Organocatalytic Asymmetric Aldol Reaction of Hydroxyacetone with β,γ-Unsaturated α-Keto Esters: Facile Access to Chiral Tertiary Alcohols

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Supporting Information

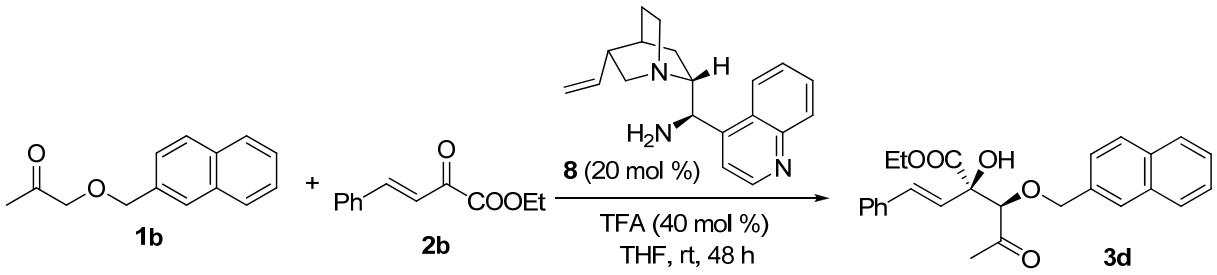
A. General Information

Chemicals and solvents were purchased from commercial suppliers and used as received. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker ACF300 or an AMX500 (500 MHz) spectrometer. Chemical shifts were reported in parts per million (ppm), and the residual solvent peak was used as an internal reference: proton (chloroform δ 7.26), carbon (chloroform δ 77.0). Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet), br s (broad singlet). Coupling constants were reported in Hertz (Hz). Low resolution mass spectra were obtained on a Finnigan/MAT LCQ spectrometer in ESI mode, and a Finnigan/MAT 95XL-T mass spectrometer in FAB mode. All high resolution mass spectra were obtained on a Finnigan/MAT 95XL-T spectrometer. For thin-layer chromatography (TLC), Merck pre-coated TLC plates (Merck 60 F_{254}) were used, and compounds were visualized under a UV light at 254 nm. Further visualization was achieved by staining with iodine, or ceric ammonium molybdate followed by heating on a hot plate. Flash chromatographic separations were performed on Merck 60 (0.040 - 0.063 mm) mesh silica gel. The enantiomeric excesses of products were determined by HPLC analysis on a chiral stationary phase.

1-(Benzyloxy)propan-2-one was purchased from Sigma-Aldrich. Other protected hydroxyacetone [1] and β,γ-unsaturated α-keto esters [2] were prepared following literature procedures.

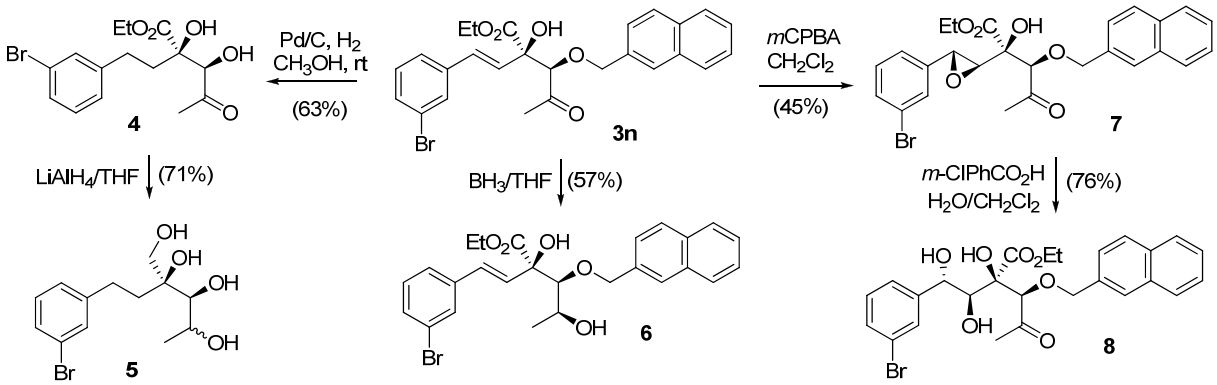
[1] Luo, S.; Qiao, Y,; Zhang, L.; Li, J.; Li, X.; Cheng, J. *J. Org. Chem.* **2009**, *74*, 9521.
[2] Wu, Y.-C.; Liu, L.; Li, H.-J.; Wang, D.; Chen, Y.-J. *J. Org. Chem.* **2006**, *71*, 6592.

B. Representative Procedure for the Aldol Reaction



1-(Naphthalen-2-ylmethoxy)propan-2-one **1b** (21.4 mg, 0.1 mmol) was added to a mixture of 9-amino(9-deoxy)*epi*-cinchonine **8** (5.8 mg, 0.02 mmol), TFA (3.0 μL, 0.04 mmol) and ethyl 2-oxo-4-phenylbut-3-enoate **2b** (22.4 mg, 0.12 mmol) in THF (0.2 mL) in a sample vial at room temperature. The vial was then sealed and the reaction mixture was stirred at room temperature for 48 hours. The mixture was then concentrated *in vacuo* and the residue was purified by flash column chromatography (ethyl acetate/hexanes = 1:10 to 1:3) to afford the desired product as a yellow oil (33.9 mg, 81%). The enantiometric excess of product was determined by chiral HPLC analysis.

C. Synthetic Manipulations of the Aldol Product 3n



Following the representative procedure, the aldol product **3n** was obtained, which was then subjected to the subsequent reactions as illustrated above.

(2*R*,3*R*)-Ethyl 2-(3-bromophenethyl)-2,3-dihydroxy-4-oxopentanoate 4

A flask containing (2*R*,3*R*)-ethyl 2-(3-bromostyryl)-2-hydroxy-3-(naphthalen-2-ylmethoxy)-4-oxopentanoate **3n** (74.5 mg, 0.15 mmol) and 10% Pd/C (32.5 mg, 0.03 mmol) in methanol (2 mL) was evacuated and back-filled with hydrogen (1 atm). The reaction mixture was stirred under hydrogen. When TLC showed the completion of the reaction, the mixture was concentrated, and the residue was purified by flash column chromatography (ethyl acetate/hexanes = 1:3) to afford **4** as a colorless oil (34.0 mg, 63%).

(2*S*,3*S*)-2-(3-Bromophenethyl)pentane-1,2,3,4-tetraol 5

To lithium aluminum hydride (7.6 mg, 0.2 mmol) in THF (1.0 mL) was added a solution of the (2*R*,3*R*)-ethyl 2-(3-bromophenethyl)-2,3-dihydroxy-4-oxopentanoate **4** (27.0 mg, 0.075 mmol) in THF (1.0 mL) at 0 ^{o}C under nitrogen. The mixture was then allowed to warm up slowly and stirred at room temperature. Upon the completion of the reaction, as indicated by TLC analysis, the reaction mixture was cooled to 0 ^{o}C and carefully quenched with successive additions of water, 15% aqueous sodium hydroxide and water. After being stirred for 30 minutes, the reaction mixture was extracted with ethyl acetate (3 x 3 mL). The combined organic extracts were washed with brine and dried over $MgSO_4$. After concentration, the residue was purified by flash column chromatography (ethyl acetate) to afford **5** as colorless oil (17.0 mg, 71%).

(2*R*,3*S*,4*R*)-Ethyl-2-((*E*)-3-bromostyryl)-2,4-dihydroxy-3-(naphthalen-2-ylmethoxy)pentanoate **6**

To a solution of **3n** (34.7 mg, 0.07 mmol) in THF (1.0 mL) at 0 ^{o}C was added borane-dimethyl sulfide complex (BH_3-SMe_2 in THF, 2 M) (0.35 mL, 0.7 mmol) under N_2, and the

resulting solution was warmed up slowly from 0 ^{o}C to room temperature and stirred for 4 hours. The reaction was then re-cooled to 0 ^{o}C and quenched by addition of cold water. The resulting mixture was extracted with CH_2Cl_2 (3 x 10 mL), and the combined organic extracts were then washed with saturated $NaHCO_3$, brine and dried over $MgSO_4$. After concentration, the residue was purified by flash column chromatography (dichloromethane) to afford **6** as colorless oil (19.9 mg, 57%).

A colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.24-1.27 (t, *J* = 7.3 Hz, 3H), 1.30-1.31 (d, *J* = 6.3 Hz, 3H), 3.83 (d, *J* = 1.9 Hz, 1H), 4.10-4.27 (m, 4H), 4.80-4.83 (d, *J* = 11.4 Hz, 1H), 4.95-4.97 (d, *J* = 11.4 Hz, 1H), 6.31-6.35 (d, *J* = 16.4 Hz, 1H), 6.93-6.96 (d, *J* = 15.8 Hz, 1H), 7.17-7.20 (t, *J* = 7.6 Hz, 1H), 7.27(s, 1H), 7.38-7.40 (d, *J* = 8.2 Hz, 1H), 7.43-7.45 (d, *J* = 8.8 Hz, 1H), 7.48-7.49 (t, *J* = 4.1 Hz, 2H), 7.56 (s, 1H), 7.78 (s, 1H), 7.83-7.84 ((d, *J* = 7.6 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$,) δ 14.1, 21.3, 29.7, 62.8, 66.7, 76.0, 81.3, 84.8, 122.9, 125.5, 125.8, 126.1, 126.3, 127.7, 127.9, 128.3, 129.4, 130.2, 130.4, 131.0, 133.1, 133.3, 135.1, 138.2, 172.8; HRMS (ESI) m/z calcd for $C_{26}H_{27}NaO_5{}^{79}Br$ $[M+Na]^+$ 521.0934, found 521.0914, calcd for $C_{26}H_{27}NaO_5{}^{81}Br$ $[M+Na]^+$ 523.0914, found 523.0917.

(2*R*,3*R*)-Ethyl-2-((2*S*,3*R*)-3-(3-bromophenyl)oxiran-2-yl)-2-hydroxy-3-(naphthalen-2-ylmethoxy)-4-oxopentanoate **7**

The aldol product **3n** (34.7 mg, 0.07 mmol) was dissolved in CH_2Cl_2 (0.7 mL), *m*CPBA (14.5 mg, 0.084 mmol) was added to the solution under N_2, and the resulting solution was stirred at room temperature for 48 hours. The mixture was concentrated *in vacuo* and the residue was purified by flash column chromatography (ethyl acetate/hexanes = 1:5 to 1:3) to afford **7** as a white solid (16.1 mg, 45%).

A white solid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.24-1.27 (t, *J* = 7.3 Hz, 3H), 1.52 (s, 3H), 3.98 (s, 1H), 4.04-4.11 (m, 1H), 4.22-4.24 (d, *J* = 10.7 Hz, 1H), 4.25-4.31 (m, 1H), 4.39 (s, 1H), 4.72-4.75 (d, *J* = 11.3 Hz, 1H), 4.88-4.90 (d, *J* = 11.3 Hz, 1H), 4.96-4.99 (d, *J* = 10.7 Hz, 1H), 5.72 (s, 1H), 7.21-7.24 (t, *J* = 7.9 Hz, 1H), 7.31-7.34 (d, *J* = 7.6 Hz, 1H), 7.41-7.52 (m, 4H), 7.59 (s, 1H), 7.75 (s, 1H), 7.83-7.86 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$,) δ 14.1, 26.2, 54.3, 63.7, 71.2, 75.4, 79.6, 79.8, 99.1, 122.4, 125.8, 126.3, 126.4, 127.0, 127.2, 127.7, 128.0, 128.5, 129.7, 130.7, 131.9, 133.1. 133.2, 134.1, 139.9, 170.5; MS (EI) m/z calcd for $C_{26}H_{25}O_6{}^{79}Br$ 512.5, found 512.4.

(2*R*,3*R*)-Ethyl-2-((1*S*,2*S*)-2-(3-bromophenyl)-1,2-dihydroxyethyl)-2-hydroxy-3-(naphthalen-2-ylmethoxy)-4-oxopentanoate **8**

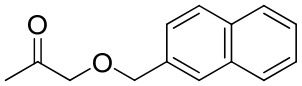
The aldol product **3n** (34.7 mg, 0.07 mmol) was dissolved in CH_2Cl_2 (0.7 mL), *m*CPBA(14.5 mg, 0.084 mmol) was added to the solution under N_2, and the resulting solution was stirred at room temperature for 48 hours. Distilled water (20 mg, 20 μL) was then added, and the resulting mixture was heated at 40 ^{o}C for 12 h. After cooling down to room temperature, the reaction mixture was concentrated *in vacuo* and the residue was purified by flash column chromatography (ethyl acetate/hexanes = 1:3 to 1:1) to afford **8** as a colorless oil (12.7 mg, 45%).

A colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.21-1.24 (t, *J* = 7.3 Hz, 3H), 1.55 (s, 3H), 3.81-3.83 (d, *J* = 9.5 Hz, 1H), 3.87 (s, 1H), 4.03-4.10 (m, 1H), 4.21-4.27 (m, 1H), 4.32 (s, 1H), 4.67-4.69 (d, *J* = 10.1 Hz, 1H), 4.72-4.74 (d, *J* = 11.4 Hz, 1H), 4.89-4.91 (d, *J* = 11.4 Hz, 1H), 5.72 (s, 1H), 7.21-7.24 (t, *J* = 7.9 Hz, 1H), 7.34-7.36 (d, *J* = 7.6 Hz, 1H), 7.42-7.46 (m, 2H), 7.48-7.52

(m, 2H), 7.63 (s, 1H), 7.75 (s, 1H), 7.83-7.86 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$,) δ 14.0, 26.2, 63.3, 70.8, 73.3, 75.2, 79.2, 80.0, 98.3, 122.6, 125.8, 126.3, 126.4, 126.6, 127.0, 127.7, 128.0, 128.4, 129.8, 130.5, 131.4, 133.1. 133.2, 134.3, 140.5, 171.8; HRMS (ESI) m/z calcd for $C_{26}H_{27}{}^{79}BrNaO_7$ $[M+Na]^+$ 553.0832, found 553.0836.

D. Analytical Data and HPLC Chromatogram of Starting Materials and Aldol Products

1-(Naphthalen-2-ylmethoxy)propan-2-one **1b**

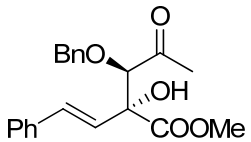


1b

Compound **1b** was prepared following the literature procedure.[1]

A colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.19 (s, 3H), 4.12 (s, 2H), 4.78 (s, 2H), 7.51-7.55 (m, 3H), 7.83-7.90 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$,) δ 26.4, 73.3, 75.1, 125.7, 126.1, 126.2, 126.8, 127.7, 127.9, 128.4, 133.1, 133.2, 134.6, 206.5; HRMS (ESI) m/z calcd for $C_{14}H_{14}O_2$ $[M+Na]^+$ 237.0891, found 237.0896.

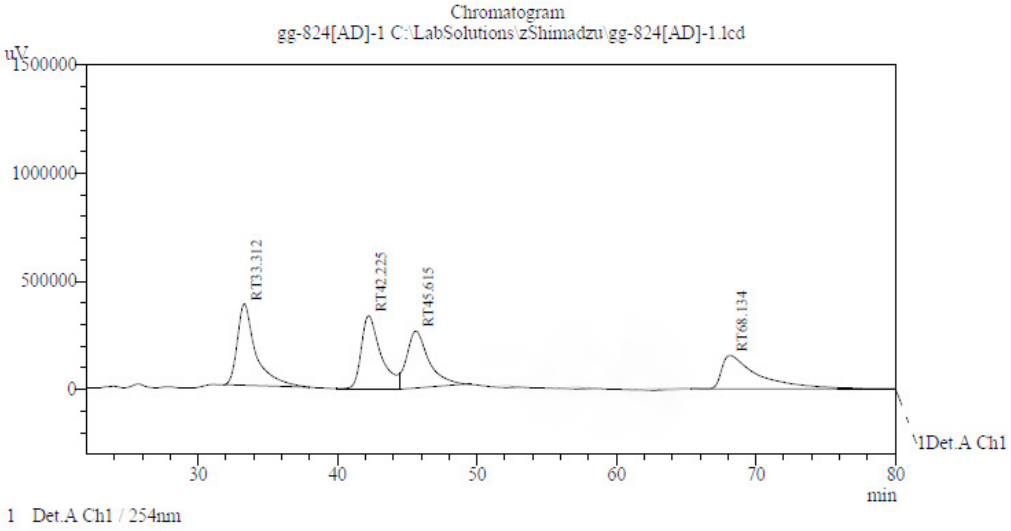
(2*R*,3*R*)-Methyl 3-(benzyloxy)-2-hydroxy-4-oxo-2-styrylpentanoate **3a**



3a

A yellow oil; two diastereomers were obtained in a ratio of 2 to 1, with ee values at 87% and 64%, respectively; the *syn* isomer: t_R (major) = 33.04 min, t_R (minor) = 42.12 min; the *anti* isomer: t_R (minor) = 45.43 min, t_R (major) = 68.56 min (Chiralcel AD-H, λ = 254 nm, 5% *i*PrOH/hexanes, flow rate = 0.8 mL/min); ^{1}H NMR (500 MHz, $CDCl_3$, mixture of diastereomers)

δ 2.25 (s, 3H), 2.29 (s, 3H), 3.70, (s, 6H), 3.86 (s, 2H), 4.10 (s, 1H), 4.11 (s, 1H), 4.23-4.78 (m, 4H, CH_2 for both diastereomers), 6.20 (d, *J* = 15.7 Hz, 1H), 6.25 (d, *J* = 15.7 Hz, 1H), 6.87 (d, *J* = 15.7 Hz, 1H), 6.96 (d, *J* = 15.7 Hz, 1H), 7.28-7.41 (m, 20H); ^{13}C NMR (125 MHz, $CDCl_3$, mixture of diastereomers) δ 28.1, 53.4, 79.7, 87.4, 123.9, 126.1, 126.7, 126.9, 127.9, 128.1, 128.3, 128.5, 131.7, 131.9, 135.9, 136.3, 172.6, 207.7; HRMS (ESI) m/z calcd for $C_{21}H_{22}O_5$ $[M+Na]^+$ 377.1359, found 377.1359.

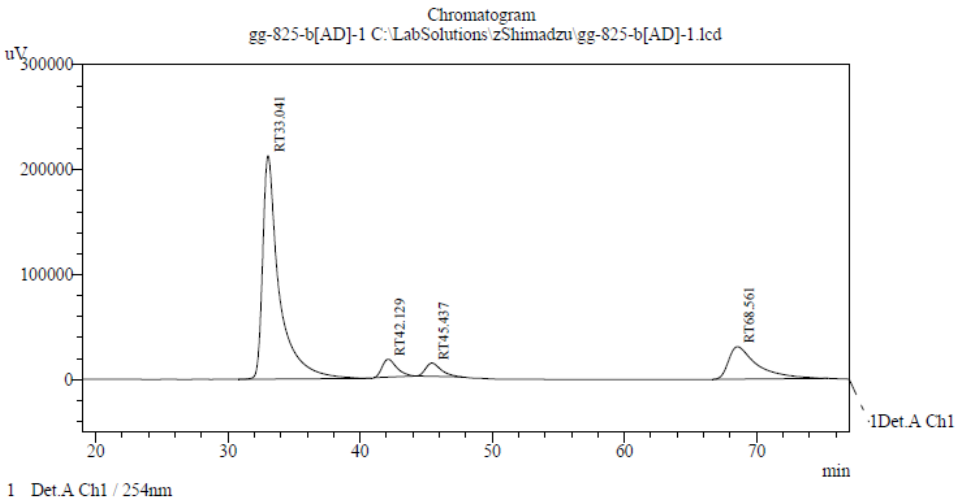


PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT33.312	33.312	34414185	375782	27.553	mg/L
2	RT42.225	42.225	33105053	338166	26.505	mg/L
3	RT45.615	45.615	28877252	263894	23.120	mg/L
4	RT68.134	68.134	28503400	153962	22.821	mg/L
Total			124899890	1131805	100.000	

(racemic **3a**)



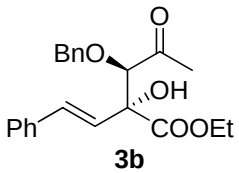
PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT33.041	33.041	18535305	212810	72.391	mg/L
2	RT42.129	42.129	1320825	17070	5.159	mg/L
3	RT45.437	45.437	1040041	12606	4.062	mg/L
4	RT68.561	68.561	4708315	30963	18.389	mg/L
Total			25604486	273450	100.000	

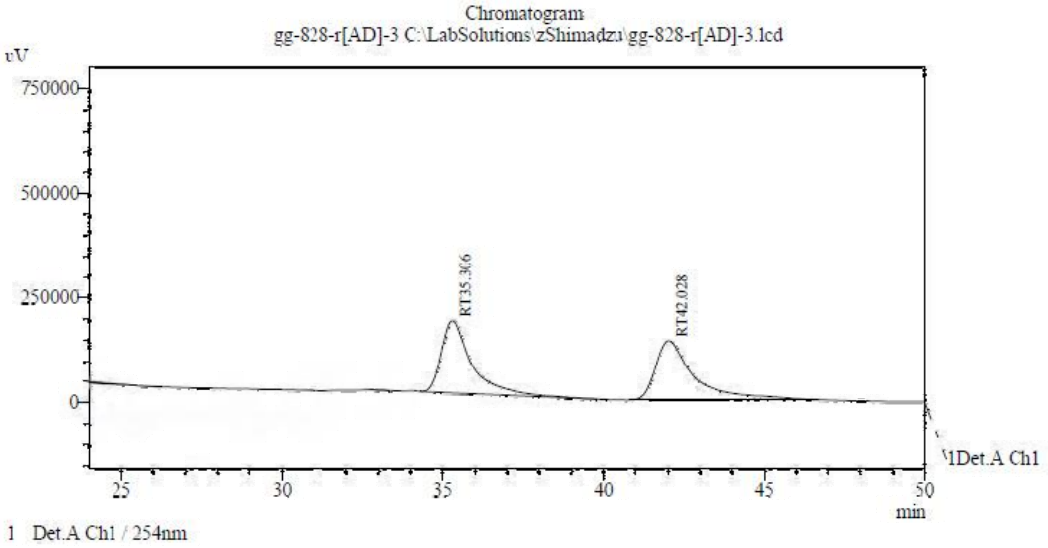
(enatiometrically enriched **3a**)

(2*R*,3*R*)-Ethyl 3-(benzyloxy)-2-hydroxy-4-oxo-2-styrylpentanoate **3b**



3b

The *syn* diastereomer was isolated as a yellow oil; the ee value was 91%; t_R (major) = 34.50 min, t_R (minor) = 41.02 min (Chiralcel AD-H, λ = 254 nm, 3% *i*PrOH/hexanes, flow rate = 0.8 mL/min); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.21 (t, *J* = 7.2 Hz, 3H), 2.25 (s, 3H), 3.88 (s, 1H), 4.10-4.27 (m, 3H), 4.43 (d, *J* = 11.3 Hz, 1H), 4.69 (d, *J* = 11.3 Hz, 1H), 6.26 (d, *J* = 15.7 Hz, 1H), 6.88 (d, *J* = 15.8 Hz, 1H), 7.28-7.41 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.0, 26.4, 62.8, 73.3, 79.2, 87.7, 124.1, 126.9, 127.9, 128.0, 128.2, 128.5, 128.6, 131.6, 136.0, 137.1, 172.1, 206.7; HRMS (ESI) m/z calcd for $C_{22}H_{24}O_5$ $[M+Na]^+$ 391.1516, found 391.1526.



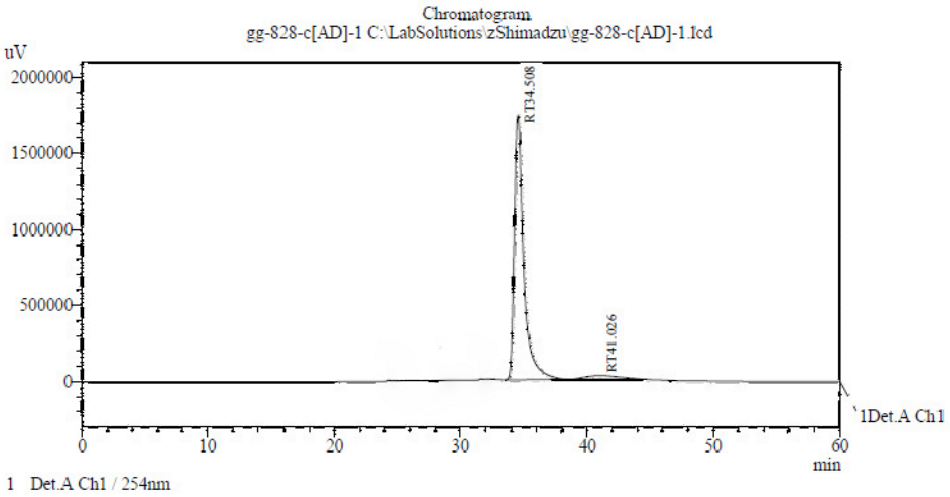
1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT35.306	35.306	11306270	172835	48.640	mg/L
2	RT42.028	42.028	11938397	142336	51.360	mg/L
Total			23244667	315170	100.000	

(racemic **3b**)



1 Det.A Ch1 / 254nm

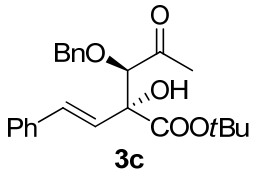
PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT34.508	34.508	89855472	1738321	95.466	mg/L
2	RT41.026	41.026	4267289	22622	4.534	mg/L
Total			94122760	1760944	100.000	

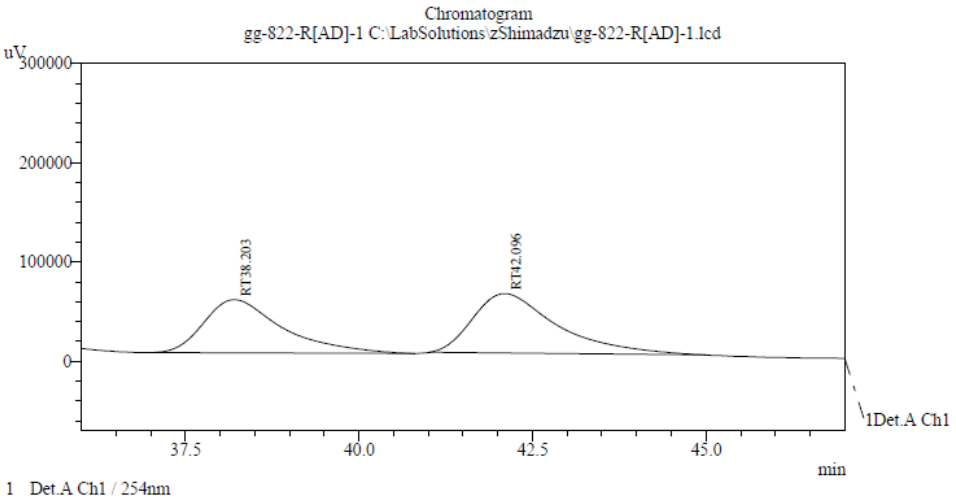
(enatiometrically enriched **3b**)

(2*R*,3*R*)-*tert*-Butyl-3-(benzyloxy)-2-hydroxy-4-oxo-2-styrylpentanoate **3c**



3c

The *syn* diastereomer was isolated as a yellow oil; the ee value was 55%; t_R (major) = 37.71 min, t_R (minor) = 41.74 min (Chiralcel AD-H, λ = 254 nm, 3% *i*PrOH/hexanes, flow rate = 0.8 mL/min); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.46 (s, 9H), 2.22, 2.26 (2s, 3H, CH_3 for two diastereomers), 3.88 (s, 1H), 4.20 (s, 1H), 4.23-4.62 (m, 2H), 6.25 (d, *J* = 15.8 Hz, 1H), 6.86 (d, *J* = 15.7 Hz, 1H), 7.26-7.46 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.4, 27.9, 73.3, 79.1, 83.9, 88.2, 124.7, 126.9, 127.9, 128.2, 128.3, 128.5, 129.0, 131.4, 136.2, 137.1, 171.1, 208.4; HRMS (ESI) m/z calcd for $C_{24}H_{28}O_5$ $[M+Na]^+$ 419.1829, found 419.1829.

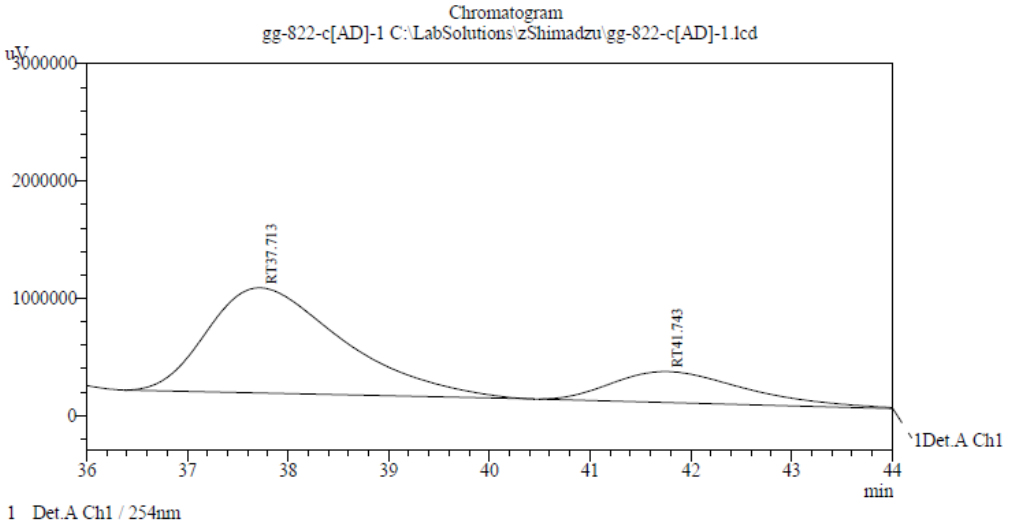


PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT38.203	38.203	4209433	53598	45.399	mg/L
2	RT42.096	42.096	5062573	59789	54.601	mg/L
Total			9272006	113387	100.000	

(racemic **3c**)



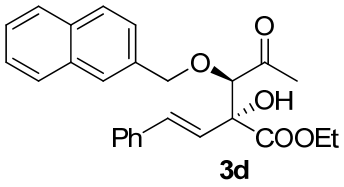
PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT37.713	37.713	84205418	897107	77.570	mg/L
2	RT41.743	41.743	24348376	263148	22.430	mg/L
Total			108553794	1160255	100.000	

(enatiometrically enriched **3c**)

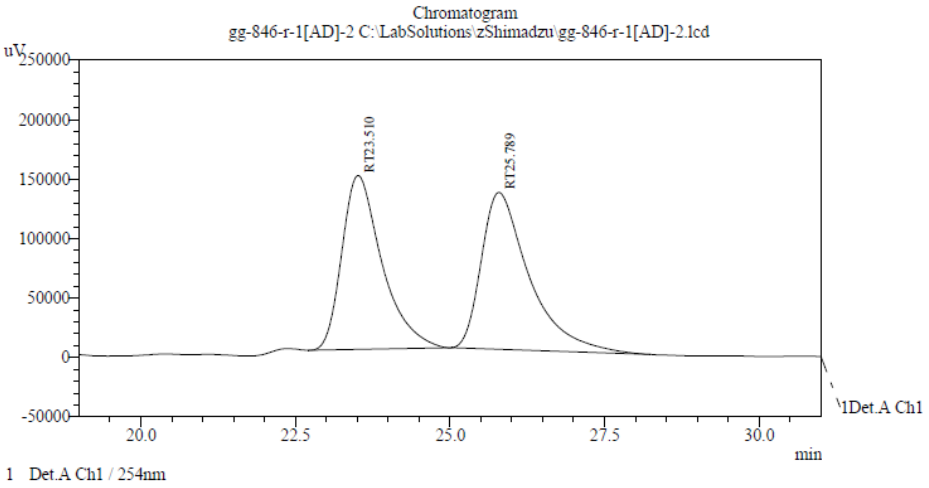
(2*R*,3*R*)-Ethyl 2-hydroxy-3-(naphthalen-2-ylmethoxy)-4-oxo-2-styrylpentanoate **3d**



3d

The *syn* diastereomer was isolated as a yellow oil; the ee value was 91%; t_R (major) = 23.75 min, t_R (minor) = 25.75 min (Chiralcel AD-H, λ = 254 nm, 10% *i*PrOH/hexanes, flow rate = 0.8 mL/min); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.09 (t, *J* = 7.2 Hz, 3H), 2.28 (s, 3H), 3.91 (s, 1H), 4.01-4.07 (m, 1H), 4.17-4.23 (m, 1H), 4.30 (s, 1H), 4.57 (d, *J* = 11.3 Hz, 1H), 4.87 (d, *J* = 11.3 Hz, 1H), 6.26 (d, *J* = 15.7 Hz, 1H), 6.88 (d, *J* = 15.7 Hz, 1H), 7.26-7.88 (m, 12H); ^{13}C NMR (125 MHz, $CDCl_3$, *syn* isomer) δ13.9, 26.4, 62.8, 73.4, 79.2, 87.5, 124.1,125.7, 126.1, 126.2, 126.4, 126.8, 126.9, 127.3, 127.7, 127.9, 128.4, 128.5, 131.6, 133.1, 134.6, 136.0, 172.1, 206.6;

$[\alpha]^{25}_{D}$ = –39.9 (c = 1.00 in $CHCl_3$); HRMS (ESI) m/z calcd for $C_{26}H_{26}O_5$ $[M+Na]^+$ 441.1672, found 441.1686.

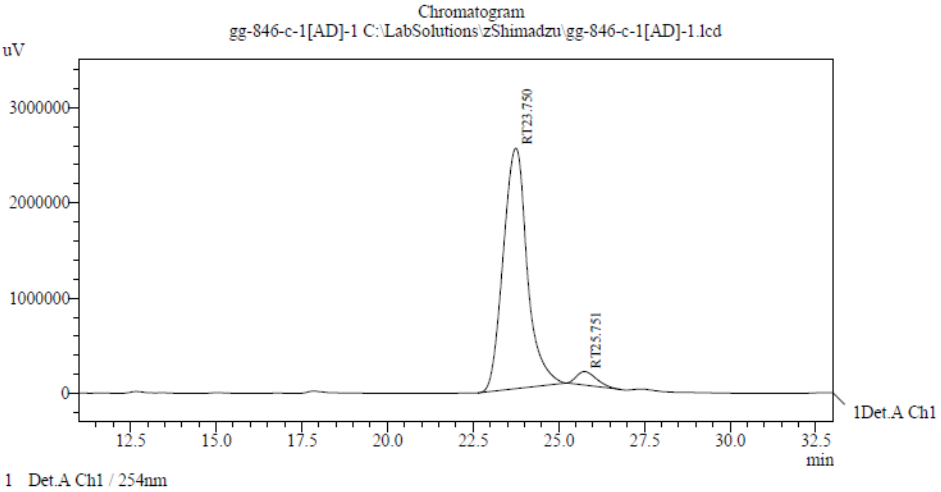


PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT23.510	23.510	6608412	146358	48.124	mg/L
2	RT25.789	25.789	7123756	131973	51.876	mg/L
Total			13732168	278331	100.000	

(racemic **3d**)



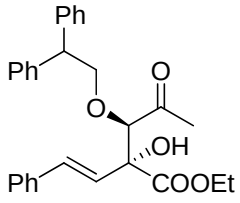
PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT23.750	23.750	123413192	2523577	95.536	mg/L
2	RT25.751	25.751	5766987	141576	4.464	mg/L
Total			129180179	2665153	100.000	

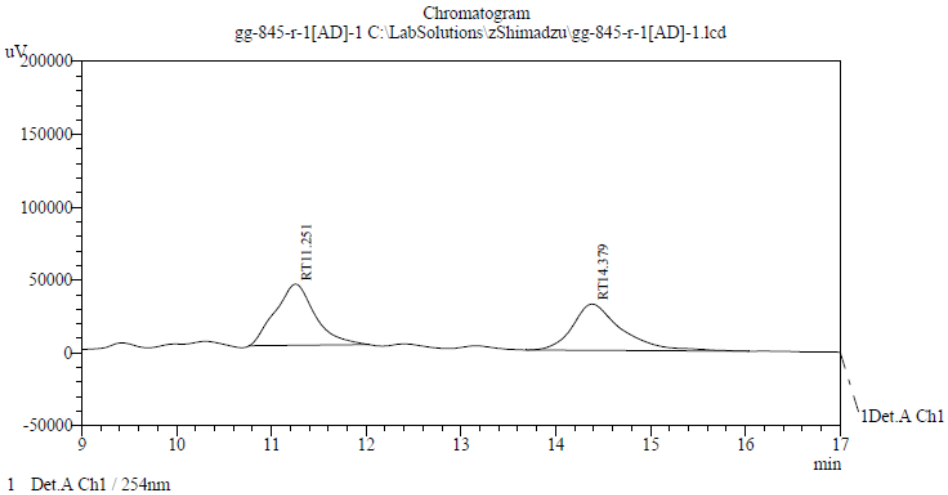
(enatiometrically enriched **3d**)

(2*R*,3*R*)-Ethyl 3-(2,2-diphenylethoxy)-2-hydroxy-4-oxo-2-styrylpentanoate **3e**



3e

A 2:1 (*syn:anti*) diastereomeric mixture was isolated as a yellow oil; the ee values were 44% and 87%; the *syn* isomer: t_R (major) = 11.05 min, t_R (minor) = 14.07 min, the *anti* isomer: t_R (minor) = 27.01 min, t_R (major) = 33.94 min (Chiralcel AD-H, λ = 254 nm, 10% *i*PrOH/hexanes, flow rate = 1.0 mL/min); ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.92 (t, *J* = 6.9 Hz, 3H), 1.11 (t, *J* = 7.4 Hz, 3H), 2.25 (s, 6H), 4.02 (s, 1H), 4.10 (s, 1H), 4.16-4.32 (m, 8H), 6.15 (d, *J* = 16.4 Hz, 1H), 6.23 (d, *J* = 15.7 Hz, 1H), 6.90 (d, *J* = 15.7 Hz, 1H), 6.94 (d, *J* = 15.7 Hz, 1H), 7.28-7.48 (m, 30H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.1, 26.8, 62.5, 80.0, 85.4, 126.5, 126.8, 127.1, 127.4, 127.5, 127.7, 127.8, 128.4, 128.5, 129.0, 131.6, 134.0, 141.1, 142.1, 148.5, 172.2, 207.0; HRMS (ESI) m/z calcd for $C_{28}H_{28}O_5$ $[M+Na]^+$ 467.1829, found 467.1843.

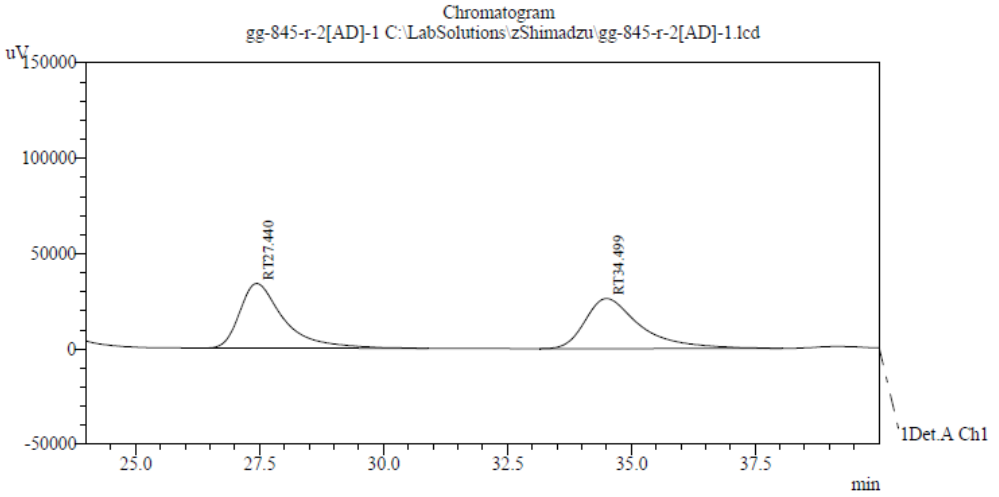


1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT11.251	11.251	1234392	42038	52.091	ppm
2	RT14.379	14.379	1135274	31692	47.909	ppm
Total			2369665	73730	100.000	



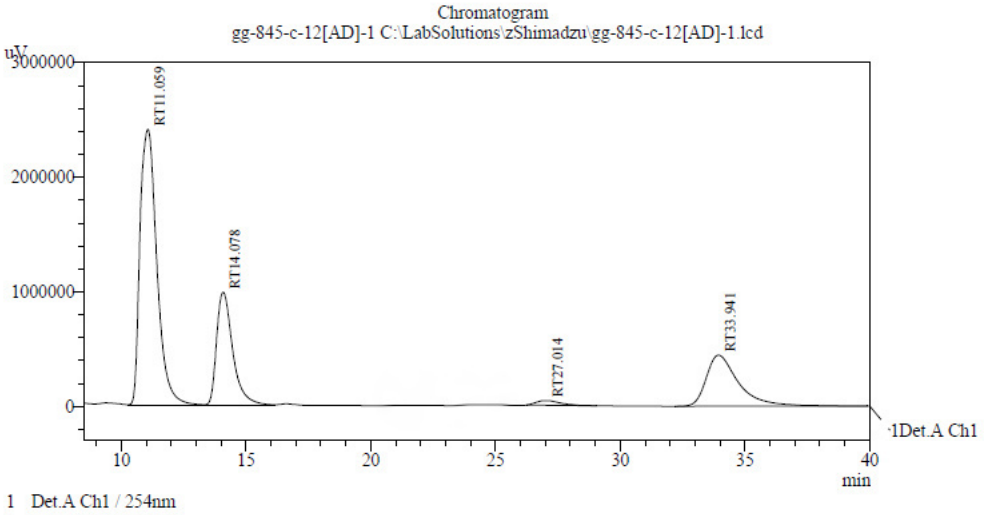
1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT27.440	27.440	2106400	33985	50.901	ppm
2	RT34.499	34.499	2031841	26269	49.099	ppm
Total			4138241	60254	100.000	

(racemic **3e**)



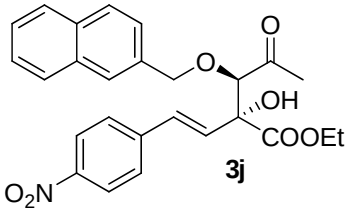
PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT11.059	11.059	112628156	2406504	56.529	ppm
2	RT14.078	14.078	43887350	983569	22.027	ppm
3	RT27.014	27.014	2737182	41045	1.374	ppm
4	RT33.941	33.941	39987132	445163	20.070	ppm
Total			199239820	3876281	100.000	

(enatiometrically enriched **3e**)

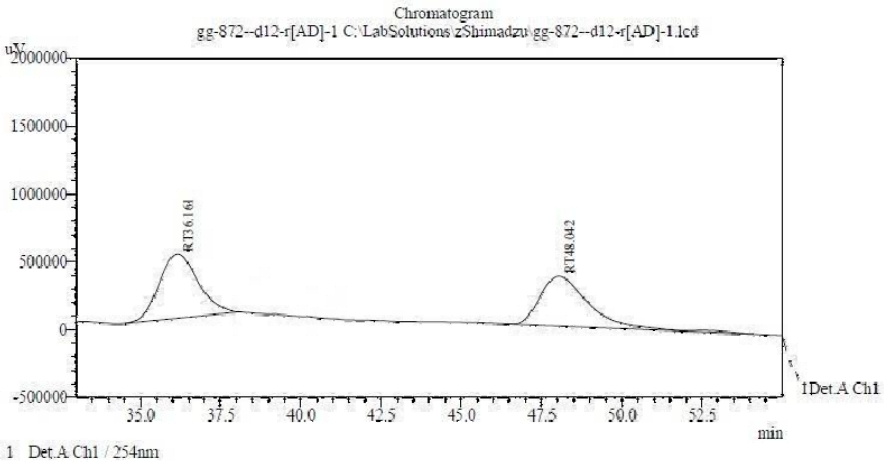
(2*R*,3*R*)-Ethyl 2-hydroxy-3-(naphthalen-2-ylmethoxy)-2-(4-nitrostyryl)-4-oxopentanoate **3j**



3j

The diastereomeric mixture was isolated as a yellow oil in an 86% combined yield, and both diastereomers were easily separated; The ee value of the *syn* isomer was 99%; t_R (major) = 38.19 min, t_R (minor) = 49.60 min (Chiralcel AD-H, λ = 254 nm, 5% *i*PrOH/hexanes, flow rate = 0.8 mL/min); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.09 (t, *J* = 6.9 Hz, 3H), 2.29 (s, 3H), 3.96 (s, 1H), 4.04-4.25 (m, 2H), 4.27 (s, 1H), 4.58 (d, *J* = 12.0 Hz, 1H), 4.88 (d, *J* = 12.0 Hz, 1H), 6.43 (d, *J* = 15.8 Hz, 1H), 6.92 (d, *J* = 15.8 Hz, 1H), 7.37-8.18 (m, 11H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.9, 28.0, 63.2, 73.7, 79.4, 87.4, 123.9, 125.9, 126.4, 126.5, 127.3, 127.4, 127.5, 127.7, 127.9,

128.5, 129.0, 129.5, 129.8, 133.1, 133.2, 133.4, 142.4, 147.2, 171.5, 208.3; $[\alpha]^{25}_{D}$ = –37.4 (c = 2.00 in $CHCl_3$); HRMS (ESI) m/z calcd for $C_{26}H_{25}NO_7$ $[M+Na]^+$ 486.1523, found 486.1521.

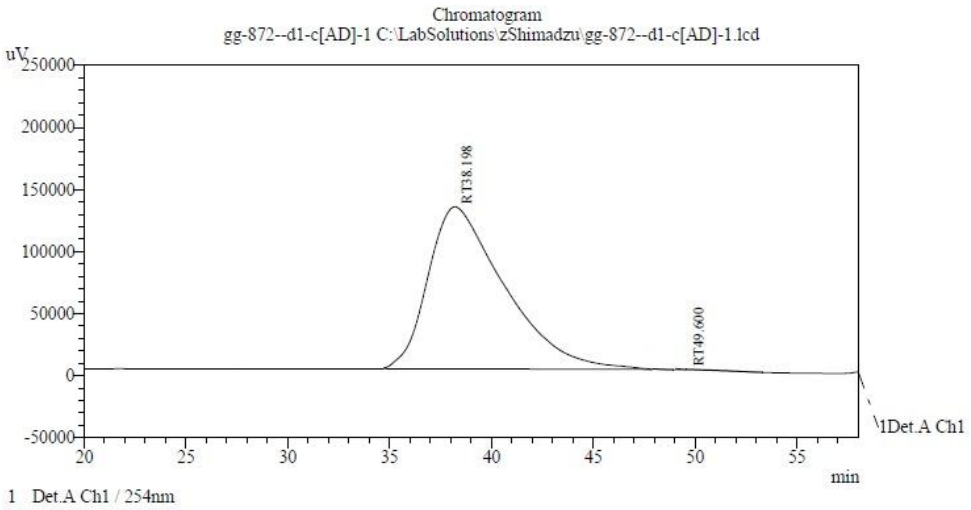


PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT36.161	36.161	39732018	470766	50.268	mg/L
2	RT48.042	48.042	39308784	366531	49.732	mg/L
Total			79040802	837297	100.000	

(racemic **3j**)



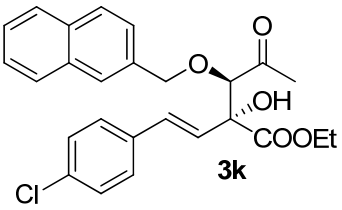
PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT38.198	38.198	37213436	130705	99.904	mg/L
2	RT49.600	49.600	35783	0	0.096	mg/L
Total			37249219	130705	100.000	

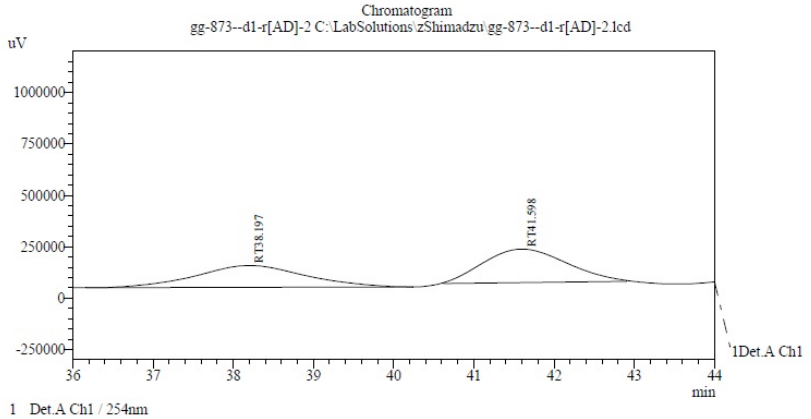
(enatiometrically enriched **3j**)

(2*R*,3*R*)-Ethyl 2-(4-chlorostyryl)-2-hydroxy-3-(naphthalen-2-ylmethoxy)-4-oxopentanoate **3k**



3k

The diastereomeric mixture was isolated as a yellow oil in an 82% combined yield, and both diastereomers were easily separated; The ee value of the *syn* isomer was 99%; t_R (minor) = 38.67 min, t_R (major) = 41.08 min (Chiralcel AD-H, λ = 254 nm, 5% *i*PrOH/hexanes, flow rate = 0.8 mL/min); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.09 (t, *J* = 7.2 Hz, 3H), 2.28 (s, 3H), 3.92 (s, 1H), 4.01-4.08 (m, 1H), 4.17-4.24 (m, 1H), 4.27 (s, 1H), 4.57 (d, *J* = 11.9 Hz, 1H), 4.87 (d, *J* = 12.0 Hz, 1H), 6.23 (d, *J* = 15.7 Hz, 1H), 6.82 (d, *J* = 15.8 Hz, 1H), 7.27-7.89 (m, 11H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.9, 28.1, 62.9, 73.4, 79.2, 87.5, 124.8, 125.7, 125.9, 126.1, 126.3, 126.4, 126.8, 127.3, 127.7, 127.9, 128.1, 128.4, 128.7, 130.3, 133.1, 133.7, 134.6, 171.9, 206.6; $[\alpha]^{25}_D$ = –34.6 (c = 2.00 in $CHCl_3$); HRMS (ESI) m/z calcd for $C_{26}H_{25}ClO_5$ $[M+Na]^+$ 475.1283, found 475.1286.

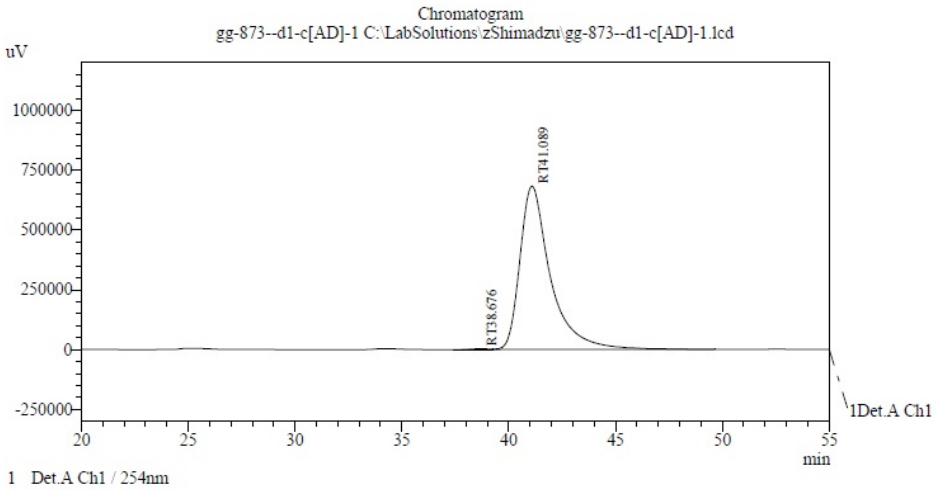


PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT38.197	38.197	10131036	106639	46.081	mg/L
2	RT41.598	41.598	11854298	163199	53.919	mg/L
Total			21985334	269838	100.000	

(racemic **3k**)



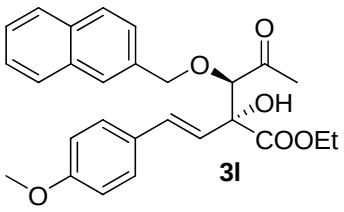
PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT38.676	38.676	154878	2511	0.228	mg/L
2	RT41.089	41.089	67830422	682816	99.772	mg/L
Total			67985299	685328	100.000	

(enatiometrically enriched **3k**)

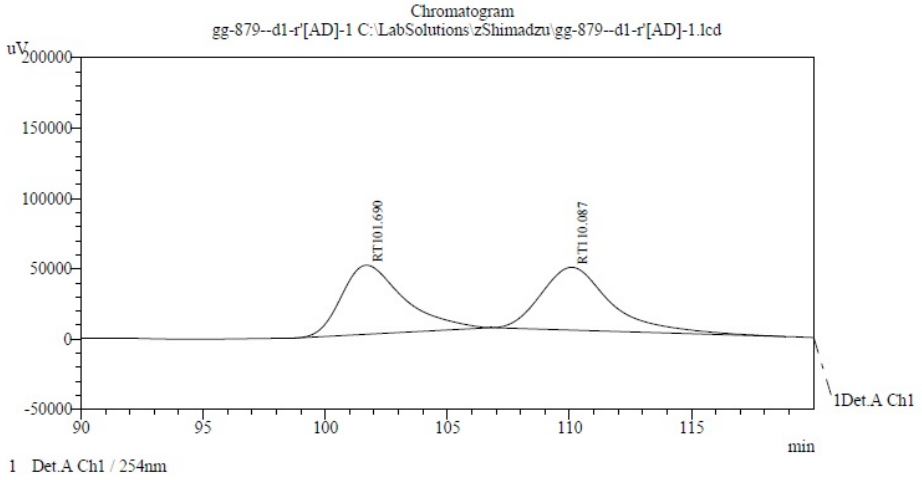
(2*R*,3*R*)-Ethyl 2-hydroxy-2-(4-methoxystyryl)-3-(naphthalen-2-ylmethoxy)-4-oxopentanoate **3l**



3l

The diastereomeric mixture was isolated as a yellow oil in 93% combined yield, and both diastereomers were easily separated; The ee value of the *syn* isomer was 77%; t_R (minor) = 99.65 min, t_R (major) = 107.20 min (Chiralcel AD-H, λ = 254 nm, 5% *i*PrOH/hexanes, flow rate = 0.8 mL/min); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.08 (t, *J* = 7.2 Hz, 3H), 2.27 (s, 3H), 3.82 (s, 3H), 3.87 (s, 1H), 4.02-4.21 (m, 2H), 4.28 (s, 1H), 4.56 (d, *J* = 11.3 Hz, 1H), 4.88 (d, *J* = 11.3 Hz, 1H), 6.11 (d, *J* = 15.7 Hz, 1H), 6.80-7.88 (m, 12H); ^{13}C NMR (125 MHz, $CDCl_3$) δ13.9, 28.2, 55.3, 62.8, 73.4, 79.2, 87.6, 113.9, 121.8, 125.9, 126.3, 126.4, 127.3, 127.7, 127.9, 128.1, 128.4, 128.7, 131.0, 133.1, 133.8, 159.6, 172.3, 207.9; $[\alpha]^{25}_D$ = –7.4 (c = 1.00 in $CHCl_3$); HRMS (ESI) m/z

calcd for $C_{27}H_{28}O_6$ [M+Na]$^+$ 471.1778, found 471.1790.

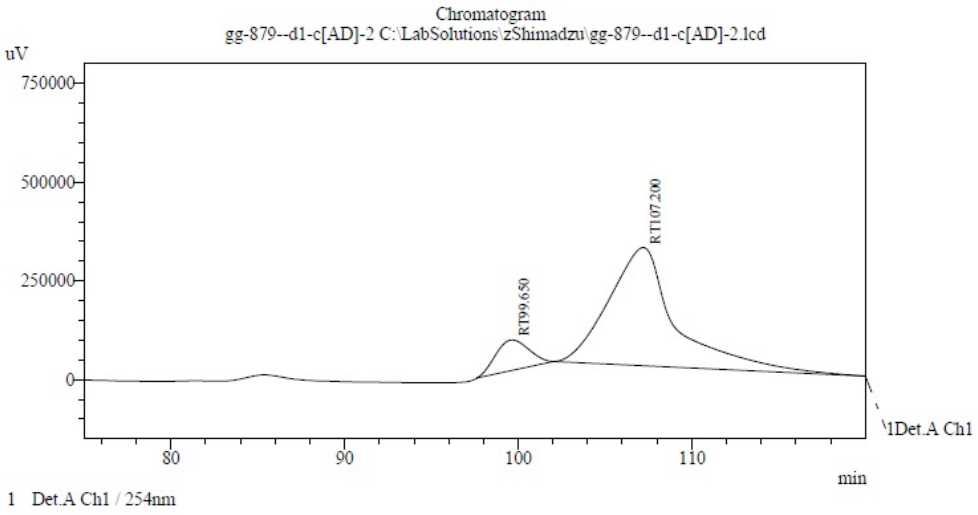


PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT101.690	101.690	8745755	49079	49.644	mg/L
2	RT110.087	110.087	8871290	44613	50.356	mg/L
Total			17617045	93692	100.000	

(racemic **3l**)



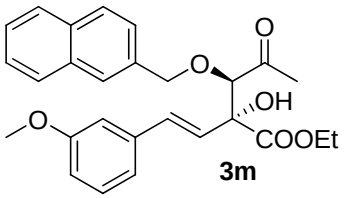
PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT99.650	99.650	10334001	77133	11.403	mg/L
2	RT107.200	107.200	80290262	299298	88.597	mg/L
Total			90624263	376431	100.000	

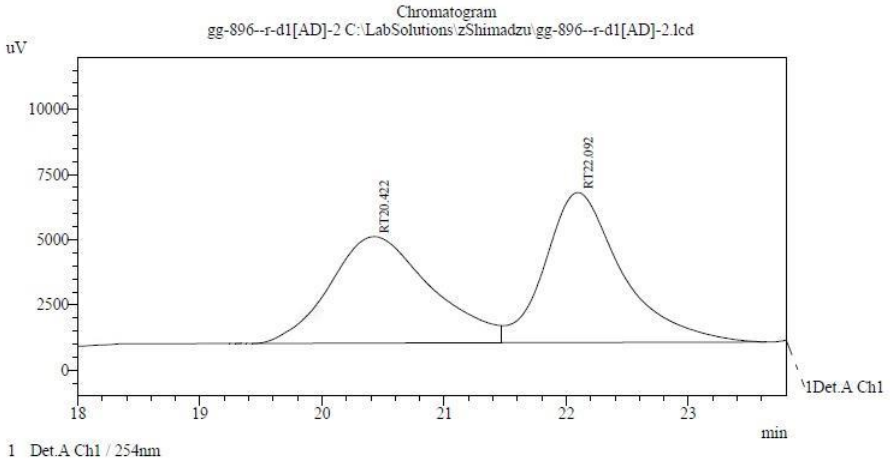
(enatiometrically enriched **3l**)

(2*R*,3*R*)-Ethyl 2-hydroxy-2-(3-methoxystyryl)-3-(naphthalen-2-ylmethoxy)-4-oxopentanoate **3m**



3m

The diastereomeric mixture was isolated as a yellow oil in an 81% combined yield, and both diastereomers were easily separated; The ee value of the *syn* isomer was 99%; t_R (minor) = 18.48 min, t_R (major) = 21.31 min (Chiralcel AD-H, λ = 254 nm, 10% *i*PrOH/hexanes, flow rate = 1.0 mL/min); ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.11 (t, *J* = 7.2 Hz, 3H), 2.30 (s, 3H), 3.82 (s, 3H), 3.93 (s, 1H), 4.03-4.25 (m, 2H), 4.31 (s, 1H), 4.58 (d, *J* = 11.8 Hz, 1H), 4.89 (d, *J* = 11.8 Hz, 1H), 6.28 (d, *J* = 15.7 Hz, 1H), 6.82-7.89 (m, 12H); ^{13}C NMR (75 MHz, $CDCl_3$,) δ 13.9, 28.1, 29.7, 62.9, 73.5 (d, *J* = 5.5 Hz), 75.3, 79.4, 87.7, 115.8 (d, *J* = 13.1 Hz), 124.1 (d, *J* = 2.2 Hz), 124.5 (d, *J* = 1.6 Hz), 125.8 (d, *J* = 15.8 Hz), 126.4 (d, *J* = 4.4 Hz), 126.9 (d, *J* = 3.3 Hz), 127.3, 127.7, 128.0, 128.2 (d, *J* = 2.2 Hz), 128.4, 129.4 (d, *J* = 5.5 Hz), 133.2 (d, *J* = 3.8 Hz), 133.8, 160.5 (d, *J* = 149.8 Hz), 172.0, 207.9; $[\alpha]^{25}_D$ = –70.8 (c = 4.00 in $CHCl_3$); HRMS (ESI) m/z calcd for $C_{27}H_{28}O_6$ $[M+Na]^+$ 471.1778, found 471.1782.

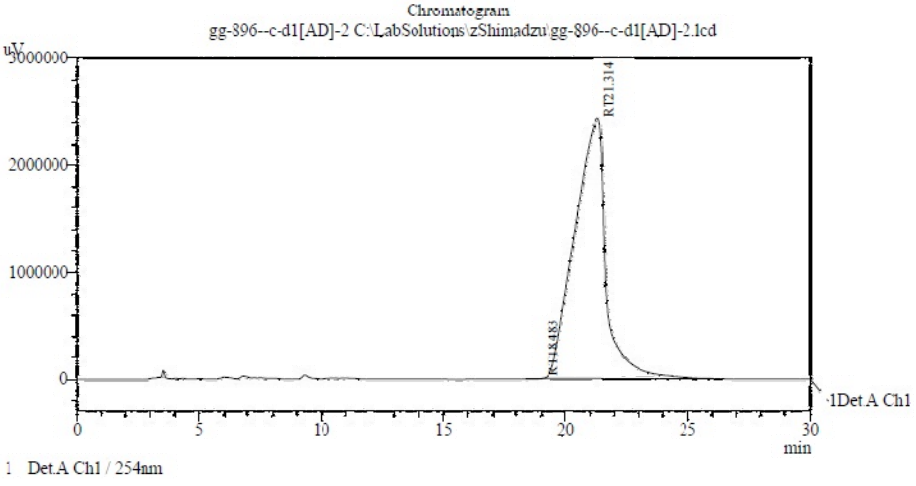


PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT20.422	20.422	251210	4080	49.967	mg/L
2	RT22.092	22.092	251540	5748	50.033	mg/L
Total			502750	9829	100.000	

(racemic **3m**)



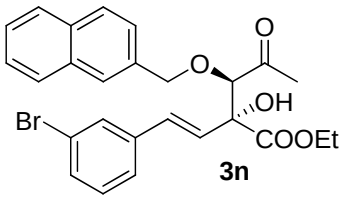
PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT18.483	19.025	77596	4885	0.035	mg/L
2	RT21.314	21.314	218541087	2430263	99.965	mg/L
Total			218618683	2435148	100.000	

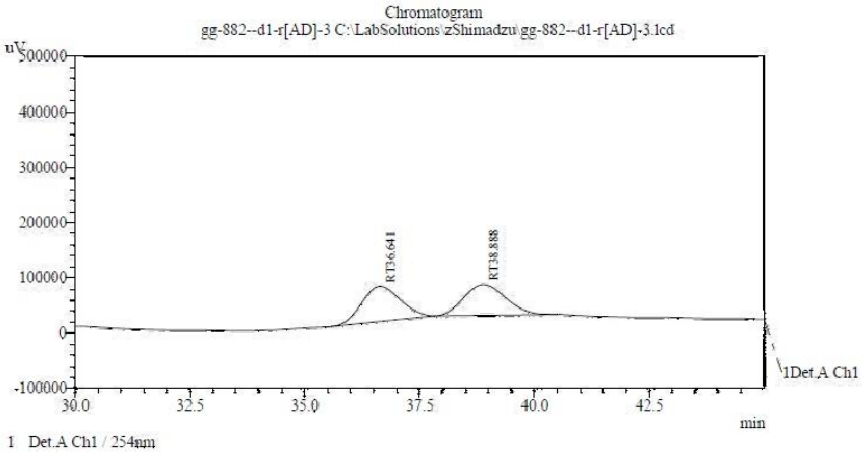
(enatiometrically enriched **3m**)

(2*R*,3*R*)-Ethyl 2-(3-bromostyryl)-2-hydroxy-3-(naphthalen-2-ylmethoxy)-4-oxopentanoate **3n**



3n

The diastereomeric mixture was isolated as a white solid in an 87% combined yield, and both diastereomers were easily separated; The ee value of the *syn* isomer was 95%; t_R (minor) = 35.71 min, t_R (major) = 38.34 min (Chiralcel AD-H, λ = 254 nm, 5% *i*PrOH/hexanes, flow rate = 0.8 mL/min); ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.10 (t, *J* = 7.3 Hz, 3H), 2.30 (s, 3H), 3.82 (s, 3H), 3.99 (s, 1H), 4.04-4.25 (m, 2H), 4.30 (s, 1H), 4.58 (d, *J* = 11.8 Hz, 1H), 4.88 (d, *J* = 11.8 Hz, 1H), 6.30 (d, *J* = 15.6 Hz, 1H), 6.83 (d, *J* = 15.7 Hz, 1H), 7.15-7.90 (m, 10H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 13.8, 28.0, 62.9, 73.3, 79.1, 87.4, 122.6, 125.6, 125.8, 126.0, 126.2, 126.3, 126.7, 127.2, 127.6, 127.8, 128.3, 129.5, 130.8, 133.0, 133.6, 134.5, 138.0, 171.8, 207.9; $[\alpha]^{25}_D$ = –25.3 (c = 1.00 in $CHCl_3$); HRMS (ESI) m/z calcd for $C_{26}H_{25}BrO_5$ $[M+Na]^+$ 519.0778, found 519.0793.

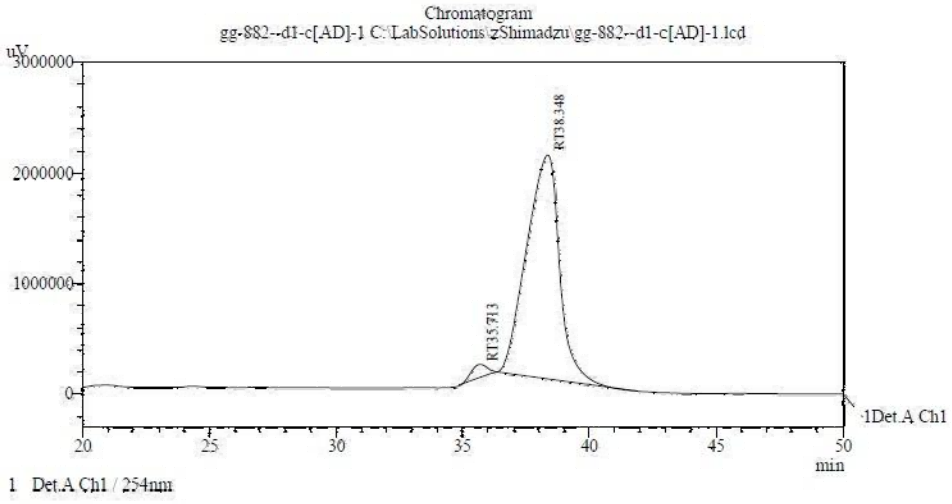


PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT36.641	36.641	3758474	64180	51.399	mg/L
2	RT38.888	38.888	3553849	55914	48.601	mg/L
Total			7312323	120095	100.000	

(racemic **3n**)



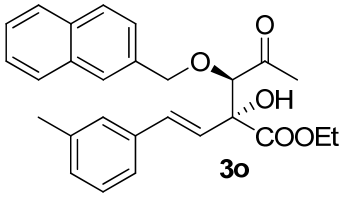
PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT35.713	35.713	5163361	118335	2.723	mg/L
2	RT38.348	38.348	184462261	2027574	97.277	mg/L
Total			189625622	2145909	100.000	

(enatiometrically enriched **3n**)

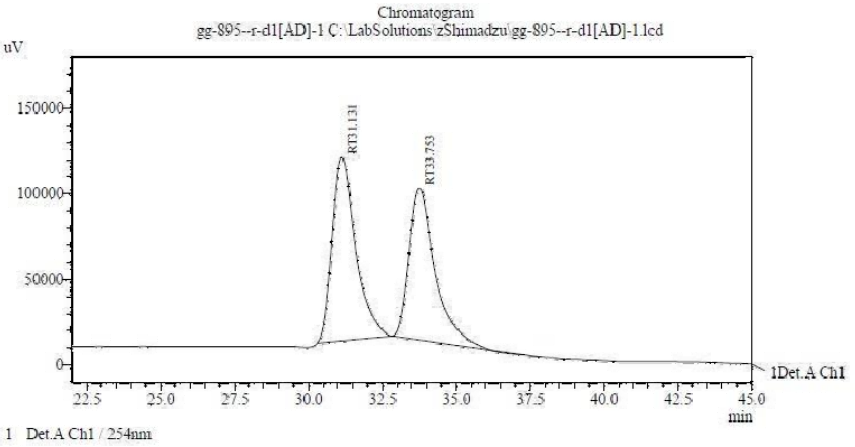
(2*R*,3*R*)-Ethyl 2-hydroxy-2-(3-methylstyryl)-3-(naphthalen-2-ylmethoxy)-4-oxopentanoate **3o**



3o

The diastereomeric mixture was isolated as a white solid in an 81% combined yield, and both diastereomers were easily separated; The ee value of the *syn* isomer was 91%; t_R (minor) = 31.29 min, t_R (major) = 34.09 min (Chiralcel AD-H, λ = 254 nm, 5% *i*PrOH/hexanes, flow rate = 0.8 mL/min); ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.11 (t, *J* = 7.0 Hz, 3H), 2.30 (s, 3H), 2.36 (s, 3H), 3.92 (s, 1H), 4.03-4.27 (m, 2H), 4.31 (s, 1H), 4.58 (d, *J* = 11.8 Hz, 1H), 4.89 (d, *J* = 11.8 Hz, 1H), 6.27 (d, *J* = 15.7 Hz, 1H), 6.87 (d, *J* = 15.7 Hz, 1H), 7.09-7.91 (m, 10H); ^{13}C NMR (75 MHz,

$CDCl_3$, *syn* isomer) δ 13.8, 21.2, 28.0, 62.7, 73.3, 79.1, 87.5, 123.8, 124.1, 125.8, 126.2, 126.3, 127.2, 127.4, 127.6, 127.9, 128.3, 128.8, 131.6, 133.0, 133.1, 133.7, 135.8, 138.0, 172.0, 207.7; $[\alpha]^{25}_{D} = -50.6$ (c = 4.00 in $CHCl_3$); HRMS (ESI) m/z calcd for $C_{27}H_{28}O_5$ $[M+Na]^+$ 455.1829, found 455.1824.

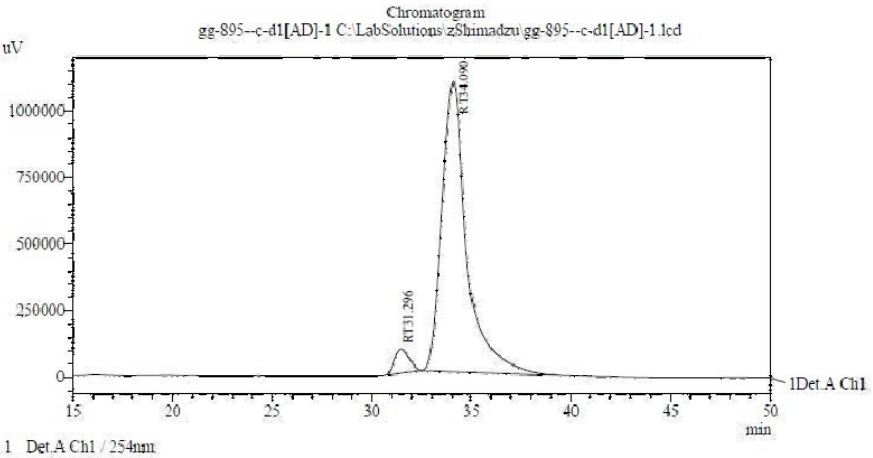


PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT31.131	31.131	5891116	106932	51.378	mg/L
2	RT33.753	33.753	5575055	88430	48.622	mg/L
Total			11466171	195362	100.000	

(racemic **3o**)



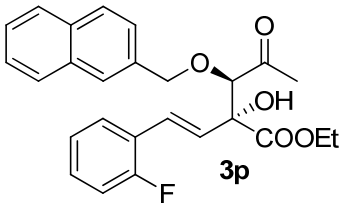
PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT31.296	31.296	4547677	89902	4.581	mg/L
2	RT34.090	34.090	94720365	1087787	95.419	mg/L
Total			99268042	1177688	100.000	

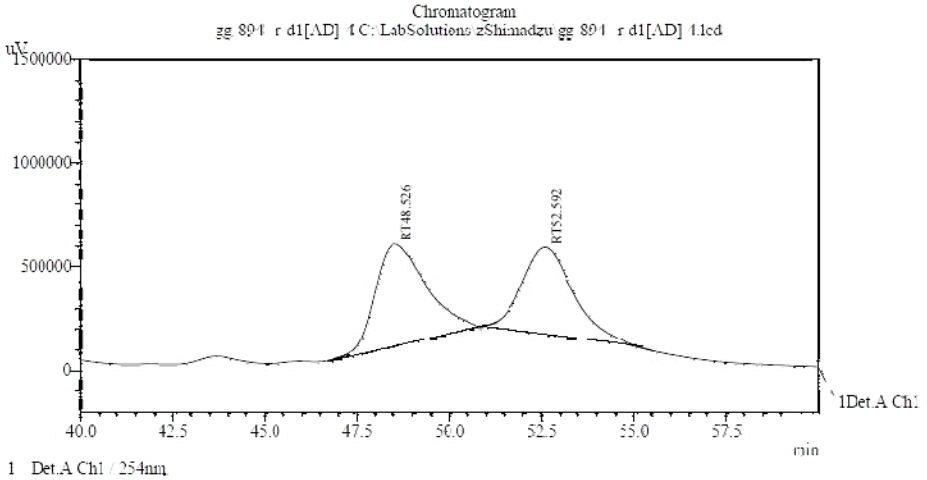
(enatiometrically enriched **3o**)

(2*R*,3*R*)-Ethyl 2-(2-fluorostyryl)-2-hydroxy-3-(naphthalen-2-ylmethoxy)-4-oxopentanoate **3p**



3p

The diastereomeric mixture was isolated as a white solid in an 83% combined yield, and both diastereomers were easily separated; The ee value of the *syn* isomer was 90%; t_R (minor) = 51.45 min, t_R (major) = 55.43 min (Chiralcel AD-H, λ = 254 nm, 5% *i*PrOH/hexanes, flow rate = 0.5 mL/min); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.09 (t, *J* = 7.2 Hz, 3H), 2.29 (s, 3H), 3.93 (s, 1H), 4.03-4.23 (m, 2H), 4.30 (s, 1H), 4.58 (d, *J* = 11.3 Hz, 1H), 4.87 (d, *J* = 11.3 Hz, 1H), 6.38 (d, *J* = 15.7 Hz, 1H), 7.00-7.86 (m, 12H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.8, 28.1, 62.9, 73.5, 79.3, 87.6, 115.6, 115.8, 124.0, 124.4, 125.9, 126.3, 126.4, 126.9, 127.3, 127.7, 127.9, 128.2, 128.4, 129.4, 133.2, 133.7, 159.4, 161.4, 171.9, 207.9; $[\alpha]^{25}_D$ = −102.8 (c = 4.00 in $CHCl_3$); HRMS (ESI) m/z calcd for $C_{26}H_{25}FO_5$ $[M+Na]^+$ 459.1578, found 459.1584.

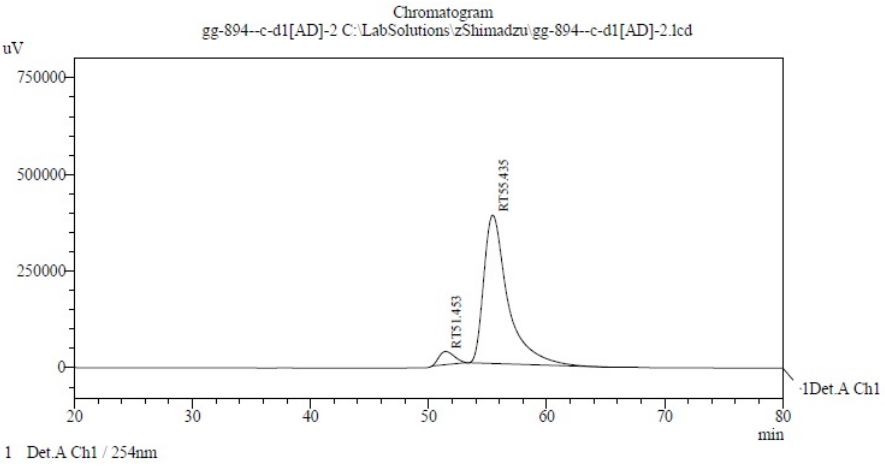


PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT48.526	48.526	63779978	470414	49.688	mg/L
2	RT52.592	52.592	32651654	368566	50.312	mg/L
Total			96431632	838980	100.000	

(racemic **3p**)



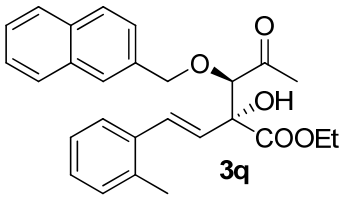
PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT51.453	51.453	3052540	33969	5.284	mg/L
2	RT55.435	55.435	54711939	383845	94.716	mg/L
Total			57764479	417814	100.000	

(enatiometrically enriched **3p**)

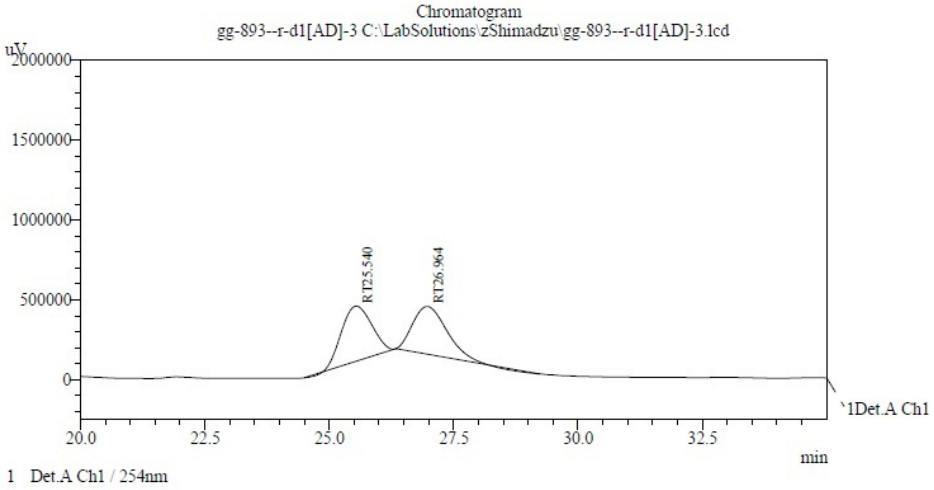
(2*R*,3*R*)-Ethyl 2-hydroxy-2-(2-methylstyryl)-3-(naphthalen-2-ylmethoxy)-4-oxopentanoate **3q**



3q

The diastereomeric mixture was isolated as a white solid in a 97% combined yield, and both diastereomers were easily separated; the ee value of the *syn* isomer was 99%; t_R (minor) = 29.90 min, t_R (major) = 32.21 min (Chiralcel AD-H, λ = 254 nm, 5% *i*PrOH/hexanes, flow rate = 0.8 mL/min); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.09 (t, *J* = 7.0 Hz, 3H), 2.30 (s, 3H), 2.34 (s, 3H), 3.93 (s, 1H), 4.02-4.23 (m, 2H), 4.30 (s, 1H), 4.57 (d, *J* = 11.3 Hz, 1H), 4.87 (d, *J* = 11.9 Hz, 1H), 6.15 (d, *J* = 15.1 Hz, 1H), 7.09-7.86 (m, 12H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.9, 19.7, 26.4,

62.8, 73.4, 79.3, 87.7, 125.7, 126.1, 126.2, 126.8, 127.7, 127.9, 128.4, 129.6, 130.2, 133.1, 133.2, 134.6, 135.9, 143.0, 172.2, 206.5; $[\alpha]^{25}_{D}$ = –73.0 (c = 5.00 in $CHCl_3$); HRMS (ESI) m/z calcd for $C_{27}H_{28}O_5$ $[M+Na]^+$ 455.1829, found 455.1835.

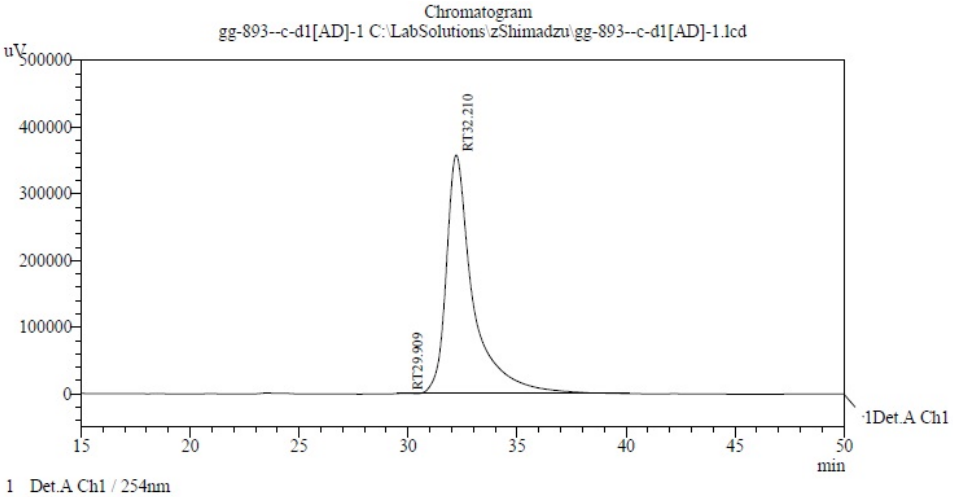


PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT25.540	25.540	14642990	346275	50.940	mg/L
2	RT26.964	26.964	14102341	298773	49.060	mg/L
Total			28745330	645048	100.000	

(racemic **3q**)



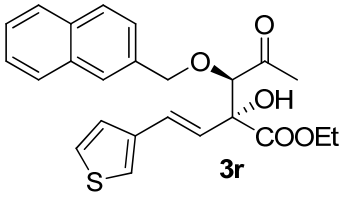
PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT29.909	29.909	14283	481	0.046	mg/L
2	RT32.210	32.210	30747042	357845	99.954	mg/L
Total			30761325	358326	100.000	

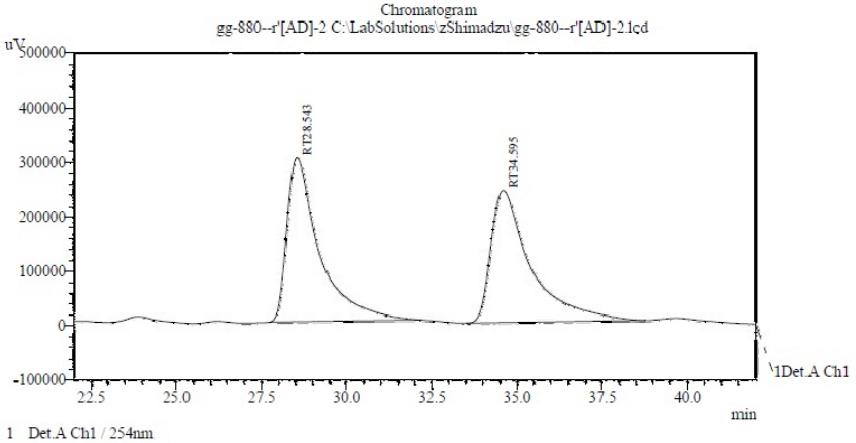
(enatiometrically enriched **3q**)

(2*R*,3*R*)-Ethyl 2-hydroxy-3-(naphthalen-2-ylmethoxy)-4-oxo-2-((E)-2-(thiophen-3-yl)vinyl)pentanoate **3r**



3r

The diastereomeric mixture was isolated as a white solid in a 90% combined yield, and both diastereomers were easily separated; the ee value of the *syn* isomer was 91%; t_R (minor) = 28.77 min, t_R (major) = 34.60 min (Chiralcel AD-H, λ = 254 nm, 20% *i*PrOH/hexanes, flow rate = 1.0 mL/min); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.34 (t, *J* = 7.2 Hz, 3H), 2.32 (s, 3H), 3.74 (s, 1H), 4.13 (s, 1H), 4.26-4.36 (m, 2H), 4.79 (d, *J* = 12.0 Hz, 1H), 4.95 (d, *J* = 12.0 Hz, 1H), 6.08 (d, *J* = 15.7 Hz, 1H), 6.97-7.86 (m, 11H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.0, 28.3, 63.0, 73.5, 79.5, 87.4, 124.1, 125.0, 125.3, 125.6, 126.1, 126.2, 126.8, 127.4, 127.7, 128.0, 128.4, 133.1, 134.0, 141.1, 171.9, 206.4; $[\alpha]^{25}_D$ = −15.4 (c = 1.50 in $CHCl_3$); HRMS (ESI) m/z calcd for $C_{24}H_{24}O_5S$ $[M+Na]^+$ 447.1237, found 447.1236.

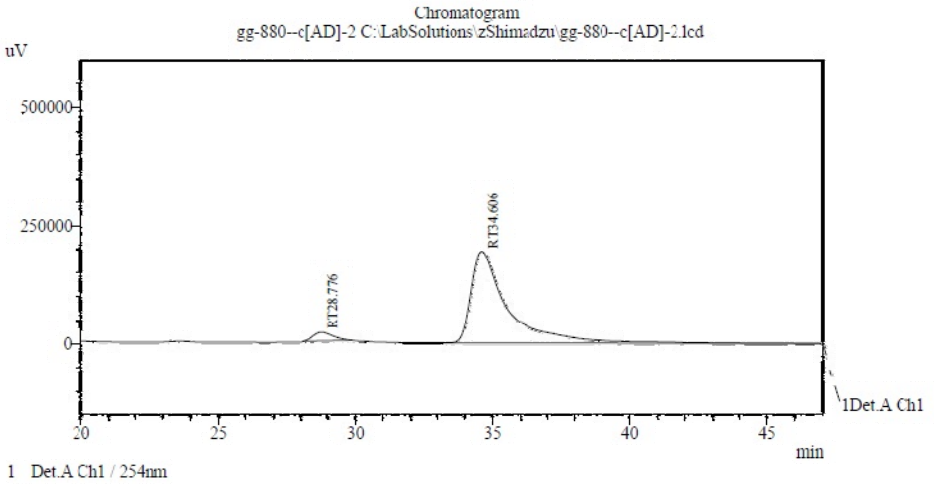


PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT28.543	28.543	20014851	300854	50.289	mg/L
2	RT34.595	34.595	19784432	242003	49.711	mg/L
Total			39799284	542857	100.000	

(racemic **3r**)



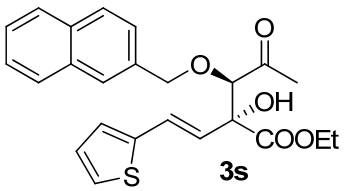
PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT28.776	28.776	899549	18218	4.787	mg/L
2	RT34.606	34.606	17893084	193735	95.213	mg/L
Total			18792632	211952	100.000	

(enatiometrically enriched **3r**)

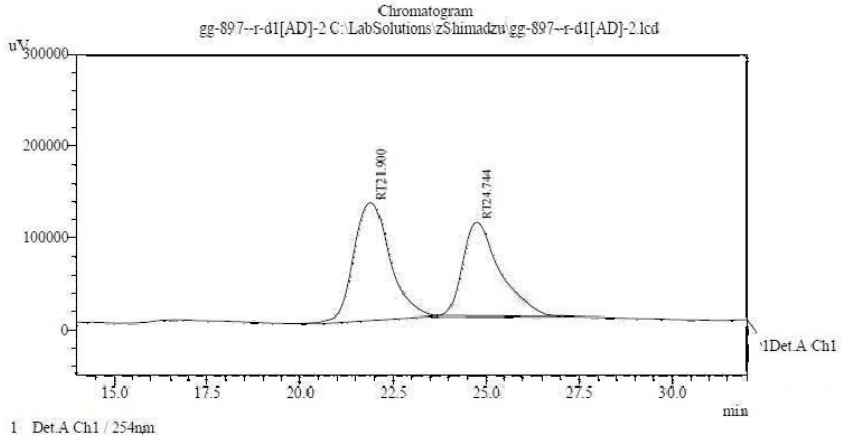
(2*R*,3*R*)-Ethyl 2-hydroxy-3-(naphthalen-2-ylmethoxy)-4-oxo-2-((E)-2-(thiophen-2-yl)vinyl)pentanoate **3s**



3s

The diastereomeric mixture was isolated as a white solid in a 92% combined yield, and both diastereomers were easily separated; the ee value of the *syn* isomer was 88%; t_R (major) = 22.53 min, t_R (minor) = 24.69 min (Chiralcel AD-H, λ = 254 nm, 10% *i*PrOH/hexanes, flow rate = 1.0 mL/min); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.09 (t, *J* = 6.9 Hz, 3H), 2.28 (s, 3H), 3.90 (s, 1H), 4.01-4.24 (m, 2H), 4.27 (s, 1H), 4.56 (d, *J* = 12.0 Hz, 1H), 4.86 (d, *J* = 12.0 Hz, 1H), 6.11 (d, *J* = 15.7 Hz, 1H), 6.95-7.89 (m, 11H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.9, 28.2, 62.9, 73.4, 79.0,

87.4, 123.4, 124.9, 125.1, 125.9, 126.3, 126.4, 126.9, 127.3, 127.4, 127.7, 127.9, 128.4, 133.1, 134.6, 140.9, 171.9, 207.6; $[\alpha]^{25}_{D}$ = –82.6 (c = 4.50 in $CHCl_3$); HRMS (ESI) m/z calcd for $C_{24}H_{24}O_5S$ $[M+Na]^+$ 447.1237, found 447.1249.

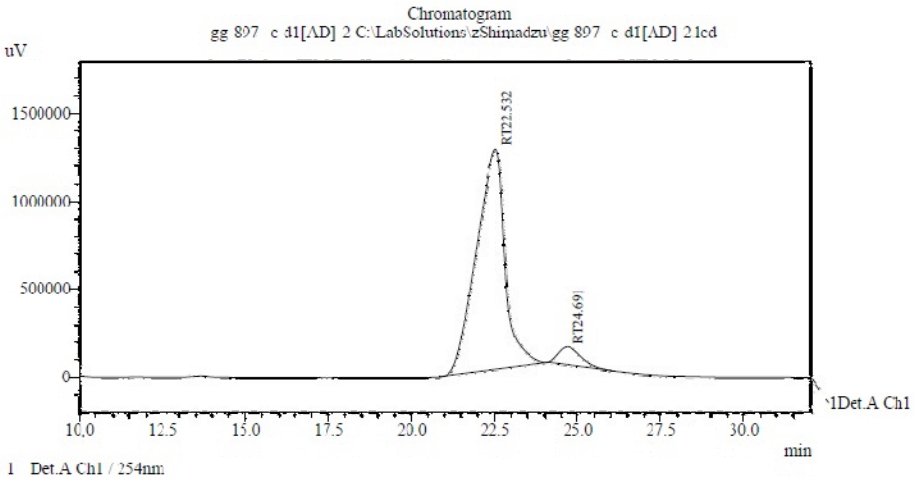


PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT21.900	21.900	8633201	127713	48.178	mg/L
2	RT24.744	24.744	9286171	101633	51.822	mg/L
Total			17919372	229347	100.000	

(racemic **3s**)



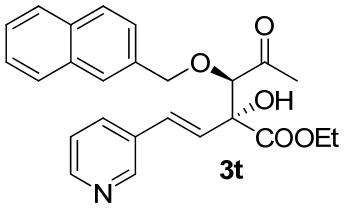
PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT22.532	22.532	75009676	1250141	94.145	mg/L
2	RT24.691	24.691	4665201	99195	5.855	mg/L
Total			79674878	1349336	100.000	

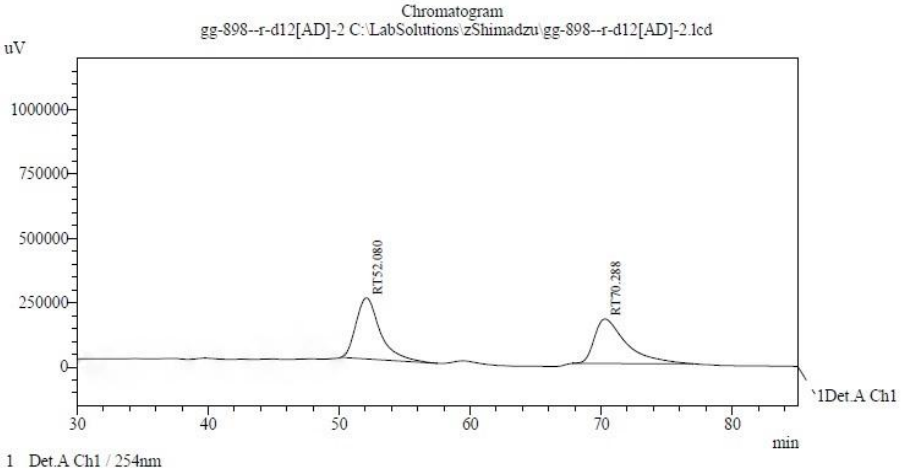
(enatiometrically enriched **3s**)

(2*R*,3*R*)-Ethyl 2-hydroxy-3-(naphthalen-2-ylmethoxy)-4-oxo-2-((E)-2-(pyridin-3-yl)vinyl)pentanoate **3t**



3t

The diastereomeric mixture was isolated as a white solid in a 95% combined yield, and both diastereomers were easily separated; the ee value of the *syn* isomer was 85%; t_R (major) = 52.98 min, t_R (minor) = 70.28 min (Chiralcel AD-H, λ = 254 nm, 10% *i*PrOH/hexanes, flow rate = 1.0 mL/min); ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.08 (t, *J* = 6.9 Hz, 3H), 1.27 (t, *J* = 6.9 Hz, 3H), 2.29 (s, 3H), 2.33 (s, 3H), 4.04-4.33 (m, 8H), 4.57 (d, *J* = 12.0 Hz, 1H), 4.73 (d, *J* = 12.0 Hz, 1H), 4.86 (d, *J* = 11.3 Hz, 1H), 4.97 (d, *J* = 11.3 Hz, 1H), 6.23 (d, *J* = 15.7 Hz, 1H), 6.35 (d, *J* = 15.7 Hz, 1H), 6.87 (d, *J* = 16.4 Hz, 1H), 6.95 (d, *J* = 15.7 Hz, 1H), 7.21-8.61 (m, 22H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.9, 28.1, 63.0, 73.6, 79.3, 87.6, 123.4, 125.9, 126.4, 126.6, 127.3, 127.7, 127.9, 128.1, 128.4, 128.8, 131.7, 133.1, 133.4, 133.6, 148.6, 149.0, 171.7, 208.1; $[\alpha]^{25}_D$ = –81.0 (c = 5.00 in $CHCl_3$); HRMS (ESI) m/z calcd for $C_{25}H_{26}NO_5$ $[M+H]^+$ 420.1805, found 420.1809.

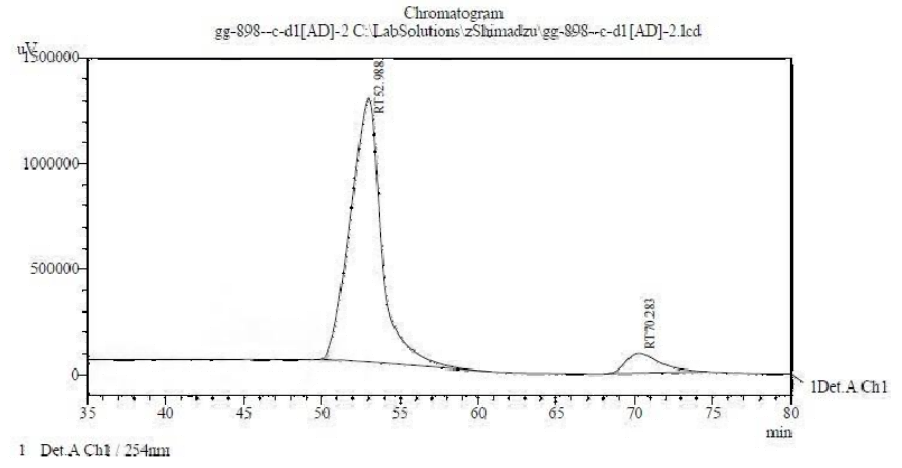


PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT52.080	52.080	30415861	237389	50.901	mg/L
2	RT70.288	70.288	29338737	172589	49.099	mg/L
Total			59754598	409977	100.000	

(racemic **3t**)



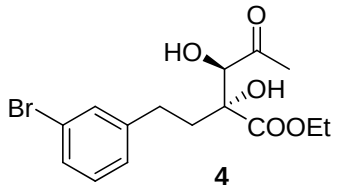
PeakTable

Detector A Ch1 254nm

Peak#	Name	Ret. Time	Area	Height	Area %	Units
1	RT52.988	52.988	173186593	1247874	92.351	mg/L
2	RT70.283	70.283	14344679	94550	7.649	mg/L
Total			187531271	1342424	100.000	

(enatiometrically enriched **3t**)

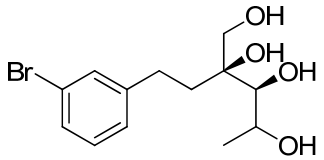
(2*R*,3*R*)-Ethyl 2-(3-bromophenethyl)-2,3-dihydroxy-4-oxopentanoate **4**



4

A colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.35 (t, *J* = 7.1 Hz, 3H), 2.10-2.19 (m, 2H), 2.27 (s, 3H), 2.29-2.55 (m, 1H), 2.91-2.91 (m, 1H), 3.71 (d, *J* = 8.7 Hz, 1H), 3.79 (s, 1H), 4.24-4.31 (m, 3H), 7.20-7.35 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.0, 28.4, 29.5, 37.3, 62.6, 78.8, 80.2, 126.0, 128.3, 140.9, 174.0, 208.3; $[\alpha]^{25}_{D}$ = –30.2 (c = 1.00 in $CHCl_3$); HRMS (ESI) m/z calcd for $C_{15}H_{19}BrO_5$ $[M+H]^+$ 359.0489, found 359.0494.

(2*S*,3*S*)-2-(3-Bromophenethyl)pentane-1,2,3,4-tetraol **5**



5

A a colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.27-1.39 (m, 5H), 1.74-2.08 (m, 3H), 262-2.87 (m, 3H), 3.27-3.89 (m, 3H), 4.03-4.06 (2s, 1H), 7.21-7.74 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 20.7, 29.0, 36.2, 66.7, 69.1, 75.2, 77.8, 125.8, 128.1, 128.2, 128.4, 128.5, 142.3; HRMS (ESI) m/z calcd for $C_{13}H_{19}BrO_4$ $[M+H]^+$ 319.0539, found 319.0545.

E. X-ray crystallographic data for compound 3n

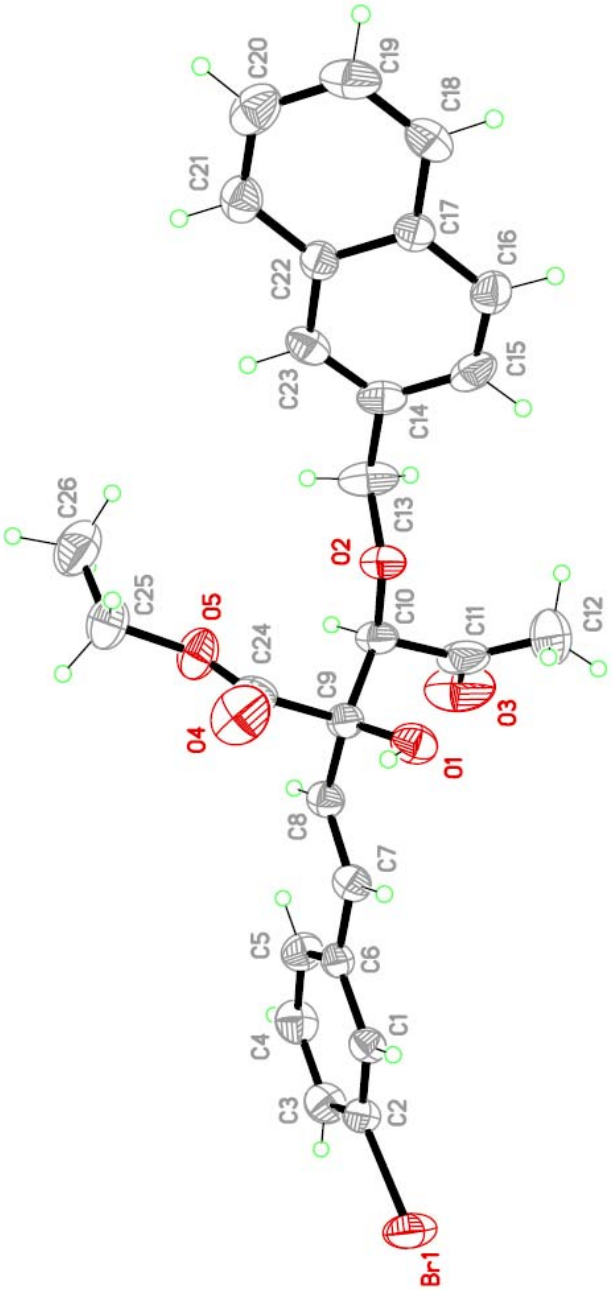


Table 1. Crystal data and structure refinement for B301.

Identification code	b301	
Empirical formula	C26 H25 Br O5	
Formula weight	497.37	
Temperature	223(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 13.1314(9) Å	α= 90°.
	b = 5.8958(4) Å	β= 109.439(2)°.
	c = 15.9344(11) Å	γ = 90°.
Volume	1163.32(14) $Å^3$	
Z	2	
Density (calculated)	1.420 Mg/m^3	
Absorption coefficient	1.801 mm^{-1}	
F(000)	512	
Crystal size	1.00 x 0.30 x 0.16 mm^3	
Theta range for data collection	1.64 to 27.50°.	
Index ranges	-14<=h<=17, -7<=k<=7, -20<=l<=19	
Reflections collected	8235	
Independent reflections	4635 [R(int) = 0.0330]	
Completeness to theta = 27.50°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7615 and 0.2660	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4635 / 1 / 292	
Goodness-of-fit on F^2	1.061	
Final R indices [I>2sigma(I)]	R1 = 0.0521, wR2 = 0.1045	
R indices (all data)	R1 = 0.0591, wR2 = 0.1082	
Absolute structure parameter	0.009(10)	
Largest diff. peak and hole	0.798 and -0.266 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for B301. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	12391(1)	4303(1)	9913(1)	52(1)
O(1)	6389(2)	6291(5)	8053(2)	39(1)
O(2)	4408(2)	4697(5)	6902(1)	36(1)
O(3)	6392(3)	3225(9)	5983(2)	84(1)
O(4)	5641(2)	4239(8)	9205(2)	61(1)
O(5)	5154(2)	1159(5)	8348(2)	45(1)
C(1)	10138(3)	3505(6)	9173(2)	30(1)
C(2)	11145(3)	2583(7)	9314(2)	33(1)
C(3)	11260(3)	451(7)	9000(3)	40(1)
C(4)	10339(3)	-739(10)	8522(2)	43(1)
C(5)	9328(3)	169(7)	8360(3)	40(1)
C(6)	9209(3)	2322(6)	8689(2)	29(1)
C(7)	8161(3)	3457(7)	8494(2)	31(1)
C(8)	7196(3)	2597(7)	8075(2)	30(1)
C(9)	6171(3)	3946(6)	7904(2)	30(1)
C(10)	5382(3)	3605(7)	6949(2)	34(1)
C(11)	5867(3)	4484(13)	6272(2)	55(1)
C(12)	5672(5)	6931(12)	6005(4)	90(2)
C(13)	3485(3)	3689(8)	6263(3)	50(1)
C(14)	2495(3)	5040(7)	6212(2)	38(1)
C(15)	2324(3)	7162(8)	5785(2)	41(1)
C(16)	1427(3)	8398(7)	5701(2)	38(1)
C(17)	621(3)	7575(7)	6036(2)	31(1)
C(18)	-359(3)	8724(6)	5914(2)	39(1)
C(19)	-1129(3)	7810(9)	6207(3)	48(1)
C(20)	-965(4)	5714(9)	6636(3)	50(1)
C(21)	-24(3)	4565(8)	6780(2)	39(1)
C(22)	786(3)	5431(6)	6474(2)	31(1)
C(23)	1751(2)	4229(8)	6560(2)	36(1)
C(24)	5609(3)	3129(7)	8564(2)	37(1)
C(25)	4571(4)	204(8)	8908(3)	55(1)

C(26)	3755(4)	-1388(9)	8361(3)	63(1)

Table 3. Bond lengths [Å] and angles [°] for B301.

Br(1)-C(2)	1.891(4)
O(1)-C(9)	1.416(5)
O(2)-C(10)	1.411(4)
O(2)-C(13)	1.428(4)
O(3)-C(11)	1.204(7)
O(4)-C(24)	1.202(5)
O(5)-C(24)	1.299(5)
O(5)-C(25)	1.468(5)
C(1)-C(2)	1.377(5)
C(1)-C(6)	1.394(5)
C(2)-C(3)	1.381(5)
C(3)-C(4)	1.387(6)
C(4)-C(5)	1.374(5)
C(5)-C(6)	1.401(5)
C(6)-C(7)	1.468(5)
C(7)-C(8)	1.321(5)
C(8)-C(9)	1.507(5)
C(9)-C(10)	1.542(4)
C(9)-C(24)	1.548(5)
C(10)-C(11)	1.516(5)
C(11)-C(12)	1.502(9)
C(13)-C(14)	1.505(5)
C(14)-C(23)	1.360(5)
C(14)-C(15)	1.406(6)
C(15)-C(16)	1.353(6)
C(16)-C(17)	1.421(5)
C(17)-C(18)	1.410(5)
C(17)-C(22)	1.425(5)
C(18)-C(19)	1.358(6)
C(19)-C(20)	1.394(7)
C(20)-C(21)	1.361(6)
C(21)-C(22)	1.406(5)
C(22)-C(23)	1.418(5)
C(25)-C(26)	1.472(7)

C(10)-O(2)-C(13)	112.8(3)
C(24)-O(5)-C(25)	118.0(3)
C(2)-C(1)-C(6)	120.6(3)
C(1)-C(2)-C(3)	121.0(4)
C(1)-C(2)-Br(1)	119.6(3)
C(3)-C(2)-Br(1)	119.4(3)
C(2)-C(3)-C(4)	118.6(4)
C(5)-C(4)-C(3)	121.2(5)
C(4)-C(5)-C(6)	120.2(4)
C(1)-C(6)-C(5)	118.4(3)
C(1)-C(6)-C(7)	118.3(3)
C(5)-C(6)-C(7)	123.2(3)
C(8)-C(7)-C(6)	127.3(4)
C(7)-C(8)-C(9)	122.5(3)
O(1)-C(9)-C(8)	111.5(3)
O(1)-C(9)-C(10)	108.9(3)
C(8)-C(9)-C(10)	112.1(3)
O(1)-C(9)-C(24)	107.8(3)
C(8)-C(9)-C(24)	108.2(3)
C(10)-C(9)-C(24)	108.3(3)
O(2)-C(10)-C(11)	113.2(3)
O(2)-C(10)-C(9)	107.1(3)
C(11)-C(10)-C(9)	110.7(3)
O(3)-C(11)-C(12)	123.3(5)
O(3)-C(11)-C(10)	119.5(6)
C(12)-C(11)-C(10)	117.2(5)
O(2)-C(13)-C(14)	109.3(3)
C(23)-C(14)-C(15)	119.3(4)
C(23)-C(14)-C(13)	121.2(4)
C(15)-C(14)-C(13)	119.5(4)
C(16)-C(15)-C(14)	121.3(4)
C(15)-C(16)-C(17)	120.8(4)
C(18)-C(17)-C(16)	123.0(4)
C(18)-C(17)-C(22)	118.5(3)
C(16)-C(17)-C(22)	118.4(3)

C(19)-C(18)-C(17)	120.8(4)
C(18)-C(19)-C(20)	120.6(4)
C(21)-C(20)-C(19)	120.5(4)
C(20)-C(21)-C(22)	120.7(4)
C(21)-C(22)-C(23)	122.7(4)
C(21)-C(22)-C(17)	118.8(4)
C(23)-C(22)-C(17)	118.5(3)
C(14)-C(23)-C(22)	121.6(4)
O(4)-C(24)-O(5)	126.5(4)
O(4)-C(24)-C(9)	121.2(4)
O(5)-C(24)-C(9)	112.3(3)
O(5)-C(25)-C(26)	108.0(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for B301. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	30(1)	54(1)	62(1)	-7(1)	1(1)	-4(1)
O(1)	37(2)	38(2)	37(2)	-4(1)	7(1)	4(1)
O(2)	26(1)	46(2)	32(1)	-5(1)	7(1)	1(1)
O(3)	49(2)	168(5)	46(2)	-13(2)	29(2)	1(2)
O(4)	71(2)	77(2)	44(1)	-22(2)	31(1)	-14(2)
O(5)	55(2)	50(2)	41(2)	-3(1)	29(1)	-7(1)
C(1)	32(2)	28(2)	28(2)	-2(1)	7(1)	1(1)
C(2)	29(2)	36(2)	30(2)	1(2)	7(2)	0(2)
C(3)	39(2)	39(2)	44(2)	1(2)	15(2)	9(2)
C(4)	46(2)	33(2)	52(2)	-1(2)	19(2)	12(3)
C(5)	39(2)	37(2)	41(2)	-6(2)	11(2)	-5(2)
C(6)	29(2)	36(2)	23(2)	4(2)	9(1)	-1(2)
C(7)	32(2)	33(2)	28(2)	-1(1)	10(2)	3(2)
C(8)	31(2)	35(2)	26(2)	-1(2)	10(2)	1(2)
C(9)	27(2)	36(3)	26(2)	1(2)	9(1)	-1(2)
C(10)	24(2)	49(2)	28(2)	-2(2)	8(1)	1(2)
C(11)	30(2)	102(4)	27(2)	0(3)	3(2)	-12(3)
C(12)	77(4)	123(6)	66(3)	55(4)	19(3)	-17(4)
C(13)	28(2)	67(3)	49(2)	-21(2)	4(2)	0(2)
C(14)	26(2)	45(2)	35(2)	-11(2)	1(2)	-2(2)
C(15)	31(2)	56(3)	35(2)	-8(2)	10(2)	-17(2)
C(16)	39(2)	38(2)	31(2)	-2(2)	6(2)	-12(2)
C(17)	30(2)	34(2)	23(2)	-4(2)	3(1)	-5(2)
C(18)	40(2)	38(2)	31(2)	0(2)	2(2)	7(2)
C(19)	32(2)	71(3)	39(2)	-7(2)	8(2)	12(2)
C(20)	48(3)	69(3)	38(2)	-7(2)	21(2)	-13(2)
C(21)	45(2)	42(2)	30(2)	-1(2)	13(1)	-4(2)
C(22)	32(2)	34(2)	24(2)	-1(1)	6(1)	-5(2)
C(23)	35(2)	36(2)	29(2)	-2(2)	1(1)	4(2)
C(24)	30(2)	52(2)	27(2)	-3(2)	8(2)	6(2)
C(25)	56(3)	73(3)	49(2)	10(2)	33(2)	-3(2)

C(26)	60(3)	61(3)	83(3)	6(3)	43(3)	-3(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for B301.

	x	y	z	U(eq)
H(1)	6402	6627	8562	58
H(1A)	10076	4945	9405	36
H(3)	11949	-182	9107	48
H(4)	10407	-2192	8304	52
H(5)	8714	-657	8029	47
H(7)	8179	4965	8690	38
H(8)	7146	1087	7876	37
H(10)	5238	1962	6847	40
H(12A)	6184	7411	5720	134
H(12B)	5763	7852	6530	134
H(12C)	4942	7109	5593	134
H(13A)	3403	2123	6436	60
H(13B)	3580	3662	5678	60
H(15)	2844	7734	5553	50
H(16)	1336	9817	5416	45
H(18)	-482	10140	5627	47
H(19)	-1779	8598	6120	57
H(20)	-1509	5089	6828	60
H(21)	86	3176	7087	47
H(23)	1880	2836	6864	43
H(25A)	4221	1418	9132	66
H(25B)	5075	-591	9419	66
H(26A)	3164	-542	7950	95
H(26B)	3482	-2310	8744	95
H(26C)	4080	-2360	8029	95

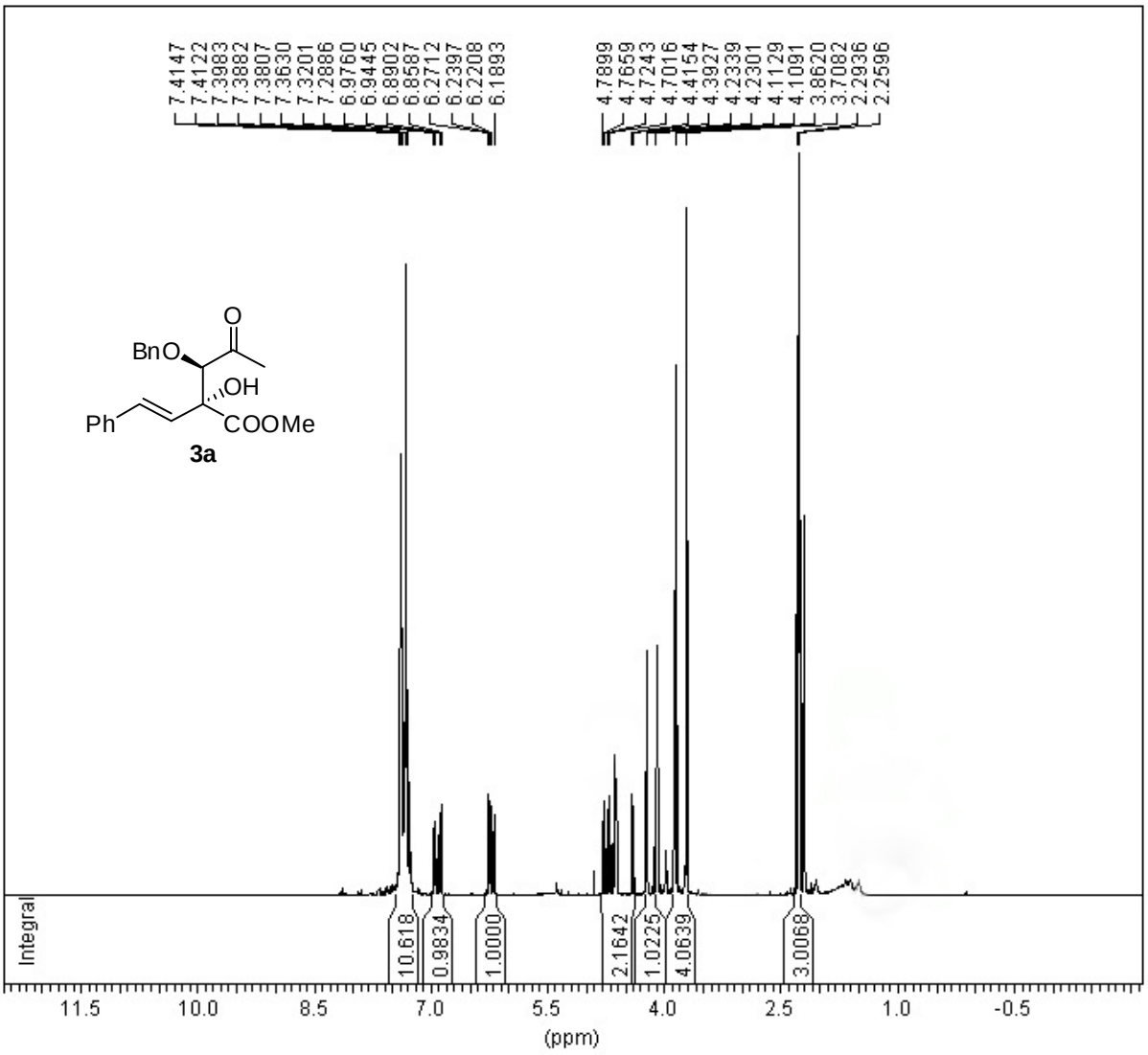
Table 6. Hydrogen bonds for B301 [Å and °].

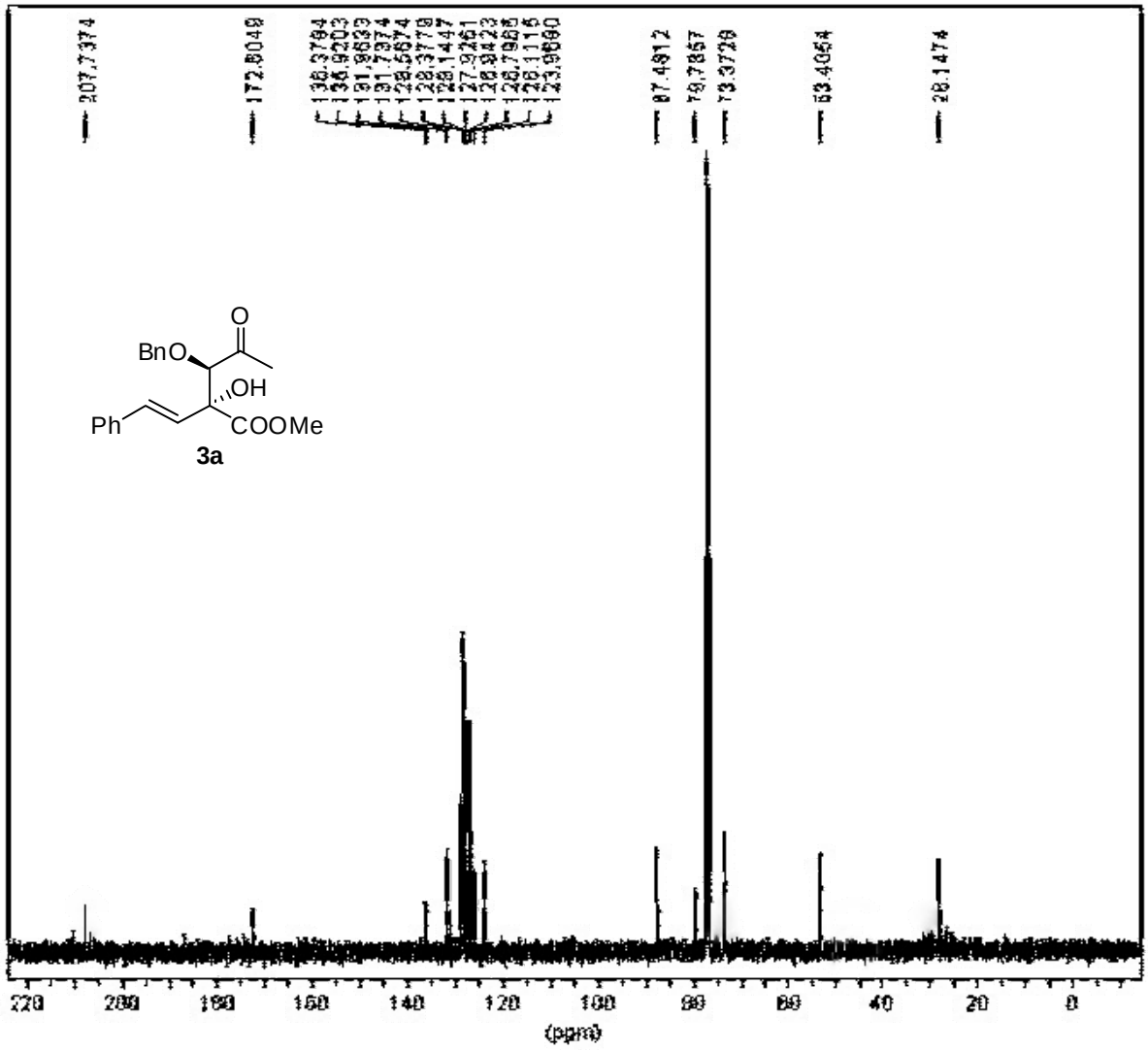
D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1)...O(4)	0.83	2.17	2.646(4)	116.4
O(1)-H(1)...Br(1)#1	0.83	2.89	3.573(3)	141.2

Symmetry transformations used to generate equivalent atoms:

#1 -x+2,y+1/2,-z+2

F. NMR Spectra of the Products

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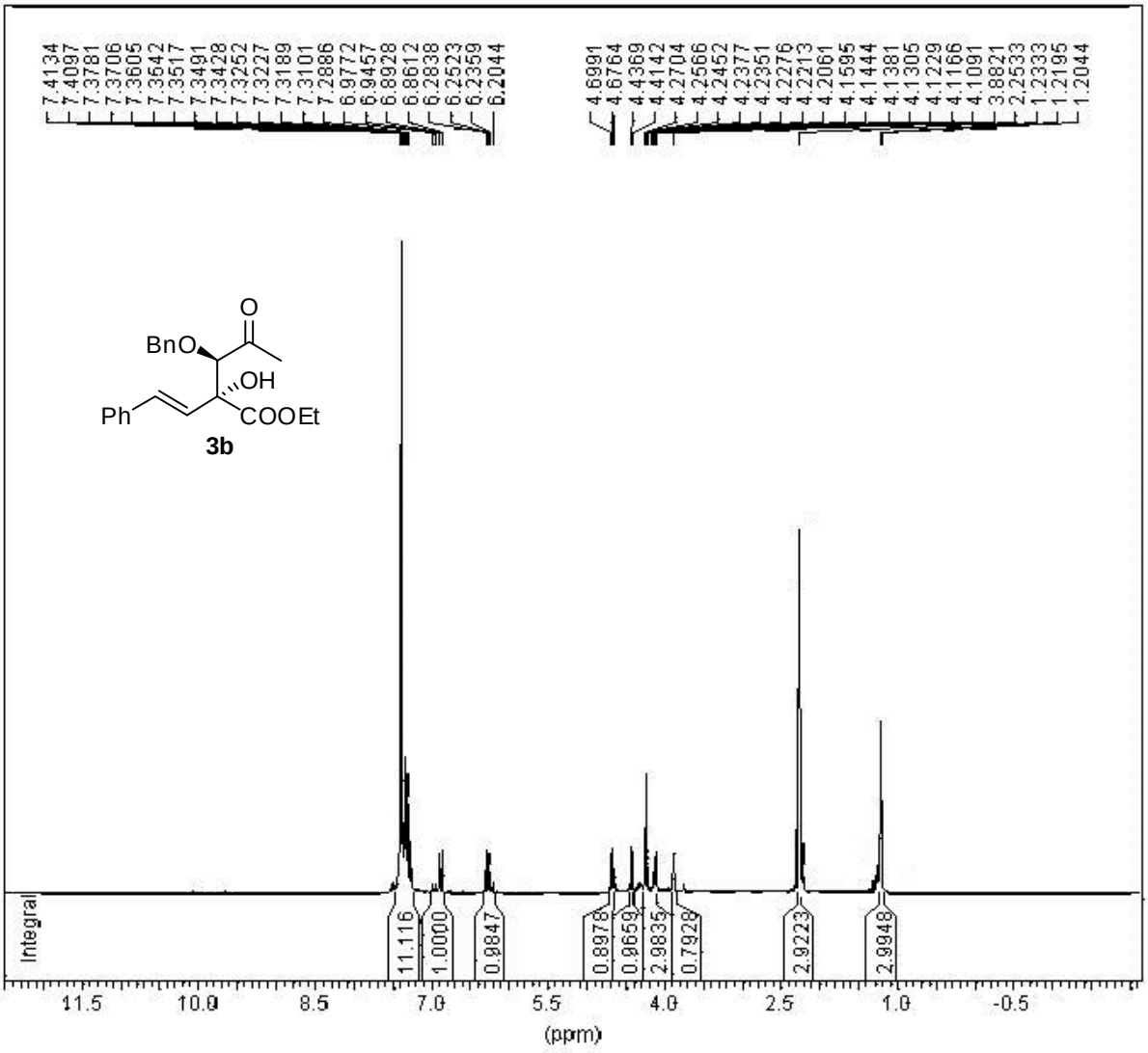
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4.2566
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4.2377
4.2351
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BnO
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Ph
COOEt
3b
Integral
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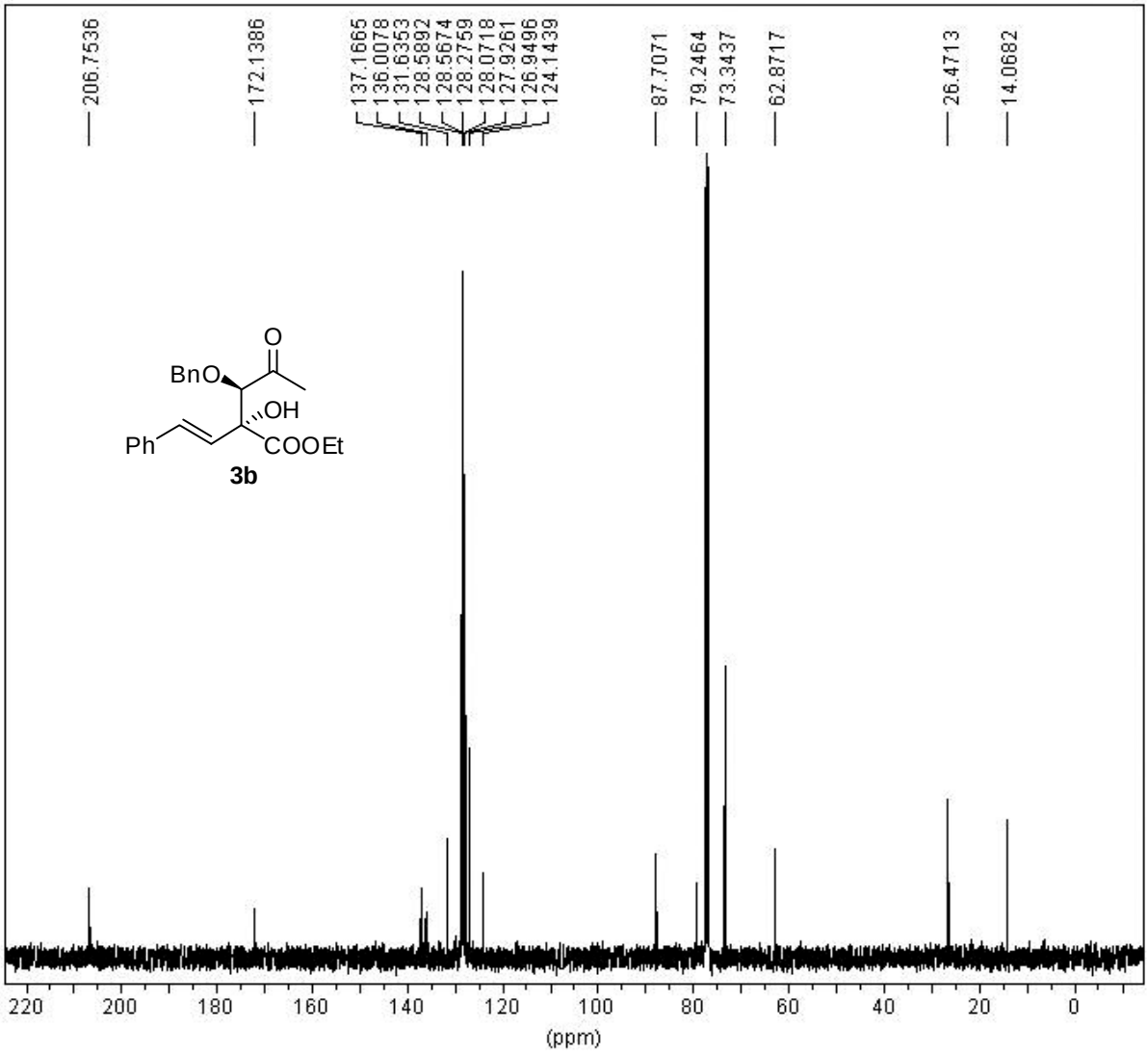
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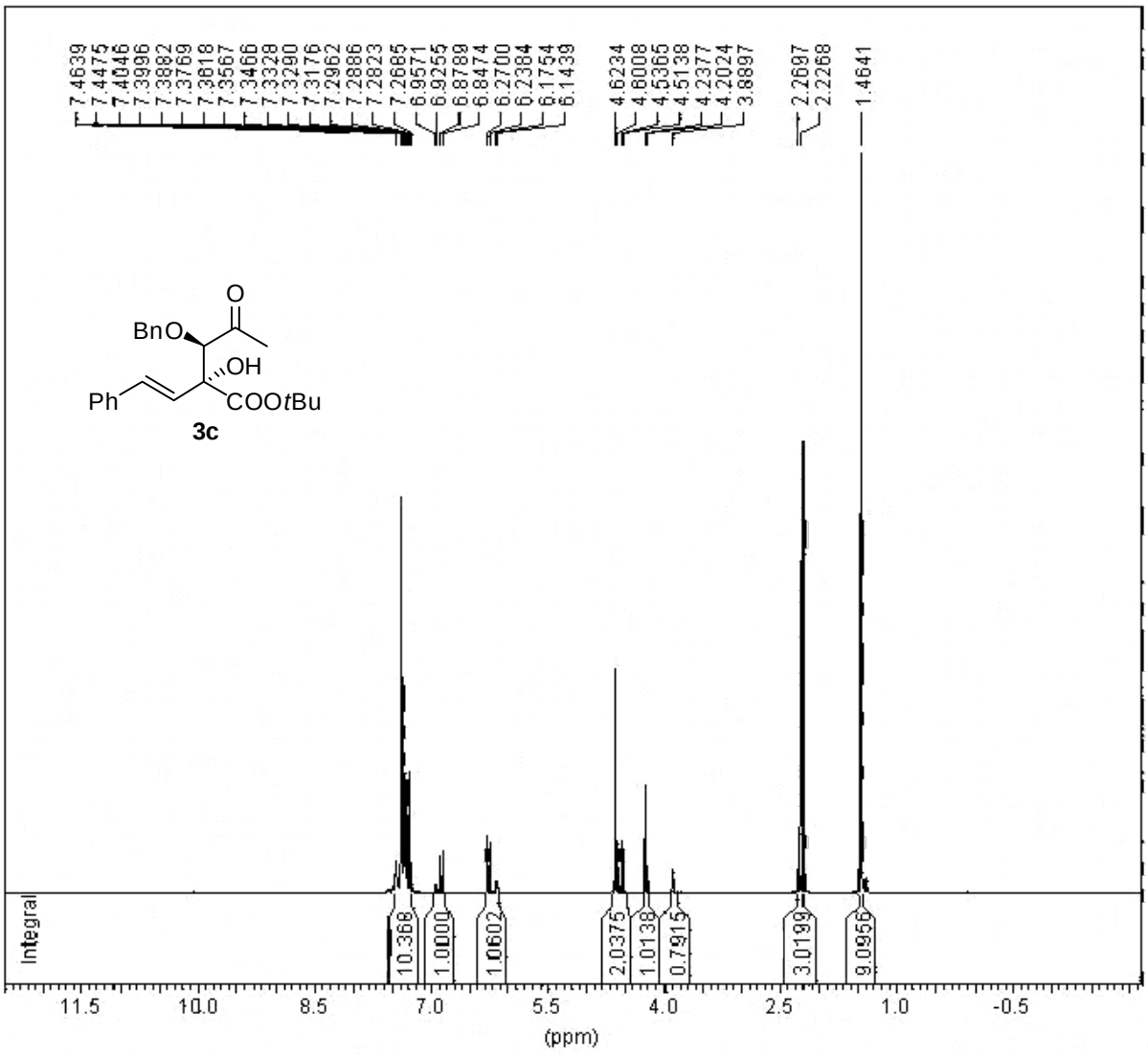
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COOtBu
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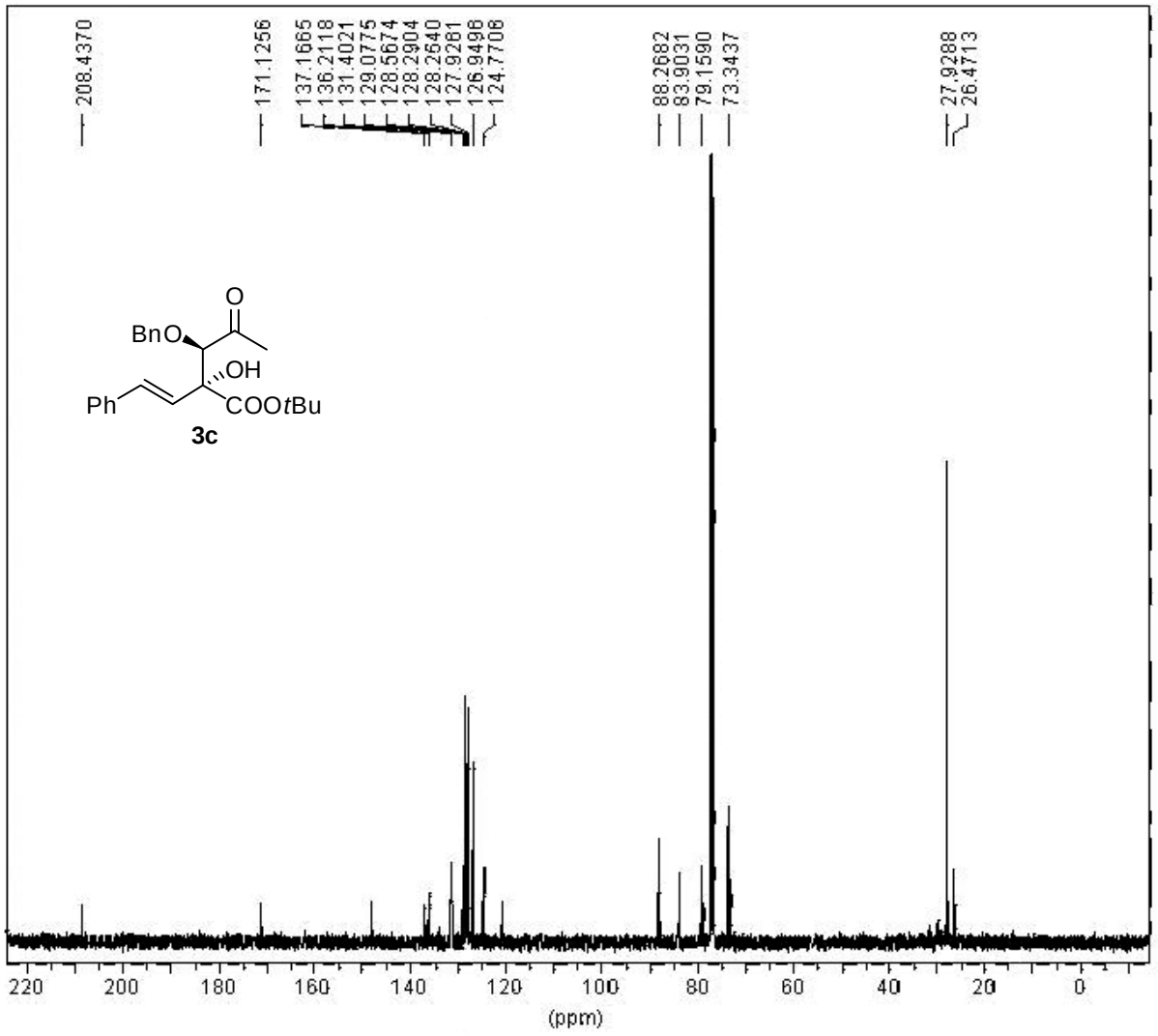
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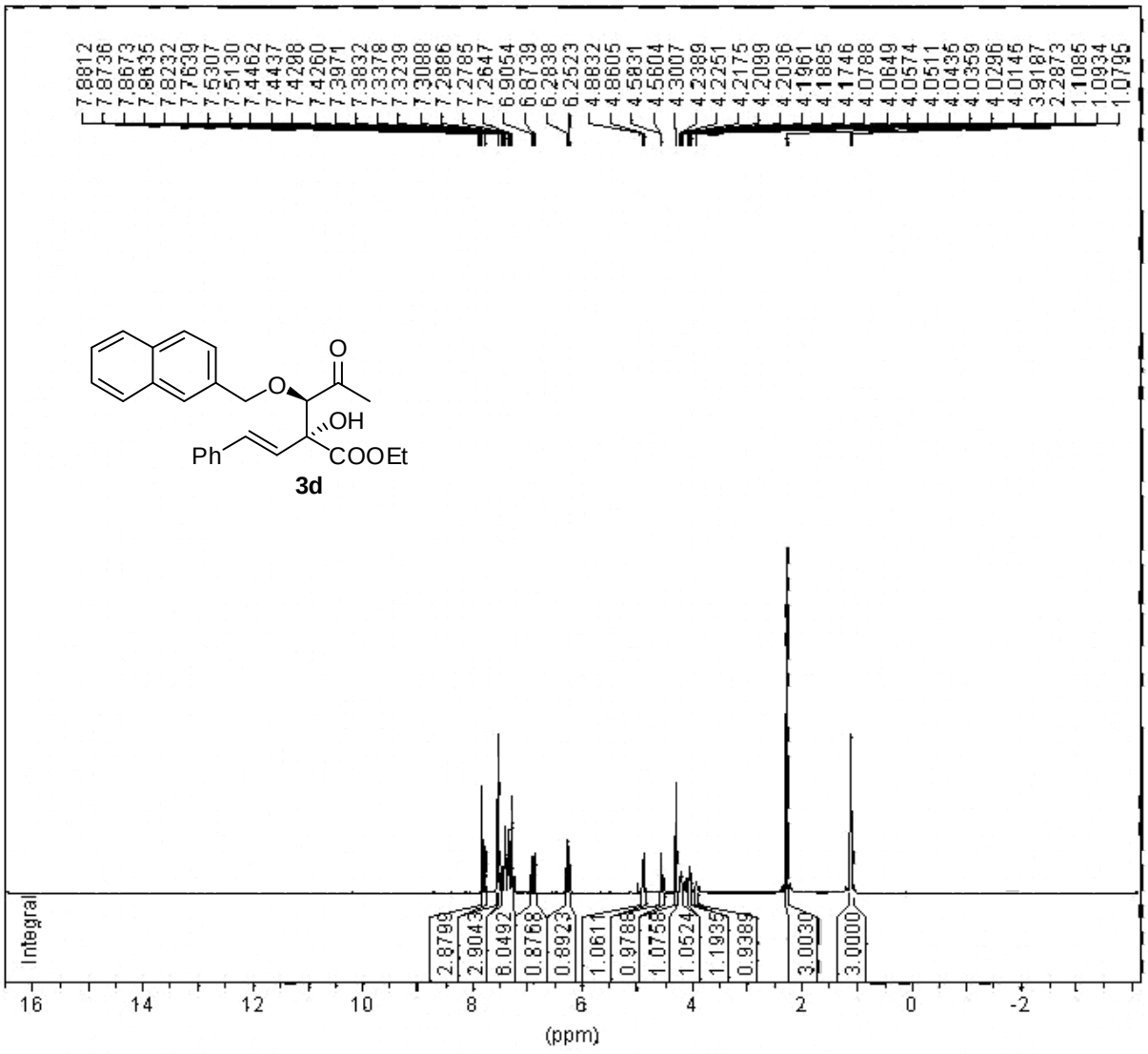
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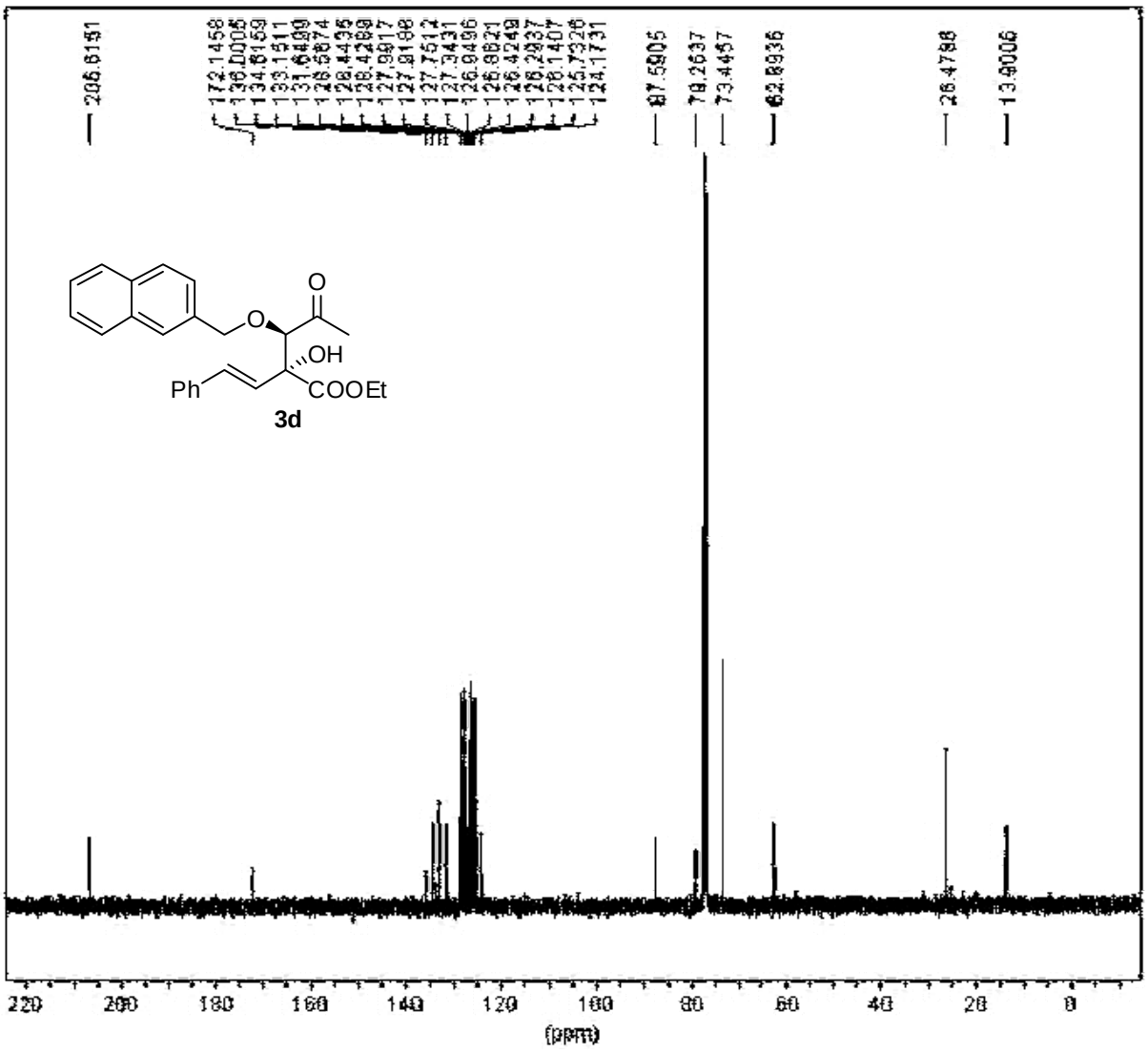
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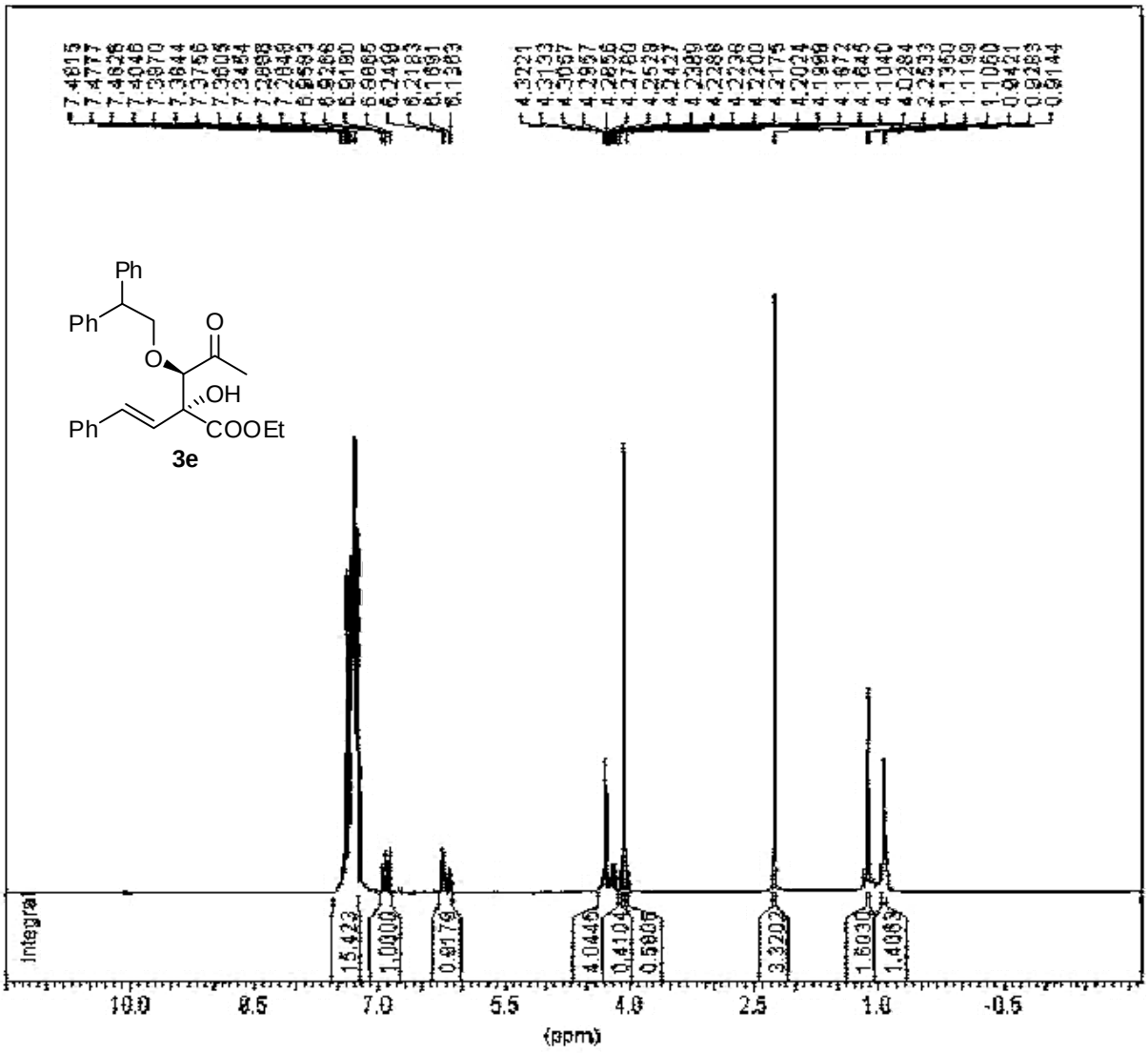
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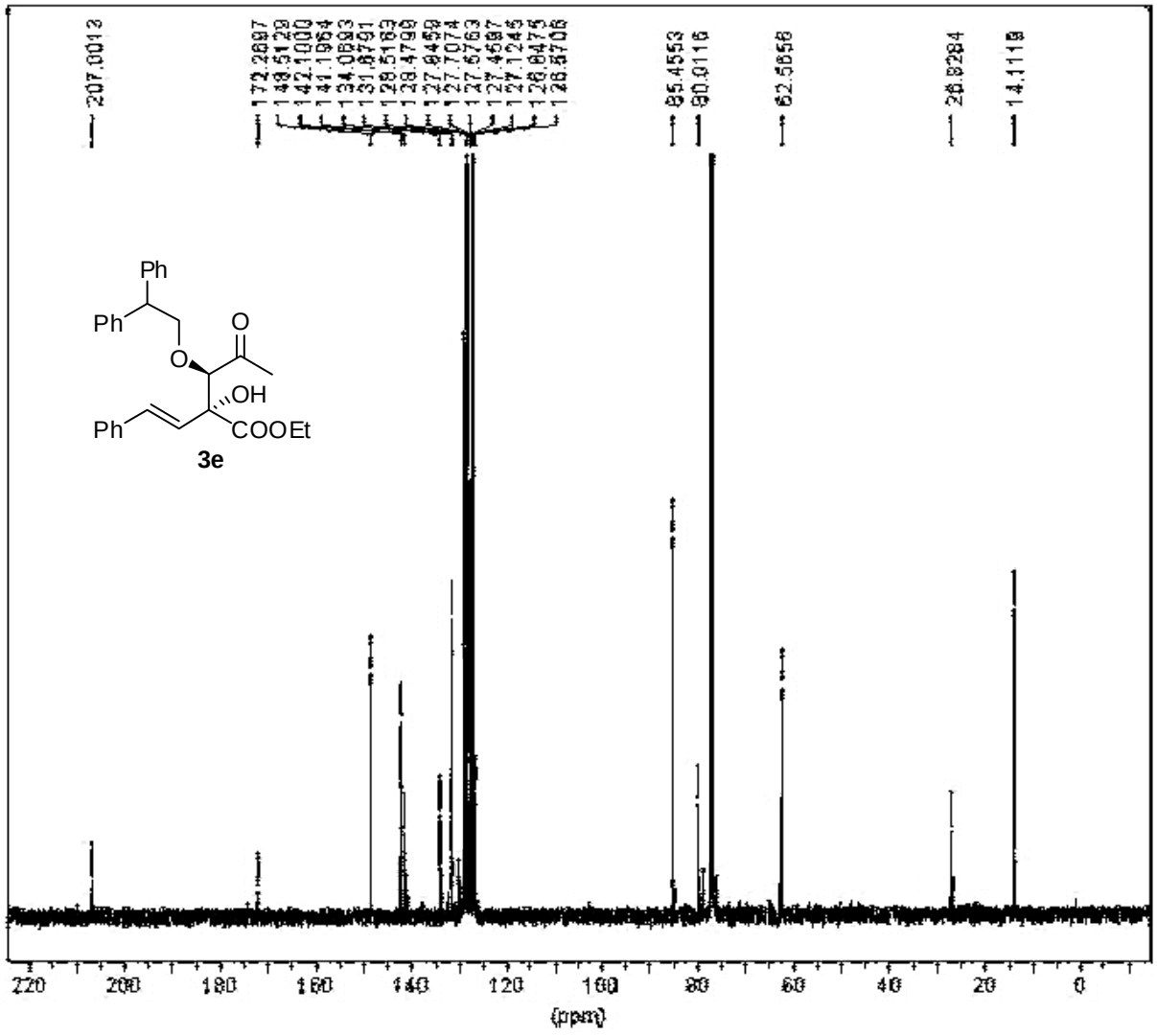
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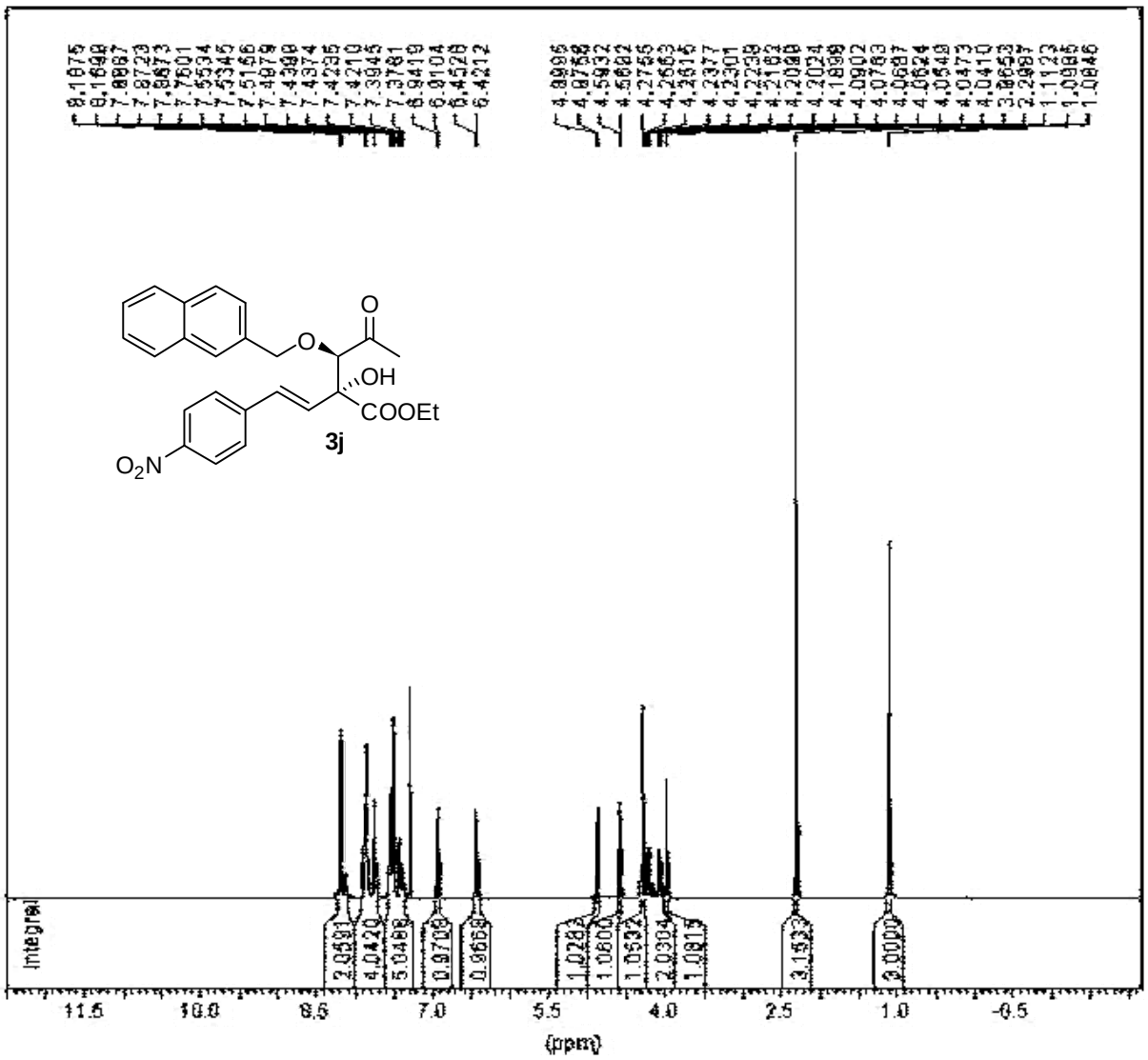
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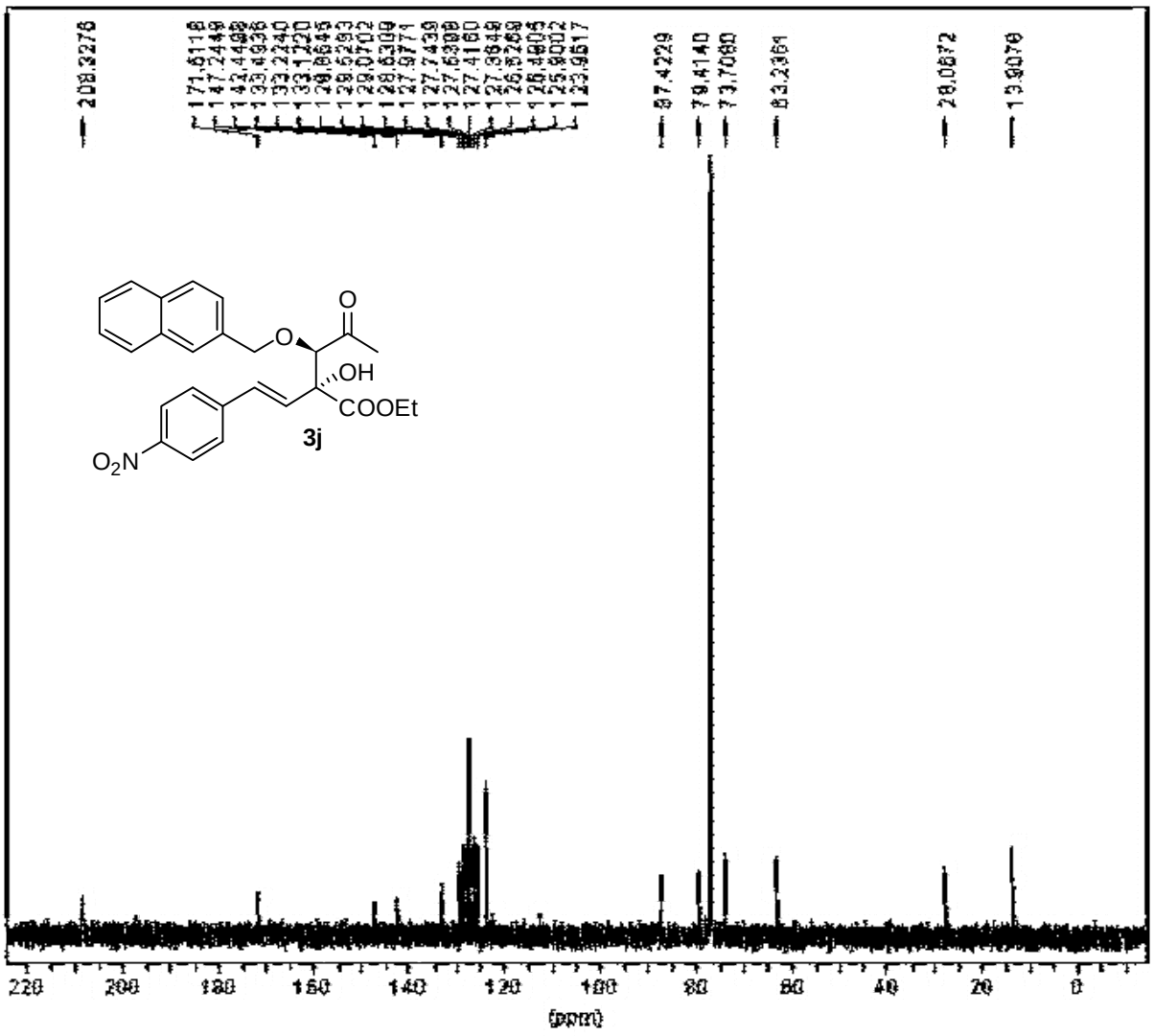
O
OH
COOEt
O_2N
3j
Integral
(ppm)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133.2240
131.1220
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128.5293
128.0702
128.8309
127.9771
127.7439
127.6388
127.4160
127.3648
126.8268
126.4804
125.8002
123.9617
87.4229
79.4140
73.7080
63.2361
28.0872
13.9078
O
O
OH
COOEt
O2N
3j
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200
180
160
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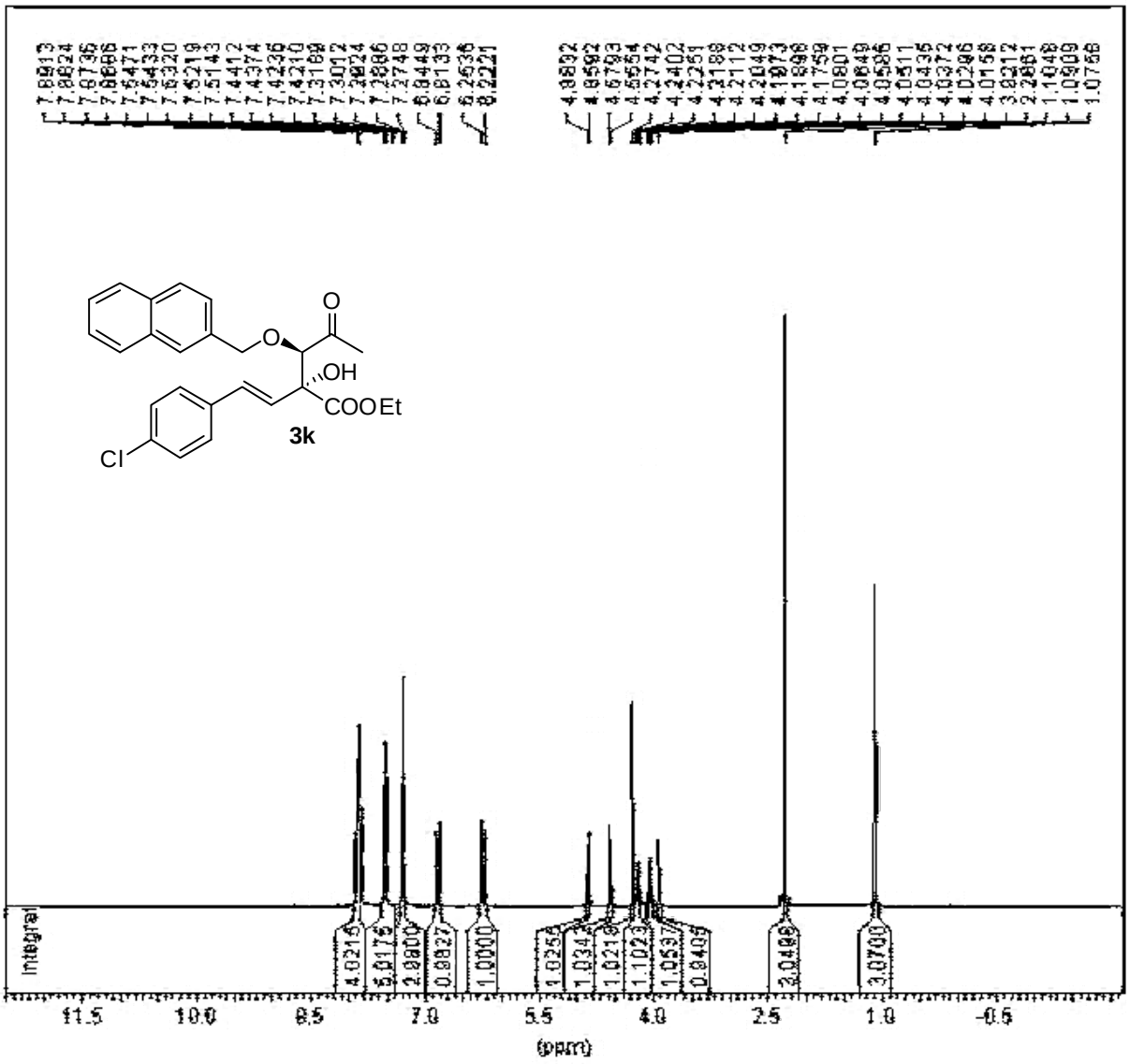
120
100
80
60
40
20
0
(ppm)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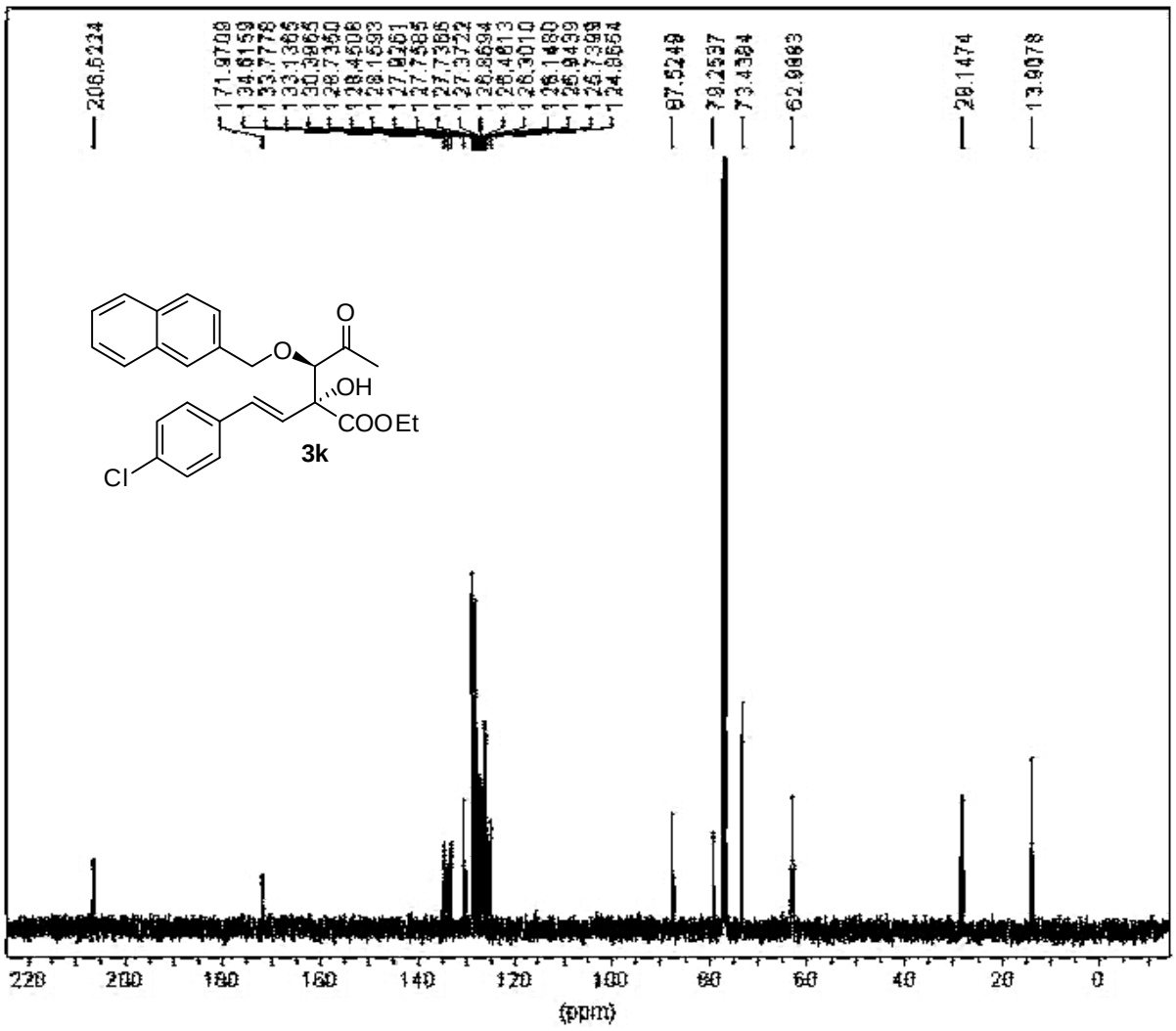
COOEt
Cl
3k
Integral
(ppm)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127.7585
127.7386
127.3722
126.8694
126.4613
126.3010
126.1480
125.9439
125.7399
124.8554
87.5248
79.2537
73.4384
62.9863
28.1474
13.9078
O
O
OH
COOEt
Cl
3k
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200
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120
100
80
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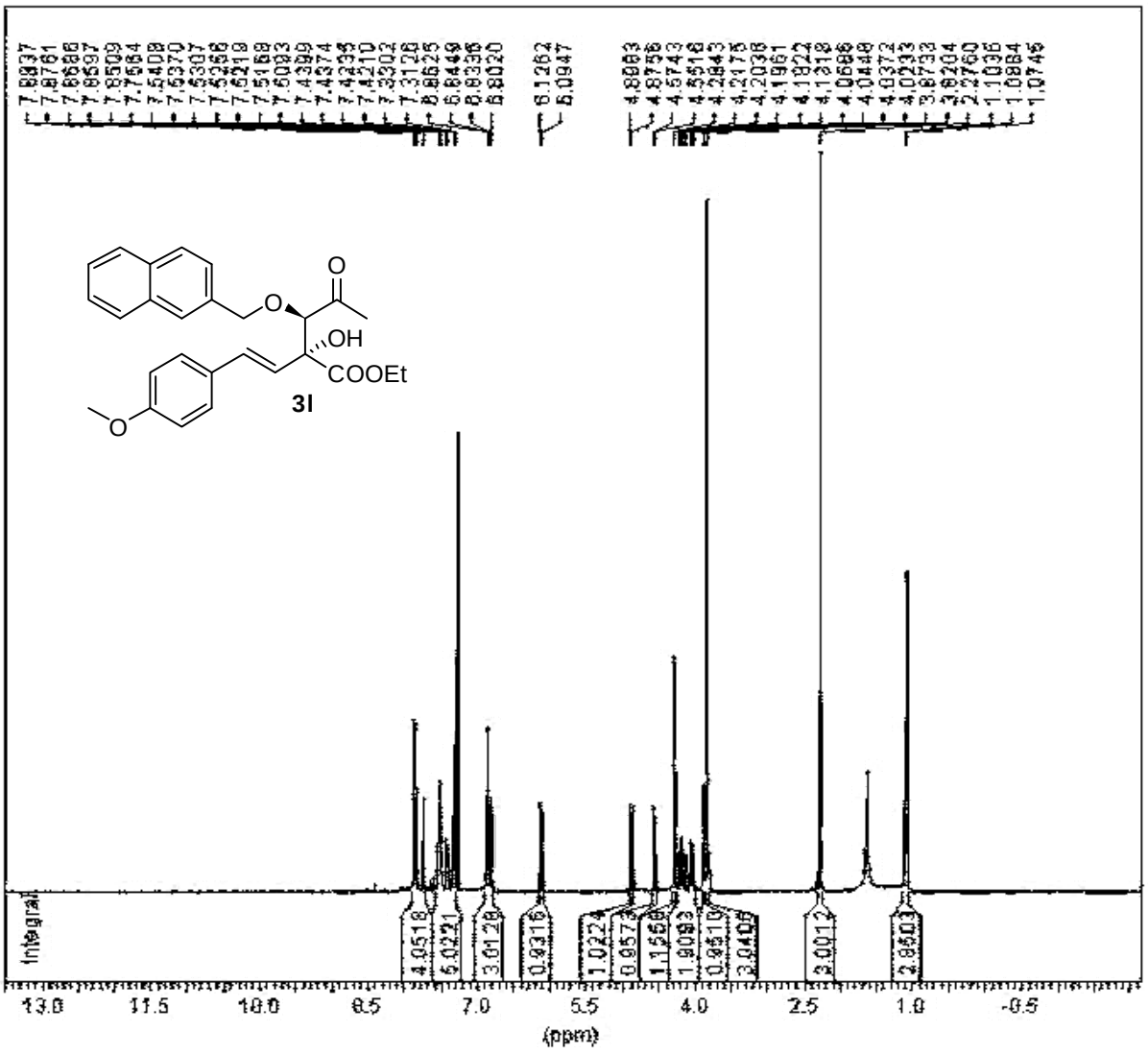
40
20
0
(ppm)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O
OH
COOEt
O
3l
Integral
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6.0221
3.0128
0.9316
1.0224
0.9573
1.1560
1.9093
0.9519
3.0409
2.0012
2.9503
13.0
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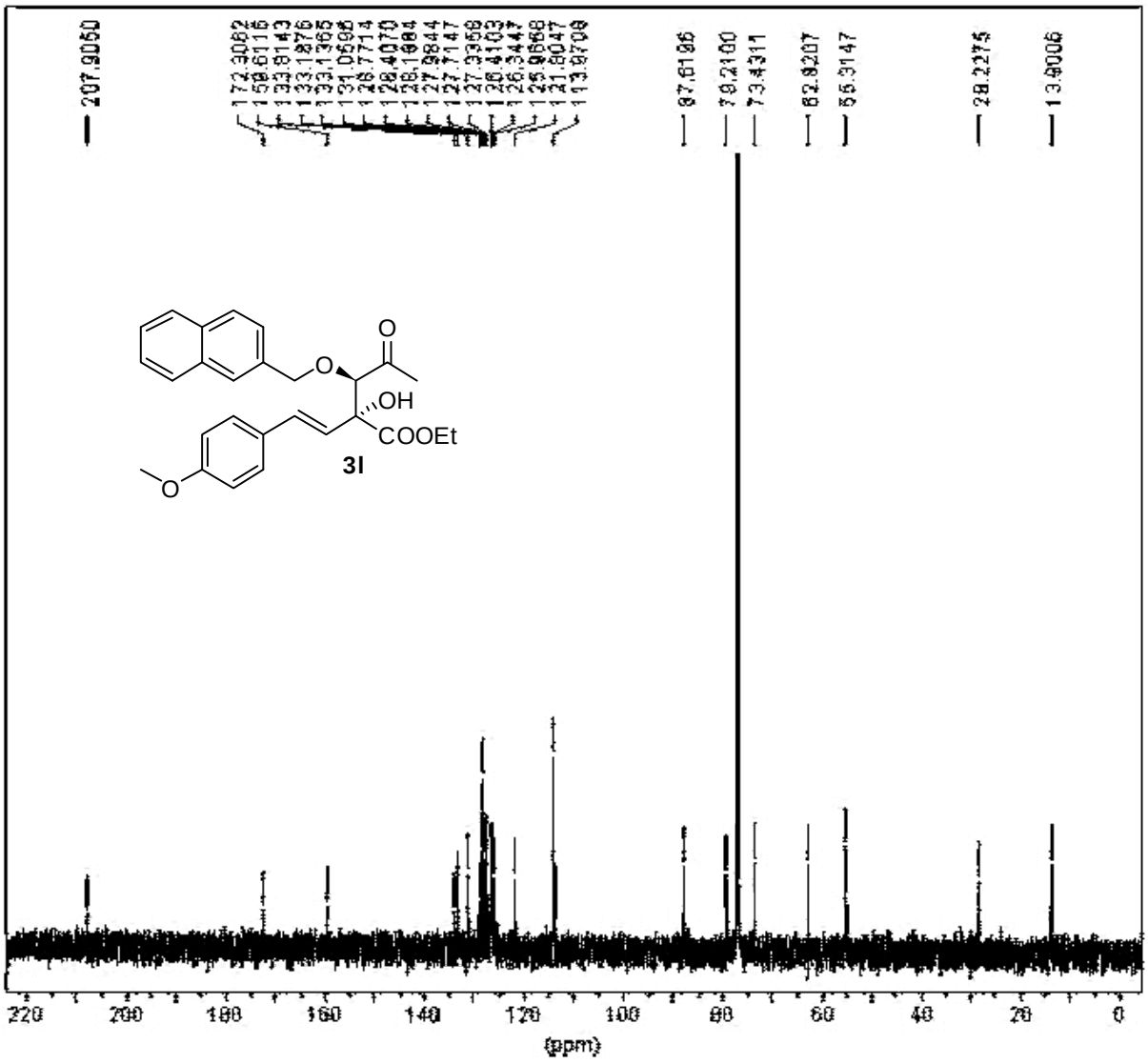
10.0
8.5
7.0
5.5
4.0
2.5
1.0
-0.5
(ppm)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159.6116
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128.4070
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127.9844
127.7147
127.3358
126.4103
126.3447
125.9568
121.8047
113.9709
87.6186
79.2100
73.4311
62.9207
55.3147
28.2275
13.9006
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O
OH
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O
3l
220
200
180
160
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120
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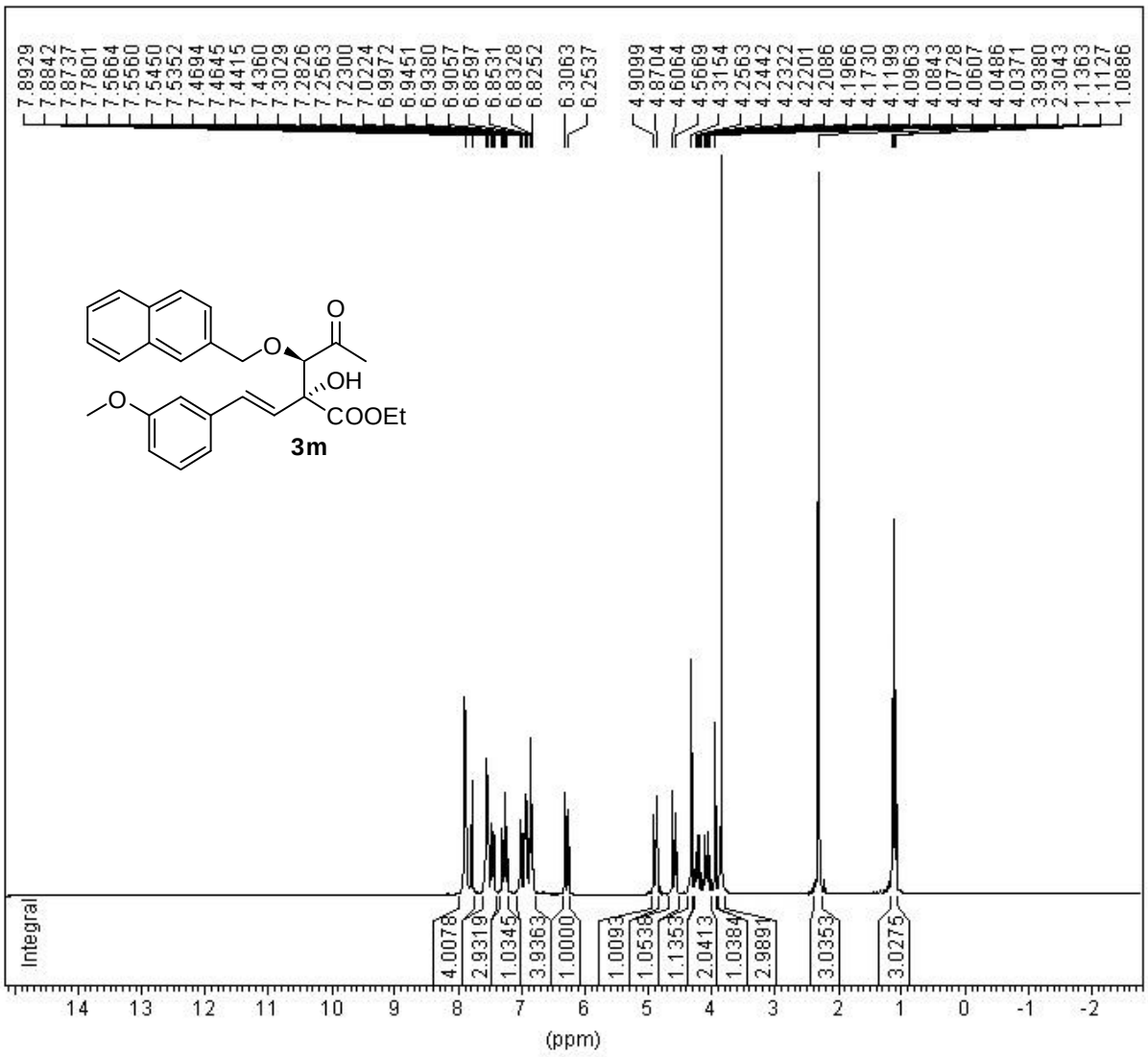
0
(ppm)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7.8737
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7.5664
7.5560
7.5450
7.5352
7.4694
7.4645
7.4415
7.4360
7.3029
7.2826
7.2563
7.2300
7.0224
6.9972
6.9451
6.9380
6.9057
6.8597
6.8531
6.8328
6.8252
6.3063
6.2537
4.9099
4.8704
4.6064
4.5669
4.3154
4.2563
4.2442
4.2322
4.2201
4.2086
4.1966
4.1730
4.1199
4.0963
4.0843
4.0728
4.0607
4.0486
4.0371
3.9380
2.3043
1.1363
1.1127
1.0886
O
O
OH
COOEt
O
3m
Integral
4.0078
2.9319
1.0345
3.9363
1.0000
1.0093
1.0538
1.1353
2.0413
1.0384
2.9891
3.0353
3.0275
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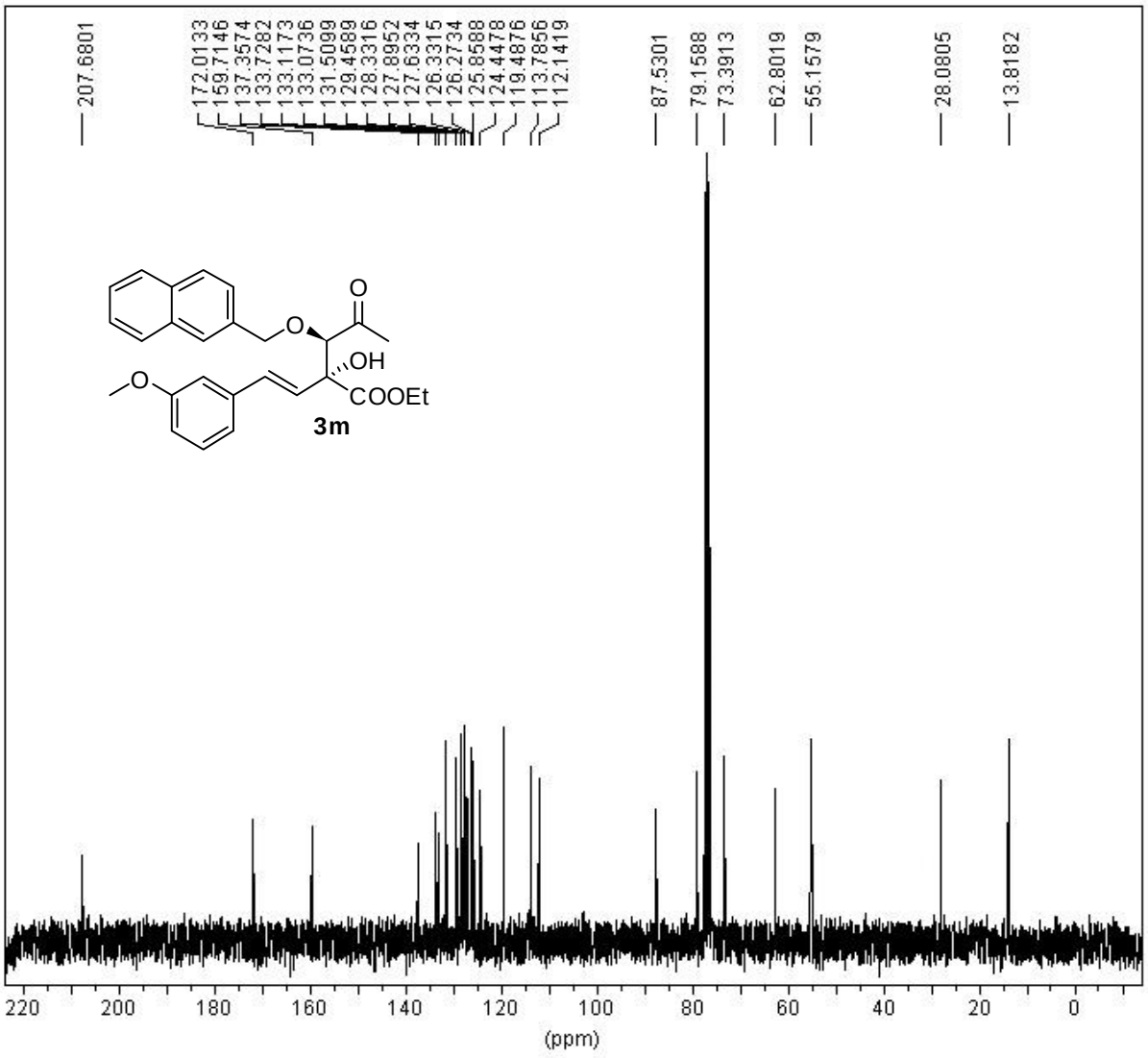
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
(ppm)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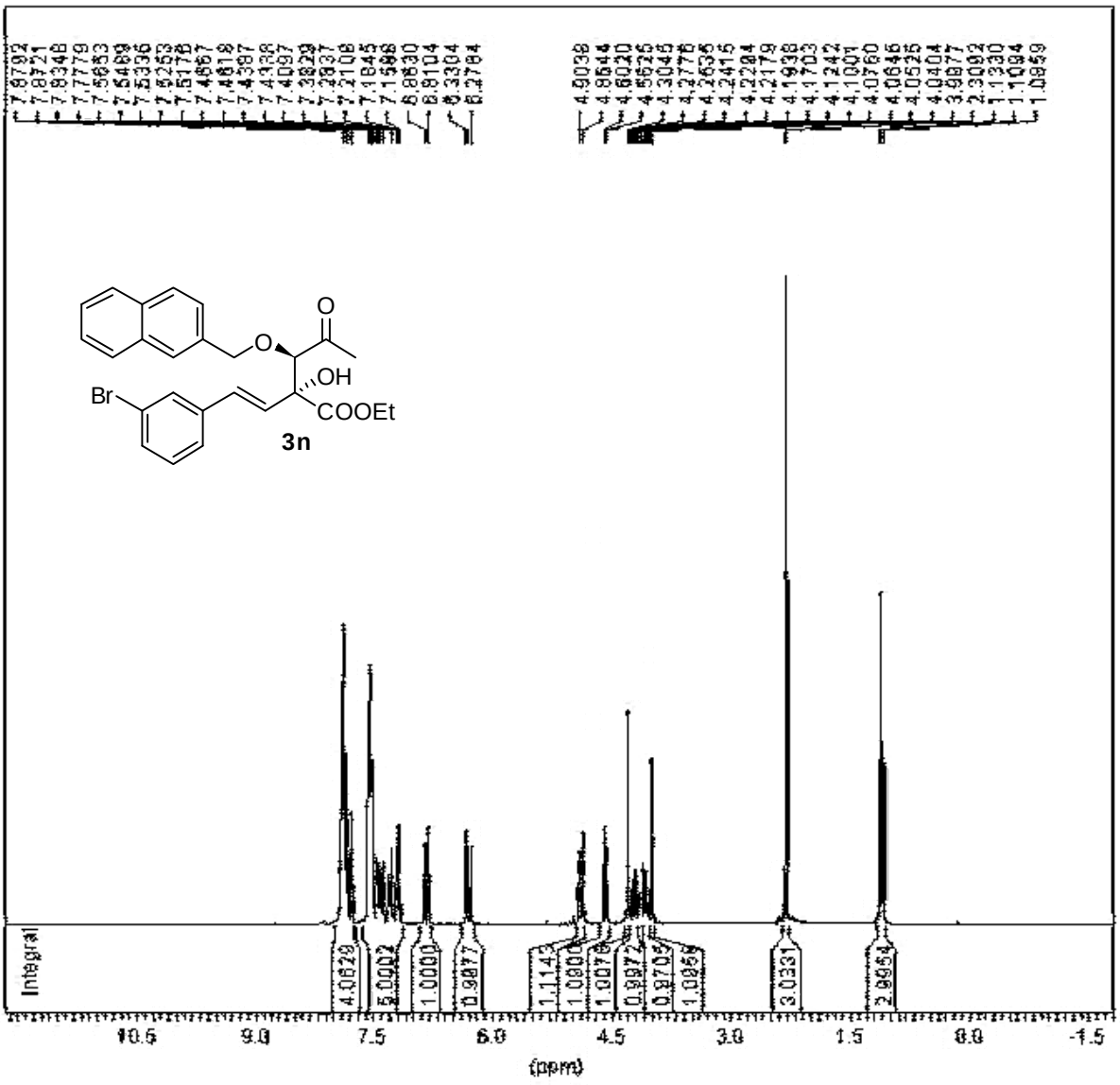
133.7282
133.1173
133.0736
131.5099
129.4589
128.3316
127.8952
127.6334
126.3315
126.2734
125.8588
124.4478
119.4876
113.7856
112.1419
87.5301
79.1588
73.3913
62.8019
55.1579
28.0805
13.8182
O
O
OH
O
COOEt
3m
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)


O
OH
Br
COOEt
3n
Integral
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5.0002
1.0000
0.9977
1.1143
1.0800
1.0078
0.9972
0.9705
1.0859
3.0331
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9.0
7.5
6.0
4.5
3.0
1.5
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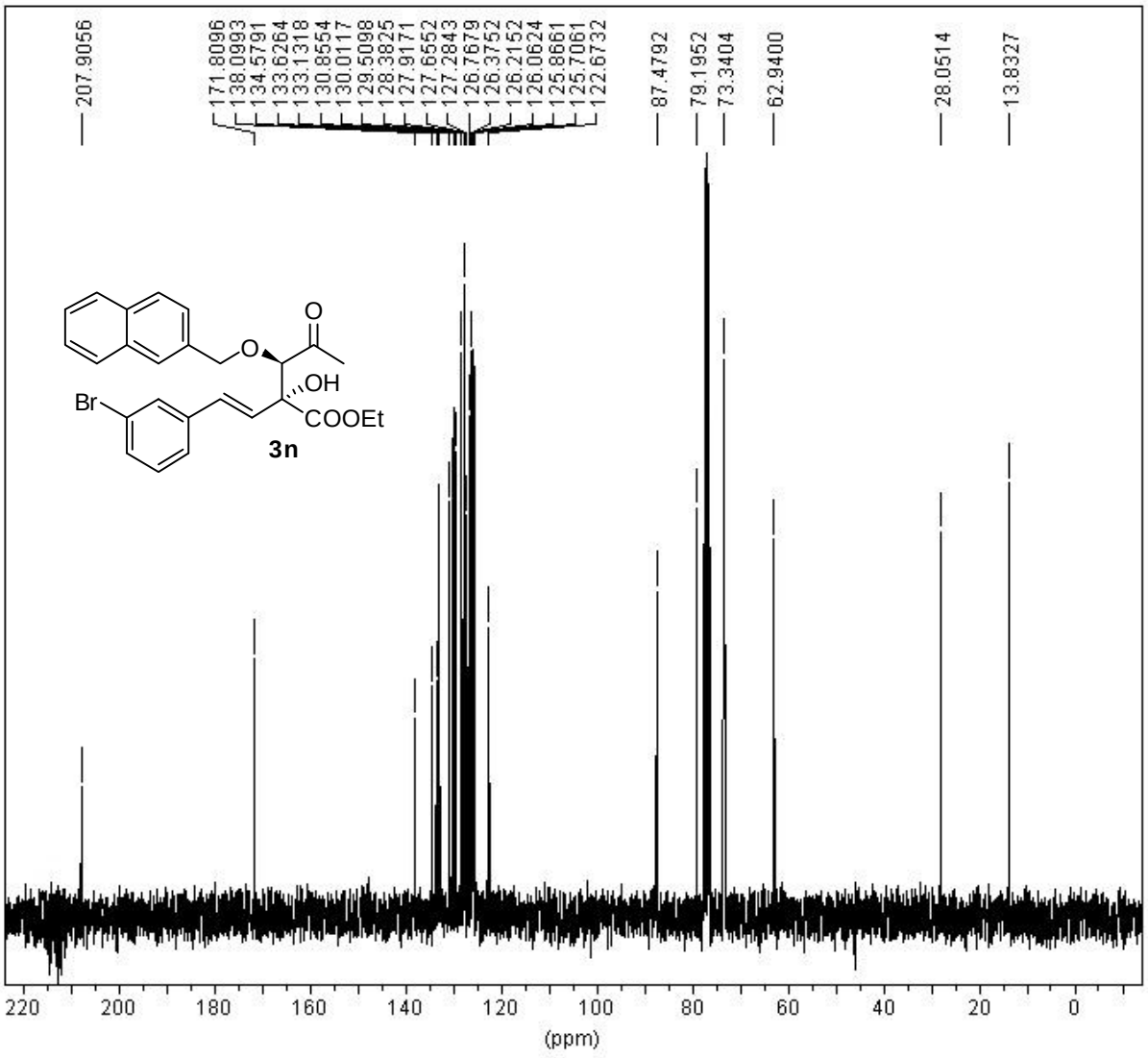
-1.5
(ppm)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130.8554
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129.5098
128.3825
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127.6552
127.2843
126.7679
126.3752
126.2152
126.0624
125.8661
125.7061
122.6732
87.4792
79.1952
73.3404
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13.8327
O
O
OH
COOEt
Br
3n
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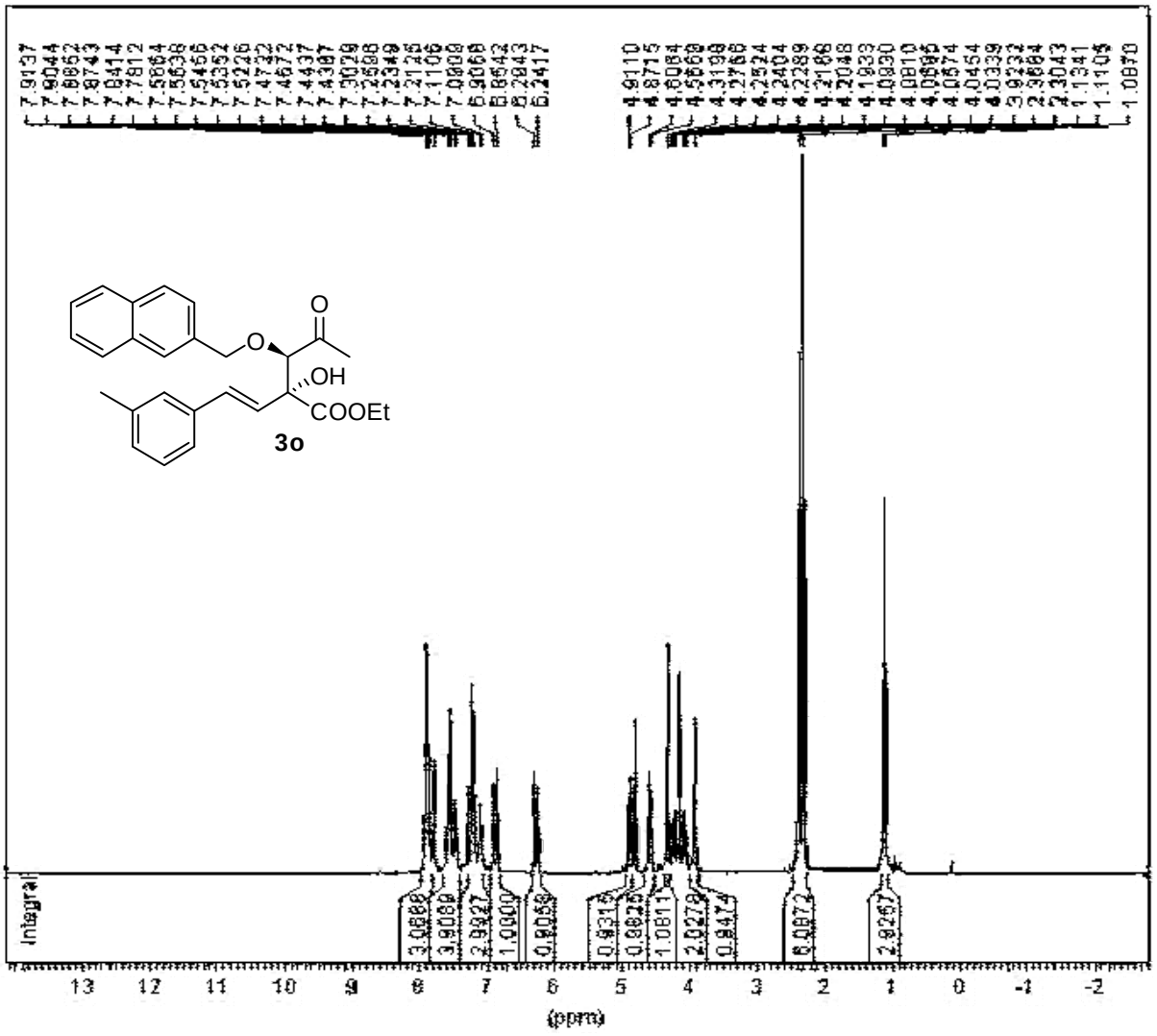
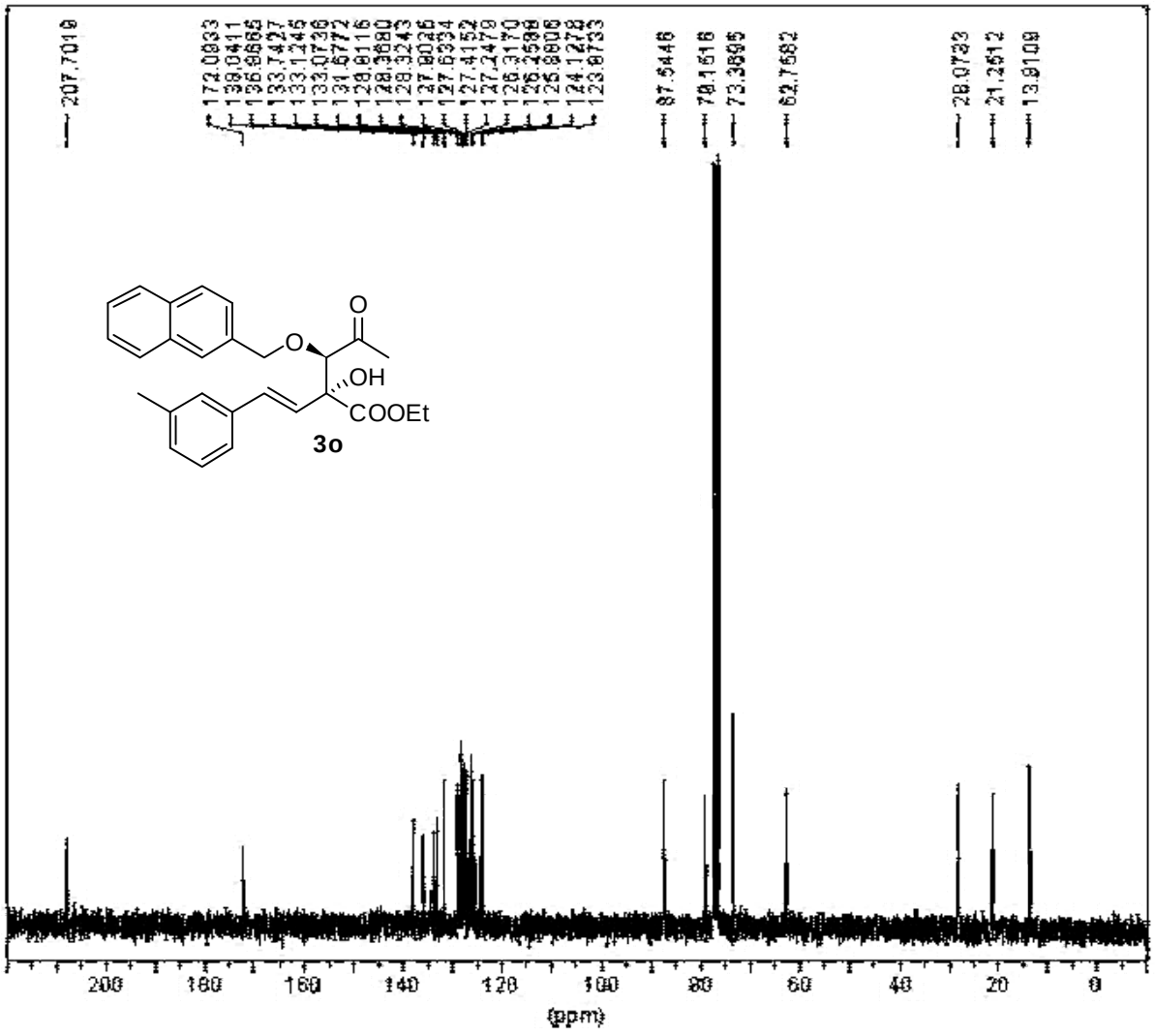
200
180
160
140
120
100
80
60
40
20
0
(ppm)


O
O
OH
COOEt
3o
Integral
(ppm)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132.0726
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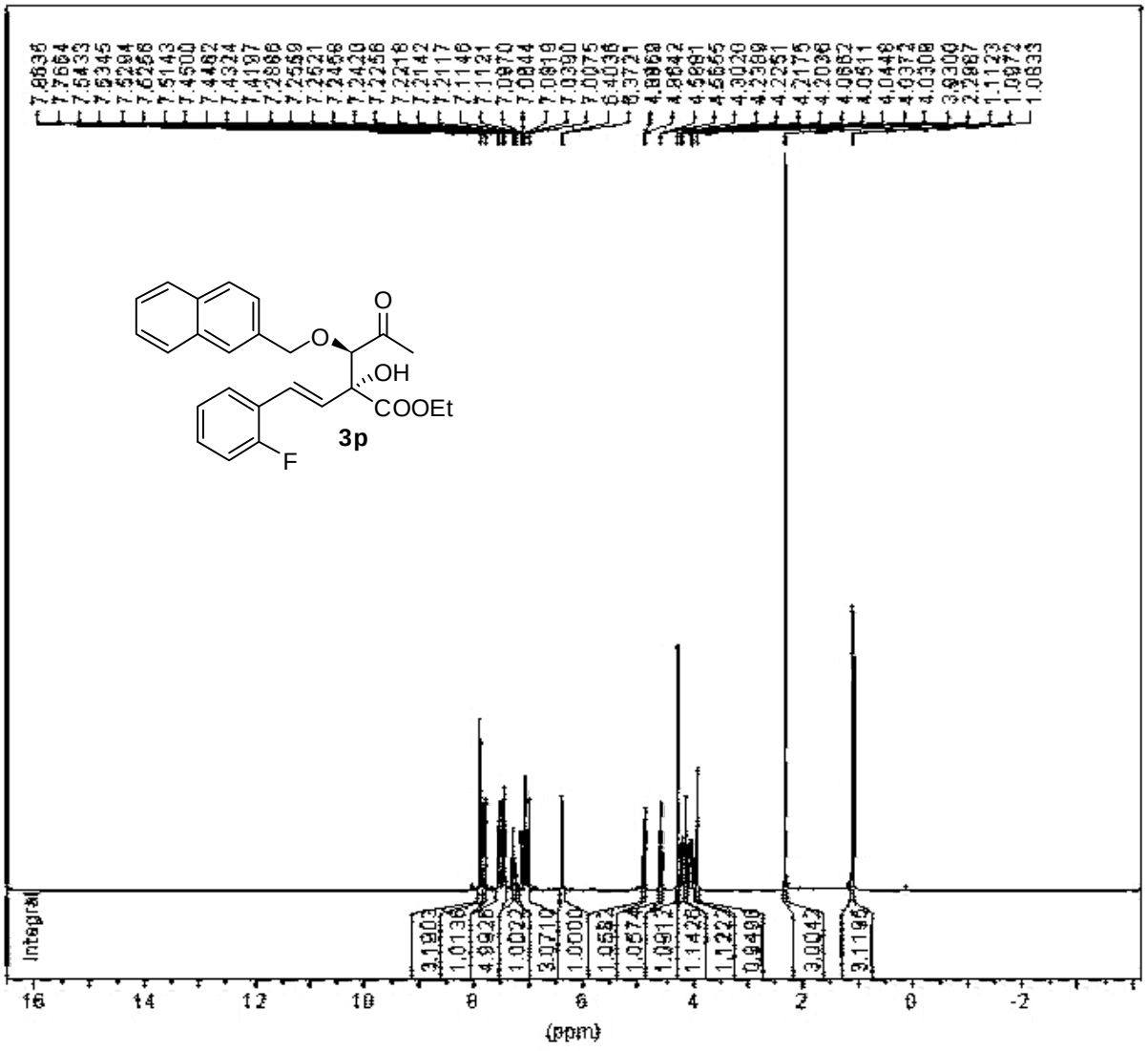
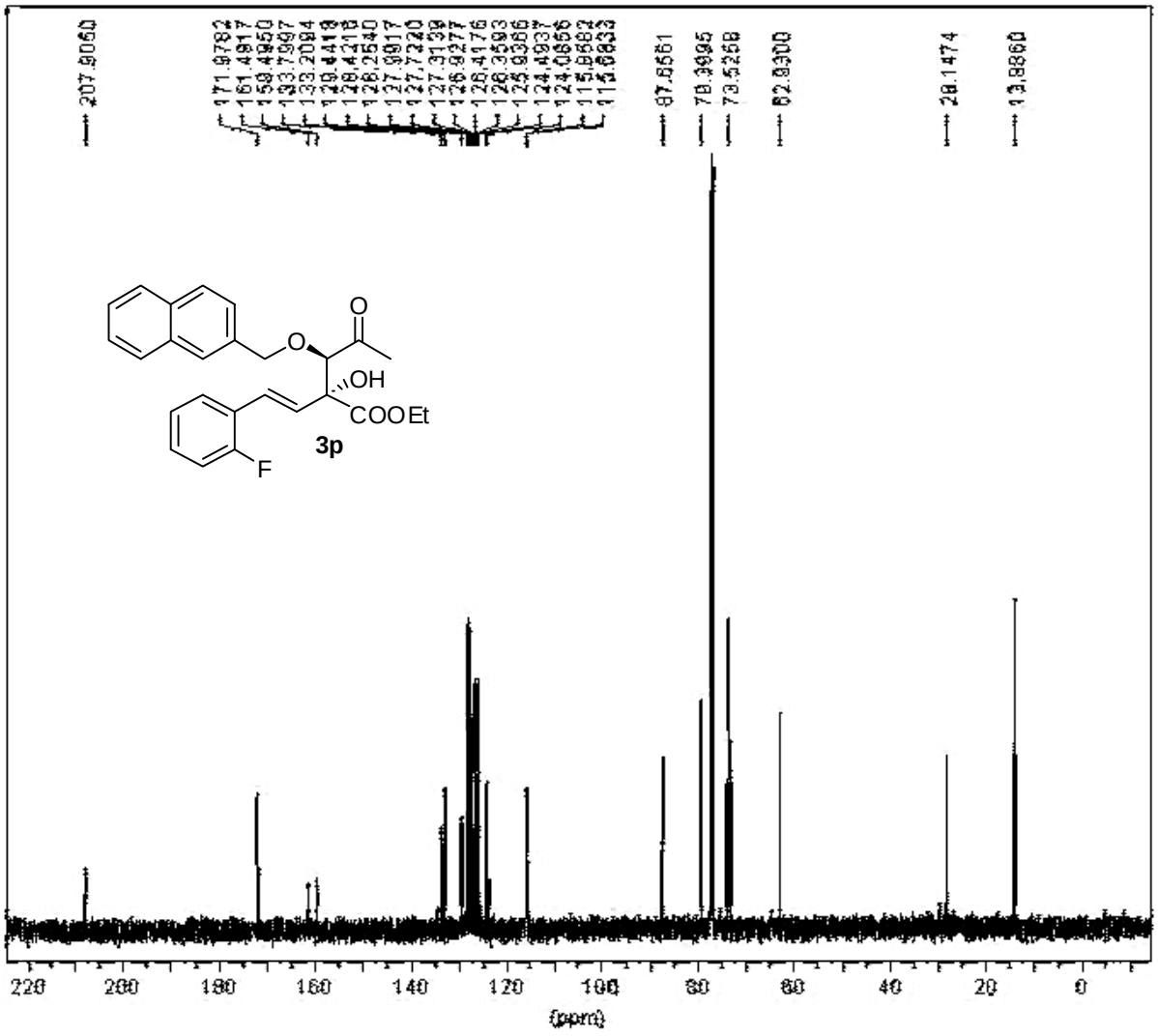
128.3243
127.8026
127.6334
127.4152
127.2478
126.3170
125.4598
125.8806
124.1278
123.8733
87.5446
78.1618
73.3895
62.7582
28.0723
21.2612
13.9109
O
O
OH
COOEt
3o
200
180
160
140
120
100
80
60
40
20
0
(ppm)


O
O
OH
COOEt
F
3p
Integral
(ppm)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127.3139
126.9277
126.4176
126.3593
125.9386
124.4937
124.0856
115.8682
115.6833
97.8551
78.3995
73.5258
62.8300
28.1474
13.8860
O
O
OH
COOEt
F
3p
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200
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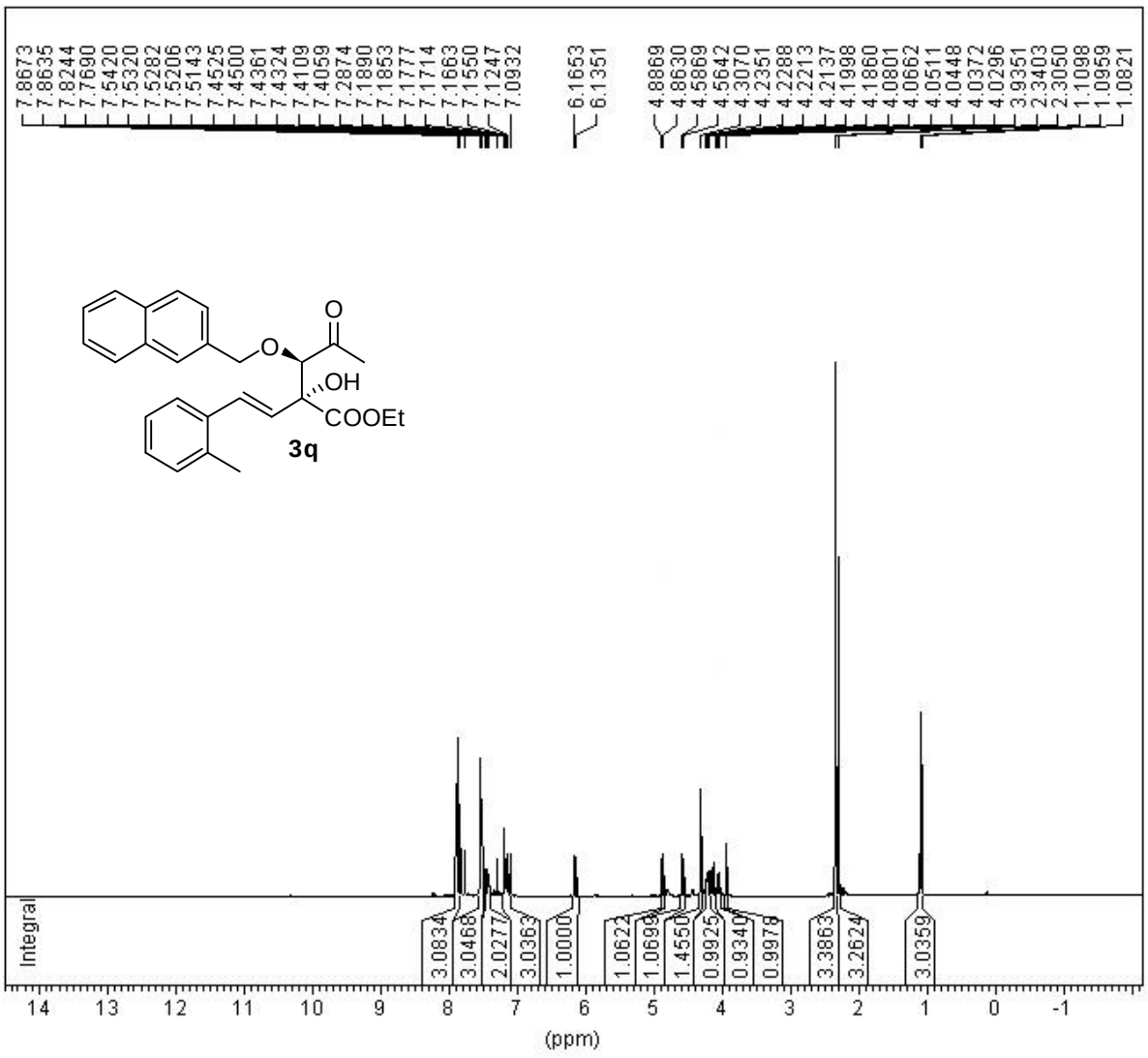
160
140
120
100
80
60
40
20
0
(ppm)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7.5143
7.4525
7.4500
7.4361
7.4324
7.4109
7.4059
7.2874
7.1890
7.1853
7.1777
7.1714
7.1663
7.1550
7.1247
7.0932
6.1653
6.1351
4.8869
4.8630
4.5869
4.5642
4.3070
4.2351
4.2288
4.2213
4.2137
4.1998
4.1860
4.0801
4.0662
4.0511
4.0448
4.0372
4.0296
3.9351
2.3403
2.3050
1.1098
1.0959
1.0821
O
O
OH
COOEt
3q
Integral
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3.0369
1.0000
1.0622
1.0698
1.4550
0.9925
0.9340
0.9979
3.3863
3.2624
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12
11
10
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8
7
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5
4
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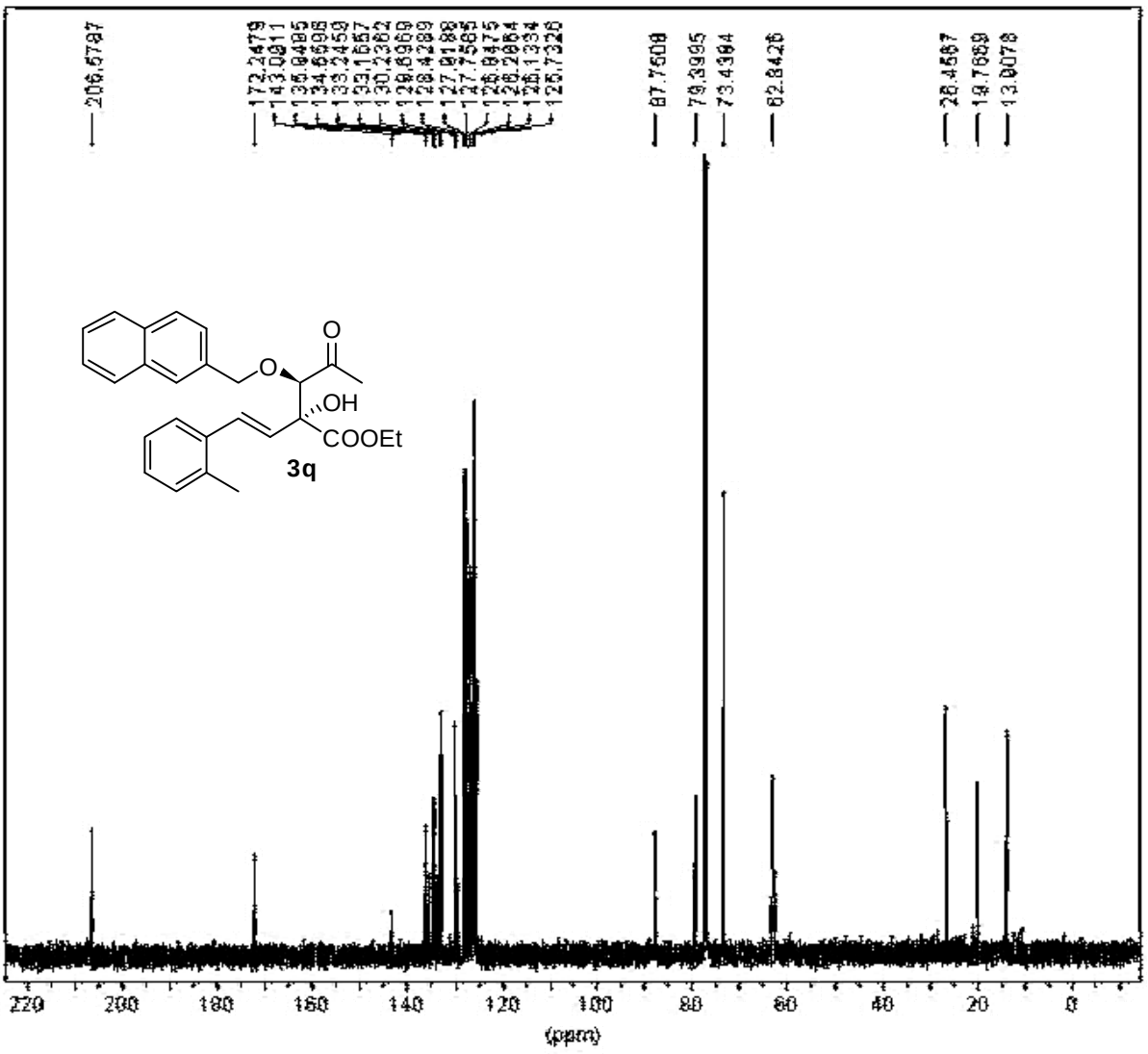
(ppm)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126.0475
126.2064
126.1334
125.7326
87.7509
79.3995
73.4394
62.8426
28.4587
19.7659
13.9078
O
O
OH
COOEt
3q
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200
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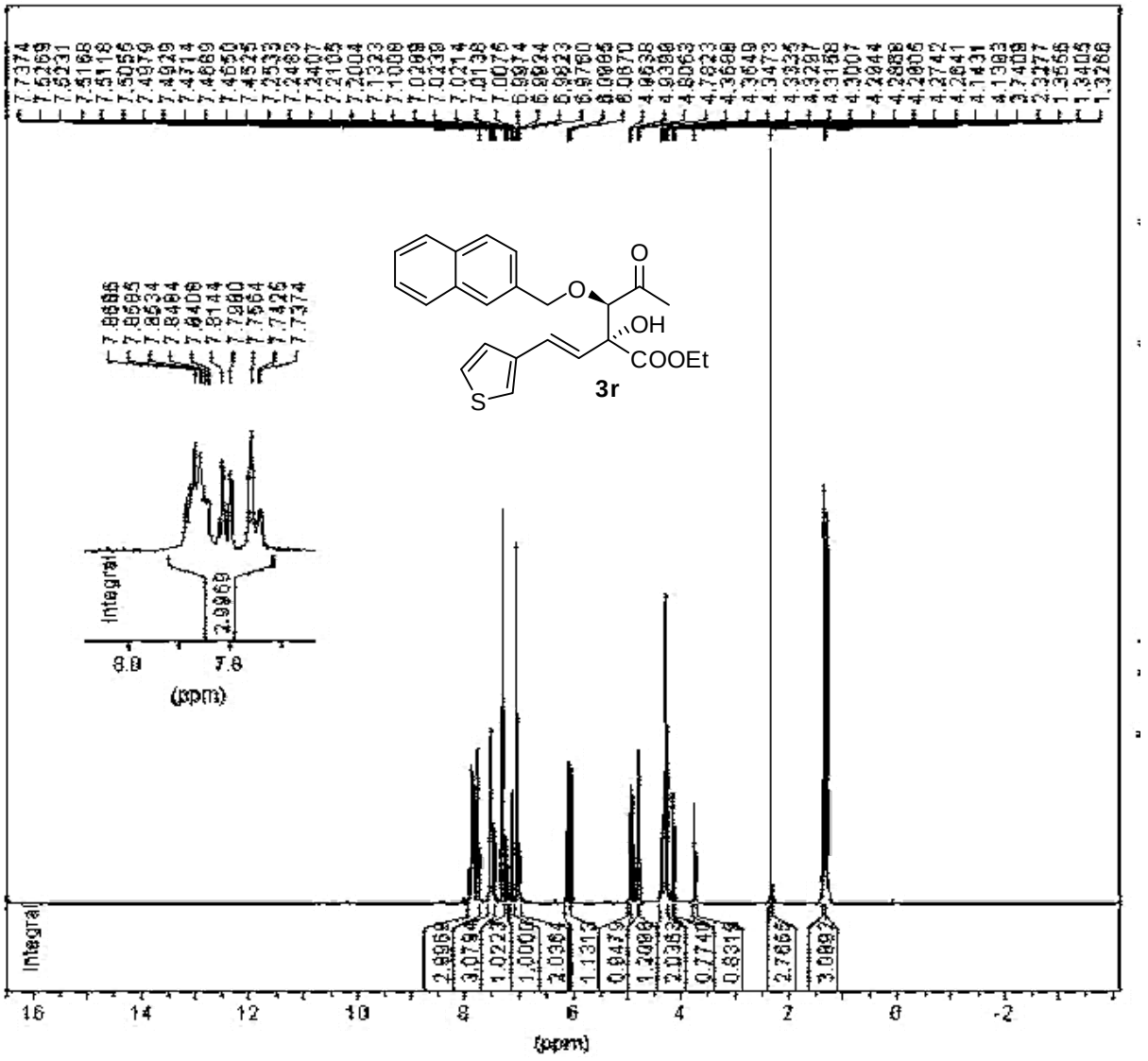
160
140
120
100
80
60
40
20
0
(ppm)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O
OH
COOEt
S
3r
Integral
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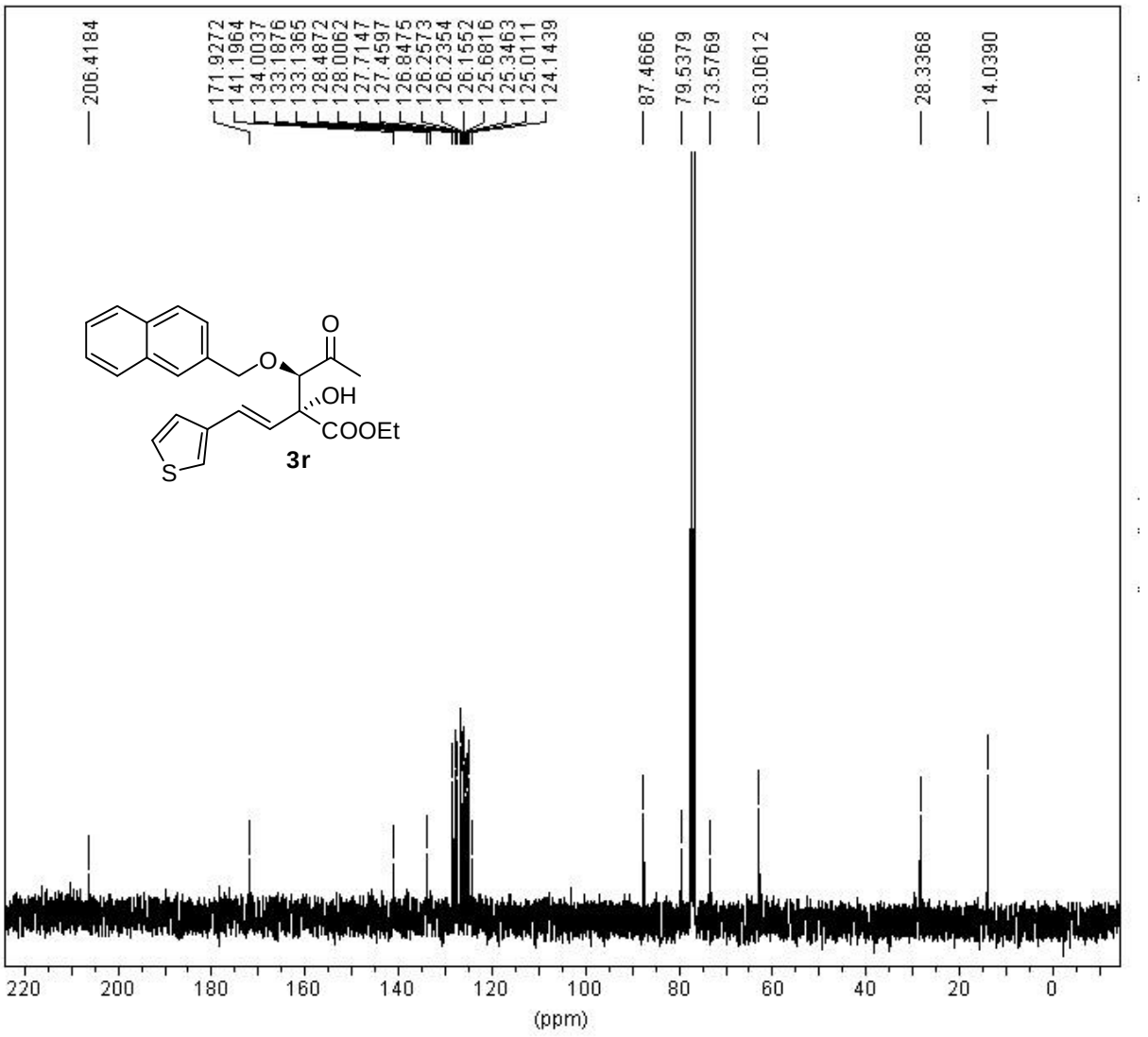
7.8
(ppm)
Integral
16
14
12
10
8
6
4
2
0
-2
(ppm)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125.6816
125.3463
125.0111
124.1439
87.4666
79.5379
73.5769
63.0612
28.3368
14.0390
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O
OH
COOEt
S
3r
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200
180
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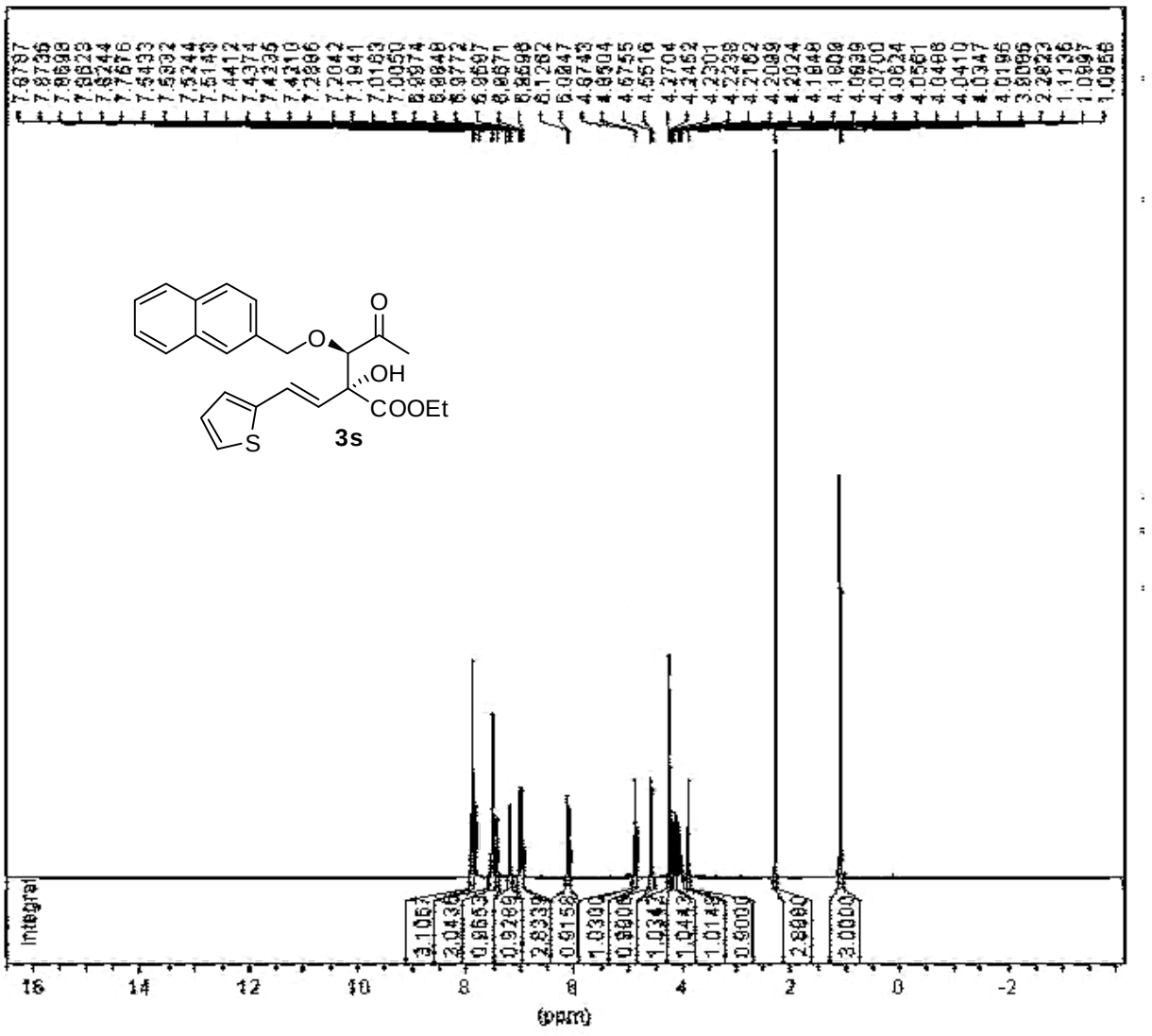
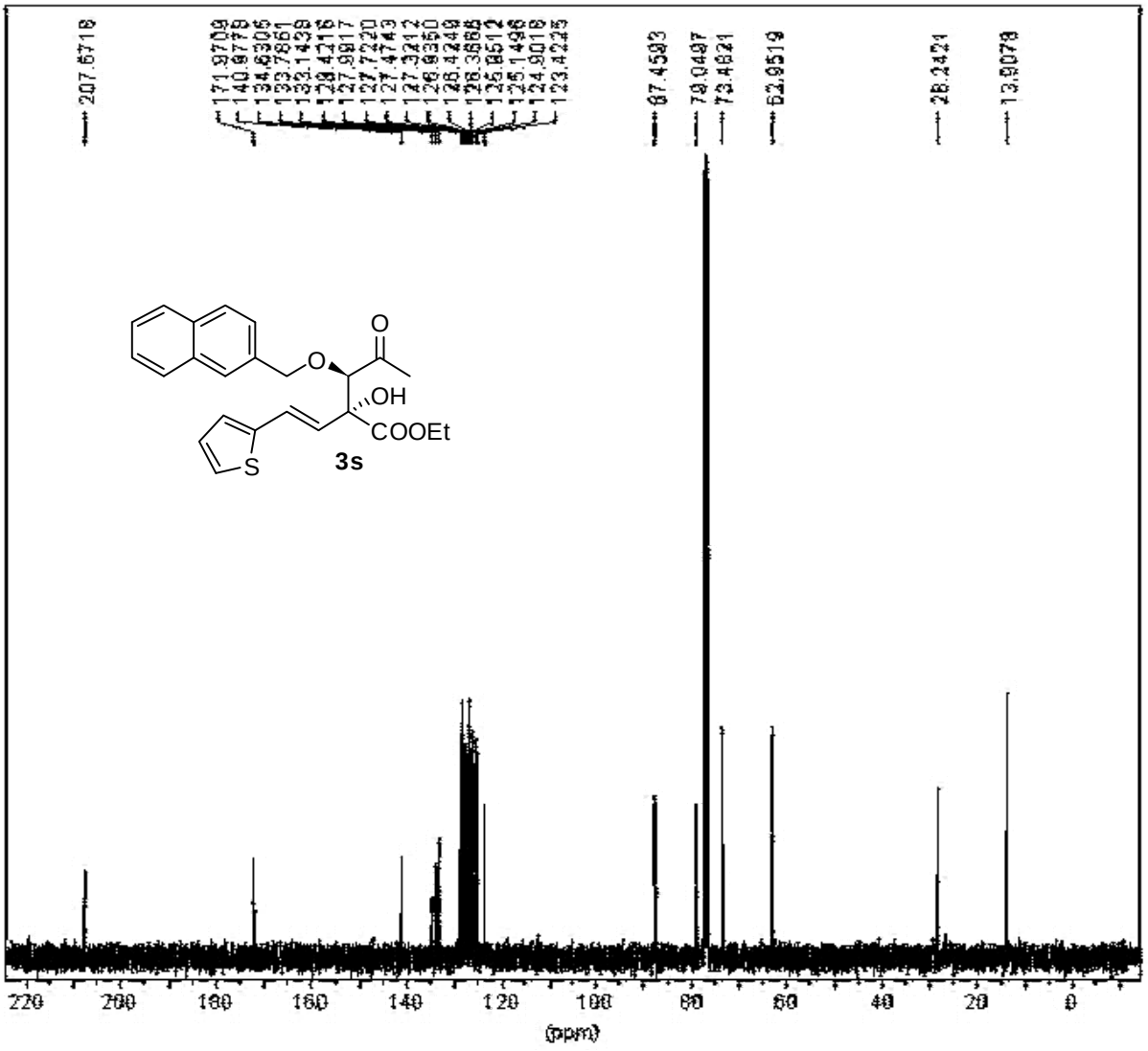
120
100
80
60
40
20
0
(ppm)


O
O
OH
COOEt
S
3s
Integral
(ppm)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126.4249
126.3888
125.8512
125.1498
124.9018
123.4225
67.4593
78.0487
73.4621
62.9519
28.2421
13.9079
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O
OH
COOEt
S
3s
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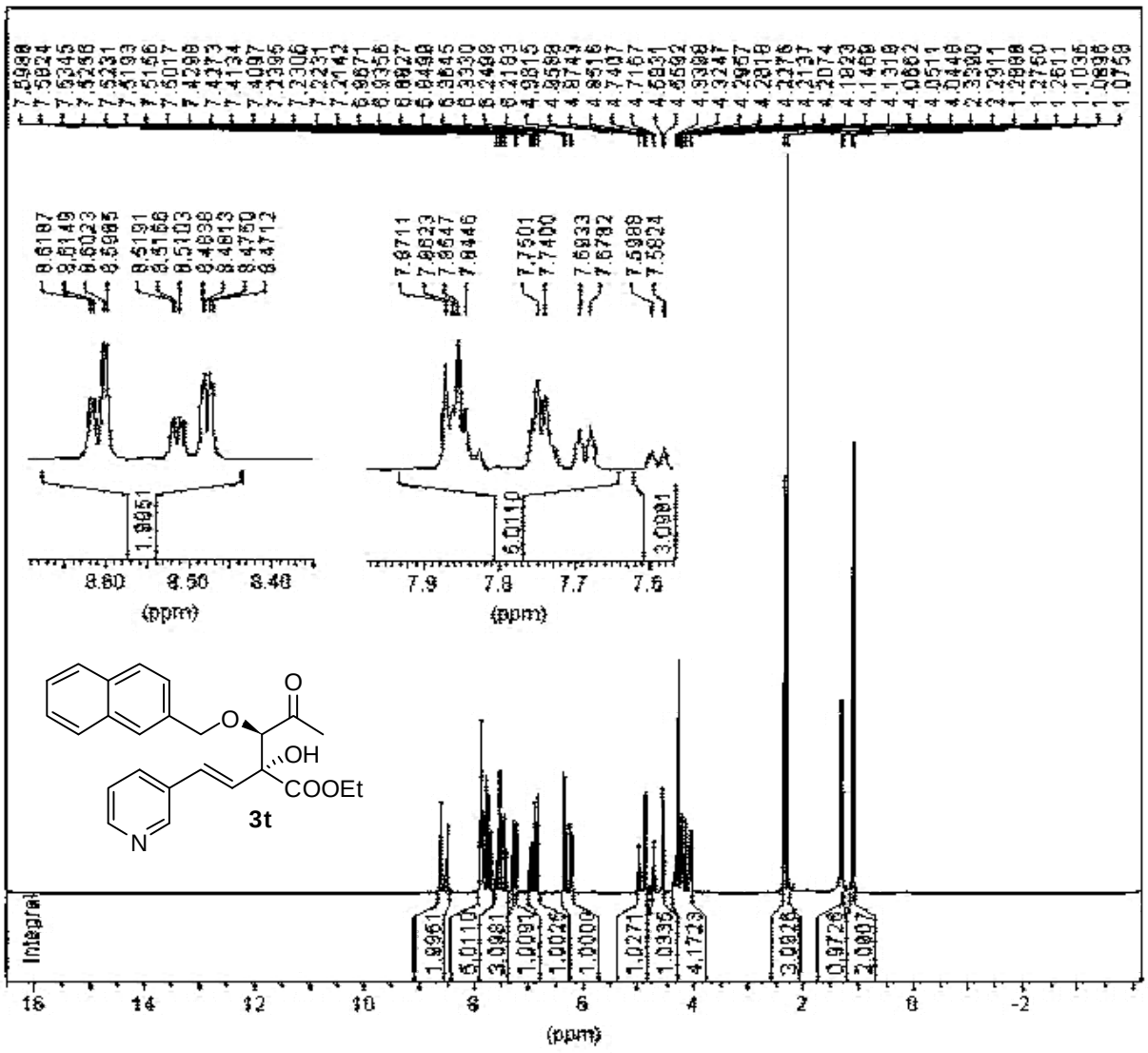
200
180
160
140
120
100
80
60
40
20
0
(ppm)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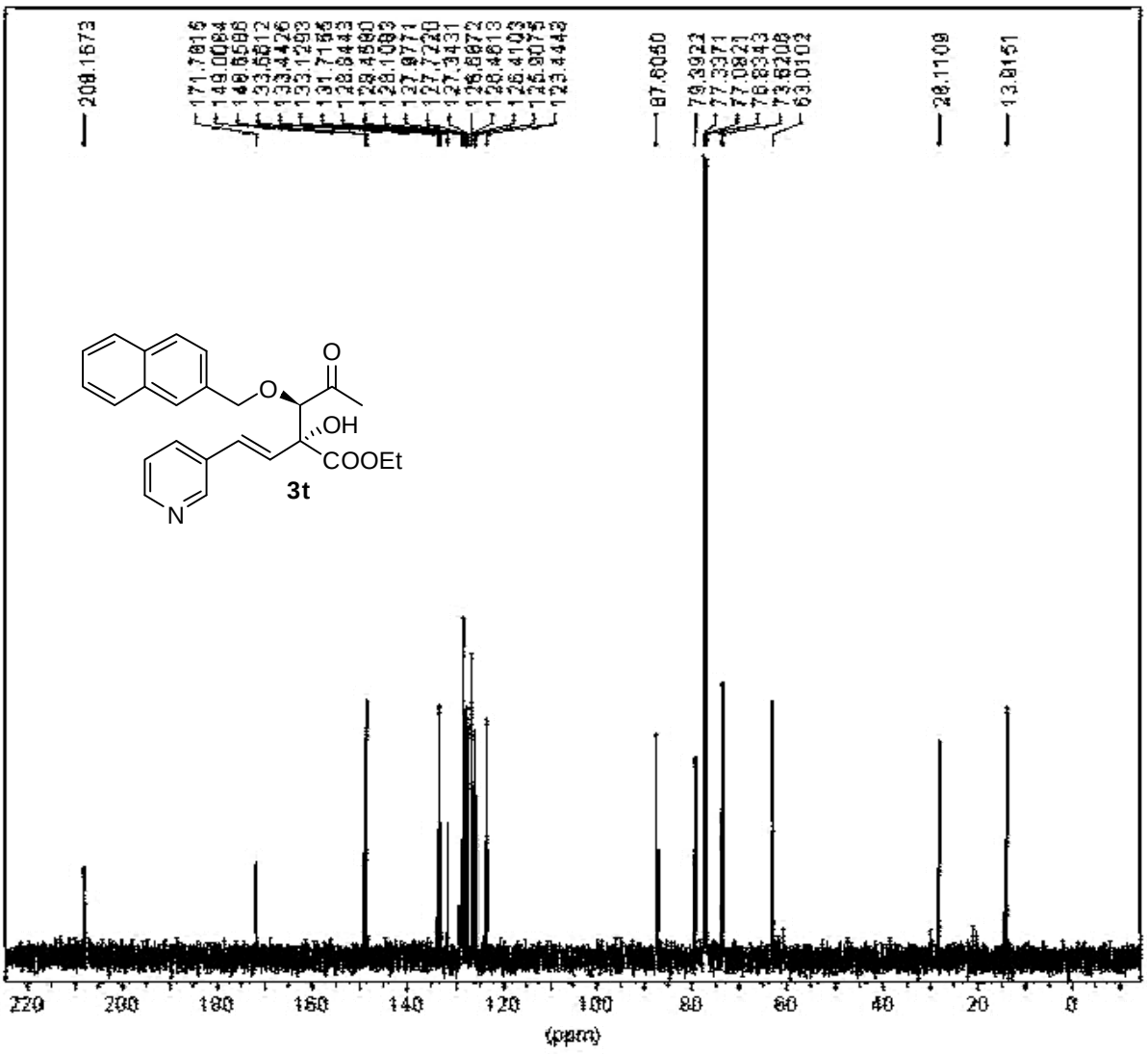
OH
COOEt
N
3t
(ppm)
Integral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133.1293
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128.1083
127.9771
127.7220
127.3431
126.8672
126.4613
126.4103
125.9075
123.4448
87.6050
79.3922
77.3371
77.0821
76.8343
73.6208
63.0102
28.1109
13.9151
O
O
OH
COOEt
N
3t
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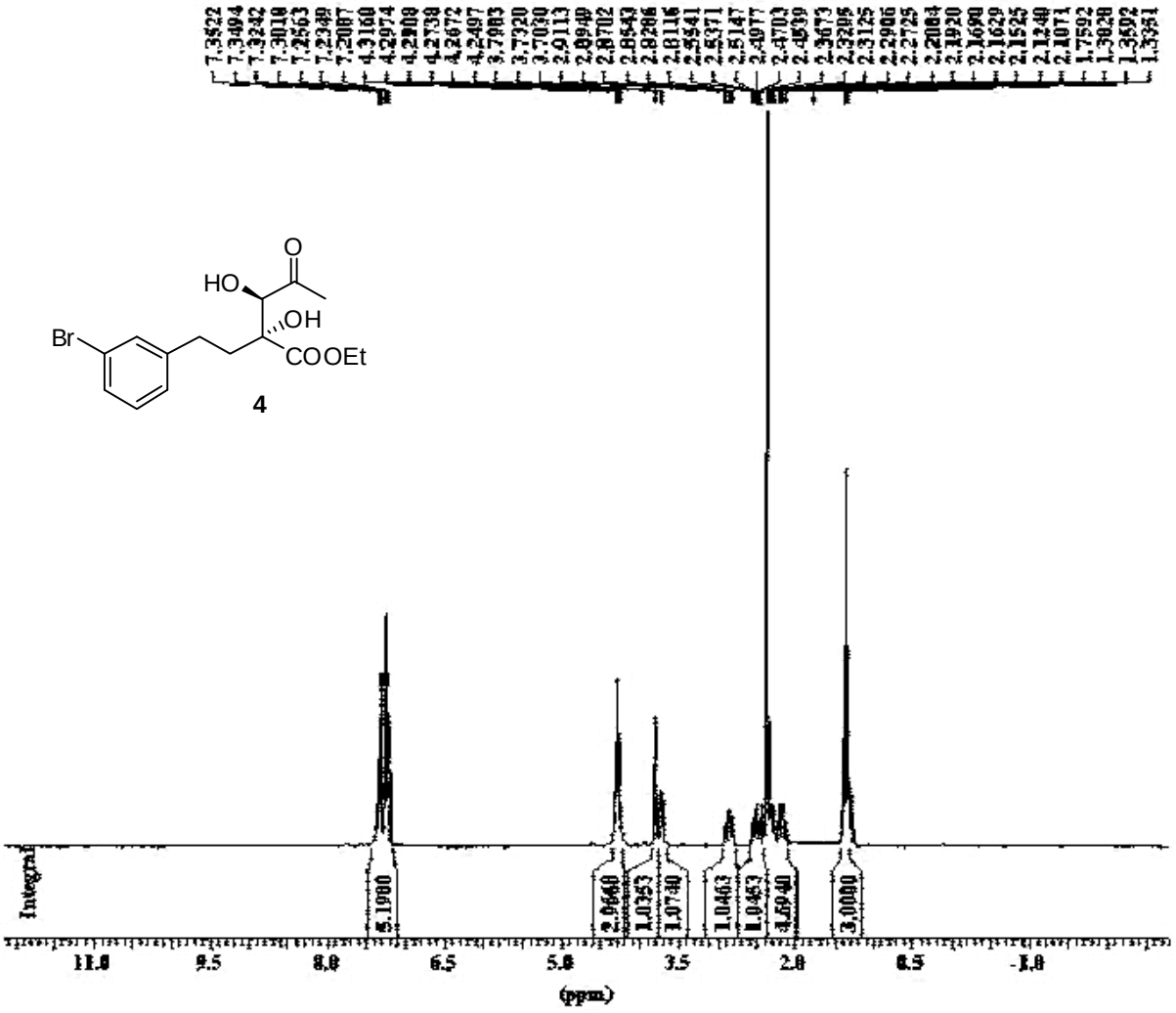
200
180
160
140
120
100
80
60
40
20
0
(ppm)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OH
COOEt
Br
4
Integral
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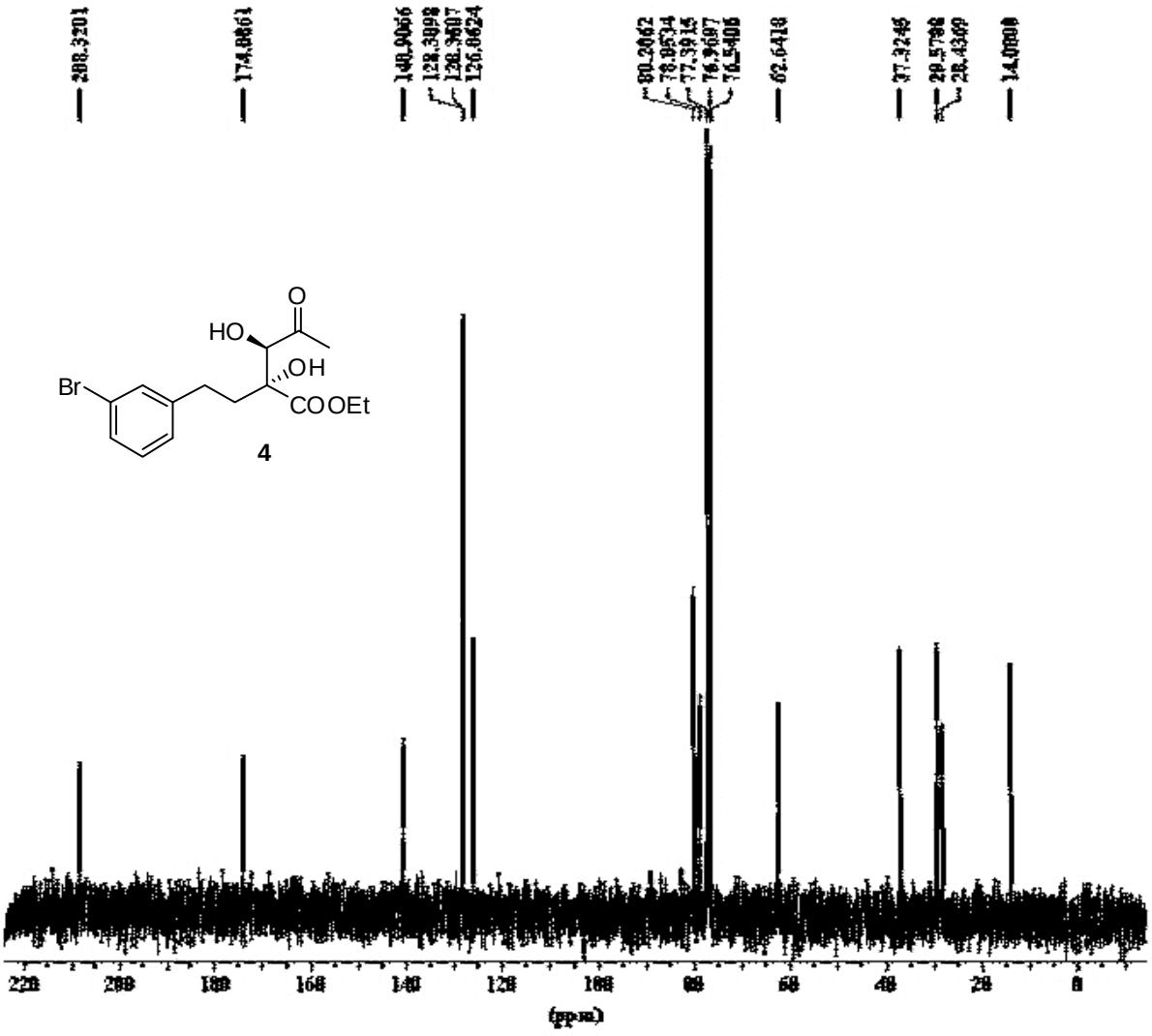
1.0353
1.0740
1.0463
1.0453
4.6940
3.0000
11.0
9.5
8.0
6.5
5.0
3.5
2.0
0.5
-1.0
(ppm)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80.2062
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77.3915
76.9607
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37.3246
29.5788
28.4369
14.0808
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OH
Br
COOEt
4
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200
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140
120
100
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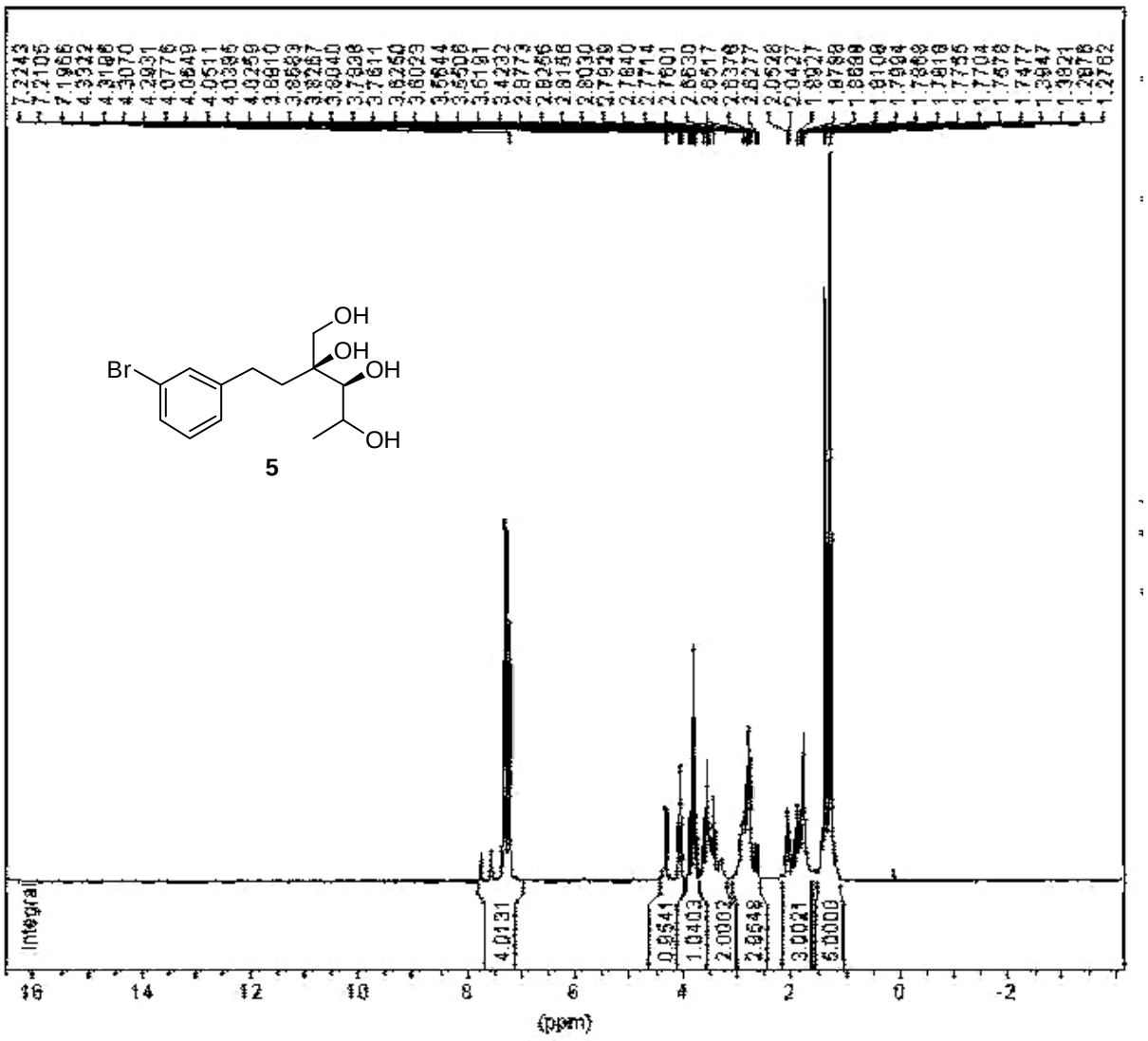
60
40
20
0
(ppm)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OH
OH
Br
OH
5
Integral
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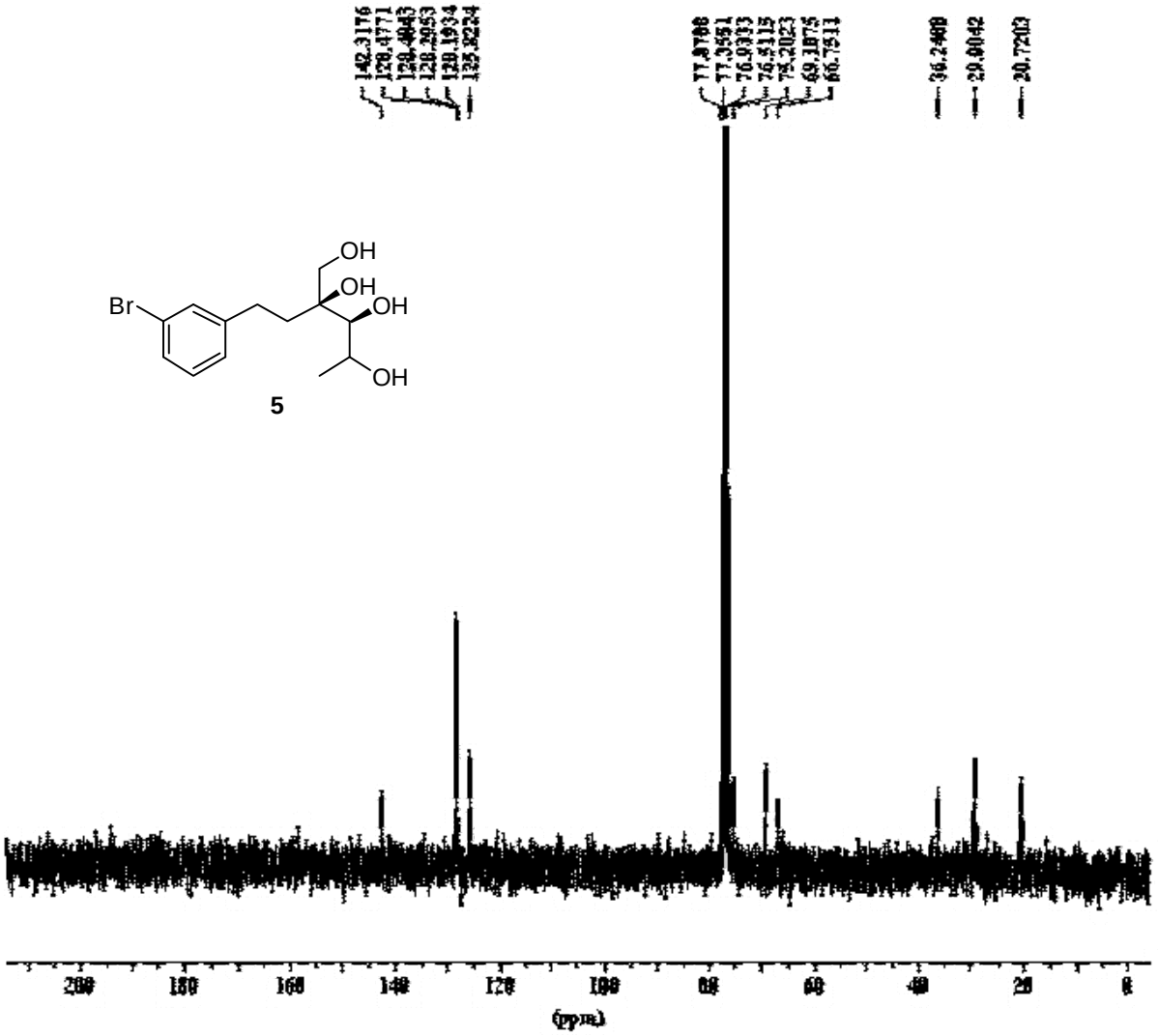
3.0021
6.0000
16
14
12
10
8
6
4
2
0
-2
(ppm)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128.1934
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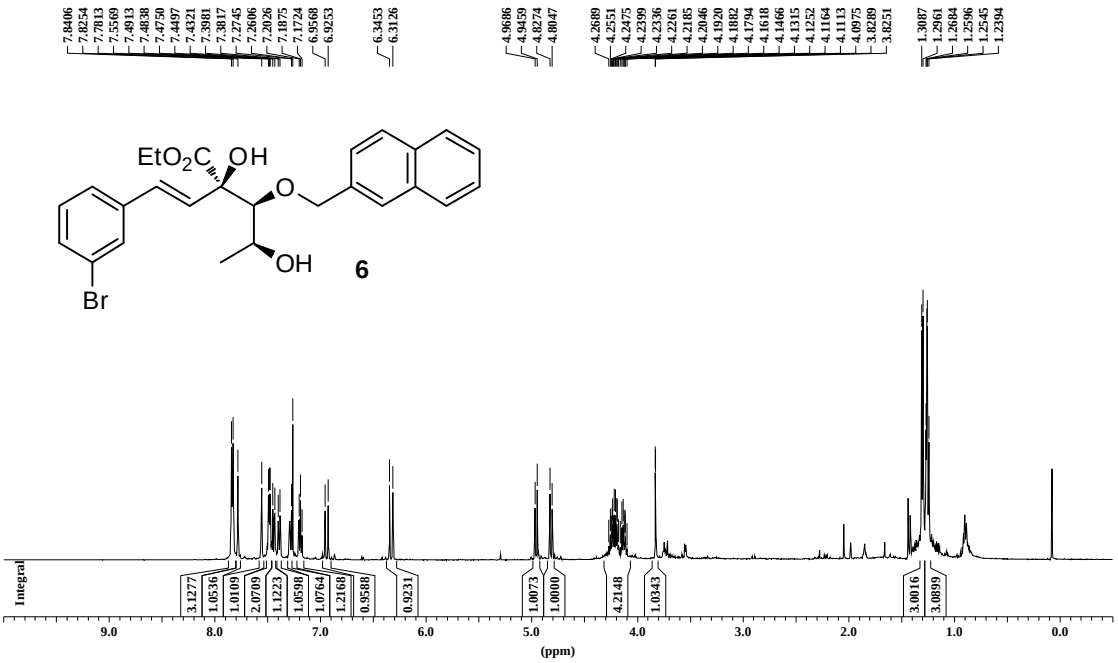
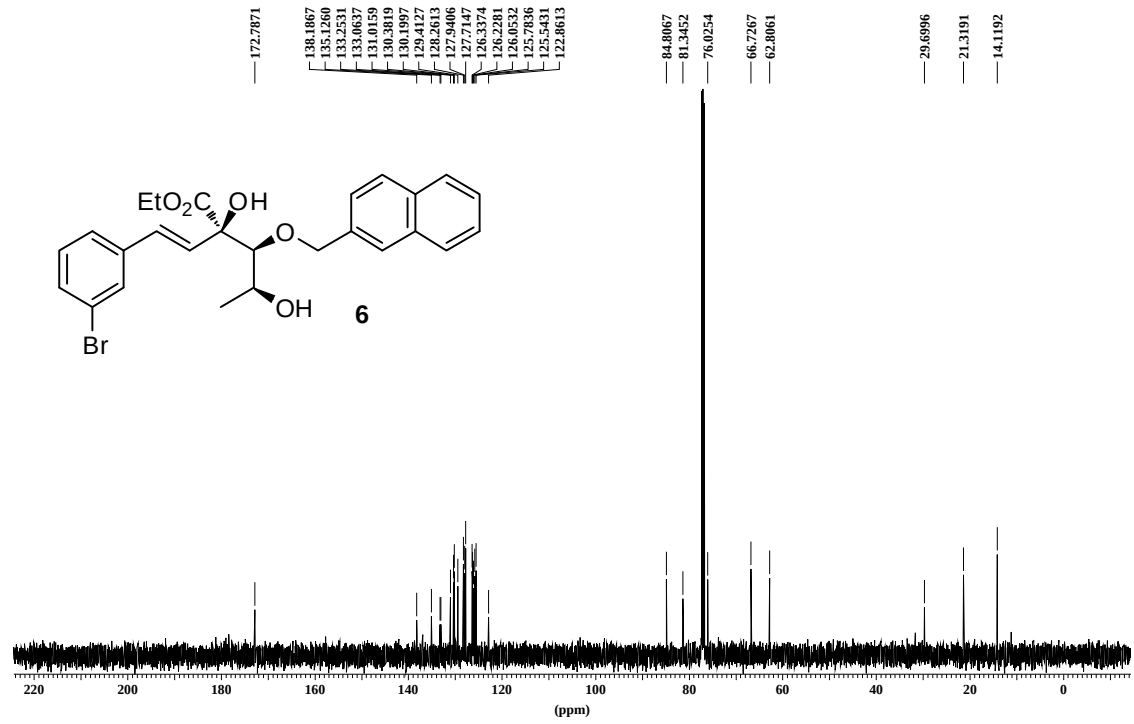
77.3551
76.0333
76.5115
75.2023
69.1875
60.7511
36.2400
29.0042
20.7203
OH
OH
OH
OH
Br
5
200
180
160
140
120
100
80
60
40
20
0
(ppm)

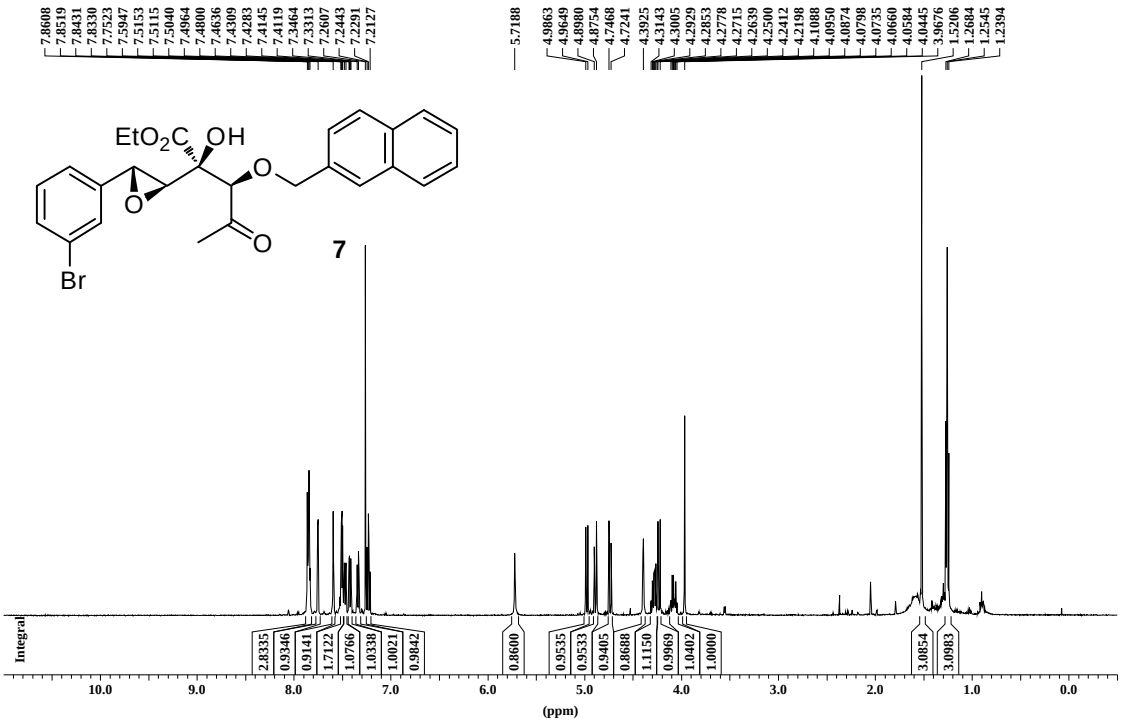
1H AMX500 dxw0825-3 1088-1

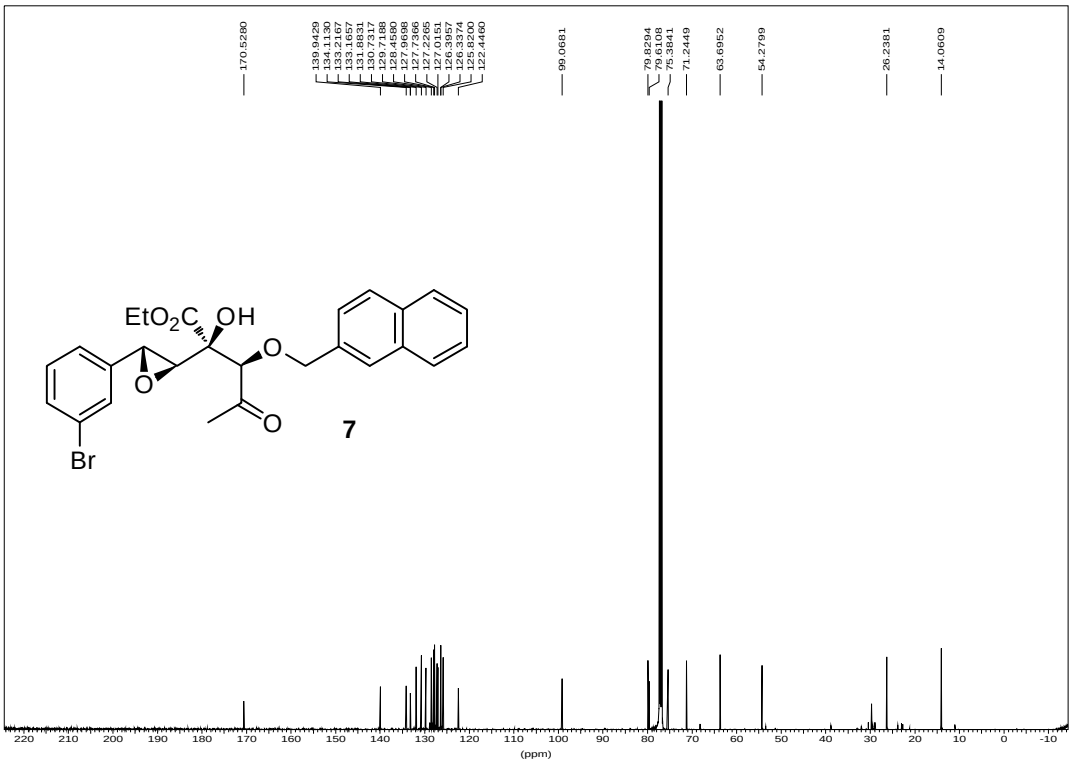


13C AMX500 dxw0825-13 1088-1



1H AMX500 dxw0825-6 epo-2





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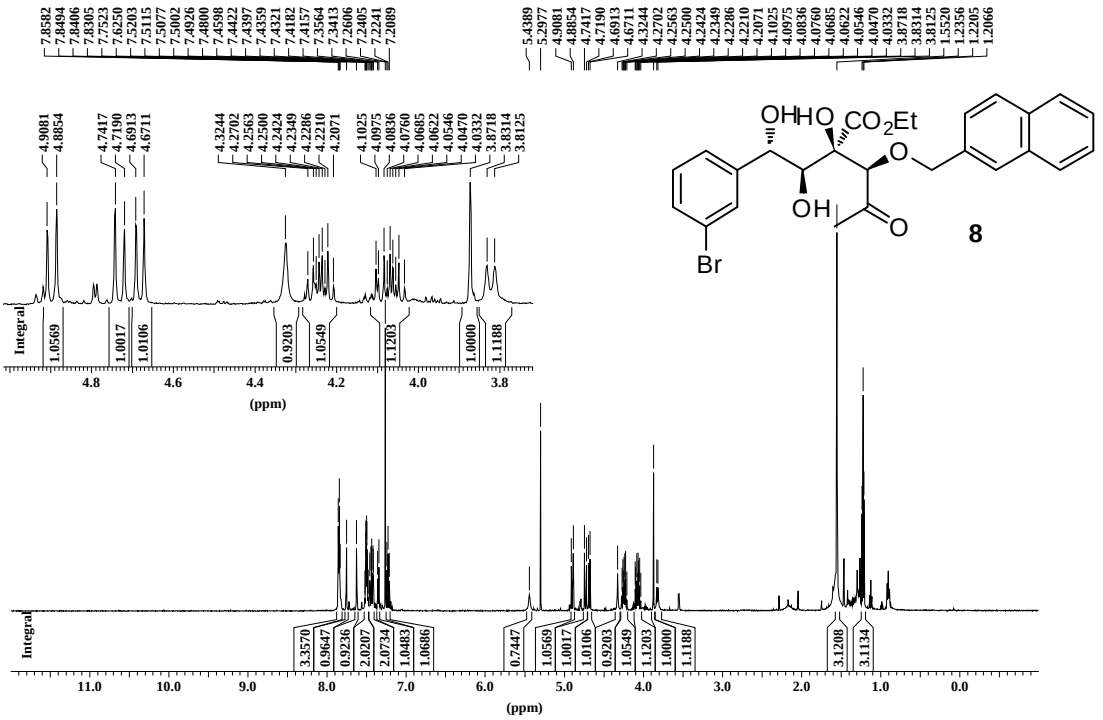

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