



Supporting Information

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Enantioselective Intramolecular Michael Reaction Catalyzed by *N*-Heterocyclic Carbenes

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Table of Contents

<i>General Information</i>	S2
<i>General Procedure for Intramolecular Michael Reaction</i>	S2
<i>Selected NMR Spectra</i>	S7
<i>HPLC and GC Traces of Racemic and Enantioenriched Compounds</i>	S18
<i>X-Ray Crystallography</i>	S36

General Information

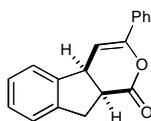
All reactions were carried out under a nitrogen atmosphere in flame-dried glassware with magnetic stirring. CH_2Cl_2 was purified by passage through a bed of activated alumina.¹ Reagents were purified prior to use unless otherwise stated following the guidelines of Perrin and Armarego.² Purification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

Enals **1a**, **1b**, **1c**, **1d**, **1e**, **9**, and **10** were prepared according to List.³ Enal **13** was prepared according to Roush.⁴

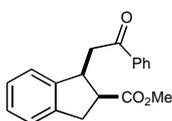
General Procedure for Intramolecular Michael Reaction

To a flame-dried 10 mL round bottom flask containing a magnetic stirring bar was added azolium salt **D** (4.2 mg, 0.01 mmol) and the corresponding enal (0.1 mmol). The flask was then sealed with rubber septum and placed under positive pressure of nitrogen. The heterogeneous mixture was then diluted with CH_2Cl_2 (2 mL, 0.05 M). Once the material dissolved, diisopropylethylamine (2 μL , 0.01 mmol) was added via syringe. The reaction stirred at 23 °C under N_2 atmosphere until complete consumption of enal (as observed by thin layer chromatography). Methanol (5 ml) was added to the reaction and stirred at 23 °C under N_2 for five hours. The mixture was partially concentrated under reduced pressure and the remaining residue was purified by silica gel chromatography (5% EtOAc/Hexanes) affording the pure annulation product.

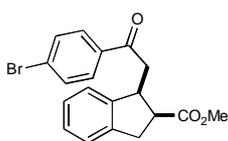
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1. Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometal.* **1996**, *15*, 1518-1520.
 2. Perrin, D. D. and Armarego, W. L. *Purification of Laboratory Chemicals*; 3rd Ed., Pergamon Press, Oxford. 1988.
 3. Yang, J.-W.; Hechavarria, M.; List, B. *J. Am. Chem. Soc.* **2005**, *127*, 15036-15037
 4. Frank, S. A.; Mergott, D. J.; Roush, W. R. *J. Am. Chem. Soc.* **2002**, *124*, 2404-2405.



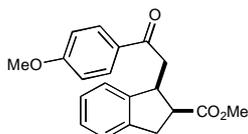
(4aS,9aS)-3-phenyl-9,9a-dihydro-4aH-2-oxa-fluoren-1-one (3): Prepared according to general procedure excluding the methanol addition using (*E*)-3-(2-((*E*)-3-oxo-3-phenylprop-1-enyl)phenyl)prop-2-enal (52 mg, 0.2 mmol) to afford 36 mg (68%) of **3** as a white solid. Methanol was not added to quench the reaction. Analytical data for **3**: IR (film) 2914, 2850, 1757, 1199, 1137, 1090, 1022, 757 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.65 (d, $J = 7.1$ Hz, 2H), 7.31 (m, 7H), 7.37 (d, $J = 4.2$ Hz, 1H), 4.25 (m, 1H), 3.54 (m, 2H), 3.42 (m, 1H); ^{13}C NMR (125 MHz, CDCl_3) 170.55, 148.62, 143.36, 132.58, 129.36, 128.71, 127.86, 127.74, 125.11, 124.98, 123.96, 100.68, 42.74, 42.42, 36.30; LRMS (ES): Mass calcd for $\text{C}_{18}\text{H}_{14}\text{O}_2$ $[\text{M}+\text{H}]^+$, 263.1. Found $[\text{M}+\text{H}]^+$, 263.1; $[\alpha]_{\text{D}}$: 57.5 (CHCl_3 , $c = 0.15$, $\text{er} = 99.5:0.5$). Enantiomeric ratio was measured by HPLC (Chiralcel AD-H, 15% IPA/Hexanes, 1 mL/min, $\text{Rt}_1 = 9.14$, $\text{Rt}_2 = 14.18$).



(1S,2S)-methyl 1-(2-oxo-2-phenylethyl)-2,3-dihydro-1H-indene-2-carboxylate (4): Prepared according to general procedure using (*E*)-3-(2-((*E*)-3-oxo-3-phenylprop-1-enyl)phenyl)prop-2-enal (52 mg, 0.2 mmol) to afford 40 mg (69%) of **4** as a colorless oil. Analytical data for **4**: IR (film) 3024, 2947, 1730, 1685, 1442, 1365 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.95 (d, $J = 7.3$ Hz, 2H), 7.57 (t, $J = 7.3$ Hz, 1H), 7.46 (t, $J = 7.6$ Hz, 2H), 7.27 (d, $J = 6.4$ Hz, 1H), 7.19 (m, 3H), 4.28 (q, $J = 7.3$ Hz, 1H), 3.59 (m, 4H), 3.42 (m, 2H), 3.15 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) 198.72, 174.72, 144.87, 137.26, 133.32, 128.84, 128.26, 127.53, 127.08, 124.81, 124.35, 51.90, 47.69, 42.75, 40.81, 34.73; LRMS (ES): Mass calcd for $\text{C}_{19}\text{H}_{18}\text{O}_3$ $[\text{M}+\text{H}]^+$, 295.3. Found $[\text{M}+\text{H}]^+$, 295.5; $[\alpha]_{\text{D}}$: -16.6 (CH_2Cl_2 , $c = 1.0$, $\text{er} = 99.5:0.5$). Enantiomeric ratio was measured by HPLC (Chiralcel AD-H, 15% IPA/Hexanes, 1 mL/min, $\text{Rt}_1 = 8.84$, $\text{Rt}_2 = 13.88$).

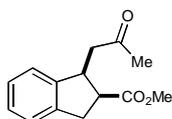


(1S,2S)-methyl 1-(2-(4-bromophenyl)-2-oxoethyl)-2,3-dihydro-1H-indene-2-carboxylate (5): Prepared according to general procedure using (*E*)-3-(2-((*E*)-3-(4-bromophenyl)-3-oxoprop-1-enyl)phenyl)prop-2-enal (34 mg, 0.1 mmol) to afford 23 mg (62%) of **5** as a colorless oil. Analytical data for **5**: IR (film) 3023, 2949, 1729, 1687, 1435, 1365 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.80 (d, $J = 8.3$ Hz, 2H), 7.59 (d, $J = 8.5$ Hz, 2H), 7.26 (d, $J = 8.5$ Hz, 1H), 7.21 (m, 3H), 4.25 (m, 1H), 3.60 (m, 4H), 3.40 (m, 2H), 3.16 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 197.76, 174.72, 144.66, 141.53, 135.94, 132.15, 129.79, 128.50, 127.62, 127.13, 124.86, 124.28, 51.96, 47.57, 42.69, 40.78, 34.74; LRMS (ES): Mass calcd for $\text{C}_{19}\text{H}_{17}\text{BrO}_3$ $[\text{M}+\text{H}]^+$, 373.2. Found $[\text{M}+\text{H}]^+$, 373.4; $[\alpha]_{\text{D}}$: -16.6 (CH_2Cl_2 , $c = 0.5$, $\text{er} = 99.5:0.5$). Enantiomeric ratio was measured by HPLC (Chiralcel AD-H, 20% IPA/Hexanes, 1 mL/min, $\text{Rt}_1 = 8.10$, $\text{Rt}_2 = 9.40$).



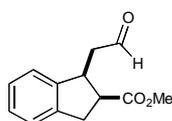
(1S,2S)-methyl 1-(2-(4-methoxyphenyl)-2-oxoethyl)-2,3-dihydro-1H-indene-2-carboxylate (6): Prepared according to general procedure using (*E*)-3-(2-((*E*)-3-(4-methoxyphenyl)-3-oxoprop-1-enyl)phenyl)prop-2-enal (29 mg, 0.1 mmol) to afford 26 mg (80%)

of **6** as a yellow oil. Analytical data for **6**: IR (film) 3006, 2949, 2841, 1730, 1674, 1600, 1508 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.92 (d, $J = 7.8$ Hz, 2H), 7.25 (m, 1H), 7.20 (m, 3H), 6.92 (d, $J = 8.9$ Hz, 2H), 4.26 (m, 1H), 3.87 (s, 3H), 3.59 (m, 4H), 3.37 (m, 2H), 3.10 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 197.19, 174.74, 163.71, 144.98, 141.58, 130.53, 130.37, 127.46, 127.04, 113.95, 55.17, 51.90, 47.73, 42.86, 40.37, 34.68; LRMS (ES): Mass calcd for $\text{C}_{20}\text{H}_{20}\text{O}_4$ $[\text{M}+\text{H}]^+$, 325.4. Found $[\text{M}+\text{H}]^+$, 325.5; $[\alpha]_{\text{D}}$: -28.9 (CH_2Cl_2 , $c = 1.0$, $\text{er} = 99.5:0.5$). Enantiomeric ratio was measured by HPLC (Chiralcel AD-H, 15% IPA/Hexanes, 1 mL/min, $\text{Rt}_1 = 11.41$, $\text{Rt}_2 = 14.05$).



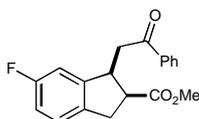
(1S,2S)-methyl 1-(2-oxopropyl)-2,3-dihydro-1H-indene-2-carboxylate

(7): Prepared according to general procedure using (*E*)-3-(2-((*E*)-3-oxobut-1-enyl)phenyl)prop-2-enal (20 mg, 0.1 mmol) to afford 14 mg (59%) of **7** as a colorless oil. Analytical data for **7**: IR (film) 3020, 2950, 1727, 1435, 1365 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.20 (m, 4H), 4.35 (m, 1H), 3.68 (s, 3H), 3.49 (q, $J = 8.3$, 1H), 3.29 (dd, $J = 15.9, 8.9$ Hz, 1H), 3.06 (dd, $J = 15.9, 7.9$ Hz, 1H), 2.86 (dd, $J = 18.1, 8.3$ Hz, 1H), 2.63 (dd, $J = 11.9, 6.1$ Hz, 1H), 2.13 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 207.33, 174.75, 144.75, 141.43, 127.49, 127.10, 124.78, 124.15, 51.94, 47.44, 45.58, 42.44, 34.60, 30.68; LRMS (ES): Mass calcd for $\text{C}_{14}\text{H}_{16}\text{O}_3$ $[\text{M}+\text{H}]^+$, 233.3. Found $[\text{M}+\text{H}]^+$, 233.5; $[\alpha]_{\text{D}}$: -26.4 (CH_2Cl_2 , $c = 1.0$, $\text{er} = 99.5:0.5$). Enantiomeric ratio was measured by HPLC (Chiralcel AD-H, 1% IPA/Hexanes, 0.5 mL/min, $\text{Rt}_1 = 23.39$, $\text{Rt}_2 = 25.01$).



(1S,2S)-methyl 1-(2-oxoethyl)-2,3-dihydro-1H-indene-2-carboxylate

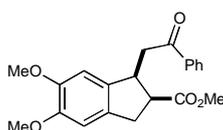
(8): Prepared according to general procedure using (*2E,2'E*)-3,3'-(1,2-phenylene)diprop-2-enal (37 mg, 0.2 mmol) to afford 29 mg (68%) of **8** as a colorless oil. Analytical data for **8**: IR (film) 3023, 2922, 1727, 1436, 1367 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 9.80 (s, 1H), 7.24 (m, 4H), 4.07 (m, 1H), 3.70 (s, 3H), 3.53 (q, $J = 8.2$ Hz, 1H), 3.35 (dd, $J = 15.9, 8.8$ Hz, 1H), 3.09 (dd, $J = 15.9, 8.2$ Hz, 1H), 2.82 (dd, $J = 7.6, 0.9$ Hz, 1H), 2.65 (dd, $J = 17.7, 6.4$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 200.87, 174.24, 144.19, 141.35, 127.70, 127.21, 124.91, 124.20, 51.99, 47.63, 46.11, 41.40, 34.33; LRMS (ES): Mass calcd for $\text{C}_{13}\text{H}_{14}\text{O}_3$ $[\text{M}+\text{H}]^+$, 219.3. Found $[\text{M}+\text{H}]^+$, 219.6; $[\alpha]_{\text{D}}$: -34.8 (CH_2Cl_2 , $c = 1.0$, $\text{er} = 99.5:0.5$). Enantiomeric ratio was measured by HPLC (Chiralcel AD-H, 3% IPA/Hexanes, 1 mL/min, $\text{Rt}_1 = 8.33$, $\text{Rt}_2 = 9.14$).



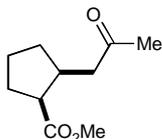
(1S,2S)-methyl 6-fluoro-1-(2-oxo-2-phenylethyl)-2,3-dihydro-1H-indene-2-carboxylate

(10): Prepared according to general procedure using (*2E,2'E*)-3,3'-(4-fluoro-1,2-phenylene)diprop-2-enal (56 mg, 0.2 mmol) to afford 42 mg (68%) of **10** as a colorless oil. Analytical data for **10**: IR (film) 3060, 2951, 2851, 1732, 1685, 1597, 1436, 1230, 1168, 813, 753, 690 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.93 (d, $J = 7.9$ Hz, 2H), 7.56 (t, $J = 7.6$ Hz, 1H), 7.45 (t, $J = 7.6$ Hz, 2H), 7.16 (m, 2H), 6.88 (m, 2H), 4.23 (q, $J = 7.3$ Hz, 1H), 3.62 (q, $J = 7.9$ Hz, 1H), 3.56 (s, 3H), 3.40 (dd, $J = 17.7, 17.7$ Hz, 1H), 3.30 (dd, $J = 15.6, 7.9$ Hz, 1H), 3.14 (dd, $J = 17.7, 6.4$ Hz, 1H), 3.06 (dd, $J = 15.6, 7.9$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 198.34, 174.38, 163.40, 161.46, 146.98, 146.92, 137.08, 136.90, 133.45,

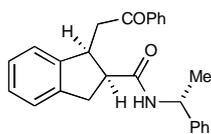
128.24, 125.69, 125.63, 114.47, 114.29, 111.68, 111.50, 51.95, 48.20, 42.68, 40.42, 33.90; LRMS (ES): Mass calcd for $C_{19}H_{17}FO_3$ $[M+H]^+$, 313.3. Found $[M+H]^+$, 313.4; $[\alpha]_D$: -21.9 (CH_2Cl_2 , $c = 1.0$, $er = 99.5:0.5$). Enantiomeric ratio was measured by HPLC (Chiralcel OD-H, 2.5% EtOH/2.5% IPA/95% Hexanes, 1 mL/min, $R_{t1} = 8.80$, $R_{t2} = 9.54$).



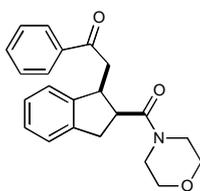
(1S,2S)-methyl 5,6-dimethoxy-1-(2-oxo-2-phenylethyl)-2,3-dihydro-1H-indene-2-carboxylate (12): Prepared according to general procedure using (2*E*,2'*E*)-3,3'-(4,5-dimethoxy-1,2-phenylene)diprop-2-enal (32 mg, 0.1 mmol) to afford 26 mg (73%) of **12** as a yellow solid. Analytical data for **12**: IR (film) 3061, 2948, 1729, 1684, 1563, 1449 cm^{-1} ; 1H NMR (500 MHz, $CDCl_3$) δ 7.92 (d, $J = 7.3$ Hz, 2H), 7.55 (m, 1H), 7.45 (m, 2H), 6.78 (s, 1H), 6.74 (s, 1H), 4.20 (m, 1H), 3.87 (s, 3H), 3.80 (s, 3H), 3.60 (m, 4H), 3.35 (m, 2H), 3.12 (dd, $J = 17.4, 6.7$ Hz, 1H), 3.05 (dd, $J = 15.5, 8.3$ Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 199.04, 174.64, 148.92, 148.49, 137.33, 136.39, 133.34, 133.00, 128.25, 107.82, 107.74, 56.27, 56.25, 51.92, 48.24, 42.84, 40.91, 34.46; LRMS (ES): Mass calcd for $C_{21}H_{22}O_5$ $[M+H]^+$, 355.4. Found $[M+H]^+$, 355.4; $[\alpha]_D$: -9.3 (CH_2Cl_2 , $c = 1.0$, $er = 99.5:0.5$). Enantiomeric ratio was measured by HPLC (Chiralcel AD-H, 15% IPA/Hexanes, 1 mL/min, $R_{t1} = 8.29$, $R_{t2} = 10.99$).



(1S,2S) methyl 2-(2-oxopropyl)cyclopentanecarboxylate (14): To a flame-dried 50 mL round bottom flask was added azolium salt **D** (46 mg, 0.11 mmol). The flask was then sealed with rubber septum and put N_2 atmosphere. A mixture of (2*E*,6*E*)-8-oxonona-2,6-dienal (80 mg, 0.53 mmol) and CH_2Cl_2 (10 mL, 0.05 M) was added via syringe. Diisopropylethylamine (18.6 μL , 0.11 mmol) was then added via syringe. The reaction stirred at 23 $^\circ C$ under N_2 atmosphere. Methanol (20 ml) and diisopropylethylamine (0.53 mmol) were added to the reaction after six hours. The reaction was then stirred under nitrogen at 23 $^\circ C$ for 12 hr. The mixture was then taken up in pentane and washed with water. The layers were then separated. The aqueous layer was then extracted with pentane (2 x 20 ml). The combined organic layers were dried with Na_2SO_4 , filtered, and condensed under reduced pressure. The mixture was then purified by flash chromatography on silica gel (25% ether/pentane) to afford 64 mg (66%) of **14** as a yellow oil. Analytical data for **14**: IR (film) 2953, 2872, 1730, 1721, 1435, 1361, 1168 cm^{-1} ; 1H NMR (500 MHz, $CDCl_3$) δ 3.66 (s, 3H), 2.93(m, 1H), 2.61 (m, 2H), 2.41 (m, 1H), 2.14 (s, 3H), 1.90 (m, 4H), 1.54 (m, 1H), 1.32 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 208.14, 176.06, 51.51, 46.64, 45.36, 38.37, 31.63, 30.50, 28.62, 23.83; LRMS (ES): Mass calcd for $C_{10}H_{16}O_3$ $[3M+Na]^+$, 575.3. Found $[3M+Na]^+$, 575.8; $[\alpha]_D$: 42.2 ($CHCl_3$, $c = 0.5$, $er = 99.5:0.5$). Enantiomeric ratio was measured by GC (Beta Dex 225, 23.00 psi, 80 $^\circ C$ – 170 $^\circ C$, $R_{t1} = 33.81$, $R_{t2} = 34.05$).

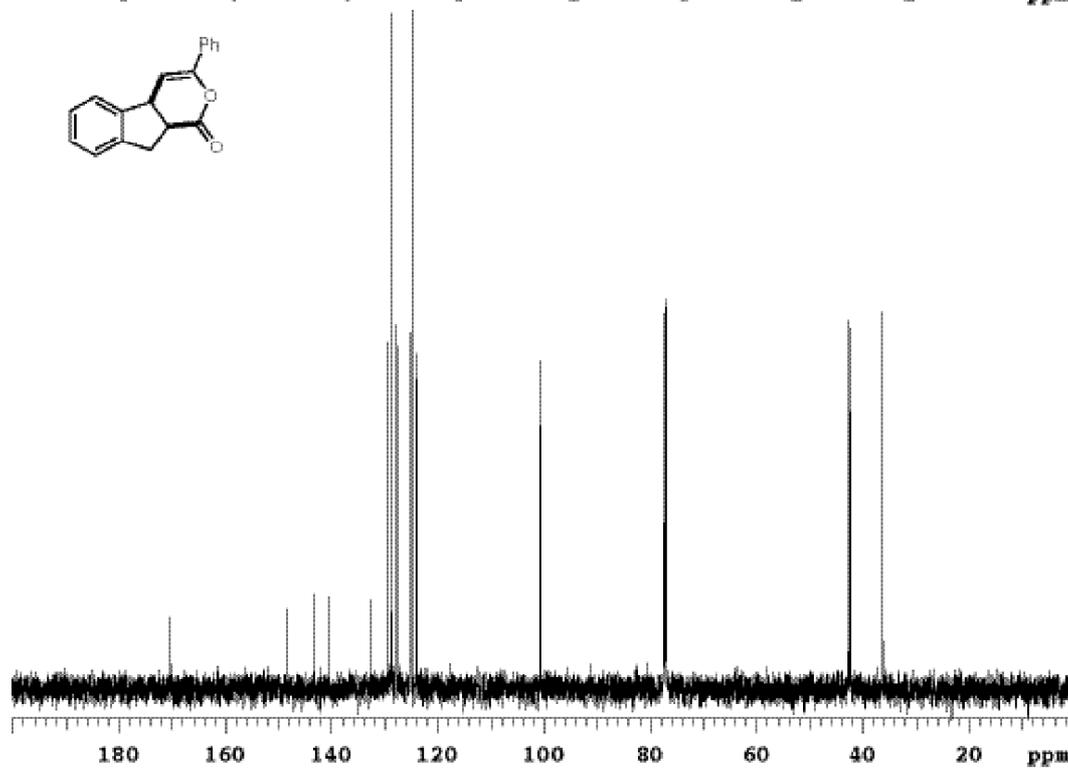
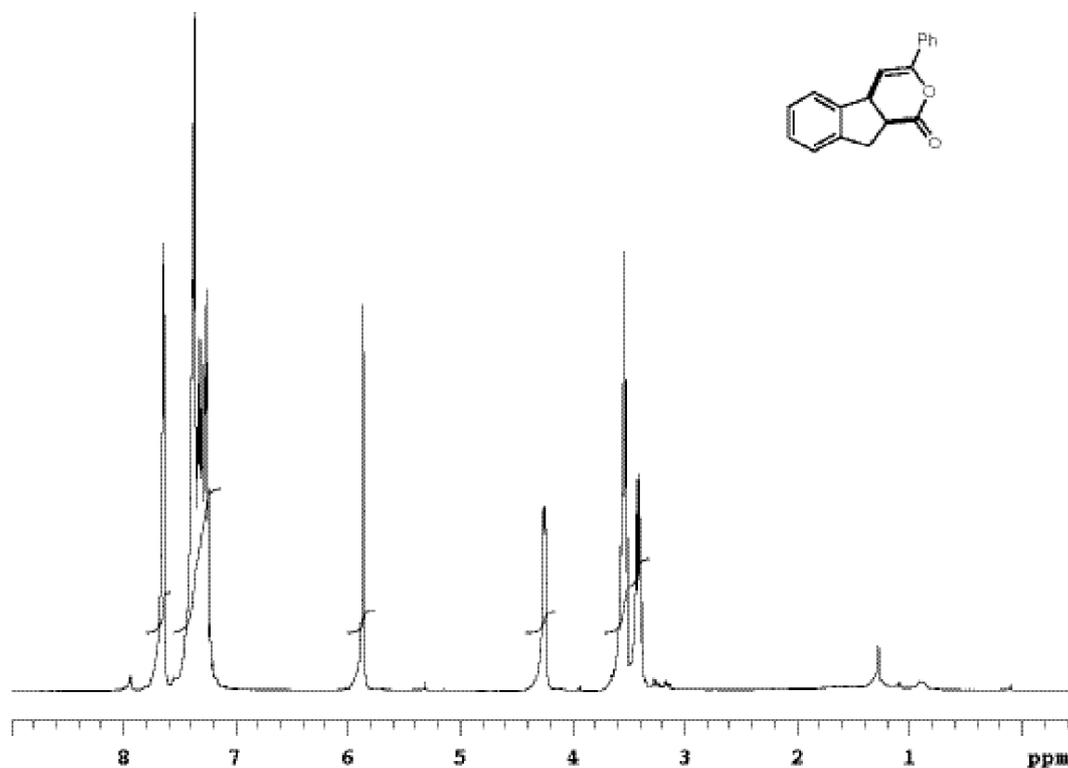


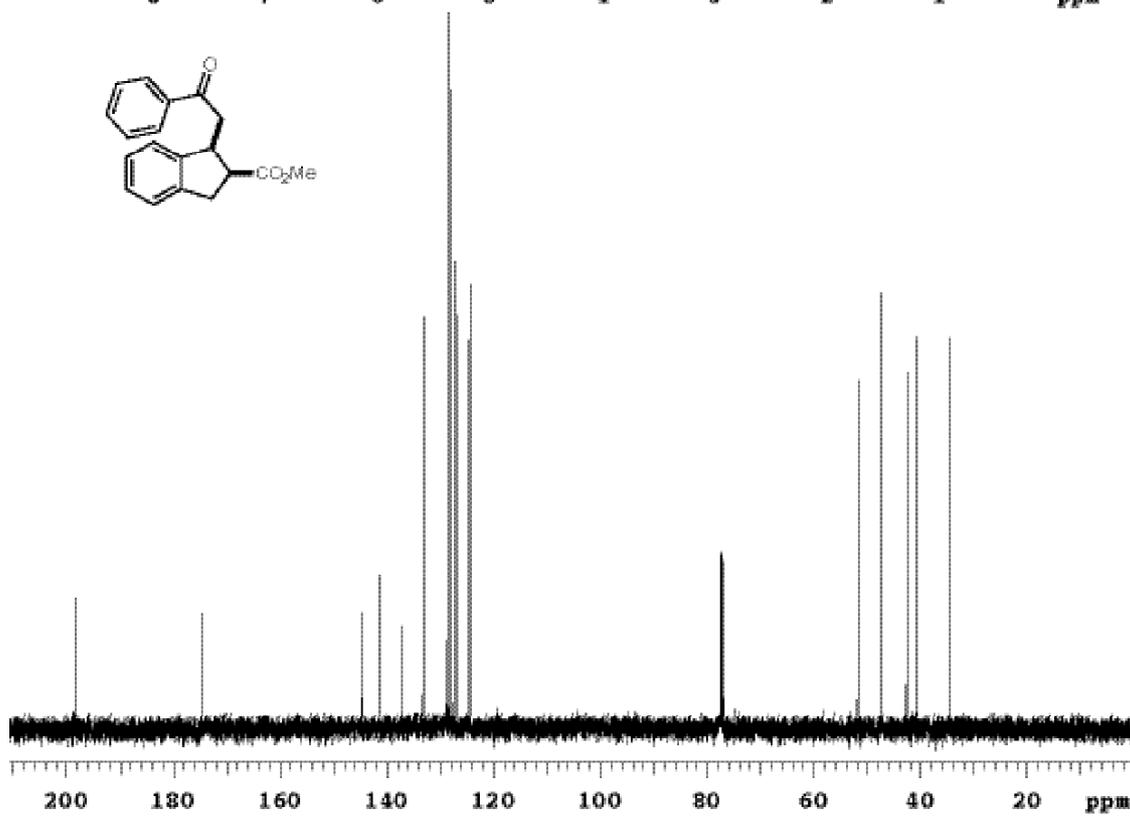
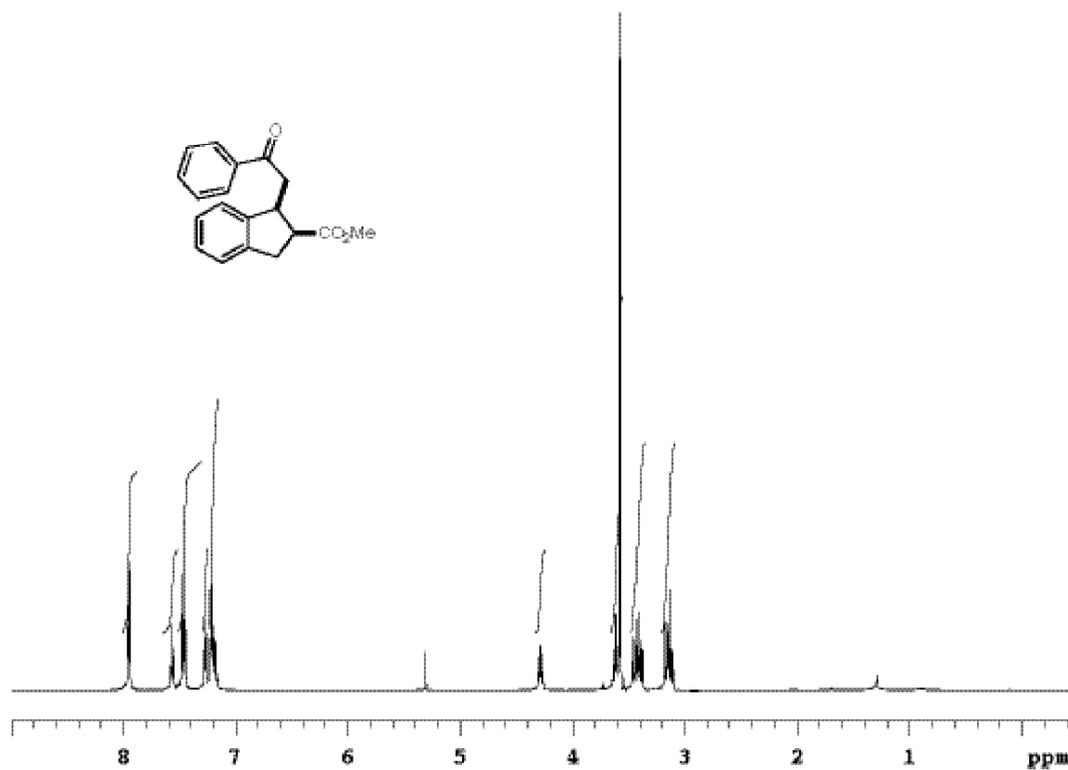
(1S,2S)-1-(2-oxo-2-phenylethyl)-N-((R)-1-phenylethyl)-2,3-dihydro-1H-indene-2-carboxamide (17): Prepared according to general procedure using (*E*)-3-(2-((*E*)-3-oxo-3-phenylprop-1-enyl)phenyl)prop-2-enal (52 mg, 0.2 mmol) to afford 70 mg (68%) of **17** as a white solid. Upon consumption of enal, (*S*)- α -methyl benzylamine (3 equiv.) was added via syringe to the reaction flask after 3 hr. The reaction stirred at 23 °C for an additional 6 hr. Analytical data for **17**: IR (film) 3315, 3062, 3029, 2974, 2928, 1682, 1640, 1534, 1239, 1215, 1000, 747, 691 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.78 (d, $J = 7.7$ Hz, 2H), 7.55 (t, $J = 7.3$ Hz, 1H), 7.40 (t, $J = 7.5$ Hz, 2H), 7.26 (d, $J = 7.7$ Hz, 1H), 7.15 (m, 6H), 7.06 (m, 2H), 6.06 (d, $J = 7.3$ Hz, 1H), 4.98 (m, 1H), 4.12 (m, 1H), 3.42 (m, 2H), 3.21 (m, 2H), 3.09 (m, 1H) 1.43 (d, $J = 6.9$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 199.67, 172.61, 144.93, 143.36, 142.34, 137.03, 133.27, 128.71, 128.33, 127.39, 127.37, 126.84, 126.38, 124.69, 124.02, 49.38, 49.22, 42.87, 40.47, 35.13, 22.04; LRMS (electrospray): Mass calcd for $\text{C}_{26}\text{H}_{25}\text{NO}_2$ $[\text{M}+\text{H}]^+$, 384.2. Found $[\text{M}+\text{H}]^+$, 384.7; $[\alpha]_{\text{D}}$: -100.0 (MeOH, $c = 0.3$, $er = 99.5:0.5$).

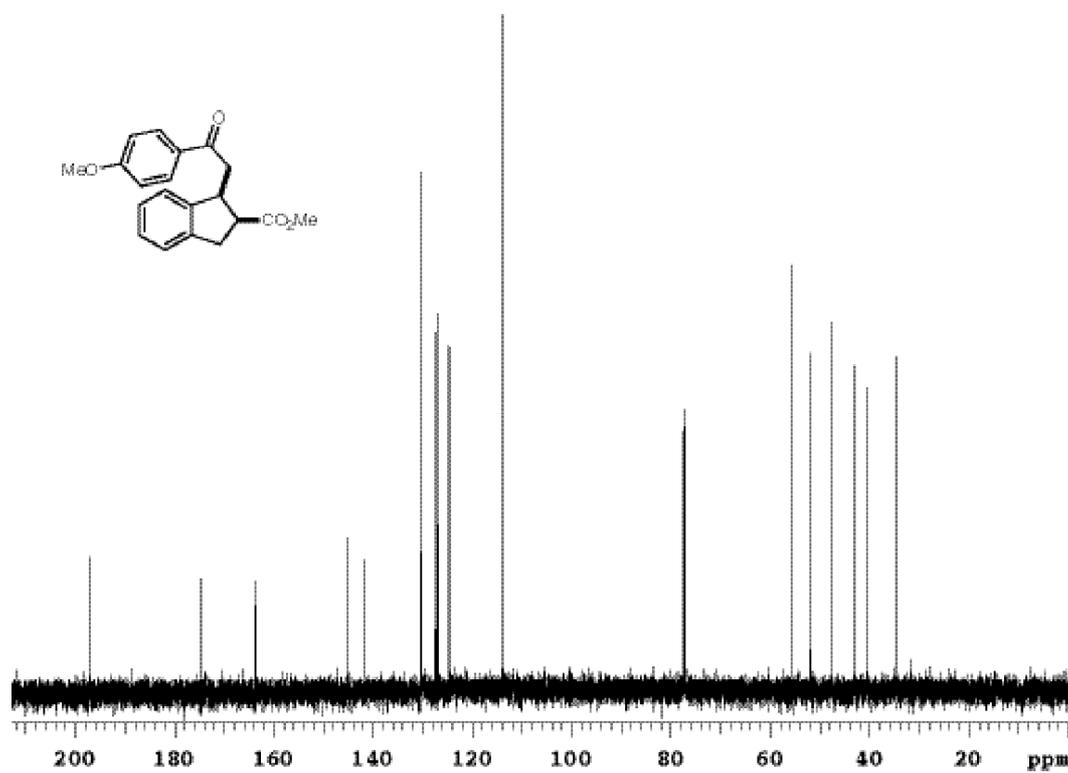
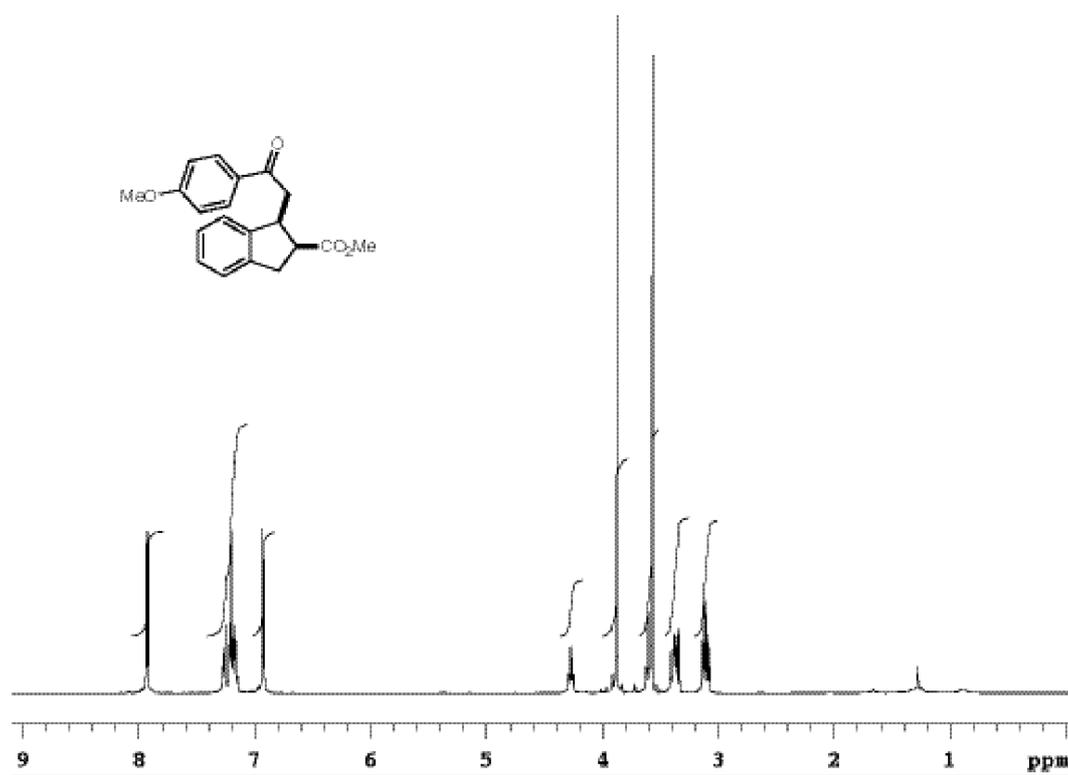


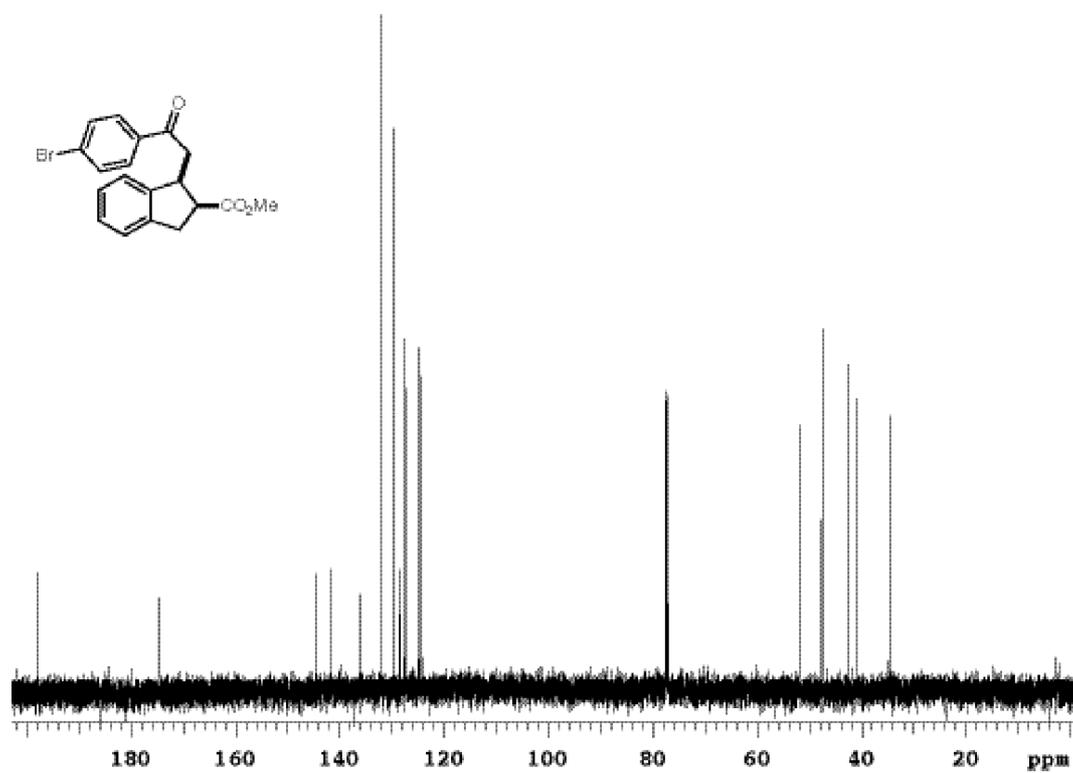
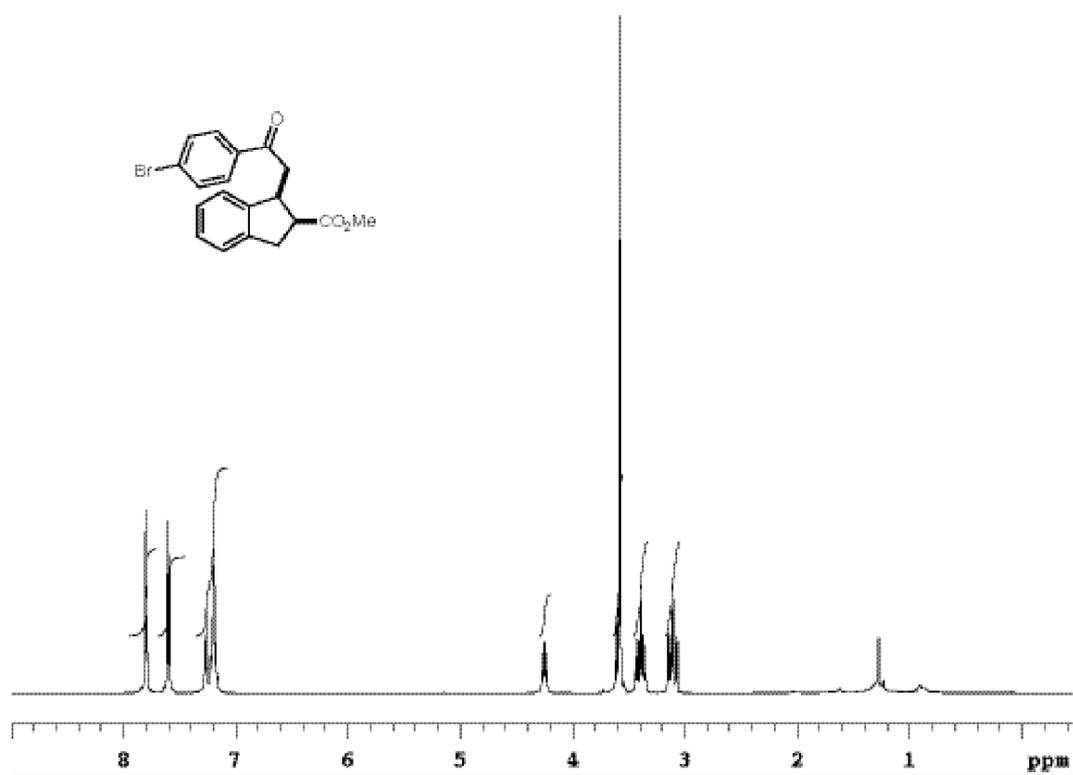
2-((1S,2S)-2-(morpholine-4-carbonyl)-2,3-dihydro-1H-inden-1-yl)-1-phenylethanone (18): Prepared according to general procedure using (*E*)-3-(2-((*E*)-3-oxo-3-phenylprop-1-enyl)phenyl)prop-2-enal (52 mg, 0.2 mmol) to afford 49 mg (70%) of **18** as a white solid. Upon consumption of the enal, morpholine (5 equiv.) was added via syringe to the reaction flask after 3 hr. The reaction stirred at room temperature for an additional 3 hr. Solvent was then removed *in vacuo*. The mixture was purified via flash chromatography on silica gel (15% EtOAc/Hexanes). Analytical data for **18**: IR (film) 3062, 2960, 2586, 1682, 1634, 1442, 1230, 1115 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.92 (d, $J = 7.6$ Hz, 2H), 7.54 (m, 1H), 7.43 (m, 2H), 7.27 (d, $J = 4.9$ Hz, 1H), 7.20 (m, 3H), 4.21 (m, 1H), 3.60 (m, 11H), 2.94 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 198.75, 171.57, 145.28, 141.95, 137.10, 133.36, 128.77, 127.43, 124.76, 124.22, 66.84, 66.62, 46.30, 44.88, 42.15, 42.09, 40.81, 35.00; LRMS (ES): Exact mass calcd for $\text{C}_{22}\text{H}_{23}\text{NO}_3$ $[\text{2M}+\text{Na}]^+$, 721.3. Found $[\text{2M}+\text{Na}]^+$, 721.9; $[\alpha]_{\text{D}}$: -72.4 (CH_2Cl_2 , $c = 1.0$, $er = 99.5:0.5$).

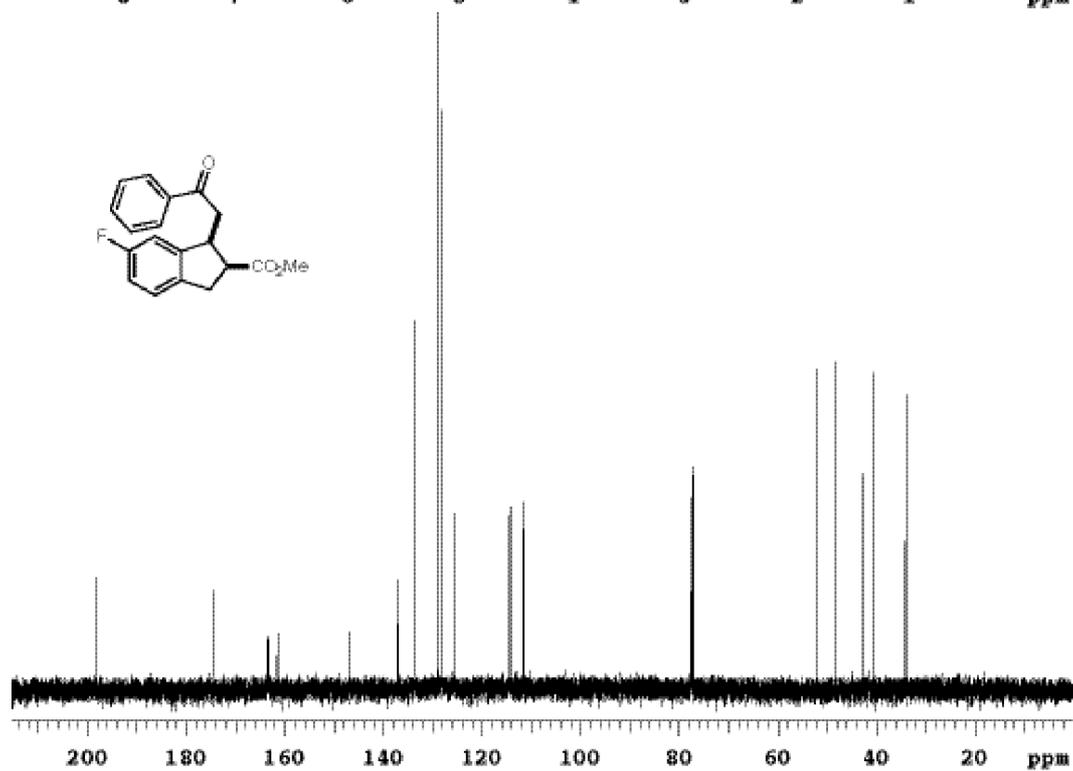
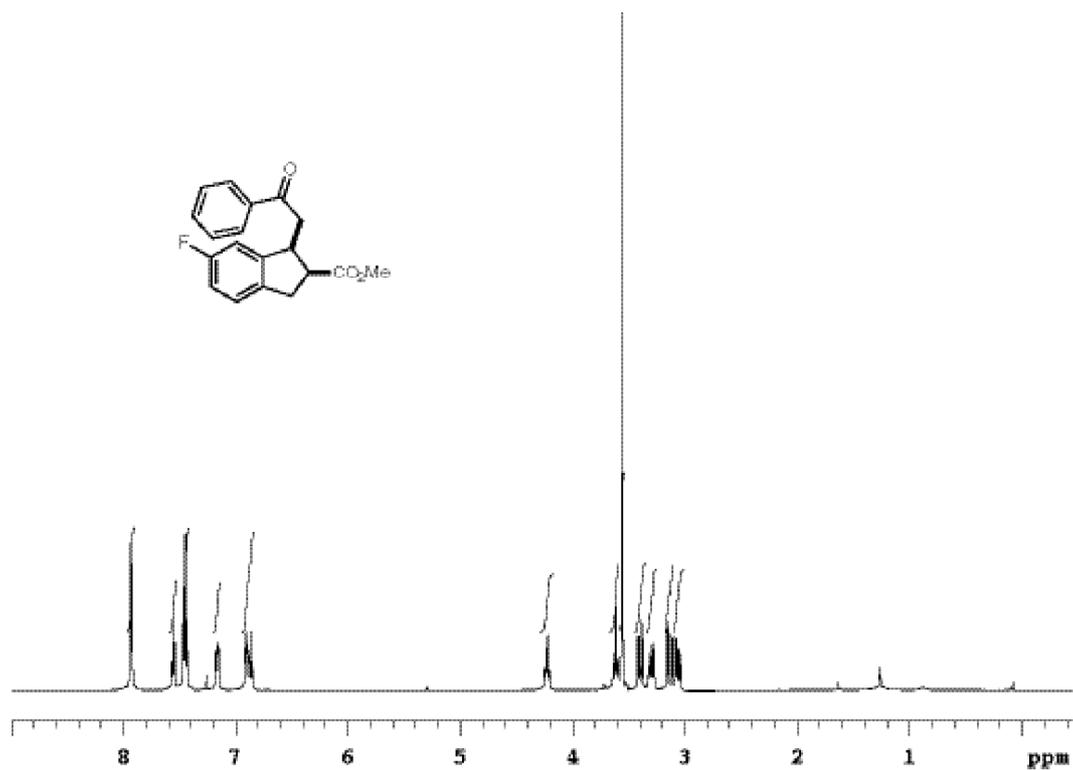
Selected NMR Spectra

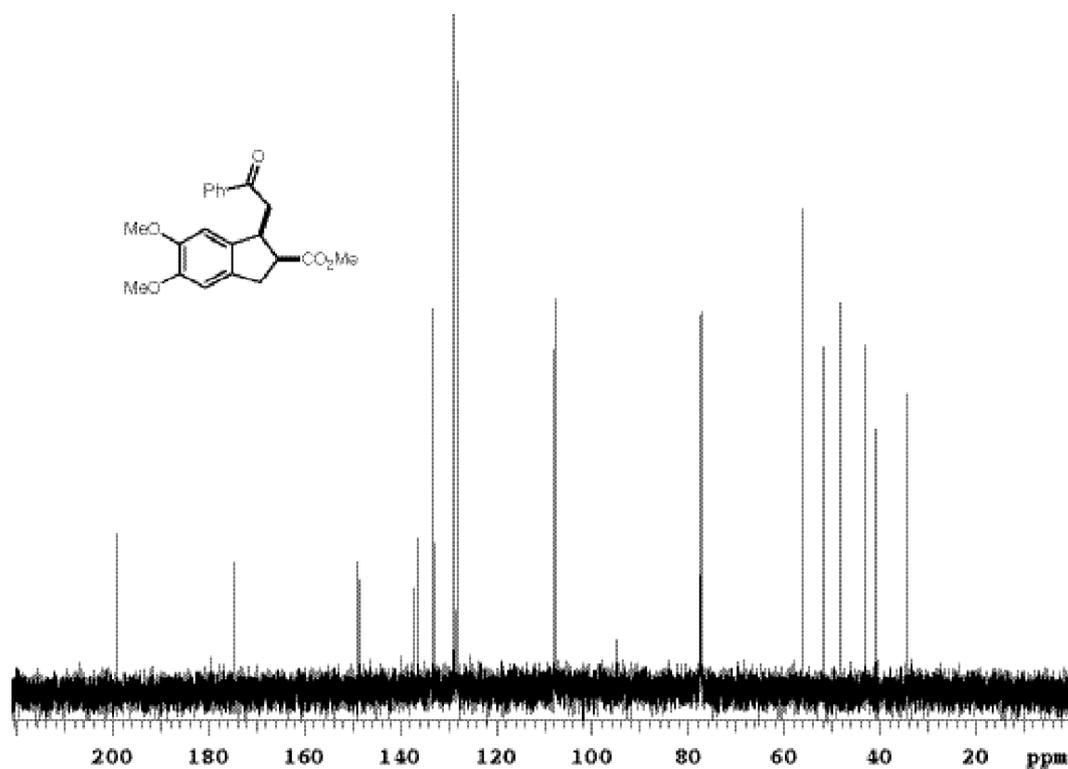
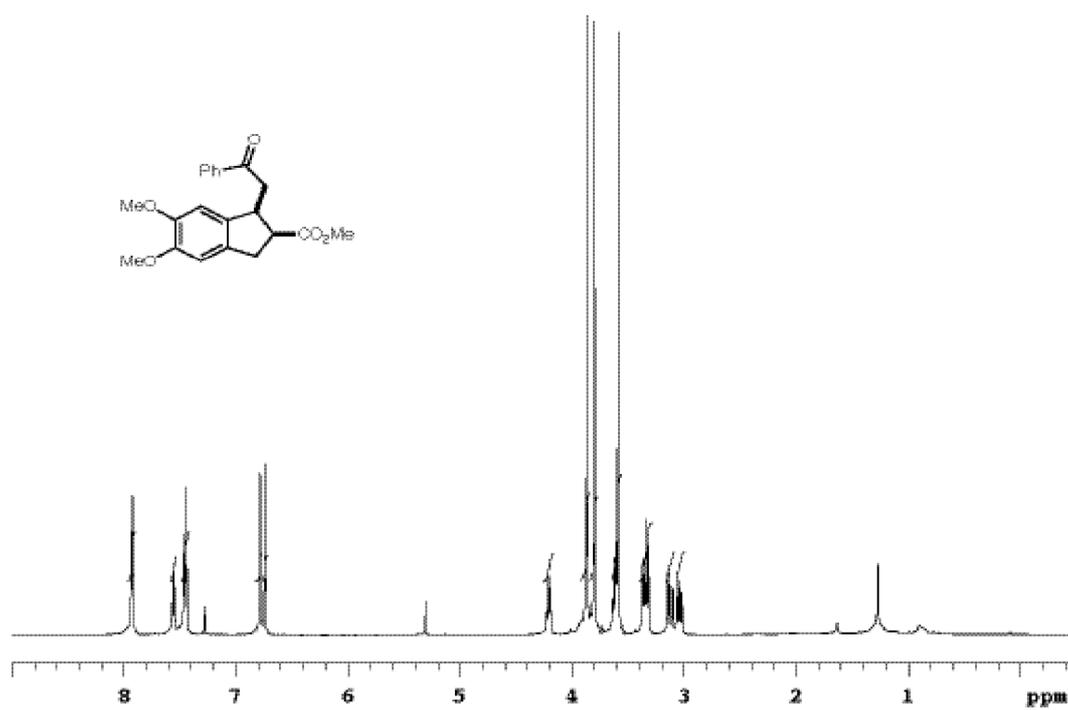


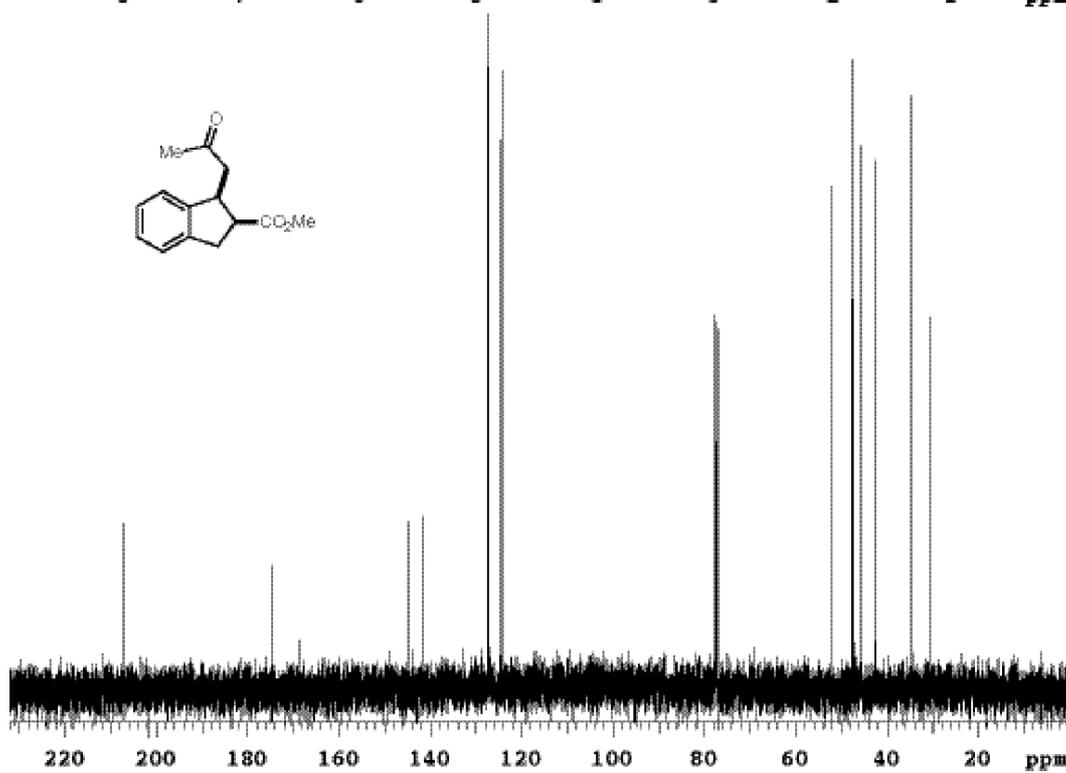
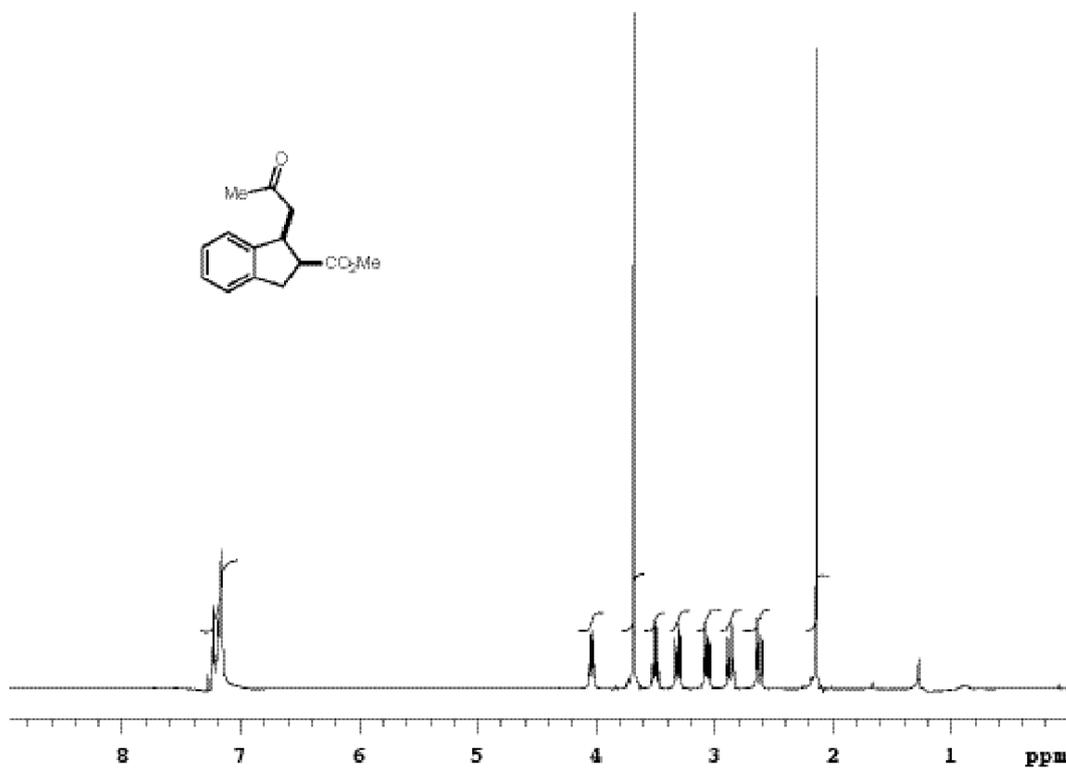


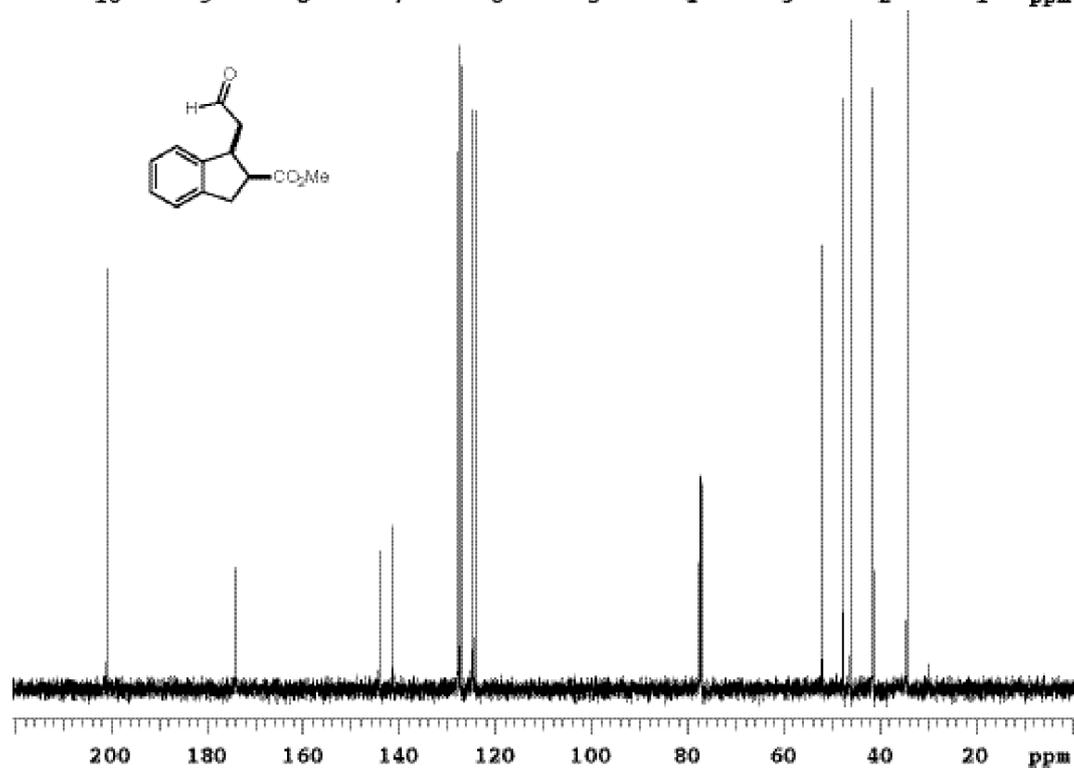
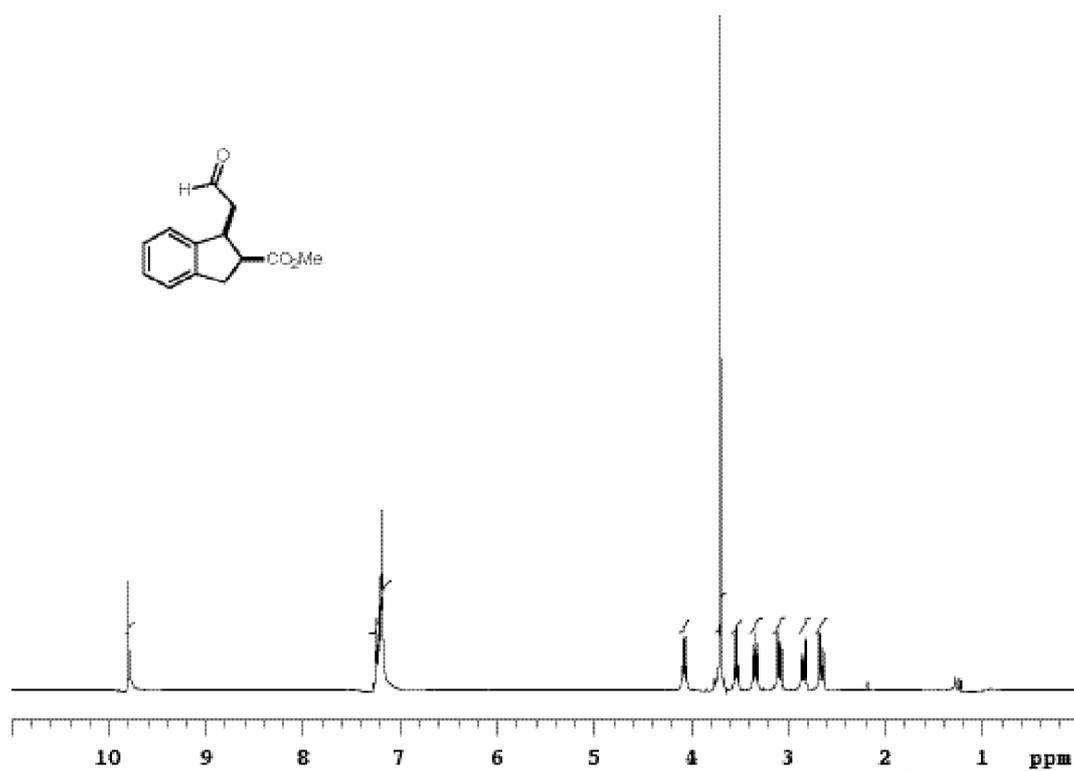


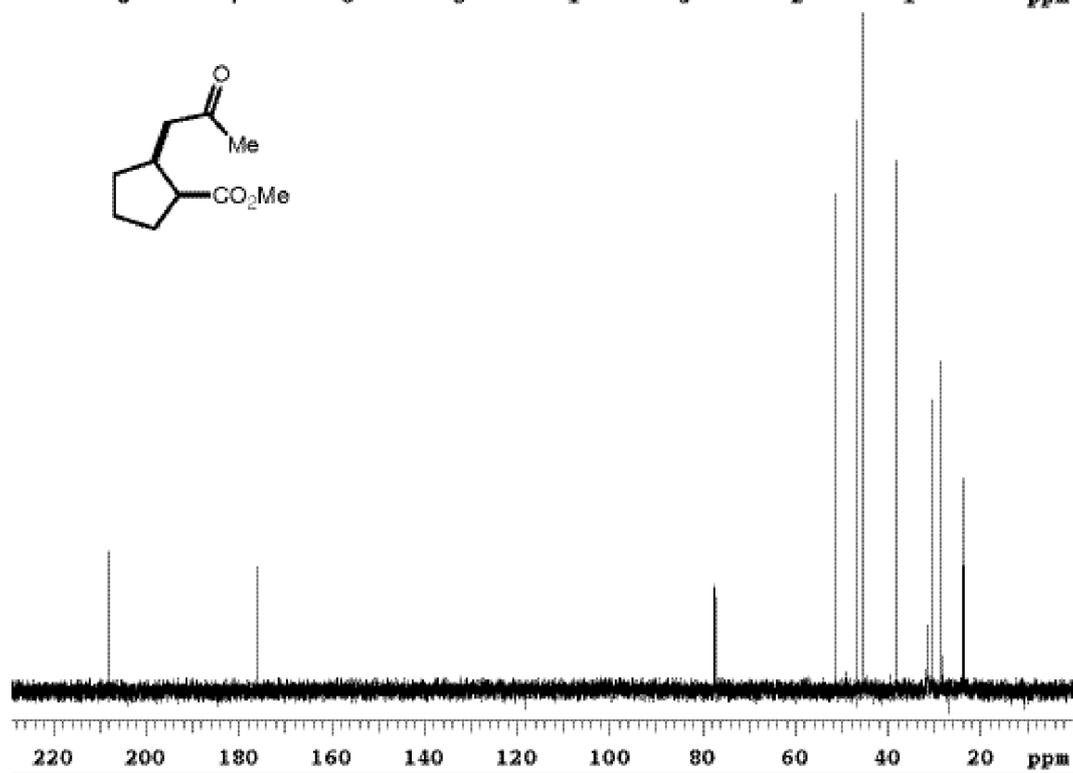
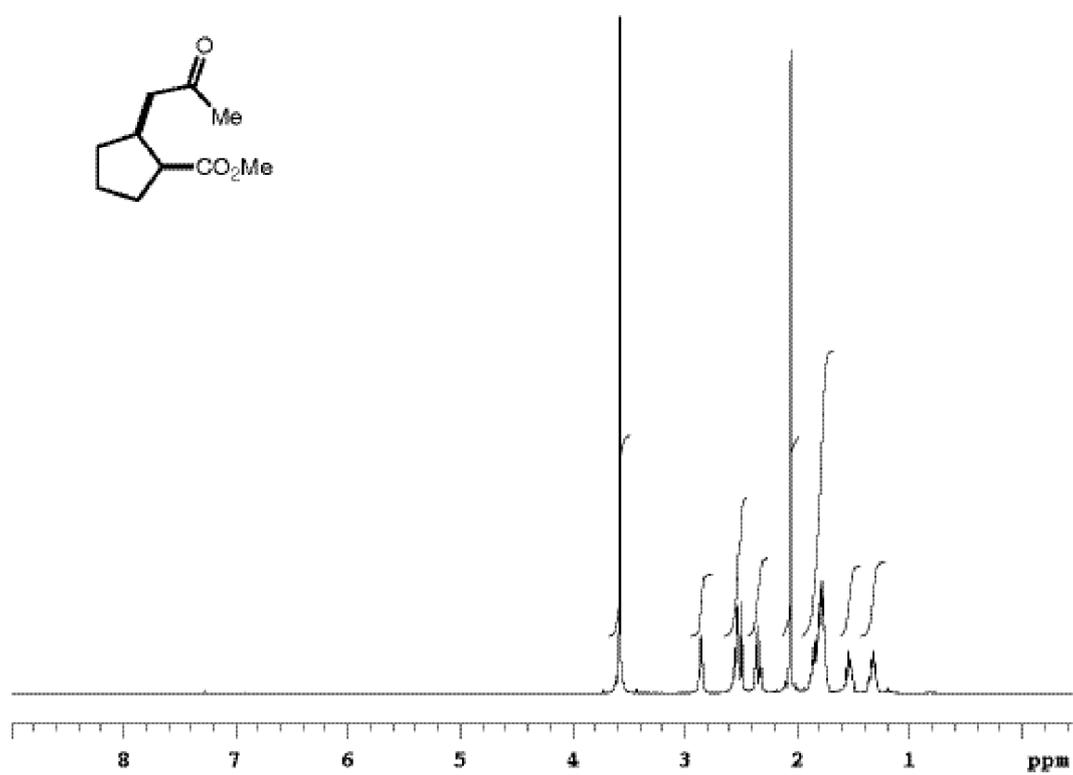


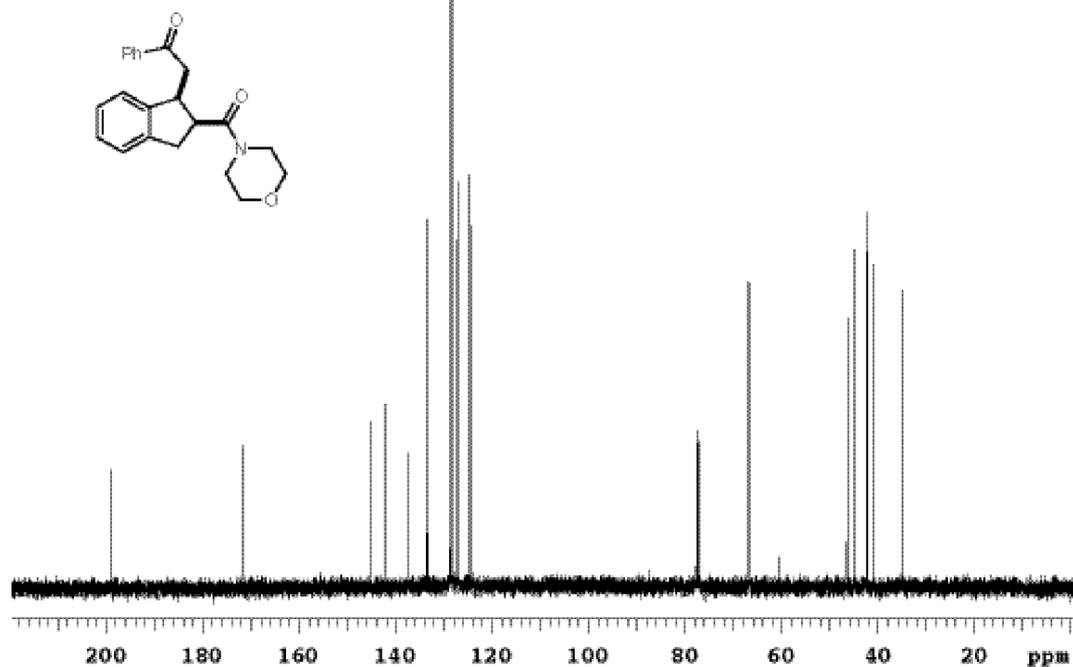
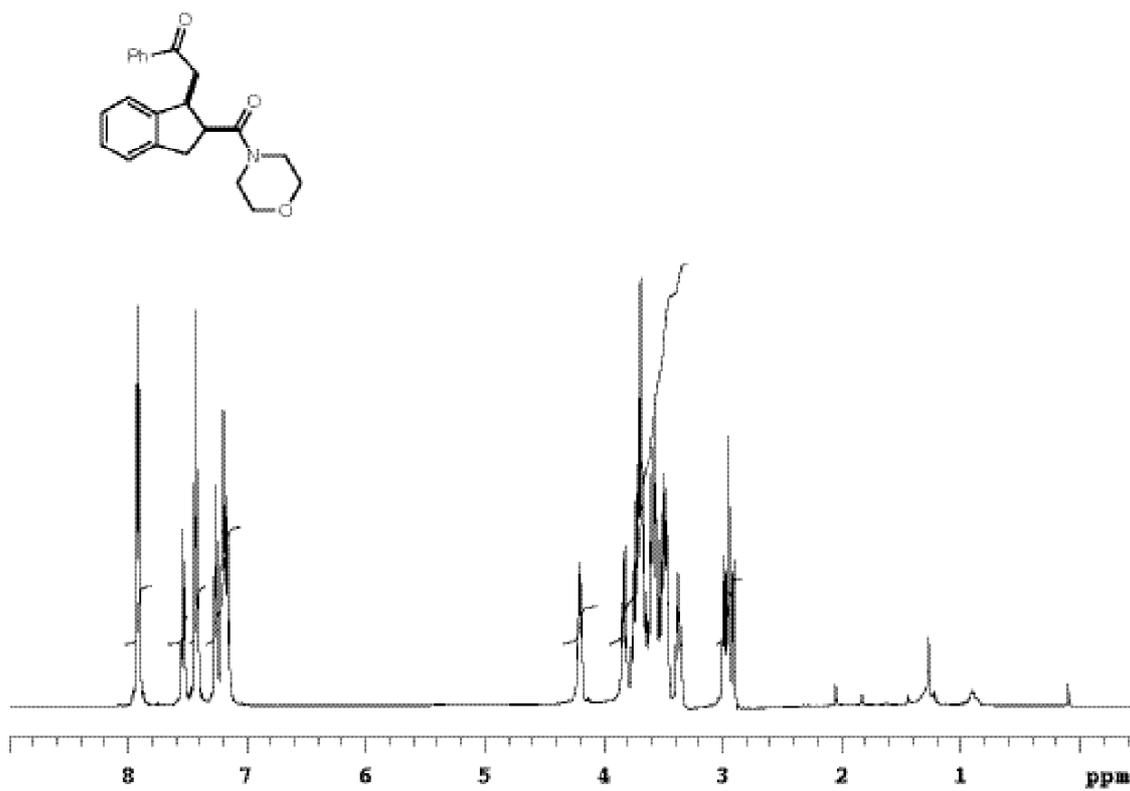


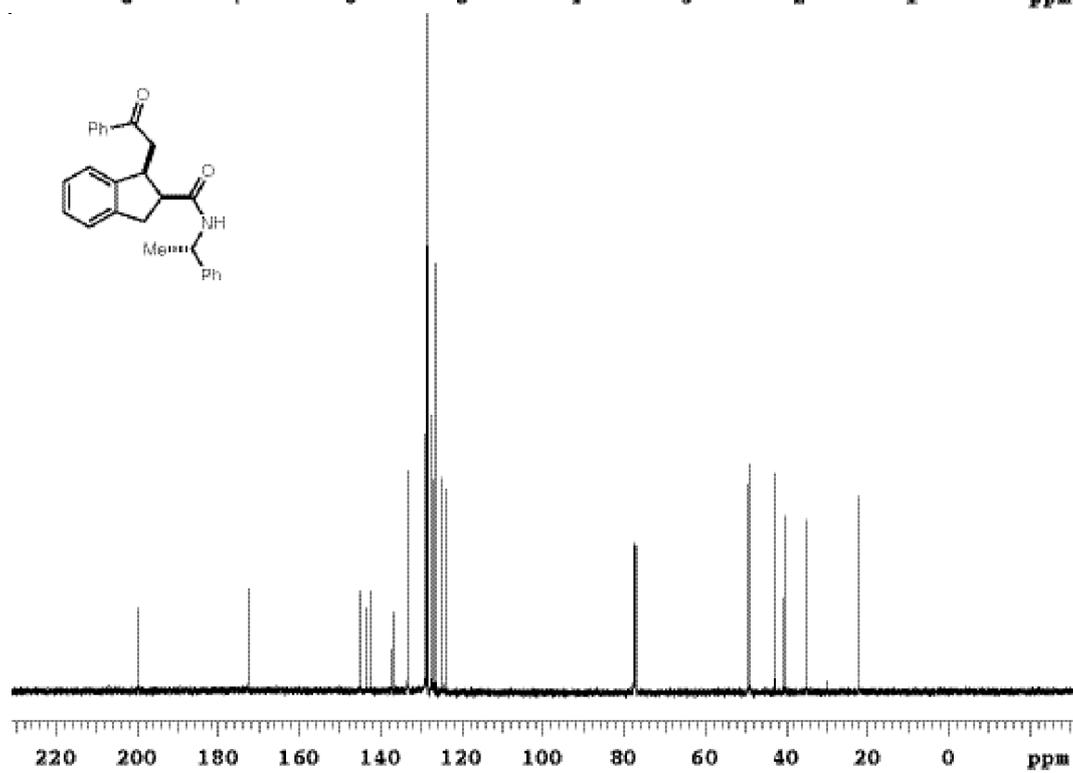
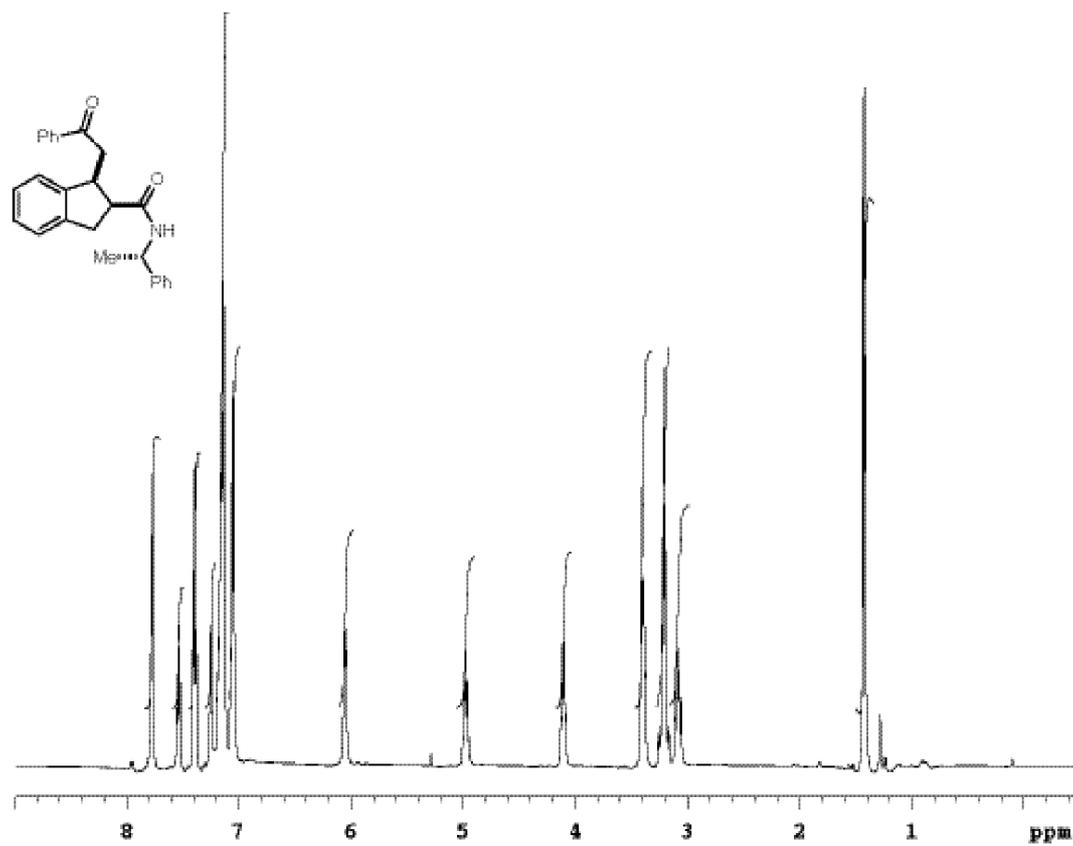












HPLC and GC Traces of Racemic and Enantioenriched Compounds

Racemic 3

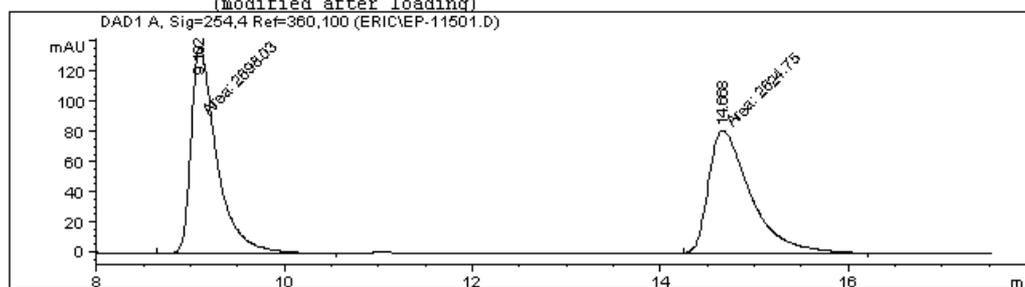
Data File C:\HPCHEM\2\DATA\ERIC\EP-11501.D

Sample Name: EP2-115

racemic

```

=====
Injection Date : 9/18/2006 4:21:21 PM
Sample Name    : EP2-115
Acq. Operator  : Eric
Location       : Vial 41
Inj Volume    : 5 µl
Acq. Method    : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 9/18/2006 4:21:42 PM by Eric
                (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 11/8/2006 1:11:17 AM by MANABU
                (modified after loading)
    
```



Area Percent Report

```

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

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.102	MM	0.3262	2698.03345	137.85426	50.6884
2	14.668	MM	0.5318	2624.75024	82.25320	49.3116

Totals : 5322.78369 220.10747

Results obtained with enhanced integrator!

*** End of Report ***

Enantioenriched 3

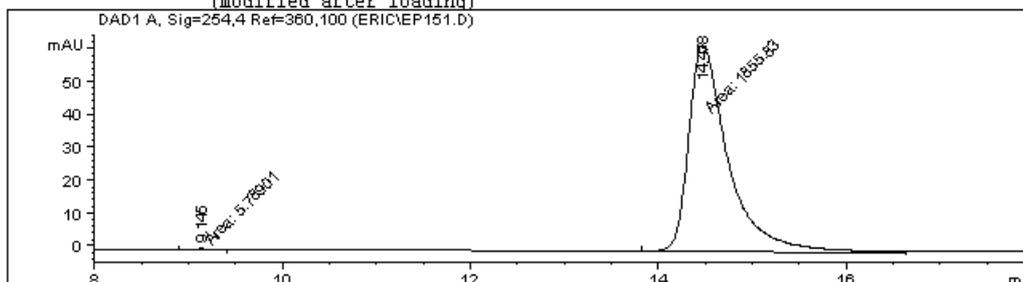
Data File C:\HPCHEM\2\DATA\ERIC\EP151.D

Sample Name: EP-02-151

chiral

```

=====
Injection Date : 10/2/2006 8:36:28 AM
Sample Name    : EP-02-151                Location : Vial 41
Acq. Operator  : mmb                      Inj Volume : 5 µl
Acq. Method    : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 10/2/2006 8:26:26 AM by mmb
                (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 11/8/2006 1:10:55 AM by MANABU
                (modified after loading)
    
```



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

```

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

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.145	MM	0.3217	5.78901	2.99910e-1	0.3110
2	14.478	MM	0.4919	1855.82959	62.87340	99.6890

Totals : 1861.61860 63.17331

Results obtained with enhanced integrator!

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

Racemic 4

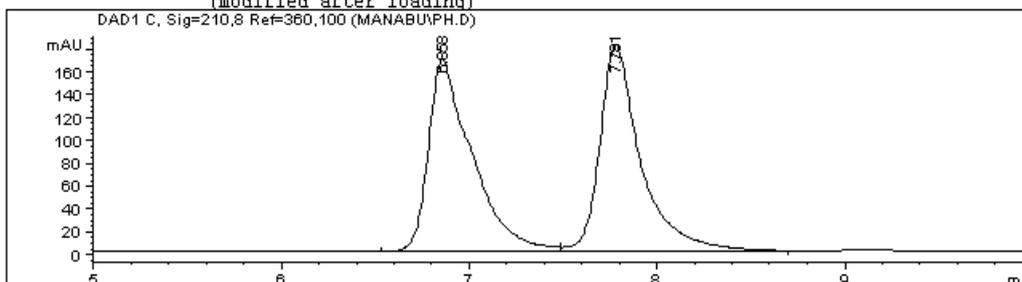
Data File C:\HPCHEM\2\DATA\MANABU\PH.D

Sample Name: Ph

AD-H 15%

```

=====
Injection Date : 11/7/2006 1:43:14 PM
Sample Name    : Ph                               Location : Vial 1
Acq. Operator  : MANABU                          Inj Volume : 20 µl
Acq. Method    : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 11/7/2006 1:50:58 PM by MANABU
                (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 11/7/2006 1:57:43 PM by MANABU
                (modified after loading)
    
```



Area Percent Report

```

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

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.858	VV	0.2233	2700.54321	168.21289	49.6053
2	7.781	VB	0.2192	2743.51733	180.64406	50.3947

Totals : 5444.06055 348.85695

Results obtained with enhanced integrator!

*** End of Report ***

Enantioenriched 4

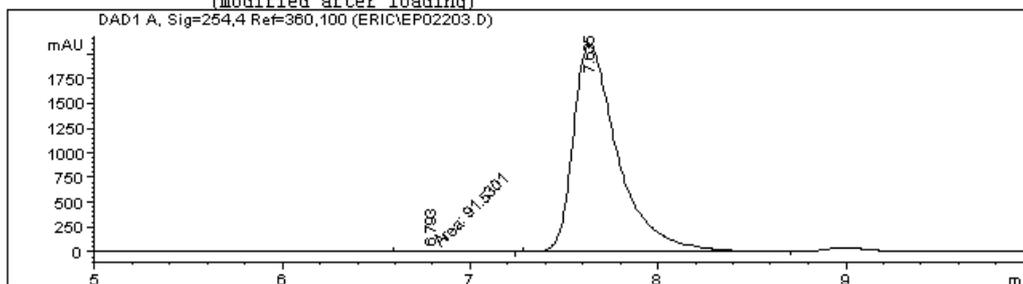
Data File C:\HPCHEM\2\DATA\ERIC\EP02203.D

Sample Name: EP-02-203

pure

```

=====
Injection Date   : 10/30/2006 10:46:54 AM
Sample Name     : EP-02-203                Location : Vial 41
Acq. Operator   : mmb                    Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed    : 10/30/2006 9:17:03 AM by mmb
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\SCHWIN1.M
Last changed    : 11/7/2006 4:48:54 PM by MANABU
                  (modified after loading)
    
```



Area Percent Report

```

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

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.793	MM	0.2606	91.53013	5.85323	0.2630
2	7.635	BV	0.2500	3.47052e4	2086.98047	99.7370

Totals : 3.47967e4 2092.83370

Results obtained with enhanced integrator!

*** End of Report ***

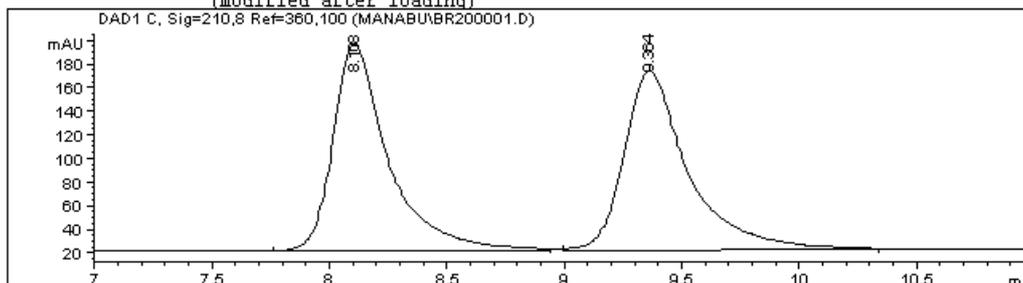
Racemic 5

Data File C:\HPCHEM\2\DATA\MANABU\BR200001.D

Sample Name: br2

```

=====
Injection Date : 11/14/2006 2:53:31 PM
Sample Name    : br2
Acq. Operator  : DAN
Location       : Vial 35
Inj Volume     : 5 µl
Acq. Method    : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 11/14/2006 2:52:08 PM by DAN
                 (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 11/14/2006 3:24:30 PM by DAN
                 (modified after loading)
    
```



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

```

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

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.108	BB	0.2363	2841.84619	174.22916	49.9502
2	9.364	BB	0.2760	2847.51367	151.02719	50.0498

Totals : 5689.35986 325.25635

Results obtained with enhanced integrator!

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

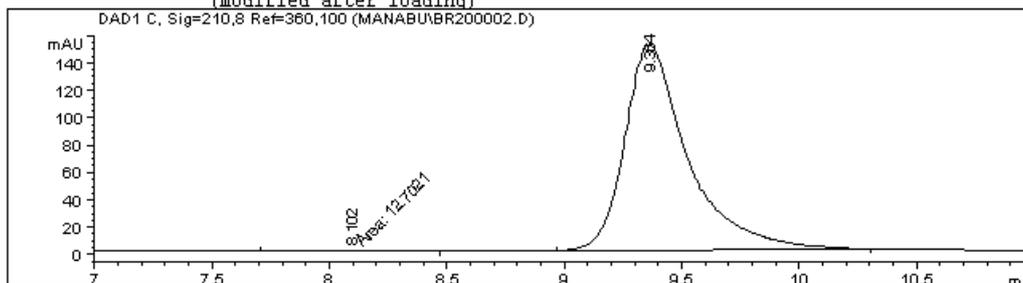
Enantioenriched 5

Data File C:\HPCHEM\2\DATA\MANABU\BR200002.D

Sample Name: br2

```

=====
Injection Date : 11/14/2006 3:09:10 PM
Sample Name    : br2
Acq. Operator  : DAN
Location       : Vial 55
Inj Volume     : 5 µl
Acq. Method    : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 11/14/2006 2:52:08 PM by DAN
                 (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 11/14/2006 3:23:59 PM by DAN
                 (modified after loading)
    
```



```

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

```

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

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.102	MM	0.4302	12.70207	4.92102e-1	0.4484
2	9.364	BB	0.2727	2820.13818	150.45482	99.5516

Totals : 2832.84025 150.94692

Results obtained with enhanced integrator!

```

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

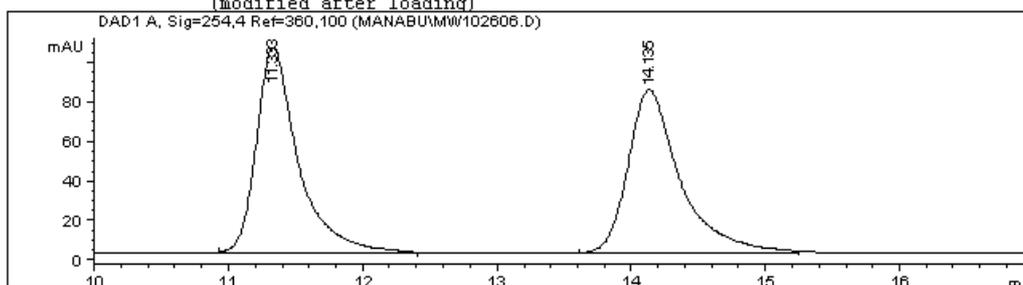
Racemic 6

Data File C:\HPCHEM\2\DATA\MANABU\MW102606.D

Sample Name: MW102606

```

=====
Injection Date : 10/26/2006 9:55:28 AM
Sample Name    : MW102606
Acq. Operator  : mmb
Location       : Vial 1
Inj Volume     : 5 µl
Acq. Method    : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 10/26/2006 8:25:54 AM by mmb
                 (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 11/7/2006 1:50:48 PM by MANABU
                 (modified after loading)
    
```



Area Percent Report

```

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

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.333	BB	0.3161	2243.50635	103.81903	50.2785
2	14.135	BB	0.3961	2218.65283	82.40121	49.7215

Totals : 4462.15918 186.22025

Results obtained with enhanced integrator!

*** End of Report ***

Enantioenriched 6

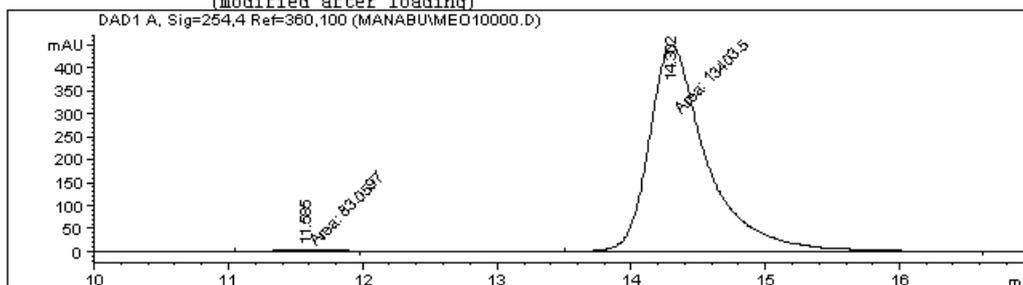
Data File C:\HPCHEM\2\DATA\MANABU\MEO10000.D

Sample Name: Me01

chiral

```

=====
Injection Date : 11/7/2006 10:50:57 PM
Sample Name    : Me01                      Location : Vial 41
Acq. Operator  : MANABU                    Inj Volume : 5 µl
Acq. Method    : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 10/5/2006 10:43:41 AM by mmb
Analysis Method : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 11/7/2006 11:36:55 PM by MANABU
                (modified after loading)
    
```



Area Percent Report

```

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

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.585	MM	0.3698	83.05973	3.74339	0.6159
2	14.302	MM	0.4989	1.34035e4	447.73129	99.3841

Totals : 1.34866e4 451.47468

Results obtained with enhanced integrator!

*** End of Report ***

Racemic 7

Data File C:\HPCHEM\2\DATA\MANABU\ME.D

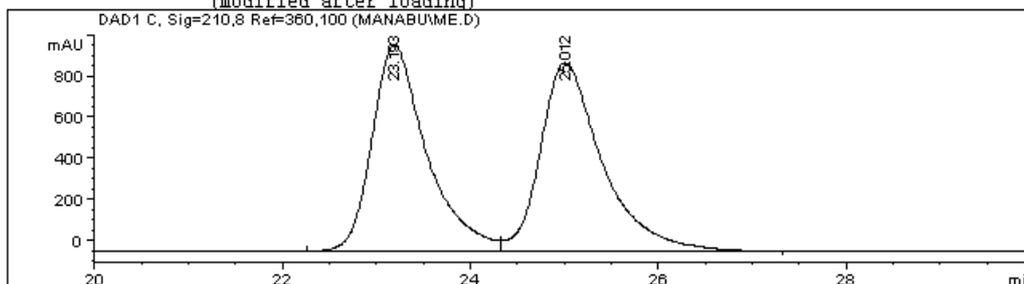
Sample Name: Me

Me

```

=====
Injection Date   : 10/31/2006 1:44:44 PM
Sample Name     : Me                               Location : Vial 1
Acq. Operator   : mmb                               Inj Volume : 5 µl
Acq. Method    : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 10/31/2006 1:43:34 PM by mmb
                (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 11/7/2006 1:41:13 PM by MANABU
                (modified after loading)

```



```

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

```

```

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

```

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.193	BV	0.5958	4.05637e4	1003.09741	49.1400
2	25.012	VB	0.6772	4.19836e4	918.57159	50.8600

```
Totals :                8.25473e4  1921.66901
```

Results obtained with enhanced integrator!

```

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

```

Enantioenriched 7

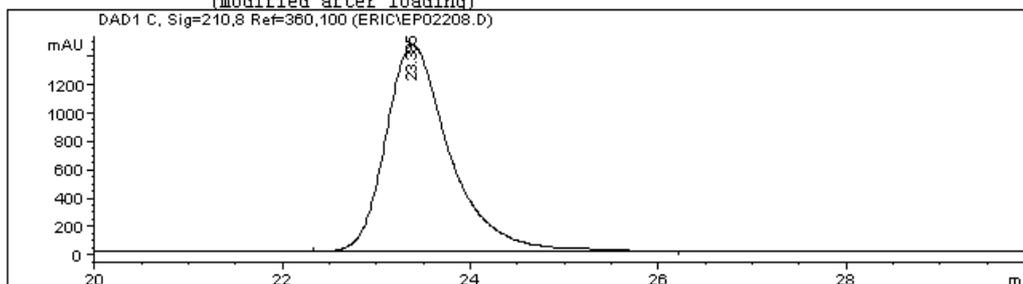
Data File C:\HPCHEM\2\DATA\ERIC\EP02208.D

Sample Name: EP-02-208

ee

```

=====
Injection Date   : 10/31/2006 2:55:51 PM
Sample Name     : EP-02-208                Location : Vial 41
Acq. Operator   : mmb                    Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed    : 10/31/2006 2:55:59 PM by mmb
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\SCHWIN1.M
Last changed    : 11/7/2006 4:51:25 PM by MANABU
                  (modified after loading)
    
```



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

```

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

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.385	BB	0.7088	6.87979e4	1451.85193	100.0000

Totals : 6.87979e4 1451.85193

Results obtained with enhanced integrator!

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

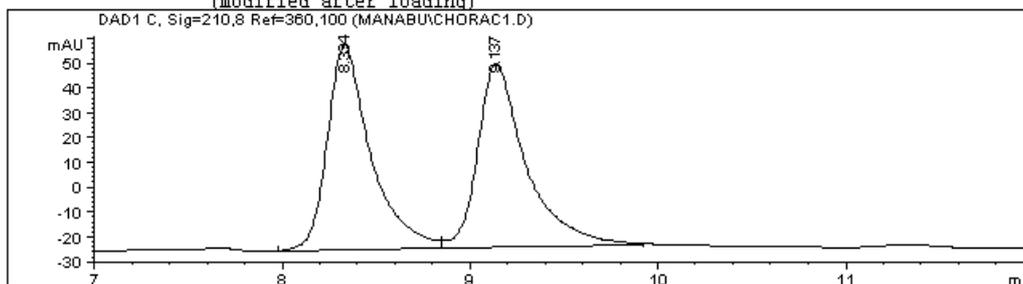
Racemic 8

Data File C:\HPCHEM\2\DATA\MANABU\CHORAC1.D

Sample Name: CHOrac

ee

```
=====
Injection Date : 11/6/2006 10:08:03 PM
Sample Name    : CHOrac                      Location : Vial 1
Acq. Operator  : MANABU                      Inj Volume : 20 µl
Acq. Method    : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 11/6/2006 10:10:19 PM by MANABU
                (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 11/7/2006 1:37:49 PM by MANABU
                (modified after loading)
=====
```



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

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

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.334	BV	0.2353	1322.68250	82.40218	49.9301
2	9.137	VB	0.2607	1326.38330	74.13223	50.0699

Totals : 2649.06580 156.53441

Results obtained with enhanced integrator!

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

Enantioenriched 8

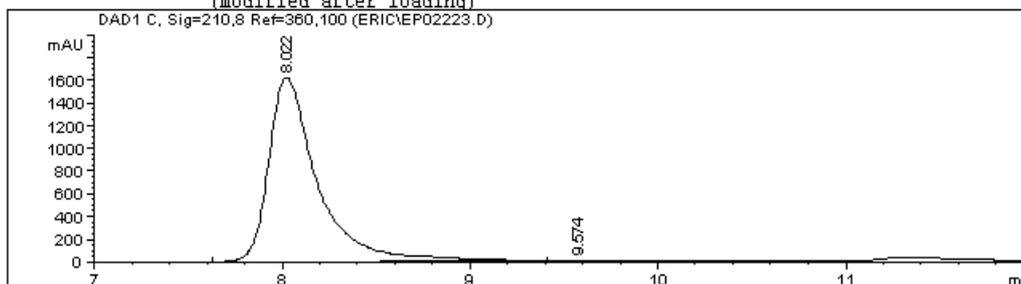
Data File C:\HPCHEM\2\DATA\ERIC\EP02223.D

Sample Name: EP-02-223

ee

```

=====
Injection Date : 11/6/2006 7:05:54 PM
Sample Name    : EP-02-223                Location : Vial 41
Acq. Operator  : MANABU                    Inj Volume : 5 µl
Acq. Method    : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 11/6/2006 6:53:06 PM by MANABU
                (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\SCHWIN1.M
Last changed   : 11/7/2006 4:30:06 PM by MANABU
                (modified after loading)
    
```



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

```

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

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.022	VB	0.2652	2.93498e4	1621.05823	99.3714
2	9.574	BB	0.3618	185.65695	6.90183	0.6286

Totals : 2.95354e4 1627.96006

Results obtained with enhanced integrator!

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

Racemic 10

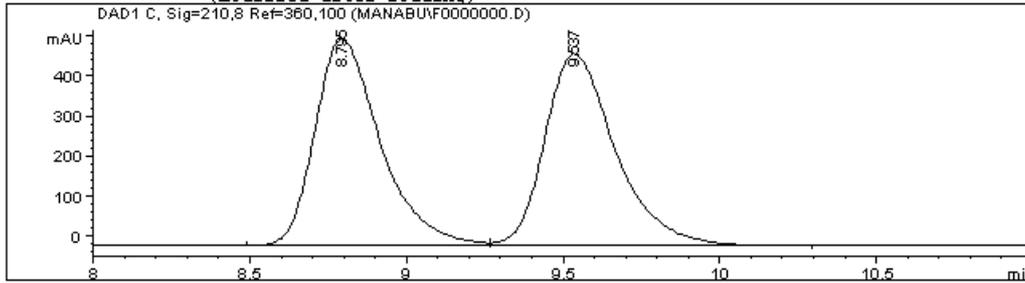
Data File C:\HPCHEM\2\DATA\MANABU\F0000000.D

Sample Name: F

EtOH-IPA 2.5 2.5 Hex 99 OD-H

```

=====
Injection Date : 11/2/2006 5:53:26 PM
Sample Name    : F                               Location : Vial 41
Acq. Operator  : mmb                               Inj Volume : 5 µl
Acq. Method    : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 11/2/2006 5:51:22 PM by mmb
                (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed   : 11/7/2006 2:00:38 PM by MANABU
                (modified after loading)
    
```



Area Percent Report

```

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

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.795	BV	0.2195	7490.45801	515.46973	50.0240
2	9.537	VB	0.2384	7483.25830	478.72235	49.9760

Totals : 1.49737e4 994.19208

Results obtained with enhanced integrator!

*** End of Report ***

Enantioenriched 10

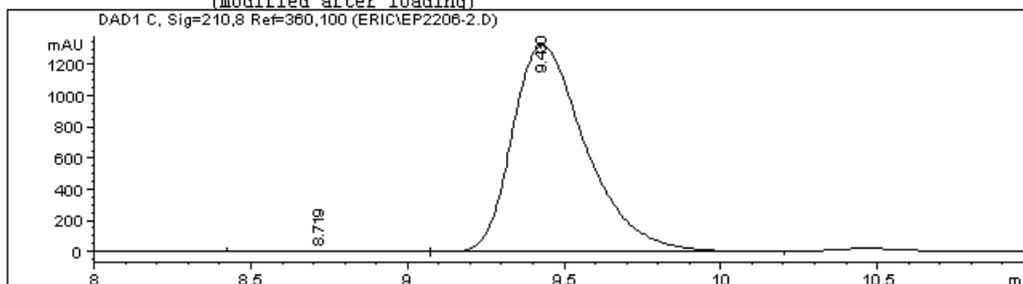
Data File C:\HPCHEM\2\DATA\ERIC\EP2206-2.D

Sample Name: EP-02-206

ee

```

=====
Injection Date   : 11/2/2006 6:18:46 PM
Sample Name     : EP-02-206                Location : Vial 41
Acq. Operator   : mmb                    Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed    : 11/2/2006 5:51:22 PM by mmb
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\SCHWIN1.M
Last changed    : 11/7/2006 4:50:23 PM by MANABU
                  (modified after loading)
    
```



Area Percent Report

```

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

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.719	BV	0.2508	89.48604	5.41465	0.4114
2	9.430	VB	0.2510	2.16637e4	1323.23816	99.5886

Totals : 2.17532e4 1328.65281

Results obtained with enhanced integrator!

*** End of Report ***

Racemic 12

Data File C:\HPCHEM\2\DATA\MANABU\2MEO.D

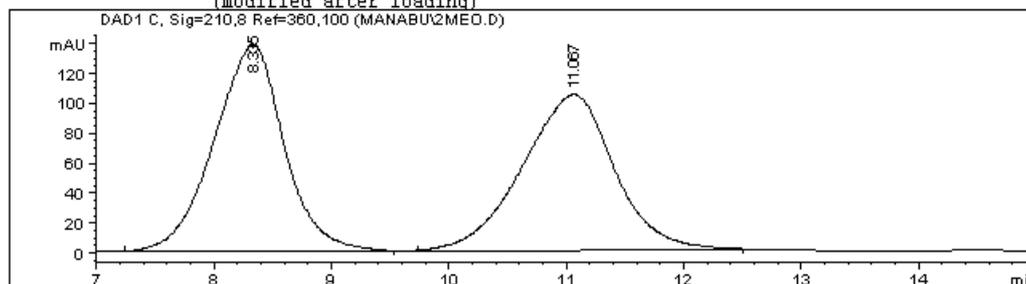
Sample Name: 2MeO

2MeO

```

=====
Injection Date   : 11/1/2006 1:30:46 PM
Sample Name     : 2MeO                      Location   : Vial 1
Acq. Operator   : mmb                      Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed    : 11/1/2006 12:50:58 PM by mmb
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed    : 11/7/2006 1:30:19 PM by MANABU
                  (modified after loading)
=====

```



```

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

```

```

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

```

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.335	BP	0.6417	5699.05176	137.46022	50.3045
2	11.067	BB	0.8359	5630.04932	103.62591	49.6955

```
Totals :                      1.13291e4  241.08613
```

Results obtained with enhanced integrator!

```

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

```

Enantioenriched 12

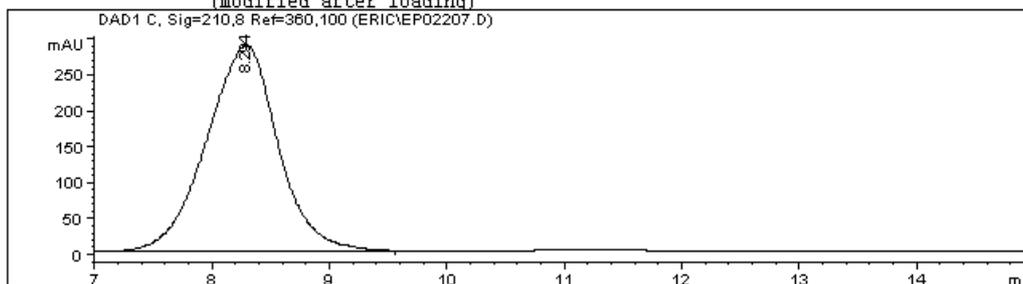
Data File C:\HPCHEM\2\DATA\ERIC\EP02207.D

Sample Name: EP-02-207

ee

```

=====
Injection Date   : 11/1/2006 9:18:58 PM
Sample Name     : EP-02-207
Acq. Operator   : mmb
Location        : Vial 41
Inj Volume      : 5 µl
Acq. Method     : C:\HPCHEM\2\METHODS\EPROCKS.M
Last changed    : 11/1/2006 12:50:58 PM by mmb
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\SCHWIN1.M
Last changed    : 11/7/2006 5:05:00 PM by MANABU
                  (modified after loading)
    
```



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

```

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

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

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.294	BB	0.6574	1.21844e4	286.96872	100.0000

Totals : 1.21844e4 286.96872

Results obtained with enhanced integrator!

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

Racemic 14

Data File C:\HPCHEM\1\DATA\MANABU\RACEMIC.D

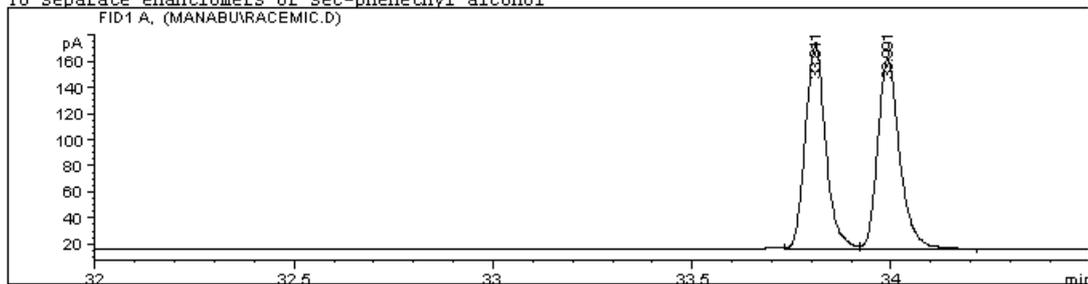
Sample Name: racemic

alkyl

```

=====
Injection Date : 11/6/06 3:29:49 PM
Sample Name    : racemic                Location : Vial 21
Acq. Operator  : Manabu                 Inj      : 1
                                           Inj Volume : 3 µl
Acq. Method    : C:\HPCHEM\1\METHODS\MW.M
Last changed   : 11/6/06 3:23:30 PM by BrianA
Analysis Method : C:\HPCHEM\1\METHODS\MW.M
Last changed   : 11/7/06 9:21:13 PM by Manabu
                 (modified after loading)
    
```

To separate enantiomers of sec-phenethyl alcohol



Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
    
```

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	33.811	VV	0.0496	562.08044	157.67152	50.12238
2	33.991	VB	0.0560	559.33557	146.71539	49.87762

Totals : 1121.41602 304.38692

Results obtained with enhanced integrator!

*** End of Report ***

Enantioenriched 14

Data File C:\HPCHEM\1\DATA\MANABU\EE.D

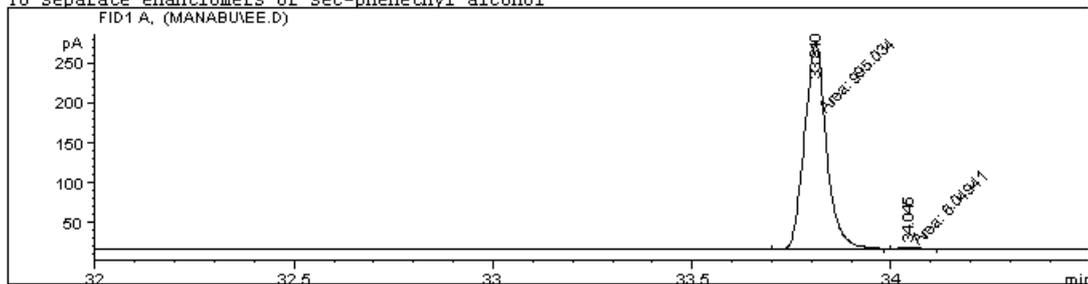
Sample Name: ee

alkyl

```

=====
Injection Date : 11/7/06 7:41:02 PM
Sample Name    : ee                      Location : Vial 25
Acq. Operator  : Manabu                  Inj      : 1
                                           Inj Volume : 3 µl
Acq. Method    : C:\HPCHEM\1\METHODS\MW.M
Last changed   : 11/6/06 3:23:49 PM by BrianA
Analysis Method : C:\HPCHEM\1\METHODS\MW.M
Last changed   : 11/7/06 9:20:35 PM by Manabu
                 (modified after loading)
    
```

To separate enantiomers of sec-phenethyl alcohol



Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/ul] (not used in calc.)
    
```

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	33.810	MM	0.0641	995.03387	258.90158	99.39571
2	34.045	MM	0.0540	6.04941	1.86830	0.60429

Totals : 1001.08329 260.76989

Results obtained with enhanced integrator!

*** End of Report ***

X-Ray Crystallography of Amide 17

X-ray diffraction was performed at $-120\text{ }^{\circ}\text{C}$ and raw frame data were processed using SAINT. Molecular structure was solved using direct methods and refined by F2 by full-matrix least-squares techniques. The GOF = 1.084 for 267 variables refined to $R1 = 0.0409$ for 4634 reflections with $I > 2\sigma(I)$. There was no absorption correction of Flack parameters. Further information is contained in the CIF file.