Supporting Information

for

Cobalt-catalyzed directed C–H alkenylation of pivalophenone N–H imine with alkenyl phosphates

Wengang Xu and Naohiko Yoshikai*

Address: Division of Chemistry and Biological Chemistry, School of Physical and Mathematical Sciences, Nanyang Technological University, Singapore 637371, Singapore

Email: Naohiko Yoshikai - nyoshikai@ntu.edu.sg

*Corresponding author

Experimental details and characterization data of new compounds

Table of Contents

Material and methods

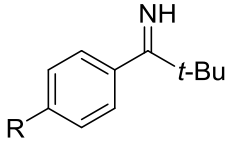
General. All reactions dealing with air and moisture-sensitive compounds were carried out in oven-dried reaction vessels under nitrogen atmosphere. Analytical thin layer chromatography (TLC) was performed on Merck 60 F254 silica gel plates. Flash column chromatography was performed using 40–63μm silica gel (Si 60, Merck). ^{1}H, ^{13}C and ^{31}P nuclear magnetic resonance (NMR) spectra were recorded on a JEOL ECA-400 (400 MHz), Bruker AV-300 (300 MHz), or AV-400 (400 MHz) NMR spectrometer. ^{1}H and ^{13}C NMR spectra are reported in parts per million (ppm) downfield from the internal standard, tetramethylsilane (0 ppm), and ^{31}P NMR spectra are reported in reference to an external standard, 85% phosphoric acid (0 ppm). Gas chromatography (GC) analysis was performed on a Shimadzu GC-2010 system equipped with glass capillary column DB-5 (Agilent J&W, 0.25 mm id × 30 m, 0.25 μm film thickness). High-resolution mass spectra (HRMS) were obtained with a Q-Tof Premier LC HR mass spectrometer. Melting points were determined using a capillary melting point apparatus and are uncorrected.

Materials. Unless otherwise noted, commercial reagents were purchased from Aldrich, Alfa Aesar, and other commercial suppliers and were used as received. THF was distilled over Na/benzophenone. Anhydrous $CoBr_2$ (>98%) was purchased from Alfa Aesar and used as received. Grignard reagents were prepared from the corresponding alkyl halides and magnesium turnings in THF, and titrated before use. IEt·HCl,[1] **L1**·HBr,[2] and **L2**·HCl[3] were prepared according to literature procedures.

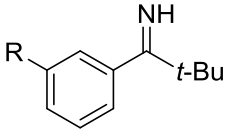
Preparation of the starting materials

Preparation of aryl imines

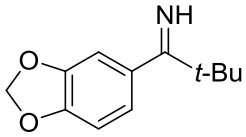
All imines shown below were synthesized according to the literature procedures[4] and purified by recrystallization or distillation. Spectral data for **1a**[5], **1b,c**[6], **1d**[4], **1e–g**[6], **1h**[5], and **1i–l**[6], showed good agreement with the literature data.



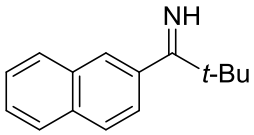
1a (R = H)
1b (R = Me)
1c (R = Ph)
1d (R = OMe)
1e (R = OCF_3)
1f (R = NMe_2)
1g (R = F)



1h (R = Me)
1i (R = OMe)
1j (R = F)



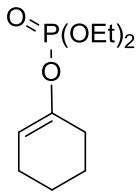
1k



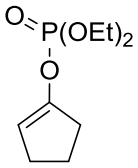
1l

Preparation of alkenyl phosphates

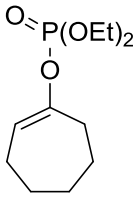
Enol phosphate esters **2a–j** were prepared according to the procedure described by Kumada and coworkers[7]. Spectral data for **2a**[7], **2b**[7], **2c**[8], **2f**[7], and **2i**[7] showed good agreement with the literature data.



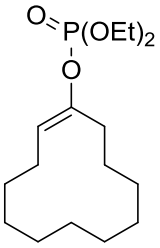
2a



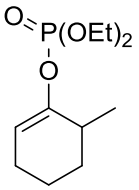
2b



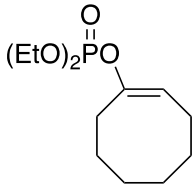
2c



2f

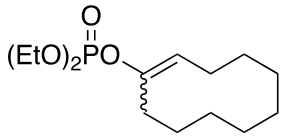


2i

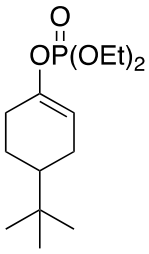


(*E*)-Cyclooct-1-en-1-yl diethyl phosphate (**2d**): Colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.37 (tt, *J* = 8.6, 2.2 Hz, 1H), 4.14–4.04 (m, 4H), 2.32–2.26 (dd, *J* = 8.2, 4.2 Hz, 2H), 2.06–1.97 (m, 2H), 1.64–1.55 (m, 2H), 1.54–1.43 (m, 6H), 1.33–1.25 (m, 6H); ^{13}C NMR (100 MHz,

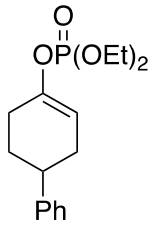
$CDCl_3$): δ 149.7 (d, $J_{C\text{-}P}$ = 9.3 Hz), 112.7 (d, $J_{C\text{-}P}$ = 4.9 Hz), 64.1 (d, $J_{C\text{-}P}$ = 6.0 Hz), 30.1, 29.4 (d, $J_{C\text{-}P}$ = 4.4 Hz), 27.6, 26.2, 26.0, 25.2, 16.2 (d, $J_{C\text{-}P}$ = 6.9 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ -5.6; HRMS (ESI) Calcd for $C_{12}H_{24}O_4P$ $[M + H]^+$ 263.1412, found 263.1408.



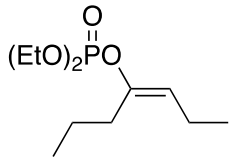
Cyclodec-1-en-1-yl diethyl phosphate (*E*/*Z* = ca. 1:1) (**2e**): Light yellow liquid; ^1H NMR (400 MHz, $CDCl_3$, both isomers): δ 5.21 (td, *J* = 8.7, 2.1 Hz, 1H, minor), 4.99 (td, *J* = 7.8, 0.9 Hz, 1H, major), 4.14–4.04 (m, 4H, both), 2.38–2.09 (m, 4H, both), 1.57–1.25 (m, 18H, both); ^{13}C NMR (100 MHz, $CDCl_3$, both isomers): δ 148.1 (d, *J* = 9.3 Hz), 147.0 (d, *J* = 7.6 Hz), 117.4 (d, *J* = 6.9 Hz), 114.9 (d, *J* = 4.5 Hz), 64.13 (d, *J* = 6.2 Hz), 64.06 (d, *J* = 6.2 Hz), 34.8, 27.4 (d, *J* = 4.3 Hz), 27.1, 26.9, 26.1 (two signals overlapped), 25.5, 25.4, 25.2, 25.0, 24.6 (two signals overlapped), 24.2, 23.9, 20.9, 20.6, 16.22 (d, *J* = 6.6 Hz), 16.18 (d, *J* = 6.6 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ -5.4. HRMS (ESI) Calcd for $C_{14}H_{28}O_4P$ $[M + H]^+$ 291.1725, found 291.1728.



4-(*tert*-Butyl)cyclohex-1-en-1-yl diethyl phosphate (**2g**): Light yellow liquid; ^1H NMR (400 MHz, $CDCl_3$): δ 5.41 (dd, *J* = 5.1, 1.7 Hz, 1H), 4.13-4.09 (m, 4H), 2.28–2.00 (m, 3H), 1.86–1.76 (m, 2H), 1.34–1.21 (m, 8H), 0.83 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.8 (d, $J_{C\text{-}P}$ = 8.6 Hz), 110.6 (d, $J_{C\text{-}P}$ = 5.4 Hz), 64.2 (d, $J_{C\text{-}P}$ = 6.0 Hz), 43.5, 32.2, 28.9 (d, $J_{C\text{-}P}$ = 3.8 Hz), 27.4, 25.1, 24.1, 16.3 (d, $J_{C\text{-}P}$ = 6.8 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ -5.4; HRMS (ESI) Calcd for $C_{14}H_{28}O_4P$ $[M + H]^+$ 291.1725, found 291.1729.

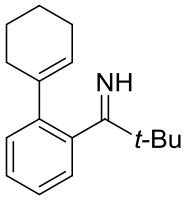


Diethyl (1,2,3,6-tetrahydro-[1,1'-biphenyl]-4-yl) phosphate (**2h**): Light yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29 (t, *J* = 7.5 Hz, 2H), 7.22–7.17 (m, 3H), 5.59–5.55 (m, 1H), 4.21–4.12 (m, 4H), 2.83–2.75 (m, 1H), 2.49–2.19 (m, 4H), 2.03–1.83 (m, 2H), 1.36 (t, *J* = 7.1 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.5 (d, $J_{C\text{-}P}$ = 9.0 Hz), 145.7, 128.4, 126.8, 126.2, 110.1 (d, $J_{C\text{-}P}$ = 5.4 Hz), 64.1 (d, $J_{C\text{-}P}$ = 6.0 Hz), 39.3, 31.5, 29.6, 28.1 (d, $J_{C\text{-}P}$ = 4.1 Hz), 16.1 (d, $J_{C\text{-}P}$ = 6.7 Hz); ^{31}P{^{1}H} NMR (162 MHz, $CDCl_3$): δ -5.5; HRMS (ESI) Calcd for $C_{16}H_{24}O_4P$ $[M + H]^+$ 311.1412, found 311.1413.



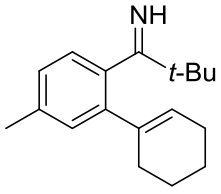
(*E*)-Diethyl hept-3-en-4-yl phosphate (**2j**): Light yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.45–5.41 (m, 1H), 4.14–4.06 (m, 4H), 2.34 (dd, *J* = 12.8, 6.0 Hz, 1H), 2.04–1.98 (m, 2H), 1.82–1.74 (m, 1H), 1.63–1.32 (m, 4H), 1.30 (t, *J* = 7.0 Hz, 6H), 1.05 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.6 (d, $J_{C\text{-}P}$ = 9.6 Hz), 110.1 (d, $J_{C\text{-}P}$ = 4.0 Hz), 64.2 (d, $J_{C\text{-}P}$ = 5.4 Hz), 32.2 (d, $J_{C\text{-}P}$ = 4.7 Hz), 31.4, 24.2, 19.6, 18.4, 16.2 (d, $J_{C\text{-}P}$ = 6.8 Hz); ^{31}P{^{1}H} NMR (162 MHz, $CDCl_3$): δ -5.5; HRMS (ESI) Calcd for $C_{11}H_{24}O_4P$ $[M + H]^+$ 251.1412, found 251.1416.

Cobalt-catalyzed C–H alkenylation of pivalophenone imines with alkenyl phosphates



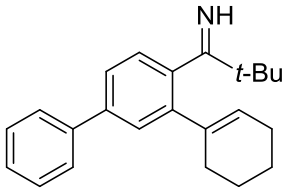
Typical procedure: 2,2-dimethyl-1-(2',3',4',5'-tetrahydro-[1,1'-biphenyl]-2-yl)propan-1-imine (3aa). A 10 mL Schlenk tube equipped with a magnetic stirring bar was charged with **L2**·HCl (9.5 mg, 0.020 mmol), $CoBr_2$ (4.4 mg, 0.020 mmol), and THF (0.30 mL). The resulting solution was cooled in an ice bath, followed by the addition of *t*-$BuCH_2MgBr$ (2.0 M in THF, 0.20 mL, 0.40 mmol). After stirring for 30 min, 2,2-dimethyl-1-phenylpropan-1-imine (**1a**, 33 mg, 0.20 mmol) and cyclohex-1-en-1-yl diethyl phosphate (**2a**, 70 mg, 0.30 mmol) were added. The resulting mixture was warmed to room temperature, stirred for 12 h, and then filtered through a short silica gel column, which was washed with ethyl acetate (5 mL). The filtrate was concentrated under reduced pressure. Silica gel chromatography (eluent: hexane/EtOAc/NEt_3 50:1:1) of the crude product afforded the title compound as a colorless oil (43 mg, 88%).

R_f 0.54 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.33 (brs, 1H), 7.28–7.23 (m, 1H), 7.20–7.15 (m, 2H), 7.03–6.99 (m, 1H), 5.70 (brs, 1H), 2.32–2.25 (m, 2H), 2.15–2.09 (m, 2H), 1.75–1.67 (m, 2H), 1.65–1.58 (m, 2H), 1.18 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.9, 141.0, 140.5, 137.6, 129.4, 128.1, 127.8, 127.4, 126.0, 40.4, 30.3, 29.4, 25.8, 23.2, 22.0; HRMS (ESI) Calcd for $C_{17}H_{24}N$ $[M + H]^+$ 242.1909, found 242.1910.

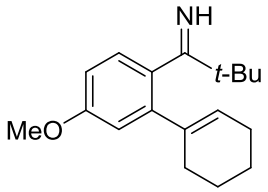


2,2-Dimethyl-1-(5-methyl-2',3',4',5'-tetrahydro-[1,1'-biphenyl]-2-yl)propan-1-imine (3ba): Light yellow oil (43 mg, 85%). R_f 0.53 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.24 (brs, 1H), 7.01–6.97 (m, 2H), 6.90 (d, J = 7.6 Hz, 1H), 5.67 (t, J = 3.4 Hz, 1H), 2.33 (s, 3H), 2.30–2.24 (m, 2H), 2.14–2.08 (m, 2H), 1.74–1.57 (m, 2H), 1.17 (s, 9H); ^{13}C NMR

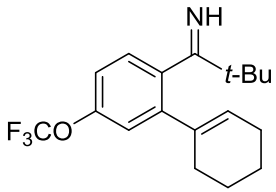
(100 MHz, $CDCl_3$): δ 192.1, 141.0, 137.8, 137.5, 129.2, 128.9 (two signals overlapped), 127.4, 126.8, 40.4, 30.4, 29.5, 25.9, 23.3, 22.1, 21.4; HRMS (ESI) Calcd for $C_{18}H_{26}N$ $[M + H]^+$ 256.2065, found 256.2062.



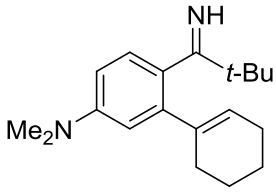
2,2-Dimethyl-1-(2'',3'',4'',5''-tetrahydro-[1,1':3',1''-terphenyl]-4'-yl)propan-1-imine (3ca): Light yellow oil (55 mg, 86%). R_f 0.48 (hexane/EtOAc/NEt_3 = 10/1/1); ^1H NMR (400 MHz, $CDCl_3$): δ 8.91 (brs, 1H), 7.60 (d, *J* = 7.1 Hz, 2H), 7.46–7.40 (m, 4H), 7.38–7.32 (m, 1H), 7.09 (d, *J* = 8.6 Hz, 1H), 5.77–5.74 (m, 1H), 2.36–2.30 (m, 2H), 2.17–2.11 (td, *J* = 6.1, 2.6 Hz, 2H), 1.76–1.70 (m, 2H), 1.67–1.61 (m, 2H), 1.21 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.8, 141.6, 140.9, 140.8, 139.5, 137.8, 129.6, 129.0, 128.0, 127.7, 127.3, 127.1, 124.8, 40.5, 30.5, 29.5, 25.9, 23.3, 22.1; HRMS (ESI) Calcd for $C_{23}H_{28}N$ $[M + H]^+$ 318.2222, found 318.2223.



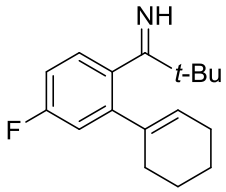
1-(5-Methoxy-2',3',4',5'-tetrahydro-[1,1'-biphenyl]-2-yl)-2,2-dimethylpropan-1-imine (3da): Light yellow oil (46 mg, 83%). R_f 0.37 (hexane/EtOAc/NEt_3 = 10/1/1); ^1H NMR (400 MHz, $CDCl_3$): δ 8.85 (brs, 1H), 6.94 (d, *J* = 9.2 Hz, 1H), 6.73–6.69 (m, 2H), 5.71–5.68 (m, 1H), 3.79 (s, 3H), 2.28–2.23 (m, 2H), 2.13–2.08 (m, 2H), 1.73–1.65 (m, 2H), 1.64–1.57 (m, 2H), 1.16 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.9, 159.0, 142.7, 137.7, 133.3, 129.4, 128.6, 113.5, 111.4, 55.4, 40.5, 30.2, 29.5, 25.8, 23.2, 22.0; HRMS (ESI) Calcd for $C_{18}H_{26}NO$ $[M + H]^+$ 272.2014, found 272.2015.



2,2-Dimethyl-1-(5-(trifluoromethoxy)-2',3',4',5'-tetrahydro-[1,1'-biphenyl]-2-yl)propan-1-imine (3ea): Light yellow oil (51 mg, 79%). R_f 0.52 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.45 (brs, 1H), 7.04 (s, 3H), 5.75 (s, 1H), 2.30–2.23 (m, 2H), 2.29–2.24 (m, 2H), 1.74–1.59 (m, 4H), 1.16 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.9, 148.7, 143.2, 136.7, 130.8 (two signals overlapped), 129.0, 120.7 (d, $^1J_{C\text{-}F}$ = 257.2 Hz), 120.6, 118.4, 40.6, 30.1, 29.3, 25.8, 23.1, 21.9; HRMS (ESI) Calcd for $C_{18}H_{23}NOF_3$ $[M + H]^+$ 326.1732, found 326.1732.

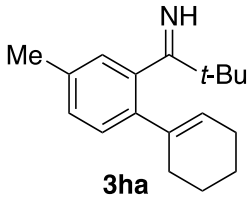


6-(1-Imino-2,2-dimethylpropyl)-*N*,*N*-dimethyl-2',3',4',5'-tetrahydro-[1,1'-biphenyl]-3-amine (3fa): Light yellow oil (47 mg, 82%). R_f 0.45 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.45 (brs, 1H), 6.91 (d, J = 8.4 Hz, 1H), 6.54 (dd, J = 8.5, 2.6 Hz, 1H), 6.51 (d, J = 2.5 Hz, 1H), 5.70–5.66 (m, 1H), 2.95 (s, 6H), 2.28–2.24 (m, 2H), 2.14–2.08 (m, 2H), 1.73–1.58 (m, 4H), 1.19 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 192.2, 150.0, 142.3, 138.7, 129.3, 128.6, 128.3, 112.0, 110.0, 40.7, 40.5, 30.4, 29.7, 25.8, 23.3, 22.1; HRMS (ESI) Calcd for $C_{19}H_{29}N_2$ $[M + H]^+$ 285.2331, found 285.2333.

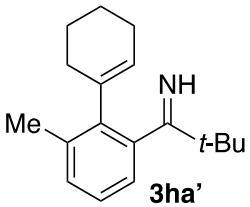


1-(5-Fluoro-2',3',4',5'-tetrahydro-[1,1'-biphenyl]-2-yl)-2,2-dimethylpropan-1-imine (3ga): Light yellow oil (42 mg, 81%). R_f 0.52 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.39 (brs, 1H), 6.98 (dd, J = 8.2, 5.9 Hz, 1H), 6.92–6.85 (m, 2H), 5.76–5.72 (m, 1H), 2.28–2.23 (m, 2H), 2.14–2.09 (m, 2H), 1.74–1.67 (m, 2H), 1.65–1.57 (m, 2H), 1.15 (s, 9H); ^{13}C

NMR (100 MHz, $CDCl_3$): δ 191.2, 162.2 (d, $^1J_{C\text{-}F}$ = 246.4 Hz), 143.4 (d, $^3J_{C\text{-}F}$ = 8.0 Hz), 136.8, 130.4, 129.2 (d, $^3J_{C\text{-}F}$ = 7.7 Hz), 114.8 (d, $^2J_{C\text{-}F}$ = 21.1 Hz), 113.0 (d, $^2J_{C\text{-}F}$ = 21.3 Hz), 40.6, 30.1, 29.4, 25.8, 23.2, 21.9; HRMS (ESI) Calcd for $C_{17}H_{23}NF$ $[M + H]^+$ 260.1815, found 260.1815.

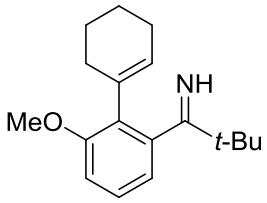


3ha

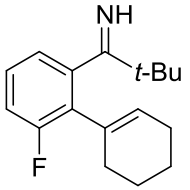


3ha'

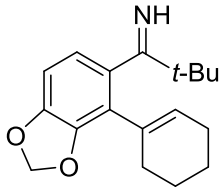
2,2-Dimethyl-1-(4-methyl-2',3',4',5'-tetrahydro-[1,1'-biphenyl]-2-yl)propan-1-imine (3ha): Light yellow oil (39 mg, 76%, obtained as an inseparable mixture with minor regioisomer **3ha'** (ratio = 3:1 as determined by ^{1}H NMR)). R_f 0.54 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$, major isomer): δ 9.28 (s, 1H), 7.07 (d, *J* = 0.6 Hz, 2H), 6.81 (s, 1H), 5.66 (s, 1H), 2.31 (s, 3H), 2.28–2.22 (m, 2H), 2.13–2.07 (m, 2H), 1.73–1.66 (m, 2H), 1.64–1.57 (m, 2H), 1.17 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, major isomer): δ 192.2, 138.2, 137.5, 135.7, 129.1, 128.6, 128.0, 125.8, 123.4, 40.4, 30.4, 29.5, 25.9, 23.3, 22.1, 21.2; HRMS (ESI) Calcd for $C_{18}H_{26}N$ $[M + H]^+$ 256.2065, found 256.2065.



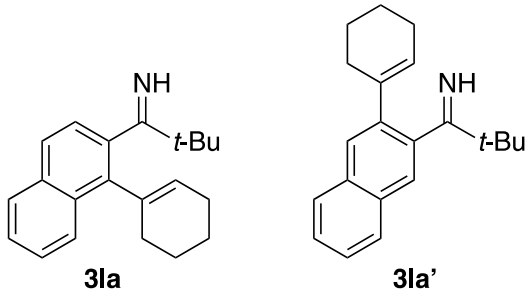
1-(6-Methoxy-2',3',4',5'-tetrahydro-[1,1'-biphenyl]-2-yl)-2,2-dimethylpropan-1-imine (3ia): Light yellow oil (43 mg, 79%). R_f 0.46 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.25 (brs, 1H), 7.15 (dd, *J* = 8.3, 7.7 Hz, 1H), 6.81 (dd, *J* = 8.4, 1.3 Hz, 1H), 6.66 (dd, *J* = 7.7, 1.2 Hz, 1H), 5.55–5.50 (m, 1H), 3.80 (s, 3H), 2.29–2.09 (m, 4H), 1.76–1.58 (m, 4H), 1.20 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.9, 157.4, 143.2, 135.0, 130.0, 129.3, 127.1, 119.1, 110.2, 56.0, 39.9, 30.0, 29.9, 25.7, 23.1, 22.2; HRMS (ESI) Calcd for $C_{18}H_{26}NO$ $[M + H]^+$ 272.2014, found 272.2017.



1-(6-Fluoro-2',3',4',5'-tetrahydro-[1,1'-biphenyl]-2-yl)-2,2-dimethylpropan-1-imine (3ja): Light yellow oil (41 mg, 79%). R_f 0.52 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.26 (brs, 1H), 7.18–7.12 (m, 1H), 7.00–6.95 (m, 1H), 6.83 (d, J = 7.6 Hz, 1H), 5.66–5.62 (m, 1H), 2.21–2.17 (m, 2H), 2.16–2.09 (m, 2H), 1.74–1.60 (m, 4H), 1.18 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.0, 160.3 (d, $^1J_{C\text{-}F}$ = 245.8 Hz), 143.5, 132.1, 131.3, 128.8 (d, $^2J_{C\text{-}F}$ = 17.1 Hz), 127.6 (d, $^3J_{C\text{-}F}$ = 8.9 Hz), 122.6, 115.1 (d, $^2J_{C\text{-}F}$ = 23.5 Hz), 40.2, 30.1, 29.6, 25.7, 23.0, 22.0; HRMS (ESI) Calcd for $C_{17}H_{23}NF$ $[M + H]^+$ 260.1815, found 260.1817.



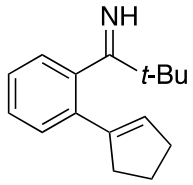
1-(4-(Cyclohex-1-en-1-yl)benzo[*d*][1,3]dioxol-5-yl)-2,2-dimethylpropan-1-imine (3ka): Light yellow oil (48 mg, 84%). R_f 0.45 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.87 (s, 1H), 6.66 (d, J = 8.0 Hz, 1H), 6.51 (d, J = 8.0 Hz, 1H), 5.94 (s, 2H), 5.73–5.69 (m, 1H), 2.33–2.27 (m, 2H), 2.16–2.10 (m, 2H), 1.73–1.59 (m, 4H), 1.16 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.0, 146.8, 144.8, 132.6, 131.2, 123.4, 120.8, 106.4, 101.0, 100.2, 40.4, 29.5, 29.0, 25.8, 23.0, 22.0; HRMS (ESI) Calcd for $C_{17}H_{24}N$ $[M + H]^+$ 242.1909, found 242.1912.



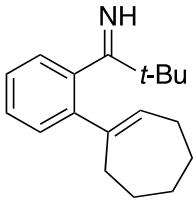
3la **3la'**

1-(1-(Cyclohex-1-en-1-yl)naphthalen-2-yl)-2,2-dimethylpropan-1-imine (3la): Light yellow oil (48 mg, 82%, obtained as an inseparable mixture with minor regioisomer **3la'** (ratio = 4:1 as determined by ^{1}H NMR)). R_f 0.52 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$,

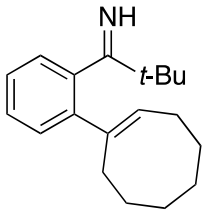
major isomer): δ 9.29 (s, 1H), 7.97–7.93 (m, 1H), 7.84–7.77 (m, 1H), 7.70 (d, *J* = 8.4 Hz, 1H), 7.50–7.44 (m, 2H), 7.20 (d, *J* = 8.5 Hz, 1H), 5.72–5.68 (m, 1H), 2.31–2.15 (m, 4H), 1.82–1.63 (m, 4H), 1.28 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, major isomer): δ 191.3, 138.8, 138.1, 136.1, 133.1, 132.4, 129.7, 128.2, 126.6, 126.5, 126.4, 126.1, 124.1, 39.9, 32.3, 30.4, 25.7, 23.2, 22.3; HRMS (ESI) Calcd for $C_{21}H_{26}N$ $[M + H]^+$ 292.2065, found 292.2068.



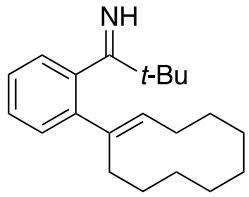
1-(2-(Cyclopent-1-en-1-yl)phenyl)-2,2-dimethylpropan-1-imine (3ab): Light yellow oil (39 mg, 85%). R_f 0.48 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.41 (brs, 1H), 7.27–7.25 (m, 2H), 7.20–7.16 (m, 1H), 7.01 (d, J = 7.7 Hz, 1H), 5.85–5.82 (m, 1H), 2.68–2.63 (m, 2H), 2.50–2.41 (m, 2H), 1.99–1.89 (m, 2H), 1.15 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 192.2, 142.6, 135.1, 134.8, 131.8, 127.8, 127.7, 127.5, 126.4, 47.4, 36.6, 34.0, 29.0, 23.7; HRMS (ESI) Calcd for $C_{16}H_{22}N$ $[M + H]^+$ 228.1752, found 228.1757.



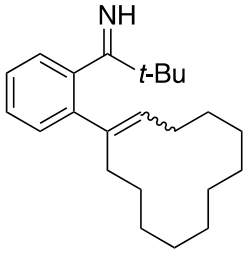
1-(2-(Cyclohept-1-en-1-yl)phenyl)-2,2-dimethylpropan-1-imine (3ac): Light yellow oil (40 mg, 79%). R_f 0.52 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.34 (brs, 1H), 7.23 (dd, *J* = 8.1, 1.8 Hz, 1H), 7.18–7.14 (m, 2H), 7.02 (dd, *J* = 7.3, 1.9 Hz, 1H), 5.80 (t, *J* = 6.5 Hz, 1H), 2.45–2.41 (m, 2H), 2.22–2.17 (m, 2H), 1.82–1.76 (m, 2H), 1.67–1.61 (m, 2H), 1.57–1.51 (m, 2H), 1.18 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.5, 144.7, 143.3, 134.1, 128.6, 128.2, 127.8, 127.1, 125.9, 43.7, 35.9, 32.7, 29.7, 29.2, 27.4, 26.8; HRMS (ESI) Calcd for $C_{18}H_{26}N$ $[M + H]^+$ 256.2065, found 256.2065.



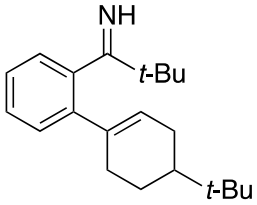
1-(2-(Cyclooct-1-en-1-yl)phenyl)-2,2-dimethylpropan-1-imine (3ad): Light yellow oil (46 mg, 86%). R_f 0.54 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.25 (brs, 1H), 7.26–7.23 (m, 1H), 7.21–7.17 (m, 2H), 7.05 (d, *J* = 7.3 Hz, 1H), 5.64 (t, *J* = 8.3 Hz, 1H), 2.45–2.40 (m, 2H), 2.24–2.17 (m, 2H), 1.64–1.51 (m, 8H), 1.18 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.8, 142.0, 140.9, 140.7, 132.4, 129.2, 127.8, 127.0, 126.1, 40.3, 31.4, 29.8 (two signals overlapped), 29.5, 27.2, 27.0, 26.5; HRMS (ESI) Calcd for $C_{19}H_{28}N$ $[M + H]^+$ 270.2222, found 270.2226.



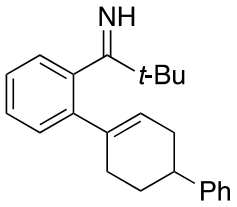
(*E*)-1-(2-(cyclodec-1-en-1-yl)phenyl)-2,2-dimethylpropan-1-imine (3ae): Light yellow oil (49 mg, 82%, *E*/*Z* > 10:1 as judged from ^{1}H NMR). R_f 0.54 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.36 (brs, 1H), 7.26–7.23 (m, 1H), 7.21–7.17 (m, 2H), 7.05 (d, *J* = 7.3 Hz, 1H), 5.64 (t, *J* = 8.3 Hz, 1H), 2.45–2.40 (m, 2H), 2.24–2.17 (m, 2H), 1.64–1.51 (m, 8H), 1.18 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.8, 141.5, 140.8 (two signals overlapped), 133.5, 130.0, 128.9, 126.5, 126.2, 40.3, 30.0, 28.3, 28.0, 27.4, 27.1, 25.5, 25.1, 21.5, 21.0; HRMS (ESI) Calcd for $C_{21}H_{32}N$ $[M + H]^+$ 298.2535, found 298.2531. The *E*-stereochemistry was assigned in light of our previous study on a related C–H alkenylation reaction.[2]



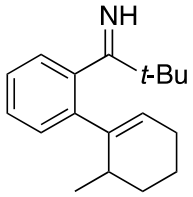
1-(2-(Cyclododec-1-en-1-yl)phenyl)-2,2-dimethylpropan-1-imine (*E*/*Z* = 4/1) (3af): Light yellow oil (51 mg, 78%). R_f 0.56 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$, both isomers): δ 9.33 (brs, 1H, both isomers), 7.35–7.09 (m, 3H, both isomers), 7.04 (dd, *J* = 7.4, 1.4 Hz, 1H, major isomer), 6.97 (dd, *J* = 7.5, 0.9 Hz, 1H, minor isomer), 5.78 (t, *J* = 7.9 Hz, 1H minor isomer), 5.34 (t, *J* = 7.9 Hz, 1H major isomer), 2.46–2.16 (m, 4H, both isomers), 1.60–1.19 (m, 25H, both isomers); ^{13}C NMR (100 MHz, $CDCl_3$, major isomer): δ 191.3, 141.7, 141.4, 140.7, 133.3, 131.3, 127.8, 126.4, 126.1, 47.4, 40.3, 29.9, 27.6, 27.3, 25.7, 25.2, 24.9, 24.8, 24.7, 22.62, 22.56; HRMS (ESI) Calcd for $C_{23}H_{37}N$ $[M + H]^+$ 326.2848, found 326.2846. The E-stereochemistry of the major isomer was assigned in light of our previous study on a related C–H alkenylation reaction.[2]



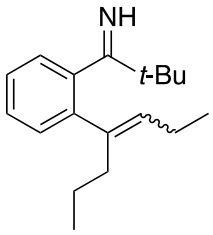
1-(4'-(*tert*-Butyl)-2',3',4',5'-tetrahydro-[1,1'-biphenyl]-2-yl)-2,2-dimethylpropan-1-imine (3ag): Light yellow oil (50 mg, 84%). R_f 0.64 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.27 (brs, 1H), 7.28–7.23 (m, 1H), 7.20–7.15 (m, 2H), 7.01 (d, *J* = 7.2 Hz, 1H), 5.72–5.68 (m, 1H), 2.45–2.11 (m, 3H), 1.94–1.82 (m, 2H), 1.32–1.14 (m, 11H), 0.89 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.9, 140.7, 140.5, 137.5, 129.7, 128.2, 127.8, 127.4, 126.1, 43.7, 40.4, 32.4, 31.9, 29.5, 27.4, 24.7; HRMS (ESI) Calcd for $C_{21}H_{32}N$ $[M + H]^+$ 298.2535, found 298.2534.



2,2-Dimethyl-1-(2',3',4',5'-tetrahydro-[1,1':4',1''-terphenyl]-2-yl)propan-1-imine (3ah): Light yellow oil (54 mg, 85%). R_f 0.48 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.29 (brs, 1H), 7.34–7.18 (m, 8H), 7.05 (d, *J* = 7.3 Hz, 1H), 5.82–5.78 (m, 1H), 2.88–2.80 (m, 1H), 2.59–2.35 (m, 3H), 2.30–2.20 (m, 1H), 2.08–2.02 (m, 1H), 1.90–1.79 (m, 1H), 1.21 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.8, 146.9 (two signals overlapped), 140.5, 137.7, 128.9, 128.7, 128.3, 128.0, 127.5, 127.1, 126.4, 126.3, 40.5, 39.6, 34.1, 31.1, 30.4, 29.5; HRMS (ESI) Calcd for $C_{23}H_{28}N$ $[M + H]^+$ 318.2222, found 318.2222.

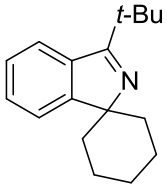


2,2-Dimethyl-1-(2'-methyl-2',3',4',5'-tetrahydro-[1,1'-biphenyl]-2-yl)propan-1-imine (3ai): Light yellow oil (45 mg, 88%). R_f 0.52 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.34 (brs, 1H), 7.26–7.17 (m, 2H), 7.11 (d, *J* = 7.3 Hz, 1H), 7.03 (d, *J* = 7.3 Hz, 1H), 5.68–5.58 (m, 1H), 2.58–2.42 (m, 1H), 2.22–2.06 (m, 2H), 1.89–1.64 (m, 2H), 1.44–1.19 (m, 11H), 0.84 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.6, 144.1, 140.8, 140.5, 130.6, 128.7, 127.7, 126.4, 126.0, 40.3, 33.0, 31.5, 30.0, 26.2, 20.3, 19.8; HRMS (ESI) Calcd for $C_{18}H_{26}NO$ $[M + H]^+$ 256.2065, found 256.2061.



1-(2-(Hept-3-en-4-yl)phenyl)-2,2-dimethylpropan-1-imine (*E*/*Z* = 4/1) (3aj): Light yellow oil (46 mg, 90%). R_f 0.52 (hexane/EtOAc/NEt_3 = 10/1/1); ^{1}H NMR (400 MHz, $CDCl_3$, major isomer): δ 9.30 (brs, 1H), 7.26–7.13 (m, 3H), 7.05–7.02 (m, 1H), 5.37 (t, *J* = 7.2 Hz, 1H), 2.29–2.24 (m, 2H), 2.14 (p, *J* = 7.5 Hz, 2H), 1.30 (q, *J* = 7.5 Hz, 2H), 1.21 (s, 9H), 1.01 (t, *J* = 7.6 Hz, 3H), 0.86 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, major isomer): δ 191.4, 141.6, 140.7, 140.5, 134.4, 130.5, 127.8, 126.6, 126.1, 40.3, 34.2, 29.9, 22.1, 21.8, 14.4, 14.2; HRMS (ESI) Calcd for $C_{18}H_{28}N$ $[M + H]^+$ 258.2222, found 258.2224. The *E*-stereochemistry of the major isomer was assigned in light of our previous study on a related C–H alkenylation reaction.[2]

Cyclization of alkenylated N–H imine under peroxide photolysis



3'-(*tert*-Butyl)spiro[cyclohexane-1,1'-isoindole] (4): 2,2-Dimethyl-1-(2',3',4',5'-tetrahydro-[1,1'-biphenyl]-2-yl)propan-1-imine (**3aa**, 48 mg, 0.20 mmol) was weighed into a 10 mL vial containing a stirring bar and then dissolved in *t*-BuO*O*t-Bu (0.5 mL). The vial was placed in Luzchem LZC-4V photoreactor and irradiated at 254 nm for 12 h. Then the mixture was concentrated under reduced pressure, and the residue was subjected to silica gel chromatography (eluent: hexane/EtOAc 100:1) to afford the title product as a yellow oil (39 mg, 81%).

R_f 0.41 (hexane/EtOAc = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72–7.69 (m, 1H), 7.55–7.51 (m, 1H), 7.33–7.29 (m, 2H), 2.05–1.93 (m, 2H), 1.82–1.72 (m, 2H), 1.71–1.56 (m, 4H), 1.49–1.40 (m, 11H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 175.4, 160.8, 137.1, 127.2, 126.7, 123.9, 122.0, 75.6, 36.3, 35.5, 28.9, 26.2, 23.6; HRMS (ESI) Calcd for $C_{17}H_{24}N$ $[M + H]^+$ 242.1909, found 242.1912.

References

(1) Bernardi, T.; Badel, S.; Mayer, P.; Groelly, J.; De Fremont, P.; Jacques, B.; Braunstein, P.; Teyssot, M.-L.; Gaulier, C.; Cisnetti, F.; Gautier, A.; Roland, S. *ChemMedChem* **2014**, *9*, 1140.

(2) Lee, P.-S.; Xu, W.; Yoshikai, N. *Adv. Synth. Catal*. **2017**, *359*, 4340.

(3) Grieco, G.; Blacque, O.; Berke, H. *Beilstein J. Org. Chem.* **2015**, *11*, 1656.

(4) He, R.; Huang, Z.-T.; Zheng, Q.-Y.; Wang, C. *Angew. Chem. Int. Ed.* **2014**, *53*, 4950.

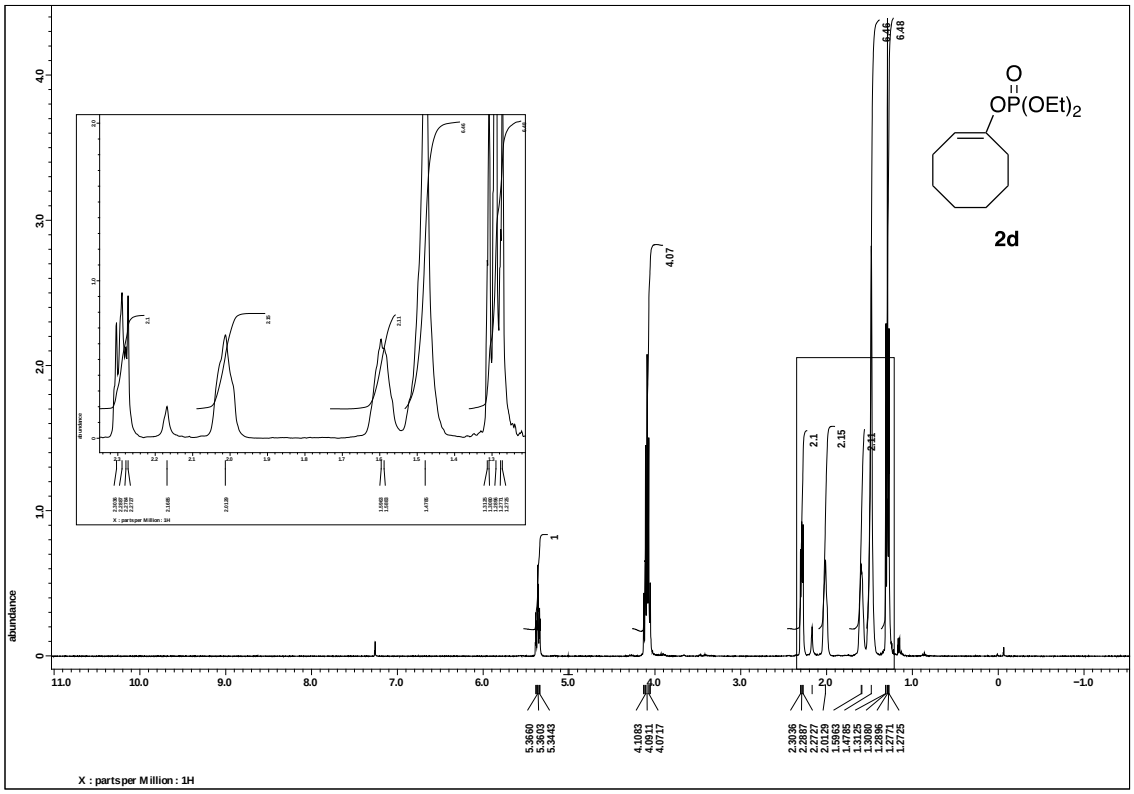
(5) Port, A.; Sanchez-Aris, M.; Cervelló, E.; Jaime, C.; Virgili, A.; Farriol, M.; Gallardo, I. *Polycyclic Aromat. Compd.* **2003**, *23*, 457.

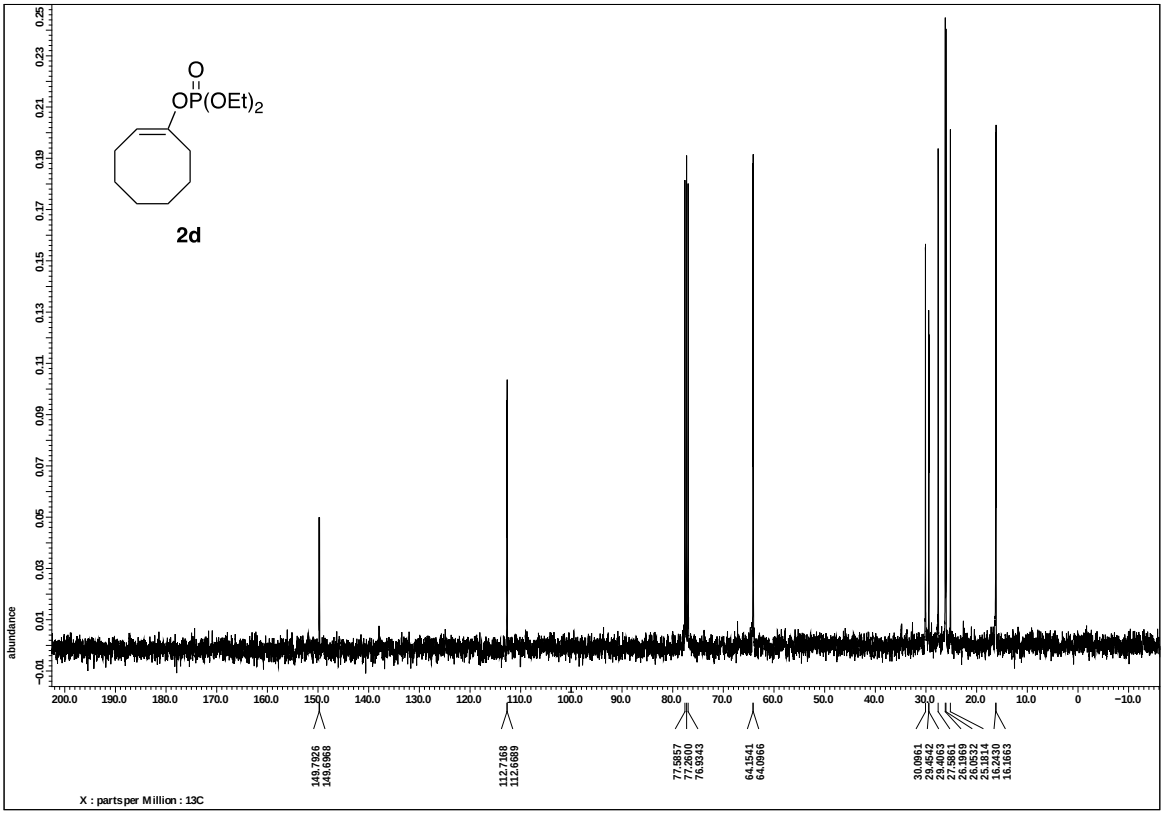
(6) Xu, W. G.; Yoshikai, N. *Chem. Sci.* **2017**, *8*, 5299.

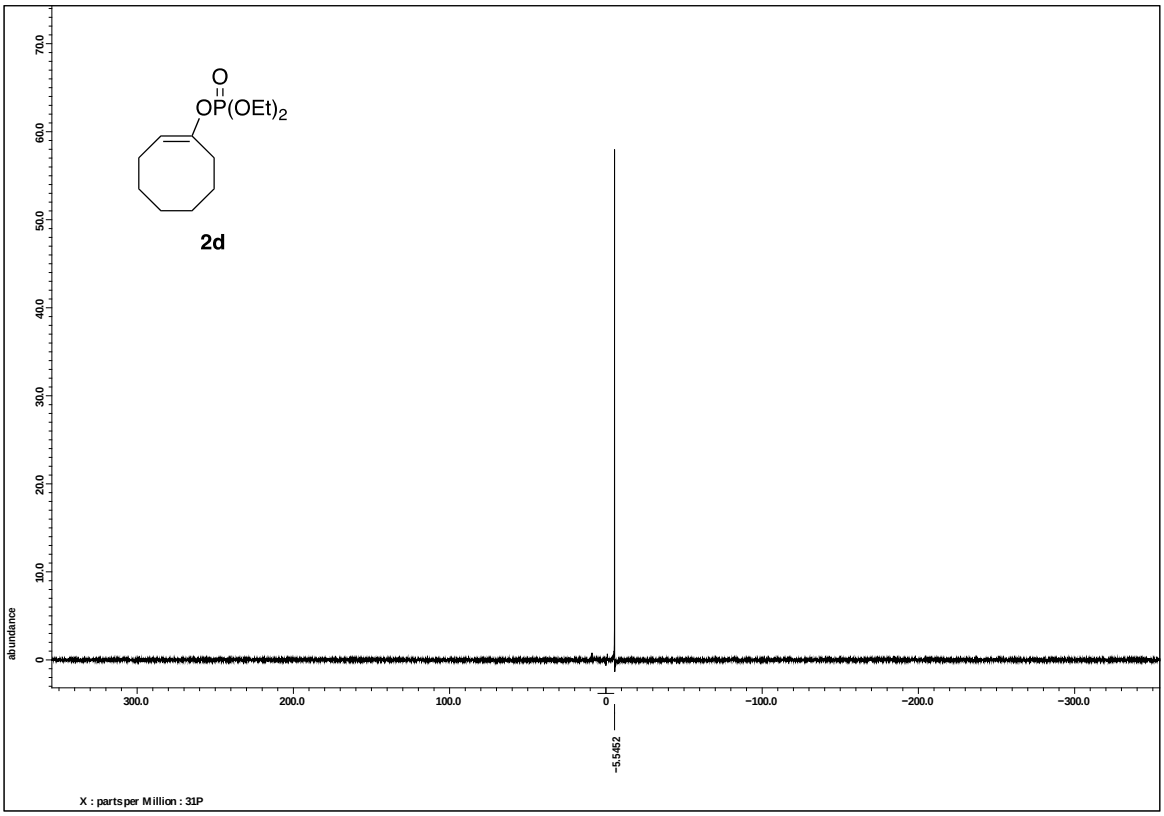
(7) Hayashi, T.; Fujiwa, T.; Okamoto, Y.; Katsuro, Y.; Kumada, M. *Synthesis* **1981**, *12*, 1001.

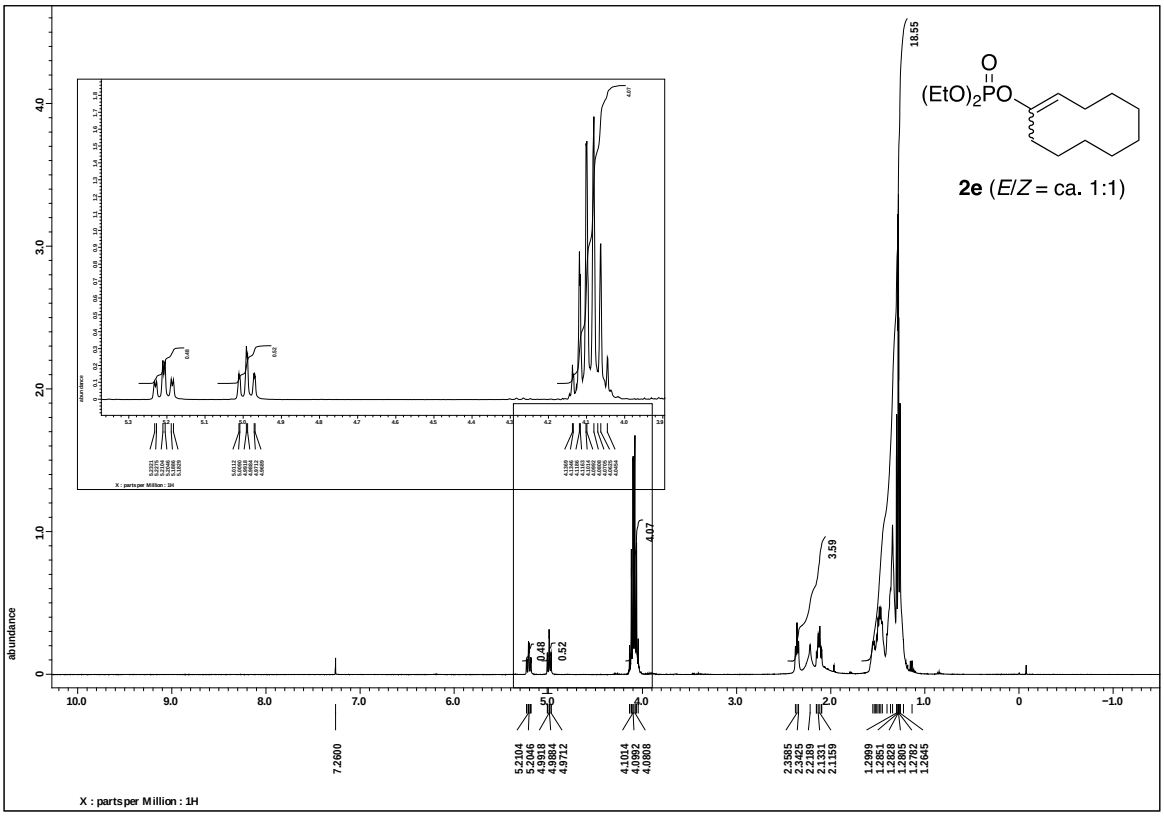
(8) Emmett, E. J.; Hayter, R. B.; Willis, C. M. *Angew. Chem. Int. Ed.* **2014**, *53*, 10204.

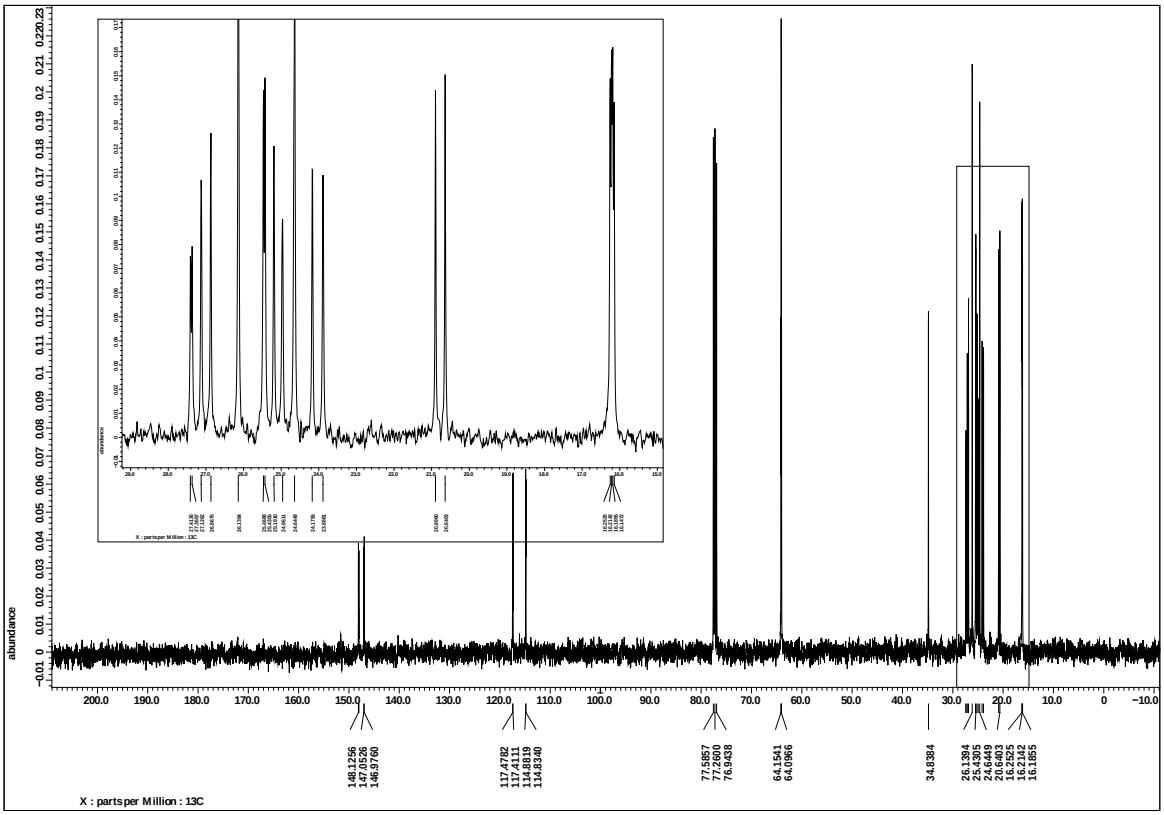
NMR Spectra

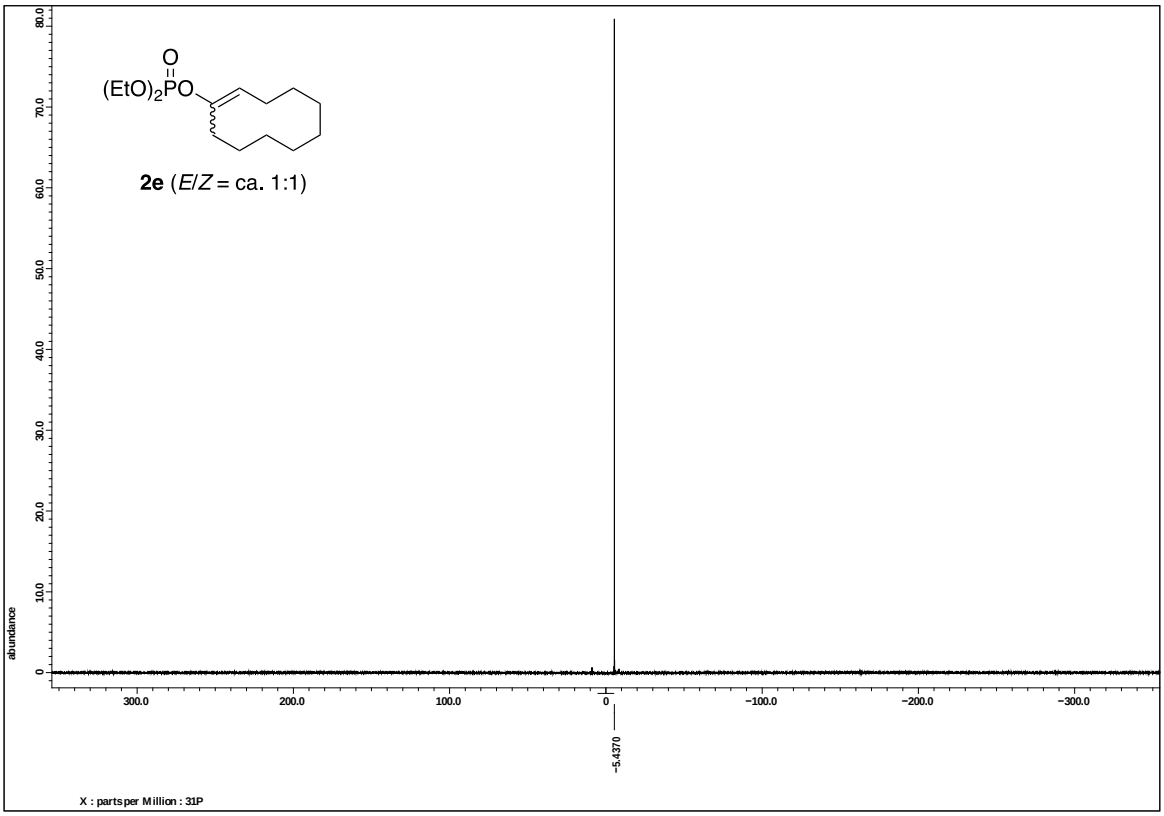

O
OP(OEt)2
2d
abundance
X : parts per Million : 1H

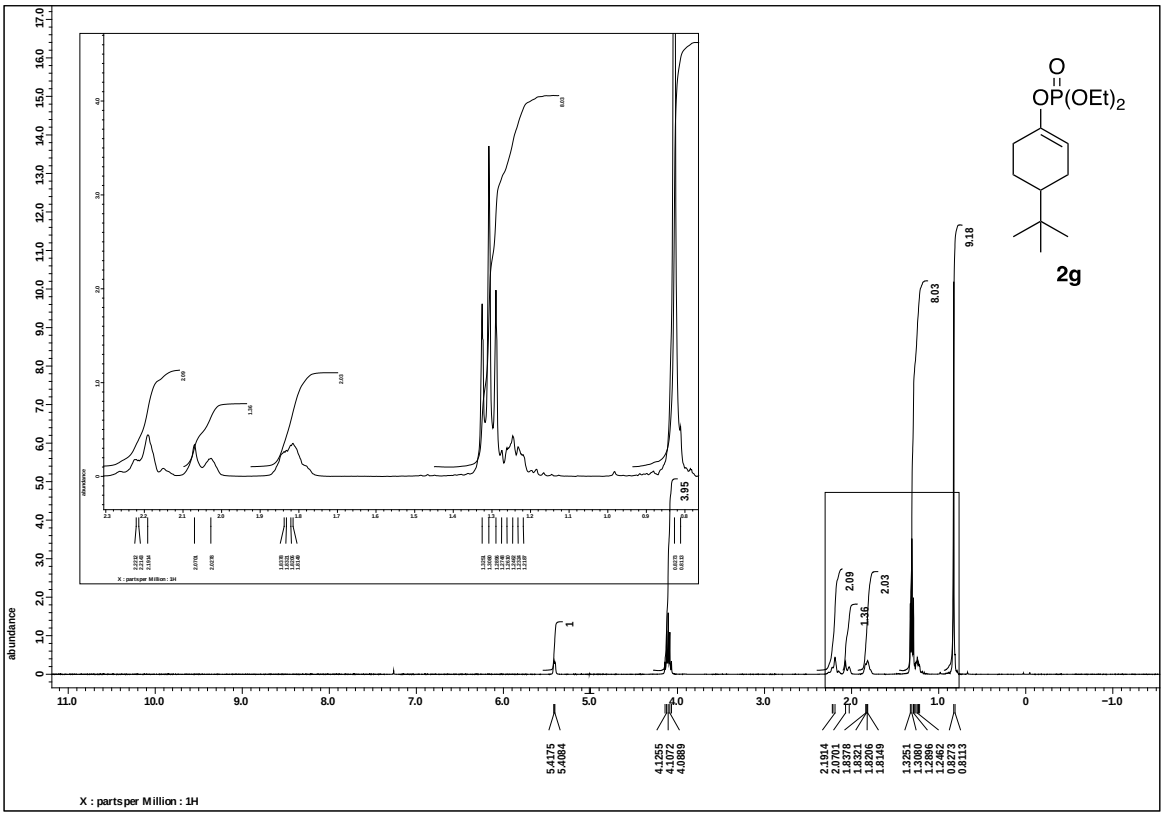

O
OP(OEt)2
2d
abundance
X : parts per Million : 13C

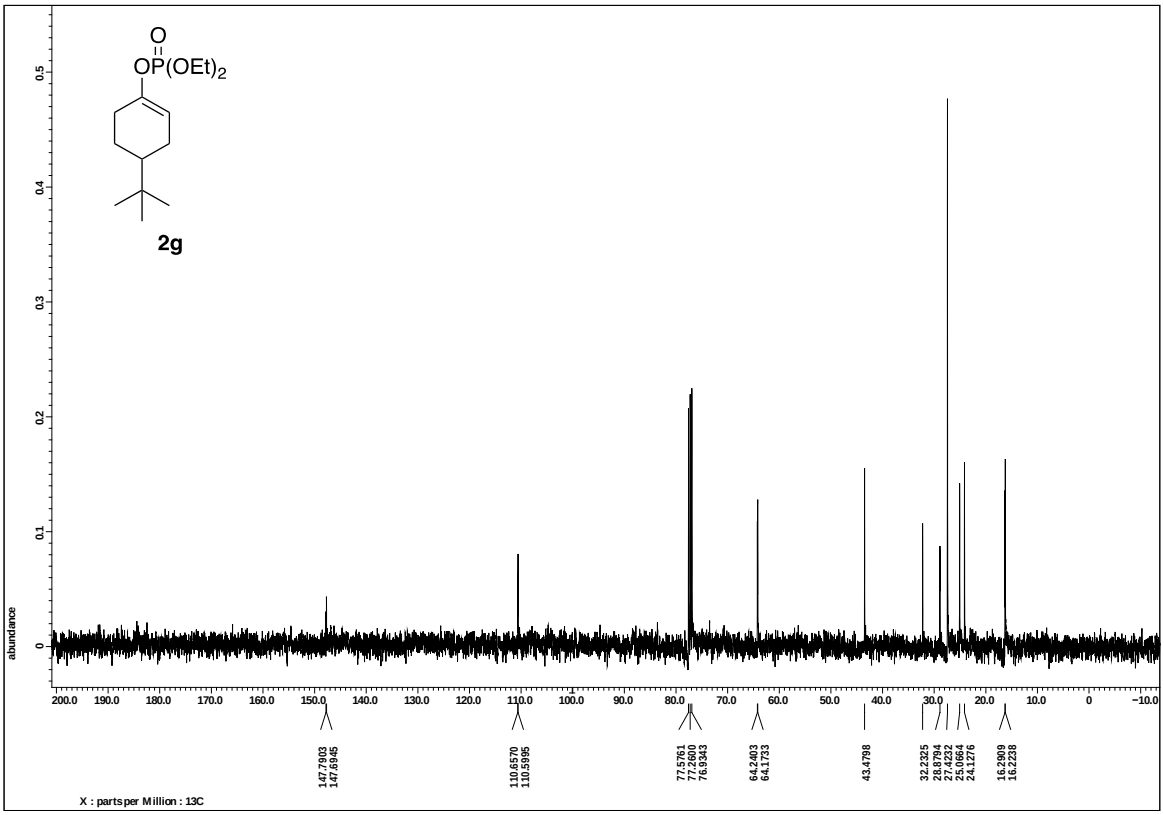

O
OP(OEt)2
2d
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
abundance
300.0
200.0
100.0
0
−100.0
−200.0
−300.0
−5.5462
X : parts per Million : 31P

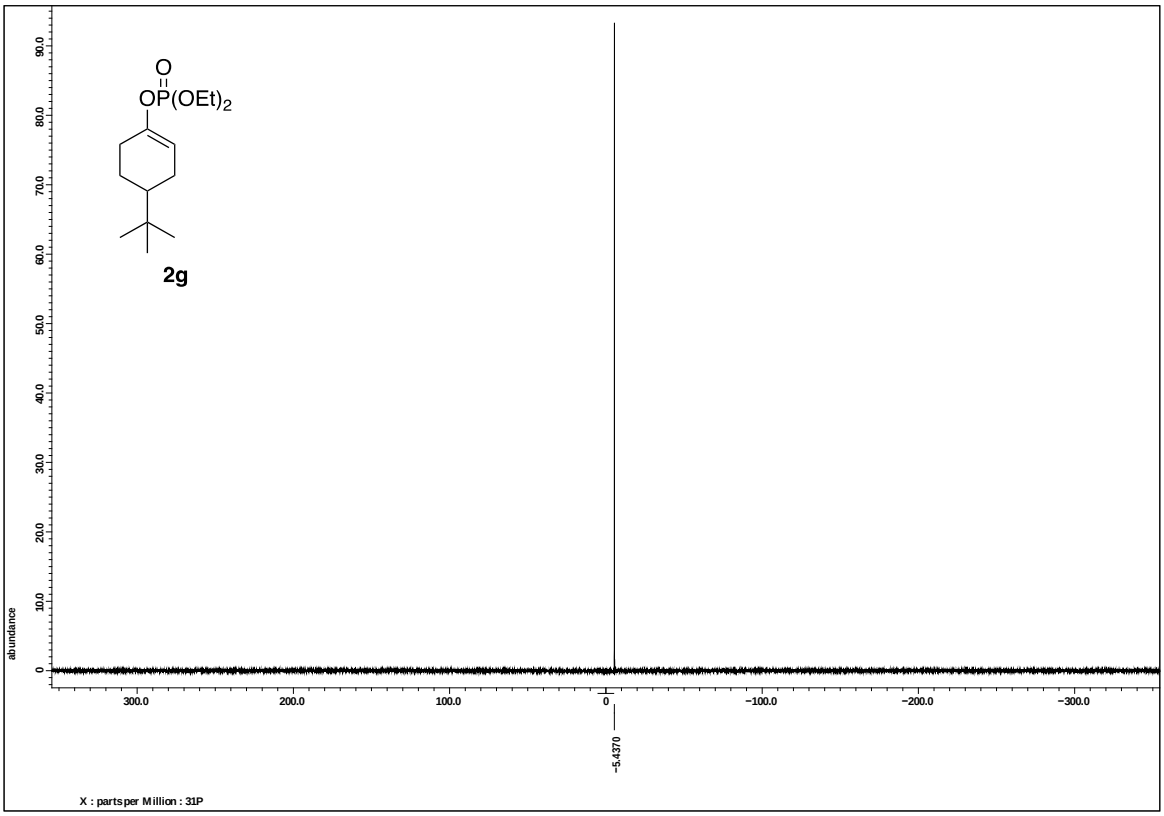

18.55
(EtO)2PO
2e (E/Z = ca. 1:1)
4.07
3.59
0.48
0.52
abundance
X : parts per Million : 1H

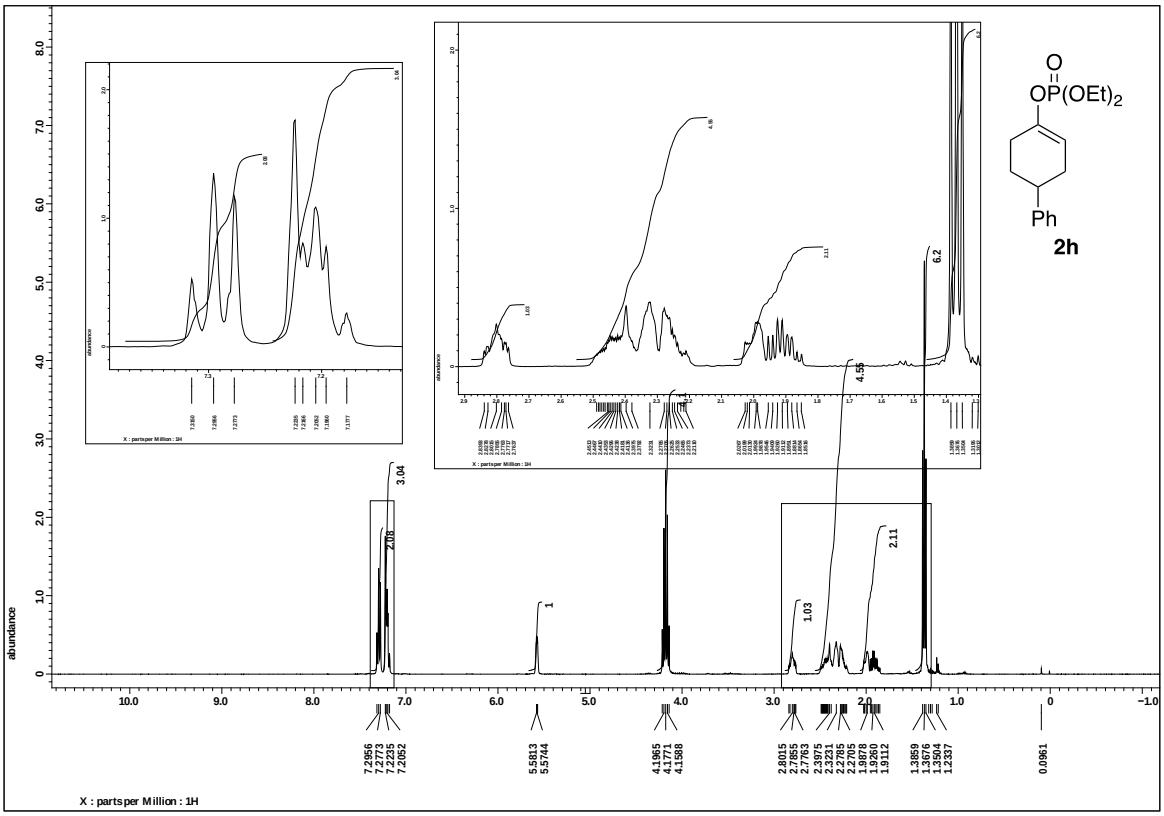

abundance
X : parts per Million : 13C

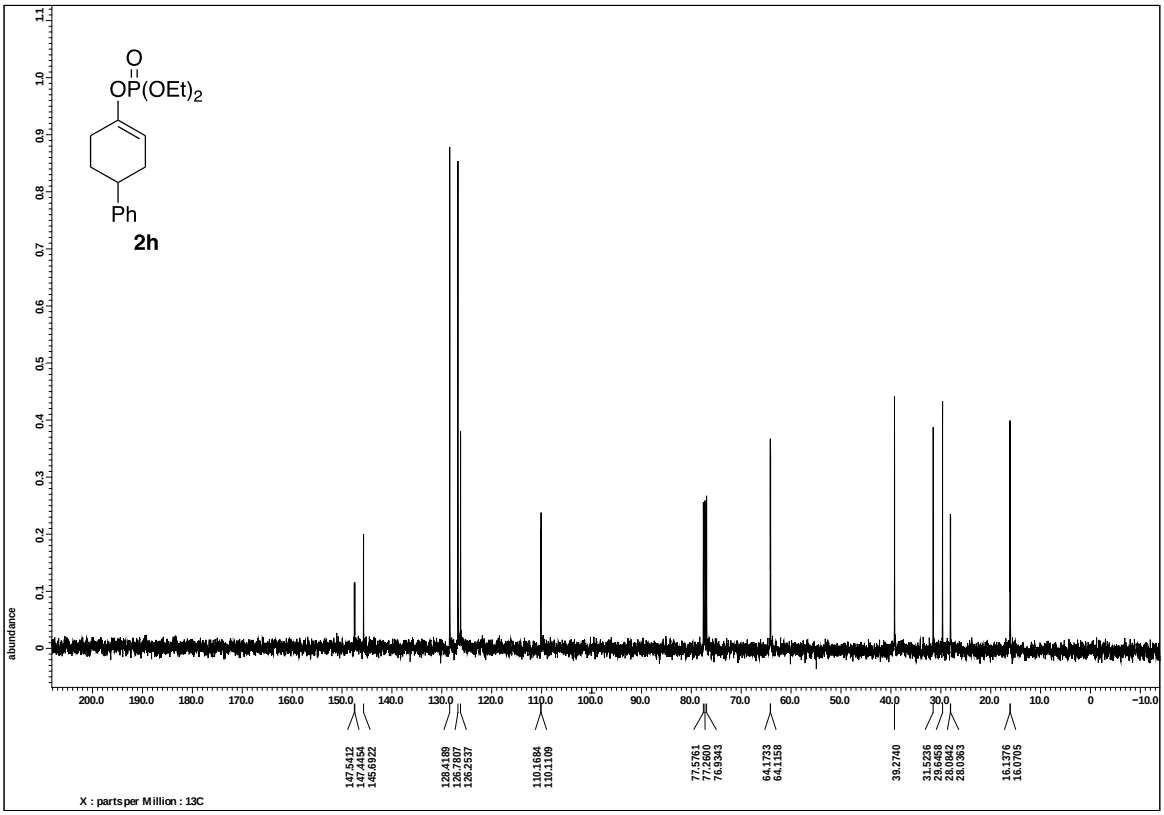

O
(EtO)2PO
2e (E/Z = ca. 1:1)
abundance
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
300.0
200.0
100.0
0
−100.0
−200.0
−300.0
−5.4370
X : parts per Million : 31P

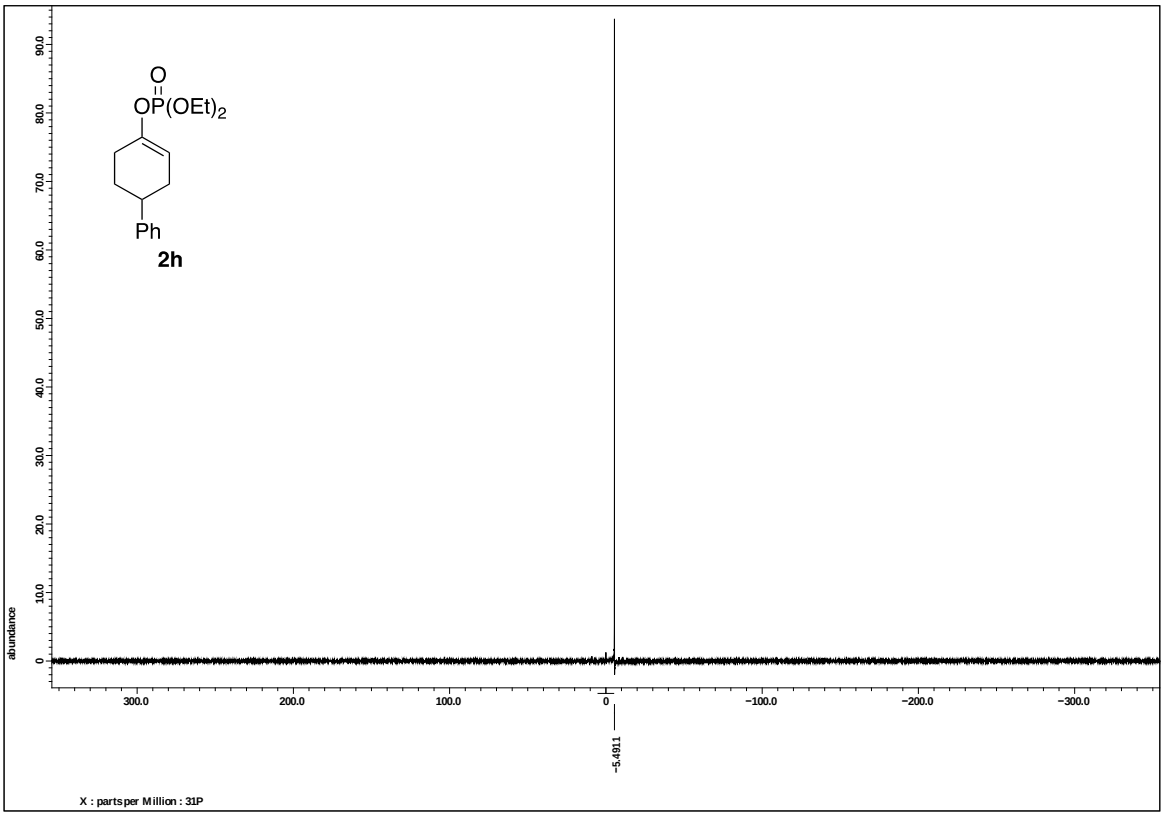

O
OP(OEt)2
2g
9.18
8.03
3.95
2.09
2.03
1.36
1
abundance
X : parts per Million : 1H

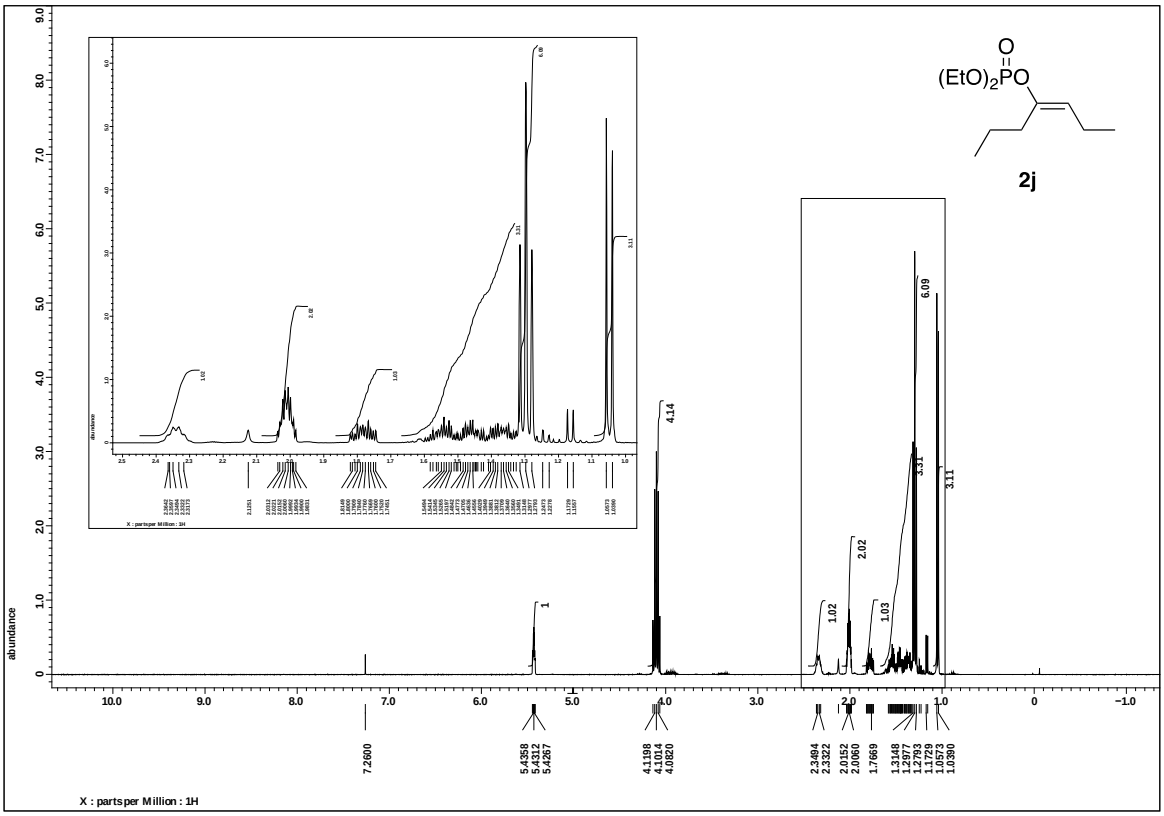

O
OP(OEt)2
2g
abundance
X : parts per Million : 13C

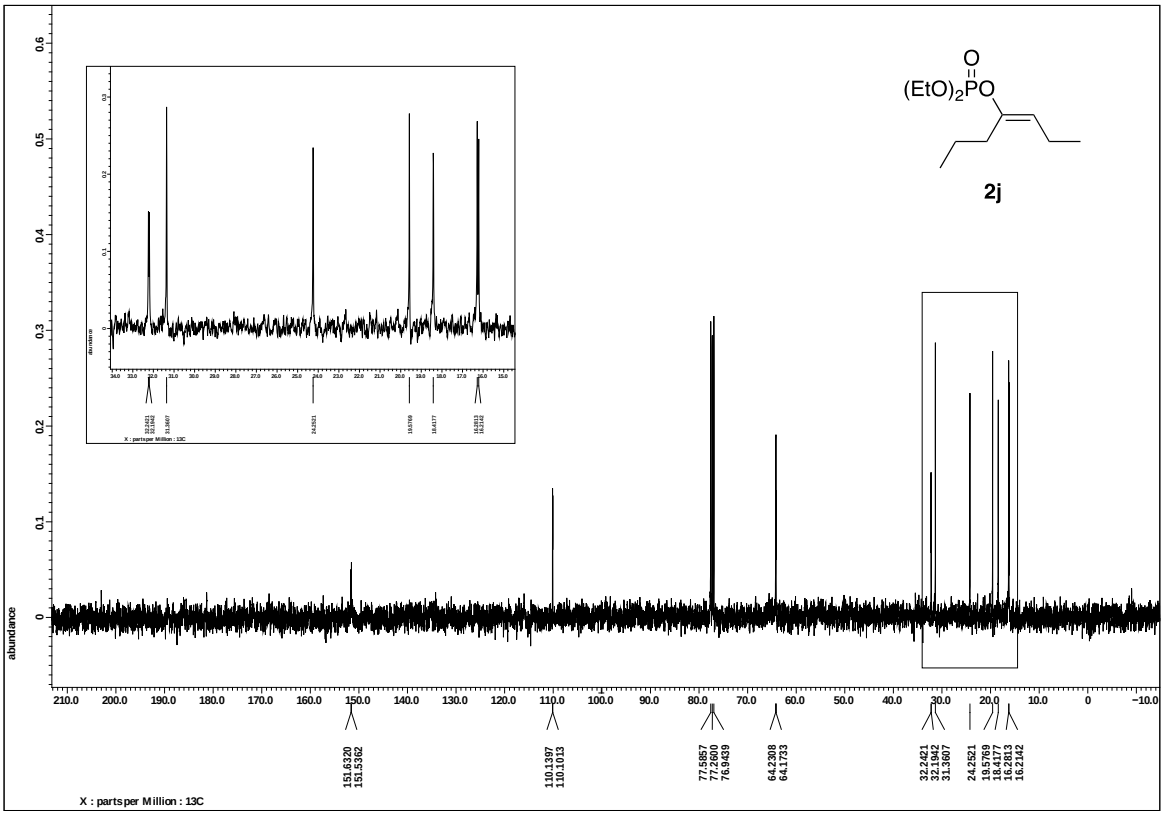

O
OP(OEt)2
2g
abundance
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
300.0
200.0
100.0
0
−100.0
−200.0
−300.0
−5.4370
X : parts per Million : 31P

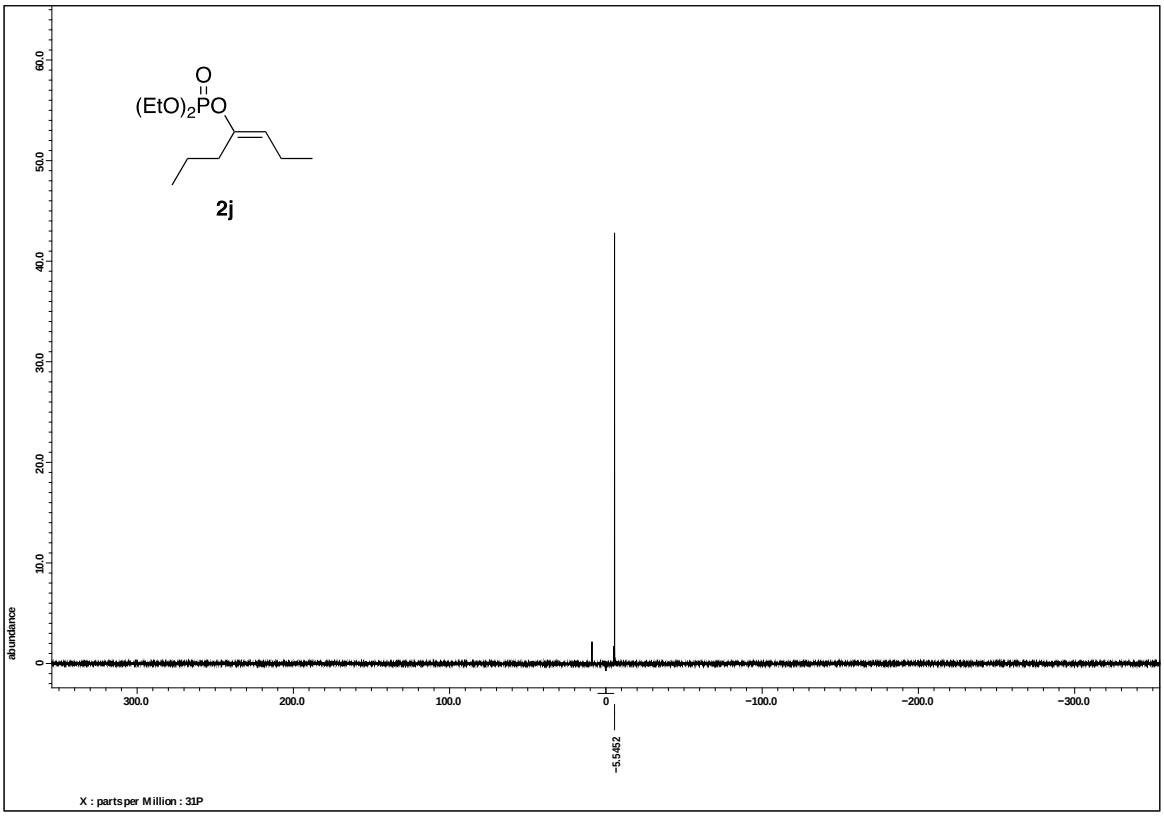

O
OP(OEt)2
Ph
2h
abundance
X : parts per Million : 1H

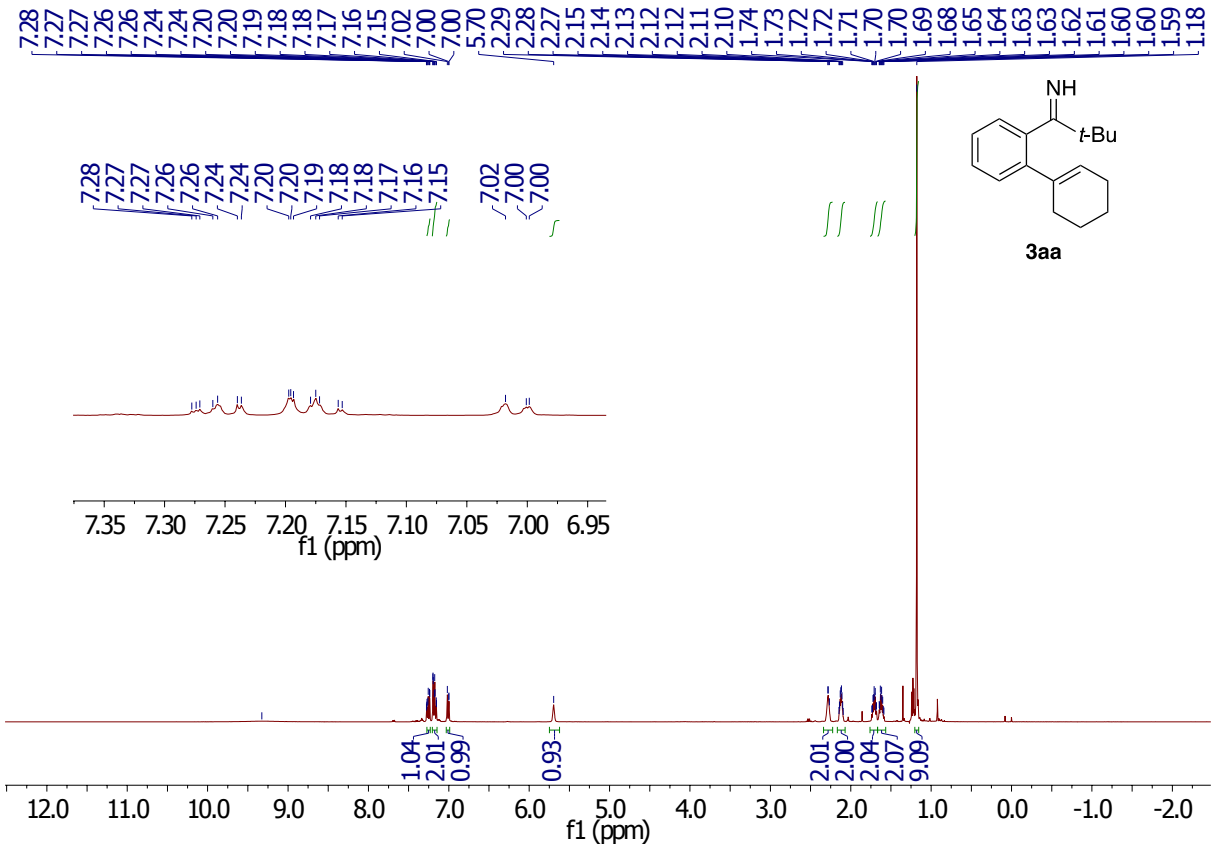

O
OP(OEt)2
Ph
2h
abundance
X : parts per Million : 13C

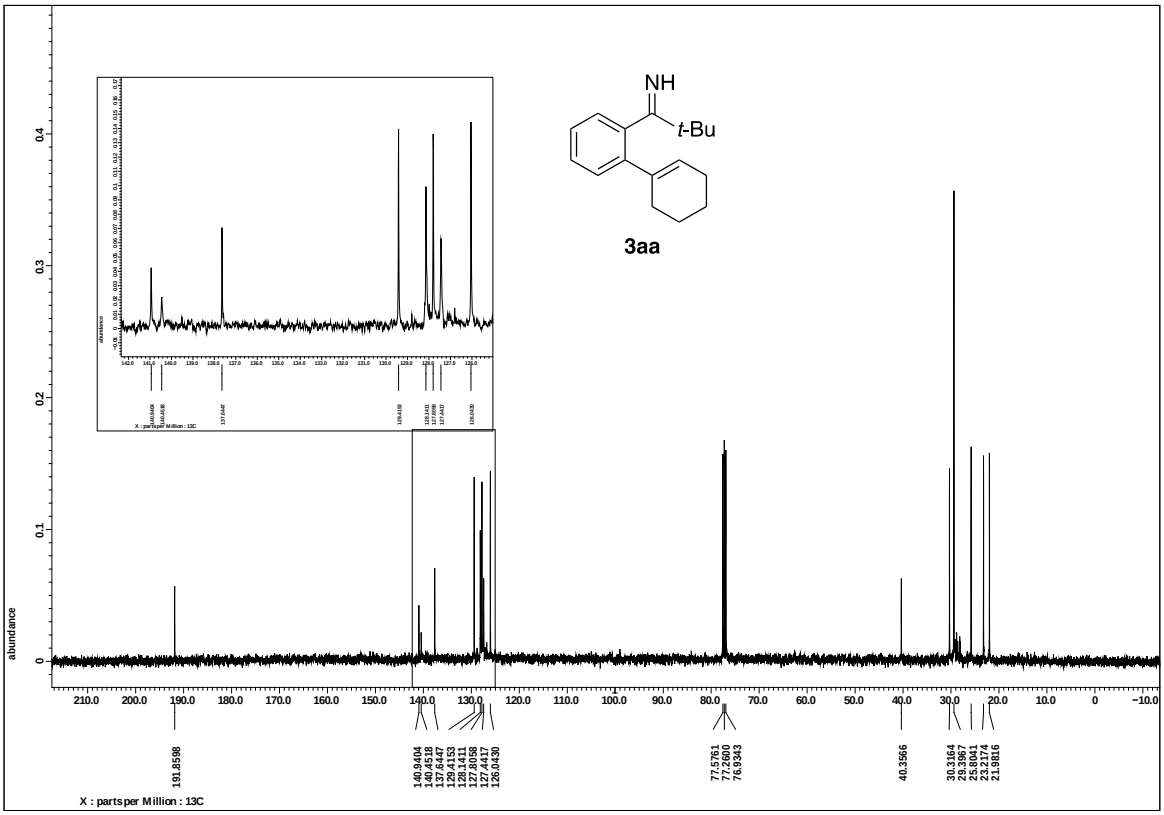

O
OP(OEt)2
Ph
2h
abundance
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
300.0
200.0
100.0
0
−100.0
−200.0
−300.0
−5.4911
X : parts per Million : 31P

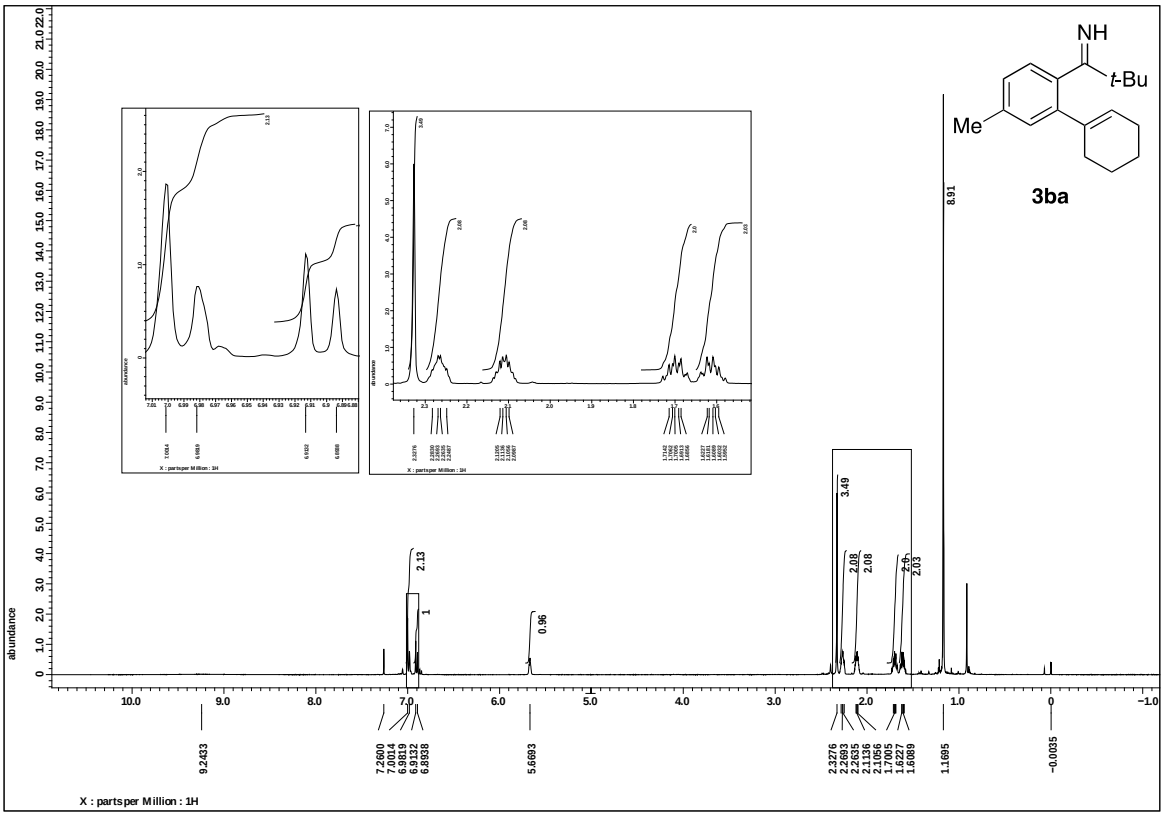

O
(EtO)2PO
2j
abundance
X : parts per Million : 1H
7.2600
5.4358
5.4312
5.4267
4.1198
4.1014
4.0820
2.3494
2.3322
2.0152
2.0060
1.7669
1.3148
1.3077
1.2793
1.1729
1.0573
1.0390

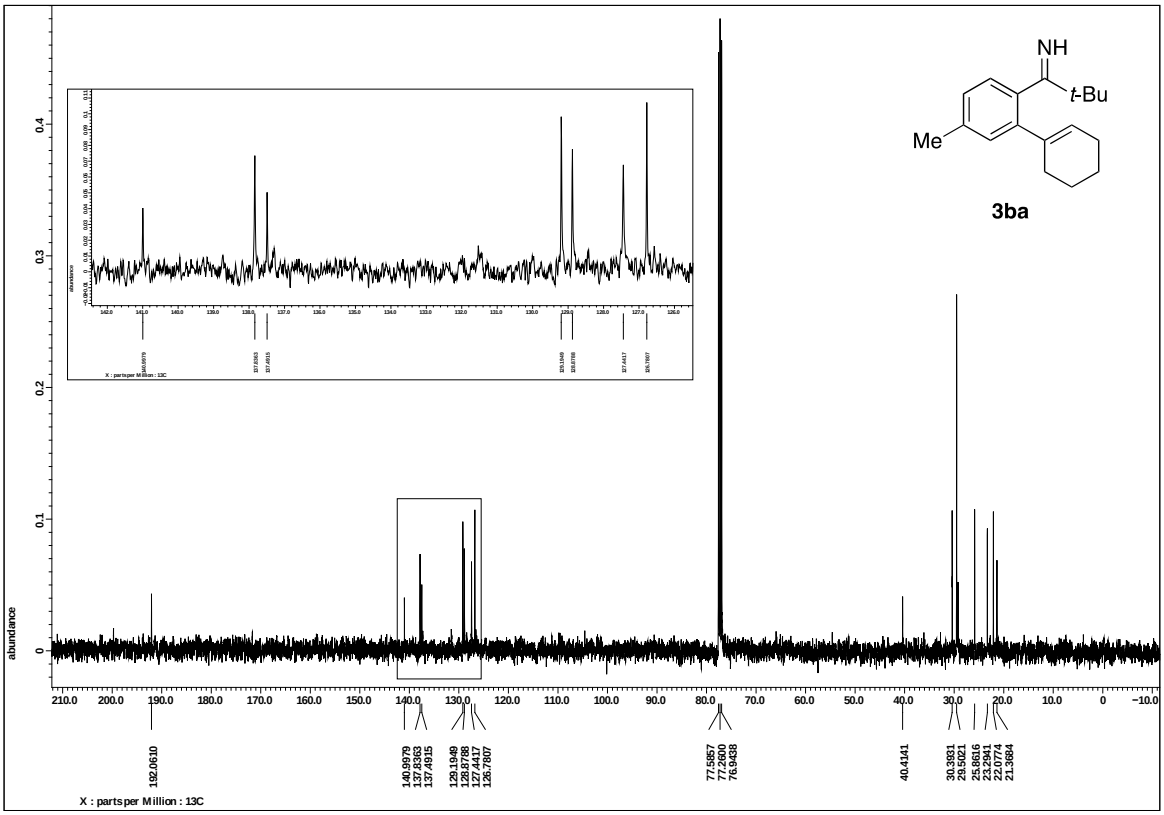

O
(EtO)2PO
2j
abundance
X : parts per Million : 13C
151.6320
151.5302
110.1397
110.1013
77.5857
77.2600
76.9439
64.2308
64.1733
32.2421
32.0942
31.9607
24.2521
19.5769
18.4177
16.2813
16.2142

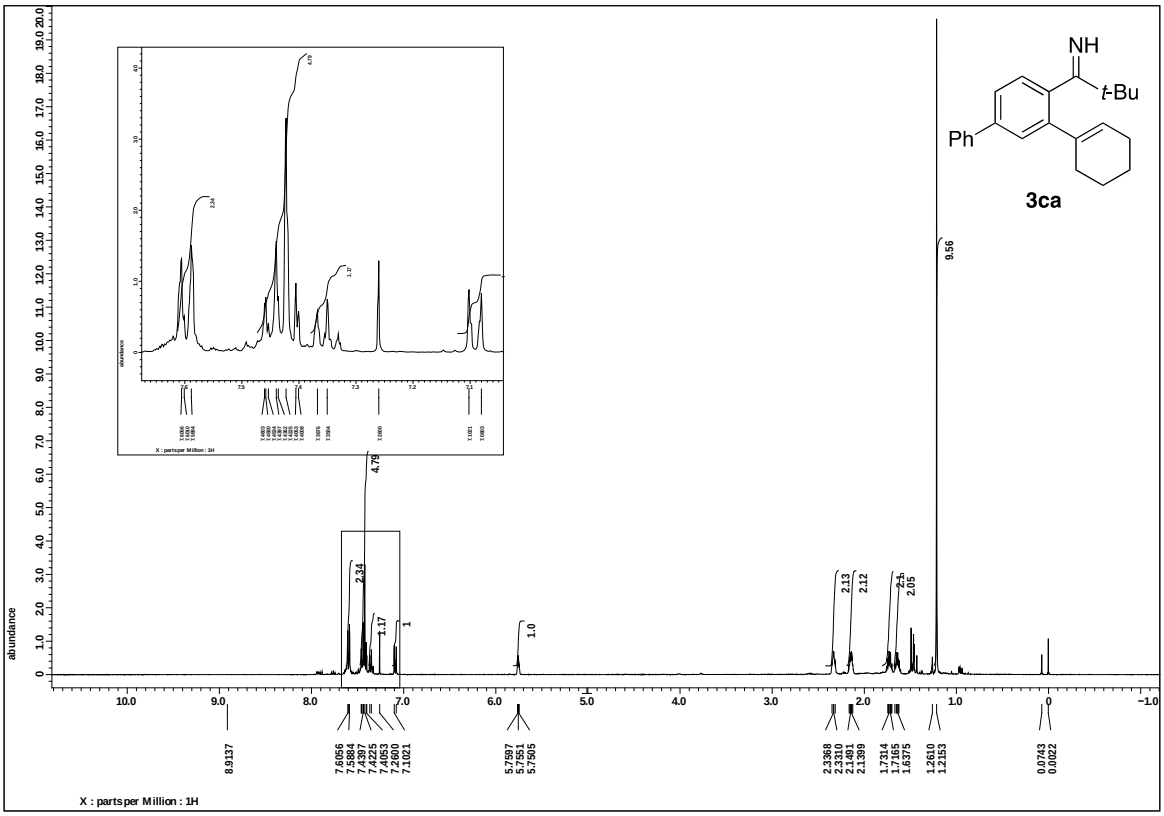

O
(EtO)2PO
2j
60.0
50.0
40.0
30.0
20.0
10.0
0
abundance
300.0
200.0
100.0
0
−100.0
−200.0
−300.0
−5.5462
X : parts per Million : 31P

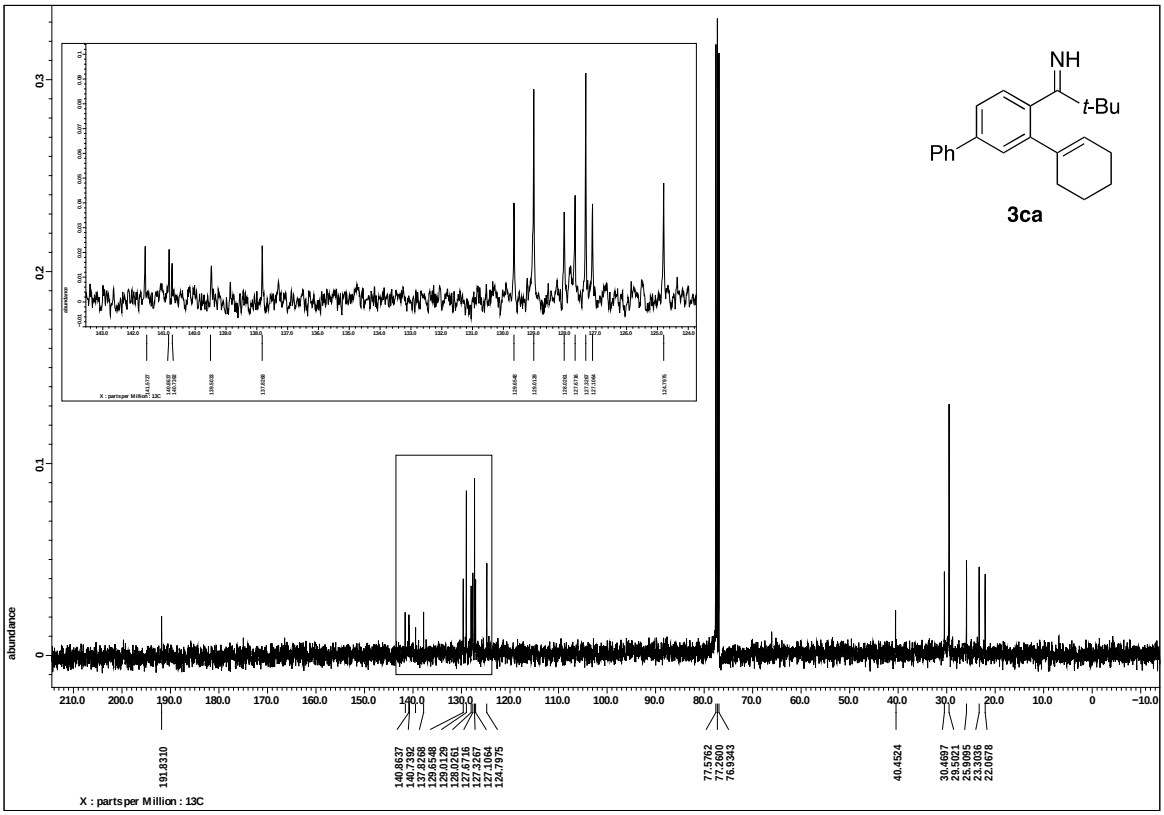

7.28 7.27 7.27 7.26 7.26 7.24 7.24 7.20 7.20 7.19 7.18 7.17 7.16 7.15 7.02 7.00 7.00 5.70 2.29 2.28 2.27 2.15 2.14 2.13 2.12 2.12 2.11 2.10 1.74 1.73 1.72 1.72 1.71 1.70 1.70 1.69 1.68 1.65 1.64 1.63 1.63 1.62 1.61 1.60 1.60 1.59 1.18
NH
t-Bu
3aa
7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95
f1 (ppm)
1.04 2.01 0.99 0.93 2.01 2.00 2.04 2.07 9.09
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0 -2.0
f1 (ppm)

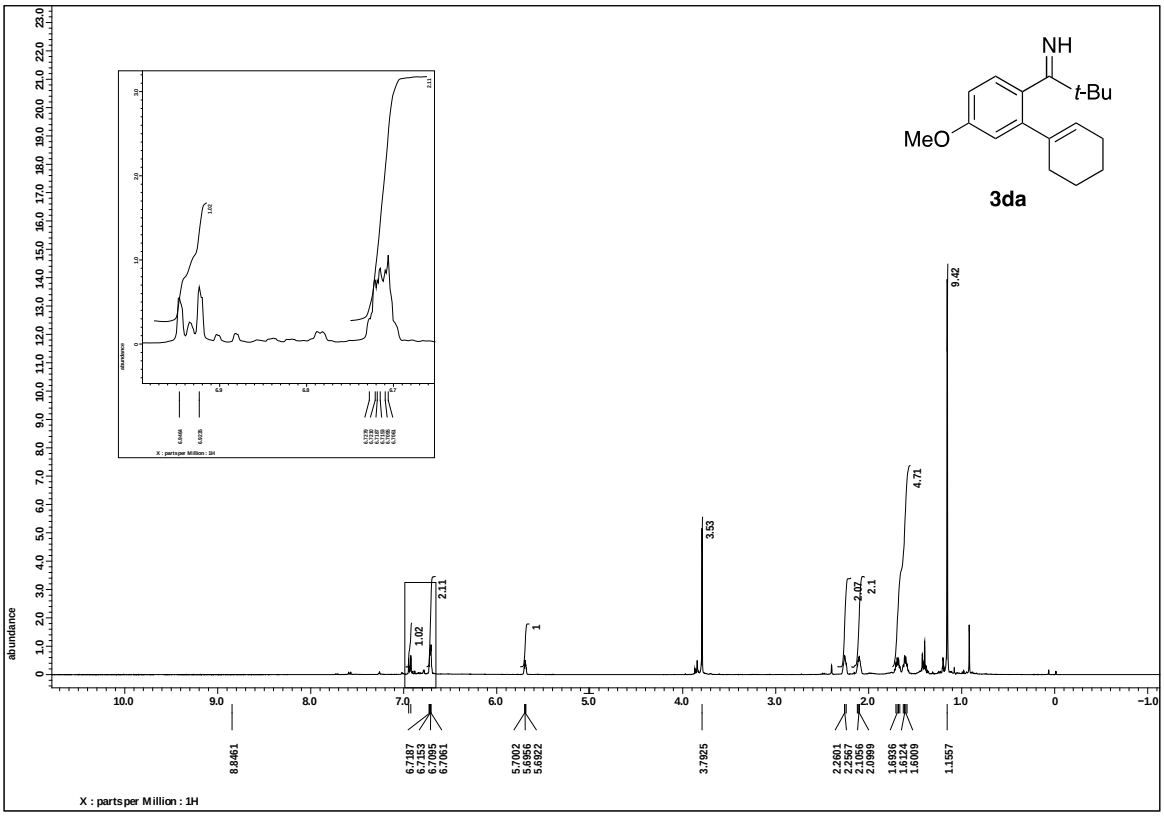

NH
t-Bu
3aa
abundance
191.8598
140.9404
140.4598
137.6447
129.4153
128.1411
127.8058
127.4417
126.0430
77.5761
77.2800
76.9343
40.3566
30.3164
29.3867
25.8041
23.2174
21.9816
X : parts per Million : 13C

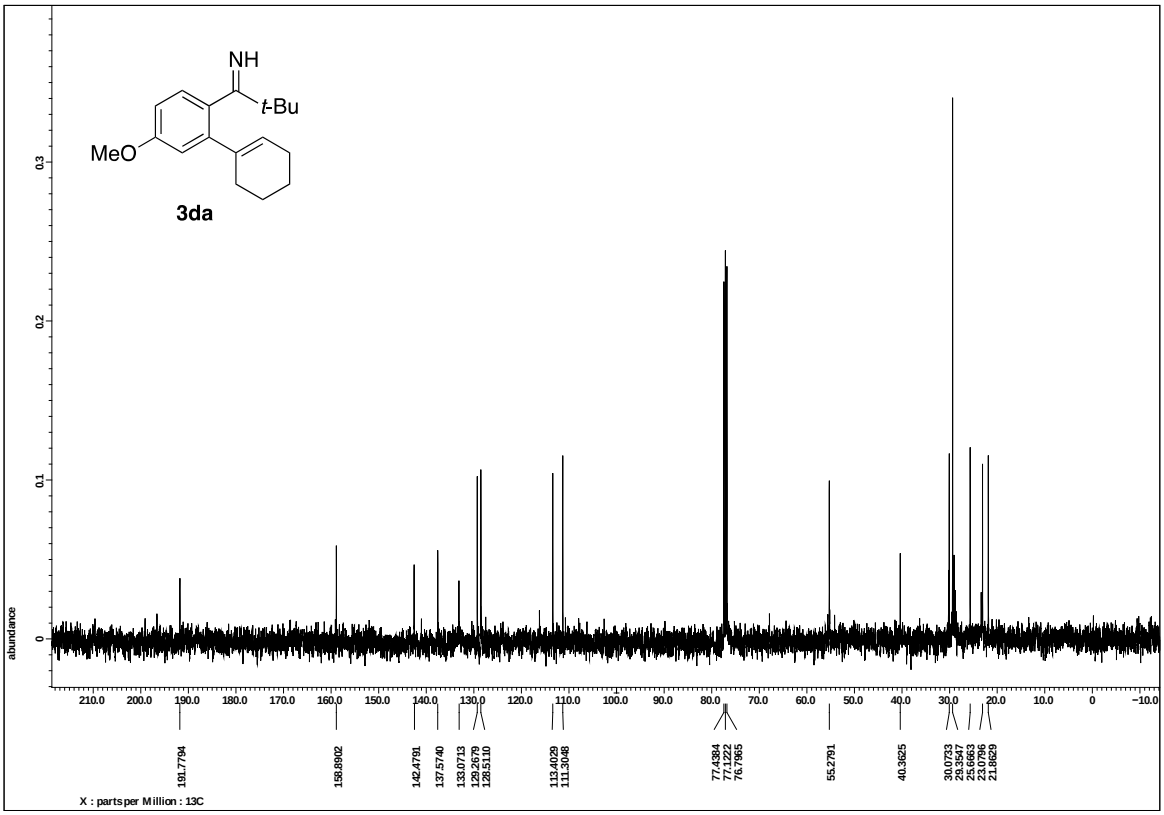

NH
t-Bu
Me
3ba
8.91
3.49
2.13
2.08
2.08
2.03
1
0.96
abundance
X : parts per Million : 1H

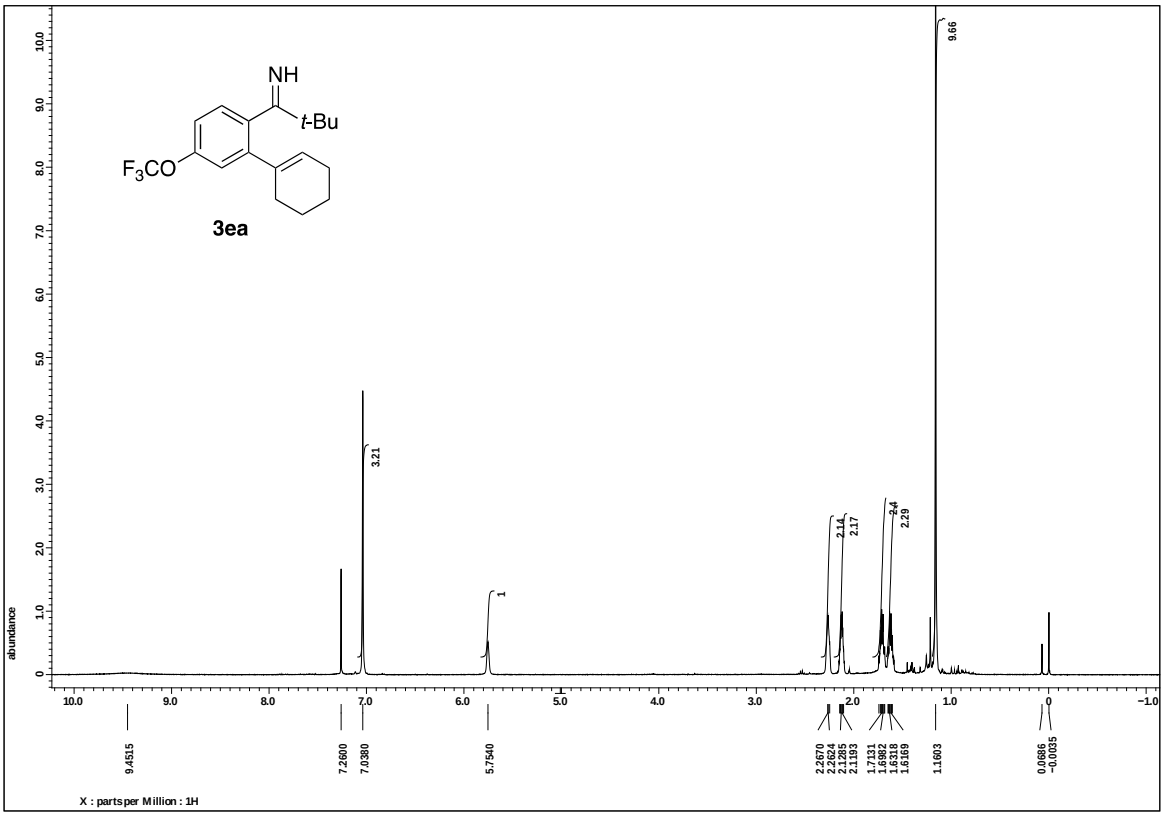

NH
t-Bu
Me
3ba
abundance
X : parts per Million : 13C

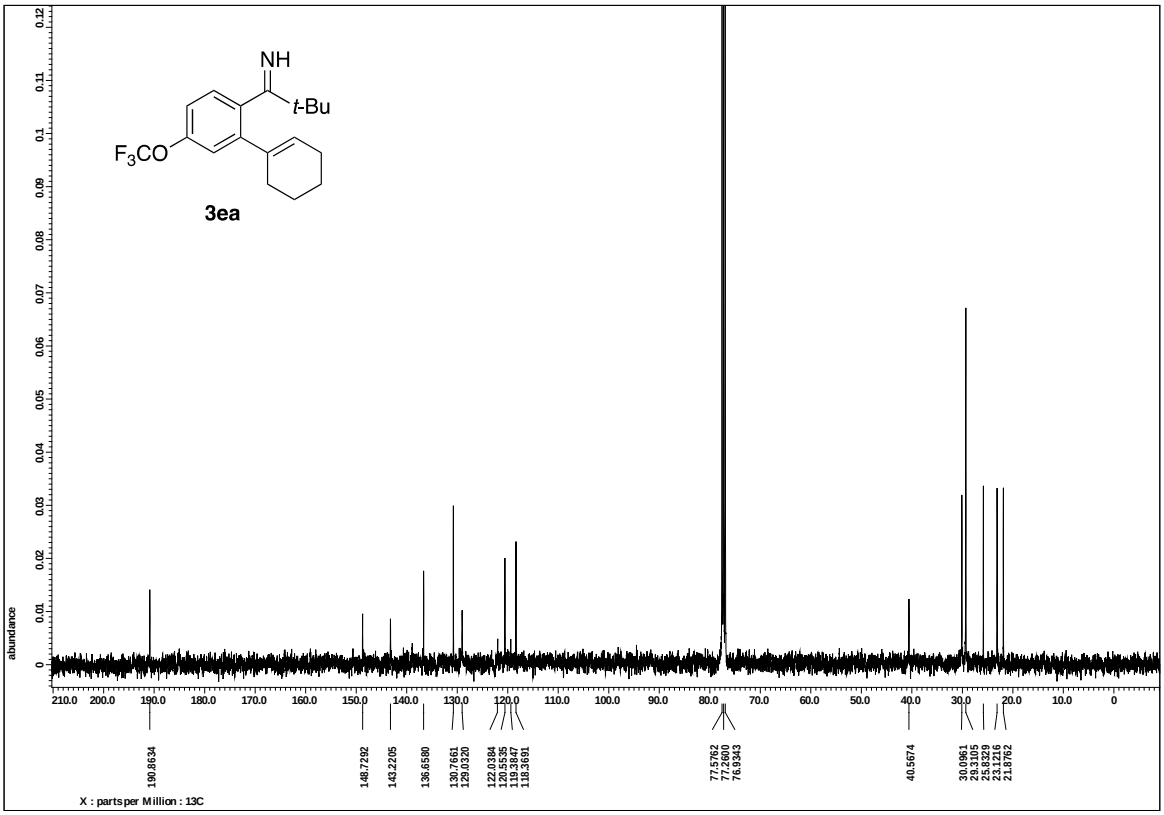

NH
t-Bu
Ph
3ca
abundance
X : parts per Million : 1H

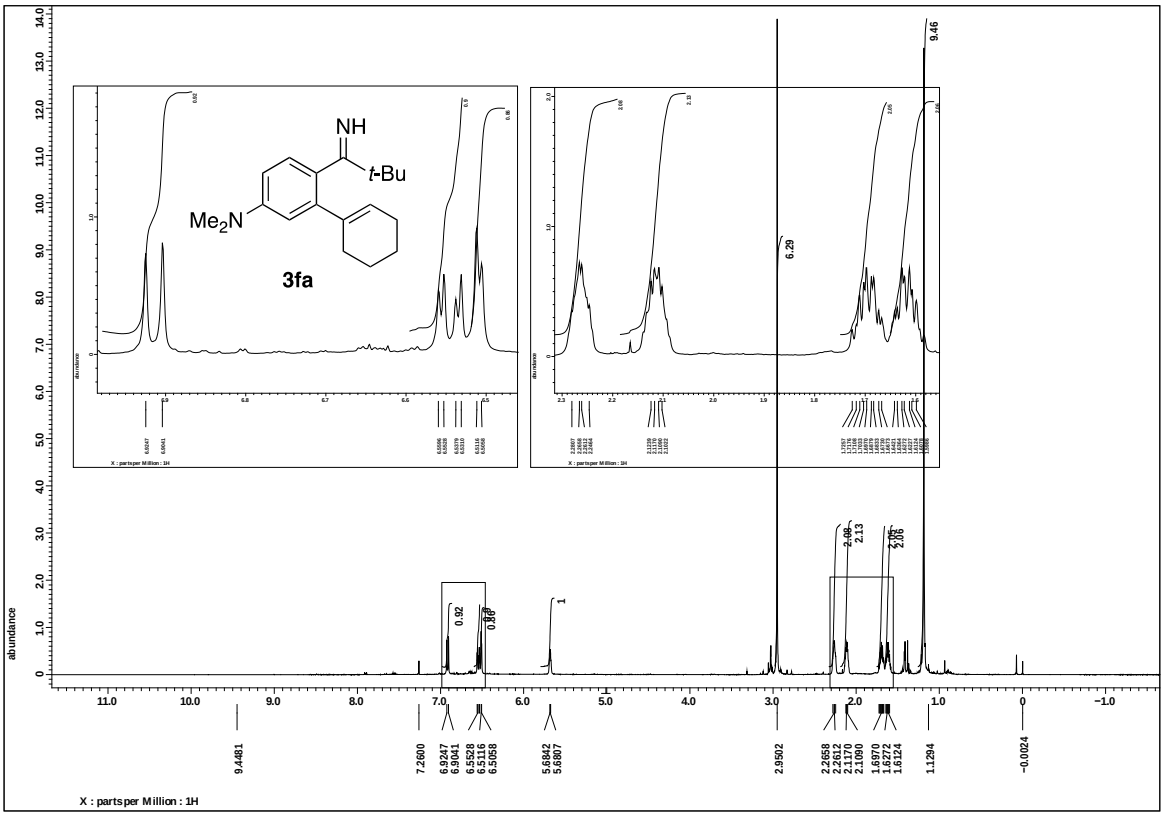

NH
t-Bu
Ph
3ca
abundance
X : parts per Million : 13C

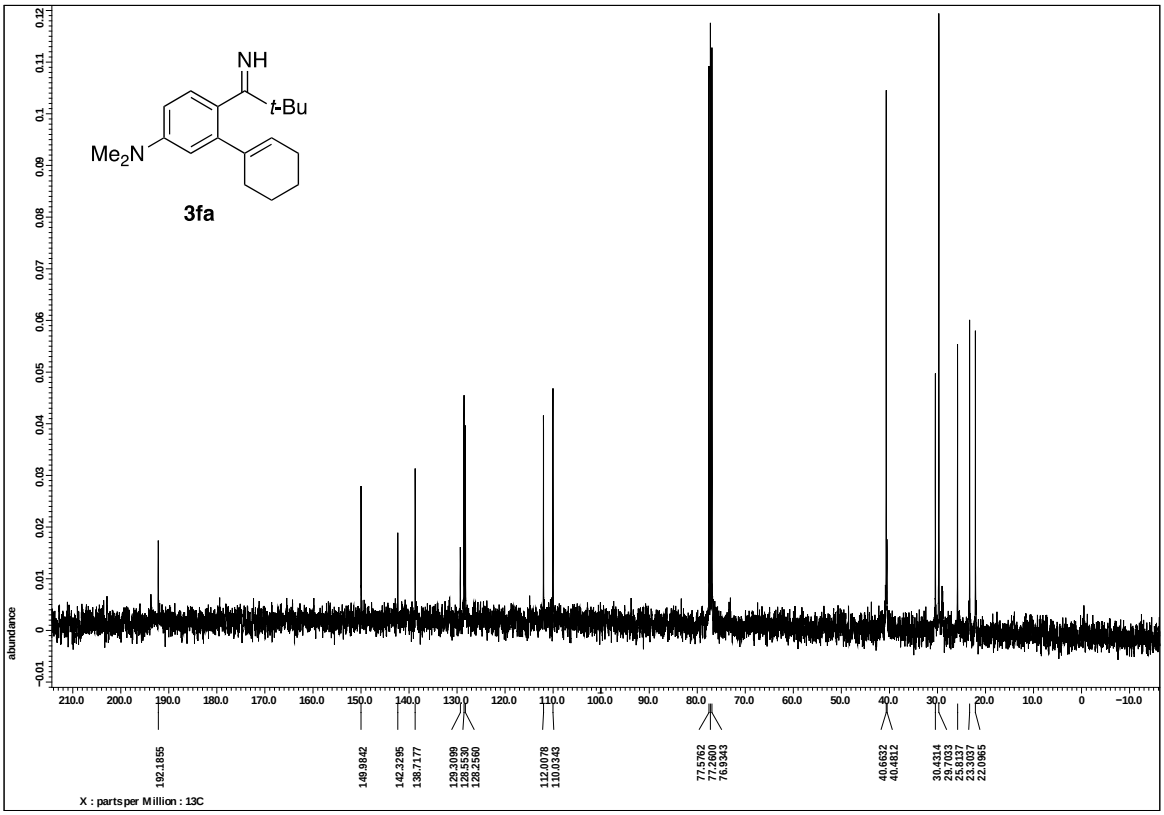

NH
t-Bu
MeO
3da
abundance
9.42
4.71
3.53
2.11
2.07
2.1
1.02
1
X : parts per Million : 1H

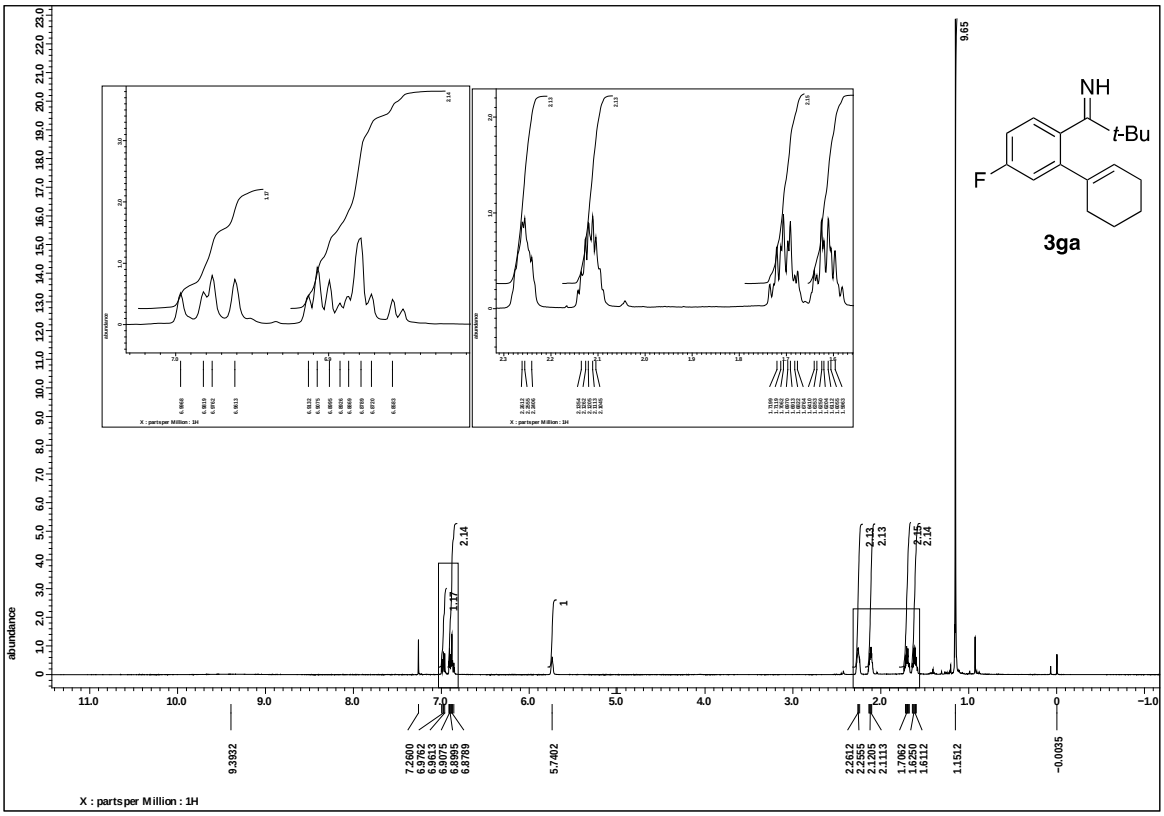

NH
t-Bu
MeO
3da
abundance
X : parts per Million : 13C

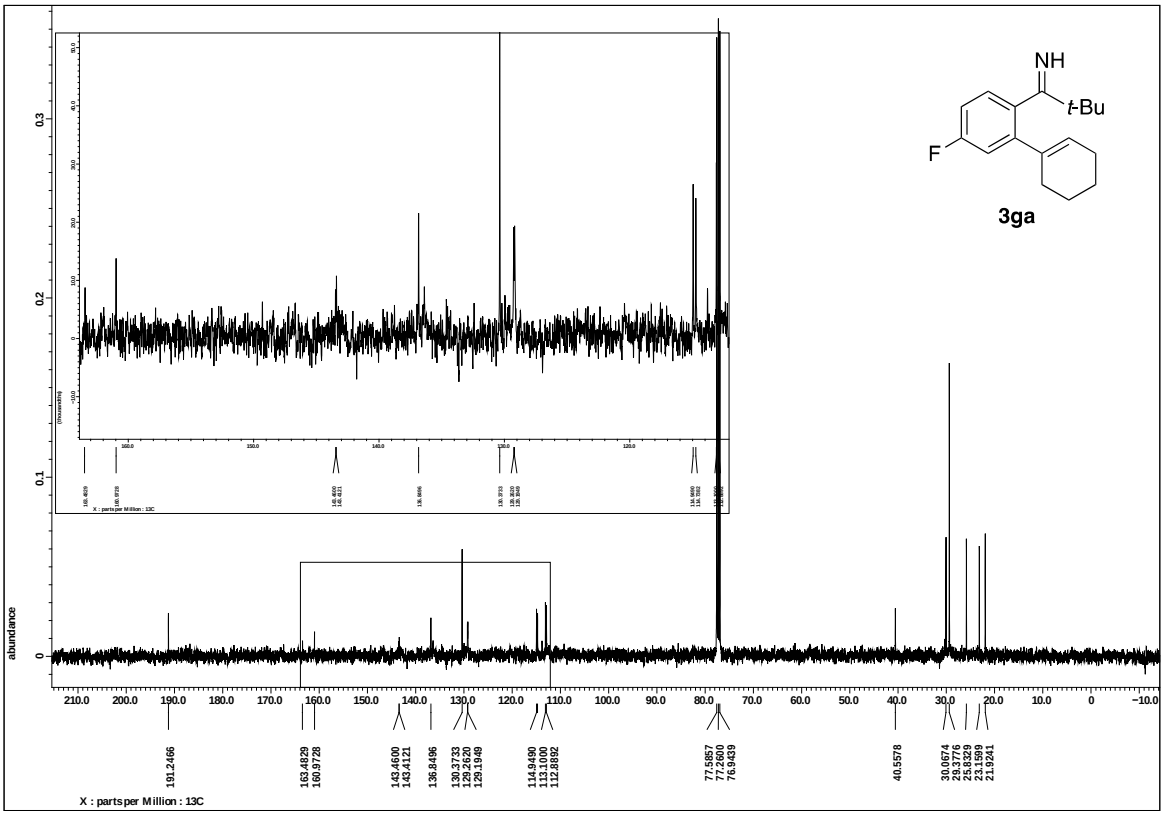

NH
t-Bu
F3CO
3ea
abundance
9.66
3.21
1
2.14
2.17
2.44
2.29
9.4515
7.2600
7.0380
5.7540
2.2670
2.2624
2.1285
2.1193
1.7131
1.6982
1.6318
1.6169
1.1603
0.0686
-0.0035
X : parts per Million : 1H

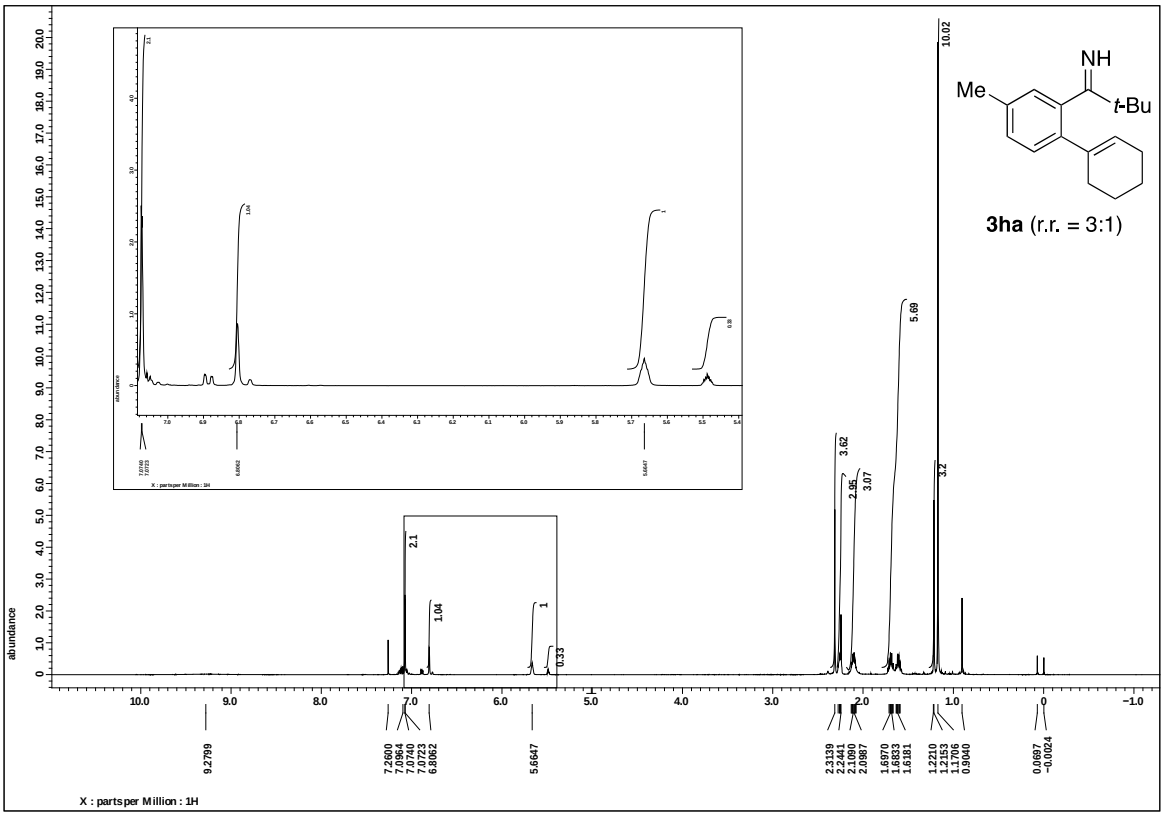

NH
t-Bu
F3CO
3ea
abundance
190.8634
148.7292
143.2205
136.6580
130.7661
129.0320
122.0384
120.5535
119.3827
118.3691
77.5762
77.2600
76.9343
40.5674
30.0961
29.3105
25.8329
23.1216
21.8762
X : parts per Million : 13C

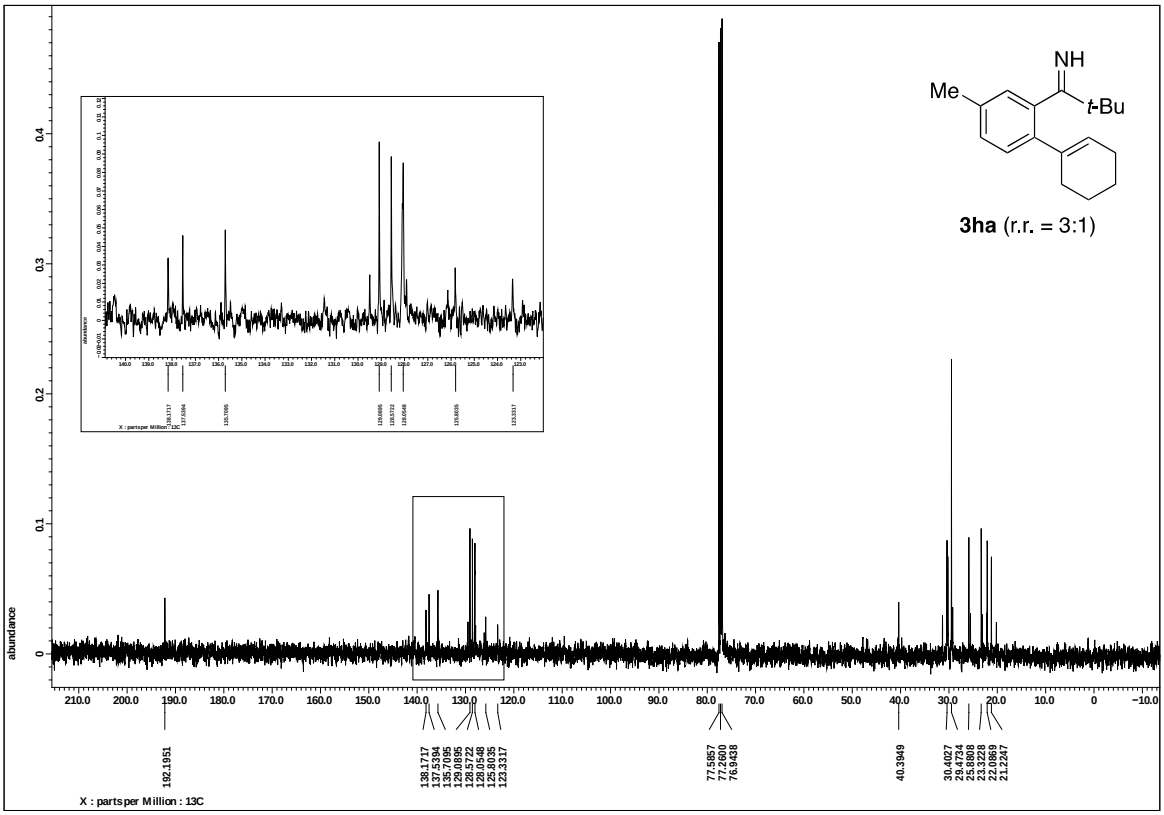

NH
t-Bu
Me2N
3fa
9.46
6.29
0.92
1
2.13
X : parts per Million : 1H

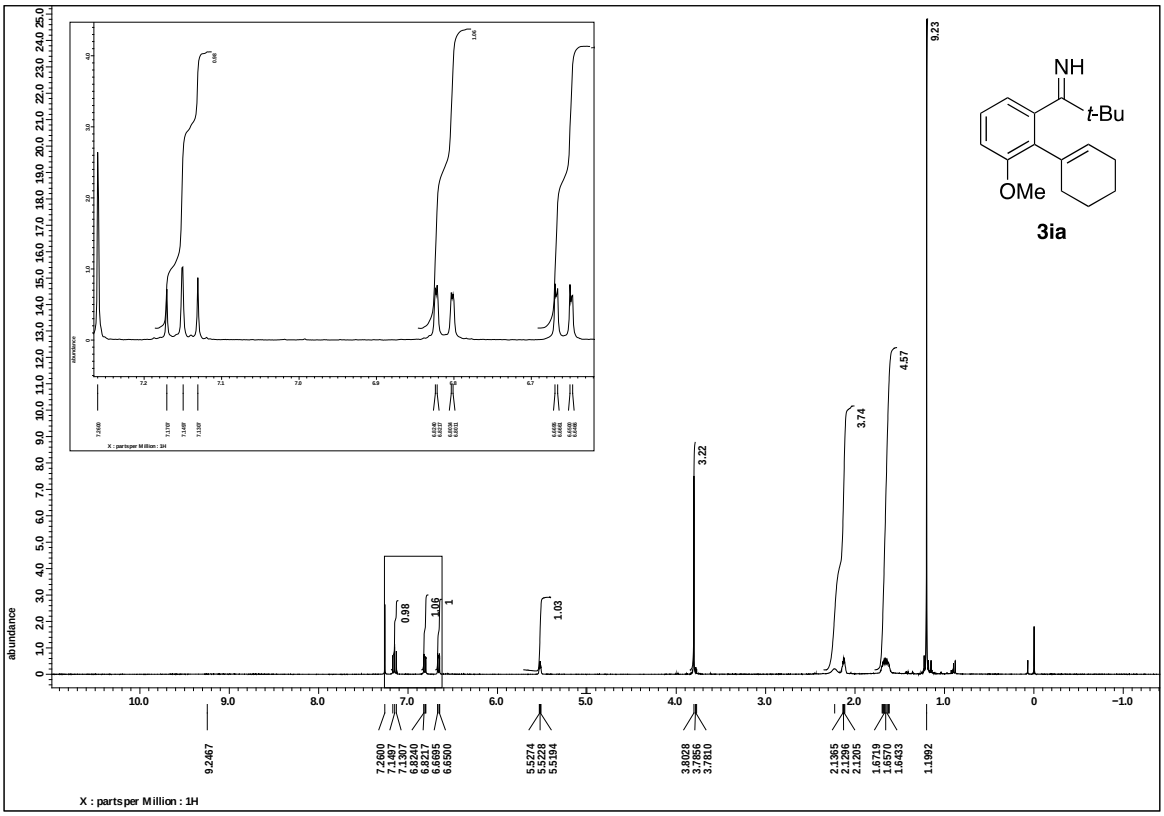

NH
t-Bu
Me2N
3fa
X : parts per Million : 13C

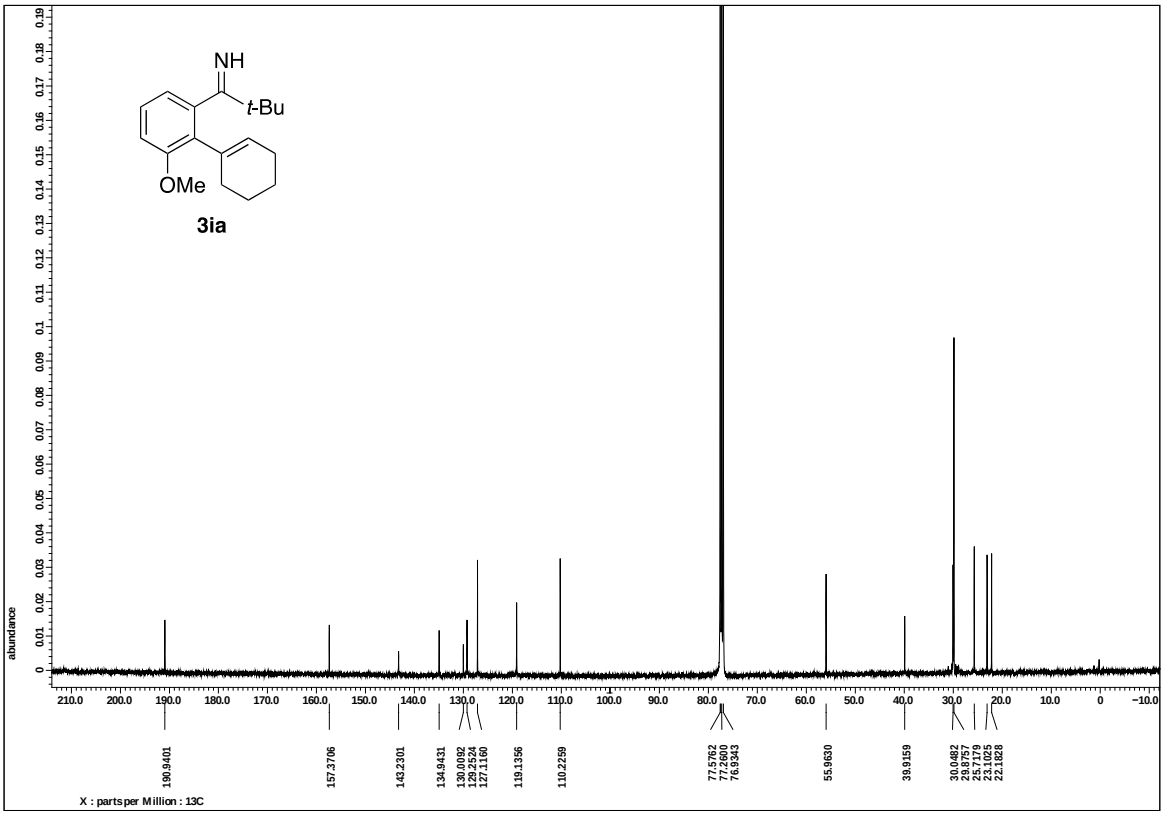

NH
t-Bu
F
3ga
abundance
X : parts per Million : 1H

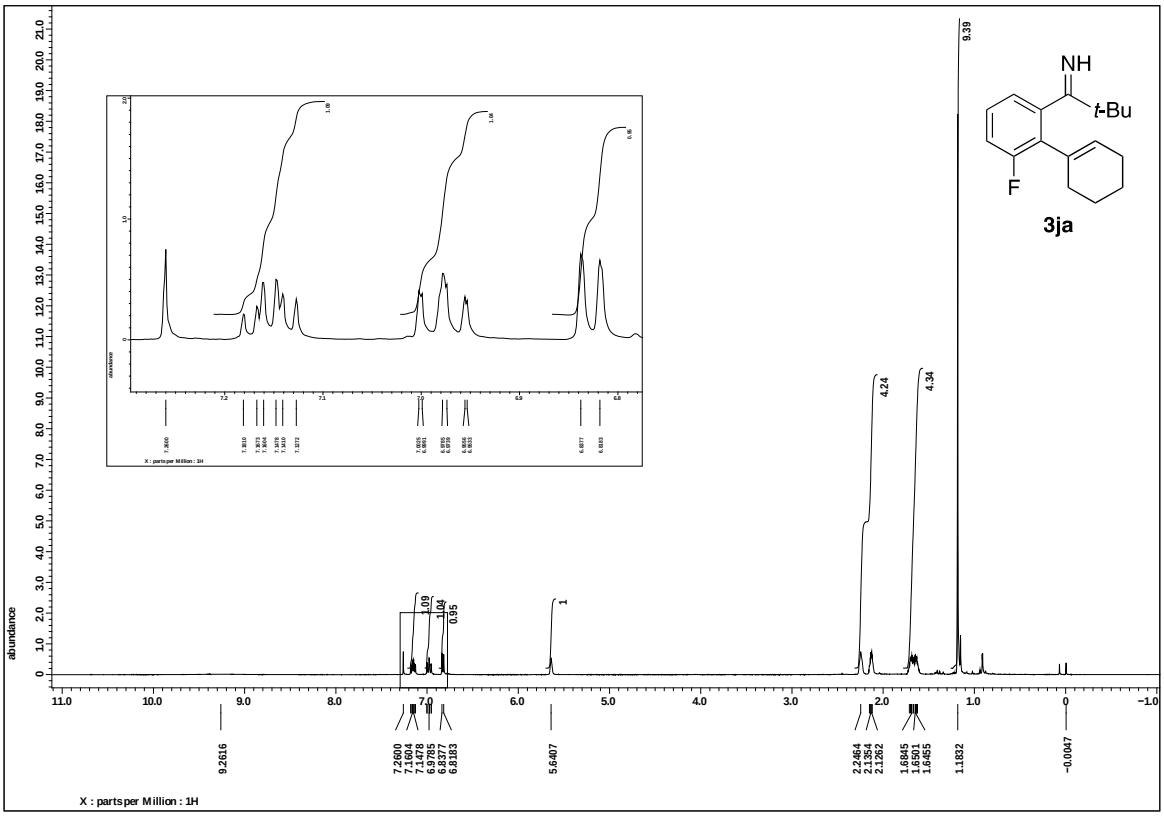

NH
t-Bu
F
3ga
abundance
X : parts per Million : 13C

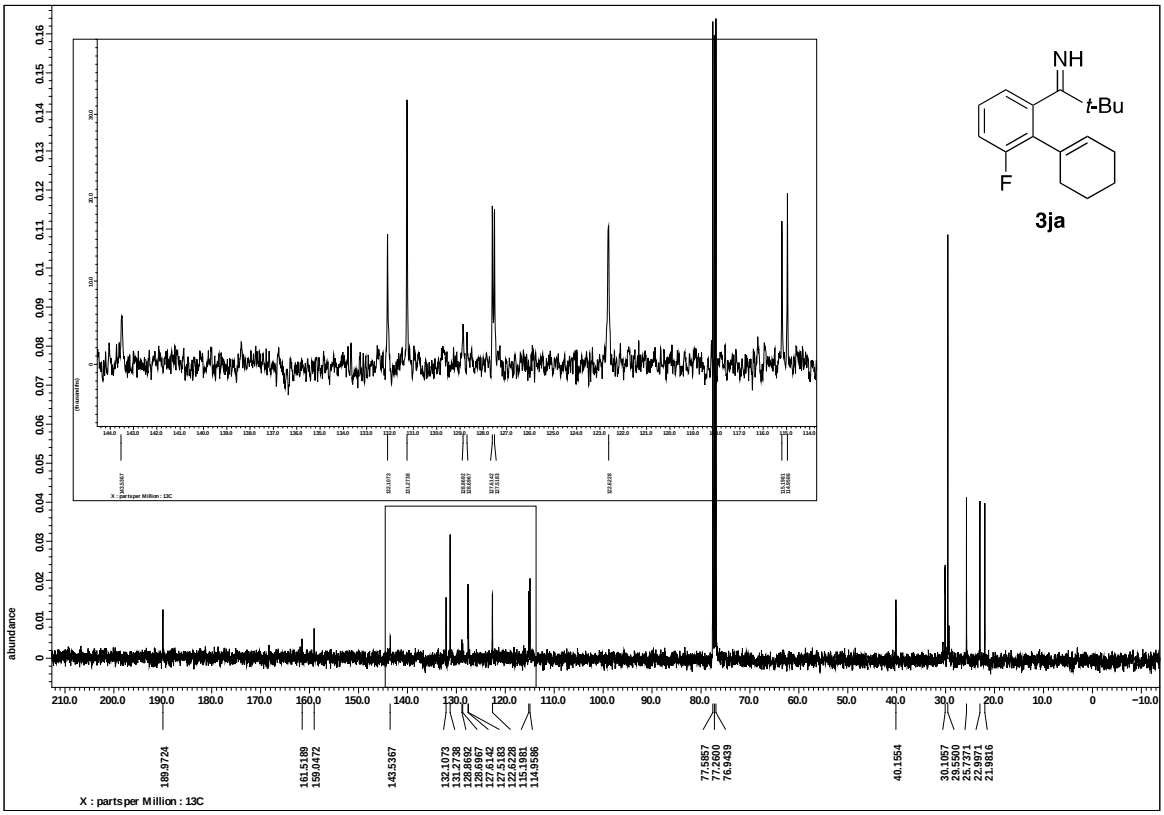

NH
Me
t-Bu
3ha (r.r. = 3:1)
X : parts per Million : 1H

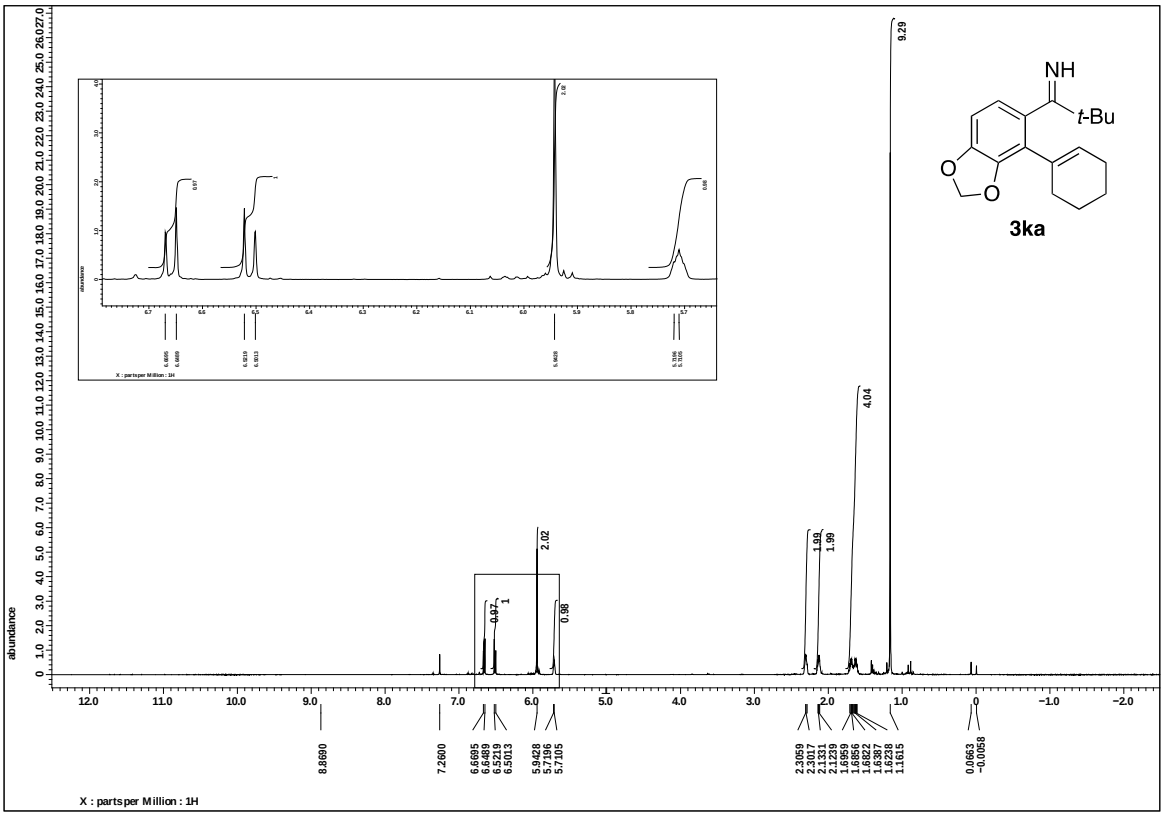

NH
Me
t-Bu
3ha (r.r. = 3:1)
X : parts per Million : 13C

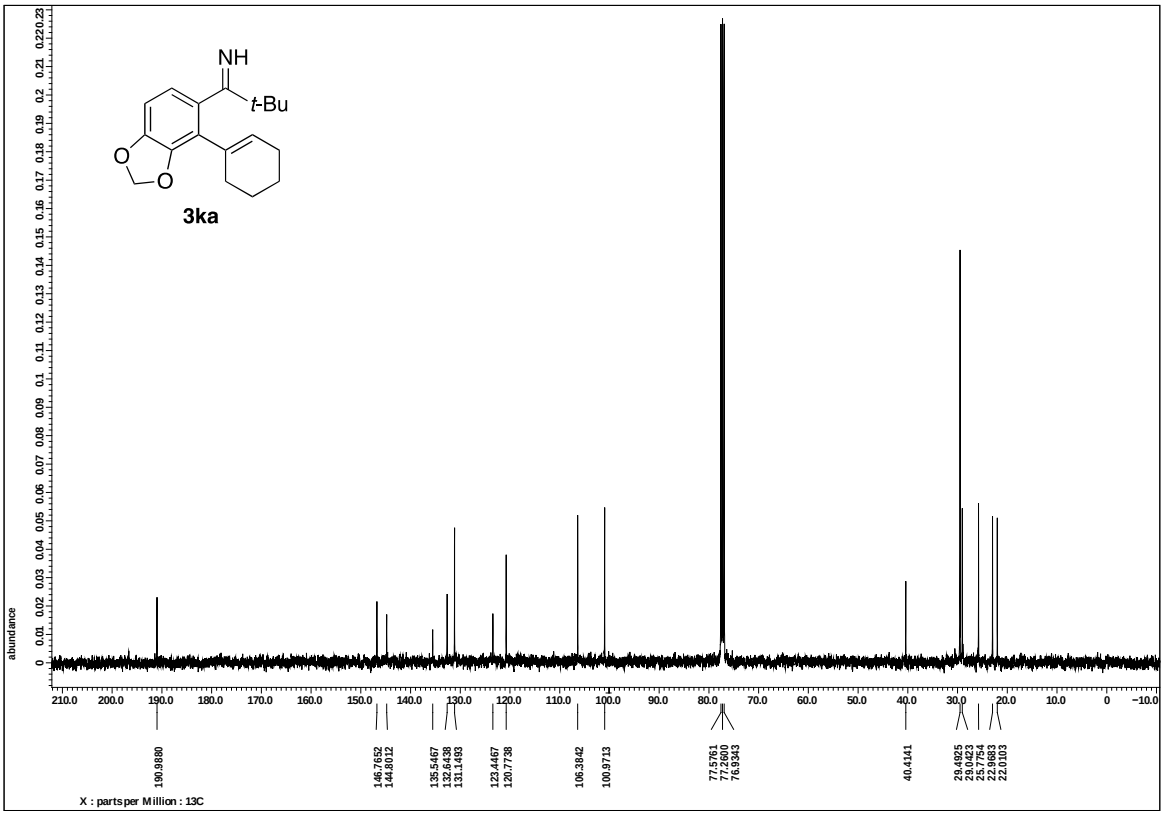

NH
t-Bu
OMe
3ia
9.23
4.57
3.74
3.22
1.03
0.98
1.06
1
abundance
X : parts per Million : 1H

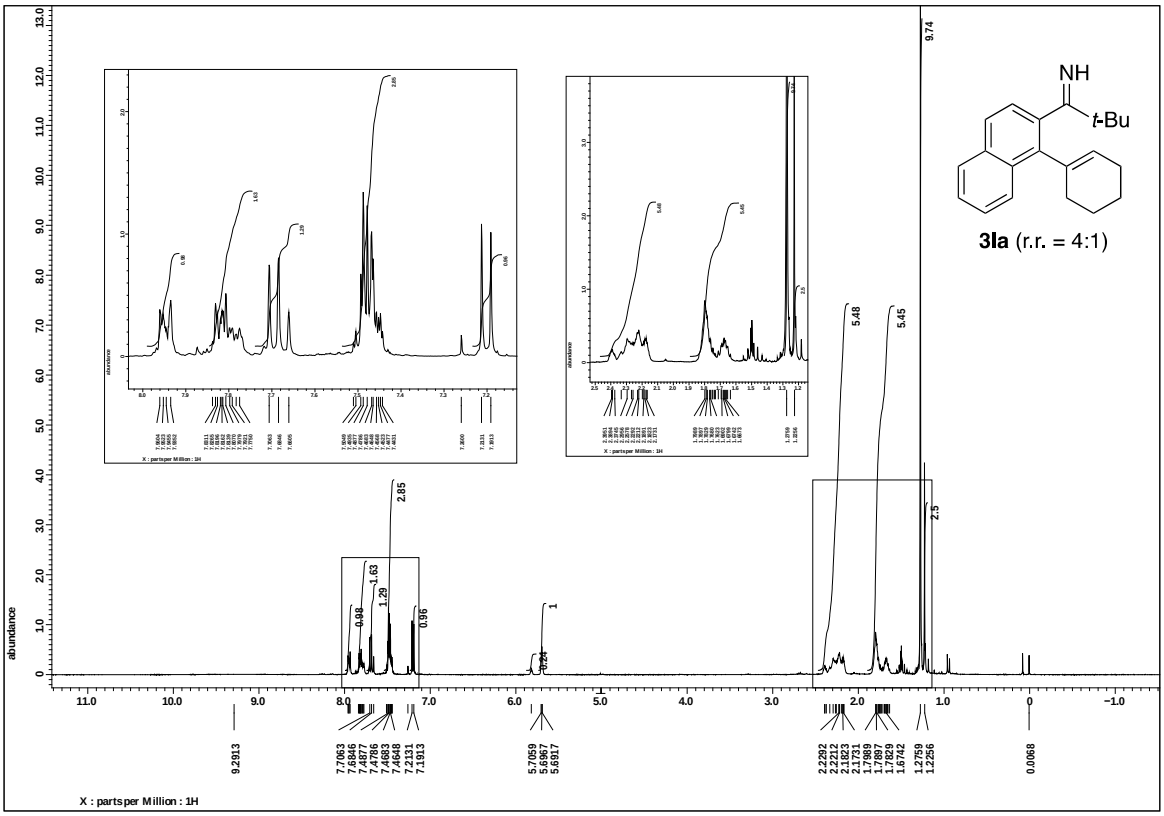

NH
t-Bu
OMe
3ia
abundance
X : parts per Million : 13C

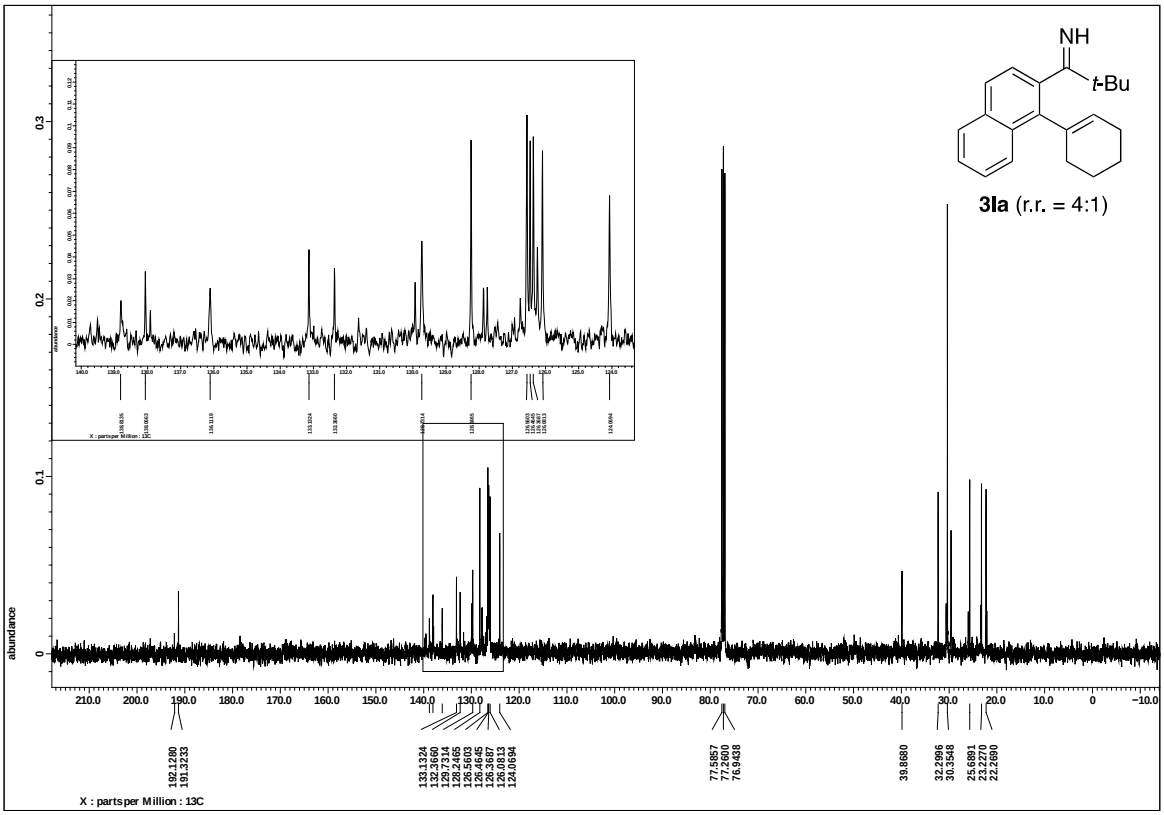

NH
t-Bu
F
3ja
9.39
4.24
4.34
1.09
1.04
0.95
1
abundance
X : parts per Million : 1H

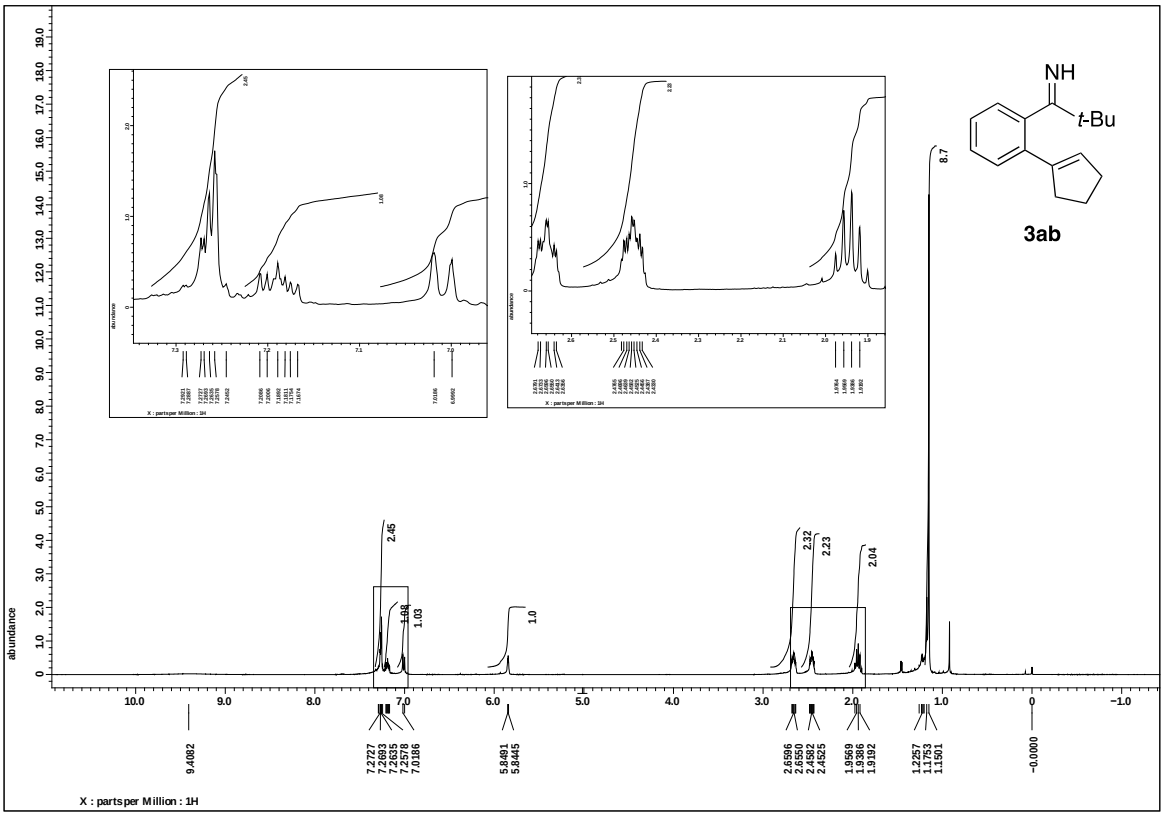

NH
t-Bu
F
3ja
abundance
189.9724
161.5199
159.0472
143.5367
132.1073
131.2738
128.8692
128.6967
127.6142
127.5128
122.4528
115.1981
114.9586
77.5857
77.2600
76.9439
40.1554
30.1057
29.5500
25.7371
22.9971
21.9816
X : parts per Million : 13C

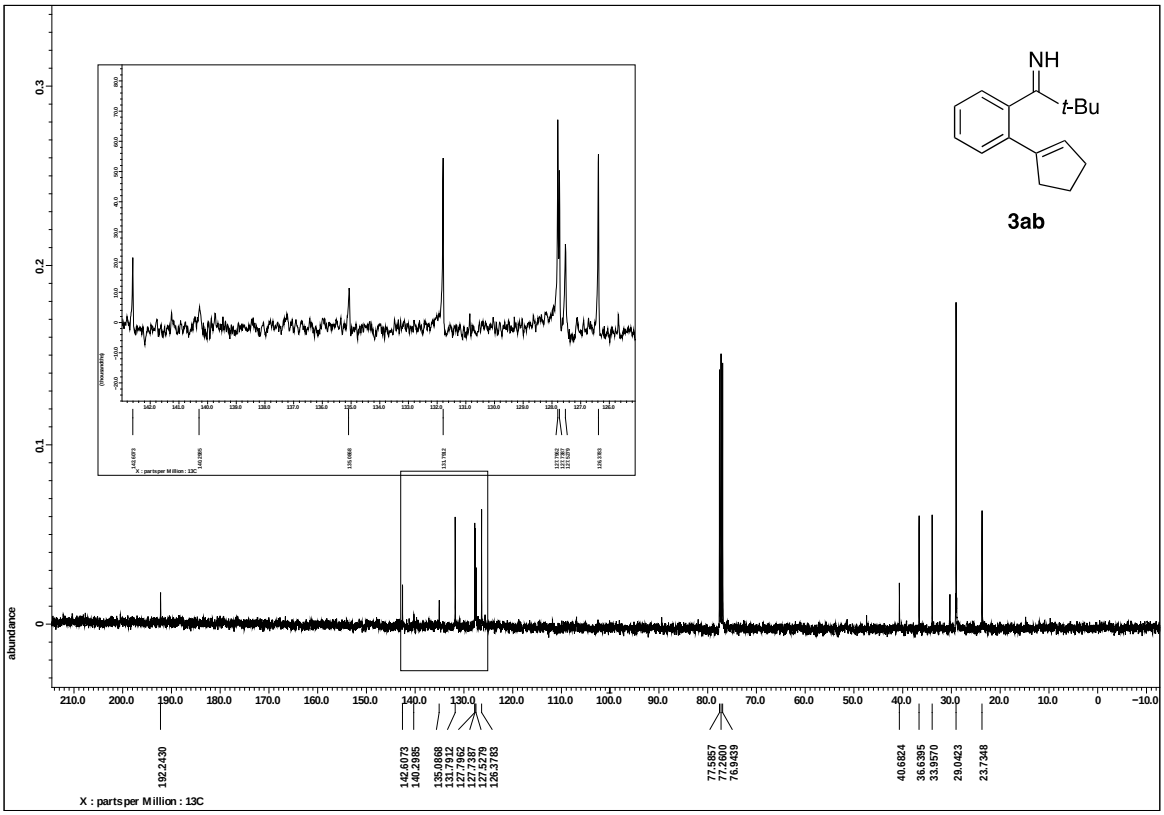

NH
t-Bu
O
O
3ka
abundance
X : parts per Million : 1H

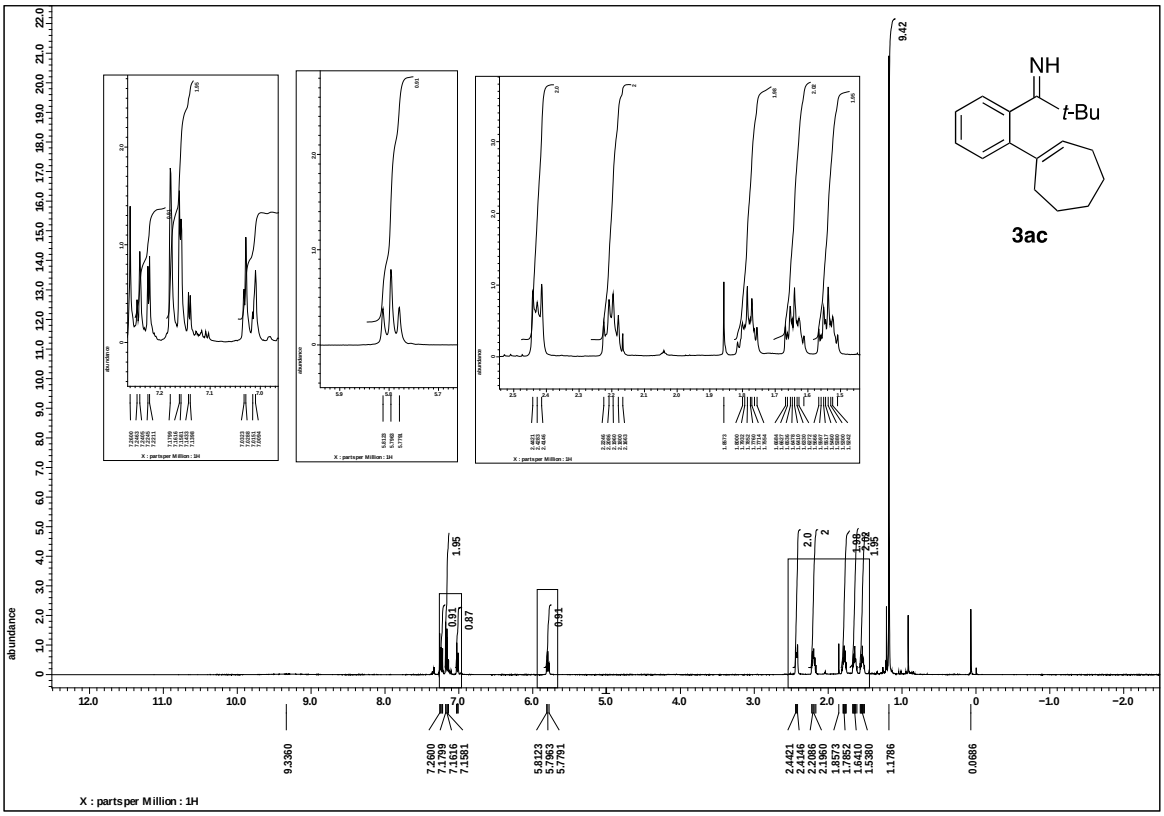

NH
t-Bu
O
O
3ka
abundance
X : parts per Million : 13C

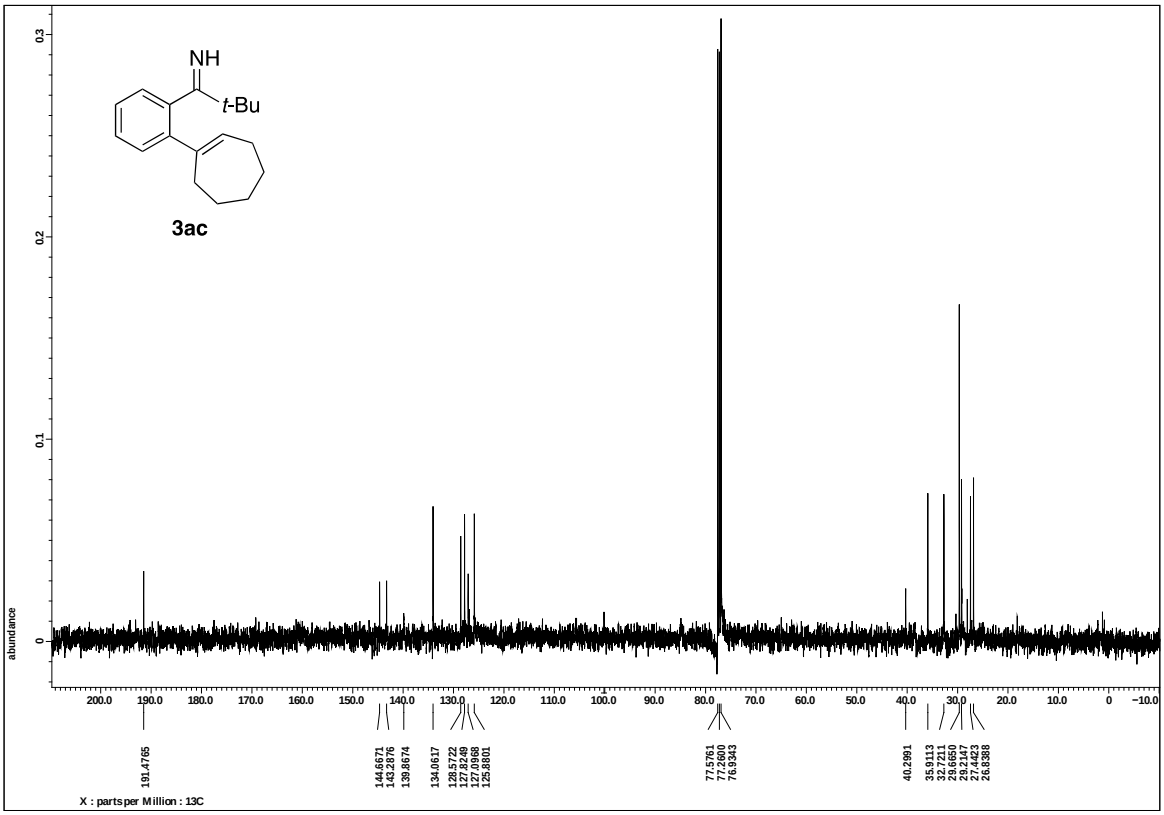

NH
t-Bu
3la (r.r. = 4:1)
abundance
X : parts per Million : 1H

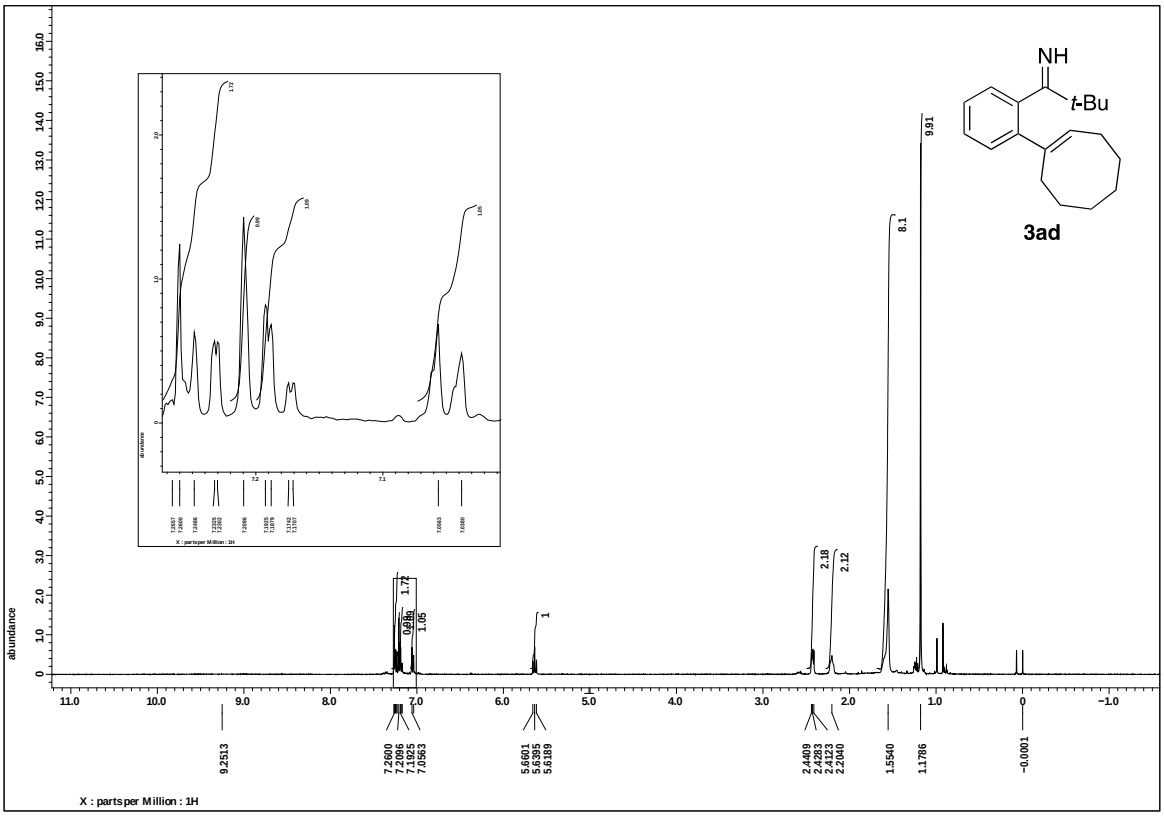

NH
t-Bu
3la (r.r. = 4:1)
abundance
X : parts per Million : 13C

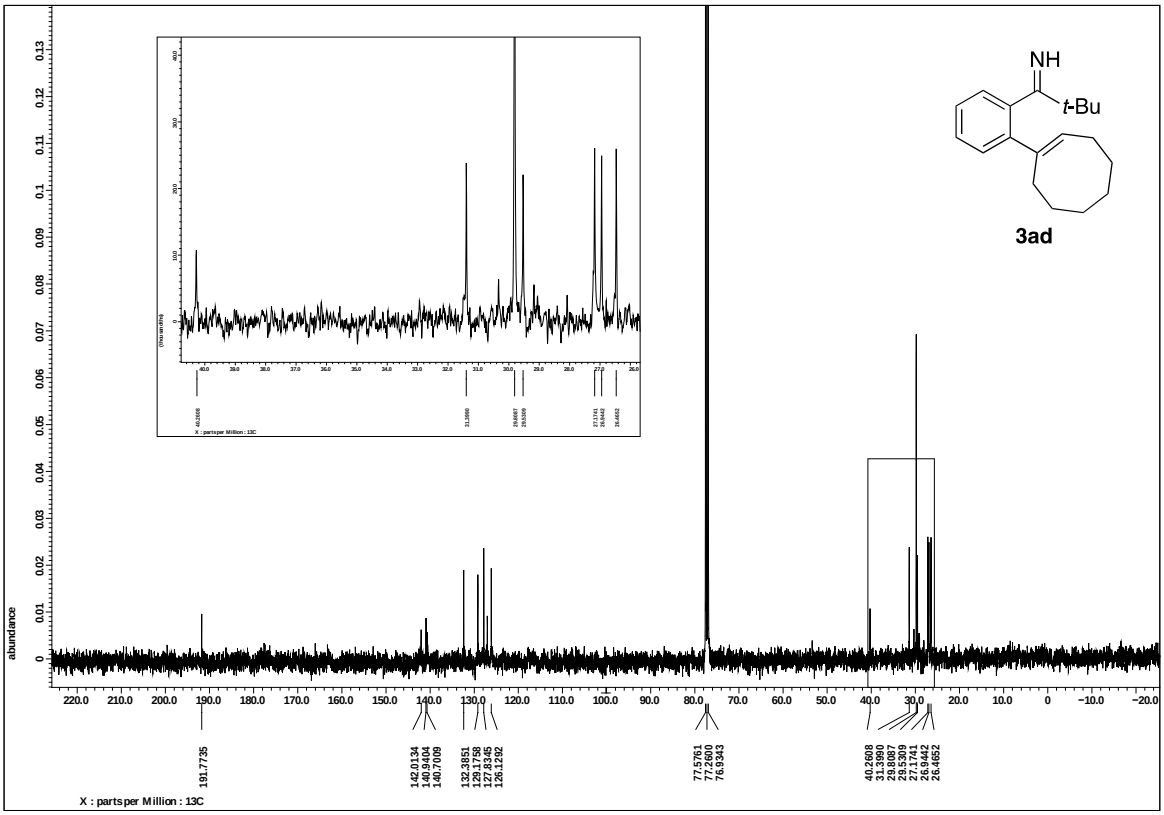

NH
t-Bu
3ab
abundance
X : parts per Million : 1H

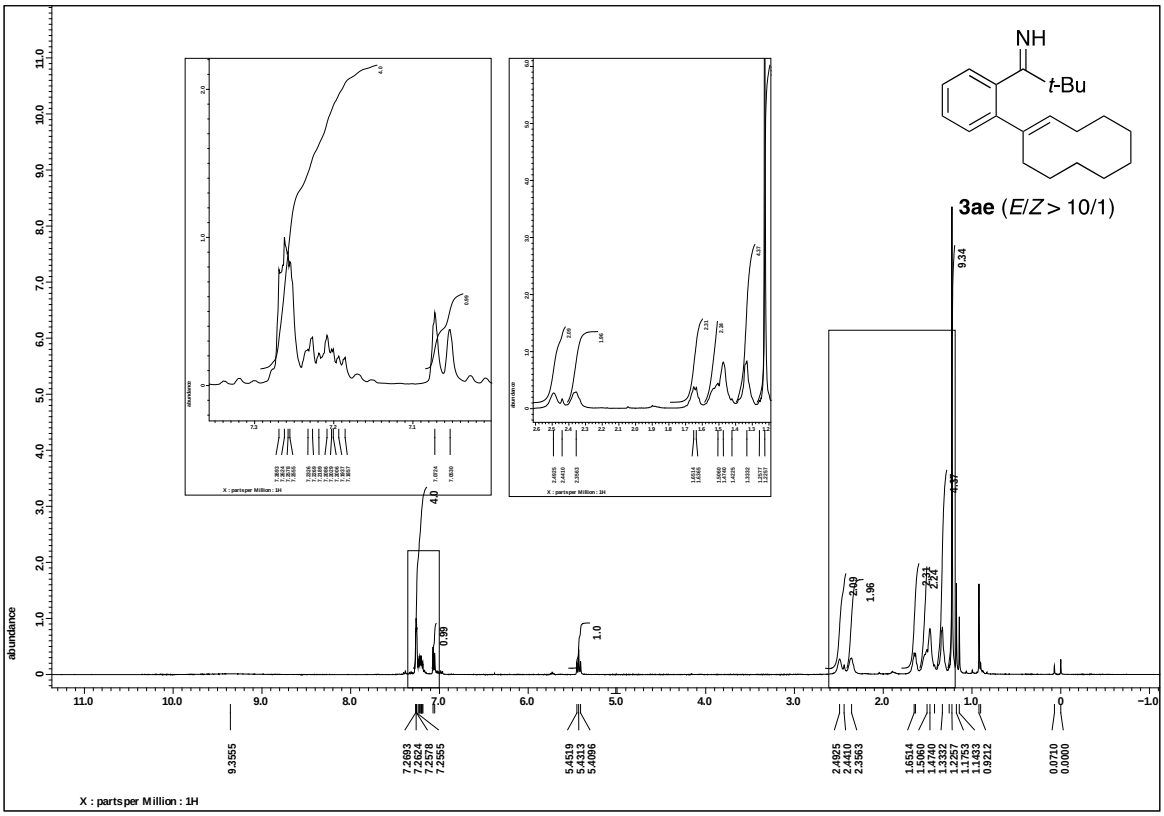

NH
t-Bu
3ab
abundance
X : parts per Million : 13C

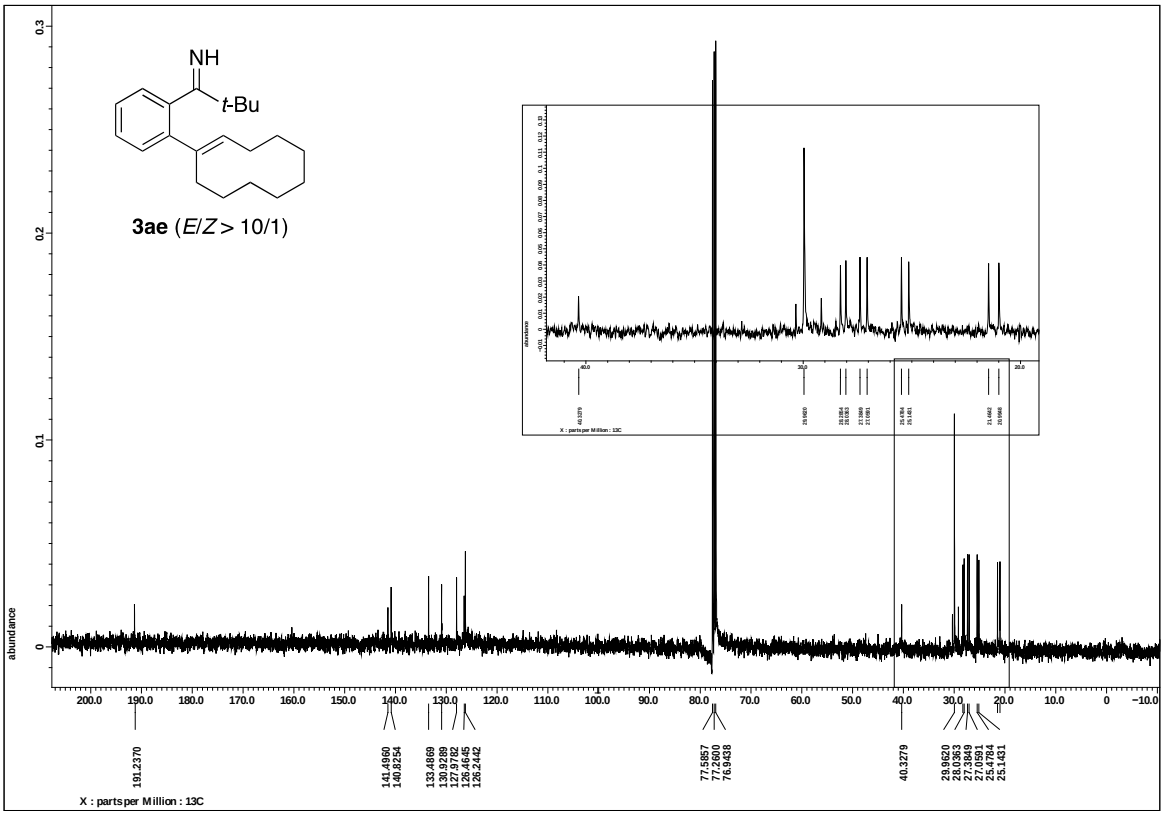

NH
t-Bu
3ac
abundance
X : parts per Million : 1H

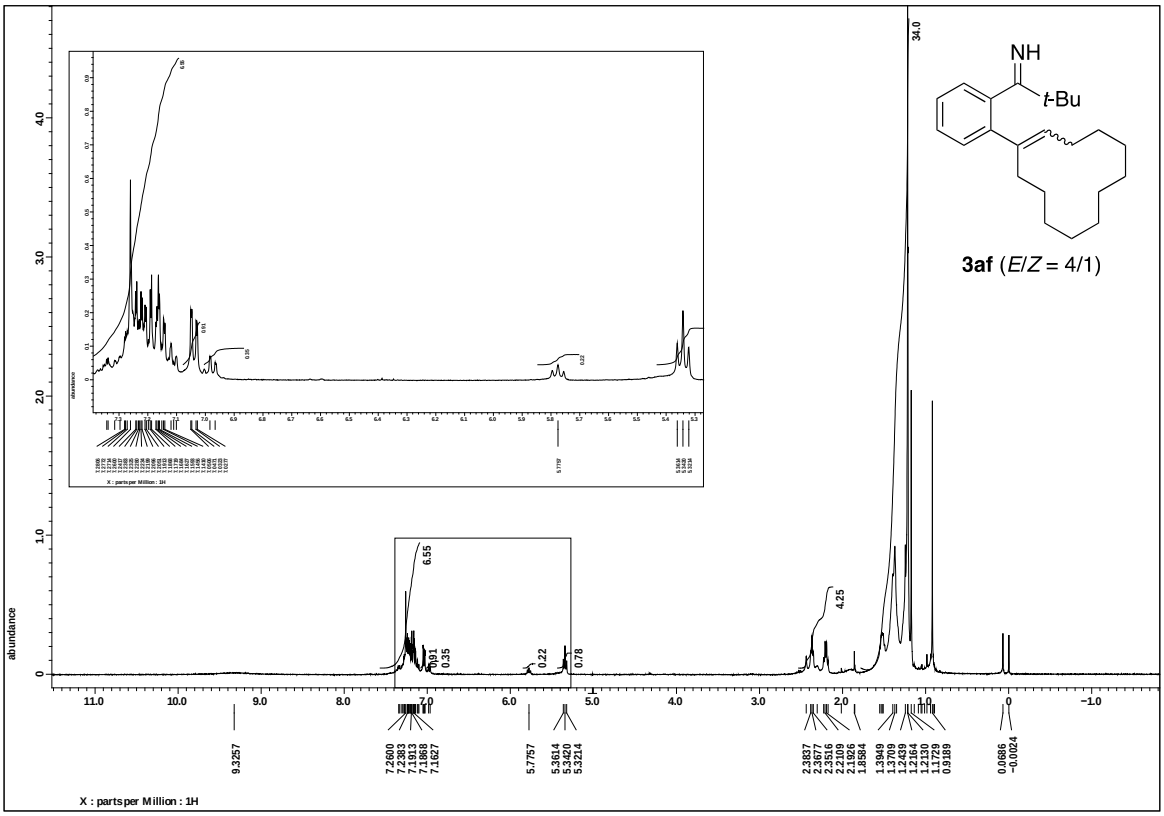

NH
t-Bu
3ac
abundance
X : parts per Million : 13C

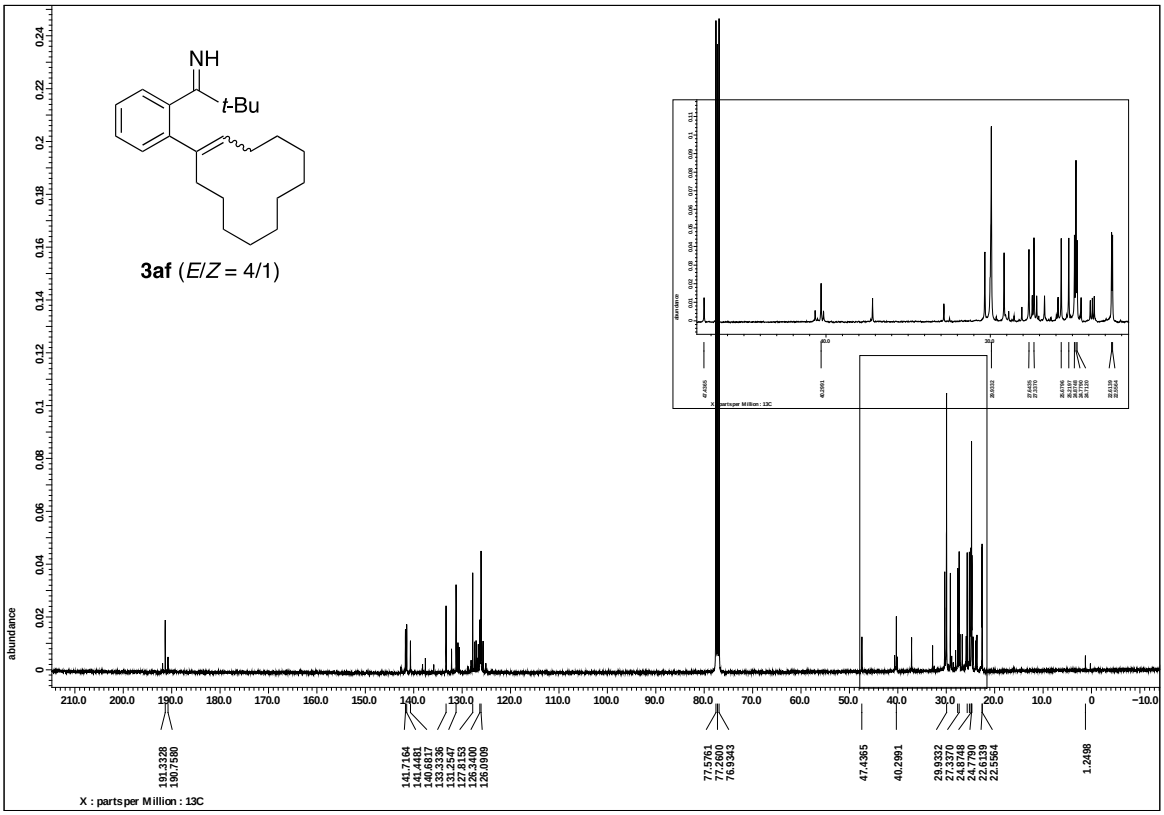

NH
t-Bu
3ad
abundance
X : parts per Million : 1H

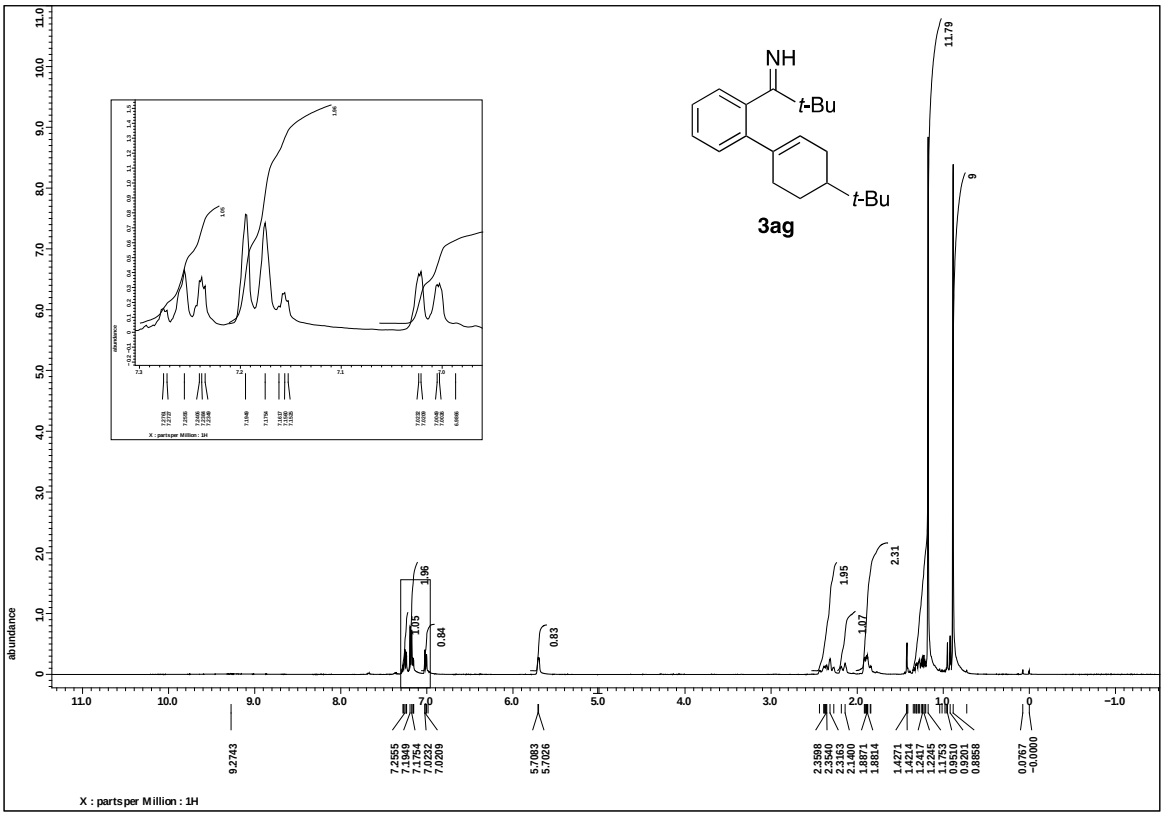

NH
t-Bu
3ad
abundance
X : parts per Million : 13C

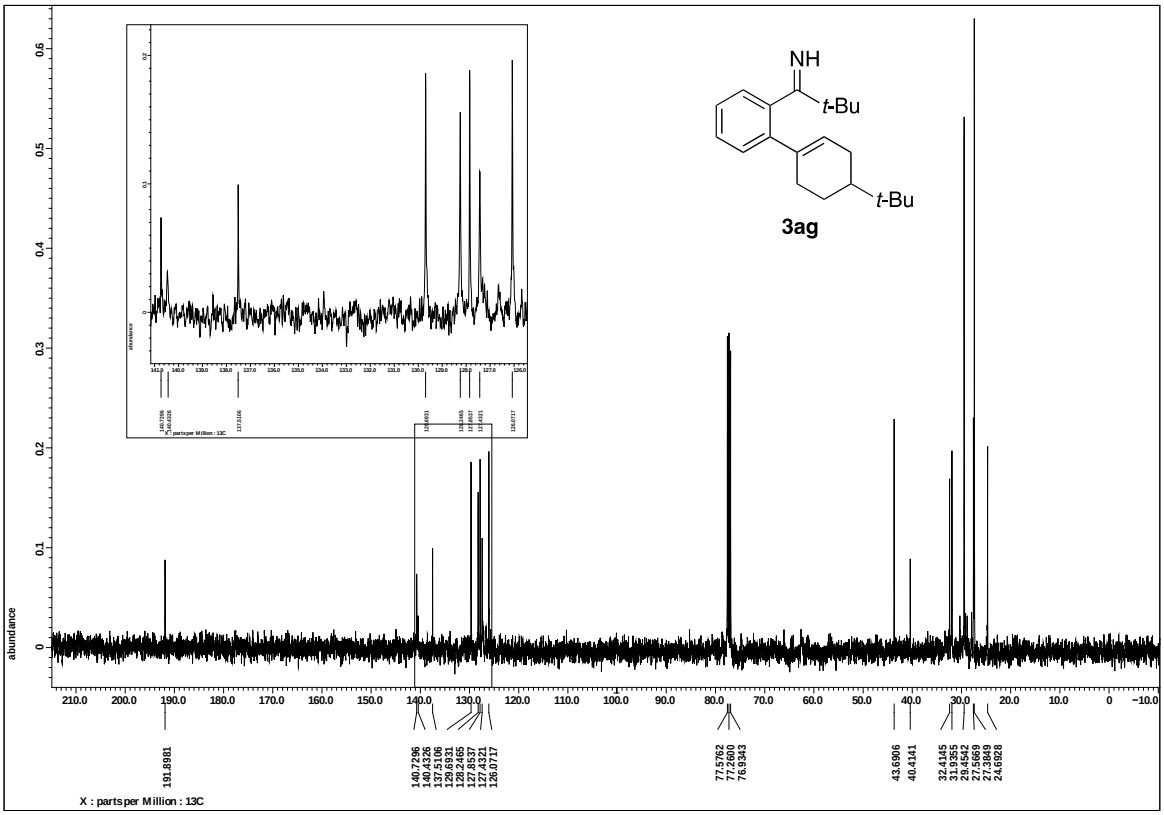

NH
t-Bu
3ae (E/Z > 10/1)
abundance
X : parts per Million : 1H

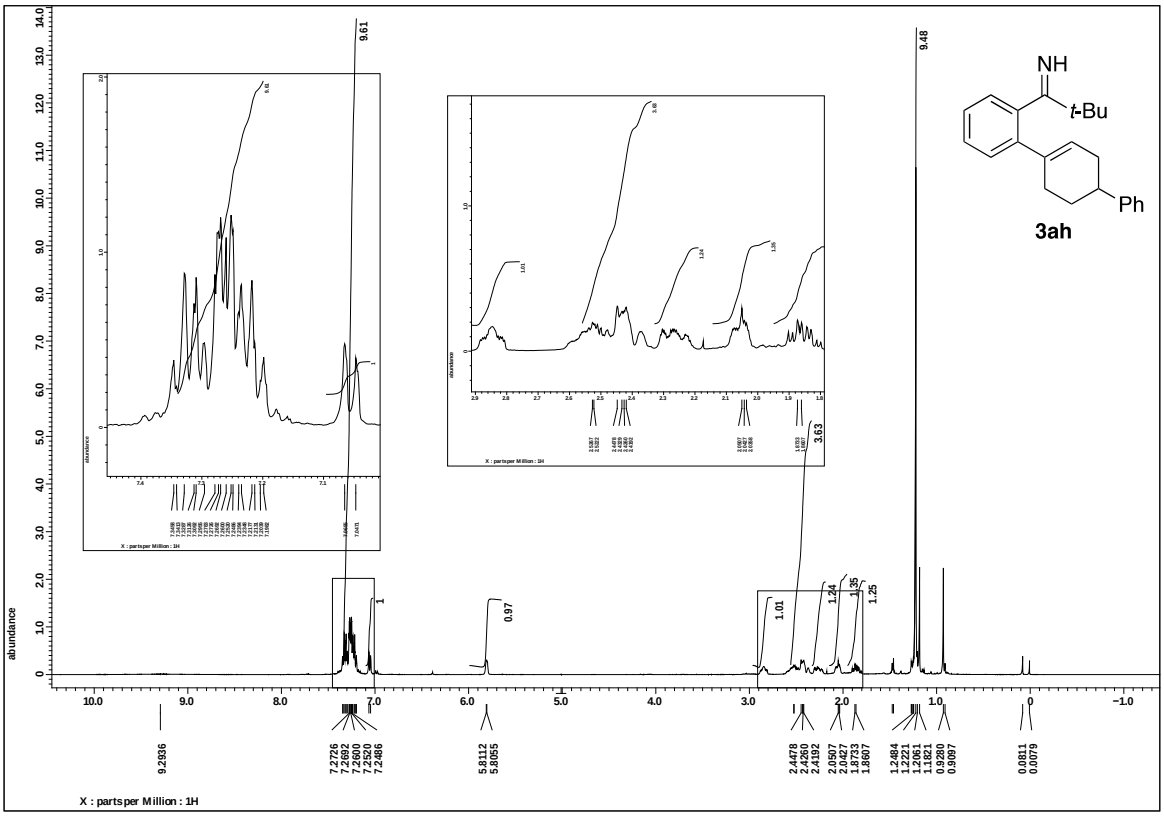

NH
t-Bu
3ae (E/Z > 10/1)
abundance
X : parts per Million : 13C

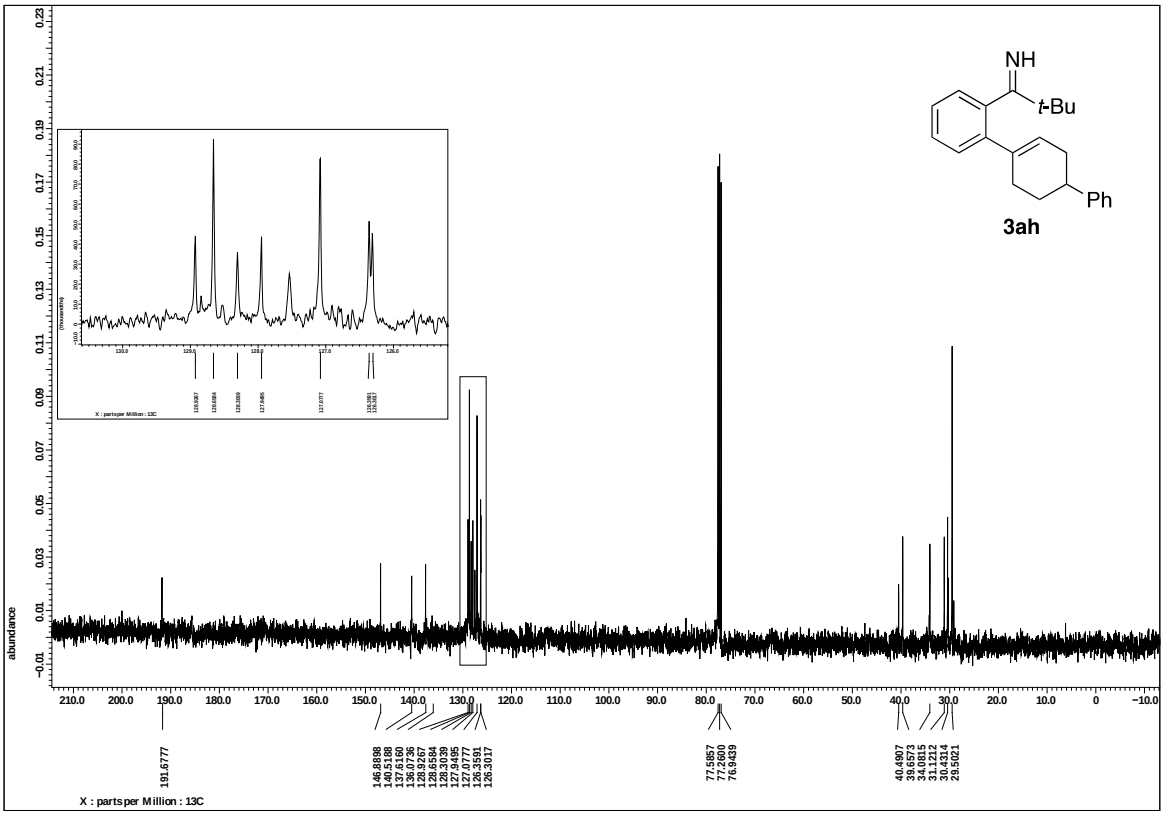

NH
t-Bu
3af (E/Z = 4/1)
abundance
X : parts per Million : 1H

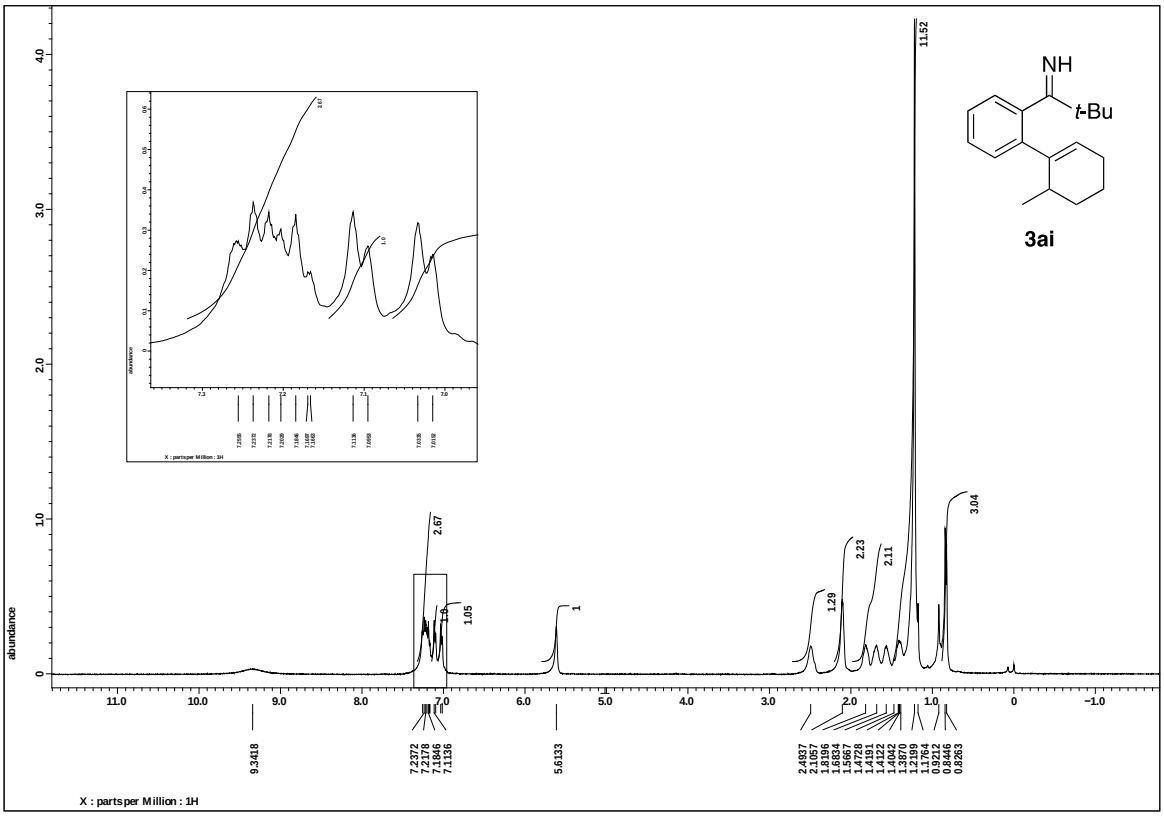

NH
t-Bu
3af (E/Z = 4/1)
abundance
X : parts per Million : 13C

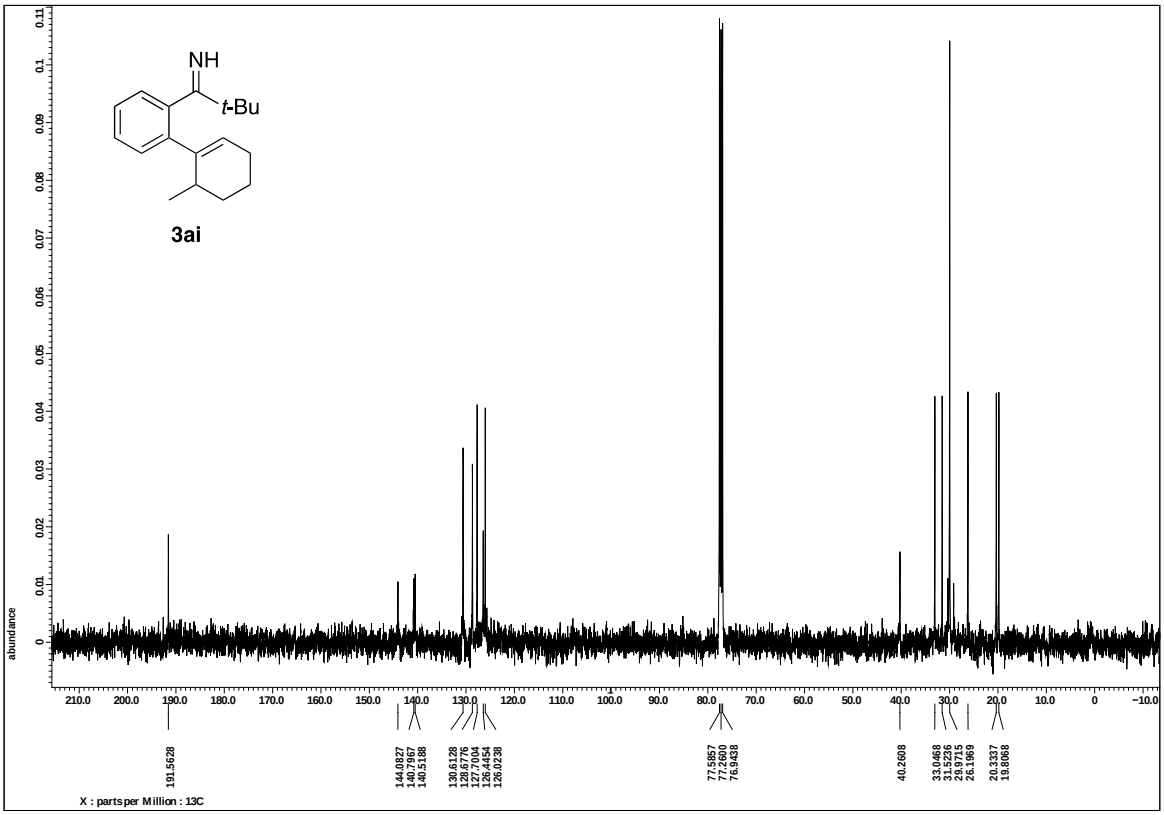

NH
t-Bu
t-Bu
3ag
abundance
X : parts per Million : 1H

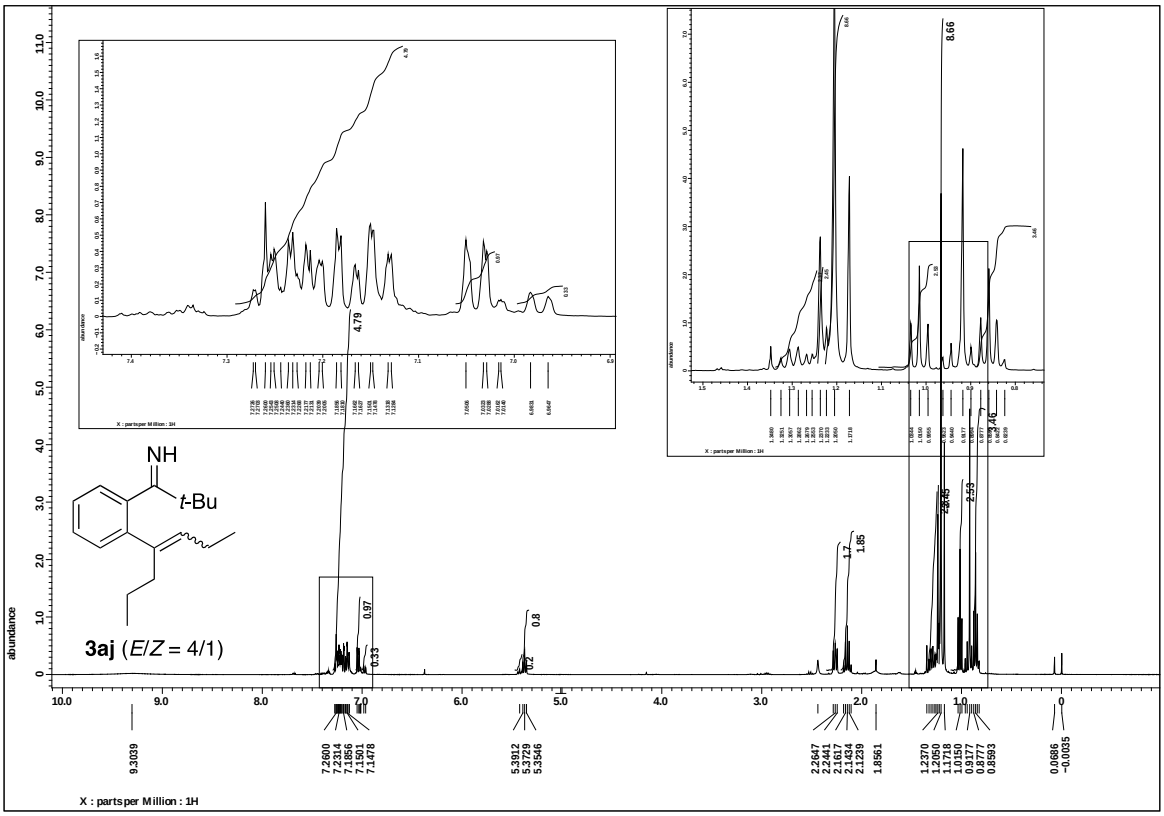

NH
t-Bu
t-Bu
3ag
abundance
X : parts per Million : 13C

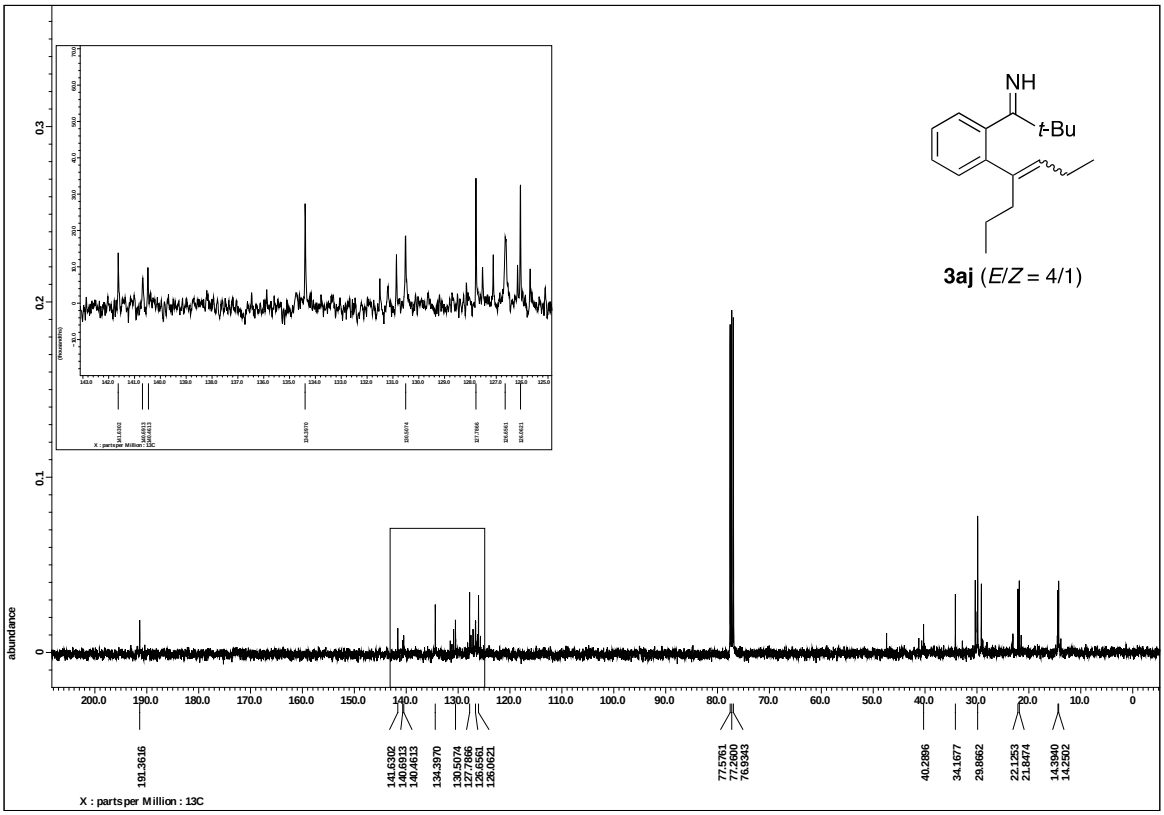

NH
t-Bu
Ph
3ah
abundance
X : parts per Million : 1H

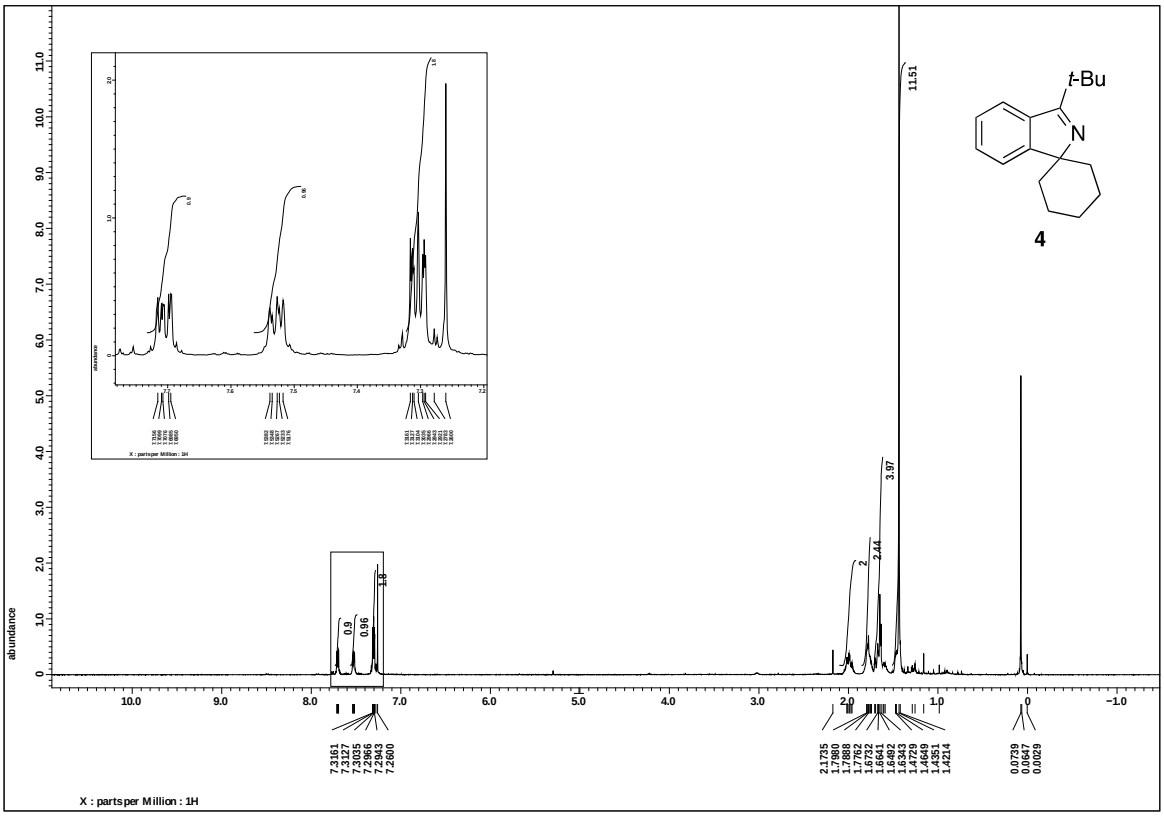

NH
t-Bu
Ph
3ah
abundance
X : parts per Million : 13C

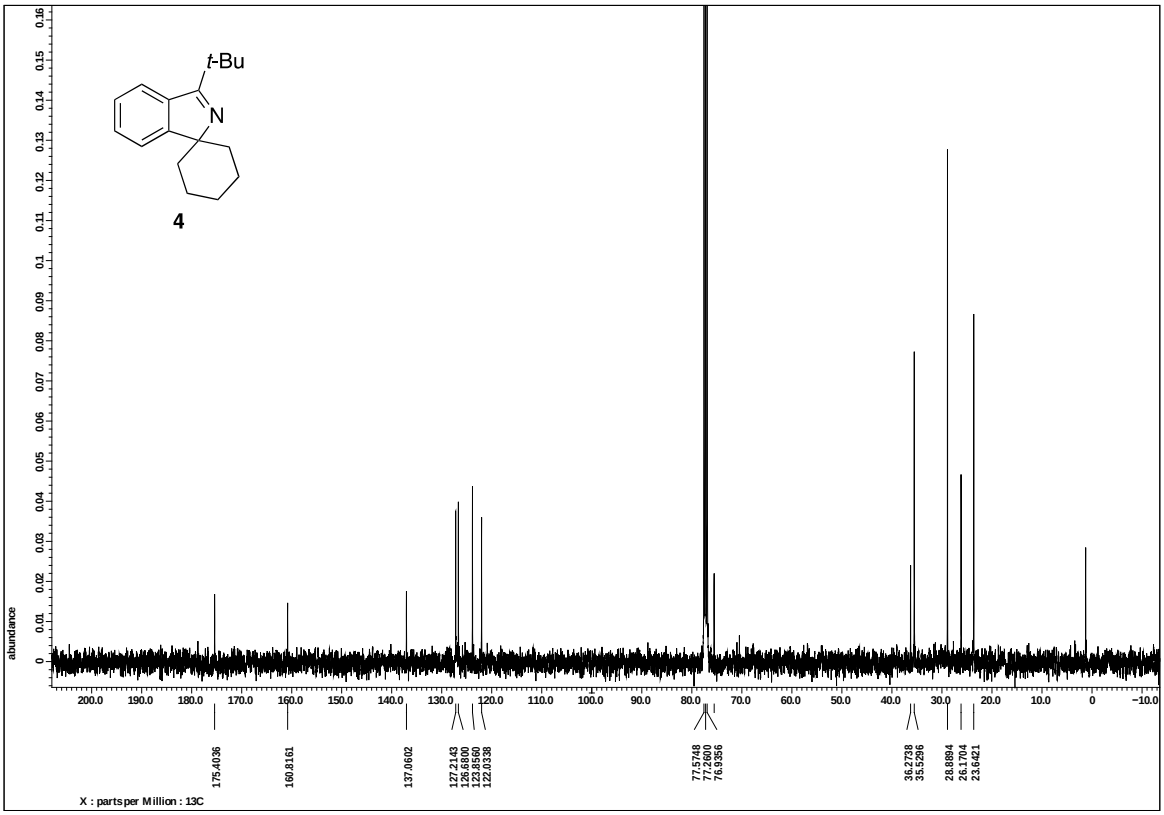

NH
t-Bu
3ai
abundance
X : parts per Million : 1H


NH
t-Bu
3ai
abundance
X : parts per Million : 13C


NH
t-Bu
3aj (E/Z = 4/1)
abundance
X : parts per Million : 1H


NH
t-Bu
3aj (E/Z = 4/1)
abundance
X : parts per Million : 13C


t-Bu
N
4
11.51
3.97
2.44
2
1.9
0.9
0.96
abundance
X : parts per Million : 1H


t-Bu
N
4
abundance
X : parts per Million : 13C