Supporting Information

4-HO-TEMPO-Catalyzed Redox Annulation of Cyclopropanols with Oxime Acetates toward Pyridine Derivatives

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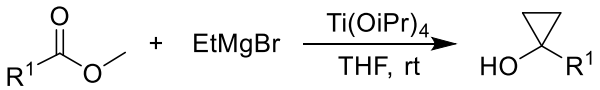
General:

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 or 300 MHz）and ^{13}C NMR (100 MHz or 75 MHz) spectra were recorded in $CDCl_3$ or DMSO-d_6. 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), quintet (quin), sextet (sex) septet (sep) 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. Enantiomeric excess is determined by HPLC analysis (Agilent 1260).

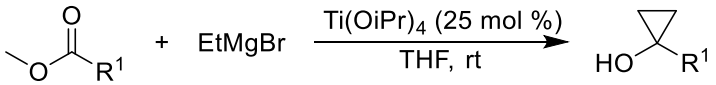
Substrates preparation:

General procedure for the synthesis of arylcyclopropanols 1a-1i.



In a fume hood, 22 mmol of ethylmagnesium bromide in THF (2M) was added dropwise over 30 minutes at room temperature to a solution of 10 mmol of ester and 10 mmol of titanium tetraisopropoxide in 30 mL THF. The reacton mixture was stirred overnight. Then the solution was quenched with 30 mL H_2SO_4 (1 M) and stirred while still in the fume hood. The solution was extracted with 3 x 30 mL of diethyl ether, washed with distilled water, and dried over Na_2SO_4 followed by filtration and concentration. The mixture was subjected to column chromatography to afford the arycyclopropanols. [1]

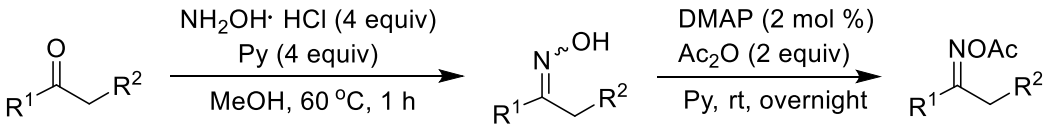
General procedure for the synthesis of alkylcyclopropanols 1j-1v.



In a fume hood, 22 mmol of ethylmagnesium bromide in THF was added dropwise over 30 minutes at room temperature to a solution of 10 mmol of ester and 2.5 mmol of

titanium tetraisopropoxide in 30 mL THF. The reacton mixture was stirred overnight. Then the solution was quenched with 10 mL H_2SO_4 (1 M) and stirred while still in the fume hood. The solution was extracted with 3 x 30 mL of diethyl ether, washed with distilled water, and dried over Na_2SO_4 followed by filtration and concentration. The mixture was subjected to column chromatography to afford the alkylcyclopropanols. [1]

General procedure for the synthesis of oxime esters 2.

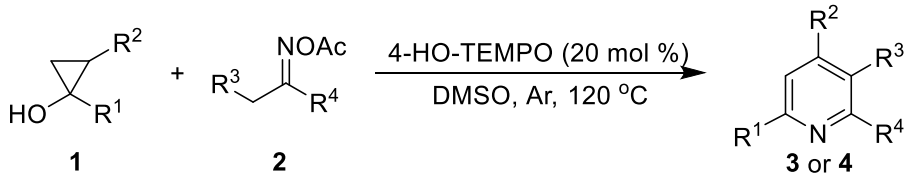


1) Ketone (10 mmol), hydroxylamine hydrochloride (4 equiv), pyridine (4 equiv) in CH_3OH (20 mL) were stirred at 60 °C for 1 h. After completion of the reaction, the solvent was removed under reduced pressure. The mixture was diluted with H_2O (30 mL) and extracted with EtOAc (3 × 20 mL). The organic layer was washed with 1 M HCl, dried over anhydrous Na_2SO_4, and filtered. The solvent was removed under reduced pressure. All the obtained products were used for the next step without further purification. [2]

2) The ketoxime, DMAP (2 mol %) and Ac_2O (2 equiv) were dissolved in pyridine (5 mL). After completion of the reaction, the reaction mixture was extracted three times with EA. The combined organic layers were washed with 1 M HCl and dried over anhydrous Na_2SO_4 and filtered. The solvent was evaporated under reduced pressure and the residue was purified by a flash column chromatography on a silica gel eluting with petroleum ether and ethyl acetate to afford the corresponding oxime esters. [2]

General experimental procedure:

Typical experimental procedure for the synthesis of pyridines 3 and 4.

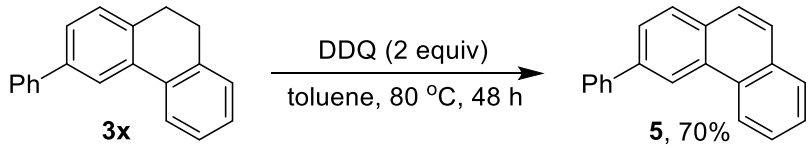


A 25 mL oven-dried reaction tube were charged with cyclopropanols **1** (0.5 mmol), oxime esters **2** (1.25 mmol, 2.5 equiv), 4-HO-TEMPO (0.1 mmol, 20 mol %) and 2.5 mL DMSO. The tube was then sealed and the displacement of argon gas for three times.

Then the mixture was stirred for 24-36 h at 120 °C. Upon completion of the reaction, the solvent was then removed under vacuo. The residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/100 to 1/3) to give the corresponding products in yields listed in Scheme 3-7. The identity and purity of the product was confirmed by ^{1}H and ^{13}C spectroscopic analysis.

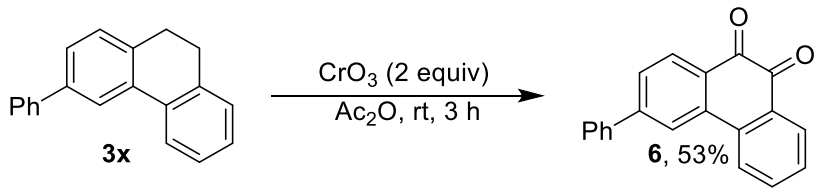
Follow-up transformations of 3x:

General procedure for the synthesis of 2-phenylbenzo[*h*]quinoline 5.



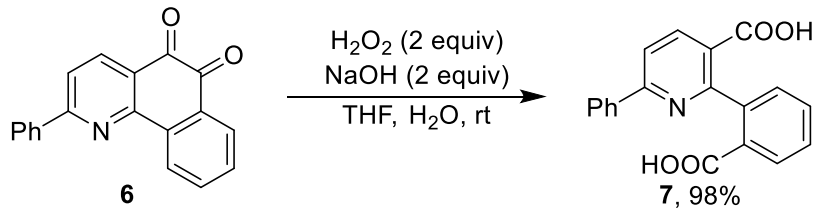
A 25 mL oven-dried reaction tube were charged with **3x** (0.3 mmol), DDQ (0.6 mmol, 2 equiv), 2 mL toluene. Then the mixture was stirred for 48 h at 80 °C. The reaction mixture was isolated by silica gel column chromatography to give the product **5** in 70 % yield. [3]

General procedure for the synthesis of 2-phenylbenzo[*h*]quinoline-5,6-dione 6.



A 25 mL oven-dried reaction tube were charged with **3x** (0.3 mmol), CrO_3 (0.6 mmol, 2 equiv), 2.5 mL Ac_2O. Then the mixture was stirred for 3 h at rt. The reaction mixture was isolated by silica gel column chromatography to give the product **6** in 53 % yield. [4]

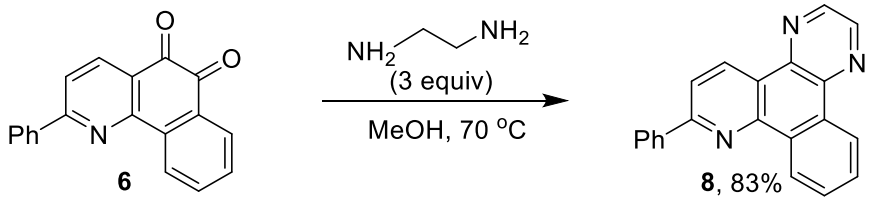
General procedure for the synthesis of 2-(2-carboxyphenyl)-6-phenylnicotinic acid 7.



1) Diketone **6** (0.3 mmol), 30% H_2O_2 (0.6 mol, 2 equiv), KOH (2 equiv) in H_2O (2 mL) and THF (4 mL) were stirred at room temperature. After completion of the reaction, the mixture was regulated PH to 2-3 by using concentrated hydrochloric acid. Then extracted

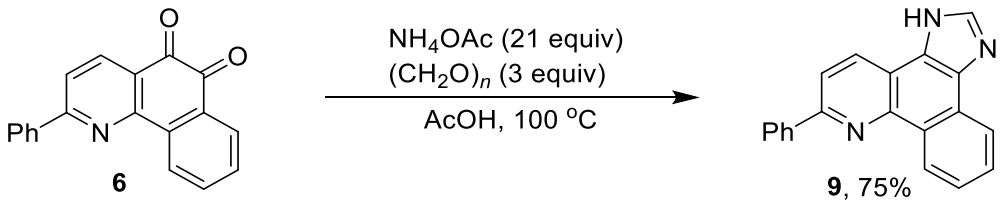
with EtOAc (3 × 20 mL) and dried over anhydrous Na_2SO_4 and filtered. The solvent was evaporated under reduced pressure and the residue was purified by a flash column chromatography on a silica gel eluting with DCM and MeOH to afford the corresponding product **7** in 98% yield. [5]

General procedure for the synthesis of 7-phenylbenzo[*f*]pyrido[2,3-*h*]quinoxaline 8.



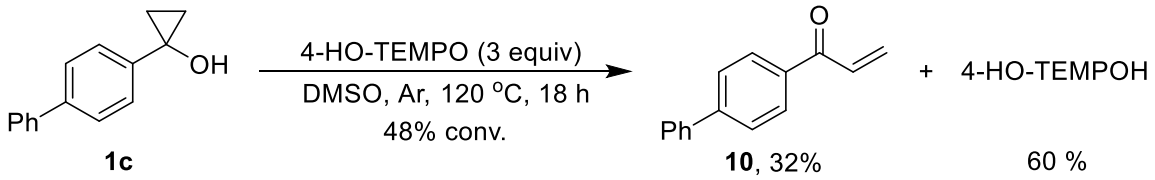
A mixture of diketone **6** (0.3 mmol) and ethylenediamine (0.9 mmol, 3 equiv) in 2 mL MeOH was refluxed for 4 h. After cooling, the reaction mixture was isolated by silica gel column chromatography to give the product **8** in 83 % yield. [6]

General procedure for the synthesis of 6-phenyl-3H-benzo[h]imidazo[4,5-f]quinoline 9.



A mixture of diketone **6** (0.3 mmol), formaldehyde (0.9 mol, 3 equiv) and NH_4OAc (6.3 mmol) in AcOH 1.5 mL was stirred at 100 °C for 8 h. After cooling, the reaction mixture was diluted with water and neutralized with concentrated aqueous ammonia (28-30% wt) to pH 7. Then, extracted with EtOAc (3 × 20 mL) and dried over anhydrous Na_2SO_4 and filtered. The solvent was evaporated under reduced pressure and the residue was purified by a flash column chromatography on a silica gel eluting to afford the corresponding product **9** in 75% yield. [7]

Control experiments:

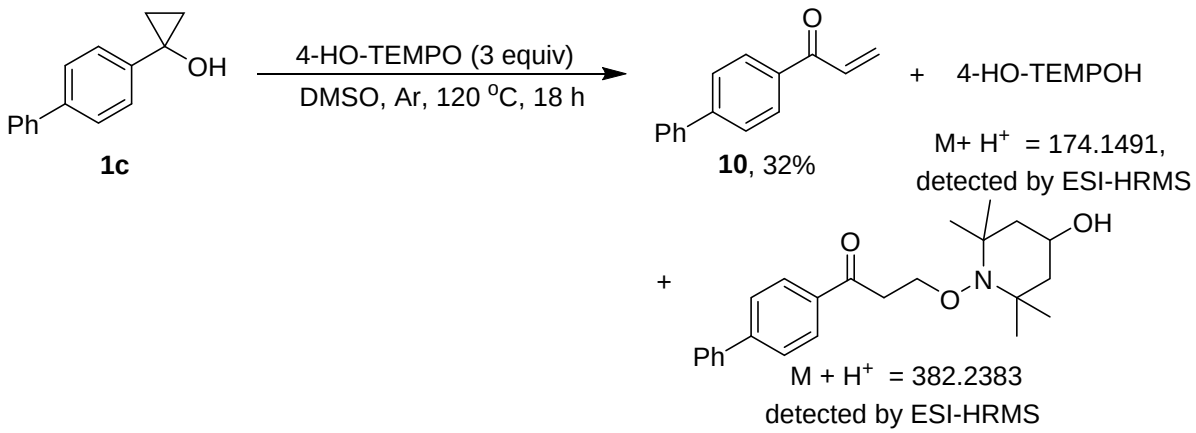


A 25 mL oven-dried reaction tube were charged with 1-([1,1'-biphenyl]-4-yl)

cyclopropanol **1c** (0.3 mmol), 4-HO-TEMPO (0.9 mmol, 3 equiv) and 1.5 mL DMSO. The tube was then sealed and the displacement of argon gas for three times. Then the mixture was stirred for 18 h at 120 °C. The reaction mixture was isolated by silica gel column chromatography to give the products **10** in 32% yield and 4-HO-TEMPOH was isolated in 60% yield.

HRMS analysis for reaction intermediates.

In a reaction tube, 1-([1,1'-biphenyl]-4-yl)cyclopropan-1-ol **1c** (0.3 mmol), 4-HO-TEMPO (0.9 mmol, 3 equiv) and DMSO (1.5 mL) were added. The reaction mixture was stirred at 120 °C for 18 h in an oil bath with vigorous stirring. Then, the reaction was cooled to room temperature and diluted with DCM prior to the injection into the high-resolution mass spectrometer.



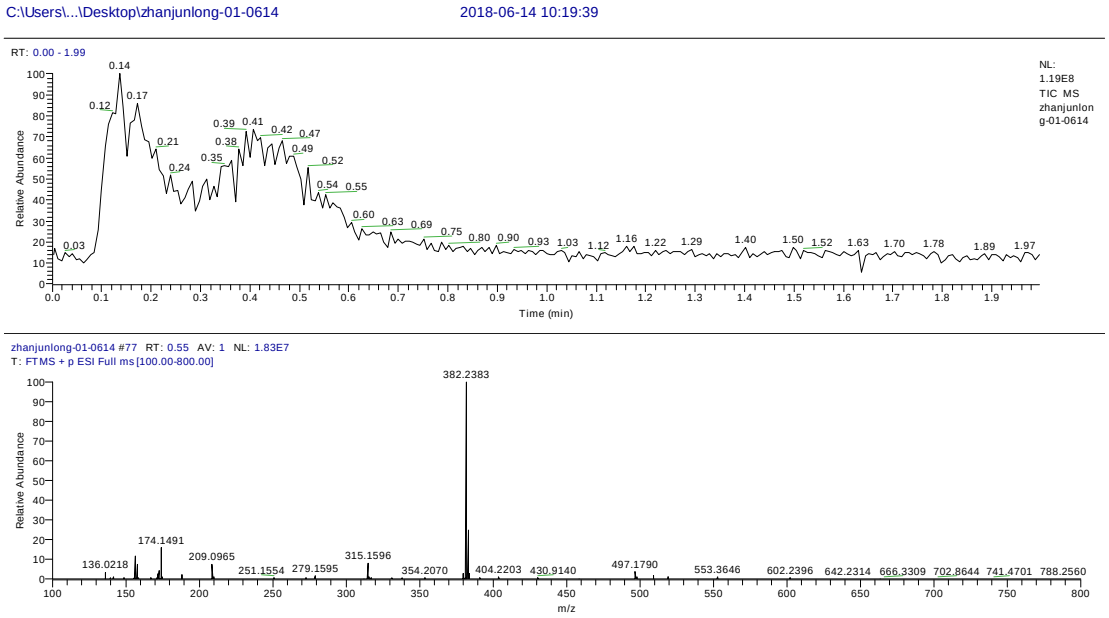
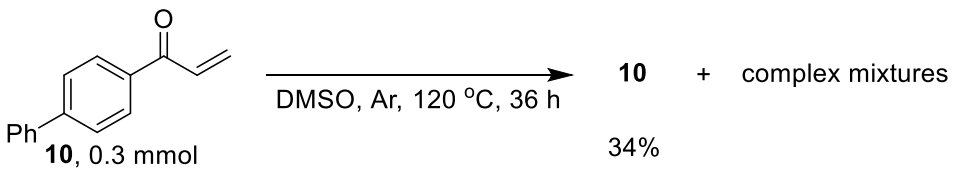
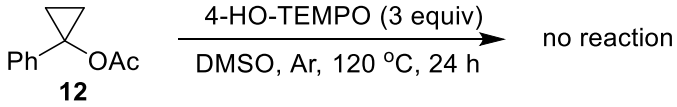


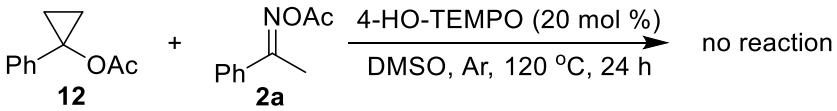
Figure S1. ESI-HRMS analysis for reaction study.



A 25 mL oven-dried reaction tube were charged with α,β-unsaturated ketone **10** (0.3 mmol), and 1.5 mL DMSO. The tube was then sealed and the displacement of argon gas for three times. Then the mixture was stirred under standard reaction conditions for 36 h. The α,β-unsaturated ketone **10** remained only in 34% and some complex mixtures were detected.



A 25 mL oven-dried reaction tube were charged with 1-phenylcyclopropyl acetate **12** (0.3 mmol), 4-HO-TEMPO (3 equiv) and 1.5 mL DMSO. The tube was then sealed and the displacement of argon gas for three times. Then the mixture was stirred under standard reaction conditions for 24 h. No reaction were detected.



A 25 mL oven-dried reaction tube were charged with 1-phenylcyclopropyl acetate **12** (0.3 mmol), **2a** (2.5 equiv), 4-HO-TEMPO (20 mol %) and 1.5 mL DMSO. The tube was then sealed and the displacement of argon gas for three times. Then the mixture was stirred under standard reaction conditions for 24 h. No reaction were detected.

Cyclic Voltammetry (CV) Experiments

Cyclic voltammograms were recorded in a single cell, constructed from a glass vial, fitted with three electrodes. A glassy carbon disk electrode was used as a working electrode and a platinum wire was used as a counter electrode. The potential was recorded with the reference electrode saturated calomel electrode (SCE) immersed in 0.1 M solution of *n*-Bu_4NBF_4 in 20 ml CH_3CN. The solutions were deoxygenated with a stream of nitrogen for 15 min before each experiment. A scan rate of 100 mV/s was used.

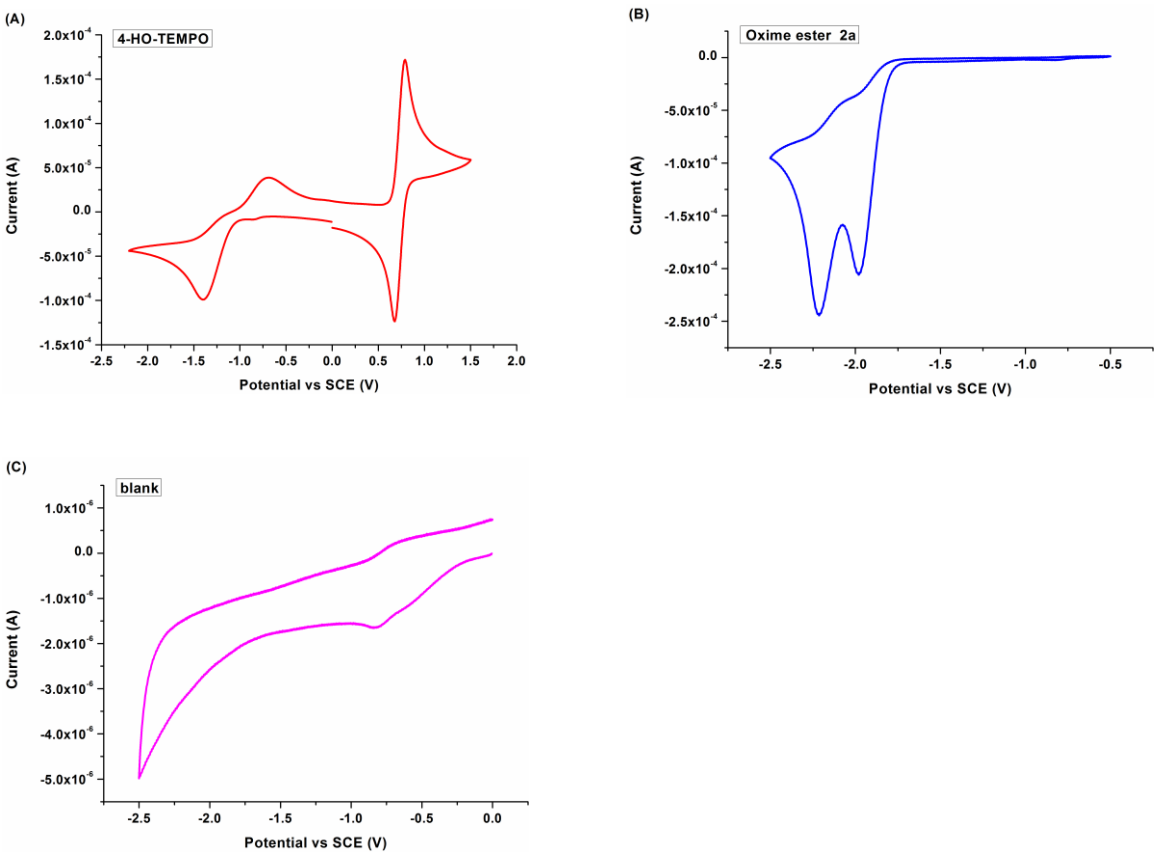
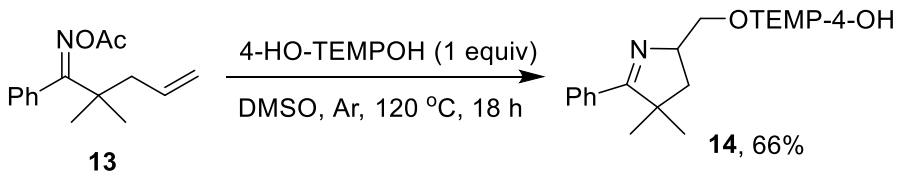
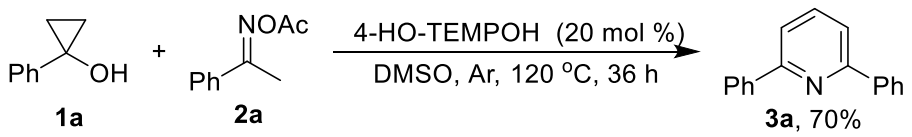


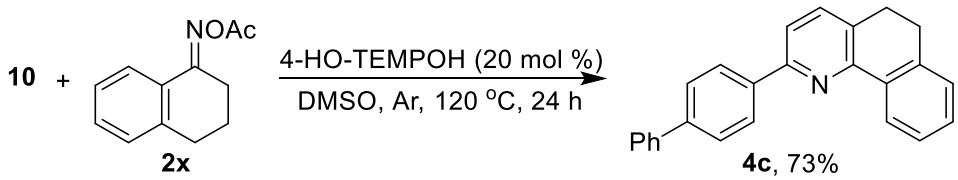
Figure S2. (A) 5 mM 4-HO-TEMPO, E_{red} = -1.2 V vs SCE; (B) 5 mM acetophenone oxime acetate **2a**, E_{red} = -1.9 V vs SCE. (C) The blank experiment.



A 25 mL oven-dried reaction tube were charged with oxime ester **13** (0.3 mmol), 4-HO-TEMPOH (0.3 mmol, 1 equiv) and 1.5 mL DMSO. The tube was then sealed and the displacement of argon gas for three times. Then the mixture was stirred for 18 h at 120 °C. The reaction mixture was isolated by silica gel column chromatography to give the product **14** in 66% yield. The identity and purity of the product was confirmed by ^{1}H and ^{13}C NMR spectroscopic analysis.

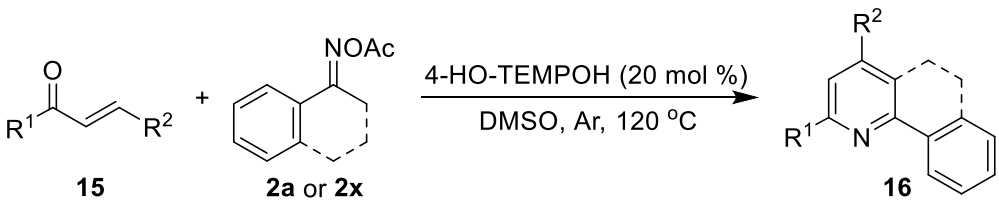


A 25 mL oven-dried reaction tube were charged with 1-phenylcyclopropanol **1a** (0.3 mmol), acetophenone oxime acetate **2a** (0.75 mmol, 2.5 equiv), 4-HO-TEMPOH (0.06 mmol, 20 mol %) and 1.5 mL DMSO. The tube was then sealed and the displacement of argon gas for three times. Then the mixture was stirred for 36 h at 120 °C. The reaction mixture was isolated by silica gel column chromatography to give the product **3a** in 70% yield.



A 25 mL oven-dried reaction tube were charged with α,β-unsaturated ketone **10** (0.3 mmol), oxime acetate **2x** (0.36 mmol, 1.2 equiv), 4-HO-TEMPOH (0.06 mmol, 20 mol %) and 1.5 mL DMSO. The tube was then sealed and the displacement of argon gas for three times. Then the mixture was stirred for 24 h at 120 °C. The reaction mixture was isolated by silica gel column chromatography to give the product **4c** in 73% yield.

General procedure for 4-HO-TEMPOH catalyzed [3+3] annulation



A 25 mL oven-dried reaction tube were charged with α,β-unsaturated aldehyde/ketone **15** (0.3 mmol), oxime esters **2a or 2x** (0.36 mmol, 1.2 equiv), 4-HO-TEMPOH (0.06 mmol, 20 mol %) and 1.5 mL DMSO. The tube was then sealed and the displacement of argon gas for three times. Then the mixture was stirred for 24 h at 120 °C. Upon completion of

the reaction, the solvent was then removed under vacuo. The residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/25 to 1/15) to give the corresponding products **16** in yields listed in Scheme 10. The identity and purity of the product was confirmed by ^{1}H and ^{13}C spectroscopic analysis.

Theoretical calculations

Calculation method for potential energy surface

All the Density functional theory (DFT) calculations were performed with the GAUSSIAN 09 series of programs[8]. DFT method M06-2X[9] with a standard 6-31+G(d,p)[10] basis set was used for geometry optimizations. The vibrational frequencies at the same level were computed to characterize all optimized structure as minima or a transition state and to evaluate its zero-point vibrational energy (ZPVE) and thermal corrections at 298 K. The energies given in this work are Gibbs free energies in gas phase (Δ*G*).

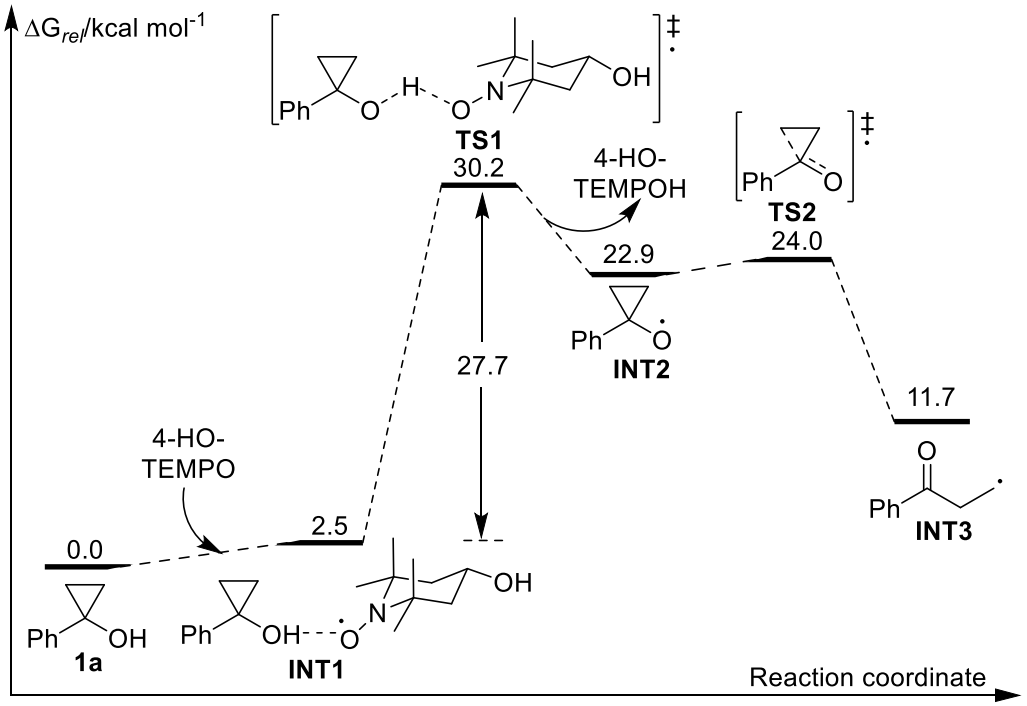


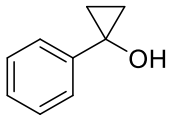
Figure S3. DFT-computed energy profiles for 4-HO-TEMPO mediated the generation of β-keto alkyl radical.

Table S1. Sum of electronic and thermal free energies (G, in a.u.), sum of electronic and thermal enthalpies (H, in a.u.), Sum of electronic and zero-point energies (E, in a.u.), and imaginary frequencies for the transition states (IF).

structure	*G*	*H*	*E*	*IF*
1a	-423.873053	-423.830445	-423.839773	
4-HO-TEMPO	-558.488961	-558.437295	-558.451195	
INT1	-982.357967	-982.281667	-982.305784	
TS1	-982.313867	-982.239476	-982.262679	-2079.7
4-HO-TEMPOH	-559.092059	-559.041405	-559.055381	
INT2	-423.233522	-423.190602	-423.199782	
TS2	-423.231651	-423.188896	-423.197913	-484.9
INT3	-423.251305	-423.205538	-423.215774	

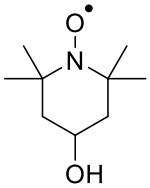
Coordinates for the ground states and transition states involved

1a



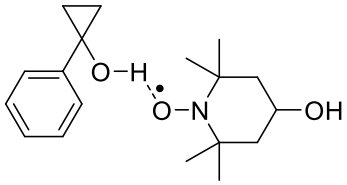
C	-2.24696400	0.73685000	-0.83591900
C	-1.42824600	-0.19051400	0.01047900
C	-2.28479400	0.86256200	0.66455000
H	-3.10946300	0.29355300	-1.32061300
H	-3.17729600	0.50130600	1.16423100
H	-1.79703600	1.70760700	1.13835600
O	-1.92514500	-1.50006300	0.05101400
H	-1.82745700	-1.83392200	0.95149600
C	0.06464000	-0.06957200	0.01241500
C	0.69628600	1.17993000	0.06250300
C	0.85931400	-1.21731700	-0.05450900
C	2.08391100	1.27664300	0.05474600
H	0.10515000	2.09110600	0.10395000
C	2.25053000	-1.11963700	-0.05959200
H	0.38104600	-2.18845000	-0.12438400
C	2.86979300	0.12545400	-0.00267500
H	2.55325000	2.25498600	0.09527300
H	2.84972500	-2.02368200	-0.11286700
H	3.95241500	0.20152800	-0.00701300
H	-1.71599500	1.49007400	-1.40853500

4-HO-TEMPO



C	-1.80405200	0.12810800	0.19898800
C	-1.03659800	1.31312600	-0.35732200
C	0.45615900	1.28980100	-0.01297900
C	0.27865400	-1.33814200	-0.01678600
C	-1.20314900	-1.15308200	-0.36362600
H	-1.16591000	1.30639900	-1.44736900
H	-1.47078200	2.24655000	0.01700000
H	-1.32676900	-1.12321200	-1.45394600
H	-1.74980400	-2.03217500	0.00329600
O	2.28857400	-0.15619600	-0.25157000
N	1.01728300	-0.06852900	-0.27383000
C	0.71066300	1.66908300	1.45487900
H	1.76374900	1.49814800	1.69420500
H	0.48310200	2.72920400	1.60304800
H	0.09472700	1.09252900	2.14941600
C	1.20280700	2.26904000	-0.92032400
H	1.12698300	1.95412300	-1.96493700
H	0.75708600	3.26317600	-0.81833500
H	2.25835700	2.31816700	-0.64872400
C	0.47446200	-1.75419800	1.45017100
H	0.09159400	-2.76921700	1.59642000
H	1.54110700	-1.74349800	1.68988900
H	-0.04625200	-1.09279900	2.14700000
C	0.88577800	-2.40760200	-0.92666900
H	0.31260100	-3.33441300	-0.82573800
H	0.85131100	-2.08344200	-1.97064800
H	1.92588200	-2.59758500	-0.65757300
O	-3.15502900	0.29043600	-0.20586900
H	-3.67609000	-0.44889300	0.12711900
H	-1.74858000	0.12592000	1.29819500

INT1



C	4.67700400	1.16212700	-0.21735100

C	3.21325100	0.87921200	-0.22286200
C	3.76616200	1.83971500	0.78874800
H	5.04597200	1.76584700	-1.03908000
H	3.54277100	2.88819100	0.62627000
H	3.83294800	1.52478200	1.82518900
O	2.52856700	1.42642900	-1.31640000
H	1.60492600	1.55895100	-1.04058500
C	-3.59951100	-0.66350500	-0.13121700
C	-2.43788300	-1.14287900	-0.97992600
C	-1.43282600	-0.04171800	-1.33327700
C	-2.08468500	1.08038900	0.96437600
C	-3.04662800	-0.09533400	1.16872400
H	-1.92688100	-1.93999200	-0.42388200
H	-2.81384000	-1.58297300	-1.90977000
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H	-3.85987400	0.24437800	1.82368700
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H	0.31097200	-1.29707400	-1.04607600
H	-0.40092400	-1.36000000	-2.67238400
H	0.57936300	0.03649400	-2.18562700
C	-2.82700400	2.37095200	0.58088300
H	-3.41996800	2.71694400	1.43317900
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H	-0.61765700	2.16392800	2.14453100
C	2.69133400	-0.45992000	0.21387600
C	1.77767300	-0.55485300	1.26419700
C	3.04191600	-1.61611500	-0.48870900
C	1.21140900	-1.78307600	1.60415200
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C	2.48512700	-2.84702600	-0.14872800
H	3.74828000	-1.53802200	-1.31138900
C	1.56422700	-2.93235400	0.89710400
H	0.49899700	-1.84706800	2.42269300
H	2.76468800	-3.73995800	-0.69971000

H	1.12763600	-3.89067300	1.16203600
H	5.34934800	0.39155900	0.14481400
O	-4.42674800	-1.79188900	0.10502900
H	-5.19261700	-1.52178000	0.62437500
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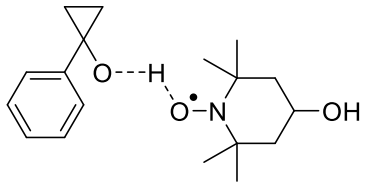
TS1



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H	3.63762500	3.12170800	-0.70648200
H	2.09726000	2.84551800	1.25584300
H	3.09593000	1.41654000	1.85776600
O	1.48402900	1.81775400	-1.01196200
H	0.29195800	1.91135900	-0.38615100
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C	-2.76402200	-0.72725700	-1.13788200
C	-2.25822900	0.71439300	-0.99510400
C	-1.40897900	0.08971800	1.36323900
C	-1.94641200	-1.32731700	1.12851400
H	-1.97611200	-1.34357000	-1.59116000
H	-3.62235900	-0.72747000	-1.82361600
H	-1.15349900	-1.94393100	0.68495100
H	-2.21557300	-1.77493500	2.09181200
O	-0.62149400	1.94188000	0.15678800
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H	-3.38863000	0.86844600	1.96505600
C	-0.04423100	-0.01120400	2.05195000

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C	2.65950800	-0.19289600	-0.32228800
C	3.69904800	-0.82462300	0.37021100
C	1.75885100	-0.97972900	-1.04622900
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H	1.17453500	-2.96056200	-1.63345000
H	3.02535900	-4.06887700	-0.39485600
H	-3.98736200	-0.82992400	0.64513300
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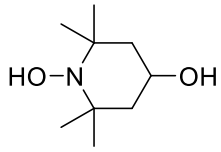
4-HO-TEMPOH



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C	0.37865200	-1.27182000	-0.01783300
C	-1.12304500	-1.27383800	-0.33346200
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O	2.30995800	0.06163600	-0.17090000
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C	0.56118800	1.60896100	1.46455300
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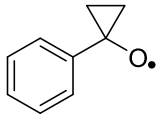
H	0.35242600	2.66829100	1.64577600
H	-0.06986000	1.03022200	2.14066600
C	0.92838500	2.41360600	-0.86158400
H	0.86309900	2.16786600	-1.92585600
H	0.39134700	3.35068100	-0.68359200
H	1.97761800	2.56661500	-0.59440000

C	0.64771400	-1.57426200	1.46819700
H	0.47331900	-2.63859200	1.65550300
H	1.68944600	-1.34987600	1.71267300
H	0.00203800	-1.01375300	2.14582900
C	1.05338000	-2.36414700	-0.85538900
H	0.56404800	-3.32680200	-0.67767600
H	0.97654700	-2.12331000	-1.92001900
H	2.10870600	-2.46309800	-0.58655400
H	-1.81116400	-0.05452200	1.30609900
O	-3.20149600	-0.15905300	-0.21500400
H	-3.69182000	0.60302100	0.11312000

INT2



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C	2.34553200	-0.74760000	0.72722000
H	3.17430000	-0.28923400	-1.25130000
H	3.17326200	-0.28833600	1.25299200
H	1.77775200	-1.49784400	1.26718400
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C	-0.88293300	1.23684200	0.00011500
C	-2.02010900	-1.30467000	-0.00022300
H	-0.01515800	-2.05383500	-0.00073500
C	-2.26750600	1.09128000	0.00038600
H	-0.42233800	2.21922400	0.00030400
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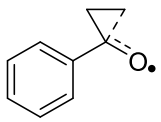
TS2



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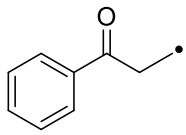
C	-1.41650100	0.38886400	-0.17156200
C	-2.36464000	-0.75280900	-0.59416800
H	-3.11986900	-0.21511600	1.40709500

H	-3.24723400	-0.35474500	-1.08471200
H	-1.88660800	-1.57770000	-1.11552600
O	-1.87566800	1.55117400	-0.10241100
H	-1.71382500	-1.43298600	1.41861400
C	0.05818100	0.12665600	-0.07992700
C	0.62312200	-1.15304700	-0.12603600
C	0.89965300	1.23747300	0.03494200
C	2.00413200	-1.31721500	-0.05740500
H	-0.00535200	-2.03589800	-0.20886600
C	2.27976000	1.07212300	0.10010200
H	0.44775900	2.22336200	0.06897000
C	2.83715700	-0.20513200	0.05464300
H	2.42903900	-2.31556100	-0.09325900
H	2.92269600	1.94274100	0.18580100
H	3.91366800	-0.33462900	0.10583200

INT3



C	2.62174700	1.23474800	0.77601000
C	1.34005200	-0.63822500	-0.21797300
C	2.39768400	0.44210600	-0.46925100
H	3.08983600	0.76196300	1.63042800
H	3.30348100	-0.09851400	-0.76020000
H	2.09186900	1.08485600	-1.30016000
O	1.67322400	-1.79638700	-0.07138500
H	2.21589600	2.23038700	0.89901700
C	-0.10141000	-0.23445300	-0.11164000
C	-0.52515600	1.08785000	-0.27533600
C	-1.04624400	-1.23340000	0.14824600
C	-1.87728900	1.40800400	-0.17743300
H	0.19306200	1.87631100	-0.47671300
C	-2.39562100	-0.91365600	0.24570000
H	-0.69600200	-2.25346600	0.27011600
C	-2.81256200	0.40828600	0.08310400
H	-2.20036700	2.43624500	-0.30476200
H	-3.12435000	-1.69260700	0.44656100
H	-3.86643100	0.65835800	0.15822700

Calculation method for bond dissociation energies

All the Density functional theory (DFT) calculations were performed with the GAUSSIAN

09 series of programs.[8] DFT method uB3LYP[11] with a standard 6-31+G(d)[12] basis set was used for geometry optimizations. The vibrational frequencies at the same level were computed to characterize all optimized structure as minima or a transition state and to evaluate its zero-point vibrational energy (ZPVE) and thermal corrections at 298 K. The energies given in this work are electronic total energy in gas phase (HF).

compound	BDE (kcal mol^{-1})
1a	**89.5**

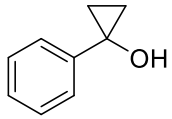
Figure S4. DFT-Calculated O-H Bond Dissociation Energy.

Table S2. Sum of electronic and thermal free energies (G, in a.u.), sum of electronic and thermal enthalpies (H, in a.u.), Sum of electronic and zero-point energies (E, in a.u.), Electronic total energy (HF, in a.u.) and imaginary frequencies for the transition states (IF).

structure	***H***	***G***	***E***	***HF***
1a	-424.001282	-424.044429	-424.010731	-424.17764500
1a'	-423.371940	-423.415585	-423.381416	-423.53479312
H-atom	-0.497912	-0.510927	-0.500273	-0.50027278

Coordinates for the ground states and transition states involved

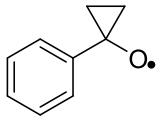
1a



C	2.10717900	-1.27526000	0.08857200
C	0.71434800	-1.18753900	0.11076800
C	0.06719800	0.05760400	0.02680200
C	0.86064800	1.21011700	-0.08593500
C	2.25554100	1.12243900	-0.10460800
C	2.88710000	-0.11955900	-0.01584200
H	2.58332500	-2.25044100	0.15674700
H	0.13477200	-2.10314000	0.19435700
H	0.37711000	2.17750700	-0.17531300
H	2.84734400	2.03036300	-0.19365700
H	3.97175000	-0.18854500	-0.02991800
C	-1.43113500	0.17988400	0.03000300
C	-2.32320700	-0.91309000	0.58462400
C	-2.26912900	-0.65721000	-0.90484100
O	-1.92366000	1.49729300	0.17267000
H	-1.78602600	1.77692500	1.09298500
H	-3.21041100	-0.57713000	1.11484500

H	-1.87010400	-1.81597100	0.98474000
H	-3.11770000	-0.15891600	-1.36528300
H	-1.75203300	-1.37330400	-1.53812100

1a'



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C	0.64031200	-1.16297100	-0.00032900
C	0.05437200	0.11430000	-0.00027200
C	0.89594000	1.23805400	0.00007800
C	2.28332800	1.08880100	0.00023500
C	2.85716100	-0.18619500	0.00010700
H	2.46169700	-2.30900100	-0.00019800
H	0.02283600	-2.05805800	-0.00050400
H	0.44354500	2.22445500	0.00012900
H	2.91799200	1.97158700	0.00044500
H	3.93788300	-0.30224800	0.00024900
C	-1.43161200	0.34699800	-0.00025900
C	-2.37131300	-0.75179900	0.72734100
C	-2.37152100	-0.75186500	-0.72669500
O	-1.90928800	1.54101800	-0.00030200
H	-3.19349000	-0.28584900	1.25965300
H	-1.81719500	-1.51192300	1.27024000
H	-3.19399400	-0.28632400	-1.25887600
H	-1.81743300	-1.51222400	-1.26931400

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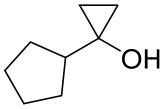
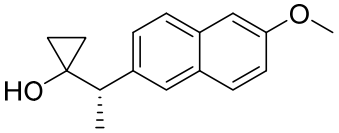
Analytical Data for Substrates:

1-Cyclopentylcyclopropan-1-ol (1m)



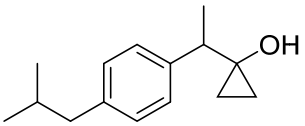
Colorless oil; (0.96 g, 76%); R_f = 0.38 (hexanes/ethyl acetate 5:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.92–1.83 (m, 2H), 1.77–1.68 (m, 2H), 1.67–1.61 (m, 2H), 1.59–1.50 (m, 2H), 1.40–1.31 (m, 2H), 0.70 (dd, *J* = 5.2 Hz, *J* = 6.8 Hz, 2H), 0.48 (dd, *J* = 5.2 Hz, *J* = 6.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 57.5, 46.5, 28.6, 25.6, 12.4; MS (ESI, m/z): Calcd for $C_8H_{15}O$, $[M+H]^+$ 127.1, found 127.5.

(S)-1-(1-(6-Methoxynaphthalen-2-yl)ethyl)cyclopropan-1-ol (1r)



White solid; (2.18 g, 90%); mp: 95–97 °C; R_f = 0.27 (hexanes/ethyl acetate 5:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73–7.68 (m, 3H), 7.44 (dd, *J* = 8.4 Hz, *J* = 1.6 Hz, 1H), 7.16–7.12 (m, 2H), 3.91 (s, 3H), 2.69 (q, *J* = 7.2 Hz, 1H), 1.83 (s, 1H), 1.47 (d, *J* = 7.2 Hz, 3H), 0.89–0.83 (m, 1H), 0.76–0.69 (m, 2H), 0.68–0.63 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.3, 138.9, 133.4, 129.2, 128.9, 127.2, 126.7, 125.9, 118.7, 105.4, 59.6, 55.2, 45.6, 16.4, 13.4, 12.7; MS (ESI, m/z): Calcd for $C_{16}H_{19}O_2$, $[M+H]^+$ 243.1, found 243.9.

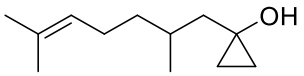
1-(1-(4-Isobutylphenyl)ethyl)cyclopropan-1-ol (1s)



White solid; (1.92 g, 88%); mp: 31–33 °C; R_f = 0.43 (hexanes/ethyl acetate 5:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 (d, *J* = 8.0 Hz, 2H), 7.11 (d, *J* = 7.6 Hz, 2H), 2.53 (q, *J* = 7.2 Hz, 1H), 2.46 (d, *J* = 7.2 Hz, 2H), 1.86 (sep, *J* = 6.8 Hz, 1H), 1.77 (s, 1H), 1.38 (d, *J* = 7.2 Hz, 3H),

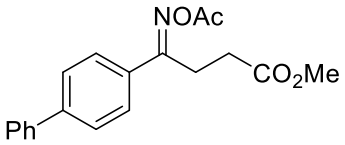
0.91 (d, J = 6.4 Hz, 6H), 0.86–0.81 (m, 1H), 0.74–0.65 (m, 2H), 0.63–0.58 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.8, 139.6, 128.9, 127.5, 59.4, 45.3, 44.9, 30.1, 22.33, 22.31, 16.4, 13.2, 12.5; MS (ESI, m/z): Calcd for $C_{15}H_{22}NaO$, $[M+Na]^+$ 241.2, found 241.1.

1-(2,6-Dimethylhept-5-en-1-yl)cyclopropan-1-ol (1t)



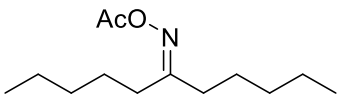
Colorless oil; (1.55 g, 85%); R_f = 0.43 (hexanes/ethyl acetate 5:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.11–5.10 (m, 1H), 2.04–1.96 (m, 2H), 1.87–1.80 (m, 1H), 1.75–1.71 (m, 1H), 1.69 (s, 3H), 1.61 (s, 3H), 1.50–1.41 (m, 1H), 1.34–1.14 (m, 3H), 1.00 (d, J = 6.4 Hz, 3H), 0.79–0.70 (m, 2H), 0.50–0.46 (m, 1H), 0.42–0.38 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 131.2, 124.8, 54.3, 45.2, 37.4, 30.2, 25.7, 25.5, 20.0, 17.7, 14.3, 13.3; MS (ESI, m/z): Calcd for $C_{12}H_{23}O$, $[M+H]^+$ 183.2, found 183.4.

Methyl (Z)-4-([1,1'-biphenyl]-4-yl)-4-(acetoxyimino)butanoate (2r)



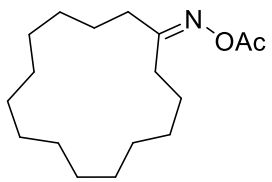
White solid; (2.35 g, 62%); mp: 57–59 ^{o}C; R_f = 0.53 (hexanes/ethyl acetate 2:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 (d, J = 8.0 Hz, 2H), 7.63 (dd, J = 15.2 Hz, J = 8.0 Hz, 4H), 7.46 (t, J = 7.6 Hz, 2H), 7.38 (t, J = 7.2 Hz, 1H), 3.68 (s, 3H), 3.21 (t, J = 7.6 Hz, 2H), 2.62 (t, J = 8.0 Hz, 2H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.2, 168.7, 164.0, 143.6, 140.0, 132.0, 128.8, 127.9, 127.7, 127.4, 127.0, 51.9, 30.8, 23.5, 19.8; MS (ESI, m/z): Calcd for $C_{19}H_{19}NNaO_4$, $[M+Na]^+$ 348.1, found 348.4.

Undecan-6-one O-acetyl oxime (2s)



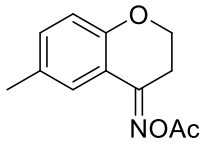
Colorless oil; (1.91 g, 84%); R_f = 0.42 (hexanes/ethyl acetate 5:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.38–2.29 (m, 4H), 2.16 (s, 3H), 1.60–1.48 (m, 4H), 1.37–1.30 (m, 8H), 0.92–0.88 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.5, 168.4, 33.7, 31.4, 31.1, 28.7, 25.7, 25.2, 22.0, 21.8, 19.3, 13.5, 13.4; MS (ESI, m/z): Calcd for $C_{13}H_{26}NO_2$, $[M+H]^+$: 228.2, found 228.0.

Cyclopentadecanone O-acetyl oxime (2w)



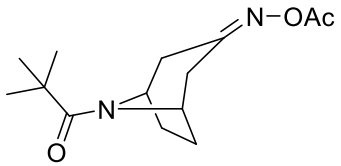
Colorless oil; (2.44 g, 87%); R_f = 0.32 (hexanes/ethyl acetate 5:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.41–2.32 (m, 4H), 2.16 (s, 3H), 1.60 (dt, J = 6.8 Hz, J = 21.6 Hz, 4H), 1.44–1.33 (m, 20H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.8, 168.7, 34.1, 28.8, 27.5, 27.2, 26.3, 26.14, 26.11, 26.03, 25.97, 24.9, 24.4, 19.5; MS (ESI, m/z): Calcd for $C_{17}H_{31}NNaO_2$, $[M+Na]^+$ 304.2, found 304.2.

6-Methylchroman-4-one O-acetyl oxime (2y)



White solid; (1.91 g, 87%); mp: 94–98 °C; R_f = 0.38 (hexanes/ethyl acetate 5:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (s, 1H), 7.13–7.11 (m, 1H), 6.80–6.78 (m, 1H), 4.21–4.18 (m, 2H), 2.99 (t, J = 6.4 Hz, 2H), 2.27 (s, 3H), 2.24 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.4, 155.8, 155.5, 133.6, 130.6, 124.8, 117.3, 115.8, 64.3, 25.0, 20.2, 19.4; MS (ESI, m/z): Calcd for $C_{12}H_{14}NO_3$, $[M+H]^+$ 220.1, found 220.3.

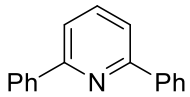
1-((1R,5S,Z)-3-(Acetoxyimino)-8-azabicyclo[3.2.1]octan-8-yl)-2,2-dimethylpropan-1-one (2z)



White solid; (1.33 g, 50%); mp: 151–153 °C; R_f = 0.28 (hexanes/ethyl acetate 2:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.84 (d, J = 18.4 Hz, 2H), 3.15 (d, J = 15.6 Hz, 1H), 2.68–2.58 (m, 2H), 2.36 (dd, J = 4.0 Hz, J = 15.6 Hz, 1H), 2.17 (s, 3H), 1.99 (brs, 2H), 1.82–1.78 (m, 1H), 1.60–1.58 (m, 1H), 1.29 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 174.4, 168.3, 163.4, 53.4, 52.3, 39.1, 38.5, 34.4, 27.9, 19.5; MS (ESI, m/z): Calcd for $C_{14}H_{23}N_2O_3$, $[M+H]^+$ 267.2, found 267.6.

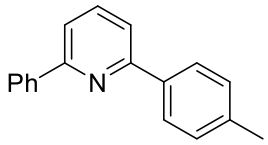
Analytical data for products:

2,6-Diphenylpyridine (3a)



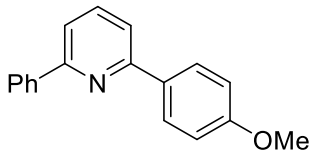
Yellow solid; (88 mg, 76%); mp: 78–80 °C; R_f = 0.44 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.15 (d, *J* = 7.2 Hz, 4H), 7.78 (t, *J* = 8.0 Hz, 1H), 7.66 (d, *J* = 8.0 Hz, 2H), 7.49 (t, *J* = 7.2 Hz, 4H), 7.42 (t, *J* = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.7, 139.4, 137.4, 128.9, 128.6, 126.9, 118.6; MS (ESI, m/z): Calcd for $C_{17}H_{14}N$, $[M+H]^+$:232.1, found 232.3.

2-Phenyl-6-(p-tolyl)pyridine (3b)



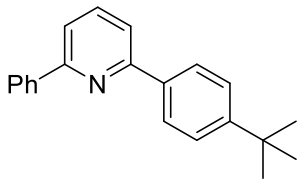
White solid; (87 mg, 71%); mp: 83–85 °C; R_f = 0.44 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14 (d, *J* = 7.2 Hz, 2H), 8.04 (d, *J* = 7.6 Hz, 2H), 7.75 (t, *J* = 8.0 Hz, 1H), 7.64 (d, *J* = 7.6 Hz, 2H), 7.48 (t, *J* = 7.2 Hz, 2H), 7.41 (t, *J* = 7.2 Hz, 1H), 7.28 (d, *J* = 7.6 Hz, 2H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.7, 156.6, 139.5, 138.9, 137.4, 136.6, 129.4, 128.8, 128.6, 126.9, 126.8, 118.3, 118.2, 21.3; MS (ESI, m/z): Calcd for $C_{18}H_{16}N$, $[M+H]^+$: 246.1, found 246.8.

2-(4-Methoxyphenyl)-6-phenylpyridine (3c)



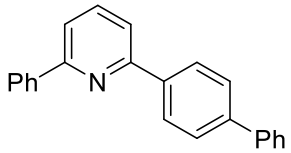
White solid; (79 mg, 60%); mp: 131–133 °C; R_f = 0.35 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.16–8.11 (m, 4H), 7.78 (t, *J* = 8.0 Hz, 1H), 7.64 (dd, *J* = 1.6 Hz, *J* = 8.0 Hz, 2H), 7.52–7.48 (m, 2H), 7.44–7.41 (m, 1H), 7.02 (dd, *J* = 2.0 Hz, *J* = 6.8 Hz, 2H), 3.88 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.4, 156.6, 156.4, 139.6, 137.4, 132.1, 128.9, 128.6, 128.2, 126.9, 117.9, 117.8, 114.0, 55.4; MS (ESI, m/z): Calcd for $C_{18}H_{16}NO$, $[M+H]^+$: 262.1, found 262.2.

2-(4-(Tert-butyl)phenyl)-6-phenylpyridine (3d)



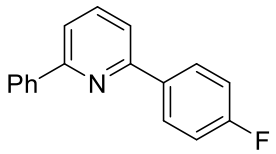
Yellow solid; (104 mg, 72%); mp: 93–95 °C; R_f = 0.53 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.15 (d, J = 7.2 Hz, 2H), 8.08 (m, J = 8.4 Hz, 2H), 7.78 (t, J = 8.0 Hz, 1H), 7.66 (d, J = 7.6 Hz, 2H), 7.53–7.47 (m, 4H), 7.42 (t, J = 7.2 Hz, 1H), 1.37(s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.8, 156.6, 152.0, 139.5, 137.4, 136.7, 128.9, 128.6, 126.9, 126.7, 125.6, 118.4, 118.3, 34.7, 31.3; MS (ESI, m/z): Calcd for $C_{21}H_{22}N$, $[M+H]^+$: 288.2, found 288.8.

2-([1,1'-Biphenyl]-4-yl)-6-phenylpyridine (3e)



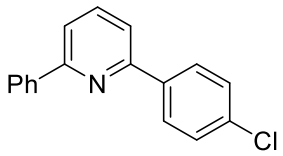
White solid; (103 mg, 67%); mp: 125–128 °C; R_f = 0.45 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.24 (d, J = 8.0 Hz, 2H), 8.17 (d, J = 7.6 Hz, 2H), 7.82 (t, J = 7.6 Hz, 1H), 7.74–7.66 (m, 6H), 7.53–7.42 (m, 5H), 7.37 (t, J = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.8, 156.3, 141.6, 140.6, 139.4, 138.3, 137.5, 129.0, 128.8, 128.7, 127.5, 127.4, 127.3, 127.1, 127.0, 118.6, 118.5; MS (ESI, m/z): Calcd for $C_{23}H_{18}N$, $[M+H]^+$: 308.1, found 308.2.

2-(4-Fluorophenyl)-6-phenylpyridine (3f)



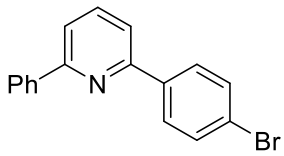
Yellow solid; (102 mg, 82%); mp: 74–76 °C; R_f = 0.58 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14–8.11 (m, 4H), 7.77 (t, J = 8.0 Hz, 1H), 7.66 (dd, J = 0.8 Hz, J = 8.0 Hz, 1H), 7.61 (dd, J = 0.8 Hz, J = 7.6 Hz, 1H), 7.51–7.47 (m, 2H), 7.45–7.41 (m, 1H), 7.16 (t, J = 8.8 Hz, 2H), ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.5 (d, $^{C,F}J_1$ = 246 Hz), 162.3, 156.8, 155.7, 139.3, 137.5, 135.5 (d, $^{C,F}J_3$ = 3 Hz), 129.0, 128.8, 128.7, 126.9, 118.4 (d, $^{C,F}J_2$ = 29 Hz); MS (ESI, m/z): Calcd for $C_{17}H_{13}FN$, $[M+H]^+$: 250.1, found 250.4.

2-(4-Chlorophenyl)-6-phenylpyridine (3g)



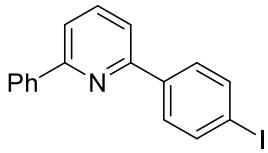
Yellow solid; (110 mg, 83%); mp: 75–77 °C; R_f = 0.58 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.13–8.04 (m, 4H), 7.77 (t, J = 8.0 Hz, 1H), 7.67 (d, J = 7.6 Hz, 1H), 7.61 (d, J = 7.6 Hz, 1H), 7.50–7.40 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.8, 155.4, 139.2, 137.8, 137.6, 135.0, 129.1, 128.8, 128.7, 128.2, 126.9, 118.8, 118.3; MS (ESI, m/z): Calcd for $C_{17}H_{13}ClN$, $[M+H]^+$: 266.1, found 266.5.

2-(4-Bromophenyl)-6-phenylpyridine (3h)



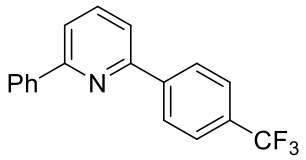
Yellow solid; (127 mg, 82%); mp: 96–98 °C; R_f = 0.55 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14–8.11 (m, 2H), 8.02 (d, J = 8.4 Hz, 2H), 7.79 (t, J = 8.0 Hz, 1H), 7.69 (d, J = 7.6 Hz, 1H), 7.65–7.60 (m, 3H), 7.50 (t, J = 7.6 Hz, 2H), 7.45–7.42 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.9, 155.5, 139.2, 138.2, 137.6, 131.8, 129.1, 128.7, 128.5, 126.9, 123.4, 118.9, 118.3; MS (ESI, m/z): Calcd for $C_{17}H_{12}BrKN$, $[M+K]^+$: 348.0, found 347.8.

2-(4-Iodophenyl)-6-phenylpyridine (3i)



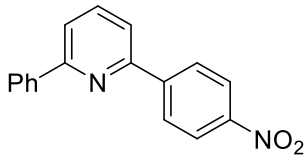
Yellow solid; (152 mg, 85%); mp: 128–130 °C; R_f = 0.53 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.12 (d, J = 7.6 Hz, 2H), 7.89 (d, J = 8.4 Hz, 2H), 7.82–7.78 (m, 3H), 7.70 (d, J = 7.6 Hz, 1H), 7.64 (d, J = 7.6 Hz, 1H), 7.52–7.48 (m, 2H), 7.45–7.42 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.9, 155.6, 139.2, 138.9, 137.8, 137.6, 129.1, 128.70, 128.67, 126.9, 119.0, 118.3, 95.3; MS (ESI, m/z): Calcd for $C_{17}H_{13}IN$, $[M+H]^+$: 358.0, found 357.9.

2-Phenyl-6-(4-(trifluoromethyl)phenyl)pyridine (3j)



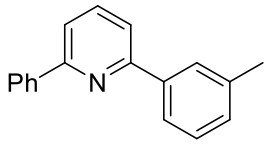
Yellow solid; (120 mg, 80%); mp: 109–112 °C; R_f = 0.45 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25 (d, J = 8.0 Hz, 2H), 8.16–8.13 (m, 2H), 7.85 (t, J = 8.0 Hz, 1H), 7.52 (t, J = 7.2 Hz, 2H), 7.47–7.43 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.1, 155.2, 142.7, 139.1, 137.7, 130.7 (q, $^{C,F}J_2$ = 32 Hz), 129.2, 128.8, 127.2, 126.9, 125.6 (q, $^{C,F}J_3$ = 4 Hz), 124.2 (q, $^{C,F}J_1$ = 270 Hz), 119.5, 119.0; MS (ESI, m/z): Calcd for $C_{18}H_{13}F_3N$, $[M+H]^+$: 300.1, found 300.5.

2-(4-Nitrophenyl)-6-phenylpyridine (3k)



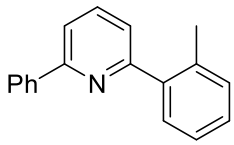
White solid; (112 mg, 81%); mp: 127−129 °C; R_f = 0.63 (hexanes/ethyl acetate 3:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.32−8.26 (m, 4H), 8.14−8.12 (m, 2H), 7.85 (t, J = 8.0 Hz, 1H), 7.76 (d, J = 7.2 Hz, 1H), 7.73−7.71 (m, 1H), 7.54−7.50 (m, 2H), 7.48−7.44 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.2, 154.0, 148.0, 145.2, 138.7, 137.8, 129.3, 128.8, 127.6, 126.9, 123.8, 120.0, 119.2; MS (ESI, m/z): Calcd for $C_{17}H_{13}N_2O_2$, $[M+H]^+$: 277.1, found 277.2.

2-Phenyl-6-(m-tolyl)pyridine (3l)



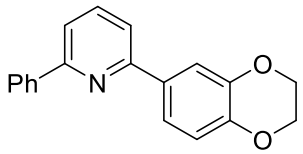
Yellow oil; (90 mg, 73%); R_f = 0.47 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14 (d, J = 7.2 Hz, 2H), 7.97 (s, 1H), 7.91 (d, J = 7.6 Hz, 1H), 7.76 (t, J = 7.6 Hz, 1H), 7.65 (d, J = 7.2 Hz, 2H), 7.48 (t, J = 7.2 Hz, 2H), 7.43–7.35 (m, 2H), 7.23 (d, J = 7.2 Hz, 1H), 2.45(s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.9, 156.7, 139.5, 139.4, 138.2, 137.4, 129.7, 128.9, 128.6, 128.5, 127.6, 127.0, 124.0, 118.7, 118.6, 21.6; MS (ESI, m/z): Calcd for $C_{18}H_{15}KN$, $[M+K]^+$: 284.1, found 284.0.

2-Phenyl-6-(o-tolyl)pyridine (3m)



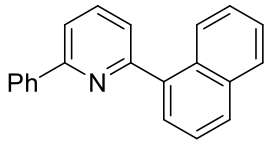
Yellow oil; (43 mg, 35%); R_f = 0.47 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.08 (d, J = 7.6 Hz, 2H), 7.81 (t, J = 7.6 Hz, 1H), 7.69 (d, J = 8.0 Hz, 1H), 7.50–7.45 (m, 3H), 7.41 (d, J = 7.2 Hz, 1H), 7.38–7.35 (m, 1H), 7.31–7.28 (m, 3H), 2.49 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.8, 156.5, 140.6, 139.5, 136.9, 136.1, 130.9, 129.8, 128.9, 128.6, 128.2, 127.0, 125.8, 122.4, 118.2, 20.7; MS (ESI, m/z): Calcd for $C_{18}H_{15}KN$, $[M+K]^+$: 284.1, found 284.0.

2-(2,3-Dihydrobenzo[b][1,4]dioxin-6-yl)-6-phenylpyridine (3n)



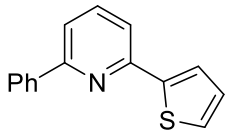
Colorless oil; (90 mg, 62%); R_f = 0.58 (hexanes/ethyl acetate 3:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.15–8.12 (m, 2H), 7.75–7.71 (m, 2H), 7.65–7.57 (m, 3H), 7.49–7.46 (m, 2H), 7.42–7.39 (m, 1H), 6.97 (d, J = 8.4 Hz, 1H), 4.28 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.4, 156.0, 144.5, 143.6, 139.4, 137.3, 133.0, 128.9, 128.6, 126.9, 120.1, 118.0, 117.9, 117.3, 115.9, 64.5, 64.3; ESI-HRMS: m/z Calcd for $C_{19}H_{16}NO_2$, $[M+H]^+$: 290.1176, found 290.1173.

2-(Naphthalen-1-yl)-6-phenylpyridine (3o)



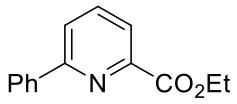
Colorless oil; (106 mg, 75%); R_f = 0.52 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25 (d, J = 8.0 Hz, 1H), 8.10 (d, J = 7.6 Hz, 2H), 7.90 (d, J = 8.0 Hz, 2H), 7.82 (t, J = 7.6 Hz, 1H), 7.74 (d, J = 6.8 Hz, 1H), 7.67 (d, J = 6.8 Hz, 1H), 7.56–7.37 (m, 7H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.9, 156.8, 139.3, 138.7, 137.1, 133.9, 131.2, 128.9, 128.8, 128.7, 128.3, 127.6, 127.0, 126.3, 125.85, 125.77, 125.3, 123.4, 118.5; ESI-HRMS: m/z Calcd for $C_{21}H_{16}N$, $[M+H]^+$: 282.1277, found 282.1271.

2-Phenyl-6-(thiophen-2-yl)pyridine (3p)



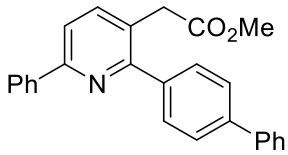
Yellow solid; (81 mg, 68%); mp: 69–71 °C; R_f = 0.55 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.12–8.09 (m, 2H), 7.70 (t, J = 8.0 Hz, 1H), 7.63 (dd, J = 1.2 Hz, J = 3.6 Hz, 1H), 7.59–7.54 (m, 2H), 7.50–7.45 (m, 2H), 7.43–7.38 (m, 2H), 7.11 (dd, J = 3.6 Hz, J = 5.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.6, 152.2, 145.5, 138.9, 137.3, 129.1, 128.6, 127.9, 127.6, 126.9, 124.5, 118.2, 116.9; MS (ESI, m/z): Calcd for $C_{15}H_{12}NS$, $[M+H]^+$: 238.1, found 238.4.

Ethyl 6-phenylpicolinate (3q)



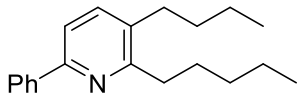
White solid; (48 mg, 42%); mp: 38–42 °C; R_f = 0.26 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.08–8.03 (m, 3H), 7.90–7.86 (m, 2H), 7.51–7.42 (m, 3H), 4.49 (q, J = 7.2 Hz, 2H), 1.47 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.5, 157.6, 148.4, 138.5, 137.6, 129.4, 128.8, 127.2, 123.4, 123.2, 61.8, 14.3; MS (ESI, m/z): Calcd for $C_{14}H_{14}NO_2$, $[M+H]^+$: 228.1, found 228.0.

Methyl 2-([1,1':3',1'':4'',1'''-quaterphenyl]-4'-yl)acetate (3r)



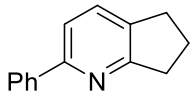
White solid; (95 mg, 50%); mp: 123–125 °C; R_f = 0.36 (hexanes/ethyl acetate 5:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.07 (d, J = 7.2 Hz, 2H), 7.77–7.64 (m, 8H), 7.47–7.43 (m, 4H), 7.40–7.34 (m, 2H), 3.76 (s, 2H), 3.68 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.8, 158.4, 155.8, 141.0, 140.7, 139.4, 139.1, 139.0, 129.6, 128.9, 128.8, 128.6, 127.4, 127.1, 126.98, 126.97, 125.8, 118.9, 52.2, 38.0; ESI-HRMS: m/z Calcd for $C_{26}H_{22}NO_2$, $[M+H]^+$: 380.1645, found 380.1643.

3-Butyl-2-pentyl-6-phenylpyridine (3s)



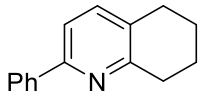
Yellow oil; (68 mg, 48%); R_f = 0.78 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 (d, J = 8.0 Hz, 2H), 7.47–7.41 (m, 4H), 7.35 (t, J = 7.2 Hz, 1H), 2.84 (t, J = 7.6 Hz, 2H), 2.63 (t, J = 8.0 Hz, 2H), 1.82 (quin, J = 7.2 Hz, 2H), 1.62–1.54 (m, 2H), 1.46–1.37 (m, 6H), 0.98–0.91 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.0, 154.0, 139.9, 137.2, 133.8, 128.5, 128.2, 126.7, 117.5, 34.9, 32.8, 32.0, 31.7, 29.0, 22.6, 14.1, 14.0; ESI-HRMS: m/z Calcd for $C_{20}H_{28}N$, $[M+H]^+$: 282.2216, found 282.2218.

2-Phenyl-6,7-dihydro-5H-cyclopenta[b]pyridine (3t)



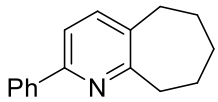
Yellow solid; (45 mg, 46%); mp: 77–79 ^{o}C; R_f = 0.45 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95–7.93 (m, 2H), 7.55 (d, J = 8.0 Hz, 1H), 7.47–7.43 (m, 3H), 7.40–7.36 (m, 1H), 3.09 (t, J = 7.6 Hz, 2H), 2.96 (t, J = 7.6 Hz, 2H), 2.16 (quin, J = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.8, 155.8, 140.0, 135.4, 132.6, 128.6, 128.3, 126.9, 118.2, 34.4, 30.5, 23.2; MS (ESI, m/z): Calcd for $C_{14}H_{14}N$, $[M+H]^+$: 196.1, found 196.0.

2-Phenyl-5,6,7,8-tetrahydroquinoline (3u)



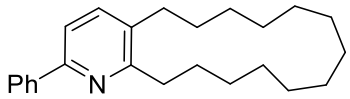
Colorless oil; (54 mg, 51%); R_f = 0.47 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95–7.92 (m, 2H), 7.45–7.33 (m, 5H), 3.00 (t, J = 6.4 Hz, 2H), 2.79 (t, J = 6.4 Hz, 2H), 1.95–1.89 (m, 2H), 1.86–1.80 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.2, 154.6, 139.9, 137.4, 130.7, 128.6, 128.3, 126.8, 117.9, 32.8, 28.5, 23.2, 22.8; MS (ESI, m/z): Calcd for $C_{15}H_{16}N$, $[M+H]^+$: 210.1, found 210.0.

2-Phenyl-6,7,8,9-tetrahydro-5H-cyclohepta[b]pyridine (3v)



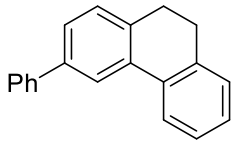
Yellow oil; (54 mg, 48%); R_f = 0.59 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98–7.95 (m, 2H), 7.45–7.40 (m, 4H), 7.38–7.33 (m, 1H), 3.14–3.11 (m, 2H), 2.81–2.78 (m, 2H), 1.92–1.86 (m, 2H), 1.76–1.67 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.1, 154.0, 139.8, 137.2, 136.5, 128.6, 128.2, 126.8, 117.9, 39.7, 35.0, 32.6, 28.1, 26.6; MS (ESI, m/z): Calcd for $C_{16}H_{18}N$, $[M+H]^+$: 224.1, found 224.0.

2-Phenyl-6,7,8,9,10,11,12,13,14,15,16,17-dodecahydro-5H-cyclopentadeca[b]pyridine (3w)



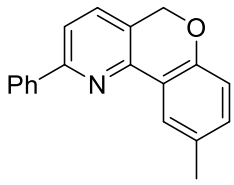
White solid; (89 mg, 53%); mp: 40–42 °C; R_f = 0.68 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99–7.97 (m, 2H), 7.45–7.41 (m, 4H), 7.36–7.33 (m, 1H), 2.87–2.83 (m, 2H), 2.64–2.60 (m, 2H), 1.87–1.79 (m, 2H), 1.66–1.61 (m, 2H), 1.59–1.54 (m, 2H), 1.53–1.48 (m, 2H), 1.46–1.42 (m, 4H), 1.34–1.28 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.1, 154.2, 139.9, 137.6, 134.0, 128.5, 128.2, 126.7, 117.7, 35.0, 32.1, 29.5, 28.1, 27.5, 27.4, 27.0, 26.9, 26.7, 26.0, 22.95, 25.6; ESI-HRMS: m/z Calcd for $C_{24}H_{34}N$, $[M+H]^+$: 336.2686, found 336.2684.

2-Phenyl-5,6-dihydrobenzo[h]quinoline (3x)



Yellow oil; (114 mg, 88%); R_f = 0.37 (hexanes/ethyl acetate 40:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.54–8.52 (m, 1H), 8.16–8.13 (m, 2H), 7.60–7.54 (m, 2H), 7.50–7.47 (m, 2H), 7.42–7.37 (m, 2H), 7.34–7.30 (m, 1H), 7.24–7.23 (m, 1H), 2.97 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.0, 152.0, 139.6, 138.2, 136.3, 134.8, 130.2, 129.0, 128.6, 127.7, 127.1, 126.7, 125.2, 118.7, 28.1, 27.8; MS (ESI, m/z): Calcd for $C_{19}H_{16}N$, $[M+H]^+$: 258.1, found 258.7.

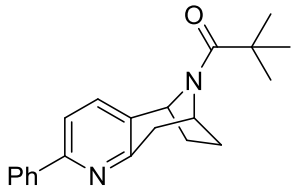
9-Methyl-2-phenyl-5H-chromeno[4,3-b]pyridine (3y)



White solid; (68 mg, 50%); mp: 76–78 °C; R_f = 0.33 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.20 (s, 1H), 8.11 (d, J = 7.6 Hz, 2H), 7.58 (d, J = 8.0 Hz, 1H), 7.49 (t, J = 7.6 Hz, 2H), 7.42 (d, J = 7.2 Hz, 2H), 7.12 (d, J = 7.6 Hz, 1H), 6.87 (d, J = 8.0 Hz, 1H), 5.19 (s, 2H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.7, 154.4, 148.4, 139.3, 132.7, 131.9, 131.6, 129.0, 128.7, 126.9, 125.0, 124.6, 123.0, 118.8, 116.6, 67.8, 20.8; MS (ESI,

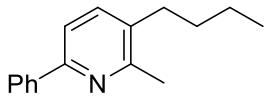
m/z): Calcd for $C_{19}H_{16}NO$, $[M+H]^+$: 274.1, found 274.2.

2,2-Dimethyl-1-(2-phenyl-6,7,8,9-tetrahydro-5H-6,9-epiminocyclohepta[b]pyridin-10-yl) propan-1-one (3z)



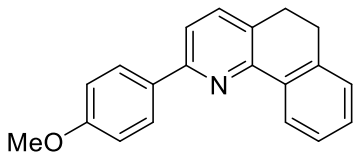
White solid; (112 mg, 70%); mp: 246–247 °C; R_f = 0.36 (hexanes/ethyl acetate 3:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 (d, J = 7.2 Hz, 2H), 7.51–7.39 (m, 5H), 5.39 (brs, 1H), 5.07 (brs, 1H), 3.56 (dd, J = 17.6 Hz, J = 4.8 Hz, 1H), 2.91 (d, J = 17.6 Hz, 1H), 2.27–2.23 (m, 2H), 2.00–1.95 (m, 1H), 1.79 (brs, 1H), 1.26 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 174.6, 156.4, 154.2, 139.3, 134.4, 133.2, 128.8, 128.7, 126.8, 118.1, 56.8, 52.9, 40.8, 39.2, 35.4, 28.1; ESI-HRMS: m/z Calcd for $C_{21}H_{25}N_2O$, $[M+H]^+$: 321.1961, found 321.1956.

3-Butyl-2-methyl-6-phenylpyridine (3aa)



Colorless oil; (53 mg, 47%); R_f = 0.28 (hexanes/ethyl acetate 40:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 (d, J = 1.6 Hz, 1H), 7.96 (d, J = 0.8Hz, 1H), 7.50–7.43 (m, 4H), 7.39–7.35 (m, 1H), 2.65–2.61 (m, 5H), 1.63–1.55 (m, 2H), 1.47–1.37 (m, 2H), 0.97 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.4, 154.2, 139.7, 137.0, 134.4, 128.6, 128.3, 126.7, 118.0, 32.2, 32.0, 22.6, 22.5, 14.0; ESI-HRMS: m/z Calcd for $C_{16}H_{20}N$, $[M+H]^+$: 226.1590, found 226.1588.

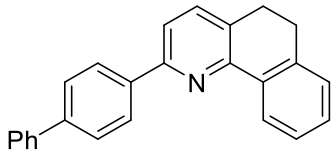
2-(4-Methoxyphenyl)-5,6-dihydrobenzo[h]quinoline (4b)



White solid; (116 mg, 81%); mp: 76–79 °C; R_f = 0.17 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.51 (d, J = 7.2 Hz, 1H), 8.08 (d, J = 8.8 Hz, 2H), 7.48 (dd, J = 8.0 Hz, J = 11.2 Hz, 2H), 7.38 (t, J = 7.2 Hz, 1H), 7.32–7.28 (m, 1H), 7.21 (d, J = 7.2 Hz, 1H), 6.99 (d, J = 8.4 Hz, 2H), 3.84 (s, 3H), 2.92 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.2, 154.7, 151.8, 138.1, 136.2, 134.9, 132.3, 129.4, 128.9, 127.9, 127.7, 127.0, 125.1, 117.9,

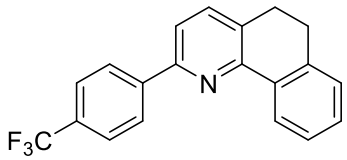
113.9, 55.3, 28.1, 27.7; MS (ESI, m/z): Calcd for $C_{20}H_{18}NO$, $[M+H]^+$: 288.1, found 288.6.

2-([1,1'-Biphenyl]-4-yl)-5,6-dihydrobenzo[h]quinoline (4c)



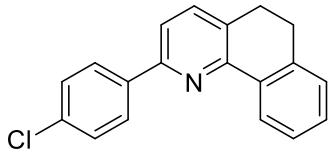
White solid; (127 mg, 76%); mp: 152–154 °C; R_f = 0.24 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.55 (dd, J = 1.2 Hz, J = 7.6 Hz, 1H), 8.22 (d, J = 8.0 Hz, 2H), 7.72–7.70 (m, 2H), 7.68–7.65 (m, 2H), 7.62 (d, J = 7.6 Hz, 1H), 7.54 (d, J = 7.6 Hz, 1H), 7.46 (t, J = 8.0 Hz, 2H), 7.42–7.30 (m, 3H), 7.24 (d, J = 8.0 Hz, 1H), 2.96 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.6, 152.0, 141.3, 140.8, 138.6, 138.2, 136.3, 134.8, 130.3, 129.0, 128.8, 127.7, 127.4, 127.3, 127.09, 127.06, 125.2, 118.6, 28.1, 27.8; ESI-HRMS: m/z Calcd for $C_{25}H_{20}N$, $[M+H]^+$: 334.1590, found 334.1592.

2-(4-(Trifluoromethyl)phenyl)-5,6-dihydrobenzo[h]quinoline (4d)



White solid; (133 mg, 82%); mp: 76–78 °C; R_f = 0.59 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.49 (d, J = 7.6 Hz, 1H), 8.23 (d, J = 8.0 Hz, 2H), 7.72 (d, J = 8.0 Hz, 2H), 7.57 (dd, J = 8.0 Hz, J = 13.6 Hz, 2H), 7.39 (t, J = 7.6 Hz, 1H), 7.33 (t, J = 7.2 Hz, 1H), 7.25–7.23 (m, 1H), 2.97 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.4, 152.4, 143.0, 138.2, 136.4, 134.5, 131.3, 130.4 (q, $^{C,F}J_2$ = 32 Hz), 129.3, 127.8, 127.2, 126.9, 125.5 (q, $^{C,F}J_3$ = 4 Hz), 125.2, 124.3 (q, $^{C,F}J_1$ = 271 Hz), 119.0, 28.0, 27.8; ESI-HRMS: m/z Calcd for $C_{20}H_{15}F_3N$, $[M+H]^+$: 326.1151, found 326.1152.

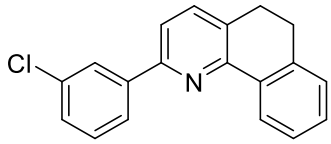
2-(4-Chlorophenyl)-5,6-dihydrobenzo[h]quinoline (4e)



White solid; (125 mg, 86%); mp: 79–81 °C; R_f = 0.34 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.48 (dd, J = 1.2 Hz, J = 7.6 Hz, 1H), 8.05 (dt, J = 2.4 Hz, J = 8.8 Hz, 2H), 7.49 (s, 2H), 7.43–7.39 (m, 2H), 7.36 (dd, J = 1.2 Hz, J = 7.6 Hz, 1H), 7.30 (td, J = 1.6 Hz, J = 7.2 Hz, 1H), 7.21 (d, J = 7.2 Hz, 1H), 2.93 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ

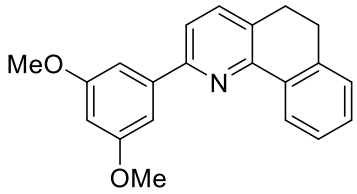
153.7, 152.1, 138.1, 138.0, 136.3, 134.61, 134.59, 130.5, 129.1, 128.7, 127.9, 127.8, 127.1, 125.2, 118.4, 28.0, 27.7; ESI-HRMS: m/z Calcd for $C_{19}H_{15}ClN$, $[M+H]^+$: 292.0888, found 292.0890.

2-(3-Chlorophenyl)-5,6-dihydrobenzo[h]quinoline (4f)



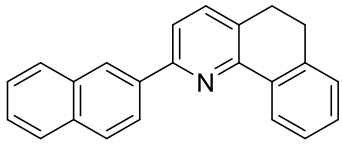
Colorless oil; (111 mg, 76%); R_f = 0.46 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.50 (dd, *J* = 1.2 Hz, *J* = 7.6 Hz, 1H), 8.16–8.15 (m, 1H), 7.98 (dt, *J* = 1.6 Hz, *J* = 7.2 Hz, 1H), 7.54 (s, 2H), 7.41–7.35 (m, 3H), 7.32 (td, *J* = 1.6 Hz, *J* = 7.2 Hz, 1H), 7.23 (d, *J* = 6.4 Hz, 1H), 2.96 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.5, 152.2, 141.5, 138.2, 136.4, 134.7, 134.6, 130.9, 129.8, 129.2, 128.5, 127.8, 127.1, 126.9, 125.2, 124.7, 118.7, 28.0, 27.8; ESI-HRMS: m/z Calcd for $C_{19}H_{15}ClN$, $[M+H]^+$: 292.0888, found 292.0889.

2-(3,5-Dimethoxyphenyl)-5,6-dihydrobenzo[h]quinoline (4g)



Yellow oil; (111 mg, 70%); R_f = 0.18 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.50 (d, *J* = 7.6 Hz, 1H), 7.55–7.50 (m, 2H), 7.39–7.31 (m, 4H), 7.22 (d, *J* = 7.2 Hz, 1H), 6.53 (s, 1H), 3.88 (s, 6H), 2.94 (s, 4H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 161.0, 154.6, 151.9, 141.8, 138.1, 136.2, 134.7, 130.6, 129.0, 127.7, 127.0, 125.2, 118.8, 104.9, 100.7, 55.4, 28.0, 27.7; ESI-HRMS: m/z Calcd for $C_{21}H_{20}NO_2$, $[M+H]^+$: 318.1489, found 318.1494.

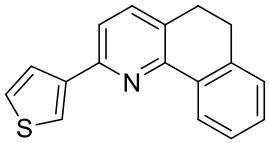
2-(Naphthalen-2-yl)-5,6-dihydrobenzo[h]quinoline (4h)



Yellow solid; (123 mg, 80%); mp: 98–101 ^{o}C; R_f = 0.37 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.59 (d, J = 7.6 Hz, 1H), 8.55 (s, 1H), 8.30 (dd, *J* = 1.6 Hz, *J* = 8.4 Hz, 1H), 7.95–7.90 (m, 2H), 7.85–7.83 (m, 1H), 7.66 (d, *J* = 8.0 Hz, 1H), 7.50–7.44 (m, 3H), 7.41 (t, *J* = 7.2 Hz, 1H), 7.31 (td, *J* = 1.2 Hz, *J* = 7.2 Hz, 1H), 7.21 (d, J = 7.2 Hz, 1H), 2.92 (s,

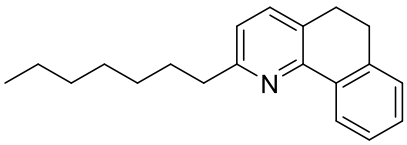
4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.8, 152.1, 138.2, 137.0, 136.3, 134.8, 133.5, 130.3, 129.0, 128.6, 128.2, 127.7, 127.6, 127.1, 126.2, 126.1, 125.8, 125.2, 124.7, 118.9, 28.1, 27.7; ESI-HRMS: m/z Calcd for $C_{23}H_{18}N$, $[M+H]^+$: 308.1434, found 308.1435.

2-(Thiophen-3-yl)-5,6-dihydrobenzo[h]quinoline (4i)



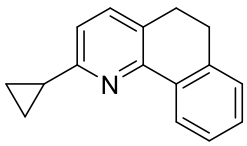
Yellow oil; (112 mg, 85%); R_f = 0.37 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.47 (d, J = 7.6 Hz, 1H), 7.95 (d, J = 2.4 Hz, 1H), 7.76 (d, J = 5.2 Hz, 1H), 7.48–7.41 (m, 2H), 7.38–7.35 (m, 2H), 7.30 (t, J = 7.2 Hz, 1H), 7.21 (d, J = 7.6 Hz, 1H), 2.92 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 152.0, 151.5, 142.7, 138.2, 136.2, 134.7, 129.9, 129.0, 127.7, 127.0, 126.4, 125.9, 125.1, 122.8, 118.6, 28.1, 27.8; MS (ESI, m/z): Calcd for $C_{17}H_{14}NS$, $[M+H]^+$: 264.1, found 264.0

2-Heptyl-5,6-dihydrobenzo[h]quinoline (4j)



Colorless oil; (103 mg, 74%); R_f = 0.46 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.35 (d, J = 7.6 Hz4, 1H), 7.37–7.32 (m, 2H), 7.27 (td, J = 1.6 Hz, J = 7.6 Hz, 1H), 7.19 (d, J = 6.8 Hz, 1H), 6.95 (d, J = 7.6 Hz, 1H), 2.92–2.84 (m, 4H), 2.81 (t, J = 8.0 Hz, 2H), 1.78 (quin, J = 7.6 Hz, 2H), 1.41–1.33 (m, 4H), 1.31–1.26 (m, 4H), 0.88 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.5, 151.6, 138.1, 135.6, 135.0, 128.7, 128.6, 127.6, 127.0, 125.0, 121.0, 38.2, 31.8, 29.8, 29.4, 29.2, 28.2, 27.7, 22.7, 14.1; ESI-HRMS: m/z Calcd for $C_{20}H_{26}N$, $[M+H]^+$: 280.2060, found 280.2056.

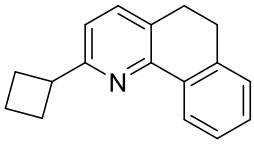
2-Cyclopropyl-5,6-dihydrobenzo[h]quinoline (4k)



Yellow oil; (90 mg, 81%); R_f = 0.44 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.30 (d, J = 7.6 Hz, 1H), 7.33–7.30 (m, 2H), 7.28–7.24 (m, 1H), 7.18 (d, J = 7.2 Hz, 1H), 6.96 (d, J = 7.6 Hz, 1H), 2.92–2.84 (m, 4H), 2.07–2.01 (m, 1H), 1.14–1.10 (m, 2H),

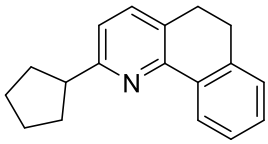
0.98–0.93 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.4, 151.4, 138.1, 135.4, 135.0, 128.6, 128.2, 127.6, 126.9, 124.9, 119.8, 28.2, 27.6, 16.9, 9.5; ESI-HRMS: m/z Calcd for $C_{16}H_{16}N$, $[M+H]^+$: 222.1277, found 222.1275.

2-Cyclobutyl-5,6-dihydrobenzo[h]quinoline (4l)



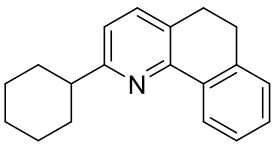
Yellow oil; (82 mg, 70%); R_f = 0.44 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.43 (dd, *J* = 0.8 Hz, *J* = 8.0 Hz, 1H), 7.38–7.33 (m, 2H), 7.27 (td, *J* = 1.2 Hz, *J* = 7.6 Hz, 1H), 7.19 (d, *J* = 7.2 Hz, 1H), 6.97 (d, *J* = 7.6 Hz, 1H), 3.69 (quin, *J* = 8.4 Hz, 1H), 2.93–2.85 (m, 4H), 2.48–2.32 (m, 4H), 2.11–2.02 (m, 1H), 2.00–1.90 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.6, 151.4, 138.1, 135.6, 135.1, 128.71, 128.69, 127.6, 127.0, 125.0, 119.5, 42.2, 28.6, 28.2, 27.7, 18.4; ESI-HRMS: m/z Calcd for $C_{17}H_{18}N$, $[M+H]^+$: 236.1434, found 236.1433.

2-Cyclopentyl-5,6-dihydrobenzo[h]quinoline (4m)



Yellow oil; (77 mg, 62%); R_f = 0.44 (hexanes/ethyl acetate 20:1); 8.37 (d, *J* = 7.6 Hz, 1H), 7.37–7.31 (m, 2H), 7.28–7.23 (m, 1H), 7.19 (d, *J* = 7.2 Hz, 1H), 6.98 (d, *J* = 7.6 Hz, 1H), 3.21 (quin, *J* = 8.0 Hz, 1H), 2.93–2.85 (m, 4H), 2.12–2.06 (m, 2H), 1.91–1.84 (m, 4H), 1.71–1.70 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.7, 151.4, 138.0, 135.6, 135.2, 128.63, 128.59, 127.6, 127.0, 125.0, 120.1, 47.7, 33.6, 28.2, 27.7, 25.9; ESI-HRMS: m/z Calcd for $C_{18}H_{20}N$, $[M+H]^+$:250.1590, found 250.1589.

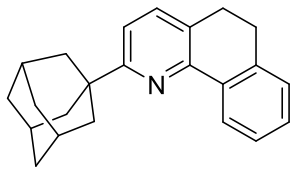
2-Cyclohexyl-5,6-dihydrobenzo[h]quinoline (4n)



Yellow oil; (100 mg, 76%); R_f = 0.44 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.38 (dd, *J* = 0.8 Hz, *J* = 7.6 Hz, 1H), 7.38–7.31 (m, 2H), 7.26 (td, *J* = 1.2 Hz, *J* = 7.2 Hz, 1H), 7.18 (d, *J* = 7.2 Hz, 1H), 6.96 (d, *J* = 7.6 Hz, 1H), 2.92–2.84 (m, 4H), 2.73 (tt, *J*

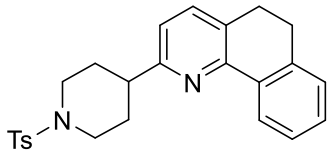
= 3.2 Hz, *J* = 7.6 Hz, 1H), 2.02–1.99 (m, 2H), 1.88–1.85 (m, 2H), 1.78–1.74 (m, 1H), 1.64–1.54 (m, 2H), 1.49–1.38 (m, 2H), 1.36–1.28 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.4, 151.3, 138.0, 135.7, 135.1, 128.7, 128.6, 127.6, 127.0, 125.0, 119.3, 46.3, 33.0, 28.2, 27.7, 26.6, 26.2; ESI-HRMS: m/z Calcd for $C_{19}H_{22}N$, $[M+H]^+$: 264.1747, found 264.1742.

2-((1s,3s)-Adamantan-1-yl)-5,6-dihydrobenzo[h]quinoline (4o)



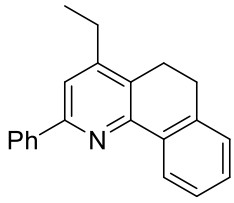
White solid; (131 mg, 83%); mp: 113–115 °C; R_f = 0.50 (hexanes/ethyl acetate 40:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.43 (d, *J* = 7.6 Hz, 1H), 7.38 (d, *J* = 7.6 Hz, 1H), 7.34 (t, *J* = 7.2 Hz, 1H), 7.26 (td, *J* = 1.2 Hz, *J* = 7.2 Hz, 1H), 7.18 (d, *J* = 7.6 Hz, 1H), 7.07 (d, *J* = 8.0 Hz, 1H), 2.92–2.84 (m, 4H), 2.12 (brs, 3H), 2.06 (d, *J* = 2.4 Hz, 6H), 1.81–1.80 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.9, 150.8, 138.0, 135.6, 135.4, 128.6, 128.3, 127.6, 126.9, 125.0, 117.2, 42.1, 39.0, 37.0, 28.9, 28.2, 27.6; ESI-HRMS: m/z Calcd for $C_{23}H_{26}N$, $[M+H]^+$: 316.2060, found 316.2057.

2-(1-Tosylpiperidin-4-yl)-5,6-dihydrobenzo[h]quinoline (4p)



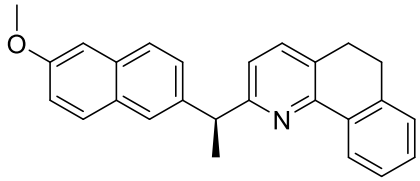
White solid; (131 mg, 48%); mp: 220–222 °C; R_f = 0.36 (hexanes/ethyl acetate 3:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.28 (dd, *J* = 0.8 Hz, *J* = 7.6 Hz, 1H), 7.70 (d, *J* = 7.6 Hz, 2H), 7.40 (d, *J* = 7.6 Hz, 1H), 7.36–7.26 (m, 4H), 7.20 (d, *J* = 7.2 Hz, 1H), 6.91 (d, *J* = 8.0 Hz, 1H), 3.93 (d, *J* = 11.6 Hz, 2H), 2.89 (brs, 4H), 2.65 (quin, *J* = 7.6 Hz, 1H), 2.47–2.41 (m, 5H), 2.04–1.99 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.4, 151.6, 143.4, 138.0, 136.0, 134.7, 133.3, 129.6, 129.4, 128.9, 127.74, 127.68, 127.0, 124.9, 119.3, 46.5, 43.2, 31.1, 28.0, 27.6, 21.5; ESI-HRMS: m/z Calcd for $C_{25}H_{27}N_2O_2S$, $[M+H]^+$: 419.1788, found 419.1785.

4-Ethyl-2-phenyl-5,6-dihydrobenzo[h]quinoline (4q)



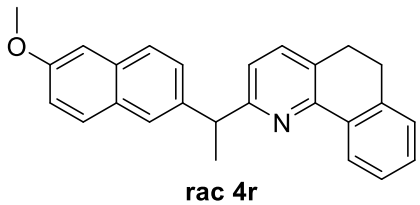
Colorless oil; (62 mg, 43%); R_f = 0.52 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.53 (dd, J = 1.2 Hz, J = 7.6 Hz, 1H), 8.16–8.13 (m, 2H), 7.49–7.46 (m, 3H), 7.41–7.35 (m, 2H), 7.30 (td, J = 1.2 Hz, J = 7.6 Hz, 1H), 7.23–7.21 (m, 1H), 2.93 (s, 4H), 2.73 (q, J = 7.6 Hz, 2H), 1.26 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.5, 151.7, 150.7, 139.9, 137.8, 135.2, 128.7, 128.5, 128.4, 127.3, 127.0, 126.7, 125.6, 118.9, 27.9, 26.0, 23.4, 14.0; ESI-HRMS: m/z Calcd for $C_{21}H_{20}N$, $[M+H]^+$: 286.1590, found 286.1591.

(S)-2-(1-(6-Methoxynaphthalen-2-yl)ethyl)-5,6-dihydrobenzo[h]quinoline (4r)



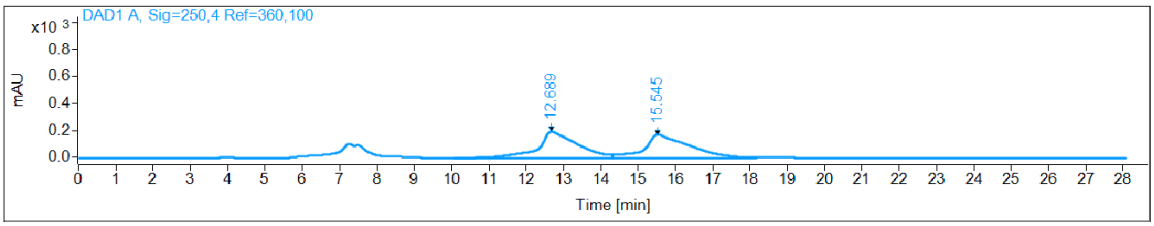
Green oil; (115 m*g,* 63%); 48% ee, $[\alpha]_D^{17}$ = +117, (c 1.0, EA); R_f = 0.30 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.46 (d, J = 7.6 Hz, 1H), 7.72 (s, 1H), 7.69–7.63 (m, 2H), 7.48 (d, J = 8.4 Hz, 1H), 7.37 (t, J = 7.6 Hz, 1H), 7.29–7.26 (m, 2H), 7.18 (d, J = 6.8 Hz, 1H), 7.11–7.07 (m, 2H), 6.89 (d, J = 7.6 Hz, 1H), 4.43 (q, J = 7.2 Hz, 1H), 3.85 (s, 3H), 2.88–2.86 (m, 2H), 2.84–2.80 (m, 2H), 1.83 (d, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.8, 157.3, 151.3, 140.8, 138.0, 135.8, 135.0, 133.2, 129.2, 129.0, 128.9, 128.8, 127.6, 127.2, 127.0, 126.8, 125.6, 125.1, 120.7, 118.6, 105.6, 55.2, 47.2, 28.1, 27.6, 21.0; ESI-HRMS: m/z Calcd for $C_{26}H_{24}NO$, $[M+H]^+$: 366.1852, found 366.1851. HPLC (CHIRALCEL® AD-H column, hexane/i-PrOH: 99.3/0.7, 1.0 mL min^{-1}, 250 nm), t_r (minor) = 15.6 min, t_r (major) = 12.7 min.

HPLC spectra for compound 4r



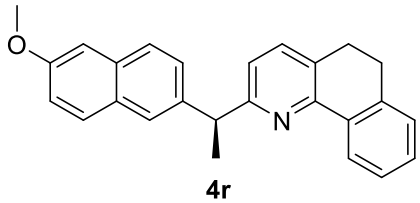
rac 4r

Sample name: 20190126-zjl-race-006



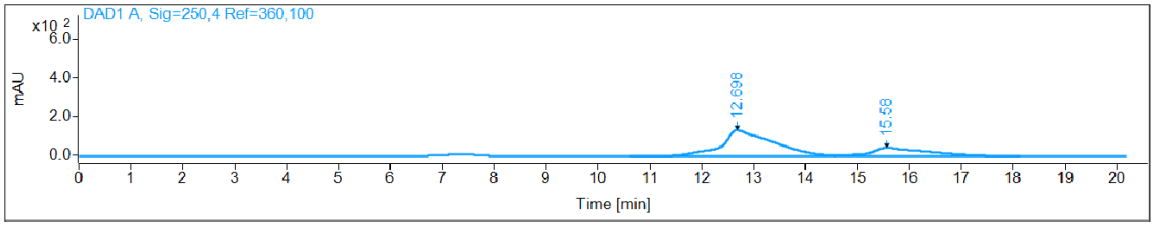
Signal: DAD1 A, Sig=250,4 Ref=360,100

RT [min]	Height	Symm.	Width (50%)	Area	Area%
12.689	197.75220	0.58	0.9667	14550.77734	49.39
15.545	175.65091	0.46	1.1867	14910.58105	50.61



4r

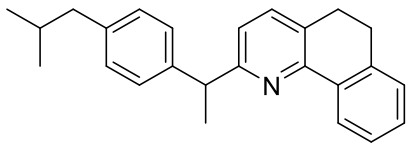
Sample name: 20190126-zjl-chirt-001



Signal: DAD1 A, Sig=250,4 Ref=360,100

RT [min]	Height	Symm.	Width (50%)	Area	Area%
12.698	137.37633	0.50	0.9867	9638.82617	73.65
15.580	44.47153	0.42	1.1467	3447.73730	26.35

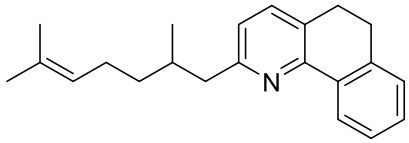
2-(1-(4-Isobutylphenyl)ethyl)-5,6-dihydrobenzo[h]quinoline (4s)



Yellow oil; (106 mg, 62%); R_f = 0.72 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.43 (d, J = 7.2 Hz, 1H), 7.36 (t, J = 7.2 Hz, 1H), 7.32–7.26 (m, 4H), 7.21–7.18 (m, 1H), 7.06 (d, J = 8.0 Hz, 2H), 6.89 (d, J = 7.6 Hz, 1H), 4.27 (q, J = 7.2 Hz, 1H), 2.93–2.88 (m, 2H), 2.86–2.82 (m, 2H), 2.42 (d, J = 7.2 Hz, 2H), 1.82 (sep, J = 6.8 Hz, 1H), 1.74 (d, J = 7.2 Hz, 3H), 0.88 (d, J = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.0, 151.3, 142.8, 139.4,

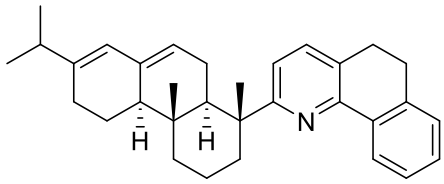
138.0, 135.8, 135.1, 129.0, 128.8, 128.7, 127.6, 127.5, 127.0, 125.1, 120.6, 46.9, 45.0, 30.2, 28.1, 27.6, 22.4, 21.1; ESI-HRMS: m/z Calcd for $C_{25}H_{28}N$, $[M+H]^+$: 342.2216, found 342.2219.

2-(2,6-Dimethylhept-5-en-1-yl)-5,6-dihydrobenzo[h]quinoline (4t)



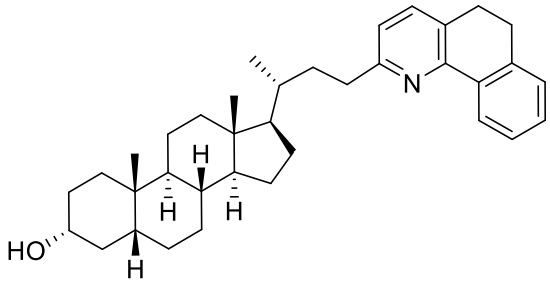
Colorless oil; (57 mg, 57%); R_f = 0.68 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.35 (dd, *J* = 1.2 Hz, *J* = 7.6 Hz, 1H), 7.36–7.32 (m, 2H), 7.27 (td, *J* = 1.2 Hz, *J* = 7.2 Hz, 1H), 7.20 (d, *J* = 7.2 Hz, 1H), 6.92 (d, *J* = 7.6 Hz, 1H), 5.12 (tt, *J* = 1.2 Hz, *J* = 7.2 Hz, 1H), 2.92–2.81 (m, 5H), 2.63–2.58 (m, 1H), 2.13–1.98 (m, 3H), 1.68 (s, 3H), 1.61 (s, 3H), 1.49–1.40 (m, 1H), 1.29–1.20 (m, 1H), 0.93 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.4, 151.6, 138.0, 135.4, 135.0, 131.0, 128.7, 128.6, 127.6, 127.0, 125.0, 124.9, 121.9, 45.6, 36.9, 33.3, 28.2, 27.7, 25.7, 25.6, 19.5, 17.7; ESI-HRMS: m/z Calcd for $C_{22}H_{28}N$, $[M+H]^+$: 306.2216, found 306.2218.

2-((1R,4aR,4bR,10aR)-7-Isopropyl-1,4a-dimethyl-1,2,3,4,4a,4b,5,6,10,10a-decahydrophenanthren-1-yl)-5,6-dihydrobenzo[h]quinoline (4u)



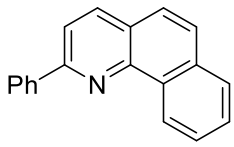
Colorless oil; (88 mg, 40%); R_f = 0.54 (hexanes/ethyl acetate 40:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.38 (dd, *J* = 1.2 Hz, *J* = 8.0 Hz, 1H), 7.36–7.32 (m, 2H), 7.26 (td, *J* = 1.6 Hz, *J* = 7.6 Hz, 1H), 7.19–7.14 (m, 2H), 5.70 (s, 1H), 5.23 (t, *J* = 2.4 Hz, 1H), 2.93–2.86 (m, 4H), 2.34–2.18 (m, 3H), 2.09 (d, *J* = 5.6 Hz, 2H), 2.01–1.96 (m, 3H), 1.89–1.85 (m, 1H), 1.80–1.75 (m, 1H), 1.67–1.62 (m, 1H), 1.51 (s, 3H), 1.46–1.40 (m, 1H), 1.37–1.32 (m, 1H), 1.30–1.21 (m, 2H), 0.99 (dd, *J* = 2.8 Hz, *J* = 6.8 Hz, 6H), 0.94 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.9, 150.5, 144.8, 138.0, 135.3, 135.1, 128.6, 128.0, 127.6, 126.9, 125.1, 122.6, 121.6, 119.0, 51.3, 48.4, 44.0, 40.1, 39.0, 35.1, 34.8, 28.2, 27.54, 27.51, 24.6, 22.6, 21.4, 20.8, 19.1, 18.7, 14.2; ESI-HRMS: m/z Calcd for $C_{32}H_{40}N$, $[M+H]^+$: 438.3155, found 438.3156.

(3R,5R,8R,9S,10S,13R,14S,17R)-17-((R)-4-(5,6-Dihydrobenzo[h]quinolin-2-yl)butan-2-yl)-10,13-dimethylhexadecahydro-1H-cyclopenta[a]phenanthren-3-ol (4v)



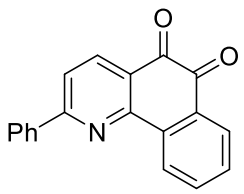
White solid; (223 mg, 87%); mp: 167–169 °C; R_f = 0.43 (hexanes/ethyl acetate 3:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.35 (d, J = 6.8 Hz, 1H), 7.37–7.32 (m, 2H), 7.27 (td, J = 1.2 Hz, J = 7.2 Hz, 1H), 7.20 (d, J = 7.2 Hz, 1H), 6.95 (d, J = 7.6 Hz, 1H), 3.63–3.57 (m, 1H), 2.93–2.86 (m, 5H), 2.73–2.66 (m, 1H), 1.99 (d, J = 11.6 Hz, 1H), 1.93–1.82 (m, 3H), 1.80–1.73 (m, 3H), 1.66–1.63 (m, 1H), 1.57–1.48 (m, 4H), 1.38–1.16 (m, 11H), 1.09–1.03 (m, 6H), 0.99–0.94 (m, 1H), 0.91 (s, 3H), 0.64 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.9, 151.5, 138.0, 135.7, 134.9, 128.6, 128.5, 127.6, 127.0, 125.0, 120.9, 71.8, 56.5, 56.2, 42.7, 42.1, 40.4, 40.2, 36.4, 36.1, 35.8, 35.7, 35.3, 34.9, 34.5, 30.5, 28.24, 28.19, 27.7, 27.7, 26.4, 24.2, 23.3, 20.8, 18.6, 12.0; ESI-HRMS: m/z Calcd for $C_{36}H_{50}NO$, $[M+H]^+$: 512.3887, found 512.3890.

2-Phenylbenzo[h]quinoline (5)



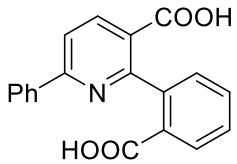
White solid; (54 mg, 70%); mp: 76–78 °C; R_f = 0.40 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.49 (d, J = 8.0 Hz, 1H), 8.32 (d, J = 7.6 Hz, 2H), 8.15 (d, J = 8.4 Hz, 1H), 7.95 (d, J = 8.4 Hz, 1H), 7.87 (d, J = 7.6 Hz, 1H), 7.73 (t, J = 8.0 Hz, 2H), 7.69–7.62 (m, 2H), 7.54 (t, J = 7.6 Hz, 2H), 7.46 (t, J = 7.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.4, 146.2, 139.7, 136.4, 133.8, 131.8, 129.2, 128.8, 128.1, 127.7, 127.42, 127.40, 126.8, 125.1, 125.0, 124.7, 118.8; MS (ESI, m/z): Calcd for $C_{19}H_{14}N$, $[M+H]^+$: 256.1, found 255.9.

2-Phenylbenzo[h]quinoline-5,6-dione (6)



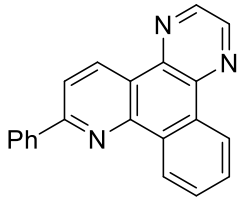
Yellow solid; (45 mg, 53%); mp: 205–208 °C; R_f = 0.23 (hexanes/ethyl acetate 5:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.80 (d, J = 8.0 Hz, 1H), 8.39 (d, J = 8.4 Hz, 1H), 8.19–8.16 (m, 3H), 7.81–7.78 (m, 2H), 7.59–7.53 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.3, 161.9, 153.3, 137.9, 137.5, 136.5, 136.1, 131.6, 131.1, 130.9, 129.7, 129.0, 127.6, 126.4, 125.1, 120.3; MS (ESI, m/z): Calcd for $C_{19}H_{12}NO_2$, $[M+H]^+$: 286.1, found 286.0.

2-(2-Carboxyphenyl)-6-phenylnicotinic acid (7)



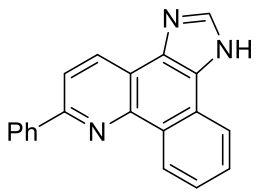
White solid; (94 mg, 98%); mp: 258–261 °C; R_f = 0.21 (DCM/MeOH 3:1); ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.02–7.97 (m, 4H), 7.58–7.56 (m, 1H), 7.48–7.40 (m, 5H), 7.07–7.05 (m, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 171.2, 170.4, 156.6, 154.6, 139.2, 138.0, 136.8, 136.5, 132.3, 129.2, 128.7, 128.6, 127.6, 127.4, 126.8, 118.7; ESI-HRMS: m/z Calcd for $C_{19}H_{14}NO_4$, $[M+H]^+$: 320.0917, found 320.0919.

7-Phenylbenzo[f]pyrido[2,3-h]quinoxaline (8)



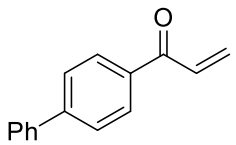
White solid; (76 mg, 83%); mp: 165–168 °C; R_f = 0.33 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.36 (d, J = 7.6 Hz, 1H), 9.29 (d, J = 8.4 Hz, 1H), 9.08 (d, J = 7.6 Hz, 1H), 8.83 (d, J = 11.2 Hz, 2H), 8.32 (d, J = 7.6 Hz, 2H), 8.04 (d, J = 8.4 Hz, 1H), 7.84–7.77 (m, 2H), 7.58–7.48 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.3, 147.1, 143.5, 143.4, 141.2, 140.3, 138.9, 133.5, 132.4, 131.0, 129.5, 129.4, 128.9, 128.7, 127.4, 124.8, 124.4, 123.1, 119.1; MS (ESI, m/z): Calcd for $C_{21}H_{14}N_3$, $[M+H]^+$: 308.1, found 308.2.

6-Phenyl-3H-benzo[h]imidazo[4,5-f]quinoline (9)



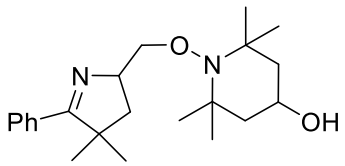
Yellow solid; (66 mg, 75%); mp: 271–276 °C; R_f = 0.27 (ethyl acetate); ^{1}H NMR (400 MHz, DMSO-d_6): δ 13.60 (brs, 1H), 9.43 (d, J = 8.0 Hz, 1H), 8.86 (d, J = 6.8 Hz, 1H), 8.43–8.41 (m, 4H), 8.34 (d, J = 8.0 Hz, 1H), 7.85–7.81 (m, 1H), 7.73 (t, J = 7.2 Hz, 1H), 7.59 (t, J = 7.6 Hz, 1H), 7.50 (t, J = 7.2 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 152.6, 143.3, 140.0, 139.0, 130.5, 129.1, 128.9, 128.6, 128.5, 126.8, 125.3, 125.2, 121.3, 119.0; MS (ESI, m/z): Calcd for $C_{20}H_{14}N_3$, $[M+H]^+$: 296.1, found 296.1.

1-([1,1'-Biphenyl]-4-yl)prop-2-en-1-one (10)



White solid; (20 mg, 32%); mp: 77–79 °C; R_f = 0.17 (hexanes/ethyl acetate 20:1); ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.02 (d, J = 8.4 Hz, 2H), 7.69 (d, J = 8.1 Hz, 2H), 7.62 (d, J = 7.2 Hz, 2H), 7.48–7.36 (m, 3H), 7.20 (dd, J = 10.5 Hz, J = 17.1 Hz, 1H), 6.46 (dd, J = 1.2 Hz, J = 17.1 Hz, 1H), 5.93 (dd, J = 1.2 Hz, J = 10.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 190.4, 145.7, 139.8, 135.9, 132.3, 130.0, 129.3, 128.9, 128.2, 127.2; ESI-HRMS: m/z Calcd for $C_{15}H_{13}O$, $[M+H]^+$: 209.0961, found 209.0965.

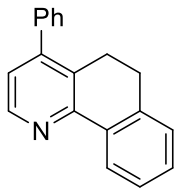
1-((4,4-Dimethyl-5-phenyl-3,4-dihydro-2H-pyrrol-2-yl)methoxy)-2,2,6,6-tetramethylpiperidin-4-ol (14)



Corlorless oil; (71 mg, 66%); R_f = 0.22 (hexanes/ethyl acetate 2:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70–7.68 (m, 2H), 7.39–7.34 (m, 3H), 4.24–4.18 (m, 1H), 4.11 (dd, J = 4.8 Hz, J = 8.8 Hz, 1H), 3.98–3.89 (m, 2H), 2.06 (dd, J = 7.2 Hz, J = 12.4 Hz, 1H), 1.87 (dd, J = 8.4 Hz, J = 12.4 Hz, 1H), 1.78 (dd, J = 3.2 Hz, J = 12.4 Hz, 2H), 1.46 (t, J = 12.0 Hz, 2H), 1.39 (s, 3H), 1.36 (s, 3H), 1.26 (s, 6H), 1.17 (s, 3H), 1.14 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 180.1, 134.8, 129.3, 128.0, 127.8, 79.3, 67.3, 63.1, 60.2, 50.4, 48.2, 44.7, 33.20, 33.16, 27.1,

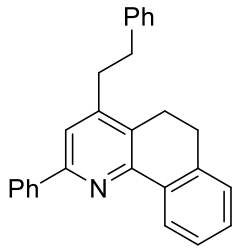
26.5, 21.2, 21.0; MS (ESI, m/z): Calcd for $C_{22}H_{34}N_2O_2$, $[M+H]^+$: 359.3, found 359.5.

4-Phenyl-5,6-dihydrobenzo[h]quinoline (16a)



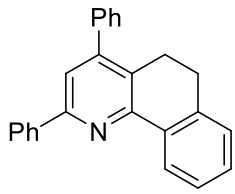
Yellow solid; (56 mg, 73%); mp: 99–103 °C; R_f = 0.26 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.57 (d, J = 4.8 Hz, 1H), 8.36–8.34 (m, 1H), 7.49–7.30 (m, 7H), 7.21 (d, J = 7.2 Hz, 1H), 7.12 (d, J = 5.2 Hz, 1H), 2.92–2.88 (m, 2H), 2.83–2.79 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 152.9, 148.4, 147.2, 138.8, 138.0, 134.9, 129.4, 129.0, 128.7, 128.4, 128.0, 127.4, 127.1, 125.3, 123.3, 28.0, 25.4; ESI-HRMS: m/z Calcd for $C_{19}H_{16}N$, $[M+H]^+$: 258.1277, found 258.1274.

4-Phenethyl-2-phenyl-5,6-dihydrobenzo[h]quinoline (16b)



Yellow solid; (50 mg, 46%); mp: 125–128 °C; R_f = 0.42 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.53 (d, J = 7.6 Hz, 1H), 8.10 (d, J = 7.6 Hz, 2H), 7.48 (t, J = 7.6 Hz, 2H), 7.42–7.36 (m, 3H), 7.32–7.27 (m, 3H), 7.22–7.16 (m, 4H), 3.03–2.99 (m, 2H), 2.94–2.91 (m, 2H), 2.86 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.3, 151.8, 148.2, 140.8, 139.6, 137.9, 135.1, 128.8, 128.54, 128.50, 128.46, 127.4, 127.0, 126.7, 125.6, 119.8, 36.3, 34.8, 27.8, 23.5; ESI-HRMS: m/z Calcd for $C_{27}H_{24}N$, $[M+H]^+$: 362.1903, found 362.1900.

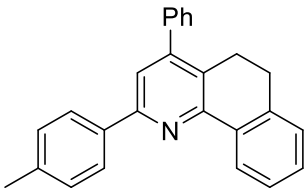
2,4-Diphenyl-5,6-dihydrobenzo[h]quinoline (16c)



White solid; (60 mg, 60%); mp: 122–126 °C; R_f = 0.37 (hexanes/ethyl acetate 20:1); ^{1}H

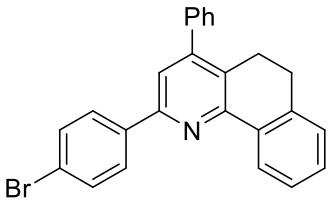
NMR (400 MHz, $CDCl_3$): δ 8.58 (d, J = 7.6 Hz, 1H), 8.17 (d, J = 7.6 Hz, 2H), 7.59 (s, 1H), 7.50–7.46 (m, 4H), 7.44–7.38 (m, 5H), 7.32 (t, J = 7.2 Hz, 1H), 7.21 (d, J = 6.0 Hz, 1H), 2.92 (dd, J = 5.6 Hz, J = 8.8 Hz, 2H), 2.84 (dd, J = 5.6 Hz, J = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.3, 152.5, 149.2, 139.5, 139.2, 138.1, 135.1, 129.0, 128.8, 128.7, 128.6, 128.4, 128.0, 127.8, 127.4, 127.0, 126.7, 125.7, 119.9, 28.1, 25.2; ESI-HRMS: m/z Calcd for $C_{25}H_{20}N$, $[M+H]^+$: 334.1590, found 334.1589.

4-Phenyl-2-(p-tolyl)-5,6-dihydrobenzo[h]quinoline (16d)



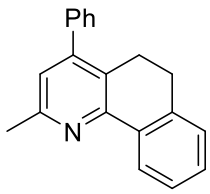
Light yellow solid; (47 mg, 45%); mp: 163–167 °C; R_f = 0.70 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.57 (dd, J = 1.2 Hz, J = 7.6 Hz, 1H), 8.07 (d, J = 8.0 Hz, 2H), 7.56 (s, 1H), 7.50–7.46 (m, 2H), 7.44–7.39 (m, 4H), 7.34–7.27 (m, 3H), 7.21 (d, J = 6.8 Hz, 1H), 2.93–2.90 (m, 2H), 2.85–2.82 (m, 2H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.3, 152.4, 149.1, 139.3, 138.6, 138.1, 136.7, 135.2, 129.3, 128.9, 128.8, 128.4, 127.9, 127.5, 127.4, 127.0, 126.6, 125.6, 119.6, 28.1, 25.2, 21.3; ESI-HRMS: m/z Calcd for $C_{26}H_{22}N$, $[M+H]^+$: 348.1747, found 348.1748.

2-(4-Bromophenyl)-4-phenyl-5,6-dihydrobenzo[h]quinoline (16e)



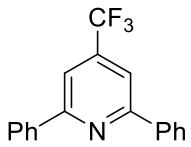
White solid; (85 mg, 69%); mp: 141–145 °C; R_f = 0.40 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.53 (d, J = 7.6 Hz, 1H), 8.03 (d, J = 8.4 Hz, 2H), 7.57 (d, J = 8.4 Hz, 2H), 7.53 (s, 1H), 7.48–7.30 (m, 7H), 7.21 (s, 1H), 2.90–2.88 (m, 2H), 2.83–2.82 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.1, 152.6, 149.3, 139.0, 138.3, 138.1, 134.9, 131.7, 129.1, 128.7, 128.4, 128.3, 128.2, 128.0, 127.5, 127.0, 125.6, 123.0, 119.5, 28.0, 25.2; ESI-HRMS: m/z Calcd for $C_{25}H_{19}BrN$, $[M+H]^+$: 412.0695, found 412.0690.

2-Methyl-4-phenyl-5,6-dihydrobenzo[h]quinoline (16f)



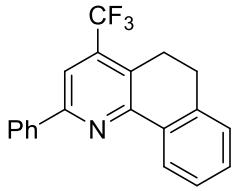
Yellow oil; (20 mg, 25%); R_f = 0.50 (hexanes/ethyl acetate 10:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.37 (d, J = 7.2 Hz, 1H), 7.48–7.40 (m, 3H), 7.39–7.34 (m, 3H), 7.30 (td, J = 1.2 Hz, J = 7.2 Hz, 1H), 7.21 (d, J = 7.2 Hz, 1H), 7.01 (s, 1H), 2.88–2.85 (m, 2H), 2.82–2.78 (m, 2H), 2.63 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.7, 152.3, 148.7, 139.2, 138.1, 135.1, 128.8, 128.7, 128.3, 127.8, 127.4, 127.1, 126.3, 125.4, 122.8, 28.2, 25.1, 24.4; ESI-HRMS: m/z Calcd for $C_{20}H_{18}N$, $[M+H]^+$: 272.1434, found 272.1435.

2,6-Diphenyl-4-(trifluoromethyl)pyridine (16g)

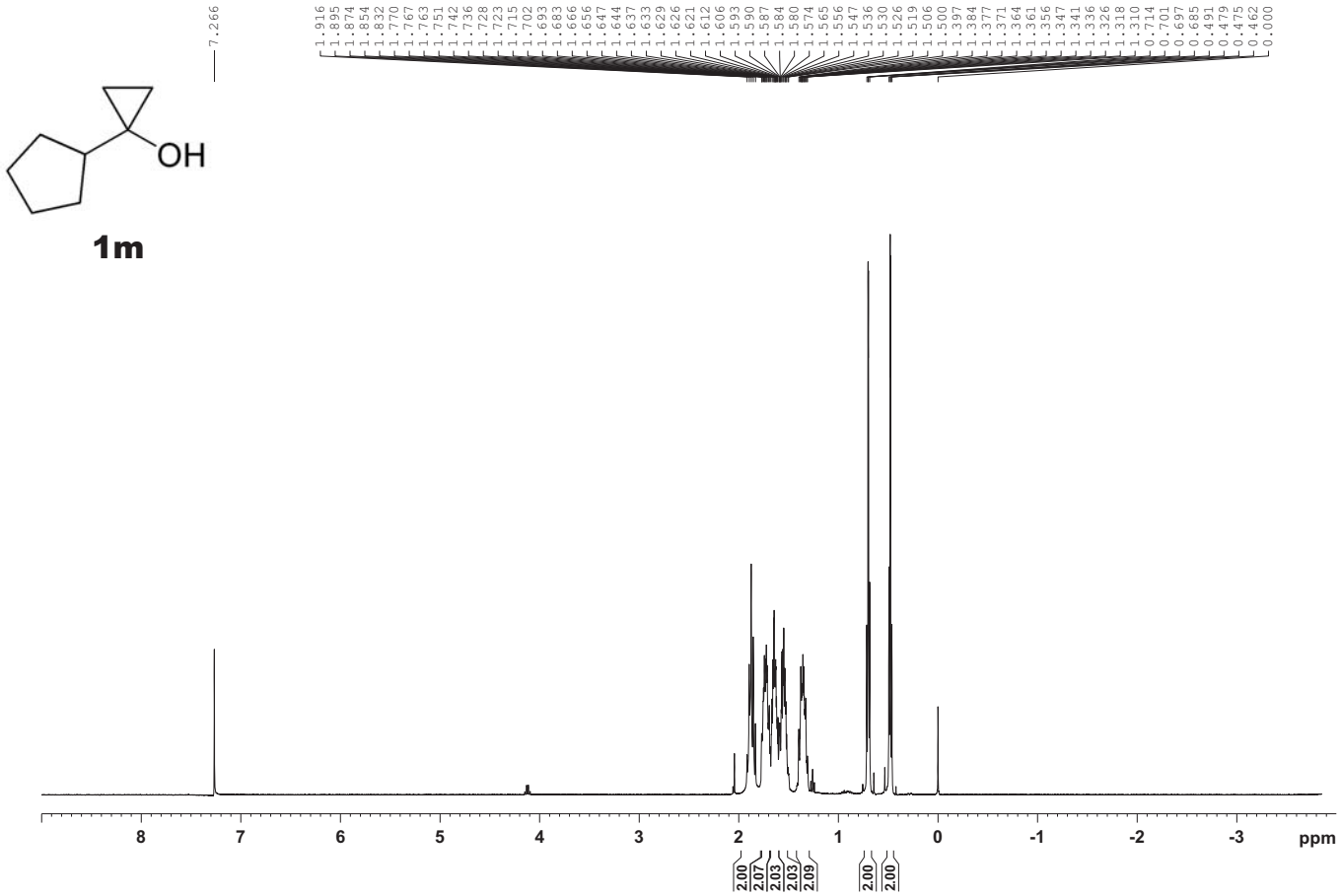


White solid; (81 mg, 90%); mp: 85–88 °C; R_f = 0.57 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.15 (d, J = 7.2 Hz, 4H), 7.85 (s, 2H), 7.53–7.44 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.1, 139.9 (q, $^{C,F}J_2$ = 33 Hz), 138.1, 129.8, 128.9, 127.1, 123.2 (q, $^{C,F}J_1$ = 272 Hz), 114.0 (q, $^{C,F}J_3$ = 3 Hz); ESI-HRMS: m/z Calcd for $C_{18}H_{13}F_3N$, $[M+H]^+$: 300.0995, found 300.0999.

2-Phenyl-4-(trifluoromethyl)-5,6-dihydrobenzo[h]quinoline (16h)



White solid; (91 mg, 93%); mp: 141–145 °C; R_f = 0.57 (hexanes/ethyl acetate 20:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.52–8.50 (m, 1H), 8.16 (d, J = 7.2 Hz, 2H), 7.85 (s, 1H), 7.52 (t, J = 7.6 Hz, 2H), 7.47–7.35 (m, 3H), 7.26 (d, J = 7.2 Hz, 1H), 3.14 (t, J = 7.6 Hz, 2H), 2.98 (t, J = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.4, 154.0, 138.4, 138.0, 136.6 (q, $^{C,F}J_2$ = 30 Hz), 133.9, 129.8, 129.4, 128.8, 127.6, 127.2, 126.8, 125.8, 123.5 (q, $^{C,F}J_1$ = 273 Hz), 114.5 (q, $^{C,F}J_3$ = 5 Hz), 27.2, 24.0; ESI-HRMS: m/z Calcd for $C_{20}H_{15}F_3N$, $[M+H]^+$: 326.1151, found 326.1153.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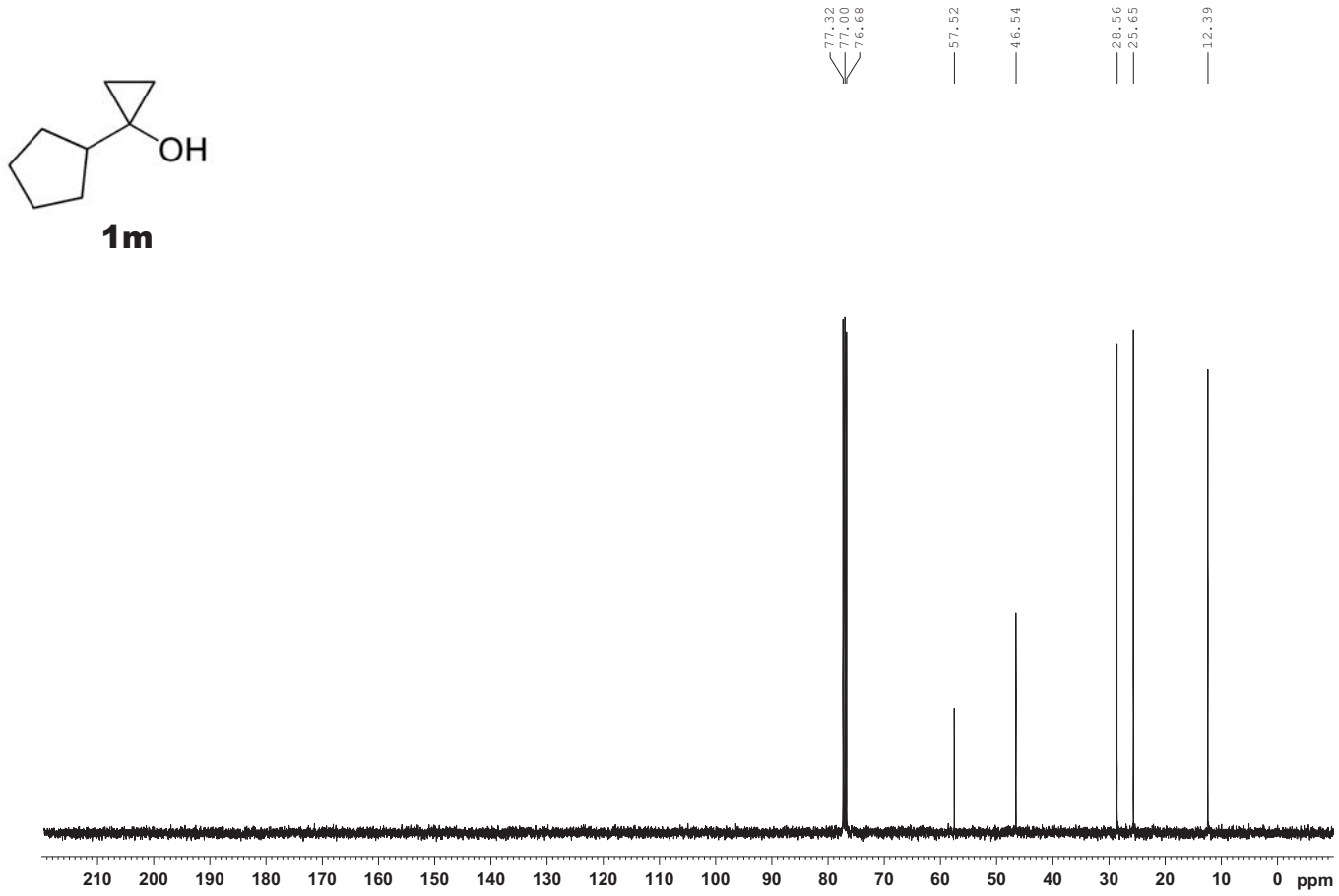
1.895
1.874
1.854
1.832
1.770
1.767
1.763
1.751
1.742
1.736
1.728
1.723
1.715
1.702
1.693
1.683
1.666
1.656
1.647
1.644
1.637
1.633
1.629
1.626
1.621
1.612
1.606
1.593
1.590
1.587
1.584
1.580
1.574
1.565
1.556
1.547
1.536
1.530
1.526
1.519
1.506
1.500
1.397
1.384
1.377
1.371
1.364
1.361
1.356
1.347
1.341
1.336
1.326
1.318
1.310
0.714
0.701
0.697
0.685
0.491
0.479
0.475
0.462
0.000
OH
1m
8
7
6
5
4
3
2
1
0
-1
-2
-3
ppm
2.00
2.07
2.03
2.03
2.09
2.00
2.00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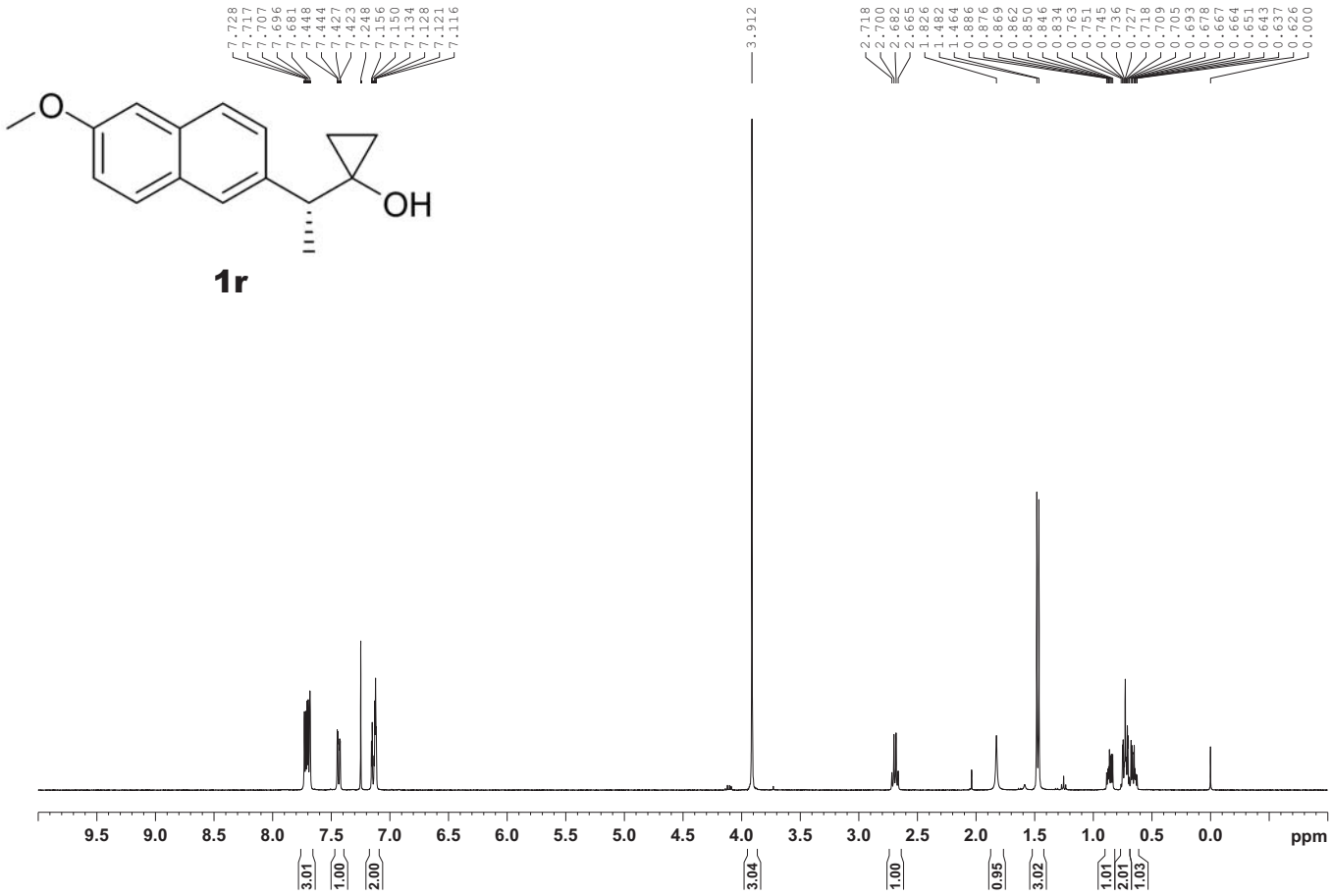
77.00
76.68
57.52
46.54
28.56
25.65
12.39
OH
1m
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.728
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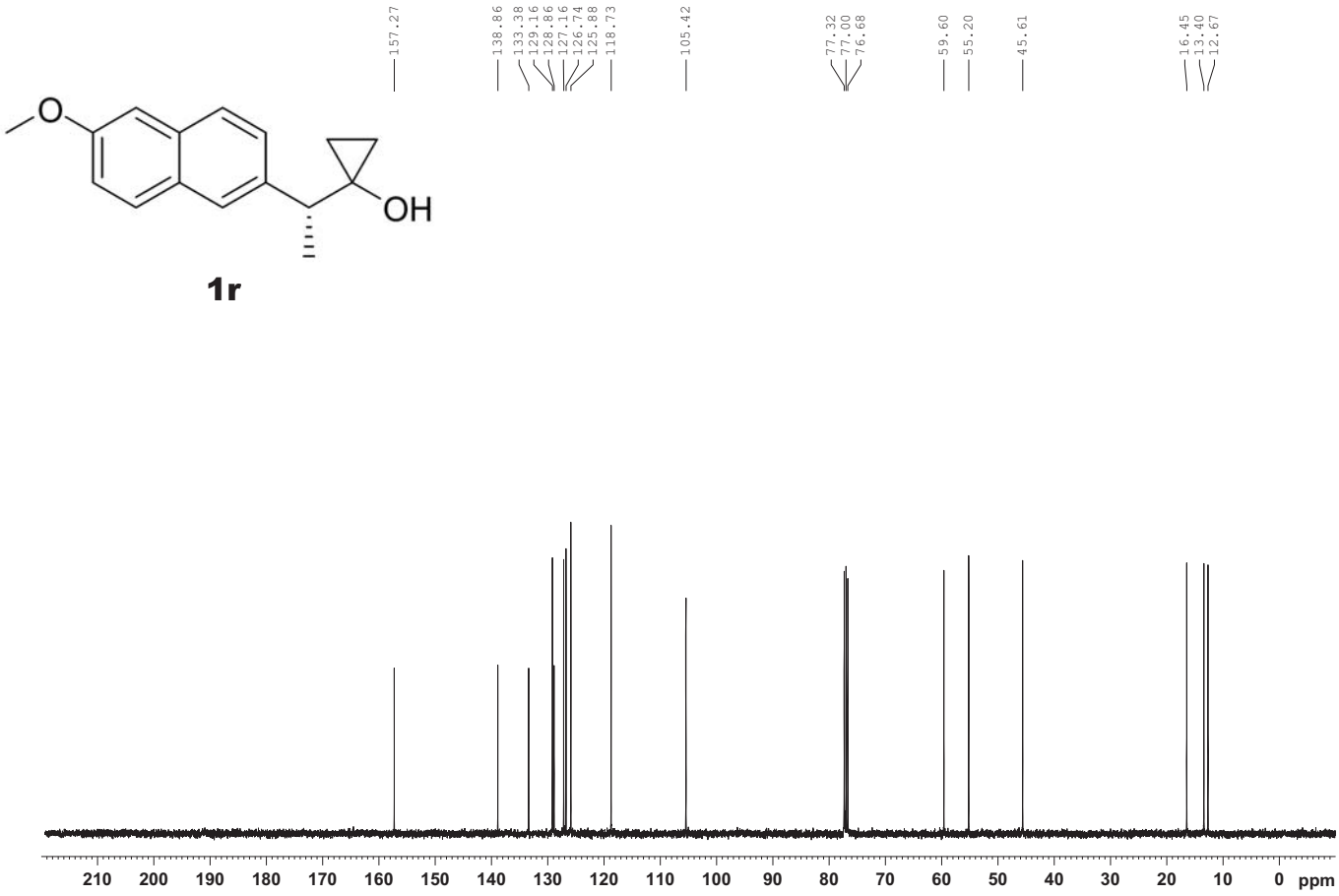
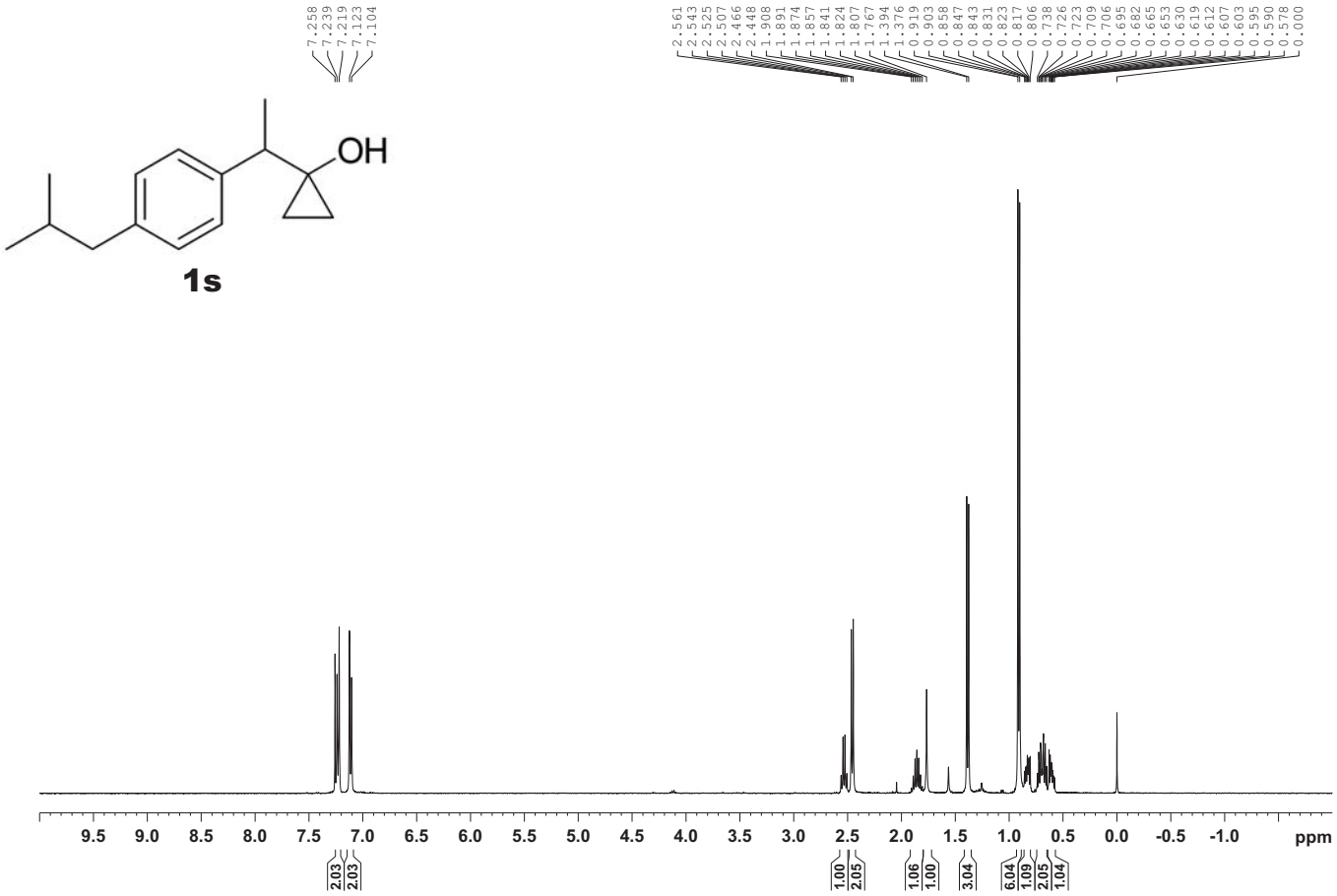
7.707
7.696
7.681
7.448
7.444
7.427
7.423
7.248
7.156
7.150
7.134
7.128
7.121
7.116
3.912
2.718
2.700
2.682
2.665
1.826
1.482
1.464
0.886
0.876
0.869
0.862
0.850
0.846
0.834
0.763
0.751
0.745
0.736
0.727
0.718
0.709
0.705
0.693
0.678
0.667
0.664
0.651
0.643
0.637
0.626
0.000
O
OH
1r
9.5 9.0 8.5 8.0 7.5 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 ppm
3.01
1.00
2.00
3.04
1.00
0.95
3.02
1.01
2.01
1.03


157.27
138.86
133.38
129.16
128.86
127.16
126.74
125.88
118.73
105.42
77.32
77.00
76.68
59.60
55.20
45.61
16.45
13.40
12.67
O
OH
1r
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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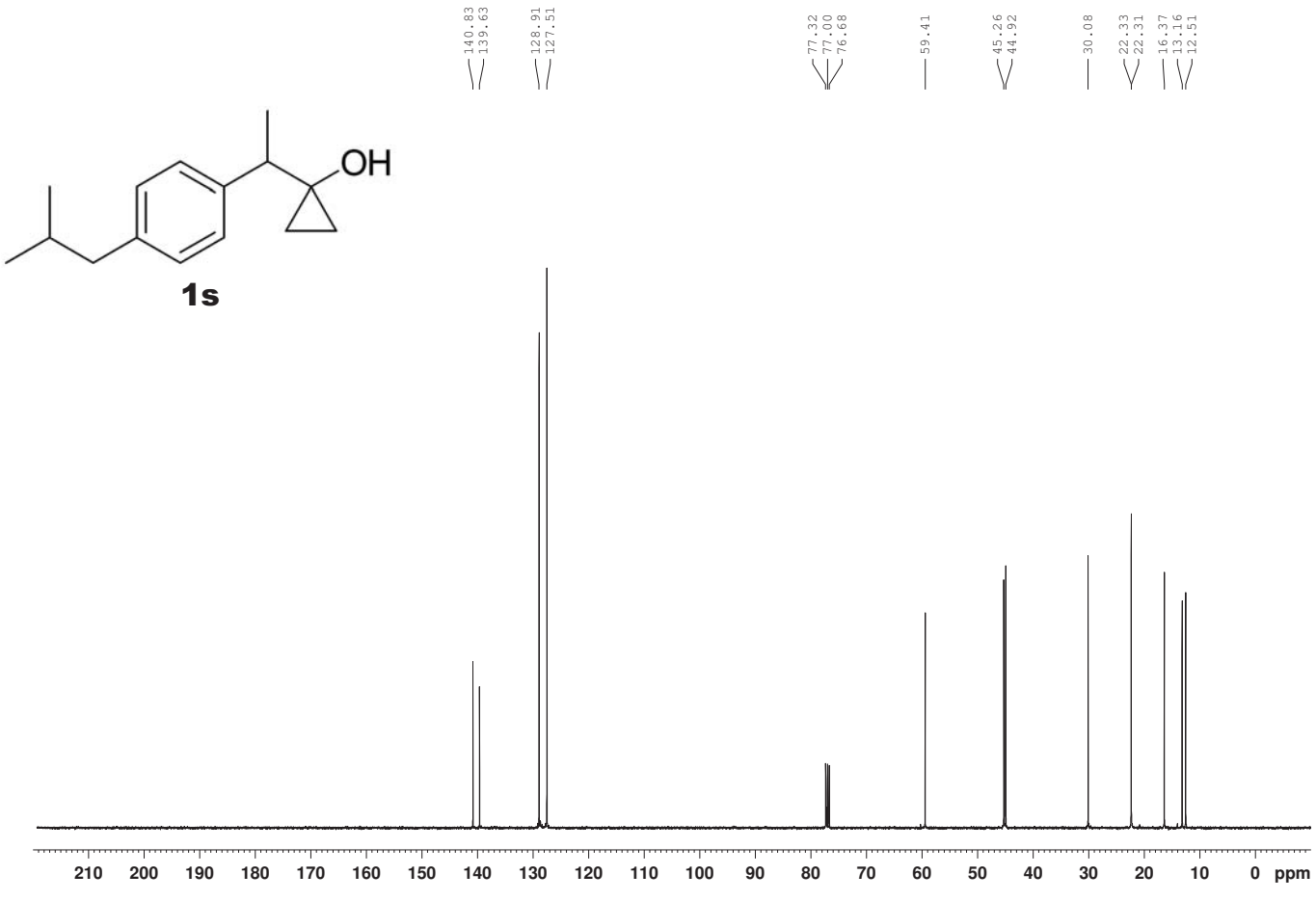
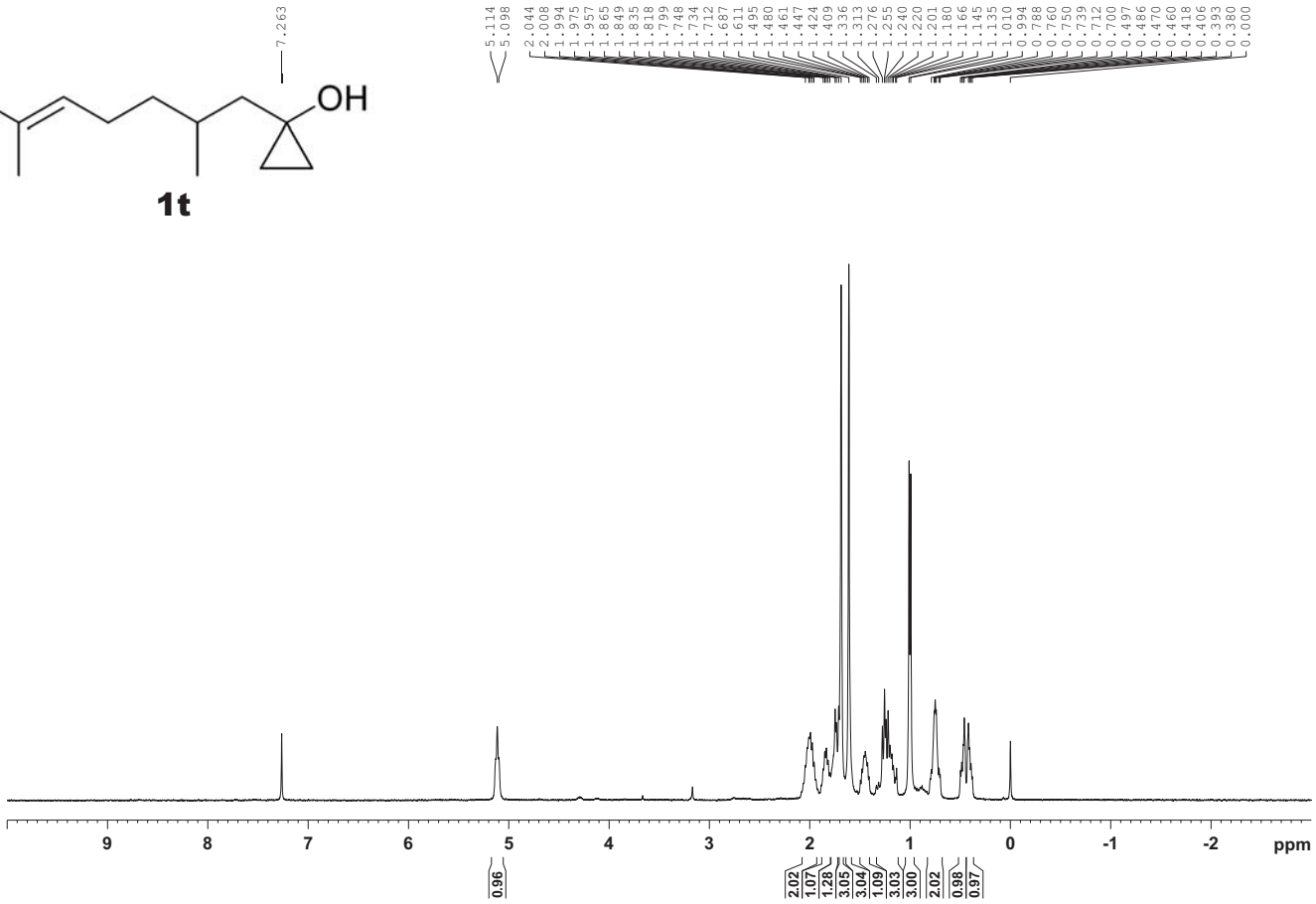
7.239
7.219
7.123
7.104
2.561
2.543
2.525
2.507
2.466
2.448
1.908
1.891
1.874
1.857
1.841
1.824
1.807
1.767
1.394
1.376
0.919
0.903
0.858
0.847
0.843
0.831
0.823
0.817
0.806
0.738
0.726
0.723
0.709
0.706
0.695
0.682
0.665
0.653
0.630
0.619
0.612
0.607
0.603
0.595
0.590
0.578
0.000
OH
1s
9.5 9.0 8.5 8.0 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
2.03
2.03
1.00
2.05
1.06
1.00
3.04
6.04
1.09
2.05
1.04


140.83
139.63
128.91
127.51
77.32
77.00
76.68
59.41
45.26
44.92
30.08
22.33
22.31
16.37
13.16
12.51
OH
1s
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

OH

1t


7.263
5.114
5.098
2.044
2.008
1.994
1.975
1.957
1.865
1.849
1.835
1.818
1.799
1.748
1.734
1.712
1.687
1.611
1.495
1.480
1.461
1.447
1.424
1.409
1.336
1.313
1.276
1.255
1.240
1.220
1.201
1.180
1.166
1.145
1.135
1.010
0.994
0.788
0.760
0.750
0.739
0.712
0.700
0.497
0.486
0.470
0.460
0.418
0.406
0.393
0.380
0.000
9
8
7
6
5
4
3
2
1
0
-1
-2
ppm
0.96
2.02
1.07
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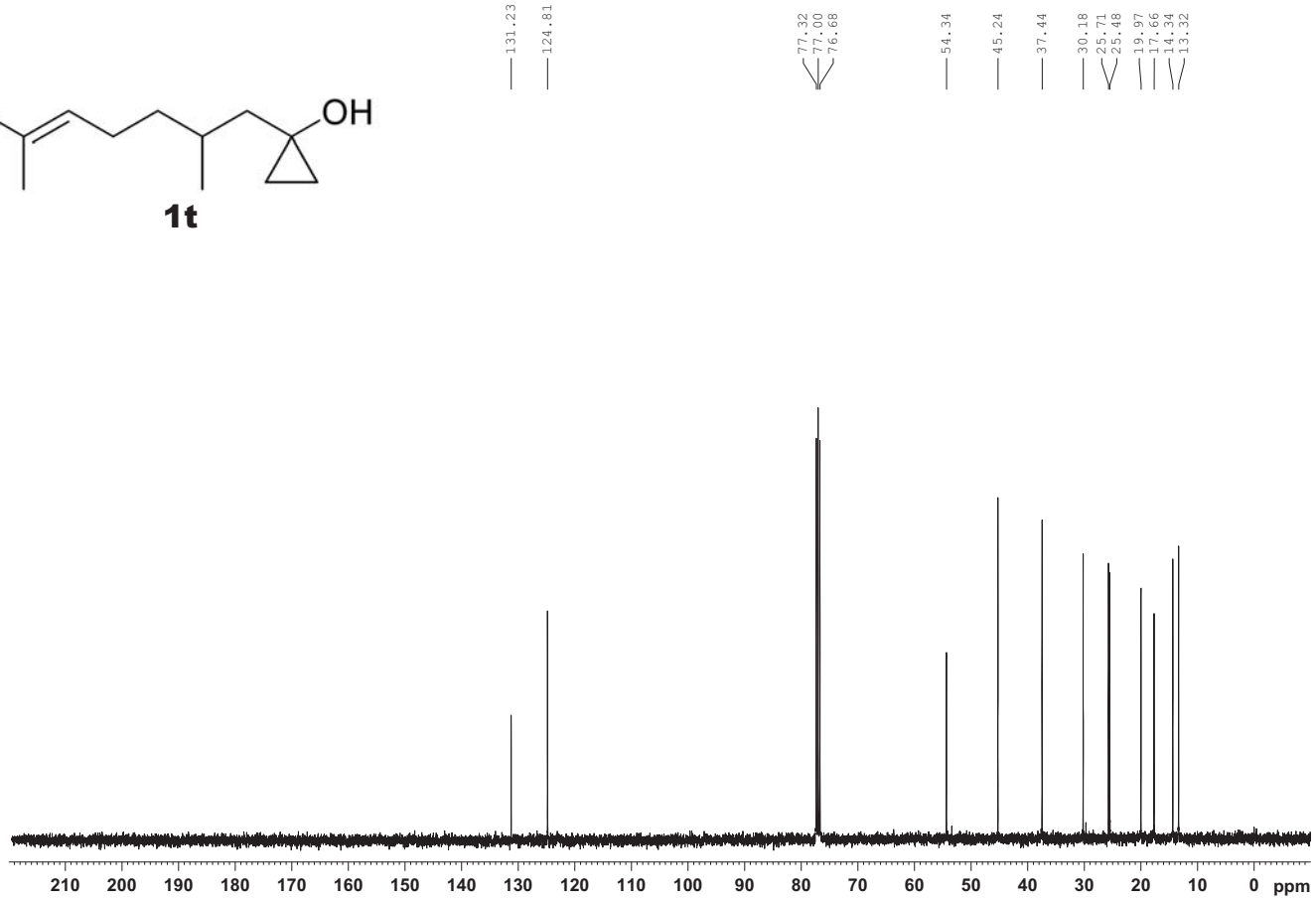
3.05
3.04
1.09
3.03
3.00
2.02
0.98
0.97

OH

1t


131.23
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54.34
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37.44
30.18
25.71
25.48
19.97
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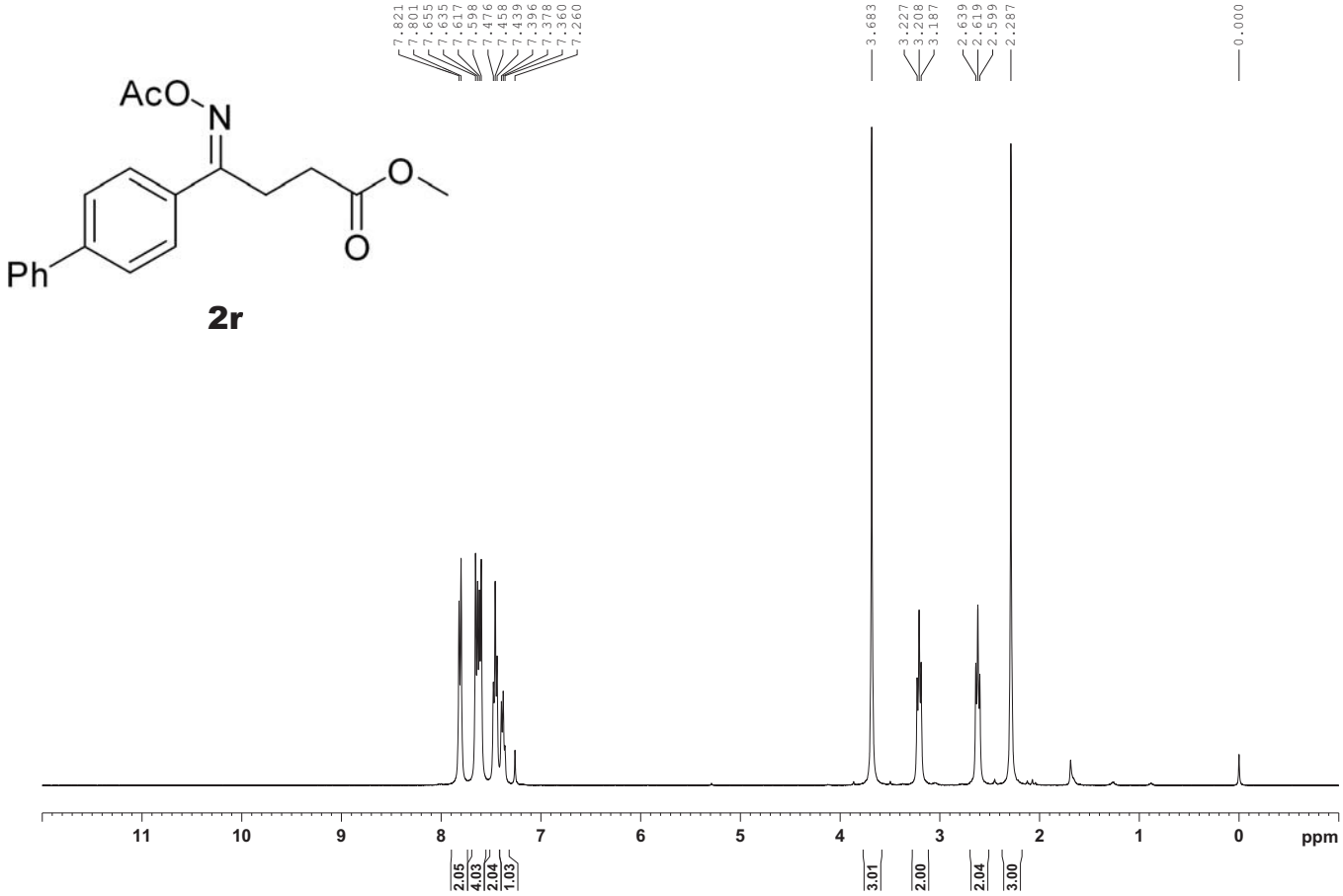
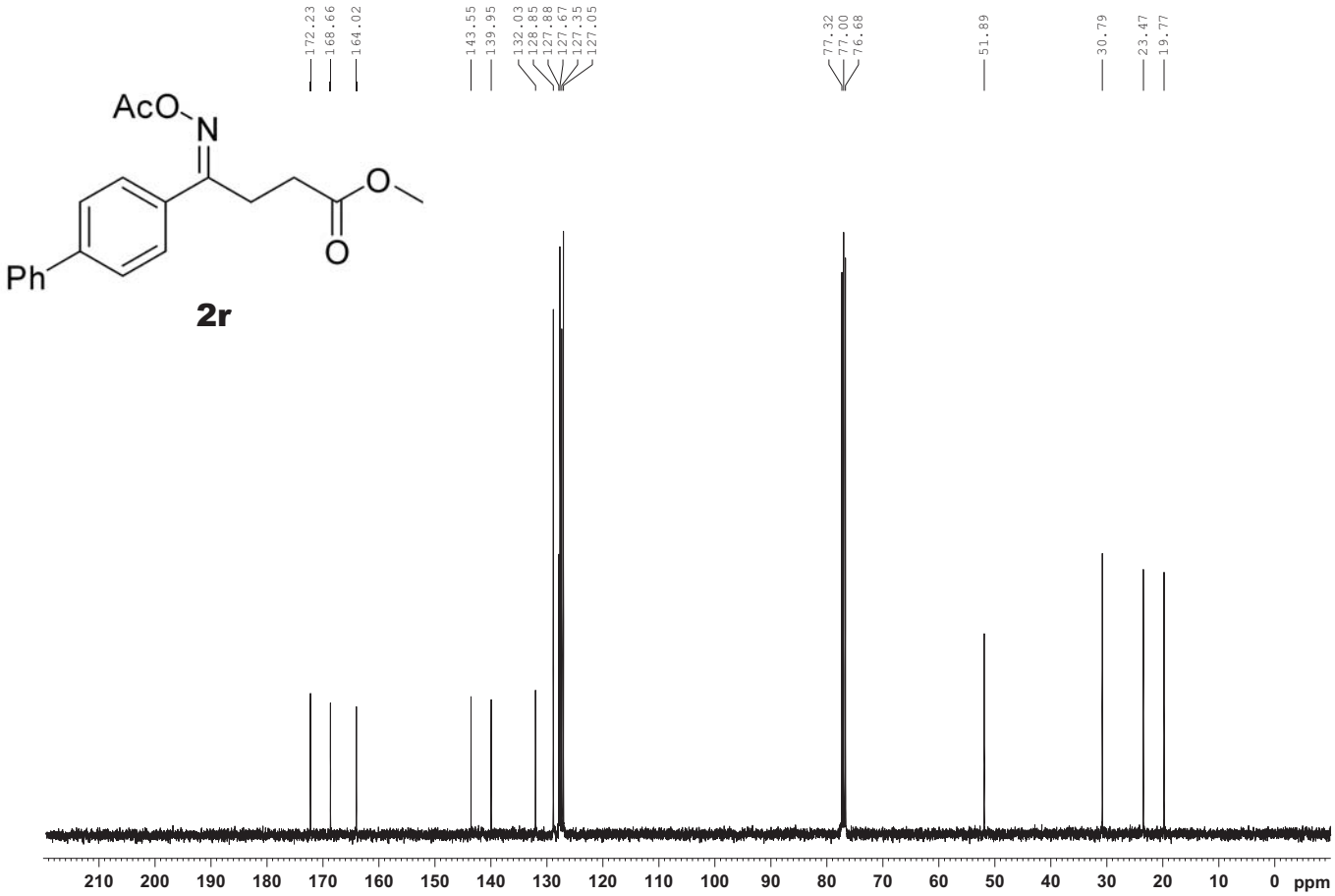
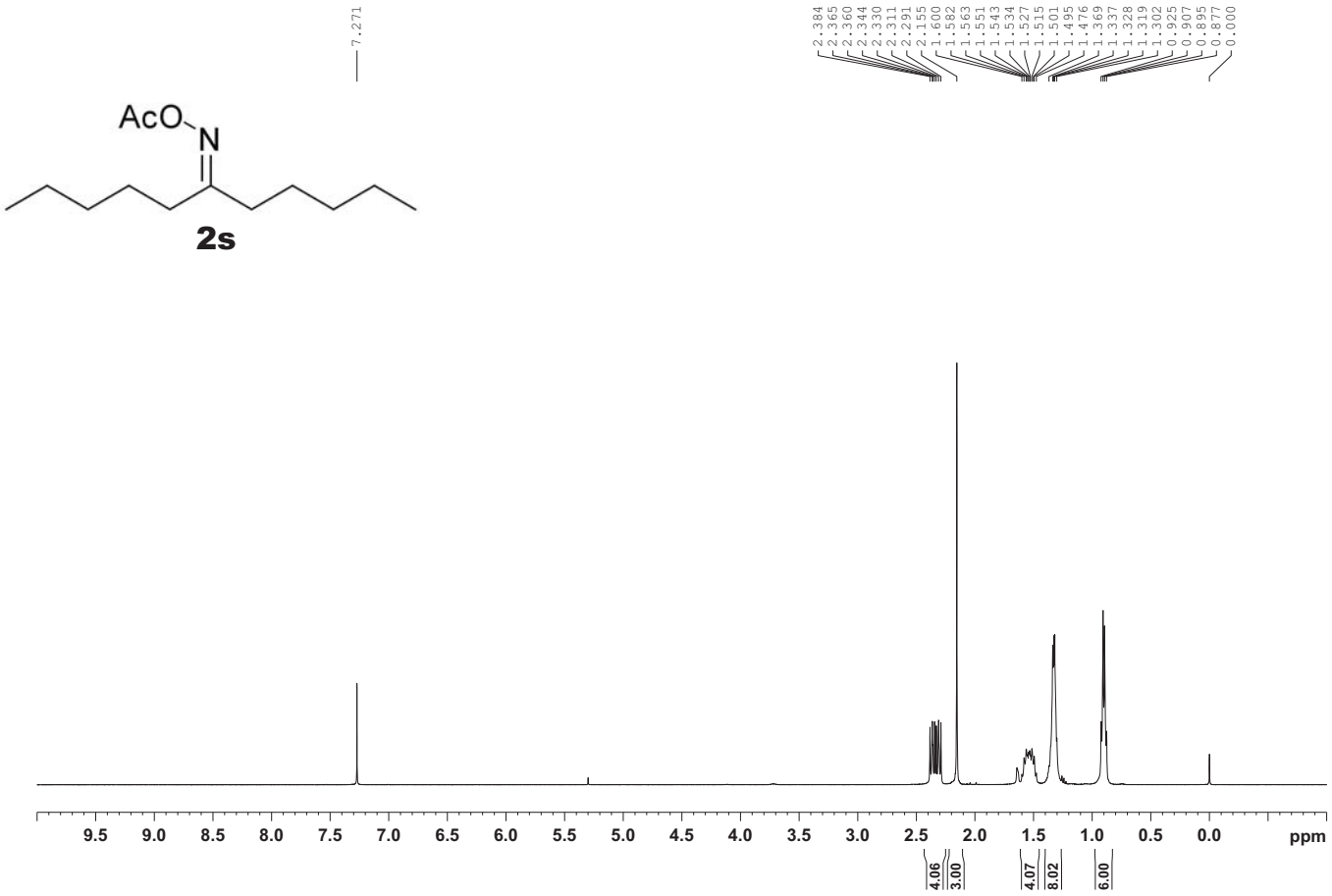
14.34
13.32
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.821
7.801
7.655
7.635
7.617
7.598
7.476
7.458
7.439
7.396
7.378
7.360
7.260
3.683
3.227
3.208
3.187
2.639
2.619
2.599
2.287
0.000
AcO
N
O
O
Ph
2r
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.05
4.03
2.04
1.03
3.01
2.00
2.04
3.00


172.23
168.66
164.02
143.55
139.95
132.03
128.85
127.88
127.67
127.35
127.05
77.32
77.00
76.68
51.89
30.79
23.47
19.77
AcO
N
O
O
Ph
2r
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


AcO
N
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2.365
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2.344
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2.291
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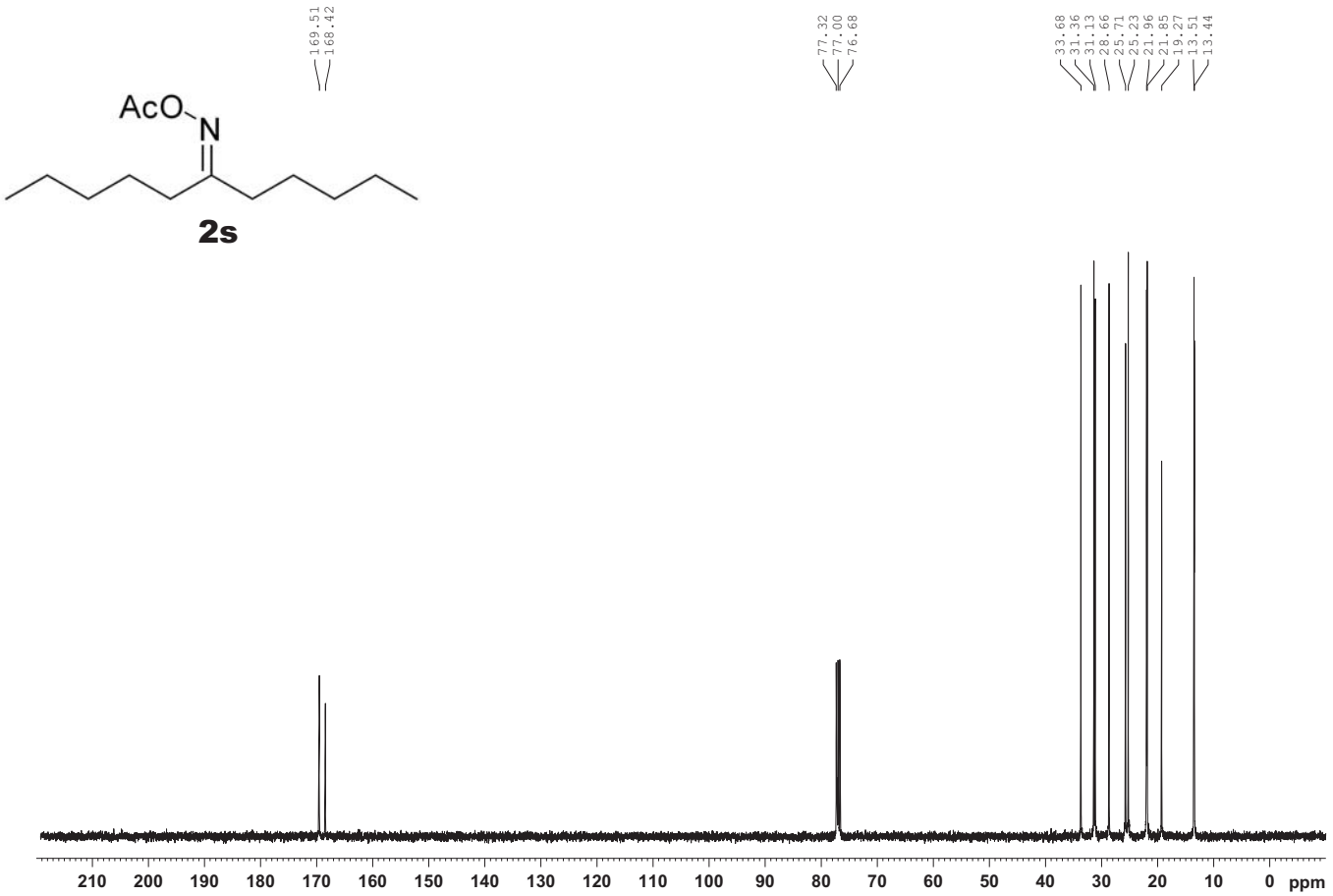
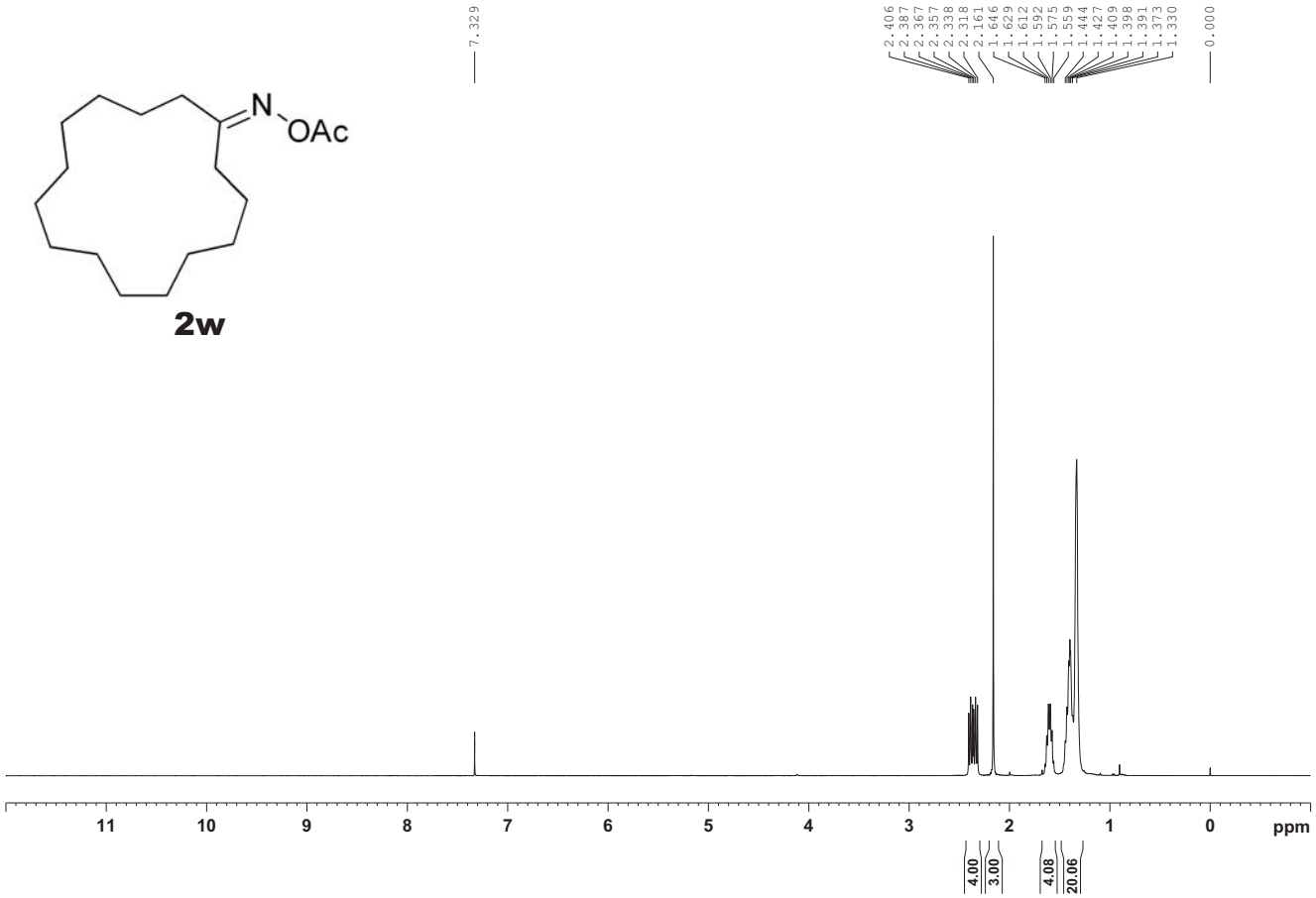
1.551
1.543
1.534
1.527
1.515
1.501
1.495
1.476
1.369
1.337
1.328
1.319
1.302
0.925
0.907
0.895
0.877
0.000
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
4.06
3.00
4.07
8.02
6.00


AcO
N
2s
169.51
168.42
77.32
77.00
76.68
33.68
31.36
31.13
28.66
25.71
25.23
21.96
21.85
19.27
13.51
13.44
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

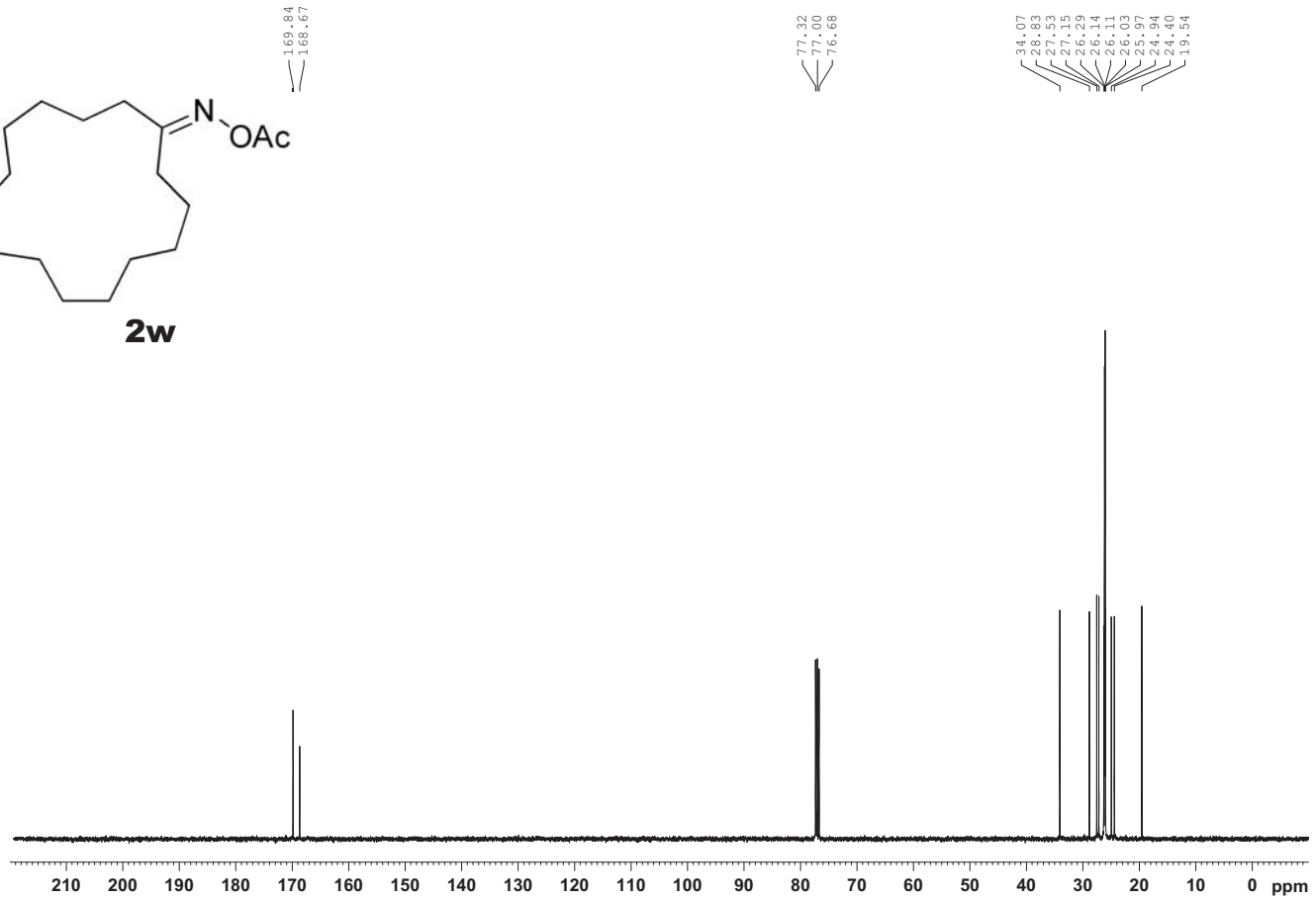
N
OAc

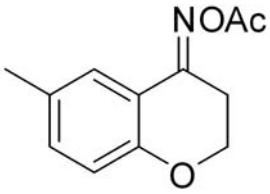
2w


7.329
2.406
2.387
2.367
2.357
2.338
2.318
2.161
1.646
1.629
1.612
1.592
1.575
1.559
1.444
1.427
1.409
1.398
1.391
1.373
1.330
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
4.00
3.00
4.08
20.06

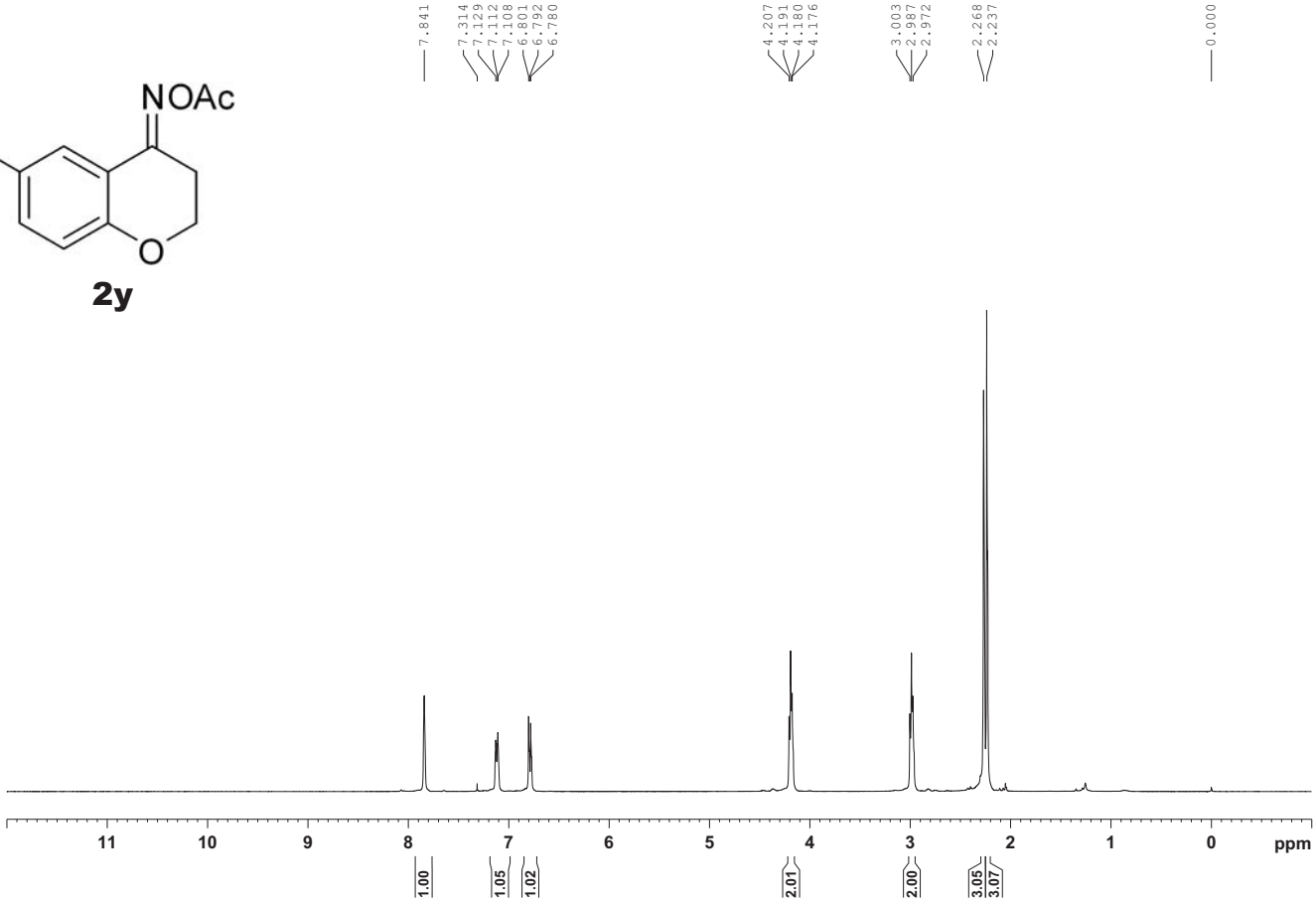
N
OAc

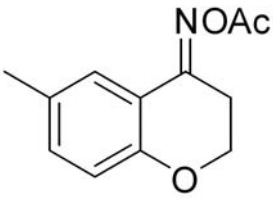
2w


169.84
168.67
77.32
77.00
76.68
34.07
28.83
27.53
27.15
26.29
26.14
26.11
26.03
25.97
24.94
24.40
19.54
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

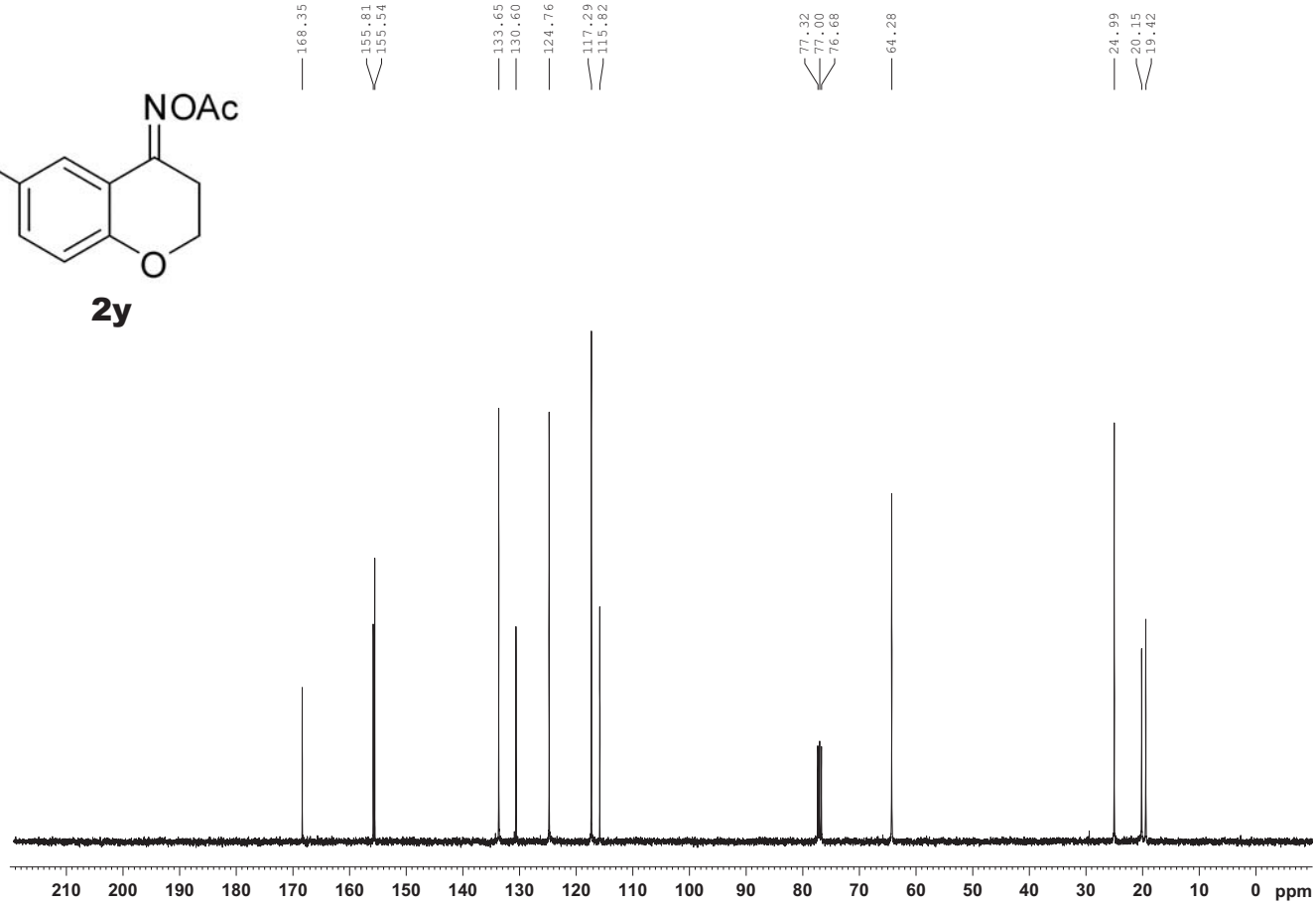

NOAc
O

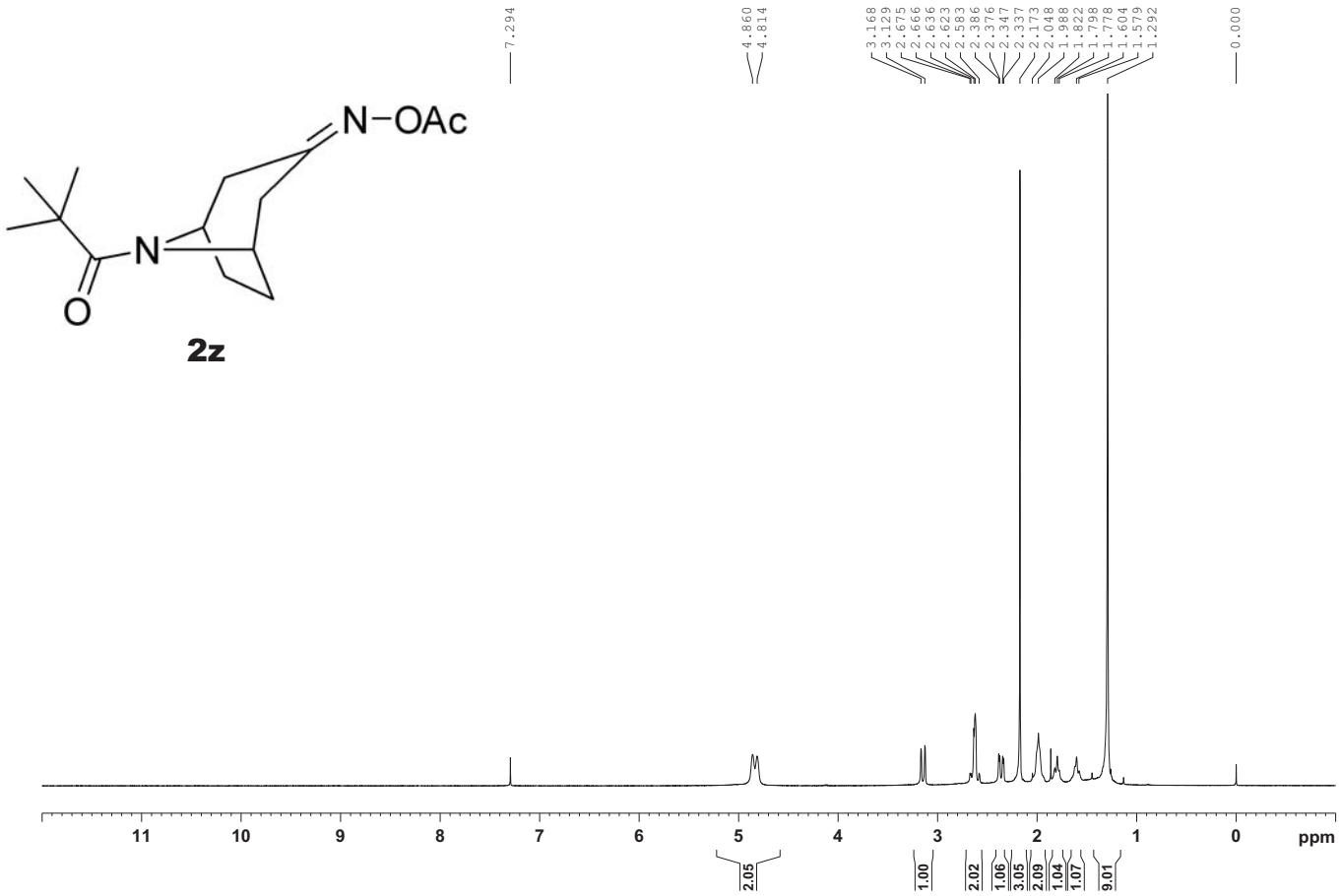
2y

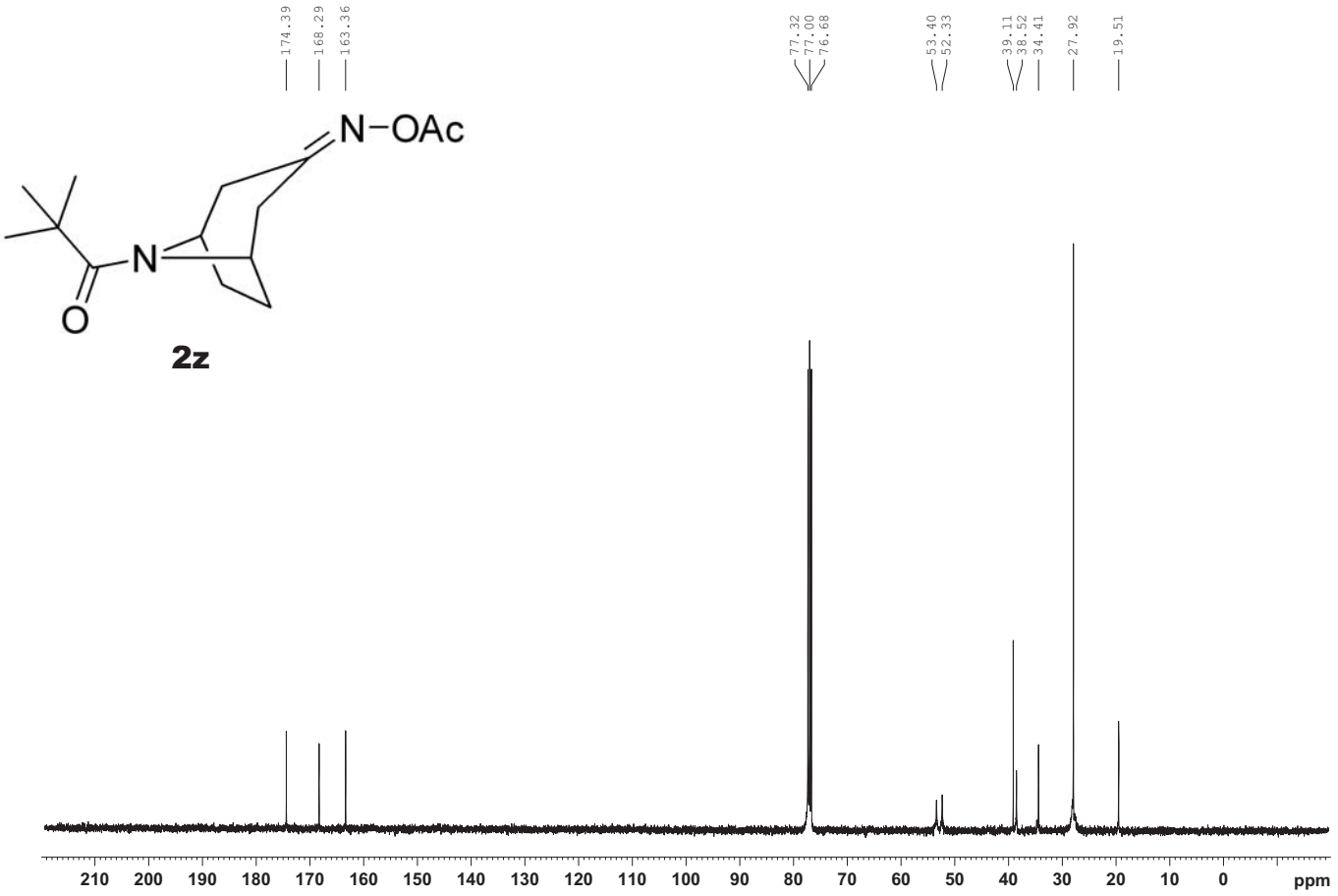

7.841
7.314
7.129
7.112
7.108
6.801
6.792
6.780
4.207
4.191
4.180
4.176
3.003
2.987
2.972
2.268
2.237
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
1.05
1.02
2.01
2.00
3.05
3.07

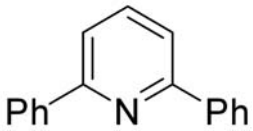

NOAc
O

2y

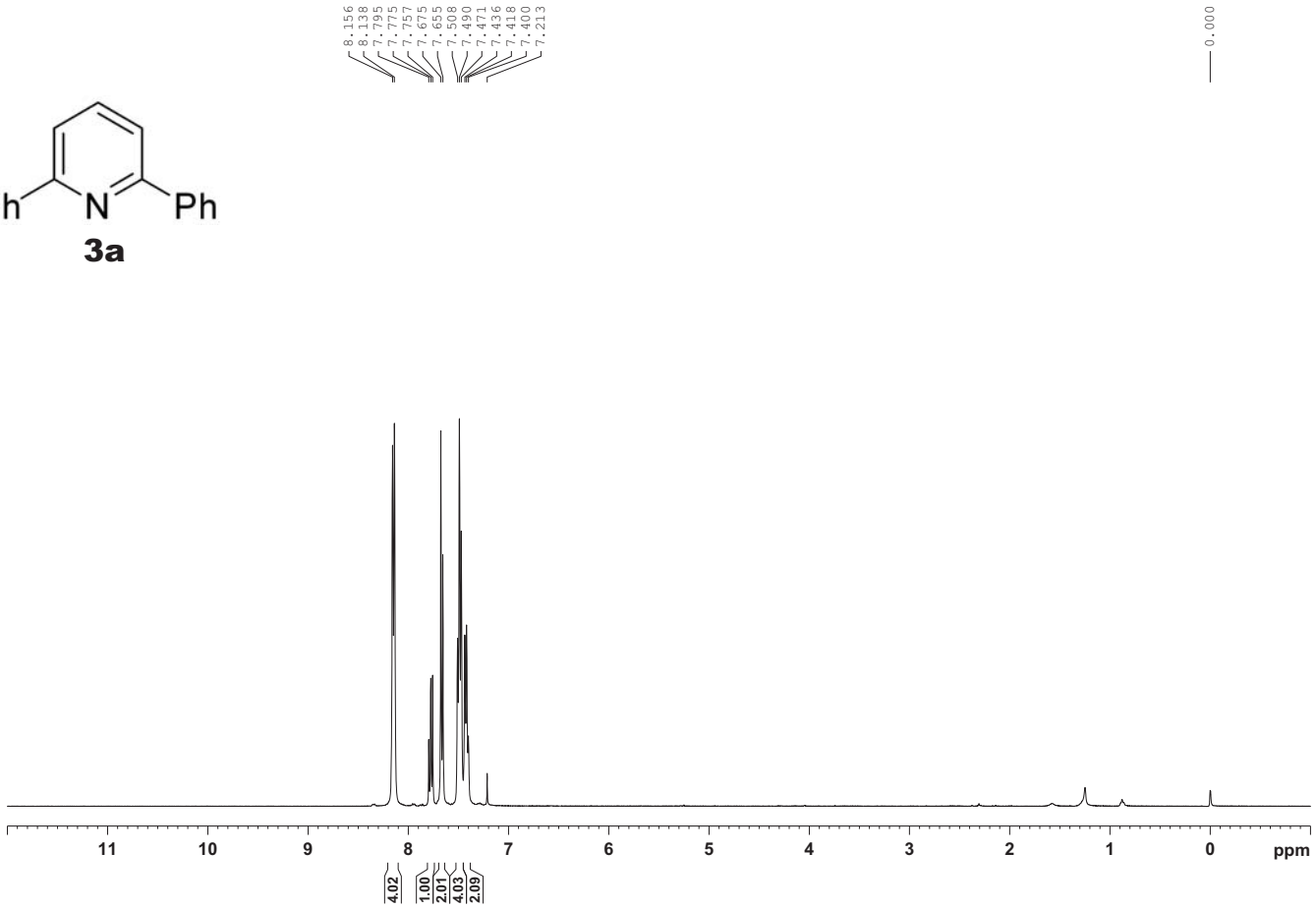

168.35
155.81
155.54
133.65
130.60
124.76
117.29
115.82
77.32
77.00
76.68
64.28
24.99
20.15
19.42
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

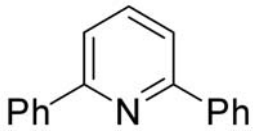

7.294
4.860
4.814
3.168
3.129
2.675
2.666
2.636
2.623
2.583
2.386
2.376
2.347
2.337
2.173
2.048
1.988
1.822
1.798
1.778
1.604
1.579
1.292
0.000
N−OAc
N
O
2z
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.05
1.00
2.02
1.06
3.05
2.09
1.04
1.07
9.01


174.39
168.29
163.36
77.32
77.00
76.68
53.40
52.33
39.11
38.52
34.41
27.92
19.51
N−OAc
N
O
2z
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

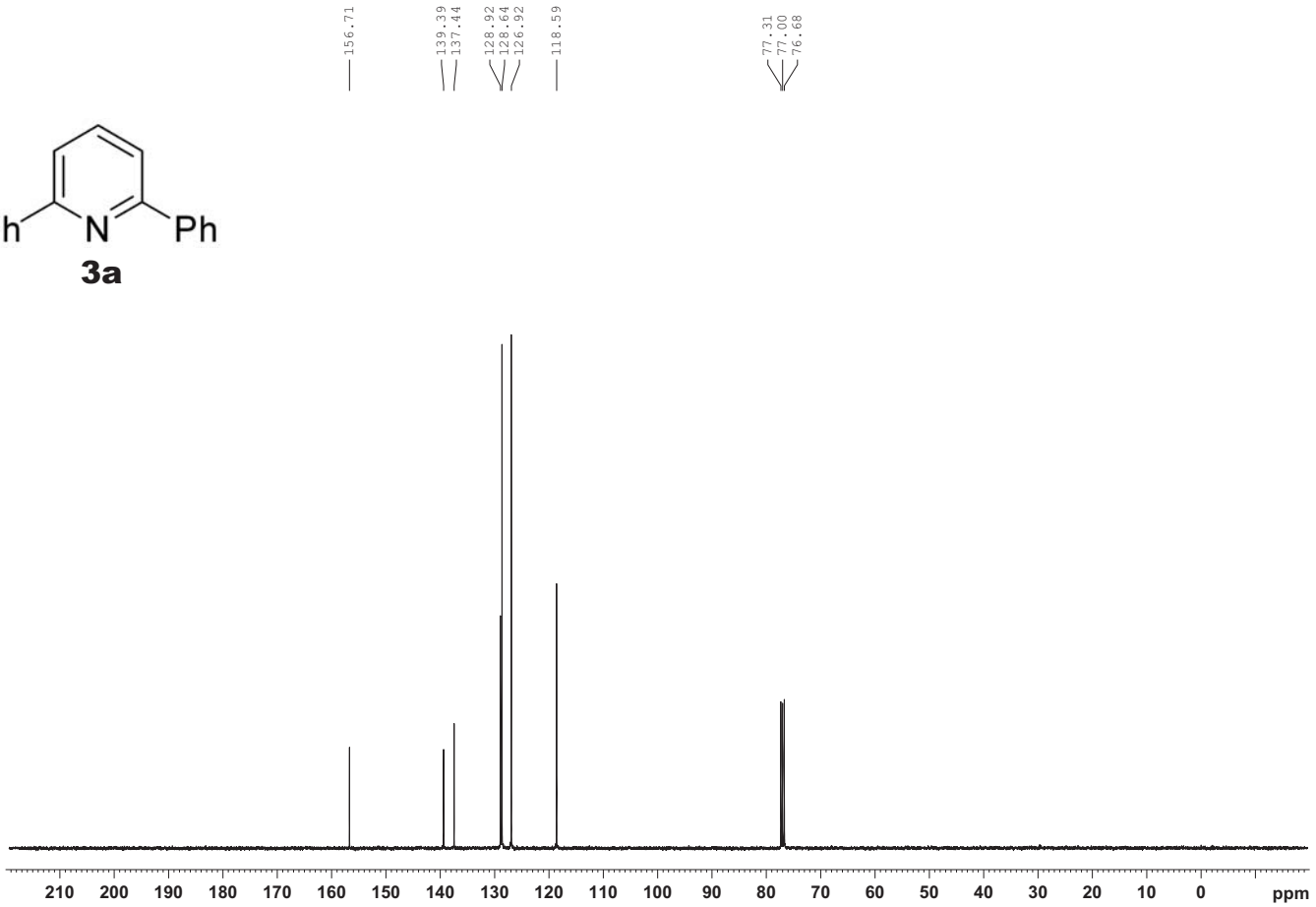

Ph
N
Ph

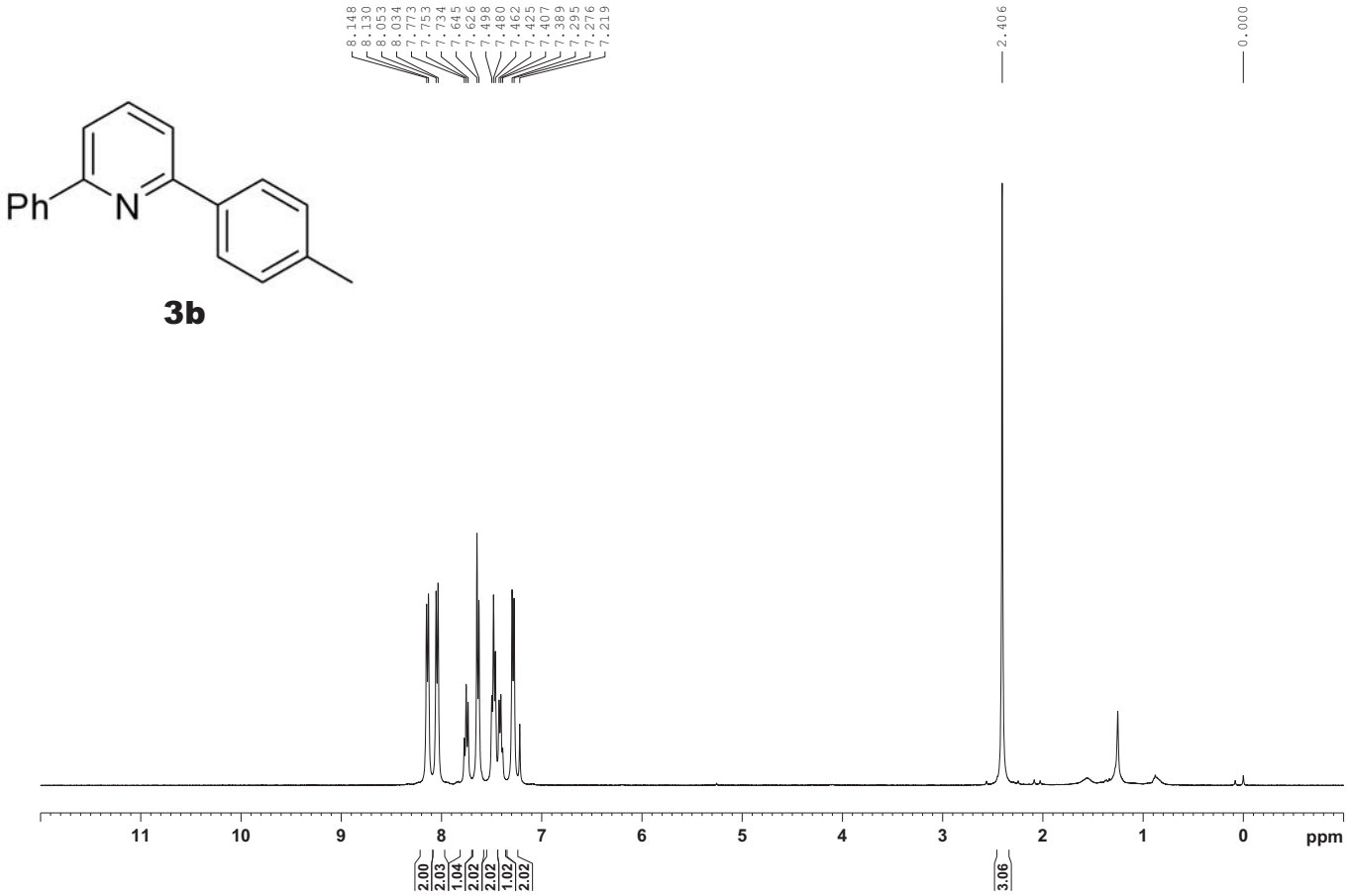
3a


8.156
8.138
7.795
7.775
7.757
7.675
7.655
7.508
7.490
7.471
7.436
7.418
7.400
7.213
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.02
1.00
2.01
4.03
2.09

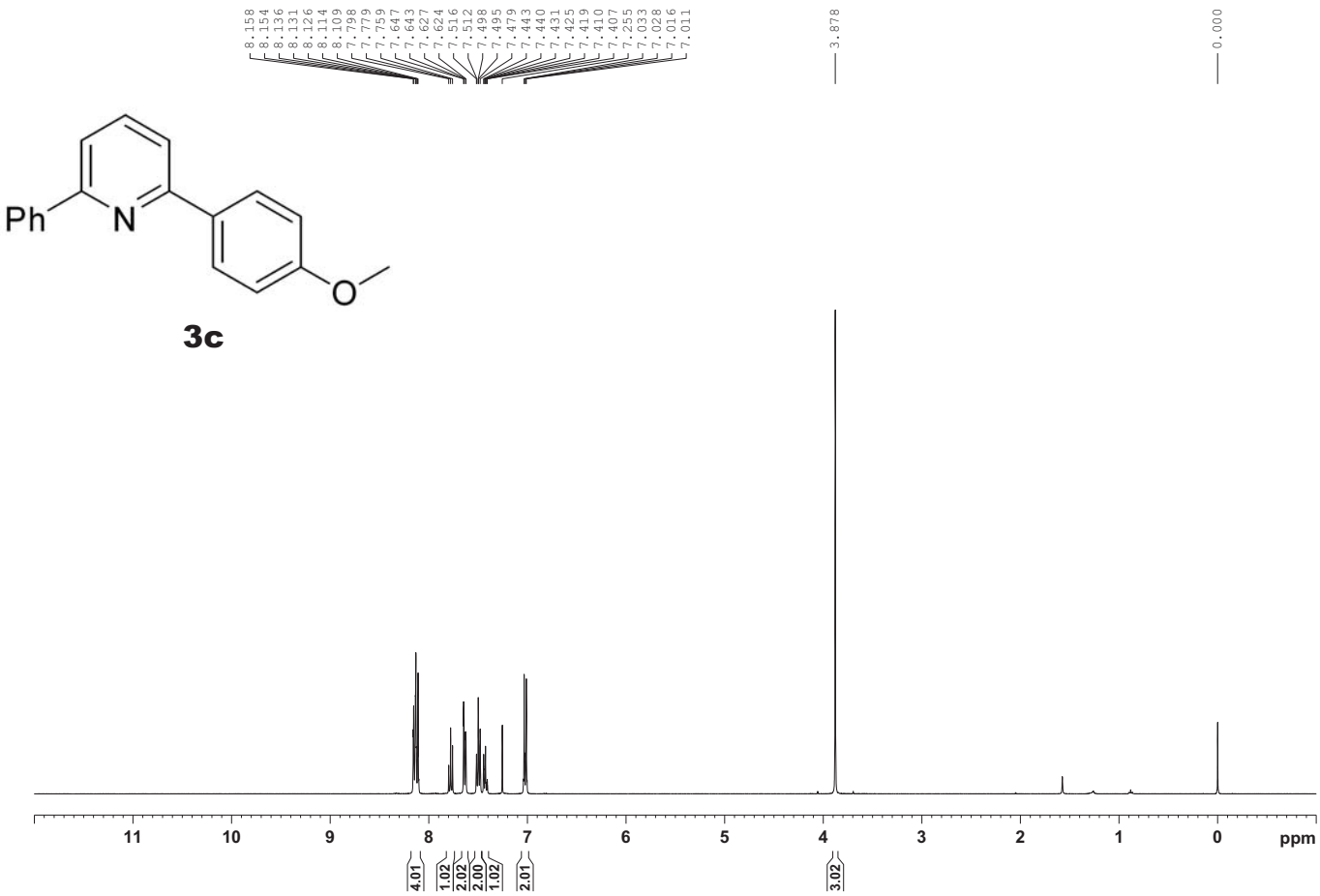

Ph
N
Ph

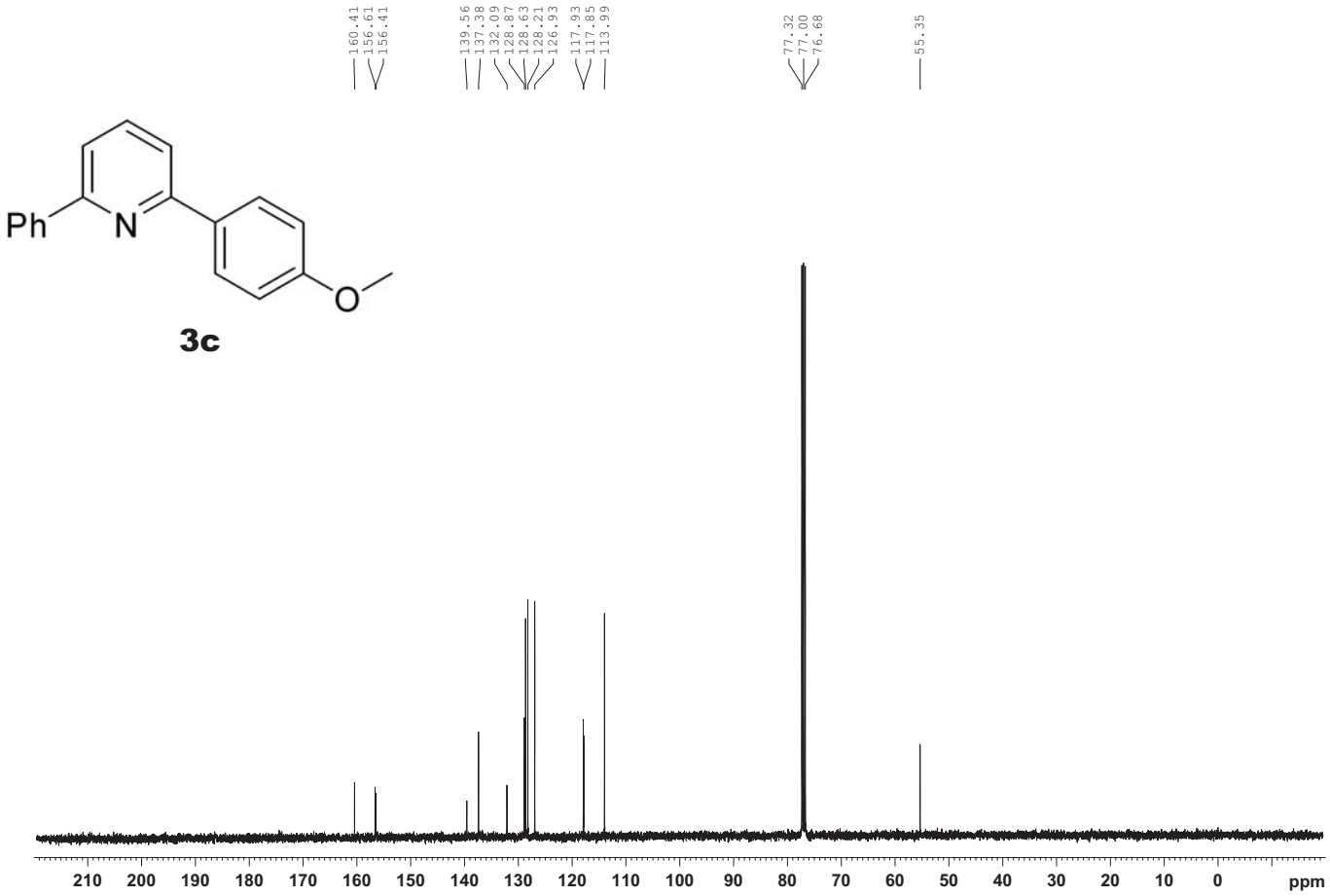
3a

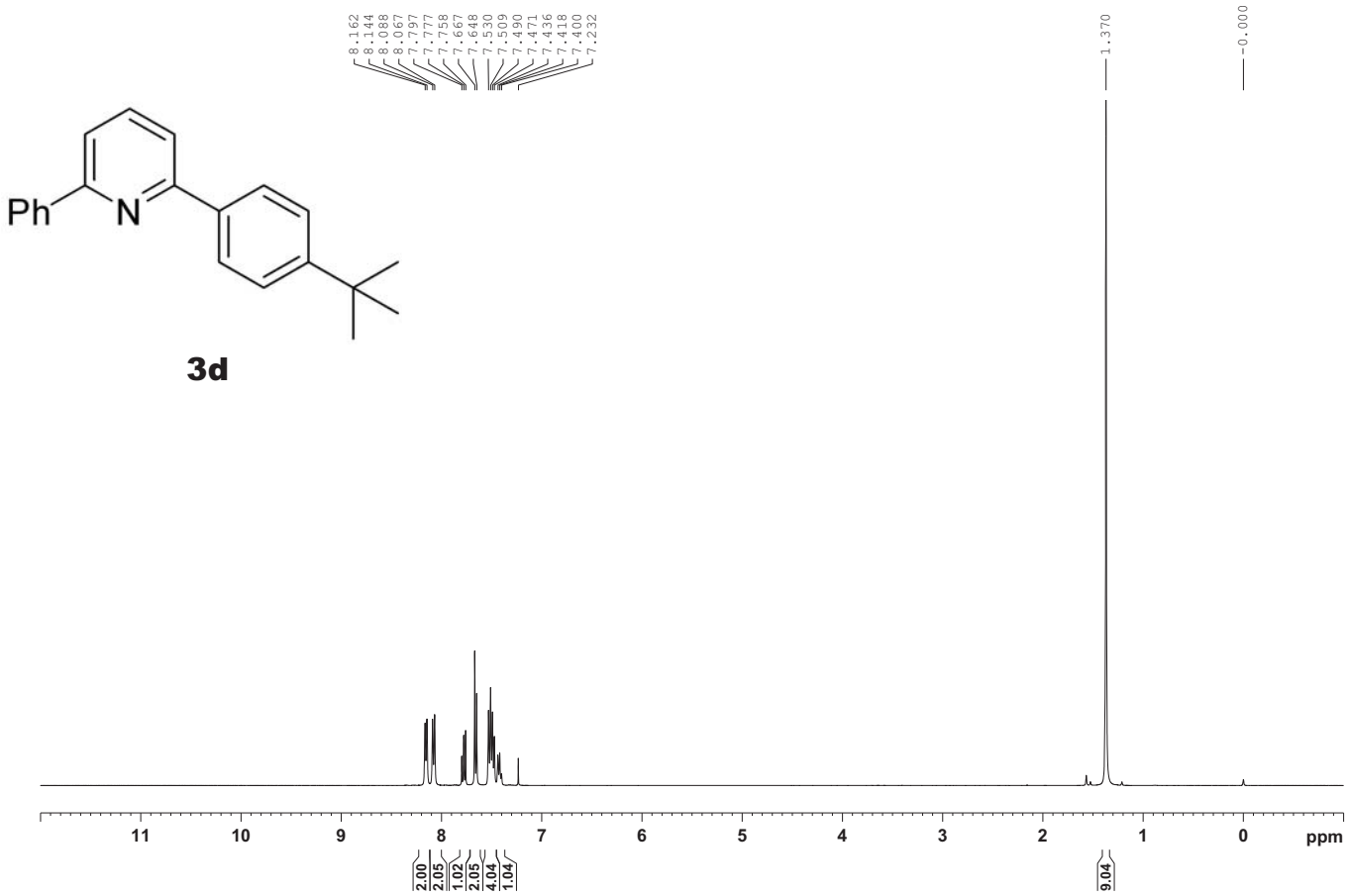

156.71
139.39
137.44
128.92
128.64
126.92
118.59
77.31
77.00
76.68
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

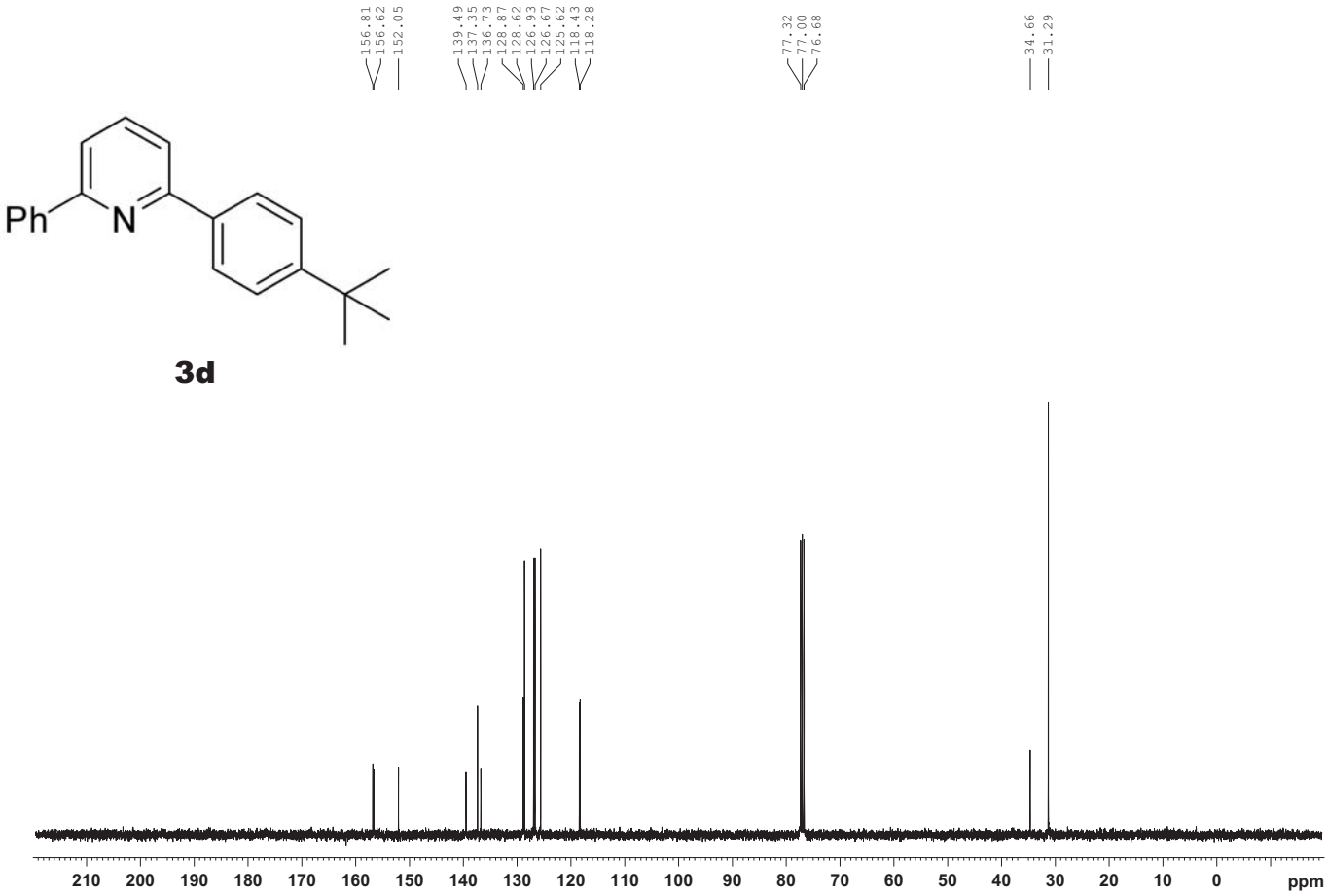

8.148
8.130
8.053
8.034
7.773
7.753
7.734
7.645
7.626
7.498
7.480
7.462
7.425
7.407
7.389
7.295
7.276
7.219
2.406
0.000
Ph
N
3b
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.00
2.03
1.04
2.02
2.02
1.02
2.02
3.06

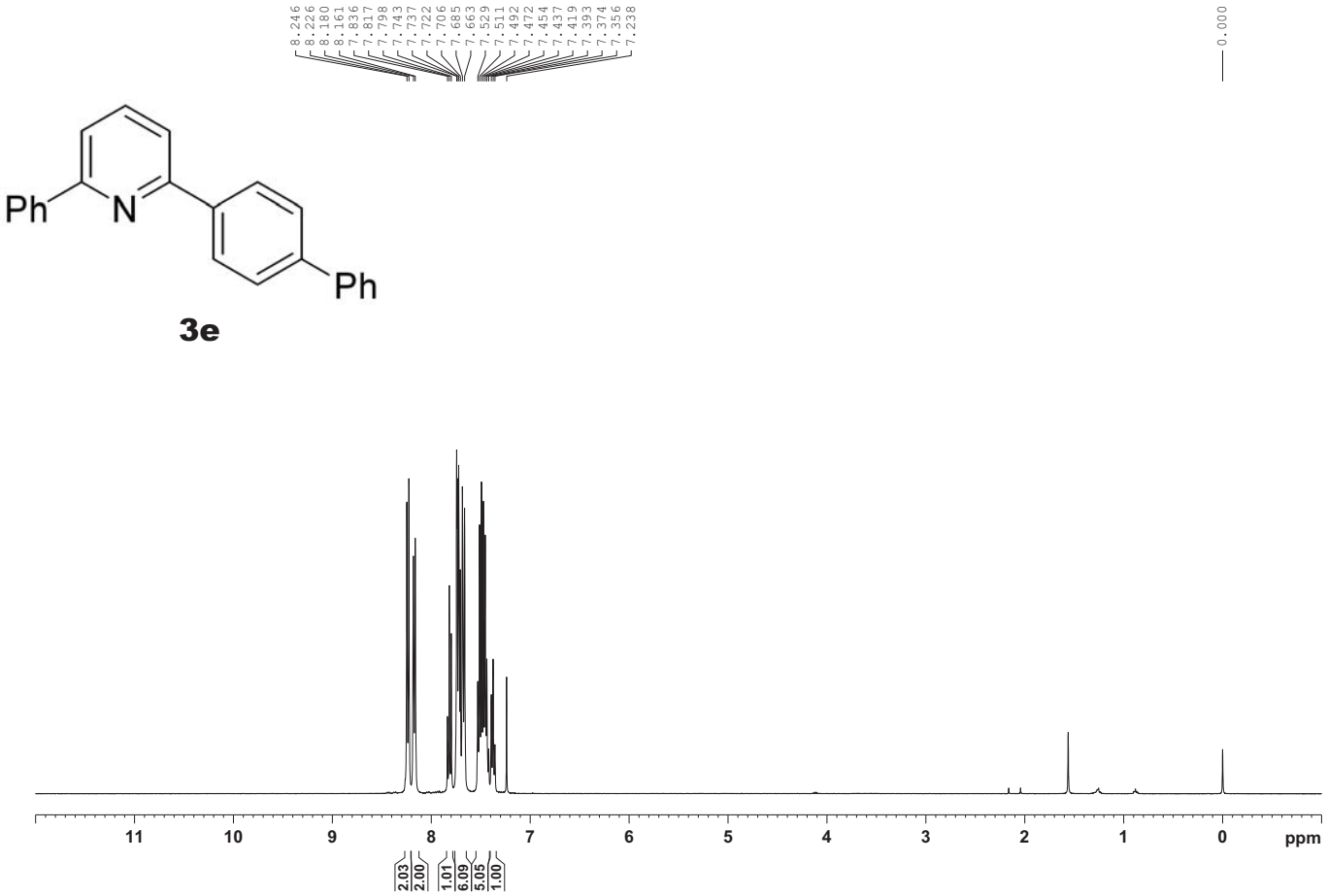

156.68
156.57
139.46
138.87
137.35
136.58
129.35
128.85
128.60
126.90
126.76
118.28
118.25
21.28
Ph
N
3b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


8.158
8.154
8.136
8.131
8.126
8.114
8.109
7.798
7.779
7.759
7.647
7.643
7.627
7.624
7.516
7.512
7.498
7.495
7.479
7.443
7.440
7.431
7.425
7.419
7.410
7.407
7.255
7.033
7.028
7.016
7.011
3.878
0.000
Ph
N
O
3c
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.01
1.02
2.02
2.00
1.02
2.01
3.02


160.41
156.61
156.41
139.56
137.38
132.09
128.87
128.63
128.21
126.93
117.93
117.85
113.99
77.32
77.00
76.68
55.35
Ph
N
O
3c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.162
8.144
8.088
8.067
7.797
7.777
7.758
7.667
7.648
7.530
7.509
7.490
7.471
7.436
7.418
7.400
7.232
1.370
-0.000
Ph
N
3d
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
2.05
1.02
2.05
4.04
1.04
9.04


156.81
156.62
152.05
139.49
137.35
136.73
128.87
128.62
126.93
126.67
125.62
118.43
118.28
77.32
77.00
76.68
34.66
31.29
Ph
N
3d
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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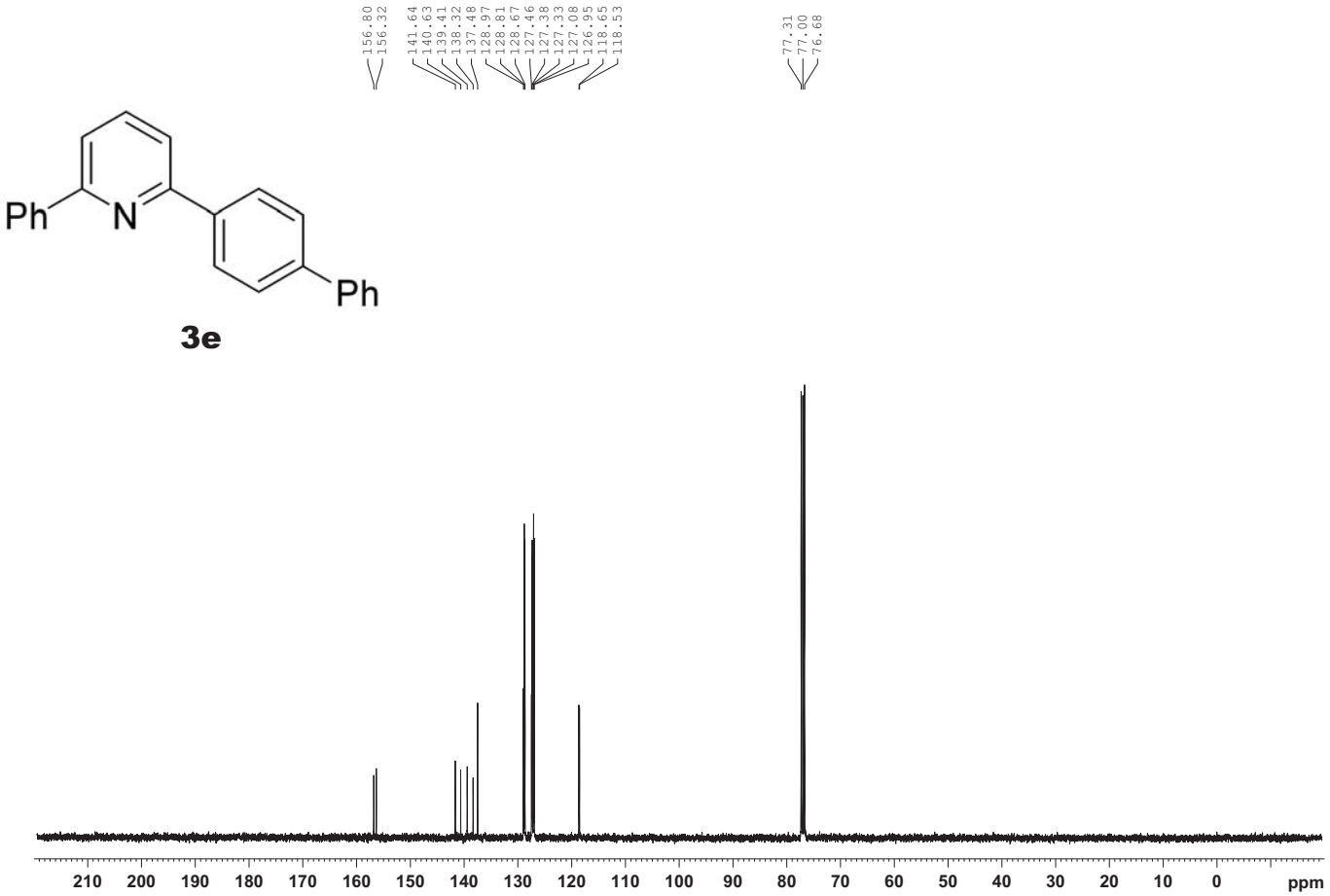
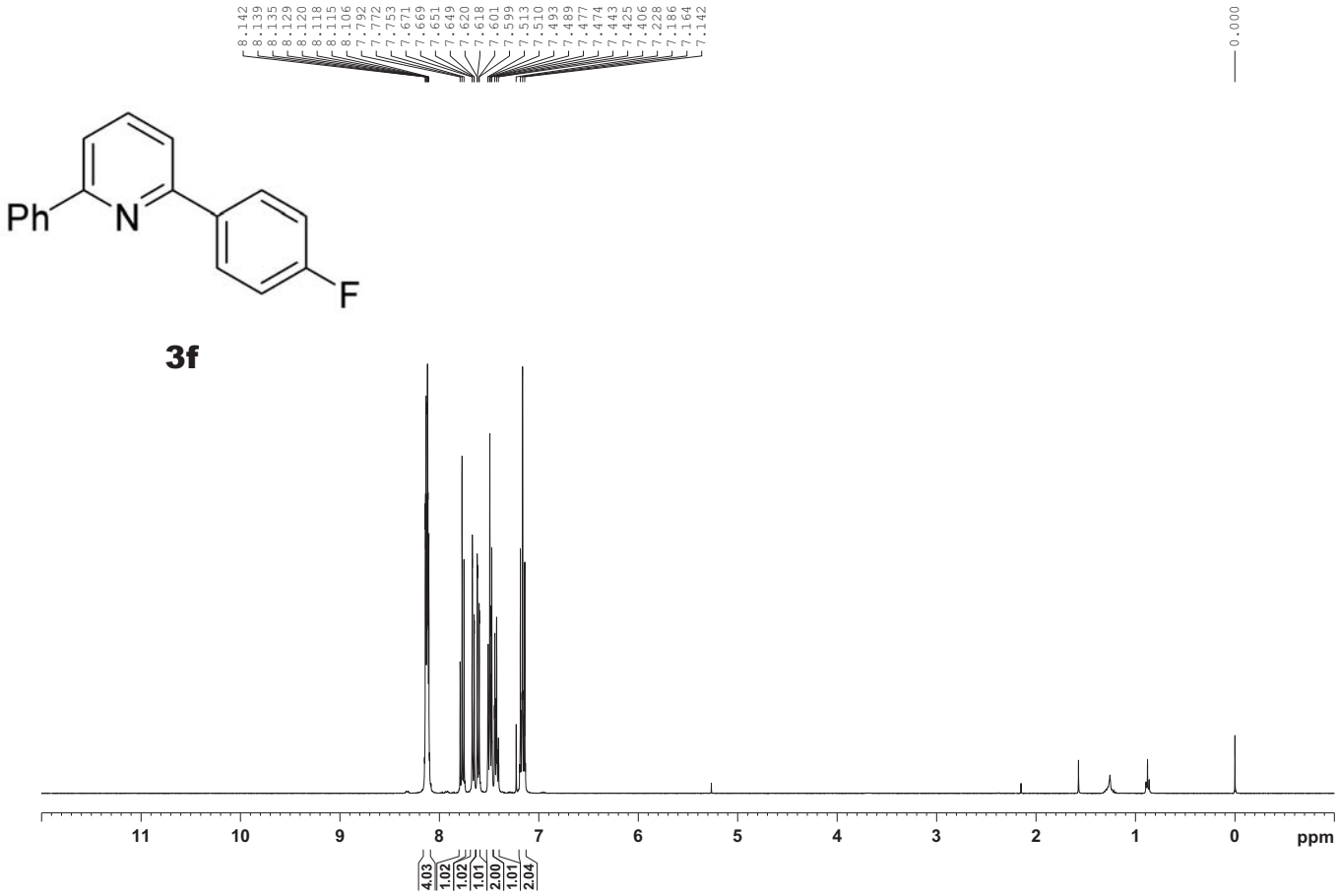
8.226
8.180
8.161
7.836
7.817
7.798
7.743
7.737
7.722
7.706
7.685
7.663
7.529
7.511
7.492
7.472
7.454
7.437
7.419
7.393
7.374
7.356
7.238
0.000
Ph
N
Ph
3e
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.03
2.00
1.01
6.09
5.05
1.00


156.80
156.32
141.64
140.63
139.41
138.32
137.48
128.97
128.81
128.67
127.46
127.38
127.33
127.08
126.95
118.65
118.53
77.31
77.00
76.68
Ph
N
Ph
3e
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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8.139
8.135
8.129
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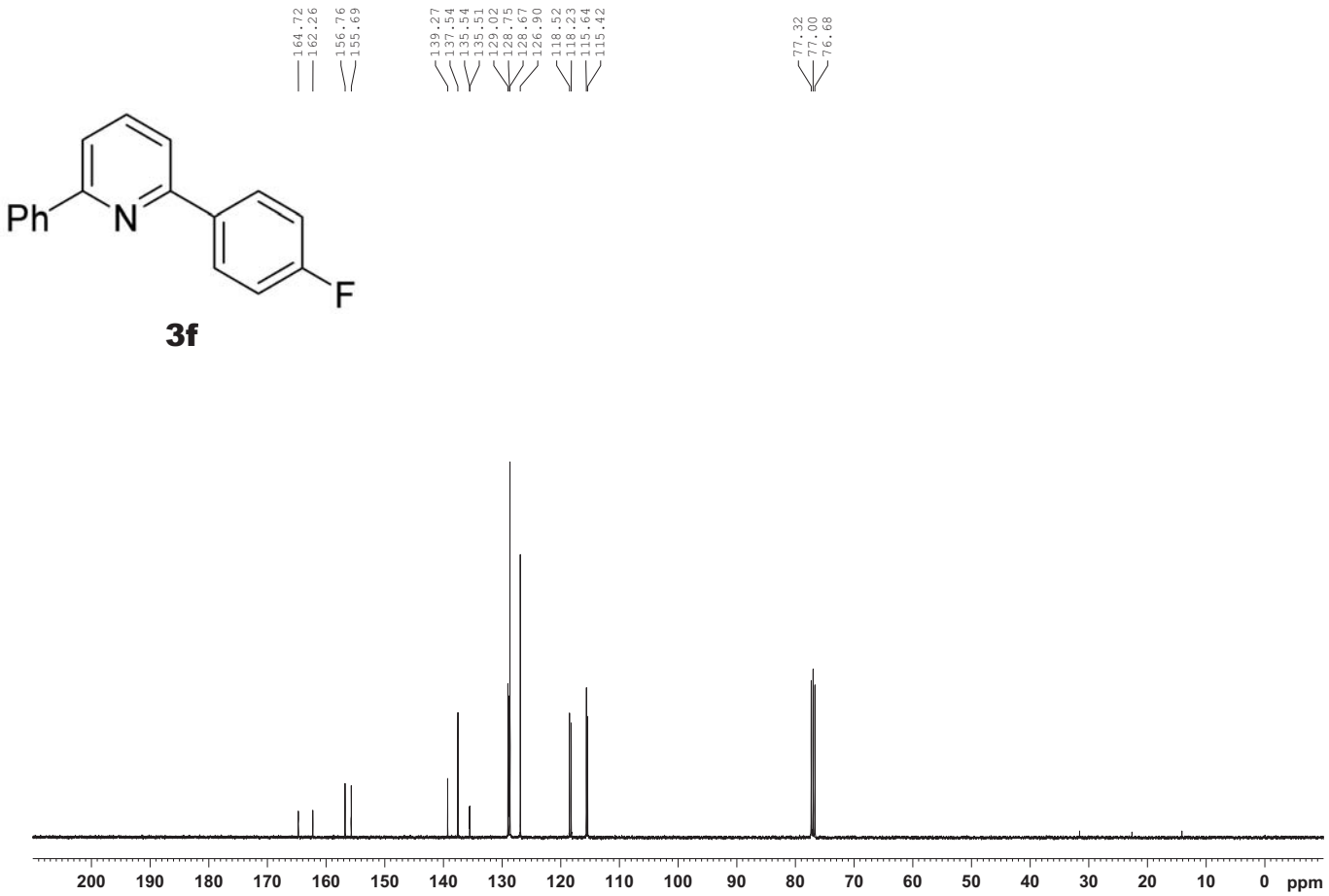
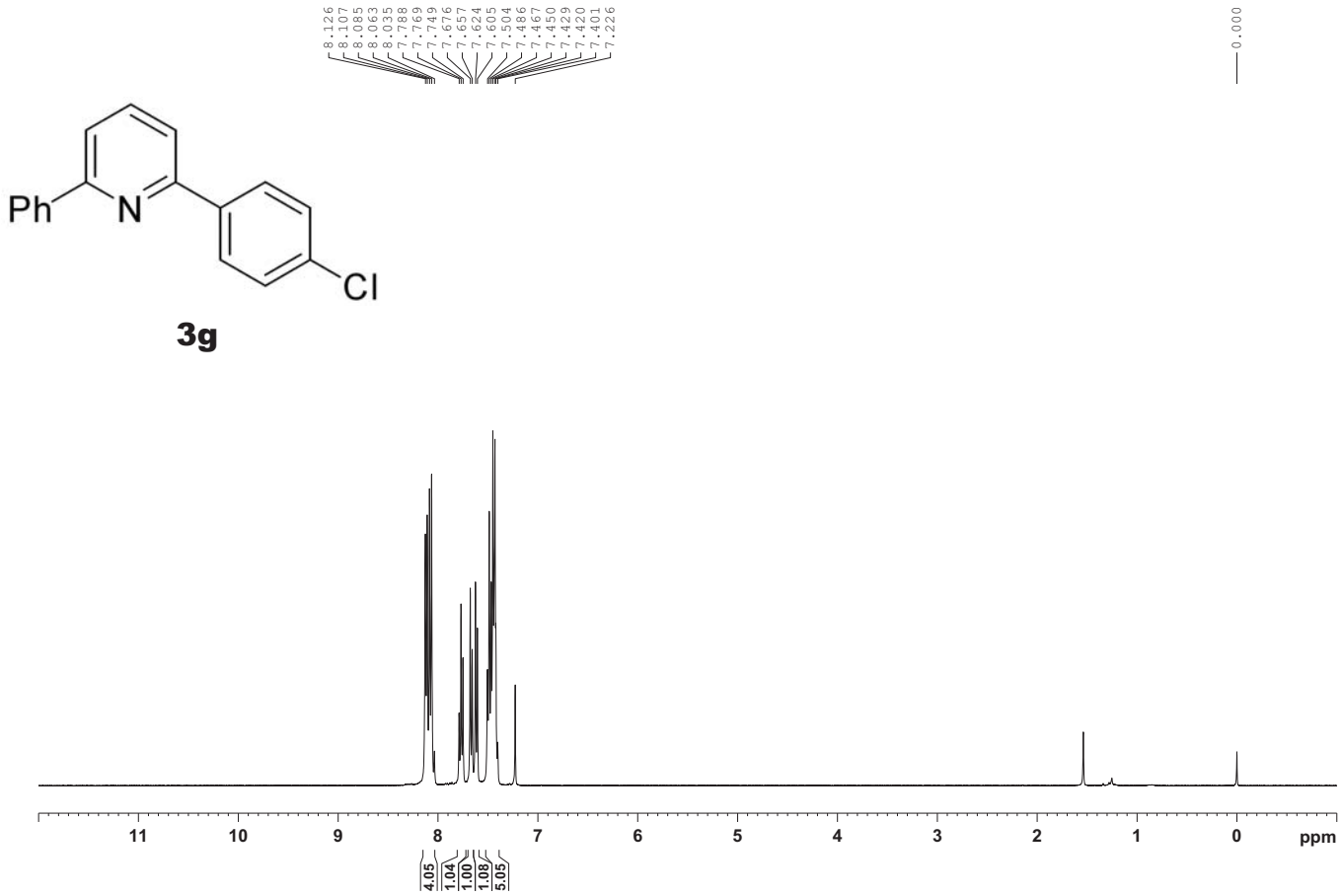
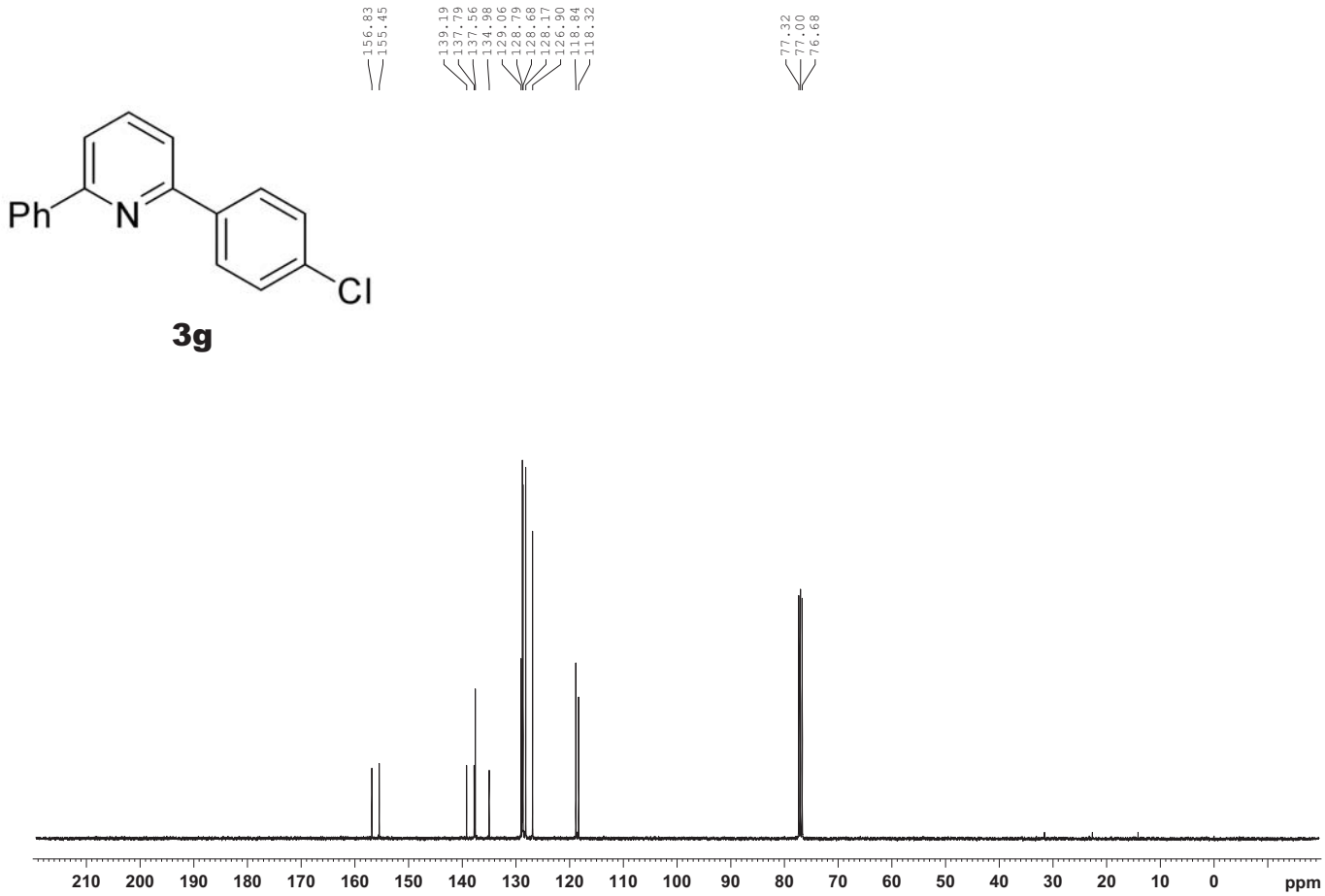
8.118
8.115
8.106
7.792
7.772
7.753
7.671
7.669
7.651
7.649
7.620
7.618
7.601
7.599
7.513
7.510
7.493
7.489
7.477
7.474
7.443
7.425
7.406
7.228
7.186
7.164
7.142
0.000
Ph
N
F
3f
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.03
1.02
1.02
1.01
2.00
1.01
2.04


164.72
162.26
156.76
155.69
139.27
137.54
135.54
135.51
129.02
128.75
128.67
126.90
118.52
118.23
115.64
115.42
77.32
77.00
76.68
Ph
N
F
3f
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.126
8.107
8.085
8.063
8.035
7.788
7.769
7.749
7.676
7.657
7.624
7.605
7.504
7.486
7.467
7.450
7.429
7.420
7.401
7.226
0.000
Ph
N
Cl
3g
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.05
1.04
1.00
1.08
5.05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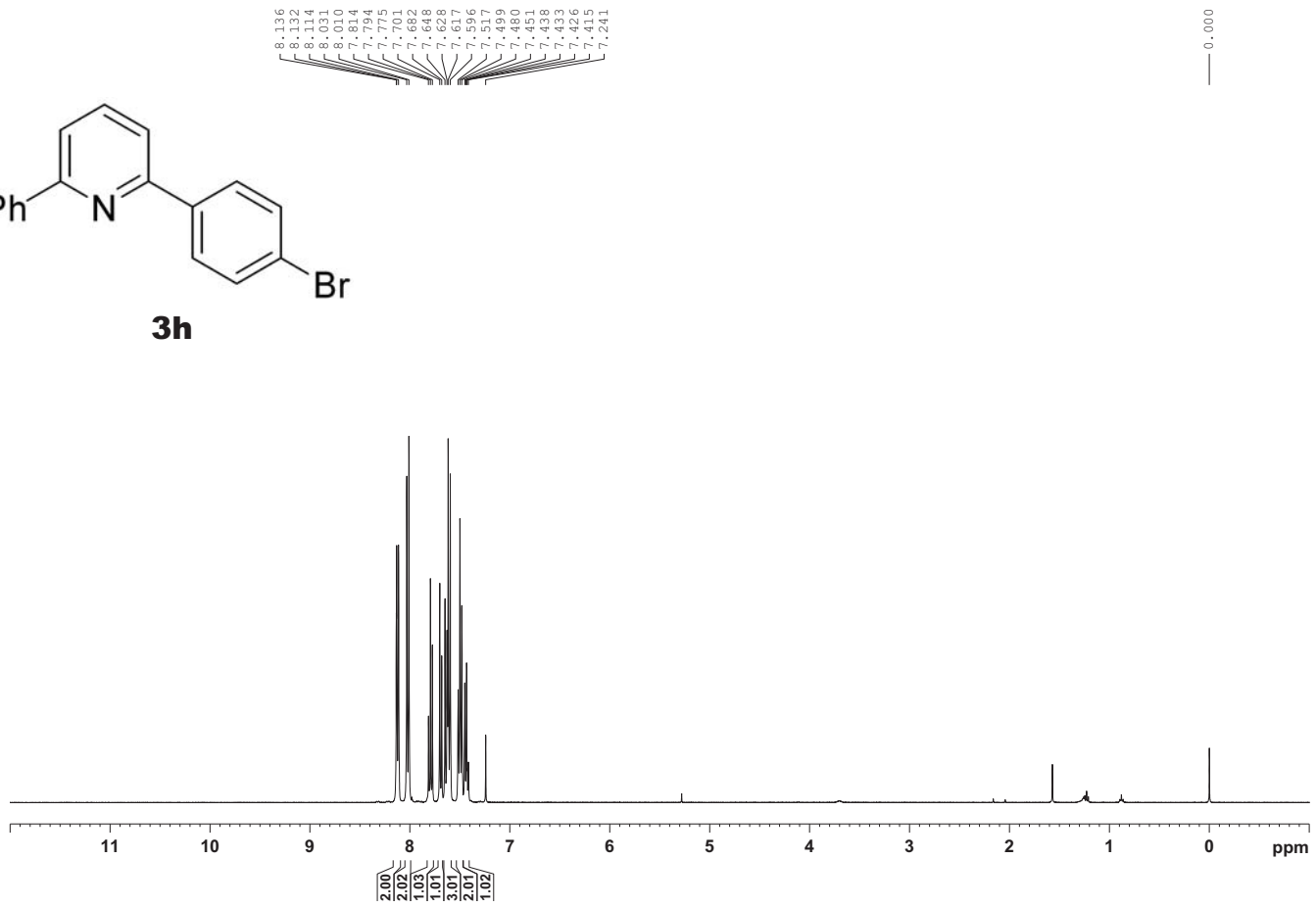
137.79
137.56
134.98
129.06
128.79
128.68
128.17
126.90
118.84
118.32
77.32
77.00
76.68
Ph
N
Cl
3g
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

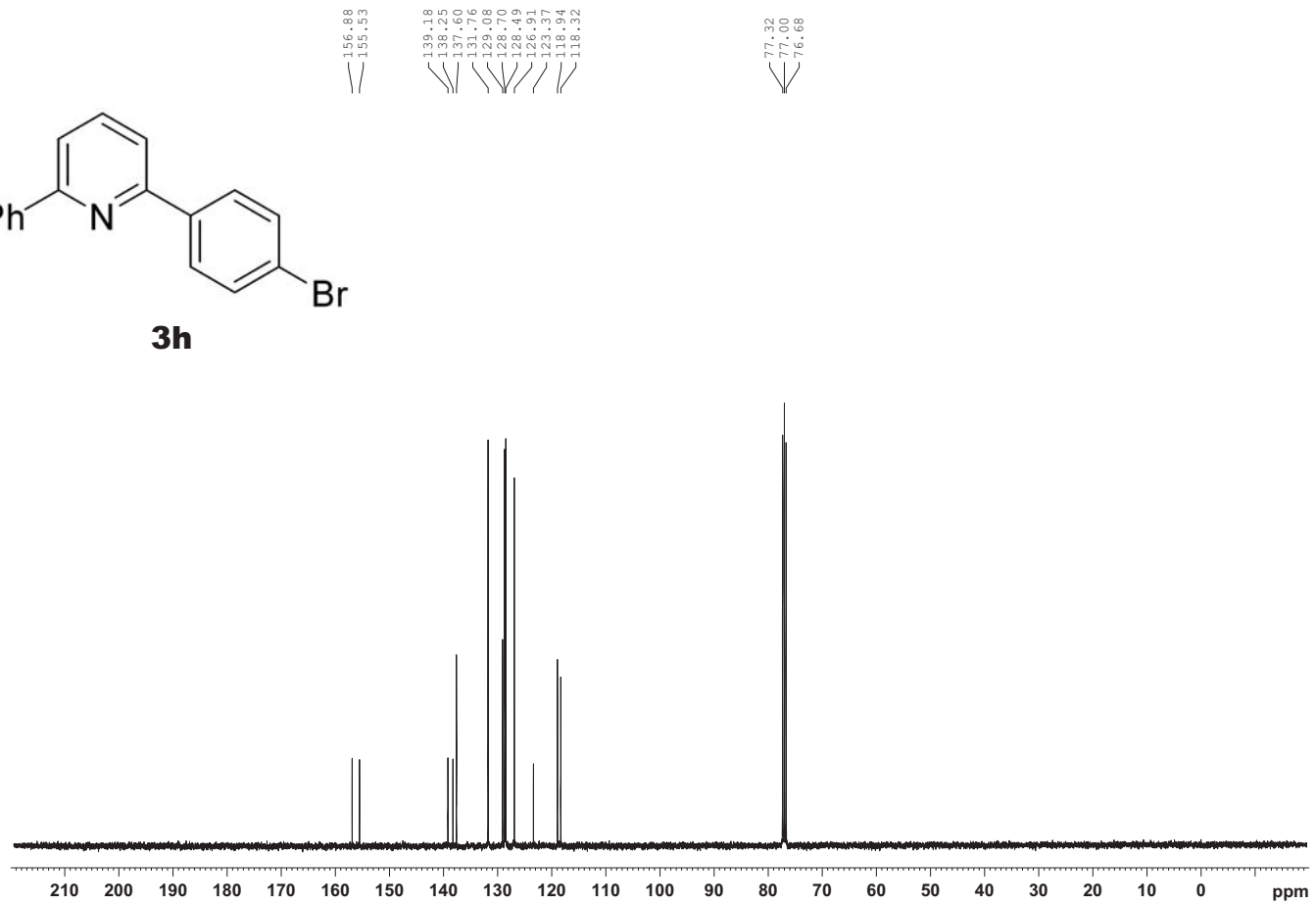
Ph
N
Br

3h


8.136
8.132
8.114
8.031
8.010
7.814
7.794
7.775
7.701
7.682
7.648
7.628
7.617
7.596
7.517
7.499
7.480
7.451
7.438
7.433
7.426
7.415
7.241
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
2.02
1.03
1.01
3.01
2.01
1.02

Ph
N
Br

3h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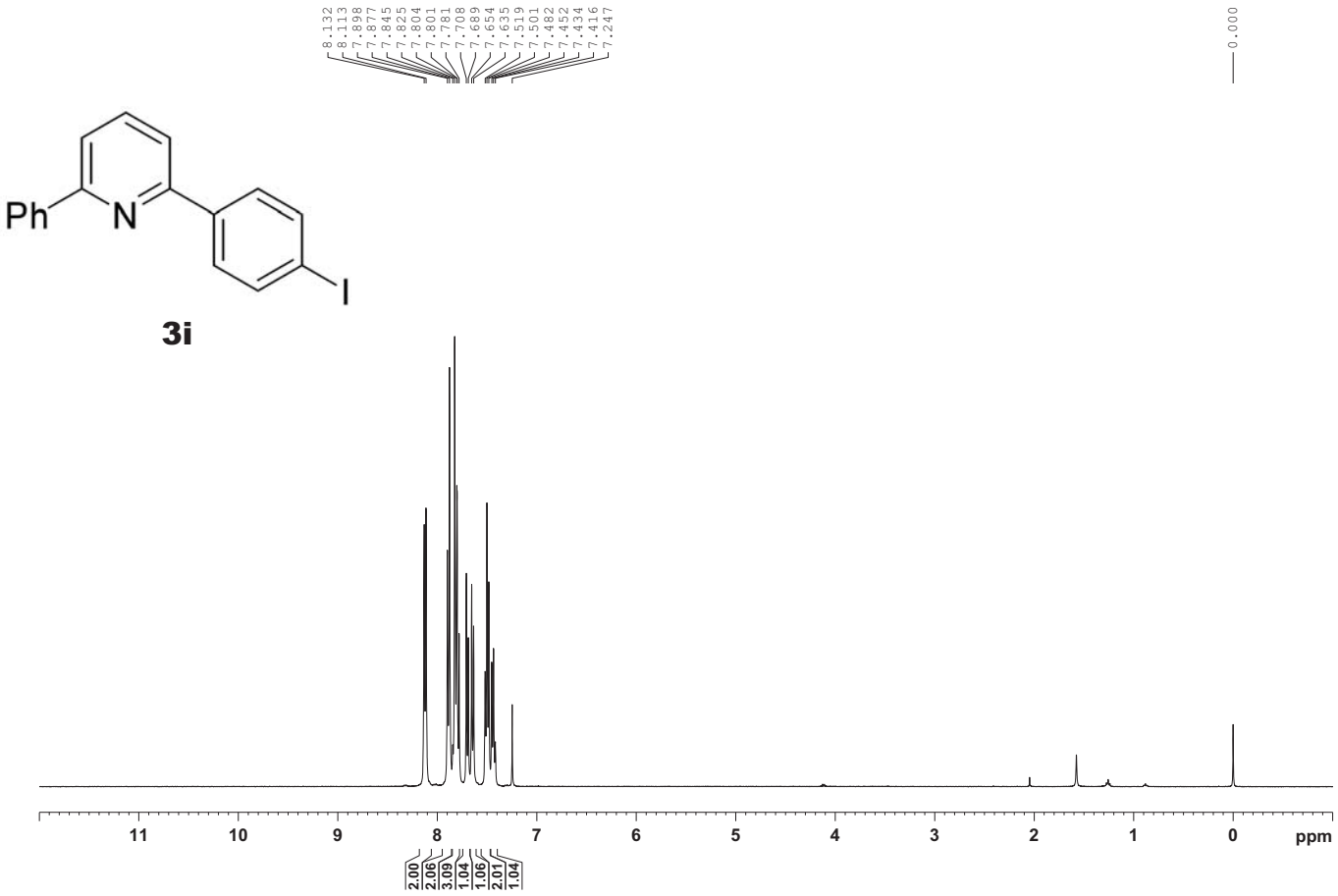
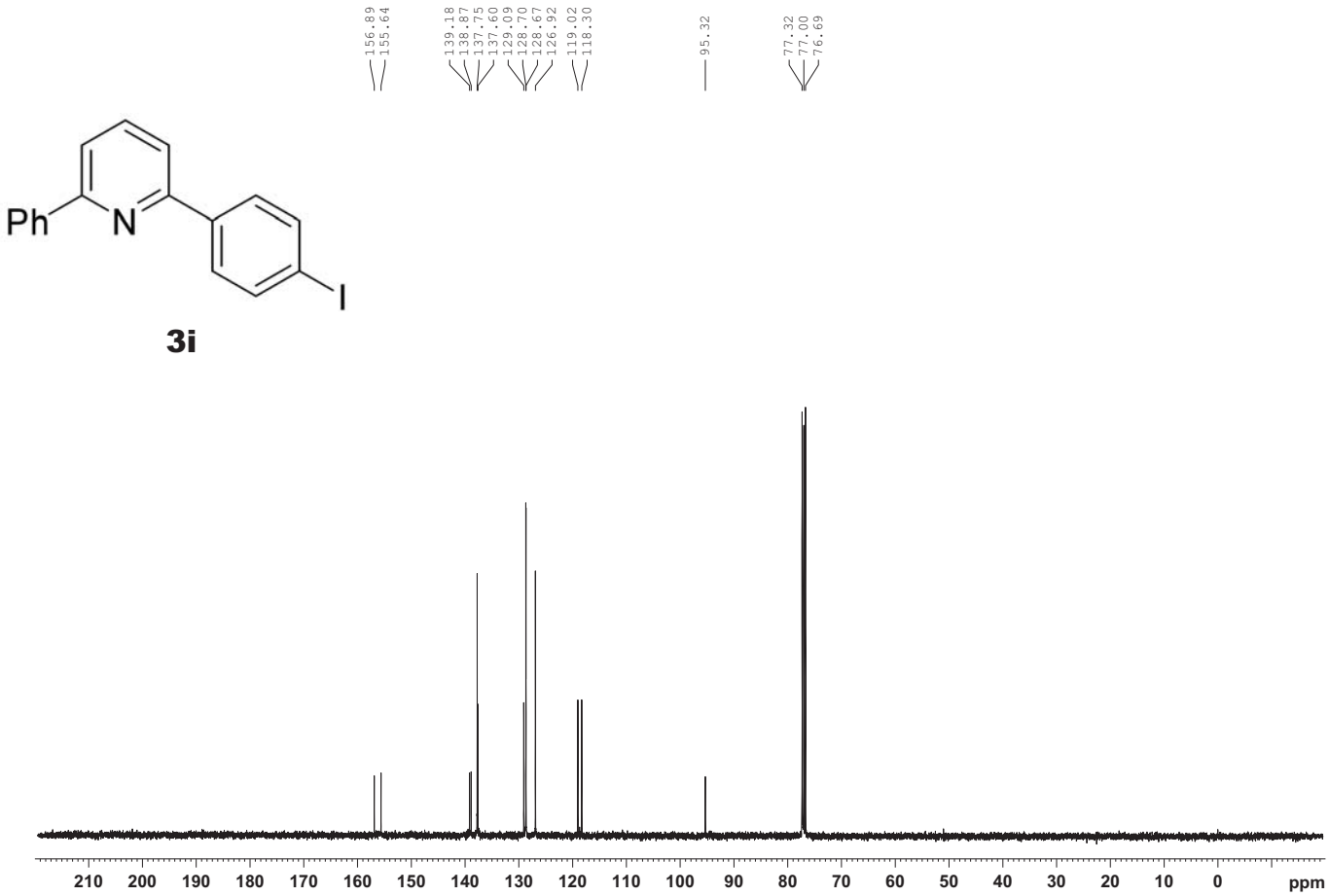
139.18
138.25
137.60
131.76
129.08
128.70
128.49
126.91
123.37
118.94
118.32
77.32
77.00
76.68
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.132
8.113
7.898
7.877
7.845
7.825
7.804
7.801
7.781
7.708
7.689
7.654
7.635
7.519
7.501
7.482
7.452
7.434
7.416
7.247
0.000
Ph
N
I
3i
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
2.06
3.09
1.04
1.06
2.01
1.04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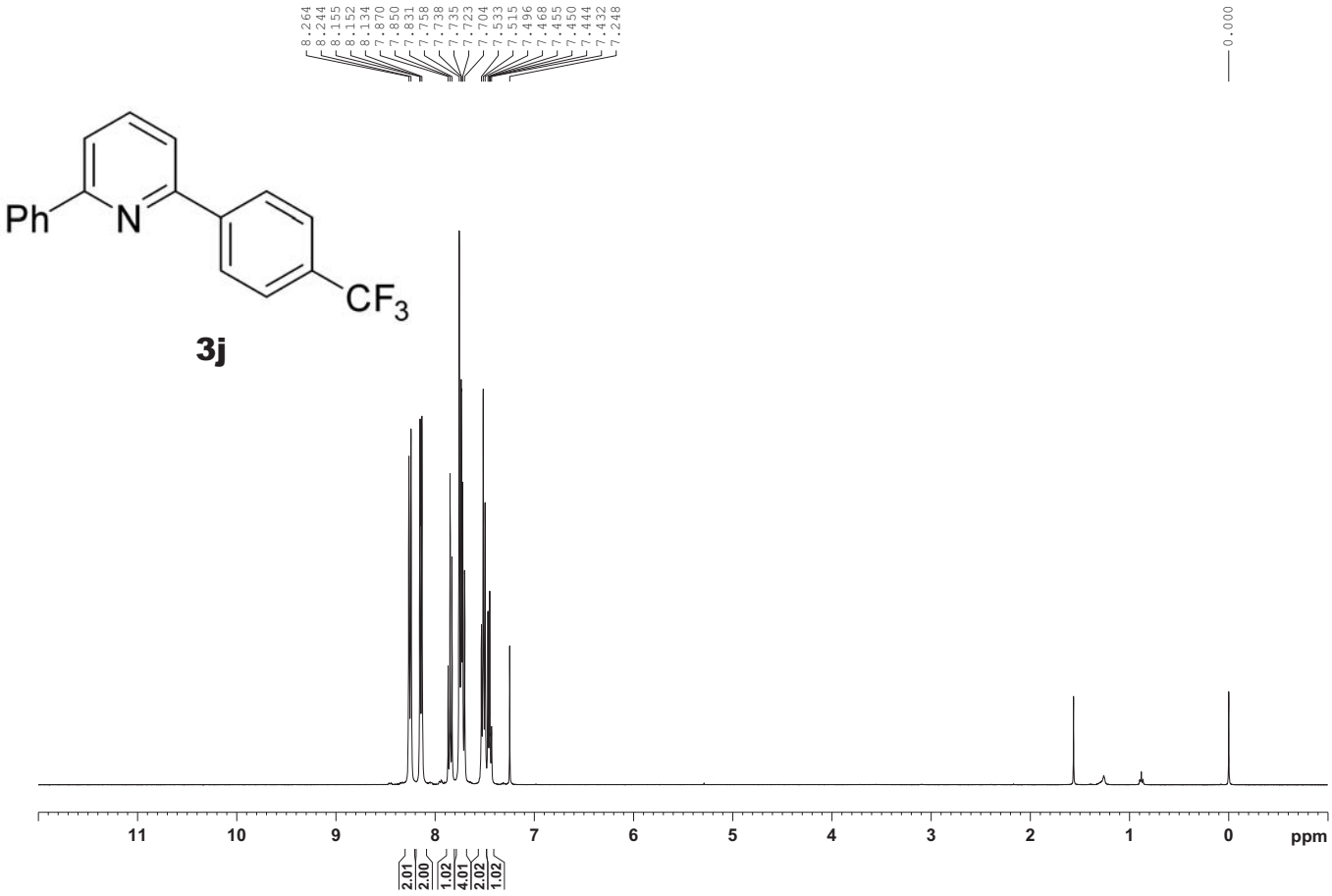
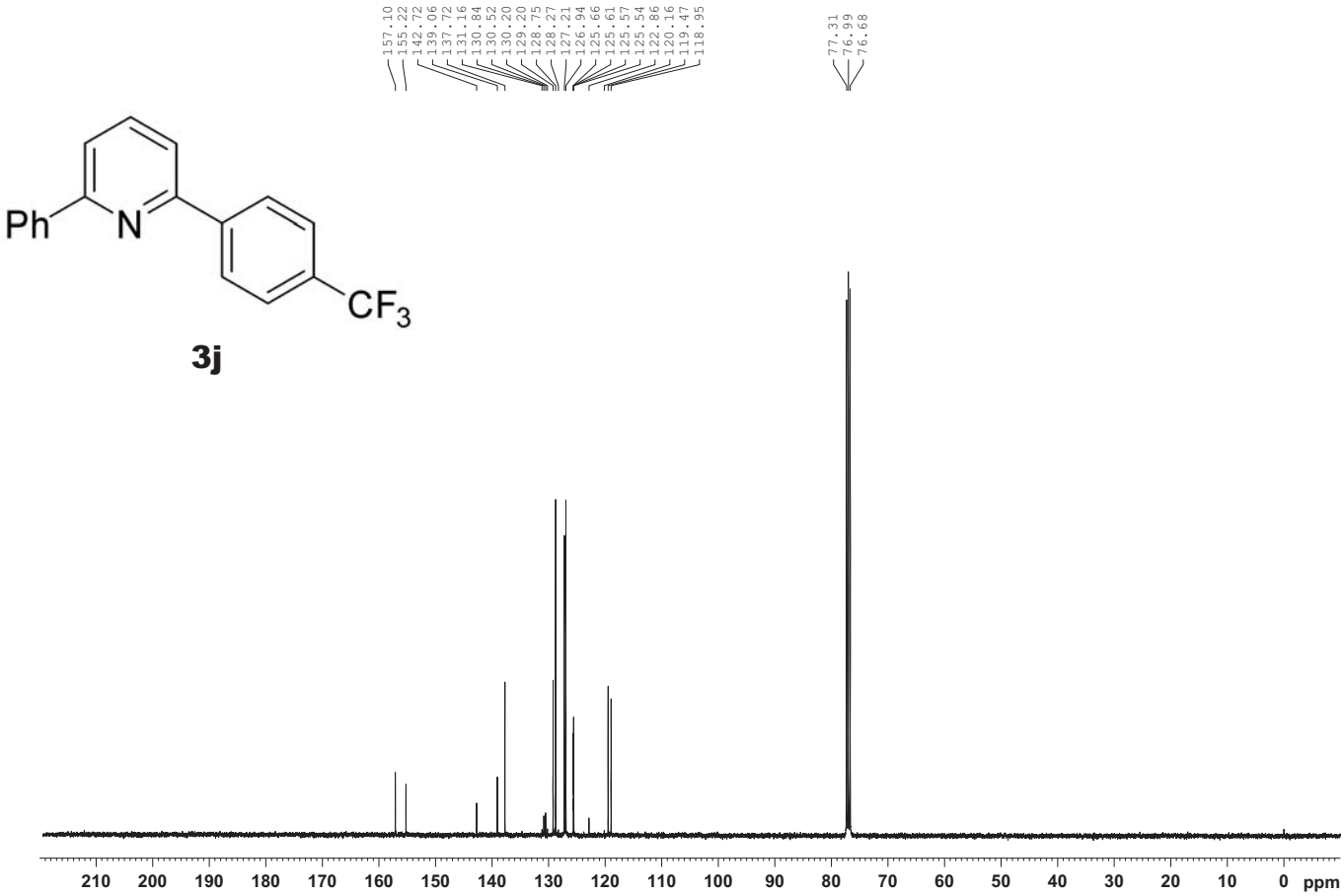
155.64
139.18
138.87
137.75
137.60
129.09
128.70
128.67
126.92
119.02
118.30
95.32
77.32
77.00
76.69
Ph
N
I
3i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.264
8.244
8.155
8.152
8.134
7.870
7.850
7.831
7.758
7.738
7.735
7.723
7.704
7.533
7.515
7.496
7.468
7.455
7.450
7.444
7.432
7.248
0.000
Ph
N
CF3
3j
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.01
2.00
1.02
4.01
2.02
1.02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142.72
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131.16
130.84
130.52
130.20
129.20
128.75
128.27
127.21
126.94
125.66
125.61
125.57
125.54
122.86
120.16
119.47
118.95
77.31
76.99
76.68
Ph
N
CF3
3j
210
200
190
180
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150
140
130
120
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100
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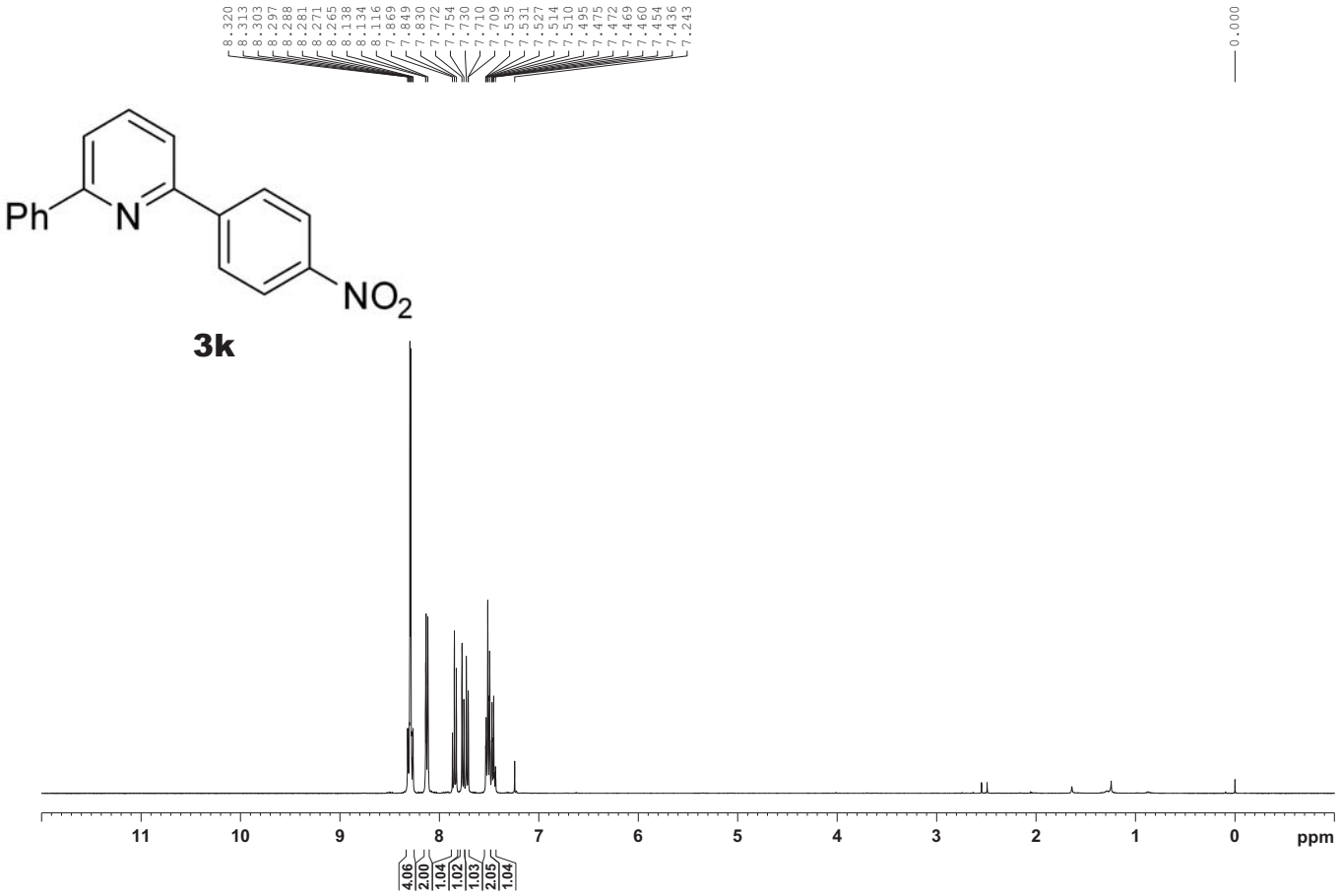
80
70
60
50
40
30
20
10
0
ppm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8.313
8.303
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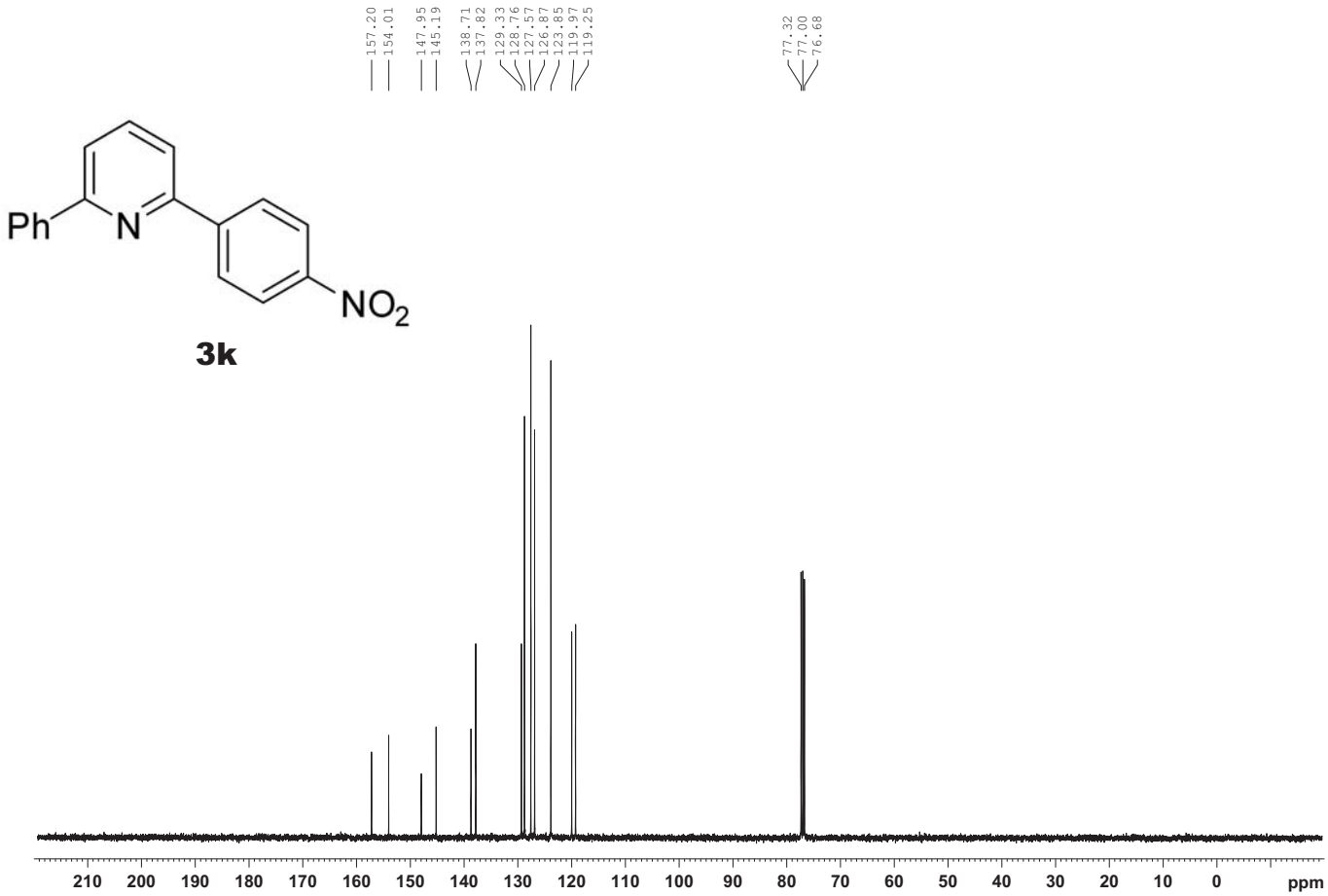
8.281
8.271
8.265
8.138
8.134
8.116
7.869
7.849
7.830
7.772
7.754
7.730
7.710
7.709
7.535
7.531
7.527
7.514
7.510
7.495
7.475
7.472
7.469
7.460
7.454
7.436
7.243
0.000
Ph
N
NO2
3k
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.06
2.00
1.04
1.02
1.03
2.05
1.04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154.01
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137.82
129.33
128.76
127.57
126.87
123.85
119.97
119.25
77.32
77.00
76.68
Ph
N
NO2
3k
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
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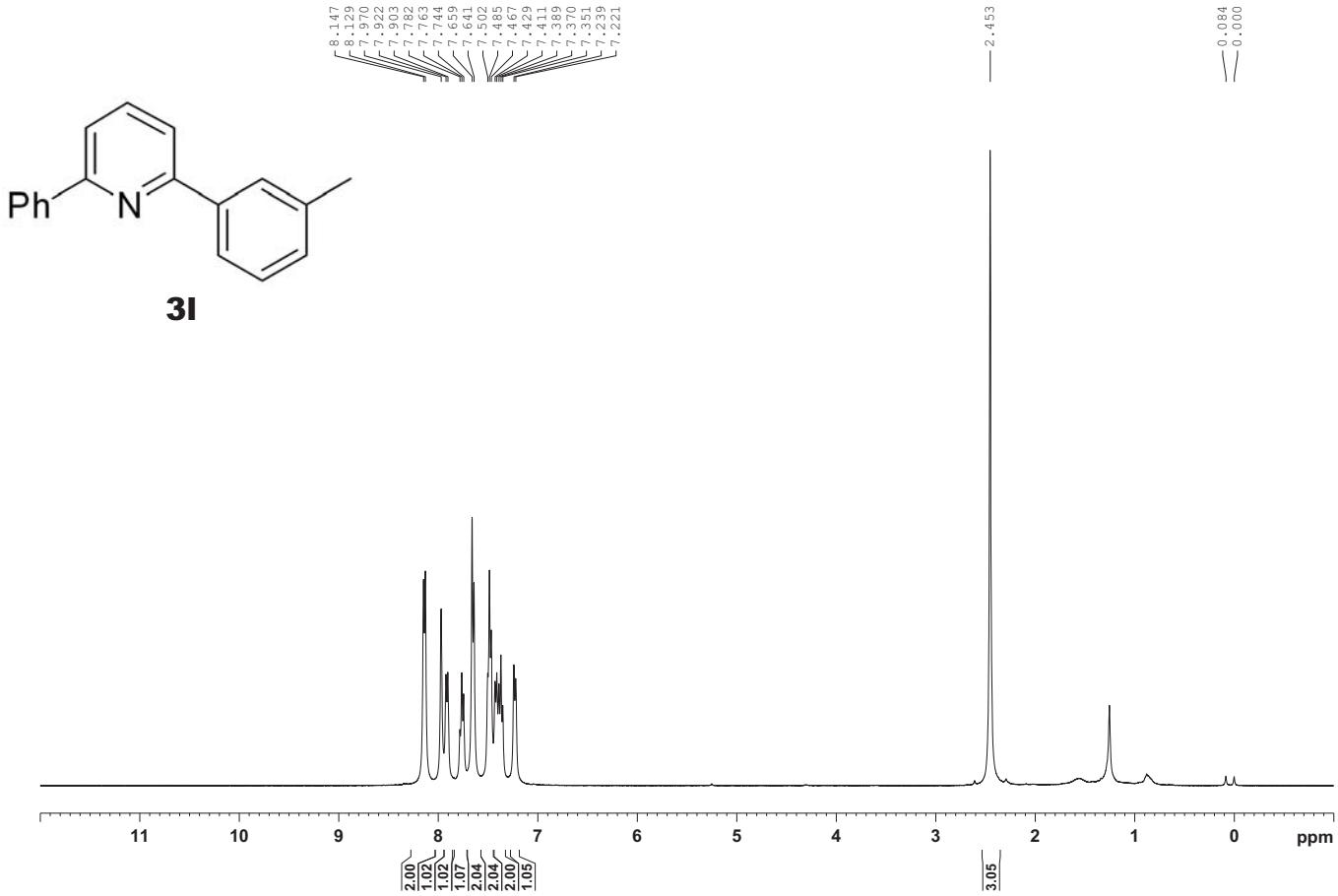
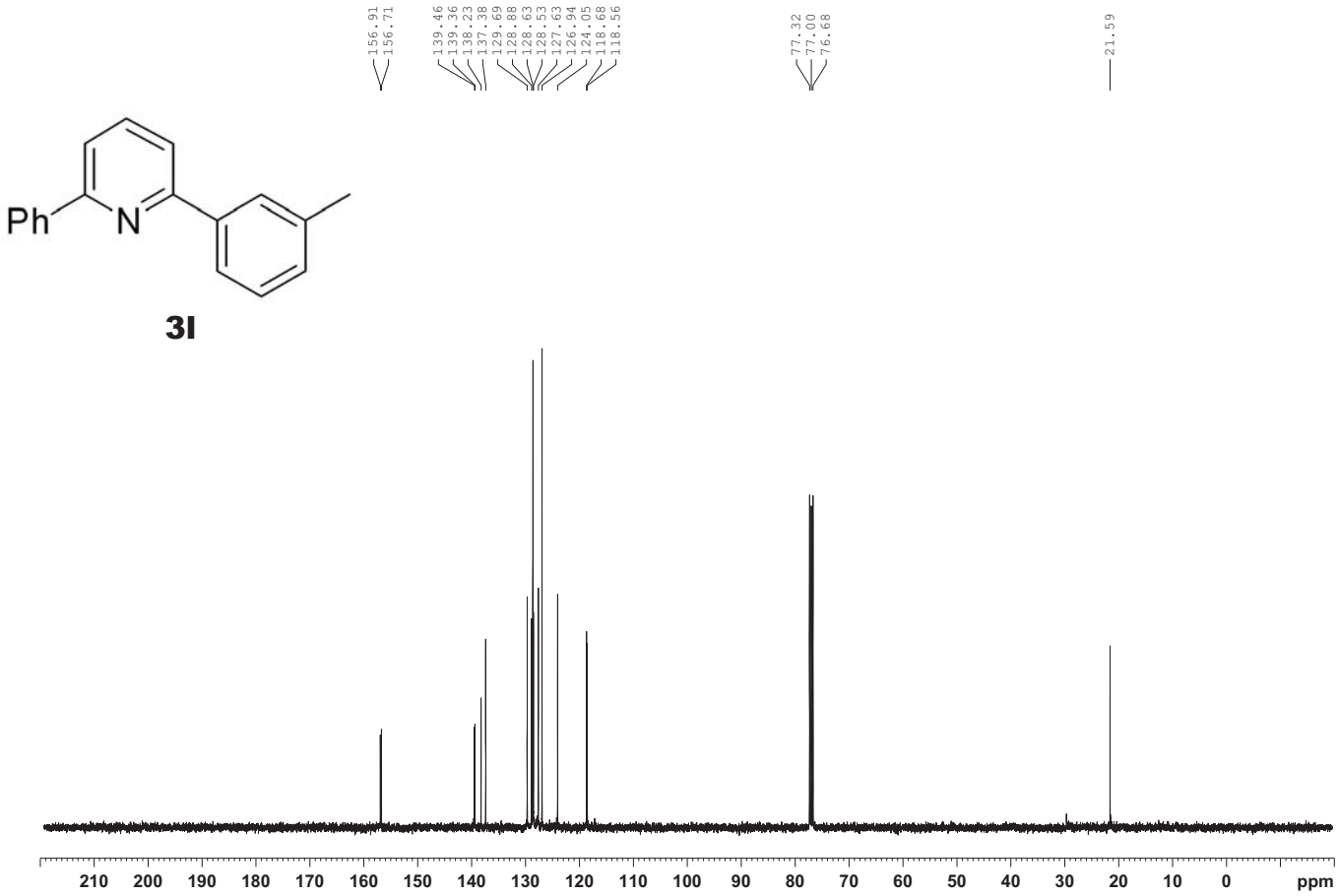
30
20
10
0
ppm


8.147
8.129
7.970
7.922
7.903
7.782
7.763
7.744
7.659
7.641
7.502
7.485
7.467
7.429
7.411
7.389
7.370
7.351
7.239
7.221
2.453
0.084
0.000
Ph
N
3l
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.00 1.02 1.02 1.07 2.04 2.04 2.00 1.05
3.05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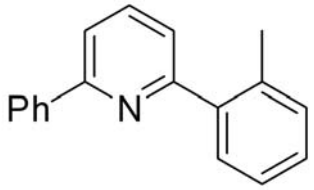
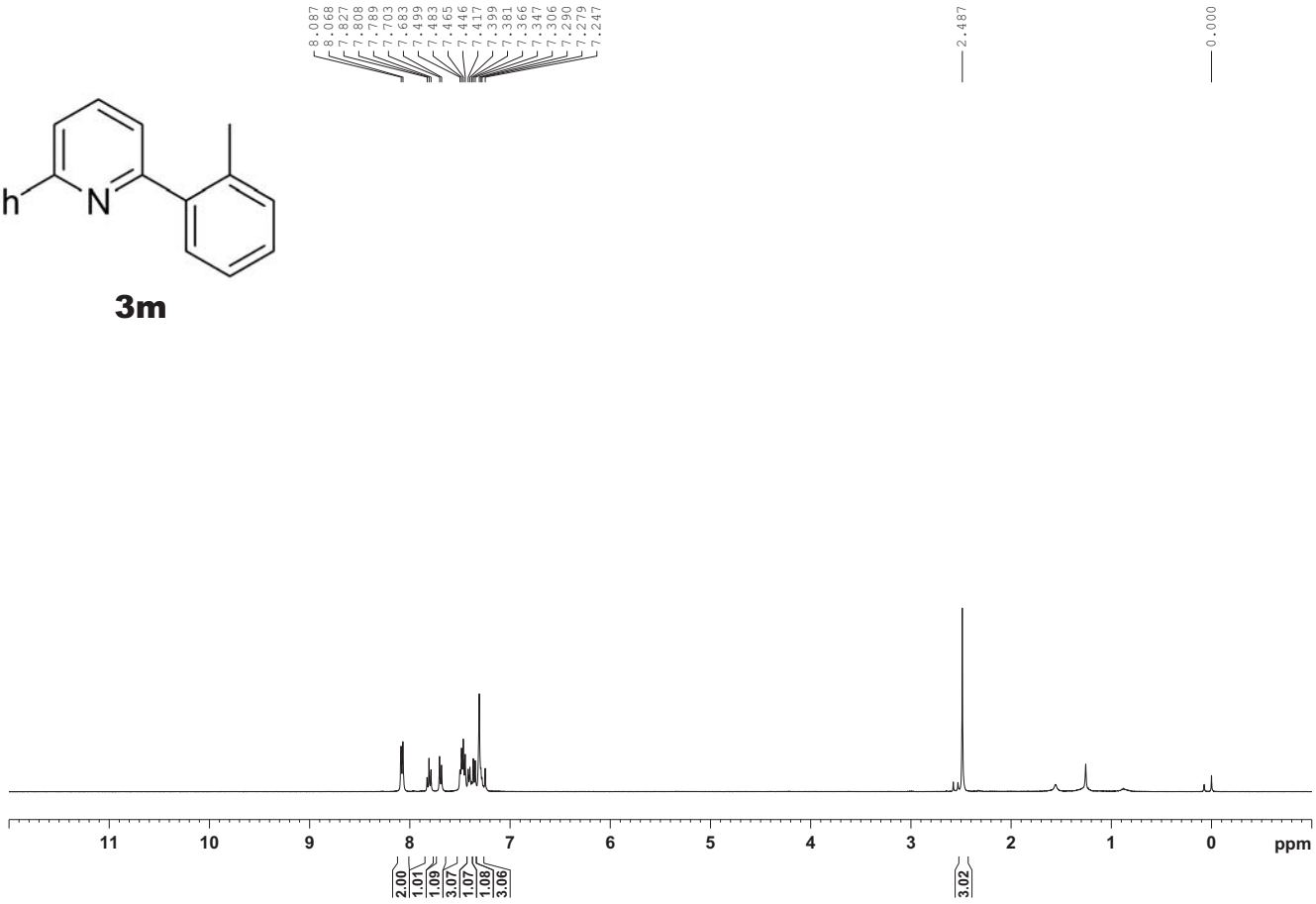
156.71
139.46
139.36
138.23
137.38
129.69
128.88
128.63
128.53
127.63
126.94
124.05
118.68
118.56
77.32
77.00
76.68
21.59
Ph
N
3l
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



3m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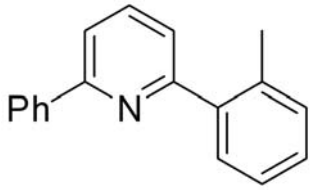
8.068
7.827
7.808
7.789
7.703
7.683
7.499
7.483
7.465
7.446
7.417
7.399
7.381
7.366
7.347
7.306
7.290
7.279
7.247
2.487
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.00
1.01
1.09
3.07
1.07
1.08
3.06
3.02



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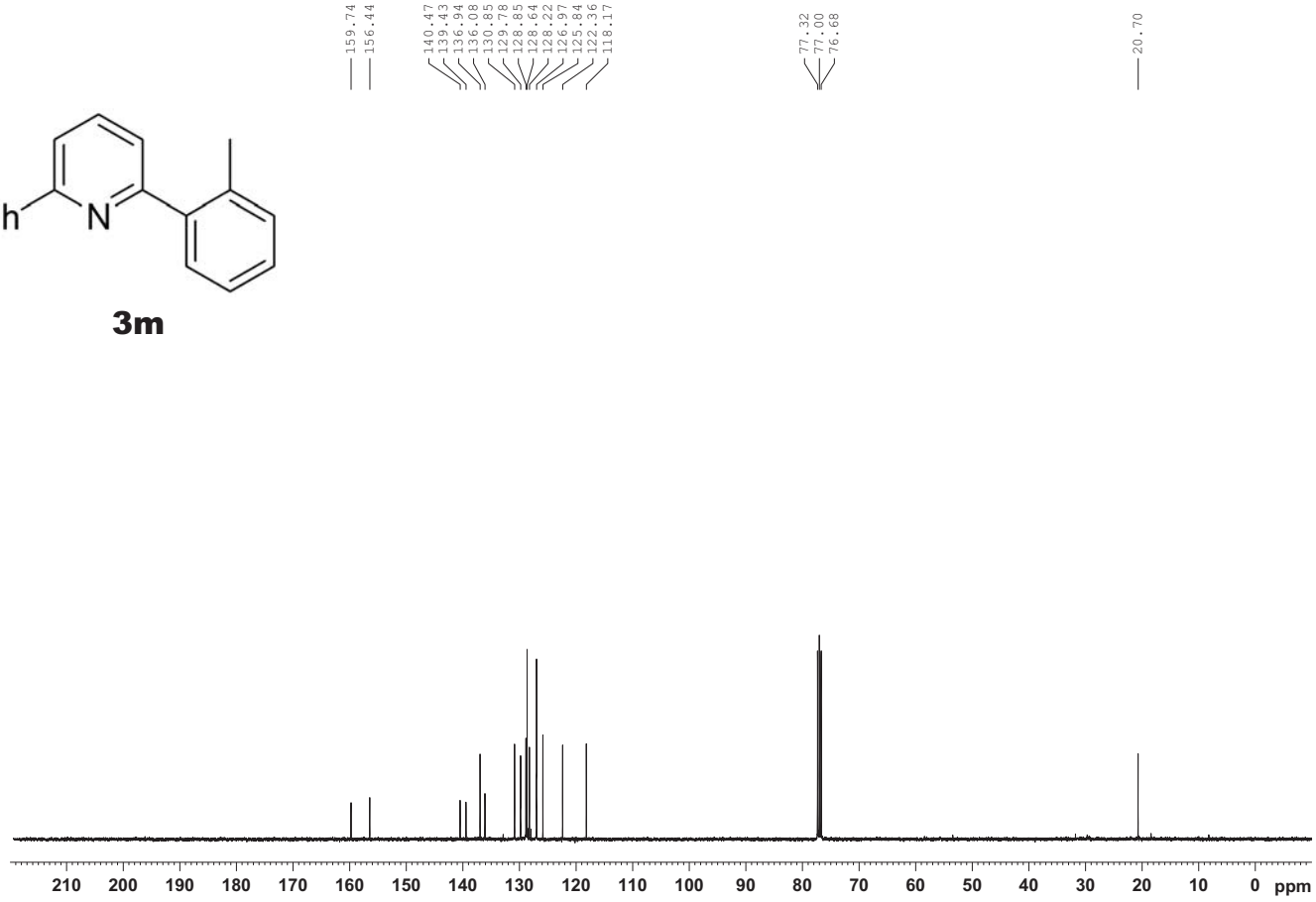
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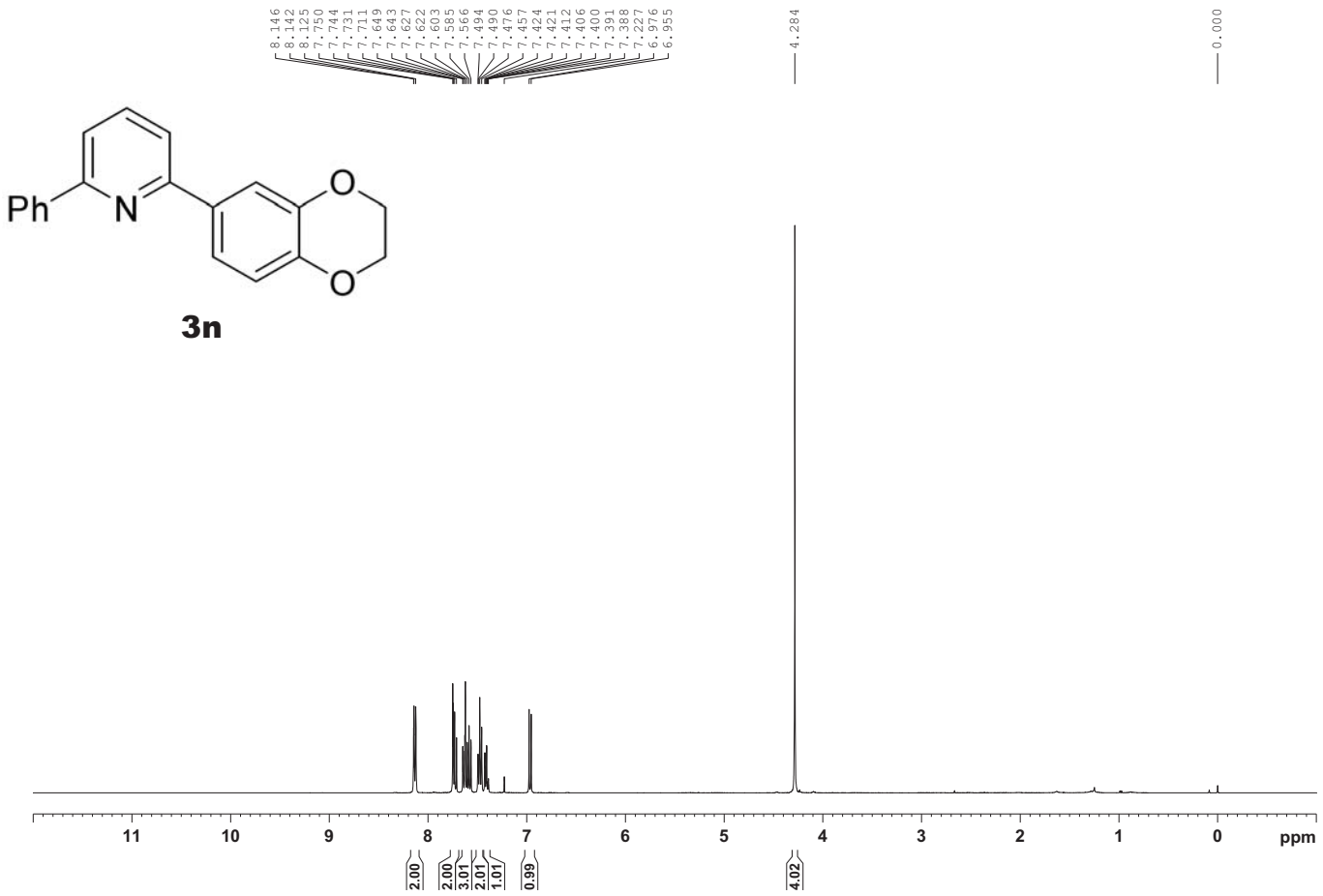
156.44
140.47
139.43
136.94
136.08
130.85
129.78
128.85
128.64
128.22
126.97
125.84
122.36
118.17
77.32
77.00
76.68
20.70
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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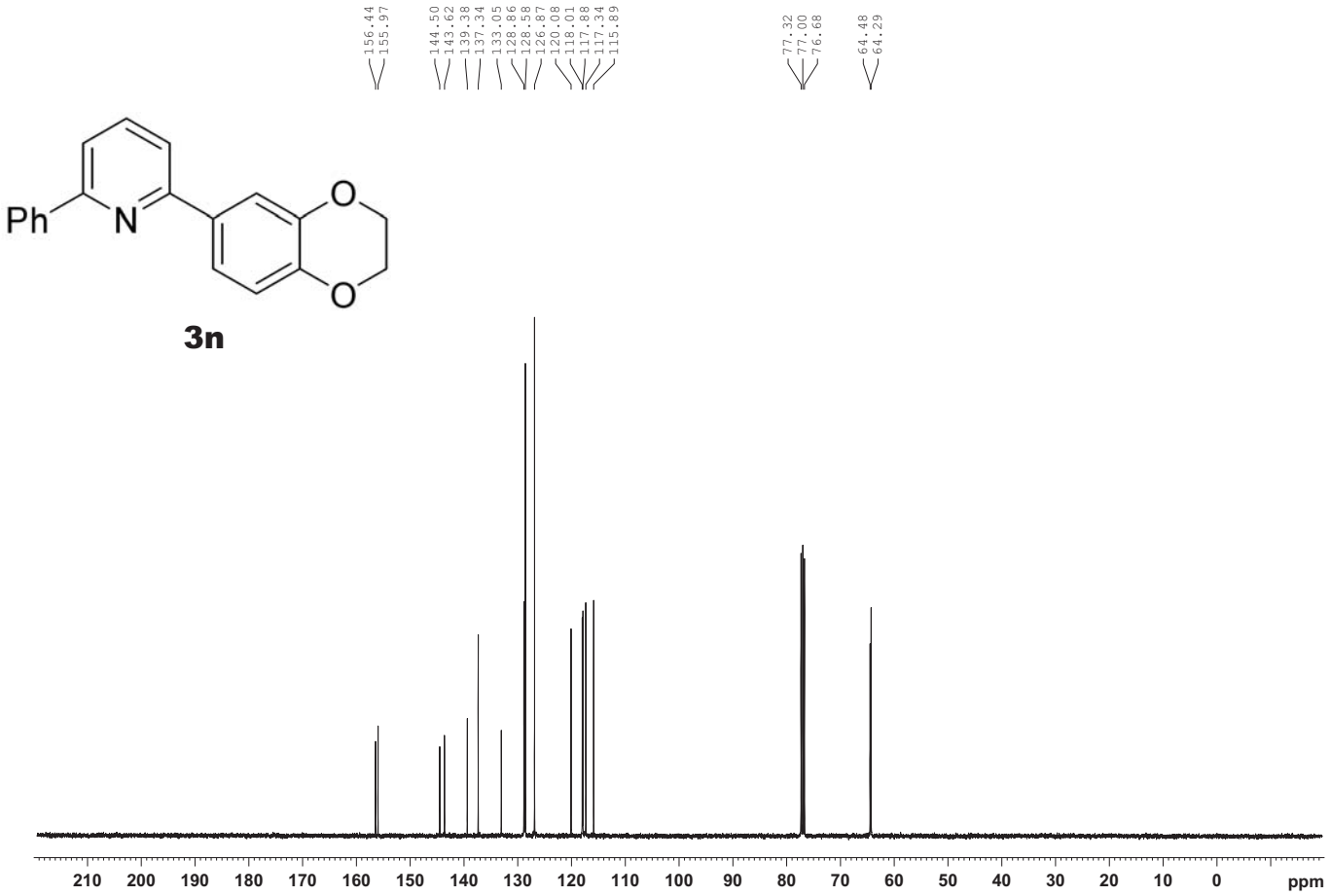
8.142
8.125
7.750
7.744
7.731
7.711
7.649
7.643
7.627
7.622
7.603
7.585
7.566
7.494
7.490
7.476
7.457
7.424
7.421
7.412
7.406
7.400
7.391
7.388
7.227
6.976
6.955
4.284
0.000
Ph
N
O
O
3n
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
2.00
3.01
2.01
1.01
0.99
4.02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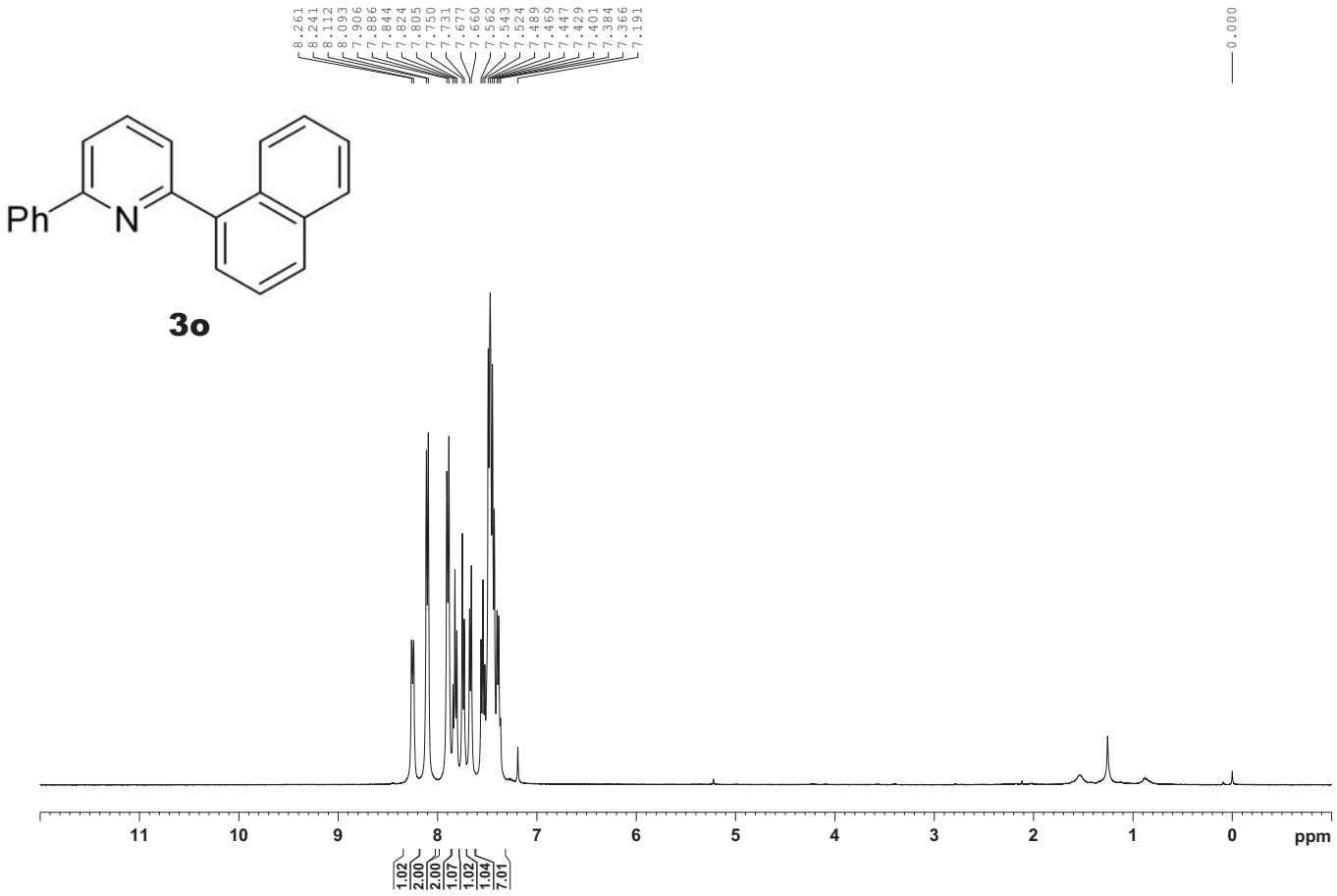
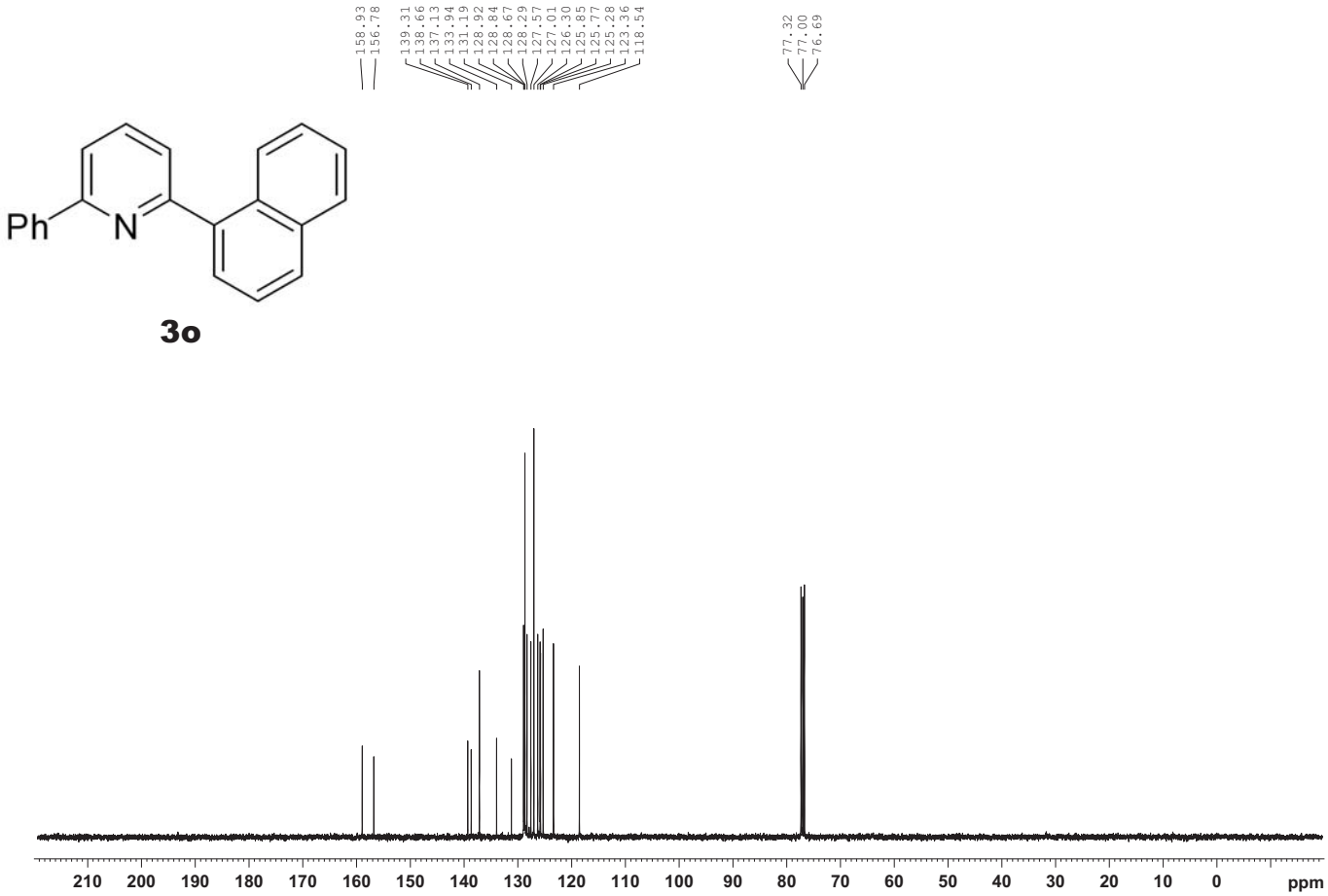
155.97
144.50
143.62
139.38
137.34
133.05
128.86
128.58
126.87
120.08
118.01
117.88
117.34
115.89
77.32
77.00
76.68
64.48
64.29
Ph
N
O
O
3n
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.261
8.241
8.112
8.093
7.906
7.886
7.844
7.824
7.805
7.750
7.731
7.677
7.660
7.562
7.543
7.524
7.489
7.469
7.447
7.429
7.401
7.384
7.366
7.191
0.000
Ph
N
3o
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.02
2.00
2.00
1.07
1.02
1.04
7.01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138.66
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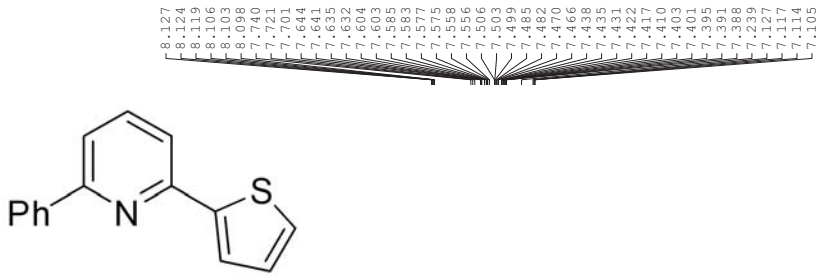
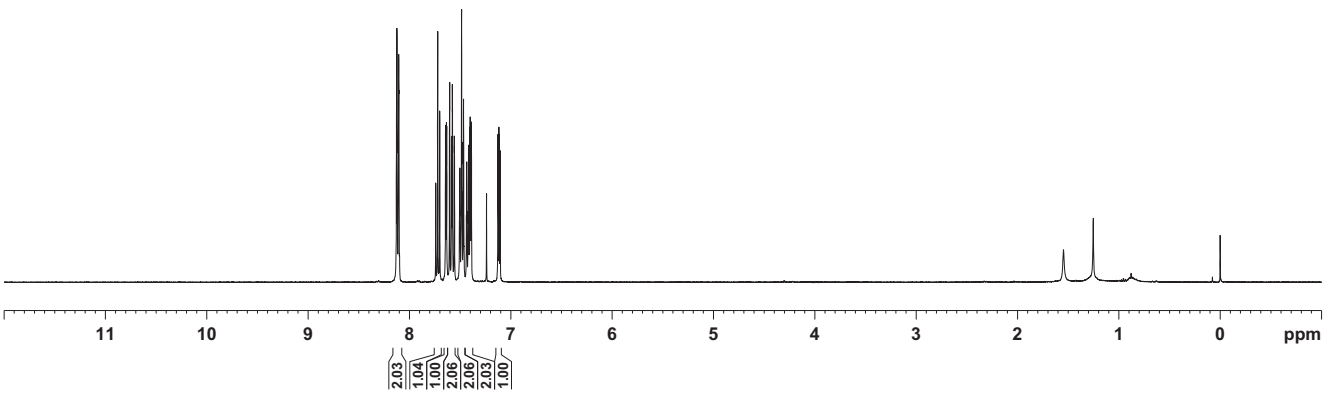
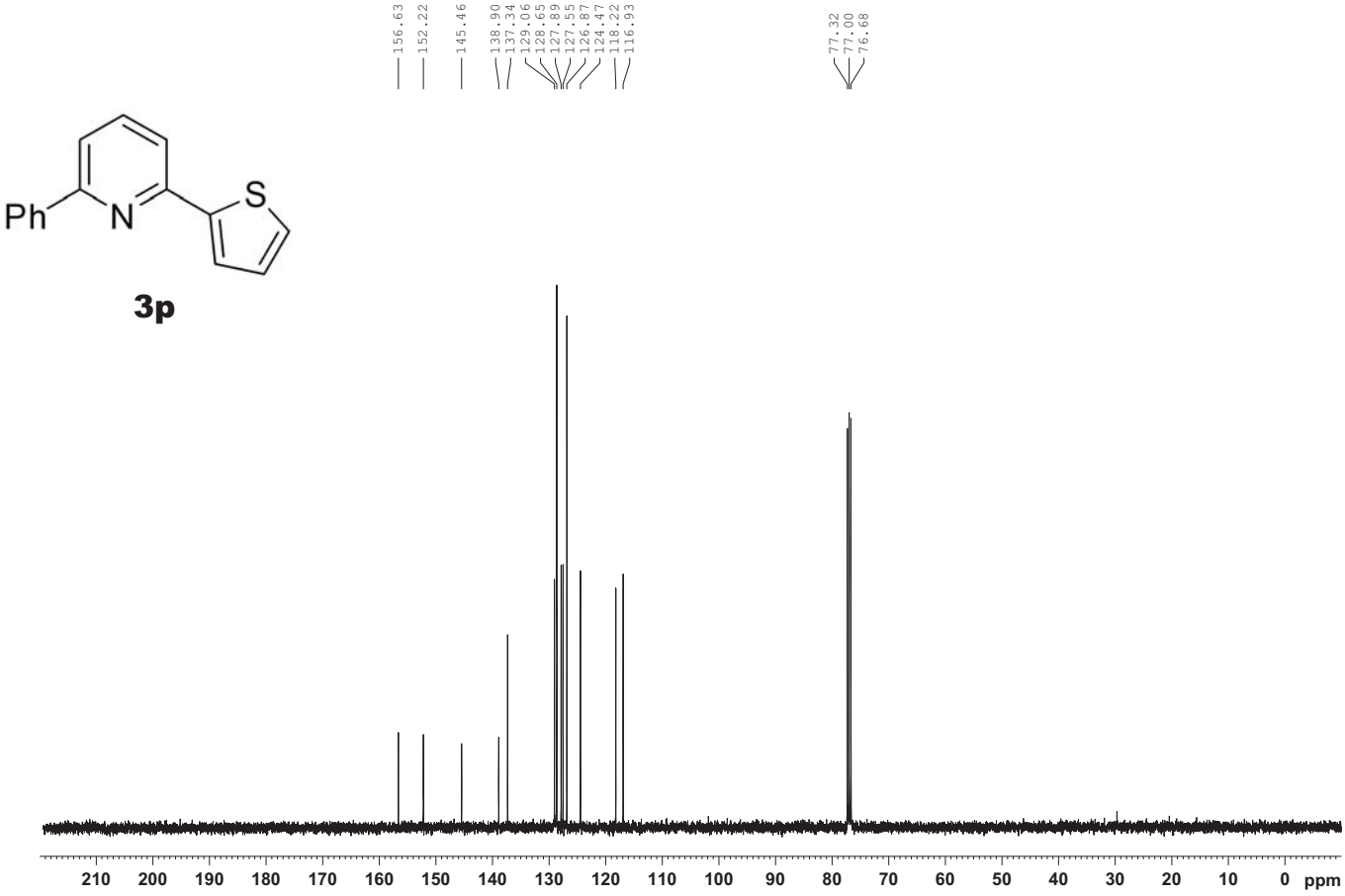
133.94
131.19
128.92
128.84
128.67
128.29
127.57
127.01
126.30
125.85
125.77
125.28
123.36
118.54
77.32
77.00
76.69
Ph
N
3o
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

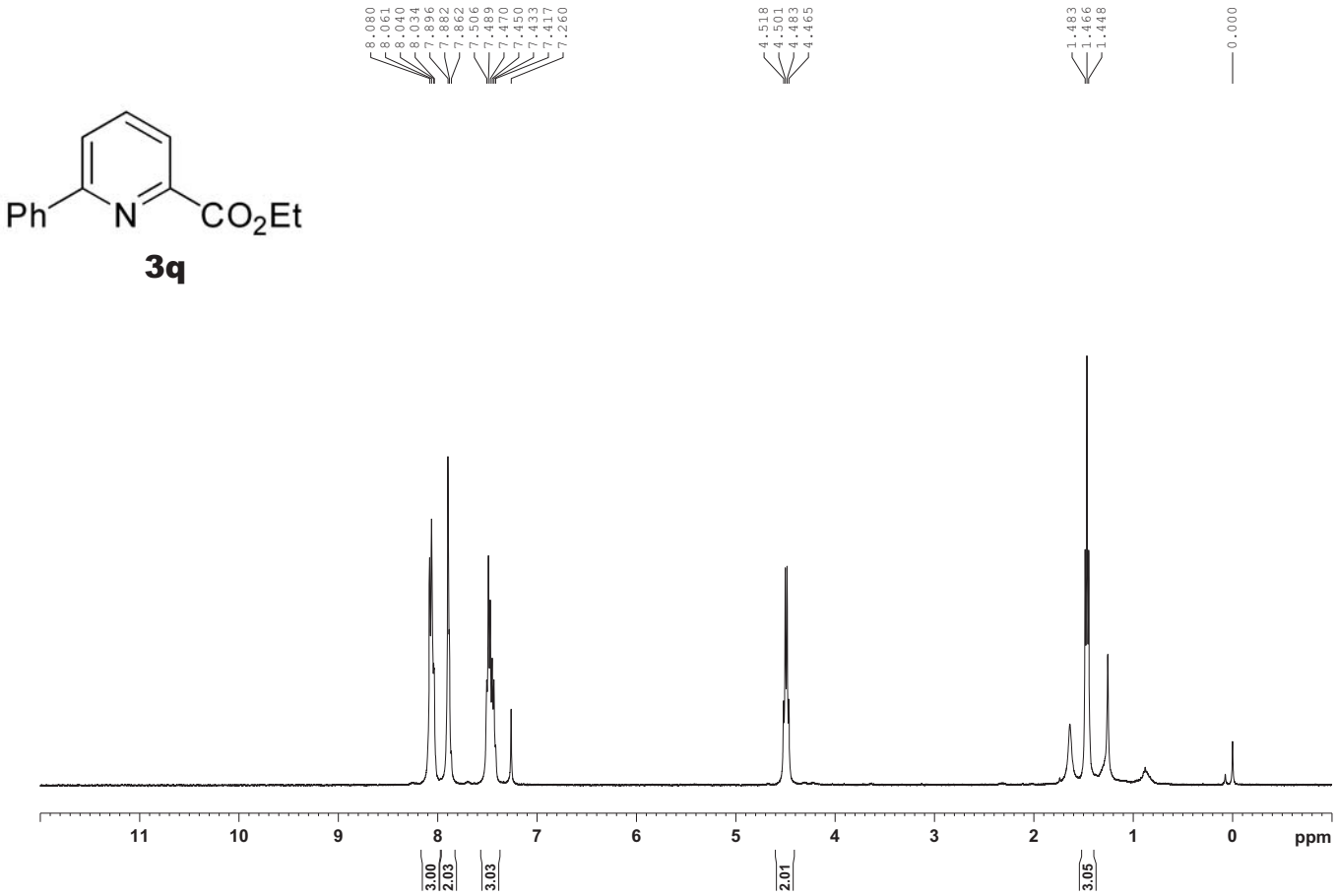

8.127
8.124
8.119
8.106
8.103
8.098
7.740
7.721
7.701
7.644
7.641
7.635
7.632
7.604
7.603
7.585
7.583
7.577
7.575
7.558
7.556
7.506
7.503
7.499
7.485
7.482
7.470
7.466
7.438
7.435
7.431
7.422
7.417
7.410
7.403
7.401
7.395
7.391
7.388
7.239
7.127
7.117
7.114
7.105
0.000
Ph
N
S

3p


11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.03
1.04
1.00
2.06
2.06
2.03
1.00


156.63
152.22
145.46
138.90
137.34
129.06
128.65
127.89
127.55
126.87
124.47
118.22
116.93
77.32
77.00
76.68
Ph
N
S
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3p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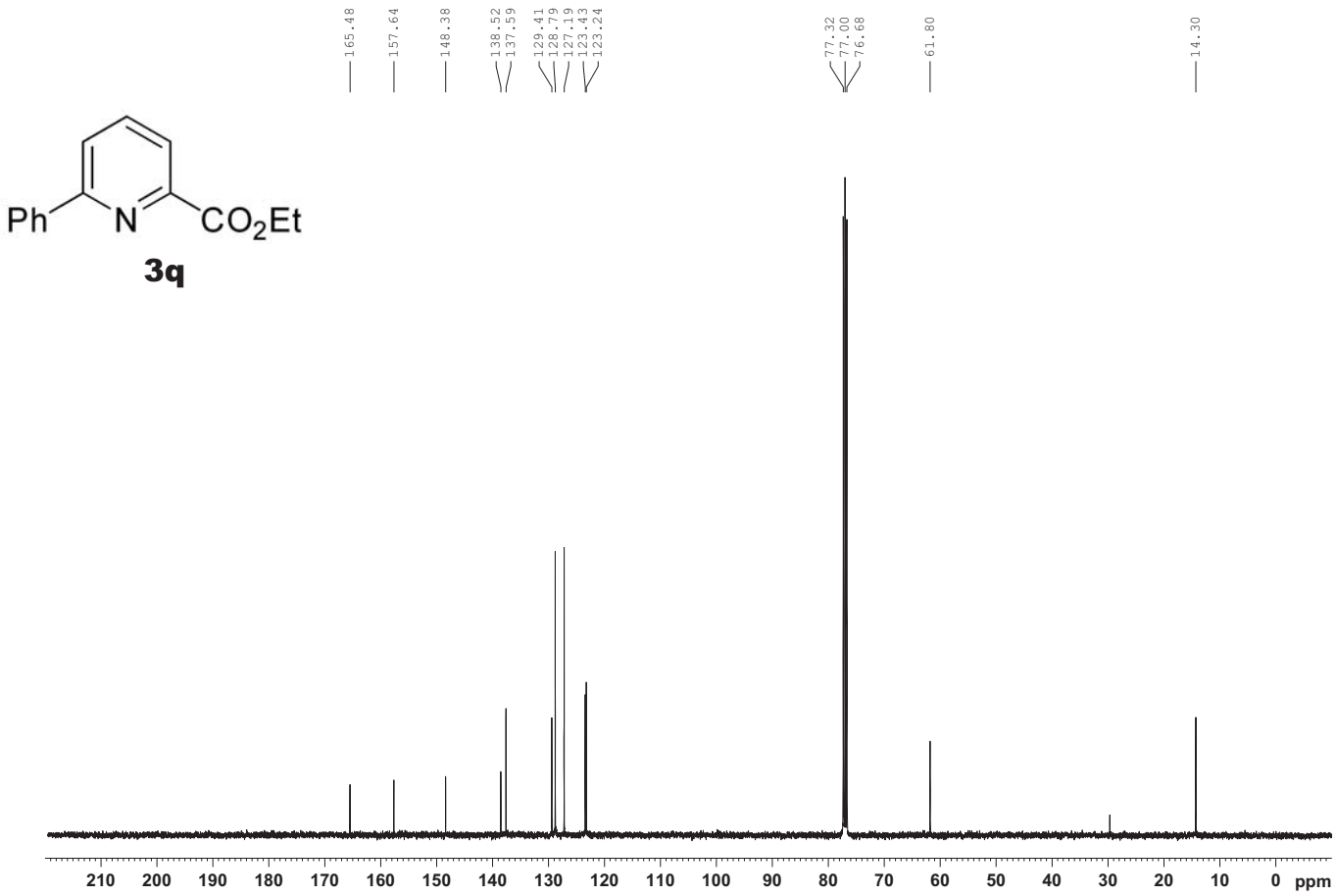
8.040
8.034
7.896
7.882
7.862
7.506
7.489
7.470
7.450
7.433
7.417
7.260
4.518
4.501
4.483
4.465
1.483
1.466
1.448
0.000
Ph
N
CO2Et
3q
11
10
9
8
7
6
5
4
3
2
1
0
ppm
3.00
2.03
3.03
2.01
3.05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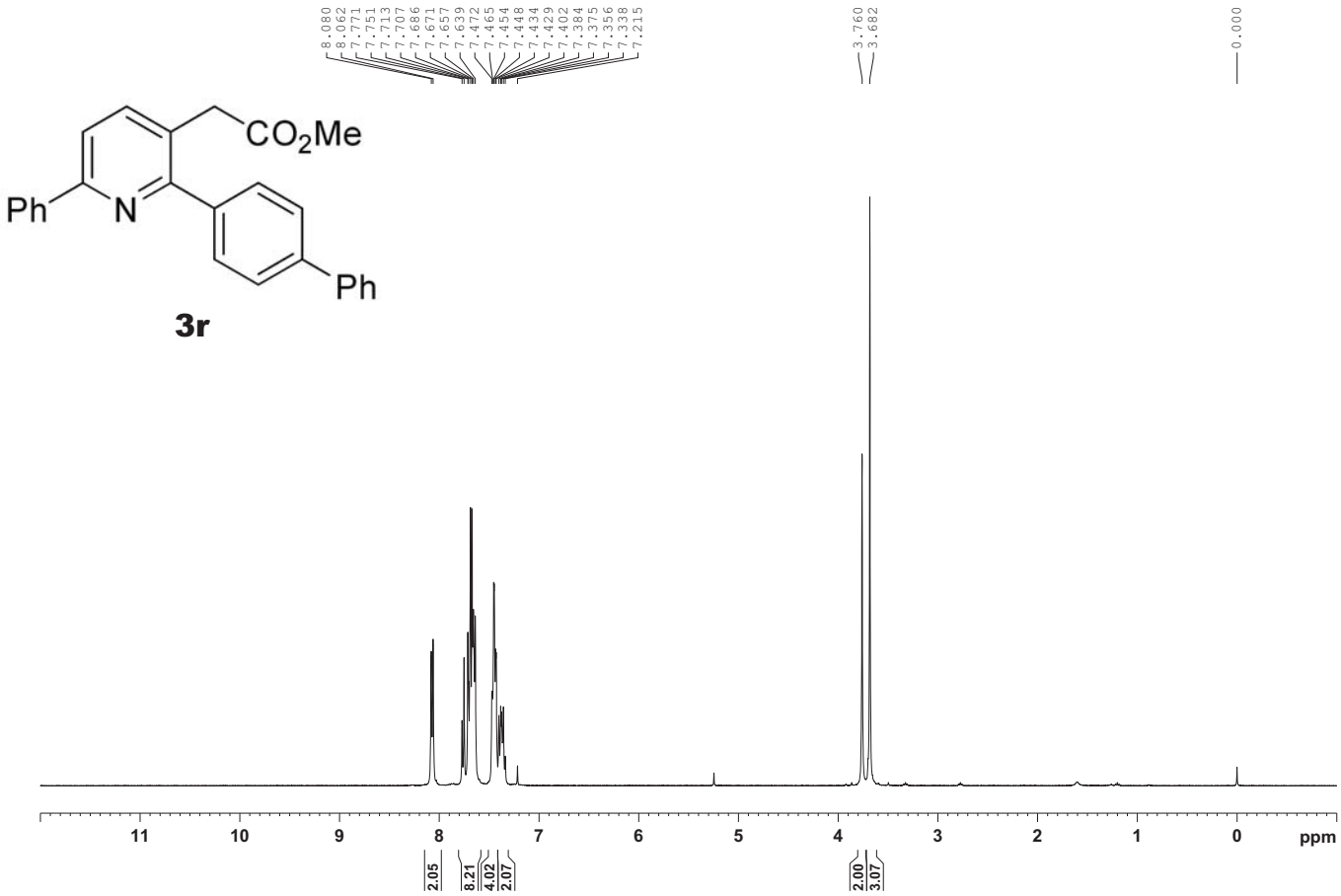
148.38
138.52
137.59
129.41
128.79
127.19
123.43
123.24
77.32
77.00
76.68
61.80
14.30
Ph
N
CO2Et
3q
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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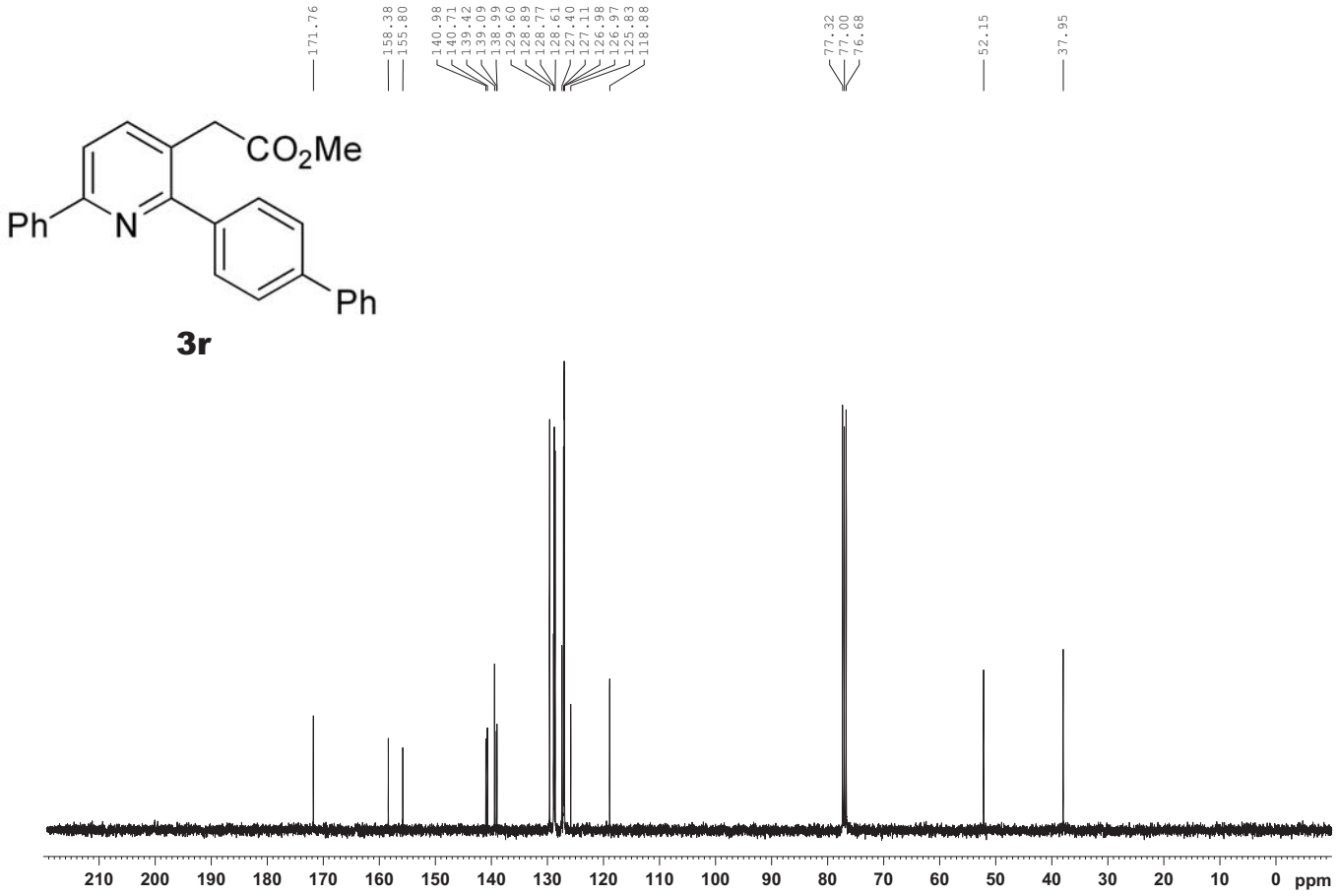
8.062
7.771
7.751
7.713
7.707
7.686
7.671
7.657
7.639
7.472
7.465
7.454
7.448
7.434
7.429
7.402
7.384
7.375
7.356
7.338
7.215
3.760
3.682
0.000
CO2Me
Ph
N
Ph
3r
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.05
8.21
4.02
2.07
2.00
3.07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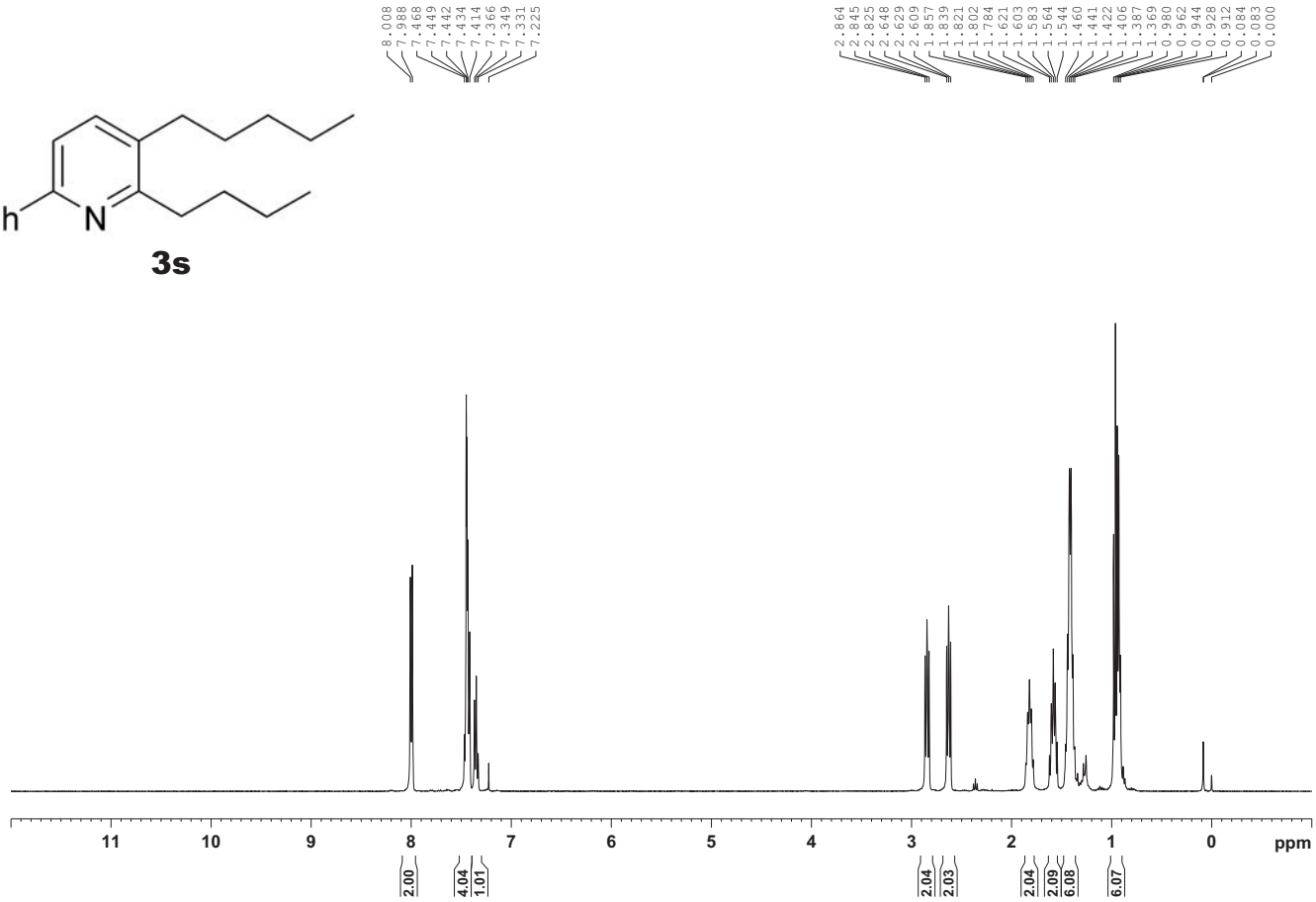
158.38
155.80
140.98
140.71
139.42
139.09
138.99
129.60
128.89
128.77
128.61
127.40
127.11
126.98
126.97
125.83
118.88
77.32
77.00
76.68
52.15
37.95
CO2Me
Ph
N
Ph
3r
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

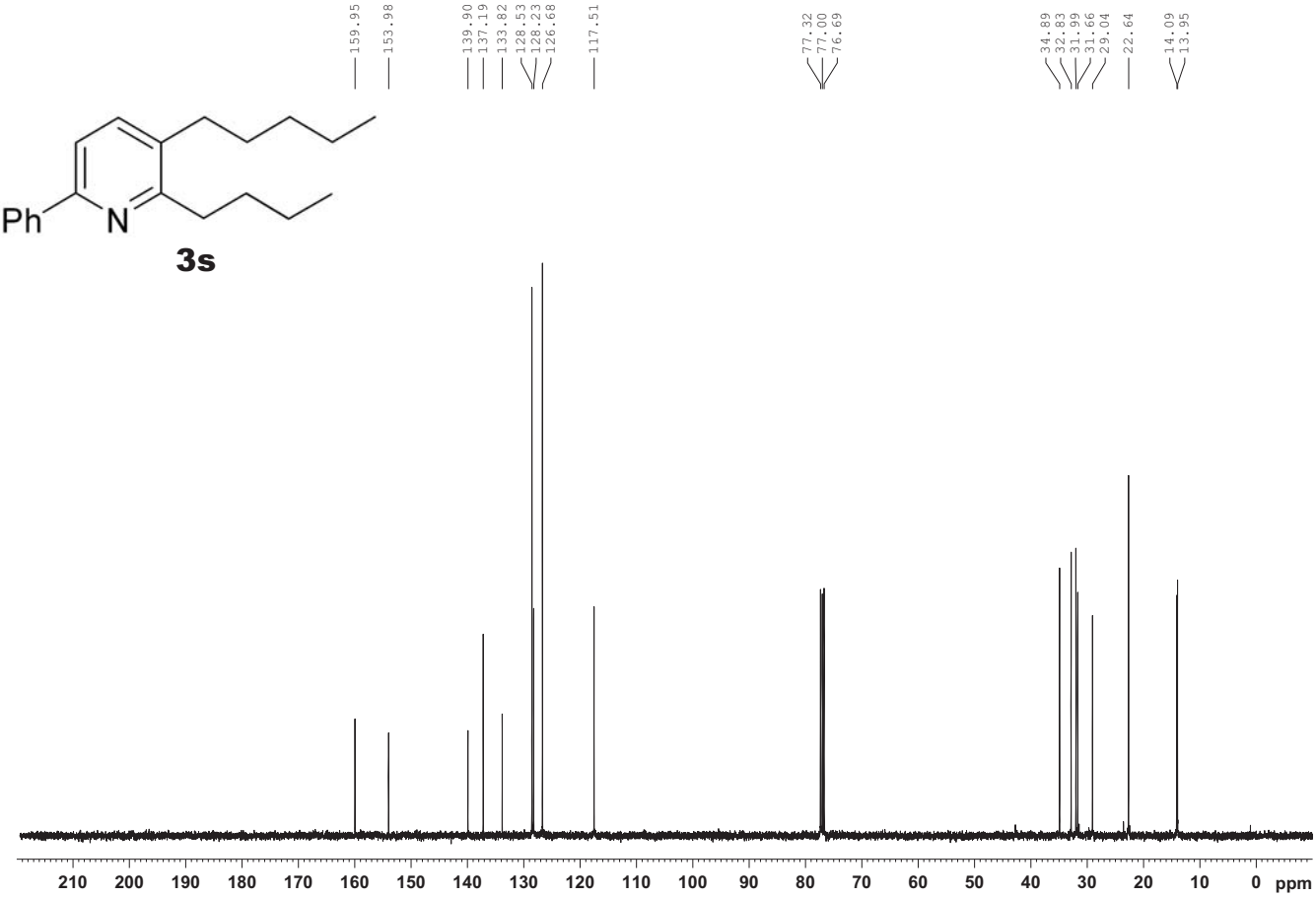
Ph
N

3s


8.008
7.988
7.468
7.449
7.442
7.434
7.414
7.366
7.349
7.331
7.225
2.864
2.845
2.825
2.648
2.629
2.609
1.857
1.839
1.821
1.802
1.784
1.621
1.603
1.583
1.564
1.544
1.460
1.441
1.422
1.406
1.387
1.369
0.980
0.962
0.944
0.928
0.912
0.084
0.083
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.00
4.04
1.01
2.04
2.03
2.04
2.09
6.08
6.07

Ph
N

3s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153.98
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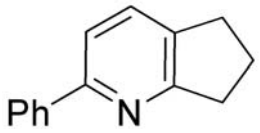
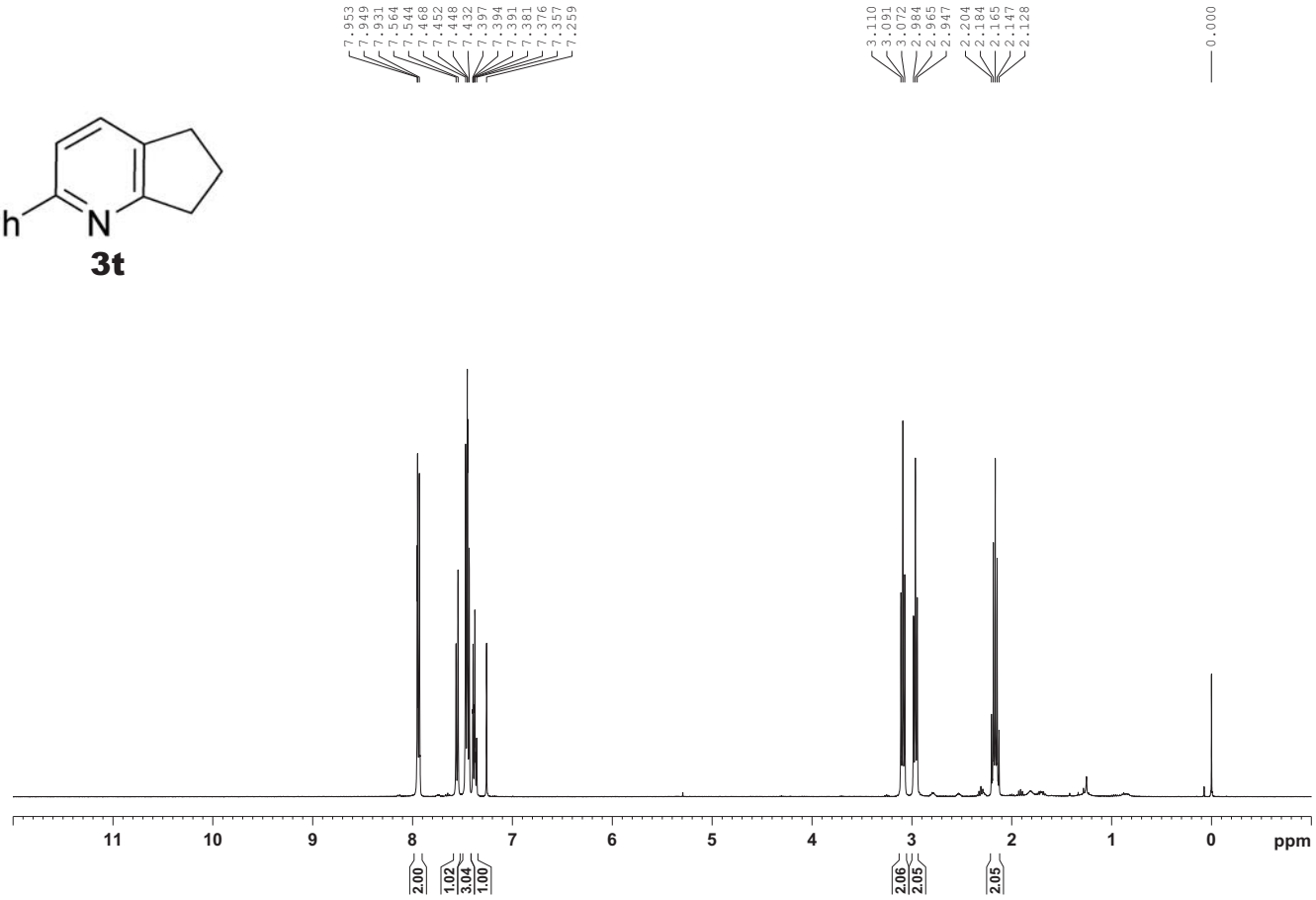
137.19
133.82
128.53
128.23
126.68
117.51
77.32
77.00
76.69
34.89
32.83
31.99
31.66
29.04
22.64
14.09
13.95
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



3t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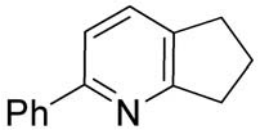
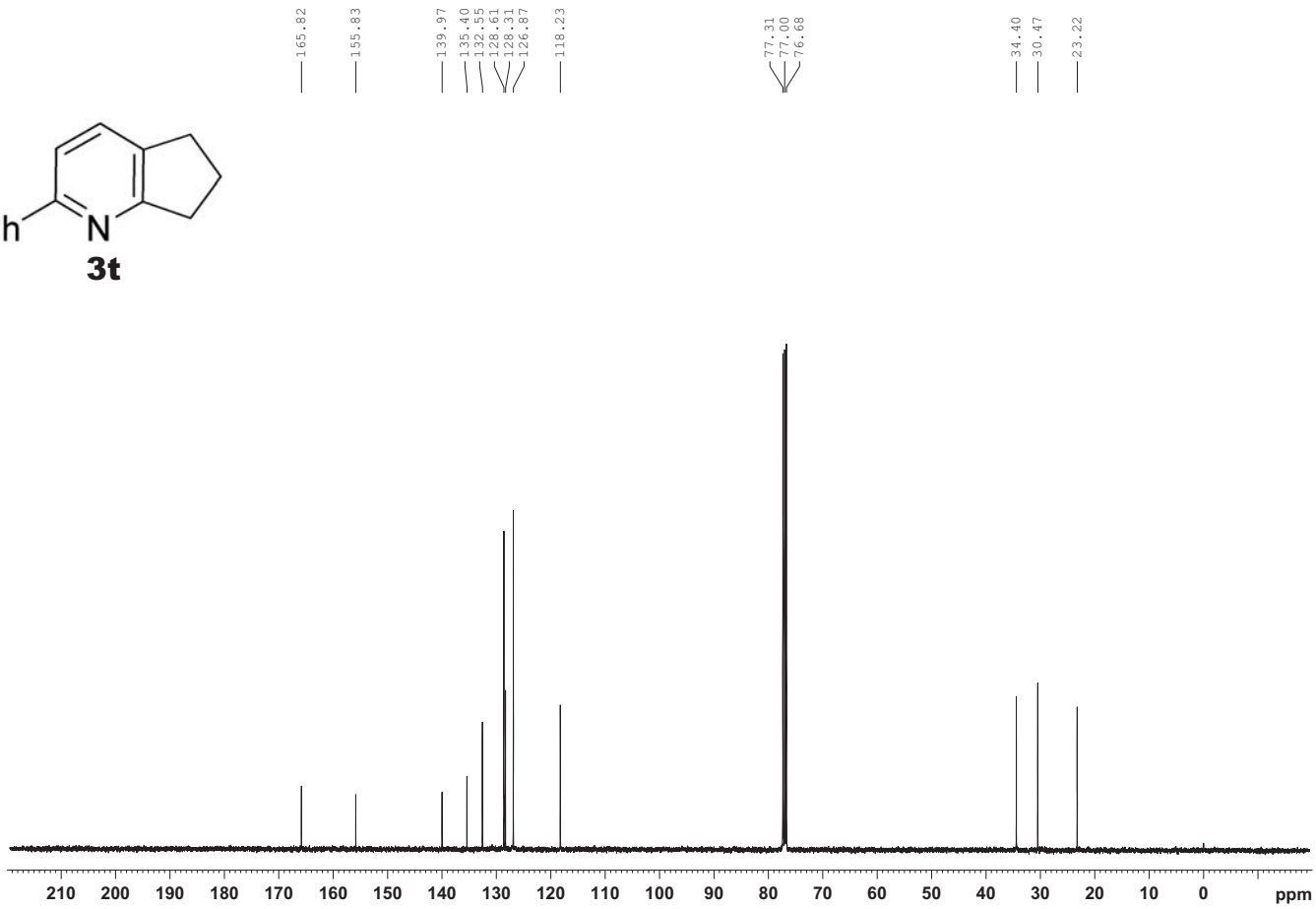
7.949
7.931
7.564
7.544
7.468
7.452
7.448
7.432
7.397
7.394
7.391
7.381
7.376
7.357
7.259
3.110
3.091
3.072
2.984
2.965
2.947
2.204
2.184
2.165
2.147
2.128
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.00
1.02
3.04
1.00
2.06
2.05
2.05



3t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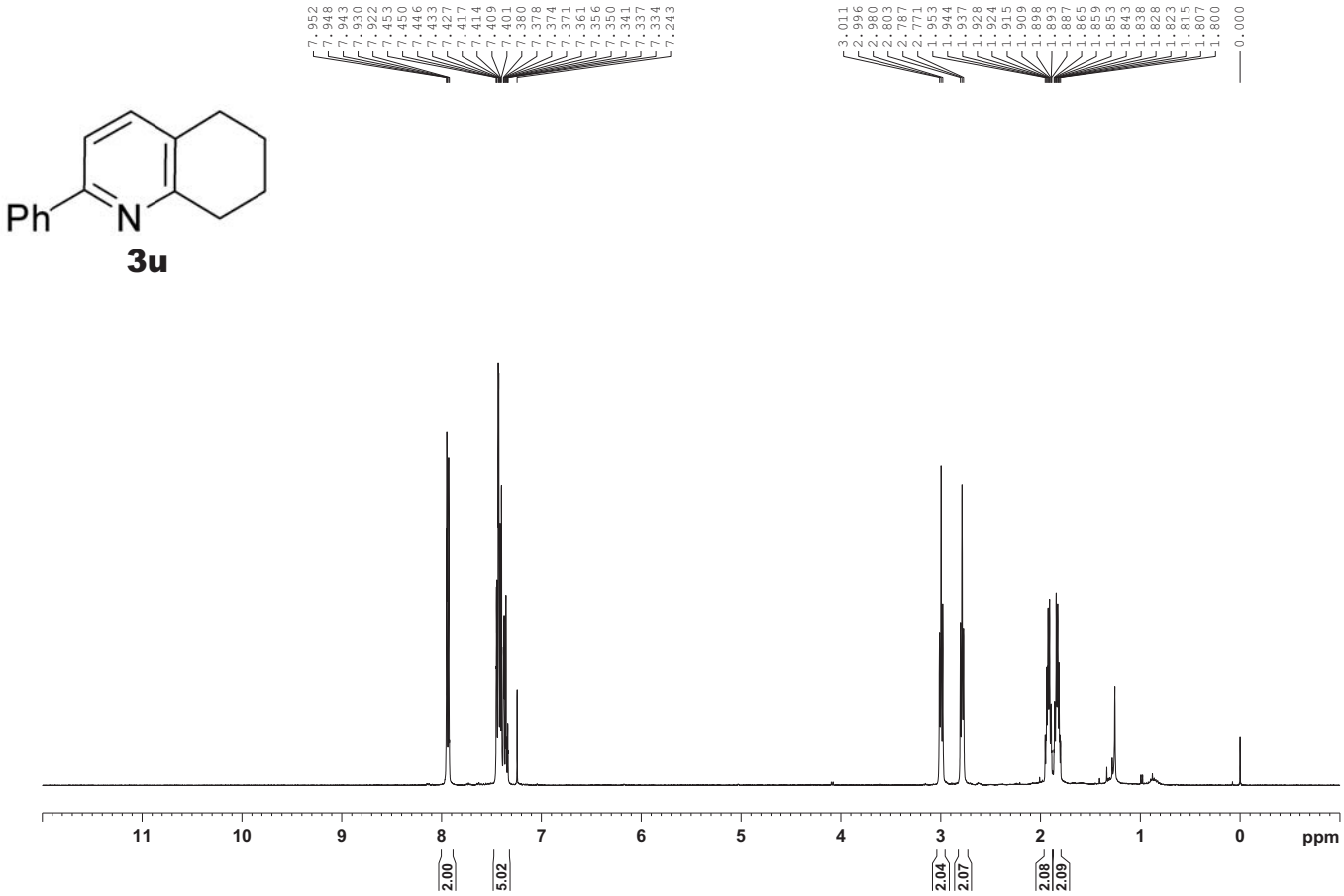
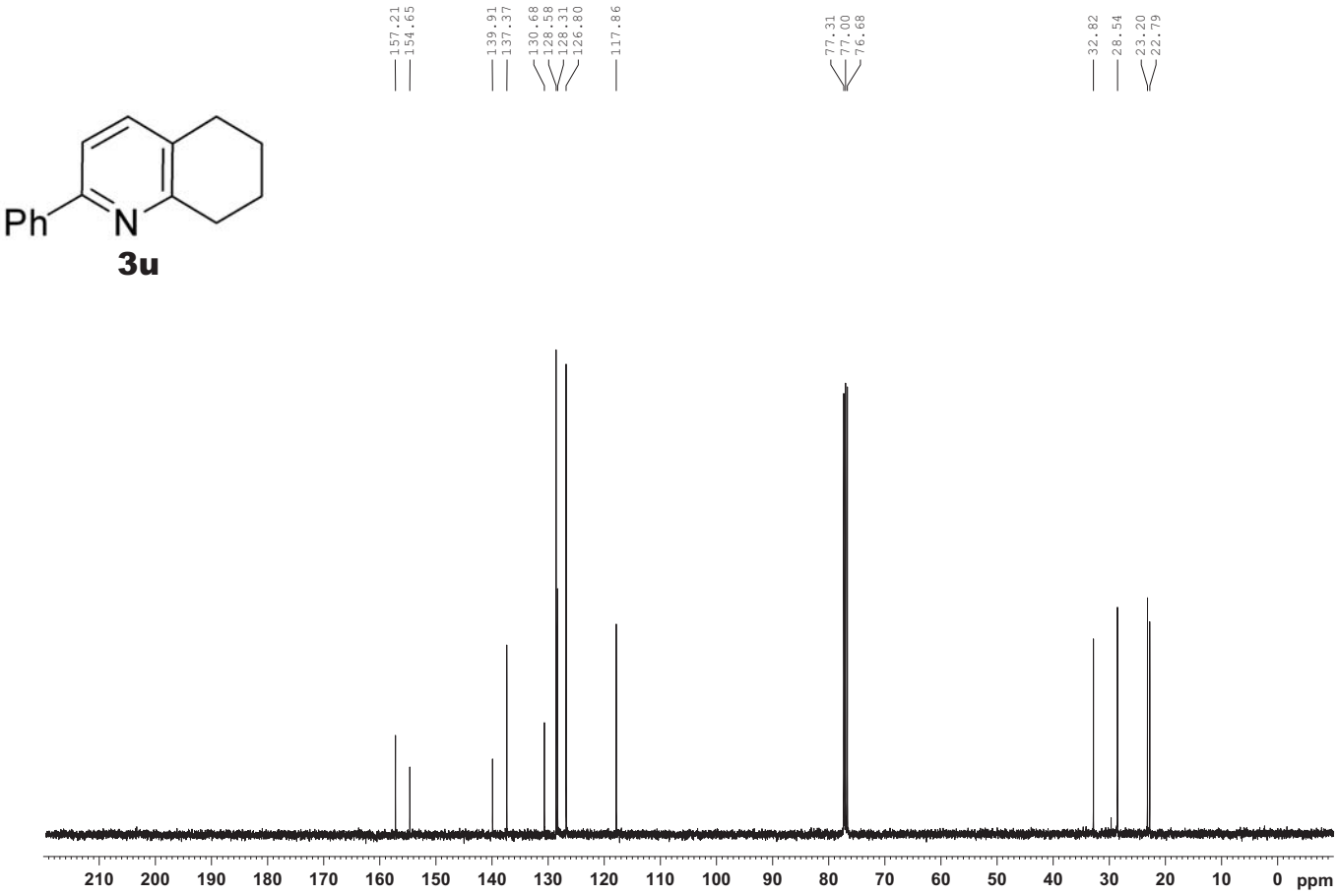
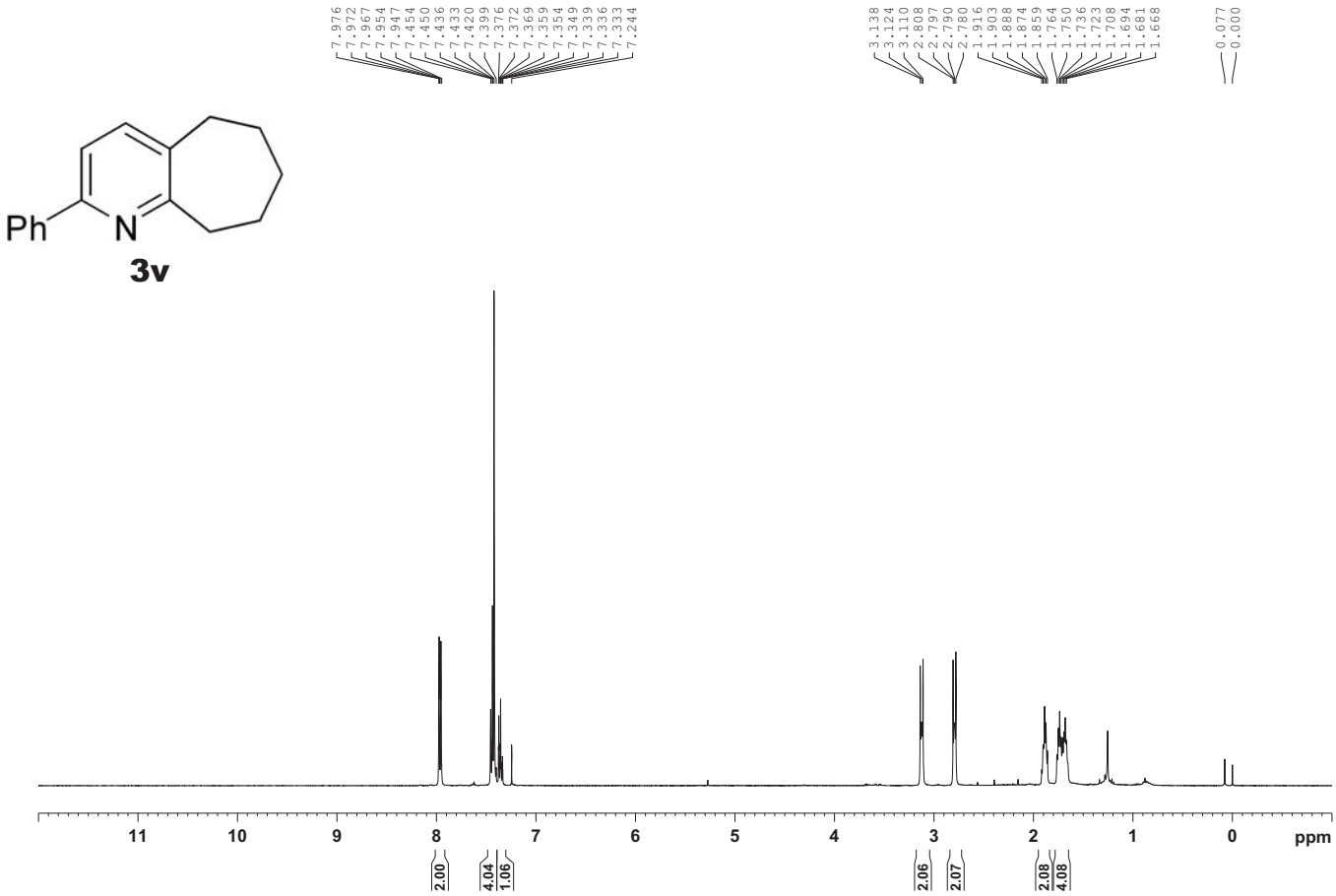
155.83
139.97
135.40
132.55
128.61
128.31
126.87
118.23
77.31
77.00
76.68
34.40
30.47
23.22
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.952
7.948
7.943
7.930
7.922
7.453
7.450
7.446
7.433
7.427
7.417
7.414
7.409
7.401
7.380
7.378
7.374
7.371
7.361
7.356
7.350
7.341
7.337
7.334
7.243
3.011
2.996
2.980
2.803
2.787
2.771
1.953
1.944
1.937
1.928
1.924
1.915
1.909
1.898
1.893
1.887
1.865
1.859
1.853
1.843
1.838
1.828
1.823
1.815
1.807
1.800
0.000
Ph
N
3u
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
5.02
2.04
2.07
2.08
2.09


157.21
154.65
139.91
137.37
130.68
128.58
128.31
126.80
117.86
77.31
77.00
76.68
32.82
28.54
23.20
22.79
Ph
N
3u
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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7.349
7.339
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7.333
7.244
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3.124
3.110
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2.797
2.790
2.780
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1.903
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1.874
1.859
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1.723
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1.694
1.681
1.668
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0.000
Ph
N
3v
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10
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8
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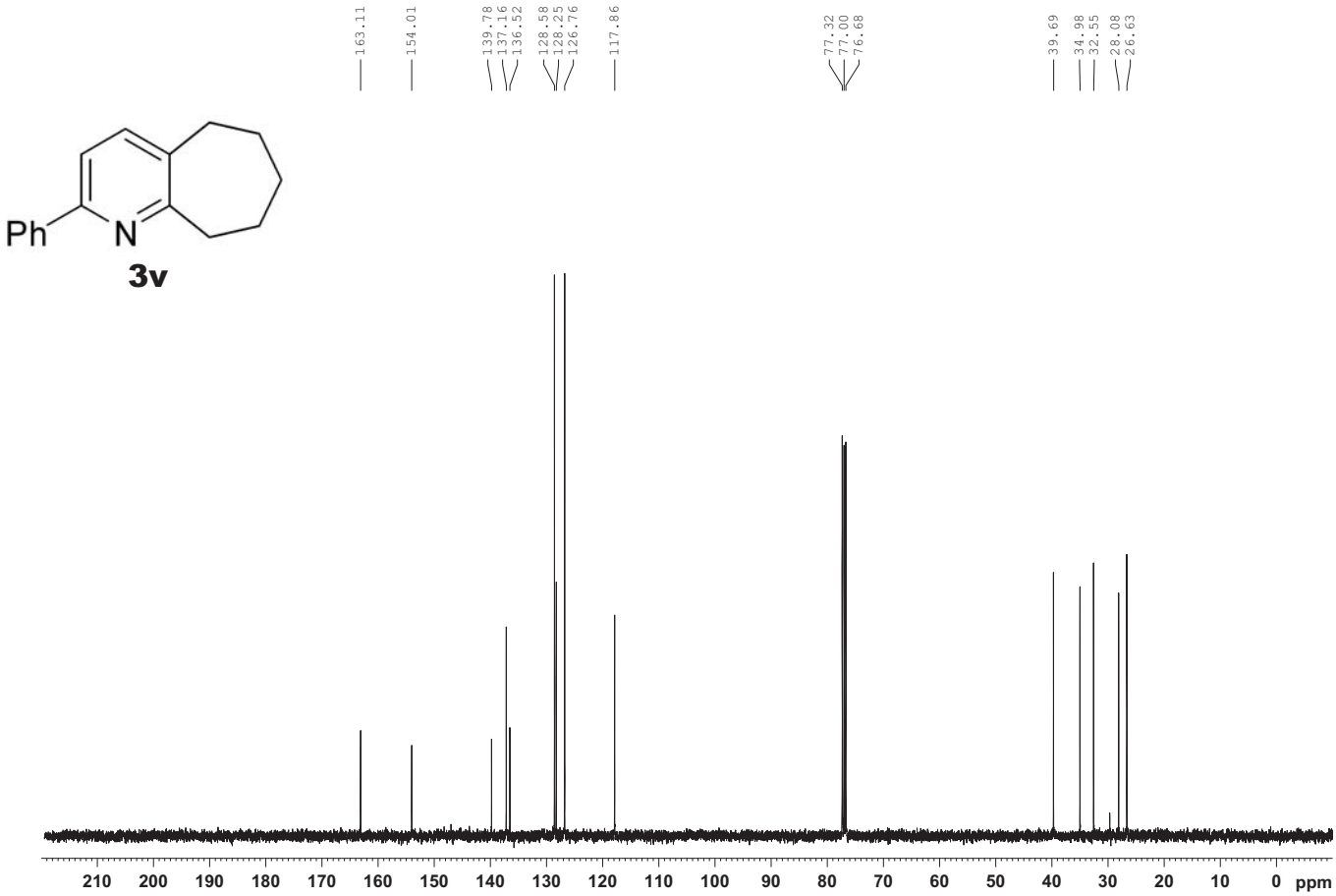
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32.55
28.08
26.63
Ph
N
3v
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200
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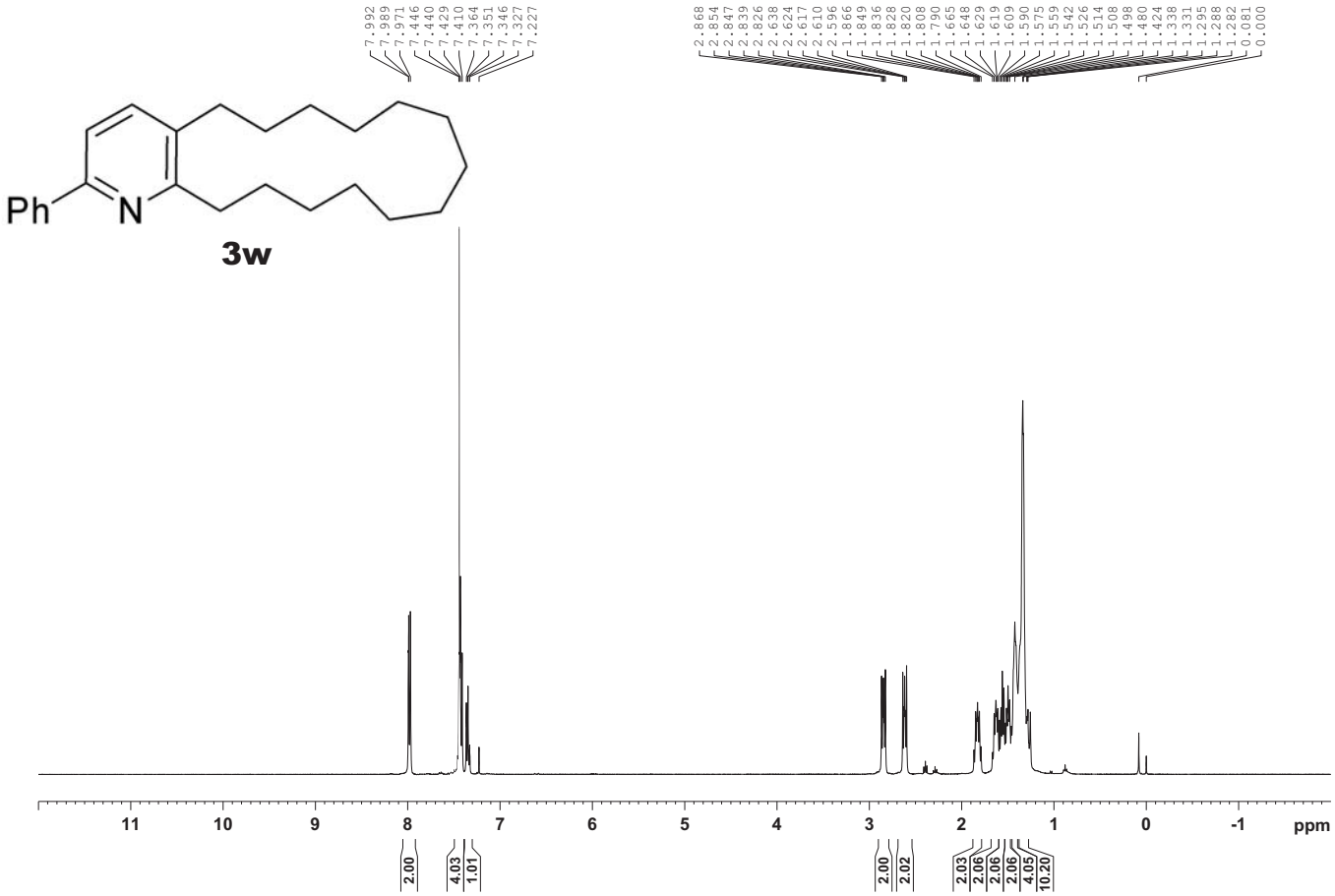
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1.526
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1.480
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1.295
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1.282
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0.000
Ph
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3w
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10
9
8
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6
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4
3
2
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2.06
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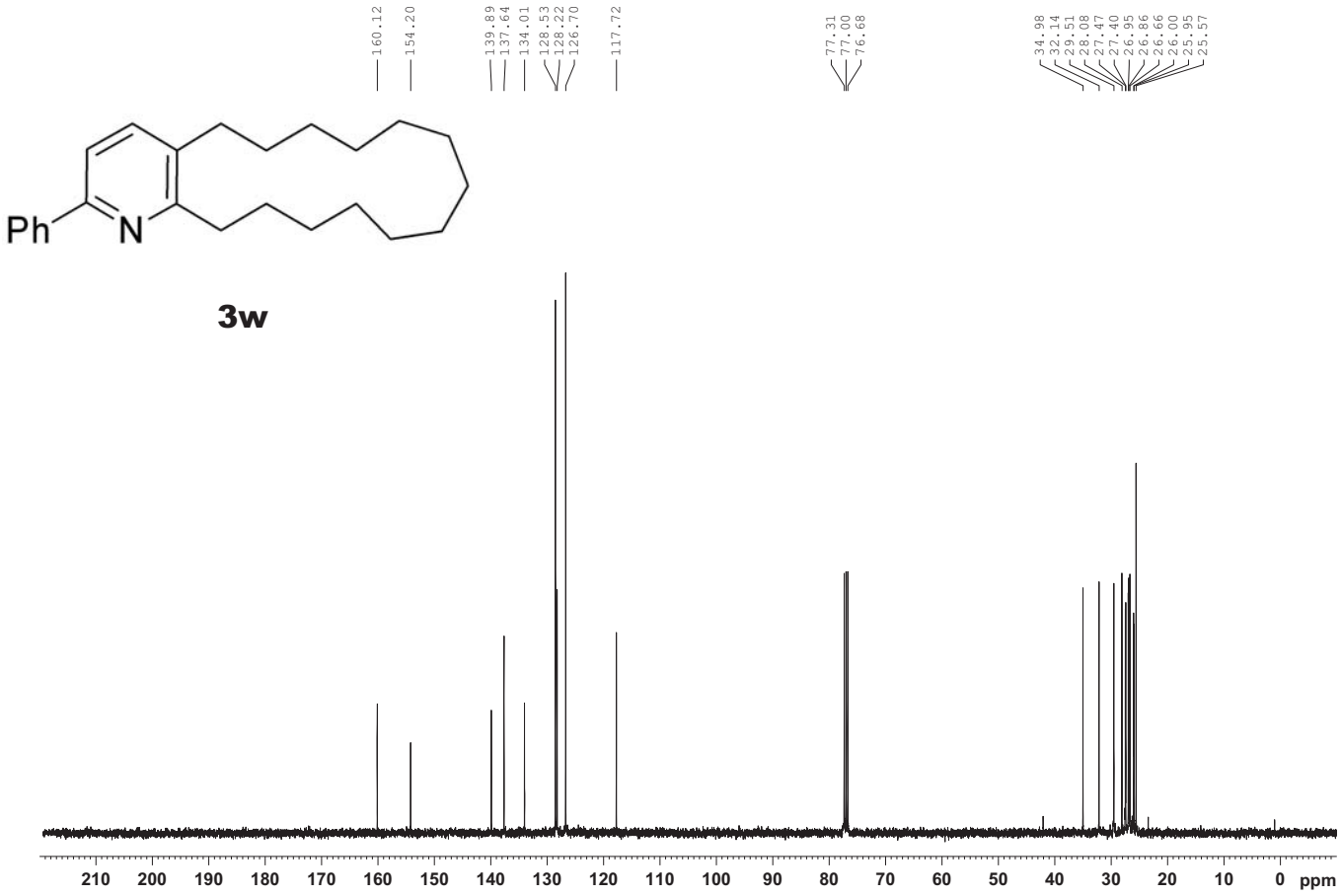
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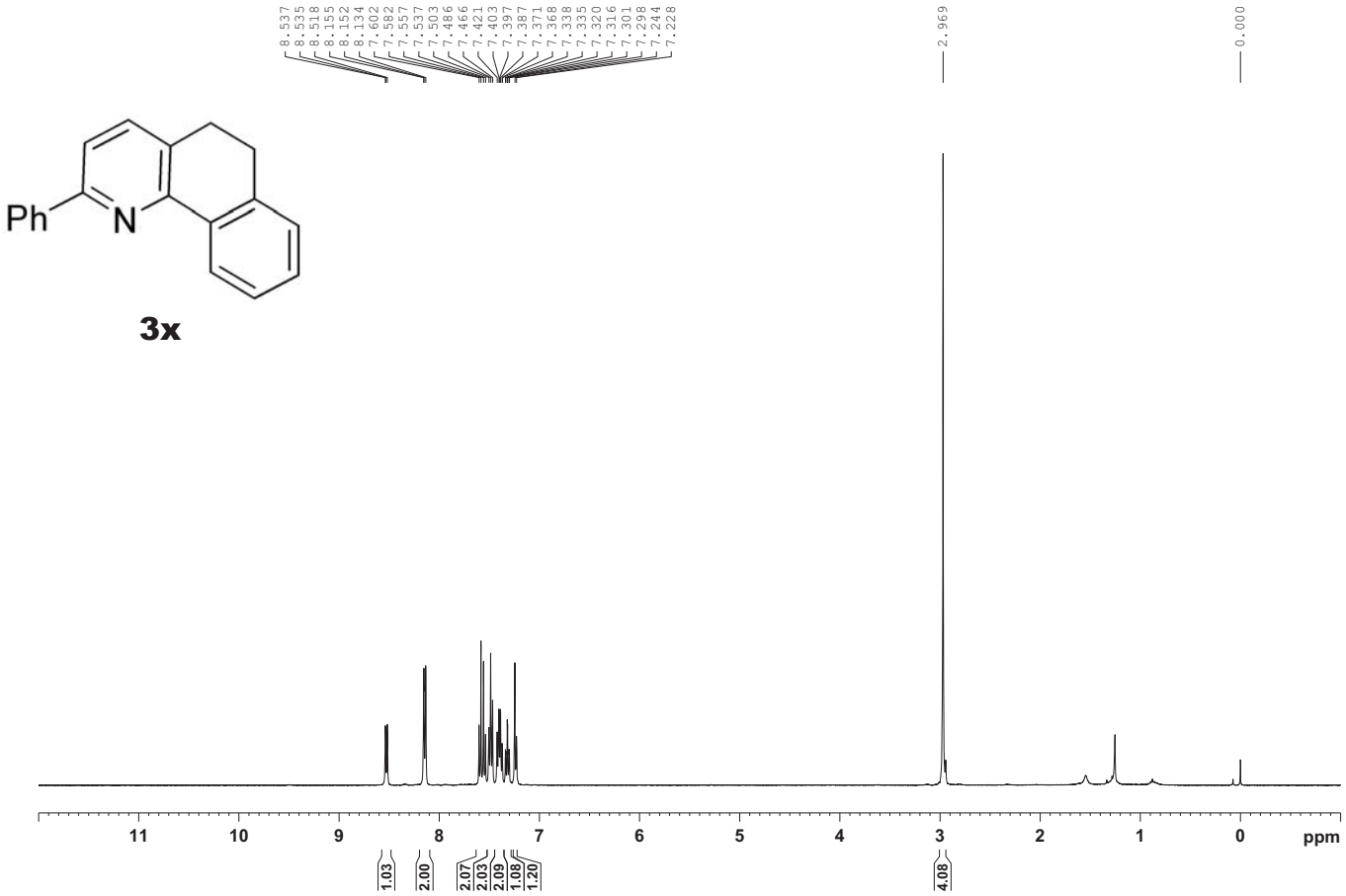
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7.421
7.403
7.397
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7.368
7.338
7.335
7.320
7.316
7.301
7.298
7.244
7.228
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0.000
Ph
N
3x
11
10
9
8
7
6
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ppm
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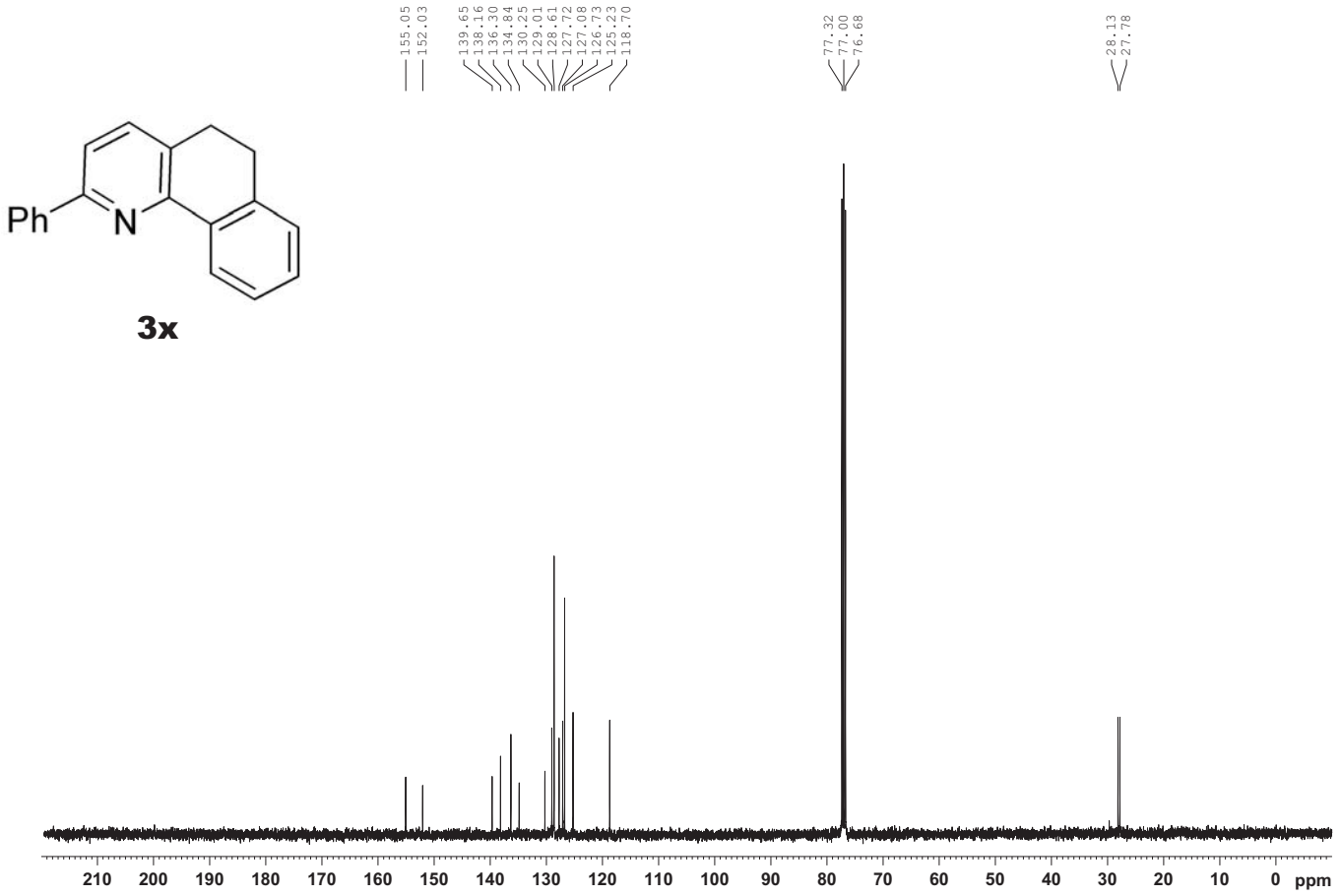
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125.23
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77.32
77.00
76.68
28.13
27.78
Ph
N
3x
210
200
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180
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140
130
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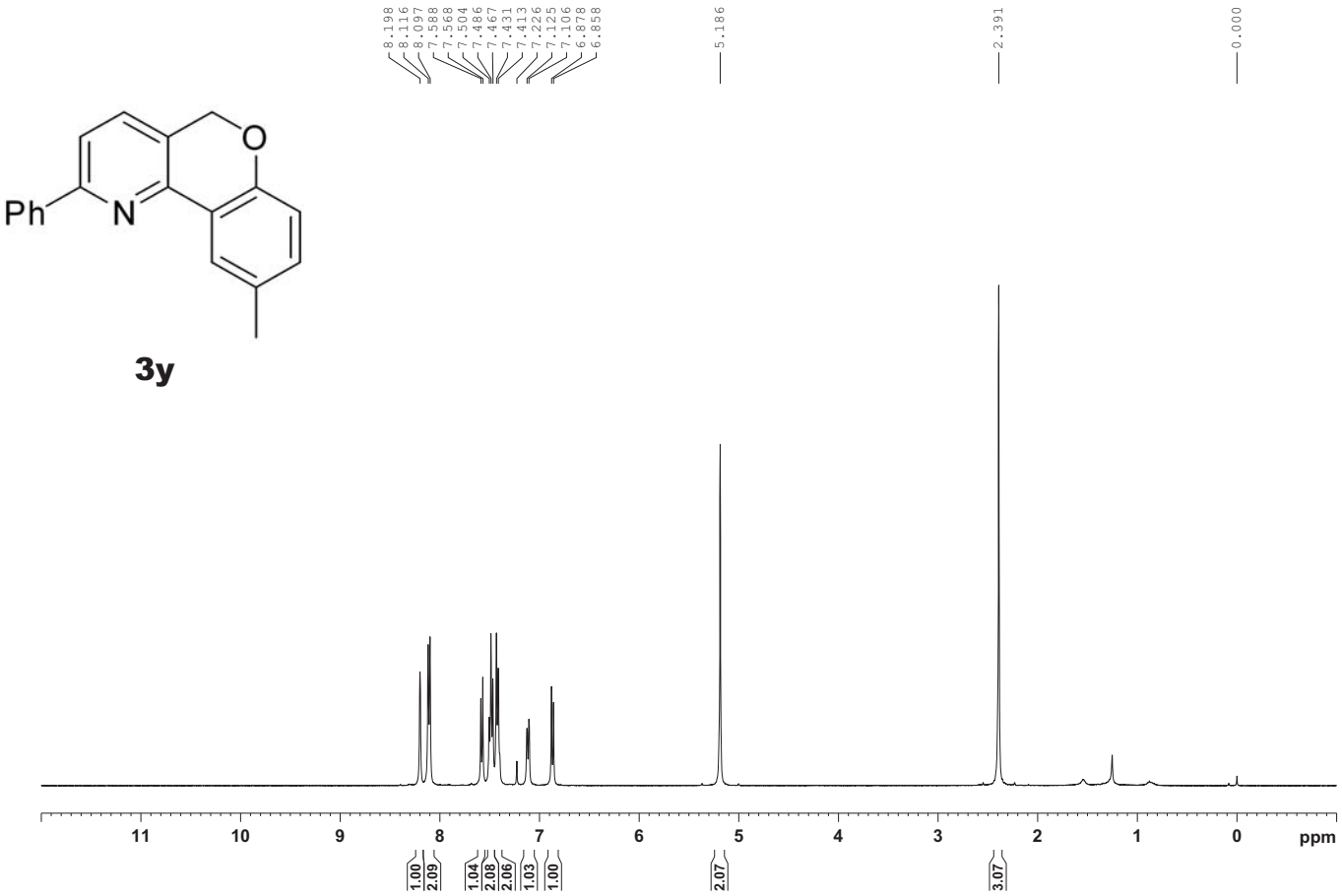
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7.106
6.878
6.858
5.186
2.391
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Ph
N
O
3y
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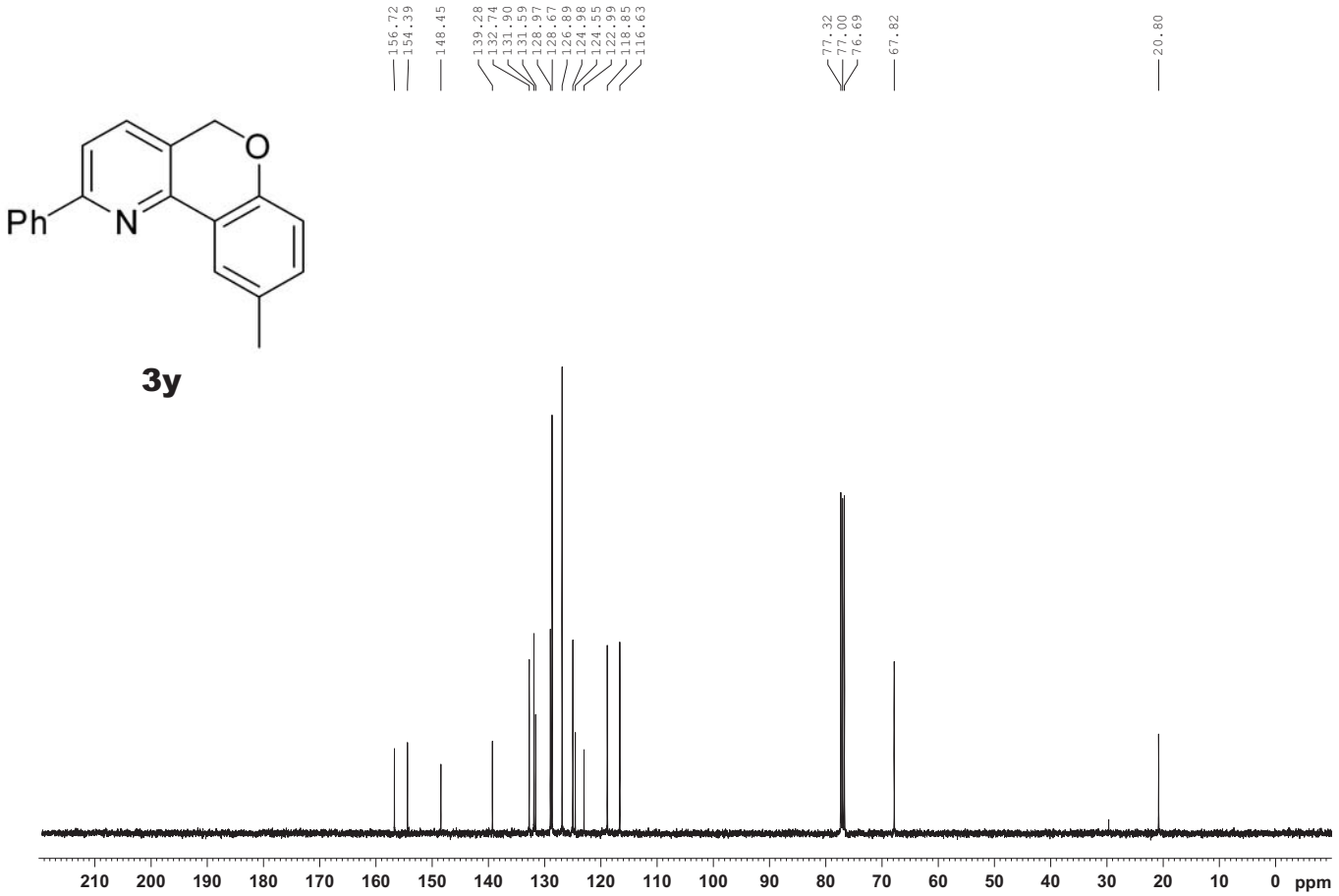
2.08
2.06
1.03
1.00
2.07
3.07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128.97
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124.98
124.55
122.99
118.85
116.63
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67.82
20.80
Ph
N
O
3y
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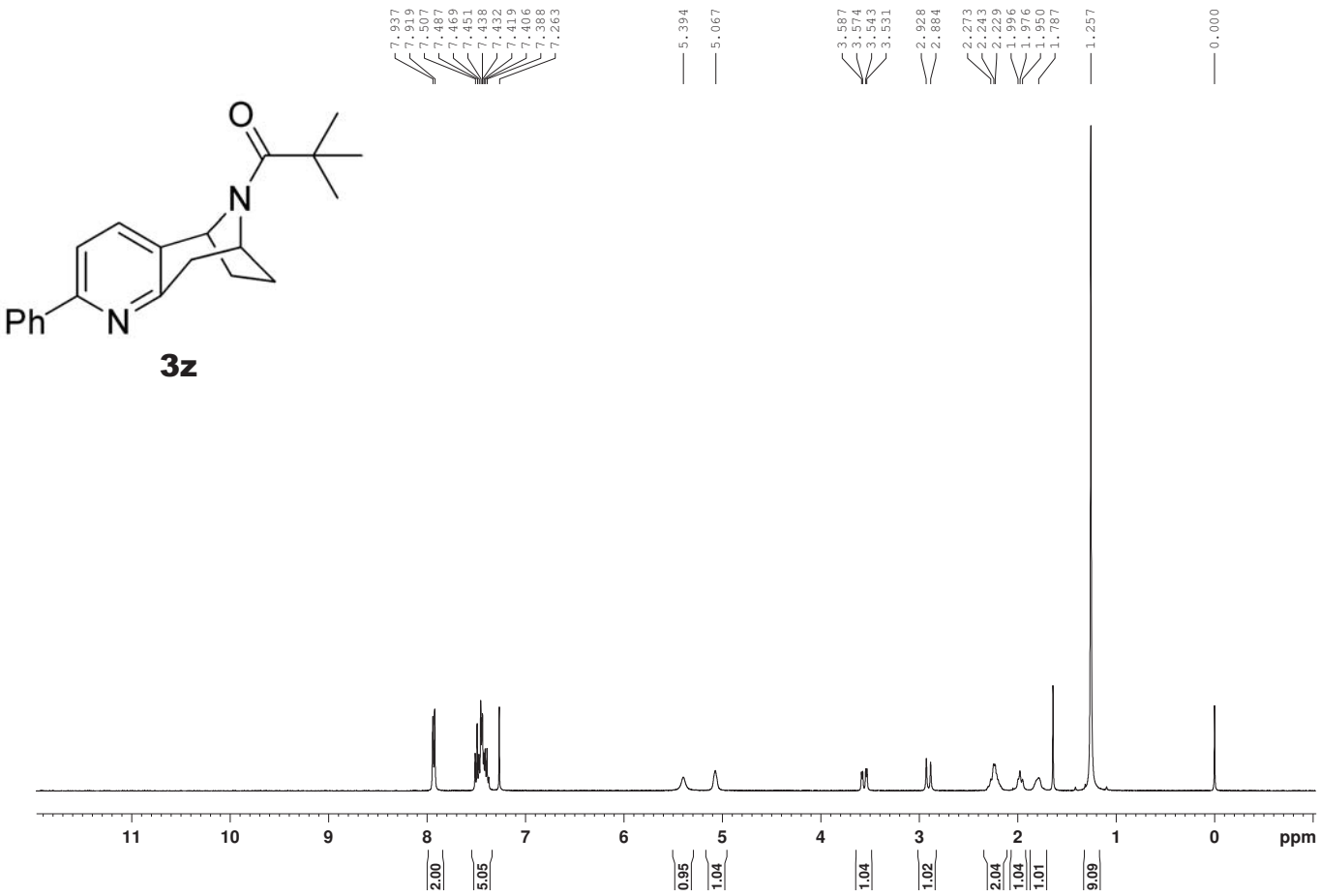
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5.394
5.067
3.587
3.574
3.543
3.531
2.928
2.884
2.273
2.243
2.229
1.996
1.976
1.950
1.787
1.257
0.000
O
N
Ph
N
3z
11
10
9
8
7
6
5
4
3
2
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ppm
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5.05
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1.04
1.04
1.02
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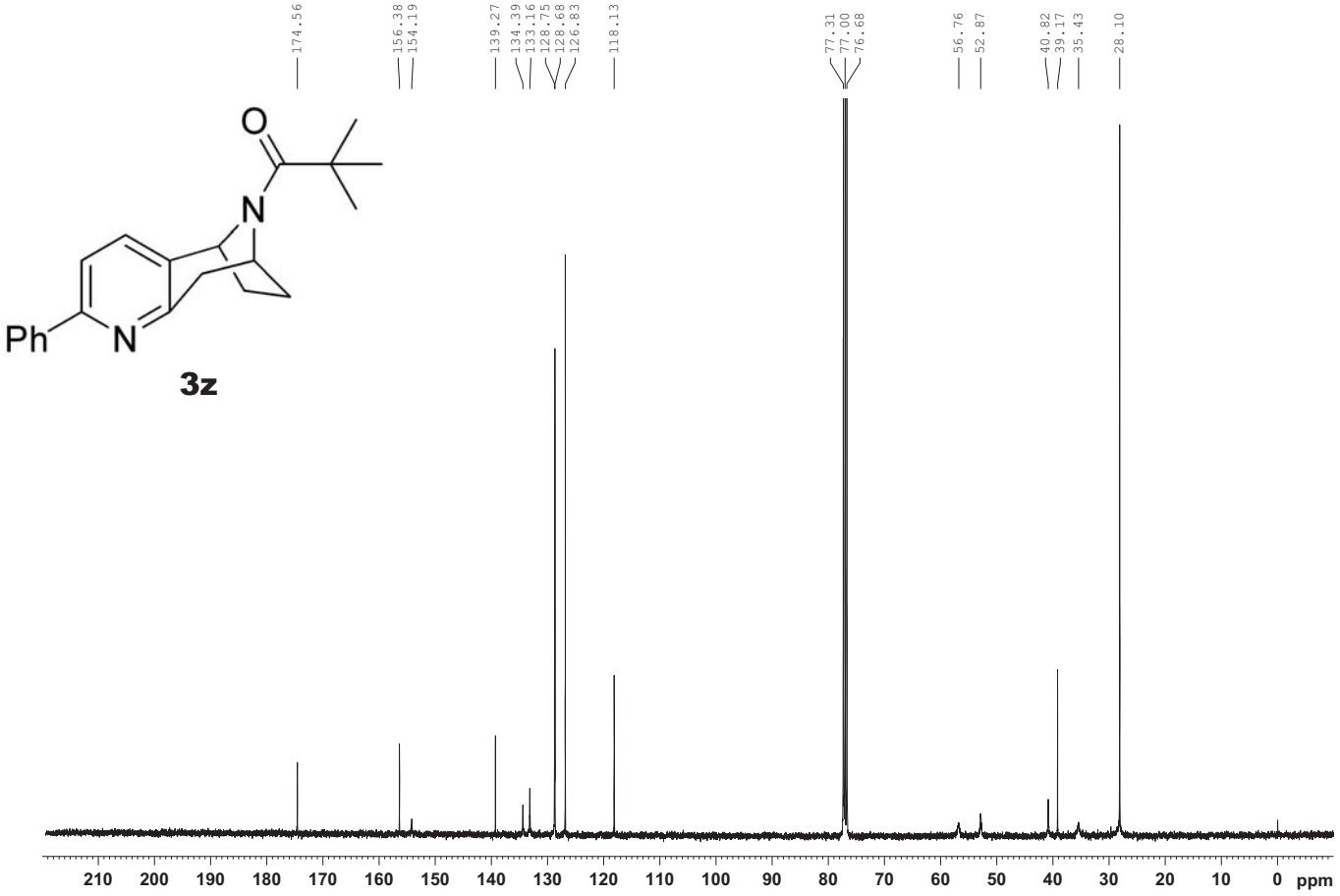
1.04
1.01
9.09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76.68
56.76
52.87
40.82
39.17
35.43
28.10
O
N
Ph
N
3z
210
200
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180
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140
130
120
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80
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60
50
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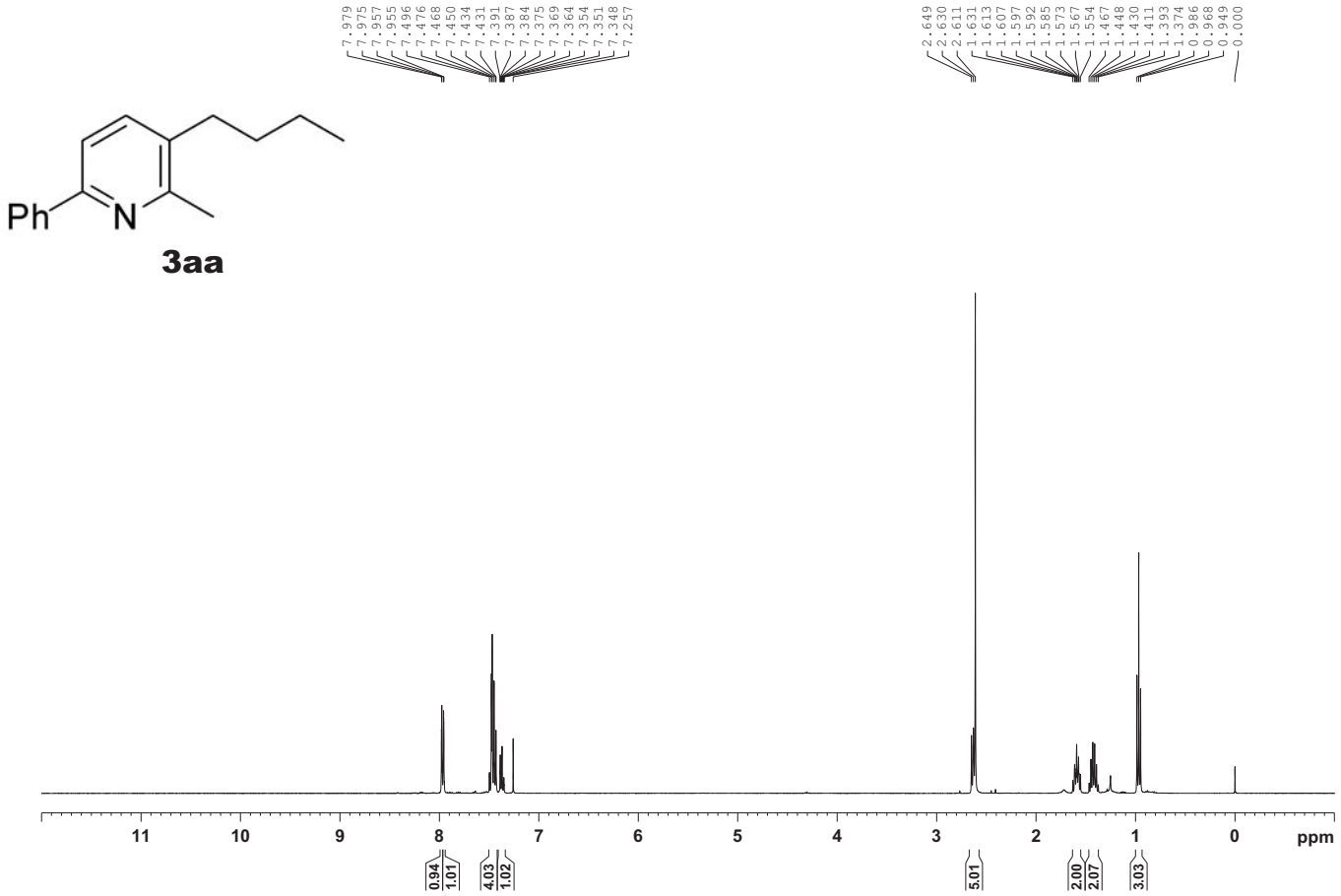
30
20
10
0
ppm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7.364
7.354
7.351
7.348
7.257
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2.630
2.611
1.631
1.613
1.607
1.597
1.592
1.585
1.573
1.567
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0.949
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3aa
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10
9
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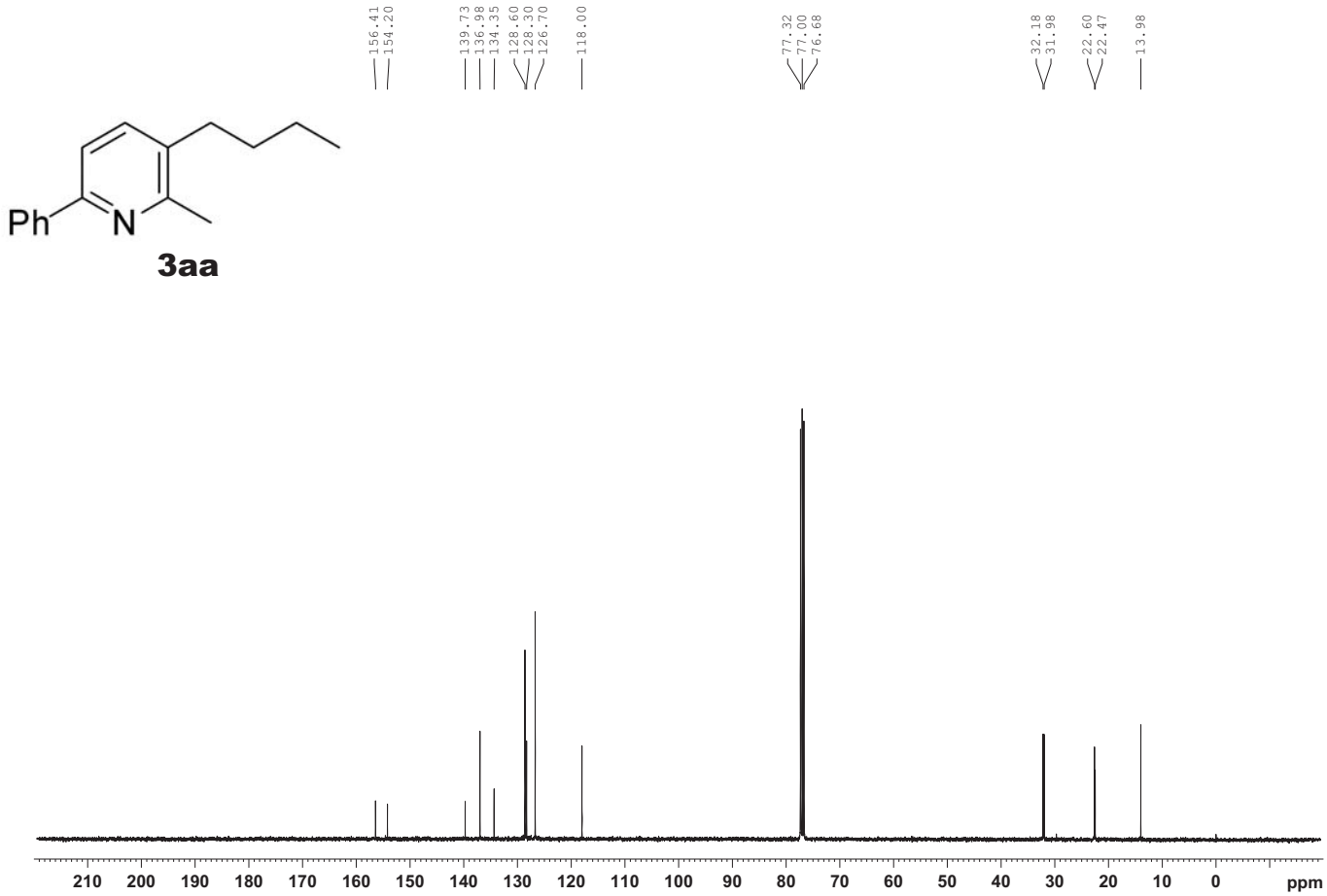
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77.00
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32.18
31.98
22.60
22.47
13.98
Ph
N
3aa
210
200
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180
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140
130
120
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60
50
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10
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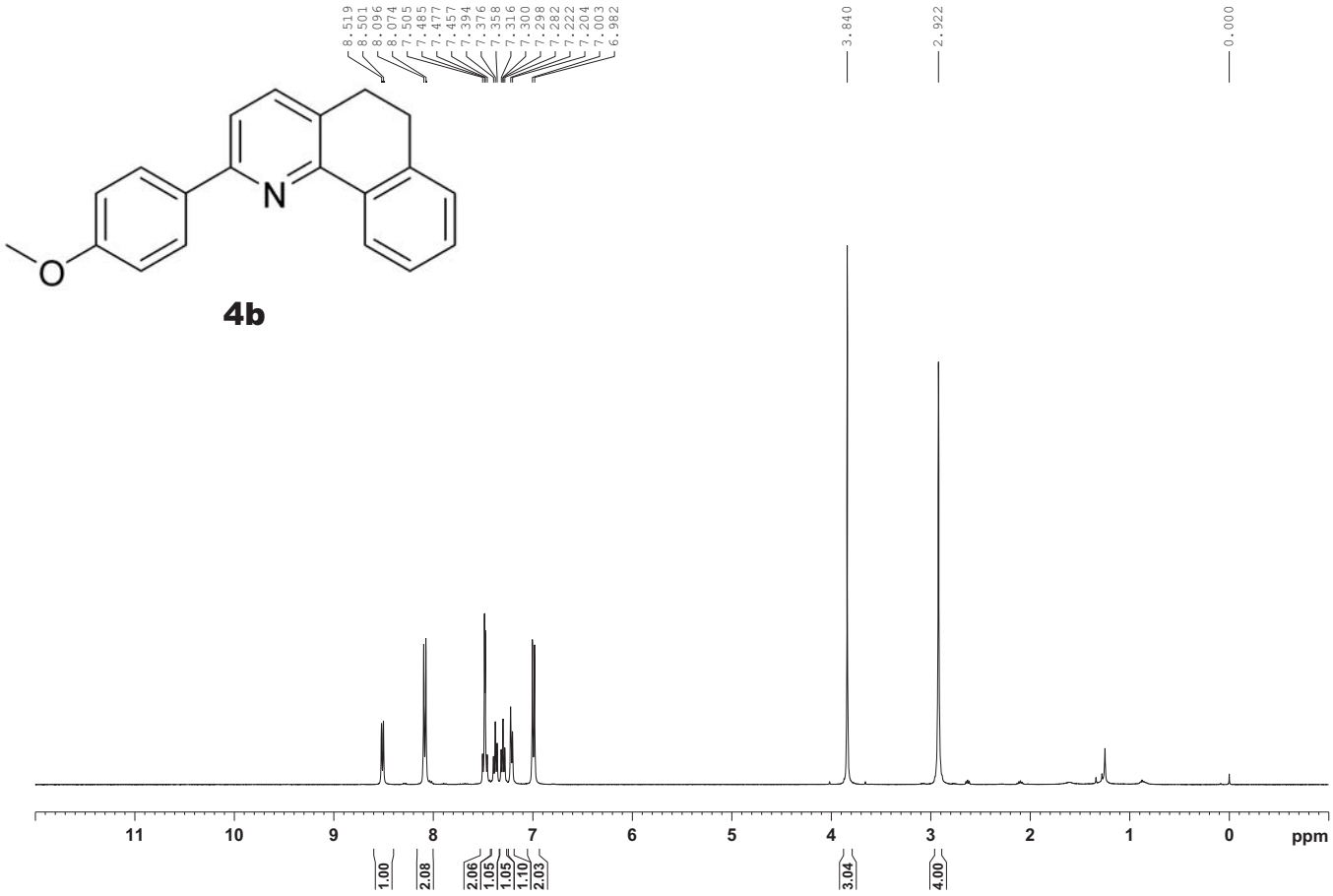
ppm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7.300
7.298
7.282
7.222
7.204
7.003
6.982
3.840
2.922
0.000
N
O
4b
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1.05
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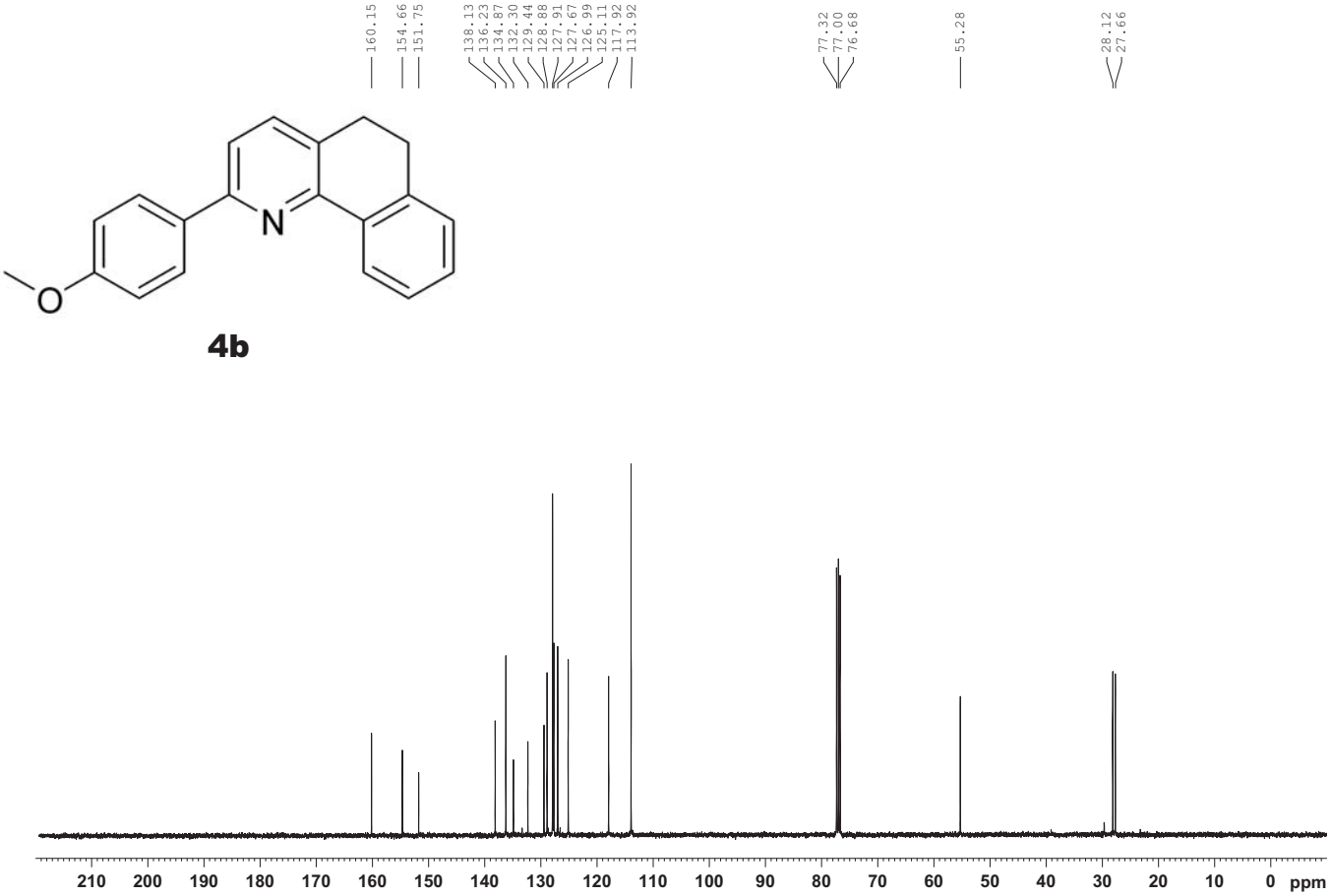
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127.91
127.67
126.99
125.11
117.92
113.92
77.32
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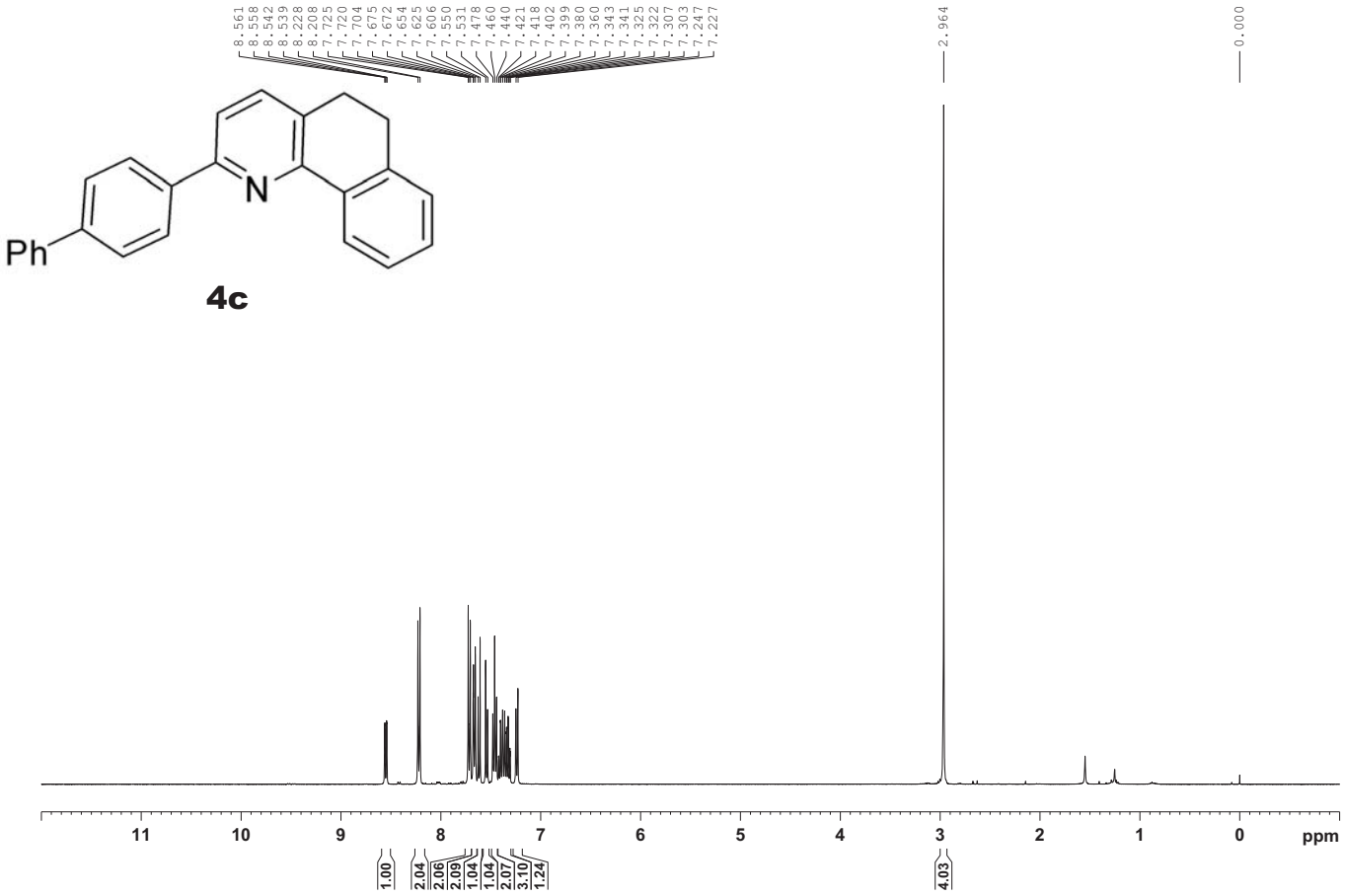
76.68
55.28
28.12
27.66
N
O
4b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.606
7.550
7.531
7.478
7.460
7.440
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7.418
7.402
7.399
7.380
7.360
7.343
7.341
7.325
7.322
7.307
7.303
7.247
7.227
2.964
0.000
N
Ph
4c
11
10
9
8
7
6
5
4
3
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2.06
2.09
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1.04
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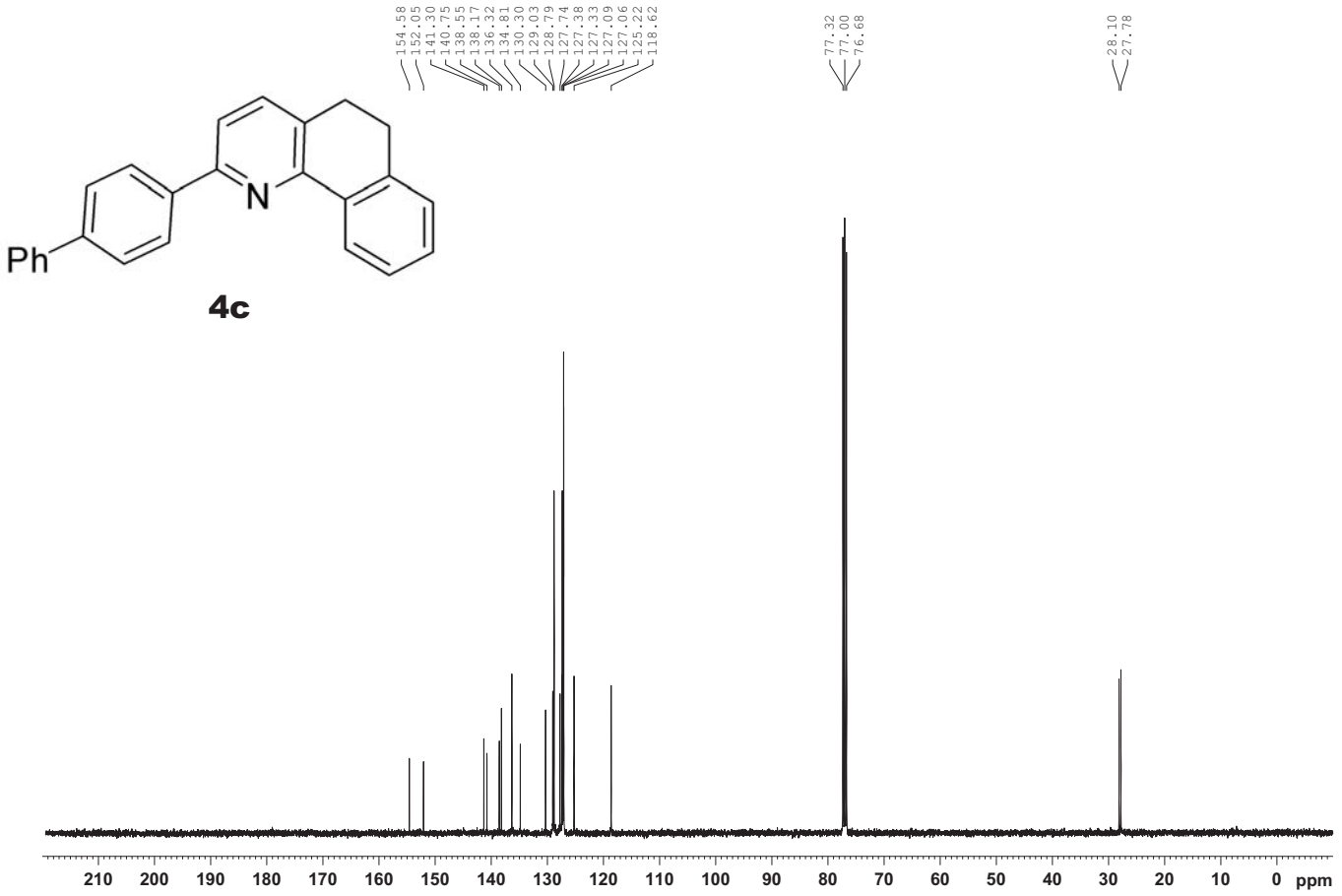
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127.09
127.06
125.22
118.62
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77.00
76.68
28.10
27.78
N
Ph
4c
210
200
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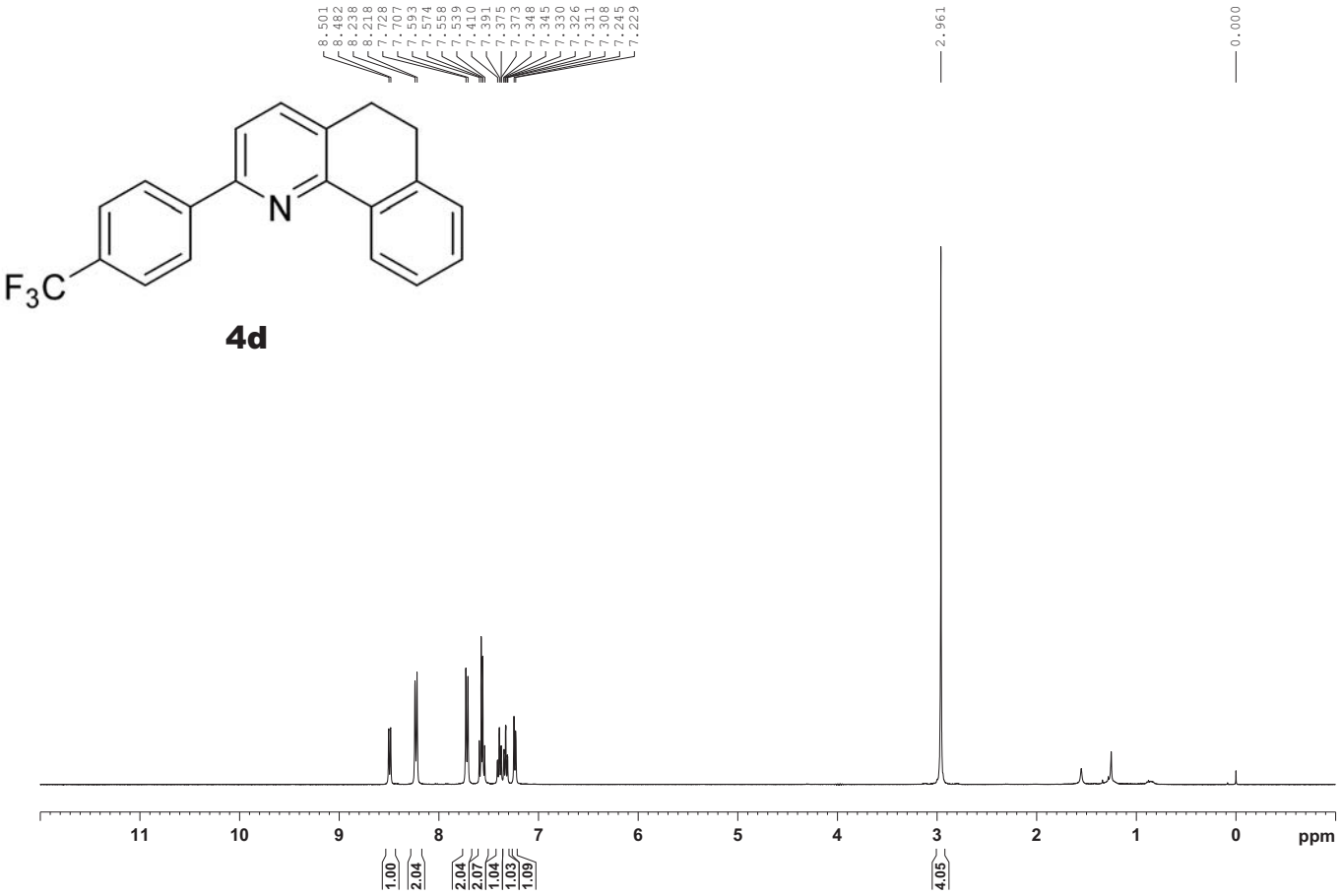
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7.373
7.348
7.345
7.330
7.326
7.311
7.308
7.245
7.229
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N
F3C
4d
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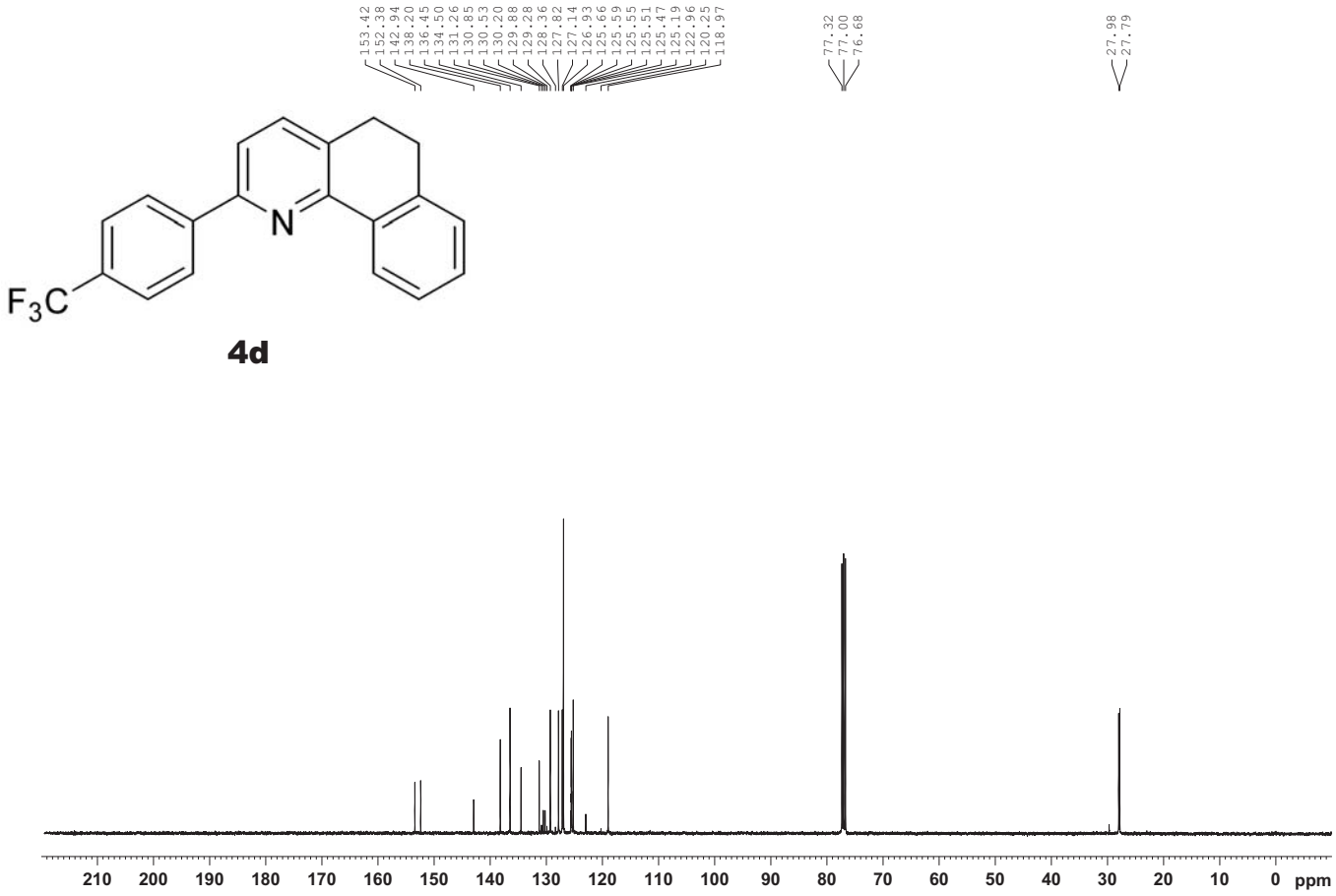
2.04
2.07
1.04
1.03
1.09
4.05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128.36
127.82
127.14
126.93
125.66
125.59
125.55
125.51
125.47
125.19
122.96
120.25
118.97
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F3C
4d
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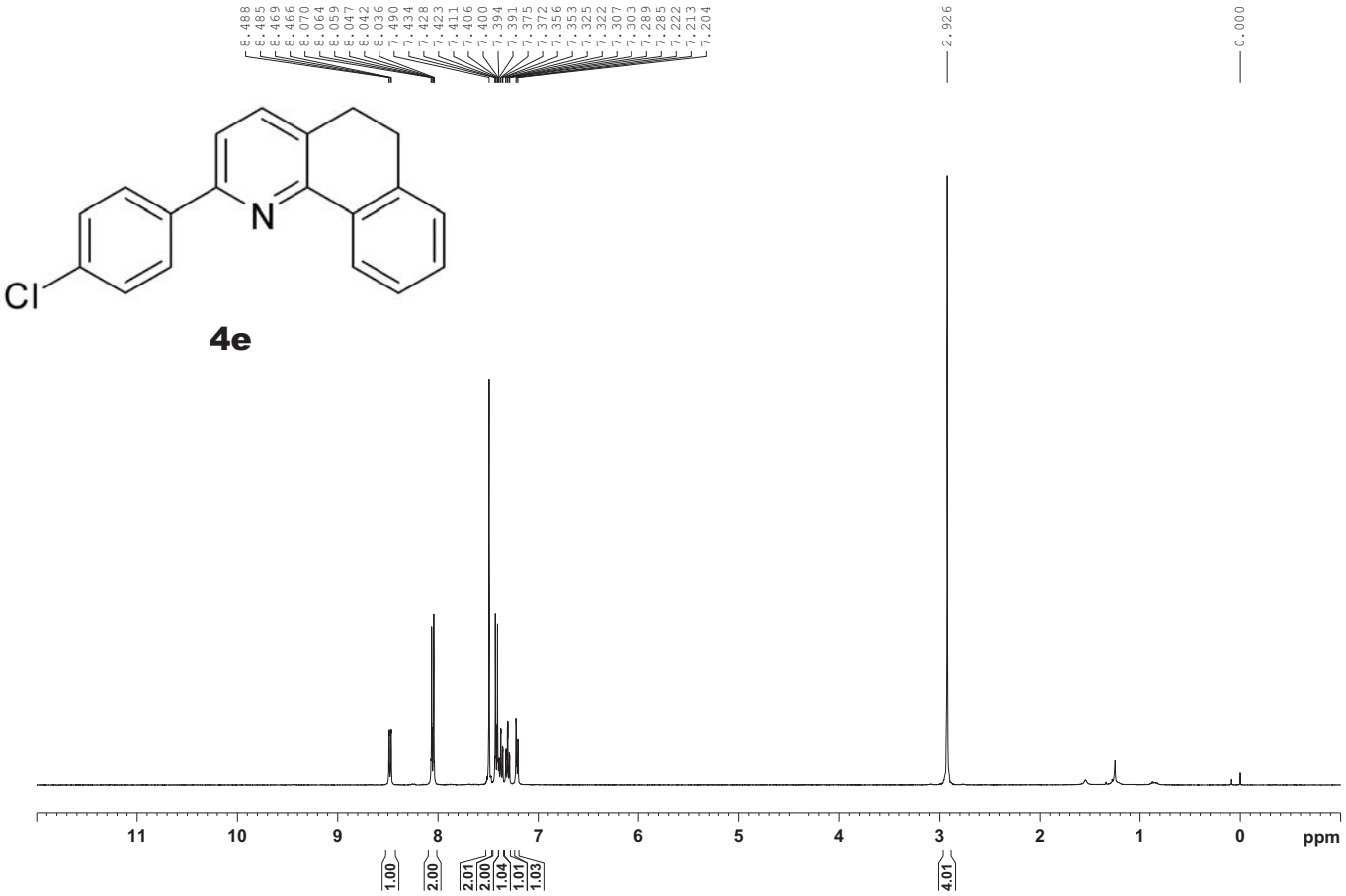
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7.434
7.428
7.423
7.411
7.406
7.400
7.394
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7.375
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7.325
7.322
7.307
7.303
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7.285
7.222
7.213
7.204
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Cl
4e
11
10
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8
7
6
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2.00
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1.01
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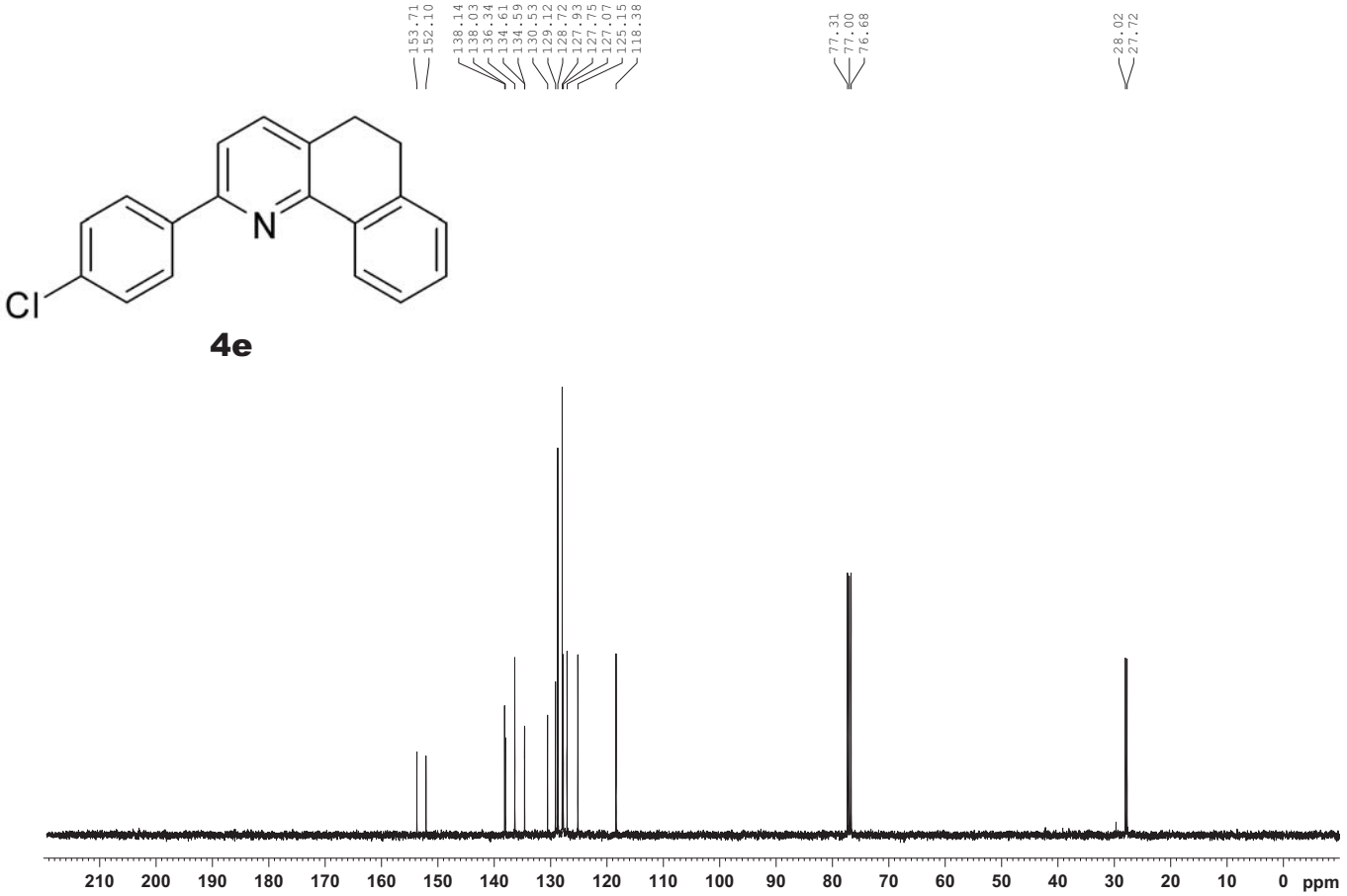
4.01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77.00
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28.02
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N
Cl
4e
210
200
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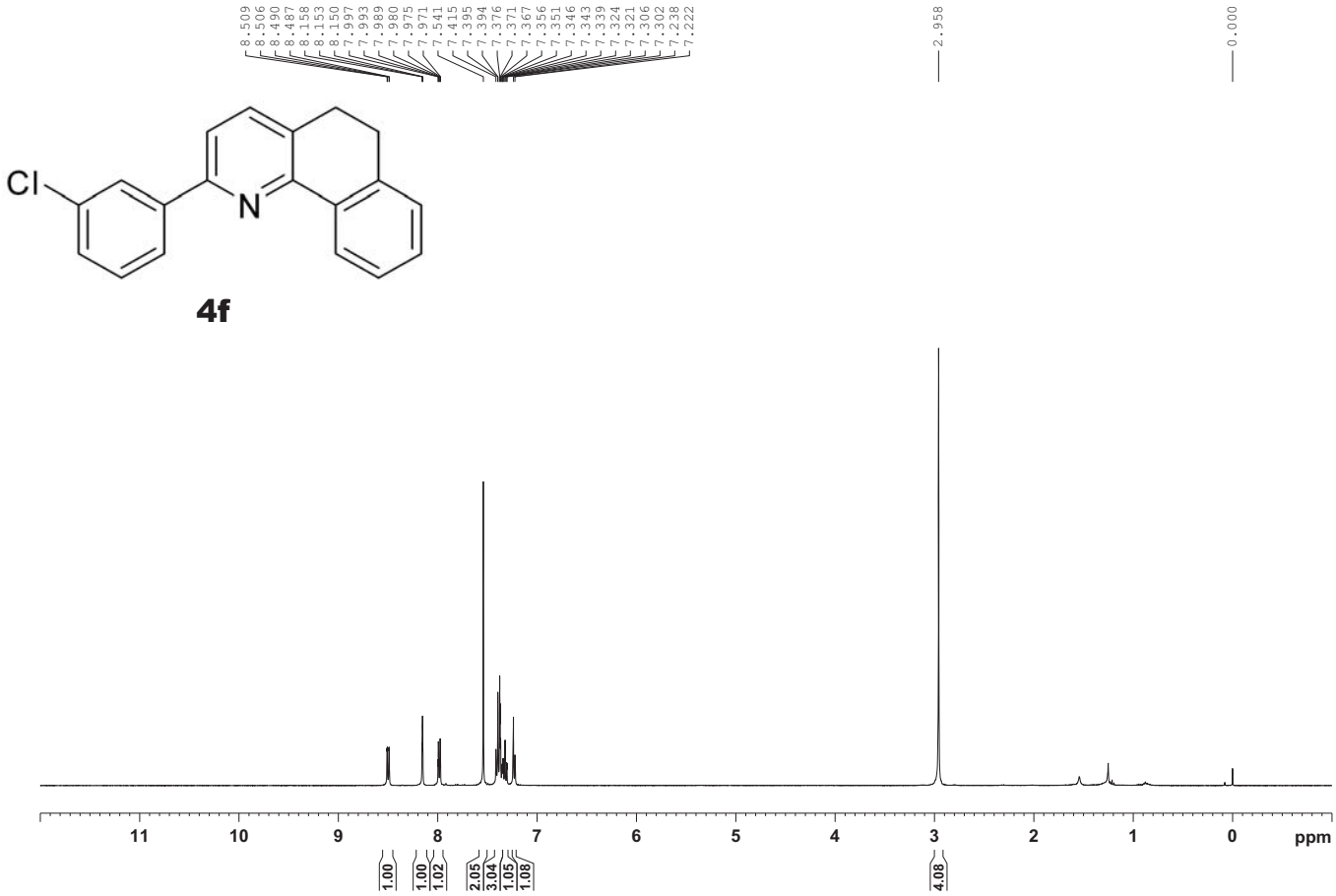
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7.371
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7.346
7.343
7.339
7.324
7.321
7.306
7.302
7.238
7.222
2.958
0.000
Cl
N
4f
11
10
9
8
7
6
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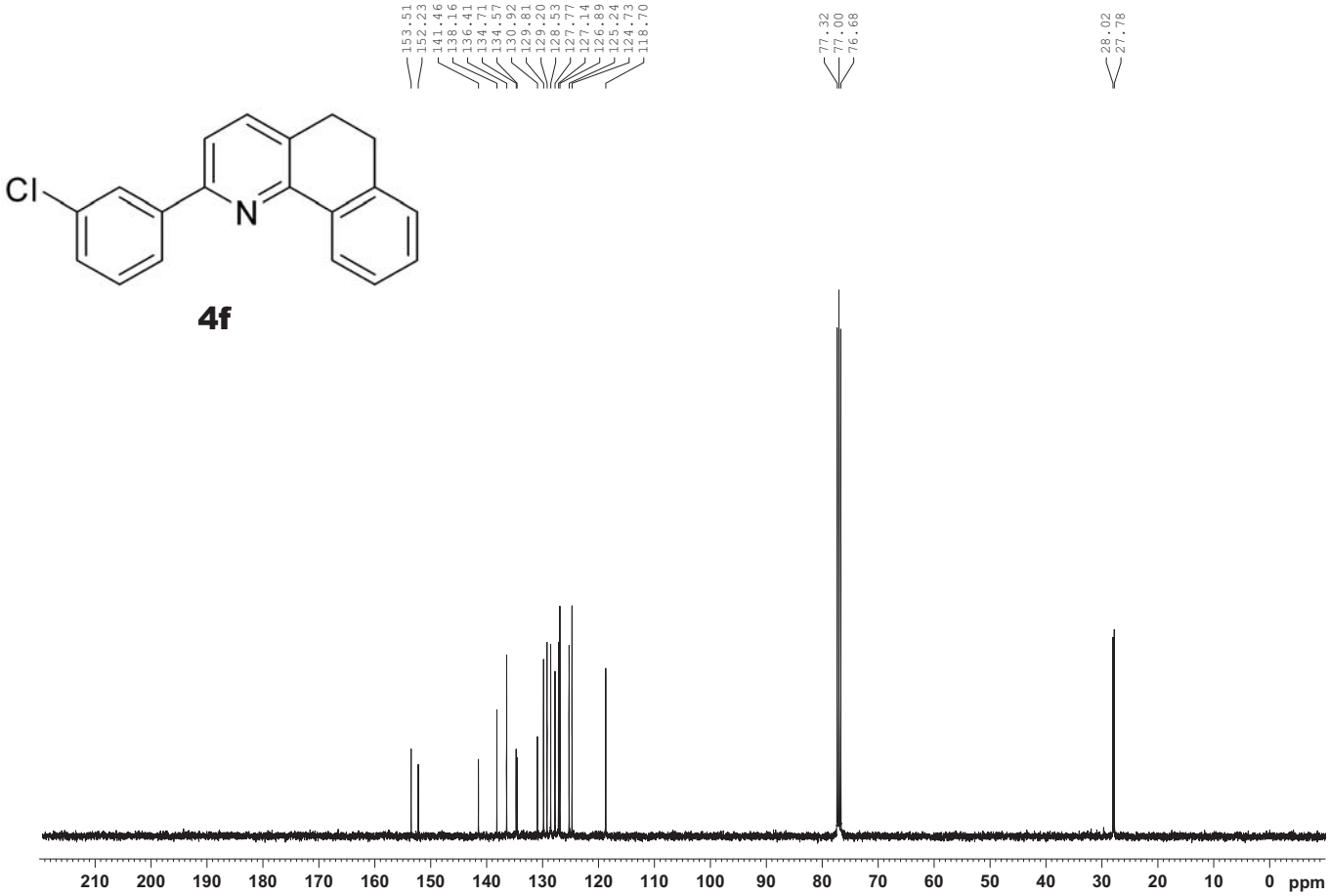
4.08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77.00
76.68
28.02
27.78
Cl
N
4f
210
200
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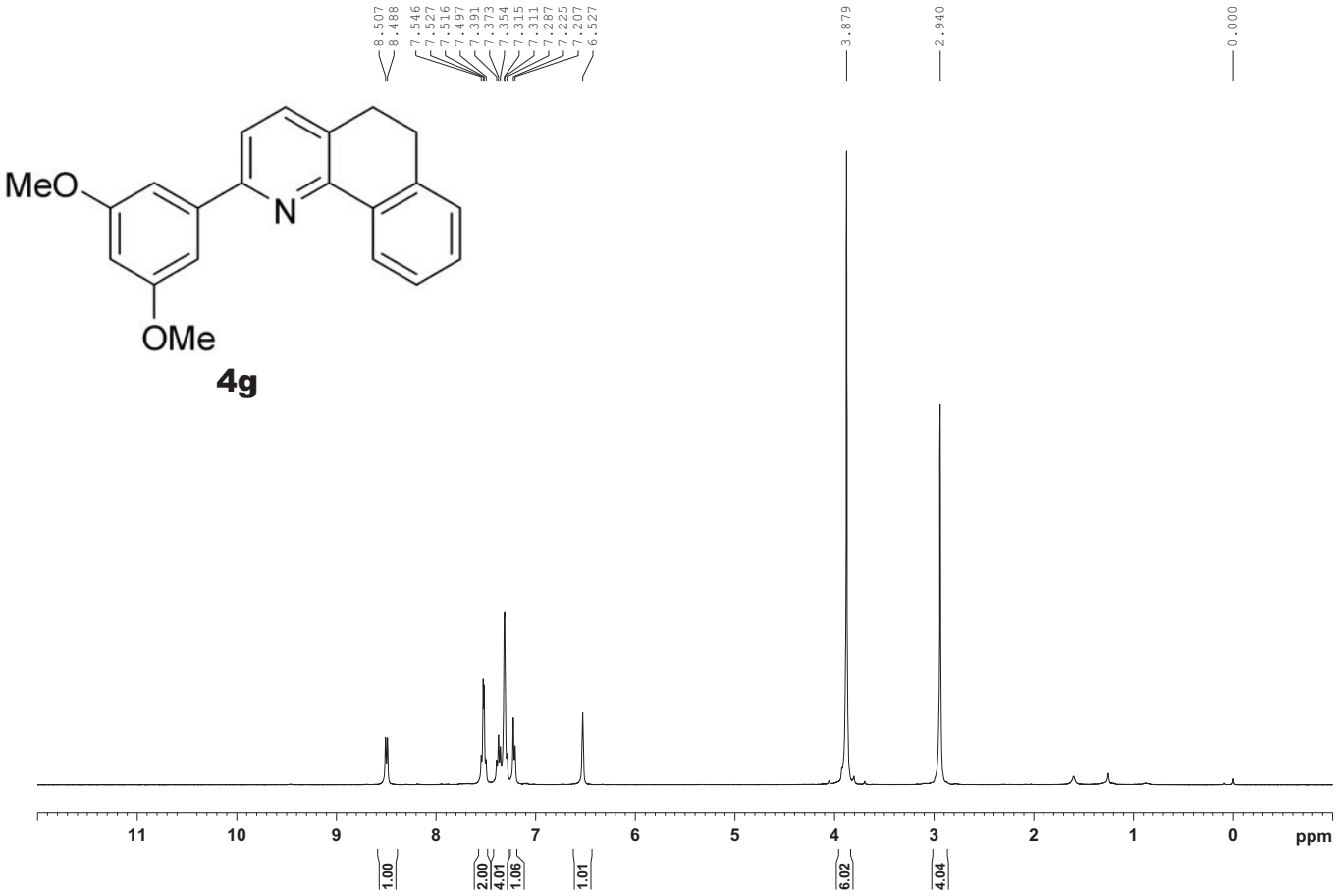
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7.527
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7.373
7.354
7.315
7.311
7.287
7.225
7.207
6.527
3.879
2.940
0.000
MeO
N
OMe
4g
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10
9
8
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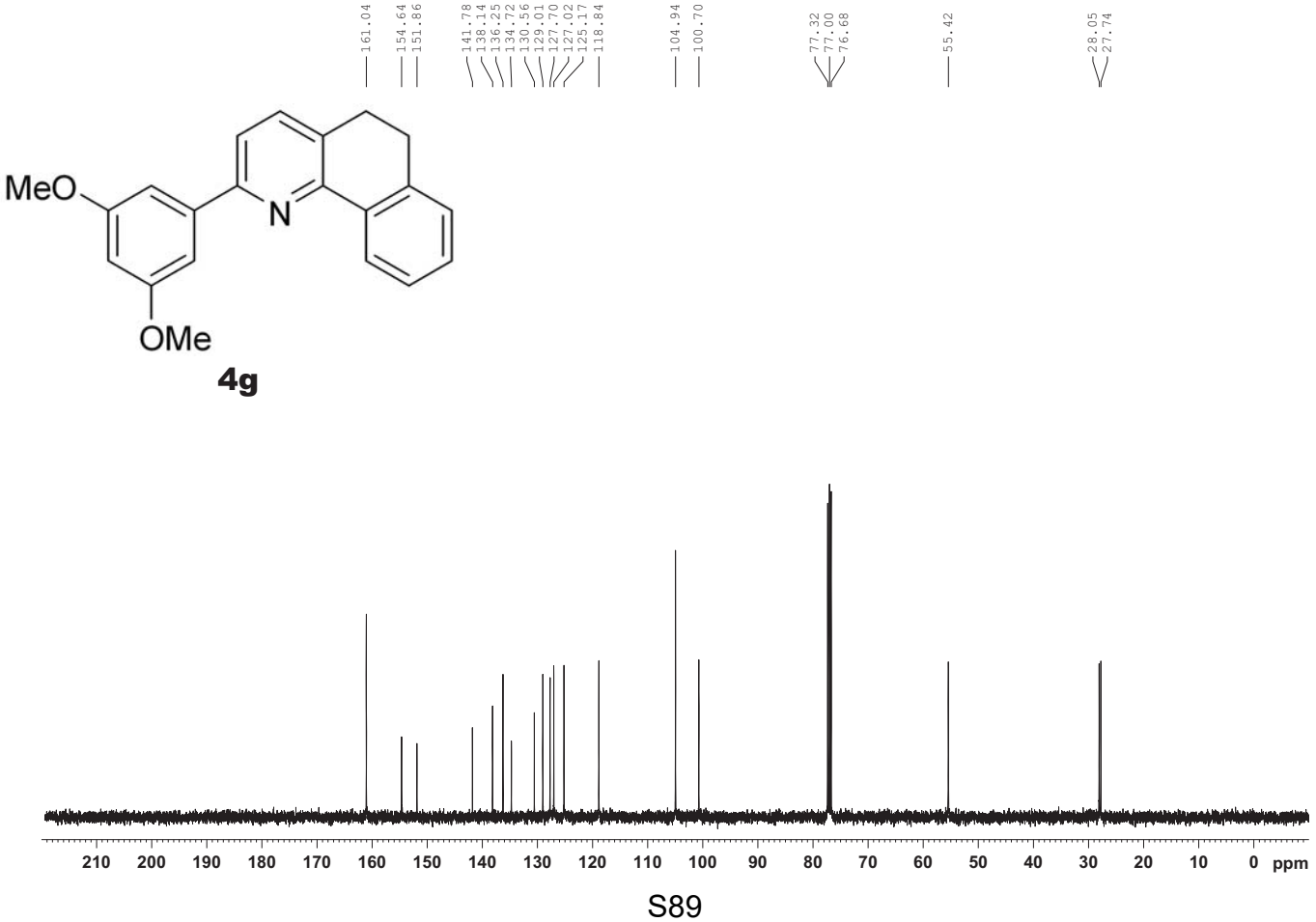
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127.02
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55.42
28.05
27.74
MeO
N
OMe
4g
210
200
190
180
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120
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10
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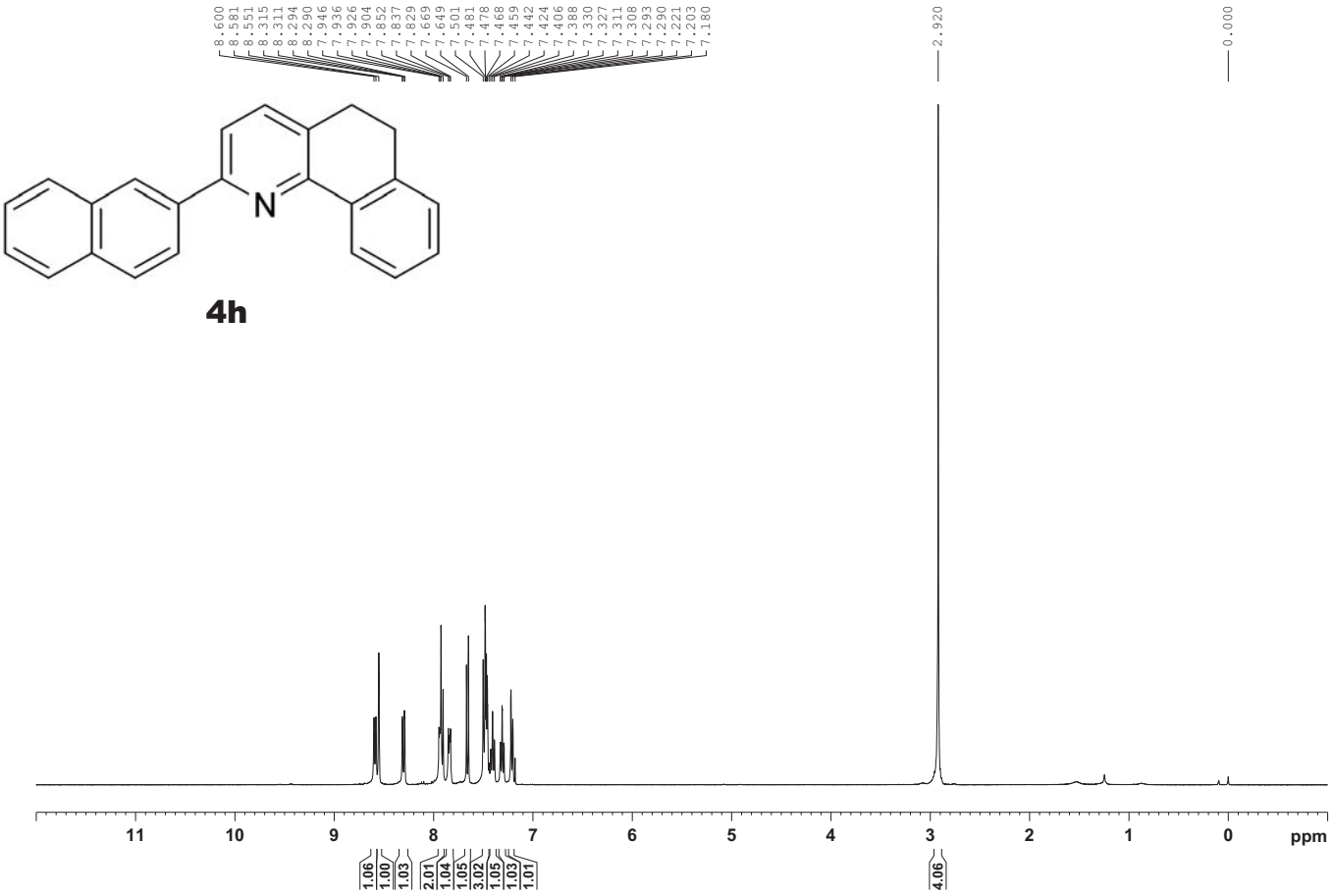
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7.829
7.669
7.649
7.501
7.481
7.478
7.468
7.459
7.442
7.424
7.406
7.388
7.330
7.327
7.311
7.308
7.293
7.290
7.221
7.203
7.180
2.920
0.000
N
4h
11
10
9
8
7
6
5
4
3
2
1
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1.03
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3.02
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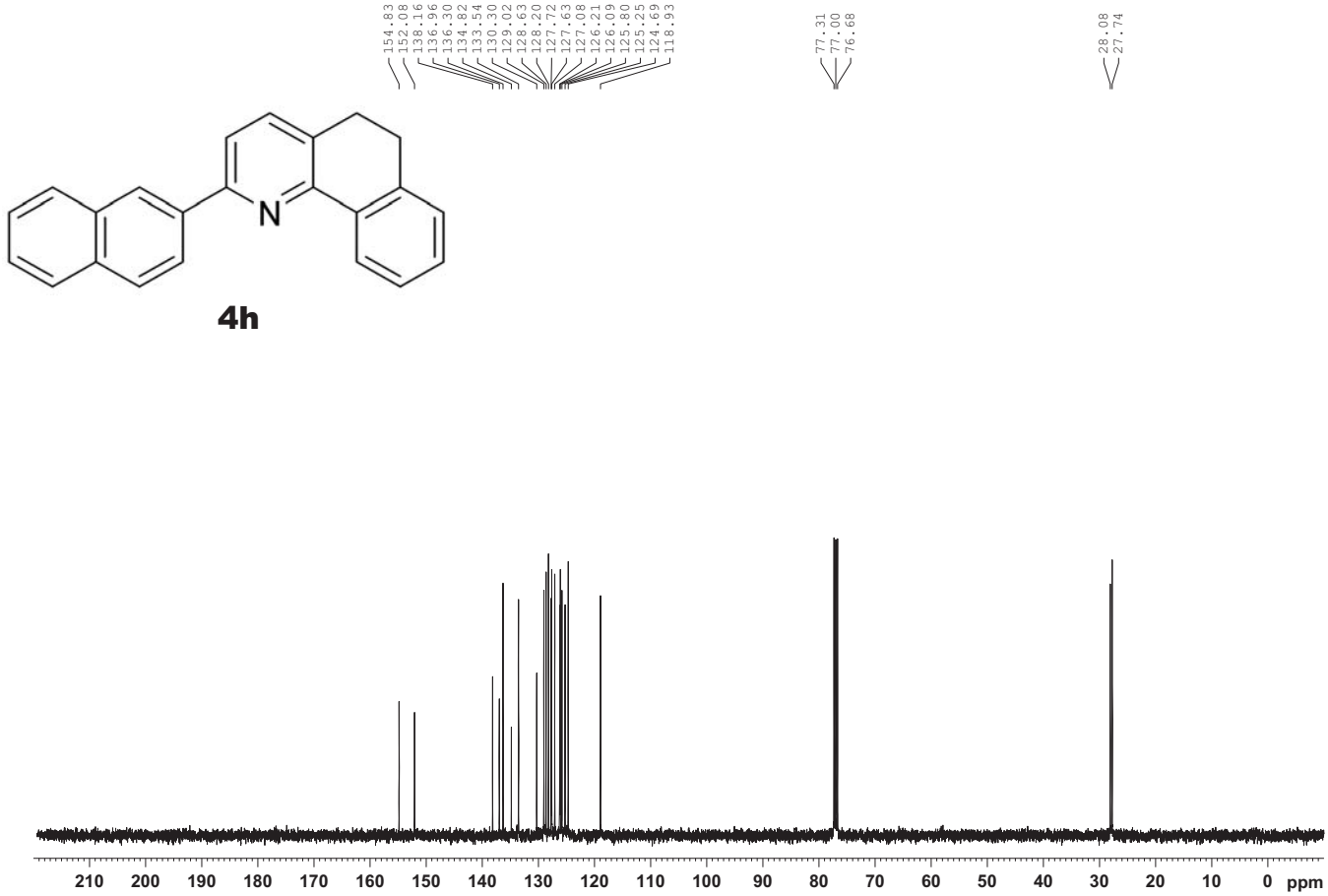
1.01
4.06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127.63
127.08
126.21
126.09
125.80
125.25
124.69
118.93
77.31
77.00
76.68
28.08
27.74
N
4h
210
200
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180
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140
130
120
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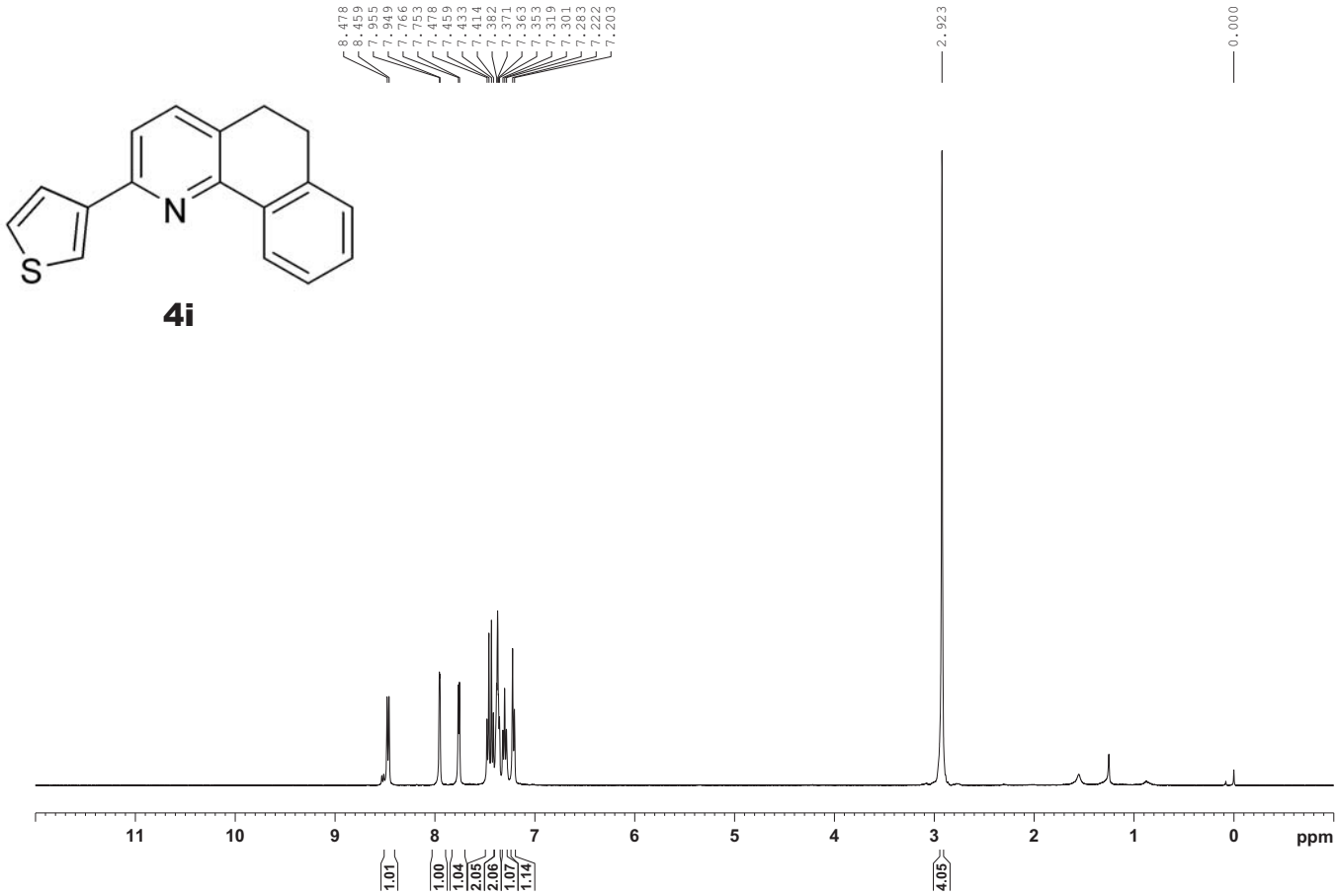
ppm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7.363
7.353
7.319
7.301
7.283
7.222
7.203
2.923
0.000
N
S
4i
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2.05
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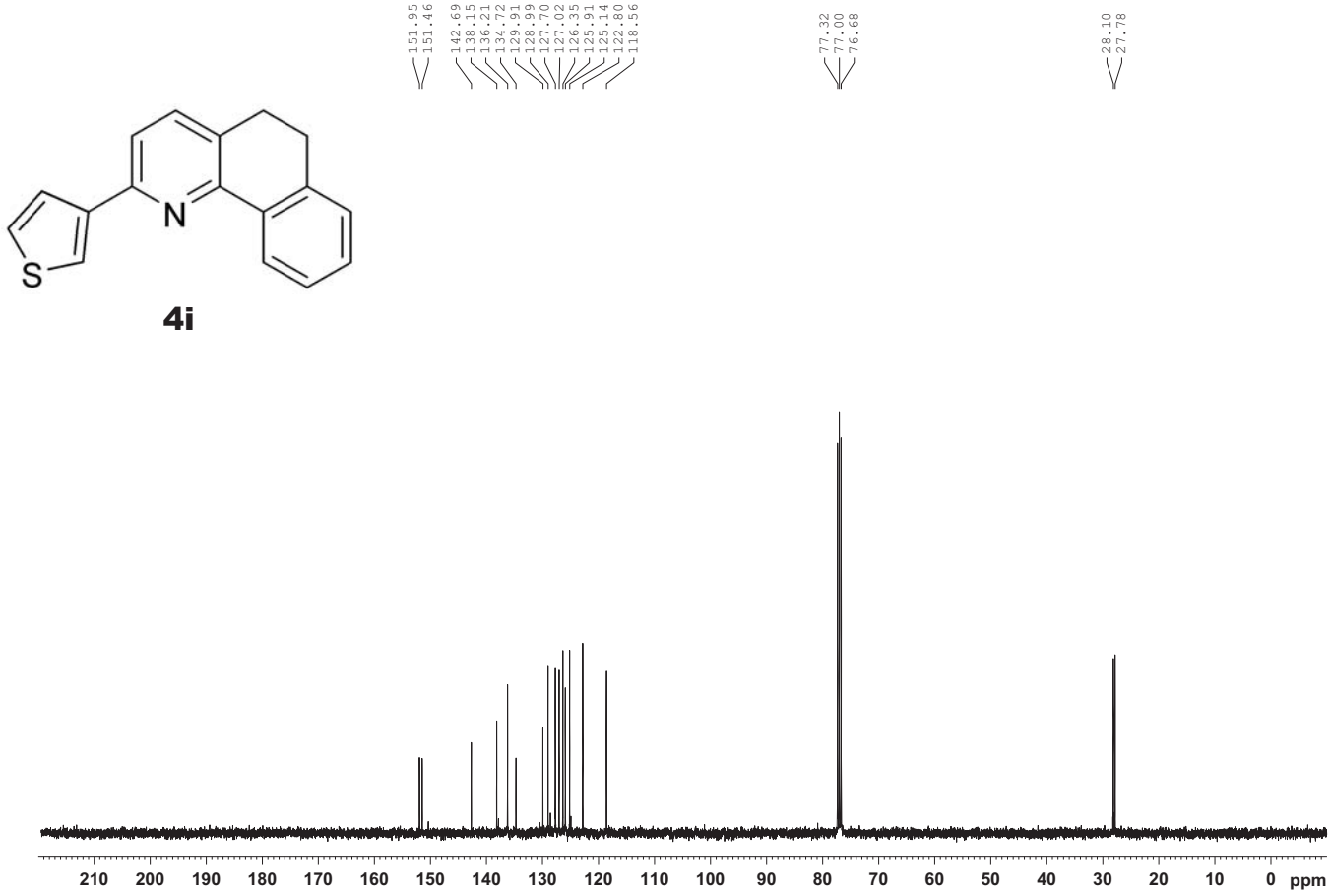
1.07
1.14
4.05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127.02
126.35
125.91
125.14
122.80
118.56
77.32
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76.68
28.10
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S
4i
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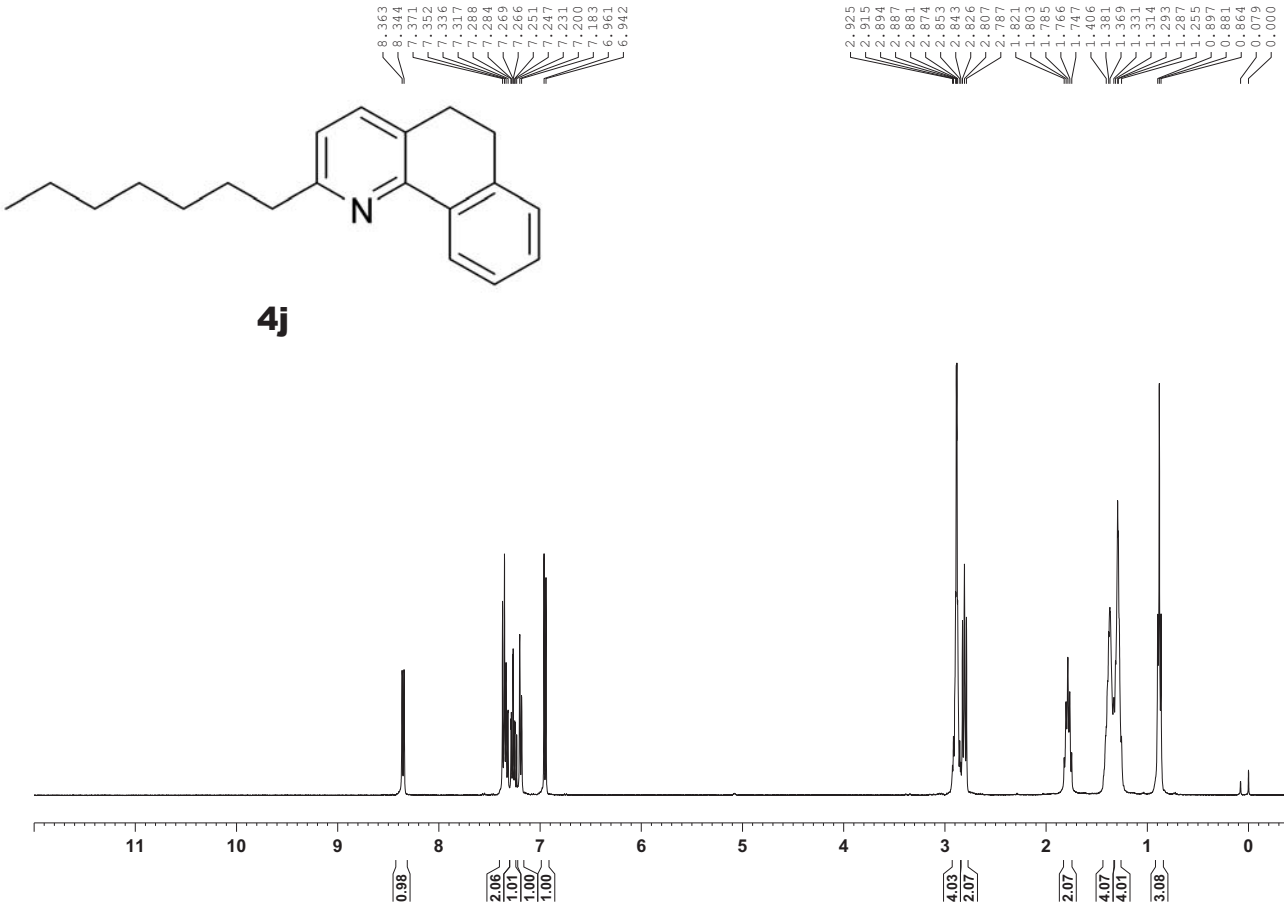
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7.247
7.231
7.200
7.183
6.961
6.942
2.925
2.915
2.894
2.887
2.881
2.874
2.853
2.843
2.826
2.807
2.787
1.821
1.803
1.785
1.766
1.747
1.406
1.381
1.369
1.331
1.314
1.293
1.287
1.255
0.897
0.881
0.864
0.079
0.000
N
4j
11
10
9
8
7
6
5
4
3
2
1
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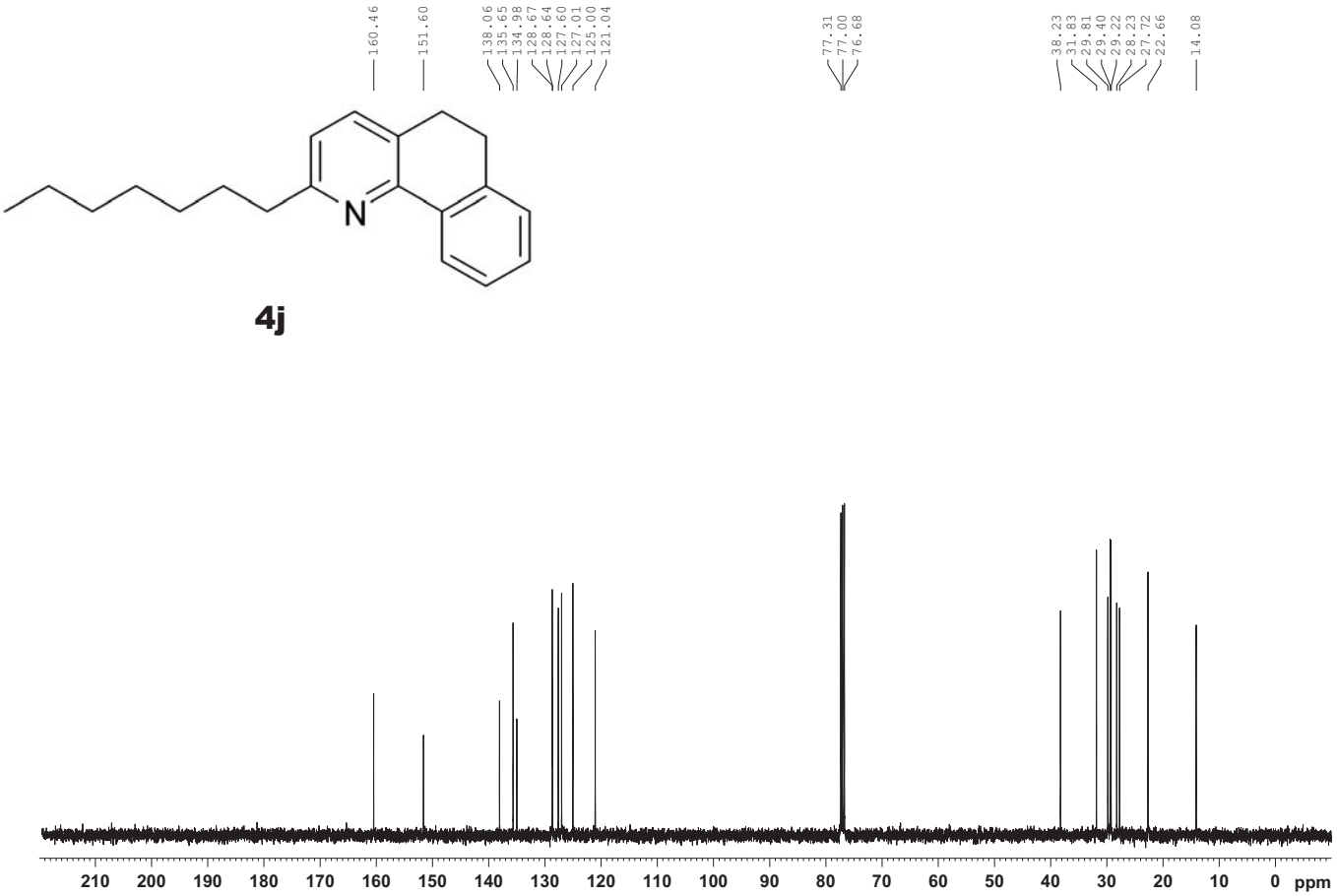
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77.00
76.68
38.23
31.83
29.81
29.40
29.22
28.23
27.72
22.66
14.08
N
4j
210
200
190
180
170
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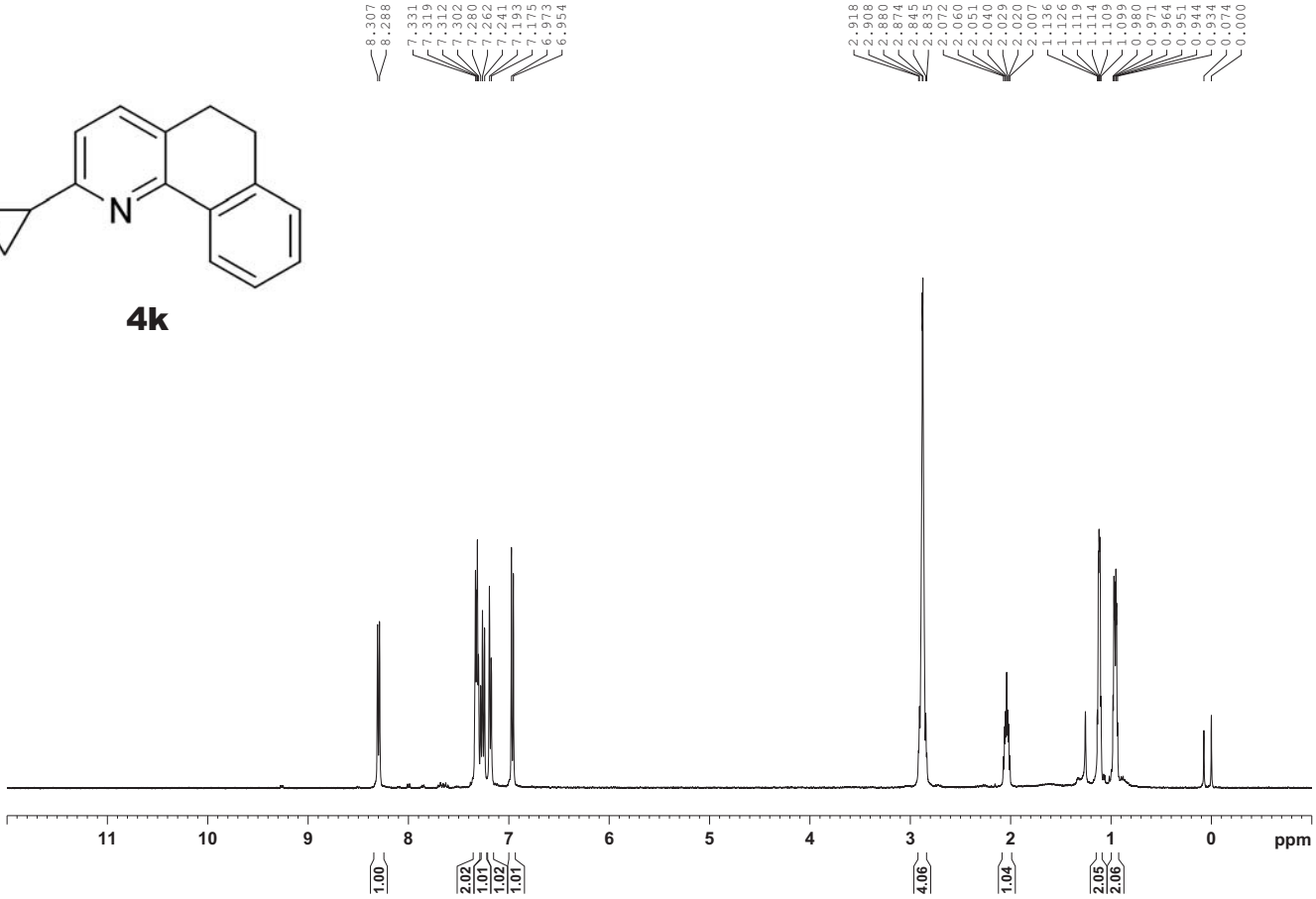
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

4k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7.241
7.193
7.175
6.973
6.954
2.918
2.908
2.880
2.874
2.845
2.835
2.072
2.060
2.051
2.040
2.029
2.020
2.007
1.136
1.126
1.119
1.114
1.109
1.099
1.080
0.971
0.964
0.951
0.944
0.934
0.074
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
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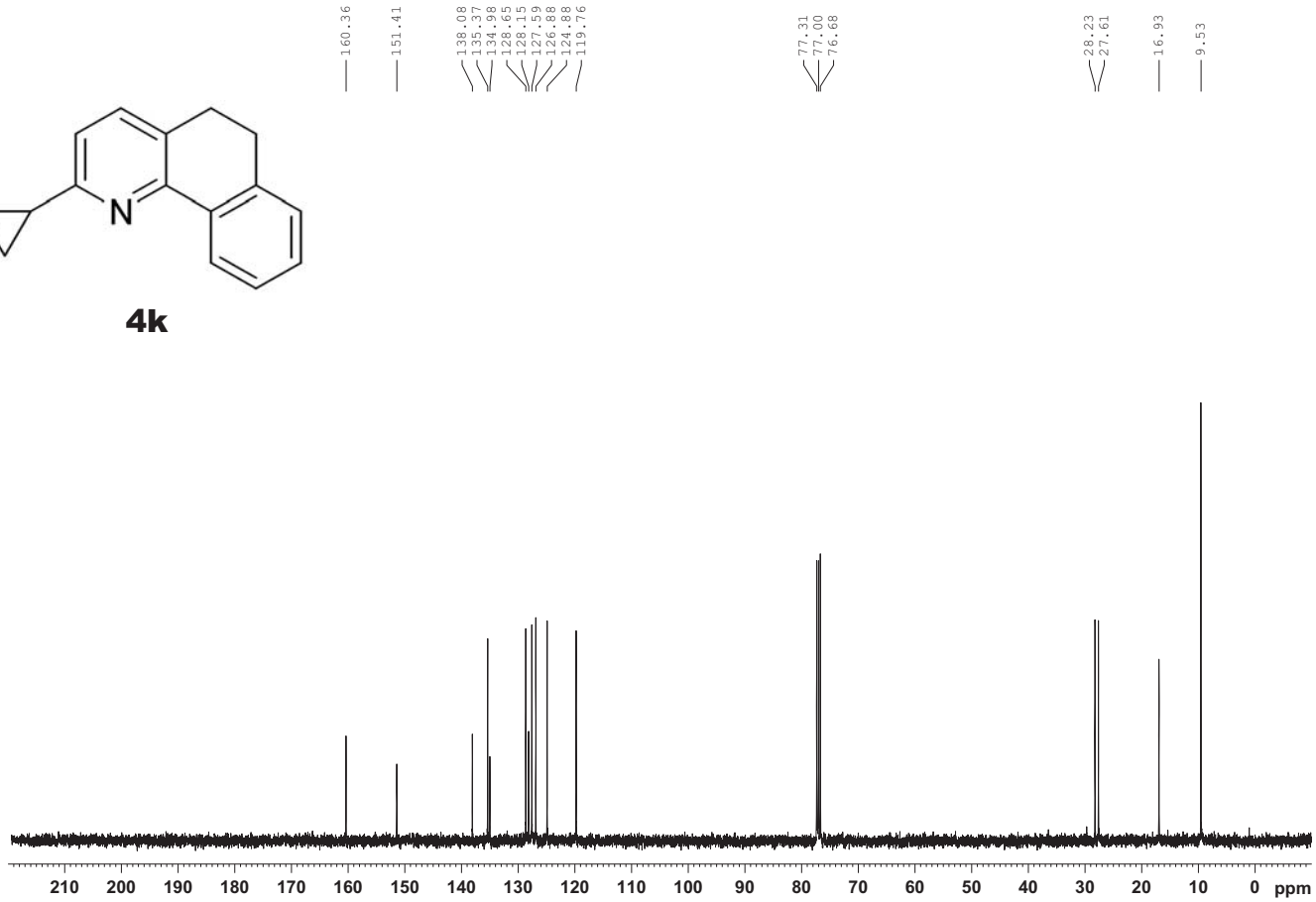
2.02
1.01
1.02
1.01
4.06
1.04
2.05
2.06

4k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128.65
128.15
127.59
126.88
124.88
119.76
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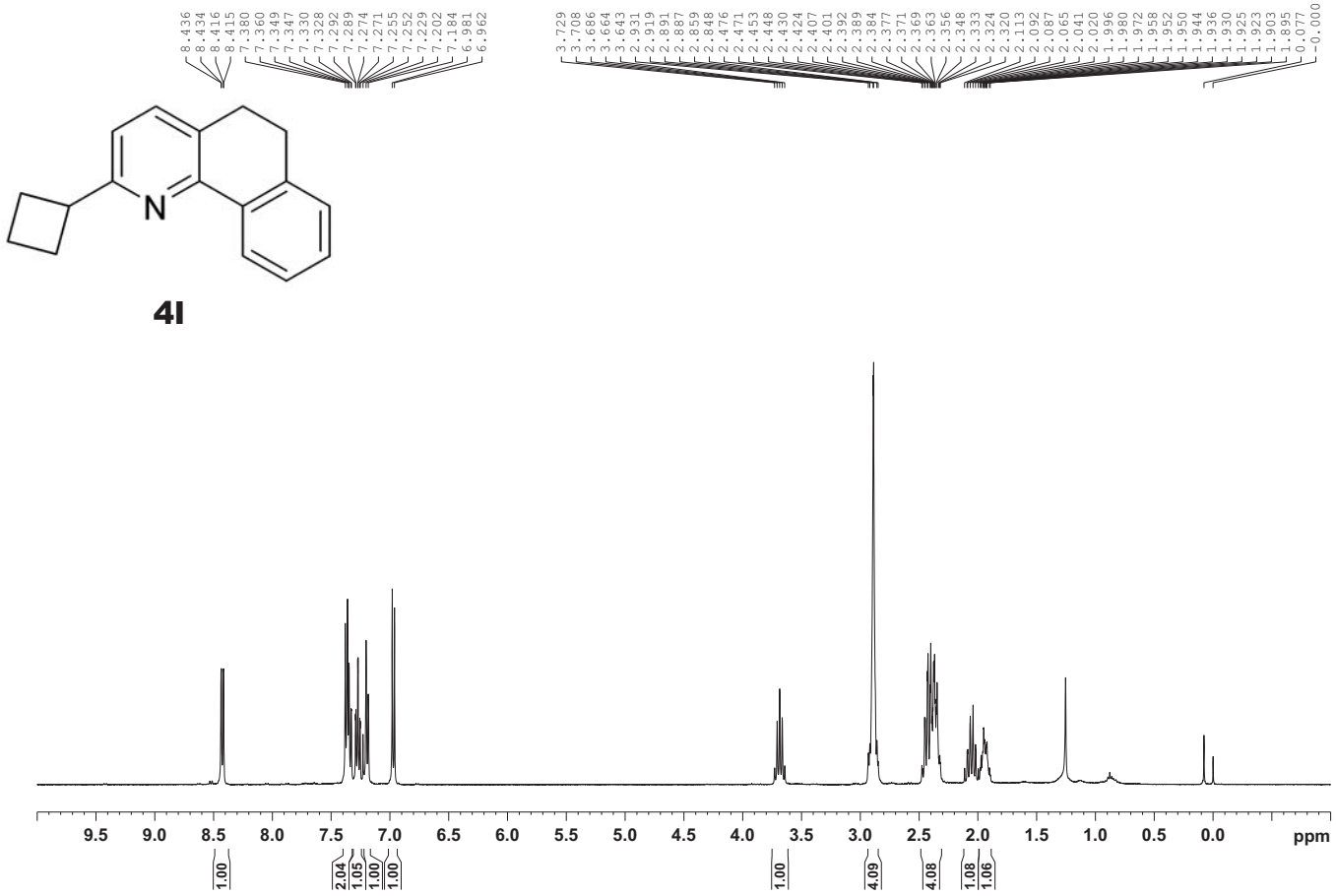
77.00
76.68
28.23
27.61
16.93
9.53
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.347
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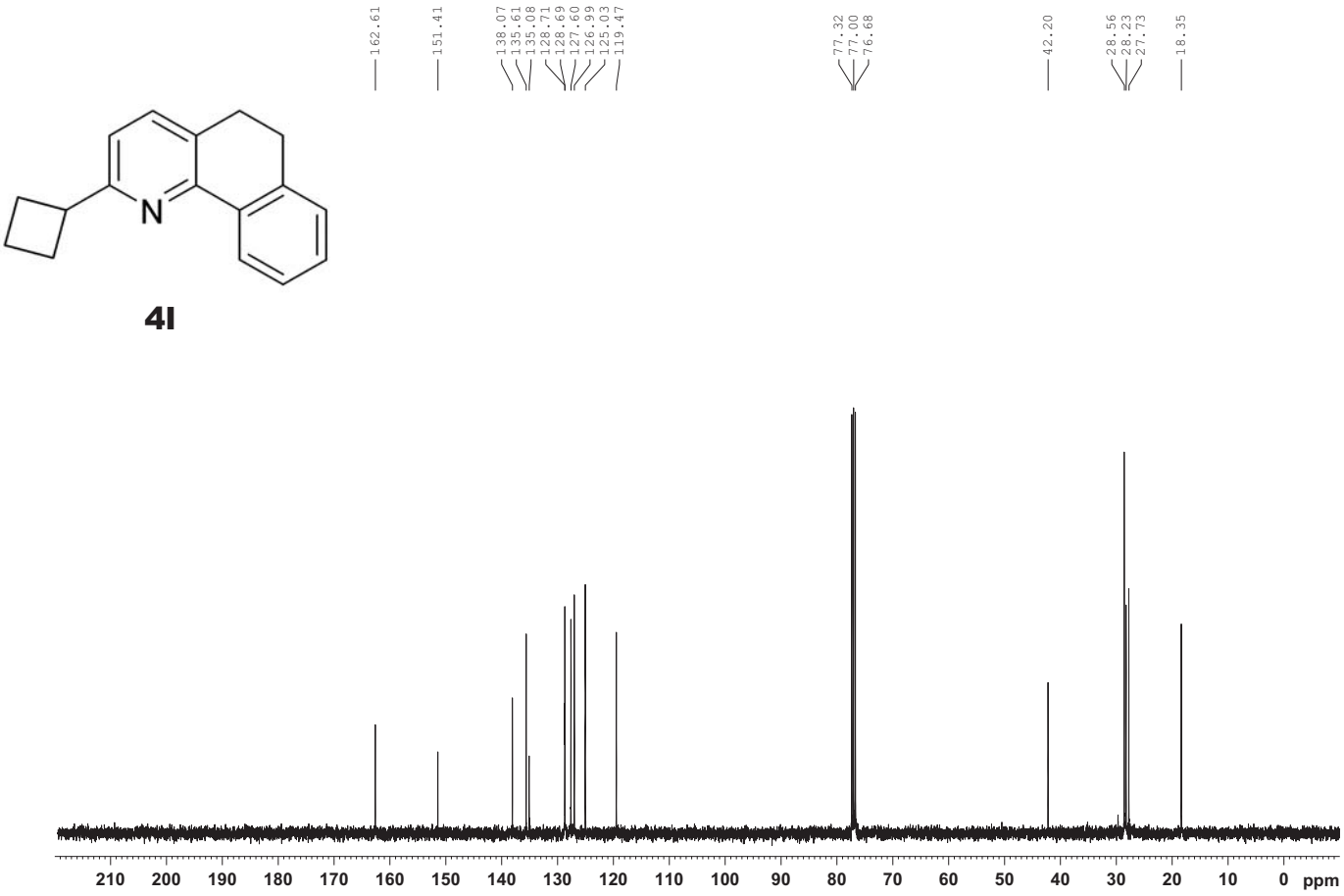
7.292
7.289
7.274
7.271
7.255
7.252
7.229
7.202
7.184
6.981
6.962
3.729
3.708
3.686
3.664
3.643
2.931
2.919
2.891
2.887
2.859
2.848
2.476
2.471
2.453
2.448
2.430
2.424
2.407
2.401
2.392
2.389
2.384
2.377
2.371
2.369
2.363
2.356
2.348
2.333
2.324
2.320
2.113
2.092
2.087
2.065
2.041
2.020
1.996
1.980
1.972
1.958
1.952
1.950
1.944
1.936
1.930
1.925
1.923
1.903
1.895
0.077
-0.000
N
4l
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 ppm
1.00
2.04
1.05
1.00
1.00
1.00
4.09
4.08
1.08
1.06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77.32
77.00
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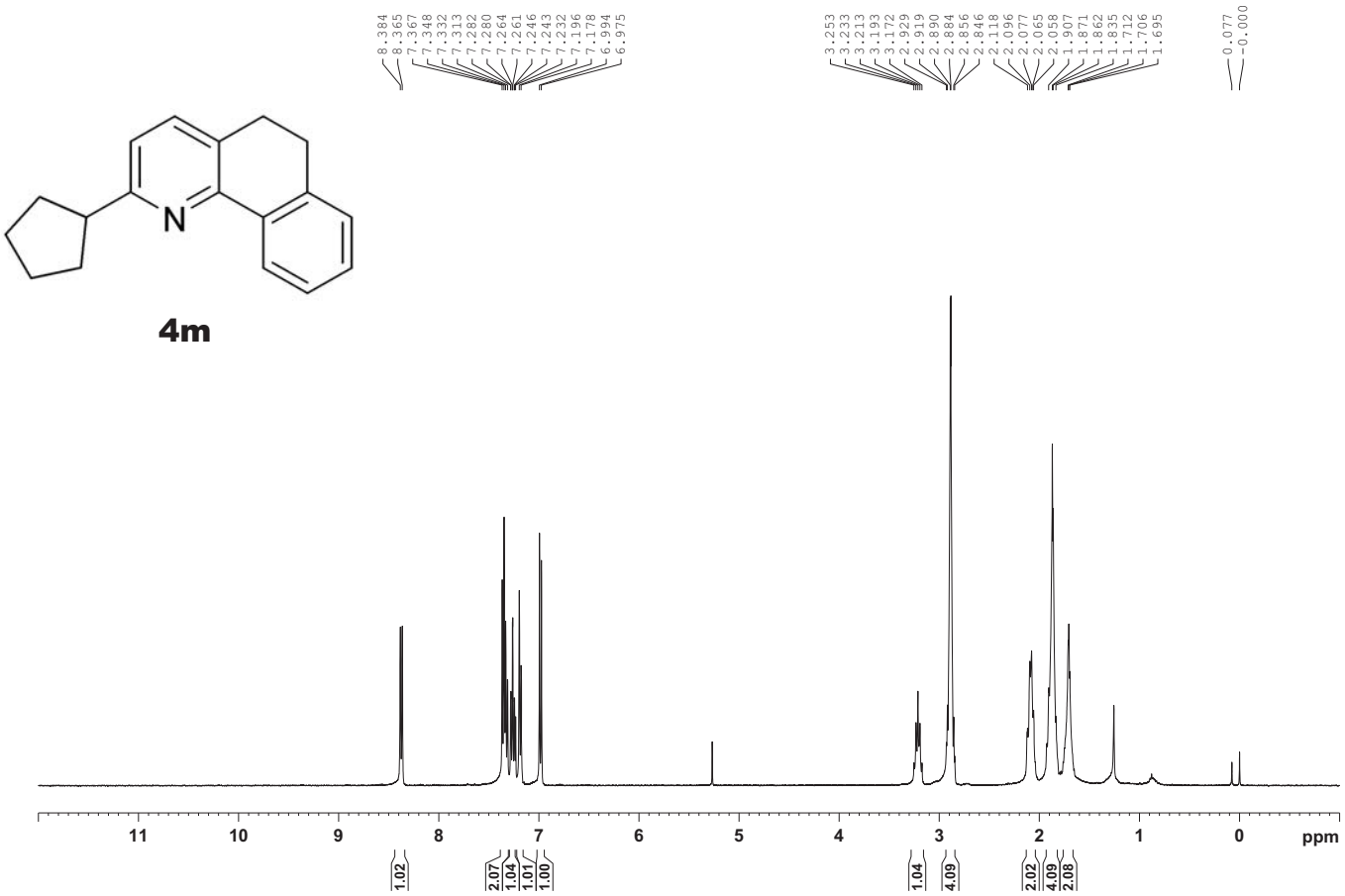
28.23
27.73
18.35
N
4l
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.261
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7.196
7.178
6.994
6.975
3.253
3.233
3.213
3.193
3.172
2.929
2.919
2.890
2.884
2.856
2.846
2.118
2.096
2.077
2.065
2.058
1.907
1.871
1.862
1.835
1.712
1.706
1.695
0.077
-0.000
N
4m
11
10
9
8
7
6
5
4
3
2
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ppm
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2.07
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1.01
1.00
1.04
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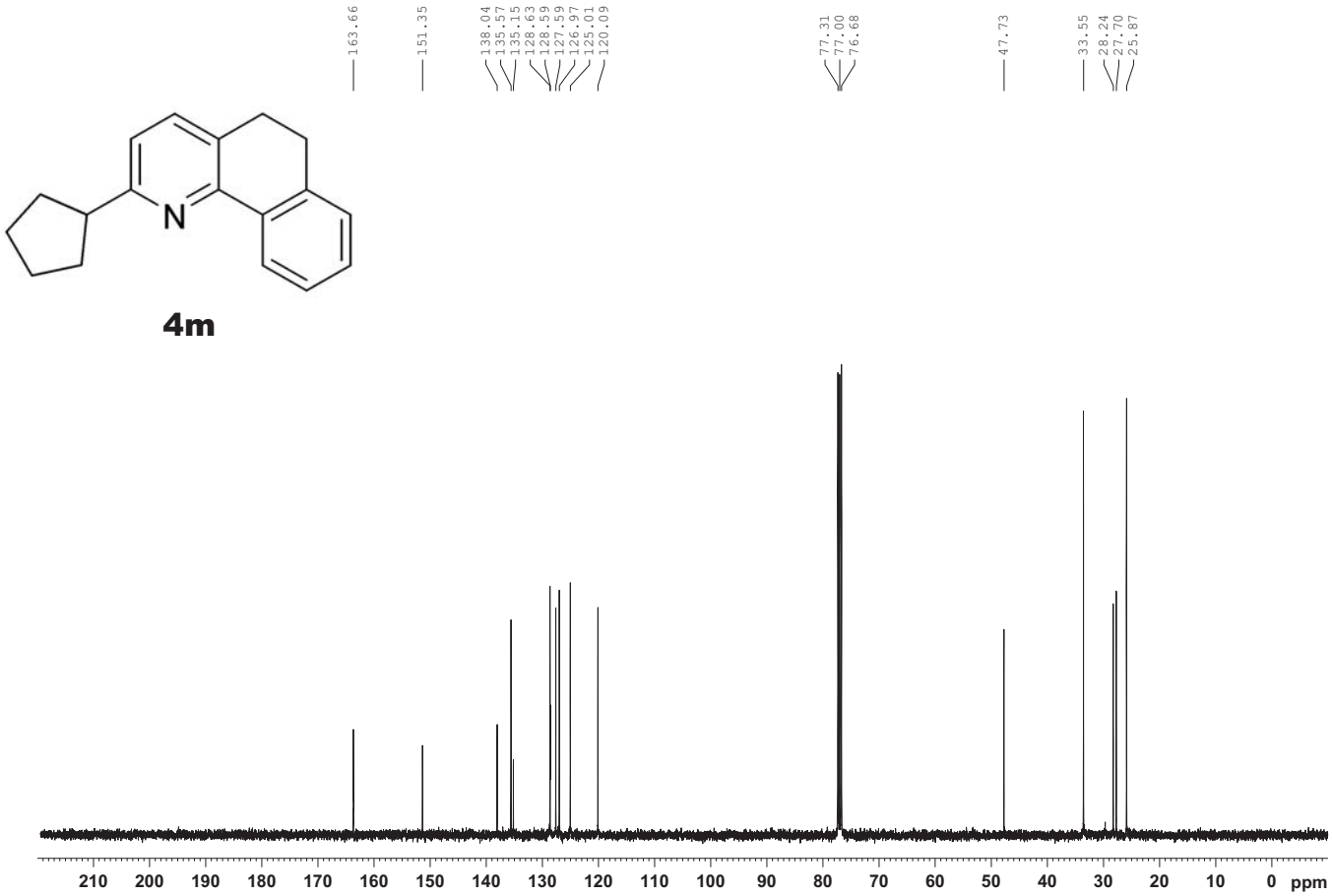
2.02
4.09
2.08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127.59
126.97
125.01
120.09
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77.00
76.68
47.73
33.55
28.24
27.70
25.87
N
4m
210
200
190
180
170
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150
140
130
120
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80
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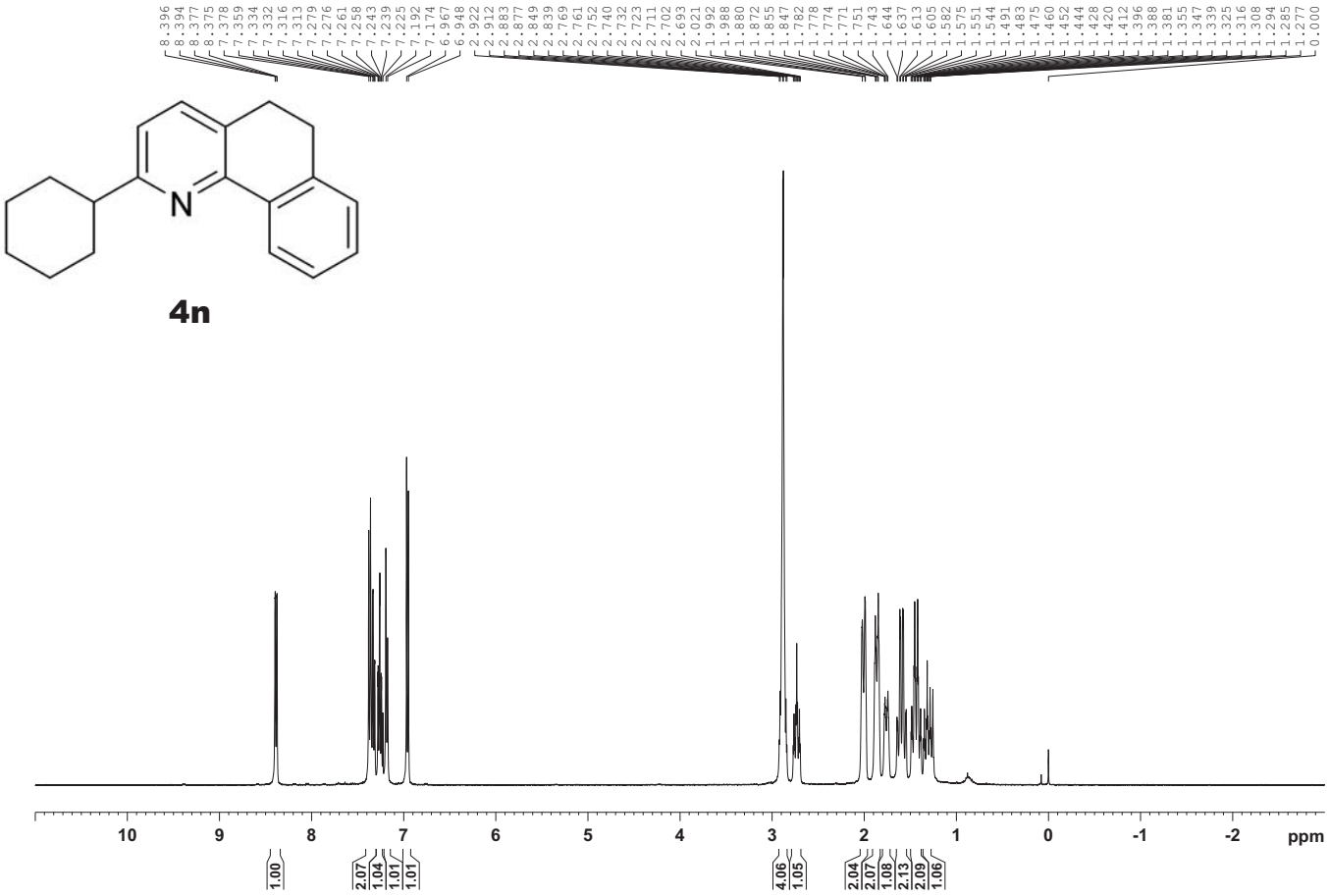
10
0
ppm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7.276
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7.258
7.243
7.239
7.225
7.192
7.174
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6.948
2.922
2.912
2.883
2.877
2.849
2.839
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2.761
2.752
2.740
2.732
2.723
2.711
2.702
2.693
2.021
1.992
1.988
1.880
1.872
1.855
1.847
1.782
1.778
1.774
1.771
1.751
1.743
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1.613
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1.582
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1.483
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1.452
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1.412
1.396
1.388
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1.355
1.347
1.339
1.325
1.316
1.308
1.294
1.285
1.277
0.000
N
4n
10
9
8
7
6
5
4
3
2
1
0
-1
-2
ppm
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1.01
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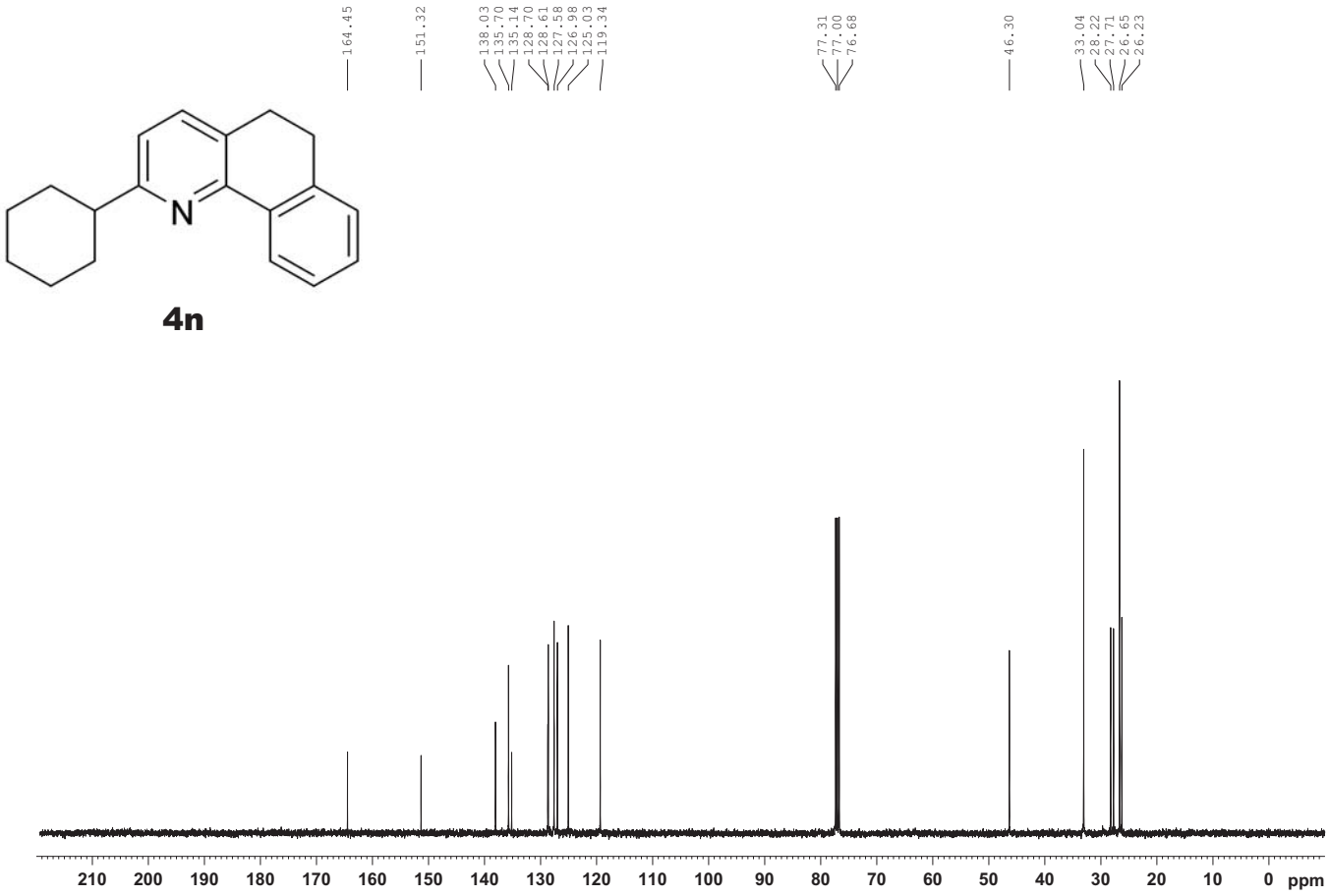
2.07
1.08
2.13
2.09
1.06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127.58
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125.03
119.34
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77.00
76.68
46.30
33.04
28.22
27.71
26.65
26.23
N
4n
210
200
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140
130
120
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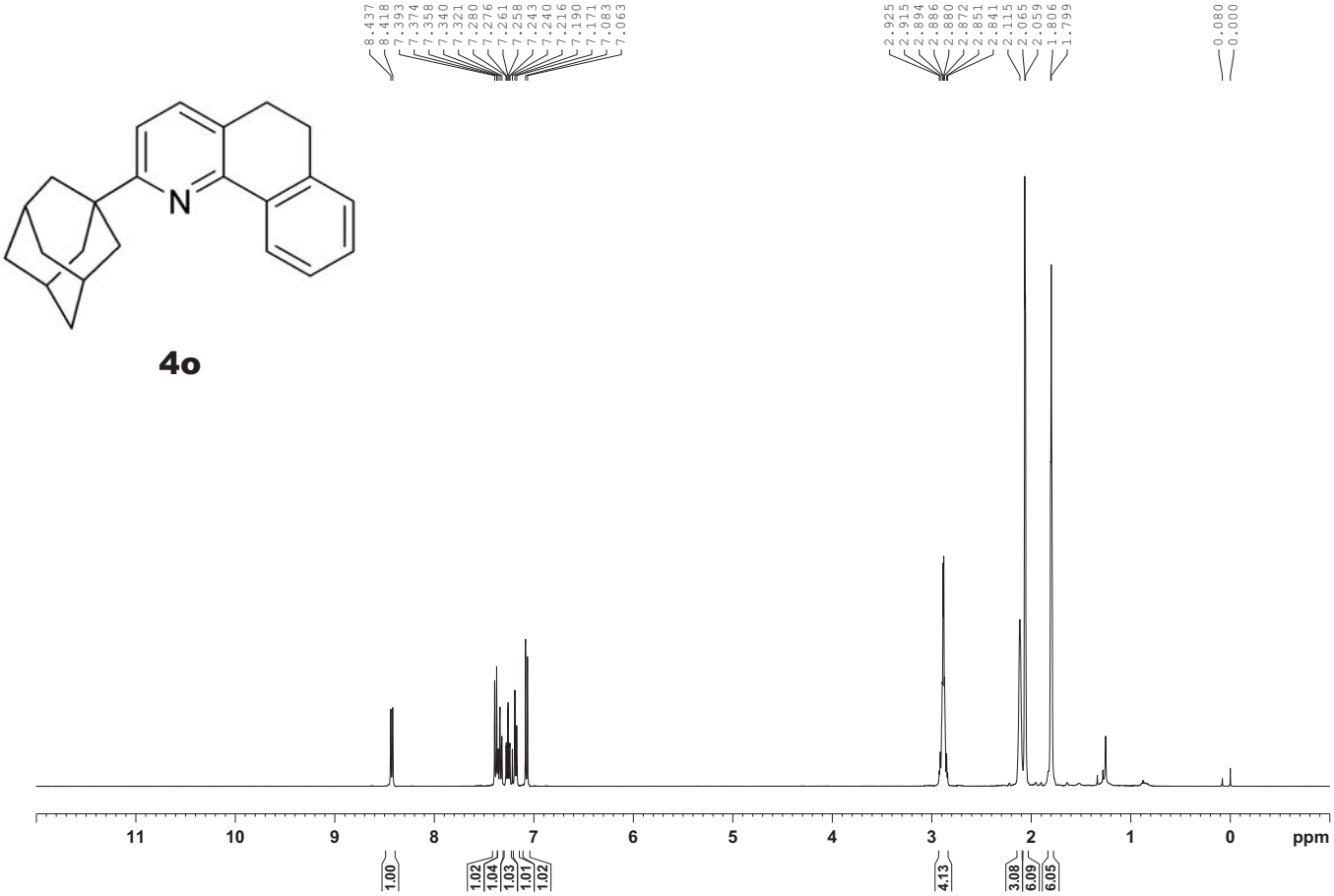
90
80
70
60
50
40
30
20
10
0
ppm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7.276
7.261
7.258
7.243
7.240
7.216
7.190
7.171
7.083
7.063
2.925
2.915
2.894
2.886
2.880
2.872
2.851
2.841
2.115
2.065
2.059
1.806
1.799
0.080
0.000
N
4o
11 10 9 8 7 6 5 4 3 2 1 0 ppm
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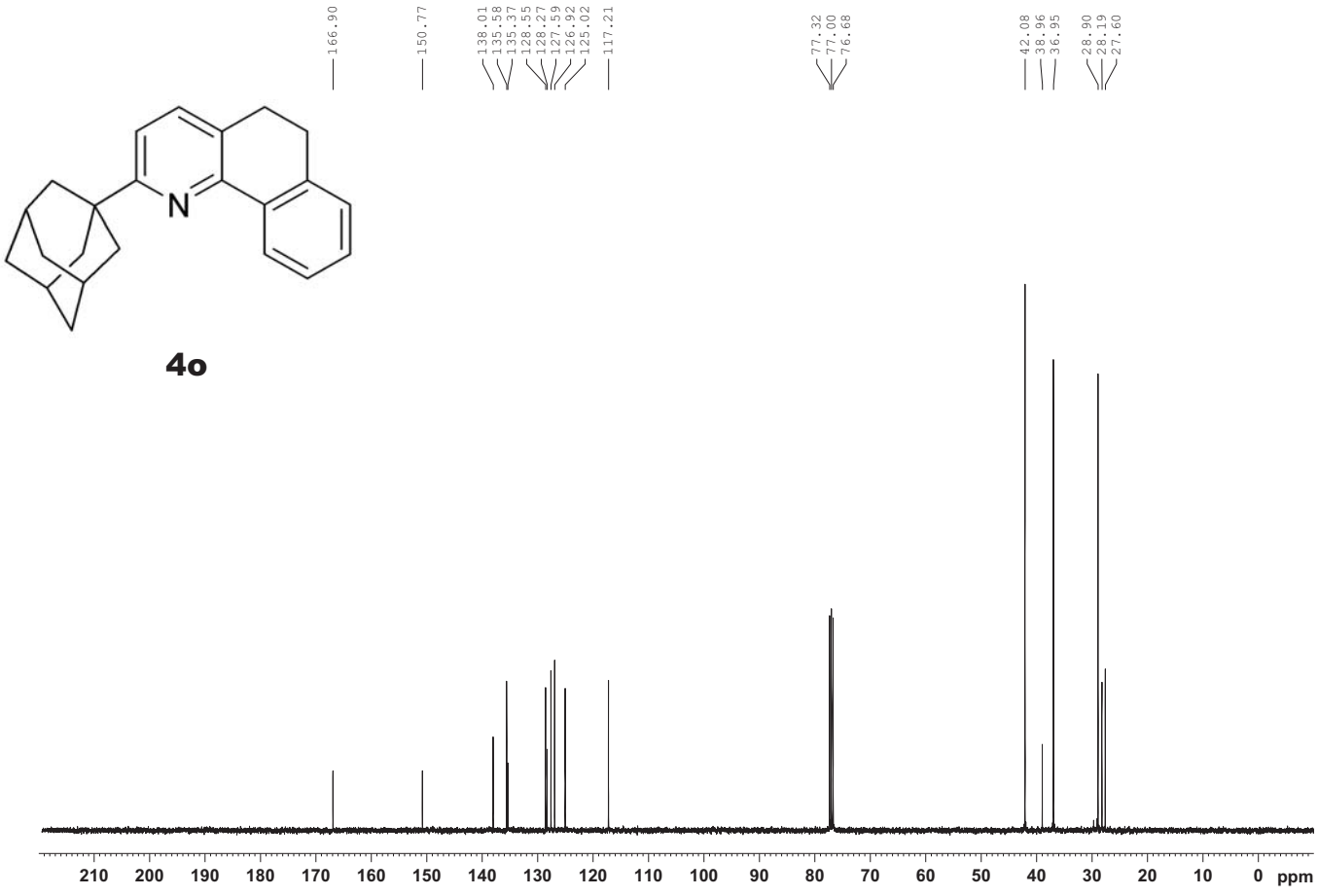
1.02
1.04
1.03
1.01
1.02
4.13
3.08
6.09
6.05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128.27
127.59
126.92
125.02
117.21
77.32
77.00
76.68
42.08
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28.90
28.19
27.60
N
4o
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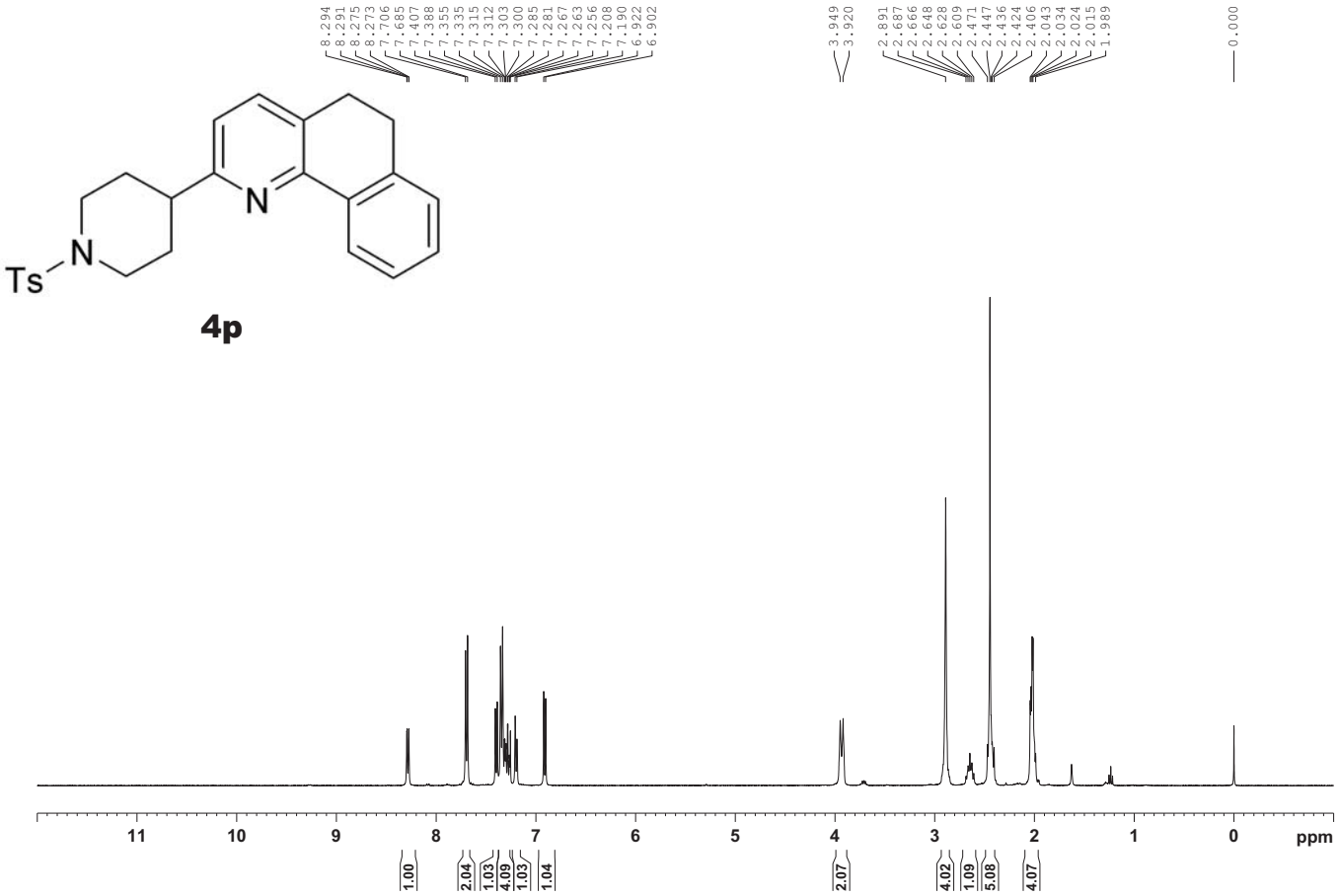
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7.312
7.303
7.300
7.285
7.281
7.267
7.263
7.256
7.208
7.190
6.922
6.902
3.949
3.920
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2.666
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2.628
2.609
2.471
2.447
2.436
2.424
2.406
2.043
2.034
2.024
2.015
1.989
0.000
N
N
Ts
4p
11 10 9 8 7 6 5 4 3 2 1 0 ppm
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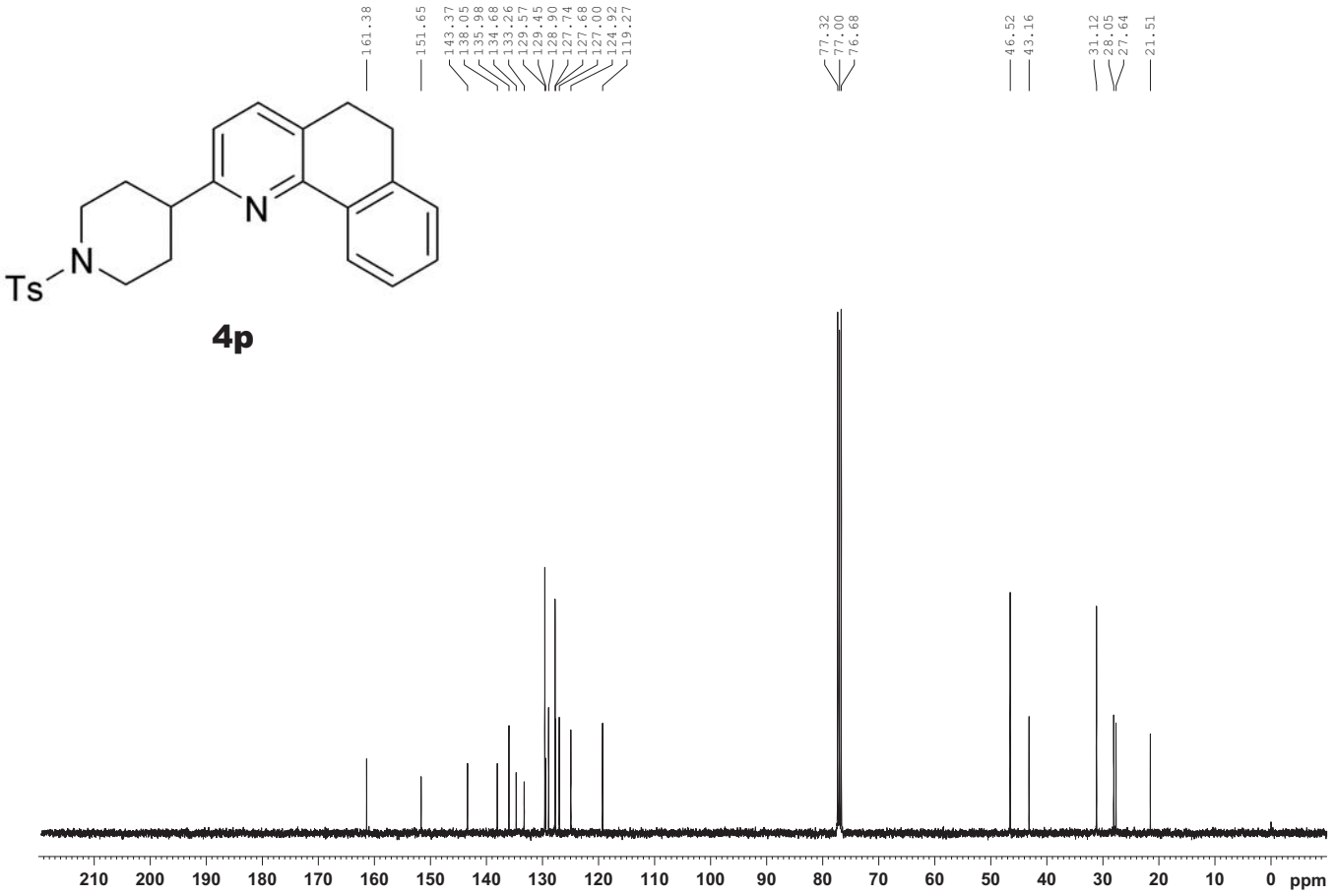
1.03
4.09
1.03
1.04
2.07
4.02
1.09
5.08
4.07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124.92
119.27
77.32
77.00
76.68
46.52
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27.64
21.51
N
N
Ts
4p
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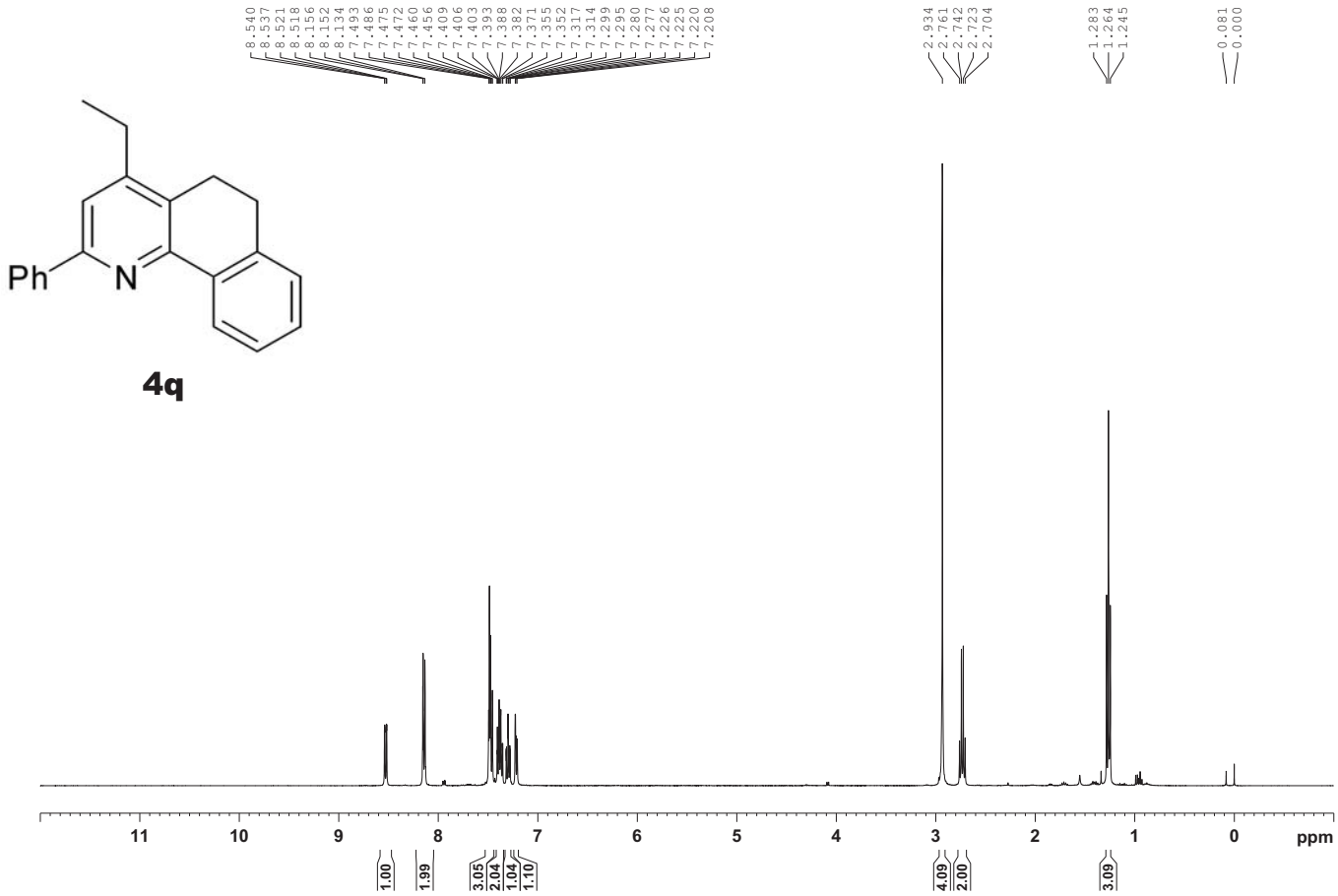
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7.456
7.409
7.406
7.403
7.393
7.388
7.382
7.371
7.355
7.352
7.317
7.314
7.299
7.295
7.280
7.277
7.226
7.225
7.220
7.208
2.934
2.761
2.742
2.723
2.704
1.283
1.264
1.245
0.081
0.000
Ph
N
4q
11
10
9
8
7
6
5
4
3
2
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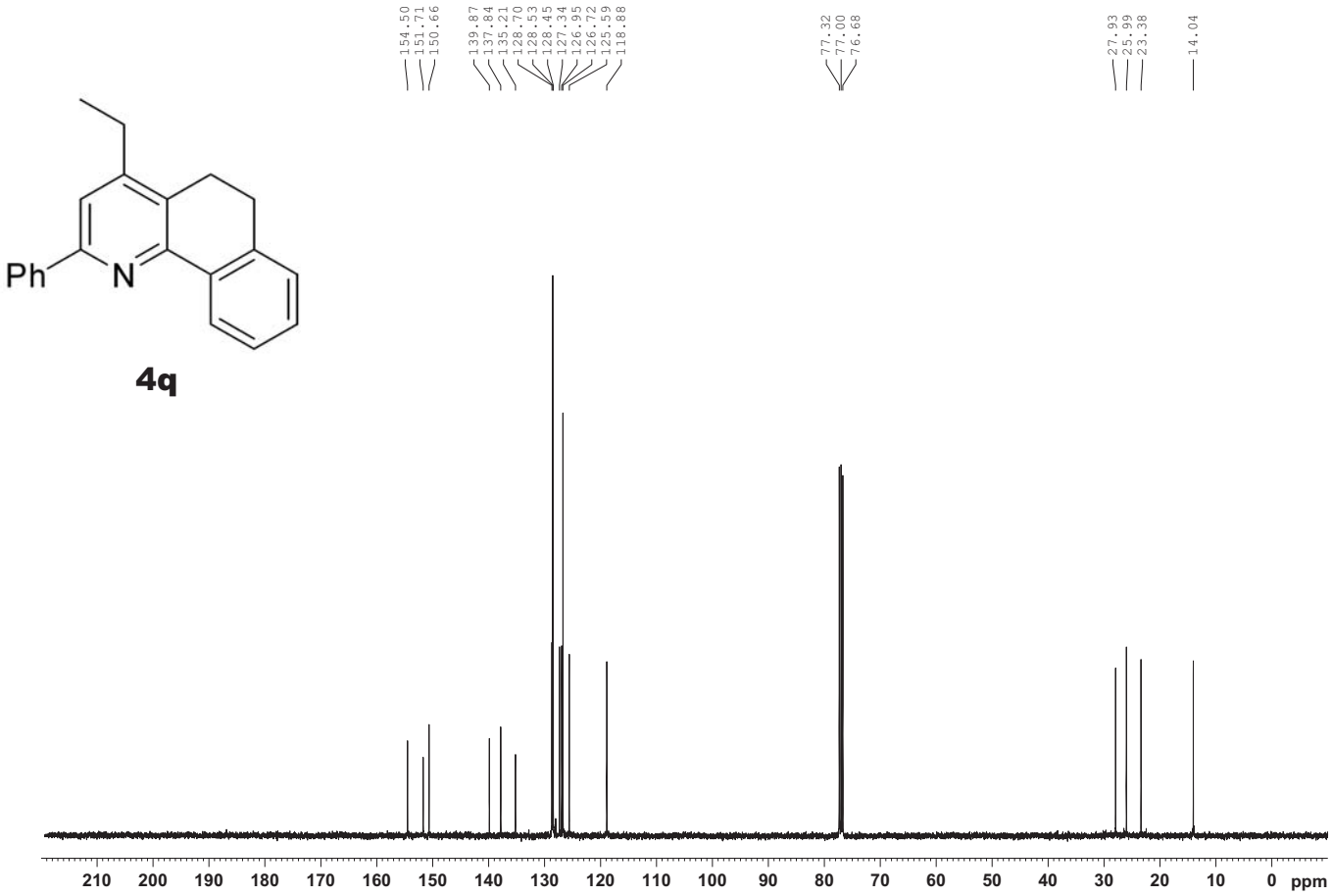
ppm
1.00
1.99
3.05
2.04
1.04
1.10
4.09
2.00
3.09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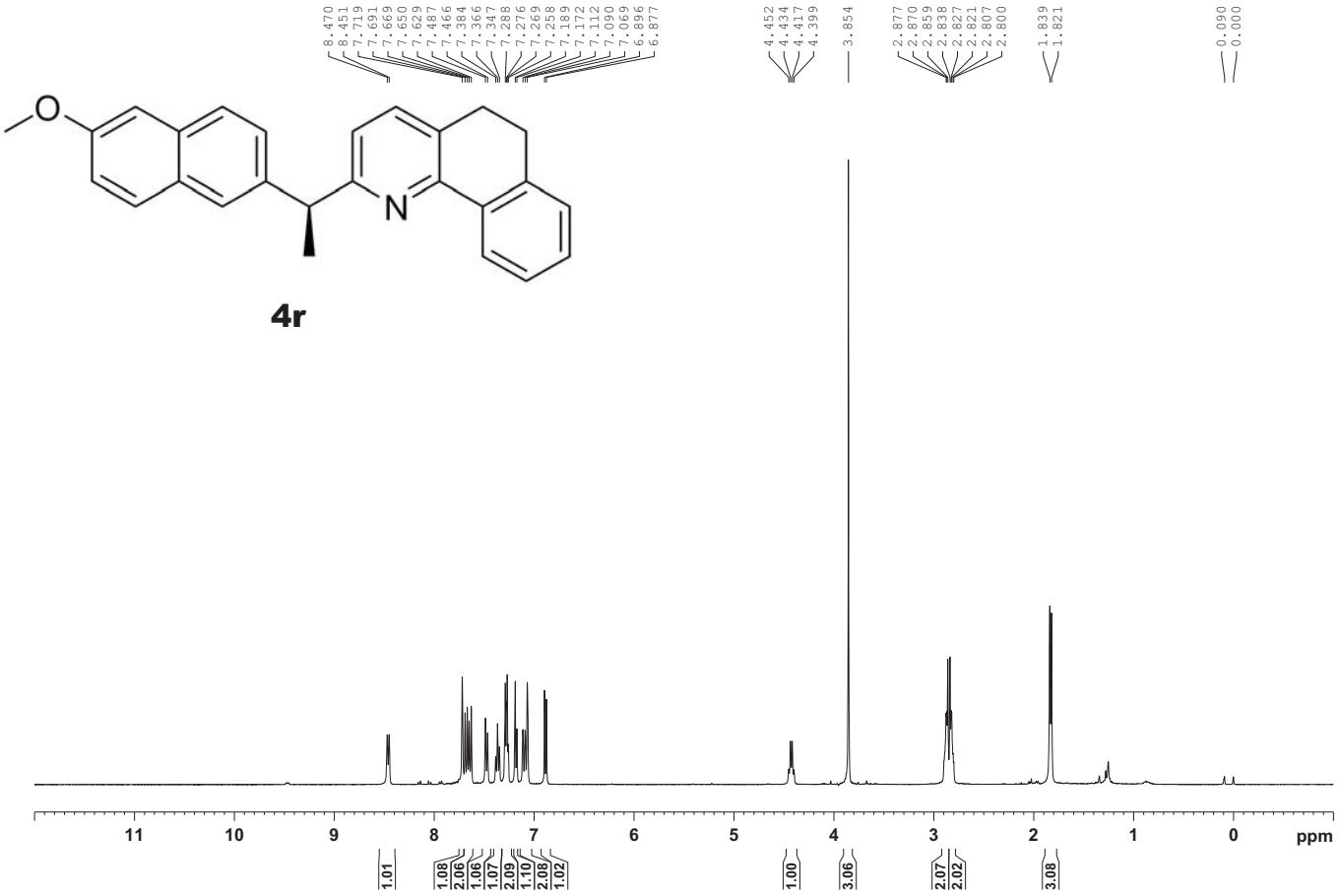
137.84
135.21
128.70
128.53
128.45
127.34
126.95
126.72
125.59
118.88
77.32
77.00
76.68
27.93
25.99
23.38
14.04
Ph
N
4q
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.669
7.650
7.629
7.487
7.466
7.384
7.366
7.347
7.288
7.276
7.269
7.258
7.189
7.172
7.112
7.090
7.069
6.896
6.877
4.452
4.434
4.417
4.399
3.854
2.877
2.870
2.859
2.838
2.827
2.821
2.807
2.800
1.839
1.821
0.090
0.000
O
N
4r
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.01
1.08
2.06
1.06
1.07
2.09
1.10
2.08
1.02
1.00
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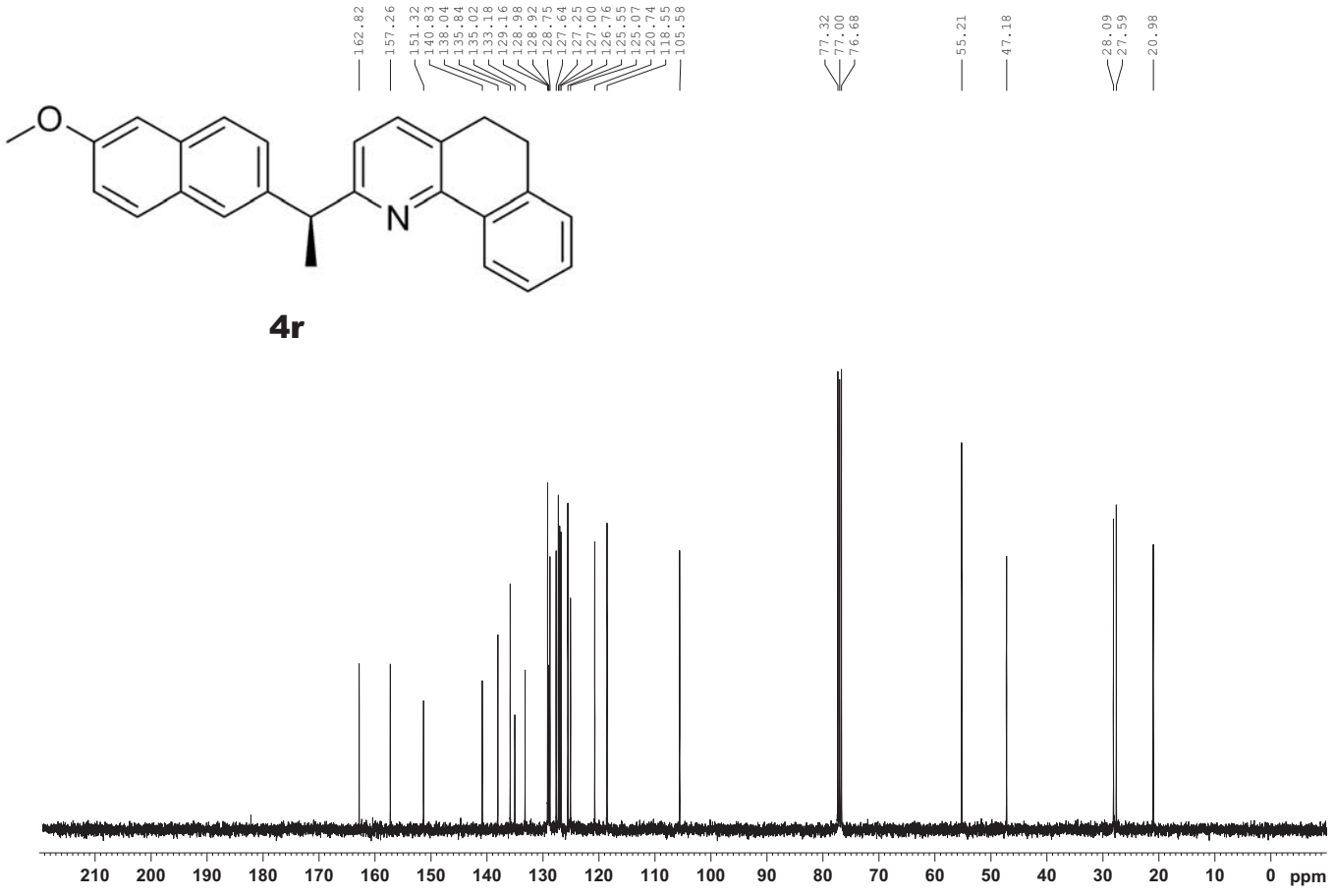
2.02
3.08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135.84
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128.92
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127.25
127.00
126.76
125.55
125.07
120.74
118.55
105.58
77.32
77.00
76.68
55.21
47.18
28.09
27.59
20.98
O
N
4r
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
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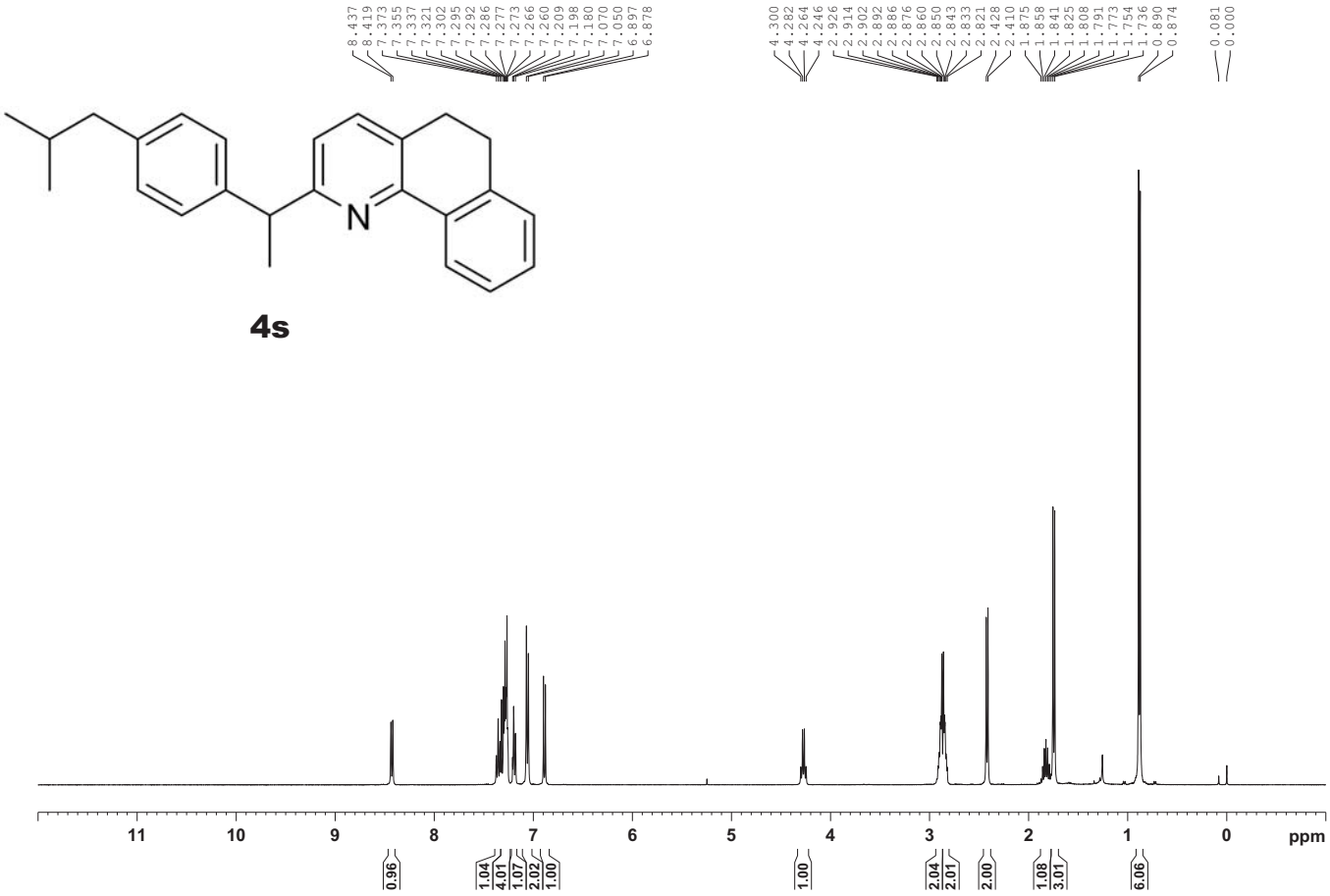
30
20
10
0
ppm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7.295
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7.273
7.266
7.260
7.209
7.198
7.180
7.070
7.050
6.897
6.878
4.300
4.282
4.264
4.246
2.926
2.914
2.902
2.892
2.886
2.876
2.860
2.850
2.843
2.833
2.821
2.428
2.410
1.875
1.858
1.841
1.825
1.808
1.791
1.773
1.754
1.736
0.890
0.874
0.081
0.000
N
4s
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.96
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1.07
2.02
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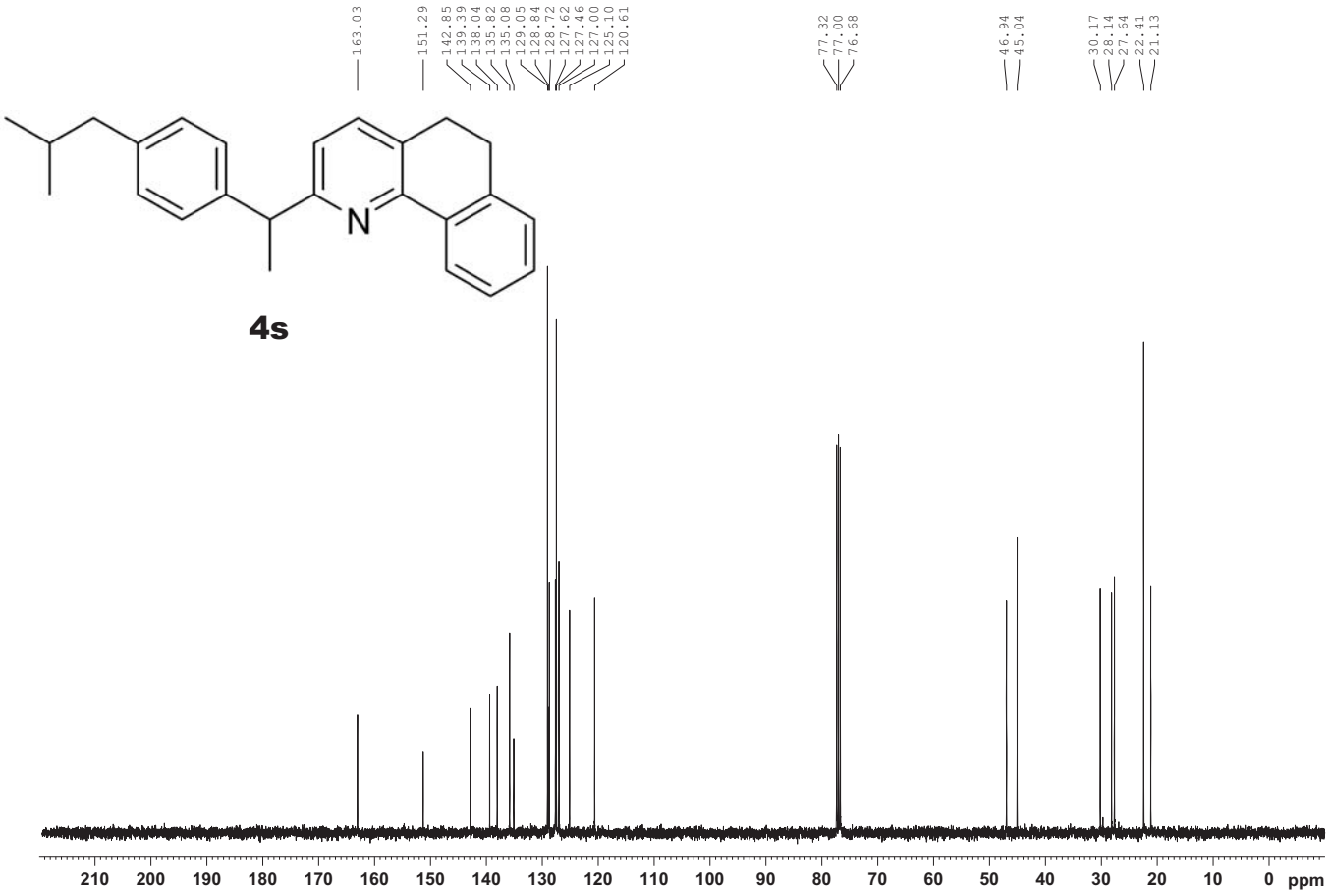
1.00
2.04
2.01
2.00
1.08
3.01
6.06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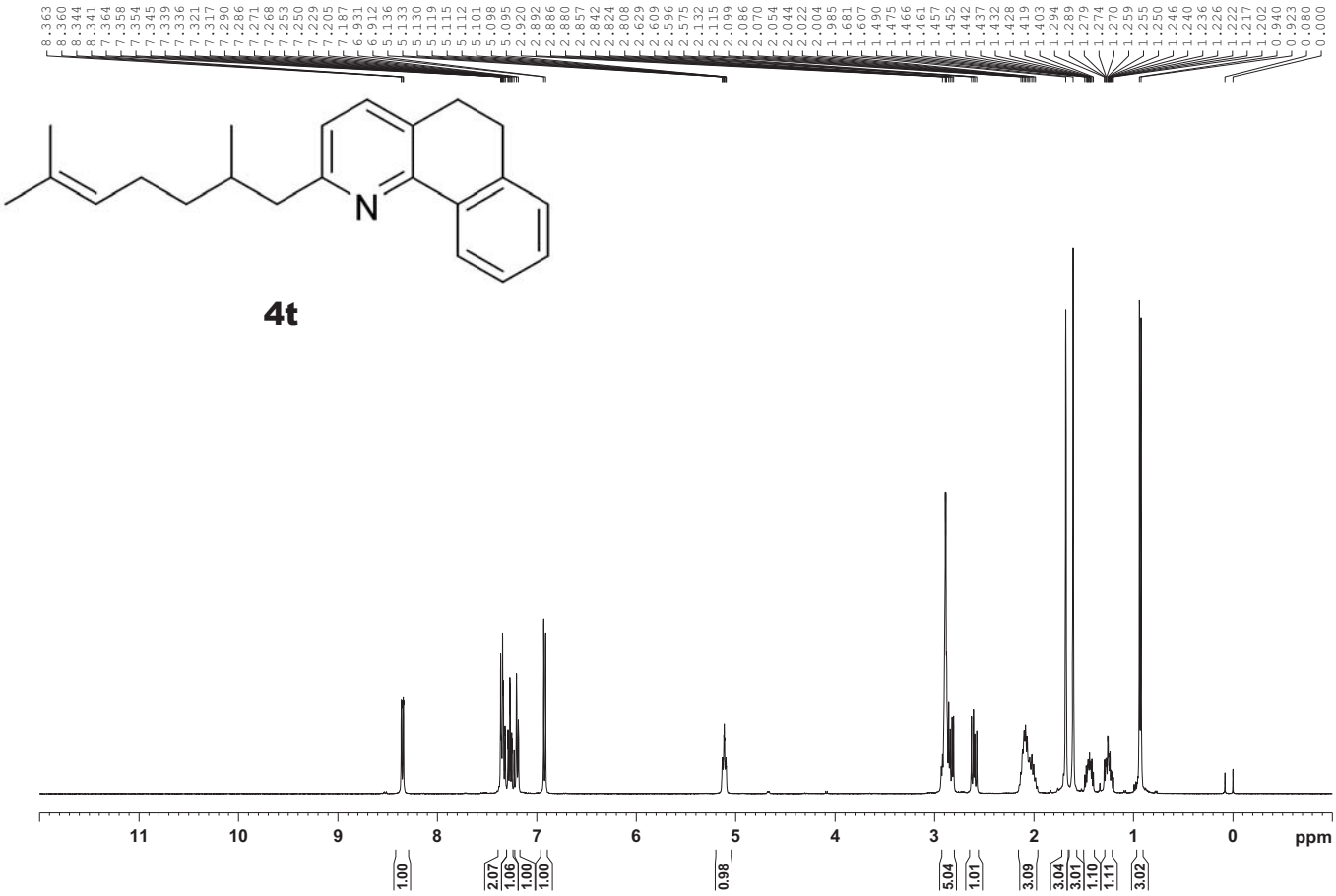
151.29
142.85
139.39
138.04
135.82
135.08
129.05
128.84
128.72
127.62
127.46
127.00
125.10
120.61
77.32
77.00
76.68
46.94
45.04
30.17
28.14
27.64
22.41
21.13
N
4s
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.345
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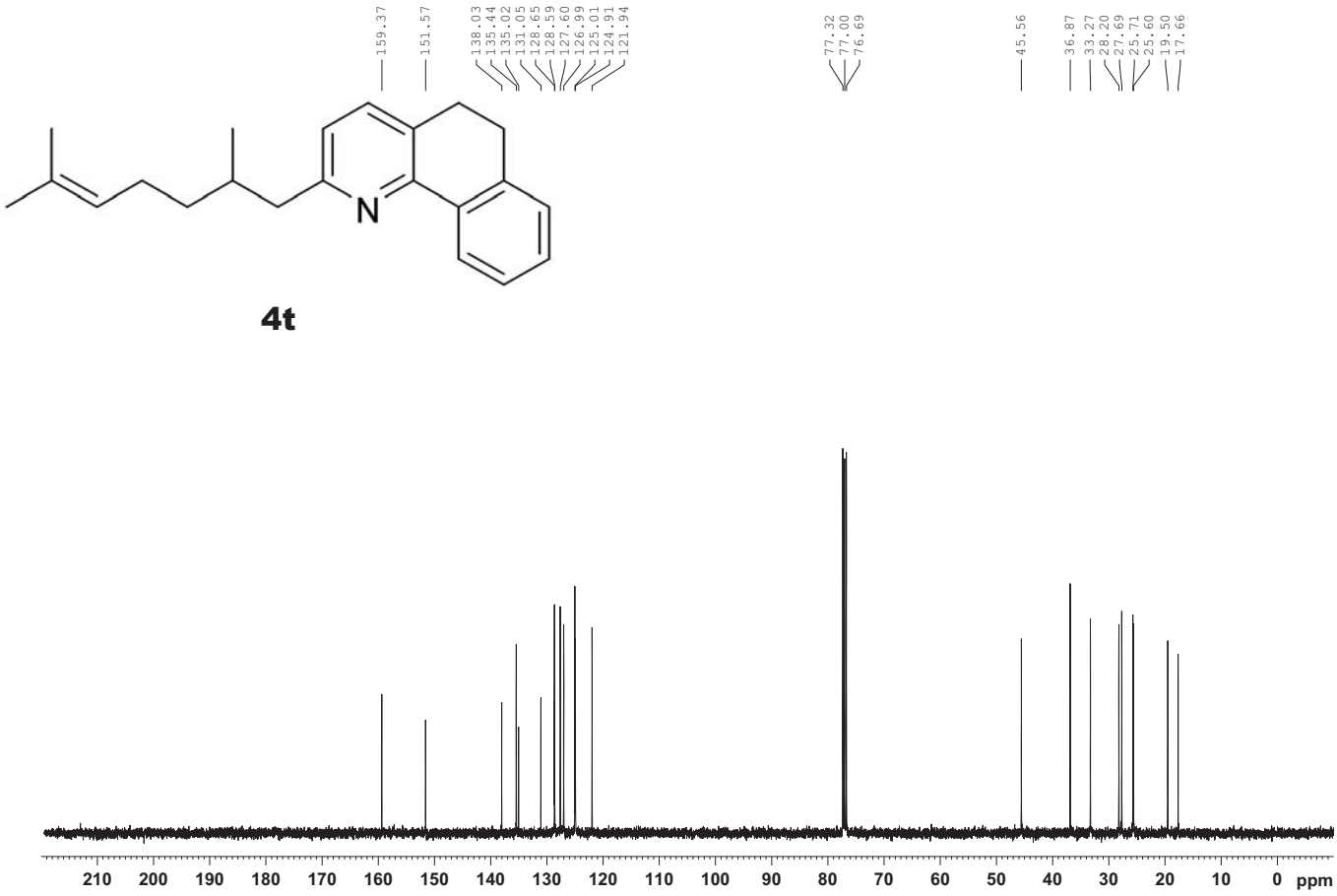
7.290
7.286
7.271
7.268
7.253
7.250
7.229
7.205
7.187
6.931
6.912
5.136
5.133
5.130
5.119
5.115
5.112
5.101
5.098
5.095
2.920
2.892
2.886
2.880
2.857
2.842
2.824
2.808
2.629
2.609
2.596
2.575
2.132
2.115
2.099
2.086
2.070
2.054
2.044
2.022
2.004
1.985
1.681
1.607
1.490
1.475
1.466
1.461
1.457
1.452
1.442
1.437
1.432
1.428
1.419
1.403
1.294
1.289
1.279
1.274
1.270
1.259
1.255
1.250
1.246
1.240
1.236
1.226
1.222
1.217
1.202
0.940
0.923
0.080
0.000
N
4t
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.07
1.06
1.00
1.00
0.98
5.04
1.01
3.09
3.04
3.01
1.10
1.11
3.02


159.37
151.57
138.03
135.44
135.02
131.05
128.65
128.59
127.60
126.99
125.01
124.91
121.94
77.32
77.00
76.69
45.56
36.87
33.27
28.20
27.69
25.71
25.60
19.50
17.66
N
4t
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
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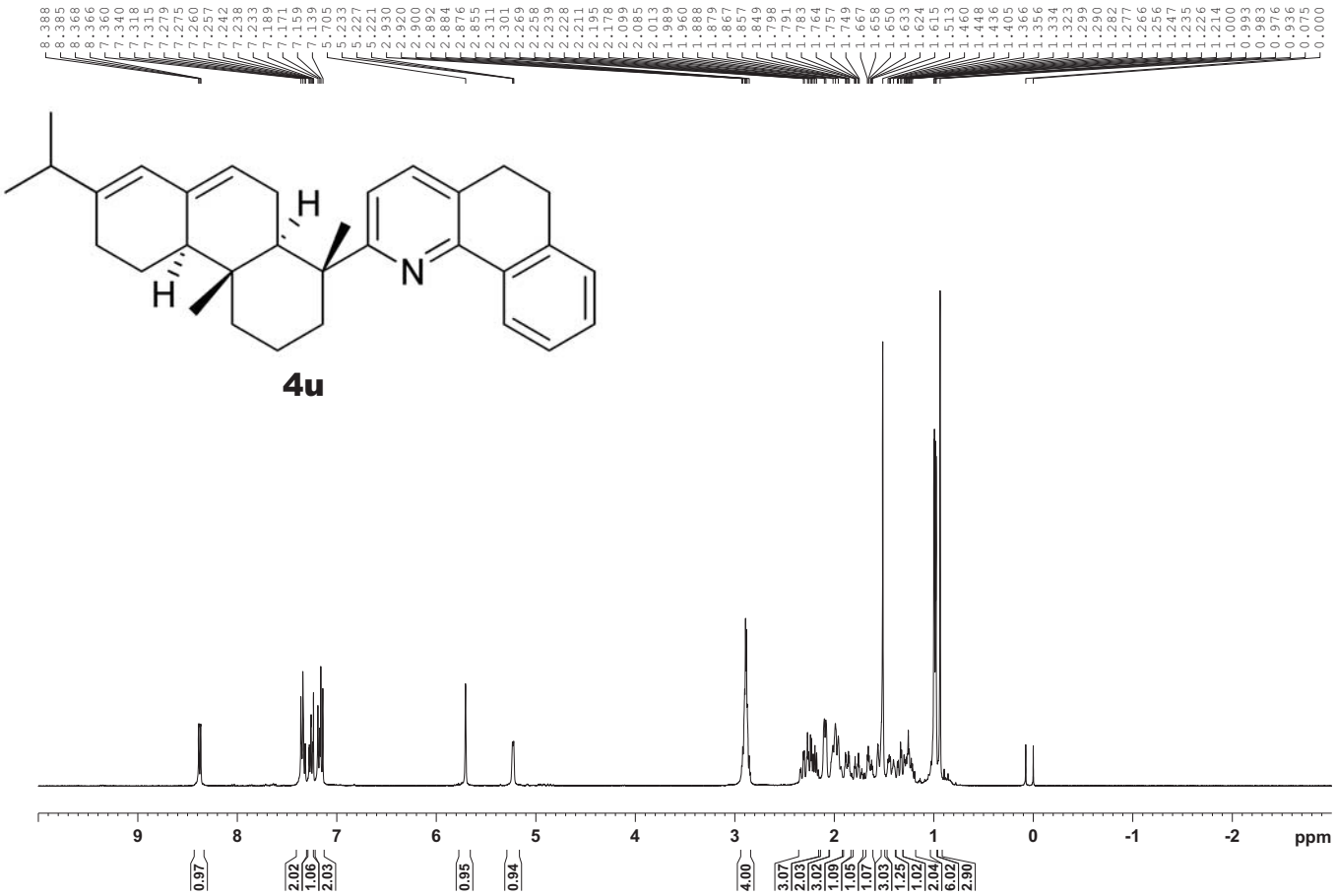
ppm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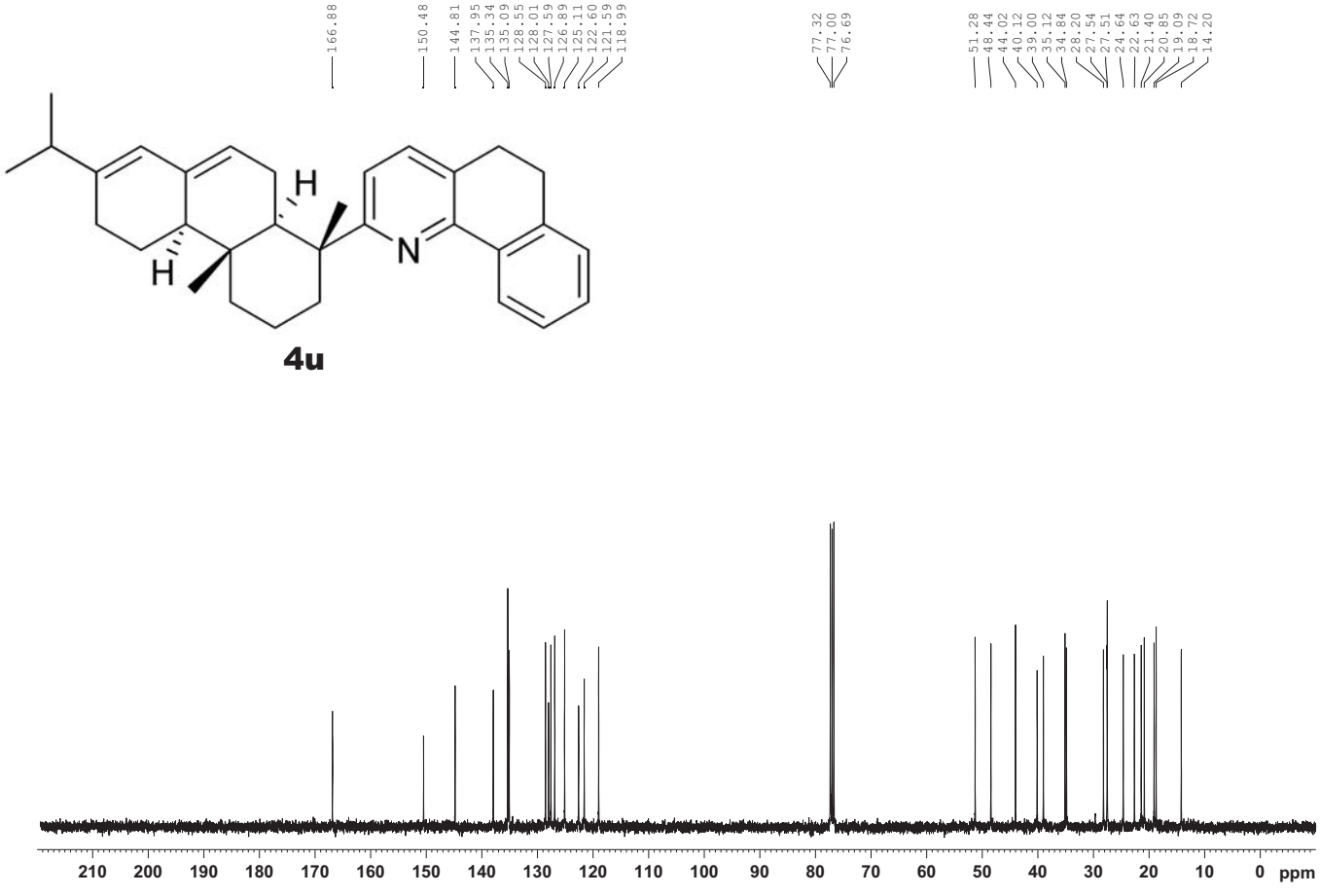
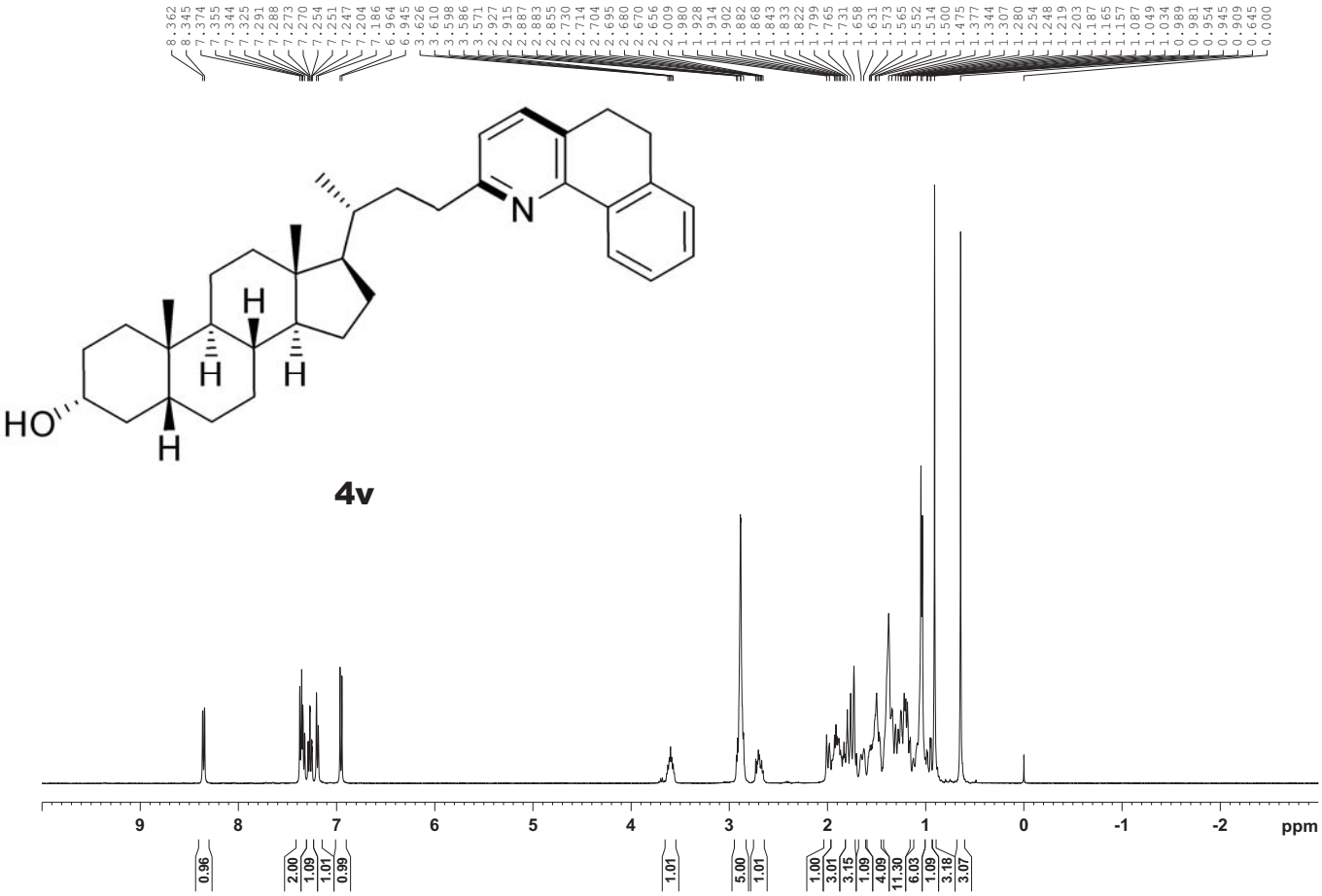
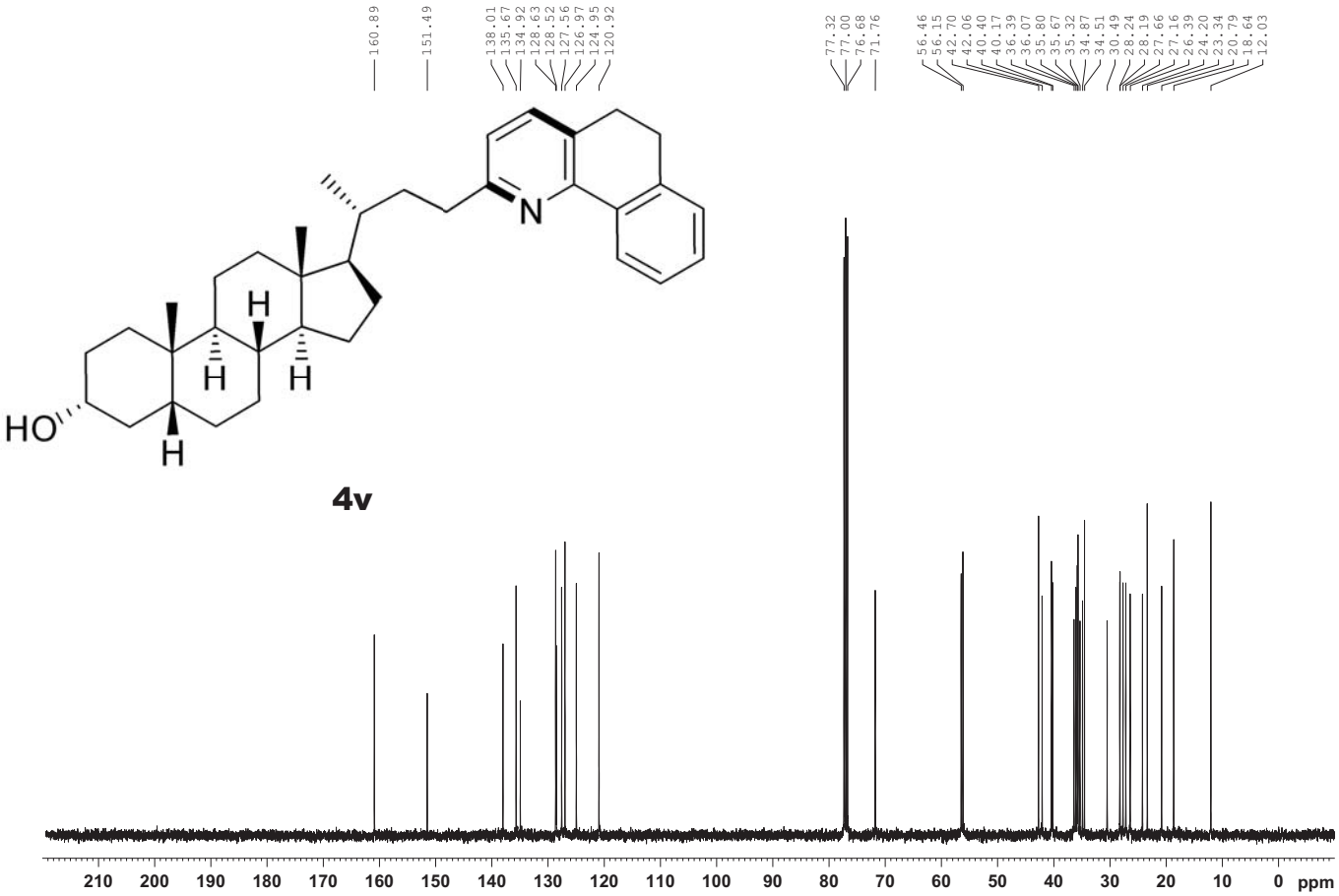
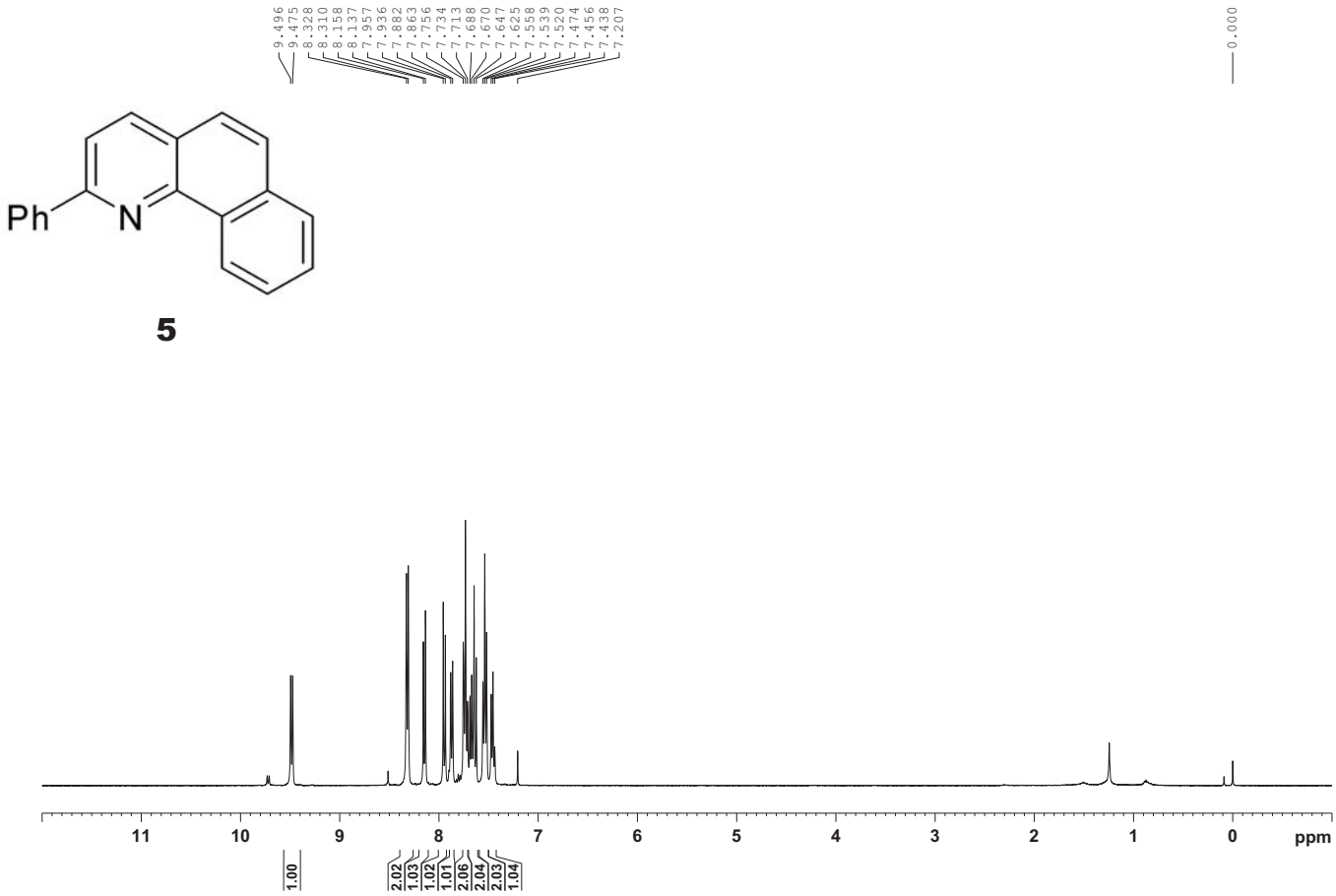
H
H
N
4u
9 8 7 6 5 4 3 2 1 0 -1 -2 ppm
0.97 2.02 1.06 2.03 0.95 0.94 4.00 3.07 2.03 3.02 1.09 1.05 1.07 3.03 1.25 1.02 2.04 6.02 2.90


166.88 150.48 144.81 137.95 135.34 135.09 128.55 128.01 127.59 126.89 125.11 122.60 121.59 118.99
77.32 77.00 76.69
51.28 48.44 44.02 40.12 39.00 35.12 34.84 28.20 27.54 27.51 24.64 22.63 21.40 20.85 19.09 18.72 14.20
H
H
N
4u
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


4v
ppm


4v
ppm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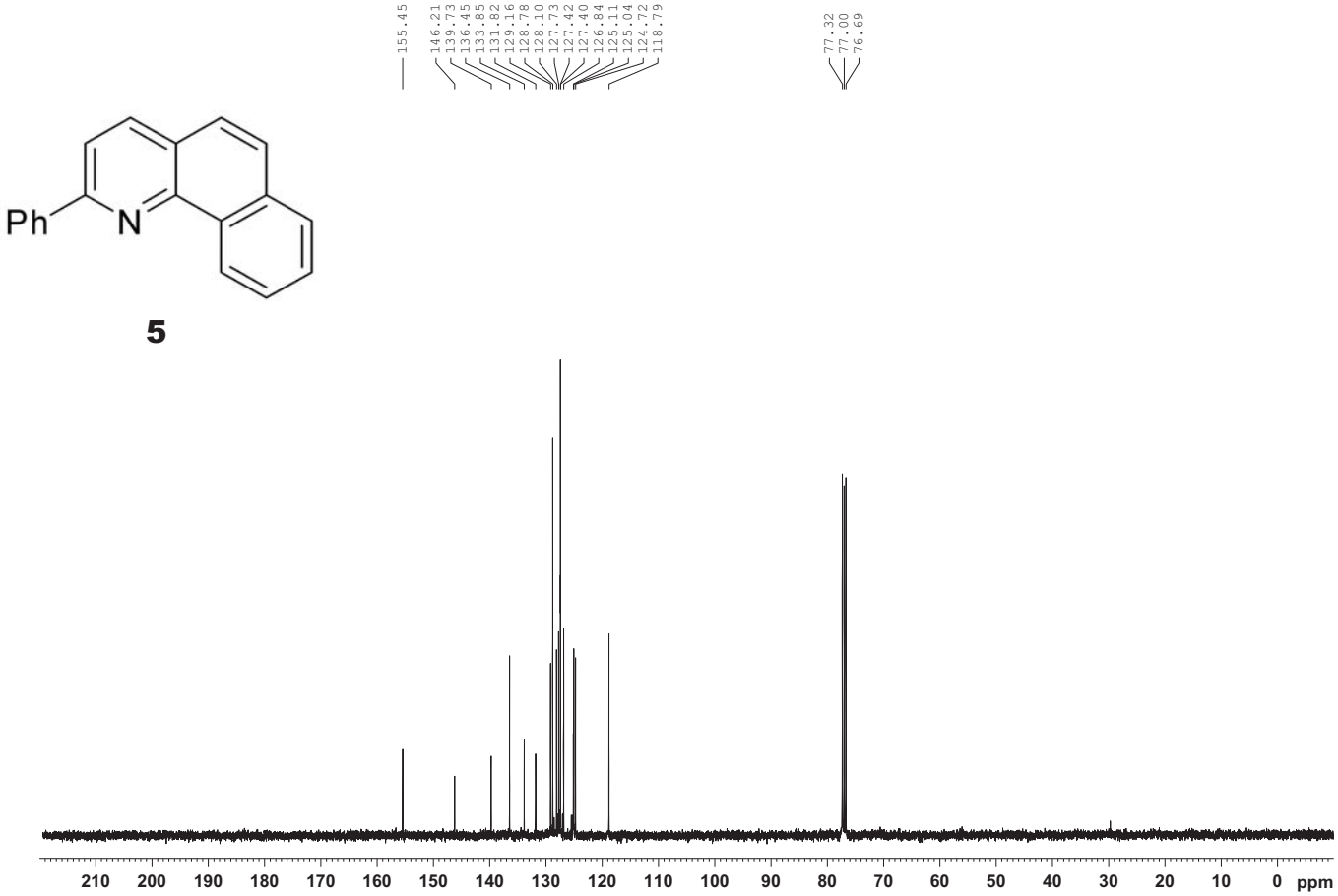
9.475
8.328
8.310
8.158
8.137
7.957
7.936
7.882
7.863
7.756
7.734
7.713
7.688
7.670
7.647
7.625
7.558
7.539
7.520
7.474
7.456
7.438
7.207
0.000
Ph
N
5
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.02
1.03
1.02
1.01
2.06
2.04
2.03
1.04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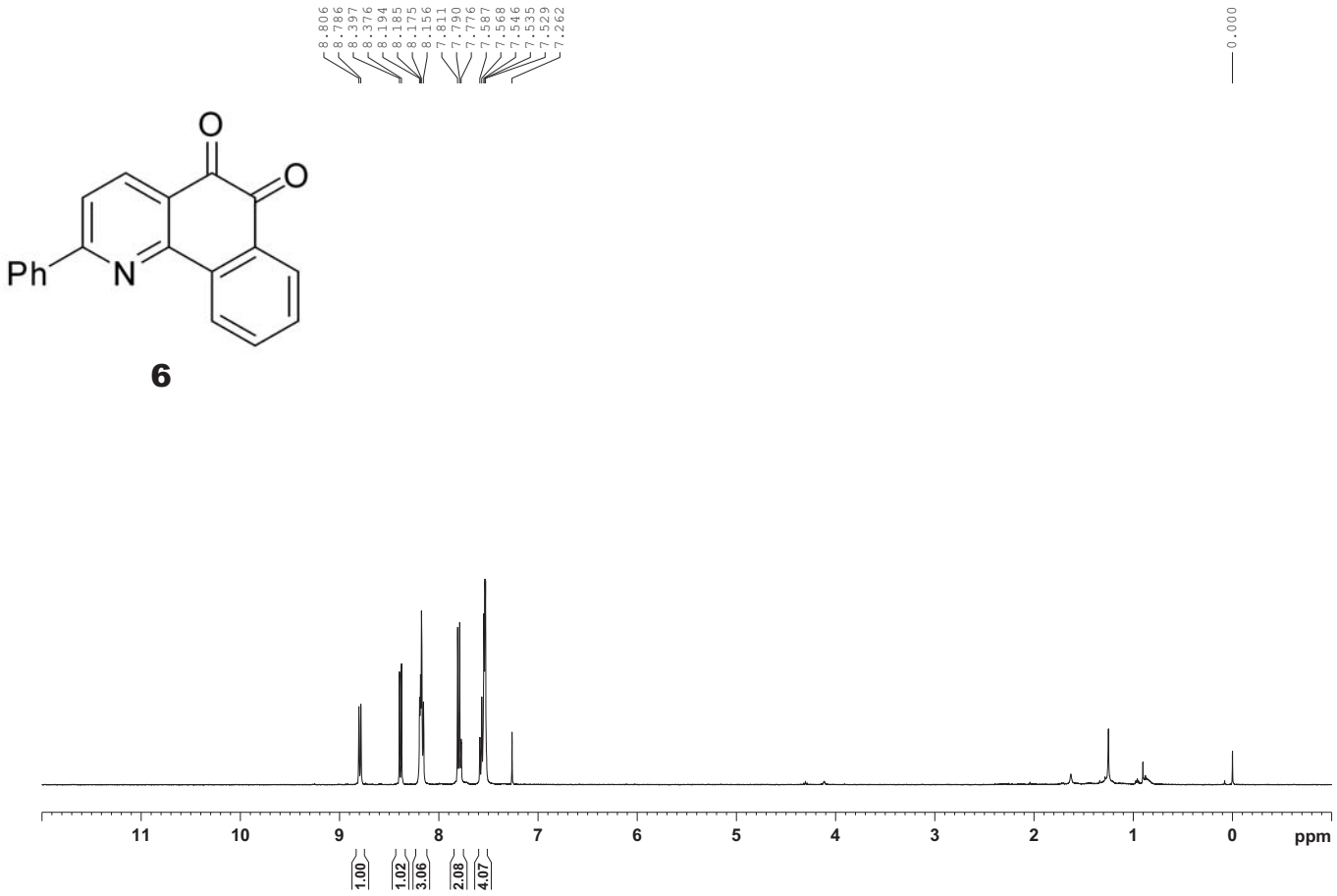
139.73
136.45
133.85
131.82
129.16
128.78
128.10
127.73
127.42
127.40
126.84
125.11
125.04
124.72
118.79
77.32
77.00
76.69
Ph
N
5
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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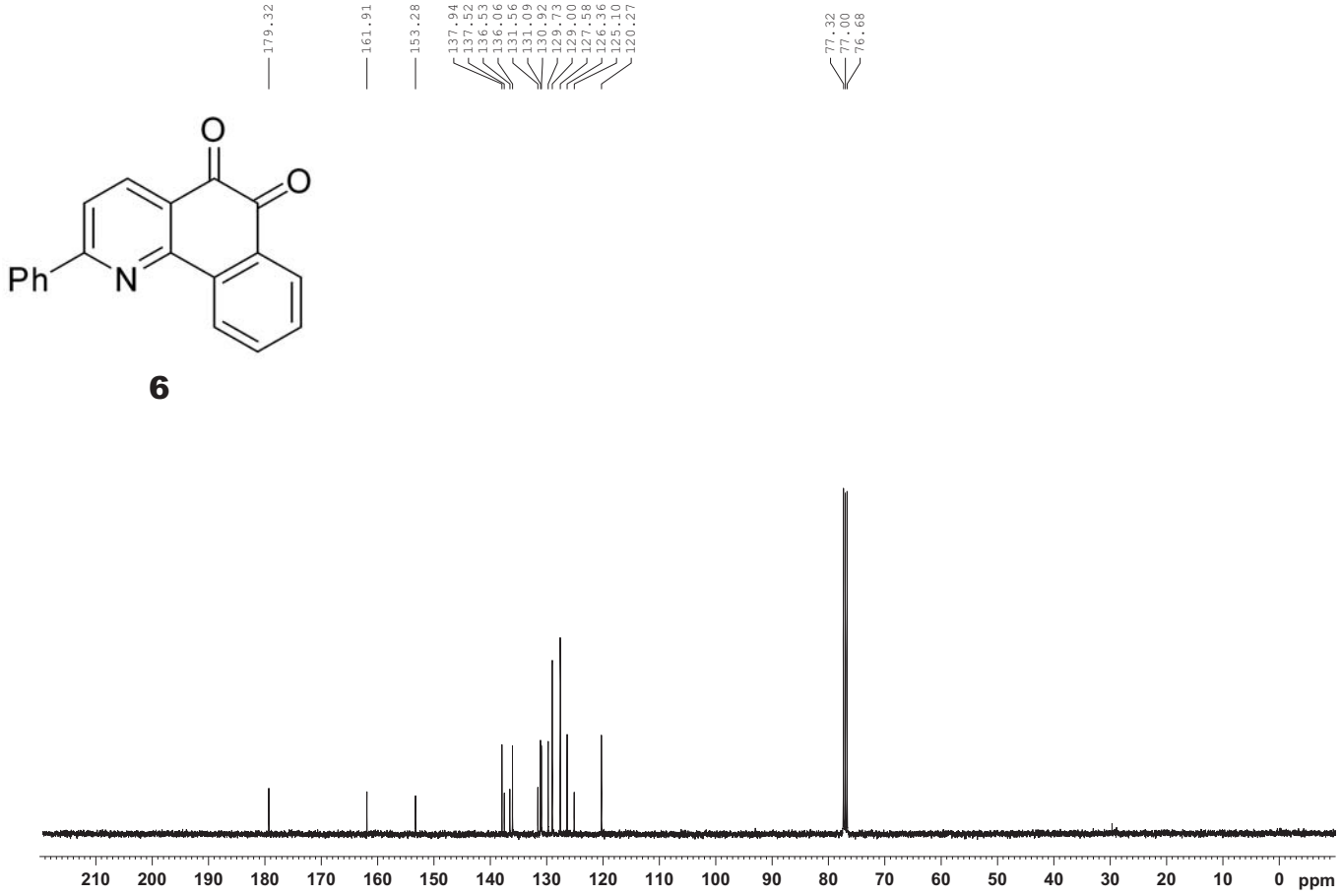
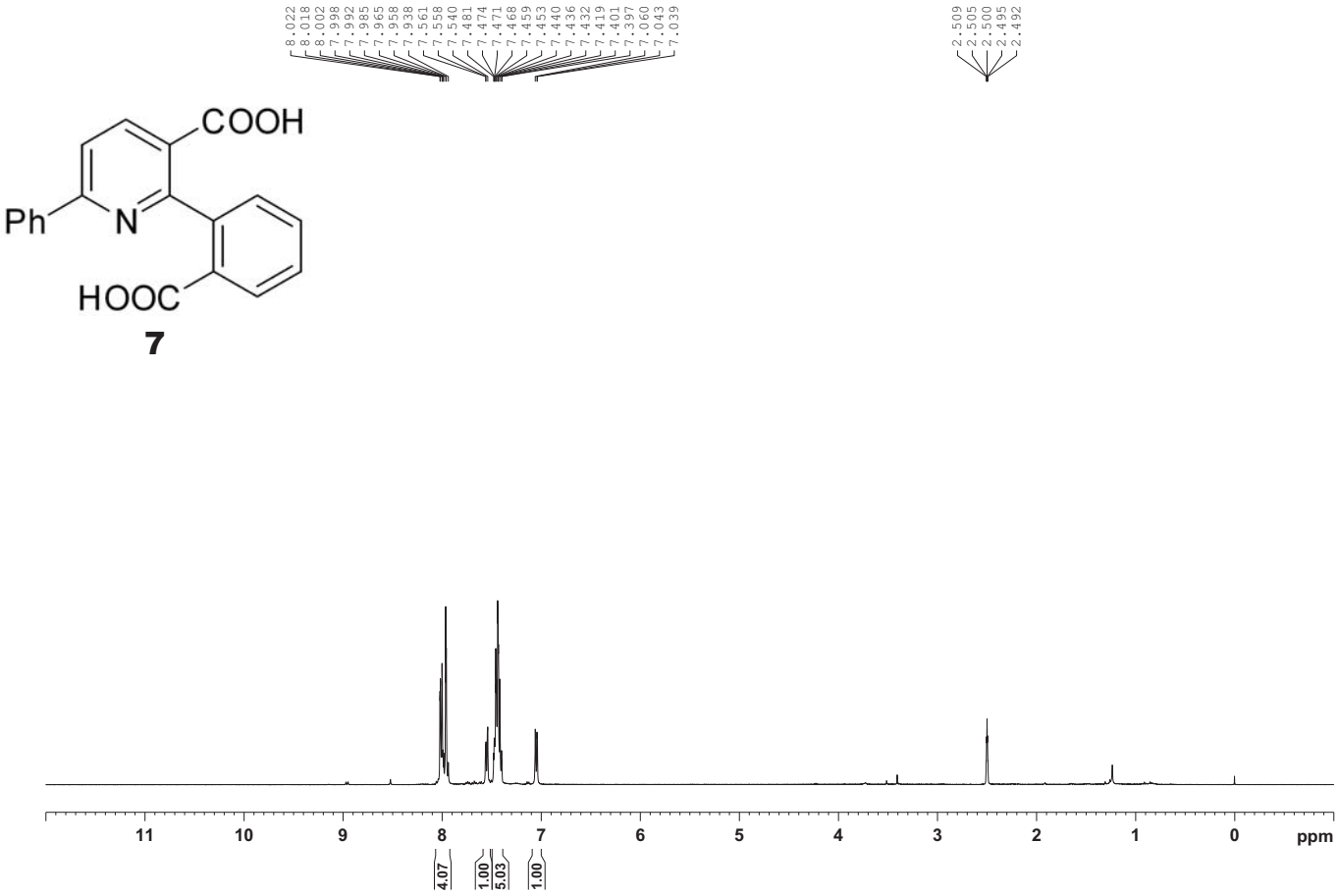
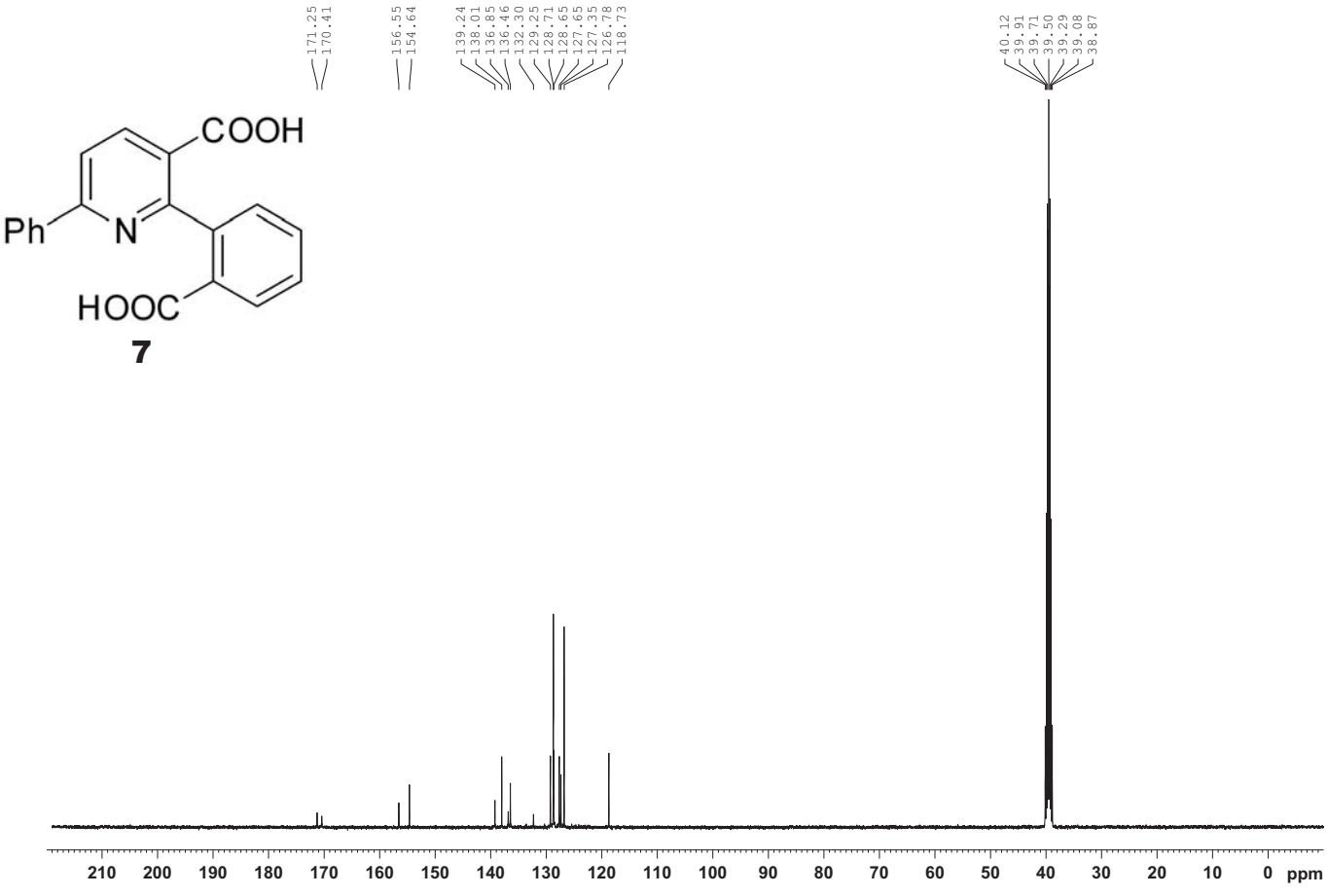
8.786
8.397
8.376
8.194
8.185
8.175
8.156
7.811
7.790
7.776
7.587
7.568
7.546
7.535
7.529
7.262
0.000
O
O
Ph
N
6
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.02
3.06
2.08
4.07


179.32
161.91
153.28
137.94
137.52
136.53
136.06
131.56
131.09
130.92
129.73
129.00
127.58
126.36
125.10
120.27
77.32
77.00
76.68
O
O
Ph
N
6
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.022
8.018
8.002
7.998
7.992
7.985
7.965
7.958
7.938
7.561
7.558
7.540
7.481
7.474
7.471
7.468
7.459
7.453
7.440
7.436
7.432
7.419
7.401
7.397
7.060
7.043
7.039
2.509
2.505
2.500
2.495
2.492
COOH
Ph
N
HOOC
7
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.07
1.00
5.03
1.00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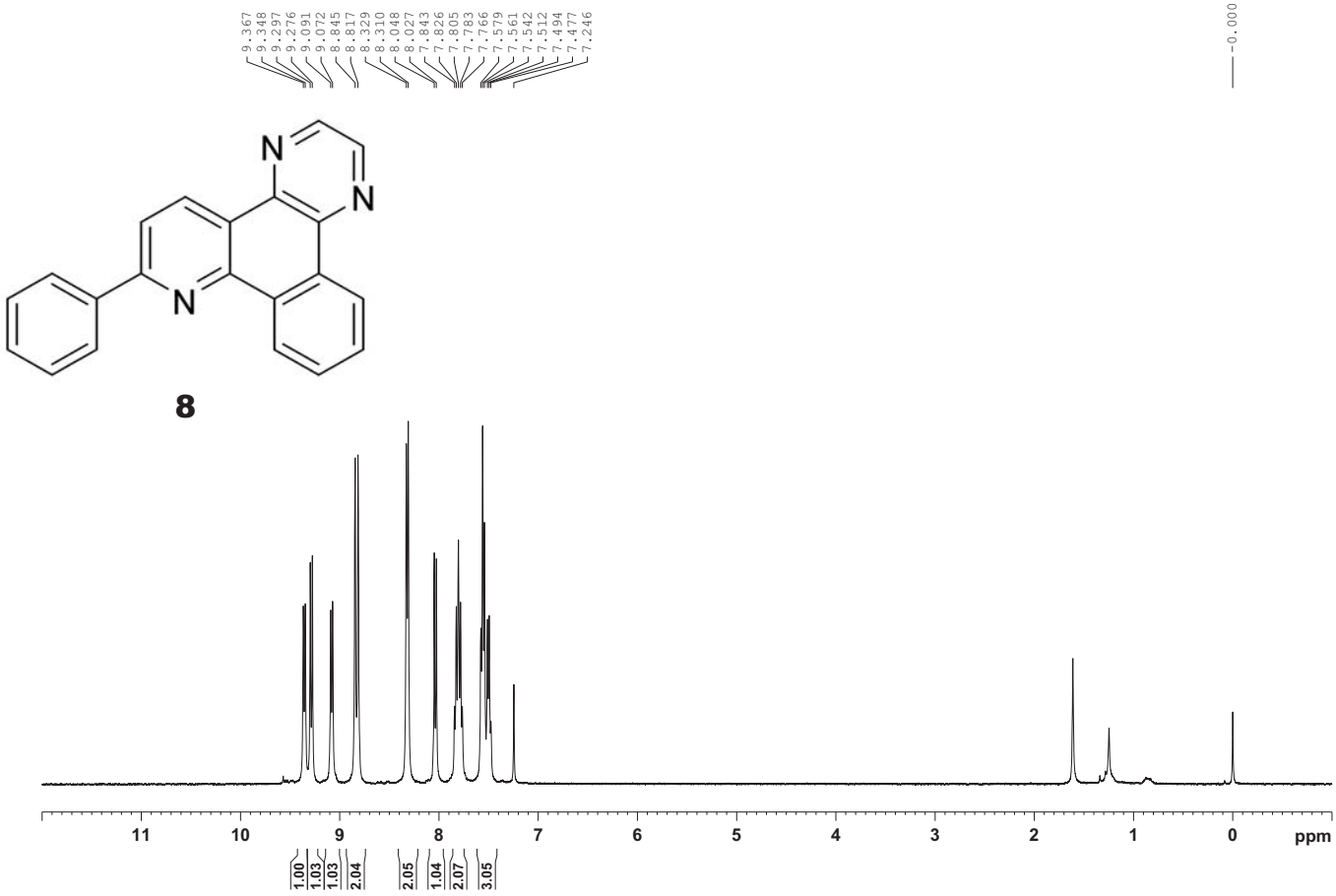
170.41
156.55
154.64
139.24
138.01
136.85
136.46
132.30
129.25
128.71
128.65
127.65
127.35
126.78
118.73
40.12
39.91
39.71
39.50
39.29
39.08
38.87
COOH
Ph
N
HOOC
7
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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9.072
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8.817
8.329
8.310
8.048
8.027
7.843
7.826
7.805
7.783
7.766
7.579
7.561
7.542
7.512
7.494
7.477
7.246
-0.000
N
N
N
8
11
10
9
8
7
6
5
4
3
2
1
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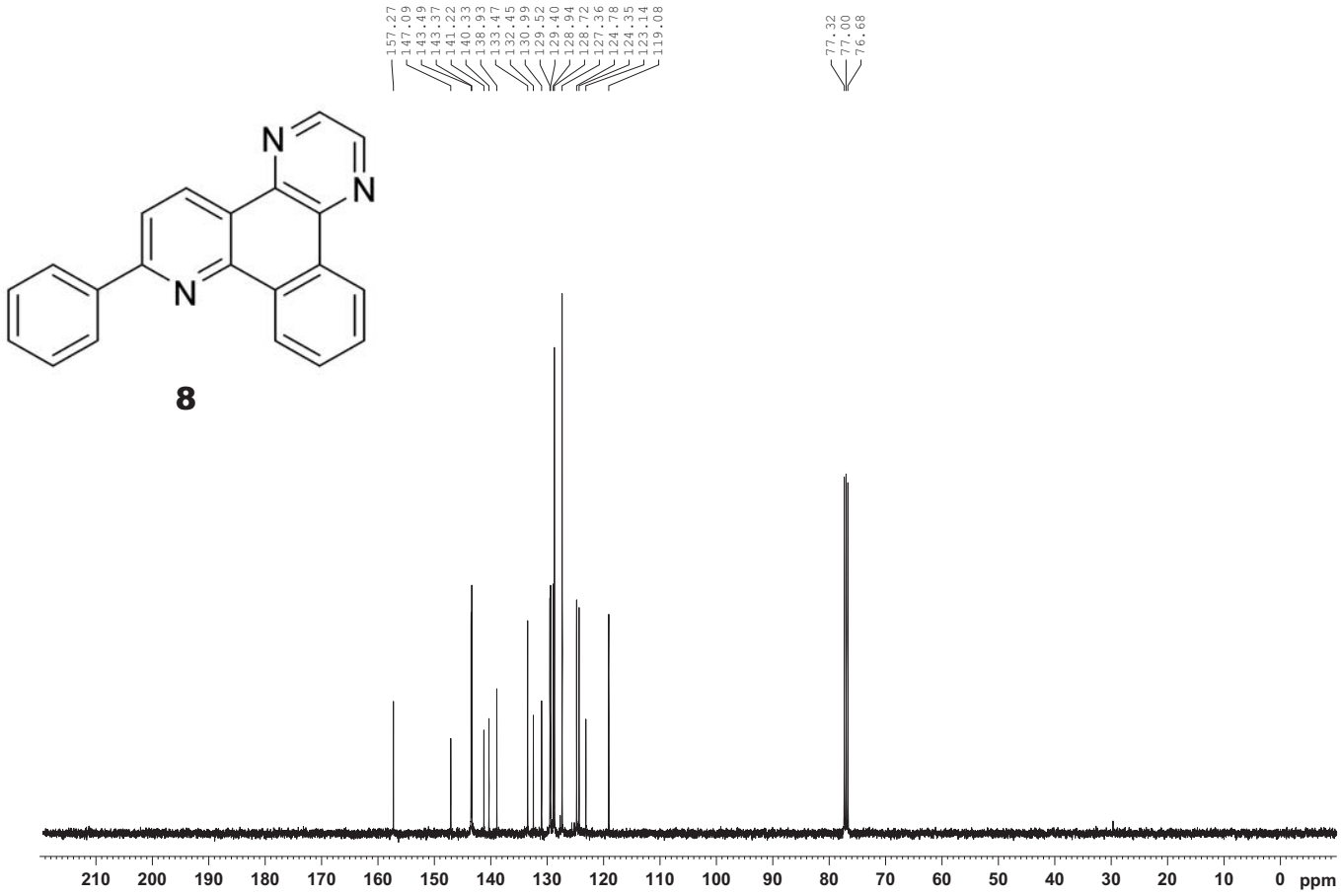
ppm
1.00
1.03
1.03
2.04
2.05
1.04
2.07
3.05


157.27
147.09
143.49
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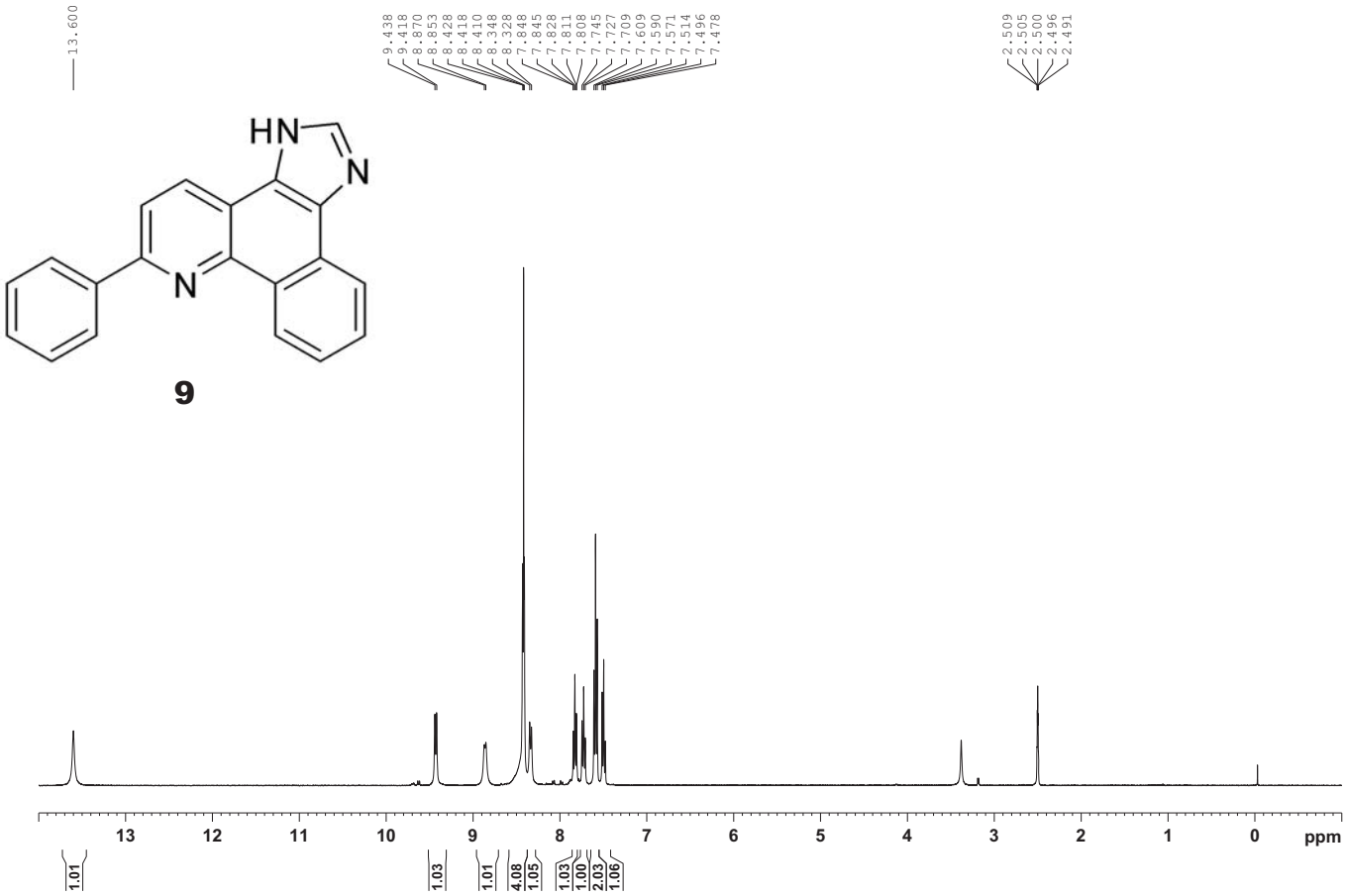
141.22
140.33
138.93
133.47
132.45
130.99
129.52
129.40
128.94
128.72
127.36
124.78
124.35
123.14
119.08
77.32
77.00
76.68
N
N
N
8
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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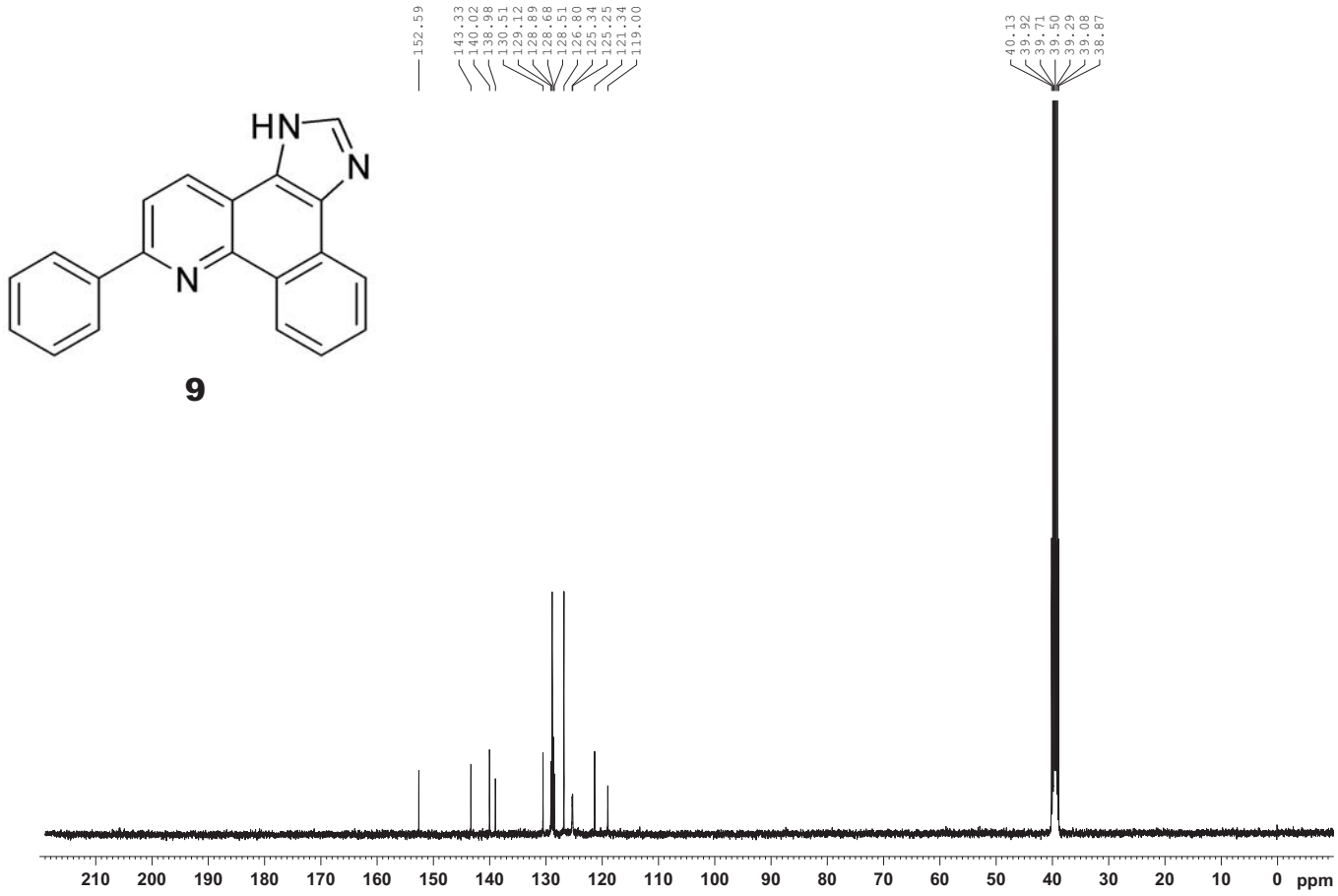
9.418
8.870
8.853
8.428
8.418
8.410
8.348
8.328
7.848
7.845
7.828
7.811
7.808
7.745
7.727
7.709
7.609
7.590
7.571
7.514
7.496
7.478
2.509
2.505
2.500
2.496
2.491
HN
N
N
9
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.01
1.03
1.01
4.08
1.05
1.03
1.00
2.03
1.06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140.02
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130.51
129.12
128.89
128.68
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126.80
125.34
125.25
121.34
119.00
40.13
39.92
39.71
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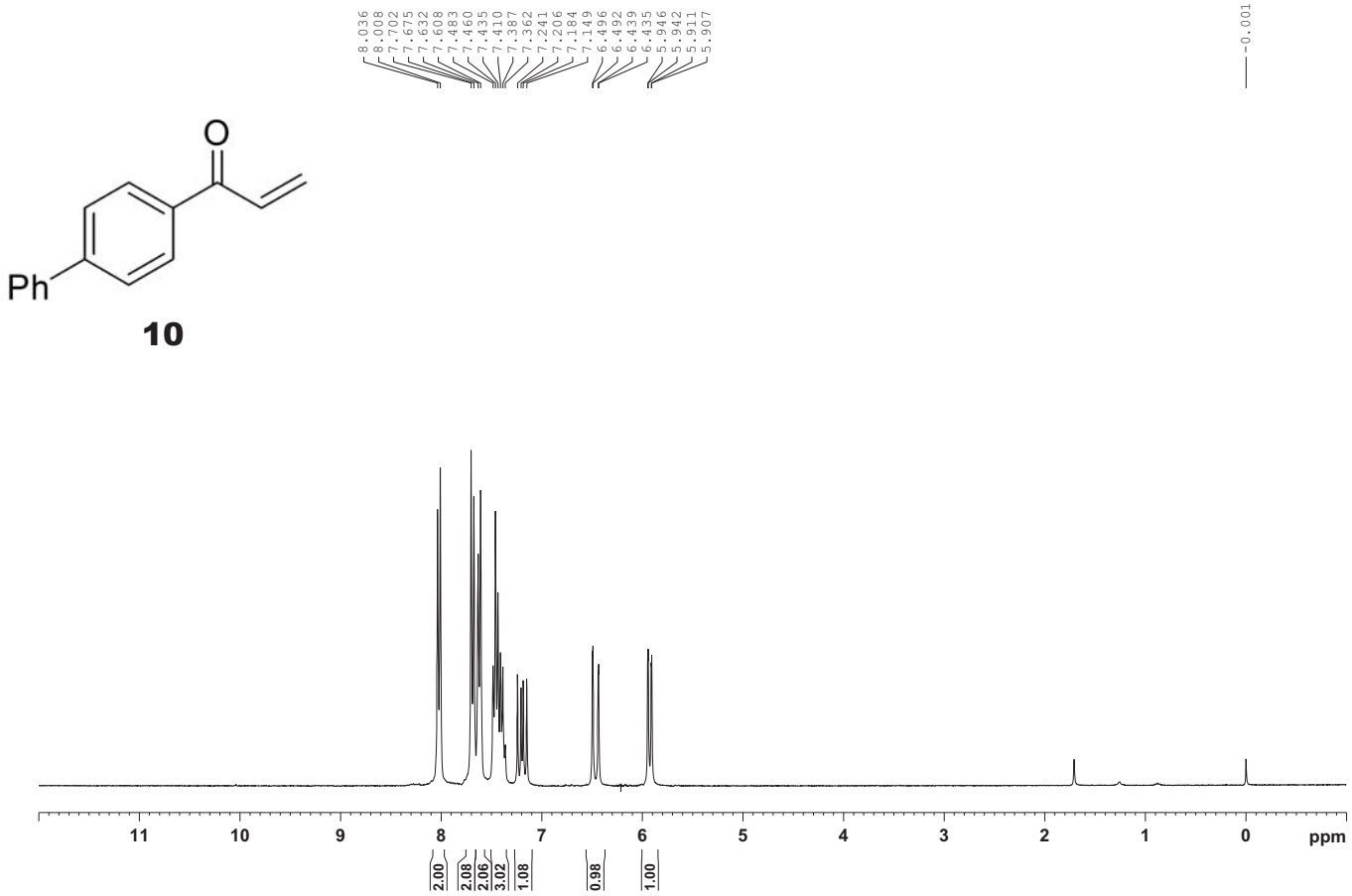
39.29
39.08
38.87
HN
N
N
9
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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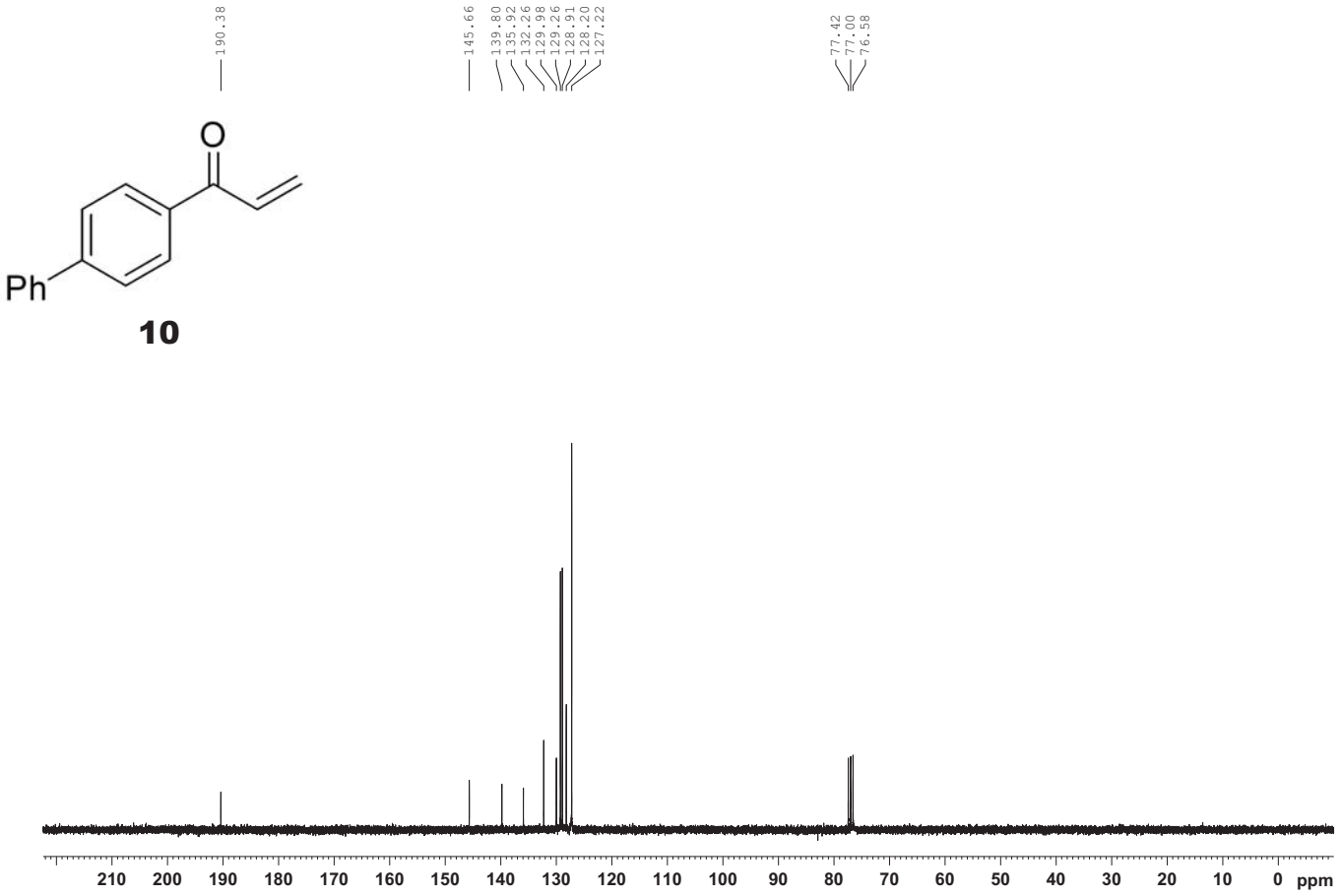
7.675
7.632
7.608
7.483
7.460
7.435
7.410
7.387
7.362
7.241
7.206
7.184
7.149
6.496
6.492
6.439
6.435
5.942
5.911
5.907
-0.001
O
Ph
10
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
2.08
2.06
3.02
1.08
0.98
1.00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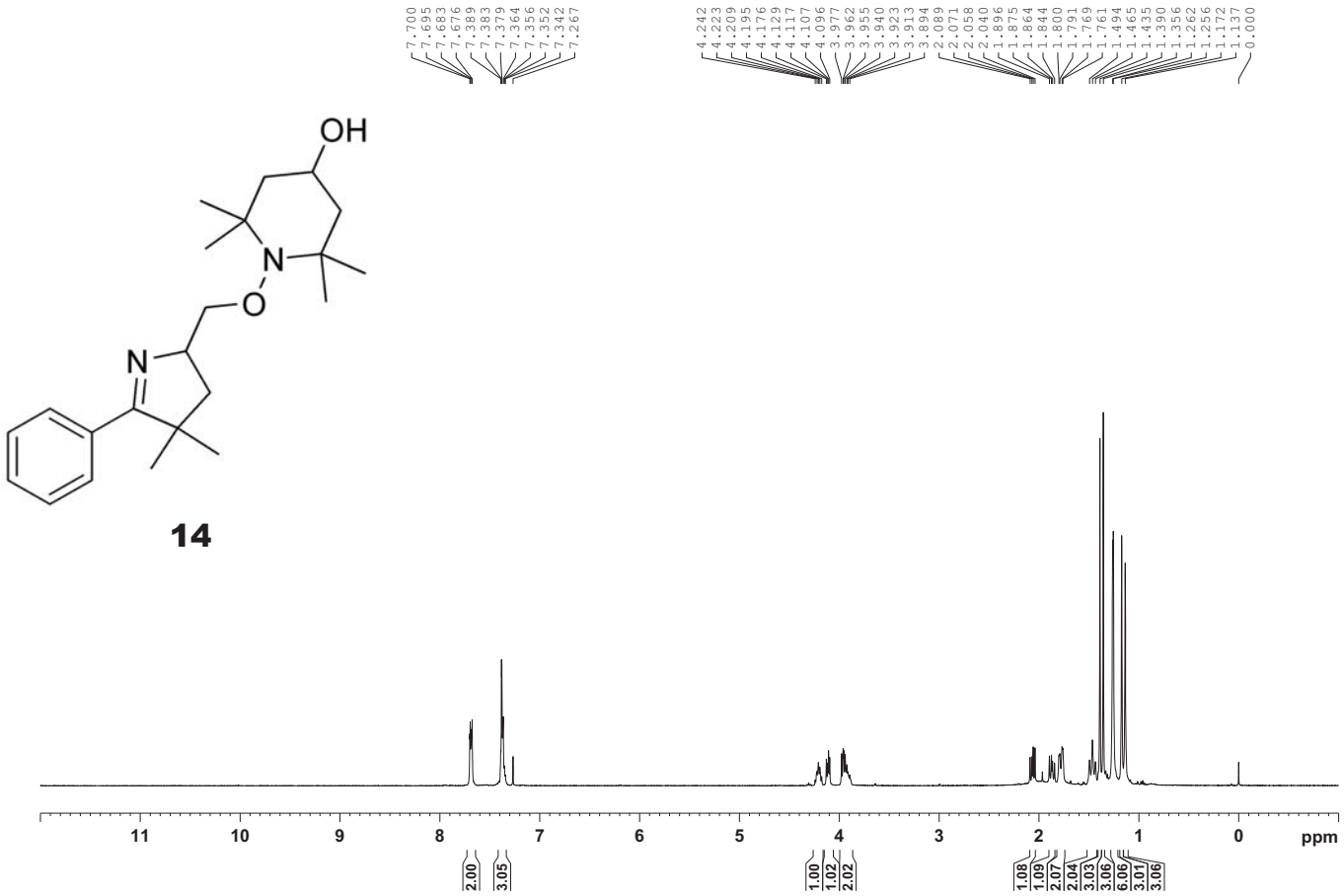
145.66
139.80
135.92
132.26
129.98
129.26
128.91
128.20
127.22
77.42
77.00
76.58
O
Ph
10
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.383
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7.364
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7.352
7.342
7.267
4.242
4.223
4.209
4.195
4.176
4.129
4.117
4.107
4.096
3.977
3.962
3.955
3.940
3.923
3.913
3.894
2.089
2.071
2.058
2.040
1.896
1.875
1.864
1.844
1.800
1.791
1.769
1.761
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1.435
1.390
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1.137
0.000
OH
N
O
N
14
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
3.05
1.00
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2.02
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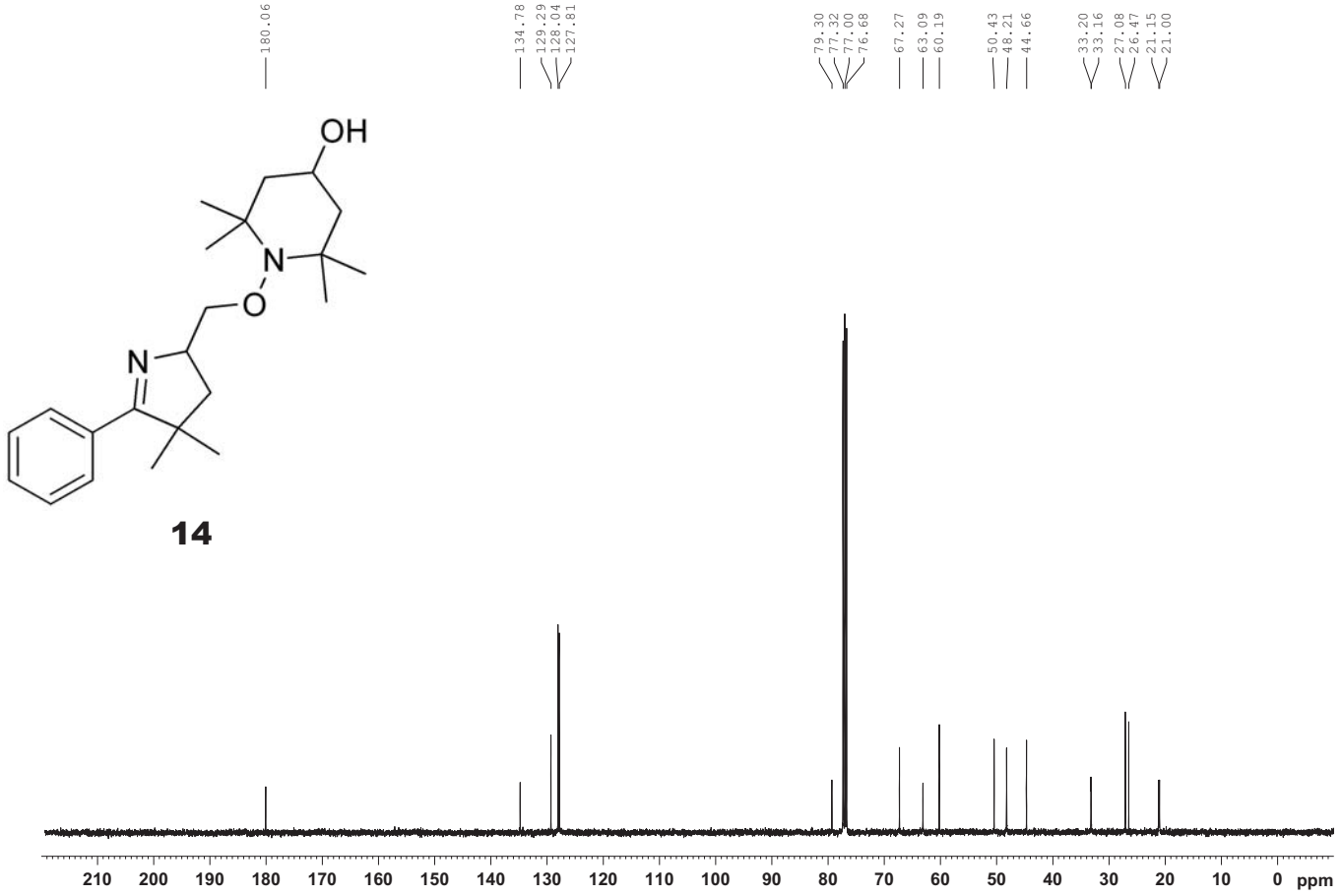
2.07
2.04
3.03
3.06
6.06
3.01
3.06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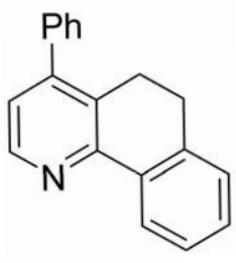
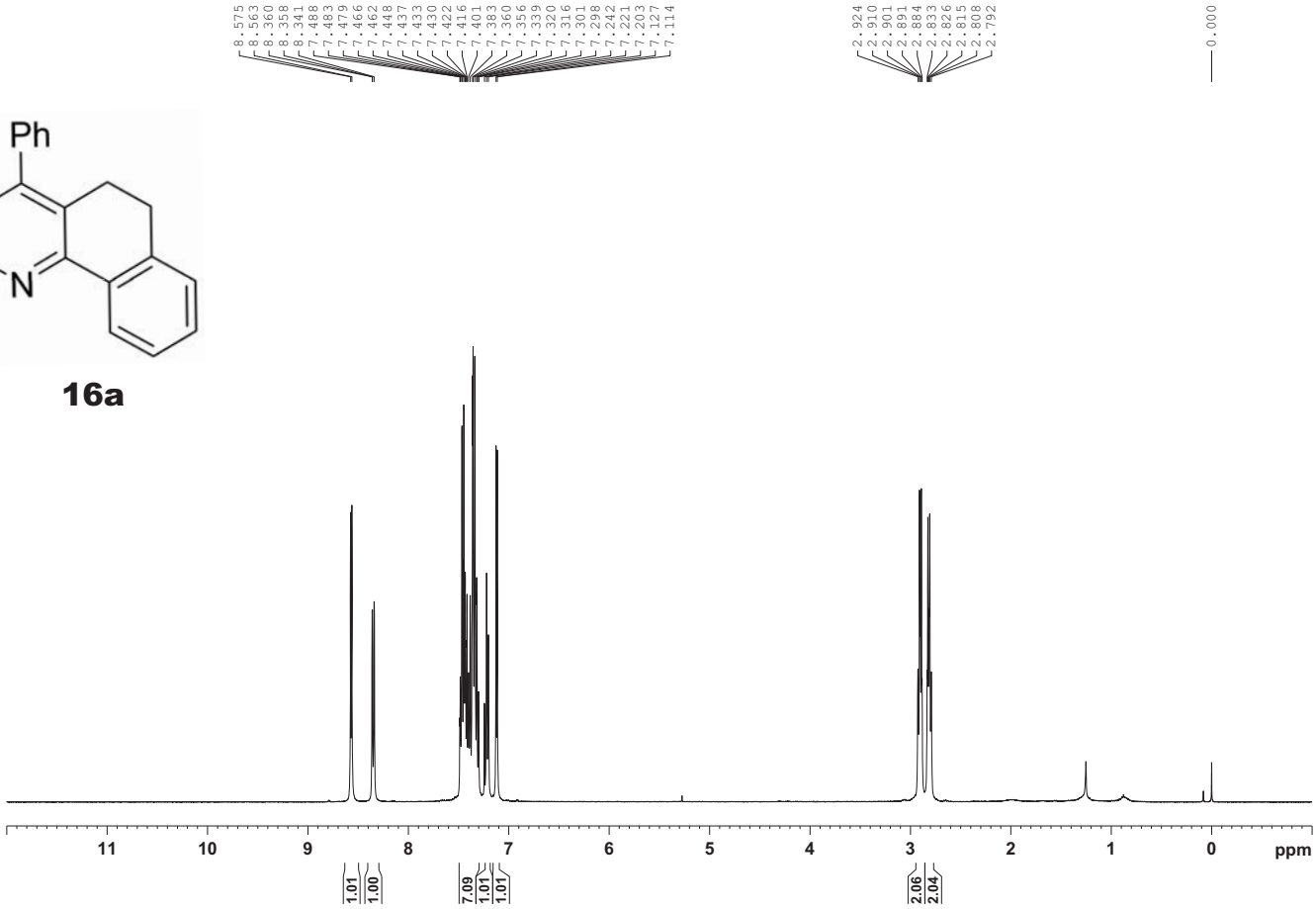
77.00
76.68
67.27
63.09
60.19
50.43
48.21
44.66
33.20
33.16
27.08
26.47
21.15
21.00
OH
N
O
N
14
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


Ph
N

16a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7.448
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7.433
7.430
7.422
7.416
7.401
7.383
7.360
7.356
7.339
7.320
7.316
7.301
7.298
7.242
7.221
7.203
7.127
7.114
2.924
2.910
2.901
2.891
2.884
2.833
2.826
2.815
2.808
2.792
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
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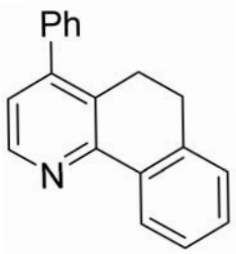
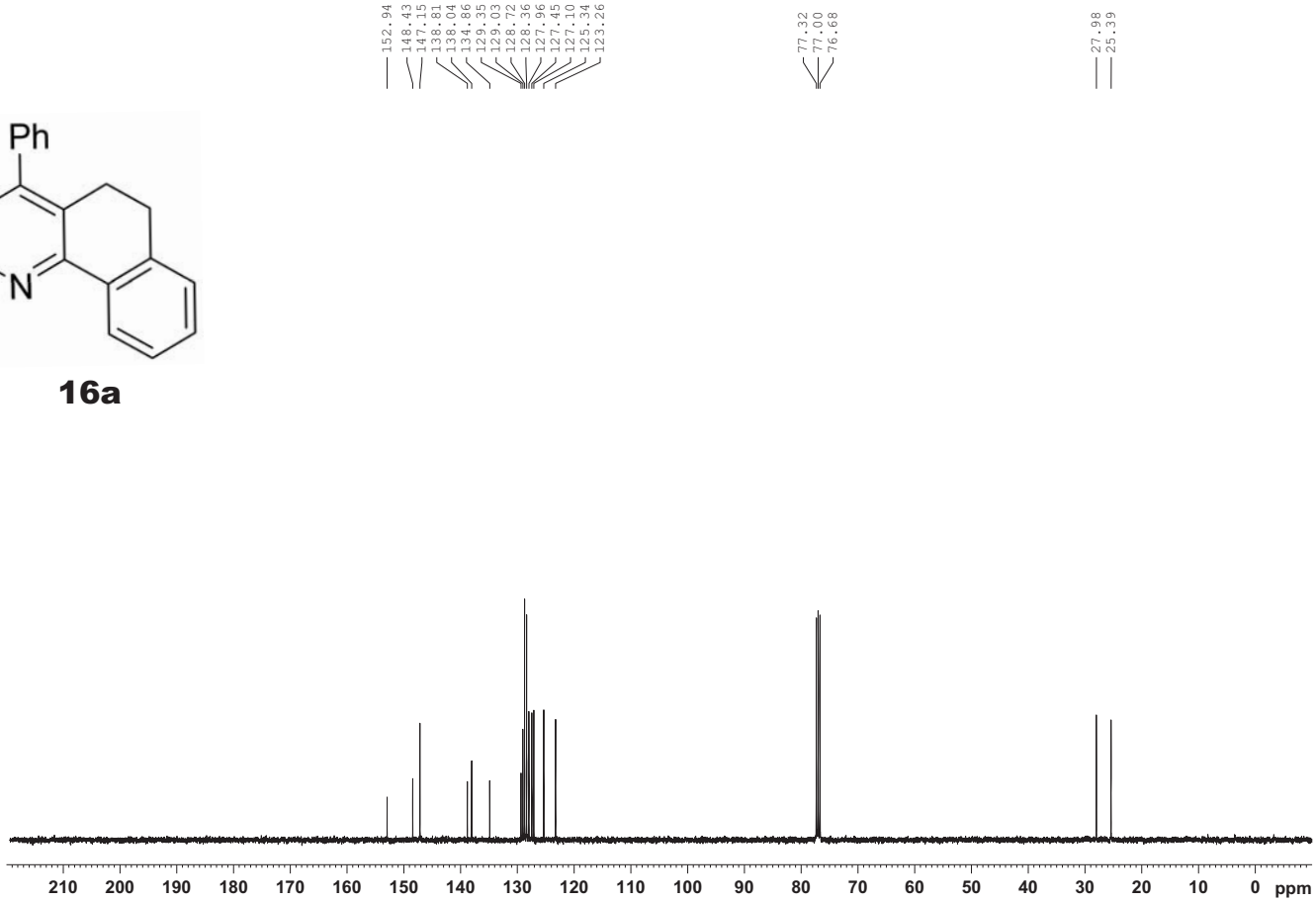
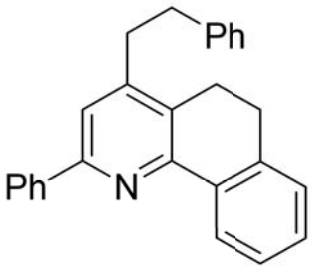
1.00
7.09
1.01
1.01
2.06
2.04

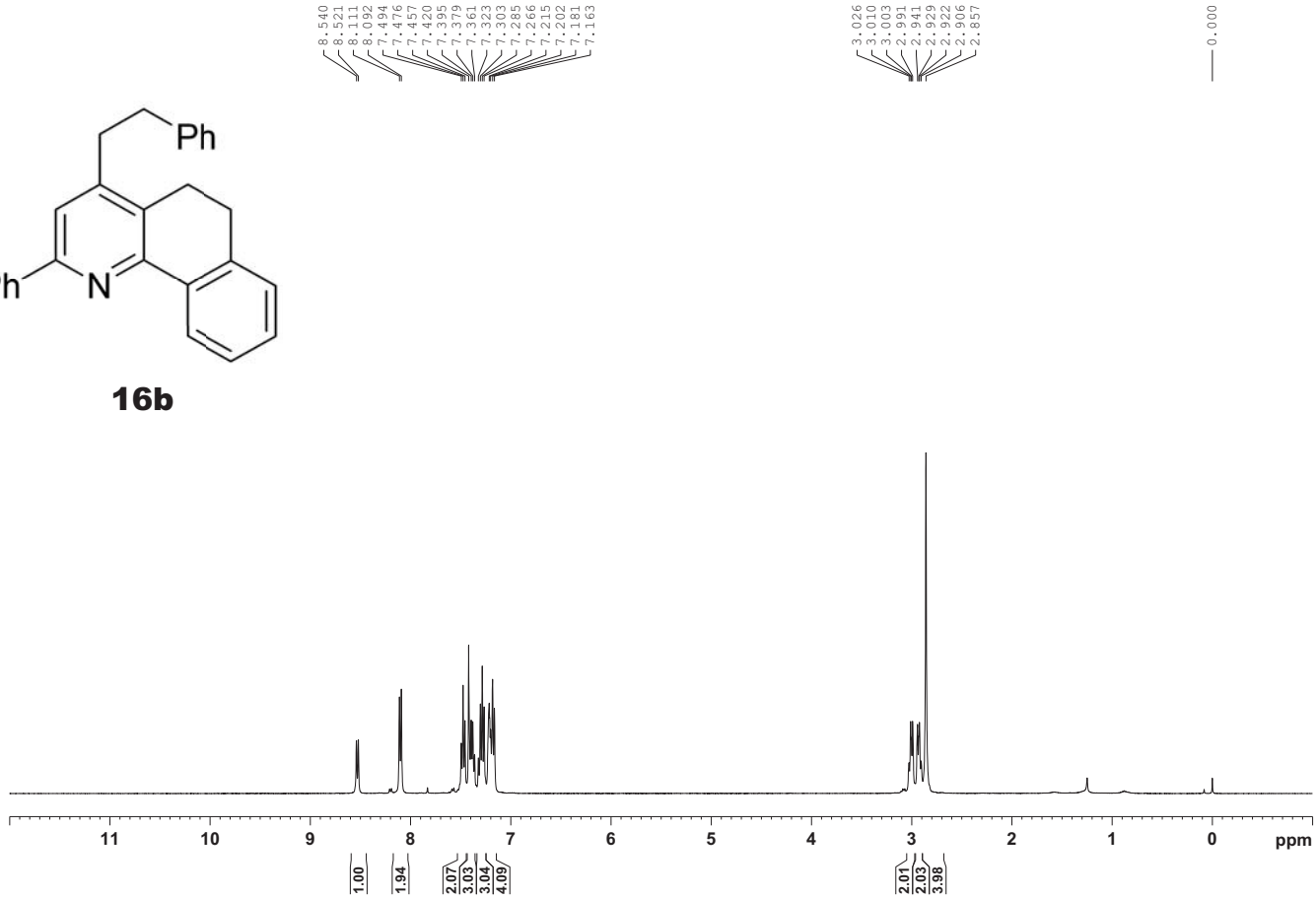

Ph
N

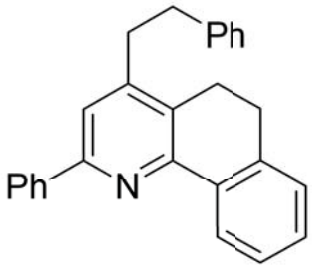
16a


152.94
148.43
147.15
138.81
138.04
134.86
129.35
129.03
128.72
128.36
127.96
127.45
127.10
125.34
123.26
77.32
77.00
76.68
27.98
25.39
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

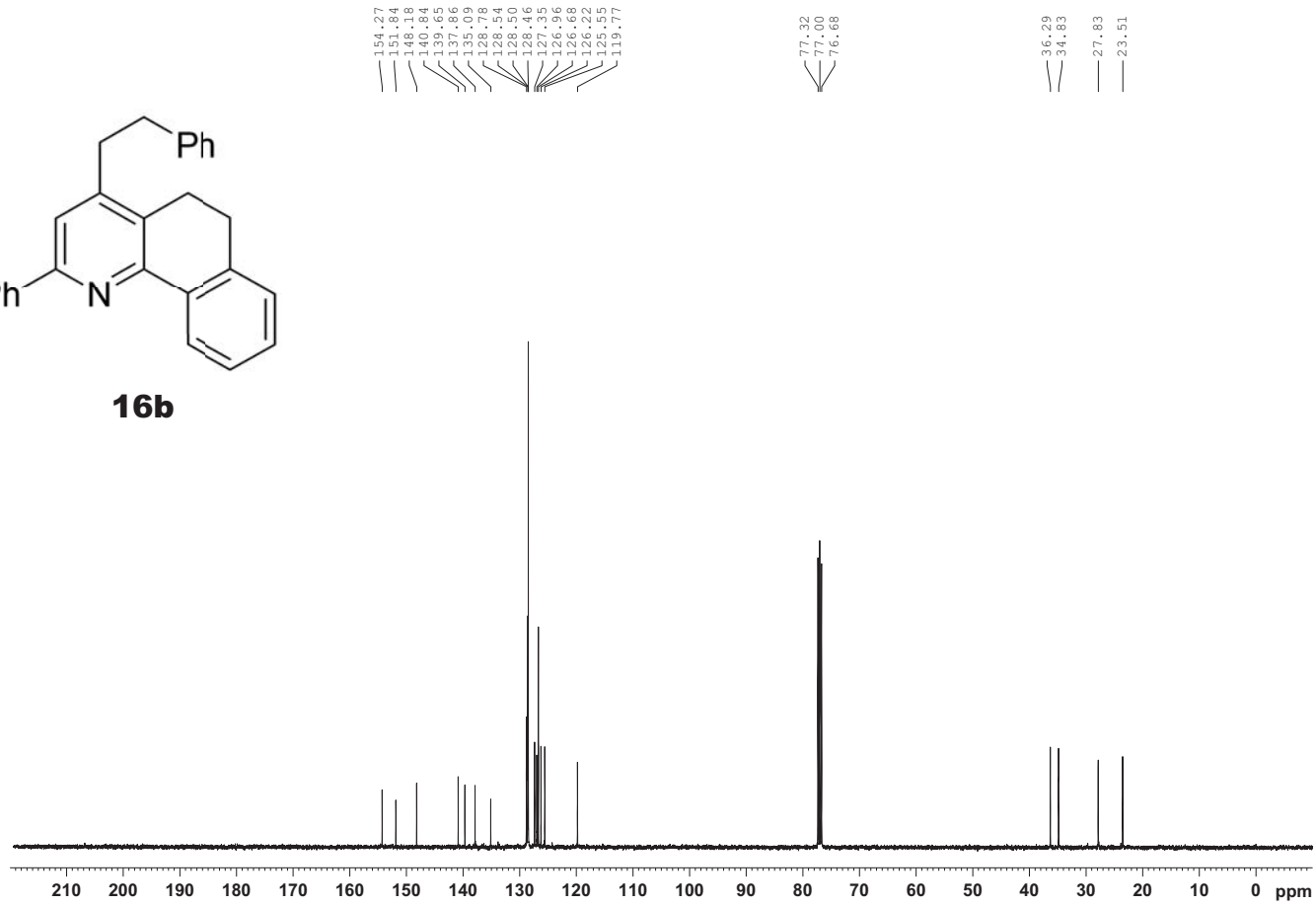

Ph
Ph
N

16b


8.540
8.521
8.111
8.092
7.494
7.476
7.457
7.420
7.395
7.379
7.361
7.323
7.303
7.285
7.266
7.215
7.202
7.181
7.163
3.026
3.010
3.003
2.991
2.941
2.929
2.922
2.906
2.857
0.000
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
1.94
2.07
3.03
3.04
4.09
2.01
2.03
3.98

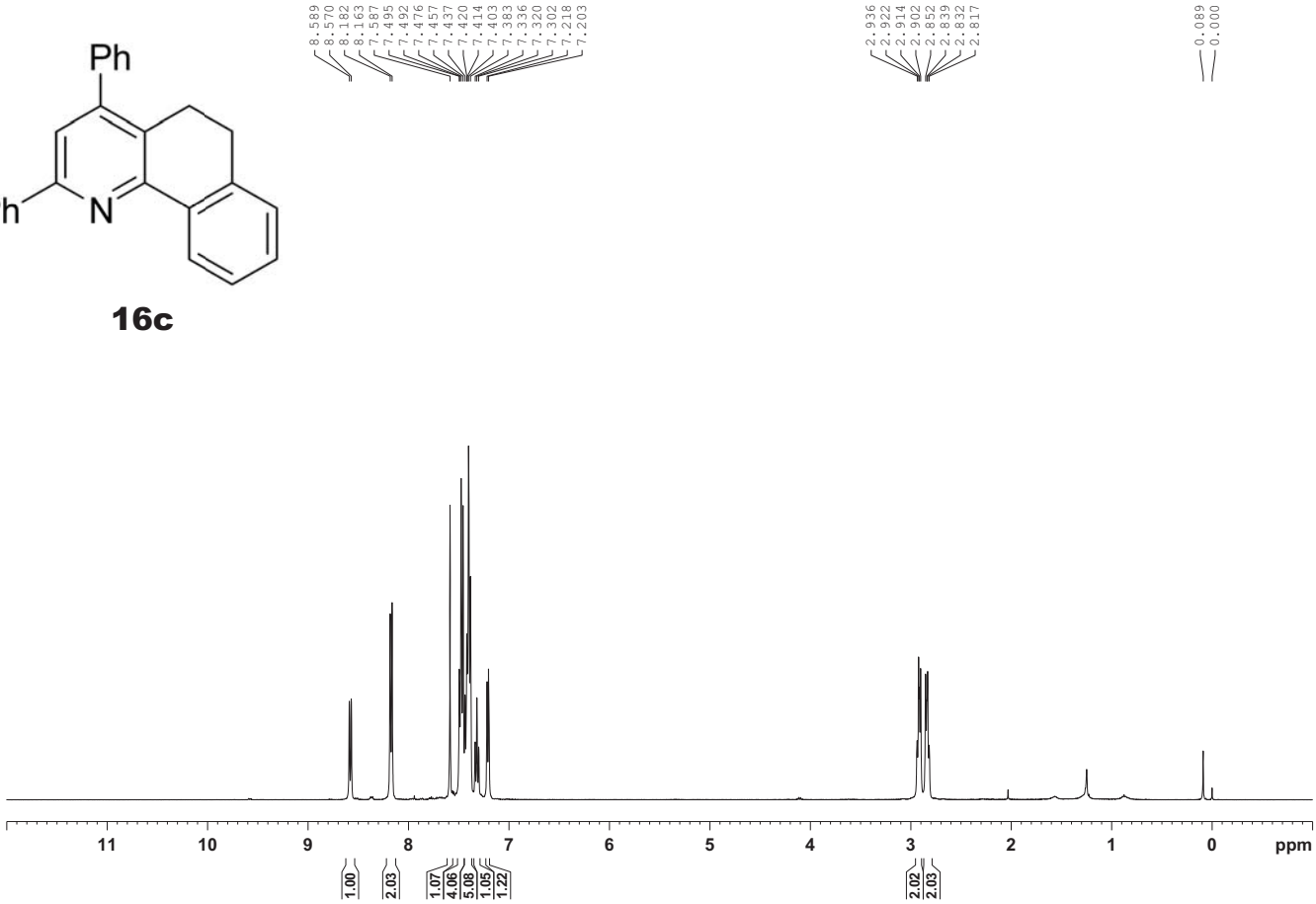

Ph
Ph
N

16b


154.27
151.84
148.18
140.84
139.65
137.86
135.09
128.78
128.54
128.50
128.46
127.35
126.96
126.68
126.22
125.55
119.77
77.32
77.00
76.68
36.29
34.83
27.83
23.51
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

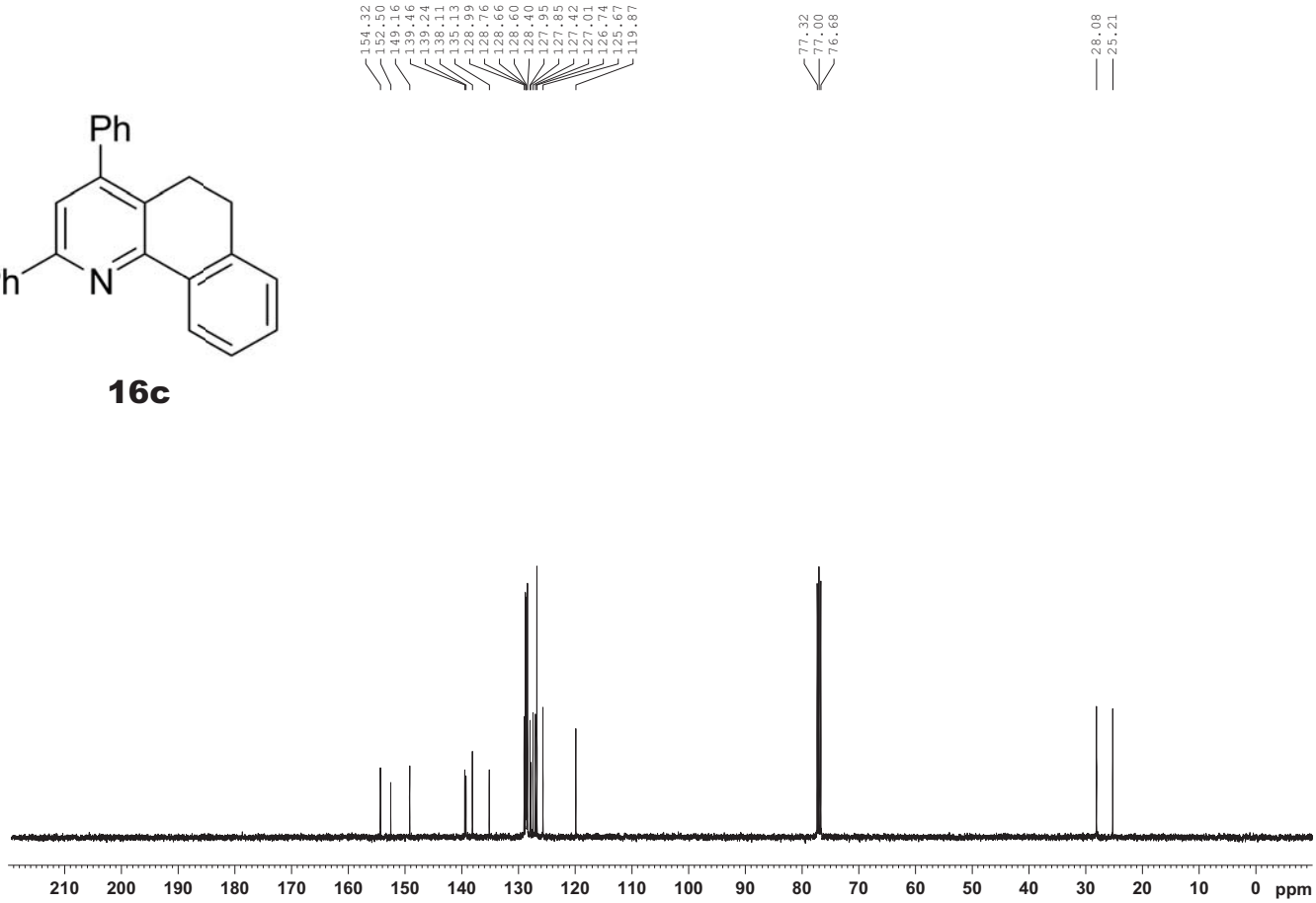
Ph
Ph
N

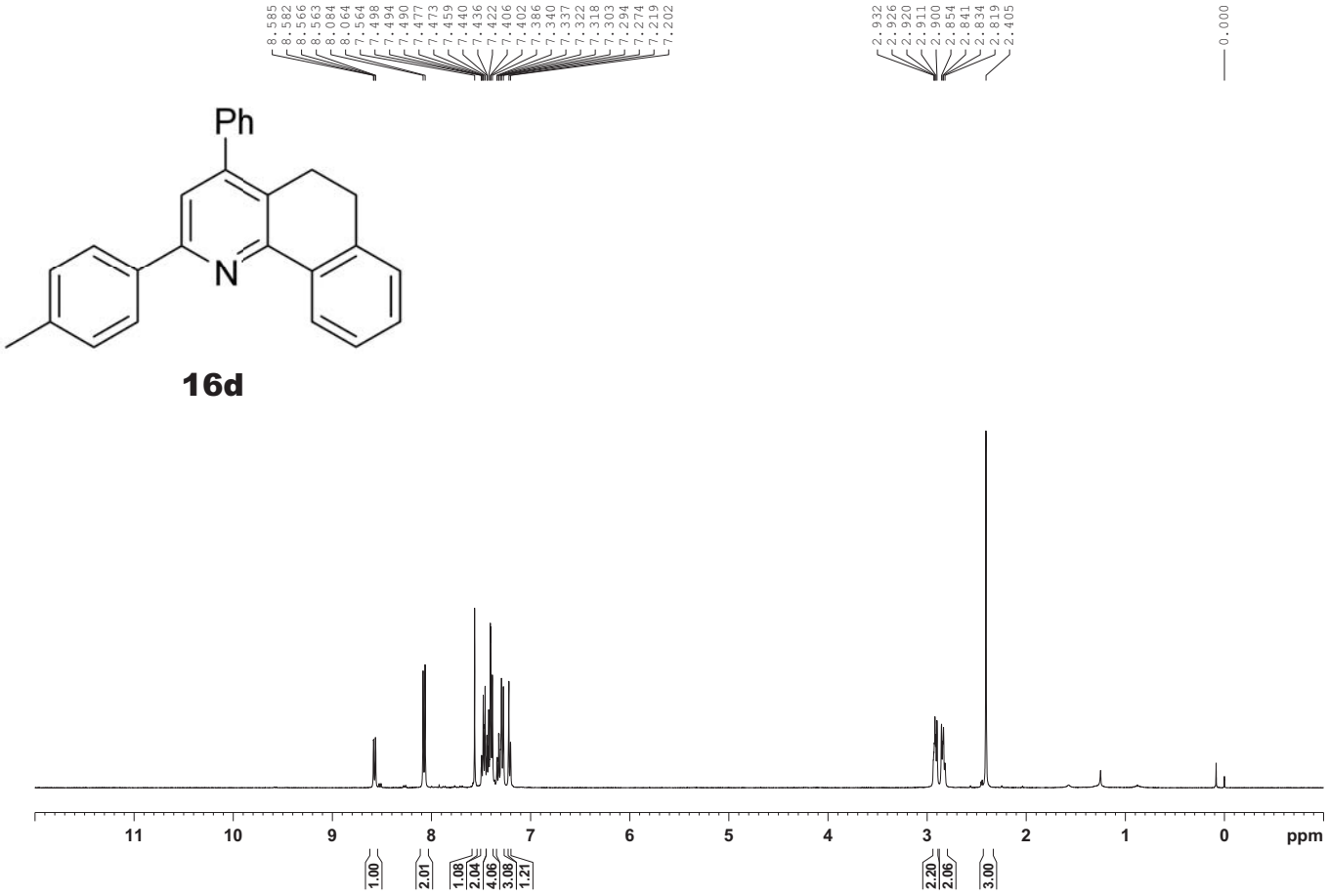
16c

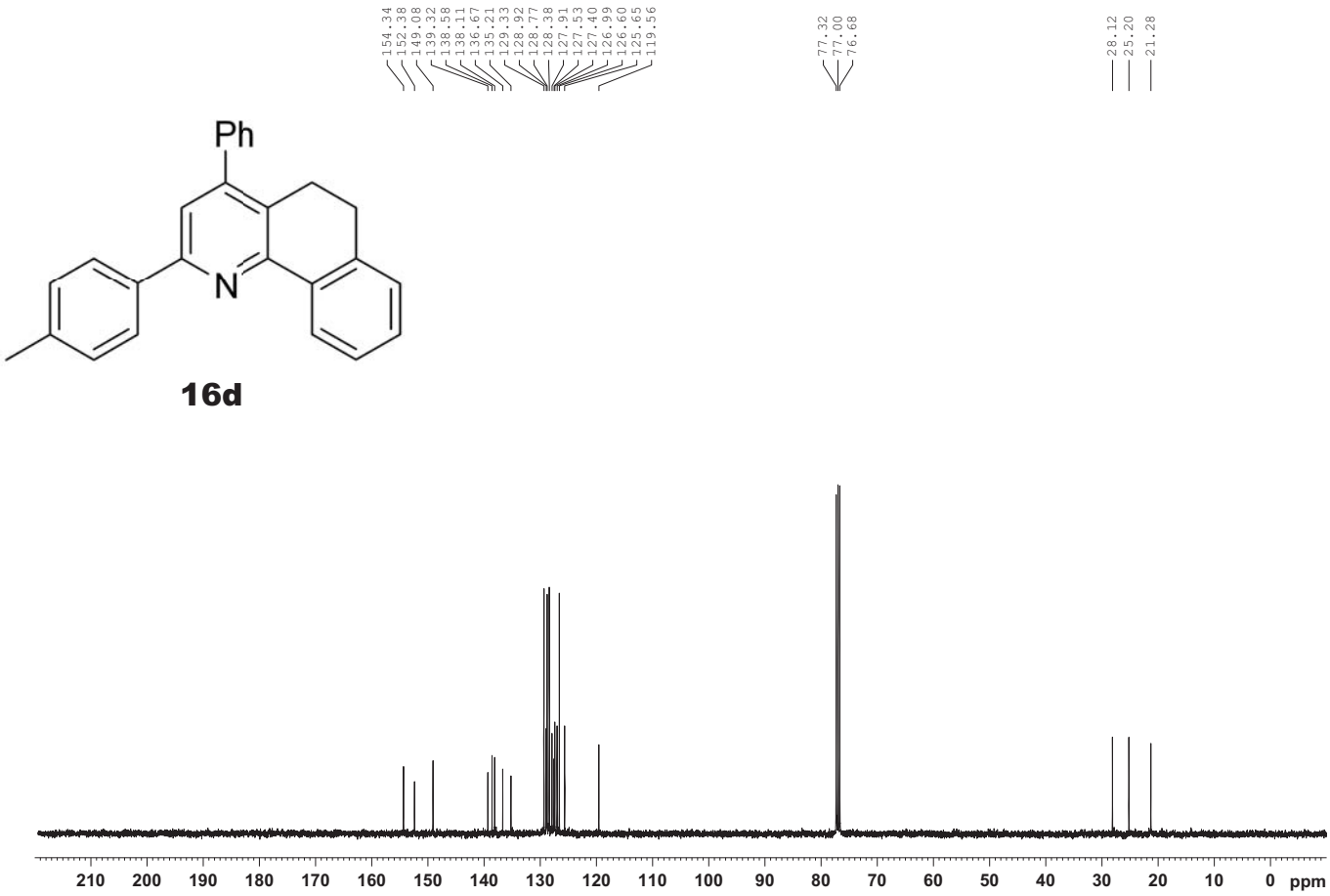

8.589
8.570
8.182
8.163
7.587
7.495
7.492
7.476
7.457
7.437
7.420
7.414
7.403
7.383
7.336
7.320
7.302
7.218
7.203
2.936
2.922
2.914
2.902
2.852
2.839
2.832
2.817
0.089
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.03
1.07
4.06
5.08
1.05
1.22
2.02
2.03

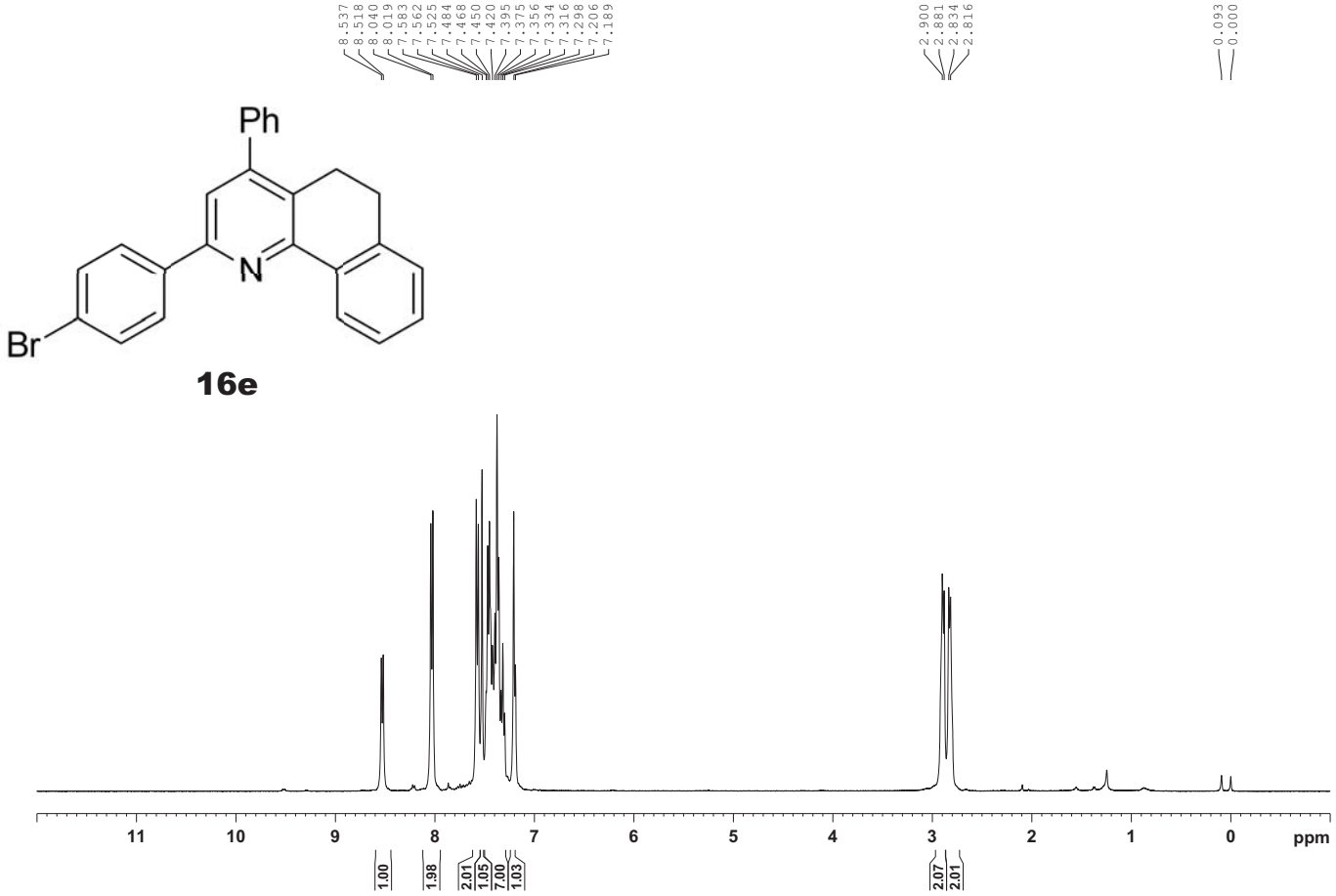
Ph
Ph
N

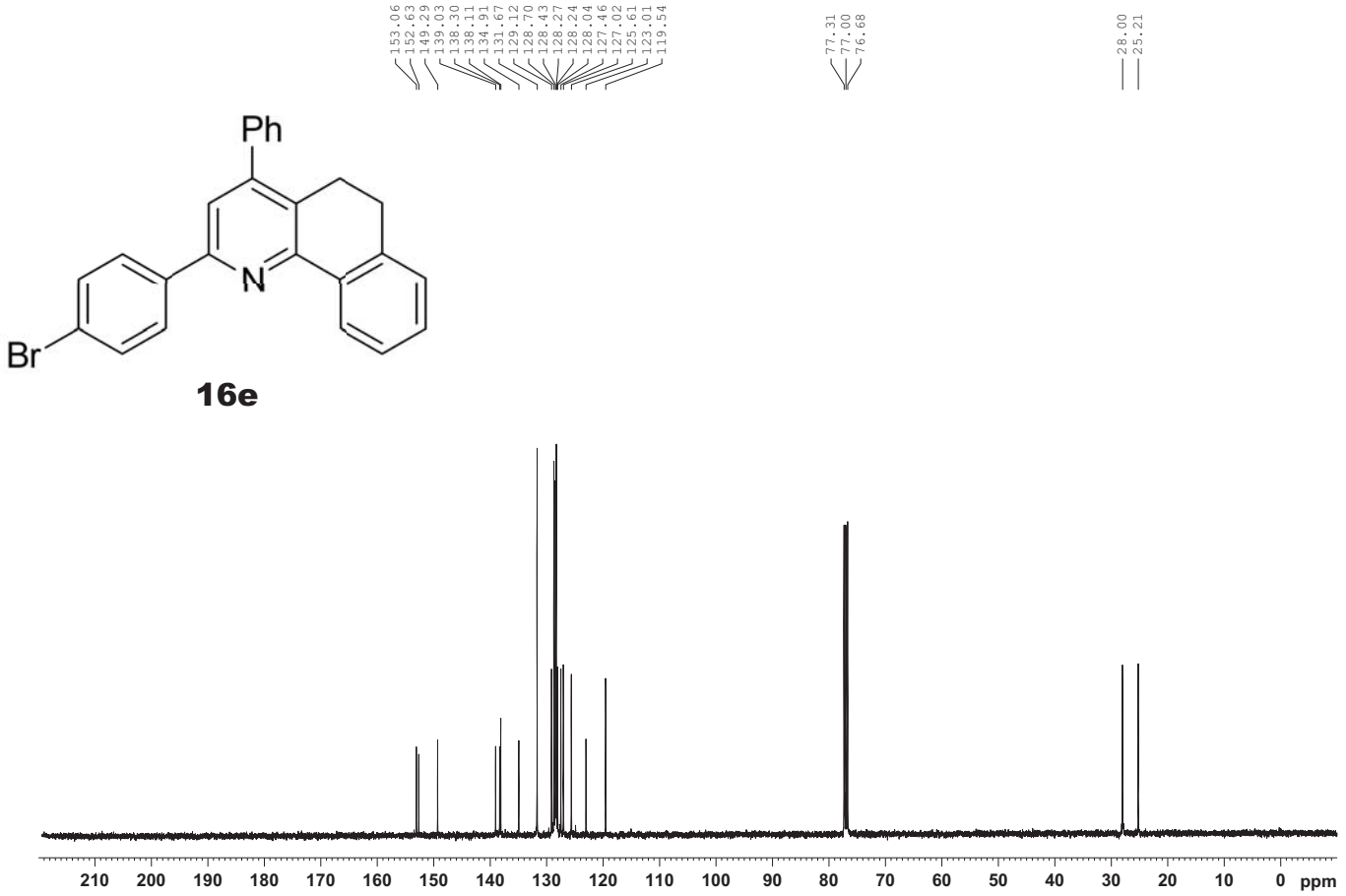
16c

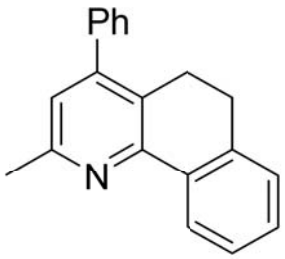

154.32
152.50
149.16
139.46
139.24
138.11
135.13
128.99
128.76
128.66
128.60
128.40
127.95
127.85
127.42
127.01
126.74
125.67
119.87
77.32
77.00
76.68
28.08
25.21
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.585
8.582
8.566
8.563
8.084
8.064
7.564
7.498
7.494
7.490
7.477
7.473
7.459
7.440
7.436
7.422
7.406
7.402
7.386
7.340
7.337
7.322
7.318
7.303
7.294
7.274
7.219
7.202
2.932
2.926
2.920
2.911
2.900
2.854
2.841
2.834
2.819
2.405
0.000
Ph
N
16d
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.01
1.08
2.04
4.06
3.08
1.21
2.20
2.06
3.00

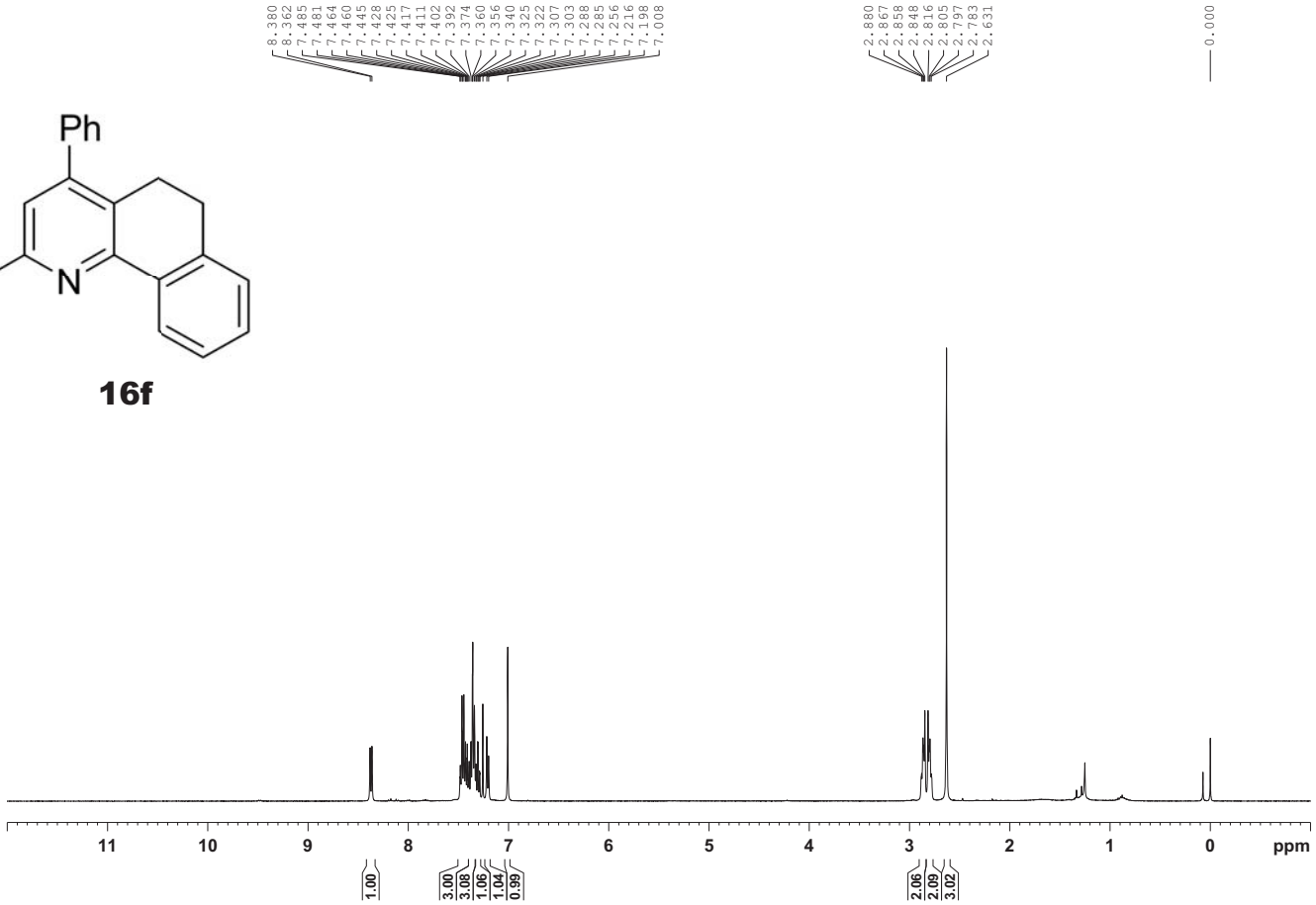

154.34
152.38
149.08
139.32
138.58
138.11
136.67
135.21
129.33
128.92
128.77
128.38
127.91
127.53
127.40
126.99
126.60
125.65
119.56
77.32
77.00
76.68
28.12
25.20
21.28
Ph
N
16d
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

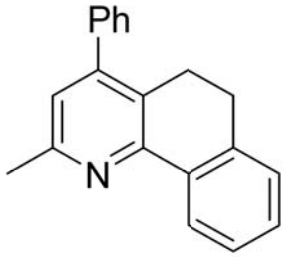

8.537
8.518
8.040
8.019
7.583
7.562
7.525
7.484
7.468
7.450
7.420
7.395
7.375
7.356
7.334
7.316
7.298
7.206
7.189
2.900
2.881
2.834
2.816
0.093
0.000
Ph
N
Br
16e
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.98
2.01
1.05
7.00
1.03
2.07
2.01


153.06
152.63
149.29
139.03
138.30
138.11
134.91
131.67
129.12
128.70
128.43
128.27
128.24
128.04
127.46
127.02
125.61
123.01
119.54
77.31
77.00
76.68
28.00
25.21
Ph
N
Br
16e
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

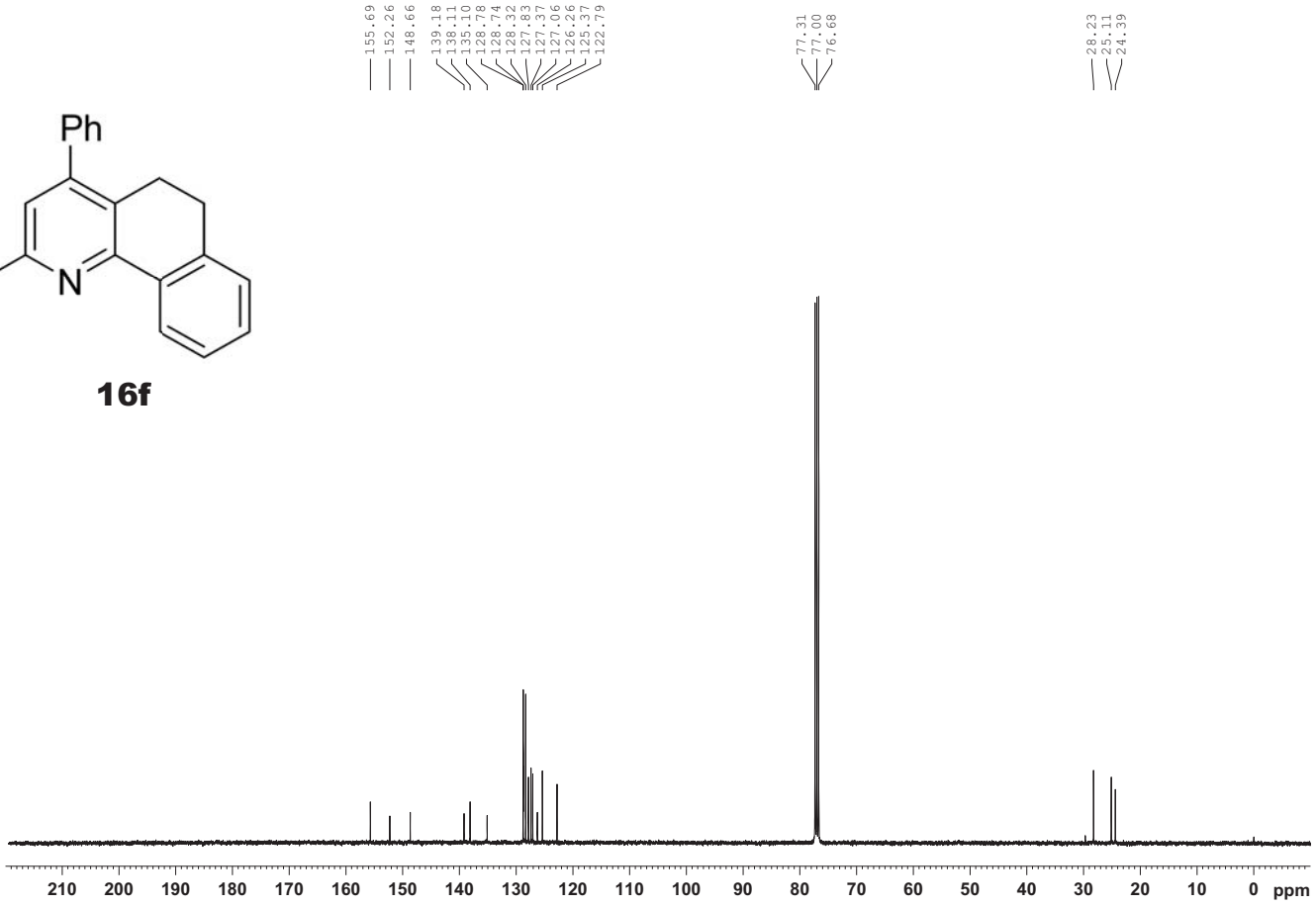

Ph
N

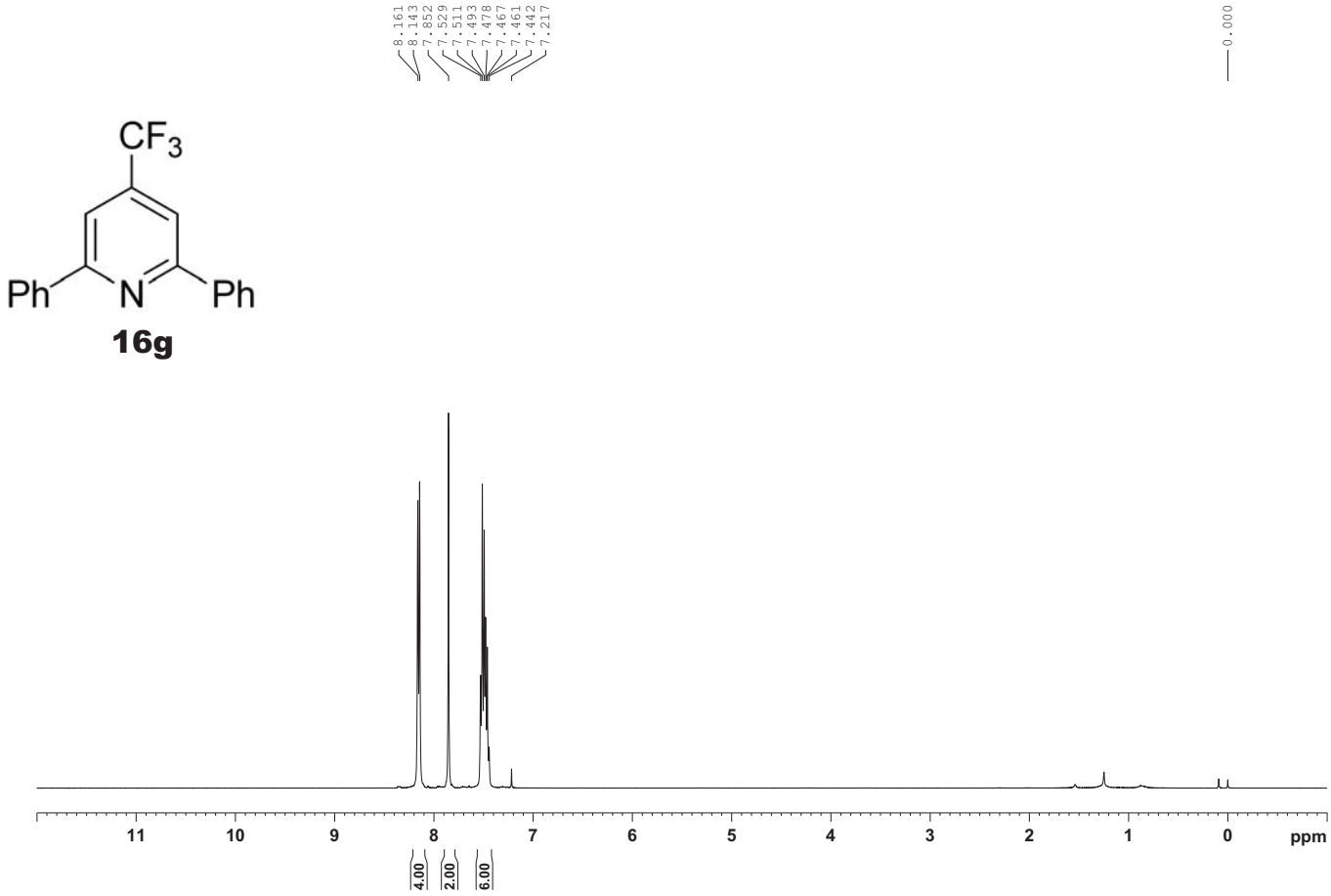
16f

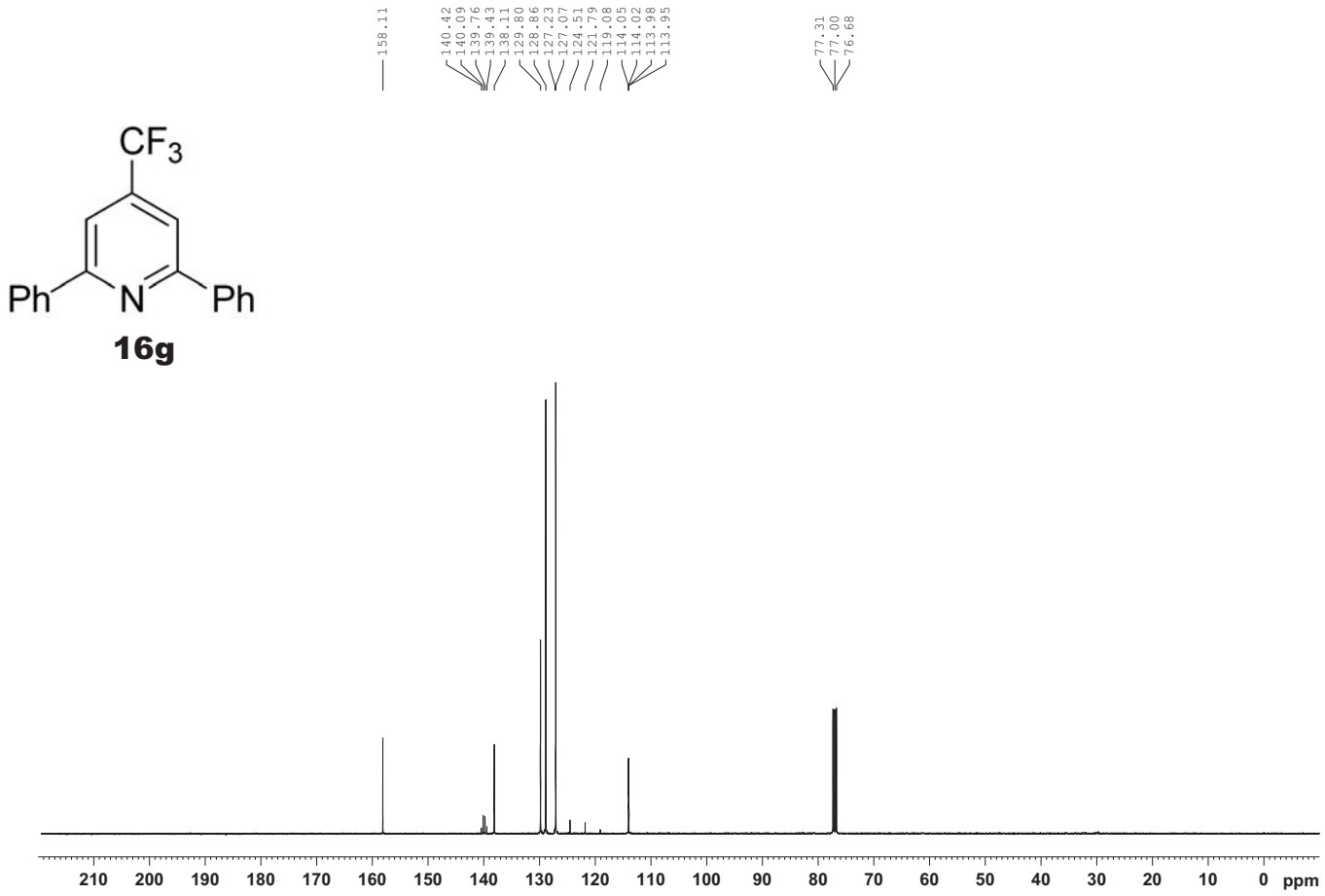

8.380
8.362
7.485
7.481
7.464
7.460
7.445
7.428
7.425
7.417
7.411
7.402
7.392
7.374
7.360
7.356
7.340
7.325
7.322
7.307
7.303
7.288
7.285
7.256
7.216
7.198
7.008
2.880
2.867
2.858
2.848
2.816
2.805
2.797
2.783
2.631
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
3.00
3.08
1.06
1.04
0.99
2.06
2.09
3.02

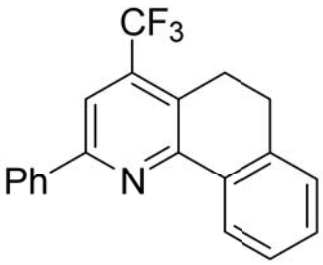

Ph
N

16f

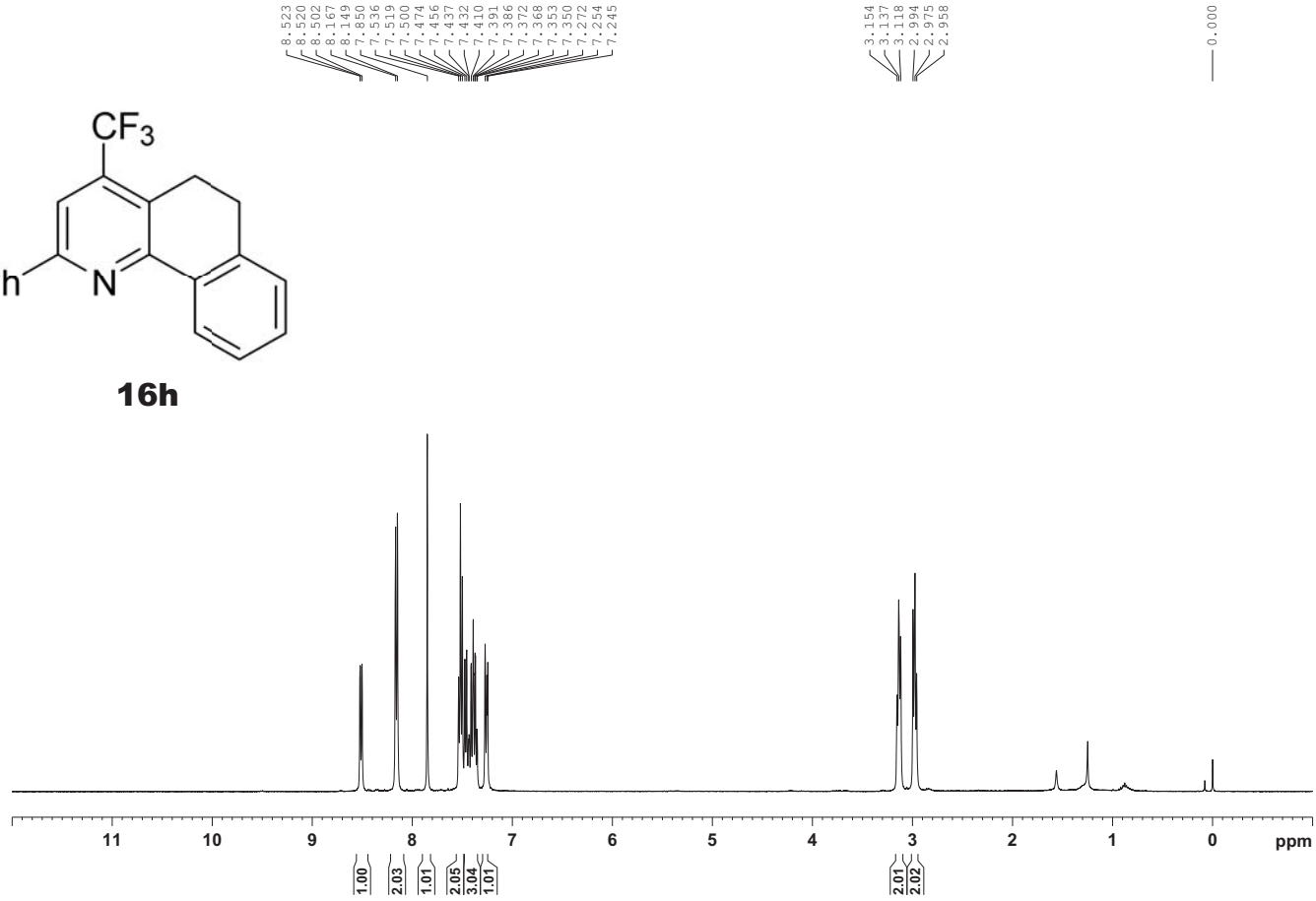

155.69
152.26
148.66
139.18
138.11
135.10
128.78
128.74
128.32
127.83
127.37
127.06
126.26
125.37
122.79
77.31
77.00
76.68
28.23
25.11
24.39
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.161
8.143
7.852
7.529
7.511
7.493
7.478
7.467
7.461
7.442
7.217
0.000
CF3
Ph
N
Ph
16g
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.00
2.00
6.00


158.11
140.42
140.09
139.76
139.43
138.11
129.80
128.86
127.23
127.07
124.51
121.79
119.08
114.05
114.02
113.98
113.95
77.31
77.00
76.68
CF3
Ph
N
Ph
16g
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


CF3
Ph
N

16h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7.519
7.500
7.474
7.456
7.437
7.432
7.410
7.391
7.386
7.372
7.368
7.353
7.350
7.272
7.254
7.245
3.154
3.137
3.118
2.994
2.975
2.958
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
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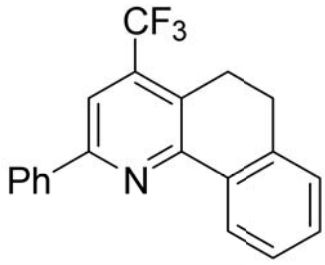
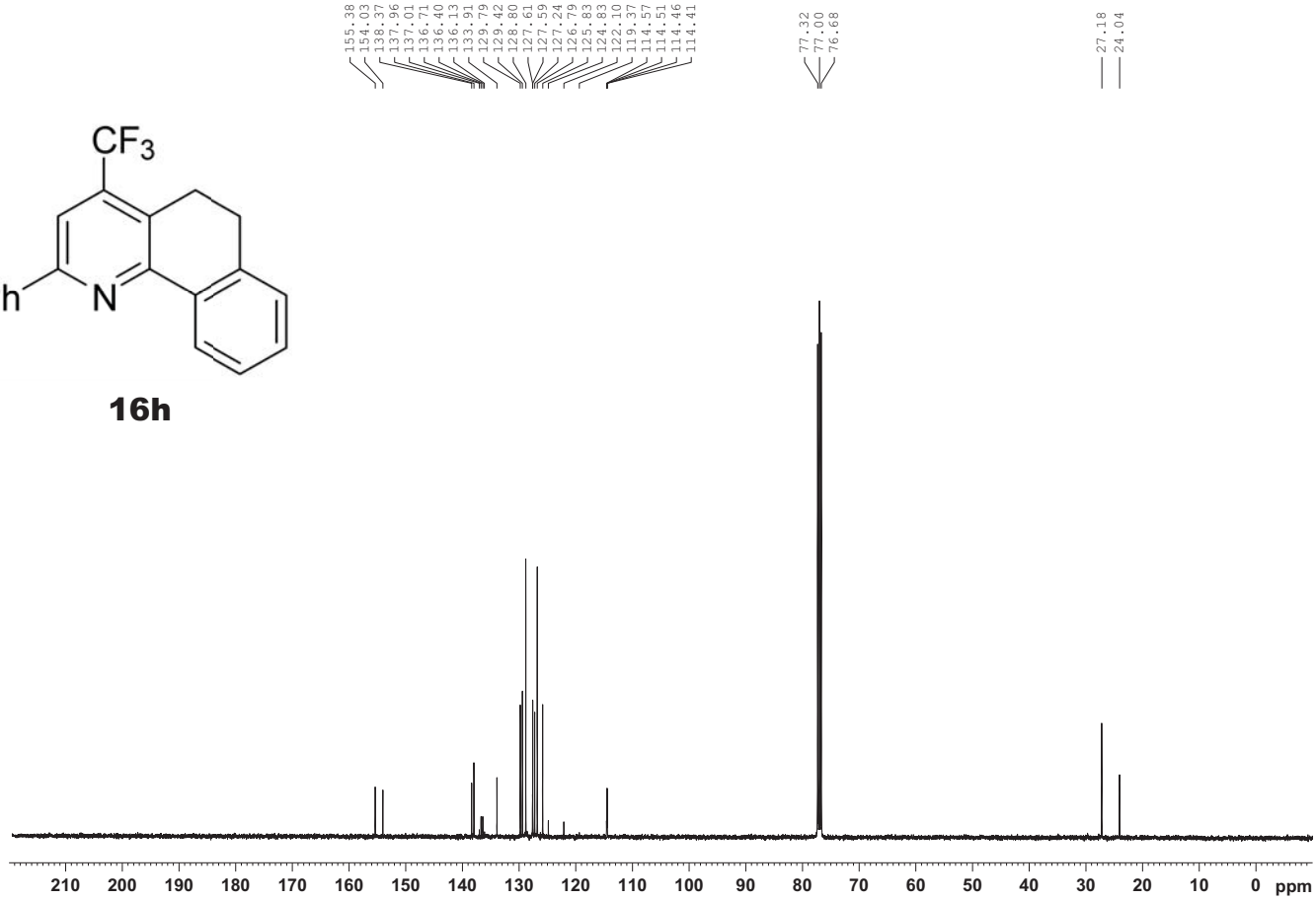
2.03
1.01
2.05
3.04
1.01
2.01
2.02


CF3
Ph
N

16h


155.38
154.03
138.37
137.96
137.01
136.71
136.40
136.13
133.91
129.79
129.42
128.80
127.61
127.59
127.24
126.79
125.83
124.83
122.10
119.37
114.57
114.51
114.46
114.41
77.32
77.00
76.68
27.18
24.04
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm