Exploitation of Differential Electronic Densities for the Stereoselective Reduction of Ketones Bearing a Masked Amino Surrogate

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Supporting Information

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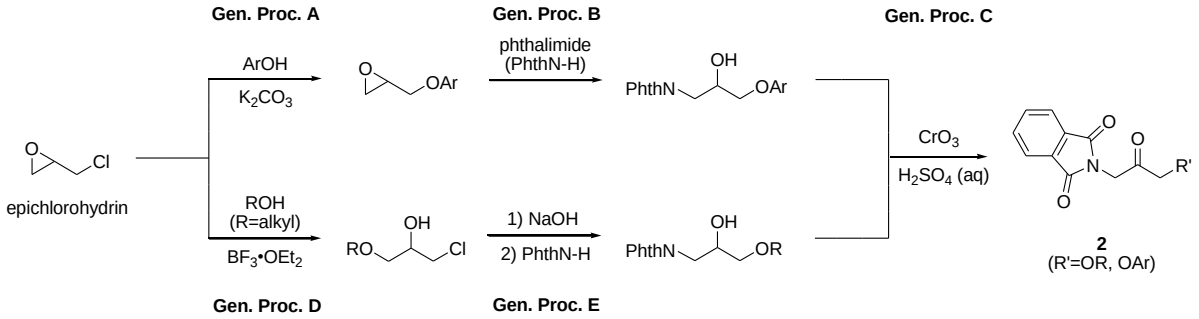
I. General Information

Analytical grade solvents were used directly without further purification as purchased from commercial sources: chloroform, acetone and tetrahydrofuran from VWR Chemicals; toluene, acetonitrile and concentrated sulphuric acid from Fischer Scientific; dichloromethane and 1,2-dichloroethane from Sigma Aldrich. Chiral tethered ruthenium catalyst (*R*,*R*)-**1a** supplied by Johnson Matthey and (*S*)-oxiranylanisole [97% sum of enantiomers] and AD-mix-*α* from Sigma Aldrich was used directly without further purification. Flash chromatography on silica was conducted on Sigma Aldrich silica gel (technical grade, pore size 60Å, 230-400 mesh, 40-63 μm particle size). Room temperature is defined to be approximately 20 ^{o}C.

NMR spectra were recorded on Bruker Avance III HDF 400 and 500 spectrometers. ^{1}H NMR spectra chemical shifts were reported in δ ppm relative to chloroform (δ = 7.26 ppm) or tetramethylsilane (δ = 0.00 ppm). Multiplicities were given as: s (singlet), d (doublet), t (triplet), q (quartet) and m (multiplet). The number of protons (n) for a given resonance was indicated by nH while coupling constants were reported as *J* value in Hertz (Hz). ^{13}C NMR spectra chemical shifts were recorded relative to solvent resonance ($CDCl_3$: δ = 77.26 ppm). Optical rotations of optically active alcohols were measured in the specified solution using a 2 dm cell with an Optical Activity Ltd. AA-1000 polarimeter. Chiral HPLC was performed on a Hewlett Packard 1050 HPLC machine incorporating a Diacel CHIRAPAK® IA, IC or Diacel CHIRALCEL OD-H column.

II. Experimental Section

Synthetic scheme for the preparation of α-phthalimyl-α'-ketoethers 2



(1) General Procedure A: Preparation of aryl glycidyl ethers

Procedure is adapted and modified from *Med. Chem. Res.* **2004**, *13*, 631. Mixture of phenol (ca. 10.0 mmol, 1 equiv.), K_2CO_3 (1.2 equiv.) and epichlorohydrin (5 equiv.) was refluxed for 5 hours in a round bottom flask. Upon completion, mixture was filtered through Celite and the filter cake washed with excess ethyl acetate. The filtrate was washed with water, the organic layer dried over Na_2SO_4, then subject to rotary evaporation to give the crude product before purification by Kugelrohr distillation.

(2) General Procedure B: Epoxide ring opening with phthalimide

Procedure is adapted and modified from *Tetrahedron: Asymmetry* **2008**, *17*, 1898. To a solution of the epoxide (ca. 10.0 mmol, 1 equiv.), phthalimide (1.2 equiv.) and isopropyl alcohol (ca. 201 mL; toluene for alkyl ethers) was added catalytic amount of pyridine (0.05 equiv.) before refluxing for 2 hours. The solution was subject to solvent strip under reduced pressure then the residue purified by flash chromatography on silica to afford the desired alcohols.

(3) General Procedure C: Preparation of α-phthalimyl-α'-ketoethers 2

Procedure is adapted and modified from *Tetrahedron: Asymmetry* **2007**, *18*, 1202. A solution of the alcohol (ca. 3.44 mmol, 1 equiv.) in acetone (5.2 mL) was cooled in an ice-water bath before the dropwise addition of a solution of chromic acid [prepared from CrO_3 (4.89 mmol, 1.45 equiv.), concentrated sulfuric acid (0.08 mL) and water (0.6 mL)]. Mixture was removed from the ice bath then stirred at room temperature for 1 hour before dropwise addition of isopropyl alcohol to quench the reaction. The mixture was filtered through Celite and the filter cake washed with excess acetone before subjecting the filtrate to rotary evaporation. The residue obtained is subsequently purified by flash chromatography on silica to give the desired ketone.

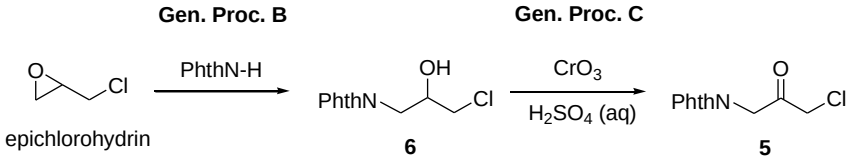
(4) General Procedure D: Epoxide ring opening with alkyl alcohols

Procedure was adapted and modified from Patent number: CN105218324 A. To a round bottom flask containing the alcohol (ca. 10 mmol, 1 equiv.) and dichloromethane (20 mL; when R= Me, ^{i}Pr, neat conditions) was cooled to 0 ^{o}C before the addition of catalytic amounts of BF_3•OEt_2 (0.01 equiv.). Epichlorohydrin (1.5 equiv.) was subsequently added dropwise over 5 minutes and was allowed to stir for 20 hours at rt. Upon completion, solution was subjected to rotary evaporation to remove volatiles before purification of the crude product by Kugelrohr distillation.

(5) General Procedure E: Preparation of *α*-phthalimyl-*α'*-alkyloxy alcohols

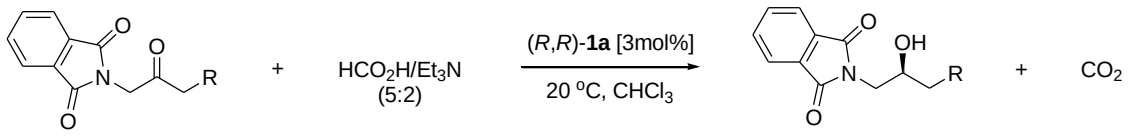
A mixture of halohydrin (ca. 6 mmol, 1 equiv.), tetrabutylammonium bromide (0.5 equiv.), saturated sodium hydroxide (1 equiv.) and toluene (ca. 15 mL) was stirred at 80 ^{o}C for 1.5 hours. Additional toluene (50 mL) was added before charging with phthalimide (1 equiv.) and the solution refluxed overnight. Upon completion, the solution was subject to solvent strip under reduced pressure then the residue purified by flash chromatography on silica to afford the desired alcohols.

(6) Preparation of 2-(3-chloro-2oxopropyl)isoindoline-1,3-dione 5 & *rac*-halohydrin 6 from epichloridrin



5 and **6** are prepared in accordance to **General Procedure C** and **B** respectively from epichlorohydrin instead of the glycidyl ethers.

(7) General Procedure F: Chiral tethered Ru/TsDPEN 1a catalyzed asymmetric transfer hydrogenation of 2 and 5



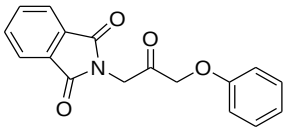
R = O-alkyl/aryl (**2**)
Cl (**5**)

R = O-alkyl/aryl (**3**)
Cl (**6**)

To a nitrogen flushed Schlenk tube was charged with ketone **2**,**5** (0.10 - 0.20 mmol) and catalyst (*R*,*R*)-**1a** (3 mol%) before the addition of equivalent volumes of chloroform and 5:2 formic acid/triethylamine solution (TEAF) such that the total concentration of the ketone is 1M (unless otherwise stated). Reaction is allowed to stir overnight (>15 hrs) at room temperature (ca. 20 ^{o}C)

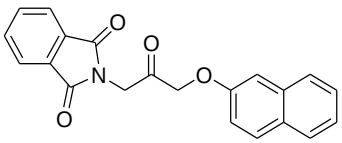
before quenching with excess saturated sodium bicarbonate solution and subsequently extracting the mixture with ethyl acetate (2 x 3 mL). The combined organic layers were concentrated then purified by flash chromatography on silica to afford the desired chiral alcohols.

(8A) Characterization of compounds - Ketones



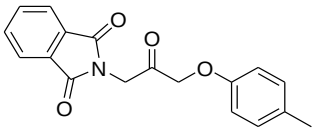
2a

Prepared in accordance to **General Procedure A**-**C** (white solid, 134 mg, 48%): ^{1}H ($CDCl_3$, 500 MHz): δ 4.72 (s, 2H), 4.85 (s, 2H), 6.95 (d, 2H, *J* = 9Hz), 7.05 (t, 1H, *J* = 8Hz), 7.34-7.27 (m, 2H), 7.74-7.90 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 45.26 (1C), 72.43 (1C), 114.78 (2C), 122.47 (1C), 123.84 (2C), 130.12 (2C), 132.35 (2C), 134.45 (2C), 157.63 (1C), 167.89 (2C), 200.29 (1C); Melting range: 164-166 ^{o}C; HRMS (ESI) calcd. for $C_{17}H_{12}NO_4$ $[M-H]^-$: 294.0772, found 294.0782.



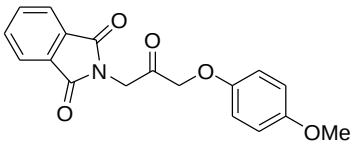
2b

Prepared in accordance to **General Procedure A-C** (white solid, 28.6 mg, 28%): ^{1}H ($CDCl_3$, 400 MHz): δ 4.84 (s, 2H), 4.91 (s, 2H), 7.14-7.15 (m, 1H), 7.24 (m, 1H), 7.40 (t, 1H, *J* = 7Hz), 7.49 (t, 1H, *J* = 7Hz), 7.74-7.90 (m, 7H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 45.30 (1C), 72.47 (1C), 107.46 (1C), 118.46 (1C), 123.86 (2C), 124.66 (1C), 127.04 (1C), 127.28 (1C), 127.95 (1C), 129.80 (1C), 130.33 (1C), 132.35 (2C), 134.46 (2C), 134.59 (1C), 155.53 (1C), 167.89 (2C), 200.12 (1C); Melting range: 195-198 ^{o}C; HRMS (ESI) calcd. for $C_{21}H_{15}NO_4Na$ $[M+Na]^+$: 368.0893, found 368.0892.



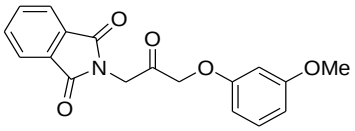
2c

Prepared in accordance to **General Procedure A-C** (white solid, 267 mg, 45%): ^{1}H ($CDCl_3$, 500 MHz): δ 2.31 (s, 3H), 4.69 (s, 2H), 4.84 (s, 2H), 6.85 (d, 2H, *J* = 9Hz), 7.14 (d, 2H, *J* = 9Hz), 7.74-7.89 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 20.75 (1C), 45.27 (1C), 72.65 (1C), 114.60 (2C), 123.82 (2C), 130.52 (2C), 131.80 (1C), 132.35 (2C), 134.42 (2C), 155.58 (1C), 167.89 (2C), 200.57 (1C); Melting range: 166-169 ^{o}C; HRMS (ESI) calcd. for $C_{18}H_{15}NO_4Na$ $[M+Na]^+$: 332.0893, found 332.0896.



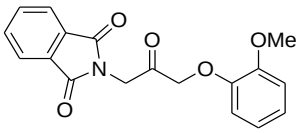
2d

Prepared in accordance to **General Procedure A-C** (white solid, 38.5 mg, 26%): ^{1}H ($CDCl_3$, 500 MHz): δ 3.79 (s, 3H), 4.67 (s, 2H), 4.84 (s, 2H), 6.89 (m, 4H), 7.74-7.89 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 45.26 (1C), 55.96 (1C), 73.26 (1C), 115.19 (2C), 115.84 (2C), 123.84 (2C), 132.36 (2C), 134.44 (2C), 151.81 (1C), 155.06 (1C), 167.90 (2C), 200.58 (1C); Melting range: 132-134 ^{o}C; HRMS (ESI) calcd. for $C_{18}H_{14}NO_5$ $[M-H]^-$: 324.0877, found 324.0879.



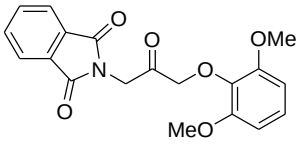
2e

Prepared in accordance to **General Procedure A-C** (white solid, 43.0 mg, 7%): ^{1}H ($CDCl_3$, 500 MHz): δ 3.82 (s, 3H), 4.70 (s, 2H), 4.84 (s, 2H), 6.52-6.53 (m, 2H), 6.60-6.62 (m, 1H), 7.23-7.24 (m, 1H, coincide with $CHCl_3$), 7.74-7.89 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 45.25 (1C), 55.63 (1C), 72.48 (1C), 101.44 (1C), 106.59 (1C), 108.17 (1C), 123.85 (2C), 130.58 (1C), 132.35 (2C), 134.45 (2C), 158.85 (1C), 161.31 (1C), 167.88 (2C), 200.21 (1C); Melting range: 156-158 ^{o}C; HRMS (ESI) calcd. for $C_{18}H_{15}NO_5Na$ $[M+Na]^+$: 348.0842, found 348.0839.



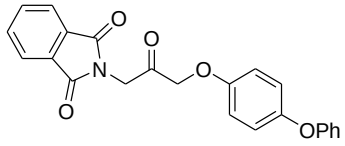
2f

Prepared in accordance to **General Procedure A-C** (white solid, 85.2 mg, 10%): ^{1}H ($CDCl_3$, 500 MHz): δ 3.91 (s, 3H), 4.73 (s, 2H), 4.93 (s, 2H), 6.93-6.97 (m, 3H), 7.04-7.07 (m, 1H), 7.74-7.89 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 45.35 (1C), 55.95 (1C), 74.93 (1C), 112.55 (1C), 116.50 (1C), 121.24 (1C), 123.74 (1C), 123.77 (2C), 132.43 (2C), 134.37 (2C), 147.54 (1C), 150.32 (1C), 167.96 (2C), 201.16 (1C); Melting range: 170-173 ^{o}C; HRMS (ESI) calcd. for $C_{18}H_{15}NO_5Na$ $[M+Na]^+$: 348.0842, found 348.0842.



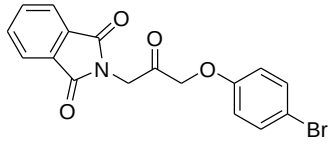
2g

Prepared in accordance to **General Procedure A-C** (white solid, 107 mg, 26%): ^{1}H ($CDCl_3$, 500 MHz): δ 3.90 (s, 6H), 4.63 (s, 2H), 5.16 (s, 2H), 6.61 (d, 2H, *J* = 9Hz), 7.04 (t, 1H, *J* = 8Hz), 7.73-7.89 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 45.69 (1C), 56.17 (2C), 78.30 (1C), 105.21 (2C), 123.66 (2C), 124.55 (1C), 132.55 (2C), 134.26 (2C), 137.34 (1C), 152.96 (2C), 168.12 (2C), 202.26 (1C); Melting range: 135-136 ^{o}C; HRMS (ESI) calcd. for $C_{19}H_{17}NO_6Na$ $[M+Na]^+$: 378.0948, found 378.0949.



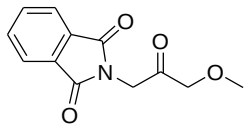
2h

Prepared in accordance to **General Procedure A-C** (white solid, 372 mg, 63%): ^{1}H ($CDCl_3$, 500 MHz): δ 4.71 (s, 2H), 4.85 (s, 2H), 6.92-7.34 (m, 9H), 7.75-7.90 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 45.22 (1C), 73.01 (1C), 115.99 (2C), 118.27 (2C), 121.04 (2C), 123.09 (1C), 123.85 (2C), 129.95 (2C), 132.33 (2C), 134.47 (2C), 151.85 (1C), 153,76 (1C), 158.22 (1C), 167.88 (2C), 200.13 (1C); Melting range: 133-136 ^{o}C; HRMS (ESI) calcd. for $C_{23}H_{17}NO_5Na$ $[M+Na]^+$: 410.0999, found 410.0997.



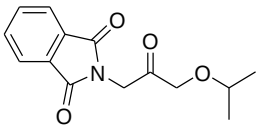
2i

Prepared in accordance to **General Procedure A-C** (white solid, 182 mg, 82%): ^{1}H ($CDCl_3$, 500 MHz): δ 4.70 (s, 2H), 4.81 (s, 2H), 6.84 (d, 2H, *J* = 9 Hz), 7.45 (d, 2H, *J* = 9Hz), 7.75-7.90 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 45.14 (1C), 72.51 (1C), 114.84 (1C), 116.60 (2C), 123.88 (2C), 132.30 (2C), 132.97 (2C), 134.51 (2C), 156.73 (1C), 167.85 (2C), 199.57 (1C); Melting range: 165-166 ^{o}C; HRMS (ESI) calcd. for $C_{17}H_{12}NO_4BrNa$ $[M+Na]^+$: 395.9846, 397.9828, found 395.9842, 397.9823.



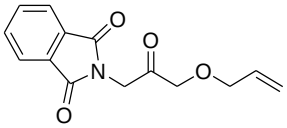
2j

Prepared in accordance to **General Procedure D,E,C** (white solid, 443 mg, 83%): ^{1}H ($CDCl_3$, 500 MHz): δ 3.49 (s, 3H), 4.14 (s, 2H), 4.69 (s, 2H), 7.73-7.88 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 44.88 (1C), 59.87 (1C), 77.01 (1C, coincide with $CDCl_3$ signal), 123.78 (2C), 132.37 (2C), 134.38 (2C), 167.93 (2C), 201.42 (1C); Melting range: 82-85 ^{o}C; HRMS (ESI) calcd. for $C_{12}H_{11}NO_4Na$ $[M+Na]^+$: 256.0580, found 256.0572.



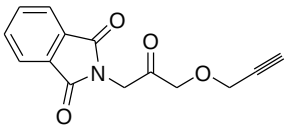
2k

Prepared in accordance to **General Procedure D,E,C** (white solid, 628 mg, 72%): ^{1}H ($CDCl_3$, 500 MHz): δ 1.24 (d, 2H, J = 6Hz), 3.67-3.72 (m, 1H), 4.16 (s, 2H), 4.73 (s, 2H), 7.73-7.88 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 22.06 (2C), 45.23 (1C), 73.11 (1C), 73.41 (1C), 123.74 (2C), 132.41 (2C), 134.33 (2C), 168.01 (2C), 202.39 (1C); Melting range: 59-61 ^{o}C; HRMS (ESI) calcd. for $C_{14}H_{14}NO_4$ $[M-H]^-$: 260.0928, found 260.0927.



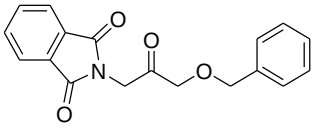
2l

Prepared in accordance to **General Procedure D,E,C** (white solid, 142 mg, 43%): ^{1}H ($CDCl_3$, 500 MHz): δ 4.12 (d, 2H, J = 6Hz), 4.19 (s, 2H), 4.72 (s, 2H), 5.28 (dd, 1H, J = 1, 11Hz), 5.36 (dd, 1H, J = 1, 17Hz), 5.90-5.97 (m, 1H), 7.72-7.88 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 45.10 (1C), 72.91 (1C), 74.46 (1C), 118.73 (1C), 123.78 (2C), 132.39 (2C), 133.59 (1C), 134.38 (2C), 167.96 (2C), 201.56 (1C); Melting range: 84-86 ^{o}C; HRMS (ESI) calcd. for $C_{14}H_{14}NO_4$ $[M+H]^+$: 260.0917, found 260.0917.



2m

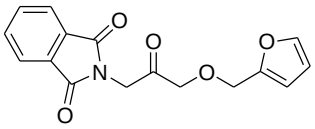
Prepared in accordance to **General Procedure D,E,C** (white solid, 352 mg, 76%): ^{1}H ($CDCl_3$, 500 MHz): δ 2.55 (t, 1H, J = 3Hz), 4.30 (s, 2H), 4.31 (d, 2H, J = 3 Hz), 4.72 (s, 2H), 7.73-7.88 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 45.09 (1C), 59.11 (1C), 73.79 (1C), 76.51 (1C), 78.35 (1C), 123.77 (2C), 132.35 (2C), 134.38 (2C), 167.89 (2C), 200.73 (1C); Melting range: 98-101 ^{o}C; HRMS (ESI) calcd. for $C_{14}H_{11}NO_4Na$ $[M+Na]^+$: 280.0580, found 280.0583.



2n

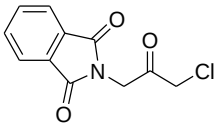
Prepared in accordance to **General Procedure D,E,C** (white solid, 293 mg, 39%): ^{1}H ($CDCl_3$, 500 MHz): δ 4.21 (s, 2H), 4.65 (s, 2H), 4.74 (s, 2H), 7.34-7.39 (m, 5H), 7.73-7.88 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 45.12 (1C), 74.03 (1C), 74.50 (1C), 123.79 (2C), 128.23 (2C), 128.51 (1C), 128.90 (2C), 132.38

(2C), 134.38 (2C), 136.94 (1C), 167.95 (2C), 201.39 (1C); Melting range: 100-102 °C; HRMS (ESI) calcd. for $C_{18}H_{15}NO_4Na$ $[M+Na]^+$: 332.0893, found 332.0897.



2o

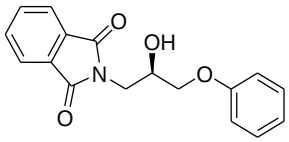
Prepared in accordance to **General Procedure D,E,C** (pale yellow oil, 148 mg, 14%): ^1H ($CDCl_3$, 500 MHz): δ 4.20 (s, 2H), 4.60 (s, 2H), 4.68 (s, 2H), 6.38-6.40 (m, 2H), 7.46 (d, 1H, J = 1Hz), 7.73-7.88 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 45.04 (1C), 65.49 (1C), 74.07 (1C), 110.71 (1C), 110.84 (1C), 123.77 (2C), 132.38 (2C), 134.36 (2C), 143.72 (1C), 150.51 (1C), 167.92 (2C), 201.33 (1C); HRMS (ESI) calcd. for $C_{16}H_{13}NO_5Na$ $[M+Na]^+$: 322.0686, found 322.0688.



5

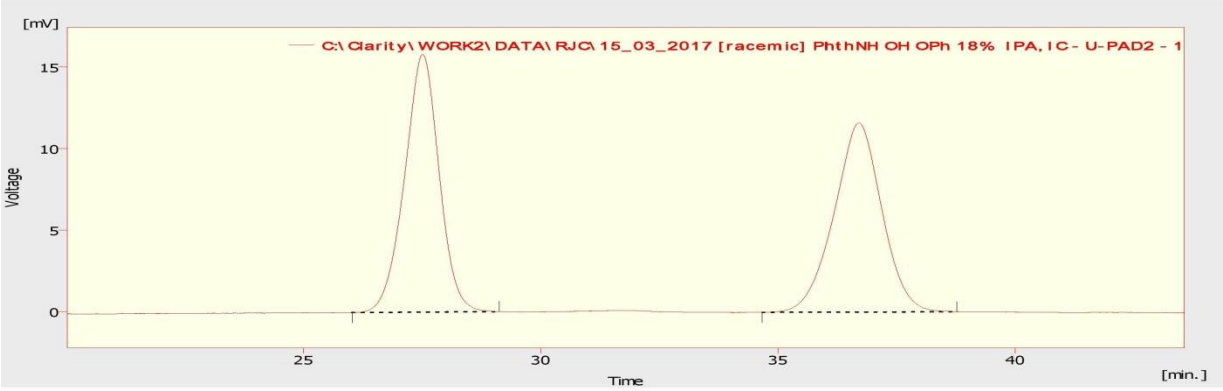
Prepared in accordance to **General Procedure C,B** (white solid, 160 mg, 42%): ^1H ($CDCl_3$, 400 MHz): δ 4.22 (s, 2H), 4.77 (s, 2H), 7.75-7.90 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 44.93 (1C), 46.42 (1C), 123.93 (2C), 132.22 (2C), 134.57 (2C), 167.67 (2C), 195.70 (1C); Melting range: 141-145 °C; HRMS (ESI) calcd. for $C_{11}H_8NO_3ClNa$ $[M+Na]^+$: 260.0085, 262.0055, found 260.0085, 260.0056.

(8B) Characterization of compounds – Optically active alcohols



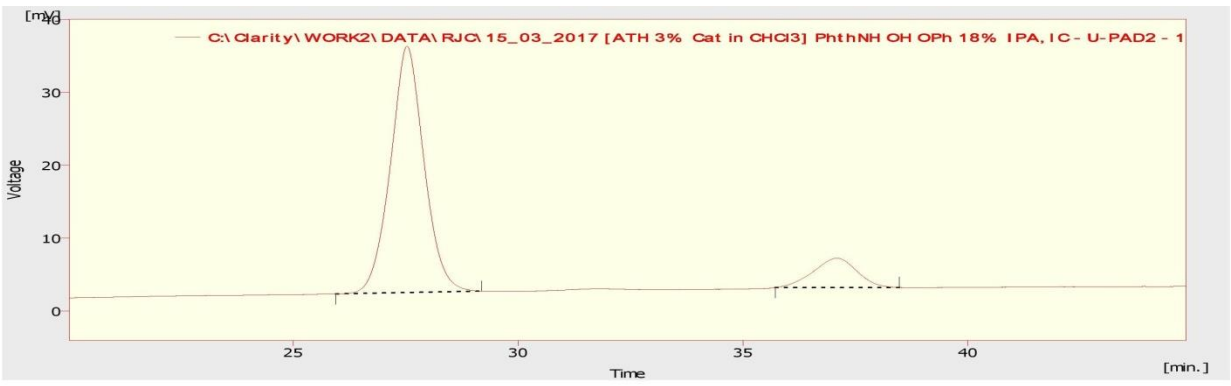
(*R*)-**3a**

Prepared in accordance to **General Procedure F** except that [**2a**]=0.5M, v/v TEAF:$CHCl_3$=1:3 (white solid, 44.1 mg, 91%, *ee* = 73%): ^{1}H ($CDCl_3$, 400 MHz): δ 2.82 (d, 1H, *J* = 6Hz), 3.94-4.09 (m, 4H), 4.29-4.33 (m, 1H), 6.92 (d, 1H, *J* = 8Hz), 6.97 (1H, t, *J* = 7Hz), 7.26-7.30 (m, 2H, coincide with $CHCl_3$ signal), 7.72-7.89 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 41.52 (1C), 69.13 (1C), 69.86 (1C), 114.85 (2C), 121.60 (1C), 123.75 (2C), 129.78 (2C), 132.20 (2C), 134.43 (2C), 158.58 (1C), 169.02 (2C); Melting range: 121-123 °C; HRMS (ESI) calcd. for $C_{17}H_{14}NO_4$ $[M-H]^-$: 296.0928, found 296.0922; HPLC (Diacel IC column, Hexane:IPA = 82:18, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 27.5 min, t_2 = 37.1 min; $[\alpha]_D^{27}$ = +28.32° (c = 1.01, $CHCl_3$).



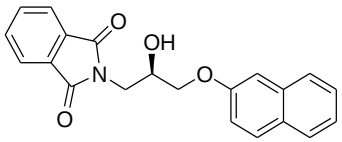
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\15_03_2017 [racemic] PhthNH OH OPh 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	27.516	802.504	15.805	49.7	57.7	0.77	
2	36.704	812.321	11.604	50.3	42.3	1.07	
	Total	1614.824	27.408	100.0	100.0		



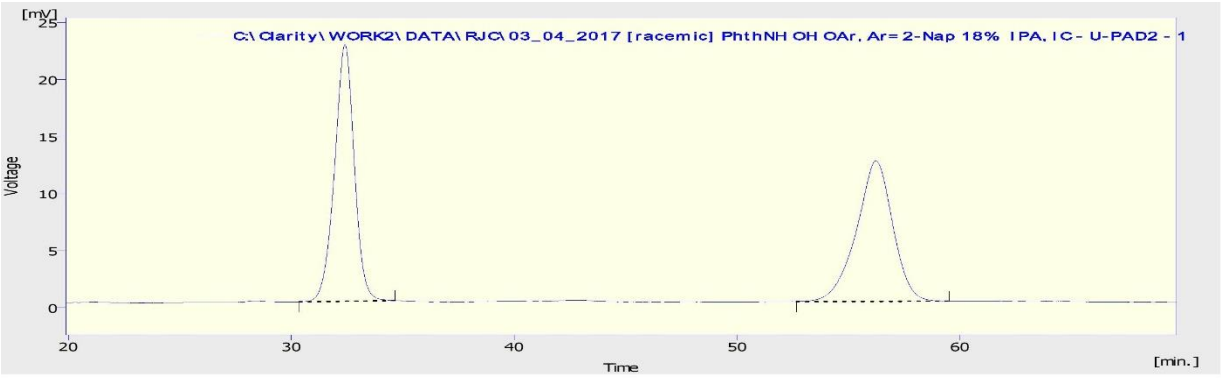
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\15_03_2017 [ATH 3% Cat in CHCl3] PhthNH OH OPh 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	27.532	1755.485	33.803	86.6	89.3	0.78	
2	37.084	271.368	4.031	13.4	10.7	1.06	
	Total	2026.853	37.834	100.0	100.0		



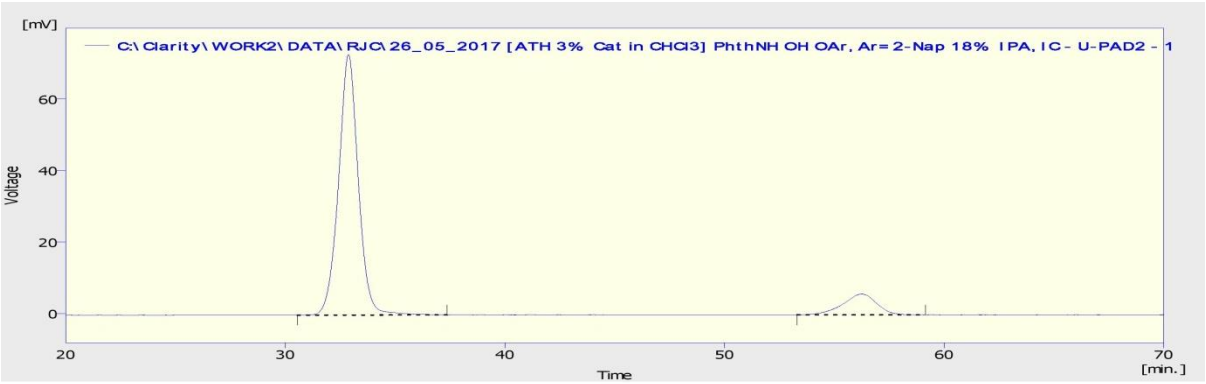
(*R*)-**3b**

Prepared in accordance to **General Procedure F** except that [**2b**]=0.066M, v/v TEAF:$CHCl_3$=1:28 (white solid, 24.6 mg, 92%, *ee* = 76%): ^1H ($CDCl_3$, 500 MHz): δ 2.90 (d, 1H, *J* = 6Hz), 4.00-4.21 (m, 4H), 4.36-4.40 (m, 1H), 7.15-7.89 (m, 11H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 41.58 (1C), 69.12 (1C), 69.92 (1C), 107.21 (1C), 118.86 (1C), 123.77 (2C), 124.13 (1C), 126.71 (1C), 127.08 (1C), 127.89 (1C), 129.46 (1C), 129.80 (1C), 132.20 (2C), 134.45 (2C), 134.63 (1C), 156.52 (1C), 169.05 (2C); Melting range: 164-167 ^oC; HRMS (ESI) calcd. for $C_{21}H_{17}NO_4Na$ $[M+Na]^+$: 370.1050, found 370.1044; HPLC (Diacel IC column, Hexane:IPA = 82:18, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 32.9 min, t_2 = 56.2 min; $[\alpha]_D^{32}$ = +17.30^o (c = 1.00, $CHCl_3$).



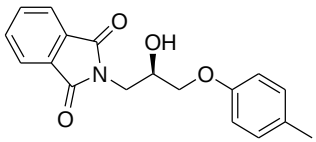
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\03_04_2017 [racemic] PhthNH OH OAr, Ar=2-Nap 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	32.408	1390.672	22.512	49.9	64.6	0.93	
2	56.228	1397.182	12.335	50.1	35.4	1.70	
	Total	2787.854	34.846	100.0	100.0		



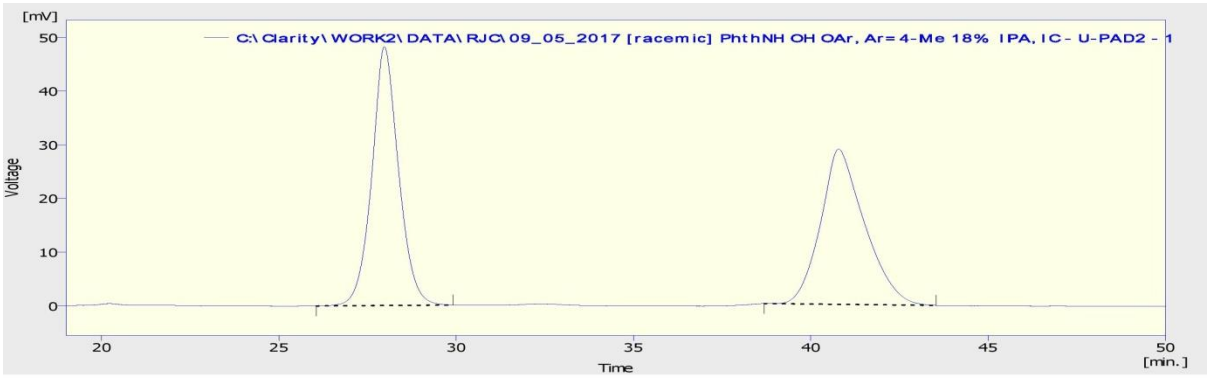
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\26_05_2017 [ATH 3% Cat in CHCl3] PhthNH OH OAr, Ar=2-Nap 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	32.876	4556.694	72.846	87.9	92.6	0.93	
2	56.244	627.922	5.829	12.1	7.4	1.63	
	Total	5184.616	78.675	100.0	100.0		



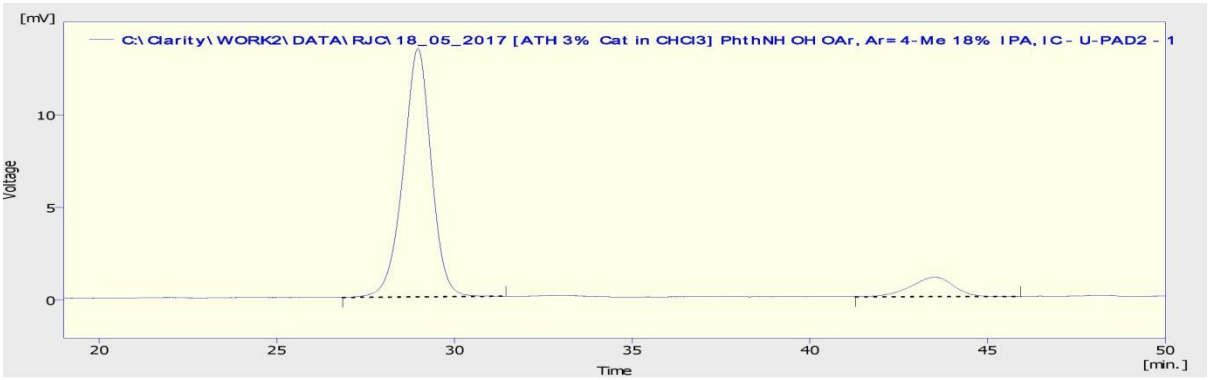
(*R*)-**3c**

Prepared in accordance to **General Procedure F** (off-white solid, 31.6 mg, 95%, *ee* = 78%): ^{1}H ($CDCl_3$, 500 MHz): δ 2.28 (s, 3H), 2.81 (d, 1H, *J* = 7Hz), 3.93-4.06 (m, 4H), 4.27-4.31 (m, 1H), 6.81 (d, 2H, *J* = 9Hz), 7.07 (d, 2H, *J* = 9Hz), 7.73-7.88 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 20.72 (1C), 41.52 (1C), 69.12 (1C), 70.07 (1C), 114.72 (2C), 123.73 (2C), 130.20 (2C), 130.86 (1C), 132.21 (2C), 134.40 (2C), 156.50 (1C), 169.00 (2C); Melting range: 108-109 ^{o}C; HRMS (ESI) calcd. for $C_{18}H_{17}NO_4Na$ $[M+Na]^+$: 334.1050, found 334.1051; HPLC (Diacel IC column, Hexane:IPA = 82:18, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 29.0 min, t_2 = 43.5 min; $[\alpha]_D^{26}$ = +22.84^{o} (c = 1.02, $CHCl_3$).



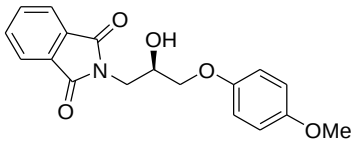
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\09_05_2017 [racemic] PhthNH OH OAr, Ar=4-Me 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	27.972	2534.781	48.245	50.4	62.5	0.78	
2	40.784	2493.115	28.924	49.6	37.5	1.30	
	Total	5027.897	77.168	100.0	100.0		



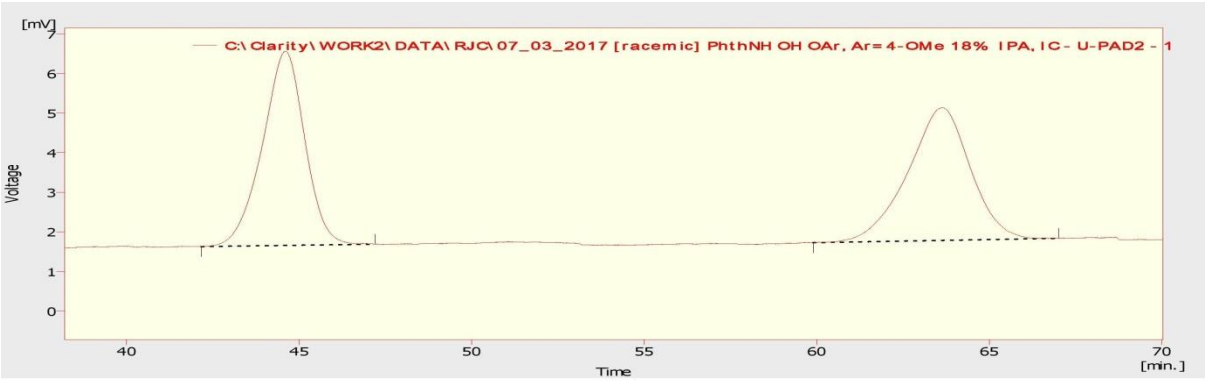
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\18_05_2017 [ATH 3% Cat in CHCl3] PhthNH OH OAr, Ar=4-Me 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	28.968	735.236	13.398	89.2	92.7	0.82	
2	43.520	88.863	1.057	10.8	7.3	1.29	
	Total	824.099	14.455	100.0	100.0		



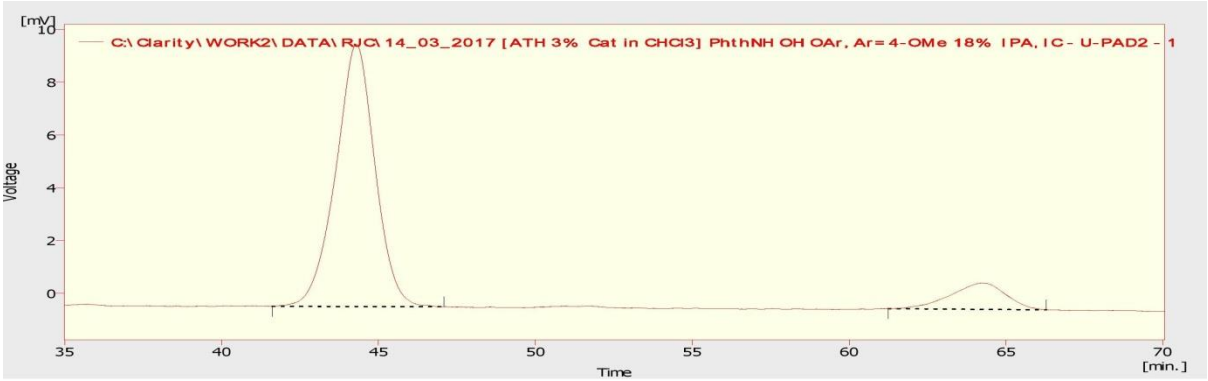
(*R*)-**3d**

Prepared in accordance to **General Procedure F** (white solid, 38.9 mg, 88%, *ee* = 75%): ^{1}H ($CDCl_3$, 500 MHz): δ 2.81 (d, 1H, *J* = 6Hz), 3.77 (s, 3H), 3.93-4.05 (m, 4H), 4.26-4.29 (m, 1H), 6.84 (q, 4H, *J* = 9Hz), 7.73-7.88 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 41.51 (1C), 55.96 (1C), 69.16 (1C), 70.72 (1C), 114.92 (2C), 115.92 (2C), 123.73 (2C), 132.21 (2C), 134.41 (2C), 152.76 (1C), 154.51 (1C), 169.01 (2C); Melting range: 144-147 °C; HRMS (ESI) calcd. for $C_{18}H_{16}NO_5$ [M-H]$^-$: 326.1034, found 326.1035; HPLC (Diacel IC column, Hexane:IPA = 82:18, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 44.3 min, t_2 = 64.2 min; $[\alpha]_D^{27}$ = +18.31° (c = 1.02, $CHCl_3$).



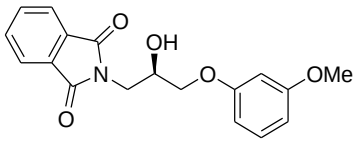
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\07_03_2017 [racemic] PhthNH OH OAr, Ar=4-OMe 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	44.588	419.394	4.906	49.8	59.4	1.30	
2	63.668	422.974	3.351	50.2	40.6	1.90	
	Total	842.368	8.257	100.0	100.0		



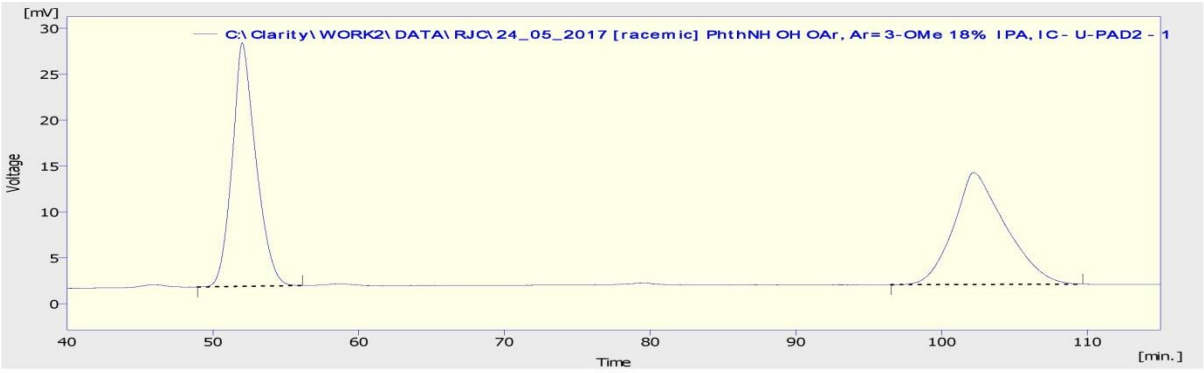
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\14_03_2017 [ATH 3% Cat in CHCl3] PhthNH OH OAr, Ar=4-OMe 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	44.296	866.546	9.944	87.6	90.9	1.32	
2	64.192	122.383	0.999	12.4	9.1	1.91	
	Total	988.929	10.942	100.0	100.0		



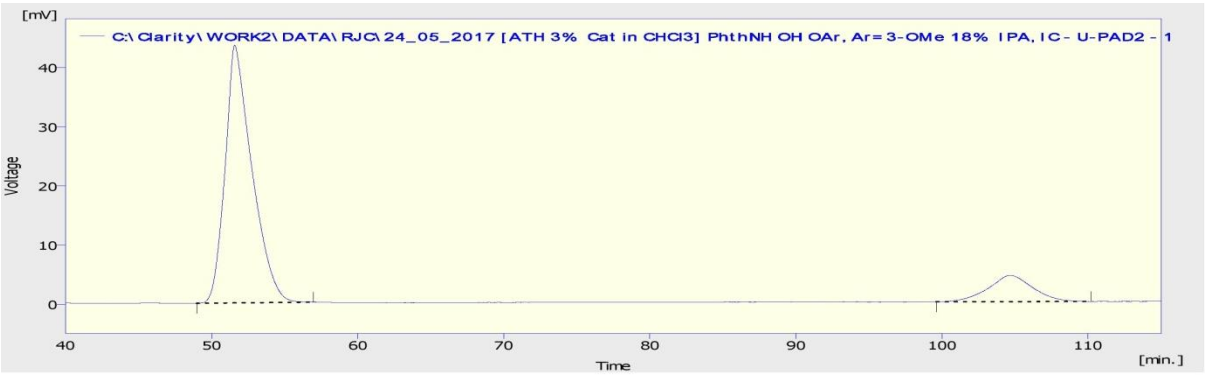
(*R*)-**3e**

Prepared in accordance to **General Procedure F** except that [**2e**]=0.5M, v/v TEAF:$CHCl_3$=1:3 (pale brown viscous oil, 30.4 mg, 88%, *ee* = 71%): ^1H ($CDCl_3$, 500 MHz): δ 2.81 (d, 1H, *J* = 5Hz), 3.78 (s, 3H), 3.94-4.07 (m, 4H), 4.30 (m, 1H), 6.47-6.54 (m, 3H), 7.17 (t, 1H, *J* = 8Hz), 7.73-7.88 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 41.54 (1C), 55.55 (1C), 69.08 (1C), 69.95 (1C), 101.36 (1C), 106.84 (1C), 107.31 (1C), 123.75 (2C), 130.21 (1C), 132.20 (2C), 134.43 (2C), 159.83 (1C), 161.08 (1C), 169.01 (2C); HRMS (ESI) calcd. for $C_{18}H_{17}NO_5Na$ $[M+Na]^+$: 350.0999, found 350.1007; HPLC (Diacel IC column, Hexane:IPA = 82:18, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 51.6 min, t_2 = 104.7 min; $[\alpha]_D^{26}$ = +17.47° (c = 1.01, $CHCl_3$).



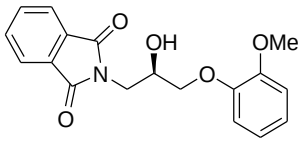
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\24_05_2017 [racemic] PhthNH OH OAr, Ar=3-OMe 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	52.012	3008.489	26.539	49.9	68.5	1.70	
2	102.176	3022.801	12.184	50.1	31.5	3.74	
	Total	6031.290	38.723	100.0	100.0		



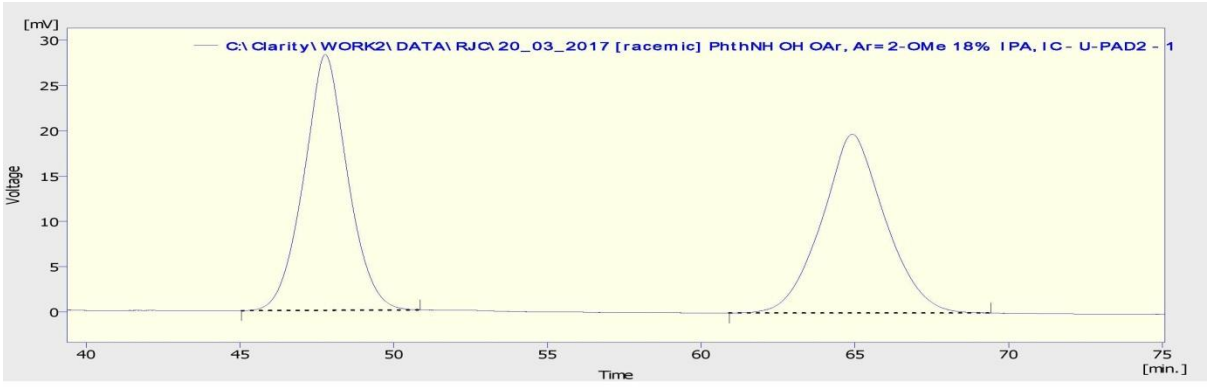
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\24_05_2017 [ATH 3% Cat in CHCl3] PhthNH OH OAr, Ar=3-OMe 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	51.580	5568.857	43.571	85.6	90.8	1.92	
2	104.684	934.783	4.426	14.4	9.2	3.15	
	Total	6503.640	47.997	100.0	100.0		



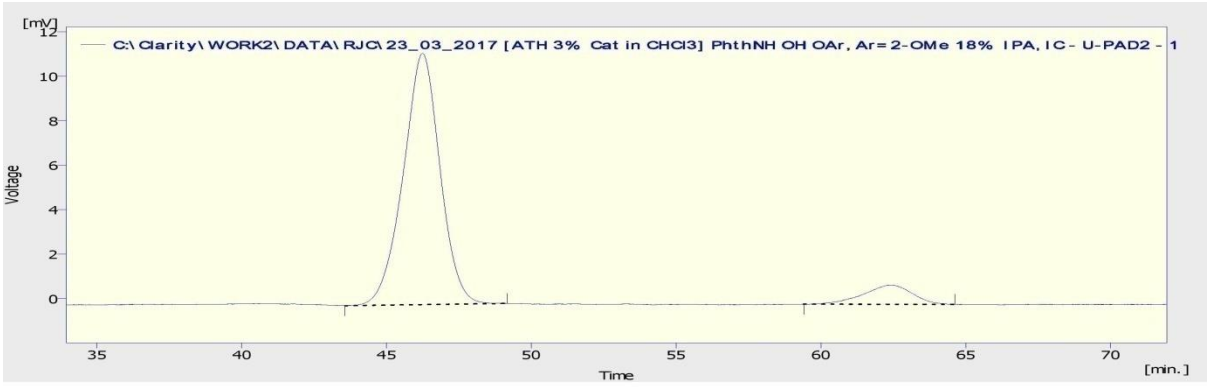
(*R*)-**3f**

Prepared in accordance to **General Procedure F** (white solid, 40.3 mg, 92%, *ee* = 82%): ^{1}H ($CDCl_3$, 500 MHz): δ 3.28 (d, 1H, *J* = 5Hz), 3.82 (s, 3H), 3.89-4.14 (m, 4H), 4.28-4.29 (m, 1H), 6.87-6.97 (m, 4H), 7.71-7.87 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 41.15 (1C), 56.04 (1C), 68.93 (1C), 72.97 (1C), 112.32 (1C), 116.45 (1C), 121.28 (1C), 122.87 (1C), 123.62 (2C), 132.26 (2C), 134.28 (2C), 148.32 (1C), 150.86 (1C), 168.86 (2C); Melting range: 109-111 ^{o}C; HRMS (ESI) calcd. for $C_{18}H_{17}NO_5Na$ $[M+Na]^+$: 350.0999, found 350.1000; HPLC (Diacel IC column, Hexane:IPA = 82:18, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 46.2 min, t_2 = 62.4 min; $[\alpha]_D^{29}$ = +12.35^{o} (c = 1.00, $CHCl_3$).



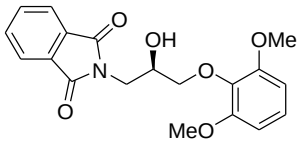
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\20_03_2017 [racemic] PhthNH OH OAr, Ar=2-OMe 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	47.780	2803.701	28.250	49.8	58.9	1.50	
2	64.916	2831.774	19.740	50.2	41.1	2.16	
	Total	5635.475	47.990	100.0	100.0		



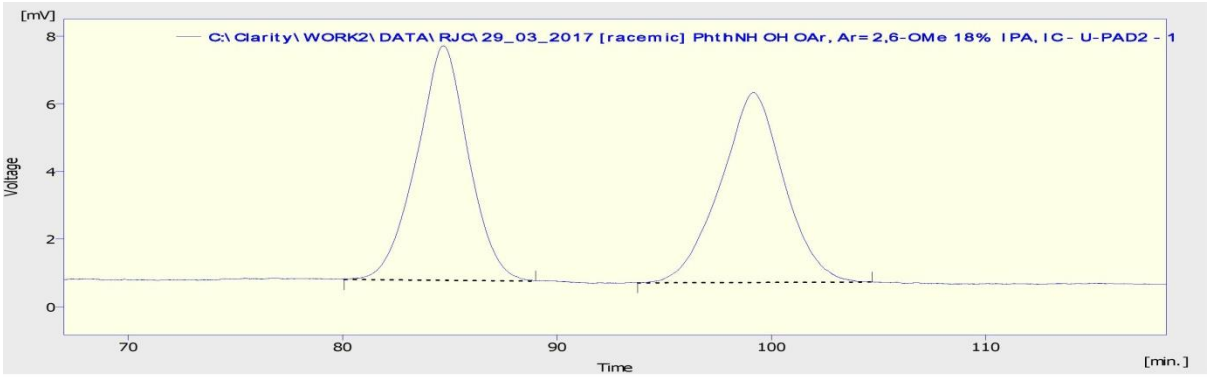
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\23_03_2017 [ATH 3% Cat in CHCl3] PhthNH OH OAr, Ar=2-OMe 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	46.240	999.882	11.300	90.8	92.9	1.34	
2	62.384	101.544	0.869	9.2	7.1	1.81	
	Total	1101.425	12.169	100.0	100.0		



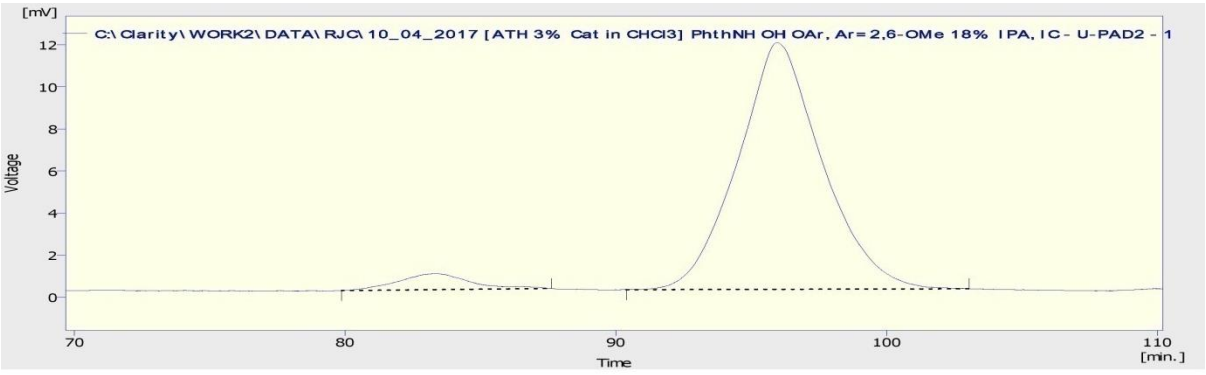
(*R*)-**3g**

Prepared in accordance to **General Procedure F** (white solid, 45.4 mg, 93%, *ee* = 90%): ^{1}H ($CDCl_3$, 500 MHz): δ 3.76-4.20 (m, 5H), 3.86 (s, 6H), 4.14 (br s, 1H), 6.58 (d, 2H, *J* = 9Hz), 7.00 (t, 1H, *J* = 9Hz), 7.70-7.86 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 40.56 (1C), 56.35 (1C), 68.89 (1C), 76.66 (1C), 105.45 (2C), 123.55 (2C), 124.31 (1C), 132.40 (2C), 134.15 (2C), 137.35 (1C), 153.39 (2C), 168.75 (2C); Melting range: 145-148 °C; HRMS (ESI) calcd. for $C_{19}H_{19}NO_6Na$ $[M+Na]^+$: 380.1105, found 380.1108; HPLC (Diacel IC column, Hexane:IPA = 82:18, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 83.4 min, t_2 = 96.0 min; $[\alpha]_D^{24}$ = -22.64° (c = 1.02, $CHCl_3$).



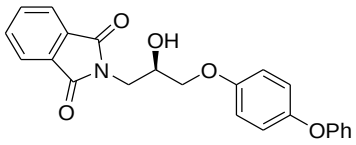
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\29_03_2017 [racemic] PhthNH OH OAr, Ar=2,6-OMe 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	84.692	1169.967	6.940	50.2	55.3	2.56	
2	99.196	1160.975	5.617	49.8	44.7	3.12	
	Total	2330.942	12.557	100.0	100.0		



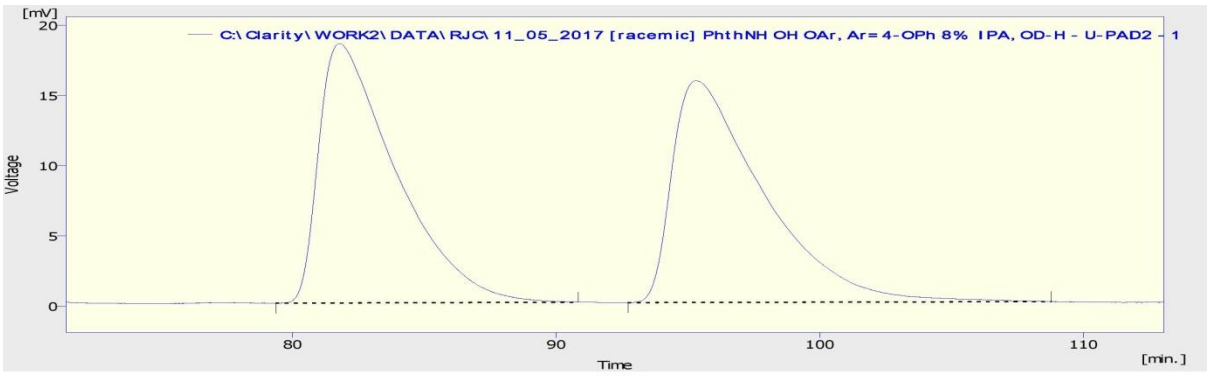
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\10_04_2017 [ATH 3% Cat in CHCl3] PhthNH OH OAr, Ar=2,6-OMe 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	83.376	139.912	0.764	5.2	6.1	2.65	
2	95.952	2538.803	11.727	94.8	93.9	3.24	
	Total	2678.715	12.491	100.0	100.0		



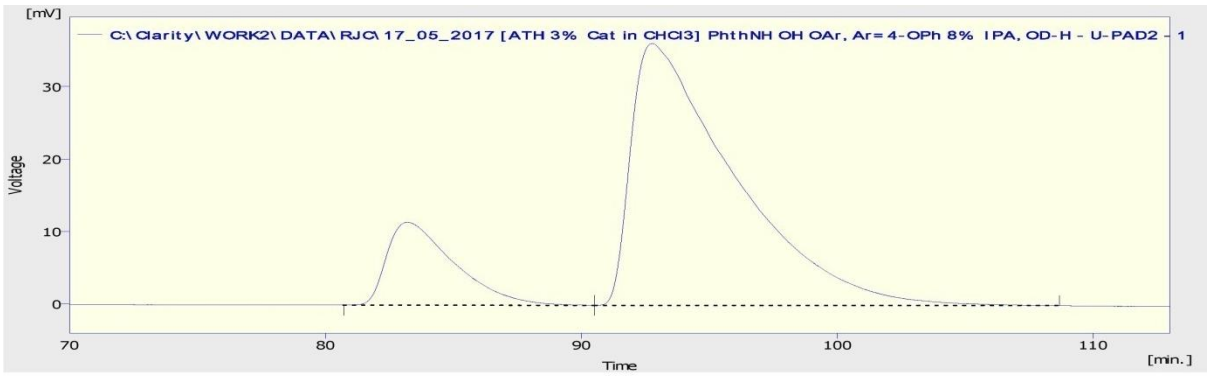
(*R*)-**3h**

Prepared in accordance to **General Procedure F** except that [**2h**]=0.5M, v/v TEAF:$CHCl_3$=1:3 (off-white solid, 35.3 mg, 89%, *ee* = 66%): ^1H ($CDCl_3$, 400 MHz): δ 2.84 (d, 1H, J = 6Hz), 3.95-4.08 (m, 4H), 4.29-4.33 (m, 1H), 6.88-7.32 (m, 9H), 7.73-7.89 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 41.50 (1C), 69.13 (1C), 70.51 (1C), 115.97 (2C), 117.98 (2C), 120.99 (2C), 122.81 (1C), 123.76 (2C), 129.88 (2C), 132.19 (2C), 134.45 (2C), 151.01 (1C), 154.83 (1C), 158.53 (1C), 169.02 (2C); Melting range: 98-101 ^oC; HRMS (ESI) calcd. for $C_{23}H_{19}NO_5Na$ $[M+Na]^+$: 412.1155, found 412.1159; HPLC (Diacel OD-H column, Hexane:IPA = 92:8, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 83.2 min, t_2 = 92.8 min; $[\alpha]_D^{28}$ = +12.44^o (c = 1.01, $CHCl_3$).



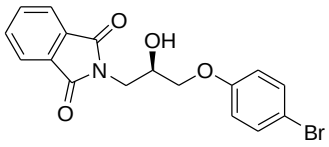
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\11_05_2017 [racemic] PhthNH OH OAr, Ar=4-OPh 8% IPA, OD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	81.796	3745.029	18.471	49.7	53.9	3.06	
2	95.300	3793.142	15.786	50.3	46.1	3.57	
	Total	7538.171	34.258	100.0	100.0		



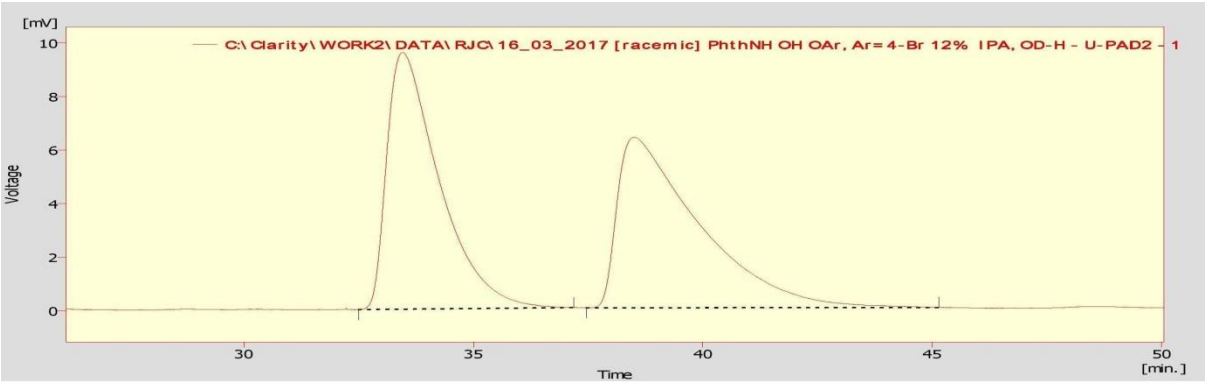
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\17_05_2017 [ATH 3% Cat in CHCl3] PhthNH OH OAr, Ar=4-OPh 8% IPA, OD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	83.204	2085.416	11.459	17.1	24.1	2.78	
2	92.760	10143.610	36.137	82.9	75.9	4.14	
	Total	12229.026	47.596	100.0	100.0		



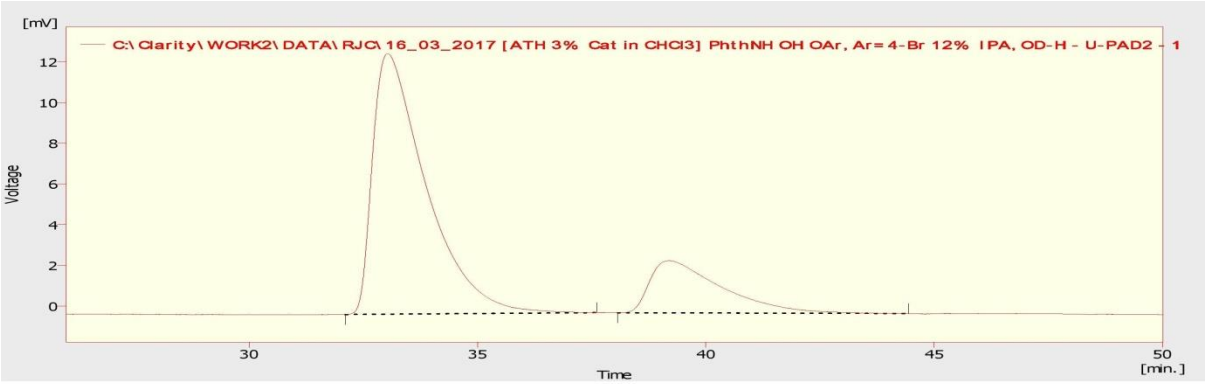
(*R*)-**3i**

Prepared in accordance to **General Procedure F** except that [**2i**]=0.25M, v/v TEAF:$CHCl_3$=1:7 (white solid, 48.7 mg, 95%, *ee* = 58%): ^1H ($CDCl_3$, 500 MHz): δ 2.84 (d, 1H, *J* = 6Hz), 3.94-4.05 (m, 4H), 4.28-4.30 (m, 1H), 6.79 (d, 2H, *J* = 9Hz), 7.37 (d, 2H, *J* = 9Hz), 7.74-7.88 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 41.46 (1C), 69.05 (1C), 70.17 (1C), 113.83 (1C), 116.64 (2C), 123.79 (2C), 132.15 (2C), 132.60 (2C), 134.50 (2C), 157.71 (1C), 169.03 (2C); Melting range: 163-166 °C; HRMS (ESI) calcd. for $C_{17}H_{14}NO_4BrNa$ $[M+Na]^+$: 397.9998, 399.9980, found 398.0001, 399.9981; HPLC (Diacel OD-H column, Hexane:IPA = 88:12, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 33.0 min, t_2 = 39.2 min; $[\alpha]_D^{28}$ = +13.49° (c = 1.01, $CHCl_3$).



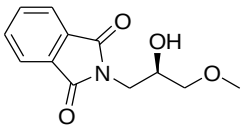
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\16_03_2017 [racemic] PhthNH OH OAr, Ar=4-Br 12% IPA, OD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	33.460	761.107	9.588	49.1	60.1	1.19	
2	38.496	788.846	6.363	50.9	39.9	1.82	
	Total	1549.953	15.951	100.0	100.0		



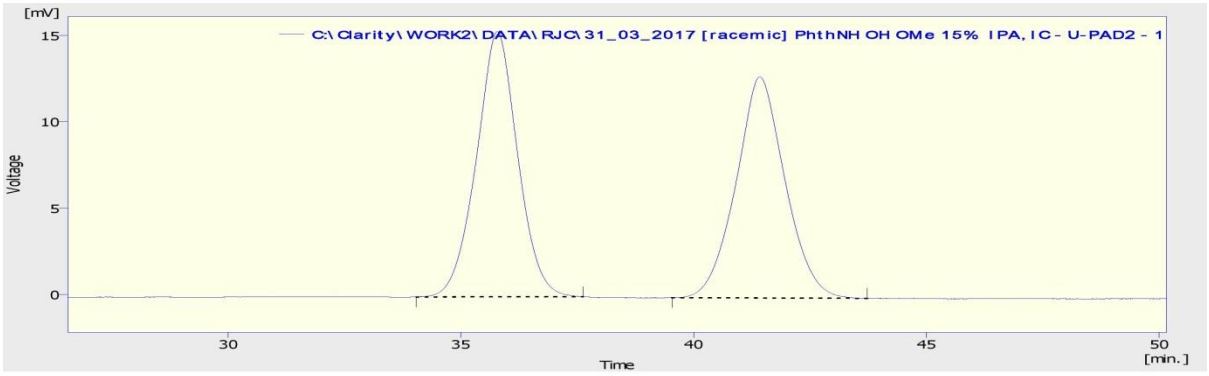
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\16_03_2017 [ATH 3% Cat in CHCl3] PhthNH OH OAr, Ar=4-Br 12% IPA, OD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	33.032	1041.399	12.811	78.8	83.3	1.21	
2	39.204	279.755	2.566	21.2	16.7	1.62	
	Total	1321.154	15.377	100.0	100.0		



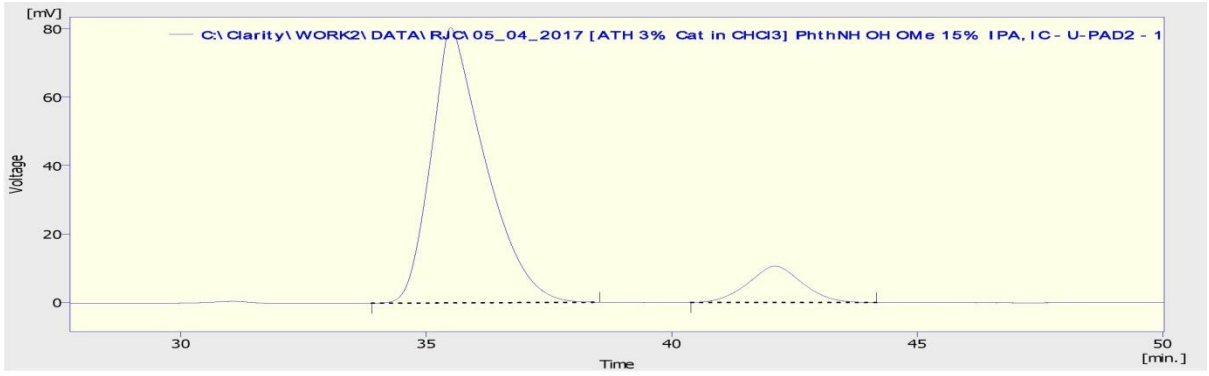
(*R*)-**3j**

Prepared in accordance to **General Procedure F** (viscous colourless oil, 31.2 mg, 90%, *ee* = 77%): ^{1}H ($CDCl_3$, 500 MHz): δ 2.64 (br s, 1H), 3.40 (s, 3H), 3.41-3.51 (m, 2H), 3.79-3.91 (m, 2H), 4.08 (br s, 1H), 7.72-7.87 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 41.40 (1C), 59.54 (1C), 69.19 (1C), 74.47 (1C), 123.66 (2C), 132.25 (2C), 134.32 (2C), 168.96 (2C); HRMS (ESI) calcd. for $C_{12}H_{13}NO_4Na$ $[M+Na]^+$: 258.0737, found 258.0737; HPLC (Diacel IC column, Hexane:IPA = 85:15, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 35.5 min, t_2 = 42.1 min; $[\alpha]_D^{27}$ = +18.80° (c = 0.99, $CHCl_3$).



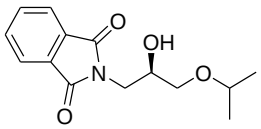
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\31_03_2017 [racemic] PhthNH OH OMe 15% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	35.776	948.102	15.368	50.3	54.5	0.94	
2	41.424	937.264	12.807	49.7	45.5	1.11	
	Total	1885.366	28.174	100.0	100.0		



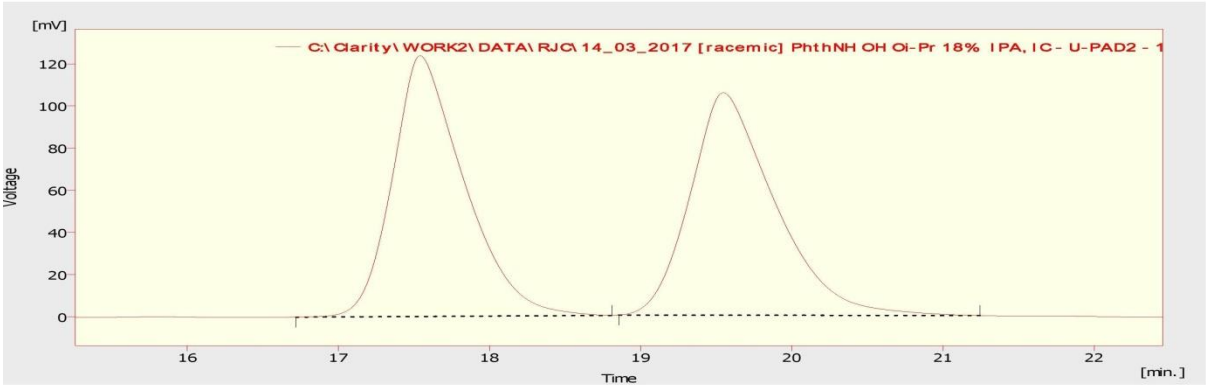
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\05_04_2017 [ATH 3% Cat in CHCl3] PhthNH OH OMe 15% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	35.500	6172.639	80.418	88.3	88.3	1.16	
2	42.096	817.133	10.641	11.7	11.7	1.18	
	Total	6989.773	91.059	100.0	100.0		



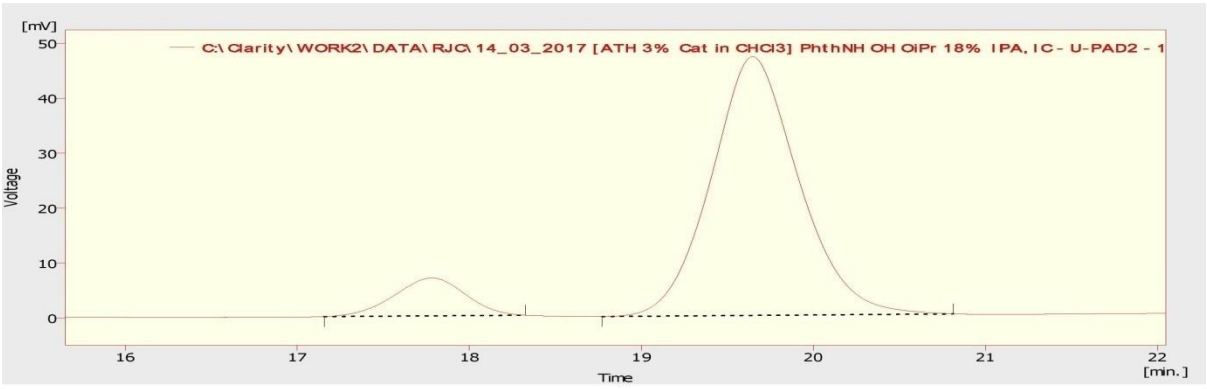
(*R*)-**3k**

Prepared in accordance to **General Procedure F** (pale brown oil, 37.7 mg, 76%, *ee* = 79%): ^{1}H ($CDCl_3$, 500 MHz): δ 1.14 (d, 6H, *J* = 6Hz), 2.70 (d, 1H, *J* = 6Hz), 3.42-4.04 (m, 6H), 7.71-7.86 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 22.16 (1C), 22.24 (1C), 41.57 (1C), 69.26 (1C), 69.92 (1C), 72.60 (1C), 123.58 (2C), 132.30 (2C), 134.24 (2C), 168.94 (2C); HRMS (ESI) calcd. for $C_{14}H_{16}NO_4$ $[M\text{-}H]^-$: 262.1085, found 262.1080; HPLC (Diacel IC column, Hexane:IPA = 82:18, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 17.8 min, t_2 = 19.6 min; $[\alpha]_D^{26}$ = +21.16° (c = 1.01, $CHCl_3$).



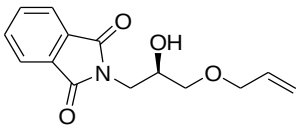
Result Table (Uncal - C:\|Clarity\|WORK2\|DATA\|RJC\|14_03_2017 [racemic] PhthNH OH Oi-Pr 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	17.540	4081.560	123.656	49.8	54.0	0.50	
2	19.548	4112.538	105.529	50.2	46.0	0.59	
	Total	8194.098	229.186	100.0	100.0		



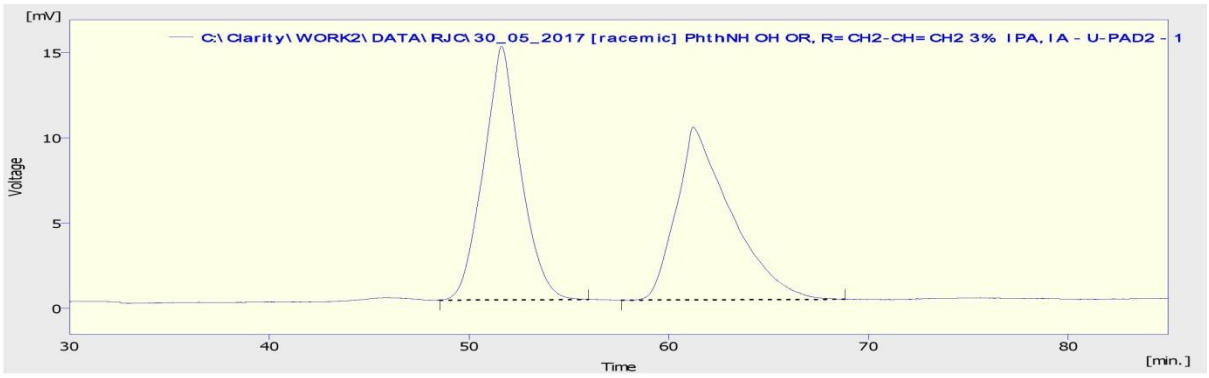
Result Table (Uncal - C:\|Clarity\|WORK2\|DATA\|RJC\|14_03_2017 [ATH 3% Cat in CHCl3] PhthNH OH OiPr 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	17.780	197.481	6.884	10.6	12.7	0.45	
2	19.644	1663.910	47.131	89.4	87.3	0.54	
	Total	1861.390	54.016	100.0	100.0		



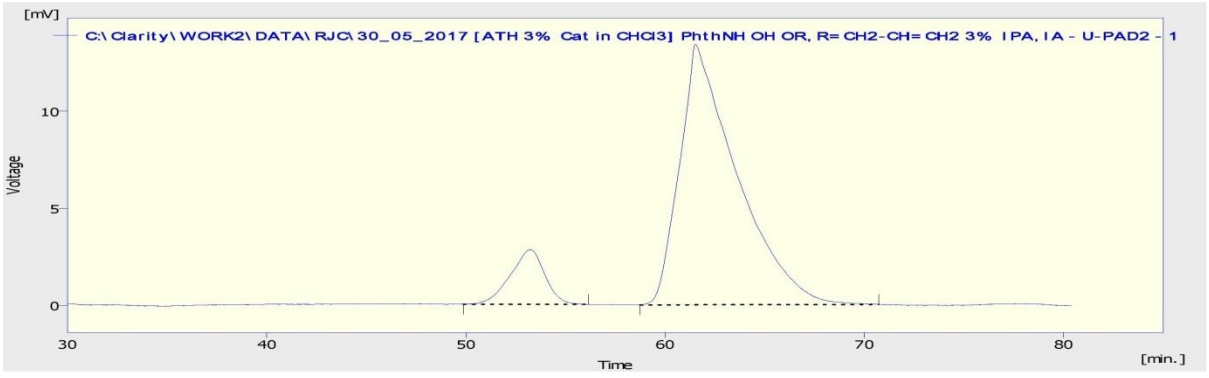
(*R*)-**3l**

Prepared in accordance to **General Procedure F** except that [**2l**]=0.5M, v/v TEAF:$CHCl_3$=1:3 (pale yellow oil, 33.1 mg, 84%, *ee* = 78%): ^{1}H ($CDCl_3$, 500 MHz): δ 2.75 (d, 1H, *J* = 6Hz), 3.46-4.08 (m, 7H), 5.16 (dd, 1H, *J* = 1, 11Hz), 5.25 (dd, 1H, *J* = 1, 17Hz), 5.83-5.90 (m, 1H), 7.69-7.85 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 41.47 (1C), 69.17 (1C), 72.00 (1C), 72.62 (1C), 117.60 (1C), 123.59 (2C), 132.22 (2C), 134.26 (2C), 134.50 (1C), 168.90 (2C); HRMS (ESI) calcd. for $C_{14}H_{15}NO_4Na$ $[M+Na]^+$: 262.1074, found 262.1075; HPLC (Diacel IA column, Hexane:IPA = 97:3, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 53.2 min, t_2 = 61.5 min; $[\alpha]_D^{31}$ = +13.44° (c = 1.02, $CHCl_3$).



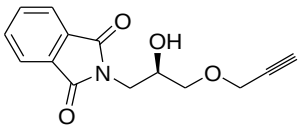
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\30_05_2017 [racemic] PhthNH OH OR, R=CH2-CH=CH2 3% IPA, IA - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	51.628	1912.296	14.875	49.7	59.5	1.94	
2	61.224	1931.904	10.122	50.3	40.5	2.86	
	Total	3844.200	24.997	100.0	100.0		



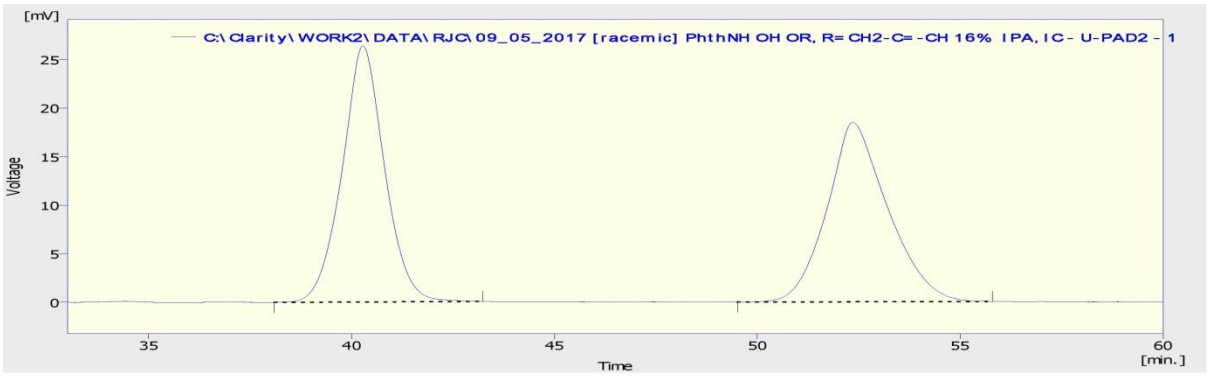
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\30_05_2017 [ATH 3% Cat in CHCl3] PhthNH OH OR, R=CH2-CH=CH2 3% IPA, IA - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	53.216	339.078	2.805	11.2	17.3	1.86	
2	61.520	2677.518	13.407	88.8	82.7	2.96	
	Total	3016.595	16.211	100.0	100.0		



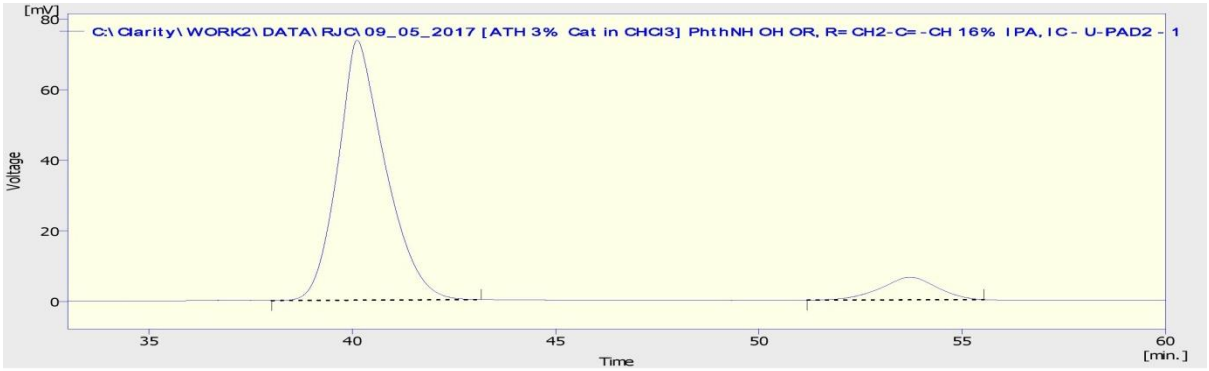
(*R*)-**3m**

Prepared in accordance to **General Procedure F** except that [**2m**]=0.5M, v/v TEAF:$CHCl_3$=1:3 (white solid, 34.7 mg, 89%, *ee* = 81%): ^1H ($CDCl_3$, 400 MHz): δ 2.43 (t, 1H, *J* = 2Hz), 2.74 (d, 1H, *J* = 6Hz), 3.57-4.12 (m, 5H), 4.21 (d, 1H, *J* = 2Hz), 7.72-7.88 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 41.45 (1C), 58.94 (1C), 69.20 (1C), 71.74 (1C), 75.23 (1C), 79.42 (1C), 123.68 (2C), 132.25 (2C), 134.34 (2C), 168.97 (2C); Melting range: 120-123 °C; HRMS (ESI) calcd. for $C_{14}H_{13}NO_4Na$ $[M+Na]^+$: 282.0737, found 282.0737; HPLC (Diacel IC column, Hexane:IPA = 84:16, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 40.1 min, t_2 = 53.7 min; $[\alpha]_D^{30}$ = +19.97° (c = 0.99, $CHCl_3$).



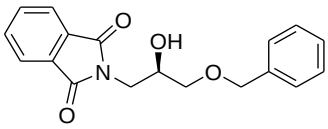
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\09_05_2017 [racemic] PhthNH OH OR, R=CH2-C=-CH 16% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	40.272	1919.255	26.368	49.9	58.8	1.09	
2	52.348	1925.475	18.457	50.1	41.2	1.56	
	Total	3844.730	44.825	100.0	100.0		



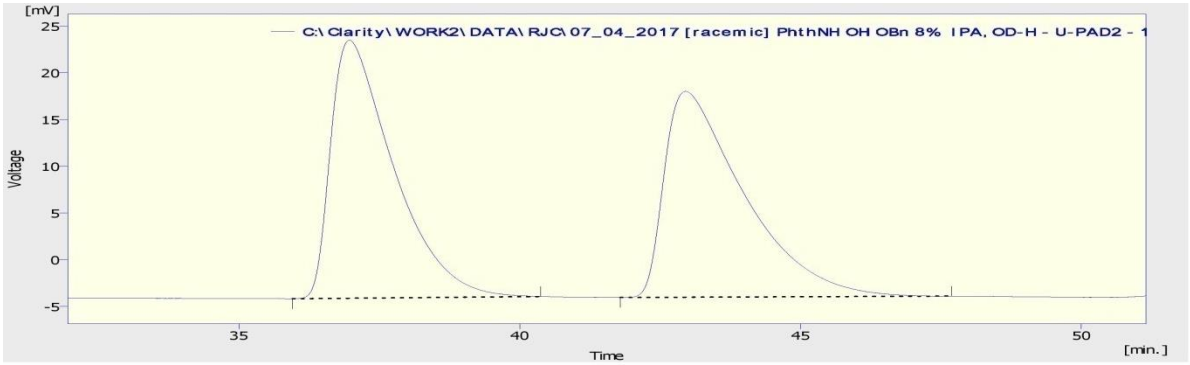
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\09_05_2017 [ATH 3% Cat in CHCl3] PhthNH OH OR, R=CH2-C=-CH 16% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	40.116	5984.035	73.649	90.7	92.0	1.22	
2	53.728	615.811	6.432	9.3	8.0	1.46	
	Total	6599.846	80.081	100.0	100.0		



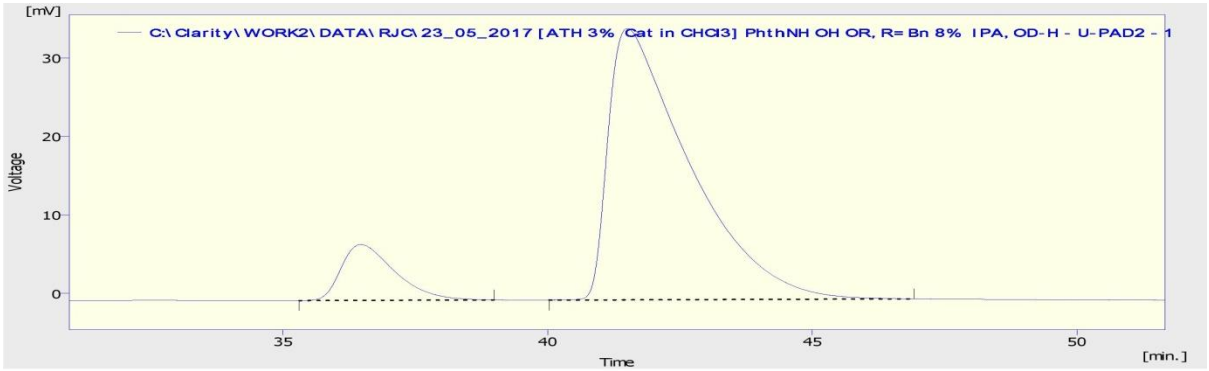
(*R*)-**3n**

Prepared in accordance to **General Procedure F** except that [**2n**]=0.5M, v/v TEAF:$CHCl_3$=1:3 (viscous colourless oil, 32.8 mg, 99%, *ee* = 76%): ^1H ($CDCl_3$, 500 MHz): δ 2.69 (br s, 1H), 3.51-4.11 (m, 5H), 4.56 (s, 2H), 7.27-7.7.35 (m, 5H, coincide with $CDCl_3$ signal), 7.71-7.86 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 41.52 (1C), 69.27 (1C), 72.06 (1C), 73.80 (1C), 123.63 (2C), 128.02 (2C), 128.05 (1C), 128.69 (2C), 132.25 (2C), 134.29 (2C), 137.94 (1C), 168.96 (2C); HRMS (ESI) calcd. for $C_{18}H_{17}NO_4Na$ $[M+Na]^+$: 334.1050, found 334.1053; HPLC (Diacel OD-H column, Hexane:IPA = 92:8, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 36.5 min, t_2 = 41.5 min; $[\alpha]_D^{27}$ = +14.24° (c = 1.03, $CHCl_3$).



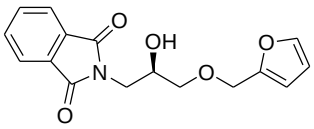
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\07_04_2017 [racemic] PhthNH OH OBn 8% IPA, OD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	36.968	2144.013	27.600	49.7	55.6	1.18	
2	42.956	2167.726	22.024	50.3	44.4	1.48	
	Total	4311.739	49.624	100.0	100.0		



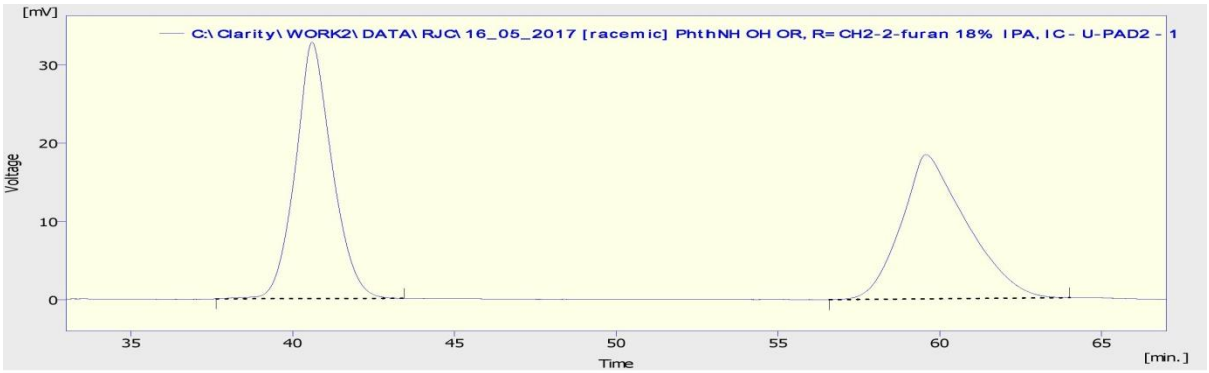
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\23_05_2017 [ATH 3% Cat in CHCl3] PhthNH OH OR, R=Bn 8% IPA, OD-H - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	36.468	499.787	7.122	12.0	17.1	1.08	
2	41.504	3674.849	34.584	88.0	82.9	1.60	
	Total	4174.637	41.706	100.0	100.0		



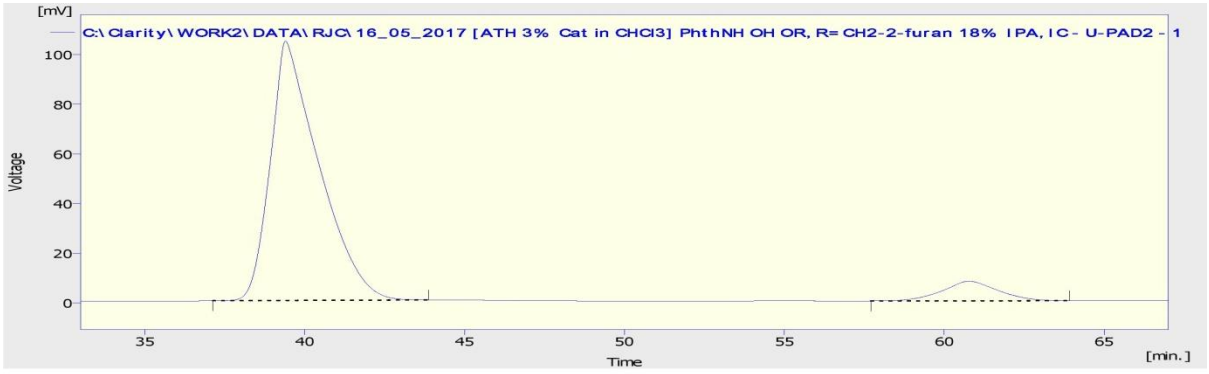
(*R*)-**3o**

Prepared in accordance to **General Procedure F** except that [**2o**]=0.5M, v/v TEAF:$CHCl_3$=1:3 (pale yellow oil, 25.4 mg, 86%, *ee* = 84%): ^1H ($CDCl_3$, 500 MHz): δ 2.69 (d, 1H, *J* = 6Hz), 3.49-3.59 (m, 2H), 3.76-3.91 (m, 2H), 4.05-4.08 (m, 1H), 4.50 (s, 2H), 6.31-6.32 (m, 2H), 7.37 (s, 1H), 7.71-7.85 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 41.42 (1C), 65.45 (1C), 69.17 (1C), 71.83 (1C), 109.86 (1C), 110.52 (1C), 123.61 (2C), 132.24 (2C), 134.26 (2C), 143.16 (1C), 151.16 (1C), 168.91 (2C); HRMS (ESI) calcd. for $C_{16}H_{15}NO_5Na$ $[M+Na]^+$: 324.0842, found 324.0838; HPLC (Diacel IC column, Hexane:IPA = 82:18, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 39.4 min, t_2 = 60.8 min; $[\alpha]_D^{27}$ = +16.09° (c = 1.02, $CHCl_3$).



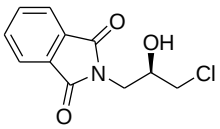
Result Table (Uncal - C:\|Clarity\|WORK2\|DATA\|RJC\|16_05_2017 [racemic] PhthNH OH OR, R=CH2-2-furan 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	40.600	2593.494	32.758	50.0	64.0	1.18	
2	59.572	2589.301	18.445	50.0	36.0	2.14	
	Total	5182.794	51.203	100.0	100.0		



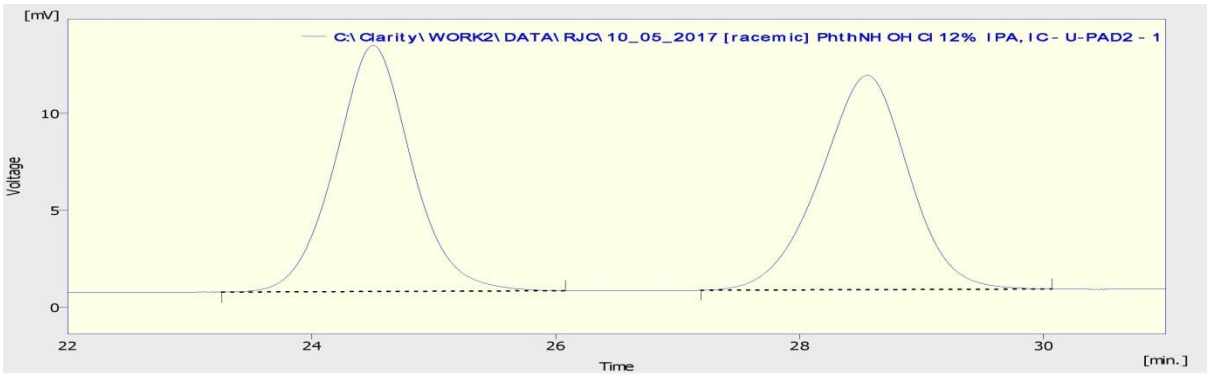
Result Table (Uncal - C:\|Clarity\|WORK2\|DATA\|RJC\|16_05_2017 [ATH 3% Cat in CHCl3] PhthNH OH OR, R=CH2-2-furan 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	39.400	11050.228	104.490	92.1	93.0	1.61	
2	60.768	952.291	7.903	7.9	7.0	1.82	
	Total	12002.519	112.394	100.0	100.0		



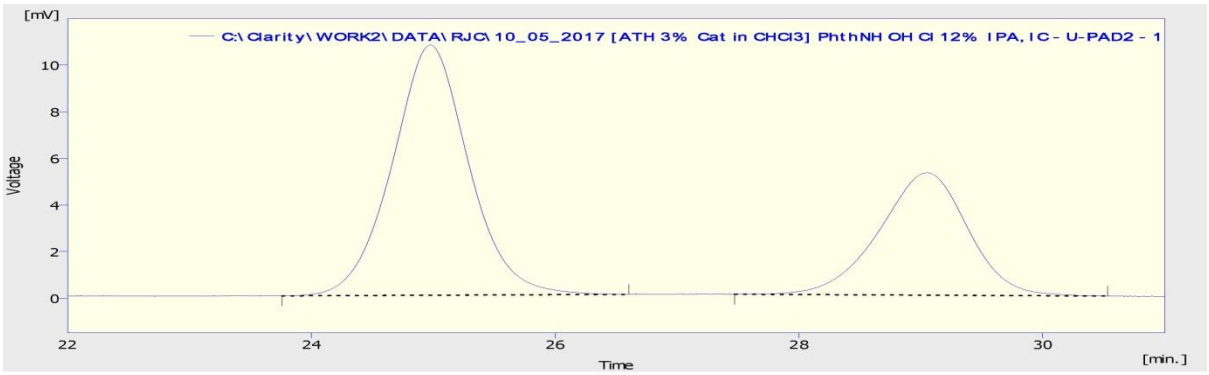
(*R*)-**6**

Prepared in accordance to **General Procedure F** except that [**5**]=0.5M, v/v TEAF:$CHCl_3$=1:3 (white solid, 26.9 mg, 92%, *ee* = 28%): ^1H ($CDCl_3$, 400 MHz): δ 2.81 (d, 1H, J = 8Hz), 3.60-3.70 (m, 2H), 3.87-4.00 (m, 2H), 4.15-4.20 (m, 1H), 7.74-7.89 (m, 4H); ^{13}C (jmod) ($CDCl_3$, 126 MHz): δ 41.83 (1C), 47.53 (1C), 70.03 (1C), 123.83 (2C), 132.09 (2C), 134.54 (2C), 168.92 (2C); HRMS (ESI) calcd. for $C_{11}H_{10}NO_3ClNa$ $[M+Na]^+$: 262.0241, 263.0273, found 262.0242, 263.0278; Melting range = 105-106 °C; HPLC (Diacel IC column, Hexane:IPA = 88:12, detection wavelength: λ = 254 nm, flow rate = 1 mL/min): t_1 = 25.0 min, t_2 = 29.1 min; $[\alpha]_D^{30}$ = +6.12° (c = 1.00, $CHCl_3$).



Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\10_05_2017 [racemic] PhthNH OH Cl 12% IPA, IC - U-PAD2 - 1)

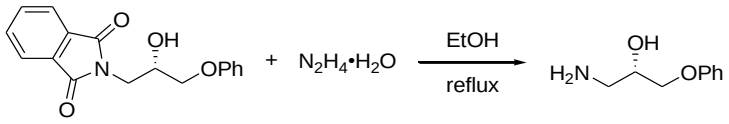
	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	24.500	556.384	12.698	49.8	53.4	0.66	
2	28.556	560.862	11.062	50.2	46.6	0.78	
	Total	1117.246	23.760	100.0	100.0		



Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\10_05_2017 [ATH 3% Cat in CHCl3] PhthNH OH Cl 12% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	24.976	468.414	10.732	63.8	67.1	0.66	
2	29.052	266.006	5.257	36.2	32.9	0.78	
	Total	734.420	15.989	100.0	100.0		

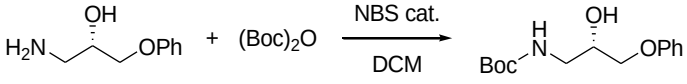
(9) Deprotection of phthalimyl alcohol (*S*)-3a



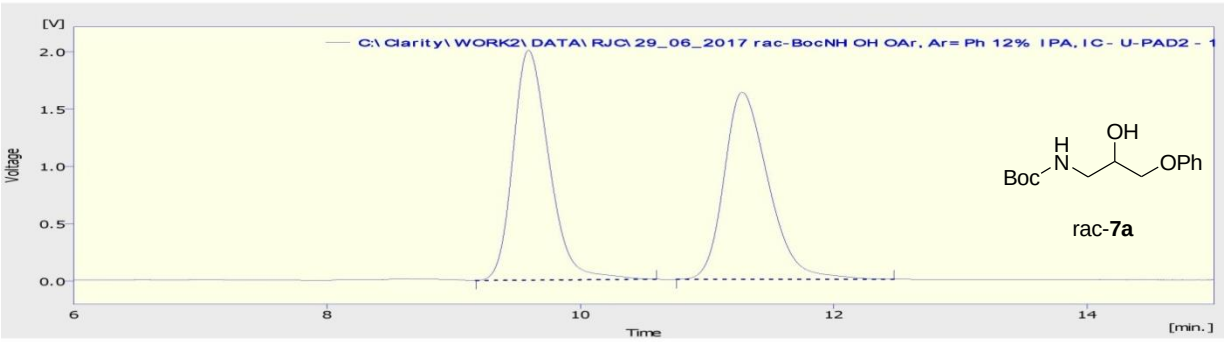
(*S*)-**3a** (*S*)-**4a**

Phthalimyl alcohol (*S*)-**3a** (294 mg, 0.99 mmol, 1 equiv.), hydrazine hydrate (0.29 mL, 5.94 mmol, 6 equiv.) was added to ethanol (40 mL) and the solution refluxed for 2 hours. Consequently, the setup was cooled in ice water and white solid formed were filtered off by Celite and the cake washed with excess ethyl acetate. The filtrate was subject to solvent strip under reduced pressure and the residue purified by Kugelrohr distillation to afford (*S*)-**4a** (white solid, 142 mg, 86%). Characterization data is consistent with reported literature: *Bioorg. Med. Chem. 2012, 20*, 5787.

As it was difficult to separate **4a** on HPLC, a *tert*-butyloxycarbonyl (Boc) group was introduced to **4a** before attempting to resolve the enantiomers. Procedure for the preparation of **7a** is adopted from *Tetrahedron Lett.* **2016**, *57*, 4807. HPLC analysis (Diacel IC column, Hexane:IPA = 82:18, detection wavelength: λ = 254 nm, t_1 = 9.6 min, t_2 = 11.3 min, flow rate = 1 mL/min) revealed that deprotection of (*S*)-**3a** did not affect the optical purity of the derived amino alcohol (*S*)-**4a**.

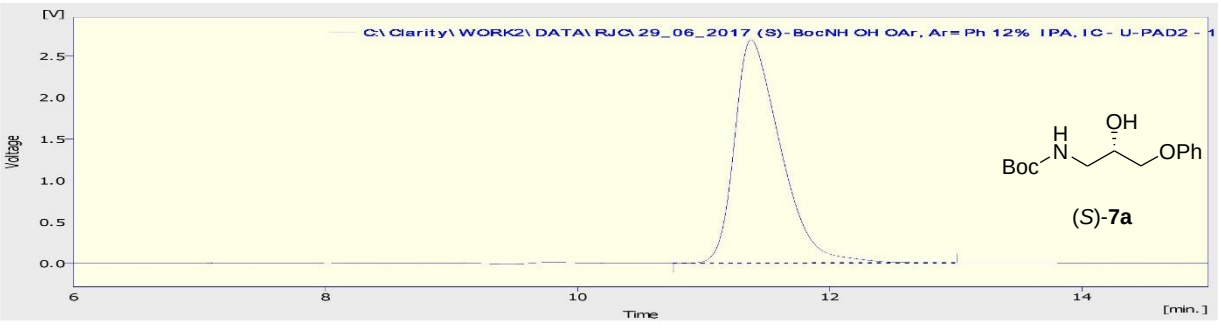


(*S*)-**4a** (*S*)-**7a**



Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\29_06_2017 rac-BocNH OH OAr, Ar=Ph 12% IPA, IC - U-PAD2 - 1)

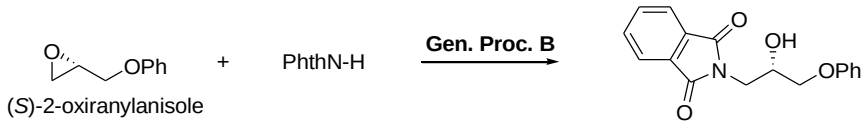
	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	9.588	1325.819	66.914	50.1	55.1	0.30	
2	11.276	1321.930	54.484	49.9	44.9	0.37	
	Total	2647.749	121.398	100.0	100.0		



Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\29_06_2017 (S)-BocNH OH OAr, Ar=Ph 12% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	11.376	2275.251	89.684	100.0	100.0	0.39	
	Total	2275.251	89.684	100.0	100.0		

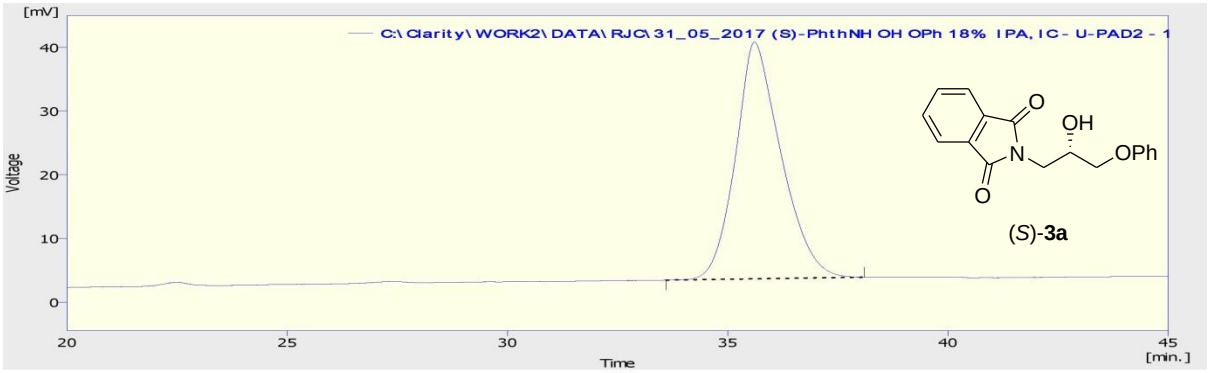
(10) Determination of absolute configuration (for selected alcohols)



(*S*)-2-oxiranylanisole

(*S*)-**3a**

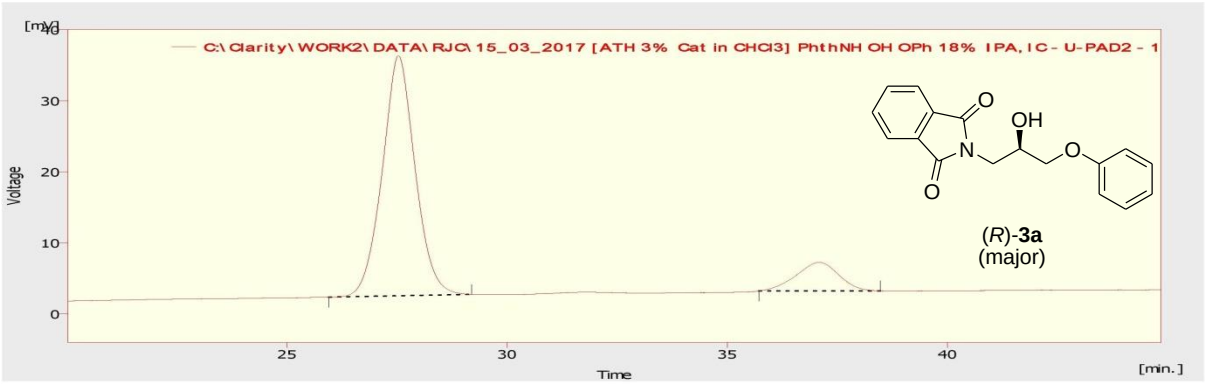
Enantiopure (*S*)-**3a** was prepared according to **General Procedure B** from commercially available (*S*)-2-oxiranylanisole before analysis by HPLC (Diacel IC column, Hexane:IPA = 82:18, detection wavelength: λ = 254 nm, flow rate = 1 mL/min)



Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\31_05_2017 (S)-PhthNH OH OPh 18% IPA, IC - U-PAD2 - 1)

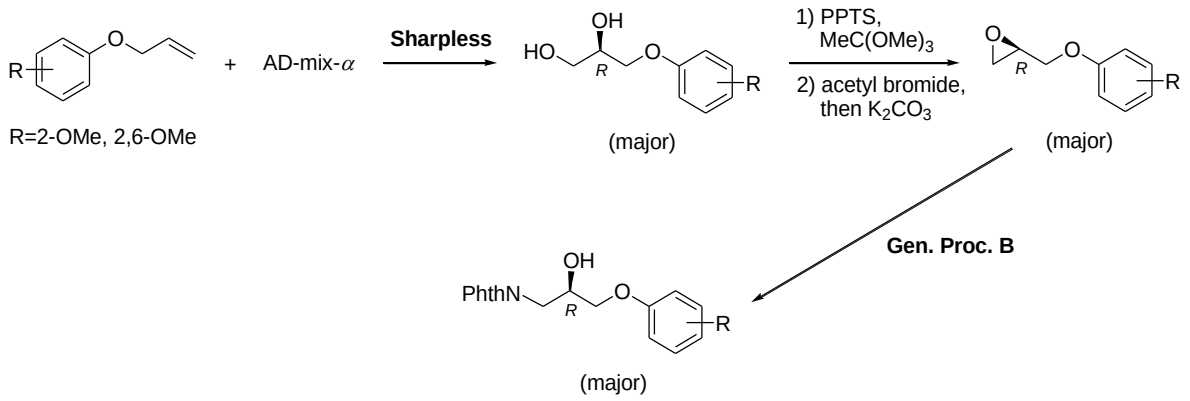
	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	35.612	2675.080	37.187	100.0	100.0	1.08	
	Total	2675.080	37.187	100.0	100.0		

From the ATH reaction, confirms major enantiomer possess an *R* configuration.



Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\15_03_2017 [ATH 3% Cat in CHCl3] PhthNH OH OPh 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	27.532	1755.485	33.803	86.6	89.3	0.78	
2	37.084	271.368	4.031	13.4	10.7	1.06	
	Total	2026.853	37.834	100.0	100.0		



R=2-OMe, 2,6-OMe

(major)

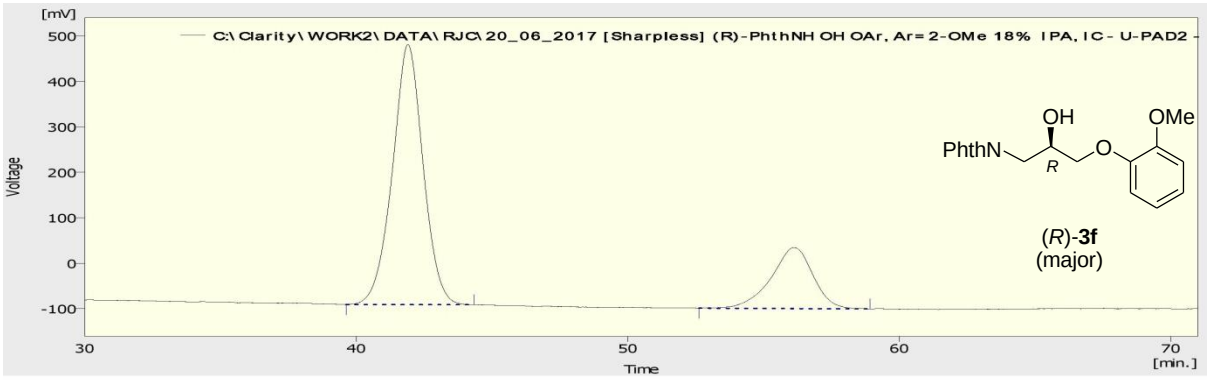
(major)

(major)

Procedure for the Sharpless dihydroxylation of the aromatic allyl ethers is adopted and modified from *Tetrahedron Lett.* **1993**, *34*, 2267, and the subsequent ring closing to give the optically active epoxides follows *Tetrahedron* **1992**, *48*, 10515.

• For alcohol 3f (R=2-OMe)

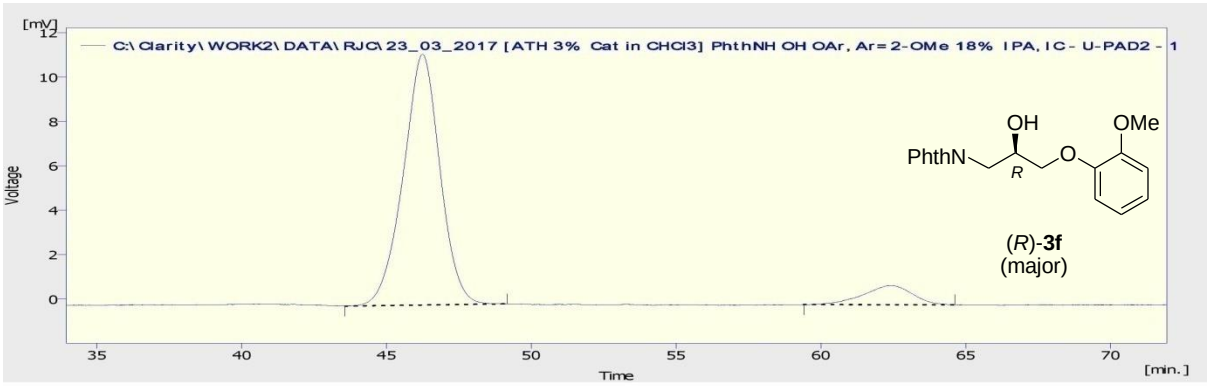
- Derived from the Sharpless pathway



Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\20_06_2017 [Sharpless] (R)-PhthNH OH OAr, Ar=2-OMe 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	41.908	1502.193	19.092	75.7	81.0	1.19	
2	56.100	483.346	4.485	24.3	19.0	1.60	
	Total	1985.539	23.578	100.0	100.0		

- Derived from the ATH reaction, confirms major enantiomer possess an *R* configuration.

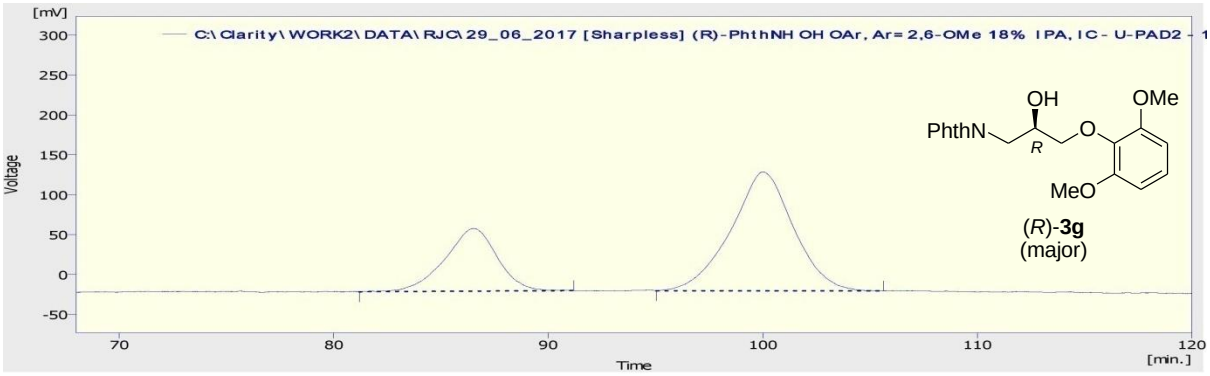


Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\23_03_2017 [ATH 3% Cat in CHCl3] PhthNH OH OAr, Ar=2-OMe 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	46.240	999.882	11.300	90.8	92.9	1.34	
2	62.384	101.544	0.869	9.2	7.1	1.81	
	Total	1101.425	12.169	100.0	100.0		

• **For alcohol 3g (R=2,6-OMe)**

- Derived from the Sharpless pathway



Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\29_06_2017 [Sharpless] (R)-PhthNH OH OAr, Ar=2,6-OMe 18% IPA, IC - U-PAD2 - 1)

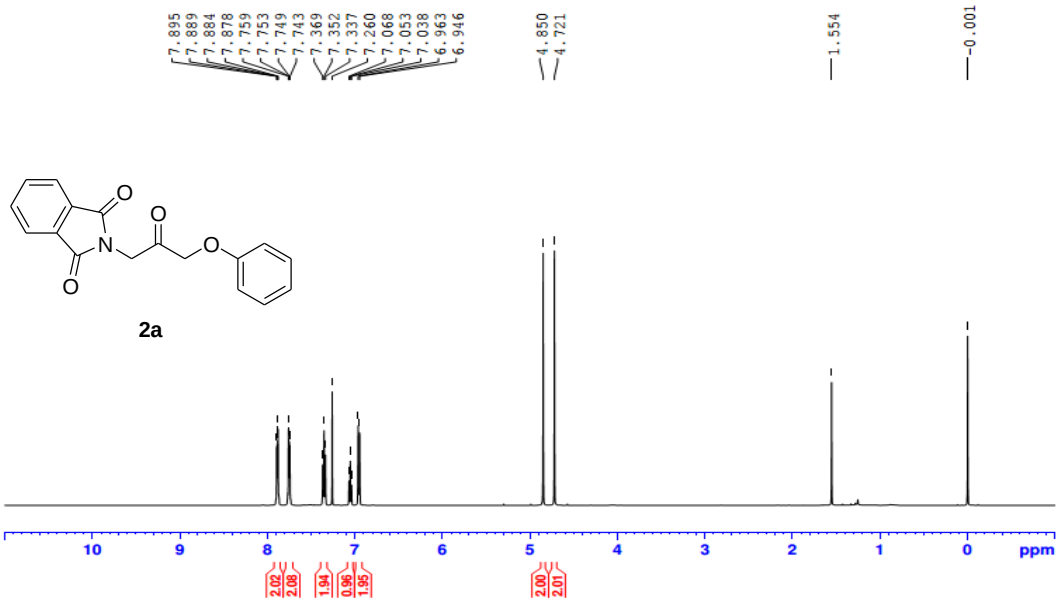
	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	86.480	444.266	2.621	30.6	34.5	2.54	
2	100.012	1008.281	4.973	69.4	65.5	3.03	
	Total	1452.547	7.594	100.0	100.0		

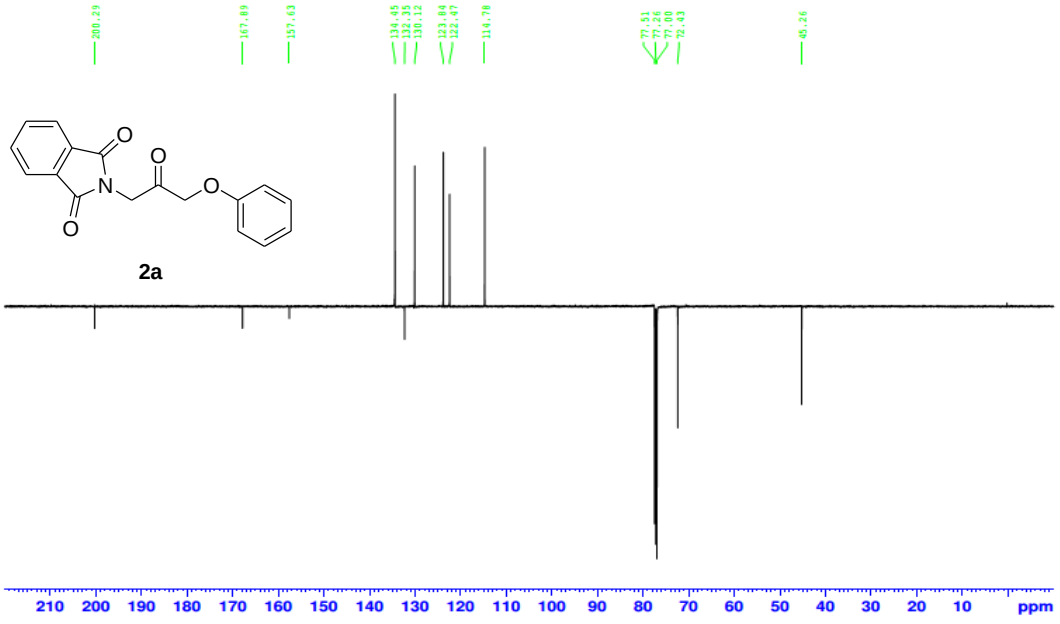
- Derived from the ATH reaction, confirms major enantiomer possess an *R* configuration.

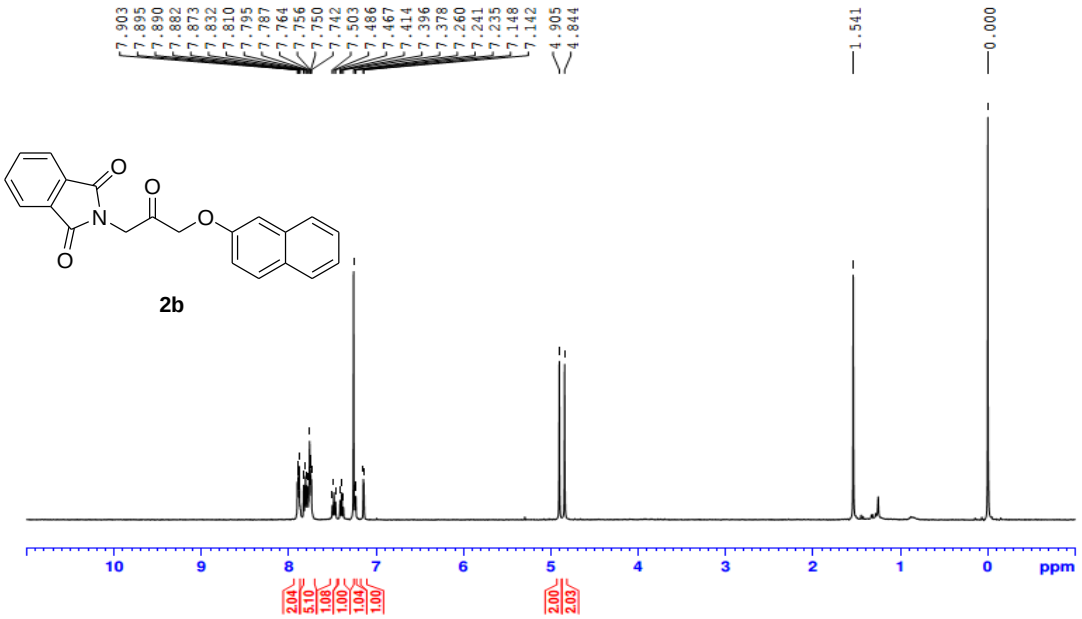
Result Table (Uncal - C:\Clarity\WORK2\DATA\RJC\10_04_2017 [ATH 3% Cat in CHCl3] PhthNH OH OAr, Ar=2,6-OMe 18% IPA, IC - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	83.376	139.912	0.764	5.2	6.1	2.65	
2	95.952	2538.803	11.727	94.8	93.9	3.24	
	Total	2678.715	12.491	100.0	100.0		

III. NMR Spectra



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7.787
7.764
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7.742
7.503
7.486
7.467
7.414
7.396
7.378
7.260
7.241
7.235
7.148
7.142
4.905
4.844
1.541
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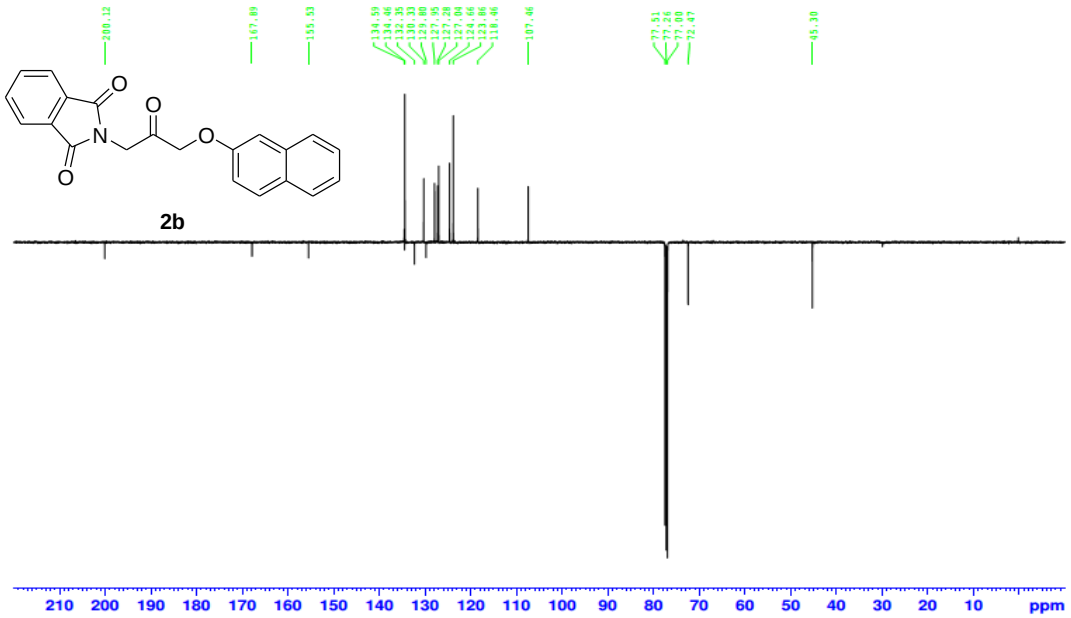
2b
10 9 8 7 6 5 4 3 2 1 0 ppm
2.04 5.10 1.08 1.00 1.04 1.00
2.00 2.03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127.28
127.04
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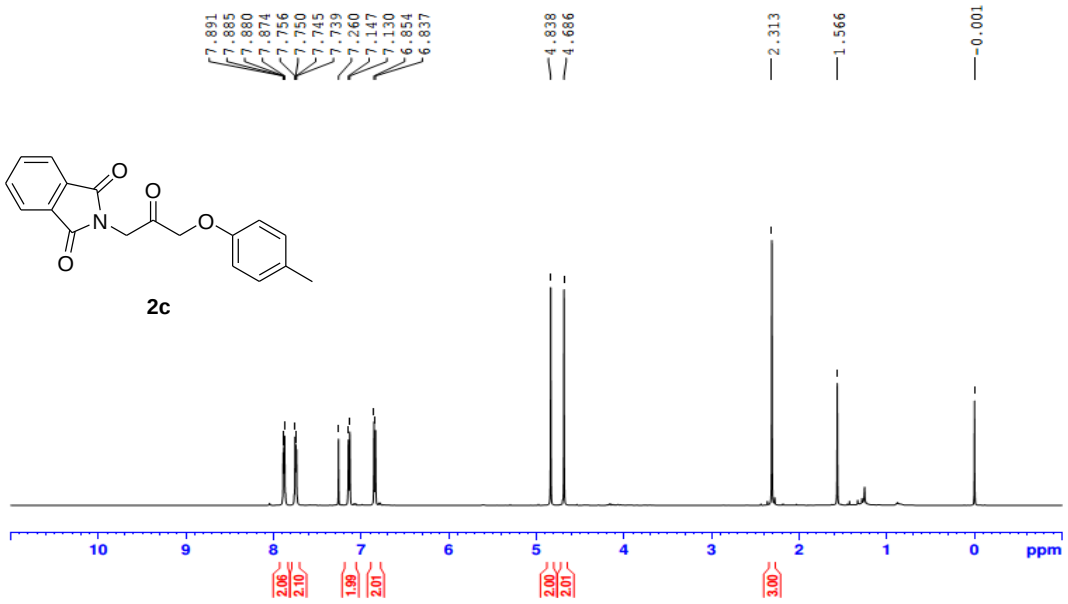
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6.837
4.838
4.686
2.313
1.566
0.001
2c
10
9
8
7
6
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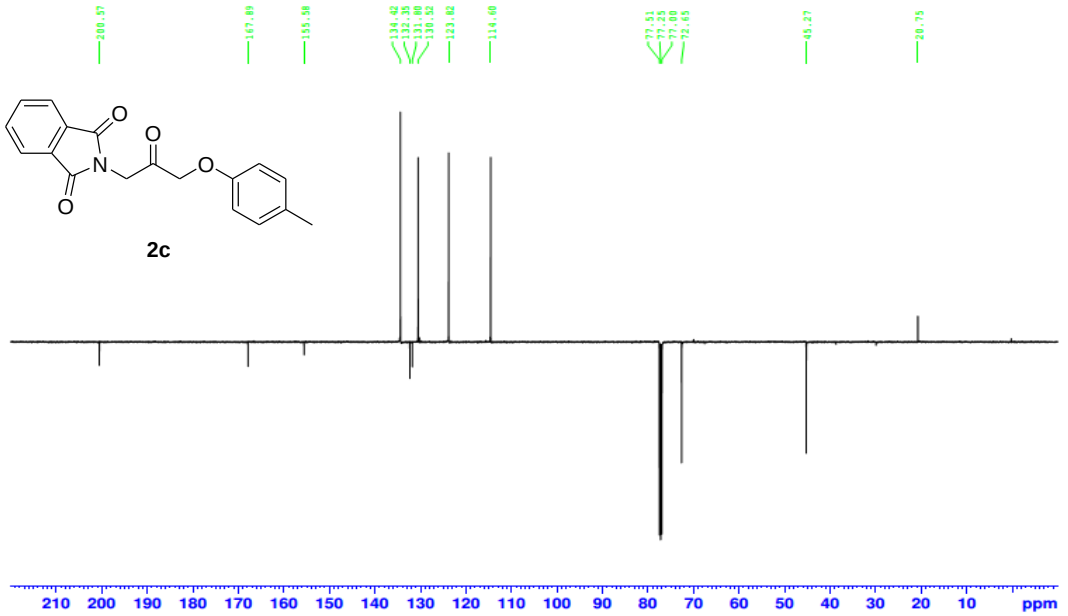
3.00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45.27
20.75
2c
210
200
190
180
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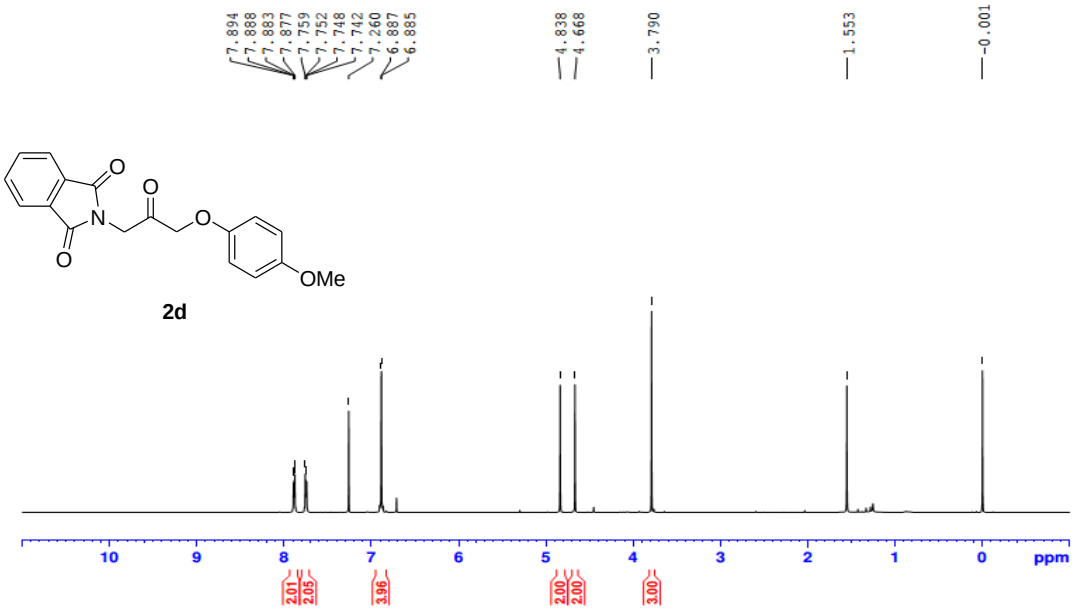
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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3.790
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O
O
N
O
O
O
OMe
2d
10
9
8
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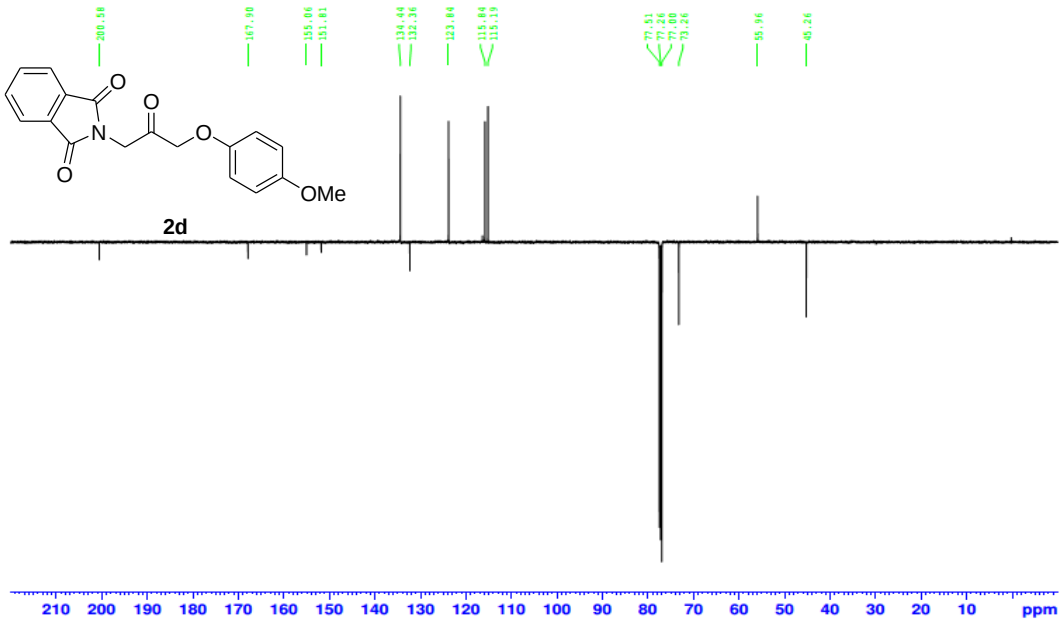
3.00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200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
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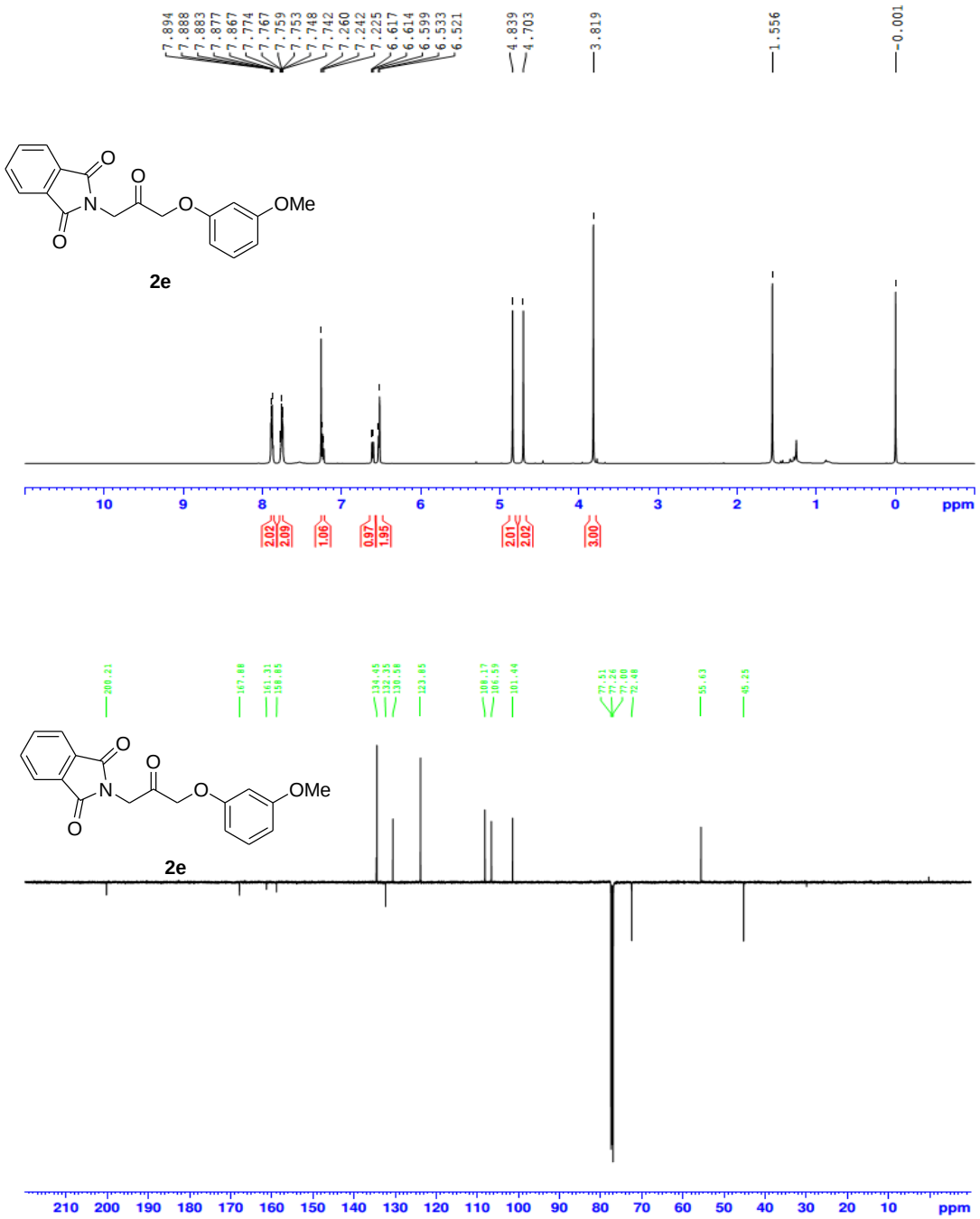
ppm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7.742
7.260
7.242
7.225
6.617
6.614
6.599
6.533
6.521
4.839
4.703
3.819
1.556
0.001
O
O
N
O
O
O
OMe
2e
10
9
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7
6
5
4
3
2
1
0
ppm
2.02
2.09
1.06
0.97
1.95
2.01
2.02
3.00
200.21
167.88
161.31
158.85
134.45
132.35
130.58
123.85
108.17
106.59
101.44
77.51
77.26
77.00
72.48
55.63
45.25
O
O
N
O
O
O
OMe
2e
210
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150
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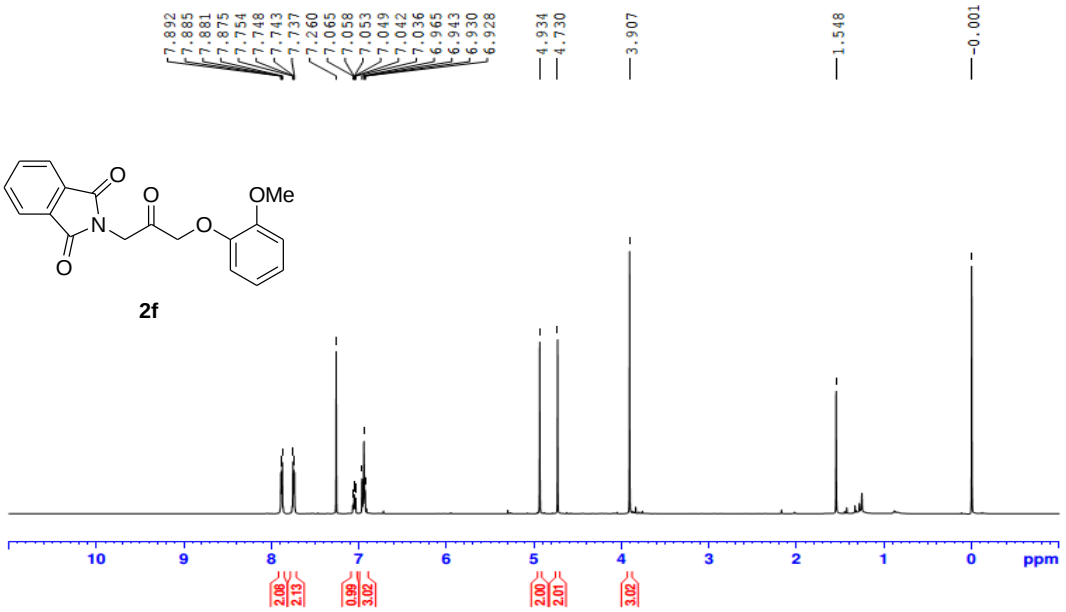
10
ppm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7.737
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7.065
7.058
7.053
7.049
7.042
7.036
6.965
6.943
6.930
6.928
4.934
4.730
3.907
1.548
-0.001
O
O
N
O
O
OMe
2f
10
9
8
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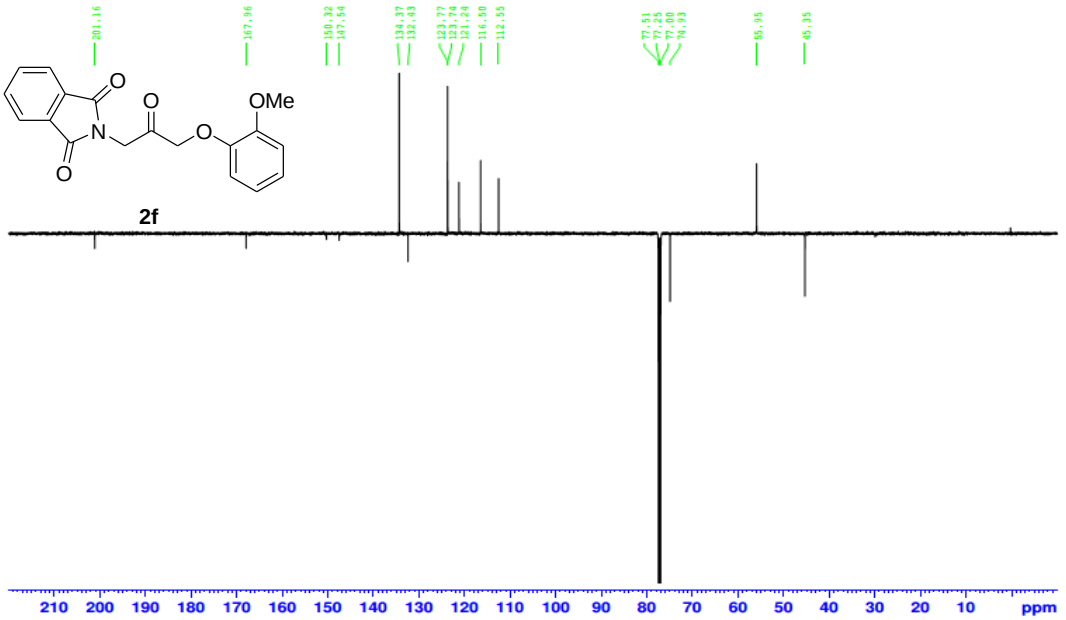
2.01
3.02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112.55
77.51
77.25
77.00
74.93
55.95
65.35
O
O
N
O
O
OMe
2f
210
200
190
180
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150
140
130
120
110
100
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70
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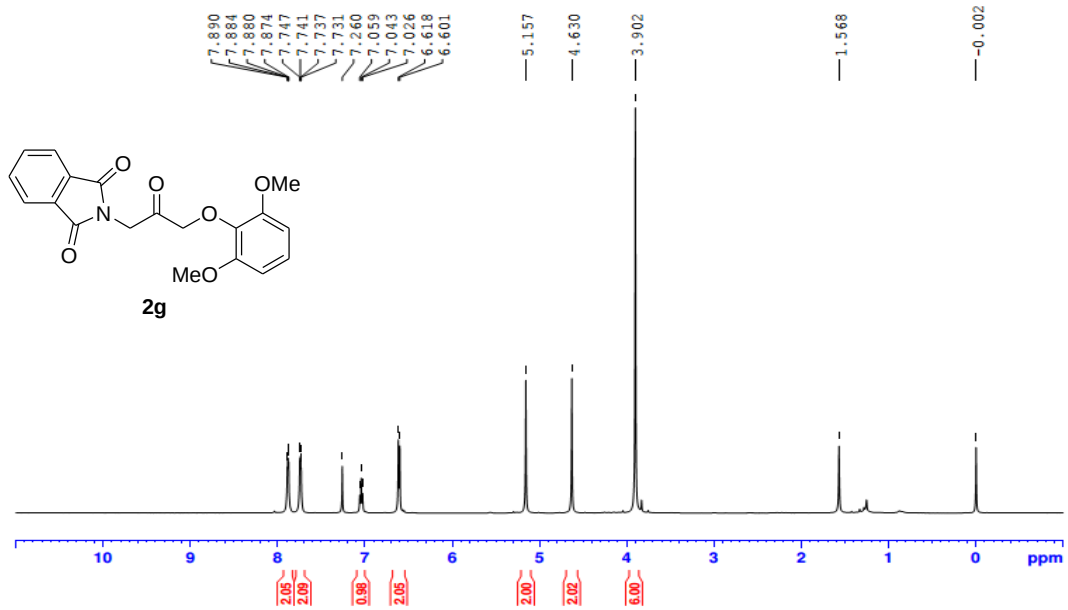
20
10
ppm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7.741
7.737
7.731
7.260
7.059
7.043
7.026
6.618
6.601
5.157
4.630
3.902
1.568
0.002
O
O
N
O
O
O
OMe
MeO
2g
10
9
8
7
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5
4
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2
1
0
ppm
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2.00
0.98
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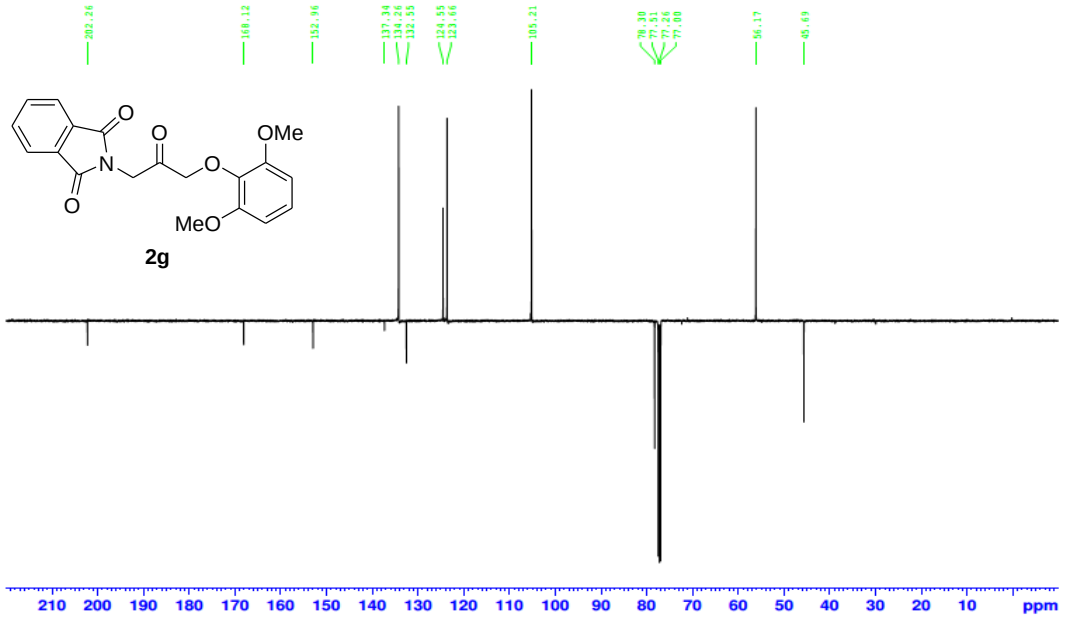
2.00
2.02
6.00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152.96
137.34
134.26
132.55
124.55
123.66
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78.30
77.51
77.26
77.00
56.17
45.69
O
O
N
O
O
O
OMe
MeO
2g
210
200
190
180
170
160
150
140
130
120
110
100
90
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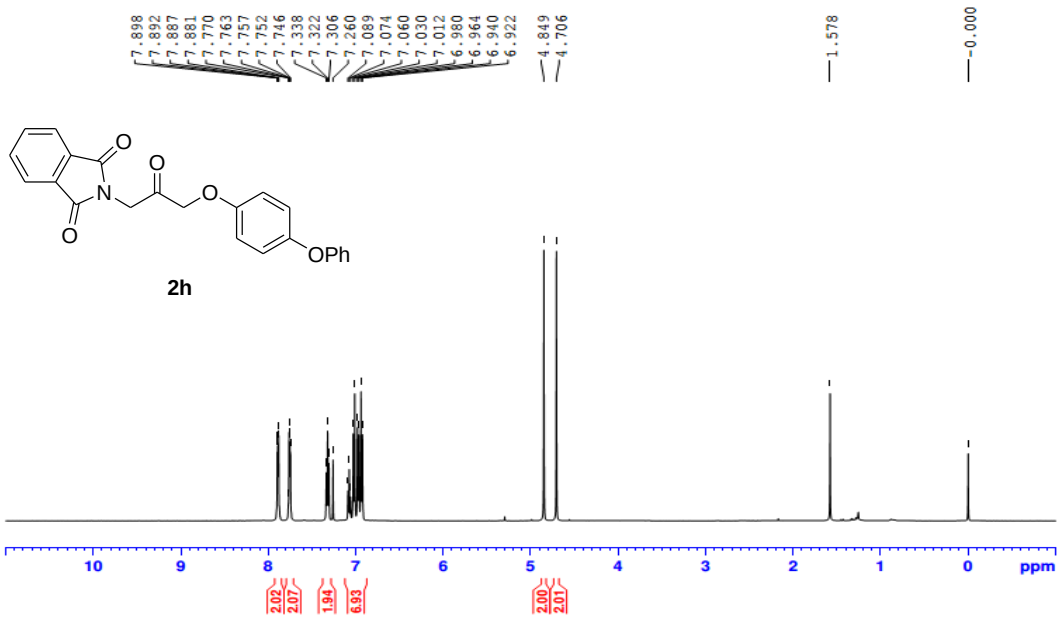
30
20
10
ppm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7.887
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7.763
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7.752
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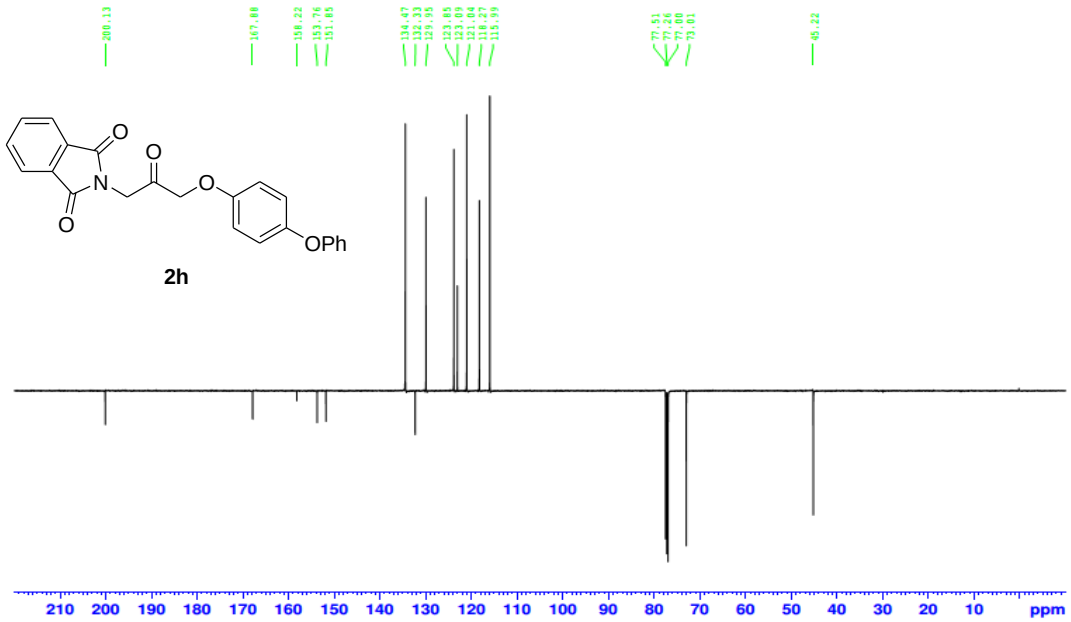
7.338
7.322
7.306
7.260
7.089
7.074
7.060
7.030
7.012
6.980
6.964
6.940
6.922
4.849
4.706
1.578
0.000
O
O
N
O
O
O
OPh
2h
10
9
8
7
6
5
4
3
2
1
0
ppm
2.02
2.07
1.94
6.93
2.00
2.01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158.22
153.76
151.85
134.47
132.33
129.95
123.65
123.09
121.04
116.27
115.99
77.51
77.26
77.00
73.01
45.22
O
O
N
O
O
O
OPh
2h
210
200
190
180
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160
150
140
130
120
110
100
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80
70
60
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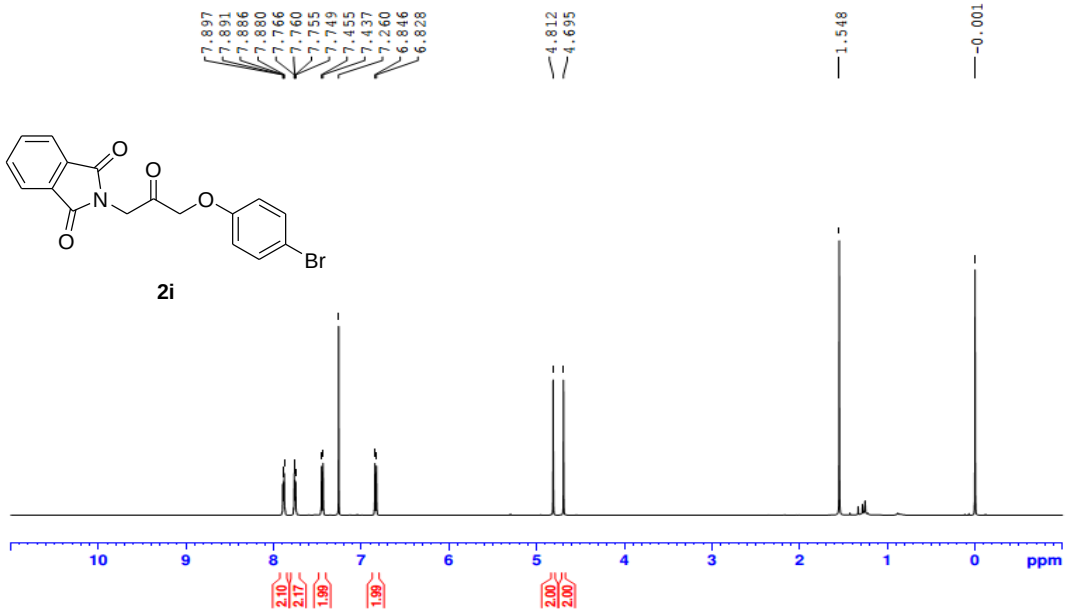
40
30
20
10
ppm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7.886
7.880
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7.760
7.755
7.749
7.455
7.437
7.260
6.846
6.828
4.812
4.695
1.548
0.001
O
O
N
O
O
Br
2i
10
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8
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6
5
4
3
2
1
0
ppm
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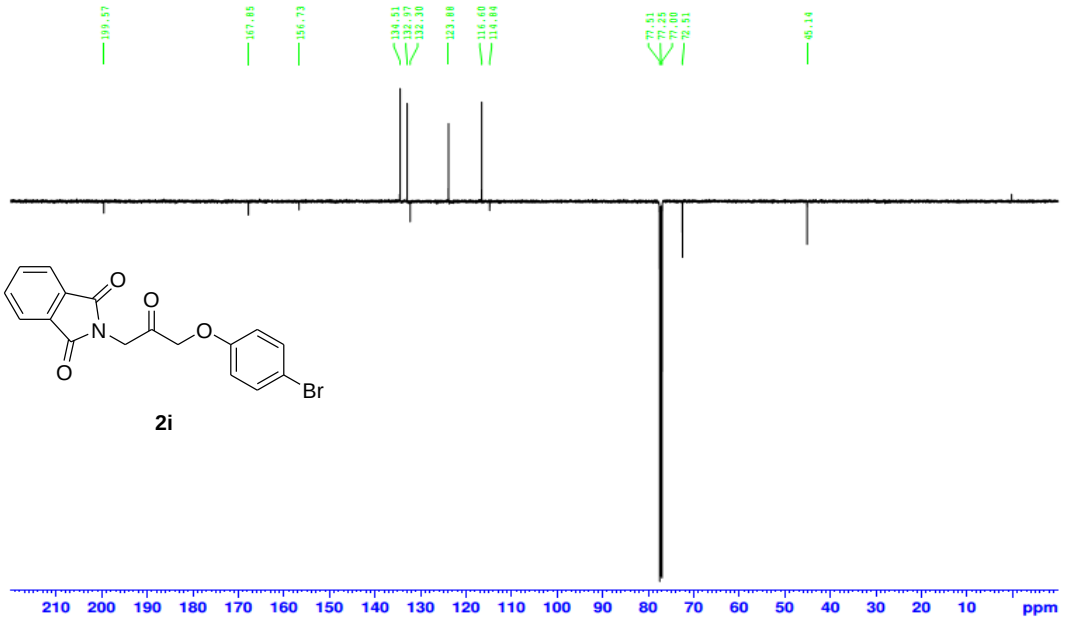
2.17
1.99
1.99
2.00
2.00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156.73
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132.30
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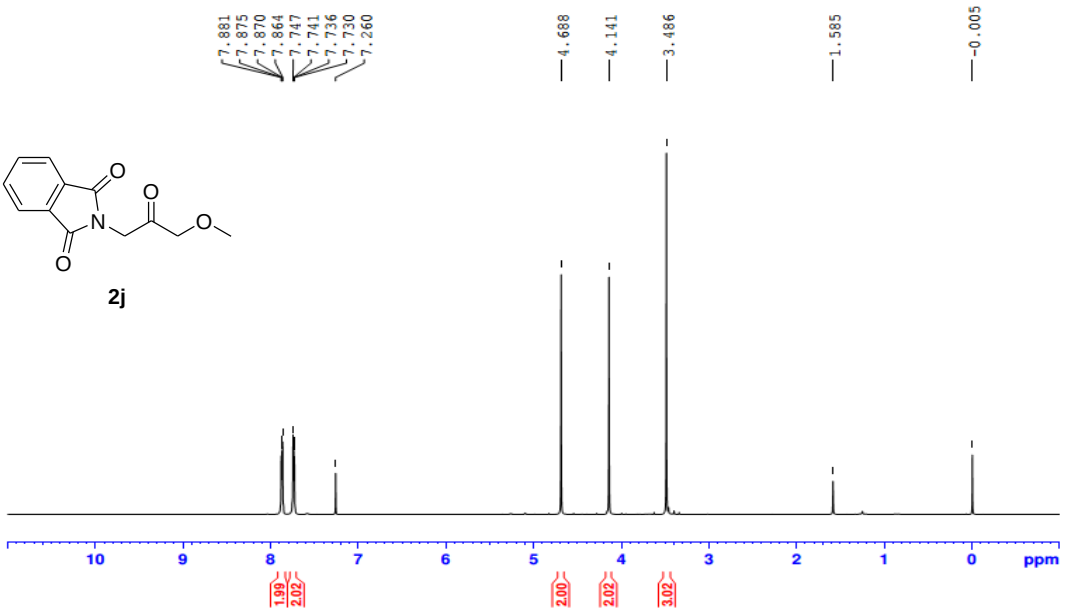
116.60
114.84
77.51
77.25
77.00
72.51
65.14
O
O
N
O
O
Br
2i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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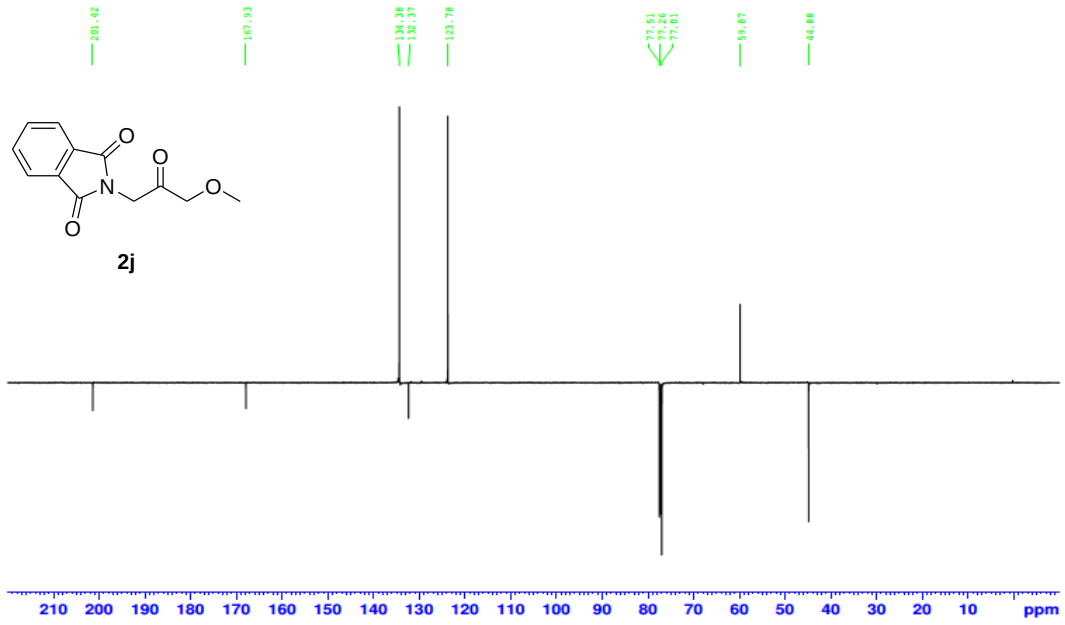
7.730
7.260
4.688
4.141
3.486
1.585
-0.005
O
O
N
O
O
O
2j
10
9
8
7
6
5
4
3
2
1
0
ppm
1.99
2.02
2.00
2.02
3.02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134.38
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77.26
77.01
59.87
44.88
O
O
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O
O
O
2j
210
200
190
180
170
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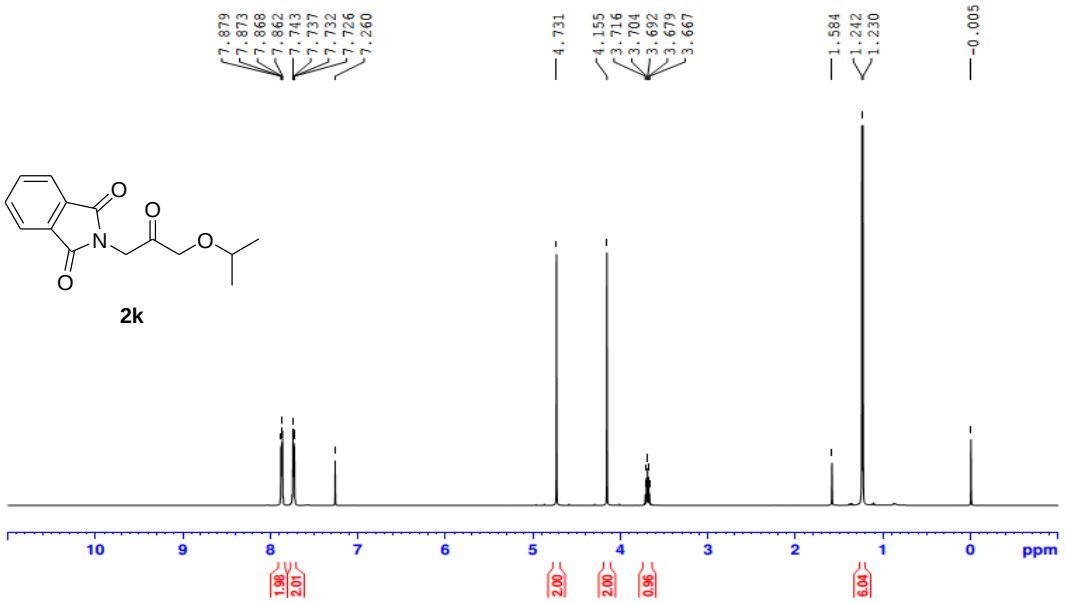
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.726
7.260
4.731
4.155
3.716
3.704
3.692
3.679
3.667
1.584
1.242
1.230
-0.005
2k
10
9
8
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5
4
3
2
1
0
ppm
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2.01
2.00
2.00
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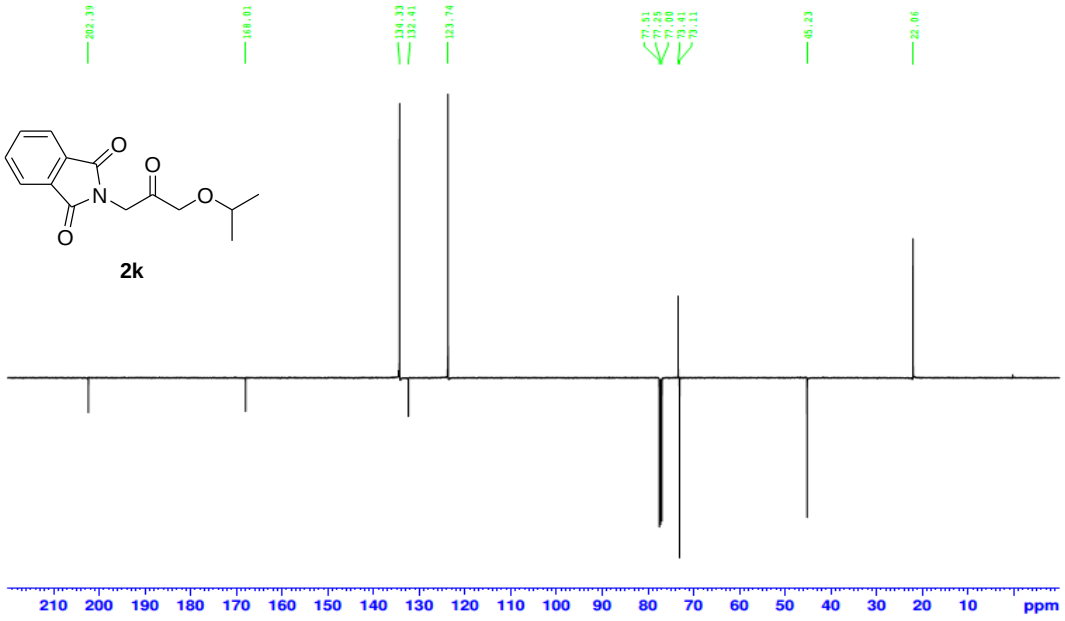
6.04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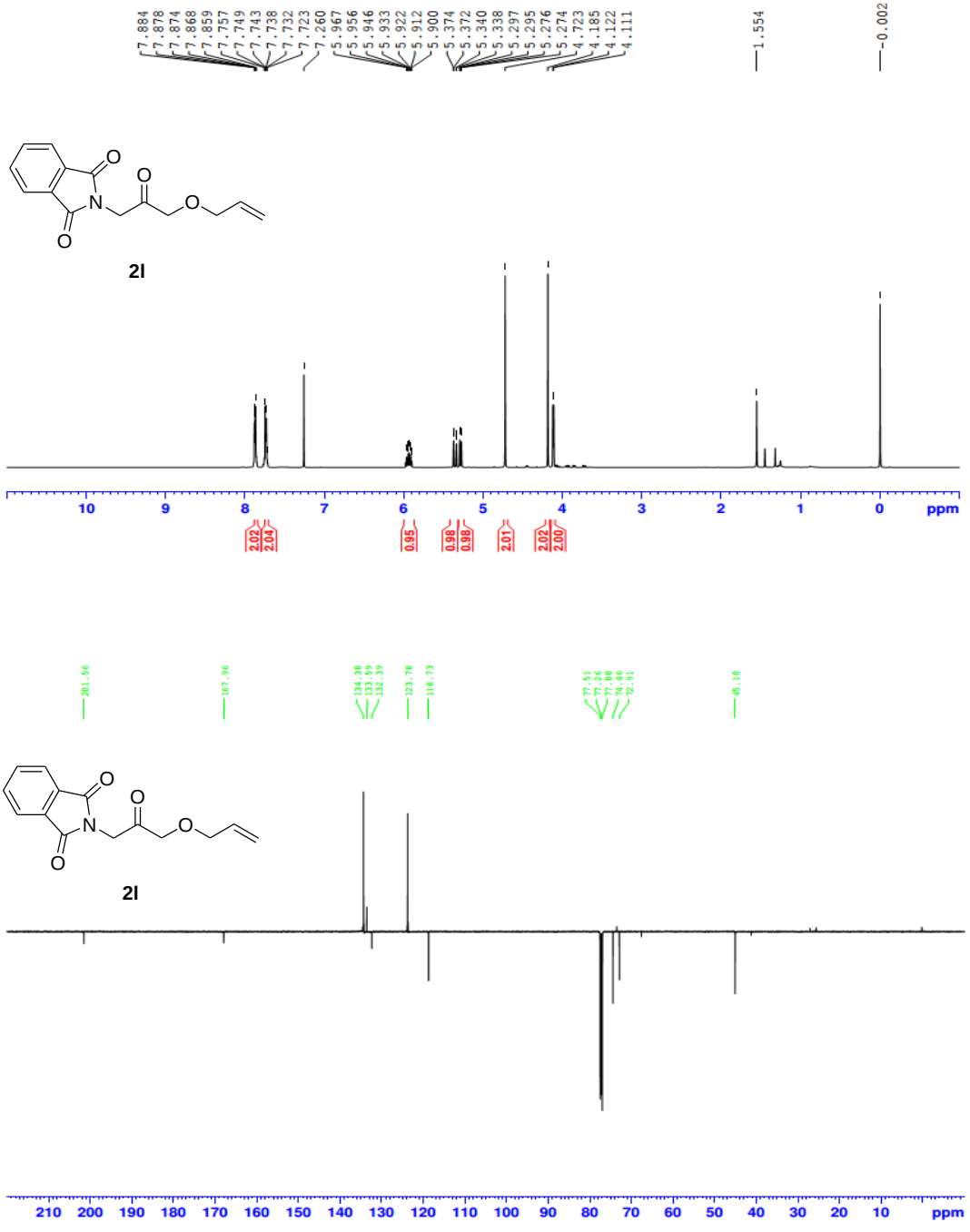
132.41
123.74
77.51
77.25
77.00
73.41
73.11
45.23
22.06
2k
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.749
7.743
7.738
7.732
7.723
7.260
5.967
5.956
5.946
5.933
5.922
5.912
5.900
5.374
5.372
5.340
5.338
5.297
5.295
5.276
5.274
4.723
4.185
4.122
4.111
1.554
0.002
2l
10
9
8
7
6
5
4
3
2
1
0
ppm
2.02
2.04
0.95
0.98
0.98
2.01
2.02
2.00
201.56
167.96
134.38
133.59
132.39
123.78
118.73
77.51
77.26
77.00
76.48
72.91
45.10
2l
210
200
190
180
170
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150
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110
100
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70
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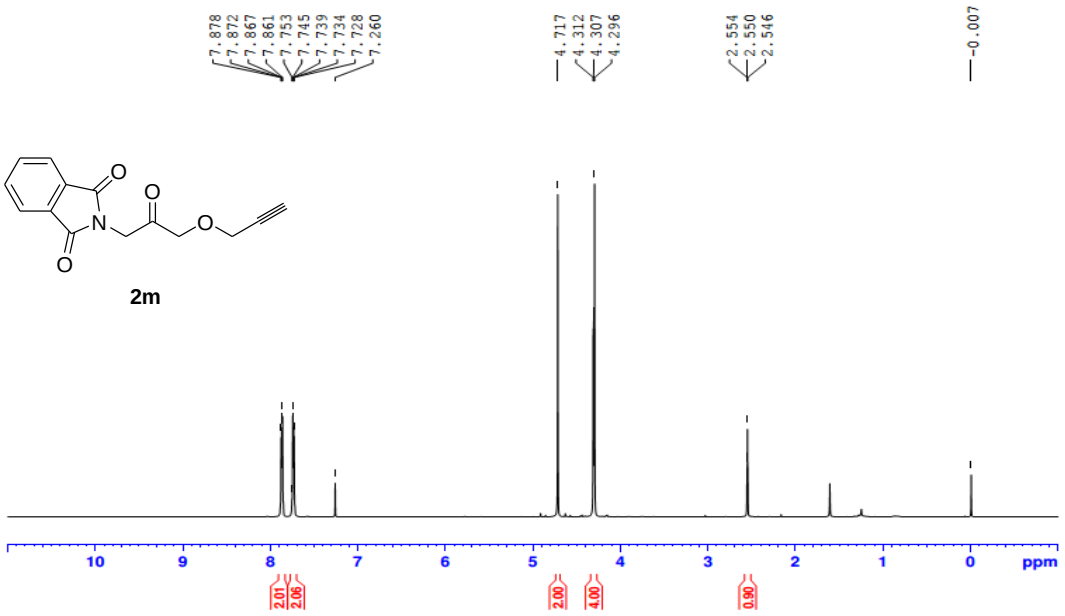
30
20
10
ppm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7.734
7.728
7.260
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4.312
4.307
4.296
2.554
2.550
2.546
0.007
O
O
N
O
O
2m
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6
5
4
3
2
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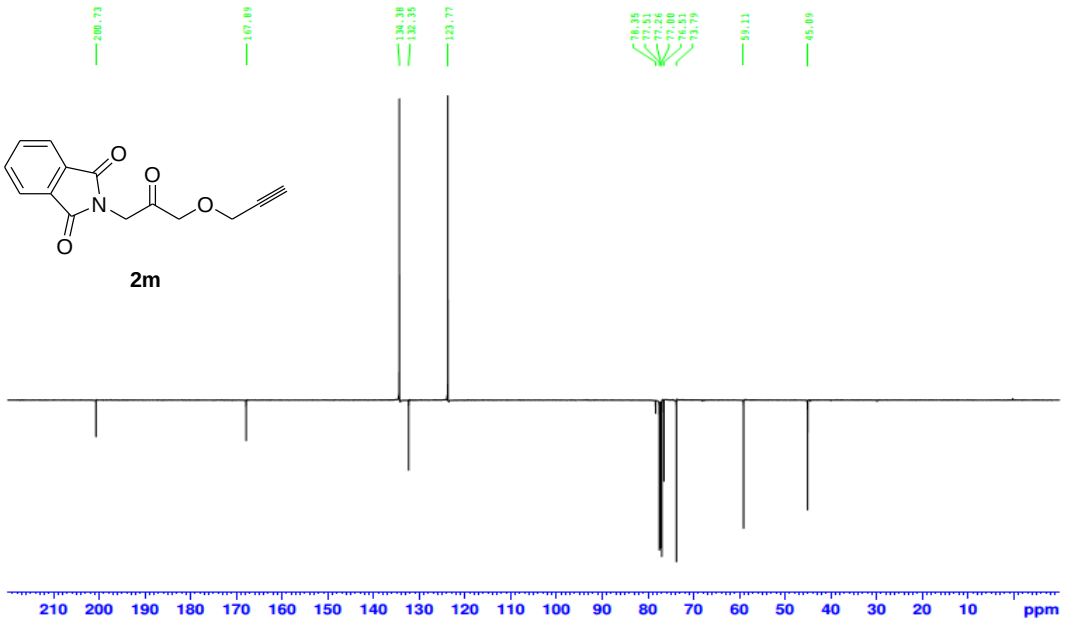
0
ppm
2.01
2.06
2.00
4.00
0.90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132.35
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78.35
77.51
77.26
77.00
76.51
73.79
59.11
45.09
O
O
N
O
O
2m
210
200
190
180
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150
140
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70
60
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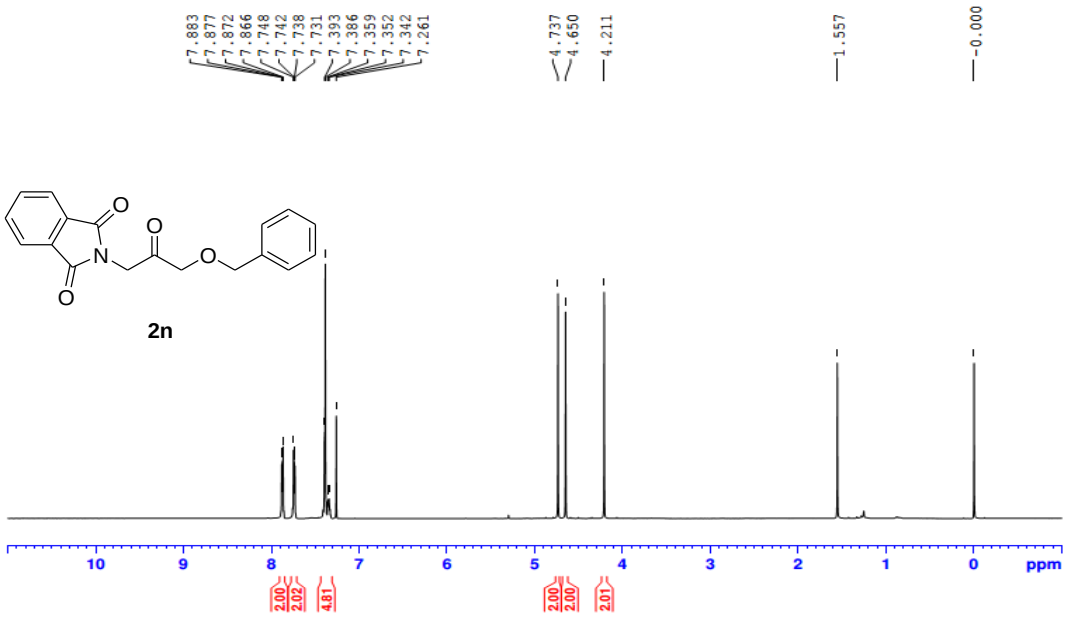
30
20
10
ppm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7.731
7.393
7.386
7.359
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7.342
7.261
4.737
4.650
4.211
1.557
0.000
2n
10
9
8
7
6
5
4
3
2
1
0
ppm
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2.02
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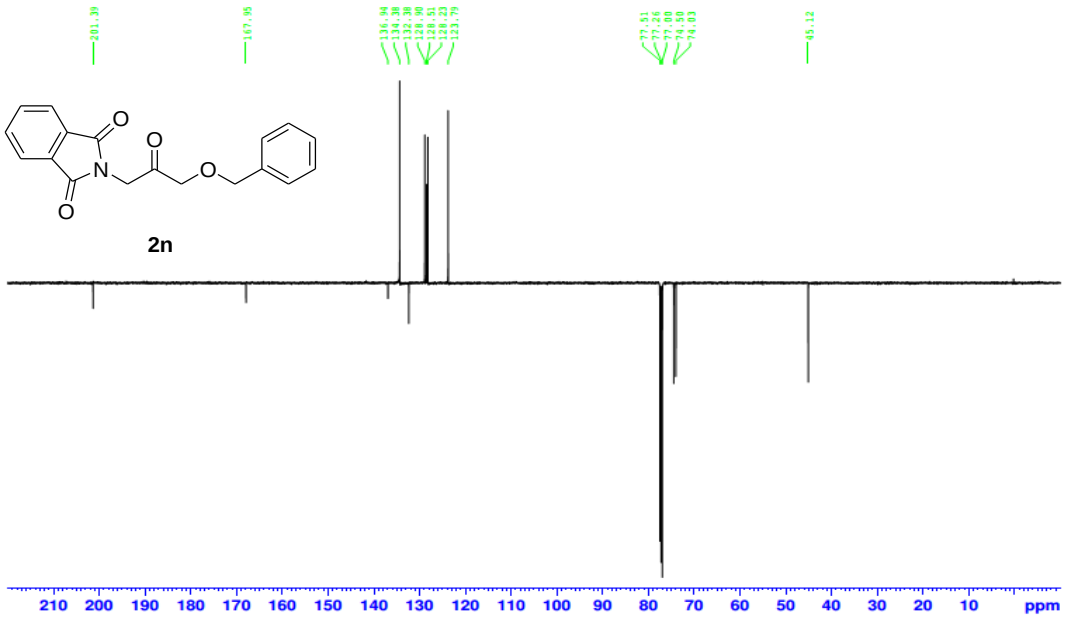
2.00
2.00
2.01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134.38
132.38
128.90
128.51
128.23
123.79
77.51
77.26
77.00
74.59
74.03
45.12
2n
210
200
190
180
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150
140
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110
100
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70
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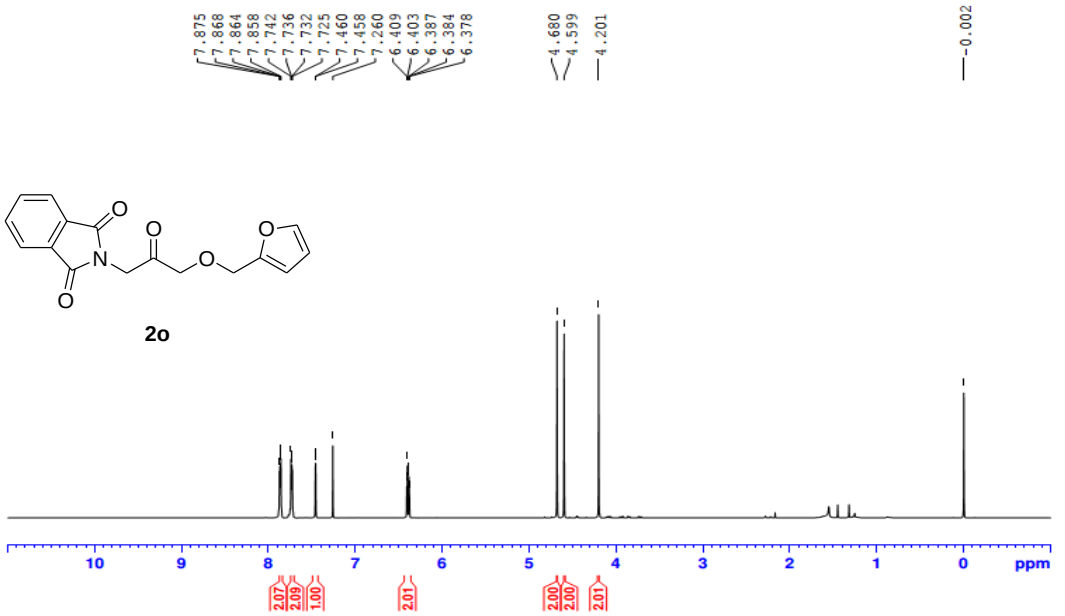
30
20
10
ppm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7.725
7.460
7.458
7.260
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6.403
6.387
6.384
6.378
4.680
4.599
4.201
0.002
2o
10
9
8
7
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5
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ppm
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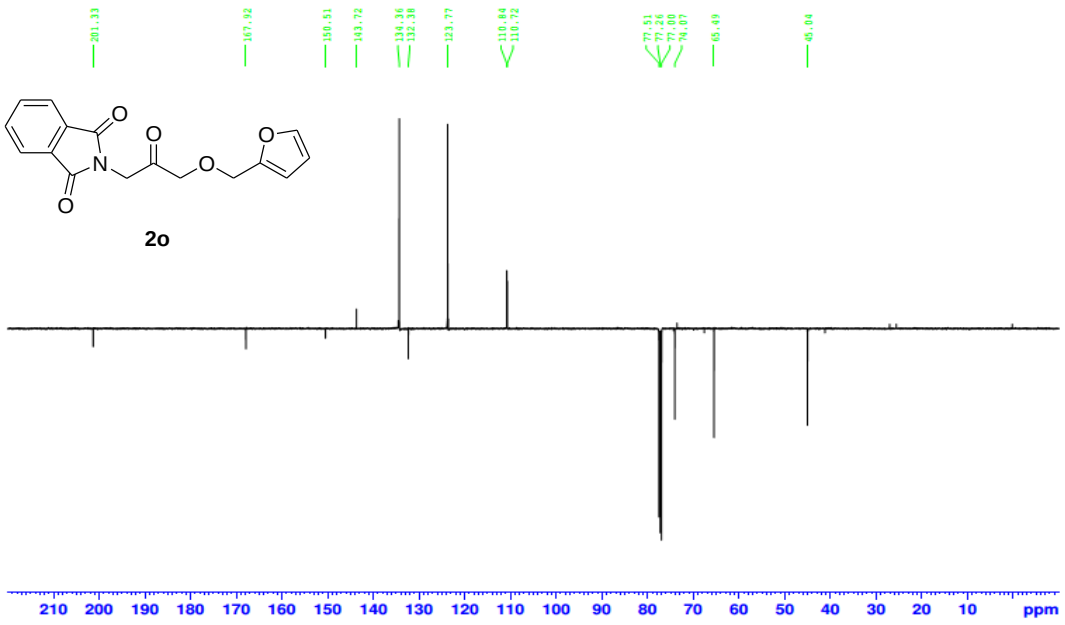
2.09
1.00
2.01
2.00
2.00
2.01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110.84
110.72
77.51
77.26
77.00
74.07
65.49
45.04
2o
210
200
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180
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120
110
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70
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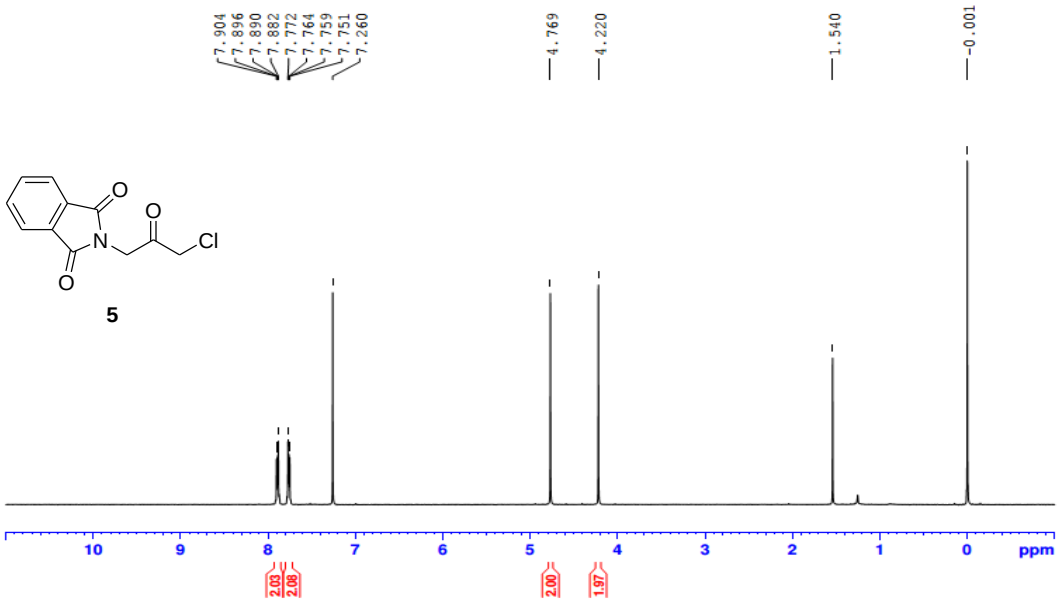
30
20
10
ppm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7.759
7.751
7.260
4.769
4.220
1.540
-0.001
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O
N
Cl
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ppm
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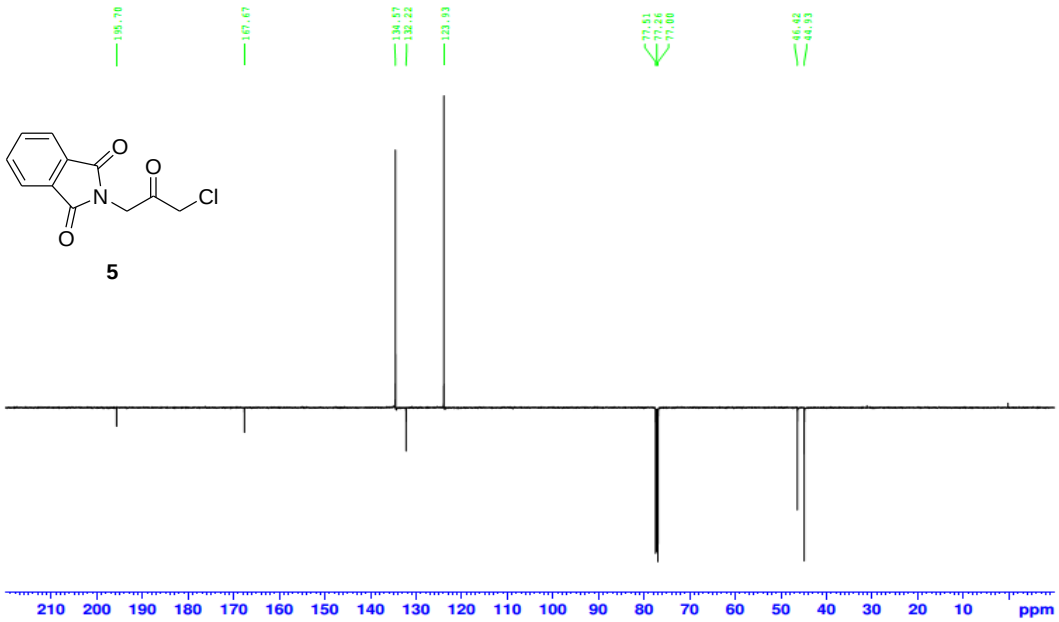
2.00
1.97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77.26
77.00
46.62
44.93
O
O
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5
210
200
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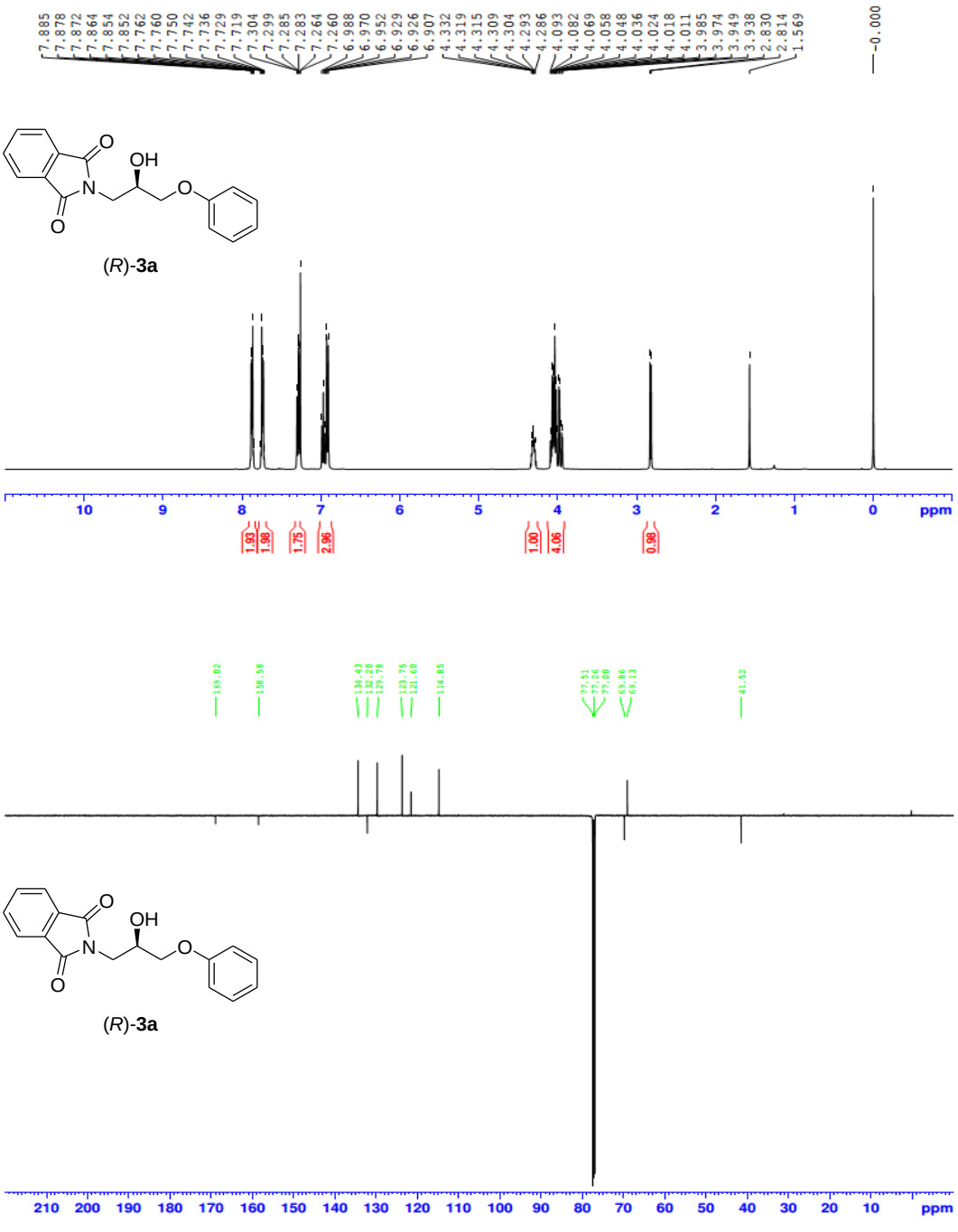
10
ppm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7.762
7.760
7.750
7.742
7.736
7.729
7.719
7.304
7.299
7.285
7.283
7.264
7.260
6.988
6.970
6.952
6.929
6.926
6.907
4.332
4.319
4.315
4.309
4.304
4.293
4.286
4.093
4.082
4.069
4.058
4.048
4.036
4.024
4.018
4.011
3.985
3.974
3.949
3.938
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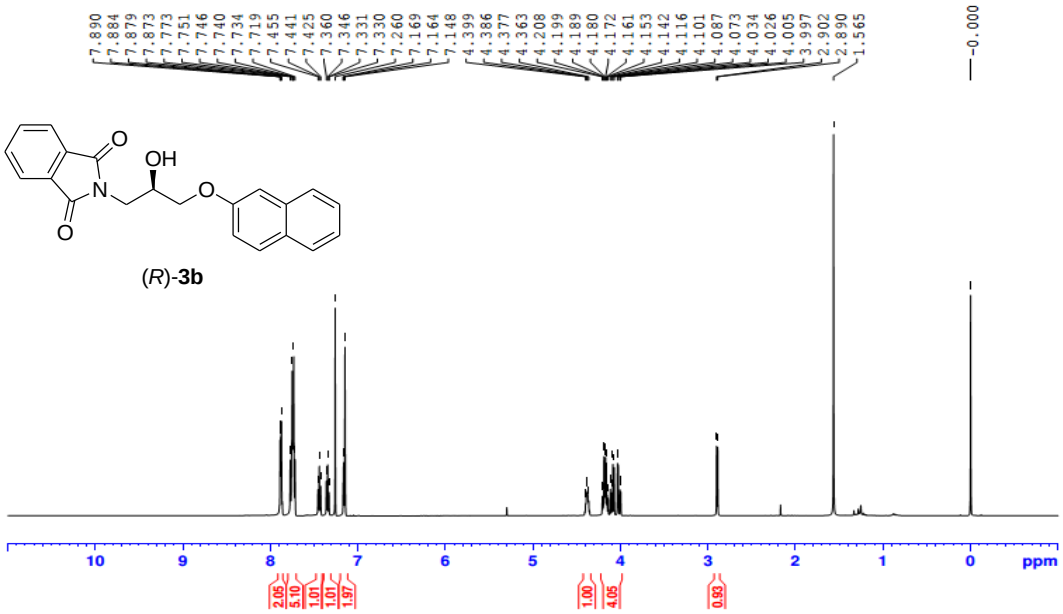
2.814
1.569
0.000
O
OH
N
O
O
10
9
8
7
6
5
4
3
2
1
0
ppm
1.93
1.98
1.75
2.96
1.00
4.06
0.98

(*R*)-**3a**

169.02
150.58
134.43
132.20
129.78
123.75
121.60
114.85
77.51
77.26
77.00
69.86
69.13
41.52
O
OH
N
O
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

(*R*)-**3a**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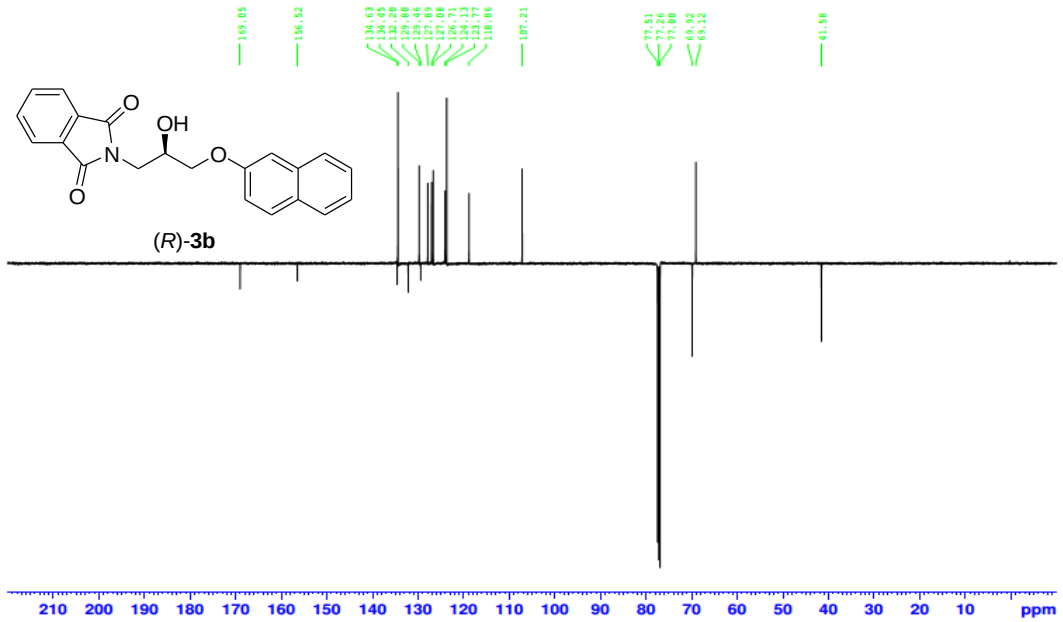
7.873
7.773
7.751
7.746
7.740
7.734
7.719
7.455
7.441
7.425
7.360
7.346
7.331
7.330
7.260
7.169
7.164
7.148
4.399
4.386
4.377
4.363
4.208
4.199
4.189
4.180
4.172
4.161
4.153
4.142
4.116
4.101
4.087
4.073
4.034
4.026
4.005
3.997
2.902
2.890
1.565
0.000
OH
O
N
O
O
(R)-3b
10
9
8
7
6
5
4
3
2
1
0
ppm
2.05
5.10
1.01
1.01
1.97
1.00
4.06
0.93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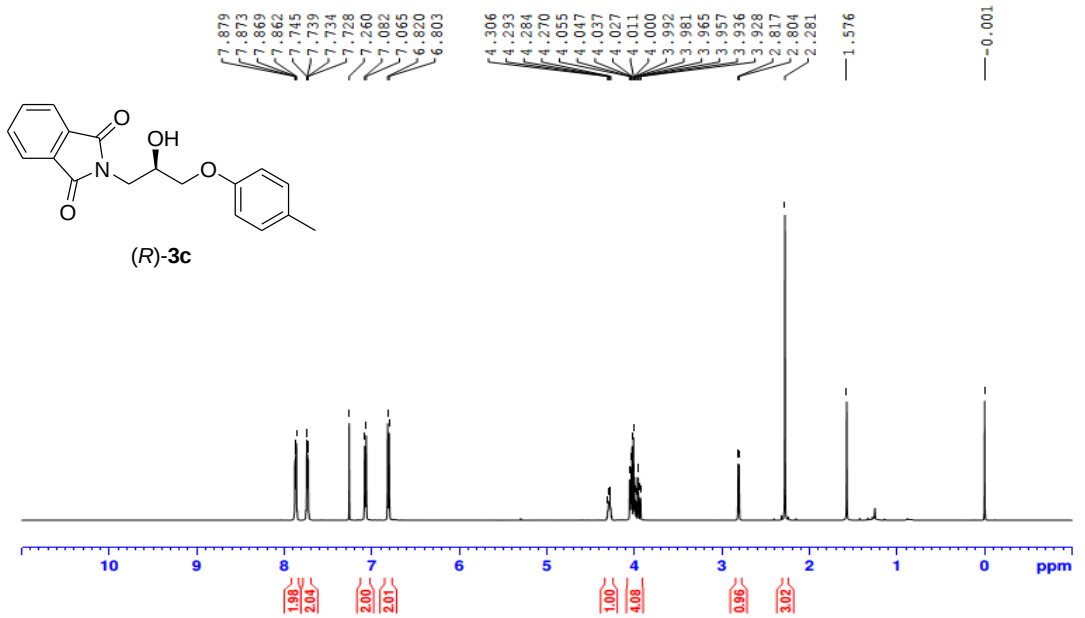
134.45
132.20
129.40
128.66
127.89
127.08
126.71
124.13
123.77
118.86
107.21
77.51
77.26
77.00
69.92
69.12
41.58
OH
O
N
O
O
(R)-3b
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.728
7.260
7.082
7.065
6.820
6.803
4.306
4.293
4.284
4.270
4.055
4.047
4.037
4.027
4.011
4.000
3.992
3.981
3.965
3.957
3.936
3.928
2.817
2.804
2.281
1.576
0.001
OH
O
N
O
O
(R)-3c
10
9
8
7
6
5
4
3
2
1
0
ppm
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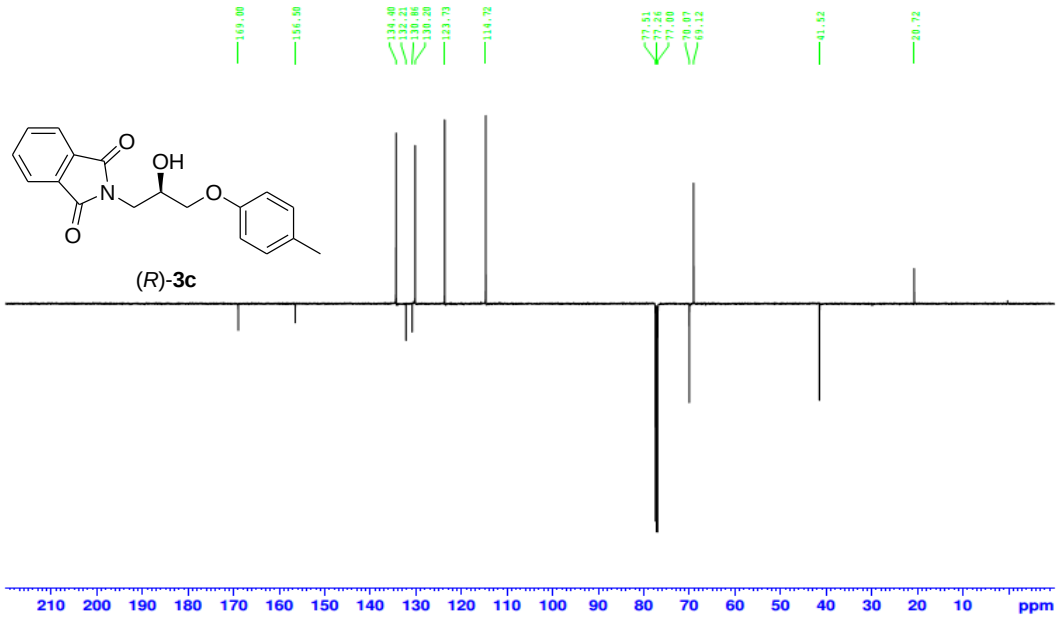
2.04
2.00
2.01
1.00
4.08
0.96
3.02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114.72
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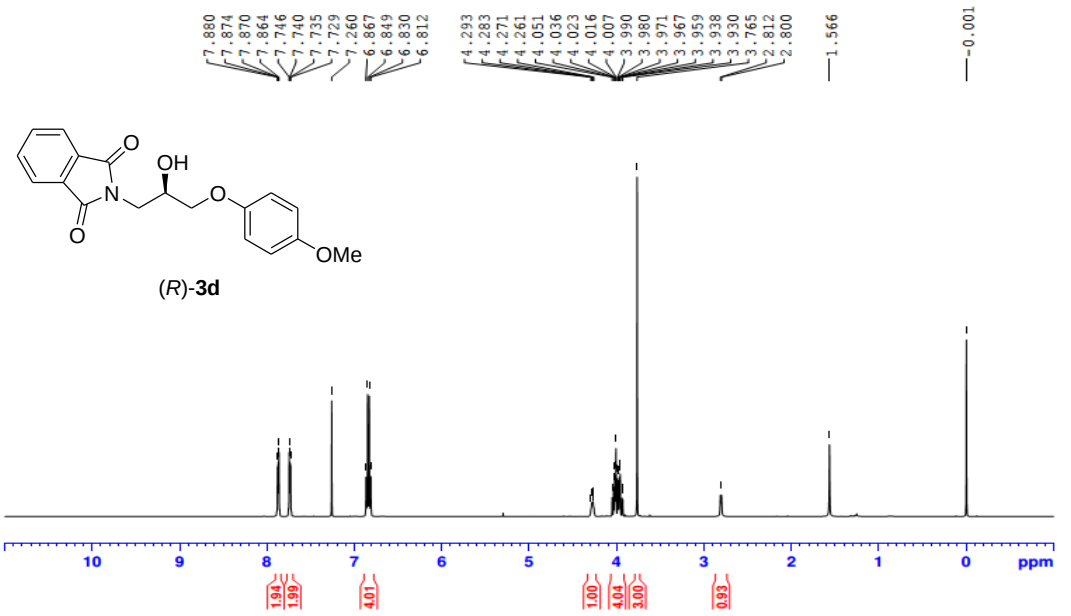
77.26
77.00
70.07
69.12
41.52
20.72
OH
O
N
O
O
(R)-3c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.870
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7.740
7.735
7.729
7.260
6.867
6.849
6.830
6.812
4.293
4.283
4.271
4.261
4.051
4.036
4.023
4.016
4.007
3.990
3.980
3.971
3.967
3.959
3.938
3.930
3.765
2.812
2.800
1.566
0.001
OH
O
N
O
O
OMe
(R)-3d
10
9
8
7
6
5
4
3
2
1
0
ppm
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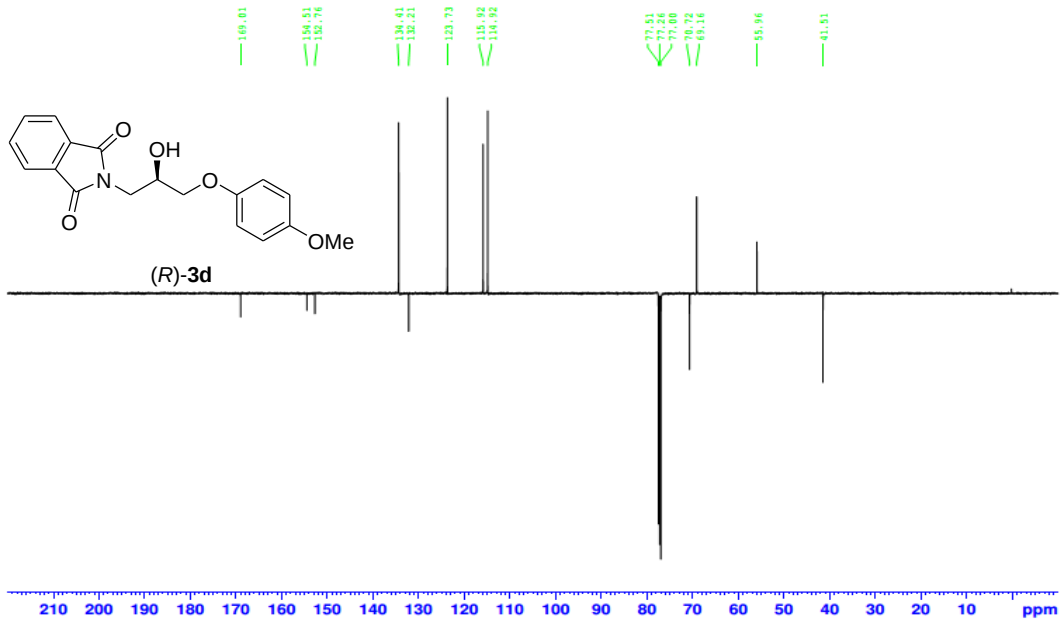
1.99
4.01
1.00
4.04
3.00
0.93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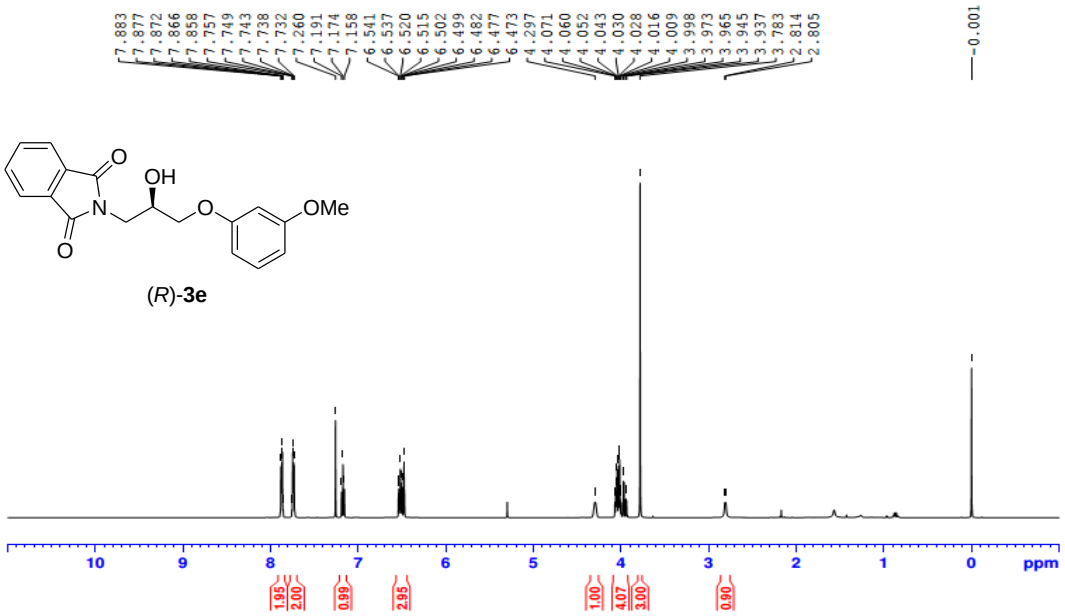
152.76
134.41
132.21
123.73
115.92
114.92
77.51
77.26
77.00
70.72
69.16
55.96
41.51
OH
O
N
O
O
OMe
(R)-3d
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.877
7.872
7.866
7.858
7.757
7.749
7.743
7.738
7.732
7.260
7.191
7.174
7.158
6.541
6.537
6.520
6.515
6.502
6.499
6.482
6.477
6.473
4.297
4.071
4.060
4.052
4.043
4.030
4.028
4.016
4.009
3.998
3.973
3.965
3.945
3.937
3.783
2.814
2.805
0.001
OH
O
N
O
OMe
(R)-3e
10
9
8
7
6
5
4
3
2
1
0
ppm
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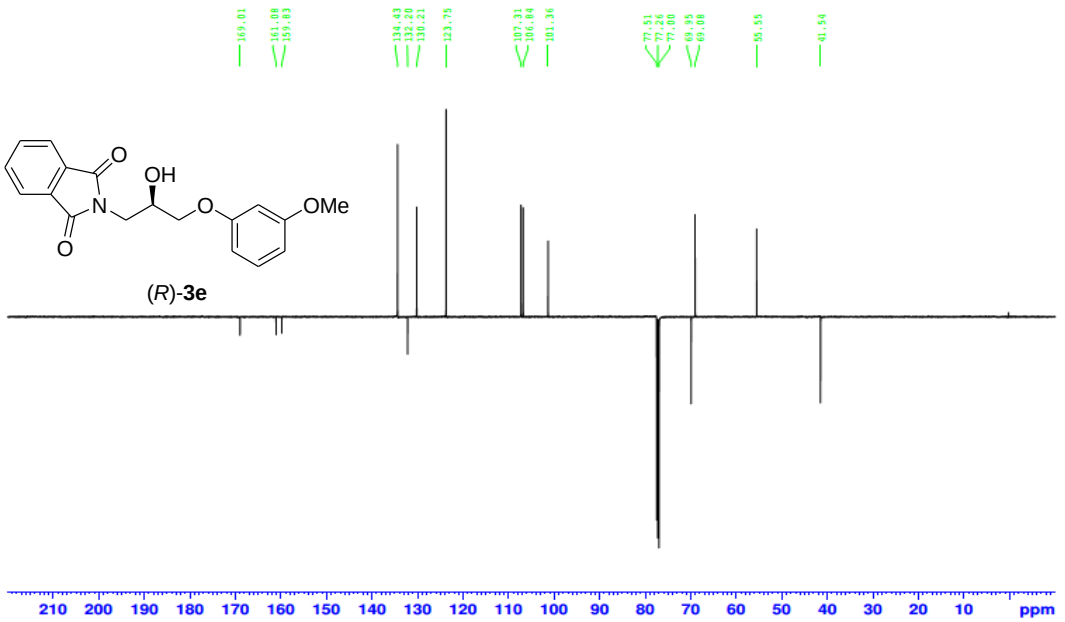
2.00
0.99
2.95
1.00
4.07
3.00
0.90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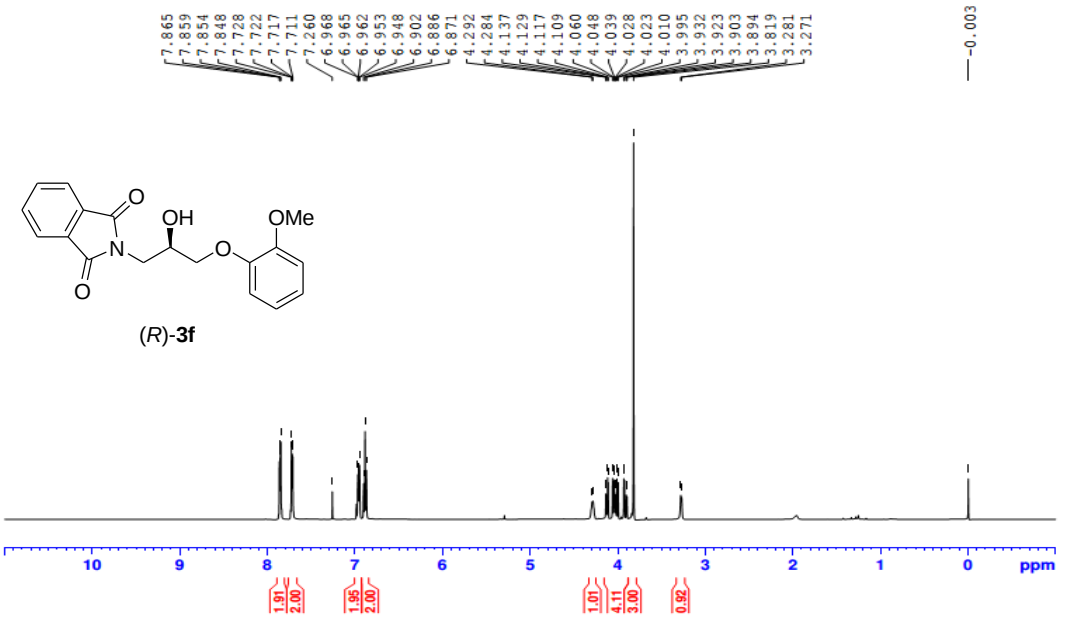
134.43
132.20
130.21
123.75
107.31
106.84
101.36
77.51
77.26
77.00
69.95
69.08
55.55
41.54
OH
O
N
O
OMe
(R)-3e
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.859
7.854
7.848
7.728
7.722
7.717
7.711
7.260
6.968
6.965
6.962
6.953
6.948
6.902
6.886
6.871
4.292
4.284
4.137
4.129
4.117
4.109
4.060
4.048
4.039
4.028
4.023
4.010
3.995
3.932
3.923
3.903
3.894
3.819
3.281
3.271
-0.003
O
OH
OMe
N
O
O
(R)-3f
10
9
8
7
6
5
4
3
2
1
0
ppm
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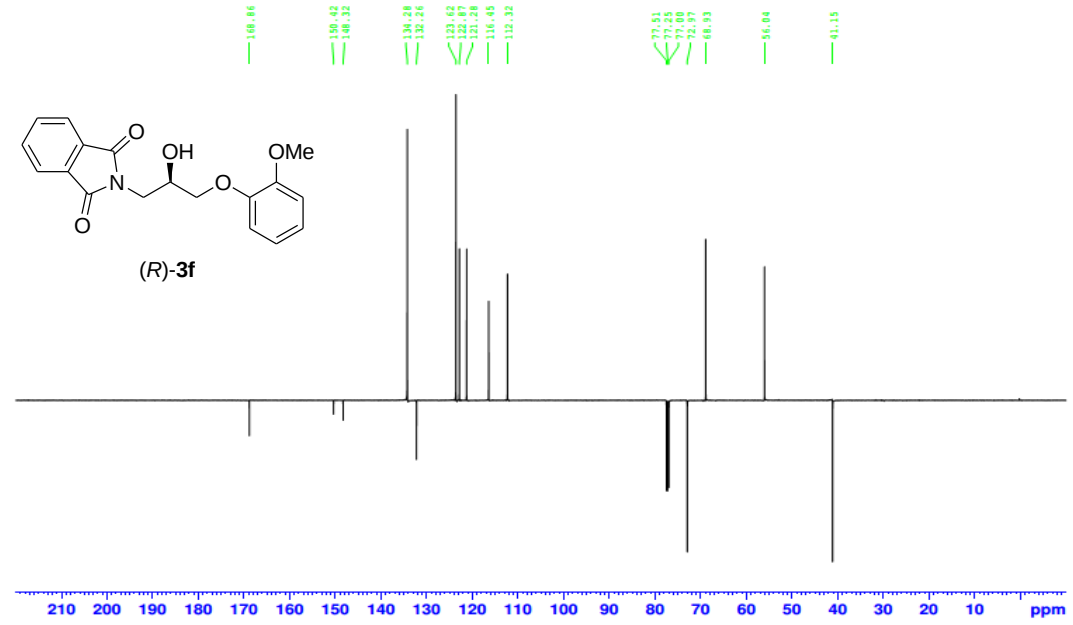
2.00
1.95
2.00
1.01
4.11
3.00
0.92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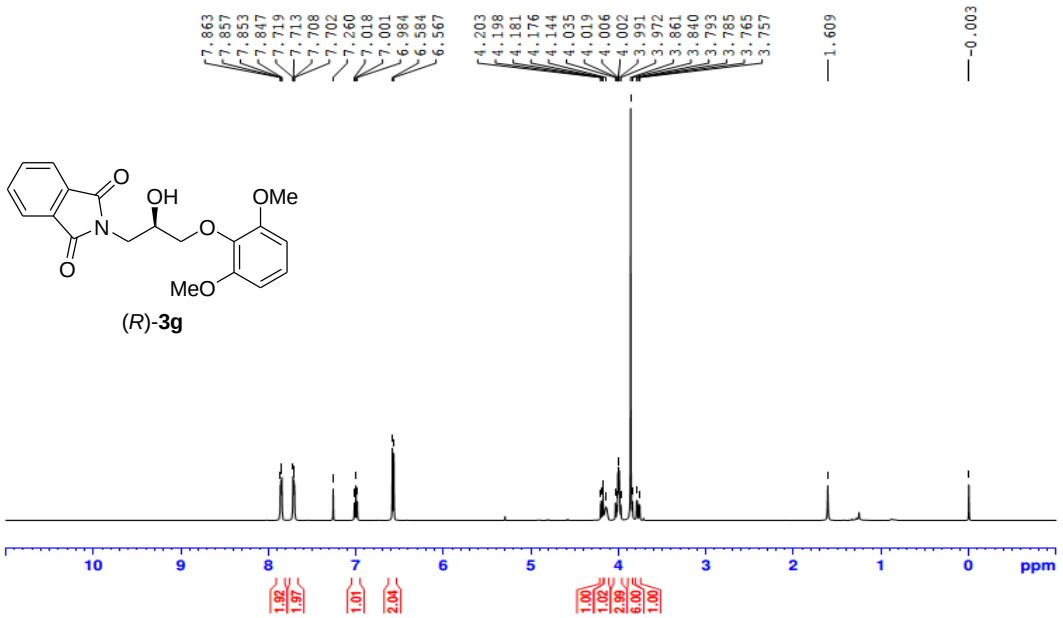
140.32
134.28
132.26
123.62
122.87
121.28
116.45
112.32
77.51
77.25
77.00
72.97
68.93
56.04
41.15
O
OH
OMe
N
O
O
(R)-3f
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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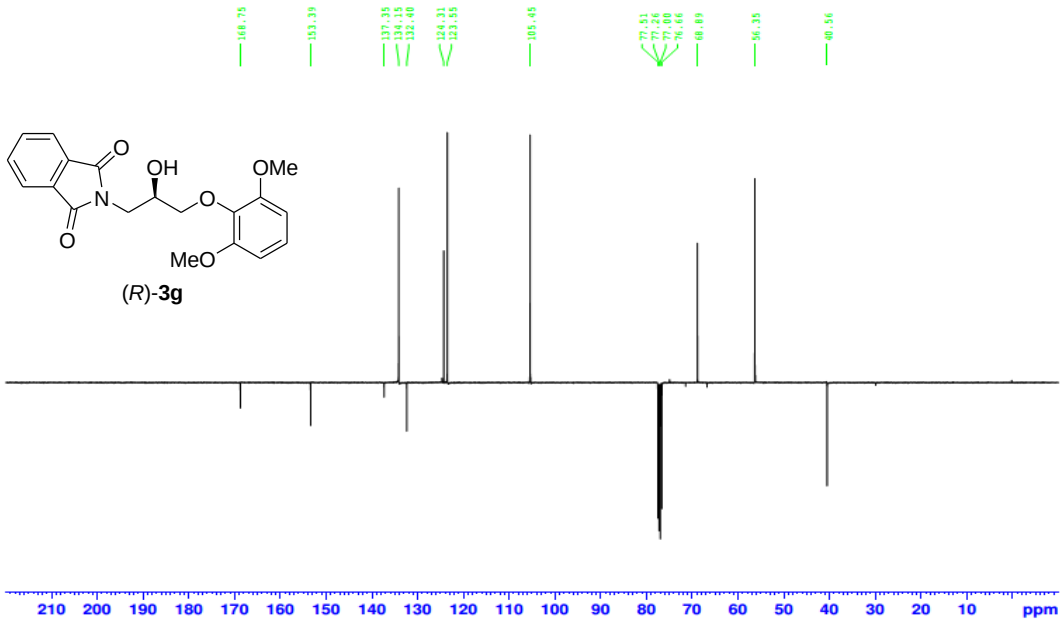
7.857
7.853
7.847
7.719
7.713
7.708
7.702
7.260
7.018
7.001
6.984
6.584
6.567
4.203
4.198
4.181
4.176
4.144
4.035
4.019
4.006
4.002
3.991
3.972
3.861
3.840
3.793
3.785
3.765
3.757
1.609
0.003
OH
OMe
O
N
O
O
MeO
(R)-3g
10
9
8
7
6
5
4
3
2
1
0
ppm
1.92
1.97
1.01
2.04
1.00
1.02
2.99
6.00
1.00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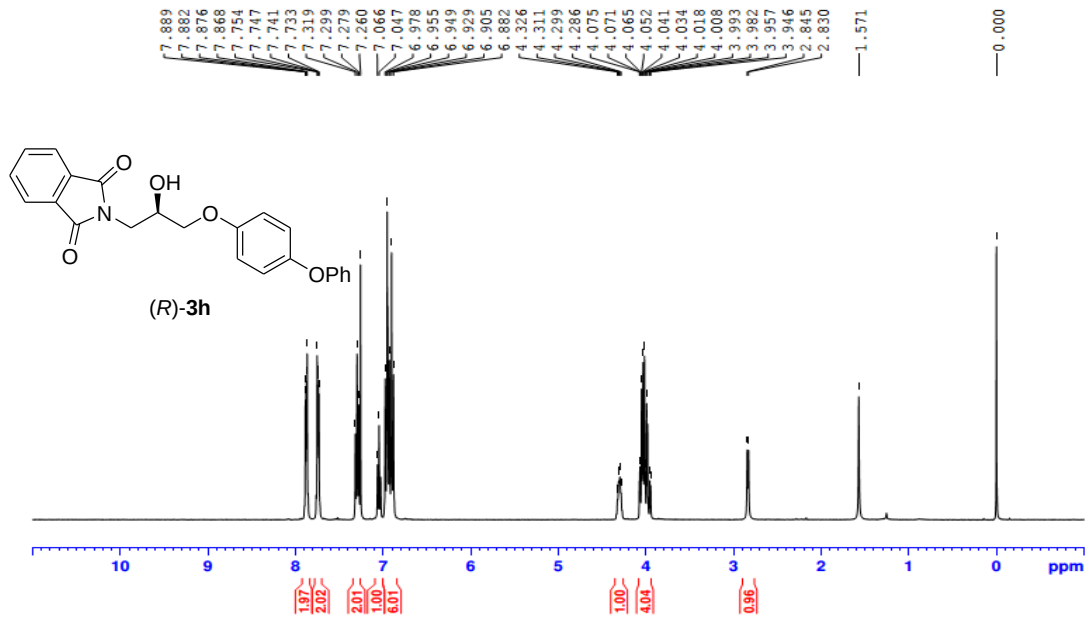
137.35
134.15
132.40
124.31
123.55
105.45
77.51
77.26
77.00
76.66
68.89
56.35
40.56
OH
OMe
O
N
O
O
MeO
(R)-3g
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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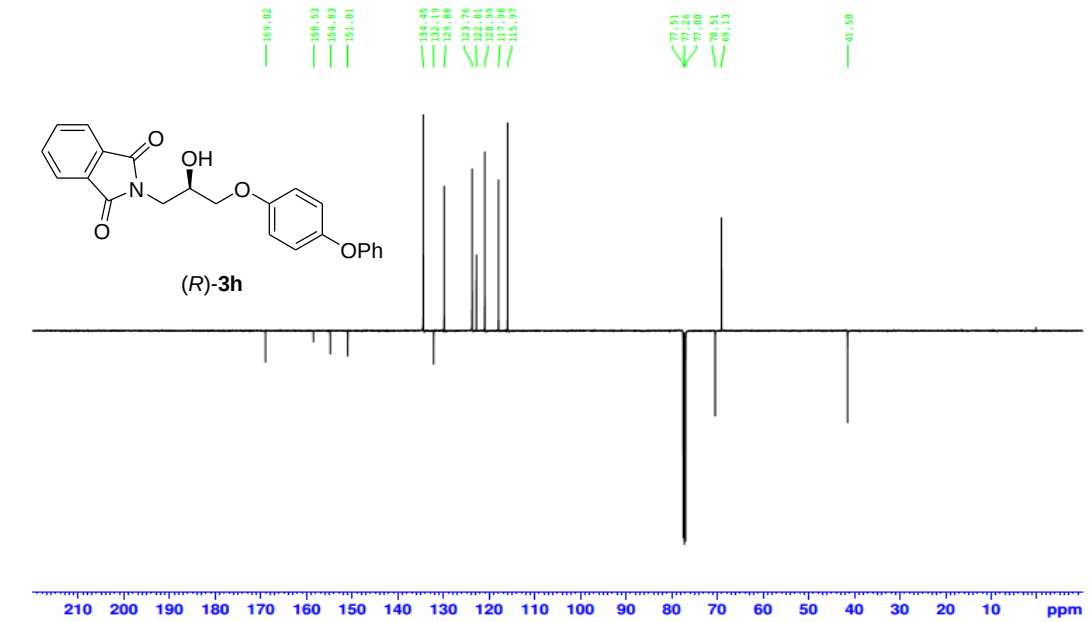
7.882
7.876
7.868
7.754
7.747
7.741
7.733
7.319
7.299
7.279
7.260
7.066
7.047
6.978
6.955
6.949
6.929
6.905
6.882
4.326
4.311
4.299
4.286
4.075
4.071
4.065
4.052
4.041
4.034
4.018
4.008
3.993
3.982
3.957
3.946
2.845
2.830
1.571
0.000
O
OH
N
O
O
OPh
(R)-3h
10
9
8
7
6
5
4
3
2
1
0
ppm
1.97
2.02
2.01
1.00
6.01
1.00
4.04
0.96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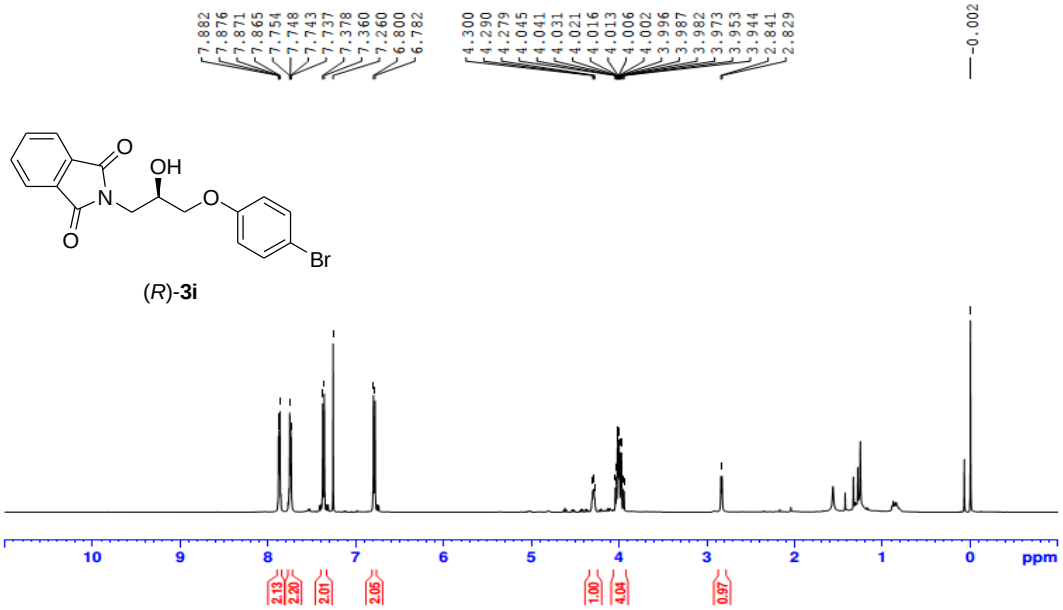
154.03
151.01
134.45
132.19
129.88
123.76
122.81
120.99
117.98
115.97
77.51
77.26
77.00
70.51
69.13
41.50
O
OH
N
O
O
OPh
(R)-3h
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.743
7.737
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7.360
7.260
6.800
6.782
4.300
4.290
4.279
4.045
4.041
4.031
4.021
4.016
4.013
4.006
4.002
3.996
3.987
3.982
3.973
3.953
3.944
2.841
2.829
-0.002
O
OH
N
O
O
Br
(R)-3i
10
9
8
7
6
5
4
3
2
1
0
ppm
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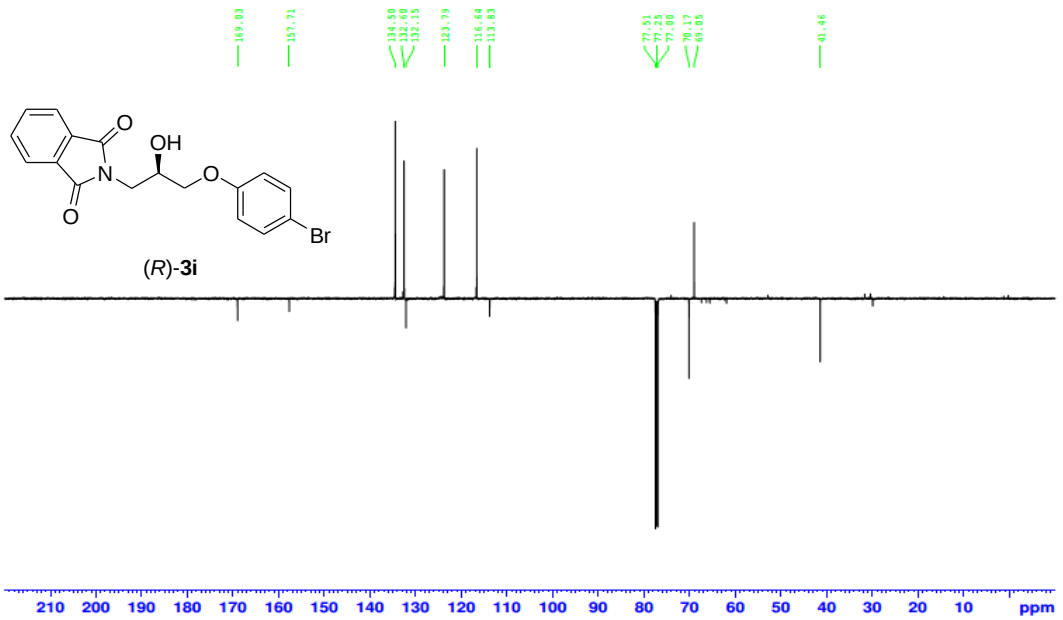
2.20
2.01
2.05
1.00
4.04
0.97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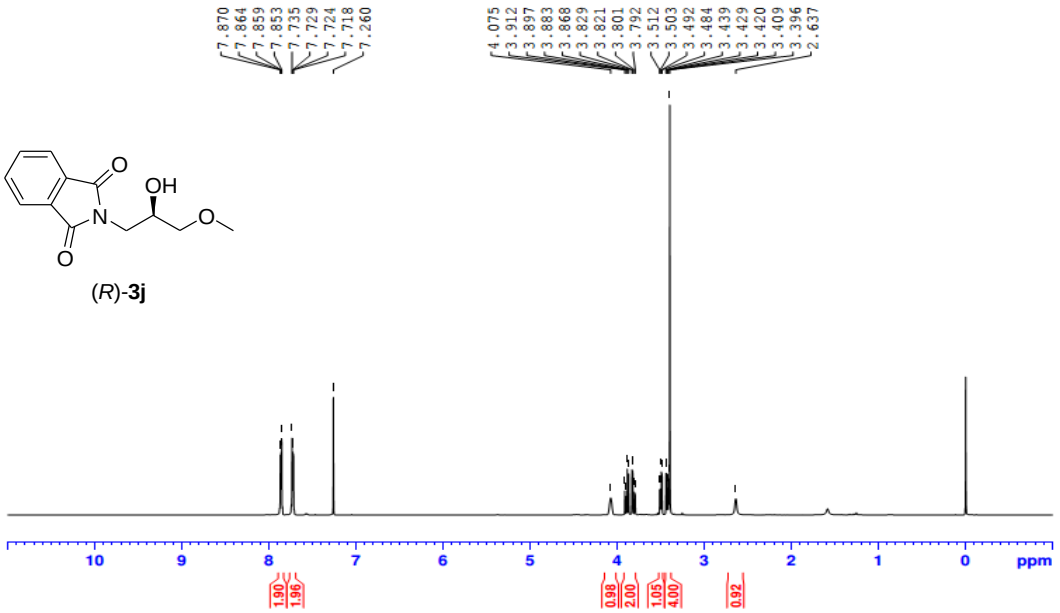
134.50
132.60
132.15
123.79
116.64
113.83
77.51
77.25
77.00
70.17
69.05
41.46
O
OH
N
O
O
Br
(R)-3i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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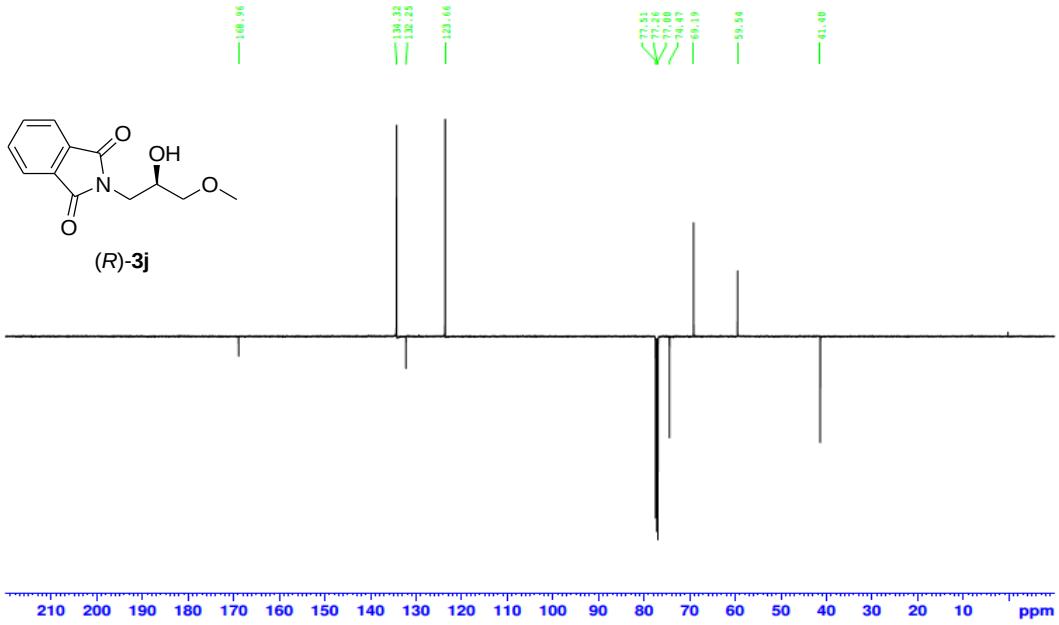
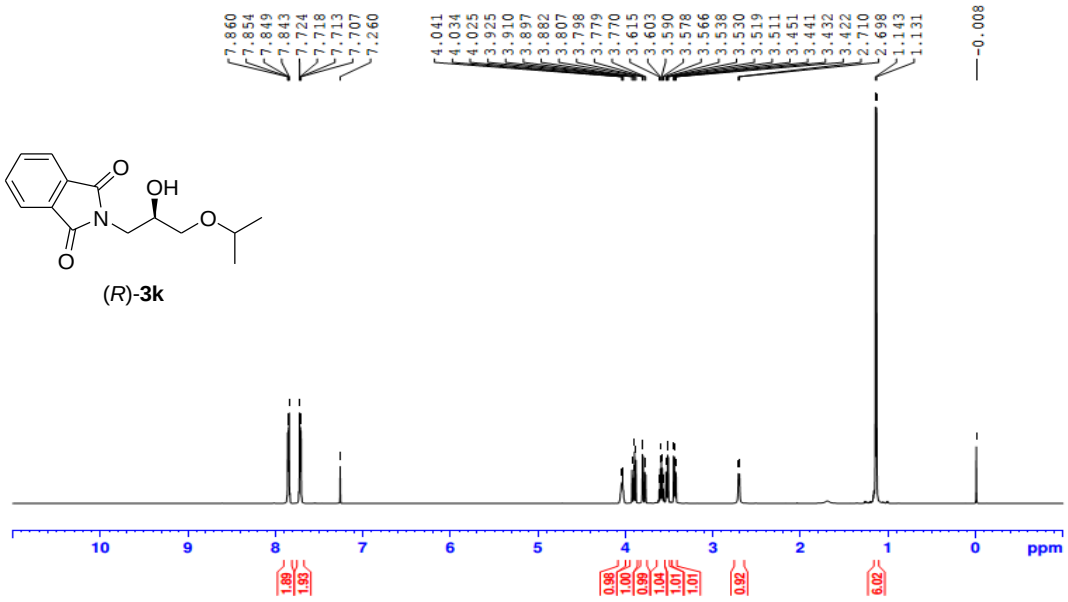
7.859
7.853
7.735
7.729
7.724
7.718
7.260
4.075
3.912
3.897
3.883
3.868
3.829
3.821
3.801
3.792
3.512
3.503
3.492
3.484
3.439
3.429
3.420
3.409
3.396
2.637
O
OH
N
O
O
10
9
8
7
6
5
4
3
2
1
0
ppm
1.90
1.96
0.98
2.00
1.05
4.00
0.92

(*R*)-**3j**


160.96
134.32
132.25
123.66
77.51
77.26
77.00
74.97
69.19
59.54
41.40
O
OH
N
O
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

(*R*)-**3j**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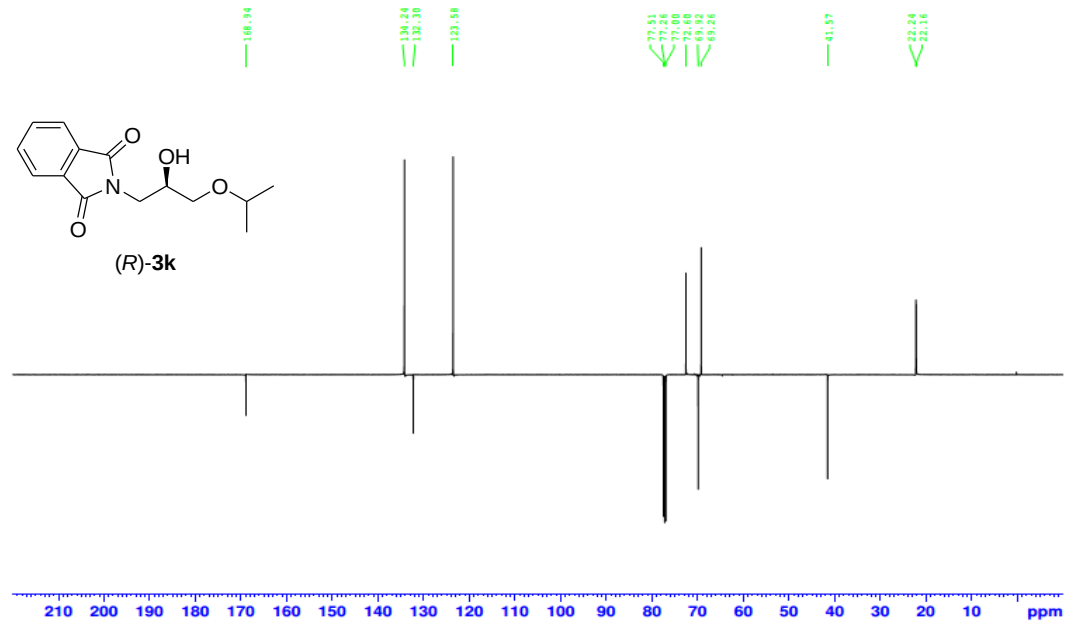
7.854
7.849
7.843
7.724
7.718
7.713
7.707
7.260
4.041
4.034
4.025
3.925
3.910
3.897
3.882
3.807
3.798
3.779
3.770
3.615
3.603
3.590
3.578
3.566
3.538
3.530
3.519
3.511
3.451
3.441
3.432
3.422
2.710
2.698
1.143
1.131
0.008
O
OH
N
O
O
10
9
8
7
6
5
4
3
2
1
0
ppm
1.88
1.93
0.90
1.00
0.98
1.04
1.01
1.01
0.92
6.02

(*R*)-**3k**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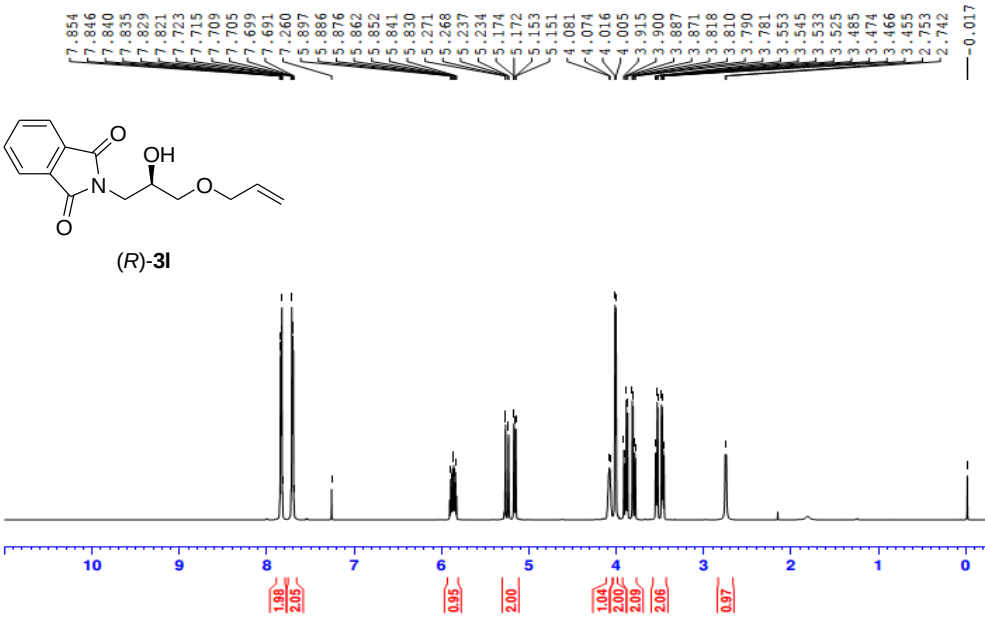
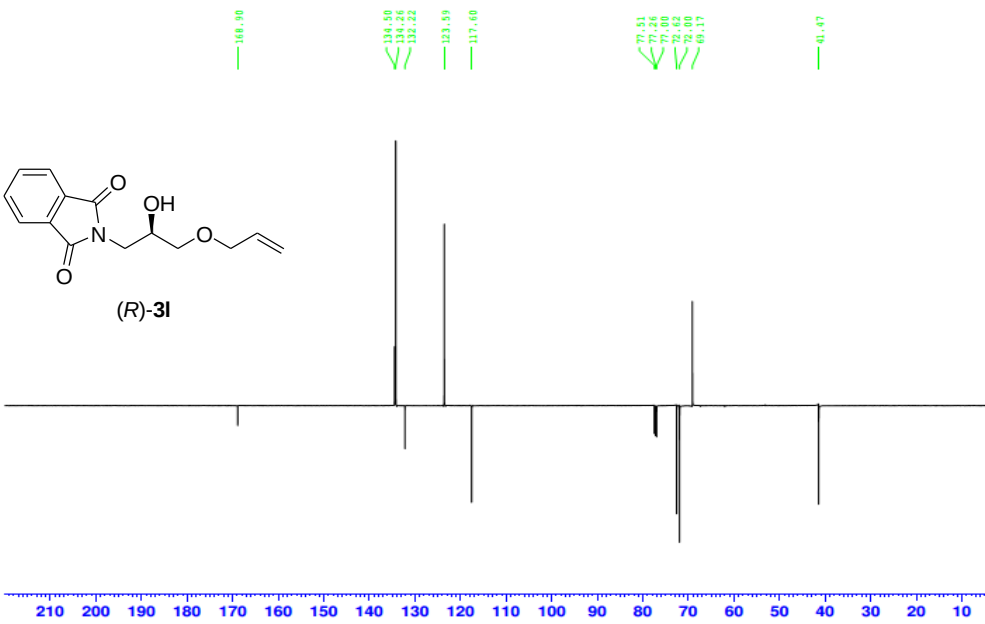
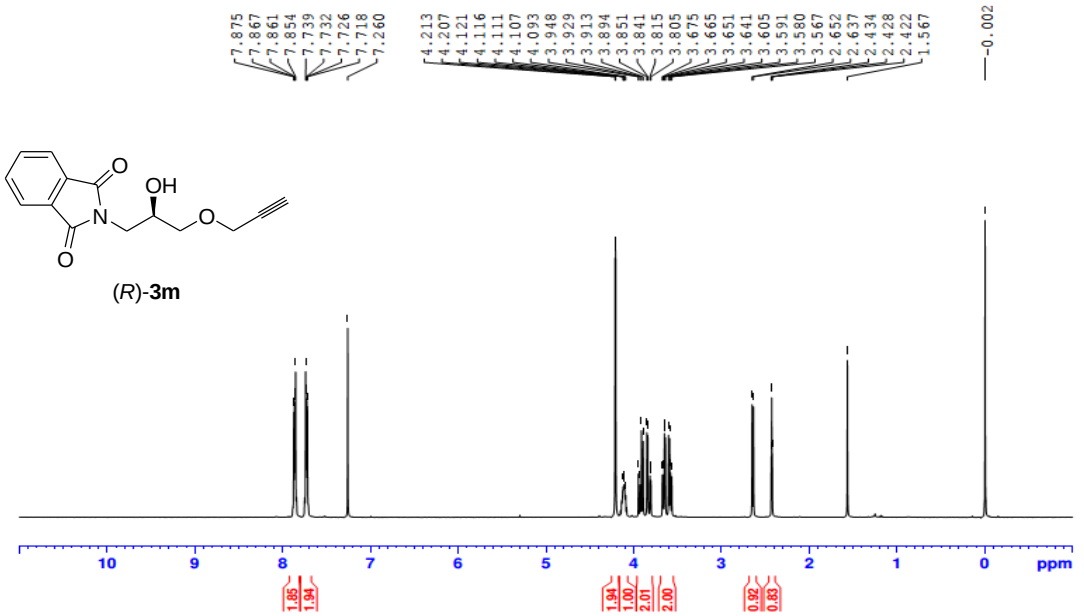
134.24
132.30
123.58
77.51
77.26
77.00
72.60
69.92
69.26
41.57
22.24
22.14
O
OH
N
O
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

(*R*)-**3k**


7.854
7.846
7.840
7.835
7.829
7.821
7.723
7.715
7.709
7.705
7.699
7.691
7.260
5.897
5.886
5.876
5.862
5.852
5.841
5.830
5.271
5.268
5.237
5.234
5.174
5.172
5.153
5.151
4.081
4.074
4.016
4.005
3.915
3.900
3.887
3.871
3.818
3.810
3.790
3.781
3.553
3.545
3.533
3.525
3.485
3.474
3.466
3.455
2.753
2.742
0.017
O
OH
N
O
O
(R)-3l
10
9
8
7
6
5
4
3
2
1
0
ppm
1.98
2.05
0.95
2.00
1.04
2.00
2.09
2.06
0.97


168.90
134.50
132.56
132.22
123.59
117.60
77.51
77.26
77.00
72.62
72.00
69.17
41.47
O
OH
N
O
O
(R)-3l
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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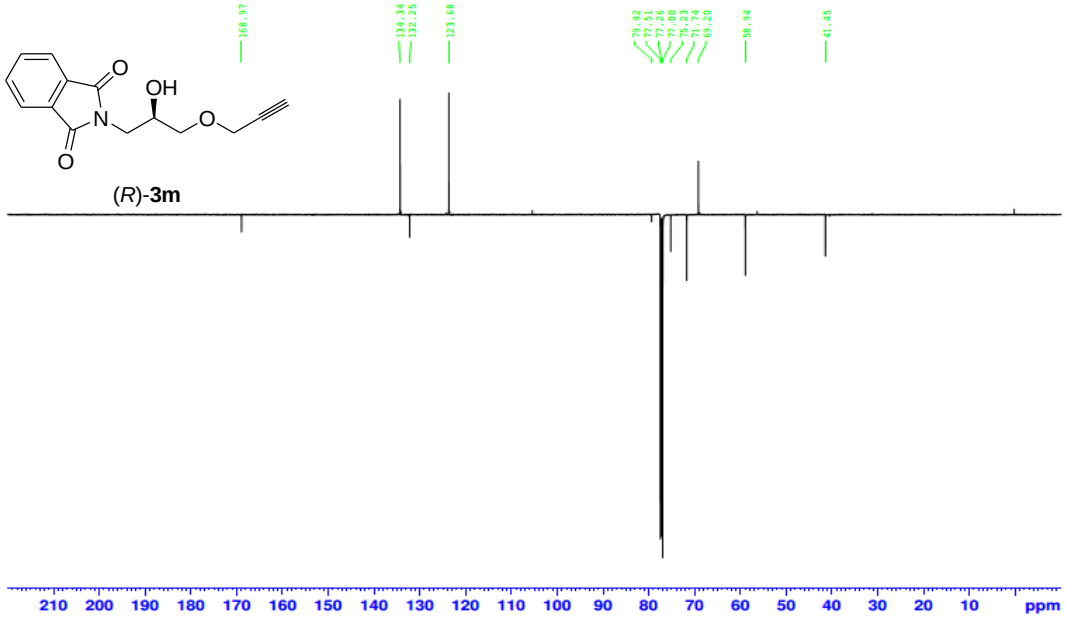
7.861
7.854
7.739
7.732
7.726
7.718
7.260
4.213
4.207
4.121
4.116
4.111
4.107
4.093
3.948
3.929
3.913
3.894
3.851
3.841
3.815
3.805
3.675
3.665
3.651
3.641
3.605
3.591
3.580
3.567
2.652
2.637
2.434
2.428
2.422
1.567
0.002
O
OH
N
O
O
(R)-3m
10
9
8
7
6
5
4
3
2
1
0
ppm
1.85
1.94
1.94
1.00
2.01
2.00
0.92
0.83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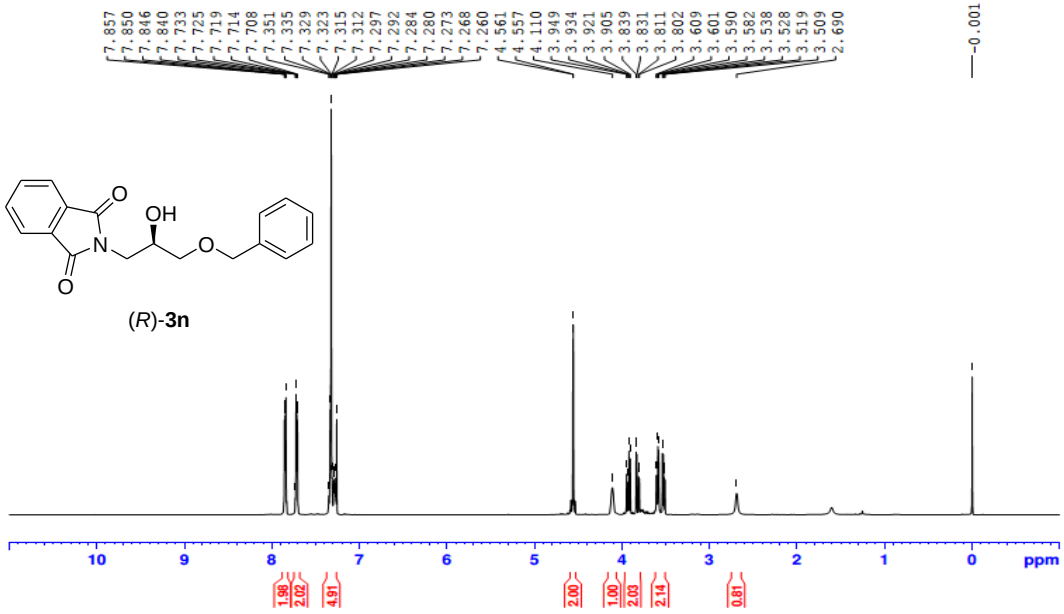
132.25
123.68
79.42
77.51
77.26
77.00
76.74
75.73
69.20
58.94
41.45
O
OH
N
O
O
(R)-3m
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.850
7.846
7.840
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7.725
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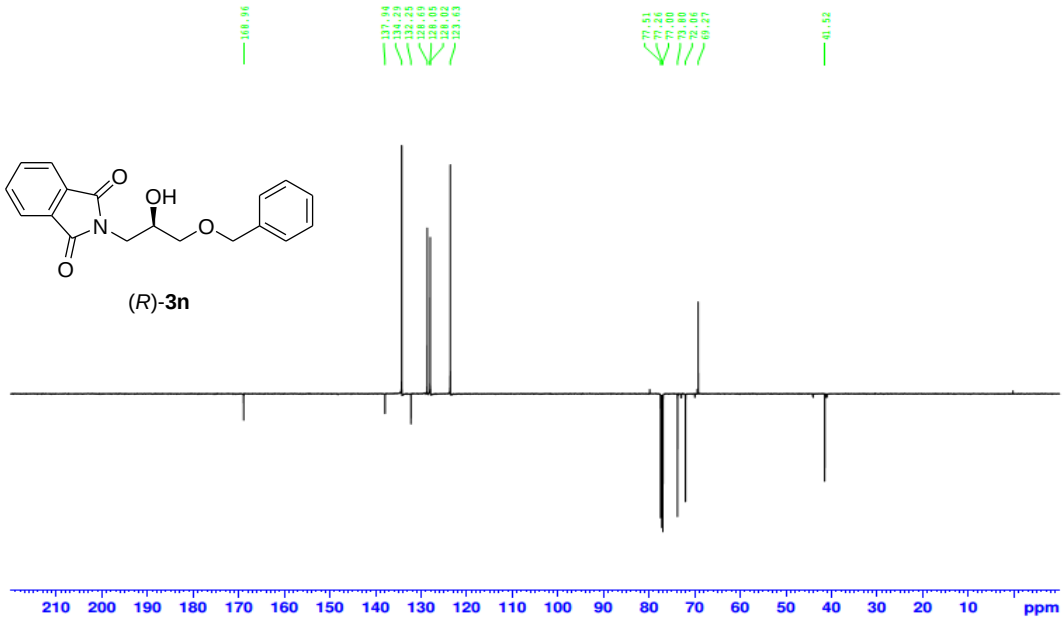
7.714
7.708
7.351
7.335
7.329
7.323
7.315
7.312
7.297
7.292
7.284
7.280
7.273
7.268
7.260
4.561
4.557
4.110
3.949
3.934
3.921
3.905
3.839
3.831
3.811
3.802
3.609
3.601
3.590
3.582
3.538
3.528
3.519
3.509
2.690
0.001
O
OH
N
O
O
(R)-3n
10
9
8
7
6
5
4
3
2
1
0
ppm
1.98
2.02
4.91
2.00
1.00
2.03
2.14
0.81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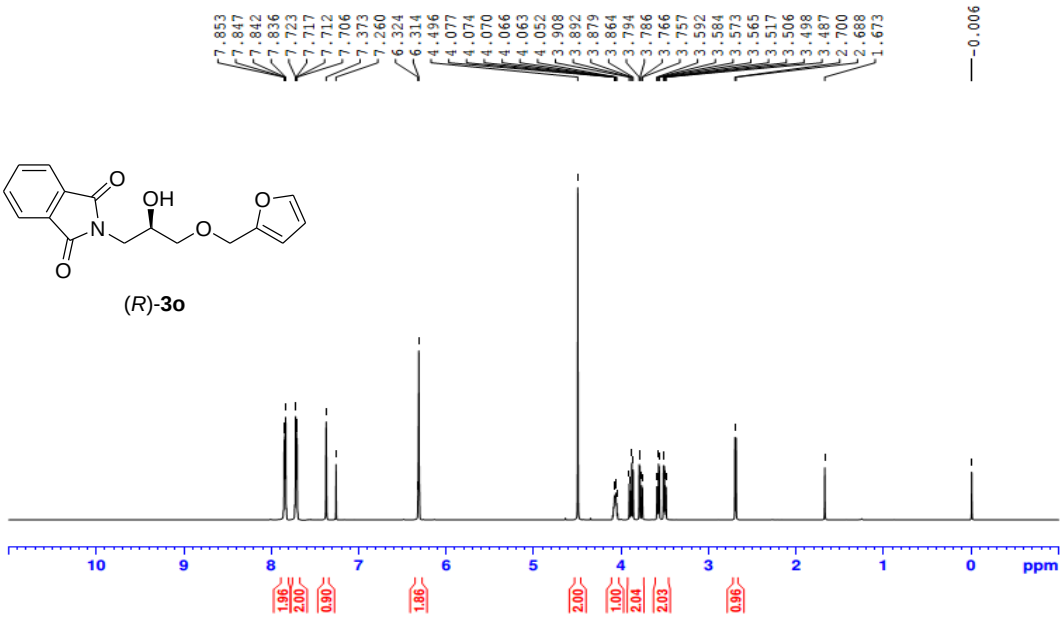
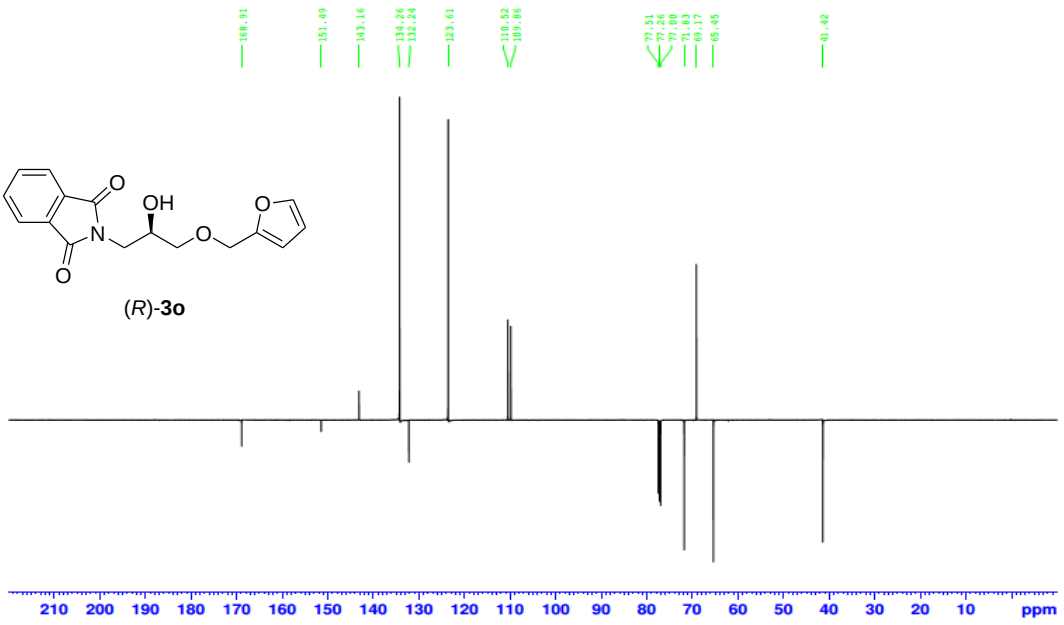
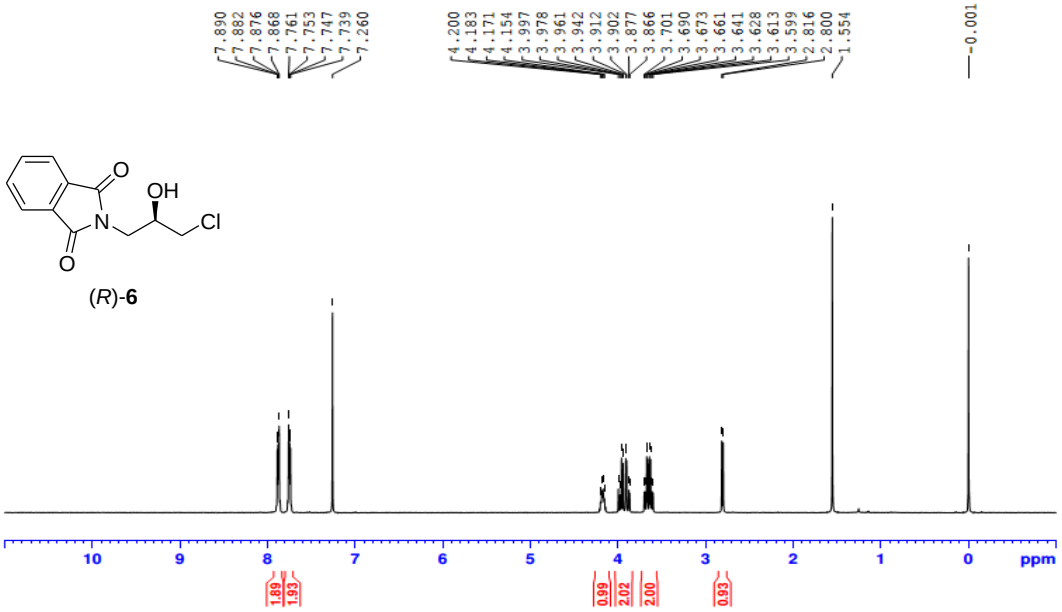
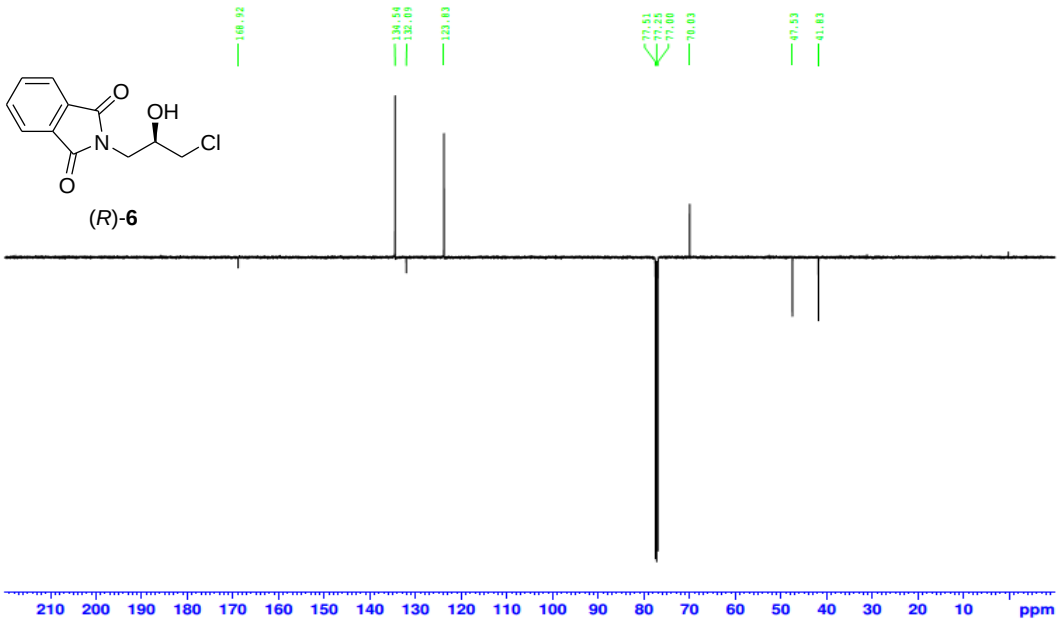
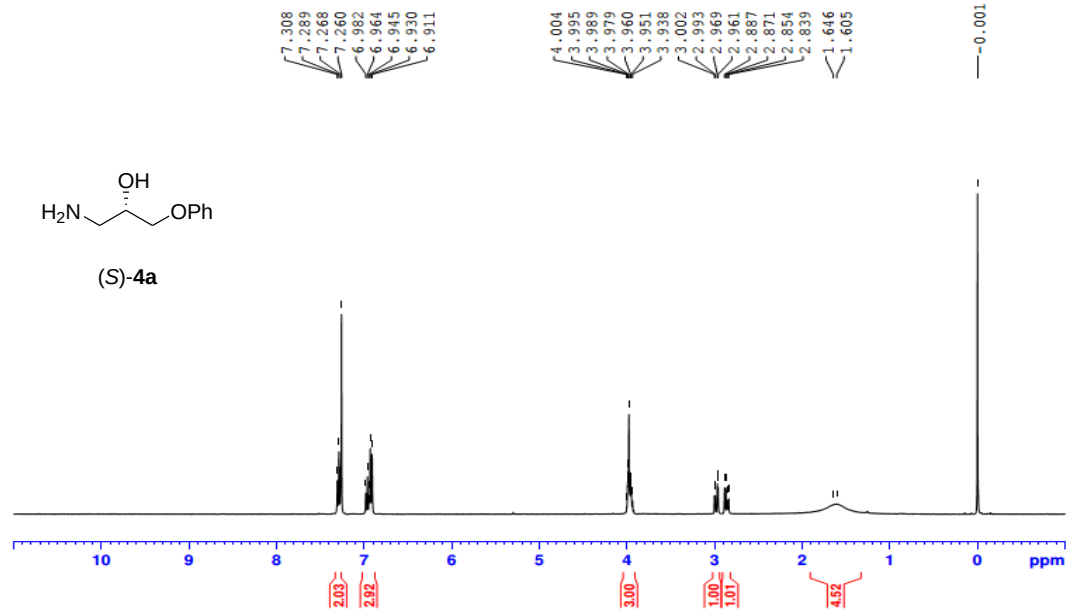
137.94
134.29
132.25
128.69
128.05
126.02
123.63
77.51
77.26
77.00
73.80
72.06
69.27
41.52
O
OH
N
O
O
(R)-3n
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

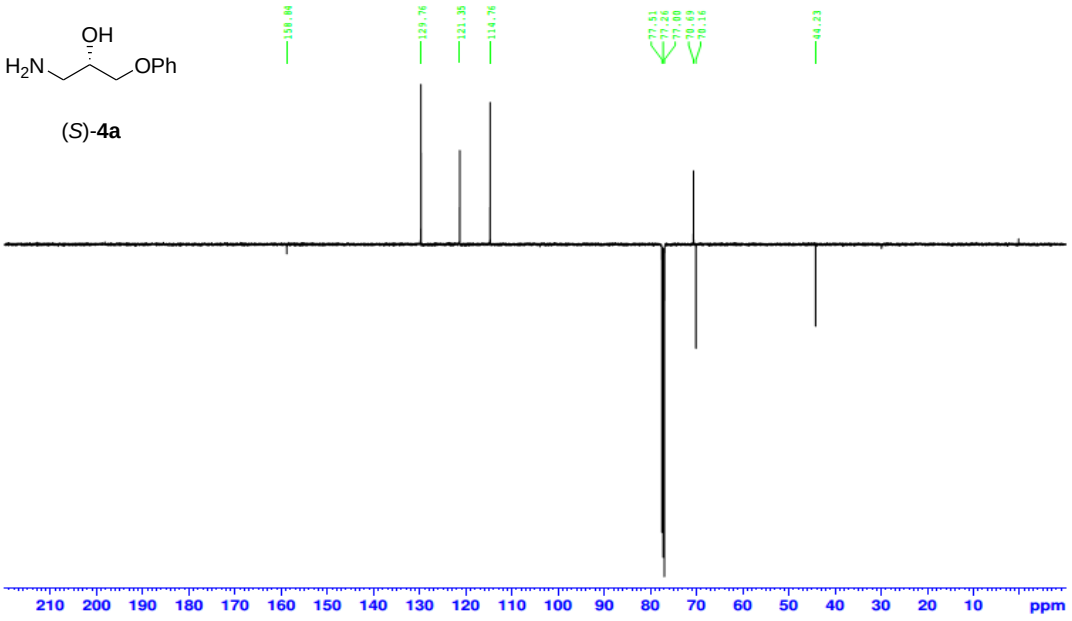

7.853
7.847
7.842
7.836
7.723
7.717
7.712
7.706
7.373
7.260
6.324
6.314
4.496
4.077
4.074
4.070
4.066
4.063
4.052
3.908
3.892
3.879
3.864
3.794
3.786
3.766
3.757
3.592
3.584
3.573
3.565
3.517
3.506
3.498
3.487
2.700
2.688
1.673
-0.006
O
OH
N
O
O
O
(R)-3o
10
9
8
7
6
5
4
3
2
1
0
ppm
1.96
2.00
0.90
1.86
2.00
1.00
2.04
2.03
0.96


168.91
151.49
143.16
134.26
132.24
123.61
110.52
109.86
77.51
77.26
77.00
71.03
69.17
65.45
41.42
O
OH
N
O
O
O
(R)-3o
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


7.890
7.882
7.876
7.868
7.761
7.753
7.747
7.739
7.260
4.200
4.183
4.171
4.154
3.997
3.978
3.961
3.942
3.912
3.902
3.877
3.866
3.701
3.690
3.673
3.661
3.641
3.628
3.613
3.599
2.816
2.800
1.554
-0.001
O
OH
N
Cl
O
(R)-6
10
9
8
7
6
5
4
3
2
1
0
ppm
1.09
1.93
0.99
2.02
2.00
0.93


168.92
134.54
132.09
123.83
77.51
77.25
77.00
70.03
47.53
41.83
O
OH
N
Cl
O
(R)-6
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


7.308
7.289
7.268
7.260
6.982
6.964
6.945
6.930
6.911
4.004
3.995
3.989
3.979
3.960
3.951
3.938
3.002
2.993
2.969
2.961
2.887
2.871
2.854
2.839
1.646
1.605
0.001
OH
H2N
OPh
(S)-4a
10
9
8
7
6
5
4
3
2
1
0
ppm
2.03
2.92
3.00
1.00
1.01
4.52


158.84
129.76
121.35
114.76
77.51
77.26
77.00
76.69
70.16
44.23
OH
H2N
OPh
(S)-4a
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm