Electronic Supplementary Material (ESI) for Chemical Science.


# Supporting Information

# Catalytic Asymmetric Direct Aldol Reaction of *α*-Alkyl Azlactones and Aliphatic Aldehydes

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## 1. General information.

$^{1}H$ and $^{13}C$ NMR spectra were recorded on a Varian instrument (400 MHz and 100 MHz, respectively) and internally referenced to tetramethylsilane signal or residual protio solvent signals. Data for $^{1}H$ NMR are recorded as follows: chemical shift (δ, ppm), multiplicity (s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet), integration, coupling constant (Hz). Data for $^{13}C$ NMR are reported in terms of chemical shift (δ, ppm). Infrared spectra were recorded on a Perkin Elmer FT-IR Spectrometer and are reported in frequency of absorption. Low resolution and high resolution mass spectra were recorded on either a Micromass 70-VSE-B instrument (EI, CI) or a Micromass Q-TOF instrument (ESI). Specific rotations were measured on a Jasco Digital Polarimeter.

High performance liquid chromatography (HPLC) analyses were performed on a Hewlett Packard 1100 Series instrument equipped with a quaternary pump, using Daicel Chiralpak AD, AS, AD-H and AS-H columns, Daicel Chiralcel OJ, OJ-H Columns. UV absorption was monitored at 254 nm.

## 2. Materials and methods

**A. Aliphatic aldehydes**:

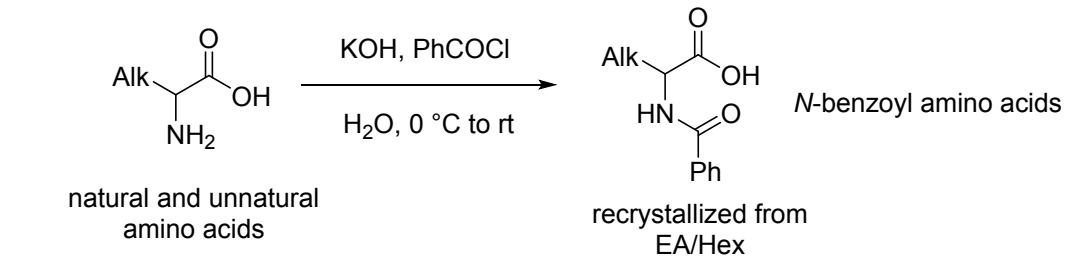
Aldehydes **7a-c** were purchased from Sigma-Aldrich and were distilled before use. Aldehyde **7d** was purchased from Alfa Aesar and was purified by flash column chromatography before use.

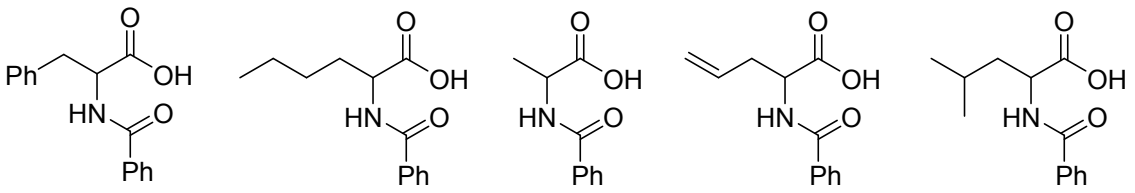
**B. 4-Alkyl-2-phenyl-5-(*4H*)-oxazolones (azlactones)**:

All natural and unnatural amino acids were purchased from Sigma Aldrich and used as received. Benzoyl chloride was purchased from Sigma Aldrich and used without out further

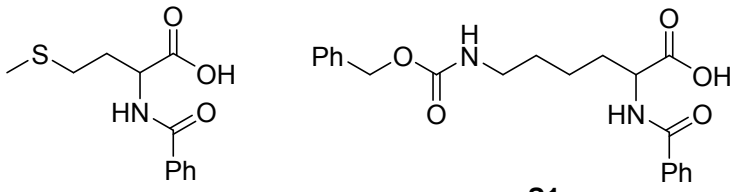
purification. EDCI was purchased from TCI and used without further purification. Thionyl chloride was purchased from Sigma Aldrich and distilled before use.

General procedure for the preparation of *N*-benzoyl amino acids:



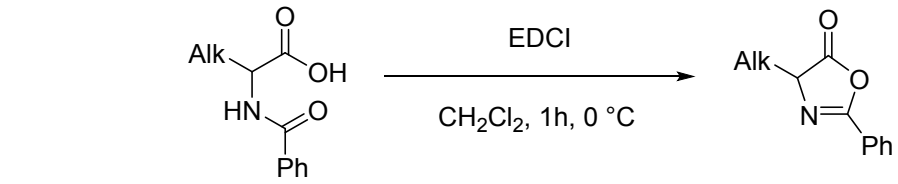

**S1a** **S1b** **S1c** **S1d** **S1e**

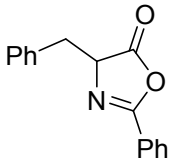



**S1f** **S1g**

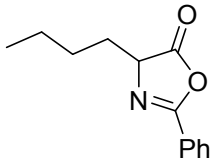
To the mixture of a DL-amino acid (10 mmol) and KOH (23 mmol, 2.3 eq) in water was added benzoyl chloride (11 mmol, 1.1 eq) at 0 °C dropwise. The reaction was allowed to warm to room temperature and was stirred for one day. The reaction mixture was acidified with 6N HCl carefully to adjust the pH to 1 and was then extracted with EA. The combined organic phase was evaporated and the crude solid product was recrystallized from EA/Hex to give the corresponding *N*-benzoyl amino acid. All spectroscopic data were in accordance with reported literature data[1-5].

General procedure for the preparation of azlactones:




**6a**

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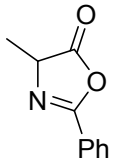


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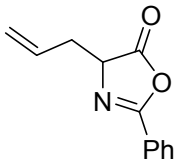
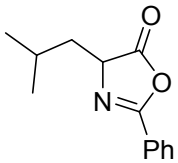
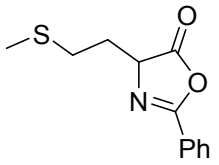


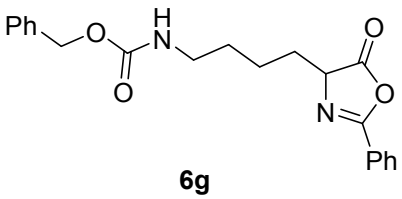

**6d**




**6e**



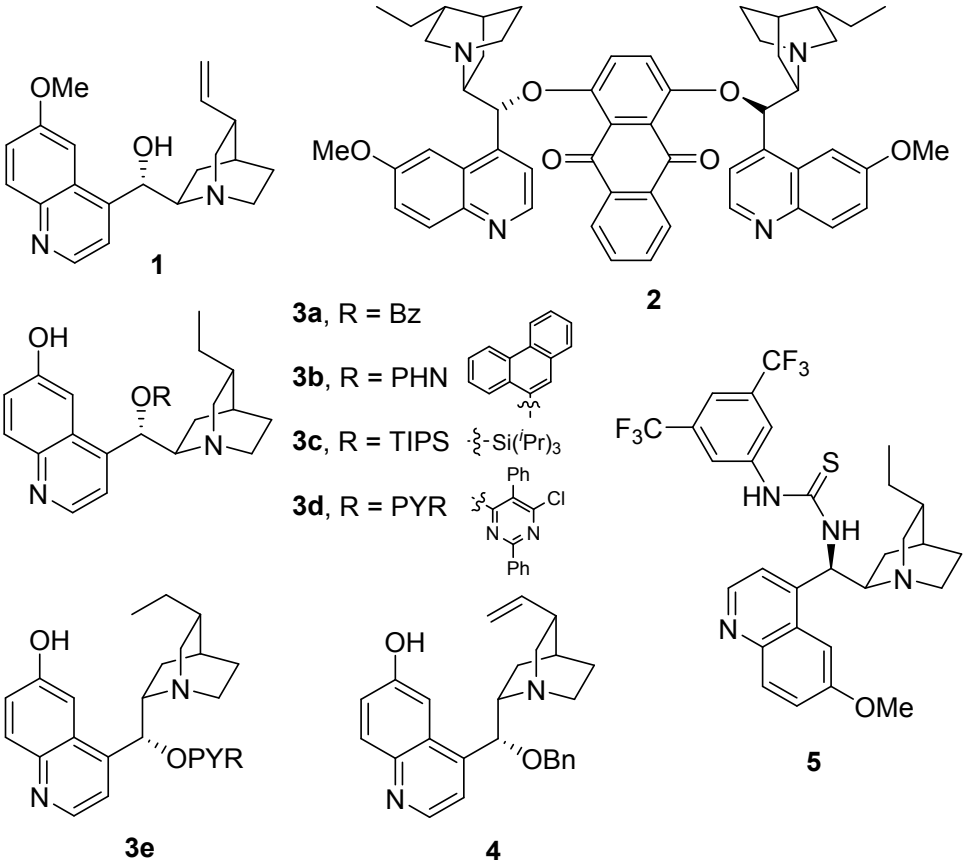

**6f**



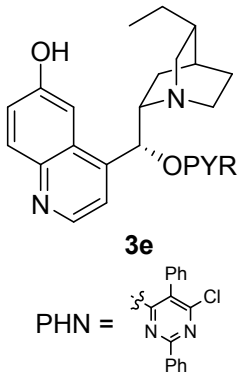

**6g**

To the stirred suspension of an *N*-benzoyl amino acid (0.5 mmol) in $CH_2Cl_2$ (5 mL, 0.1 M) cooled in an ice bath was added EDCI in one portion (0.5 mmol, 1 eq). After 1h, the reaction was diluted with 20 mL of $Et_2O$, washed with water (15 mL x 2) and brine (10 mL). The combined organic phase was dried over $MgSO_4$, filtered and evaporated to give the corresponding azlactone in reasonably pure form without further purification. The spectroscopic data of all azlactones (**6a-h**) were in accordance with reported literature data[6-8].

**C. Catalysts**




Catalysts **1** and **2** were purchased from Sigma Aldrich and used without further purification. All the other catalysts were synthesized according to literature procedures[9-14].




Catalyst **3e** was obtained as a white solid. $[\alpha]_D^{20}$ = +312.3 (c = 1.22, $CH_2Cl_2$). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 9.45 (br, 1H), 8.74 – 8.49 (m, 2H), 7.98 (d, *J* = 9.0 Hz, 1H), 7.88 (d, *J* = 6.4 Hz, 2H), 7.59 (t, *J* = 7.3 Hz, 2H), 7.51 (t, *J* = 9.2 Hz, 3H), 7.26 (d, *J* = 9.5 Hz, 1H), 7.23 – 7.14 (m, 2H), 7.04 – 6.92 (m, 3H), 3.18 – 2.98 (m, 3H), 2.74 – 2.56 (m, 1H), 2.27 (d, *J* = 11.8 Hz, 1H), 1.81 – 1.67 (m, 1H), 1.59 (s, 1H), 1.53 – 1.30 (m, 2H), 1.20 – 1.01 (m, 3H), 0.89 – 0.76 (m, 1H), 0.69 (t, *J* = 7.3 Hz, 3H). **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ = 166.13, 162.84, 159.82, 157.22, 146.46, 144.08, 143.72, 135.26, 132.31, 131.27, 131.08, 130.02, 128.77, 128.74, 128.43, 128.25, 127.41, 123.18, 118.69, 117.27, 107.71, 77.48, 77.16, 76.84, 76.53, 58.60, 58.37, 43.50, 36.89, 27.35, 26.73, 25.01, 20.54, 11.94. **IR**

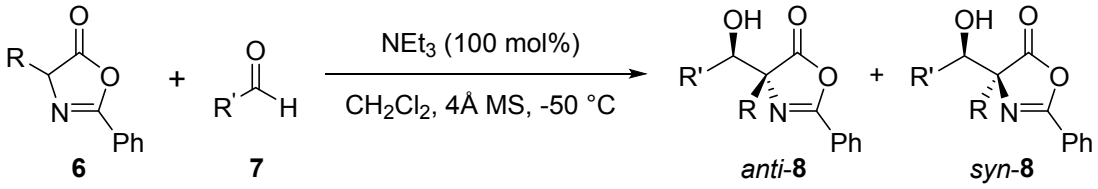
(neat): v = 3054, 2957, 2953, 2877, 2360, 2338, 1619, 1571, 1515, 1407, 1377, 1264,1210, 1030, 999, 846, 809, 735, 700 $cm^{-1}$. **HRMS** (ESI/$[M+H]^+$) = Calcd. for $C_{26}H_{29}N_2O_3$ m/z = 577.2370, found m/z = 577.2368.

**D. The other materials**:

Molecular Sieves (4Å, purchased from Alfa Aesar) were dried in a round-bottom flask under vacuum with a Bunsen burner. Triethylamine, dihydropyran (DHP) and pyridinium *p*-toluenesulfonate (PPTS) were purchased from Alfa Aesar and used without further purification. HCl in MeOH (~1.25 M) was purchased from Sigma Aldrich and was used without further purification. Thionyl chloride was purchased from Sigma Aldrich and distilled before use. 4-(Dimethylamino)pyridine (DMAP) was purchased from Sigma Aldrich and used as received.

## 3. Aldol reaction of azlactones and aliphatic aldehydes

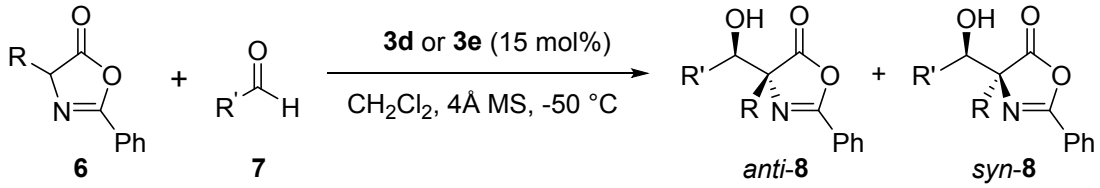
General procedure for racemic reactions (Table 1 & 2):



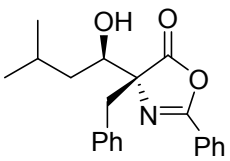

Freshly prepared azlactone **6** (0.5 mmol), freshly distilled or purified aldehyde **7** (1 mmol) and freshly dried 4Å Molecular Sieves (50 mg) were added to a vial (22 mL), followed by $CH_2Cl_2$ (5 mL). The vial was gently shaken and then placed in a -50 °C freezer. After 15 minutes, triethylamine (70 µL) was added by a micro syringe. The vial was gently shaken again and was placed back to the -50 °C freezer. The reaction progress was monitored by TLC. Usually it takes 3 days to complete. The reaction mixture was passed through a short pad of silica gel with washing by $Et_2O$. The filtrate was concentrated to give a crude product which was suitable

for HPLC analysis and further transformations. All crude products were stored in a -50 °C freezer due to instability.

General procedure for asymmetric reactions (Table 1 & 2):




Freshly prepared azlactone **6** (0.1mmol), catalyst **3d** or **3e** (8.7 mg, 0.015 mmol) and freshly dried 4Å Molecular Sieves (10 mg) were added to a vial (2 mL), followed by $CH_2Cl_2$ (1 mL). The vial was gently shaken and then placed in a -50 °C freezer. After 15 minutes, freshly distilled or purified aldehyde **7** (0.15 mmol) was added by a micro syringe. The vial was gently shaken again and was placed back to the -50 °C freezer. The reaction progress was monitored by TLC (All reaction times in Table 1 and 2 were not optimized). The reaction mixture was passed through a short pad of silica gel with washing by $Et_2O$. The filtrate was concentrated to give the crude product, which was subject to vacuum to remove solvent and aldehyde residues to give product **8** in reasonably pure form. Product **8ad** was an exception, which was purified by a short deactivated silica gel column (Hex to 1/20 = EA/Hex). All products were stored in a -50 °C freezer due to instability.

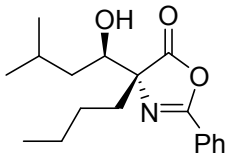



**8aa**

92% yield, 94% ee, *anti*/*syn* = 97.5/2.5, 88 h

Product **8aa** was obtained as a colorless oil (31.2 mg) from a reaction catalyzed by **3d**. Enantiomeric excess and diastereometric ratio were determined by HPLC analysis: Daicel Chiralcel AS-H, Hexanes/IPA = 95/5, 1.0 ml/min, λ = 254 nm, t (*anti*, minor) = 13.91 min, t (*anti*, major) = 16.59 min, t (*syn*, major) = 25.53, t (*syn*, minor) = 39.98. $[\alpha]_D^{20}$ = +129.3 (c = 0.31, $CH_2Cl_2$). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 7.78 (d, *J* = 7.7 Hz, 2H), 7.54 – 7.45 (m, 1H), 7.38 (t, *J* = 7.7 Hz, 2H), 7.19 – 7.04 (m, 5H), 4.07 (dd, *J* = 10.3, 5.5 Hz, 1H), 3.38 (d, *J* = 13.4 Hz, 1H), 3.25 (d, *J* = 13.4 Hz, 1H), 2.66 (d, *J* = 6.6 Hz, 1H), 2.00 – 1.84 (m, 1H), 1.73 –

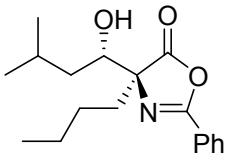
1.62 (m, 1H), 1.36 – 1.26 (m, 1H), 0.96 (dd, *J* = 11.3, 6.6 Hz, 6H). **$^{13}$C NMR** (100 MHz, CDCl3): δ = 178.00, 161.37, 134.21, 132.84, 130.26, 128.79, 128.26, 127.97, 127.29, 125.36, 78.93, 73.12, 40.15, 40.12, 24.63, 24.00, 21.26. **IR** (neat): v = 3242, 2949, 2925, 2601, 2496, 2360, 1818, 1724, 1649, 1602, 1581, 1496, 1450, 1367, 1385, 1322, 1337, 1292, 1238, 1212, 1171, 1141, 1086, 1058, 977, 924, 902, 883, 858, 808, 776, 743, 698, 643, 620, 611 cm$^{-1}$. **HRMS** (ESI/[M+H]$^+$) = Calcd. for $C_{21}H_{23}NO_3$ m/z = 338.1756, found m/z = 338.1752.




**8ba**

98% yield, 95% ee,
*anti*/*syn* = 98/2, 102 h

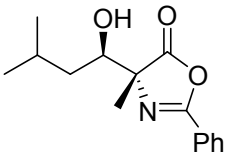
Product **8ba** was obtained as a colorless oil (29.6 mg) from a reaction catalyzed by **3d**. Enantiomeric excess and diastereometric ratio were determined by HPLC analysis: Daicel Chiralpak AD-H connected with AD, Hexanes/IPA = 95/5, 0.8 ml/min, λ = 254 nm, t (*anti*, minor) = 17.02 min, t (*syn*, major) = 18.38 min, t (*anti*, major) = 19.24, t (*syn*, minor) = 22.86. $[\alpha]_D^{20}$ = +13.1 (c = 0.36, $CH_2Cl_2$). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 7.96 (d, J = 8.0 Hz, 2H), 7.57 (t, J = 7.3 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 3.95 (d, J = 10.8 Hz, 1H), 2.67 (s, 1H), 2.08 – 1.79 (m, 3H), 1.72 – 1.58 (m, 1H), 1.37 – 1.04 (m, 5H), 0.94 (dd, J = 12.4, 6.6 Hz, 6H), 0.85 (t, J = 7.3 Hz, 3H). **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ = 178.71, 161.56, 132.97, 128.94, 128.14, 125.55, 77.95, 73.06, 39.50, 33.74, 25.99, 24.56, 24.00, 22.74, 21.21, 13.95. **IR** (neat): v = 3255, 2959, 2944, 2863, 1820, 1642, 1464, 1451, 1365, 1342, 1323, 1289, 1261, 1184, 1153, 1077, 1067, 986, 963, 889, 854, 840, 797, 778, 734, 697, 684, 614 cm$^{-1}$. **HRMS** (ESI/[M+H]$^+$) = Calcd. for $C_{18}H_{26}NO_3$ m/z = 304.1913, found m/z = 304.1909.




*ent*-**8ba**

97% yield, -90% ee,
*anti*/*syn* = 97.5/2.5, 142 h

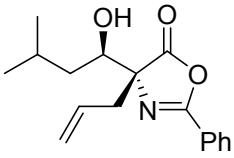
Product *ent*-**8ba** was obtained as a colorless oil (29.5 mg) from a reaction catalyzed by **3e**. Enantiomeric excess and diastereometric ratio were determined by HPLC analysis: Daicel Chiralpak AD-H connected with AD, Hexanes/IPA = 95/5, 0.8 ml/min, λ = 254 nm, t (*anti*, major) = 17.57 min, t (*syn*, minor) = 18.85 min, t (*anti*, minor) = 19.66, t (*syn*, major) = 23.30. $[\alpha]_D^{20}$ = +10.7 (c = 0.37, $CH_2Cl_2$). All the other data were the same as product **8ba**.



**8ca**

92% yield, 95% ee, *anti*/*syn* = 98/2, 103 h

Product **8ca** was obtained as a colorless oil (24 mg) from a reaction catalyzed by **3d**. Enantiomeric excess and diastereometric ratio were determined by HPLC analysis: Daicel Chiralpak AD-H, Hexanes/IPA = 99/1, 1.0 ml/min, λ = 254 nm, t (*anti*, minor) = 27.84 min, t (*anti*, major) = 31.96 min, t (*anti*, major) = 37.02, t (*syn*, minor) = 38.29. $[\alpha]_D^{20}$ = +10.1 (c = 0.33, $CH_2Cl_2$). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 8.00 (d, J = 7.4 Hz, 1H), 7.59 (t, J = 7.4 Hz, 1H), 7.49 (t, J = 7.6 Hz, 2H), 3.90 (dd, J = 10.5, 4.0 Hz, 1H), 2.20 (s, 1H), 1.95 – 1.78 (m, 1H), 1.69 – 1.58 (m, 1H), 1.55 (s, 3H), 1.29 – 1.22 (m, 1H), 0.94 (dd, J = 12.4, 6.6 Hz, 6H). **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ = 179.00, 161.47, 133.00, 128.93, 128.11, 125.61, 73.62, 73.49, 39.35, 24.59, 23.96, 21.20, 20.48. **IR** (neat): v = 3290, 2955, 2870, 2359, 2337, 1819, 1739, 1649, 1602, 1581, 1495, 1451, 1368, 1323, 1292, 1216, 1173, 1109, 1094, 1069, 1031, 1000, 957, 929, 885, 849, 780, 738, 696, 613 $cm^{-1}$. **HRMS** (ESI/$[M+H]^+$) = Calcd. for $C_{15}H_{20}NO_3$ m/z = 262.1443, found m/z = 262.1435.

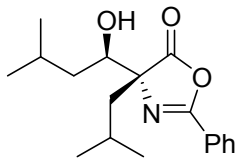


**8da**

95% yield, 94% ee, *anti*/*syn* = 98/2, 109 h

Product **8ca** was obtained as a light yellow oil (27.4 mg) from a reaction catalyzed by **3d**. Enantiomeric excess and diastereometric ratio were determined by HPLC analysis: Daicel Chiralpak AD-H, Hexanes/IPA = 96/4, 1.0 ml/min, λ = 254 nm, t (*anti*, minor) = 16.41 min, t (*syn*, major) = 18.02 min, t (*anti*, major) = 19.49, t (*syn*, minor) = 26.28. $[\alpha]_D^{20}$ = +61 (c = 0.72, $CH_2Cl_2$). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 8.00 (d, J = 7.8 Hz, 2H), 7.59 (t, J = 7.4 Hz, 1H), 7.49 (t, J = 7.6 Hz, 2H), 5.62 (td, J = 17.0, 7.4 Hz, 1H), 5.19 (d, J = 17.0 Hz, 1H), 5.10 (d, J = 10.1 Hz, 1H), 3.97 (d, J = 10.3 Hz, 1H), 2.83 (dd, J = 13.6, 6.7 Hz, 1H), 2.67 (dd, J = 13.7, 8.0 Hz, 1H), 2.16 (d, J = 21.5 Hz, 1H), 1.96 – 1.79 (m, 1H), 1.60 (ddd, J = 14.6, 11.1, 3.8 Hz, 1H), 1.25 – 1.15 (m, 1H), 0.93 (dd, J = 11.1, 6.6 Hz, 6H). **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ = 177.98, 161.62, 133.03, 130.58, 128.93, 128.17, 125.50, 120.80, 77.91, 72.69, 39.69, 38.24, 24.55, 23.99, 21.22. **IR** (neat): v = 3318, 2955, 2910, 2870, 2359, 1816, 1739, 1648, 1580, 1495, 1647, 1451, 1368, 1322, 1289, 1217, 1178, 1042, 1026, 970, 925, 900, 850, 779, 700,

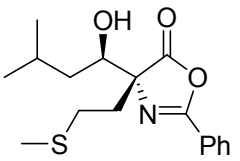
689, 668 $cm^{-1}$. **HRMS** (ESI/$[M+H]^+$) = Calcd. for $C_{17}H_{22}NO_3$ m/z = 288.1600, found m/z = 288.1598.




**8ea**

89% yield, 93% ee, *anti*/*syn* = 97.5/2.5, 99 h

Product **8ea** was obtained as a colorless yellow oil (27 mg) from a reaction catalyzed by **3d**. Enantiomeric excess and diastereometric ratio were determined by HPLC analysis: Daicel Chiralpak AD-H, Hexanes/IPA = 96/4, 1.0 ml/min, λ = 254 nm, t (*anti*, minor) = 12.65 min, t (*anti*, major) = 15.16 min, t (*syn*, major) = 17.11, t (*syn*, minor) = 25.16. $[\alpha]_D^{20}$ = +52.7 (c = 0.34, $CH_2Cl_2$). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 8.03 – 7.95 (m, 2H), 8.04 – 7.97 (m, 2H), 7.59 (t, J = 7.4 Hz, 1H), 7.49 (t, J = 7.5 Hz, 2H), 3.88 (dd, J = 10.3, 5.5 Hz, 1H), 2.37 – 2.14 (m, 1H), 2.05 (dd, J = 14.0, 5.1 Hz, 1H), 1.92 – 1.77 (m, 2H), 1.70 – 1.52 (m, 2H), 1.30 – 1.19 (m, 1H), 0.95 (d, J = 6.7 Hz, 3H), 0.91 (dd, J = 6.5, 3.9 Hz, 6H), 0.83 (d, J = 6.6 Hz, 3H). **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ = 179.21, 161.34, 132.95, 128.97, 128.11, 125.62, 77.48, 77.16, 76.85, 74.01, 42.61, 38.89, 24.99, 24.59, 24.33, 24.02, 22.96, 21.18. **IR** (neat): v = 3289, 2955, 2927, 2870, 1817, 1649, 1580, 1495, 1467, 1450, 1387, 1368, 1323, 1289, 1166, 1078, 1043, 1024, 970, 885, 855, 779, 698, 654, 623, 613, 602 $cm^{-1}$. **HRMS** (ESI/$[M+H]^+$) = Calcd. for $C_{18}H_{26}NO_3$ m/z = 304.1913, found m/z = 304.1913.

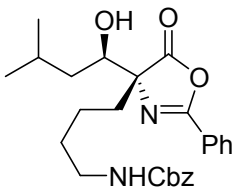



**8fa**

96% yield, 92% ee, *anti*/*syn* = 97/3, 120 h

Product **8fa** was obtained as a light yellow oil (31 mg) from a reaction catalyzed by **3d**. Enantiomeric excess and diastereometric ratio were determined by HPLC analysis: Daicel Chiralpak AD-H, Hexanes/IPA = 96.5/3.5, 1.0 ml/min, λ = 254 nm, t (*anti*, minor) = 32.10 min, t (*syn*, major) = 34.33 min, t (*syn*, minor) = 44.90, t (*anti*, major) = 47.92. $[\alpha]_D^{20}$ = +29.8 (c = 0.4, $CH_2Cl_2$). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 7.98 (d, J = 7.6 Hz, 2H), 7.58 (t, J = 7.4 Hz, 1H), 7.48 (t, J = 7.7 Hz, 2H), 3.92 (dd, J = 10.6, 4.2 Hz, 1H), 2.56 (d, J = 6.0 Hz, 1H), 2.51 – 2.23 (m, 4H), 2.04 (s, 3H), 1.94 – 1.76 (m, 1H), 1.68 – 1.58 (m, 1H), 1.25 – 1.15 (m, 1H), 0.93 (dd, J = 14.1, 6.6 Hz, 6H). **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ = 178.59, 162.23, 133.13, 128.96, 128.18, 125.48, 76.63, 73.30, 39.25, 32.71, 28.91, 24.53, 23.96, 21.19, 15.21. **IR** (neat): v =

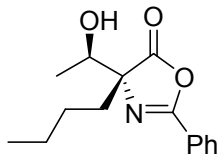
3460, 2956, 2911, 2869, 2360, 2337, 1812, 1739, 1649, 1580, 1495, 1451, 1367, 1321, 1292, 1216, 1139, 1070, 1066, 1009, 1001, 963, 908, 881, 850, 780, 730, 695, 647, 609 $cm^{-1}$. **HRMS** (ESI/$[M+H]^+$) = Calcd. for $C_{17}H_{24}NO_3S$ m/z = 322.1477, found m/z = 322.1478.




**8ga**

95% yield, 94% ee,
*anti*/*syn* = 97.5/2.5, 112 h

Product **8ga** was obtained as a colorless oil (47 mg) from a reaction catalyzed by **3d**. Enantiomeric excess and diastereometric ratio were determined by HPLC analysis: Daicel Chiralpak AD-H, Hexanes/IPA = 87/13, 1.0 ml/min, λ = 254 nm, t (*anti*, minor) = 26.73 min, t (*anti*, major) = 32.71 min, t (*syn*, minor) = 36.01, t (*syn*, major) = 37.89. $[\alpha]_D^{20}$ = +12.1 (c = 0.56, $CH_2Cl_2$). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 7.98 (d, J = 7.4 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.47 (t, J = 7.6 Hz, 2H), 7.38 – 7.27 (m, 5H), 5.05 (s, 2H), 4.79 (s, 1H), 3.92 (d, J = 10.3 Hz, 1H), 3.26 – 2.97 (m, 2H), 2.63 (s, 1H), 2.11 – 1.75 (m, 3H), 1.67 – 1.55 (m, 1H), 1.55 – 1.39 (m, 2H), 1.24 – 1.04 (m, 3H), 0.91 (dd, J = 12.2, 6.6 Hz, 6H). **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ = 178.57, 161.60, 156.41, 136.57, 133.06, 128.95, 128.59, 128.18, 128.14, 125.43, 77.75, 72.94, 66.71, 40.63, 39.59, 33.40, 29.81, 24.50, 23.94, 21.17, 21.05. **IR** (neat): v = 3334, 2954, 2910, 2867, 1813, 1697, 1650, 1580, 1526, 1496, 1452, 1368, 1321, 1288, 1254, 1178, 1134, 1044, 1022, 972, 882, 850, 777, 736, 694, 606 $cm^{-1}$. **HRMS** (ESI/$[M+H]^+$) = Calcd. for $C_{26}H_{33}N_2O_5$ m/z = 453.2389, found m/z = 453.2390.

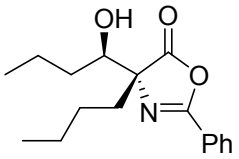



**8bb**

93% yield, 88% ee,
*anti*/*syn* = 90.5/9.5, 114 h

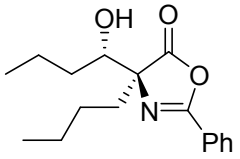
Product **8bb** was obtained as a colorless oil (24.2 mg) from a reaction catalyzed by **3d**. Enantiomeric excess and diastereometric ratio were determined by HPLC analysis: Daicel Chiralcel OJ-H connected with OJ, Hexanes/IPA = 96/4, 0.8 ml/min, λ = 254 nm, t (*anti*, minor) = 40.78 min, t (*anti*, major) = 59.11 min, t (*syn*, minor) = 63.58, t (*syn*, minor) = 73.38. $[\alpha]_D^{20}$ = +1.2 (c = 1.2, $CH_2Cl_2$). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 8.01 (d, J = 7.3 Hz, 2H), 7.59 (t, J = 7.3 Hz, 1H), 7.49 (t, J = 7.6 Hz, 2H), 4.16 – 3.97 (m, 1H), 2.37 (s, 1H), 2.07 – 1.85 (m, 2H), 1.41 – 1.02 (m, 7H), 0.86 (t, J = 7.2 Hz, 3H). **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ = 178.54, 161.73, 133.03, 128.95, 128.17, 125.56, 77.89, 71.18, 33.56, 26.08,

22.73, 16.98, 13.94. **IR** (neat): v = 3392, 2957, 2930, 2872, 2360, 2321, 1813, 1739, 1650, 1602, 1580, 1520, 1494, 1451, 1378, 1321, 1291, 1230, 1198, 1164, 1074, 1037, 1022, 954, 913, 881, 780, 694, 671, 605 $cm^{-1}$. **HRMS** (ESI/$[M+H]^+$) = Calcd. for $C_{15}H_{20}NO_3$ m/z = 262.1443, found m/z = 262.1443.



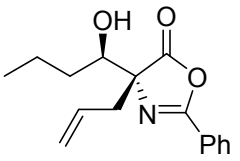

**8bc**
95% yield, 94% ee,
*anti*/*syn* = 97.5/2.5, 112 h

Product **8bc** was obtained as a colorless oil (27.5 mg) from a reaction catalyzed by **3d**. Enantiomeric excess and diastereometric ratio were determined by HPLC analysis: Daicel Chiralpak AD-H connected with AD, Hexanes/IPA = 95/5, 0.8 ml/min, λ = 254 nm, t (*anti*, minor) = 62.88 min, t (*anti*, major) = 83.69 min, t (*syn*, major) = 87.09, t (*syn*, minor) = 90.83. $[\alpha]_D^{20}$ = +17.1 (c = 0.35, $CH_2Cl_2$). $^1H$ NMR (400 MHz, $CDCl_3$): δ = 8.01 (d, *J* = 7.5 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 2H), 3.93 – 3.75 (m, 1H), 2.31 – 2.19 (m, 1H), 2.05 (td, *J* = 12.9, 4.8 Hz, 1H), 2.00 – 1.90 (m, 1H), 1.72 – 1.02 (m, 8H), 0.93 (t, *J* = 7.2 Hz, 3H), 0.86 (t, *J* = 7.3 Hz, 3H). **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ = 178.81, 161.52, 132.97, 128.93, 128.14, 125.58, 77.93, 77.48, 77.16, 76.84, 74.72, 33.68, 32.90, 25.98, 22.74, 19.49, 13.94, 13.92. **IR** (neat): v = 3444, 2958, 2930, 2872, 2337, 1815, 1739, 1649, 1602, 1581, 1495, 1451, 1377, 1322, 1290, 1229, 1217, 1157, 1076, 1041, 1023, 966, 880, 855, 804, 778, 732, 696, 612 $cm^{-1}$. **HRMS** (ESI/$[M+H]^+$) = Calcd. for $C_{17}H_{24}NO_3$ m/z = 290.1756, found m/z = 290.1760.




*ent*-**8bc**
98% yield, -92% ee,
*anti*/*syn* = 97/3,132 h

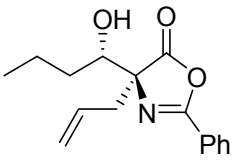
Product *ent*-**8bc** was obtained as a colorless oil (28.3 mg) from a reaction catalyzed by **3d**. Enantiomeric excess and diastereometric ratio were determined by HPLC analysis: Daicel Chiralpak AD-H connected with AD, Hexanes/IPA = 95/5, 0.8 ml/min, λ = 254 nm, t (*anti*, major) = 68.47 min, t (*anti*, minor) = 94.59 min, t (*syn*, major) = 97.69, t (*syn*, minor) = 101.46. All the other data were the same as product **8bc**.



**8dc**

97% yield, 94% ee, *anti*/*syn* = 97.5/2.5, 95 h

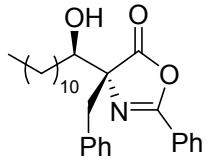
Product **8dc** was obtained as a yellow oil (26.5 mg) from a reaction catalyzed by **3d**. Enantiomeric excess and diastereometric ratio were determined by HPLC analysis: Daicel Chiralcel OJ-H, Hexanes/IPA = 97/3, 1.0 ml/min, λ = 254 nm, t (*anti*, minor) = 18.09 min, t (*anti*, major) = 21.01 min, t (*syn*, minor) = 26.48, t (*syn*, minor) = 34.97. $[\alpha]_D^{20}$ = +26.9 (c = 0.33, $CH_2Cl_2$). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 8.02 – 7.95 (m, 2H), 7.59 (t, *J* = 6.8 Hz, 1H), 7.53 – 7.44 (m, 2H), 5.62 (td, *J* = 17.0, 7.8 Hz, 1H), 5.19 (d, *J* = 17.0 Hz, 1H), 5.10 (d, *J* = 10.1 Hz, 1H), 3.93 – 3.82 (m, 1H), 2.85 (dd, *J* = 13.6, 6.5 Hz, 1H), 2.68 (dd, *J* = 13.6, 7.9 Hz, 1H), 2.45 – 2.19 (m, 1H), 1.69 – 1.51 (m, 2H), 1.51 – 1.30 (m, 2H), 0.93 (t, *J* = 7.2 Hz, 3H). **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ = 178.00, 161.61, 133.04, 130.59, 128.92, 128.16, 125.47, 120.80, 77.92, 77.48, 77.16, 76.84, 74.28, 38.17, 33.07, 19.41, 13.91. **IR** (neat): v = 3400, 2960, 2900, 2848, 1815, 1648, 1580, 1494, 1451, 1322, 1289, 1179, 1156, 1042, 1024, 969, 926, 889, 779, 690 $cm^{-1}$. **HRMS** (ESI/$[M+H]^+$) = Calcd. for $C_{16}H_{20}NO_3$ m/z = 274.1443, found m/z = 274.1446.



*ent*-**8dc**

94% yield, -92% ee, *anti*/*syn* = 97/3, 136 h

Product *ent*-**8dc** was obtained as a yellow oil (25.8 mg) from a reaction catalyzed by **3d**. Enantiomeric excess and diastereometric ratio were determined by HPLC analysis: Daicel Chiralcel OJ-H, Hexanes/IPA = 97/3, 1.0 ml/min, λ = 254 nm, t (*anti*, major) = 18.21 min, t (*anti*, minor) = 20.54 min, t (*syn*, major) = 25.48, t (*syn*, minor) = 35.65. $[\alpha]_D^{20}$ = -26.2 (c = 0.33, $CH_2Cl_2$). All the other data were the same as product **8dc**.



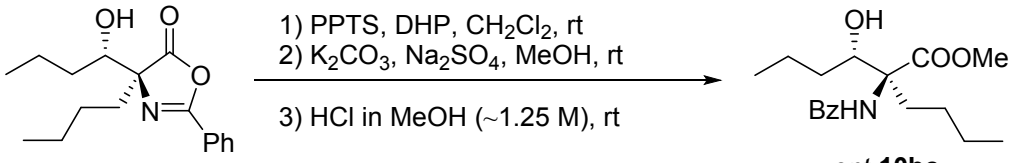
**8ad**

83% yield, 92% ee, *anti*/*syn* = 95.5/4.5, 131 h

Product **8ad** was obtained as a colorless oil (36 mg) from a reaction catalyzed by **3d**. Enantiomeric excess and diastereometric ratio were determined by HPLC analysis: Daicel Chiralcel AS-H, Hexanes/IPA = 96.5/3.5, 1.0 ml/min, λ = 254 nm, t (*anti*, minor) = 16.62 min, t (*anti*, major) = 20.50 min, t (*syn*, major) = 29.36, t (*syn*, minor) = 44.78. $[\alpha]_D^{20}$ = +74 (c = 0.45, $CH_2Cl_2$). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 7.81 (d, *J* = 7.5 Hz, 2H), 7.51 (t, *J* = 7.5 Hz, 1H), 7.39 (t, *J* = 7.6 Hz, 2H), 7.17 – 7.05 (m, 5H), 3.91

(dd, *J* = 12.5, 6.7 Hz, 1H), 3.39 (d, *J* = 13.4 Hz, 1H), 3.23 (d, *J* = 13.4 Hz, 1H), 2.16 (d, *J* = 7.2 Hz, 1H), 1.65 – 1.47 (m, 5H), 1.22 (s, 20H), 0.85 (t, *J* = 6.7 Hz, 3H). **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ = 178.05, 161.35, 134.23, 132.84, 130.27, 128.78, 128.25, 127.97, 127.27, 125.37, 78.94, 77.48, 77.16, 76.84, 74.97, 40.10, 32.04, 31.48, 29.75, 29.72, 29.70, 29.48, 26.32, 22.82, 14.27. **IR** (neat): v = 3293, 2922, 2852, 1816, 1714, 1650, 1581, 1495, 1452, 1322, 1292, 1237, 1171, 1056, 973, 893, 777, 696, 611 $cm^{-1}$. **HRMS** (ESI/$[M+H]^+$) = Calcd. for $C_{16}H_{20}NO_3$ m/z = 436.2852, found m/z = 436.2852.

## 4. Transformation of Aldol products



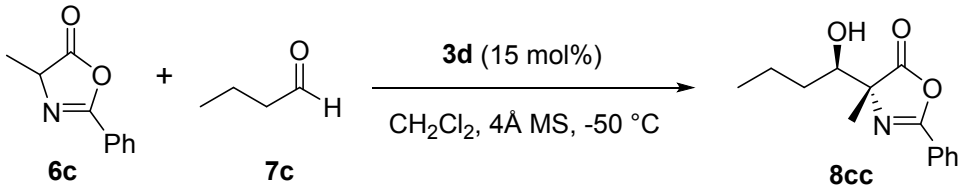

*ent*-**8bc**

*ent*-**10bc**

87% yield, -92% ee

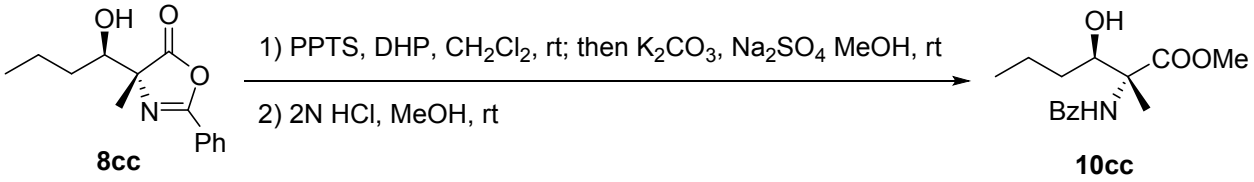
Aldol product *ent*-**8bc** was made according to the general procedure reported in previous section. To a 4 mL vial containing Aldol product *ent*-**8bc** (from 0.1 mmol scale reaction) were added dihydropyran (DHP, 0.14 mL, 1.5 mmol, 15 eq), $CH_2Cl_2$ (0.5 mL) and pyridinium *p*-toluenesulfonate (PPTS, 25.1 mg, 0.1 mmol, 1 eq) sequentially. The reaction mixture was stirred at room temperature for 20 h. After protection with DHP was complete, anhydrous $Na_2SO_4$ (100 mg), anhydrous MeOH (0.5 mL) and $K_2CO_3$ (34.6 mg, 0.25 mmol, 2.5 eq) were added to the reaction mixture. After 16 h, HCl in MeOH (1 mL, ~1.25 M) was added. After stirring at room temperature for 19.5 h, the reaction was concentrated and purified by flash column chromatography (1/10 = EA/Hex to 1/5) to give *ent*-**10bc** as a colorless oil (28 mg, 87% yield based on a 0.1 mmol Aldol reaction). All reaction times were not optimized. Racemic product was obtained according to the same procedure. Enantiomeric excess of *ent*-**10bc** was determined by HPLC analysis: Daicel Chiralpak AD-H, Hexanes/IPA = 90/10, 1.0 ml/min, λ = 254 nm, t (*anti*, major) = 9.17 min, t (*anti*, minor) = 10.23 min. $[\alpha]_D^{20}$ = -11.1 (c = 0.35, $CH_2Cl_2$). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 7.85 (d, *J* = 7.4 Hz, 2H), 7.70 (s, 1H), 7.57 (t, *J* = 7.3 Hz, 1H), 7.49 (t, *J* = 7.5 Hz, 2H), 5.67 (d, *J* = 10.9 Hz, 1H), 4.06 – 3.96 (m, 1H), 3.86 (s, 3H), 2.66 (td, *J*

= 14.0, 3.9 Hz, 1H), 2.01 – 1.87 (m, 1H), 1.61 (tt, *J* = 11.4, 5.9 Hz, 1H), 1.46 – 1.17 (m, 6H), 1.13 – 0.92 (m, 2H), 0.86 (q, *J* = 7.0 Hz, 6H). **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ = 174.21, 167.93, 133.78, 132.29, 128.92, 127.29, 75.36, 71.10, 53.63, 36.33, 32.22, 26.56, 22.66, 19.32, 14.05, 14.04. **IR** (neat): v = 3396, 2957, 2931, 2872, 1729, 1645, 1602, 1579, 1516, 1485, 1440, 1339, 1290, 1249, 1215, 1185, 1128, 1074, 1016, 960, 914, 866, 822, 711, 690, 664 $cm^{-1}$. **HRMS** (ESI/$[M+H]^+$) = Calcd. for $C_{18}H_{28}NO_4$ m/z = 322.2018, found m/z = 322.2011.




**6c** 105 mg, 0.6 mmol **7c** 81 µL, 0.9 mmol **8cc**

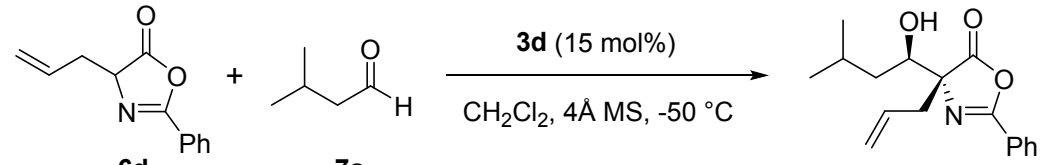
Aldol product **8cc** was made from a 0.6 mmol reaction (4 d) according to the general procedure reported in the previous section. It was used directly for the following steps.




**8cc** **10cc** 81% yield, 95% ee

To a 22 mL vial containing Aldol product **8cc** (from 0.6 mmol scale reaction) were added dihydropyran (DHP, 0.82 mL, 9 mmol, 15 eq), $CH_2Cl_2$ (3 mL) and pyridinium *p*-toluenesulfonate (PPTS, 151 mg, 0.6 mmol, 1 eq) sequentially. The reaction mixture was stirred at room temperature for 9 h. After protection with DHP was complete, anhydrous $Na_2SO_4$ (600 mg), anhydrous MeOH (3 mL) and $K_2CO_3$ (207 mg, 1.5 mmol, 2.5 eq) were added to the reaction mixture. After 6.5 h, the reaction mixture was passed through a short silica gel column and concentrated. Then 2N HCl (1 mL) and MeOH (4 mL) was added. After stirring at room temperature for 17 h, the reaction was concentrated and purified by flash column chromatography (1/5 = EA/Hex to 2/5) to give **10cc** as a colorless oil (135 mg, 81%

yield based on a 0.6 mmol Aldol reaction). All reaction times were not optimized. Racemic product was obtained according to the same procedure. Enantiomeric excess of **10cc** was determined by HPLC analysis: Daicel Chiralpak AD-H, Hexanes/IPA = 90/10, 1.0 ml/min, λ = 254 nm, t (*anti*, minor) = 12.87 min, t (*anti*, major) = 13.80 min. $[\alpha]_D^{20}$ = -8.4 (c = 0.45, $CH_2Cl_2$). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 7.83 (d, *J* = 7.6 Hz, 2H), 7.64 (s, 1H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.48 (t, *J* = 7.4 Hz, 2H), 5.18 (d, *J* = 10.6 Hz, 1H), 3.98 (t, *J* = 10.1 Hz, 1H), 3.86 (s, 3H), 1.74 (s, 3H), 1.69 – 1.53 (m, 1H), 1.46 – 1.27 (m, 2H), 1.24 – 1.10 (m, 1H), 0.88 (t, *J* = 7.0 Hz, 3H). **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ = 174.96, 167.95, 133.89, 132.31, 128.94, 127.36, 75.13, 66.15, 53.70, 35.91, 20.80, 19.53, 14.15. **IR** (neat): v = 3394, 2964, 2875, 1731, 1655, 1622, 1575, 1537, 1490, 1463, 1441, 1379, 1325, 1236, 1160, 1115, 1063, 1027, 999, 973, 941, 917, 874, 829, 804, 715, 694, 613 $cm^{-1}$. **HRMS** (ESI/$[M+H]^+$) = Calcd. for $C_{15}H_{22}NO_4$ m/z = 280.1549, found m/z = 280.1549.

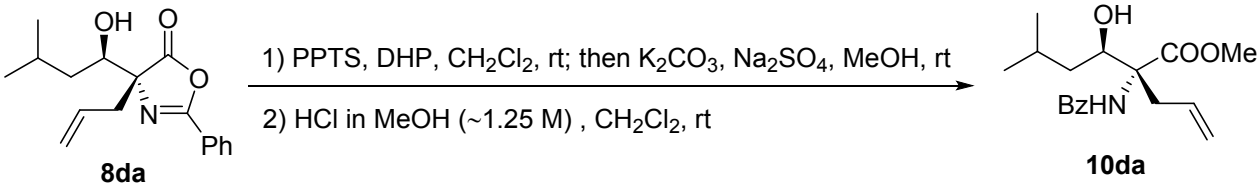



**6d** 101 mg, 0.5 mmol

**7a** 81 µL, 0.75 mmol

**8da**

Aldol product **8da** was made from a 0.5 mmol reaction (5 d) according to the general procedure reported in the previous section. It was used directly for the following steps.



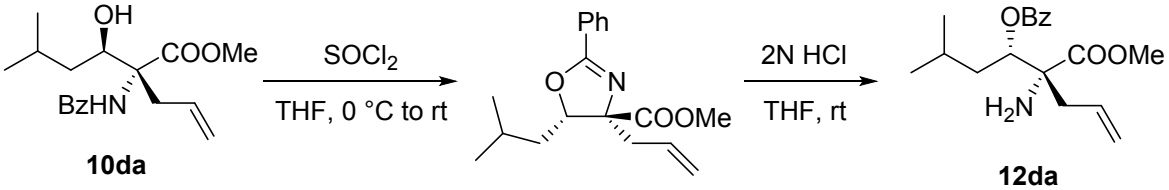

**8da**

**10da**

90% yield, 95% ee

To a 22 mL vial containing Aldol product **8da** (from 0.5 mmol scale reaction) were added dihydropyran (DHP, 0.68 mL, 7.5 mmol, 15 eq), $CH_2Cl_2$ (3 mL) and pyridinium *p*-toluenesulfonate (PPTS, 126 mg, 0.5 mmol, 1 eq) sequentially. The reaction mixture was

stirred at room temperature for 17.5 h. After protection with DHP was complete, anhydrous $Na_2SO_4$ (500 mg), anhydrous MeOH (2.5 mL) and $K_2CO_3$ (138 mg, 1 mmol, 2 eq) were added to the reaction mixture. After 18 h, the reaction mixture was passed through a short silica gel column and concentrated to remove all volatiles. Then HCl in MeOH (1 mL, ~1.25 M) and $CH_2Cl_2$ (2 mL) were added. After stirring at room temperature for 6.5 h, the reaction was concentrated and purified by flash column chromatography (1/10 = EA/Hex) to give **10da** as a colorless oil (144 mg, 90% yield based on a 0.5 mmol Aldol reaction). All reaction times were not optimized. Racemic product was obtained according to the same procedure. Enantiomeric excess of **10da** was determined by HPLC analysis: Daicel Chiralpak AD-H, Hexanes/IPA = 90/10, 1.0 ml/min, λ = 254 nm, t (*anti*, minor) = 8.96 min, t (*anti*, major) = 9.76 min. $[\alpha]_D^{20}$ = +28.1 (c = 0.59, $CH_2Cl_2$). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 7.83 (d, *J* = 7.2 Hz, 2H), 7.65 – 7.53 (m, 2H), 7.48 (t, *J* = 7.4 Hz, 2H), 5.68 – 5.54 (m, 2H), 5.14 – 5.04 (m, 2H), 4.11 (t, *J* = 10.7 Hz, 1H), 3.85 (s, 3H), 3.40 (dd, *J* = 14.3, 7.8 Hz, 1H), 2.68 (dd, *J* = 14.4, 7.3 Hz, 1H), 2.01 – 1.82 (m, 1H), 1.45 – 1.34 (m, 1H), 0.87 (dd, *J* = 9.7, 6.8 Hz, 6H), 0.81 (d, *J* = 13.2 Hz, 1H). **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ = 173.44, 168.18, 133.79, 132.33, 132.12, 128.92, 127.34, 119.94, 73.46, 71.02, 53.58, 43.36, 37.06, 24.53, 23.98, 21.55. **IR** (neat): v = 3396, 2954, 2869, 1731, 1644, 1602, 1579, 1515, 1485, 1439, 1334, 1259, 1224, 1179, 1132, 1089, 1058, 1032, 984, 957, 922, 890, 837, 799, 739, 712, 690, 666, 623 $cm^{-1}$. **HRMS** (ESI/$[M+H]^+$) = Calcd. for $C_{18}H_{26}NO_4$ m/z = 320.1862, found m/z = 320.1867.




**10da**
*anti*, 95% ee

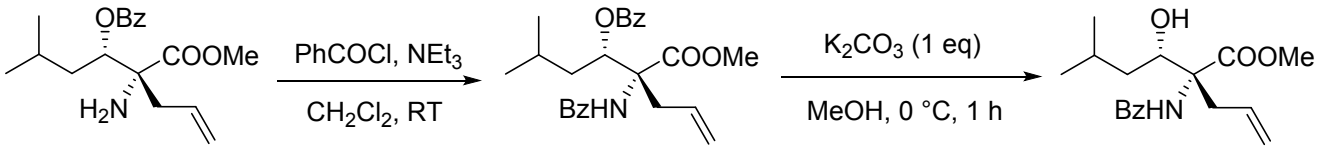
**11da**

**12da**
85% yield, *syn*

To a 22 mL vial containing product **10da** (52.8 mg, 0.165 mmol) was charged 1.5 mL anhydrous THF, followed by addition of freshly distilled thionyl chloride (120 μL, 1.65 mmol, 10 eq). The reaction was stirred at room temperature for 24 h. Then 1 mL 2N HCl and 0.5 mL THF was added to the reaction mixture. After stirring at room temperature for 22.5 h, saturated $NaHCO_3$ (aq) was added to quench the reaction and render the reaction mixture basic (pH ≈

10), followed by extraction with $Et_2O$ (10 mL x 3). The combined organic layer was washed by brine, dried over $MgSO_4$ and concentrated. The crude was purified by flash column chromatography (1/5 = EA/Hex) to give product **12da** as a pale yellow oil (44.7 mg, 85 % yield over 2 steps). All reaction times were not optimized. $[\alpha]_D^{20}$ = -23.6 (c = 1.00, $CH_2Cl_2$). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 8.01 (d, *J* = 7.6 Hz, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.6 Hz, 2H), 5.64 (td, *J* = 16.7, 9.0 Hz, 1H), 5.54 (d, *J* = 10.5 Hz, 1H), 5.24 – 5.13 (m, 2H), 3.65 (s, 3H), 2.65 (dd, *J* = 13.3, 6.2 Hz, 1H), 2.28 (dd, *J* = 13.3, 8.5 Hz, 1H), 1.90 – 1.79 (m, 1H), 1.73 (s, 2H), 1.66 – 1.52 (m, 1H), 1.50 – 1.38 (m, 1H), 0.99 (d, *J* = 6.4 Hz, 3H), 0.92 (d, *J* = 6.6 Hz, 3H). **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ = 175.62, 165.66, 133.17, 131.86, 129.94, 129.77, 128.52, 120.50, 76.59, 64.39, 52.49, 40.20, 38.34, 24.51, 24.02, 21.68. **IR** (neat): v = 3074, 2956, 2929, 2871, 2366, 2343, 1728, 1720, 1602, 1451, 1269, 1217, 1177, 1109, 1070, 1026, 997, 925, 855, 815, 712, 667 $cm^{-1}$. **HRMS** (ESI/$[M+H]^+$) = Calcd. for $C_{18}H_{26}NO_4$ m/z = 320.1862, found m/z = 320.1856.

Determination of enantiomeric excess of product **11da**:

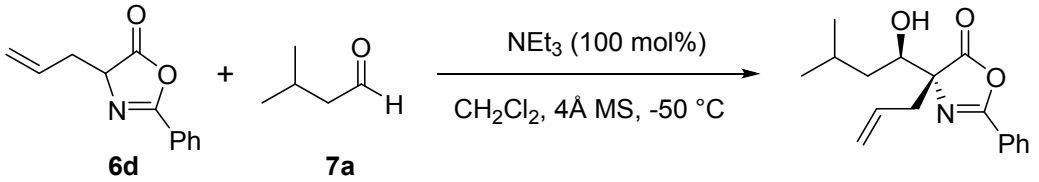



**12da** **13da** **14da**, 95% ee

To a solution of **12da** (23.2 mg, 0.073 mmol) and benzoyl chloride (12.7 µL, 1.5 eq) in $CH_2Cl_2$ (1 mL) was added triethylamine (30 µL, 3 eq). The reaction was stirred at room temperature for 17 h. Then 1N HCl was added to quench the reaction, followed by extraction with $Et_2O$ (10 mL x 3). The combine organic layer was washed with sat. $NaHCO_3$ and brine. Then it was dried over $MgSO_4$ and concentrated. The crude was purified by flash column chromatography (1/10 = EA/Hex) to give **13da** as a colorless oil (27.3 mg, 89% yield) which contained small amount of impurities. Half of this compound (13.5 mg) was dissolved in MeOH (1 mL) and cooled in an ice-bath with stirring. Then $K_2CO_3$ (6.6 mg) was added in one portion to the reaction. After stirring at 0 C for 1 h, the reaction was diluted by water and extracted with $Et_2O$ (5 mL x 3). The combined organic layer was washed with brine, dried over $MgSO_4$ and concentrated. The

crude was purified by preparative thin-layer chromatography to give **14da** as a pale white solid (8.4 mg, 83% yield, 95% ee). Enantiomeric excess of **14da** was determined by HPLC analysis: Daicel Chiralpak AD-H, Hexanes/IPA = 90/10, 1.0 ml/min, λ = 254 nm, t (*anti*, minor) = 7.79 min, t (*anti*, major) = 12.99 min. $[\alpha]_D^{20}$ = -148.1 (c = 0.15, $CH_2Cl_2$). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 7.78 (d, *J* = 7.7 Hz, 2H), 7.54 (t, *J* = 7.2 Hz, 1H), 7.46 (t, *J* = 7.4 Hz, 2H), 6.71 (s, 1H), 5.69 (dt, *J* = 15.3, 9.5 Hz, 1H), 5.39 – 5.23 (m, 2H), 4.76 (d, *J* = 11.0 Hz, 1H), 4.13 (t, *J* = 10.6 Hz, 1H), 3.83 (s, 3H), 2.95 (dd, *J* = 13.7, 4.8 Hz, 1H), 2.71 (dd, *J* = 13.7, 9.8 Hz, 1H), 2.03 – 1.87 (m, 1H), 1.44 – 1.32 (m, 1H), 1.32 – 1.20 (m, 2H), 0.93 (t, *J* = 7.0 Hz, 6H). **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ = 171.96, 168.23, 134.41, 132.48, 132.05, 128.83, 127.15, 121.01, 73.38, 69.01, 53.02, 41.62, 38.84, 24.72, 24.09, 21.59. **IR** (neat): ν = 3460, 3389, 2954, 2865, 1730, 1637, 1603, 1579, 1517, 1481, 1435, 1413, 1330, 1292, 1258, 1231, 1176, 1137, 1092, 1055, 1030, 988, 960, 919, 887, 871, 846, 803, 773, 707, 687, 626 $cm^{-1}$. **HRMS** (ESI/$[M+H]^+$) = Calcd. for $C_{18}H_{26}NO_4$ m/z = 320.1862, found m/z = 320.1862.

## 5. Determination of relative configurations

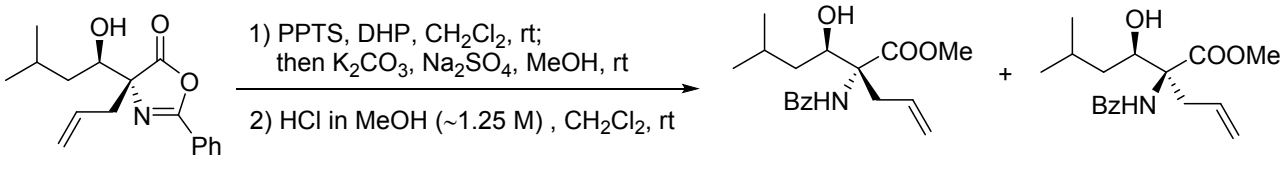



**6d** 0.53 mmol

**7a** 0.8 mmol, 1.5 eq

*rac*-**8da**

Aldol product *rac*-**8da** was made from a 0.53 mmol reaction (4 d) according to the general procedure reported in the previous section. It was used directly for the following steps.

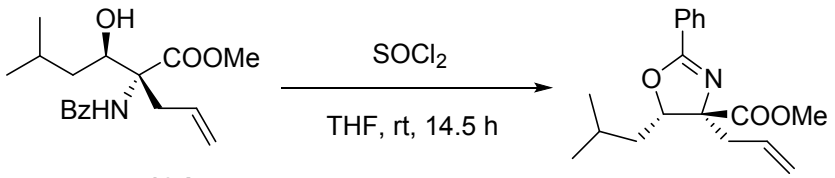



*rac*-**8da**

*anti*, *rac*-**10da**

*syn*, *rac*-**14da**

The reation was carried out by following the same procedure used for compound **10da**, giving *rac*-**10da** as a colorless oil (90.6 mg, 53% yield) and *rac*-**14da** as a pale yellow solid (64.5 mg, 38% yield). All spectroscopic data were the same as those of **10da** and **14da**.

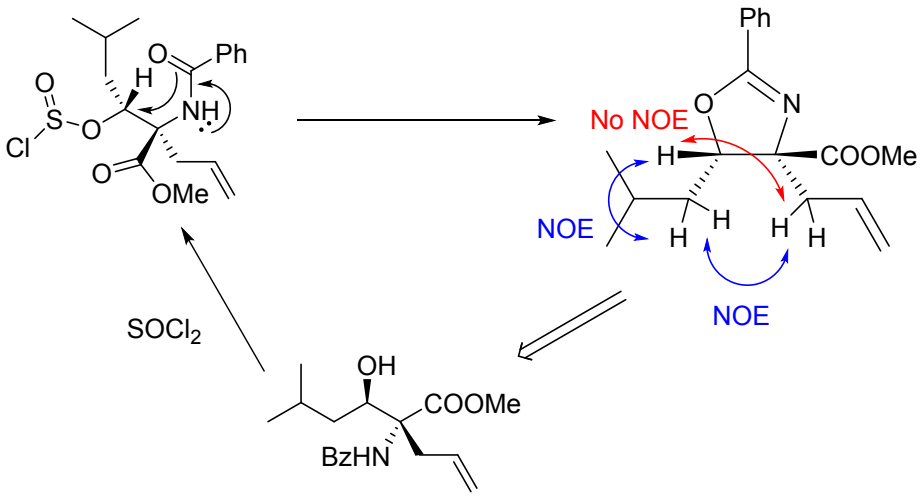



*anti*, *rac*-**10da**

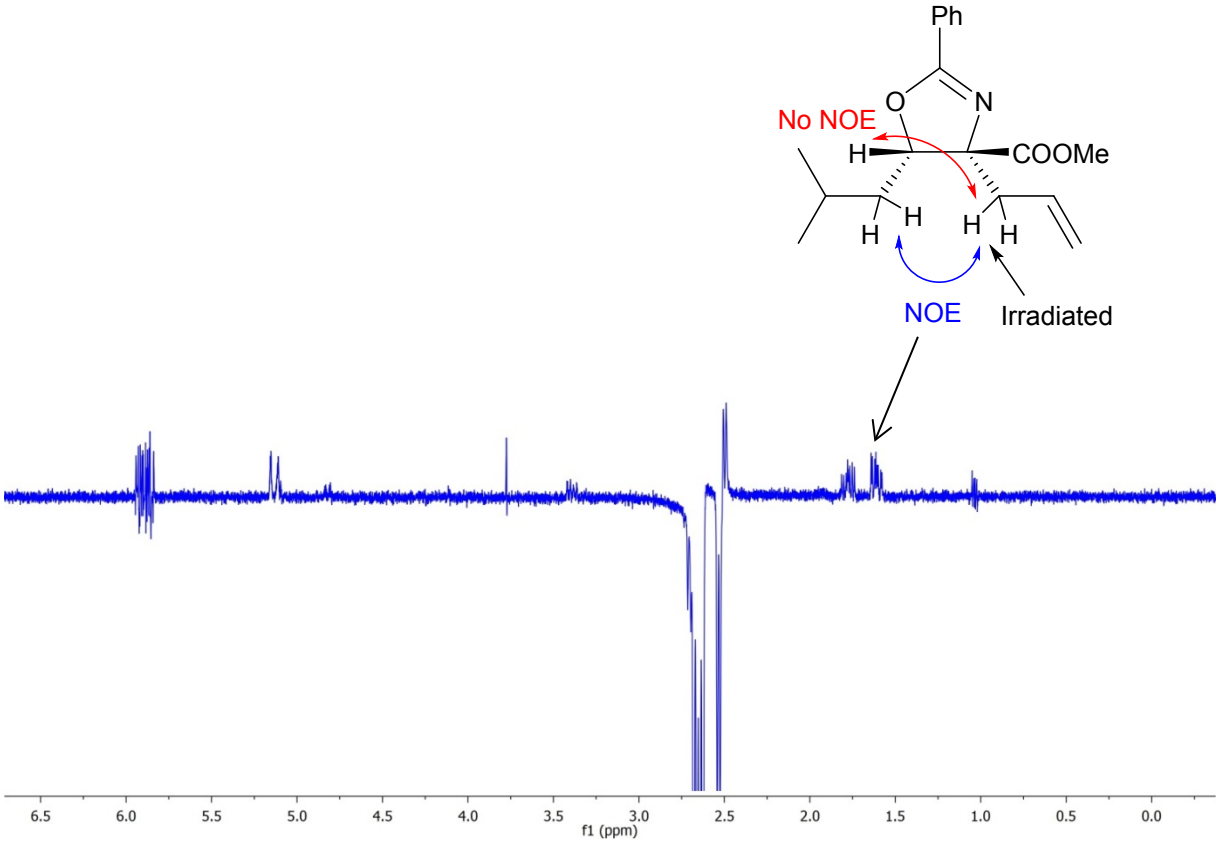
*rac*-**11da**, 89% yield

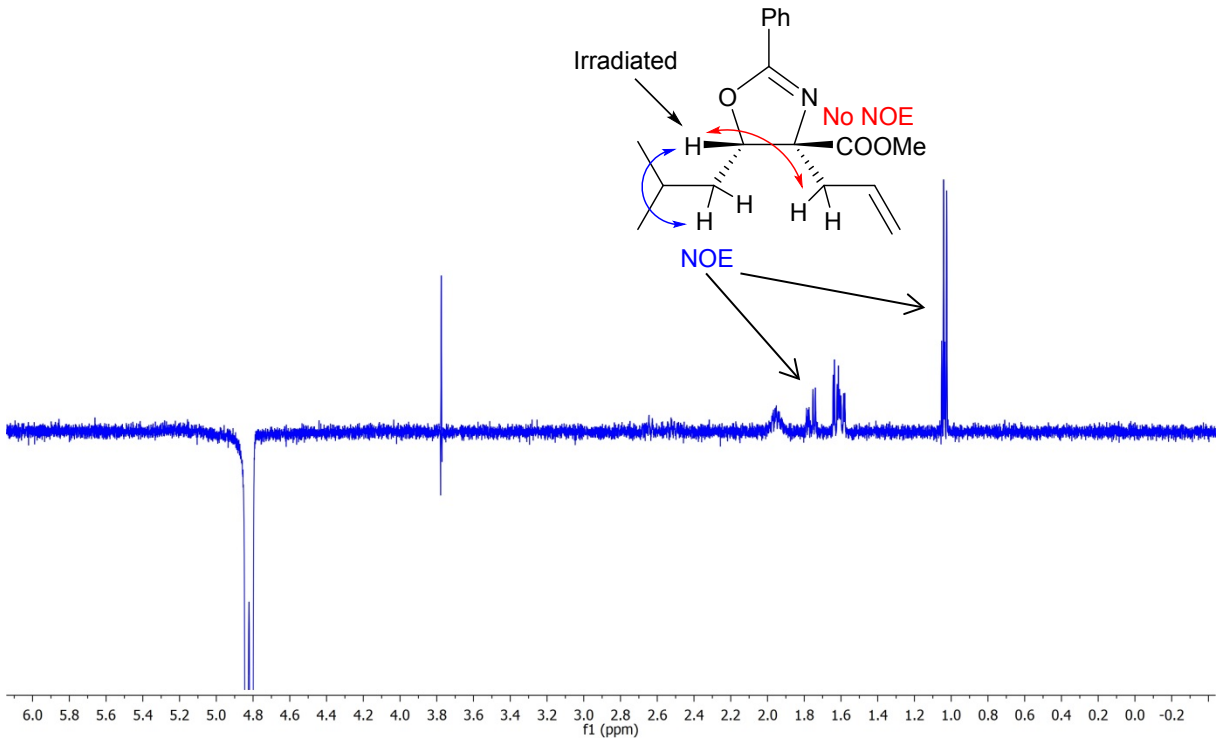
To a solution of rac-**10da** (30.7 mg, 0.096 mmol) in 1 mL anhydrous THF was added freshly distilled thionyl chloride (35 μL, 0.48 mmol, 5 eq) at room temperature. The reaction was stirred at room temperature for 14.5 h. Then saturated $NaHCO_3$ (aq) was added to quench the reaction and render the reaction mixture basic (pH ≈ 10), followed by extraction with $Et_2O$ (10 mL x 3). The combined organic layer was washed by brine, dried over $MgSO_4$ and concentrated. The crude was purified by flash column chromatography (1/5 = EA/Hex) to give product *rac*-**11da** as a brown oil (25.9 mg, 89% yield). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 8.02 – 7.97 (m, 2H), 7.52 – 7.45 (m, 1H), 7.44 – 7.36 (m, 2H), 5.97 – 5.81 (m, 1H), 5.18 – 5.08 (m, 2H), 4.82 (dd, *J* = 10.9, 2.6 Hz, 1H), 3.78 (s, 3H), 2.65 (dd, *J* = 13.9, 7.6 Hz, 1H), 2.52 (dd, *J* = 13.9, 6.5 Hz, 1H), 2.03 – 1.87 (m, 1H), 1.78 (ddd, *J* = 14.1, 10.9, 5.2 Hz, 1H), 1.61 (ddd, *J* = 14.0, 8.5, 2.7 Hz, 1H), 1.04 (dd, *J* = 6.6, 4.2 Hz, 6H). **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ = 173.80, 164.23, 132.96, 131.80, 128.67, 128.35, 127.59, 118.85, 83.68, 79.08, 52.65, 38.55, 38.31, 26.08, 23.59, 21.89. **IR** (neat): v = 3073, 2956, 2877, 2341, 1963, 1911, 1730, 1643, 1580, 1495, 1450, 1444, 1354, 1322, 1250, 1228, 1136, 1086, 1069, 1026, 975, 919, 836, 780, 697 $cm^{-1}$. **HRMS** (ESI/$[M+H]^+$) = Calcd. for $C_{18}H_{26}NO_4$ m/z = 302.1756, found m/z = 302.1763.

Relative configuration of *rac*-**11da** was determined by 1D NOESY experiment. Therefore, relative configuration of *rac*-**10da** was determined to be *anti*. Relative configurations of all the other products were assigned based on these results.

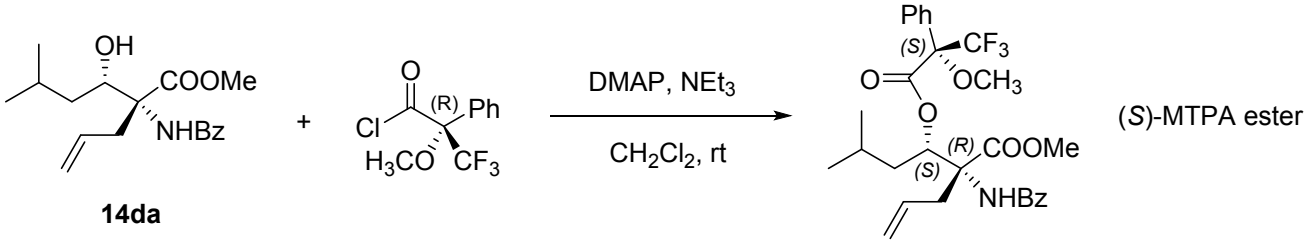

Ph
O
N
No NOE
H
COOMe
NOE
H
H
H
H
NOE
SOCl2
OH
COOMe
BzHN

**10da**


Ph
O
N
No NOE
H
COOMe
H
H
H
H
NOE
Irradiated
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

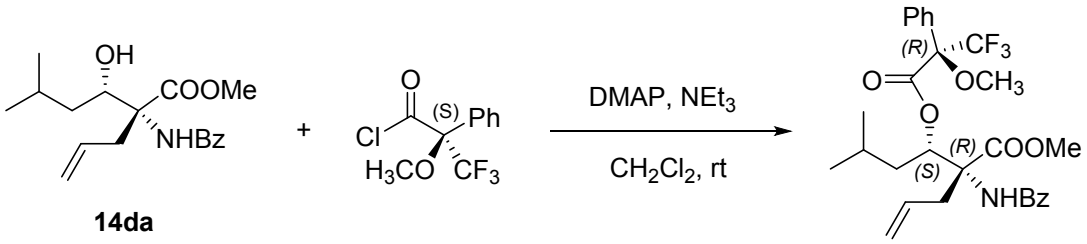



## 6. Determination of absolute configurations




To a solution of **14da** (4.2 mg, 0.013 mmol) in $CH_2Cl_2$ (0.5 mL) were added (*R*)-(−)-α-Methoxy-α-(trifluoromethyl)phenylacetyl chloride ((*R*)-(−)-MTPA-Cl, Mosher's acid chloride, 4 mg, 0.0156 mmol, 1.2 equiv), triethylamine (18 μL, 0.13 mmol, 10 equiv) and 4-dimethylaminopyridine (DMAP, 3.2 mg, 0.026 mmol, 2 equiv) sequentially at room temperature. After stirring for 14 h, the reaction mixture was concentrated and purified by preparative thin-

layer chromatography (1/5 = EA/Hex) to give *(S)*-**15da** as a colorless oil (4.6 mg, 66% yield). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 7.72 (d, *J* = 7.7 Hz, 2H), 7.58 (d, *J* = 7.1 Hz, 2H), 7.52 (t, *J* = 7.3 Hz, 1H), 7.48 – 7.33 (m, 5H), 7.03 (s, 1H), 6.09 (d, *J* = 8.9 Hz, 1H), 5.57 (td, *J* = 17.3, 7.8 Hz, 1H), 5.10 – 4.95 (m, 2H), 3.77 (s, 3H), 3.54 (s, 3H), 3.47 (dd, *J* = 13.8, 7.0 Hz, 1H), 2.55 (dd, *J* = 13.7, 7.6 Hz, 1H), 1.66 – 1.53 (m, 2H), 1.53 – 1.45 (m, 1H), 0.90 (d, *J* = 6.3 Hz, 3H), 0.85 (d, *J* = 6.5 Hz, 3H). **HRMS** (ESI/$[M+H]^+$) = Calcd. for $C_{18}H_{26}NO_4$ m/z = 536.2260, found m/z = 536.2265.

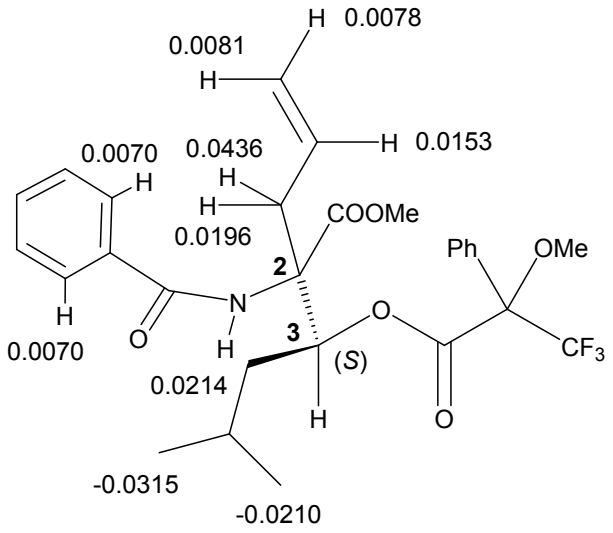



**14da**

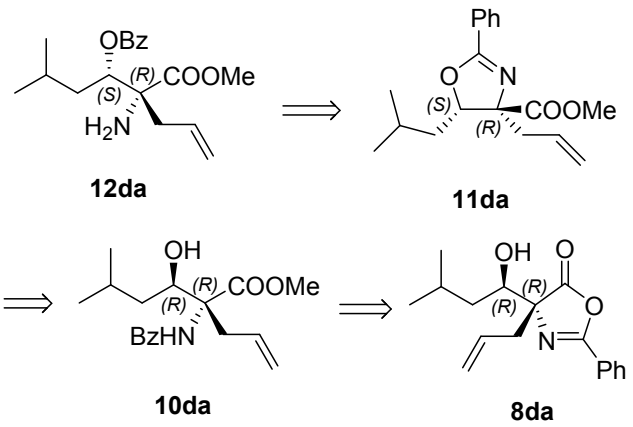
(*R*)-**15da**

(*R*)-MTPA ester

By following the same procedure, product *(R)*-**15da** was synthesized from (*S*)-(−)-MTPA-Cl and obtained as a colorless oil (4.5 mg, 65% yield). **$^1$H NMR** (400 MHz, $CDCl_3$): δ = 7.72 (d, *J* = 7.7 Hz, 2H), 7.58 (d, *J* = 7.1 Hz, 2H), 7.52 (t, *J* = 7.3 Hz, 1H), 7.48 – 7.33 (m, 5H), 7.03 (s, 1H), 6.09 (d, *J* = 8.9 Hz, 1H), 5.57 (td, *J* = 17.3, 7.8 Hz, 1H), 5.10 – 4.95 (m, 2H), 3.77 (s, 3H), 3.54 (s, 3H), 3.47 (dd, *J* = 13.8, 7.0 Hz, 1H), 2.55 (dd, *J* = 13.7, 7.6 Hz, 1H), 1.66 – 1.53 (m, 2H), 1.53 – 1.45 (m, 1H), 0.90 (d, *J* = 6.3 Hz, 3H), 0.85 (d, *J* = 6.5 Hz, 3H). **HRMS** (ESI/$[M+H]^+$) = Calcd. for $C_{18}H_{26}NO_4$ m/z = 536.2260, found m/z = 536.2267.




MPTA ester **15da**




By employing the modified Mosher's method[15-17], the absolute configuration at the C3 position of **15da** was determined to be *S* on the basis of the Δδ values ($\delta_{(S)\text{-MTPA}}$ - $\delta_{(R)\text{-MTPA}}$ in ppm). Based on the relative stereochemistry, the absolute configuration at the C2 position of **15da** was determined to be *R*. Therefore, the absolute configurations of **12da**, **11da**, **10da** and **8da** were all assigned. Absolute configurations of all the other products were assigned based on these results.

## 7. Reference: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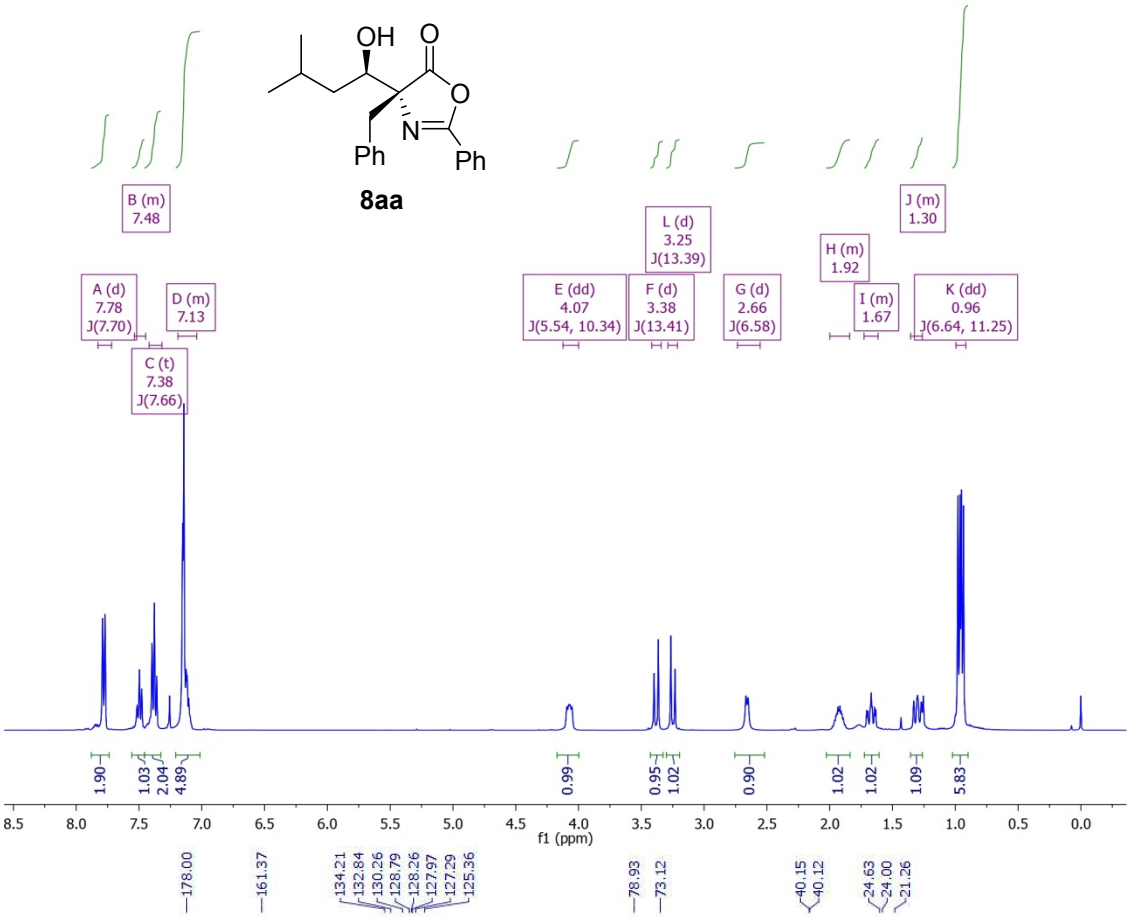
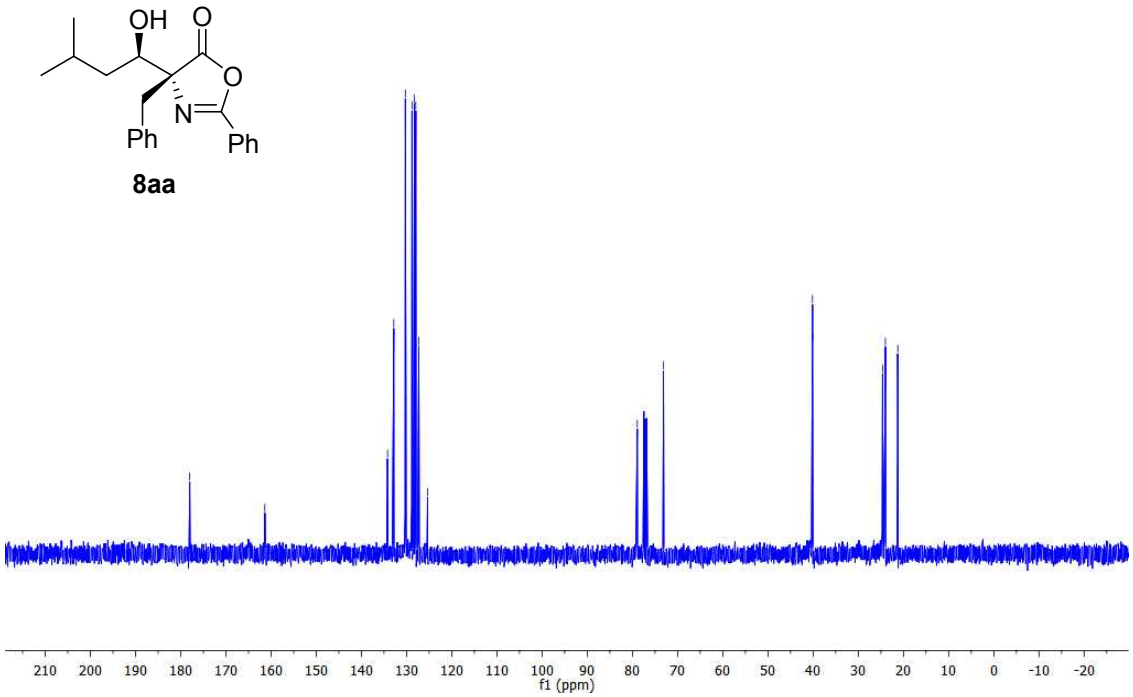
## 8. $^{1}H$ and $^{13}C$ NMR Spectra for New Compounds, HPLC Spectra for chiral products


OH
O
O
N
Ph
Ph
B (m)
7.48
A (d)
7.78
J(7.70)
D (m)
7.13
C (t)
7.38
J(7.66)
L (d)
3.25
J(13.39)
H (m)
1.92
J (m)
1.30
E (dd)
4.07
J(5.54, 10.34)
F (d)
3.38
J(13.41)
G (d)
2.66
J(6.58)
I (m)
1.67
K (dd)
0.96
J(6.64, 11.25)
1.90
1.03
2.04
4.89
0.99
0.95
1.02
0.90
1.02
1.02
1.09
5.83
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
178.00
161.37
134.21
132.84
130.26
128.79
128.26
127.97
127.29
125.36
78.93
73.12
40.15
40.12
24.63
24.00
21.26

**8aa**


OH
O
O
N
Ph
Ph
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

**8aa**

Data File C:\CHEM32\1\DATA\YZHENG\3-63RAC_1.D
Sample Name: 3-63rac

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Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
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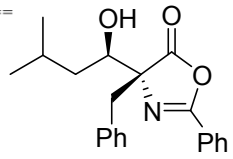
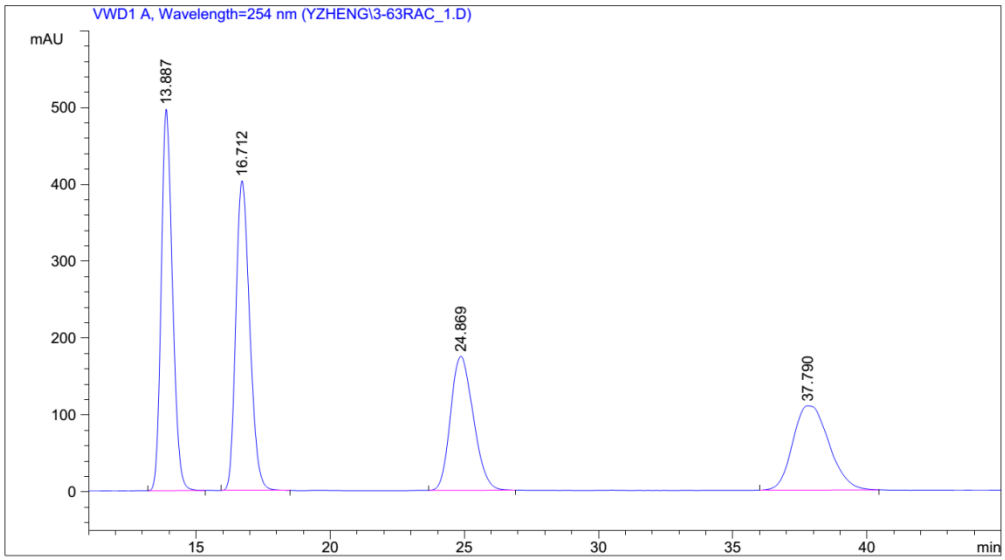
Last changed    : 8/1/2011 2:00:23 PM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 8/1/2011 5:37:18 PM by yang
                  (modified after loading)
Sample Info     : AS-H, Hex:IPA = 95:5, 1.0 mL/min, 254 nm, 44 bar, Right
```




**8aa**

Racemic




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

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

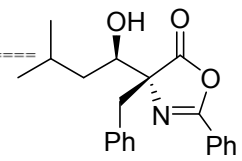

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  13.887 BB    0.4461 1.44565e4   496.35040  29.2101
   2  16.712 BB    0.5614 1.44665e4   403.04199  29.2304
   3  24.869 BB    0.9422 1.02947e4   174.34625  20.8009
   4  37.790 BB    1.4103 1.02737e4   109.66785  20.7586

Totals :                 4.94914e4  1183.40650


==========================================================================
                          *** End of Report ***
```

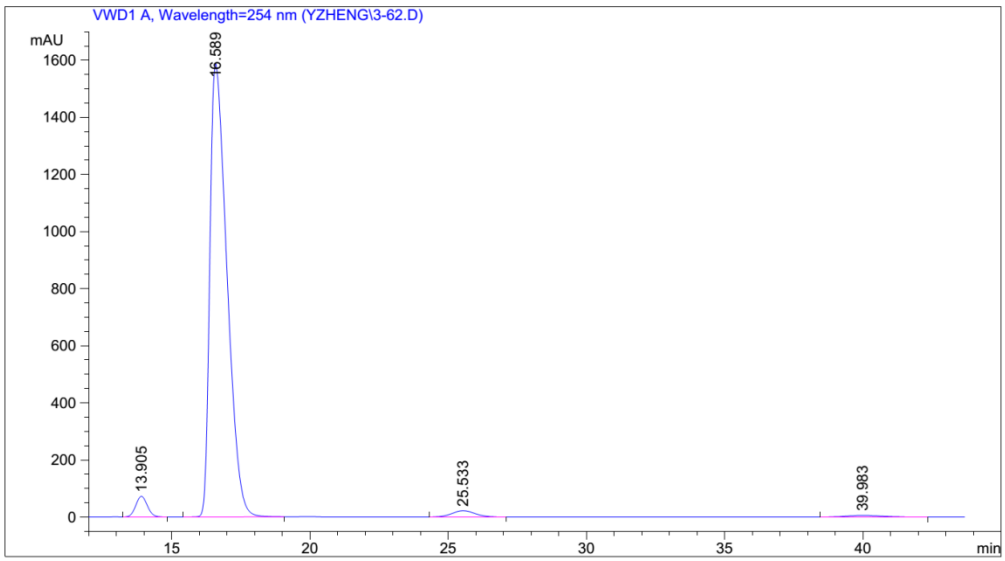
Data File C:\CHEM32\1\DATA\YZHENG\3-62.D
Sample Name: 3-62




**8aa**

94% ee, *anti*/*syn* = 97.5/2.5

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 8/1/2011 4:17:13 PM
Acq. Method     : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 8/1/2011 3:55:52 PM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\WASH.M
Last changed    : 8/10/2011 11:12:50 AM by jhl
                  (modified after loading)
Sample Info     : AS-H, Hex:IPA = 95:5, 1.0 mL/min, 254 nm, 44 bar, Right
```




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

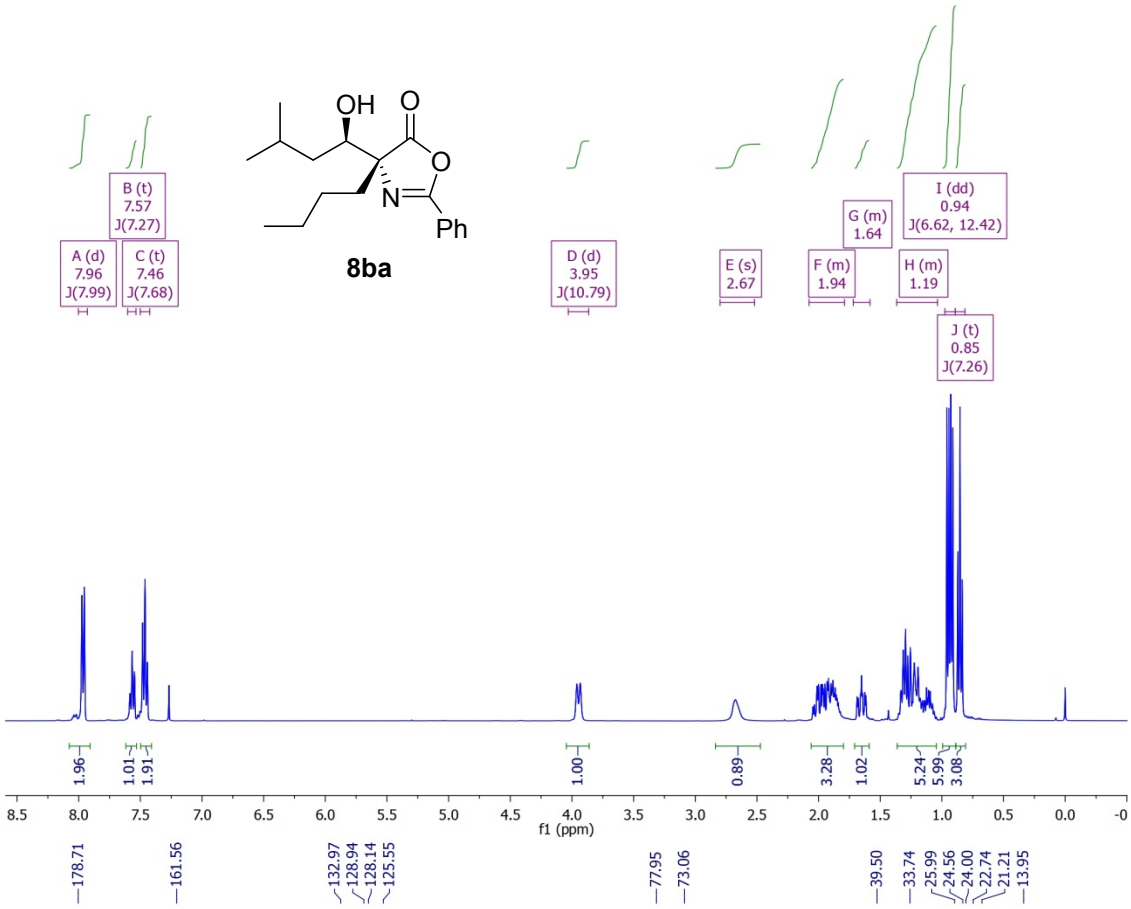
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

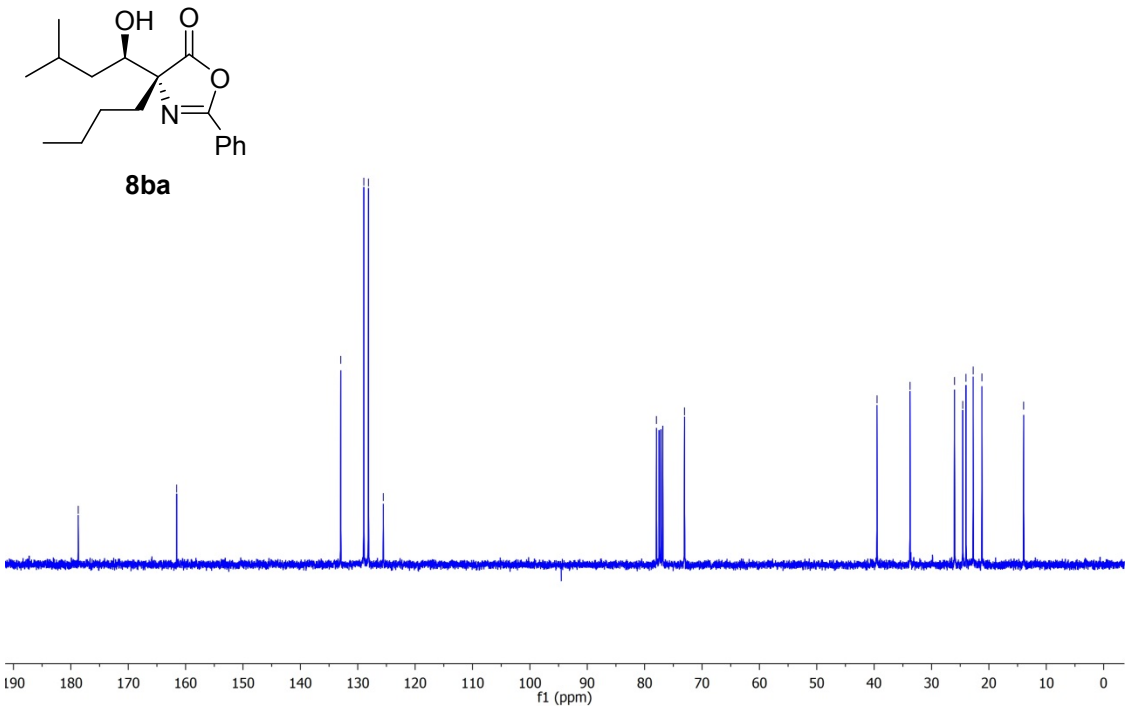

Signal 1: VWD1 A, Wavelength=254 nm

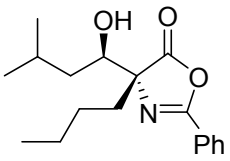
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  13.905 VB    0.4595 2129.86011   72.44035   2.9648
   2  16.589 BB    0.6545 6.78960e4  1588.21118  94.5130
   3  25.533 BB    0.8975 1288.76758   21.95257   1.7940
   4  39.983 BB    1.1716  523.13538    5.65183   0.7282

Totals :                  7.18378e4  1688.25594

==========================================================================
                        *** End of Report ***
```


OH
O
O
N
Ph
8ba
A (d) 7.96 J(7.99)
B (t) 7.57 J(7.27)
C (t) 7.46 J(7.68)
D (d) 3.95 J(10.79)
E (s) 2.67
F (m) 1.94
G (m) 1.64
H (m) 1.19
I (dd) 0.94 J(6.62, 12.42)
J (t) 0.85 J(7.26)
1.96
1.01
1.91
1.00
0.89
3.28
1.02
5.24
5.99
3.08
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)
178.71
161.56
132.97
128.94
128.14
125.55
77.95
73.06
39.50
33.74
25.99
24.56
24.00
22.74
21.21
13.95


OH
O
O
N
Ph
8ba
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

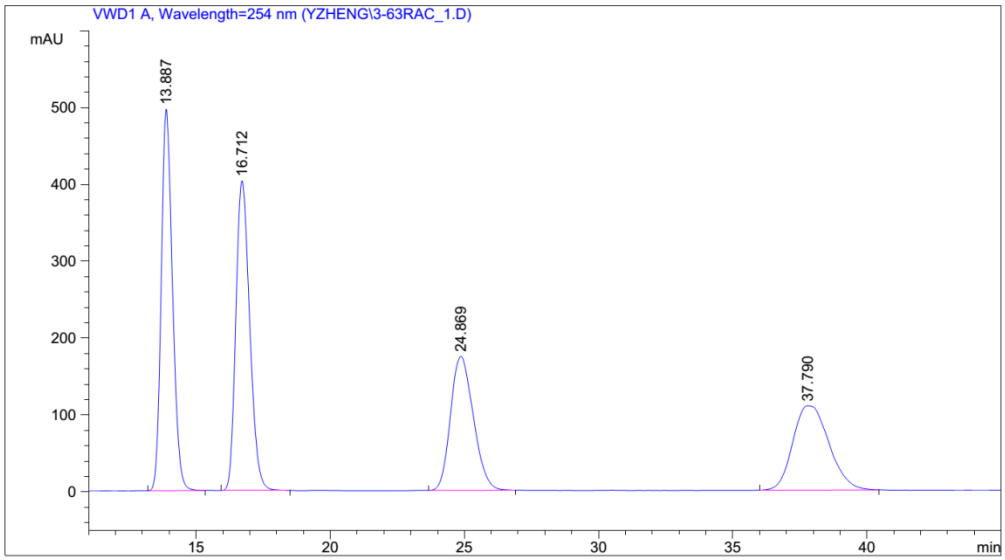



**8ba**

Racemic

```
Data File C:\CHEM32\1\DATA\YZHENG\3-63RAC_1.D
 Sample Name: 3-63rac

    ========================================================================
    Acq. Operator   : yang
    Acq. Instrument : Instrument 1                    Location :   -
    Injection Date  : 8/1/2011 2:32:52 PM
    Acq. Method     : C:\CHEM32\1\METHODS\METHOD-1_LC.M
    Last changed    : 8/1/2011 2:00:23 PM by yang
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\METHOD-1_LC.M
    Last changed    : 8/1/2011 5:37:18 PM by yang
                      (modified after loading)
    Sample Info     : AS-H, Hex:IPA = 95:5, 1.0 mL/min, 254 nm, 44 bar, Right
```




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

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


    Signal 1: VWD1 A, Wavelength=254 nm

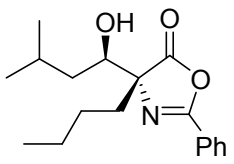
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  13.887 BB    0.4461 1.44565e4   496.35040  29.2101
       2  16.712 BB    0.5614 1.44665e4   403.04199  29.2304
       3  24.869 BB    0.9422 1.02947e4   174.34625  20.8009
       4  37.790 BB    1.4103 1.02737e4   109.66785  20.7586

    Totals :                  4.94914e4  1183.40650


    ========================================================================
                           *** End of Report ***

Instrument 1 8/1/2011 5:37:22 PM yang                          Page   1 of 1
```

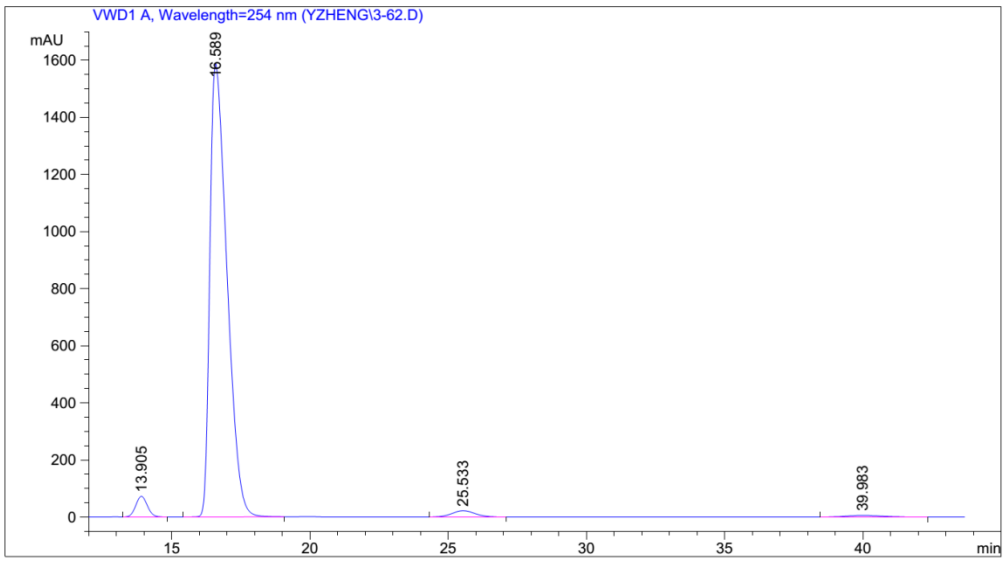
Data File C:\CHEM32\1\DATA\YZHENG\3-62.D
Sample Name: 3-62




**8ba**

95% ee, *anti*/*syn* = 98/2

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 8/1/2011 4:17:13 PM
Acq. Method     : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 8/1/2011 3:55:52 PM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\WASH.M
Last changed    : 8/10/2011 11:12:50 AM by jhl
                  (modified after loading)
Sample Info     : AS-H, Hex:IPA = 95:5, 1.0 mL/min, 254 nm, 44 bar, Right
```




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

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

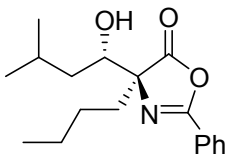

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  13.905 VB    0.4595 2129.86011   72.44035   2.9648
   2  16.589 BB    0.6545 6.78960e4  1588.21118  94.5130
   3  25.533 BB    0.8975 1288.76758   21.95257   1.7940
   4  39.983 BB    1.1716  523.13538    5.65183   0.7282

Totals :                  7.18378e4  1688.25594

==========================================================================
                        *** End of Report ***
```

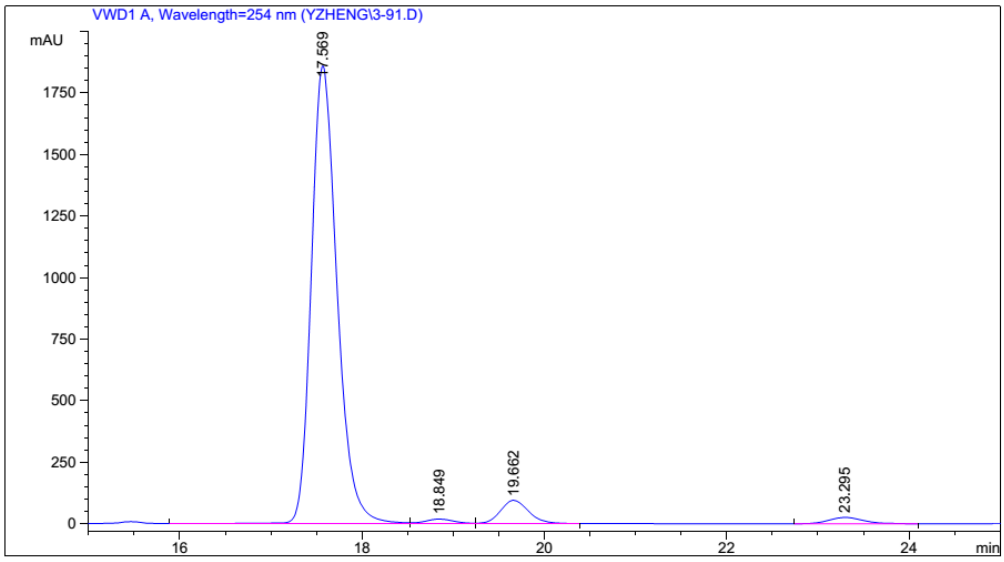
Data File C:\CHEM32\1\DATA\YZHENG\3-91.D
Sample Name: 3-91




*ent*-**8ba**

-90% ee, *anti*/*syn* = 97.5/2.5

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location : Vial 51
Injection Date  : 9/12/2011 5:33:19 PM
                                                  Inj Volume : 5 µl
Acq. Method     : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 9/12/2011 5:13:01 PM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\METHOD1.M
Last changed    : 9/12/2011 6:07:44 PM by xiao
                  (modified after loading)
Sample Info     : AD-H+AD, Hex:IPA = 95:5, 0.8 mL/min, 254 nm, 47 bar, Le
                  ft,
```



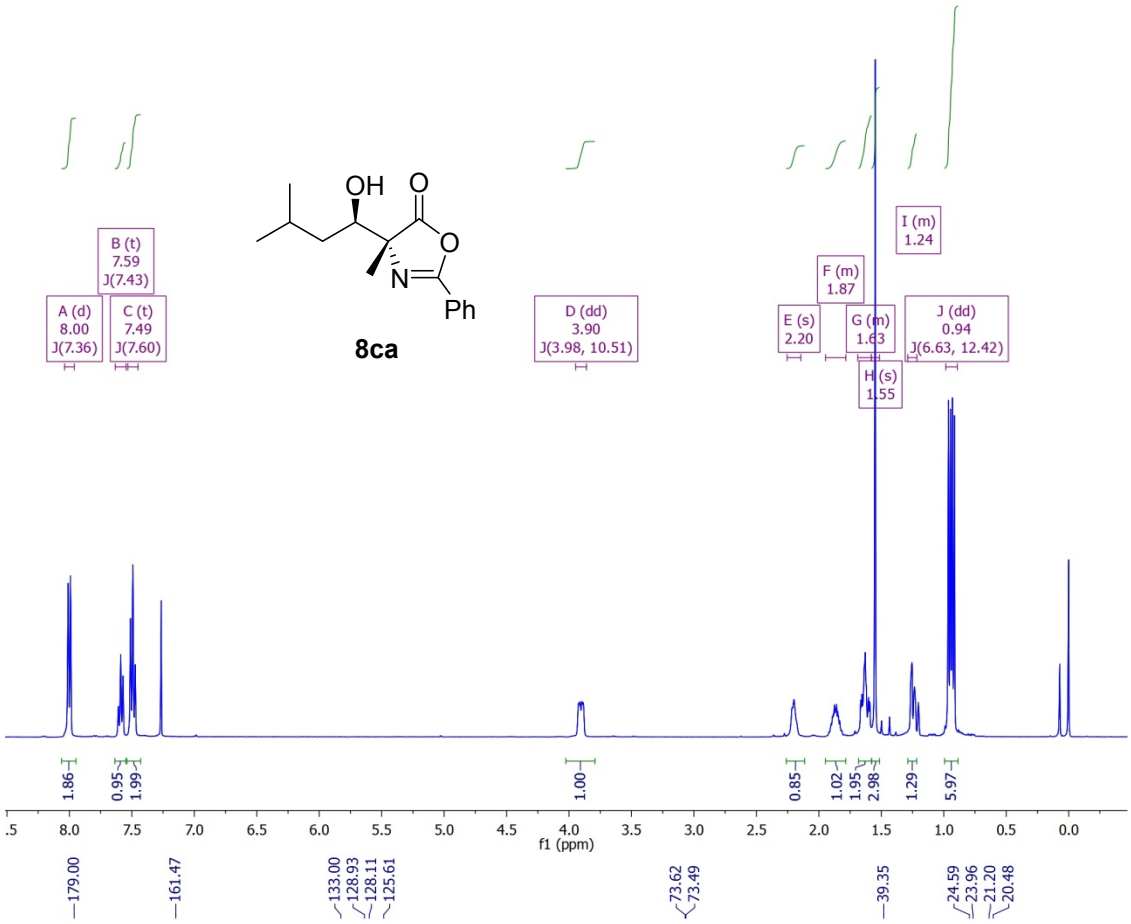

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

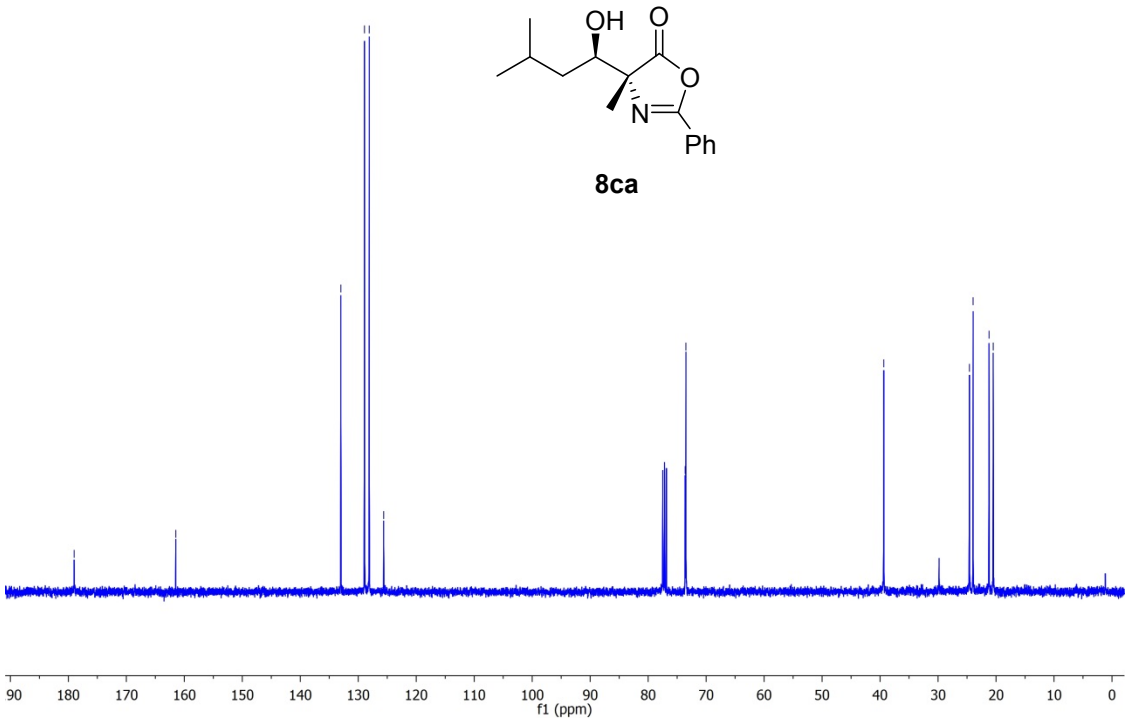

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  17.569 VV    0.3010 3.63684e4  1859.21899  92.2223
   2  18.849 VV    0.3221  391.98923   18.67219   0.9940
   3  19.662 VB    0.3268 2006.05396   94.84164   5.0869
   4  23.295 BB    0.4001  669.13727   25.92393   1.6968

Totals :                  3.94355e4  1998.65674
```

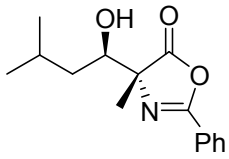

A (d)
8.00
J(7.36)
B (t)
7.59
J(7.43)
C (t)
7.49
J(7.60)
D (dd)
3.90
J(3.98, 10.51)
E (s)
2.20
F (m)
1.87
G (m)
1.63
H (s)
1.55
I (m)
1.24
J (dd)
0.94
J(6.63, 12.42)
1.86
0.95
1.99
1.00
0.85
1.02
1.95
2.98
1.29
5.97
f1 (ppm)
179.00
161.47
133.00
128.93
128.11
125.61
73.62
73.49
39.35
24.59
23.96
21.20
20.48

**8ca**


f1 (ppm)

**8ca**

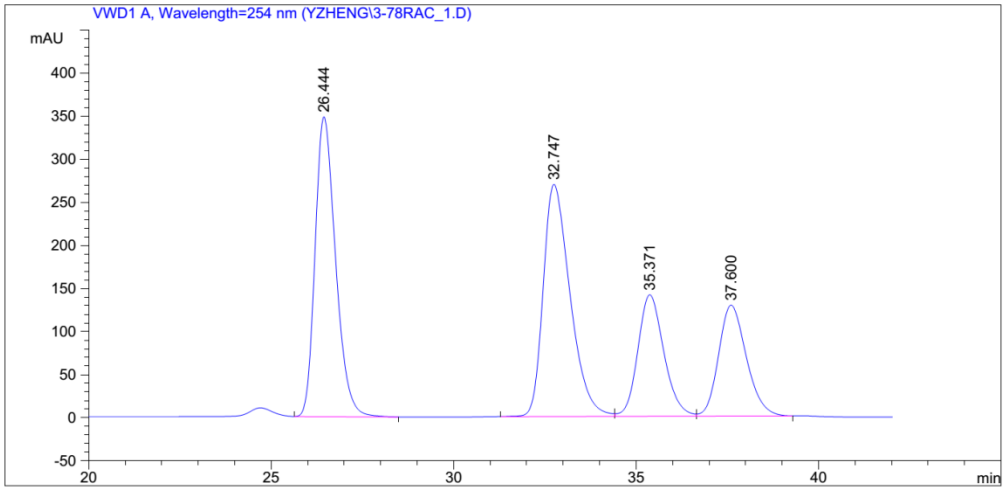
Data File C:\CHEM32\1\DATA\YZHENG\3-78RAC_1.D
Sample Name: 3-78rac




**8ca**

Racemic

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location : Vial 61
Injection Date  : 8/15/2011 10:48:35 PM
                                                  Inj Volume : 5 µl
Acq. Method     : C:\CHEM32\1\METHODS\METHOD1.M
Last changed    : 8/15/2011 10:29:10 PM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\METHOD1.M
Last changed    : 8/30/2011 2:14:12 PM by xiao
                  (modified after loading)
Sample Info     : AD-H, Hex:IPA = 99:1, 1mL/min, 254 nm, 45 bar, Left
```




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

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

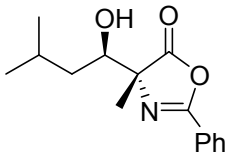

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  26.444 VB    0.6009 1.35843e4   348.07138  33.0314
   2  32.747 BV    0.7833 1.37832e4   269.39120  33.5150
   3  35.371 VV    0.7583 6964.91602  140.99208  16.9358
   4  37.600 VB    0.8142 6792.98926  128.61781  16.5177

Totals :                  4.11254e4   887.07248

==========================================================================
                        *** End of Report ***
```

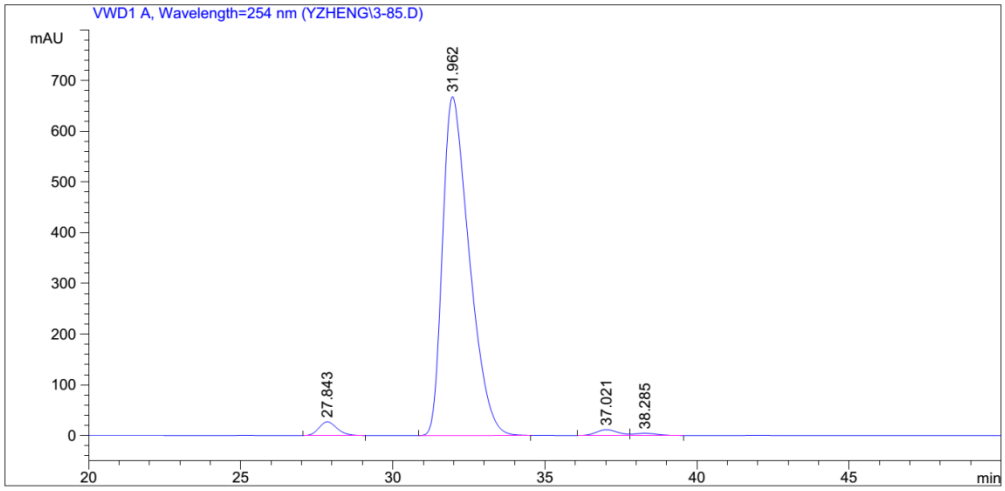
Data File C:\CHEM32\1\DATA\YZHENG\3-85.D
Sample Name: 3-85




**8ca**

95% ee, *anti*/*syn* = 98/2

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location : Vial 51
Injection Date  : 8/30/2011 11:48:31 AM
                                                  Inj Volume : 5 µl
Acq. Method     : C:\CHEM32\1\METHODS\METHOD1.M
Last changed    : 8/30/2011 11:00:10 AM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\METHOD1.M
Last changed    : 8/30/2011 2:13:00 PM by xiao
                  (modified after loading)
Sample Info     : AD-H, Hex:IPA = 99:1, 1 mL/min, 254 nm, 44 bar, Left
```




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

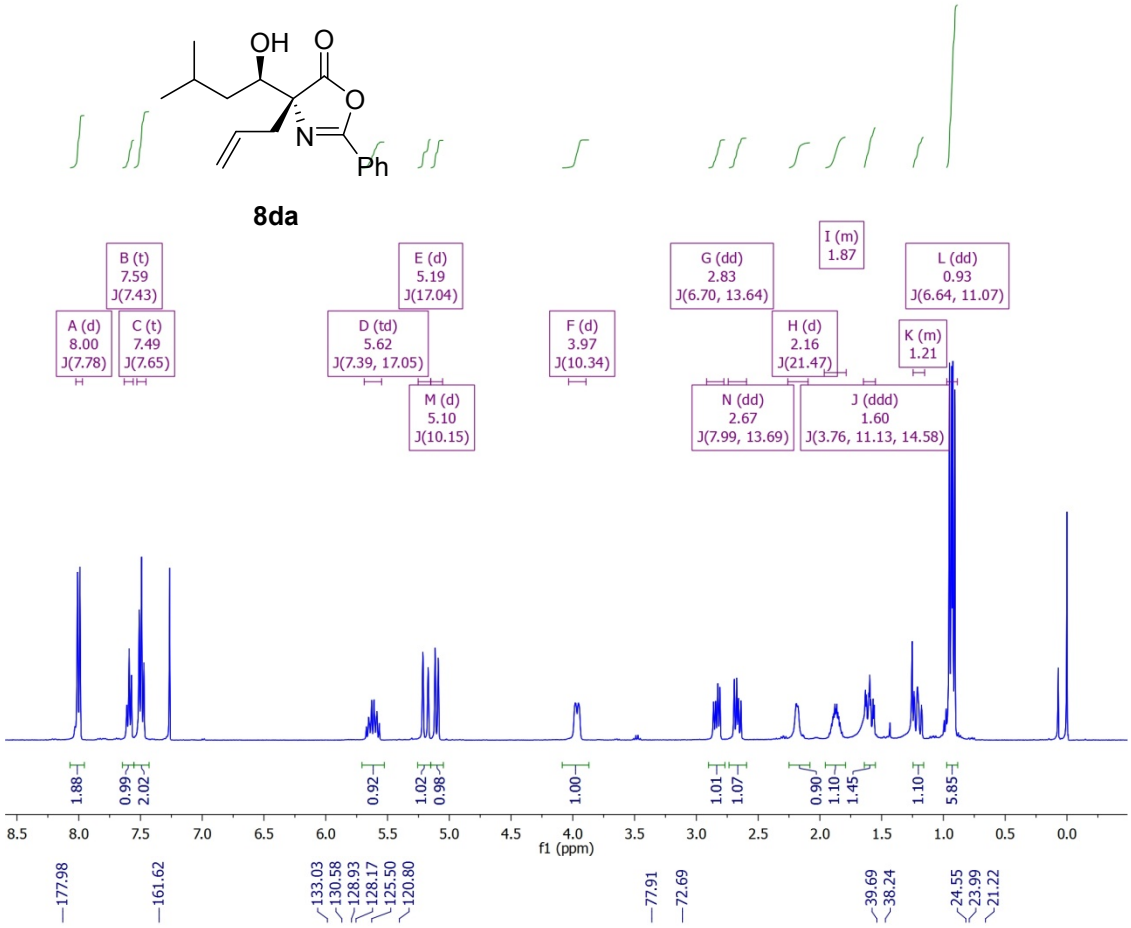
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

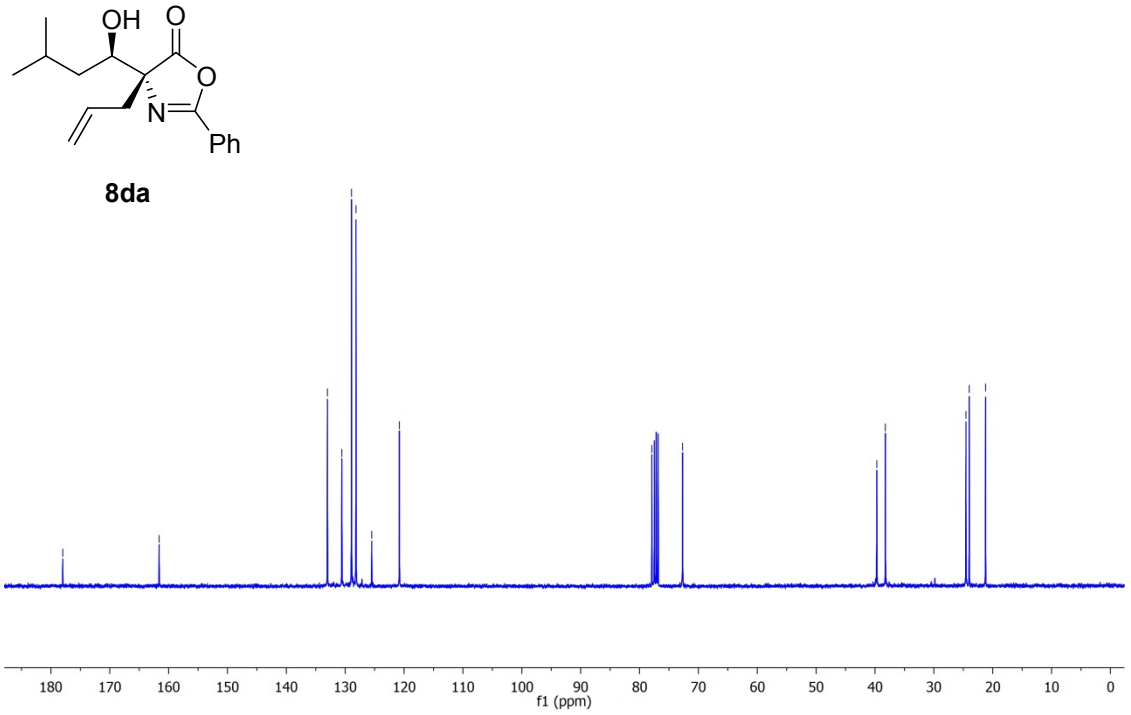
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  27.843 BB    0.6423 1125.41760   26.98795   2.6308
   2  31.962 BB    0.9270 4.08507e4   668.41473  95.4950
   3  37.021 BV    0.7711  567.44141   11.40712   1.3265
   4  38.285 VB    0.7853  234.28592    4.43397   0.5477

Totals :                  4.27778e4   711.24377

==========================================================================
                        *** End of Report ***
```

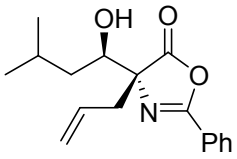

OH
O
O
N
Ph
A (d) 8.00 J(7.78)
B (t) 7.59 J(7.43)
C (t) 7.49 J(7.65)
D (td) 5.62 J(7.39, 17.05)
E (d) 5.19 J(17.04)
M (d) 5.10 J(10.15)
F (d) 3.97 J(10.34)
G (dd) 2.83 J(6.70, 13.64)
H (d) 2.16 J(21.47)
I (m) 1.87
N (dd) 2.67 J(7.99, 13.69)
J (ddd) 1.60 J(3.76, 11.13, 14.58)
K (m) 1.21
L (dd) 0.93 J(6.64, 11.07)
1.88
0.99
2.02
0.92
1.02
0.98
1.00
1.01
1.07
0.90
1.10
1.45
1.10
5.85
f1 (ppm)
177.98
161.62
133.03
130.58
128.93
128.17
125.50
120.80
77.91
72.69
39.69
38.24
24.55
23.99
21.22

**8da**


OH
O
O
N
Ph
f1 (ppm)

**8da**

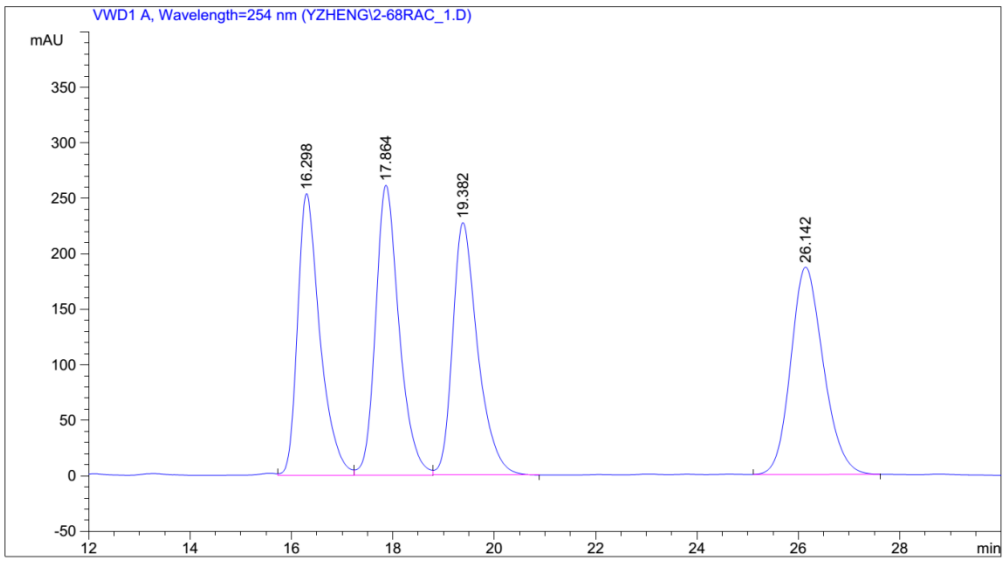
Data File C:\CHEM32\1\DATA\YZHENG\2-68RAC_1.D
Sample Name: 2-68rac




**8da**

Racemic

```
==================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 1/13/2011 11:35:19 AM
Acq. Method     : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 1/13/2011 10:39:23 AM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\WASH.M
Last changed    : 8/29/2011 10:29:09 AM by yang
                  (modified after loading)
Sample Info     : AD-H, Hex:IPA = 96:4, 1 mL/min, 254 nm, 40 bar, Right
```




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

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

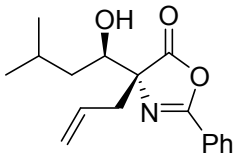

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  16.298 VV    0.4573 7755.66309  253.59787  23.9816
   2  17.864 VV    0.4866 8413.77637  261.07626  26.0166
   3  19.382 VB    0.5166 7826.41553  227.39288  24.2004
   4  26.142 BB    0.6843 8344.20117  186.77571  25.8014

Totals :                  3.23401e4   928.84273

==================================================================
                        *** End of Report ***
```

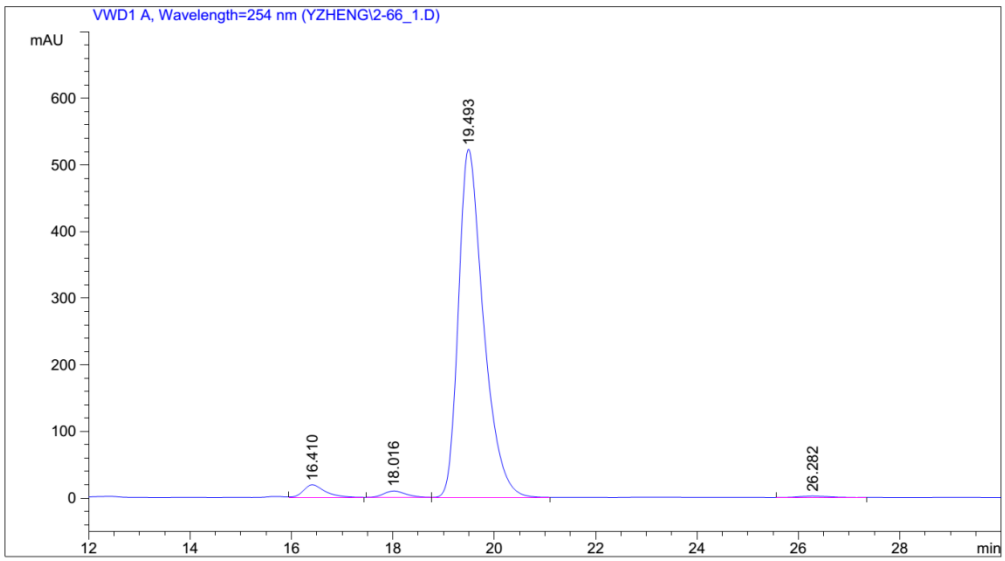
Data File C:\CHEM32\1\DATA\YZHENG\2-66_1.D
Sample Name: 2-66




**8da**

94% ee, *anti*/*syn* = 98/2

```
================================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 1/13/2011 12:11:33 PM
Acq. Method     : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 1/13/2011 12:12:37 PM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\WASH.M
Last changed    : 8/29/2011 10:28:01 AM by yang
                  (modified after loading)
Sample Info     : AD-H, Hex:IPA = 96:4, 1 mL/min, 254 nm, 40 bar, Right
```




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

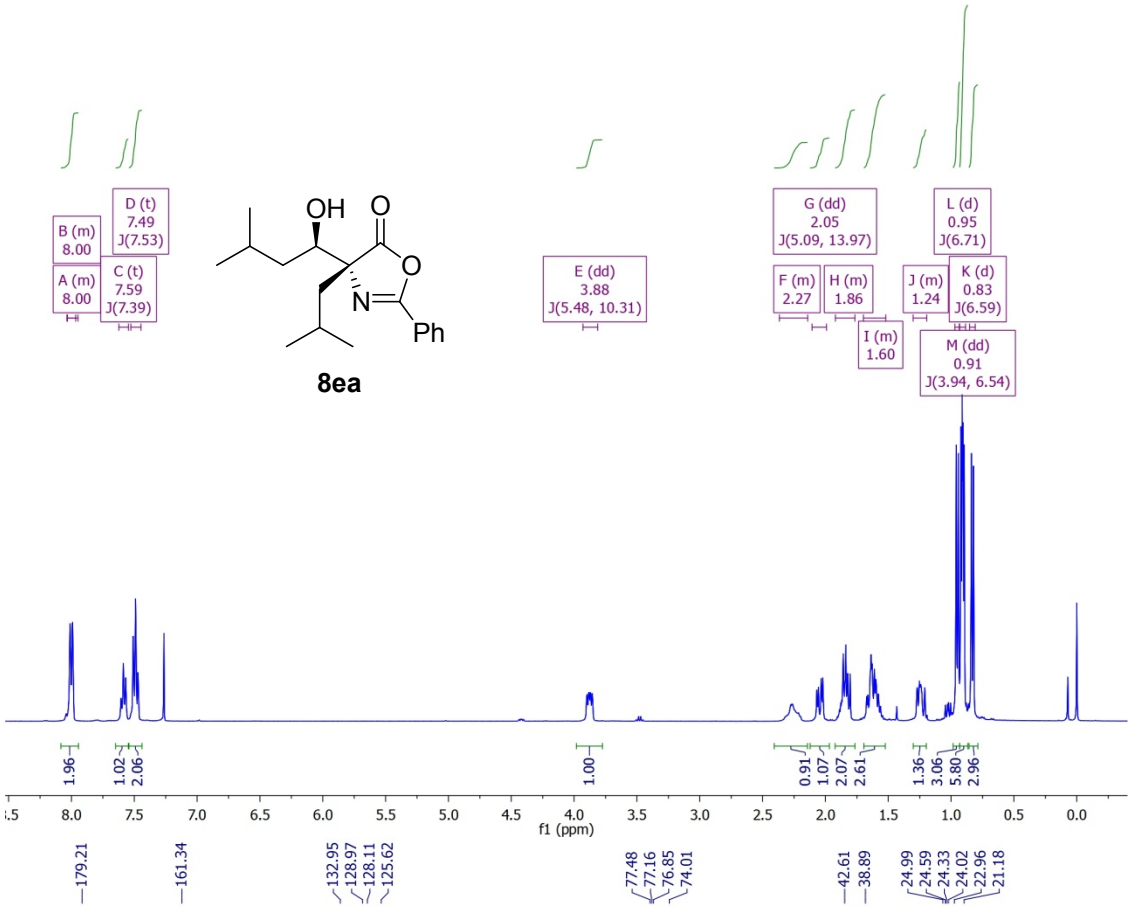
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

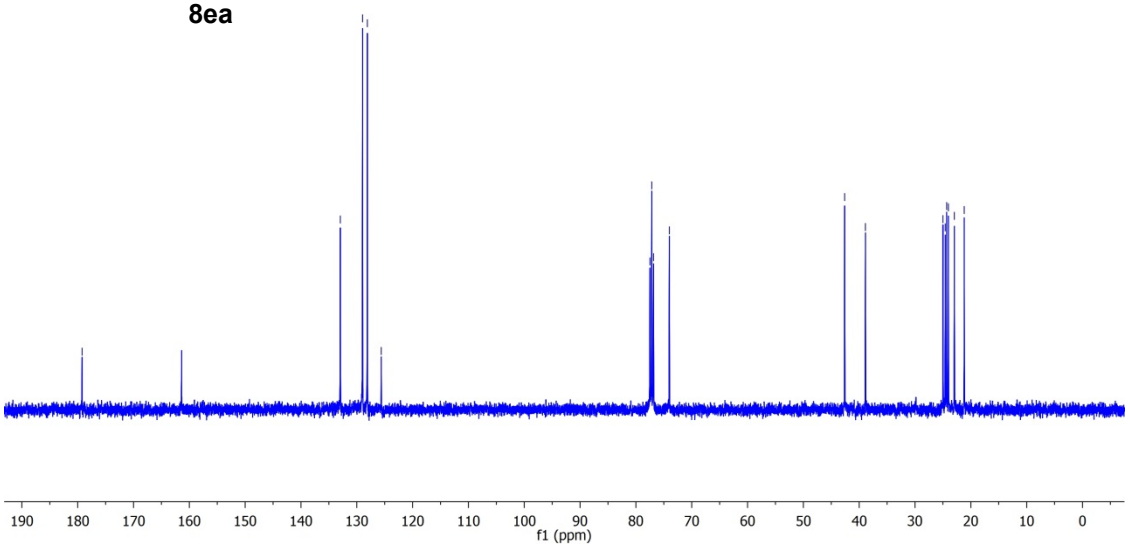
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  16.410 VB    0.4480  578.97461   19.11654   3.0657
   2  18.016 BV    0.4745  304.32053    9.79644   1.6114
   3  19.493 VB    0.5146 1.79064e4   522.82092  94.8149
   4  26.282 BB    0.6333   95.94662    2.20955   0.5080

Totals :                  1.88856e4   553.94345

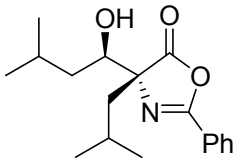
================================================================================
                          *** End of Report ***
```


B (m)
8.00
A (m)
8.00
D (t)
7.49
J(7.53)
C (t)
7.59
J(7.39)
OH
O
O
N
Ph
E (dd)
3.88
J(5.48, 10.31)
G (dd)
2.05
J(5.09, 13.97)
F (m)
2.27
H (m)
1.86
I (m)
1.60
J (m)
1.24
L (d)
0.95
J(6.71)
K (d)
0.83
J(6.59)
M (dd)
0.91
J(3.94, 6.54)
1.96
1.02
2.06
1.00
0.91
1.07
2.07
2.61
1.36
3.06
5.80
2.96
f1 (ppm)
179.21
161.34
132.95
128.97
128.11
125.62
77.48
77.16
76.85
74.01
42.61
38.89
24.99
24.59
24.33
24.02
22.96
21.18

**8ea**


f1 (ppm)

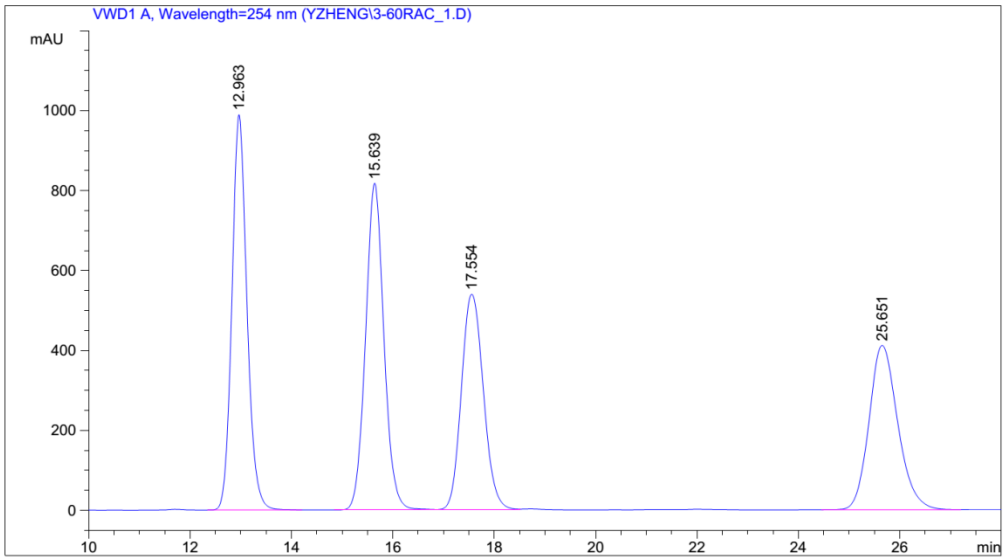
Data File C:\CHEM32\1\DATA\YZHENG\3-60RAC_1.D
Sample Name: 3-60rac




**8ea**

Racemic

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 7/28/2011 11:21:48 AM
Acq. Method     : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 7/28/2011 10:55:57 AM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 8/1/2011 12:58:37 PM by yang
                  (modified after loading)
Sample Info     : AD-H, Hex:IPA = 96:4, 1.0 mL/min, 254 nm, 39 bar, Right
```




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

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

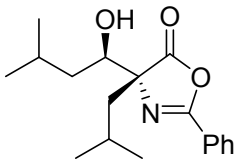

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  12.963 BB    0.3132 2.03779e4   989.14178  27.7510
   2  15.639 BV    0.3821 2.05489e4   817.36218  27.9840
   3  17.554 VV    0.4744 1.62889e4   539.76801  22.1826
   4  25.651 BB    0.5942 1.62153e4   411.04782  22.0823

Totals :                  7.34310e4  2757.31979

==========================================================================
                          *** End of Report ***
```

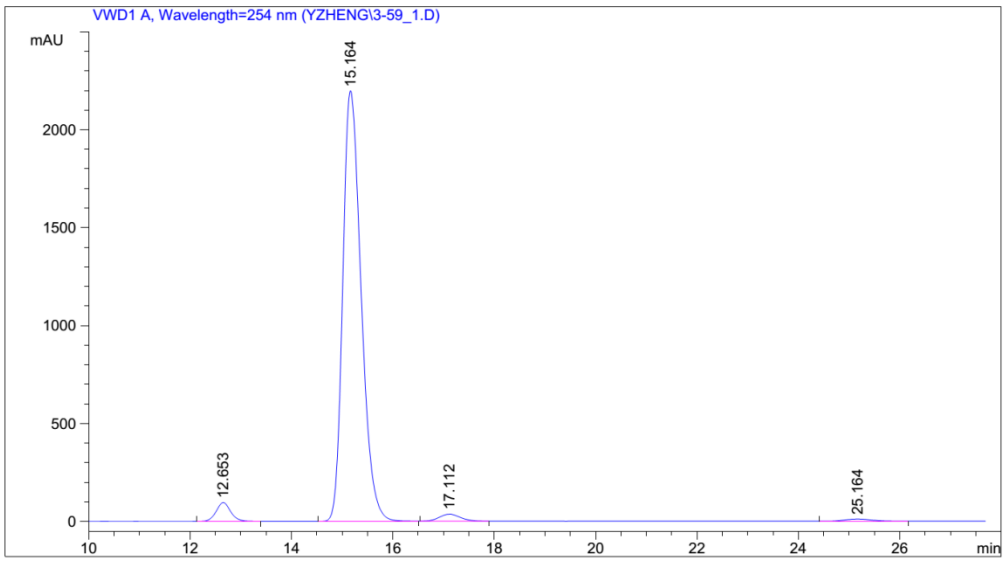
Data File C:\CHEM32\1\DATA\YZHENG\3-59_1.D
Sample Name: 3-59




**8ea**

93% ee, *anti*/*syn* = 97.5/2.5

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 7/29/2011 12:00:11 AM
Acq. Method     : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 7/28/2011 11:37:54 PM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 8/1/2011 12:56:49 PM by yang
                  (modified after loading)
Sample Info     : AD-H, Hex:IPA = 96:4, 1.0 mL/min, 254 nm, 39 bar, Right
```




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

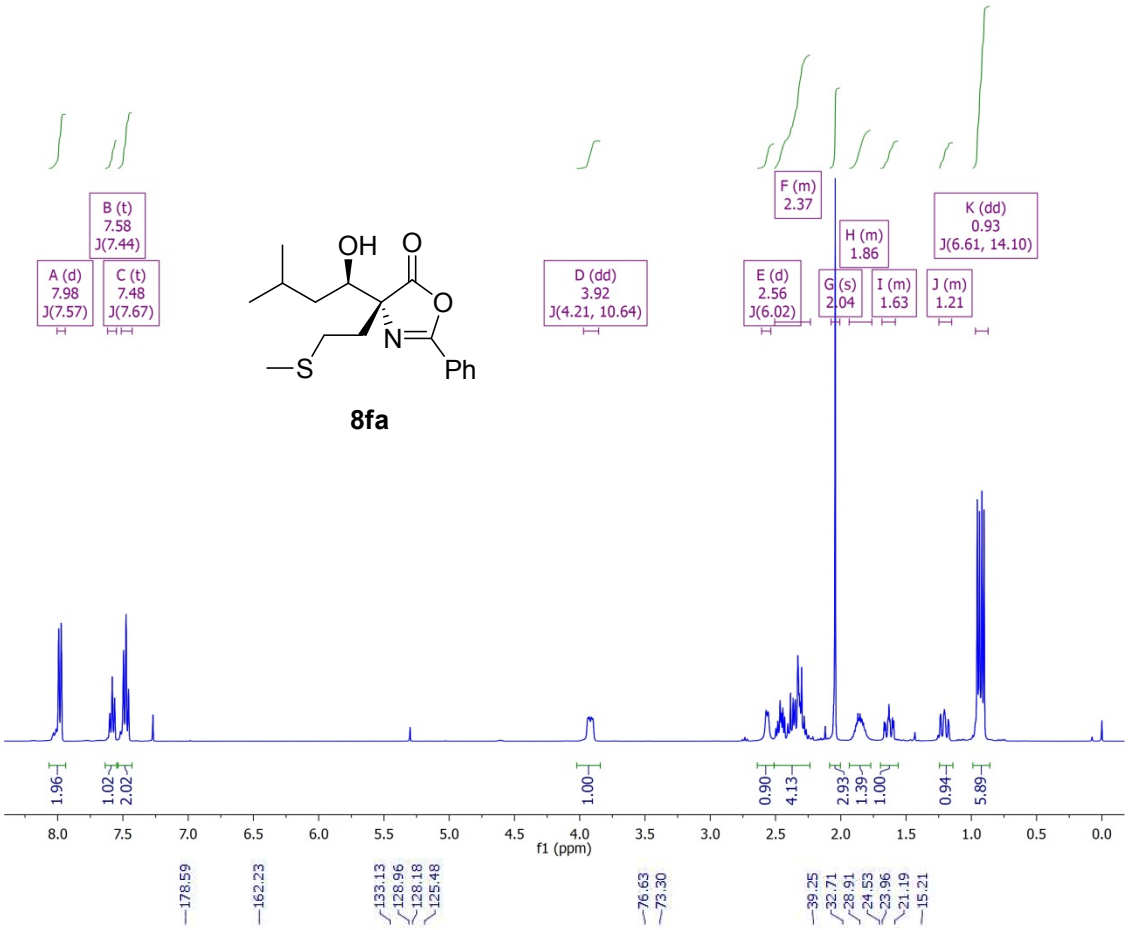
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

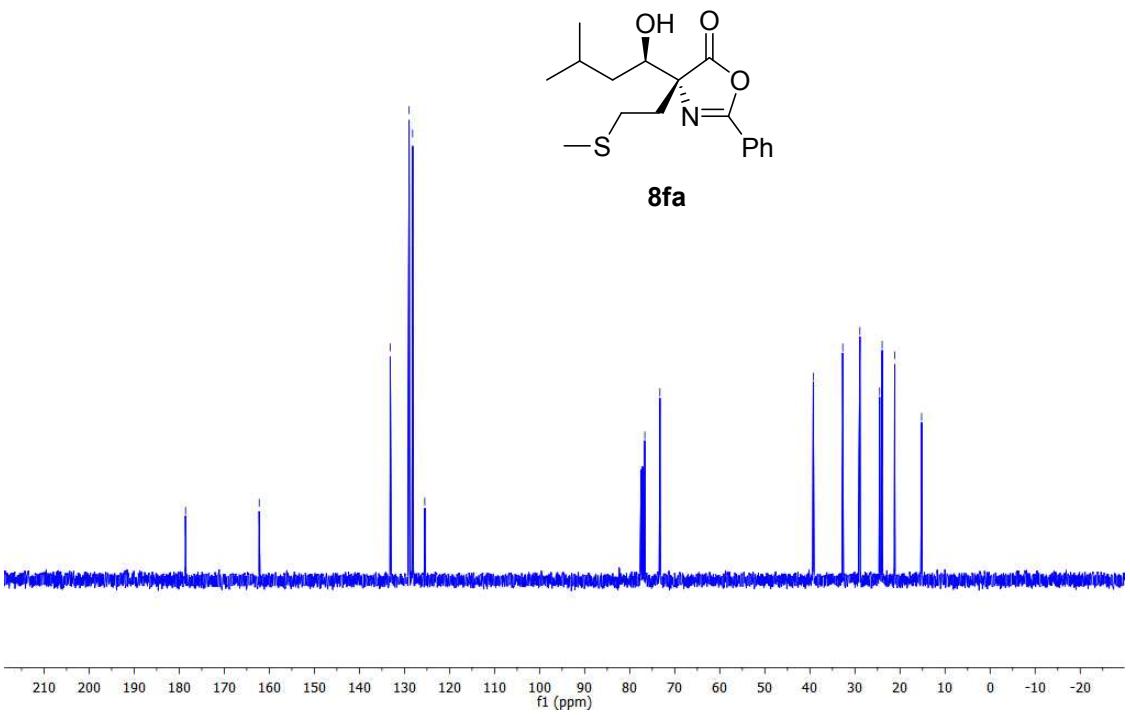
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  12.653 BB    0.3011 1925.33533   96.53149   3.2858
   2  15.164 BB    0.3891 5.51910e4  2197.30835  94.1898
   3  17.112 BB    0.4435 1045.66577   36.33644   1.7845
   4  25.164 BB    0.5818  433.50134   11.44081   0.7398

Totals :                  5.85955e4  2341.61710


==========================================================================
                        *** End of Report ***
```

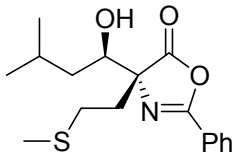

A (d)
7.98
J(7.57)
B (t)
7.58
J(7.44)
C (t)
7.48
J(7.67)
D (dd)
3.92
J(4.21, 10.64)
E (d)
2.56
J(6.02)
F (m)
2.37
G (s)
2.04
H (m)
1.86
I (m)
1.63
J (m)
1.21
K (dd)
0.93
J(6.61, 14.10)
OH
O
O
N
S
Ph
1.96
1.02
2.02
1.00
0.90
4.13
2.93
1.39
1.00
0.94
5.89
f1 (ppm)

**8fa**


178.59
162.23
133.13
128.96
128.18
125.48
76.63
73.30
39.25
32.71
28.91
24.53
23.96
21.19
15.21
f1 (ppm)

**8fa**

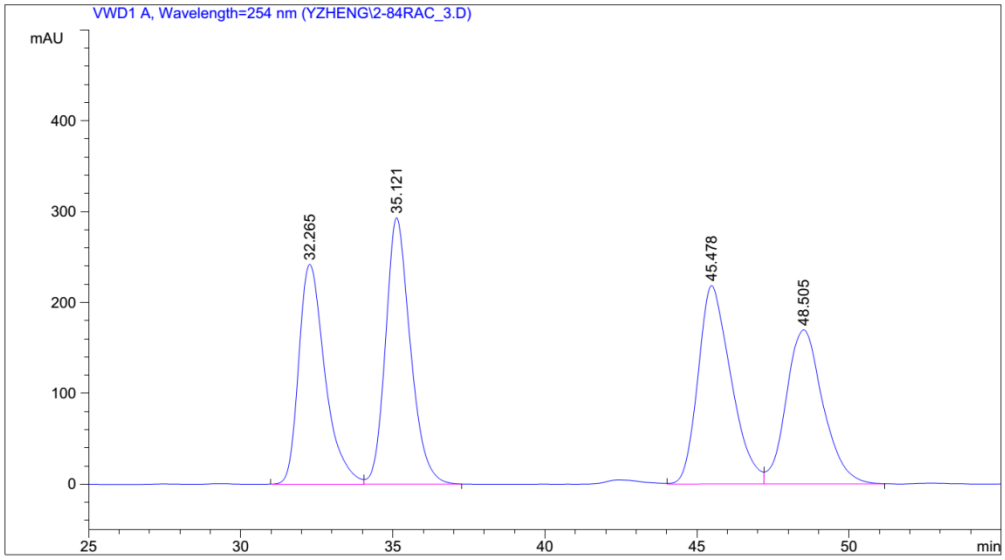
Data File C:\CHEM32\1\DATA\YZHENG\2-84RAC_3.D
Sample Name: 2-84rac




**8fa**

Racemic

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 1/27/2011 6:18:51 PM
Acq. Method     : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 1/27/2011 6:08:05 PM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\WASH.M
Last changed    : 8/31/2011 11:14:37 AM by yang
                  (modified after loading)
Sample Info     : AD-H, Hex:IPA = 96.5:3.5, 1 mL/min, 254 nm, 39 bar, Rig
                  ht
```




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

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

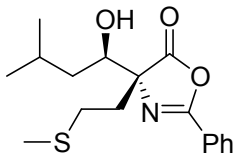

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  32.265 BV    0.9091 1.45334e4   241.92194  23.5229
   2  35.121 VB    0.8421 1.65952e4   293.30771  26.8599
   3  45.478 VV    1.0786 1.64092e4   218.47810  26.5589
   4  48.505 VB    1.2715 1.42463e4   169.54929  23.0582

Totals :                  6.17841e4   923.25703

==========================================================================
                          *** End of Report ***
```

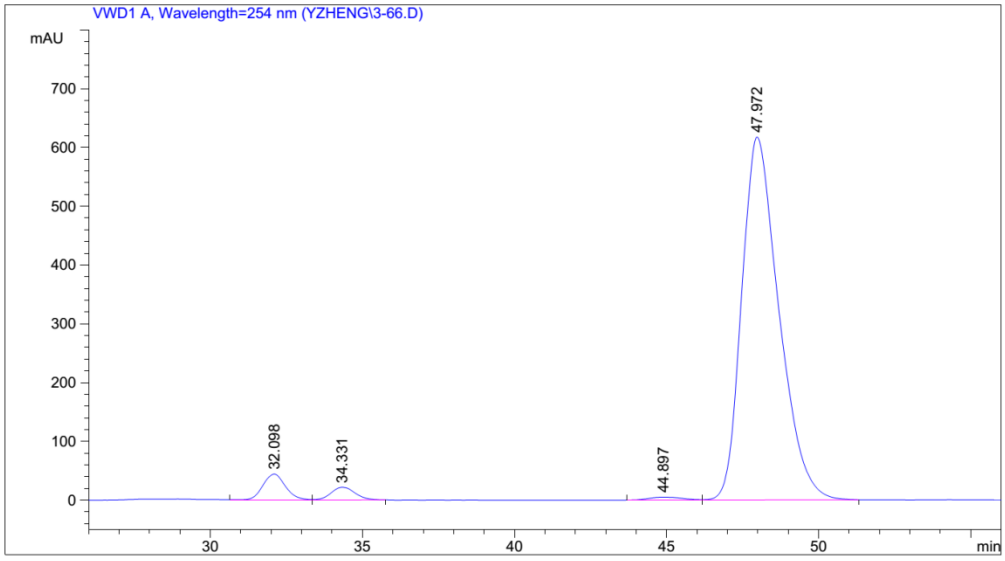
Data File C:\CHEM32\1\DATA\YZHENG\3-66.D
Sample Name: 3-66




**8fa**

92% ee, *anti*/*syn* = 97/3

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 8/15/2011 12:54:41 PM
Acq. Method     : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 8/15/2011 11:01:25 AM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\WASH.M
Last changed    : 8/15/2011 5:03:25 PM by yang
                  (modified after loading)
Sample Info     : AD-H, Hex:IPA = 96.5:3.5, 1 mL/min, 254 nm, 40 bar, Rig
                  ht
```




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

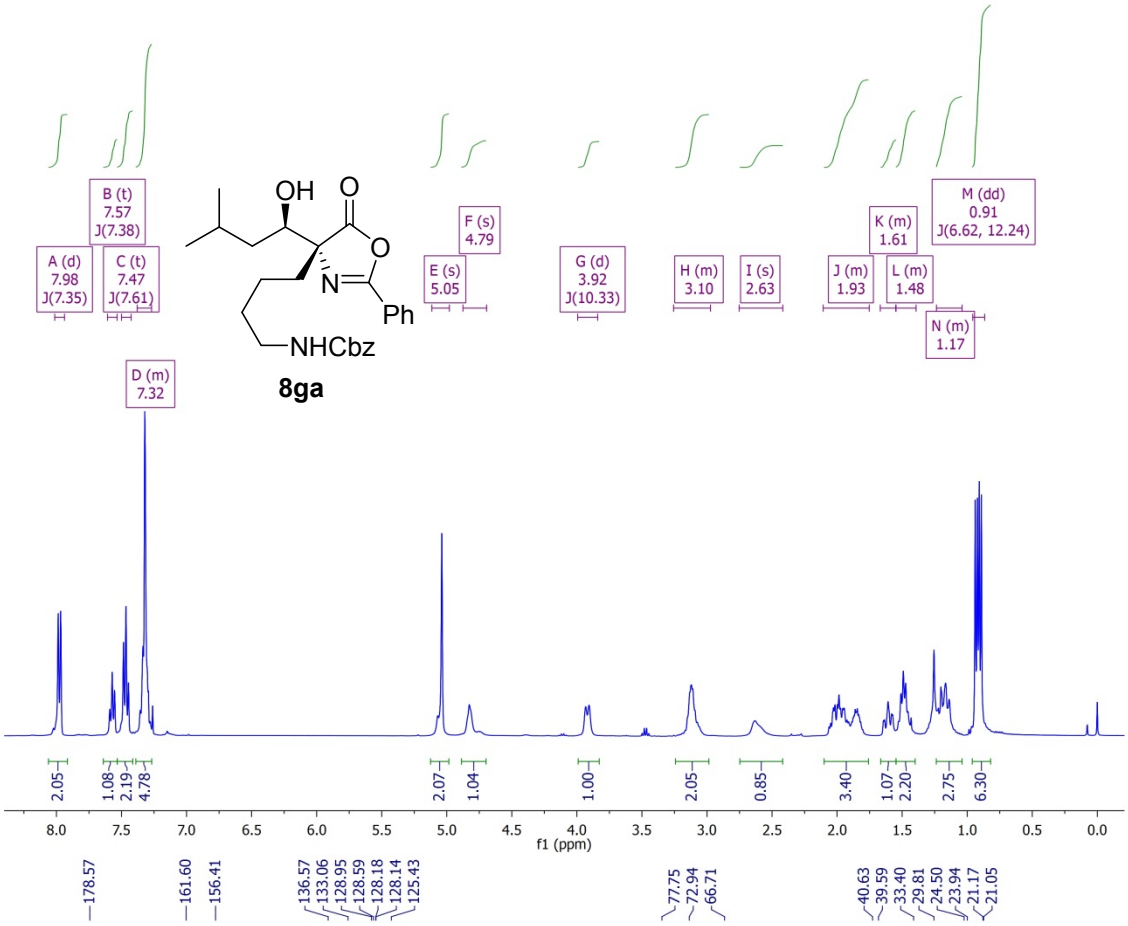
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

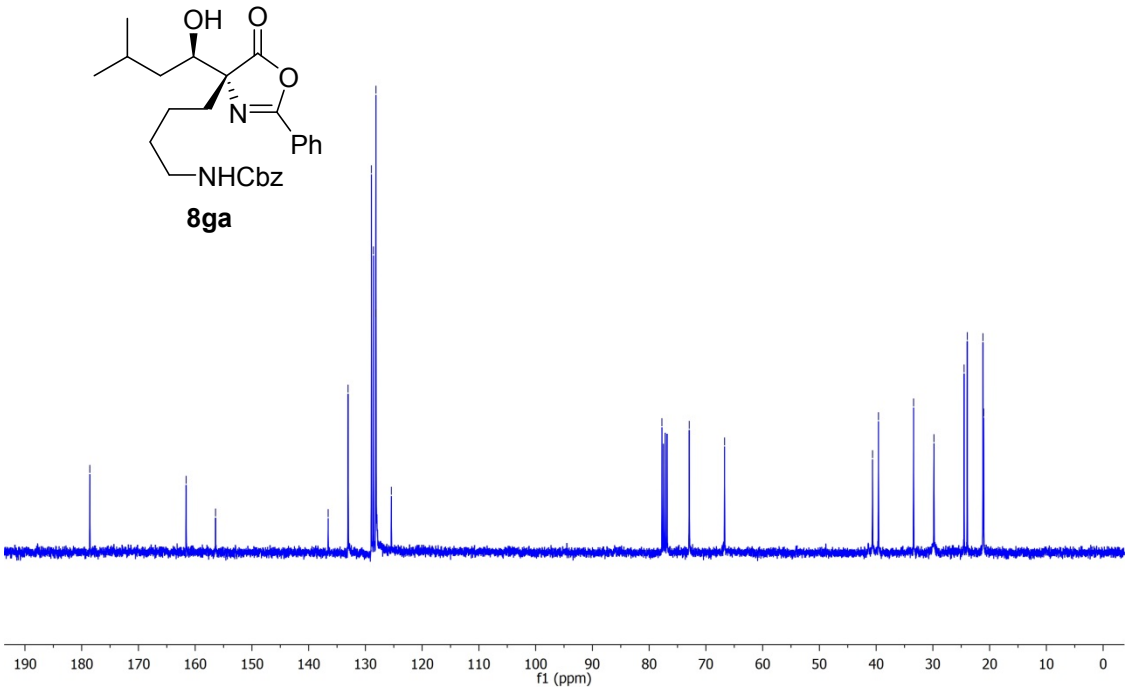
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  32.098 VV    0.8085 2286.37842   44.11148   4.0707
   2  34.331 VB    0.8557 1195.80103   21.49694   2.1290
   3  44.897 BV    0.9209  370.31412    4.92522   0.6593
   4  47.972 VB    1.2040 5.23148e4   617.45117  93.1410

Totals :                  5.61673e4   687.98482

==========================================================================
                        *** End of Report ***
```

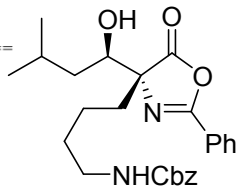

A (d) 7.98 J(7.35)
B (t) 7.57 J(7.38)
C (t) 7.47 J(7.61)
D (m) 7.32
E (s) 5.05
F (s) 4.79
G (d) 3.92 J(10.33)
H (m) 3.10
I (s) 2.63
J (m) 1.93
K (m) 1.61
L (m) 1.48
M (dd) 0.91 J(6.62, 12.24)
N (m) 1.17
OH
O
N
Ph
NHCbz
2.05
1.08
2.19
4.78
2.07
1.04
1.00
2.05
0.85
3.40
1.07
2.20
2.75
6.30
f1 (ppm)
178.57
161.60
156.41
136.57
133.06
128.95
128.59
128.18
128.14
125.43
77.75
72.94
66.71
40.63
39.59
33.40
29.81
24.50
23.94
21.17
21.05

**8ga**


OH
O
N
Ph
NHCbz
f1 (ppm)

**8ga**

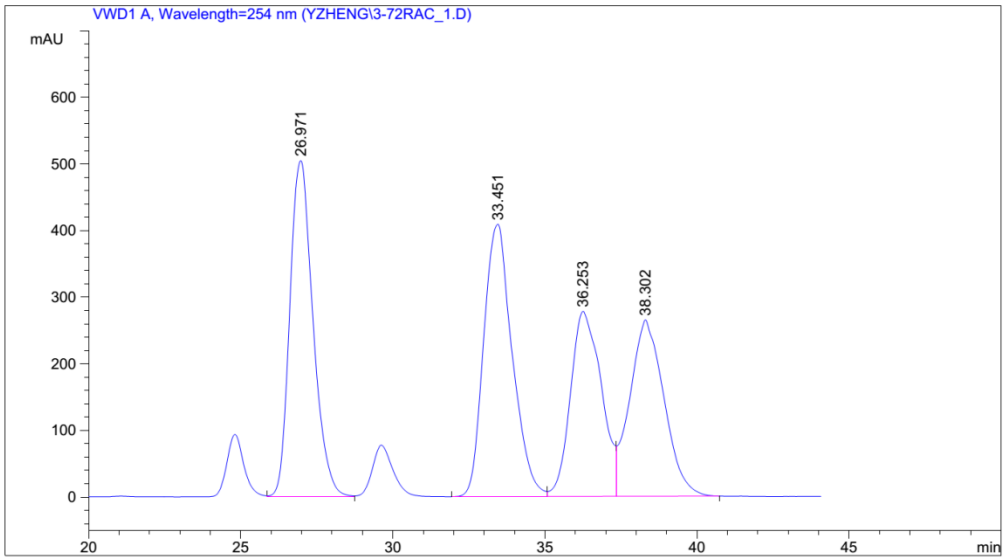
Data File C:\CHEM32\1\DATA\YZHENG\3-72RAC_1.D
Sample Name: 2-72rac




**8ga**

Racemic

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 8/11/2011 3:44:19 PM
Acq. Method     : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 8/11/2011 2:59:20 PM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 8/11/2011 6:27:53 PM by yang
                  (modified after loading)
Sample Info     : AD-H, Hex:IPA = 87:13, 1 mL/min, 254 nm, 43 bar, Right
```




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

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

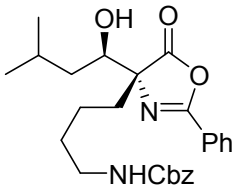

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  26.971 VV    0.7904 2.67048e4   504.80145  28.5004
   2  33.451 BV    1.0009 2.66542e4   408.83020  28.4464
   3  36.253 VV    0.9966 1.96091e4   277.86295  20.9276
   4  38.302 VB    1.0630 2.07317e4   264.91617  22.1257

Totals :                 9.36999e4  1456.41077


==========================================================================
                        *** End of Report ***
```

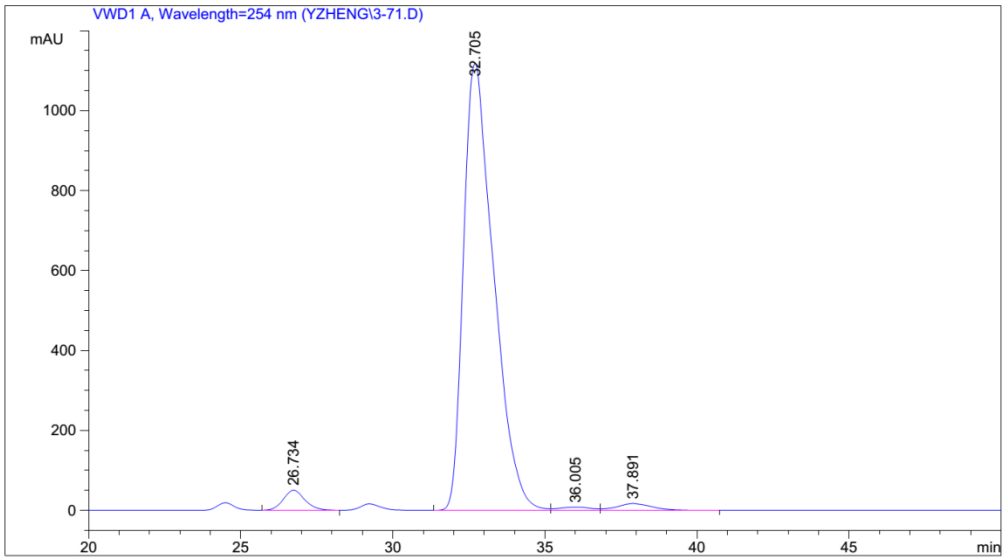
Data File C:\CHEM32\1\DATA\YZHENG\3-71.D
Sample Name: 3-71




**8ga**

94% ee, *anti*/*syn* = 97.5/2.5

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 8/11/2011 5:17:51 PM
Acq. Method     : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 8/11/2011 4:31:09 PM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 8/11/2011 6:25:54 PM by yang
                  (modified after loading)
Sample Info     : AD-H, Hex:IPA = 87:13, 1 mL/min, 254 nm, 43 bar, Right
```




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

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

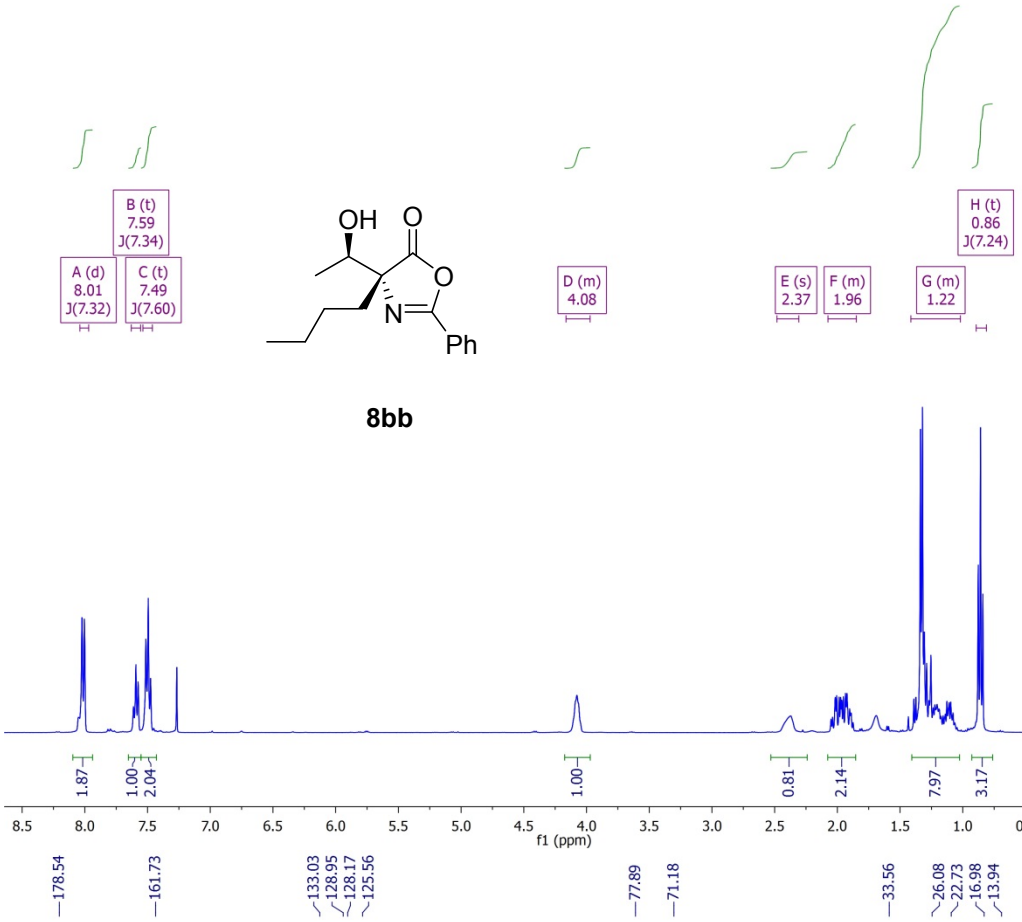

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  26.734 BB    0.7527 2456.97339   50.22921   3.0263
   2  32.705 BB    0.9954 7.68489e4  1118.29761  94.6549
   3  36.005 BV    0.9143  567.14624    7.90414   0.6986
   4  37.891 VB    0.9769 1315.50061   16.92087   1.6203

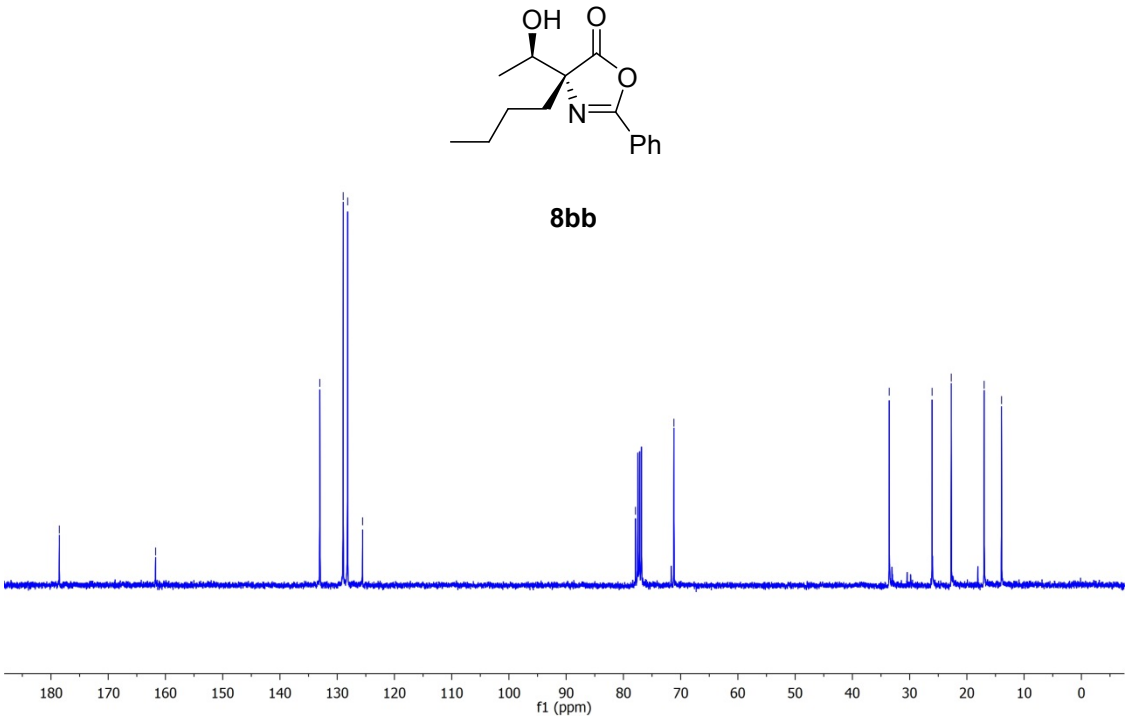
Totals :                 8.11885e4  1193.35183

==========================================================================
                        *** End of Report ***

Instrument 1 8/11/2011 6:26:12 PM yang                    Page   1 of 1
```

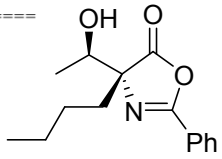

A (d)
8.01
J(7.32)
B (t)
7.59
J(7.34)
C (t)
7.49
J(7.60)
D (m)
4.08
E (s)
2.37
F (m)
1.96
G (m)
1.22
H (t)
0.86
J(7.24)
OH
O
O
N
Ph
1.87
1.00
2.04
1.00
0.81
2.14
7.97
3.17
f1 (ppm)
178.54
161.73
133.03
128.95
128.17
125.56
77.89
71.18
33.56
26.08
22.73
16.98
13.94

**8bb**


OH
O
O
N
Ph
f1 (ppm)

**8bb**

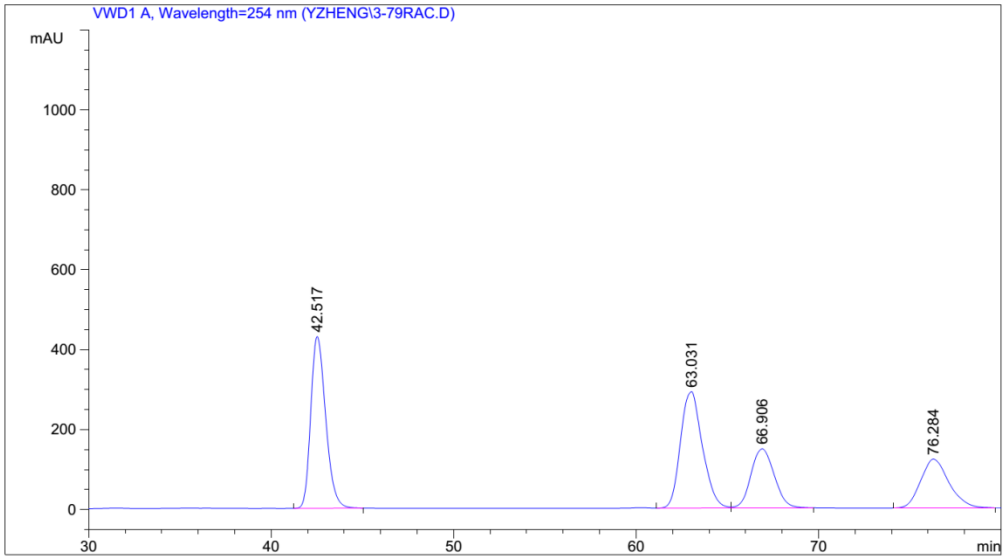
Data File C:\CHEM32\1\DATA\YZHENG\3-79RAC.D
Sample Name: 3-79rac




**8bb**

Racemic

```
==================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 8/16/2011 5:35:41 PM
Acq. Method     : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 8/16/2011 5:15:02 PM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\WASH.M
Last changed    : 8/16/2011 10:22:15 PM by yang
                  (modified after loading)
Sample Info     : OJ-H+OJ, Hex:IPA = 96:4, 0.8 mL/min, 254 nm, 51 bar, Ri
                  ght
```




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

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


Signal 1: VWD1 A, Wavelength=254 nm

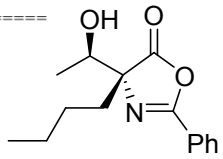
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  42.517 BB    0.8859 2.44250e4   429.74283  32.5804
   2  63.031 VV    1.2815 2.43056e4   291.49854  32.4211
   3  66.906 VB    1.4139 1.31029e4   148.06441  17.4778
   4  76.284 BB    1.6199 1.31349e4   122.15461  17.5206

Totals :                 7.49684e4   991.46038

==================================================================
                        *** End of Report ***
```

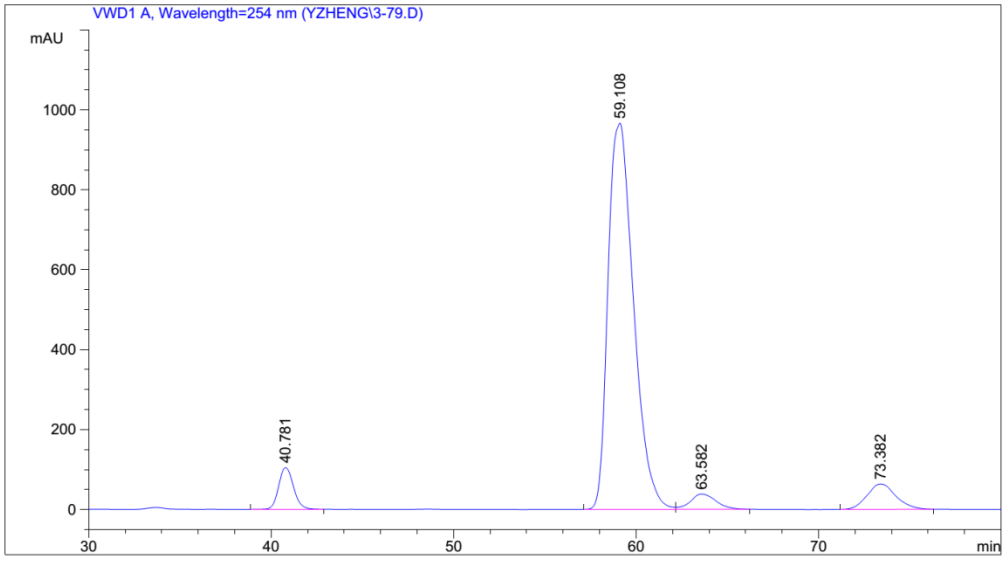
```
Data File C:\CHEM32\1\DATA\YZHENG\3-79.D
 Sample Name: 3-79

    ========================================================================
    Acq. Operator    : yang
    Acq. Instrument  : Instrument 1                    Location :   -
    Injection Date   : 8/16/2011 8:43:08 PM
    Acq. Method      : C:\CHEM32\1\METHODS\METHOD-1_LC.M
    Last changed     : 8/16/2011 5:15:02 PM by yang
                       (modified after loading)
    Analysis Method  : C:\CHEM32\1\METHODS\WASH.M
    Last changed     : 8/16/2011 10:21:37 PM by yang
                       (modified after loading)
    Sample Info      : OJ-H+OJ, Hex:IPA = 96:4, 0.8 mL/min, 254 nm, 52 bar, Ri
                       ght
```




**8bb**

88% ee, *anti*/*syn* = 90.5/9.5




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

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

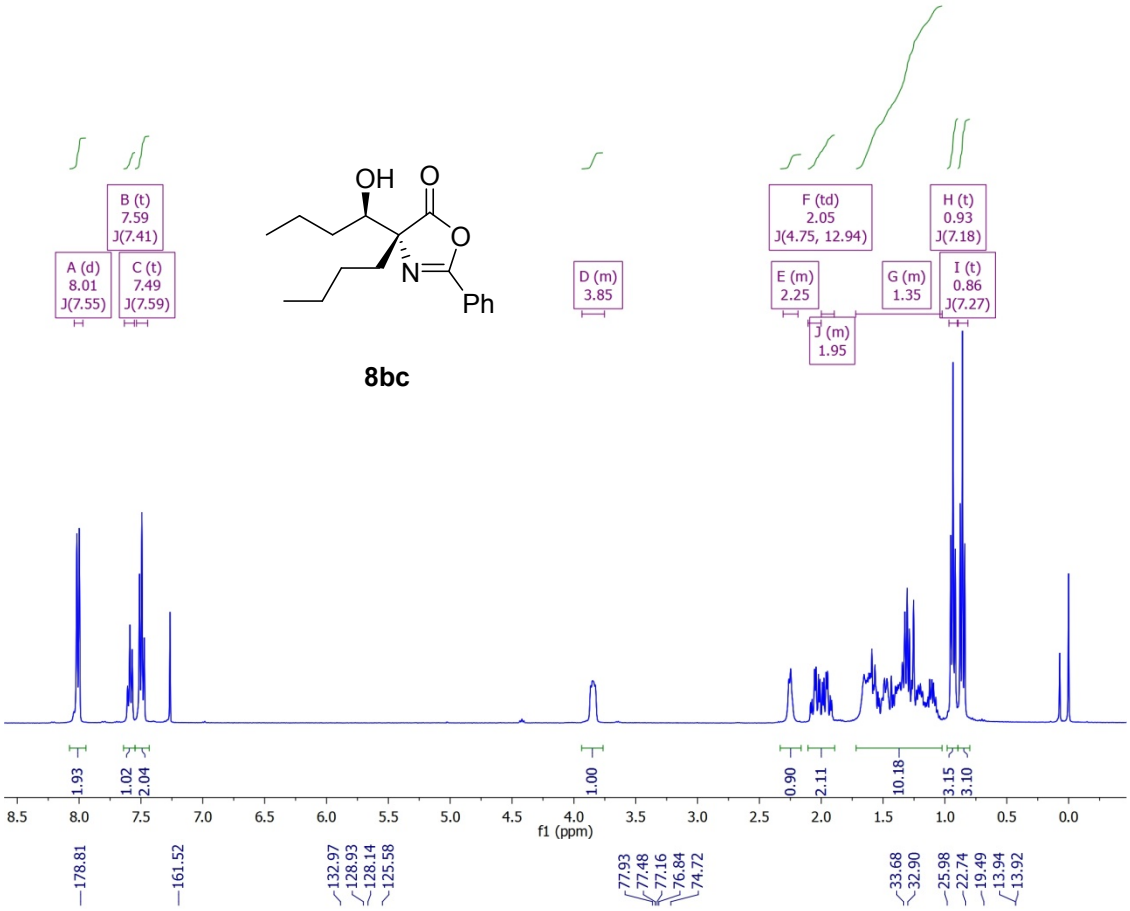

    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  40.781 BB    0.8862 5945.25244  104.31563   5.3903
       2  59.108 BB    1.3108 9.36414e4   966.55273  84.9013
       3  63.582 BB    1.3658 3728.47119   38.25199   3.3805
       4  73.382 BB    1.6048 6979.34521   63.24734   6.3279

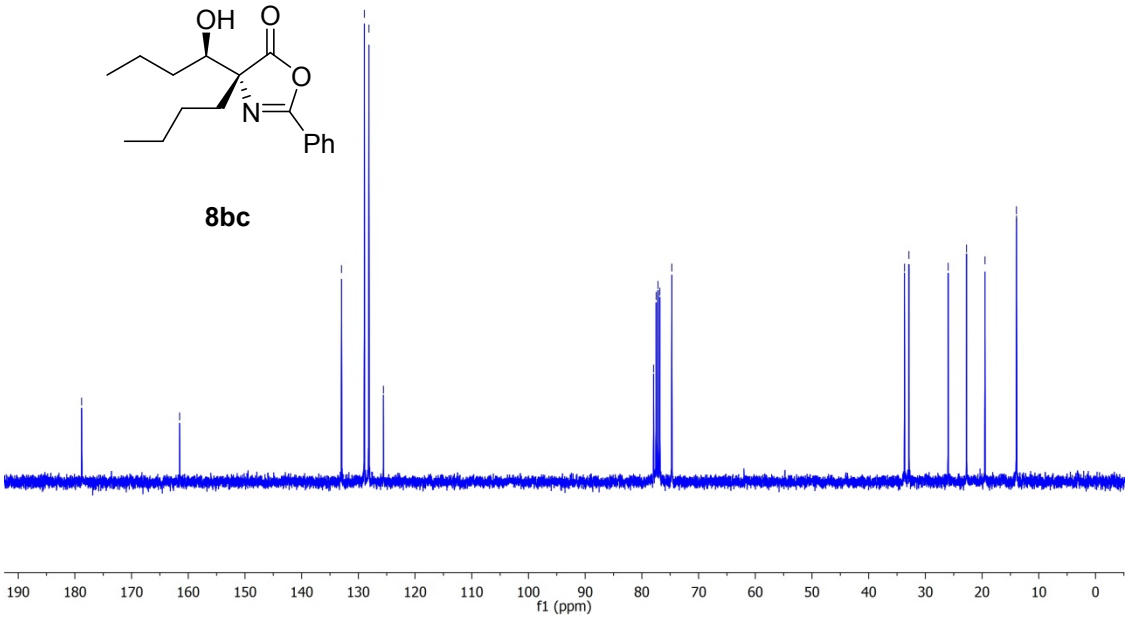
    Totals :                  1.10294e5  1172.36769

    ========================================================================
                              *** End of Report ***

Instrument 1 8/16/2011 10:21:50 PM yang                        Page   1 of 1
```

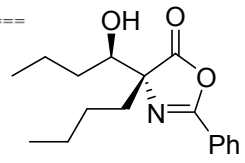

A (d)
8.01
J(7.55)
B (t)
7.59
J(7.41)
C (t)
7.49
J(7.59)
D (m)
3.85
E (m)
2.25
F (td)
2.05
J(4.75, 12.94)
G (m)
1.35
H (t)
0.93
J(7.18)
I (t)
0.86
J(7.27)
J (m)
1.95
OH
O
O
N
Ph
1.93
1.02
2.04
1.00
0.90
2.11
10.18
3.15
3.10
f1 (ppm)
178.81
161.52
132.97
128.93
128.14
125.58
77.93
77.48
77.16
76.84
74.72
33.68
32.90
25.98
22.74
19.49
13.94
13.92

**8bc**


OH
O
O
N
Ph
f1 (ppm)

**8bc**

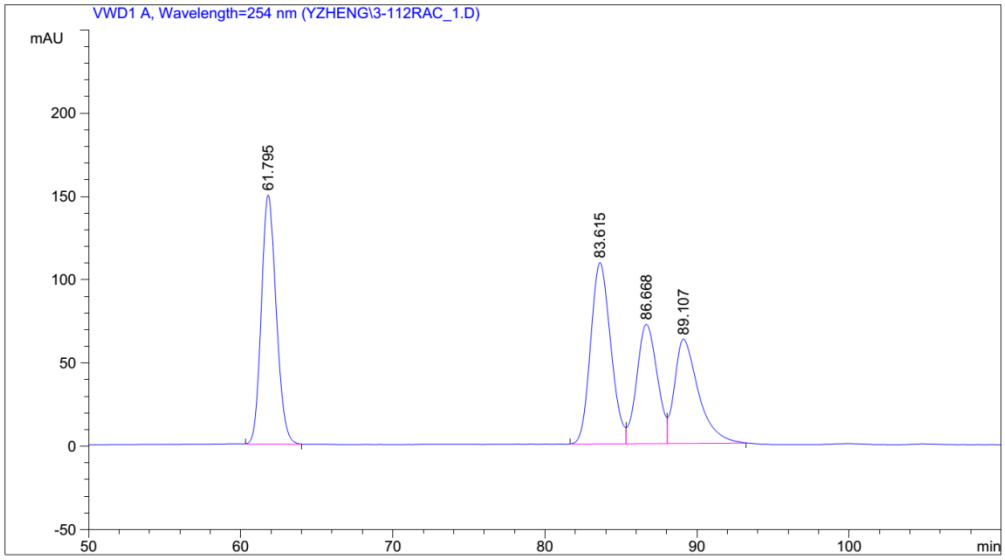
Data File C:\CHEM32\1\DATA\YZHENG\3-112RAC_1.D
Sample Name: 3-22




**8bc**

Racemic

```
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 10/3/2011 8:02:06 PM
Acq. Method     : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 10/3/2011 7:27:20 PM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 10/4/2011 12:55:14 PM by yang
                  (modified after loading)
Sample Info     : AD-H+AD, Hex:IPA = 97.2:2.8, 0.8 mL/min, 254 nm, 43 bar
                  , Right
```




```
                          Area Percent Report

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

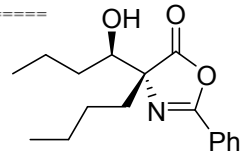

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  61.795 BB    1.0586 1.01515e4   149.51230  30.1216
   2  83.615 BV    1.4019 9997.66309  108.86904  29.6651
   3  86.668 VV    1.4206 6694.03369   71.64605  19.8626
   4  89.107 VB    1.5286 6858.53320   62.67318  20.3507

Totals :                  3.37017e4   392.70057


                          *** End of Report ***
```

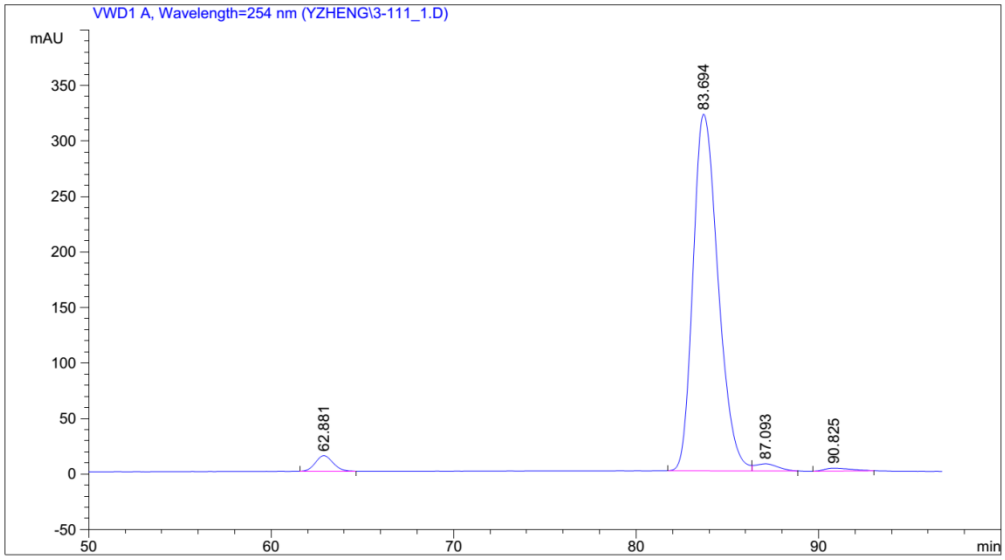
Data File C:\CHEM32\1\DATA\YZHENG\3-111_1.D
Sample Name: 3-111




```
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 10/4/2011 1:48:56 PM
Acq. Method     : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 10/4/2011 1:08:48 PM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 10/4/2011 3:52:38 PM by yang
                  (modified after loading)
Sample Info     : AD-H+AD, Hex:IPA = 97.2:2.8, 0.8 mL/min, 254 nm, 43 bar
                  , Right
```

**8bc**

94% ee, *anti*/*syn* = 97.5/2.5




Area Percent Report

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


Signal 1: VWD1 A, Wavelength=254 nm

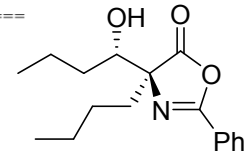
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  62.881 BB    1.0241  947.42053   14.12772   2.9253
   2  83.694 BB    1.4483 3.06197e4   321.25366  94.5426
   3  87.093 BB    1.0888  557.07318    6.46186   1.7200
   4  90.825 BB    1.1914  263.00912    2.59781   0.8121

Totals :                  3.23872e4   344.44105
```

*** End of Report ***

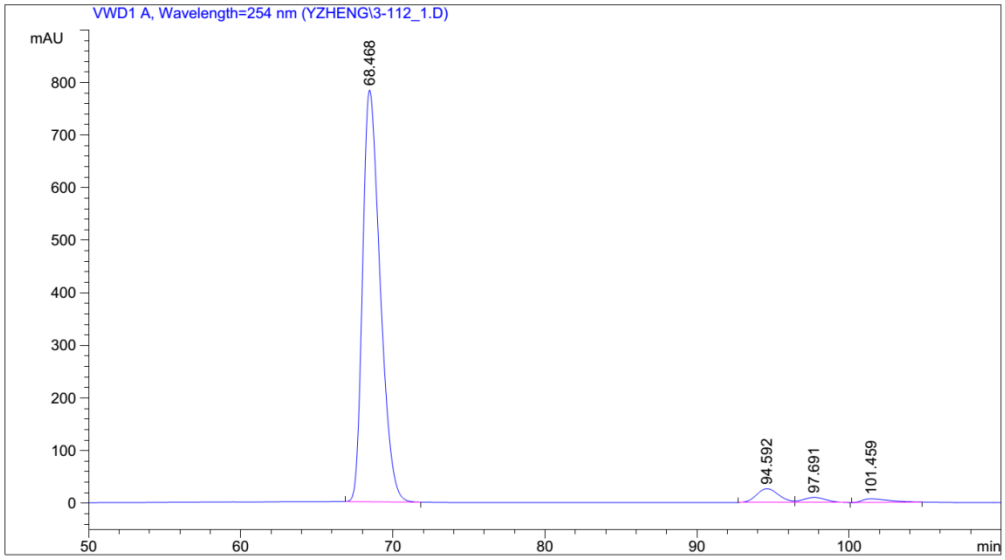
Data File C:\CHEM32\1\DATA\YZHENG\3-112_1.D
Sample Name: 3-22

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 10/3/2011 5:01:00 PM
Acq. Method     : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 10/3/2011 4:37:38 PM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 10/4/2011 12:48:26 PM by yang
                  (modified after loading)
Sample Info     : AD-H+AD, Hex:IPA = 97:3, 0.8 mL/min, 254 nm, 43 bar, Ri
                  ght
```




***ent*-8bc**

-92% ee, *anti*/*syn* = 97/3




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

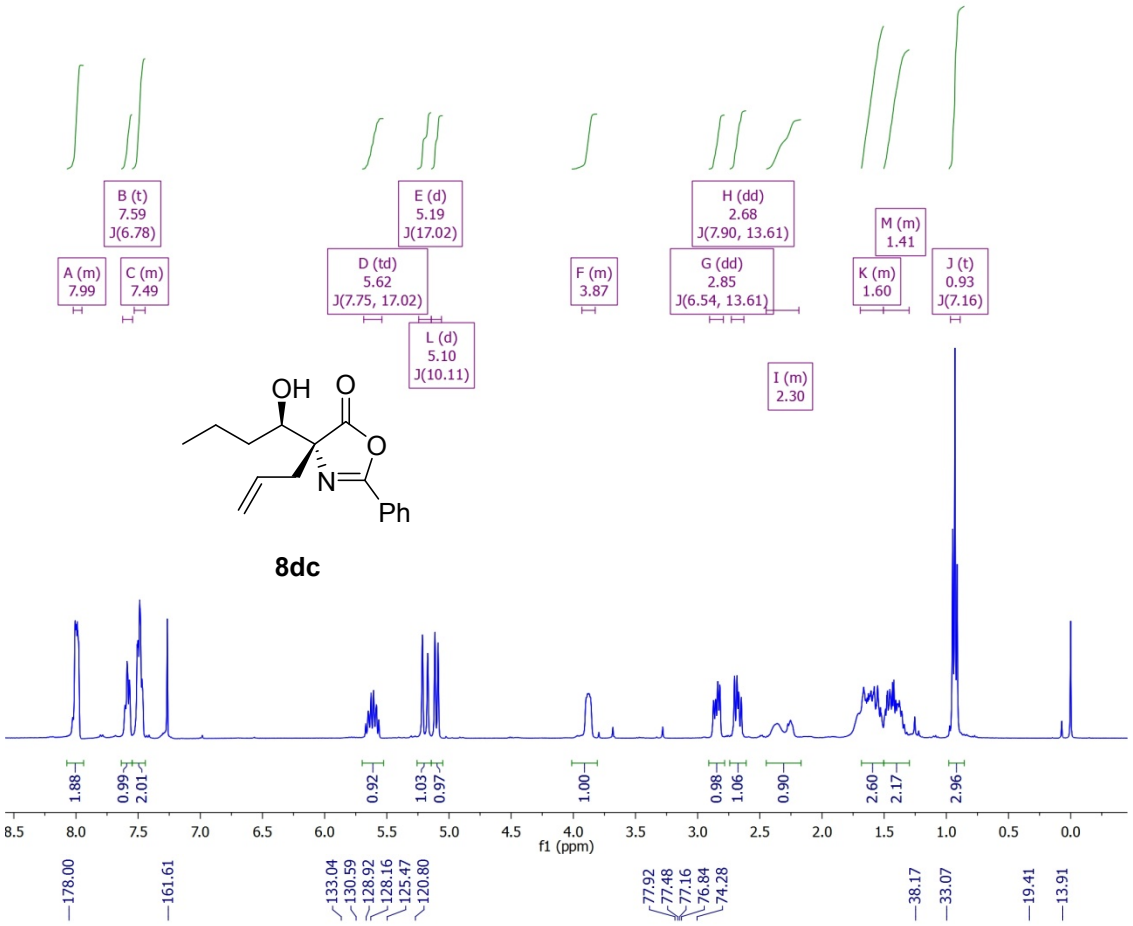
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

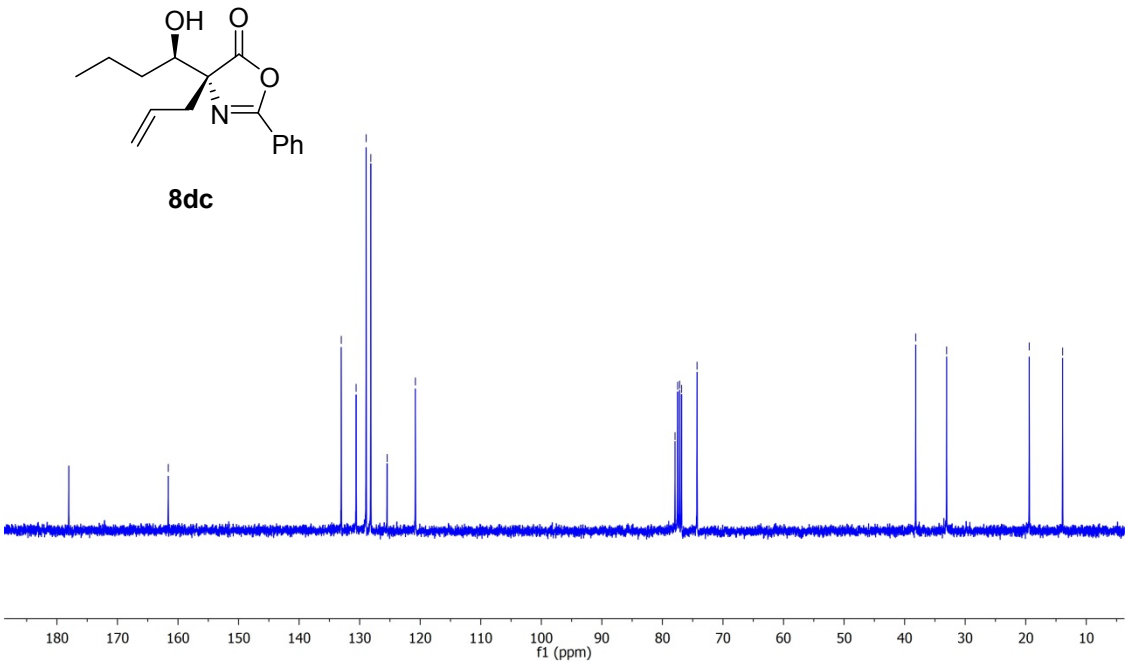
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  68.468 BB    1.2534 6.26077e4   782.81177  93.2501
   2  94.592 BV    1.5157 2681.97754   26.33486   3.9946
   3  97.691 VB    1.3537 1016.94580    9.79685   1.5147
   4 101.459 BB    1.5890  832.90808    6.82155   1.2406

Totals :                  6.71396e4   825.76503

==========================================================================
                          *** End of Report ***
```

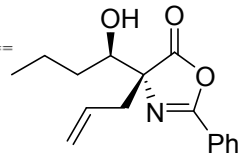

A (m)
7.99
B (t)
7.59
J(6.78)
C (m)
7.49
D (td)
5.62
J(7.75, 17.02)
E (d)
5.19
J(17.02)
L (d)
5.10
J(10.11)
F (m)
3.87
G (dd)
2.85
J(6.54, 13.61)
H (dd)
2.68
J(7.90, 13.61)
I (m)
2.30
K (m)
1.60
M (m)
1.41
J (t)
0.93
J(7.16)
OH
O
O
N
Ph
1.88
0.99
2.01
0.92
1.03
0.97
1.00
0.98
1.06
0.90
2.60
2.17
2.96
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
178.00
161.61
133.04
130.59
128.92
128.16
125.47
120.80
77.92
77.48
77.16
76.84
74.28
38.17
33.07
19.41
13.91

**8dc**


OH
O
O
N
Ph
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

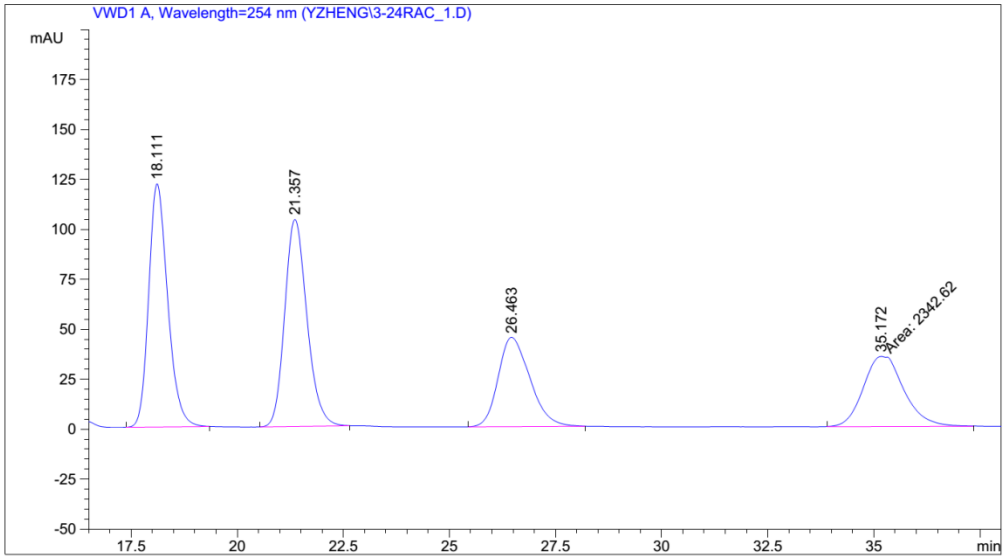
**8dc**

Data File C:\CHEM32\1\DATA\YZHENG\3-24RAC_1.D
Sample Name: 3-24rac




**8dc**
Racemic

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 6/28/2011 10:49:47 PM
Acq. Method     : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 4/2/2011 5:29:05 PM by jhl
Analysis Method : C:\CHEM32\1\METHODS\WASH.M
Last changed    : 8/10/2011 11:23:50 AM by jhl
                  (modified after loading)
Sample Info     : OJ-H, Hex:IPA = 97:3, 1 mL/min, 254 nm,  39 bar, Right
```




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

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

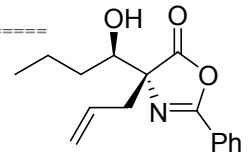

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  18.111 BB    0.4802 3796.21582  121.75442  31.0119
   2  21.357 BB    0.5592 3776.37964  103.59920  30.8499
   3  26.463 BB    0.7773 2325.93457   44.70980  19.0010
   4  35.172 MM    1.1125 2342.61719   35.09529  19.1372

Totals :                 1.22411e4   305.15870


==========================================================================
                          *** End of Report ***
```

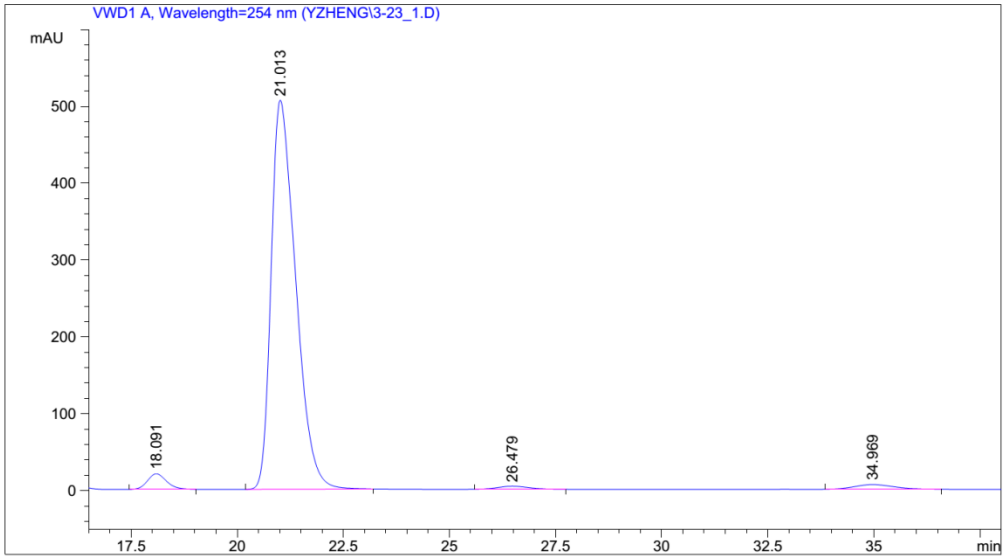
Data File C:\CHEM32\1\DATA\YZHENG\3-23_1.D
Sample Name: 3-23




**8dc**

94% ee, *anti*/*syn* = 97.5/2.5

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 6/28/2011 11:44:45 PM
Acq. Method     : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 4/2/2011 5:29:05 PM by jhl
Analysis Method : C:\CHEM32\1\METHODS\WASH.M
Last changed    : 8/10/2011 11:22:00 AM by jhl
                  (modified after loading)
Sample Info     : OJ-H, Hex:IPA = 97:3, 1 mL/min, 254 nm, 39 bar, Right
```




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

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

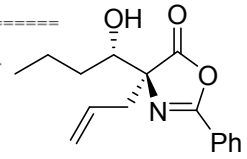

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|-------|
   1  18.091 BB    0.4825  631.86621   20.30002   2.9376
   2  21.013 BB    0.5856 2.02909e4   506.16669  94.3347
   3  26.479 BB    0.7396  197.53523    3.95765   0.9184
   4  34.969 BB    0.8847  389.17010    6.14300   1.8093

Totals :                  2.15094e4   536.56736


==========================================================================
                          *** End of Report ***
```

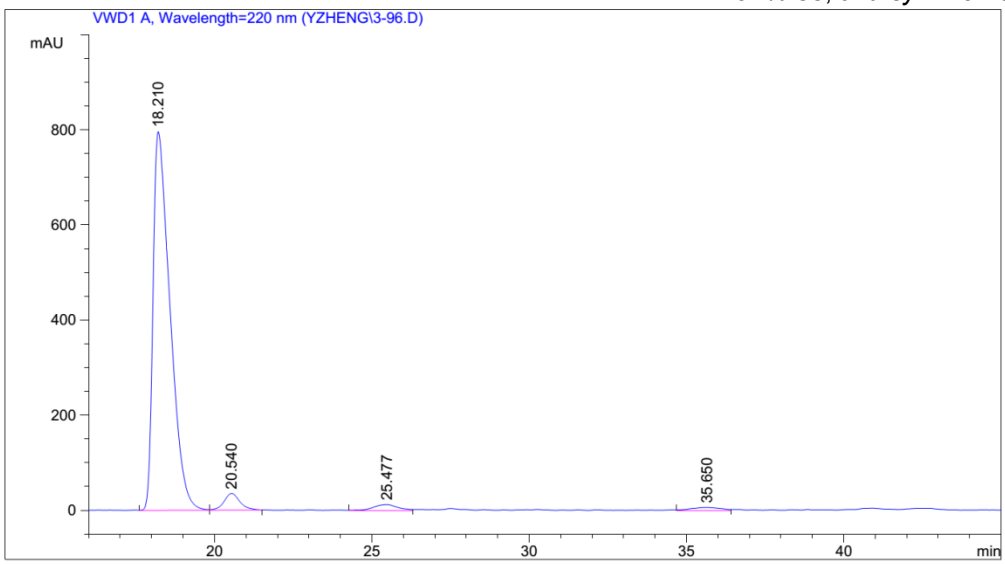
Data File C:\CHEM32\1\DATA\YZHENG\3-96.D
Sample Name: 3-96




**8dc-*ent***

-92% ee, *anti*/*syn* = 97/3

```
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 9/14/2011 4:14:53 PM
Acq. Method     : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 9/14/2011 3:30:28 PM by yang
Analysis Method : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 9/23/2011 3:13:05 PM by yang
                  (modified after loading)
Sample Info     : OJ-H, Hex:IPA = 97:3, 1 mL/min, 254 nm, 39 bar, Right
```




```
                          Area Percent Report

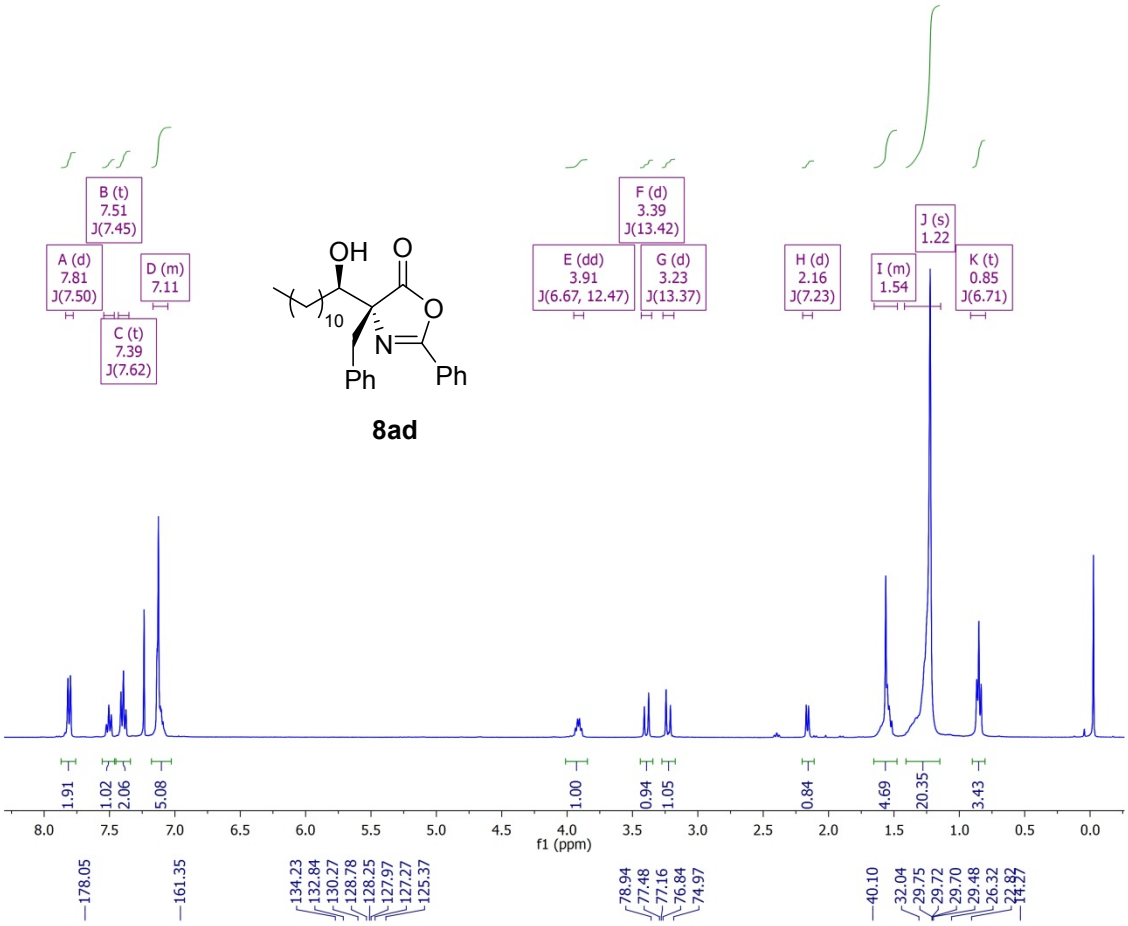
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=220 nm

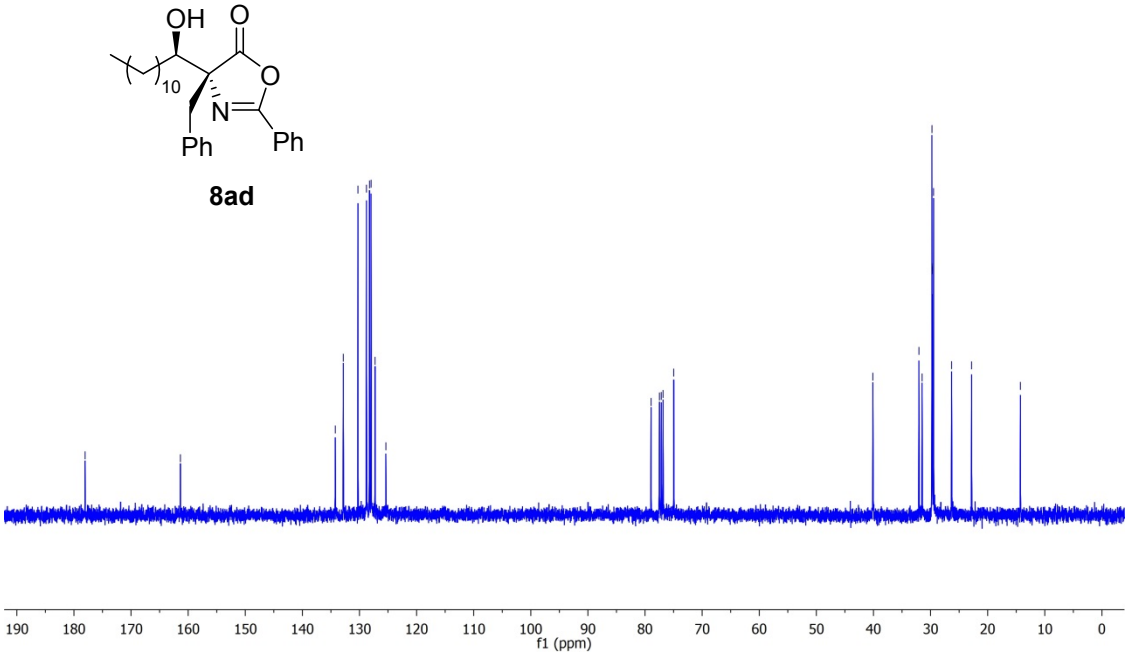
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  18.210 VV    0.5513 2.97630e4   795.91199  92.9582
   2  20.540 VB    0.5288 1218.60913   34.97573   3.8061
   3  25.477 VV    0.8019  641.35376   11.95811   2.0031
   4  35.650 VV    0.8521  394.65192    5.97526   1.2326

Totals :                  3.20176e4   848.82110


                          *** End of Report ***
```


B (t)
7.51
J(7.45)
A (d)
7.81
J(7.50)
D (m)
7.11
C (t)
7.39
J(7.62)
OH
O
10
O
N
Ph
Ph
F (d)
3.39
J(13.42)
E (dd)
3.91
J(6.67, 12.47)
G (d)
3.23
J(13.37)
H (d)
2.16
J(7.23)
J (s)
1.22
I (m)
1.54
K (t)
0.85
J(6.71)
1.91
1.02
2.06
5.08
1.00
0.94
1.05
0.84
4.69
20.35
3.43
f1 (ppm)
178.05
161.35
134.23
132.84
130.27
128.78
128.25
127.97
127.27
125.37
78.94
77.48
77.16
76.84
74.97
40.10
32.04
29.75
29.72
29.70
29.48
26.32
22.82
14.27

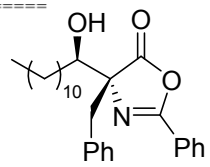
**8ad**


OH
O
10
O
N
Ph
Ph
f1 (ppm)

**8ad**

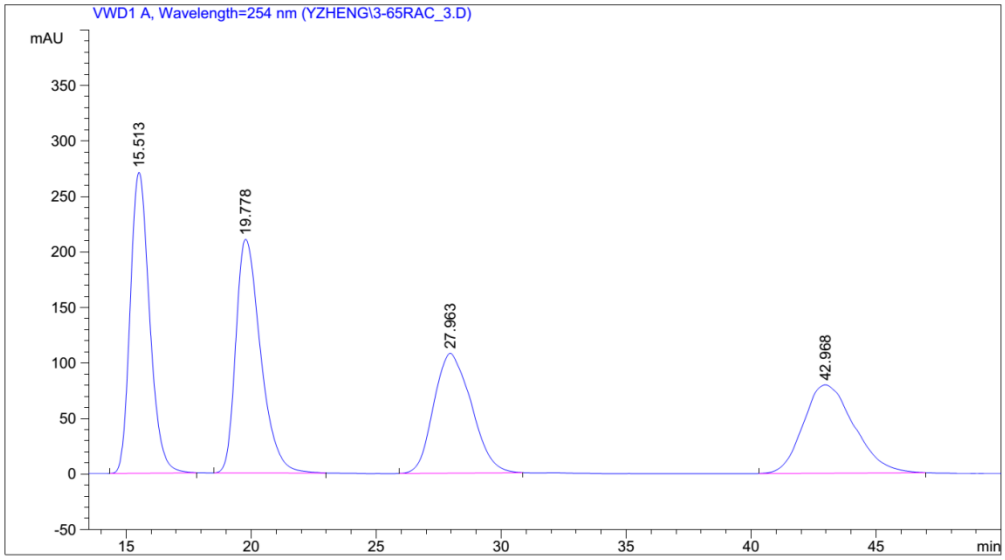
Data File C:\CHEM32\1\DATA\YZHENG\3-65RAC_3.D
Sample Name: 3-65rac

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 8/3/2011 4:49:15 PM
Acq. Method     : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 8/3/2011 4:22:01 PM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\WASH.M
Last changed    : 8/10/2011 11:17:35 AM by jhl
                  (modified after loading)
Sample Info     : AS-H, Hex:IPA = 96.5:3.5, 1.0 mL/min, 254 nm, 44 bar, R
                  ight
```




**8ad**

Racemic




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

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


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  15.513 BB    0.8414 1.45775e4   271.07748  28.2494
   2  19.778 BB    1.0537 1.44936e4   210.49646  28.0867
   3  27.963 BB    1.5207 1.12952e4   107.95037  21.8887
   4  42.968 BB    1.9230 1.12367e4    79.62708  21.7752

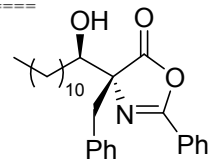
Totals :                 5.16030e4   669.15139

==========================================================================
                          *** End of Report ***
```

Instrument 1 8/10/2011 11:17:40 AM jhl  Page 1 of 1

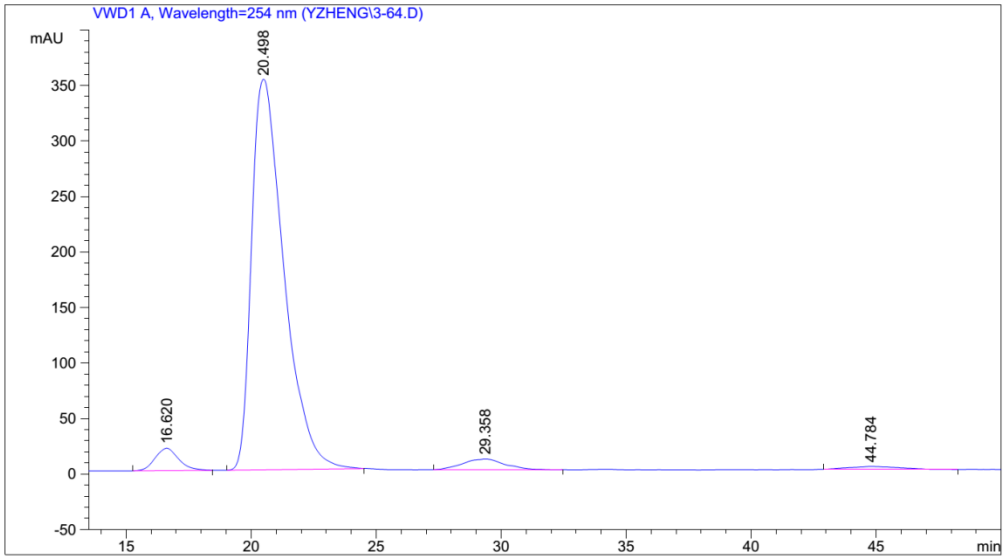
Data File C:\CHEM32\1\DATA\YZHENG\3-64.D
Sample Name: 3-64

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 8/3/2011 10:57:06 AM
Acq. Method     : C:\CHEM32\1\METHODS\METHOD-1_LC.M
Last changed    : 8/2/2011 5:34:32 PM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\WASH.M
Last changed    : 8/10/2011 11:16:24 AM by jhl
                  (modified after loading)
Sample Info     : AS-H, Hex:IPA = 96.5:3.5, 1.0 mL/min, 254 nm, 44 bar, R
                  ight
```




**8ad**

92% ee, *anti*/*syn* = 95.5/4.5




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

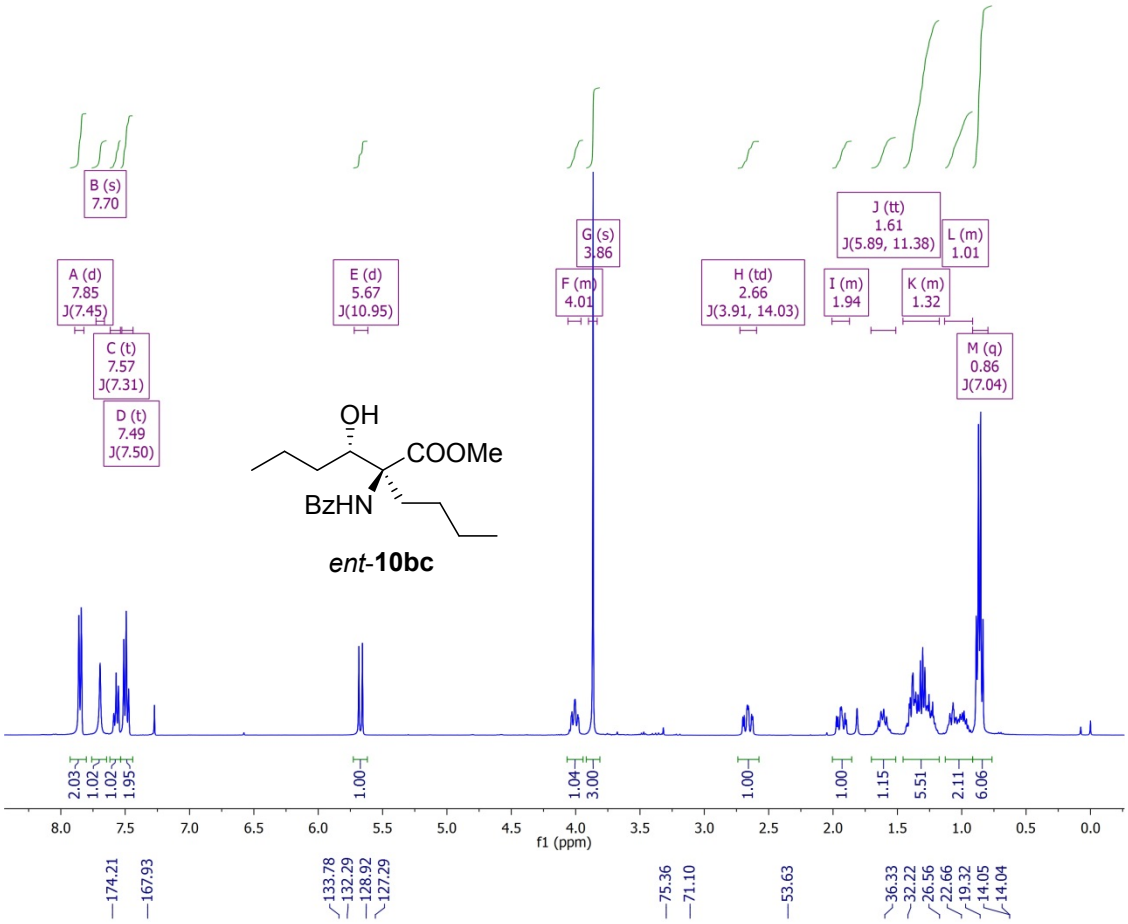
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

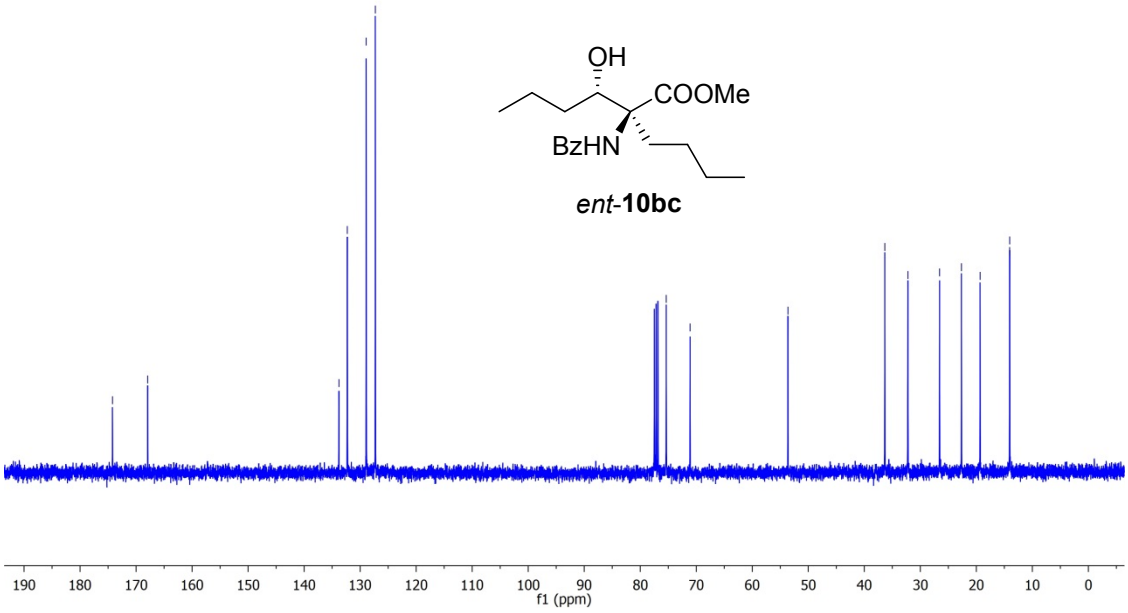
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  16.620 BB    1.0042 1316.72168   20.18391   3.8245
   2  20.498 BB    1.3238 3.15790e4   351.84460  91.7241
   3  29.358 BB    1.5167 1124.23438    9.63799   3.2654
   4  44.784 BB    1.7626  408.27173    2.79471   1.1859

Totals :                  3.44282e4   384.46122

==========================================================================
                          *** End of Report ***
```

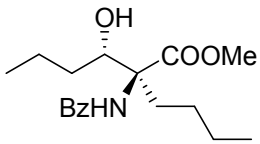



*ent*-**10bc**




*ent*-**10bc**

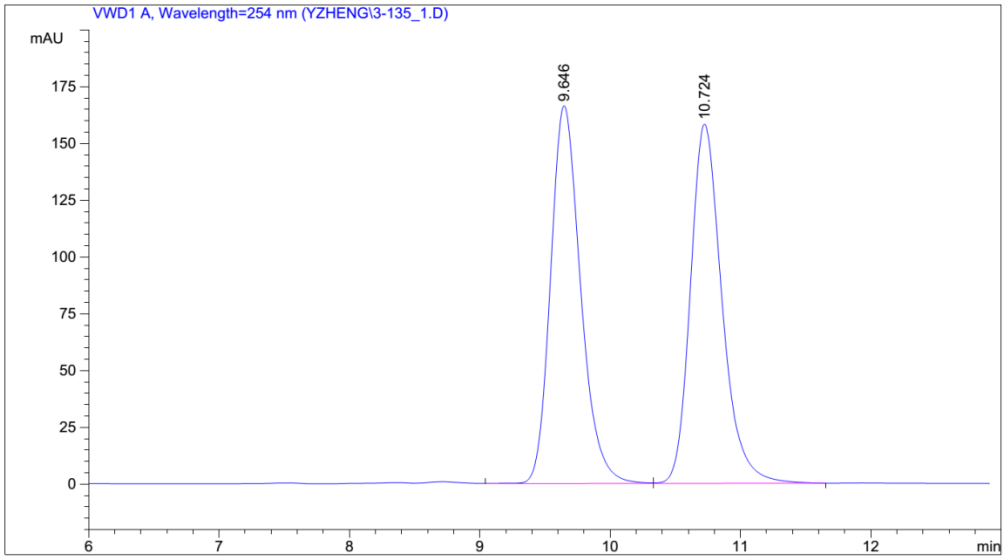
Data File C:\CHEM32\1\DATA\YZHENG\3-135_1.D
Sample Name: 3-135rac




***ent*-10bc**

Racemic

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 10/20/2011 11:34:21 AM
Acq. Method     : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 10/20/2011 11:08:32 AM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\WASH.M
Last changed    : 10/26/2011 10:41:01 AM by jhl
                  (modified after loading)
Sample Info     : AD-H, Hex:IPA = 90:10, 1.0 mL/min, 254 nm, 42 bar, Righ
                  t
```




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

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

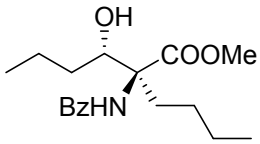

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   9.646 VV    0.2451 2663.87866  166.34001  50.0445
   2  10.724 VB    0.2572 2659.14600  158.23003  49.9555

Totals :                  5323.02466  324.57004


==========================================================================
                        *** End of Report ***
```

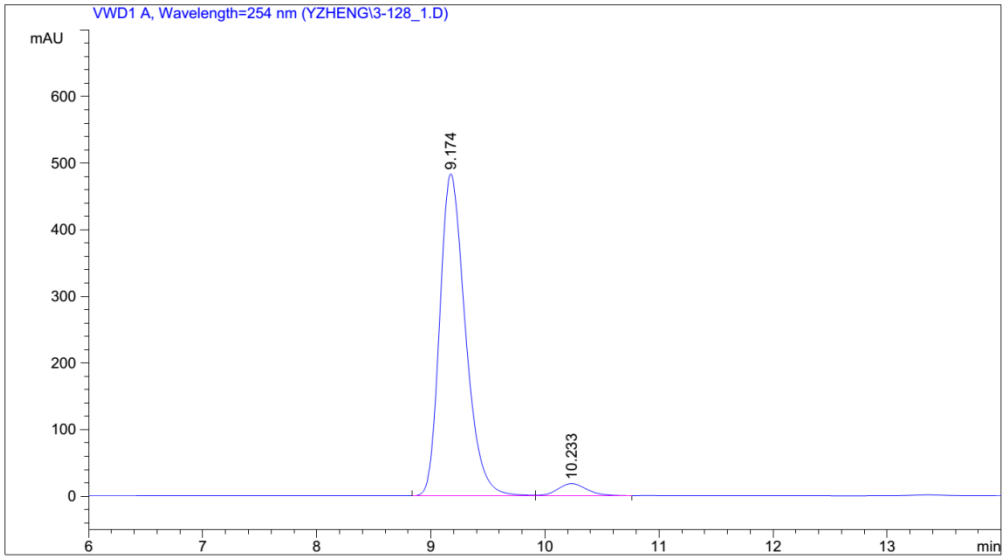
Data File C:\CHEM32\1\DATA\YZHENG\3-128_1.D
Sample Name: 3-128




*ent*-**10bc**

-92% ee

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 10/20/2011 12:12:47 PM
Acq. Method     : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 10/20/2011 11:08:32 AM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\WASH.M
Last changed    : 10/26/2011 10:39:21 AM by jhl
                  (modified after loading)
Sample Info     : AD-H, Hex:IPA = 90:10, 1.0 mL/min, 254 nm, 42 bar, Righ
                  t
```




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

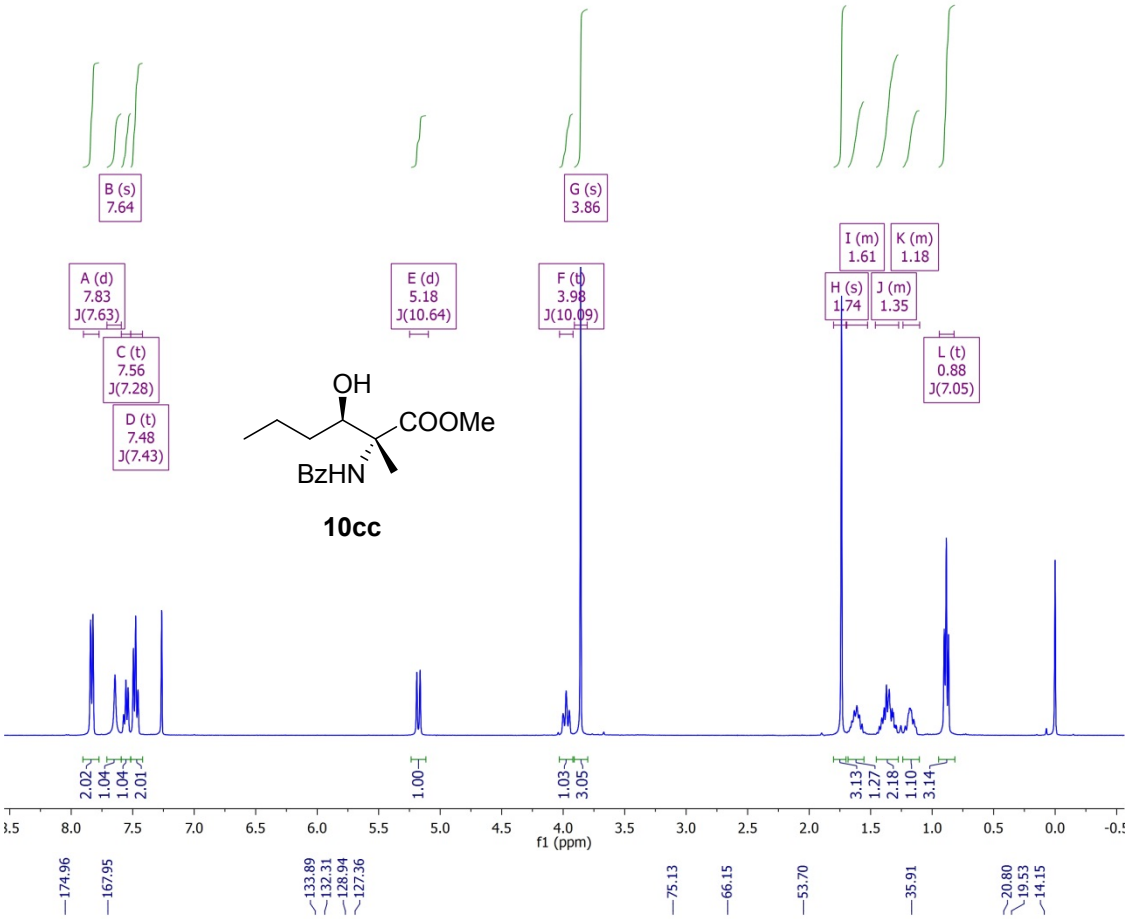
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

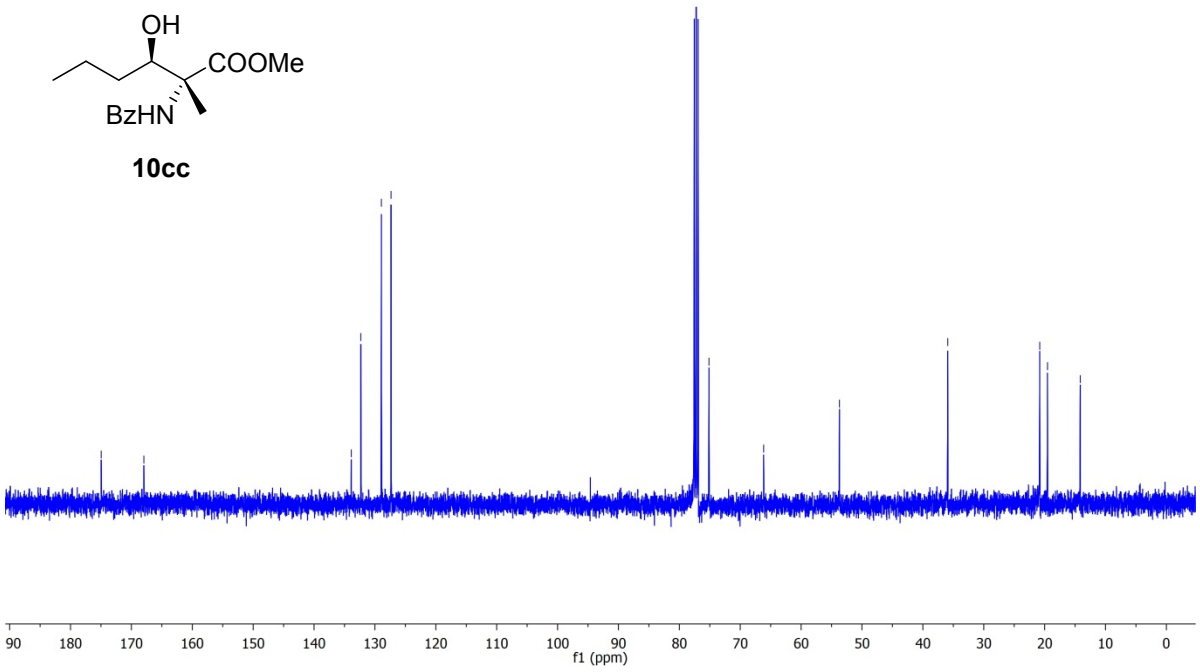
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   9.174 BV    0.2400 7529.78955  483.25226  95.9212
   2  10.233 VB    0.2699  320.18448   18.27895   4.0788

Totals :                  7849.97403  501.53121


==========================================================================
                        *** End of Report ***
```


B (s)
7.64
A (d)
7.83
J(7.63)
C (t)
7.56
J(7.28)
D (t)
7.48
J(7.43)
E (d)
5.18
J(10.64)
G (s)
3.86
F (t)
3.98
J(10.09)
I (m)
1.61
K (m)
1.18
H (s)
1.74
J (m)
1.35
L (t)
0.88
J(7.05)
OH
COOMe
BzHN
2.02
1.04
1.04
2.01
1.00
1.03
3.05
3.13
1.27
2.18
1.10
3.14
f1 (ppm)
174.96
167.95
133.89
132.31
128.94
127.36
75.13
66.15
53.70
35.91
20.80
19.53
14.15

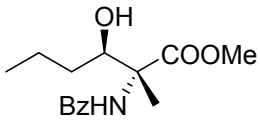
**10cc**


OH
COOMe
BzHN
f1 (ppm)

**10cc**

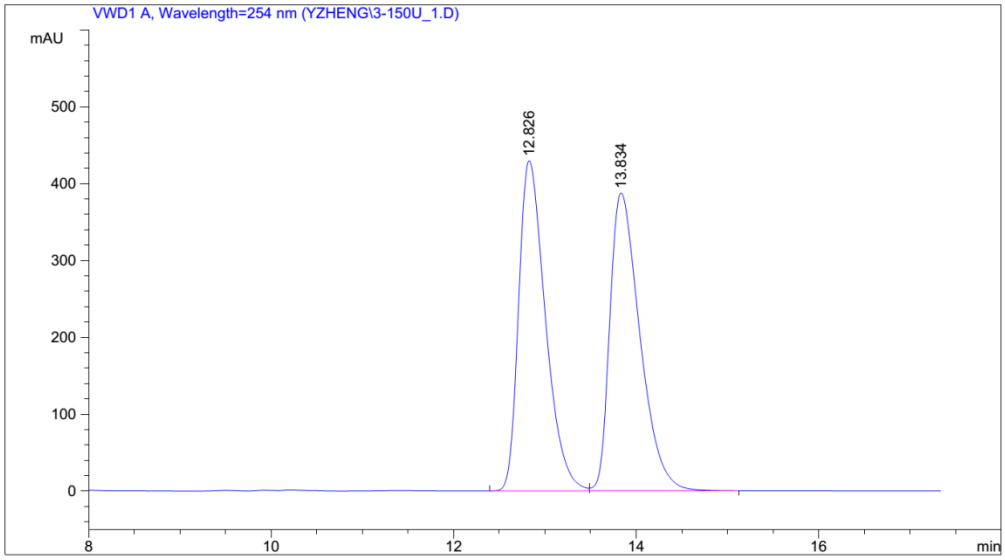
Data File C:\CHEM32\1\DATA\YZHENG\3-150U_1.D
Sample Name: 3-150U

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 2/22/2012 10:59:30 AM
Acq. Method     : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 2/22/2012 10:21:20 AM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 2/22/2012 12:36:15 PM by yang
                  (modified after loading)
Sample Info     : AD-H, Hex:IPA = 90:10, 1.0 mL/min, 254 nm, 41 bar, Righ
                  t
```




**10cc**

Racemic




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

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


Signal 1: VWD1 A, Wavelength=254 nm

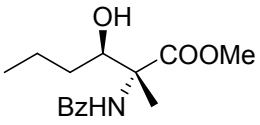
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  12.826 BV    0.3132 8744.77539  429.55450  49.7204
   2  13.834 VB    0.3506 8843.12598  387.57666  50.2796

Totals :                 1.75879e4   817.13116

==========================================================================
                          *** End of Report ***
```

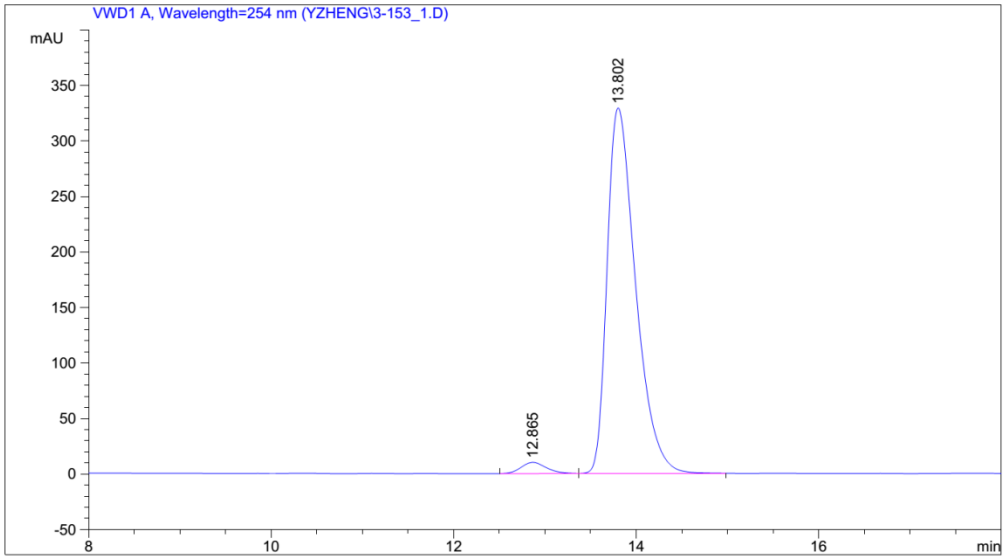
Data File C:\CHEM32\1\DATA\YZHENG\3-153_1.D
Sample Name: 3-153

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 2/22/2012 11:19:36 AM
Acq. Method     : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 2/22/2012 10:21:20 AM by yang
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 2/22/2012 12:35:10 PM by yang
                  (modified after loading)
Sample Info     : AD-H, Hex:IPA = 90:10, 1.0 mL/min, 254 nm, 41 bar, Righ
                  t
```




**10cc**

95% ee




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

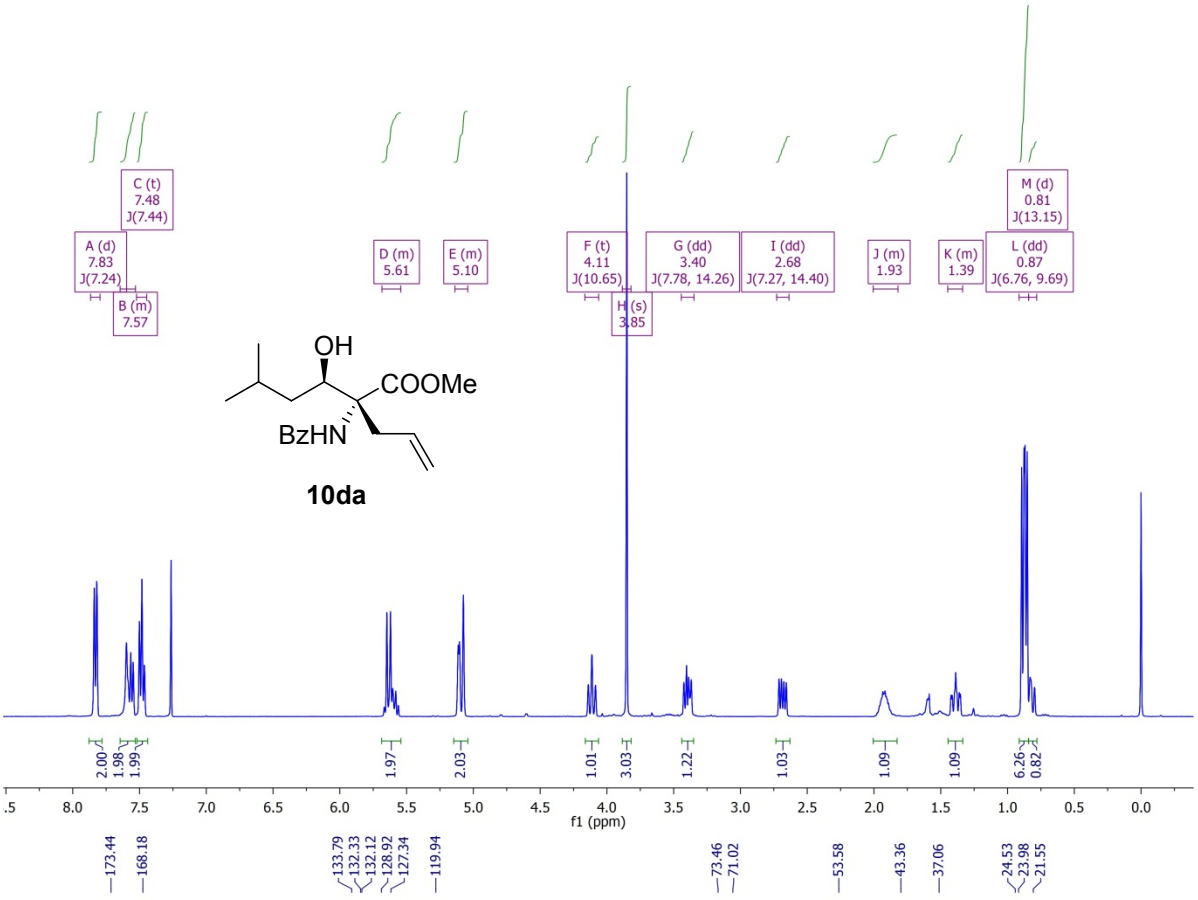
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

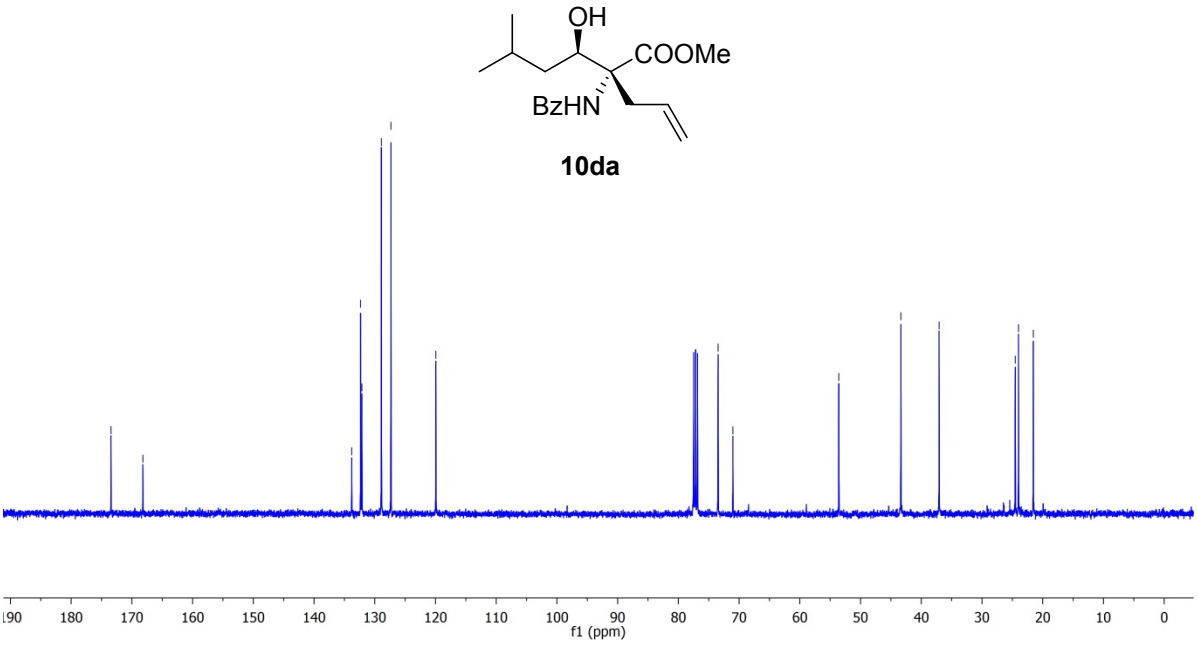
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  12.865 BV    0.2930  194.25142   10.15565   2.6504
   2  13.802 VB    0.3328 7134.79004  329.33212  97.3496

Totals :                  7329.04146  339.48777

==========================================================================
                        *** End of Report ***
```

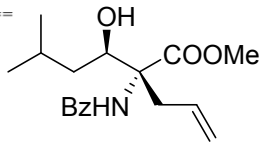

A (d) 7.83 J(7.24)
B (m) 7.57
C (t) 7.48 J(7.44)
D (m) 5.61
E (m) 5.10
F (t) 4.11 J(10.65)
H (s) 3.85
G (dd) 3.40 J(7.78, 14.26)
I (dd) 2.68 J(7.27, 14.40)
J (m) 1.93
K (m) 1.39
L (dd) 0.87 J(6.76, 9.69)
M (d) 0.81 J(13.15)
OH
COOMe
BzHN
2.00
1.98
1.99
1.97
2.03
1.01
3.03
1.22
1.03
1.09
1.09
6.26
0.82
f1 (ppm)

**10da**


OH
COOMe
BzHN
173.44
168.18
133.79
132.33
132.12
128.92
127.34
119.94
73.46
71.02
53.58
43.36
37.06
24.53
23.98
21.55
f1 (ppm)

**10da**

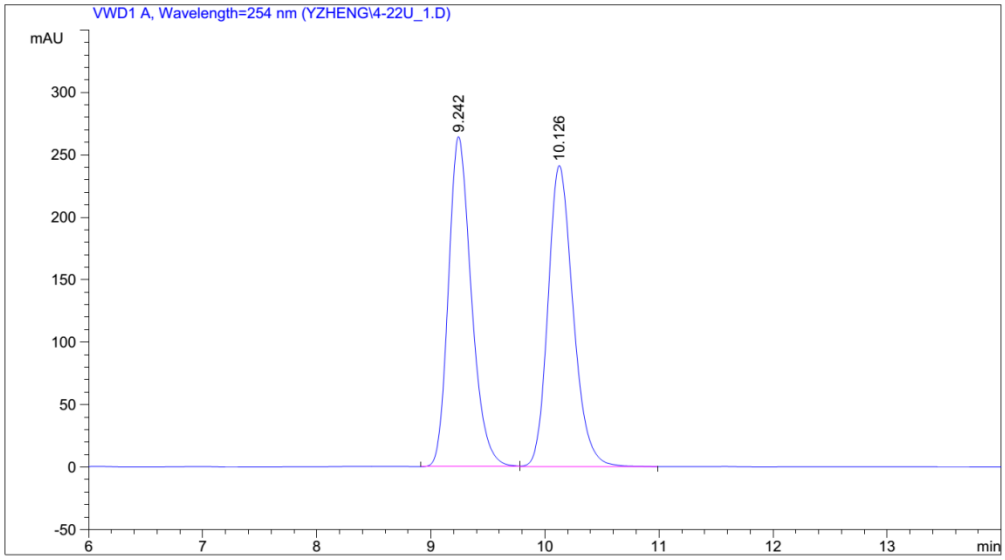
Data File C:\CHEM32\1\DATA\YZHENG\4-22U_1.D
Sample Name: 4-22U




**10da**

Racemic

```
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 2/21/2012 4:31:25 PM
Acq. Method     : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 2/21/2012 3:23:10 PM by jhl
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 2/22/2012 12:37:27 PM by yang
                  (modified after loading)
Sample Info     : AD-H, Hex:IPA = 90:10, 1.0 mL/min, 254 nm, 41 bar, Righ
                  t
```




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

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

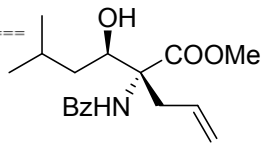

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   9.242 VV    0.2142 3697.71021  264.33917  50.0371
   2  10.126 VB    0.2354 3692.22241  241.17551  49.9629

Totals :                  7389.93262  505.51468

=====================================================================
                         *** End of Report ***
```

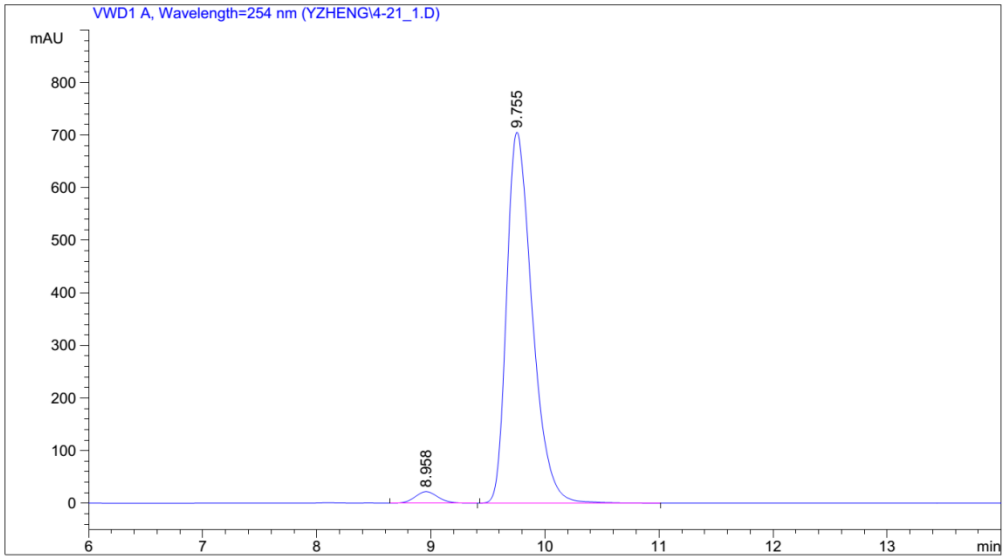
Data File C:\CHEM32\1\DATA\YZHENG\4-21_1.D
Sample Name: 4-22U




**10da**

95% ee

```
==========================================================================
Acq. Operator   : yang
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 2/21/2012 4:48:08 PM
Acq. Method     : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 2/21/2012 3:23:10 PM by jhl
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\YANG.M
Last changed    : 2/22/2012 12:38:09 PM by yang
                  (modified after loading)
Sample Info     : AD-H, Hex:IPA = 90:10, 1.0 mL/min, 254 nm, 41 bar, Righ
                  t
```




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

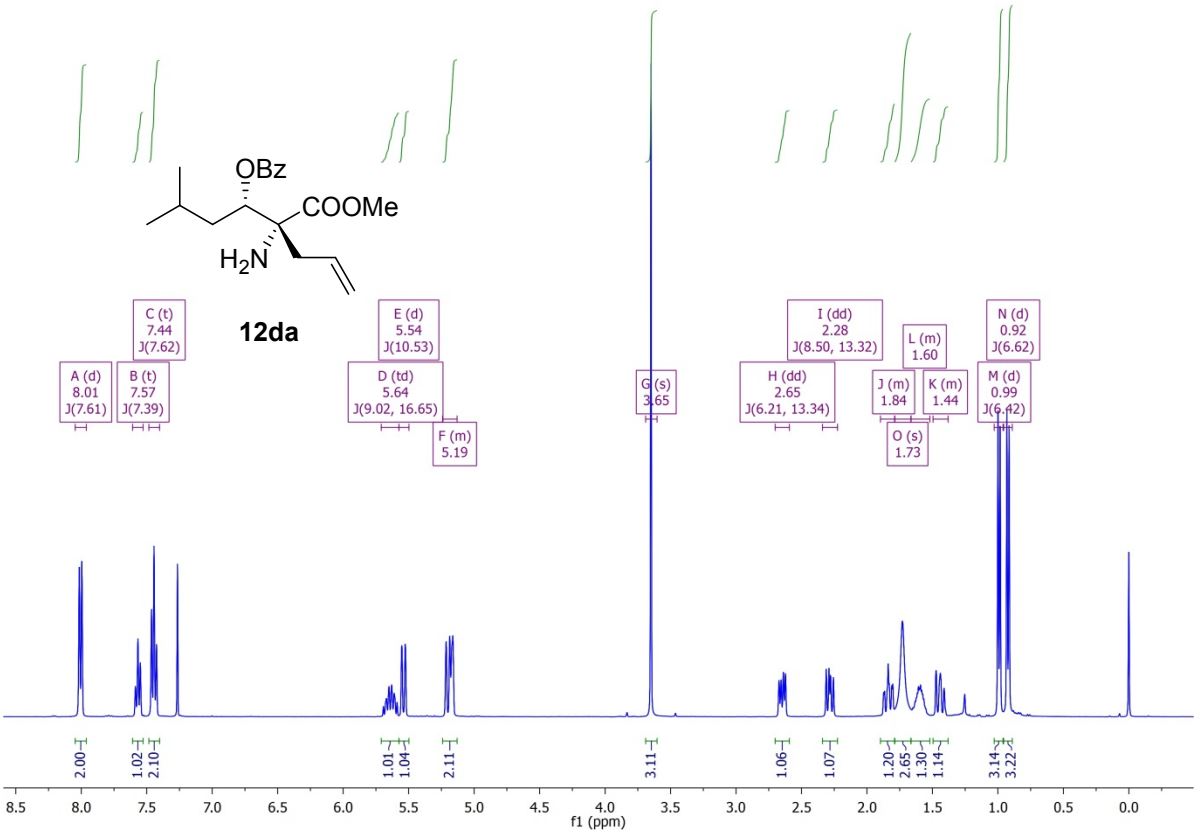
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

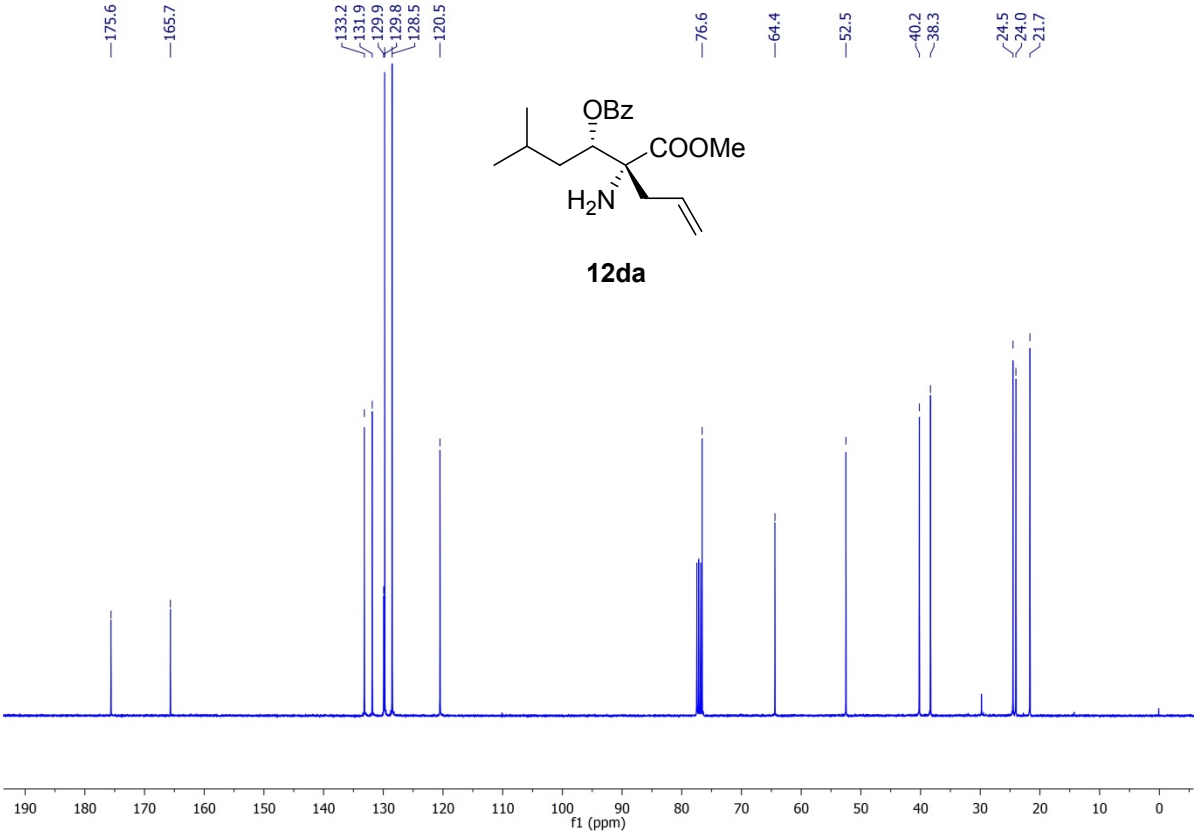

Signal 1: VWD1 A, Wavelength=254 nm

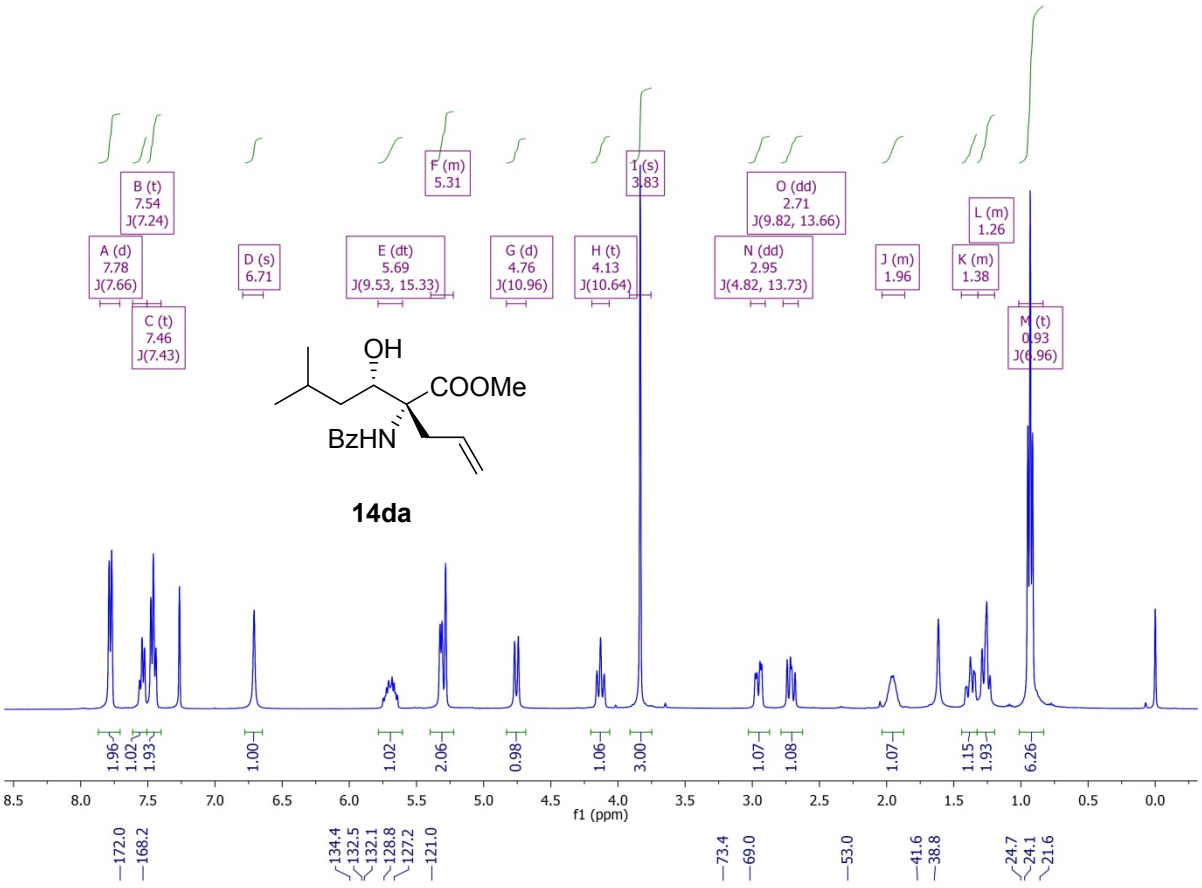
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   8.958 VB    0.2151  306.92242   22.00724   2.7158
   2   9.755 BB    0.2401 1.09945e4   705.41644  97.2842

Totals :                  1.13014e4   727.42368

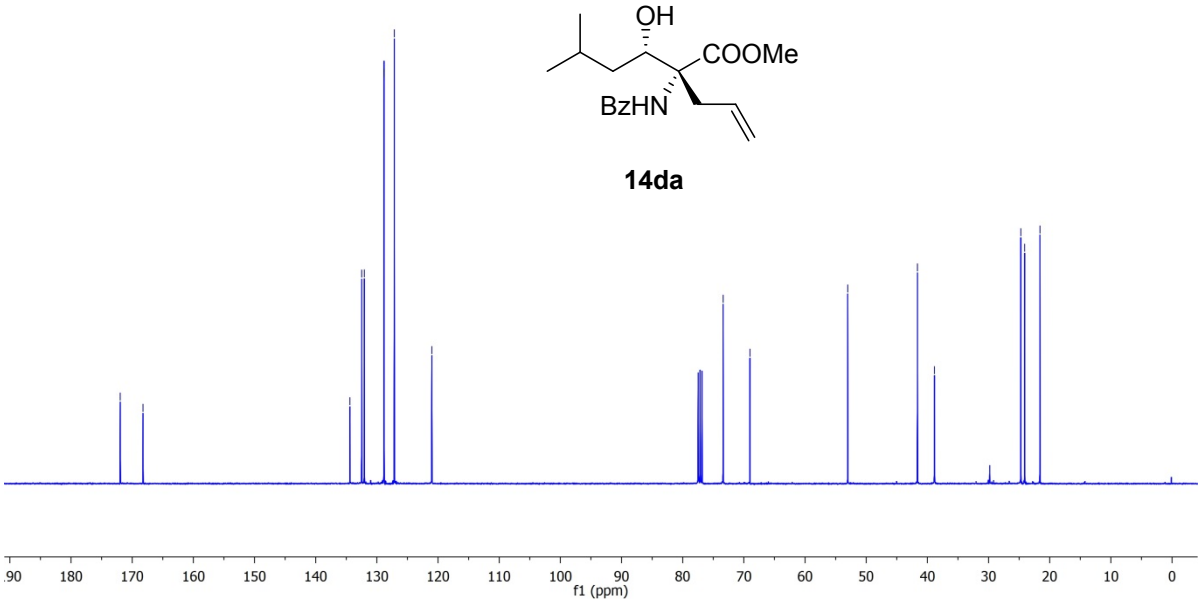

==========================================================================
                        *** End of Report ***
```


OBz
COOMe
H2N
12da
A (d) 8.01 J(7.61)
B (t) 7.57 J(7.39)
C (t) 7.44 J(7.62)
D (td) 5.64 J(9.02, 16.65)
E (d) 5.54 J(10.53)
F (m) 5.19
G (s) 3.65
H (dd) 2.65 J(6.21, 13.34)
I (dd) 2.28 J(8.50, 13.32)
J (m) 1.84
K (m) 1.44
L (m) 1.60
M (d) 0.99 J(6.42)
N (d) 0.92 J(6.62)
O (s) 1.73
2.00
1.02
2.10
1.01
1.04
2.11
3.11
1.06
1.07
1.20
2.65
1.30
1.14
3.14
3.22
f1 (ppm)


175.6
165.7
133.2
131.9
129.9
129.8
128.5
120.5
76.6
64.4
52.5
40.2
38.3
24.5
24.0
21.7
OBz
COOMe
H2N
12da
f1 (ppm)


A (d)
7.78
J(7.66)
B (t)
7.54
J(7.24)
C (t)
7.46
J(7.43)
D (s)
6.71
E (dt)
5.69
J(9.53, 15.33)
F (m)
5.31
G (d)
4.76
J(10.96)
H (t)
4.13
J(10.64)
I (s)
3.83
N (dd)
2.95
J(4.82, 13.73)
O (dd)
2.71
J(9.82, 13.66)
J (m)
1.96
K (m)
1.38
L (m)
1.26
M (t)
0.93
J(6.96)
OH
COOMe
BzHN
1.96
1.02
1.93
1.00
1.02
2.06
0.98
1.06
3.00
1.07
1.08
1.07
1.15
1.93
6.26
f1 (ppm)
172.0
168.2
134.4
132.5
132.1
128.8
127.2
121.0
73.4
69.0
53.0
41.6
38.8
24.7
24.1
21.6

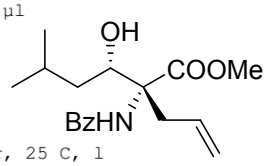
**14da**


OH
COOMe
BzHN
f1 (ppm)

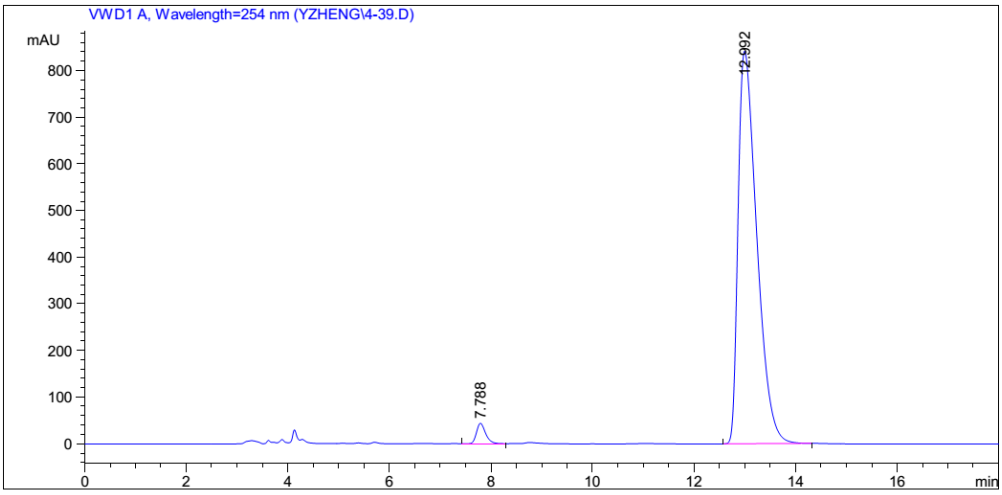
**14da**

Data File C:\HPCHEM\1\DATA\YZHENG\4-39.D
Sample Name: 4-39

```
==========================================================================
Acq. Operator   : YANG
Acq. Instrument : Deng Lab LC                    Location : Vial 21
Injection Date  : 10/3/2014 3:36:36 PM
                                                 Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\METHOD 1.M
Last changed    : 10/3/2014 3:25:09 PM by YANG
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\METHOD 1.M
Last changed    : 10/4/2014 3:59:26 PM by CHAO
                  (modified after loading)
Sample Info     : AD-H, Hex/IPA=90/10, 1.0 mL/min, 254nm, 47 bar, 25 C, l
                  eft
```




**14da**, 95% ee




```
==========================================================================
                        Fraction Information
==========================================================================
Fraction collection off
==========================================================================
No Fractions found.
==========================================================================
==========================================================================
                       Area Percent Report
==========================================================================

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

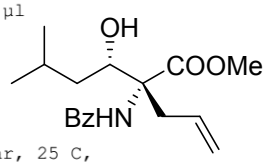

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   7.788 VV    0.1921  560.22424   44.05143   2.5360
   2  12.992 BB    0.3944 2.15309e4   842.17053  97.4640

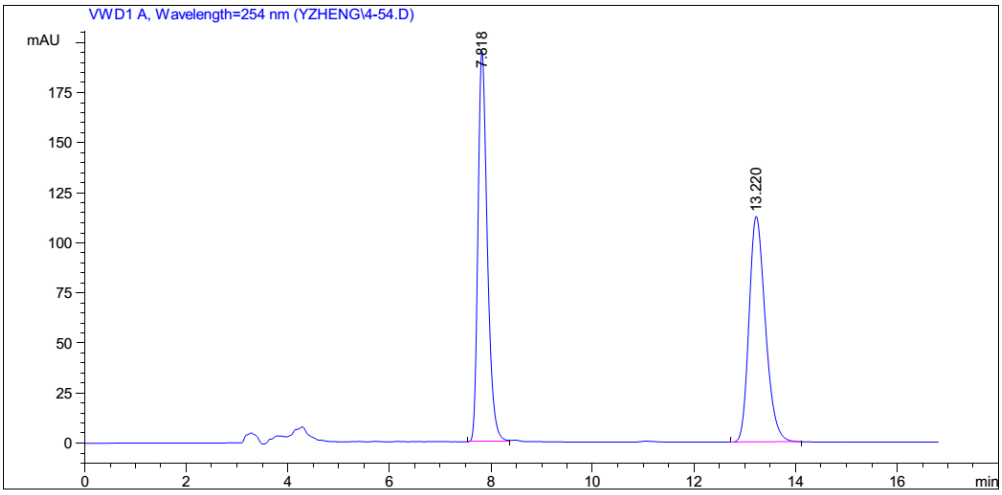
Totals :                  2.20912e4   886.22196
```

Data File C:\HPCHEM\1\DATA\YZHENG\4-54.D
Sample Name: 4-54

```
======================================================================
Acq. Operator   : YANG
Acq. Instrument : Deng Lab LC                    Location : Vial 21
Injection Date  : 10/4/2014 4:20:21 PM
                                                 Inj Volume : 20 µl
Acq. Method     : C:\HPCHEM\1\METHODS\METHOD 1.M
Last changed    : 10/4/2014 4:02:24 PM by YANG
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\METHOD 1.M
Last changed    : 10/4/2014 4:38:43 PM by CHAO
                  (modified after loading)
Sample Info     : AD-H, Hex/IPA=90/10, 1.0 mL/min, 254 nm, 47 bar, 25 C,
                  left
```




**14da**, Racemic



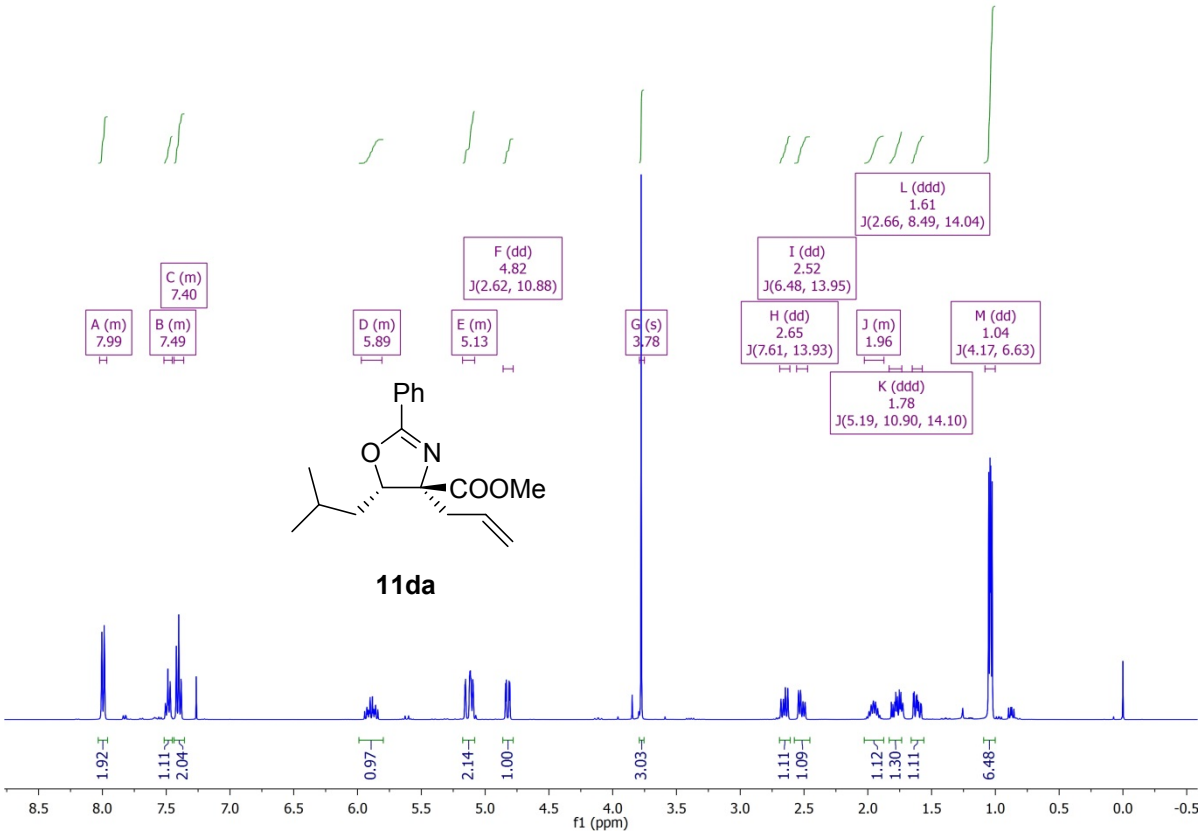

```
==========================================================================
                        Fraction Information
==========================================================================
Fraction collection off
==========================================================================
No Fractions found.
==========================================================================
======================================================================
                      Area Percent Report
======================================================================

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

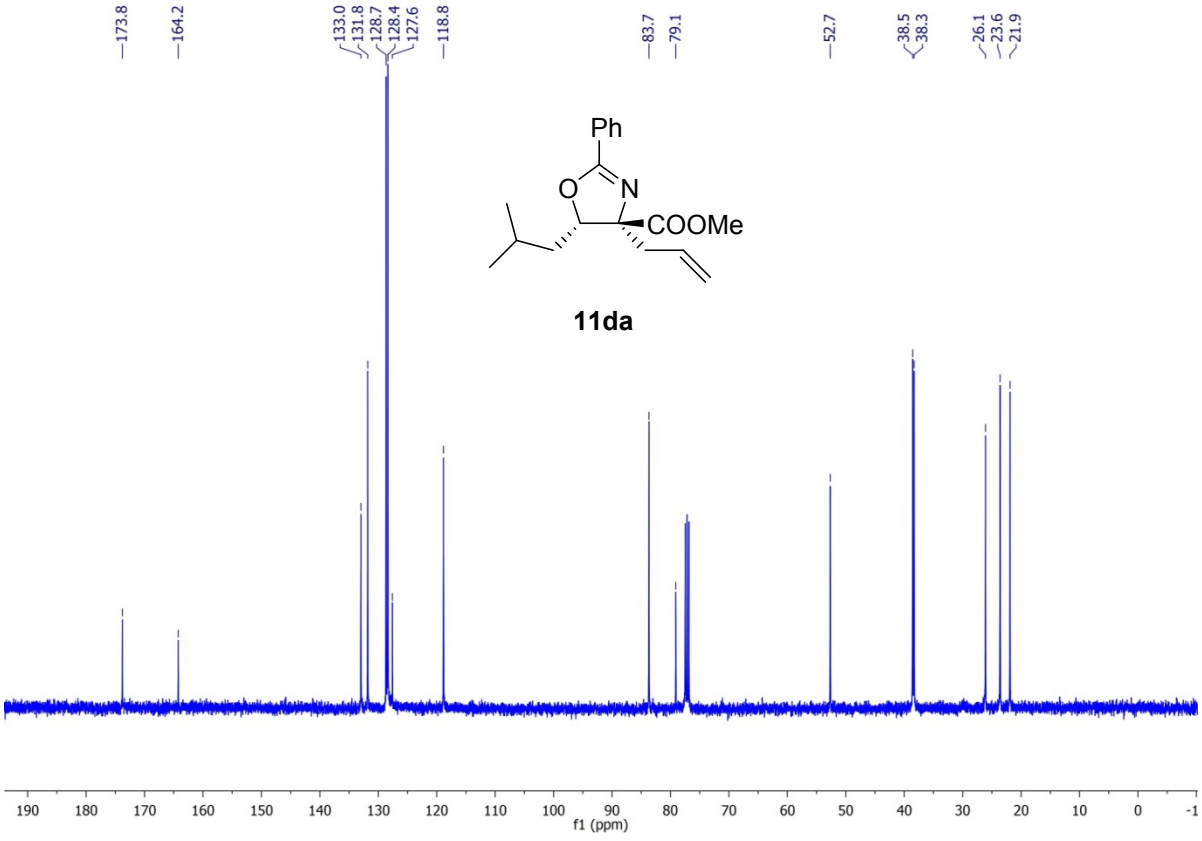

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   7.818 BV    0.1944 2497.13208  195.17357  49.8004
   2  13.220 BB    0.3436 2517.15137  112.67712  50.1996

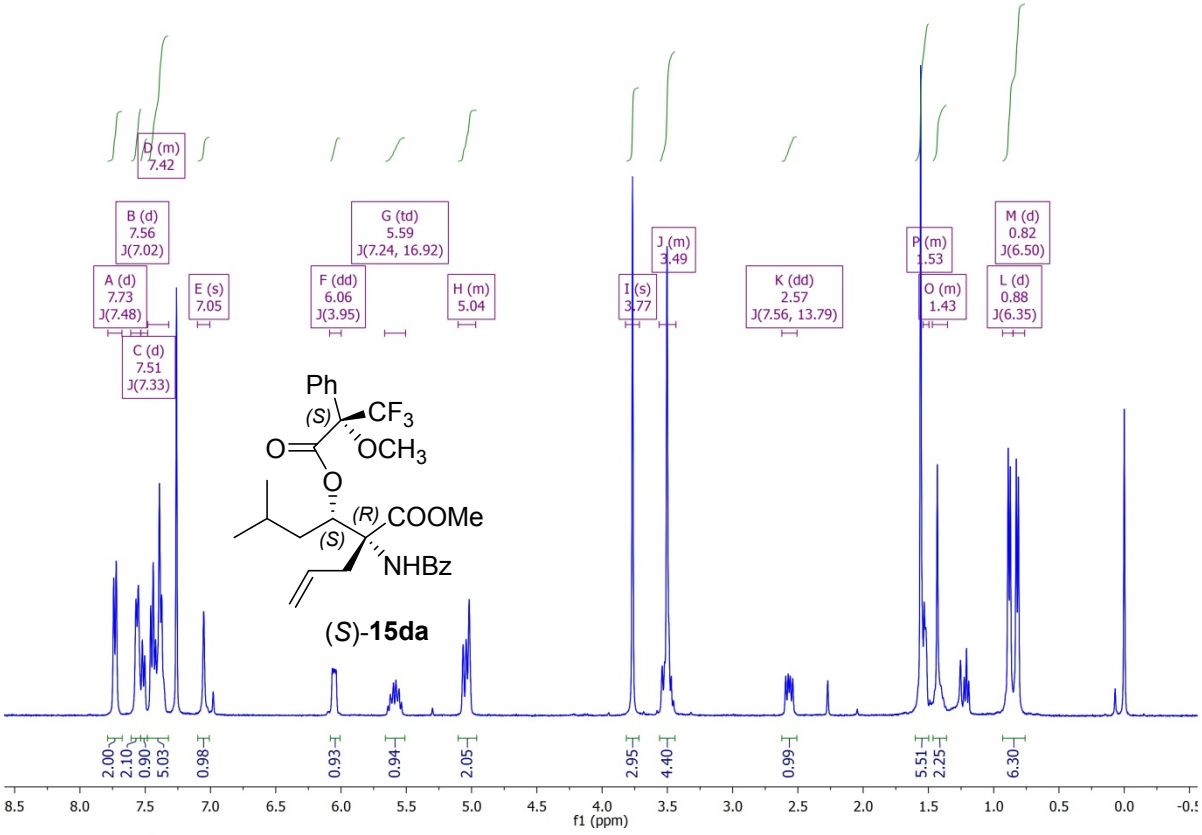
Totals :                  5014.28345  307.85069
```

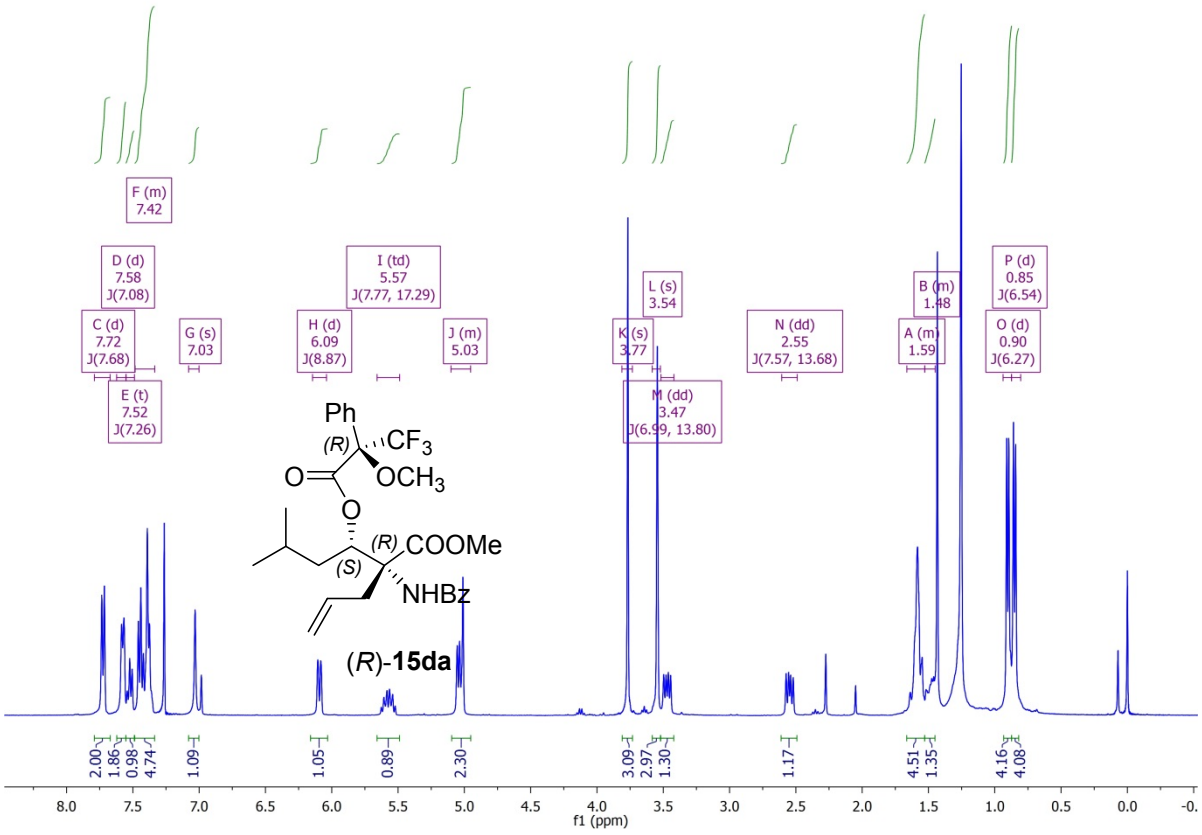

A (m)
7.99
B (m)
7.49
C (m)
7.40
D (m)
5.89
E (m)
5.13
F (dd)
4.82
J(2.62, 10.88)
G (s)
3.78
H (dd)
2.65
J(7.61, 13.93)
I (dd)
2.52
J(6.48, 13.95)
J (m)
1.96
K (ddd)
1.78
J(5.19, 10.90, 14.10)
L (ddd)
1.61
J(2.66, 8.49, 14.04)
M (dd)
1.04
J(4.17, 6.63)
Ph
O
N
COOMe
1.92
1.11
2.04
0.97
2.14
1.00
3.03
1.11
1.09
1.12
1.30
1.11
6.48
f1 (ppm)

**11da**


173.8
164.2
133.0
131.8
128.7
128.4
127.6
118.8
83.7
79.1
52.7
38.5
38.3
26.1
23.6
21.9
Ph
O
N
COOMe
f1 (ppm)

**11da**


D (m)
7.42
B (d)
7.56
J(7.02)
A (d)
7.73
J(7.48)
C (d)
7.51
J(7.33)
E (s)
7.05
G (td)
5.59
J(7.24, 16.92)
F (dd)
6.06
J(3.95)
H (m)
5.04
J (m)
3.49
I (s)
3.77
K (dd)
2.57
J(7.56, 13.79)
P (m)
1.53
O (m)
1.43
M (d)
0.82
J(6.50)
L (d)
0.88
J(6.35)
Ph
(S)
CF3
O
OCH3
O
(R)
COOMe
(S)
NHBz
(S)-15da
2.00
2.10
0.90
5.03
0.98
0.93
0.94
2.05
2.95
4.40
0.99
5.51
2.25
6.30
f1 (ppm)


F (m)
7.42
D (d)
7.58
J(7.08)
C (d)
7.72
J(7.68)
E (t)
7.52
J(7.26)
G (s)
7.03
I (td)
5.57
J(7.77, 17.29)
H (d)
6.09
J(8.87)
J (m)
5.03
L (s)
3.54
K (s)
3.77
M (dd)
3.47
J(6.99, 13.80)
N (dd)
2.55
J(7.57, 13.68)
B (m)
1.48
A (m)
1.59
P (d)
0.85
J(6.54)
O (d)
0.90
J(6.27)
Ph
(R)
CF3
O
OCH3
O
(R)
COOMe
(S)
NHBz
(R)-15da
2.00
1.86
0.98
4.74
1.09
1.05
0.89
2.30
3.09
2.97
1.30
1.17
4.51
1.35
4.16
4.08
f1 (ppm)