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Two phenyls are better than one or three: Synthesis and application of terminal olefin-oxazoline (TOlefOx) ligands

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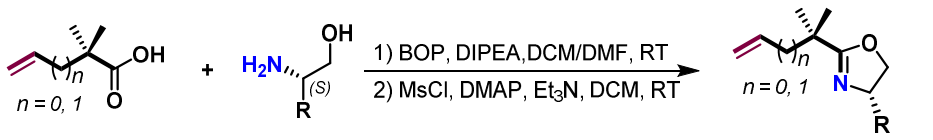
1. General Information

All solvents were dried before use following the standard procedures. Unless otherwise indicated, all starting materials purchased from commercial suppliers were used without further purification. The ^{1}H and ^{13}C NMR spectra were recorded on Bruker AV-400 MHz in the indicated solvents. Chemical shifts are reported in δ (ppm) referenced to an internal TMS standard for ^{1}H NMR and $CDCl_3$ (δ = 77.10 ppm) for ^{13}C NMR. Coupling constants (*J*) are quoted in Hz. IR spectra were recorded on Nicolet iN 10 MX. ESI mass spectra were recorded on Agilent 1200/G6100A. (BOP = Benzotriazol-1-yloxytris(dimethylamino) phosphoniumhexafluorophosphate; DIPEA = Ethyldiisopropylamine; DMAP = 4-Dimethylaminopyridine; MsCl = Methanesulfonyl chloride;

2. Ligand Preparation

2.1 General Procedure for Preparation of TOlefOx Type I Ligands



4 **5** **TOlefOx Type I Ligands**

5a (R = CH_2Ph)
5b (R = $CHPh_2$)

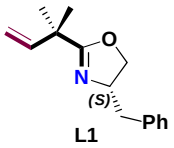
L1 (*n* = *0*, R = CH_2Ph)
L2 (*n* = *1*, R = CH_2Ph)
L3 (*n* = *1*, R = $CHPh_2$)

To a well-stirred solution of enoic acid **4**[1] (1 mmol) in DCM / DMF (20:3, 10 mL) was added the optically pure amino alcohol **5** (1 mmol), BOP (1.2 mmol) and DIPEA (2 mmol). The resulted solution was stirred overnight at room temperature. Then the reaction mixture was diluted with water (30 mL) and extracted with DCM (30 mL × 3). The combined organic phases were washed with brine (30 mL), dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was used in the next step without any further purification.

To a well-stirred solution of the previous residue, Et_3N (8 mmol) and DMAP (0.1 mmol) in DCM (10 mL) was added MsCl (2.5 mmol) dropwise at 0 °C. The resulting solution was warmed to room temperature and stirred overnight. The reaction mixture was quenched with water (30 mL) and extracted with DCM (30 mL × 3). The combined organic phases were washed with brine (30 mL), dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash column chromatography using hexane / ethyl acetate eluent to afford the desired product **L1**-**L3**.

[1] 2,2-Dimethylbut-3-enoic acid (**4a**, CAS 10276-09-2), 2,2-Dimethylpent-4-enoic acid (**4b**, CAS 16386-93-9), and (*S*)-Phenylalaninol (**5a**, CAS 3182-95-4) were purchased from Alfa Aesar. (*S*)-Diphenylalaninol (**5b**, CAS 162118-01-6) was purchased from Oakwood Chemical.

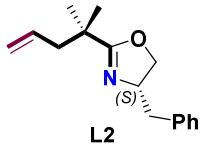
(*S*)-4-Benzyl-2-(2-methylbut-3-en-2-yl)-4,5-dihydrooxazole (L1)



L1

Yellow oil. 74% yield for two steps. $[\alpha]_D^{23}$–56.8 (*c* 1.24 $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.30–7.18 (m, 5H), 6.03–5.95 (m, 1H), 5.10–5.03 (m, 2H), 4.38–4.35 (m, 1H), 4.15–3.95 (m, 2H), 3.10 (dd, *J* = 13.6 Hz, *J* = 4.4 Hz, 1H), 2.68–2.61 (m, 1H), 1.30 (s, 3H), 1.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 172.16, 143.27, 141.83, 129.02, 128.39, 126.66, 112.27, 70.80, 69.12, 56.03, 39.17, 25.22, 25.02; ESI-MS: $[M+H]^{\oplus}$ 230.1; HRMS (ESI): $[M+H]^{\oplus}$ calcd for $C_{15}H_{20}NO^{\oplus}$ 230.1539, found: 230.1545; IR (KBr) *v* (cm^{-1}): 2975, 1658, 1495, 1451, 1413, 1262, 1130, 982, 917, 742, 701.

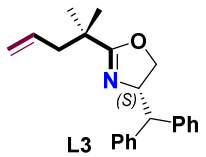
(*S*)-4-Benzyl-2-(2-methylpent-4-en-2-yl)-4,5-dihydrooxazole (L2)



L2

Yellow oil. 94% yield for two steps. $[\alpha]_D^{23}$–19.0 (*c* 1.24 $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.32–7.18 (m, 5H), 5.74–5.64 (m, 1H), 5.06–5.00 (m, 2H), 4.36–4.30 (m, 1H), 4.11 (t, J = 11.8 Hz, 1H), 3.96 (dd, J = 11.2 Hz, J = 9.2 Hz, 1H), 3.10 (dd, *J* = 18.0 Hz, *J* = 6.0 Hz, 1H), 2.61 (dd, J = 18.0, 12.0 Hz, 1 H), 2.25 (d, *J* = 10.0 Hz, 2H), 1.18 (s, 3H), 1.16 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 172.85, 137.62, 134.26, 129.26, 128.22, 126.23, 117.39, 71.14, 66.78, 44.74, 41.50, 36.26, 25.24, 25.22; ESI-MS: $[M+H]^{\oplus}$ 244.1; HRMS (ESI): $[M+H]^{\oplus}$ calcd for $C_{16}H_{22}NO^{\oplus}$ 244.1696, found: 244.1701; IR (KBr) *v* (cm^{-1}): 2974, 2926, 1656, 1473, 1138, 983, 917, 752, 701.

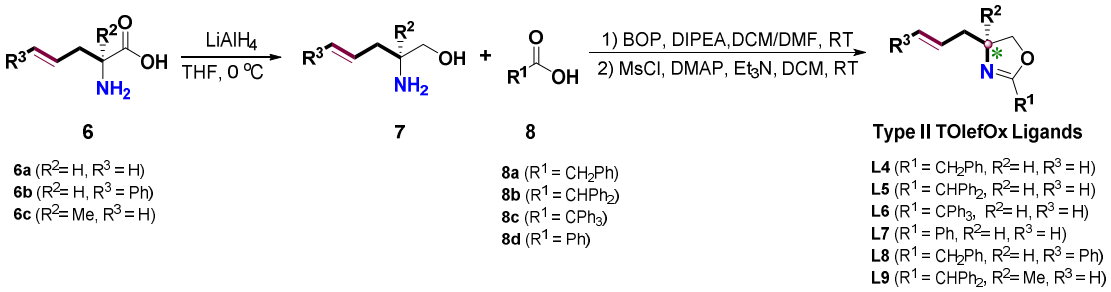
(*S*)-4-Benzhydryl-2-(2-methylpent-4-en-2-yl)-4,5-dihydrooxazole (L3)



L3

Yellow oil. 62% yield for two steps. $[\alpha]_D^{23}$–69.7 (*c* 1.07 $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.29–7.19 (m, 10H), 5.69–5.57 (m, 1H), 5.02–4.96 (m, 2H), 4.89–4.81 (m, 1 H), 4.29–4.23 (m, 1H), 4.08–3.99 (m, 2H), 2.19 (d, *J* = 9.6 Hz, 2H), 1.12 (s, 3H), 1.06 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 172.92, 141.90, 134.65, 129.09, 128.16, 126.54, 117.39, 70.57, 69.24, 56.25, 44.80, 36.38, 25.30, 25.26; ESI-MS: $[M+H]^{\oplus}$ 320.2; HRMS (ESI): $[M+H]^{\oplus}$ calcd for $C_{22}H_{26}NO^{\oplus}$ 320.2009, found: 320.2020; IR (KBr) *v* (cm^{-1}): 2972.4, 1657.6, 1599.9, 1494.8, 1354.8, 1214.0, 987.5, 914.5, 833.3, 700.5, 560.7

2.2 General Procedure for Preparation of TOlefOx Type II Ligands

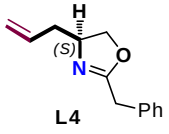


To a well-stirred solution of the optically pure amino acid **6**[2] (1 mmol) in THF (10 mL) under argon atmosphere was added $LiAlH_4$ (2 mmol) in several portions at 0 °C and allowed to stir for 2 h. The resulting solution was warmed to room temperature and stirred for 36 h. The reaction mixture was quenched with 10% NaOH aqueous solution (30 mL) and extracted with ethyl acetate (30 mL × 3). The combined organic phases were washed with brine (30 mL), dried over anhydrous Na_2SO_4 and concentrated under reduced pressure to afford the crude compound **7**, which was used in the next step without any further purification.

To a well-stirred solution of the crude compound **7** in DCM / DMF (20:3, 10 mL) was added the carboxylic acid **8** (1 mmol), BOP (1.2 mmol) and DIPEA (2 mmol). The resulted solution was stirred overnight at room temperature. Then the reaction mixture was diluted with water (30 mL) and extracted with DCM (30 mL × 3). The combined organic phases were washed with brine (30 mL), dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was used in the next step without any further purification.

To a well-stirred solution of the previous residue, Et_3N (8 mmol) and DMAP (0.1 mmol) in DCM (10 mL) was added MsCl (2.5 mmol) dropwise at 0 °C. The resulting solution was warmed to room temperature and stirred overnight. The reaction mixture was quenched with water (30 mL) and extracted with DCM (30 mL × 3). The combined organic phases were washed with brine (30 mL), dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash column chromatography using hexane / ethyl acetate eluent to afford the desired product **L4**-**L9**.

(*S*)-4-Allyl-2-benzyl-4,5-dihydrooxazole (L4)



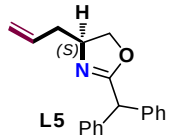
L4

Colorless oil. 32% yield for three steps. $[\alpha]_D^{23}$–2.3(*c* 0.56 $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.34–7.23 (m, 5H), 5.81–5.70 (m, 1H), 5.12–5.06 (m, 2H), 4.27–4.14 (m, 2H), 3.91 (t, J = 7.2 Hz, 1H), 3.61 (s, 2H), 2.47–2.41 (m, 1H), 2.26–2.17 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm)

[2] (*S*)-2-Aminopent-4-enoic acid (**6a**, CAS 16338-48-0), (*S*)-2-Amino-5-phenylpent-4-enoic acid (**6b**, CAS 52161-76-9) were purchased from Aldrich. (*S*)-2-Amino-2-methylpent-4-enoic acid (**6c**, CAS 96886-55-4) was purchased from Alfa Aesar.

166.30, 135.27, 134.07, 128.96, 128.61, 127.01, 117.64, 71.93, 65.47, 39.81, 34.88; ESI-MS: $[M+Na]^{\oplus}$ 224.1; HRMS (ESI): $[M+H]^{\oplus}$ calcd for $C_{13}H_{16}NO^{\oplus}$ 202.1226, found: 202.1228; IR (KBr) ν (cm^{-1}): 3296.0, 3064.9, 2923.4, 1662.9, 1543.8, 1362.9, 1247.6, 1164.3, 982.9, 918.4, 720.0

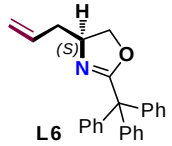
(*S*)-4-Allyl-2-benzhydryl-4,5-dihydrooxazole (L5）



L5

Colorless oil. 57% yield for three steps. $[\alpha]_D^{23}$–21.6 (*c* 0.74 $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.32–7.25 (m, 10H), 5.79–5.70 (m, 1H), 5.11–5.07 (m, 3H), 4.15–4.38 (m, 2H), 4.00–3.97 (m, 1H), 2.51–2.45 (m, 1H), 2.29–2.24 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 167.62, 139.35, 134.01, 128.64, 128.52, 127.13, 117.77, 71.88, 65.38, 51.04, 39.74; ESI-MS: $[M+H]^{\oplus}$ 278.2; HRMS (ESI): $[M+H]^{\oplus}$ calcd for $C_{19}H_{20}NO^{\oplus}$ 278.1539, found: 278.1544; IR (KBr) ν (cm^{-1}) 3062.6, 3028.2, 2967.4, 2924.7, 2896.3, 1659.8, 1601.2, 1495.1, 1452.5, 1376.3, 1356.8, 1129.4, 1078.2, 1031.9, 993.2, 918.4, 749.2, 699.0, 640.8

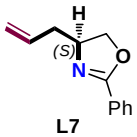
(*S*)-4-Allyl-2-trityl-4,5-dihydrooxazole (L6)



L6

Colorless oil. 56% yield for three steps. $[\alpha]_D^{23}$–32.9 (*c* 0.93, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.28–7.21 (m, 15H), 5.81–5.71 (m, 1H), 5.12–5.07 (m, 2H), 4.34–4.26 (m, 2H), 4.06 (t, *J* = 6.8 Hz, 1H), 2.53–2.49 (m, 1H), 2.32–2.25 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 169.71, 143.48, 134.10, 130.19, 127.60, 126.80, 117.75, 71.45, 65.52, 61.43, 39.50; ESI-MS: $[M+H]^{\oplus}$ 354.3; HRMS (ESI): $[M+H]^{\oplus}$ calcd for $C_{25}H_{24}NO^{\oplus}$ 354.1853, found: 354.1861; IR (KBr) ν (cm^{-1}) 3087.2, 3050.4, 3030.6, 2916.9, 1643.7, 1597.1, 1490.4, 1443.2, 1309.9, 1213.1, 1189.3, 1155.9, 1034.6, 990.0, 913.4, 763.1, 746.5, 698.5, 635.7, 507.2.

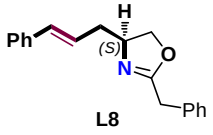
(*S*)-4-Allyl-2-phenyl-4,5-dihydrooxazole (L7)



L7

Colorless oil. 63.3% yield for three steps. $[\alpha]_D^{23}$–35.8 (*c* 0.53 $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.94 (d, *J* = 8.0 Hz, 2H), 7.49–7.38 (m, 3H), 5.88–5.77 (m, 1H), 5.16–5.10 (m, 2H), 4.47–4.35 (m, 2H), 4.12–4.08 (m, 1H), 2.60–2.54 (m, 1H), 2.37–2.29 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 163.91, 134.13, 131.33, 128.33, 128.28, 127.77, 117.72, 71.76, 65.96, 39.90; ESI-MS: $[M+H]^{\oplus}$ 188.1; HRMS (ESI): $[M+H]^{\oplus}$ calcd for $C_{12}H_{14}NO^{\oplus}$ 188.1070, found: 188.1069; IR (KBr) ν (cm^{-1}) 3071.2, 2917.2, 1649.3, 1579.6, 1449.9, 1357.1, 1081.7, 1025.2, 968.8, 780.7, 695.3.

(*S*)-2-Benzyl-4-cinnamyl-4,5-dihydrooxazole (L8)

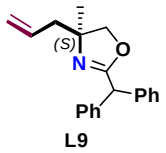


L8

Colorless oil. 32% yield for three steps. $[\alpha]_D^{23}$–31.4 (*c* 0.60 $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.31–7.18 (m, 10H), 6.44 (d, *J* = 16.0 Hz, 1H), 6.15–6.06 (m, 1H), 4.29–4.24 (m, 2H), 4.01–3.95 (m, 1H), 3.68–3.57 (m, 2H), 2.59–2.53 (m,

1H), 2.64–2.39 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 166.51, 137.29, 135.27, 133.00, 128.97, 128.67, 128.55, 127.30, 127.03, 126.20, 125.43, 71.80, 65.74, 38.86, 34.93; ESI-MS: $[M+Na]^{\oplus}$ 300.1; HRMS (ESI): $[M+Na]^{\oplus}$ calcd for $C_{19}H_{19}NNaO^{\oplus}$ 300.1359, found: 300.1358; IR (KBr) ν (cm^{-1}): 3290.6, 3028.0, 2924.5, 1734.1, 1652.2, 1541.1, 1454.7, 1362.0, 1260.0, 1148.3, 1073.0, 967.6, 802.9, 745.0, 725.5, 694.0.

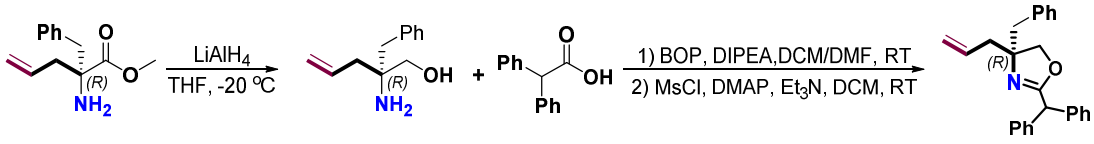
(*S*)-4-Allyl-2-benzhydryl-4-methyl-4,5-dihydrooxazole (L9)



L9

Colorless oil. 57% yield for three steps. $[\alpha]_D^{23}$–6.5 (*c* 0.87, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.31–7.26 (m, 10H), 5.74–5.64 (m, 1H), 5.11–5.05 (m, 3H), 4.11 (d, *J* = 8.4 Hz, 1H), 3.85 (d, *J* = 8.4 Hz, 1H), 2.32 (m, 2H), 1.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 166.11, 139.39, 133.64, 128.69, 128.50, 127.10, 118.58, 76.75, 69.69, 51.00, 45.26, 26.94; ESI-MS: $[M+H]^{\oplus}$ 292.2; HRMS (ESI): $[M+H]^{\oplus}$ calcd for $C_{20}H_{22}NO^{\oplus}$ 292.1695, found: 292.1700; IR (KBr) ν (cm^{-1}): 3062.6, 2967.4, 1659.8, 1601.2, 1495.1, 1452.5, 1376.3, 1129.4, 993.2, 918.4, 749.2, 699.0, 640.8.

2.3 Preparation Of L10



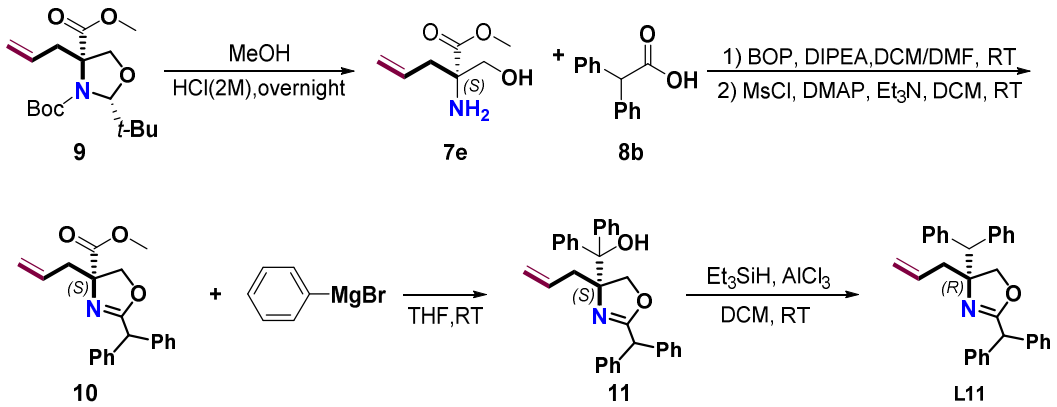
6d 7d 8b L10

The preparation of compound **6d** followed the procedure of Rodney and coworkers[3]. The preparation of **L10** followed the general procedure for preparation of TOlefOx type II ligands.

(*R*)-4-Allyl-2-benzhydryl-4-benzyl-4,5-dihydrooxazole (L10) Colorless oil. 73% yield for three steps. $[\alpha]_D^{23}$+26.9 (*c* 1.21, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ(ppm) 7.27–7.05 (m, 15H), 5.76–5.66 (m, 1H), 5.11 (d, *J* = 6.4 Hz, 1H), 5.07 (s, 1 H), 5.03 (s, 1 H), 4.09 (d, *J* = 8.4 Hz, 1H), 4.02, (d, *J* = 8.4 Hz, 1H), 3.02 (d, *J* = 13.6 Hz, 1H), 2.76 (d, *J* = 13.6 Hz, 1H), 2.49 (dd, *J* = 13.6 Hz, *J* = 6.8 Hz, 1H), 2.32 (dd, *J* = 13.6 Hz, *J* = 6.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 166.66, 139.34, 139.13, 136.67, 133.41, 130.92, 128.78, 128.61, 128.37, 128.36, 128.09, 127.00, 126.90, 126.47, 119.01, 73.22, 73.10, 51.03, 45.16, 44.78; ESI-MS: $[M+H]^{\oplus}$ 368.2; HRMS (ESI): $[M+H]^{\oplus}$ calcd for $C_{26}H_{26}NO^{\oplus}$ 368.2009, found: 368.2007; IR (KBr) ν (cm^{-1}): 3061.3, 3027.6, 2976.9, 1660.4, 1601.6, 1494.9, 1452.7, 1363.2, 1181.8, 1031.3, 992.6, 918.2, 749.6, 700.1.

[3] Kristine, D.; Giuseppe, F. P.; Suresh K. G.; Ram K. M. and Rodney L. J. *J. Med. Chem.* **2003**, *46*, 727−733.

2.4 Preparation of L11



The preparation of compound **9** followed the procedure of Koskinen and coworkers[4].

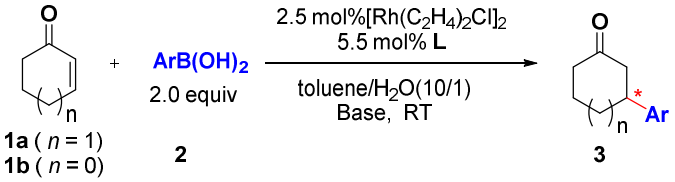
(*S*)-Methyl 4-allyl-2-benzhydryl-4,5-dihydrooxazole-4-carboxylate (10) To a well-stirred solution of compound **9** (8.5 mmol) in MeOH (40 mL) under argon atmosphere was added HCl (2M, 16 mL) dropwise in an hour at room temperature. The resulting solution was allowed to stir overnight at room temperature. Then the reaction mixture was quenched with saturated $NaHCO_3$(50 mL) and evaporated the solvent MeOH in vacuo, then extracted with ethyl acetate (50 mL × 3), dried over anhydrous Na_2SO_4 and concentrated under reduced pressure to afford the crude compound **7e**. The residue was used in the next step without any further purification. The preparation of **10** followed the general procedure for preparation of TOlefOx type I ligands to afford the desired product **10** as white solid, 29% yield for three steps. m.p. 57–58℃; $[\alpha]_D^{23}$–10.6 (*c* 0.59, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.32–7.26 (m, 10H), 5.64–5.54 (m, 1H), 5.21 (s, 1H), 5.14–5.07 (m, 2H), 4.62 (d, J = 9.2 Hz, 1H), 4.19 (d, J = 9.2 Hz, 1H), 3.78 (s, 3H), 2.65–2.55 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 172.91, 169.03, 138.88, 131.48, 128.69, 128.54, 127.24, 119.98, 76.93, 73.35, 52.76, 50.93, 41.98; ESI-MS: $[M+H]^{\oplus}$ 336.2; HRMS (ESI): $[M+Na]^{\oplus}$ calcd for $C_{21}H_{22}NO_3^{\oplus}$ 336.1600, found: 336.1601; IR (KBr) ν (cm^{-1}): 1737.8, 1649.0, 1495.9, 1290.6, 1216.8, 1141.3, 976.2, 919.6, 748.9, 727.4, 703.3, 644.8.

(*R*)-4-Allyl-2,4-dibenzhydryl-4,5-dihydrooxazole (L11) To a well-stirred solution of compound **10** (1 mmol) in THF (10 mL) under argon atmosphere was added phenylmagnesium bromide (3.0 M solution in diethyl ether, 1 mL, 3.0 mmol) and allowed to stir for 10 min at room temperature. Then the reaction mixture was quenched with saturated NH_4Cl solution (50 mL) extracted with ethyl acetate (50 mL × 3). The combined organic phases were washed with brine (20 mL), dried over anhydrous Na_2SO_4 and concentrated under reduced pressure to afford the crude compound **11.** To a well-stirred solution of the previous crude compound **11** in THF (10 mL) under argon atmosphere was added Et_3SiH (30 mmol) and $AlCl_3$ (1.5 mmol)

[4] Brunner, M.; Saarenketo, P.; Straub, T.; Rissanen, K. and Koskinen, A. M. P. *Eur. J. Org. Chem.* **2004**, 3879–3883.

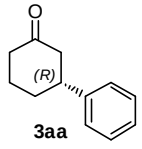
at room temperature and stirred overnight. Then the reaction mixture was quenched with saturated NH_4Cl solution (50 mL) extracted with ethyl acetate (50 mL × 3). The combined organic phases were washed with brine (20 mL), dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash column chromatography using hexane / ethyl acetate (30:1) eluent to afford the desired product **L11** as colorless oil, 93% yield for two steps. $[\alpha]_D^{23}$+63.6 (*c* 0.95 $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.51 (d, *J* = 7.2 Hz, 2H), 7.28–7.07 (m, 18H), 5.69–5.58 (m, 1H), 5.07–4.92 (m, 3H), 4.27 (d, *J* = 8.4 Hz, 1H), 4.19 (d, *J* = 8.4 Hz, 1H), 4.10 (s, 1H), 2.41–2.33 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 166.72, 141.40, 140.67, 139.64, 139.29, 133.45, 130.20, 128.98, 128.78, 128.41, 128.24, 127.04, 126.93, 126.54, 119.08, 76.14, 73.19, 59.81, 51.24, 44.34; ESI-MS: $[M+H]^{\oplus}$ 444.3; HRMS (ESI): $[M+Na]^{\oplus}$ calcd for $C_{32}H_{29}NNaO^{\oplus}$ 466.2141, found: 466.2138; IR (KBr) *v* (cm^{-1}): 3405.6, 3286.1, 2959.0, 2872.0, 1632.8, 1548.9, 1466.1, 1370.1, 1042.5, 917.0, 842.2, 746.1, 558.8.

3 General Procedure of Rh-Catalyzed Conjugate Addition



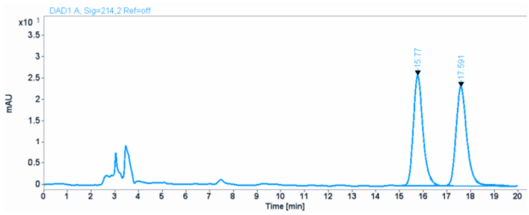
A dried Schlenk flask was charged with $[Rh(C_2H_4)_2Cl]_2$ (5 mol% Rh), **L** (10 mol%), arylboronic acid **2** (0.4 mmol) in toluene / H_2O (1mL / 0.1mL), the reaction mixture was stirred at room temperature for 15 min under argon atmosphere. Then the base (30 mol%) and enone **1** (0.2 mmol) were added and allowed to stir at room temperature. After the reaction was finished tracked by TLC, the reaction mixture was quenched with saturated aqueous NH_4Cl (5 mL) and extracted with ethyl acetate (5 mL × 3). The organic phase was washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash column chromatography using hexane / ethyl acetate eluent to afford the desired product **3**.

(*R*)-3-Phenylcyclohexanone (3aa)[5]



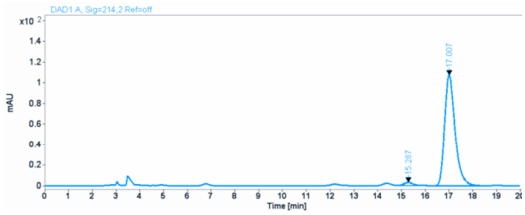
33.6 mg, 97% yield. $[\alpha]_D^{24}$ +18.4 (*c* 1.00, $CHCl_3$) for 95% *ee*; [lit.: $[\alpha]_D^{23}$–19.5 (*c* 0.95, $CHCl_3$) for 93% *ee* in the S-isomer]. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.24 (t, *J* = 7.6Hz, 2H), 7.16–7.11 (m, 3H), 2.95–2.88 (m, 1H), 2.53–2.24 (m, 4H), 2.07–1.95 (m, 2H), 1.80–1.65 (m, 2H); HPLC: Chiracel OD-H Column (250 mm), detected at 214 nm, *n*-hexane / *i*-propanol = 99/1, flow = 0.7 mL/min; Retention time: 15.3 min ((*S*)-**3aa**), 17.0 min((*R*)-**3aa**).

[5] Feng, C.-G.; Wang, Z.-Q; Shao, C.; Xu, M.-H.; Lin, G.-Q. *Org. lett.* **2008**, *10*, 4101



Signal: DAD1 A, Sig=214,2 Ref=off

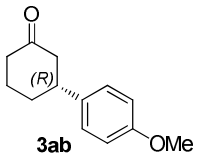
RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
15.770	0.431	725.139	25.886	49.5561	RAC
17.591	0.486	738.130	23.270	50.4439	



Signal: DAD1 A, Sig=214,2 Ref=off

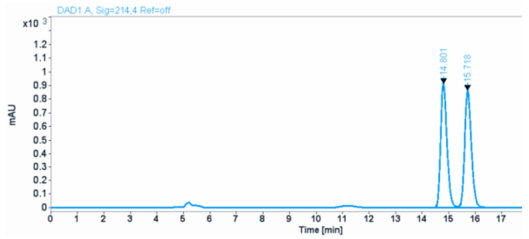
RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
15.287	0.399	81.883	3.113	2.4399	95.120
17.007	0.465	3274.040	107.606	97.5601	

(*R*)-3-(4-Methoxyphenyl)cyclohexanone (3ab)[5]



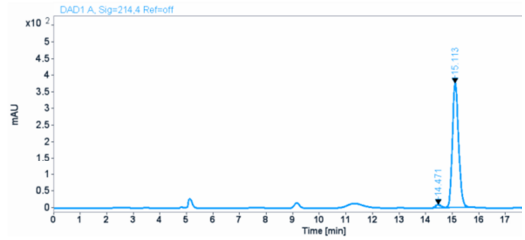
3ab

28.8mg, 71% yield. $[\alpha]_D^{24}$+11.4 (*c* 1.35, $CHCl_3$) for 96% ee. [lit.: $[\alpha]_D^{23}$–14.2 (*c* 1.02, $CHCl_3$) for 92% ee in the S-isomer]. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.14 (d, *J* = 8.4 Hz, 2H), 6.87 (d, *J* = 8.4 Hz, 2H), 3.81 (s, 3H), 3.10–2.90 (m, 1H), 2.60–2.33 (m, 4H), 2.21-2.00 (m, 2H), 1.88-1.70 (m, 2H); HPLC: Chiracel AD-H Column (250 mm), detected at 214 nm, *n*-hexane / *i*-propanol = 95/5, flow = 0.6 mL/min; Retention time: 14.4 min ((*S*)-**3ab**), 15.1 min ((*R*)-**3ab**).



Signal: DAD1 A, Sig=214,4 Ref=off

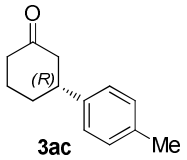
RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
14.801	0.256	15189.552	914.355	49.9165	RAC
15.718	0.272	15240.391	862.557	50.0835	



Signal: DAD1 A, Sig=214,4 Ref=off

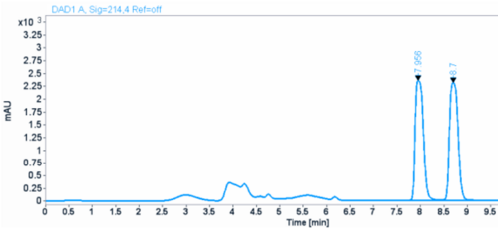
RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
14.471	0.227	138.044	9.537	2.2347	95.5
15.113	0.250	6039.228	374.743	97.7653	

(*R*)-3-(*p*-Tolyl)cyclohexanone (3ac)[5]



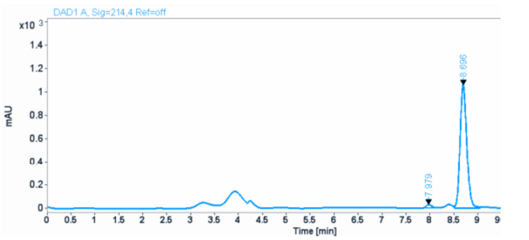
3ac

34.1 mg, 91% yield. $[\alpha]_D^{24}$ +13.1 (*c* 1.72, $CHCl_3$) for 96 % ee; [lit.: $[\alpha]_D^{23}$–15.0 (*c* 1.09, $CHCl_3$) for 94% ee in the S-isome]. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.15–7.10(m, 4H), 3.00–2.94 (m, 1H), 2.60–2.36 (m, 4H), 2.33 (s, 3H), 2.17–2.04 (m, 2H), 1.88–1.74 (m, 2H); HPLC: Chiracel AD-H Column (250 mm), detected at 214 nm, *n*-hexane / *i*-propanol = 95/5, flow = 0.6 mL/min; Retention time: 8.0 min ((*S*)-**3ac**), 8.7 min ((*R*)-**3ac**).



Signal: DAD1 A, Sig=214,4 Ref=off

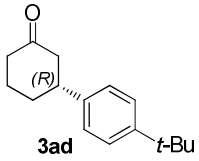
RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
7.956	0.196	28512.941	2345.253	49.2969	RAC
8.700	0.204	29326.234	2305.560	50.7031	



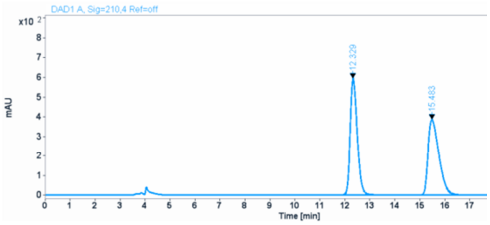
Signal: DAD1 A, Sig=214,4 Ref=off

RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
7.979	0.128	217.120	26.432	2.0572	95.886
8.696	0.163	10336.943	1057.866	97.9428	

(*R*)-3-(4-(*tert*-Butyl)phenyl)cyclohexanone (3ad)[6]

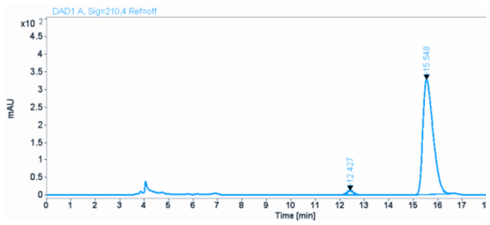


36.6 mg, 80% yield. $[\alpha]_D^{24}$ +10.1 (*c* 1.73, $CHCl_3$) for 95% ee; [lit.: $[\alpha]_D^{20}$–9.9 (*c* 0.9, $CHCl_3$) for 83% ee in the S-isomer]. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.35 (d, *J* = 8.4 Hz, 2H), 7.15 (d, *J* = 8.4 Hz, 2H), 3.02–2.95 (m, 1H), 2.62–2.33 (m, 4H), 2.17–2.07 (m, 2H), 1.89–1.74 (m, 2H), 1.31 (s, 9H); HPLC: Chiracel AS-H Column (250 mm), detected at 214 nm, *n*-hexane / *i*-propanol = 98/2, flow = 0.6 mL/min; Retention time: 12.4 min ((*S*)-**3ad**), 15.5 min ((*R*)-**3ad**).



Signal: DAD1 A, Sig=210,4 Ref=off

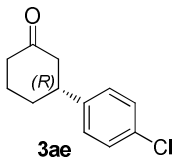
RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
12.329	0.301	11564.500	593.334	50.0458	0.092
15.483	0.466	11543.352	385.261	49.9542	



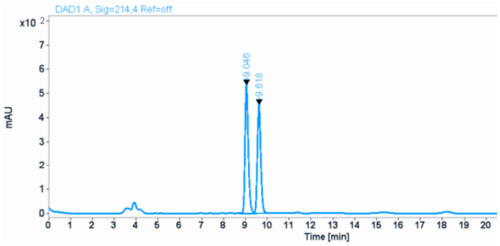
Signal: DAD1 A, Sig=210,4 Ref=off

RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
12.427	0.291	228.077	11.918	2.3481	95.304
15.548	0.453	9485.258	326.349	97.6519	

(*R*)-3-(4-Chlorophenyl)cyclohexanone (3ae)[6]

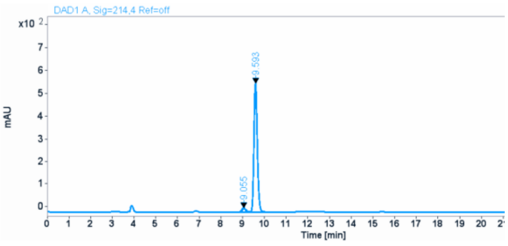


36.4 mg, 87% yield. $[\alpha]_D^{24}$ +8.1 (*c* 1.75, $CHCl_3$) for 94% ee; [lit.: $[\alpha]_D^{20}$–7.3 (*c* 1.0, $CHCl_3$) for 90% ee in the S-isomer]. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.30 (d, *J* = 8.4 Hz, 2H), 7.15 (d, *J* = 8.4 Hz, 2H), 3.03–2.95 (m, 1H), 2.60–2.33 (m, 4H), 2.17–2.12 (m, 1H), 2.08–2.05 (m, 1H), 1.87–1.75 (m, 2H); HPLC: Chiracel AD-H Column, (250 mm), detected at 214 nm, *n*-hexane / *i*-propanol = 95/5, flow = 0.8 mL/min; Retention time: 9.0 min ((*S*)-**3ae**), 9.6 min ((*R*)-**3ae**).



Signal: DAD1 A, Sig=214,4 Ref=off

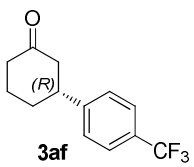
RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
9.046	0.162	5555.166	533.298	53.4233	RAC
9.618	0.165	4843.225	451.287	46.5767	



Signal: DAD1 A, Sig=214,4 Ref=off

RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
9.055	0.189	200.427	16.773	3.2206	93.559
9.593	0.164	6022.946	565.580	96.7794	

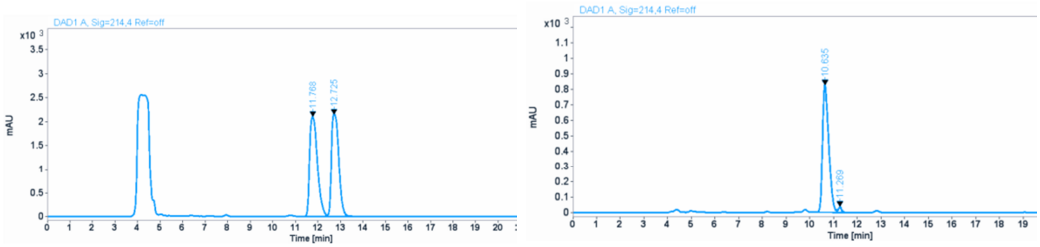
(*R*)-3-(4-(Trifluoromethyl)phenyl)cyclohexanone (3af)[5]



37.7 mg, 79 % yield. $[\alpha]_D^{24}$ +14.6 (*c* 1.30, $CHCl_3$) for 95% ee; [lit.: $[\alpha]_D^{23}$–11.4 (*c* 0.95, $CHCl_3$) for 95% ee in the S-isomer]. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.59 (d, *J* = 8.4 Hz, 2H), 7.33 (d, *J* = 8.4 Hz, 2H), 3.15–3.02 (m, 1H), 2.65–2.30 (m, 4H), 2.25–2.04 (m, 2H), 1.99–1.80 (m, 2H); HPLC: Chiracel AS-H Column (250 mm), detected at 214 nm, *n*-hexane / *i*-propanol = 90/10, flow =

[6] Xue, F.; Li, X.-C. and Wan, B.-S. *J. Org. Chem.* **2011**, *76*, 7256.

0.7 mL/min; Retention time: 10.6 min ((*R*)-**3af**), 11.3 min ((*S*)-**3af**).



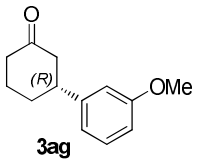
Signal: DAD1 A, Sig=214,4 Ref=off

RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
11.768	0.376	50478.289	2091.914	51.0960	RAC
12.725	0.359	48312.816	2146.366	48.9040	

Signal: DAD1 A, Sig=214,4 Ref=off

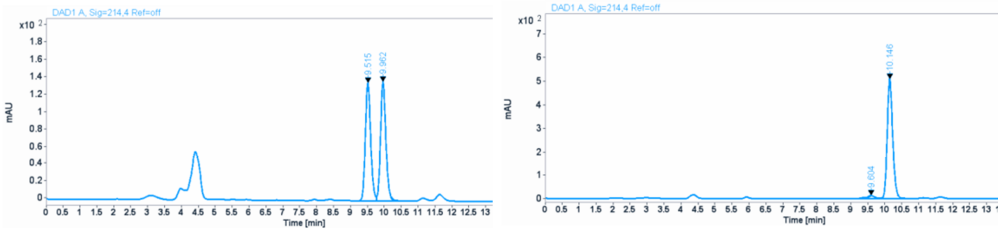
RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
10.635	0.304	15063.067	825.685	97.7048	95.410
11.269	0.168	353.854	35.126	2.2952	

(*R*)-3-(3-Methoxyphenyl)cyclohexanone (3ag) [5]



3ag

31.9 mg, 78% yield. $[\alpha]_D^{24}$+12.8 (*c* 1.41, $CHCl_3$) for 93% ee; [lit.: $[\alpha]_D^{25}$–13.0 (*c* 1.06, $CHCl_3$) for 93% ee in the S-isomer]. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.26–7.22 (m, 1H), 6.82–6.76 (m, 3H), 3.80 (s, 3H), 3.00–2.94 (m, 1H), 2.56–2.36 (m, 4H), 2.16–2.06 (m, 2H), 1.94–1.70 (m, 2H); HPLC: Chiracel AD-H Column (250 mm), detected at 214 nm, *n*-hexane / *i*-propanol = 90/10, flow = 0.7 mL/min; Retention time: 9.6 min ((*S*)-**3ag**), 10.1 min ((*R*)-**3ag**).



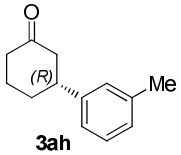
Signal: DAD1 A, Sig=214,4 Ref=off

RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
9.515	0.170	1504.089	136.773	49.6064	RAC
9.962	0.171	1527.960	137.685	50.3936	

Signal: DAD1 A, Sig=214,4 Ref=off

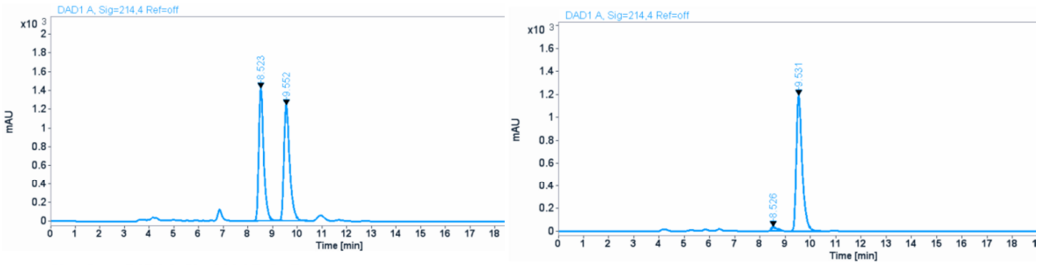
RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
9.604	0.221	201.873	12.880	3.3945	93.211
10.146	0.173	5745.244	510.683	96.6055	

(*R*)-3-(*m*-Tolyl)cyclohexanone (3ah) [5]



3ah

36.9 mg, 98% yield. $[\alpha]_D^{24}$+15.5 (*c* 1.79, $CHCl_3$) for 95% ee; [lit.: $[\alpha]_D^{25}$–17.5 (*c* 1.12, $CHCl_3$) for 94% ee in the S-isomer]. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.18–7.11 (m, 1H), 7.01–6.92 (m, 3H), 2.95–2.84 (m, 1H), 2.52–2.28 (m, 4H), 2.28(s, 3H), 2.14–1.95 (m, 2H), 1.85–1.61 (m, 2H); HPLC: Chiracel OD-H Column (250 mm), detected at 214 nm, *n*-hexane / *i*-propanol = 90/10, flow = 0.7 mL/min; Retention time: 8.5 min ((*S*)-**3ah**), 9.5 min ((*R*)-**3ah**).



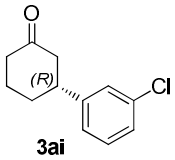
Signal: DAD1 A, Sig=214,4 Ref=off

RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
8.523	0.212	19704.742	1418.466	49.5552	RAC
9.552	0.245	20058.496	1234.481	50.4448	

Signal: DAD1 A, Sig=214,4 Ref=off

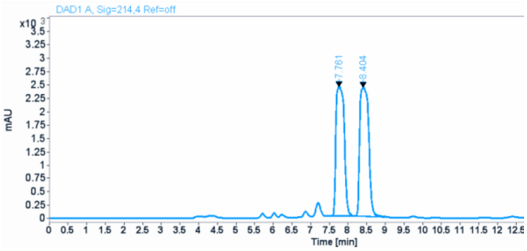
RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
8.526	0.258	495.196	31.977	2.5071	94.986
9.531	0.243	19256.439	1189.541	97.4929	

(*R*)-3-(3-Chlorophenyl)cyclohexanone (3ai)[5]



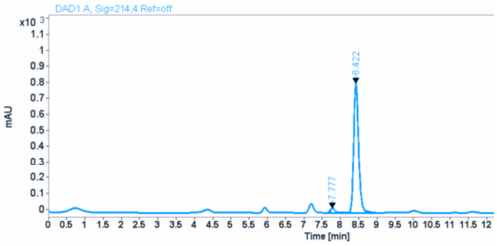
3ai

35.0 mg, 84% yield. $[\alpha]_D^{23}$ +8.3 (*c* 1.18, $CHCl_3$) for 93% ee. [lit.: $[\alpha]_D^{25}$–10.1 (*c* 1.05, $CHCl_3$) for 94% ee in the S-isomer]. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.28–7.21 (m, 3H), 7.09 (d, *J* = 7.2 Hz, 1H), 3.02–2.95 (m, 1H), 2.61–2.33 (m, 4H), 2.18–2.09 (m, 2H), 1.88–1.72 (m, 2H); HPLC: Chiracel AD-H Column (250 mm), detected at 214 nm, *n*-hexane / *i*-propanol = 90/10, flow = 0.7 mL/min; Retention time: 7.8 min ((*S*)-**3ai**), 8.4 min ((*R*)-**3ai**).



Signal: DAD1 A, Sig=214,4 Ref=off

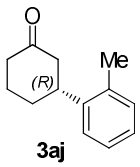
RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
7.761	0.258	37483.539	2417.237	48.1094	RAC
8.404	0.280	40429.543	2409.860	51.8906	



Signal: DAD1 A, Sig=214,4 Ref=off

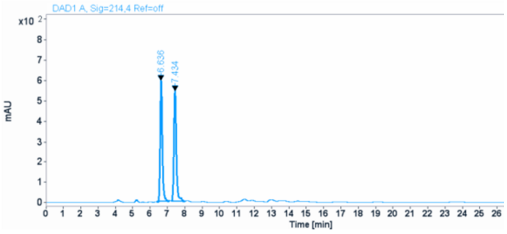
RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
7.777	0.137	260.868	29.092	3.3071	93.386
8.422	0.143	7627.288	814.104	96.6929	

(*R*)-3-(*o*-Tolyl)cyclohexanone (3aj)[6]



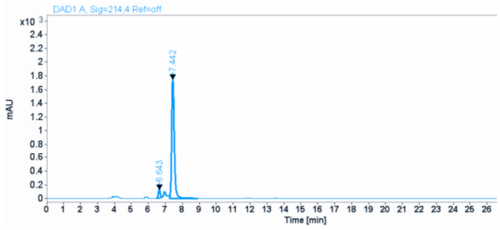
3aj

37.4 mg, 99% yield. $[\alpha]_D^{24}$ +29.3 (*c* 1.84, $CHCl_3$) for 90% ee. [lit.: $[\alpha]_D^{20}$–32.6 (*c* 1.0, $CHCl_3$) for 90% ee in the S-isomer]. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.25–7.20 (m, 2H), 7.19–7.13 (m, 2H), 3.25–3.16 (m, 1H), 2.52–2.50 (m, 2H), 2.47–2.38 (m, 2H), 2.32 (s, 3H), 2.20–2.15 (m, 1H), 2.02–2.00 (m, 1H), 1.89–1.76 (m, 2H); HPLC: Chiracel AD-H Column (250 mm); detected at 214 nm,*n*-hexane / *i*-propanol = 90/10, flow = 0.7 mL/min; Retention time: 6.6 min ((*S*)-**3aj**), 7.4 min ((*R*)-**3aj**).



Signal: DAD1 A, Sig=214,4 Ref=off

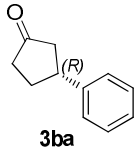
RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
6.636	0.143	5106.898	594.834	49.3935	RAC
7.434	0.160	5232.304	543.588	50.6065	



Signal: DAD1 A, Sig=214,4 Ref=off

RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
6.643	0.129	919.676	119.170	4.6302	90.740
7.442	0.181	18942.680	1743.891	95.3698	

(*R*)-3-Phenylcyclopentanone (3ba) [7]

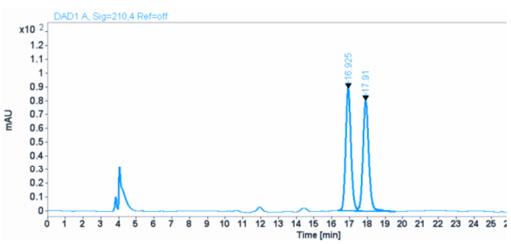


3ba

19.4 mg, 61% yield. $[\alpha]_D^{24}$ +35.7 (*c* 0.40, $CHCl_3$) for 94% ee. [lit.: $[\alpha]_D^{20}$ –92 (*c* 0.82, $CHCl_3$) for 97% ee in the S-isomer]. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.36–7.33 (m, 2H), 7.26–7.29 (m, 3H), 3.47–3.38 (m, 1H), 2.67(dd, *J* = 18.0 Hz, *J* = 7.6 Hz, 1H), 2.50–2.41 (m, 2H), 2.35–2.28 (m, 2H), 2.05–1.94 (m, 1H); HPLC: Chiracel AS-H Column (250 mm); detected at 214 nm;

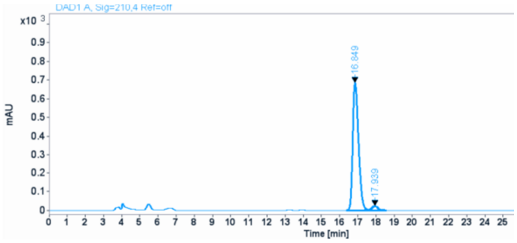
[7] Takaya, Y.; Ogasawara, M. and Hayashi, T.; *J. Am. Chem. Soc.* **1998**, *120*, 5579

n-hexane / *i*-propanol = 98/2; flow = 0.7 mL/min; Retention time: 16.8 min ((*R*)-**3ba**), 17.9 min ((*S*)-**3ba**).



Signal: DAD1 A, Sig=210,4 Ref=off

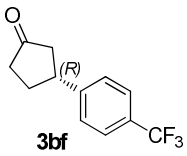
RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
16.925	0.318	1820.762	88.689	50.1615	RAC
17.910	0.345	1809.035	80.137	49.8385	



Signal: DAD1 A, Sig=210,4 Ref=off

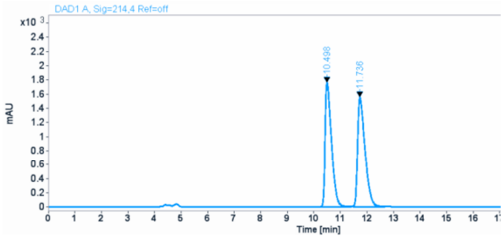
RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
16.849	0.343	15072.535	684.762	96.8592	93.718
17.939	0.343	488.745	22.001	3.1408	

(*R*)-3-(4-(Trifluoromethyl)phenyl)cyclopentanone (3bf) [5]



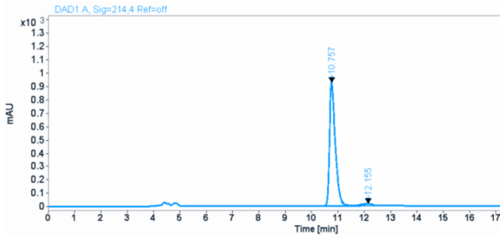
3bf

31.5 mg, 69% yield. $[\alpha]_D^{24}$ +52.6 (*c* 1.58, $CHCl_3$) for 94% ee. [lit.: $[\alpha]_D^{24}$ –52.1 (*c* 1.36, CHCl3) for 94% ee in the S-isomer]. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.60 (d, *J* = 8.0 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 3.53–3.44 (m, 1H), 2.70 (dd, *J* = 18.0 Hz, *J* = 7.6 Hz, 1H), 2.52–2.45 (m, 2H), 2.38–2.28 (m, 2H), 2.06–1.85 (m, 1H). HPLC: Chiracel OB-H Column (250 mm); detected at 214 nm; *n*-hexane / *i*-propanol = 90/10; flow = 0.7 mL/min; Retention time: 10.7 min ((*R*)-**3bf**), 12.2 min ((*S*)-**3bf**).



Signal: DAD1 A, Sig=214,4 Ref=off

RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
10.498	0.253	29540.668	1764.045	49.5036	RAC
11.736	0.323	30133.076	1555.231	50.4964	



Signal: DAD1 A, Sig=214,4 Ref=off

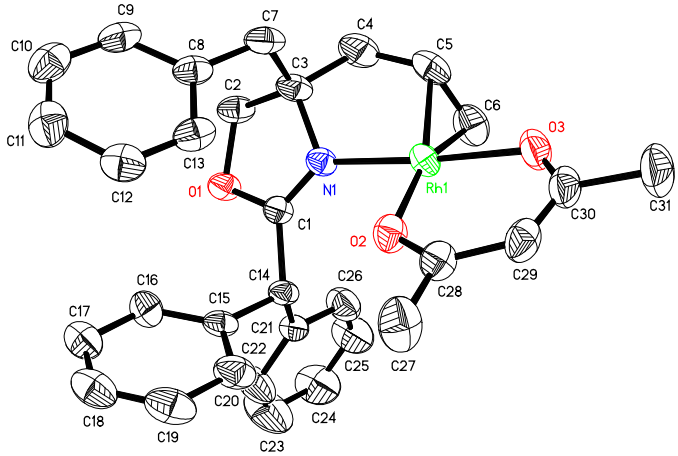
RT [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area%	ee value%
10.757	0.253	14000.456	922.796	97.1062	94.212
12.155	0.432	417.217	16.092	2.8938	

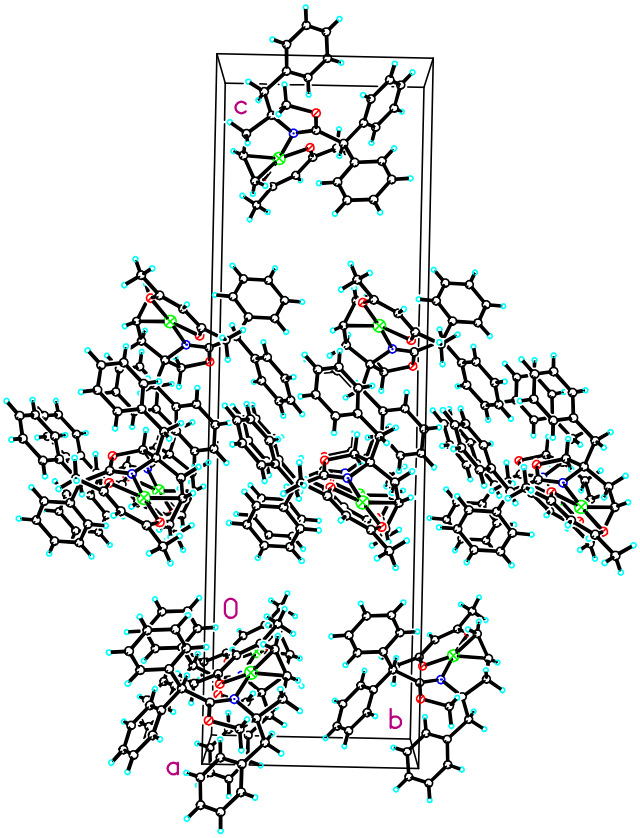
4. X-Ray Single Crystal Structure Of [Rh(L10)(acac)] And [Rh(L11)(acac)]

4.1 Preparation of [Rh(L10)(acac)] and [Rh(L11)(acac)]

To a small vessel was added $[Rh(C_2H_4)_2(acac)]$ (10 mg, 0.04 mmol), **L10/L11** (0.056 mmol, 1.4 equiv) and 1,4-dioxane (0.1 mL). The small vessel was placed in a sealed bottle, which was charged with n-pentane (5 mL). After the solvent exchange of 1,4-dioxane and n-pentane for 4 days, the red-brown crystals were obtained.

4.2 X-Ray Single Crystal Structure Of [Rh(L10)(acac)]

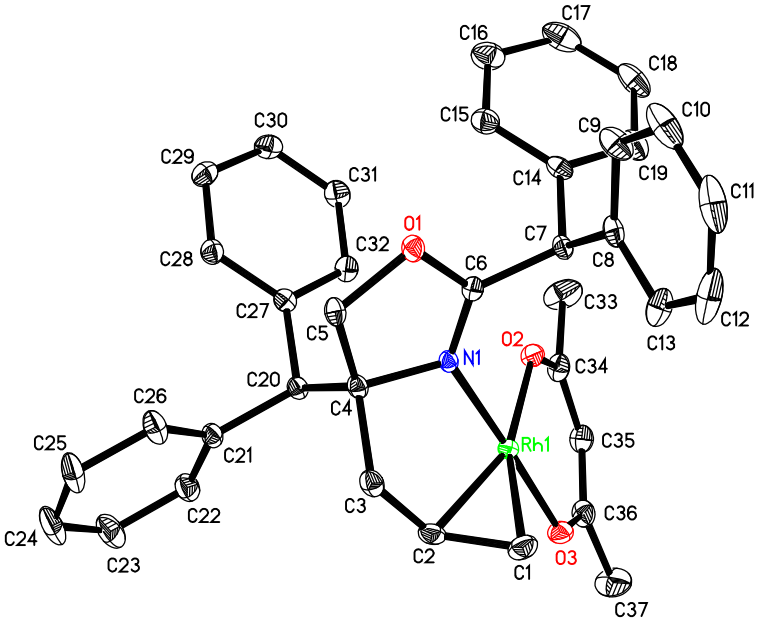

C9
C10
C8
C7
C4
C5
C3
C2
C6
O3
C11
C13
N1
Rh1
C12
O1
C1
C30
C31
O2
C29
C28
C16
C14
C26
C17
C15
C21
C22
C25
C27
C20
C24
C18
C19
C23

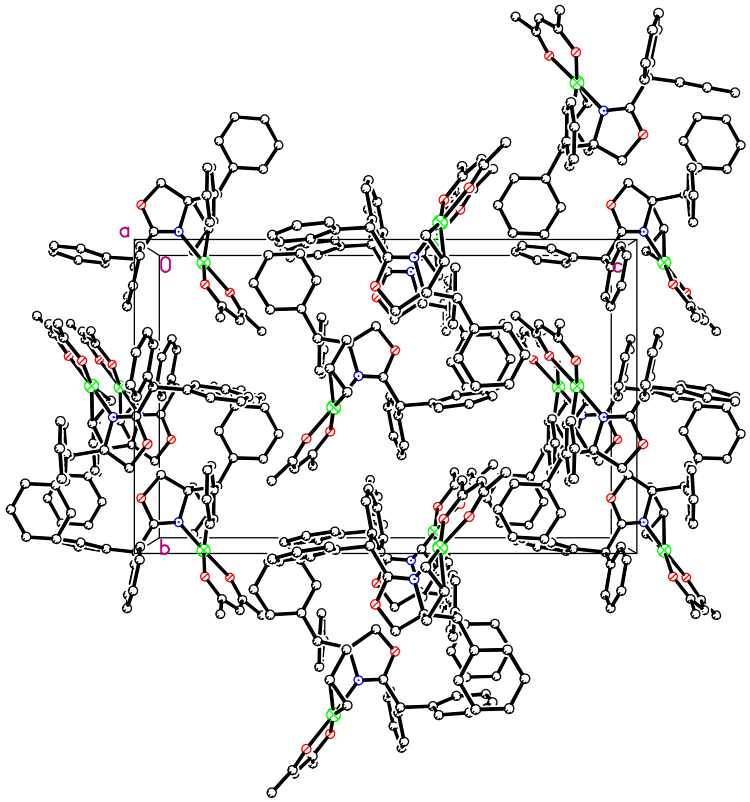

c
0
b
a

CCDC 1448857 for [Rh(L10)(acac)]

Identification code	cd214540	
Empirical formula	C31 H32 N O3 Rh	
Formula weight	569.48	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P 21 21 21	
Unit cell dimensions	a = 9.7915(7) Å	= 90°.
	b = 10.0496(6) Å	= 90°.
	c = 33.148(2) Å	= 90°.
Volume	3261.8(4) $Å^3$	
Z	4	
Density (calculated)	1.160 Mg/m^3	
Absorption coefficient	0.550 mm^{-1}	
F(000)	1176	
Crystal size	0.211 x 0.154 x 0.086 mm^3	
Theta range for data collection	2.118 to 25.994°.	
Index ranges -10<=k<=12, -40<=l<=40	-12<=h<=11,	
Reflections collected	19903	
Independent reflections	6411 [R(int) = 0.0428]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from eqalents	
Max. and min. transmission	0.7457 and 0.6123	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6411 / 0 / 327	
Goodness-of-fit on F^2	1.021	
Final R indices [I>2sigma(I)]	R1 = 0.0419, wR2 = 0.0963	
R indices (all data)	R1 = 0.0499, wR2 = 0.1004	
Absolute structure parameter	0.011(18)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.516 and -0.401 $e.Å^{-3}$	

4.3 X-Ray Single Crystal Structure Of [Rh(L11)(acac)]

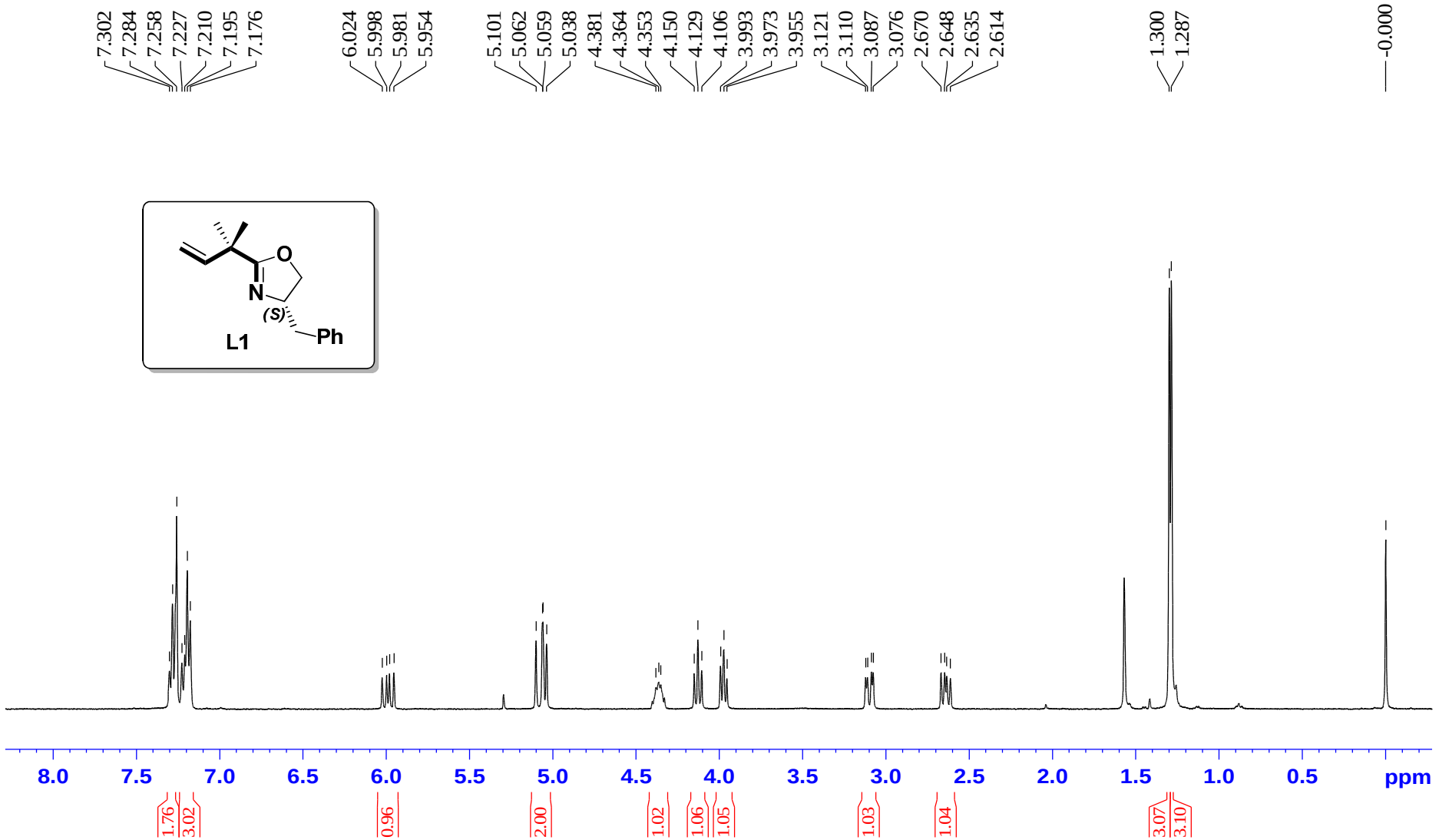




CCDC 1448858 for [Rh(L11)(acac)]

Identification code	mo_dm15248_0m	
Empirical formula	C37 H36 N O3 Rh	
Formula weight	645.58	
Temperature	130 K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P 21 21 21	
Unit cell dimensions	a = 10.8993(7) Å	= 90°.
	b = 13.2135(8) Å	= 90°.
	c = 21.2588(13) Å	= 90°.
Volume	3061.6(3) $Å^3$	
Z	4	
Density (calculated)	1.401 Mg/m^3	
Absorption coefficient	0.595 mm^{-1}	
F(000)	1336	
Crystal size	0.2 x 0.18 x 0.15 mm^3	
Theta range for data collection	1.815 to 30.529°.	
Index ranges	-15<=h<=15, -18<=k<=18, -28<=l<=30	
Reflections collected	31142	
Independent reflections	9356 [R(int) = 0.0373]	
Completeness to theta = 26.000°	100.0 %	
Absorption correction	Semi-empirical from eqalents	
Max. and min. transmission	0.7461 and 0.6898	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9356 / 0 / 381	
Goodness-of-fit on F^2	1.018	
Final R indices [I>2sigma(I)]	R1 = 0.0300, wR2 = 0.0607	
R indices (all data)	R1 = 0.0347, wR2 = 0.0626	
Absolute structure parameter	-0.010(10)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.479 and -0.467 $e.Å^{-3}$	

5. ^{1}H NMR,^{13}CNMR Copies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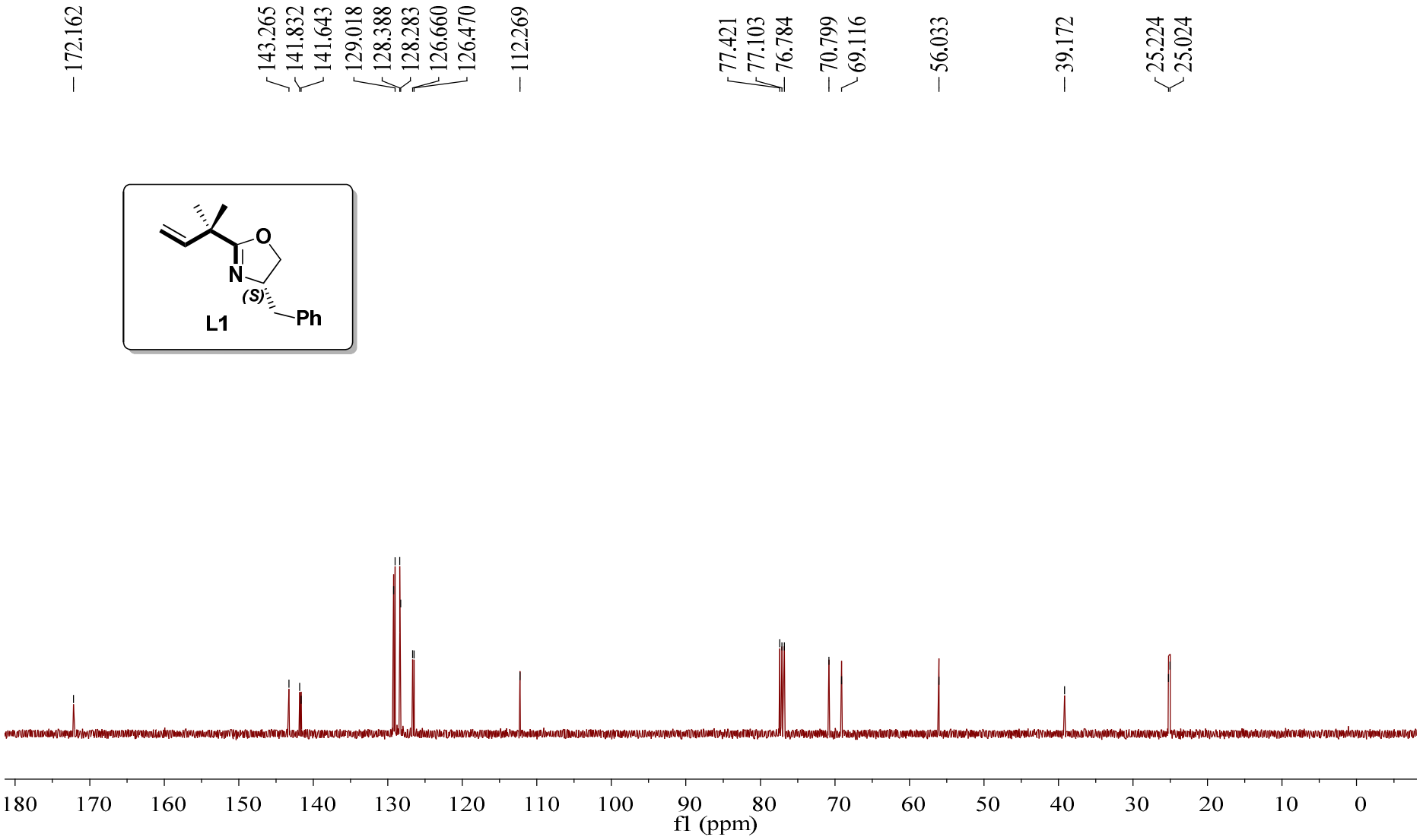
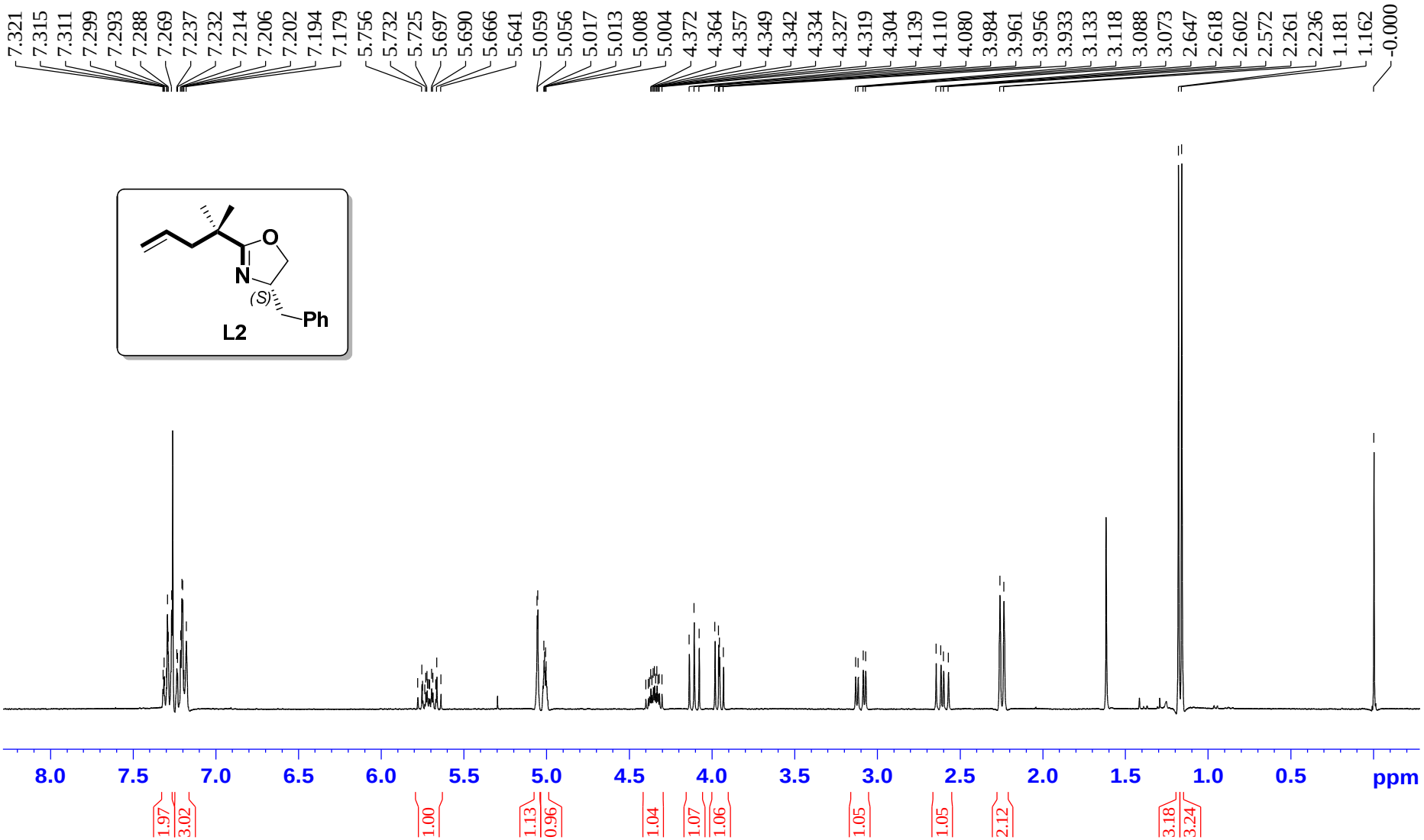
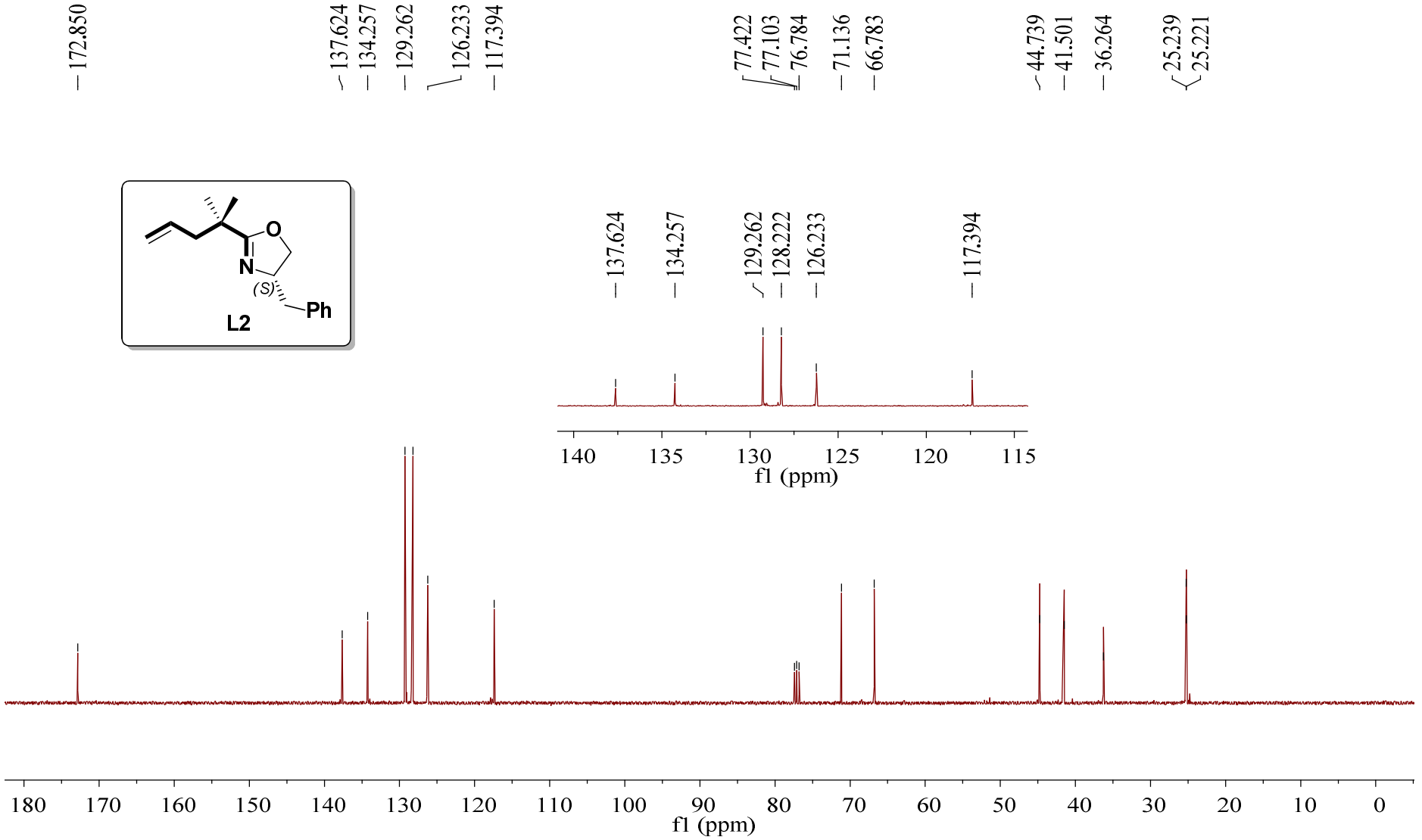
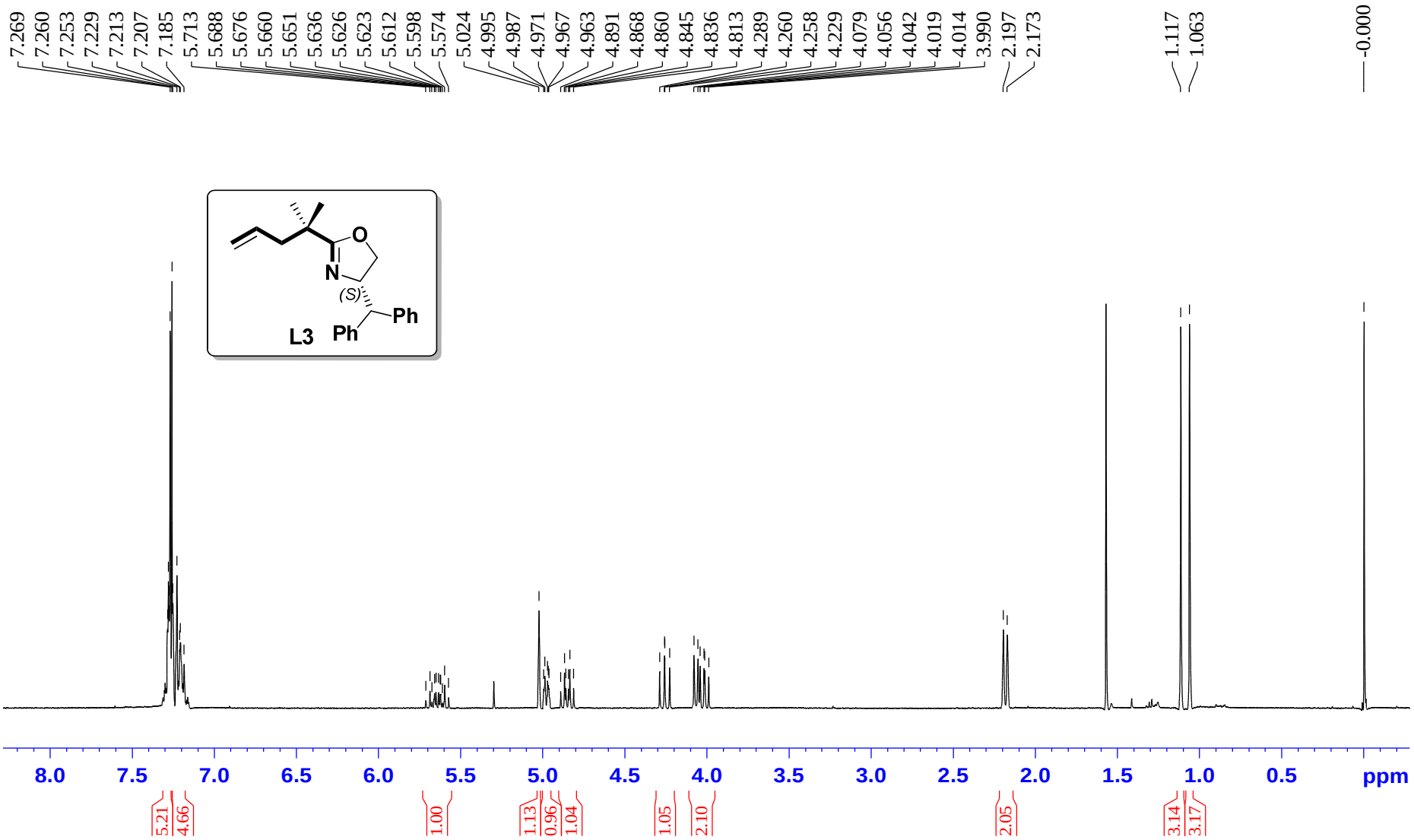
7.258
7.227
7.210
7.195
7.176
6.024
5.998
5.981
5.954
5.101
5.062
5.059
5.038
4.381
4.364
4.353
4.150
4.129
4.106
3.993
3.973
3.955
3.121
3.110
3.087
3.076
2.670
2.648
2.635
2.614
1.300
1.287
-0.000
O
N
(S)
Ph
L1
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.76
3.02
0.96
2.00
1.02
1.06
1.05
1.03
1.04
3.07
3.10


172.162
143.265
141.832
141.643
129.018
128.388
128.283
126.660
126.470
112.269
77.421
77.103
76.784
70.799
69.116
56.033
39.172
25.224
25.024
O
N
(S)
Ph
L1
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.321
7.315
7.311
7.299
7.293
7.288
7.269
7.237
7.232
7.214
7.206
7.202
7.194
7.179
5.756
5.732
5.725
5.697
5.690
5.666
5.641
5.059
5.056
5.017
5.013
5.008
5.004
4.372
4.364
4.357
4.349
4.342
4.334
4.327
4.319
4.304
4.139
4.110
4.080
3.984
3.961
3.956
3.933
3.133
3.118
3.088
3.073
2.647
2.618
2.602
2.572
2.261
2.236
1.181
1.162
-0.000
O
N
(S)
Ph
L2
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.97
3.02
1.00
1.13
0.96
1.04
1.07
1.06
1.05
1.05
2.12
3.18
3.24


172.850
137.624
134.257
129.262
126.233
117.394
77.422
77.103
76.784
71.136
66.783
44.739
41.501
36.264
25.239
25.221
O
N
(S)
Ph
L2
137.624
134.257
129.262
128.222
126.233
117.394
140
135
130
125
120
115
f1 (ppm)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.269
7.260
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7.229
7.213
7.207
7.185
5.713
5.688
5.676
5.660
5.651
5.636
5.626
5.623
5.612
5.598
5.574
5.024
4.995
4.987
4.971
4.967
4.963
4.891
4.868
4.860
4.845
4.836
4.813
4.289
4.260
4.258
4.229
4.079
4.056
4.042
4.019
4.014
3.990
2.197
2.173
1.117
1.063
-0.000
O
N
(S)
Ph
Ph
L3
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
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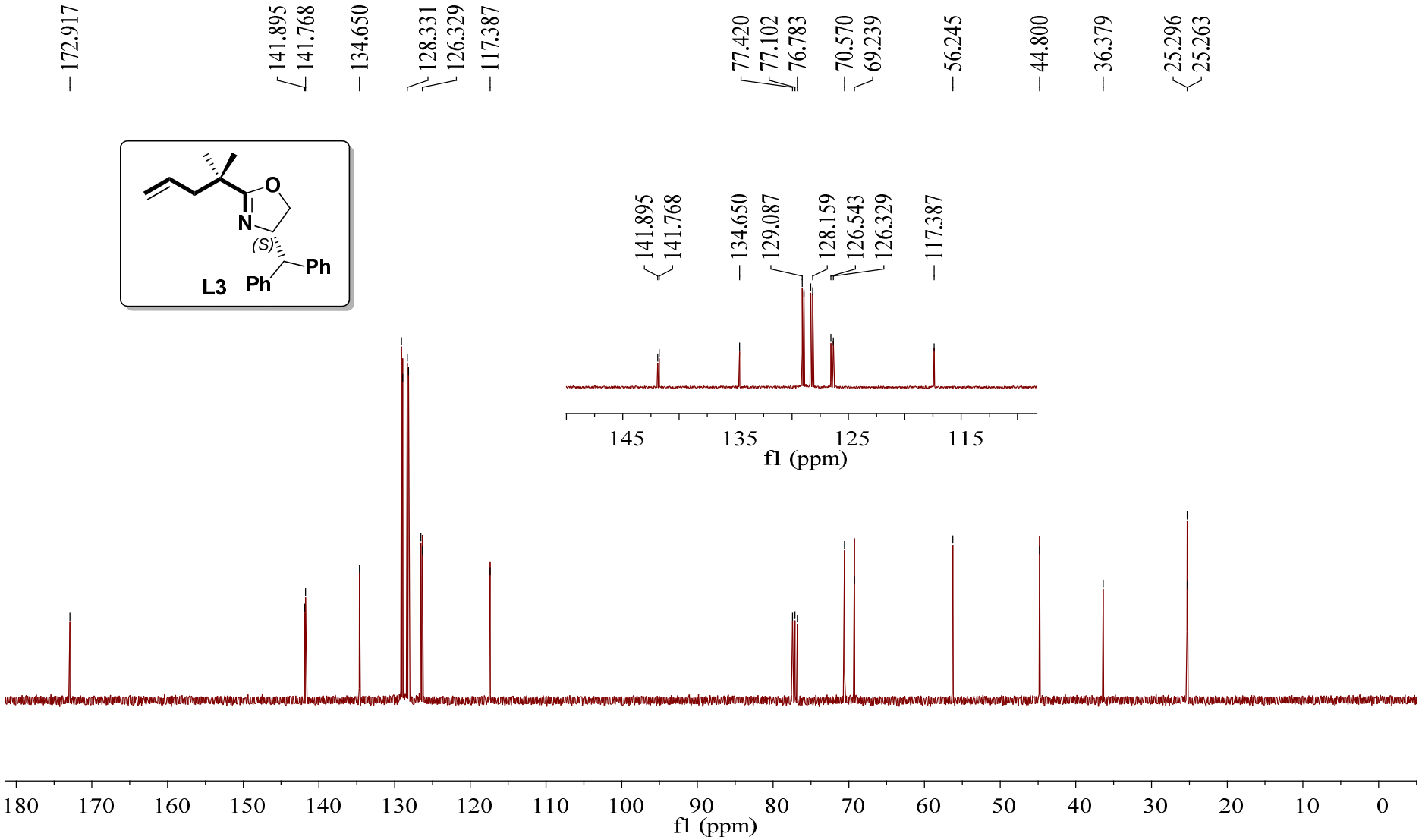
0.96
1.04
1.05
2.10
2.05
3.14
3.17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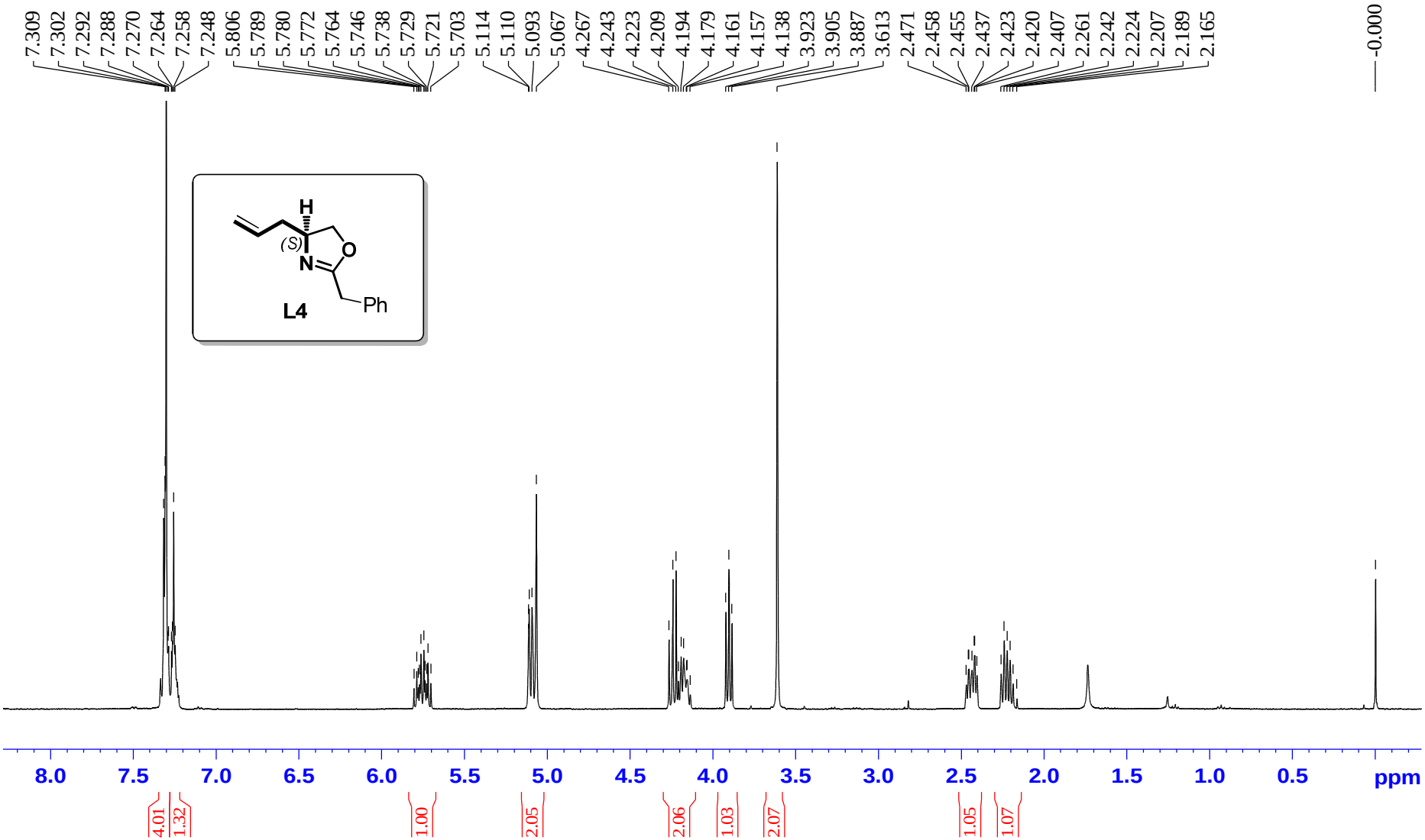
141.895
141.768
134.650
128.331
126.329
117.387
77.420
77.102
76.783
70.570
69.239
56.245
44.800
36.379
25.296
25.263
O
N
(S)
Ph
Ph
L3
141.895
141.768
134.650
129.087
128.159
126.543
126.329
117.387
145
135
125
115
f1 (ppm)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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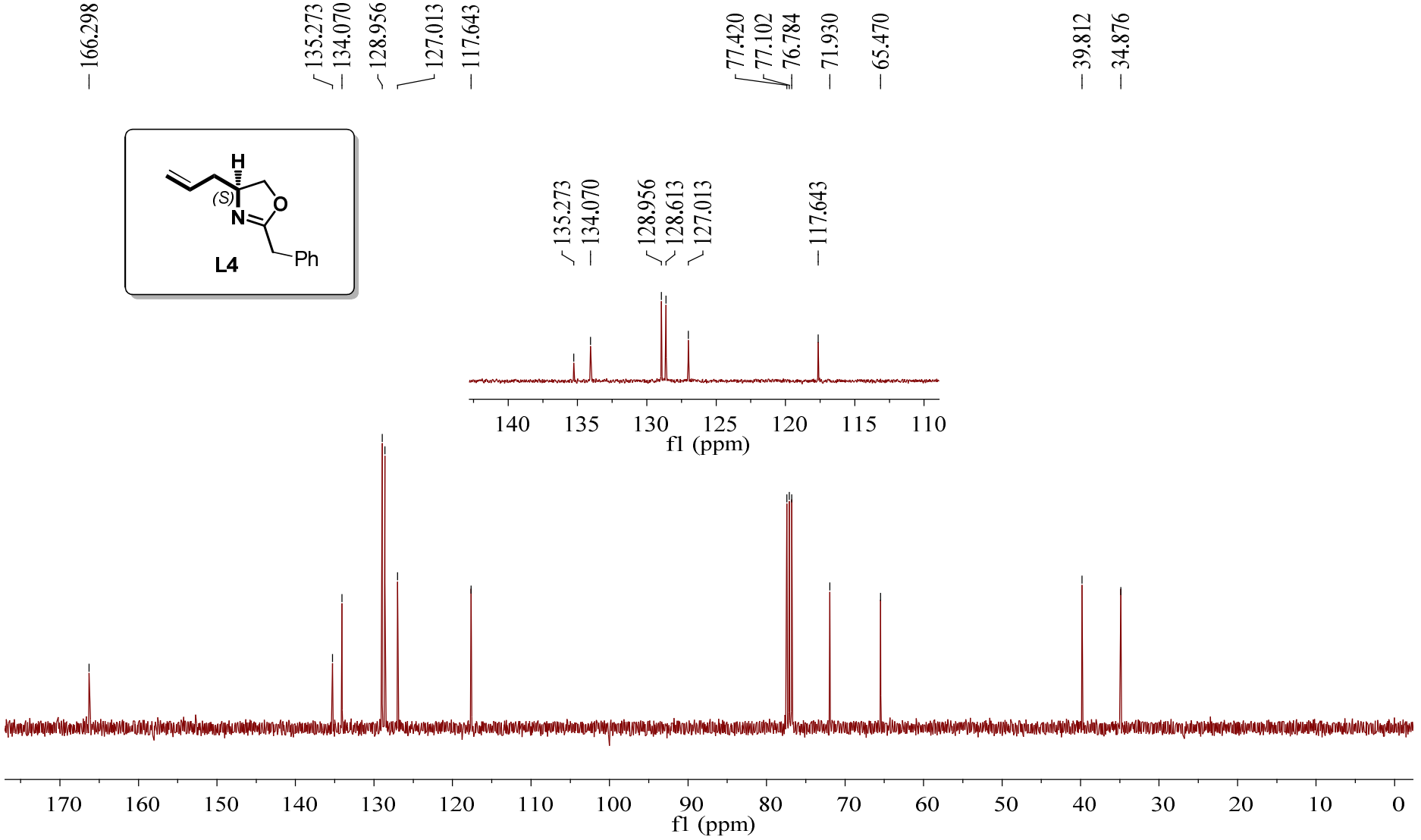
7.302
7.292
7.288
7.270
7.264
7.258
7.248
5.806
5.789
5.780
5.772
5.764
5.746
5.738
5.729
5.721
5.703
5.114
5.110
5.093
5.067
4.267
4.243
4.223
4.209
4.194
4.179
4.161
4.157
4.138
3.923
3.905
3.887
3.613
2.471
2.458
2.455
2.437
2.423
2.420
2.407
2.261
2.242
2.224
2.207
2.189
2.165
-0.000
H
(S)
N
O
Ph
L4
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.01
1.32
1.00
2.05
2.06
1.03
2.07
1.05
1.07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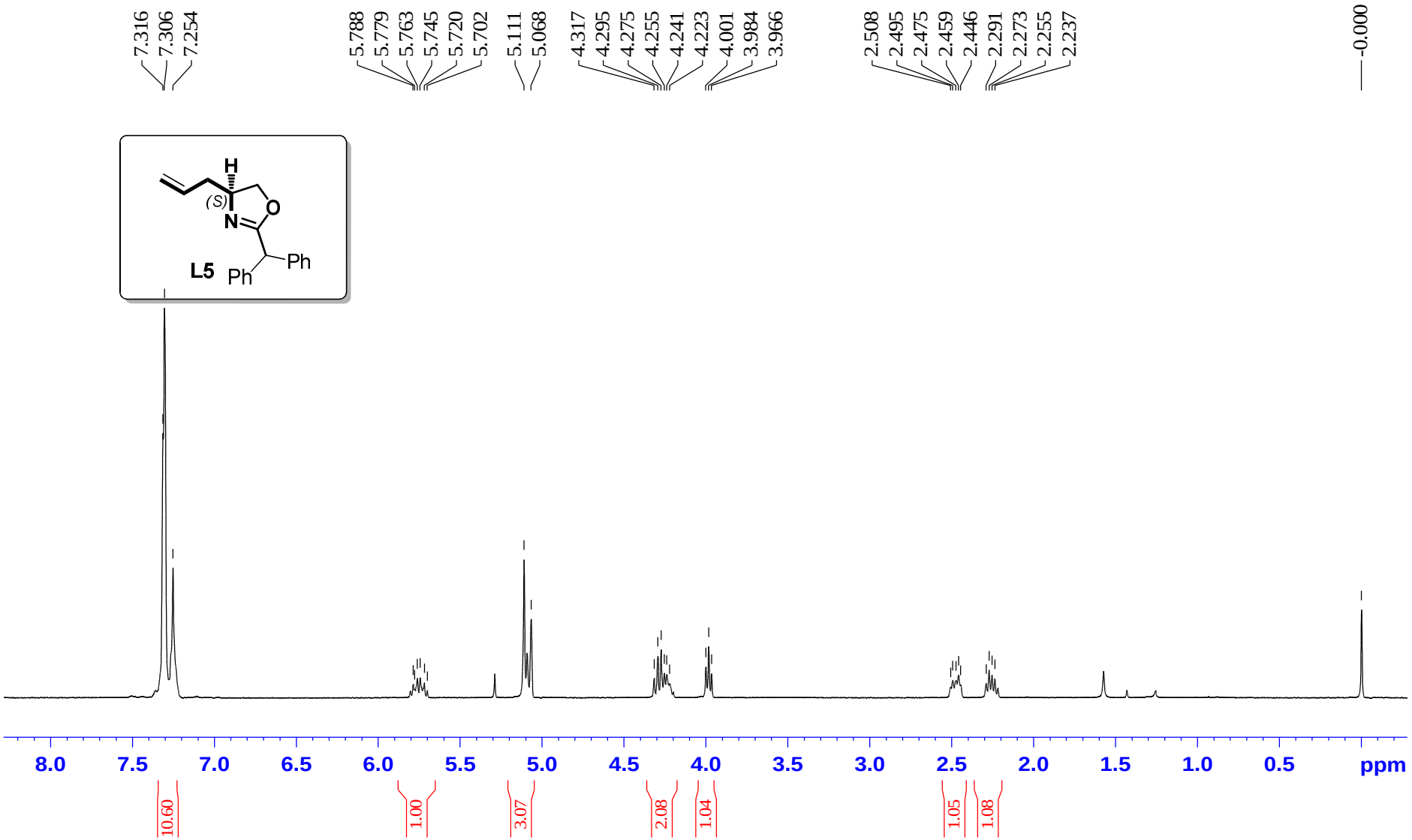
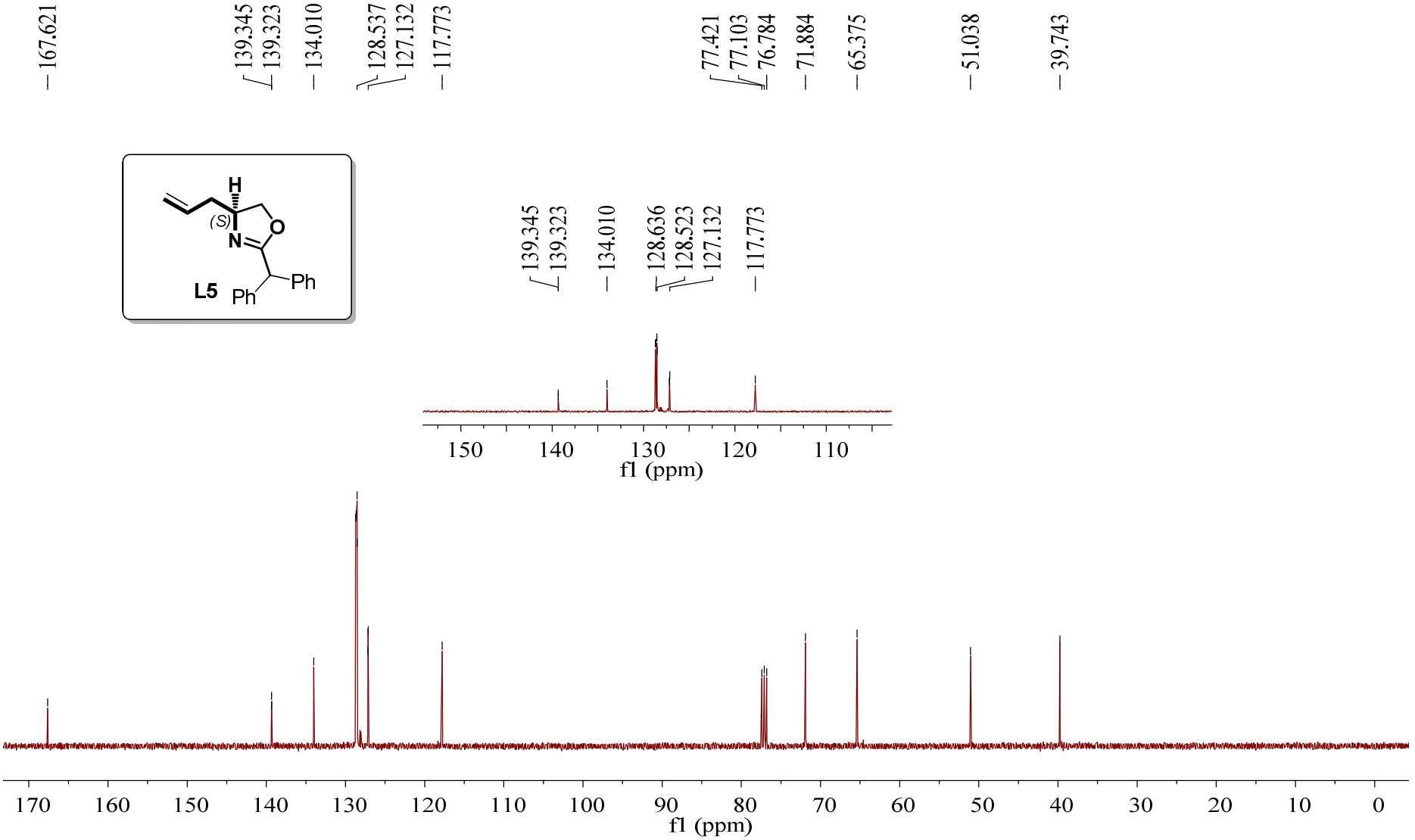
135.273
134.070
128.956
127.013
117.643
77.420
77.102
76.784
71.930
65.470
39.812
34.876
H
(S)
N
O
Ph
L4
135.273
134.070
128.956
128.613
127.013
117.643
140
135
130
125
120
115
110
f1 (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.316
7.306
7.254
5.788
5.779
5.763
5.745
5.720
5.702
5.111
5.068
4.317
4.295
4.275
4.255
4.241
4.223
4.001
3.984
3.966
2.508
2.495
2.475
2.459
2.446
2.291
2.273
2.255
2.237
-0.000
H
(S)
N
O
Ph
L5
Ph
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
10.60
1.00
3.07
2.08
1.04
1.05
1.08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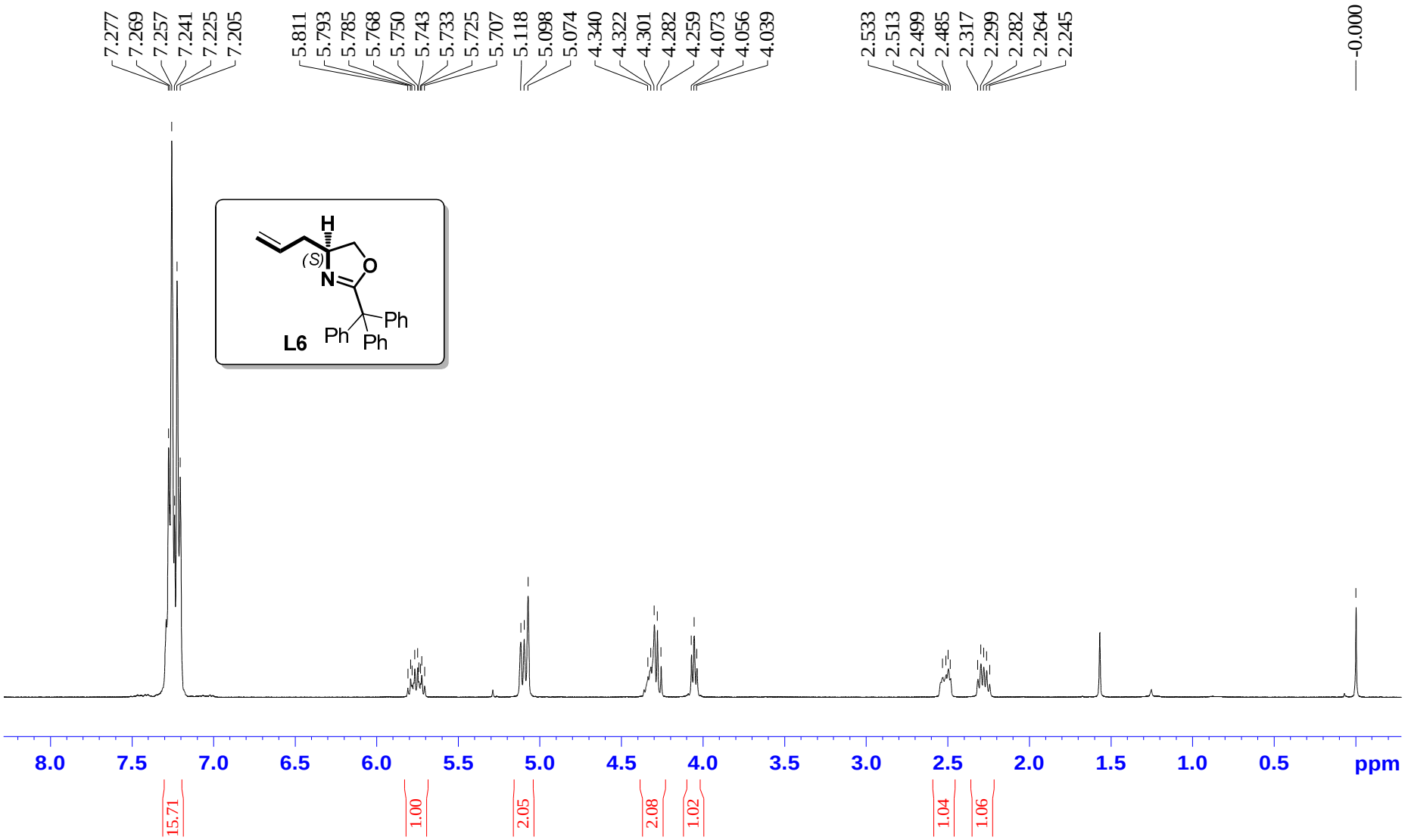
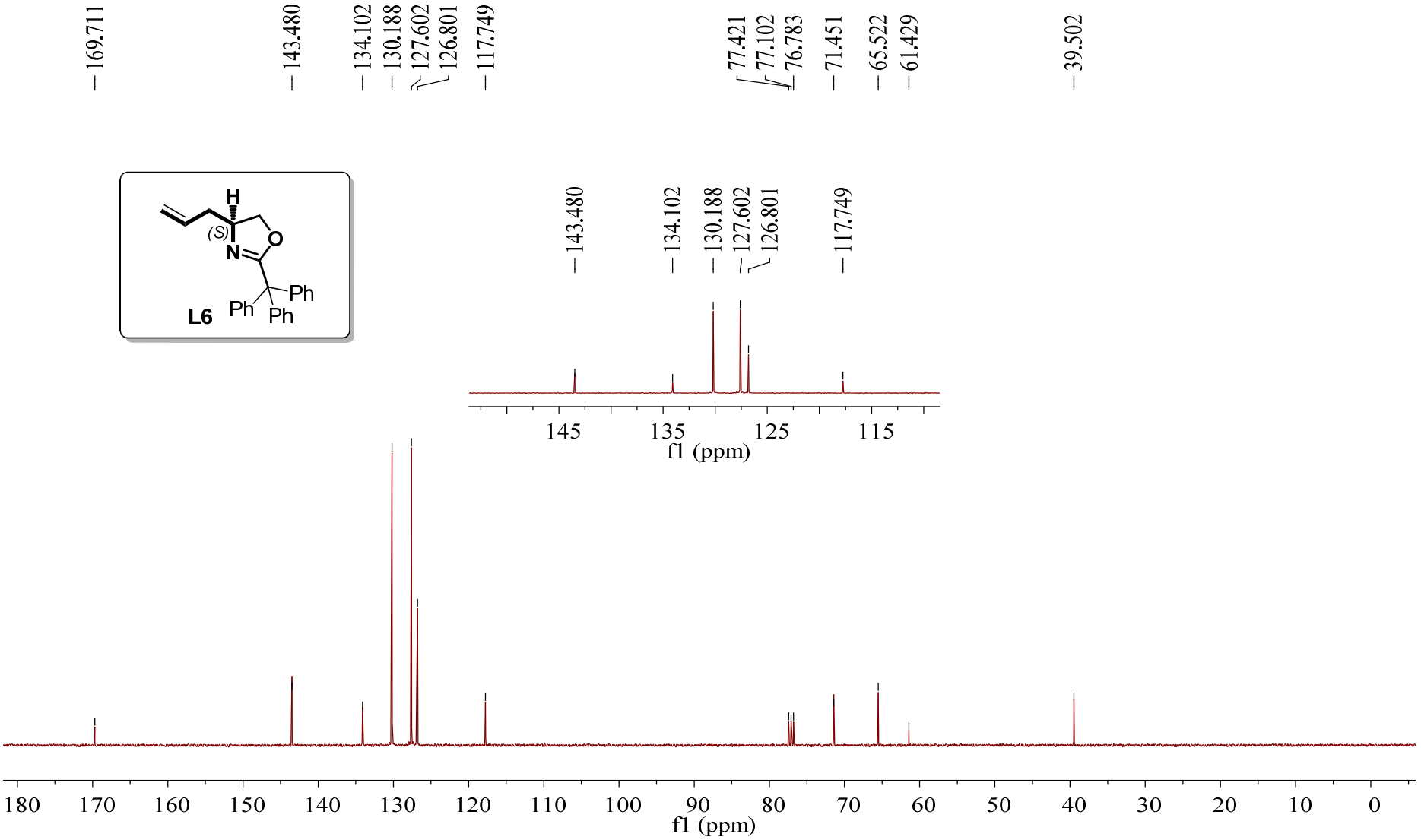
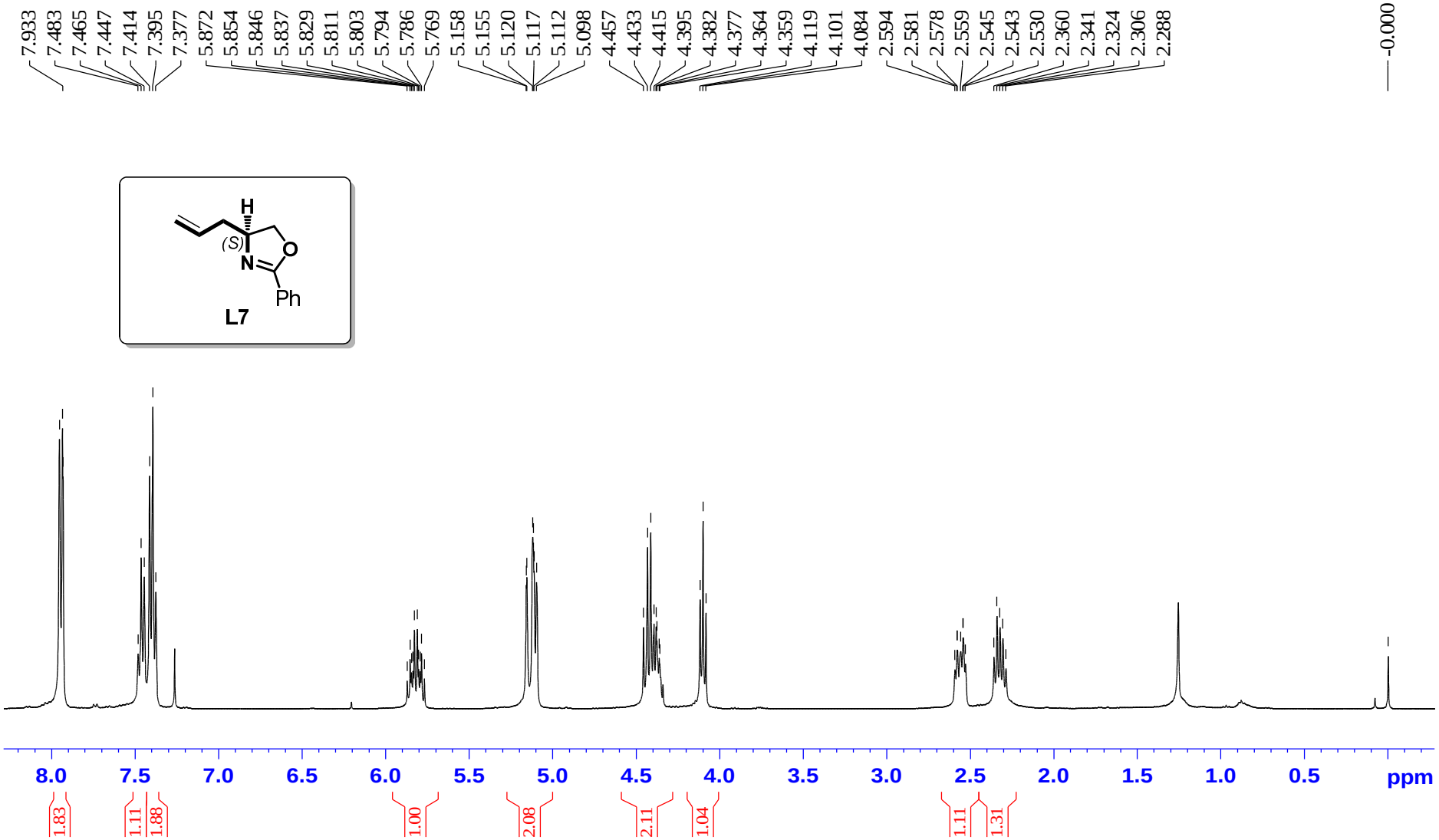
139.345
139.323
134.010
128.537
127.132
117.773
77.421
77.103
76.784
71.884
65.375
51.038
39.743
L5
Ph
Ph
150
140
130
120
110
f1 (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.277
7.269
7.257
7.241
7.225
7.205
5.811
5.793
5.785
5.768
5.750
5.743
5.733
5.725
5.707
5.118
5.098
5.074
4.340
4.322
4.301
4.282
4.259
4.073
4.056
4.039
2.533
2.513
2.499
2.485
2.317
2.299
2.282
2.264
2.245
-0.000
H
(S)
N
O
Ph
Ph
Ph
L6
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
15.71
1.00
2.05
2.08
1.02
1.04
1.06


169.711
143.480
134.102
130.188
127.602
126.801
117.749
77.421
77.102
76.783
71.451
65.522
61.429
39.502
H
(S)
N
O
Ph
Ph
Ph
L6
143.480
134.102
130.188
127.602
126.801
117.749
145
135
125
115
f1 (ppm)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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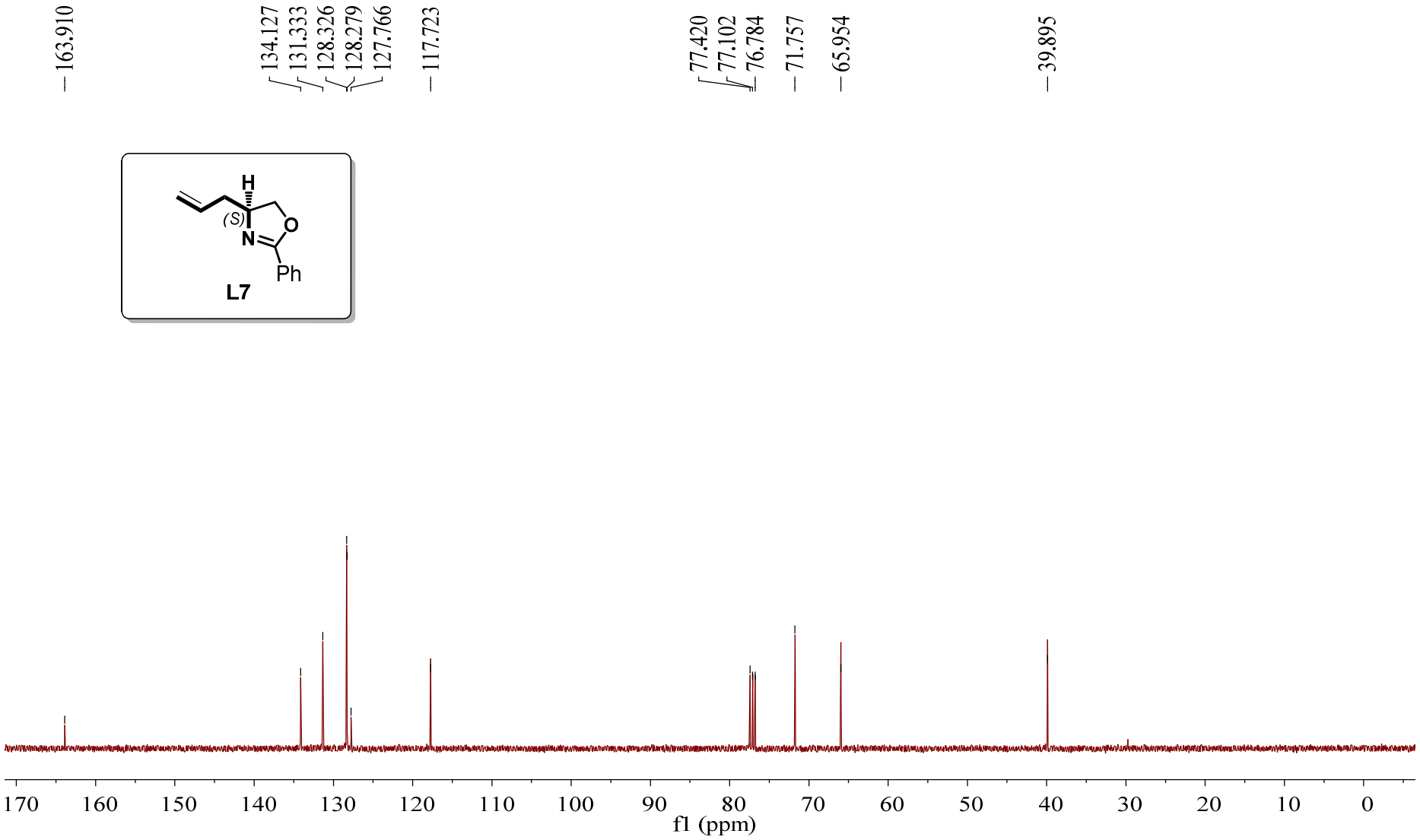
7.483
7.465
7.447
7.414
7.395
7.377
5.872
5.854
5.846
5.837
5.829
5.811
5.803
5.794
5.786
5.769
5.158
5.155
5.120
5.117
5.112
5.098
4.457
4.433
4.415
4.395
4.382
4.377
4.364
4.359
4.119
4.101
4.084
2.594
2.581
2.578
2.559
2.545
2.543
2.530
2.360
2.341
2.324
2.306
2.288
-0.000
H
(S)
N
O
Ph
L7
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.83
1.11
1.88
1.00
2.08
2.11
1.04
1.11
1.31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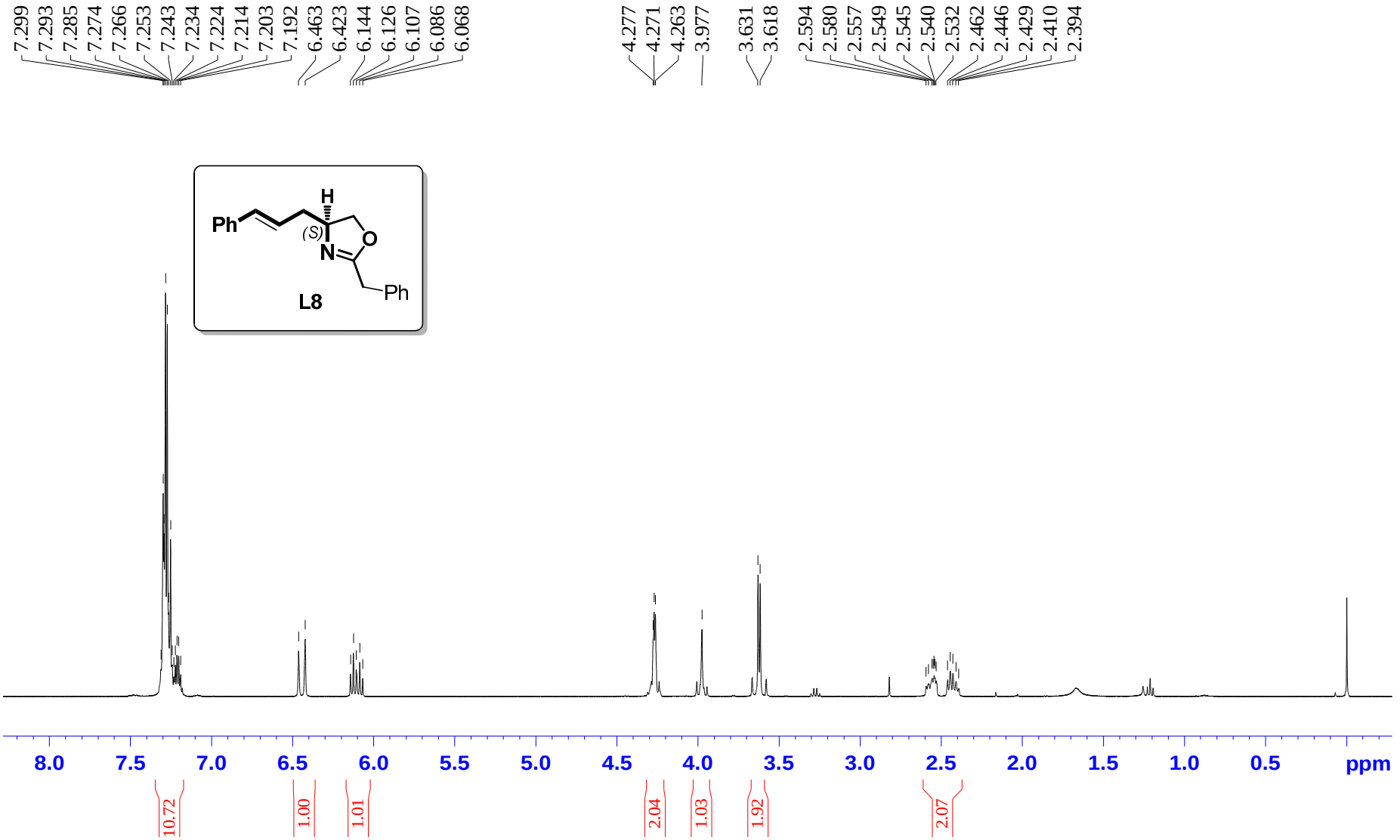
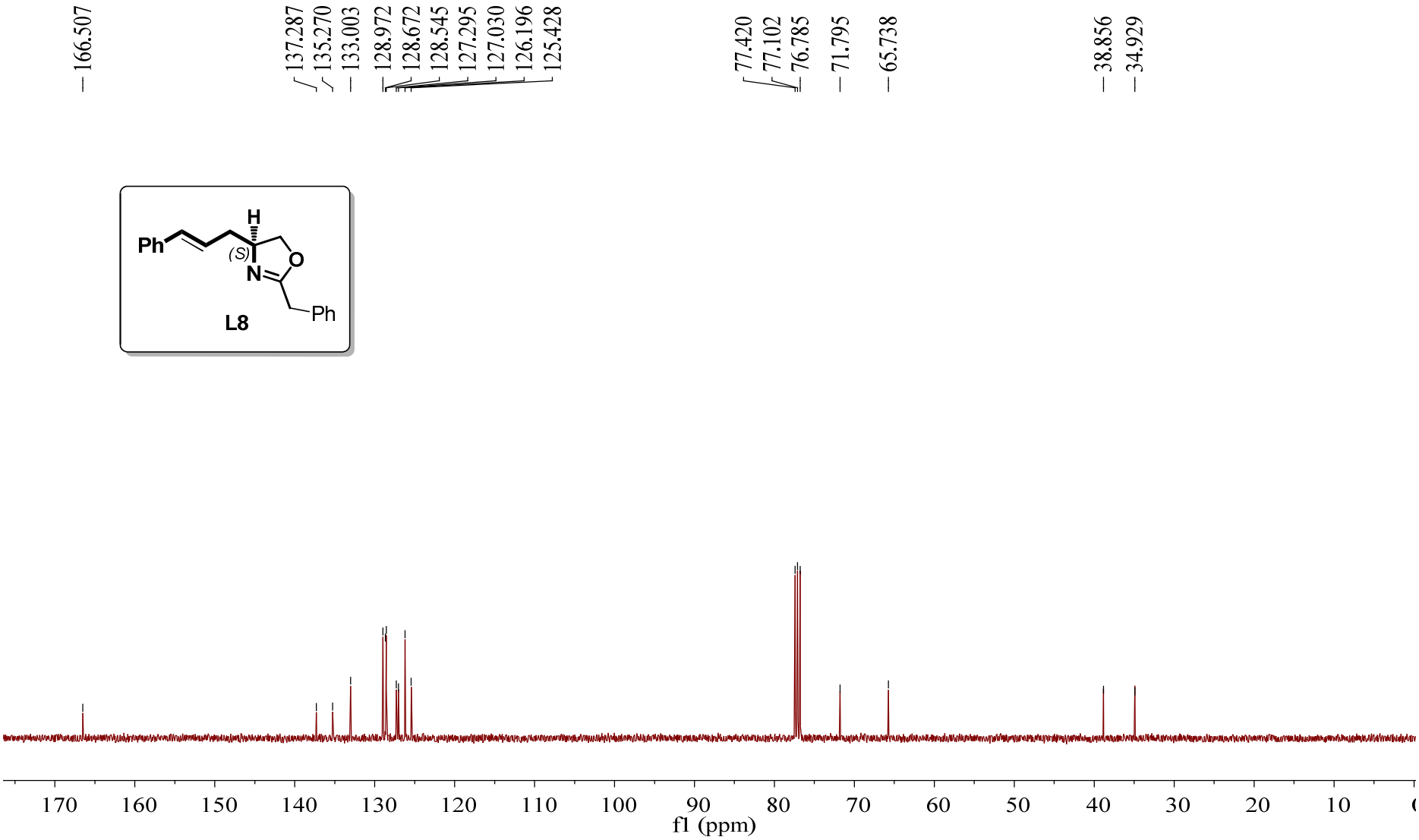
134.127
131.333
128.326
128.279
127.766
117.723
77.420
77.102
76.784
71.757
65.954
39.895
H
(S)
N
O
Ph
L7
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.299
7.293
7.285
7.274
7.266
7.253
7.243
7.234
7.224
7.214
7.203
7.192
6.463
6.423
6.144
6.126
6.107
6.086
6.068
4.277
4.271
4.263
3.977
3.631
3.618
2.594
2.580
2.557
2.549
2.545
2.540
2.532
2.462
2.446
2.429
2.410
2.394
Ph
H
(S)
N
O
Ph
L8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
10.72
1.00
1.01
2.04
1.03
1.92
2.07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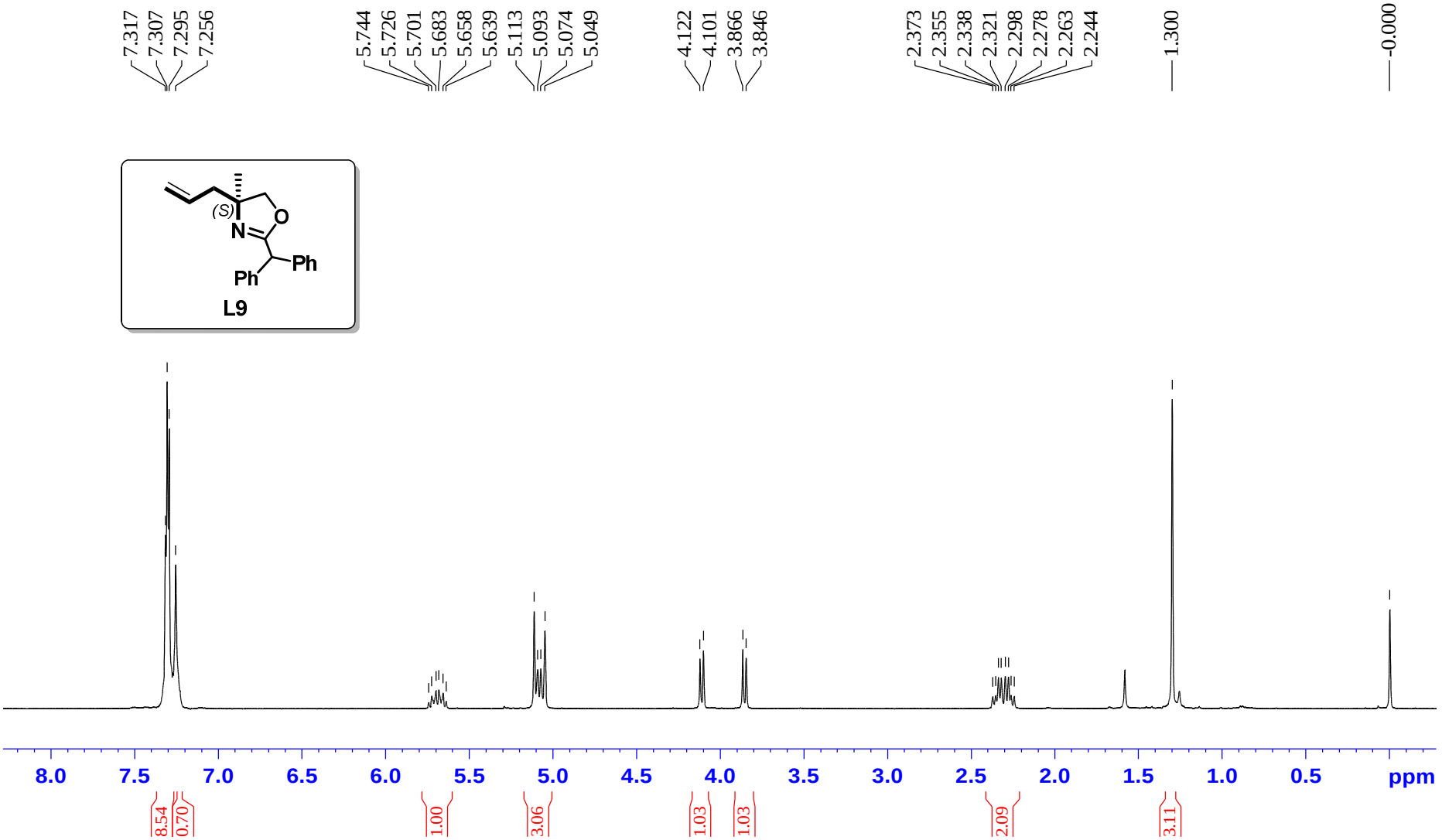
135.270
133.003
128.972
128.672
128.545
127.295
127.030
126.196
125.428
77.420
77.102
76.785
71.795
65.738
38.856
34.929
Ph
H
(S)
N
O
Ph
L8
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.295
7.256
5.744
5.726
5.701
5.683
5.658
5.639
5.113
5.093
5.074
5.049
4.122
4.101
3.866
3.846
2.373
2.355
2.338
2.321
2.298
2.278
2.263
2.244
1.300
-0.000
(S)
N
O
Ph
Ph
L9
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
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0.5
ppm
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0.70
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3.06
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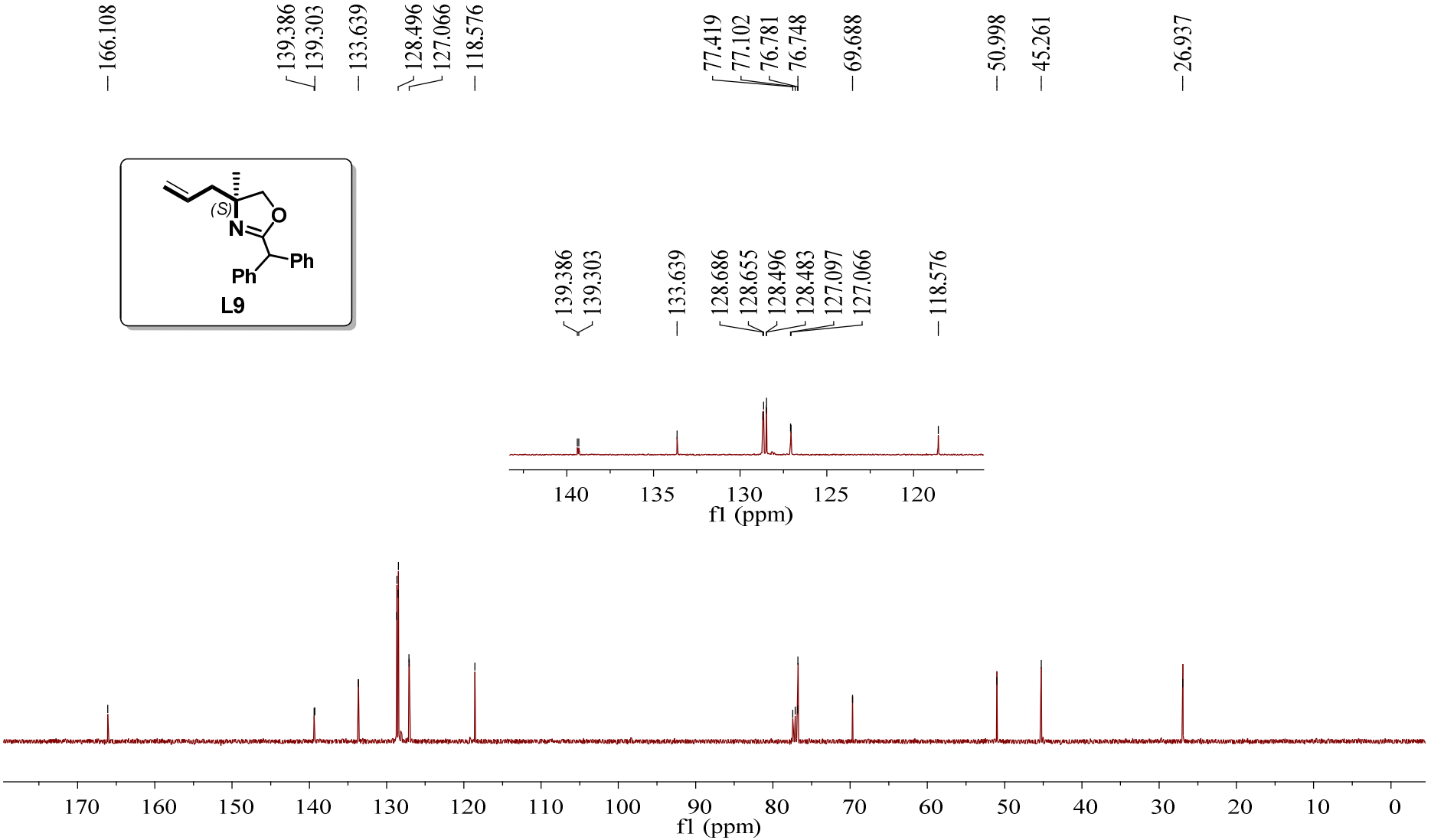
1.03
2.09
3.11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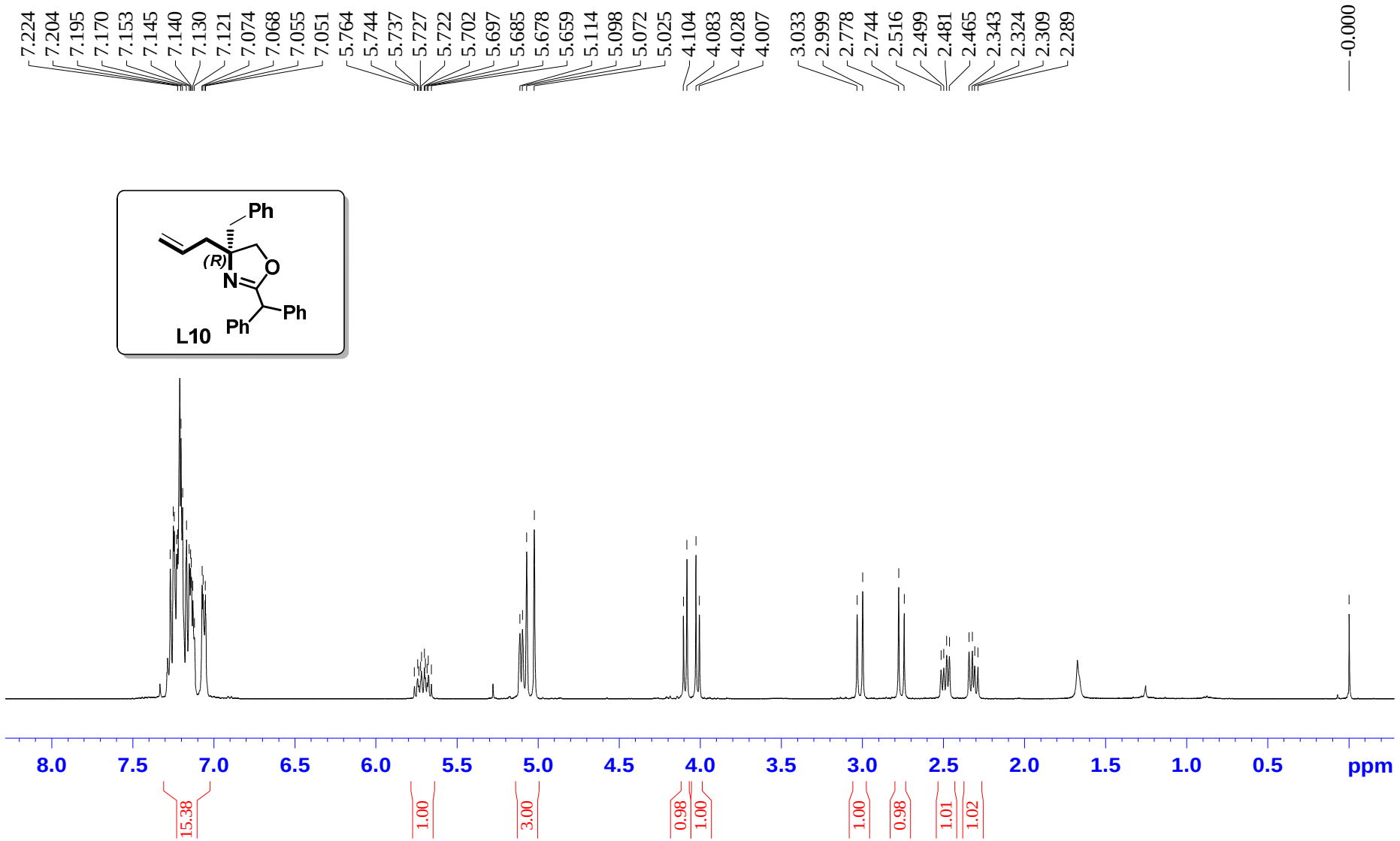
139.303
133.639
128.496
127.066
118.576
77.419
77.102
76.781
76.748
69.688
50.998
45.261
26.937
(S)
N
O
Ph
Ph
L9
139.386
139.303
133.639
128.686
128.655
128.496
128.483
127.097
127.066
118.576
140
135
130
125
120
f1 (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.224
7.204
7.195
7.170
7.153
7.145
7.140
7.130
7.121
7.074
7.068
7.055
7.051
5.764
5.744
5.737
5.727
5.722
5.702
5.697
5.685
5.678
5.659
5.114
5.098
5.072
5.025
4.104
4.083
4.028
4.007
3.033
2.999
2.778
2.744
2.516
2.499
2.481
2.465
2.343
2.324
2.309
2.289
-0.000
Ph
(R)
N
O
Ph
Ph
L10
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
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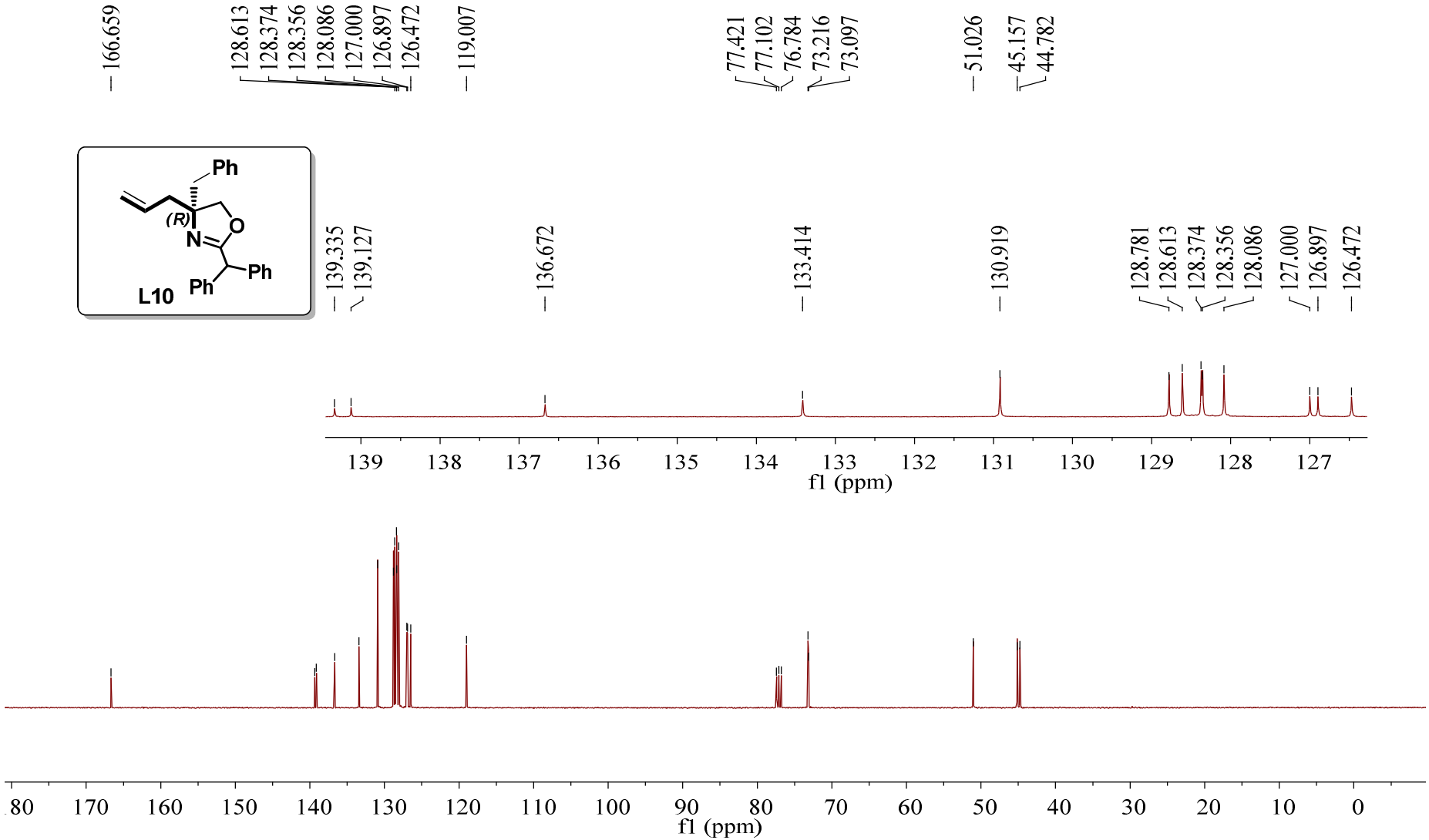
3.00
0.98
1.00
1.00
0.98
1.01
1.02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128.374
128.356
128.086
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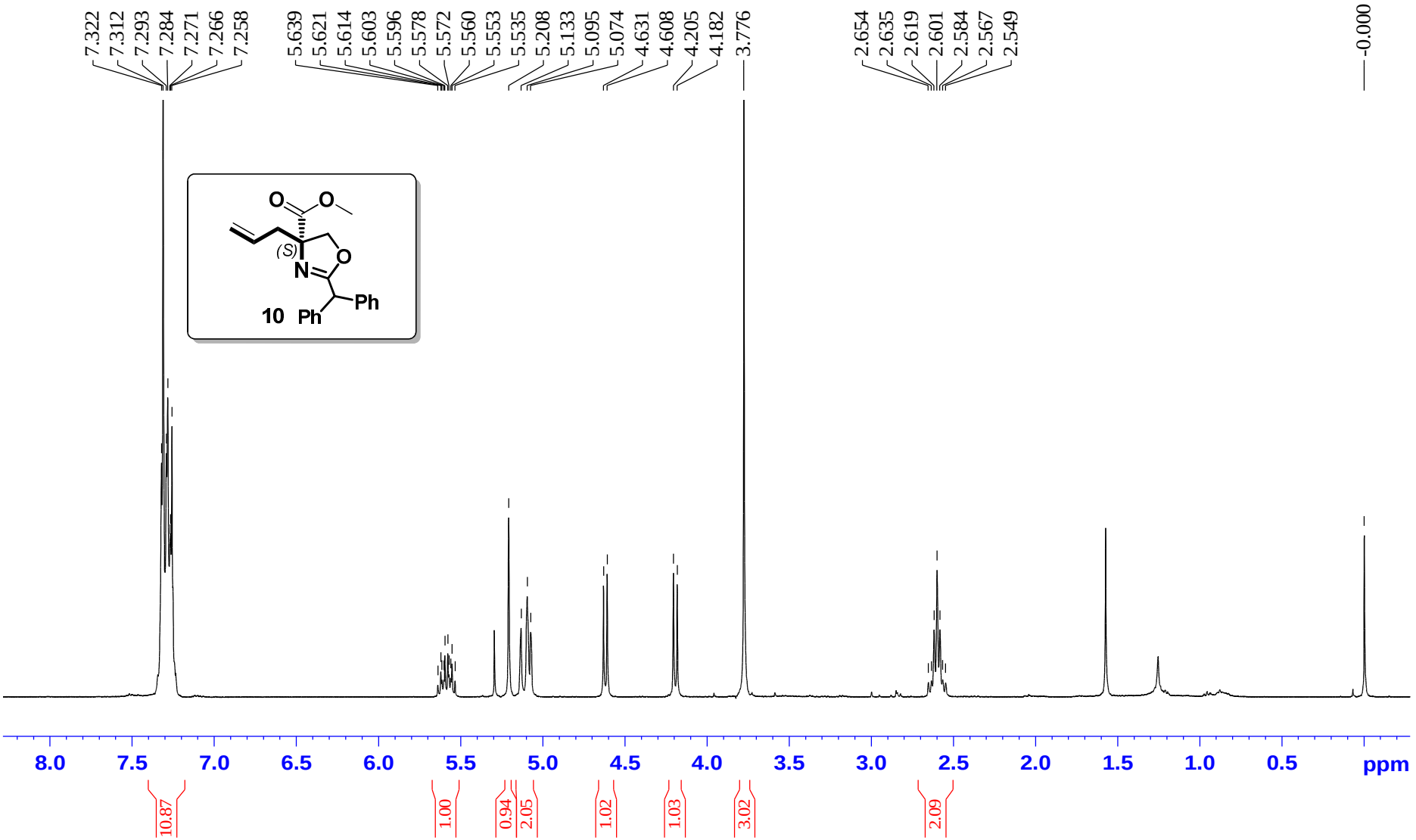
126.897
126.472
119.007
77.421
77.102
76.784
73.216
73.097
51.026
45.157
44.782
Ph
(R)
N
O
Ph
Ph
L10
139.335
139.127
136.672
133.414
130.919
128.781
128.613
128.374
128.356
128.086
127.000
126.897
126.472
139 138 137 136 135 134 133 132 131 130 129 128 127
f1 (ppm)
80 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.312
7.293
7.284
7.271
7.266
7.258
5.639
5.621
5.614
5.603
5.596
5.578
5.572
5.560
5.553
5.535
5.208
5.133
5.095
5.074
4.631
4.608
4.205
4.182
3.776
2.654
2.635
2.619
2.601
2.584
2.567
2.549
-0.000
(S)
10 Ph
Ph
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
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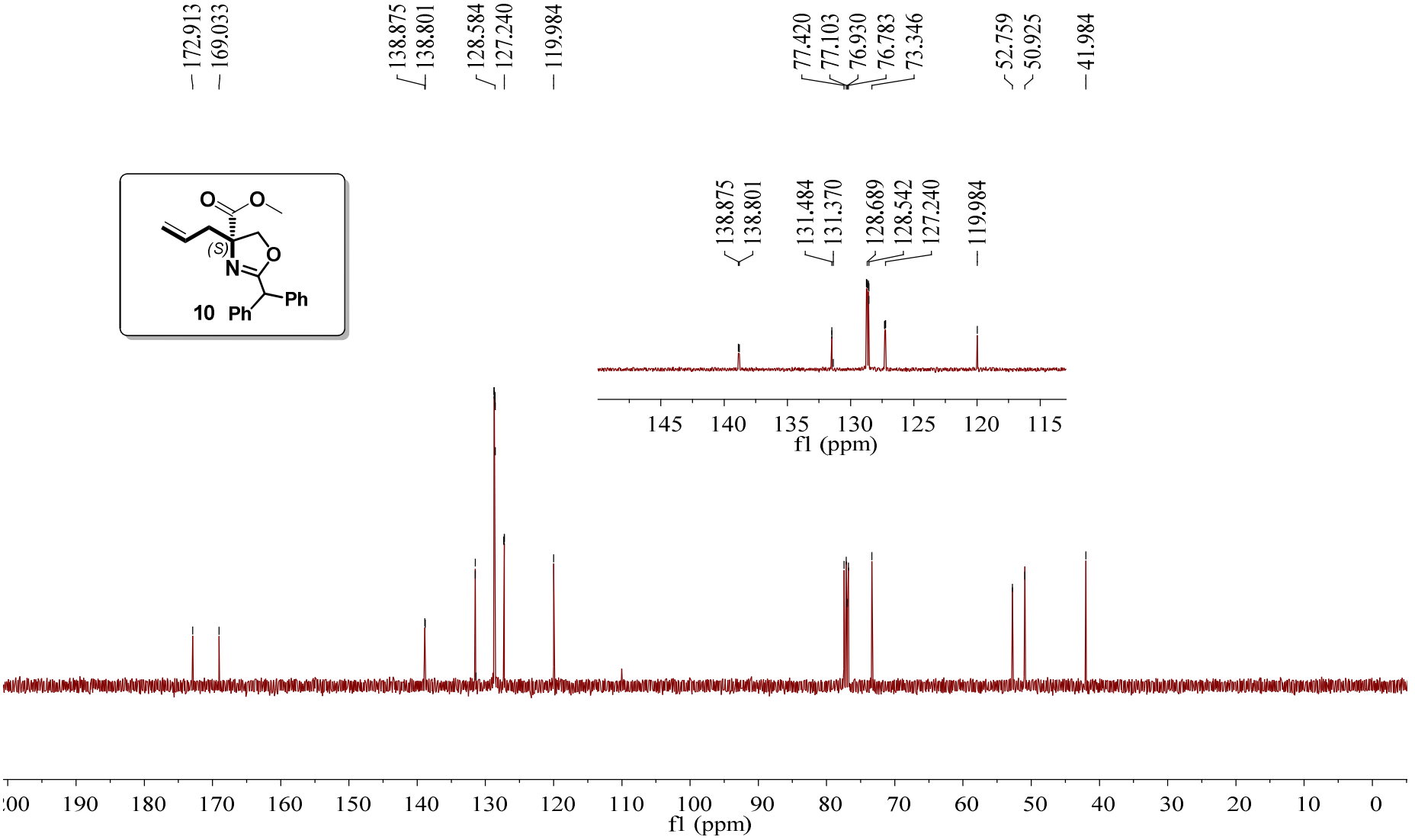
0.94
2.05
1.02
1.03
3.02
2.09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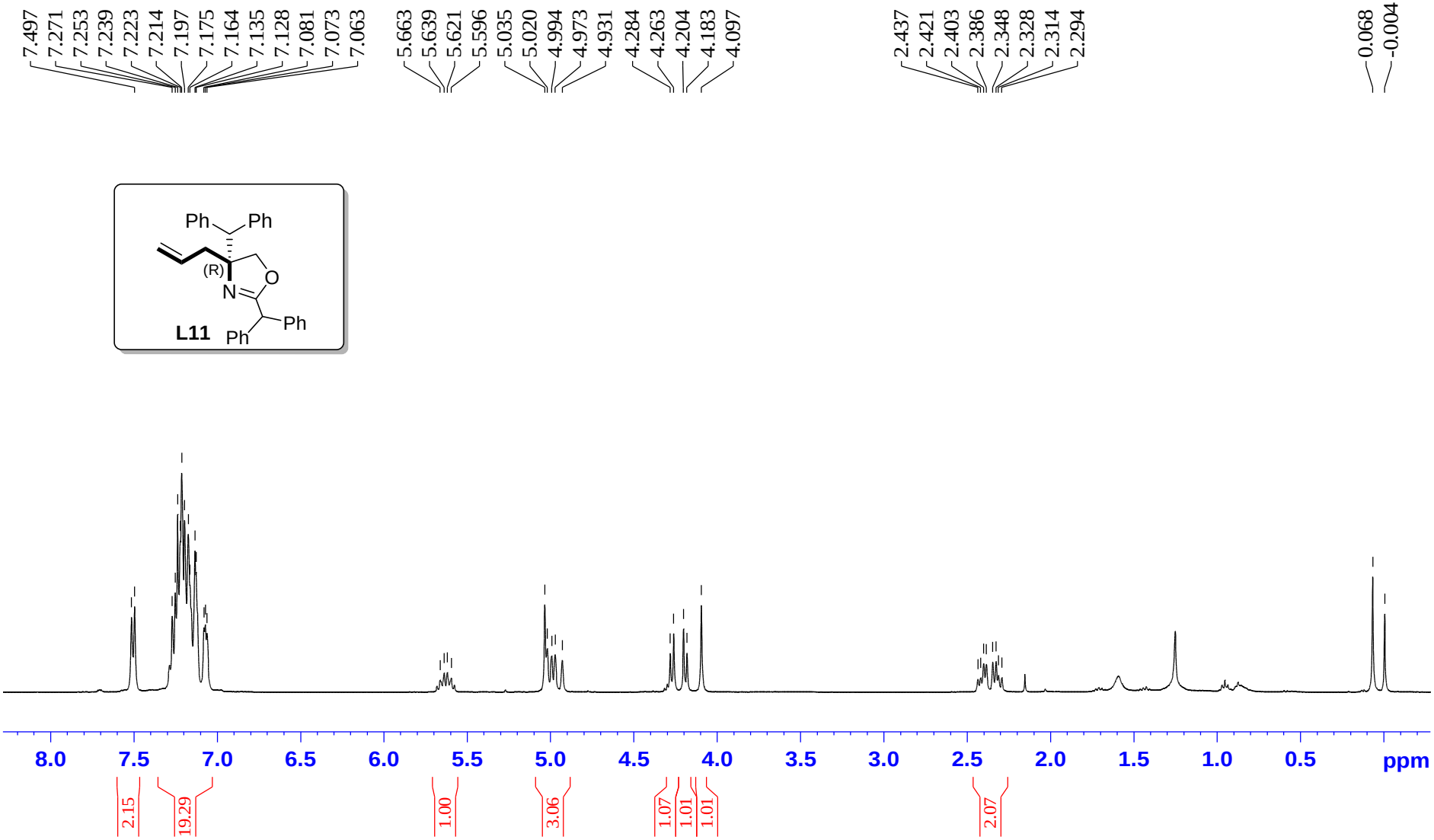
138.875
138.801
128.584
127.240
119.984
77.420
77.103
76.930
76.783
73.346
52.759
50.925
41.984
O
O
(S)
N
O
Ph
10 Ph
138.875
138.801
131.484
131.370
128.689
128.542
127.240
119.984
145 140 135 130 125 120 115
f1 (ppm)
:00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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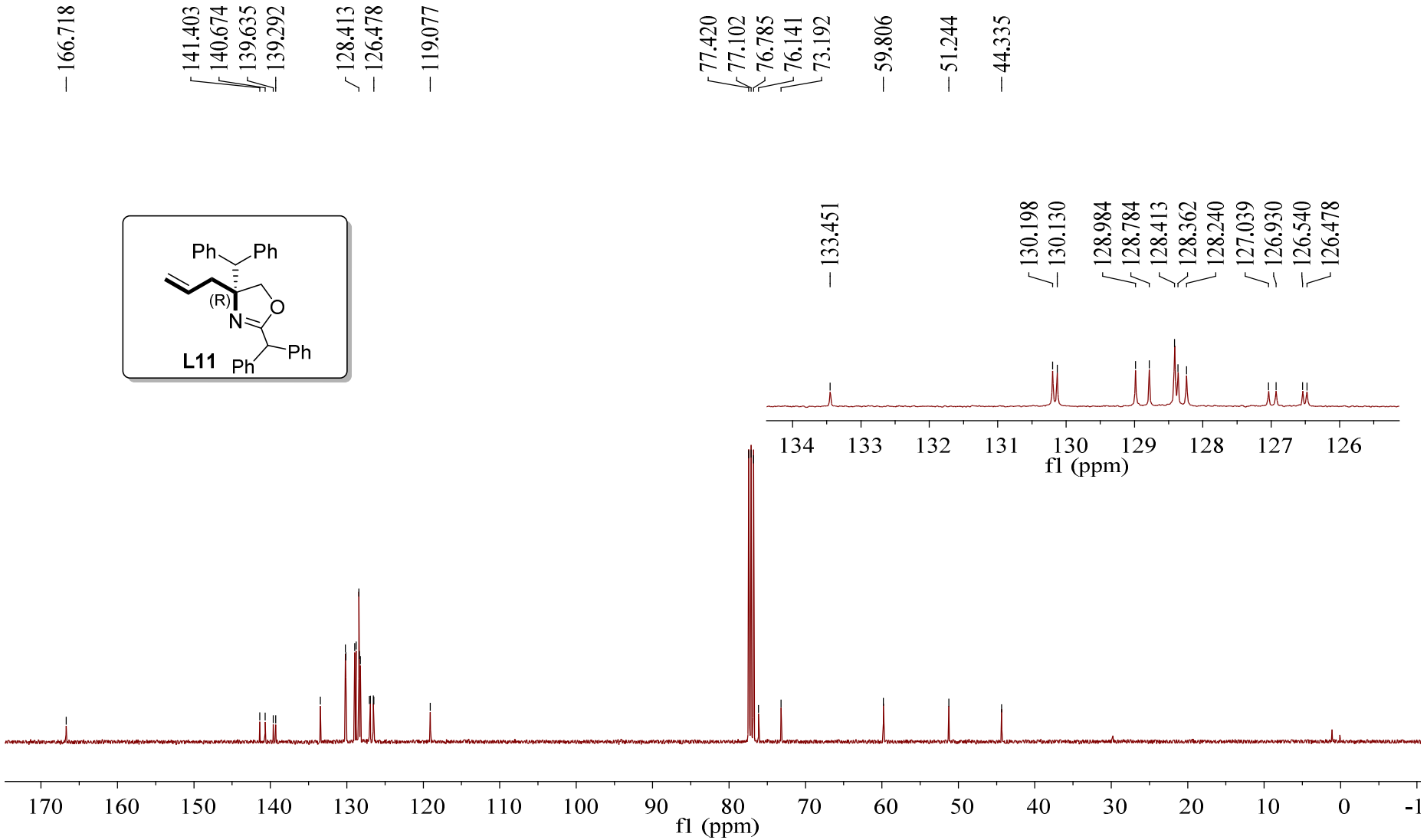
7.271
7.253
7.239
7.223
7.214
7.197
7.175
7.164
7.135
7.128
7.081
7.073
7.063
5.663
5.639
5.621
5.596
5.035
5.020
4.994
4.973
4.931
4.284
4.263
4.204
4.183
4.097
2.437
2.421
2.403
2.386
2.348
2.328
2.314
2.294
0.068
-0.004
Ph
Ph
(R)
N
O
Ph
Ph
L11
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.15
19.29
1.00
3.06
1.07
1.01
1.01
2.07


166.718
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126.478
119.077
77.420
77.102
76.785
76.141
73.192
59.806
51.244
44.335
133.451
130.198
130.130
128.984
128.784
128.413
128.362
128.240
127.039
126.930
126.540
126.478
Ph
Ph
(R)
N
O
Ph
Ph
L11
134
133
132
131
130
129
128
127
126
f1 (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)