Enantioselective Synthesis of α,α-Disubstituted Cyclopentenes by an *N*-Heterocyclic Carbene-Catalyzed Desymmetrization of 1,3-Diketones

Manabu Wadamoto, Eric M. Phillips, Troy E. Reynolds and Karl A. Scheidt*

Department of Chemistry, Northwestern University,
2145 Sheridan Road, Evanston, Illinois 60208

Supporting Information

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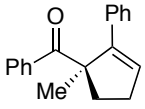
General Information

All reactions were carried out under a nitrogen atmosphere in flame-dried glassware with magnetic stirring. CH_2Cl_2 was purified by passage through a bed of activated alumina.[1] Reagents were purified prior to use unless otherwise stated following the guidelines of Perrin and Armarego.[2] Purification of reaction products was carried out by flash chromatography using EM Reagent silica gel 60 (230-400 mesh). Analytical thin layer chromatography was performed on EM Reagent 0.25 mm silica gel 60-F plates. Visualization was accomplished with UV light and ceric ammonium nitrate stain or potassium permangenate stain followed by heating. Infrared spectra were recorded on a Perkin Elmer 1600 series FT-IR spectrometer. ^{1}H-NMR spectra were recorded on a Varian Inova 500 (500 MHz) spectrometer and are reported in ppm using solvent as an internal standard ($CDCl_3$ at 7.26 ppm). Data are reported as (ap = apparent, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, b = broad; coupling constant(s) in Hz; integration. Proton-decoupled ^{13}C-NMR spectra were recorded on a Varian Inova 500 (125 MHz) spectrometer and are reported in ppm using solvent as an internal standard ($CDCl_3$ at 77.0 ppm). Mass spectra data were obtained on a Varian 1200 Quadrupole Mass Spectrometer and Micromass Quadro II Spectrometer.

General Procedure for Desymmetrization of 1,3-Diketone

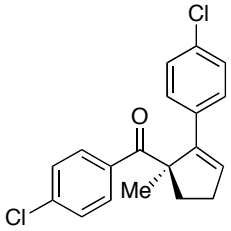
To an oven dried 10 mL round bottom flask containing a magnetic stirring bar was added azolium salt **D** (4.2 mg, 0.01 mmol) and the corresponding enal 1,3-diketone (0.1 mmol) in dry box. The heterogeneous mixture was then diluted with CH_2Cl_2 (2 mL, 0.05 M). Once the material dissolved, diisopropylethylamine (17 μL, 0.1 mmol) was added via syringe. The reaction was stirred at 40 ˚C under N_2 atmosphere until consumption of diketone was observed by TLC. The reaction mixture was diluted with hexane and applied directly to a silica gel column to provide the pure cyclopentene.



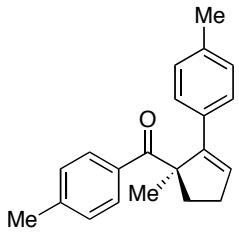
(*R*)-(1-methyl-2-phenylcyclopent-2-enyl)(phenyl)methanone (4): Prepared according to general procedure using (*E*)-5-methyl-6-oxo-6-phenyl-5-(phenylcarbonyl)hex-2-enal (92 mg, 0.3 mmol), azolium salt (15 mg, 0.03 mmol) diisopropylethylamine (54μL, 0.3 mmol) to afford 63 mg (80%) of **4** as a colorless oil. Analytical data for **4**: IR (film) 3057, 2962, 2929, 2847, 1670, 1596, 1496, 1446, 1372, 1261, 1240, 1172, 976, 758, 692 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 (d, *J* = 7.7 Hz, 2H), 7.40 (t, *J* = 7.3 Hz, 1H), 7.34-7.38 (m, 4H), 7.21 (t, *J* = 7.3 Hz, 2H), 7.15 (m, 1H), 6.36 (s, 1H), 2.73-2.60 (m, 3H), 1.99-1.97 (m, 1H), 1.55 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 205.0, 148.6, 137.5, 135.2, 132.0, 129.3, 128.9, 128.7, 128.3, 127.5, 126.5, 62.1, 39.0, 31.1, 24.1; GCMS (CI); Exact mass cals for $C_{19}H_{18}O$ $[M]^+$, 262.14 Found [M + H], 263; $[\alpha]_D$: +229.0 ($CHCl_3$, c = 1.0, er = 96.5 : 3.5). Enantiomeric ratio

1. Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometal.* **1996**, *15*, 1518-1520.
2. Perrin, D. D. and Armarego, W. L. *Purification of Laboratory Chemicals;* 3rd Ed., Pergamon Press, Oxford. 1988.

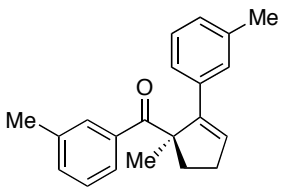
was measured by chral HPLC (Chiralcel OD-H, 3% IPA/Hexanes, 1mL/min, Rt_{major} = 5.87, Rt_{minor} = 9.22).



(*R*)-(4-chlorophenyl)(2-(4-chlorophenyl)-1-methylcyclopent-2-enyl)methanone (5): Prepared according to general procedure using (*E*)-6-(4-chlorophenyl)-5-(4-chlorophenylcarbonyl)-5-methyl-6-oxohex-2-enal (38 mg, 0.1 mmol), azolium salt (5 mg, 0.01 mmol) diisopropylethylamine (17 μL, 0.1 mmol) to afford 25 mg (76%) of **5** as a colorless oil. Analytical data for **5**: IR (film) 3445, 3026, 2957, 2931, 2860, 1684, 1496, 1455, 1377, 1181, 1049, 748, 699 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.92 (d, *J* = 8.6 Hz, 2H), 7.27 (d, *J* = 8.4 Hz, 2H), 7.22 (d, *J* = 8.6 Hz, 2H), 7.16 (d, *J* = 8.6 Hz, 2H), 6.37 (d, *J* = 2.2 Hz, 1H), 2.76-2.57 (m, 3H), 2.03-2.57 (m, 1H), 1.53 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 203.2, 147.6, 138.7, 135.2, 133.4, 133.3, 130.8, 129.5, 129.0, 128.7, 127.6, 612.0, 38.9, 31.1, 23.8; GCMS (CI): Exact mass calcd for $C_{19}H_{16}Cl_2O$ $[M]^+$, 330.06. Found [M+Na], 331; $[\alpha]_D$: +20.6 (MeOH, c = 1.0, er = 97:3). Enantiomeric ratio was measured by HPLC (Chiralcel OD-H, 5% IPA/Hexanes, 1mL/min, Rt_1 = 6.95, Rt_2 = 7.97).

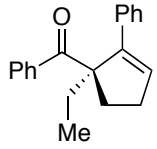


(*R*)-(1-methyl-2-*p*-tolylcyclopent-2-enyl)(*p*-tolyl)methanone (6): Prepared according to general procedure using (*E*)-5-methyl-5-(4-methylphenylcarbonyl)-6-oxo-6-*p*-tolylhex-2-enal (33 mg, 0.1 mmol), azolium salt (5 mg, 0.01 mmol) diisopropylethylamine (17 μL, 0.1 mmol) to afford 17 mg (60%) of **6** as a colorless oil. Analytical data for **6**: IR (film) 2924, 2848, 1666, 1606, 1512, 1545, 1371, 1265, 1242, 1172, 991, 976, 813 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.92 (d, *J* = 8.1 Hz, 2H), 7.22 (d, *J* = 8.1 Hz, 2H), 7.09 (d, *J* = 8.05 Hz, 2H), 7.00 (d, *J* = 7.9 Hz, 2H), 6,31 (s, 1H), 2.72–2.58 (m, 3H), 2.31 (s, 3H), 2.25 (s, 3H), 1.98–1.94 (m, 1H), 1.52 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 204.5, 148.8, 142.6, 137.2, 134.7, 132.3, 129.5, 129.4, 129.0, 127.5, 126.4, 62.0, 39.1, 31.0, 24.0, 21.7, 21.3; GCMS (electrospray): Exact mass calcd for $C_{21}H_{22}O$ $[M]^+$, 290.17. Found [M+H], 291; $[\alpha]_D$: +183 (c=1.0, $CHCl_3$, er = 97:3), Enantiomeric ratio was measured by HPLC (Chiralcel AD-H, 3% IPA/Hexanes, 1mL/min, Rt_{major}, = 4.71, Rt_{minor} = 5.27).

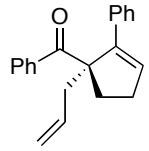


(*R*)-(1-methyl-2-*p*-tolylcyclopent-2-enyl)(*m*-tolyl)methanone (7): Prepared according to general procedure using (*E*)-5-methyl-5-(4-methylphenylcarbonyl)-6-oxo-6-*m*-tolylhex-2-enal (33 mg, 0.1 mmol), azolium salt (5 mg, 0.01 mmol) diisopropylethylamine (17 μL, 0.1 mmol) to afford 19 mg (65%) of **7** as a colorless oil. Analytical data for **7**: IR (film) 2924, 2845, 1668, 1599, 1486, 1454, 1370, 1267, 1156, 1092, 782, 732 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.3 Hz, 2H), 7.22 (d, *J* = 7.3 Hz, 1H), 7.18-7.15 (m, 2H), 7.11 (d, *J* = 4.4 Hz, 2H), 6.98 (m, 1H), 6.35-6.34 (m, 1H), 2.72–2.58 (m, 3H), 2.33 (s, 3H), 2.28 (s, 3H), 2.00–1.96 (m, 1H), 1.55 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 205.4,

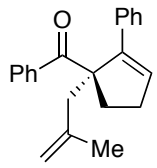
148.7, 138.1, 138.0, 137.6, 135.2, 132.7, 129.9, 128.7, 128.6, 128.3, 128.0, 127.3, 126.3, 123.6, 62.9, 38.9, 31.0, 24.2, 21.7, 21.7; GCMS (electrospray): Exact mass calcd for $C_{21}H_{22}O$ $[M]^+$, 290.17. Found [M+H], 291; $[\alpha]_D$: +183 (c=1.0, $CHCl_3$, er = 97:3), Enantiomeric ratio was measured by HPLC (Chiralcel AD-H, 3% IPA/Hxanes, 1mL/min, Rt_{major}, = 5.29, Rt_{minor} = 5.93).



(*R*)-(1-ethyl-2-phenylcyclopent-2-enyl)(phenyl)methanone (8): Prepared according to general procedure using (*E*)-5,5-di(phenylcarbonyl)hept-2-enal (35 mg, 0.1 mmol), azolium salt (5 mg, 0.01 mmol) diisopropylethylamine (17 μL, 0.1 mmol) to afford 20 mg (73%) of **8** as a colorless oil. Analytical data for **8**: IR (film) 3054, 2962, 2846, 1668, 1596, 1444, 1258, 1260, 1168, 1075, 1026, 985, 827, 764, 691 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.3 Hz, 2H), 7.44-7.42 (m, 1H), 7.35-7.16 (m, 7H), 6.44 (s, 1H), 2.68-2.63 (m, 2H), 2.55-2.51 (m, 1H), 2.15-2.11 (m, 1H), 2.02 (q, *J* = 7.8 Hz, 2H), 0.81 (t, *J* = 7.8 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 205.4, 145.6, 138.5, 125.7, 131.8, 131.6, 128.9, 128.7, 128.3, 127.5, 126.7, 66.3, 34.6, 32.2, 28.6, 8.2; MS (CI): Exact mass calcd for $C_{20}H_{20}O$ $[M]^+$, 276.15. Found [M+H], 297; $[\alpha]_D$: +63.6 ($CHCl_3$, c = 1.0, er = 95:5). Enantiomeric ratio was measured by HPLC (Chiralcel AD-H, 3% IPA/Hexanes, 1mL/min, Rt_{major} = 6.23, Rt_{minor} = 8.02).

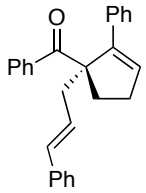


(*R*)-(1-allyl-2-phenylcyclopent-2-enyl)(phenyl)methanone (9): Prepared according to general procedure using (*E*)-5,5-di(phenylcarbonyl)octa-2,7-dienal (33 mg, 0.1 mmol), azolium salt (5 mg, 0.01 mmol) diisopropylethylamine (17 μL, 0.1 mmol) to afford 20 mg (70%) of **9** as a colorless oil. Analytical data for **9**: IR (film) 3064, 2925, 1668, 1445, 1239, 1179, 1102, 1070, 1000, 917, 698 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.98 (d, *J* = 7.3 Hz, 2H), 7.44 (t, *J* = 7.3 Hz, 1H), 7.35-7.17 (m, 7H), 6.42 (t, *J* = 2.4 Hz, 1H), 5.77-5.69 (m, 1H), 5.06-5.02 (m, 2H), 2.75-2.72 (t, *J* = 7.8 Hz, 2H), 2.62-2.60 (m, 2H), 2.57-2.47 (m, 1H), 2.24-2.20 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 204.6, 146.0, 138.1, 135.4, 134.8, 132.0, 131.6, 129.0, 128.7, 128.4, 127.6, 126.8, 118.1, 65.9, 40.6, 34.5, 31.9; MS (CI): Exact mass calcd for $C_{21}H_{20}O$ $[M]^+$, 288.15. Found [M+H], 289; $[\alpha]_D$: +223 ($CHCl_3$, c = 1.0, er = 91.5:8.5). Enantiomeric ratio was measured by HPLC (Chiralcel AD-H, 3% IPA/Hexanes, 1mL/min, Rt_{major} = 5.28, Rt_{minor} = 6.23).

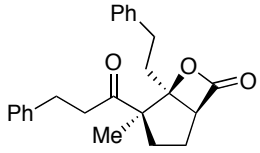


(*R*)-(1-(2-methylallyl)-2-phenylcyclopent-2-enyl)(phenyl)methanone (10): Prepared according to general procedure using (*E*)-7-methyl-5,5-di(phenylcarbonyl)octa-2,7-dienal (33 mg, 0.1 mmol), azolium salt (5 mg, 0.01 mmol) diisopropylethylamine (17 μL, 0.1 mmol) to afford 21 mg (69%) of **10** as a colorless oil. Analytical data for **10**: IR (film) 3066, 2918, 2846, 1669, 1596, 1495, 1444, 1227, 1165, 1073, 982, 892, 761, 693 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.88 (d, *J* = 7.3 Hz, 2H), 7.41 (t, *J* = 7.3 Hz, 1H), 7.37-7.18 (m, 7H), 6.43 (t, *J* = 2.4 Hz, 1H), 4.83 (s, 1H), 4.67 (s, 1H), 2.99 (d, *J* = 4.2 Hz, 1H),

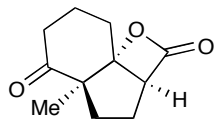
2.71-2.65 (m, 2H), 2.57-2.52 (m, 2H), 2.41-2.38 (m, 1H), 1.65 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 205.3, 146.5, 143.4, 138.5, 135.0, 132.0, 131.7, 128.9, 128.7, 128.2, 127.5, 126.9, 115.1, 65.8, 43.5, 34.5, 31.9, 24.8; GCMS (CI): Exact mass calcd for $C_{22}H_{22}O$ $[M]^+$, 302.17. Found [M+H], 303; $[\alpha]_D$: +50.9 ($CHCl_3$, c = 1.0, er = 91.5:8.5). Enantiomeric ratio was measured by HPLC (Chiralcel AD-H, 3% IPA/Hexanes, 1mL/min, Rt_{major} = 6.62, Rt_{minor} = 8.37).



(*R*,*E*)-(1-cinnamyl-2-phenylcyclopent-2-enyl)(phenyl)methanone (11): Prepared according to general procedure using (2*E*,7*E*)-8-phenyl-5,5-di(phenylcarbonyl)octa-2,7-dienal (33 mg, 0.1 mmol), azolium salt (5 mg, 0.01 mmol) diisopropylethylamine (17 μL, 0.1 mmol) to afford 23 mg (64%) of **11** as a colorless oil. Analytical data for **11**: IR (film) 3445, 3026, 2957, 2931, 2860, 1684, 1496, 1455, 1377, 1181, 1049, 748, 699 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 (d, *J* = 7.7 Hz, 2H), 7.44 (t, *J* = 7.5 Hz, 1H), 7.36-7.19 (m, 12H), 6.44 (bs, 1H), 6.37 (d, *J* = 16 Hz, 1H), 6.17-6.11 (m, 1H), 2.92 (t, *J* = 8.4, 2H) 2.61-2.50 (m, 3H), 2.28-2.23 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 204.5, 146.2, 138.1, 138.0, 135.4, 133.2, 131.6, 129.1, 128.8, 128.7, 128.4, 127.7, 127.2, 126.9, 126.7, 126.3, 66.0, 39.7, 34.9, 31.9; GCMS (CI): Exact mass calcd for $C_{27}H_{24}O$ $[M]^+$, 364.18. Found [M+H], 365; $[\alpha]_D$: +65.2 ($CHCl_3$, c = 1.0, er = 91:9). Enantiomeric ratio was measured by HPLC (Chiralcel AD-H, 5% IPA/Hexanes, 1mL/min, Rt_{minor} = 8.89, Rt_{major} = 9.70).



(1*R*,4*S*,5*R*)-4-methyl-5-phenethyl-4-(3-phenylpropanoyl)-6-oxabicyclo[3.2.0]heptan-7-one (12): Prepared according to general procedure using (*E*)-5-methyl-6-oxo-8-phenyl-5-(3-phenylpropanoyl)oct-2-enal (33 mg, 0.1 mmol), azolium salt (10 mg, 0.02 mmol) diisopropylethylamine (17 μL, 0.1 mmol) to afford 23 mg (65%) of **12** as a colorless oil. Analytical data for **12**: IR (film) 3445, 3026, 2957, 2931, 2860, 1684, 1496, 1455, 1377, 1181, 1049, 748, 699 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33–7.16 (m, 10H), 3.63 (d, *J* = 7.8, 1H), 3.00-2.94 (m, 1H), 2.88-2.81 (m, 3H), 2.78-2.71 (m, 2H), 2.31-2.24 (m, 1H), 2.15-2.09 (m, 1H), 1.90-1.81 (m, 2H), 1.78-1.71 (m, 2H), 1.27 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 211.3, 171.5, 141.1, 128.8, 128.8, 128.6, 128.6, 126.6, 126.5, 92.2, 59.4, 57.0, 40.7, 35.0, 32.3, 30.7, 30.0, 29.6, 24.1, 15.8; LRMS (electrospray): Exact mass calcd for $C_{18}H_{26}O_2$ $[M]^+$, 362.19. Found [M+Na], 747.5; $[\alpha]_D$: –1.2 ($CHCl_3$, c = 1.0, er = 96:3). Enantiomeric ratio was measured by HPLC (Chiralcel AD-H, 5% EtOH/Hexanes, 1mL/min, Rt_{minor} = 8.43, Rt_{major} = 9.41).

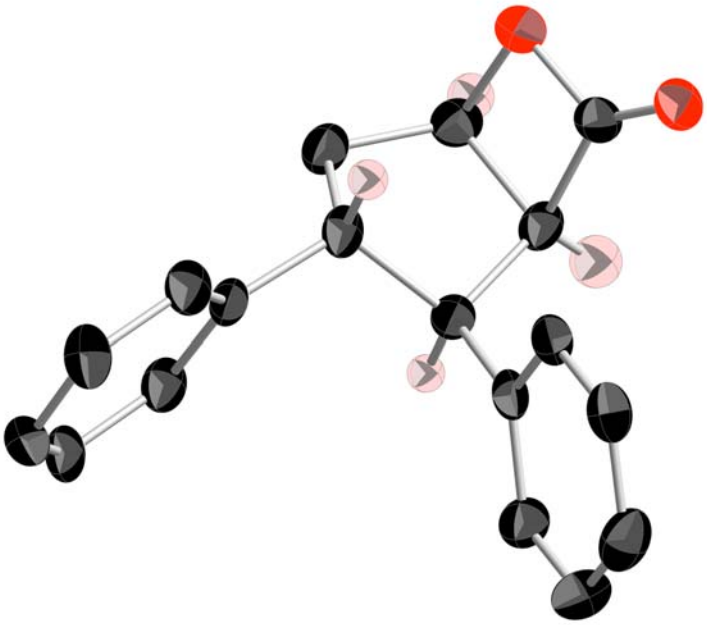


(2aS, 4aS, 8aS)-4a-Methyl-hexahydro-1-oxacyclobuta[*c*]indene-2,5-dione (13): Prepared according to general procedure using (*E*)-4-(1-methyl-2,6-dioxocyclohexyl)but-2-enal (19 mg, 0.1 mmol), azolium salt (10 mg, 0.02 mmol) diisopropylamine (17 μL, 0.1 mmol) to afford 10 mg (51%) of **13** as a white solid. Analyical data for **13**: IR (film) 3445, 3026, 2957, 2931, 2860, 1684, 1496, 1455, 1377, 1181, 1049, 748, 699 cm^{-1}; ^{1}H NMR (500

MHz, $CDCl_3$) δ 3.48 (d, *J* = 7.2 Hz, 1H), 2.77-2.73 (m, 1H), 2.58-2.52 (m, 1H), 2.39-2.33 (m, 2H), 2.27-2.24 (m, 1H), 2.13-2.10 (m, 1H), 1.98-1.94 (m, 1H), 1.64-1.56 (m, 2H), 1.55-1.40 (m, 1H), 1.29 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 210.4, 170.7, 89.6, 59.2, 58.9, 37.3, 32.1, 28.2, 24.8, 19.7, 18.5; LRMS (electrospray): Exact mass calcd for $C_{11}H_{14}O_3$ $[M]^+$, 194.09. Found [2M+Na], 411.5; $[\alpha]_D$: –27.3 ($CHCl_3$, c = 1.0, er = 97.5:2.5). Enantiomeric ratio was measured by GC (Beta Dex 225, 23.00 psi, 80 °C – 170 °C, Rt_{minor}= 27.44, Rt_{major} = 27.70).

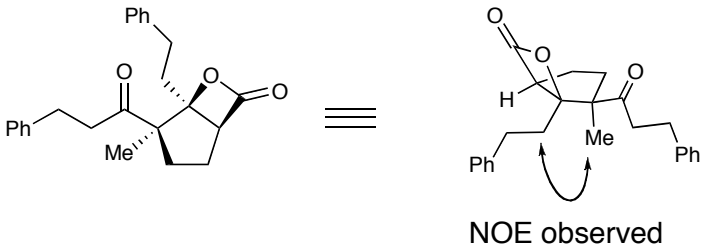
X-ray crystal structure of cinnamaldehyde dimer 3:

X-ray diffraction was performed at –120 °C and raw frame data were processed using SAINT. Molecular structure was solved using direct methods and refined by F2 by full-matrix least-squares techniques. The GOF = 0.836 for 549 variables refined to R1 = 0.0305 for 9531 reflections with I>2α(I). Further information is contained in the CIF file.



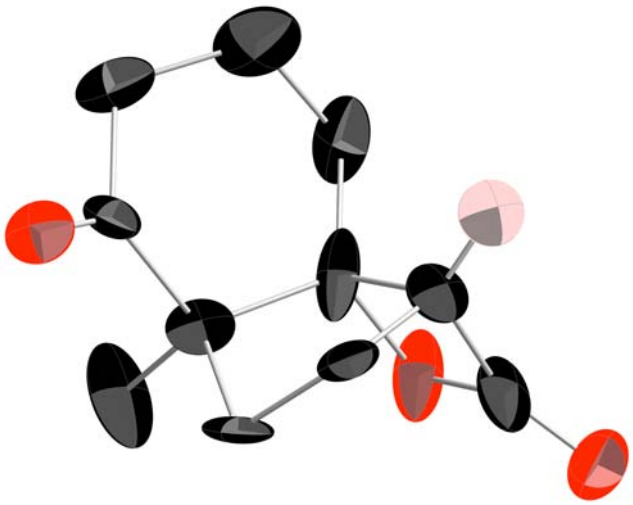
Stereochemical Determination of 12 and 13

Relative stereochemistry of compound **12** was determined by NOE experiment (Inova 500 MHz).



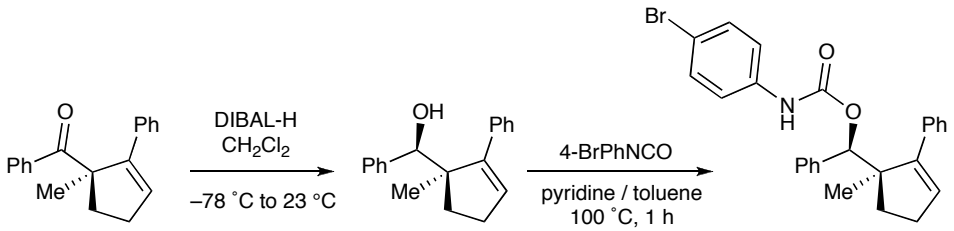
Relative stereochemistry of compound **13** was determined by X-ray diffraction.

X-ray crystal structure **13:** X-ray diffraction was performed at –120 °C and raw frame data were processed using SAINT. Molecular structure was solved using direct methods and refined by F2 by full-matrix least-squares techniques. The GOF = 1.655 for 261 variables refined to R1 = 0.1608 for 3176 reflections with I>2α(I). Further information is contained in the CIF file.



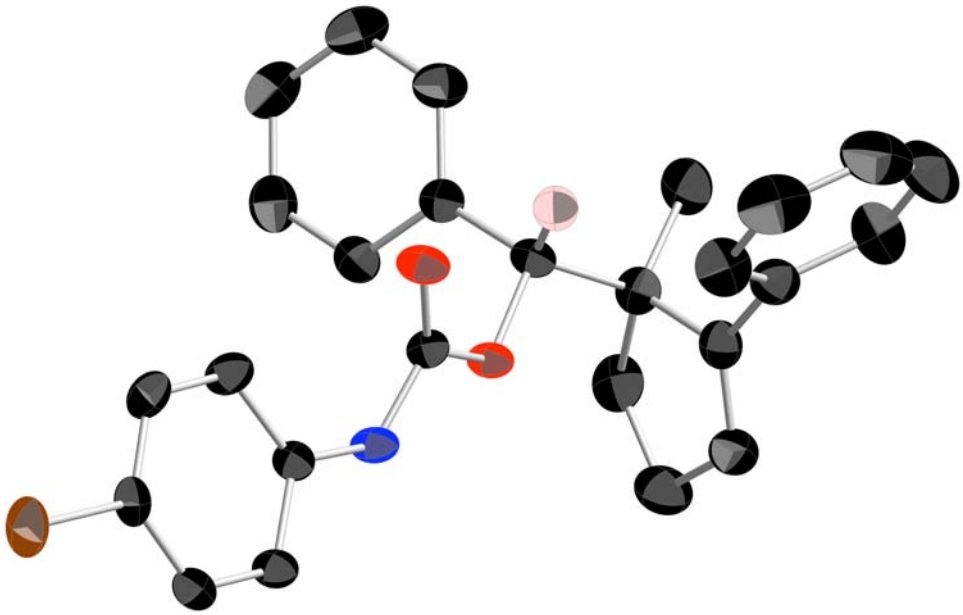
Determination of Absolute Stereochemistry of 4

The absolute stereochemistry of **4** was determined by the X-ray diffraction of a heavy atom derivative, (*R*)-((*R*)-1-methyl-2-phenylcyclopent-2-enyl)(phenyl)methyl 4-bromophenylcarbamate. This compound was synthesized by the method shown below.

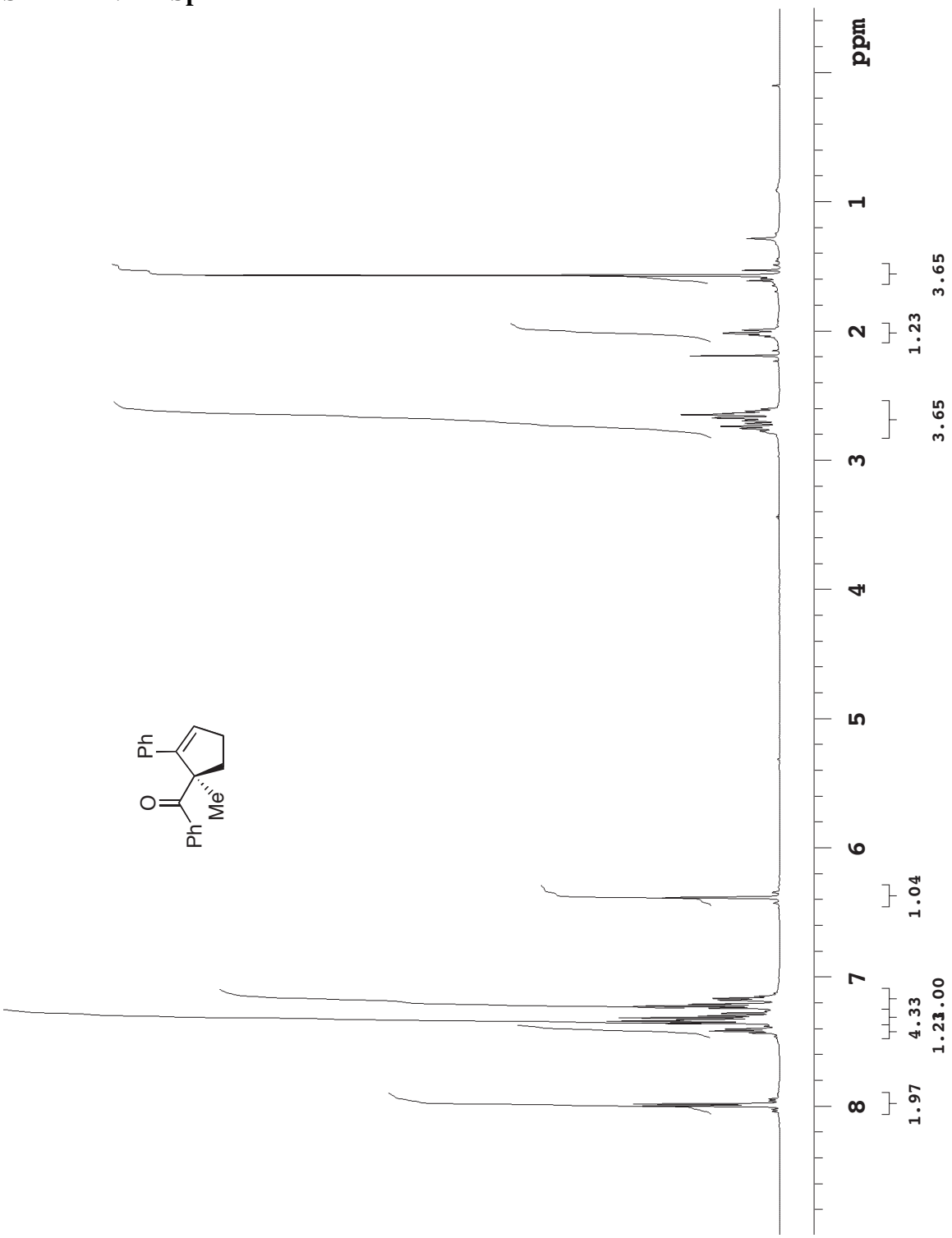


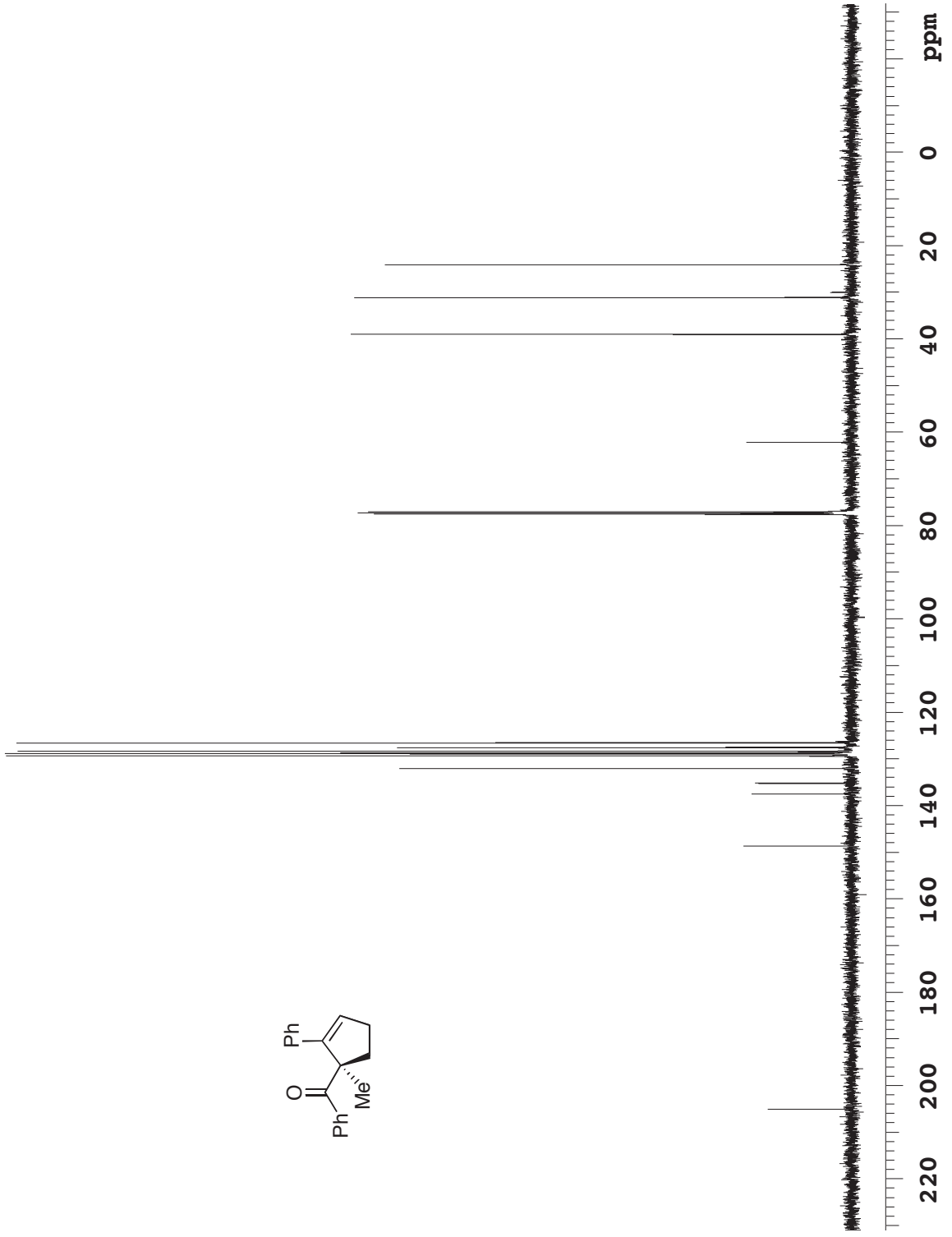
X-ray crystal structure of (*R*)-((*R*)-1-methyl-2-phenylcyclopent-2-enyl)(phenyl)methyl 4-bromophenylcarbamate**:**

X-ray diffraction was performed at –120 °C and raw frame data were processed using SAINT. Molecular structure was solved using direct methods and refined by F2 by full-matrix least-squares techniques. The GOF = 0.836 for 549 variables refined to R1 = 0.0305 for 9531 reflections with I>2α(I). Further information is contained in the CIF file.



Selected NMR Spectra

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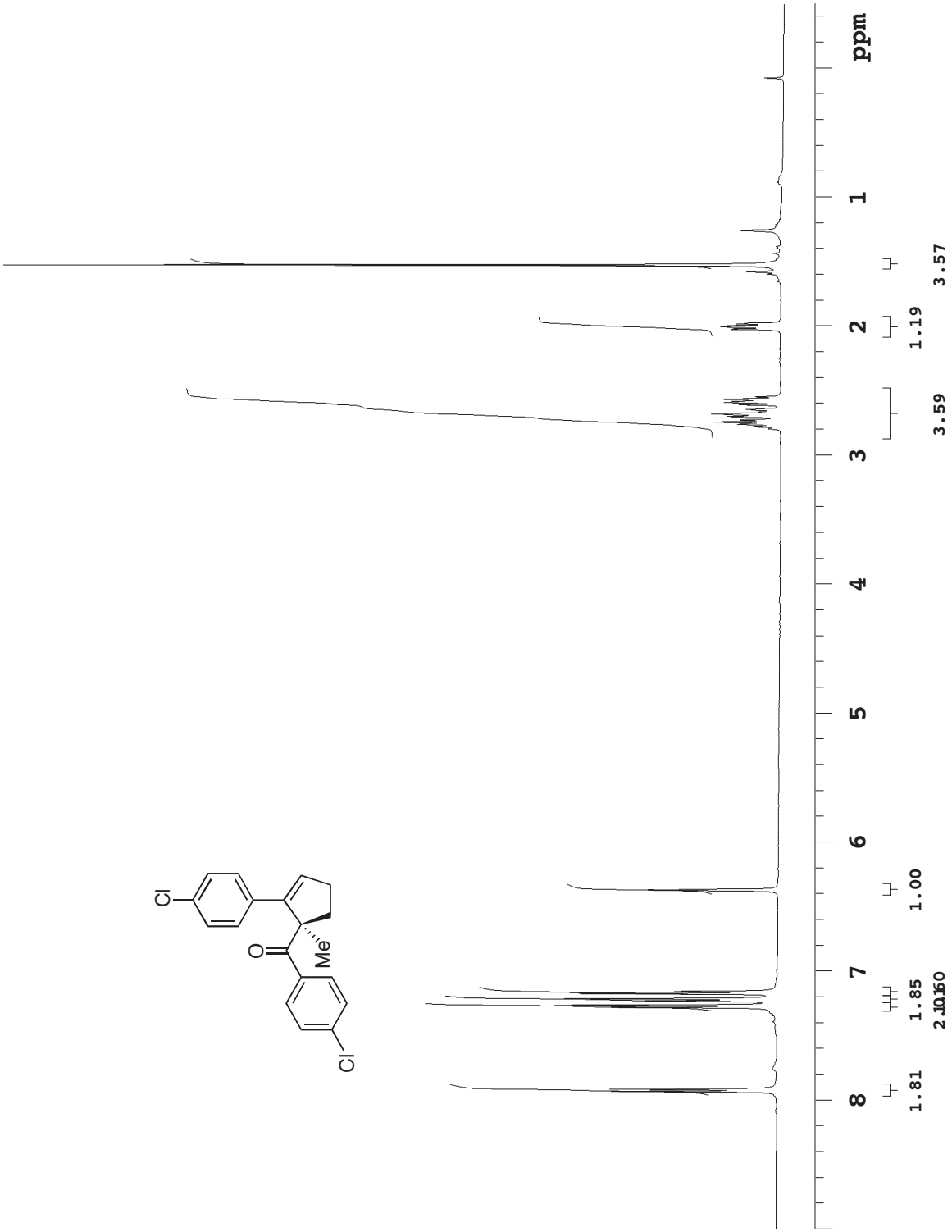
100
120
140
160
180
200
220
Ph
O
Me
Ph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O
Me
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ppm
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3
4
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6
7
8
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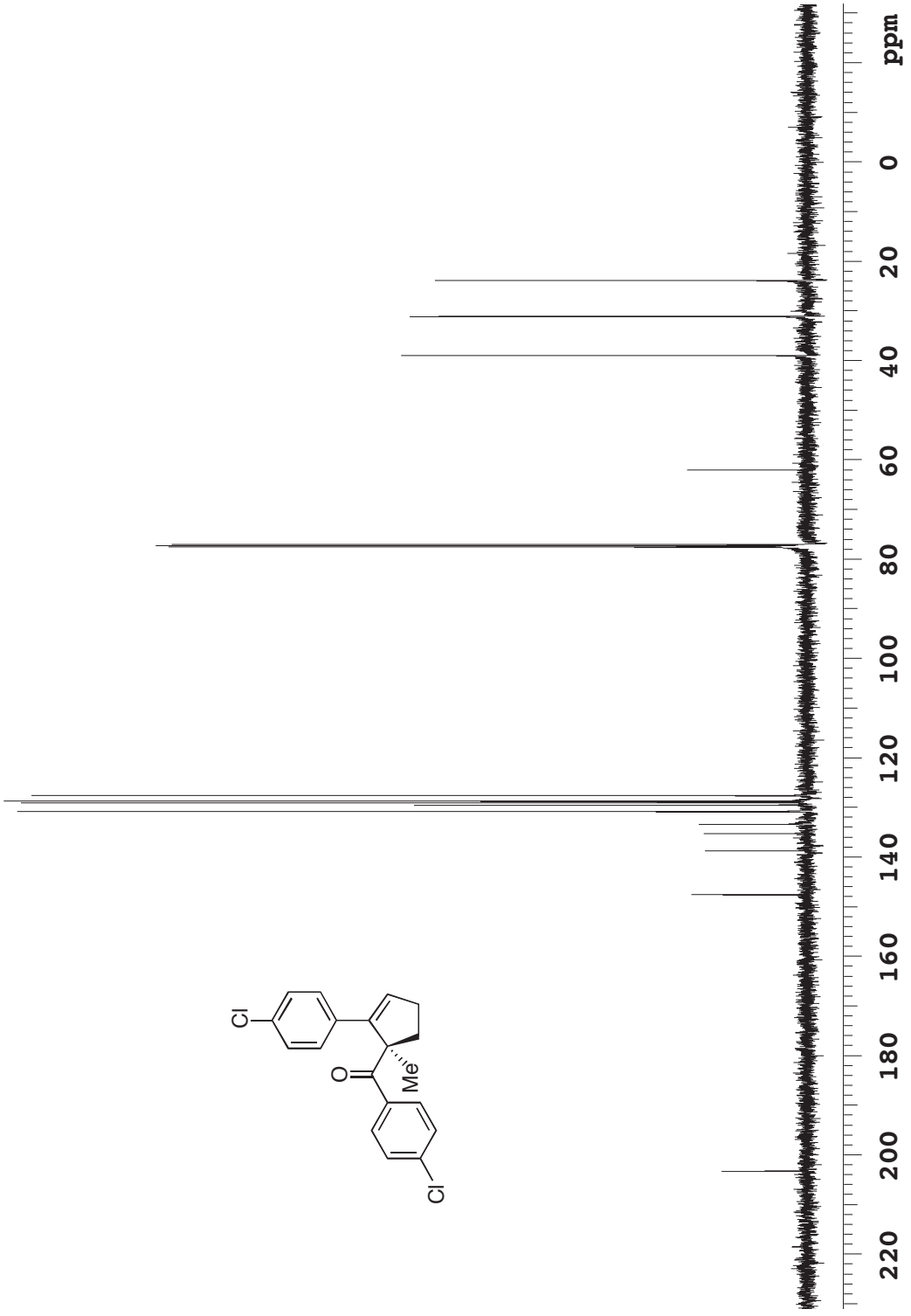
3.59
1.00
1.85
1.81


ppm
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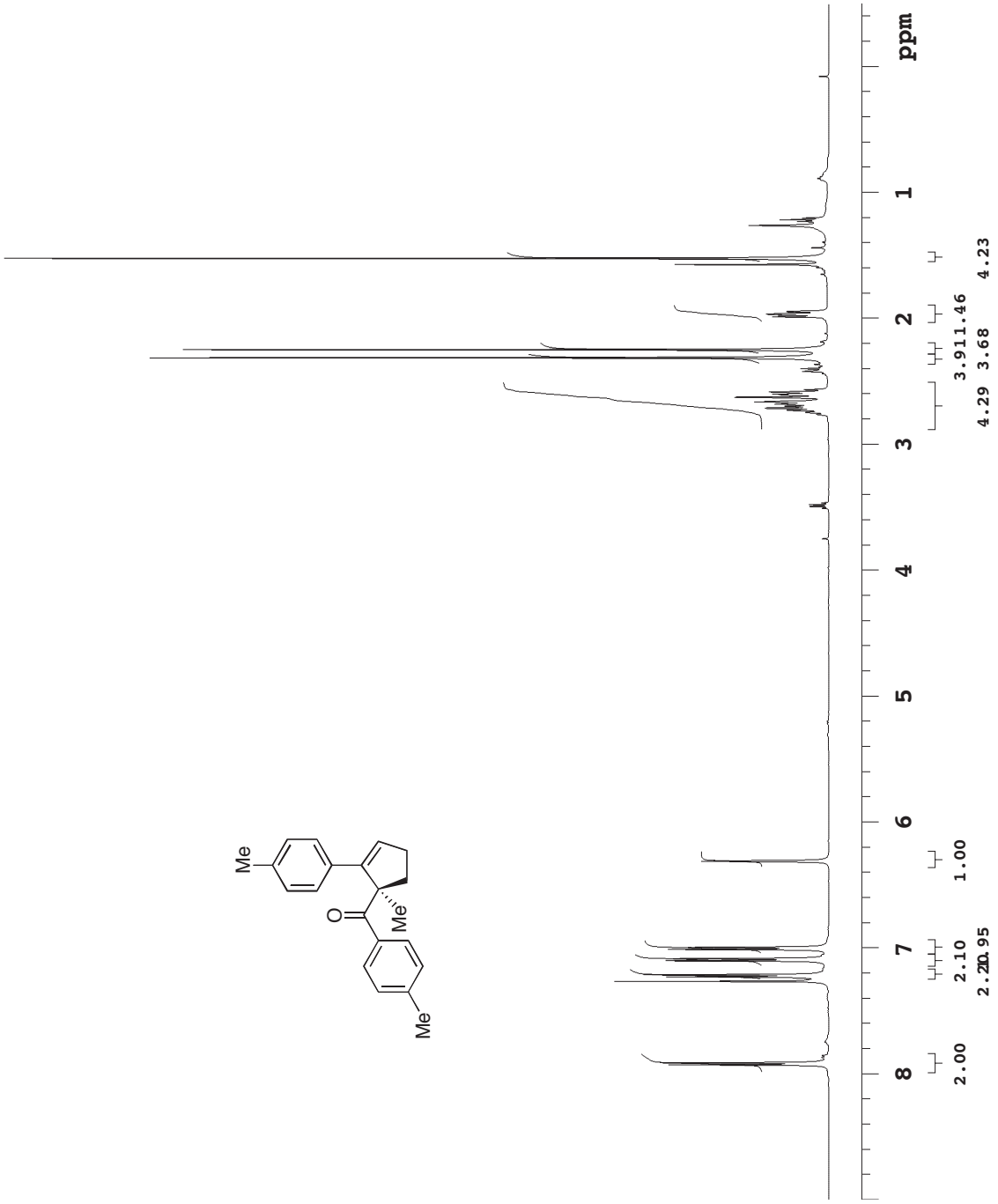
100
120
140
160
180
200
220
Cl
O
Me
Cl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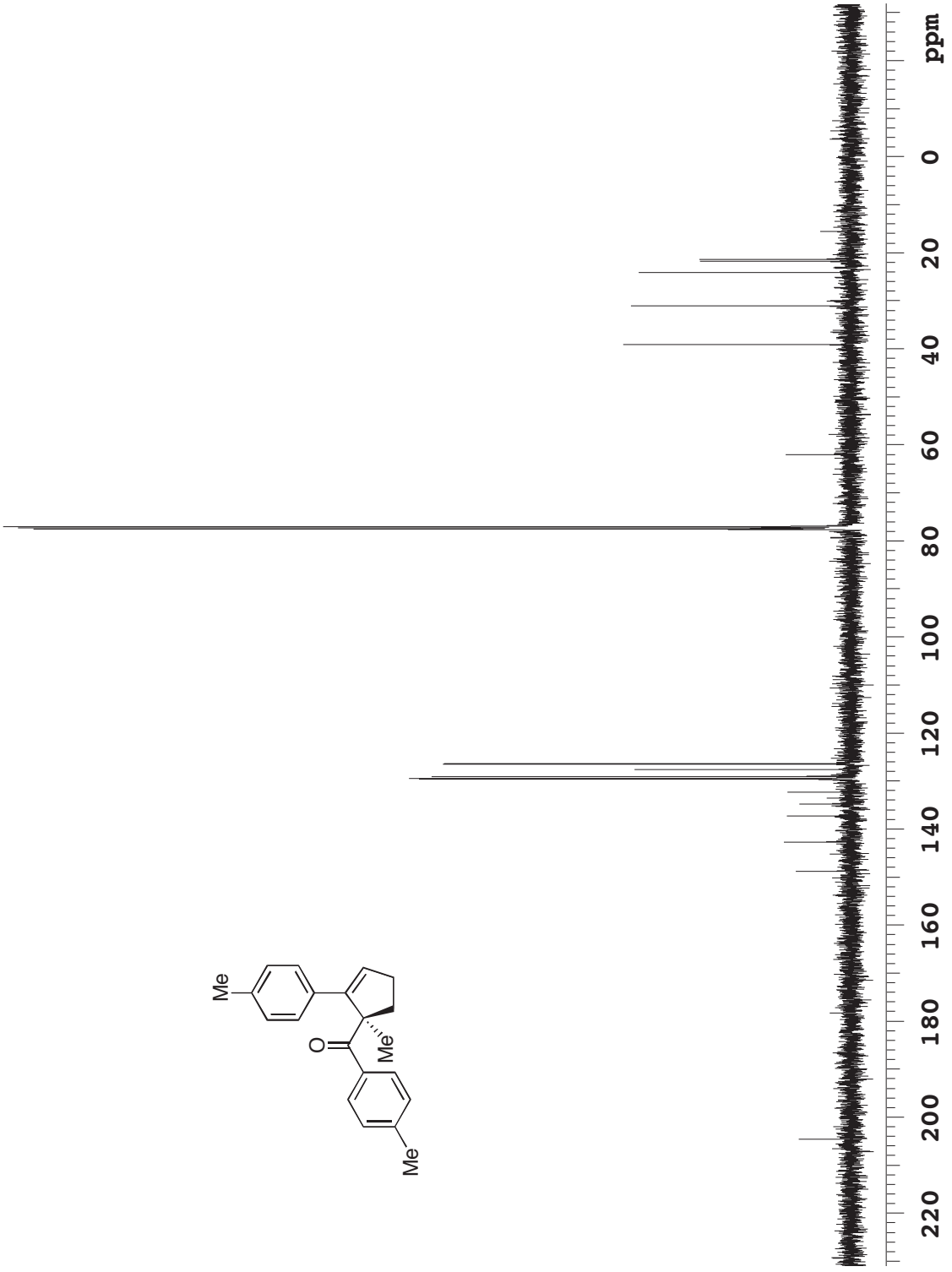
1.00
2.10
2.20
0.95
2.00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O
Me
Me
ppm
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20
40
60
80
100
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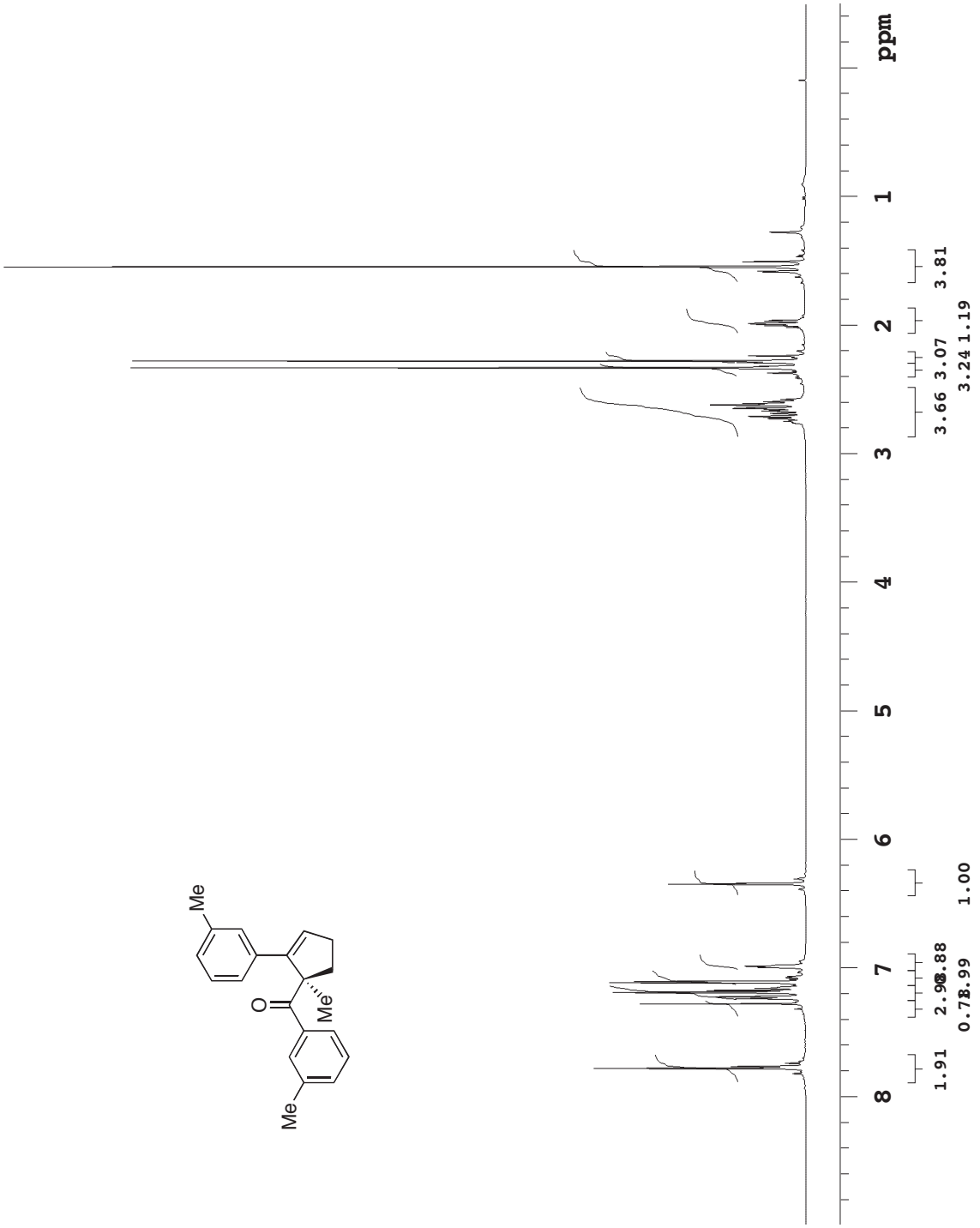
160
180
200
220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ppm
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3
4
5
6
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3.07
3.24
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1.00
1.91
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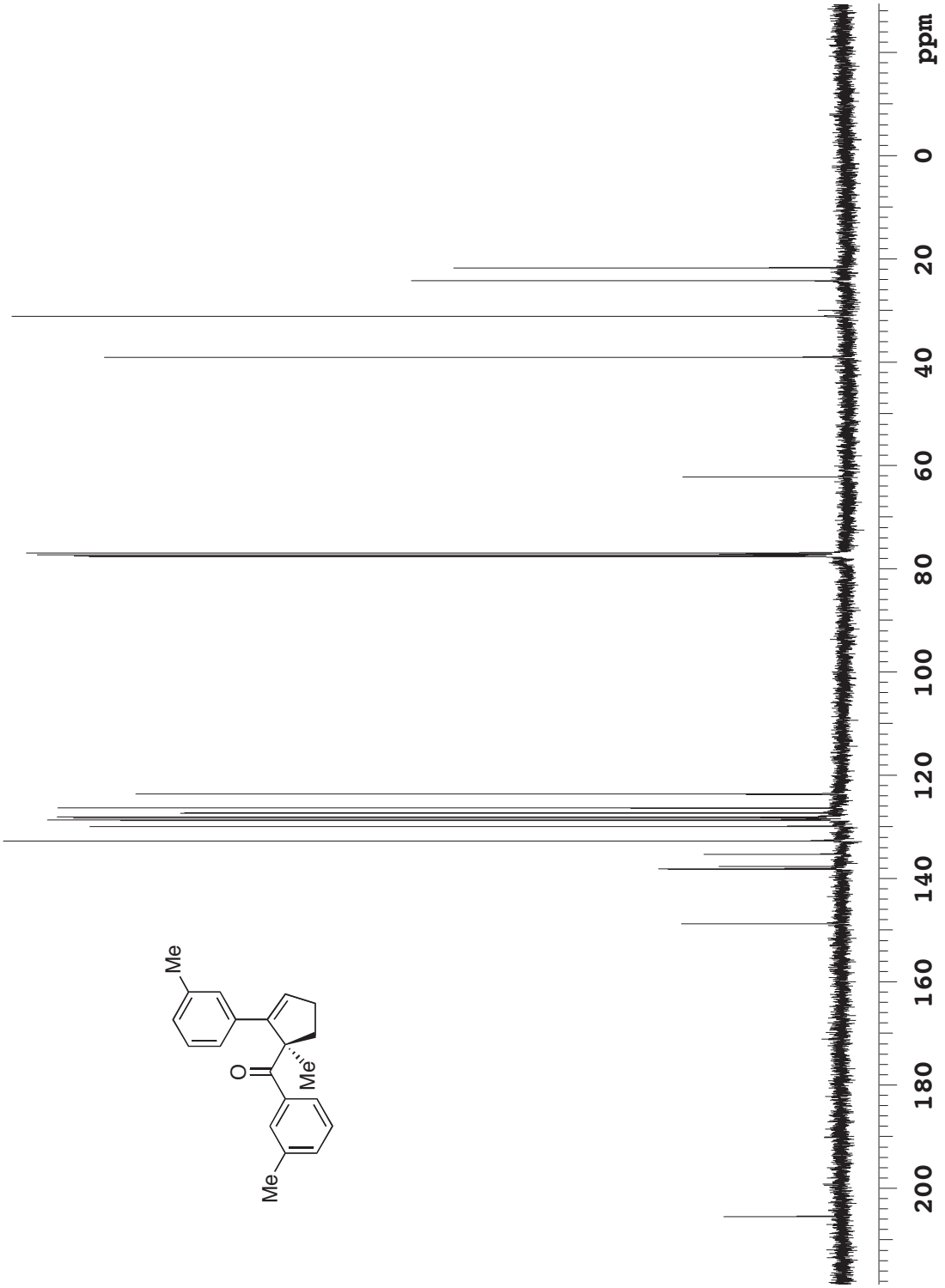
8.88
0.78
8.99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Me
O
Me
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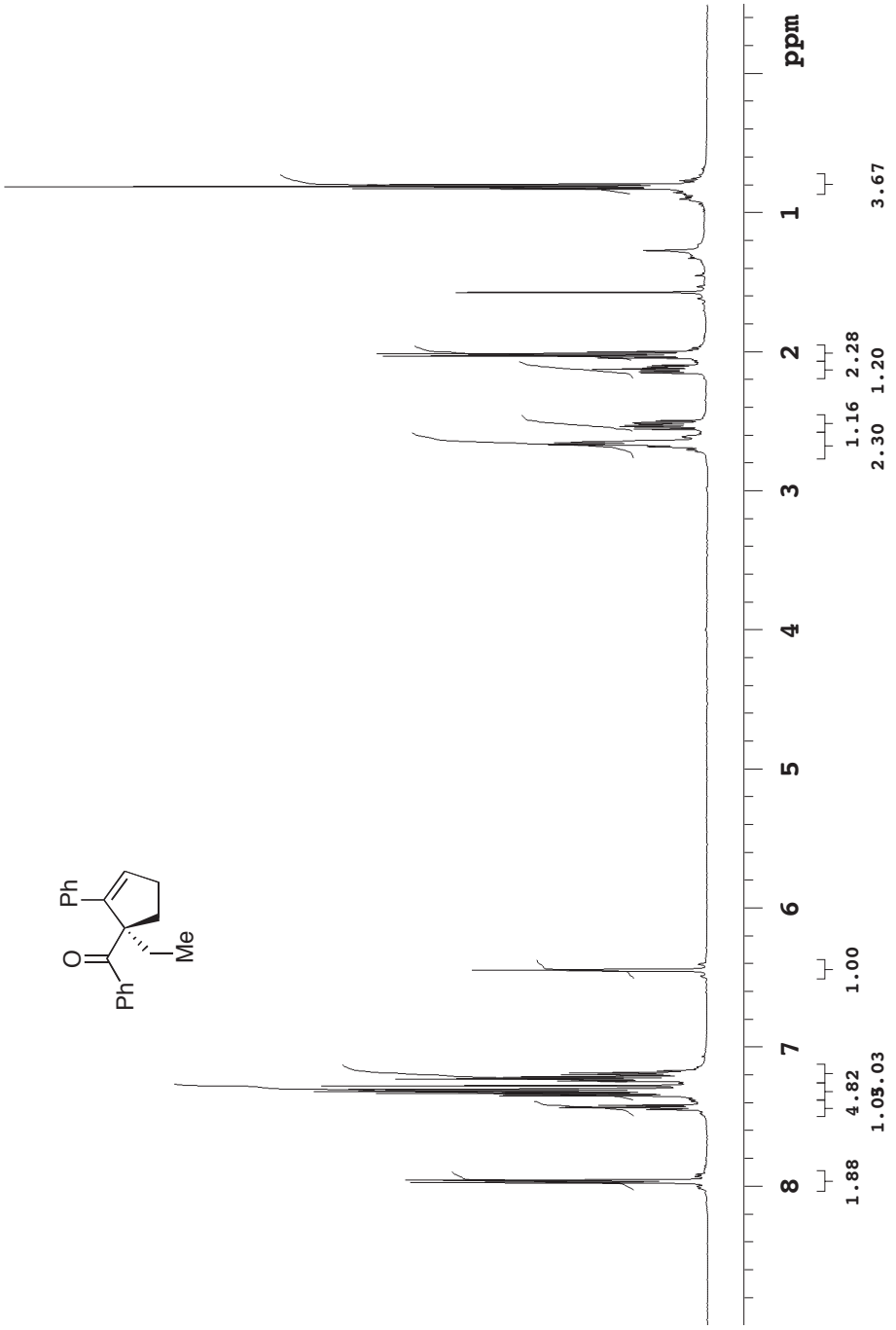
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Ph
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ppm
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2
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6
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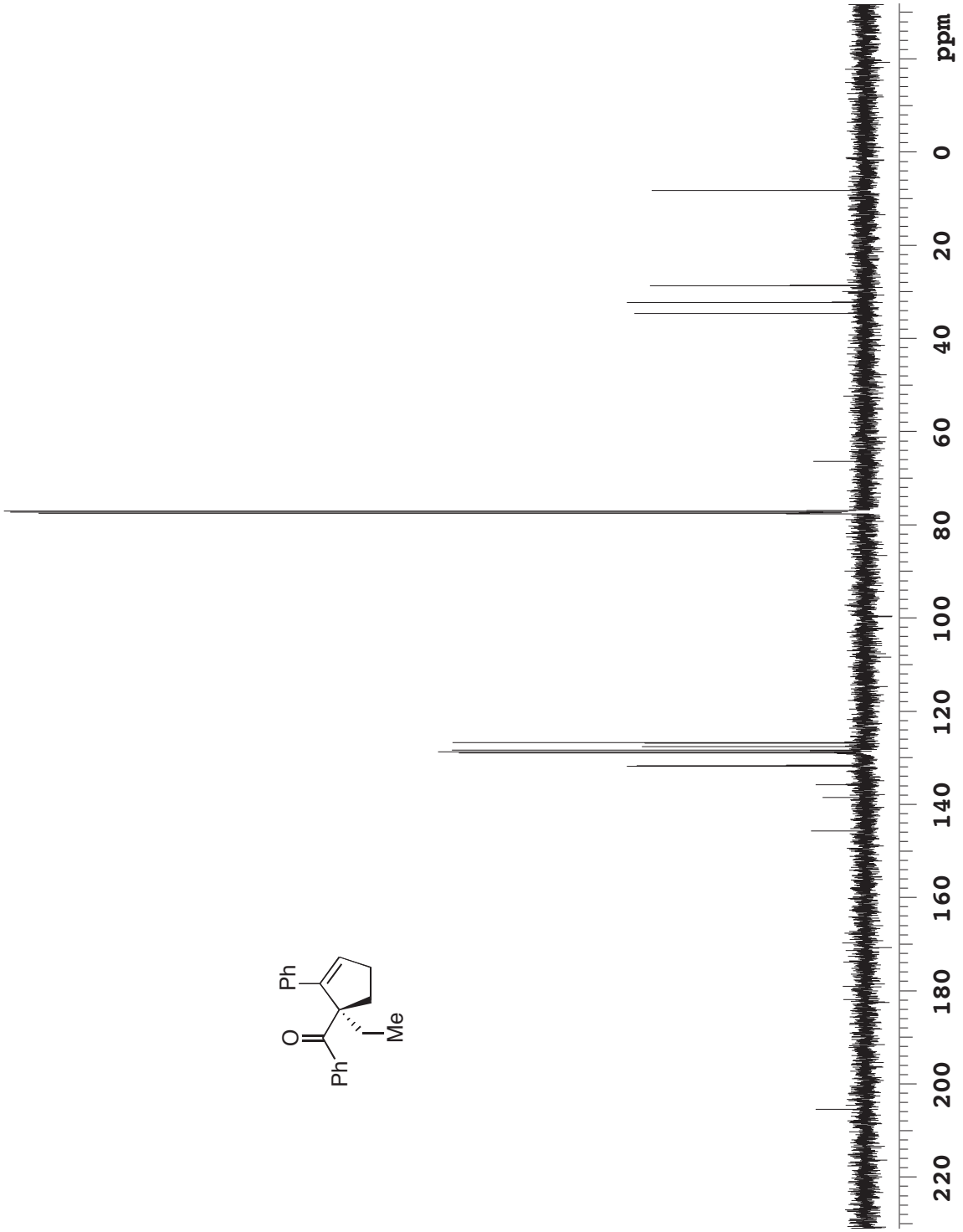
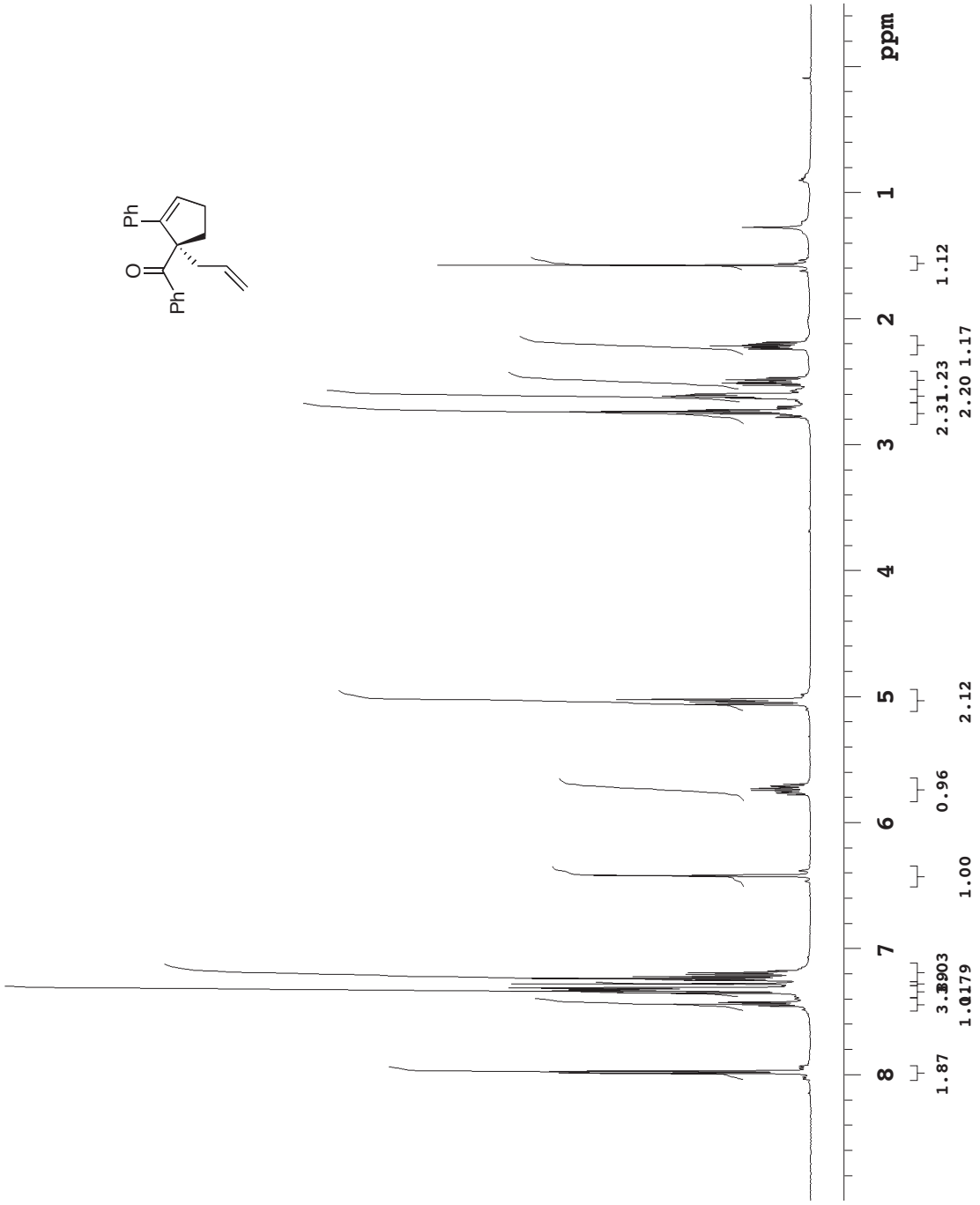
1.16
2.30
1.00
4.82
1.05.03
1.88


