Direct β-C–H Ketoalkylation of Enaminoesters with Cyclopropanols under Metal-Free Conditions

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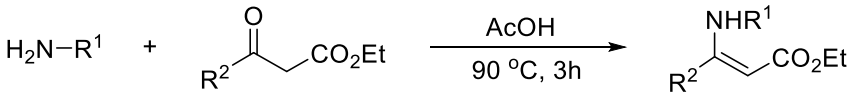
Supporting Information

General:

All reagents were purchased from commercial suppliers and used without further purification. Flash chromatography was carried out with silica gel (200-300 mesh). Analytical TLC was performed with silica gel GF254 plates, and the products were visualized by UV detection. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded in $CDCl_3$. Chemical shifts (δ) are reported in ppm using TMS as internal standard and spin-spin coupling constants (*J*) are given in Hz. Proton and carbon multiplicity are recorded as singlet (s), doublet (d), triplet (t), quartet (q), multiplet (m) and broad (br). Mass spectra (MS) were measured on Ion trap mass spectrometer by ESI. The high-resolution mass spectra (HRMS) were measured on a Bruker Daltonics APEX II 47e spectrometer by ESI.

Substrates preparation:

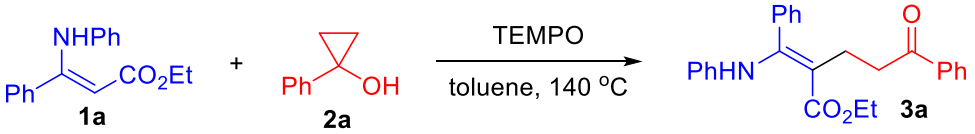
General procedure for the synthesis of enamines 1a-q.



A mixture of β-keto ester (1.16 g, 10 mmol), aniline (0.93 g, 10 mmol) and acetic acid (60.1 mg, 1 mmol) was placed in a 100 mL round-bottomed flask at 90 °C for 3 h. After cooling, the reaction mixture was added 30 mL water and extracted with EA (40 mL x 3). Next, the combined organic layer was dried with Na_2SO_4, filtered and concentrated under reduced pressure. The desired product was obtained by silica gel chromatography to give the pure product. [1]

General experimental procedure:

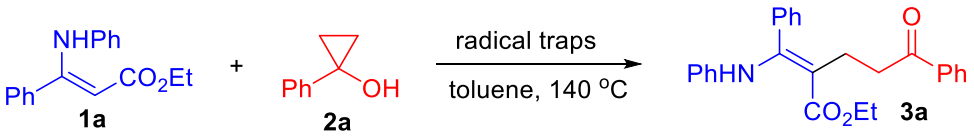
Optimization of the amount of TEMPO



Entry	Solvent	The amount of TEMPO	Yield (%)
1	Toluene	3 equiv.	87

2	Toluene	2 equiv.	85
3	Toluene	1 equiv.	49
4	Toluene	0.2 equiv.	38

Optimization of the radical traps

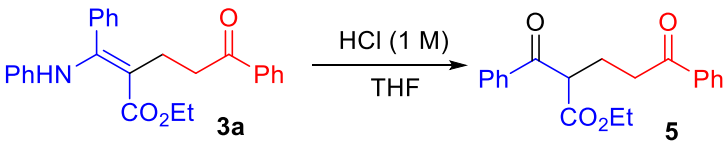


Entry	Solvent	The radical traps	Yield (%)
1	Toluene	TEMPO	85
2	Toluene	4-HO-TEMPO	78
3	Toluene	4-Ac-NH-TEMPO	41
4	Toluene	BHT	-
5	Toluene	NHPI	-

Typical experimental procedure for β-ketoalkylation of enamines:

A 25 mL oven-dried reaction pressure tube were charged with enamines **1** (0.5 mmol, 2.5 equiv.), cyclopropanols **2** (0.2 mmol), TEMPO (0.4 mmol, 2 equiv.) and toluene (2.0 mL). The tube was then sealed and the mixture was stirred for 36 h at 140 °C under air. Upon completion of the reaction, the solvent was then removed under vacuo. The residue was purified by chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/20 to 1/3) to give the corresponding products **3** and **4**. The identity and purity of the product was confirmed by ^{1}H and ^{13}C spectroscopic analysis.

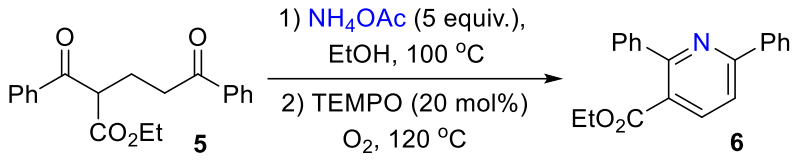
The experimental procedure for the synthesis of 5:



To a solution of **3a** (0.3 mmol) in THF (2 mL) was added 2 mL HCl (1 M) at 0 °C, and the mixture was stirred at room temperature. After **3a** was complete consumption, the mixture was concentrated under reduced pressure and then added water (10 mL), which was subsequently extracted with EtOAc (15 mL × 3). The combined organic phase

was then washed with brine, dried over Na_2SO_4. After the solvent had been removed under reduced pressure, the residue was purified by column chromatography on silica gel using petroleum ether/ethyl acetate as eluent to give the target 1,5-dicarbonyl compound **5** in 93% yield. [2]

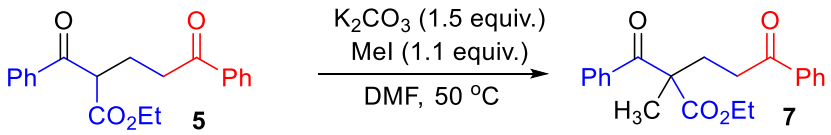
The experimental procedure for the synthesis of 6:



1) An oven-dried reaction pressure tube was charged with 1,5-dicarbonyl compound **5** (0.3 mmol), EtOH (2 mL) and NH_4OAc (5 equiv.). The resulting mixture was reflux at 100 °C for 3 h. After **5** was complete consumption, the mixture was cooled at room temperature. [3]

2) TEMPO (20 %) was added to the above solution and stirred at 120 °C under O_2 atmosphere for 24 h. The resulting reaction mixture was mixed with few silica gel and concentrated, then purified by flash column chromatography on silica gel with ethyl acetate/petroleum ether as eluent to give the target ethyl 2,6-diphenylnicotinate **6** in 70% yield. [4]

The experimental procedure for the synthesis of 7:

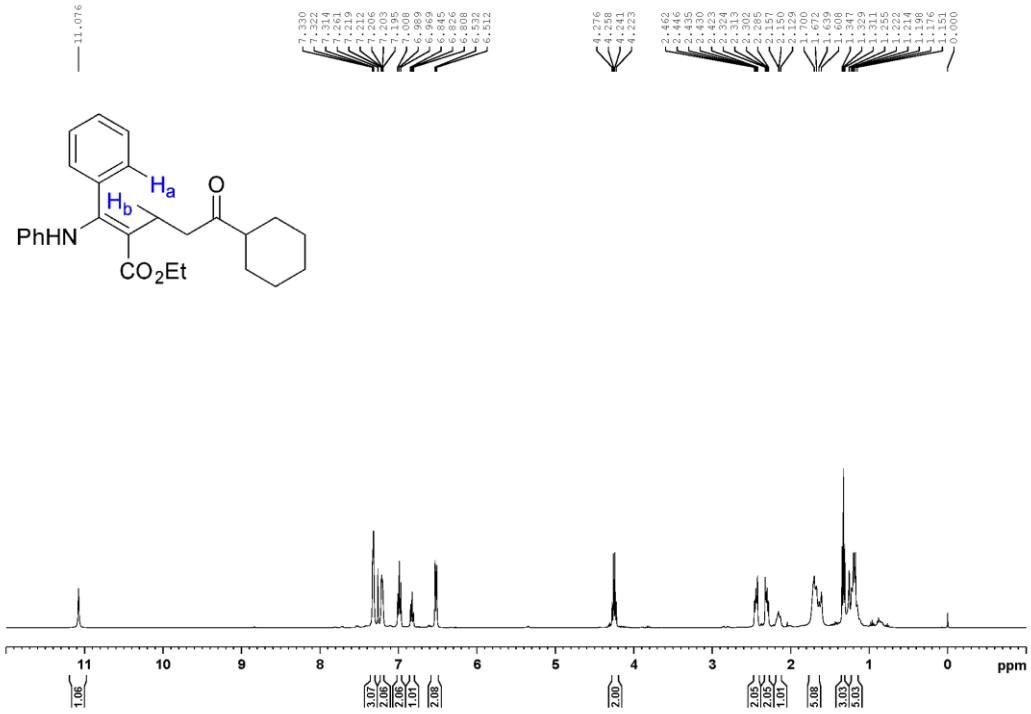


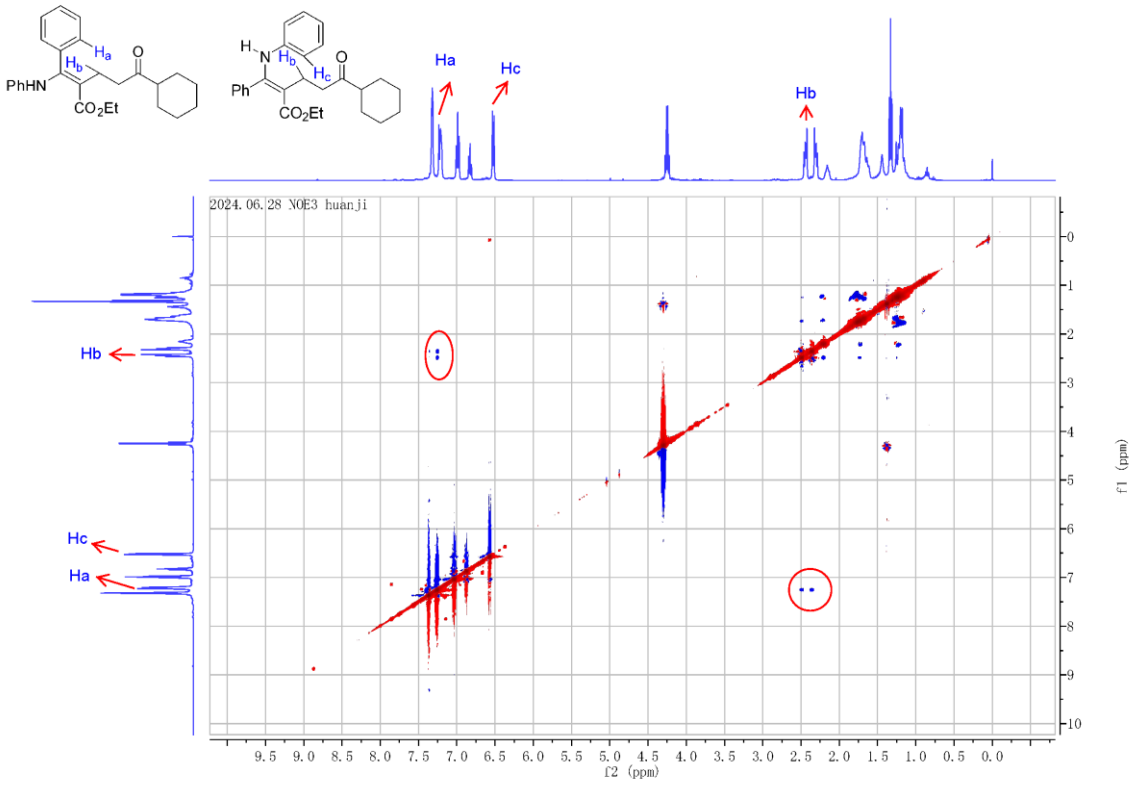
To a solution of **5** (0.3 mmol) in DMF (2 mL) was added K_2CO_3 (4.5 mmol, 1.5 equiv.) and MeI (3.3 mmol, 1.1 equiv.) and the mixture was stirred at 50 °C for 12 h. After **5** was complete consumption, the mixture was added water (10 mL), which was subsequently extracted with EtOAc (15 mL × 3). The combined organic phase was then washed with brines (10 mL × 6), dried over Na_2SO_4. After the solvent had been removed under reduced pressure, the residue was purified by column chromatography on silica gel using petroleum ether/ethyl acetate as eluent to give the target compound **7** in 82% yield. [5]

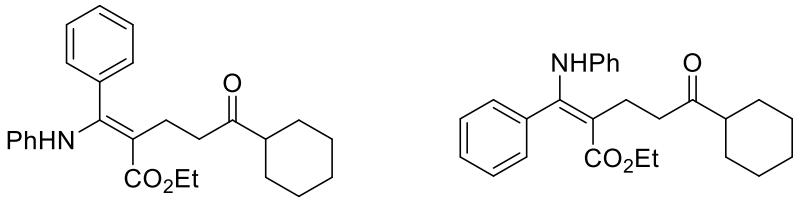
Study on the stereoconfiguration of products:

The NOE experiment of 4m

The product **4m** may have two different structures (A and B) as shown in below. In order to identify the configuration of **4m**, the NOE experiment has been carried out and the result is shown as below:







(A) (B)

From the above NOE experiment, we found that H_a and H_b of the configuration **A** are adjacent in space, and H_b and H_c of the configuration **B** are not adjacent in space. Thus, the configuration **B** could be excluded and the product **4m** should be Z-isomer as reported.

DFT Computation Method

The M06-2X functional combined with the conductor-like polarizable continuum model was used to optimize all the complexes by the ORCA package[6]. The 6-311G** basis set was adopted for all atoms. Frequency analyses were performed at the same level.

Cartesian Coordinates

3a

C	1.43487800	0.29934700	-0.27196200
C	0.32570000	1.05566900	-0.57058300
C	1.48514800	-1.15103500	-0.62287500
C	1.48855600	-1.57288300	-1.95208100
C	1.56254200	-2.09529700	0.39985700
C	1.57256600	-2.92641900	-2.25469700
H	1.43717700	-0.83476800	-2.74509700
C	1.63634300	-3.44974100	0.09593600
H	1.57685900	-1.76288100	1.43216100
C	1.64580700	-3.86665200	-1.23093100
H	1.58061000	-3.24696100	-3.28935100
H	1.69285700	-4.17834300	0.89547100
H	1.70986400	-4.92181000	-1.46766300
C	0.28599600	2.46212800	-0.17170800
O	1.15936700	3.07458700	0.42540800
O	-0.86692600	3.06430600	-0.52260900
C	-0.99556100	4.45670700	-0.19243200
H	-2.06868800	4.64062300	-0.21087500
H	-0.61619000	4.61863200	0.81679800
C	-0.26771900	5.32751400	-1.19897900
H	-0.44392400	6.38093500	-0.97294700
H	0.80489700	5.13831700	-1.15935200
H	-0.63048500	5.12434600	-2.20778800
C	-0.92788900	0.46915700	-1.18103500
H	-1.34387500	1.16442900	-1.91038500
H	-0.71304900	-0.45148400	-1.71794300
C	-1.98833000	0.17389900	-0.11986000
H	-1.59797300	-0.54011300	0.61538300
H	-2.23772200	1.08550200	0.43325800

C	-3.26072200	-0.39658100	-0.71127500
O	-3.36655500	-0.59519200	-1.90061100
C	-4.40003700	-0.71686900	0.21520700
C	-4.32580000	-0.50311700	1.59263300
C	-5.56891200	-1.24698500	-0.33564100
C	-5.40814800	-0.81574100	2.40793000
H	-3.42722800	-0.09270600	2.03696700
C	-6.64840600	-1.55855500	0.47761000
H	-5.60742000	-1.40631800	-1.40623800
C	-6.56874100	-1.34265700	1.85174700
H	-5.34538200	-0.64772000	3.47617300
H	-7.55236700	-1.96938400	0.04448600
H	-7.41154300	-1.58524800	2.48815100
N	2.49956400	0.78838500	0.42717000
H	2.36965000	1.73594200	0.77200000
C	3.75913100	0.20055700	0.66524500
C	4.38278300	0.49384800	1.88158200
C	4.42446800	-0.60066200	-0.26629800
C	5.64188500	-0.01570100	2.16804000
H	3.86559500	1.11912200	2.60077300
C	5.67583400	-1.12189600	0.03825600
H	3.97367100	-0.80678200	-1.22723600
C	6.29215200	-0.83605200	1.25201000
H	6.11058900	0.22347500	3.11512100
H	6.17827900	-1.74566400	-0.69141100
H	7.27061200	-1.24134800	1.47766600

3a´

C	-1.18018300	-0.06648500	-0.71079200
C	-1.53441600	-1.37568300	-0.61866600

C	-1.94315400	1.08111900	-0.15583500
C	-2.50168500	1.01549000	1.12155300
C	-2.05659500	2.26140600	-0.89103700
C	-3.18106600	2.10565100	1.64786500
H	-2.39017900	0.10685500	1.70478100
C	-2.74376200	3.34975300	-0.36766200
H	-1.60524900	2.31628700	-1.87481500
C	-3.30731000	3.27481600	0.90206900
H	-3.60682800	2.04649100	2.64273800
H	-2.83547200	4.25886300	-0.94962100
H	-3.83537500	4.12672100	1.31346900
C	-2.88829500	-1.83209800	-0.22770800
O	-3.10498700	-2.89533400	0.30933800
O	-3.86728800	-0.98546400	-0.57976300
C	-5.19953500	-1.34567500	-0.18052100
H	-5.84446800	-0.76532100	-0.83811400
H	-5.34920000	-2.40887500	-0.37047600
C	-5.44860700	-1.00792400	1.27757800
H	-6.47795600	-1.25654300	1.54337600
H	-4.77928700	-1.58253600	1.91944600
H	-5.28975600	0.05687900	1.45343300
C	-0.53035500	-2.48269000	-0.87389600
H	-1.03268800	-3.42578700	-0.66918300
H	-0.22042700	-2.51625200	-1.92366200
C	0.72524000	-2.37755600	0.01242600
H	0.51597400	-1.76144600	0.89049100
H	0.99635600	-3.36842600	0.39600300
C	1.97277600	-1.85259500	-0.67770300
O	2.05514400	-1.79443600	-1.88726200

C	3.11328800	-1.39676400	0.18063000
C	3.14645000	-1.60812300	1.56132600
C	4.16546500	-0.71799100	-0.43657800
C	4.21723800	-1.14028800	2.31286100
H	2.34477200	-2.14099900	2.05768000
C	5.22937600	-0.24252400	0.31627900
H	4.12052900	-0.55983000	-1.50671700
C	5.25561100	-0.45233100	1.69225700
H	4.24079400	-1.31036000	3.38232400
H	6.03555300	0.29646000	-0.16676300
H	6.08550100	-0.08056500	2.28163600
N	-0.00459100	0.26470500	-1.36781100
H	0.33872000	-0.42495600	-2.02473600
C	0.99750400	1.13356600	-0.88940700
C	1.94509200	1.61049600	-1.79857900
C	1.11373100	1.46426700	0.46218900
C	2.99483200	2.40466300	-1.35960600
H	1.85243400	1.34324800	-2.84542800
C	2.16335100	2.26966400	0.88941000
H	0.39742200	1.07698300	1.17701700
C	3.10876300	2.74220600	-0.01381500
H	3.72715100	2.76206500	-2.07419900
H	2.24850900	2.51361500	1.94174100
H	3.92982700	3.36014400	0.32792600

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[1] (a) M. Zhao, F. Wang and X.-W. Li, *Org. Lett.* 2012, **14**, 1412; (b) G. Bartoli, M. Bosco, M. Locatelli, E. Marcantoni, P. Melchiorre and L. Sambri, *Synlett.,* 2004, **2**, 239; (c) S. Tang, X.-L. Gao and A. Lei, *Chem. Commun.,* 2017, **53**, 3354; (d) J.-L. Zhan, L. Zhu, J.-N. Bai, J.-B. Liu, S.-H. Zhang, Y.-Q. Xie, B.-M. Hu, Y. Wang and W.-J. Han, *Org.*

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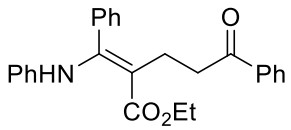
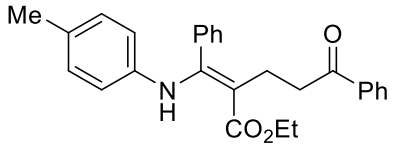
Analytical Data for Products:

Ethyl (Z)-5-oxo-5-phenyl-2-(phenyl(phenylamino)methylene)pentanoate (3a)



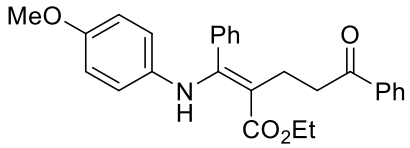
Yellow solid; (69 mg, 87%); mp: 62–66 °C; R_f = 0.21 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.17 (s, 1H), 7.79 (d, *J* = 7.6 Hz, 2H), 7.49 (t, *J* = 7.2 Hz, 1H), 7.37 (t, *J* = 7.2 Hz, 2H), 7.32 (s, 3H), 7.24 (brs, 2H), 6.99 (t, *J* = 7.2 Hz, 2H), 6.83 (t, *J* = 7.2 Hz, 1H), 6.54 (d, *J* = 8.0 Hz, 2H), 4.27 (q, *J* = 6.8 Hz, 2H), 2.99–2.95 (m, 2H), 2.55–2.51 (m, 2H), 1.33 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.9, 168.8, 155.7, 138.2, 134.5, 132.5, 130.7, 126.8, 126.7, 126.6, 126.38, 126.35, 126.0, 120.6, 120.0, 94.9, 57.6, 38.2, 21.6, 12.5; ESI-HRMS: m/z Calcd for $C_{26}H_{25}NO_3Na$, $[M + Na]^+$: 422.1727, found 422.1725.

Ethyl (Z)-5-oxo-5-phenyl-2-(phenyl(p-tolylamino)methylene)pentanoate (3b)



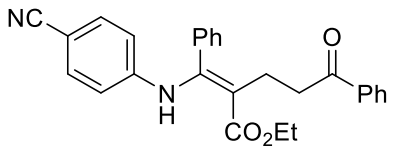
Yellow solid; (71 mg, 86%); mp: 80–84 °C; R_f = 0.24 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.11 (s, 1H), 7.79–7.77 (m, 2H), 7.49 (t, *J* = 7.6 Hz, 1H), 7.37 (t, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 3.2 Hz, 3H), 7.24–7.21 (m, 2H), 6.80 (d, *J* = 8.4 Hz, 2H), 6.46 (d, *J* = 8.0 Hz, 2H), 4.26 (q, *J* = 7.2 Hz, 2H), 2.98–2.94 (m, 2H), 2.53–2.49 (m, 2H), 2.15 (s, 3H), 1.32 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.1, 170.9, 158.3, 137.7, 136.7, 134.8, 132.6, 132.3, 129.0, 128.9, 128.6, 128.5, 128.4, 128.0, 122.3, 96.2, 59.5, 40.3, 23.7, 20.5, 14.5; ESI-HRMS: m/z Calcd for $C_{27}H_{27}NO_3Na$, $[M + Na]^+$: 436.1883, found 436.1887.

Ethyl (Z)-2-(((4-methoxyphenyl)amino)(phenyl)methylene)-5-oxo-5-phenylpentanoate (3c)



Yellow oil; (80 mg, 93%); R_f = 0.2 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.01 (s, 1H), 7.78 (d, *J* = 7.6 Hz, 2H), 7.49 (t, *J* = 7.6 Hz, 1H), 7.36 (t, *J* = 7.6 Hz, 2H), 7.29–7.27 (m, 3H), 7.20–7.18 (m, 2H), 6.55 (s, 4H), 4.26 (q, *J* = 7.2 Hz, 2H), 3.64 (s, 3H), 2.98–2.94 (m, 2H), 2.52–2.48 (m, 2H), 1.32 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.0, 170.8, 158.9, 155.5, 136.5, 134.5, 133.2, 132.5, 128.8, 128.4, 128.3, 128.2, 127.9, 124.4, 113.5, 95.4, 59.3, 55.0, 40.2, 23.7, 14.4; ESI-HRMS: m/z Calcd for $C_{27}H_{27}NO_4Na$, $[M + Na]^+$: 452.1832, found 452.1837.

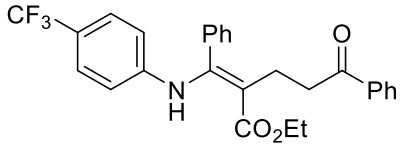
Ethyl (Z)-2-(((4-cyanophenyl)amino)(phenyl)methylene)-5-oxo-5-phenylpentanoate (3d)



White solid; (37 mg, 44%); mp: 143–147 °C; R_f = 0.29 (hexanes/ethyl acetate 5:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.37 (s, 1H), 7.80 (d, *J* = 7.6 Hz, 2H), 7.52 (t, *J* = 7.6 Hz, 1H),

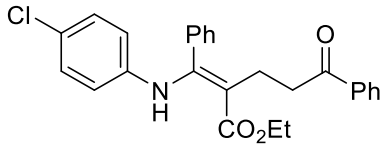
7.28–7.23 (m, 4H), 6.43 (d, *J* = 8.8 Hz, 2H), 4.28 (q, *J* = 7.2 Hz, 2H), 3.00–2.96 (m, 2H), 2.59–2.55 (m, 2H), 1.33 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.5, 170.7, 155.0, 144.6, 136.6, 134.0, 132.9, 132.8, 129.6, 129.2, 128.7, 128.5, 128.0, 120.0, 119.1, 104.3, 101.6, 60.3, 39.8, 23.4, 14.4; ESI-HRMS: m/z Calcd for $C_{27}H_{24}N_2O_3Na$, $[M + Na]^+$: 447.1679, found 447.1684.

Ethyl-(Z)-5-oxo-5-phenyl-2-(phenyl((4-(trifluoromethyl)phenyl)amino)methylene)pentanoate (3e)



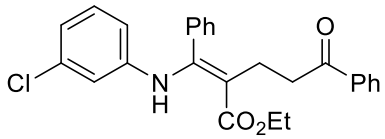
White solid; (75 mg, 80%); mp: 94–96 °C; R_f = 0.21 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.32 (s, 1H), 7.81–7.79 (m, 2H), 7.51 (t, *J* = 7.2 Hz, 1H), 7.40–7.36 (m, 5H), 7.28–7.22 (m, 4H), 6.50 (d, *J* = 8.4 Hz, 2H), 4.28 (q, *J* = 7.2 Hz, 2H), 3.00–2.96 (m, 2H), 2.58–2.54 (m, 2H), 1.33 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.7, 170.8, 156.0, 143.6, 136.6, 134.3, 132.8, 129.3, 129.0, 128.8, 128.4, 128.2, 128.0, 125.8 (q, $^{C,F}J_3$ = 4 Hz), 124.2 (q, $^{C,F}J_1$ = 269 Hz), 123.7 (q, $^{C,F}J_2$ = 32 Hz), 120.2, 99.9, 60.1, 40.0, 23.5, 14.4; ^{19}F NMR (376 MHz, $CDCl_3$): δ -63.1; ESI-HRMS: m/z Calcd for $C_{27}H_{24}F_3NO_3Na$, $[M + Na]^+$: 490.1600, found 490.1605.

Ethyl (Z)-2-(((4-chlorophenyl)amino)(phenyl)methylene)-5-oxo-5-phenylpentanoate (3f)



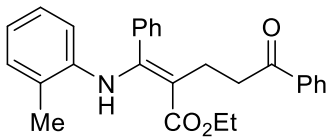
White solid; (74 mg, 86%); mp: 85–88 °C; R_f = 0.21 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.14 (s, 1H), 7.79 (d, *J* = 7.6 Hz, 2H), 7.50 (t, *J* = 7.6 Hz, 1H), 7.39–7.32 (m, 5H), 7.23–7.21 (m, 2H), 6.94 (d, *J* = 8.8 Hz, 2H), 6.45 (d, *J* = 8.8 Hz, 2H), 4.26 (q, *J* = 7.2 Hz, 2H), 2.98–2.94 (m, 2H), 2.55–2.51 (m, 2H), 1.32 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.9, 170.8, 157.3, 139.0, 136.6, 134.3, 132.7, 129.0, 128.9, 128.7, 128.5, 128.4, 128.0, 127.8, 123.0, 97.9, 59.8, 40.1, 23.6, 14.4; ESI-HRMS: m/z Calcd for $C_{26}H_{24}ClNO_3Na$, $[M + Na]^+$: 456.1337, found 456.1335.

Ethyl (Z)-2-(((3-chlorophenyl)amino)(phenyl)methylene)-5-oxo-5-phenylpentanoate (3g)



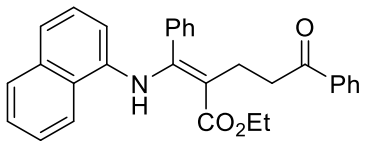
White solid; (81 mg, 93%); mp: 57–59 °C; R_f = 0.24 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.17 (s, 1H), 7.80–7.78 (m, 2H), 7.50 (t, *J* = 7.6 Hz, 1H), 7.40–7.34 (m, 5H), 7.26–7.23 (m, 2H), 6.89 (t, *J* = 8.0 Hz, 1H), 6.79–6.77 (m, 1H), 6.49 (t, *J* = 2.0 Hz, 1H), 6.37 (dd, *J* = 1.6 Hz, *J* = 8.0 Hz, 1H), 4.27 (q, *J* = 7.2 Hz, 2H), 2.99–2.95 (m, 2H), 2.56–2.52 (m, 2H), 1.32 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.8, 170.7, 156.8, 141.7, 136.6, 134.2, 134.0, 132.7, 129.3, 129.1, 128.79, 128.77, 128.4, 128.0, 122.4, 121.5, 119.6, 98.6, 59.8, 40.0, 23.5, 14.4; ESI-HRMS: m/z Calcd for $C_{26}H_{24}ClNO_3Na$, $[M + Na]^+$: 456.1337, found 456.1332.

Ethyl (Z)-5-oxo-5-phenyl-2-(phenyl(o-tolylamino)methylene)pentanoate (3h)



Yellow solid; (75 mg, 91%); mp: 80–82 °C; R_f = 0.25 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.92 (s, 1H), 7.80 (d, *J* = 7.2 Hz, 2H), 7.49 (t, *J* = 7.2 Hz, 1H), 7.37 (t, *J* = 7.6 Hz, 2H), 7.25 (t, *J* = 3.2 Hz, 3H), 7.19–7.17 (m, 2H), 7.05 (d, *J* = 7.2 Hz, 1H), 6.78 (t, *J* = 7.2 Hz, 1H), 6.70 (t, *J* = 7.2 Hz, 1H), 6.30 (d, *J* = 8.0 Hz, 1H), 4.27 (q, *J* = 7.2 Hz, 2H), 3.01–2.97 (m, 2H), 2.57–2.53 (m, 2H), 2.38 (s, 3H), 1.33 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.0, 170.8, 158.5, 138.8, 136.6, 134.7, 132.5, 130.6, 129.9, 128.6, 128.4, 128.21, 128.19, 127.9, 125.5, 124.0, 123.3, 96.6, 59.4, 40.2, 23.7, 18.2, 14.4; ESI-HRMS: m/z Calcd for $C_{27}H_{27}NO_3Na$, $[M + Na]^+$: 436.1883, found 436.1887.

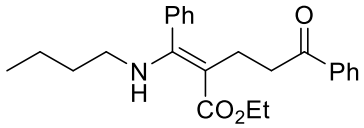
Ethyl (Z)-2-((naphthalen-1-ylamino)(phenyl)methylene)-5-oxo-5-phenylpentanoate (3i)



Yellow oil; (81 mg, 90%); R_f = 0.30 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.45 (s, 1H), 8.27 (d, *J* = 8.4 Hz, 1H), 7.81 (d, *J* = 7.2 Hz, 2H), 7.73 (d, *J* = 8.0 Hz,

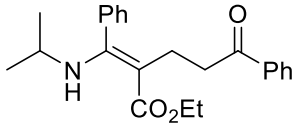
1H), 7.55 (t, *J* = 7.2 Hz, 1H), 7.50–7.43 (m, 2H), 7.40–7.34 (m, 3H), 7.21–7.15 (m, 5H), 6.99 (t, *J* = 8.0 Hz, 1H), 6.54 (d, *J* = 7.2 Hz, 1H), 4.32 (q, *J* = 7.2 Hz, 2H), 3.05–3.01 (m, 2H), 2.63–2.59 (m, 2H), 1.35 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): *δ* 200.1, 171.2, 159.4, 136.7, 136.2, 134.8, 133.9, 132.7, 129.2, 128.7, 128.5, 128.4, 128.2, 128.12, 128.06, 126.2, 126.0, 125.0, 124.1, 122.3, 121.8, 97.5, 59.7, 40.3, 23.9, 14.5; ESI-HRMS: m/z Calcd for $C_{30}H_{27}NO_3Na$, $[M + Na]^+$: 472.1883, found 472.1888.

Ethyl (Z)-2-((butylamino)(phenyl)methylene)-5-oxo-5-phenylpentanoate (3j)



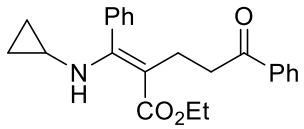
Yellow oil; (65 mg, 86%); R_f = 0.32 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): *δ* 9.23 (t, *J* = 5.6 Hz, 1H), 7.78–7.56 (m, 2H), 7.48 (t, *J* = 7.2 Hz, 1H), 7.44–7.34 (m, 5H), 7.20 (dd, *J* = 1.6 Hz, *J* = 7.6 Hz, 2H), 4.21 (q, *J* = 7.2 Hz, 2H), 2.90–2.86 (m, 2H), 2.81–2.76 (m, 2H), 2.32–2.27 (m, 2H), 1.44–1.36 (m, 2H), 1.32–1.24 (m, 5H), 0.82 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): *δ* 200.3, 170.8, 163.2, 136.6, 135.0, 132.4, 128.5, 128.3, 128.2, 127.9, 127.5, 91.5, 58.8, 44.0, 40.6, 32.8, 24.1, 19.6, 14.5, 13.5; ESI-HRMS: m/z Calcd for $C_{24}H_{30}NO_3Na$, $[M + Na]^+$: 402.2040, found 402.2044.

Ethyl (Z)-2-((isopropylamino)(phenyl)methylene)-5-oxo-5-phenylpentanoate (3k)



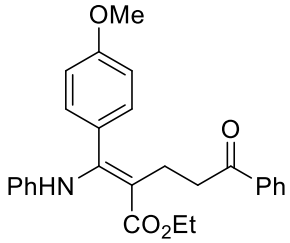
White solid; (72 mg, 98%); mp: 64–67 °C; R_f = 0.24 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): *δ* 9.17 (d, *J* = 9.6 Hz, 1H), 7.77–7.75 (m, 2H), 7.48 (t, *J* = 7.2 Hz, 1H), 7.44–7.34 (m, 5H), 7.23 (dd, *J* = 1.6 Hz, *J* = 7.6 Hz, 2H), 4.21 (q, *J* = 7.2 Hz, 2H), 3.12–3.03 (m, 1H), 2.89–2.85 (m, 2H), 2.30–2.26 (m, 2H), 1.30 (t, *J* = 7.2 Hz, 3H), 1.06 (d, *J* = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): *δ* 200.3, 170.8, 162.2, 136.6, 135.2, 132.4, 128.5, 128.4, 128.2, 128.0, 127.5, 91.5, 58.8, 45.6, 40.6, 24.3, 24.0, 14.5; ESI-HRMS: m/z Calcd for $C_{23}H_{27}NO_3Na$, $[M + Na]^+$: 388.1883, found 388.1888.

Ethyl (Z)-2-((cyclopropylamino)(phenyl)methylene)-5-oxo-5-phenylpentanoate (3l)



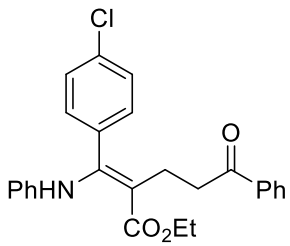
Yellow oil; (60 mg, 83%); R_f = 0.38 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.13 (d, *J* = 2.4 Hz, 1H), 7.77–7.75 (m, 2H), 7.48 (t, *J* = 7.2 Hz, 1H), 7.44–7.34 (m, 5H), 7.27–7.25 (m, 2H), 4.19 (q, *J* = 7.2 Hz, 2H), 2.90–2.86 (m, 2H), 2.34–2.30 (m, 2H), 2.21–2.15 (m, 1H), 1.29 (t, *J* = 7.2 Hz, 3H), 0.46–0.42 (m, 2H), 0.42–0.36 (m,2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.3, 170.7, 164.0, 136.6, 135.7, 132.5, 128.4, 128.3, 128.2, 128.0, 127.7, 92.9, 59.0, 40.5, 26.5, 24.0, 14.5, 8.2; ESI-HRMS: m/z Calcd for $C_{23}H_{25}NO_3Na$, $[M + Na]^+$: 386.1727, found 386.1734.

Ethyl (Z)-2-((4-methoxyphenyl)(phenylamino)methylene)-5-oxo-5-phenylpentanoate (3m)



Yellow oil; (62 mg, 72%); R_f = 0.20 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.12 (s, 1H), 7.81–7.79 (m, 2H), 7.51 (t, *J* = 7.6 Hz, 1H), 7.38 (t, *J* = 8.0 Hz, 2H), 7.15 (dd, *J* = 2.0 Hz, *J* = 6.8 Hz, 2H), 7.02 (t, *J* = 8.0 Hz, 2H), 6.86–6.82 (m, 3H), 6.55 (d, *J* = 7.6 Hz, 2H), 4.26 (q, *J* = 7.2 Hz, 2H), 3.78 (s, 3H), 2.99–2.95 (m, 2H), 2.58–2.54 (m, 2H), 1.33 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.2, 170.9, 159.8, 157.7, 140.6, 136.7, 132.7, 130.3, 128.5, 128.4, 128.1, 126.8, 122.5, 122.0, 114.0, 97.2, 59.6, 55.2, 40.4, 23.8, 14.5; ESI-HRMS: m/z Calcd for $C_{27}H_{27}NO_4Na$, $[M + Na]^+$: 452.1838, found 452.1843.

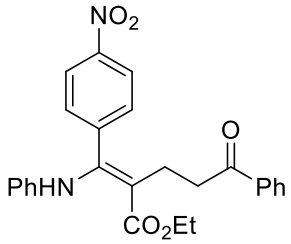
Ethyl (Z)-2-((4-chlorophenyl)(phenylamino)methylene)-5-oxo-5-phenylpentanoate (3n)



White solid; (52 mg, 60%); mp: 76–79 °C; R_f = 0.43 (hexanes/ethyl acetate 10:1); ^{1}H NMR

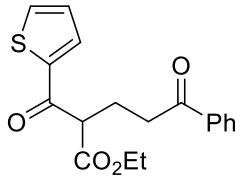
(400 MHz, $CDCl_3$): δ 11.16 (s, 1H), 7.71–7.68 (m, 2H), 7.35–7.32 (m, 5H), 7.24–7.22 (m, 2H), 7.00 (t, *J* = 7.6 Hz, 2H), 6.84 (t, *J* = 7.2 Hz, 1H), 6.54 (d, *J* = 7.6 Hz, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 2.94–2.90 (m, 2H), 2.53–2.49 (m, 2H), 1.34 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.9, 170.8, 158.0, 140.2, 139.1, 134.9, 134.6, 129.5, 128.9, 128.8, 128.7, 128.6, 128.5, 122.8, 122.2, 96.7, 59.7, 40.3, 23.8, 14.5; ESI-HRMS: m/z Calcd for $C_{26}H_{24}ClNO_3Na$, $[M + Na]^+$: 456.1342, found 456.1344.

Ethyl (Z)-2-((4-nitrophenyl)(phenylamino)methylene)-5-oxo-5-phenylpentanoate (3o)



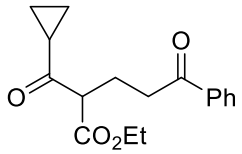
Red oil; (47 mg, 53%); R_f = 0.13 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.99 (s, 1H), 8.13 (d, *J* = 8.4 Hz, 2H), 7.83 (d, *J* = 7.6 Hz, 2H), 7.55–7.49 (m, 1H), 7.42–7.38 (m, 4H), 7.03 (t, *J* = 7.6 Hz, 2H), 6.89 (d, *J* = 7.6 Hz, 1H), 6.58 (d, *J* = 8.0 Hz, 2H), 4.29 (q, *J* = 7.2 Hz, 2H), 3.00 (t, *J* = 7.6 Hz, 2H), 2.50 (t, *J* = 7.6 Hz, 2H), 1.35 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.9, 169.8, 154.5, 147.0, 140.7, 139.0, 135.8, 132.3, 129.8, 128.1, 127.8, 127.3, 123.0, 122.4, 97.5, 59.4, 39.1, 22.8, 13.8; ESI-HRMS: m/z Calcd for $C_{26}H_{24}N_2O_5Na$, $[M + Na]^+$: 467.1583, found 467.1588.

Ethyl 5-oxo-5-phenyl-2-(thiophene-2-carbonyl)pentanoate (3p')



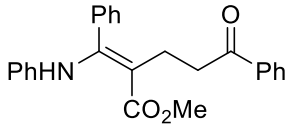
Yellow oil; (45 mg, 68%); R_f = 0.38 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97–7.94 (m, 3H), 7.48 (dd, *J* = 0.8 Hz, *J* = 4.8 Hz, 1H), 7.56 (t, *J* = 7.2 Hz, 1H), 7.46 (t, *J* = 7.2 Hz, 2H), 7.17–7.15 (m, 1H), 4.43 (t, *J* = 7.2 Hz, 1H), 4.21–4.13 (m, 2H), 3.24–3.16 (m, 1H), 3.13–3.06 (m, 1H), 2.52–2.38 (m, 2H), 1.20 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.2, 188.0, 169.4, 143.3, 136.6, 135.0, 133.5, 133.2, 128.6, 128.4, 128.0, 61.6, 54.0, 35.5, 23.4, 14.0; ESI-HRMS: m/z Calcd for $C_{18}H_{18}O_4SNa$, $[M + Na]^+$: 353.0823, found 353.0821.

Ethyl 2-(cyclopropanecarbonyl)-5-oxo-5-phenylpentanoate (3q')



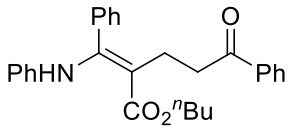
Yellow oil; (35 mg, 61%); R_f = 0.35 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97–7.94 (m, 2H), 7.57 (t, *J* = 7.2 Hz, 1H), 7.46 (t, *J* = 7.2 Hz, 2H), 4.26–4.18 (m, 2H), 3.78 (dd, *J* = 2.8 Hz, *J* = 7.6 Hz, 1H), 3.06 (t, *J* = 7.2 Hz, 2H), 2.41–2.26 (m, 2H), 2.16–2.10 (m, 1H), 1.27 (t, *J* = 7.6 Hz, 3H), 1.15–1.07 (m, 2H), 0.98–0.93 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 205.3, 199.1, 169.6, 136.6, 133.2, 128.6, 128.0, 61.4, 58.5, 35.7, 22.3, 20.0, 14.1, 11.9, 11.7; ESI-HRMS: m/z Calcd for $C_{17}H_{20}O_4Na$, $[M + Na]^+$: 311.1259, found 311.1264.

Methyl (Z)-5-oxo-5-phenyl-2-(phenyl(phenylamino)methylene)pentanoate (3r)



Colorless, viscous liquid; (62 mg, 80%); R_f = 0.25 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.13 (s, 1H), 7.78–7.76 (m, 2H), 7.52–7.49 (m, 1H), 7.38 (d, *J* = 7.6 Hz, 2H), 7.33–7.31 (m, 3H), 7.25–7.22 (m, 2H), 7.02–6.98 (m, 2H), 6.84 (t, *J* = 7.6 Hz, 1H), 6.55 (d, *J* = 8.0 Hz, 2H), 3.78 (s, 3H), 2.97–2.93 (m, 2H), 2.54–2.50 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.8, 171.0, 157.7, 139.9, 136.3, 134.2, 132.4, 128.6, 128.5, 128.3, 128.2, 128.1, 127.7, 122.5, 121.9, 96.4, 50.8, 39.9, 23.2; ESI-HRMS: m/z Calcd for $C_{25}H_{23}NO_3Na$, $[M + Na]^+$: 408.1570, found 408.1574.

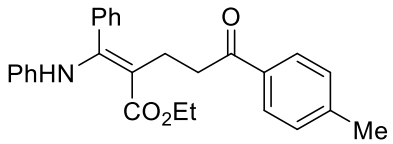
Butyl (Z)-5-oxo-5-phenyl-2-(phenyl(phenylamino)methylene)pentanoate (3s)



Colorless, viscous liquid; (63 mg, 74%); R_f = 0.32 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.16 (s, 1H), 7.79–7.77 (m, 2H), 7.53–7.49 (m, 1H), 7.38 (t, *J* = 7.6 Hz, 2H), 7.34–7.31 (m, 3H), 7.25–7.23 (m, 2H), 7.00 (t, *J* = 7.6 Hz, 2H), 6.84 (t, *J* = 7.6 Hz, 1H), 6.54 (d, *J* = 7.6 Hz, 2H), 4.21 (t, *J* = 6.4 Hz, 2H), 2.99–2.95 (m, 2H), 2.54–2.50 (m, 2H), 1.72–1.65 (m, 2H), 1.47–1.38 (m, 2H), 0.92 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$):

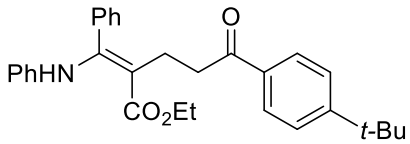
δ 200.0, 171.0, 157.9, 140.4, 136.7, 134.7, 132.7, 128.9, 128.8, 128.6, 128.45, 128.40, 128.0, 122.7, 122.1, 97.1, 63.6, 40.3, 30.9, 23.6, 19.3, 13.7; ESI-HRMS: m/z Calcd for $C_{28}H_{29}NO_3Na$, $[M + Na]^+$: 450.2040, found 450.2038.

Ethyl (Z)-5-oxo-2-(phenyl(phenylamino)methylene)-5-(p-tolyl)pentanoate (4b)



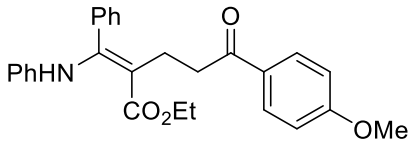
White solid; (65 mg, 79%); mp: 101–104 °C; R_f = 0.29 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.14 (s, 1H), 7.69 (d, *J* = 8.0 Hz, 2H), 7.34–7.32 (m, 3H), 7.26–7.23 (m, 2H), 7.18 (d, *J* = 8.0 Hz, 2H), 7.00 (t, *J* = 8.0 Hz, 2H), 6.84 (t, *J* = 7.2 Hz, 1H), 6.54 (d, *J* = 8.0 Hz, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 2.96–2.92 (m, 2H), 2.53–2.49 (m, 2H), 2.38 (s, 3H), 1.34 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.8, 171.0, 157.9, 143.4, 140.4, 134.7, 134.2, 129.1, 129.0, 128.8, 128.6, 128.5, 128.2, 122.7, 122.1, 97.2, 59.7, 40.3, 23.8, 21.6, 14.5; ESI-HRMS: m/z Calcd for $C_{27}H_{27}NO_3Na$, $[M + Na]^+$: 436.1889, found 436.1889.

Ethyl (Z)-5-(4-(tert-butyl)phenyl)-5-oxo-2-(phenyl(phenylamino)methylene)pentanoate (4c)



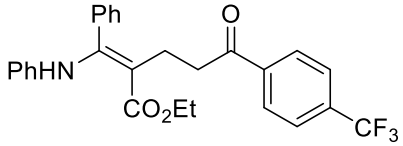
Red oil; (64 mg, 70%); R_f = 0.43 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.15 (s, 1H), 7.75 (d, *J* = 8.0 Hz, 2H), 7.40 (d, *J* = 8.0 Hz, 2H), 7.32 (brs, 3H), 7.26–7.24 (m, 2H), 7.00 (t, *J* = 7.6 Hz, 2H), 6.84 (t, *J* = 7.2 Hz, 1H), 6.54 (d, *J* = 8.0 Hz, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 2.95 (t, *J* = 7.6 Hz, 2H), 2.51 (t, *J* = 7.6 Hz, 2H), 1.35–1.33 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.8, 171.0, 157.8, 156.4, 140.4, 134.7, 134.2, 129.0, 128.8, 128.6, 128.5, 128.0, 125.3, 122.7, 122.1, 97.2, 59.7, 40.2, 35.0, 31.1, 23.7, 14.5; ESI-HRMS: m/z Calcd for $C_{30}H_{33}NO_3Na$ $[M + Na]^+$: 478.2362, found 478.2358.

Ethyl (Z)-5-(4-methoxyphenyl)-5-oxo-2-(phenyl(phenylamino)methylene)pentanoate (4d)



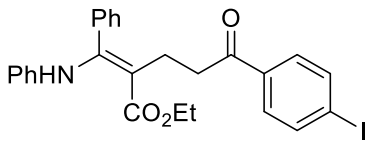
Reddish-brown oil; (79 mg, 92%); R_f = 0.27 (hexanes/ethyl acetate 5:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.14 (s, 1H), 7.76 (d, *J* = 8.8 Hz, 2H), 7.32 (t, *J* = 3.2 Hz, 3H), 7.25–7.23 (m, 2H), 6.99 (t, *J* = 7.6 Hz, 2H), 6.86–6.81 (m, 3H), 6.54 (d, *J* = 7.6 Hz, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 3.84 (s, 3H), 2.93–2.89 (m, 2H), 2.53–2.49 (m, 2H), 1.34 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.8, 170.9, 163.2, 157.8, 140.4, 134.7, 130.3, 129.8, 129.0, 128.8, 128.6, 128.4, 122.7, 122.1, 113.5, 97.2, 59.7, 55.4, 40.0, 23.9, 14.5; ESI-HRMS: m/z Calcd for $C_{27}H_{27}NO_4Na$, $[M + Na]^+$: 452.1838, found 452.1842.

Ethyl (Z)-5-oxo-2-(phenyl(phenylamino)methylene)-5-(4-(trifluoromethyl)phenyl)pentanoate (4e)



Yellow solid; (63 mg, 67%); mp: 77–80 °C; R_f = 0.41 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.16 (s, 1H), 7.86 (d, *J* = 8.0 Hz, 2H), 7.63 (d, *J* = 8.4 Hz, 2H), 7.34–7.30 (m, 3H), 7.24–7.22 (m, 2H), 7.00 (t, *J* = 8.0 Hz, 2H), 6.84 (t, *J* = 7.2 Hz, 1H), 6.55 (d, *J* = 8.0 Hz, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 2.99–2.95 (m, 2H), 2.56–2.52 (m, 2H), 1.34 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.6, 170.3, 157.6, 139.8, 138.8, 134.1, 133.5 (q, $^{C,F}J_2$ = 32 Hz), 128.5, 128.4, 128.2, 128.0, 127.9, 125.0 (q, $^{C,F}J_3$ = 3 Hz), 123.1 (q, $^{C,F}J_1$ = 271 Hz), 122.4, 121.8, 96.1, 59.3, 40.1, 23.2, 14.1; ESI-HRMS: m/z Calcd for $C_{27}H_{24}F_3NO_3Na$, $[M + Na]^+$: 490.1606, found 490.1608.

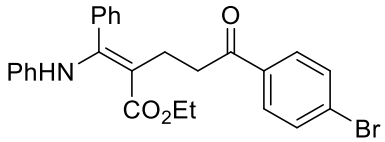
Ethyl (Z)-5-(4-iodophenyl)-5-oxo-2-(phenyl(phenylamino)methylene)pentanoate (4f)



Yellow solid; (93 mg, 89%); mp: 94–96 °C; R_f = 0.41 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.15 (s, 1H), 7.72 (d, *J* = 8.0 Hz, 2H), 7.46 (d, *J* = 8.4 Hz, 2H), 7.35–7.29 (m, 3H), 7.24–7.21 (m, 2H), 6.99 (t, *J* = 8.0 Hz, 2H), 6.83 (t, *J* = 7.6 Hz, 1H), 6.54 (d, *J* = 7.6 Hz, 2H), 4.26 (q, *J* = 7.2 Hz, 2H), 2.92–2.88 (m, 2H), 2.52–2.48 (m, 2H), 1.33 (t, *J*

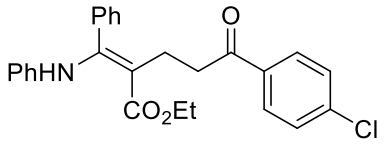
= 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.0, 170.4, 157.6, 139.9, 137.4, 135.5, 134.3, 129.2, 128.6, 128.5, 128.3, 128.1, 122.5, 121.8, 100.3, 96.4, 59.4, 39.9, 23.4, 14.2; ESI-HRMS: m/z Calcd for $C_{26}H_{24}INO_3Na$, $[M + Na]^+$: 548.0699, found 548.0705.

Ethyl (Z)-5-(4-bromophenyl)-5-oxo-2-(phenyl(phenylamino)methylene)pentanoate (4g)



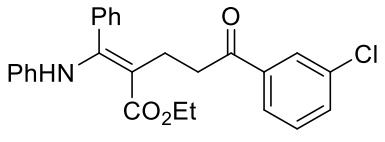
Yellow solid; (84 mg, 88%); mp: 63–67 °C; R_f = 0.41 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.15 (s, 1H), 7.62 (d, *J* = 8.4 Hz, 2H), 7.50 (d, *J* = 8.4 Hz, 2H), 7.33–7.32 (m, 3H), 7.24–7.23 (m, 2H), 6.99 (t, *J* = 8.0 Hz, 2H), 6.83 (t, *J* = 7.2 Hz, 1H), 6.54 (d, *J* = 8.0 Hz, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 2.93–2.89 (m, 2H), 2.53–2.49 (m, 2H), 1.33 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.0, 170.7, 157.9, 140.2, 135.3, 134.6, 131.6, 129.6, 128.9, 128.8, 128.6, 128.4, 127.8, 122.8, 122.1, 96.7, 59.7, 40.2, 23.8, 14.5; ESI-HRMS: m/z Calcd for $C_{26}H_{24}BrNO_3Na$, $[M + Na]^+$: 500.0837, found 500.0844.

Ethyl (Z)-5-(4-chlorophenyl)-5-oxo-2-(phenyl(phenylamino)methylene)pentanoate (4h)



Grayish-brown solid; (81 mg, 93%); mp: 74–76 °C; R_f = 0.41 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.15 (s, 1H), 7.69 (dt, *J* = 2.0 Hz, *J* = 8.8 Hz, 2H), 7.35–7.31 (m, 5H), 7.25–7.22 (m, 2H), 7.00 (t, *J* = 7.6 Hz, 2H), 6.84 (t, *J* = 7.6 Hz, 1H), 6.54 (d, *J* = 8.0 Hz, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 2.94–2.90 (m, 2H), 2.53–2.49 (m, 2H), 1.33 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.8, 170.8, 157.9, 140.2, 139.1, 134.9, 134.6, 129.5, 128.9, 128.8, 128.7, 128.6, 128.4, 122.8, 122.1, 96.7, 59.7, 40.3, 23.8, 14.5; ESI-HRMS: m/z Calcd for $C_{26}H_{24}ClNO_3Na$, $[M + Na]^+$: 456.1342, found 456.1346.

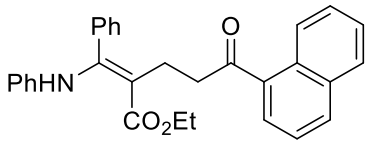
Ethyl (Z)-5-(3-chlorophenyl)-5-oxo-2-(phenyl(phenylamino)methylene)pentanoate (4i)



Reddish-brown oil; (75 mg, 87%); R_f = 0.41 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.16 (s, 1H), 7.82 (brs, 1H), 7.64 (d, *J* = 7.6 Hz, 1H), 7.48–7.45 (m, 1H),

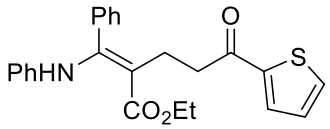
7.32–7.31 (m, 4H), 7.25–7.21 (m, 2H), 7.00 (t, *J* = 8.0 Hz, 2H), 6.83 (t, *J* = 7.6 Hz, 1H), 6.54 (d, *J* = 8.0 Hz, 2H), 4.28 (q, *J* = 7.2 Hz, 2H), 2.96–2.92 (m, 2H), 2.53–2.49 (m, 2H), 1.34 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.7, 170.7, 158.0, 140.2, 138.2, 134.7, 134.5, 132.6, 129.7, 128.8, 128.6, 128.4, 128.1, 126.1, 122.8, 122.1, 96.6, 59.7, 40.3, 23.7, 14.5; ESI-HRMS: m/z Calcd for $C_{26}H_{24}ClNO_3Na$, $[M + Na]^+$: 456.1342, found 456.1347.

Ethyl (Z)-5-(naphthalen-1-yl)-5-oxo-2-(phenyl(phenylamino)methylene)pentanoate (4j)



Yellowish-brown oil; (52 mg, 58%); R_f = 0.36 (hexanes/ethyl acetate 5:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.12 (s, 1H), 8.48 (d, *J* = 8.8 Hz, 1H), 7.93 (d, *J* = 8.0 Hz, 1H), 7.85–7.83 (m, 1H), 7.61 (dd, *J* = 1.2 Hz, *J* = 7.2 Hz, 1H), 7.55–7.47 (m, 2H), 7.39 (t, *J* = 7.2 Hz, 1H), 7.33–7.28 (m, 3H), 7.23–7.20 (m, 2H), 6.99 (t, *J* = 8.0 Hz, 2H), 6.83 (t, *J* = 7.2 Hz, 1H), 6.53 (d, *J* = 8.0 Hz, 2H), 4.26 (q, *J* = 7.2 Hz, 2H), 3.08–3.04 (m, 2H), 2.59–2.55 (m, 2H), 1.33 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 204.5, 170.9, 157.9, 140.4, 135.8, 134.7, 133.9, 132.2, 130.2, 128.9, 128.8, 128.6, 128.5, 128.3, 127.7, 127.4, 126.3, 125.8, 124.3, 122.7, 122.1, 97.0, 59.7, 43.7, 23.9, 14.5; ESI-HRMS: m/z Calcd for $C_{30}H_{27}NO_3Na$, $[M + Na]^+$: 472.1889, found 472.1893.

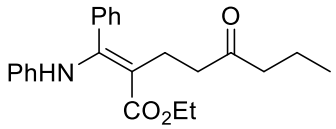
Ethyl (Z)-5-oxo-2-(phenyl(phenylamino)methylene)-5-(thiophen-2-yl)pentanoate (4k)



Yellowish-brown oil; (70 mg, 86%); R_f = 0.39 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.15 (s, 1H), 7.54 (dd, *J* = 1.2 Hz, *J* = 4.8 Hz, 1H), 7.43 (dd, *J* = 1.2 Hz, *J* = 3.6 Hz, 1H), 7.33–7.30 (m, 3H), 7.25–7.22 (m, 2H), 7.03 (dd, *J* = 3.6 Hz, *J* = 4.8 Hz, 1H), 7.02–6.96 (m, 2H), 6.85–6.81 (m, 1H), 6.54 (d, *J* = 7.6 Hz, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 2.91–2.87 (m, 2H), 2.56–2.52 (m, 2H), 1.33 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.0, 170.8, 157.9, 144.3, 140.3, 134.6, 133.1, 131.6, 128.9, 128.8, 128.6, 128.4, 127.8, 122.7, 122.1, 96.8, 59.7, 41.0, 24.0, 14.5; ESI-HRMS: m/z Calcd for $C_{24}H_{23}NO_3SNa$,

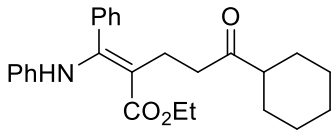
[M + Na]$^+$: 428.1296, found 428.1300.

Ethyl (Z)-5-oxo-2-(phenyl(phenylamino)methylene)octanoate (4l)



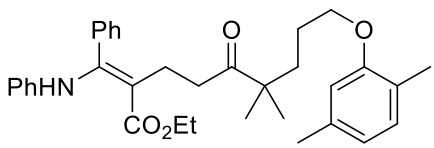
Reddish-brown oil; (34 mg, 46%); R_f = 0.42 (hexanes/ethyl acetate 5:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.08 (s, 1H), 7.32 (t, *J* = 3.2 Hz, 3H), 7.22–7.20 (m, 2H), 6.99 (t, *J* = 8.0 Hz, 2H), 6.83 (t, *J* = 7.2 Hz, 1H), 6.52 (d, *J* = 8.0 Hz, 2H), 4.25 (q, *J* = 7.2 Hz, 2H), 2.44–2.39 (m, 2H), 2.35–2.31 (m, 2H), 2.20 (t, *J* = 7.2 Hz, 2H), 1.49 (sex, *J* = 3.2 Hz, 2H), 1.33 (t, *J* = 7.2 Hz, 3H), 0.84 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 211.2, 170.9, 157.7, 140.4, 134.8, 128.9, 128.8, 128.5, 128.4, 122.6, 122.0, 97.2, 59.6, 44.4, 44.1, 22.6, 17.3, 14.5, 13.7; ESI-HRMS: m/z Calcd for $C_{23}H_{27}NO_3Na$, [M + Na]$^+$: 388.1889, found 388.1893.

Ethyl (Z)-5-cyclohexyl-5-oxo-2-(phenyl(phenylamino)methylene)pentanoate (4m)



Reddish-brown oil; (53 mg, 65%); R_f = 0.39 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.07 (s, 1H), 7.32–7.31 (m, 3H), 7.22–7.21 (m, 2H), 6.99 (t, *J* = 8.0 Hz, 2H), 6.82 (t, *J* = 7.2 Hz, 1H), 6.52 (d, *J* = 7.6 Hz, 2H), 4.25 (q, *J* = 7.2 Hz, 2H), 2.46–2.42 (m, 2H), 2.32–2.29 (m, 2H), 2.16 (brs, 1H), 1.70–1.64 (m, 5H), 1.33 (t, *J* = 7.2 Hz, 3H), 1.22–1.15 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 213.9, 170.9, 157.6, 140.4, 134.8, 128.9, 128.8, 128.5, 128.4, 122.6, 122.0, 97.4, 59.6, 50.4, 41.9, 28.3, 25.8, 25.6, 22.6, 14.5; ESI-HRMS: m/z Calcd for $C_{26}H_{31}NO_3Na$, [M + Na]$^+$: 428.2202, found 428.2205.

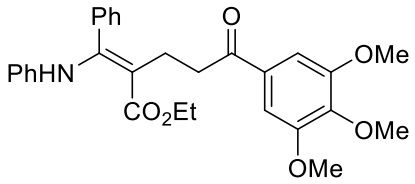
Ethyl (Z)-9-(2,5-dimethylphenoxy)-6,6-dimethyl-5-oxo-2-(phenyl(phenylamino)methylene)nonanoate (4n)



White solid; (50 mg, 47%); mp: 85–87 °C; R_f = 0.25 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.09 (s, 1H), 7.31–7.25 (m, 3H), 7.21–7.19 (m, 2H), 7.01–6.96 (m, 3H), 6.82 (t, *J* = 7.2 Hz, 1H), 6.66 (d, *J* = 7.2 Hz, 1H), 6.59 (s, 1H), 6.52 (d, *J* = 8.0 Hz, 2H),

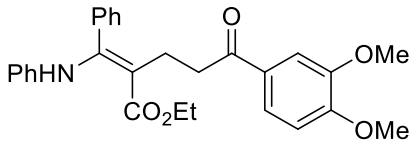
4.24 (q, *J* = 7.2 Hz, 2H), 3.85 (t, *J* = 6.0 Hz, 2H), 2.53–2.49 (m, 2H), 2.33–2.28 (m, 5H), 2.17 (s, 3H), 1.62–1.59 (m, 2H), 1.56–1.51 (m, 2H), 1.31 (t, *J* = 7.2 Hz, 3H), 1.04 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): *δ* 214.8, 170.5, 157.2, 156.4, 140.0, 136.0, 134.3, 129.9, 128.4, 128.3, 128.1, 128.0, 123.0, 122.2, 121.6, 120.3, 111.4, 96.9, 67.4, 59.2, 46.7, 37.7, 35.9, 24.5, 23.7, 22.4, 21.0, 15.3, 14.1; ESI-HRMS: m/z Calcd for $C_{34}H_{41}NO_4Na$, $[M + Na]^+$: 550.2933, found 550.2935.

Ethyl (Z)-5-oxo-2-(phenyl(phenylamino)methylene)-5-(3,4,5-trimethoxyphenyl)pentanoate (4o)



Yellow oil; (76 mg, 78%); R_f = 0.12 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): *δ* 11.13 (s, 1H), 7.29 (t, *J* = 3.2 Hz, 3H), 7.26–7.20 (m, 2H), 7.14 (s, 2H), 6.99 (t, *J* = 8.0 Hz, 2H), 6.83 (t, *J* = 7.2 Hz, 1H), 6.53 (d, *J* = 8.0 Hz, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 3.90 (s, 3H), 3.88 (s, 6H), 2.97 (t, *J* = 8.0 Hz, 2H), 2.54 (t, *J* = 8.0 Hz, 2H), 1.32 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): *δ* 198.6, 170.8, 157.8, 152.9, 142.4, 140.3, 134.6, 132.2, 128.9, 128.8, 128.5, 128.4, 122.7, 122.1, 105.6, 97.1, 60.8, 59.6, 56.2, 39.8, 23.4, 14.5; ESI-HRMS: m/z Calcd for $C_{29}H_{31}NO_6Na$, $[M + Na]^+$: 512.2049, found 512.2055.

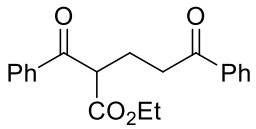
Ethyl (Z)-5-(3,4-dimethoxyphenyl)-5-oxo-2-(phenyl(phenylamino)methylene)pentanoate (4p)



Red solid; (63 mg, 69%); mp: 126–128 °C; R_f = 0.19 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): *δ* 11.15 (s, 1H), 7.45 (d, *J* = 2.0 Hz, 1H), 7.38 (dd, *J* = 1.6 Hz, *J* = 8.4 Hz, 1H), 7.32–7.31 (m, 3H), 7.26–7.22 (m, 2H), 6.99 (t, *J* = 8.0 Hz, 2H), 6.83 (t, *J* = 7.2 Hz, 1H), 6.79 (d, *J* = 8.4 Hz, 1H), 6.54 (d, *J* = 7.6 Hz, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 3.92 (s, 3H), 3.89 (s, 3H), 2.96–2.92 (m, 2H), 2.54–2.50 (m, 2H), 1.34 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): *δ* 198.7, 170.9, 157.7, 152.9, 148.9, 140.3, 134.7, 130.0, 128.9, 128.7, 128.5, 128.4, 122.7, 122.6, 122.0, 110.0, 109.7, 97.2, 59.6, 55.9, 55.8, 39.8, 23.9, 14.5;

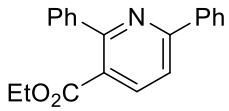
ESI-HRMS: m/z Calcd for $C_{28}H_{29}NO_5Na$, $[M + Na]^+$: 482.1943, found 482.1945.

Ethyl 2-benzoyl-5-oxo-5-phenylpentanoate (5)



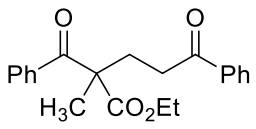
Yellow oil; (739 mg, 93%); R_f = 0.18 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.09–8.07 (m, 2H), 7.97–7.94 (m, 2H), 7.61–7.54 (m, 2H), 7.51–7.43 (m, 4H), 4.58 (dd, *J* = 2.4 Hz, *J* = 6.4 Hz, 1H), 4.21–4.09 (m, 2H), 3.24–3.06 (m, 2H), 2.51–2.35 (m, 2H), 1.16 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.2, 195.3, 169.8, 136.6, 135.8, 133.6, 133.2, 128.71, 128.69, 128.6, 127.9, 61.4, 52.8, 35.6, 23.2, 13.9; ESI-HRMS: m/z Calcd for $C_{20}H_{20}O_4Na$, $[M + Na]^+$: 347.1259, found 347.1264.

Ethyl 2,6-diphenylnicotinate (6)



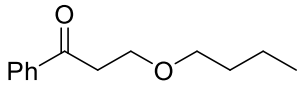
Yellow oil; (42 mg, 70%); R_f = 0.51 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.17 (d, *J* = 8.0 Hz, 1H), 8.14–8.11 (m, 2H), 7.77 (d, *J* = 8.0 Hz, 1H), 7.65–7.63 (m, 2H), 7.50–7.42 (m, 6H), 4.17 (q, *J* = 7.2 Hz, 2H), 1.07 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.3, 158.7, 158.4, 140.5, 138.8, 138.3, 129.7, 128.81, 128.75, 128.6, 128.0, 127.3, 125.3, 117.8, 61.3, 13.6; ESI-HRMS: m/z Calcd for $C_{20}H_{18}NO_2$, $[M + H]^+$: 304.1338, found 304.1343.

Ethyl 2-benzoyl-2-methyl-5-oxo-5-phenylpentanoate (7)



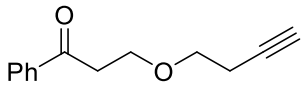
Colorless oil; (55 mg, 82%); R_f = 0.31 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92–7.90 (m, 2H), 7.87 (d, *J* = 7.2 Hz, 2H), 7.53 (dt, *J* = 1.2 Hz, *J* = 7.6 Hz, 2H), 7.42 (t, *J* = 8.0 Hz, 4H), 4.18–4.06 (m, 2H), 3.09–2.90 (m, 2H), 2.56–2.42 (m, 2H), 1.61 (s, 3H), 1.04 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.9, 197.2, 173.8, 136.5, 135.3, 133.0, 132.7, 128.45, 128.44, 128.43, 127.9, 61.4, 56.2, 33.5, 30.7, 21.4, 13.6; ESI-HRMS: m/z Calcd for $C_{21}H_{22}O_4Na$, $[M + Na]^+$: 361.1416, found 361.1421.

3-Butoxy-1-phenylpropan-1-one (8a)



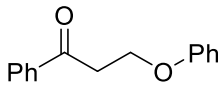
Colourless oil; (21 mg, 52%); R_f = 0.59 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97 (d, *J* = 7.6 Hz, 2H), 7.56 (t, *J* = 7.2 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 3.85 (t, *J* = 6.4 Hz, 2H), 3.47 (t, *J* = 6.4 Hz, 2H), 3.25 (t, *J* = 6.4 Hz, 2H), 1.58–1.51 (m, 2H), 1.39–1.30 (m, 2H), 0.90 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.5, 137.1, 133.0, 128.5, 128.1, 71.0, 66.0, 38.9, 31.7, 19.2, 13.8; ESI-HRMS: m/z Calcd for $C_{13}H_{19}O_2$, $[M + H]^+$: 207.1380, found 207.1382.

3-(But-3-yn-1-yloxy)-1-phenylpropan-1-one (8b)

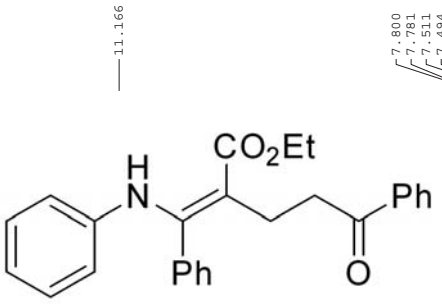


Yellow oil; (12 mg, 29%); R_f = 0.35 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97–7.95 (m, 2H), 7.57 (t, *J* = 7.2 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 3.92 (t, *J* = 6.8 Hz, 2H), 3.62 (t, *J* = 6.8 Hz, 2H), 3.27 (t, *J* = 6.8 Hz, 2H), 2.48–2.44 (m, 2H), 1.97 (t, *J* = 2.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.2, 137.0, 133.2, 128.6, 128.1, 81.2, 69.3, 69.2, 66.2, 38.8, 19.8; ESI-HRMS: m/z Calcd for $C_{13}H_{15}O_2$, $[M + H]^+$: 203.1067, found 203.1068.

3-Phenoxy-1-phenylpropan-1-one (8c)



White solid; (13 mg, 28%); mp: 55–59 ^{o}C; R_f = 0.36 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01–7.99 (m, 2H), 7.60–7.57 (m, 1H), 7.48 (t, *J* = 7.8 Hz, 2H), 7.30–7.25 (m, 2H), 6.97–6.92 (m, 3H), 4.43 (t, *J* = 6.8 Hz, 2H), 3.47 (t, *J* = 6.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.6, 158.6, 136.8, 133.3, 129.5, 128.6, 128.1, 120.9, 114.6, 63.2, 38.2; ESI-HRMS: m/z Calcd for $C_{15}H_{15}O_2$, $[M + H]^+$: 227.1067, found 227.1070.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Ph
O

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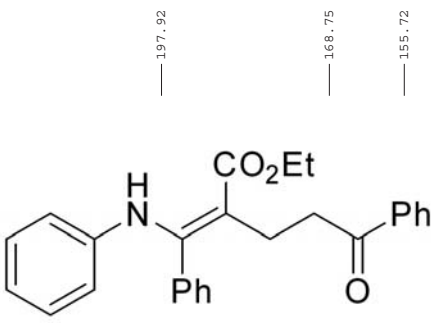
3.06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Ph
Ph
O

3a

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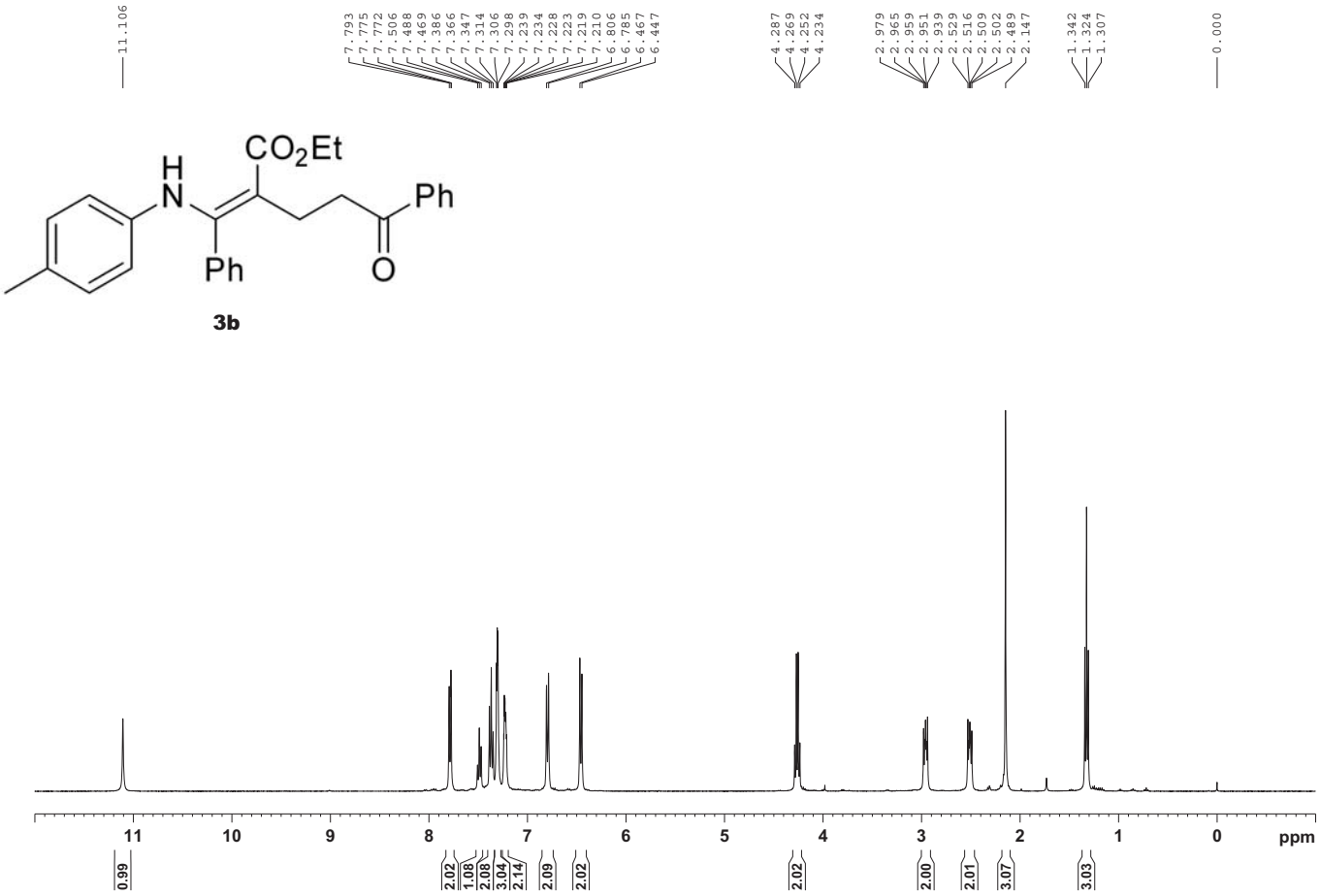
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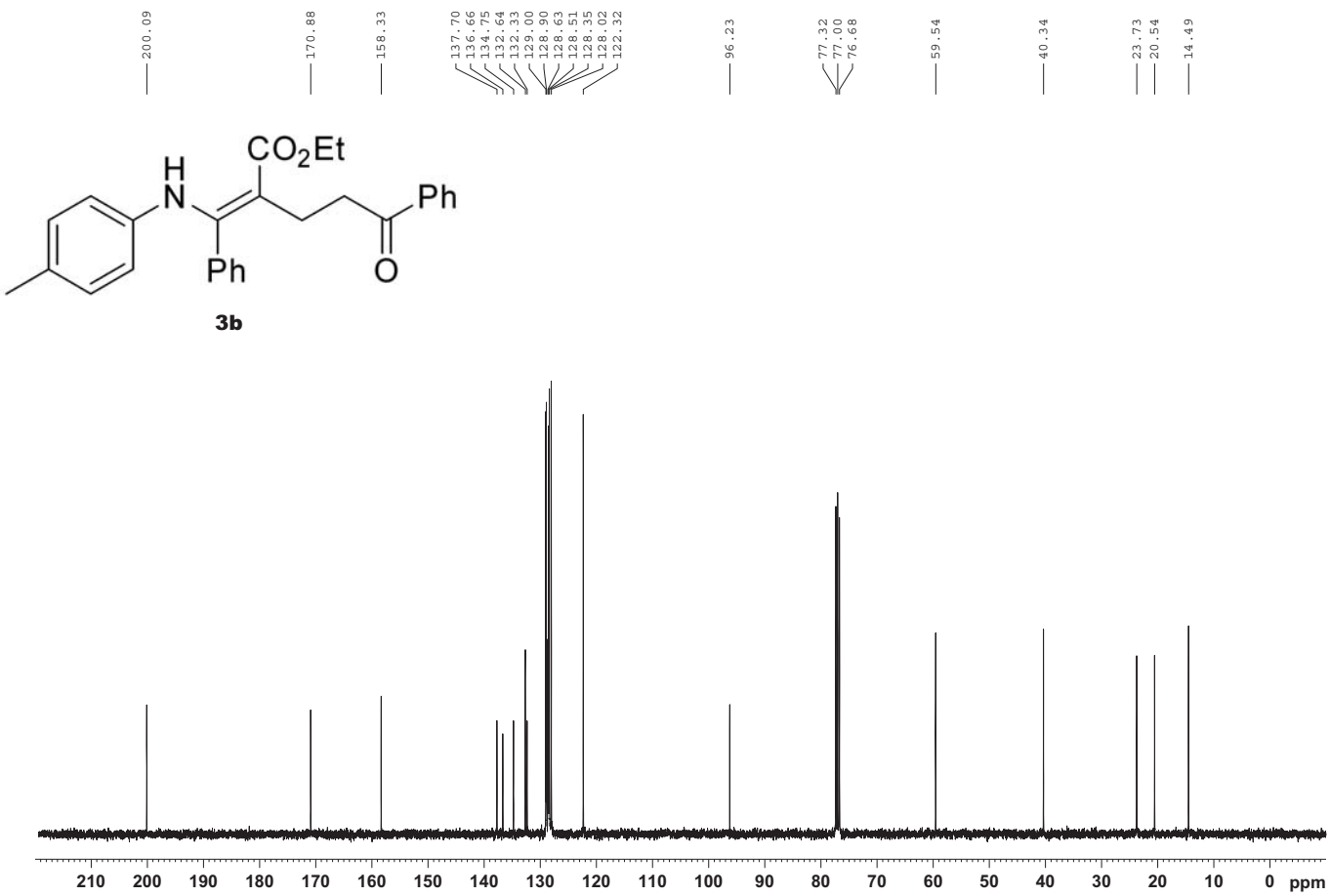
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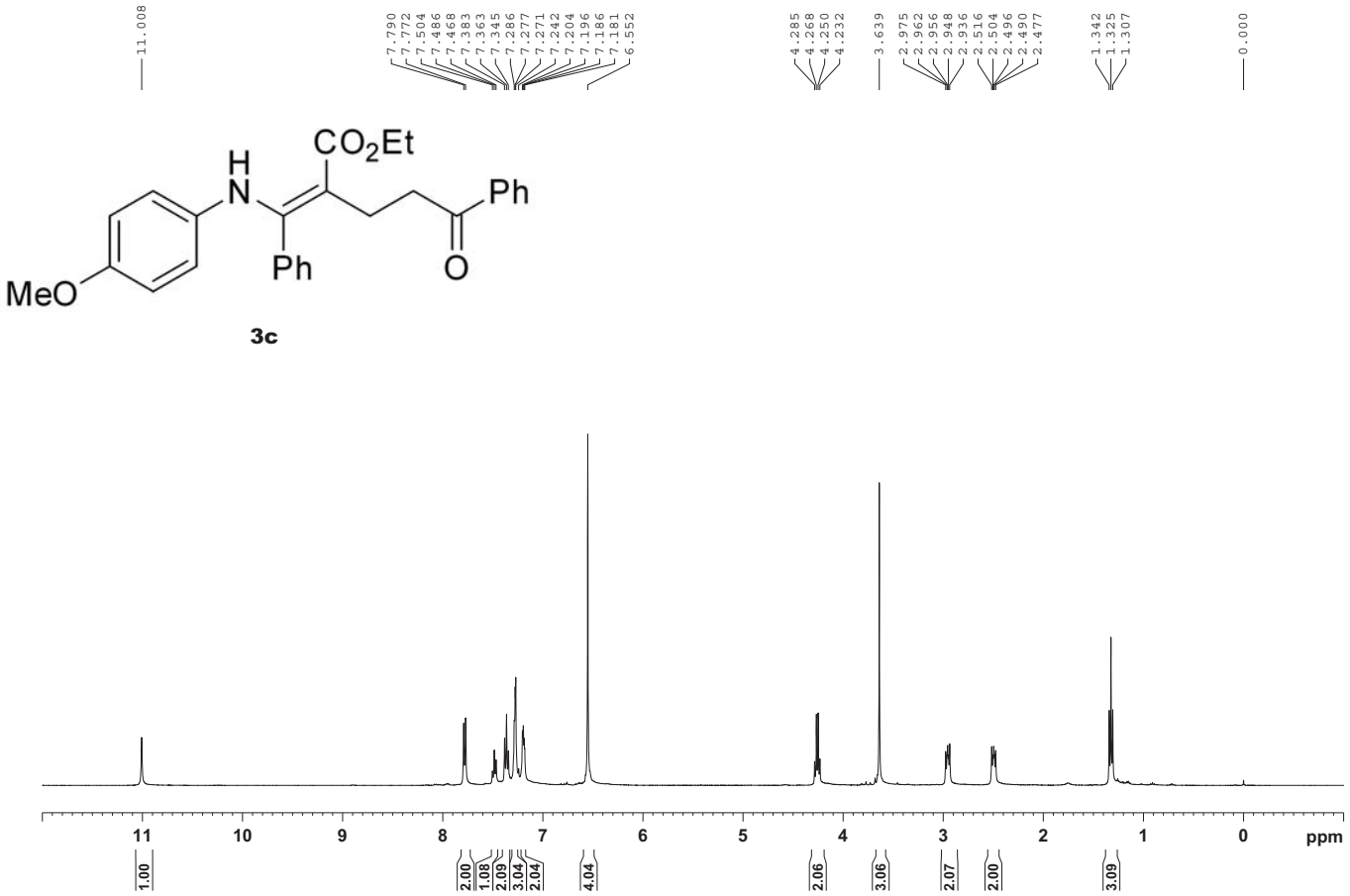
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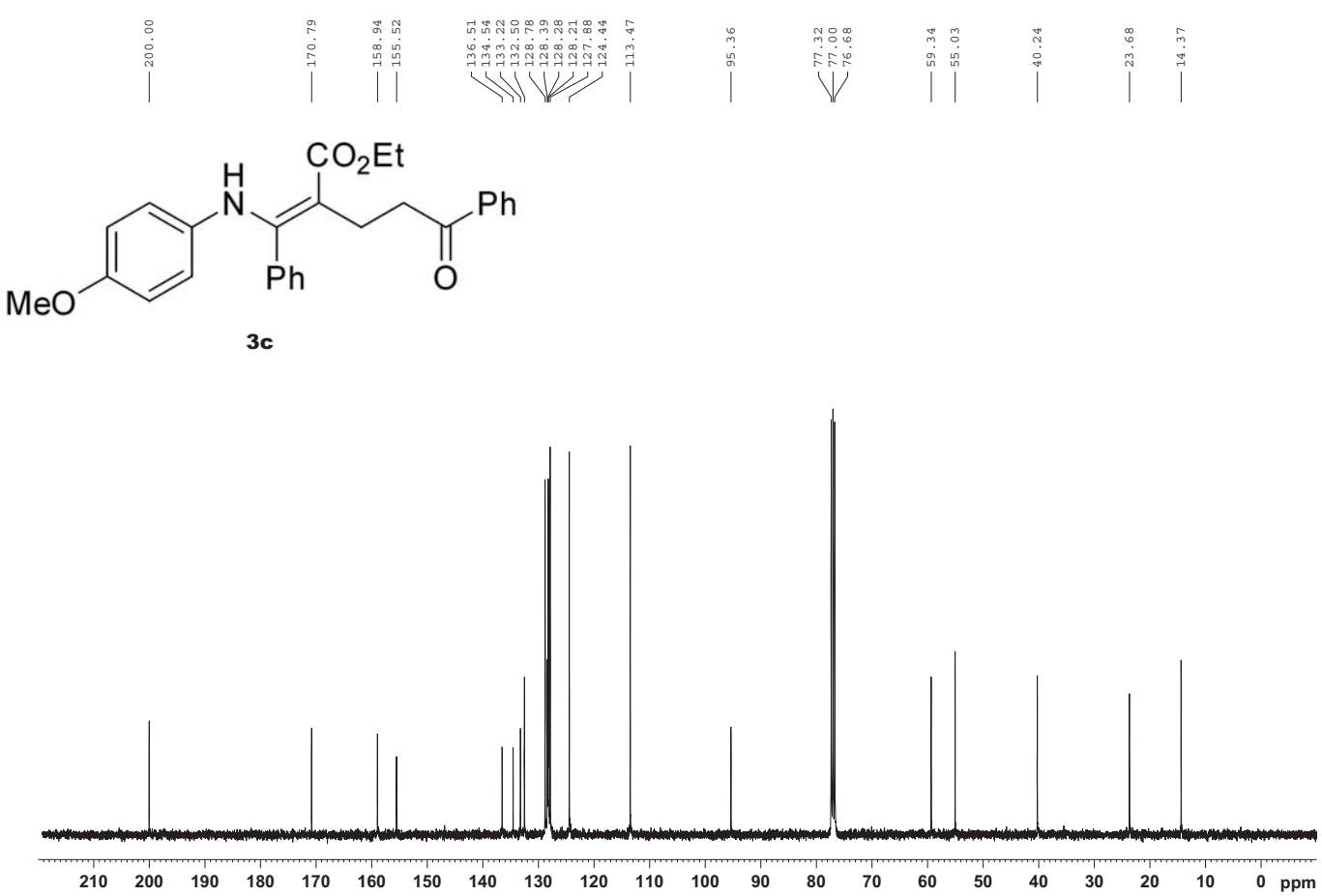
3c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MeO
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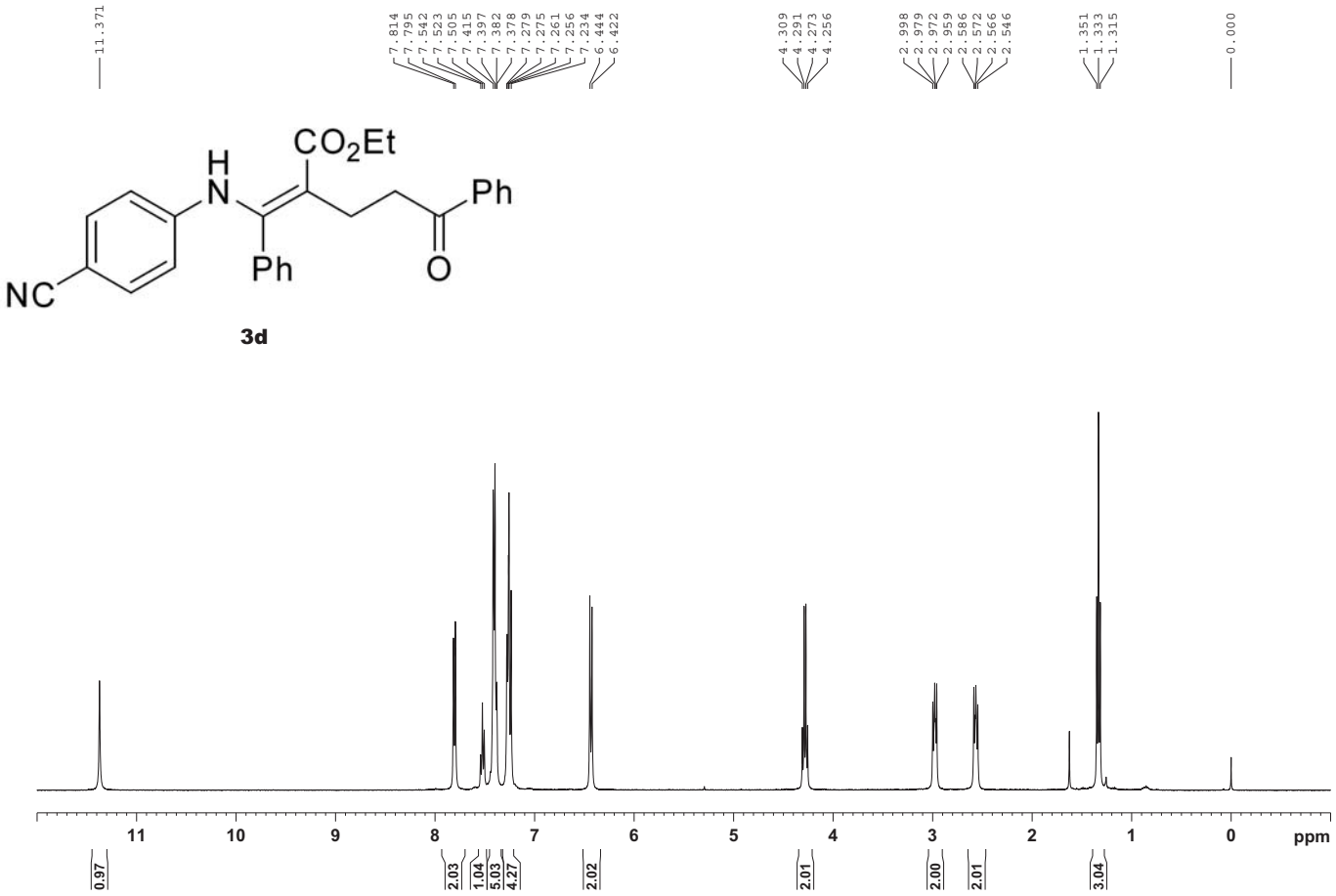
3c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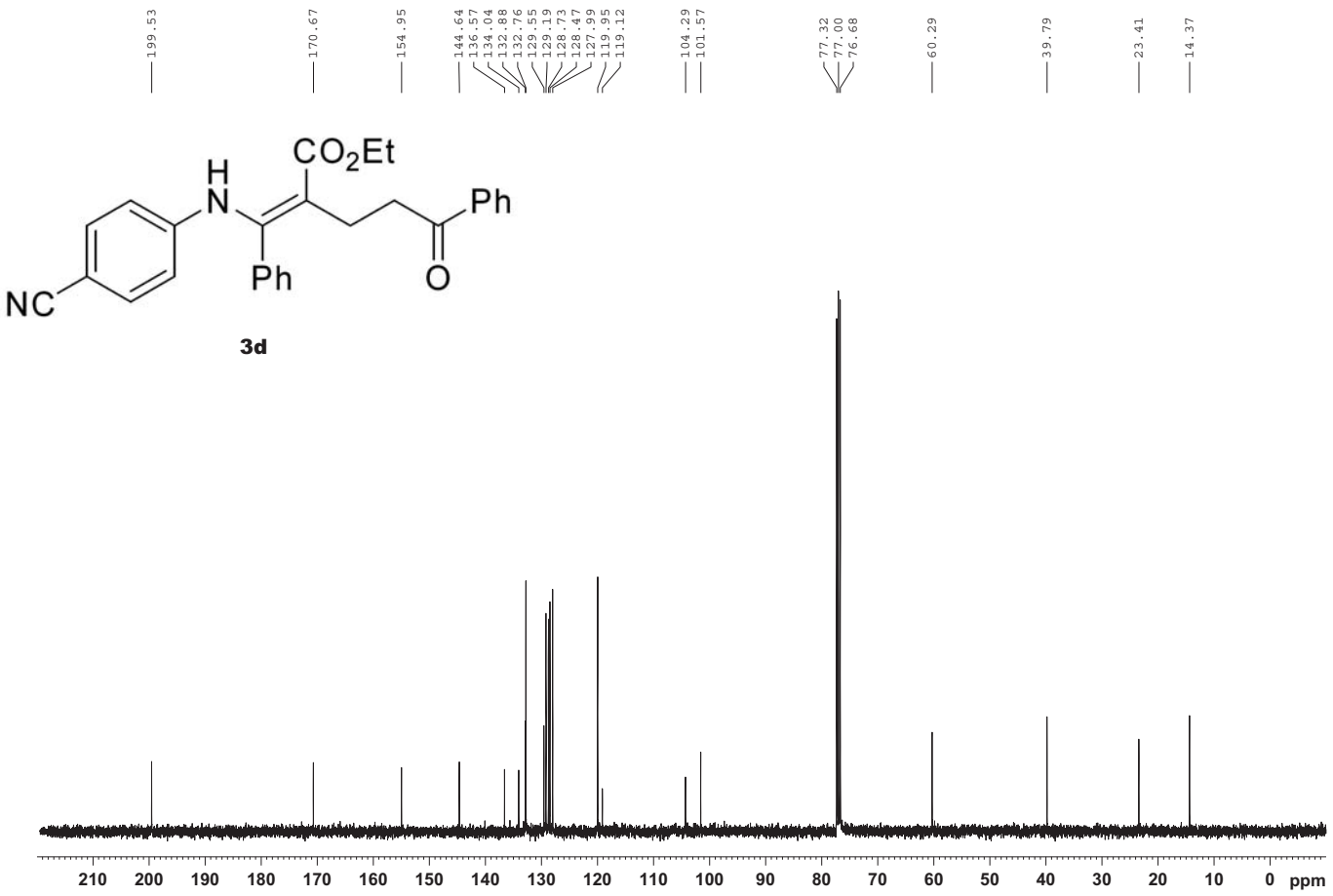
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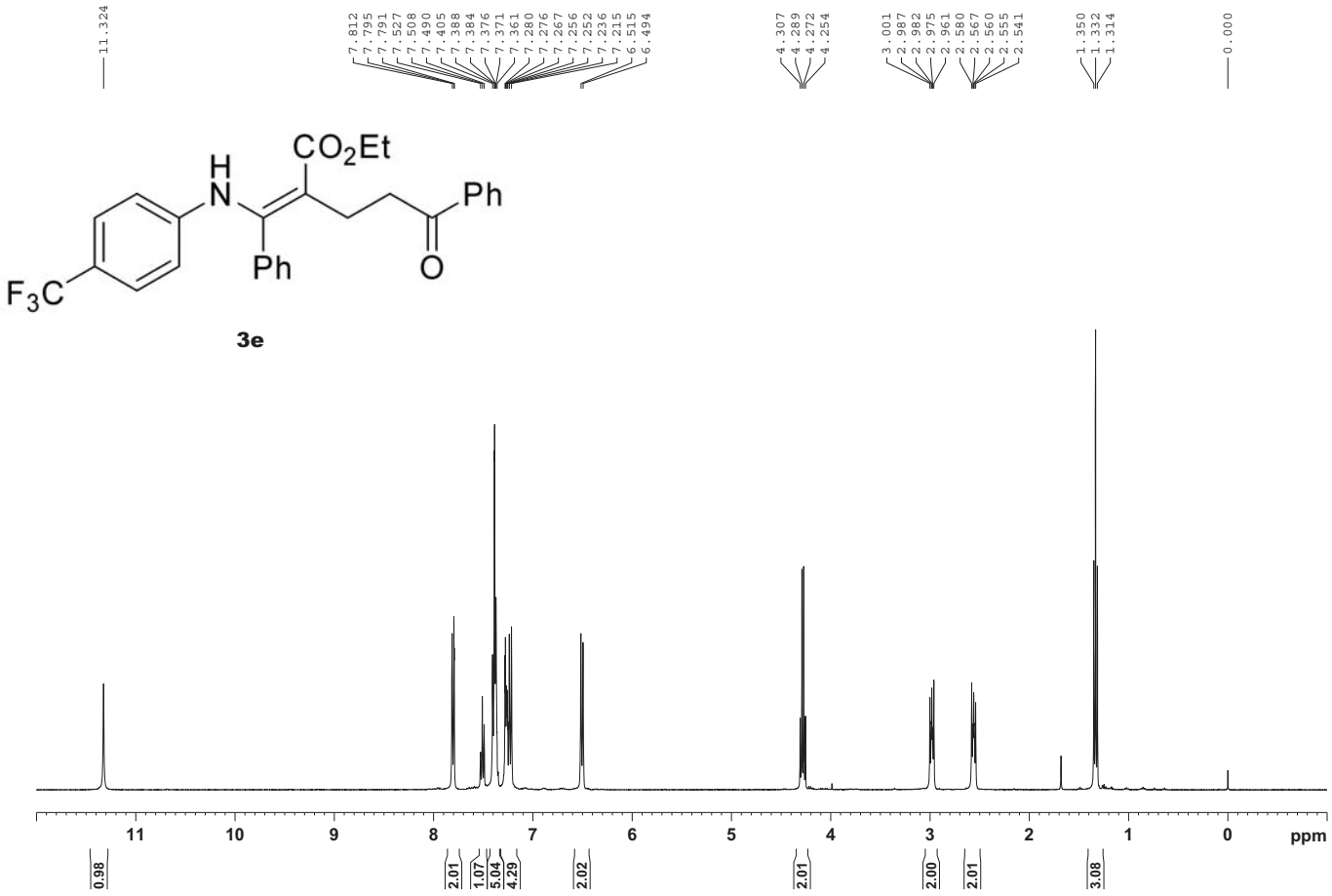
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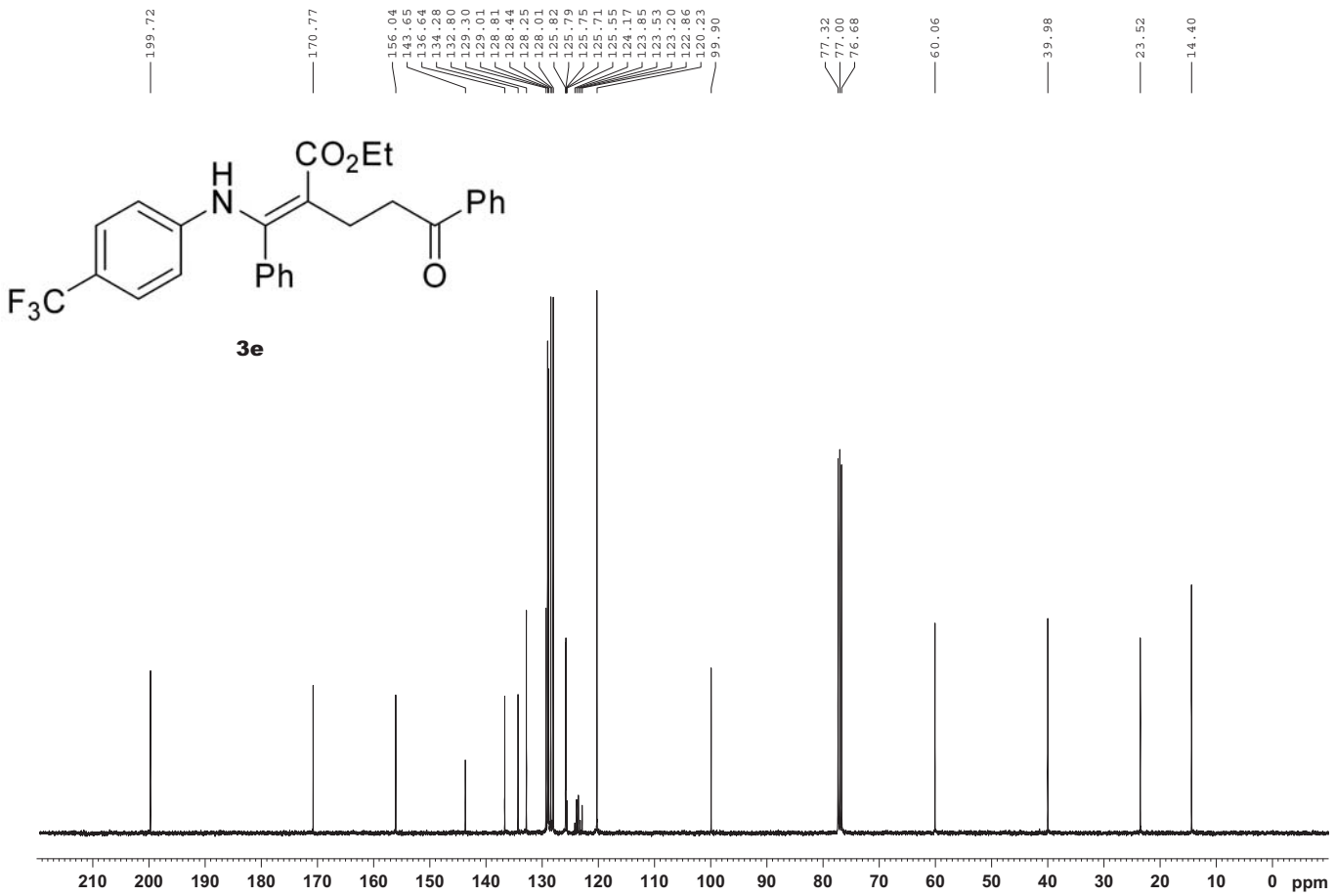
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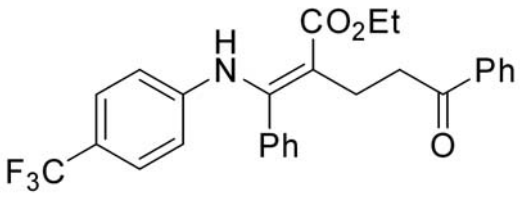
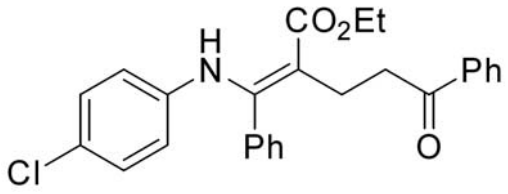
132.80
129.30
129.01
128.81
128.44
128.25
128.01
125.82
125.79
125.75
125.71
125.55
124.17
123.85
123.53
123.20
122.86
120.23
99.90
77.32
77.00
76.68
60.06
39.98
23.52
14.40
CO2Et
H
N
Ph
Ph
O
F3C
3e
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


H
N
CO2Et
Ph
Ph
O
F3C

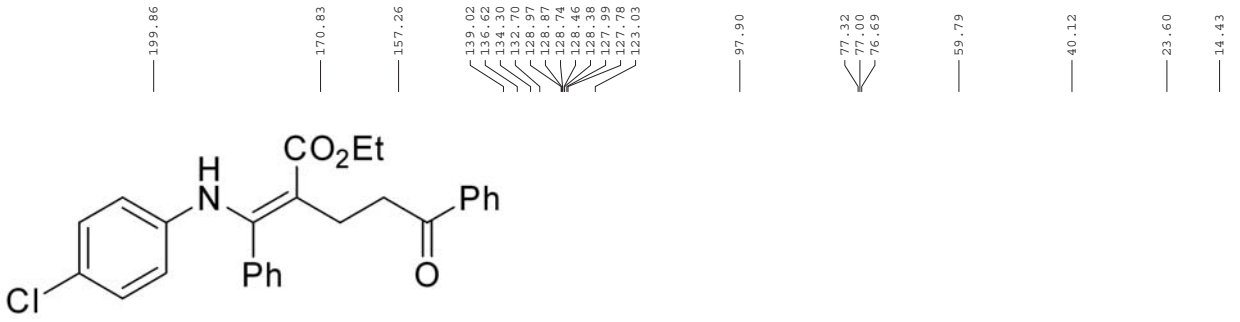
3e

-63.08
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
ppm

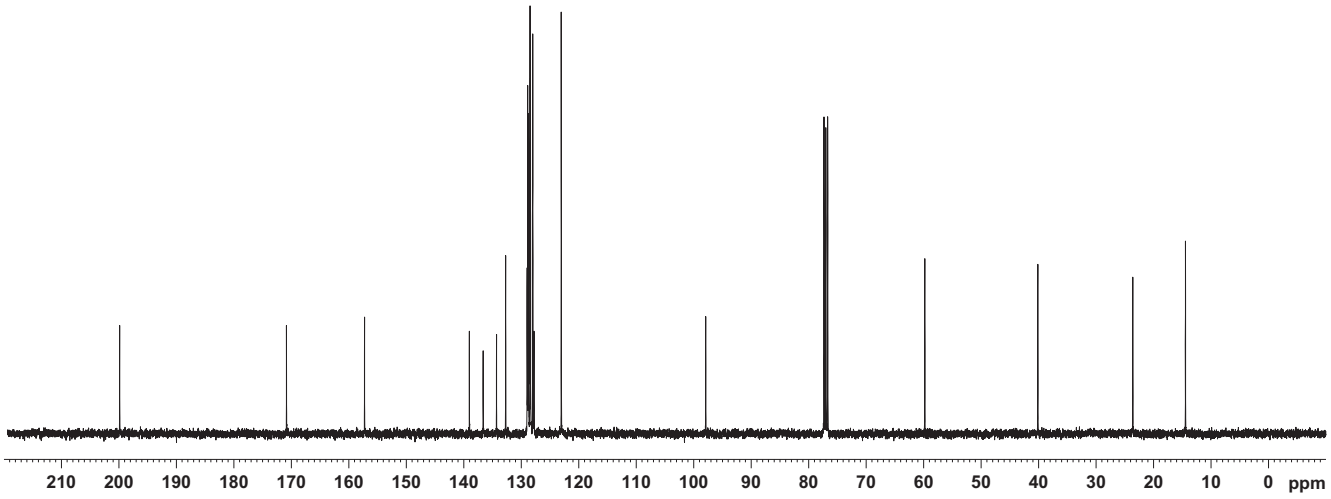

H
N
CO2Et
Ph
Ph
O
Cl

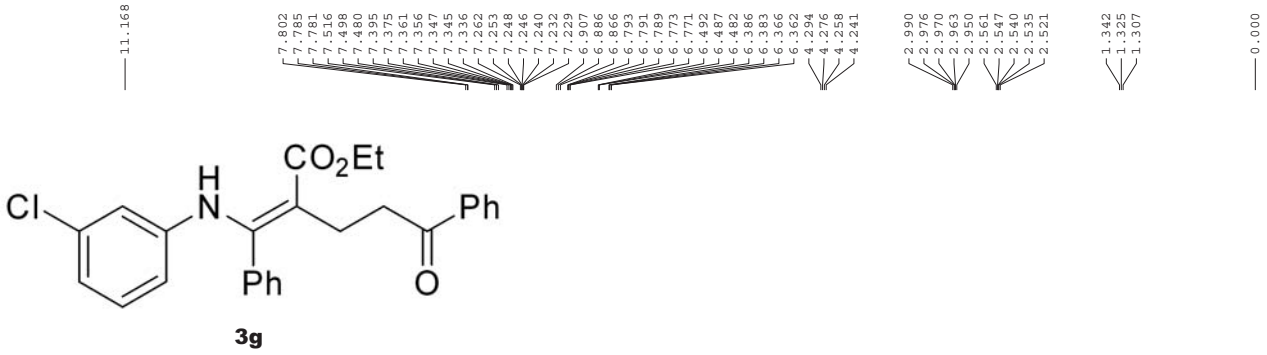
3f

11.137
7.800
7.781
7.515
7.496
7.478
7.393
7.374
7.354
7.338
7.333
7.324
7.250
7.231
7.226
7.217
7.208
6.955
6.933
6.460
6.438
4.291
4.273
4.255
4.238
2.983
2.964
2.956
2.944
2.548
2.535
2.528
2.508
1.342
1.324
1.306
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.98
2.03
1.05
5.06
2.05
2.01
2.00
2.05
2.00
2.01
3.02

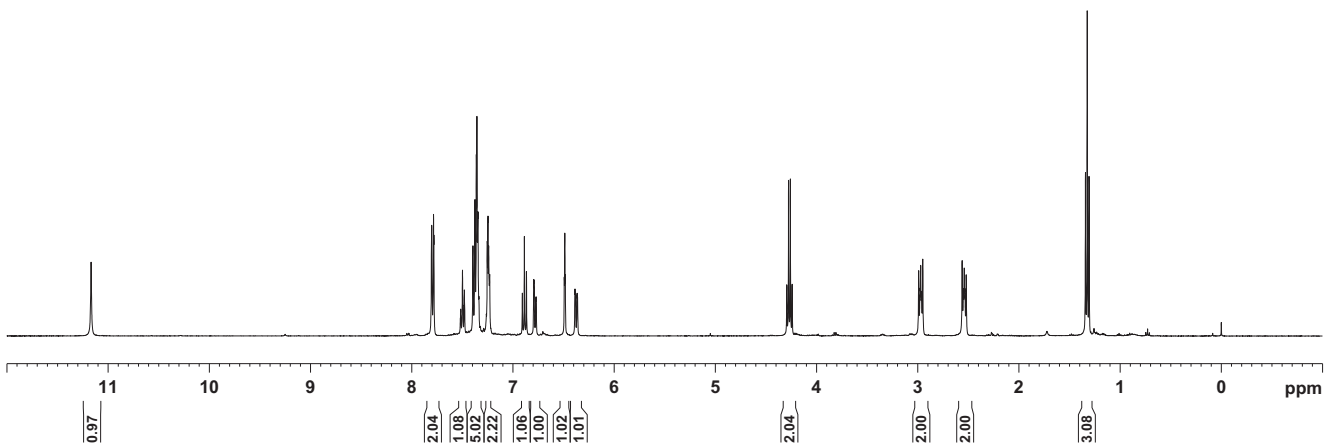

199.86
170.83
157.26
139.02
136.62
134.30
132.70
128.97
128.87
128.74
128.46
128.38
127.99
127.78
123.03
97.90
77.32
77.00
76.69
59.79
40.12
23.60
14.43
CO2Et
H
N
Ph
Ph
O
Cl

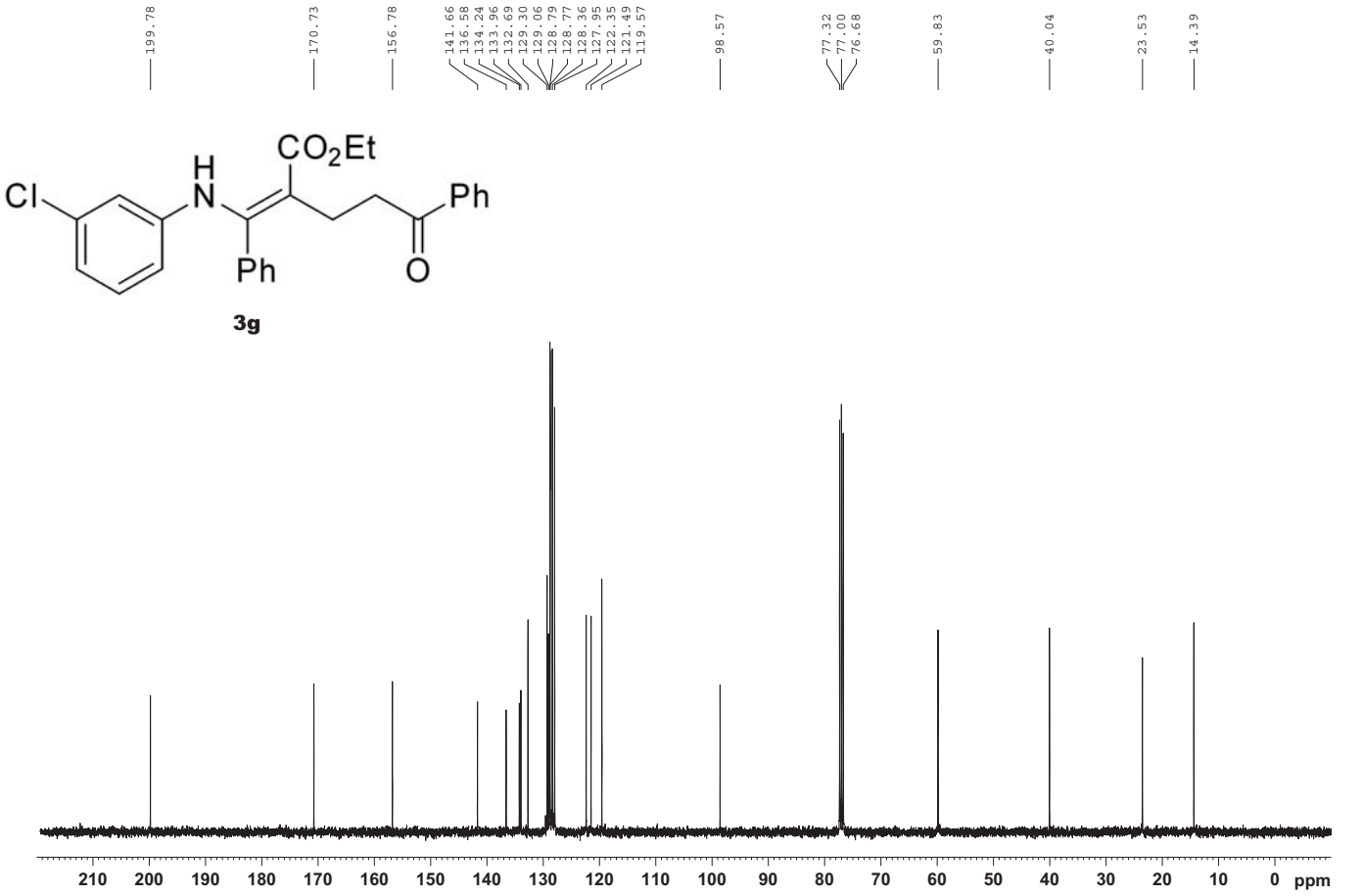
3f


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

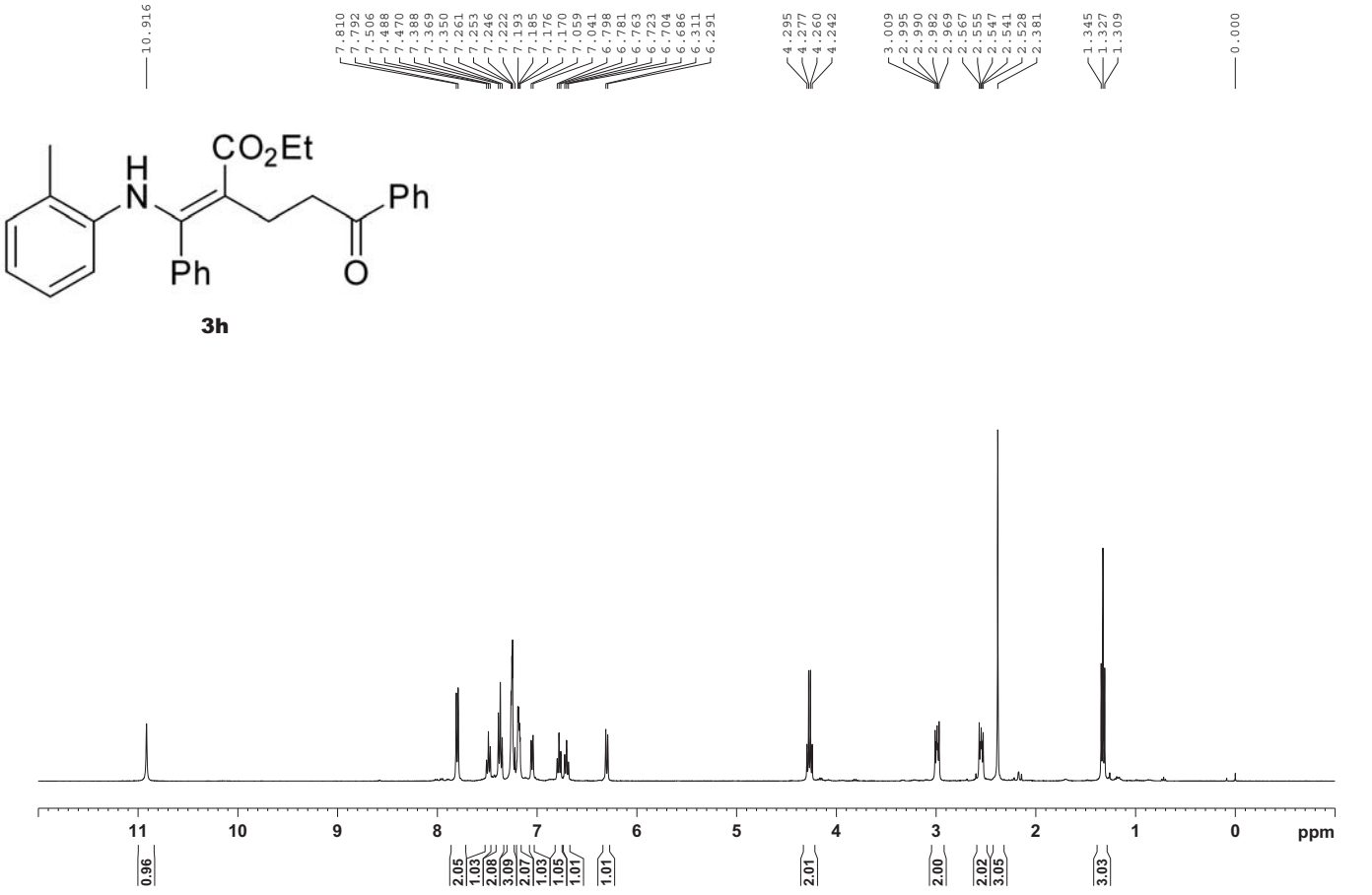

11.168
7.802
7.785
7.781
7.516
7.498
7.480
7.395
7.375
7.361
7.356
7.347
7.345
7.336
7.262
7.253
7.248
7.246
7.240
7.232
7.229
6.907
6.886
6.866
6.793
6.791
6.789
6.773
6.771
6.492
6.487
6.482
6.386
6.383
6.366
6.362
4.294
4.276
4.258
4.241
2.990
2.976
2.970
2.963
2.950
2.561
2.547
2.540
2.535
2.521
1.342
1.325
1.307
0.000
CO2Et
H
N
Cl
Ph
Ph
O

3g

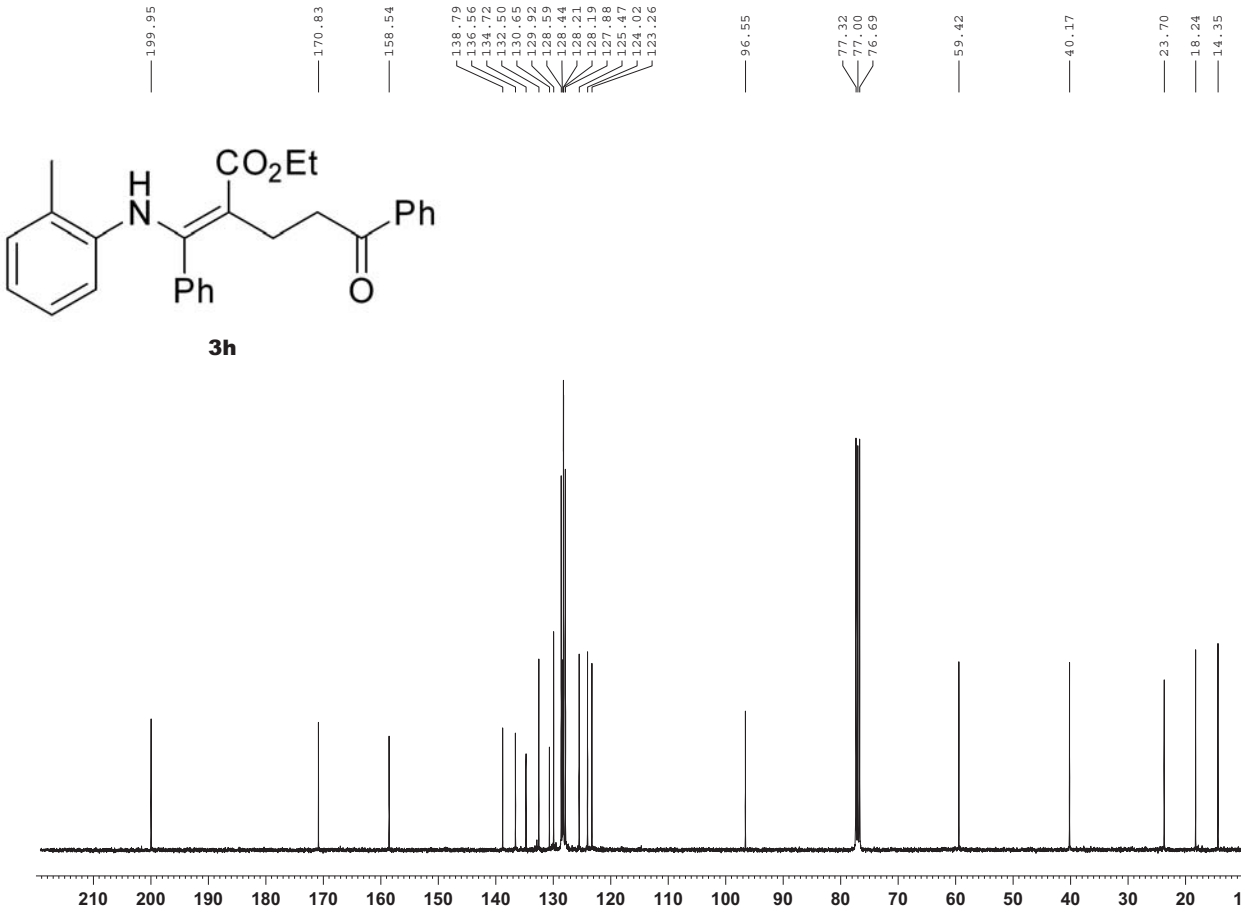

11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.97
2.04
1.08
5.02
2.22
1.06
1.00
1.02
1.01
2.04
2.00
2.00
3.08


199.78
170.73
156.78
141.66
136.58
134.24
133.96
132.69
129.30
129.06
128.79
128.77
128.36
127.95
122.35
121.49
119.57
98.57
77.32
77.00
76.68
59.83
40.04
23.53
14.39
Cl
H
N
CO2Et
Ph
Ph
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

3g


10.916
7.810
7.792
7.506
7.488
7.470
7.388
7.369
7.350
7.261
7.253
7.246
7.222
7.193
7.185
7.176
7.170
7.059
7.041
6.798
6.781
6.763
6.723
6.704
6.686
6.311
6.291
4.295
4.277
4.260
4.242
3.009
2.995
2.990
2.982
2.969
2.567
2.555
2.547
2.541
2.528
2.381
1.345
1.327
1.309
0.000
H
N
CO2Et
Ph
Ph
O
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.96
2.05
1.03
2.08
3.09
2.07
1.03
1.05
1.01
1.01
2.01
2.00
2.02
3.05
3.03

3h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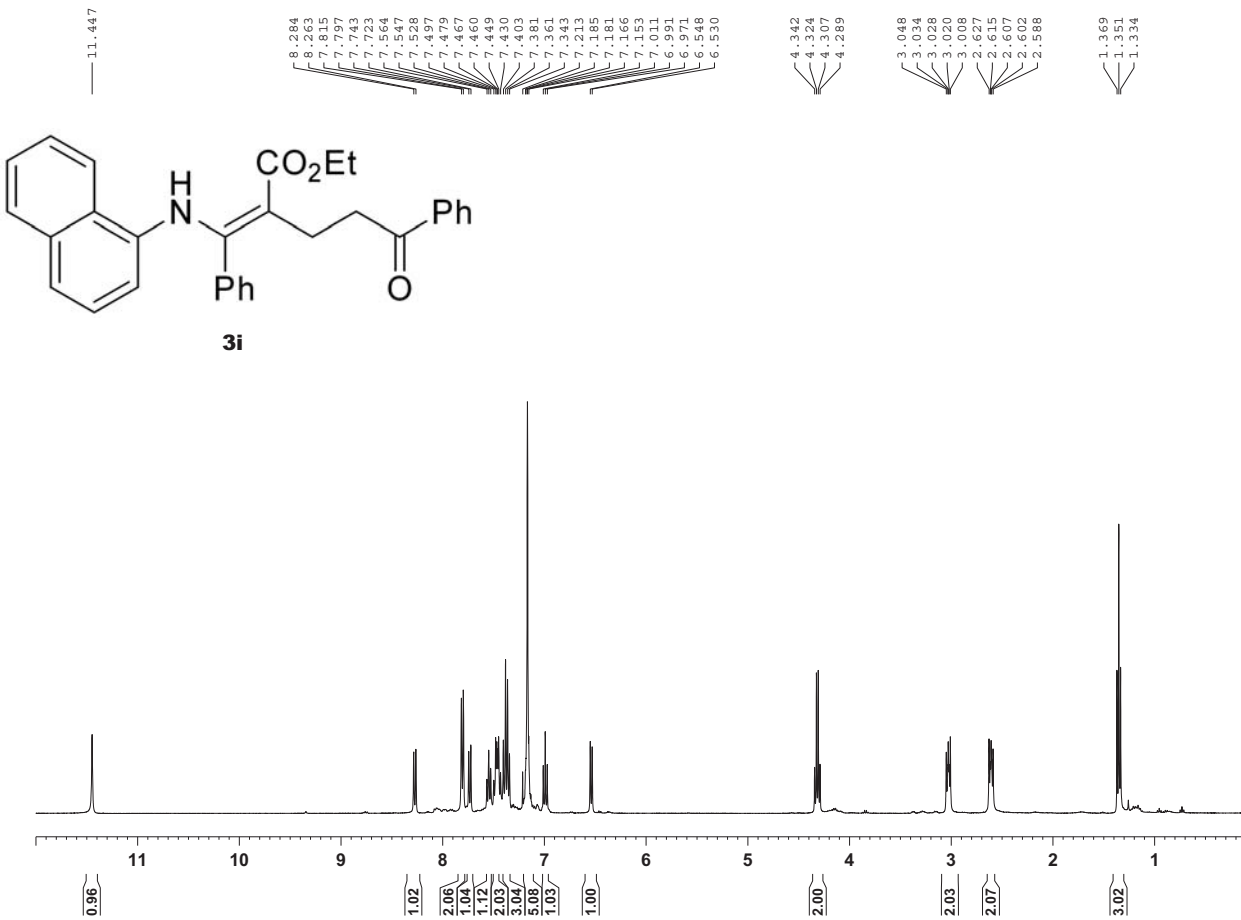
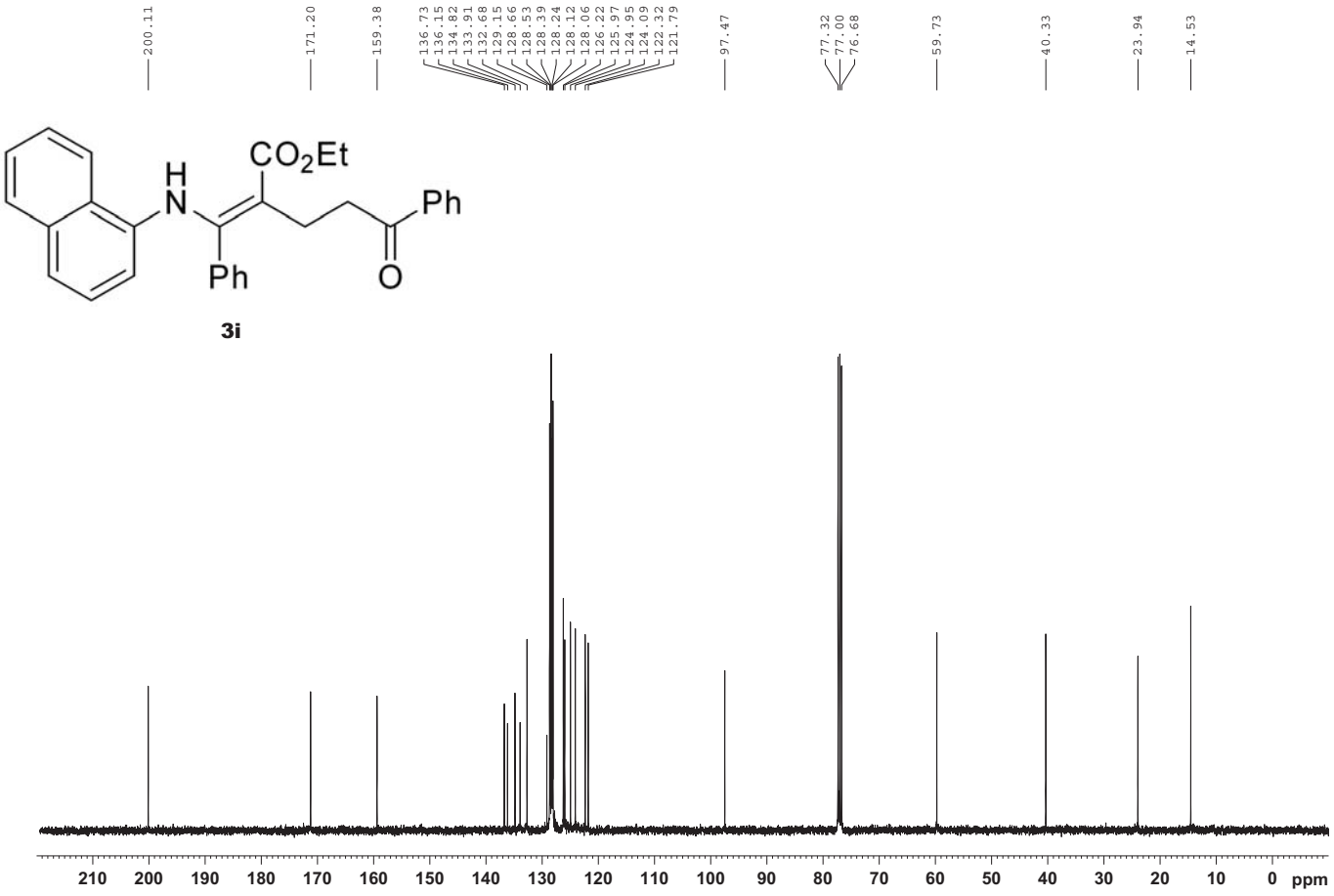
170.83
158.54
138.79
136.56
134.72
132.50
130.65
129.92
128.59
128.44
128.21
128.19
127.88
125.47
124.02
123.26
96.55
77.32
77.00
76.69
59.42
40.17
23.70
18.24
14.35
CO2Et
H
N
Ph
Ph
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

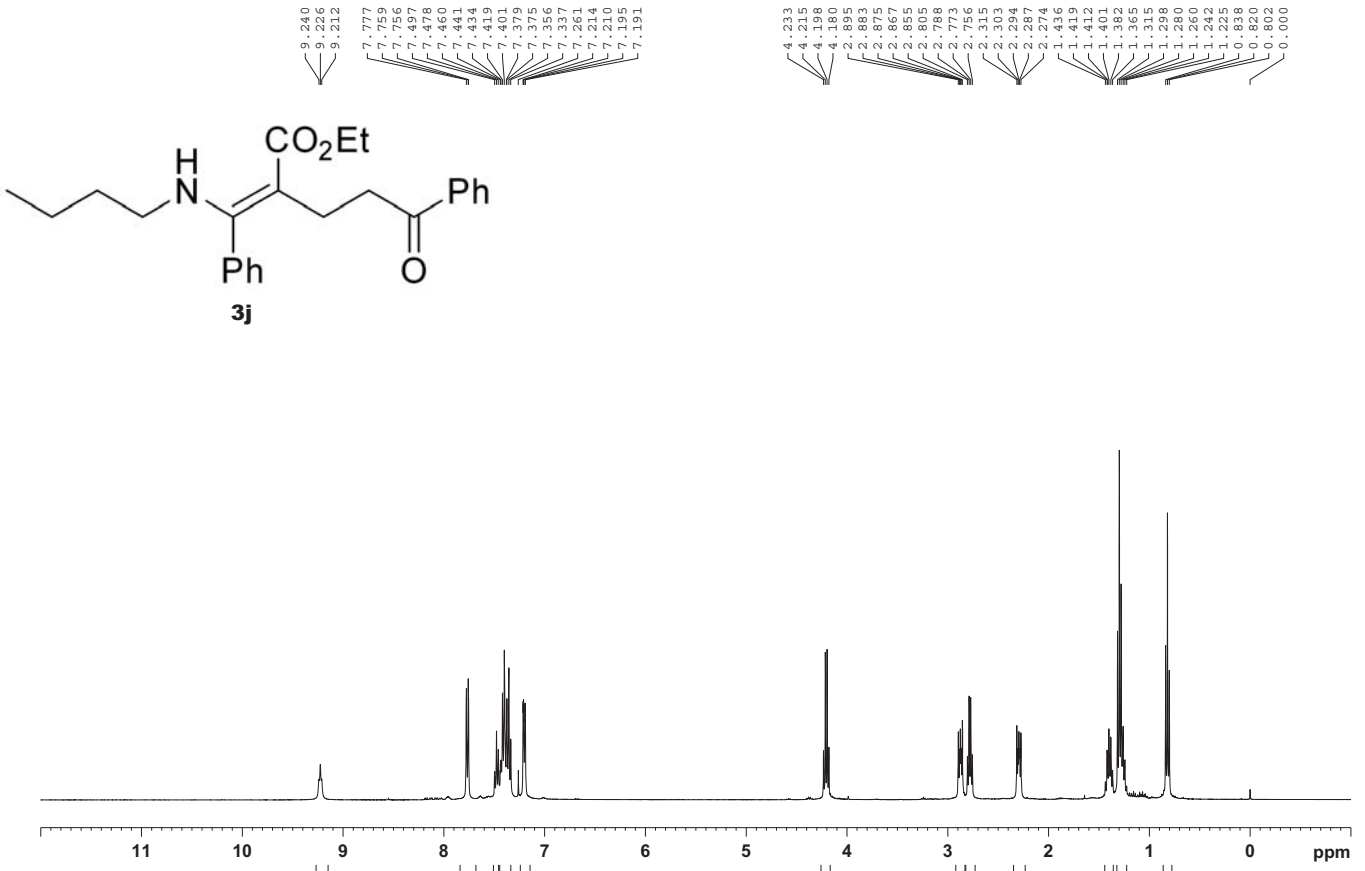
3h


11.447
8.284
8.263
7.815
7.797
7.743
7.723
7.564
7.547
7.528
7.497
7.479
7.467
7.460
7.449
7.430
7.403
7.381
7.361
7.343
7.213
7.185
7.181
7.166
7.153
7.011
6.991
6.971
6.548
6.530
4.342
4.324
4.307
4.289
3.048
3.034
3.028
3.020
3.008
2.627
2.615
2.607
2.602
2.588
1.369
1.351
1.334
0.000
CO2Et
H
N
Ph
Ph
O
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.96
1.02
2.06
1.04
1.12
2.03
3.04
5.08
1.03
1.00
2.00
2.03
2.07
3.02

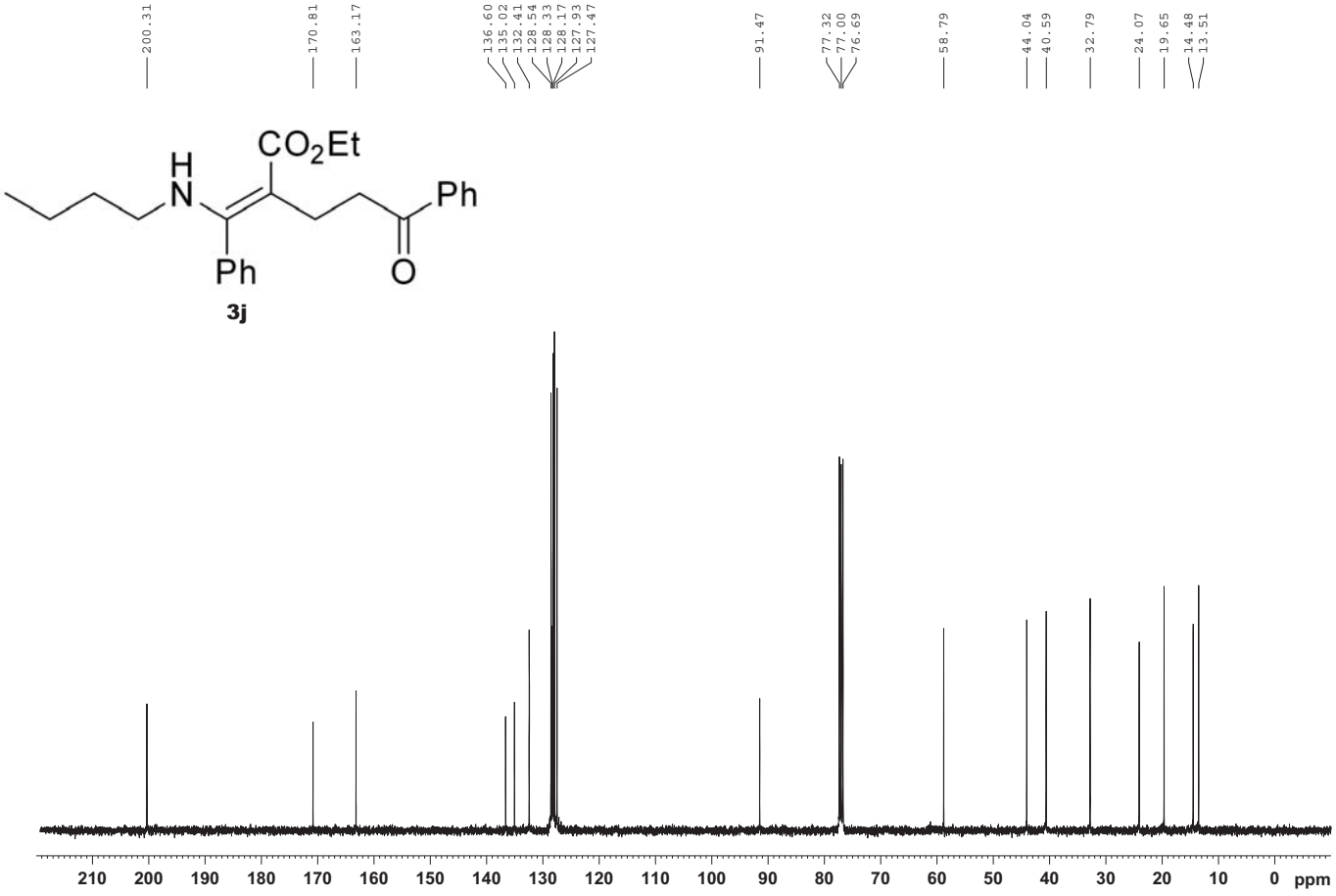
3i


200.11
171.20
159.38
136.73
136.15
134.82
133.91
132.68
129.15
128.66
128.53
128.39
128.24
128.12
128.06
126.22
125.97
124.95
124.09
122.32
121.79
97.47
77.32
77.00
76.68
59.73
40.33
23.94
14.53
CO2Et
H
N
Ph
Ph
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

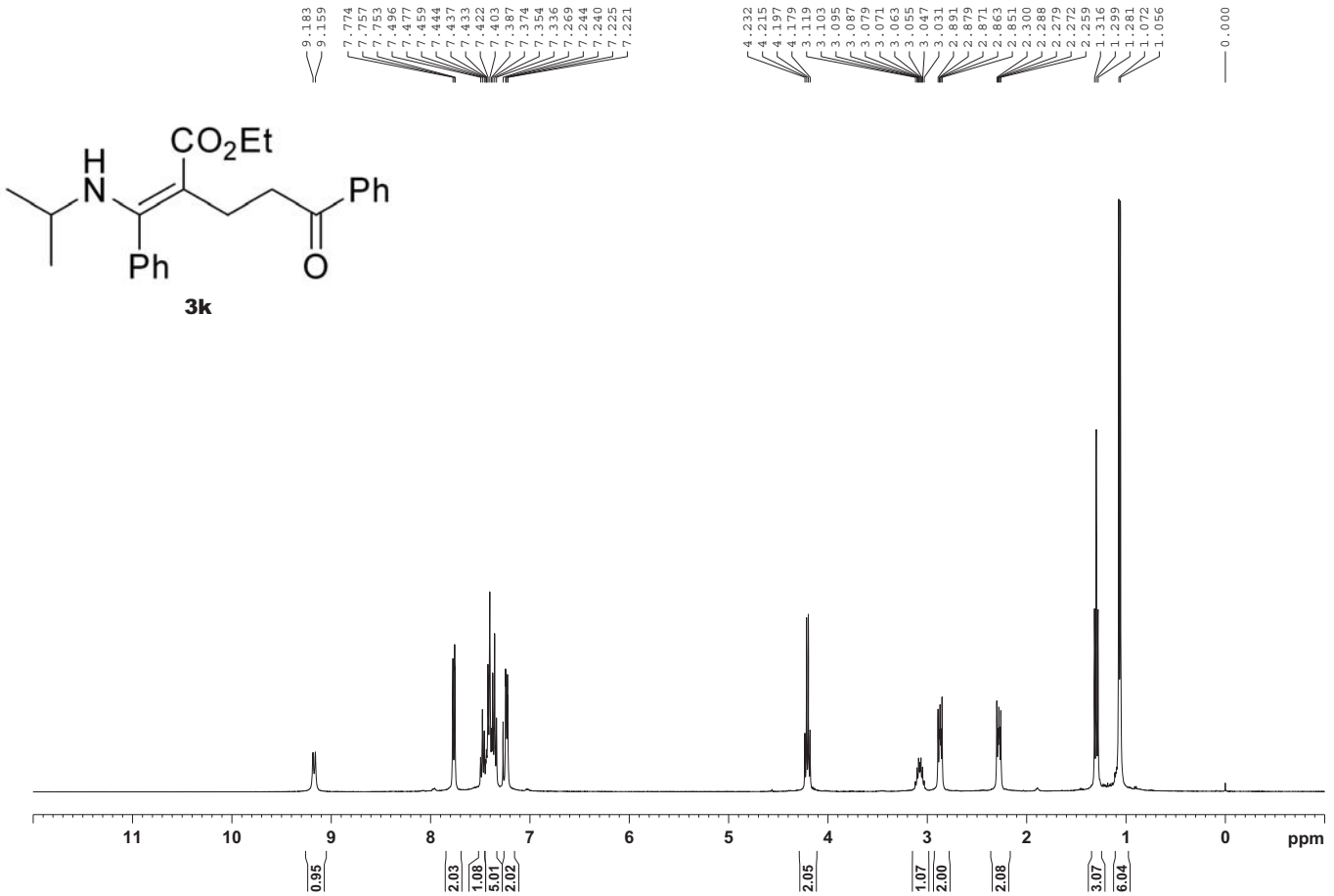
3i


9.240
9.226
9.212
7.777
7.759
7.756
7.497
7.478
7.460
7.441
7.434
7.419
7.401
7.379
7.375
7.356
7.337
7.261
7.214
7.210
7.195
7.191
4.233
4.215
4.198
4.180
2.895
2.883
2.875
2.867
2.855
2.805
2.788
2.773
2.756
2.315
2.303
2.294
2.287
2.274
1.436
1.419
1.412
1.401
1.382
1.365
1.315
1.298
1.280
1.260
1.242
1.225
0.838
0.820
0.802
0.000
CO2Et
H
N
Ph
Ph
O
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.91
2.04
1.07
5.09
2.05
2.04
2.00
2.01
2.00
2.07
5.03
3.03

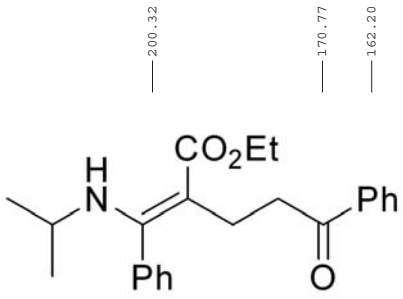
3j


200.31
170.81
163.17
136.60
135.02
132.41
128.54
128.33
128.17
127.93
127.47
91.47
77.32
77.00
76.69
58.79
44.04
40.59
32.79
24.07
19.65
14.48
13.51
CO2Et
H
N
Ph
Ph
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

3j

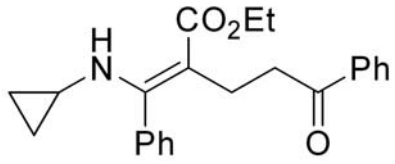

9.183
9.159
7.774
7.757
7.753
7.496
7.477
7.459
7.444
7.437
7.433
7.422
7.403
7.387
7.374
7.354
7.336
7.269
7.244
7.240
7.225
7.221
4.232
4.215
4.197
4.179
3.119
3.103
3.095
3.087
3.079
3.071
3.063
3.055
3.047
3.031
2.891
2.879
2.871
2.863
2.851
2.300
2.288
2.279
2.272
2.259
1.316
1.299
1.281
1.072
1.056
0.000
CO2Et
H
N
Ph
Ph
O
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.95
2.03
1.08
5.01
2.02
2.05
1.07
2.00
2.08
3.07
6.04

3k


CO2Et
H
N
Ph
Ph
O

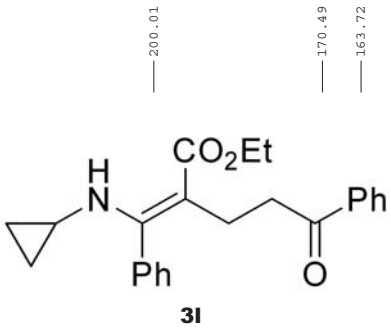
3k

200.32
170.77
162.20
136.62
135.19
132.45
128.54
128.37
128.20
127.95
127.51
91.46
77.31
77.00
76.68
58.79
45.61
40.61
24.31
24.02
14.51
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

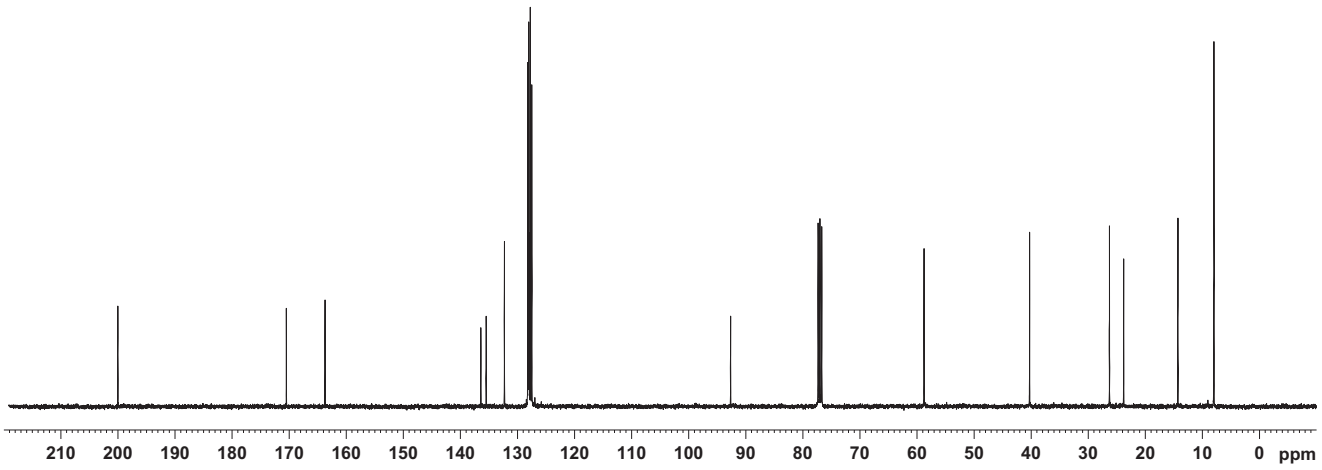

CO2Et
H
N
Ph
Ph
O

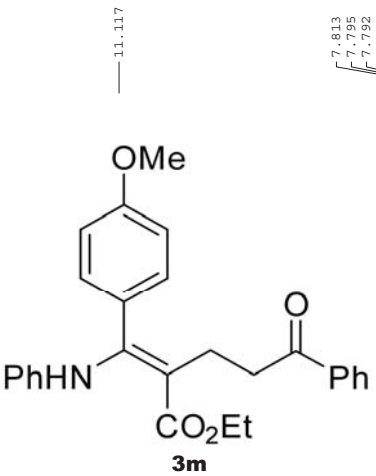
3l

9.141
9.135
7.771
7.753
7.750
7.492
7.473
7.455
7.436
7.432
7.415
7.397
7.390
7.386
7.370
7.351
7.332
7.272
7.268
7.252
4.214
4.197
4.179
4.161
2.905
2.892
2.885
2.876
2.864
2.345
2.333
2.325
2.318
2.305
2.212
2.202
2.194
2.185
2.176
2.167
2.159
2.149
1.302
1.284
1.266
0.456
0.437
0.427
0.417
0.411
0.395
0.384
0.378
0.366
0.353
0.348
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.96
1.98
1.04
5.02
2.06
2.06
2.00
2.02
1.05
3.07
2.00
2.02

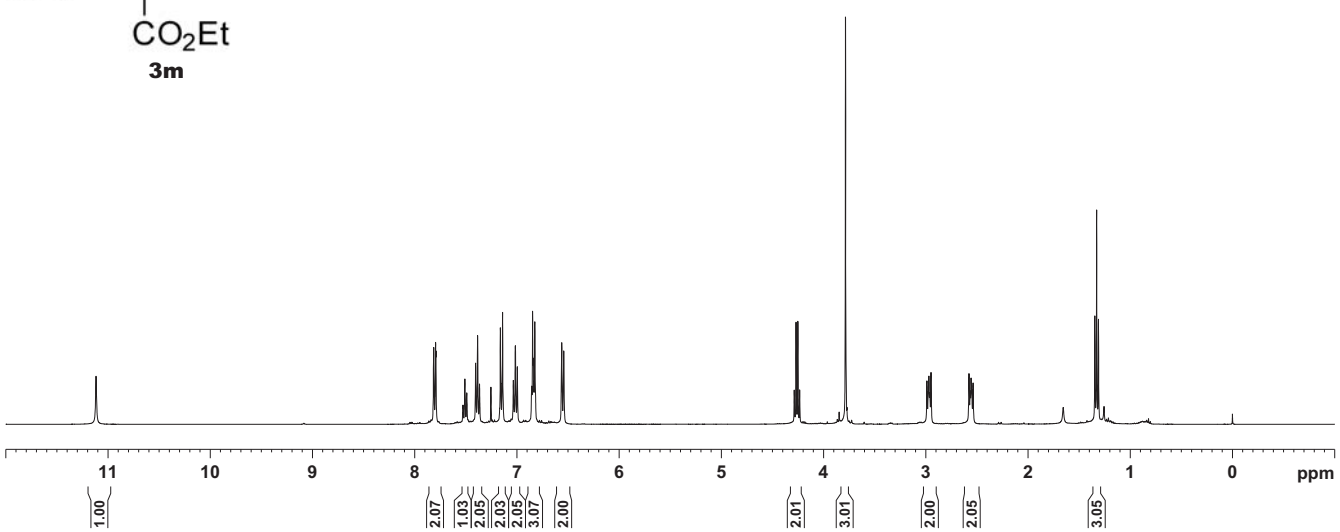

200.01
170.49
163.72
136.42
135.47
132.28
128.19
128.03
127.98
127.76
127.49
92.65
77.32
77.00
76.68
58.76
40.27
26.27
23.78
14.28
7.97
CO2Et
H
N
Ph
Ph
O

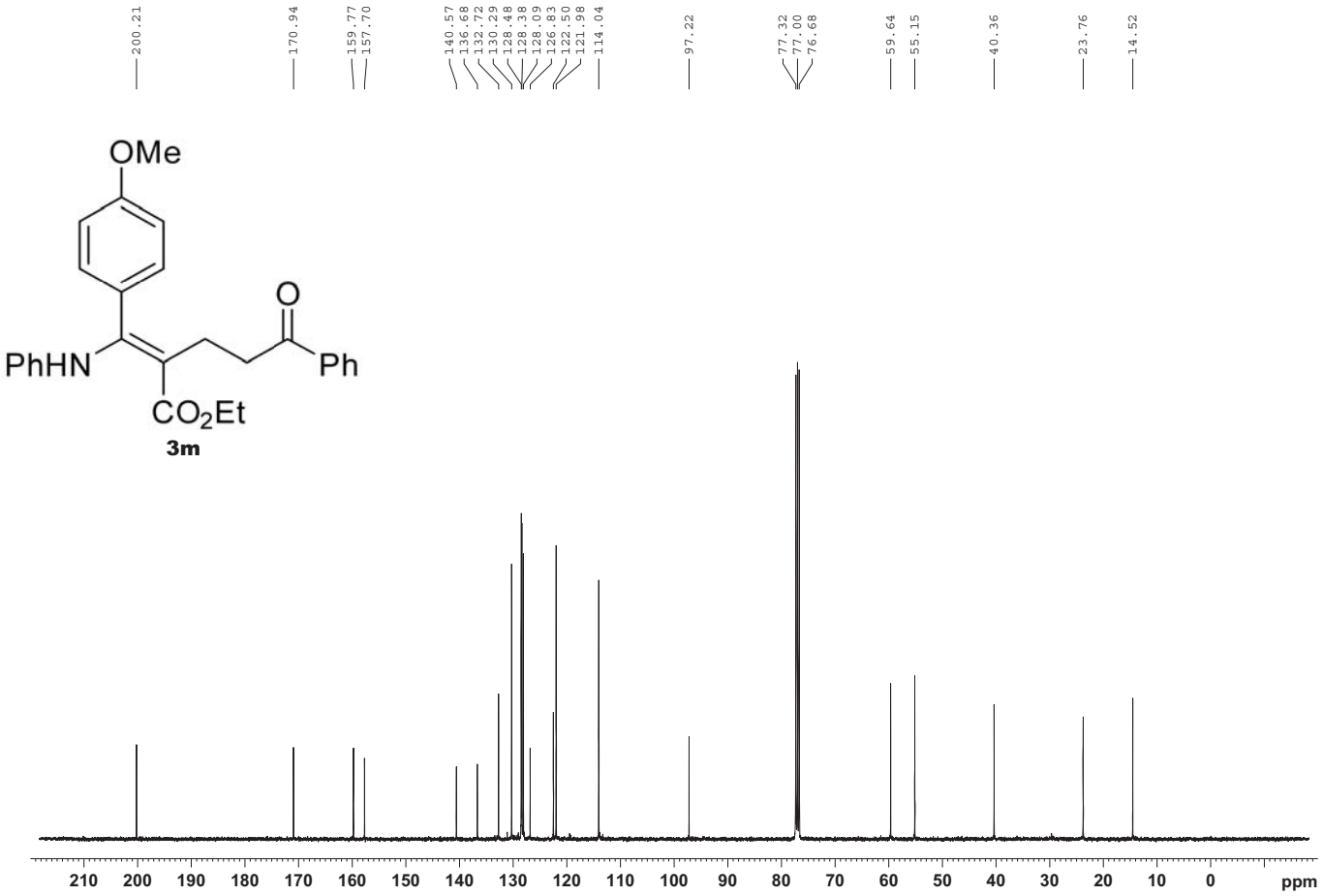
3l

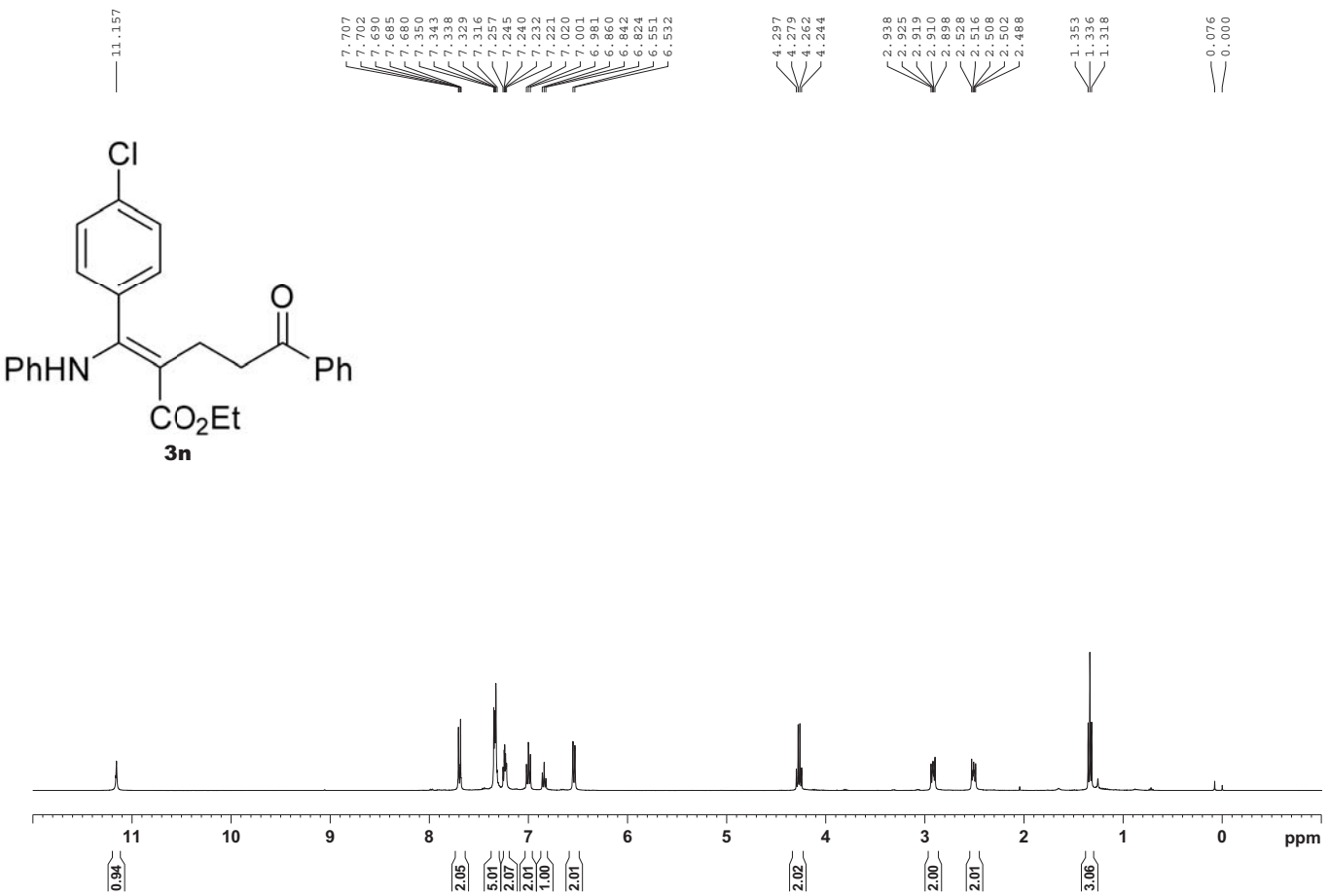

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

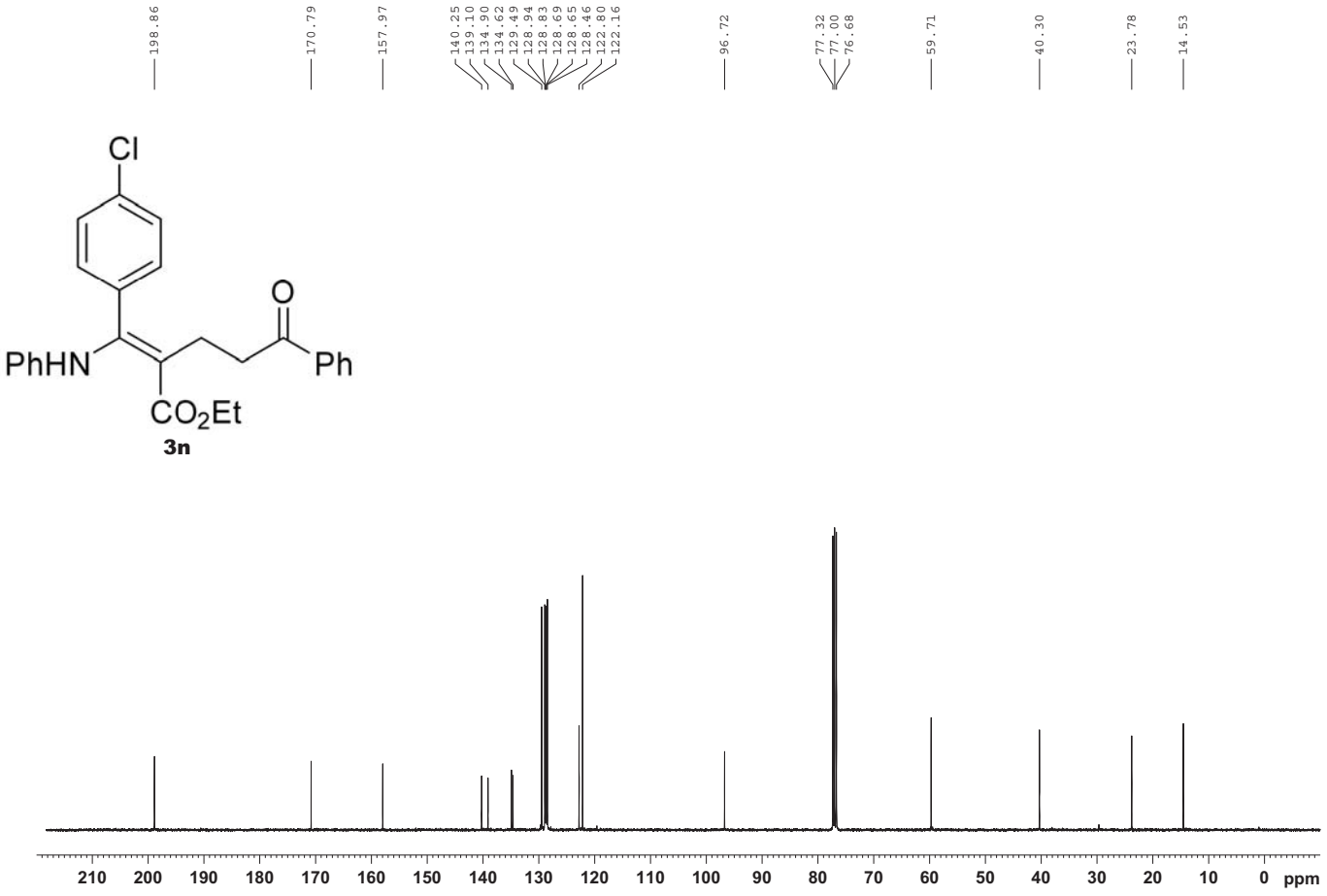

11.117
7.813
7.795
7.792
7.527
7.509
7.490
7.404
7.384
7.366
7.254
7.162
7.157
7.145
7.140
7.035
7.015
6.995
6.856
6.854
6.847
6.841
6.838
6.830
6.825
6.561
6.542
4.287
4.269
4.251
4.234
3.785
2.989
2.975
2.969
2.962
2.949
2.577
2.564
2.557
2.551
2.537
1.346
1.328
1.311
-0.000
OMe
O
PhHN
Ph
CO2Et

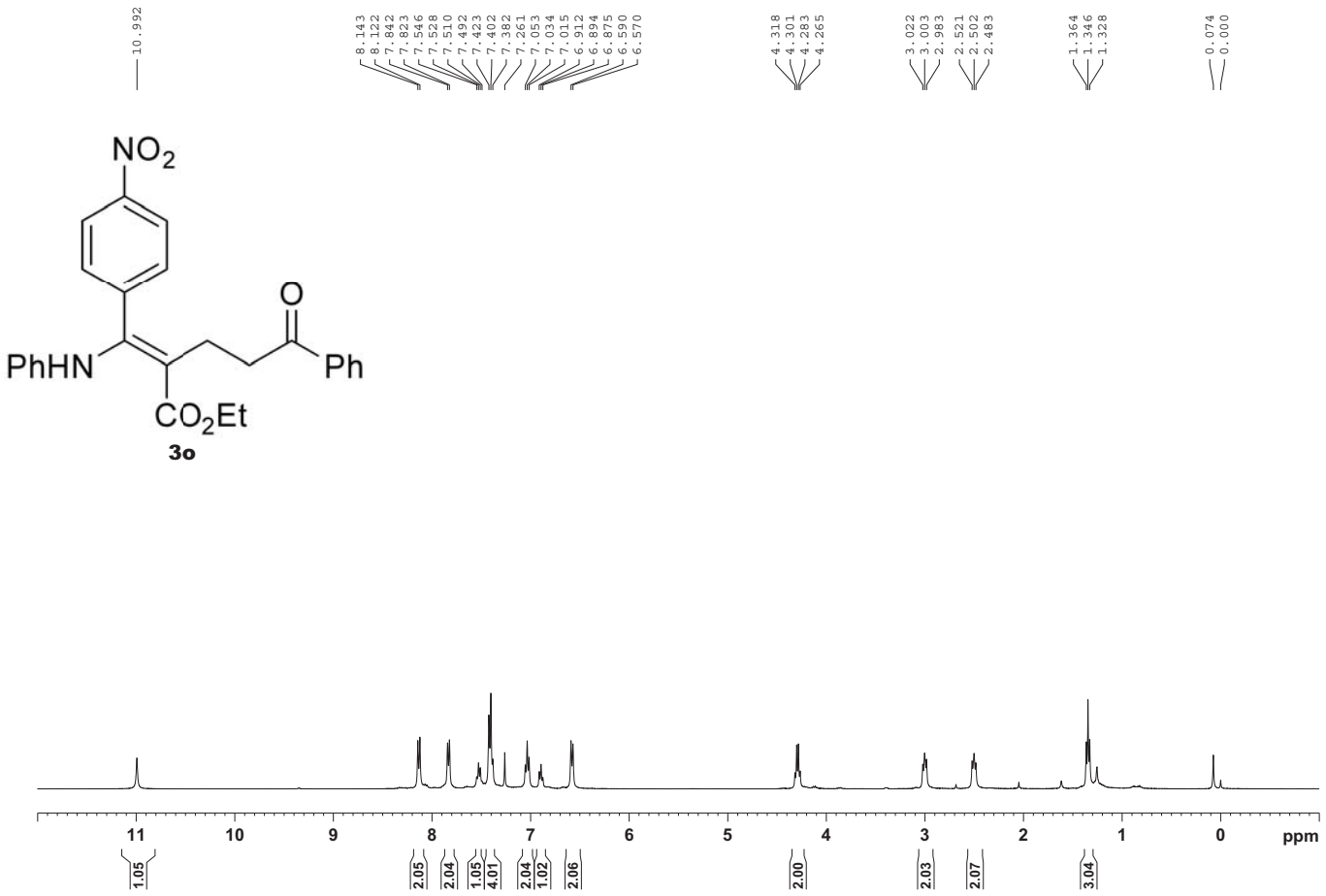
3m


11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
2.07
1.03
2.05
2.03
2.05
3.07
2.00
2.01
3.01
2.00
2.05
3.05


200.21
170.94
159.77
157.70
140.57
136.68
132.72
130.29
128.48
128.38
128.09
126.83
122.50
121.98
114.04
97.22
77.32
77.00
76.68
59.64
55.15
40.36
23.76
14.52
OMe
O
PhHN
Ph
CO2Et
3m
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


11.157
7.707
7.702
7.690
7.685
7.680
7.350
7.343
7.338
7.329
7.316
7.257
7.245
7.240
7.232
7.221
7.020
7.001
6.981
6.860
6.842
6.824
6.551
6.532
4.297
4.279
4.262
4.244
2.938
2.925
2.919
2.910
2.898
2.528
2.516
2.508
2.502
2.488
1.353
1.336
1.318
0.076
0.000
Cl
O
PhHN
Ph
CO2Et
3n
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.94
2.05
5.01
2.07
2.01
1.00
2.01
2.02
2.00
2.01
3.06


198.86
170.79
157.97
140.25
139.10
134.90
134.62
129.49
128.94
128.83
128.69
128.65
128.46
122.80
122.16
96.72
77.32
77.00
76.68
59.71
40.30
23.78
14.53
Cl
O
PhHN
Ph
CO2Et
3n
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


10.992
8.143
8.122
7.842
7.823
7.546
7.528
7.510
7.492
7.423
7.402
7.382
7.261
7.053
7.034
7.015
6.912
6.894
6.875
6.590
6.570
4.318
4.301
4.283
4.265
3.022
3.003
2.983
2.521
2.502
2.483
1.364
1.346
1.328
0.074
0.000
NO2
O
PhHN
Ph
CO2Et
3o
11 10 9 8 7 6 5 4 3 2 1 0 ppm
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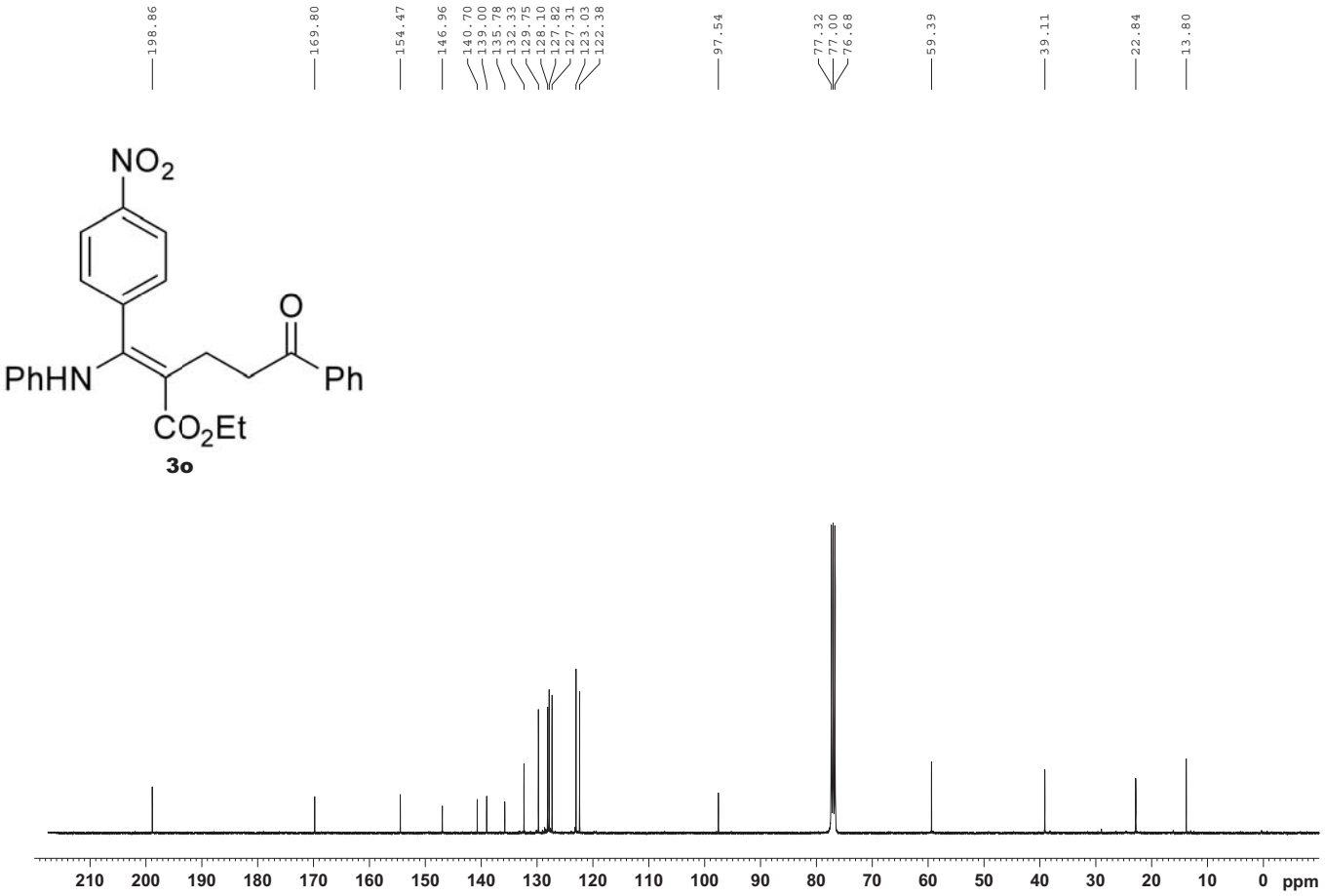
2.05
2.04
1.05
4.01
2.04
1.02
2.06
2.00
2.03
2.07
3.04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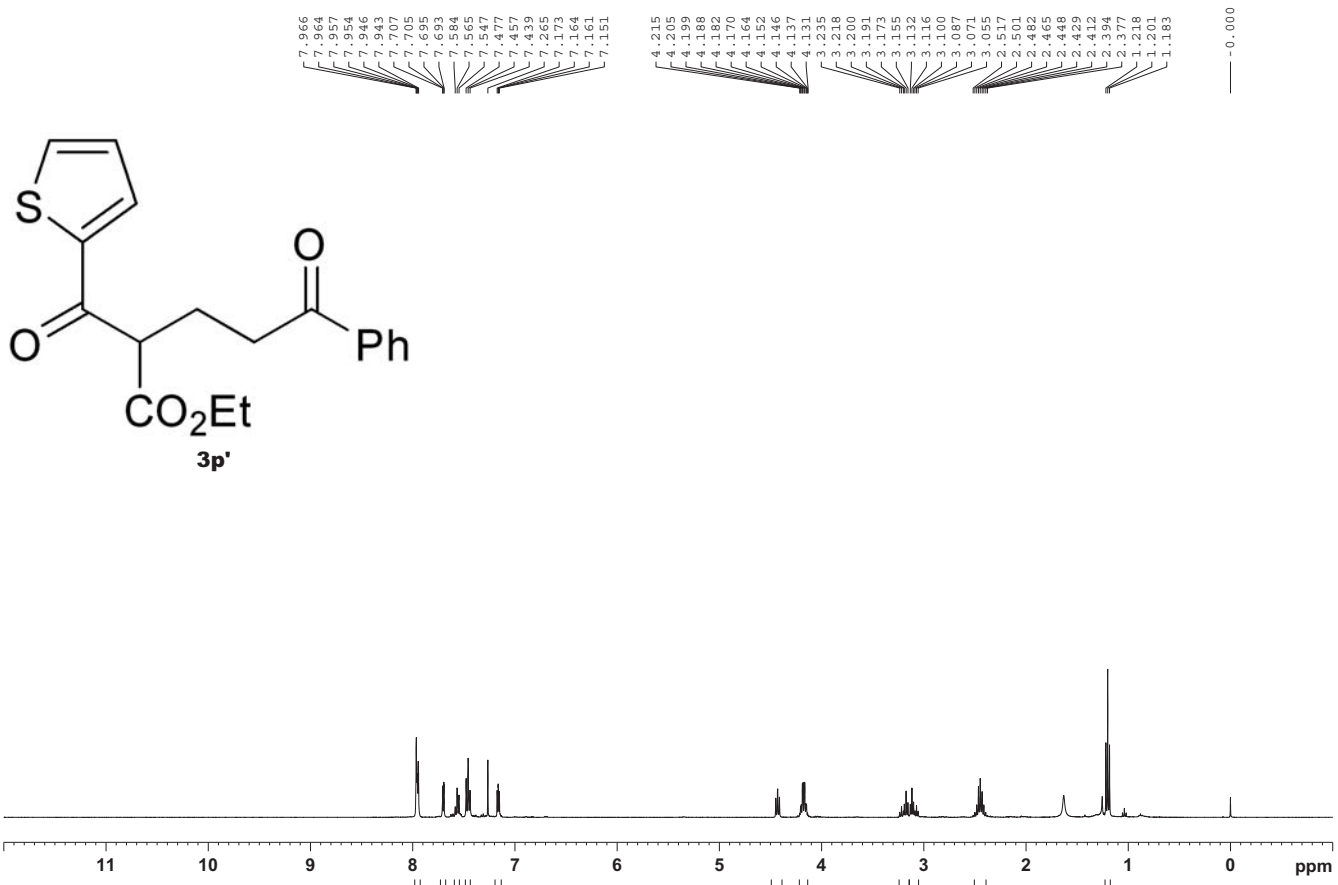
169.80
154.47
146.96
140.70
139.00
135.78
132.33
129.75
128.10
127.82
127.31
123.03
122.38
97.54
77.32
77.00
76.68
59.39
39.11
22.84
13.80
NO2
PhHN
O
Ph
CO2Et
3o
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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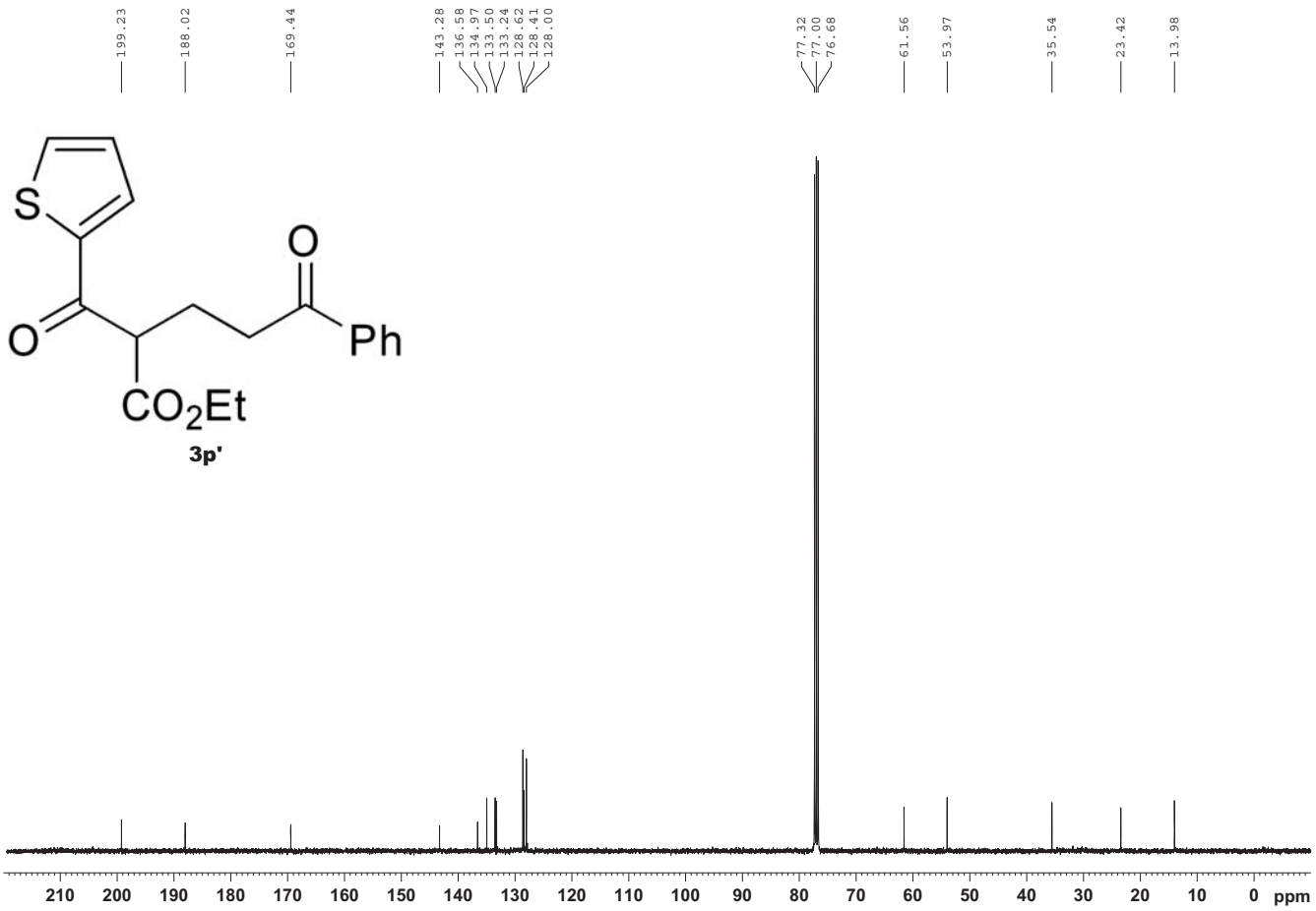
7.964
7.957
7.954
7.946
7.943
7.707
7.705
7.695
7.693
7.584
7.565
7.547
7.477
7.457
7.439
7.265
7.173
7.164
7.161
7.151
4.215
4.205
4.199
4.188
4.182
4.170
4.164
4.152
4.146
4.137
4.131
3.235
3.218
3.200
3.191
3.173
3.155
3.132
3.116
3.100
3.087
3.071
3.055
2.517
2.501
2.482
2.465
2.448
2.429
2.412
2.394
2.377
1.218
1.201
1.183
-0.000
S
O
O
Ph
CO2Et
3p'
11 10 9 8 7 6 5 4 3 2 1 0 ppm
3.04
1.03
1.04
2.02
1.06
1.00
2.02
1.02
1.08
2.06
3.04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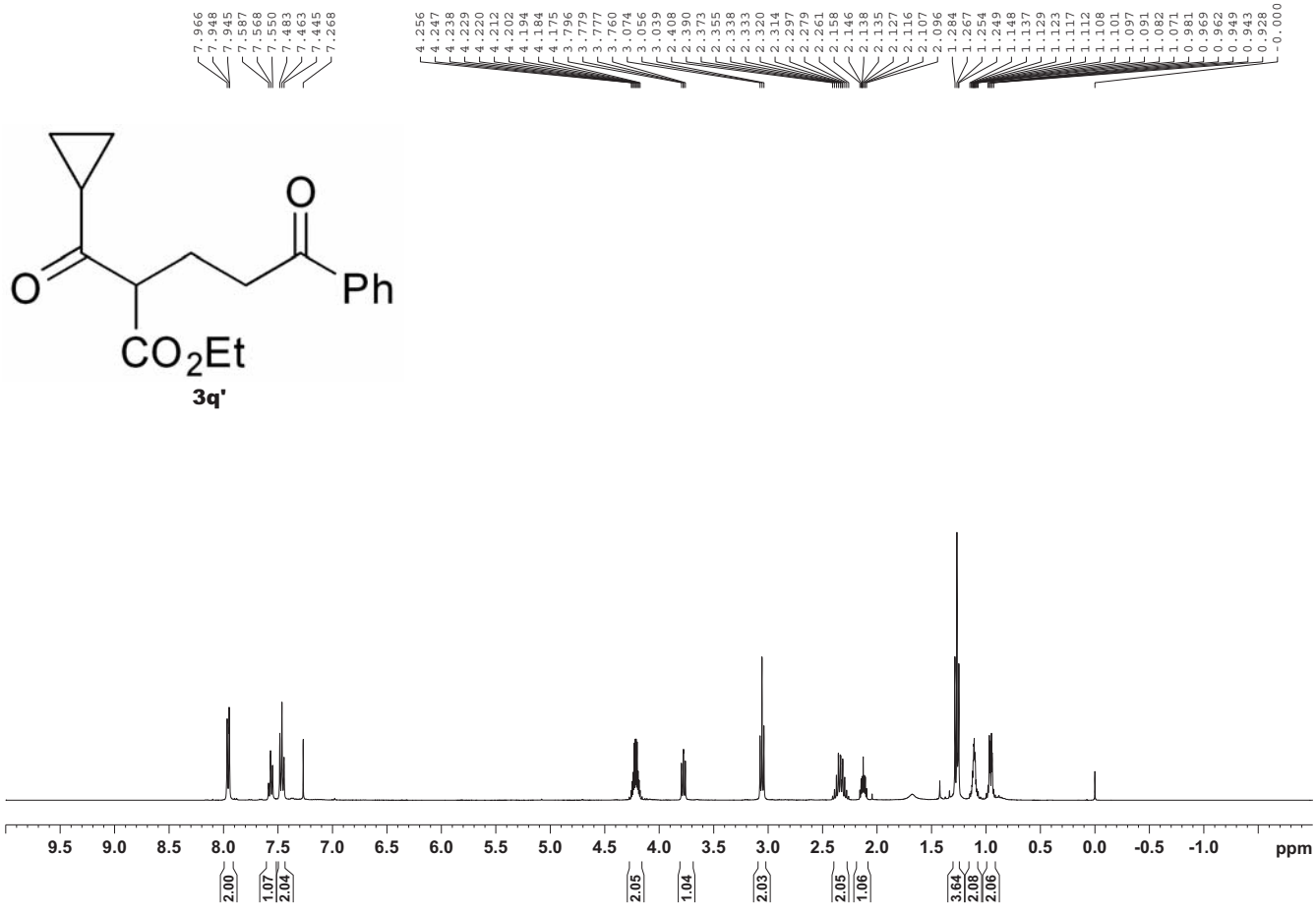
188.02
169.44
143.28
136.58
134.97
133.50
133.24
128.62
128.41
128.00
77.32
77.00
76.68
61.56
53.97
35.54
23.42
13.98
S
O
O
Ph
CO2Et
3p'
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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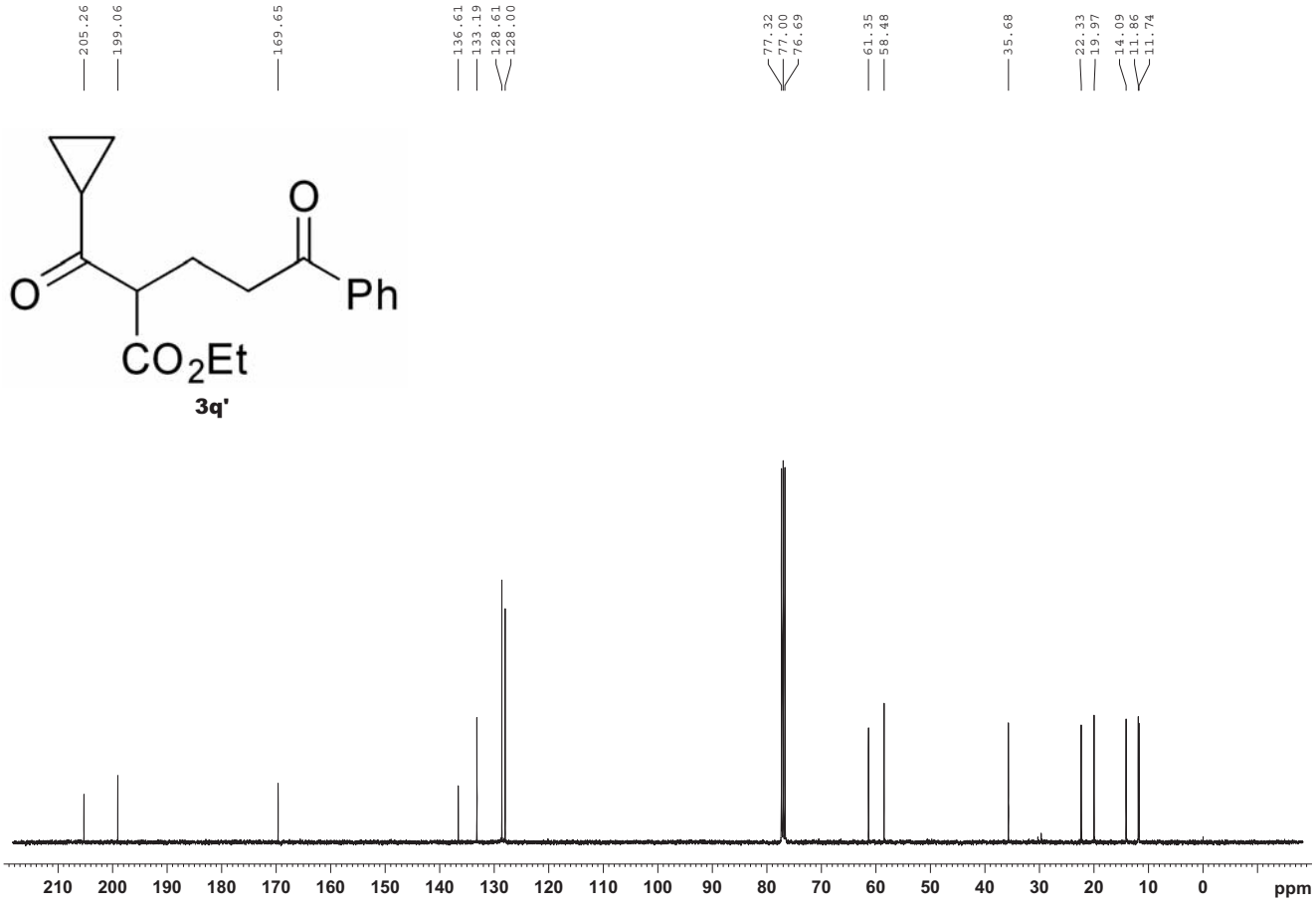
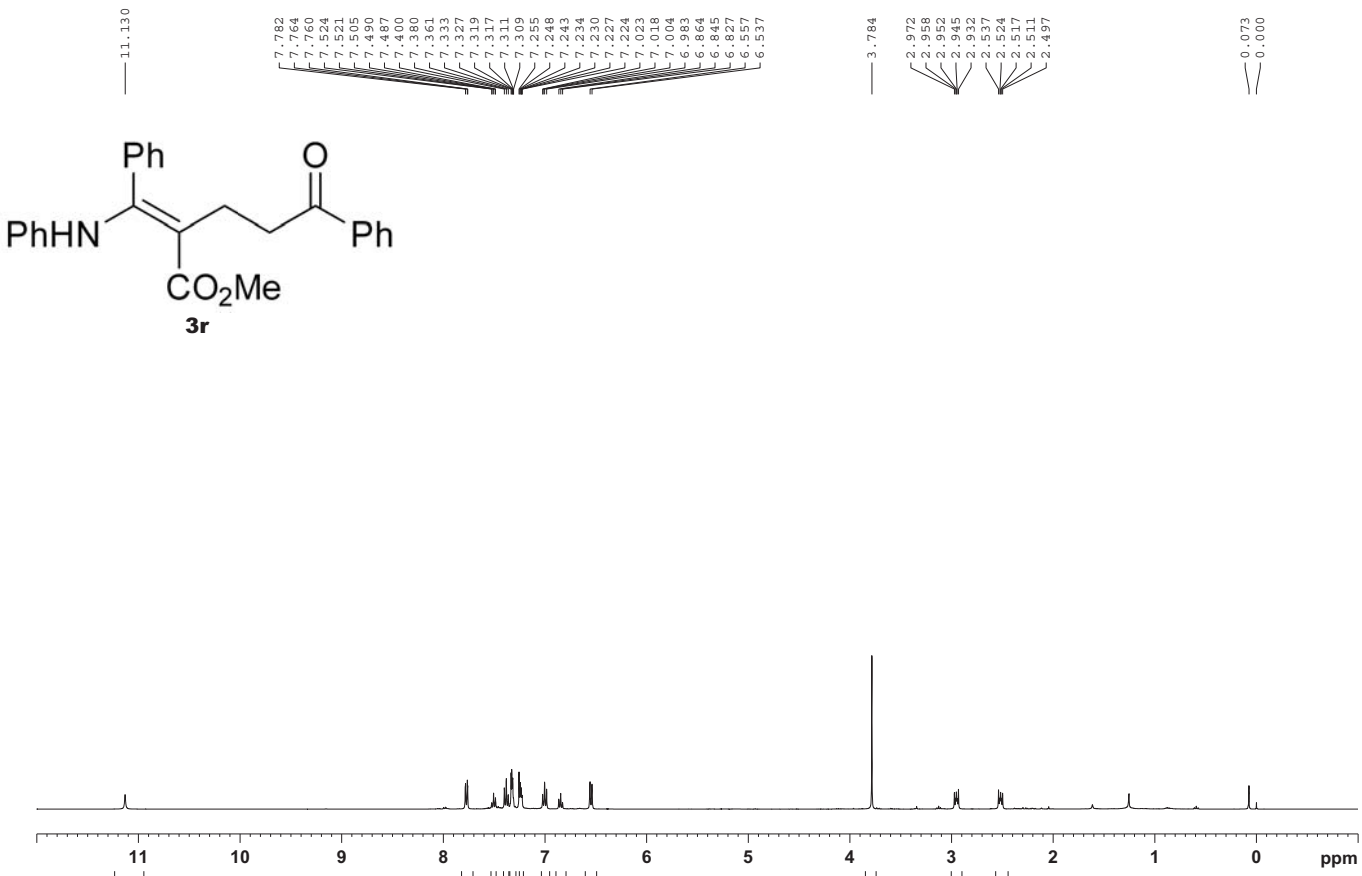
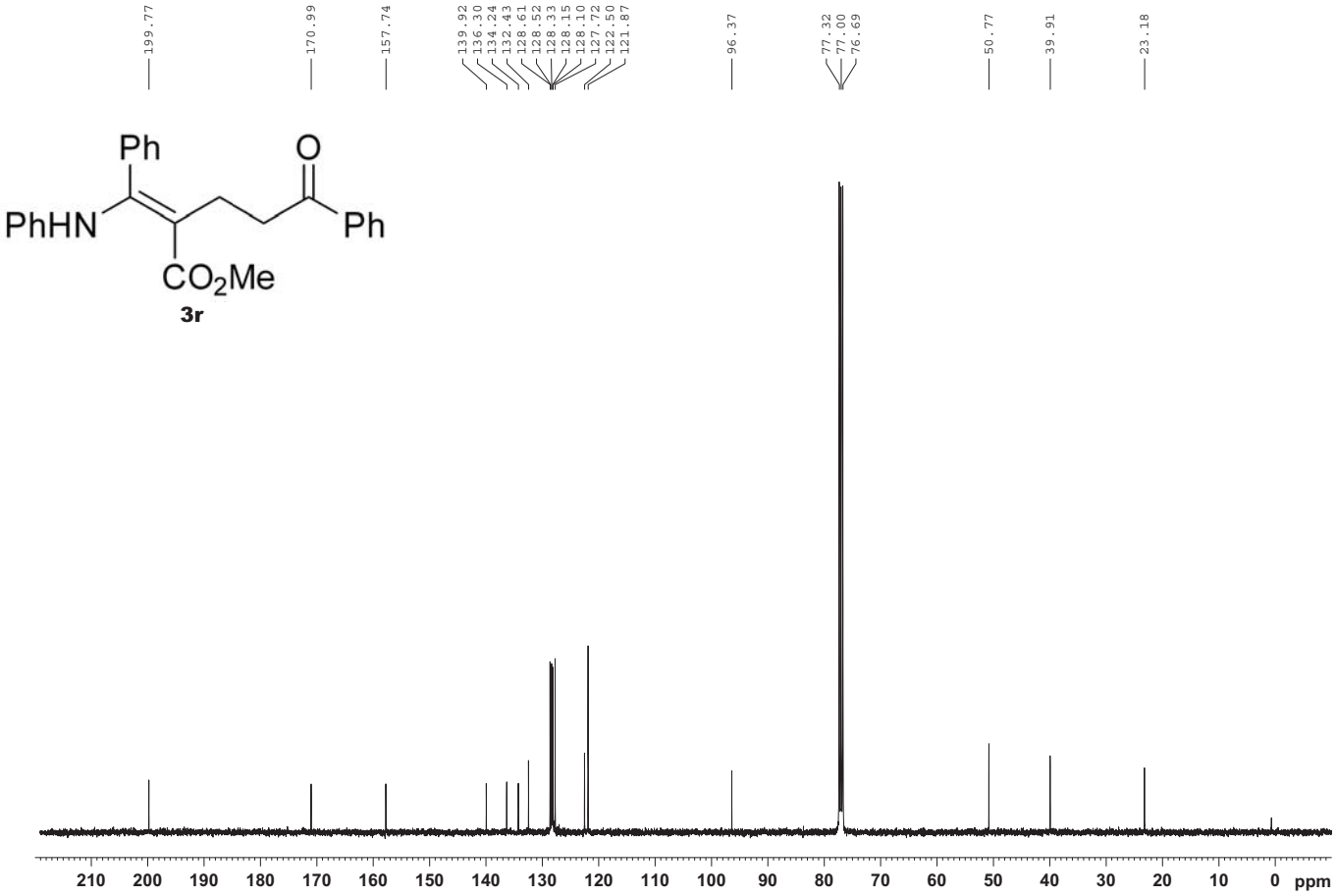
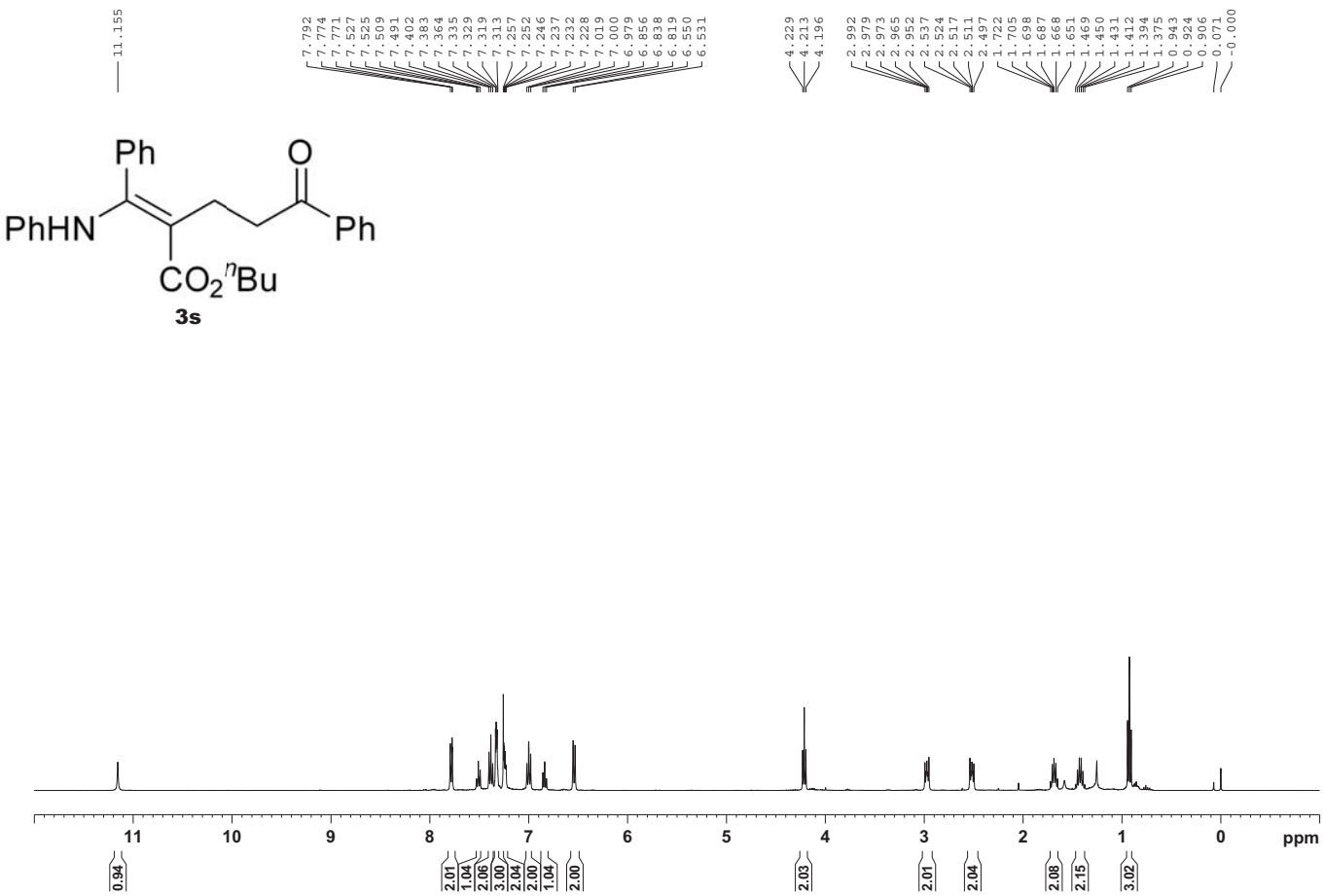
7.945
7.587
7.568
7.550
7.483
7.463
7.445
7.268
4.256
4.247
4.238
4.229
4.220
4.212
4.202
4.194
4.184
4.175
3.796
3.779
3.777
3.760
3.074
3.056
3.039
2.408
2.390
2.373
2.355
2.338
2.333
2.320
2.314
2.297
2.279
2.261
2.158
2.146
2.138
2.135
2.127
2.116
2.107
2.096
1.284
1.267
1.254
1.249
1.148
1.137
1.129
1.123
1.117
1.112
1.108
1.101
1.097
1.091
1.082
1.071
0.981
0.969
0.962
0.949
0.943
0.928
0.000
O
O
Ph
CO2Et
3q'
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 ppm
2.00
1.07
2.04
2.05
1.04
2.03
2.05
1.06
3.64
2.08
2.06


205.26
199.06
169.65
136.61
133.19
128.61
128.00
77.32
77.00
76.69
61.35
58.48
35.68
22.33
19.97
14.09
11.86
11.74
O
O
Ph
CO2Et
3q'
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


11.130
7.782
7.764
7.760
7.524
7.521
7.505
7.490
7.487
7.400
7.380
7.361
7.333
7.327
7.319
7.317
7.311
7.309
7.255
7.248
7.243
7.234
7.230
7.227
7.224
7.023
7.018
7.004
6.983
6.864
6.845
6.827
6.557
6.537
3.784
2.972
2.958
2.952
2.945
2.932
2.537
2.524
2.517
2.511
2.497
0.073
0.000
Ph
O
PhHN
Ph
CO2Me
3r
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
2.01
1.06
2.00
3.03
2.04
2.03
1.04
2.00
3.00
2.00
2.02


199.77
170.99
157.74
139.92
136.30
134.24
132.43
128.61
128.52
128.33
128.15
128.10
127.72
122.50
121.87
96.37
77.32
77.00
76.69
50.77
39.91
23.18
Ph
O
PhHN
Ph
CO2Me
3r
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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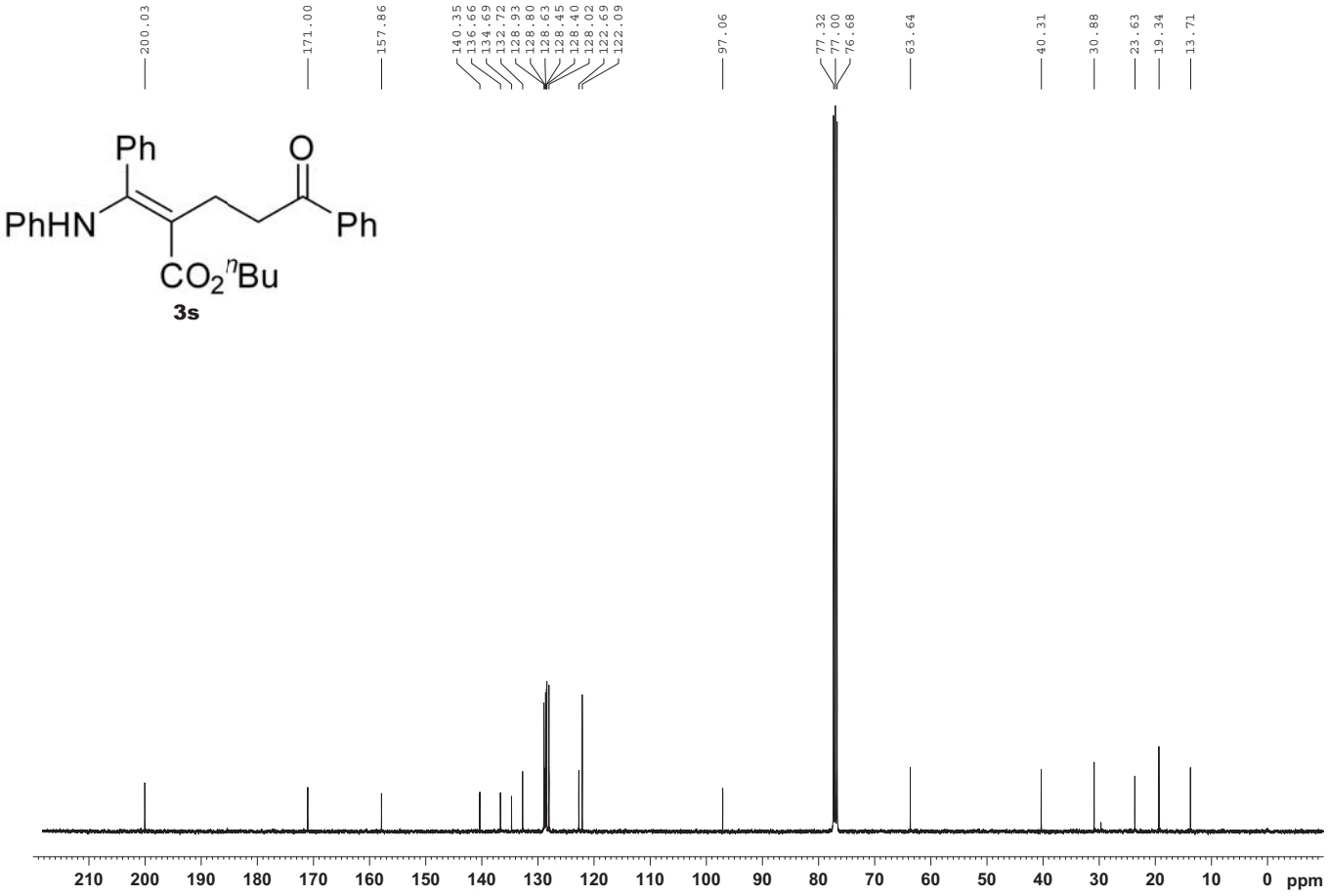
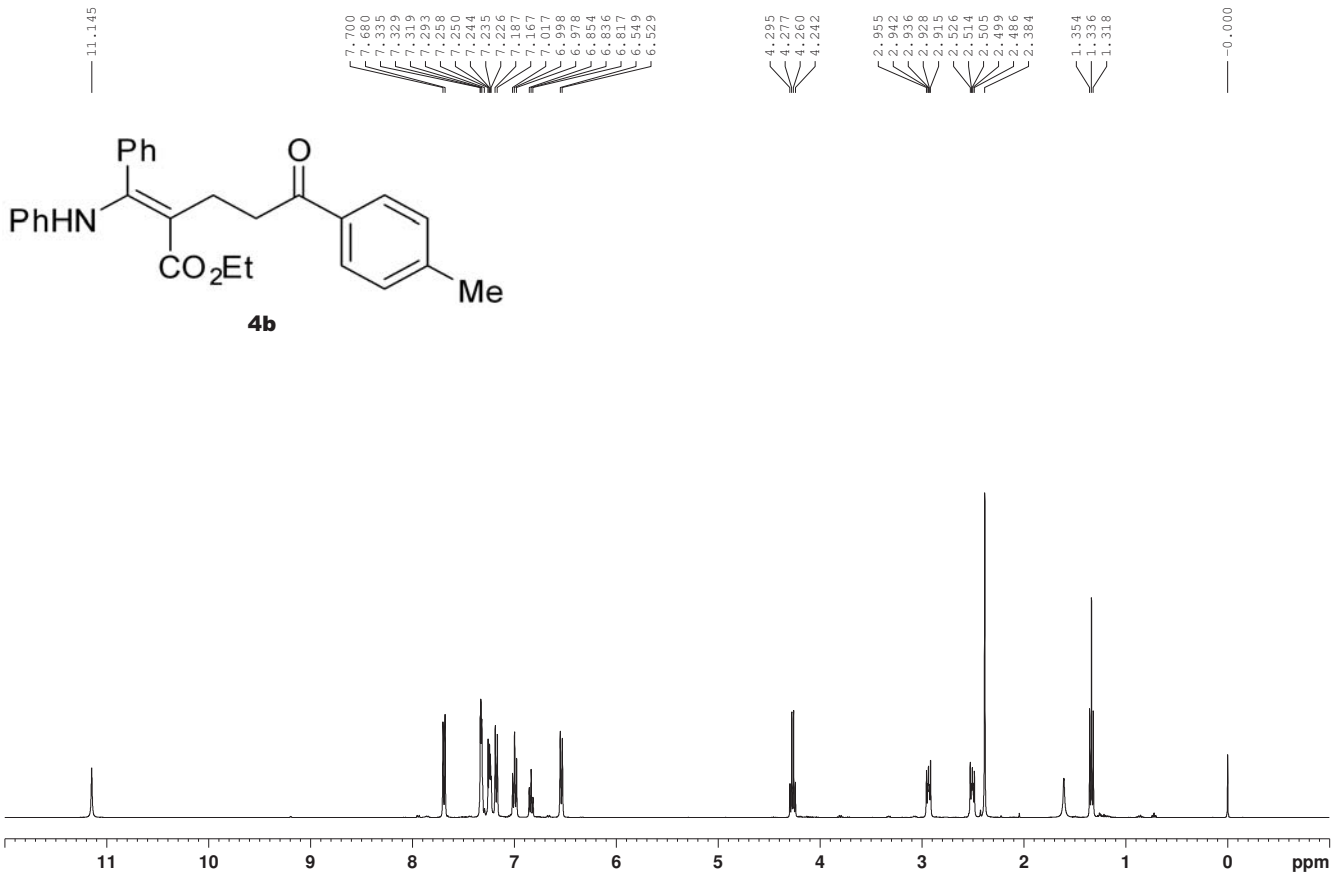
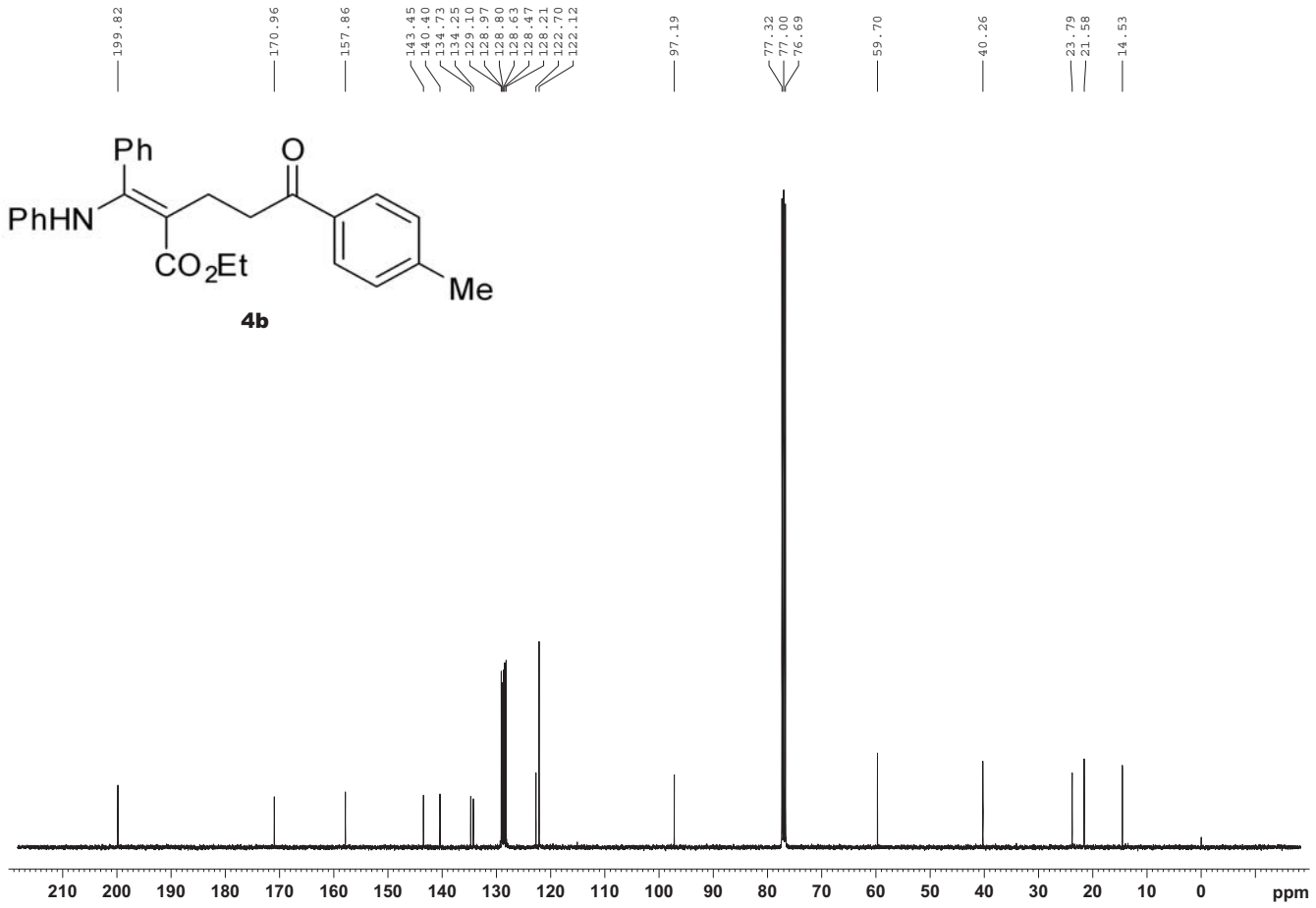
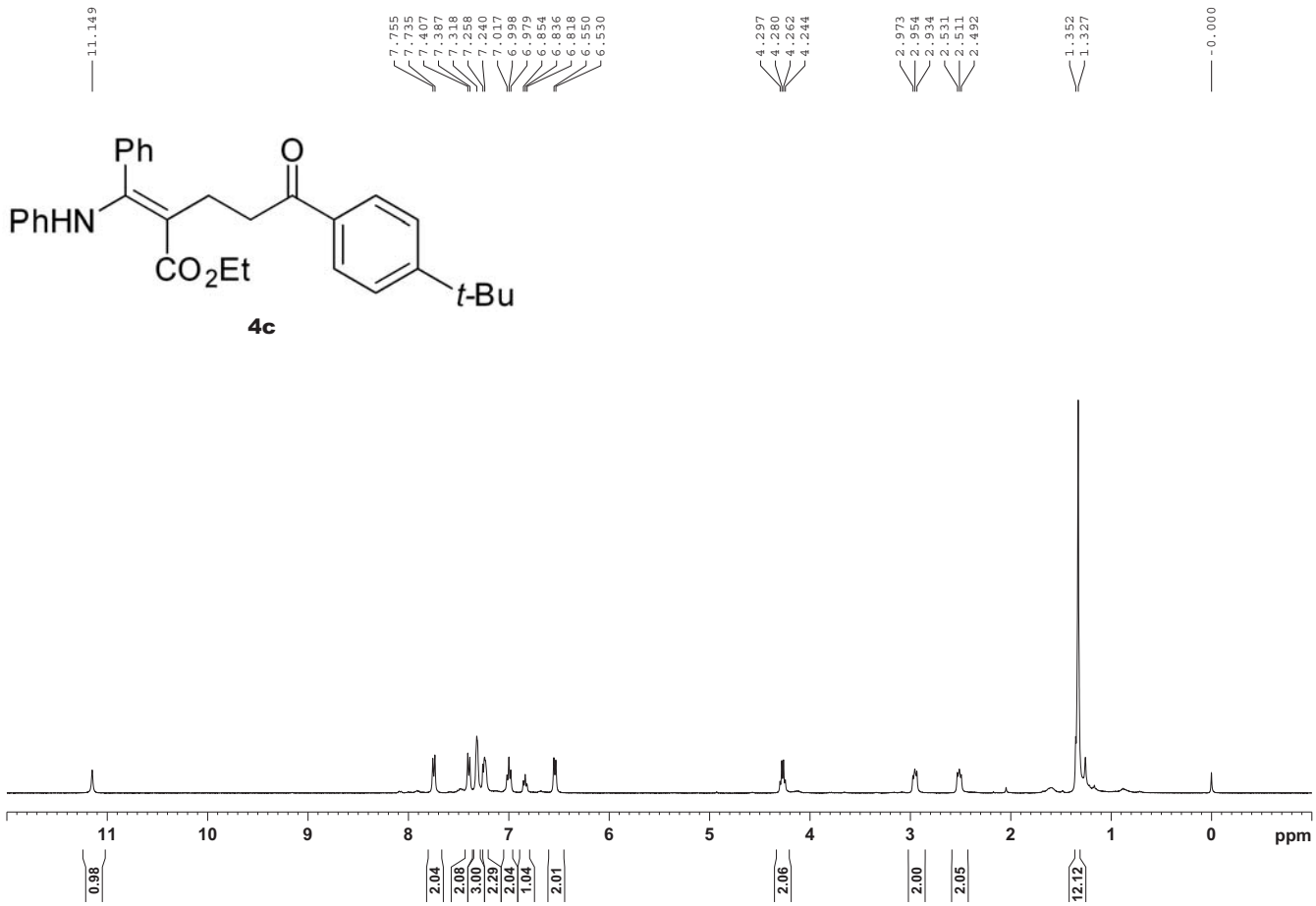
7.792
7.774
7.771
7.527
7.525
7.509
7.491
7.402
7.383
7.364
7.335
7.329
7.319
7.313
7.257
7.252
7.246
7.237
7.232
7.228
7.019
7.000
6.979
6.856
6.838
6.819
6.550
6.531
4.229
4.213
4.196
2.992
2.979
2.973
2.965
2.952
2.537
2.524
2.517
2.511
2.497
1.722
1.705
1.698
1.687
1.668
1.651
1.469
1.450
1.431
1.412
1.394
1.375
0.943
0.924
0.906
0.071
-0.000
Ph
O
PhHN
Ph
CO2nBu
3s
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.94
2.01
1.04
2.06
3.00
2.04
2.00
1.04
2.00
2.03
2.01
2.04
2.08
2.15
3.02


200.03
171.00
157.86
140.35
136.66
134.69
132.72
128.93
128.80
128.63
128.45
128.40
128.02
122.69
122.09
97.06
77.32
77.00
76.68
63.64
40.31
30.88
23.63
19.34
13.71
Ph
O
PhHN
Ph
CO2nBu
3s
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

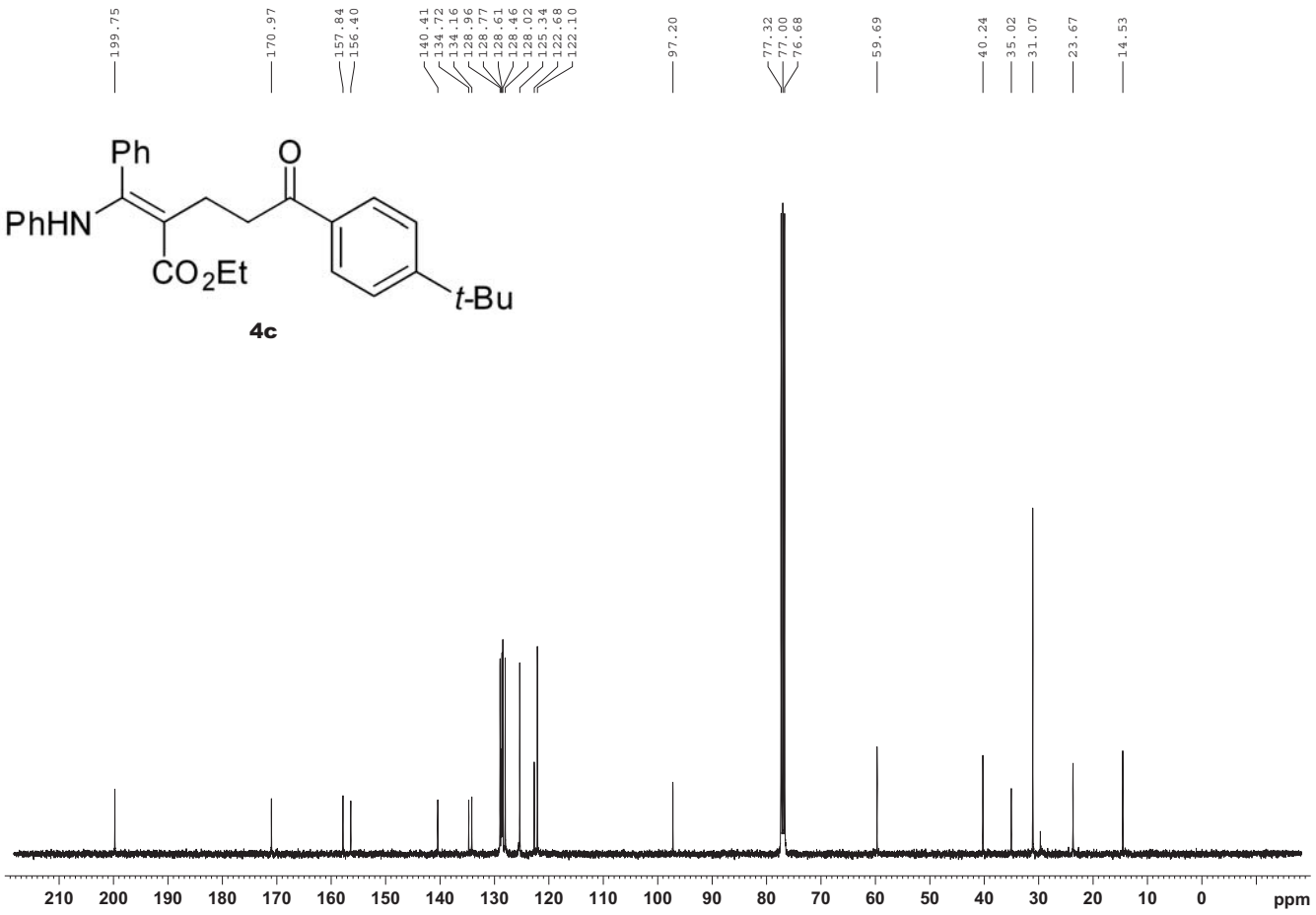

11.145
7.700
7.680
7.335
7.329
7.319
7.293
7.258
7.250
7.244
7.235
7.226
7.187
7.167
7.017
6.998
6.978
6.854
6.836
6.817
6.549
6.529
4.295
4.277
4.260
4.242
2.955
2.942
2.936
2.928
2.915
2.526
2.514
2.505
2.499
2.486
2.384
1.354
1.336
1.318
-0.000
Ph
O
PhHN
CO2Et
Me
4b
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.99
2.01
3.07
2.25
2.03
2.03
1.01
1.99
2.00
2.00
2.03
3.05
3.01

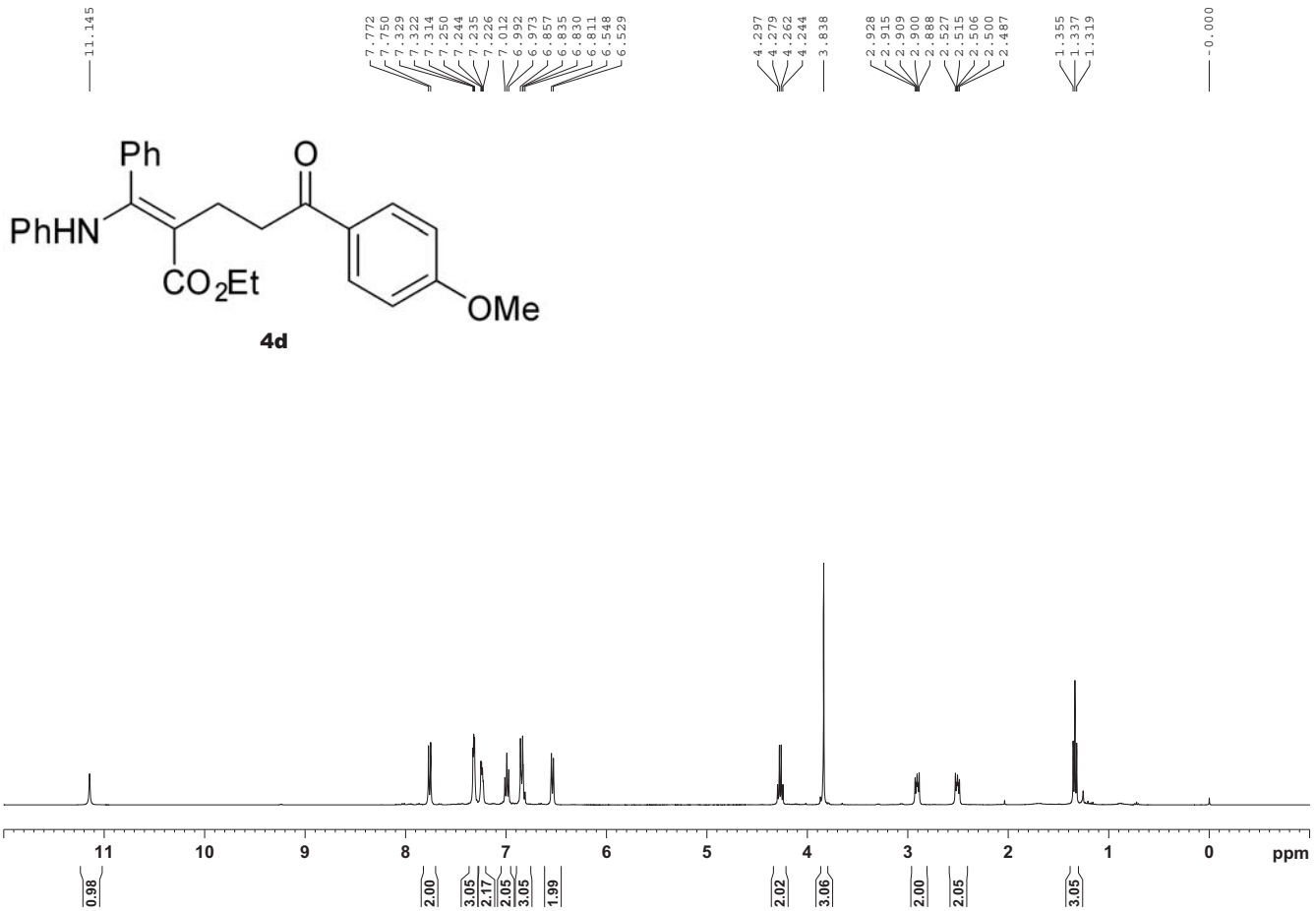

199.82
170.96
157.86
143.45
140.40
134.73
134.25
129.10
128.97
128.80
128.63
128.47
128.21
122.70
122.12
97.19
77.32
77.00
76.69
59.70
40.26
23.79
21.58
14.53
Ph
O
PhHN
CO2Et
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

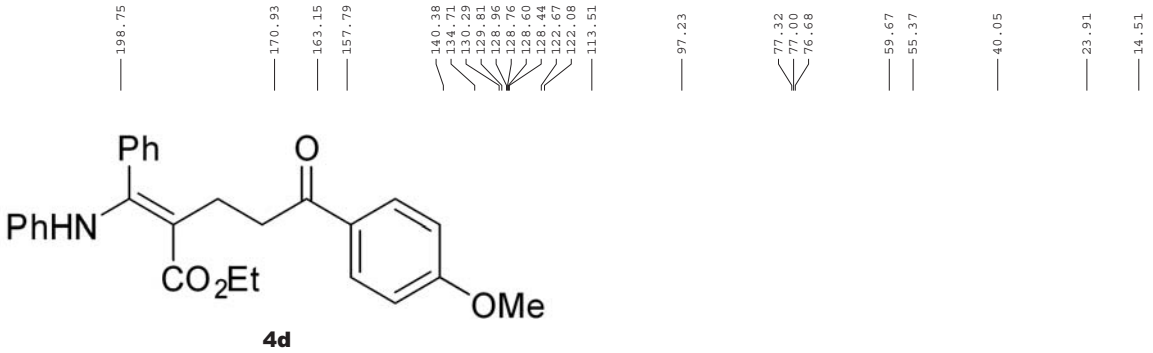
4b


11.149
7.755
7.735
7.407
7.387
7.318
7.258
7.240
7.017
6.998
6.979
6.854
6.836
6.818
6.550
6.530
4.297
4.280
4.262
4.244
2.973
2.954
2.934
2.531
2.511
2.492
1.352
1.327
-0.000
Ph
O
PhHN
CO2Et
t-Bu
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.98
2.04
2.08
3.00
2.29
2.04
1.04
2.01
2.06
2.00
2.05
12.12

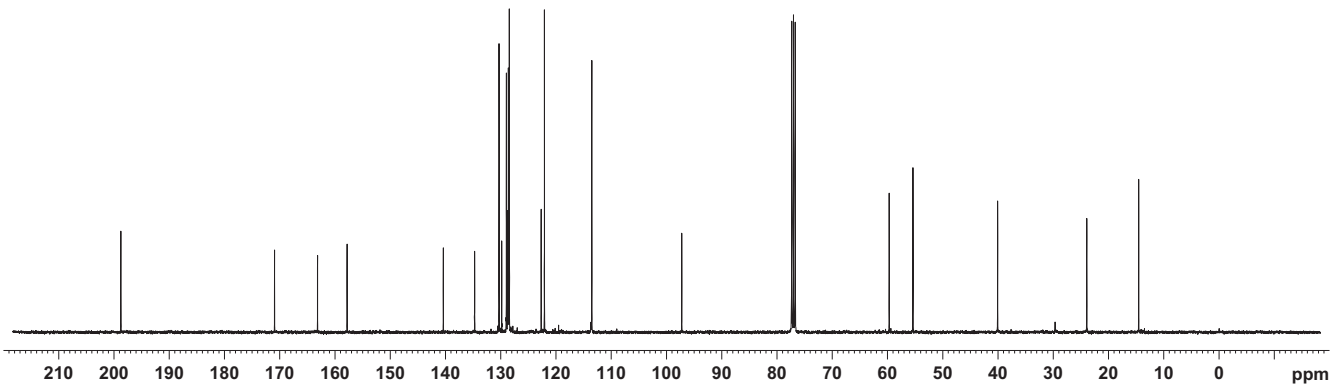
4c

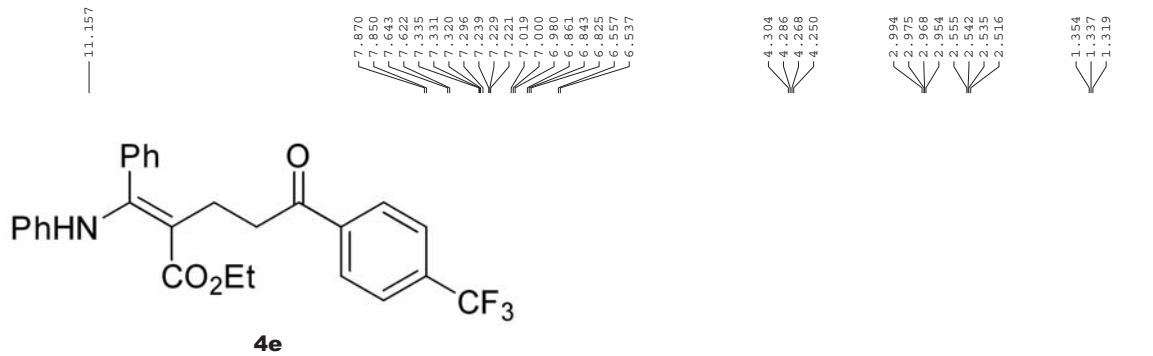

199.75
170.97
157.84
156.40
140.41
134.72
134.16
128.96
128.77
128.61
128.46
128.02
125.34
122.68
122.10
97.20
77.32
77.00
76.68
59.69
40.24
35.02
31.07
23.67
14.53
Ph
O
PhHN
CO2Et
t-Bu
4c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


11.145
7.772
7.750
7.329
7.322
7.314
7.250
7.244
7.235
7.226
7.012
6.992
6.973
6.857
6.835
6.830
6.811
6.548
6.529
4.297
4.279
4.262
4.244
3.838
2.928
2.915
2.909
2.900
2.888
2.527
2.515
2.506
2.500
2.487
1.355
1.337
1.319
-0.000
Ph
O
PhHN
CO2Et
OMe
4d
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.98
2.00
3.05
2.17
2.05
3.05
1.99
2.02
3.06
2.00
2.05
3.05

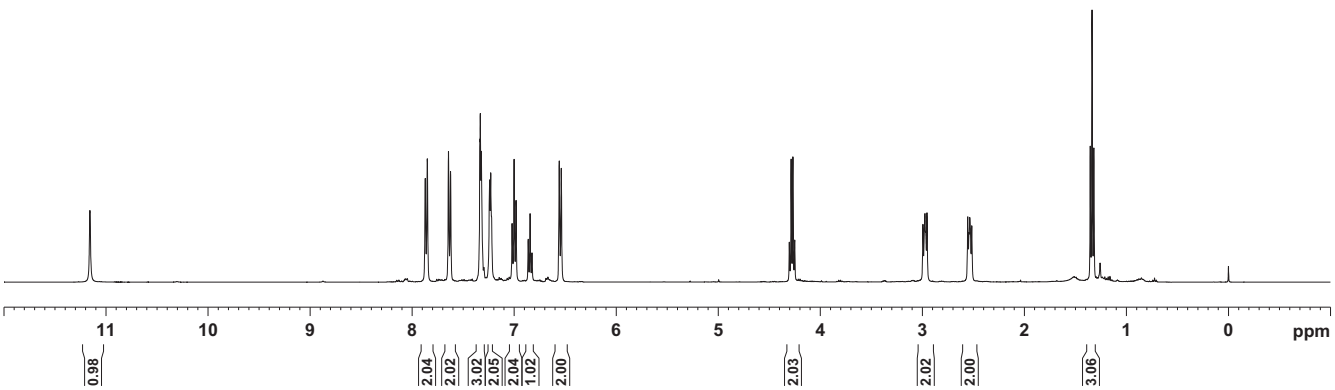

198.75
170.93
163.15
157.79
140.38
134.71
130.29
129.81
128.96
128.76
128.60
128.44
122.67
122.08
113.51
97.23
77.32
77.00
76.68
59.67
55.37
40.05
23.91
14.51
Ph
O
PhHN
CO2Et
OMe

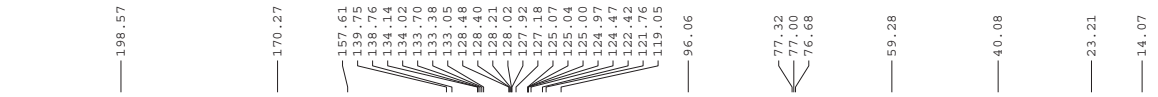
4d

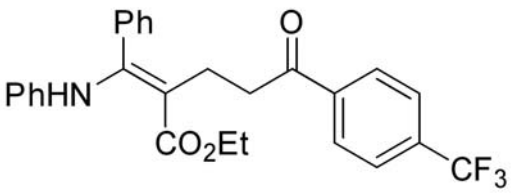

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


11.157
7.870
7.850
7.643
7.622
7.335
7.331
7.320
7.296
7.239
7.229
7.221
7.019
7.000
6.980
6.861
6.843
6.825
6.557
6.537
4.304
4.286
4.268
4.250
2.994
2.975
2.968
2.954
2.555
2.542
2.535
2.516
1.354
1.337
1.319
0.000
Ph
O
PhHN
CO2Et
CF3

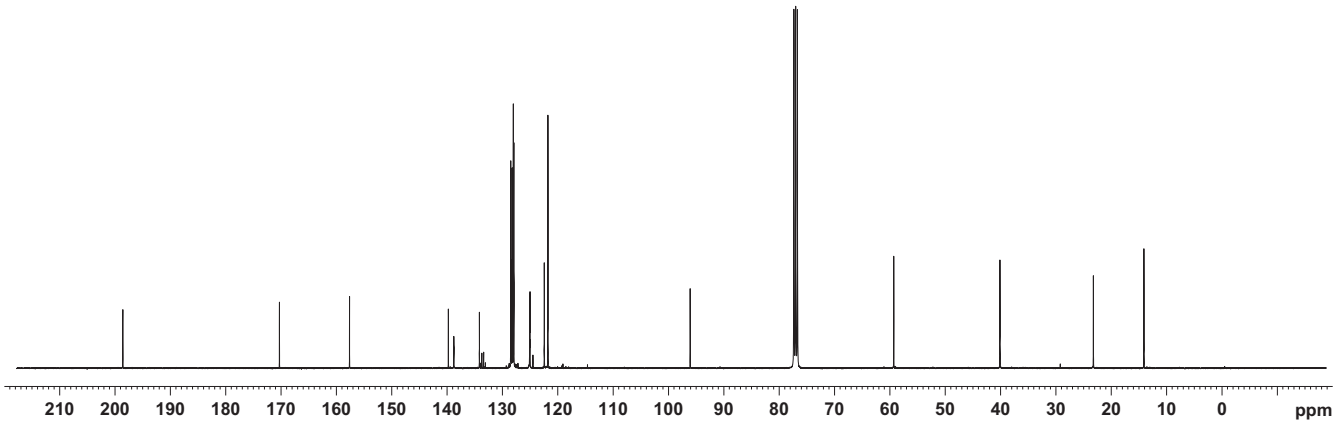
4e


11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.98
2.04
2.02
3.02
2.05
2.04
1.02
2.00
2.03
2.02
2.00
3.06

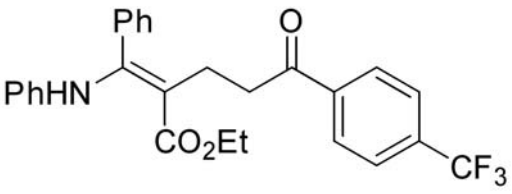

198.57
170.27
157.61
139.75
138.76
134.14
134.02
133.70
133.38
133.05
128.48
128.40
128.21
128.02
127.92
127.18
125.07
125.04
125.00
124.97
124.47
122.42
121.76
119.05
96.06
77.32
77.00
76.68
59.28
40.08
23.21
14.07


Ph
O
PhHN
CO2Et
CF3

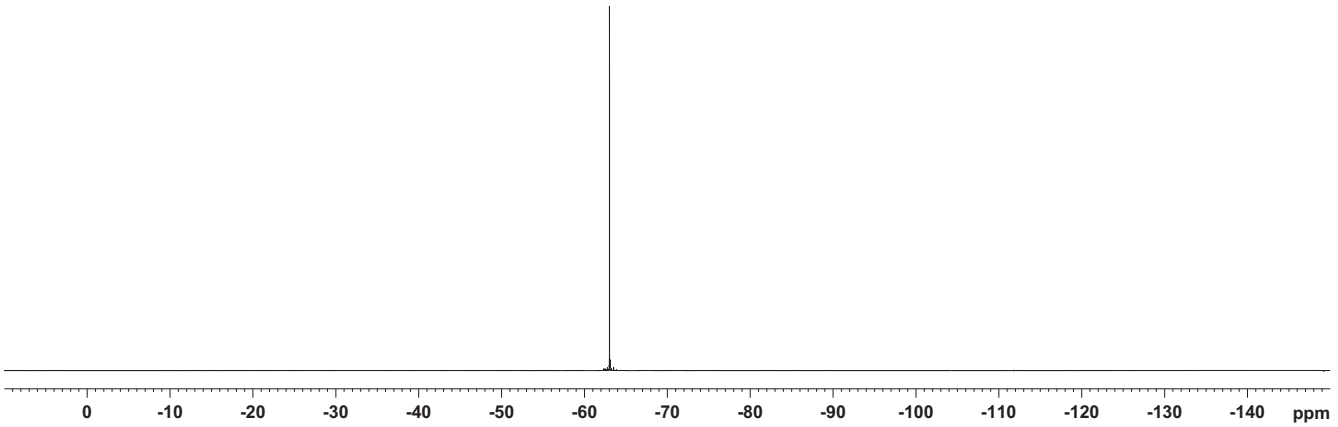
4e

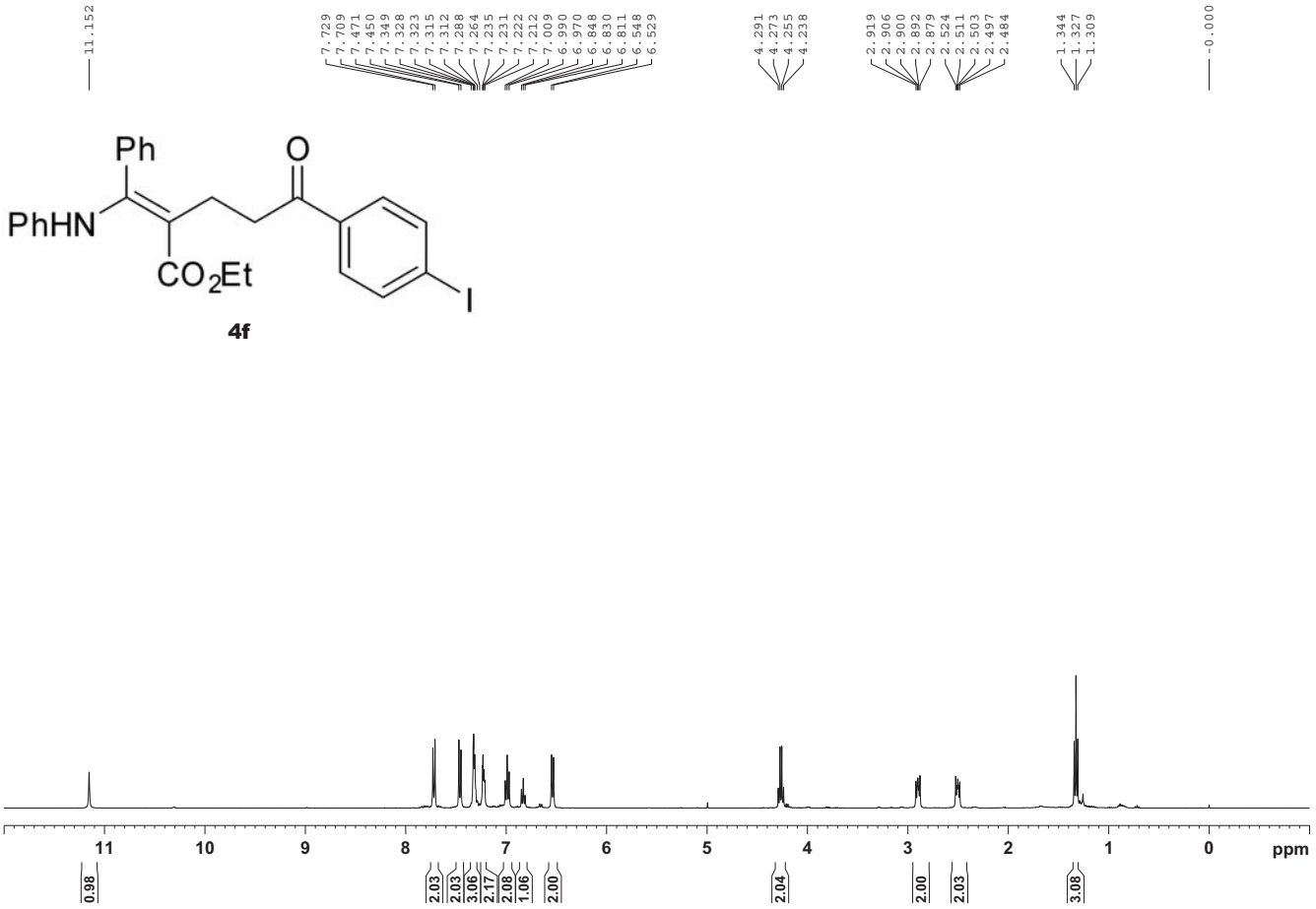

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

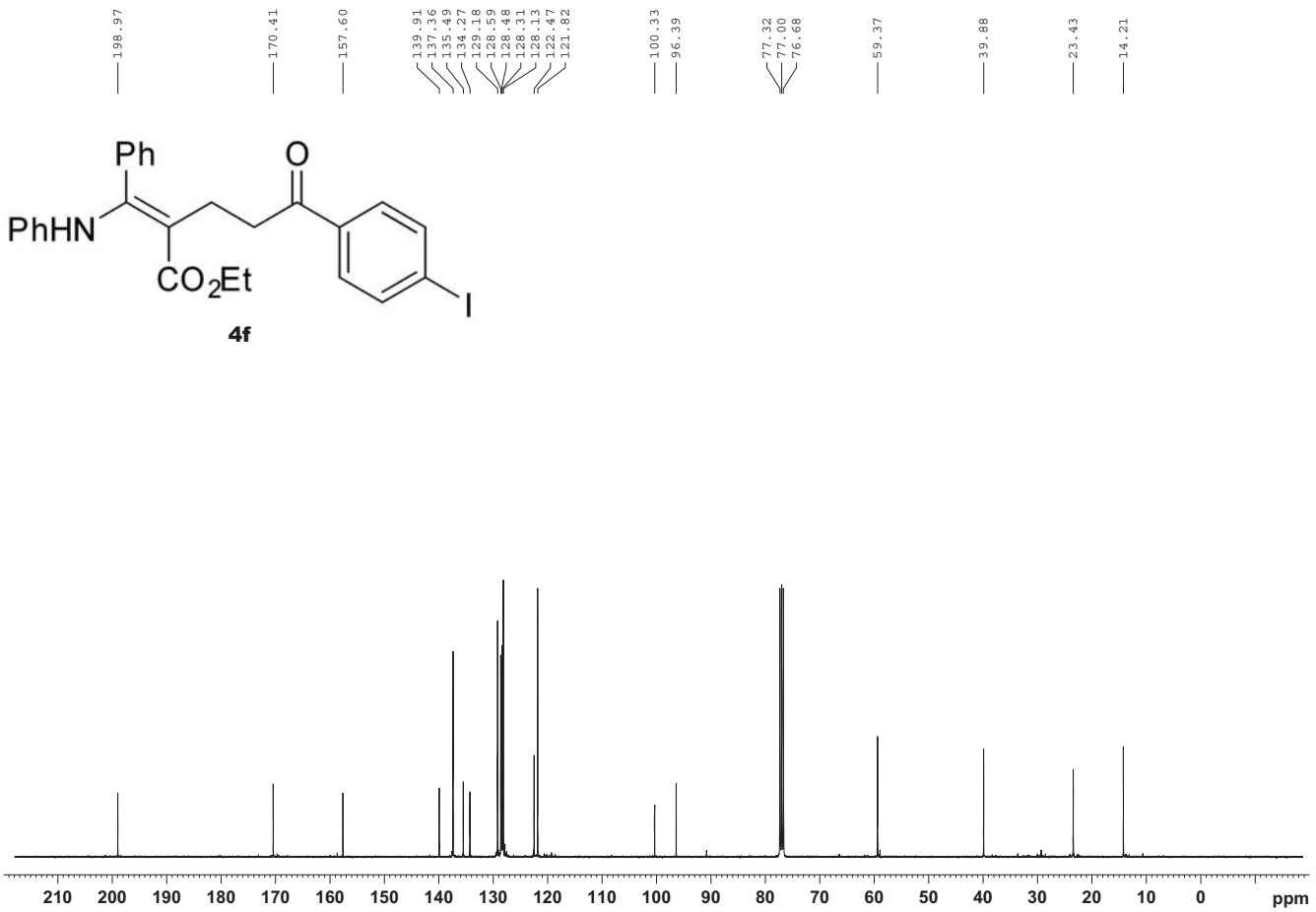

-63.02


Ph
O
PhHN
CO2Et
CF3

4e


0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
ppm


11.152
7.729
7.709
7.471
7.450
7.349
7.328
7.323
7.315
7.312
7.288
7.264
7.235
7.231
7.222
7.212
7.009
6.990
6.970
6.848
6.830
6.811
6.548
6.529
4.291
4.273
4.255
4.238
2.919
2.906
2.900
2.892
2.879
2.524
2.511
2.503
2.497
2.484
1.344
1.327
1.309
-0.000
Ph
O
PhHN
CO2Et
I
4f
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.98
2.03
2.03
3.06
2.17
2.08
1.06
2.00
2.04
2.00
2.03
3.08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128.13
122.47
121.82
100.33
96.39
77.32
77.00
76.68
59.37
39.88
23.43
14.21
Ph
O
PhHN
CO2Et
I
4f
210
200
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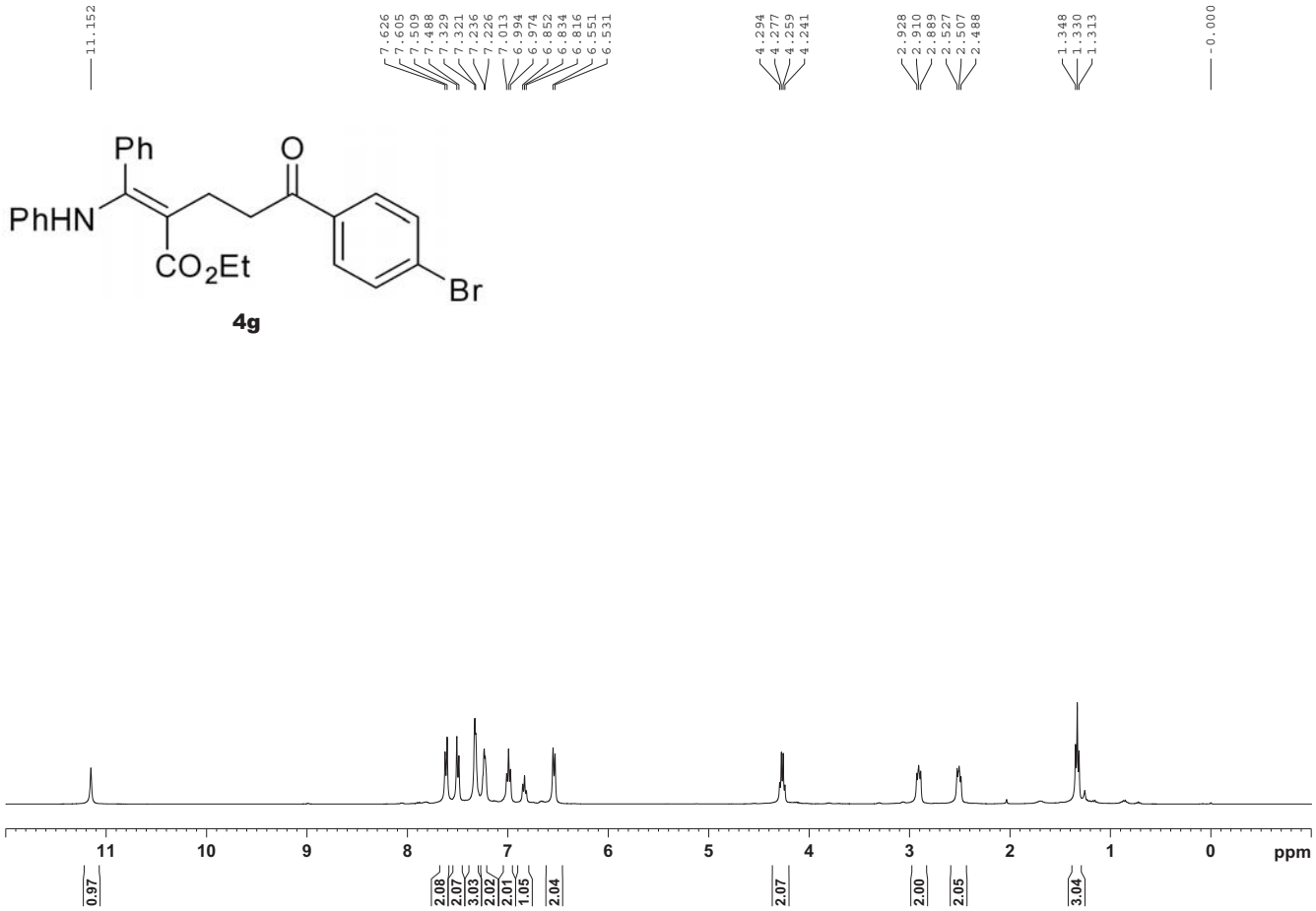
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7.321
7.236
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6.852
6.834
6.816
6.551
6.531
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4.277
4.259
4.241
2.928
2.910
2.889
2.527
2.507
2.488
1.348
1.330
1.313
0.000
Ph
O
PhHN
CO2Et
Br
4g
11
10
9
8
7
6
5
4
3
2
1
0
ppm
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1.05
2.04
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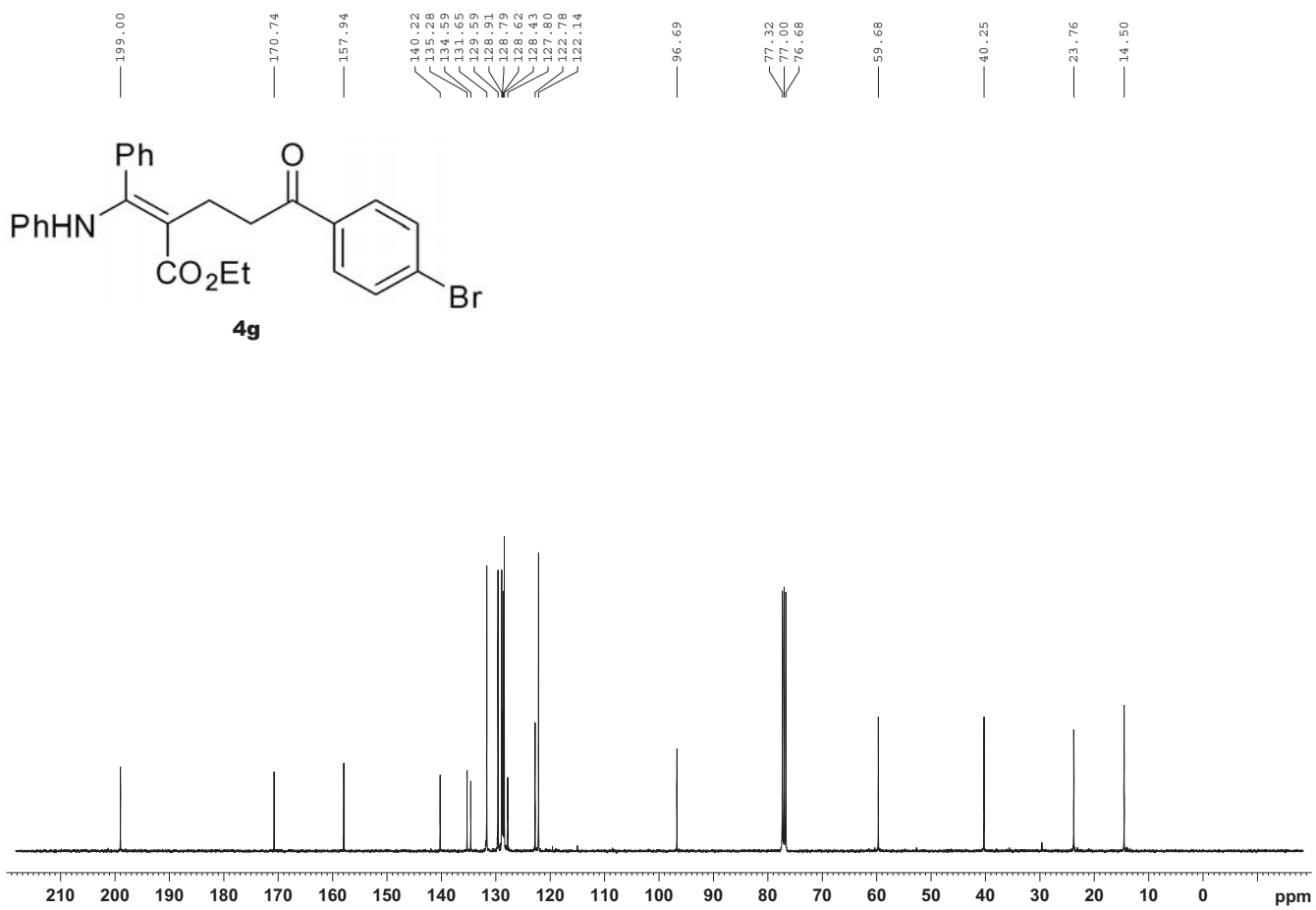
2.05
3.04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127.80
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122.14
96.69
77.32
77.00
76.68
59.68
40.25
23.76
14.50
Ph
O
PhHN
CO2Et
Br
4g
210
200
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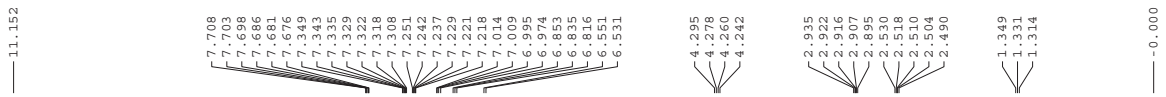
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7.242
7.237
7.229
7.221
7.218
7.014
7.009
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6.853
6.835
6.816
6.551
6.531
4.295
4.278
4.260
4.242
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2.922
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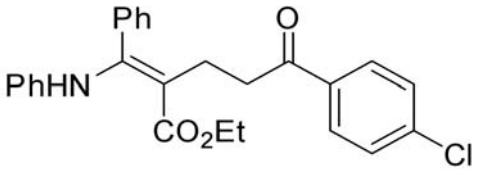
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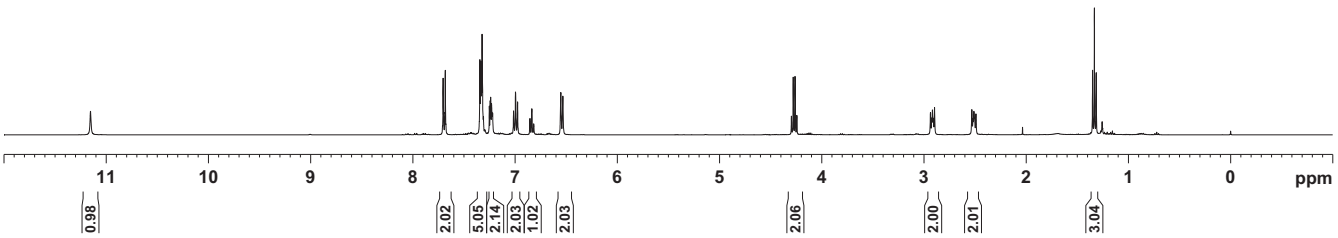
CO2Et
Cl

4h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6
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3
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ppm
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5.05
2.14
2.03
1.02
2.03
2.06
2.00
2.01
3.04


198.82
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157.94
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139.07
134.89
134.61
129.47
128.92
128.80
128.66
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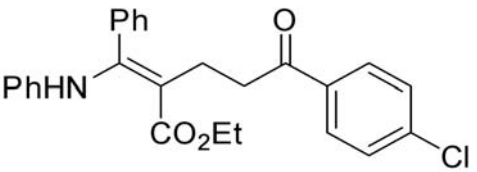
23.77
14.50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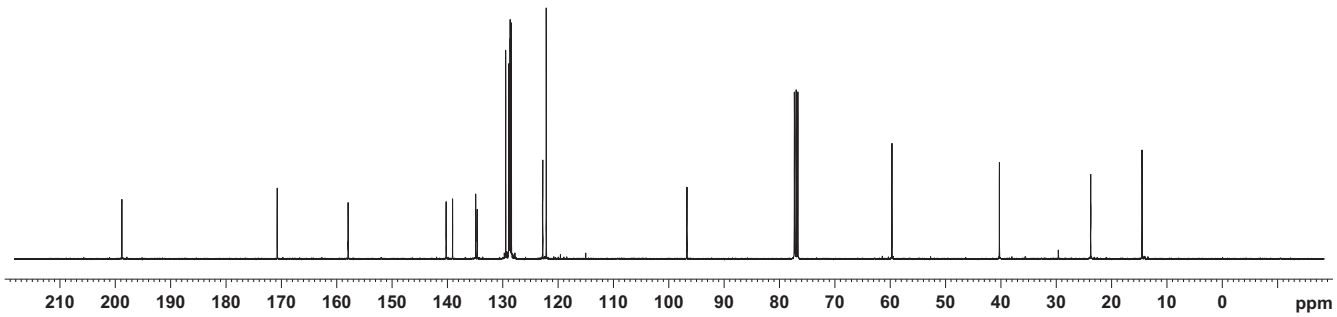
CO2Et
Cl

4h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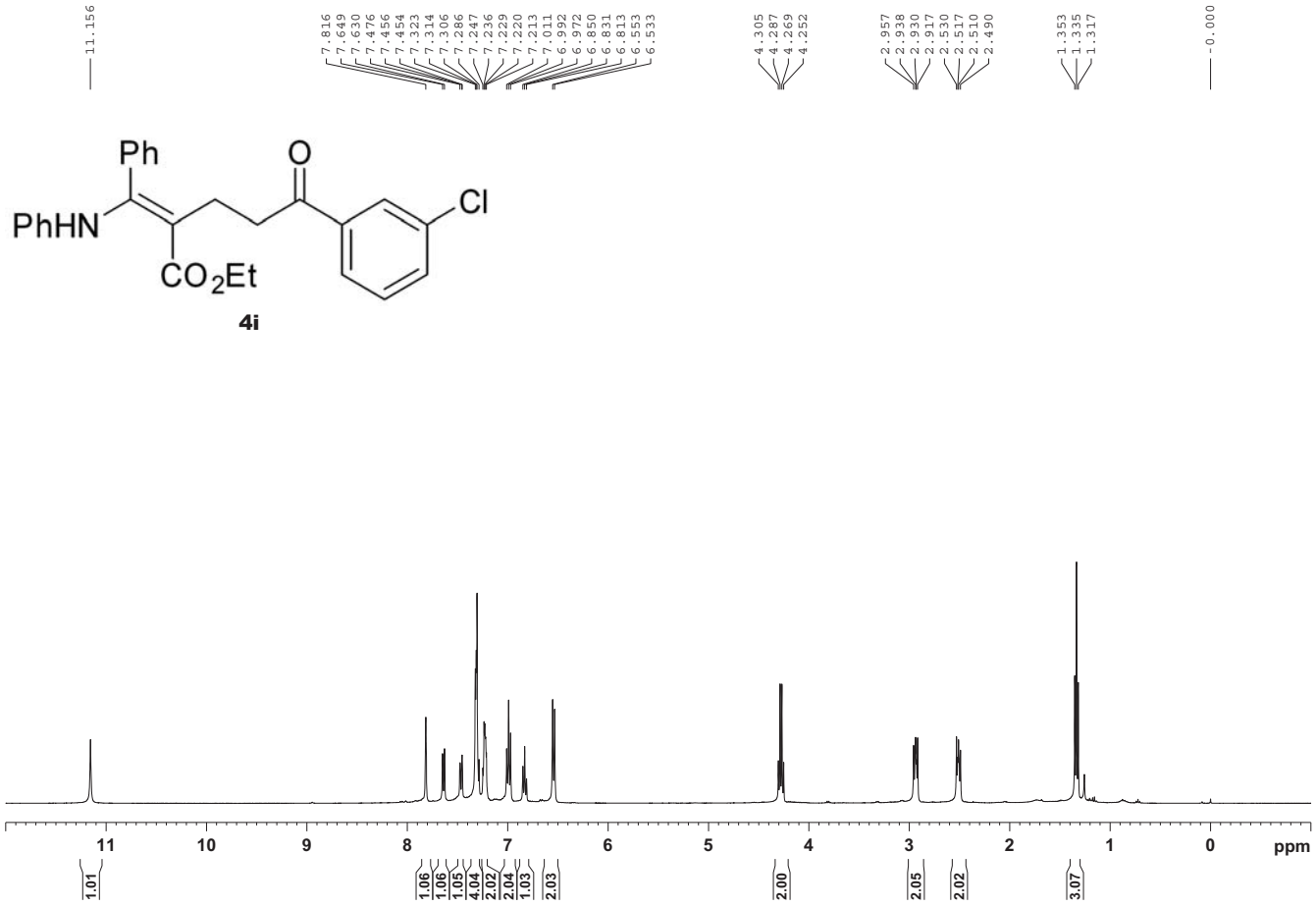
ppm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7.236
7.229
7.220
7.213
7.011
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6.972
6.850
6.831
6.813
6.553
6.533
4.305
4.287
4.269
4.252
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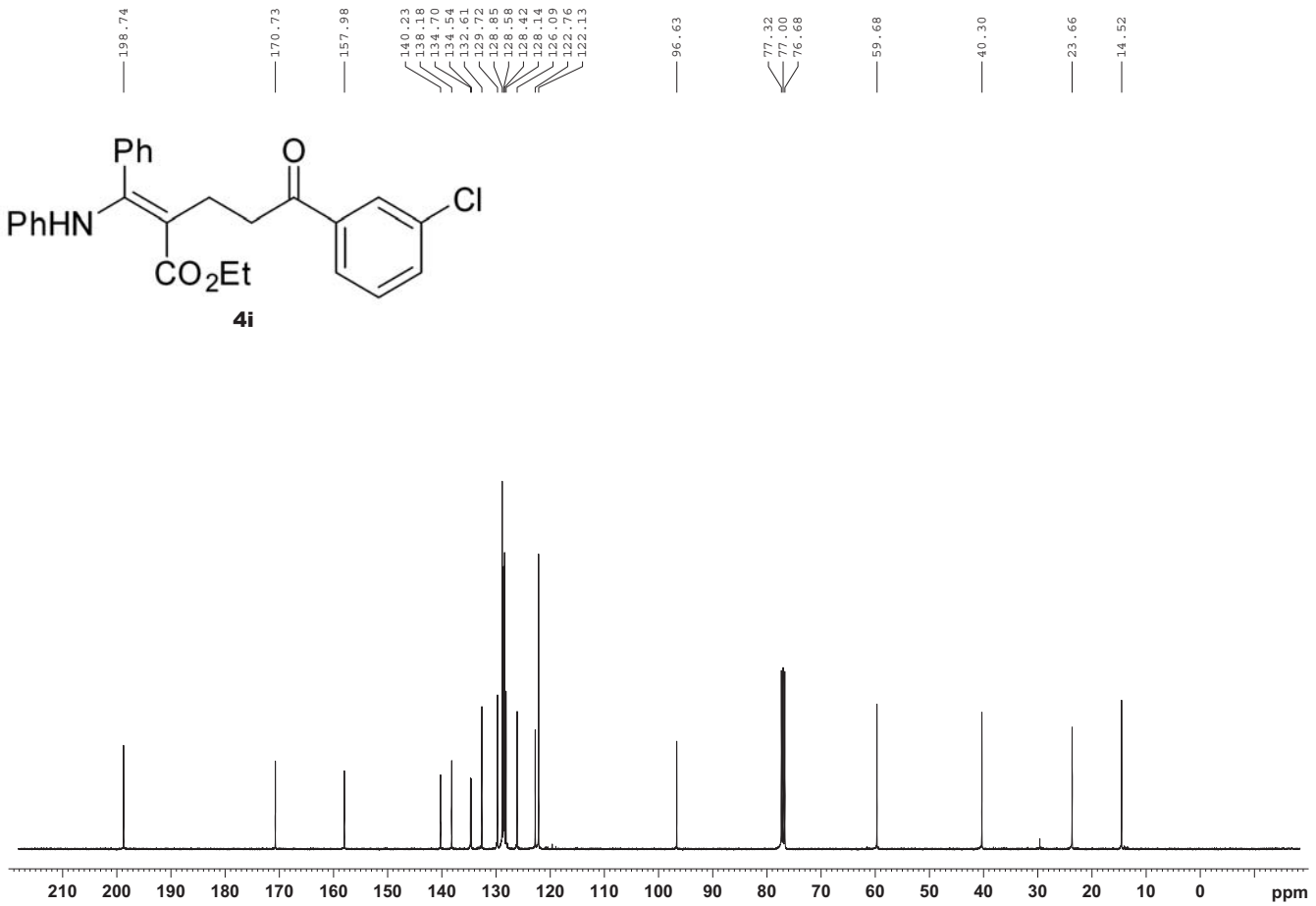
2.938
2.930
2.917
2.530
2.517
2.510
2.490
1.353
1.335
1.317
-0.000
Ph
O
PhHN
Cl
CO2Et
4i
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.01
1.06
1.06
1.05
4.04
2.02
2.04
1.03
2.03
2.00
2.05
2.02
3.07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128.42
128.14
126.09
122.76
122.13
96.63
77.32
77.00
76.68
59.68
40.30
23.66
14.52
Ph
O
PhHN
Cl
CO2Et
4i
210
200
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180
170
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140
130
120
110
100
90
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60
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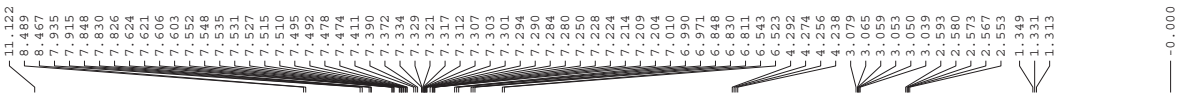
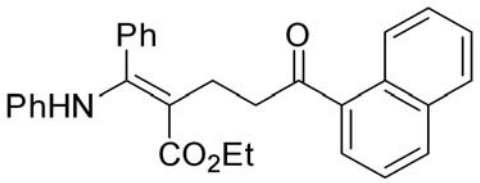
20
10
0
ppm

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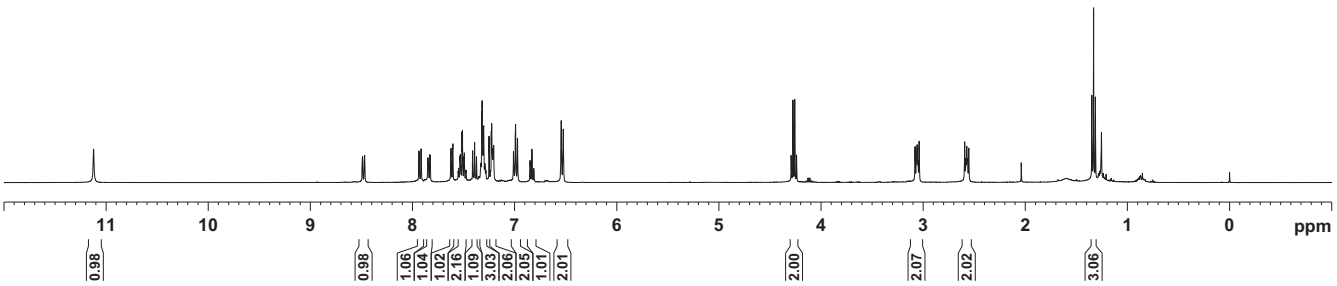
PhHN
CO2Et

4j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9
8
7
6
5
4
3
2
1
0
ppm
0.98
0.98
1.06
1.04
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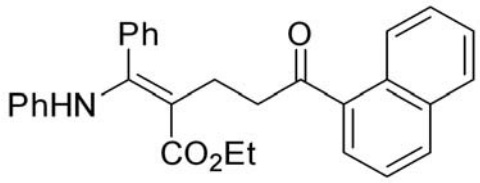
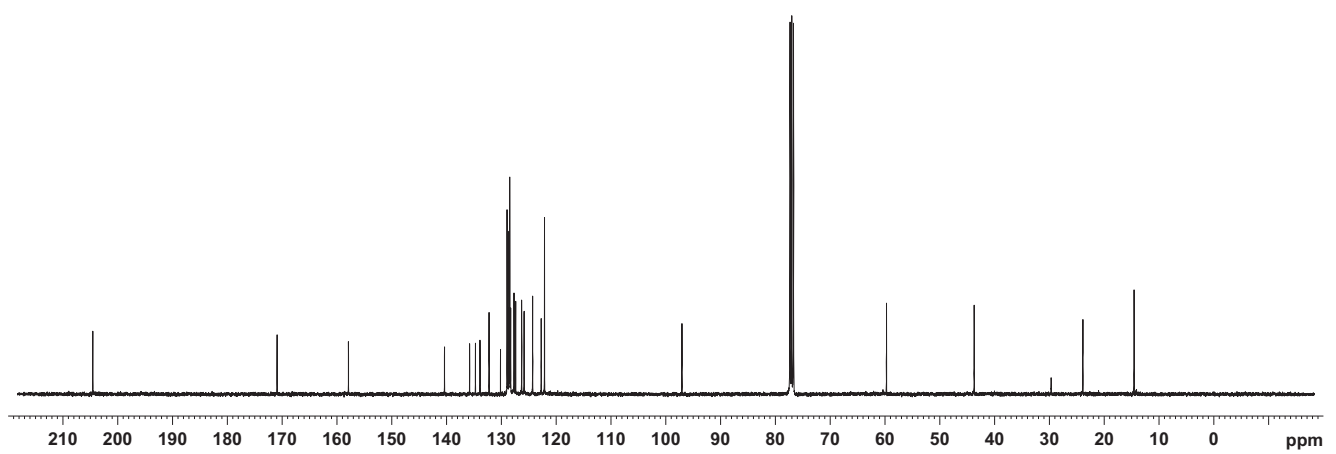
1.09
3.03
2.06
2.05
1.01
2.01
2.00
2.07
2.02
3.06


Ph
O
PhHN
CO2Et

4j


210
200
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180
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160
150
140
130
120
110
100
90
80
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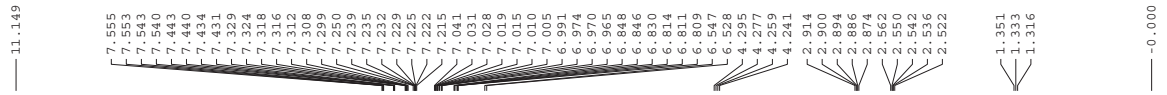
60
50
40
30
20
10
0
ppm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7.312
7.308
7.299
7.250
7.239
7.235
7.232
7.229
7.225
7.222
7.215
7.041
7.031
7.028
7.019
7.015
7.010
7.005
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6.974
6.970
6.965
6.848
6.846
6.830
6.814
6.811
6.809
6.547
6.528
4.295
4.277
4.259
4.241
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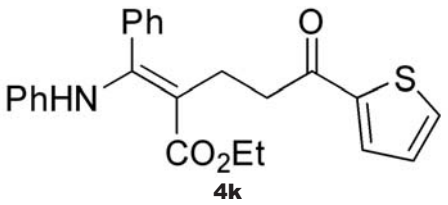
1.333
1.316
-0.000


Ph
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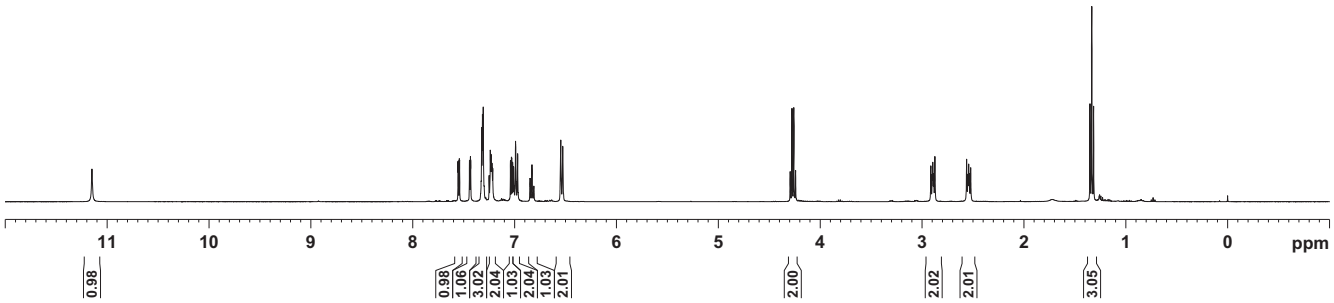
PhHN
S
CO2Et

4k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8
7
6
5
4
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2
1
0
ppm
0.98
0.98
1.06
3.02
2.04
1.03
2.04
1.03
2.01
2.00
2.02
2.01
3.05


193.03
170.80
157.93
144.28
140.27
134.58
133.14
131.63
128.91
128.79
128.59
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127.84
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122.10
96.84
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77.00
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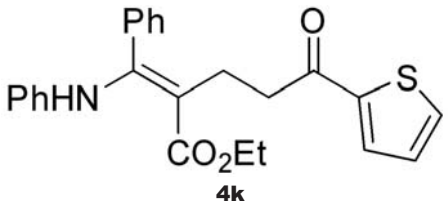
59.67
40.97
24.01
14.49


Ph
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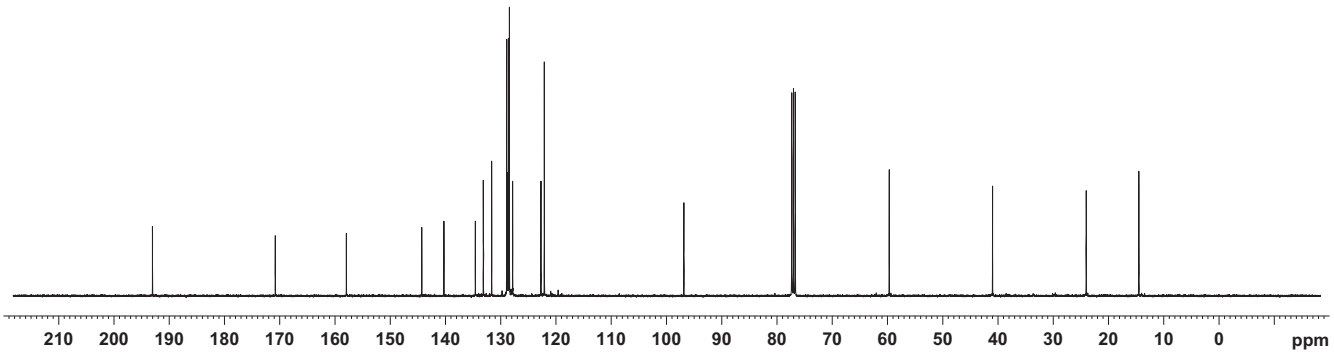
PhHN
S
CO2Et

4k


210
200
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60
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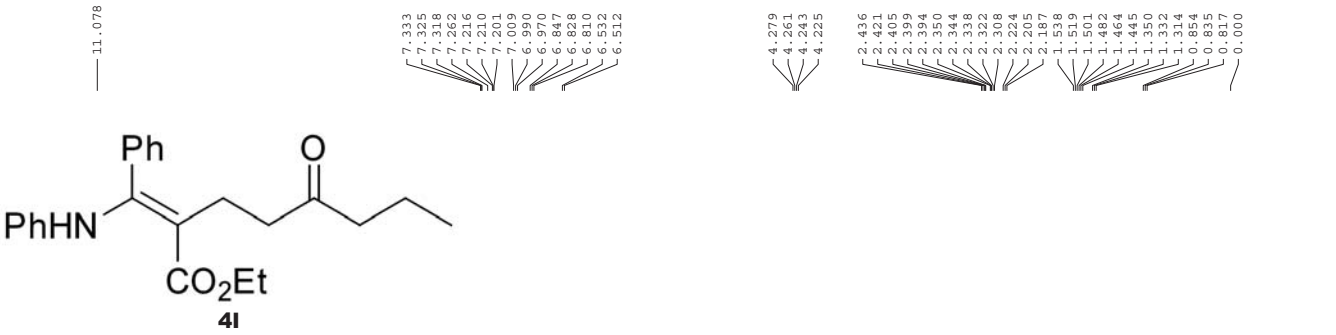
0
ppm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6.847
6.828
6.810
6.532
6.512
4.279
4.261
4.243
4.225
2.436
2.421
2.405
2.399
2.394
2.350
2.344
2.338
2.322
2.308
2.224
2.205
2.187
1.538
1.519
1.501
1.482
1.464
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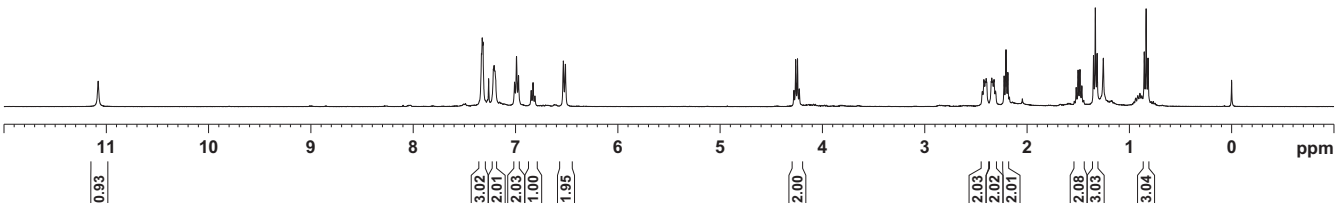
1.332
1.314
0.854
0.835
0.817
0.000

Ph
O
PhHN
CO2Et

4l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1
0
ppm
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1.00
1.95
2.00
2.03
2.02
2.01
2.08
3.03
3.04


211.15
170.86
157.66
140.40
134.75
128.91
128.79
128.54
128.45
122.65
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77.00
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59.62
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22.58
17.33
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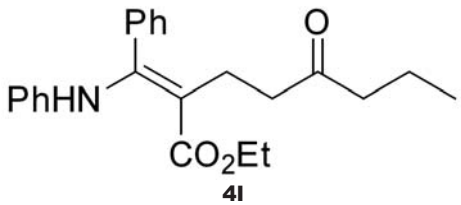
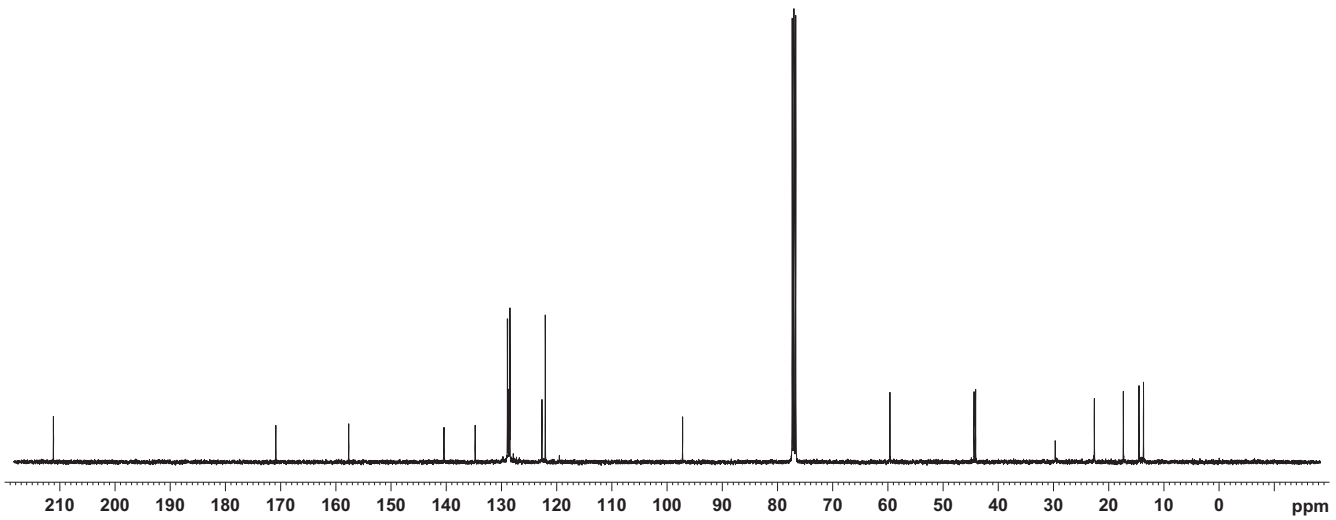
13.68


Ph
O
PhHN
CO2Et

4l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140
130
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100
90
80
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60
50
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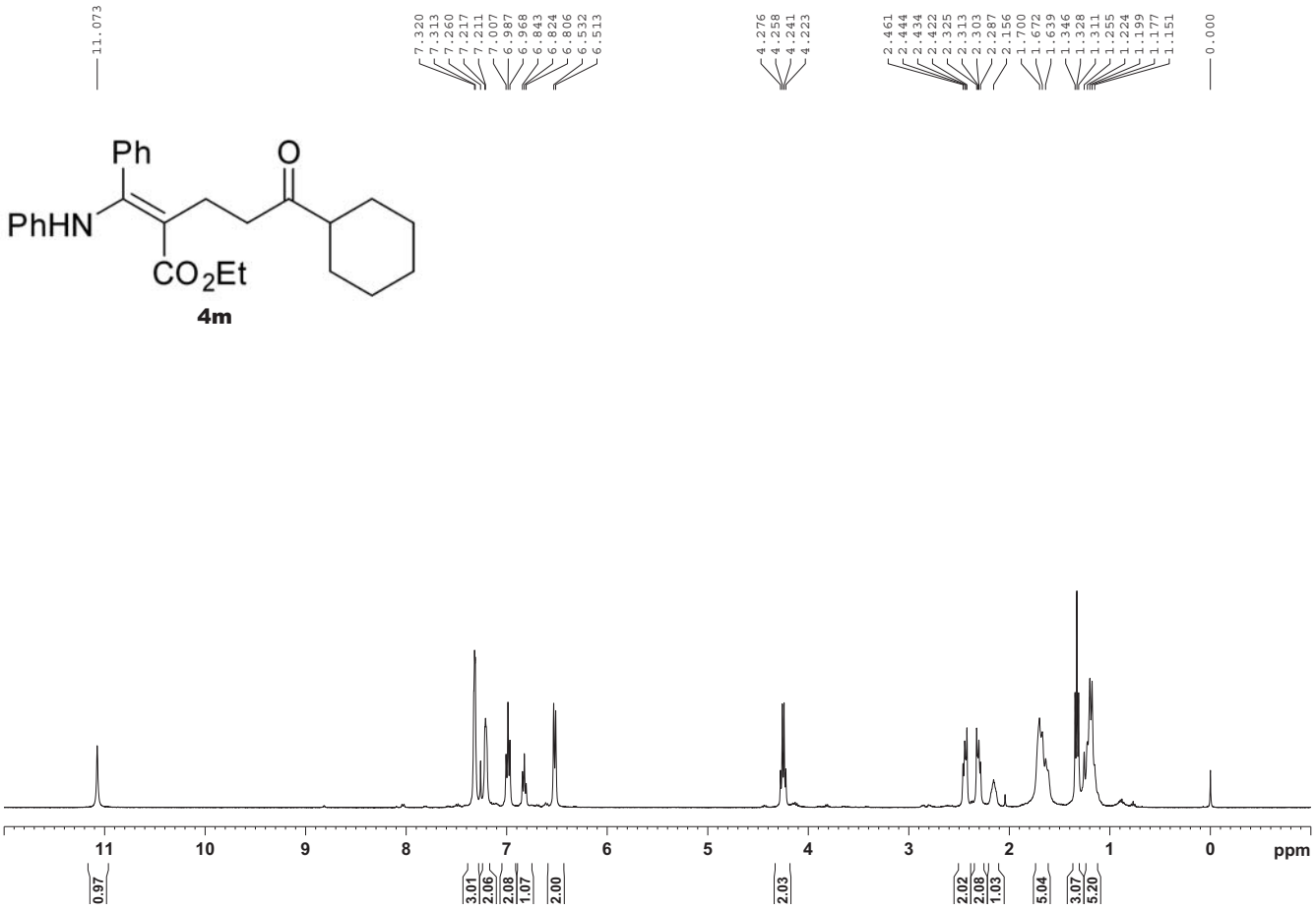
30
20
10
0
ppm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7.217
7.211
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6.968
6.843
6.824
6.806
6.532
6.513
4.276
4.258
4.241
4.223
2.461
2.444
2.434
2.422
2.325
2.313
2.303
2.287
2.156
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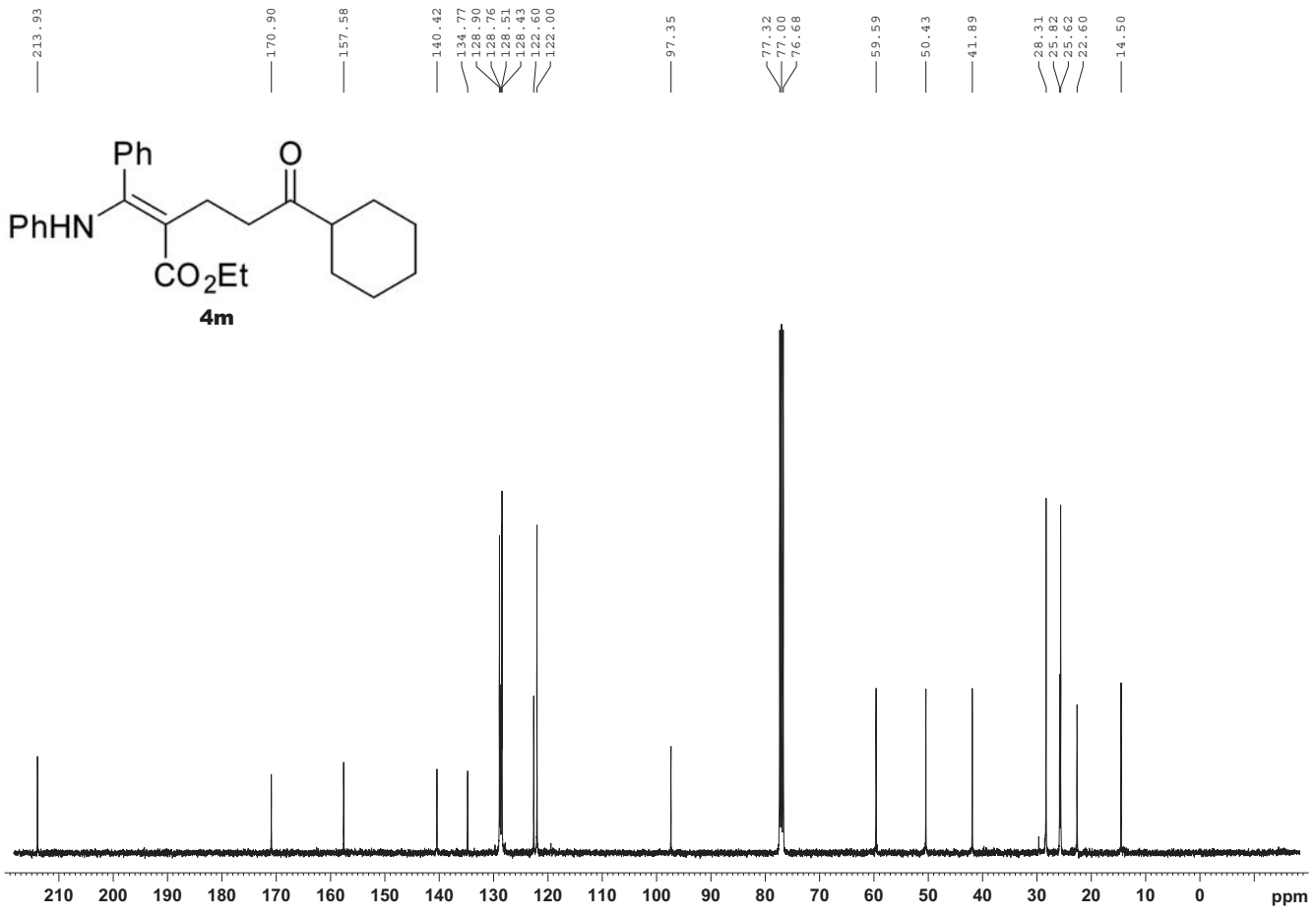
1.672
1.639
1.346
1.328
1.311
1.255
1.224
1.199
1.177
1.151
0.000
Ph
O
PhHN
CO2Et
4m
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.97
3.01
2.06
2.08
1.07
2.00
2.03
2.02
2.08
1.03
5.04
3.07
5.20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122.60
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97.35
77.32
77.00
76.68
59.59
50.43
41.89
28.31
25.82
25.62
22.60
14.50
Ph
O
PhHN
CO2Et
4m
210
200
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50
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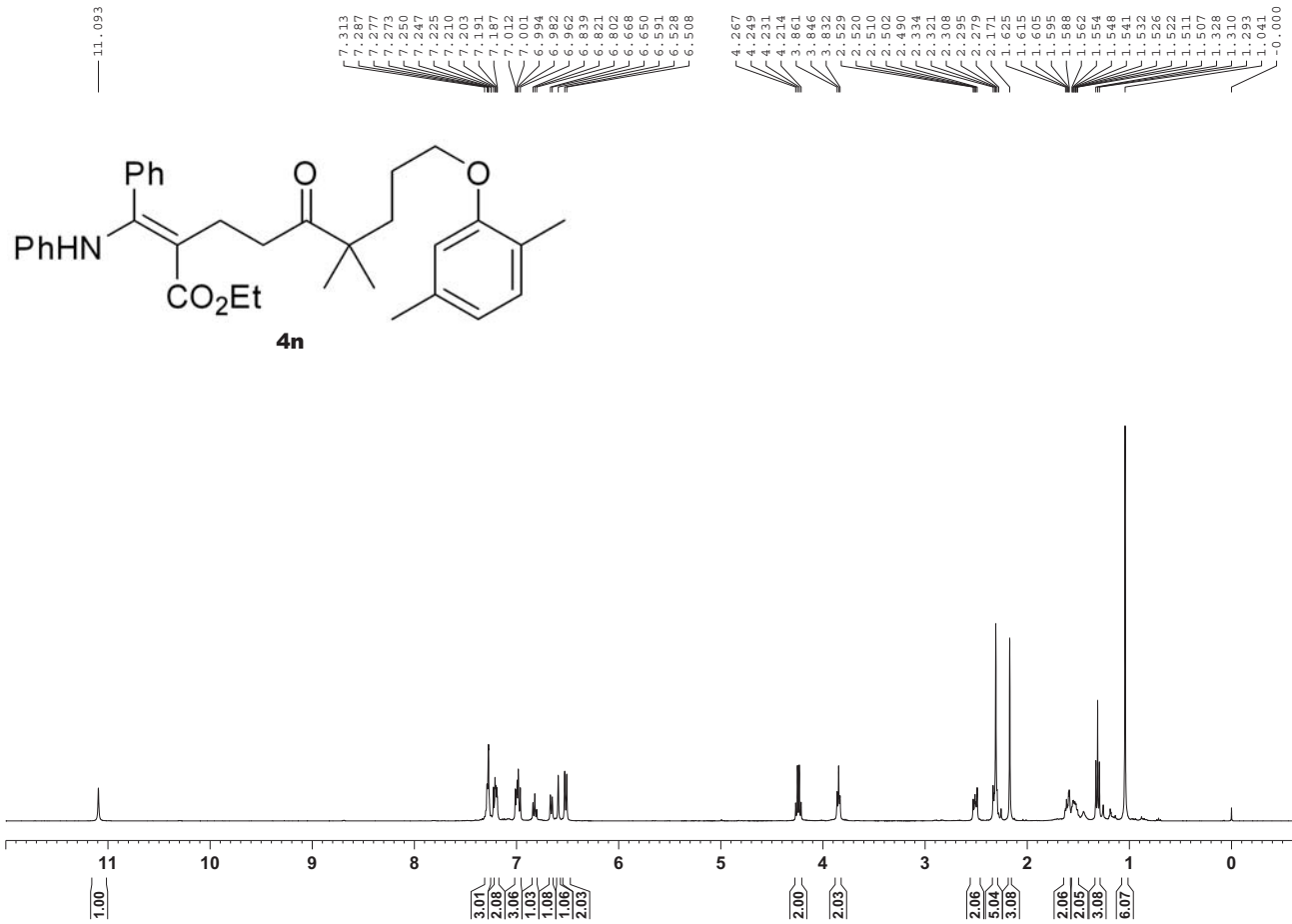
ppm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7.203
7.191
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7.012
7.001
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6.591
6.528
6.508
4.267
4.249
4.231
4.214
3.861
3.846
3.832
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2.490
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2.321
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2.295
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1.605
1.595
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1.562
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1.541
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1.522
1.511
1.507
1.328
1.310
1.293
1.041
-0.000
Ph
O
O
PhHN
CO2Et
4n
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
3.01
2.08
3.06
1.03
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1.06
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2.00
2.03
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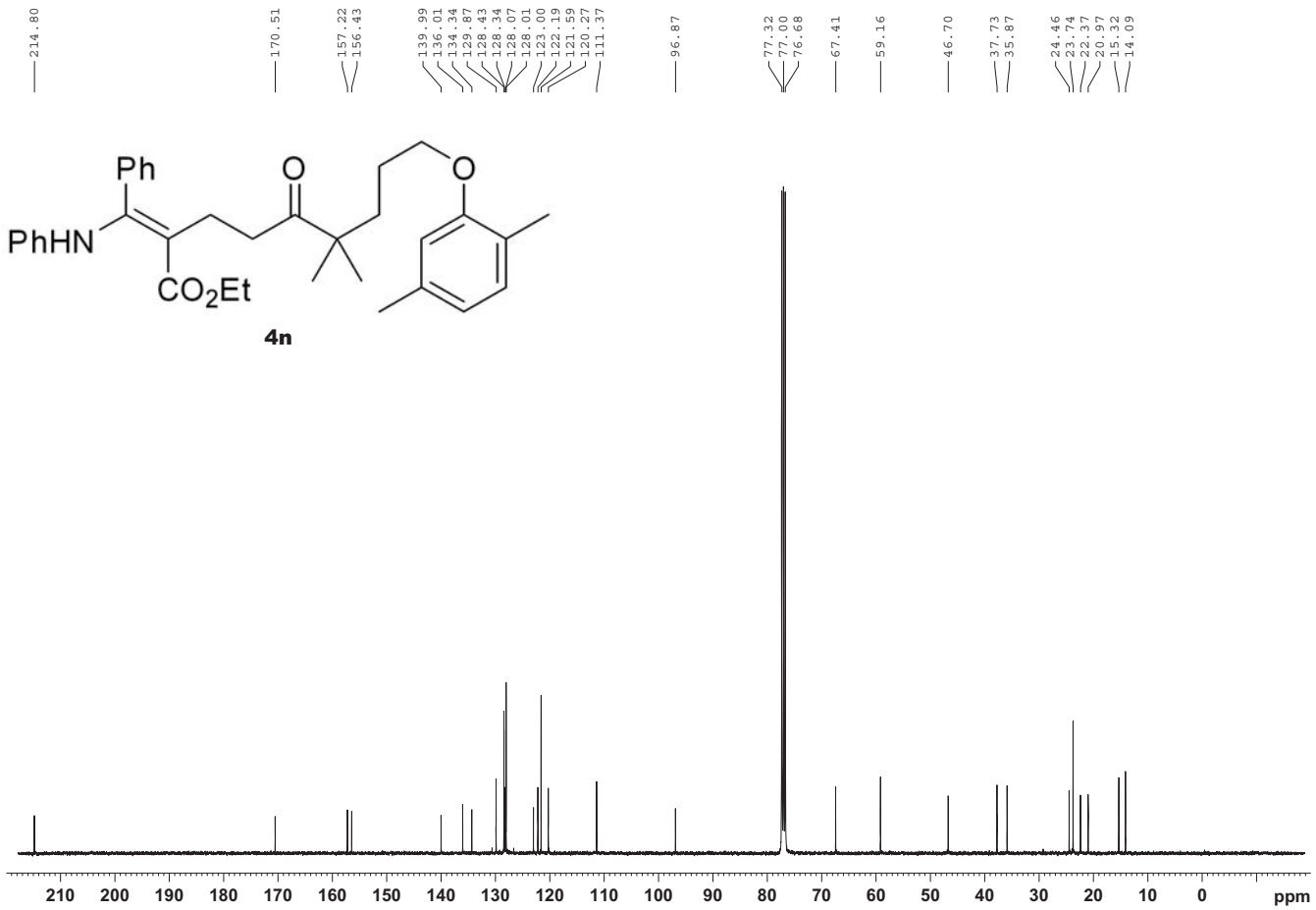
5.04
3.08
2.06
2.05
3.08
6.07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128.34
128.07
128.01
123.00
122.19
121.59
120.27
111.37
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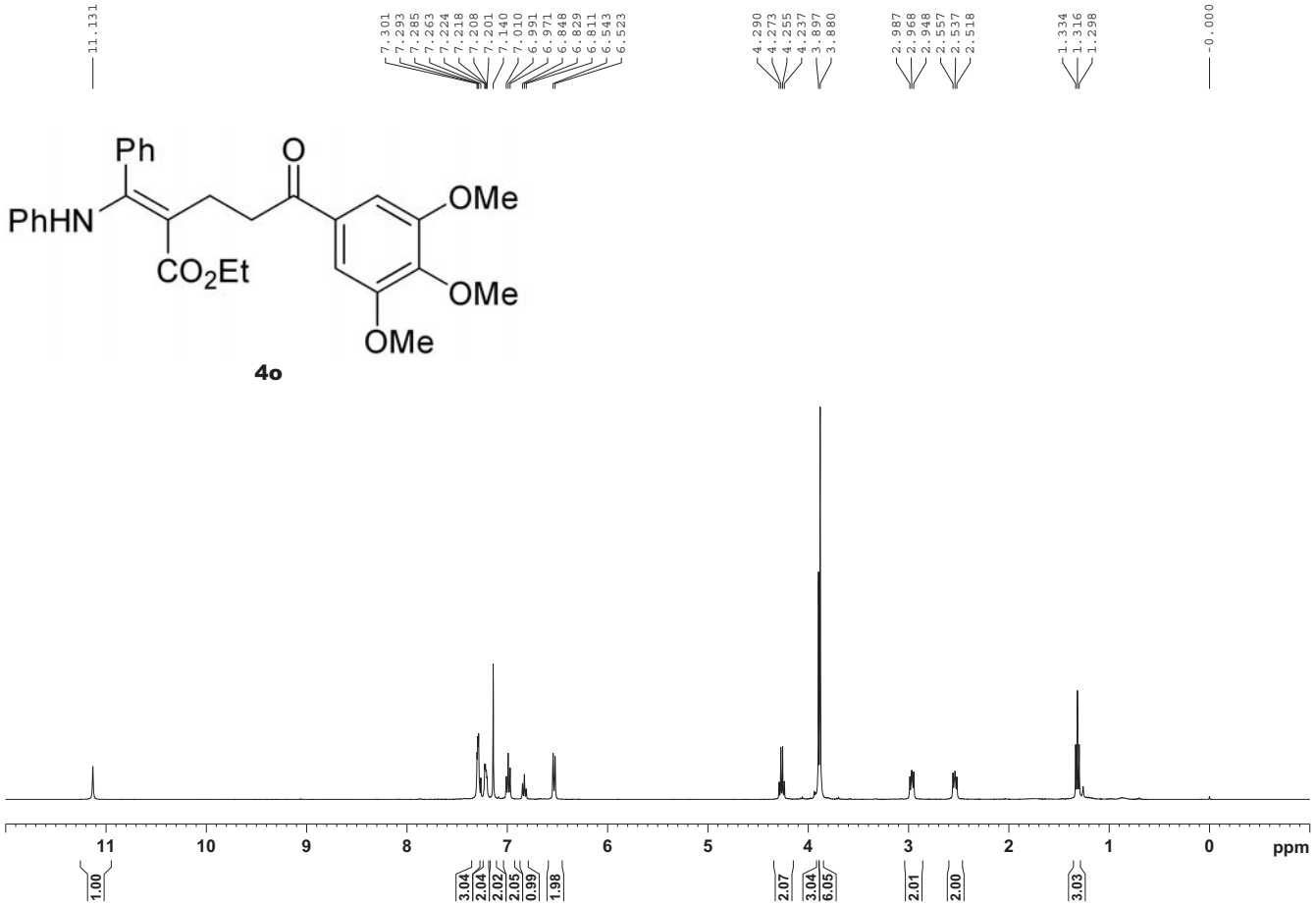
77.32
77.00
76.68
67.41
59.16
46.70
37.73
35.87
24.46
23.74
22.37
20.97
15.32
14.09
Ph
O
O
PhHN
CO2Et
4n
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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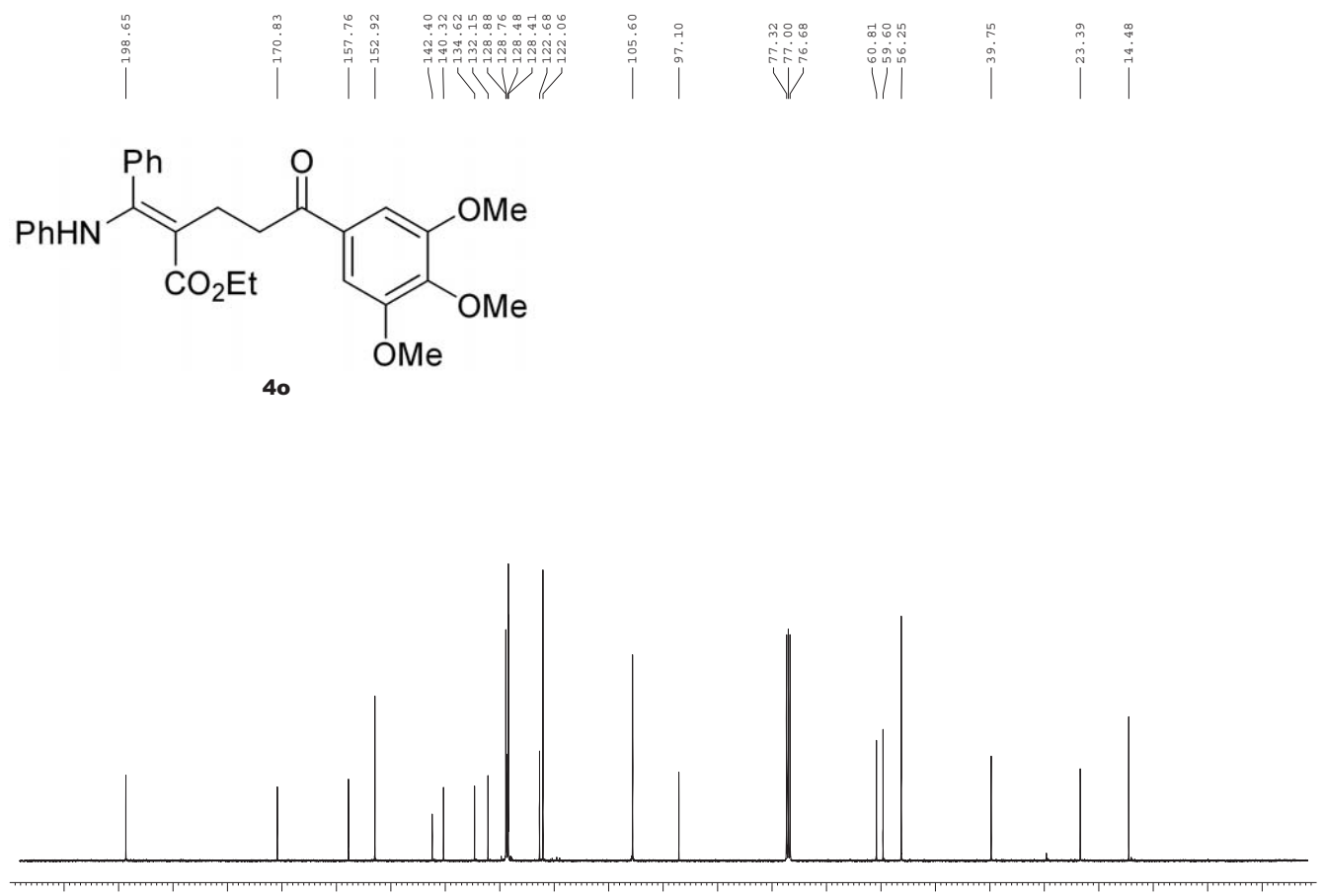
7.293
7.285
7.263
7.224
7.218
7.208
7.201
7.140
7.010
6.991
6.971
6.848
6.829
6.811
6.543
6.523
4.290
4.273
4.255
4.237
3.897
3.880
2.987
2.968
2.948
2.557
2.537
2.518
1.334
1.316
1.298
-0.000
Ph
O
PhHN
OMe
CO2Et
OMe
OMe
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
3.04
2.04
2.02
2.05
0.99
1.98
2.07
3.04
6.05
2.01
2.00
3.03

4o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157.76
152.92
142.40
140.32
134.62
132.15
128.88
128.76
128.48
128.41
122.68
122.06
105.60
97.10
77.32
77.00
76.68
60.81
59.60
56.25
39.75
23.39
14.48
Ph
O
PhHN
OMe
CO2Et
OMe
OMe
210
200
190
180
170
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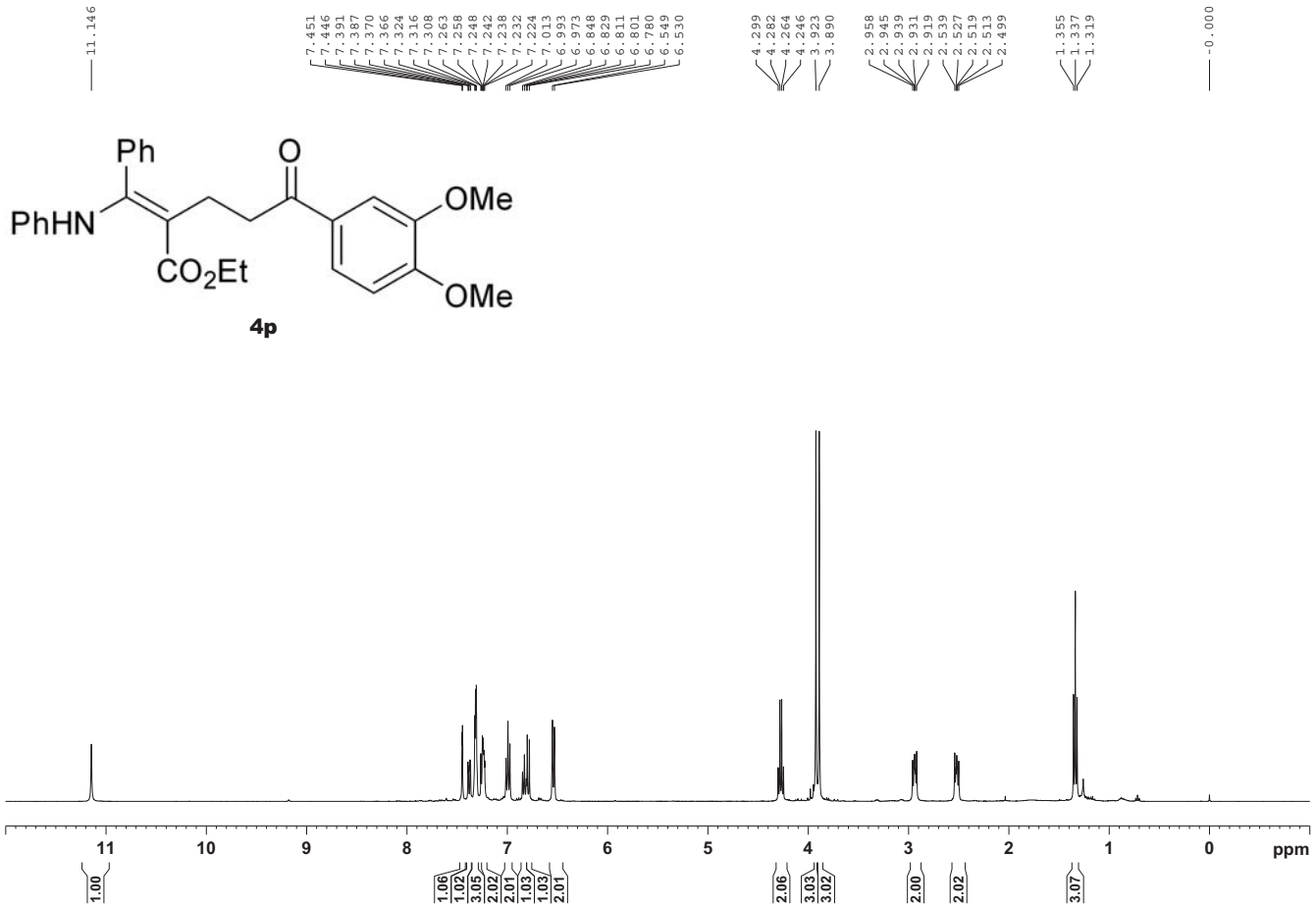
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

4o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7.446
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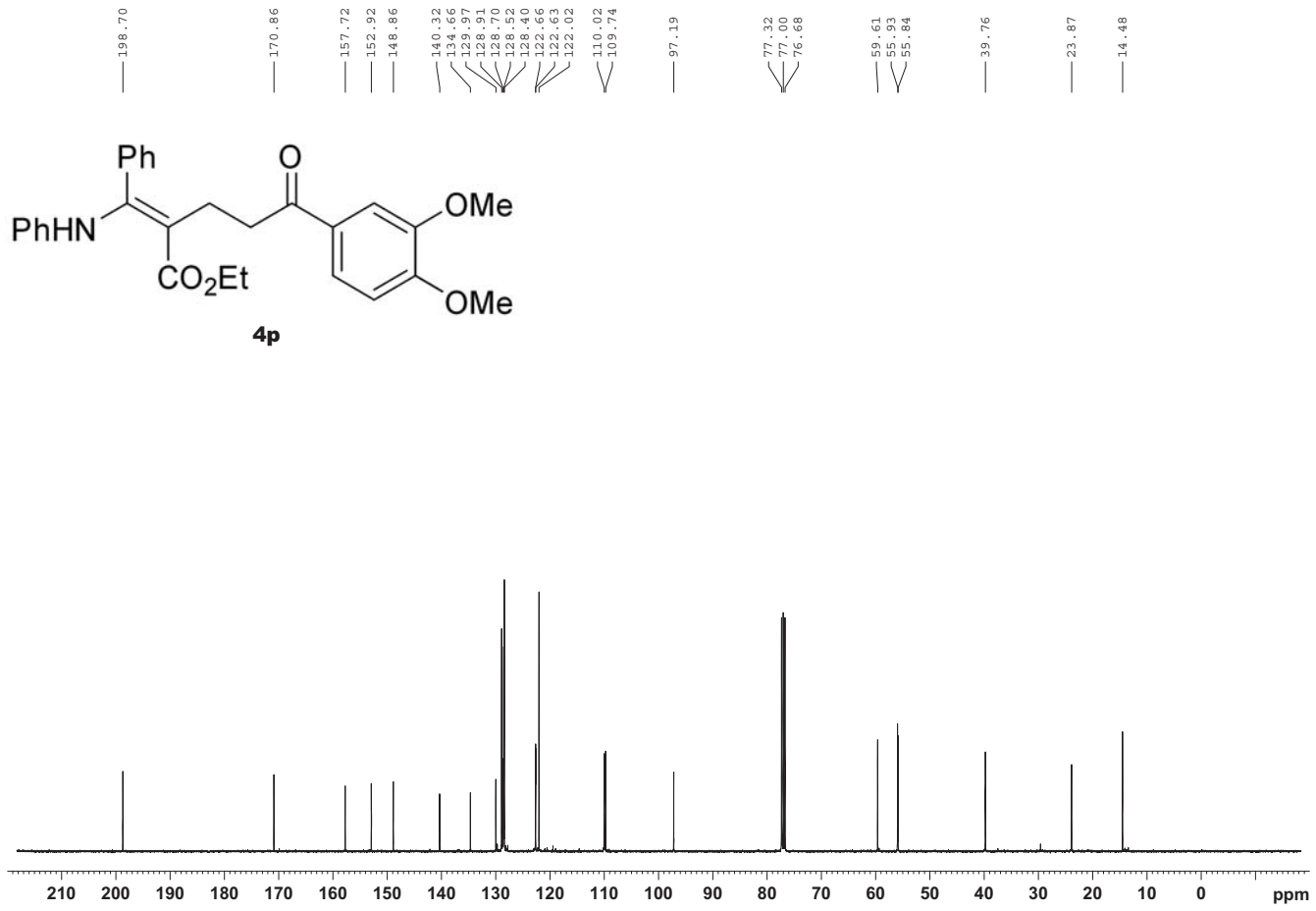
7.366
7.324
7.316
7.308
7.263
7.258
7.248
7.242
7.238
7.232
7.224
7.013
6.993
6.973
6.848
6.829
6.811
6.801
6.780
6.549
6.530
4.299
4.282
4.264
4.246
3.923
3.890
2.958
2.945
2.939
2.931
2.919
2.539
2.527
2.519
2.513
2.499
1.355
1.337
1.319
-0.000
Ph
O
PhHN
OMe
CO2Et
OMe
4p
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.06
1.02
3.05
2.02
2.01
1.03
1.03
2.01
2.06
3.03
3.02
2.00
2.02
3.07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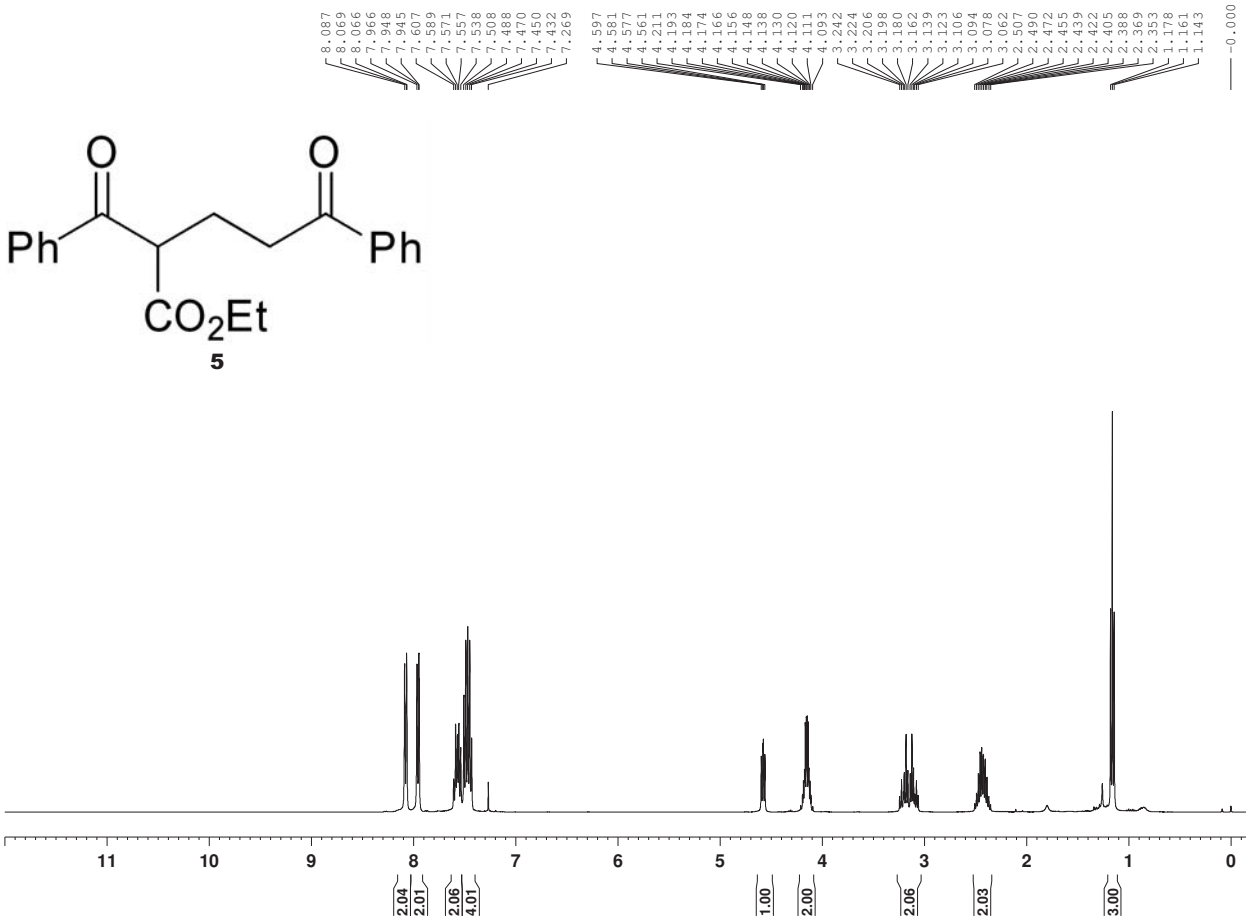
157.72
152.92
148.86
140.32
134.66
129.97
128.91
128.70
128.52
128.40
122.66
122.63
122.02
110.02
109.74
97.19
77.32
77.00
76.68
59.61
55.93
55.84
39.76
23.87
14.48
Ph
O
PhHN
OMe
CO2Et
OMe
4p
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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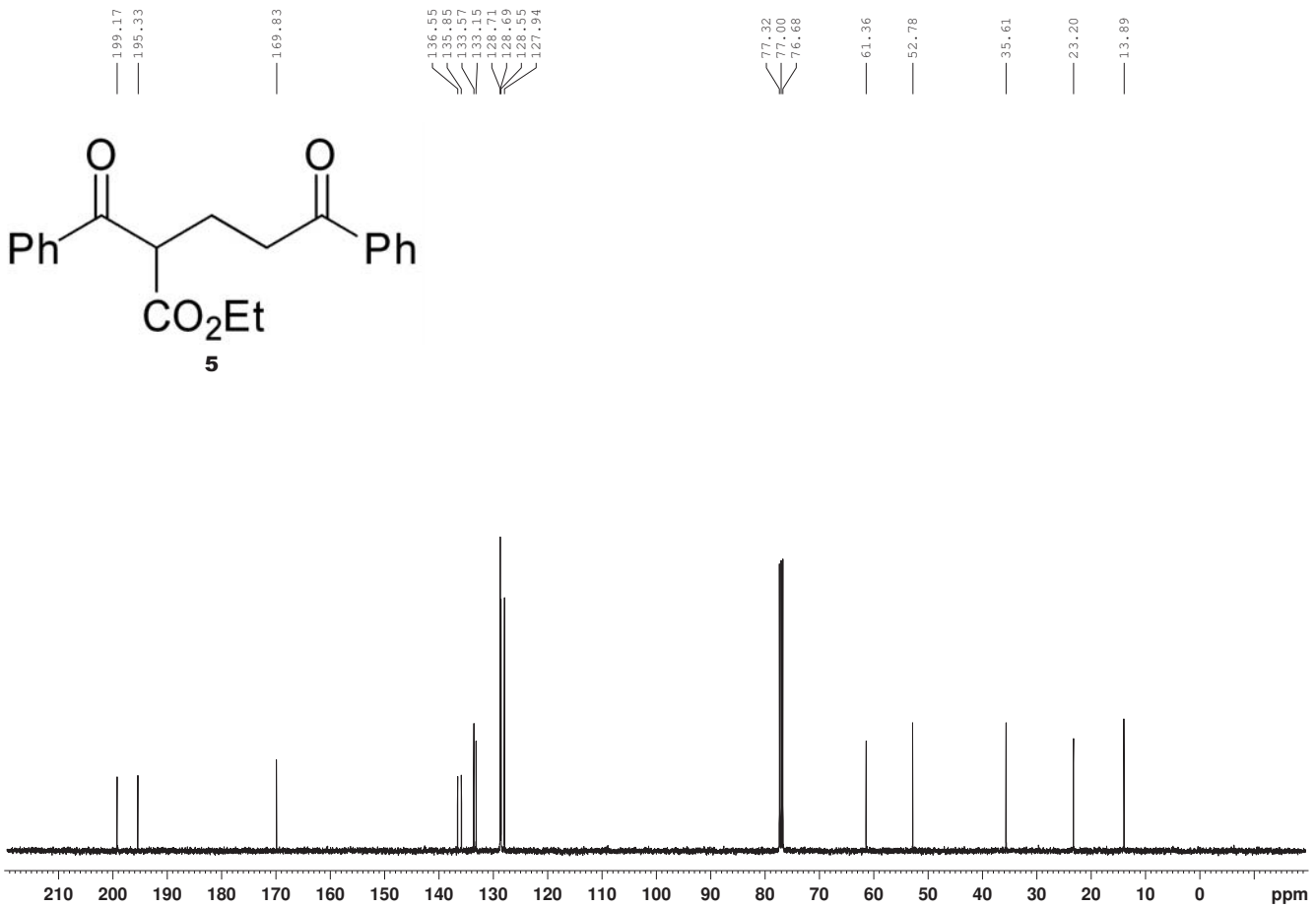
8.069
8.066
7.966
7.948
7.945
7.607
7.589
7.571
7.557
7.538
7.508
7.488
7.470
7.450
7.432
7.269
4.597
4.581
4.577
4.561
4.211
4.193
4.184
4.174
4.166
4.156
4.148
4.138
4.130
4.120
4.111
4.093
3.242
3.224
3.206
3.198
3.180
3.162
3.139
3.123
3.106
3.094
3.078
3.062
2.507
2.490
2.472
2.455
2.439
2.422
2.405
2.388
2.369
2.353
1.178
1.161
1.143
-0.000
O
O
Ph
Ph
CO2Et
5
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.04
2.01
2.06
4.01
1.00
2.00
2.06
2.03
3.00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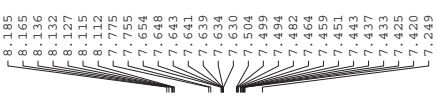
195.33
169.83
136.55
135.85
133.57
133.15
128.71
128.69
128.55
127.94
77.32
77.00
76.68
61.36
52.78
35.61
23.20
13.89
O
O
Ph
Ph
CO2Et
5
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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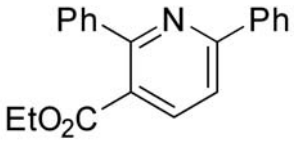
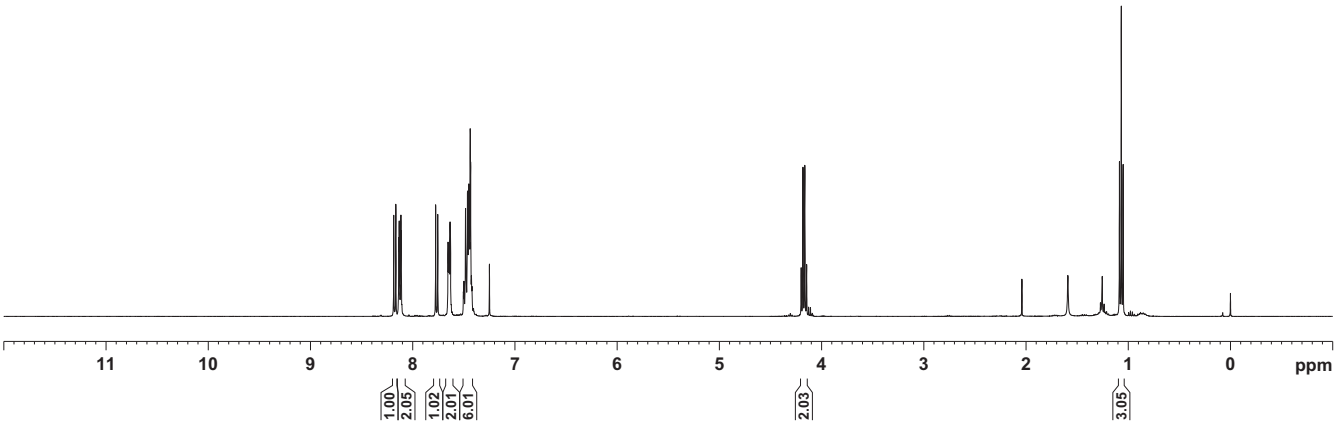
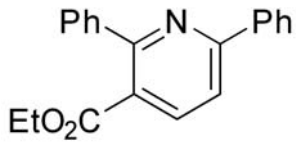
8.165
8.136
8.132
8.127
8.115
8.112
7.775
7.755
7.654
7.648
7.643
7.641
7.639
7.634
7.630
7.504
7.499
7.494
7.482
7.464
7.459
7.451
7.443
7.437
7.433
7.425
7.420
7.249
4.199
4.182
4.164
4.146
1.086
1.068
1.050
-0.000


Ph
N
Ph
EtO2C

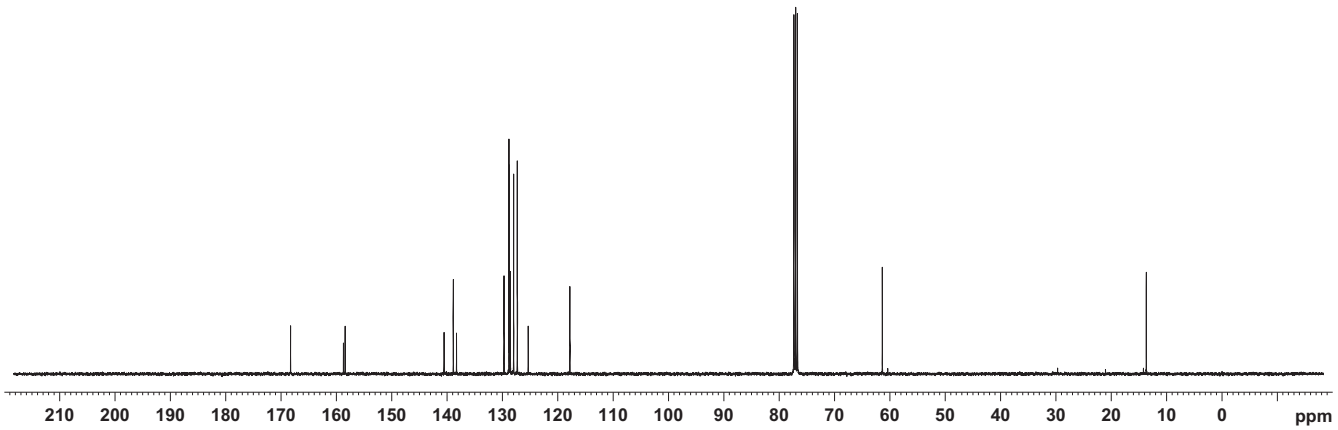
6

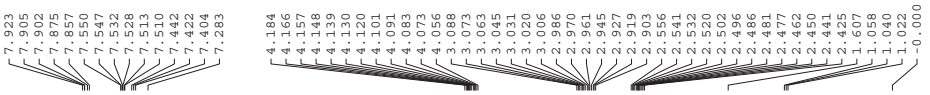

11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.05
1.02
2.01
6.01
2.03
3.05


168.26
158.71
158.40
140.53
138.85
138.28
129.70
128.81
128.75
128.55
127.96
127.30
125.32
117.80
77.32
77.00
76.68
61.34
13.65


Ph
N
Ph
EtO2C

6


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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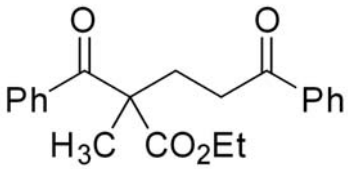
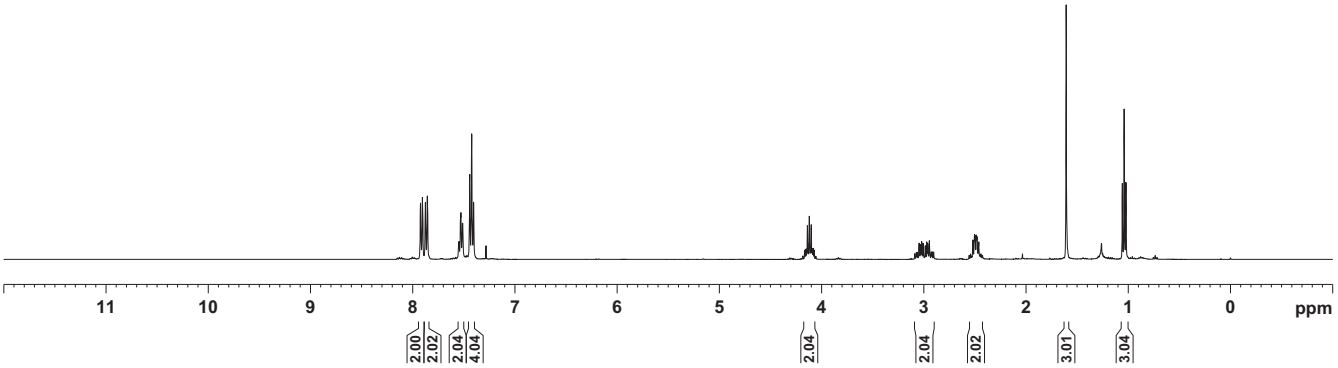
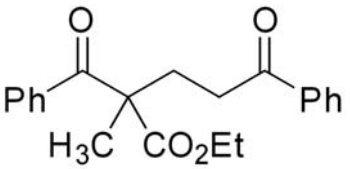
7.905
7.902
7.875
7.857
7.550
7.547
7.532
7.528
7.513
7.510
7.442
7.422
7.404
7.283
4.184
4.166
4.157
4.148
4.139
4.130
4.120
4.101
4.091
4.083
4.073
4.056
3.088
3.073
3.063
3.045
3.031
3.020
3.006
2.986
2.970
2.961
2.945
2.927
2.919
2.903
2.556
2.541
2.532
2.520
2.502
2.496
2.486
2.481
2.477
2.462
2.450
2.441
2.425
1.607
1.058
1.040
1.022
-0.000


O
O
Ph
Ph
H3C
CO2Et

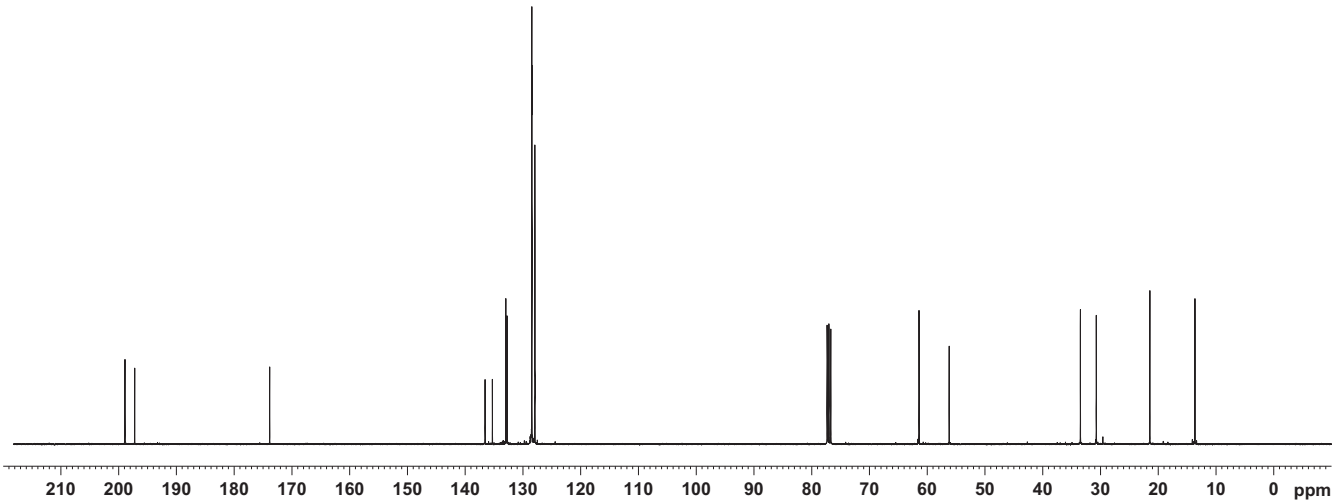
7

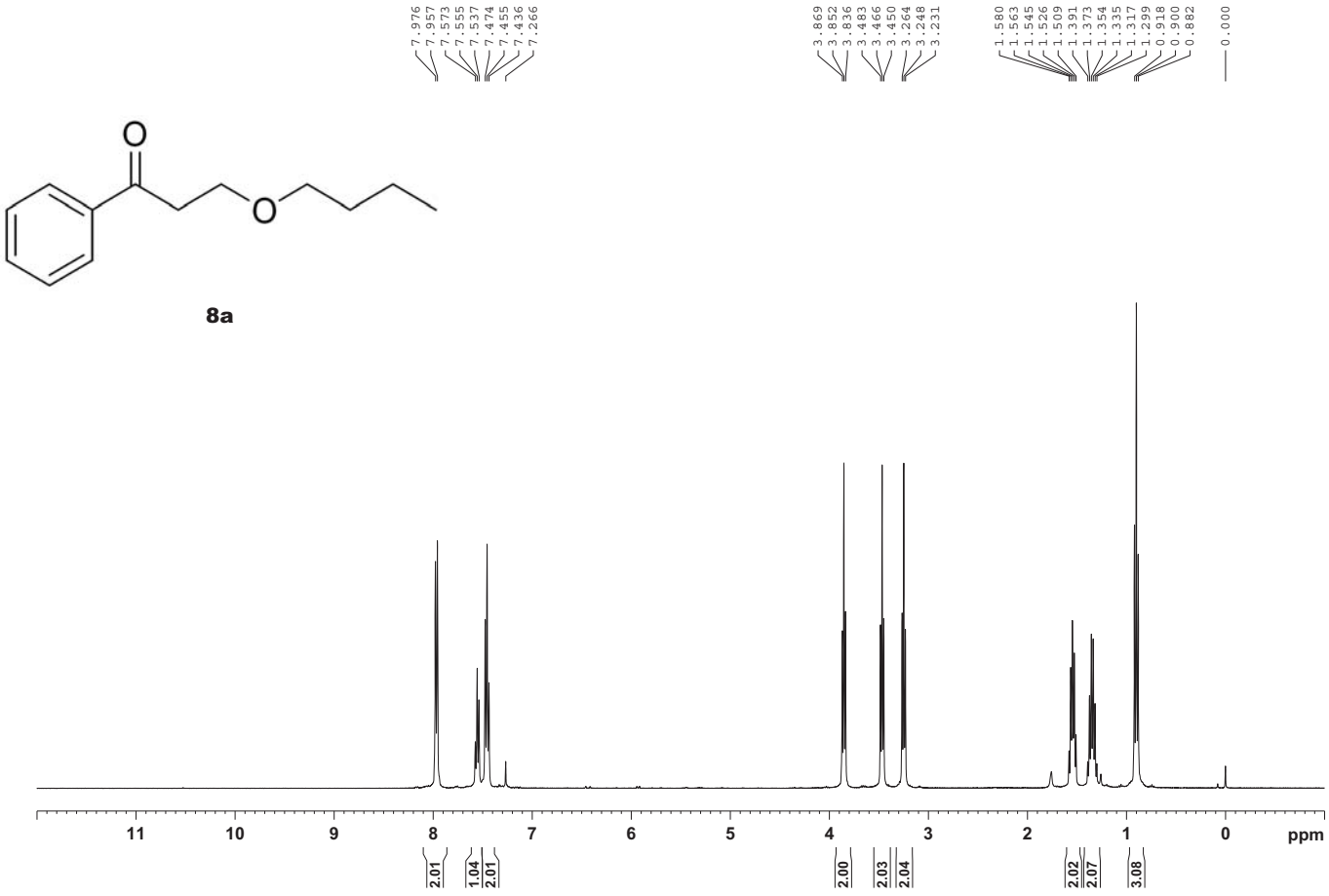

11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
2.02
2.04
4.04
2.04
2.04
2.02
3.01
3.04


198.89
197.21
173.83
136.54
135.27
132.95
132.72
128.45
128.44
128.43
127.91
77.32
77.00
76.68
61.40
56.18
33.46
30.71
21.43
13.62


O
O
Ph
Ph
H3C
CO2Et

7


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.573
7.555
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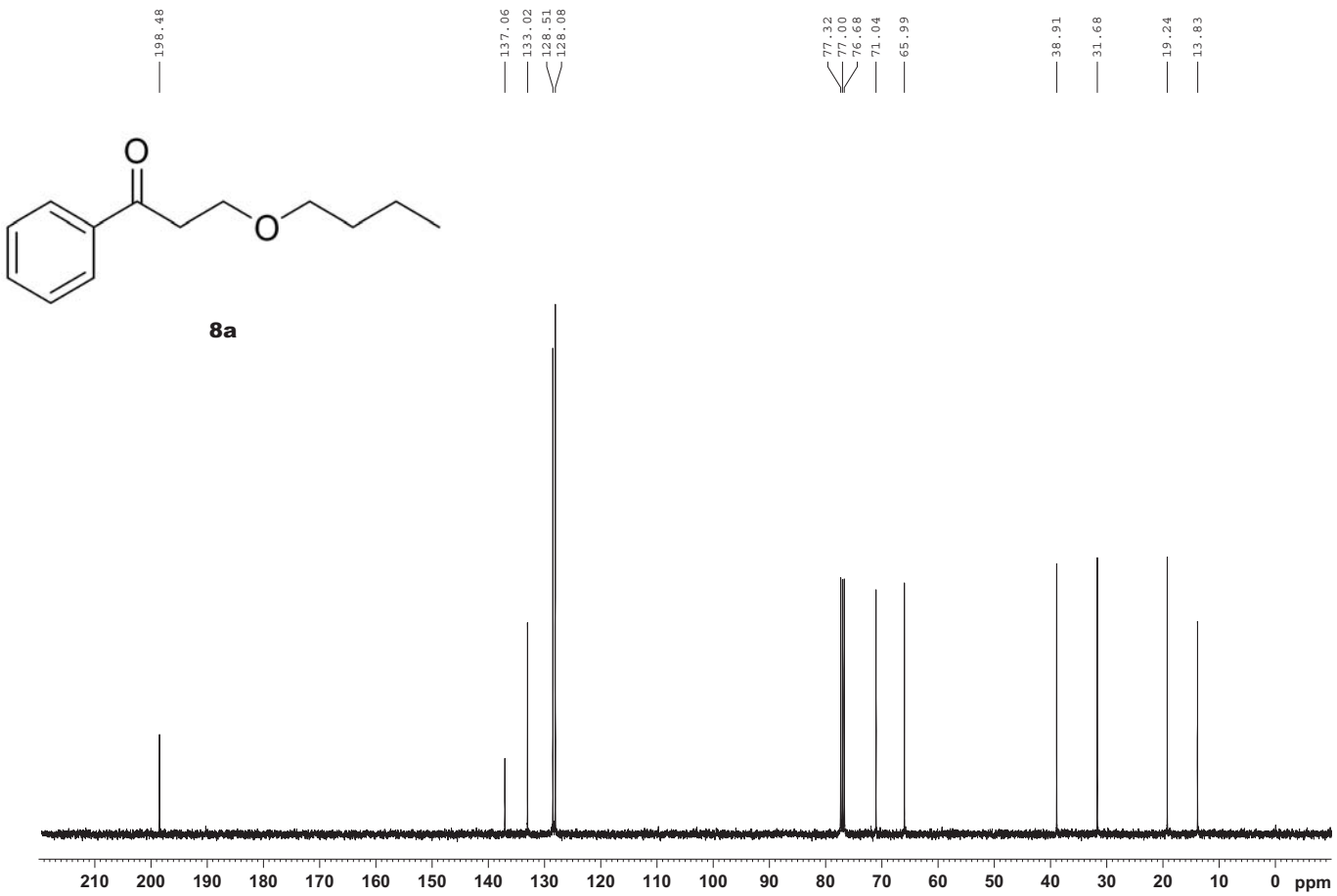
7.474
7.455
7.436
7.266
3.869
3.852
3.836
3.483
3.466
3.450
3.264
3.248
3.231
1.580
1.563
1.545
1.526
1.509
1.391
1.373
1.354
1.335
1.317
1.299
0.918
0.900
0.882
0.000
O
O
8a
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.01
1.04
2.01
2.00
2.03
2.04
2.02
2.07
3.08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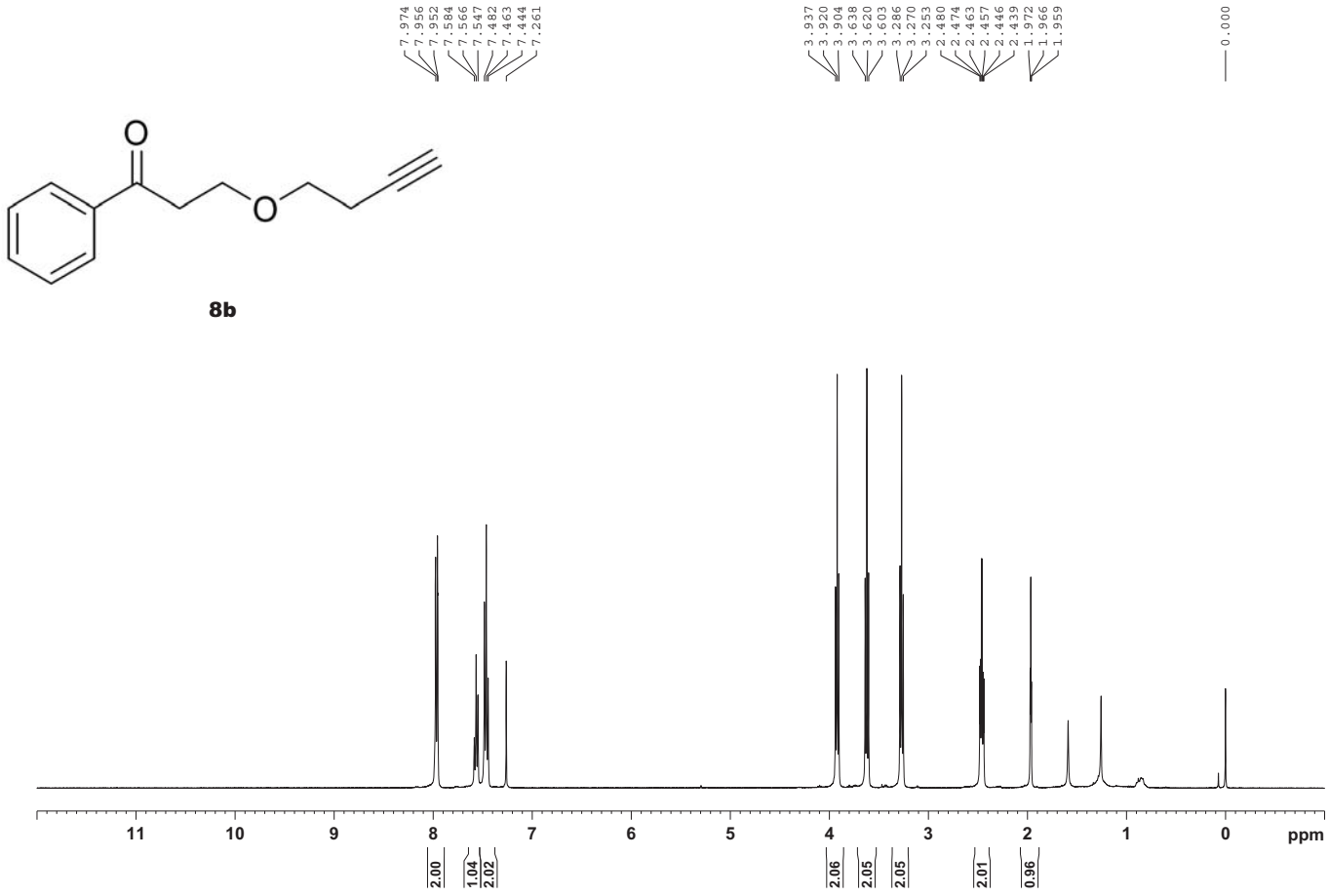
137.06
133.02
128.51
128.08
77.32
77.00
76.68
71.04
65.99
38.91
31.68
19.24
13.83
O
O
8a
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.952
7.584
7.566
7.547
7.482
7.463
7.444
7.261
3.937
3.920
3.904
3.638
3.620
3.603
3.286
3.270
3.253
2.480
2.474
2.463
2.457
2.446
2.439
1.972
1.966
1.959
0.000
8b
11
10
9
8
7
6
5
4
3
2
1
0
ppm
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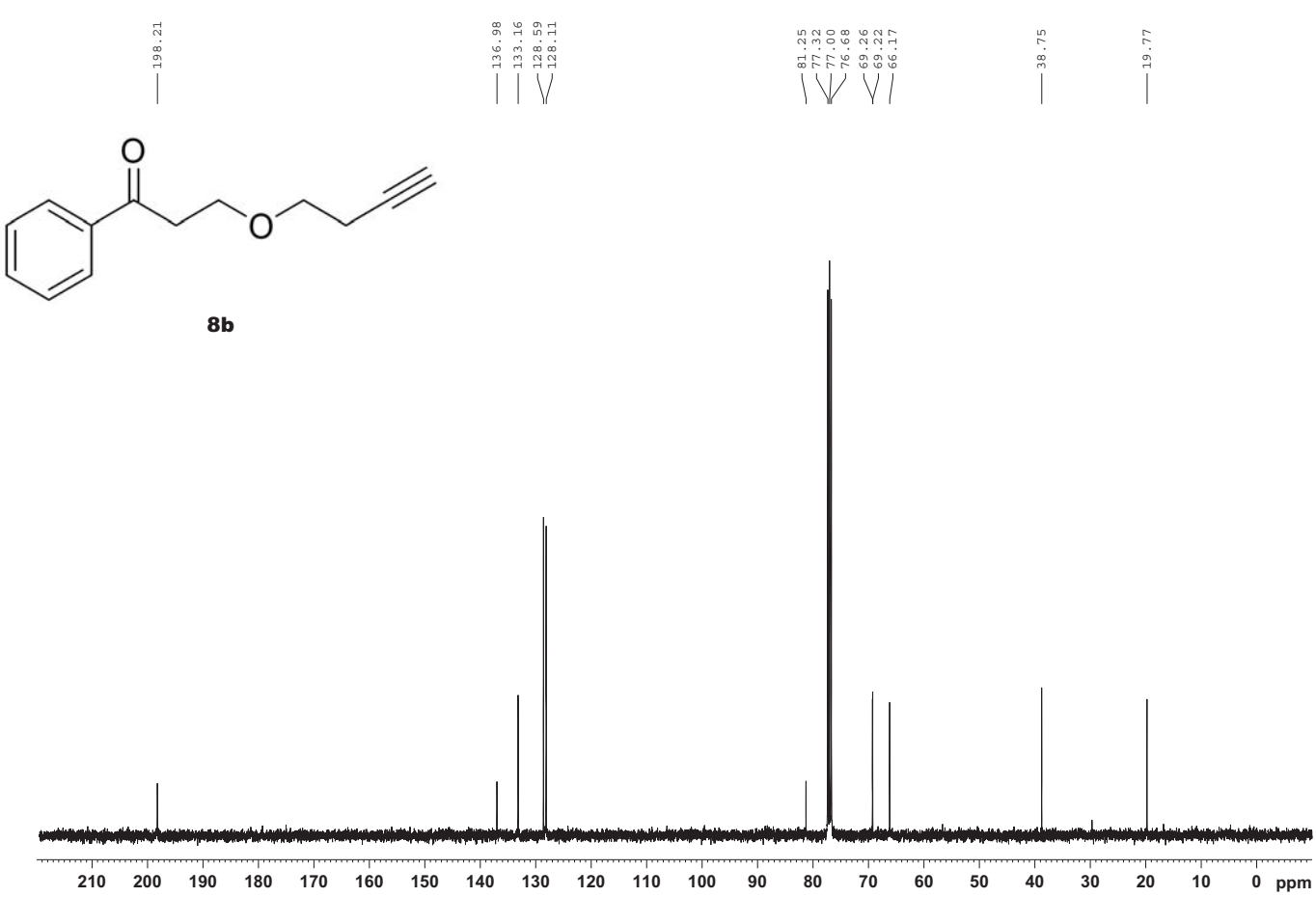
1.04
2.02
2.06
2.05
2.05
2.01
0.96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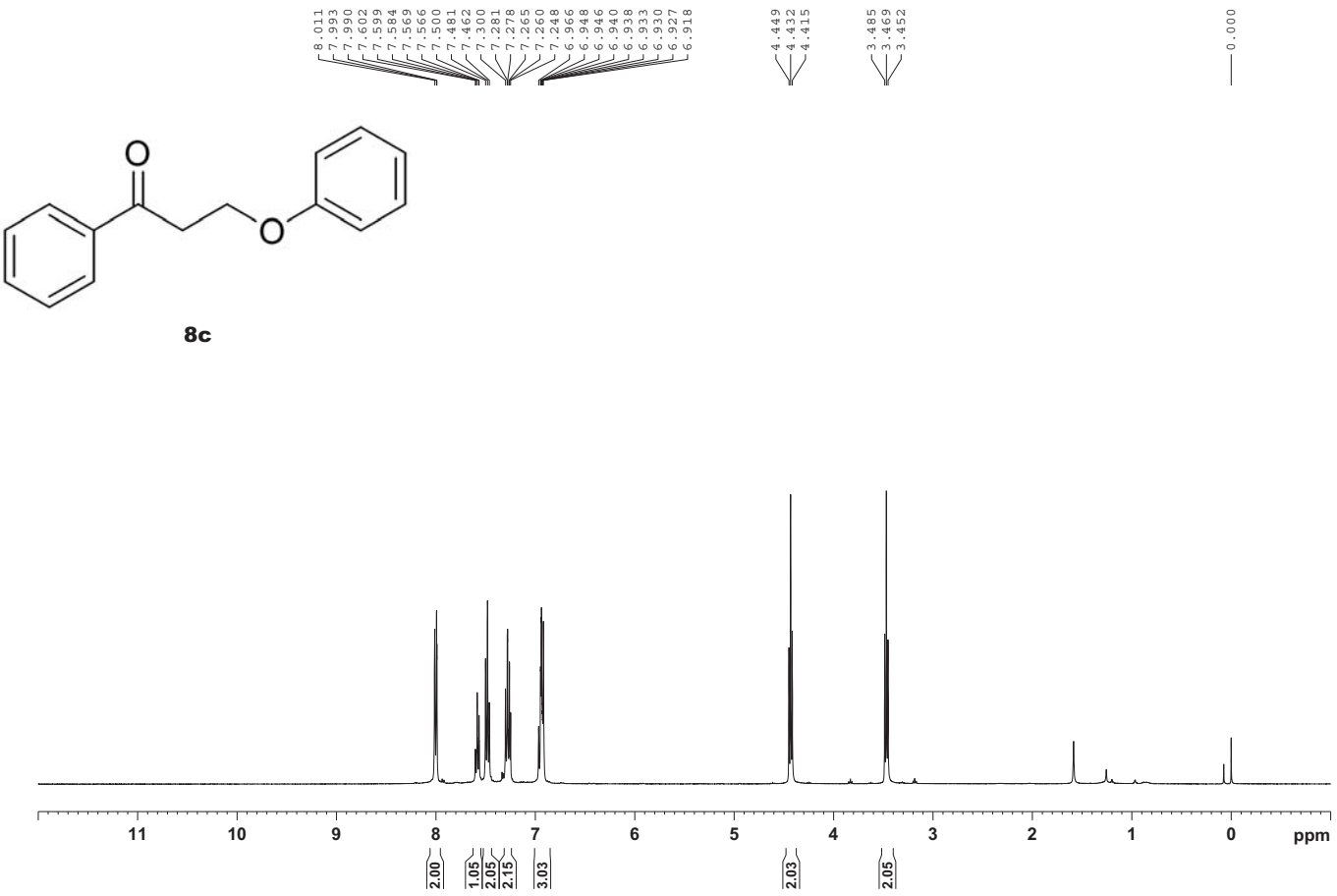
128.11
81.25
77.32
77.00
76.68
69.26
69.22
66.17
38.75
19.77
8b
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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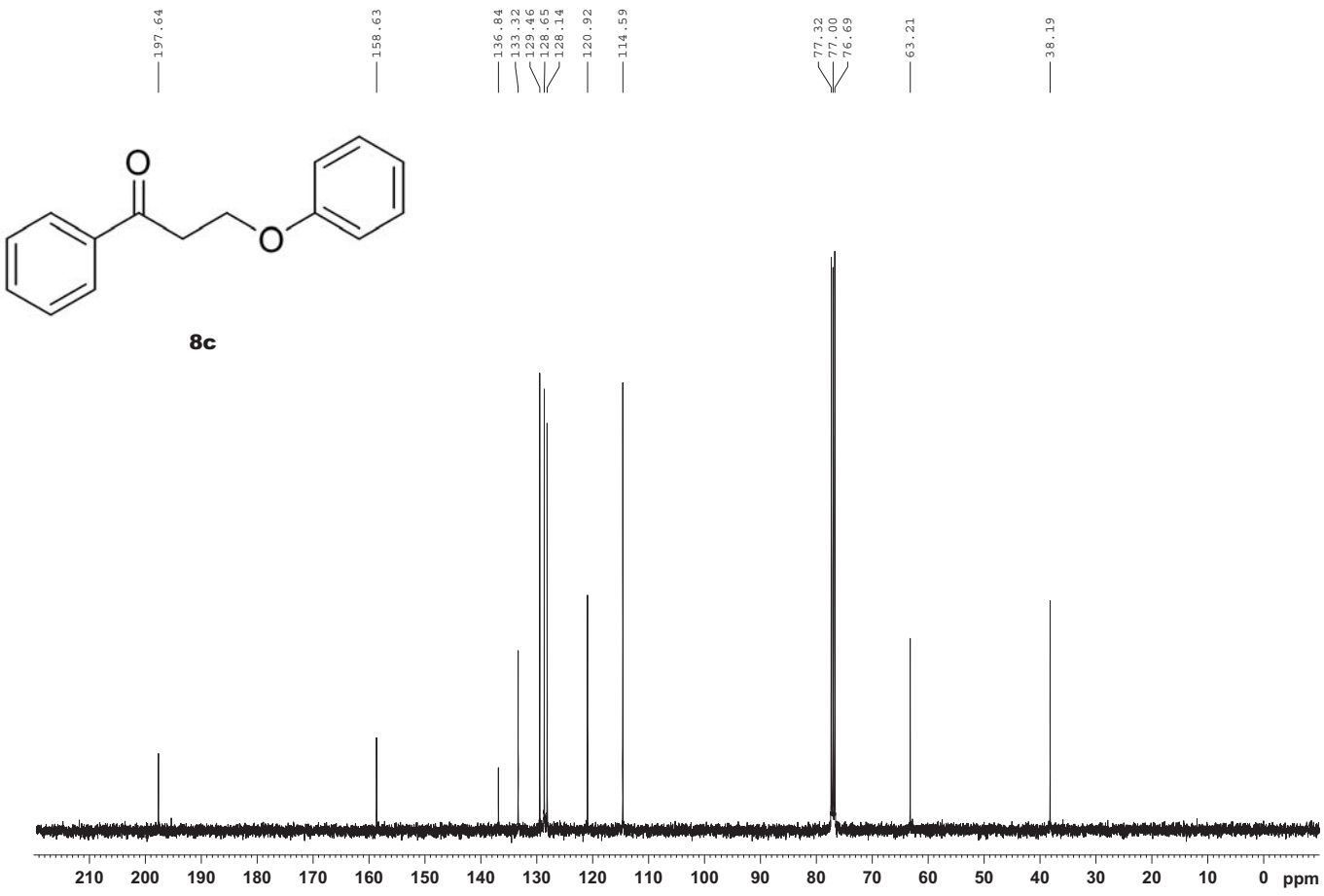
7.599
7.584
7.569
7.566
7.500
7.481
7.462
7.300
7.281
7.278
7.265
7.260
7.248
6.966
6.948
6.946
6.940
6.938
6.933
6.930
6.927
6.918
4.449
4.432
4.415
3.485
3.469
3.452
0.000
O
O
8c
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
1.05
2.05
2.15
3.03
2.03
2.05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133.32
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128.65
128.14
120.92
114.59
77.32
77.00
76.69
63.21
38.19
O
O
8c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm