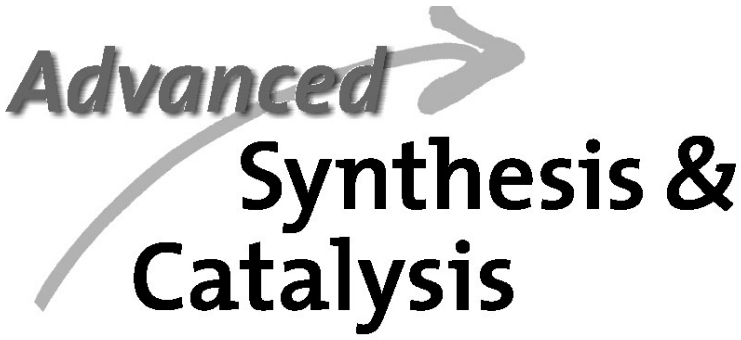

Supporting Information

1,3-Dipolar Cycloadditions of Carbonyl Ylides to Aldimines:

Scope, Limitations and Asymmetric Cycloadditions

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Supporting Information

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Chiral Lewis acids

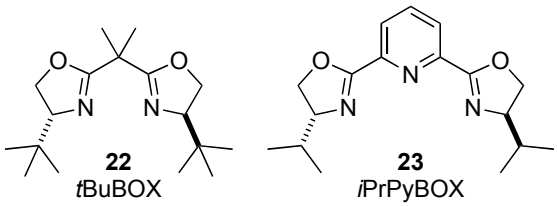
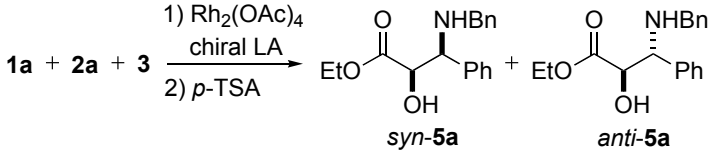


Figure 1. Chiral ligands

Table 1. 1,3-Dipolar cycloadditions using chiral Lewis acids[a]



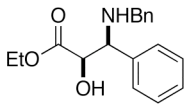
Entry	Lewis acid	Cond.[b] [°C / h]	d.r.[c] (*syn:anti*)	e.r.[d] (*syn*)	e.r.[d] (*anti*)	Yield[e] *syn*-**5a** (%)
1	**22**/Cu(OTf)$_2$	20 / 1	4.1:1	51:49	53:47	57
2	**22**/Cu(OTf)$_2$	-10 / 1	9.4:1	51:49	n.d.	52
3	**22**/Zn(OTf)$_2$	20 / 1	10.1:1	52:48	n.d.	68
4	**23**/Yb(OTf)$_3$	-15 / 1	4.5:1	rac	54:46	36
5	**23**/Sc(OTf)$_3$	-15 / 1	3.6:1	rac	53:47	34

[a] The reaction was carried out with imine **1a** (1.0 equiv), benzaldehyde (1.5 equiv), $Rh_2(OAc)_4$ (2.0 mol%), chiral Lewis acid (10 mol%) and powdered 4Å MS in CH_2Cl_2 with addition of ethyl diazoacetate (1.5 equiv). [b] Reaction temperature and addition time. [c] Determined by ^{1}H NMR spectroscopic analysis of the crude reaction mixture. [d] Determined by chiral HPLC (Chiracel OD-column). [e] Isolated yield.

General Experimental Procedure.

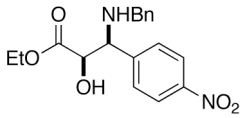
^{1}H and ^{13}C NMR spectra were recorded at 400 MHz (100 MHz) or 500 MHz (125 MHz) on a Bruker Avance 400 or a Bruker Avance DMX 500 instrument in $CDCl_3$, DMSO-d_6 or MeOH-d_4 using the residual peak of $CHCl_3$ (^{1}H NMR δ = 7.26 ppm, ^{13}C NMR δ = 77.0 ppm), DMSO (^{13}C NMR δ = 39.5 ppm) or MeOH (^{1}H NMR δ = 3.30 ppm) as internal standard. Chemical shifts are reported in the δ-scale with multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet and m = multiplet), integration and coupling constant (Hz). IR-spectra were recorded on an ATI Mattson Infinity Series FTIR and only the strongest/structurally most important peaks (ν_{max}, cm^{-1}) are listed. Optical rotations were determined on a Perkin Elmer Polarimeter 343 at the sodium D line (589 nm) and at ambient temperature. Melting points where measured with a Stuart Scientific SMP3 melting point apparatus and are uncorrected. Dichloromethane (CH_2Cl_2) was distilled from CaH_2 under a nitrogen atmosphere before use. Air- and moisture sensitive reactions were carried out in flame-dried, septum-capped flasks under an atmospheric pressure of nitrogen. All liquid reagents were transferred via oven-dried syringes. Commercially available compounds were used without further purification unless otherwise indicated. Powdered 4Å molecular sieves (4Å MS) were activated under vacuum with a heat gun for 5 min prior to use. TLC analyses were performed on Merck silica gel 60 F_{254} plates, using UV and a solution of phosphomolybdic acid in ethanol (5 wt%) for visualization. Flash chromatography was performed using SDS silica gel 60 (35-63 μm).

(2*R,3*S**)-Ethyl 3-(benzylamino)-2-hydroxy-3-phenylpropanoate (5a).**



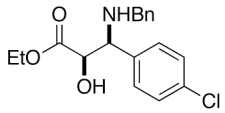
The general procedure was followed using **1a** (24.3 μL, 0.128 mmol). D.r. 93:7 (*syn*:*anti*), ^{1}H NMR spectroscopic analysis on the crude product. Flash chromatography (heptane:EtOAc 4:1 → 2:1) of the residue gave *syn*-**5a** (31.3 mg, 82%) as a white solid: mp 76-77 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.38-7.23 (m, 10H), 4.23 (d, J = 4.0 Hz, 1H), 4.21-4.11 (m, 2H), 3.95 (d, J = 4.0 Hz, 1H), 3.76 (d, J_{AB} = 13.3 Hz, 1H), 3.50 (d, J_{AB} = 13.3 Hz, 1H), 1.16 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 173.5, 140.0, 139.6, 128.5, 128.3, 128.2, 127.8, 127.0, 74.9, 63.6, 61.7, 50.7,14.0; IR (film) ν_{max} = 3467 (br), 3028, 2981, 2927, 1734, 1454, 1100 cm^{-1}; HRMS (FAB+) calcd for $C_{18}H_{22}NO_3$ (M+H): 300.1600, found: 300.1601.

(2*R,3*S**)-Ethyl 3-(benzylamino)-2-hydroxy-3-(4-nitrophenyl)propanoate (5e)**



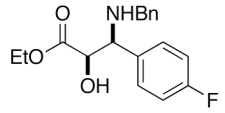
The general procedure was followed using **1e** (61.5 mg, 0.256 mmol). D.r. 91:9 (*syn*:*anti*), ^{1}H NMR spectroscopic analysis on the crude product. Addition of ethyl diazoacetate over 10h at rt. Flash chromatography (heptane:EtOAc 4:1 → 2:1) of the residue gave *syn*-**5e** (53.6 mg, 61%) as a pale yellow solid and *anti*-**5e** (7.1 mg, 8%) as a pale yellow solid: *Syn*-isomer: mp 99-100 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.24 (m, 2H), 7.56 (m, 2H), 7.33-7.19 (m, 5H), 4.27-4.16 (m, 2H), 4.24 (d, J = 3.5 Hz, 1H), 4.09 (d, J = 3.5 Hz, 1H), 3.78 (d, J_{AB} = 13.4 Hz, 1H), 3.45 (d, J_{AB} = 13.4 Hz, 1H), 1.21 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 172.8, 147.6, 147.2, 139.0, 128.9, 128.4, 128.2, 127.3, 123.7, 74.2, 62.6, 62.2, 50.4, 14.0; IR (film) ν_{max} = 3477 (br), 2981, 1731, 1521, 1347, 1098 cm^{-1}; HRMS (FAB+) calcd for $C_{18}H_{21}N_2O_5$ (M+H): 345.1450, found: 345.1443.

(2*R,3*S**)-Ethyl 3-(benzylamino)-3-(4-chlorophenyl)-2-hydroxypropanoate (5f)**



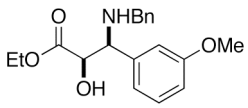
The general procedure was followed using **1f** (58.8 mg, 0.256 mmol). D.r. 98:2 (*syn*:*anti*), ^{1}H NMR spectroscopic analysis on the crude product. Flash chromatography (heptane:EtOAc 4:1) of the residue gave *syn*-**5f** (64.3 mg, 75%) as a white solid: mp 71-72 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.42-7.26 (m, 9H), 4.29-4.15 (m, 2H), 4.27 (d, J = 4.0 Hz, 1H), 3.98 (d, J = 4.0 Hz, 1H), 3.81 (d, J_{AB} = 13.4 Hz, 1H), 3.51 (d, J_{AB} = 13.4 Hz, 1H), 1.23 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 173.2, 139.3, 137.8, 133.6, 129.3, 128.7, 128.31, 128.25, 127.1, 74.6, 62.7, 61.9, 50.4, 14.0; IR (film) ν_{max} = 3468 (br), 3028, 2982, 1731, 1493, 1210, 1092 cm^{-1}; HRMS (FAB+) calcd for $C_{18}H_{21}ClNO_3$ (M+H): 334.1210, found: 334.1205.

(2*R,3*S**)-Ethyl 3-(benzylamino)-3-(4-fluorophenyl)-2-hydroxypropanoate (5g)**



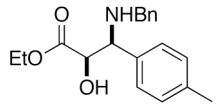
The general procedure was followed using **1g** (54.6 mg, 0.256 mmol). D.r. 97:3 (*syn*:*anti*), ^{1}H NMR spectroscopic analysis on the crude product. Flash chromatography (heptane:EtOAc 4:1) of the residue gave *syn*-**5g** (63.4 mg, 78%) as a white solid: mp 57-60 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.36-7.22 (m, 7H), 7.07 (m, 2H), 4.23 (d, J = 4.2 Hz, 1H), 4.21-4.09 (m, 2H), 3.93 (d, J = 4.2 Hz, 1H), 3.77 (d, J_{AB} = 13.4 Hz, 1H), 3.45 (d, J_{AB} = 13.4 Hz, 1H), 1.16 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 173.2, 163.7, 161.2, 139.2, 134.7, 129.6, 129.5, 128.4, 128.3, 127.2, 115.5, 115.3, 74.7, 62.7, 61.9, 50.4, 14.0; IR (film) ν_{max} = 3388 (br), 2983, 1733, 1510, 1222, 1105 cm^{-1}; HRMS (FAB+) calcd for $C_{18}H_{21}FNO_3$ (M+H): 318.1505, found: 318.1507.

(2*R,3*S**)-Ethyl 3-(benzylamino)-2-hydroxy-3-(3-methoxyphenyl)propanoate (5h)**



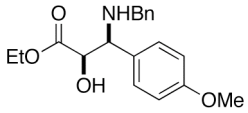
The general procedure was followed using **1h** (57.7 mg, 0.256 mmol). D.r. 98:2 (*syn*:*anti*), ^{1}H NMR spectroscopic analysis on the crude product. Flash chromatography (heptane:EtOAc 4:1 → 2:1) of the residue gave *syn*-**5h** (64.6 mg, 77%) as a white solid: mp 47-48 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.32-7.22 (m, 6H), 6.94 (m, 2H), 6.86 (m, 1H), 4.25 (d, J = 4.0 Hz, 1H), 4.21-4.12 (m, 2H), 3.93 (d, J = 4.0 Hz, 1H), 3.83 (s, 3H), 3.78 (d, J_{AB} = 13.3 Hz, 1H), 3.51 (d, J_{AB} = 13.3 Hz, 1H), 1.17 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 173.4, 159.8, 141.0, 139.7, 129.5, 128.3, 127.1, 120.1, 113.4, 113.1, 74.8, 63.5, 61.8, 55.3, 50.6, 14.0; IR (film) ν_{max} = 3449 (br), 3027, 2937, 2836, 1732, 1600, 1454, 1256, 1101 cm^{-1}; HRMS (FAB+) calcd for $C_{19}H_{24}NO_4$ (M+H): 330.1705, found: 330.1710.

(2*R,3*S**)-Ethyl 3-(benzylamino)-2-hydroxy-3-*p*-tolylpropanoate (5i)**



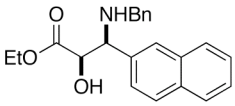
The general procedure was followed using **1i** (53.6 mg, 0.256 mmol). D.r. 97:3 (*syn*:*anti*), ^{1}H NMR spectroscopic analysis on the crude product. Flash chromatography (heptane:EtOAc 4:1) of the residue gave *syn*-**5i** (69.8 mg, 87%) as a white solid: mp 84-86 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.32-7.18 (m, 9H), 4.22 (d, J = 4 Hz, 1H), 4.17 (m, 2H), 3.93 (d, J = 4.0 Hz, 1H), 3.72 (d, J_{AB} = 13.3 Hz, 1H), 3.50 (d, J_{AB} = 13.3 Hz, 1H), 2.37 (s, 3H), 1.17 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 173.4, 139.9, 137.4, 136.3, 129.2, 128.2, 127.6, 127.0, 74.9, 63.2, 61.7, 50.6, 21.1, 14.0; IR (film) ν_{max} = 3436 (br), 3026, 2981, 2924, 1731, 1453, 1099 cm^{-1}; HRMS (FAB+) calcd for $C_{19}H_{24}NO_3$ (M+H): 314.1756, found: 314.1748.

(2*R,3*S**)-Ethyl 3-(benzylamino)-2-hydroxy-3-(4-methoxyphenyl)propanoate (5j)**



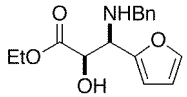
The general procedure was followed using **1j** (57.7 mg, 0.256 mmol). D.r. >94:6 (*syn*:*anti*), ^{1}H NMR spectroscopic analysis on the crude product. Addition of ethyl diazoacetate over 10h at rt. Flash chromatography (heptane:EtOAc 4:1 → 2:1) of the residue gave *syn*-**5j** (65.6 mg, 78%) as a white solid and *anti*-**5j** (3.3 mg, 4%) as a white solid: *Syn*-isomer: mp 88-90 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.33-7.23 (m, 7H), 6.92 (m, 2H), 4.21 (d, J = 4.2 Hz, 1H), 4.21-4.12 (m, 2H), 3.90 (d, J = 4.2 Hz, 1H), 3.83 (s, 3H), 3.75 (d, J_{AB} = 13.3 Hz, 1H), 3.49 (d, J_{AB} = 13.3 Hz, 1H), 1.17 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 173.5, 159.2, 140.0, 131.5, 128.8, 128.23, 128.21, 126.9, 113.9, 75.0, 62.9, 61.6, 55.2, 50.5, 14.0; IR (film) ν_{max} = 3449 (br), 2934, 2837, 1732, 1513, 1248, 1031 cm^{-1}; HRMS (FAB+) calcd for $C_{19}H_{24}NO_4$ (M+H): 330.1705, found: 330.1711.

(2*R,3*S**)-Ethyl 3-(benzylamino)-2-hydroxy-3-(naphthalen-3-yl)propanoate (5k)**



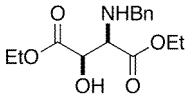
The general procedure was followed using **1k** (62.8 mg, 0.256 mmol). D.r. 98:2 (*syn*:*anti*), ^{1}H NMR spectroscopic analysis on the crude product. Flash chromatography (heptane:EtOAc 4:1) of the residue gave *syn*-**5k** (74.0 mg, 83%) as a white solid: mp 91-92 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.89-7.78 (m, 4H), 7.56 (dd, J = 8.5, 1.6 Hz, 1H), 7.50 (m, 2H), 7.33-7.24 (m, 5H), 4.36 (d, J = 4.2 Hz, 1H), 4.20-4.11 (m, 2H), 4.13 (d, J = 4.2 Hz, 1H), 3.82 (d, J_{AB} = 13.4 Hz, 1H), 3.54 (d, J_{AB} = 13.4 Hz, 1H), 1.11 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 173.4, 139.6, 136.8, 133.3, 133.2, 128.4, 128.3, 127.9, 127.7, 127.1, 126.2, 126.0, 125.5, 74.9, 63.6, 61.8, 50.5, 14.0; IR (film) ν_{max} = 3475 (br), 3057, 2981, 1732, 1453, 1097 cm^{-1}; HRMS (FAB+) calcd for $C_{22}H_{24}NO_3$ (M+H) 350.1756, found: 350.1758.

(2*R,3*R**)-Ethyl 3-(benzylamino)-3-(furan-2-yl)-2-hydroxypropanoate (5l)**



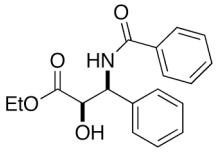
The general procedure was followed using **1l** (46.6 mg, 0.252 mmol). Addition of ethyl diazoacetate over 10h at 0° C. D.r. 92:8 (*syn*:*anti*), ^{1}H NMR spectroscopic analysis on the crude product. Flash chromatography (heptane:EtOAc 4:1) of the residue gave *syn*-**5l** (54.3 mg, 75%) as a pale orange oil and *anti*-**5l** (4.7 mg, 6%) as a pale orange oil: *Syn*-isomer: ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.41 (dd, J = 1.9, 0.9 Hz, 1H), 7.33-7.20 (m, 5H), 6.36 (dd, J = 3.2, 1.8 Hz, 1H), 6.30 (m, 1H), 4.36 (d, J = 3.8 Hz, 1H), 4.24-4.09 (m, 2H), 4.17 (m, 1H), 4.03 (d, J = 3.8 Hz, 1H), 3.84 (d, J_{AB} = 13.3 Hz, 1H), 3.58 (d, J_{AB} = 13.3 Hz, 1H), 1.18 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 172.9, 153.3, 142.1, 139.6, 128.3, 128.2, 127.0, 110.2, 107.8, 73.0, 61.9, 57.1, 50.8, 14.0; IR (film) ν_{max} = 3348 (br), 2981, 2929, 1738, 1454, 1101 cm^{-1}; HRMS (FAB+) calcd for $C_{16}H_{20}NO_4$ (M+H) 290.1392, found: 290.1385.

(2*R,3*R**)-Diethyl 2-(benzylamino)-3-hydroxysuccinate (5m)**



The general procedure was followed using **1m** (49.0 mg, 0.256 mmol). Addition of ethyl diazoacetate over 10h at 0° C. D.r. 83:17 (*syn*:*anti*), ^{1}H NMR spectroscopic analysis on the crude product. Flash chromatography (heptane:EtOAc 4:1 → 2:1) of the residue gave *syn*-**5m** (48.5 mg, 64%) as a colorless oil and *anti*-**5m** (9.5 mg, 13%) as a colorless oil: *Syn*-isomer: ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.36-7.22 (m, 5H), 4.54 (d, J = 2.6 Hz, 1H), 4.26 (m, 3H), 4.15 (qd, J = 10.7, 7.1 Hz, 1H), 3.95 (d, J_{AB} = 13.3 Hz, 1H), 3.67 (d, J = 2.6 Hz, 1H), 3.66 (d, J_{AB} = 13.3 Hz, 1H), 1.31 (t, J = 7.2 Hz, 3H), 1.22 (t, J = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 172.5, 171.8, 139.6, 128.32, 128.26, 127.1, 72.2, 62.1, 62.0, 61.5, 52.2, 14.2, 14.0; IR (film) ν_{max} = 3520, 3054, 2987, 1738, 1421, 1265 cm^{-1}; HRMS (FAB+) calcd for $C_{15}H_{22}NO_5$ (M+H) 296.1498, found: 296.1498. *Anti*-isomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ = 7.37-7.22 (m, 5H), 4.49 (dd, J = 6.5, 3.4 Hz, 1H), 4.21 (m, 4H), 3.96 (d, J_{AB} = 13.0 Hz, 1H), 3.75 (d, J_{AB} = 13.0 Hz), 3.68 (d, J = 3.5 Hz, 1H), 3.36 (d, J = 7.6 Hz, 1H), 2.28 (br s, 1H), 1.28 (t, J = 7.1 Hz, 3H), 1.27 (t, J = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ = 171.9, 171.0, 139.4, 128.5, 128.3, 127.3, 71.8, 63.2, 61.8, 61.4, 52.6, 14.2, 14.1; IR (film) ν_{max} = 3498 (br), 3059, 2985, 1739, 1454, 1267, 1201, 1027 cm^{-1}; HRMS (FAB+) calcd for $C_{15}H_{22}NO_5$ (M+H) 296.1498, found: 296.1502.

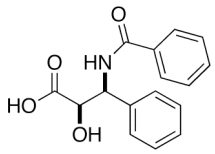
(2*R*,3*S*)-Ethyl 3-(benzamido)-2-hydroxy-3-phenylpropanoate (17)



To a solution of amine **16a** (13.6 mg, 0.043 mmol) in EtOH (1.5 mL) and HCl (150 μL, 3M in EtOH) was added a catalytic amount of $Pd(OH)_2$ (20% on C). The resultant mixture was stirred under H_2-atmosphere (1 atm) for 15h, filtered through a pad of florisil and evaporated under reduced pressure. The residue was dissolved in EtOAc:$NaHCO_3$ (4 mL, 3:4) and to the solution was added benzoylchloride (7.6 μL, 0.065 mmol) at 0 °C. The resultant two-phase solution was stirred at 0 °C for 15 min and the diluted with EtOAc. The phases were separated and the organic phase was washed with brine, dried ($MgSO_4$) and evaporated under reduced pressure. Flash chromatography (heptane:EtOAc 2:1 → 1:1) of the residue gave **17** (10.5 mg, 77%) as a white solid: mp 163-165 °C (lit.[1] mp 164-165 °C); $[\alpha]^{20}_D$ = -18.1

(*c* 0.7, $CHCl_3$) [lit.[1] $[\alpha]^{20}_D$ = -21.7 (*c* 1.0, $CHCl_3$)]; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.77 (m, 2H), 7.52-7.26 (m, 8H), 6.98 (d, *J* = 9.2 Hz, 1H), 5.77 (dd, *J* = 9.2, 1.8 Hz, 1H), 4.63 (dd, *J* = 3.8, 2.2 Hz, 2H), 4.35-4.26 (m, 2H), 3.26 (d, *J* = 3.8 Hz, 1H), 1.31 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 172.9, 166.8, 138.7, 134.1, 131.7, 128.7, 128.6, 127.9, 127.0, 126.9, 73.3, 62.8, 54.7, 14.1; IR (film) ν_{max} = 3414 (br), 3351, 1719, 1637, 1527 cm^{-1}.

(2*R*,3*S*)-3-(Benzamido)-2-hydroxy-3-phenylpropanoic acid (18)



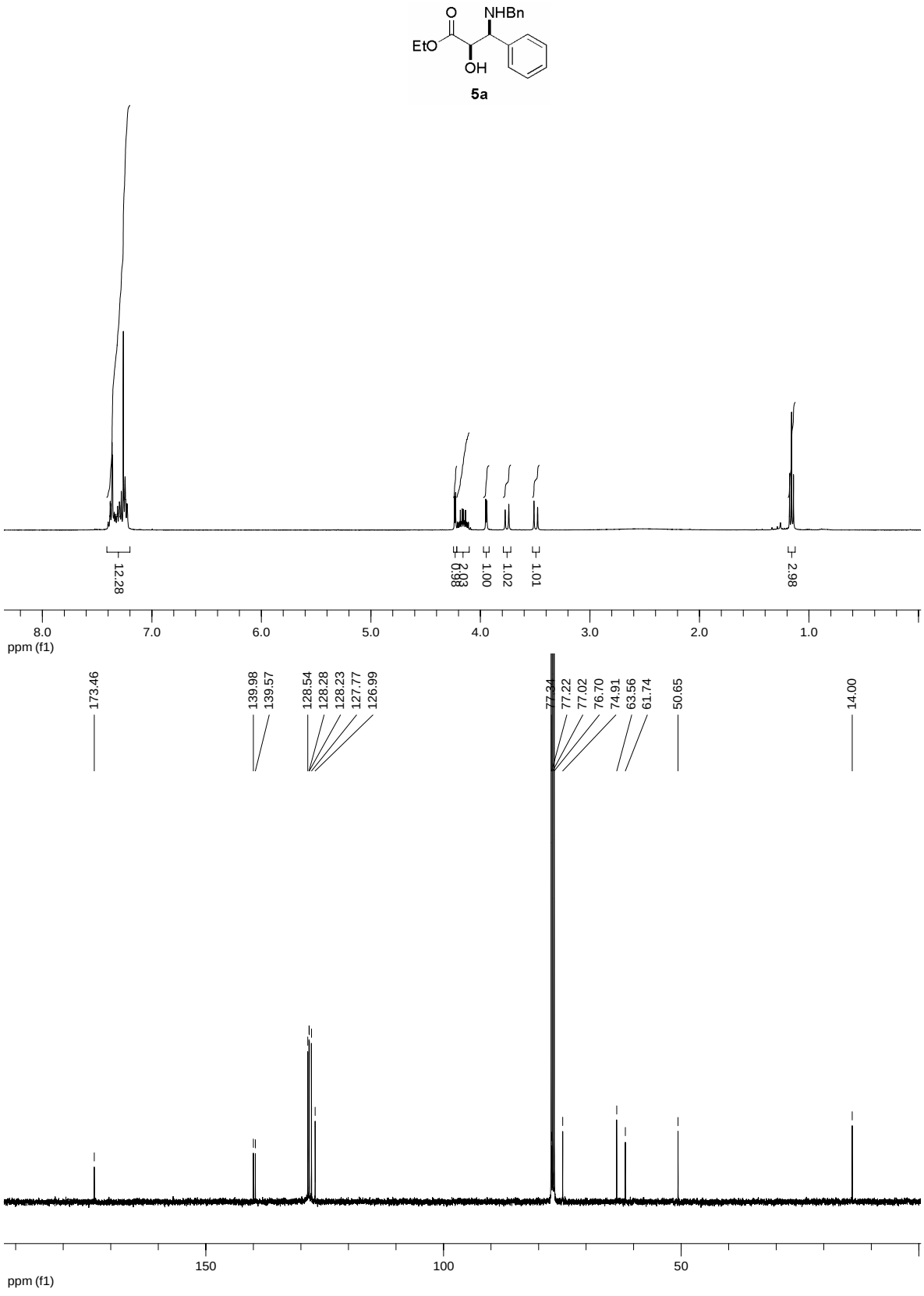
To a solution of ethyl ester **17** (8.9 mg, 0.0284mmol) in THF:MeOH:H_2O (1.1 mL, 10:5:4) was added LiOH·H_2O (2.4 mg, 0.0568 mmol). The resultant mixture was stirred at room temperature for 1h, diluted with EtOAc (1.5 mL). The phase where separated and the aqueous layer was extracted three times with EtOAc. The combined organic extracts where dried ($MgSO_4$) and concentrated under reduced pressure. Recrystallization of the residue from $CHCl_3$ gave **18** (7.2 mg, 89%) as a white solid. mp 166-168 °C (lit.[2] mp 177-179 °C, lit.[3] 166-168 °C); $[\alpha]^{20}_D$ = -35.3 (*c* 0.45, EtOH) [lit.[2] $[\alpha]^{23}_D$ = -35.9 (*c* 0.565, EtOH), lit.[3] $[\alpha]^{20}_D$ = -37.8 (*c* 1, EtOH)]; ^{1}H NMR (400 MHz, MeOH-d_4) δ = 7.83 (m, 2H), 7.57-7.25 (m, 8H), 5.63, (m, 1H), 4.55 (d, *J* = 3.1 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ = 173.4, 165.9, 140.3, 134.4, 131.4, 128.4, 128.0, 127.3, 127.2, 126.9, 73.4, 55.8.

References:

[1] R. N. Patel, A. Banerjee, J. M. Howell, C. G. McNamee, D. Brozozowski, D. Mirfakhrae, V. Nanduri, J. K. Thottathil, L. J. Szarka, *Tetrahedron: Asymmetry* **1993**, *4*, 2069-2084.

[2] L. Deng, E. N. Jacobsen, *J. Org. Chem.* **1992**, *57*, 4320-4323.

[3] D.-M. Gou, Y.-C. Liu, C.-S. Chen, *J. Org. Chem.* **1993**, *58*, 1287-1289.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1.00
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1.01
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8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)
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139.98
139.57
128.54
128.28
128.23
127.77
126.99
77.34
77.22
77.02
76.70
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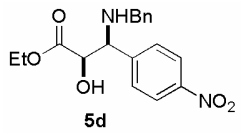
100
50
ppm (f1)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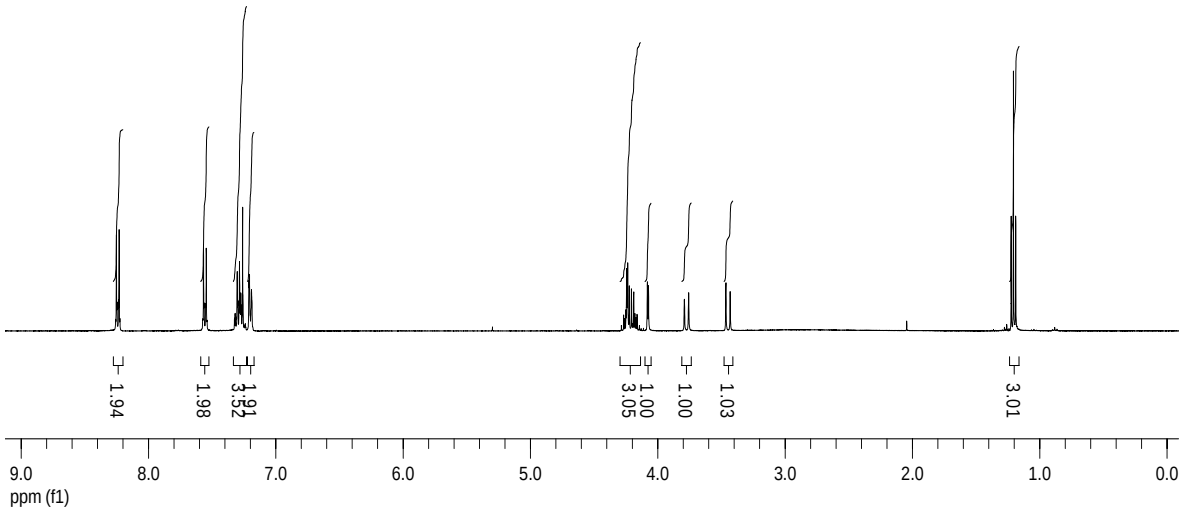
NO2

5d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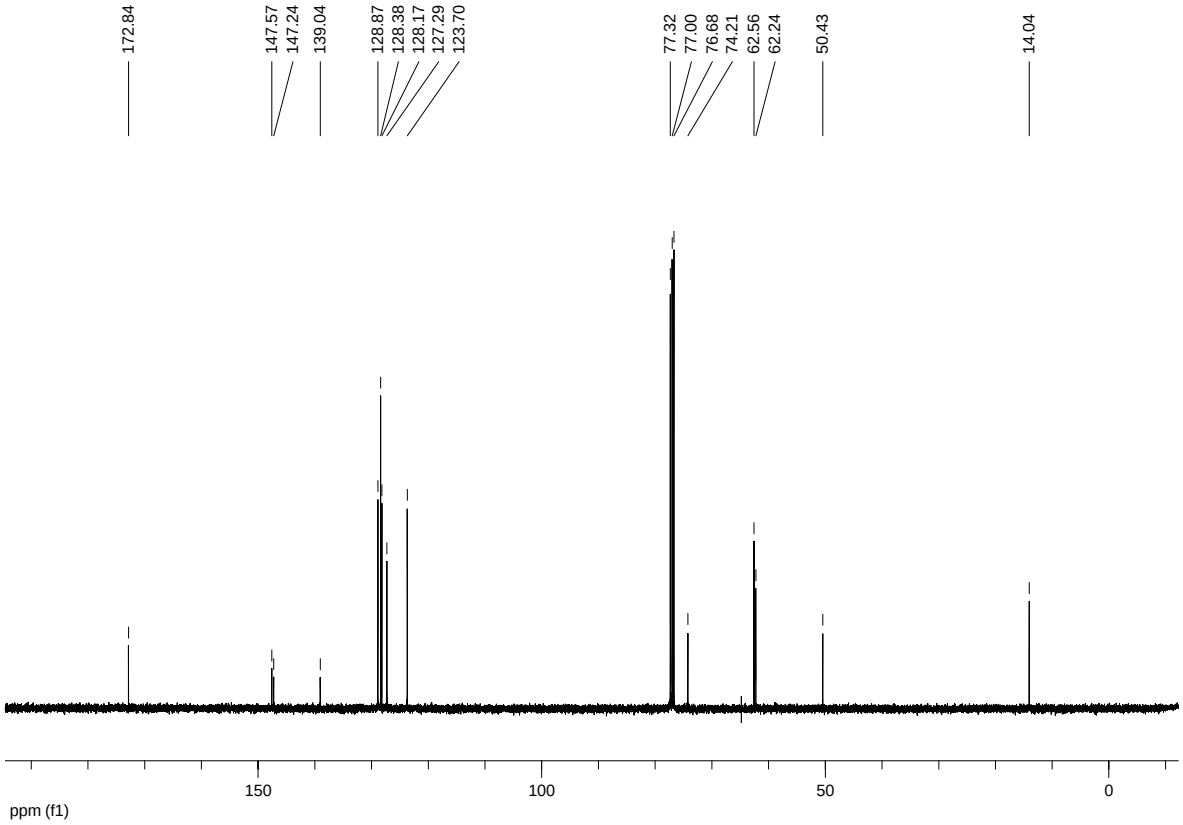
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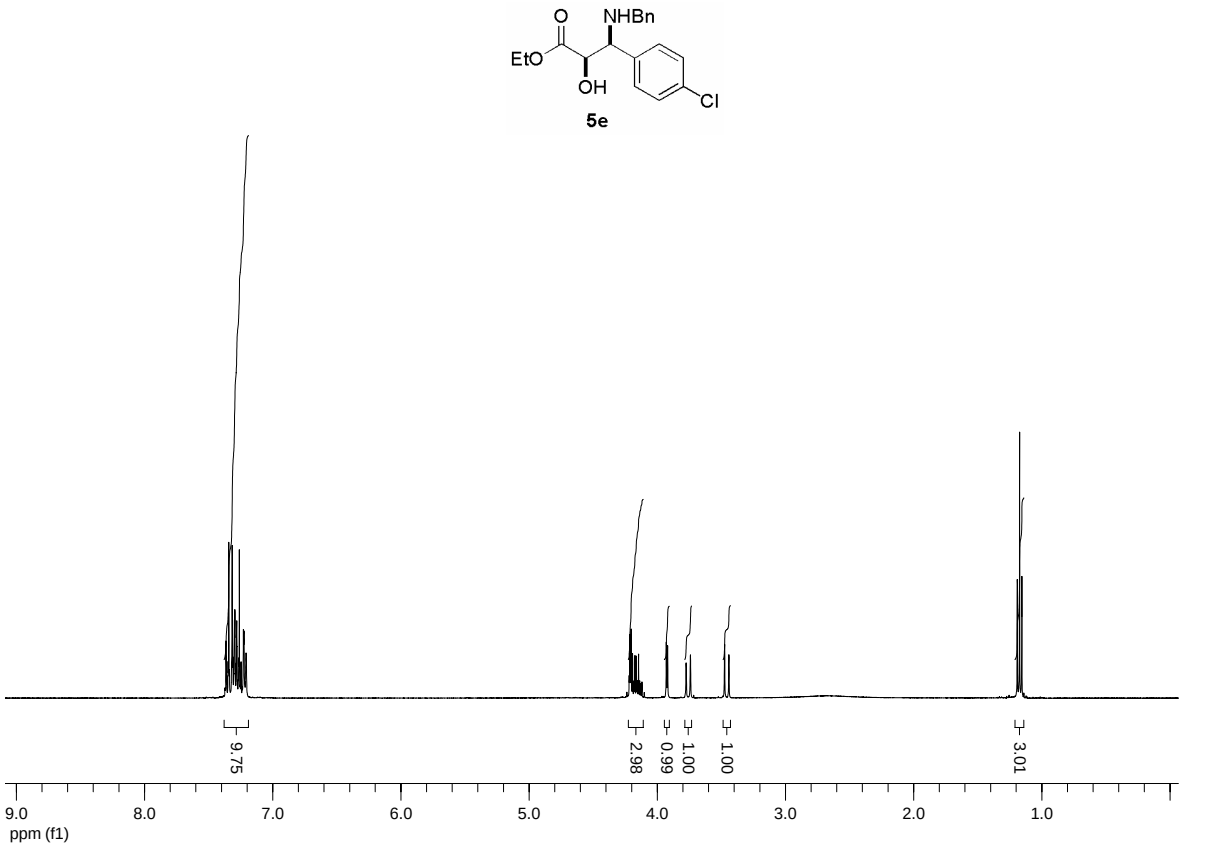
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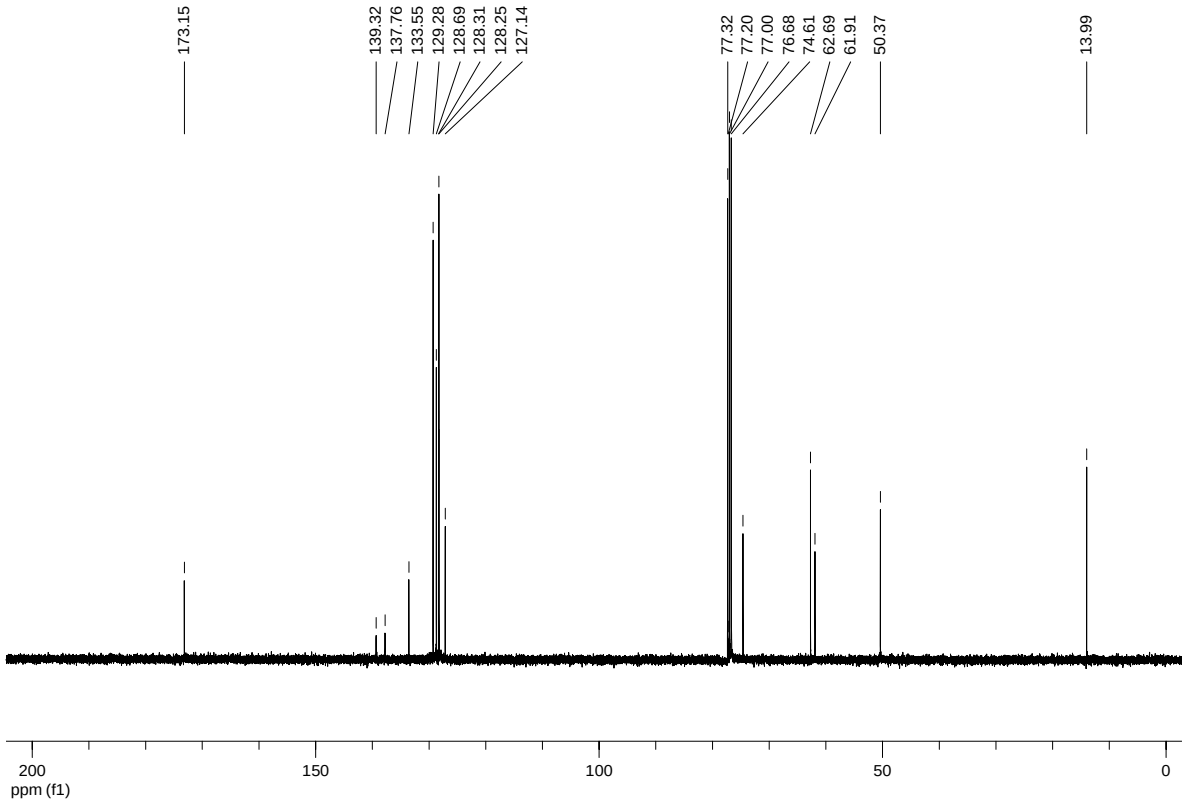
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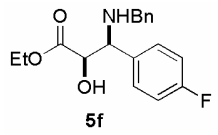
ppm (f1)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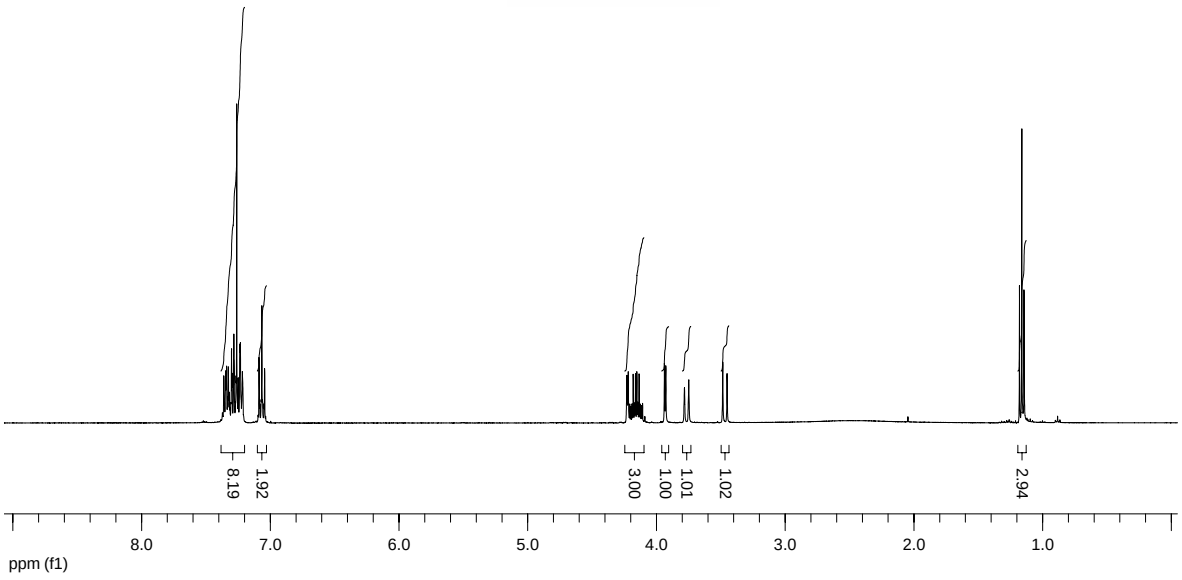
EtO
OH
F

5f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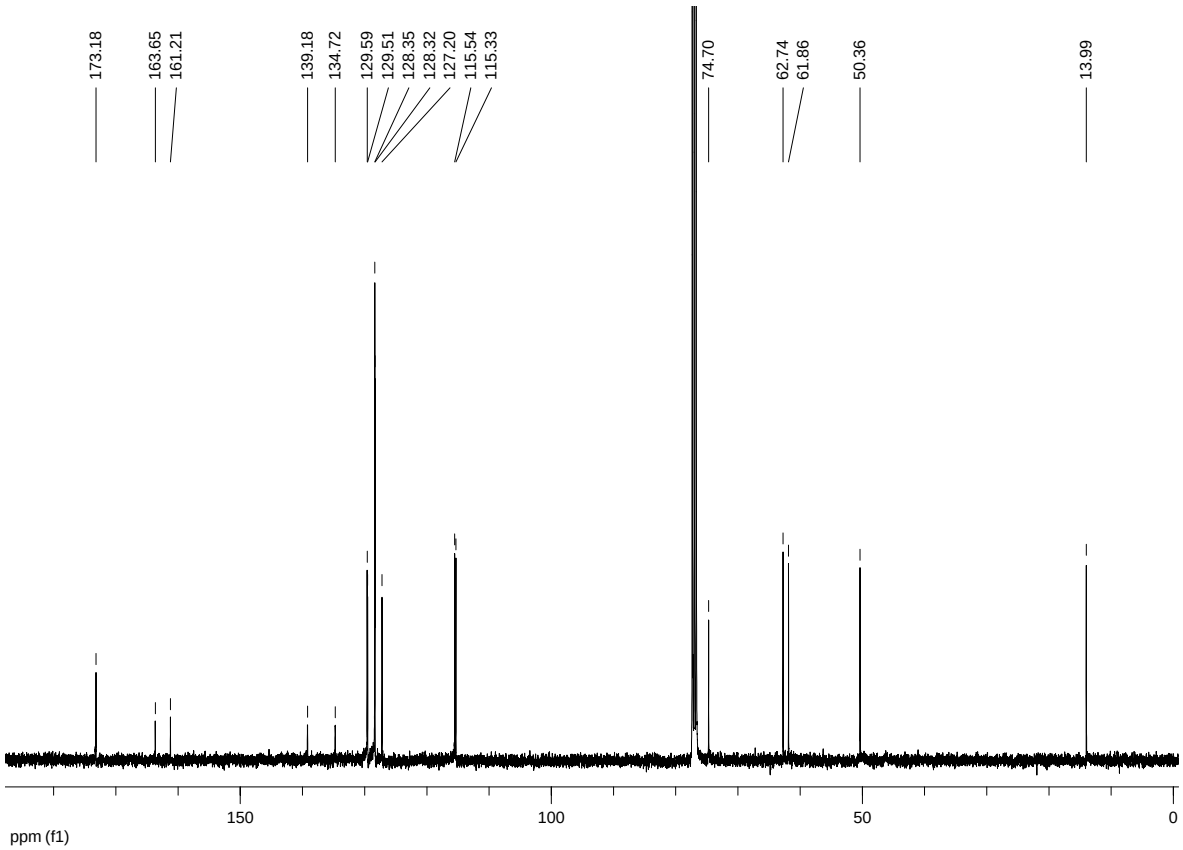
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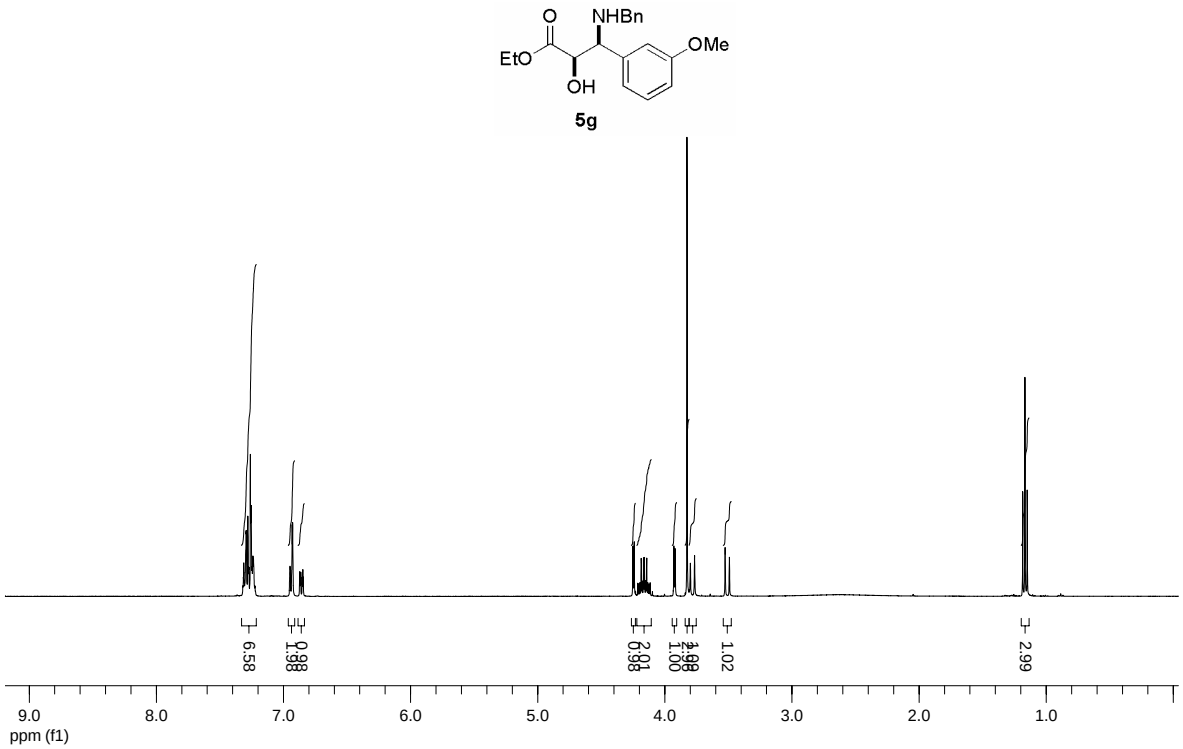
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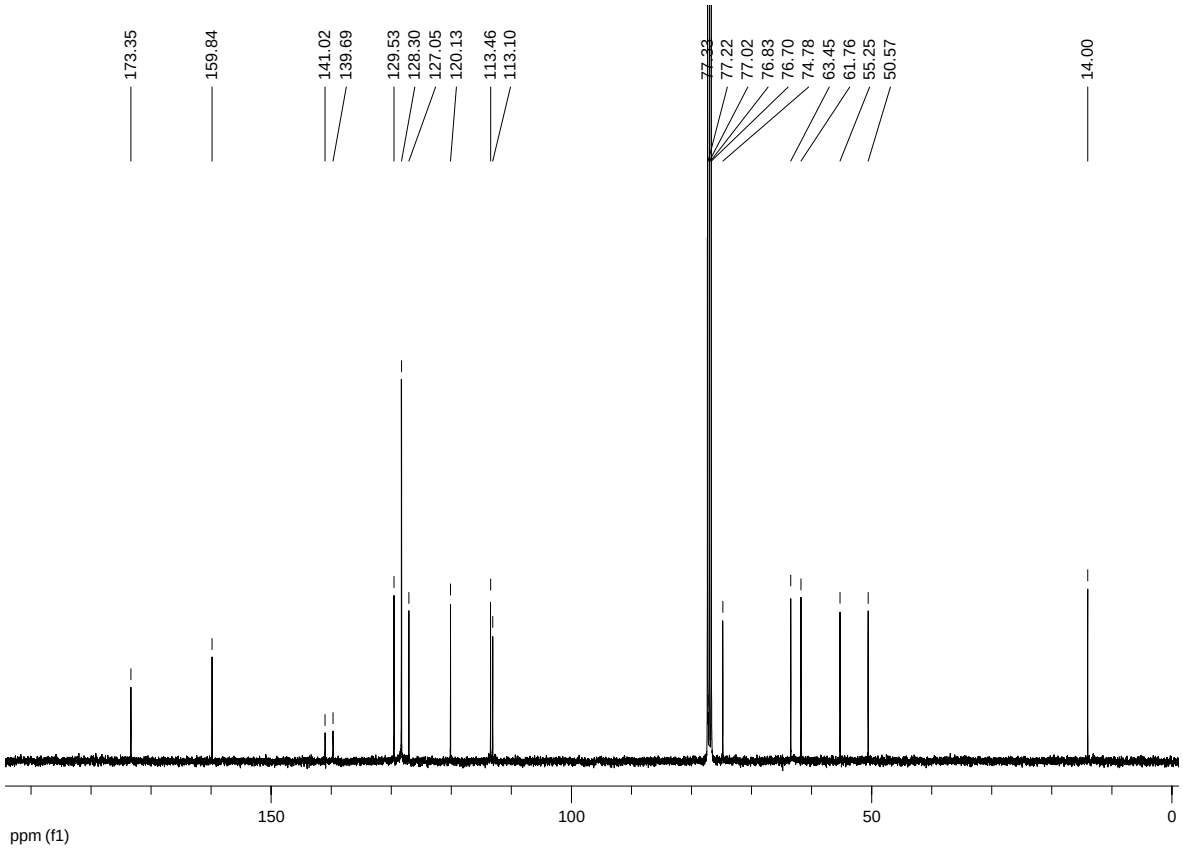
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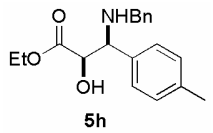
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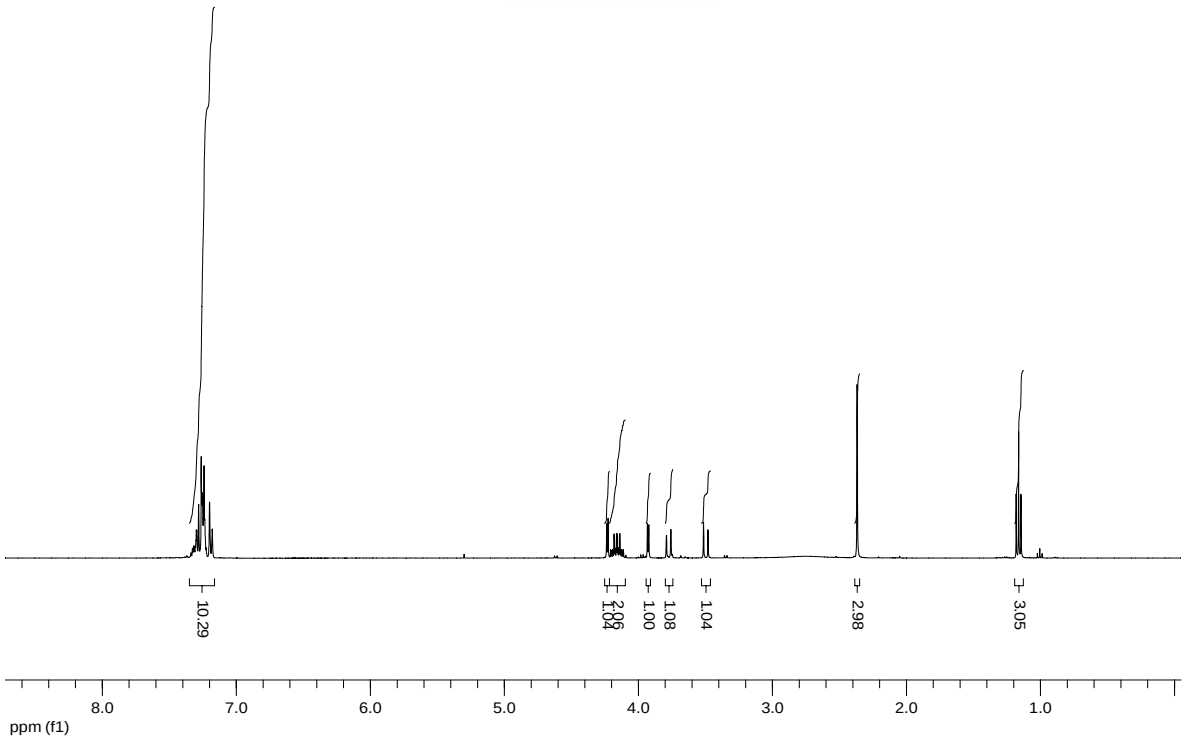
OH

5h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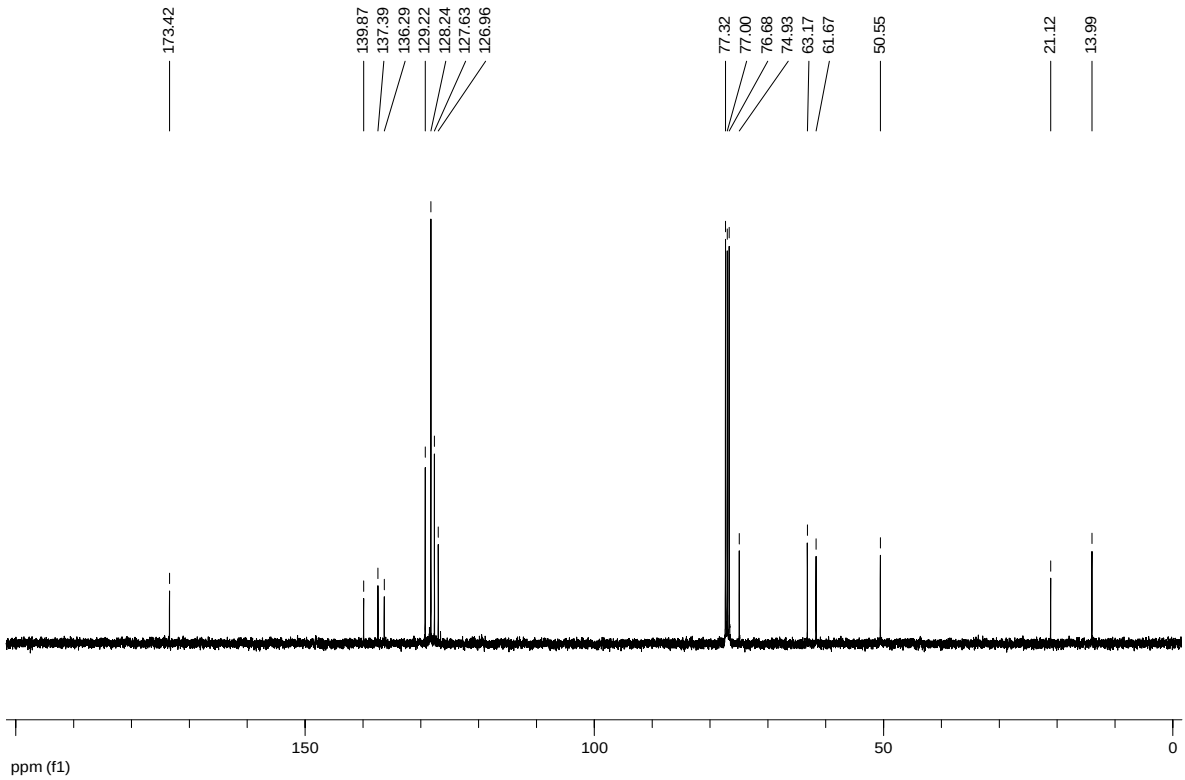
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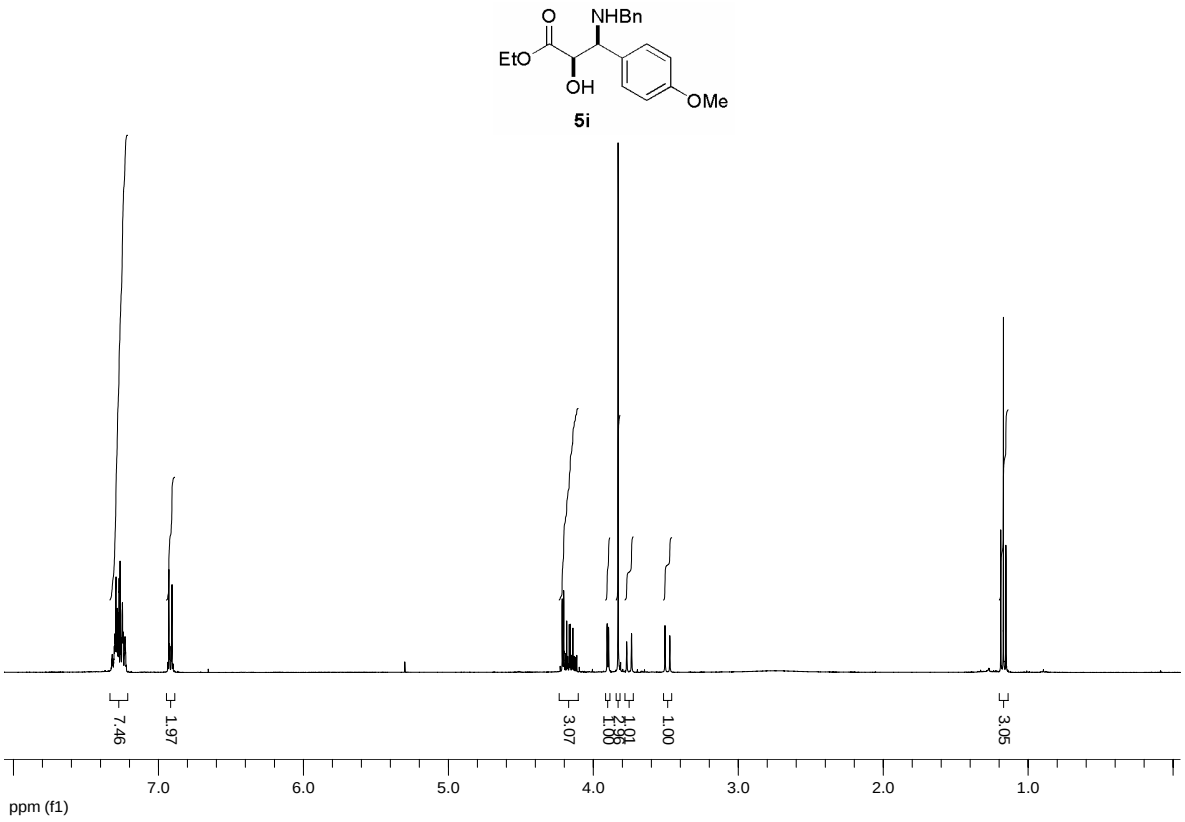
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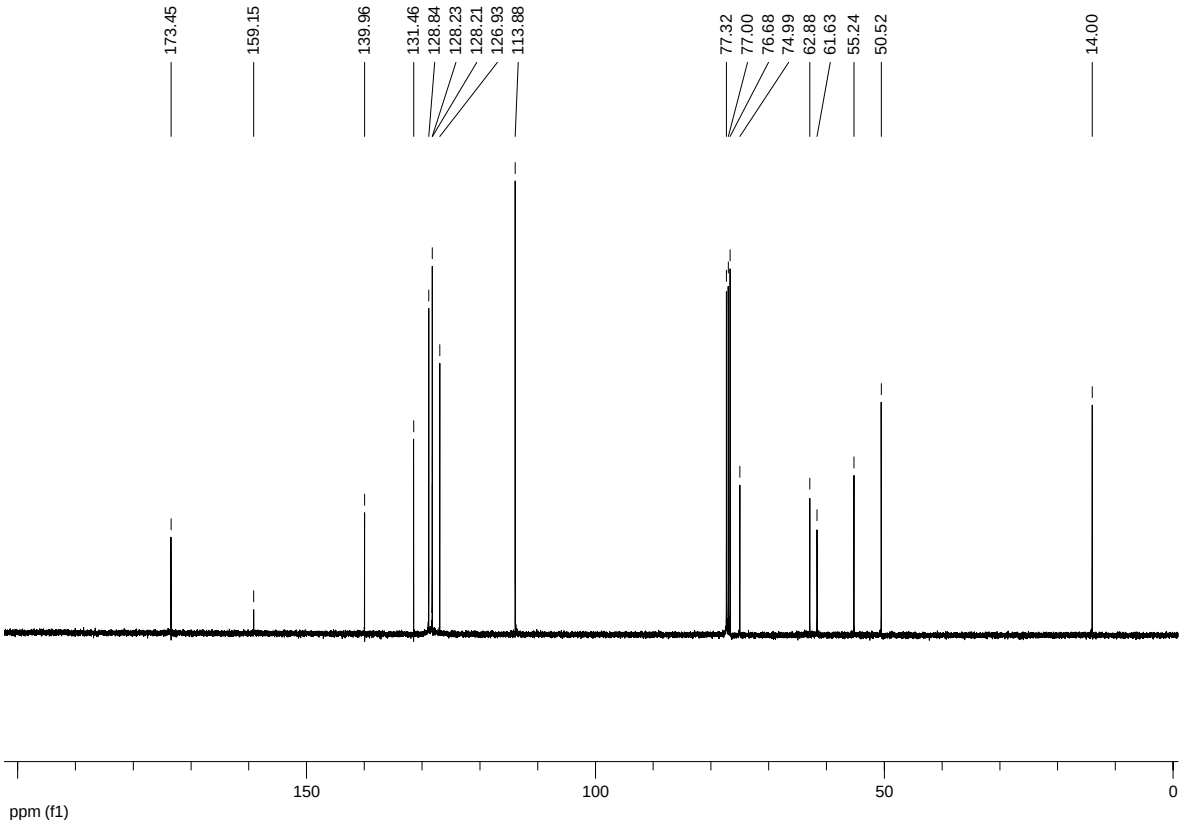
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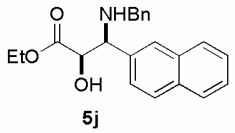
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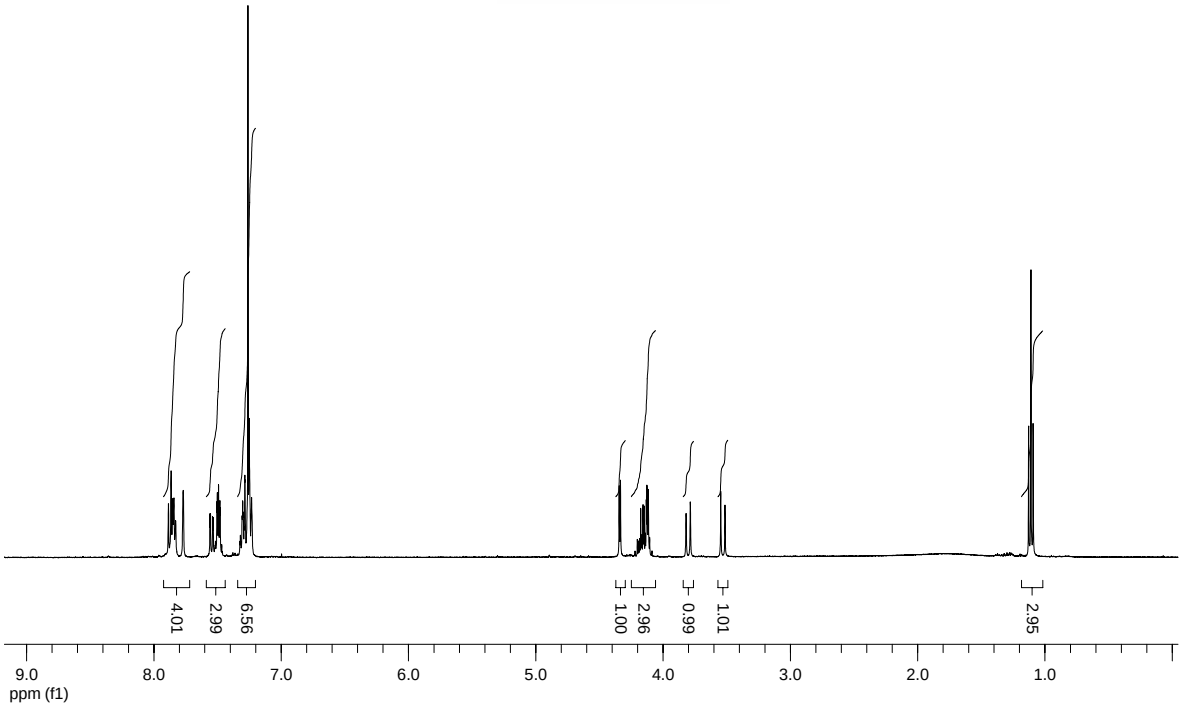
OH

5j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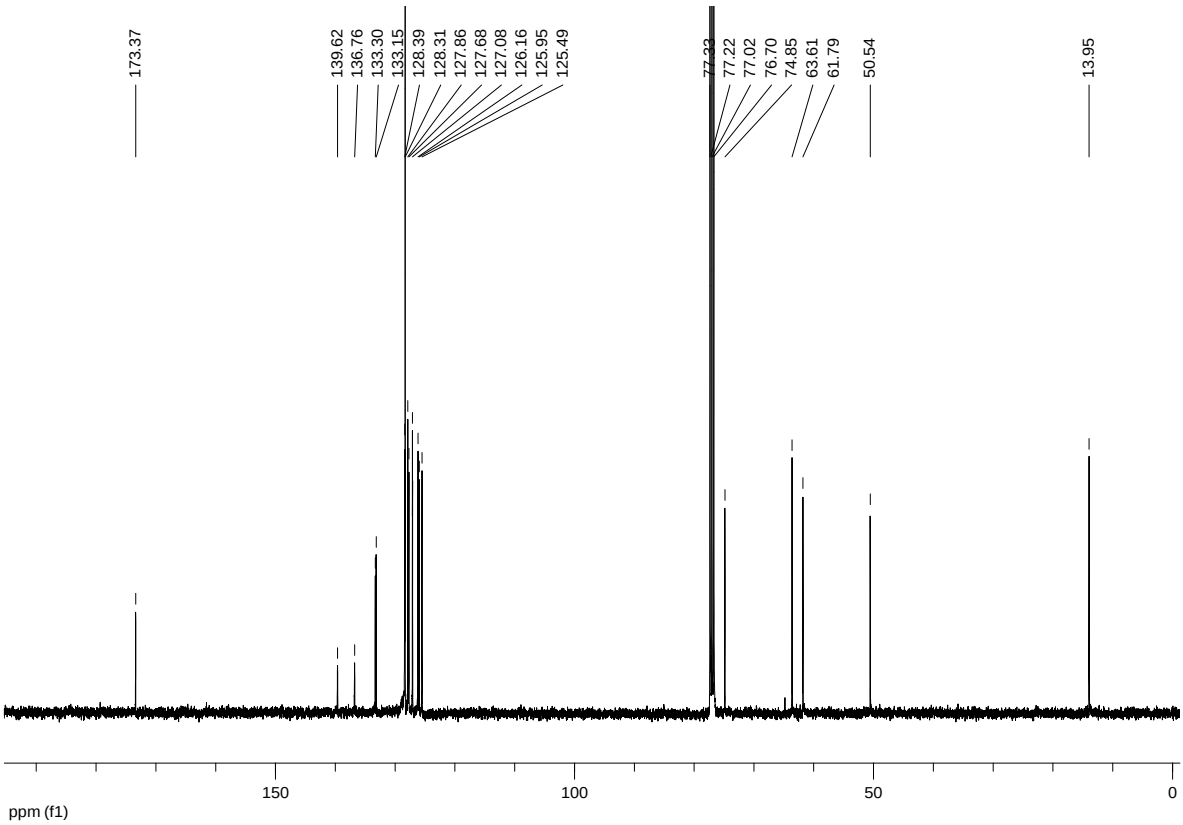
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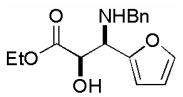
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EtO
O
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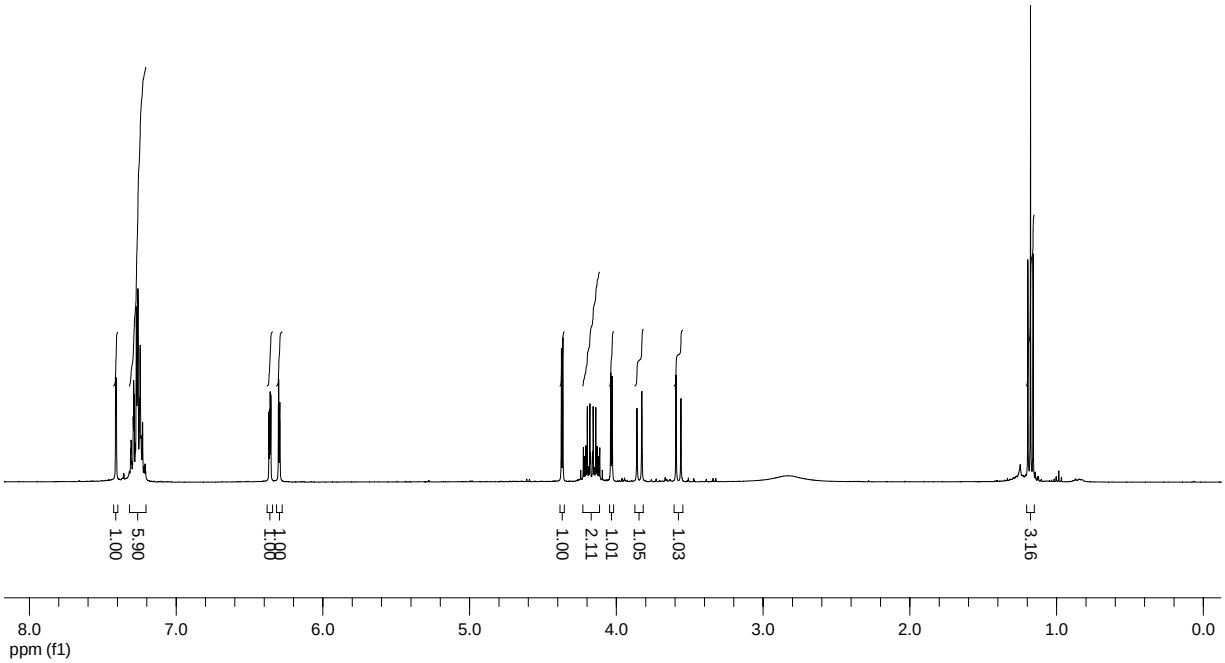
5k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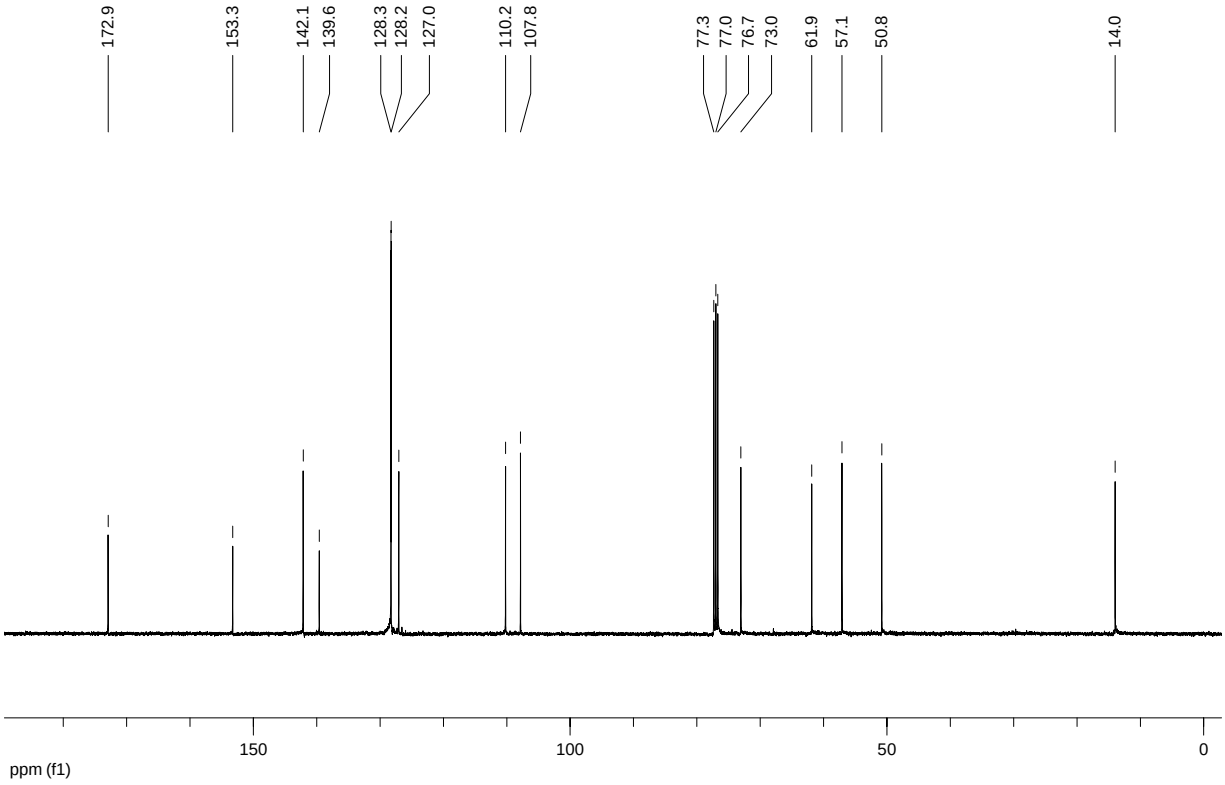
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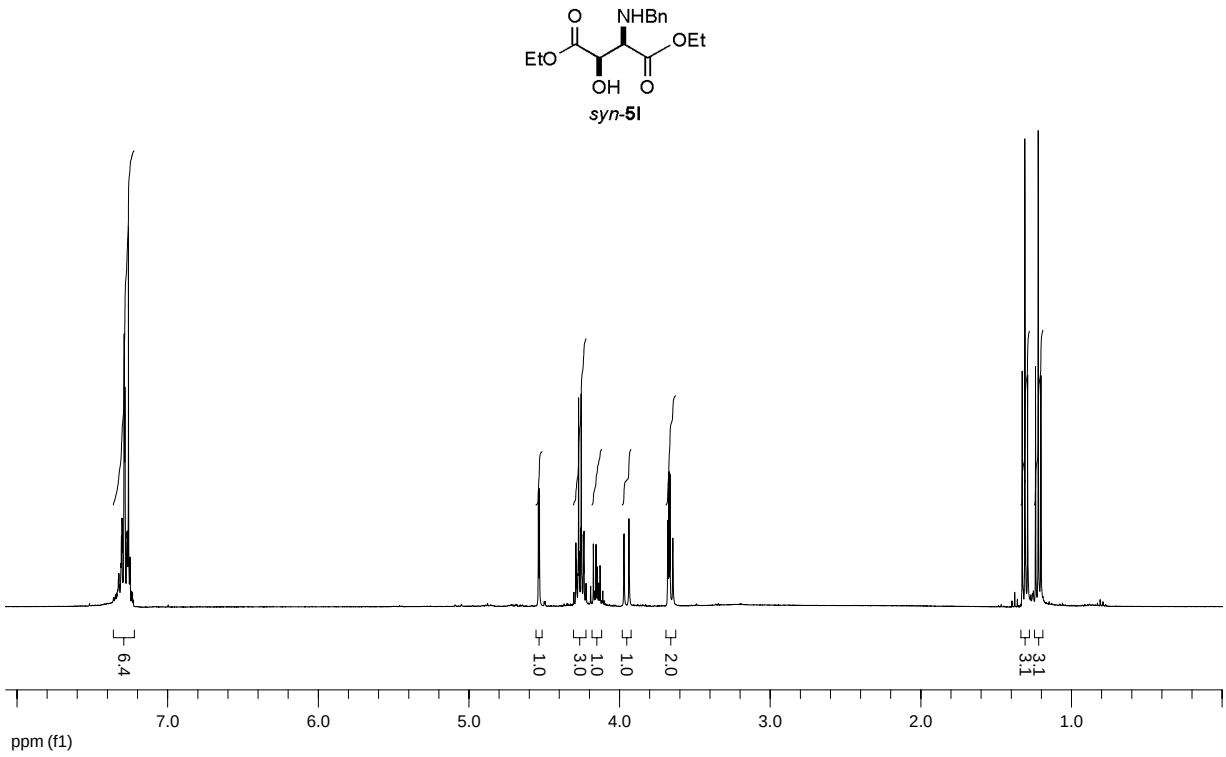
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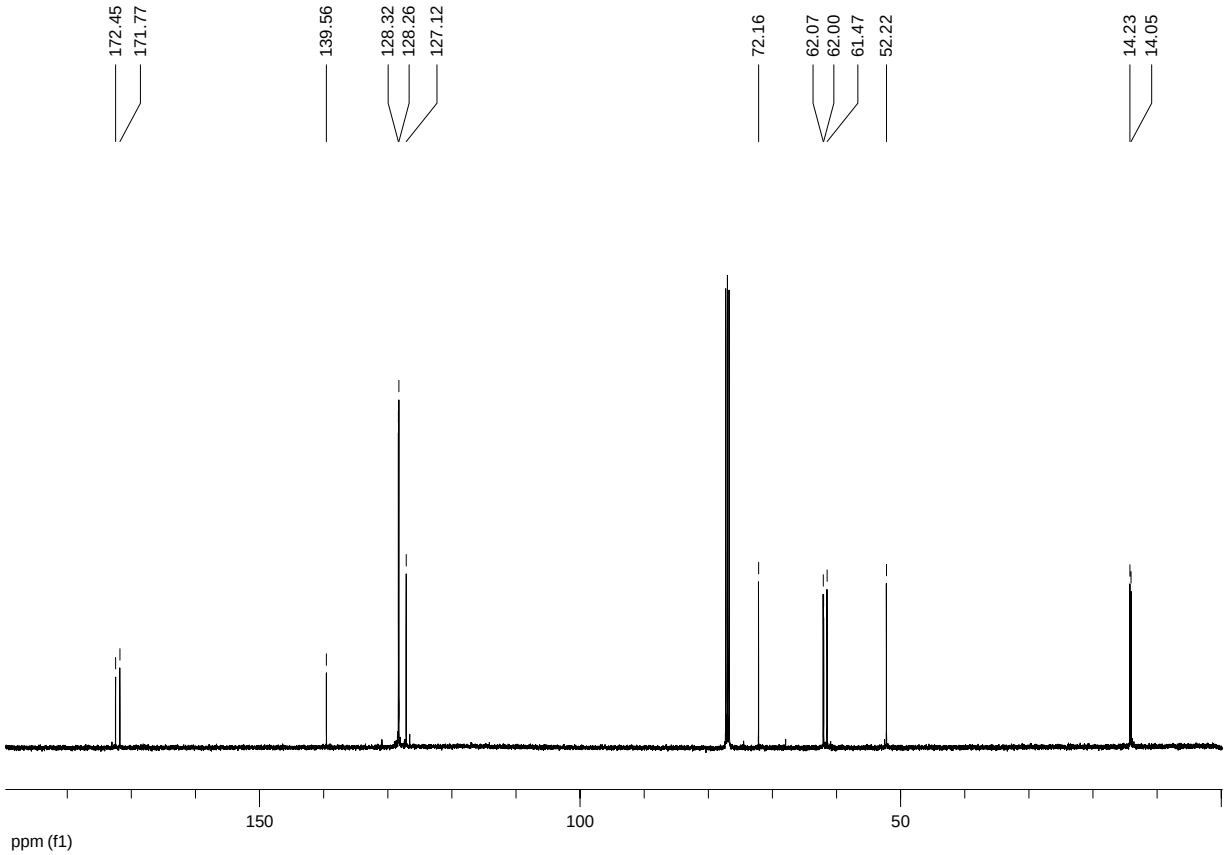
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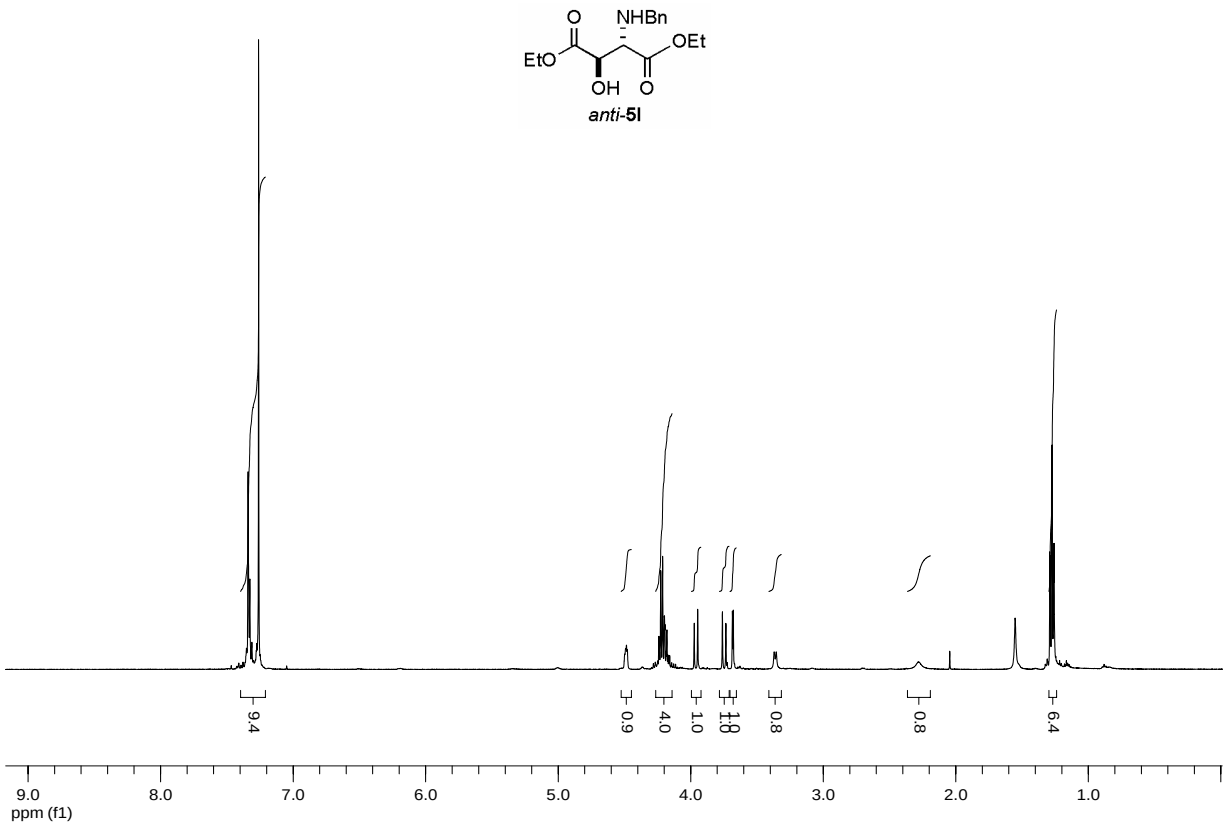
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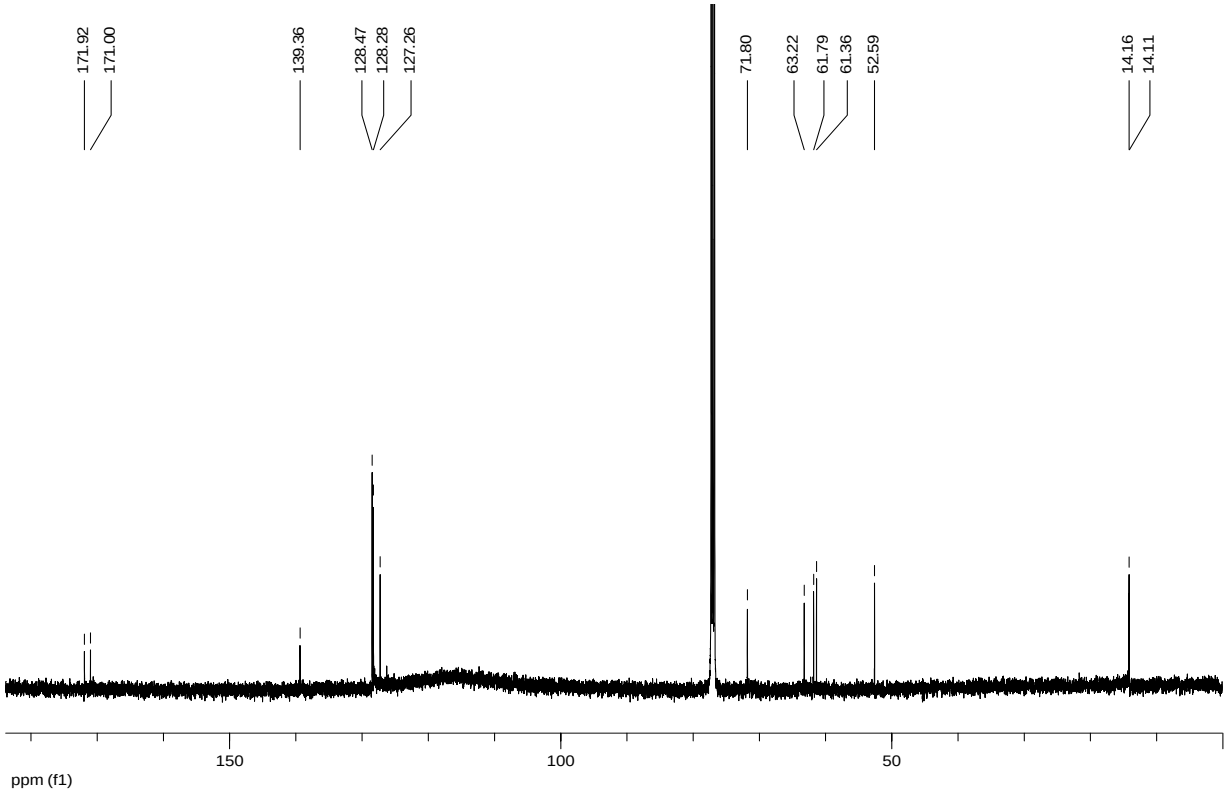
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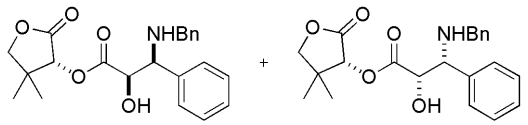
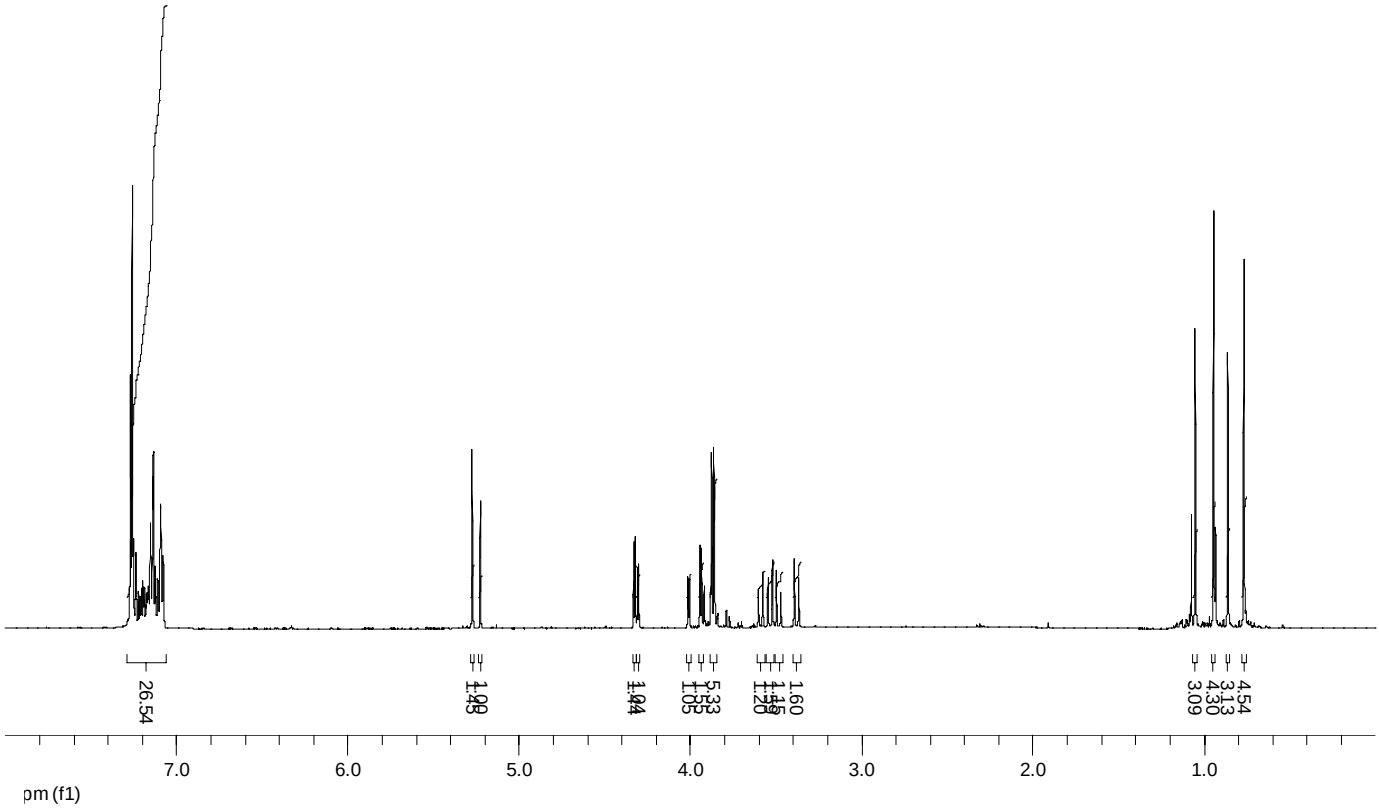


14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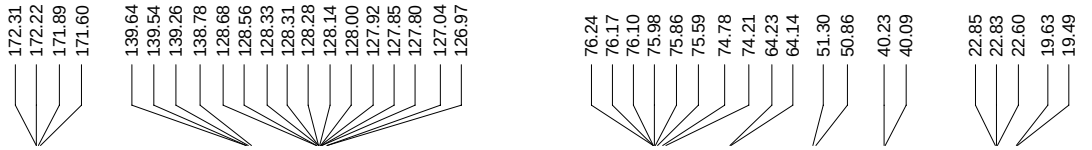
pm (f1)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127.80
127.04
126.97
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76.10
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75.86
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74.78
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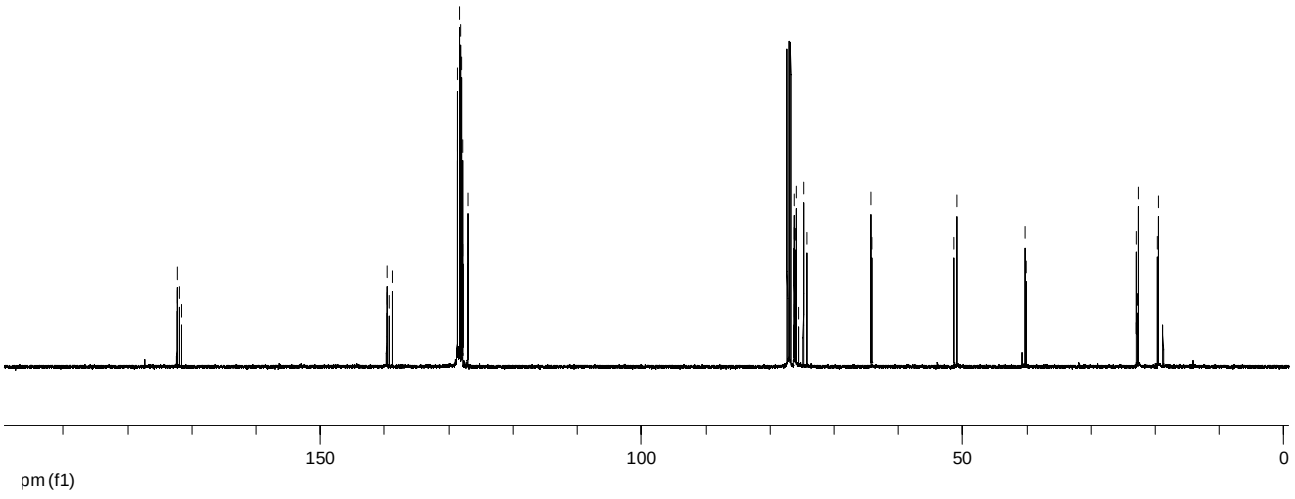
19.49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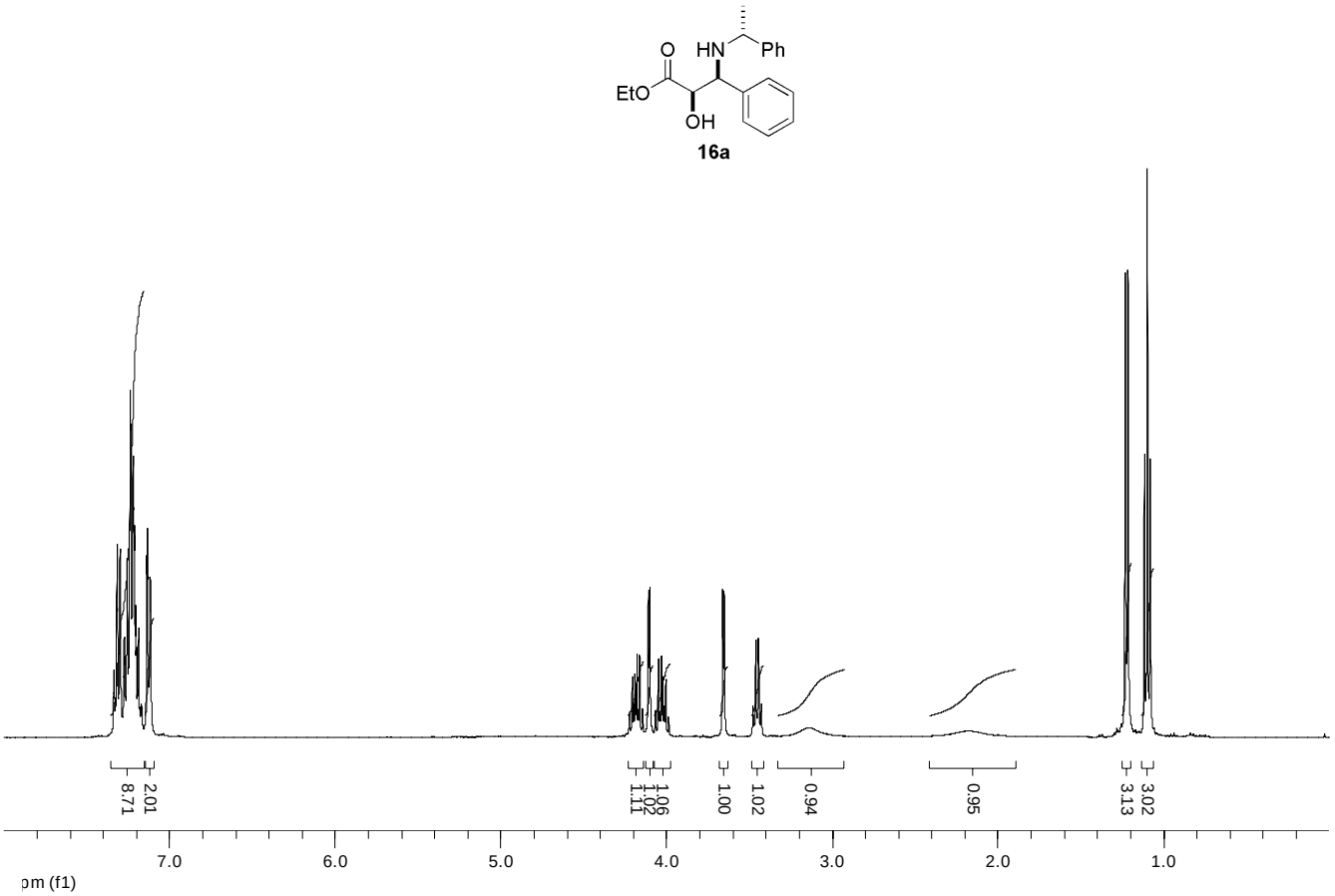
pm (f1)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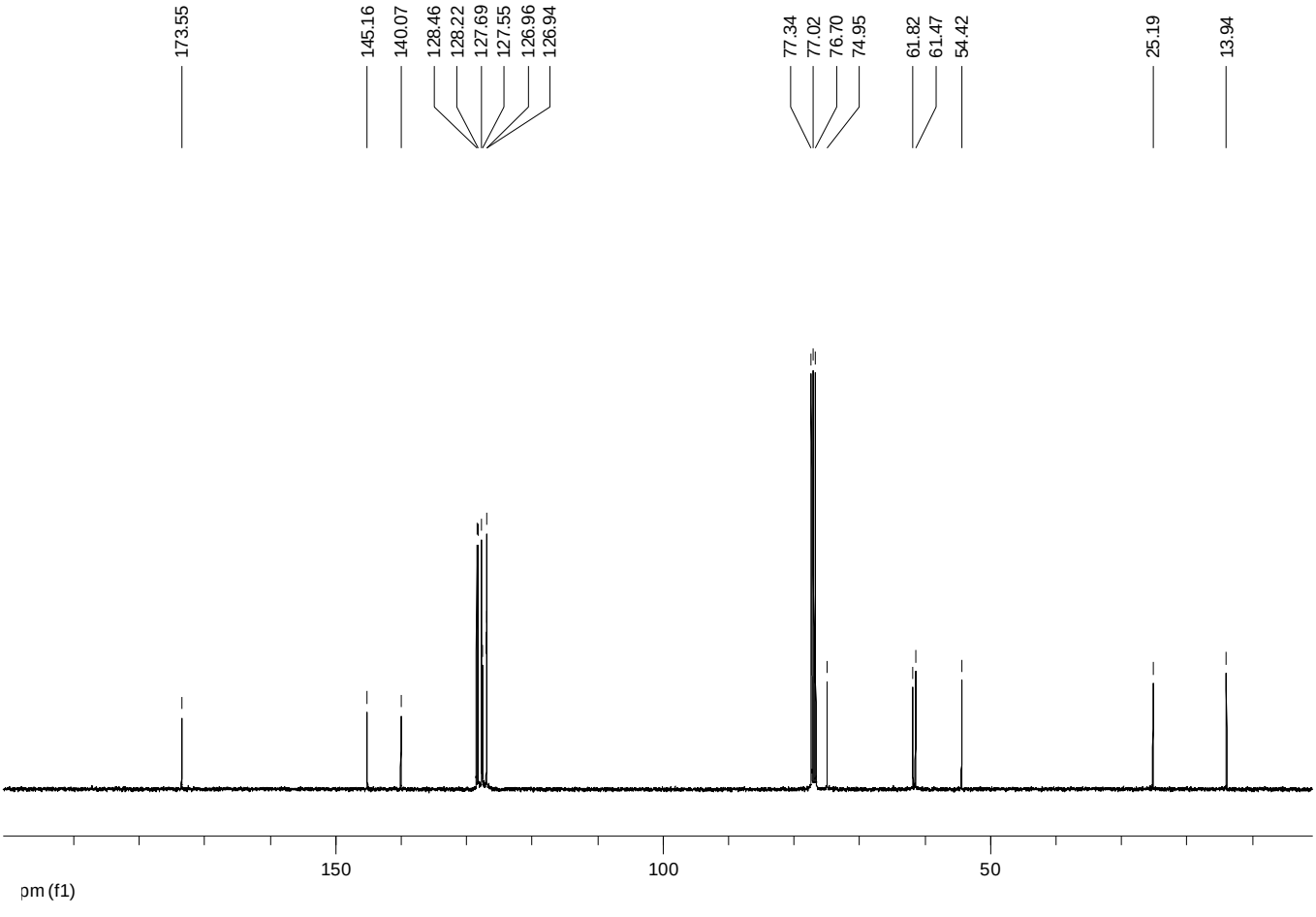
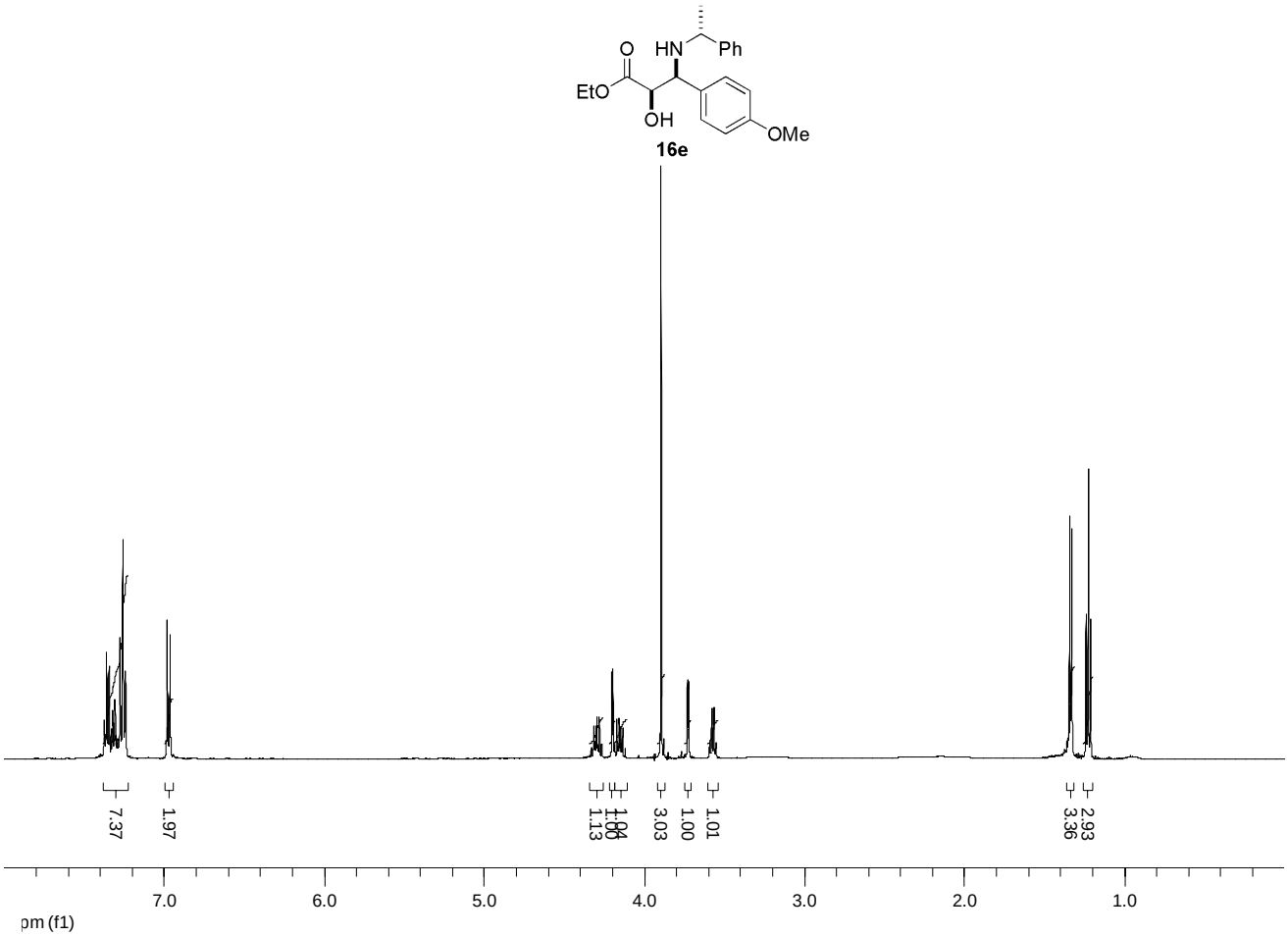
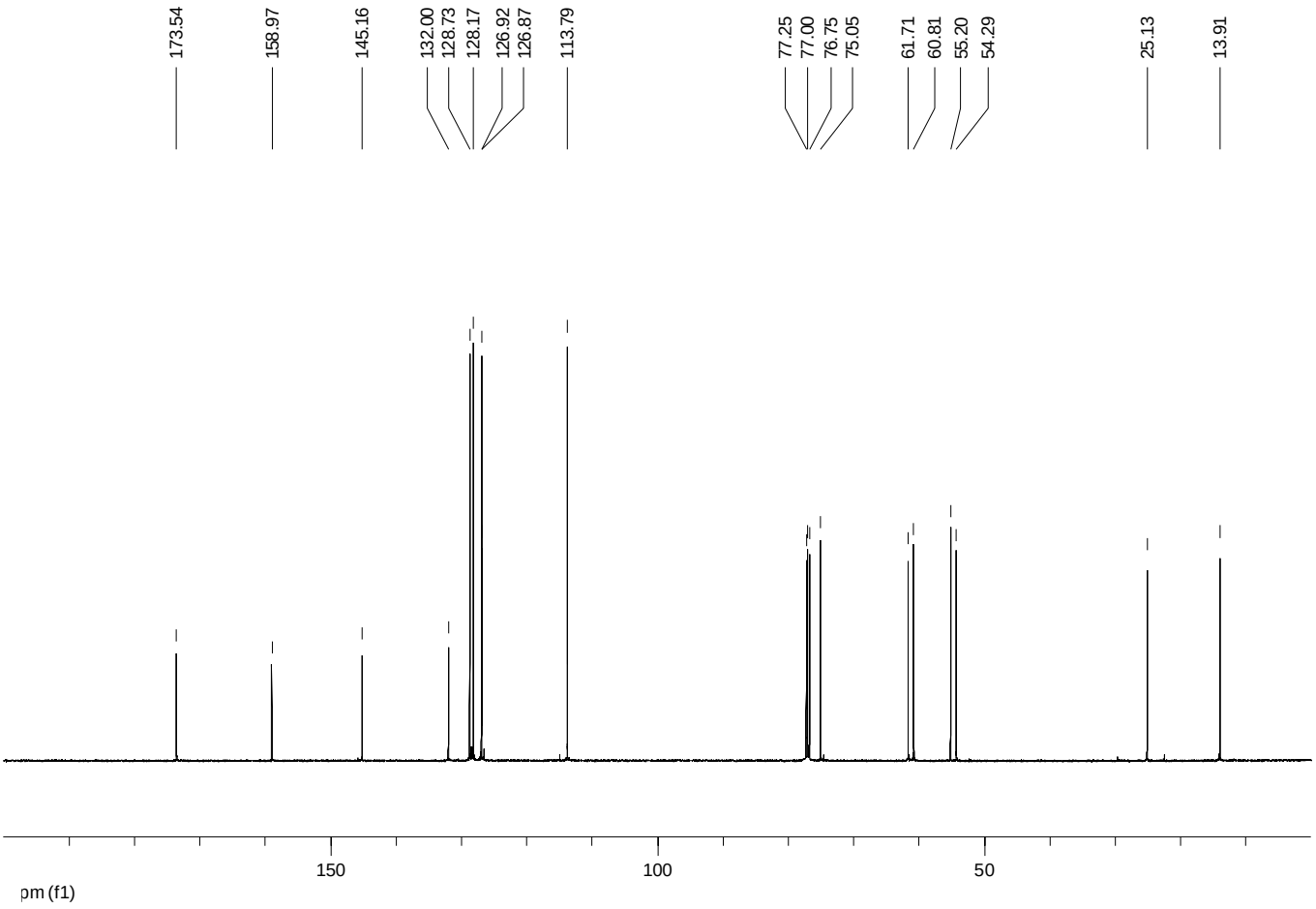
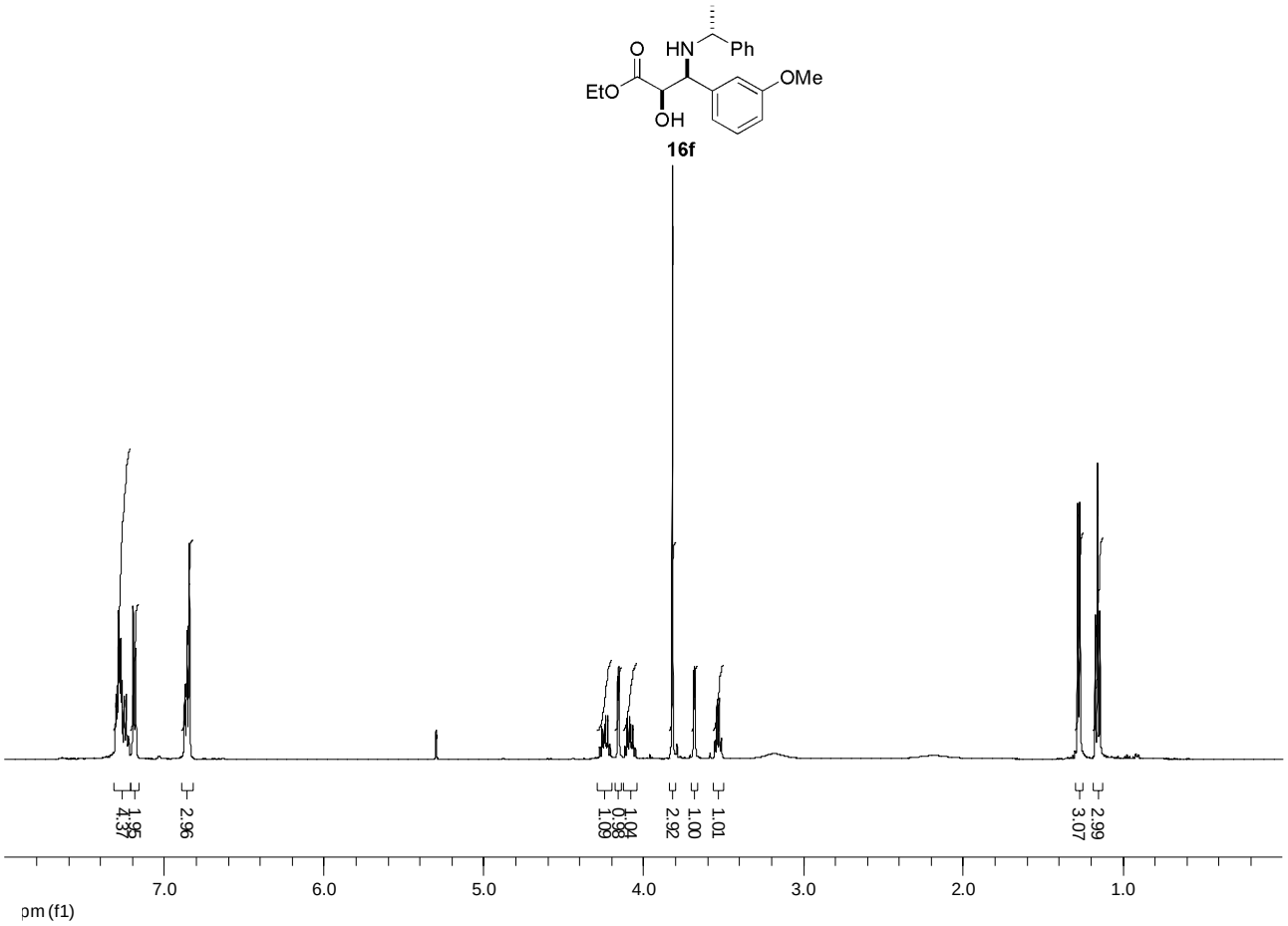
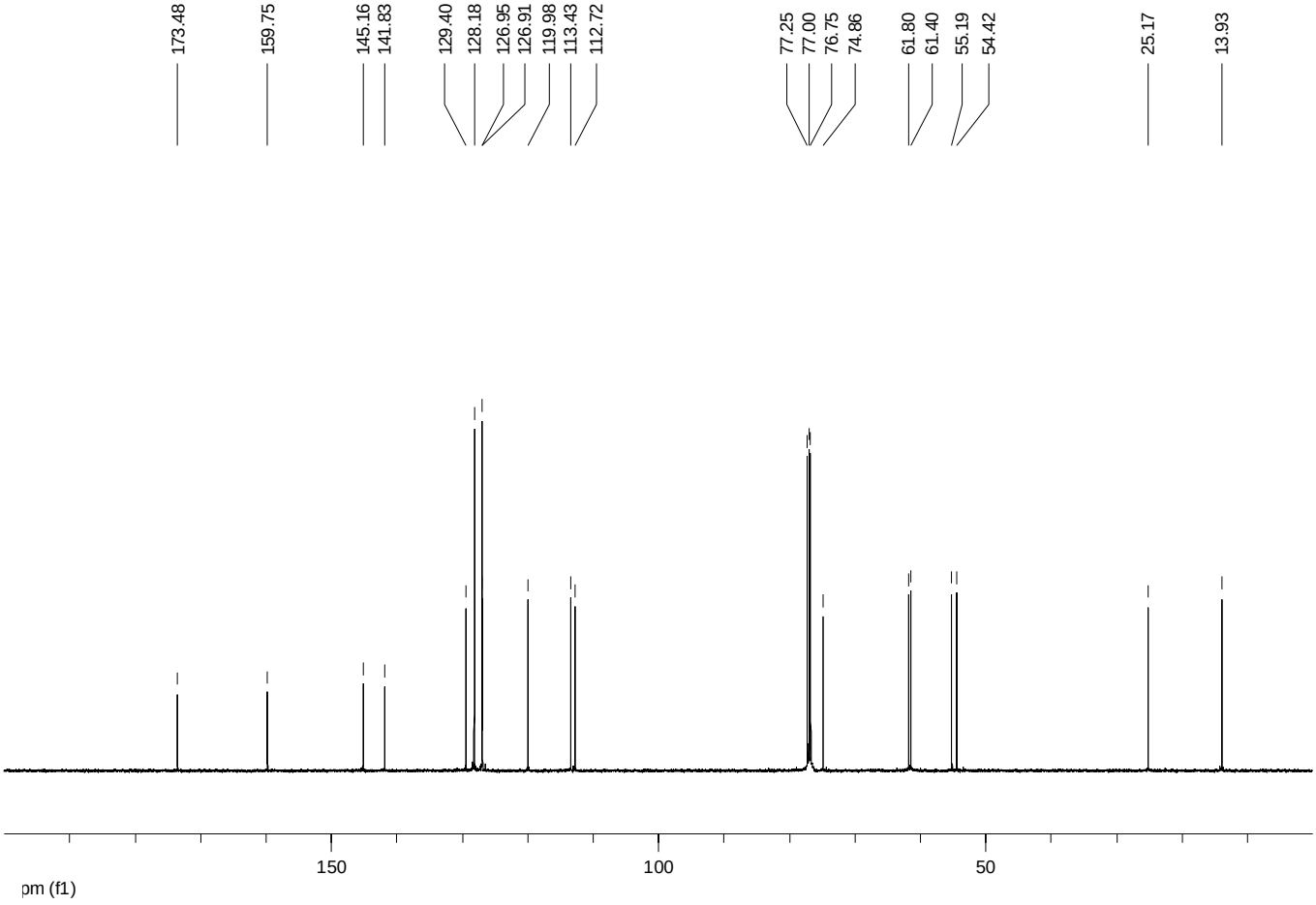
1.20
1.06
1.00
1.02
0.94
0.95
3.13
3.02
7.0
6.0
5.0
4.0
3.0
2.0
1.0
pm (f1)

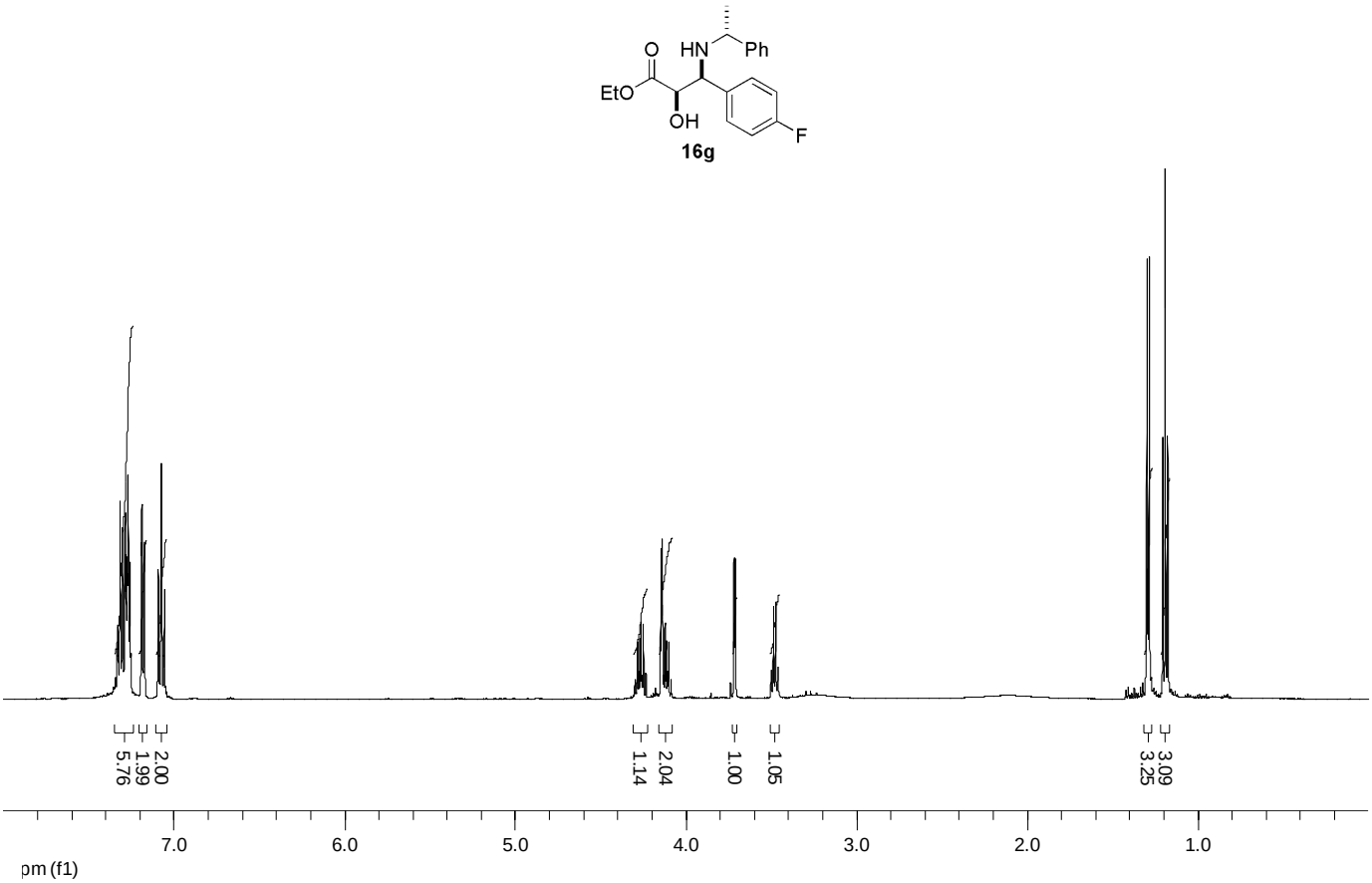

173.55
145.16
140.07
128.46
128.22
127.69
127.55
126.96
126.94
77.34
77.02
76.70
74.95
61.82
61.47
54.42
25.19
13.94
150
100
50
pm (f1)

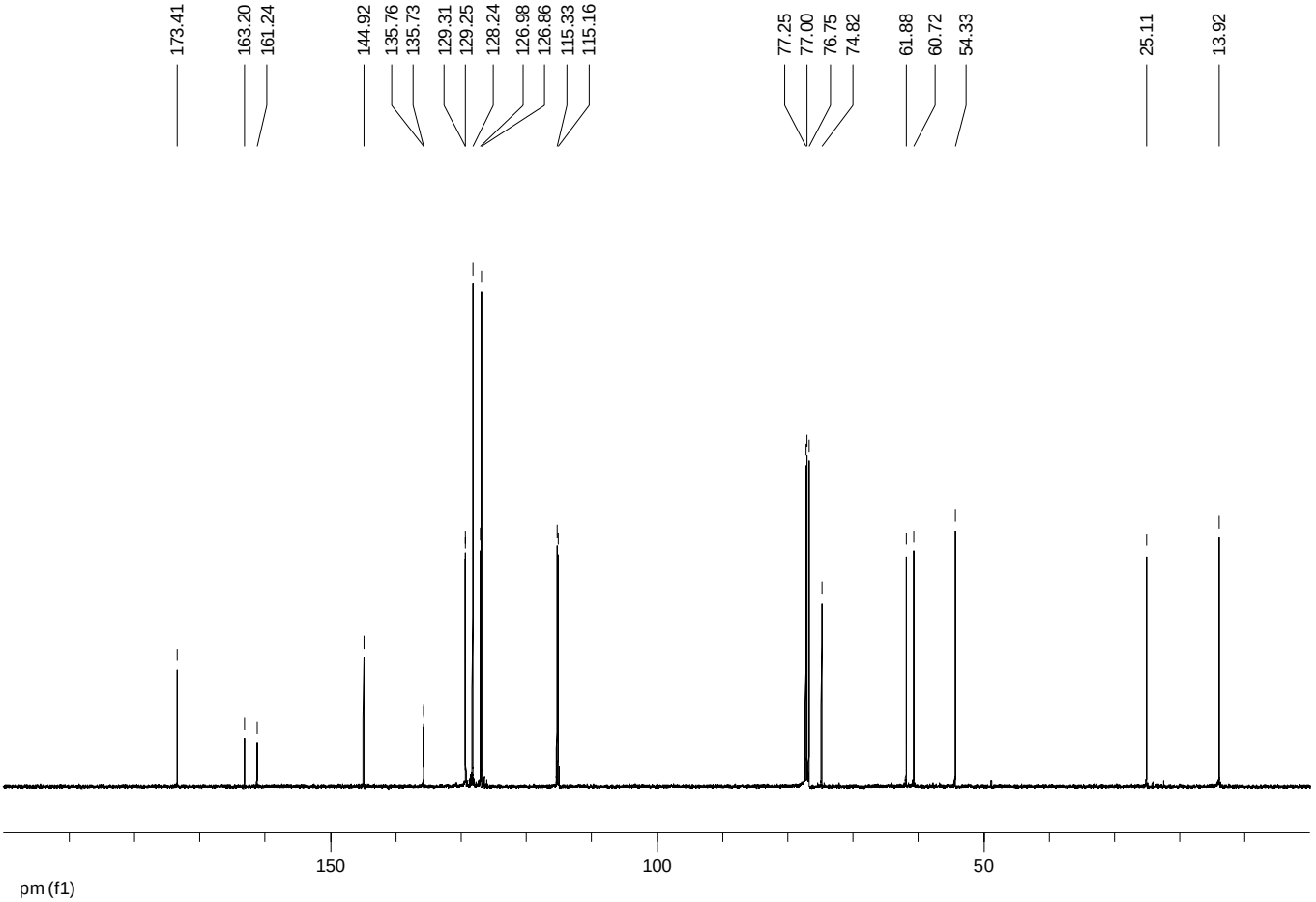

O
HN
Ph
EtO
OH
OMe
16e
7.37
1.97
1.13
1.00
1.04
3.03
1.00
1.01
3.36
2.23
7.0
6.0
5.0
4.0
3.0
2.0
1.0
pm (f1)

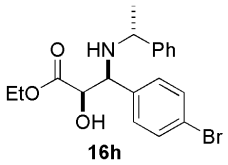

173.54
158.97
145.16
132.00
128.73
128.17
126.92
126.87
113.79
77.25
77.00
76.75
75.05
61.71
60.81
55.20
54.29
25.13
13.91
150
100
50
pm (f1)


O
HN
Ph
EtO
OMe
OH
16f
4.37
1.95
2.96
1.09
0.98
1.04
2.92
1.00
1.01
3.07
2.99
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)

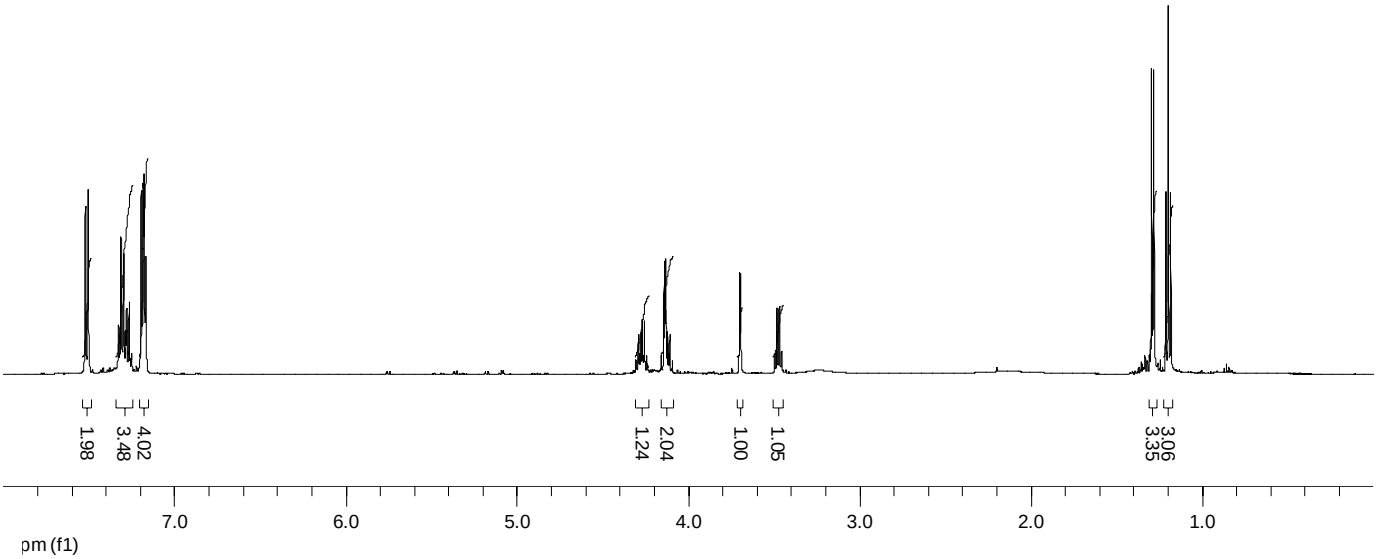

173.48
159.75
145.16
141.83
129.40
128.18
126.95
126.91
119.98
113.43
112.72
77.25
77.00
76.75
74.86
61.80
61.40
55.19
54.42
25.17
13.93
150
100
50
ppm (f1)

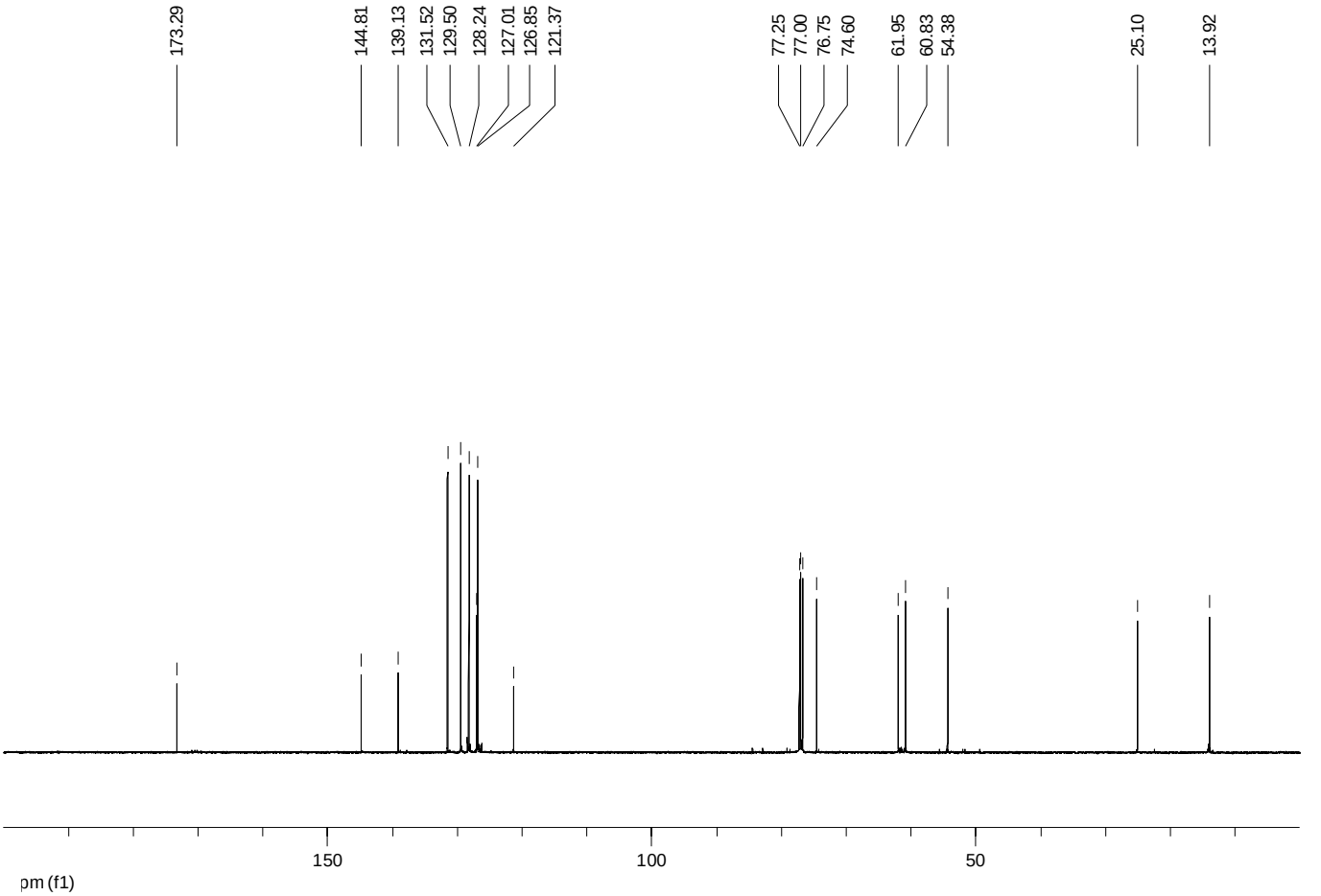

O
HN
Ph
EtO
OH
F
16g
5.79
1.99
2.00
1.14
2.04
1.00
1.05
3.25
3.09
7.0
6.0
5.0
4.0
3.0
2.0
1.0
pm (f1)


173.41
163.20
161.24
144.92
135.76
135.73
129.31
129.25
128.24
126.98
126.86
115.33
115.16
77.25
77.00
76.75
74.82
61.88
60.72
54.33
25.11
13.92
150
100
50
pm (f1)


O
HN
Ph
EtO
OH
Br

16h


1.98
3.48
4.02
1.24
2.04
1.00
1.05
3.35
3.06
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)


173.29
144.81
139.13
131.52
129.50
128.24
127.01
126.85
121.37
77.25
77.00
76.75
74.60
61.95
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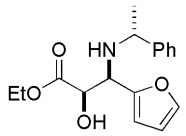
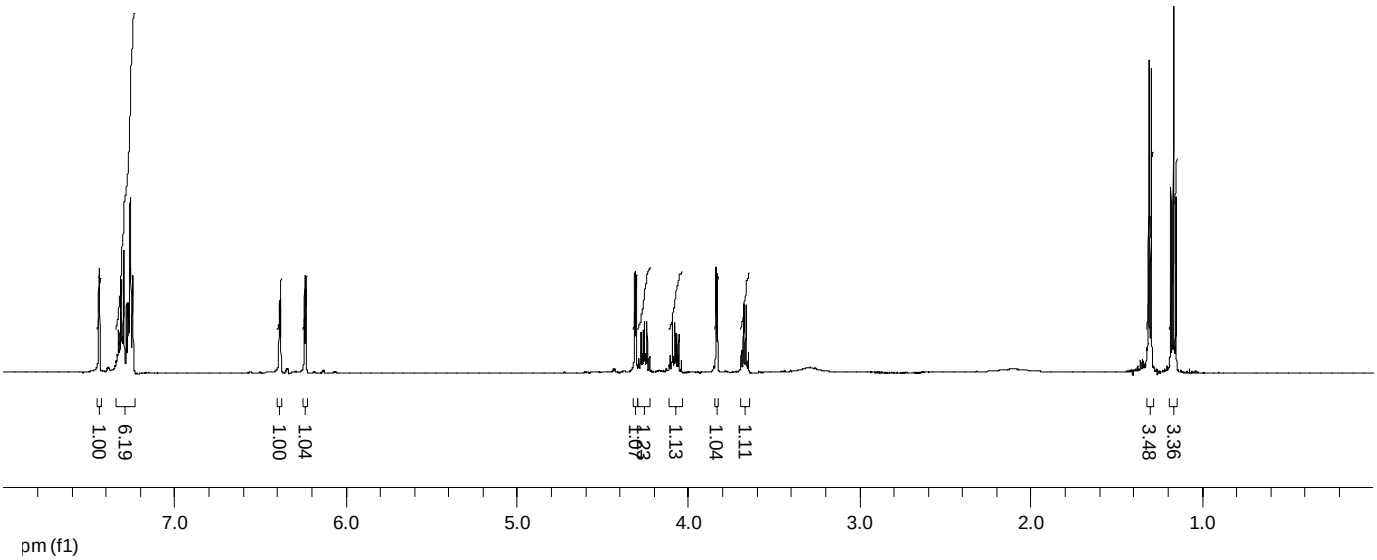
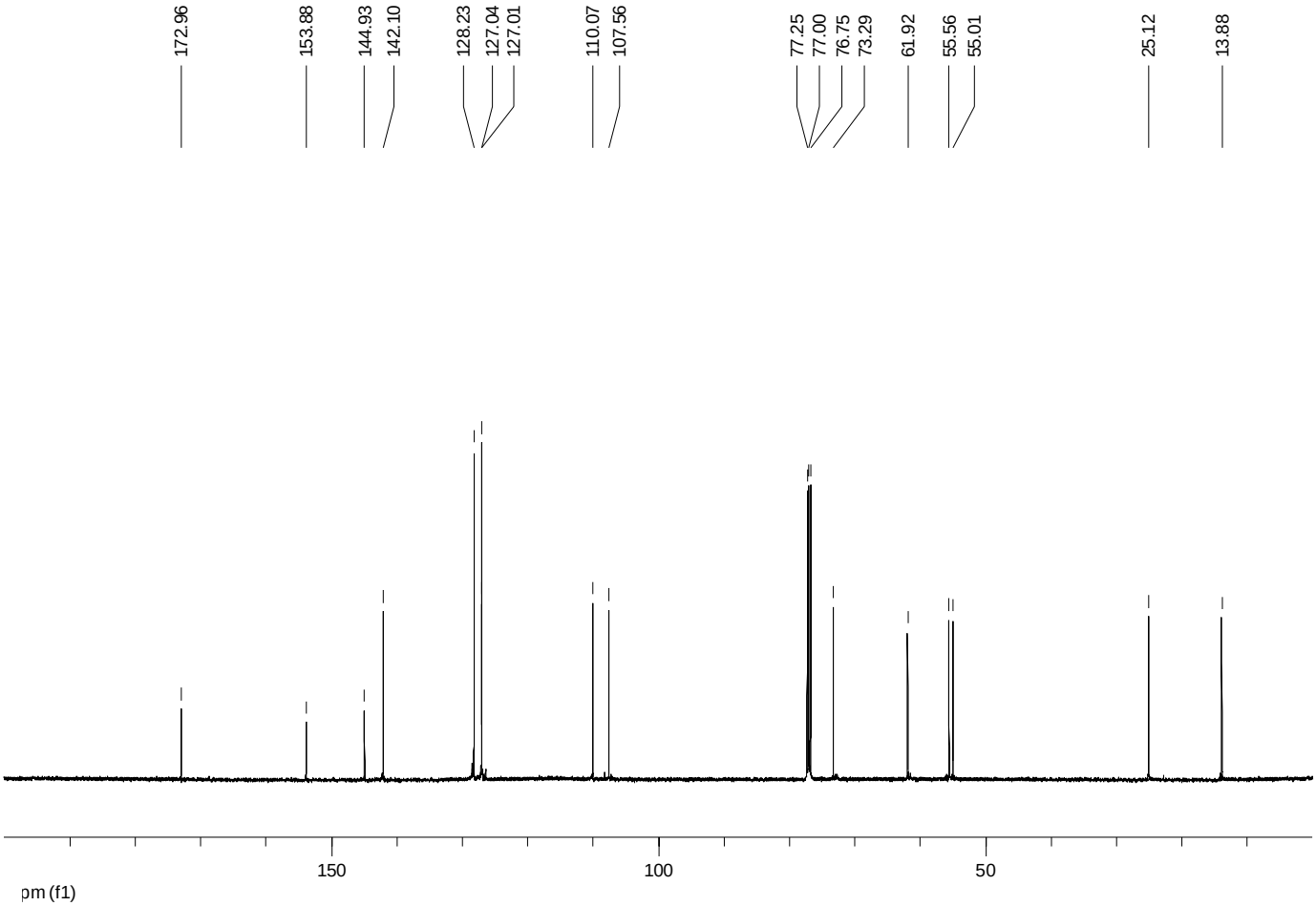
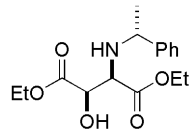
54.38
25.10
13.92
150
100
50
ppm (f1)


O
HN
Ph
EtO
O
OH

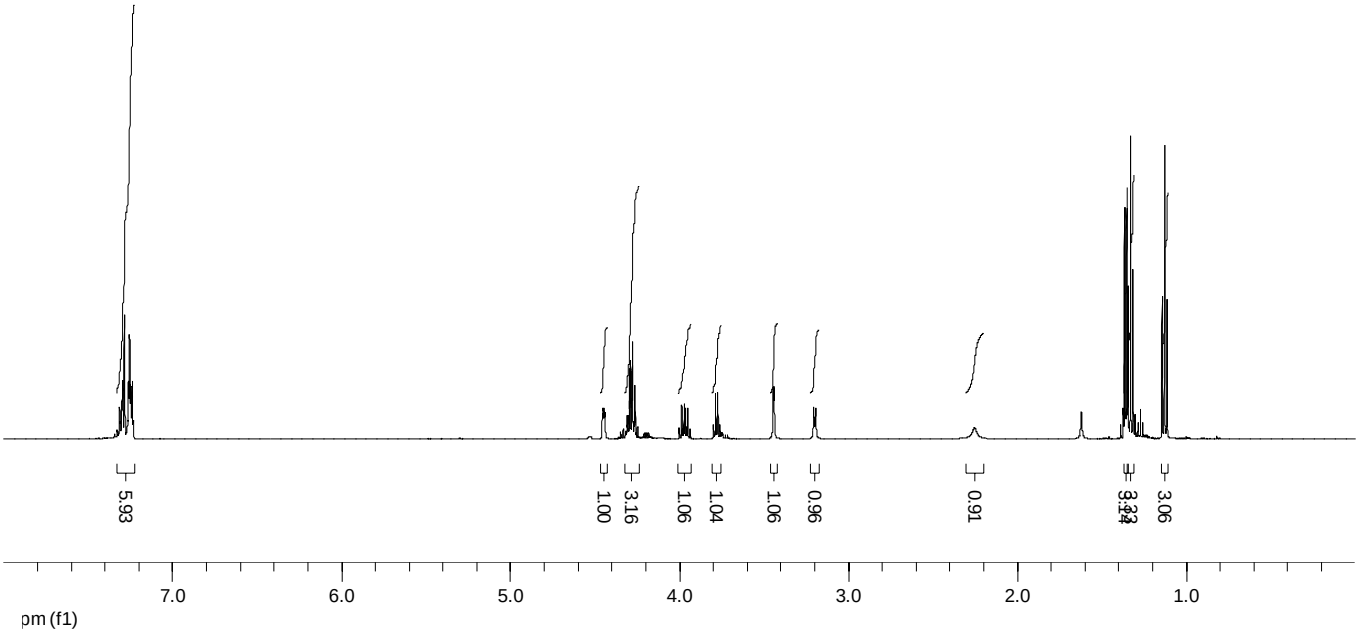
16i

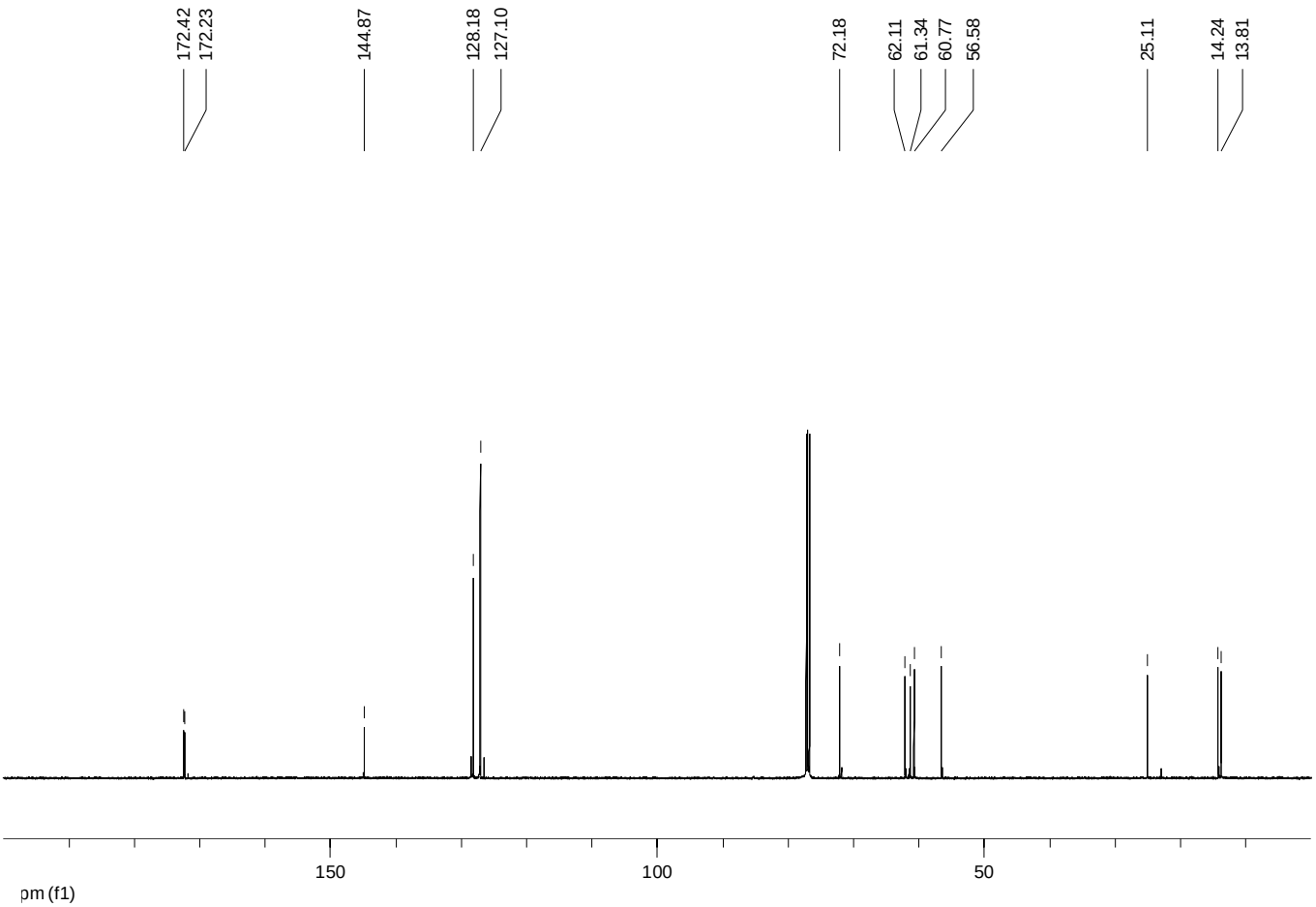

1.00
6.19
1.00
1.04
1.07
1.23
1.13
1.04
1.11
3.48
3.36
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)


172.96
153.88
144.93
142.10
128.23
127.04
127.01
110.07
107.56
77.25
77.00
76.75
73.29
61.92
55.56
55.01
25.12
13.88
150
100
50
ppm (f1)


O
HN
Ph
EtO
OEt
OH
O

16j


5.93
1.00
3.16
1.06
1.04
1.06
0.96
0.91
3.06
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)


172.42
172.23
144.87
128.18
127.10
72.18
62.11
61.34
60.77
56.58
25.11
14.24
13.81
150
100
50
ppm (f1)