Ph
O
Ph
Me
ppm
0
20
40
60
80
100
120
140
160
180
200
220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O
Ph
ppm
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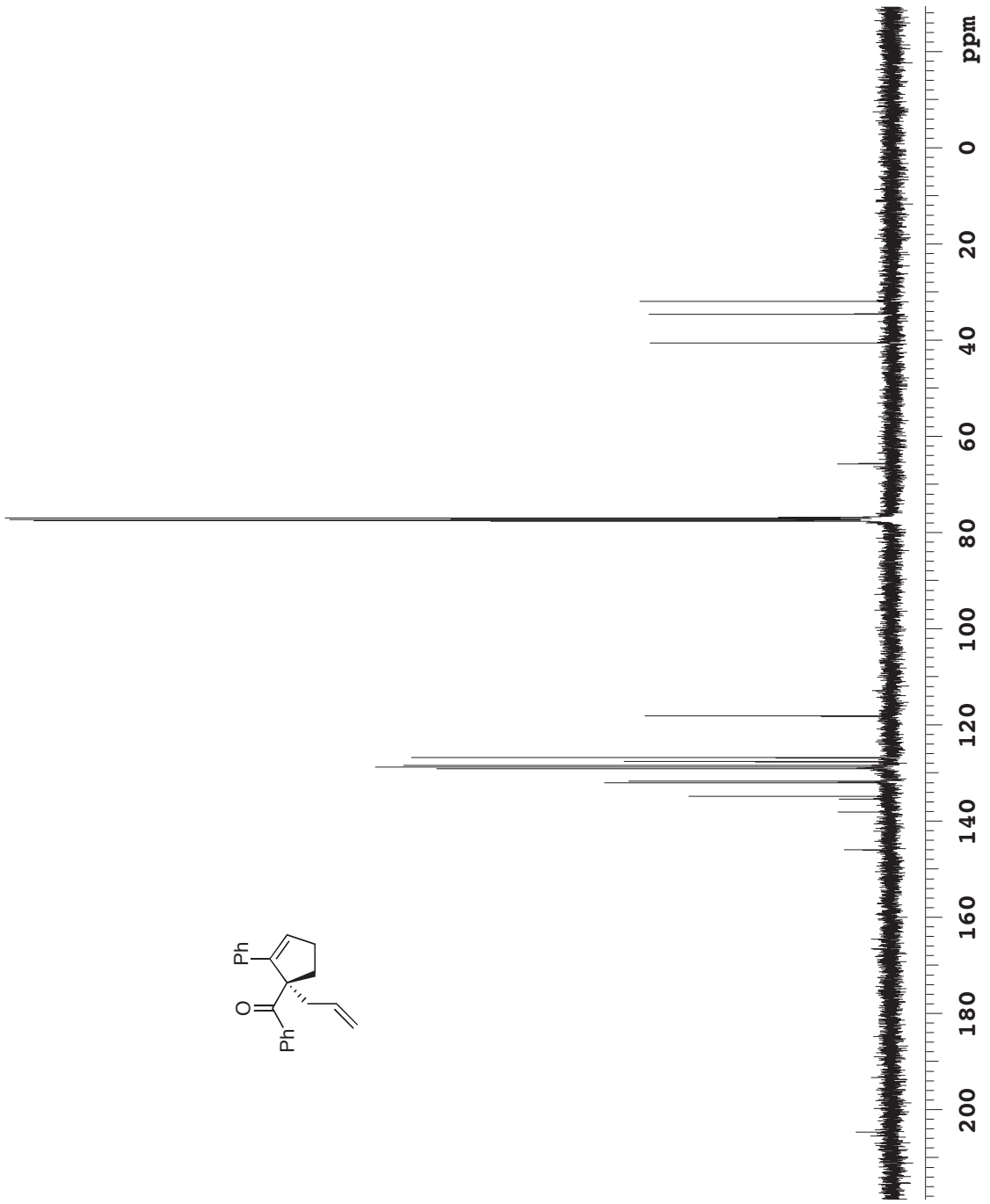
2
3
4
5
6
7
8
1.12
2.31 2.20 1.17
2.12
0.96
1.00
1.87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Ph
ppm
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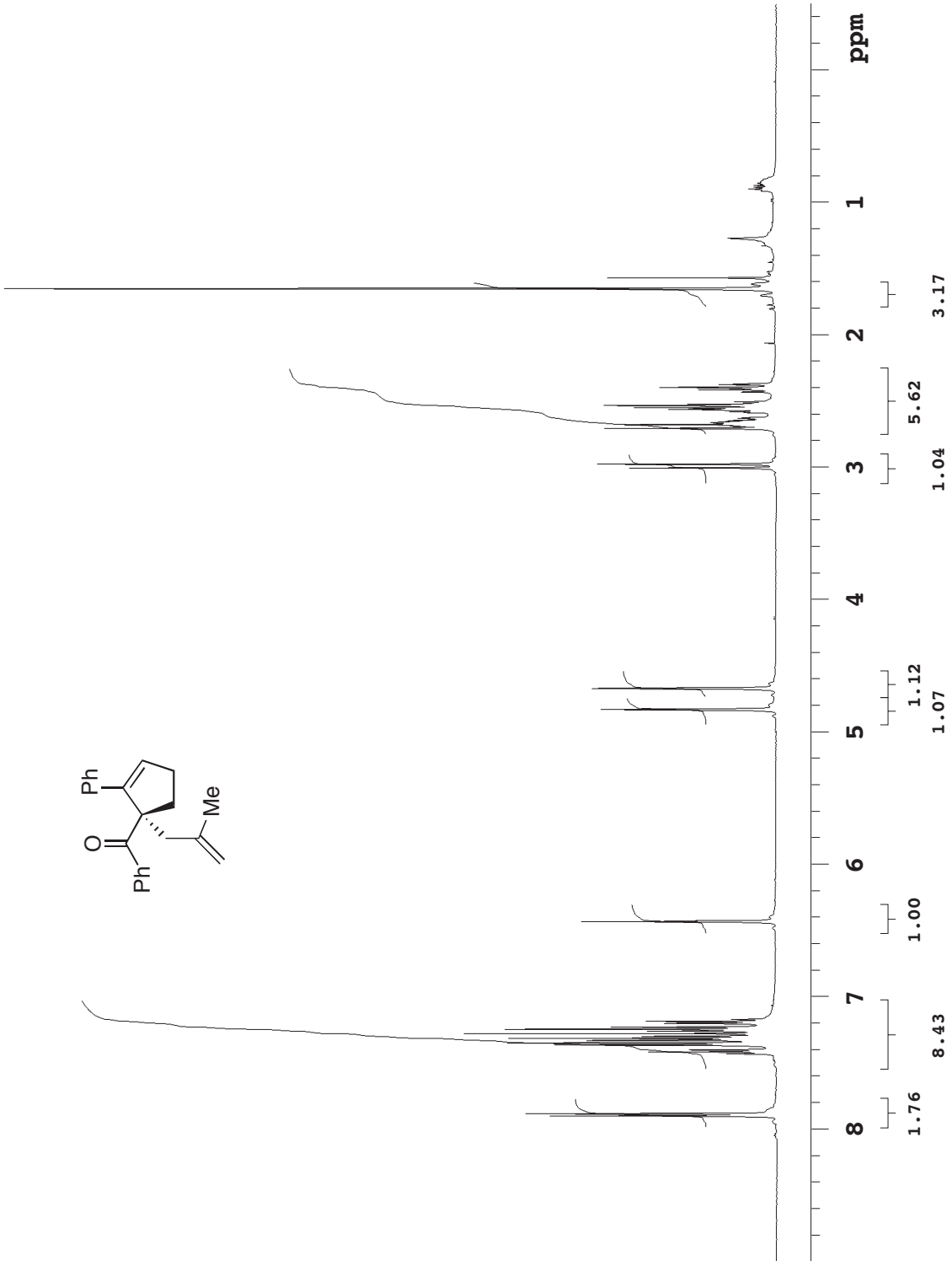
180
200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Ph
Me
ppm
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6
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1.12
1.07
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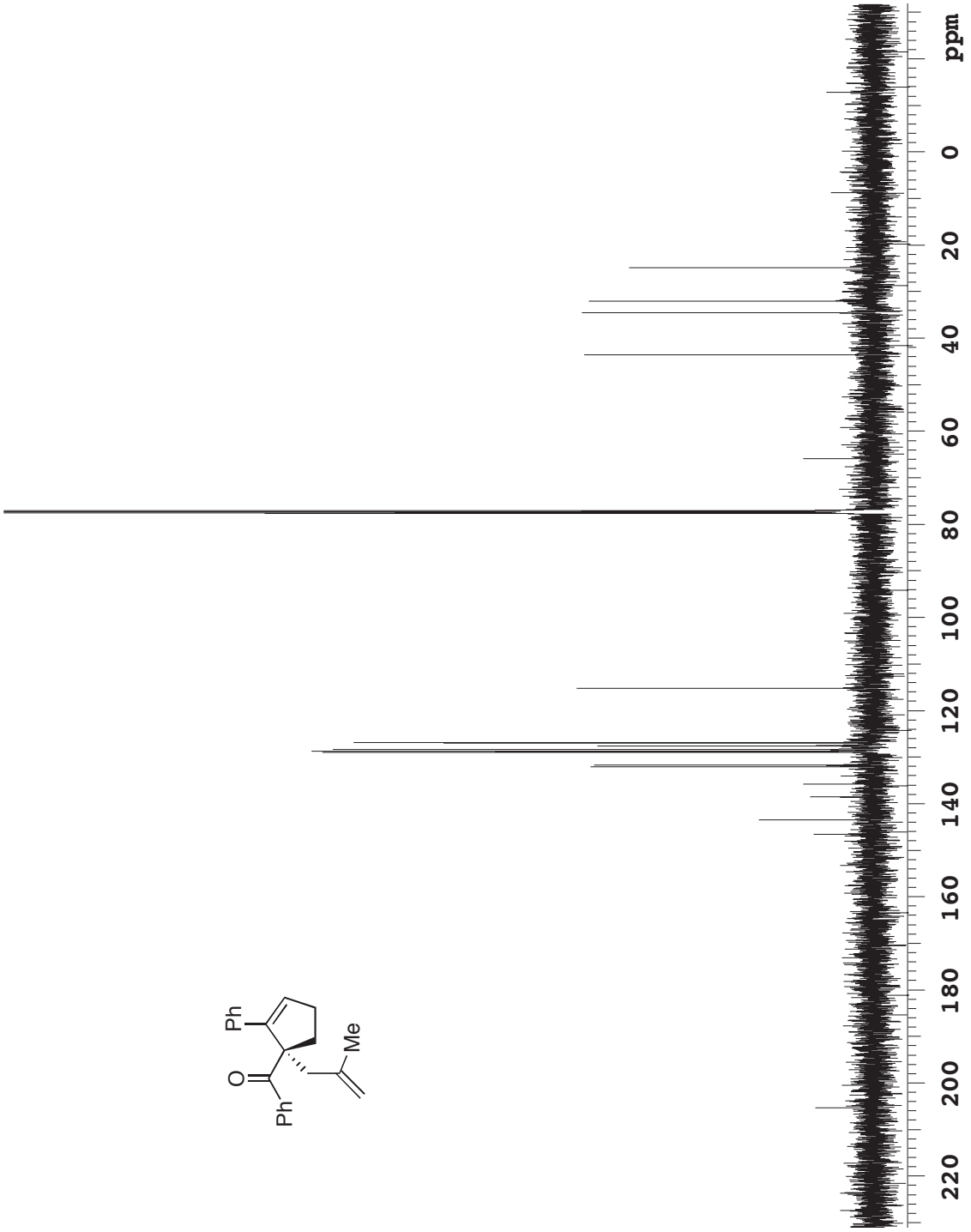
1.76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120
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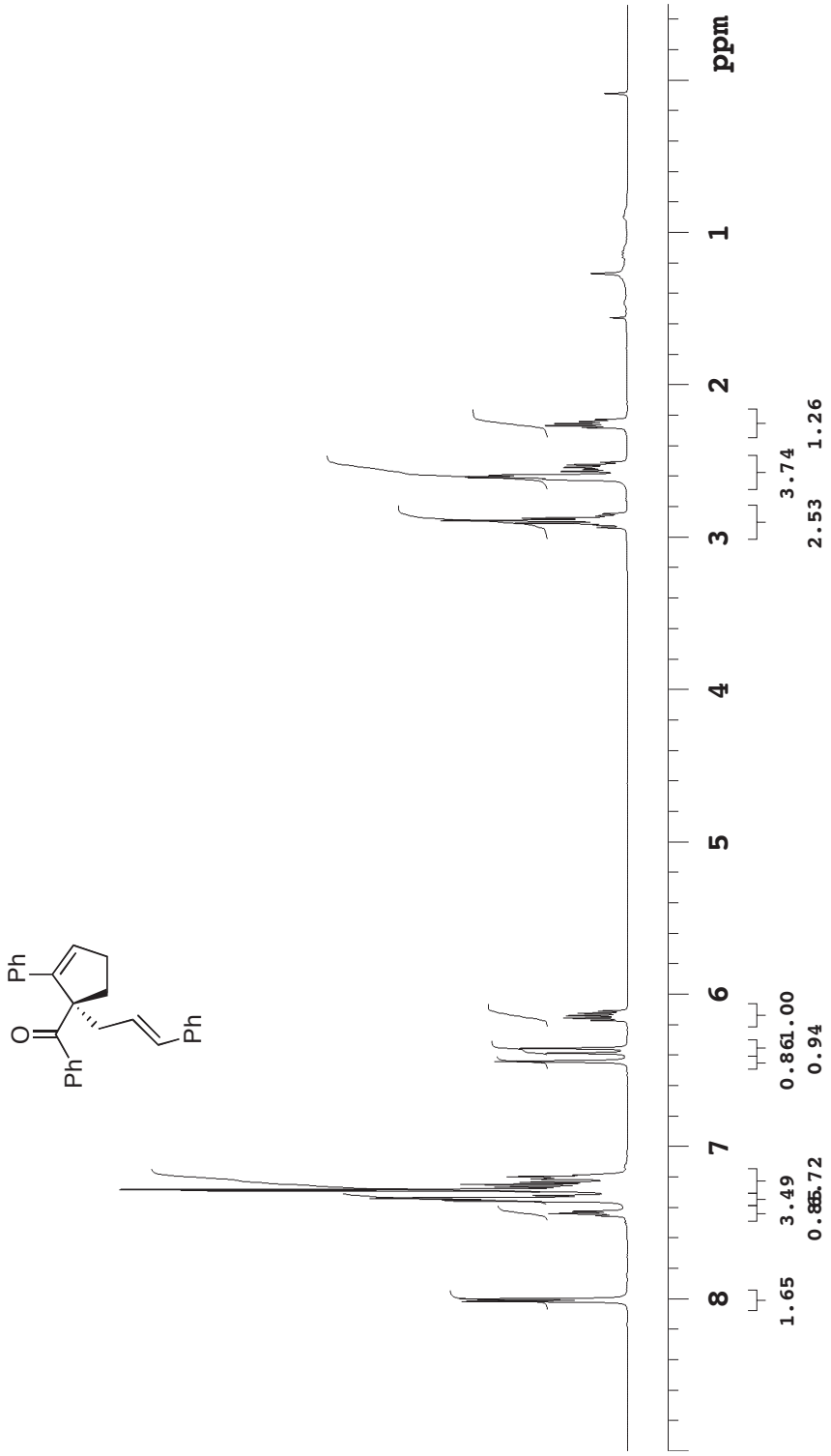
160
180
200
220
Ph
O
Ph
Me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Ph
ppm
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6
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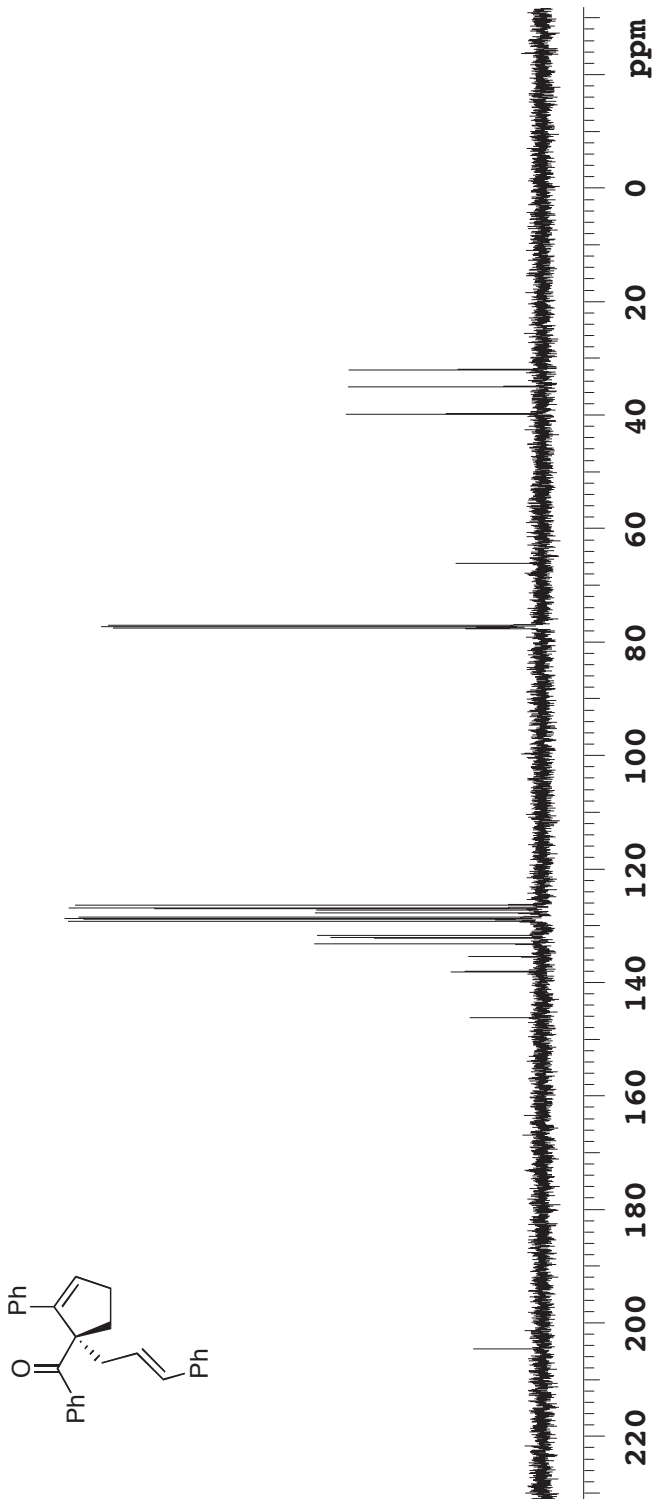
0.86
3.49
1.65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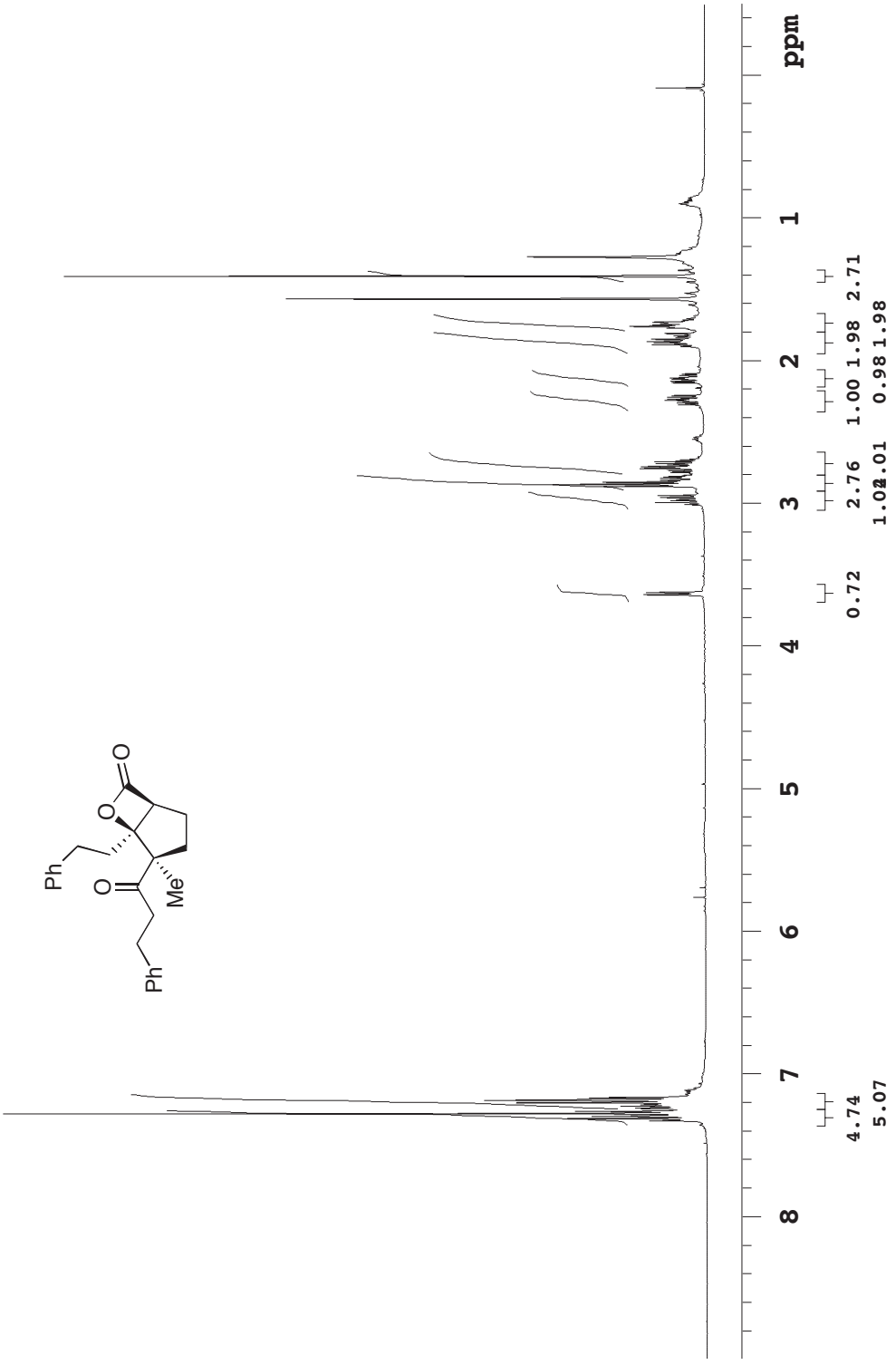
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0.98
1.98
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5.07
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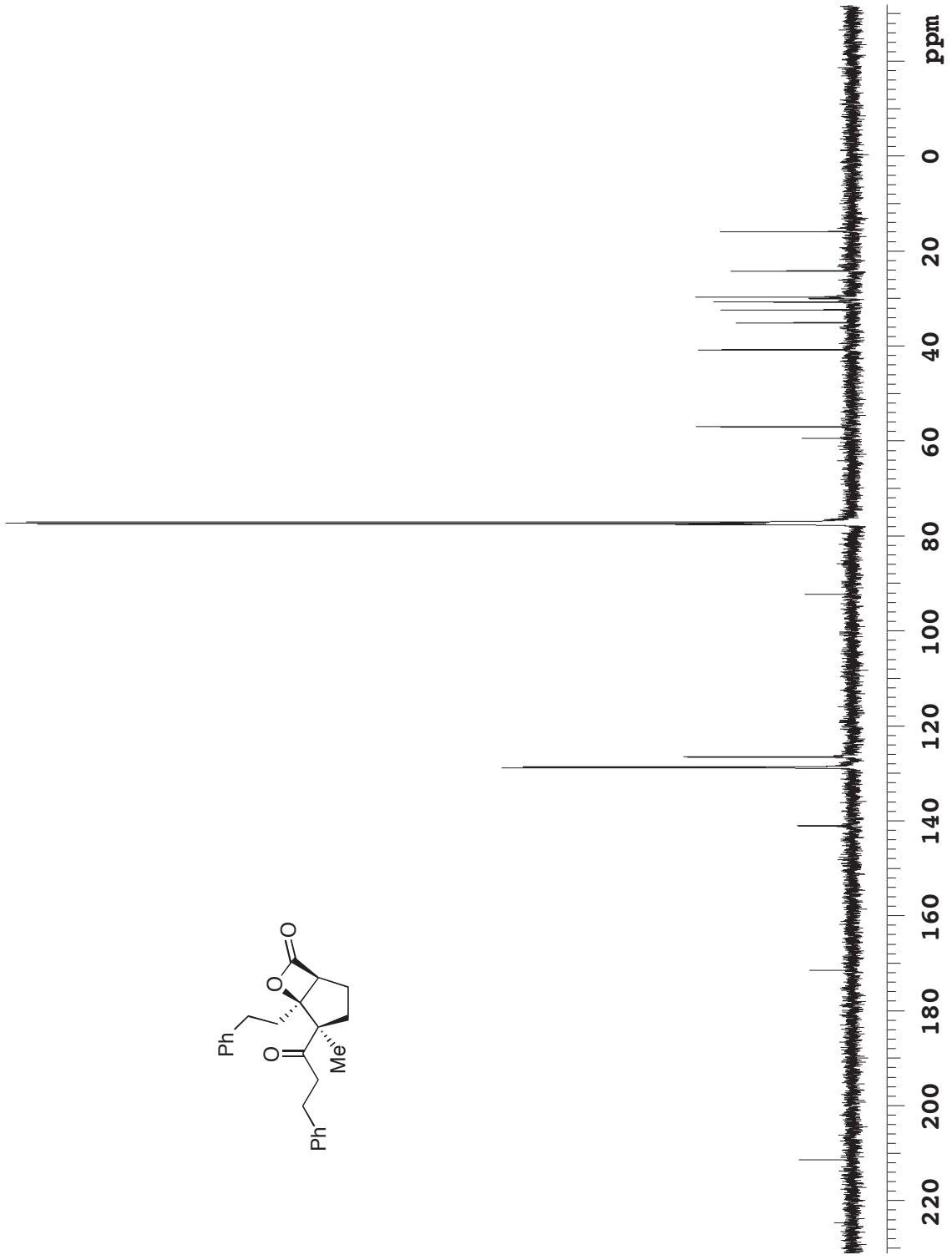
O
O
Ph
Me
Ph


O
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O
Me
Ph
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40
20
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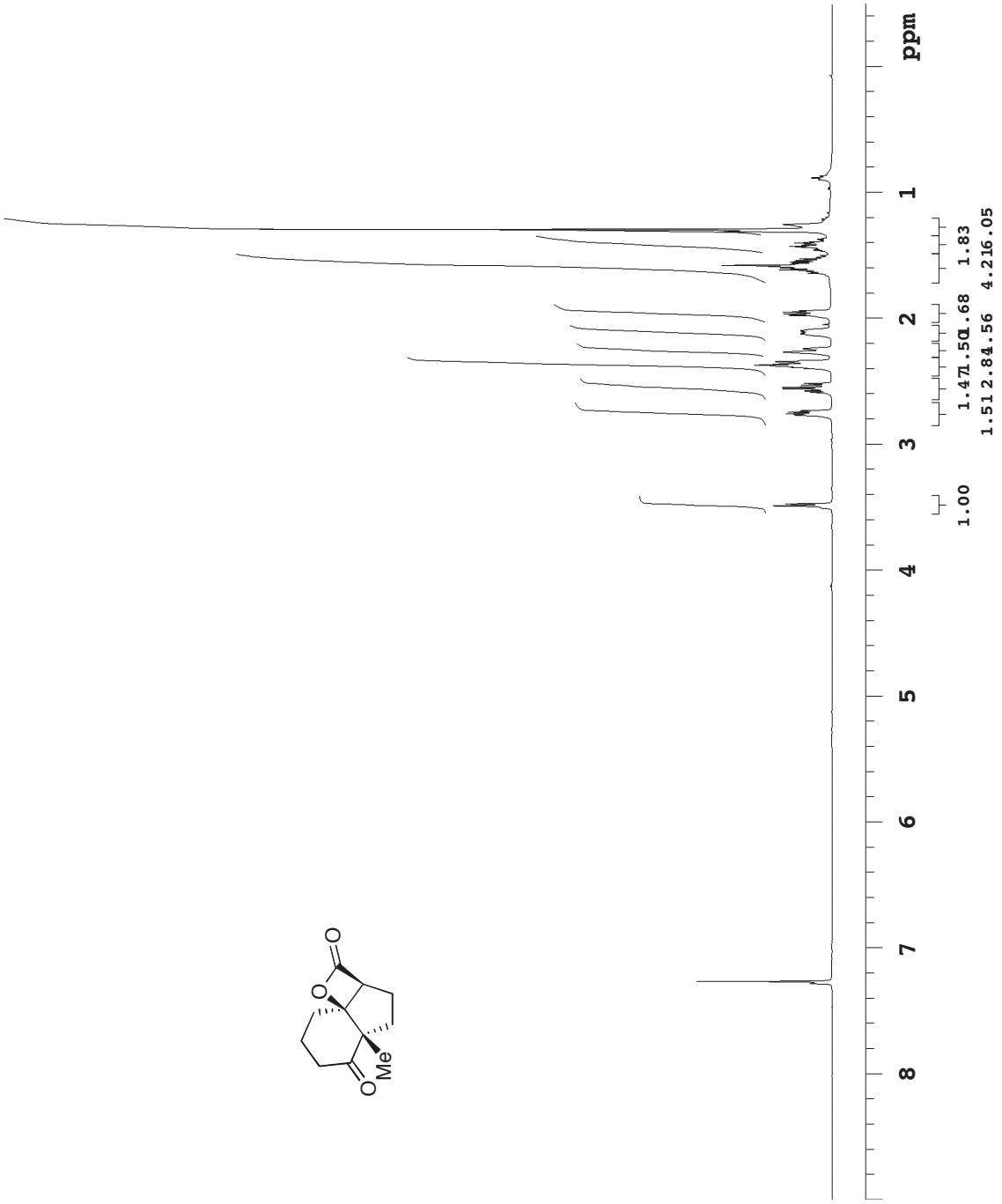
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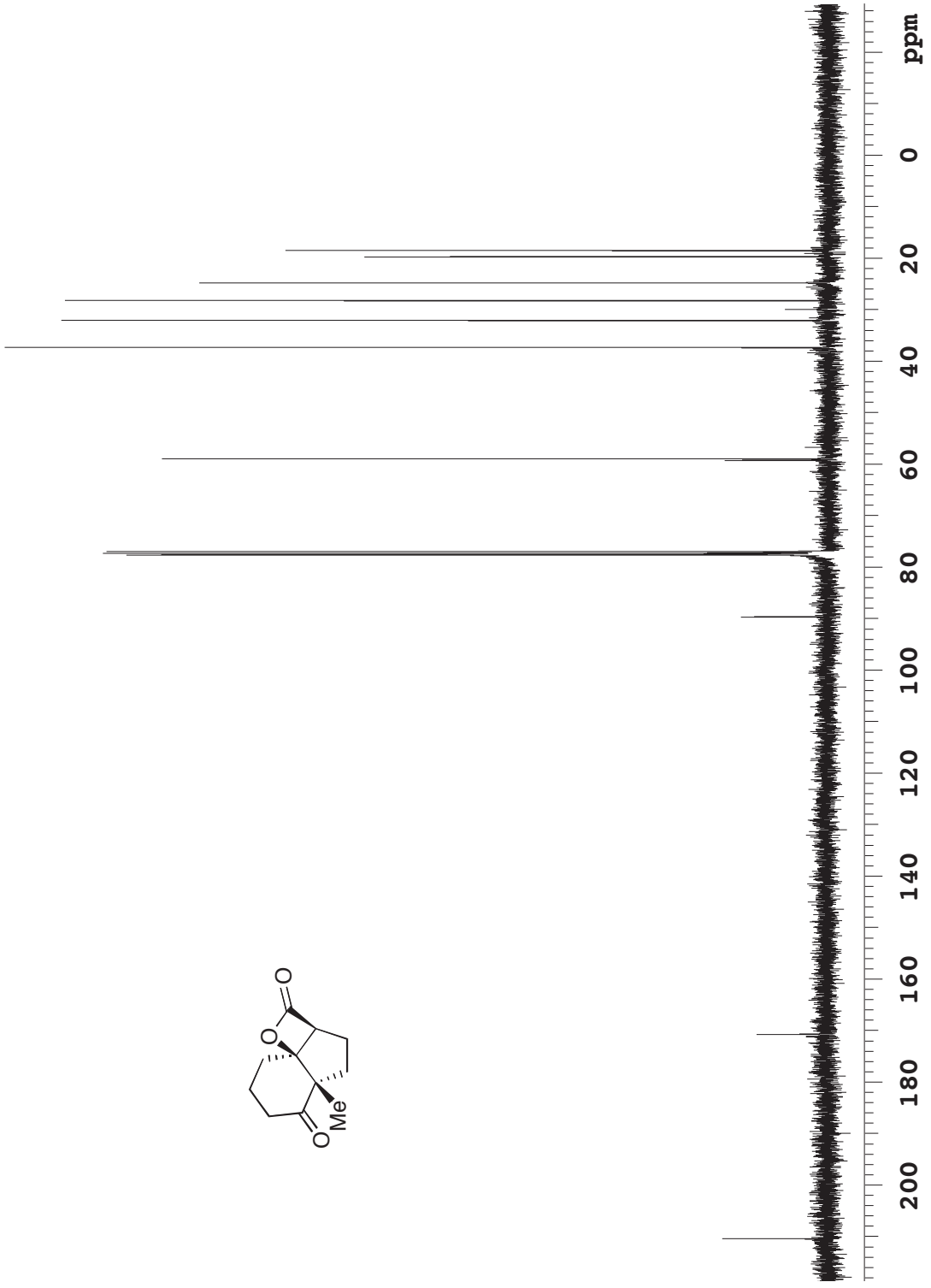
4.21
6.05
O
O
O
Me


ppm
0
20
40
60
80
100
120
140
160
180
200
O
O
O
Me

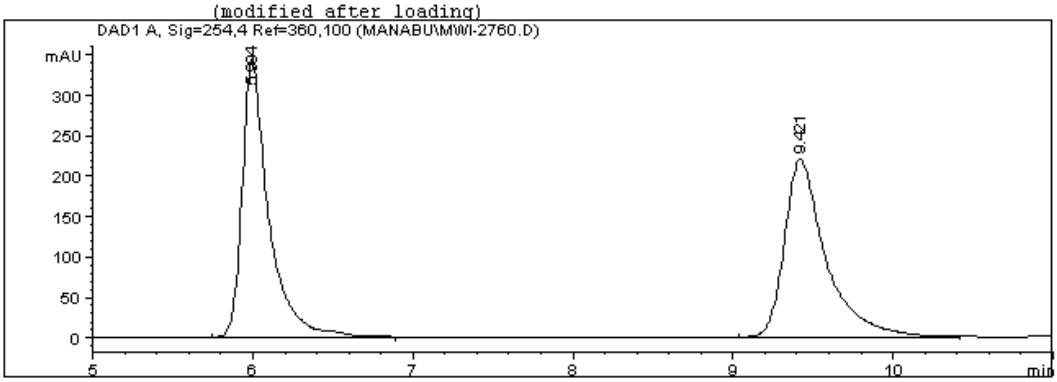
HPLC and GC Traces

Racemic 4

Data File C:\HPCHEM\2\DATA\MANABU\MWI-2760.D Sample Name: MWI-276

ee

```
======================================================================
Injection Date  : 12/29/2006 12:28:10 PM
Sample Name     : MWI-276                        Location : Vial 77
Acq. Operator   : MANABU
                                                Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed    : 12/28/2006 11:29:35 AM by MANABU
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\MMB LC.M
Last changed    : 5/14/2007 9:54:04 PM by Rob
                  (modified after loading)
```




```
======================================================================
                          Area Percent Report
======================================================================

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


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.994 BB    0.1680 4000.78589  343.31052  50.0895
   2   9.421 BB    0.2626 3986.49365  220.86250  49.9105

Totals :                  7987.27954  564.17302

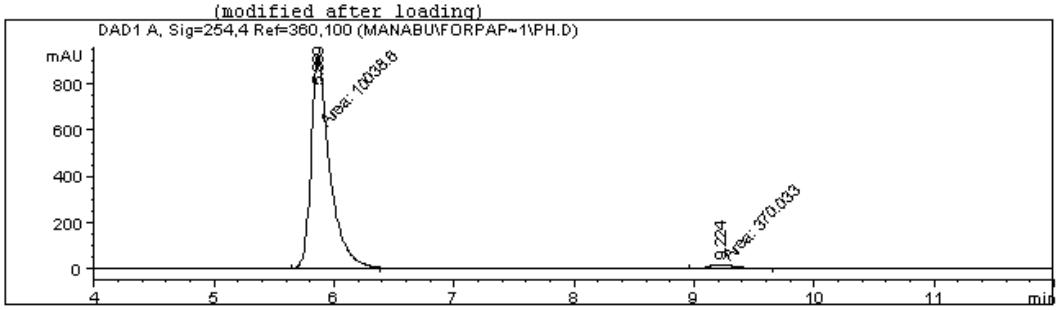
 Results obtained with enhanced integrator!
======================================================================
                          *** End of Report ***
```

Enantioenriched 4

Data File C:\HPCHEM\2\DATA\MANABU\FORPAP~1\PH.D Sample Name: MWII-32

diphenyl

```
==========================================================================
Injection Date  : 1/26/2007 7:37:18 PM
Sample Name     : MWII-32                          Location : Vial 72
Acq. Operator   : MANABU
                                                  Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed    : 1/26/2007 7:36:16 PM by MANABU
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 6/1/2007 1:28:22 PM by MANABU
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

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


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.869 MM    0.1813 1.00386e4   922.93201  96.4449
   2   9.224 MM    0.3216  370.03271   19.17678   3.5551

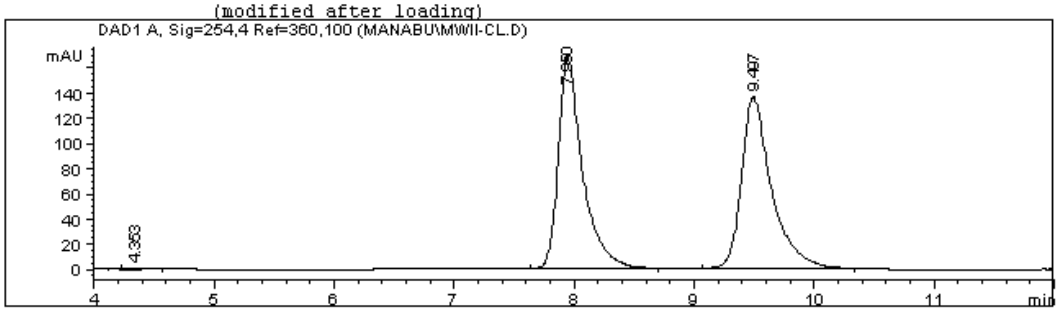
Totals :                  1.04086e4   942.10879

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Racemic 5

Data File C:\HPCHEM\2\DATA\MANABU\MWII-CL.D Sample Name: MWII-Cl

```
==================================================================
Injection Date  : 5/30/2007 3:25:00 AM
Sample Name     : MWII-Cl                      Location : Vial 2
Acq. Operator   : MANABU
                                              Inj Volume : 2 µl
Method          : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 5/30/2007 3:38:21 AM by MANABU
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

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


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   4.353 PB    0.1272    9.79792    1.08663   0.2041
   2   7.950 BB    0.2051 2387.78174  168.73271  49.7389
   3   9.497 BB    0.2548 2403.04907  136.91711  50.0570

Totals :                   4800.62873  306.73646

 Results obtained with enhanced integrator!
==================================================================
                        *** End of Report ***
```

Enantioenriched 5

Data File C:\HPCHEM\2\DATA\MANABU\CL-N0006.D Sample Name: CL-nl

```
==========================================================================
Injection Date  : 6/1/2007 11:46:05 AM
Sample Name     : CL-nl                          Location : Vial 3
Acq. Operator   : MANABU
                                                 Inj Volume : 2 µl
Acq. Method     : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 6/1/2007 10:49:08 AM by MANABU
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 6/1/2007 1:26:53 PM by MANABU
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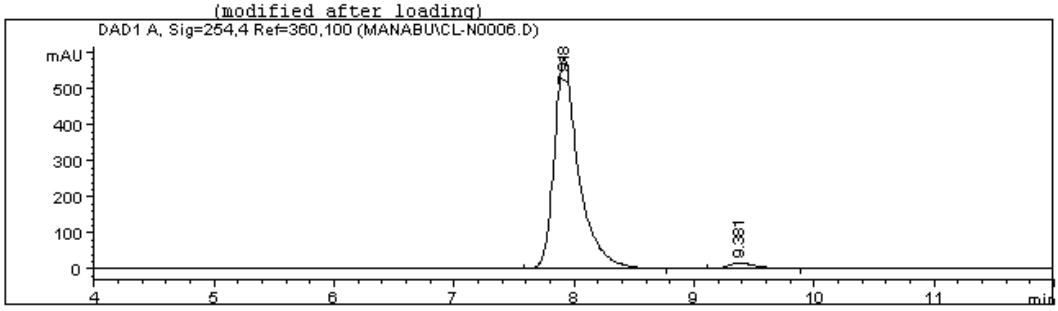
```




```
==========================================================================
                          Area Percent Report
==========================================================================

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


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.918 BB    0.2050 8338.28906  589.60474  97.0219
   2   9.381 BB    0.2431  255.94324   15.30842   2.9781

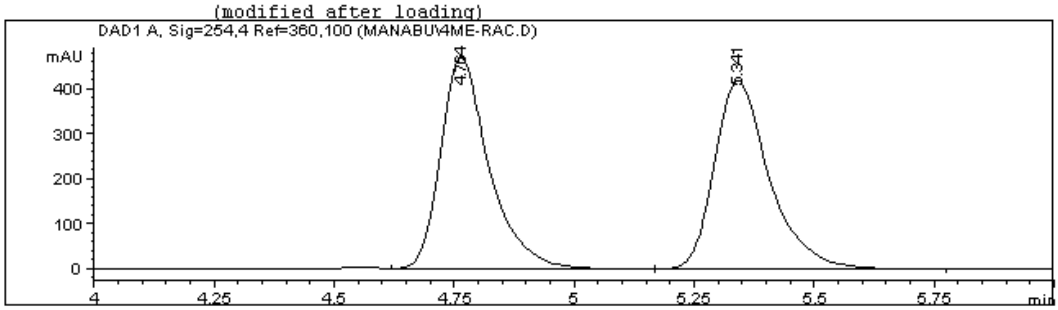
Totals :                   8594.23230  604.91316

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Racemic 6

Data File C:\HPCHEM\2\DATA\MANABU\4ME-RAC.D Sample Name: 4Me-rac

```
==========================================================================
Injection Date  : 5/26/2007 5:59:16 PM
Sample Name     : 4Me-rac                          Location : Vial 2
Acq. Operator   : MANABU
                                                  Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 5/26/2007 5:58:14 PM by MANABU
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 5/26/2007 6:17:26 PM by MANABU
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

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


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   4.764 VV    0.1050 3371.96973  473.94806  50.0800
   2   5.341 VB    0.1214 3361.20166  419.86703  49.9200

Totals :                  6733.17139  893.81509

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Enantioenriched 6

Data File C:\HPCHEM\2\DATA\MANABU\4ME.D Sample Name: 4Me

```
==========================================================================
Injection Date  : 5/26/2007 6:17:38 PM
Sample Name     : 4Me                              Location : Vial 3
Acq. Operator   : MANABU
                                                  Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 5/26/2007 6:27:01 PM by MANABU
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 5/26/2007 6:32:31 PM by MANABU
                  (modified after loading)
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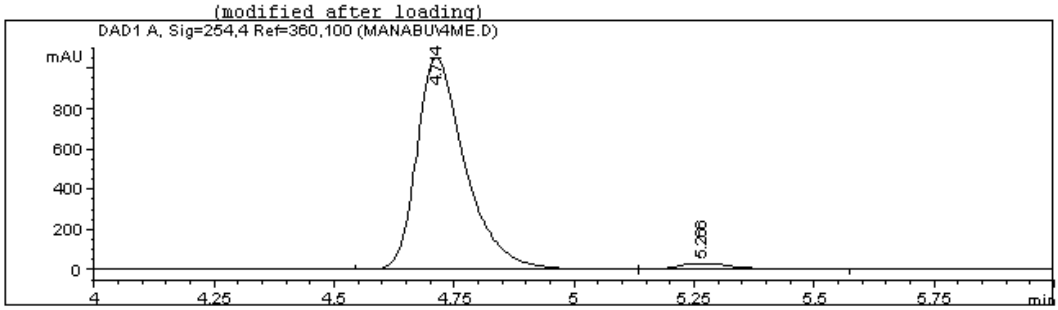



```
==========================================================================
                          Area Percent Report
==========================================================================

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


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   4.714 BV    0.1037 7399.76172 1057.30090  96.8361
   2   5.266 VP    0.1176  241.76926   30.79171   3.1639

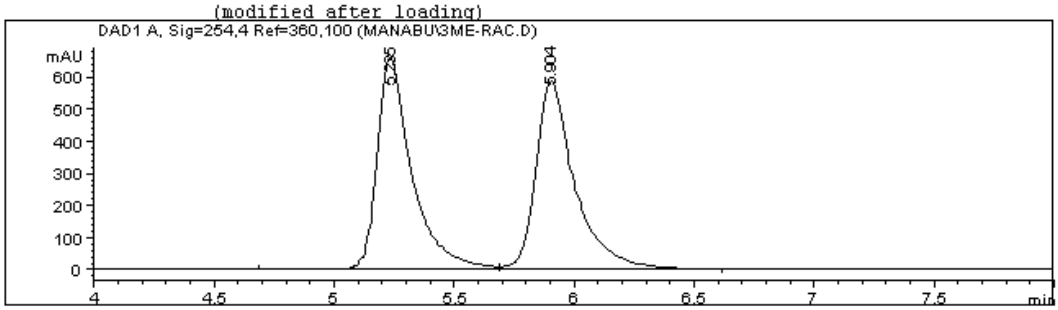
Totals :                  7641.53098 1088.09261

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Racemic 7

Data File C:\HPCHEM\2\DATA\MANABU\3ME-RAC.D Sample Name: 3Me-rac

```
==========================================================================
Injection Date  : 5/26/2007 5:40:46 PM
Sample Name     : 3Me-rac                          Location : Vial 2
Acq. Operator   : MANABU
                                                  Inj Volume : 10 µl
Acq. Method     : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 5/26/2007 5:39:16 PM by MANABU
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 5/26/2007 5:57:44 PM by MANABU
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

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


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.235 BV    0.1417 6559.88818  661.32617  49.9986
   2   5.904 VB    0.1629 6560.25537  584.67340  50.0014

Totals :                  1.3120le4  1245.99957

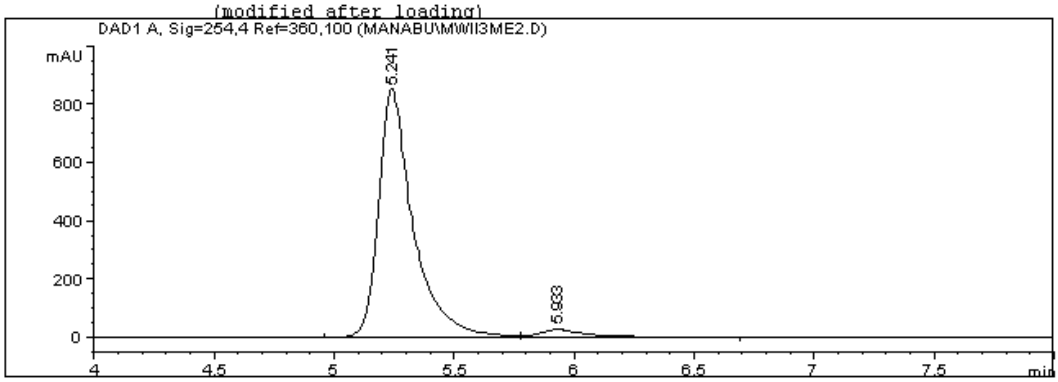
 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Enantioenriched 7

Data File C:\HPCHEM\2\DATA\MANABU\MWII3ME2.D Sample Name: MWII-3Me

3-Me

```
==========================================================================
Injection Date  : 5/22/2007 1:43:47 PM
Sample Name     : MWII-3Me                        Location : Vial 41
Acq. Operator   : MANABU
                                                 Inj Volume : 2 µl
Acq. Method     : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 5/22/2007 1:45:02 PM by MANABU
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\ROB.M
Last changed    : 5/25/2007 11:12:16 AM by Rob
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

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


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.241 VV    0.1464 8501.94336  852.08423  96.3348
   2   5.933 VB    0.1815  323.46936   25.59337   3.6652

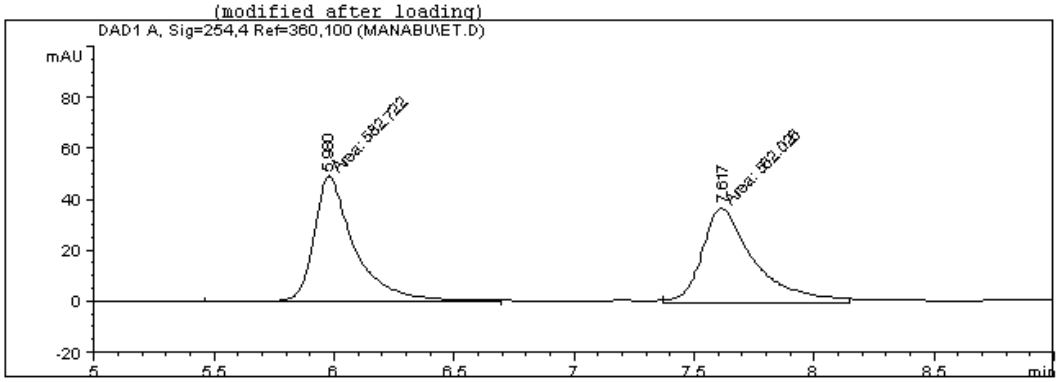
Totals :                   8825.41272  877.67760

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Racemic 8

Data File C:\HPCHEM\2\DATA\MANABU\ET.D Sample Name: Et

```
==========================================================================
Injection Date  : 4/26/2007 3:03:46 AM
Sample Name     : Et                              Location : Vial 32
Acq. Operator   : MANABU
                                                  Inj Volume : 2 µl
Acq. Method     : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 4/2/2007 2:32:21 PM by MANABU
Analysis Method : C:\HPCHEM\2\METHODS\ROB.M
Last changed    : 5/14/2007 9:06:06 PM by Rob
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

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


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.980 MM    0.1980  582.72223   49.03902  50.9039
   2   7.617 MM    0.2533  562.02637   36.97408  49.0961

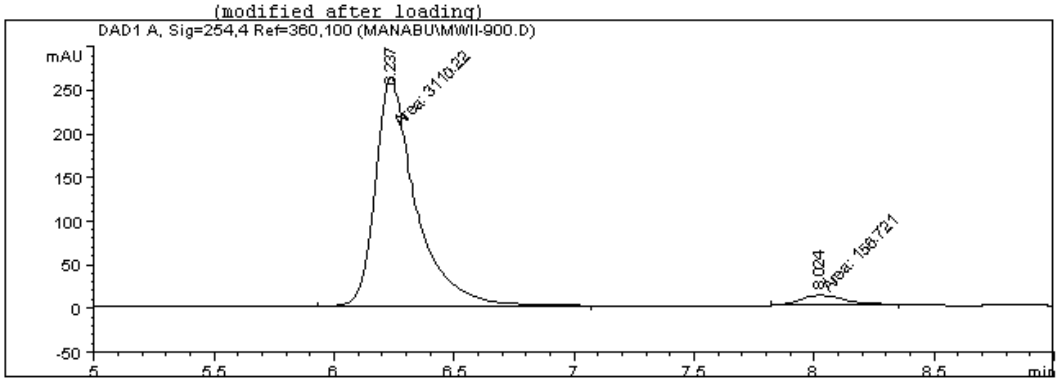
Totals :                  1144.74860   86.01310

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Enantioenriched 8

Data File C:\HPCHEM\2\DATA\MANABU\MWII-900.D Sample Name: MWII-90

```
==========================================================================
Injection Date  : 2/26/2007 12:40:51 PM
Sample Name     : MWII-90                          Location : Vial 83
Acq. Operator   : MANABU
                                                  Inj Volume : 2 µl
Acq. Method     : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 1/27/2007 11:14:36 AM by MANABU
Analysis Method : C:\HPCHEM\2\METHODS\ROB.M
Last changed    : 5/14/2007 9:03:47 PM by Rob
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

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


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.237 MM    0.1978 3110.22070  262.06119  95.2028
   2   8.024 MM    0.2246  156.72131   11.62845   4.7972

Totals :                   3266.94202  273.68964

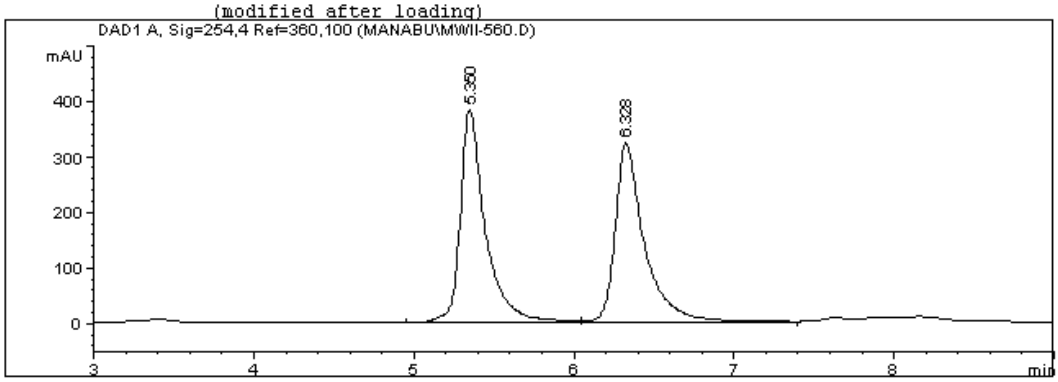
 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Racemic 9

Data File C:\HPCHEM\2\DATA\MANABU\MWII-560.D Sample Name: MWII-56

allyl rac

```
==========================================================================
Injection Date  : 2/12/2007 11:12:51 AM
Sample Name     : MWII-56                          Location : Vial 72
Acq. Operator   : MANABU
                                                  Inj Volume : 2 µl
Acq. Method     : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 2/12/2007 10:10:19 AM by MANABU
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\ROB.M
Last changed    : 5/14/2007 8:44:37 PM by Rob
                  (modified after loading)
```




```
==========================================================================
                        Area Percent Report
==========================================================================

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


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.350 BV    0.1561 4137.13379  382.65686  50.3501
   2   6.328 VV    0.1840 4079.60181  321.51462  49.6499

Totals :                   8216.73560  704.17148

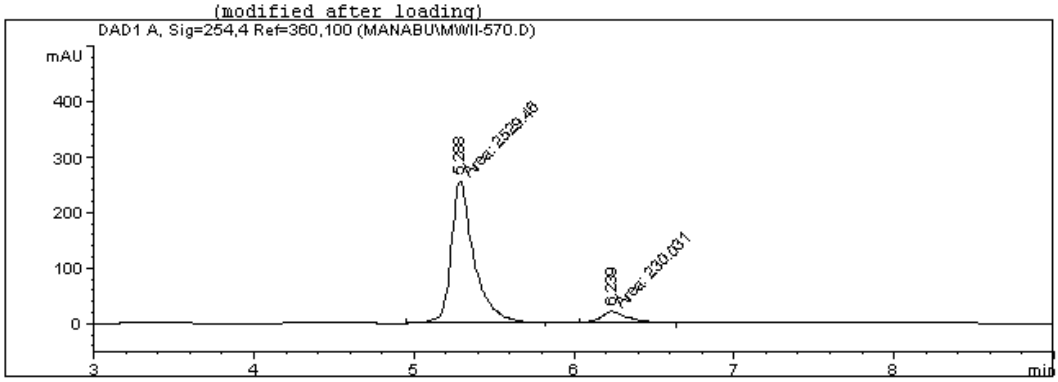
 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```

Enantioenriched 9

Data File C:\HPCHEM\2\DATA\MANABU\MWII-570.D Sample Name: MWII-57

allyl 1,2

```
==========================================================================
Injection Date  : 2/12/2007 11:26:58 AM
Sample Name     : MWII-57                          Location : Vial 73
Acq. Operator   : MANABU
                                                  Inj Volume : 2 µl
Acq. Method     : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 2/12/2007 11:26:36 AM by MANABU
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\ROB.M
Last changed    : 5/14/2007 8:45:06 PM by Rob
                  (modified after loading)
```




```
==========================================================================
                        Area Percent Report
==========================================================================

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


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.288 MM    0.1649 2529.45581  255.71126  91.6640
   2   6.239 MM    0.2023  230.03125   18.95047   8.3360

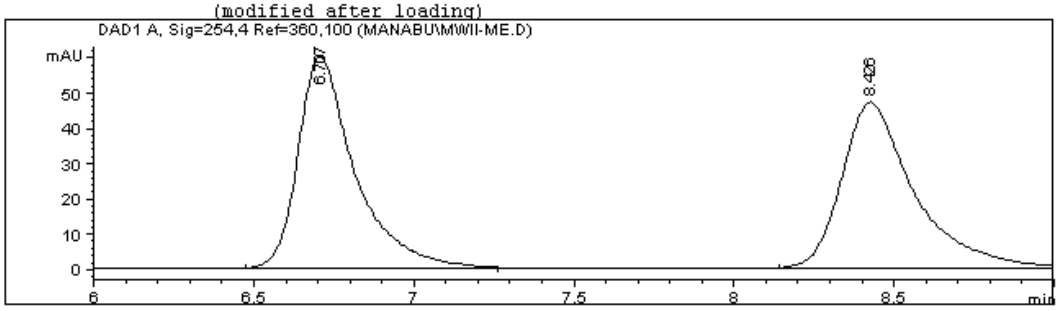
Totals :                  2759.48706  274.66173

 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***
```

Racemic 10

Data File C:\HPCHEM\2\DATA\MANABU\MWII-ME.D Sample Name: Me

```
==========================================================================
Injection Date  : 5/30/2007 4:16:27 AM
Sample Name     : Me                              Location : Vial 4
Acq. Operator   : MANABU
                                                  Inj Volume : 2 µl
Acq. Method     : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 5/30/2007 4:15:39 AM by MANABU
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 5/30/2007 4:38:44 AM by MANABU
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

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


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.707 BB    0.1802  743.91437   60.18658  49.9250
   2   8.426 BB    0.2306  746.15057   47.16357  50.0750

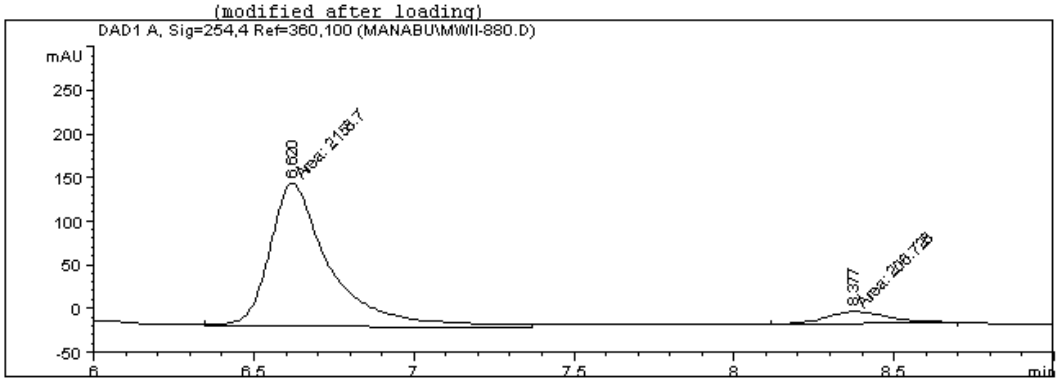
Totals :                  1490.06494  107.35015

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Enantioenriched 10

Data File C:\HPCHEM\2\DATA\MANABU\MWII-880.D Sample Name: MWII-89

```
==========================================================================
Injection Date  : 2/26/2007 11:54:06 AM
Sample Name     : MWII-89                          Location : Vial 81
Acq. Operator   : MANABU
                                                  Inj Volume : 2 µl
Acq. Method     : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 1/27/2007 11:14:36 AM by MANABU
Analysis Method : C:\HPCHEM\2\METHODS\ROB.M
Last changed    : 5/14/2007 8:57:19 PM by Rob
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

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


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.620 MM    0.2199 2158.69653  163.63376  91.2604
   2   8.377 MM    0.2434  206.72845   14.15574   8.7396

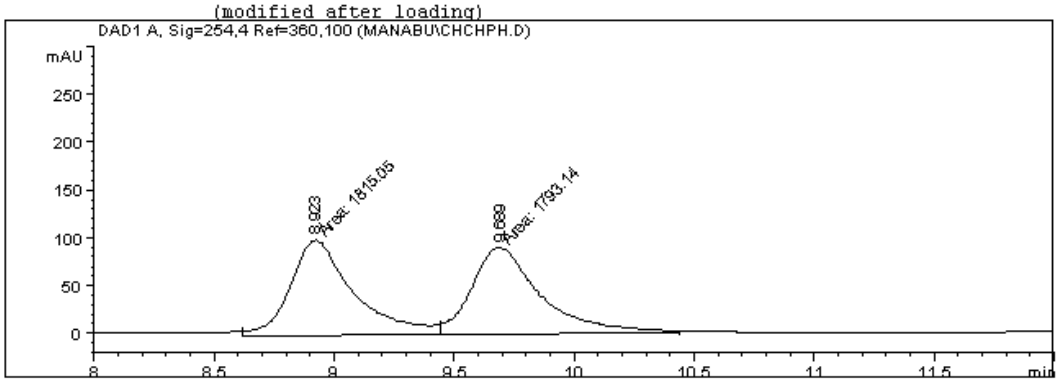
Totals :                  2365.42499  177.78950

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Racemic 11

Data File C:\HPCHEM\2\DATA\MANABU\CHCHPH.D Sample Name: CHCHPH

```
==========================================================================
Injection Date  : 4/26/2007 2:35:10 AM
Sample Name     : CHCHPH                          Location : Vial 31
Acq. Operator   : MANABU
                                                  Inj Volume : 2 µl
Acq. Method     : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 4/2/2007 2:32:21 PM by MANABU
Analysis Method : C:\HPCHEM\2\METHODS\ROB.M
Last changed    : 5/14/2007 9:08:31 PM by Rob
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

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


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.923 MF    0.3052 1815.04565   99.11434  50.3035
   2   9.689 FM    0.3307 1793.14380   90.37606  49.6965

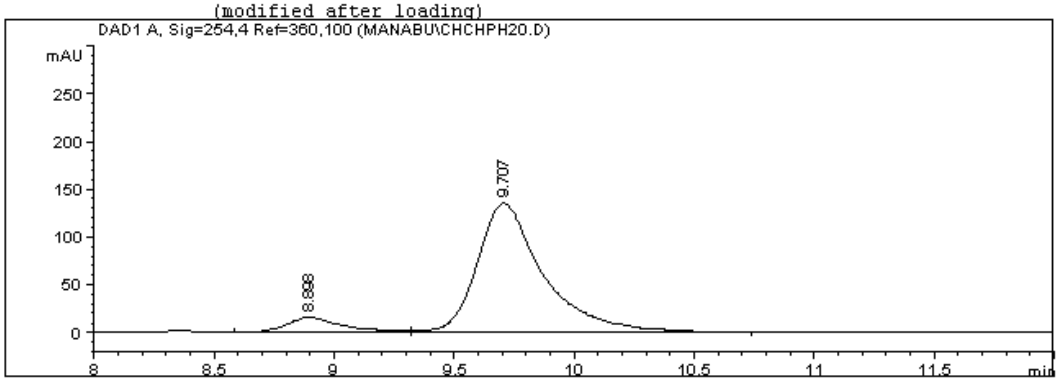
Totals :                   3608.18945  189.49040

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Enantioenriched 11

Data File C:\HPCHEM\2\DATA\MANABU\CHCHPH20.D Sample Name: CHCHPH2

```
==================================================================
Injection Date  : 4/17/2007 10:57:10 AM
Sample Name     : CHCHPH2                      Location : Vial 73
Acq. Operator   : MANABU
                                              Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 4/17/2007 10:55:13 AM by MANABU
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\ROB.M
Last changed    : 5/14/2007 9:08:31 PM by Rob
                  (modified after loading)
```




```
==================================================================
                      Area Percent Report
==================================================================

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


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

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.898 VV    0.2469  258.08749   15.29082   9.0493
   2   9.707 VB    0.2806 2593.92944  134.75366  90.9507

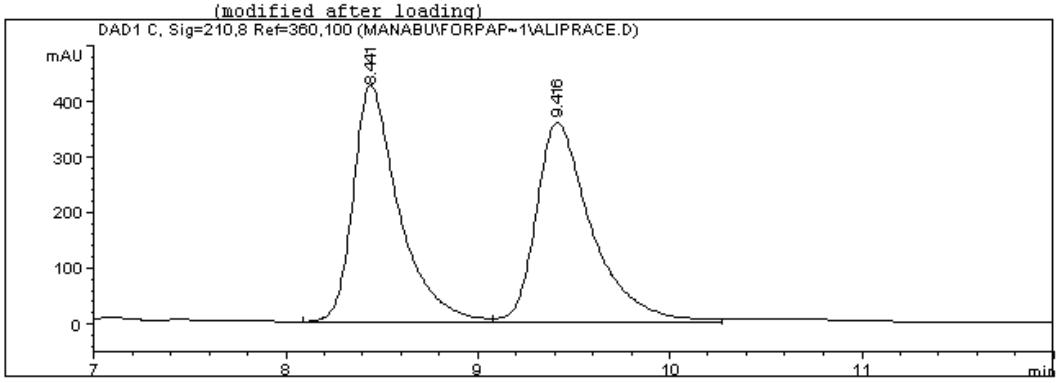
Totals :                   2852.01694  150.04449

 Results obtained with enhanced integrator!
==================================================================
                      *** End of Report ***
```

Racemic 12

Data File C:\HPCHEM\2\DATA\MANABU\FORPAP~1\ALIPRACE.D Sample Name: aliprace

```
==================================================================
Injection Date  : 4/26/2007 10:32:26 AM
Sample Name     : aliprace                    Location : Vial 75
Acq. Operator   : MANABU
                                             Inj Volume : 10 µl
Acq. Method     : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 4/26/2007 10:22:52 AM by MANABU
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\ROB.M
Last changed    : 5/25/2007 11:06:54 AM by Rob
                  (modified after loading)
```




```
==================================================================
                      Area Percent Report
==================================================================

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


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.441 PV    0.2398 7003.30420  426.06577  49.5754
   2   9.416 VB    0.2944 7123.26172  357.64194  50.4246

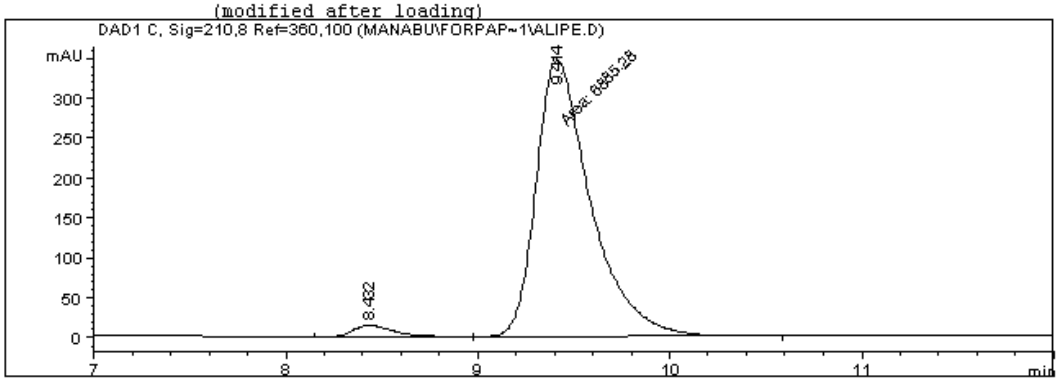
Totals :                  1.41266e4   783.70770

 Results obtained with enhanced integrator!
==================================================================
                      *** End of Report ***
```

Enantioenriched 12

Data File C:\HPCHEM\2\DATA\MANABU\FORPAP~1\ALIPE.D Sample Name: alipe

```
==================================================================
Injection Date  : 4/26/2007 10:52:32 AM
Sample Name     : alipe                          Location : Vial 74
Acq. Operator   : MANABU
                                                 Inj Volume : 10 µl
Acq. Method     : C:\HPCHEM\2\METHODS\MANABU.M
Last changed    : 4/26/2007 10:22:52 AM by MANABU
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\MMB LC.M
Last changed    : 5/15/2007 10:21:27 AM by mmb
                  (modified after loading)
```




```
==================================================================
                      Area Percent Report
==================================================================

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


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.432 VP    0.2282  236.96541   15.34524   3.3271
   2   9.414 MM    0.3300 6885.28369  347.73145  96.6729

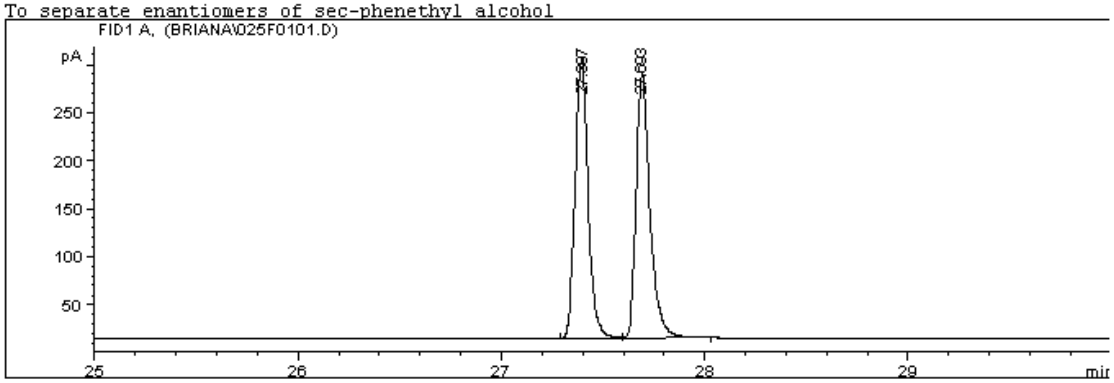
Totals :                   7122.24910  363.07669

 Results obtained with enhanced integrator!
==================================================================
                      *** End of Report ***
```

Racemic 13

Data File C:\HPCHEM\1\DATA\BRIANA\025F0101.D Sample Name: MWII

```
==========================================================================
Injection Date  : 5/5/07 2:49:05 PM              Seq. Line :   1
Sample Name     : MWII                          Location : Vial 25
Acq. Operator   : BTA                               Inj :   1
                                               Inj Volume : 3 µl
Acq. Method     : C:\HPCHEM\1\METHODS\MW.M
Last changed    : 11/6/06 3:23:49 PM by BrianA
Analysis Method : C:\HPCHEM\1\METHODS\MW.M
Last changed    : 5/5/07 3:41:14 PM by BTA
                  (modified after loading)
To separate enantiomers of sec-phenethyl alcohol
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  27.397 PV    0.0656 1291.62000  289.49869 49.90987
   2  27.693 VB    0.0664 1296.28491  275.24738 50.09013

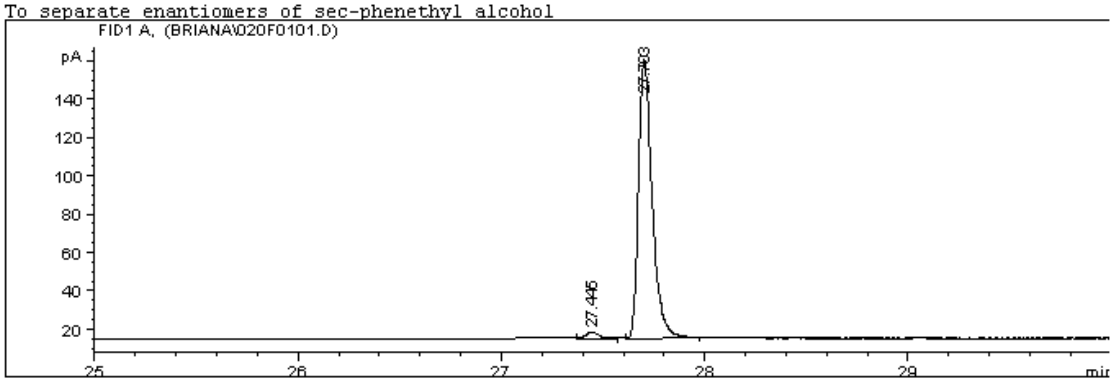
Totals :                  2587.90491  564.74606

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

Enantioenriched 13

Data File C:\HPCHEM\1\DATA\BRIANA\020F0101.D Sample Name: MWII-chi

```
==========================================================================
Injection Date  : 5/5/07 3:36:15 PM              Seq. Line :   1
Sample Name     : MWII-chi                        Location : Vial 20
Acq. Operator   : BTA                                Inj :   1
                                                 Inj Volume : 3 µl
Sequence File   : C:\HPCHEM\1\SEQUENCE\APEL.S
Acq. Method     : C:\HPCHEM\1\METHODS\MW.M
Last changed    : 5/5/07 3:41:14 PM by BTA
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\MW.M
Last changed    : 5/5/07 4:19:50 PM by BTA
                  (modified after loading)
To separate enantiomers of sec-phenethyl alcohol
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  27.445 BB    0.0562   12.20981    2.83643  1.77708
   2  27.703 PB    0.0648  674.86255  144.97050 98.22292

Totals :                   687.07236  147.80693

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```