Supporting Information

Chemoselective and Enantioselective Oxidation of Indoles Employing Aspartyl Peptide Catalysts

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Table of Contents

A. General.

^{1}H NMR data were collected at 500 MHz or 400 MHz. ^{1}H NMR chemical shifts are reported in parts-per-million (δ, ppm) relative to tetramethylsilane (δ = 0 ppm) with residual $CHCl_3$ (δ = 7.26 ppm) as the internal standard.^{1}H NMR spectral data are reported as follows: chemical shift (multiplicity [singlet (s), doublet (d), triplet (t), quartet (q), quintet (p), multiplet (m), broad (br)], coupling constants [Hz], integration). Proton decoupled ^{13}C NMR spectra were recorded at 126 MHz or 100 MHz. ^{13}C chemical shifts are reported in parts-per-million (δ) relative to tetramethylsilane (δ = 0 ppm), with the central line of the $CDCl_3$ triplet (77.23 ppm) serving as the internal standard. ^{19}F spectra were collected at 376 MHz. ^{19}F NMR chemical shifts are reported in parts-per-million (δ) relative to trichlorofluoromethane (δ = 0 ppm) using monofluorobenzene as the internal standard (δ = -113.1 ppm). All NMR data were collected at room temperature (23 °C).

Thin film and attenuated total reflectance (ATR) infrared (IR) spectra of neat samples were recorded on a FT-IR spectrometer; IR data (ν_{max} in cm^{-1}) is reported for diagnostic bands as well as other notable frequencies.

All of the samples were characterized by ultra high performance liquid chromatography-mass spectrometry (UPLC-MS) on an instrument equipped with a reverse-phase C18 column (1.7 μm particle size, 2.1 × 50 mm), dual atmospheric pressure chemical ionization (API)/electrospray (ESI) mass spectrometry detector, or Direct Analysis in Real Time (DART) ionization, and a photodiode array detector.

Chiral analytical normal phase HPLC was performed at a column temperature of 20 °C on a chromatograph equipped with a diode array detector (210 nm, 230 nm, 250 nm, 254 nm, or 280 nm).

Optical rotations were recorded on a polarimeter at 546, 578, and 589 nm, 23°C, with a 0.5 dm path length sample holder. Concentration are given in g/100 mL.

Analytical and preparative thin-layer chromatography (TLC) was performed using pre-coated plates (0.25 mm thickness); TLC visualization was accomplished by irradiation with a UV lamp (254 nm) and/or staining with *p*-anisaldehyde or $KMnO_4$ solutions.

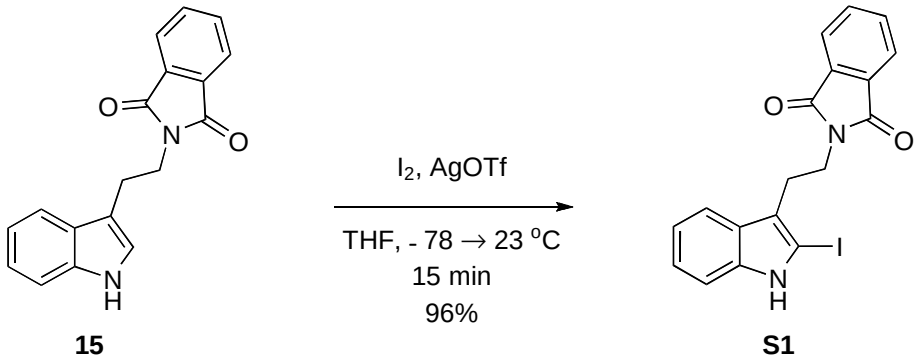
Flash column chromatography was performed using silica gel 60 Å (40-63 micron) or using an automated flash purification system. Gradient elution volumes are reported as column volumes (CV).

All yields refer to chromatographically and spectroscopically (^{1}H NMR) homogeneous materials unless indicated otherwise. Solvents were purified using a purification system. All commercially available materials were purchased from suppliers and used as received unless indicated otherwise.

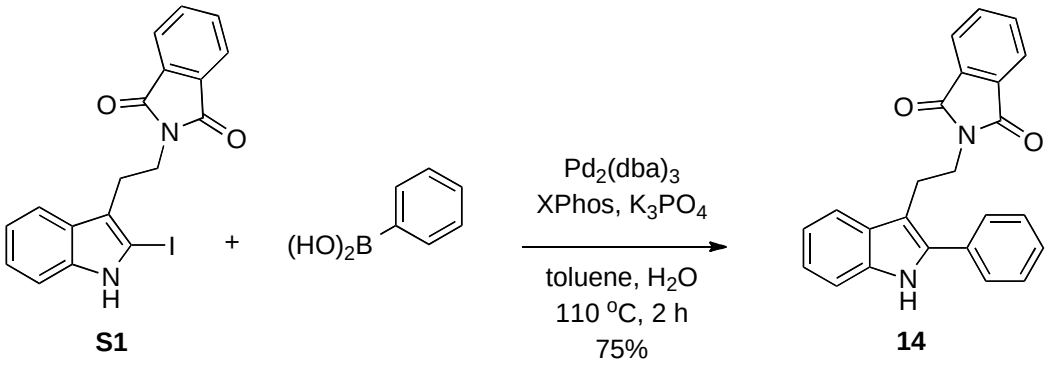
Abbreviations: AcOH = acetic acid, Boc = *tert*-butoxycarbonyl, CAM = ceric ammonium molybdate, DCM = dichloromethane, DMAP = *N*,*N*-dimethyl-4-aminopyridine, *N*,*N*-DMF = *N*,*N*-dimethylformamide, DIC = *N*,*N*-diisopropylcarbodiimide, EDC = 1-Ethyl-3-(3-dimethylaminopropyl)carbodiimide, EtOAc = ethyl acetate, Hex = Hexanes, HOBt = 1-Hydroxybenzotriazole, TEA = triethylamine, THF = tetrahydrofurane. Standard three-letter abbreviations used for proteinogenic amino acids.

B. Preparation of 2-ArylindoleDerivatives.

*With the exception of compound **19** (vide infra) all substrates used in this study were prepared according to one of two routes outlined in Scheme 1. The following detailed procedures below are representative of the conditions used to prepare the desired substrates:*

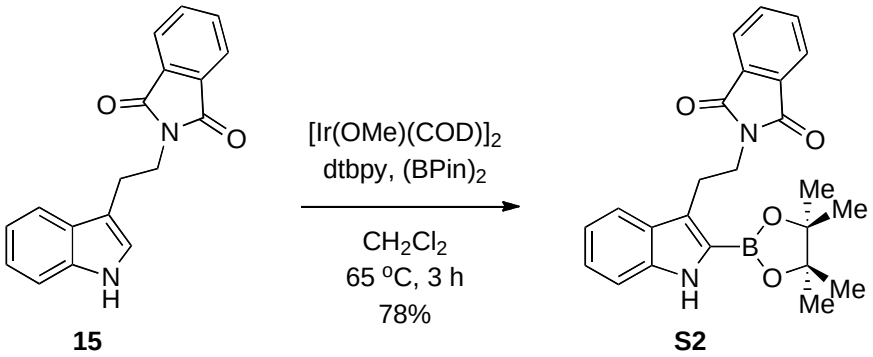


***N*,*N*-Phthaloyl-2-iodotryptamine** (**S1**, Scheme 1 - Route a). A flame dried 200-mL three–neck round bottom flask was charged with *N*,*N*-phthaloyltryptamine **15** (3.23 g, 11.1 mmol, 1 equiv.), iodine (3.12 g, 12.3 mmol, 1.11 equiv.), and a stirring bar and sealed under argon atmosphere. Anhydrous THF (110 mL) was added via syringe and the resulting mixture was agitated at 23 °C and then cooled to -78 °C. After 15 min, AgOTf (3.12 g, 12.3 mmol, 1.11 equiv.) was added via a solid addition funnel. After 5 min, the reaction mixture became a fine yellow suspension and agitation was maintained. After 25 min, TLC analysis (30% EtOAc/hexanes) indicated completion of the reaction. Solid sodium bicarbonate (2.06 g, 24.5 mmol, 2.21 equiv.) was added and the cold bath was removed. After 30 min, the resulting yellow suspension was diluted with EtOAc (100 mL) and a mixture of saturated aqueous. sodium thiosulfate–saturated aqueous sodium bicarbonate (1:1, 80 mL) at 23 °C. The mixture was filtered through Celite and the filter cake was rinsed with EtOAc (300 mL) and the organic layers were combined. The combined organic layer was washed with brine (200 mL) and dried over solid anhydrous sodium sulfate. The organic layer was filtered and concentrated at reduced pressure on a rotary evaporator to afford **S1** as a yellow crystalline solid (4.45 g, 96%). The crude product was sufficiently pure to be used in subsequent reactions without further purification. A 1-gram sample was purified for characterization by flash column chromatorgraphy on silica gel (2.4 × 14.0 cm, eluent 20% EtOAc/hexanes) to provide the product **S1** as a bright yellow solid (960 mg, 96% mass recovery). **^{1}H NMR** (500 MHz, $CDCl_3$, 20 °C, *J* in Hz) δ 7.98 (br. s, 1H, N**H**), 7.78 (dd, 2H, *J* = 3.0, 5.5 Hz, phthalimide), 7.66 (dd, 2H, *J* = 3.0, 5.5 Hz, phthalimide), 7.61 (d, 1H, *J* = 8.0 Hz, Ar**H**), 7.25 (dt, 1H, *J* = 1.0, 8.0 Hz, Ar**H**), 7.08 (dt, 1H, *J* = 1.0, 7.0 Hz, Ar**H**), 7.03 (dt, 1H, *J* = 1.5, 7.0 Hz, Ar**H**), 3.90 (t, 2H, *J* = 7.5 Hz, =C-CH_2C**H$_2$**N), 3.06 (t, 2H, *J* = 7.5 Hz, =C-C**H$_2$**CH_2N); **^{13}C NMR** (126 MHz, $CDCl_3$,20 °C) δ 168.5, 139.0, 134.0, 132.4, 127.7, 123.4, 122.6, 120.3, 118.8, 118.1, 110.6, 78.5, 37.9, 26.2; **FTIR** (neat, cm^{-1}) 3352 (br m), 3050 (w), 2935(w), 1771 (m), 1701 (s), 1653 (m), 1558 (m), 1540 (m), 1396 (s), 1362 (m), 1338 (m), 1101 (w), 742 (w), 717 (m); **HRMS** (DART) calc'd for $C_{18}H_{12}IN_2O_2$ $[M–H]^-$: 414.9949, found: 414.9965; **TLC** R_f = 0.50 (30% EtOAc/hexanes, UV, CAM).

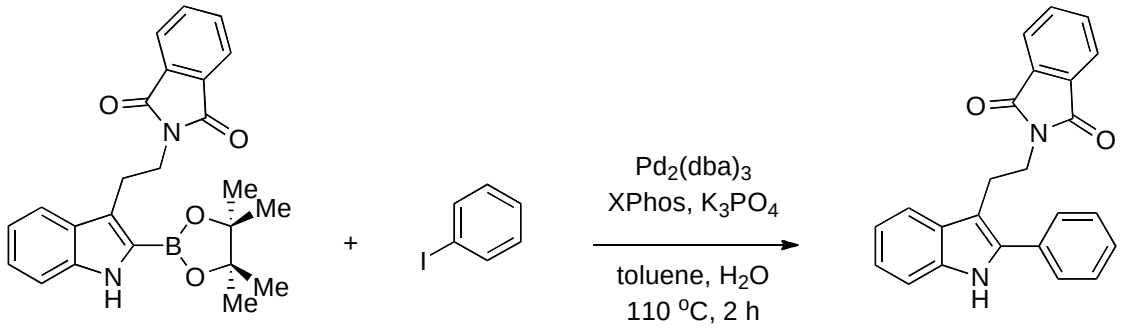


S1 **14**

***N,N*-Phthaloyl-2-phenyltryptamine** (**14**, Scheme 1 - Route a). A flame dried 15-mL pressure flask was charged with 2-iodotryptamine **S1** (100 mg, 0.240 mmol, 1 equiv.), phenylboronic acid (45.3 mg, 0.360 mmol, 1.50 equiv.), $Pd_2(dba)_3$ (5.5 mg, 6.0 μmol, 0.025 equiv.), XPhos (11.7 mg, 0.0240 mmol, 0.100 equiv.), anhydrous potassium phosphate tribasic (104 mg, 0.480 mmol, 2.00 equiv.), and a stirring bar and sealed with a septum under an atmosphere of argon. Toluene (2.4 mL) followed by deionized water (240 μL) were added via syringe and the mixture was agitated to give a clear red solution. The septum was replaced with the pressure flask's Teflon seal and the entire mixture was heated in an oil bath set to 110 °C. After 5 min, the reaction mixture turned into a pale yellow clear solution and the mixture was maintained at 110 °C. After 2 h, the oil bath was removed and the mixture was allowed to cool to 23 °C and TLC analysis (10% EtOAc in hexanes) indicated completion of the reaction. The volatiles were removed under reduced pressure and the crude mixture was purified by flash column chromatography on silica gel (3.0 × 14.0 cm, eluent 15% EtOAc/hexanes) to afford the product **14** as yellow crystals (66.2 mg, 75%). **^{1}H NMR** (500 MHz, $CDCl_3$, 20 °C, *J* in Hz) δ 8.08 (br. s, 1H, N**H**), 7.79 (d, 1H, *J* = 7.5 Hz, Ar**H**), 7.75 (dd, 2H, *J* = 3.5, 6.0 Hz, phthalimide), 7.65 (dd, 2H, *J* = 3.0, 5.5 Hz, phthalimide), 7.58-7.55 (m, 2H, Ar**H**), 7.40 (tt, 2H, *J* = 1.5, 8.0 Hz, Ar**H**), 7.35 (dt, 1H, *J* = 1.0. 8.0 Hz, Ar**H**), 7.29 (tt, 1H, *J* = 1.0, 7.0 Hz, Ar**H**), 7.19 (tt, 1H, *J* = 1.0, 8.0 Hz, Ar**H**), 7.14 (tt, 1H, *J* = 1.0, 7.0 Hz, Ar**Hz**), 3.97 (t, 2H, *J* = 8.0 Hz, =C-$CH_2C\mathbf{H_2}$N), 3.24 (t, 2H, *J* = 8.0 Hz, =C-$C\mathbf{H_2}CH_2$N); **^{13}C NMR** (126 MHz, $CDCl_3$,20 °C) δ 168.4, 136.0, 135.4, 133.9, 132.8, 132.4, 129.3, 129.2, 128.0, 128.0, 123.3, 122.7, 120.2, 119.2, 111.1, 109.3, 38.6, 24.1; **FTIR** (neat, cm^{-1}) 3359 (br. m), 3050 (w), 3021 (w), 2942 (w), 2863 (w), 1766 (m), 1701 (s), 1446 (m), 1431 (m), 1396 (s), 1356 (m), 742 (s), 716 (s), 695 (s); **HRMS** (DART) calc'd for $C_{24}H_{17}N_2O_2$ $[M–H]^-$: 365.1296, found: 365.1301; **TLC** R_f = 0.31 (20% EtOAc/hexanes, UV, CAM).

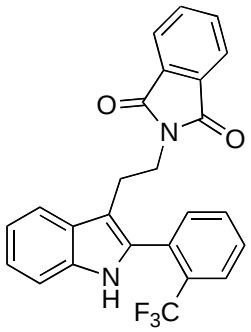


***N*,*N*-Phthaloyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)tryptamine** (**S2**, Scheme 1 - Route b). A flame dried 15-mL pressure flask was charged with (1,5-cyclooctadiene)(methoxy)iridium(I) dimer (3.4 mg, 5.2 μmol, 0.015 equiv.), 4,4'-di-*tert*-butyl-2,2'-dipyridyl (2.8 mg, 1.0 μmol, 0.03 equiv.), bis(pinacolato)diboron (177 mg, 0.690 mmol, 2.00 equiv.), *N*,*N*-phthaloyltryptamine **15** (100 mg, 0.345 mmol, 1 equiv.), and a stirring bar and sealed with a septum under an atmosphere of argon. Anhydrous dichloromethane (2.2 mL) was added via syringe to give a colorless suspension. The septum was replaced with the pressure flask's Teflon seal and the entire mixture was heated in an oil bath set to 65 °C. The reaction mixture gradually turned into a clear dark amber solution. After 3 h, the mixture was allowed to cool to 23 °C and the volatiles were removed under reduced pressure and the crude mixture was purified by flash column chromatography on silica gel (2.5 × 11.0 cm, eluent 20% EtOAc/hexanes) to afford product **S2** as a pale yellow powder (112 mg, 78%). **^{1}H NMR** (500 MHz, $CDCl_3$, 20 °C, *J* in Hz) 8.39 (br. s, 1H, N**H**), 7.75 (dd, 2H, *J* = 3.0, 5.0 Hz, phthalimide), 7.72 (d, 1H, *J* = 8.0 Hz, Ar**H**), 7.62 (dd, 2H, *J* = 3.0, 5.5 Hz, phthalimide), 7.29 (d, 1H, *J* = 8.5 Hz, Ar**H**), 7.16 (t, 1H, *J* = 7.5 Hz, Ar**H**), 7.03 (t, 1H, *J* = 7.5 Hz, Ar**H**), 3.97 (t, 2H, *J* = 7.5 Hz, =C-CH_2C**H_2**N), 3.37 (t, 2H, *J* = 7.5 Hz, =C-C**H_2**CH_2N), 1.28 (s, 12H, -B[OC(C**H_3**)$_2$]$_2$); **^{13}C NMR** (126 MHz, $CDCl_3$,20 °C) δ 168.4, 138.2, 133.8, 132.5, 128.4, 125.1, 123.9, 123.2, 119.8, 119.6, 111.5, 84.1, 39.4, 24.9, 24.7; **FTIR** (neat, cm^{-1}) 3453 (m), 3381 (br. s), 3058 (w), 2978 (s), 2935 (m), 2252 (w), 1770 (s), 1705 (s), 1619 (w), 1576 (m), 1551 (s), 1464 (m), 1436 (m), 1392 (s), 1263 (m), 1137 (s), 1105 (m), 1080 (m), 1018 (w), 961 (w), 907 (w), 857 (w), 734 (s), 713 (s); **HRMS** (DART) calc'd for $C_{24}H_{26}BN_2O_4$ $[M+H]^+$: 417.1986, found: 417.1978; **TLC** R_f = 0.48 (30% EtOAc/hexanes, UV, CAM).



S2 **14**

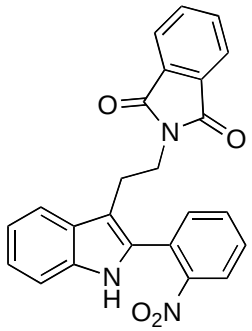
***N,N*-Phthaloyl-2-phenyltryptamine** (**14**, Scheme 1 - Route b). A flame dried 15-mL pressure flask was charged with the boronic acid ester **S2** (150 mg, 0.360 mmol, 1.50 equiv.), $Pd_2(dba)_3$ (5.5 mg, 6.0 μmol, 0.025 equiv.), XPhos (11.7 mg, 0.024 mmol, 0.10 equiv.), anhydrous potassium phosphate tribasic (104 mg, 0.480 mmol, 2.00 equiv.), and a stirring bar and sealed with a septum under an atmosphere of argon. Iodobenzene (27.4 μL, 0.240 mmol, 1 equiv.), toluene (2.4 mL), and deionized water (240 μL) were added via syringe and the mixture was agitated to give a clear red solution. The septum was replaced with the pressure flask's Teflon seal and the entire mixture was heated in an oil bath set to 110 °C. After 5 min, the reaction mixture turned into a pale yellow clear solution and the mixture was maintained at 110 °C. After 2 h, the oil bath was removed and the mixture was allowed to cool to 23 °C and TLC analysis (10% EtOAc/hexanes) indicated the completion of the reaction. The volatiles were removed under reduced pressure and the crude mixture was purified by flash column chromatography on silica gel (3.0 × 14.0 cm, eluent 15% EtOAc/hexanes) to afford the product **14** as yellow crystals (72.4 mg, 82%). (For characterization data, please see the procedure for synthesis of **14** from **S1**.)



36a

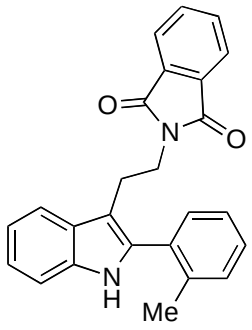
2-(2-(2-(2-(Trifluoromethyl)phenyl)-1*H*-indol-3-yl)ethyl)isoindoline-1,3-dione (36a). Purified by flash column chromatography on silica gel (20% EtOAc/Hex). Yellow foam, 188.9 mg, 90.70% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, 20 °C, *J* in Hz) δ 8.11 (br. s, 1H, N**H**), 7.81 (d, 1H, *J* = 8.0 Hz, Ar**H**), 7.78-7.72 (complex m, 3H, ArH/phthalimide), 7.64 (m, 2H, phthalimide), 7.62 (d, 1H, *J* = 7.5 Hz, Ar**H**), 7.57 (d, 1H, *J* = 7.5 Hz, Ar**H**), 7.54 (t, 1H, *J* = 8.0 Hz, Ar**H**), 7.35 (d, 1H, *J* = 8.0 Hz, Ar**H**), 7.20 (t, 1H, *J* = 8.0 Hz, Ar**H**), 7.14 (t, 1H, *J* = 7.5 Hz, Ar**H**), 3.87 (t, 2H, *J* = 8.0 Hz, =C-CH_2C**H_2**N), 3.00 (t, 2H, *J* = 8.0 Hz, =C-C**H_2**CH_2N); **^{13}C NMR** (126 MHz, $CDCl_3$,20 °C) 168.4, 135.9, 134.0, 133.7, 132.4, 132.3, 132.0, 131.1, 129.9 (q, *J* = 30.0 Hz), 129.1, 128.0, 126.6 (q, *J* = 5.2 Hz), 124.1 (q, *J* = 274.6 Hz), 123.3, 122.8, 120.1, 119.4, 111.4, 111.1, 38.4, 23.9; **FTIR** (neat, cm^{-1}) 3460 (m), 3367 (s), 3065 (m), 3022 (m), 2942 (m), 2856 (w), 1766 (m), 1709 (s), 1608 (m), 1579 (w), 1489 (m), 1450 (m), 1436 (m), 1400 (s), 1360

(m), 1310 (s), 1267 (m), 1231 (m), 1216 (m), 1173 (s), 1126 (s), 1108 (m), 1076 (m), 1036 (m), 997 (m), 961 (w), 936 (w), 867 (w), 767 (s), 749 (s), 716 (s); **HRMS** (DART) calc'd for $C_{25}H_{18}F_3N_2O_2$ $[M+H]^+$: 435.1315, found:435.1328; **TLC** R_f = 0.45 (30% EtOAc/Hex).



36b

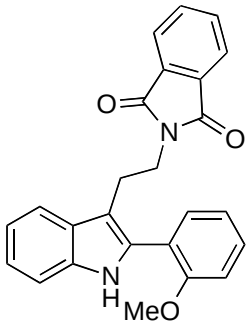
2-(2-(2-(2-Nitrophenyl)-1*H*-indol-3-yl)ethyl)isoindoline-1,3-dione (36b). Purified by flash column chromatography on silica gel (20% acetone/Hex). Orange foam, 171.8 mg, 87.00% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, 20 °C, *J* in Hz) δ 8.13 (br. s, 1H, N**H**), 7.93 (dd, 1H, *J* = 1.0, 8.0 Hz, Ar**H**), 7.78 (d, 1H, *J* = 8.0 Hz, Ar**H**), 7.74 (dd, 2H, *J* = 3.0, 5.5 Hz, phthalimide), 7.67 (dt, 1H, *J* = 1.5, 7.5 Hz, Ar**H**), 7.65 (dd, 2H, *J* = 2.5, 5.5 Hz, phthalimide), 7.61 (dd, 1H, *J* = 1.5, 7.5 Hz, Ar**H**), 7.55 (dt, 1H, *J* = 1.5, 8.0 Hz, Ar**H**), 7.33 (d, 1H, *J* = 8.0 Hz, Ar**H**), 7.20 (dd, 1H, *J* = 1.5, 8.0 Hz, Ar**H**), 7.13 (dt, 1H, *J* = 1.0, 8.0 Hz, Ar**H**), 3.86 (t, 2H, *J* = 8.0 Hz, =C-$CH_2C\mathbf{H_2}$N), 3.00 (t, 2H, *J* = 8.0 Hz, =C-$C\mathbf{H_2}CH_2$N)); **^{13}C NMR** (126 MHz, $CDCl_3$,20 °C) δ 168.3, 149.6, 136.3, 134.0, 133.6, 132.9, 132.2, 130.3, 129.7, 128.1, 127.0, 124.6, 123.3, 123.1, 120.2, 119.4, 111.7, 111.4, 38.3, 23.8; **FTIR** (neat, cm^{-1}) 3374 (s), 3058 (m), 3022 (m), 2942 (m), 2863 (m), 1766 (m), 1705 (s), 1612 (m), 1522 (s), 1453 (m), 1439 (m), 1400 (s), 1356 (s), 1346 (s), 1184 (m), 1126 (m), 1101 (m), 993 (m), 849 (m), 749 (s), 716 (s); **HRMS** (DART) calc'd for $C_{24}H_{18}N_3O_4$ $[M+H]^+$: 412.1292, found: 412.1250; **TLC** R_f = 0.21 (30% EtOAc/Hex).



36c

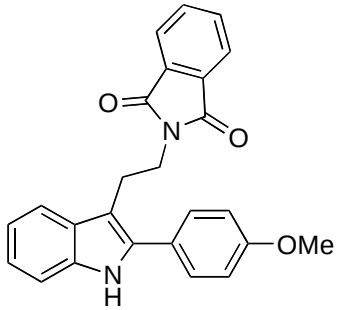
2-(2-(2-(o-Tolyl)-1*H*-indol-3-yl)ethyl)isoindoline-1,3-dione (36c). Purified by flash column chromatography on silica gel (20% EtOAc/Hex). Yellow foam, 180 mg, 99.0% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, 20 °C, *J* in Hz) δ 7.95 (br. s, 1H, N**H**), 7.76 (d, 1H, *J* = 7.5 Hz, Ar**H**), 7.71 (dd, 2H, *J* = 3.0, 5.0 Hz, phthalimide), 7.63 (dd, 2H, *J* = 3.0, 5.5 Hz), 7.34-7.29 (complex m, 2H, Ar**H**), 7.26 (dt, 1H, *J* = 1.5, 7.5 Hz, Ar**H**), 7.22 (dd, 1H, *J* = 0.5, 7.5 Hz, Ar**H**), 7.19-7.14 (complex m, 2H, Ar**H**), 7.10 (dt, 1H, *J* = 1.0, 7.5 Hz, Ar**H**), 3.86 (t, 2H, *J* = 7.5 Hz, =C-$CH_2C\mathbf{H_2}$N), 3.01 (t, 2H, *J* = 7.5 Hz, =C-$C\mathbf{H_2}CH_2$N), 2.22 (s, 3H, -CH_3); **^{13}C NMR** (126 MHz, $CDCl_3$,20 °C) δ 168.4, 137.6, 135.8, 135.5, 133.9, 132.4, 132.2,

131.0, 130.6, 128.8, 128.5, 125.9, 123.2, 122.2, 119.9, 119.1, 111.0, 110.1, 38.5, 23.9, 20.2; **FTIR** (neat, cm^{-1}) 3453 (m), 3374 (s), 3050 (m), 3022 (m), 2928 (m), 2849 (m), 2245 (m), 1770 (s), 1705 (s), 1615 (m), 1486 (m), 1453 (s), 1436 (s), 1396 (s), 1356 (s), 1338 (s), 1302 (s), 1234 (w), 1187 (w), 1169 (m), 1123 (m), 1101 (s), 1087 (m), 1040 (m), 1008 (s), 936 (m), 871 (m), 763 (s), 734 (s), 720 (s); **HRMS** (DART) calc'd for $C_{25}H_{21}N_2O_2$ $[M+H]^+$: 381.1598, found: 381.1608; **TLC** R_f = 0.24 (20% acetone/Hex).



36d

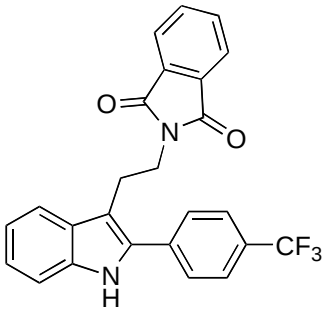
2-(2-(2-(2-methoxyphenyl)-1*H*-indol-3-yl)ethyl)isoindoline-1,3-dione (36d). Purified by flash column chromatography on silica gel (20% EtOAc/Hex). Yellow foam, 135.9 mg, 23.00% yield (unoptimized). **^{1}H NMR** (500 MHz, $CDCl_3$, 20 °C, *J* in Hz) δ 7.83 (dd, 2H, *J* = 3.0, 5.0 Hz, phthalimide), 7.77 (m, 1H, Ar**H**), 7.69 (dd, 2H, *J* = 3.0,, 5.5 Hz, phthalimide), 7.34 (dt, 1H, *J* = 2.0, 7.5 Hz, Ar**H**), 7.33 (d, 1H, *J* = 8.0 Hz, Ar**H**), 7.18-7.12 (complex m, 4H, phthalimide/ Ar**H**), 7.09-7.02 (complex m, 2H, Ar**H**), 4.04 (t, 2H, *J* = 8.0 Hz, =C-CH_2C**H_2**N), 3.73 (s, 3H, -OMe), 3.18 (t, 2H, *J* = 8.0 Hz, =C-C**H_2**CH_2N); **^{13}C NMR** (126 MHz, $CDCl_3$,20 °C) δ 168.6, 154.5, 137.2, 134.1, 132.5, 128.5, 128.3, 128.2, 128.2, 127.4, 123.4, 122.3, 121.1, 119.9, 119.2, 112.6, 112.5, 111.2, 55.9, 38.7, 24.8;**FTIR** (neat, cm^{-1}) 3050 (w), 2928 (w), 2849 (w), 2835 (w), 1770 (m), 1709 (s), 1608 (w), 1594 (m), 1507 (s), 1461 (s), 1436 (m), 1396 (s), 1371 (m), 1356 (m), 1249 (m), 1227 (m), 1094 (m), 1018 (m), 993 (m), 867 (w), 745 (s), 716 (s); **HRMS** (DART) calc'd for $C_{25}H_{21}N_2O_3$ $[M+H]^+$: 397.1547, found: 397.1535; **TLC** R_f = 0.38 (30% EtOAc/Hex).



36e

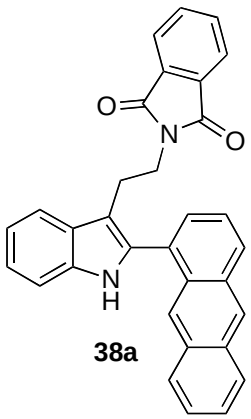
2-(2-(2-(4-Methoxyphenyl)-1*H*-indol-3-yl)ethyl)isoindoline-1,3-dione (36e). Purified by flash column chromatography on silica gel (20% EtOAc/Hex). Yellow foam, 253 mg, 99.0% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, 20 °C, *J* in Hz) δ 7.98 (br. s, 1H, N**H**), 7.76-7.72 (complex m, 3H, phthalimide/Ar**H**), 7.66 (dd, 2H, *J* = 3.0, 5.5 Hz, phthalimide), 7.49 (d, 2H, *J* = 8.0 Hz, Ar**H**), 7.33 (d, 1H, *J* = 7.5 Hz, Ar**H**), 7.17 (t, 1H, *J* = 7.0 Hz, Ar**H**), 7.13 (t, 1H, *J* = 7.0 Hz, Ar**H**), 6.93 (d, 2H, *J* = 8.0 Hz, Ar**H**), 3.96 (t, 2H, *J* = 7.5 Hz, =C-CH_2C**H_2**N), 3.81 (s, 3H, -OMe), 3.21 (t, 2H, *J* = 8.0 Hz, =C-C**H_2**CH_2N); **^{13}C NMR** (126 MHz,

$CDCl_3$, 20 °C) δ 168.5, 159.5, 135.9, 135.4, 133.9, 132.4, 129.4, 129.3, 125.4, 123.3, 122.4, 120.1, 119.0, 114.6, 110.9, 108.6, 55.5, 38.6, 24.1; **FTIR** (neat, cm^{-1}) 3367 (s), 3058 (w), 2921 (m), 2849 (m), 2835 (m), 1770 (m), 1705 (s), 1608 (m), 1507 (m), 1461 (s), 1443 (s), 1396 (s), 1356 (m), 1281 (m), 1249 (s), 1177 (m), 1123 (m), 1101 (m), 1029 (m), 997 (m), 907 (m), 835 (m), 734 (s), 716 (s); **HRMS** (DART) calc'd for $C_{25}H_{21}N_2O_3$ $[M+H]^+$: 397.1547, found: 397.1532; **TLC** R_f = 0.30 (30% EtOAc/Hex).



36f

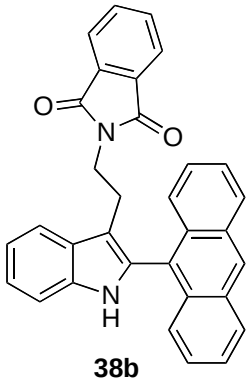
2-(2-(2-(4-(Trifluoromethyl)phenyl)-1*H*-indol-3-yl)ethyl)isoindoline-1,3-dione (36f). Purified by flash column chromatography on silica gel (20% EtOAc/Hex). Yellow foam, 268 mg, 96.4% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, 20 °C, *J* in Hz) 8.09 (br. s, 1H, N**H**), 7.78 (d, 1H, *J* = 8.0 Hz, Ar**H**), 7.72 (dd, 2H, *J* = 3.0, 5.5 Hz, phthalimide), 7.68-7.63 (complex m, 4H), 7.60 (d, 2H, *J* = 8.5 Hz, Ar**H**), 7.37 (d, 1H, *J* = 8.0 Hz, Ar**H**), 7.23 (dt, 1H, *J* = 1.0, 8.0 Hz, Ar**H**), 7.17 (dt, 1H, *J* = 1.0, 8.0 Hz, Ar**H**), 3.95 (t, 2H, *J* = 7.5 Hz, =C-CH_2C$\mathbf{H_2}$N), 3.27 (t, 2H, *J* = 8.0 Hz, =C-C$\mathbf{H_2}$$CH_2$N); **^{13}C NMR** (126 MHz, $CDCl_3$,20 °C) 168.4, 136.3, 136.3, 134.1, 133.7, 132.2, 129.6 (q, *J* = 32.9 Hz), 129.2, 128.0, 126.1 (q, *J* = 4.0 Hz), 124.2 (q, *J* = 272.8 Hz), 123.5, 123.3, 120.5, 119.4, 111.3, 111.0, 38.5, 24.0; **FTIR** (neat, cm^{-1}) 3460 (w), 3374 (m), 3065 (w), 3022 (w), 2935 (w), 2863 (w), 1770 (m), 1705 (s), 1615 (m), 1457 (m), 1439 (m), 1396 (s), 1356 (m), 1320 (s), 1256 (w), 1166 (s), 1119 (s), 1069 (s), 1015 (m), 997 (w), 936 (w), 846 (m), 749 (s), 716 (s); **HRMS** (DART) calc'd for $C_{25}H_{18}F_3N_2O_2$ $[M+H]^+$: 435.1315, found: 435.1320; **TLC** R_f = 0.38 (20% EtOAc/Hex).



38a

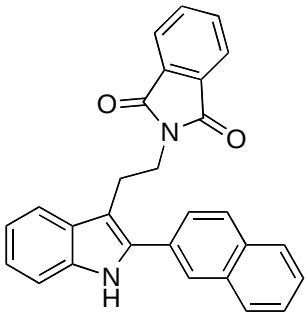
2-(2-(2-(Anthracen-1-yl)-1*H*-indol-3-yl)ethyl)isoindoline-1,3-dione (38a). Purified by flash column chromatography on silica gel (20% EtOAc/Hex). Yellow foam, 206.2 mg, 92.14% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, 20 °C, *J* in Hz) 8.32 (s, 1H, Ar**H**), 8.24 (s, 1H, Ar**H**), 8.21 (br. s, 1H, N**H**), 7.96 (d, 1H, *J* = 8.5 Hz, Ar**H**), 7.92 (d, 1H, *J* = 8.5 Hz, Ar**H**), 7.82 (d, 1H, *J* = 8.0 Hz, Ar**H**), 7.77 (d, 1H, *J* = 8.5 Hz,

Ar**H**), 7.52 (d, 1H, *J* = 6.5 Hz, Ar**H**), 7.47-7.38 (m, 3H, Ar**H**), 7.38-7.30 (m, 5H, phthalimide/Ar**H**), 7.25 (d, 1H, *J* = 7.5 Hz, Ar**H**), 7.18 (t, 1H, *J* = 8.0 Hz, Ar**H**), 3.85 (br. s, 2H, =C-$CH_2C\mathbf{H_2}$N), 3.19 (br. s, 2H, =C-$C\mathbf{H_2}CH_2$N); **^{13}C NMR** (126 MHz, $CDCl_3$,20 °C) δ168.2, 136.1, 134.6, 133.3, 132.0, 131.8, 131.8, 131.7, 130.7, 130.1, 129.3, 128.8, 128.6, 128.5, 128.0, 127.0, 125.9, 125.7, 124.9, 124.8, 122.6, 122.5, 120.0, 119.1, 111.5, 111.2, 38.7, 23.5; **FTIR** (neat, cm^{-1}) 3356 (s), 3048 (m), 3014 (w), 2924 (m), 2852 (w), 1768 (m), 1709 (s), 1614 (m), 1460 (m), 1438 (m), 1393 (s), 1362 (m), 1337 (m), 1309 (m), 1102 (m), 1018 (m), 878 (m), 747 (s), 738 (s), 713 (s), 666 (m); **HRMS** (DART) calc'd for $C_{32}H_{23}N_2O_2$ $[M+H]^+$: 467.1754, found: 467.1738; **TLC** R_f = 0.47 (30% EtOAc/Hex).



38b

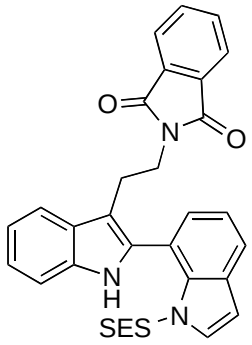
2-(2-(2-(Anthracen-9-yl)-1*H*-indol-3-yl)ethyl)isoindoline-1,3-dione (38b). Purified by flash column chromatography on silica gel (20% EtOAc/Hex). Yellow foam, 211 mg, 94.2% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, 20 °C, *J* in Hz) δ 8.51 (s, 1H, Ar**H**), 8.15 (br. s, 1H, N**H**), 8.00 (d, 2H, *J* = 8.5 Hz, Ar**H**), 7.83 (d, 1H, *J* = 7.5 Hz, Ar**H**), 7.69 (d, 2H, *J* = 8.5 Hz, Ar**H**), 7.54 (app. br. s, 4H, phthalimide), 7.41-7.37 (complex m, 3H, Ar**H**), 7.28-7.22 (complex m, 3H, Ar**H**), 7.16 (dt, 1H, *J* = 1.0, 8.0 Hz, Ar**H**), 3.75 (t, 2H, *J* = 7.0 Hz, =C-$CH_2C\mathbf{H_2}$N), 2.92 (t, 2H, *J* = 7.5 Hz, =C-$C\mathbf{H_2}CH_2$N); **^{13}C NMR** (126 MHz, $CDCl_3$,20 °C) δ 168.2, 136.5, 133.7, 132.2, 131.9, 131.8, 131.4, 128.7, 128.6, 128.5, 126.5, 126.5, 126.4, 125.5, 123.0, 122.4, 120.0, 119.2, 113.4, 111.1, 38.4, 24.1; **FTIR** (neat, cm^{-1}) 3356 (s), 3053 (m), 3025 (m), 2936 (w), 2852 (w), 1768 (m), 1706 (s), 1614 (w), 1488 (w), 1460 (m), 1438 (m), 1399 (s), 1359(m), 1334 (m), 1217 (m), 1119 (m), 1024 (m), 892 (m), 738 (s), 710 (s); **HRMS** (DART) calc'd for $C_{32}H_{23}N_2O_2$ $[M+H]^+$: 467.1754, found: 467.1763; **TLC** R_f = 0.42 (30% EtOAc/Hex).



38c

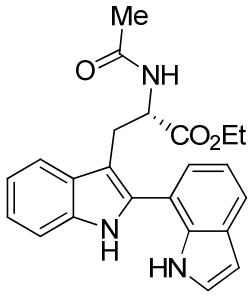
2-(2-(2-(Naphthalen-2-yl)-1*H*-indol-3-yl)ethyl)isoindoline-1,3-dione (38c). Purified by flash column chromatography on silica gel (20% acetone/Hex). Yellow foam, 266 mg, 99.0% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, 20 °C, *J* in Hz) δ : 8.18 (br. s, 1H, N**H**), 8.01 (s, 1H, Ar**H**), 7.90 (d, 1H, *J* = 8.0 Hz, Ar**H**),

7.84 (d, 1H, *J* = 8.0 Hz, Ar**H**), 7.80 (dd, 1H, *J* = 0.5, 8.0 Hz, Ar**H**), 7.77 (d, 1H, *J* = 7.5 Hz, Ar**H**), 7.68 (dd, 1H, *J* = 2.0, 8.5 Hz, Ar**H**), 7.64 (dd, 2H, *J* = 3.0, 5.5 Hz, phthalimide), 7.52 (dd, 2H, *J* = 3.0, 5.5 Hz, phthalimide), 7.49 (dt, 1H, *J* = 1.0, 7.0 Hz, Ar**H**), 7.45 (dt, 1H, *J* = 1.5, 7.5 Hz, Ar**H**), 7.39 (dd, 1H, *J* = 0.5, 8.0 Hz, Ar**H**), 7.21 (dd, 1H, *J* = 1.5, 8.0 Hz, Ar**H**), 7.17 (dt, 1H, *J* = 1.0, 7.0 Hz, Ar**H**), 4.02 (t, 2H, *J* = 7.5 Hz, =C-CH_2C**H**$_2$N), 3.34 (t, 2H, *J* = 7.5 Hz, =C-C**H**$_2$$CH_2$N); **^{13}C NMR** (126 MHz, $CDCl_3$,20 °C) 168.4, 136.2, 135.3, 133.8, 133.7, 132.7, 132.2, 130.2, 129.5, 128.9, 128.5, 127.9, 126.9, 126.7, 126.4, 125.6, 123.1, 122.9, 120.2, 119.2, 111.1, 109.9, 38.6, 24.2; **FTIR** (neat, cm^{-1}) 3453 (m), 3374 (s), 3058 (m), 3022 (m), 2935 (m), 2863 (w), 1766 (s), 1709 (s), 1612 (m), 1601 (m), 1551 (m), 1453 (m), 1436 (s), 1396 (s), 1360 (s), 1335 (m), 1306 (m), 1259 (m), 1231 (m), 1213 (m), 1144 (m), 1011 (m), 1000 (m), 860 (m), 817 (m), 749 (s), 716 (s); **HRMS** (DART) calc'd for $C_{28}H_{21}N_2O_2$ $[M+H]^+$: 417.1598, found: 417.1593; **TLC** R_f = 0.47 (30% EtOAc/Hex).



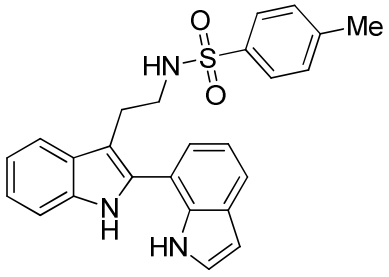
42

2-(2-(1'-((2-(trimethylsilyl)ethyl)sulfonyl)-1*H*,1'*H*-[2,7'-biindol]-3-yl)ethyl)isoindoline-1,3-dione (42). Purified by flash column chromatography on silica gel (1:1:8 CH_2Cl_2EtOAc/Hex). Yellow solid, 74.8 mg, 96.0% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, 20 °C, *J* in Hz) 8.74 (br. s, 1H, N**H**), 7.71 (dd, 2H, *J* = 3.0, 5.5 Hz, phthalimide), 7.70-7.65 (complex m, 2H, Ar**H**), 7.63 (dd, 2H, *J* = 3.0, 5.5 Hz, phthalimide), 7.51 (d, 1H, *J* = 3.5 Hz, Ar**H**), 7.39 (d, 1H, *J* = 8.0 Hz, Ar**H**), 7.36 (dd, 1H, *J* = 1.5, 7.5 Hz, Ar**H**), 7.31 (t, 1H, *J* = 7.5 Hz, Ar**H**), 7.19 (dt, 1H, *J* = 1.0, 8.0 Hz, Ar**H**), 7.07 (t, 1H, *J* = 7.5 Hz, Ar**H**), 7.66 (d, 1H, *J* = 3.5 Hz, Ar**H**), 3.81 (t, 2H, *J* = 7.5 Hz, =C-CH_2C**H**$_2$N), 2.83 (app. br. s, 2H, =C-C**H**$_2$$CH_2$N), 2.61 (m, 2H, -$SO_2$C**H**$_2$$CH_2$$SiMe_3$), 0.66 (m, 2H, -$SO_2CH_2$C**H**$_2$$SiMe_3$), –0.28 (s, 9H, -$SiMe_3$); **^{13}C NMR** (126 MHz, $CDCl_3$,20 °C) δ 168.3, 135.7, 134.6, 134.0, 133.7, 133.3, 132.3, 130.1, 130.0, 127.4, 123.5, 123.3, 123.0, 122.9, 120.2, 119.3, 118.9, 112.1, 111.5, 108.0, 51.4, 38.5, 24.3, 10.2, –2.0; **FTIR** (neat, cm^{-1})3381 (m), 3108 (w), 3050 (w), 3022 (w), 2950 (m), 2892 (w), 1766 (m), 1705 (s), 1612 (w), 1457 (m), 1436 (m), 1400 (s), 1360 (s), 1306 (m), 1249 (s), 1231 (m), 1169 (s), 1155 (s), 1126 (s), 1101 (m), 1069 (m), 1018 (m), 979 (m), 889 (w), 857 (m), 839 (m), 799 (m), 745 (s), 716 (s), 698 (m), 662 (m); **HRMS** (DART) calc'd for $C_{31}H_{30}N_3O_4SSi$ $[M–H]^–$: 568.1732, found: 568.1732; **TLC** R_f = 0.16 (1:1:8 CH_2Cl_2/EtOAc/Hex).



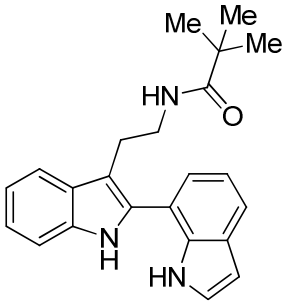
25

(2'*S*)-3-(2-*N*-Acetylamino-2-ethoxycarbonylethyl)-2-(7-indolyl)-1*H*-indole (**25**). Purified by column chromatography on silica gel (50% EtOAc/Hex). Yellow foam, 514.91 mg, 73% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 9.02 (s, 1H), 8.19 (s, 1H), 7.73 (dd, *J* = 7.5, 0.8, 1H), 7.63 (d, *J* = 7.9, 1H), 7.37 (d, *J* = 8.0, 1H), 7.27 – 7.17 (m, 5H), 6.64 (dd, *J* = 3.1, 2.0, 1H), 5.77 (d, *J* = 8.3, 1H), 4.78 (ddd, *J* = 8.3, 6.8, 5.2, 1H), 3.83 (dq, *J* = 10.7, 7.1, 1H), 3.46 (dq, *J* = 10.7, 7.1, 1H), 3.40 (dd, *J* = 14.6, 5.1, 1H), 3.30 (dd, *J* = 14.6, 6.8, 1H), 1.48 (s, 3H), 1.01 (t, *J* = 7.1, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 172.0, 170.0, 136.2, 135.0, 133.6, 129.5, 128.8, 125.3, 123.1, 122.7, 121.6, 120.3, 120.2, 119.1, 116.4, 111.3, 108.7, 103.2, 61.5, 53.1, 27.5, 22.8, 14.0; **FTIR** (cm^{-1}) 3395, 2929, 1727, 1653, 1514, 1435, 1335, 1211, 1025; **UPLC-MS** (ESI) *m/z* 376.17 (calculated for$C_{22}H_{21}N_3O_3$[M + H^+] = 376.16); **TLC** R_f = 0.31 (50% EtOAc/Hex); $[\alpha]_{546\,nm}^{20\,℃}$ = +177.7 (*c* = 0.115, $CHCl_3$).



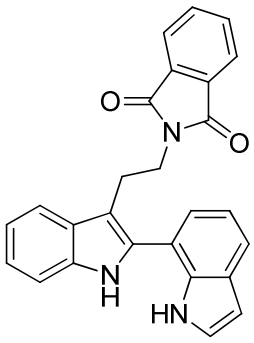
40a

3-(2-*N*-(4-Toluenesulfonyl)aminoethyl)-2-(7-indolyl)-1*H*-indole (**40a**). Purified by column chromatography on silica gel (30% EtOAc/Hex). Yellow foam, 1.30 g, 43% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 9.19 (brs, 1H), 8.56 (brs, 1H), 7.80 – 7.74 (m, 1H), 7.72 (t, *J* = 6.4, 1H), 7.63 (t, *J* = 7.0, 1H), 7.48 (t, *J* = 7.3, 2H), 7.30 – 7.23 (m, 4H), 7.19 – 7.11 (m, 3H), 6.71 – 6.65 (m, 1H), 3.28 – 3.19 (m, 2H), 2.97 (q, *J* = 6.6, 2H), 2.39 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 143.4, 141.7, 136.7, 134.5, 133.3, 130.1, 129.8, 128.7, 127.4, 127.2, 125.2, 123.1, 122.3, 121.5, 120.3, 119.9, 116.4, 109.1, 103.3, 84.2, 43.2, 25.2, 21.7; **FTIR** (cm^{-1}) 3389, 3057, 2926, 2852, 1597, 1428, 1153, 1092; **UPLC-MS** (ESI)*m/z*430.16 (calculated for $C_{25}H_{23}N_3O_2S$ [M + H^+] = 430.15); **TLC** R_f = 0.59 (6% EtOAc/DCM).



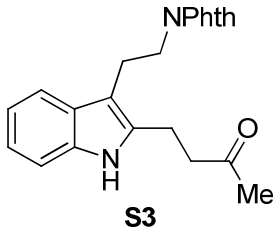
40b

3-(2-*N*-Pivaloylaminoethyl)-2-(7-indolyl)-1*H*-indole(**40b**). Purified by column chromatography on silica gel (25% EtOAc/Hex). Yellow foam, 1.46 g, 55% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 9.17 (s, 1H), 8.13 (s, 1H), 7.72 (dd, *J* = 7.6, 4.3, 2H), 7.40 (d, *J* = 7.9, 1H), 7.30 – 7.28 (m, 1H), 7.26 – 7.18 (m, 3H), 6.64 (dd, *J* = 3.1, 2.0, 1H), 5.71 – 5.63 (m, 1H), 3.52 (dd, *J* = 12.9, 6.7, 2H), 2.99 (t, *J* = 6.8, 2H), 0.91 (s, 9H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 178.8, 136.4, 134.8, 132.9, 129.3, 128.7, 125.3, 123.1, 122.6, 121.4, 120.1, 120.0, 119.4, 116.5, 111.4, 111.3, 103.0, 40.1, 38.6, 27.4, 25.0; **FTIR** (cm^{-1}) 3401, 2964, 1638, 1514, 1431, 1334, 1198; **UPLC-MS** (ESI) *m/z* 360.22 (calculated for $C_{23}H_{25}N_3O$ [M + H^+] = 360.20); **TLC** R_f = 0.47 (35% EtOAc/Hex).



40c

3-(2-*N*,*N*-Phthaloylaminoethyl)-2-(7-indolyl)-1*H*-indole (**40c**). Purified by column chromatography on silica gel (25% EtOAc/Hex). Yellow solid, 138 mg, 69% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 9.07 (s, 1H), 8.07 (s, 1H), 7.67 – 7.59 (m, 5H), 7.55 (d, *J* = 8.0 Hz, 1H), 7.33 (d, *J* = 8.1 Hz, 1H), 7.27 – 7.23 (m, 2H), 7.18 – 7.14 (m, 1H), 7.14 – 7.10 (m, 1H), 6.97 – 6.93 (m, 1H), 6.58 (dd, *J* = 3.1, 2.1 Hz, 1H), 3.88 (t, *J* = 6.7 Hz, 2H), 3.19 (t, *J* = 6.7 Hz, 2H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 168.73, 136.17, 135.10, 133.82, 133.10, 132.03, 129.18, 128.52, 125.08, 123.25, 123.06, 122.43, 121.34, 120.09, 120.03, 118.72, 116.31, 111.11, 111.08, 103.16, 39.04, 23.62; **FTIR** (cm^{-1}) 3339, 3289, 3066, 2947, 2911, 1761, 1694, 1614, 1337, 1277; **UPLC-MS** (ESI) *m/z*406.15 (calculated for $C_{26}H_{19}N_3O_2$[M + H^+] = 406.15); **TLC** R_f = 0.31 (25% EtOAc/Hex).

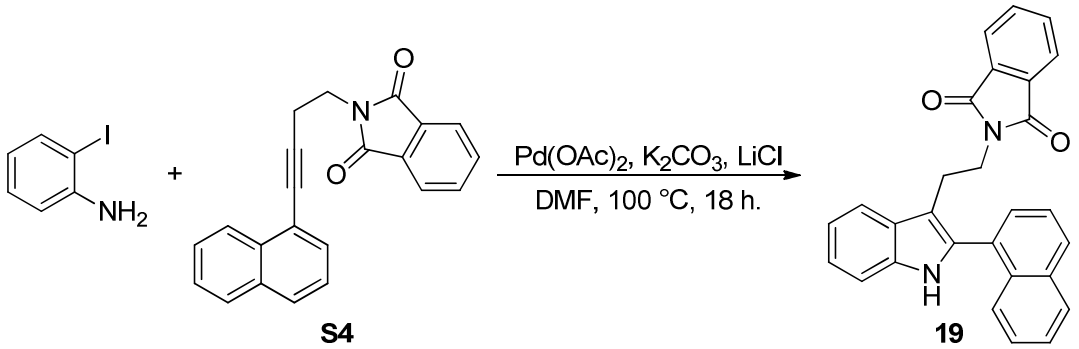


S3

3-(2-*N*,*N*-Phthaloylaminoethyl)-2-(3-oxobutyl)-1*H*-indole (**S3**). Prepared by the reported procedure.[5] Purified by preparative TLC (50% EtOAc/Hex). Yellow solid, 87.8 mg, 25% yield.**^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 8.51 (s, 1H), 7.85 (dd, *J* = 5.4, 3.0, 2H), 7.72 (dd, *J* = 5.5, 3.0, 2H), 7.66 (d, *J* = 7.7, 1H), 7.27 (d, *J* = 7.9, 2H), 7.13 – 7.09 (m, 1H), 7.09 – 7.04 (m, 1H), 3.91 – 3.85 (m, 2H), 3.09 – 3.01 (m, 4H), 2.93 – 2.87 (m, 2H), 2.19 (s, 3H);**^{13}C NMR** (126 MHz, $CDCl_3$) δ 209.9, 168.5, 135.9, 135.3, 134.1, 132.4, 128.2, 123.4, 121.6, 119.5, 118.4, 110.8, 107.7, 44.1, 38.9, 30.3, 23.7, 19.4; **FTIR** (cm^{-1}) 1770, 1703, 1615, 1436, 1395, 1359, 1265, 1162, 1020; **UPLC-MS** (ESI) *m/z* 361.15 (calculated for $C_{22}H_{20}N_2O_3$[M + H^+] = 361.15);**TLC** R_f = 0.56 (50% EtOAc/Hex).

3-(2-*N*,*N*-Phthaloylaminoethyl)-2-(1-naphthyl)-1*H*-indole (19)

Compound was prepared by a modification of a reported procedure.[6]



S4 **19**

Notes:

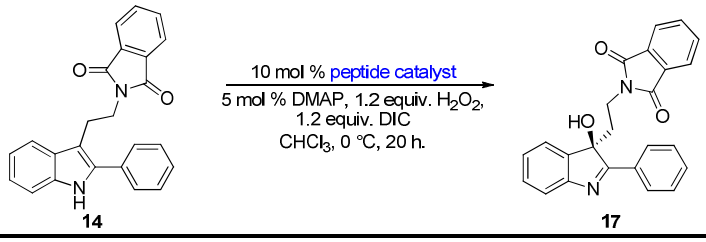
- 2-Iodoaniline was recrystallized from boiling hexanes.
- Lithium chloride was kept under high vacuum and flame-dried for 60 seconds prior to use.
- Dry *N*,*N*-dimethylformamide was degassed by freeze-pump-thaw procedure.

A flame dried 500-mL round bottom flask was charged with 2-iodoaniline (2.18 g, 9.95 mmol, 1.3 equiv.), $Pd(OAc)_2$ (340 mg, 1.51 mmol, 0.2 equiv.), K_2CO_3 (3.17 g, 22.94 mmol, 3.0 equiv.) and LiCl (200 mg, 4.72 mmol, 0.62 equiv.). The atmosphere of the flask was evacuated and refilled with nitrogen. To this mixture was added *N*,*N*-DMF (103 mL), followed by the solution of alkyne **S4** (2.49 g, 7.65 mmol, 1.0 equiv.) in *N*,*N*-DMF (50 mL). The flask was sealed, flushed with nitrogen and heated at 100 °C in an oil bath for 18 hours. After this time, alkyne **S4** could not be detected by TLC. The reaction mixture was cooled to room temperature, diluted with 500 mL of EtOAc, transferred to a separatory funnel and extracted sequentially with 500 mL of distilled water, 500 mL of 1 M HCl, 500 mL of saturated $NaHCO_3$,

and 500 mL of brine. Organic phase was dried over $MgSO_4$, filtered, and the solvent was removed *in vacuo*. The black residue was purified by column chromatography (20% EtOAc/Hex), and thus obtained material was further purified by trituration in boiling DCM and dried under high vacuum. Compound **19** was obtained as a pale yellow solid, 1.27 g (39% yield).

^{1}H NMR (500 MHz, $CDCl_3$, *J* in Hz) δ 8.15 (s, 1H), 7.88 – 7.84 (m, 2H), 7.83 (d, *J* = 7.9, 1H), 7.78 (d, *J* = 8.4, 1H), 7.64 – 7.57 (m, 5H), 7.50 (dd, *J* = 8.2, 7.1, 1H), 7.45 (ddd, *J* = 8.1, 6.8, 1.1, 1H), 7.39 (d, *J* = 8.0, 1H), 7.36 (ddd, *J* = 8.2, 6.8, 1.2, 1H), 7.24 (ddd, *J* = 8.3, 7.2, 1.1, 1H), 7.17 (ddd, *J* = 8.5, 7.2, 0.9, 1H), 3.88 (t, *J* = 7.3, 2H), 3.18 – 3.07 (m, 2H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 168.3, 136.0, 134.3, 133.9, 133.7, 132.5, 132.2, 130.2, 129.1, 128.9, 128.6, 128.5, 126.8, 126.2, 125.9, 125.5, 123.0, 122.5, 120.0, 119.2, 111.4, 111.0, 38.7, 23.8; **FTIR** (cm^{-1}) 3447, 3354, 3054, 2952, 2915, 1765, 1692, 1614, 1431, 1397, 1360, 1017, 971; **UPLC-MS** (ESI) *m/z* 417.16 (calculated for $C_{28}H_{20}N_2O_2$[M + H^+] = 417.15); **TLC** R_f = 0.44 (30% EtOAc/Hex).

C. Structure–Enantioselectivity Relationships for Peptide Catalyzed Indole Oxidation.

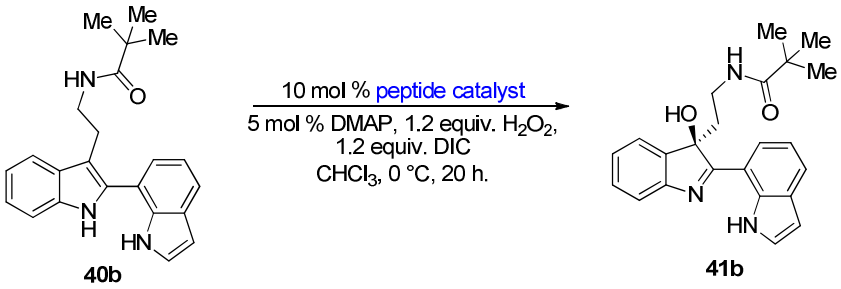


14 **17**

			Residue Position					
Entry	*i*-1	*i* = catalytic	*i*+1	*i*+2	*i*+3	*i*+4	*i*+5	*e.r.*
1		Asp	Pro	D-Val	Leu	Gly		67:33
2		Asp	Pro	D-Val	Dhl	Gly		67:33
3		Asp	Pro	D-Val	Tba	Gly		64:36
4		Asp	Pro	D-Val	Cha	Gly		61:39
5		Asp	Pro	D-Val	Cpa	Gly		59:41
6		Asp	Pro	D-Val	D-Leu	Gly		53:47
7		Asp	Pro	D-Val	Ile	Gly		62:38
8		Asp	Pro	D-Val	Val	Gly		61:39
9		Asp	Pro	D-Val	Val	Val	Gly	59:41
10		Asp	Pro	D-Val	Chg	Gly		60:40
11		Asp	Pro	D-Val	Phe			59:41
12		Asp	Pro	D-Val	MEA			57:43
13		Asp	Pro	D-Val	*trt*-Gln	Gly		56:44
14		Asp	Pro	D-Val	Boc_2Arg	Gly		55:45
15		Asp	Pro	D-Val	*tert*-Leu	Gly		52:48
16		Asp	Pro	D-Val	(*R*)-PEA			60:40
17		Asp	Pro	D-Val	(*R*)-2-NEA			62:38
18		Asp	Pro	D-Val	(*R*)-1-NEA			58:42
19		Asp	Pro	D-Val	(*S*)-1-NEA			52:48
20		Asp	Pip	D-Val	Leu	Gly		67:33
21		Asp	Bn-Hyp	D-Val	Leu	Gly		67:33
22		Asp	Ala	D-Val	Leu	Gly		62:38
23		Asp	Pro	D-*t*Bu-Thr	Leu	Gly		55:45
24		Asp	D-Pro	Val	Phe			47:53
25		Asp	D-Pro	Aib	Phe			49:51
26		Asp	Val	Pro	D-Val	Val	DPMA	52:48
27		D-Asp	D-Pro	Val	D-Val	Abi		41:59
28		Asp	Pro	D-Val	Cha	Gly	NBA	59:41
29		Asp	Pro	D-Val	Leu	NPA		62:38
30	Val-Val-Val-D-Pro-D-Val	Asp	Pro	D-Val	Val	Gly		60:40
31	Val-Val-Val-Pro-D-Val	Asp	Pro	D-Val	Val	Gly		55:45

Table S1. Peptide primary structure/enantioselectivity studies for a model indole oxidation reaction. Abbreviations: Dhl = *L*-4,5-di-dehydroleucine, Tba = L-β-*tert*-butylalanine, Cha = L-β-cyclohexylalanine, Cpa = L-β-cyclopropylalanine, Chg = L-cyclohexylglycine, MEA = methylamine, PEA = 1-phenylethylamine, 2-NEA = 1-(2-naphthyl)ethylamine, 1-NEA = 1-(1-naphthyl)ethylamine, Pip = L-pipecolic acid, Hyp = (2*S*,4*R*)-4-hydroxyproline,Aib = 2-aminoisobutyric acid,DPMA = 1,1-diphenylmethylamine, Abi = abietic acid imide, NBA = *n*-butylamine, NPA = *n*-propylamine.

D. Peptide Sequence Optimization for Enantioselective Indole Oxidation.

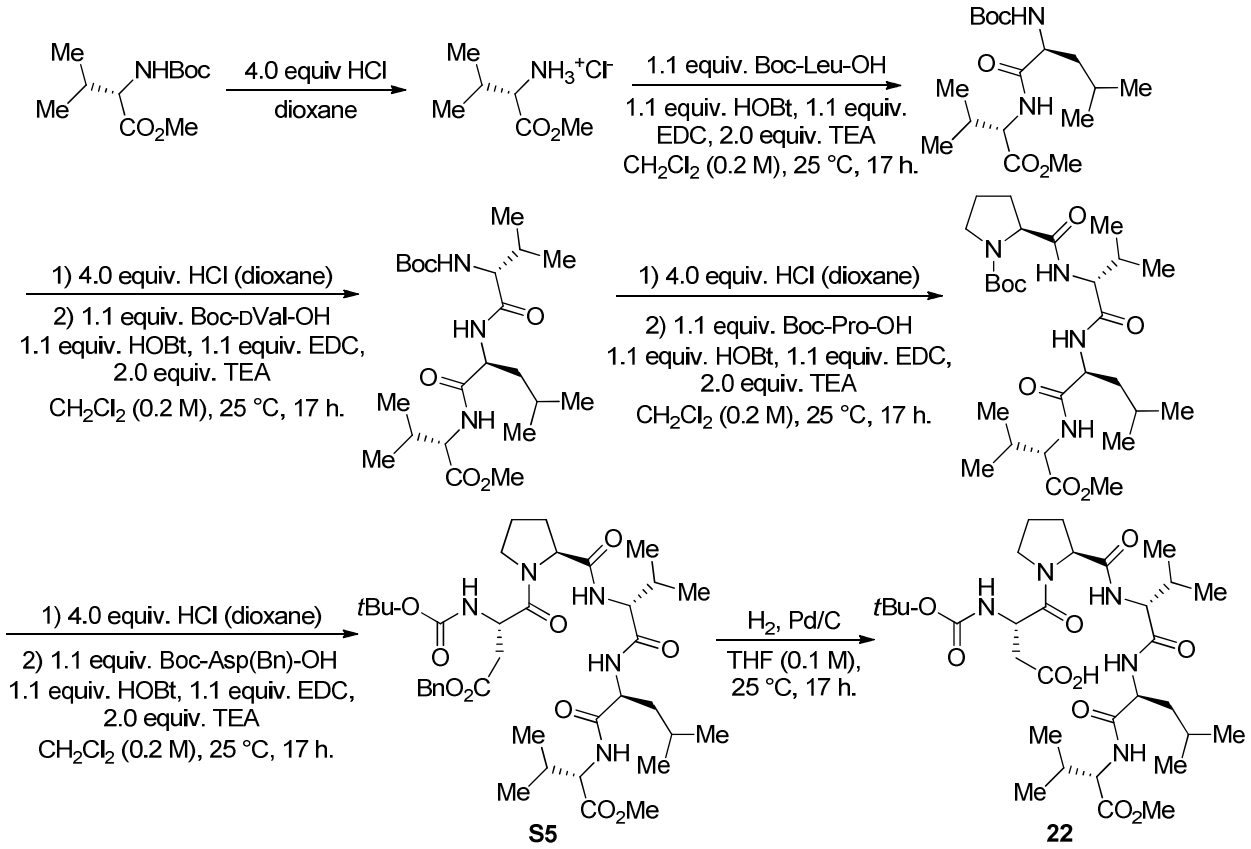


40b **41b**

			Residue Position			
Entry	*i* = catalytic	*i*+1	*i*+2	*i*+3	*i*+4	*e.r.*
1	Asp	Pro	D-Val	Leu	Gly	79:21
2	Glu	Pro	D-Val	Leu	Gly	60:40
3	Asp	*t*Bu-Hyp	D-Val	Leu	Gly	77:23
4	Asp	Pip	D-Val	Leu	Gly	75:25
5	Asp	Leu	D-Val	Leu	Gly	74:26
6	Asp	Pro	D-*tert*-Leu	Leu	Gly	79:21
7	Asp	Pro	D-Val	Cpa	Gly	75:25
8	Asp	Pro	D-Val	Tba	Gly	78:22
9	Asp	Pro	D-Val	Leu	Val	84:16
10	Asp	D-Pro	Aib	Phe	Gly	50:50
11	Asp	Pro	D-Val	Leu	Ala	82:18
12	Asp	Pro	D-Val	Leu	Phe	82:18
13	Asp	Pro	D-Val	Leu	Leu	82:18
14	Asp	Pro	D-Val	Leu	Ile	81:19
15	Asp	Pro	D-Val	Leu	Pro	76:24
16	Asp	Pro	D-Val	Leu	D-Val	64:36

Table S2. Optimization of the peptide primary structure forenantioselectivityin the oxidation of the model bis(indole)**40b**. Currently optimized sequence (peptide catalyst **22**) depicted in entry 9. Abbreviations: Hyp = (2*S*,4*R*)-4-hydroxyproline, Pip = L-pipecolic acid, Cpa = L-β-cyclopropylalanine, Tba = L-β-*tert*-butylalanine, Aib = 2-aminoisobutyric acid.

E. Preparation of Peptides 22 and *ent*-22.



Boc-Val-OMe (1.38 g, 5.96 mmol, 1.0 equiv.) was placed in a flask equipped with magnetic stir bar, flushed with nitrogen, and treated with 4 M solution of HCl in dioxane (6.0 mL). The dissolution of the material was accompanied by intense gas evolution, so in order to avoid pressure build-up inside the flask, the septum was pierced with a needle. After 0.5 h starting material could not be detected by TLC, and the reaction mixture was concentrated under reduced pressure in a rotary evaporator (caution: HCl gas released).Solvent and any residual HCl were removed by exposing the product to high vacuum for a minimum of 1 h.To the resulting white powder were added HOBt·H_2O (1.01 g, 6.56 mmol, 1.1 equiv.), EDC·HCl (1.26 g, 6.56 mmol, 1.1 equiv.), and Boc-Leu-OH·H_2O (1.64 g, 6.56 mmol, 1.1 equiv.), the mixture was suspended in dry DCM (30 mL, 0.2 M), and triethylamine (1.66 mL, 11.93 mmol, 2.0 equiv.) was added in one portion.The resulting clear, colorless solution was stirred at room temperature for 17 h. The reaction mixture was then diluted with 350 mL of EtOAc, transferred to a separatory funnel and sequentially washed with 150 mL of 0.5 M aqueous solution of citric acid and 150 mL of saturated aqueous solution of $NaHCO_3$. The organic phase was dried over $MgSO_4$, filtered, and concentrated under reduced pressure. This product was exposed to high vacuum for 1 hour, and then subjected to the removal of the Boc group in a manner described above. To the white powder obtained after extensive drying of the crude deprotection product at high vacuum were added HOBt·H_2O (1.01 g, 6.56 mmol, 1.1 equiv.), EDC·HCl (1.26 g, 6.56 mmol, 1.1 equiv.), and Boc-D-Val-OH (1.43 g, 6.56 mmol, 1.1 equiv.), the mixture was suspended in dry DCM (30 mL, 0.2 M), and triethylamine (1.66 mL, 11.93 mmol, 2.0 equiv.) was added in one portion. Subsequent coupling, work-up, and Boc removal were carried out in a manner analogous to the one described above. To the dry deprotection product thus obtained were added

HOBt·H_2O (1.01 g, 6.56 mmol, 1.1 equiv.), EDC·HCl (1.26 g, 6.56 mmol, 1.1 equiv.), and Boc-Pro-OH (1.41 g, 6.56 mmol, 1.1 equiv.), the mixture was suspended in dry DCM (30 mL, 0.2 M), and triethylamine (1.66 mL, 11.93 mmol, 2.0 equiv.) was added in one portion. After carrying out the peptide coupling, work-up, and Boc removal proceduresas described above, final peptide coupling step was performed using following amount of materials: HOBt·H_2O (1.01 g, 6.56 mmol, 1.1 equiv.), EDC·HCl (1.26 g, 6.56 mmol, 1.1 equiv.), and Boc-Asp(Bn)-OH (2.12 g, 6.56 mmol, 1.1 equiv.), dry DCM (30 mL, 0.2 M), and triethylamine (1.66 mL, 11.93 mmol, 2.0 equiv.). Crude product obtained after the final peptide coupling was purified by flash column chromatography on normal phase silica gel (30% → 50% acetone/hexanes) to yield 2.30 g (52% yield) of benzyl ester **S5** as a white solid. This compound was dissolved in THF (30 mL, 0.1 M), and the atmosphere of the flask was flushed with dry N_2 for 5 minutes. Then Pd on charcoal (10wt%, 500 mg, 0.470 mmol, 0.15 equiv.) was added,and a balloon filled with H_2 was placed on the reaction flask. The resulting suspension was stirred at room temperature for 17 hours. At this point, no starting material could be detected by thin layer chromatography.The suspension was diluted with 250 mL of EtOAc and filtered through Celite. The filtrate was concentrated under reduced pressure to afford 1.82 g of the free acid **22** as a white foam (47% yield). This material was purified via the following sequence: 1) Flash purification onan automated system using a reverse phase (C18) column, eluting with the gradient 0% → 99% MeOH/H_2O over 25 CV, then 100% MeOH for 2 CV; 30 mL/min, detection at 210 nm, 262 nm. 2) Flash chromatography on normal phase silica gel eluting with the gradient 95:5:1 → 85:15:1 DCM:MeOH:AcOH, followed by several cycles of azeotropic removal of acetic acid with toluene under reduced pressure on a rotary evaporator, and the final drying of the product under high vacuum for a minimum of 72 h. White powder (1.61 g, 41% yield).

^{1}H NMR (500 MHz, $CDCl_3$, *J* in Hz) δ 7.61 (d, *J* = 8.5, 1H), 7.56 (d, *J* = 7.9, 1H), 7.50 (d, *J* = 7.4, 1H), 5.66 (d, *J* = 9.5, 1H), 4.86 (td, *J* = 11.1, 4.1, 1H), 4.77 (d, *J* = 7.2, 1H), 4.60 – 4.54 (m, 1H), 4.41 (dd, *J* = 8.5, 5.4, 1H), 4.27 (dd, *J* = 7.4, 3.5, 1H), 3.96 (t, *J* = 8.9, 1H), 3.88 (dd, *J* = 17.3, 9.1, 1H), 3.69 (s, 3H), 3.08 (dd, *J* = 17.5, 11.5, 1H), 2.77 (dd, *J* = 17.5, 4.3, 1H), 2.60 (dd, *J* = 11.2, 5.1, 1H), 2.34 – 2.23 (m, 1H), 2.10 (dq, *J* = 13.4, 6.8, 1H), 2.05 – 1.96 (m, 1H), 1.93 – 1.82 (m, 1H), 1.82 – 1.71 (m, 2H), 1.58 – 1.42 (m, 3H), 1.39 (s, 9H), 0.93 (d, *J* = 6.9, 3H), 0.89 – 0.80 (m, 12H), 0.69 (d, *J* = 6.9, 3H);**^{13}C NMR** (126 MHz, $CDCl_3$) δ 175.0, 174.8, 173.1, 172.2, 171.8, 171.5, 155.1, 80.4, 60.2, 59.7, 57.9, 52.3, 52.1, 47.8, 39.5, 37.6, 31.0, 30.7, 28.4, 26.8, 24.8, 24.2, 22.9, 21.9, 19.8, 19.0, 17.9, 16.4; **FTIR** (cm^{-1}) 3306, 2962, 1711, 1690, 1634, 1516, 1436, 1294, 1212, 1160, 1004; **UPLC-MS** (ESI) *m/z* 656.44 (calculated for $C_{31}H_{53}N_5O_{10}$ [M + H^+] = 656.38); **TLC** R_f = 0.64 (85:15:1DCM:MeOH:AcOH). **$[\alpha]_D^{20\,℃}$** = -159.7 (c = 5.225, $CHCl_3$).

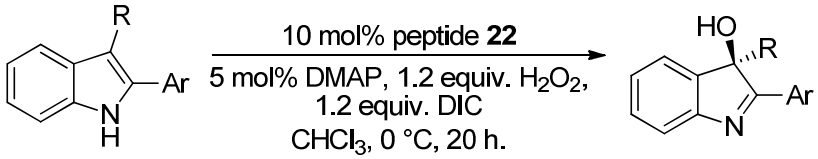
The enantiomer of the catalyst, *ent*-**22**, was prepared in an analogous fashion, using following monomers and reagents:

Boc-D-Val-OMe (1.62 g, 7.00 mmol, 1.0 equiv.), Boc-D-Leu-OH·H_2O (1.92 g, 7.7 mmol, 1.1 equiv.), Boc-Val-OH (1.67g, 7.7 mmol, 1.1 equiv.), Boc-D-Pro-OH (1.66 g, 7.7 mmol, 1.1 equiv.), Boc-D-Asp(Bn)-OH (2.49 g, 7.7 mmol, 1.1 equiv.), HOBt·H_2O (1.18 g, 7.7 mmol, 1.1 equiv.), EDC·HCl (1.48 g, 7.7 mmol, 1.1 equiv.), TEA (1.95 mL, 14.0 mmol, 2.0 equiv.). White powder (2.11 g, 46%).

^{1}H NMR (500 MHz, $CDCl_3$, *J* in Hz) δ 7.61 (d, *J* = 8.5, 1H), 7.56 (d, *J* = 7.9, 1H), 7.50 (d, *J* = 7.4, 1H), 5.66 (d, *J* = 9.5, 1H), 4.86 (td, *J* = 11.1, 4.1, 1H), 4.77 (d, *J* = 7.2, 1H), 4.60 – 4.54 (m, 1H), 4.41 (dd, *J* = 8.5, 5.4, 1H), 4.27 (dd, *J* = 7.4, 3.5, 1H), 3.96 (t, *J* = 8.9, 1H), 3.88 (dd, *J* = 17.3, 9.1, 1H), 3.69 (s, 3H),

3.08 (dd, *J* = 17.5, 11.5, 1H), 2.77 (dd, *J* = 17.5, 4.3, 1H), 2.60 (dd, *J* = 11.2, 5.1, 1H), 2.34 – 2.23 (m, 1H), 2.10 (dq, *J* = 13.4, 6.8, 1H), 2.05 – 1.96 (m, 1H), 1.93 – 1.82 (m, 1H), 1.82 – 1.71 (m, 2H), 1.58 – 1.42 (m, 3H), 1.39 (s, 9H), 0.93 (d, *J* = 6.9, 3H), 0.89 – 0.80 (m, 12H), 0.69 (d, *J* = 6.9, 3H);**^{13}C NMR** (126 MHz, $CDCl_3$) δ 175.0, 174.7, 173.1, 172.2, 171.8, 171.5, 155.1, 80.3, 60.2, 59.7, 57.9, 52.3, 52.1, 47.8, 39.5, 37.5, 31.0, 30.7, 28.4, 26.8, 24.8, 24.2, 22.9, 21.9, 19.7, 18.9, 17.9, 16.5; **FTIR** (cm^{-1}) 3306, 2963, 1634, 1516, 1437, 1367, 1293, 1249, 1211, 1159; **UPLC-MS** (ESI) *m/z* 656.47 (calculated for $C_{31}H_{53}N_5O_{10}$ [M + H^+] = 656.38);**TLC** R_f =0.64 (85:15:1DCM:MeOH:AcOH). $[\alpha]_D^{20\,℃}$ = +147.5 (c = 4.407, $CHCl_3$).

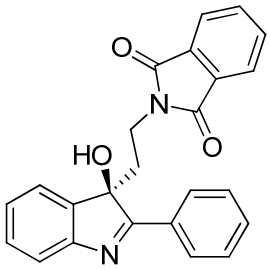
F. General Procedure for the Peptide Catalyzed Oxidation of 2,3-Disubstituted Indoles.



The oxidation of indoles was carried out in ¼-dram or 2-dram glass vials mounted to a cryogenic cooler with temperature control of ± 0.1 °C. Reactions were set up on the benchtop without exclusion of air or moisture. In a typical experiment, indole (0.1 mmol) was dissolved in 890 μL of chloroform (for deviations from the standard concentration see section G) and peptide **22** was added as a chloroform solution (typically 100 μL of 0.1 M solution, 0.01 mmol). To this solution was added a chloroform solution of *N*,*N*-dimethyl-4-aminopyridine (typically 10 μL of 0.5 M solution, 5 μmol), followed by 12.2 μL of aqueous 30% hydrogen peroxide (0.12 mmol). The biphasic mixture was stirred at 0 °C for 5 minutes, and the reaction was initiated by the addition 18.6 μL of diisopropylcarbodiimide (0.12 mmol). After 20 hours, the reaction mixture was diluted with 2.0 mL of chloroform and directly purified by flash chromatography or preparative TLC.

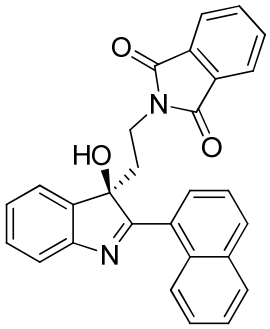
Racemic standards for the indole oxidation products were prepared by reacting the indole with 4.0 equiv. of peracetic acid (CH_3CO_3H) in chloroform (typically 0.1 M) at room temperature for 6-10 h. Standards were purified without previous workup in a manner analogous to that for the peptide catalyzed reactions.

G. Analytical Data for Indole Oxidation Products.



17

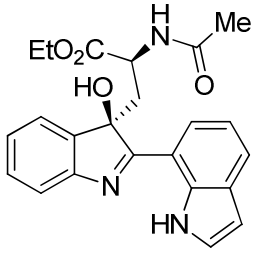
(*R*)-3-Hydroxy-3-(2-*N*,*N*-phthaloylaminoethyl)-2-phenyl-3*H*-indole (**17**). Purified by preparative TLC (50% EtOAc/Hex). Compound isolated as a mixture of conformers. White foam, 9.6 mg, 59% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 8.27 – 8.15 (m, 2H), 7.72 – 7.65 (m, 2H), 7.65 – 7.58 (m, 2H), 7.52 (d, *J* = 6.6, 1H), 7.48 (d, *J* = 7.6, 1H), 7.41 (dd, *J* = 13.2, 5.9, 1H), 7.36 (dd, *J* = 13.8, 6.2, 2H), 7.32 – 7.27 (m, 1H), 7.19 (t, *J* = 7.4, 1H), 3.58 – 3.48 (m, 1H), 3.44 – 3.35 (m, 1H), 2.82 – 2.62 (m, 2H), 2.52 – 2.43 (m, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 179.0, 167.9, 153.05, 140.2, 133.9, 132.1, 131.8, 131.6, 130.4, 128.7, 128.6, 126.8, 123.2, 122.6, 121.6, 86.0, 36.1, 33.3; **FTIR** (cm^{-1}) 3469, 1772, 1704, 1536, 1444, 1396, 1369, 1265, 1026; **UPLC-MS** (ESI) *m/z* 383.19 (calculated for $C_{24}H_{18}N_2O_3$ [M + H^+] = 383.13); **TLC** R_f = 0.57 (50% EtOAc/Hex); **Chiral HPLC analysis**: 77:23 er, performed on Chiralcel OD-H (Daicel, 4.6 mm × 250 mm, 5 μm, 20 °C), eluting at 0.20 mL/min with 80:20 hexanes/ethanol. Retention times: t_{minor} = 35.0 min, t_{major} = 37.4 min; $[\alpha]_{546\ nm}^{20\,℃}$ = -17.1 (*c* = 0.164, $CHCl_3$).



20

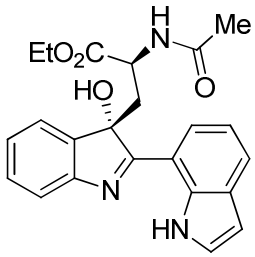
(*R*)-3-Hydroxy-3-(2-*N*,*N*-phthaloylaminoethyl)-2-(1-naphthyl)-3*H*-indole (**20**). Purified by preparative TLC (40% EtOAc/Hex). White solid, 14.9 mg, 57% yield. Product was recrystallized by slow evaporation from 95:5 2-propanol/ethanol mixture; white crystals, 11.9 mg, 45% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 9.08 (d, *J* = 8.6, 1H), 8.53 (dd, *J* = 7.3, 1.1, 1H), 7.93 (d, *J* = 8.2, 1H), 7.85 (d, *J* = 7.8, 1H), 7.66 – 7.62 (m, 3H), 7.61 – 7.56 (m, 3H), 7.56 – 7.48 (m, 3H), 7.33 (td, *J* = 7.6, 1.1, 1H), 7.18 (td, *J* = 7.5, 0.9, 1H), 3.71 – 3.63 (m, 1H), 3.56 – 3.46 (m, 1H), 2.77 (s, 1H), 2.67 – 2.58 (m, 1H), 2.34 (ddd, *J* = 13.8, 7.8, 5.9, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 179.6, 168.0, 153.6, 139.1, 134.4, 133.9, 132.0, 131.7, 131.7, 130.4, 129.3, 128.7, 128.3, 127.8, 126.9, 126.8, 126.3, 124.9, 123.2, 122.7, 122.0, 87.5, 35.6, 33.8; **FTIR** (cm^{-1}) 3352, 2943, 1764, 1698, 1567, 1507, 1401, 1339, 1273, 1033; **UPLC-MS** (ESI) *m/z* 433.20 (calculated for $C_{28}H_{20}N_2O_3$[M + H^+] = 433.15); **TLC** R_f = 0.45 (40% EtOAc/Hex); **Chiral HPLC analysis**: 94:6er (>98:2 er after recrystallization), performed on Chiralcel OD (Daicel, 4.6 mm × 250 mm,

10 μm, 20 °C), eluting at 0.50 mL/min with 90:10 hexanes/ethanol. Retention times: t_{major} = 148.0 min, t_{minor} = 222.2 min; $[\alpha]_{546\ nm}^{20\ ℃}$ =-174.8 (*c* = 0.078, $CHCl_3$).



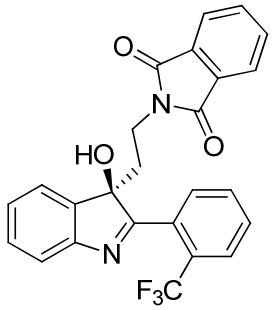
26

(**2'*S*,3*R*)-3-Hydroxy-3-(2-*N*-acetylamino-2-ethoxycarbonylethyl)-2-(7-indolyl)-3*H*-indole** (**26**). Purified by column chromatography on silica gel (60% EtOAc/Hex). Yellow foam, 586.0 mg, 73% yield. **^{1}H NMR** (400 MHz, $CDCl_3$, *J* in Hz) δ 11.12 (s, 1H), 8.16 (d, *J* = 7.5, 1H), 7.85 (d, *J* = 7.8, 1H), 7.63 (d, *J* = 7.6, 1H), 7.53 (d, *J* = 7.3, 1H), 7.46 (td, *J* = 7.6, 1.1 Hz, 1H), 7.41 – 7.37 (m, 1H), 7.31 (t, *J* = 7.4, 1H), 7.24 (t, *J* = 7.7, 1H), 6.65 – 6.61 (m, 1H), 5.02 (d, *J* = 8.5, 1H), 4.07 (td, *J* = 9.0, 3.5, 1H), 3.96 (dq, *J* = 10.8, 7.1, 1H), 3.83 (dq, *J* = 10.7, 7.1, 1H), 3.03 (dd, *J* = 14.2, 3.5, 1H), 2.72 – 2.64 (m, 2H), 1.17 – 1.08 (m, 6H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 179.1, 171.6, 169.6, 153.6, 139.1, 134.7, 130.7, 129.1, 126.6, 125.4, 125.3, 123.5, 122.6, 121.2, 119.5, 115.4, 102.9, 86.1, 61.7, 48.8, 41.7, 22.0, 14.0; **FTIR** (cm^{-1}) 3380, 2984, 1731, 1657, 1522, 1433, 1372, 1335, 1266, 1212,1059; **UPLC-MS** (ESI) *m/z* 406.23 (calculated for $C_{23}H_{23}N_3O_4$ [M + H^+] = 406.17); **TLC** R_f =0.35 (70% EtOAc/Hex). $[\alpha]_{546\ nm}^{20\ ℃}$ = +89.5 (*c* = 0.140, $CHCl_3$).



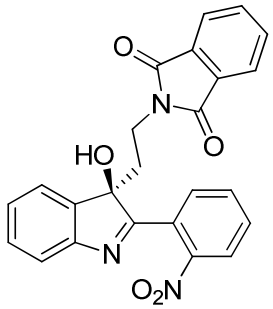
27

(**2'*S*,3*S*)-3-Hydroxy-3-(2-*N*-acetylamino-2-ethoxycarbonylethyl)-2-(7-indolyl)-3*H*-indole** (**27**). Purified by column chromatography on silica gel (60% EtOAc/Hex). White foam, 80.3 mg, 10% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 11.17 (s, 1H), 8.19 (d, *J* = 7.6, 1H), 7.85 (d, *J* = 7.8, 1H), 7.63 (d, *J* = 7.6, 1H), 7.57 (d, *J* = 7.3, 1H), 7.45 (t, *J* = 8.0, 1H), 7.42 – 7.39 (m, 1H), 7.28 (t, *J* = 7.4, 1H), 7.24 (t, *J* = 7.7, 1H), 6.67 – 6.62 (m, 1H), 6.33 (d, *J* = 6.3, 1H), 4.63 – 4.52 (m, 1H), 3.97 – 3.90 (m, 1H), 3.86 – 3.77 (m, 1H), 3.37 (s, 1H), 2.86 (dd, *J* =14.8, 8.9, 1H), 2.46 (dd, *J* = 14.7, 3.5, 1H), 1.69 (s, 3H), 1.09 (t, *J* = 7.1, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 179.9, 171.4, 170.3, 153.6, 139.2, 135.1, 130.6, 129.1, 126.3, 125.5, 125.3, 124.3, 123.4, 121.3, 119.5, 114.8, 102.9, 86.7, 61.8, 50.4, 41.1, 22.8, 14.0; **FTIR** (cm^{-1}) 3383, 3351, 2974, 2924, 2851, 1745, 1641, 1524, 1451, 1267, 1208, 1061; **UPLC-MS** (ESI) *m/z* 406.22 (calculated for $C_{23}H_{23}N_3O_4$[M + H^+] = 406.17); **TLC** R_f=0.25 (70% EtOAc/Hex); $[\alpha]_{546\ nm}^{20\ ℃}$ = -51.4 (*c* = 0.006, $CHCl_3$).



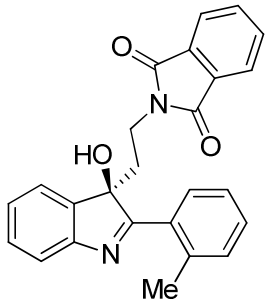
37a

(*R*)-3-Hydroxy-3-(2-*N*,*N*-phthaloylaminoethyl)-2-(2-trifluoromethylphenyl)-3*H*-indole (**37a**). The concentration of the indole in this reaction was set to 0.5 M. Purified by column chromatography on silica gel (35% EtOAc/Hex). White foam, 83.4 mg, 93% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 8.06 (d, *J* = 7.6, 1H), 7.83 (d, *J* = 7.8, 1H), 7.81 – 7.76 (m, 2H), 7.72 – 7.67 (m, 2H), 7.66 – 7.61 (m, 2H), 7.59 (d, *J* = 7.6, 2H), 7.42 (td, *J* = 7.6, 1.2, 1H), 7.32 (td, *J* = 7.5, 1.0, 1H), 3.82 – 3.69 (m, 2H), 2.90 (s, 1H), 2.29 (ddd, *J* = 13.9, 9.5, 6.6, 1H), 2.16 – 2.06 (m, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 179.4, 168.3, 153.0, 139.2, 134.2, 132.3 (q, *J* = 1.7), 132.2, 131.8, 130.5, 130.0, 130.0, 129.4 (q, *J* = 30.8), 127.5, 124.1 (q, *J* = 273.7), 123.5, 123.2, 122.3, 87.0, 34.5, 33.2; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -57.0; **FTIR** (cm^{-1}) 3470, 2924, 1772, 1704, 1579, 1522, 1446, 1398, 1369, 1309, 1130, 1113, 1034; **UPLC-MS** (ESI) *m/z*451.14 (calculated for $C_{25}H_{17}F_3N_2O_3$[M + H^+] = 451.12); **TLC** R_f =0.45 (40% EtOAc/Hex); **Chiral HPLC analysis**: 94:6 er, performed on Chiralpak IA (Daicel, 4.6 mm × 250 mm, 5 μm, 20 °C), eluting at 0.50 mL/min with 80:20 hexanes/isopropanol. Retention times: t_{minor} = 22.6 min, t_{major} = 33.3 min; $[\alpha]_{546\ nm}^{20\,℃}$= -14.5 (*c* = 3.950, $CHCl_3$).



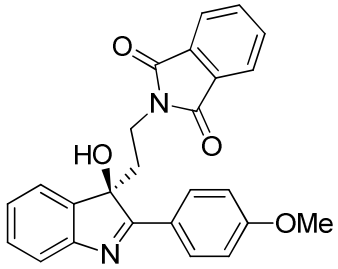
37b

(*R*)-3-Hydroxy-3-(2-*N*,*N*-phthaloylaminoethyl)-2-(2-nitrophenyl)-3*H*-indole (**37b**). The concentration of the indole in this reaction was set to 0.5 M. Purified by preparative TLC (15% EtOAc/DCM). Yellow foam, 29.2 mg, 78% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 8.07 (d, *J* = 8.2, 1H), 8.01 (d, *J* = 7.5, 1H), 7.83 – 7.75 (m, 3H), 7.72 – 7.68 (m, 2H), 7.67 – 7.62 (m, 2H), 7.55 (d, *J* = 7.6, 1H), 7.41 (t, *J* = 7.6, 1H), 7.33 (t, *J* = 7.4, 1H), 3.69 – 3.58 (m, 2H), 2.93 (s, 1H), 2.45 – 2.36 (m, 1H), 2.18 – 2.08 (m, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 179.3, 168.2, 152.8, 149.1, 139.2, 134.2, 133.6, 132.2, 130.9, 130.5, 130.5, 129.0, 127.7, 125.1, 123.4, 123.2, 122.2, 87.6, 34.7, 33.1; **FTIR** (cm^{-1}) 3453, 3058, 1772, 1704, 1614, 1529, 1445, 1398, 1368, 1265, 1188; **UPLC-MS** (ESI) *m/z* 428.13 (calculated for $C_{24}H_{17}N_3O_5$[M + H^+] = 428.12); **TLC** R_f =0.45 (15% EtOAc/DCM); **Chiral HPLC analysis**: 89:11 er, performed on Chiralcel OD-H (Daicel, 4.6 mm × 250 mm, 5 μm, 20 °C), eluting at 0.20 mL/min with 80:20 hexanes/ethanol. Retention times: t_{minor} = 54.9 min, t_{major} = 65.0 min; $[\alpha]_D^{20\,℃}$= +149.0 (*c* = 0.603, $CHCl_3$).



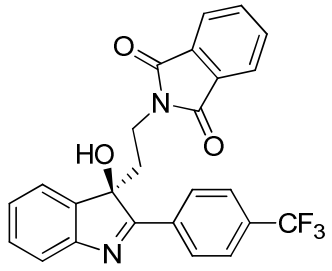
37c

(*R*)-3-Hydroxy-3-(2-*N*,*N*-phthaloylaminoethyl)-2-(2-methylphenyl)-3*H*-indole (**37c**). Purified by preparative TLC (10% EtOAc/DCM).Compound isolated as ~5:1 mixture of conformers. White foam, 11.7 mg, 76% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 8.20 (d, *J* = 7.9, 0.15H), 8.17 (d, *J* = 7.7, 0.83H), 7.74 – 7.69 (m, 2H), 7.67 – 7.62 (m, 2H), 7.56 – 7.46 (m, 2H), 7.34 – 7.28 (m, 3H), 7.26 – 7.21 (m, 1H), 7.19 – 7.12 (m, 1H), 3.62 – 3.45 (m, 2H), 2.82 (s, 1H), 2.64 (s, 3H), 2.52 (ddd, *J* = 13.9, 8.7, 7.0, 1H), 2.28 – 2.16 (m, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 180.5, 168.0, 153.4, 139.1, 139.1, 134.0, 132.1, 131.9, 131.9, 130.2, 130.2, 129.2, 126.8, 125.7, 123.3, 122.7, 121.7, 87.0, 35.2, 33.2, 22.2; **FTIR** (cm^{-1}) 3450, 3057, 2925, 1772, 1706, 1600, 1534, 1447, 1397, 1368, 1265, 1188; **UPLC-MS** (ESI) *m/z*397.17 (calculated for $C_{25}H_{20}N_2O_3$[M + H^+] = 397.15); **TLC** R_f =0.30 (10% EtOAc/DCM); **Chiral HPLC analysis**: 88:12 er, performed on Chiralcel OJ-H (Daicel, 4.6 mm × 250 mm, 5 μm, 20 °C), eluting at 0.50 mL/min with 75:25 hexanes/ethanol. Retention times: t_{minor} = 23.3 min, t_{major} = 68.7 min; $[\alpha]_{546\ nm}^{20\ ℃}$= -14.7 (*c* = 0.244, $CHCl_3$).



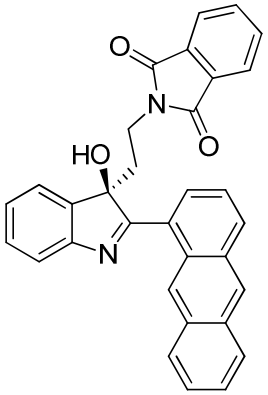
37e

(*R*)-3-Hydroxy-3-(2-*N*,*N*-phthaloylaminoethyl)-2-(4-methoxyphenyl)-3*H*-indole (**37e**). Purified by preparative TLC (50% EtOAc/Hex).Compound isolated as ~2:1 mixture of conformers. Yellow foam, 9.0 mg, 64% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 8.20 (d, *J* = 8.8, 0.67 H), 8.17 (d, *J* = 8.8, 1.29H), 7.72 – 7.66 (m, 2H), 7.66 – 7.59 (m, 2H), 7.51 (dd, *J* = 7.1, 3.4, 1H), 7.47 (d, *J* = 7.4, 0.31H), 7.42 (d, *J* = 7.6, 0.64H), 7.30 – 7.25 (m, 1H), 7.20 – 7.15 (m, 1H), 6.87 (d, *J* = 9.0, 0.58H), 6.84 (d, *J* = 9.0, 1.20H), 3.81 (s, 3H), 3.56 – 3.47 (m, 1H), 3.43 – 3.35 (m, 1H), 2.82 – 2.65 (m, 2H), 2.53 – 2.44 (m, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 178.6, 167.8, 162.1, 152.8, 139.9, 133.8, 132.0, 130.5, 130.1, 126.0, 124.4, 123.1, 122.5, 120.8, 113.9, 85.4, 55.4, 36.3, 33.2; **FTIR** (cm^{-1}) 3464, 2961, 1772, 1705, 1603, 1508, 1397, 1253, 1173, 1024; **UPLC-MS** (ESI) *m/z*413.20 (calculated for $C_{25}H_{20}N_2O_4$[M + H^+] = 413.14); **TLC** R_f =0.34 (50% EtOAc/Hex); **Chiral HPLC analysis**: 75:25 er, performed on Chiralcel OJ-H (Daicel, 4.6 mm × 250 mm, 5 μm, 20 °C), eluting at 0.50 mL/min with 75:25 hexanes/ethanol. Retention times: t_{major} = 53.1 min, t_{minor} = 78.0 min; $[\alpha]_{546\ nm}^{20\ ℃}$= +104.4 (*c* = 0.440, *N*,*N*-DMF).



37f

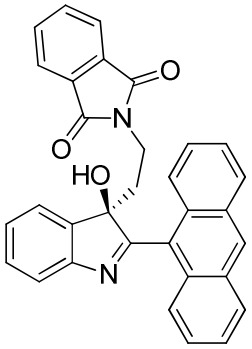
(*R*)-3-Hydroxy-3-(2-*N*,*N*-phthaloylaminoethyl)-2-(4-trifluoromethylphenyl)-3*H*-indole (**37f**). The concentration of the indole in this reaction was set to 0.5 M. Purified by preparative TLC (40% EtOAc/Hex).Compound isolated as ~2.5:1 mixture of conformers. White foam, 24.3 mg, 69% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 8.20 (d, *J* = 8.1, 0.57 H), 8.13 (d, *J* = 8.1, 1.36 H), 7.76 – 7.54 (m, 4H), 7.51 – 7.38 (m, 3.28H), 7.32 (d, *J* = 7.3, 0.69H), 7.26 – 7.11 (m, 2H), 3.53 – 3.38 (m, 1H), 3.35 – 2.92 (m, 2H), 2.78 – 2.62 (m, 1H), 2.58 – 2.39 (m, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$, *J* in Hz) δ 177.6, 177.5, 167.9, 167.8, 152.6, 152.5, 140.0, 140.0, 134.8, 134.7, 134.0, 132.6 (q, *J* = 32.6), 132.6 (q, *J*= 32.6), 131.9, 131.8, 130.6, 130.6, 128.7, 128.6, 127.4, 127.3, 124.0 (q, *J* = 272.0), 123.9 (q, *J* = 272.3), 123.2, 122.6, 122.0, 121.8, 85.8, 85.7, 36.0, 35.9, 33.1, 33.1; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -63.1; **FTIR** (cm^{-1}) 3463, 1773, 1707, 1617, 1539, 1396, 1322, 1123, 1067, 1017; **UPLC-MS** (ESI) *m/z*451.15 (calculated for $C_{25}H_{17}F_3N_2O_3$[M + H^+] = 451.12); **TLC** R_f =0.42 (40% EtOAc/Hex); **Chiral HPLC analysis**: 72:28 er, performed on Chiralpak IA (Daicel, 4.6 mm × 250 mm, 5 μm, 20 °C), eluting at 0.50 mL/min with 80:20 hexanes/isopropanol. Retention times: t_{major} = 20.2 min, t_{minor} = 25.6 min; $[\alpha]_{546\ nm}^{20\ ℃}$= -18.0 (*c* = 0.050, $CHCl_3$).



39a

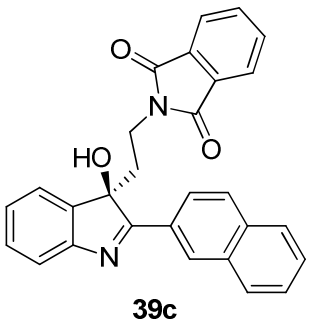
(*R*)-2-(1-Anthracenyl)-3-hydroxy-3-(2-*N*,*N*-phthaloylaminoethyl)-3*H*-indole (**39a**). Purified by column chromatography on silica gel (35% EtOAc/Hex). Yellow solid, 89.9 mg, 88% yield. Product was recrystallized by slow evaporation from DCM; yellow crystals, 72.7 mg, 71% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 9.82 (s, 1H), 8.56 (d, *J* = 7.1, 1H), 8.34 (s, 1H), 8.13 – 8.07 (m, 1H), 8.03 (d, *J* = 8.6, 1H), 7.98 – 7.93 (m, 1H), 7.70 (d, *J* = 7.6, 1H), 7.54 (d, *J* = 7.2, 1H), 7.51 – 7.45 (m, 4H), 7.43 (dd, *J* = 8.4, 7.2, 1H), 7.39 (dd, *J* = 5.5, 3.0, 2H), 7.36 (dd, *J* = 7.6, 1.2, 1H), 7.21 (t, *J* = 7.0, 1H), 3.62 (dt, *J* = 14.0, 6.9, 1H), 3.47 (dt, *J* = 14.0, 7.0, 1H), 2.90 (s, 1H), 2.66 (dt, *J* = 14.0, 6.9, 1H), 2.53 (dt, *J* = 14.0, 7.0, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 179.3, 167.9, 153.7, 139.1, 133.6, 132.9, 132.4, 132.4, 131.8,

131.6, 130.4, 129.8, 129.2, 129.2, 128.8, 127.8, 127.1, 126.9, 126.7, 126.1, 125.7, 124.2, 122.8, 122.6, 122.1, 87.7, 35.9, 33.5; **FTIR** (cm^{-1}) 3424, 3046, 2944, 1762, 1696, 1616, 1517, 1395, 1348, 1198, 1019; **UPLC-MS** (ESI) *m/z*483.16 (calculated for $C_{32}H_{22}N_2O_3$[M + H^+] = 483.16); **TLC** R_f =0.43 (40% EtOAc/Hex); **Chiral HPLC analysis**: 95:5 er (>99:1erafter recrystallization), performed on Chiralcel OD-H (Daicel, 4.6 mm × 250 mm, 5 μm, 20 °C), eluting at 0.50 mL/min with 85:15 hexanes/ethanol. Retention times: t_{minor} = 27.8 min, t_{major} = 46.7 min; $[\alpha]_{546\ nm}^{20\ ℃}$= -18.4 (*c* = 3.380, *N*,*N*-DMF) (recrystallized material).



39b

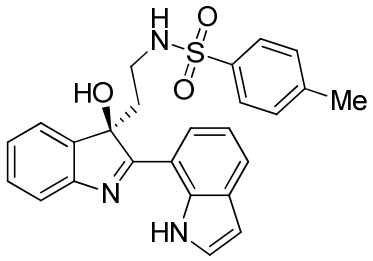
(*R*)-2-(9-Anthracenyl)-3-hydroxy-3-(2-*N*,*N*-phthaloylaminoethyl)-3*H*-indole (**39b**). Purified by preparative TLC (40% EtOAc/Hex). White solid, 1.2 mg, 7% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 8.50 (s, 1H), 8.07 – 8.98 (m, 3H), 7.97 – 7.85 (m, 1H), 7.75 (dd, *J* = 11.2, 7.5, 2H), 7.68 – 7.59 (m, 4H), 7.54 (td, *J* = 7.7, 1.1, 1H), 7.51 – 7.37 (m, 5H), 4.00 – 3.91 (m, 1H), 3.83 – 3.76 (m, 1H), 2.22 – 2.13 (m, 1H), 2.03 – 1.94 (m, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 183.7, 168.3, 153.2, 139.7, 134.1, 133.9, 132.0, 131.5, 130.4, 129.1, 128.5, 127.8, 127.3, 123.7, 123.5, 122.2, 88.7, 35.8, 33.1; **FTIR** (cm^{-1}) 3451, 3051, 2923, 2852, 1770, 1700, 1574, 1520, 1444, 1397, 1366, 1264, 1088; **UPLC-MS** (ESI) *m/z* 483.17 (calculated for $C_{32}H_{22}N_2O_3$[M + H^+] = 483.16); **TLC** R_f =0.40 (7% EtOAc/DCM); **Chiral HPLC analysis**: 84:16 er, performed on Chiralcel OD-H (Daicel, 4.6 mm × 250 mm, 5 μm, 20 °C), eluting at 0.50 mL/min with 85:15 hexanes/ethanol. Retention times: t_{minor} = 30.7 min, t_{major} = 50.2 min; $[\alpha]_{546\ nm}^{20\ ℃}$= +830.8 (*c* = 0.035, $CHCl_3$).



39c

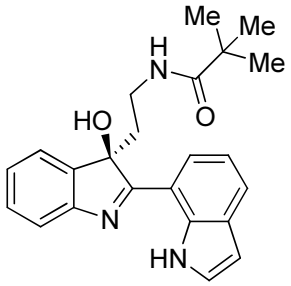
(*R*)-3-Hydroxy-2-(2-naphthyl)-3-(2-*N*,*N*-phthaloylaminoethyl)-3*H*-indole (**39c**). Purified by preparative TLC (40% EtOAc/Hex).Compound isolated as ~2:1 mixture of conformers. White solid, 14.1 mg, 74% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 8.60 (s, 0.31 H), 8.53 (s, 0.65 H), 8.14 (d, *J* = 8.6, 0.32 H), 8.08 (d, *J* = 8.6, 0.67 H), 7.78 (d, *J* = 8.0, 0.33 H), 7.70 (t, *J* = 7.0, 1H), 7.63 (d, *J* = 7.0, 1H),

7.54 (d, *J* = 8.7, 1H), 7.52 – 7.41 (m, 6H), 7.41 – 7.35 (m, 1H), 7.31 (d, *J* = 7.5, 1H), 7.21 – 7.13 (m, 1H), 7.11 (t, *J* = 7.3, 1H), 3.51 – 3.38 (m, 1H), 3.36 – 3.25 (m, 1H), 2.82 – 2.71 (m, 1H), 2.66 – 2.52 (m, 1H), 1.67 (brs, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 179.0, 178.9, 167.9, 167.8, 153.0, 152.9, 140.2, 134.7, 134.7, 133.7, 133.7, 132.9, 132.8, 131.8, 130.3, 129.5, 129.5, 129.1, 129.0, 128.3, 128.2, 127.8, 127.7, 127.6, 126.7, 126.6, 126.5, 126.4, 124.9, 124.8, 123.0, 123.0, 122.5, 121.5, 121.4, 85.9, 85.8, 36.3, 36.3, 33.3; **FTIR** (cm^{-1}) 3457, 2924, 1771, 1705, 1532, 1396, 1367, 1190, 1021; **UPLC-MS** (ESI) *m/z*433.17 (calculated for $C_{28}H_{20}N_2O_3$[M + H^+] = 433.15); **TLC** R_f =0.31 (10% EtOAc/DCM); **Chiral HPLC analysis**: 67:33 er, performed on Chiralcel OD-H (Daicel, 4.6 mm × 250 mm, 5 μm, 20 °C), eluting at 0.50 mL/min with 85:15 hexanes/ethanol. Retention times: t_{minor} = 18.7 min, t_{major} = 22.9 min; $[\alpha]_{546\ nm}^{20\ ℃}$= -24.4 (*c* = 1.000, $CHCl_3$).



41a

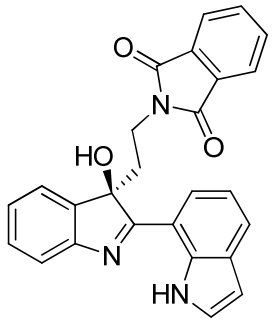
(*R*)-3-Hydroxy-3-(2-*N*-(4-toluenesulfonyl)aminoethyl)-2-(7-indolyl)-3*H*-indole (**41a**). Purified by preparative TLC (15% EtOAc/DCM). Yellow foam, 11.1 mg, 77% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ10.99 (s, 1H), 8.05 (dd, *J* = 7.5, 0.6, 1H), 7.83 (d, *J* = 7.8, 1H), 7.54 (t, *J* = 7.9, 3H), 7.39 (ddd, *J* = 7.7, 6.6, 2.2, 1H), 7.36 (dd, *J* = 3.0, 2.4, 1H), 7.21 (d, *J* = 7.9, 2H), 7.19 – 7.13 (m, 3H), 6.62 (dd, *J* = 3.1, 2.3, 1H), 4.91 – 4.84 (m, 1H), 2.93 – 2.83 (m, 2H), 2.68 (brs, 1H), 2.54 (ddd, *J* = 14.4, 8.2, 7.2, 1H), 2.43 (s, 3H), 1.89 (dt, *J* = 14.1, 5.8, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 179.6, 152.9, 143.5, 139.1, 136.7, 134.9, 130.5, 129.9, 129.1, 127.3, 126.3, 125.6, 125.3, 124.2, 122.8, 121.2, 119.4, 114.3, 102.9, 87.5, 39.2, 38.2, 21.8; **FTIR** (cm^{-1}) 3453, 3375, 3297, 3068, 2924, 1700, 1657, 1598, 1522, 1433, 1334, 1266, 1157, 1091; **UPLC-MS** (ESI) *m/z* 446.16 (calculated for [M + H^+] = 446.15); **TLC** R_f =0.30 (35% EtOAc/Hex); **Chiral HPLC analysis**: 86:14 er, performed on Chiralcel OD (Daicel, 4.6 mm × 250 mm, 10 μm, 20 °C), eluting at 0.10 mL/min with 80:20 hexanes/ethanol. Retention times: t_{minor} = 99.0 min, t_{major} = 107.8 min.



41b

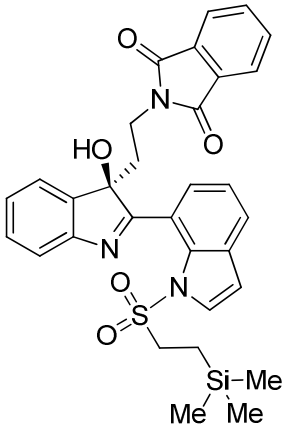
(*R*)-3-Hydroxy-2-(7-indolyl)-3-(2-*N*-pivaloylaminoethyl)-3*H*-indole (**41b**). Purified by column chromatography (35% EtOAc/Hex). White foam, 10.2 mg, 87% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 11.16 (s, 1H), 8.21 (d, *J* = 7.6, 1H), 7.85 (d, *J* = 7.8, 1H), 7.61 (d, *J* = 7.6, 1H), 7.51 (d, *J* = 7.3,

1H), 7.43 (td, *J* = 7.6, 1.2, 1H), 7.40 – 7.38 (m, 1H), 7.28 (t, *J* = 7.4, 1H), 7.23 (t, *J* = 7.7, 1H), 6.65 – 6.62 (m, 1H), 5.76 – 5.69 (m, 1H), 3.24 – 3.12 (m, 1H), 3.00 – 2.89 (m, 2H), 2.68 – 2.57 (m, 1H), 2.30 – 2.19 (m, 1H), 0.92 (s, 9H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 180.4, 178.6, 153.2, 140.1, 135.1, 130.3, 129.2, 126.6, 125.5, 125.3, 124.3, 122.9, 121.1, 119.4, 114.8, 102.9, 87.5, 39.3, 38.5, 35.5, 27.3; **FTIR** (cm^{-1}) 3377, 2964, 1634, 1520, 1335, 1265, 1201, 1051; **UPLC-MS** (ESI) *m/z*376.21 (calculated for $C_{23}H_{25}N_3O_2$[M + H^+] = 376.19); **TLC** R_f =0.39 (45% EtOAc/Hex); **Chiral HPLC analysis**: 83:17 er, performed on Chiralcel OJ-H (Daicel, 4.6 mm × 250 mm, 5 μm, 20 °C), eluting at 0.20 mL/min with 90:10 hexanes/isopropanol. Retention times:t_{major} = 128.6 min, t_{minor} = 164.5 min; $[\alpha]_{546\ nm}^{20\ ℃}$= +49.4 (*c* = 2.110, $CHCl_3$).



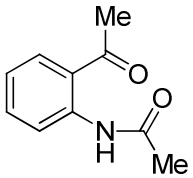
41c

(*R*)-3-Hydroxy-2-(7-indolyl)-3-(2-*N*,*N*-phthaloylaminoethyl)-3*H*-indole (**41c**). Purified by preparative TLC (40% EtOAc/Hex). Yellow solid, 5.5 mg, 89% yield. Product was recrystallized by slow evaporation from 95:5 2-propanol/ethanol mixture; yellow crystals, 2.90 mg, 47% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 11.03 (s, 1H), 8.10 (d, *J* = 7.5, 1H), 7.64 (d, *J* = 7.8, 1H), 7.59 (d, *J* = 7.6, 1H), 7.54 (d, *J* = 7.3, 1H), 7.51 – 7.46 (m, 4H), 7.36 (td, *J* = 7.7, 1.1, 1H), 7.26 – 7.24 (m, 1H), 7.21 (t, *J* = 7.4, 1H), 7.14 (td, *J* = 7.7, 1.8, 1H), 6.45 – 6.41 (m, 1H), 3.55 – 3.44 (m, 1H), 3.40 – 3.29 (m, 1H), 2.85 (t, *J* = 6.4, 2H), 2.55 (s, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 179.1, 168.0, 153.6, 139.5, 134.8, 133.6, 131.5, 130.4, 128.9, 126.5, 125.2, 125.0, 124.1, 122.8, 122.5, 121.2, 119.4, 114.8, 102.7, 86.5, 37.5, 33.4; **FTIR** (cm^{-1}) 3441, 3377, 3059, 2941, 1767, 1697, 1521, 1396, 1335, 1266, 1196, 1017; **UPLC-MS** (ESI) *m/z*422.21 (calculated for $C_{26}H_{19}N_3O_3$[M + H^+] = 422.14); **TLC** R_f =0.47 (40% EtOAc/Hex); **Chiral HPLC analysis**: 82:18er (95:5 erafter recrystallization), performed on Chiralcel OD-H (Daicel, 4.6 mm × 250 mm, 5 μm, 20 °C), eluting at 0.50 mL/min with 80:20 hexanes/ethanol. Retention times: t_{minor} = 19.2 min, t_{major} = 20.8 min; $[\alpha]_{578\ nm}^{20\ ℃}$= +136.8 (*c* = 0.100, $CHCl_3$).



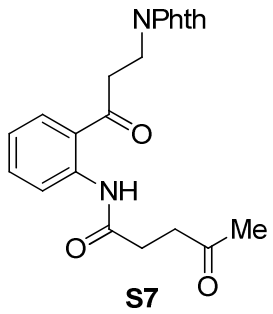
43

(*R*)-3-Hydroxy-2-(*N*-(2-trimethylsilylethanesulfonyl)indol-7-yl)-3-(2-*N*,*N*-phthaloylaminoethyl)-3*H*-indole (**43**). Purified by preparative TLC (40% EtOAc/Hex). White foam, 8.2 mg, 70% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 7.81 (dd, *J* = 5.4, 3.1, 2H), 7.75 (dd, *J* = 7.8, 1.1, 1H), 7.71 – 7.67 (m, 3H), 7.61 (d, *J* = 7.3, 1H), 7.57 (d, *J* = 3.7, 1H), 7.51 (d, *J* = 7.1, 1H), 7.48 (t, *J* = 7.7, 1H), 7.43 (td, *J* = 7.5, 1.4, 1H), 7.38 (td, *J* = 7.5, 1.1, 1H), 6.77 (d, *J* = 3.7, 1H), 4.07 (s, 1H), 3.70 – 3.54 (m, 3H), 3.49 (td, *J* = 14.0, 4.0, 1H), 2.50 (ddd, *J* = 13.0, 11.4, 5.8, 1H), 2.21 (ddd, *J* = 12.7, 10.8, 4.6, 1H), 0.87 (td, *J* = 13.8, 4.2, 1H), 0.55 (td, *J* = 13.8, 3.7, 1H), 0.02 (s, 9H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 184.06, 168.2, 152.5, 140.6, 134.1, 133.2, 132.9, 132.3, 130.0, 129.7, 127.5, 125.3, 123.6, 123.5, 123.4, 123.3, 121.2, 120.4, 108.3, 88.0, 50.6, 34.7, 33.5, 9.6, -1.7; **FTIR** (cm^{-1}) 3510, 2956, 1773, 1709, 1570, 1398, 1367, 1250, 1157, 1130, 833; **UPLC-MS** (ESI) *m/z*586.18 (calculated for [M + H^+] = 586.18); **TLC** R_f =0.40 (35% EtOAc/Hex); **Chiral HPLC analysis**: 93:7er, performed on Chiralcel OD (Daicel, 4.6 mm × 250 mm, 10 μm, 20 °C), eluting at 0.1 mL/min with 96:4 hexanes/ethanol. Retention times: t_{major} = 180.6 min, t_{minor} = 200.3min; $[\alpha]_D^{20\,℃}$= +271.1 (*c* = 0.100, $CHCl_3$).



S6

S6. Purified by preparative TLC (30% EtOAc/Hex). White solid, 25.0 mg, 45% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 11.70 (s, 1H), 8.75 (dd, *J* = 8.5, 0.9, 1H), 7.90 (dd, *J* = 8.0, 1.5, 1H), 7.62 – 7.50 (m, 1H), 7.18 – 7.06 (m, 1H), 2.68 (s, 3H), 2.24 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 203.1, 169.7, 141.3, 135.4, 131.8, 122.5, 121.9, 120.9, 28.8, 25.8; **FTIR** (cm^{-1}) 3242, 2973, 2926, 1685, 1650, 1581, 1522, 1452, 1360, 1239, 1168; **UPLC-MS** (ESI) *m/z* 178.40 (calculated for [M + H^+] =178.08); **TLC** R_f = 0.44 (30% EtOAc/Hex).

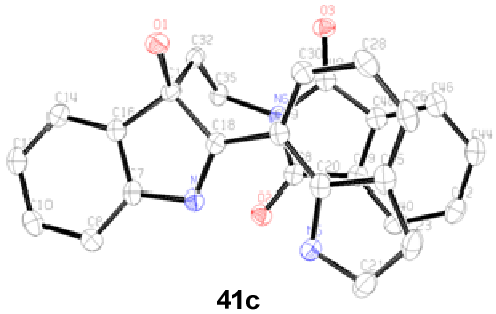


S7

S7. Purified by preparative TLC (65% EtOAc/Hex). Yellow solid, 10.0 mg, 13% yield. **^{1}H NMR** (500 MHz, $CDCl_3$, *J* in Hz) δ 11.64 (s, 1H), 8.70 (dd, *J* = 8.5, 1.0, 1H), 7.89 – 7.83 (m, 3H), 7.74 (dd, *J* = 5.5, 3.0, 2H), 7.56 – 7.50 (m, 1H), 7.12 – 7.03 (m, 1H), 4.20 – 4.09 (m, 2H), 3.54 – 3.44 (m, 2H), 2.87 (t, *J* = 6.5, 2H), 2.75 (t, *J* = 6.5, 2H), 2.24 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 207.1, 201.9, 171.4, 168.3, 141.2, 135.5, 134.3, 134.1, 132.3, 130.8, 123.6, 122.6, 121.1, 38.3, 33.8, 32.0, 30.2, 25.0; **FTIR** (cm^{-1}) 3473, 2951, 1771, 1706, 1650, 1583, 1522, 1450, 1397, 1365, 1200, 910; **UPLC-MS** (ESI) *m/z* 393.55 (calculated for $[M + H^+]$ = 393.14); **TLC** R_f = 0.62 (65% EtOAc/Hex).

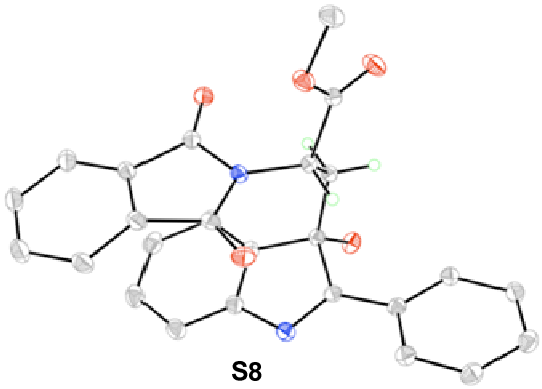
H. X-Ray Structure of the Compound 41c and Proof of Absolute Stereochemistry.

The absolute stereochemistry of the compound **41c** was established as *R* by X-ray crystallographic analysis. The details of the analysis are in section K, following the NMR spectra.



41c

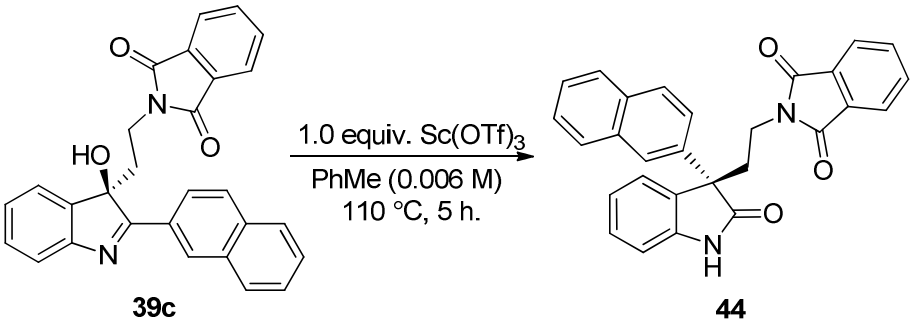
Support for the stereochemical assignment derives from the work of Movassaghi *et al*.[4], who reported a crystal structure of a related indole oxidation product **S8**. This compound is identical in spectroscopic and chromatographic properties to the *minor* diastereomer **24** obtained in the matched case of the oxidation of indole **21** using peptide catalyst **22**.



S8

I. Procedure for the Stereospecific Rearrangement of 3-Hydroxy-indolenines.

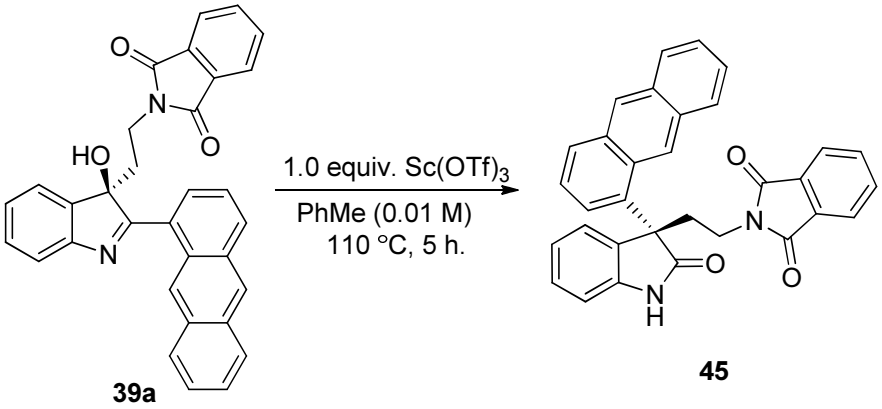
(*S*)-3-(2-*N*,*N*-phthaloylaminoethyl)-3-(2-naphthyl)oxindole (**44**)



A 5-mL round-bottom flask was charged with 3-hydroxy-indolenine **39c** (7.0 mg, 0.01619 mmol, 1.0 equiv.), scandium(III) triflate (8.0 mg, 0.01619 mmol, 1.0 equiv.) and degassed toluene (2.5 mL).The flask was flushed with nitrogen, sealed, and heated in an oil bath at 110 °C for 5 hours. The resulting yellow solution was directly purified by preparative TLC eluting with 10% EtOAc/DCM. After removal of the desorption solvent (EtOAc), the 2-oxindole **44** was isolated as white solid (5.0 mg, 71% yield).

^{1}H NMR (500 MHz, $CDCl_3$, *J* in Hz) δ 7.92 (s, 1H), 7.82 (d, *J* = 1.7, 1H), 7.79 – 7.66 (m, 5H), 7.61 (dd, *J* = 5.5, 3.1, 2H), 7.54 (dd, *J* = 8.7, 2.0, 1H), 7.45 – 7.40 (m, 2H), 7.38 (d, *J* = 7.4, 1H), 7.17 (td, *J* = 7.7, 1.2, 1H), 6.99 (td, *J* = 7.6, 1.0, 1H), 6.95 (d, *J* = 7.8, 1H), 3.88 – 3.66 (m, 2H), 2.95 (dt, *J* = 15.2, 7.6, 1H), 2.87 – 2.75 (m, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 179.6, 168.2, 140.9, 136.6, 133.9, 133.4, 132.7, 132.1, 131.9, 128.7, 128.6, 128.4, 127.6, 126.4, 126.3, 125.9, 125.6, 124.9, 123.2, 123.0, 110.5, 55.7, 34.9, 34.7; **FTIR** (cm^{-1}) 3236, 3051, 2917, 1765, 1699, 1615, 1469, 1349, 1234; **UPLC-MS** (ESI) *m/z* 433.57 (calculated for $C_{28}H_{20}N_2O_3$ [M + H^+] = 433.15); **TLC** R_f = 0.34 (10% EtOAc/DCM); **Chiral HPLC analysis**: 67:33 er, performed on Chiralcel OJ-H (Daicel, 4.6 mm × 250 mm, 5 μm, 20 °C), eluting at 0.50 mL/min with 80:20 hexanes/ethanol. Retention times: t_{minor} = 64.3 min, t_{major} = 84.8 min; $[\alpha]_{546\,nm}^{20\,℃}$ = -24.4 (*c* = 1.0, $CHCl_3$).

(*S*)-3-(2-*N*,*N*-phthaloylaminoethyl)-3-(1-anthracenyl)oxindole (**45**)



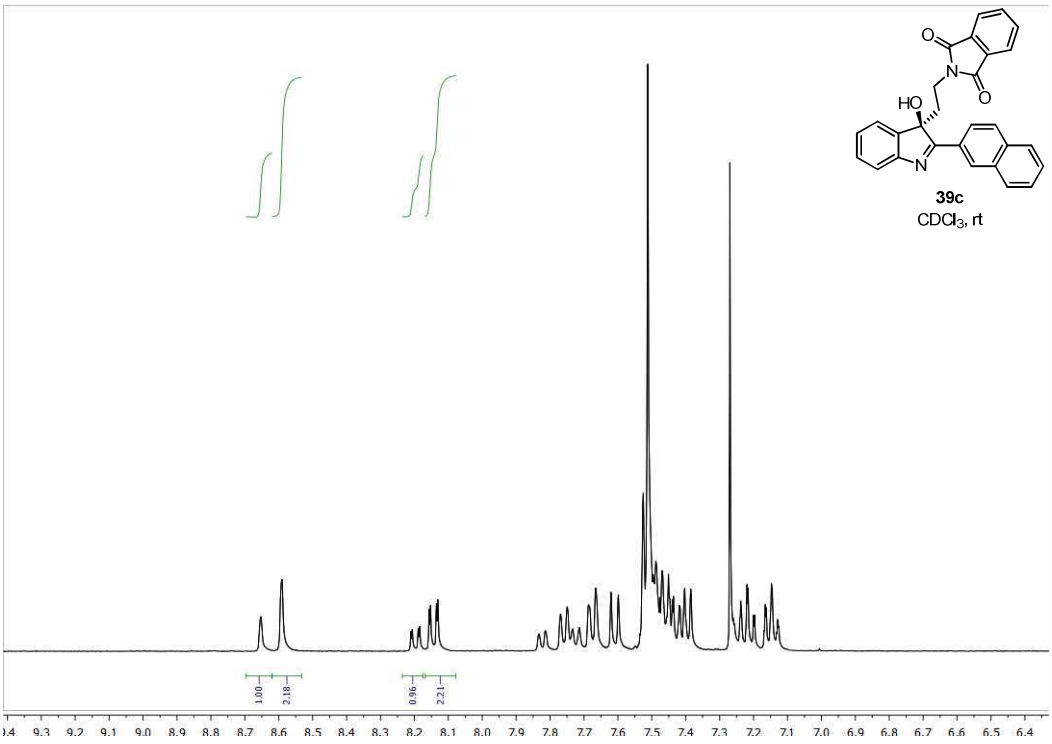
39a **45**

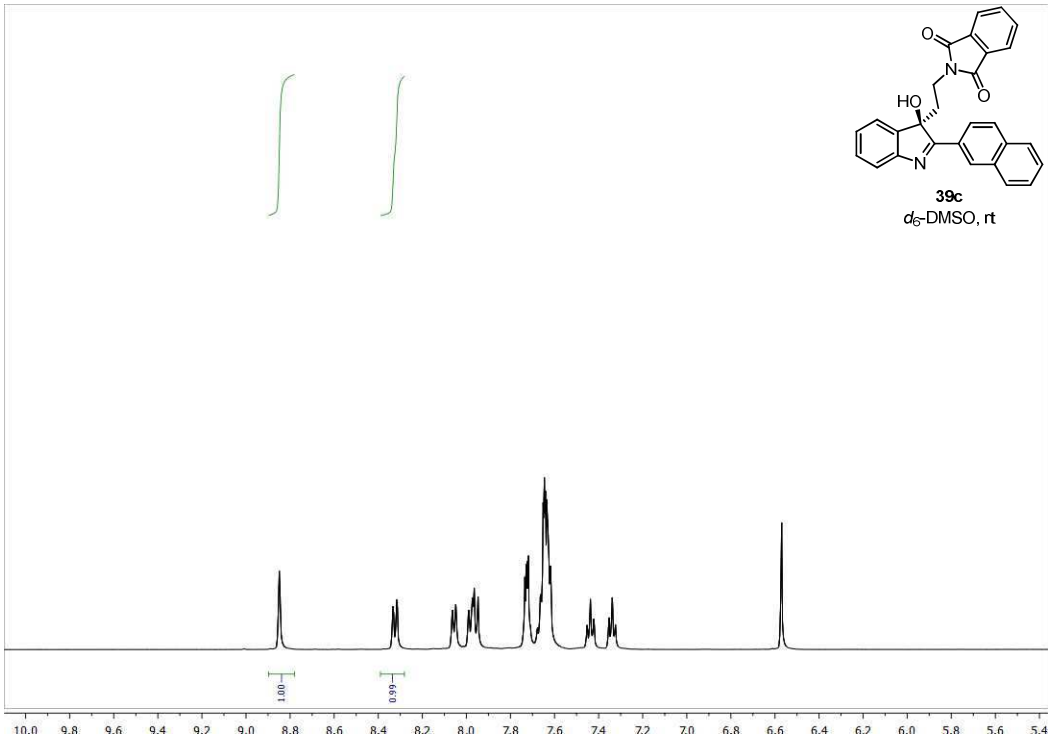
A 5-mL round-bottom flask was charged with 3-hydroxy-indolenine **39a** (19.8 mg, 0.04103 mmol, 1.0 equiv.), scandium(III) triflate (20.2 mg, 0.04103 mmol, 1.0 equiv.) and degassed toluene (4.1 mL).The flask was flushed with nitrogen, sealed, and heated in an oil bath at 110 °C for 5 hours. The resulting yellow solution was directly purified by preparative TLC eluting with 10% EtOAc/DCM. After removal of the desorption solvent (EtOAc), the 2-oxindole **45** was isolated as white solid (7.0 mg, 35% yield).

^{1}H NMR (500 MHz, $CDCl_3$, *J* in Hz) δ 8.51 (s, 1H), 8.38 (s, 1H), 8.05 (d, *J* = 7.0, 1H), 8.01 (d, *J* = 8.7, 1H), 7.91 (s, 1H), 7.88 (d, *J* = 8.3, 1H), 7.79 (dd, *J* = 5.5, 3.0, 2H), 7.68 (dd, *J* = 5.5, 3.0, 2H), 7.57 (dd, *J* = 8.4, 7.2, 1H), 7.53 (d, *J* = 8.4, 1H), 7.39 – 7.33 (m, 1H), 7.26 – 7.17 (m, 2H), 7.14 (d, *J* = 7.7, 1H), 6.95 – 6.90 (m, 1H), 6.87 (td, *J* = 7.4, 1.0, 1H), 4.02 – 3.94 (m, 1H), 3.91 – 3.83 (m, 1H), 2.96 – 2.83 (m, 2H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 180.6, 168.3, 140.1, 134.8, 134.1, 133.7, 133.0, 132.3, 131.7, 131.2, 130.0, 129.7, 128.9, 128.8, 127.6, 127.6, 126.6, 125.8, 125.5, 124.7, 124.2, 123.6, 123.3, 123.2, 110.5, 55.9, 36.4, 34.2; **FTIR** (cm^{-1}) 3245, 2926, 1770, 1708, 1617, 1399, 1369; **UPLC-MS** (ESI) *m/z* 483.62 (calculated for $C_{32}H_{22}N_2O_3$ [M + H^+] = 483.16); **TLC** R_f = 0.45 (10% EtOAc/DCM); **Chiral HPLC analysis**: 99:1 er, performed on Chiralcel OD-H (Daicel, 4.6 mm × 250 mm, 5 μm, 20 °C), eluting at 0.7 mL/min with 55:45 hexanes/ethanol. Retention times: t_{major} = 13.8 min, t_{minor} = 35.8 min; $[\alpha]_{546\ nm}^{20\ ℃}$= -1.1 (*c* = 0.82, $CHCl_3$).

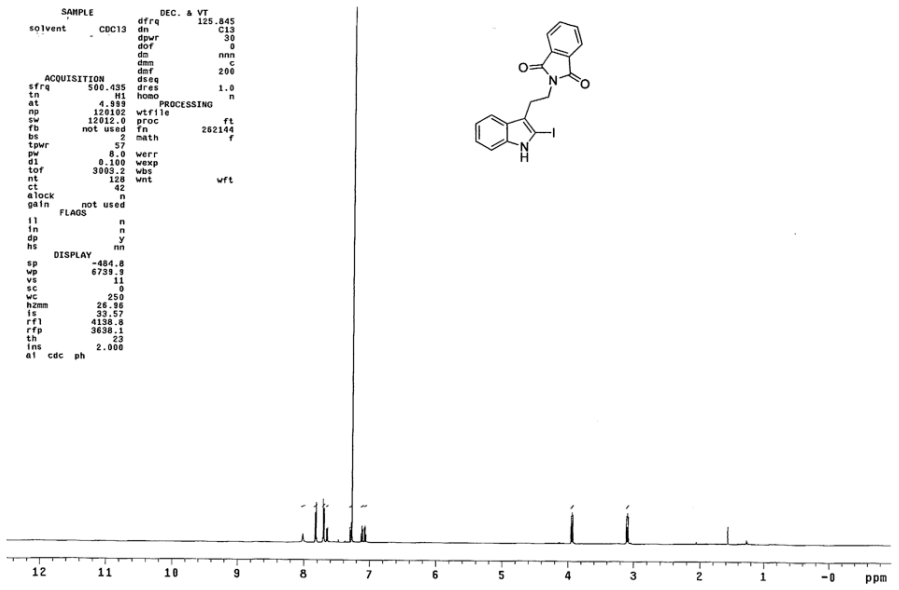
J. NMR Spectra.

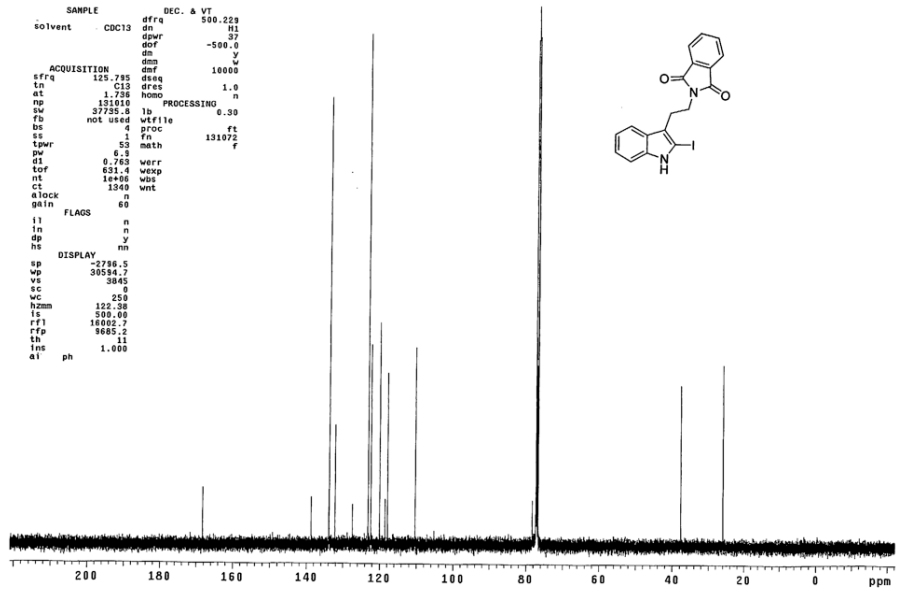
As shown below, some of the oxidation products exhibit conformational equilibria, resulting in doubling of signals at room temperature.As shown for compound **39c**, solvent exchange in the NMR experiments reveals reversible coalescence of signals with the introduction of d_6-DMSO.



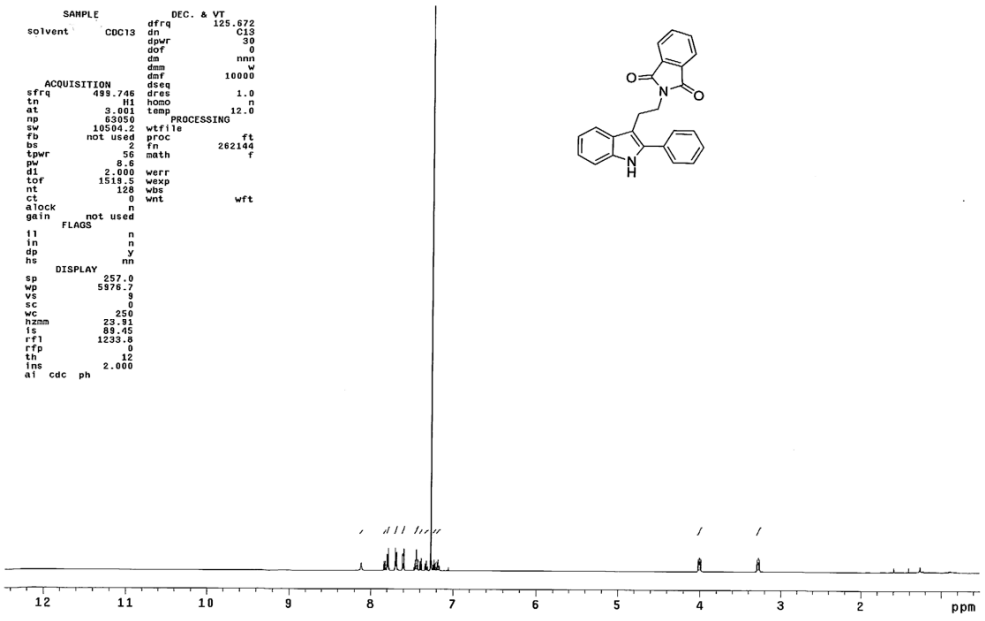


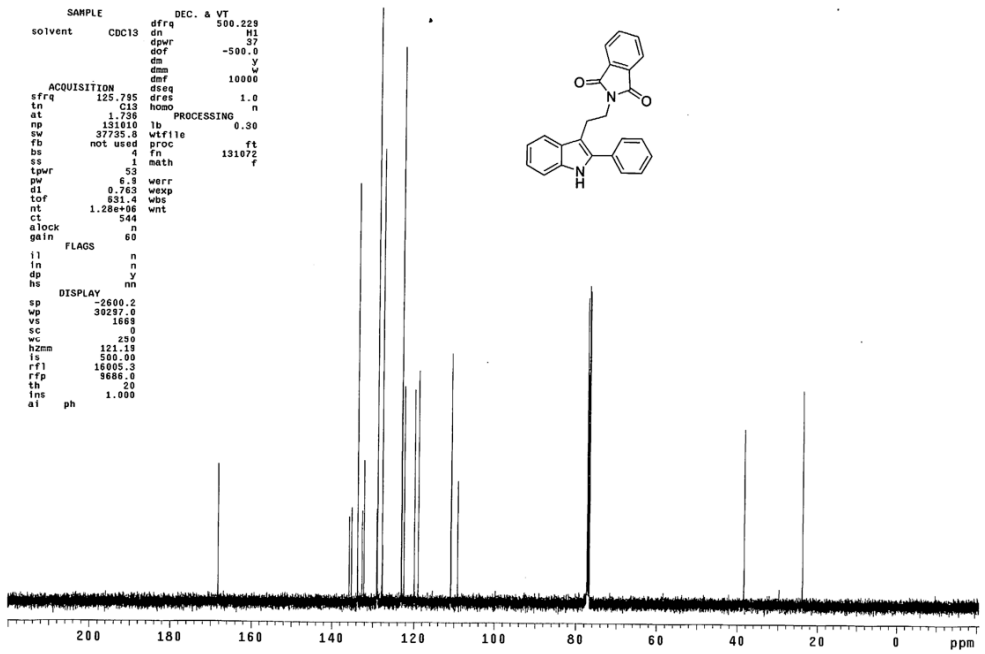
***N*,*N*-Phthaloyl-2-iodotryptamine (S1):**



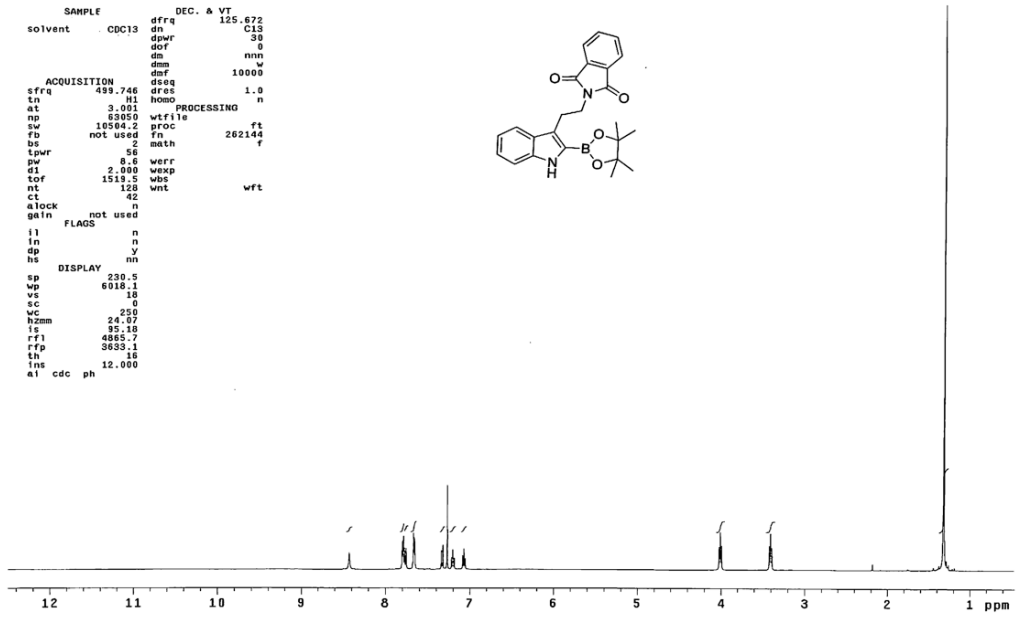


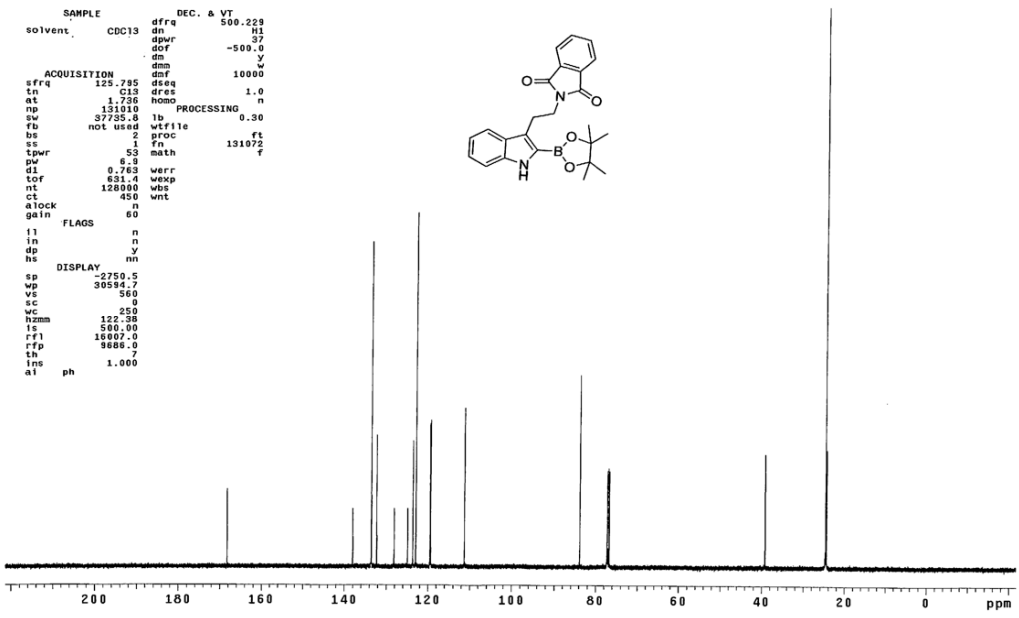
N,*N*-Phthaloyl-2-phenyltryptamine (14):



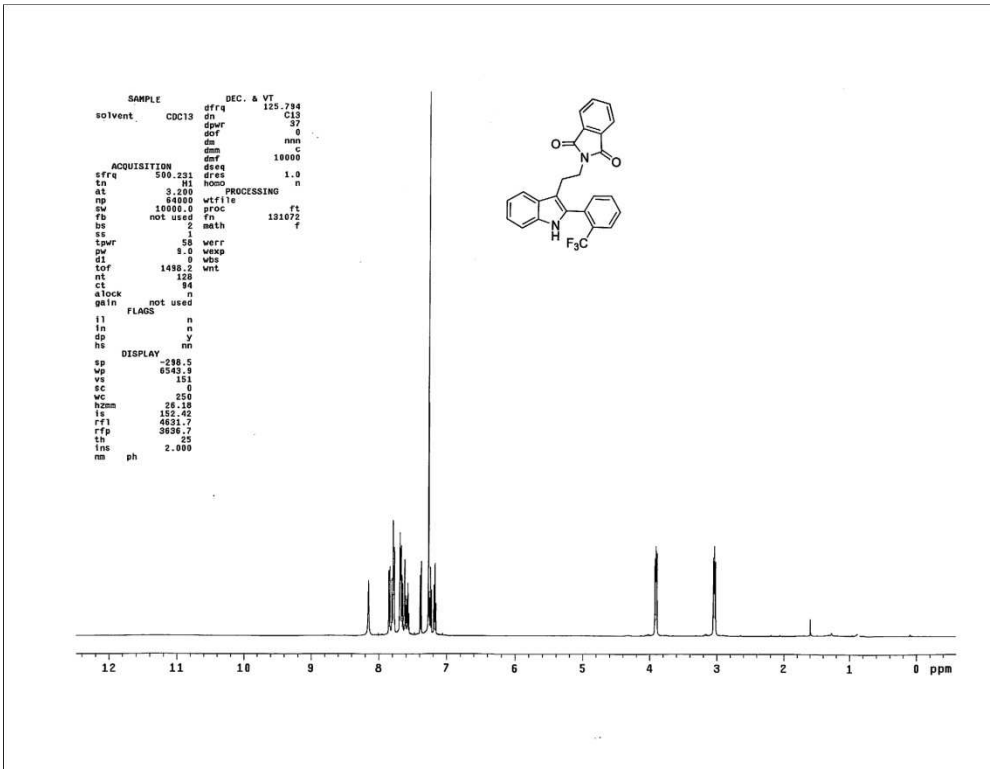


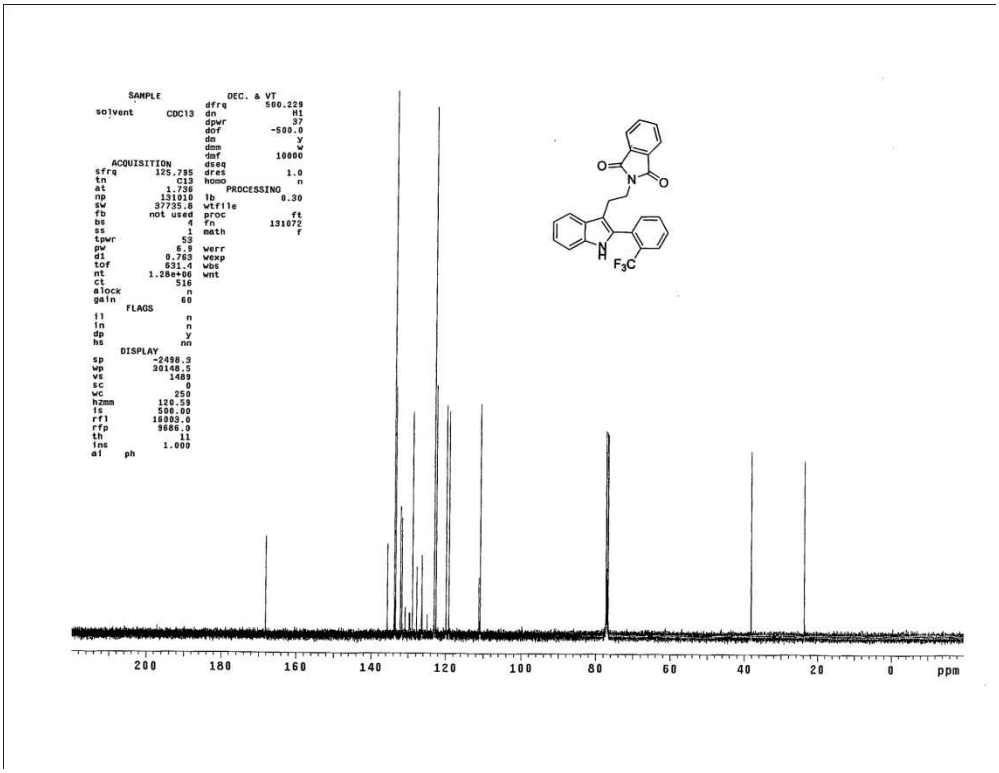
N,*N*-Phthaloyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)tryptamine (S2):



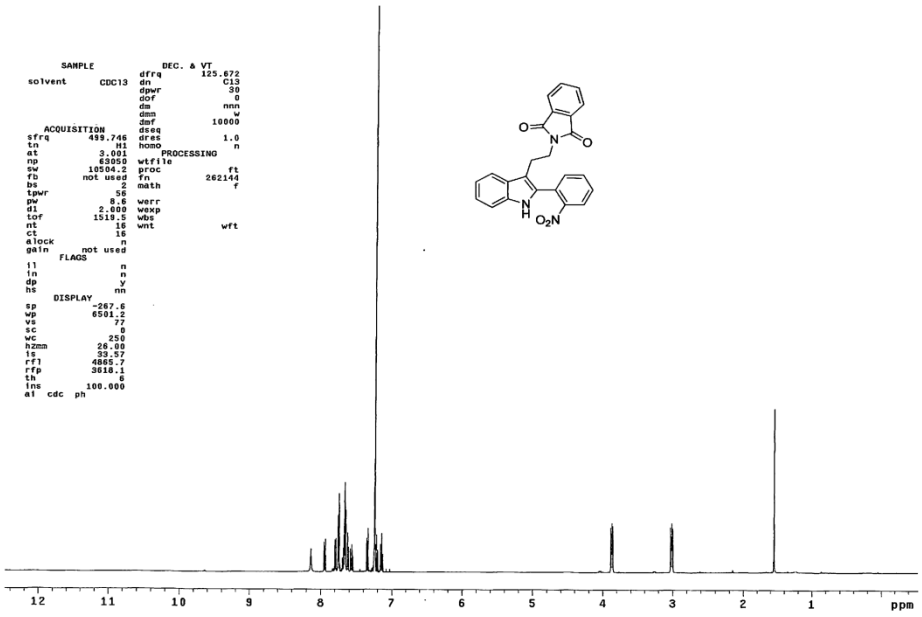


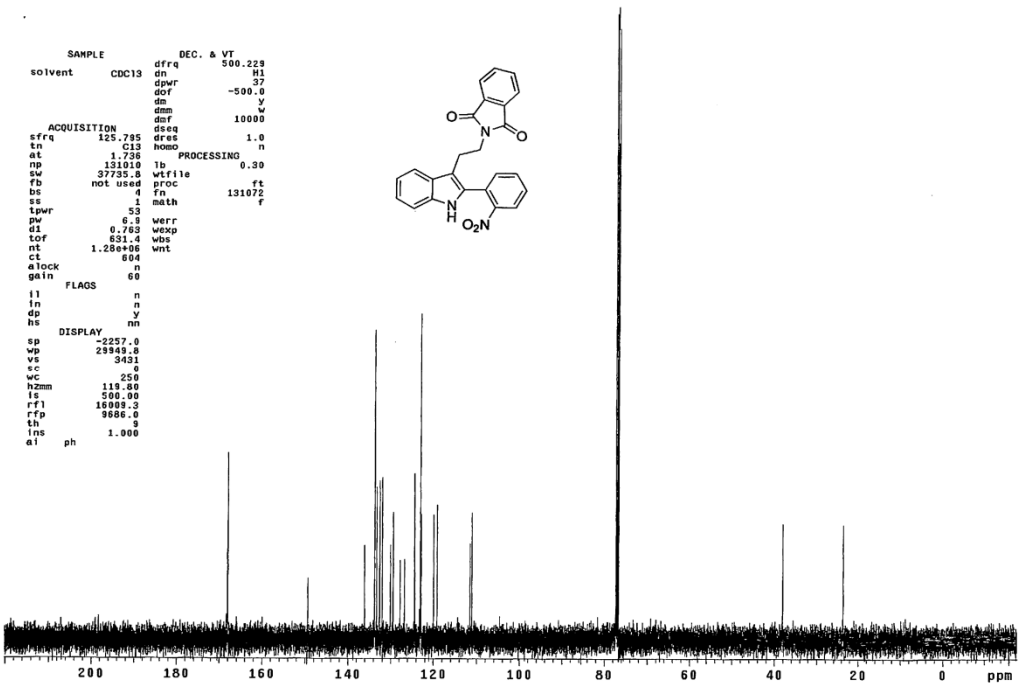
2-(2-(2-(2-(Trifluoromethyl)phenyl)-1*H*-indol-3-yl)ethyl)isoindoline-1,3-dione (Table 1, Entry 36a):



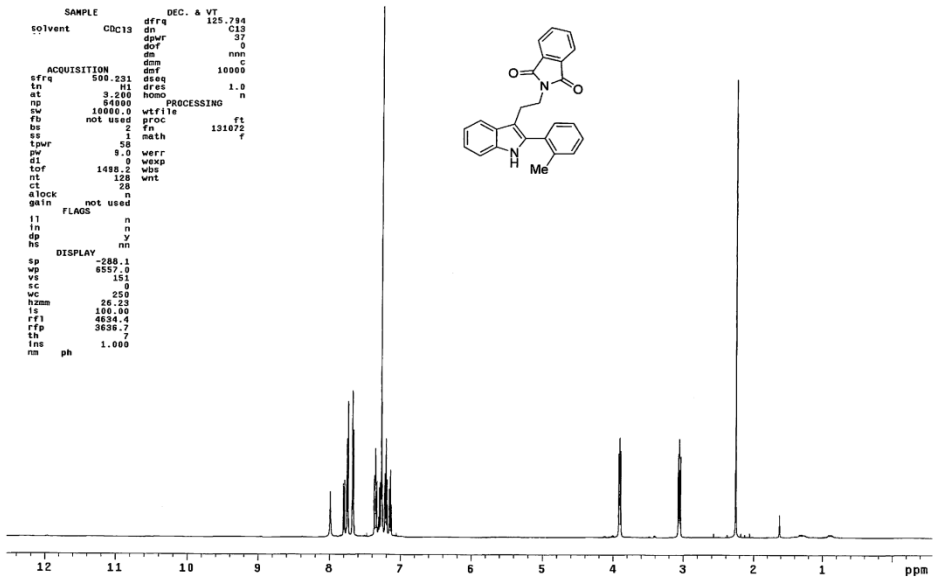


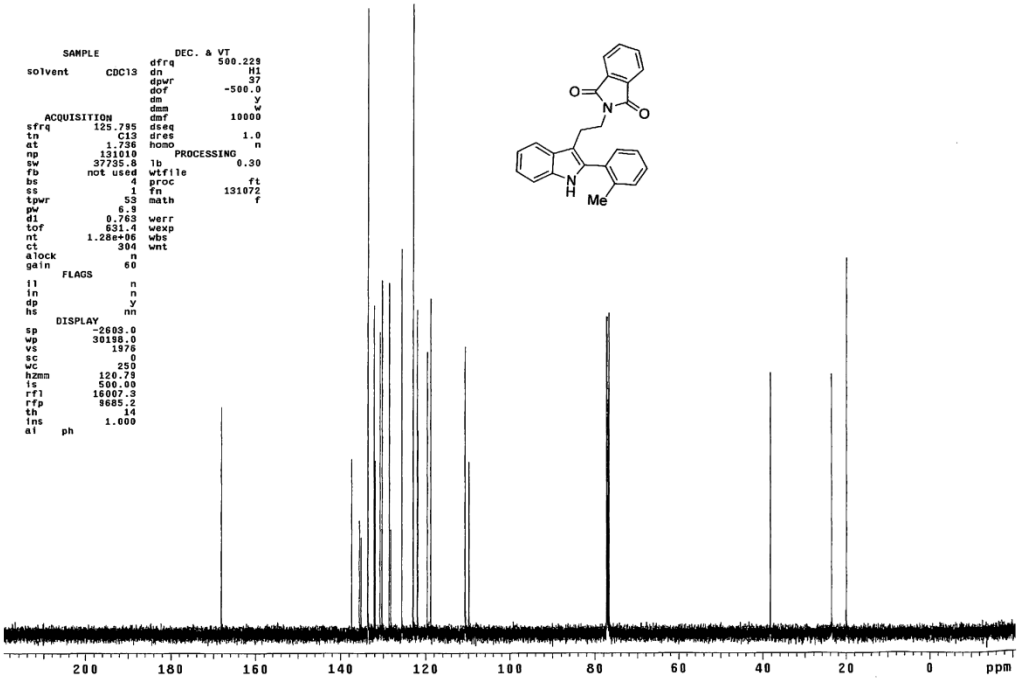
2-(2-(2-(2-Nitrophenyl)-1H-indol-3-yl)ethyl)isoindoline-1,3-dione :(Table 1, Entry 36b):



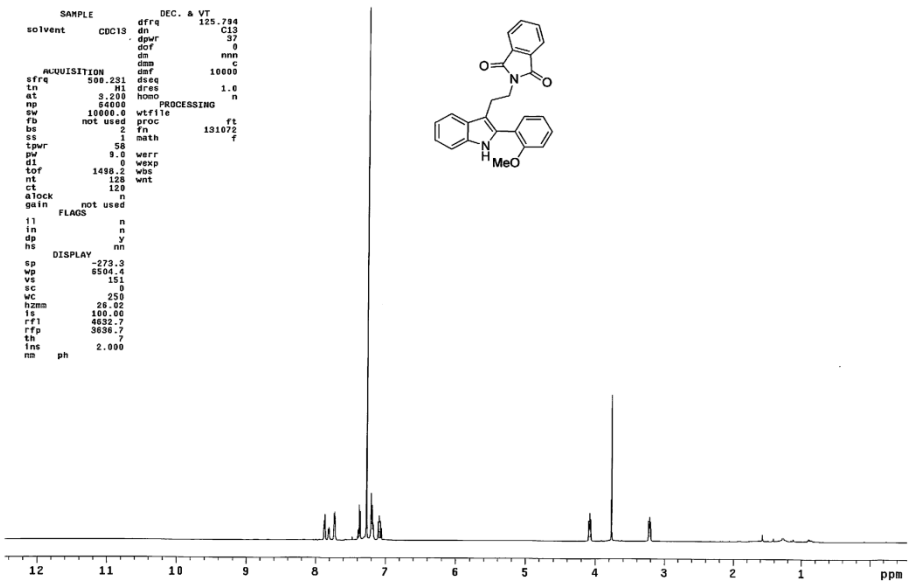


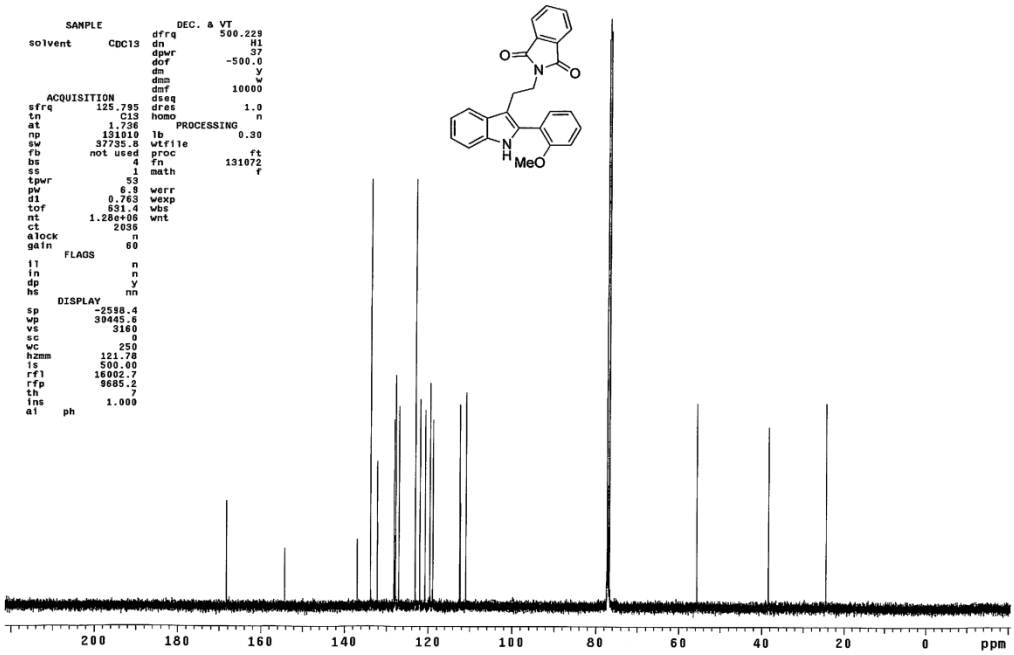
2-(2-(2-(*o*-Tolyl)-1*H*-indol-3-yl)ethyl)isoindoline-1,3-dione: (Table 1, Entry 36c):



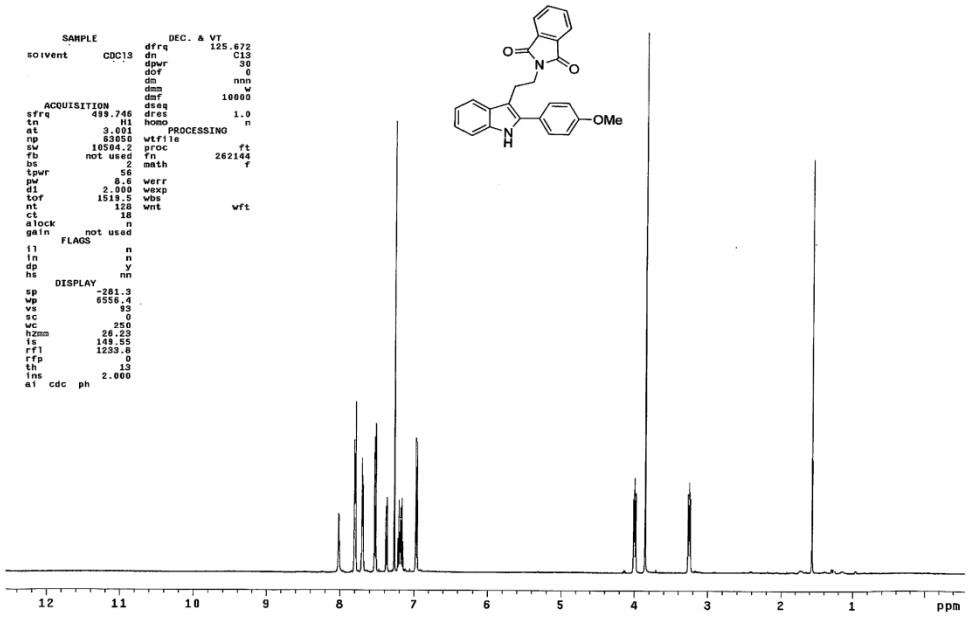


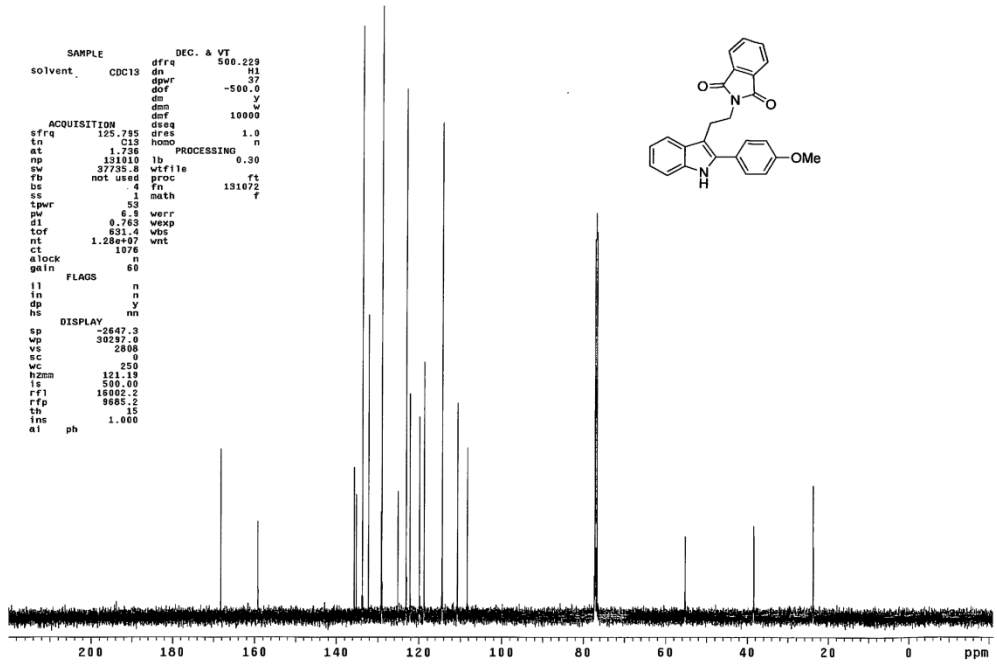
2-(2-(2-(2-Methoxyphenyl)-1*H*-indol-3-yl)ethyl)isoindoline-1,3-dione : (Table 1, Entry 36d):



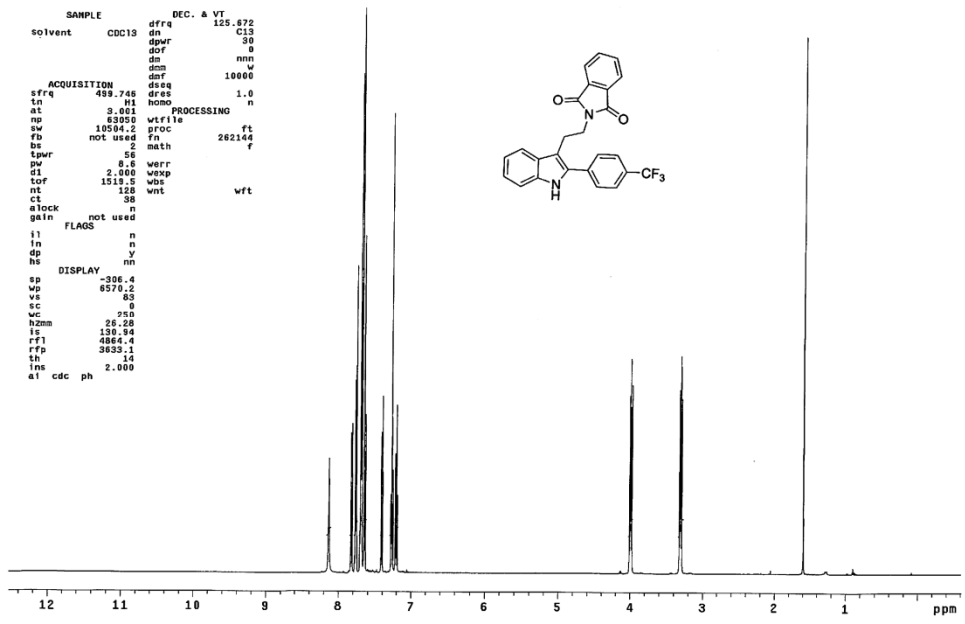


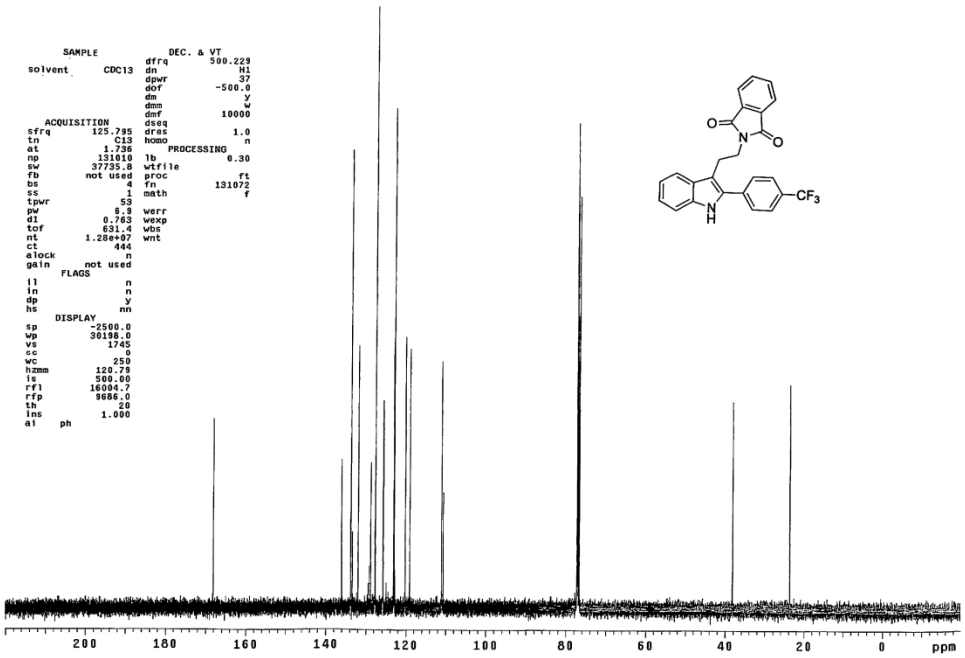
2-(2-(2-(4-Methoxyphenyl)-1*H*-indol-3-yl)ethyl)isoindoline-1,3-dione: (Table 1, Entry 36e):



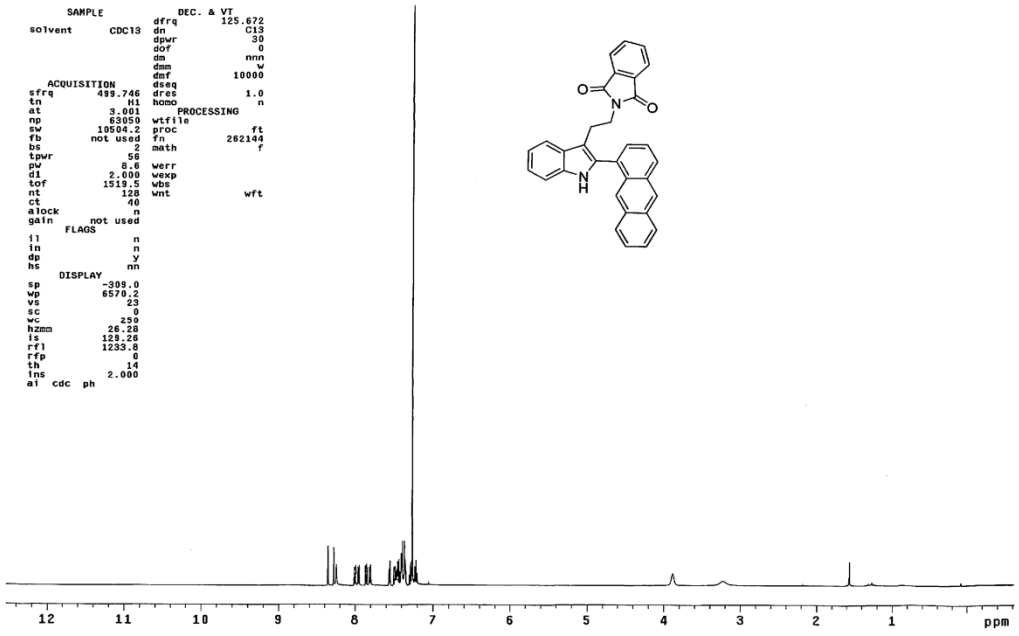


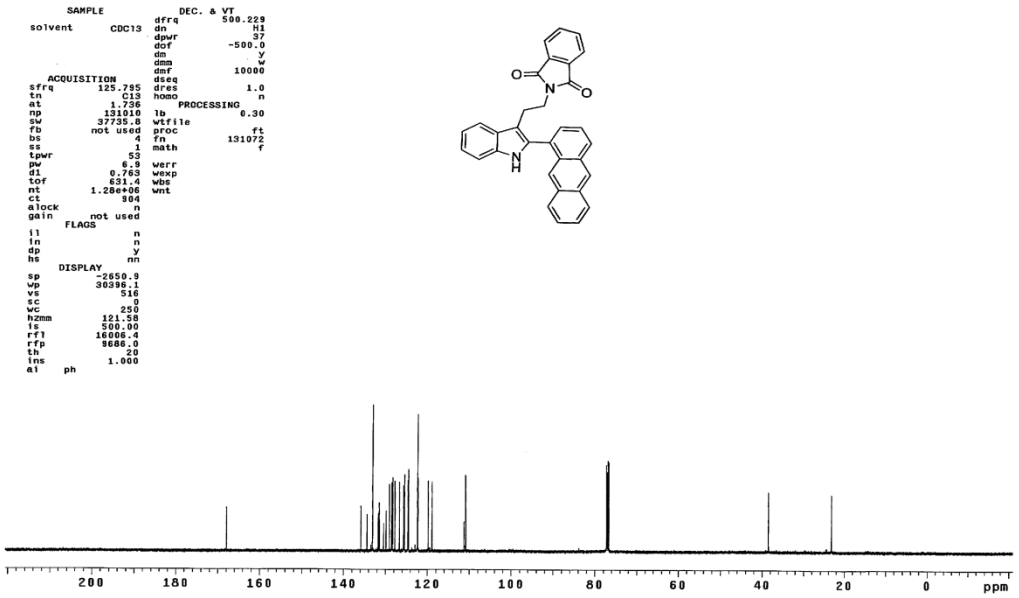
2-(2-(2-(4-(Trifluoromethyl)phenyl)-1*H*-indol-3-yl)ethyl)isoindoline-1,3-dione: (Table 1, Entry 36f):



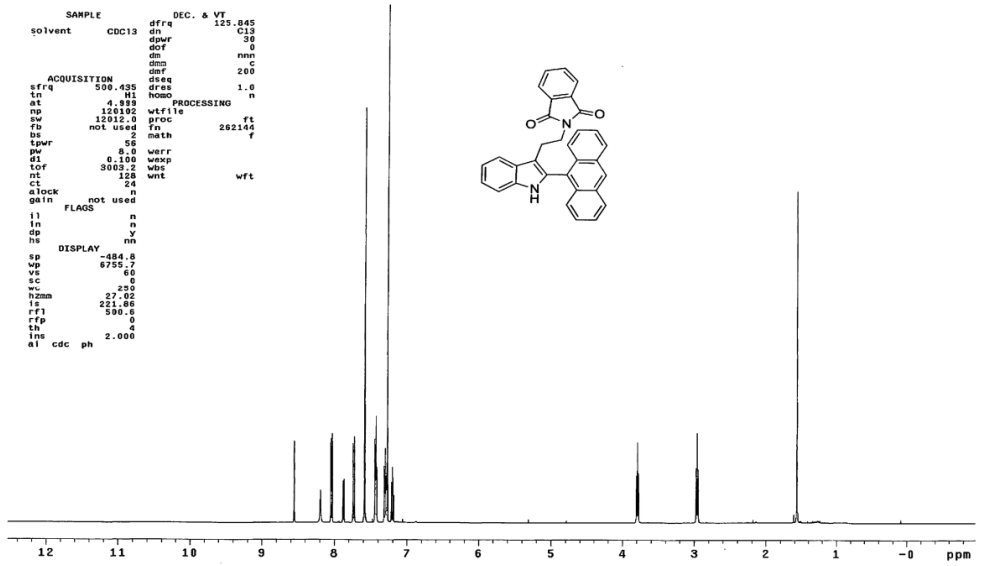


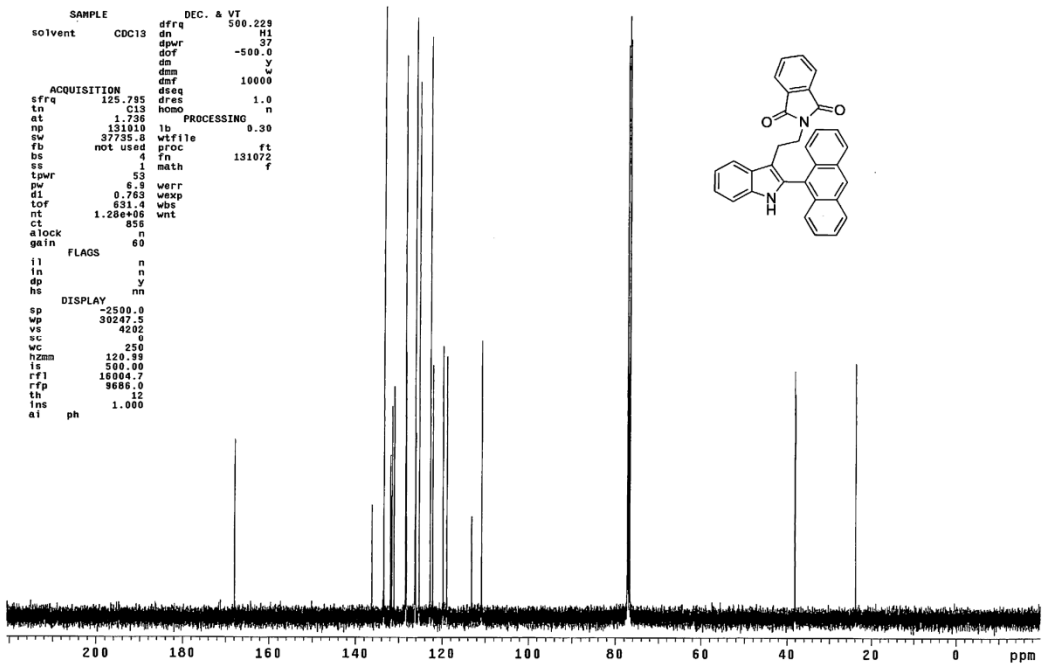
2-(2-(2-(Anthracen-1-yl)-1*H*-indol-3-yl)ethyl)isoindoline-1,3-dione: (Table 2, Entry 38a):



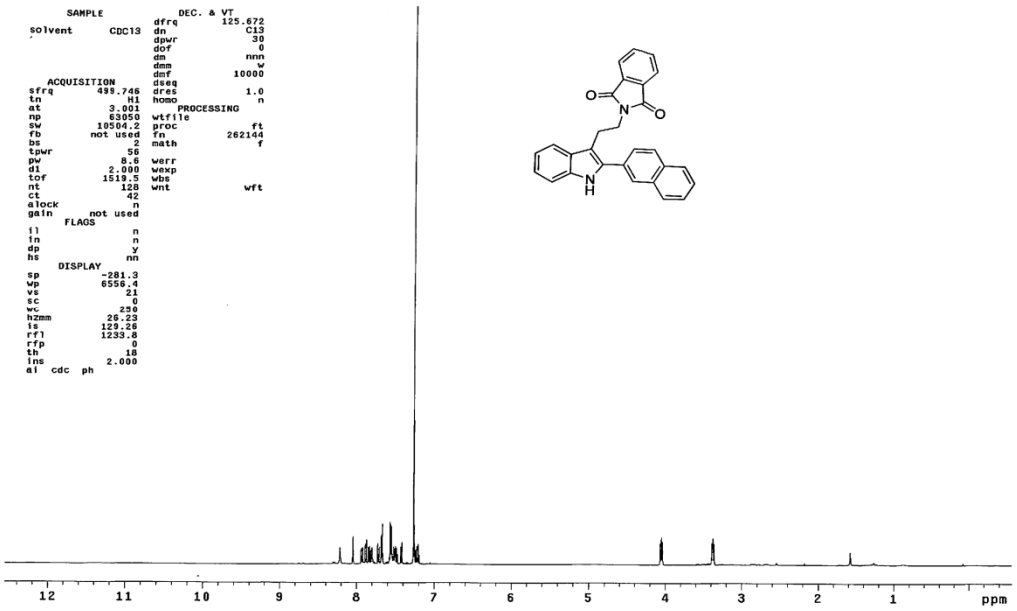


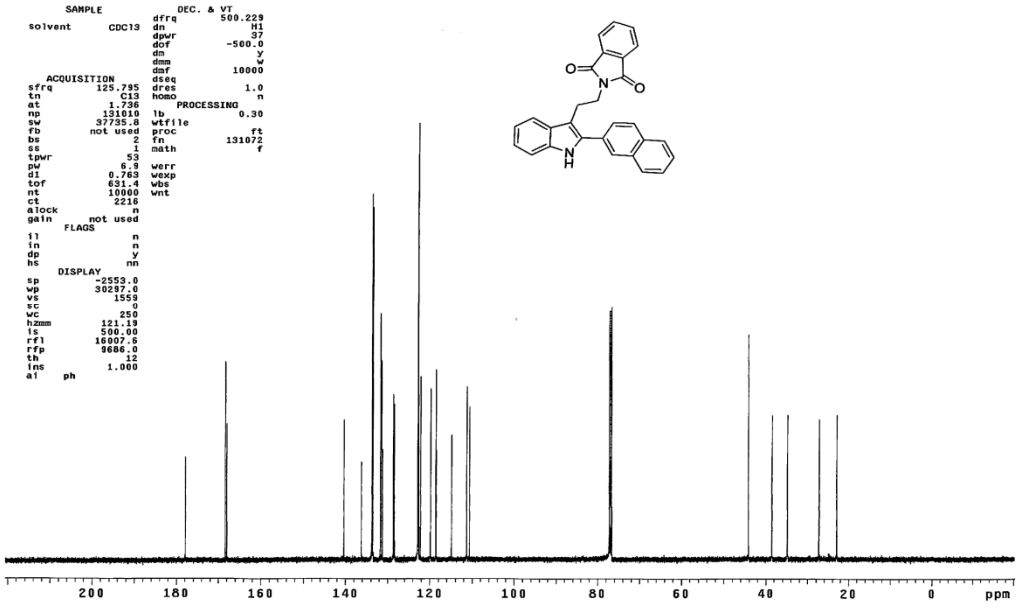
2-(2-(2-(Anthracen-9-yl)-1*H*-indol-3-yl)ethyl)isoindoline-1,3-dione: (Table 2, Entry 38b):



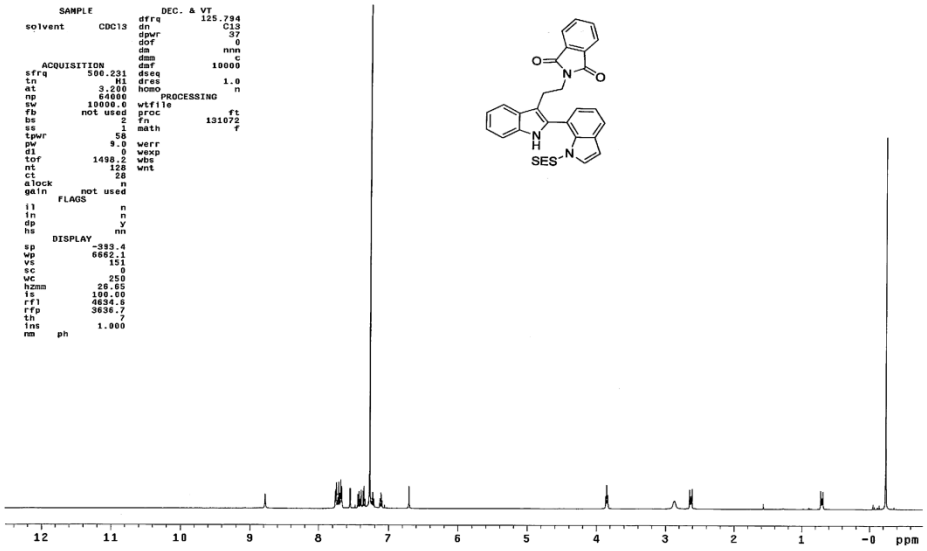


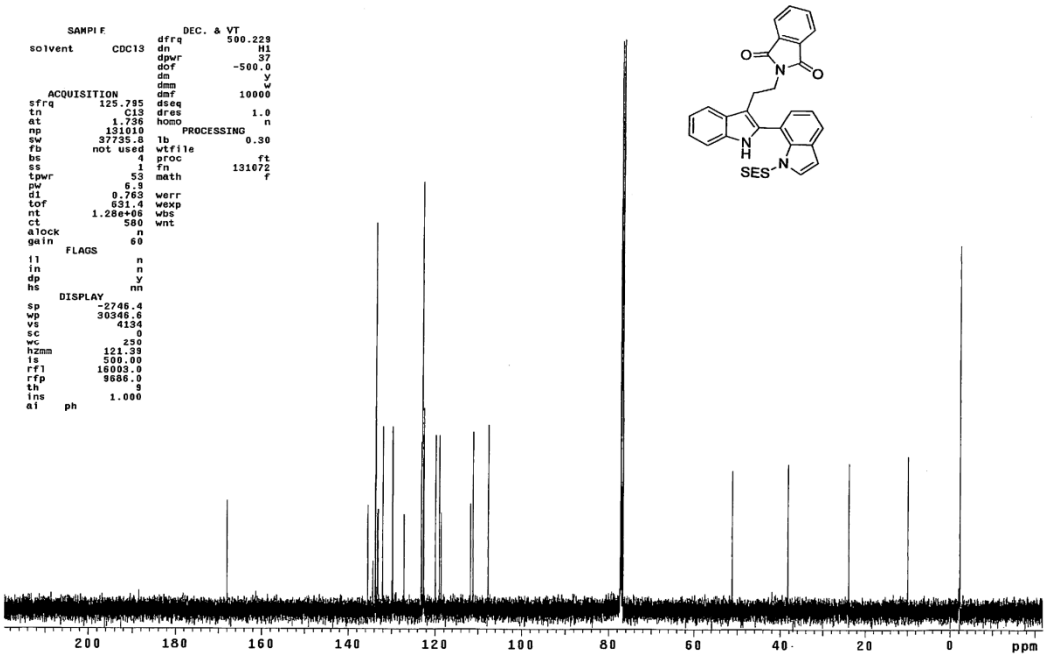
2-(2-(2-(Naphthalen-2-yl)-1*H*-indol-3-yl)ethyl)isoindoline-1,3-dione: (Table 2, Entry 38c):

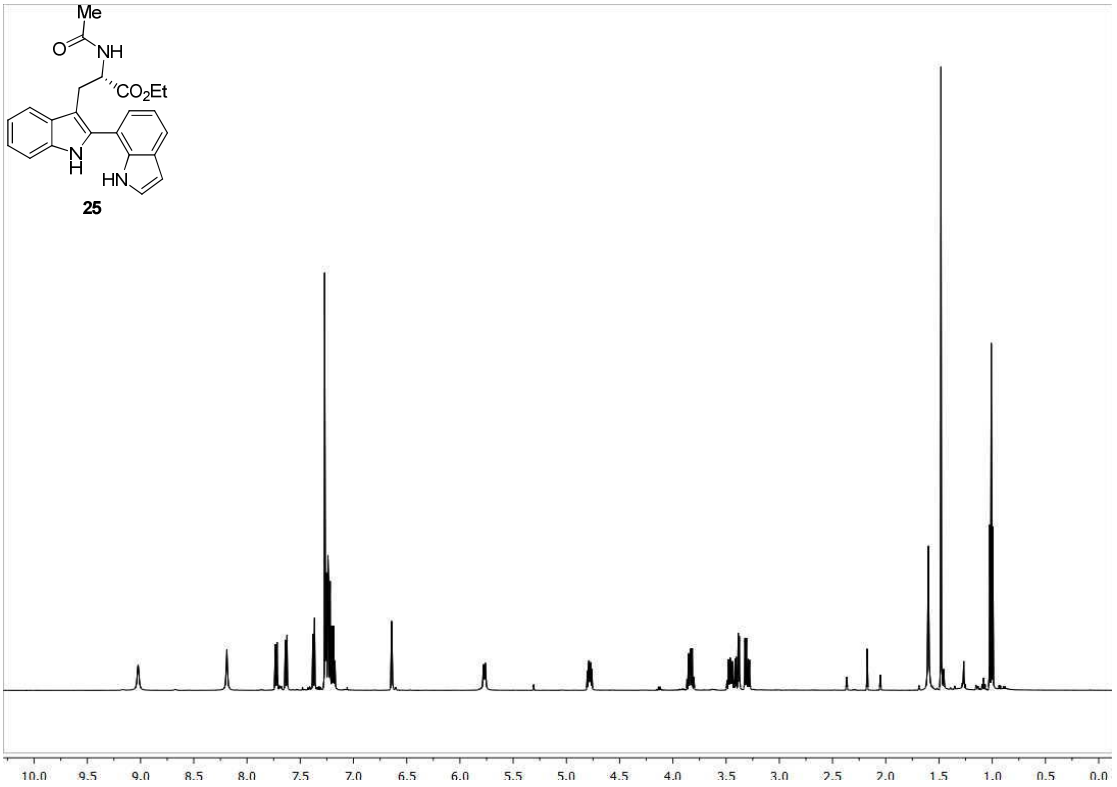




2-(2-(1'-((2-(Trimethylsilyl)ethyl)sulfonyl)-1*H*,1'*H*-[2,7'-bisindol]-3-yl)ethyl)isoindoline-1,3-dione: (Table 3, Entry 42):



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H
HN
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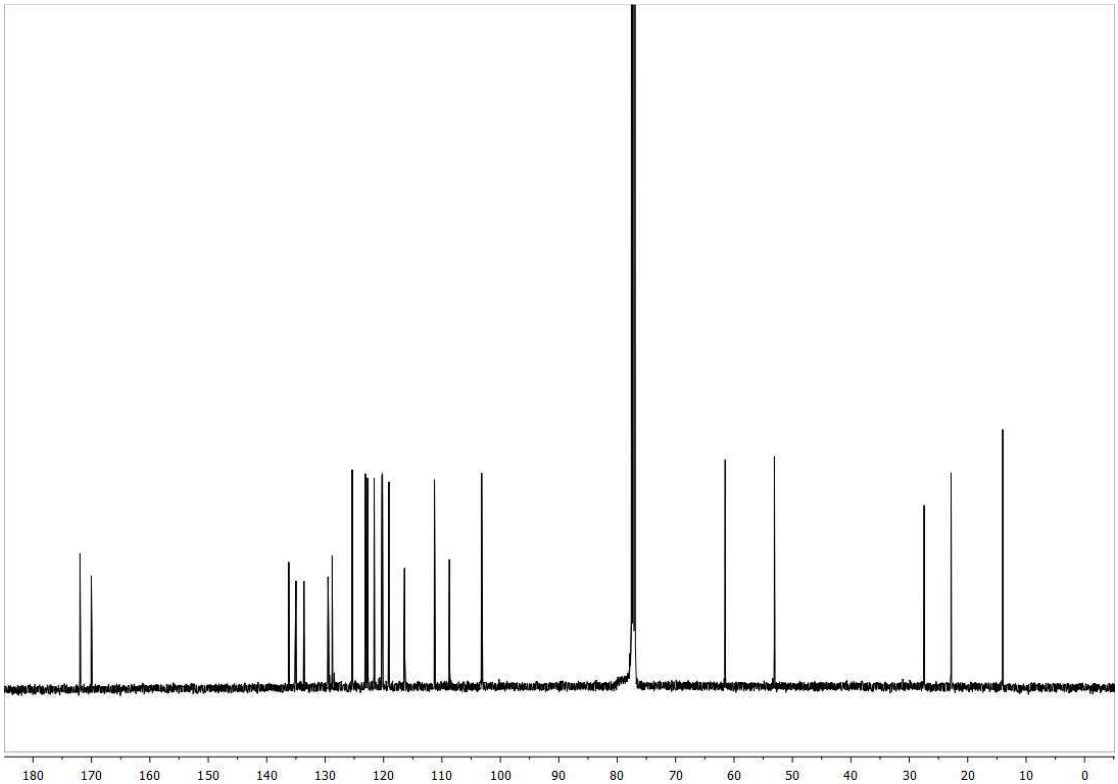
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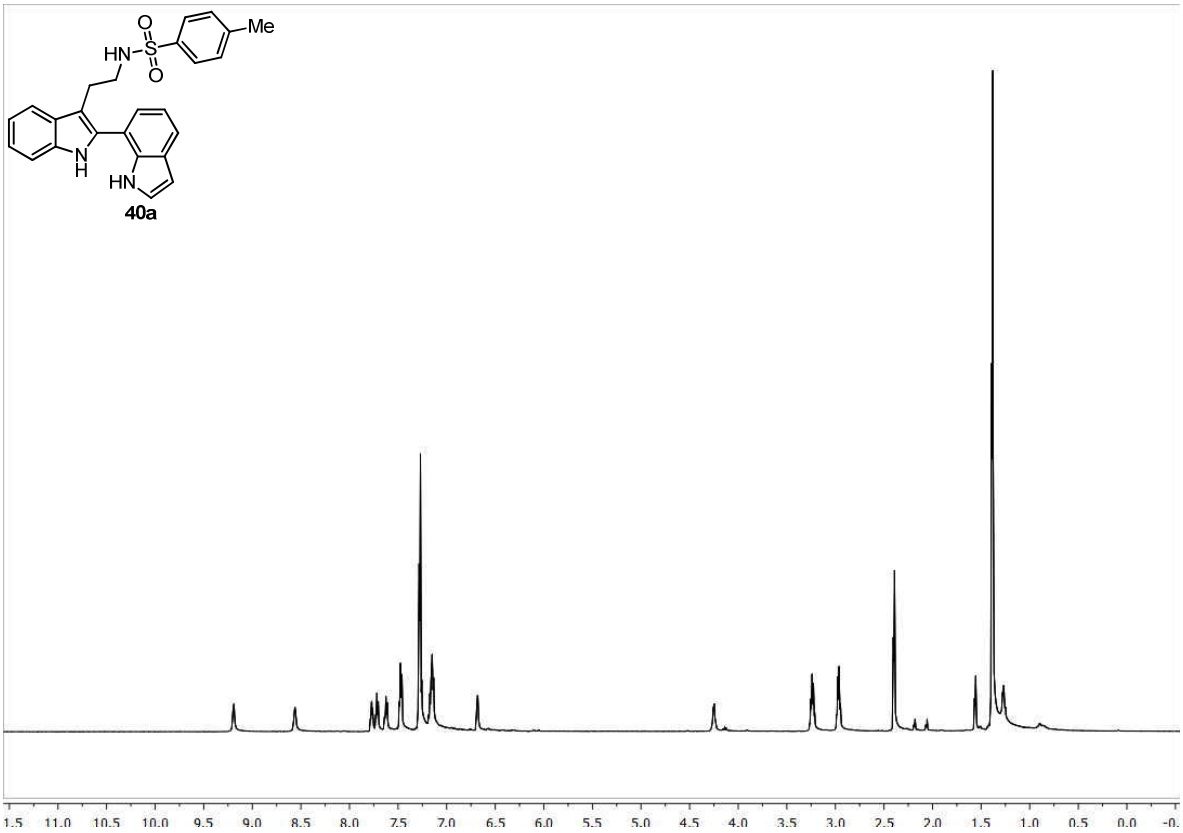
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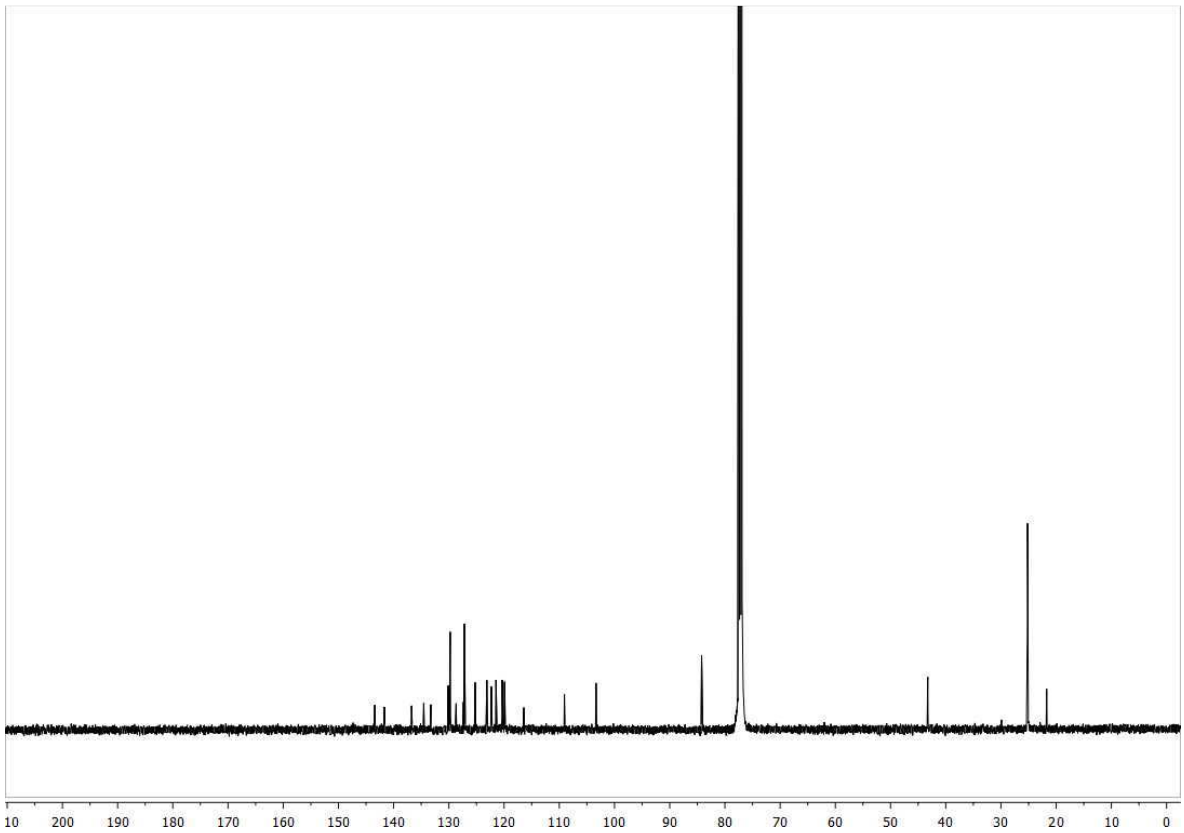

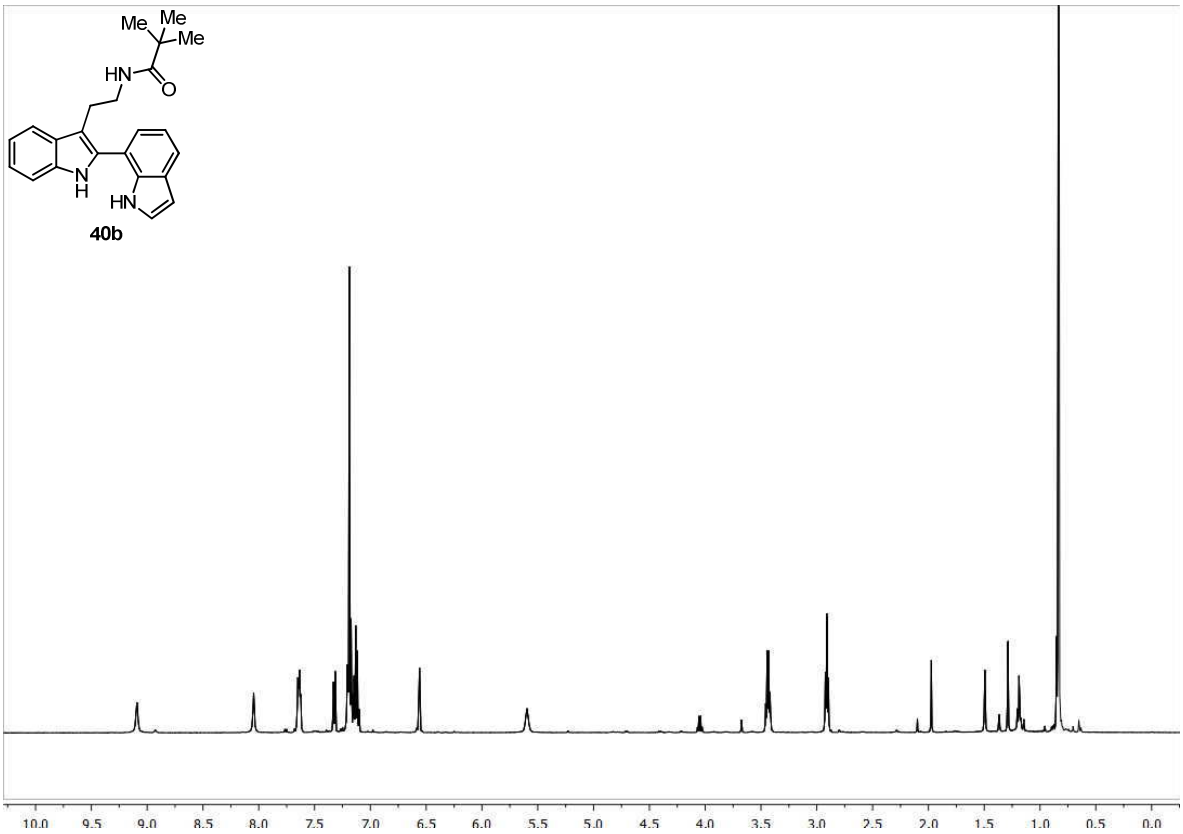
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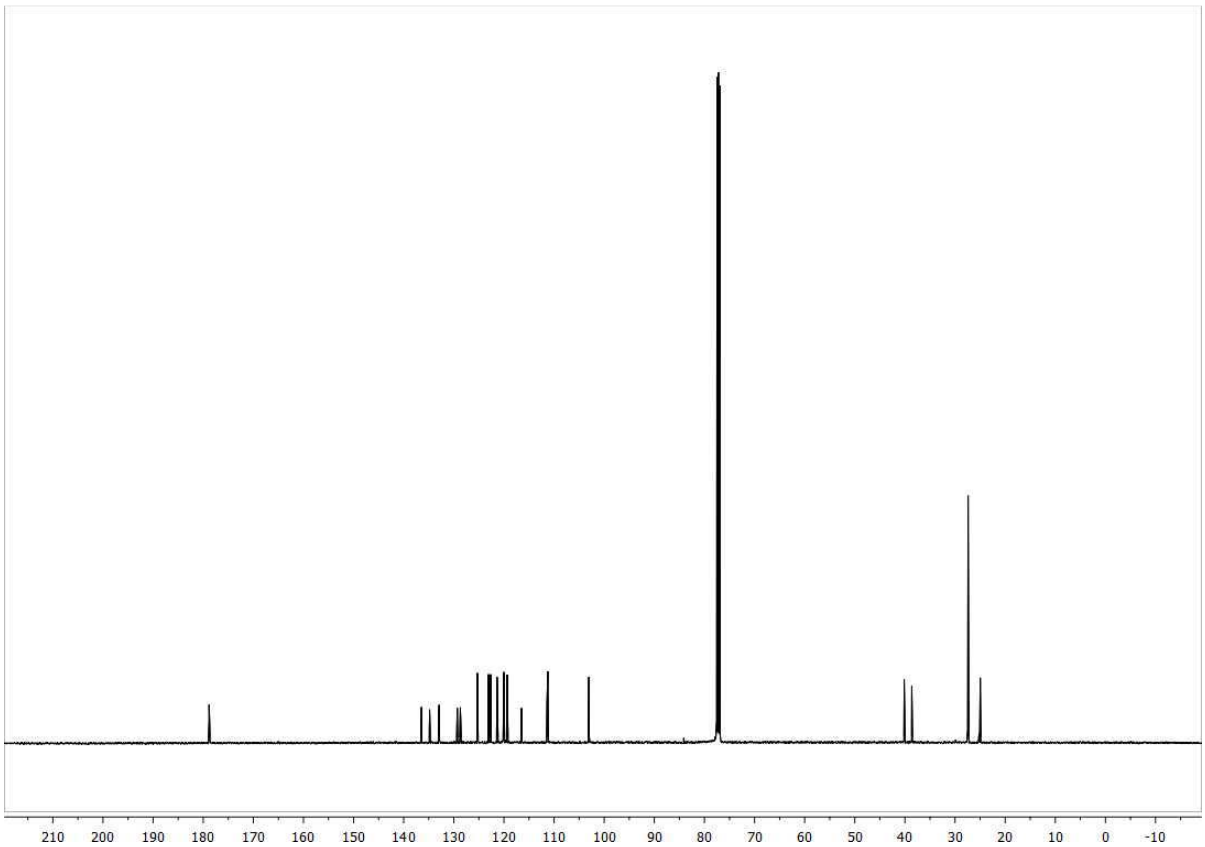
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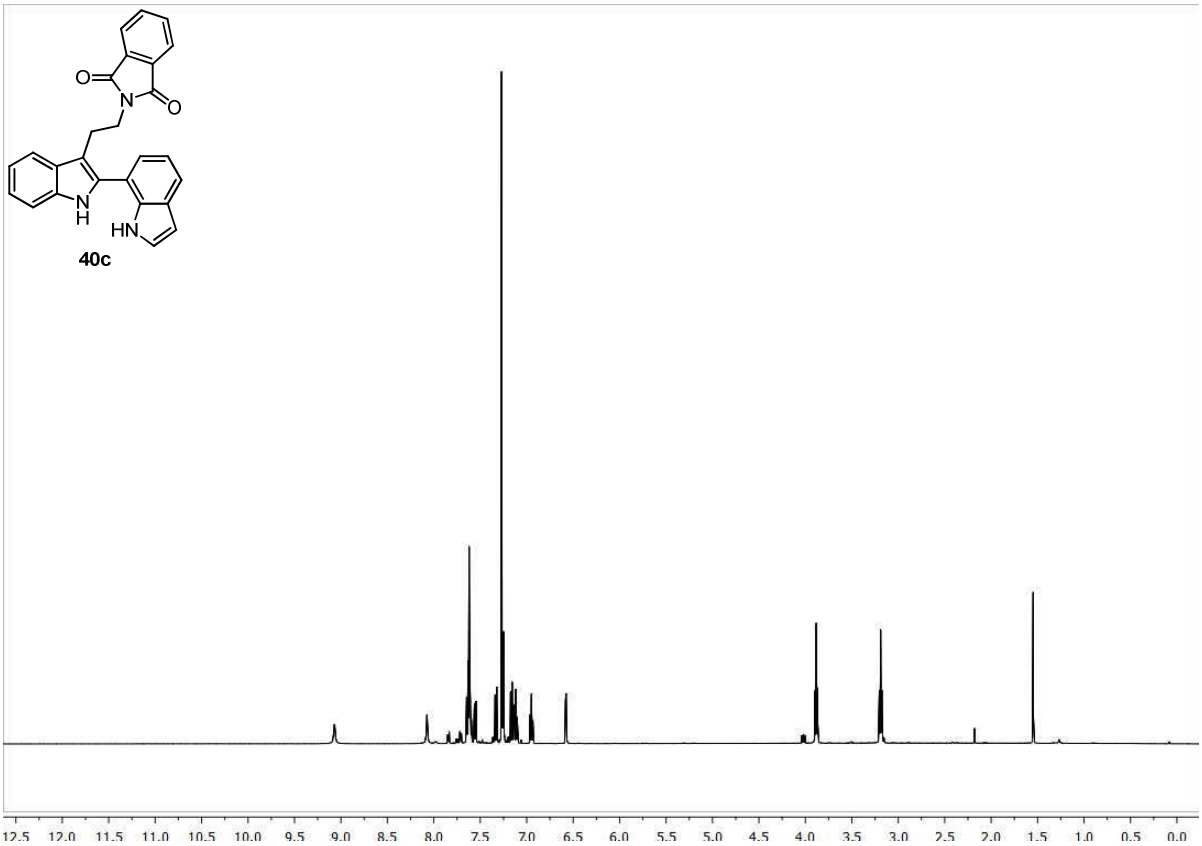
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H
HN
40c
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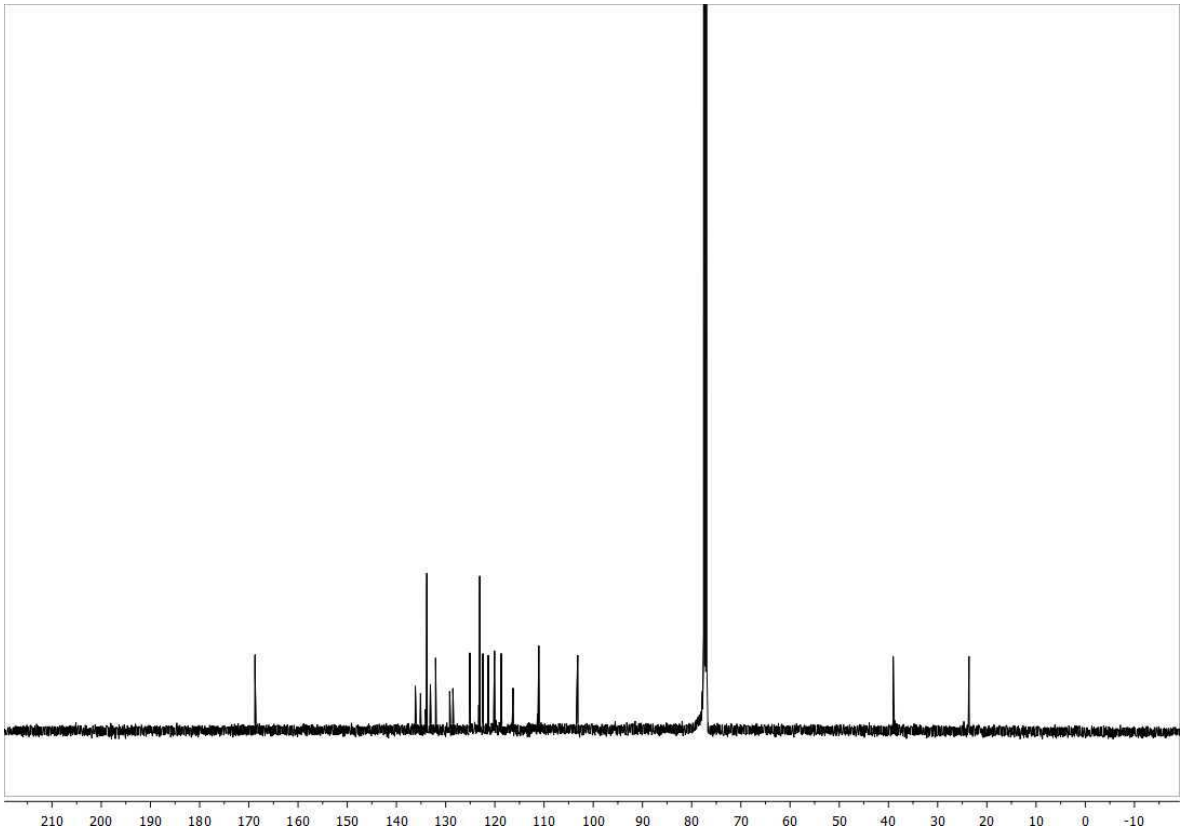
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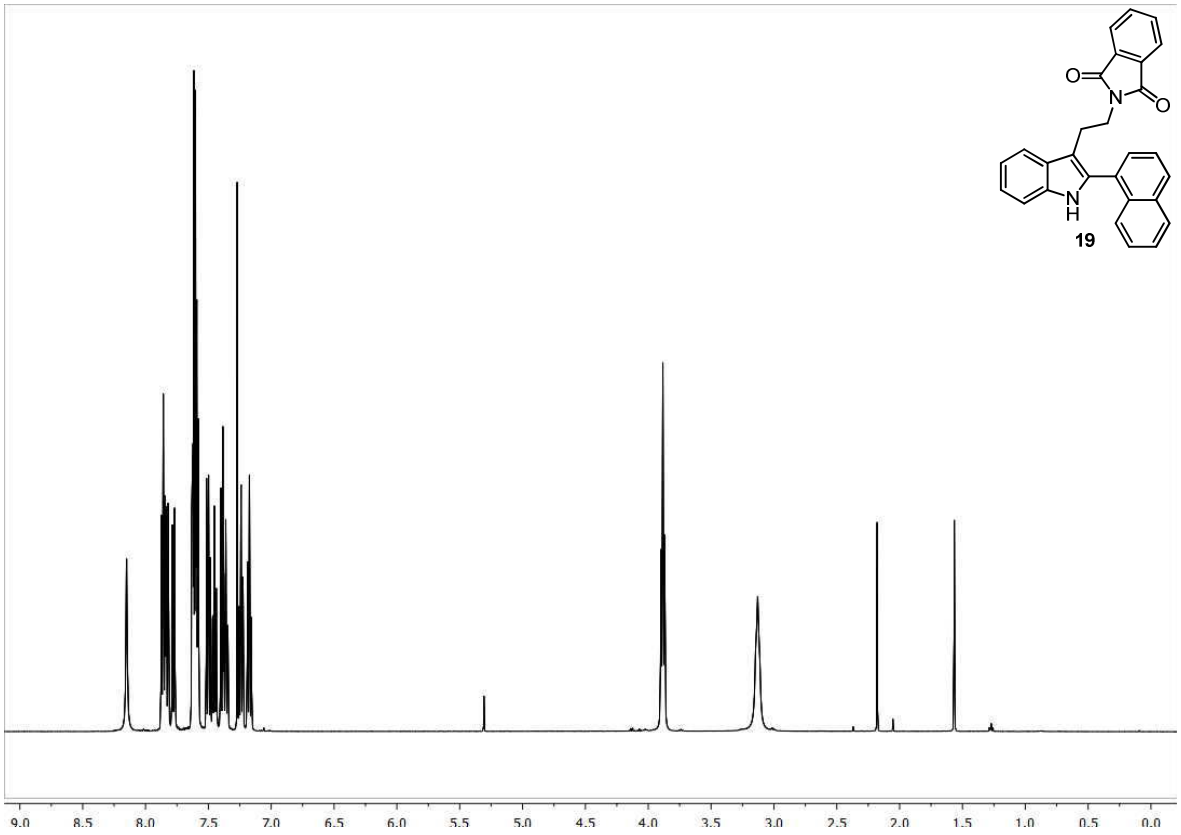
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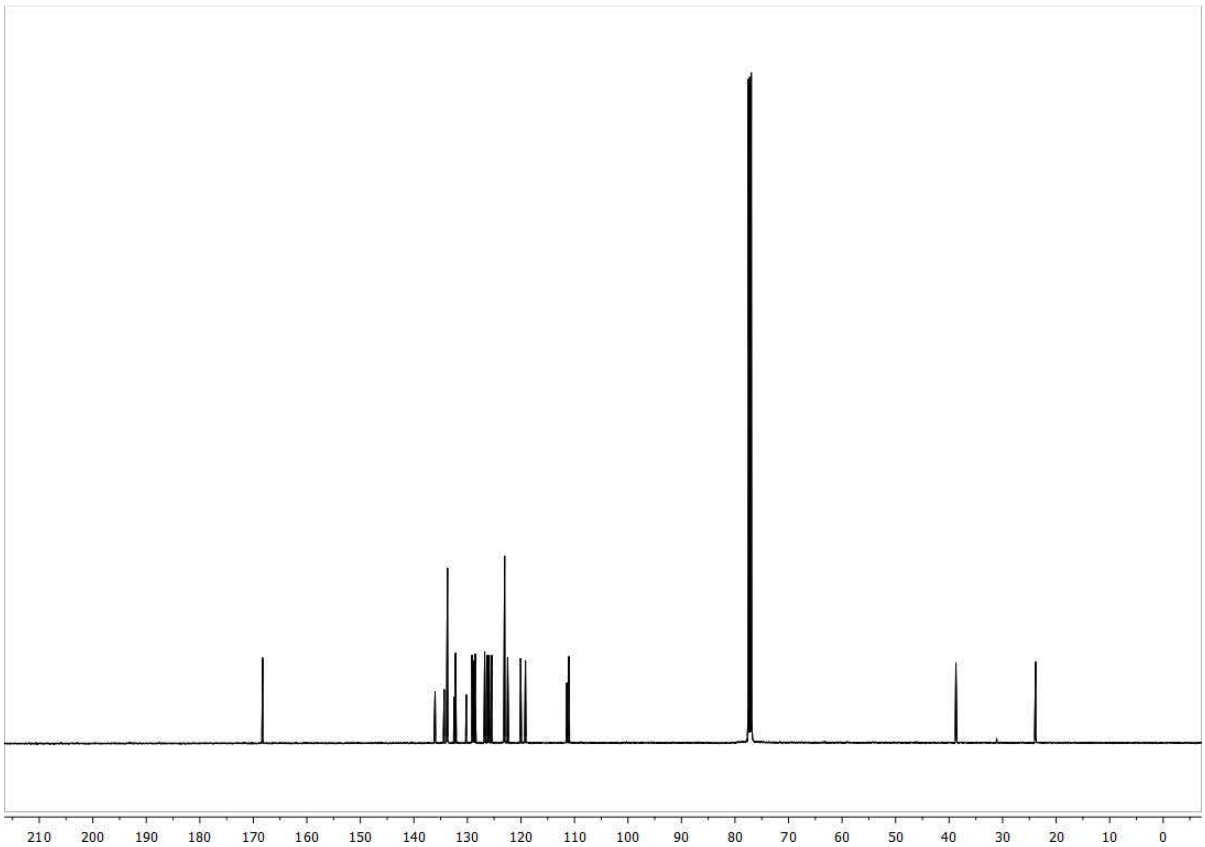
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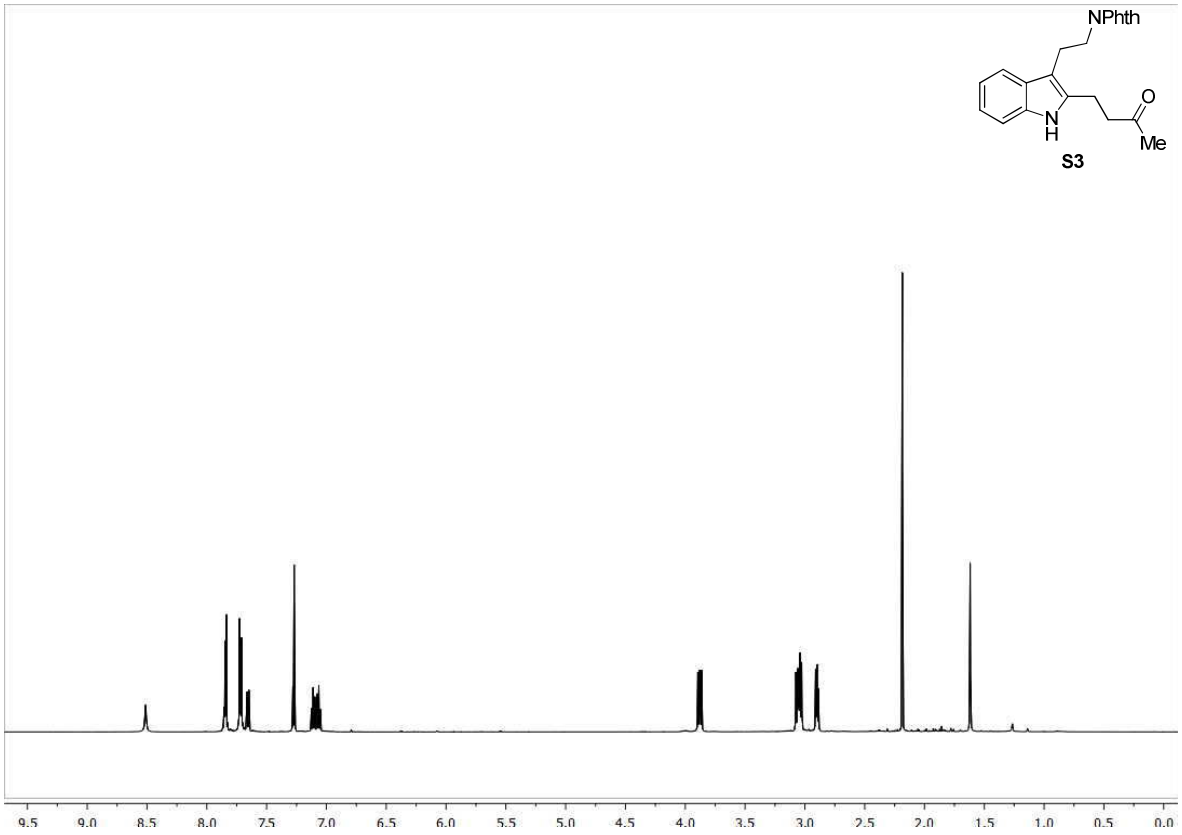
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Me
S3
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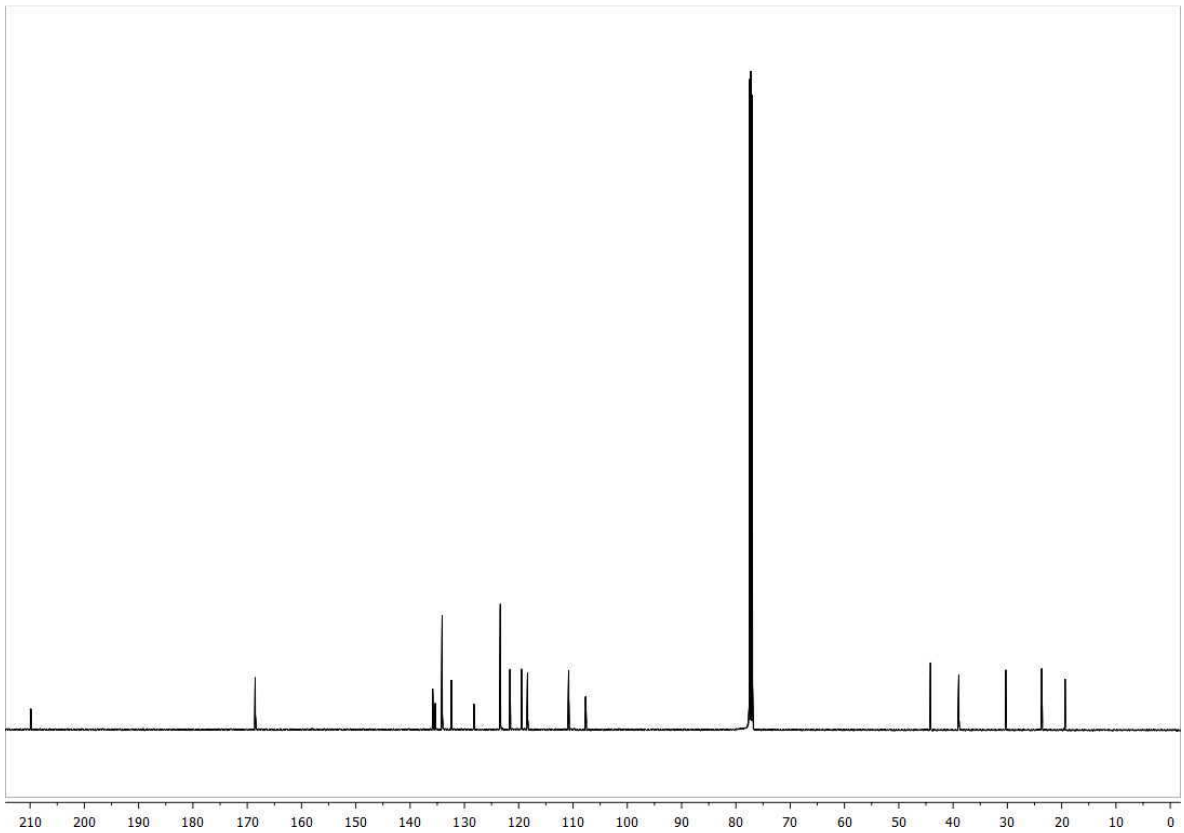
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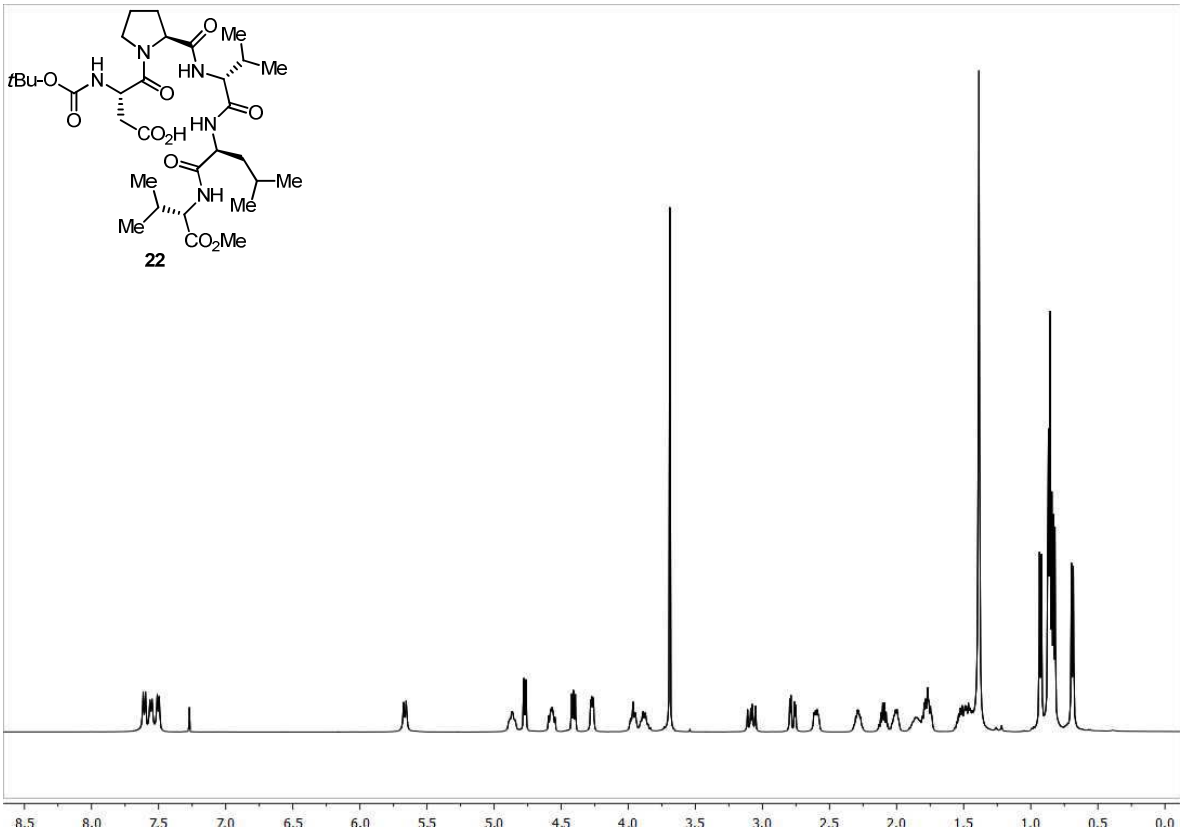
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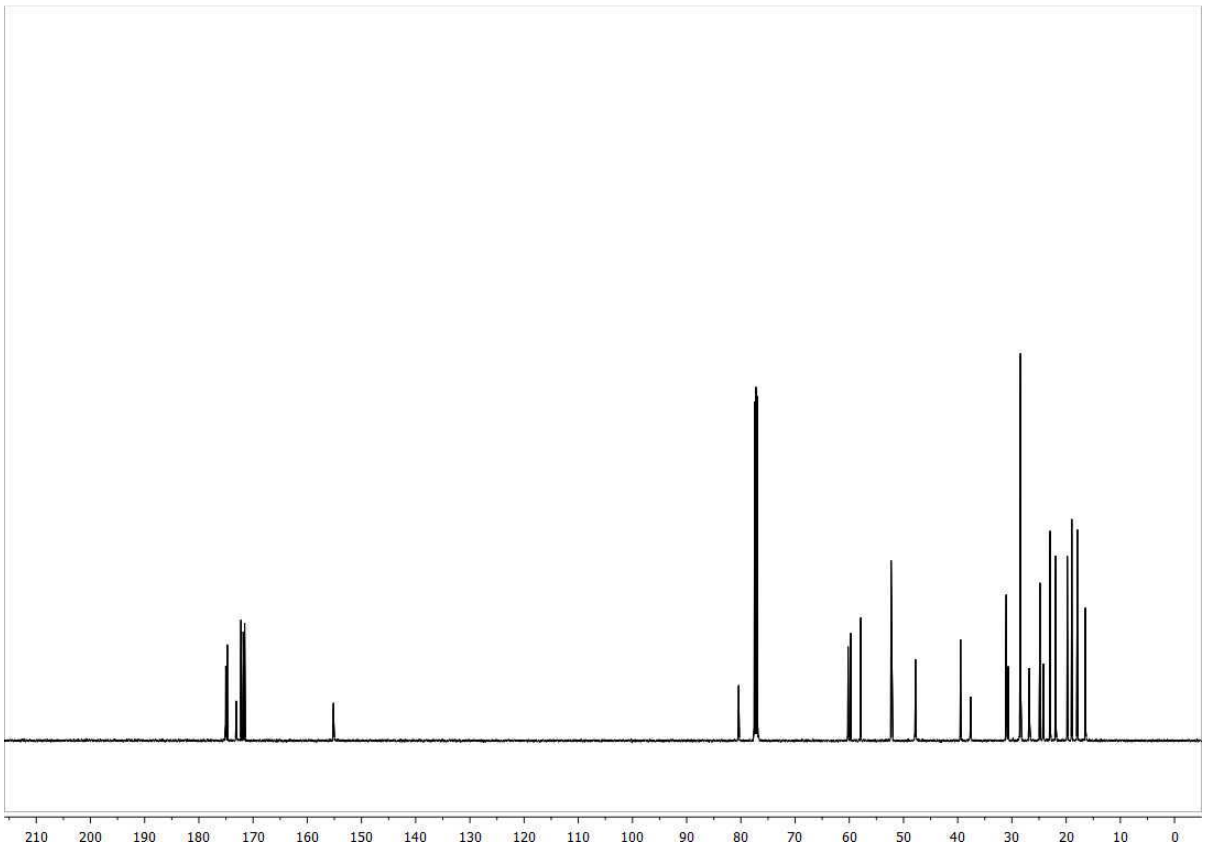
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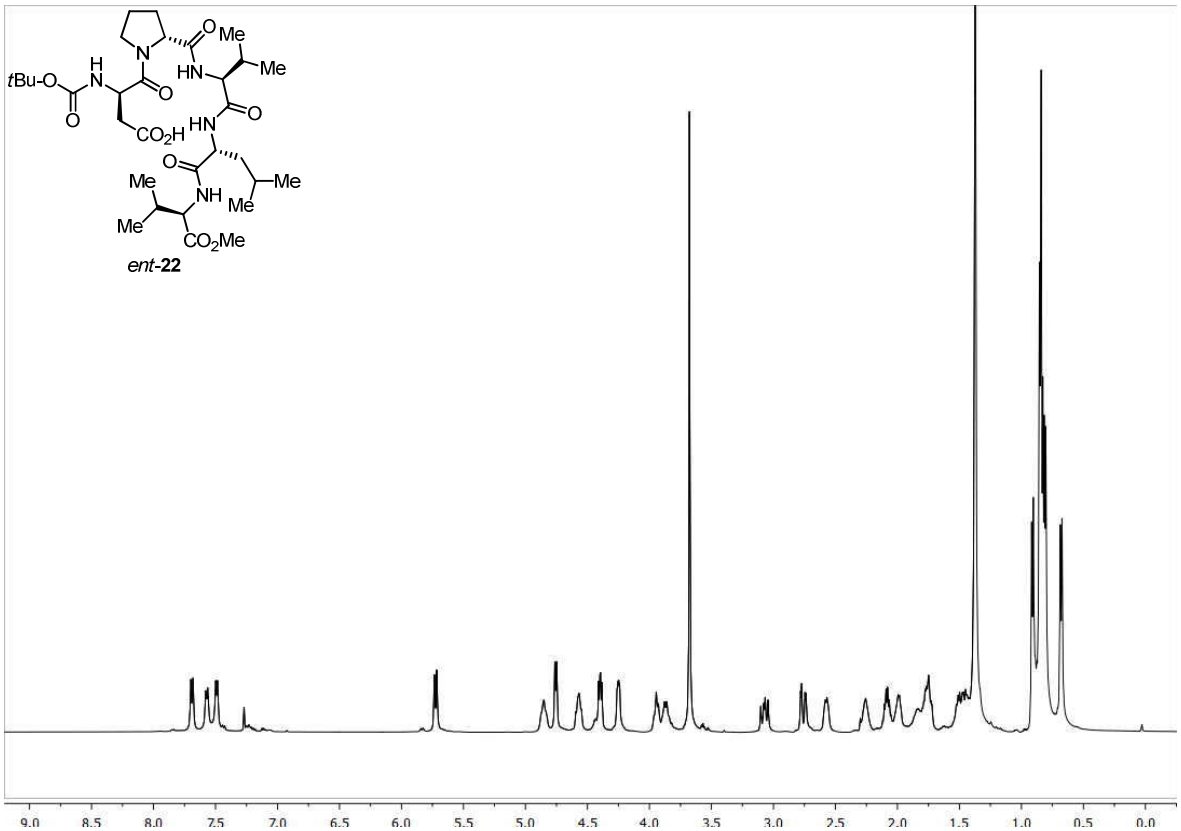
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H
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CO2H
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CO2Me
ent-22
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8.5
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7.5
7.0
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6.0
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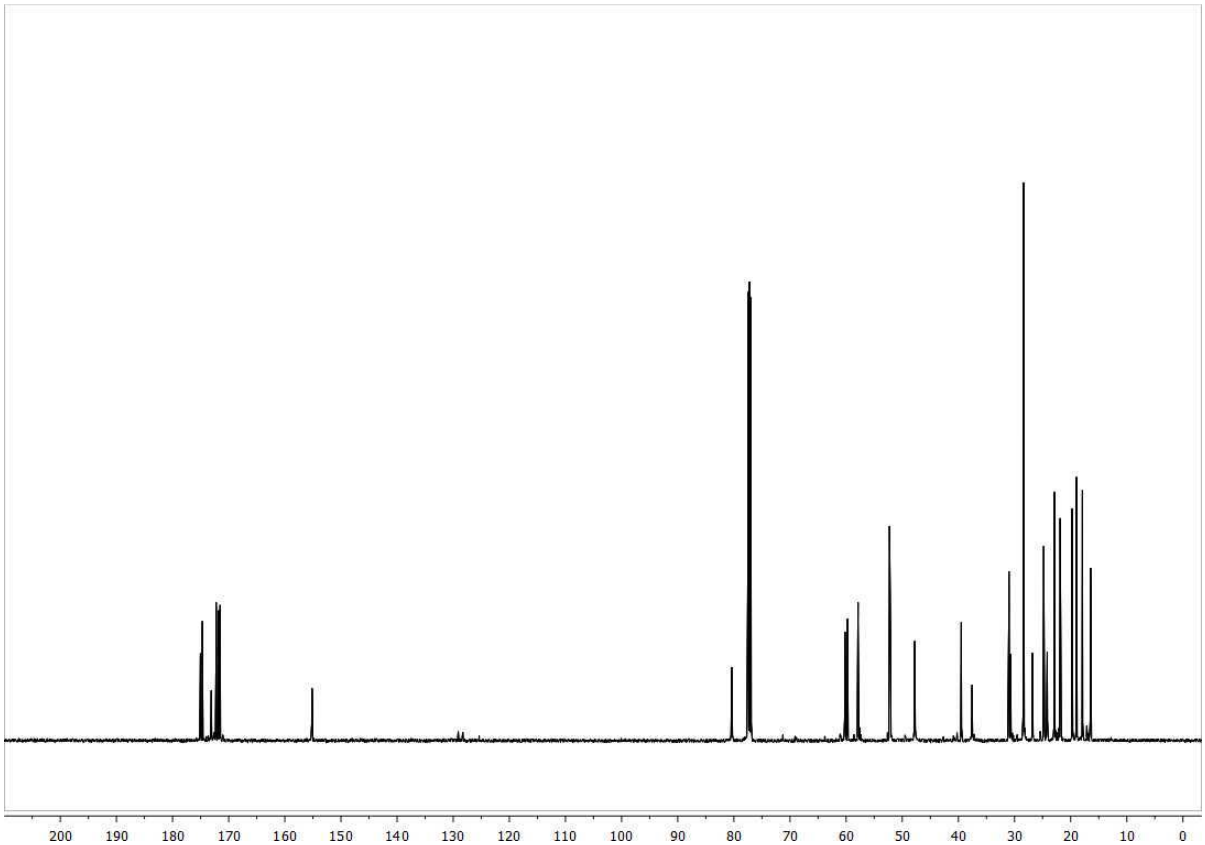
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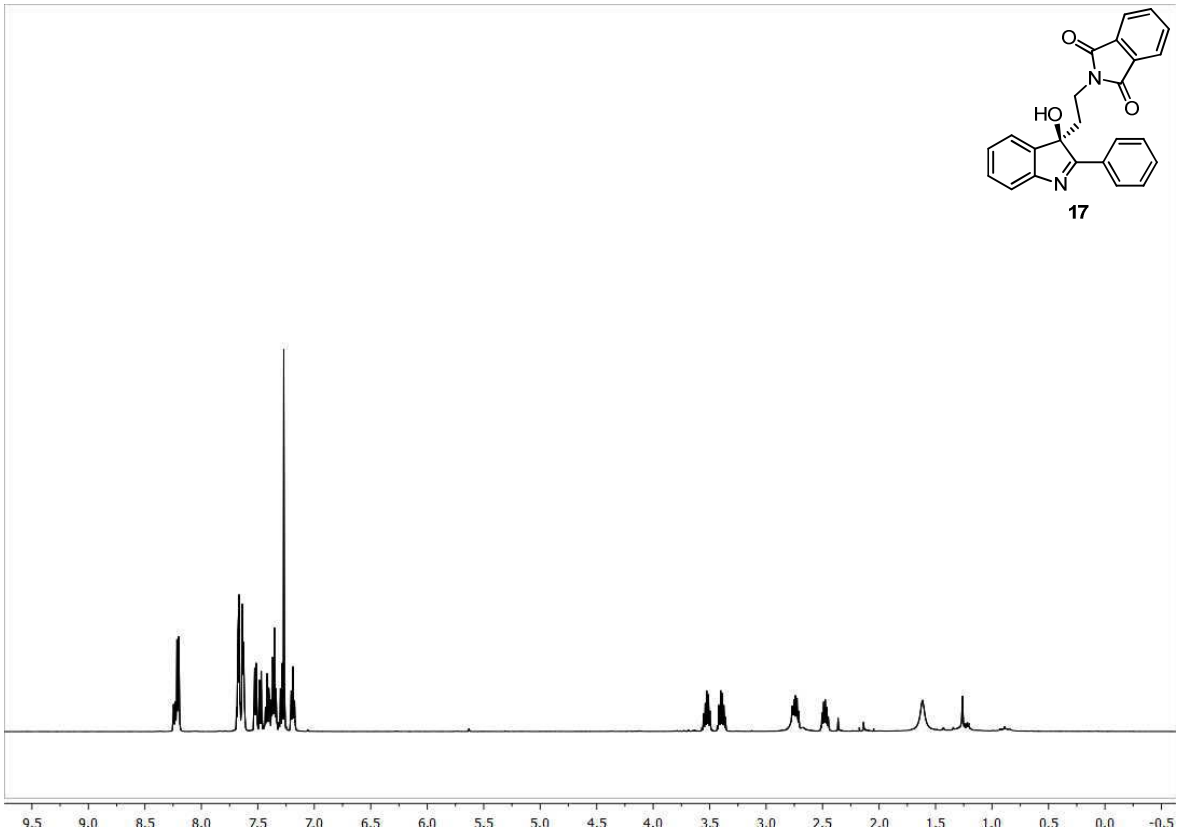
10
0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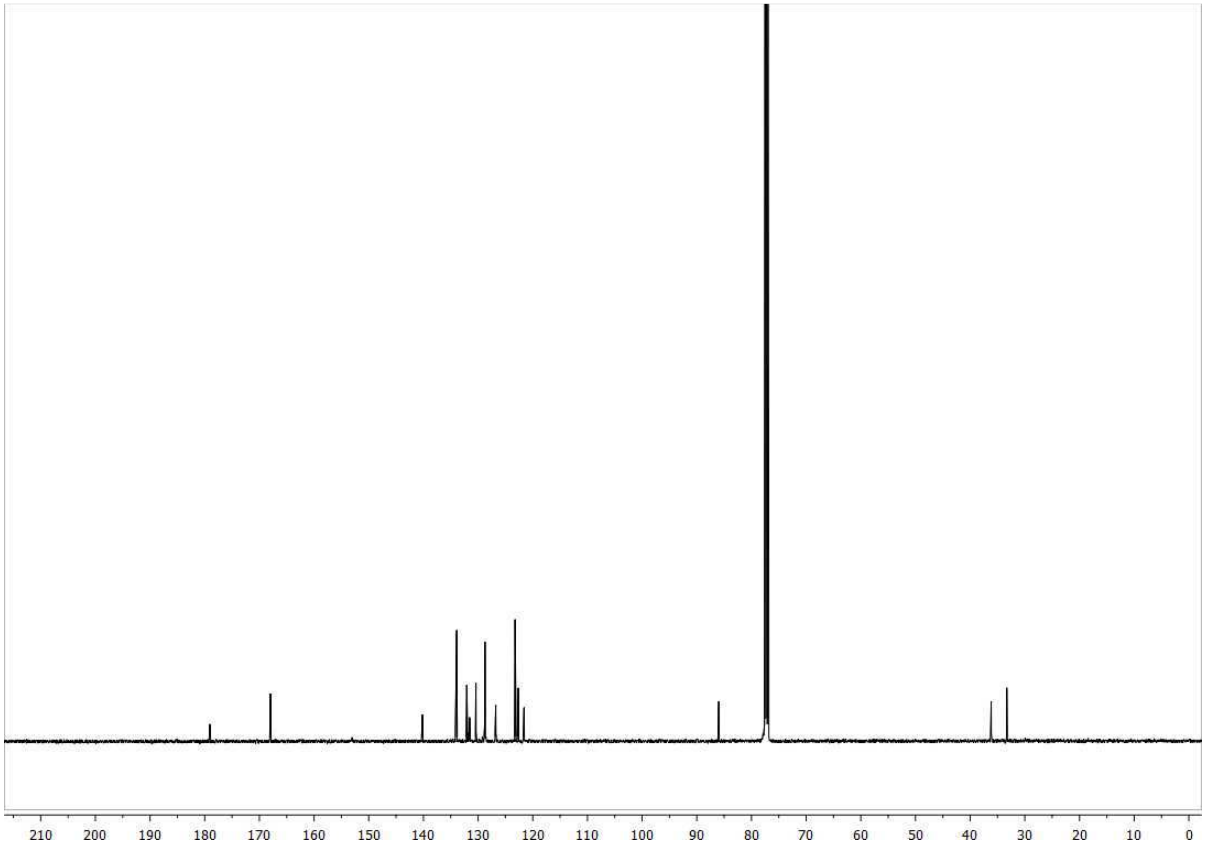
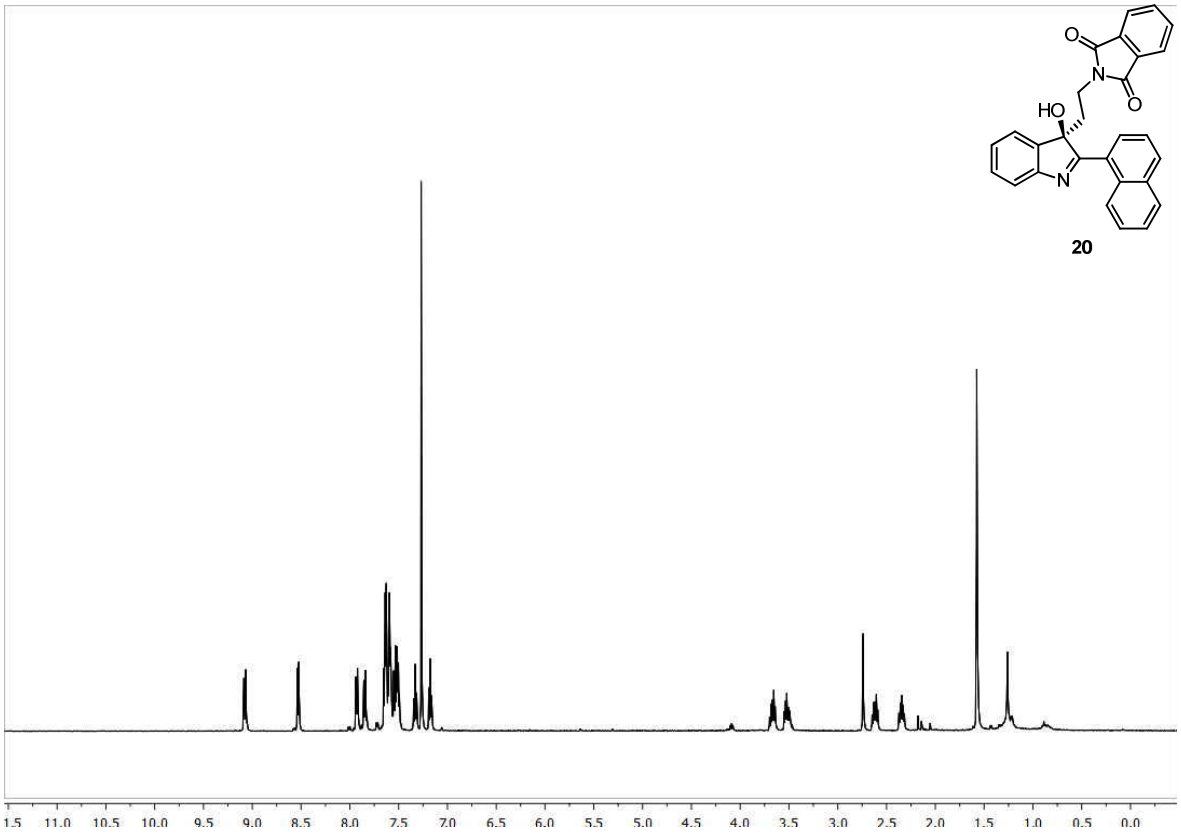
O
O
N
17





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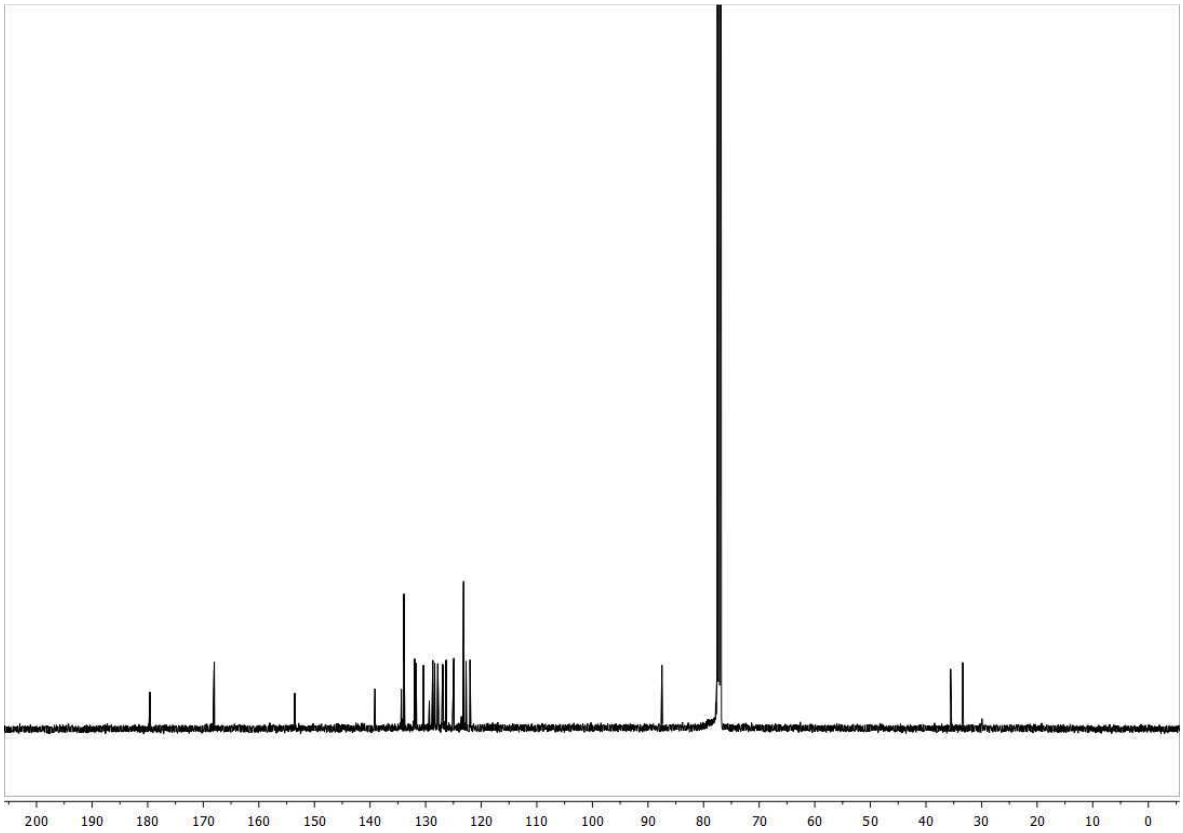

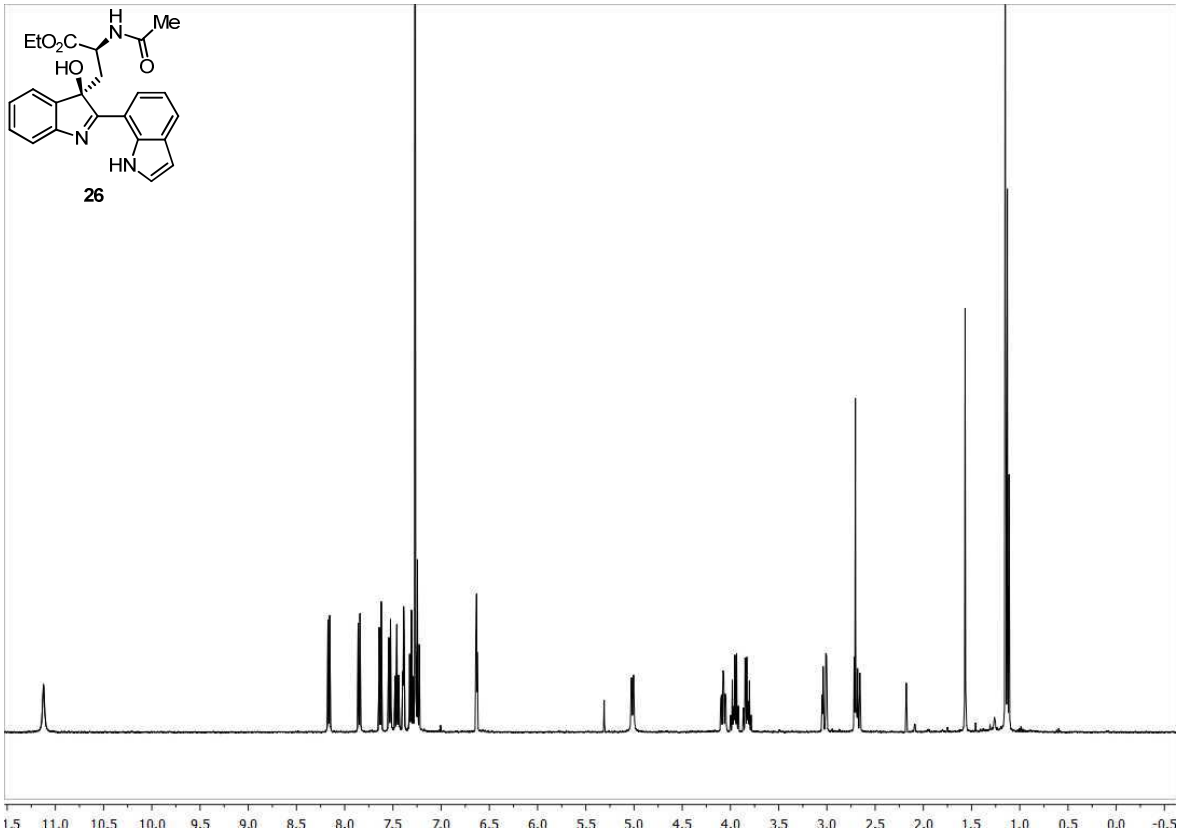
EtO2C
HO
H
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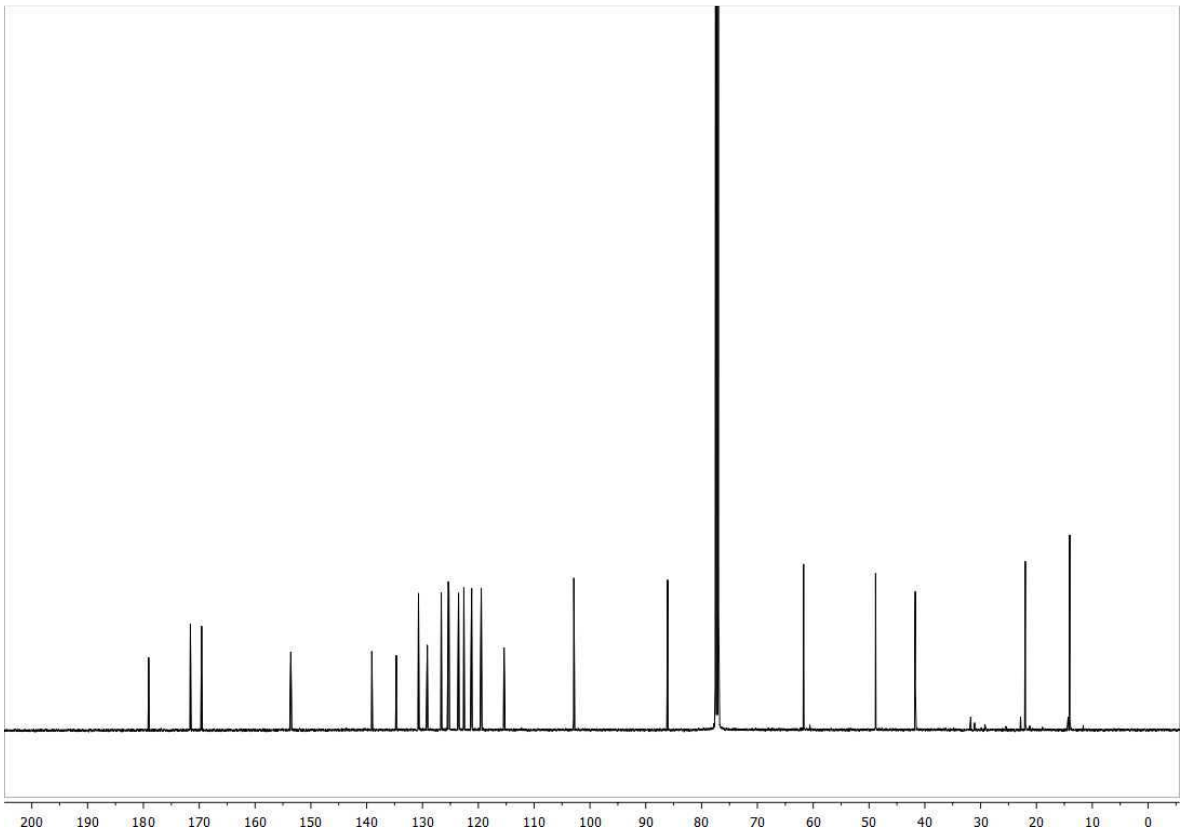
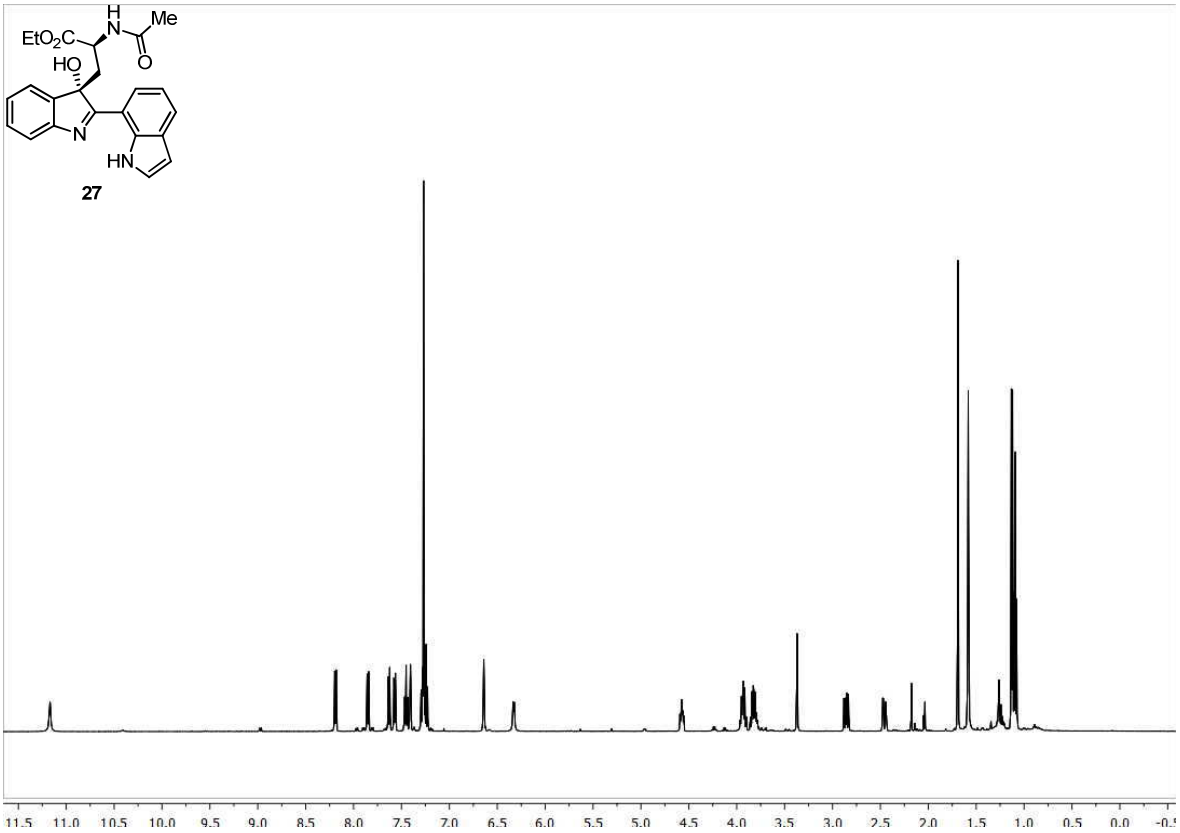
Me
O
N
HN
26

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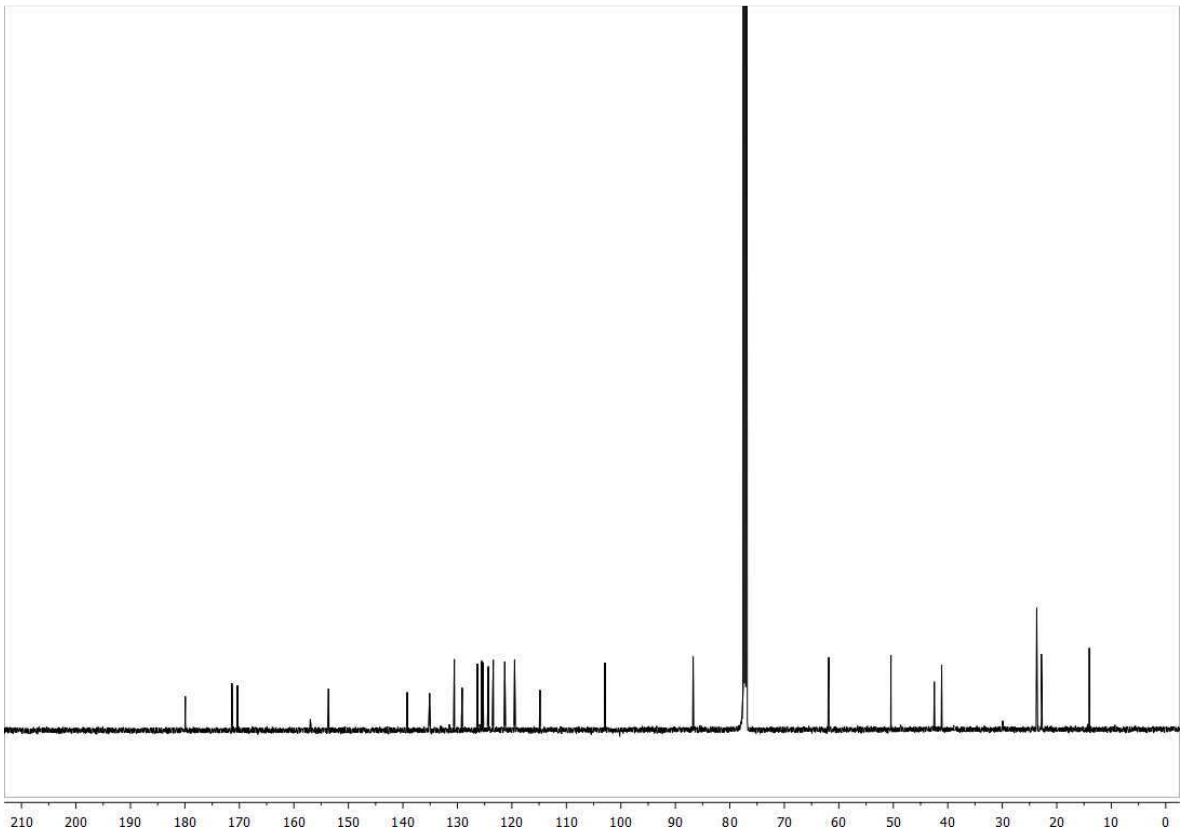
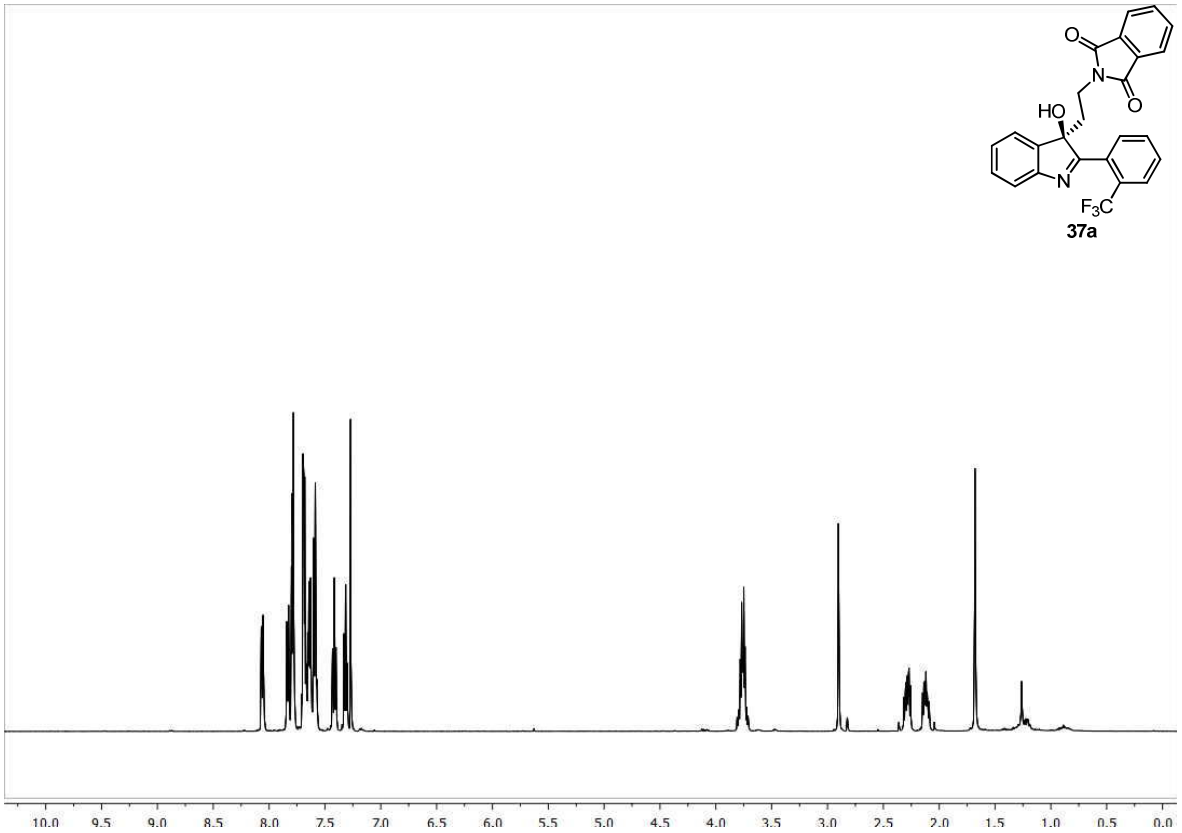
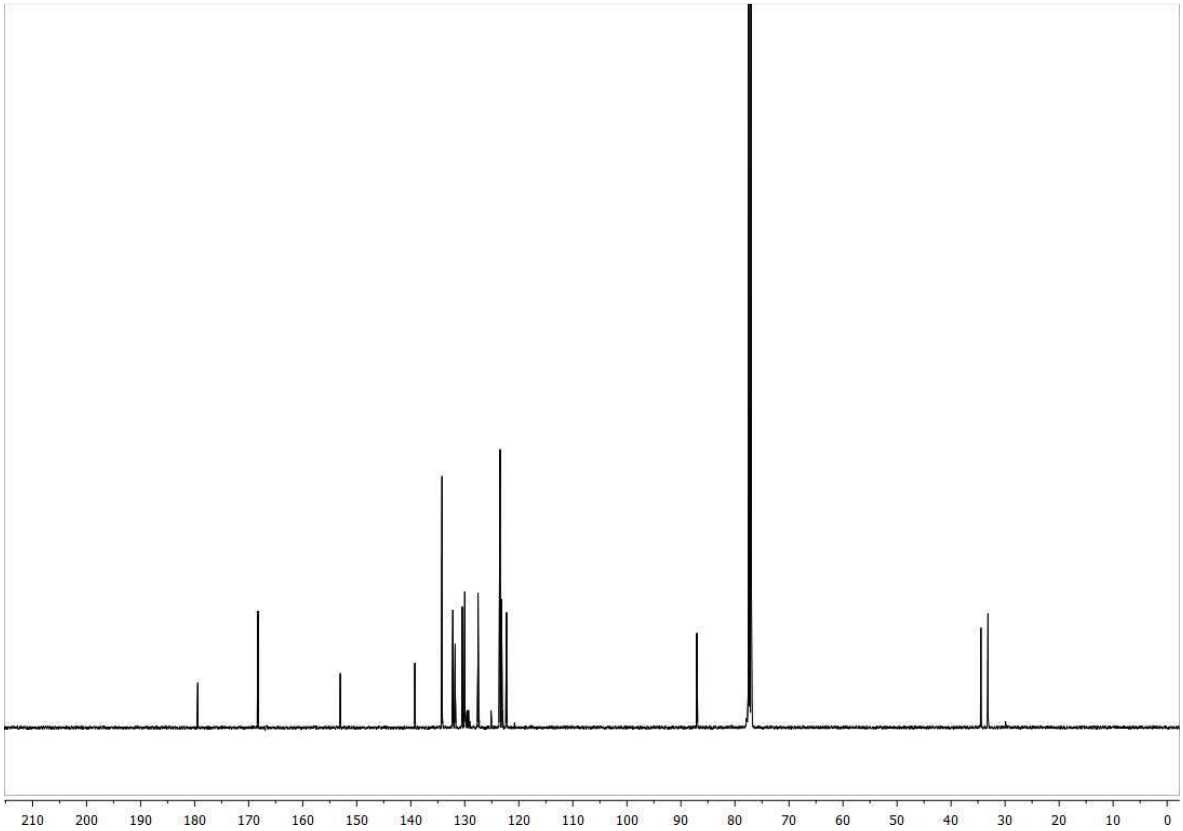
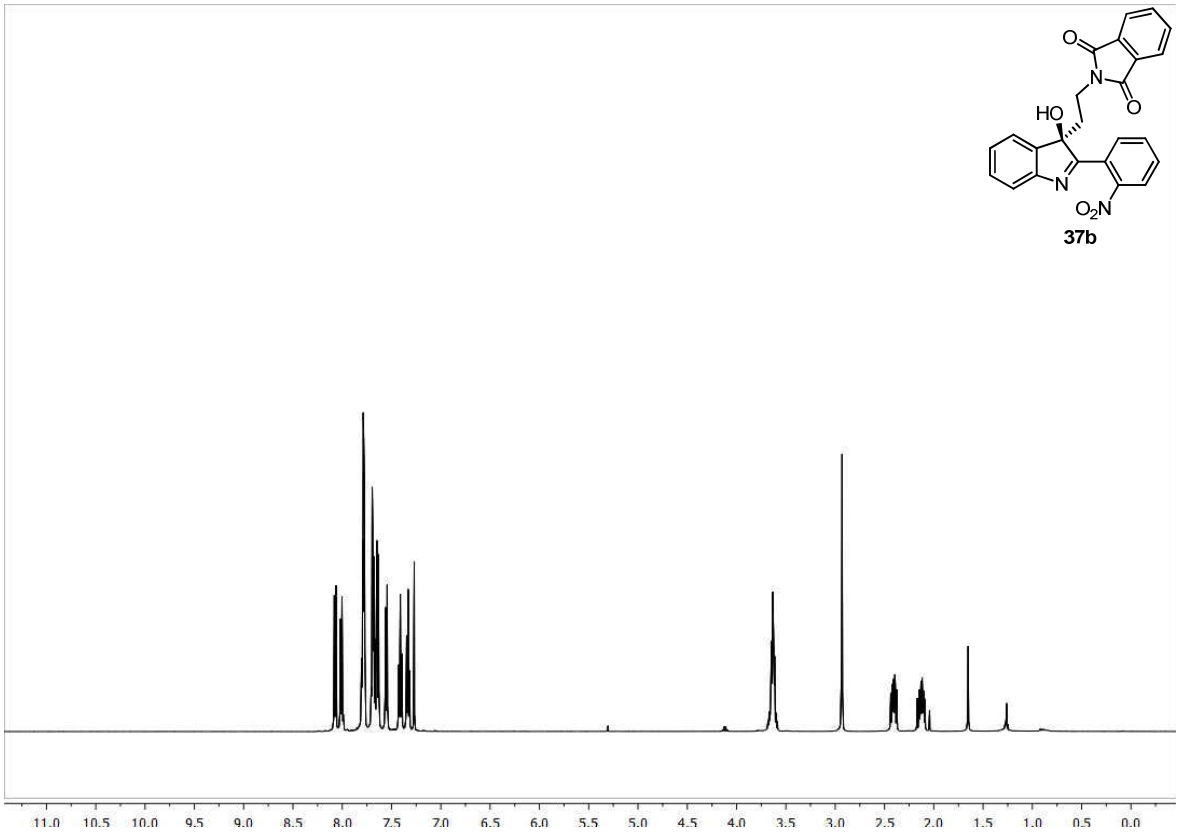
HO
H
N
Me
O
N
HN
27





37a





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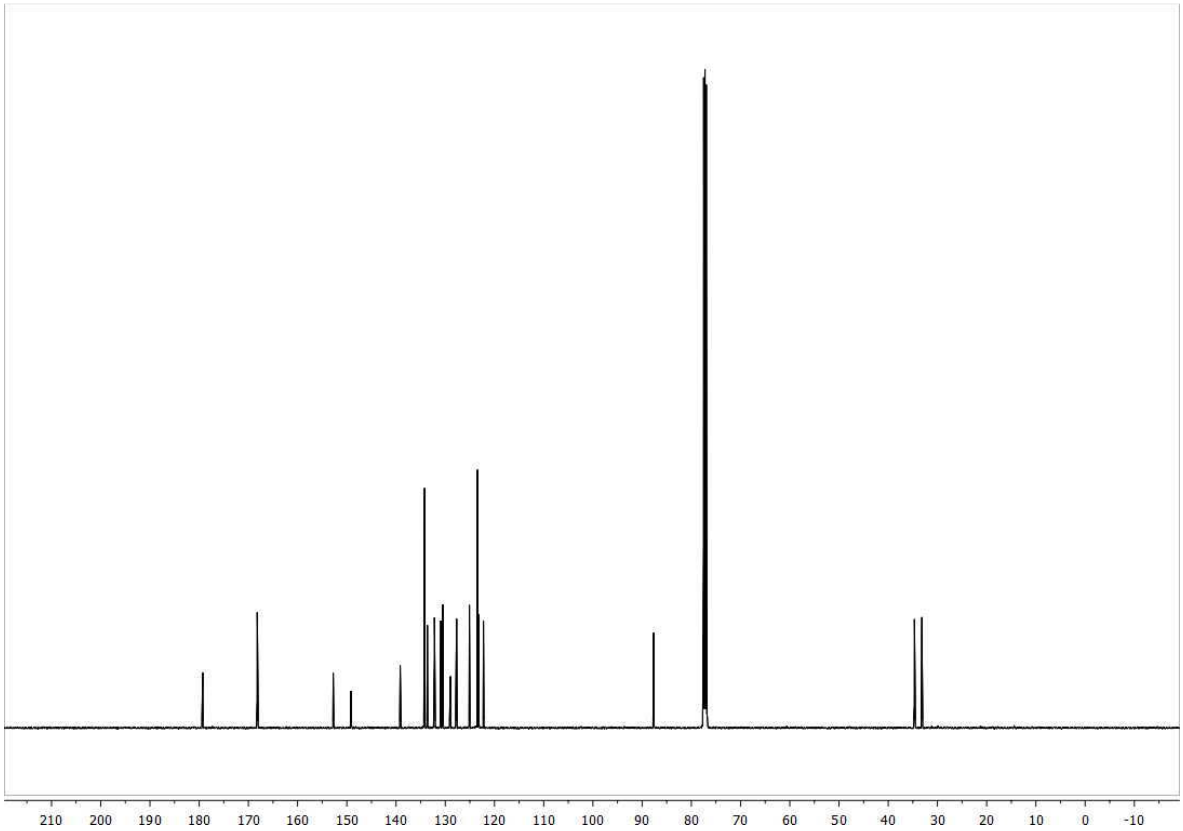
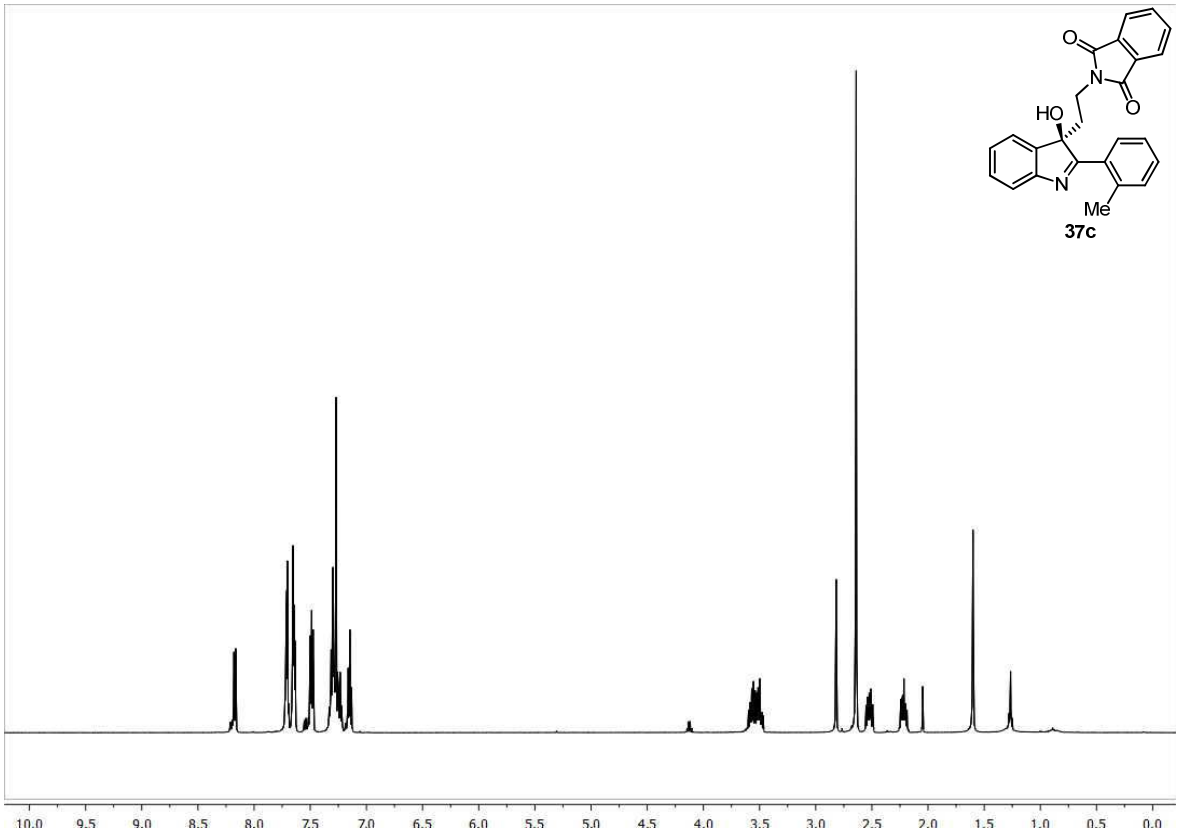
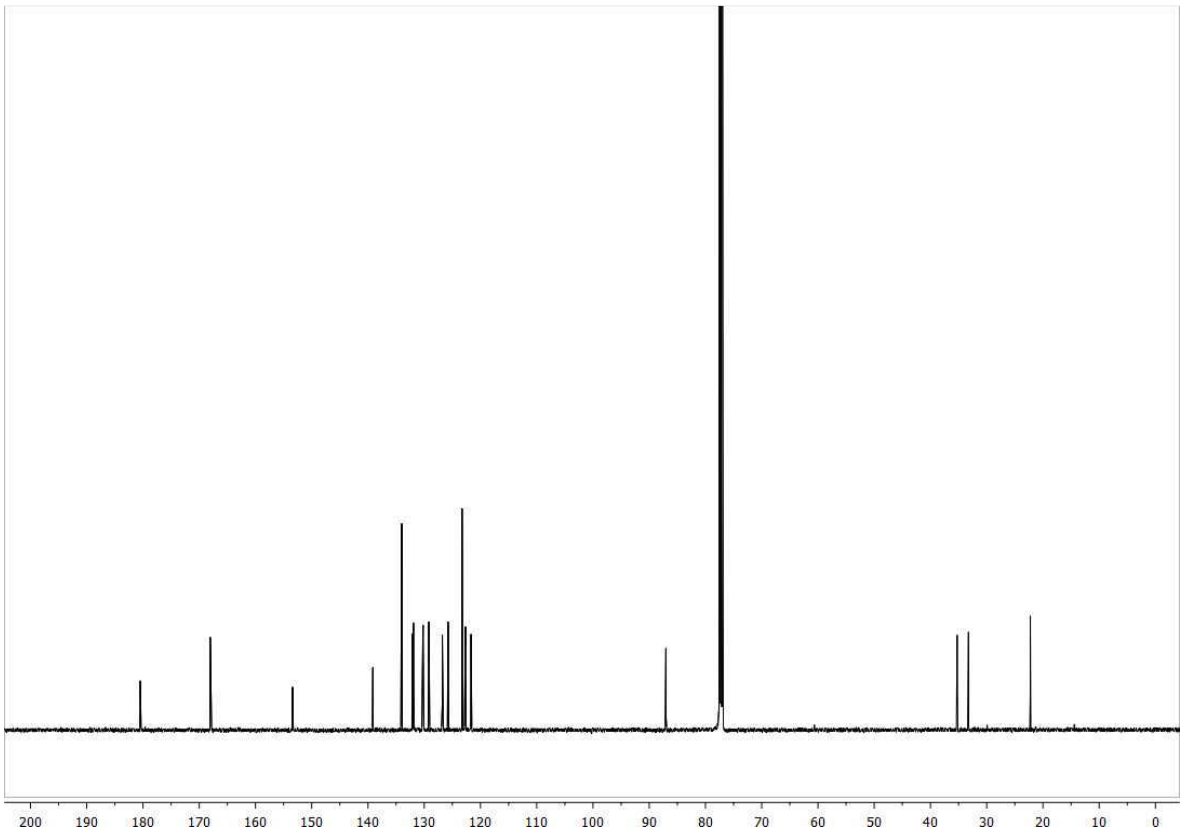



O
N
O
HO
N
Me
37c
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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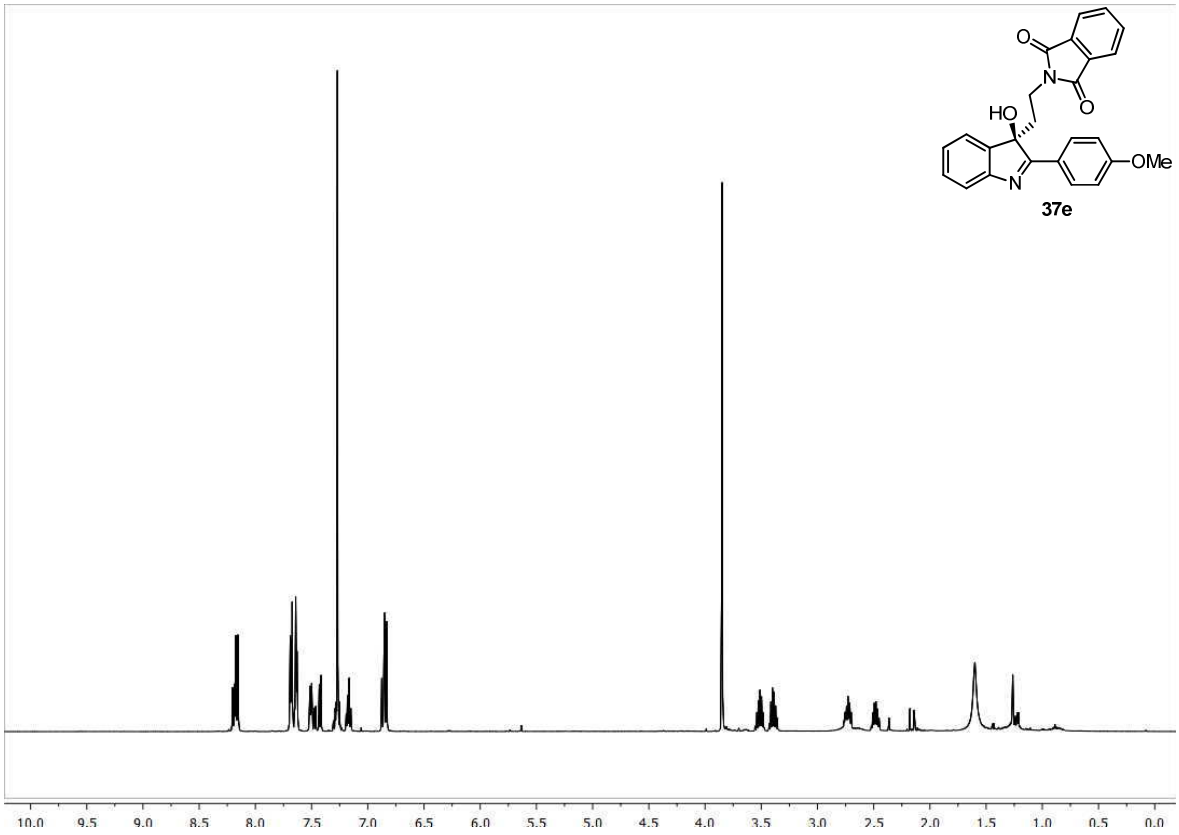
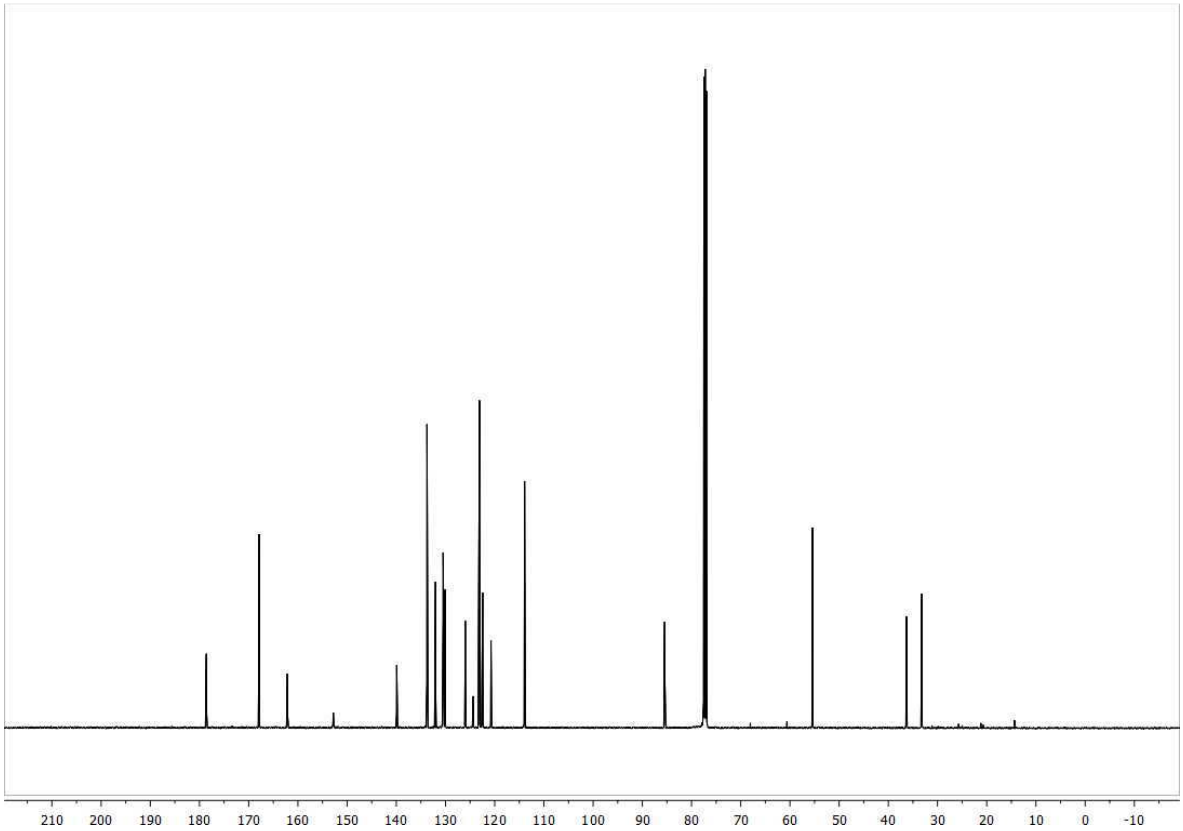

O
N
O
HO
OMe
N
37e
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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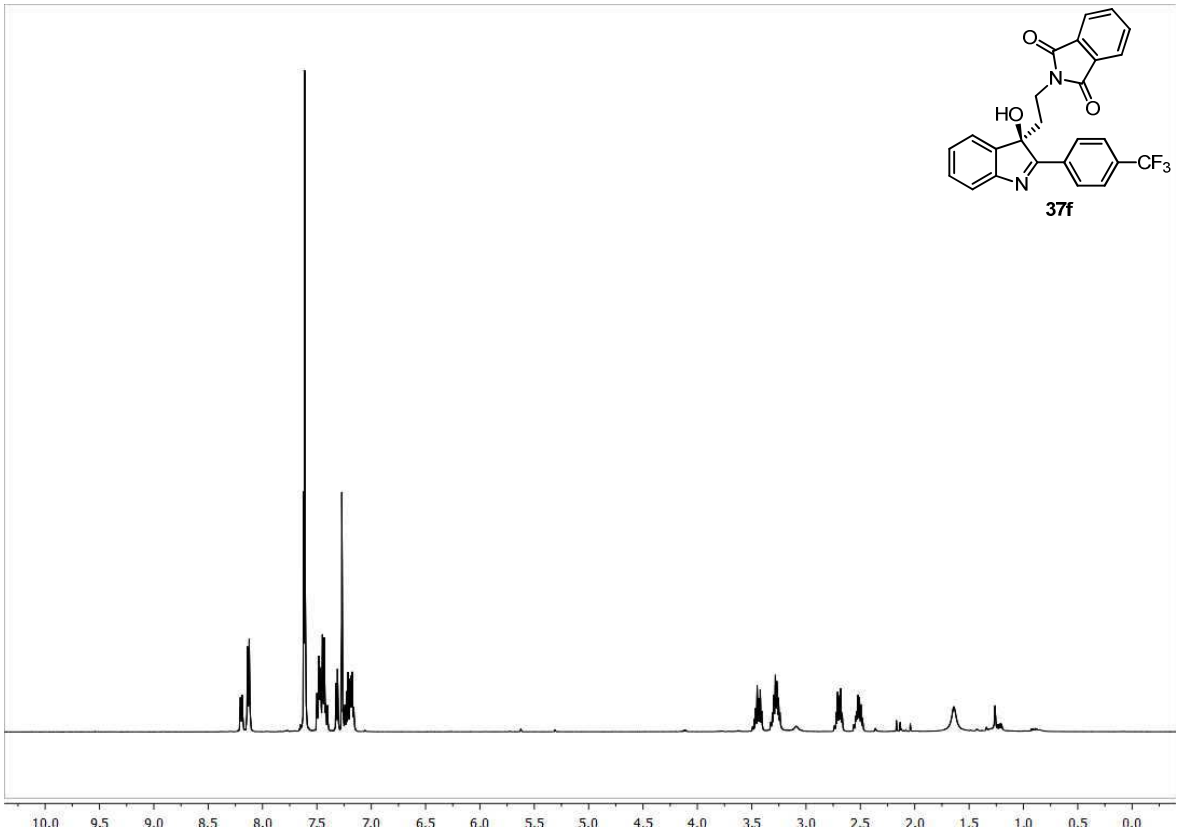
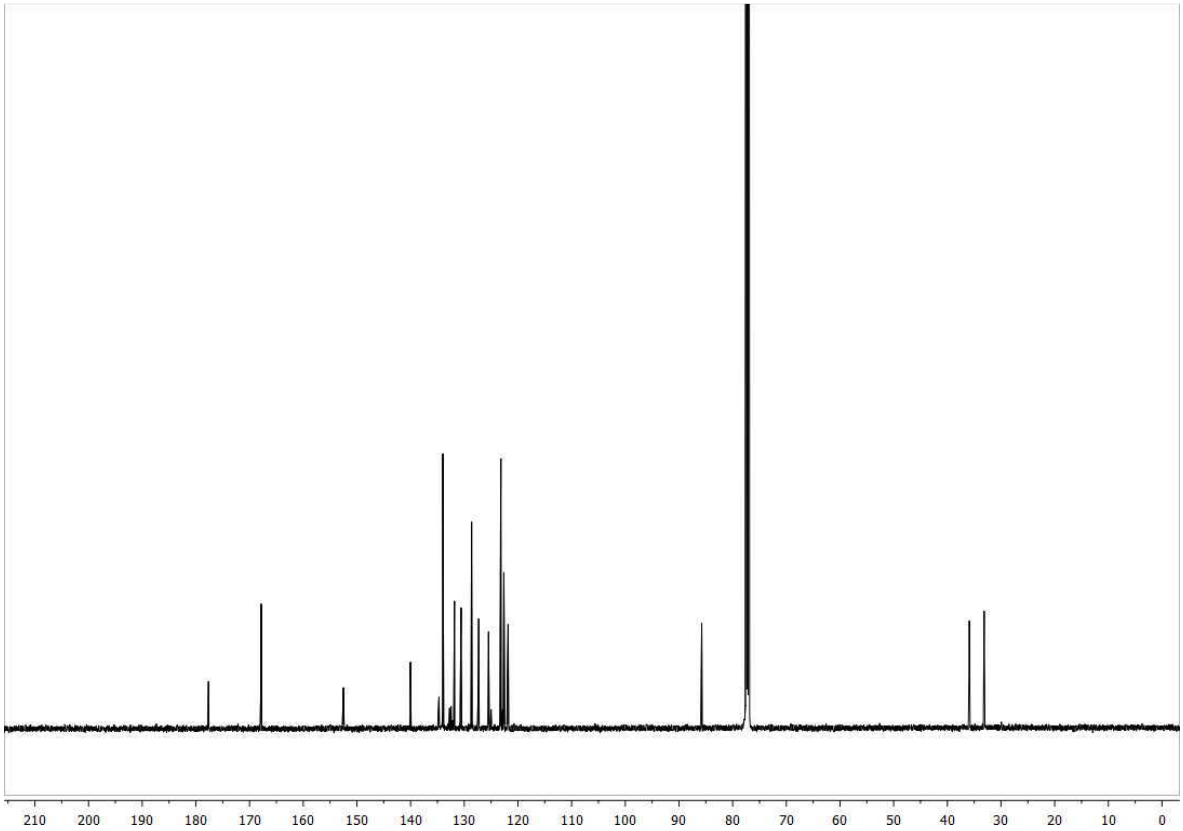
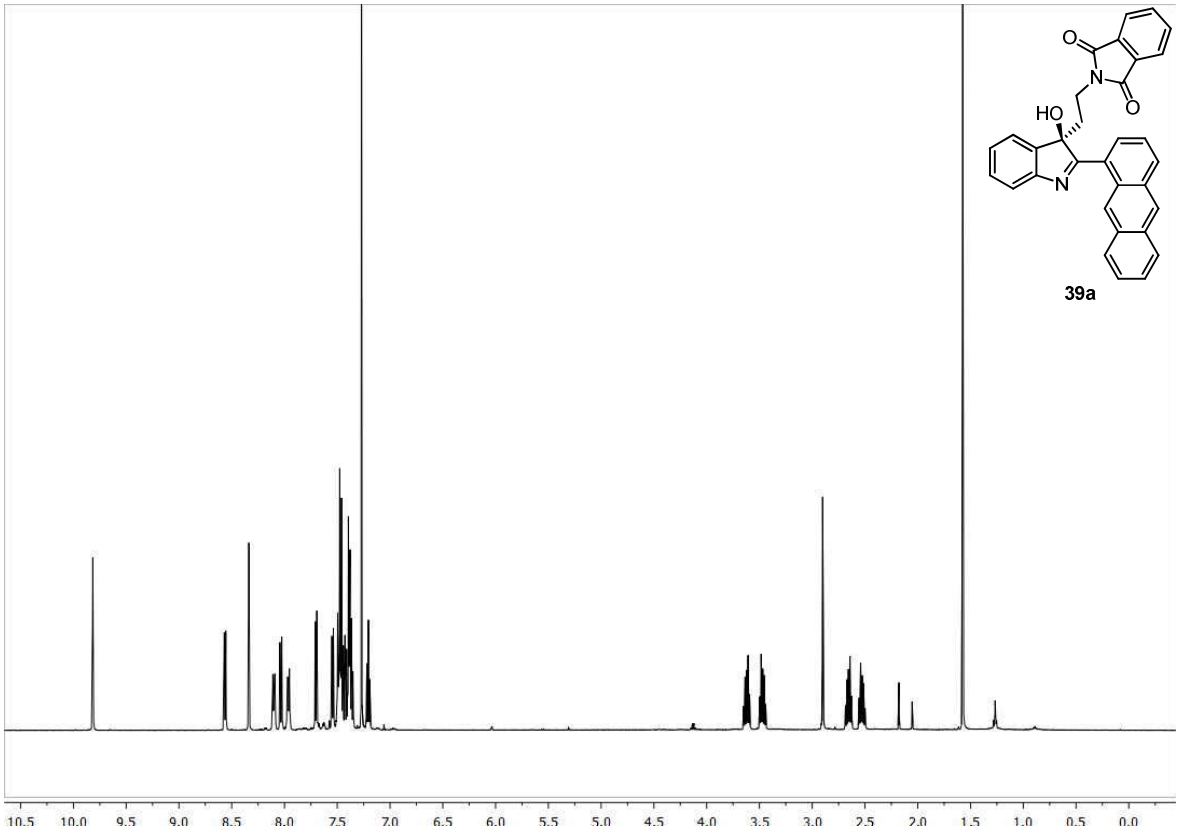
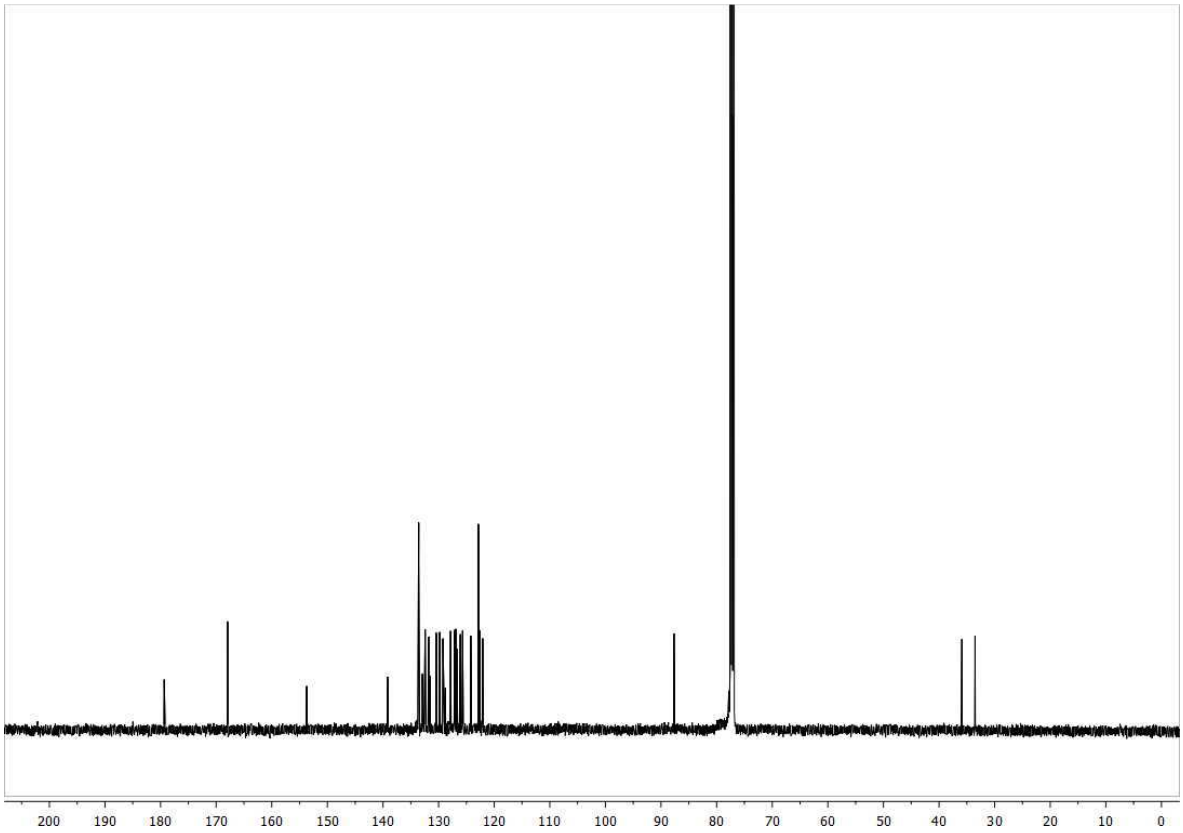
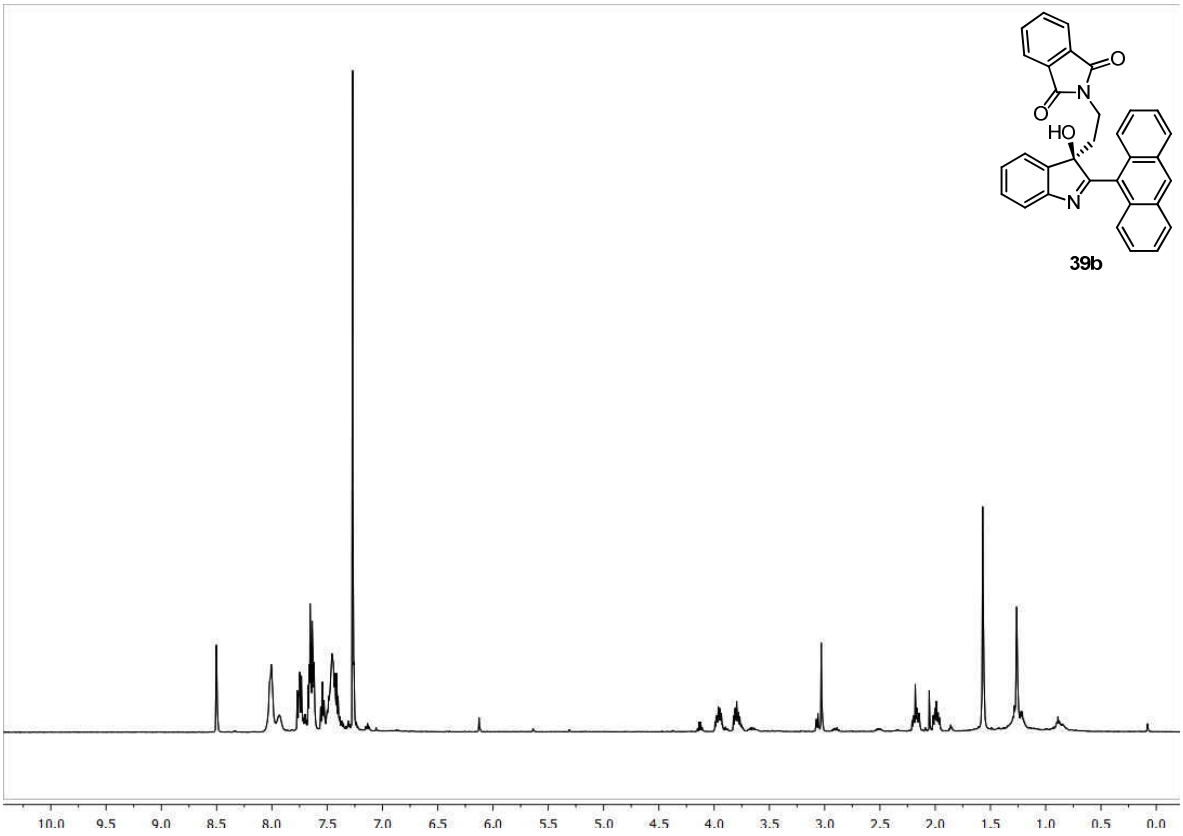


37f




O
N
O
HO
N
39a





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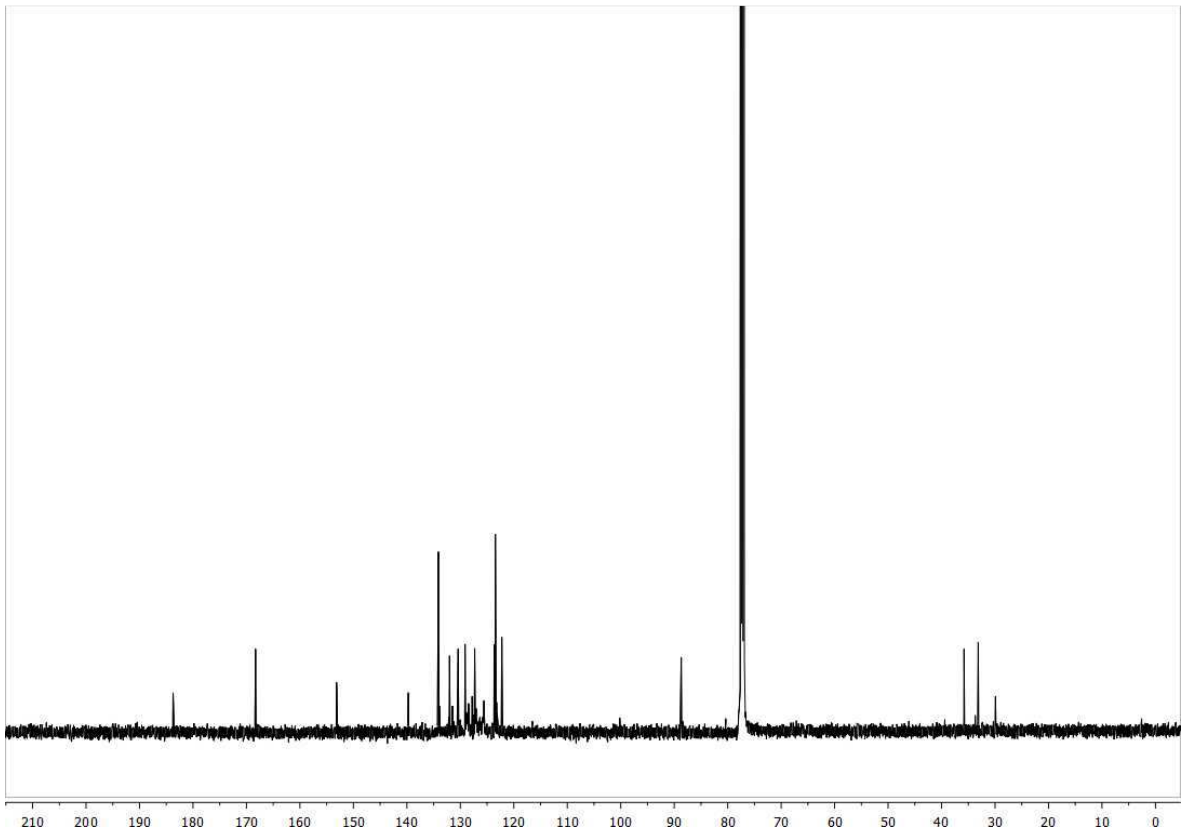
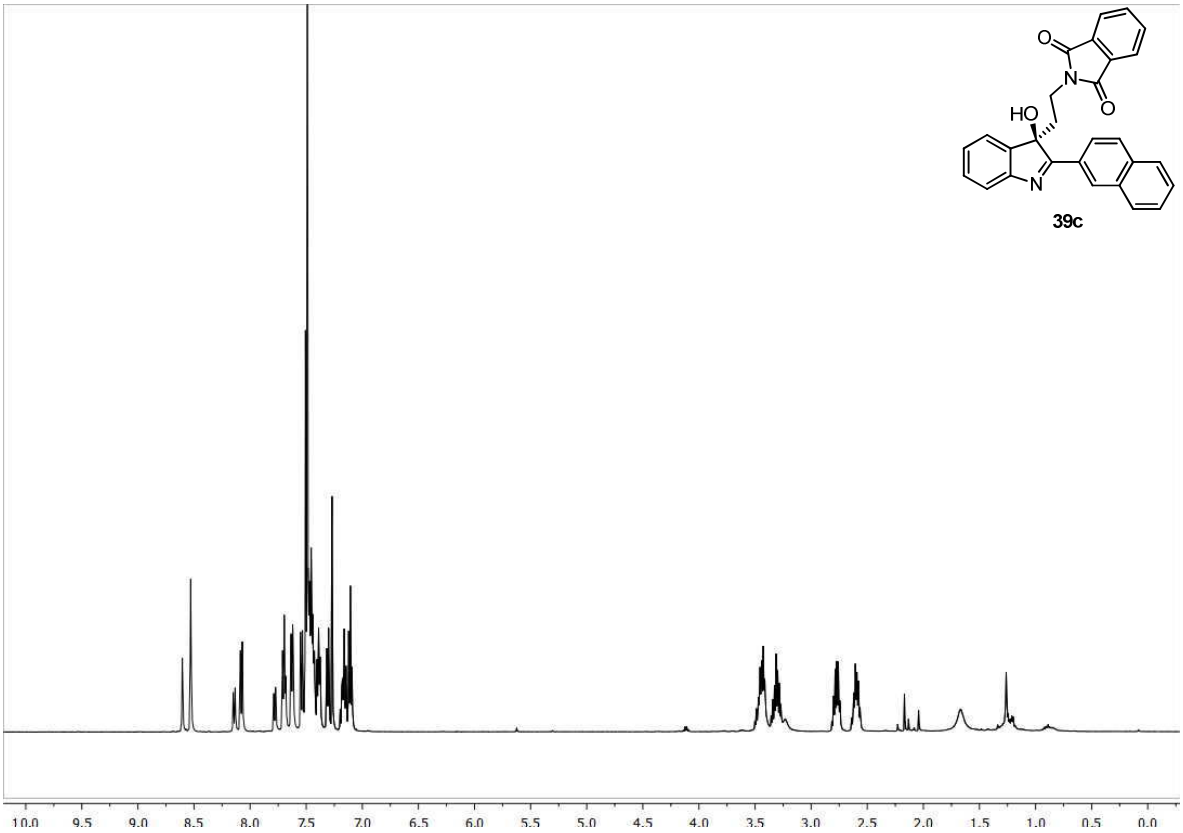
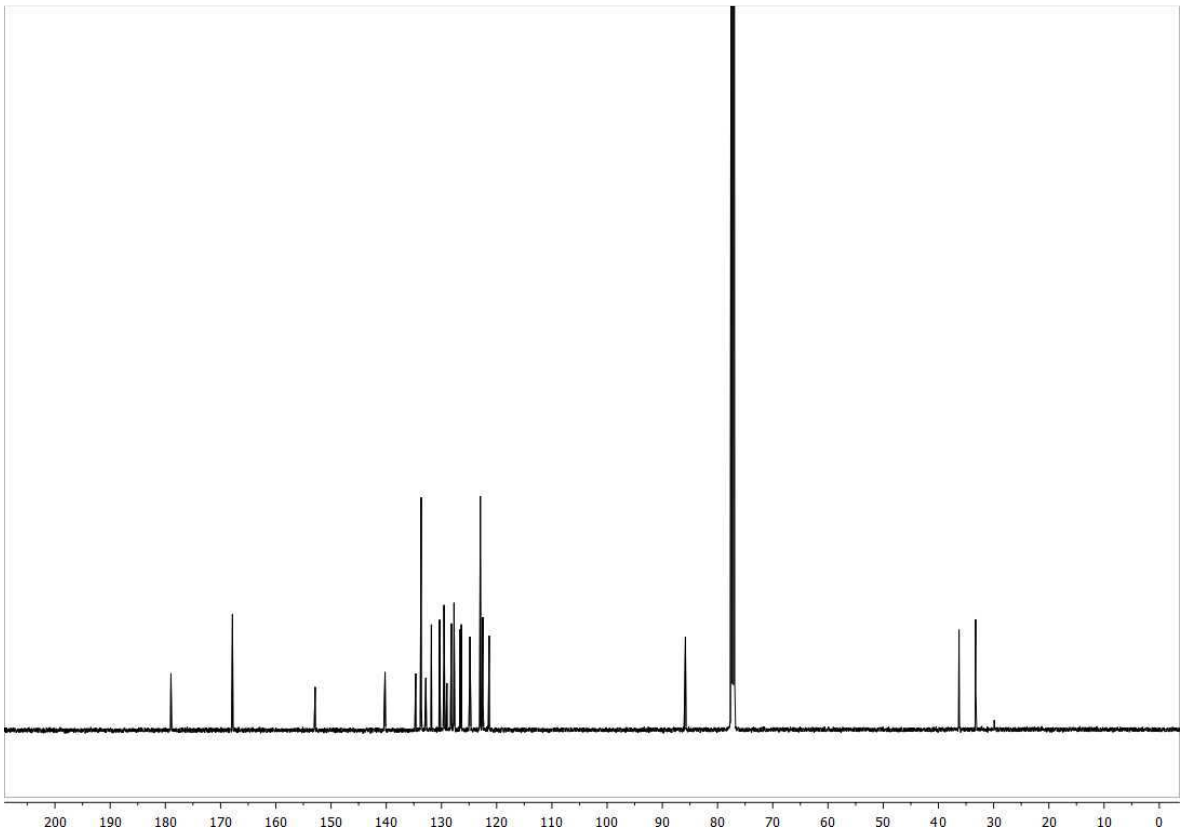



O
N
O
HO
N
39c
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0


200
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70
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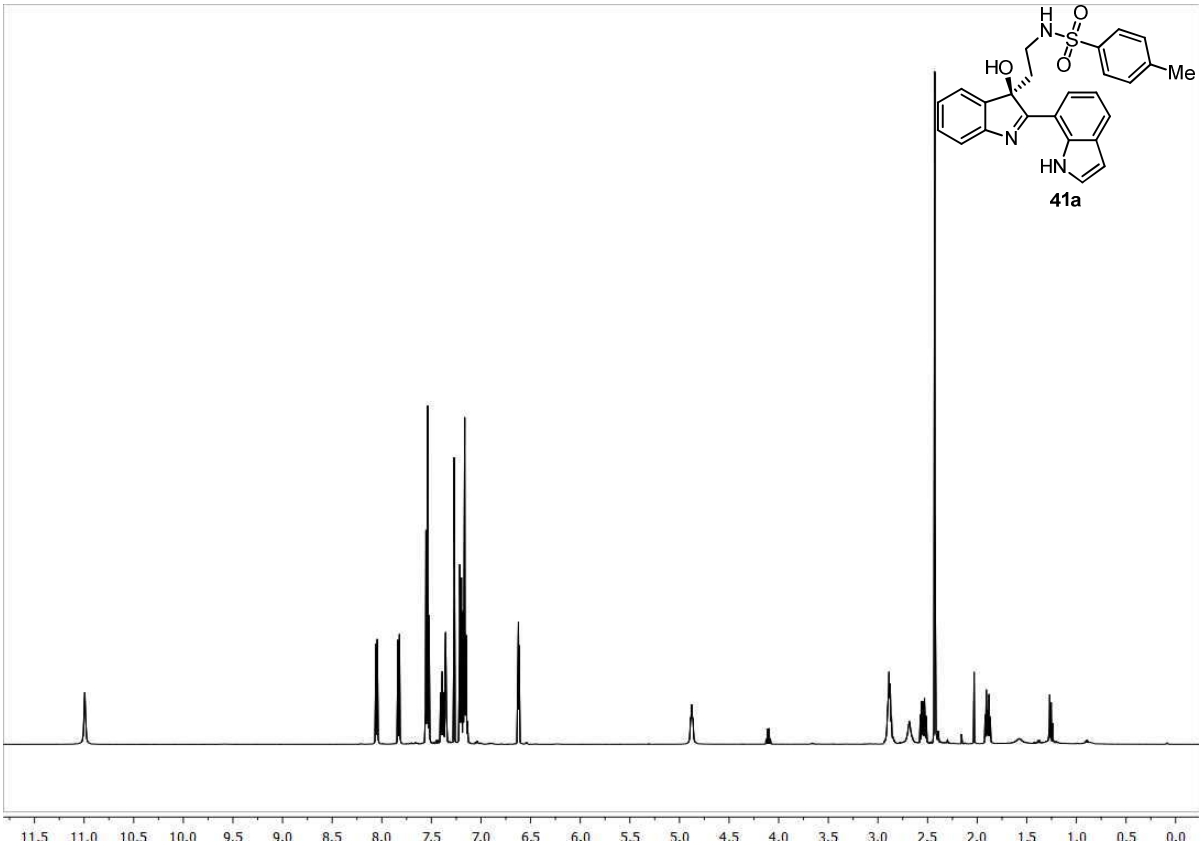
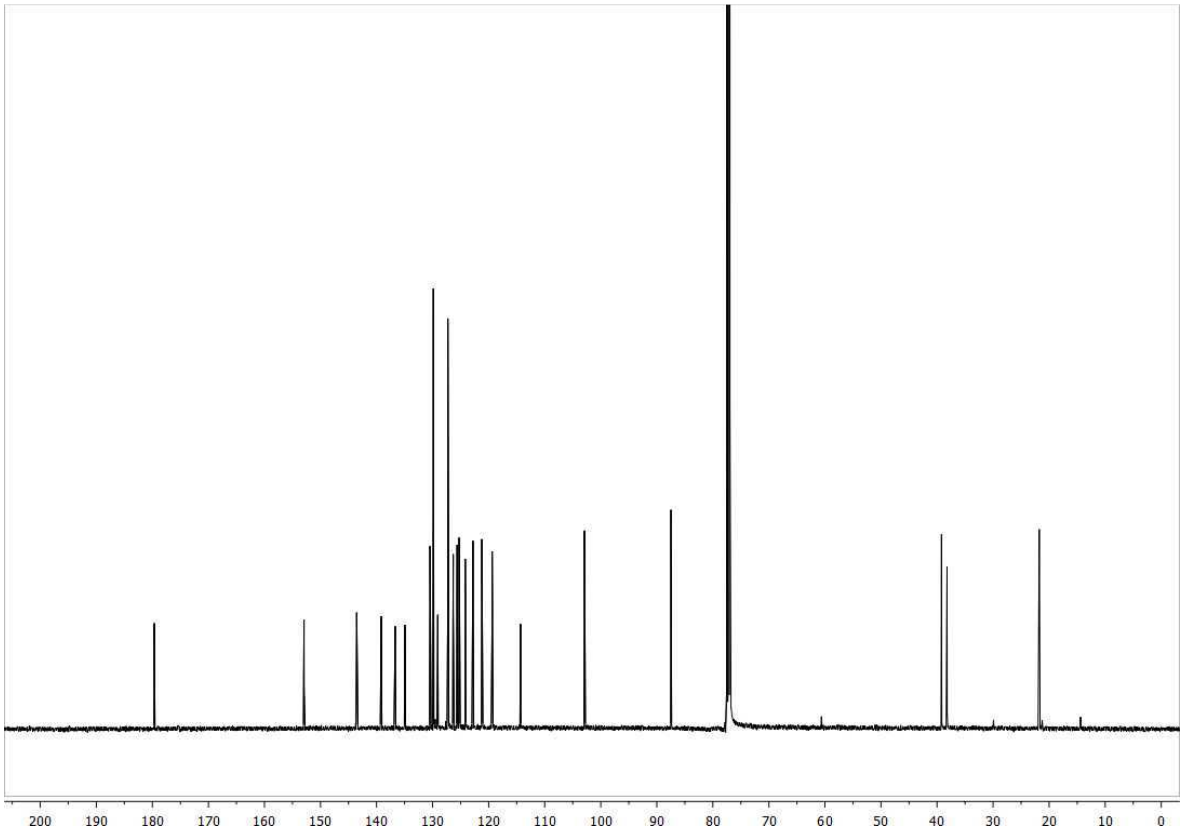
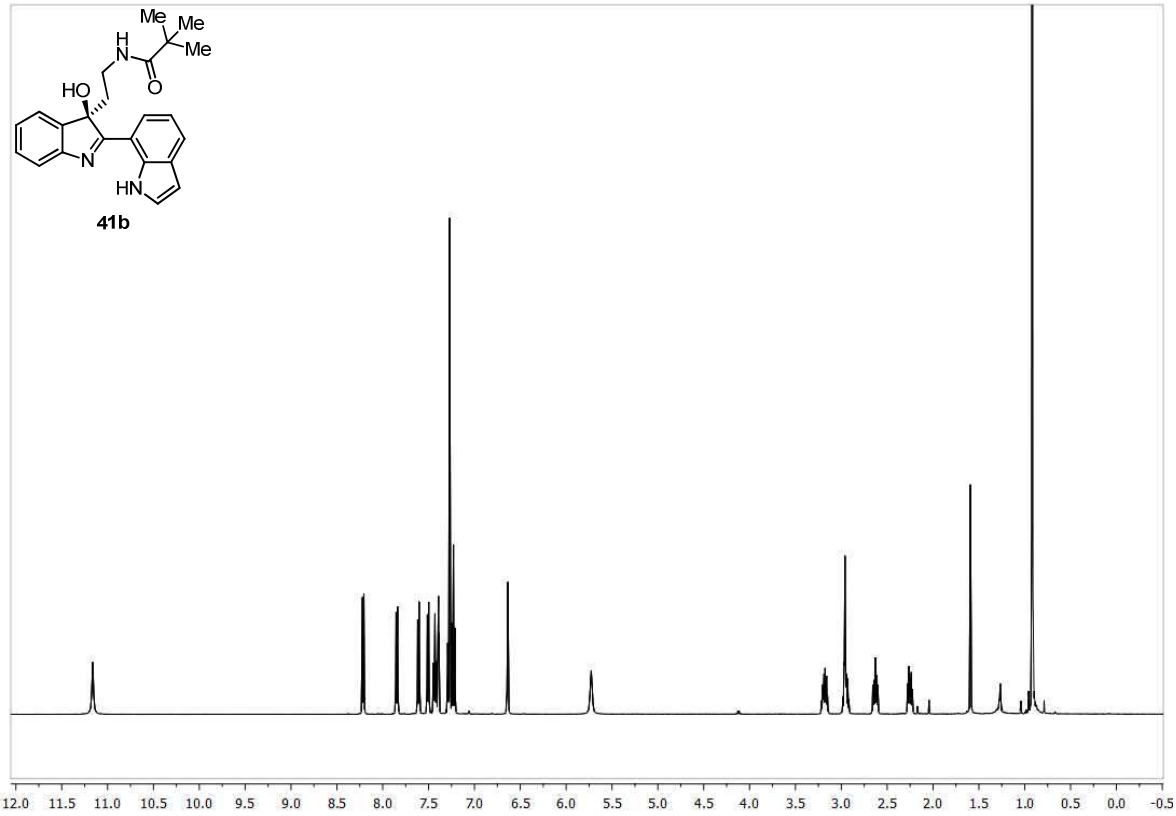
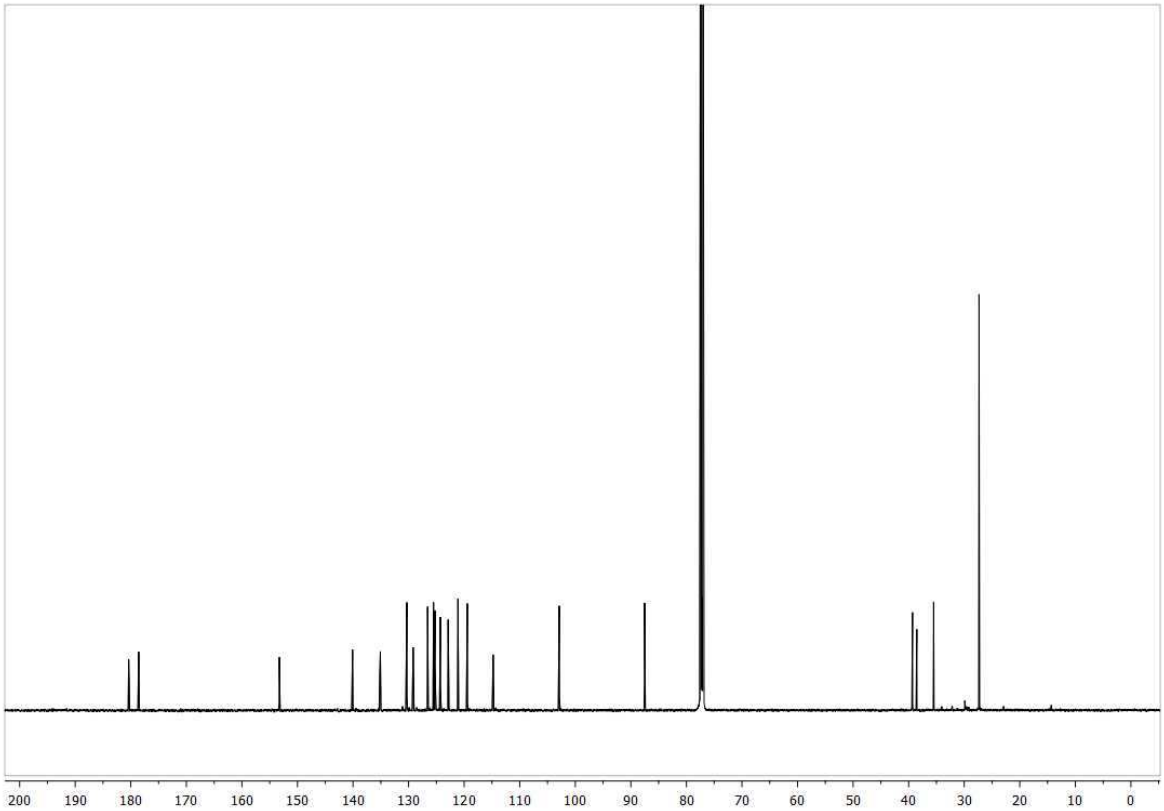
10
0


HO
N
H
N
O
S
O
Me
HN
41a




Me
Me
Me
H
N
O
HO
N
HN
41b
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 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 -0.5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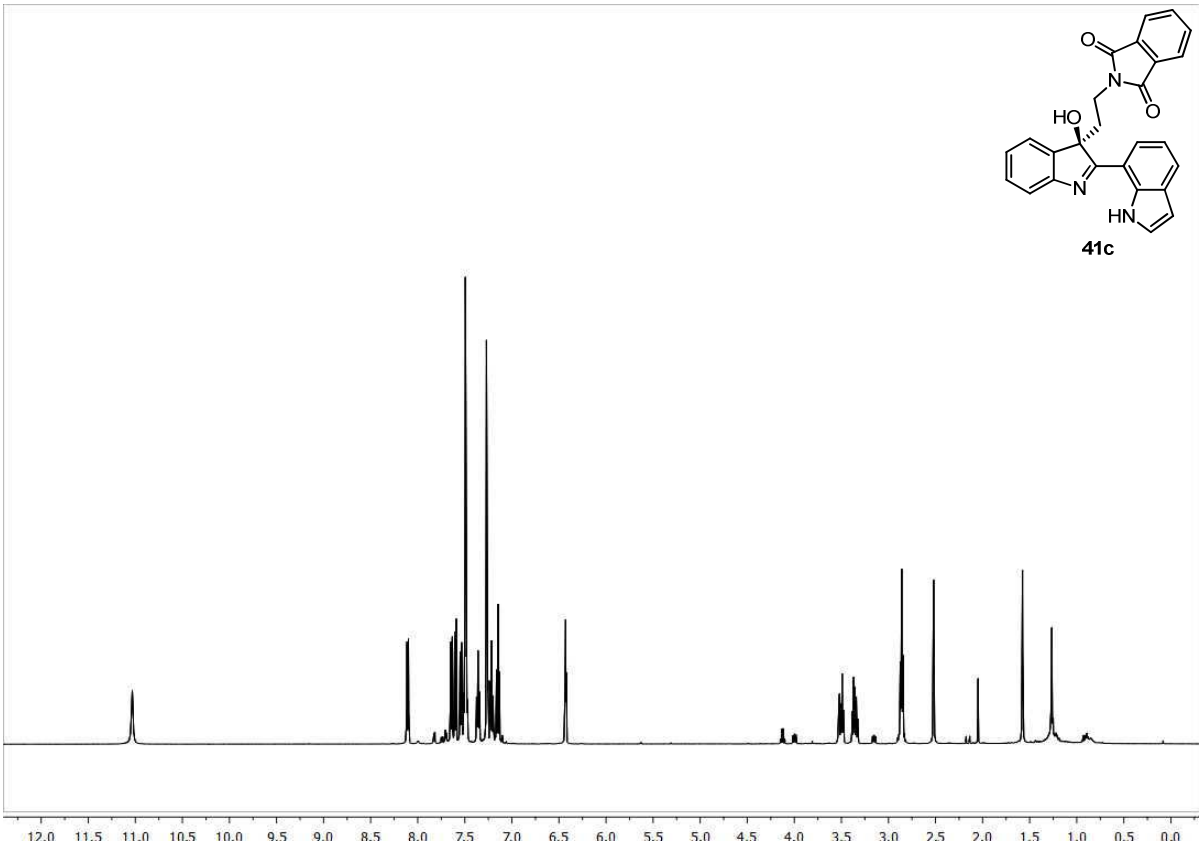
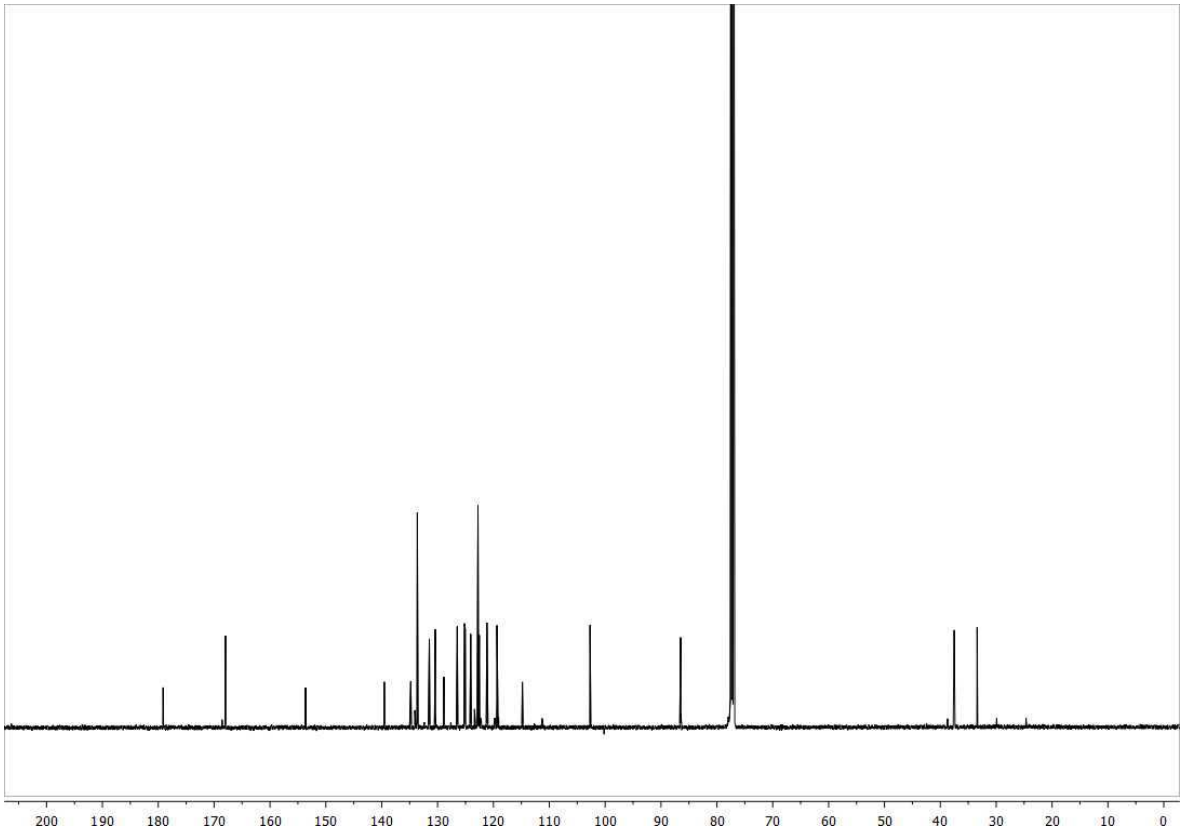
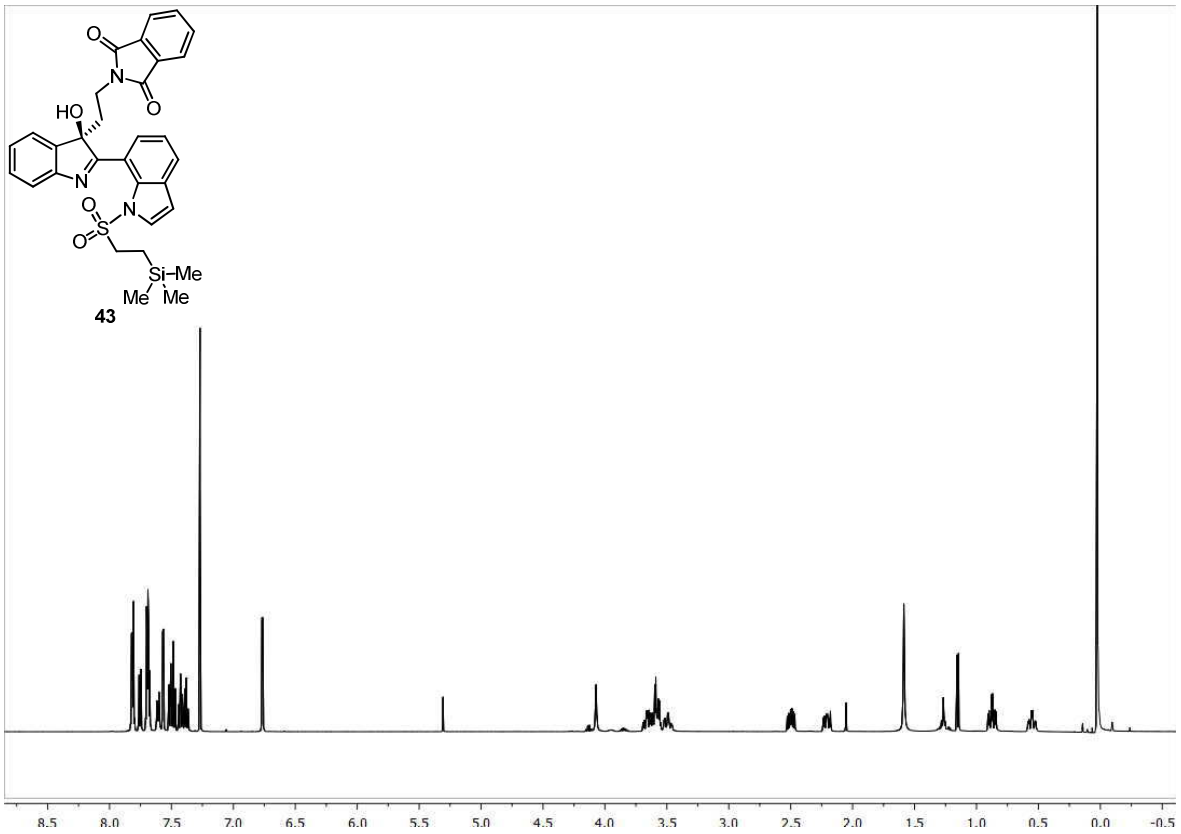

O
N
O
HO
N
HN
41c




HO
N
O
O
N
O
O
S
O
Si–Me
Me
Me
43
8.5
8.0
7.5
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6.5
6.0
5.5
5.0
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4.0
3.5
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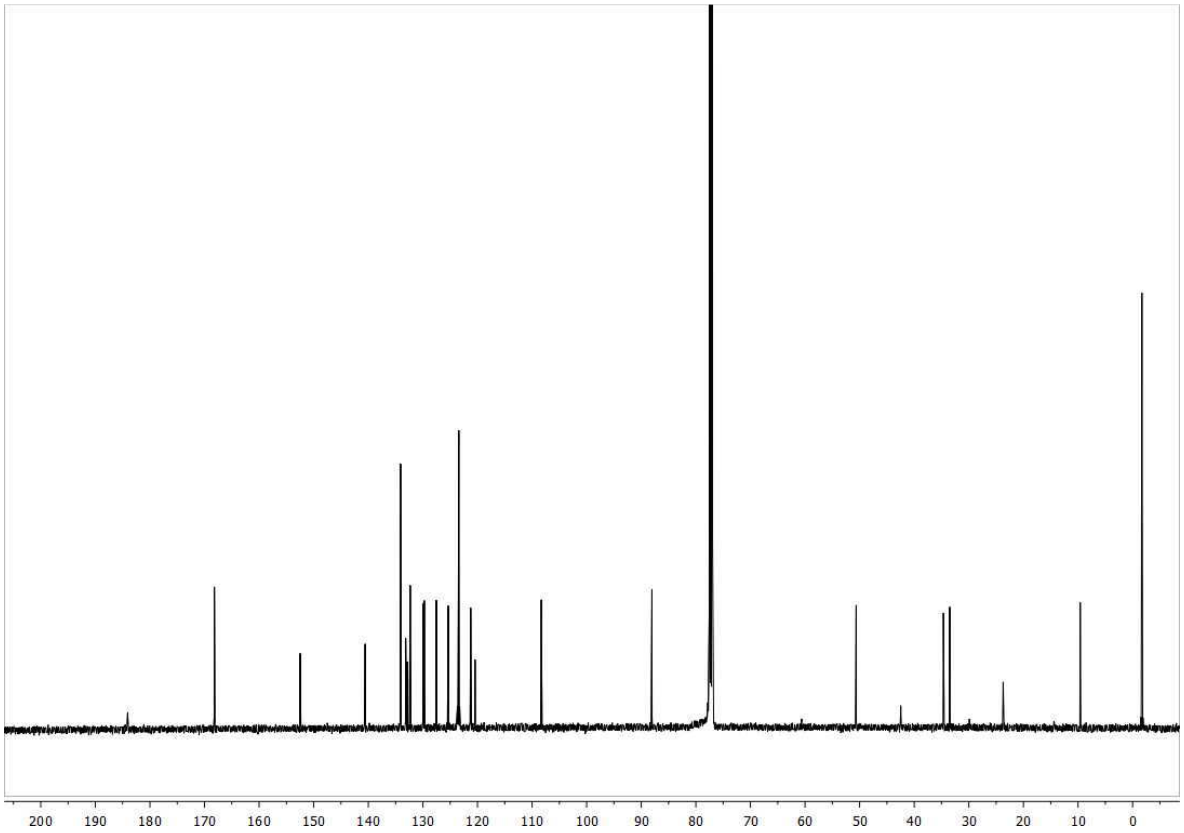
2.5
2.0
1.5
1.0
0.5
0.0
-0.5


200
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180
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150
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90
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70
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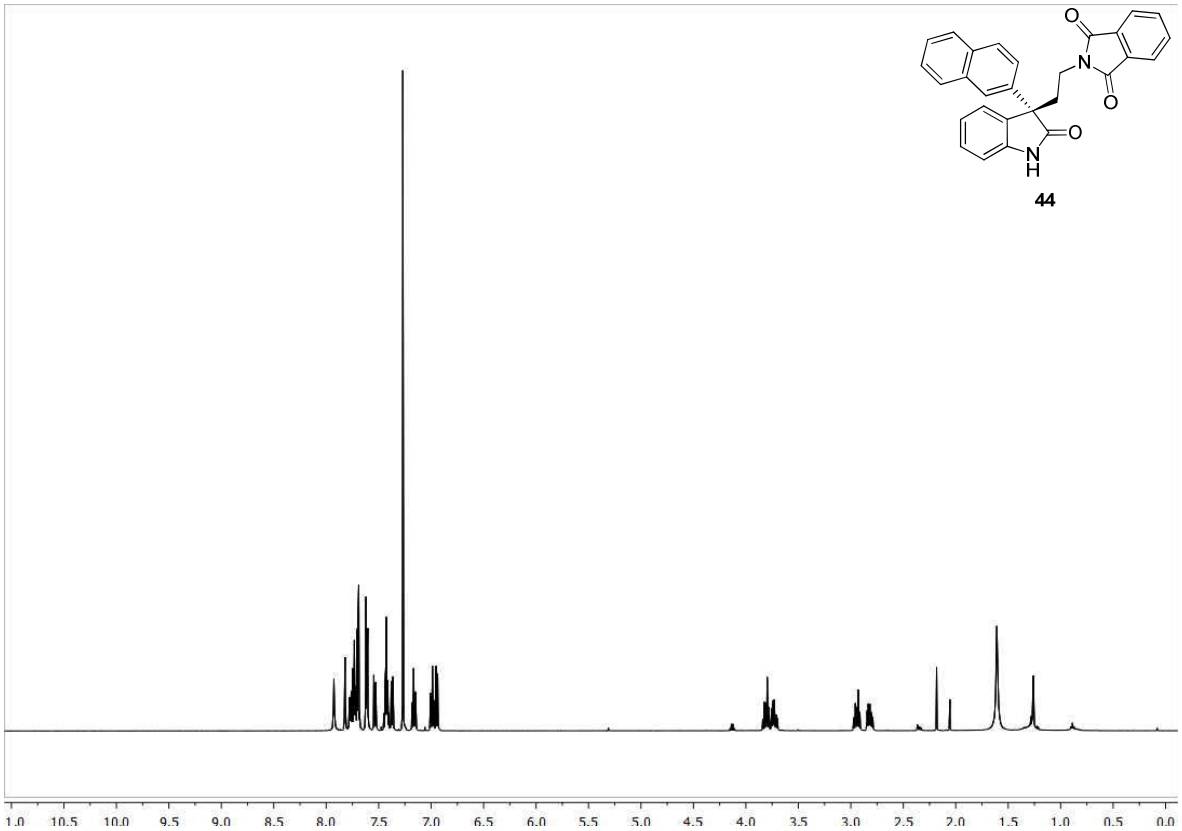
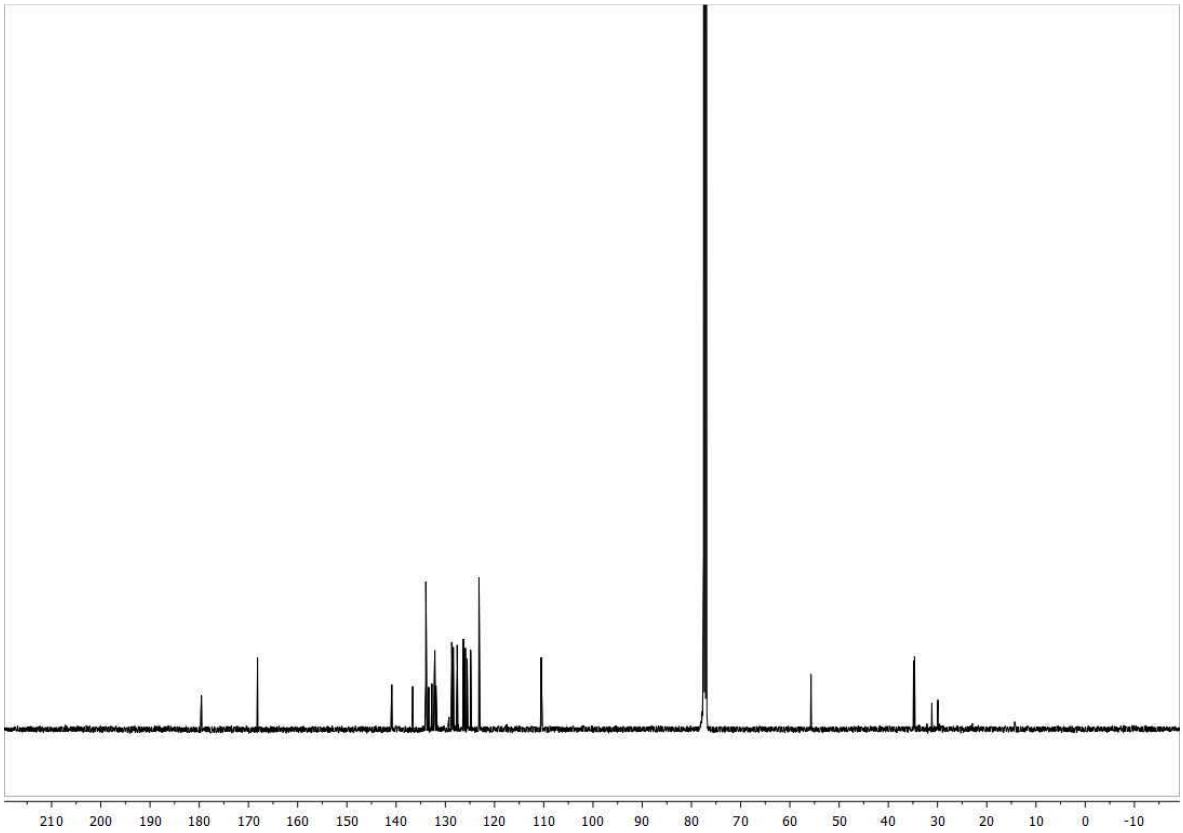
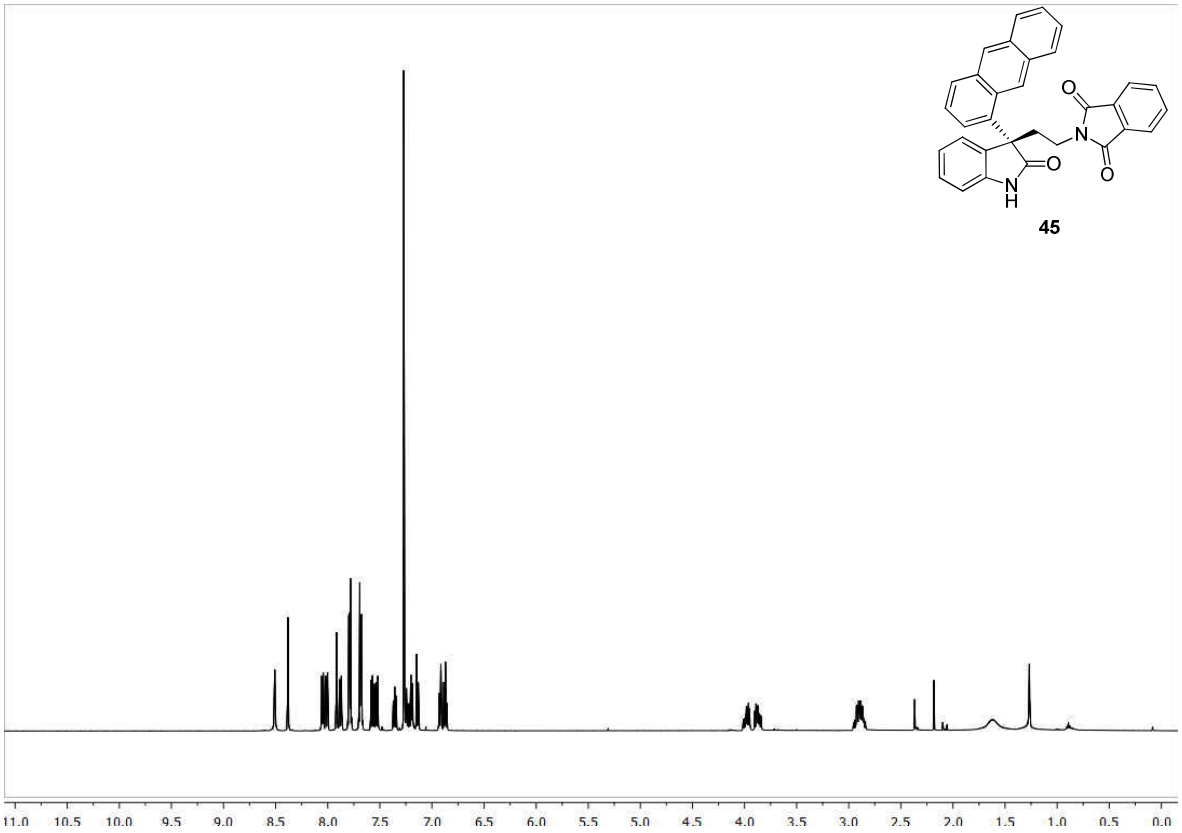
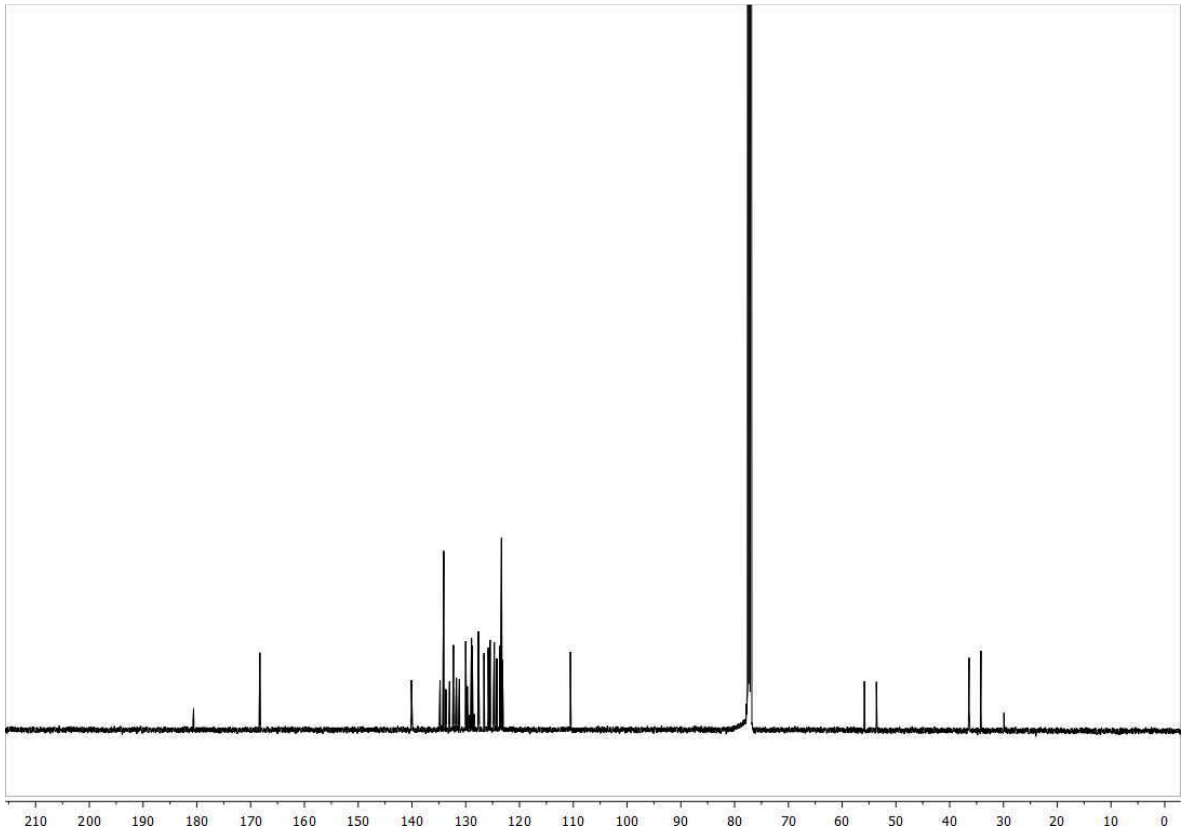
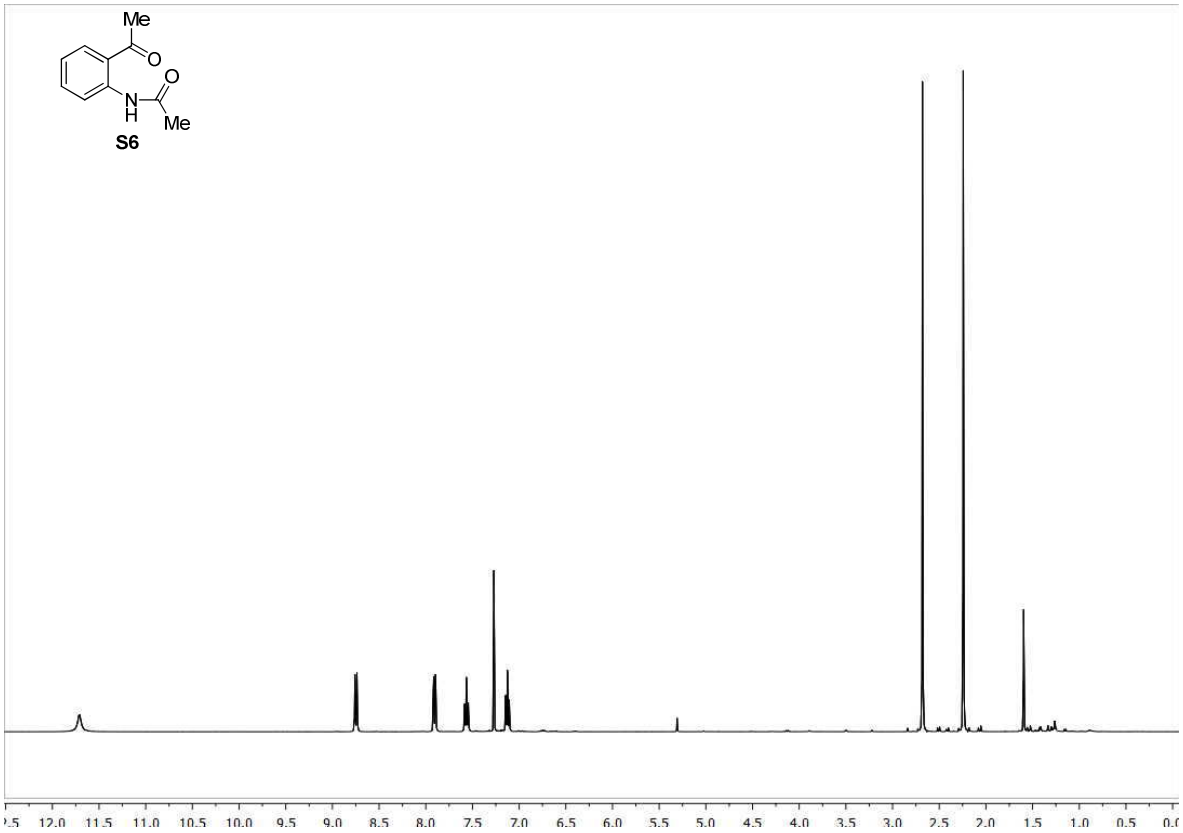
30
20
10
0

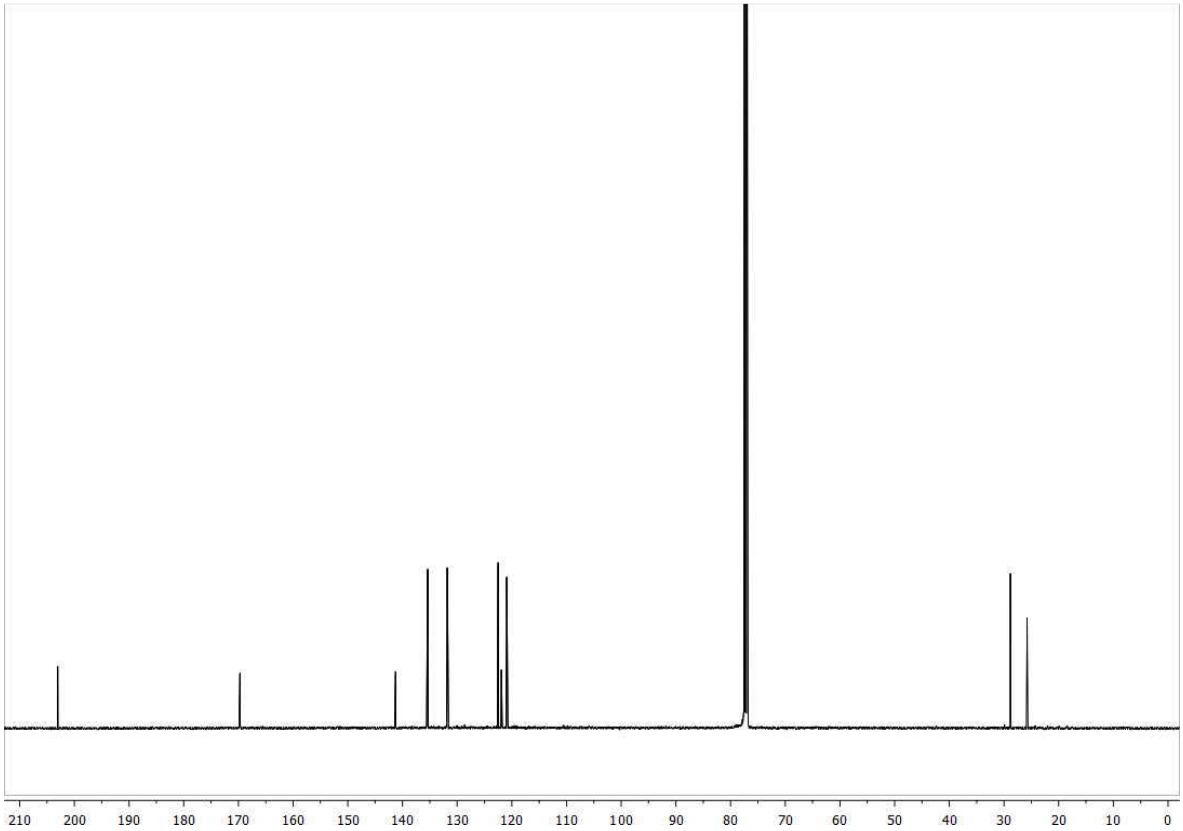

O
N
O
O
N
H
44

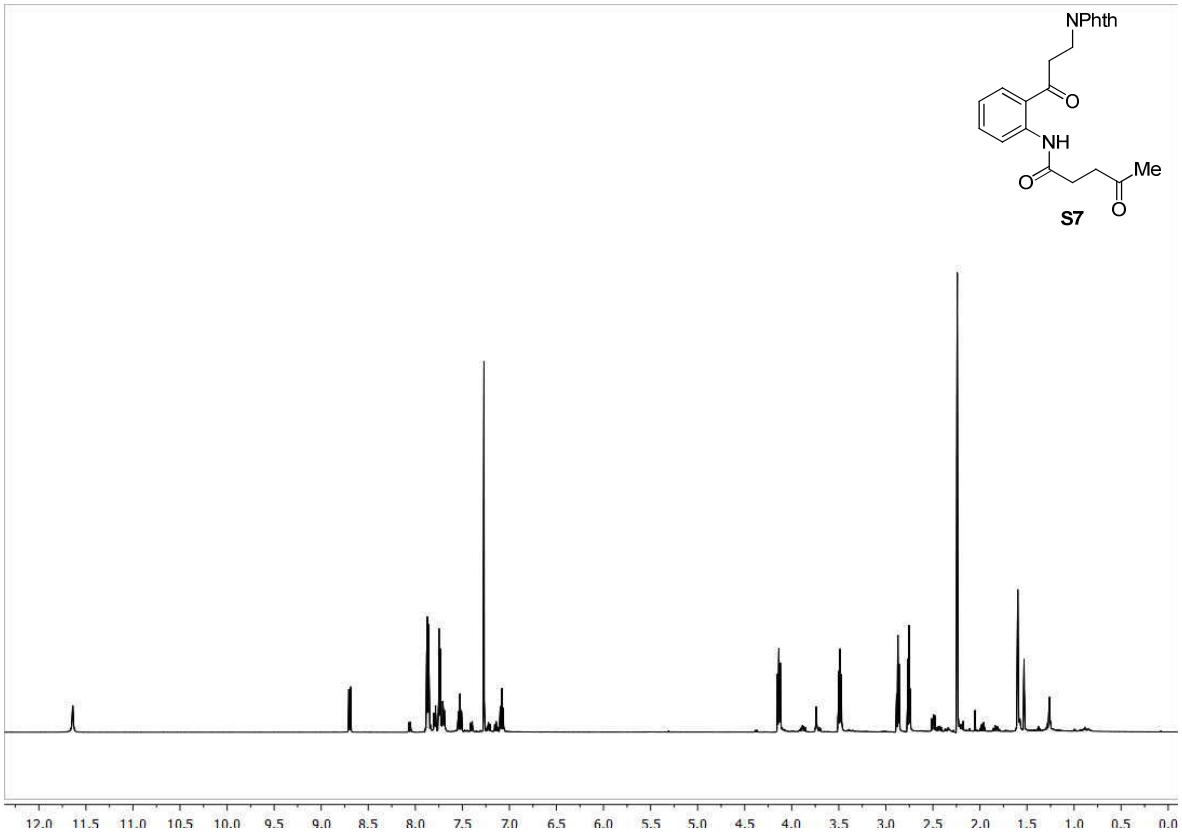


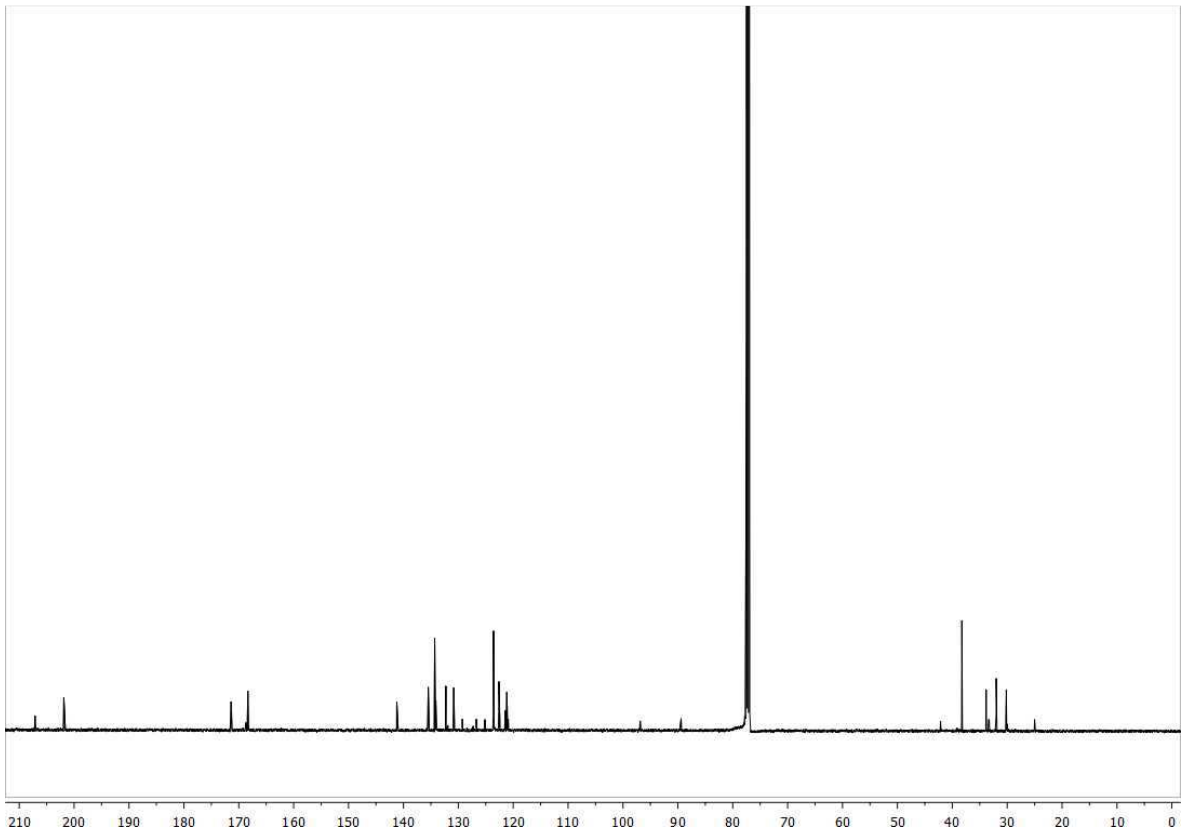

O
N
O
O
N
H
45




Me
O
O
N
H
Me
S6




NPhth
O
NH
O
Me
O
S7
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 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


210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

K. X-Ray Diffraction Report for Compound 41c.

Experimental

Data Collection

A colorless block crystal of $C_{26}H_{19}N_3O_3$ having approximate dimensions of 0.22 × 0.22 × 0.15 mm was mounted in a loop. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Cu-Kα radiation.

Indexing was performed from 4 oscillations that were exposed for 180 seconds. The crystal-to-detector distance was 127.40 mm.

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

a = 7.62626(14) Å

b = 9.13104(17) Å β = 92.6325(13)°

c = 14.3228(3) Å

V = 996.33(3) Å^3

For Z = 2 and F.W. = 421.45, the calculated density is 1.405 g/cm^3. Based on the systematic absences 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 -180±1°C to a maximum 2θ value of 136.5°. A total of 171 oscillation images were collected. A sweep of data was done using ω scans from 20.0 to 200.0° in 5.0° step, at χ=0.0° and φ = 0.0°. The exposure rate was 36.0 [sec./°]. A second sweep was performed using ω scans from 20.0 to 185.0° in 5.0° step, at χ=54.0° and φ = 0.0°. The exposure rate was 36.0 [sec./°]. Another sweep was performed using ω scans from 21.0 to 191.0° in 5.0° step, at χ=54.0° and φ = 90.0°. The exposure rate was 36.0 [sec./°]. Another sweep was performed using ω scans from 20.0 to 200.0° in 5.0° step, at χ=54.0° and φ = 270.0°. The exposure rate was 36.0 [sec./°]. Another sweep was performed using ω scans from 24.0 to 184.0° in 5.0° step, at χ=54.0° and φ = 180.0°. The exposure rate was 36.0 [sec./°]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 10803 reflections that were collected, 3418 were unique (R_{int} = 0.030).

The linear absorption coefficient, μ, for Cu-Kα radiation is 7.601 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.781 to 0.892. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[7]and expanded using Fourier techniques[8]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[9] on F^2 was based on 3413 observed reflections and 298 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$R1 = \Sigma \, ||F_o| - |F_c|| \, / \, \Sigma \, |F_o| = 0.0305$

$wR2 = [\Sigma (w (F_o^2 - F_c^2)^2) / \Sigma \, w(F_o^2)^2]^{1/2} = 0.0770$

The standard deviation of an observation of unit weight[10] was 1.08. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.18 and -0.16 $e^-/Å^3$, respectively. The absolute structure was deduced based on Flack parameter, 0.24(17), using 1489 Friedel pairs.[11]

Neutral atom scattering factors were taken from Cromer and Waber[12]. Anomalous dispersion effects were included in F_{calc}[13]; the values for Δf' and Δf'' were those of Creagh and McAuley[14]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[15]. All calculations were performed using the CrystalStructure[16] crystallographic software package except for refinement, which was performed using SHELXL-97[17].

Experimental Details

a. Crystal Data

Empirical Formula	$C_{26}H_{19}N_3O_3$
Formula Weight	421.45
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.22 × 0.22 × 0.15 mm
Crystal System	monoclinic
Lattice Type	Primitive
Indexing Images	4 oscillations @ 180.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 7.62626(14) Å
	b = 9.13104(17) Å
	c = 14.3228(3) Å

$\beta = 92.6325(13)^{\circ}$

$V = 996.33(3)\ Å^3$

Space Group	$P2_1$ (#4)
Z value	2
D_{calc}	1.405 g/cm^3
F_{000}	440.00
μ(CuKα)	7.601 cm^{-1}

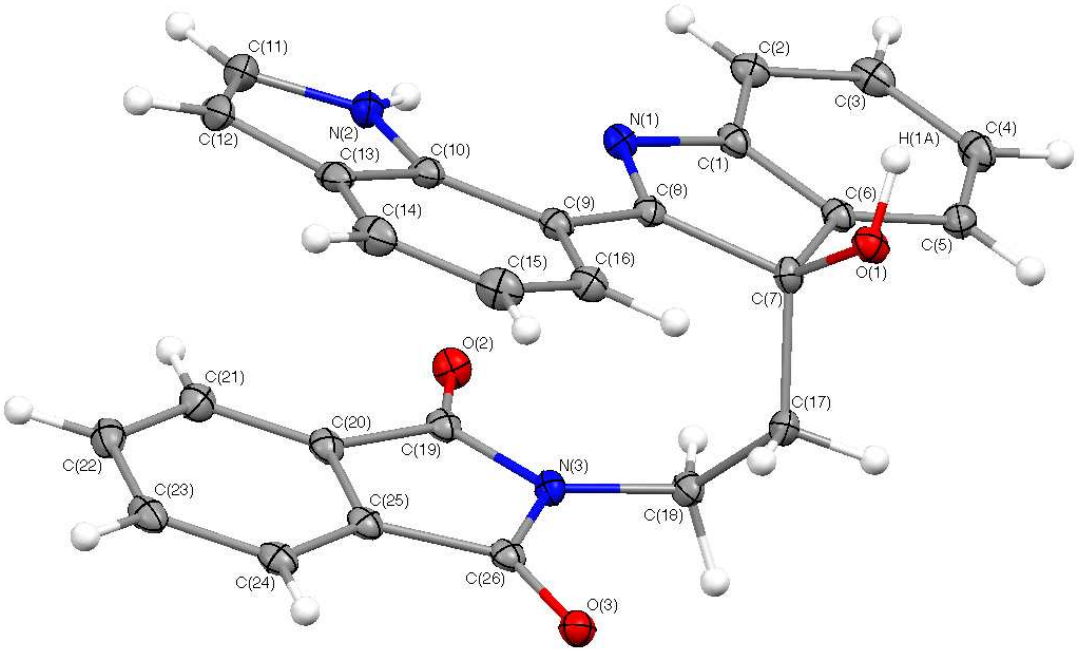
b. Intensity Measurements

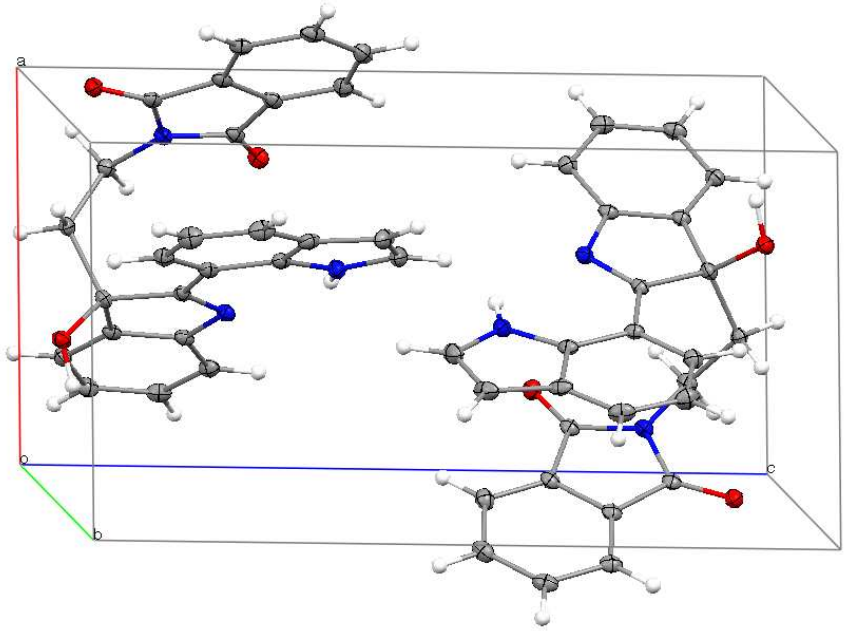
Diffractometer	Rigaku RAXIS-RAPID	
Radiation	CuKα (λ = 1.54187 Å)	
	graphite monochromated	
Detector Aperture	280 mm × 256 mm	
Data Images	171 exposures	
ω oscillation Range (χ=0.0, φ=0.0)		20.0 - 200.0°
Exposure Rate	36.0 sec./°	
ω oscillation Range (χ=54.0, φ=0.0)		20.0 - 185.0°
Exposure Rate	36.0 sec./°	
ω oscillation Range (χ=54.0, φ=90.0)		21.0 - 191.0°
Exposure Rate	36.0 sec./°	
ω oscillation Range (χ=54.0, φ=270.0)		20.0 - 200.0°
Exposure Rate	36.0 sec./°	
ω oscillation Range (χ=54.0, φ=180.0)		24.0 - 184.0°
Exposure Rate	36.0 sec./°	
Detector Position	127.40 mm	
Pixel Size	0.100 mm	
$2\theta_{max}$	136.5°	

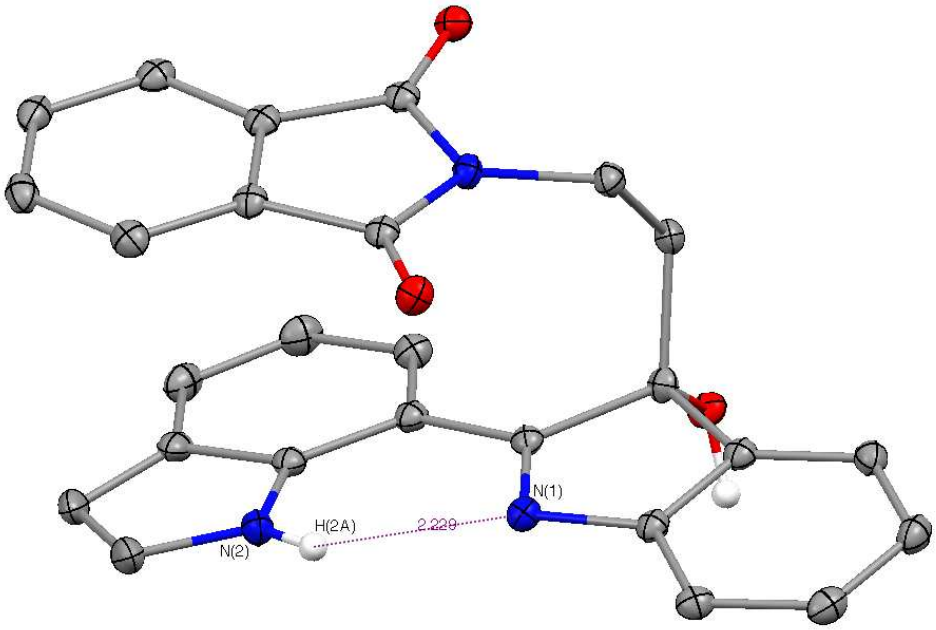
No. of Reflections Measured	Total: 10803
	Unique: 3413 (R_{int} = 0.030)
	Friedel pairs: 1489
Corrections	Lorentz-polarization
	Absorption
	(trans. factors: 0.781 - 0.892)

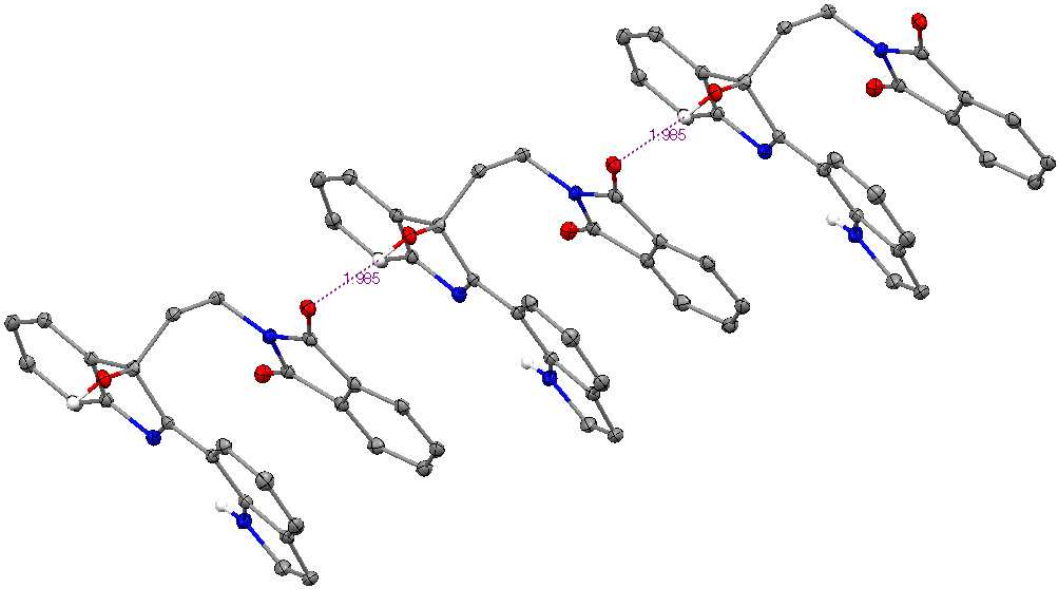
c. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELX97)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\Sigma\ w\ (F_o^2 - F_c^2)^2$
Least Squares Weights	$w = 1/[\ \sigma^2(F_o^2) + (0.0399 \cdot P)^2 + 0.0984 \cdot P\ ]$
	where $P = (Max(F_o^2,0) + 2F_c^2)/3$
$2\theta_{max}$ cutoff	136.5°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	3413
No. Variables	298
Reflection/Parameter Ratio	11.45
Residuals: R1 (I>2.00σ(I))	0.0305
Residuals: R (All reflections)	0.0323
Residuals: wR2 (All reflections)	0.0770
Goodness of Fit Indicator	1.080
Flack Parameter	0.24(17)
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.18 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.16$e^-/Å^3$

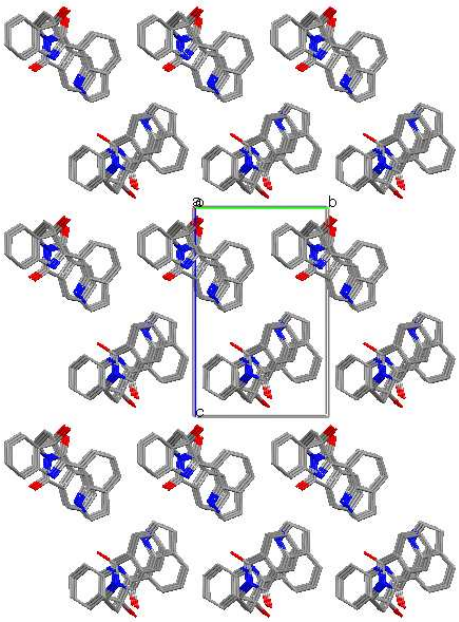

C(11)
C(12)
N(2)
C(13)
C(10)
C(14)
C(15)
C(16)
C(9)
N(1)
C(8)
C(1)
C(2)
C(3)
C(4)
H(1A)
C(6)
C(5)
O(1)
C(7)
C(17)
O(2)
C(21)
C(20)
C(19)
C(22)
C(23)
C(25)
N(3)
C(18)
C(24)
C(26)
O(3)


a
o
b
c

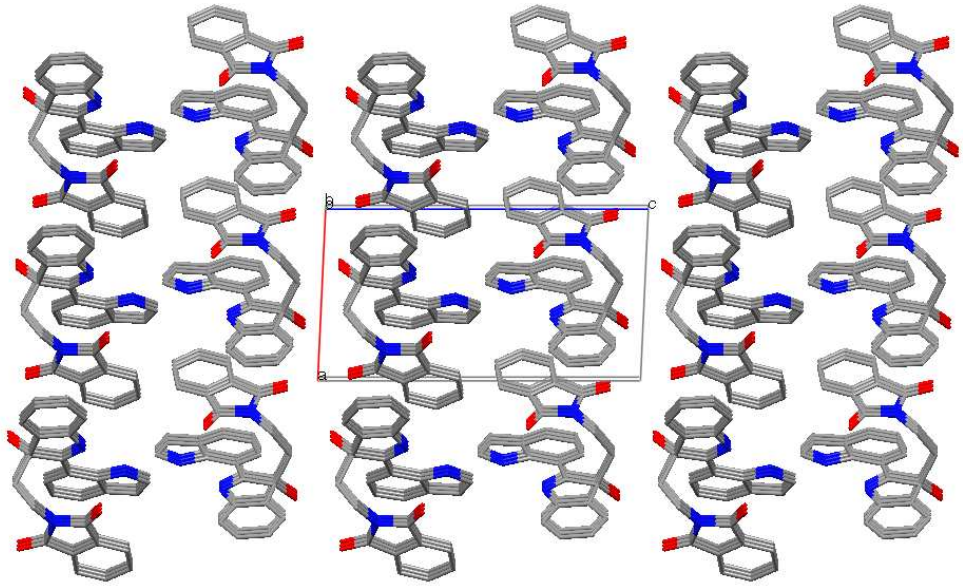

N(1)
H(2A)
2.229
N(2)


1.985
1.985

Packing diagram along the crystallographic a-axis



Packing diagram along the crystallographic b-axis



Packing diagram along the crystallographic c-axis

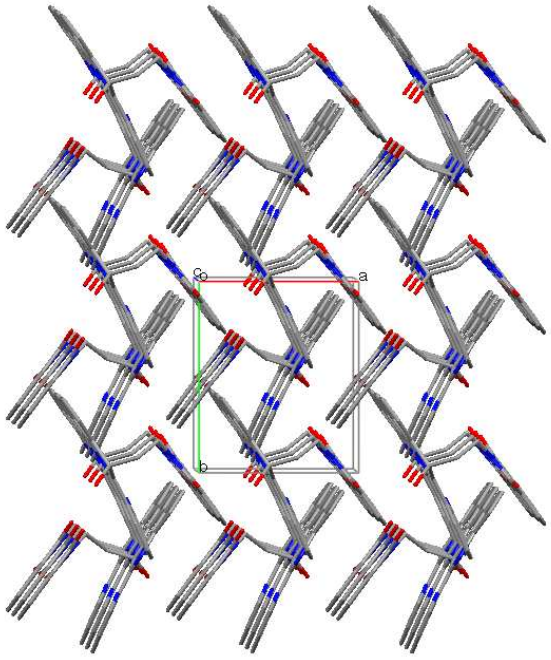


Table S3. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
O(1)	0.67725(16)	0.54037(14)	0.94714(8)	1.86(2)
O(2)	0.25987(16)	0.29457(13)	0.65759(8)	1.94(2)
O(3)	0.04021(15)	0.54743(14)	0.90183(8)	1.90(2)
N(1)	0.62529(17)	0.43015(15)	0.71619(9)	1.55(2)
N(2)	0.4759(2)	0.62345(18)	0.58607(10)	1.88(2)
N(3)	0.18701(17)	0.40790(16)	0.79575(10)	1.53(2)
C(1)	0.7121(2)	0.30881(19)	0.76023(12)	1.58(2)
C(2)	0.8079(2)	0.20117(19)	0.71699(13)	1.79(3)
C(3)	0.8908(2)	0.0963(2)	0.77401(13)	2.03(3)
C(4)	0.8788(2)	0.0985(2)	0.87045(13)	2.14(3)

Table S3. Atomic coordinates and B_{iso}/B_{eq} (continued)

C(5)	0.7831(2)	0.2073(2)	0.91289(12)	1.83(3)
C(6)	0.6999(2)	0.31174(19)	0.85742(12)	1.56(2)
C(7)	0.5913(2)	0.44398(19)	0.88092(11)	1.56(3)

C(8)	0.5630(2)	0.51060(18)	0.78191(11)	1.45(2)
C(9)	0.4767(2)	0.64975(19)	0.76040(12)	1.60(3)
C(10)	0.4430(2)	0.6947(2)	0.66736(12)	1.66(3)
C(11)	0.4259(2)	0.7110(2)	0.51166(13)	2.44(3)
C(12)	0.3579(2)	0.8380(2)	0.54324(14)	2.44(3)
C(13)	0.3679(2)	0.8319(2)	0.64334(13)	2.01(3)
C(14)	0.3238(2)	0.9278(2)	0.71549(14)	2.32(3)
C(15)	0.3525(2)	0.8845(2)	0.80680(14)	2.33(3)
C(16)	0.4273(2)	0.7480(2)	0.82939(13)	1.85(3)
C(17)	0.4182(2)	0.4009(2)	0.92477(11)	1.65(2)
C(18)	0.2862(2)	0.3148(2)	0.86318(12)	1.74(3)
C(19)	0.1846(2)	0.39121(19)	0.69811(12)	1.59(2)
C(20)	0.0738(2)	0.51268(19)	0.65939(12)	1.68(3)
C(21)	0.0312(2)	0.5514(2)	0.56793(12)	1.99(3)
C(22)	-0.0771(2)	0.6728(2)	0.55342(14)	2.12(3)
C(23)	-0.1394(2)	0.7516(2)	0.62804(13)	2.13(3)
C(24)	-0.0972(2)	0.71174(19)	0.72008(13)	1.85(3)
C(25)	0.0099(2)	0.59064(19)	0.73377(12)	1.66(3)
C(26)	0.0760(2)	0.51973(19)	0.82132(12)	1.61(3)

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

Table S4. Atomic coordinates and B_{iso} involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.8166	0.1992	0.6511	2.15
H(2)	0.9573	0.0212	0.7463	2.43
H(3)	0.9364	0.0252	0.9076	2.57
H(4)	0.7752	0.2097	0.9789	2.20
H(5)	0.4372	0.6865	0.4478	2.93
H(6)	0.3121	0.9165	0.5060	2.93
H(7)	0.2748	1.0212	0.7013	2.79
H(8)	0.3211	0.9484	0.8556	2.80
H(9)	0.4448	0.7215	0.8933	2.21
H(10)	0.4471	0.3420	0.9814	1.98

H(11)	0.3602	0.4916	0.9453	1.98
H(12)	0.3494	0.2387	0.8287	2.09
H(13)	0.2029	0.2646	0.9034	2.09
H(14)	0.0741	0.4974	0.5170	2.39
H(15)	-0.1091	0.7023	0.4913	2.55
H(16)	-0.2124	0.8345	0.6159	2.55
H(17)	-0.1400	0.7652	0.7712	2.22
H(1A)	0.790(2)	0.552(2)	0.9342(13)	2.8(4)
H(2A)	0.522(2)	0.539(2)	0.5857(13)	2.6(4)

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

Table S5. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.0206(6)	0.0266(6)	0.0232(6)	-0.0019(5)	-0.0013(5)	-0.0055(5)
O(2)	0.0265(6)	0.0209(6)	0.0260(6)	0.0019(5)	-0.0004(5)	-0.0042(5)
O(3)	0.0222(6)	0.0283(6)	0.0218(6)	-0.0020(5)	0.0008(5)	-0.0030(5)
N(1)	0.0193(7)	0.0180(7)	0.0215(7)	0.0001(6)	-0.0008(5)	-0.0021(6)
N(2)	0.0239(8)	0.0271(9)	0.0203(7)	0.0034(6)	0.0001(6)	0.0029(6)
N(3)	0.0197(7)	0.0168(7)	0.0214(7)	-0.0005(6)	-0.0026(5)	0.0007(6)
C(1)	0.0168(8)	0.0173(8)	0.0257(9)	-0.0016(6)	-0.0019(7)	0.0009(7)
C(2)	0.0217(8)	0.0202(9)	0.0261(9)	-0.0029(7)	0.0009(7)	-0.0033(7)
C(3)	0.0210(8)	0.0177(9)	0.0383(10)	-0.0002(7)	0.0008(7)	-0.0037(8)
C(4)	0.0229(9)	0.0213(9)	0.0369(10)	0.0015(7)	-0.0031(7)	0.0052(8)
C(5)	0.0216(8)	0.0245(9)	0.0233(9)	-0.0018(7)	-0.0015(7)	0.0034(8)
C(6)	0.0172(8)	0.0179(8)	0.0240(8)	-0.0024(6)	-0.0011(6)	0.0001(7)
C(7)	0.0208(8)	0.0209(9)	0.0175(8)	-0.0004(7)	-0.0028(6)	-0.0016(7)
C(8)	0.0159(8)	0.0185(8)	0.0205(8)	-0.0039(6)	-0.0016(6)	0.0006(7)
C(9)	0.0163(8)	0.0189(8)	0.0255(9)	-0.0017(6)	0.0006(7)	0.0011(7)
C(10)	0.0161(8)	0.0214(9)	0.0257(9)	-0.0022(7)	0.0020(6)	0.0027(8)
C(11)	0.0245(9)	0.0434(12)	0.0249(9)	0.0010(8)	0.0007(7)	0.0114(9)
C(12)	0.0229(9)	0.0345(11)	0.0355(10)	0.0026(8)	0.0021(8)	0.0187(9)
C(13)	0.0174(8)	0.0240(10)	0.0353(10)	-0.0010(7)	0.0054(7)	0.0090(8)
C(14)	0.0245(9)	0.0176(9)	0.0463(11)	0.0028(7)	0.0032(8)	0.0048(8)

C(15)	0.0295(10)	0.0200(9)	0.0393(11)	0.0021(7)	0.0046(8)	-0.0049(8)
C(16)	0.0230(8)	0.0210(9)	0.0260(9)	-0.0024(7)	0.0000(7)	-0.0029(8)
C(17)	0.0222(8)	0.0221(8)	0.0181(8)	0.0013(7)	-0.0005(6)	0.0037(7)
C(18)	0.0213(8)	0.0199(9)	0.0246(8)	-0.0008(7)	-0.0010(7)	0.0041(7)
C(19)	0.0178(8)	0.0180(9)	0.0242(8)	-0.0046(7)	-0.0016(6)	-0.0001(7)
C(20)	0.0193(8)	0.0192(9)	0.0250(9)	-0.0049(6)	-0.0034(7)	-0.0004(7)
C(21)	0.0270(9)	0.0247(9)	0.0238(9)	-0.0054(8)	-0.0015(7)	-0.0018(7)
C(22)	0.0271(9)	0.0240(10)	0.0288(9)	-0.0052(7)	-0.0058(7)	0.0065(8)
C(23)	0.0201(8)	0.0202(9)	0.0401(11)	-0.0027(7)	-0.0034(8)	0.0069(8)
C(24)	0.0191(8)	0.0184(9)	0.0328(10)	-0.0037(7)	0.0004(7)	-0.0013(8)
C(25)	0.0176(8)	0.0196(9)	0.0258(8)	-0.0055(6)	-0.0014(7)	-0.0018(7)
C(26)	0.0169(8)	0.0196(9)	0.0246(9)	-0.0053(6)	-0.0004(7)	-0.0007(7)

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 S6. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
O(1)	C(7)	1.431(2)	O(2)	C(19)	1.215(2)
O(3)	C(26)	1.224(2)	N(1)	C(1)	1.423(2)
N(1)	C(8)	1.301(2)	N(2)	C(10)	1.367(2)
N(2)	C(11)	1.372(2)	N(3)	C(18)	1.470(2)
N(3)	C(19)	1.406(2)	N(3)	C(26)	1.386(2)
C(1)	C(2)	1.387(2)	C(1)	C(6)	1.400(2)
C(2)	C(3)	1.392(2)	C(3)	C(4)	1.389(2)
C(4)	C(5)	1.389(2)	C(5)	C(6)	1.377(2)
C(6)	C(7)	1.511(2)	C(7)	C(8)	1.549(2)
C(7)	C(17)	1.539(2)	C(8)	C(9)	1.457(2)
C(9)	C(10)	1.407(2)	C(9)	C(16)	1.399(2)
C(10)	C(13)	1.414(2)	C(11)	C(12)	1.356(2)
C(12)	C(13)	1.433(2)	C(13)	C(14)	1.407(2)
C(14)	C(15)	1.374(2)	C(15)	C(16)	1.402(2)
C(17)	C(18)	1.526(2)	C(19)	C(20)	1.486(2)
C(20)	C(21)	1.381(2)	C(20)	C(25)	1.388(2)
C(21)	C(22)	1.393(2)	C(22)	C(23)	1.390(2)
C(23)	C(24)	1.391(2)	C(24)	C(25)	1.384(2)

C(25)	C(26)	1.479(2)

Table S7. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
O(1)	H(1A)	0.89(2)	N(2)	H(2A)	0.85(2)
C(2)	H(1)	0.950	C(3)	H(2)	0.950
C(4)	H(3)	0.950	C(5)	H(4)	0.950
C(11)	H(5)	0.950	C(12)	H(6)	0.950
C(14)	H(7)	0.950	C(15)	H(8)	0.950
C(16)	H(9)	0.950	C(17)	H(10)	0.990
C(17)	H(11)	0.990	C(18)	H(12)	0.990
C(18)	H(13)	0.990	C(21)	H(14)	0.950
C(22)	H(15)	0.950	C(23)	H(16)	0.950
C(24)	H(17)	0.950			

Table S8. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(1)	N(1)	C(8)	107.27(13)	C(10)	N(2)	C(11)	109.20(15)
C(18)	N(3)	C(19)	125.01(14)	C(18)	N(3)	C(26)	123.67(14)
C(19)	N(3)	C(26)	111.24(13)	N(1)	C(1)	C(2)	126.69(15)
N(1)	C(1)	C(6)	112.03(14)	C(2)	C(1)	C(6)	121.19(15)
C(1)	C(2)	C(3)	117.39(16)	C(2)	C(3)	C(4)	121.64(16)
C(3)	C(4)	C(5)	120.38(16)	C(4)	C(5)	C(6)	118.69(16)
C(1)	C(6)	C(5)	120.71(15)	C(1)	C(6)	C(7)	107.40(14)
C(5)	C(6)	C(7)	131.87(15)	O(1)	C(7)	C(6)	113.60(13)
O(1)	C(7)	C(8)	113.94(13)	O(1)	C(7)	C(17)	105.19(12)
C(6)	C(7)	C(8)	99.48(12)	C(6)	C(7)	C(17)	112.08(14)
C(8)	C(7)	C(17)	112.81(13)	N(1)	C(8)	C(7)	113.60(13)
N(1)	C(8)	C(9)	121.04(14)	C(7)	C(8)	C(9)	125.35(14)
C(8)	C(9)	C(10)	121.04(15)	C(8)	C(9)	C(16)	122.92(15)
C(10)	C(9)	C(16)	116.01(15)	N(2)	C(10)	C(9)	129.45(16)
N(2)	C(10)	C(13)	107.61(15)	C(9)	C(10)	C(13)	122.92(16)
N(2)	C(11)	C(12)	109.64(16)	C(11)	C(12)	C(13)	107.24(17)

C(10)	C(13)	C(12)	106.30(16)	C(10)	C(13)	C(14)	118.73(16)
C(12)	C(13)	C(14)	134.97(17)	C(13)	C(14)	C(15)	119.15(17)
C(14)	C(15)	C(16)	121.36(18)	C(9)	C(16)	C(15)	121.79(17)
C(7)	C(17)	C(18)	116.84(13)	N(3)	C(18)	C(17)	112.82(14)
O(2)	C(19)	N(3)	124.67(15)	O(2)	C(19)	C(20)	129.57(15)
N(3)	C(19)	C(20)	105.76(14)	C(19)	C(20)	C(21)	130.52(16)
C(19)	C(20)	C(25)	108.05(14)	C(21)	C(20)	C(25)	121.43(16)
C(20)	C(21)	C(22)	117.20(16)	C(21)	C(22)	C(23)	121.26(17)
C(22)	C(23)	C(24)	121.36(17)	C(23)	C(24)	C(25)	116.94(16)
C(20)	C(25)	C(24)	121.79(16)	C(20)	C(25)	C(26)	107.96(14)
C(24)	C(25)	C(26)	130.24(16)	O(3)	C(26)	N(3)	124.51(15)
O(3)	C(26)	C(25)	128.79(15)	N(3)	C(26)	C(25)	106.67(14)

Table S9. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(7)	O(1)	H(1A)	110.4(13)	C(10)	N(2)	H(2A)	122.0(13)
C(11)	N(2)	H(2A)	128.8(13)	C(1)	C(2)	H(1)	121.3
C(3)	C(2)	H(1)	121.3	C(2)	C(3)	H(2)	119.2
C(4)	C(3)	H(2)	119.2	C(3)	C(4)	H(3)	119.8
C(5)	C(4)	H(3)	119.8	C(4)	C(5)	H(4)	120.7
C(6)	C(5)	H(4)	120.7	N(2)	C(11)	H(5)	125.2
C(12)	C(11)	H(5)	125.2	C(11)	C(12)	H(6)	126.4
C(13)	C(12)	H(6)	126.4	C(13)	C(14)	H(7)	120.4
C(15)	C(14)	H(7)	120.4	C(14)	C(15)	H(8)	119.3
C(16)	C(15)	H(8)	119.3	C(9)	C(16)	H(9)	119.1
C(15)	C(16)	H(9)	119.1	C(7)	C(17)	H(10)	108.1
C(7)	C(17)	H(11)	108.1	C(18)	C(17)	H(10)	108.1
C(18)	C(17)	H(11)	108.1	H(10)	C(17)	H(11)	107.3
N(3)	C(18)	H(12)	109.0	N(3)	C(18)	H(13)	109.0
C(17)	C(18)	H(12)	109.0	C(17)	C(18)	H(13)	109.0
H(12)	C(18)	H(13)	107.8	C(20)	C(21)	H(14)	121.4
C(22)	C(21)	H(14)	121.4	C(21)	C(22)	H(15)	119.4
C(23)	C(22)	H(15)	119.4	C(22)	C(23)	H(16)	119.3

C(24)	C(23)	H(16)	119.3	C(23)	C(24)	H(17)	121.5	
C(25)	C(24)	H(17)	121.5					

Table S10. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(1)	N(1)	C(8)	C(7)	-4.10(18)	C(1)	N(1)	C(8)	C(9)	175.61(14)
C(8)	N(1)	C(1)	C(2)	-175.08(16)	C(8)	N(1)	C(1)	C(6)	1.49(18)
C(10)	N(2)	C(11)	C(12)	-1.0(2)	C(11)	N(2)	C(10)	C(9)	-178.15(17)
C(11)	N(2)	C(10)	C(13)	0.58(19)	C(18)	N(3)	C(19)	O(2)	2.5(2)
C(18)	N(3)	C(19)	C(20)	-177.70(14)	C(19)	N(3)	C(18)	C(17)	120.80(16)
C(18)	N(3)	C(26)	O(3)	-4.6(2)	C(18)	N(3)	C(26)	C(25)	177.11(14)
C(26)	N(3)	C(18)	C(17)	-62.6(2)	C(19)	N(3)	C(26)	O(3)	172.35(15)
C(19)	N(3)	C(26)	C(25)	-5.91(18)	C(26)	N(3)	C(19)	O(2)	-174.42(16)
C(26)	N(3)	C(19)	C(20)	5.37(18)	N(1)	C(1)	C(2)	C(3)	176.47(15)
N(1)	C(1)	C(6)	C(5)	-176.80(14)	N(1)	C(1)	C(6)	C(7)	1.69(18)
C(2)	C(1)	C(6)	C(5)	-0.0(2)	C(2)	C(1)	C(6)	C(7)	178.48(15)
C(6)	C(1)	C(2)	C(3)	0.2(2)	C(1)	C(2)	C(3)	C(4)	-0.04(19)
C(2)	C(3)	C(4)	C(5)	-0.3(2)	C(3)	C(4)	C(5)	C(6)	0.4(2)
C(4)	C(5)	C(6)	C(1)	-0.3(2)	C(4)	C(5)	C(6)	C(7)	-178.36(17)
C(1)	C(6)	C(7)	O(1)	-125.05(14)	C(1)	C(6)	C(7)	C(8)	-3.59(16)
C(1)	C(6)	C(7)	C(17)	115.88(14)	C(5)	C(6)	C(7)	O(1)	53.2(2)
C(5)	C(6)	C(7)	C(8)	174.67(17)	C(5)	C(6)	C(7)	C(17)	-65.9(2)
O(1)	C(7)	C(8)	N(1)	126.09(15)	O(1)	C(7)	C(8)	C(9)	-53.6(2)
O(1)	C(7)	C(17)	C(18)	171.62(13)	C(6)	C(7)	C(8)	N(1)	4.87(17)
C(6)	C(7)	C(8)	C(9)	-174.81(15)	C(6)	C(7)	C(17)	C(18)	-64.47(18)
C(8)	C(7)	C(17)	C(18)	46.8(2)	C(17)	C(7)	C(8)	N(1)	-114.05(16)
C(17)	C(7)	C(8)	C(9)	66.3(2)	N(1)	C(8)	C(9)	C(10)	6.2(2)
N(1)	C(8)	C(9)	C(16)	-171.88(15)	C(7)	C(8)	C(9)	C(10)	-174.14(15)
C(7)	C(8)	C(9)	C(16)	7.8(2)	C(8)	C(9)	C(10)	N(2)	1.9(2)
C(8)	C(9)	C(10)	C(13)	-176.68(15)	C(8)	C(9)	C(16)	C(15)	176.69(16)
C(10)	C(9)	C(16)	C(15)	-1.5(2)	C(16)	C(9)	C(10)	N(2)	-179.92(16)
C(16)	C(9)	C(10)	C(13)	1.5(2)	N(2)	C(10)	C(13)	C(12)	0.03(14)
N(2)	C(10)	C(13)	C(14)	-179.16(15)	C(9)	C(10)	C(13)	C(12)	178.86(15)

C(9)	C(10)	C(13)	C(14)	-0.3(2)	N(2)	C(11)	C(12)	C(13)	1.0(2)
C(11)	C(12)	C(13)	C(10)	-0.63(19)	C(11)	C(12)	C(13)	C(14)	178.36(19)
C(10)	C(13)	C(14)	C(15)	-1.0(2)	C(12)	C(13)	C(14)	C(15)	-179.87(19)
C(13)	C(14)	C(15)	C(16)	1.0(2)	C(14)	C(15)	C(16)	C(9)	0.2(2)
C(7)	C(17)	C(18)	N(3)	-78.36(18)	O(2)	C(19)	C(20)	C(21)	-2.4(3)
O(2)	C(19)	C(20)	C(25)	177.15(17)	N(3)	C(19)	C(20)	C(21)	177.87(17)
N(3)	C(19)	C(20)	C(25)	-2.63(18)	C(19)	C(20)	C(21)	C(22)	-179.95(13)
C(19)	C(20)	C(25)	C(24)	179.56(15)	C(19)	C(20)	C(25)	C(26)	-0.84(18)
C(21)	C(20)	C(25)	C(24)	-0.9(2)	C(21)	C(20)	C(25)	C(26)	178.72(15)

Table S11. Torsion angles (°) (continued)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(25)	C(20)	C(21)	C(22)	0.6(2)	C(20)	C(21)	C(22)	C(23)	0.1(2)
C(21)	C(22)	C(23)	C(24)	-0.5(2)	C(22)	C(23)	C(24)	C(25)	0.2(2)
C(23)	C(24)	C(25)	C(20)	0.4(2)	C(23)	C(24)	C(25)	C(26)	-179.07(17)
C(20)	C(25)	C(26)	O(3)	-174.09(17)	C(20)	C(25)	C(26)	N(3)	4.07(18)
C(24)	C(25)	C(26)	O(3)	5.5(3)	C(24)	C(25)	C(26)	N(3)	-176.38(17)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table S12. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(1)	O(3)[1)]	2.8727(16)	O(1)	N(1)	3.4629(17)
O(1)	C(1)	3.431(2)	O(1)	C(5)	3.190(2)
O(1)	C(6)	2.462(2)	O(1)	C(8)	2.4988(19)
O(1)	C(9)	3.180(2)	O(1)	C(16)	3.127(2)
O(1)	C(17)	2.360(2)	O(2)	N(1)	3.1286(17)
O(2)	N(2)	3.597(2)	O(2)	N(3)	2.3230(18)
O(2)	C(8)	3.4681(19)	O(2)	C(11)[2)]	3.569(2)
O(2)	C(14)[3)]	3.479(2)	O(2)	C(18)	2.948(2)
O(2)	C(20)	2.446(2)	O(2)	C(21)	3.159(2)
O(2)	C(22)[4)]	3.453(2)	O(2)	C(25)	3.510(2)
O(2)	C(26)	3.463(2)	O(3)	O(1)[5)]	2.8727(16)

O(3)	N(3)	2.3111(18)	O(3)	C(4)[6)]	3.324(2)
O(3)	C(5)[6)]	3.265(2)	O(3)	C(6)[5)]	3.409(2)
O(3)	C(7)[5)]	3.551(2)	O(3)	C(17)	3.181(2)
O(3)	C(18)	2.904(2)	O(3)	C(19)	3.472(2)
O(3)	C(20)	3.508(2)	O(3)	C(24)	3.142(2)
O(3)	C(25)	2.440(2)	N(1)	O(1)	3.4629(17)
N(1)	O(2)	3.1286(17)	N(1)	N(2)	2.773(2)
N(1)	N(3)	3.5863(18)	N(1)	C(2)	2.512(2)
N(1)	C(6)	2.341(2)	N(1)	C(7)	2.389(2)
N(1)	C(9)	2.402(2)	N(1)	C(10)	2.858(2)
N(1)	C(17)	3.453(2)	N(1)	C(18)	3.568(2)
N(1)	C(19)	3.378(2)	N(1)	C(24)[1)]	3.329(2)
N(1)	C(25)[1)]	3.278(2)	N(2)	O(2)	3.597(2)
N(2)	N(1)	2.773(2)	N(2)	C(8)	3.033(2)
N(2)	C(9)	2.508(2)	N(2)	C(12)	2.231(2)
N(2)	C(12)[2)]	3.471(2)	N(2)	C(13)	2.244(2)
N(2)	C(14)	3.564(2)	N(2)	C(19)	3.512(2)
N(2)	C(20)	3.439(2)	N(2)	C(21)	3.452(2)
N(2)	C(22)[1)]	3.491(2)	N(2)	C(23)[1)]	3.190(2)
N(3)	O(2)	2.3230(18)	N(3)	O(3)	2.3111(18)
N(3)	N(1)	3.5863(18)	N(3)	C(2)[5)]	3.591(2)
N(3)	C(7)	3.280(2)	N(3)	C(8)	3.032(2)
N(3)	C(9)	3.181(2)	N(3)	C(17)	2.496(2)
N(3)	C(20)	2.307(2)	N(3)	C(25)	2.299(2)
C(1)	O(1)	3.431(2)	C(1)	C(3)	2.374(2)
C(1)	C(4)	2.759(2)	C(1)	C(5)	2.413(2)

Table S12. Distances beyond the asymmetric unit out to 3.60 Å (continued)

atom	atom	distance	atom	atom	distance
C(1)	C(7)	2.347(2)	C(1)	C(8)	2.195(2)
C(1)	C(9)	3.594(2)	C(1)	C(17)	3.431(2)
C(1)	C(25)[1)]	3.465(2)	C(1)	C(26)[1)]	3.457(2)
C(2)	N(1)	2.512(2)	C(2)	N(3)[1)]	3.591(2)

C(2)	C(4)	2.428(2)	C(2)	C(5)	2.821(2)
C(2)	C(6)	2.428(2)	C(2)	C(8)	3.535(2)
C(2)	C(19)[1)]	3.377(2)	C(3)	C(1)	2.374(2)
C(3)	C(5)	2.410(2)	C(3)	C(6)	2.753(2)
C(3)	C(24)[7)]	3.597(2)	C(4)	O(3)[8)]	3.324(2)
C(4)	C(1)	2.759(2)	C(4)	C(2)	2.428(2)
C(4)	C(6)	2.380(2)	C(5)	O(1)	3.190(2)
C(5)	O(3)[8)]	3.265(2)	C(5)	C(1)	2.413(2)
C(5)	C(2)	2.821(2)	C(5)	C(3)	2.410(2)
C(5)	C(7)	2.637(2)	C(5)	C(17)	3.308(2)
C(6)	O(1)	2.462(2)	C(6)	O(3)[1)]	3.409(2)
C(6)	N(1)	2.341(2)	C(6)	C(2)	2.428(2)
C(6)	C(3)	2.753(2)	C(6)	C(4)	2.380(2)
C(6)	C(8)	2.335(2)	C(6)	C(17)	2.530(2)
C(6)	C(18)	3.160(2)	C(6)	C(26)[1)]	3.498(2)
C(7)	O(3)[1)]	3.551(2)	C(7)	N(1)	2.389(2)
C(7)	N(3)	3.280(2)	C(7)	C(1)	2.347(2)
C(7)	C(5)	2.637(2)	C(7)	C(9)	2.671(2)
C(7)	C(16)	3.120(2)	C(7)	C(18)	2.611(2)
C(8)	O(1)	2.4988(19)	C(8)	O(2)	3.4681(19)
C(8)	N(2)	3.033(2)	C(8)	N(3)	3.032(2)
C(8)	C(1)	2.195(2)	C(8)	C(2)	3.535(2)
C(8)	C(6)	2.335(2)	C(8)	C(10)	2.493(2)
C(8)	C(16)	2.509(2)	C(8)	C(17)	2.572(2)
C(8)	C(18)	3.039(2)	C(8)	C(19)	3.262(2)
C(8)	C(24)[1)]	3.329(2)	C(8)	C(25)[1)]	3.584(2)
C(9)	O(1)	3.180(2)	C(9)	N(1)	2.402(2)
C(9)	N(2)	2.508(2)	C(9)	N(3)	3.181(2)
C(9)	C(1)	3.594(2)	C(9)	C(7)	2.671(2)
C(9)	C(13)	2.478(2)	C(9)	C(14)	2.855(2)
C(9)	C(15)	2.448(2)	C(9)	C(17)	3.317(2)
C(9)	C(19)	3.339(2)	C(9)	C(20)	3.563(2)
C(9)	C(24)[1)]	3.374(2)	C(9)	C(26)	3.428(2)

Table S12. Distances beyond the asymmetric unit out to 3.60 Å (continued)

atom	atom	distance	atom	atom	distance
C(10)	N(1)	2.858(2)	C(10)	C(8)	2.493(2)
C(10)	C(11)	2.233(2)	C(10)	C(12)	2.278(2)
C(10)	C(14)	2.427(2)	C(10)	C(15)	2.756(2)
C(10)	C(16)	2.380(2)	C(10)	C(19)	3.441(2)
C(10)	C(20)	3.267(2)	C(10)	C(23)[1)]	3.300(2)
C(10)	C(24)[1)]	3.556(2)	C(11)	O(2)[9)]	3.569(2)
C(11)	C(10)	2.233(2)	C(11)	C(13)	2.247(2)
C(11)	C(21)	3.472(2)	C(12)	N(2)	2.231(2)
C(12)	N(2)[9)]	3.471(2)	C(12)	C(10)	2.278(2)
C(12)	C(14)	2.624(2)	C(13)	N(2)	2.244(2)
C(13)	C(9)	2.478(2)	C(13)	C(11)	2.247(2)
C(13)	C(15)	2.398(2)	C(13)	C(16)	2.790(2)
C(14)	O(2)[10)]	3.479(2)	C(14)	N(2)	3.564(2)
C(14)	C(9)	2.855(2)	C(14)	C(10)	2.427(2)
C(14)	C(12)	2.624(2)	C(14)	C(16)	2.421(2)
C(15)	C(9)	2.448(2)	C(15)	C(10)	2.756(2)
C(15)	C(13)	2.398(2)	C(16)	O(1)	3.127(2)
C(16)	C(7)	3.120(2)	C(16)	C(8)	2.509(2)
C(16)	C(10)	2.380(2)	C(16)	C(13)	2.790(2)
C(16)	C(14)	2.421(2)	C(16)	C(17)	3.453(2)
C(16)	C(26)	3.392(2)	C(17)	O(1)	2.360(2)
C(17)	O(3)	3.181(2)	C(17)	N(1)	3.453(2)
C(17)	N(3)	2.496(2)	C(17)	C(1)	3.431(2)
C(17)	C(5)	3.308(2)	C(17)	C(6)	2.530(2)
C(17)	C(8)	2.572(2)	C(17)	C(9)	3.317(2)
C(17)	C(16)	3.453(2)	C(17)	C(26)	3.135(2)
C(18)	O(2)	2.948(2)	C(18)	O(3)	2.904(2)
C(18)	N(1)	3.568(2)	C(18)	C(6)	3.160(2)
C(18)	C(7)	2.611(2)	C(18)	C(8)	3.039(2)
C(18)	C(19)	2.551(2)	C(18)	C(26)	2.518(2)
C(19)	O(3)	3.472(2)	C(19)	N(1)	3.378(2)

C(19)	N(2)	3.512(2)	C(19)	C(2)[5)]	3.377(2)
C(19)	C(8)	3.262(2)	C(19)	C(9)	3.339(2)
C(19)	C(10)	3.441(2)	C(19)	C(18)	2.551(2)
C(19)	C(21)	2.604(2)	C(19)	C(25)	2.327(2)
C(19)	C(26)	2.305(2)	C(20)	O(2)	2.446(2)
C(20)	O(3)	3.508(2)	C(20)	N(2)	3.439(2)

Table S12. Distances beyond the asymmetric unit out to 3.60 Å (continued)

atom	atom	distance	atom	atom	distance
C(20)	N(3)	2.307(2)	C(20)	C(9)	3.563(2)
C(20)	C(10)	3.267(2)	C(20)	C(22)	2.367(2)
C(20)	C(23)	2.746(2)	C(20)	C(24)	2.422(2)
C(20)	C(26)	2.319(2)	C(21)	O(2)	3.159(2)
C(21)	N(2)	3.452(2)	C(21)	C(11)	3.472(2)
C(21)	C(19)	2.604(2)	C(21)	C(23)	2.425(2)
C(21)	C(24)	2.837(2)	C(21)	C(25)	2.415(2)
C(22)	O(2)[11)]	3.453(2)	C(22)	N(2)[5)]	3.491(2)
C(22)	C(20)	2.367(2)	C(22)	C(24)	2.425(2)
C(22)	C(25)	2.742(2)	C(23)	N(2)[5)]	3.190(2)
C(23)	C(10)[5)]	3.300(2)	C(23)	C(20)	2.746(2)
C(23)	C(21)	2.425(2)	C(23)	C(25)	2.365(2)
C(24)	O(3)	3.142(2)	C(24)	N(1)[5)]	3.329(2)
C(24)	C(3)[12)]	3.597(2)	C(24)	C(8)[5)]	3.329(2)
C(24)	C(9)[5)]	3.374(2)	C(24)	C(10)[5)]	3.556(2)
C(24)	C(20)	2.422(2)	C(24)	C(21)	2.837(2)
C(24)	C(22)	2.425(2)	C(24)	C(26)	2.597(2)
C(25)	O(2)	3.510(2)	C(25)	O(3)	2.440(2)
C(25)	N(1)[5)]	3.278(2)	C(25)	N(3)	2.299(2)
C(25)	C(1)[5)]	3.465(2)	C(25)	C(8)[5)]	3.584(2)
C(25)	C(19)	2.327(2)	C(25)	C(21)	2.415(2)
C(25)	C(22)	2.742(2)	C(25)	C(23)	2.365(2)
C(26)	O(2)	3.463(2)	C(26)	C(1)[5)]	3.457(2)
C(26)	C(6)[5)]	3.498(2)	C(26)	C(9)	3.428(2)
C(26)	C(16)	3.392(2)	C(26)	C(17)	3.135(2)

C(26)	C(18)	2.518(2)	C(26)	C(19)	2.305(2)
C(26)	C(20)	2.319(2)	C(26)	C(24)	2.597(2)

Symmetry Operators:

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

Table S13. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O(1)	H(3)[1)]	3.531	O(1)	H(4)	3.139
O(1)	H(8)[2)]	2.947	O(1)	H(9)	2.519
O(1)	H(10)	2.585	O(1)	H(10)[3)]	3.102
O(1)	H(11)	2.457	O(1)	H(13)[3)]	3.070
O(1)	H(17)[4)]	3.582	O(2)	H(1)[5)]	3.488
O(2)	H(5)[6)]	2.985	O(2)	H(7)[7)]	2.574
O(2)	H(12)	2.565	O(2)	H(13)	3.578
O(2)	H(14)	3.038	O(2)	H(15)[8)]	2.521
O(2)	H(2A)	3.20(2)	O(3)	H(3)[3)]	2.734
O(3)	H(4)[3)]	2.622	O(3)	H(9)	3.478
O(3)	H(11)	2.542	O(3)	H(13)	2.865
O(3)	H(17)	3.018	O(3)	H(1A)[5)]	1.99(2)
N(1)	H(1)	2.752	N(1)	H(5)[6)]	3.254
N(1)	H(6)[6)]	3.241	N(1)	H(12)	3.224
N(1)	H(1A)	3.492(19)	N(1)	H(2A)	2.23(2)
N(2)	H(5)	2.071	N(2)	H(6)	3.147
N(2)	H(6)[6)]	2.849	N(2)	H(14)	3.378
N(2)	H(15)[4)]	3.574	N(2)	H(16)[4)]	3.075
N(3)	H(10)	3.299	N(3)	H(11)	2.580
N(3)	H(12)	2.022	N(3)	H(13)	2.022
C(1)	H(1)	2.049	C(1)	H(2)	3.235

C(1)	H(4)	3.274	C(1)	H(5)[6)]	3.333
C(1)	H(12)	3.045	C(1)	H(1A)	3.37(2)
C(1)	H(2A)	3.52(2)	C(2)	H(2)	2.032
C(2)	H(3)	3.279	C(2)	H(5)[6)]	2.946
C(3)	H(1)	2.053	C(3)	H(3)	2.035
C(3)	H(4)	3.271	C(3)	H(7)[9)]	3.226
C(3)	H(13)[4)]	3.325	C(3)	H(16)[9)]	3.361
C(3)	H(17)[9)]	3.032	C(4)	H(1)	3.287
C(4)	H(2)	2.029	C(4)	H(4)	2.044
C(4)	H(11)[2)]	3.418	C(4)	H(13)[4)]	2.919
C(4)	H(17)[9)]	3.360	C(5)	H(2)	3.262
C(5)	H(3)	2.036	C(5)	H(9)[2)]	3.345
C(5)	H(10)	3.045	C(5)	H(11)[2)]	3.067
C(5)	H(12)	3.480	C(5)	H(13)[4)]	3.252
C(5)	H(1A)	3.16(2)	C(6)	H(1)	3.290
C(6)	H(3)	3.239	C(6)	H(4)	2.033

Table S13. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(6)	H(10)	2.695	C(6)	H(11)	3.361
C(6)	H(12)	2.767	C(6)	H(1A)	2.53(2)
C(7)	H(4)	2.886	C(7)	H(9)	2.779
C(7)	H(10)	2.072	C(7)	H(11)	2.072
C(7)	H(12)	2.711	C(7)	H(13)	3.414
C(7)	H(1A)	1.93(2)	C(8)	H(9)	2.683
C(8)	H(10)	3.398	C(8)	H(11)	2.868
C(8)	H(12)	3.060	C(8)	H(17)[4)]	3.254
C(8)	H(1A)	2.75(2)	C(8)	H(2A)	2.824(19)
C(9)	H(8)	3.295	C(9)	H(9)	2.038
C(9)	H(11)	3.178	C(9)	H(17)[4)]	3.105
C(9)	H(1A)	3.49(2)	C(9)	H(2A)	2.74(2)
C(10)	H(5)	3.144	C(10)	H(6)	3.198
C(10)	H(7)	3.291	C(10)	H(9)	3.244

C(10)	H(16)[4)]	3.042	C(10)	H(17)[4)]	3.508
C(10)	H(2A)	1.96(2)	C(11)	H(1)[10)]	2.910
C(11)	H(6)	2.067	C(11)	H(6)[6)]	3.367
C(11)	H(14)	3.321	C(11)	H(15)[4)]	3.573
C(11)	H(16)[4)]	3.276	C(11)	H(2A)	2.02(2)
C(11)	H(2A)[10)]	3.33(2)	C(12)	H(1)[10)]	3.284
C(12)	H(5)	2.056	C(12)	H(5)[10)]	3.544
C(12)	H(7)	2.907	C(12)	H(16)[4)]	3.393
C(12)	H(2A)	3.06(2)	C(12)	H(2A)[10)]	2.79(2)
C(13)	H(5)	3.166	C(13)	H(6)	2.138
C(13)	H(7)	2.058	C(13)	H(8)	3.256
C(13)	H(16)[4)]	3.243	C(13)	H(2A)	3.05(2)
C(14)	H(2)[11)]	2.975	C(14)	H(6)	3.000
C(14)	H(8)	2.017	C(14)	H(9)	3.266
C(14)	H(12)[12)]	3.271	C(15)	H(2)[11)]	3.340
C(15)	H(7)	2.028	C(15)	H(9)	2.041
C(15)	H(10)[3)]	3.355	C(15)	H(12)[12)]	3.250
C(16)	H(4)[3)]	3.231	C(16)	H(7)	3.279
C(16)	H(8)	2.043	C(16)	H(10)[3)]	2.959
C(16)	H(11)	2.928	C(16)	H(17)[4)]	3.444
C(16)	H(1A)	3.57(2)	C(17)	H(4)	3.297
C(17)	H(4)[3)]	3.495	C(17)	H(9)	2.971
C(17)	H(9)[2)]	3.212	C(17)	H(12)	2.072

Table S13. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(17)	H(13)	2.072	C(17)	H(1A)	3.15(2)
C(18)	H(7)[7)]	3.543	C(18)	H(8)[7)]	3.358
C(18)	H(10)	2.061	C(18)	H(11)	2.061
C(19)	H(1)[5)]	3.351	C(19)	H(7)[7)]	3.447
C(19)	H(12)	2.608	C(19)	H(13)	3.157
C(19)	H(14)	2.859	C(19)	H(15)[8)]	3.244
C(19)	H(2A)	3.38(2)	C(20)	H(1)[5)]	3.469

C(20)	H(14)	2.044	C(20)	H(15)	3.229
C(20)	H(15)[8)]	3.580	C(20)	H(17)	3.283
C(21)	H(6)[8)]	3.040	C(21)	H(15)	2.035
C(21)	H(15)[8)]	3.358	C(21)	H(16)	3.275
C(22)	H(6)[8)]	3.045	C(22)	H(14)	2.054
C(22)	H(14)[13)]	3.132	C(22)	H(16)	2.032
C(22)	H(17)	3.288	C(22)	H(2A)[5)]	3.34(2)
C(23)	H(2)[11)]	3.059	C(23)	H(14)	3.287
C(23)	H(14)[13)]	3.114	C(23)	H(15)	2.033
C(23)	H(17)	2.054	C(23)	H(2A)[5)]	3.26(2)
C(24)	H(2)[11)]	2.878	C(24)	H(15)	3.274
C(24)	H(16)	2.033	C(24)	H(1A)[5)]	3.54(2)
C(25)	H(14)	3.277	C(25)	H(16)	3.228
C(25)	H(17)	2.047	C(25)	H(1A)[5)]	3.41(2)
C(26)	H(4)[3)]	3.491	C(26)	H(9)	3.479
C(26)	H(11)	2.750	C(26)	H(12)	3.305
C(26)	H(13)	2.765	C(26)	H(17)	2.854
C(26)	H(1A)[5)]	2.79(2)	H(1)	O(2)[4)]	3.488
H(1)	N(1)	2.752	H(1)	C(1)	2.049
H(1)	C(3)	2.053	H(1)	C(4)	3.287
H(1)	C(6)	3.290	H(1)	C(11)[6)]	2.910
H(1)	C(12)[6)]	3.284	H(1)	C(19)[4)]	3.351
H(1)	C(20)[4)]	3.469	H(1)	H(2)	2.348
H(1)	H(5)[6)]	2.348	H(1)	H(6)[6)]	3.123
H(1)	H(14)[6)]	3.173	H(1)	H(15)[6)]	3.090
H(1)	H(16)[9)]	3.374	H(2)	C(1)	3.235
H(2)	C(2)	2.032	H(2)	C(4)	2.029
H(2)	C(5)	3.262	H(2)	C(14)[9)]	2.975
H(2)	C(15)[9)]	3.340	H(2)	C(23)[9)]	3.059
H(2)	C(24)[9)]	2.878	H(2)	H(1)	2.348

Table S13. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance

H(2)	H(3)	2.324	H(2)	H(7)[9)]	2.534
H(2)	H(8)[9)]	3.193	H(2)	H(16)[9)]	2.801
H(2)	H(17)[9)]	2.483	H(3)	O(1)[14)]	3.531
H(3)	O(3)[2)]	2.734	H(3)	C(2)	3.279
H(3)	C(3)	2.035	H(3)	C(5)	2.036
H(3)	C(6)	3.239	H(3)	H(2)	2.324
H(3)	H(4)	2.346	H(3)	H(8)[9)]	3.139
H(3)	H(11)[2)]	3.176	H(3)	H(13)[4)]	2.987
H(3)	H(17)[9)]	3.113	H(3)	H(1A)[14)]	3.020
H(4)	O(1)	3.139	H(4)	O(3)[2)]	2.622
H(4)	C(1)	3.274	H(4)	C(3)	3.271
H(4)	C(4)	2.044	H(4)	C(6)	2.033
H(4)	C(7)	2.886	H(4)	C(16)[2)]	3.231
H(4)	C(17)	3.297	H(4)	C(17)[2)]	3.495
H(4)	C(26)[2)]	3.491	H(4)	H(3)	2.346
H(4)	H(8)[2)]	3.327	H(4)	H(9)[2)]	2.542
H(4)	H(10)	2.780	H(4)	H(11)[2)]	2.513
H(4)	H(13)[4)]	3.518	H(4)	H(1A)	3.190
H(5)	O(2)[10)]	2.985	H(5)	N(1)[10)]	3.254
H(5)	N(2)	2.071	H(5)	C(1)[10)]	3.333
H(5)	C(2)[10)]	2.946	H(5)	C(10)	3.144
H(5)	C(12)	2.056	H(5)	C(12)[6)]	3.544
H(5)	C(13)	3.166	H(5)	H(1)[10)]	2.348
H(5)	H(6)	2.467	H(5)	H(6)[6)]	3.171
H(5)	H(7)[6)]	3.477	H(5)	H(14)	3.448
H(5)	H(15)[4)]	3.491	H(5)	H(2A)	2.455
H(5)	H(2A)[10)]	3.270	H(6)	N(1)[10)]	3.241
H(6)	N(2)	3.147	H(6)	N(2)[10)]	2.849
H(6)	C(10)	3.198	H(6)	C(11)	2.067
H(6)	C(11)[10)]	3.367	H(6)	C(13)	2.138
H(6)	C(14)	3.000	H(6)	C(21)[13)]	3.040
H(6)	C(22)[13)]	3.045	H(6)	H(1)[10)]	3.123
H(6)	H(5)	2.467	H(6)	H(5)[10)]	3.171
H(6)	H(7)	2.982	H(6)	H(14)[13)]	3.040

H(6)	H(15)[13)]	3.036	H(6)	H(2A)[10)]	2.173
H(7)	O(2)[12)]	2.574	H(7)	C(3)[11)]	3.226
H(7)	C(10)	3.291	H(7)	C(12)	2.907

Table S13. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(7)	C(13)	2.058	H(7)	C(15)	2.028
H(7)	C(16)	3.279	H(7)	C(18)[12)]	3.543
H(7)	C(19)[12)]	3.447	H(7)	H(2)[11)]	2.534
H(7)	H(5)[10)]	3.477	H(7)	H(6)	2.982
H(7)	H(8)	2.320	H(7)	H(12)[12)]	2.740
H(7)	H(15)[13)]	3.409	H(8)	O(1)[3)]	2.947
H(8)	C(9)	3.295	H(8)	C(13)	3.256
H(8)	C(14)	2.017	H(8)	C(16)	2.043
H(8)	C(18)[12)]	3.358	H(8)	H(2)[11)]	3.193
H(8)	H(3)[11)]	3.139	H(8)	H(4)[3)]	3.327
H(8)	H(7)	2.320	H(8)	H(9)	2.330
H(8)	H(10)[3)]	3.022	H(8)	H(12)[12)]	2.689
H(8)	H(13)[12)]	3.110	H(8)	H(1A)[3)]	3.302
H(9)	O(1)	2.519	H(9)	O(3)	3.478
H(9)	C(5)[3)]	3.345	H(9)	C(7)	2.779
H(9)	C(8)	2.683	H(9)	C(9)	2.038
H(9)	C(10)	3.244	H(9)	C(14)	3.266
H(9)	C(15)	2.041	H(9)	C(17)	2.971
H(9)	C(17)[3)]	3.212	H(9)	C(26)	3.479
H(9)	H(4)[3)]	2.542	H(9)	H(8)	2.330
H(9)	H(10)[3)]	2.230	H(9)	H(11)	2.328
H(9)	H(1A)	3.087	H(10)	O(1)	2.585
H(10)	O(1)[2)]	3.102	H(10)	N(3)	3.299
H(10)	C(5)	3.045	H(10)	C(6)	2.695
H(10)	C(7)	2.072	H(10)	C(8)	3.398
H(10)	C(15)[2)]	3.355	H(10)	C(16)[2)]	2.959
H(10)	C(18)	2.061	H(10)	H(4)	2.780

H(10)	H(8)[2)]	3.022	H(10)	H(9)[2)]	2.230
H(10)	H(11)	1.595	H(10)	H(12)	2.466
H(10)	H(13)	2.242	H(10)	H(1A)	3.334
H(10)	H(1A)[2)]	3.458	H(11)	O(1)	2.457
H(11)	O(3)	2.542	H(11)	N(3)	2.580
H(11)	C(4)[3)]	3.418	H(11)	C(5)[3)]	3.067
H(11)	C(6)	3.361	H(11)	C(7)	2.072
H(11)	C(8)	2.868	H(11)	C(9)	3.178
H(11)	C(16)	2.928	H(11)	C(18)	2.061
H(11)	C(26)	2.750	H(11)	H(3)[3)]	3.176

Table S13. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(11)	H(4)[3)]	2.513	H(11)	H(9)	2.328
H(11)	H(10)	1.595	H(11)	H(12)	2.848
H(11)	H(13)	2.456	H(11)	H(1A)	3.333
H(12)	O(2)	2.565	H(12)	N(1)	3.224
H(12)	N(3)	2.022	H(12)	C(1)	3.045
H(12)	C(5)	3.480	H(12)	C(6)	2.767
H(12)	C(7)	2.711	H(12)	C(8)	3.060
H(12)	C(14)[7)]	3.271	H(12)	C(15)[7)]	3.250
H(12)	C(17)	2.072	H(12)	C(19)	2.608
H(12)	C(26)	3.305	H(12)	H(7)[7)]	2.740
H(12)	H(8)[7)]	2.689	H(12)	H(10)	2.466
H(12)	H(11)	2.848	H(12)	H(13)	1.600
H(13)	O(1)[2)]	3.070	H(13)	O(2)	3.578
H(13)	O(3)	2.865	H(13)	N(3)	2.022
H(13)	C(3)[5)]	3.325	H(13)	C(4)[5)]	2.919
H(13)	C(5)[5)]	3.252	H(13)	C(7)	3.414
H(13)	C(17)	2.072	H(13)	C(19)	3.157
H(13)	C(26)	2.765	H(13)	H(3)[5)]	2.987
H(13)	H(4)[5)]	3.518	H(13)	H(8)[7)]	3.110
H(13)	H(10)	2.242	H(13)	H(11)	2.456
H(13)	H(12)	1.600	H(13)	H(1A)[2)]	3.030

H(14)	O(2)	3.038	H(14)	N(2)	3.378
H(14)	C(11)	3.321	H(14)	C(19)	2.859
H(14)	C(20)	2.044	H(14)	C(22)	2.054
H(14)	C(22)[8)]	3.132	H(14)	C(23)	3.287
H(14)	C(23)[8)]	3.114	H(14)	C(25)	3.277
H(14)	H(1)[10)]	3.173	H(14)	H(5)	3.448
H(14)	H(6)[8)]	3.040	H(14)	H(15)	2.354
H(14)	H(15)[8)]	2.711	H(14)	H(16)[8)]	2.671
H(14)	H(2A)	3.534	H(15)	O(2)[13)]	2.521
H(15)	N(2)[5)]	3.574	H(15)	C(11)[5)]	3.573
H(15)	C(19)[13)]	3.244	H(15)	C(20)	3.229
H(15)	C(20)[13)]	3.580	H(15)	C(21)	2.035
H(15)	C(21)[13)]	3.358	H(15)	C(23)	2.033
H(15)	C(24)	3.274	H(15)	H(1)[10)]	3.090
H(15)	H(5)[5)]	3.491	H(15)	H(6)[8)]	3.036
H(15)	H(7)[8)]	3.409	H(15)	H(14)	2.354

Table S13. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(15)	H(14)[13)]	2.711	H(15)	H(16)	2.322
H(15)	H(2A)[5)]	3.508	H(16)	N(2)[5)]	3.075
H(16)	C(3)[11)]	3.361	H(16)	C(10)[5)]	3.042
H(16)	C(11)[5)]	3.276	H(16)	C(12)[5)]	3.393
H(16)	C(13)[5)]	3.243	H(16)	C(21)	3.275
H(16)	C(22)	2.032	H(16)	C(24)	2.033
H(16)	C(25)	3.228	H(16)	H(1)[11)]	3.374
H(16)	H(2)[11)]	2.801	H(16)	H(14)[13)]	2.671
H(16)	H(15)	2.322	H(16)	H(17)	2.353
H(16)	H(2A)[5)]	3.389	H(17)	O(1)[5)]	3.582
H(17)	O(3)	3.018	H(17)	C(3)[11)]	3.032
H(17)	C(4)[11)]	3.360	H(17)	C(8)[5)]	3.254
H(17)	C(9)[5)]	3.105	H(17)	C(10)[5)]	3.508
H(17)	C(16)[5)]	3.444	H(17)	C(20)	3.283
H(17)	C(22)	3.288	H(17)	C(23)	2.054

H(17)	C(25)	2.047	H(17)	C(26)	2.854
H(17)	H(2)[11)]	2.483	H(17)	H(3)[11)]	3.113
H(17)	H(16)	2.353	H(17)	H(1A)[5)]	3.107
H(1A)	O(3)[4)]	1.99(2)	H(1A)	N(1)	3.492(19)
H(1A)	C(1)	3.37(2)	H(1A)	C(5)	3.16(2)
H(1A)	C(6)	2.53(2)	H(1A)	C(7)	1.93(2)
H(1A)	C(8)	2.75(2)	H(1A)	C(9)	3.49(2)
H(1A)	C(16)	3.57(2)	H(1A)	C(17)	3.15(2)
H(1A)	C(24)[4)]	3.54(2)	H(1A)	C(25)[4)]	3.41(2)
H(1A)	C(26)[4)]	2.79(2)	H(1A)	H(3)[1)]	3.020
H(1A)	H(4)	3.190	H(1A)	H(8)[2)]	3.302
H(1A)	H(9)	3.087	H(1A)	H(10)	3.334
H(1A)	H(10)[3)]	3.458	H(1A)	H(11)	3.333
H(1A)	H(13)[3)]	3.030	H(1A)	H(17)[4)]	3.107
H(2A)	O(2)	3.20(2)	H(2A)	N(1)	2.23(2)
H(2A)	C(1)	3.52(2)	H(2A)	C(8)	2.824(19)
H(2A)	C(9)	2.74(2)	H(2A)	C(10)	1.96(2)
H(2A)	C(11)	2.02(2)	H(2A)	C(11)[6)]	3.33(2)
H(2A)	C(12)	3.06(2)	H(2A)	C(12)[6)]	2.79(2)
H(2A)	C(13)	3.05(2)	H(2A)	C(19)	3.38(2)
H(2A)	C(22)[4)]	3.34(2)	H(2A)	C(23)[4)]	3.26(2)
H(2A)	H(5)	2.455	H(2A)	H(5)[6)]	3.270

Table S13. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

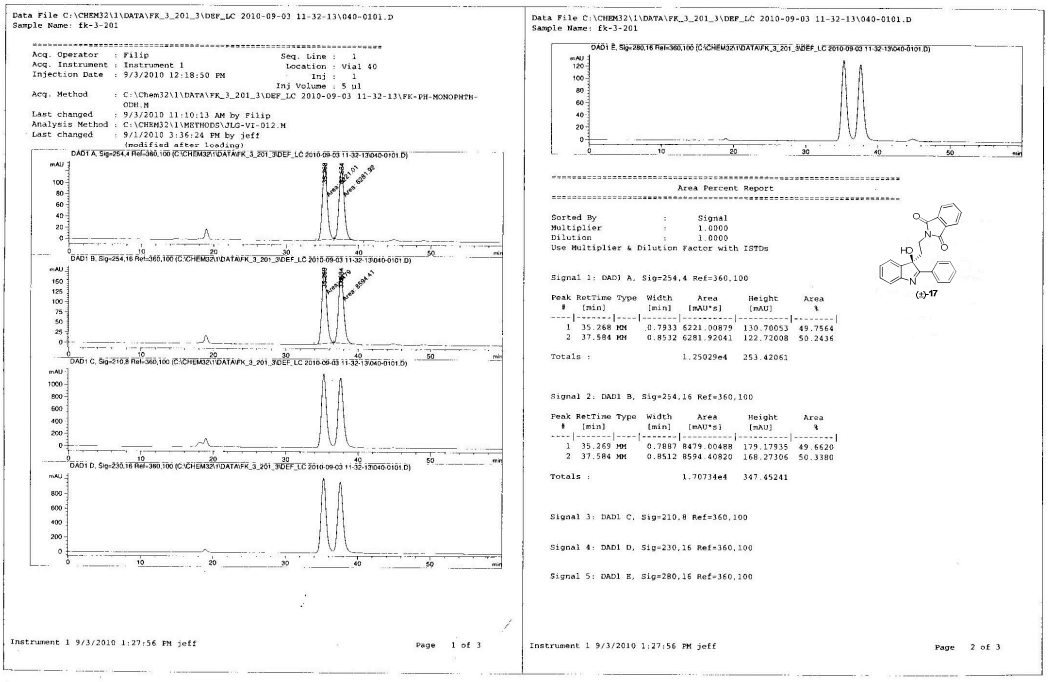
atom	atom	distance	atom	atom	distance
H(2A)	H(6)[6)]	2.173	H(2A)	H(14)	3.534
H(2A)	H(15)[4)]	3.508	H(2A)	H(16)[4)]	3.389

Symmetry Operators:

(1) -X+2,Y+1/2,-Z+2	(2) -X+1,Y+1/2-1,-Z+2
(3) -X+1,Y+1/2,-Z+2	(4) X+1,Y,Z
(5) X-1,Y,Z	(6) -X+1,Y+1/2-1,-Z+1
(7) X,Y-1,Z	(8) -X,Y+1/2-1,-Z+1
(9) X+1,Y-1,Z	(10) -X+1,Y+1/2,-Z+1
(11) X-1,Y+1,Z	(12) X,Y+1,Z
(13) -X,Y+1/2,-Z+1	(14) -X+2,Y+1/2-1,-Z+2

L. Chromatograms.

OD-H, 0.2 ml/min, 80:20 Hex: EtOH



Data File C:\CHEM32\1\DATA\FK_3_201_3\DEF_LC 2010-09-03 11-32-13\040-0101.D
Sample Name: fk-3-201

Acq. Operator : Filip
Acq. Instrument : Instrument 1
Injection Date : 9/3/2010 12:18:50 PM
Seq. Line : 1
Location : Vial 40
Inj : 1
Inj Volume : 5 µl
Acq. Method : C:\Chem32\1\DATA\FK_3_201_3\DEF_LC 2010-09-03 11-32-13\FK-PH-MONOPHTH-ODH.M
Last changed : 9/3/2010 11:10:13 AM by Filip
Analysis Method : C:\CHEM32\1\METHODS\JLG-VI-012.M
Last changed : 9/1/2010 3:36:24 PM by jeff
(modified after loading)

DAD1 A, Sig=254,4 Ref=360,100 (C:\CHEM32\1\DATA\FK_3_201_3\DEF_LC 2010-09-03 11-32-13\040-0101.D)
DAD1 B, Sig=254,16 Ref=360,100 (C:\CHEM32\1\DATA\FK_3_201_3\DEF_LC 2010-09-03 11-32-13\040-0101.D)
DAD1 C, Sig=210,8 Ref=360,100 (C:\CHEM32\1\DATA\FK_3_201_3\DEF_LC 2010-09-03 11-32-13\040-0101.D)
DAD1 D, Sig=230,16 Ref=360,100 (C:\CHEM32\1\DATA\FK_3_201_3\DEF_LC 2010-09-03 11-32-13\040-0101.D)

Instrument 1 9/3/2010 1:27:56 PM jeff Page 1 of 3

Data File C:\CHEM32\1\DATA\FK_3_201_3\DEF_LC 2010-09-03 11-32-13\040-0101.D
Sample Name: fk-3-201

DAD1 E, Sig=280,16 Ref=360,100 (C:\CHEM32\1\DATA\FK_3_201_3\DEF_LC 2010-09-03 11-32-13\040-0101.D)

Area Percent Report

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

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	35.268	MM	0.7933	6221.00879	130.70053	49.7564
2	37.584	MM	0.8532	6281.92041	122.72008	50.2436

Totals : 1.25029e4 253.42061

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	35.269	MM	0.7987	8479.00488	179.17935	49.6520
2	37.584	MM	0.8512	8594.40820	168.27306	50.3380

Totals : 1.70734e4 347.45241

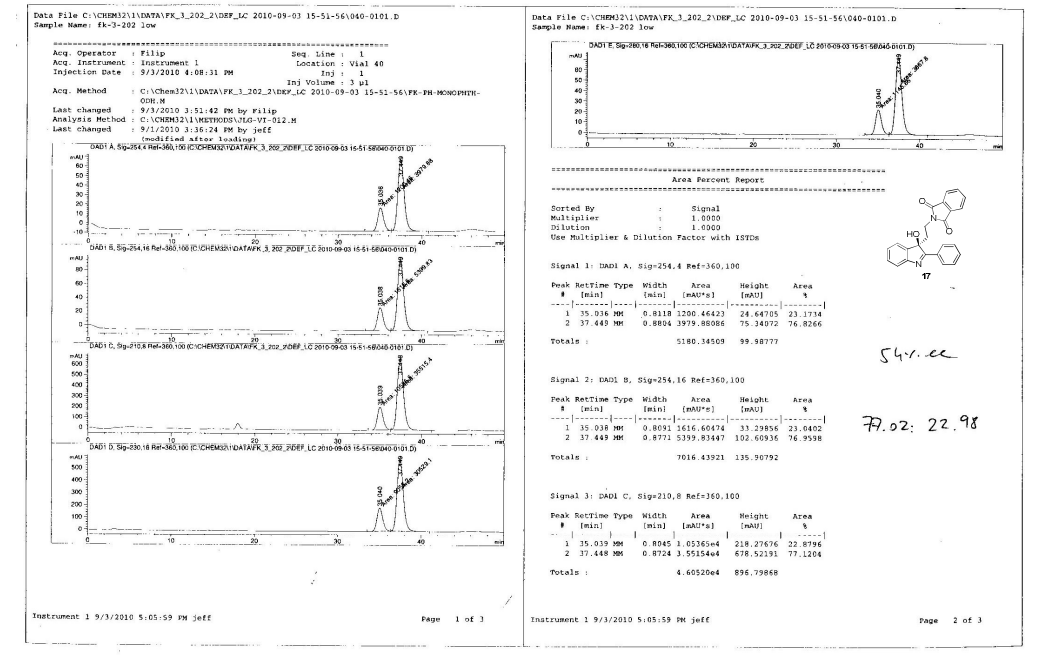
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Signal 4: DAD1 D, Sig=230,16 Ref=360,100

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

(±)-17

Instrument 1 9/3/2010 1:27:56 PM jeff Page 2 of 3



Data File C:\CHEM32\1\DATA\FK_3_202_2\DEF_LC 2010-09-03 15-51-56\040-0101.D
Sample Name: fk-3-202 low

Acq. Operator : Filip
Acq. Instrument : Instrument 1
Injection Date : 9/3/2010 4:08:31 PM
Seq. Line : 1
Location : Vial 40
Inj : 1
Inj Volume : 3 µl
Acq. Method : C:\Chem32\1\DATA\FK_3_202_2\DEF_LC 2010-09-03 15-51-56\FK-PH-MONOPHTH-ODH.M
Last changed : 9/3/2010 3:51:42 PM by Filip
Analysis Method : C:\CHEM32\1\METHODS\JLG-VI-012.M
Last changed : 9/1/2010 3:36:24 PM by jeff
(modified after loading)

DAD1 A, Sig=254,4 Ref=360,100 (C:\CHEM32\1\DATA\FK_3_202_2\DEF_LC 2010-09-03 15-51-56\040-0101.D)
DAD1 B, Sig=254,16 Ref=360,100 (C:\CHEM32\1\DATA\FK_3_202_2\DEF_LC 2010-09-03 15-51-56\040-0101.D)
DAD1 C, Sig=210,8 Ref=360,100 (C:\CHEM32\1\DATA\FK_3_202_2\DEF_LC 2010-09-03 15-51-56\040-0101.D)
DAD1 D, Sig=230,16 Ref=360,100 (C:\CHEM32\1\DATA\FK_3_202_2\DEF_LC 2010-09-03 15-51-56\040-0101.D)

Instrument 1 9/3/2010 5:05:59 PM jeff Page 1 of 3

Data File C:\CHEM32\1\DATA\FK_3_202_2\DEF_LC 2010-09-03 15-51-56\040-0101.D
Sample Name: fk-3-202 low

DAD1 E, Sig=280,16 Ref=360,100 (C:\CHEM32\1\DATA\FK_3_202_2\DEF_LC 2010-09-03 15-51-56\040-0101.D)

Area Percent Report

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

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	35.036	MM	0.8118	1200.46423	24.64705	23.1734
2	37.449	MM	0.8804	3979.86086	75.34972	76.8266

Totals : 5180.34509 99.98777

54% ee

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	35.038	MM	0.8091	1616.60474	33.29856	23.0402
2	37.449	MM	0.8771	5399.83447	102.60936	76.9598

Totals : 7016.43921 135.90792

77.02: 22.98

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	35.039	MM	0.8045	1.05365e4	218.27676	22.8796
2	37.448	MM	0.8724	3.55154e4	678.52191	77.1204

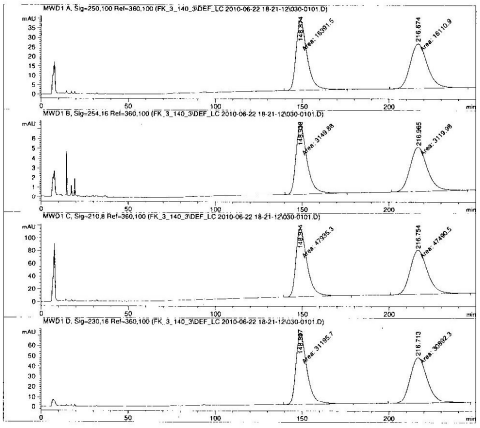
Totals : 4.60520e4 896.79868

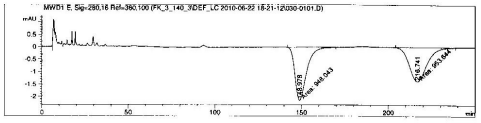
(±)-17

Instrument 1 9/3/2010 5:05:59 PM jeff Page 2 of 3

Data File C:\CHEM32\2\DATA\FK_3_140_3\DEF_LC 2010-06-22 18-21-12\030-0101.D
Sample Name: fk-3-140

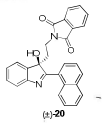
Acq. Operator : Filip
Acq. Instrument : Instrument 2
Injection Date : 6/22/2010 6:28:49 PM
Seq. Line : 1
Location : Vial 30
Inj : 1
Inj Volume : 15 ul
Acq. Method : C:\Chem32\2\DATA\FK_3_140_3\DEF_LC 2010-06-22 18-21-12\FK-NPHTH-PHTH.M
Last changed : 6/22/2010 5:19:47 PM by Filip
Analysis Method : C:\CHEM32\2\DATA\FK_3_140_3\DEF_LC 2010-06-22 18-21-12\030-0101.D\DA.M (FK-NPHTH-PHTH.M)
Last changed : 6/22/2010 5:19:47 PM by Filip
Method Info : chiralcel OD





Area Percent Report

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


(±)-20

Signal 1: MWD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	148.874	MM	7.4108	1.63915e4	36.86395	50.4317
2	216.674	MM	11.3476	1.61109e4	23.66274	49.5683
Totals :				3.25024e4	60.52669	

Signal 2: MWD1 B, Sig=254,16 Ref=360,100

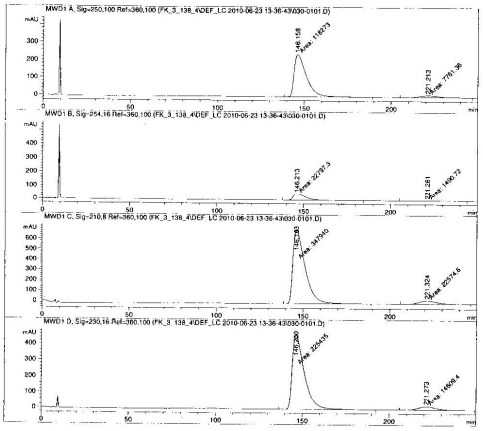
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	148.938	MM	7.4193	3149.97364	7.07585	50.2384
2	216.965	MM	11.4595	3119.97974	4.53770	49.7616
Totals :				6269.85937	11.61355	

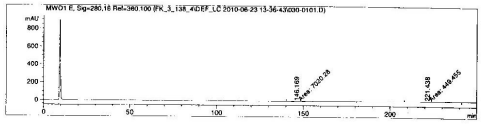
Signal 3: MWD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	148.914	MM	7.4173	4.79353e4	107.71022	50.2330
2	216.754	MM	11.4414	4.74905e4	69.17962	49.7670
Totals :				9.54258e4	176.88984	

Data File C:\CHEM32\2\DATA\FK_3_138_4\DEF_LC 2010-06-23 13-36-43\030-0101.D
Sample Name: fk-3-138

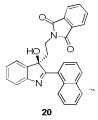
Acq. Operator : Filip
Acq. Instrument : Instrument 2
Injection Date : 6/23/2010 1:43:56 PM
Seq. Line : 1
Location : Vial 30
Inj : 1
Inj Volume : 15 ul
Acq. Method : C:\Chem32\2\DATA\FK_3_138_4\DEF_LC 2010-06-23 13-36-43\FK-NPHTH-PHTH.M
Last changed : 6/22/2010 5:19:47 PM by Filip
Analysis Method : C:\CHEM32\2\DATA\FK_3_138_4\DEF_LC 2010-06-23 13-36-43\030-0101.D\DA.M (FK-NPHTH-PHTH.M)
Last changed : 6/22/2010 5:19:47 PM by Filip
Method Info : chiralcel OD





Area Percent Report

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


20

Signal 1: MWD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	146.158	MM	8.1471	1.18273e5	241.95290	93.8418
2	221.213	MM	11.6939	7761.36377	11.06187	6.1582
Totals :				1.26034e5	253.01476	

Signal 2: MWD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	146.213	MM	8.0653	2.27973e4	47.10973	93.8623
2	221.261	MM	11.6748	1490.72253	2.12813	6.1377
Totals :				2.42880e4	49.23785	

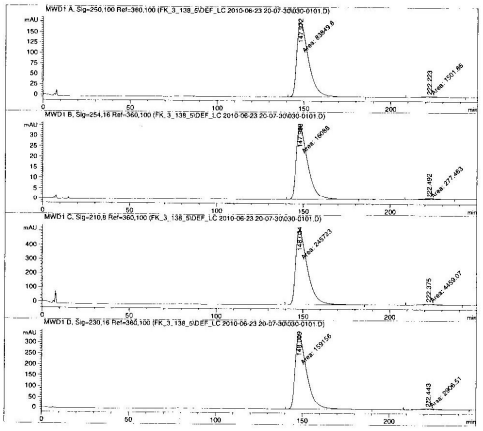
94:6

Signal 3: MWD1 C, Sig=210,8 Ref=360,100

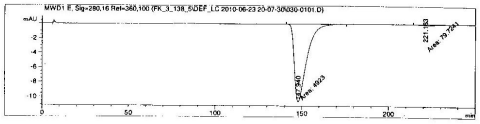
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	146.163	MM	8.1088	3.47940e5	715.14539	93.9072
2	221.324	MM	11.6944	2.25746e4	32.17287	6.0928
Totals :				3.70515e5	747.31826	

Data File C:\CHEM32\2\DATA\FK_3_138_5\DEF_LC 2010-06-23 20-07-30\030-0101.D
Sample Name: fk-3-138 recrystallized

Acq. Operator	: Filip	Seq. Line	: 1
Acq. Instrument	: Instrument 2	Location	: Vial 30
Injection Date	: 6/23/2010 8:14:32 PM	Inj	: 1
		Inj Volume	: 15 µl
Acq. Method	: C:\Chem32\2\DATA\FK_3_138_5\DEF_LC 2010-06-23 20-07-30\FK-NPHTH-PHTH.M		
Last changed	: 6/22/2010 5:19:47 PM by Filip		
Analysis Method	: C:\CHEM32\2\DATA\FK_3_138_5\DEF_LC 2010-06-23 20-07-30\030-0101.D\DA.M (FK-NPHTH-PHTH.M)		
Last changed	: 6/22/2010 5:19:47 PM by Filip		
Method Info	: chiralcel OD		



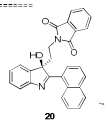
Data File C:\CHEM32\2\DATA\FK_3_138_5\DEF_LC 2010-06-23 20-07-30\030-0101.D
Sample Name: fk-3-138 recrystallized



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs



20

Signal 1: MWD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	147.972	MM	7.7946	8.38498e4	179.28931	98.2404
2	222.223	MM	10.8480	1501.85876	2.30743	1.7596
Totals :				8.53516e4	181.59673	

98.5 : 1.5

Signal 2: MWD1 B, Sig=254,16 Ref=360,100

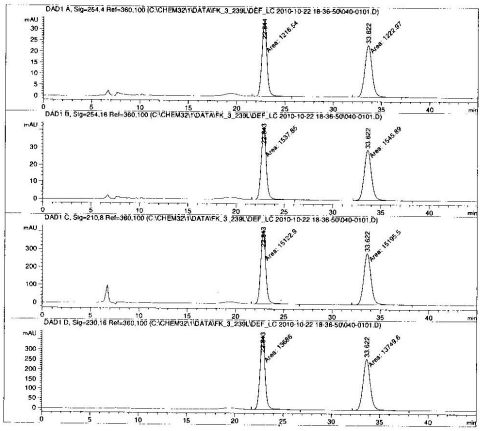
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	147.988	MM	7.7623	1.60880e4	34.54707	98.3046
2	222.492	MM	10.6769	277.46274	4.33120e-1	1.6954
Totals :				1.63654e4	34.97619	

Signal 3: MWD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	148.034	MM	7.7705	2.45723e5	527.04474	98.2177
2	222.375	MM	11.1815	4459.07080	6.64647	1.7823
Totals :				2.50182e5	533.69121	

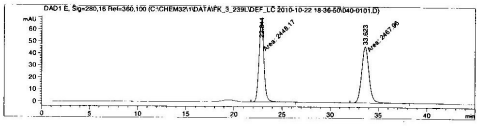
Data File C:\CHEM32\1\DATA\FK_3_239L\DEF_LC 2010-10-22 18-36-50\040-0101.D
Sample Name: fk-3-239 low

Acq. Operator : Filip
Acq. Instrument : Instrument 1
Injection Date : 10/22/2010 6:53:50 PM
Seq. Line : 1
Location : Vial 40
Inj : 1
Inj Volume : 6 µl
Acq. Method : C:\Chem32\1\DATA\FK_3_239L\DEF_LC 2010-10-22 18-36-50\FK-PHCF3-MONOPHTH-IA2.M
Last changed : 10/22/2010 5:53:52 PM by Kai
Analysis Method : C:\CHEM32\2\DATA\FK_3_TOSRAC\DEF_LC 2011-05-07 21-10-34\040-0101.D\DA.M (FK-HYDROXIND_TOS_OD_FIN.M)
Last changed : 5/8/2011 2:40:05 PM by Filip (modified after loading)
Method Info : chiralcel OD



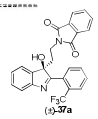
Instrument 2 5/8/2011 2:42:01 PM Filip Page 1 of 3

Data File C:\CHEM32\1\DATA\FK_3_239L\DEF_LC 2010-10-22 18-36-50\040-0101.D
Sample Name: fk-3-239 low



Area Percent Report

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



(±)-37a

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.844	MM	0.6088	1216.54041	33.30658	49.8682
2	33.622	MM	0.8772	1222.97314	23.23499	50.1318
Totals :				2439.51355	56.54157	

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.843	MM	0.6105	1537.84985	41.98073	49.8696
2	33.622	MM	0.8795	1545.89063	29.29633	50.1304
Totals :				3083.74048	71.27706	

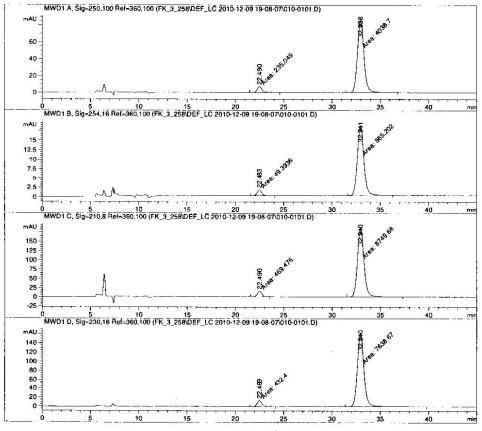
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.843	MM	0.6095	1.51229e4	413.55371	49.8804
2	33.622	MM	0.8796	1.51955e4	287.91251	50.1196
Totals :				3.03184e4	701.46622	

Instrument 2 5/8/2011 2:42:01 PM Filip Page 2 of 3

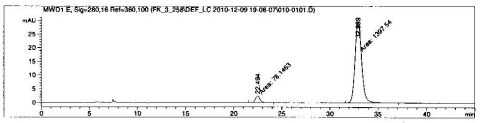
Data File C:\CHEM32\2\DATA\FK_3_258\DEF_LC 2010-12-09 19-08-07\010-0101.D
Sample Name: fk-3-258

Acq. Operator : Filip
Acq. Instrument : Instrument 2
Injection Date : 12/9/2010 7:29:18 PM
Seq. Line : 1
Location : Vial 10
Inj : 1
Inj Volume : 4 µl
Acq. Method : C:\Chem32\2\DATA\FK_3_258\DEF_LC 2010-12-09 19-08-07\FK-PHCF3-MONOPHTH-IA2.M
Last changed : 12/6/2010 7:53:30 PM by Filip
Analysis Method : C:\CHEM32\2\DATA\FK_3_TOSRAC\DEF_LC 2011-05-07 21-10-34\040-0101.D\DA.M (FK-HYDROXIND_TOS_OD_FIN.M)
Last changed : 5/8/2011 2:34:55 PM by Filip (modified after loading)
Method Info : chiralcel OD



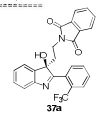
Instrument 2 5/8/2011 2:40:05 PM Filip Page 1 of 3

Data File C:\CHEM32\2\DATA\FK_3_258\DEF_LC 2010-12-09 19-08-07\010-0101.D
Sample Name: fk-3-258



Area Percent Report

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



37a

Signal 1: MWD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.490	MM	0.5628	235.04945	6.96029	5.4998
2	32.938	MM	0.7852	4038.69678	85.72643	94.5002
Totals :				4273.74623	92.68671	

95 : 5 e.r.

Signal 2: MWD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.483	MM	0.5480	49.39363	1.50222	5.4006
2	32.941	MM	0.7832	865.20197	18.41213	94.5994
Totals :				914.59559	19.91435	

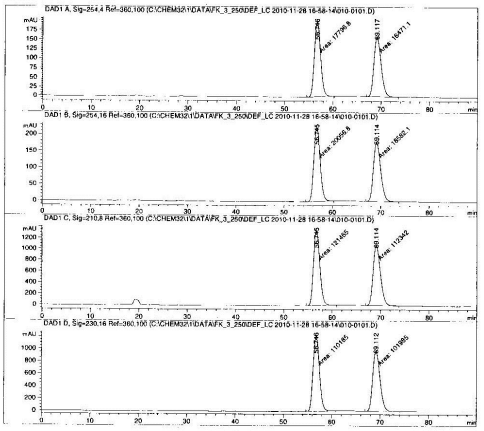
Signal 3: MWD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.490	MM	0.5258	469.47638	14.88112	5.0924
2	32.940	MM	0.7861	8749.67969	185.50757	94.9076
Totals :				9219.15607	200.38869	

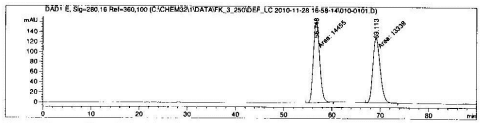
Instrument 2 5/8/2011 2:40:05 PM Filip Page 2 of 3

Data File C:\CHEM32\1\DATA\FK_3_250\DEF_LC 2010-11-28 16-58-14\010-0101.D
Sample Name: fk-3-250

Acq. Operator	: Filip
Acq. Instrument	: Instrument 1
Injection Date	: 11/28/2010 5:15:20 PM
Seq. Line	: 1
Location	: Vial 10
Inj	: 1
Inj Volume	: 7 µl
Acq. Method	: C:\Chem32\1\DATA\FK_3_250\DEF_LC 2010-11-28 16-58-14\FK-PH-MONOPHTH-ODH.M
Last changed	: 11/28/2010 3:06:27 PM by Filip
Analysis Method	: C:\CHEM32\2\METHODS\FK-MONOPHTH-CF3_FIN.M
Last changed	: 11/24/2010 4:34:13 PM by Filip
Method Info	: chiralcel OJ-H



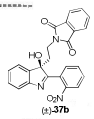
Data File C:\CHEM32\1\DATA\FK_3_250\DEF_LC 2010-11-28 16-58-14\010-0101.D
Sample Name: fk-3-250



Area Percent Report

Sorted By	: Signal
Multiplier	: 1.0000
Dilution	: 1.0000

Use Multiplier & Dilution Factor with ISTDs


(±)-37b

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	56.746	MM	1.5021	1.77968e4	197.47076	51.9343
2	69.117	MM	1.7377	1.64711e4	157.98227	48.0657
Totals :				3.42680e4	355.45303	

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	56.745	MM	1.5032	2.00568e4	222.38306	51.9083
2	69.114	MM	1.7398	1.85821e4	178.00981	48.0917
Totals :				3.86390e4	400.39287	

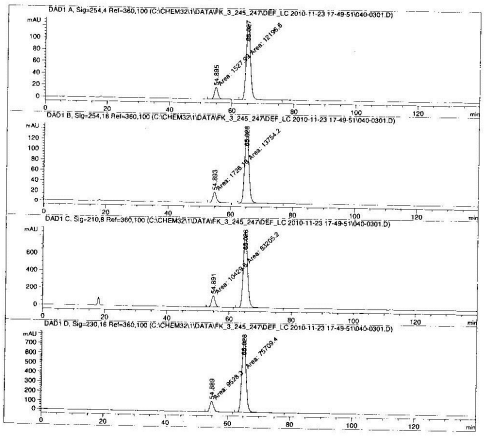
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	56.745	MM	1.5100	1.21465e5	1340.66772	51.9508
2	69.114	MM	1.7408	1.12342e5	1075.58105	48.0492
Totals :				2.33807e5	2416.24878	

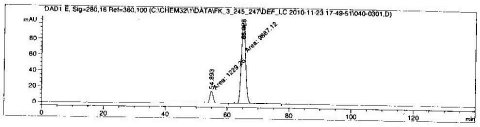
OD-H, 0.2 mL/min, 80 Hex : 20 EtOH

Data File C:\CHEM32\1\DATA\FK_3_245_247\DEF_LC 2010-11-23 17-49-51\040-0301.D
Sample Name: fk-3-247 low

Acq. Operator	: Filip
Acq. Instrument	: Instrument 1
Injection Date	: 11/23/2010 10:35:13 PM
Seq. Line	: 3
Location	: Vial 40
Inj	: 1
Inj Volume	: 4 µl
Acq. Method	: C:\Chem32\1\DATA\FK_3_245_247\DEF_LC 2010-11-23 17-49-51\FK-PH-MONOPHTH-ODH.M
Last changed	: 11/23/2010 5:28:14 PM by Filip
Analysis Method	: C:\CHEM32\2\METHODS\FK-MONOPHTH-CF3_FIN.M
Last changed	: 11/22/2010 11:18:35 AM by Filip
Method Info	: chiralcel OJ-H



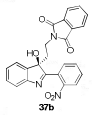
Data File C:\CHEM32\1\DATA\FK_3_245_247\DEF_LC 2010-11-23 17-49-51\040-0301.D
Sample Name: fk-3-247 low



Area Percent Report

Sorted By	: Signal
Multiplier	: 1.0000
Dilution	: 1.0000

Use Multiplier & Dilution Factor with ISTDs


37b

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	54.895	MM	1.3062	1527.98547	19.49644	11.1332
2	65.027	MM	1.6030	1.21966e4	126.80947	88.8668
Totals :				1.37246e4	146.30591	

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

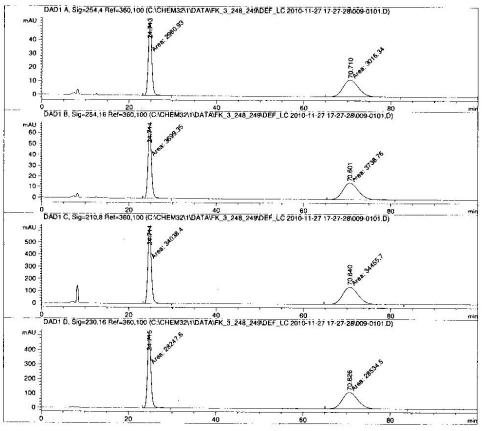
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	54.893	MM	1.3092	1726.19286	21.97551	11.1508
2	65.028	MM	1.6048	1.37542e4	142.84019	88.8492
Totals :				1.54803e4	164.81571	

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	54.891	MM	1.3102	1.04298e4	132.67923	11.1388
2	65.026	MM	1.6046	8.32052e4	864.25287	88.8612
Totals :				9.36350e4	996.93210	

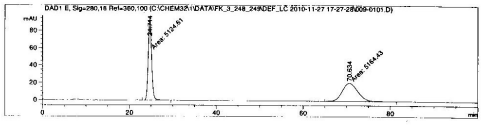
Data File C:\CHEM32\1\DATA\FK_3_248_249\DEF_LC 2010-11-27 17-27-28\009-0101.D
Sample Name: fk-3-248

Acq. Operator	: Filip	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 9
Injection Date	: 11/27/2010 5:40:15 PM	Inj	: 1
		Inj Volume	: 10 µl
Acq. Method	: C:\Chem32\1\DATA\FK_3_248_249\DEF_LC 2010-11-27 17-27-28\FK-PHORTOME-MONOPHTH-OJH.M		
Last changed	: 11/27/2010 5:12:44 PM by jeff		
Analysis Method	: C:\CHEM32\2\DATA\FK_3_TOSRAC\DEF_LC 2011-05-07 21-10-34\040-0101.D\DA.M (FK-HYDROXIND_TOS_OD_FIN.M)		
Last changed	: 5/8/2011 2:32:51 PM by Filip (modified after loading)		
Method Info	: chiralcel OD		



Instrument 2 5/8/2011 2:34:55 PM Filip Page 1 of 3

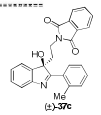
Data File C:\CHEM32\1\DATA\FK_3_248_249\DRP_LC 2010-11-27 17-27-28\009-0101.D
Sample Name: fk-3-248



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs


(±)-37c

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.713	MM	0.9212	2980.92676	53.92921	49.7048
2	70.710	MM	4.1186	3016.33984	12.20613	50.2952
Totals :				5997.26660	66.13534	

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.714	MM	0.9223	3699.35449	66.84946	49.7351
2	70.601	MM	4.1203	3738.76001	15.12320	50.2649
Totals :				7438.11450	81.97266	

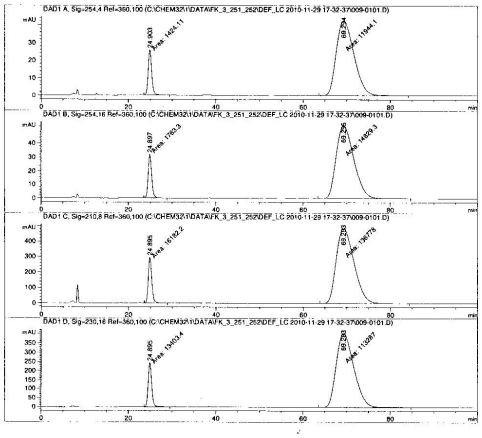
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.714	MM	0.9185	3.40384e4	617.61279	49.6954
2	70.640	MM	4.1268	3.44557e4	139.15559	50.3046
Totals :				6.84940e4	756.76839	

Instrument 2 5/8/2011 2:34:55 PM Filip Page 2 of 3

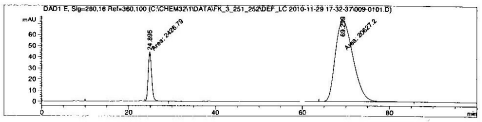
Data File C:\CHEM32\1\DATA\FK_3_251_252\DEF_LC 2010-11-29 17-32-37\009-0101.D
Sample Name: fk-3-251

Acq. Operator	: Filip	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 9
Injection Date	: 11/29/2010 5:53:59 PM	Inj	: 1
		Inj Volume	: 10 µl
Acq. Method	: C:\Chem32\1\DATA\FK_3_251_252\DEF_LC 2010-11-29 17-32-37\FK-PHORTOME-MONOPHTH-OJH.M		
Last changed	: 11/27/2010 5:12:44 PM by jeff		
Analysis Method	: C:\CHEM32\2\DATA\FK_3_TOSRAC\DEF_LC 2011-05-07 21-10-34\040-0101.D\DA.M (FK-HYDROXIND_TOS_OD_FIN.M)		
Last changed	: 5/8/2011 2:25:28 PM by Filip (modified after loading)		
Method Info	: chiralcel OD		



Instrument 2 5/8/2011 2:32:51 PM Filip Page 1 of 3

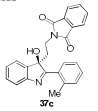
Data File C:\CHEM32\1\DATA\FK_3_251_252\DEF_LC 2010-11-29 17-32-37\009-0101.D
Sample Name: fk-3-251



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs


37c

89.4 : 10.6

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.903	MM	0.9239	1424.11414	25.69086	10.6530
2	69.294	MM	4.6954	1.19441e4	42.39601	89.3470
Totals :				1.33682e4	68.08687	

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.897	MM	0.9227	1763.30090	31.84959	10.6270
2	69.295	MM	4.7022	1.48293e4	52.56186	89.3730
Totals :				1.65926e4	84.41145	

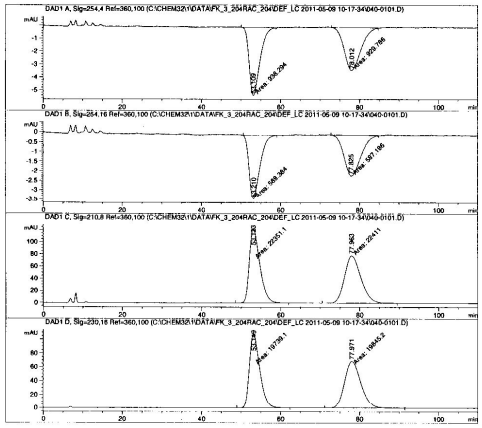
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.895	MM	0.9179	1.61822e4	293.84201	10.5794
2	69.283	MM	4.6910	1.36778e5	485.75543	89.4206
Totals :				1.52960e5	779.59744	

Instrument 2 5/8/2011 2:32:51 PM Filip Page 2 of 3

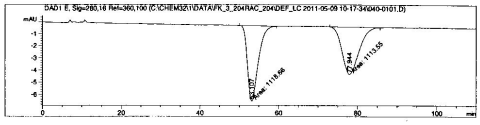
Data File C:\CHEM32\1\DATA\FK_3_204RAC_204\DEF_LC 2011-05-09 10-17-34\040-0101.D
Sample Name: fk-3-204rac

Acq. Operator : Filip
Acq. Instrument : Instrument 1
Injection Date : 5/9/2011 10:54:00 AM
Seq. Line : 1
Location : Vial 40
Inj : 1
Inj Volume : 5 ul
Acq. Method : C:\Chem32\1\DATA\FK_3_204RAC_204\DEF_LC 2011-05-09 10-17-34\FK-PHOME-MONOPHTH-OJH.M
Last changed : 5/9/2011 10:17:04 AM by Filip
Analysis Method : C:\CHEM32\1\METHODS\FK-PHOME-MONOPHTH-OJH.M
Last changed : 5/9/2011 10:17:04 AM by Filip
Method Info : chiralcel OJ-H



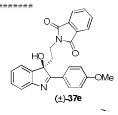
Instrument 1 5/9/2011 2:57:29 PM Filip Page 1 of 3

Data File C:\CHEM32\1\DATA\FK_3_204RAC_204\DEF_LC 2011-05-09 10-17-34\040-0101.D
Sample Name: fk-3-204rac



Area Percent Report

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



(±)-37e

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	53.109	PP N	2.9626	938.29382	5.27850	50.2277
2	78.012	PP N	4.6604	929.78644	3.32514	49.7723
Totals :				1868.08026	8.60364	

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	53.210	PP N	2.9487	588.38446	3.32563	50.0510
2	77.825	PP N	4.6480	587.18634	2.10551	49.9490
Totals :				1175.57080	5.43115	

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

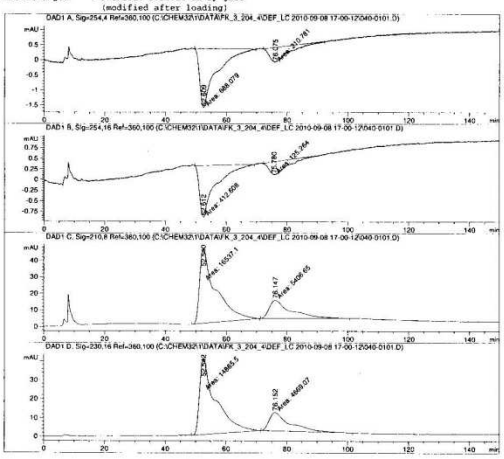
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	53.123	MM	3.0444	2.23511e4	122.36164	49.9331
2	77.963	MM	4.8071	2.24110e4	77.70162	50.0669
Totals :				4.47621e4	200.06326	

Instrument 1 5/9/2011 2:57:29 PM Filip Page 2 of 3

75:25
OJ-H, 0.5 ml/min, Hex : EtOH

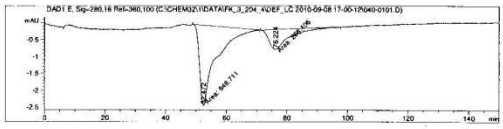
Data File C:\CHEM32\1\DATA\FK_3_204_4\DEF_LC 2010-09-08 17-00-12\040-0101.D
Sample Name: fk-3-204

Acq. Operator : Filip
Acq. Instrument : Instrument 1
Injection Date : 9/8/2010 5:05:03 PM
Seq. Line : 1
Location : Vial 40
Inj : 1
Inj Volume : 5 ul
Acq. Method : C:\Chem32\1\DATA\FK_3_204_4\DEF_LC 2010-09-08 17-00-12\FK-PHOME-MONOPHTH-ODH.M
Last changed : 9/8/2010 4:45:55 PM by Filip
Analysis Method : C:\CHEM32\1\METHODS\JLG-VI-012.M
Last changed : 9/1/2010 3:36:24 PM by jeff
(modified after loading)



Instrument 1 9/8/2010 9:06:55 PM jeff Page 1 of 3

Data File C:\CHEM32\1\DATA\FK_3_204_4\DEF_LC 2010-09-08 17-00-12\040-0101.D
Sample Name: fk-3-204



Area Percent Report

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

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	52.609	PP N	5.8459	688.07867	1.96171	76.5502
2	76.075	PP N	7.0746	210.78070	4.96565e-1	23.4498
Totals :				898.85938	2.45828	

52.2 min

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

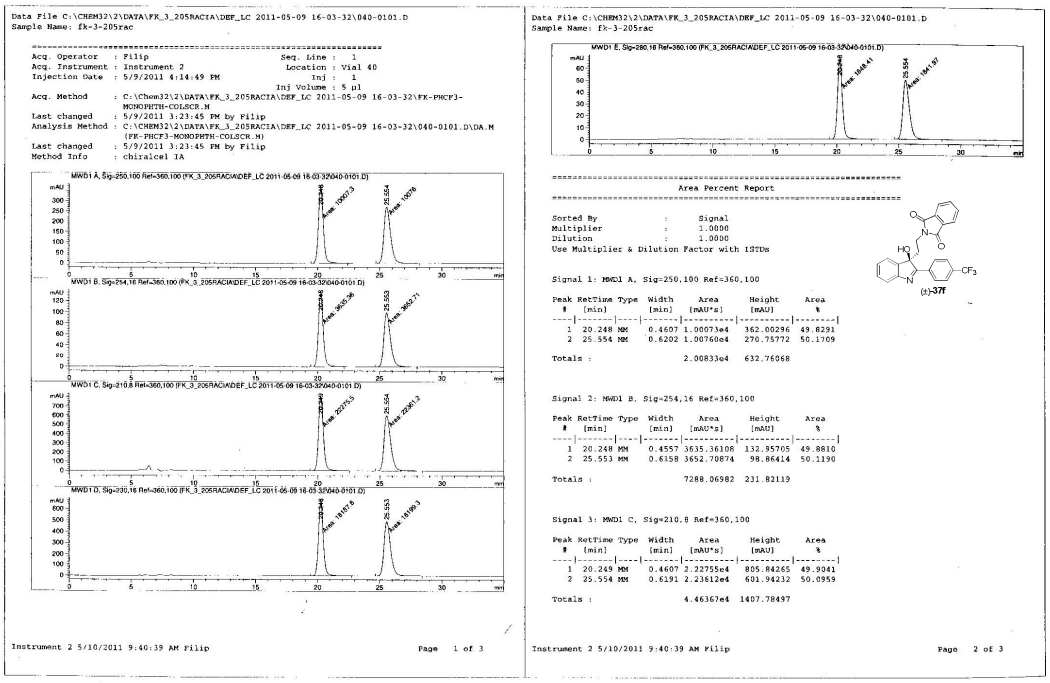
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	52.612	PP N	5.6286	412.60754	1.22154	76.7111
2	75.780	PP N	6.6562	125.26440	3.13653e-1	23.2889
Totals :				537.87195	1.53519	

76.1 : 23.9

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	52.540	MM	6.0924	1.65371e4	45.23973	75.3613
2	76.147	MM	8.1782	5406.65283	11.01848	24.6387
Totals :				2.19437e4	56.25821	

Instrument 1 9/8/2010 9:06:55 PM jeff Page 2 of 3



Data File C:\CHEM32\2\DATA\FK_3_205RACIA\DEF_LC 2011-05-09 16-03-32\040-0101.D
Sample Name: fk-3-205rac

Acq. Operator : Filip
Acq. Instrument : Instrument 2
Injection Date : 5/9/2011 4:14:49 PM
Seq. Line : 1
Location : Vial 40
Inj : 1
Inj Volume : 5 µl
Acq. Method : C:\Chem32\2\DATA\FK_3_205RACIA\DEF_LC 2011-05-09 16-03-32\FK-PHCF3-MONOPHTH-COLSCR.M
Last changed : 5/9/2011 3:23:45 PM by Filip
Analysis Method : C:\CHEM32\2\DATA\FK_3_205RACIA\DEF_LC 2011-05-09 16-03-32\040-0101.D\DA.M (FK-PHCF3-MONOPHTH-COLSCR.M)
Last changed : 5/9/2011 3:23:45 PM by Filip
Method Info : chiralcel IA

MWD1 A, Sig=250,100 Ref=360,100 (FK_3_205RACIA\DEF_LC 2011-05-09 16-03-32\040-0101.D)
MWD1 B, Sig=254,16 Ref=360,100 (FK_3_205RACIA\DEF_LC 2011-05-09 16-03-32\040-0101.D)
MWD1 C, Sig=210,8 Ref=360,100 (FK_3_205RACIA\DEF_LC 2011-05-09 16-03-32\040-0101.D)
MWD1 D, Sig=230,16 Ref=360,100 (FK_3_205RACIA\DEF_LC 2011-05-09 16-03-32\040-0101.D)

Instrument 2 5/10/2011 9:40:39 AM Filip
Page 1 of 3

Data File C:\CHEM32\2\DATA\FK_3_205RACIA\DEF_LC 2011-05-09 16-03-32\040-0101.D
Sample Name: fk-3-205rac

MWD1 E, Sig=280,16 Ref=360,100 (FK_3_205RACIA\DEF_LC 2011-05-09 16-03-32\040-0101.D)

Area Percent Report

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

(±)-37f

Signal 1: MWD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.248	MM	0.4607	1.00073e4	362.00296	49.8291
2	25.554	MM	0.6202	1.00760e4	270.75772	50.1709
Totals :				2.00833e4	632.76068	

Signal 2: MWD1 B, Sig=254,16 Ref=360,100

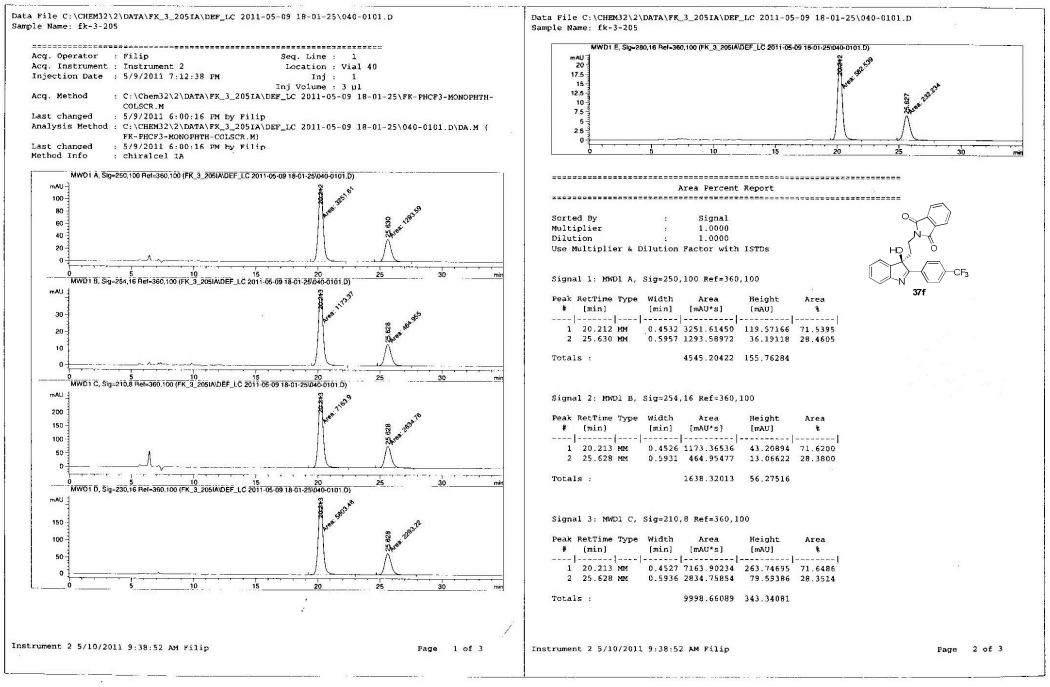
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.248	MM	0.4557	3635.36108	132.95705	49.8810
2	25.553	MM	0.6158	3652.70874	98.86414	50.1190
Totals :				7288.06982	231.82119	

Signal 3: MWD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.249	MM	0.4607	2.22755e4	805.84265	49.9041
2	25.554	MM	0.6191	2.23612e4	601.94232	50.0959
Totals :				4.46367e4	1407.78497	

Instrument 2 5/10/2011 9:40:39 AM Filip
Page 2 of 3

1A, 0.5 mL 1min, 80:20 Hex : IPA



Data File C:\CHEM32\2\DATA\FK_3_205IA\DEF_LC 2011-05-09 18-01-25\040-0101.D
Sample Name: fk-3-205

Acq. Operator : Filip
Acq. Instrument : Instrument 2
Injection Date : 5/9/2011 7:12:38 PM
Seq. Line : 1
Location : Vial 40
Inj : 1
Inj Volume : 3 µl
Acq. Method : C:\Chem32\2\DATA\FK_3_205IA\DEF_LC 2011-05-09 18-01-25\FK-PHCF3-MONOPHTH-COLSCR.M
Last changed : 5/9/2011 6:00:16 PM by Filip
Analysis Method : C:\CHEM32\2\DATA\FK_3_205IA\DEF_LC 2011-05-09 18-01-25\040-0101.D\DA.M (FK-PHCF3-MONOPHTH-COLSCR.M)
Last changed : 5/9/2011 6:00:16 PM by Filip
Method Info : chiralcel IA

MWD1 A, Sig=250,100 Ref=360,100 (FK_3_205IA\DEF_LC 2011-05-09 18-01-25\040-0101.D)
MWD1 B, Sig=254,16 Ref=360,100 (FK_3_205IA\DEF_LC 2011-05-09 18-01-25\040-0101.D)
MWD1 C, Sig=210,8 Ref=360,100 (FK_3_205IA\DEF_LC 2011-05-09 18-01-25\040-0101.D)
MWD1 D, Sig=230,16 Ref=360,100 (FK_3_205IA\DEF_LC 2011-05-09 18-01-25\040-0101.D)

Instrument 2 5/10/2011 9:38:52 AM Filip
Page 1 of 3

Data File C:\CHEM32\2\DATA\FK_3_205IA\DEF_LC 2011-05-09 18-01-25\040-0101.D
Sample Name: fk-3-205

MWD1 E, Sig=280,16 Ref=360,100 (FK_3_205IA\DEF_LC 2011-05-09 18-01-25\040-0101.D)

Area Percent Report

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

37f

Signal 1: MWD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.212	MM	0.4532	3251.61450	119.57166	71.5395
2	25.630	MM	0.5957	1293.58972	36.19118	28.4605
Totals :				4545.20422	155.76284	

Signal 2: MWD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.213	MM	0.4526	1173.36536	43.20894	71.6200
2	25.628	MM	0.5931	464.95477	13.06622	28.3800
Totals :				1638.32013	56.27516	

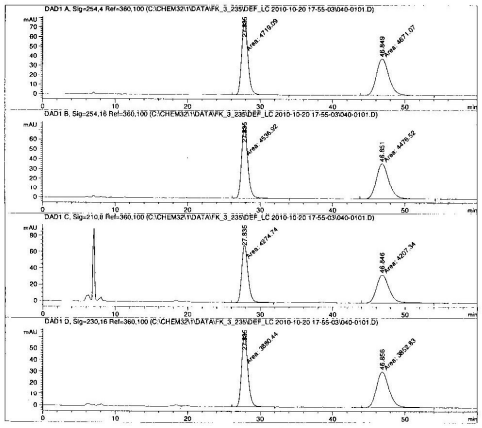
Signal 3: MWD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.213	MM	0.4527	7163.90234	263.74695	71.6486
2	25.628	MM	0.5936	2834.75854	79.59386	28.3514
Totals :				9998.66089	343.34081	

Instrument 2 5/10/2011 9:38:52 AM Filip
Page 2 of 3

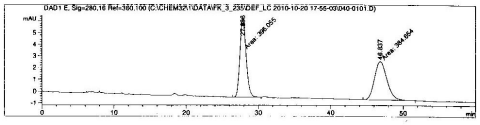
Data File C:\CHEM32\1\DATA\FK_3_235\DEF_LC 2010-10-20 17-55-03\040-0101.D
Sample Name: fk-3-235 low

Acq. Operator : Filip
Acq. Instrument : Instrument 1
Injection Date : 10/20/2010 5:58:34 PM
Seq. Line : 1
Location : Vial 40
Inj : 1
Inj Volume : 5 µl
Acq. Method : C:\Chem32\1\DATA\FK_3_235\DEF_LC 2010-10-20 17-55-03\FK-ANTRAC-PHTH.M
Last changed : 10/20/2010 4:48:10 PM by Filip
Analysis Method : C:\CHEM32\2\DATA\FK_3_TOSRAC\DEF_LC 2011-05-07 21-10-34\040-0101.D\DA.M (FK-HYDROXIND_TOS_OD_FIN.M)
Last changed : 5/8/2011 2:23:37 PM by Filip (modified after loading)
Method Info : chiralcel OD



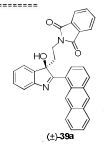
Instrument 2 5/8/2011 2:25:28 PM Filip Page 1 of 3

Data File C:\CHEM32\1\DATA\FK_3_235\DEF_LC 2010-10-20 17-55-03\040-0101.D
Sample Name: fk-3-235 low



Area Percent Report

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



(±)-39a

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.835	MM	1.0056	4719.09473	78.21732	50.2557
2	46.849	MM	2.0222	4671.06787	38.49893	49.7443
Totals :				9390.16260	116.71626	

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.835	MM	1.0084	4536.92334	74.98431	50.3351
2	46.851	MM	2.0226	4476.51855	36.88670	49.6649
Totals :				9013.44189	111.87102	

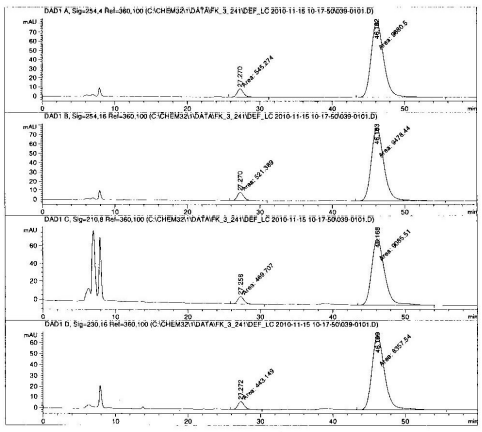
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.835	MM	1.0138	4274.73975	70.27375	50.3973
2	46.846	MM	2.0458	4207.34033	34.27620	49.6027
Totals :				8482.08008	104.54995	

Instrument 2 5/8/2011 2:25:28 PM Filip Page 2 of 3

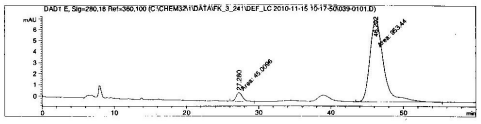
Data File C:\CHEM32\1\DATA\FK_3_241\DEF_LC 2010-11-15 10-17-50\039-0101.D
Sample Name: fk-3-241 low all

Acq. Operator : Filip
Acq. Instrument : Instrument 1
Injection Date : 11/15/2010 10:29:57 AM
Seq. Line : 1
Location : Vial 39
Inj : 1
Inj Volume : 10 µl
Acq. Method : C:\Chem32\1\DATA\FK_3_241\DEF_LC 2010-11-15 10-17-50\FK-ANTRAC-PHTH.M
Last changed : 11/15/2010 10:06:09 AM by Filip
Analysis Method : C:\CHEM32\2\DATA\FK_3_TOSRAC\DEF_LC 2011-05-07 21-10-34\040-0101.D\DA.M (FK-HYDROXIND_TOS_OD_FIN.M)
Last changed : 5/8/2011 2:20:45 PM by Filip (modified after loading)
Method Info : chiralcel OD



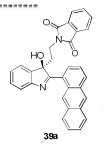
Instrument 2 5/8/2011 2:23:36 PM Filip Page 1 of 3

Data File C:\CHEM32\1\DATA\FK_3_241\DEF_LC 2010-11-15 10-17-50\039-0101.D
Sample Name: fk-3-241 low all



Area Percent Report

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



39a

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.270	MM	1.0170	545.27380	8.93626	5.2301
2	46.162	MM	2.0001	9880.49805	82.33318	94.7699
Totals :				1.0425e4	91.26945	

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.270	MM	1.0155	521.38879	8.55710	5.2140
2	46.163	MM	2.0020	9478.44434	78.90980	94.7860
Totals :				9999.83313	87.46690	

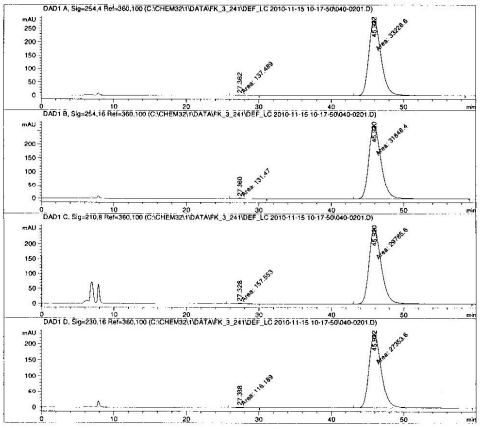
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.258	MM	0.9941	469.70676	7.87520	4.9157
2	46.168	MM	2.0382	9085.51465	74.29472	95.0843
Totals :				9555.22141	82.16991	

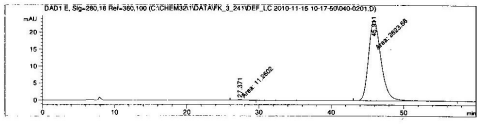
Instrument 2 5/8/2011 2:23:36 PM Filip Page 2 of 3

Data File C:\CHEM32\1\DATA\FK_3_241\DEF_LC 2010-11-15 10-17-50\040-0201.D
Sample Name: fk-3-241 low cryst

Acq. Operator	: Filip	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 40
Injection Date	: 11/15/2010 11:51:24 AM	Inj	: 1
		Inj Volume	: 10 µl
Acq. Method	: C:\Chem32\1\DATA\FK_3_241\DEF_LC 2010-11-15 10-17-50\FK-ANTRAC-PHTH.M		
Last changed	: 11/15/2010 10:06:09 AM by Filip		
Analysis Method	: C:\CHEM32\2\DATA\FK_3_TOSRAC\DEF_LC 2011-05-07 21-10-34\040-0101.D\DA.M (FK-HYDROXIND_TOS_OD_FIN.M)		
Last changed	: 5/8/2011 2:11:45 PM by Filip (modified after loading)		
Method Info	: chiralcel OD		



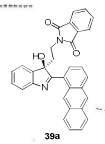
Data File C:\CHEM32\1\DATA\FK_3_241\DEF_LC 2010-11-15 10-17-50\040-0201.D
Sample Name: fk-3-241 low cryst



Area Percent Report

Sorted By	: Signal
Multiplier	: 1.0000
Dilution	: 1.0000

Use Multiplier & Dilution Factor with ISTDs



39a

99.6 : 0.4

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.362	MM	1.0812	137.48906	2.11930	0.4121
2	45.922	MM	1.9636	3.32286e4	282.04535	99.5879
Totals :				3.33661e4	284.16465	

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

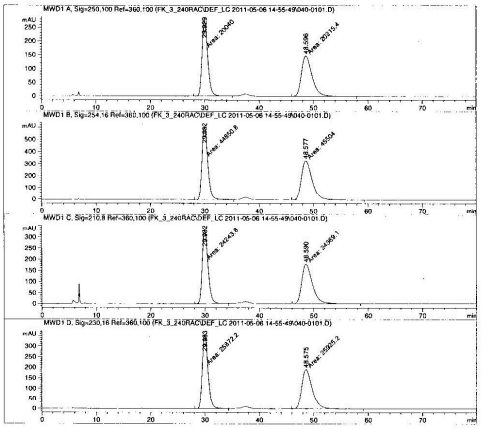
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.360	MM	1.0809	131.47032	2.02712	0.4111
2	45.920	MM	1.9651	3.18484e4	270.11804	99.5889
Totals :				3.19799e4	272.14516	

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.328	MM	1.2821	157.55267	2.04815	0.5262
2	45.920	MM	1.9688	2.97856e4	252.15018	99.4738
Totals :				2.99413e4	254.19832	

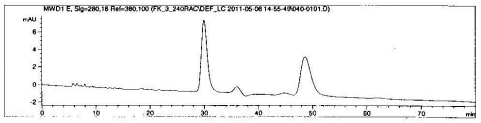
Data File C:\CHEM32\2\DATA\FK_3_240RAC\DEF_LC 2011-05-06 14-55-49\040-0101.D
Sample Name: FK-3-240 Rac

Acq. Operator	: Filip	Seq. Line	: 1
Acq. Instrument	: Instrument 2	Location	: Vial 40
Injection Date	: 5/6/2011 3:31:48 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: C:\Chem32\2\DATA\FK_3_240RAC\DEF_LC 2011-05-06 14-55-49\FK-ANTRAC-FHTH.M		
Last changed	: 5/6/2011 2:30:57 PM by Filip		
Analysis Method	: C:\CHEM32\2\DATA\FK_3_240RAC\DEF_LC 2011-05-06 14-55-49\040-0101.D\DA.M (FK-ANTRAC-FHTH.M)		
Last changed	: 5/6/2011 2:30:57 PM by Filip		
Method Info	: chiralcel OD-H		



Instrument 2 5/6/2011 5:30:52 PM Filip Page 1 of 3

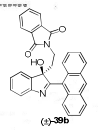
Data File C:\CHEM32\2\DATA\FK_3_240RAC\DEF_LC 2011-05-06 14-55-49\040-0101.D
Sample Name: FK-3-240



Area Percent Report

Sorted By	: Signal
Multiplier	: 1.0000
Dilution	: 1.0000

Use Multiplier & Dilution Factor with ISTDs


(±)-39b

Signal 1: MWD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.929	MM	1.2143	2.00400e4	275.05646	49.7822
2	48.596	MM	2.2793	2.02154e4	147.82066	50.2178
Totals :				4.02554e4	422.87712	

Signal 2: MWD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.932	MM	1.2249	4.48508e4	610.24048	49.6385
2	48.577	MM	2.2921	4.55040e4	330.87598	50.3615
Totals :				9.03548e4	941.11646	

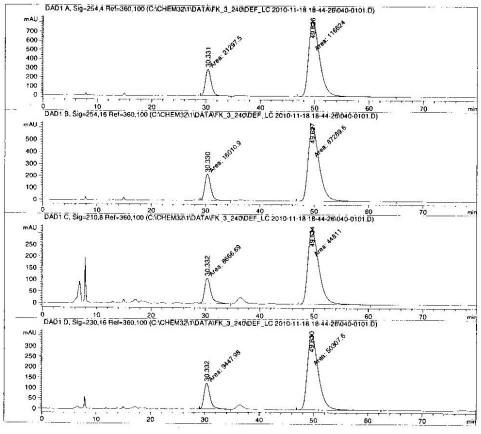
Signal 3: MWD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.932	MM	1.2090	2.42438e4	334.21313	49.8711
2	48.599	MM	2.2787	2.43691e4	178.23680	50.1289
Totals :				4.86129e4	512.44994	

Instrument 2 5/6/2011 5:30:52 PM Filip Page 2 of 3

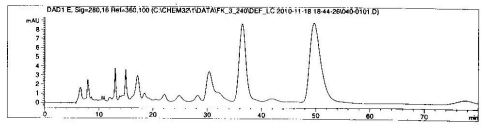
Data File C:\CHEM32\1\DATA\FK_3_240\DEF_LC 2010-11-18 18-44-26\040-0101.D
Sample Name: fk-3-240 low

Acq. Operator	: Filip	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 40
Injection Date	: 11/18/2010 7:21:06 PM	Inj	: 1
		Inj Volume	: 7 µl
Acq. Method	: C:\Chem32\1\DATA\FK_3_240\DEF_LC 2010-11-18 18-44-26\FK-ANTRAC-FHTH.M		
Last changed	: 11/18/2010 5:40:54 PM by Filip		
Analysis Method	: C:\CHEM32\2\METHODS\FK-ANTRAC-FHTH.M		
Last changed	: 11/18/2010 5:40:54 PM by Filip		
Method Info	: chiralcel OD-H		



Instrument 1 11/19/2010 10:59:01 AM Filip Page 1 of 3

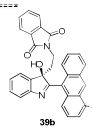
Data File C:\CHEM32\1\DATA\FK_3_240\DEF_LC 2010-11-18 18-44-26\040-0101.D
Sample Name: fk-3-240 low



Area Percent Report

Sorted By	: Signal
Multiplier	: 1.0000
Dilution	: 1.0000

Use Multiplier & Dilution Factor with ISTDs


39b

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.331	MM	1.2195	2.12975e4	291.05637	15.4417
2	49.626	MM	2.3529	1.16624e5	826.10889	84.5583
Totals :				1.37922e5	1117.16525	

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.330	MM	1.2222	1.60109e4	218.33565	15.4993
2	49.627	MM	2.3523	8.72896e4	618.47852	84.5007
Totals :				1.03301e5	836.81416	

84.2 : 15.8 e.r.

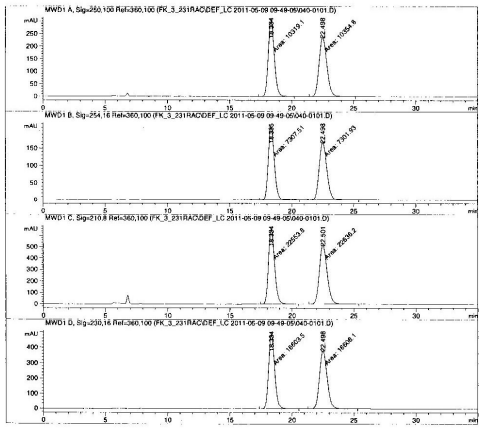
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.332	MM	1.3231	8666.68945	109.17543	16.2062
2	49.634	MM	2.3367	4.48110e4	319.61691	83.7938
Totals :				5.34777e4	428.79234	

Instrument 1 11/19/2010 10:59:01 AM Filip Page 2 of 3

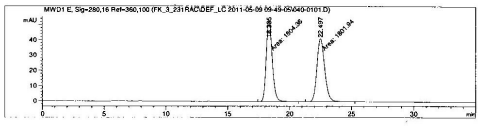
Data File C:\CHEM32\2\DATA\FK_3_231RAC\DEF_LC 2011-05-09 09-49-05\040-0101.D
Sample Name: fk-3-231rac

Acq. Operator : Filip
Acq. Instrument : Instrument 2
Injection Date : 5/9/2011 10:06:40 AM
Seq. Line : 1
Location : Vial 40
Inj : 1
Inj Volume : 5 ul
Acq. Method : C:\Chem32\2\DATA\FK_3_231RAC\DEF_LC 2011-05-09 09-49-05\FK-ANTRAC-PHTH.M
Last changed : 5/9/2011 9:16:16 AM by Filip
Analysis Method : C:\CHEM32\2\DATA\FK_3_231RAC\DEF_LC 2011-05-09 09-49-05\040-0101.D\DA.M (FK-ANTRAC-PHTH.M)
Last changed : 5/9/2011 9:16:16 AM by Filip
Method Info : chiralcel OD-H



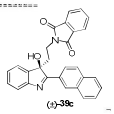
Instrument 2 5/9/2011 11:48:26 AM Filip Page 1 of 3

Data File C:\CHEM32\2\DATA\FK_3_231RAC\DEF_LC 2011-05-09 09-49-05\040-0101.D
Sample Name: fk-3-231rac



Area Percent Report

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


(±)-39c

Signal 1: MWD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.334	MM	0.5730	1.03191e4	300.16785	49.9137
2	22.498	MM	0.7314	1.03548e4	235.96573	50.0863
Totals :				2.06739e4	536.13358	

Signal 2: MWD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.335	MM	0.5713	7307.50732	213.20163	50.0191
2	22.498	MM	0.7294	7301.92578	166.84962	49.9809
Totals :				1.46094e4	380.05125	

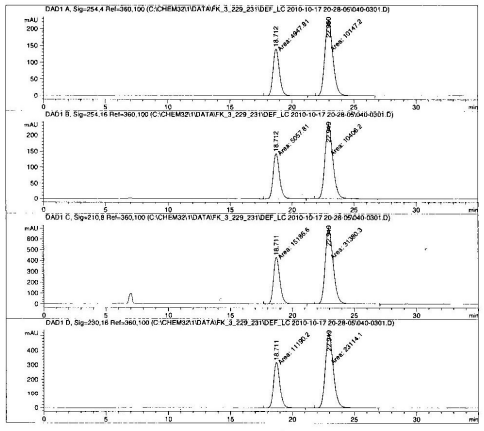
Signal 3: MWD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.334	MM	0.5726	2.25536e4	656.47266	49.9088
2	22.501	MM	0.7314	2.26362e4	515.80432	50.0912
Totals :				4.51900e4	1172.27698	

Instrument 2 5/9/2011 11:48:26 AM Filip Page 2 of 3

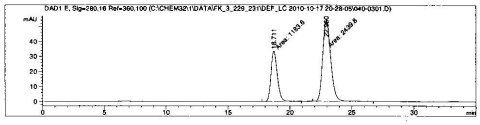
Data File C:\CHEM32\1\DATA\FK_3_229_231\DEF_LC 2010-10-17 20-28-05\040-0301.D
Sample Name: fk-3-231

Acq. Operator : Filip
Acq. Instrument : Instrument 1
Injection Date : 10/18/2010 9:57:12 AM
Seq. Line : 3
Location : Vial 40
Inj : 1
Inj Volume : 5 ul
Acq. Method : C:\Chem32\1\DATA\FK_3_229_231\DEF_LC 2010-10-17 20-28-05\FK-ANTRAC-PHTH.M
Last changed : 10/17/2010 8:28:00 PM by Filip
Analysis Method : C:\CHEM32\2\DATA\FK_3_TOSRAC\DEF_LC 2011-05-07 21-10-34\040-0101.D\DA.M (FK-HYDROXIND_TOS_OD_FIN.M)
Last changed : 5/8/2011 2:40:05 PM by Filip (modified after loading)
Method Info : chiralcel OD



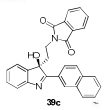
Instrument 2 5/8/2011 3:00:11 PM Filip Page 1 of 3

Data File C:\CHEM32\1\DATA\FK_3_229_231\DEF_LC 2010-10-17 20-28-05\040-0301.D
Sample Name: fk-3-231



Area Percent Report

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


39c

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.712	MM	0.5881	4947.80615	140.22806	32.7777
2	22.950	MM	0.7557	1.01472e4	223.80553	67.2223
Totals :				1.50950e4	364.03358	

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.712	MM	0.5872	5057.80762	143.55243	32.7071
2	22.949	MM	0.7541	1.04062e4	229.98517	67.2929
Totals :				1.54640e4	373.53760	

67.3:32.7 r.r.

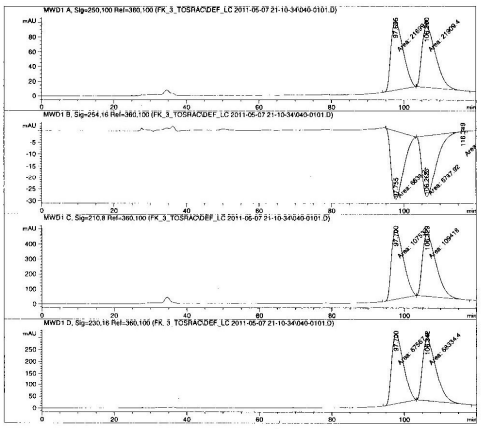
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.711	MM	0.5862	1.51866e4	431.79984	32.6124
2	22.949	MM	0.7543	3.13803e4	693.36108	67.3876
Totals :				4.65669e4	1125.16092	

Instrument 2 5/8/2011 3:00:11 PM Filip Page 2 of 3

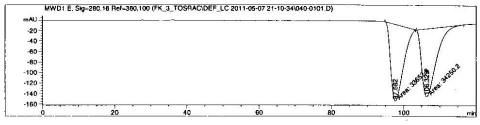
Data File C:\CHEM32\2\DATA\FK_3_TOSRAC\DEF_LC 2011-05-07 21-10-34\040-0101.D
Sample Name: Tos rac

Acq. Operator	: Filip	Seq. Line	: 1
Acq. Instrument	: Instrument 2	Location	: Vial 40
Injection Date	: 5/7/2011 11:41:41 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: C:\Chem32\2\DATA\FK_3_TOSRAC\DEF_LC 2011-05-07 21-10-34\FK-HYDROXIND_TOS_OD_FIN.M		
Last changed	: 5/7/2011 8:55:15 PM by Curren		
Analysis Method	: C:\CHEM32\2\DATA\FK_3_TOSRAC\DEF_LC 2011-05-07 21-10-34\040-0101.D\DA.M (FK-HYDROXIND_TOS_OD_FIN.M)		
Last changed	: 5/8/2011 1:40:59 PM by Filip (modified after loading)		
Method Info	: chiralcel OD		



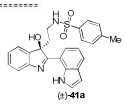
Instrument 2 5/8/2011 1:42:46 PM Filip Page 1 of 3

Data File C:\CHEM32\2\DATA\FK_3_TOSRAC\DEF_LC 2011-05-07 21-10-34\040-0101.D
Sample Name: Tos rac



Area Percent Report

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


(±)-41a

Signal 1: MWD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	97.695	MM	3.8866	2.16396e4	93.05273	49.7534
2	106.300	MM	4.4570	2.19094e4	81.92986	50.2406
Totals :				4.36090e4	174.98258	

Signal 2: MWD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	97.755	FP M	3.7781	6638.34766	29.28419	49.4144
2	106.262	PP N	4.3328	6787.92285	26.11049	50.5482
3	116.349	PM	0.8403	2.33087	4.62335e-2	0.0174
Totals :				1.34286e4	55.44091	

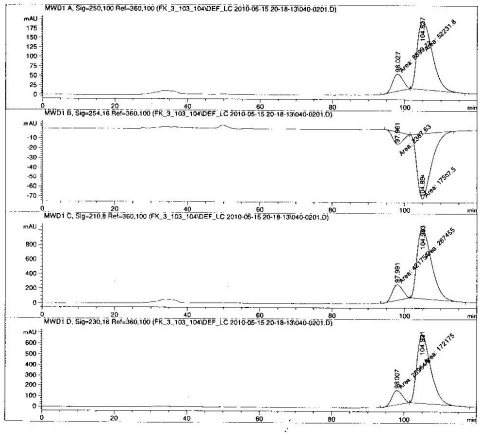
Signal 3: MWD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	97.709	MM	3.8341	1.07533e5	467.43619	49.5553
2	106.329	MM	4.4189	1.09418e5	412.68338	50.4238
3	151.565	VV	0.3201	45.29045	2.35818	0.0209
Totals :				2.16996e5	882.47775	

Instrument 2 5/8/2011 1:42:46 PM Filip Page 2 of 3

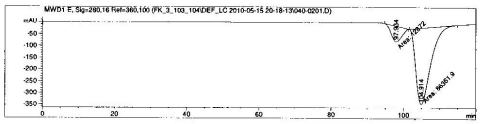
Data File C:\CHEM32\2\DATA\FK_3_103_104\DEF_LC 2010-05-15 20-18-13\040-0201.D
Sample Name: fk-3-104

Acq. Operator	: Filip	Seq. Line	: 2
Acq. Instrument	: Instrument 2	Location	: Vial 40
Injection Date	: 5/16/2010 1:01:10 AM	Inj	: 1
		Inj Volume	: 20 µl
Acq. Method	: C:\Chem32\2\DATA\FK_3_103_104\DEF_LC 2010-05-15 20-18-13\FK-HYDROXIND_TOS_OD_FIN.M		
Last changed	: 5/15/2010 8:17:26 PM by Filip		
Analysis Method	: C:\CHEM32\2\DATA\FK_3_TOSRAC\DEF_LC 2011-05-07 21-10-34\040-0101.D\DA.M (FK-HYDROXIND_TOS_OD_FIN.M)		
Last changed	: 5/8/2011 1:49:29 PM by Filip (modified after loading)		
Method Info	: chiralcel OD		



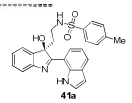
Instrument 2 5/8/2011 1:58:04 PM Filip Page 1 of 3

Data File C:\CHEM32\2\DATA\FK_3_103_104\DEF_LC 2010-05-15 20-18-13\040-0201.D
Sample Name: fk-3-104



Area Percent Report

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


41a

Signal 1: MWD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	98.027	MM	3.3193	8899.72461	44.68723	14.5583
2	104.827	MM	4.8155	5.22318e4	180.77823	85.4417
Totals :				6.11315e4	225.46546	

Signal 2: MWD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	97.961	MF N	3.2157	2387.63135	12.37489	12.0011
2	104.894	PP N	4.3278	1.75075e4	67.42208	87.9989
Totals :				1.98951e4	79.79697	

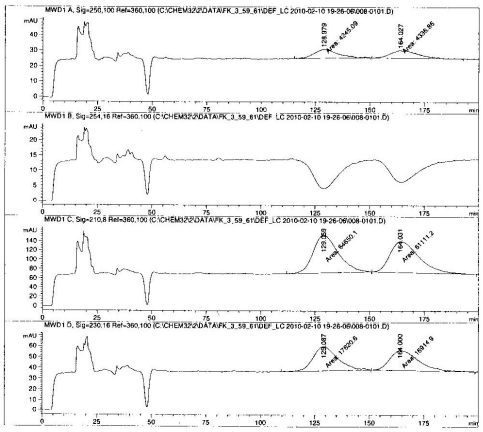
Signal 3: MWD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	97.991	MM	3.2954	4.21752e4	213.30299	13.6211
2	104.983	MM	4.6232	2.67455e5	964.17804	86.3789
Totals :				3.09630e5	1177.48103	

Instrument 2 5/8/2011 1:58:04 PM Filip Page 2 of 3

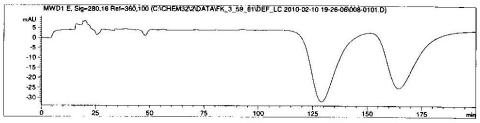
Data File C:\CHEM32\2\DATA\FK_3_59_61\DEF_LC 2010-02-10 19-26-06\008-0101.D
Sample Name: fk-3-59 lib-10

Acq. Operator : Filip
Acq. Instrument : Instrument 2
Injection Date : 2/10/2010 7:58:17 PM
Seq. Line : 1
Location : Vial 8
Inj : 1
Inj Volume : 20 ul
Acq. Method : C:\Chem32\2\DATA\FK_3_59_61\DEF_LC 2010-02-10 19-26-06\FK-HYDROXIND_MIT4.M
Last changed : 2/9/2010 6:27:15 PM by Filip
Analysis Method : C:\CHEM32\1\METHODS\PAL-I-230-ANALYSIS.M
Last changed : 12/8/2009 1:12:19 PM by liang
Method Info : 2% IPA in Hexane, 40 minutes; AD-column



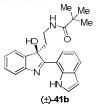
Instrument 1 2/11/2010 11:43:12 AM jeff
Page 1 of 3

Data File C:\CHEM32\2\DATA\FK_3_59_61\DEF_LC 2010-02-10 19-26-06\008-0101.D
Sample Name: fk-3-59 lib-10



Area Percent Report

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


(±)-41b

Signal 1: MWD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	128.979	MM	12.5449	4245.08740	5.63986	49.4653
2	164.027	MM	14.4061	4336.86084	5.01737	50.5347
Totals :				8581.94824	10.65724	

Signal 2: MWD1 B, Sig=254,16 Ref=360,100

Signal 3: MWD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	129.059	MM	13.1358	6.46501e4	82.02816	51.4070
2	164.031	MM	15.2059	6.11112e4	66.98608	48.5930
Totals :				1.25761e5	149.01424	

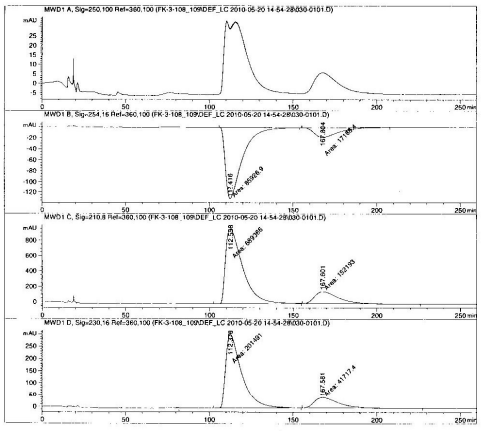
Signal 4: MWD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	129.087	MM	13.0357	1.76206e4	22.52871	51.0218
2	164.000	MM	15.0567	1.69149e4	18.72348	48.9782
Totals :				3.45355e4	41.25219	

Instrument 1 2/11/2010 11:43:12 AM jeff
Page 2 of 3

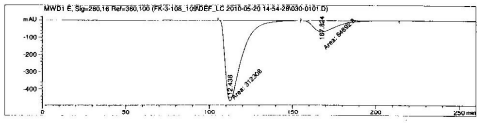
Data File C:\CHEM32\2\DATA\FK-3-108_109\DEF_LC 2010-05-20 14-54-28\030-0101.D
Sample Name: fk-3-108

Acq. Operator : Filip
Acq. Instrument : Instrument 2
Injection Date : 5/20/2010 3:06:43 PM
Seq. Line : 1
Location : Vial 30
Inj : 1
Inj Volume : 10 ul
Acq. Method : C:\Chem32\2\DATA\FK-3-108_109\DEF_LC 2010-05-20 14-54-28\FK-HYDROXIND_YALE_PIV.M
Last changed : 5/20/2010 1:58:51 PM by Filip
Analysis Method : C:\CHEM32\2\DATA\FK-3-108_109\DEF_LC 2010-05-20 14-54-28\030-0101.D\DA.M (FK-HYDROXIND_YALE_PIV.M)
Last changed : 5/20/2010 1:58:51 PM by Filip
Method Info : chiralcel OJ-H



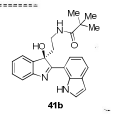
Instrument 2 5/21/2010 12:19:37 PM Filip
Page 1 of 3

Data File C:\CHEM32\2\DATA\FK-3-108_109\DEF_LC 2010-05-20 14-54-28\030-0101.D
Sample Name: fk-3-108



Area Percent Report

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


41b

66% ee

Signal 1: MWD1 A, Sig=250,100 Ref=360,100

Signal 2: MWD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	112.416	PP N	10.5227	8.55269e4	131.11415	83.3303
2	167.804	MP N	16.1411	1.71884e4	17.74805	16.6691
Totals :				1.03115e5	148.86220	

83: 17

Signal 3: MWD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	112.558	MM	11.8131	6.89366e5	972.60522	81.9154
2	167.601	MM	16.4088	1.52193e5	154.58469	18.0845
Totals :				8.41559e5	1127.18991	

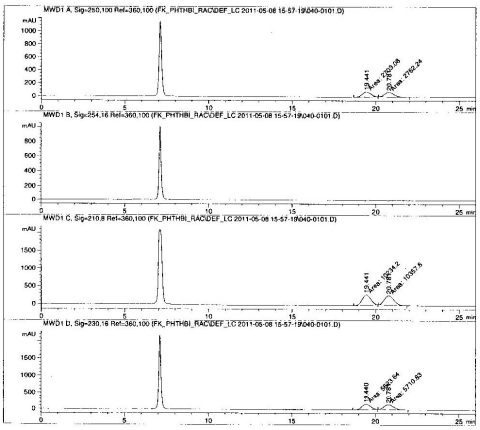
Signal 4: MWD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	112.378	MM	11.0222	2.01491e5	304.67389	82.8471
2	167.581	MM	16.4012	4.17174e4	42.39267	17.1529
Totals :				2.43209e5	347.06656	

Instrument 2 5/21/2010 12:19:37 PM Filip
Page 2 of 3

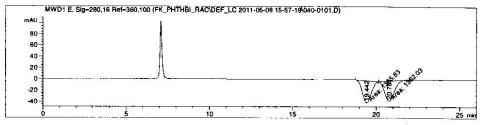
Data File C:\CHEM32\2\DATA\FK_PHTHBI_RAC\DEF_LC 2011-05-08 15-57-19\040-0101.D
Sample Name: Phth rac

Acq. Operator : Filip
Acq. Instrument : Instrument 2
Injection Date : 5/8/2011 4:01:39 PM
Seq. Line : 1
Location : Vial 40
Inj : 1
Inj Volume : 10 µl
Acq. Method : C:\Chem32\2\DATA\FK_PHTHBI_RAC\DEF_LC 2011-05-08 15-57-19\FK-MONOPHTH-ODH.M
Last changed : 5/8/2011 3:10:27 PM by Filip
Analysis Method : C:\CHEM32\2\DATA\FK_PHTHBI_RAC\DEF_LC 2011-05-08 15-57-19\040-0101.D\DA.M (FK-MONOPHTH-ODH.M)
Last changed : 5/8/2011 3:10:27 PM by Filip
Method Info : chiralcel OD-H



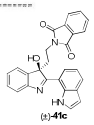
Instrument 2 5/8/2011 4:41:41 PM Filip Page 1 of 3

Data File C:\CHEM32\2\DATA\FK_PHTHBI_RAC\DEF_LC 2011-05-08 15-57-19\040-0101.D
Sample Name: Phth rac



Area Percent Report

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


(±)-41c

Signal 1: MWD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.441	MM	0.5919	2703.07861	76.10938	49.4587
2	20.781	MM	0.6558	2762.24292	70.19650	50.5413
Totals :				5465.32153	146.30588	

Signal 2: MWD1 B, Sig=254,16 Ref=360,100

Signal 3: MWD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.441	MM	0.5894	1.02342e4	289.41443	49.7002
2	20.781	MM	0.6518	1.03576e4	264.85382	50.2998
Totals :				2.05918e4	554.26825	

Signal 4: MWD1 D, Sig=230,16 Ref=360,100

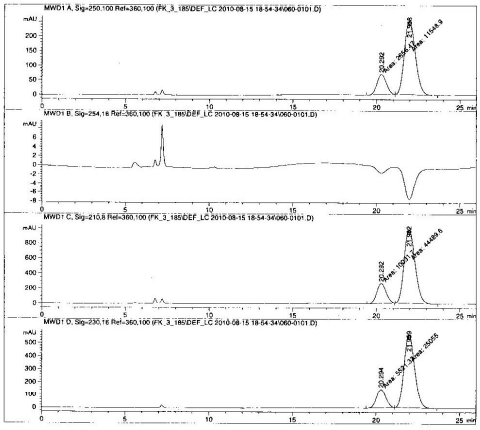
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.440	MM	0.5880	5623.64111	159.39807	49.6154
2	20.781	MM	0.6514	5710.82813	146.11992	50.3846
Totals :				1.13345e4	305.51799	

Rac

Instrument 2 5/8/2011 4:41:41 PM Filip Page 2 of 3

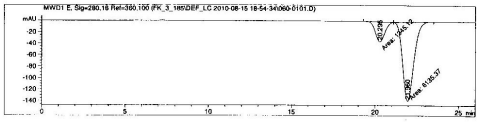
Data File C:\CHEM32\2\DATA\FK_3_185\DEF_LC 2010-08-15 18-54-34\060-0101.D
Sample Name: fk-3-185

Acq. Operator : Filip
Acq. Instrument : Instrument 2
Injection Date : 8/15/2010 7:25:38 PM
Seq. Line : 1
Location : Vial 60
Inj : 1
Inj Volume : 3 µl
Acq. Method : C:\Chem32\2\DATA\FK_3_185\DEF_LC 2010-08-15 18-54-34\FK-MONOPHTH-ODH.M
Last changed : 8/15/2010 6:53:34 PM by Filip
Analysis Method : C:\CHEM32\2\DATA\FK_3_TOSRAC\DEF_LC 2011-05-07 21-10-34\040-0101.D\DA.M (FK-HYDROXIND_TOS_OD_FIN.M)
Last changed : 5/8/2011 1:58:14 PM by Filip (modified after loading)
Method Info : chiralcel OD



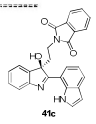
Instrument 2 5/8/2011 2:11:45 PM Filip Page 1 of 3

Data File C:\CHEM32\2\DATA\FK_3_185\DEF_LC 2010-08-15 18-54-34\060-0101.D
Sample Name: fk-3-185



Area Percent Report

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


41c

Signal 1: MWD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.292	MM	0.6420	2656.46729	68.96424	18.7005
2	21.958	MM	0.7622	1.15489e4	252.54510	81.2995
Totals :				1.42053e4	321.50935	

Signal 2: MWD1 B, Sig=254,16 Ref=360,100

82.2 : 17.8

Signal 3: MWD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.292	MM	0.6393	1.00317e4	261.54788	18.3996
2	21.962	MM	0.7580	4.44896e4	978.21533	81.6004
Totals :				5.45213e4	1239.76321	

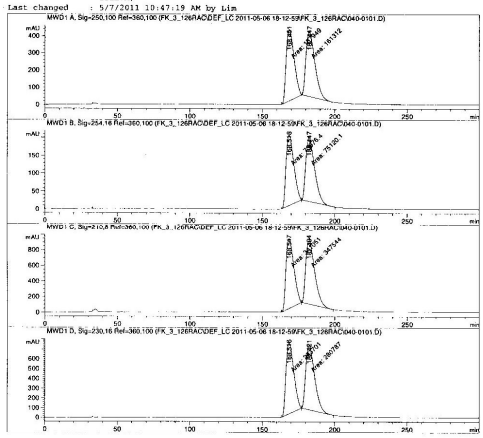
Signal 4: MWD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.294	MM	0.6398	5521.32520	144.05894	18.0575
2	21.959	MM	0.7404	2.50550e4	563.99811	81.9425
Totals :				3.05763e4	708.05705	

Instrument 2 5/8/2011 2:11:45 PM Filip Page 2 of 3

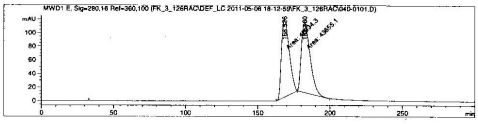
Data File C:\CHEM32\2\DATA\FK_3_126RAC\DEF_LC 2011-05-06 18-12-59\FK_3_126RAC\040-0101.D
Sample Name: fk-3-126rac

Acq. Operator	: Filip	Seq. Line	: 1
Acq. Instrument	: Instrument 2	Location	: Vial 40
Injection Date	: 5/6/2011 8:15:37 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: C:\Chem32\2\DATA\FK_3_126RAC\DEF_LC 2011-05-06 18-12-59\FK-SES_PHTH.M		
Last changed	: 5/6/2011 5:48:15 PM by Filip		
Analysis Method	: C:\CHEM32\2\DATA\DLIM\DEF_LC 2011-05-07 10-47-27\091-0101.D\DA.M (DL-II-2218.M)		
Last changed	: 5/7/2011 10:47:19 AM by Lim		



Instrument 2 5/7/2011 1:06:37 PM Lim Page 1 of 3

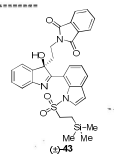
Data File C:\CHEM32\2\DATA\FK_3_126RAC\DEF_LC 2011-05-06 18-12-59\FK_3_126RAC\040-0101.D
Sample Name: fk-3-126rac



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs



(±)-43

Signal 1: MWD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	168.451	MM	6.3154	1.57949e5	416.83960	49.4734
2	182.447	MM	7.1338	1.61312e5	376.87177	50.5266
Totals :				3.19261e5	793.71136	

Signal 2: MWD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	168.518	MM	6.2505	7.36764e4	196.45477	49.5149
2	182.447	MM	7.0311	7.51201e4	178.06624	50.4851
Totals :				1.48796e5	374.52101	

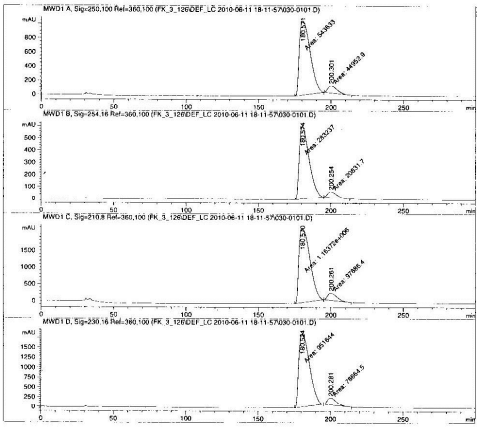
Signal 3: MWD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	168.547	MM	6.3053	3.41051e5	901.49384	49.5285
2	182.494	MM	7.1114	3.47544e5	814.51990	50.4715
Totals :				6.88596e5	1716.01373	

Instrument 2 5/7/2011 1:06:37 PM Lim Page 2 of 3

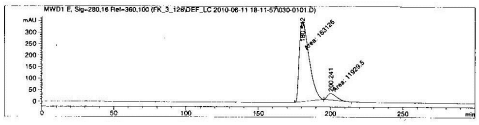
Data File C:\CHEM32\2\DATA\FK_3_126\DEF_LC 2010-06-11 18-11-57\030-0101.D
Sample Name: fk-3-126

Acq. Operator	: Filip	Seq. Line	: 1
Acq. Instrument	: Instrument 2	Location	: Vial 30
Injection Date	: 6/11/2010 6:23:29 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: C:\Chem32\2\DATA\FK_3_126\DEF_LC 2010-06-11 18-11-57\FK-SES_PHTH.M		
Last changed	: 6/11/2010 5:05:46 PM by Lindsey		
Analysis Method	: C:\CHEM32\2\DATA\FK_3_126\DEF_LC 2010-06-11 18-11-57\030-0101.D\DA.M (FK-SES_PHTH.M)		
Last changed	: 6/11/2010 5:05:46 PM by Lindsey		
Method Info	: chiralcel OD		



Instrument 2 6/12/2010 2:18:09 PM PeterJ Page 1 of 3

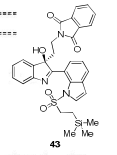
Data File C:\CHEM32\2\DATA\FK_3_126\DEF_LC 2010-06-11 18-11-57\030-0101.D
Sample Name: fk-3-126



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs



43

85% ee

Signal 1: MWD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	180.571	MM	8.6482	5.43633e5	1047.67761	92.3626
2	200.301	MM	7.1735	4.49529e4	104.44221	7.6374
Totals :				5.88586e5	1152.11982	

93:7

Signal 2: MWD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	180.574	MM	5.5043	2.83237e5	603.16412	93.1690
2	200.254	MM	7.1933	2.08317e4	48.26645	6.8510
Totals :				3.04069e5	651.43058	

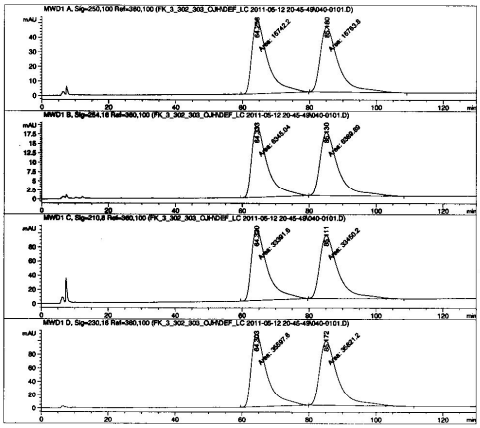
Signal 3: MWD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	180.570	MM	8.8803	1.16372e6	2184.08862	92.2411
2	200.261	MM	7.2219	9.78864e4	225.90100	7.7589
Totals :				1.26161e6	2409.98962	

Instrument 2 6/12/2010 2:18:09 PM PeterJ Page 2 of 3

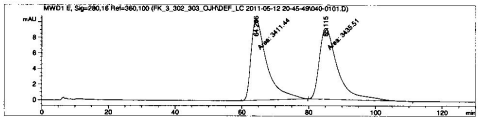
Data File C:\CHEM32\2\DATA\FK_3_302_303_OJH\DEF_LC 2011-05-12 20-45-49\040-0101.D
Sample Name: fk-3-302

Acq. Operator	: Filip	Seq. Line	: 1
Acq. Instrument	: Instrument 2	Location	: Vial 40
Injection Date	: 5/12/2011 9:57:24 PM	Inj	: 1
		Inj Volume	: 6 µl
Acq. Method	: C:\Chem32\2\DATA\FK_3_302_303_OJH\DEF_LC 2011-05-12 20-45-49\FK-NAPHTYLREARRANG.M		
Last changed	: 5/12/2011 8:00:23 PM by Filip		
Analysis Method	: C:\CHEM32\2\DATA\FK_3_302_303_OJH\DEF_LC 2011-05-12 20-45-49\040-0101.D\DA.M (FK-NAPHTYLREARRANG.M)		
Last changed	: 5/12/2011 8:00:23 PM by Filip		
Method Info	: chiralcel OJ-H		



Instrument 2 5/13/2011 8:01:30 AM Filip Page 1 of 3

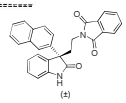
Data File C:\CHEM32\2\DATA\FK_3_302_303_OJH\DEF_LC 2011-05-12 20-45-49\040-0101.D
Sample Name: fk-3-302



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs



Signal 1: MWD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	64.258	MM	5.5448	1.67422e4	50.32381	49.9678
2	85.180	MM	6.2804	1.67638e4	44.48713	50.0322
Totals :				3.35060e4	94.81094	

Signal 2: MWD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	64.333	MM	5.5521	6345.03613	19.04701	49.8239
2	85.130	MM	6.3108	6389.88574	16.87547	50.1761
Totals :				1.27349e4	35.92248	

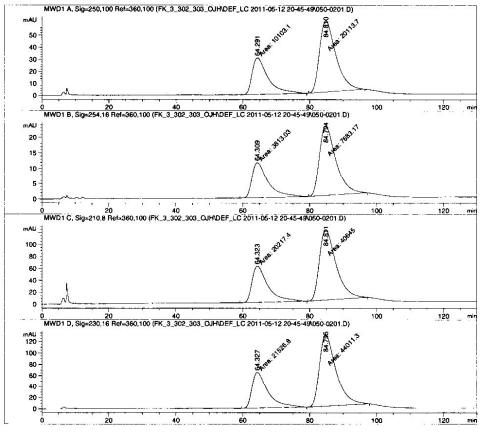
Signal 3: MWD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	64.320	MM	5.5346	3.33916e4	100.55346	49.9562
2	85.111	MM	6.2597	3.34502e4	89.06238	50.0438
Totals :				6.68418e4	189.61584	

Instrument 2 5/13/2011 8:01:30 AM Filip Page 2 of 3

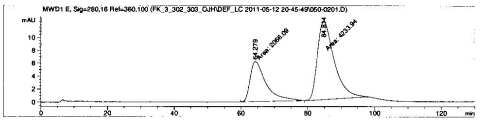
Data File C:\CHEM32\2\DATA\FK_3_302_303_OJH\DEF_LC 2011-05-12 20-45-49\050-0201.D
Sample Name: fk-3-303

Acq. Operator	: Filip	Seq. Line	: 2
Acq. Instrument	: Instrument 2	Location	: Vial 50
Injection Date	: 5/13/2011 2:57:57 AM	Inj	: 1
		Inj Volume	: 6 µl
Acq. Method	: C:\Chem32\2\DATA\FK_3_302_303_OJH\DEF_LC 2011-05-12 20-45-49\FK-NAPHTYLREARRANG.M		
Last changed	: 5/12/2011 8:00:23 PM by Filip		
Analysis Method	: C:\CHEM32\2\DATA\FK_3_302_303_OJH\DEF_LC 2011-05-12 20-45-49\050-0201.D\DA.M (FK-NAPHTYLREARRANG.M)		
Last changed	: 5/13/2011 8:03:20 AM by Filip (modified after loading)		
Method Info	: chiralcel OJ-H		



Instrument 2 5/13/2011 8:06:30 AM Filip Page 1 of 3

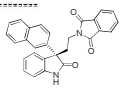
Data File C:\CHEM32\2\DATA\FK_3_302_303_OJH\DEF_LC 2011-05-12 20-45-49\050-0201.D
Sample Name: fk-3-303



Area Percent Report

Sorted By	:	Signal
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs



Signal 1: MWD1 A, Sig=250,100 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	64.291	MM	5.5758	1.01031e4	30.19909	33.4353
2	84.810	MM	5.6723	2.01137e4	59.09898	66.5647
Totals :				3.02168e4	89.29807	

Signal 2: MWD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	64.309	MM	5.5597	3813.02808	11.43055	33.1677
2	84.794	MM	5.7139	7683.16748	22.41083	66.8323
Totals :				1.14962e4	33.84138	

Signal 3: MWD1 C, Sig=210,8 Ref=360,100

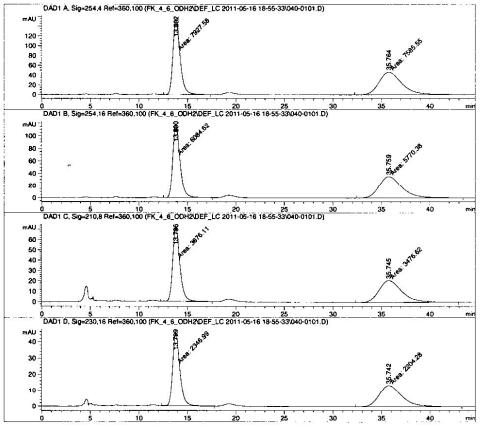
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	64.323	MM	5.5709	2.02174e4	60.48574	33.2182
2	84.811	MM	5.7259	4.06450e4	118.30669	66.7818
Totals :				6.08625e4	178.79244	

Instrument 2 5/13/2011 8:06:30 AM Filip Page 2 of 3

OD-H, 0.7 mL/min, 55:45 Hex:EtOH

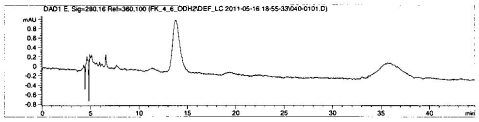
Data File C:\PURIFY\DATA\20070302.000\FK_4_6_ODH2\DEF_LC 2011-05-16 18-55-33\040-0101.D
Sample Name: fk-4-61ow

Acq. Operator : Filip
Acq. Instrument : Instrument 1
Injection Date : 5/16/2011 7:00:11 PM
Seq. Line : 1
Location : Vial 40
Inj : 1
Inj Volume : 10 ul
Acq. Method : C:\Purify\data\20070302.000\FK_4_6_ODH2\DEF_LC 2011-05-16 18-55-33\FK-ANTHRAREARRANGINST1.M
Last changed : 5/16/2011 6:16:35 PM by Filip
Analysis Method : C:\CHEM32\1\DATA\FK_4_6_ODH\DEF_LC 2011-05-16 13-54-45\040-0101.D\DA.M (FK-ANTHRAREARRANGINST1.M)
Last changed : 5/16/2011 9:02:21 PM by Filip
Method Info : chiralcel IA



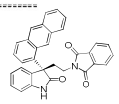
Instrument 1 5/16/2011 9:03:51 PM Filip Page 1 of 3

Data File C:\PURIFY\DATA\20070302.000\FK_4_6_ODH2\DEF_LC 2011-05-16 18-55-33\040-0101.D
Sample Name: fk-4-61ow



Area Percent Report

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



(±)-45

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.802	MM	0.8624	7927.58105	153.20509	51.1024
2	35.764	MM	2.7356	7585.54932	46.21542	48.8976
Totals :				1.55131e4	199.42052	

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.800	MM	0.8645	6084.62451	117.30924	51.3254
2	35.759	MM	2.7330	5770.37695	35.18988	48.6746
Totals :				1.18550e4	152.49912	

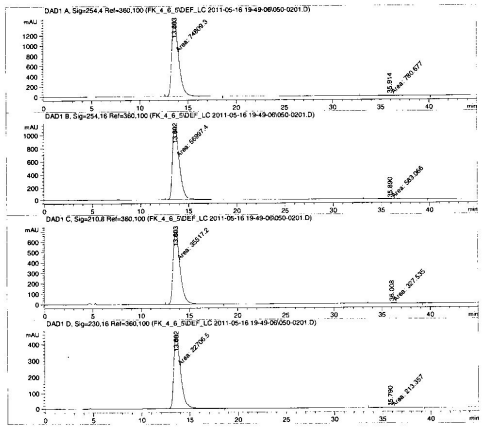
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.796	MM	0.8648	3676.10645	70.84660	51.3945
2	35.745	MM	2.7500	3476.61670	21.07026	48.6055
Totals :				7152.72314	91.91687	

Instrument 1 5/16/2011 9:03:51 PM Filip Page 2 of 3

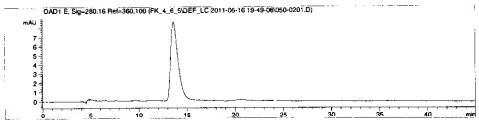
Data File C:\PURIFY\DATA\20070302.000\FK_4_6_5\DEF_LC 2011-05-16 19-49-06\050-0201.D
Sample Name: fk-4-51ow

Acq. Operator : Filip
Acq. Instrument : Instrument 1
Injection Date : 5/16/2011 8:52:09 PM
Seq. Line : 2
Location : Vial 50
Inj : 1
Inj Volume : 10 ul
Acq. Method : C:\Purify\data\20070302.000\FK_4_6_5\DEF_LC 2011-05-16 19-49-06\FK-ANTHRAREARRANGINST1.M
Last changed : 5/16/2011 7:49:04 PM by Filip
Analysis Method : C:\CHEM32\2\METHODS\FK-ANTHRAREARRANGINST1.M
Last changed : 5/16/2011 7:49:04 PM by Filip
Method Info : chiralcel od-h



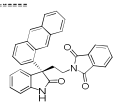
Instrument 1 5/16/2011 9:41:21 PM Filip Page 1 of 3

Data File C:\PURIFY\DATA\20070302.000\FK_4_6_5\DEF_LC 2011-05-16 19-49-06\050-0201.D
Sample Name: fk-4-51ow



Area Percent Report

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



45

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.603	MM	0.8459	7.48092e4	1473.97327	98.9672
2	35.914	MM	2.6389	780.67700	4.93064	1.0328
Totals :				7.55899e4	1478.90391	

Signal 2: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.602	MM	0.8430	5.69974e4	1126.82300	98.9874
2	35.890	MM	2.6145	583.06555	3.71690	1.0126
Totals :				5.75805e4	1130.53990	

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.603	MM	0.8239	3.55172e4	718.47443	99.0862
2	36.008	MM	2.4815	327.53485	2.19986	0.9138
Totals :				3.58447e4	720.67429	

Instrument 1 5/16/2011 9:41:21 PM Filip Page 2 of 3

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9. Least Squares function minimized: (SHELXL97)

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

10. 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

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