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Electronic Supplementary Information

Very efficient and broad-in-scope palladium-catalyzed Hiyama cross-coupling. The role of water and copper(I) salts.

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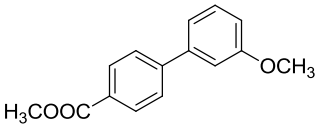
1.- Experimental Section

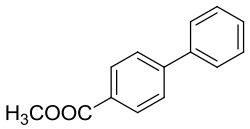
1.1.- General. ^{1}H NMR spectra were recorded in a Bruker Avance spectrometer at 300 MHz in $CDCl_3$ with tetramethylsilane (TMS) as internal standard. ^{13}C NMR spectra were recorded on the same apparatus at 75 MHz with $CDCl_3$ as solvent and reference (76.9 ppm). The chemical shifts (δ) are reported in ppm downfield from TMS and coupling constants (*J*) are expressed in hertz. Mass spectra were recorded on a Shimadzu QP2010 Plus apparatus at an ionization voltage of 70 eV equipped with a SPB^{TM}-1 capillary column (internal diameter 0.25 mm, length 30 m). The high resolution mass spectra were obtained with a Bruker MicroTOF-Q II instrument (Bruker Daltonics, Billerica, MA). Detection of the ions was performed in electrospray ionization, positive ion mode. Solvents were dried using an MBraun solvent system (SPS-800). Analytical thin–layer chromatography (TLC) was carried out with silica gel 60 F254 pre–coated aluminum sheets. Flash column chromatography was performed using Merck silica gel 60 (230-400 mesh). Elution was carried out with hexane-EtOAc mixtures, under positive pressure and employing gradient of solvent polarity techniques. Chemical reagents were purchased from commercial sources and were used without further purification unless noted otherwise. Solvents were analytical grade or were purified by standard procedures prior to use. Triethoxysilanes were commercially available except **2f** and **2g** which were prepared following the methodology described by DeShong[1].

1.2.- General Procedure: Aryl halide **1** (0.11 mmol), $Pd(PPh_3)_4$ (0.025 equiv.) and CuI (2 equiv.) were combined in a round bottom flask and placed under argon. THF (4 mL) were added, followed by phenyltriethoxysilane **2** (1.5 equiv.), TBAF (1.5 equiv., 1.0 M in THF) and H_20 (0.2 mL). The flask was fitted with a condenser and the reaction mixture was stirred 7 h at 80 ºC. The reaction mixture was evaporated and the crude product was analyzed by ^{1}H NMR and GC/MS and then purified by column chromatography (hexane-EtOAc).



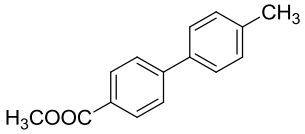
Methyl 3′-methoxy-(1,1′-biphenyl)-4-carboxylate (3aa): employing General Procedure, with methyl 4-iodobenzoate (**1a**) (30 mg, 0.11 mmol) as starting material and triethoxy(3-methoxyphenyl)silane (**2a**), desired product **3aa** was isolated in 93% yield.

Characterization of **3aa**:[2] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 3.87 (s, 3H), 3.94 (s, 3H), 6.94 (dd, *J* = 8.1 and 1.9 Hz, 1H), 7.15 (t, *J* = 1.9 Hz, 1H), 7.21 (da, *J* = 8.1, 1H), 7.38 (t, *J* = 8.1 Hz, 1H), 7.65 (d, *J* = 8.5 Hz, 2H), 8.10 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 52.2, 55.4, 113.0, 113.5, 119.8, 127.1, 129.0, 129.9, 130.1, 141.5, 145.5, 160.0, 167.0. GC/MS: ^{t}R 21.66 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 242 (M^+), 211 (100).



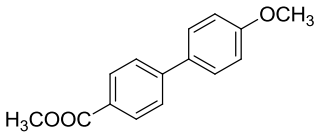
Methyl-4-biphenylcarboxylate (3ab): employing General Procedure, with methyl 4-iodobenzoate (**1a**) (30 mg, 0.11 mmol) as starting material and triethoxy(phenyl)silane (**2b**), desired product **3ab** was isolated in 84% yield.

Characterization of **3ab**:[3] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 3.95 (s, 3H), 7.36-7.51 (m, 3H), 7.60-7.70 (m, 4H), 8.12 (d, *J* = 8.26 Hz, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 52.1, 127.0, 127.2, 128.1, 128.9, 130.1, 140.0, 145.6, 167.0. GC/MS: ^{t}R 20.0 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 212 (M^+), 181 (100).



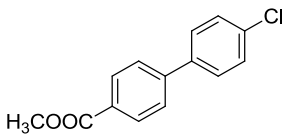
Methyl 4′-methyl-(1,1′-biphenyl)-4-carboxylate (3ac): employing General Procedure, with methyl 4-iodobenzoate (**1a**) (30 mg, 0.11 mmol) as starting material and triethoxy(p-tolyl)silane (**2c**), desired product **3ac** was isolated in 98% yield.

Characterization of **3ac**:[4] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 2.41 (s, 3H), 3.94 (s, 3H), 7.27 (d, *J* = 8.4 Hz, 2H), 7.53 (d, *J* = 8.4 Hz, 2H), 7.65 (d, *J* = 8.5 Hz, 2H), 8.09 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 21.1, 52.1, 126.7, 127.1, 128.5, 129.6, 130.0, 137.1, 138.1, 145.5, 167.0. GC/MS: ^{t}R 21.16 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 226 (M^+), 195 (100).



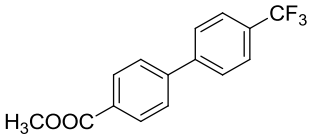
Methyl 4′-methoxy-(1,1′-biphenyl)-4-carboxylate (3ad): employing General Procedure, with methyl 4-iodobenzoate (**1a**) (30 mg, 0.11 mmol) as starting material and triethoxy(4-methoxyphenyl)silane (**2d**), desired product **3ad** was isolated in 86% yield.

Characterization of **3ad**:[2] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 3.86 (s, 3H), 3.93 (s, 3H), 6.99 (d, *J* = 8.6 Hz, 2H), [7.57 (d, *J* = 8.6 Hz), 7.61 (d, *J* = 8.3 Hz), 4H], 8.08 (d, *J* = 8.3 Hz, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 52.1, 55.4, 114.4, 126.6, 128.4, 128.5, 130.1, 132.4, 145.2, 159.8, 167.0. GC/MS: ^{t}R 22.95 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 242 (M^+, 100).



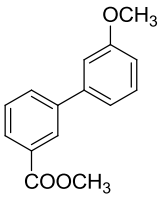
Methyl 4′-chloro-(1,1′-biphenyl)-4-carboxylate (3ae): employing General Procedure, with methyl 4-iodobenzoate (**1a**) (30 mg, 0.11 mmol) as starting material and triethoxy(4-chlorophenyl) silane (**2e**), desired product **3ae** was isolated in 93% yield.

Characterization of **3ae**:[5] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 3.94 (s, 3H), 7.43 (d, *J* = 8.6 Hz, 2H), 7.55 (d, *J* = 8.6 Hz, 2H), 7.61 (d, *J* = 8.6 Hz, 2H), 8.10 (d, *J* = 8.6 Hz, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 52.2, 126.9, 128.5, 129.1, 129,2, 130.2, 134.3, 138.4, 144.3, 166.9. GC/MS: ^{t}R 22.1 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 246 (M^+), 215 (100).



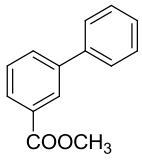
Methyl 4′-Trifluoromethyl-(1,1′-biphenyl)-4-carboxylate (3af): employing General Procedure, with methyl 4-iodobenzoate (**1a**) (30 mg, 0.11 mmol) as starting material and triethoxy(4-(trifluoromethyl)phenyl)silane (**2f**), desired product **3af** was isolated in 77% yield.

Characterization of **3af**:[6] ^{1}H NMR (300 MHz, $CDCl_3$) δ: 3.96 (s, 3H), 7.67 (d, *J* = 8.3 Hz, 2H), 7.73 (s, 4H), 8.14 (d, *J* = 8.3 Hz, 2H). ^{13}C NMR (75 MHz, CDCl3): δ 52.4, 126.0 (q, *J* = 3.8 Hz), 126.1, 127.4, 127.8, 129.9, 130.4, 143.7, 143.7, 144.2, 166.9. GC/MS: ^{t}R 19.8 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 280 (M^+), 249 (100).



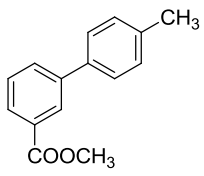
Methyl 3′-methoxy-(1,1′-biphenyl)-3-carboxylate (3ba): employing General Procedure, with methyl 3-iodobenzoate (**1b**) (30 mg, 0.11 mmol) as starting material and triethoxy(3-methoxyphenyl)silane (**2a**), desired product **3ba** was isolated in 93% yield.

Characterization of **3ba**:[7] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 3.88 (s, 3H), 3.95 (s, 3H), 6.93 (ddd, *J* = 8.0 and 2.6 Hz, 1H), 7.15 (t, *J* = 2.0 Hz, 1H), 7.21 (d, *J* = 8.0 Hz, 1H), 7.38 (t, *J* = 7.7 Hz, 1H), 7.50 (t, *J* =7.7 Hz, 1H), 7.78 (ddd, 7.7, 1.6 and 1.2 Hz, 1H), 8.02 (dt, *J* = 7.7 and 1.6 Hz, 1H), 8.28 (t, *J* =1.6 Hz, 1H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 52.1, 55.3, 112.9, 113.1, 119.6, 128.2, 128.5, 128.8, 129.9, 130.6, 131.5, 141.3, 141.6, 160.0, 167.0. GC/MS: ^{t}R 21.2 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 242 (M^+, 100).



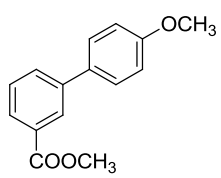
Methyl-3-biphenylcarboxylate (3bb): employing General Procedure, with methyl 3-iodobenzoate (**1b**) (30 mg, 0.11 mmol) as starting material and triethoxy(phenyl)silane (**2b**), desired product **3bb** was isolated in 78% yield.

Characterization of **3bb**:[4] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 3.95 (s, 3H), 7.34-7.56 (m, 4H), 7.63 (d, *J* = 7.1 Hz, 2H), 7.79 (d, *J* = 7.7 Hz, 1H), 8.02 (d, *J* = 7.7 Hz, 1H), 8.28 (s, 1H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 52.2, 127.1, 127.7, 128.2, 128.3, 128.8, 128.8, 130.7, 131.5, 140.1, 141.4, 167.0. GC/MS: ^{t}R 18.98 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 212 (M^+), 181 (100).



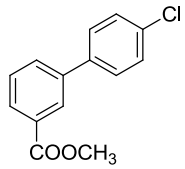
Methyl 4′-methyl-(1,1′-biphenyl)-3 –carboxylate (3bc): employing General Procedure, with methyl 3-iodobenzoate (**1b**) (30 mg, 0.11 mmol) as starting material and triethoxy(p-tolyl)silane (**2c**), desired product **3bc** was isolated in 76% yield.

Characterization of **3bc**:[8] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 2.41 (s, 3 H), 3.95 (s, 3H), 7.27 (d, *J* = 8.3 Hz, 2 H), 7.47-7.55 (m, 3H), 7.77 (dt, *J* = 7.5 and 1.3 Hz, 1H), 8.0 (dt, *J* = 7.5 and 1.3 Hz, 1H), 8.28 (t, *J* = 1.3 Hz, 1H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 21.1, 52.1, 126.9, 128.0, 128.8, 129.6, 130.6, 131.3, 137.2, 137.6, 141.4, 167.1. GC/MS: ^{t}R 19.9 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 226 (M^+, 100).



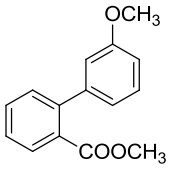
Methyl 4′-methoxy(1,1′-biphenyl)-3-carboxylate (3bd): employing General Procedure, with methyl 3-iodobenzoate (**1b**) (30 mg, 0.11 mmol) as starting material and triethoxy(4-methoxyphenyl)silane (**2d**), desired product **3bd** was isolated in 92% yield.

Characterization of **3bd**:[2] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 3.86 (s, 3H), 3.95 (s, 3H), 7.00 (d, *J* = 8.7 Hz, 2H), 7.48 (t, *J* = 7.7 Hz, 1H), 7.57 (d, *J* = 8.7 Hz, 2H), 7.74 (d, *J* = 7.7 Hz, 1H), 7.97 (d, *J* = 7.7 Hz, 1H), 8.24 (s, 1H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 52.2, 55.2, 127.1, 127.7, 128.2, 128.3, 128.8, 128.8, 130.7, 131.5, 140.1, 141.4, 167.0. GC/MS: ^{t}R 21.9 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 242 (M^+, 100).



Methyl 4′-chloro-(1,1′-biphenyl)-3-carboxylate (3be): employing General Procedure, with methyl 3-iodobenzoate (**1b**) (30 mg, 0.11 mmol) as starting material and (4-chlorophenyl)triethoxysilane (**2e**), desired product **3be** was isolated in 85% yield.

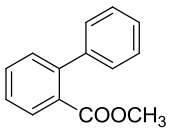
Characterization of **3be**:[9] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 3.95 (s, 3H), 7.42 (d, *J* = 8.6 Hz, 2H), 7.48-7.56 (m, 3H), 7.73 (dd, *J* = 7.6 and 1.5 Hz, 1H), 8.03 (dt, *J* = 7.6 and 1.3 Hz, 1H), 8.23 (t, *J* = 1.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 52.2, 128.0, 128.4, 128.6, 129.0, 129.0, 130.8, 131.3, 133.9, 138.5, 140.2, 166.8. GC/MS: ^{t}R 20.8 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 246 (M^+, 100).



Methyl 3′-methoxy-(1,1′-biphenyl)-2-carboxylate (3ca): employing General Procedure, with methyl 2-iodobenzoate (**1c**) (30 mg, 0.11 mmol) as starting material and triethoxy(3-methoxyphenyl)silane (**2a**), desired product **3ca** was isolated in 82% yield.

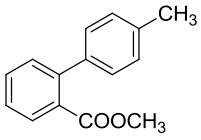
Characterization of **3ca**:[2] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 3.65 (s, 3H), 3.83 (s, 3H), 6.87-6.92 (m, 3H), 7.30 (t, *J* = 7.7 Hz, 1H), 7.37-7.44 (m, 2H), 7.52 (dt, *J* = 7.5 and 1.4 Hz, 1H), 7.79-7.82 (m, 1H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 52.0, 55.2, 112.8, 113.8, 120.9, 127.2, 129.0, 129.6, 130.5, 131.0, 131.1, 142.2,

142.7, 159.3, 169.1. GC/MS: ^{t}R 19.19 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 242 (M^+, 100).



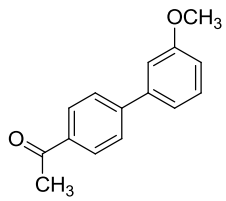
Methyl-(1,1′-biphenyl)-2-carboxylate (3cb): employing General Procedure, with methyl 2-iodobenzoate (**1c**) (30 mg, 0.11 mmol) as starting material and triethoxy(phenyl)silane (**2b**), desired product **3cb** was isolated in 90% yield.

Characterization of **3cb**:[10] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 3.64 (s, 3H), 7.30-7.44 (m, 7H), 7.52 (td, *J* = 7.5 and 1.4 Hz, 1H), 7.83 (dd, *J* = 7.7 and 0.9 Hz, 1H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 51.9, 127.1, 127.2, 128.0, 128.3, 129.7, 169.1, 130.8, 131.2, 141.3, 142.4, 169.1. GC/MS: ^{t}R 16.5 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 212 (M^+), 181 (100).



Methyl 4′-methyl-(1,1′-biphenyl)-2-carboxylate (3cc): employing General Procedure, with methyl 2-iodobenzoate (**1c**) (30 mg, 0.11 mmol) as starting material and triethoxy(p-tolyl)silane (**2c**), desired product **3cc** was isolated in 88% yield.

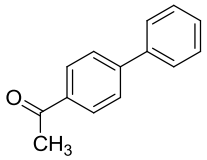
Characterization of **3cc**:[11] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 2.40 (s, 3H), 3.67 (s, 3H), 7.21 (s, 4H), 7.36-7.42 (m, 2H), 7.49-7.54 (m, 1H), 7.81 (d, *J* = 7.8 Hz, 1H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 21.2, 51.9, 126.9, 128.8, 128.2, 129.7, 130.7, 130.8, 131.2, 136.9, 138.3, 142.4, 169.2. GC/MS: ^{t}R 17.7 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 226 (M^+), 195 (100).



1-(3′-methoxy-[1,1′-biphenyl]-4-yl)ethanone (3da): employing General Procedure, with 4-iodoacetophenone (**1d**) (30 mg, 0.12 mmol) as starting material and triethoxy(3-methoxyphenyl)silane (**2a**), desired product **3da** was isolated in 91% yield.

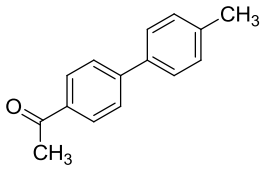
Characterization of **3da**:[16] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 2.63 (s, 3H), 3.87 (s, 3H), 6.95 (ddd, *J* = 7.9, 2.50 and 1.2 Hz, 1H), 7.15 (t, *J* = 1.2 Hz, 1H), 7.21 (dt, *J* = 7.9 and 1.2 Hz, 1H), 7.39 (t, *J* = 7.9 Hz, 1H), 7.67 (d, *J* = 8.5 Hz, 2H), 8.02 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 26.6, 55.3, 113.0, 113.5, 119.7,

127.2, 128.8, 129.9, 135.9, 141.3, 145.6, 160.0, 197.7. GC/MS: ^{t}R 21.2 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 226 (M^+), 211 (100).



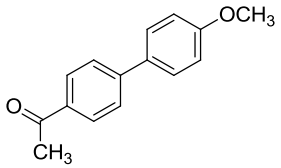
1-([1,1′-biphenyl]-4-yl)ethanone (3db): employing General Procedure, with 4-iodoacetophenone (**1d**) (30 mg, 0.12 mmol) as starting material and triethoxy(phenyl)silane (**2b**), desired product **3db** was isolated in 88% yield.

Characterization of **3db**:[4] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 2.64 (s, 2.64), 7.40-7.50 (m, 3H), 7.63 (d, *J* = 8.0 Hz, 2H), 7.69 (d, *J* = 8.0 Hz, 2H), 8.04 (d, *J* = 8.0 Hz, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 26.6**,** 127.2, 127.2, 128.2, 128.9, 128.9, 135.8, 139.8, 145.7, 197.7. GC/MS: ^{t}R 18.4 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 196 (M^+), 181(100).



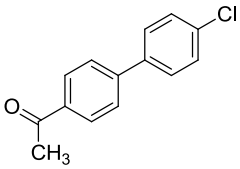
1-(4′-methyl-[1,1′-biphenyl]-4-yl)ethanone (3dc): employing General Procedure, with 4-iodoacetophenone (**1d**) (30 mg, 0.12 mmol) as starting material and triethoxy(p-tolyl)silane (**2c**), desired product **3dc** was isolated in 93% yield.

Characterization of **3dc**:[12] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 2.41 (s, 3H), 2.63 (s, 3H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.53 (d, *J* = 8.0 Hz, 2H), 7.67 (d, *J* = 8.0 Hz, 2H), 8.02 (d, *J* = 8.0 Hz, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 21.1, 26.6, 126.9, 127.1, 128.9, 129.6, 135.6, 136.9, 138.2, 145.7, 197.74. GC/MS: ^{t}R 19.8 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 210 (M^+), 195 (100).



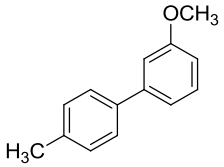
1-(4′-methoxy-[1,1′-biphenyl]-4-yl)ethanone (3dd): employing General Procedure, with 4-iodoacetophenone (**1d**) (30 mg, 0.12 mmol) as starting material and triethoxy(4-methoxyphenyl)silane (**2d**), desired product **3dd** was isolated in 85% yield.

Characterization of **3dd**:[5] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 2.62 (s, 3H), 3.86 (s, 3H), 7.00 (d, *J* = 8.8 Hz, 2H), 7.58 (d, *J* = 8.8 Hz, 2H), 7.64 (d, *J* = 8.4 Hz, 2H), 8.00 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 26.6, 55.3, 114.4, 126.6, 128.3, 128.9, 132.2, 135.2, 145.3, 159.9, 197.7. GC/MS: ^{t}R 21.4 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 226 (M^+), 211 (100).



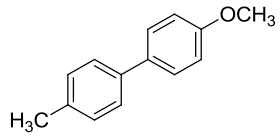
1-(4′-chloro-[1,1′-biphenyl]-4-yl)ethanone (<u>3de</u>): employing General Procedure, with 4-iodoacetophenone (**1d**) (30 mg, 0.12 mmol) as starting material and (4-chlorophenyl)triethoxysilane (**2e**), desired product **3de** was isolated in 91% yield.

Characterization of **3de**:[12] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 2.64 (s, 3H), 7.44 (d, *J* = 8.5 Hz, 2H), 7.55 (d, *J* = 8.5 Hz, 2H), 7.65 (d, *J* = 8.5 Hz, 2H), 8.03 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 26.6, 127.0, 128.5, 129.0, 129.1, 134.4, 136.1, 138.2, 144.4, 197.5. GC/MS: ^{t}R 20.6 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 230 (M^+), 215 (100).



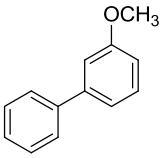
3-methoxy-4'-methyl-1,1'-biphenyl (<u>3ea</u>): employing General Procedure, with 4-iodotoluene (**1e**) (30 mg, 0.14 mmol) as starting material and triethoxy(3-methoxyphenyl)silane (**2a**), desired product **3ea** was isolated in 76% yield.

Characterization of **3ea**:[13] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 2.42 (s, 3H), 3.88 (s, 3H), 6.90 (d, *J* = 7.9 and 1.9 Hz, 1H), 7.14 (t, *J* = 1.9 Hz, 1H), 7.19 (d, *J* = 7.6 Hz, 1H), 7.27 (d, *J* = 8.0 Hz, 2H), 7.36 (dd, *J* = 7.9 and 7.6 Hz, 1H), 7.52 (d, *J* = 8.0, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 21.1, 29.7, 55.2, 112.4, 112.7, 112.9, 119.5, 119.7, 127.0, 127.2, 127.4, 128.7, 129.4, 129.7, 137.2, 138.2, 142.7, 159.9. GC/MS: ^{t}R 18.2 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 198 (M^+, 100).



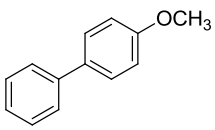
4-methoxy-4'-methyl-1,1'-biphenyl (<u>3ed</u>): employing General Procedure, with 4-iodotoluene (**1e**) (30 mg, 0.14 mmol) as starting material and triethoxy(4-methoxyphenyl)silane (**2d**), desired product **3ed** was isolated in 93% yield.

Characterization of **3ed**:[5] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 2.40 (s, 3H), 3.86 (s, 3H), 6.99 (d, *J* = 8.8 Hz, 2H), 7.25 (d, *J* = 8.2 Hz, 2H), 7.47 (d, *J* = 8.2 Hz, 2H), 7.53 (d, *J* = 8.8 Hz, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 21.0**,** 55.3, 114.1, 126.6, 126.7, 127.9, 128.1, 128.7, 129.4, 133.7, 136.3, 137.9, 158.9. GC/MS: ^{t}R 18.0 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 198 (M^+, 100).



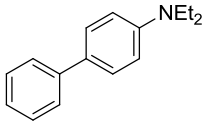
3-methoxy-1,1'-biphenyl (3fa): employing General Procedure, with 4-iodobencene (**1f**) (36.6 mg, 0.18 mmol) as starting material and triethoxy(3-methoxyphenyl)silane (**2a**), desired product **3fd** was isolated in 88% yield.

Characterization of **3fa**:[14] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 3.88 (s, 3H), 6.92 (dd, *J* = 5.7 and 2.3 Hz, 1H), 7.15-7.22 (m, 2H), 7.34-7.48 (m, 4H), 7.60-7.63 (m, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 55.3, 112.7, 112.9, 119.7, 127.2, 127.4, 128.7, 129.7, 141.1, 142.8, 159.9. GC/MS: ^{t}R 16.4 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 184 (M^+, 100).



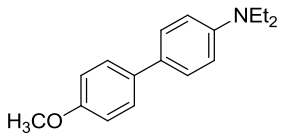
4-methoxy-1,1'-biphenyl (3fd): employing General Procedure, with 4-iodobencene (**1f**) (36.6 mg, 0.18 mmol) as starting material and triethoxy(4-methoxyphenyl)silane (**2d**), desired product **3fd** was isolated in 93% yield.

Characterization of **3fd**:[5] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 3.87 (s, 3H), 7.00 (d, *J* = 8.8 Hz, 2H), 7.31-7.35 (m, 1H), 7.41-7.45 (m, 2H), 7.55-7.60 (m, 4H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 55.3, 114.2, 126.6, 126.7, 128.1, 128.7, 133.8, 140.8, 159.1. GC/MS: ^{t}R 16.6 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 184 (M^+, 100).



N,N-diethyl-[1,1'-biphenyl]-4-amine (3gb): employing General Procedure, with N,N-diethyl-4-iodoaniline (**1g**) (45 mg, 0.16 mmol) as starting material and triethoxy(phenyl)silane (**2b**), desired product **3gb** was isolated in 65% yield.

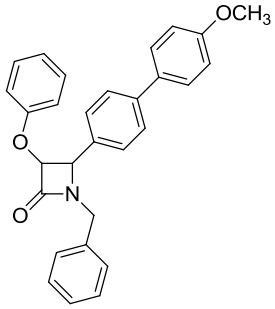
Characterization of **3gb**:[15] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 1.20 (t, *J* = 7.1 Hz, 6H), 3.40 (q, *J* = 7.1 Hz, 4H), 6.75 (d, *J* = 8.8 Hz, 2H), 7.21-7.26 (m, 1H), 7.38 (t, *J* = 7.6 Hz, 2H), 7.48 (d, *J* = 8.8 Hz, 2H), 7.55 (d, *J* = 7.1 Hz, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 12.6, 44.4, 111.9, 125.7, 126.1, 127.9, 128.1, 128.6, 141.3, 147.1. GC/MS: ^{t}R 20.27 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 225 (M^+), 210 (100).



N,N-diethyl-4'-methoxy-[1,1'-biphenyl]-4-amine (3gd): employing General Procedure, with N,N-diethyl-4-iodoaniline (**1g**) (34 mg, 0.12 mmol) as starting material and triethoxy(4-methoxyphenyl)silane (**2d**), desired product **3gd** was isolated in 71% yield.

Characterization of **3gd**: ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 1.19 (t, *J* = 7.1 Hz, 6H), 3.39 (q, *J* = 7.1 Hz, 4H), 3.83 (s, 3H), 6.74 (d, *J* = 8.8 Hz, 2H), 6.94 (d, *J* = 8.8 Hz, 2H), 7.42 (d, *J* = 8.8 Hz, 2H), 7.47 (d, *J* = 8.8 Hz, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 12.9, 44.9, 55.5, 112.9, 115.1, 127.6, 128.0, 134.6, 159.3. HRMS (ESI) m/z 256.16933 [(M + H^+); calcd for $C_{17}H_{22}NO$: 256.16959].

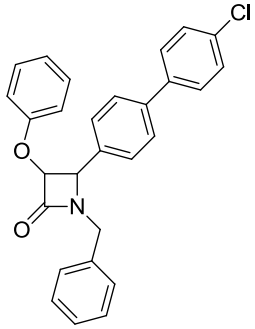
1-benzyl-4-(4'-methoxy-[1,1'-biphenyl]-4-yl)-3-phenoxyazetidin-2-one (3id):



employing General Procedure, with 1-benzyl-4-(4-iodophenyl)-3-phenoxyazetidin-2-one (**1i**) (30 mg, 0.066 mmol) as starting material and triethoxy(4-methoxyphenyl)silane (**2d**), desired product **3id** was isolated in 68% yield.

Characterization of **3id**: ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 3.85 (s, 3H), 3.91 (d, *J* = 14.7 Hz, 1H), 4.79 (d, *J* = 4.2 Hz, 1H), 4.92 (d, *J* = 14.7 Hz, 1H), 5.43 (d, *J* = 4.2 Hz, 1H), 6.75 (d, *J* = 7.6 Hz, 2H), 6.87 (t, *J* = 7.6 Hz, 1H), 6.96 (d, *J* = 8.8 Hz, 2H), 7.11 (t, *J* = 7.6 Hz, 2H), 7.18-7.21 (m, 2H), 7.31-7.34 (m, 5H), 7.46-7.52 (m, 4H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 44.2, 55.3, 61.2, 82.2, 114.2, 115.6, 122.0, 126.4, 128.0, 128.0, 128.6, 128.9, 129.1, 129.2, 131.0, 132.9, 134.8, 141.0, 157.0, 159.3, 165.6. HRMS (ESI) m/z 458.1705 [(M + Na^+); calcd for $C_{29}H_{25}O_3Na$: 458.17266].

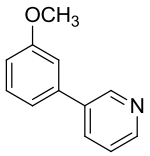
1-benzyl-4-(4'-chloro-[1,1'-biphenyl]-4-yl)-3-phenoxyazetidin-2-one (3ie):



employing General Procedure, with 1-benzyl-4-(4-iodophenyl)-3-phenoxyazetidin-2-one (**1i**) (30 mg, 0.066 mmol) as starting material and (4-chlorophenyl)triethoxysilane (**2e**), desired product **3ie** was isolated in 90% yield.

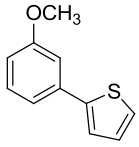
Characterization of **3ie**: ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 3.91

(d, *J* = 14.7 Hz, 1H), 4.79 (d, *J* = 4.5 Hz, 1H), 4.91 (d, *J* = 14.7 Hz, 1H), 5.42 (d, *J* = 4.5 Hz, 1H), 6.75 (d, *J* = 8.0 Hz, 2H), 6.87 (t, *J* = 7.38 Hz, 1H), 7.09-7.14 (m, 2H), 7.18-7.20 (m, 2H), 7.32-7.41 (m, 7H), 7.46-7.49 (m, 4H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 44.2, 61.1, 82.2, 115.6, 122.0, 126.8, 128.0, 128.2, 128.6, 128.9, 128.9, 129.2, 132.1, 133.6, 134.7, 138.8, 140.2, 156.9, 165.5. HRMS (ESI) m/z 462.12404 [(M + Na^+); calcd for $C_{28}H_2ClNaO_2$: 462.12313].



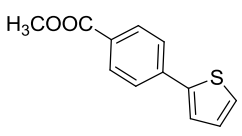
3-(3-methoxyphenyl)pyridine (<u>3ja</u>): employing General Procedure, with 3-iodopyridine (**1j**) (30 mg, 0.15 mmol) as starting material and triethoxy(3-methoxyphenyl)silane (**2a**), desired product **3ja** was isolated in 87% yield.

Characterization of **3ja**:[2] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 3.87 (s, 3H), 6.95 (ddd, *J* = 8.3, 2.4 and 0.9 Hz, 1H), 7.10 (t, *J* = 2.4 Hz, 1H), 7.15-7.18 (m, 1H), 7.33-7.42 (m, 2H), 7.85 (dt, *J* = 7.9 and 1.6 Hz, 1H), 8.59 (dd, *J* = 4.8 and 1.6 Hz, 1H), 8.85 (d, *J* = 1.6 Hz, 1H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 55.3, 112.9, 113.4, 119.6, 123.5, 130.1, 134.4, 136.5, 139.3, 148.3, 148.6, 160.1. GC/MS: ^{t}R 17.15 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 185 (M^+, 100).



2-(3-methoxyphenyl)thiophene (<u>3ka</u>): employing General Procedure, with 2-iodothiophene (**1k**) (38 mg, 0.18 mmol) as starting material and triethoxy(3-methoxyphenyl)silane (**2a**), desired product **3ka** was isolated in 93% yield.

Characterization of **3ka**:[16] ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 3.86 (s, 3H), 6.85 (ddd, *J* = 8.0, 2.4 and 0.9 Hz, 1H), 7.08 (dd, *J* = 5.0 and 3.6 Hz, 1H), 7.17 (ta, *J* = 2.1 Hz, 1H), 7.21-7.24 (m, 1H), 7.28-7.33 (m, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 55.3, 111.6, 112.9, 118.6, 123.3, 124.9, 127.9, 129.9, 135.7, 144.2, 159.9. GC/MS: ^{t}R 16.55 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 190 (M^+, 100).



2-(4-methoxycarbonylphenyl)thiophene (<u>3ag</u>): employing General Procedure, with methyl 4-iodobenzoate (**1a**) (30 mg, 0.11 mmol) as starting material and triethoxy(thiophen-2-yl)silane (**2g**), desired product **3ag** was isolated in 88% yield.

Characterization of **3ag**:[17] ^{1}H NMR ($CDCl_3$, 300 Hz) 3.93 (s, 3 H) 7.11 (dd, *J* = 5.3 and 3.8 Hz, 1H), 7.35-737 (m, 1H) 7.41-7.43 (m, 1H) 7.67 (d, *J* = 8.4 Hz, 2H), 8.04 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 52.1, 124.5, 125.5, 1126.3, 128.3, 128.8, 130.3, 138.6, 143.1, 166.7. GC/MS: ^{t}R 18.97 min (50°C (3 min), 10 °C/min, 300°C, 49.6 kPa). *m/z* (%) 187 (100), 218 (M^+).

2.- References

1.- A. S. Manoso, C. Ahn, A. Soheili, C. J. Handy, R. Correia, W. M. Seganish, and P. DeShong, *J. Org. Chem.,* 2004, **69**, 8305.

2.- P. Leowanawat, N. Zhang, M. Safi, D. J. Hoffman, M. C. Fryberger, A. George and V. J. Percec, *J. Org. Chem.,* 2012, **77**, 2885.

3.- V. Percec, G. M. Golding, J. Smidrkal and O. Weichold, *J. Org. Chem.,* 2004, **69**, 3447.

4.- L. Wu, B-L. Li, Y-Y. Huang, H-F. Zhou, Y-M. He and Q-H. Fan, *Org. Lett.,* 2006, **8**, 3605.

5.- H. Zaho, Y. Wang, J. Sha, S. Sheng and M. Cai, *Tetrahedron*, 2008, ***64,*** 7517.

6.- G. A. Molander, S. L. J. Trice and S. M. Kennedy, *J. Org. Chem.,* 2012, **77**, 8678.

7.- G. A. Molander and L. Iannazzo, *J. Org. Chem.,* 2011, **76**, 9182.

8.- C. A. Parrish and S. L. Buchwald, *J. Org. Chem.,* 2001, **66**, 3820.

9.- K. Manabe, M. Ohba and Y. Matsushima, *Org. Lett.,* 2011, **13**, 2436.

10.- S. Santra, P. Q. Hota, R. Bhattacharyya, P. Bera, P. Ghosh and S. K. Mandal, *ACS Catal.*, 2013, **3**, 2776.

11.- A. Catalano, A. Carocci, A. Di Mola, C. Bruno, P. M. L. Vanderheyden and C. Franchini, *Arch. Pharm. Chem. Life Sci.,* 2011, **344***,* 617.

12.- V. Papoian and T. Minehan, *J. Org. Chem.,* 2008, **73**, 7376.

13.- M. Marco Mor, S. Rivara, A. Lodola, P. V. Plazzi, G. Tarzia, A. Duranti, A. Tontini, P. Giovanni, S. Kathuria and D. Piomelli, *J. Med. Chem.,* 2004, **47**, 4998.

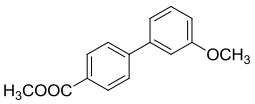
14.- W-M. Dai, Y. Li, Y. Zhang, C. Yue and J. Wu, *Chem. Eur. J.,* 2008**, 14**, 5538.

15.- H. Jasch, S. B. Hoefling and M. R. Heinrich, *J. Org. Chem.,* 2012*,* **77***,* 1520.

16.- A. Gavryushin, C. Kofink, G. Manolikakes and P. Knochel, *Org. Lett.,* 2005, **7**, 4871.

17.- P. Leowanawat, N. Zhang, A-M. Resmerita, B. M. Rosen and V. Percec, *J. Org. Chem.,* 2011, **76***,* 9946.

3.- ^{1}H NMR and ^{13}C NMR Spectra



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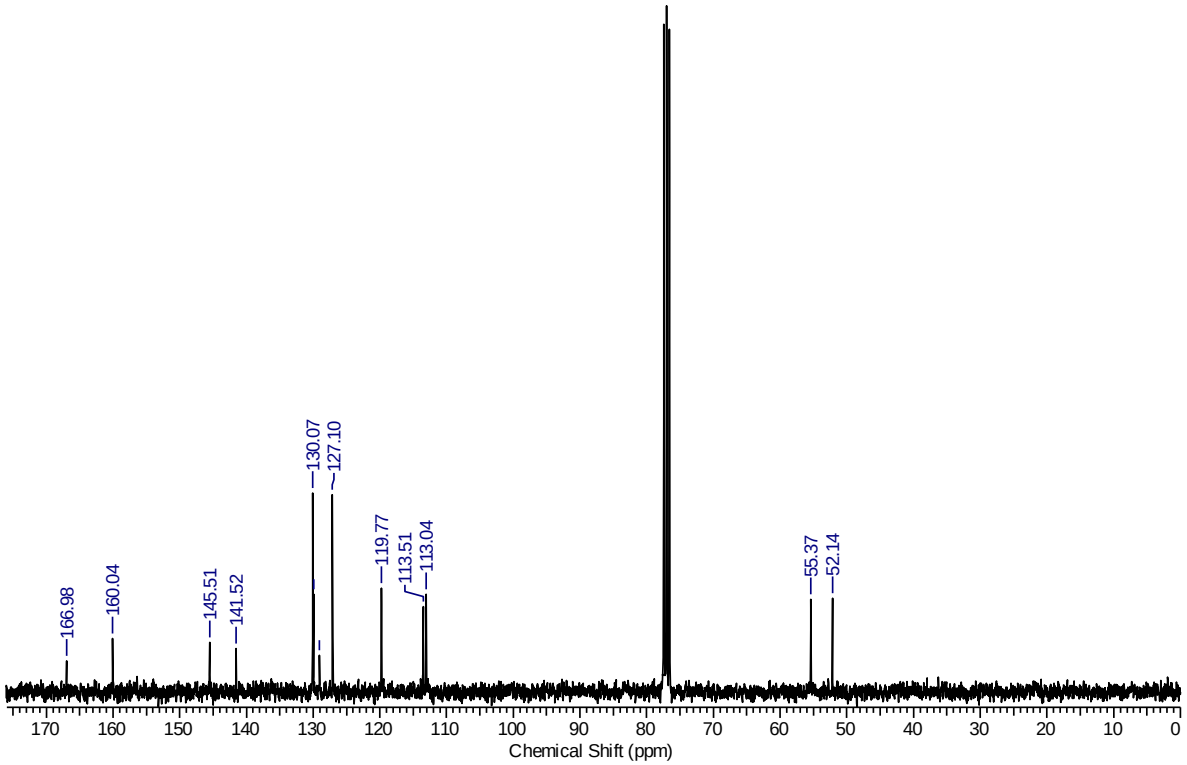

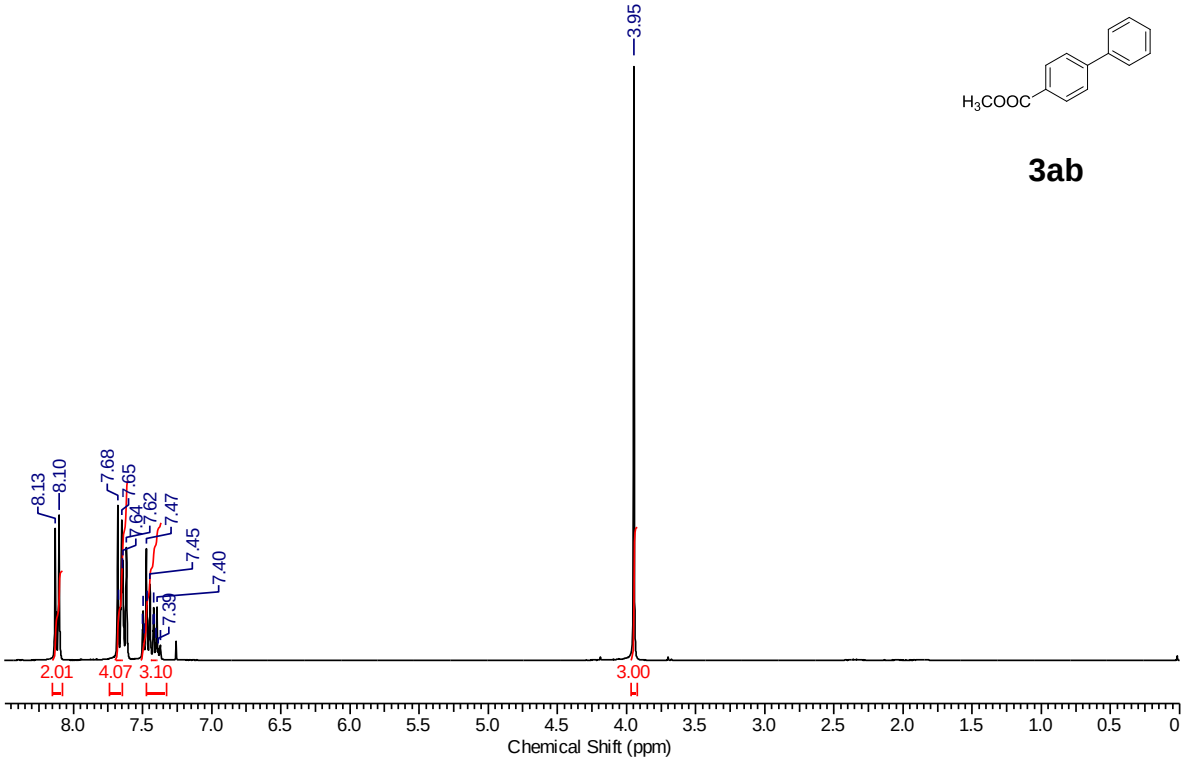
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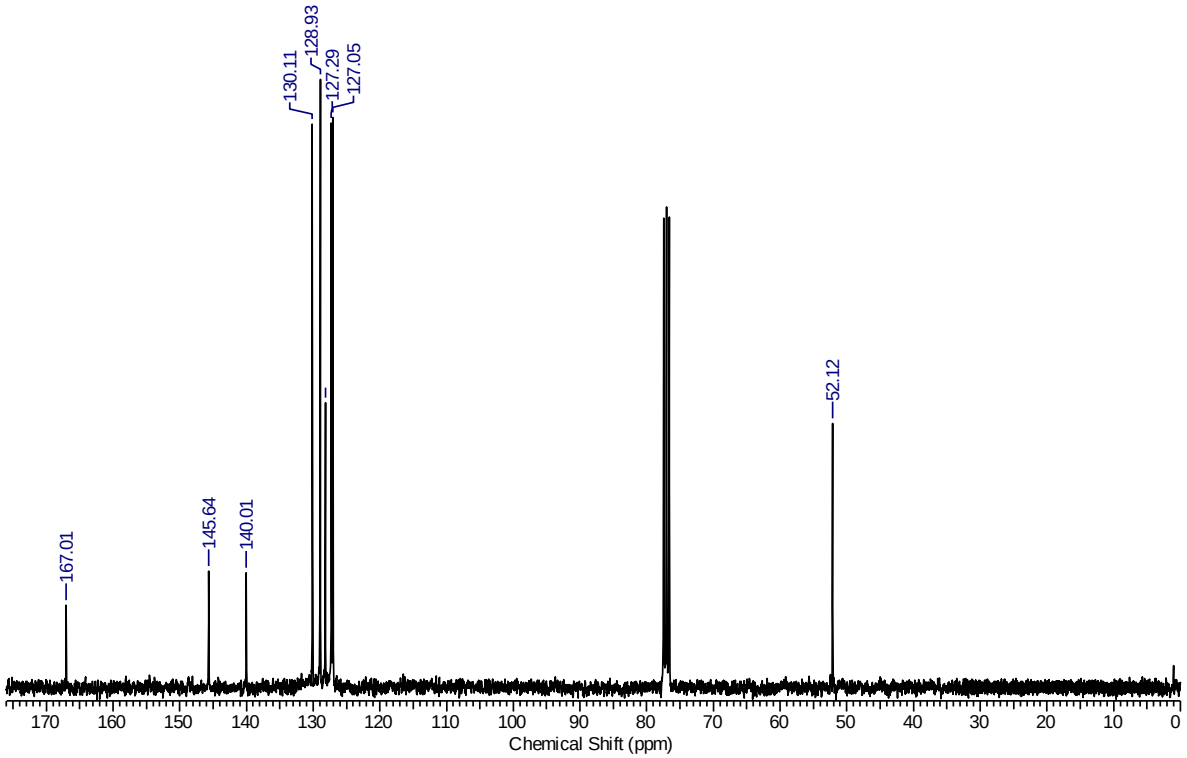
Chemical Shift (ppm)

H3COOC

3ab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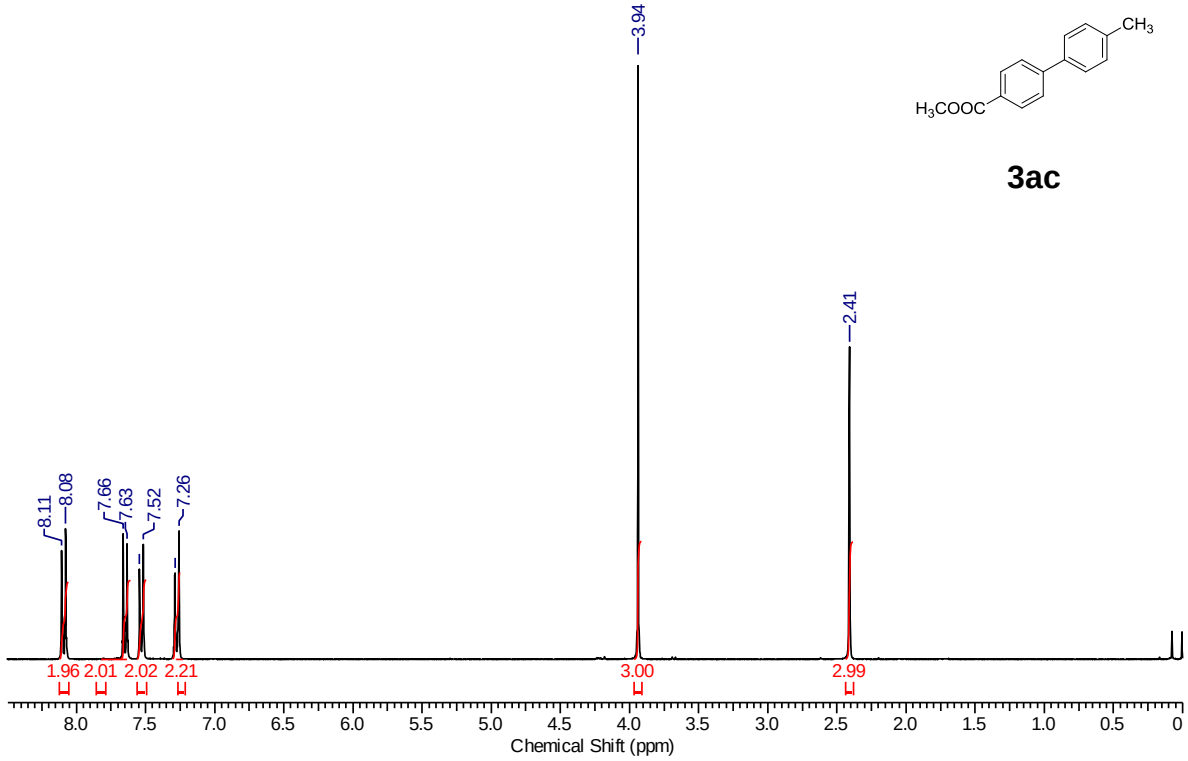
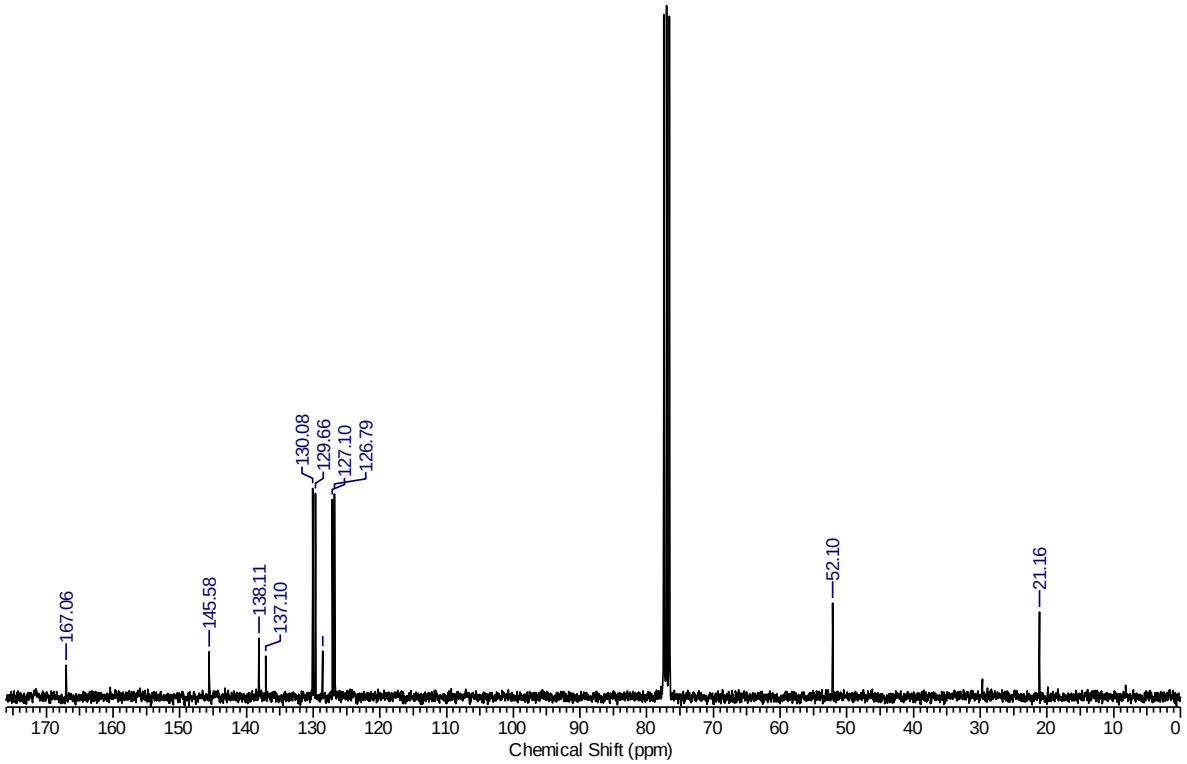
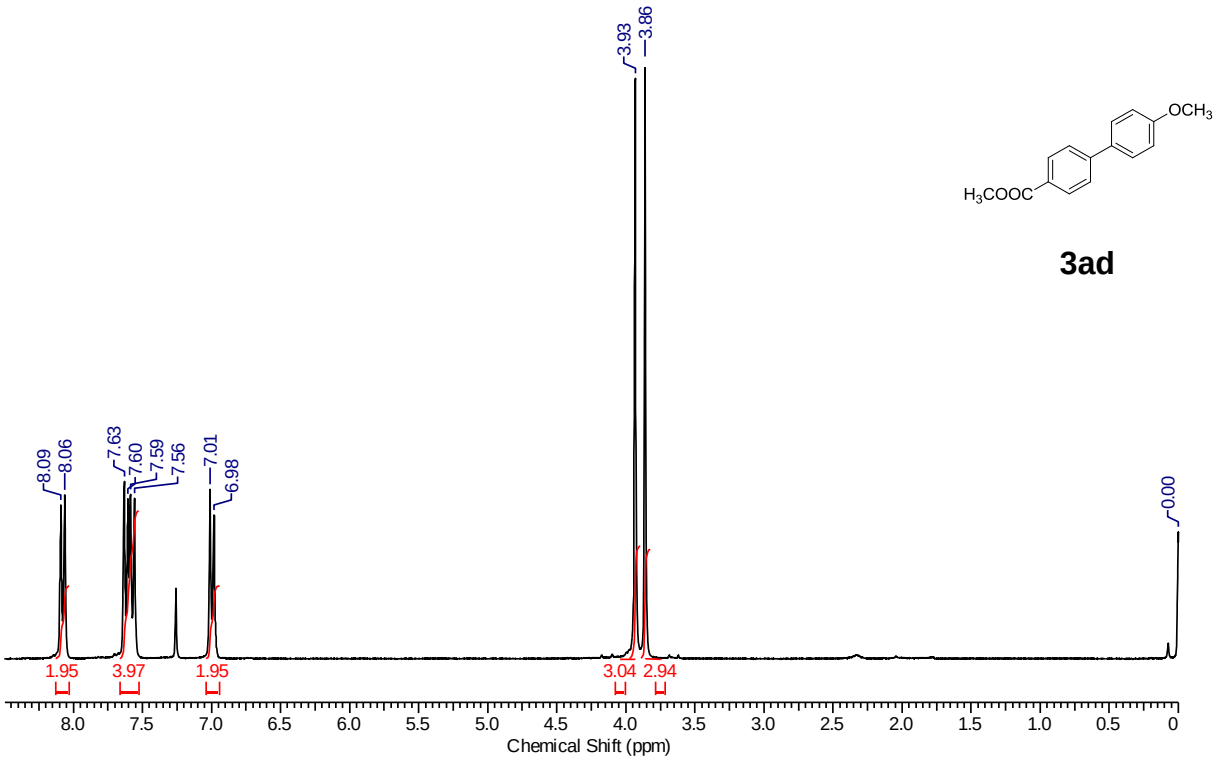
52.12
167.01
145.64
140.01
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)


3.94
2.41
8.11
8.08
7.66
7.63
7.52
7.26
1.96
2.01
2.02
2.21
3.00
2.99
Chemical Shift (ppm)

CH3
H3COOC

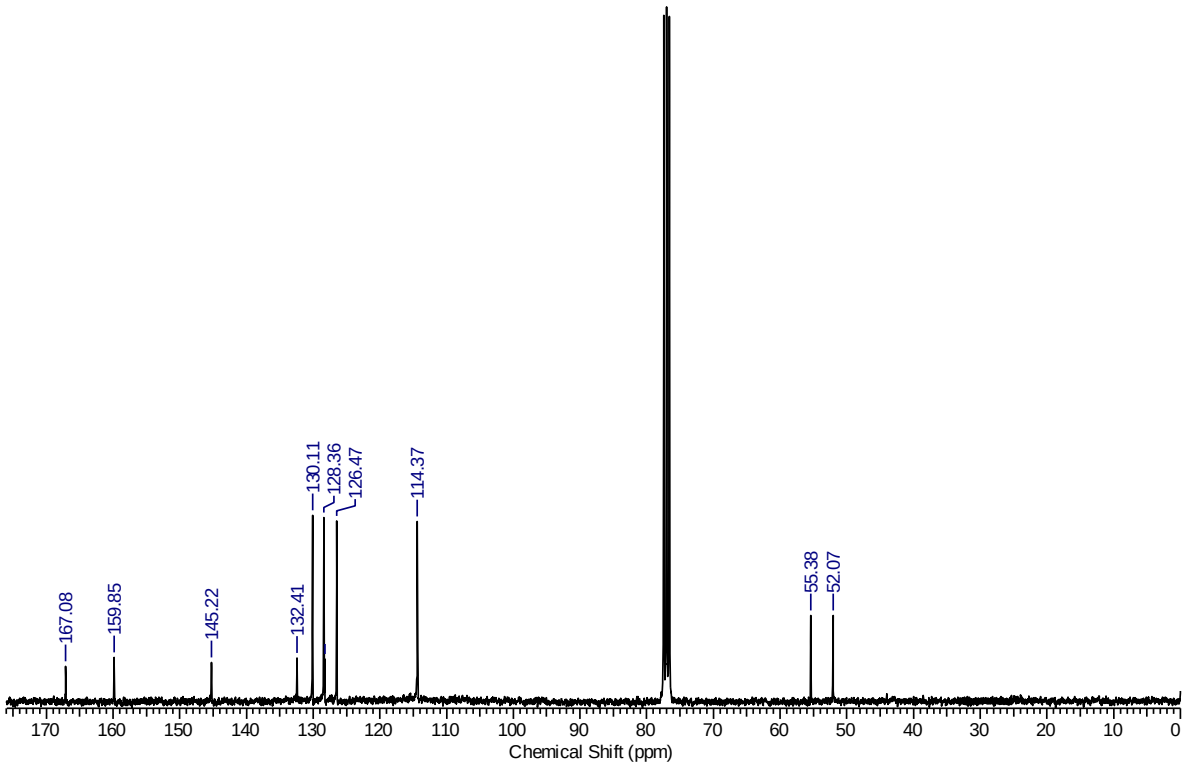
3ac

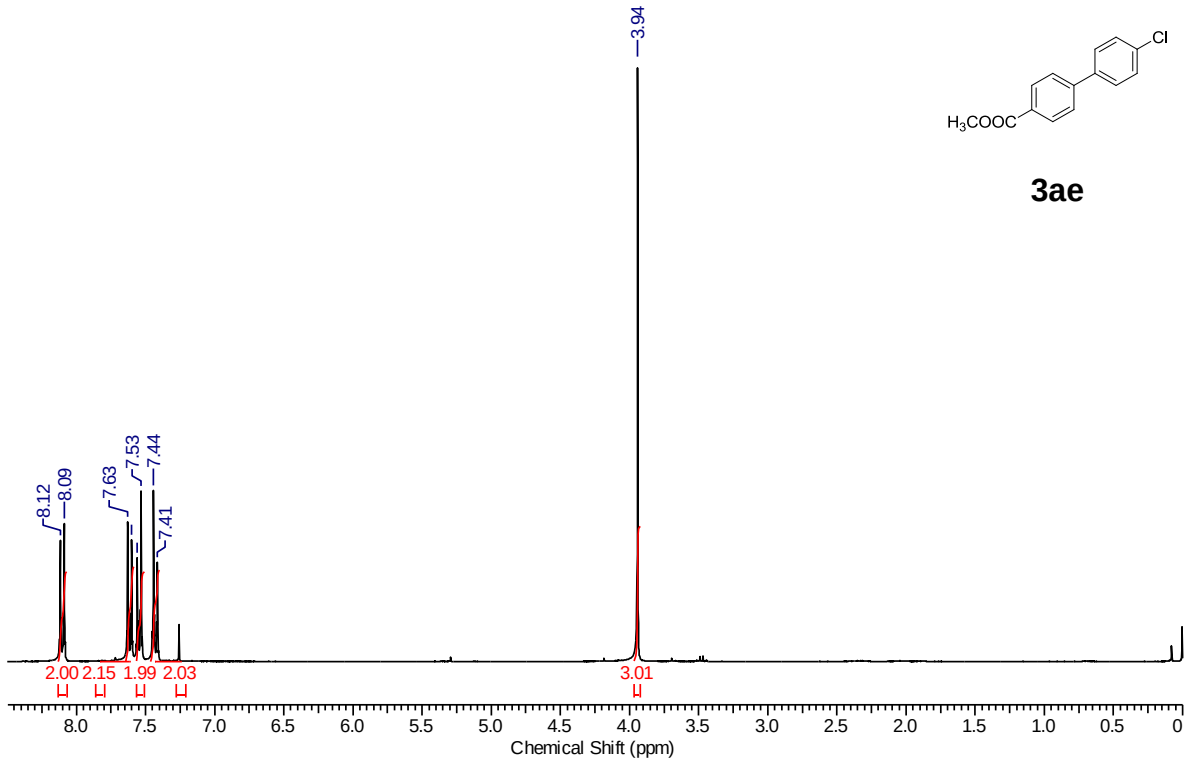

130.08
129.66
127.10
126.79
167.06
145.58
138.11
137.10
52.10
21.16
Chemical Shift (ppm)

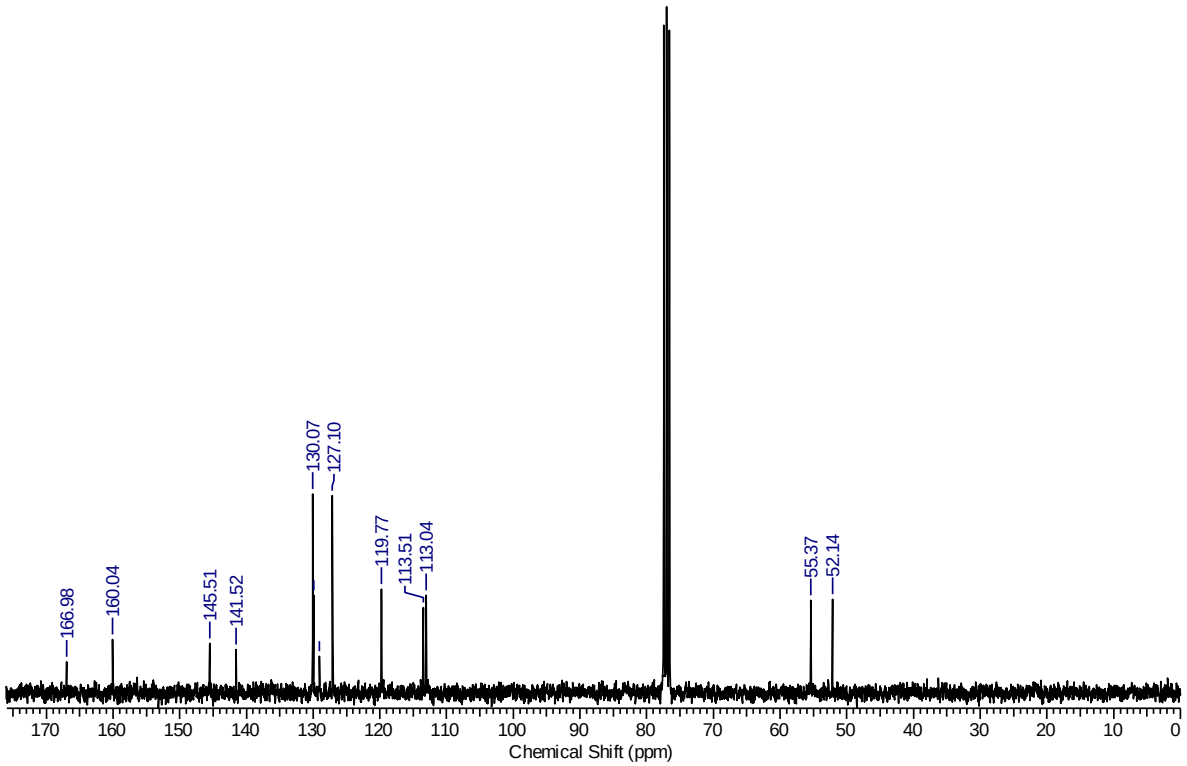

3.93
3.86
8.09
8.06
7.63
7.60
7.59
7.56
7.01
6.98
0.00
1.95
3.97
1.95
3.04
2.94
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
Chemical Shift (ppm)

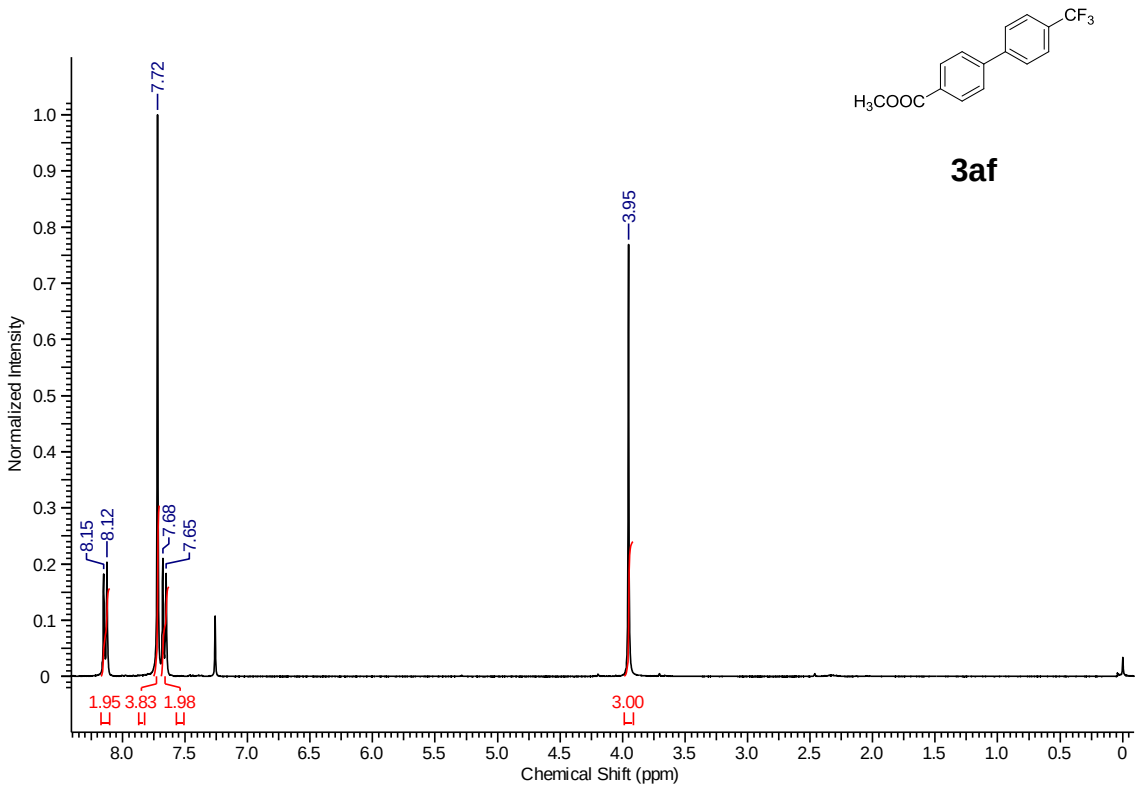
OCH3
H3COOC

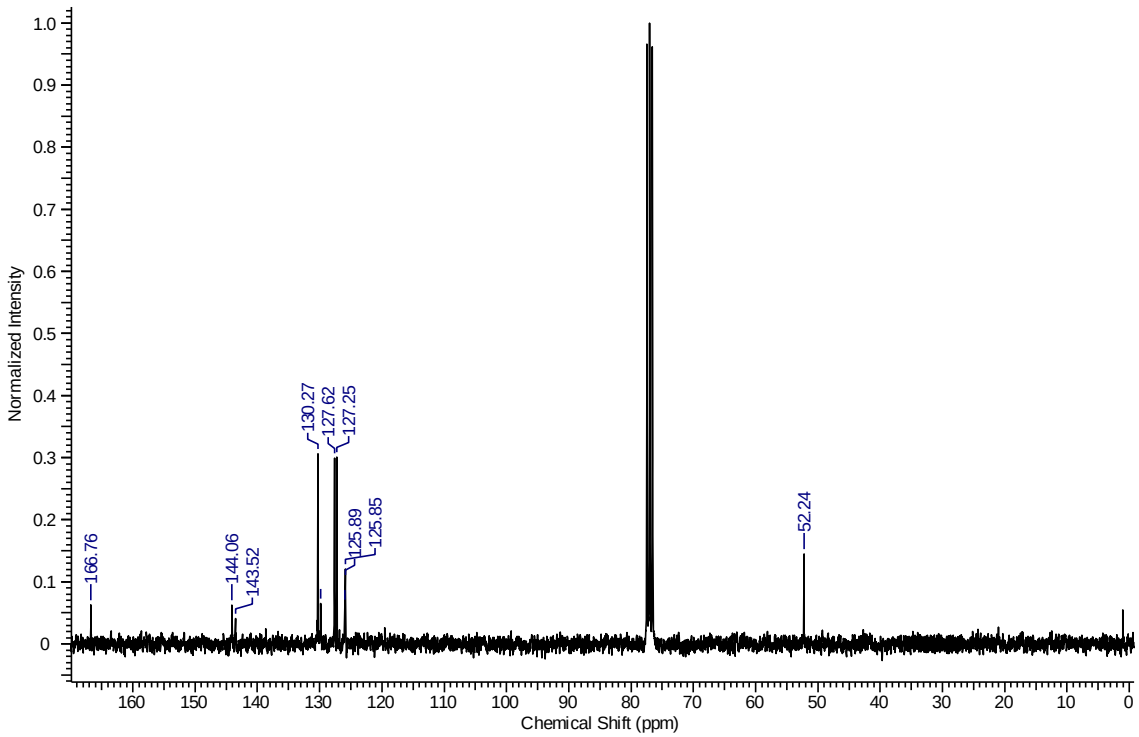
3ad

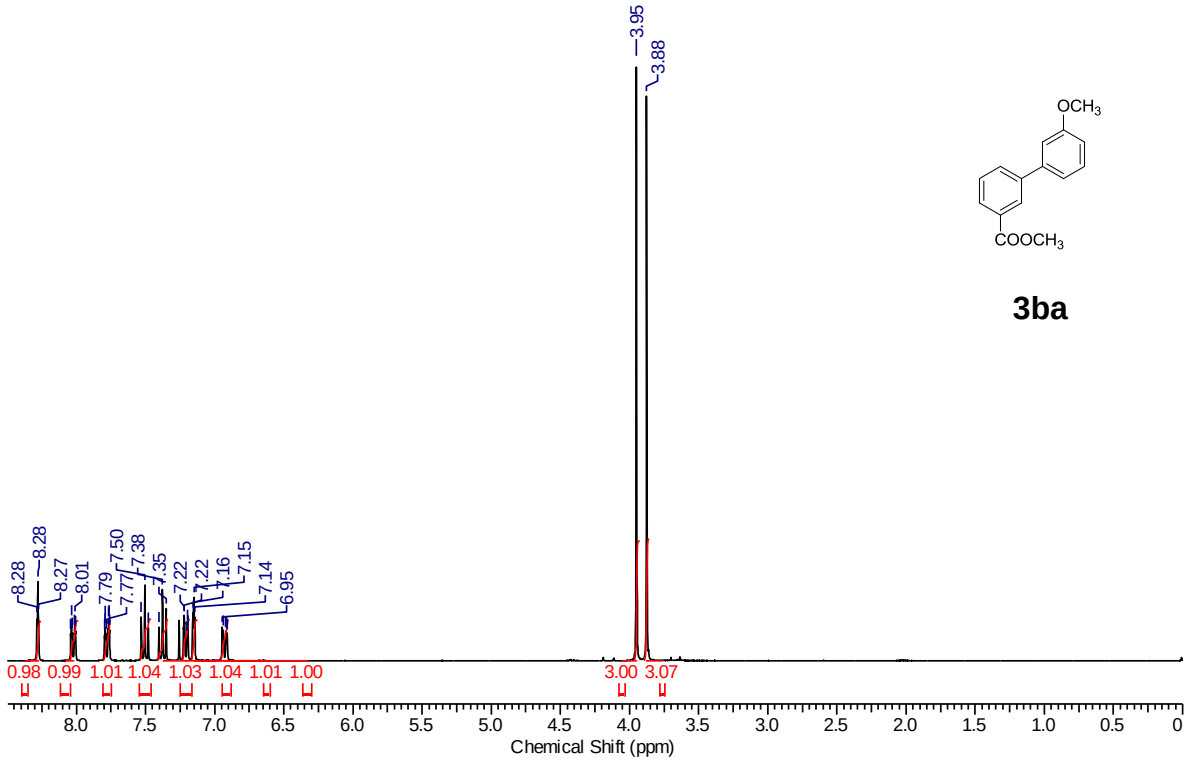

167.08
159.85
145.22
132.41
130.11
128.36
126.47
114.37
55.38
52.07
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)


3.94
Cl
H3COOC
3ae
8.12
8.09
7.63
7.53
7.44
7.41
2.00 2.15 1.99 2.03
3.01
Chemical Shift (ppm)

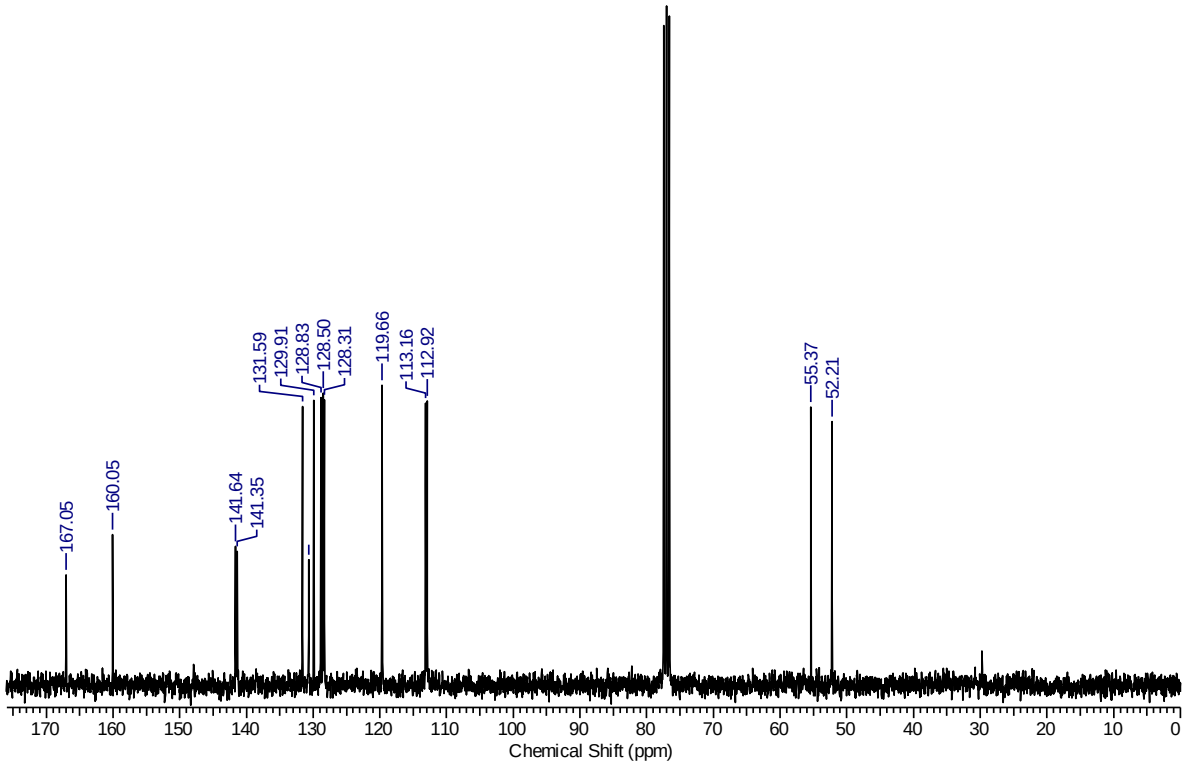

166.98
160.04
145.51
141.52
130.07
127.10
119.77
113.51
113.04
55.37
52.14
Chemical Shift (ppm)

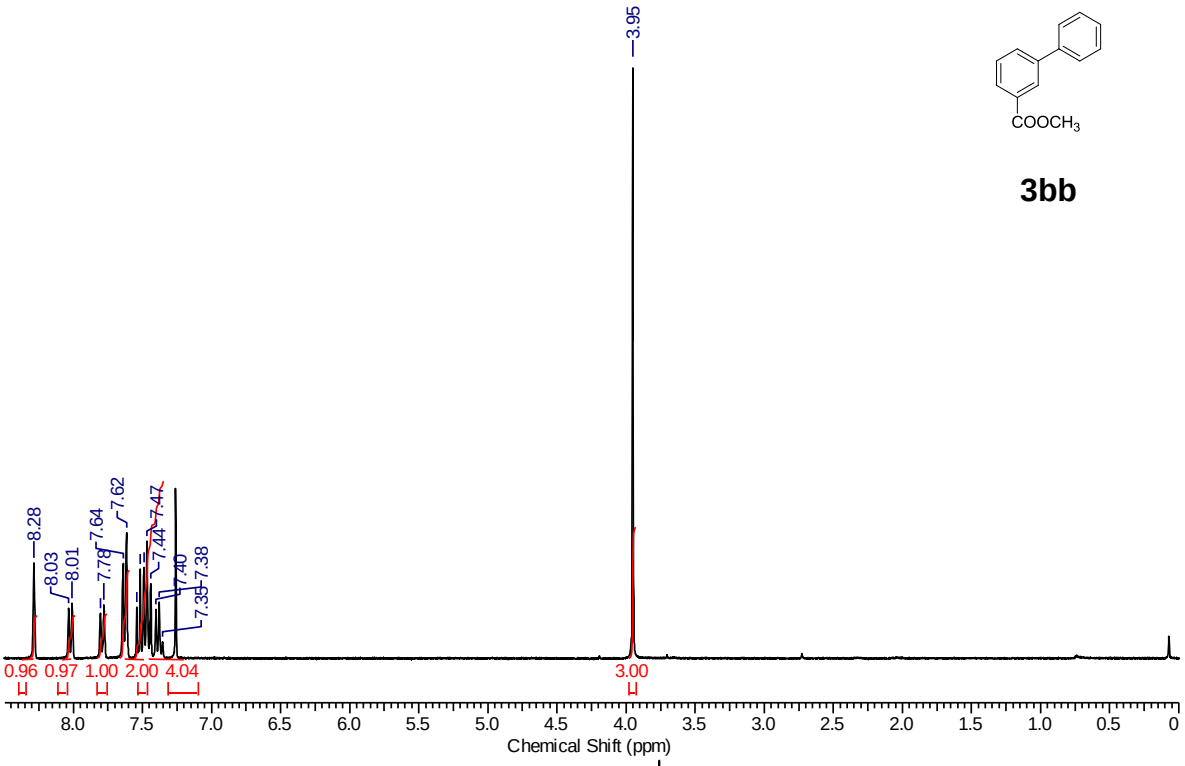

CF3
H3COOC
3af
7.72
3.95
8.15
8.12
7.68
7.65
1.95
3.83
1.98
3.00
Normalized Intensity
Chemical Shift (ppm)


166.76
144.06
143.52
130.27
127.62
127.25
125.89
125.85
52.24
Normalized Intensity
Chemical Shift (ppm)


3.95
3.88
OCH3
COOCH3
8.28
8.28
8.27
8.01
7.79
7.77
7.50
7.38
7.35
7.22
7.22
7.16
7.15
7.14
6.95
0.98 0.99 1.01 1.04 1.03 1.04 1.01 1.00
3.00 3.07
Chemical Shift (ppm)

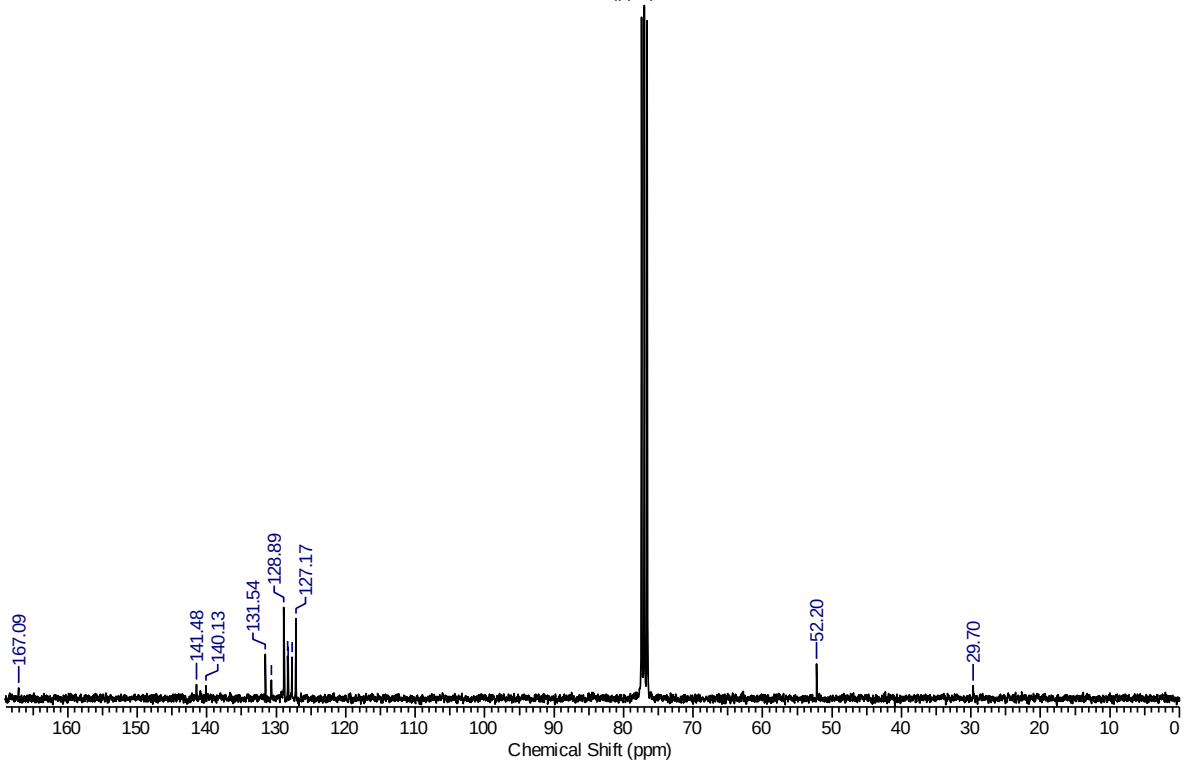
3ba

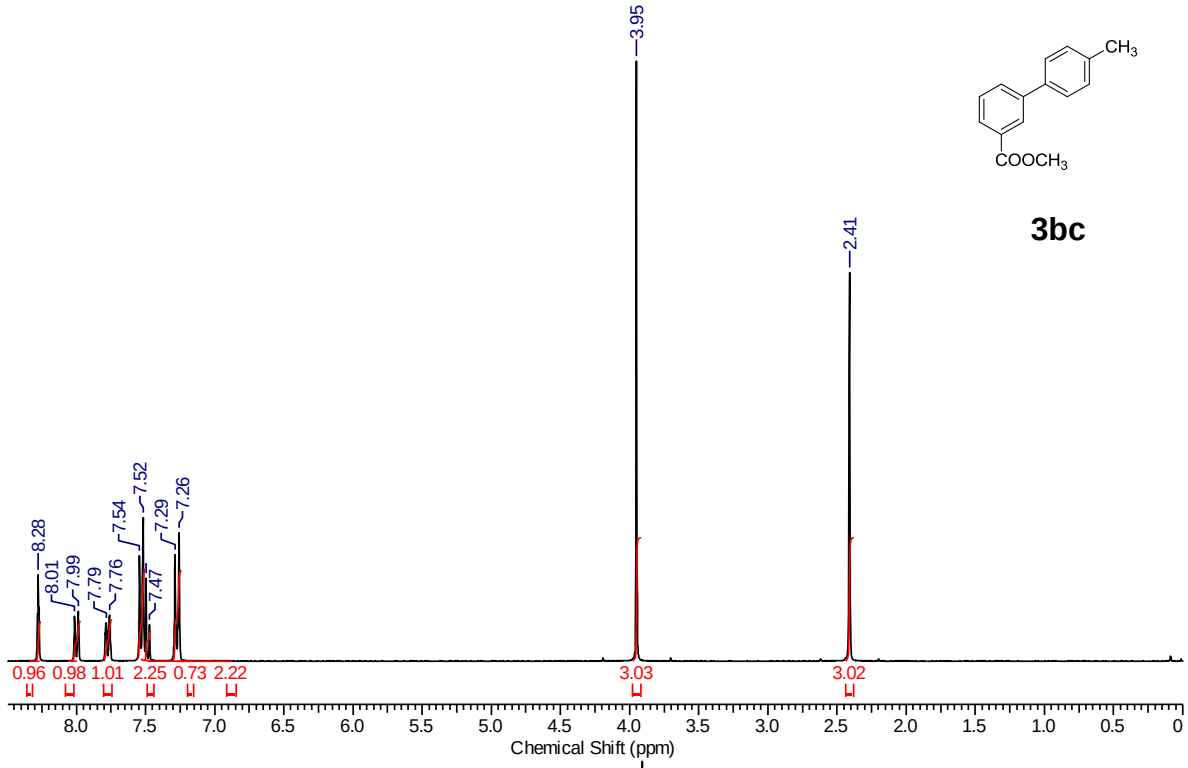

167.05
160.05
141.64
141.35
131.59
129.91
128.83
128.50
128.31
119.66
113.16
112.92
55.37
52.21
Chemical Shift (ppm)

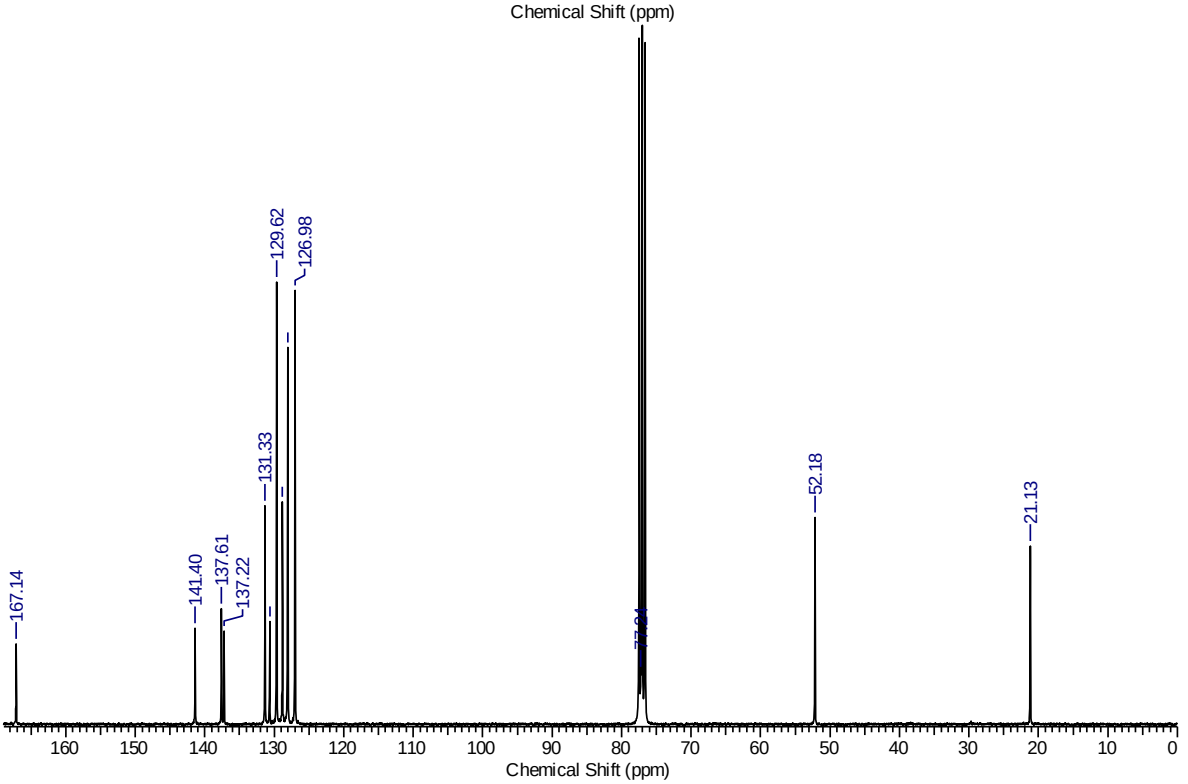

3.95
8.28
8.03
8.01
7.79
7.64
7.62
7.47
7.44
7.40
7.38
7.35
0.96 0.97 1.00 2.00 4.04
3.00
Chemical Shift (ppm)

COOCH3

3bb

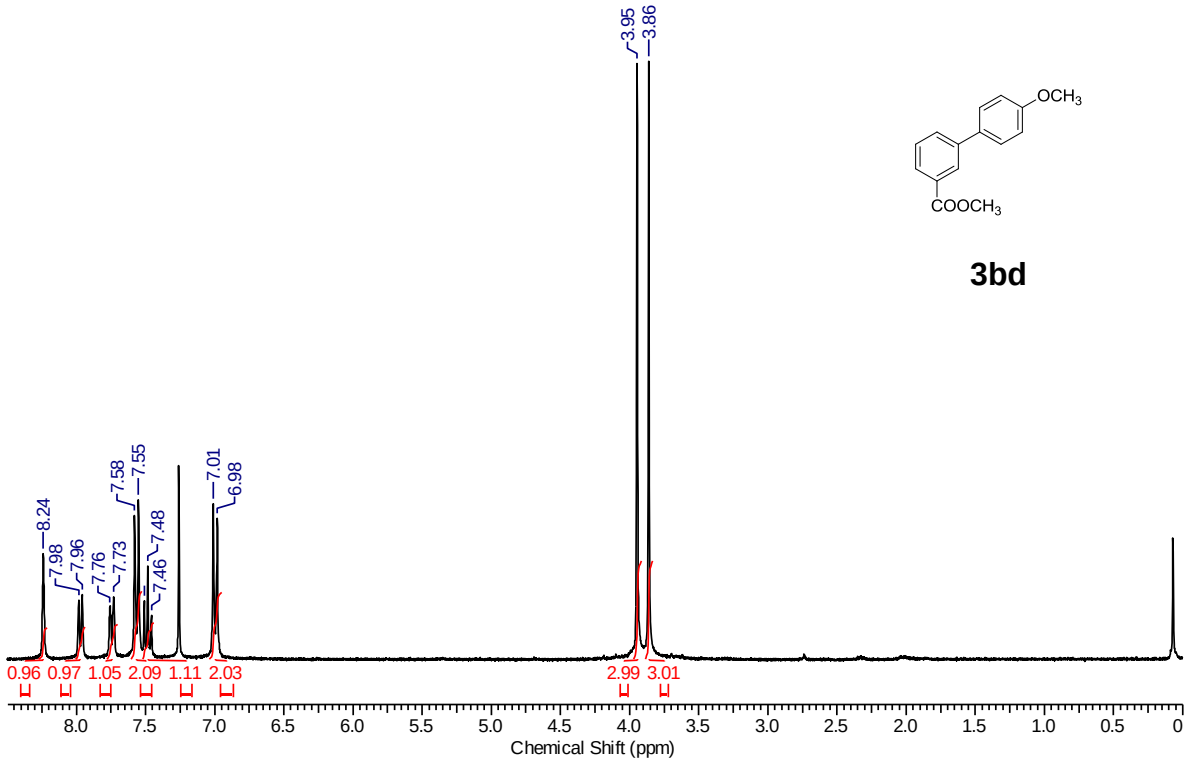

167.09
141.48
140.13
131.54
128.89
127.17
52.20
29.70
Chemical Shift (ppm)


3.95
2.41
8.28
8.01
7.99
7.79
7.76
7.54
7.52
7.47
7.29
7.26
0.96 0.98 1.01 2.25 0.73 2.22
3.03
3.02
Chemical Shift (ppm)


167.14
141.40
137.61
137.22
131.33
129.62
126.98
77.24
52.18
21.13
Chemical Shift (ppm)

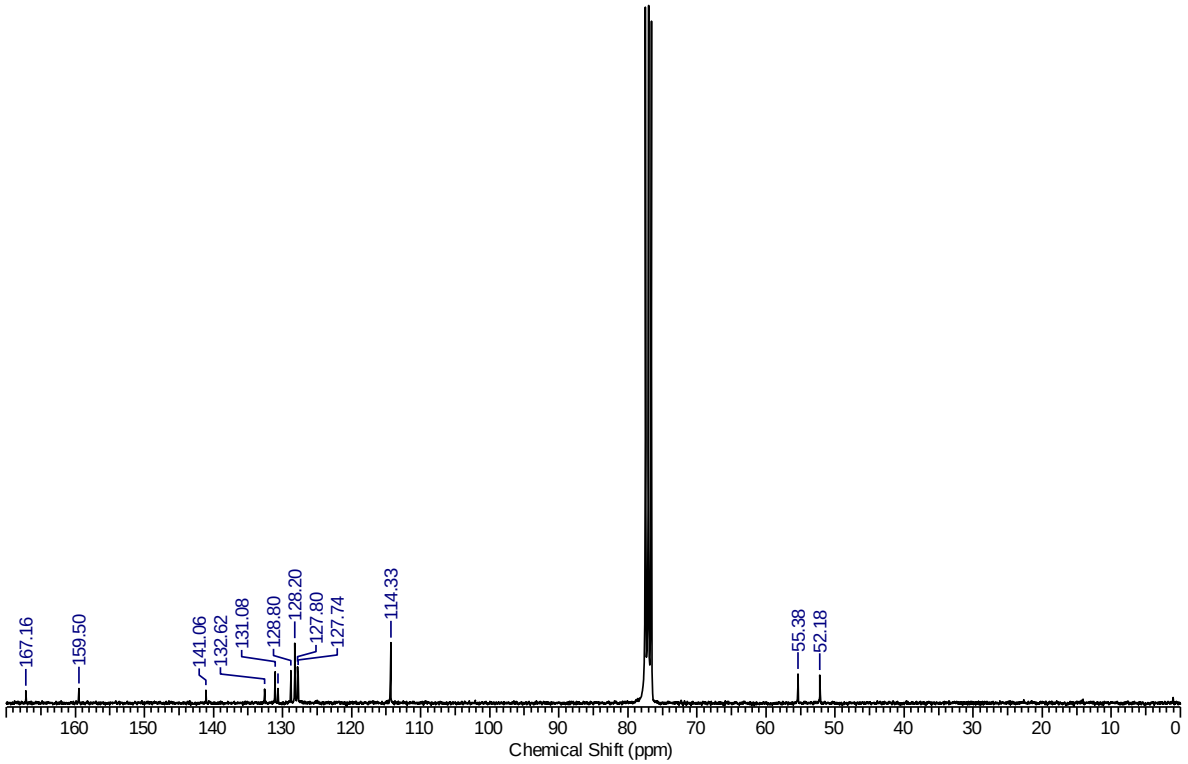
CH3
COOCH3

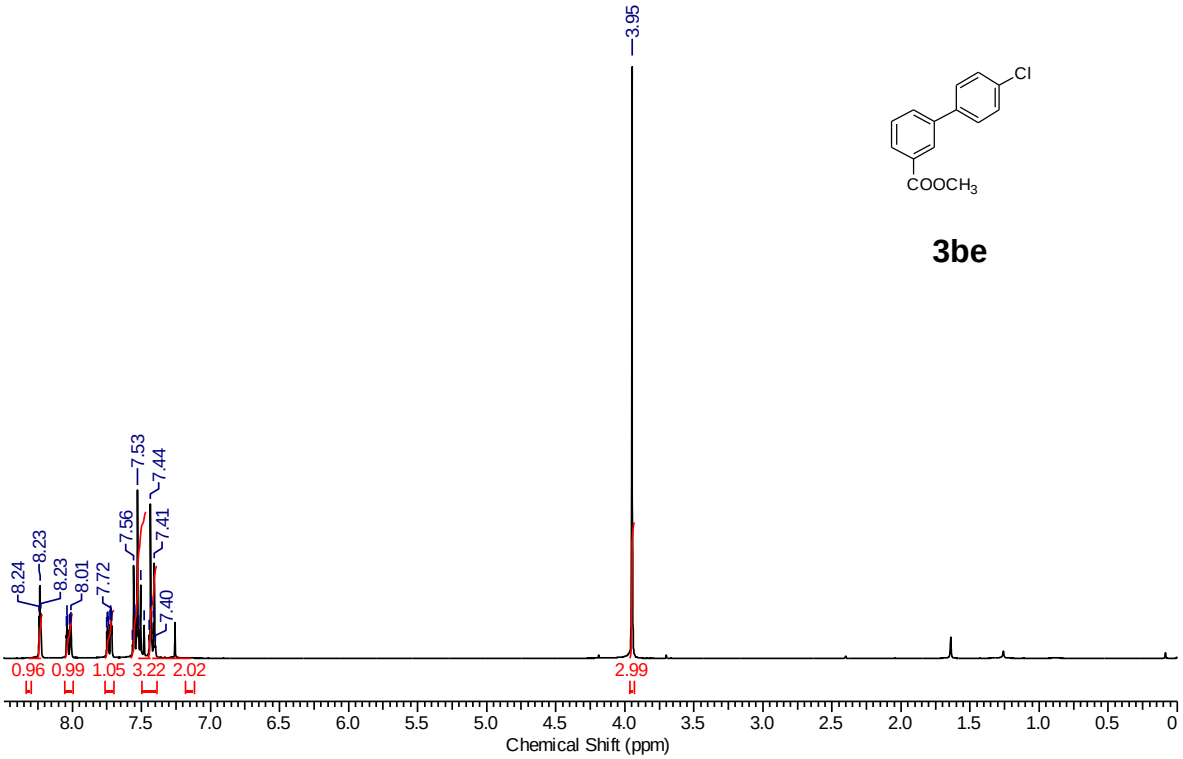
3bc

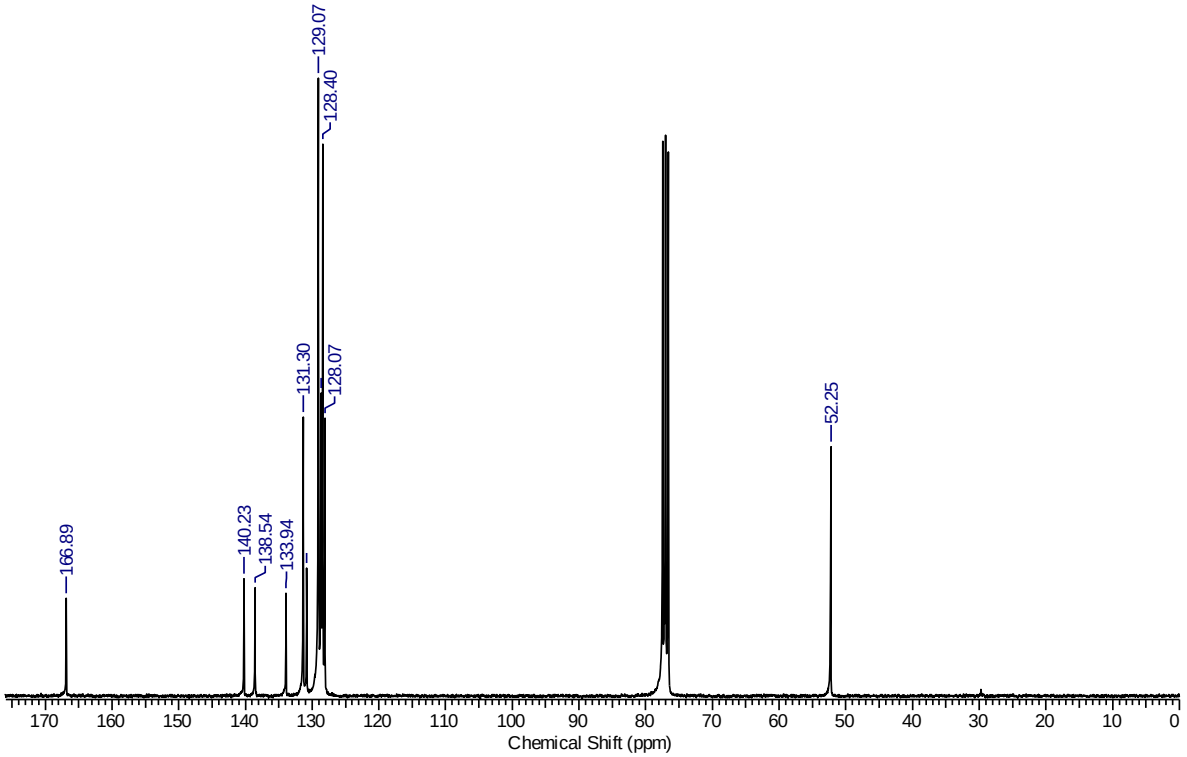

3.95
3.86
8.24
7.98
7.96
7.76
7.73
7.58
7.55
7.48
7.46
7.01
6.98
0.96 0.97 1.05 2.09 1.11 2.03
2.99 3.01
Chemical Shift (ppm)

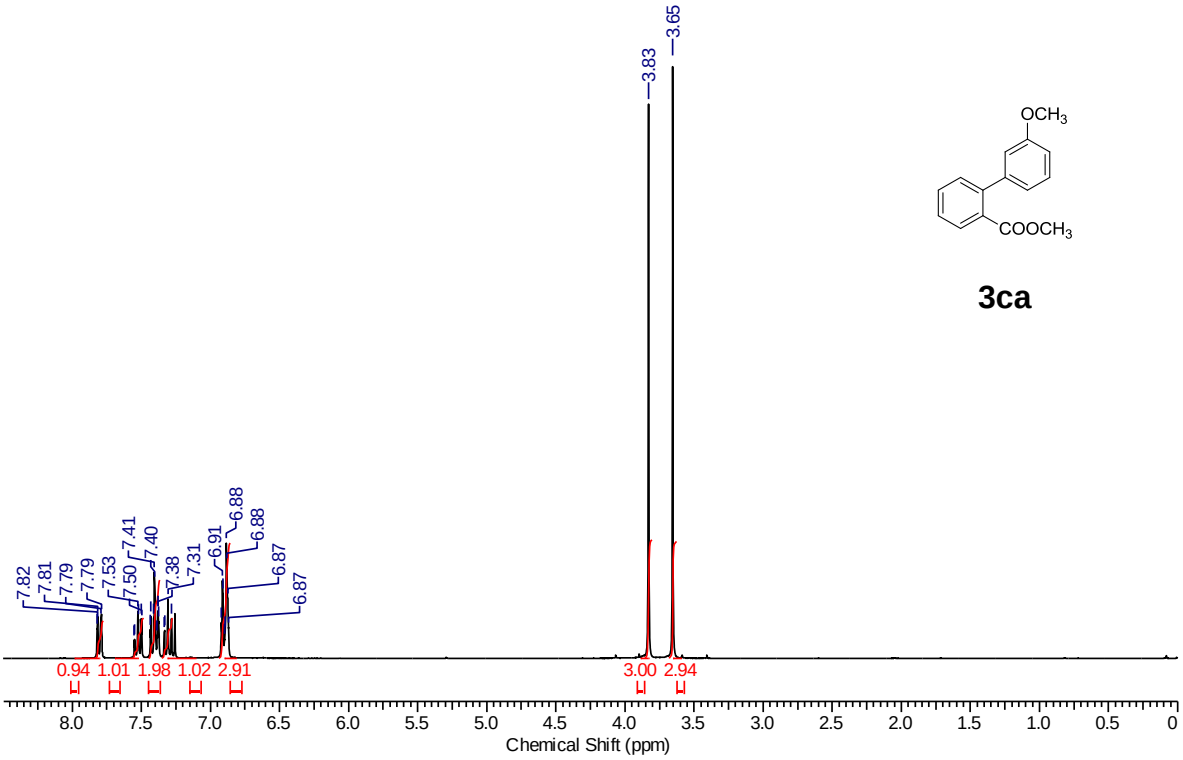
OCH3
COOCH3

3bd


167.16
159.50
141.06
132.62
131.08
128.80
128.20
127.80
127.74
114.33
55.38
52.18
Chemical Shift (ppm)

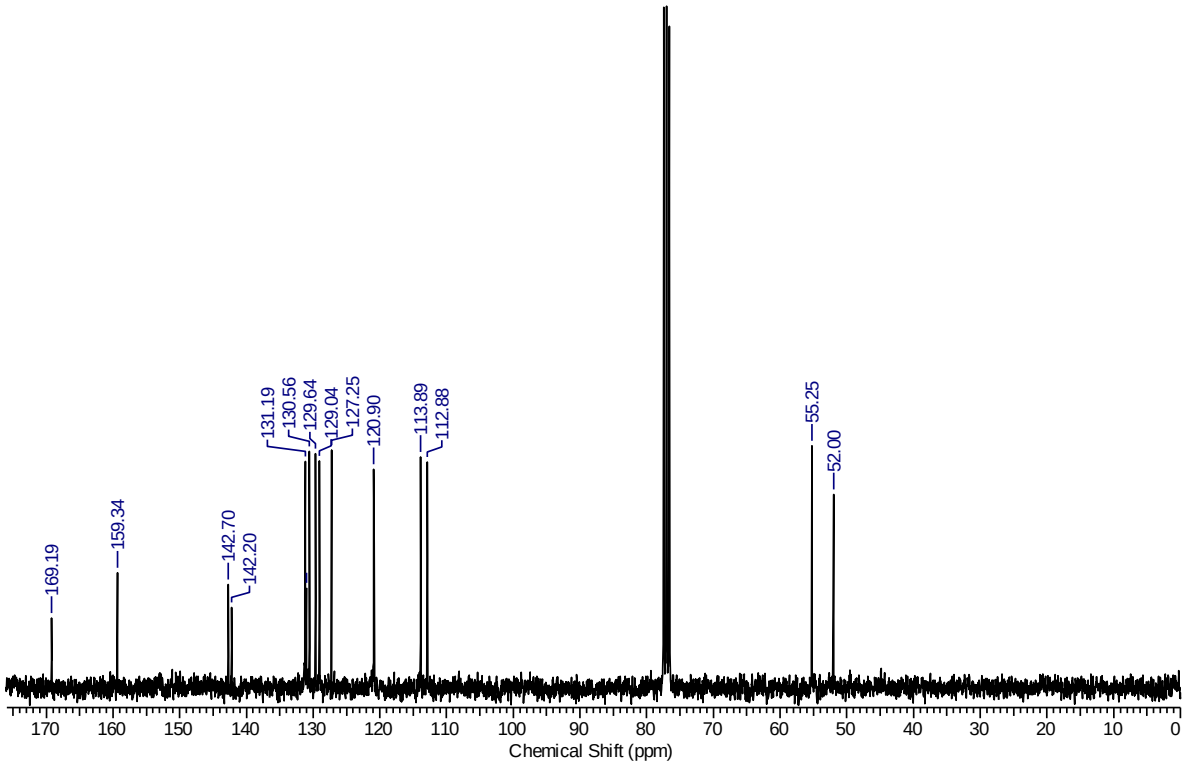

3.95
Cl
COOCH3
3be
8.24
8.23
8.23
8.01
7.72
7.56
7.53
7.44
7.41
7.40
0.96 0.99 1.05 3.22 2.02
2.99
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0
Chemical Shift (ppm)

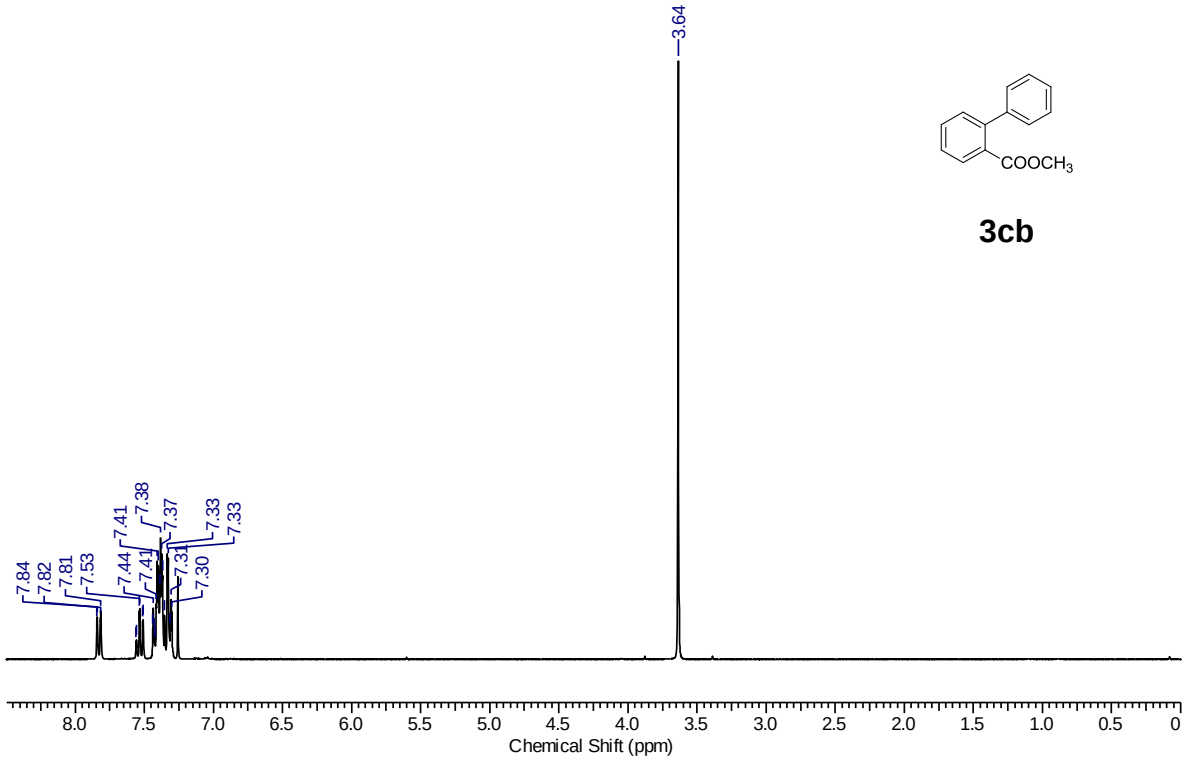

129.07
128.40
131.30
128.07
52.25
166.89
140.23
138.54
133.94
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
Chemical Shift (ppm)


3.83
3.65
7.82
7.81
7.79
7.79
7.53
7.50
7.41
7.40
7.38
7.31
6.91
6.88
6.88
6.87
6.87
0.94
1.01
1.98
1.02
2.91
3.00
2.94
Chemical Shift (ppm)

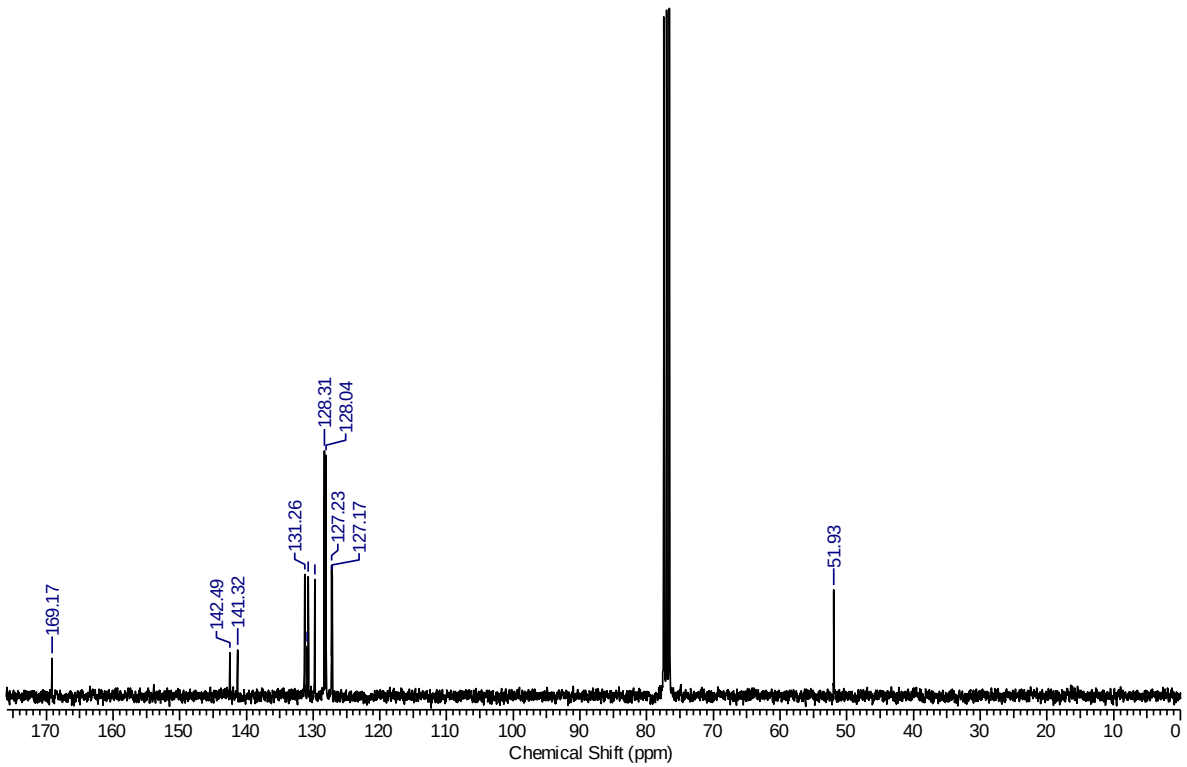
OCH3
COOCH3

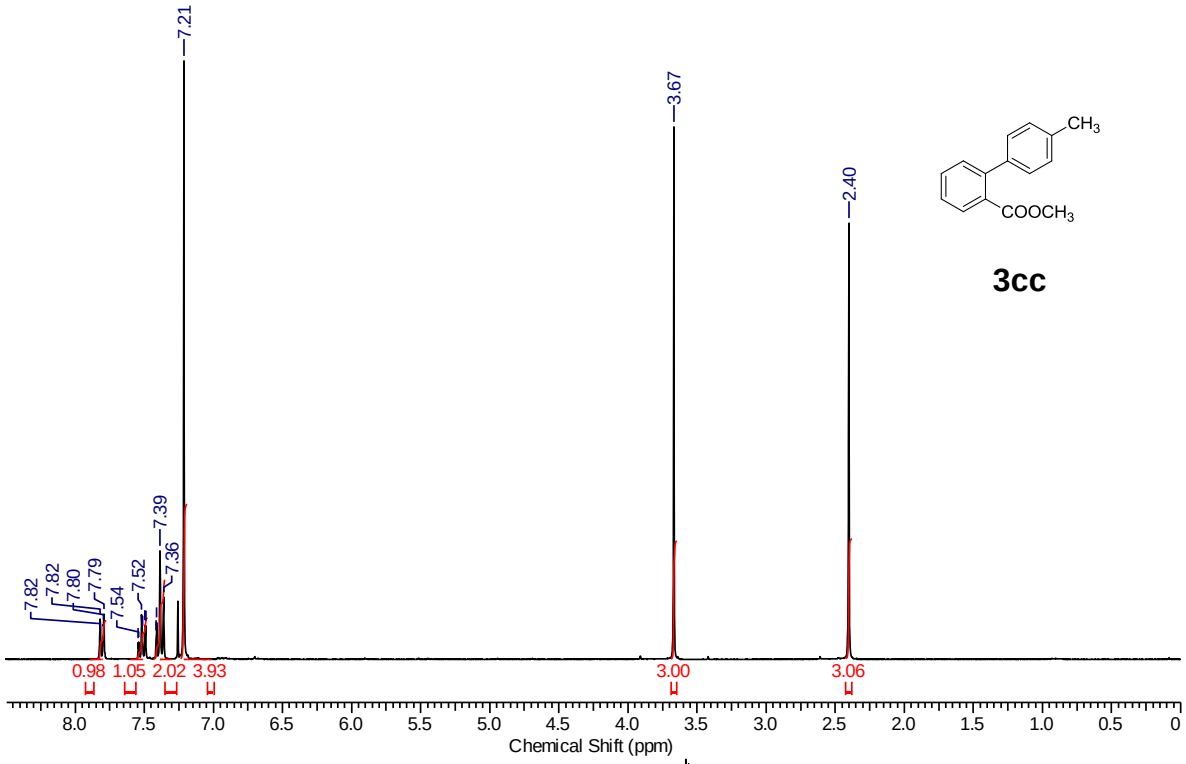
3ca


169.19
159.34
142.70
142.20
131.19
130.56
129.64
129.04
127.25
120.90
113.89
112.88
55.25
52.00
Chemical Shift (ppm)


3.64
7.84
7.82
7.81
7.53
7.44
7.41
7.41
7.38
7.37
7.33
7.33
7.31
7.30
COOCH3
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
Chemical Shift (ppm)

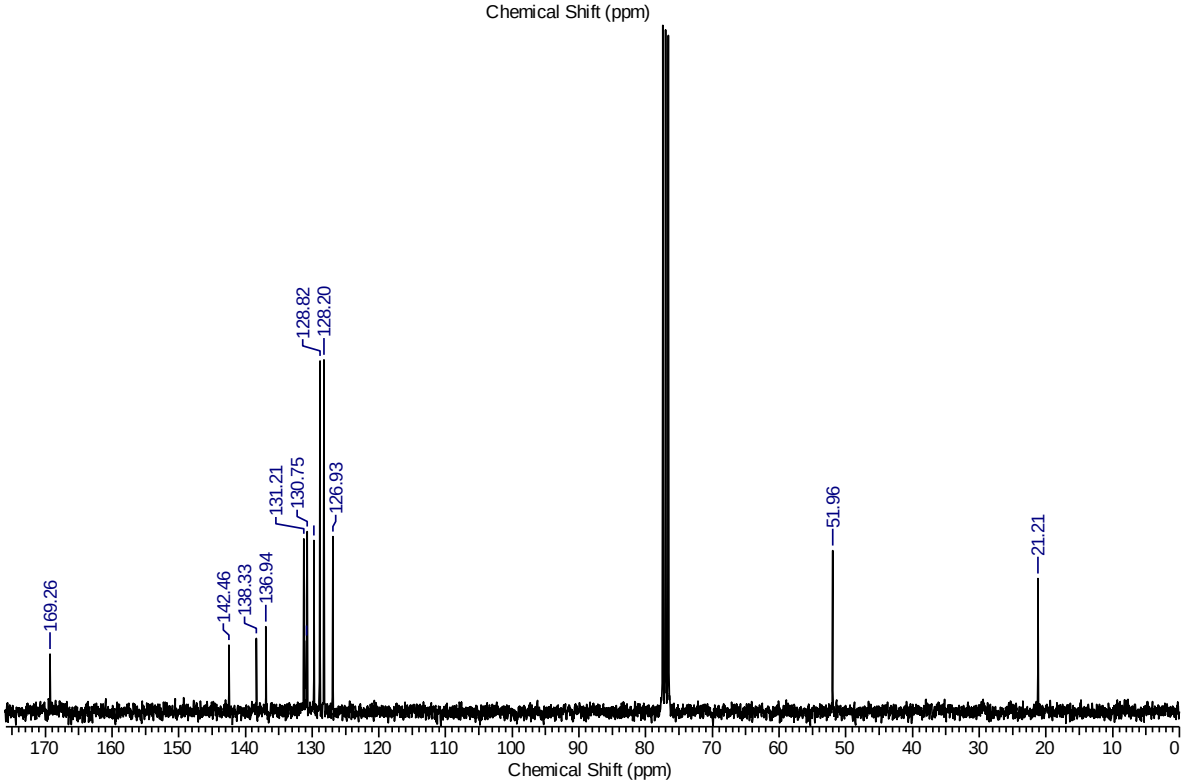
3cb

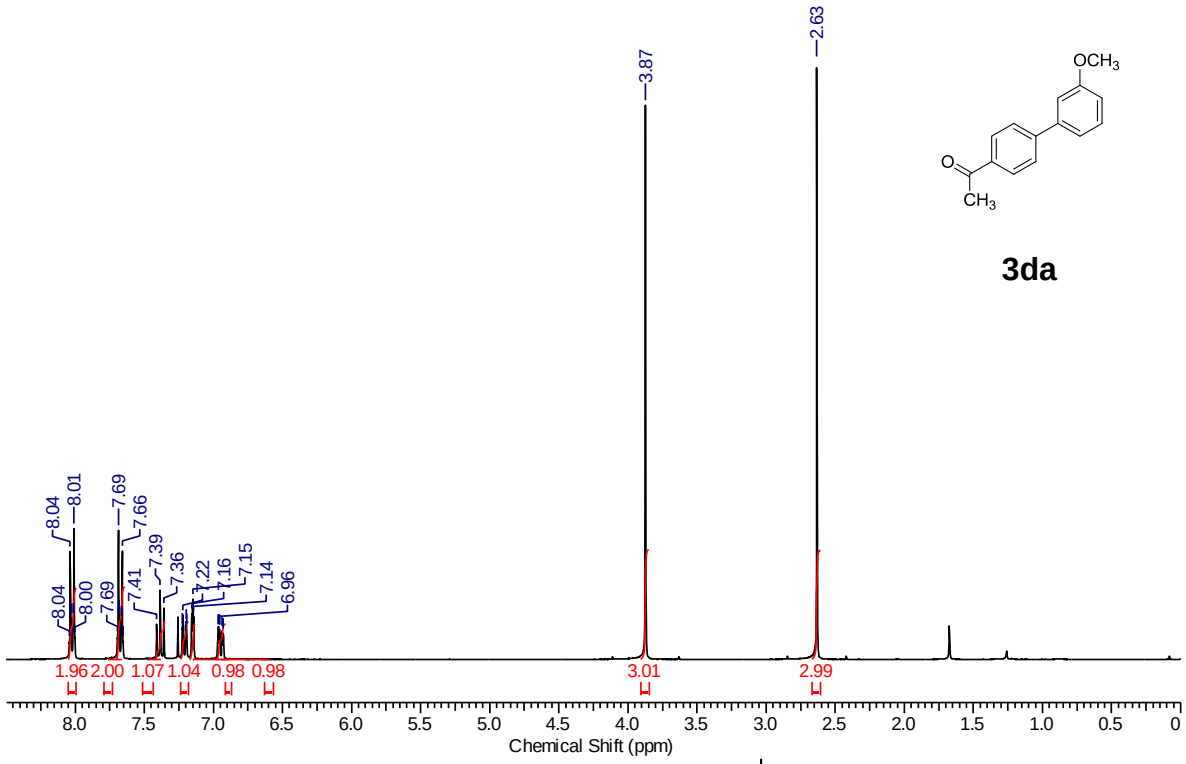

169.17
142.49
141.32
131.26
128.31
128.04
127.23
127.17
51.93
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

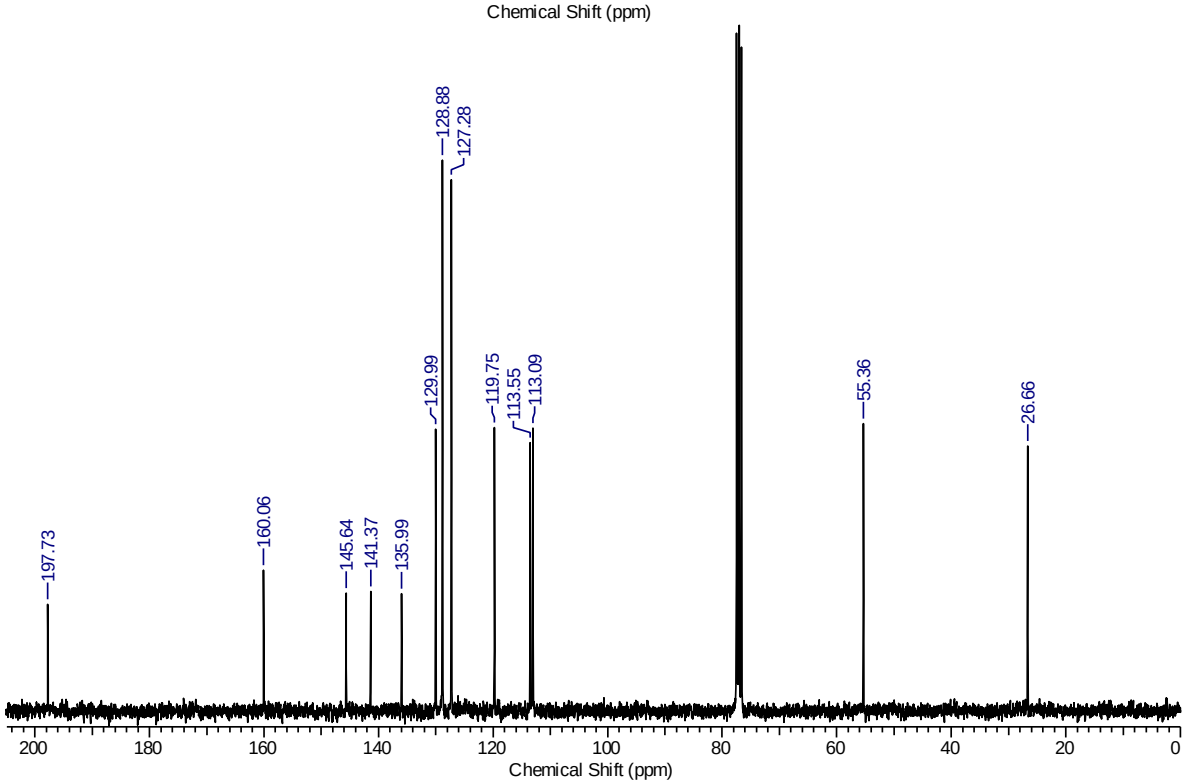

7.21
3.67
2.40
7.82
7.82
7.80
7.79
7.54
7.52
7.39
7.36
0.98 1.05 2.02 3.93
3.00
3.06
Chemical Shift (ppm)

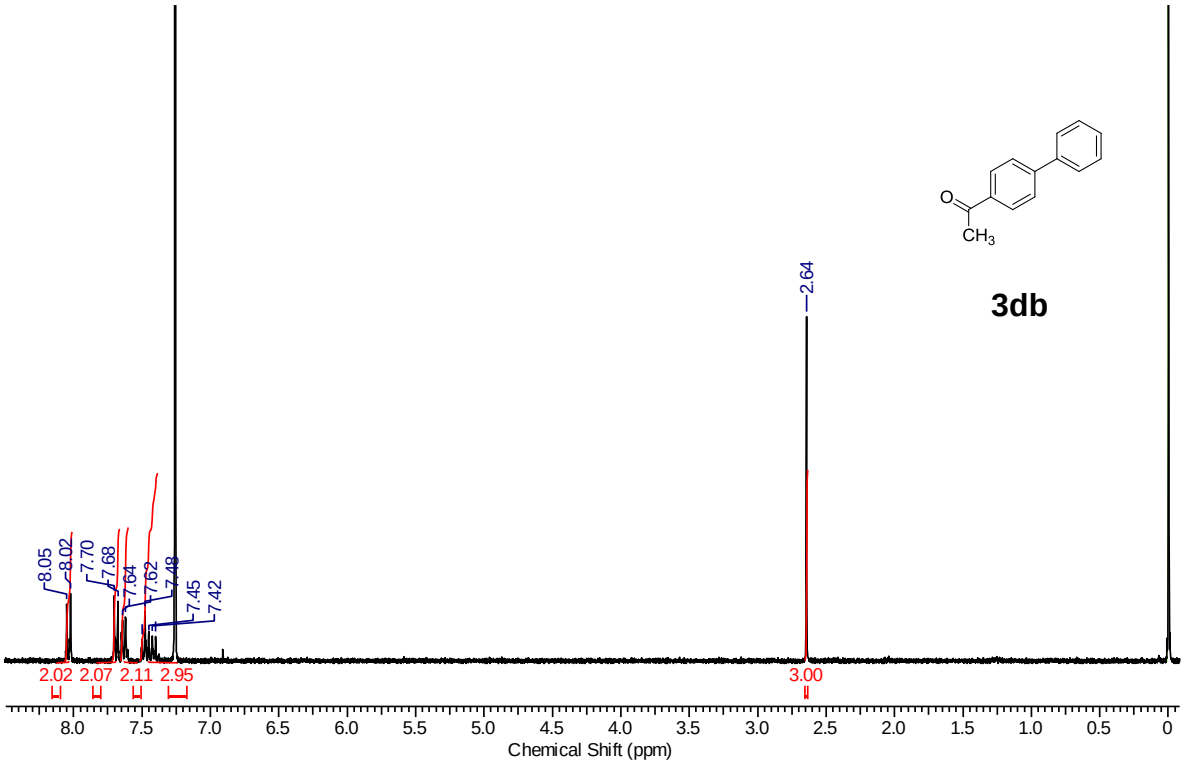
CH3
COOCH3

3cc

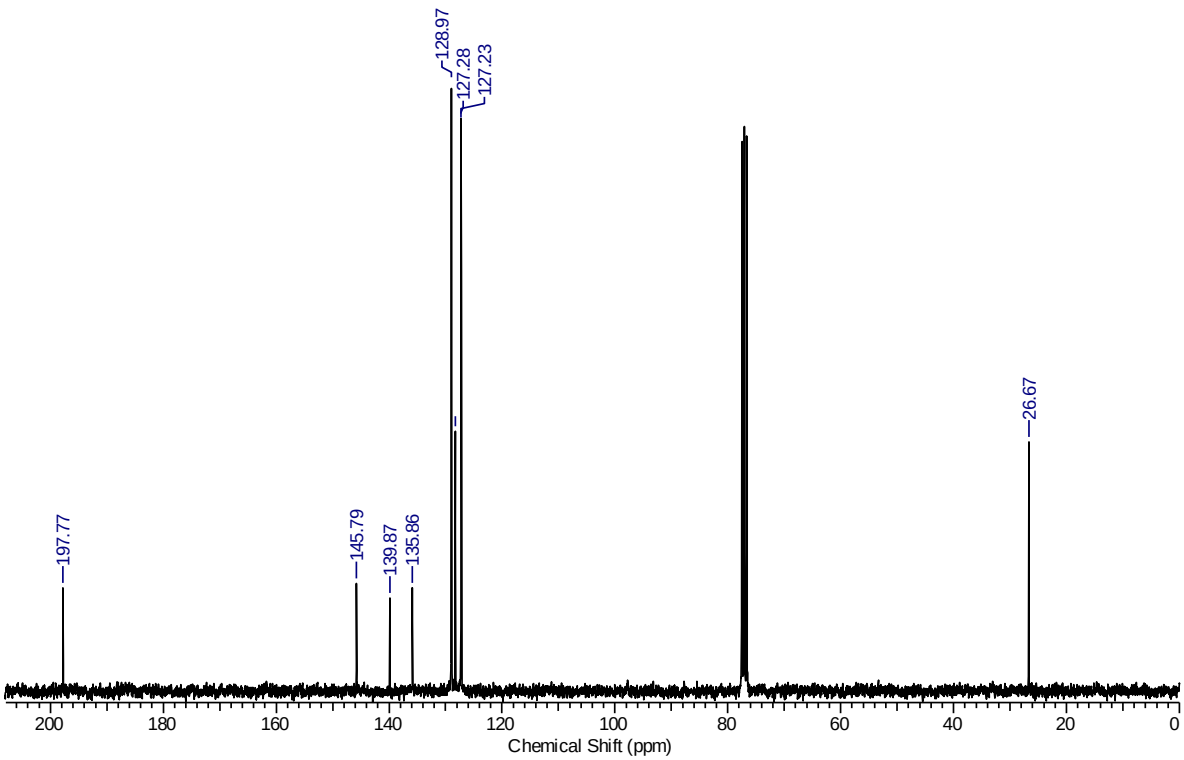

169.26
142.46
138.33
136.94
131.21
130.75
128.82
128.20
126.93
51.96
21.21
Chemical Shift (ppm)

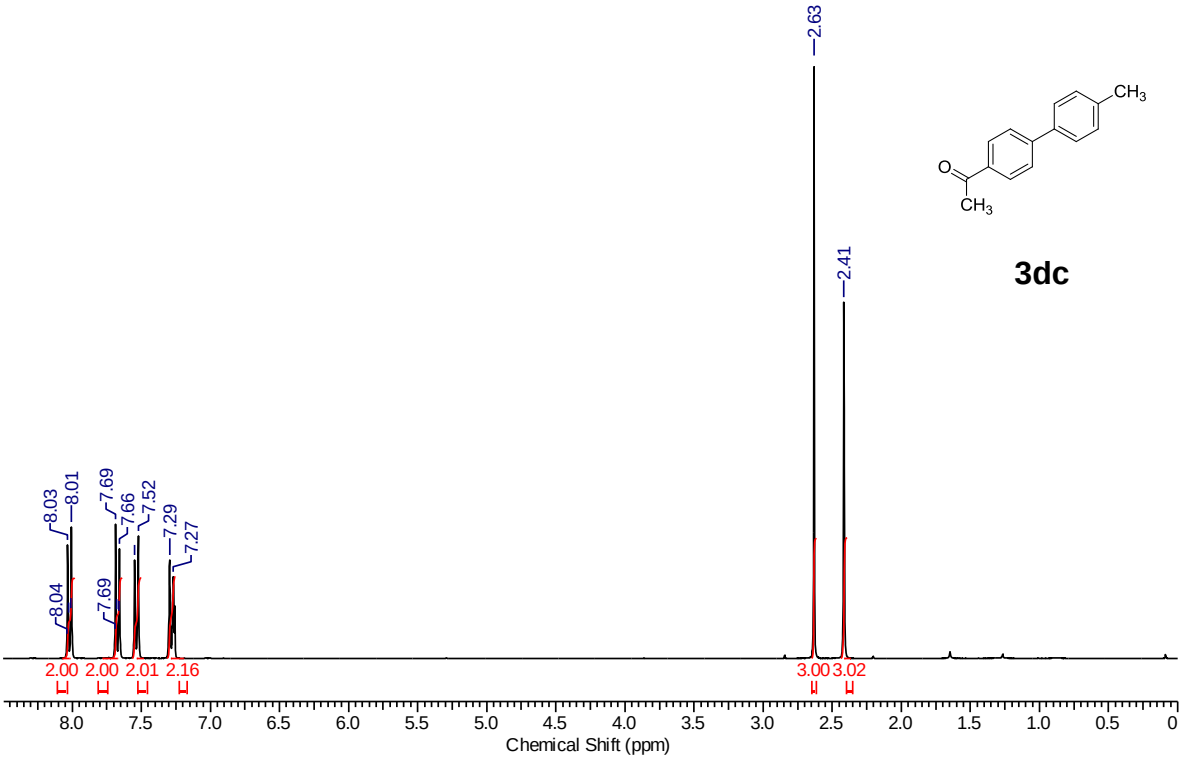

2.63
3.87
OCH3
O
CH3
3da
8.04
8.01
7.69
7.66
7.39
7.41
7.36
7.22
7.16
7.15
7.14
6.96
8.04
8.00
7.69
1.96 2.00 1.07 1.04 0.98 0.98
3.01
2.99
Chemical Shift (ppm)


128.88
127.28
129.99
119.75
113.55
113.09
55.36
26.66
197.73
160.06
145.64
141.37
135.99
Chemical Shift (ppm)


8.05
8.02
7.70
7.68
7.64
7.62
7.48
7.45
7.42
2.64
2.02 2.07 2.11 2.95
3.00
Chemical Shift (ppm)

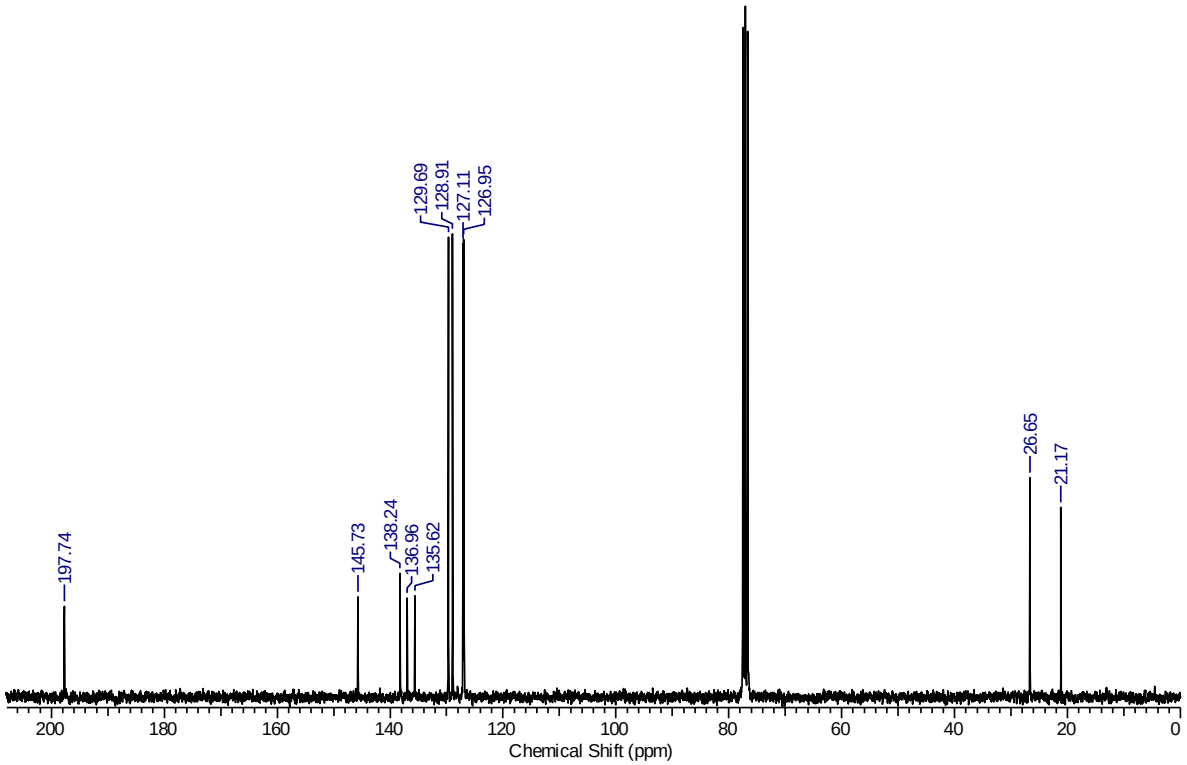
3db

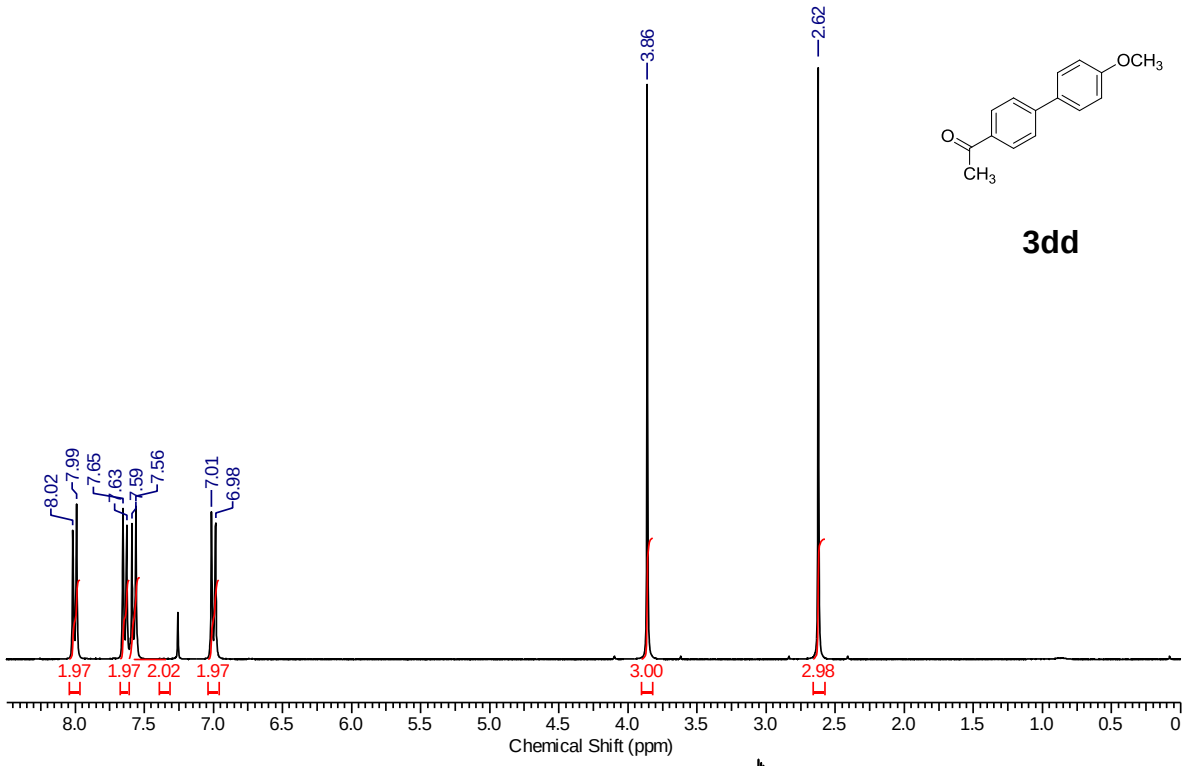

128.97
127.28
127.23
197.77
145.79
139.87
135.86
26.67
Chemical Shift (ppm)

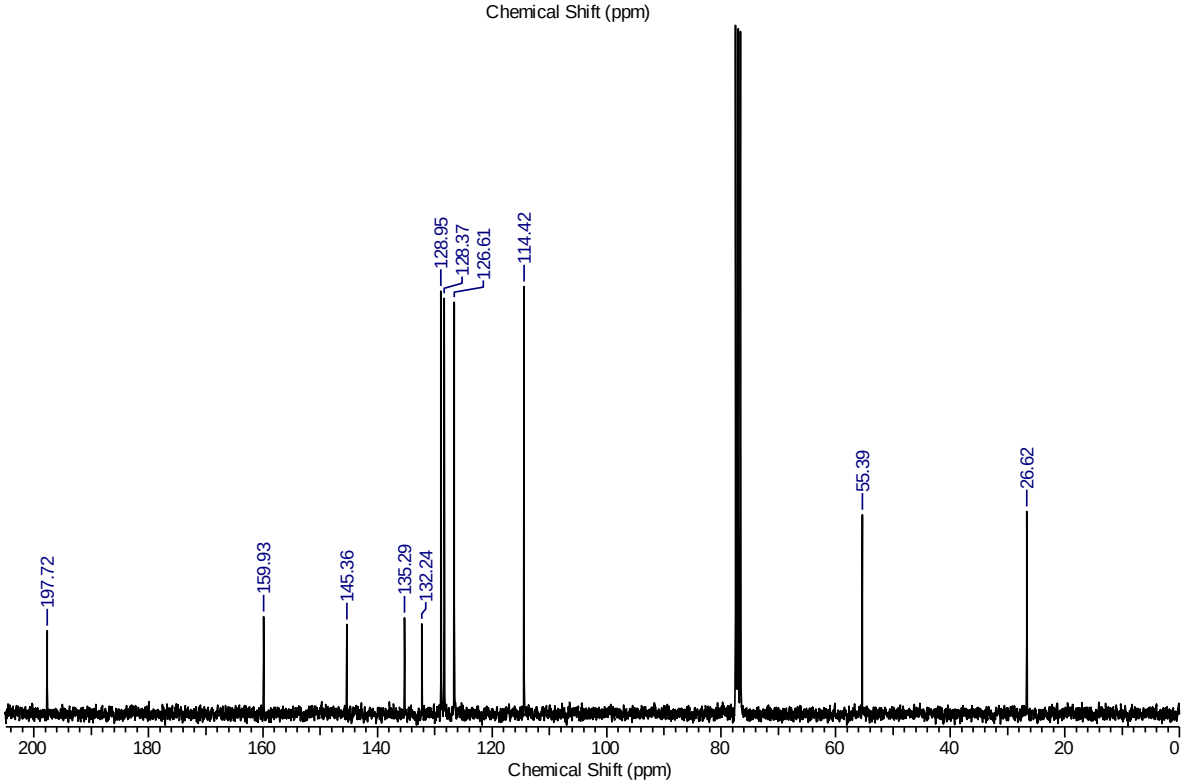

2.63
2.41
8.03
8.01
7.69
7.66
7.52
7.29
7.27
8.04
7.69
2.00 2.00 2.01 2.16
3.00 3.02
Chemical Shift (ppm)

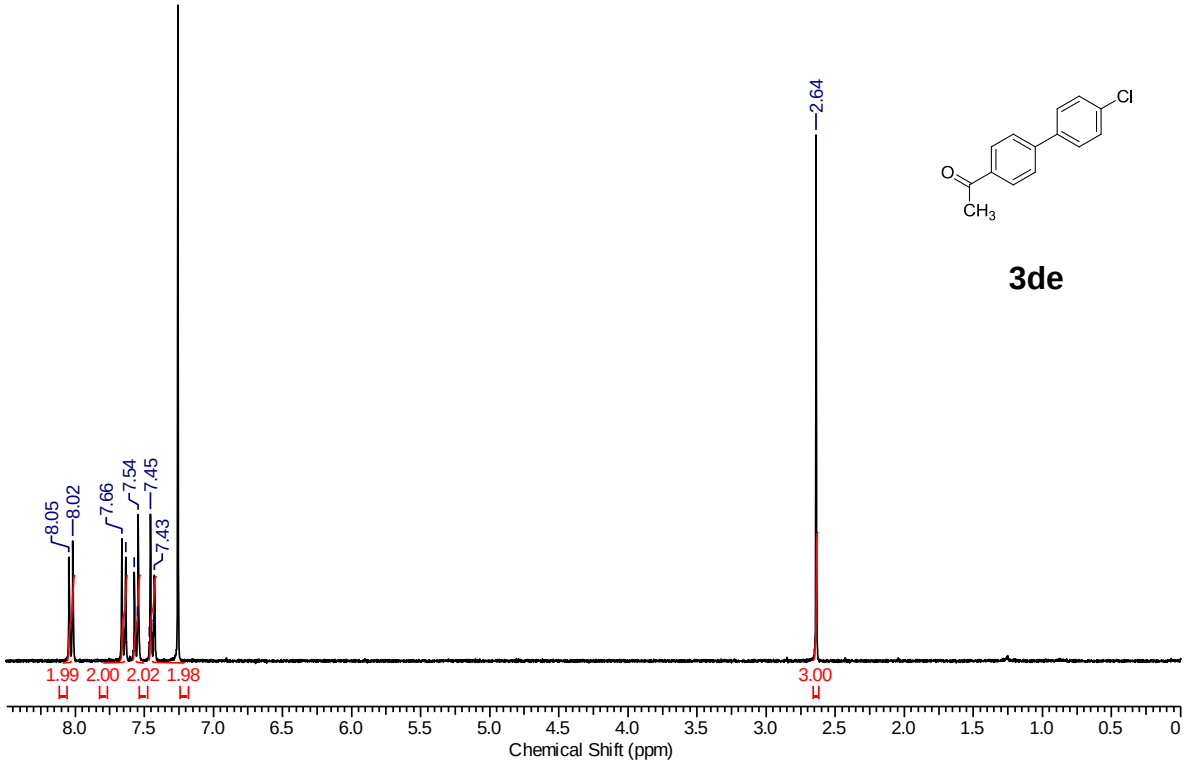
CH3
O
CH3

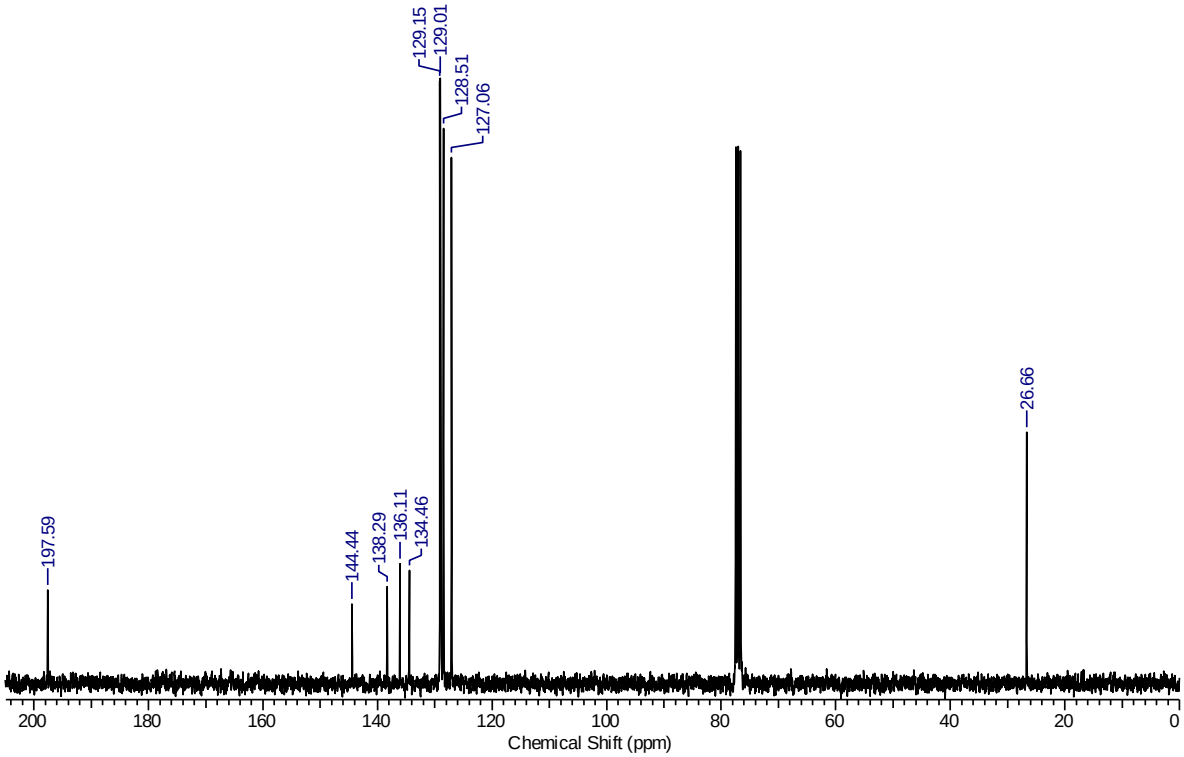
3dc

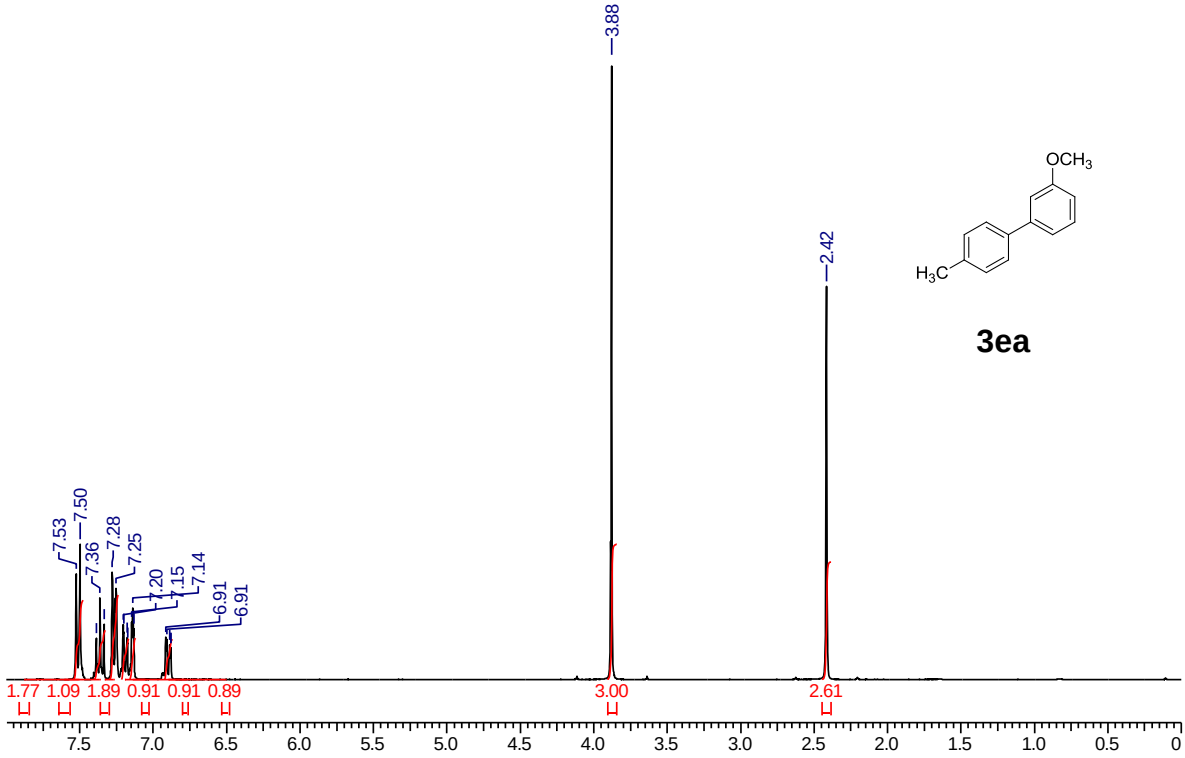

129.69
128.91
127.11
126.95
197.74
145.73
138.24
136.96
135.62
26.65
21.17
Chemical Shift (ppm)


3.86
2.62
OCH3
O
CH3
3dd
8.02
7.99
7.65
7.63
7.59
7.56
7.01
6.98
1.97
1.97
2.02
1.97
3.00
2.98
Chemical Shift (ppm)


128.95
128.37
126.61
114.42
55.39
26.62
197.72
159.93
145.36
135.29
132.24
Chemical Shift (ppm)

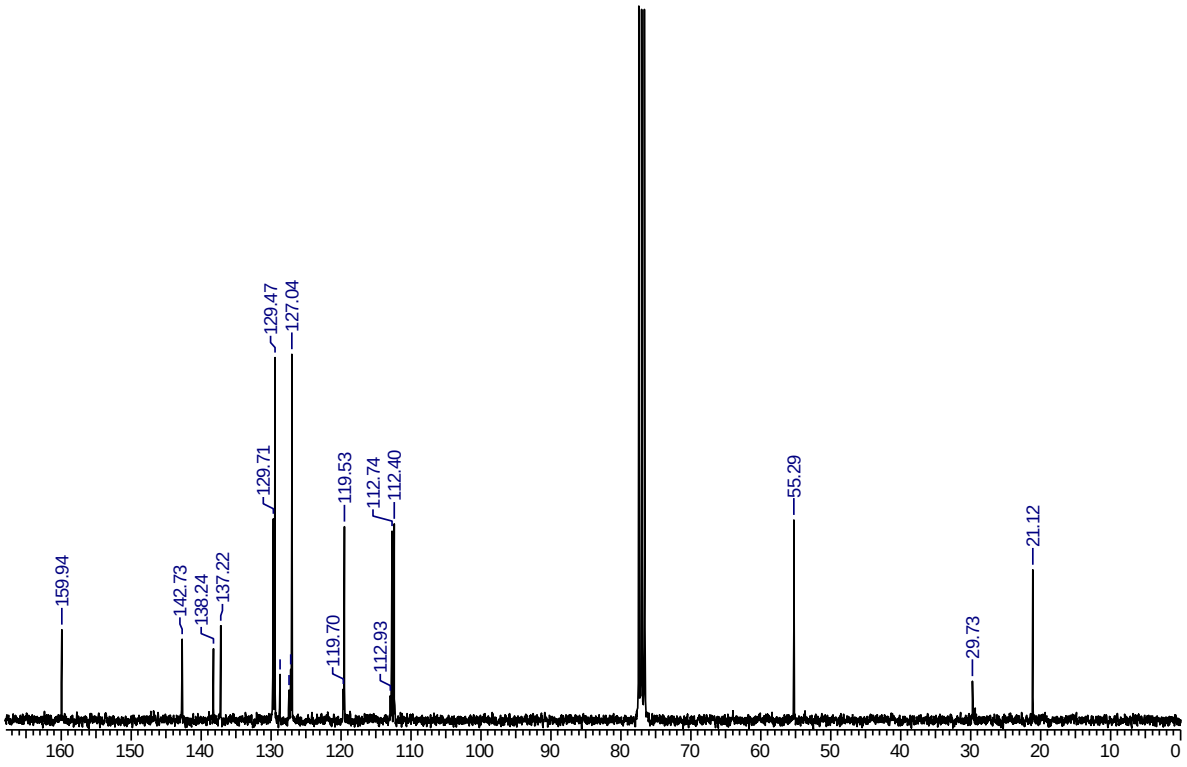

8.05
8.02
7.66
7.54
7.45
7.43
2.64
1.99
2.00
2.02
1.98
3.00
Cl
O
CH3
3de
Chemical Shift (ppm)

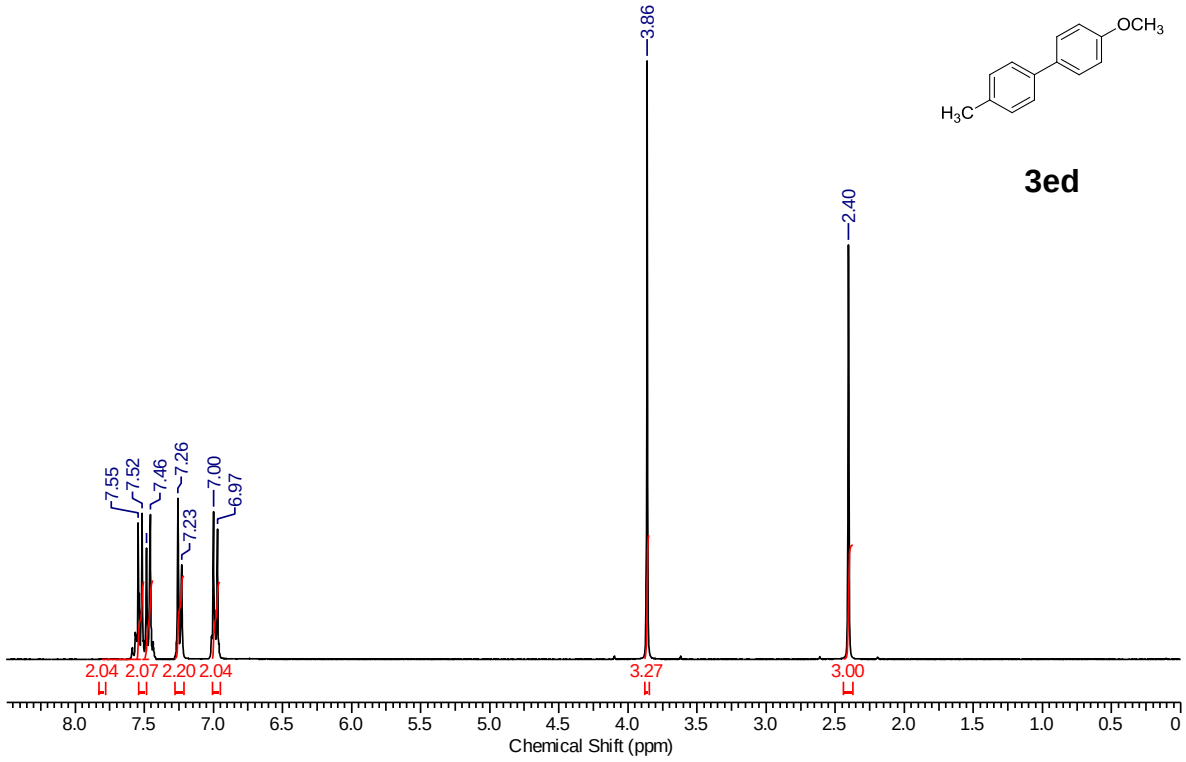

129.15
129.01
128.51
127.06
197.59
144.44
138.29
136.11
134.46
26.66
Chemical Shift (ppm)


3.88
2.42
7.53
7.50
7.36
7.28
7.25
7.20
7.15
7.14
6.91
6.91
1.77 1.09 1.89 0.91 0.91 0.89
3.00
2.61
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

OCH3
H3C

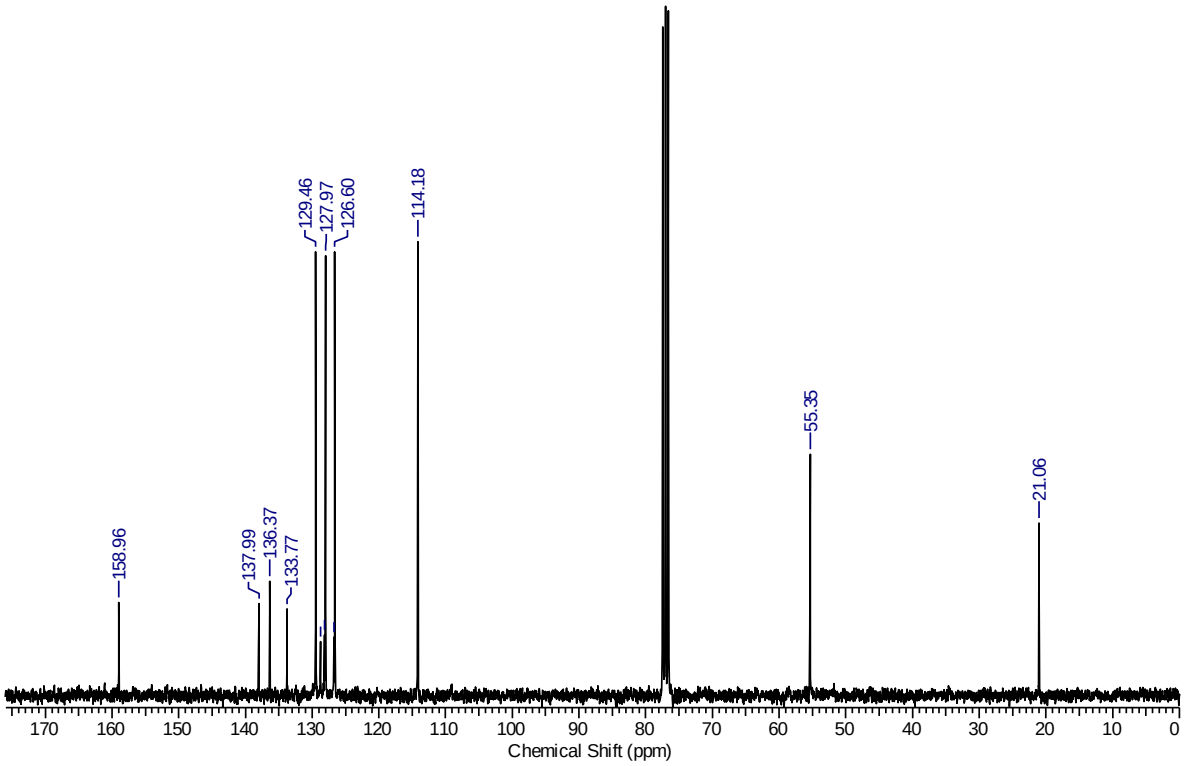
3ea

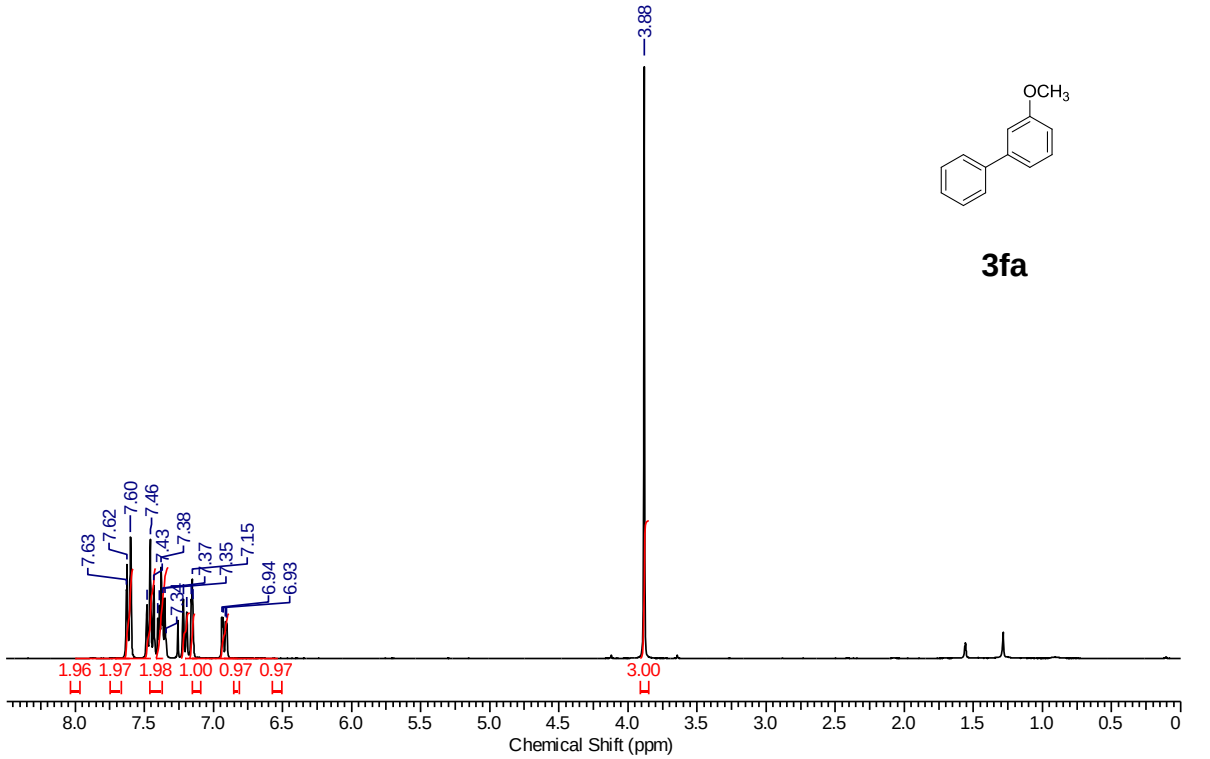

159.94
142.73
138.24
137.22
129.71
129.47
127.04
119.70
119.53
112.93
112.74
112.40
55.29
29.73
21.12
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


3.86
2.40
7.55
7.52
7.46
7.26
7.23
7.00
6.97
2.04
2.07
2.20
2.04
3.27
3.00
Chemical Shift (ppm)

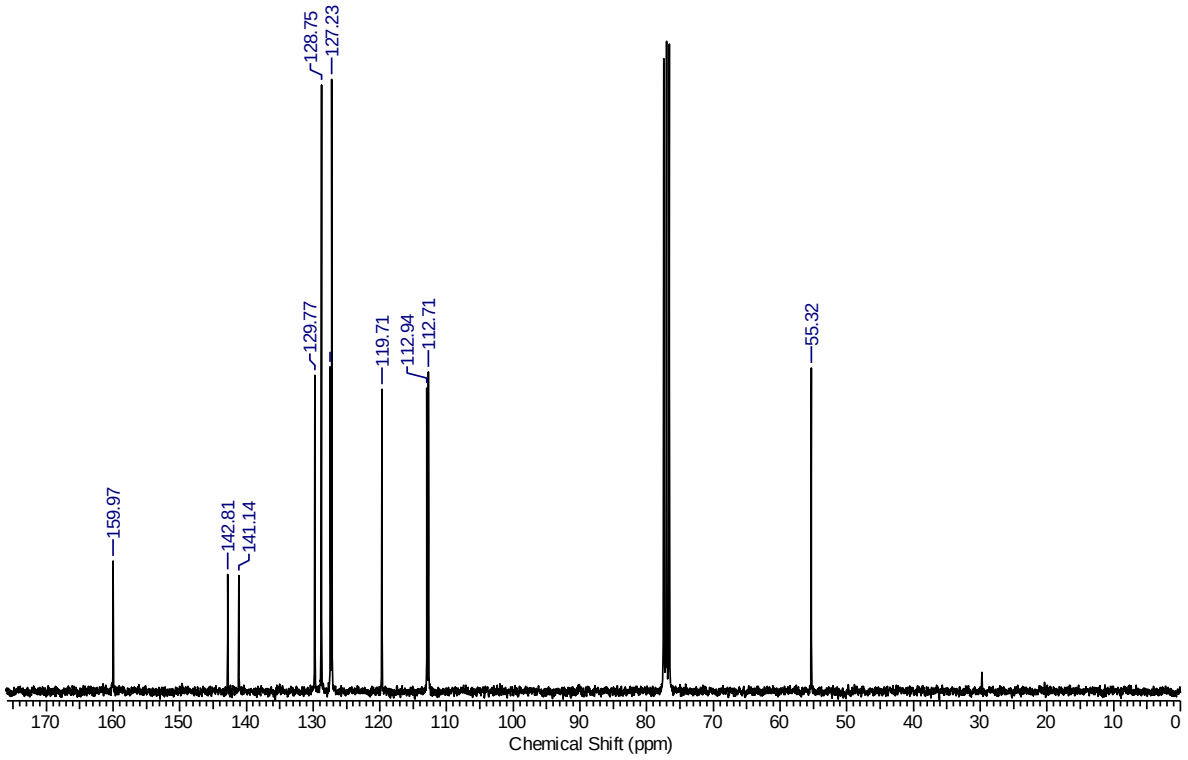
OCH3
H3C

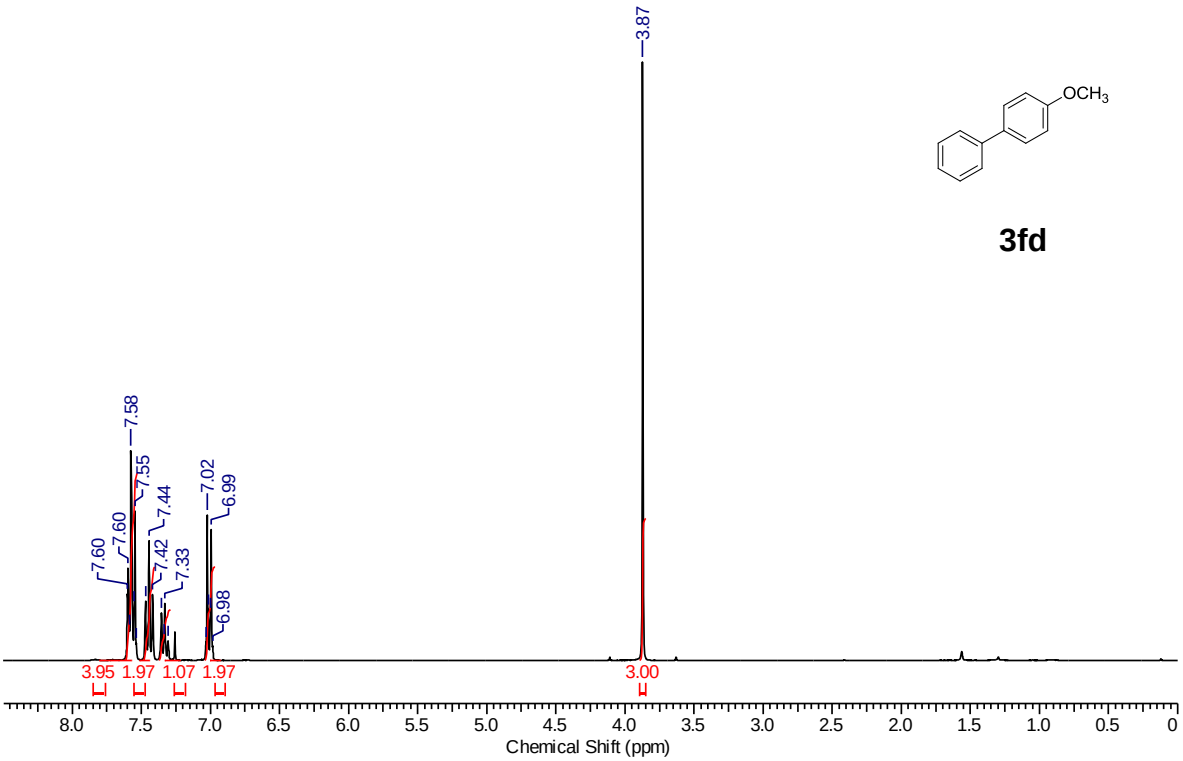
3ed


158.96
137.99
136.37
133.77
129.46
127.97
126.60
114.18
55.35
21.06
Chemical Shift (ppm)


3.88
OCH3
7.63
7.62
7.60
7.46
7.43
7.38
7.37
7.35
7.15
6.94
6.93
7.34
1.96 1.97 1.98 1.00 0.97 0.97
3.00
Chemical Shift (ppm)

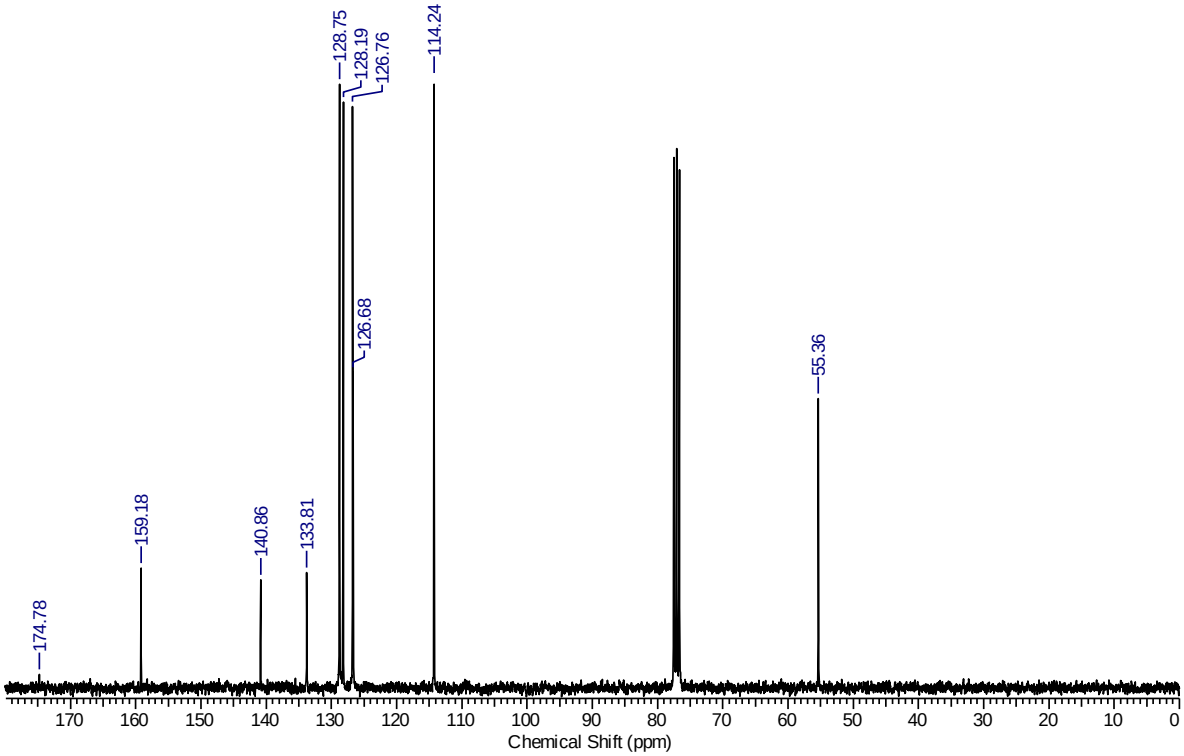
3fa

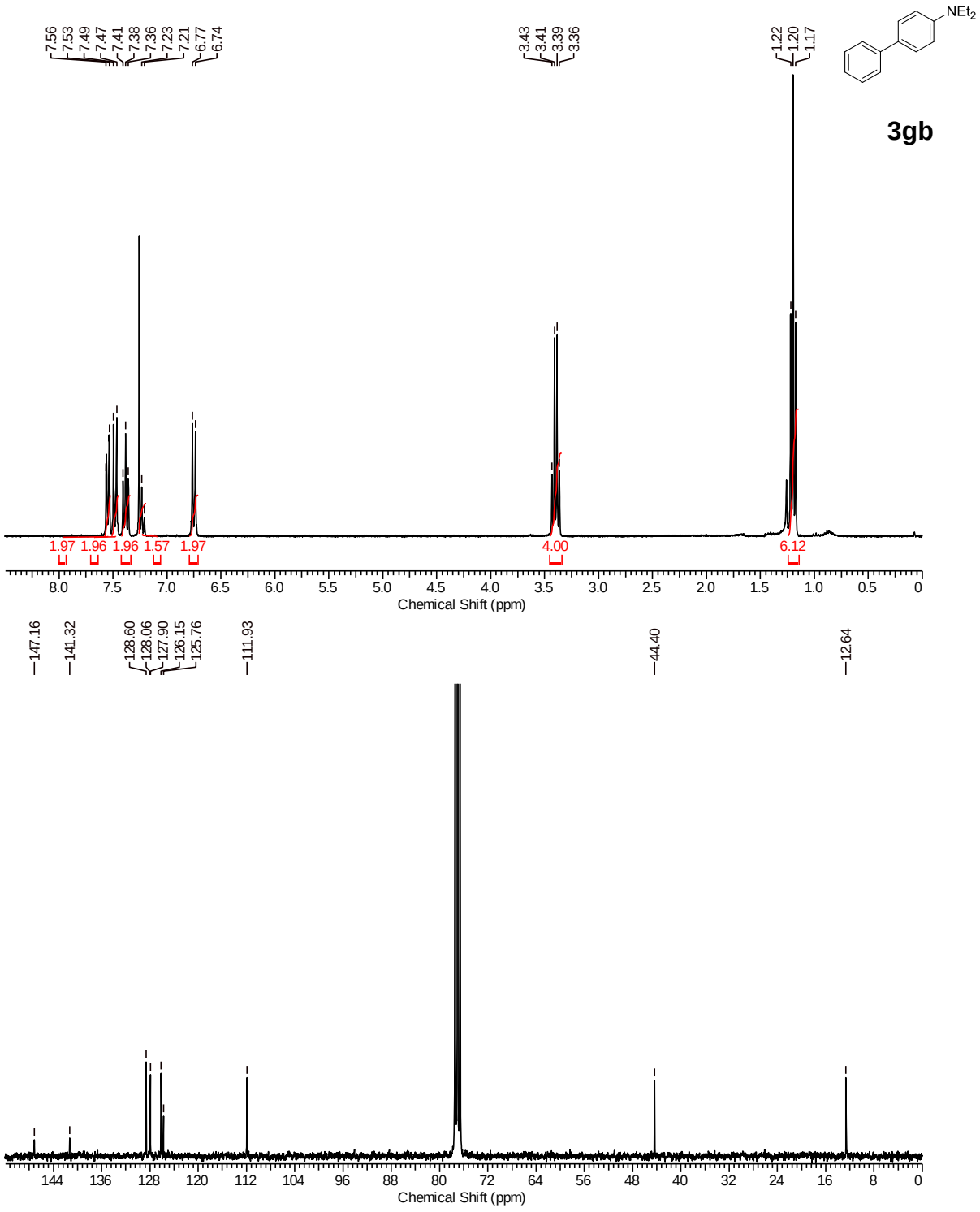

128.75
127.23
129.77
119.71
112.94
112.71
55.32
159.97
142.81
141.14
Chemical Shift (ppm)

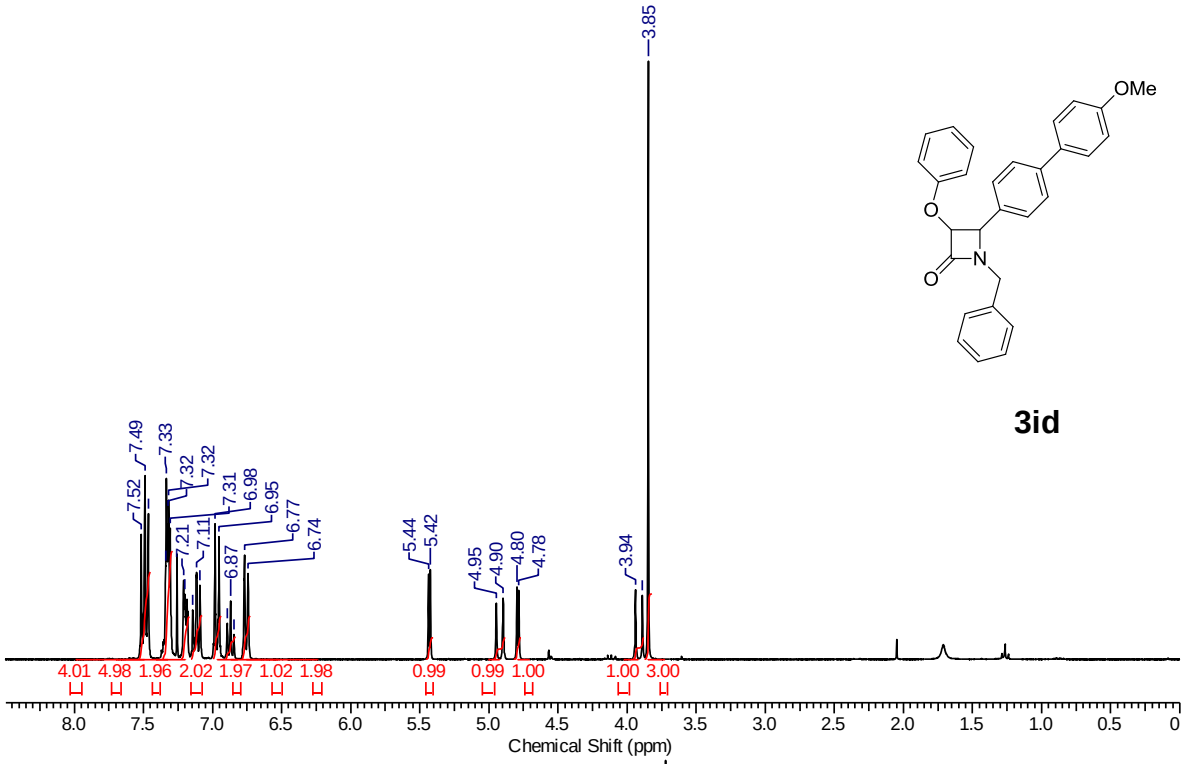

3.87
7.58
7.55
7.44
7.60
7.60
7.42
7.33
7.02
6.99
6.98
3.95
1.97
1.07
1.97
3.00
Chemical Shift (ppm)

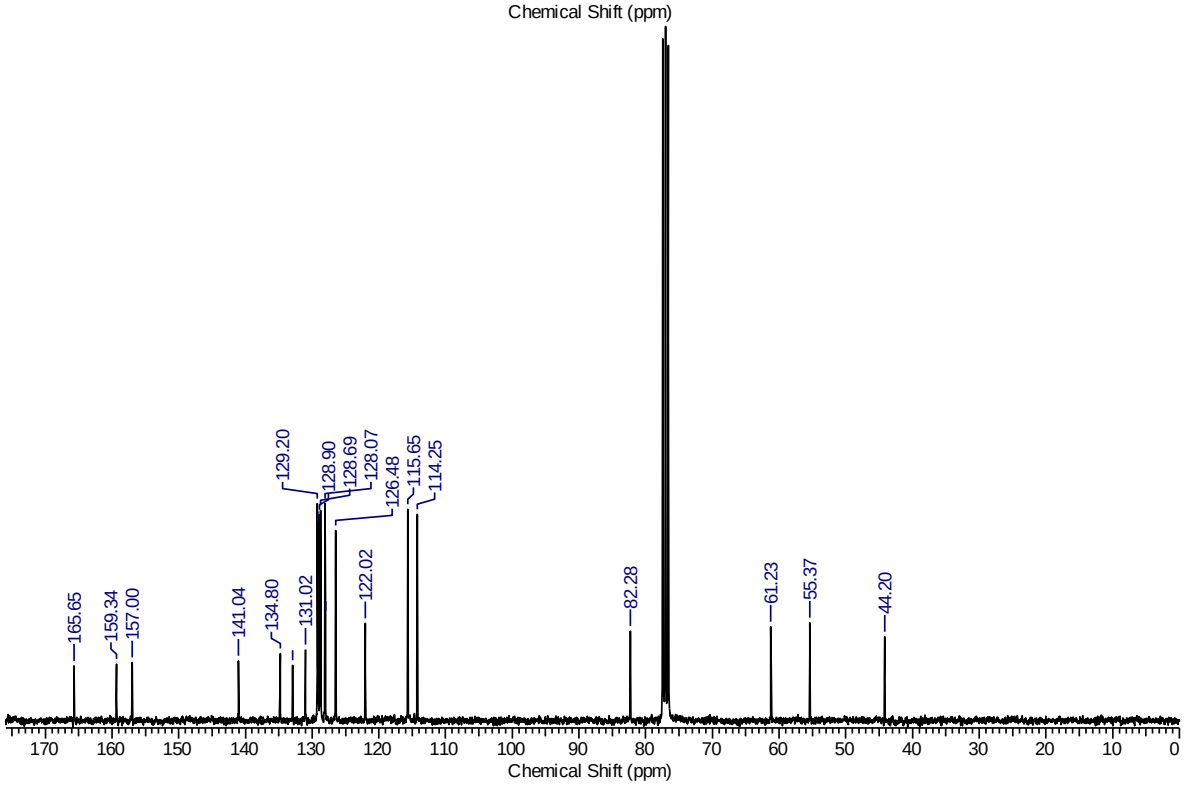
OCH3

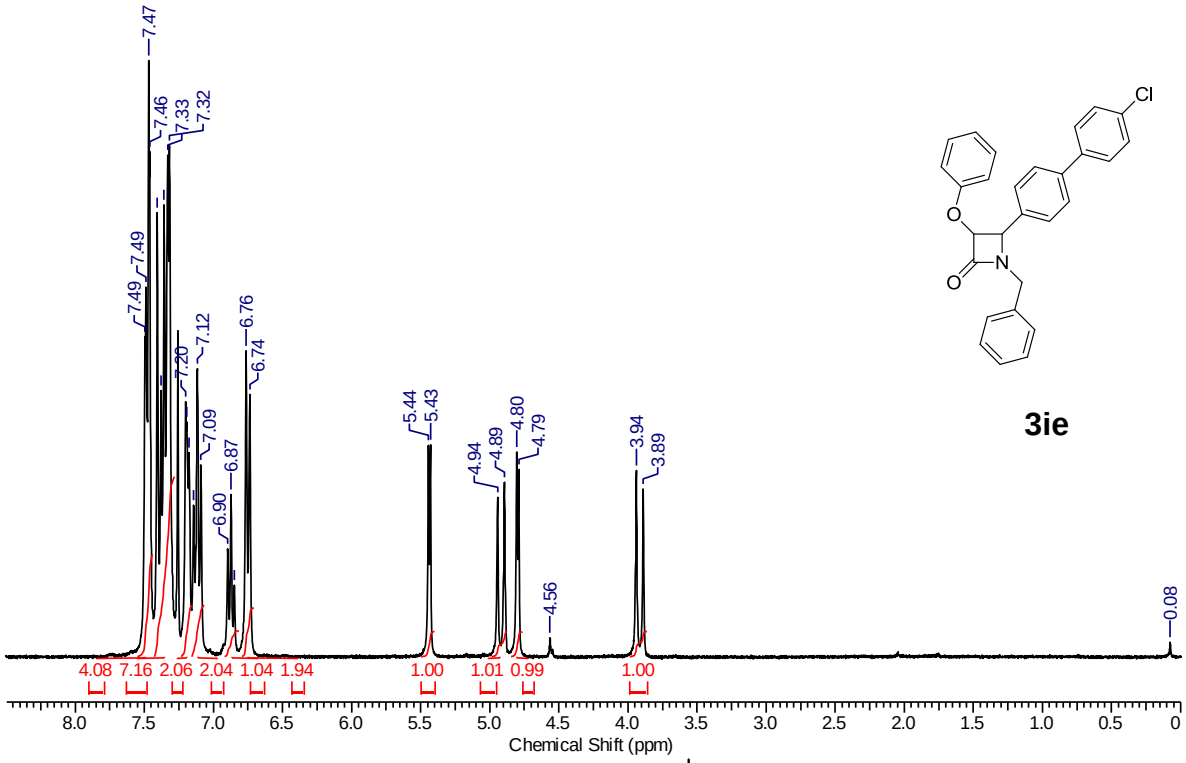
3fd

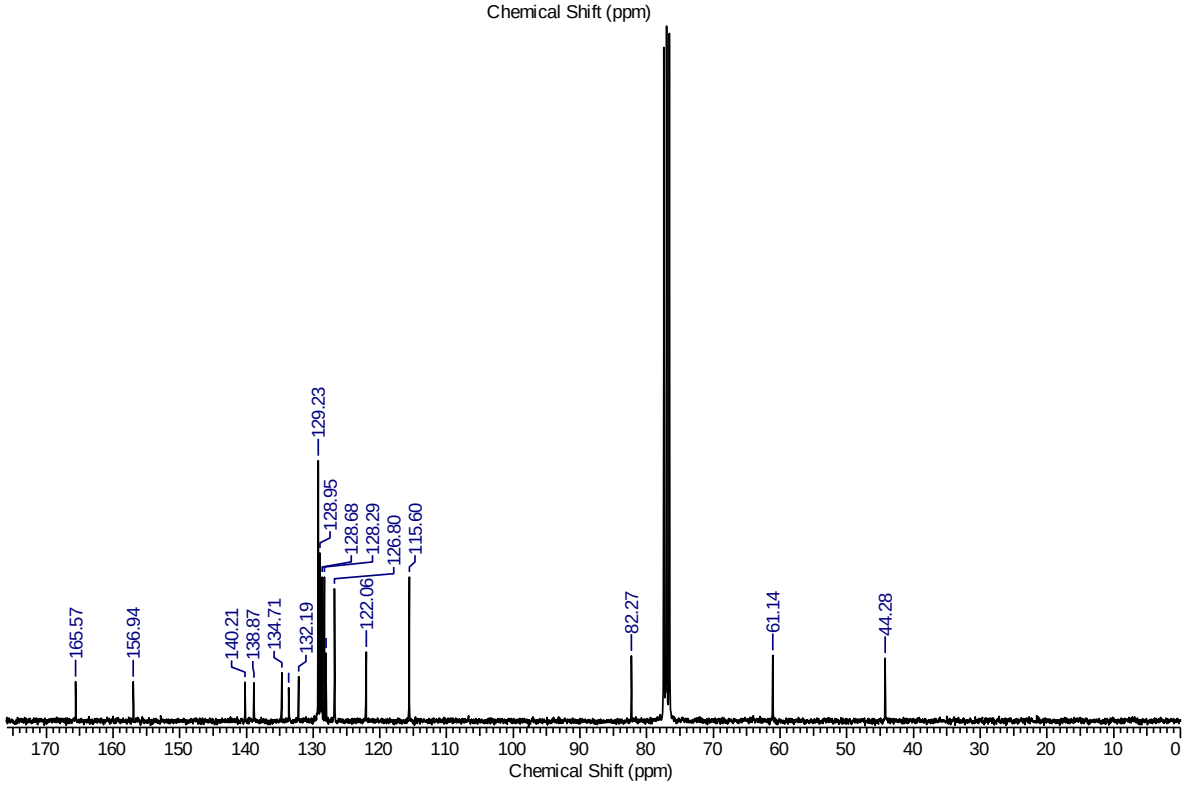

128.75
128.19
126.76
114.24
126.68
55.36
159.18
140.86
133.81
174.78
Chemical Shift (ppm)

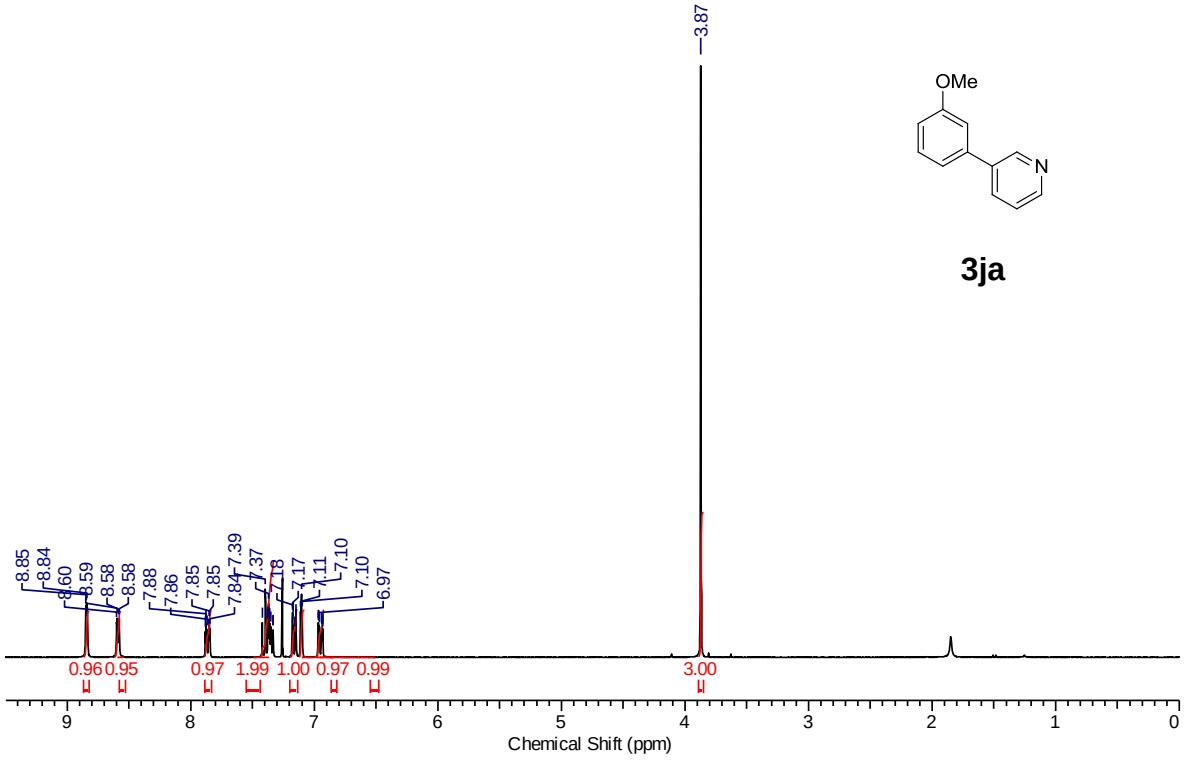

7.56
7.53
7.49
7.47
7.41
7.38
7.36
7.23
7.21
6.77
6.74
3.43
3.41
3.39
3.36
1.22
1.20
1.17
NEt2
3gb
1.97 1.96 1.96 1.57 1.97
4.00
6.12
Chemical Shift (ppm)
147.16
141.32
128.60
128.06
127.90
126.15
125.76
111.93
44.40
12.64
Chemical Shift (ppm)

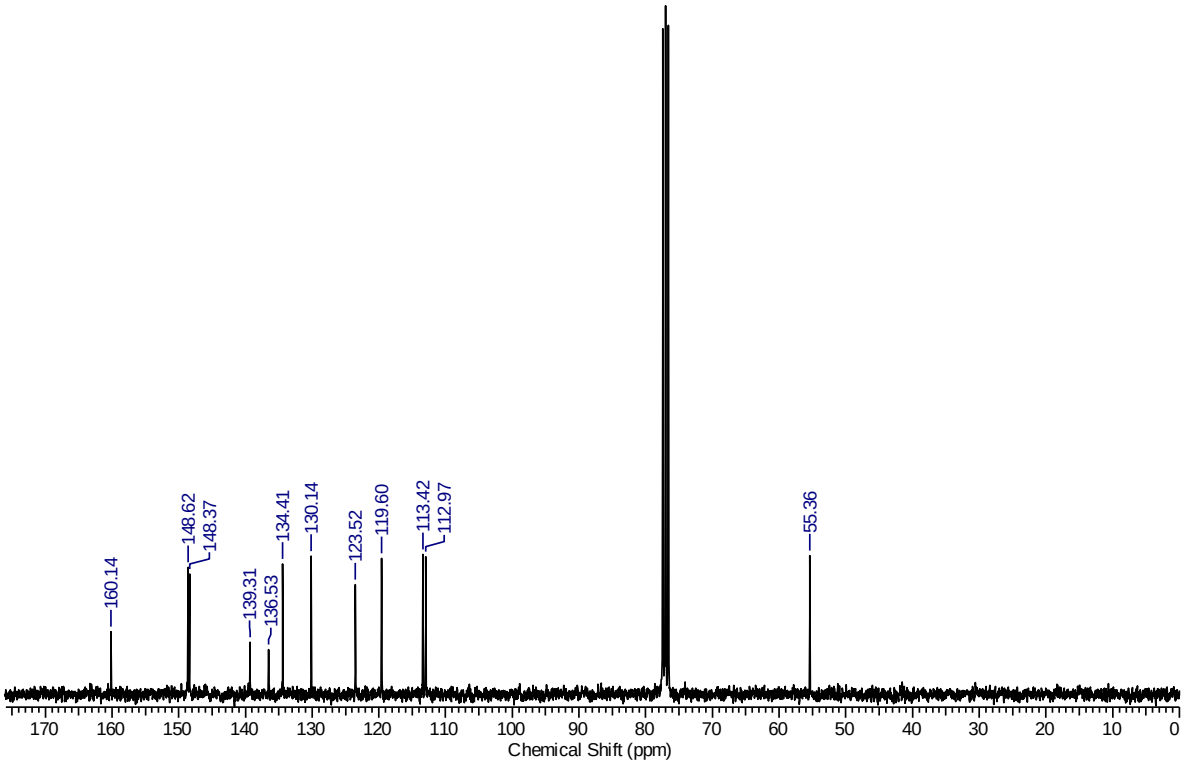

3.85
OMe
O
N
O
3id
7.49
7.33
7.32
7.32
7.52
7.31
6.98
6.95
7.21
7.11
6.77
6.74
6.87
5.44
5.42
4.95
4.90
4.80
4.78
3.94
4.01 4.98 1.96 2.02 1.97 1.02 1.98 0.99 0.99 1.00 1.00 3.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0
Chemical Shift (ppm)

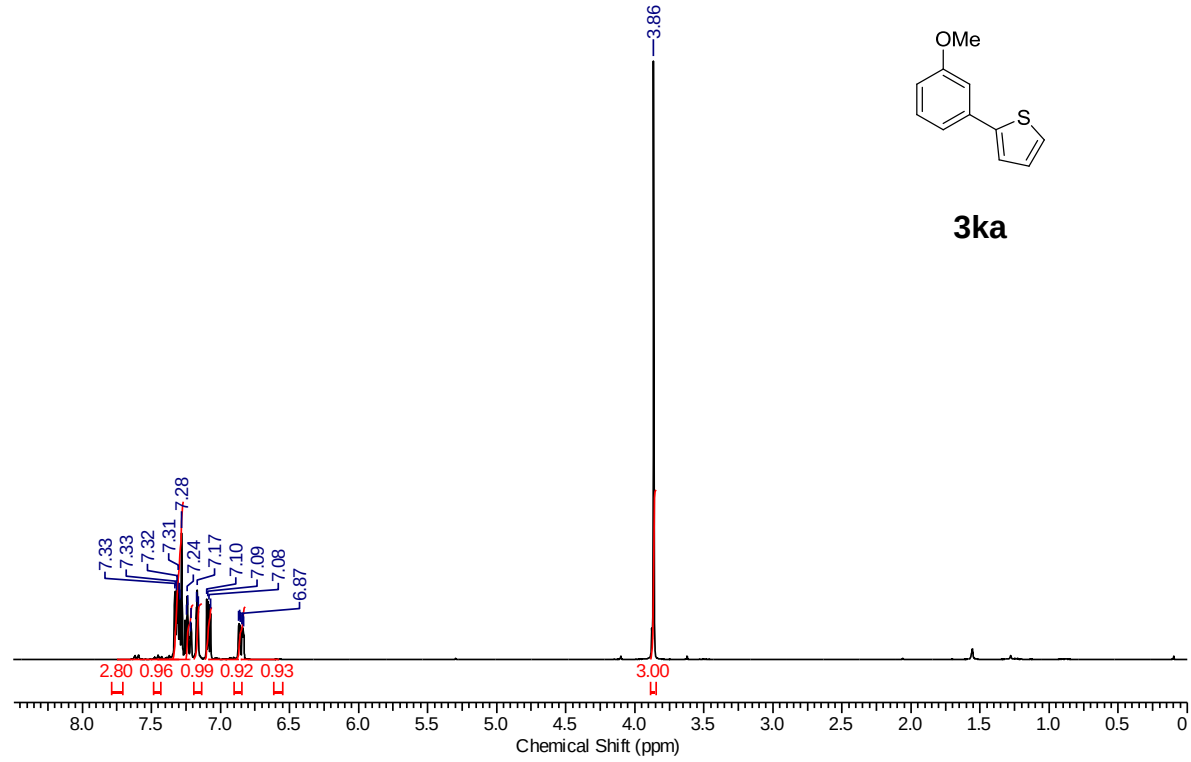

129.20
128.90
128.69
128.07
126.48
115.65
114.25
165.65
159.34
157.00
141.04
134.80
131.02
122.02
82.28
61.23
55.37
44.20
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
Chemical Shift (ppm)

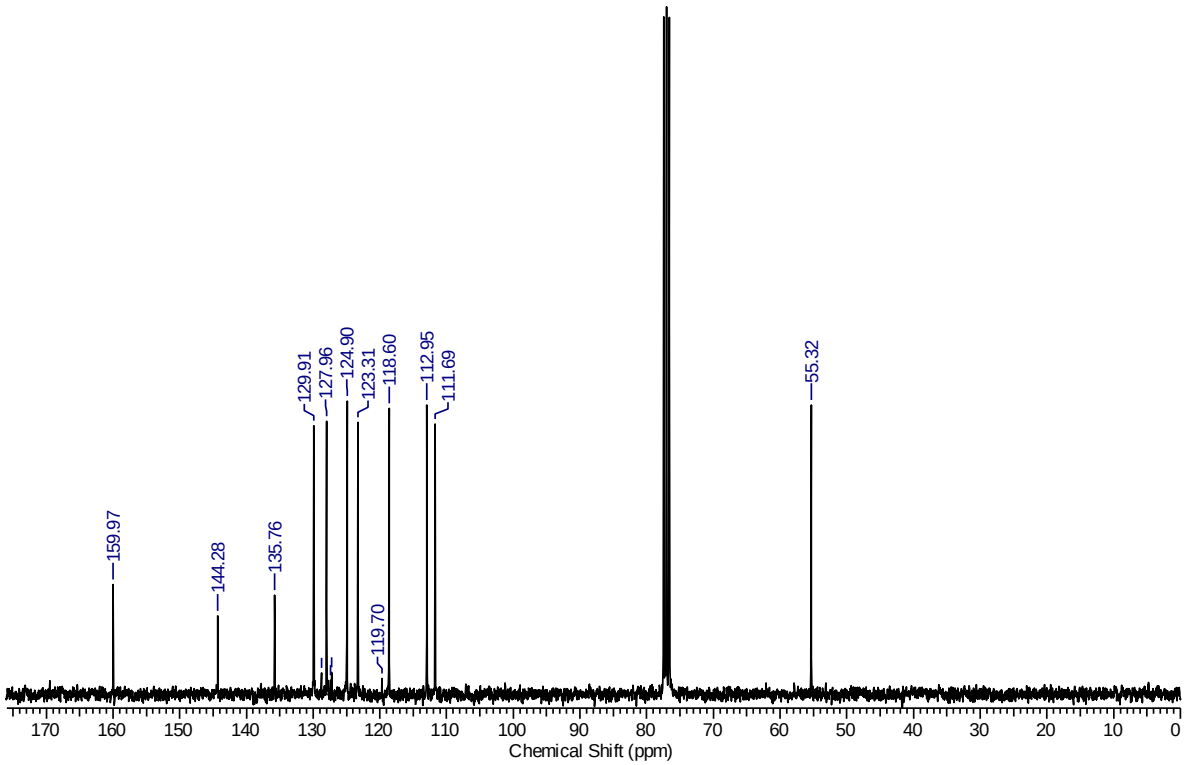

7.47
7.46
7.33
7.32
7.49
7.49
7.20
7.12
6.76
6.74
7.09
6.87
6.90
5.44
5.43
4.94
4.89
4.80
4.79
4.56
3.94
3.89
0.08
4.08 7.16 2.06 2.04 1.04 1.94
1.00 1.01 0.99 1.00
Chemical Shift (ppm)
Cl
O
N
O
3ie

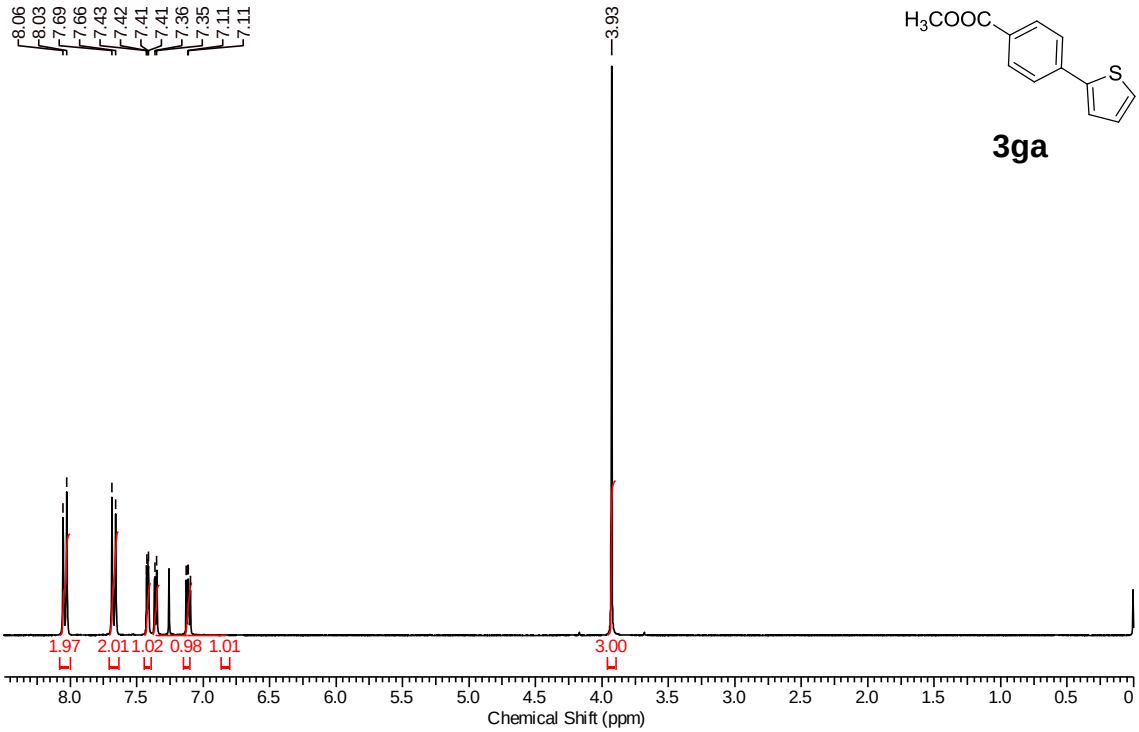

129.23
128.95
128.68
128.29
126.80
115.60
122.06
165.57
156.94
140.21
138.87
134.71
132.19
82.27
61.14
44.28
Chemical Shift (ppm)


3.87
OMe
N
3ja
8.85
8.84
8.60
8.59
8.58
8.58
7.88
7.86
7.85
7.85
7.84
7.39
7.37
7.18
7.17
7.11
7.10
7.10
6.97
0.96
0.95
0.97
1.99
1.00
0.97
0.99
3.00
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)


160.14
148.62
148.37
139.31
136.53
134.41
130.14
123.52
119.60
113.42
112.97
55.36
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

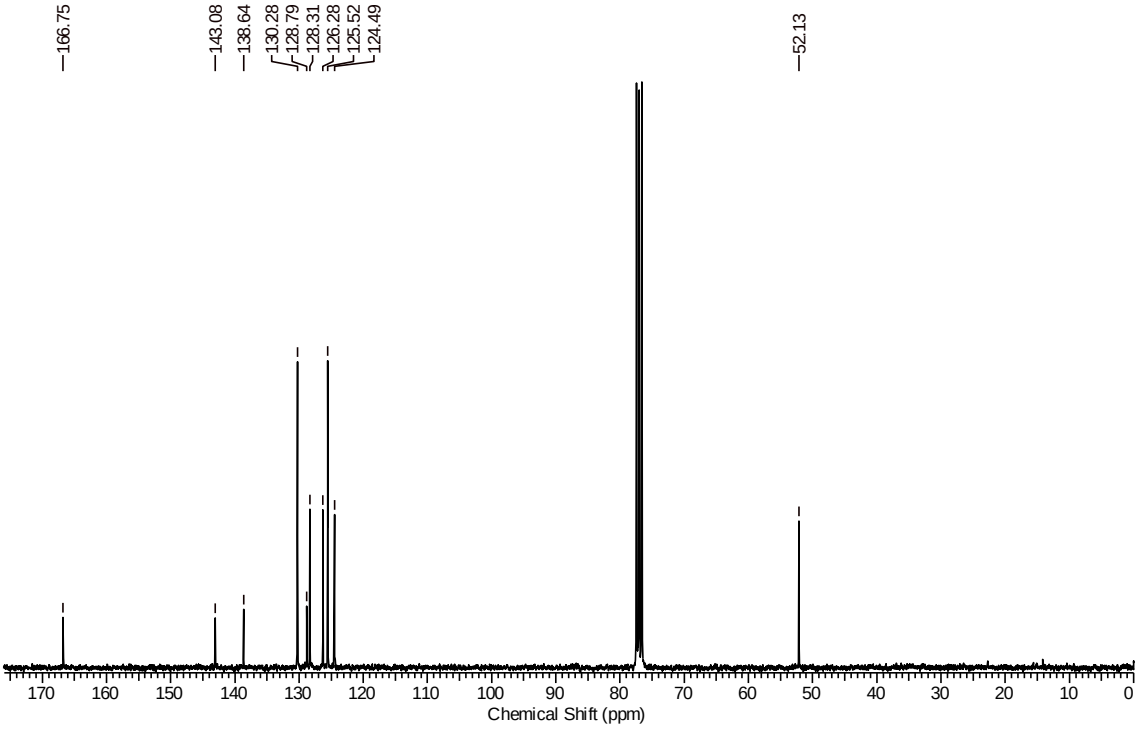

3.86
OMe
S
3ka
7.33
7.33
7.32
7.31
7.28
7.24
7.17
7.10
7.09
7.08
6.87
2.80 0.96 0.99 0.92 0.93
3.00
Chemical Shift (ppm)


159.97
144.28
135.76
129.91
127.96
124.90
123.31
119.70
118.60
112.95
111.69
55.32
Chemical Shift (ppm)


8.06
8.03
7.69
7.66
7.43
7.42
7.41
7.41
7.36
7.35
7.11
7.11
3.93
1.97
2.01
1.02
0.98
1.01
3.00
Chemical Shift (ppm)

H3COOC
S

3ga


166.75
143.08
138.64
130.28
128.79
128.31
126.28
125.52
124.49
52.13
Chemical Shift (ppm)