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C-H arylation reactions through aniline activation catalysed by PANI-g-C_3N_4-TiO_2 composite under visible light in aqueous medium

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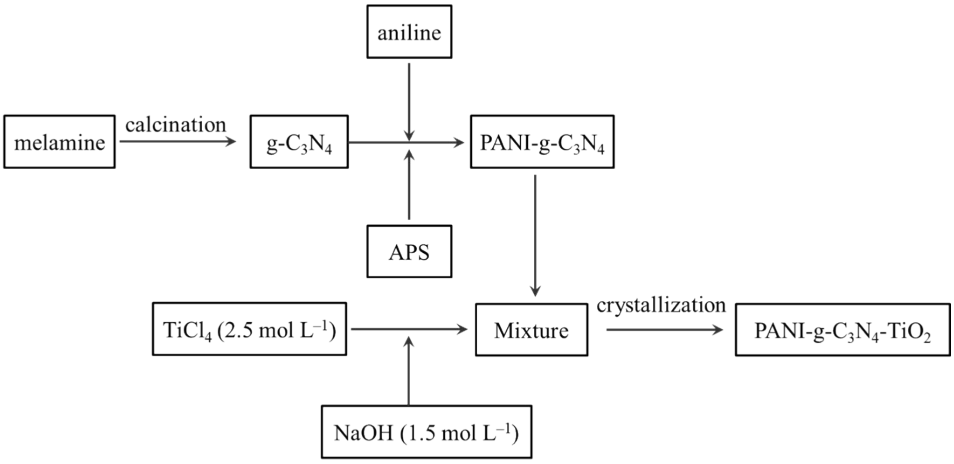
1 General experimental details

Chemicals were used as received without special purification unless stated otherwise. Column chromatography was generally performed on silica gel (300−400 mesh). Thin-layer chromatography (TLC) was visualized using UV light. ^{1}H and ^{13}C NMR were recorded in $CDCl_3$ at ambient temperature on a 300 MHz NMR spectrometer. Chemical shifts are reported in δ units, parts per million (ppm), and are referenced to $CDCl_3$ (δ 7.26 ppm for H NMR and 77.0 ppm for C NMR). The coupling constants J are given in Hz. Liquid Chromatograph-Mass Spectrometer (LCMS) was performed on a Shimadzu LCMS-2020 instrument with an ESI source. High-resolution mass spectrometry (HRMS) was performed on an Agilent 6540 Q-TOF MS instrument with an ESI source. X-ray diffraction (XRD) patterns were collected on a SHIMADZU XRD-6000diffractometer with Cu Kα radiation at 40 kV and 30 mA in the range of 5-80 ^{o} at a scanning rate of 6 min^{-1}. HRTEM (JEM-2010, JEOL) was used to study the microstructures. UV-vis absorption spectra were obtained by a UV-vis spectrophotometer (SOLID 3700, Shimadzu). The fluorescence spectra were achieved by using a fluorescence spectrophotometer (Hitachi, F-4500) at wavelengths between 200-800 nm. The excitation wavelength was 320 nm, PMT voltage was 700 V, and the spectral slit width was 2.5 nm. X-ray photoelectron spectroscopy (XPS) measurement was performed using PHOIBOS 100 hemispherical energy analyzer (SPECS) equipped with Al, Kα radiation in the fixed analyzer transmission mode. All peaks in XPS spectra have already been calibrated with C 1s peak at 285 eV. Nitrogen adsorption-desorption isotherms were obtained from a TriStar 3010 isothermal nitrogen sorption analyzer (Micromeritics, USA). The 14W CFL (wavelength: 435~610 nm), green LED (3W) and blue LED (3W) were purchased from Philips (Beijing, China).

1.1 Preparation of g-C_3N_4, PANI-g-C_3N_4 and PANI−g-C_3N_4−TiO_2 composites

The g-C_3N_4, PANI-g-C_3N_4 and PANI−g-C_3N_4−TiO_2 composites were prepared according to our previous report and was outlined in Figure S1.[1]



Scheme S1. Preparation of g-C_3N_4, PANI-g-C_3N_4 and PANI−g-C_3N_4−TiO_2 composites

g-C_3N_4 was prepared using melamine as a precursor.[2] Briefly, 10 g of melamine was put into a semi-closed crucible and statically calcinated at 580 °C with a ramping rate of 5 °C min^{-1} and tempered for another 4 h.

Then, 1.4 g of g-C_3N_4 was dispersed into 30 mL of HCl solution (0.125 M) and stirred at room temperature for 1 h, followed by addition of aniline (20 wt%~80 wt% of g-C_3N_4). The dispersion was sonicated for 15 min. Ammonium persulphate (APS) with the same molar ratio of aniline was dissolved into 30 mL of HCl solution (0.125 M). Afterwards, the APS solution was added dropwise into the above suspension at 0 °C, and then stirred for another 5 h. After that, the mixture was filtrated and the resultant solid was rinsed by deionized water and ethanol for several times until the filtrate was colorless. The obtained solid was dried under 60 °C for 10 h, grinded into dark green powder, and designated as PANI-g-C_3N_4 composite.

For the preparation of the ternary composite PANI-g-C_3N_4-TiO_2, initially, 15 mL of sodium hydroxide solution (c = 1.5 mol L^{-1}) was added slowly to 3 mL of aqueous $TiCl_4$ solution (2.5 mol L^{-1}), which was diluted with 80 mL of deionized water. The mixture was stirred and a certain amount of PANI-g-C_3N_4 was added to the above mixture (m(g-C_3N_4): m(TiO_2) = 1:2). The temperature was then raised slowly to 80 °C, and the mixture was crystallized for 2 h. Afterwards, aqueous sodium hydroxide solution (c = 2.5 mol L^{-1}) was added dropwise to maintain the pH at 5~6. The solid was filtrated, washed with deionized water, dried under vacuum, and grinded to afford PANI-g-C_3N_4-TiO_2.

1.2 Surface areas of PANI-g-C_3N_4-TiO_2 composites

Table S1. Surface areas of various PANI-g-C_3N_4-TiO_2 samples

Sample	Surface area /$m^2\ g^{-1}$
PANI(20%)-g-C_3N_4-TiO_2	95
PANI(40%)-g-C_3N_4-TiO_2	128
PANI(60%)-g-C_3N_4-TiO_2	98
PANI(80%)-g-C_3N_4-TiO_2	66

1.3 Characterization of semiconductor composites

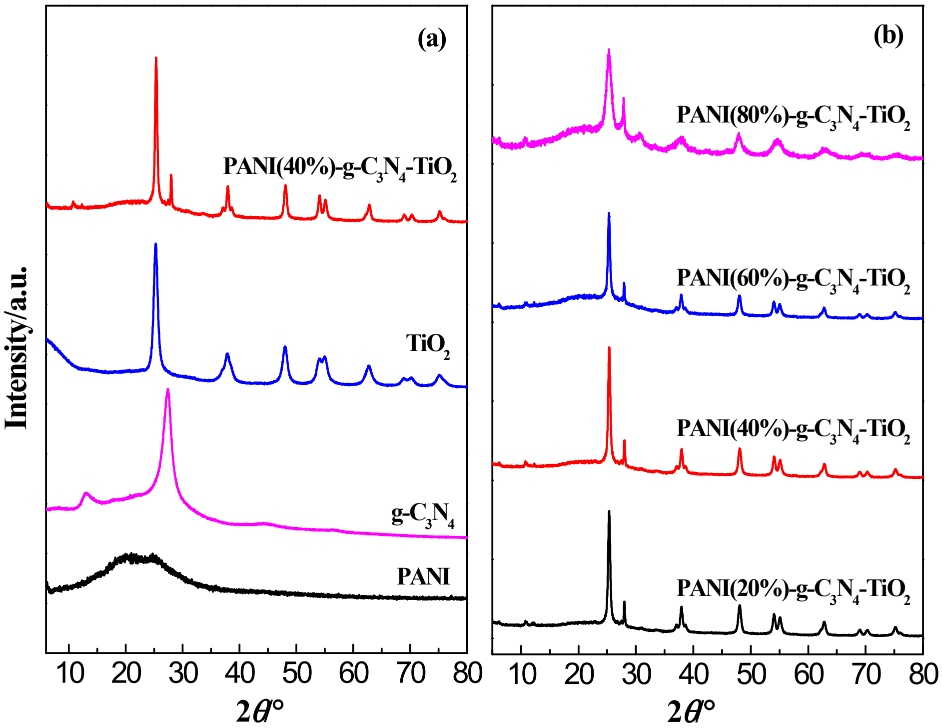


Figure S1. XRD patterns of PANI, g-C_3N_4, TiO_2, and PANI-g-C_3N_4-TiO_2 composites

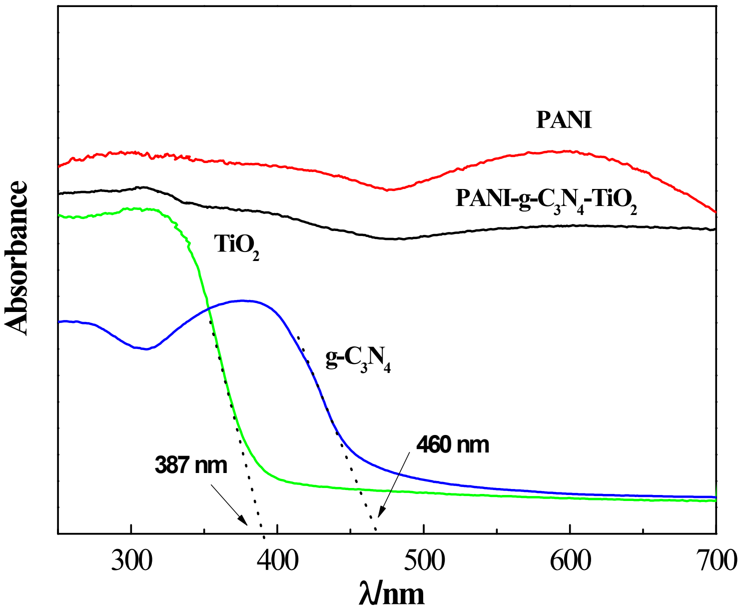


Figure S2. UV–vis DRS of TiO_2, g-C_3N_4, PANI, and PANI-g-C_3N_4-TiO_2 composite

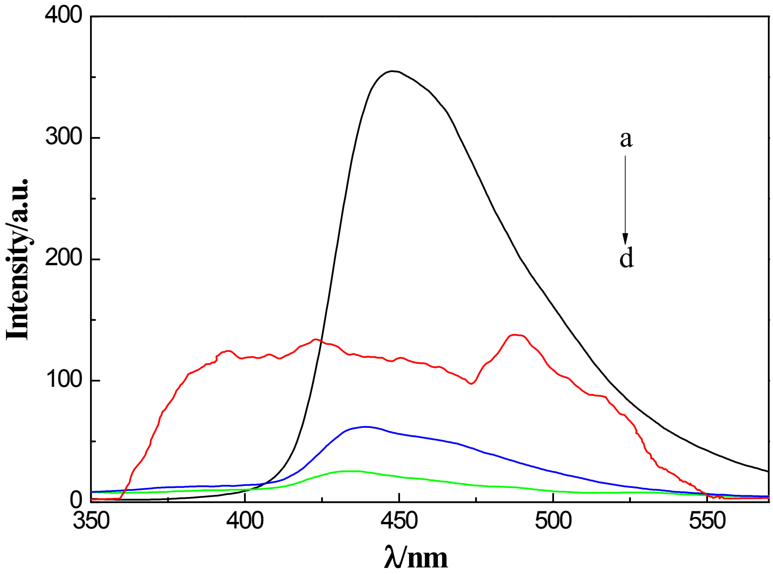


Figure S3. Photoluminescence spectra of different materials (a) g-C_3N_4, (b) TiO_2, (c) g-C_3N_4-TiO_2, and (d) PANI-g-C_3N_4-TiO_2

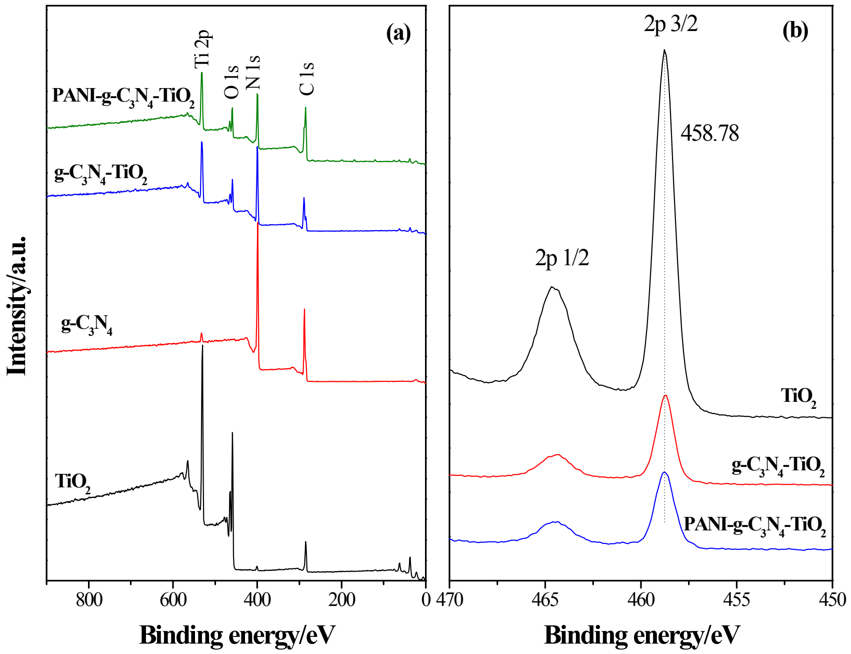


Figure S4. XPS survey (a) of TiO_2, g-C_3N_4, g-C_3N_4-TiO_2, and PANI-g-C_3N_4-TiO_2; and their corresponding Ti 2p spectra (b)

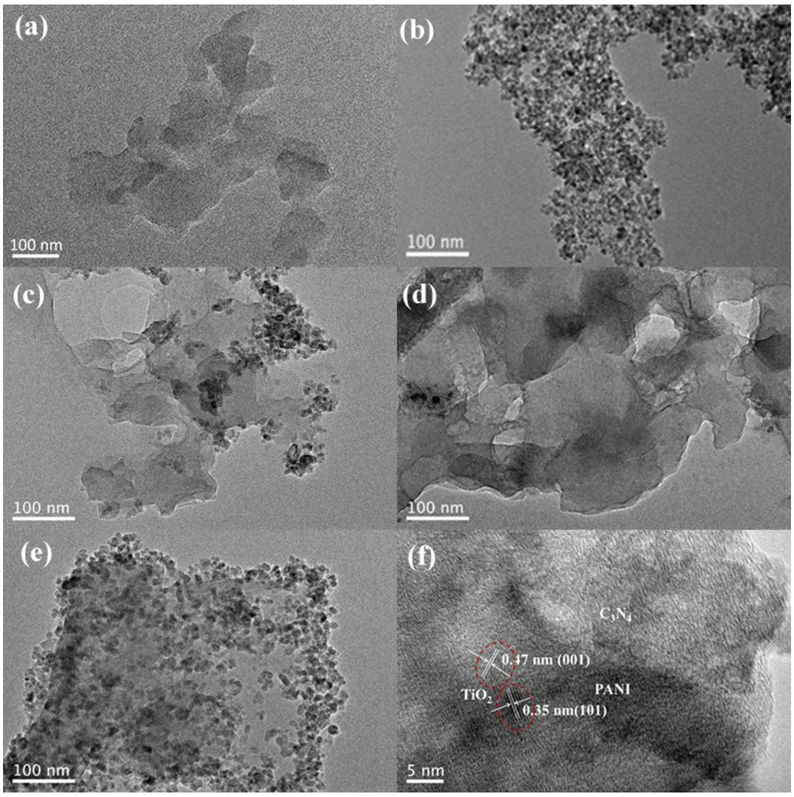


Figure S5. TEM images of (a) g-C_3N_4, (b) TiO_2, (c) g-C_3N_4-TiO_2, (d) PANI-g-C_3N_4, and (e) PANI-g-C_3N_4-TiO_2; and HRTEM image of (f) PANI-g-C_3N_4-TiO_2.

1.4 Typical procedure for synthesis of 3a

To a mixture of aniline (0.3 mmol) and $MeSO_3H$ (0.3 mmol) in acetone/H_2O (2:1, 3 mL) at 25 °C was added *t*-BuONO (0.45 mmol). The reaction mixture was stirred for 10 minutes at 25 °C. Then, furan **2a** (10 equiv.) and PANI-g-C_3N_4-TiO_2 (30 mg) were successively added. The mixture was then irradiated with a 14 W CFL and stirred at room temperature for 3 h. The distance of the reaction vial from the light is about 5 centimeters. After reaction, the mixture was diluted with EtOAc (10 mL) and H_2O (5 mL), and the solid catalyst was recovered by centrifugation. The aqueous phase was extracted with EtOAc (5 mL × 3). The collected organic extracts were dried on Na_2SO_4, filtered and evaporated to dryness. The crude was purified by flash chromatography on silica gel to give the pure product **3a** in 87% yield.

1.5 Typical procedure for synthesis of 5a

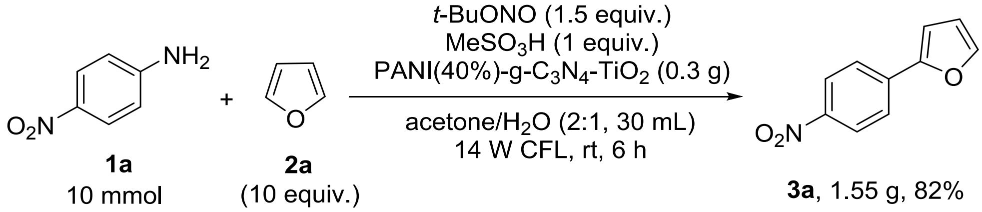
To a mixture of aniline (0.3 mmol) and $MeSO_3H$ (0.3 mmol) in acetone/H_2O (2:1, 3 mL) at 25 °C was added *t*-BuONO (0.45 mmol). The reaction mixture was stirred for 10 minutes at 25 °C. Then, enol acetate **4** (10 equiv.) and PANI-g-C_3N_4-TiO_2 (30 mg) were successively added. The mixture was then irradiated with a 14 W CFL and stirred at room temperature for 6 h. The distance of the reaction vial from the light is about 5 centimeters. After reaction, the mixture was diluted with EtOAc (10 mL) and H_2O (5 mL), and the solid catalyst was recovered by centrifugation. The aqueous phase was extracted with EtOAc (5 mL × 3). The collected organic extracts were dried on Na_2SO_4, filtered and evaporated to dryness. The crude was purified by flash chromatography on silica gel to give the pure product **5a** in 85% yield.

1.6 Typical procedure for synthesis of 7a

To a mixture of aniline (0.3 mmol) and $MeSO_3H$ (0.3 mmol) in H_2O (3 mL) at 25 °C was added *t*-BuONO (0.45 mmol). The reaction mixture was stirred for 10 minutes at 25 °C. Then, enol acetate **6** (5 equiv.) and PANI-g-C_3N_4-TiO_2 (30 mg) were successively added. The mixture was then irradiated with a 14 W CFL and stirred at room temperature for 12 h. The distance of the reaction vial from the light is about 5 centimeters. After reaction, the mixture was diluted with EtOAc (10 mL) and H_2O (5 mL), and the solid catalyst was recovered by centrifugation. The aqueous phase was extracted with EtOAc (5 mL × 3). The collected organic extracts were dried on Na_2SO_4, filtered and evaporated to dryness. The crude was purified by flash chromatography on silica gel to give the pure product **7a** in 91% yield.

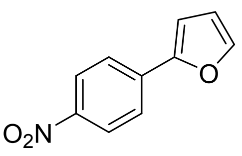
1.7 Scale-up synthesis

The scale-up experiment was conducted at a 10 mmol scale (Scheme S2). After completion of the reaction, the mixture was diluted with EtOAc (30 mL) and the catalyst was filtered off and washed with ethyl acetate. The collected organic extracts were dried, concentrated and purified by flash chromatography on silica gel to give the pure product **3a** in 82% yield (1.55 g).

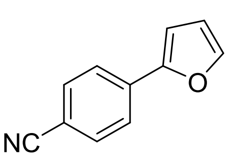


Scheme S2 Gram-scale synthesis of **3a**.

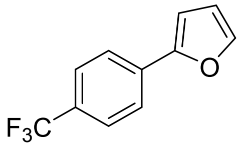
2 Characterization data of compounds



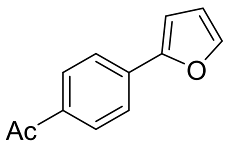
2-(4-Nitrophenyl)furan (3a).[3] ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.26 - 8.21 (m, 2H), 7.80 - 7.75 (m, 2H), 7.57 (dd, J = 1.7, 0.5 Hz, 1H), 6.87 (dd, J = 3.5, 0.5 Hz, 1H), 6.55 (dd, J = 3.5, 1.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.83, 146.50, 144.28, 136.55, 124.43, 124.04, 112.57, 109.12; MS (ESI) *m/z* 212 $[M + Na]^+$.



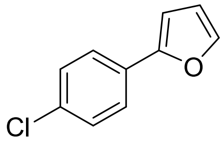
4-(Furan-2-yl)benzonitrile (3b).[3] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.74 - 7.70 (m, 2H), 7.66 - 7.62 (m, 2H), 7.53 (dd, J = 1.7, 0.6 Hz, 1H), 6.80 (dd, J = 3.5, 0.6 Hz, 1H), 6.52 (dd, J = 3.5, 1.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 152.02, 143.77 , 134.70, 132.65, 124.00, 119.06, 112.34, 110.31, 108.26; MS (ESI) *m/z* 192 $[M + Na]^+$.



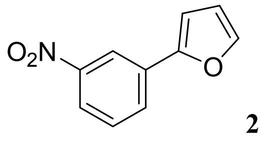
2-(4-(Trifluoromethyl)phenyl)furan (3c).[4] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.2 Hz, 2H), 7.64 (d, *J* = 8.3 Hz, 2H), 7.55 - 7.50 (m, 1H), 6.77 (d, *J* = 3.4 Hz, 1H), 6.52 (dd, *J* = 3.4, 1.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 152.63, 143.21, 134.09 (d, *J* = 1.3 Hz), 129.06 (q, *J* = 32.5 Hz), 125.82 (q, *J* = 3.9 Hz), 123.87, 122.55, 112.09, 107.10; MS (ESI) *m/z* 235 $[M + Na]^+$.



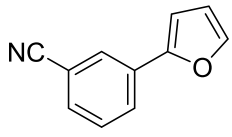
1-(4-(Furan-2-yl)phenyl)ethanone (3d).[5] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.7 Hz, 2H), 7.73 (d, *J* = 8.7 Hz, 2H), 7.52 (dd, *J* = 1.8, 0.6 Hz, 1H), 6.79 (dd, *J* = 3.4, 0.6 Hz, 1H), 6.51 (dd, *J* = 3.4, 1.8 Hz, 1H), 2.60 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.51 (s), 152.91, 143.38, 135.59, 134.96, 129.05, 123.63, 112.20, 107.59, 26.67; MS (ESI) *m/z* 209 $[M + Na]^+$.



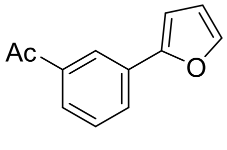
2-(4-Chlorophenyl)furan (3e).[5] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.64 - 7.59 (m, 2H), 7.48 (dd, *J* = 1.8, 0.7 Hz, 1H), 7.39 - 7.34 (m, 2H), 6.65 (dd, *J* = 3.4, 0.6 Hz, 1H), 6.49 (dd, *J* = 3.4, 1.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.02, 142.41, 133.04, 129.46, 128.96, 125.10, 111.88, 105.53; MS (ESI) *m/z* 201 $[M + Na]^+$.



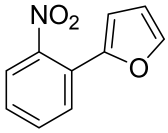
2-(3-Nitrophenyl)furan (3f).[6] ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.48 (t, *J* = 1.9 Hz, 1H), 8.08 (ddd, *J* = 8.2, 2.3, 1.0 Hz, 1H), 7.95 (ddd, *J* = 7.8, 1.6, 1.1 Hz, 1H), 7.55 (d, *J* = 8.1 Hz, 1H), 7.52 (dd, *J* = 2.8, 2.3 Hz, 1H), 6.81 (dd, *J* = 3.4, 0.6 Hz, 1H), 6.53 (dd, *J* = 3.4, 1.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.61, 148.81, 143.42, 132.46, 129.82, 129.34, 121.80, 118.61, 112.22, 107.40; MS (ESI) *m/z* 212 $[M + Na]^+$.



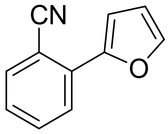
3-(Furan-2-yl)benzonitrile (3g).[7] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92 (d, *J* = 1.2 Hz, 1H), 7.86 (dt, *J* = 7.3, 1.8 Hz, 1H), 7.52 - 7.44 (m, 3H), 6.74 (dd, *J* = 3.4, 0.6 Hz, 1H), 6.51 (dd, *J* = 3.4, 1.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.65, 143.28, 132.02, 130.50, 129.63, 127.75, 127.21, 118.78, 113.05, 112.13, 106.98; MS (ESI) *m/z* 192 $[M + Na]^+$.



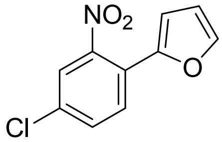
1-(3-(Furan-2-yl)phenyl)ethanone (3h). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.26 - 8.22 (m, 1H), 7.87 - 7.80 (m, 2H), 7.47 (ddd, *J* = 8.2, 4.8, 0.6 Hz, 2H), 6.74 (dd, *J* = 3.4, 0.7 Hz, 1H), 6.49 (dd, *J* = 3.4, 1.8 Hz, 1H), 2.64 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.06, 152.99, 142.66, 137.59, 131.41, 129.08, 128.19, 127.12, 123.57, 111.95, 106.08, 26.84; HRMS (ESI): Calcd for $C_{12}H_{10}NaO_2$ $[M + Na]^+$ 209.0573, found 209.0581.



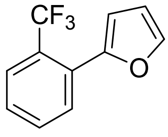
2-(2-Nitrophenyl)furan (3i).[6] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72 - 7.65 (m, 2H), 7.59 - 7.50 (m, 2H), 7.43 - 7.37 (m, 1H), 6.67 (dd, J = 3.5, 0.6 Hz, 1H), 6.49 (dd, J = 3.5, 1.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.46, 147.54, 143.86, 131.97, 128.93, 128.36, 124.15, 123.90, 111.96, 109.76; MS (ESI) m/z 212 $[M + Na]^+$.



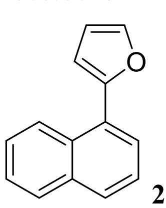
2-(Furan-2-yl)benzonitrile (3j).[5] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.89 (dd, J = 8.1, 0.7 Hz, 1H), 7.72 - 7.68 (m, 1H), 7.65 - 7.59 (m, 1H), 7.56 (dd, J = 1.8, 0.6 Hz, 1H), 7.36 - 7.30 (m, 2H), 6.56 (dd, J = 3.5, 1.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.88, 143.46, 134.29, 133.36, 133.08, 127.24, 126.06, 119.16, 112.40, 110.59, 106.97; MS (ESI) m/z 192 $[M + Na]^+$.



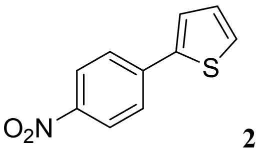
2-(4-Chloro-2-nitrophenyl)furan (3k). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.66 (dd, J = 5.3, 3.2 Hz, 2H), 7.55 - 7.50 (m, 2H), 6.68 - 6.65 (m, 1H), 6.50 (dd, J = 3.5, 1.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.48, 147.39, 144.17, 133.85, 132.12, 129.85, 124.08, 122.62, 112.15, 110.34; HRMS (ESI): Calcd for $C_{10}H_6ClNNaO_3$ $[M + Na]^+$ 245.9928, found 245.9936.



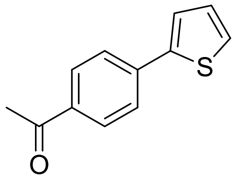
2-(2-(Trifluoromethyl)phenyl)furan (3l). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.75 (dd, J = 8.1, 0.9 Hz, 2H), 7.57 (ddd, J = 4.6, 2.5, 0.7 Hz, 2H), 7.45 - 7.39 (m, 1H), 6.74 - 6.69 (m, 1H), 6.51 (dd, J = 3.4, 1.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.60, 143.15, 131.85, 130.09, 129.86 (d, J = 1.9 Hz), 127.85, 126.75 (q, J = 6.1 Hz), 126.20 (q, J = 271.5 Hz), 122.33, 111.74, 110.21 (q, J = 3.5 Hz); HRMS (ESI): Calcd for $C_{11}H_7F_3NaO$ $[M + Na]^+$ 235.0341, found 235.0354.



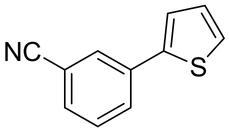
2-(Naphthalen-1-yl)furan (3m).[8] ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.43 (dd, J = 8.1, 1.6 Hz, 1H), 7.93 - 7.89 (m, 1H), 7.86 (d, J = 8.2 Hz, 1H), 7.76 (dd, J = 7.2, 1.2 Hz, 1H), 7.65 (dd, J = 1.8, 0.7 Hz, 1H), 7.54 (ddd, J = 9.3, 6.0, 4.6 Hz, 3H), 6.75 (dd, J = 3.3, 0.7 Hz, 1H), 6.61 (dd, J = 3.3, 1.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.60, 142.55, 134.08, 130.52, 128.74, 128.69, 128.64, 126.68, 126.30, 126.05, 125.69, 125.45, 111.51, 109.34; MS (ESI) m/z 217 $[M + Na]^+$.



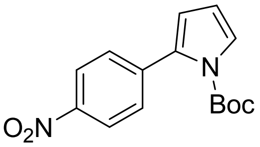
2-(4-Nitrophenyl)thiophene (3n).[3] ^{1}H NMR (300 MHz, CDCl$_3$) δ 8.24 – 8.20 (m, 2H), 7.74 – 7.71 (m, 2H), 7.47 (dd, J = 3.7, 1.1 Hz, 1H), 7.45 – 7.42 (m, 1H), 7.15 (dd, J = 5.1, 3.7 Hz, 1H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 146.69, 141.67, 140.66, 128.80, 127.79, 126.10, 125.82, 124.50; MS (ESI) m/z 228 [M + Na]$^+$.



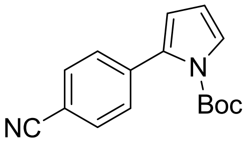
1-(4-(Thiophen-2-yl)phenyl)ethanone (3o).[5] ^{1}H NMR (300 MHz, CDCl$_3$) δ 7.96 (d, J = 8.6 Hz, 2H), 7.69 (d, J = 8.6 Hz, 2H), 7.43 (dd, J = 3.6, 1.1 Hz, 1H), 7.37 (dd, J = 5.1, 1.1 Hz, 1H), 7.12 (dd, J = 5.1, 3.7 Hz, 1H), 2.61 (s, 3H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 197.48, 143.03, 138.88, 135.82, 129.23, 128.50, 126.59, 125.76, 124.73, 26.71; MS (ESI) m/z 225 [M + Na]$^+$.



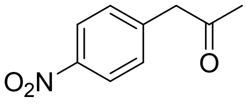
3-(Thiophen-2-yl)benzonitrile (3p).[9] ^{1}H NMR (300 MHz, CDCl$_3$) δ 7.89 – 7.85 (m, 1H), 7.83 – 7.79 (m, 1H), 7.54 (dd, J = 5.2, 3.9 Hz, 1H), 7.48 (dd, J = 11.4, 4.0 Hz, 1H), 7.40 – 7.33 (m, 2H), 7.12 (dd, J = 4.9, 3.8 Hz, 1H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 141.70, 135.73, 130.69, 130.09, 129.85, 129.25, 128.51, 126.46, 124.56, 118.69, 113.22; MS (ESI) m/z 208 [M + Na]$^+$.



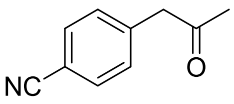
***tert*-Butyl 2-(4-nitrophenyl)-1H-pyrrole-1-carboxylate (3q)**.[6] ^{1}H NMR (300 MHz, CDCl$_3$) δ 8.24 - 8.19 (m, 2H), 7.54 - 7.49 (m, 2H), 7.41 (dd, J = 3.3, 1.8 Hz, 1H), 6.33 (dd, J = 3.4, 1.8 Hz, 1H), 6.27 (t, J = 3.3 Hz, 1H), 1.43 (s, 9H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 149.01, 146.70, 140.80, 132.88, 129.65, 124.42, 123.05, 116.65, 111.27, 84.65, 27.83; MS (ESI) m/z 311 [M + Na]$^+$.



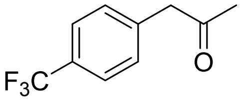
***tert*-Butyl 2-(4-cyanophenyl)-1H-pyrrole-1-carboxylate (3r)**.[3] ^{1}H NMR (300 MHz, CDCl$_3$) δ 7.66 - 7.61 (m, 2H), 7.48 - 7.43 (m, 2H), 7.39 (dd, J = 3.1, 1.9 Hz, 1H), 6.28 - 6.24 (m, 2H), 1.42 (s, 9H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 149.05, 138.92, 133.22, 131.52, 129.68, 124.08, 119.13, 116.21, 111.16, 110.64, 84.50, 27.81; MS (ESI) m/z 291 [M + Na]$^+$.



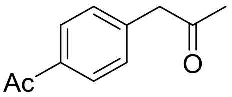
1-(4-Nitrophenyl)propan-2-one (5a).[3] ^{1}H NMR (300 MHz, CDCl$_3$) δ 8.18 (d, J = 8.7 Hz, 2H), 7.35 (d, J = 8.7 Hz, 2H), 3.85 (s, 2H), 2.24 (s, 3H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 204.38, 147.16, 141.56, 130.59, 123.87, 50.15, 30.01; MS (ESI) *m/z* 202 [M + Na]$^+$.



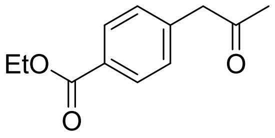
4-(2-Oxopropyl)benzonitrile (5b).[3] ^{1}H NMR (300 MHz, CDCl$_3$) δ 7.63 - 7.59 (m, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 3.79 (s, 2H), 2.21 (s, 3H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 204.50, 139.51, 132.43, 130.45, 118.79, 111.07, 50.44, 29.91; MS (ESI) *m/z* 182 [M + Na]$^+$.



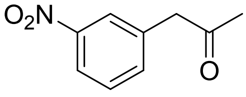
1-(4-(Trifluoromethyl)phenyl)propan-2-one (5c).[7] ^{1}H NMR (300 MHz, CDCl$_3$) δ 7.59 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 3.78 (s, 2H), 2.20 (s, 3H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 205.36, 138.18, 129.97, 125.74 (q, *J* = 3.8 Hz), 122.45, 115.57, 50.49, 29.76; MS (ESI) *m/z* 225 [M + Na]$^+$.



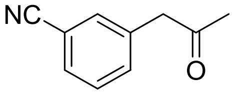
1-(4-Acetylphenyl)propan-2-one (5d).[10] ^{1}H NMR (300 MHz, CDCl$_3$) δ 7.95 - 7.91 (m, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 3.78 (s, 2H), 2.59 (s, 3H), 2.19 (s, 3H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 205.30, 197.83, 139.64, 136.11, 129.86, 128.89, 50.80, 29.76, 26.75; MS (ESI) *m/z* 199 [M + Na]$^+$.



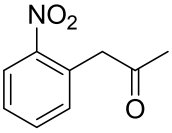
Ethyl 4-(2-oxopropyl)benzoate (5e).[11] ^{1}H NMR (300 MHz, CDCl$_3$) δ 8.01 (d, *J* = 8.4 Hz, 2H), 7.27 (d, *J* = 8.4 Hz, 2H), 4.37 (q, *J* = 7.1 Hz, 2H), 3.76 (s, 2H), 2.17 (s, 3H), 1.38 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 205.44, 166.48, 139.31, 130.09, 129.58, 129.49, 61.10, 50.90, 29.66, 14.45; MS (ESI) *m/z* 229 [M + Na]$^+$.



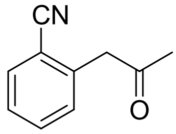
1-(3-Nitrophenyl)propan-2-one (5f).[11] ^{1}H NMR (300 MHz, CDCl$_3$) δ 8.16 - 8.09 (m, 1H), 8.07 - 8.04 (m, 1H), 7.54 - 7.49 (m, 2H), 3.85 (s, 2H), 2.25 (s, 3H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 204.50, 148.44, 135.99, 135.95, 129.60, 124.63, 122.29, 49.81, 29.96; MS (ESI) *m/z* 202 [M + Na]$^+$.



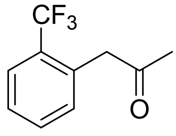
3-(2-Oxopropyl)benzonitrile (5g).[12] ^{1}H NMR (300 MHz, DMSO) δ 7.71 (ddd, *J* = 5.5, 3.6, 1.7 Hz, 1H), 7.64 (d, *J* = 0.6 Hz, 1H), 7.53 - 7.50 (m, 2H), 3.90 (s, 2H), 2.17 (s, 3H); ^{13}C NMR (75 MHz, DMSO) δ 205.23, 136.67, 134.92, 133.35, 130.34, 129.36, 118.86, 111.10, 48.43, 29.81; MS (ESI) *m/z* 182 [M + Na]$^+$.



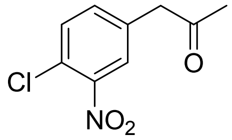
1-(2-Nitrophenyl)propan-2-one (5h).[7] ^{1}H NMR (300 MHz, CDCl$_3$) δ 8.12 (d, *J* = 8.1 Hz, 1H), 7.60 (t, *J* = 7.3 Hz, 1H), 7.46 (t, *J* = 7.5 Hz, 1H), 7.29 (s, 1H), 4.12 (s, 2H), 2.33 (s, 3H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 203.76, 148.70, 133.76, 133.67, 130.50, 128.59, 125.42), 48.72, 30.17; MS (ESI) *m/z* 202 [M + Na]$^+$.



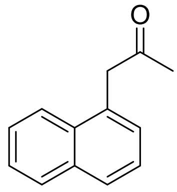
2-(2-Oxopropyl)benzonitrile (5i).[7] ^{1}H NMR (300 MHz, CDCl$_3$) δ 7.64 (dd, *J* = 7.7, 1.1 Hz, 1H), 7.55 (td, *J* = 7.7, 1.4 Hz, 1H), 7.36 (td, *J* = 7.7, 1.2 Hz, 1H), 7.29 (dd, *J* = 7.8, 0.6 Hz, 1H), 3.97 (s, 2H), 2.28 (s, 3H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 203.68, 138.18, 132.98, 132.81, 130.95, 127.75, 117.88, 113.34, 48.65, 30.16; MS (ESI) *m/z* 182 [M + Na]$^+$.



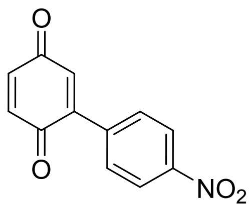
1-(2-(Trifluoromethyl)phenyl)propan-2-one (5j).[10] ^{1}H NMR (300 MHz, CDCl$_3$) δ 7.66 (d, *J* = 7.8 Hz, 1H), 7.51 (d, *J* = 7.5 Hz, 1H), 7.39 (d, *J* = 7.7 Hz, 1H), 7.27 (d, *J* = 7.6 Hz, 1H), 3.91 (d, *J* = 1.0 Hz, 2H), 2.20 (s, 3H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 204.79, 132.88 (d, *J* = 1.8 Hz), 132.83, 132.02 (d, *J* = 1.0 Hz), 128.89 (q, *J* = 29.8 Hz), 127.40, 126.22 (q, *J* = 5.5 Hz), 122.62, 47.45 (d, *J* = 1.7 Hz), 29.71; MS (ESI) *m/z* 225 [M + Na]$^+$.



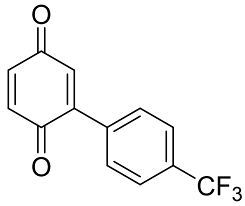
1-(4-Chloro-3-nitrophenyl)propan-2-one (5k). ^{1}H NMR (300 MHz, CDCl$_3$) δ 8.12 (d, *J* = 2.2 Hz, 1H), 7.56 (dd, *J* = 8.2, 2.2 Hz, 1H), 7.22 (d, *J* = 8.2 Hz, 1H), 4.10 (s, 2H), 2.33 (s, 3H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 203.22, 148.99, 134.67, 134.33, 133.75, 128.93, 125.57, 48.19, 30.15; HRMS (ESI): Calcd for $C_9H_8ClNNaO_3$ [M + Na]$^+$ 236.0085, found 236.0097.



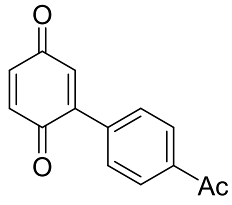
1-(Naphthalen-1-yl)propan-2-one (5l).[13] ^{1}H NMR (300 MHz, CDCl$_3$) δ 7.91 - 7.86 (m, 2H), 7.82 (d, *J* = 8.1 Hz, 1H), 7.55 - 7.49 (m, 2H), 7.47 - 7.38 (m, 2H), 4.12 (s, 2H), 2.12 (s, 3H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 207.16, 134.02, 132.29, 131.19, 128.92, 128.36, 128.22, 126.69, 126.06, 125.72, 123.94, 49.38, 29.11; MS (ESI) *m/z* 207 [M + Na]$^+$.



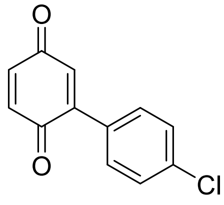
4'-Nitro-[1,1'-biphenyl]-2,5-dione (7a).[3] ^{1}H NMR (300 MHz, CDCl$_3$) δ 8.30 (d, *J* = 8.8 Hz, 2H), 7.66 (d, *J* = 8.8 Hz, 2H), 6.97 - 6.87 (m, 3H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 186.98, 185.75, 148.74, 144.03, 138.88, 137.11, 136.71, 134.21, 130.43, 123.75; MS (ESI) *m/z* 230 [M + H]$^+$.



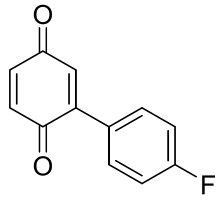
4'-(Trifluoromethyl)-[1,1'-biphenyl]-2,5-dione (7b).[14] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.2 Hz, 2H), 7.59 (d, *J* = 8.1 Hz, 2H), 6.92 - 6.84 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.29, 186.12, 144.79, 137.10, 136.56, 136.18 (d, *J* = 1.2 Hz), 133.72, 131.97 (q, *J* = 32.7 Hz), 129.75, 125.57 (q, *J* = 3.7 Hz), 123.89 (q, *J* = 270.7 Hz); MS (ESI) *m/z* 253 $[M + H]^+$.



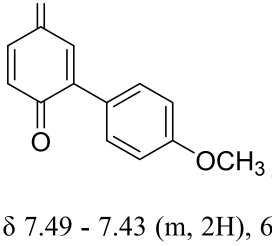
4'-Acetyl-[1,1'-biphenyl]-2,5-dione (7c).[15] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.99 (d, *J* = 7.7 Hz, 2H), 7.55 (d, *J* = 7.7 Hz, 2H), 6.87 (d, *J* = 4.9 Hz, 3H), 2.61 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.53, 187.30, 186.17, 144.98, 137.93, 137.10, 136.47, 133.50, 129.61, 128.42, 26.83; MS (ESI) *m/z* 227 $[M + H]^+$.



4'-Chloro-[1,1'-biphenyl]-2,5-dione (7d).[14] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45 - 7.38 (m, 4H), 6.89 - 6.80 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.44, 186.42, 144.81, 137.11, 136.64, 136.45, 132.76, 131.08, 130.66, 128.96; MS (ESI) *m/z* 219 $[M + H]^+$.

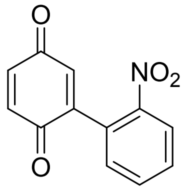


4'-Fluoro-[1,1'-biphenyl]-2,5-dione (7e).[15] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.53 - 7.45 (m, 2H), 7.18 - 7.10 (m, 2H), 6.90 - 6.80 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.57, 186.68, 165.76, 144.91, 137.13, 136.47, 132.57, 131.43 (d, *J* = 8.5 Hz), 128.77 (d, *J* = 3.4 Hz), 116.04; MS (ESI) *m/z* 203 $[M + H]^+$.

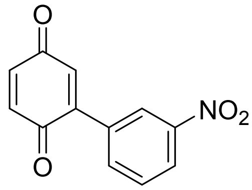


4'-Methoxy-[1,1'-biphenyl]-2,5-dione (7f).[14] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49 - 7.43 (m, 2H), 6.98 - 6.92 (m, 2H), 6.85 - 6.76 (m, 3H), 3.84 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.77, 187.17, 161.44, 145.24, 137.09, 136.32, 131.15, 130.96, 125.03, 114.20, 55.47;

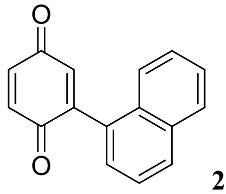
MS (ESI) *m/z* 215 [M + H]$^+$.



2'-Nitro-[1,1'-biphenyl]-2,5-dione (7g). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.19 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.74 (td, *J* = 7.5, 1.4 Hz, 1H), 7.65 (td, *J* = 7.8, 1.6 Hz, 1H), 7.39 (dd, *J* = 7.5, 1.5 Hz, 1H), 6.87 (d, *J* = 1.8 Hz, 2H), 6.82 (dd, *J* = 1.7, 0.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.06, 184.65, 148.27, 147.04, 137.09, 136.78, 134.27, 131.85, 131.37, 131.02, 128.89, 124.94; HRMS (ESI): Calcd for $C_{12}H_8NO_4$ [M + H]$^+$ 230.0448, found 230.0462.



3'-Nitro-[1,1'-biphenyl]-2,5-dione (7h). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.36 - 8.26 (m, 2H), 7.84 - 7.77 (m, 1H), 7.64 (t, *J* = 8.0 Hz, 1H), 6.96 - 6.85 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.00, 185.77, 148.26, 143.69, 137.01, 136.66, 135.19, 134.17, 133.86, 129.72, 124.71, 124.33; MS (ESI) *m/z* 230 [M + H]$^+$.



2-(Naphthalen-1-yl)cyclohexa-2,5-diene-1,4-dione (7i).[15] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.96 - 7.88 (m, 2H), 7.63 - 7.59 (m, 1H), 7.54 - 7.44 (m, 3H), 7.35 (dd, *J* = 7.1, 1.1 Hz, 1H), 6.97 - 6.89 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.70, 186.65, 147.44, 137.16, 136.66, 135.68, 133.62, 131.34, 131.01, 130.21, 128.73, 127.61, 126.83, 126.39, 125.27, 125.18; MS (ESI) *m/z* 235 [M + H]$^+$.

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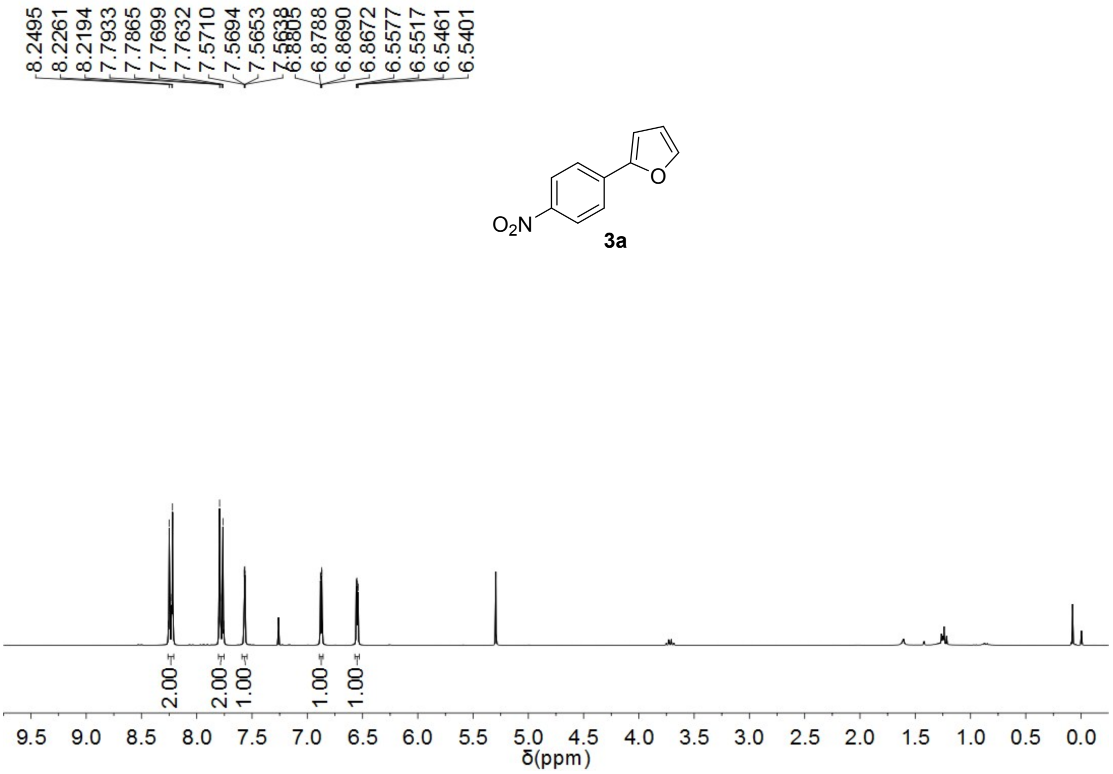
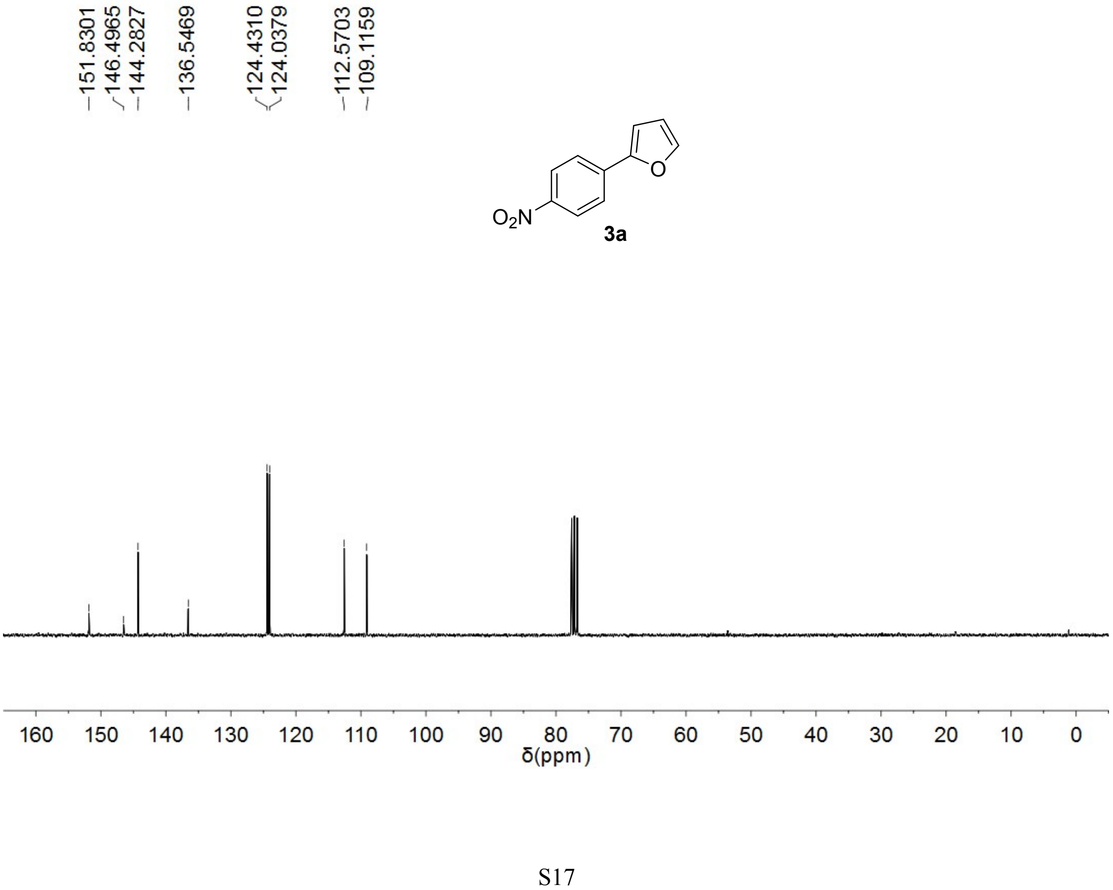
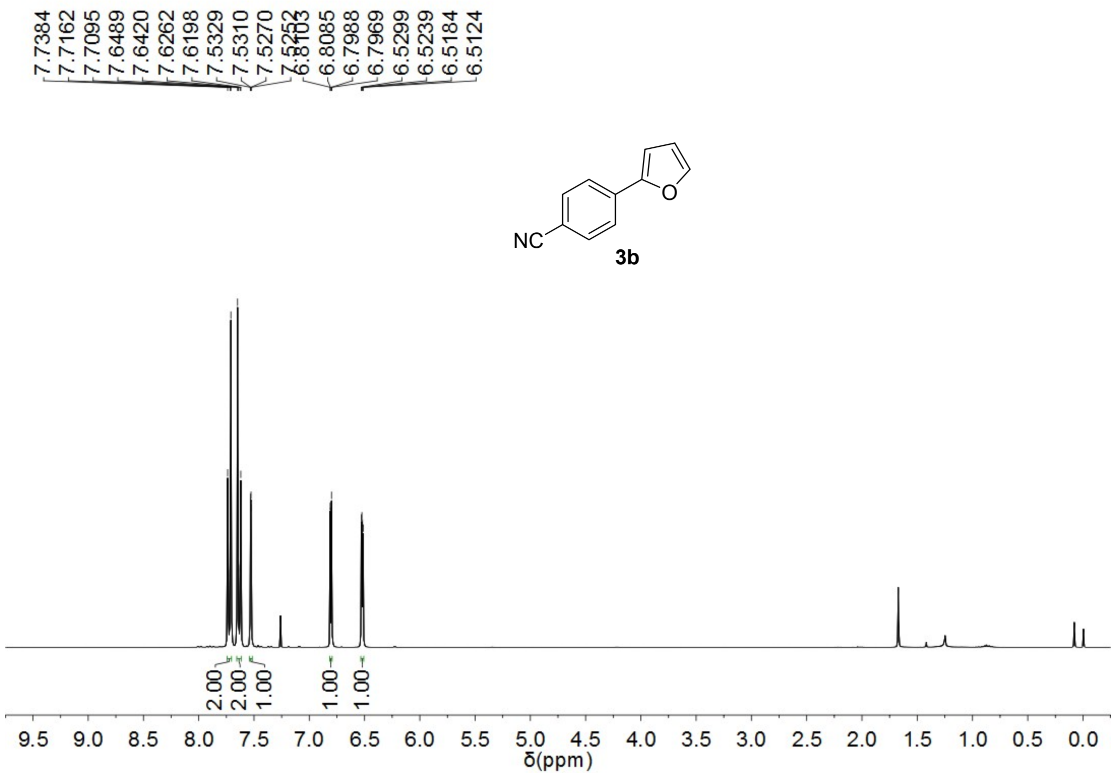
3 Copies of ^{1}H NMR and ^{13}C NMR spectra



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1.00
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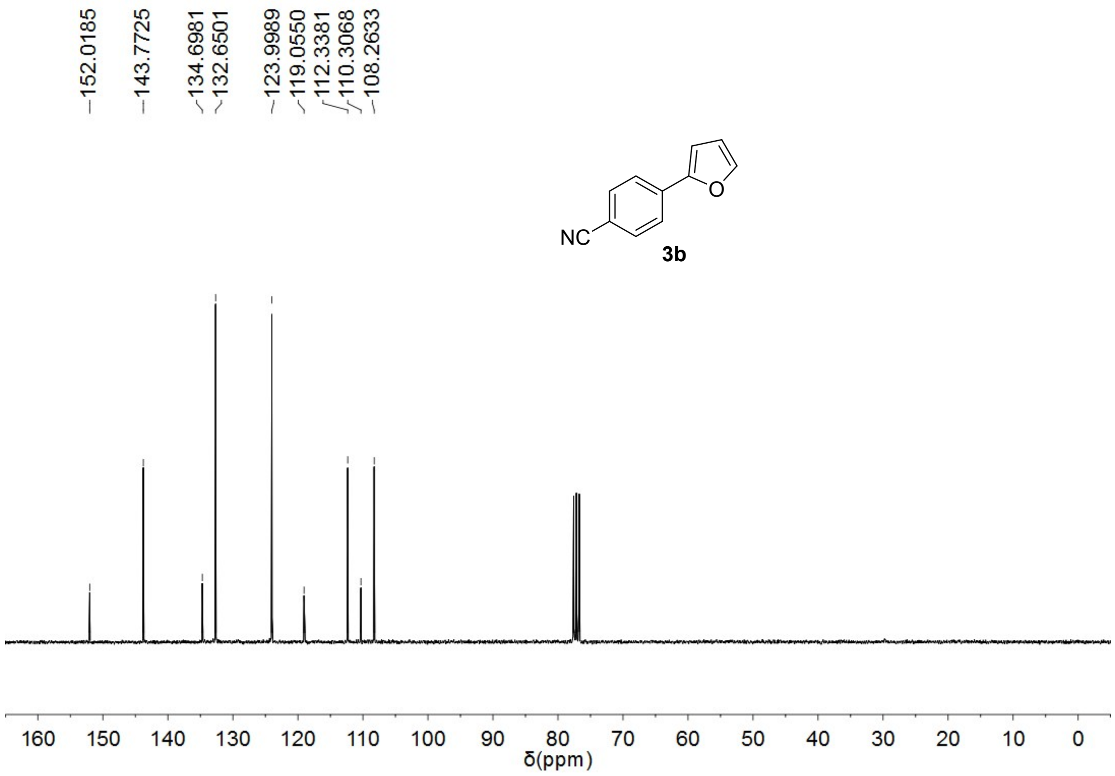
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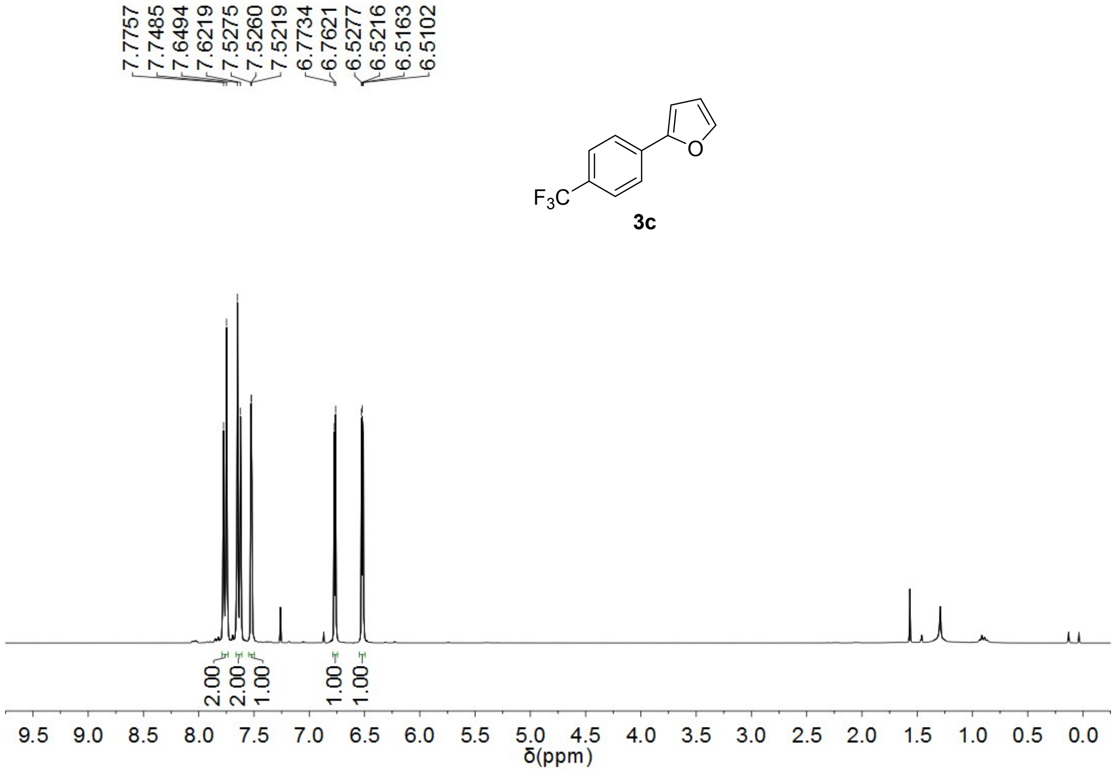
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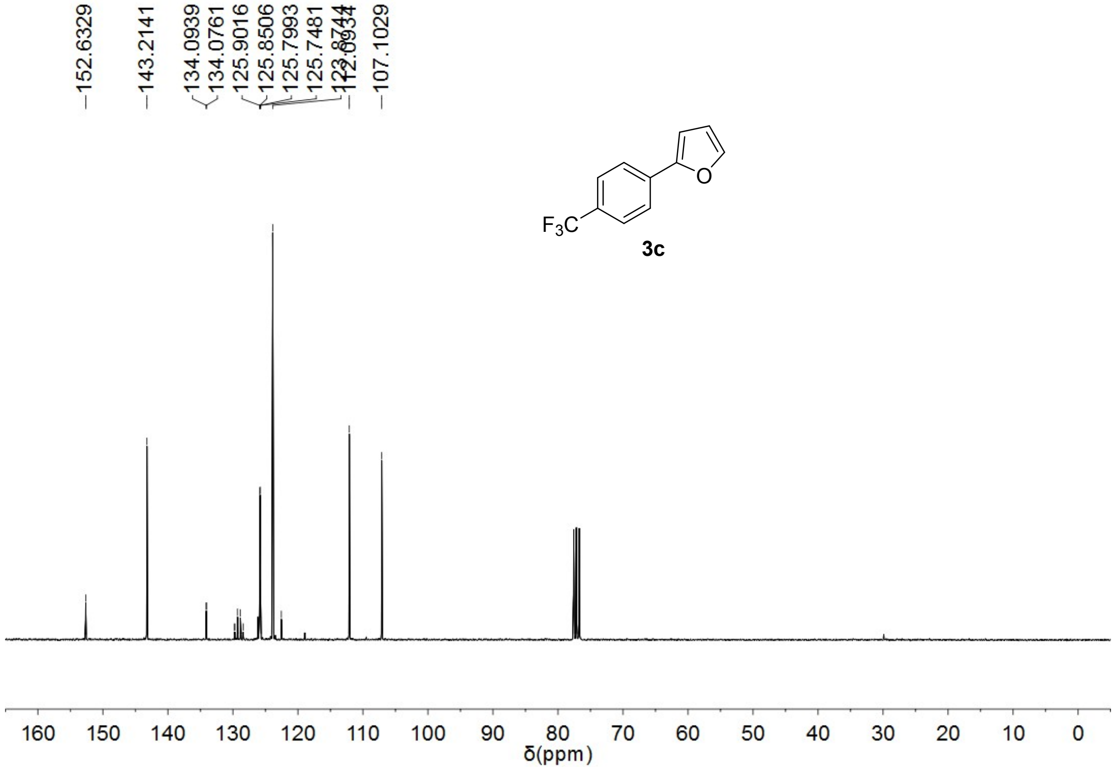
3c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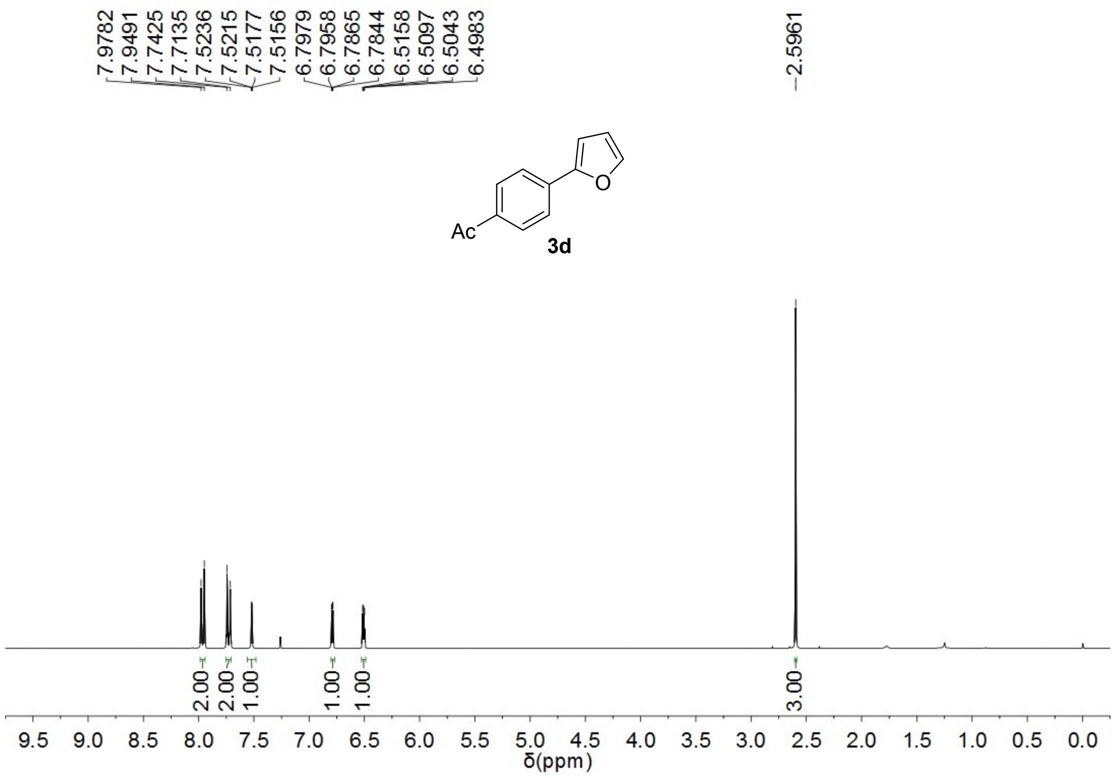
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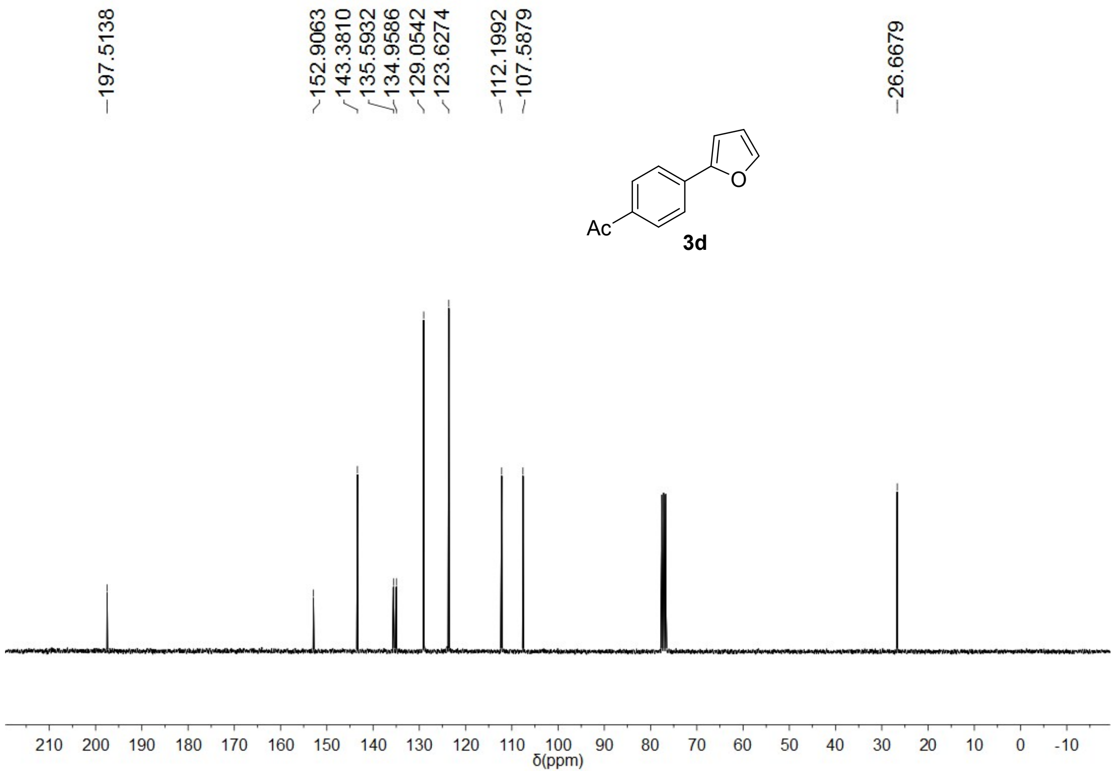
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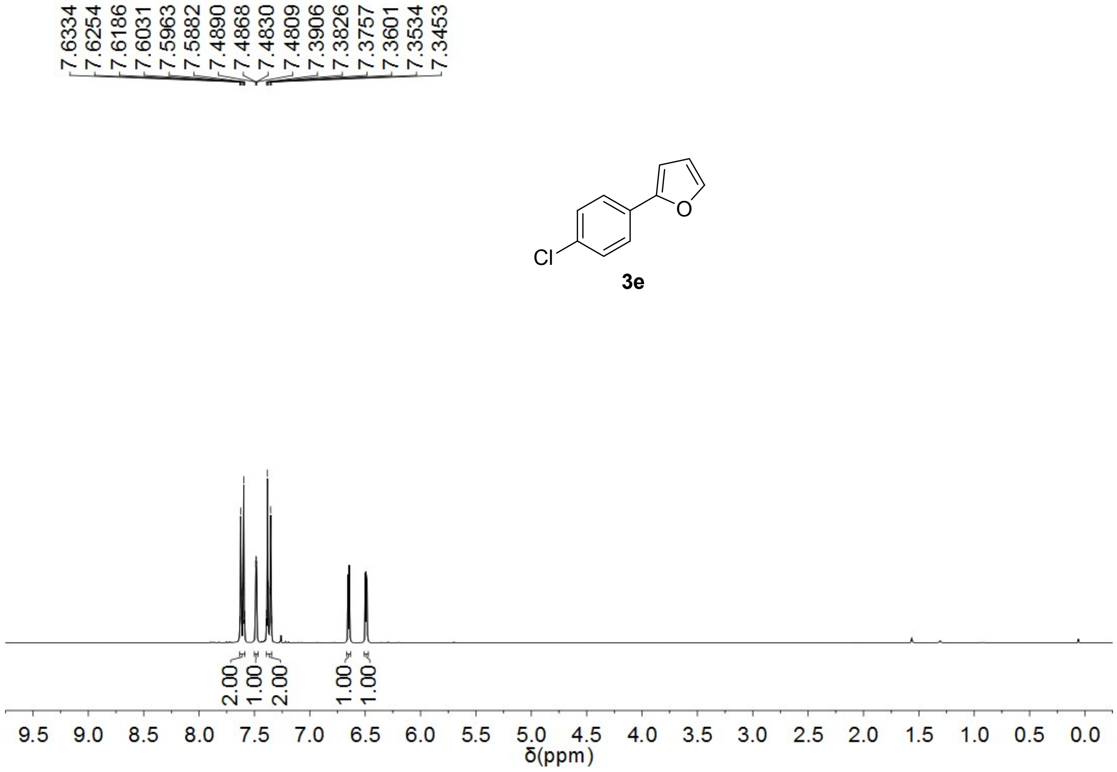
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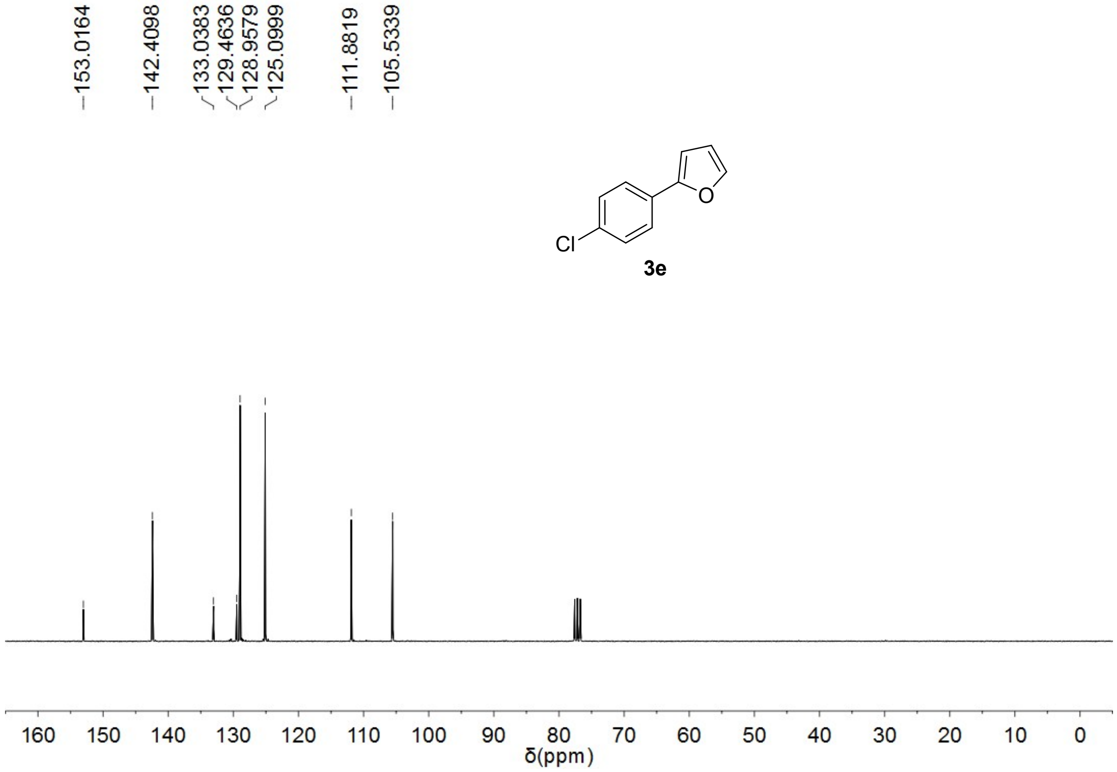
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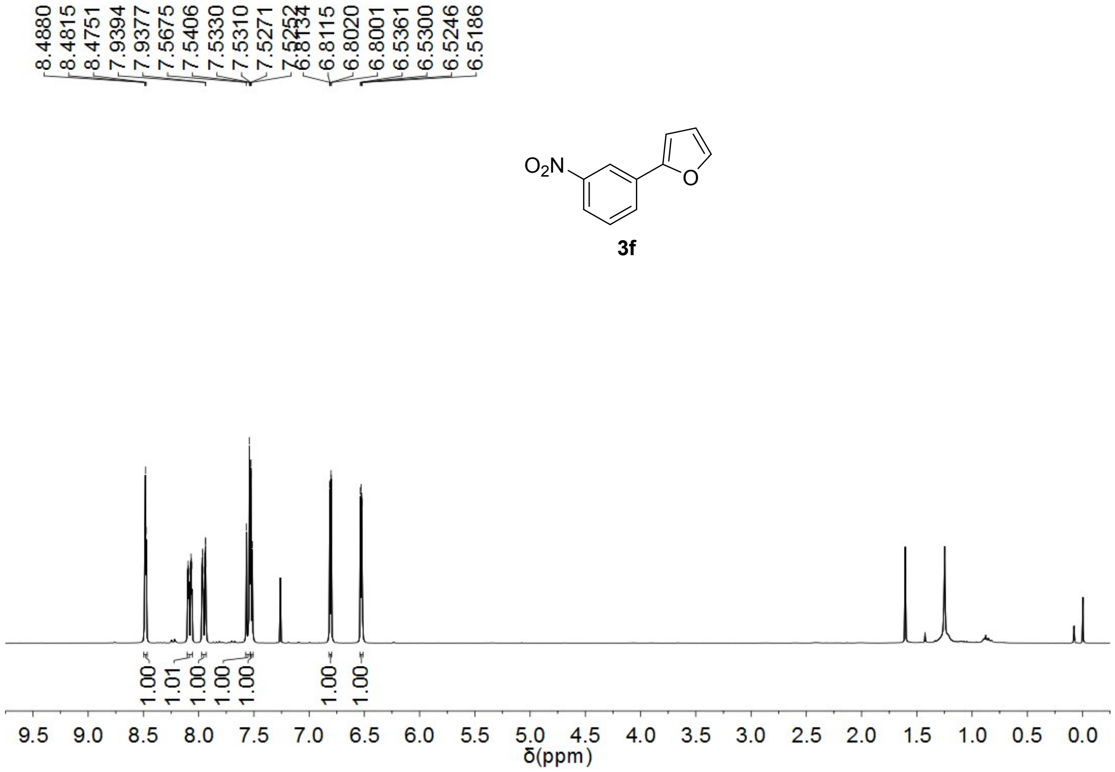
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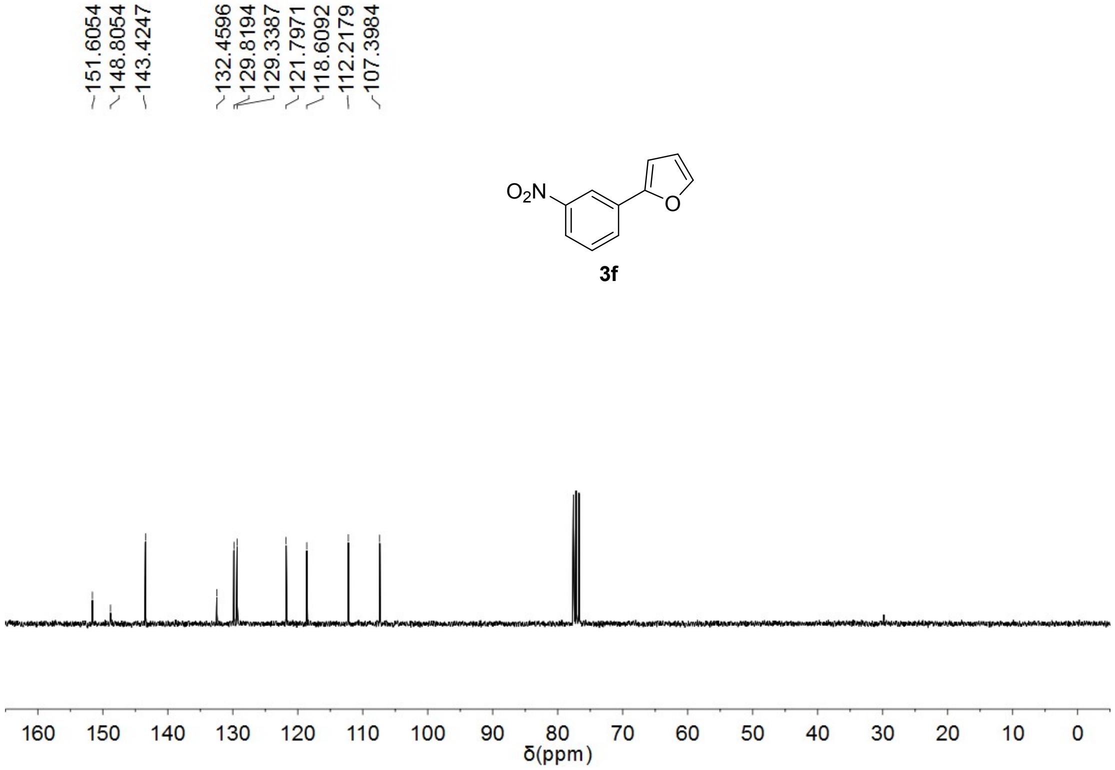
3f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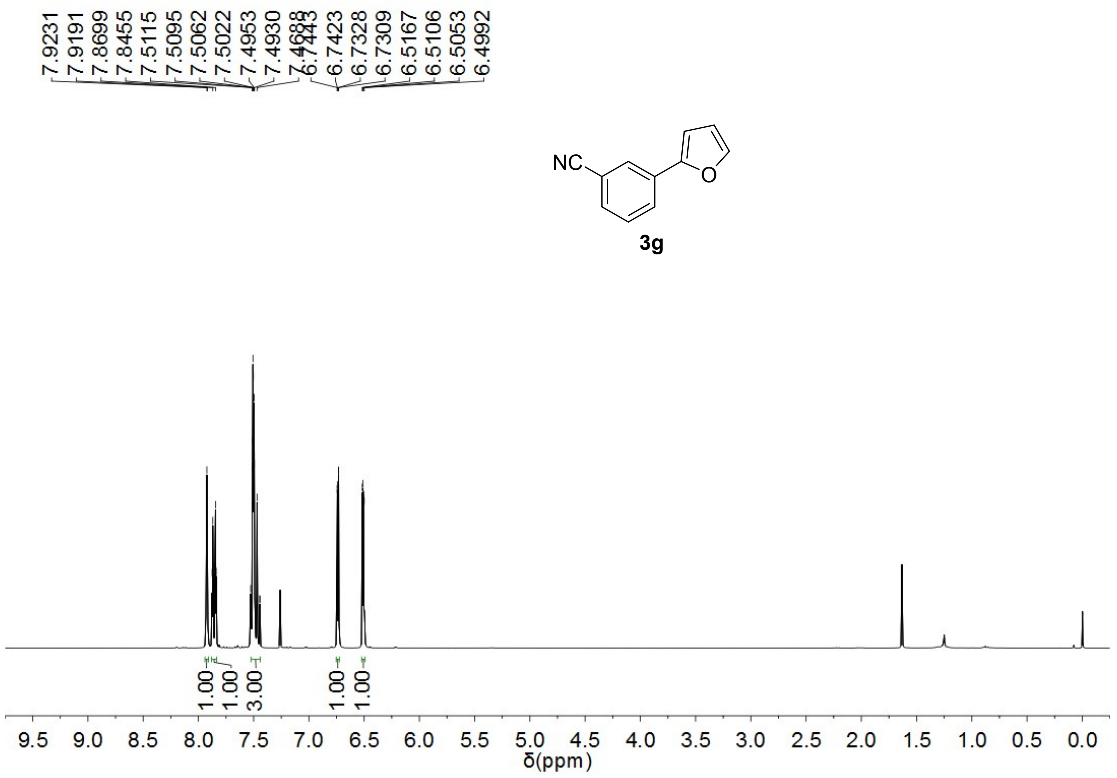
3f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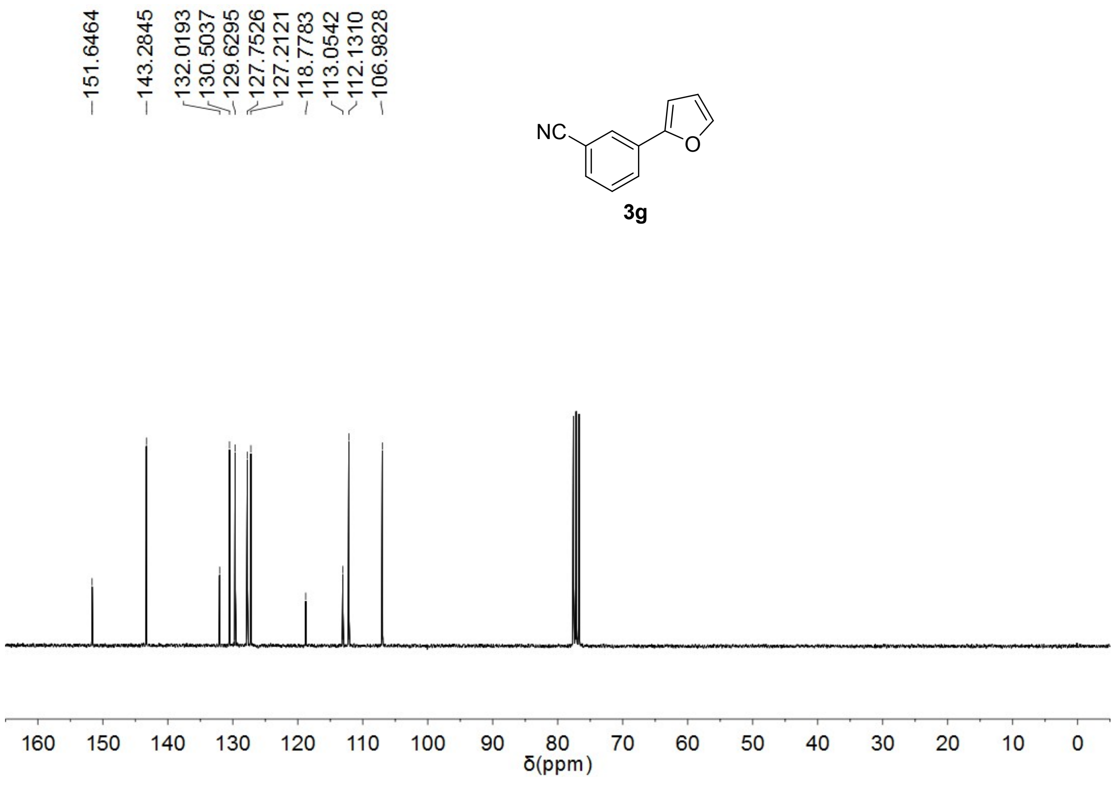
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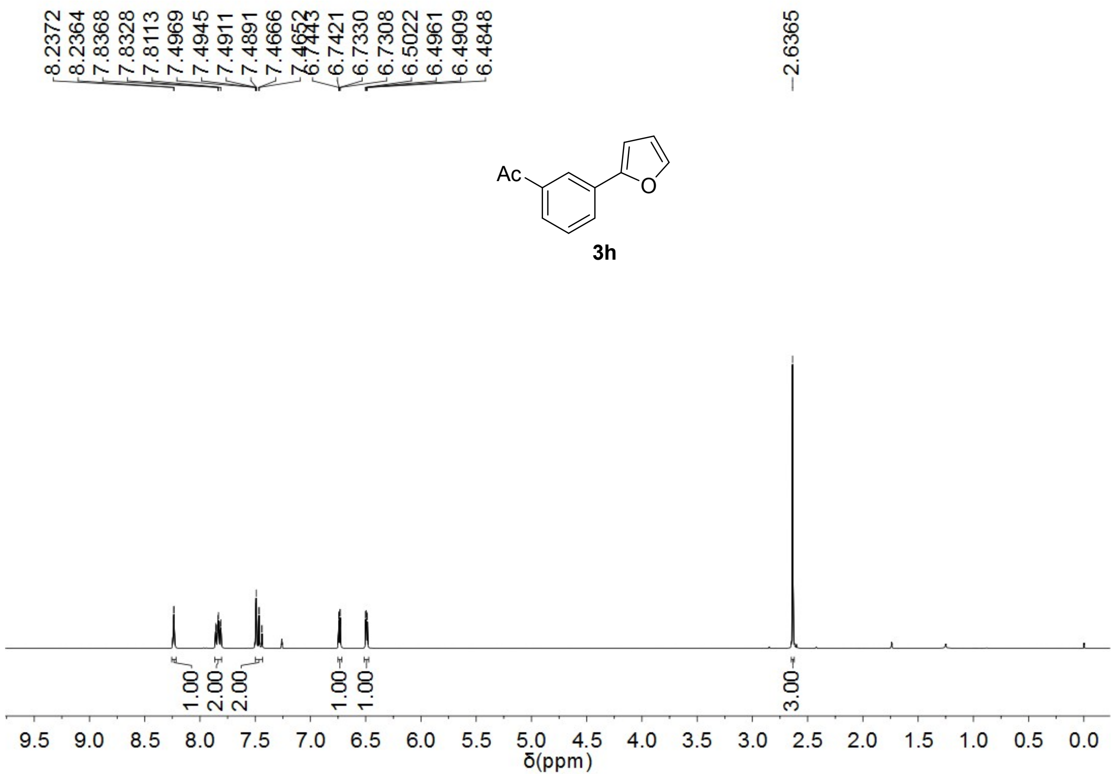
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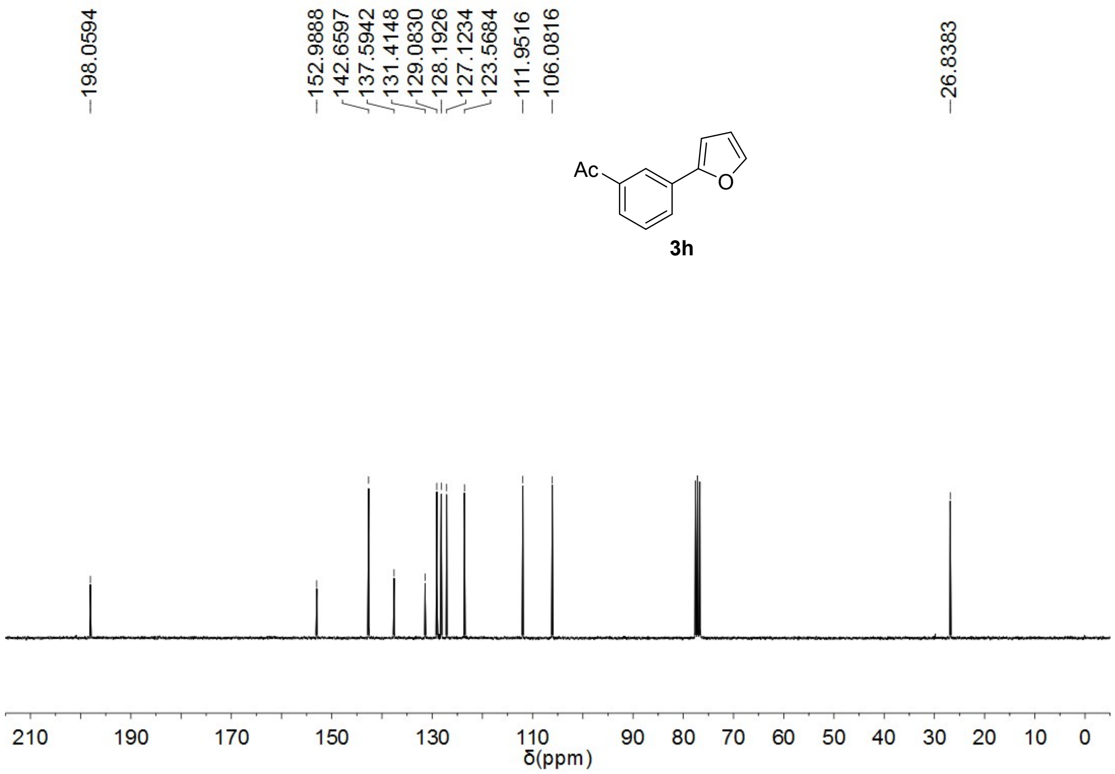
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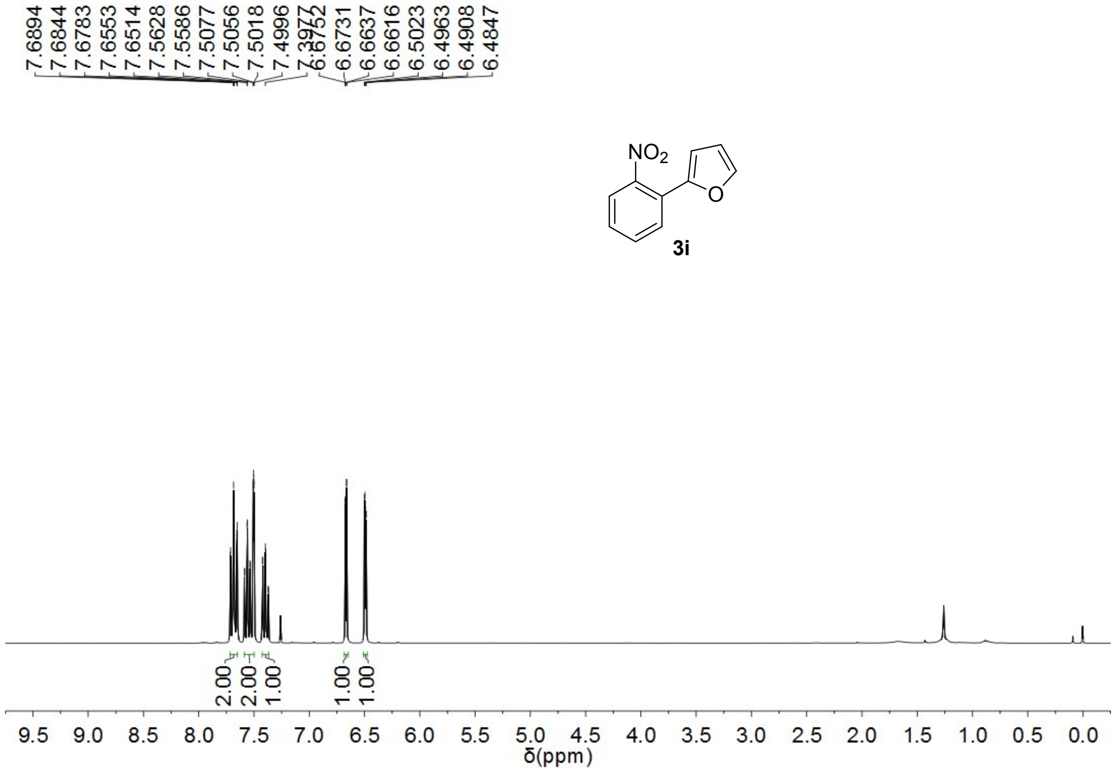
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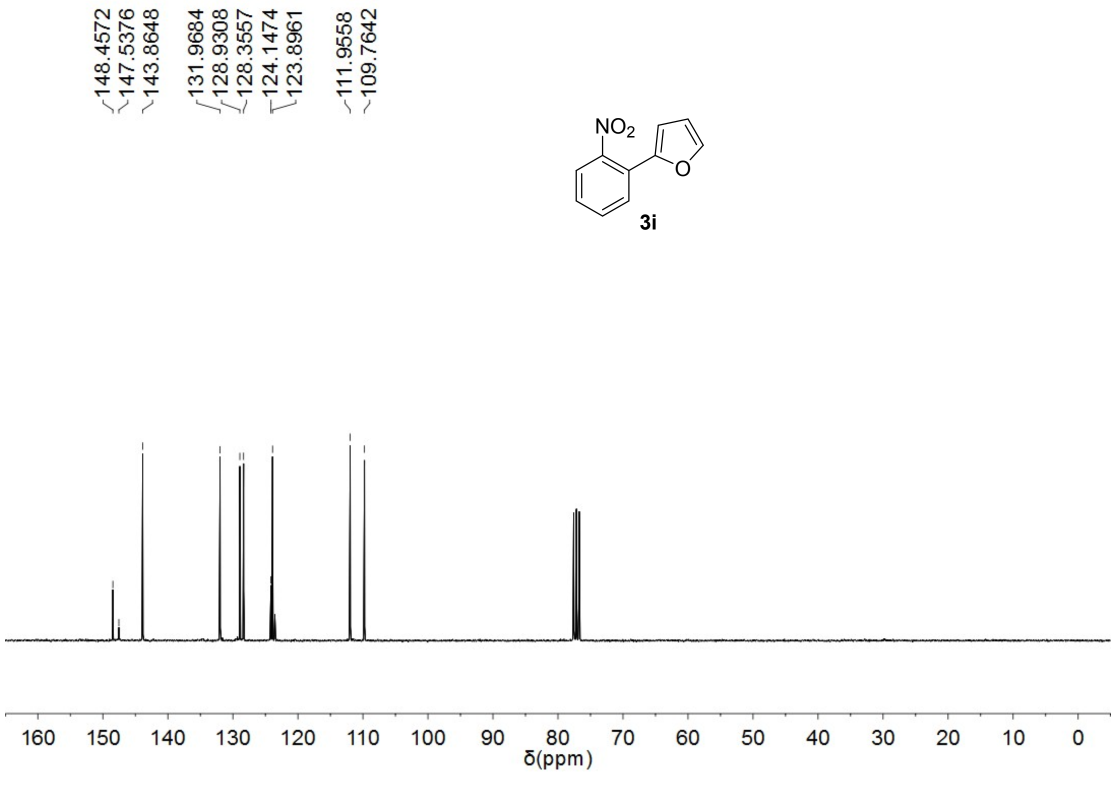
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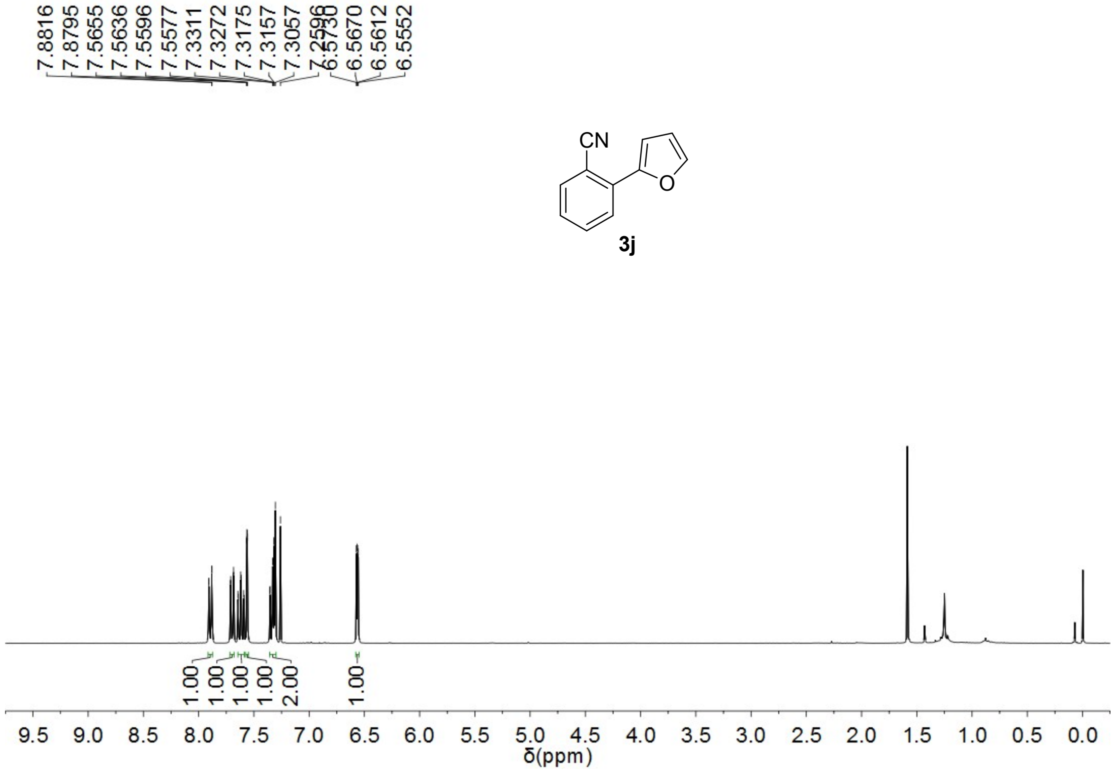
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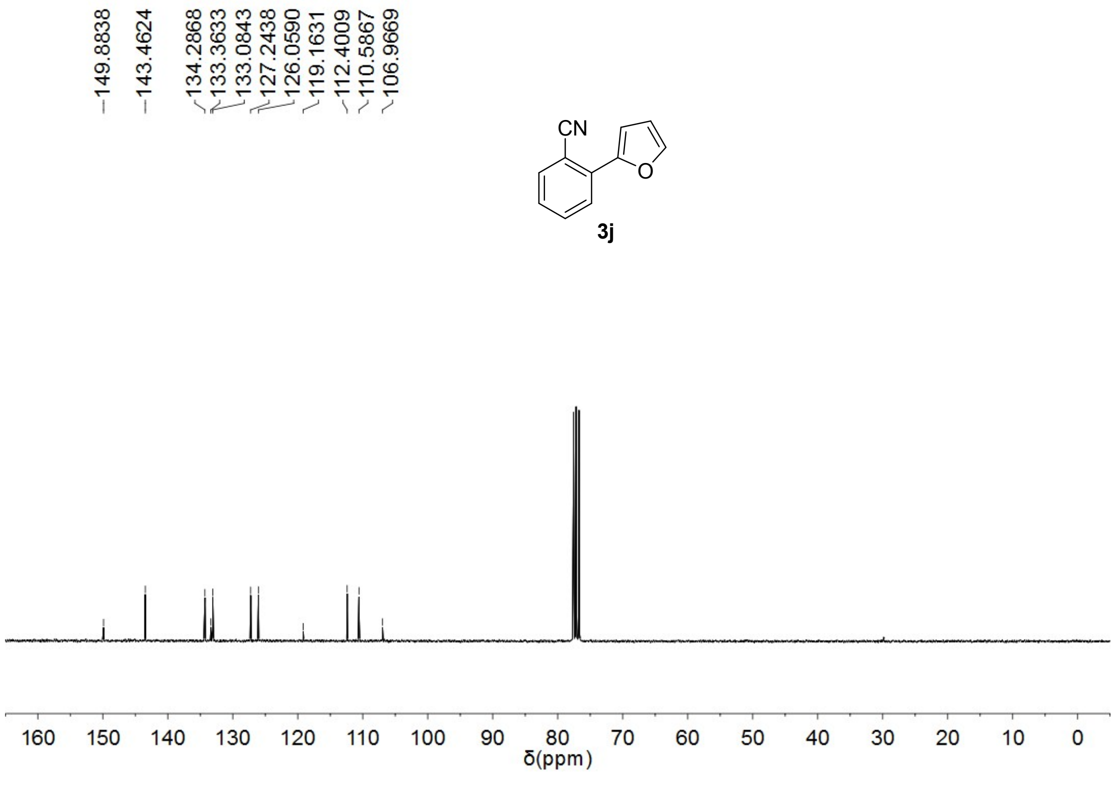
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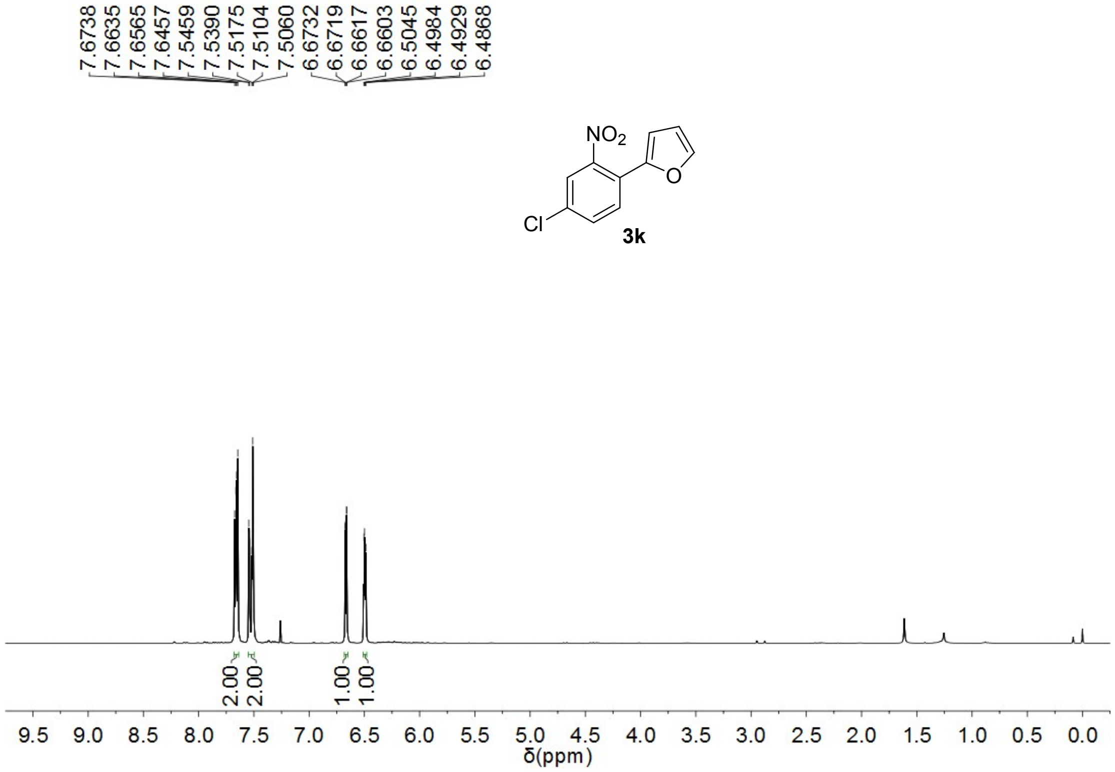
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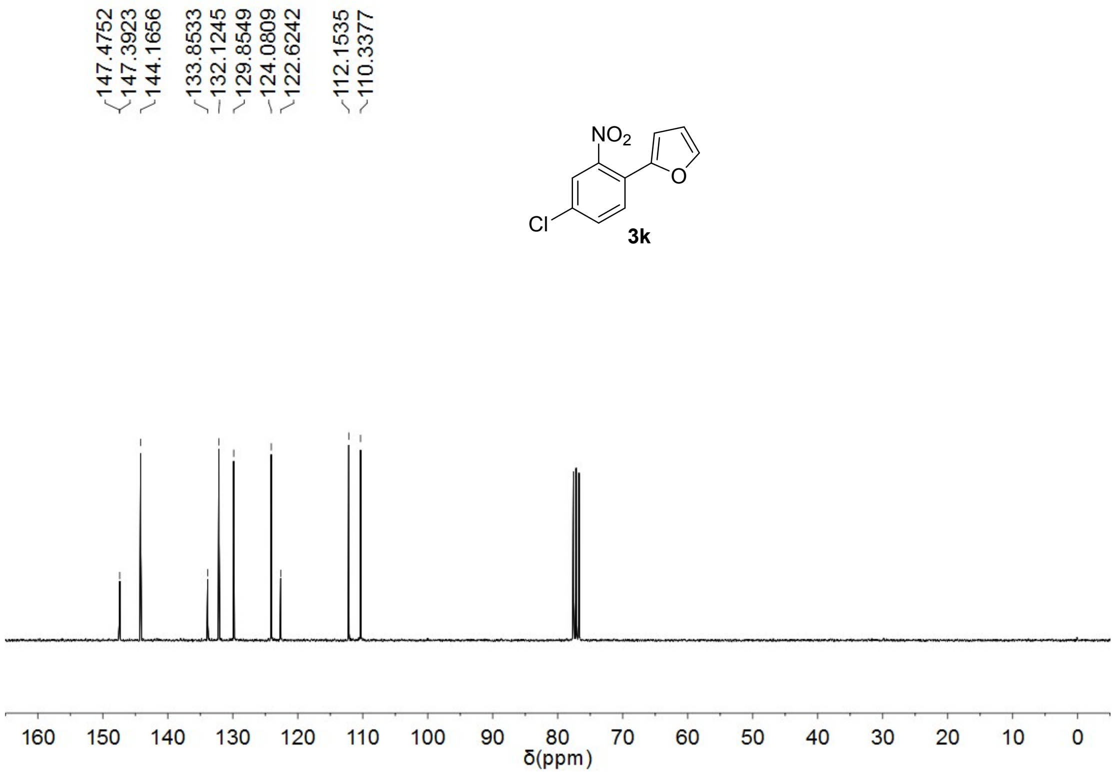
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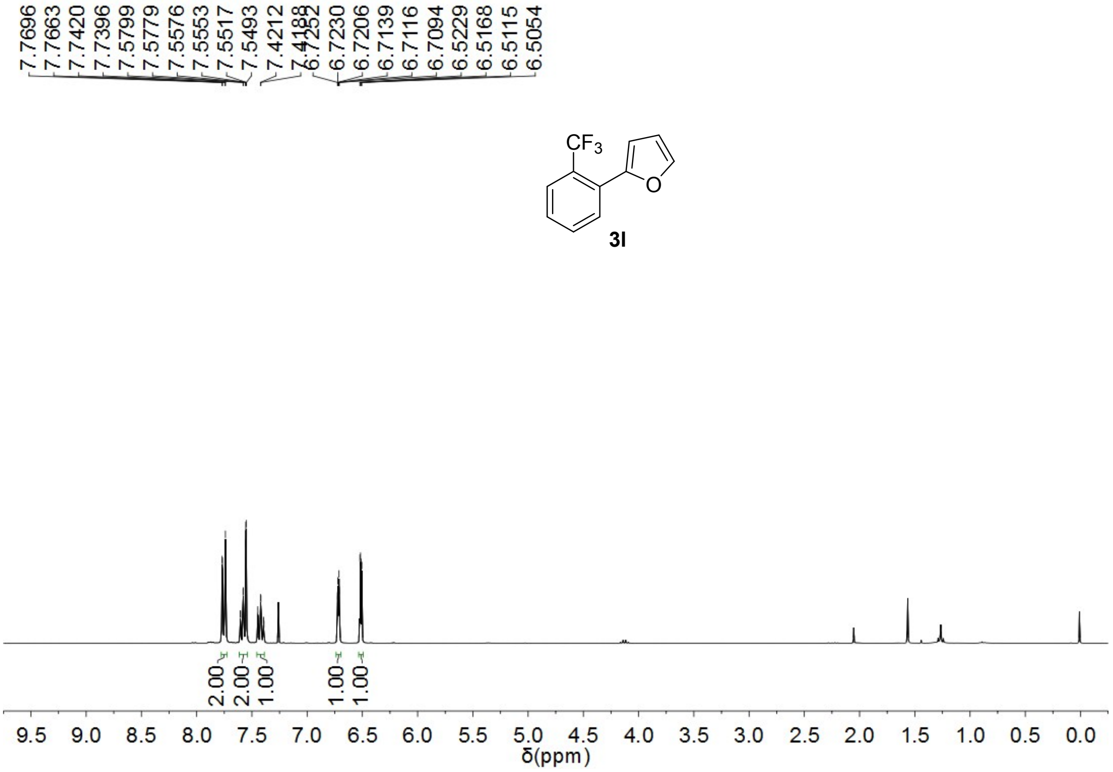
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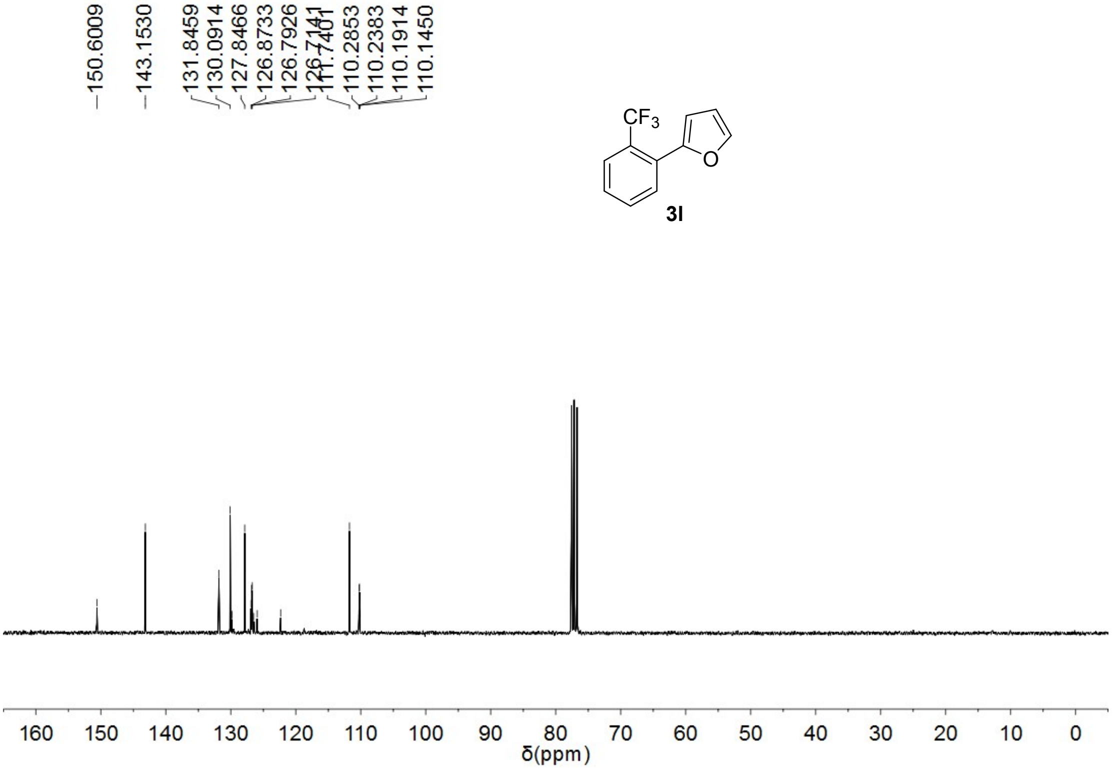
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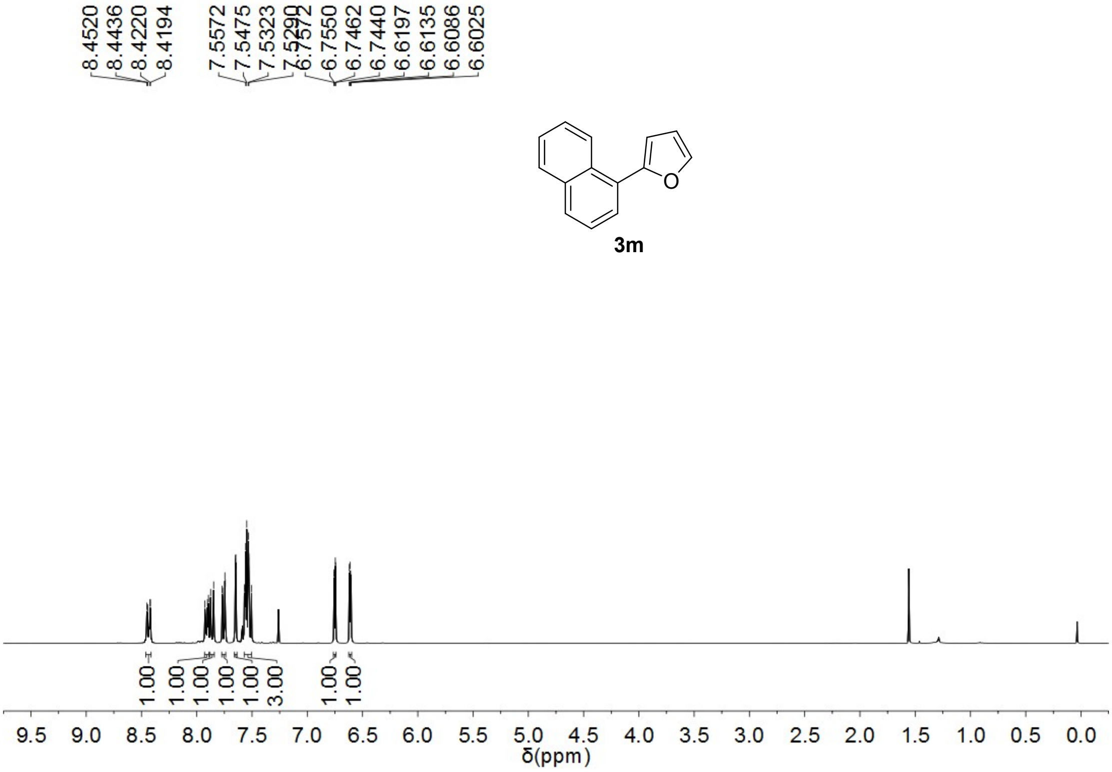
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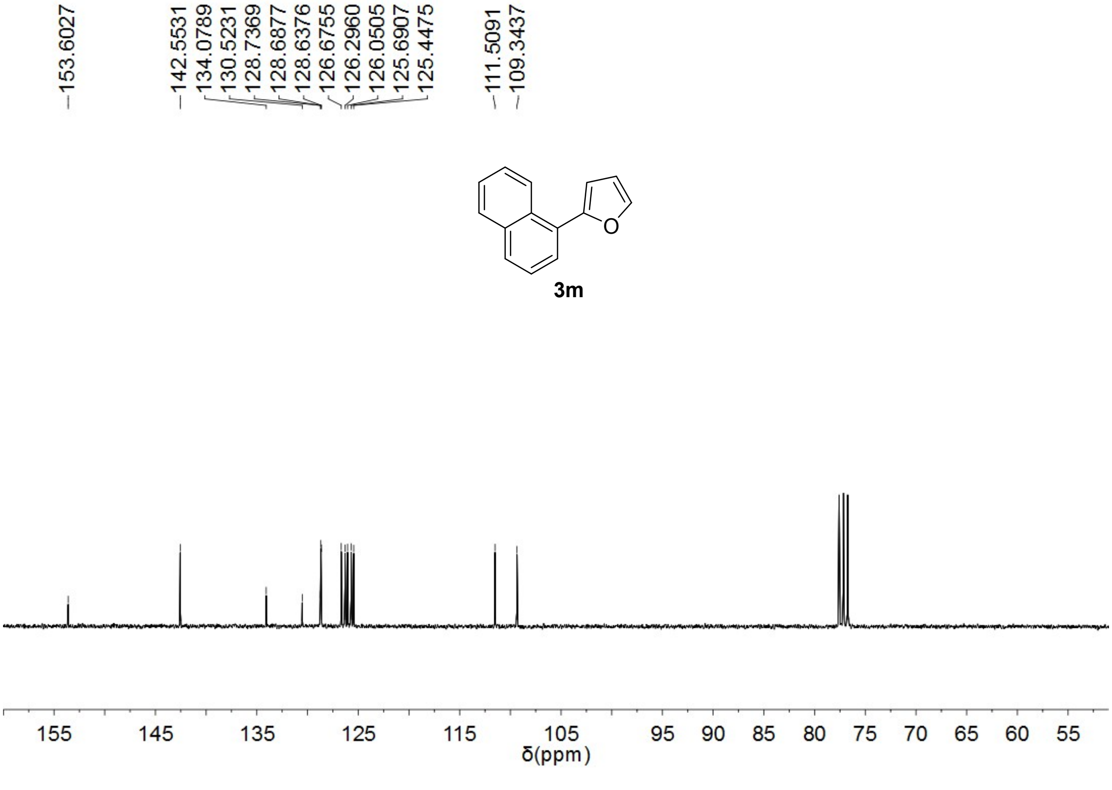
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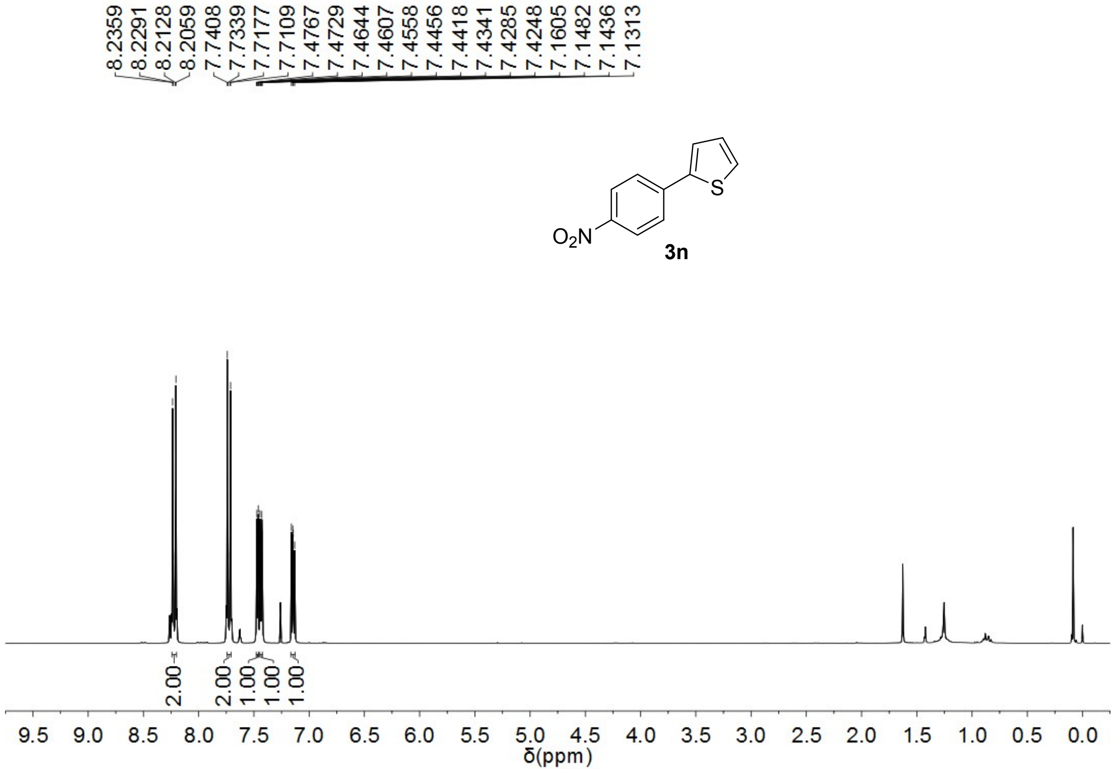
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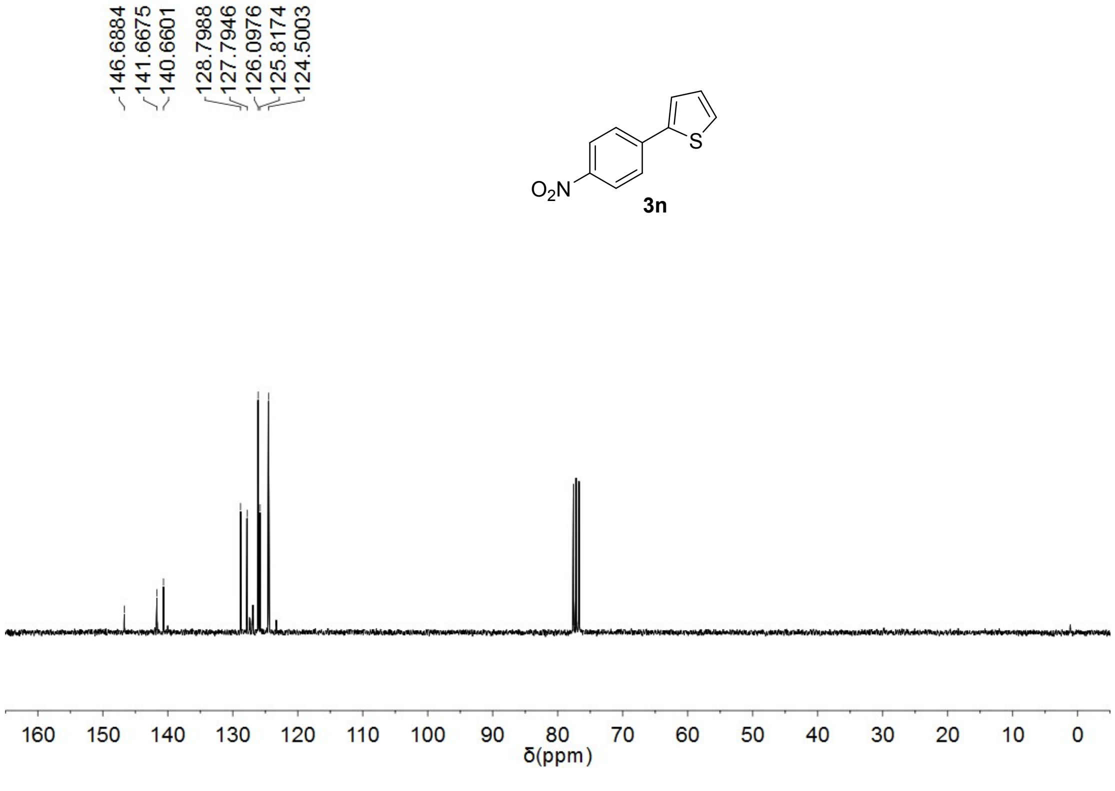
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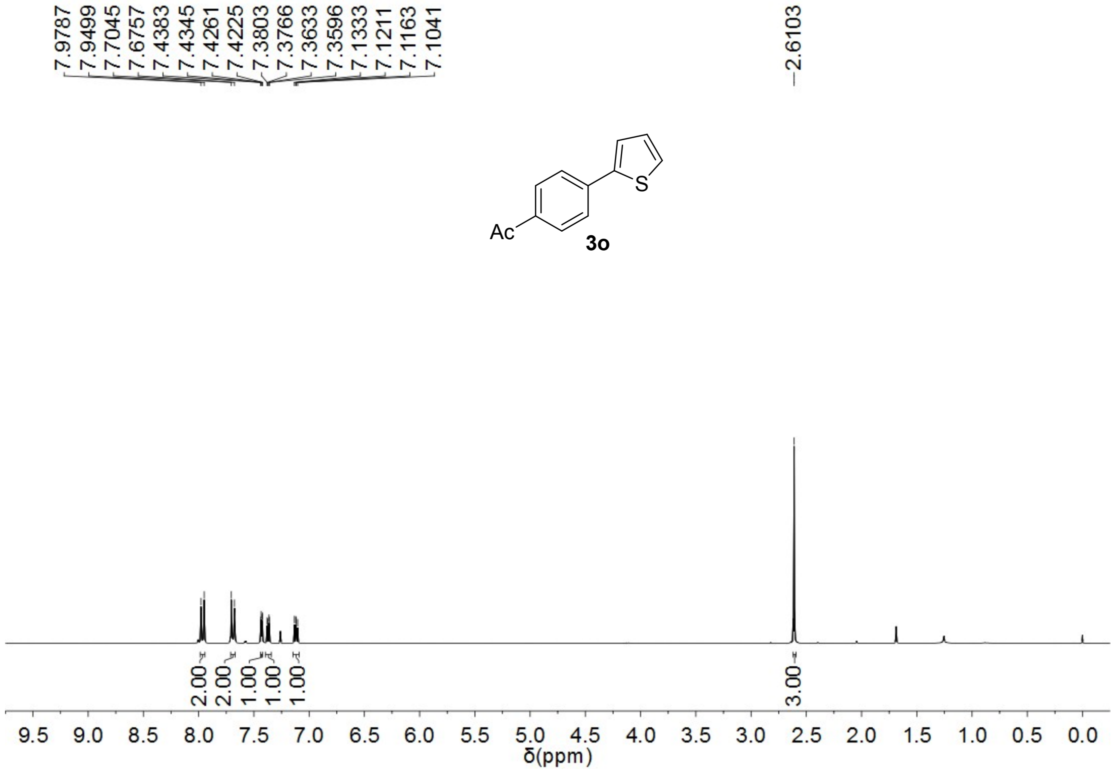
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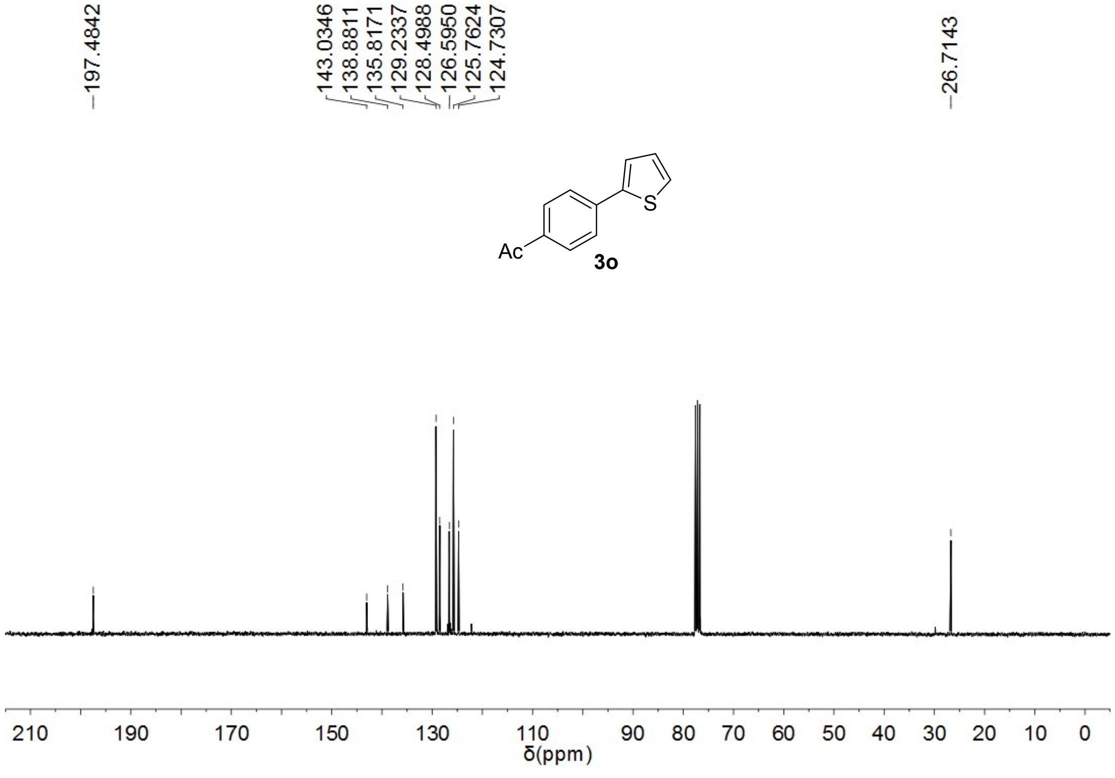
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128.4988
126.5950
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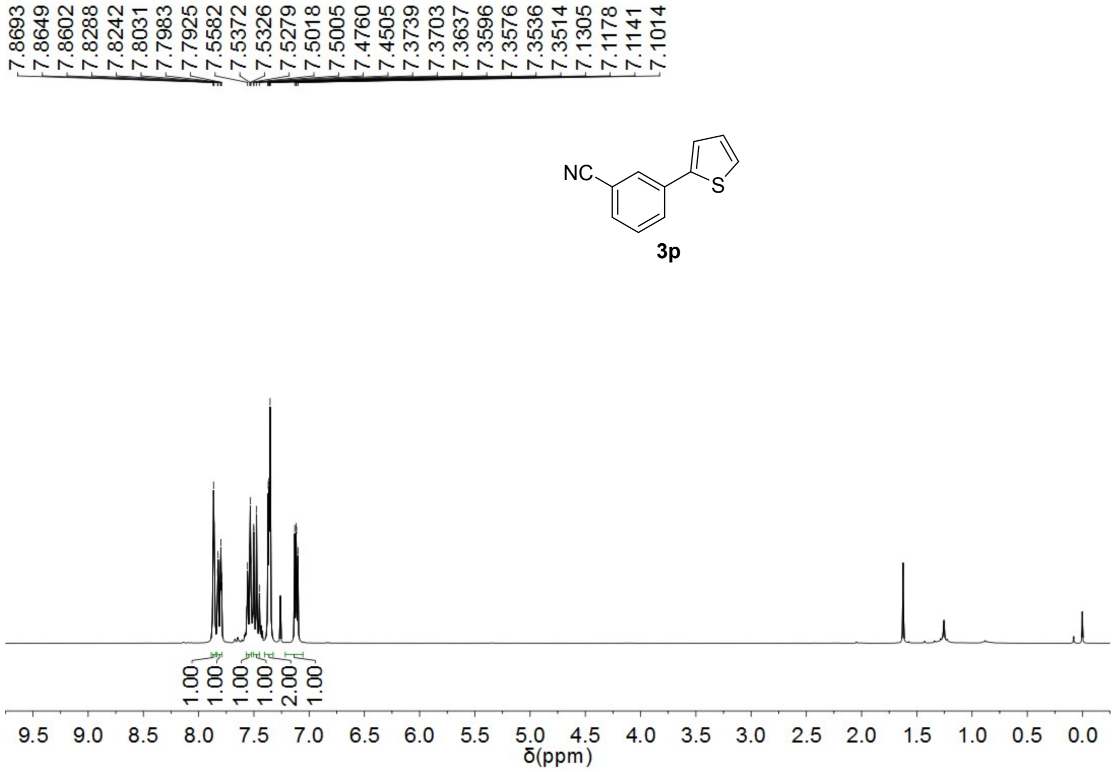
124.7307
26.7143
Ac
S
3o
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
δ(ppm)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7.8242
7.8031
7.7983
7.7925
7.5582
7.5372
7.5326
7.5279
7.5018
7.5005
7.4760
7.4505
7.3739
7.3703
7.3637
7.3596
7.3576
7.3536
7.3514
7.1305
7.1178
7.1141
7.1014
NC
S
3p
1.00
1.00
1.00
1.00
2.00
1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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3.5
3.0
2.5
2.0
1.5
1.0
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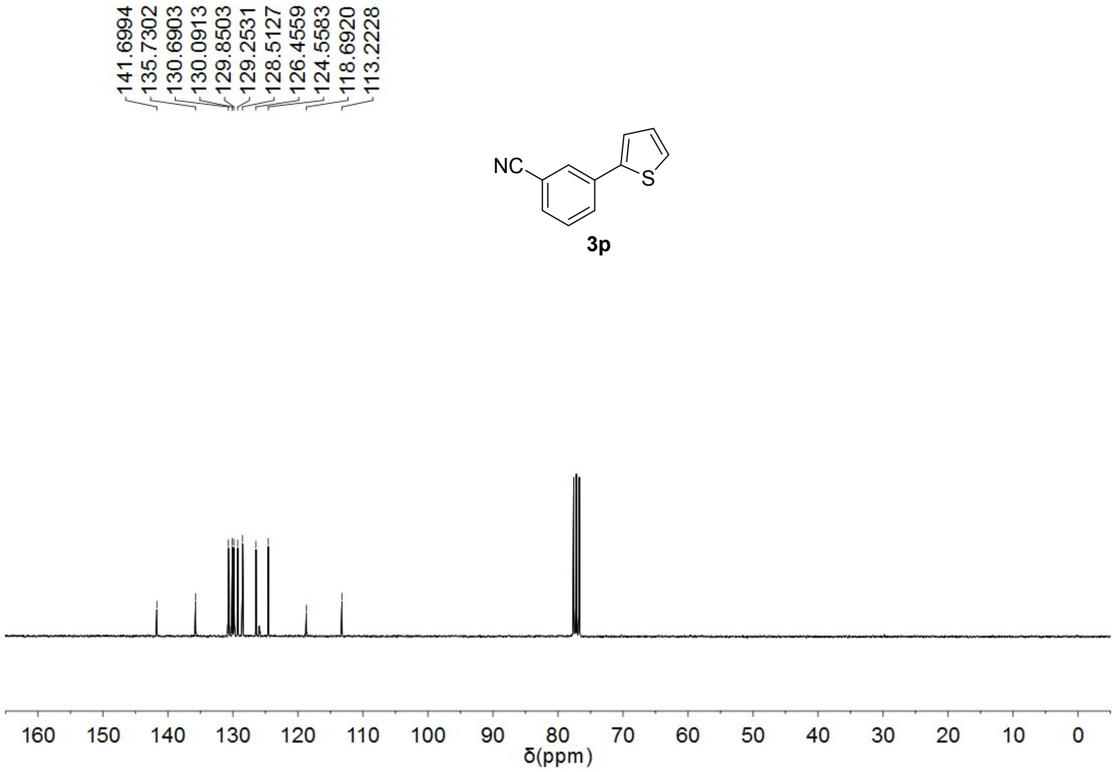
0.0
δ(ppm)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135.7302
130.6903
130.0913
129.8503
129.2531
128.5127
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113.2228
NC
S
3p
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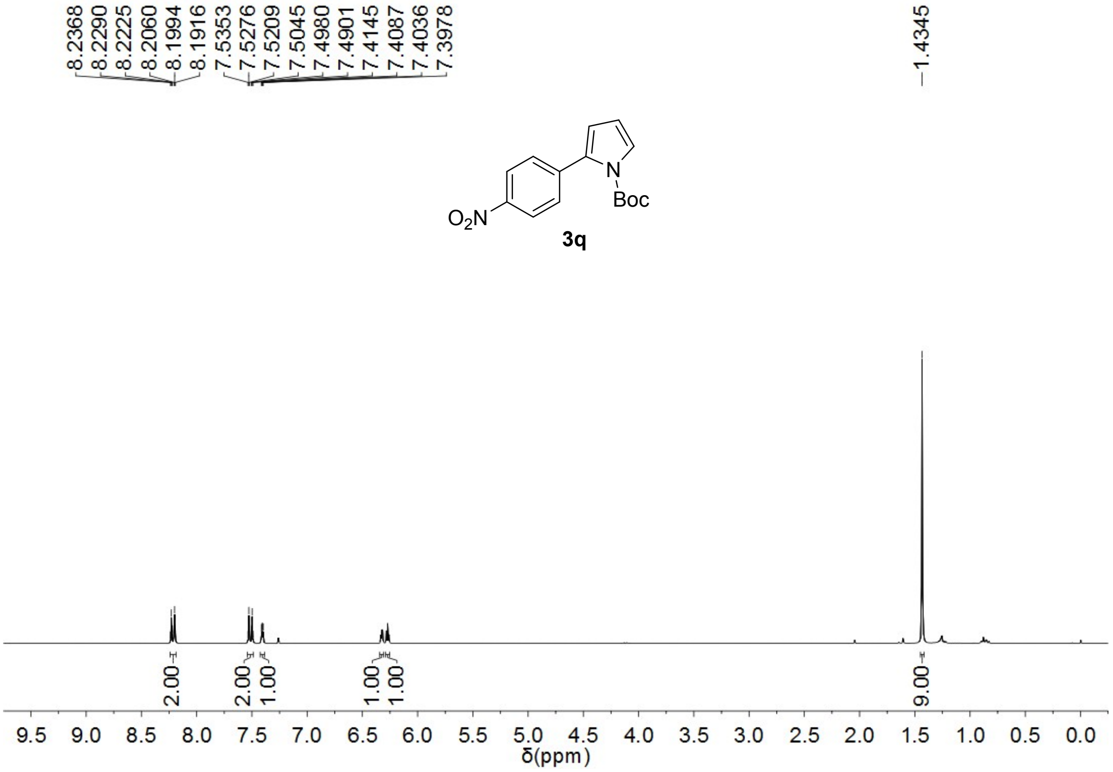
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ(ppm)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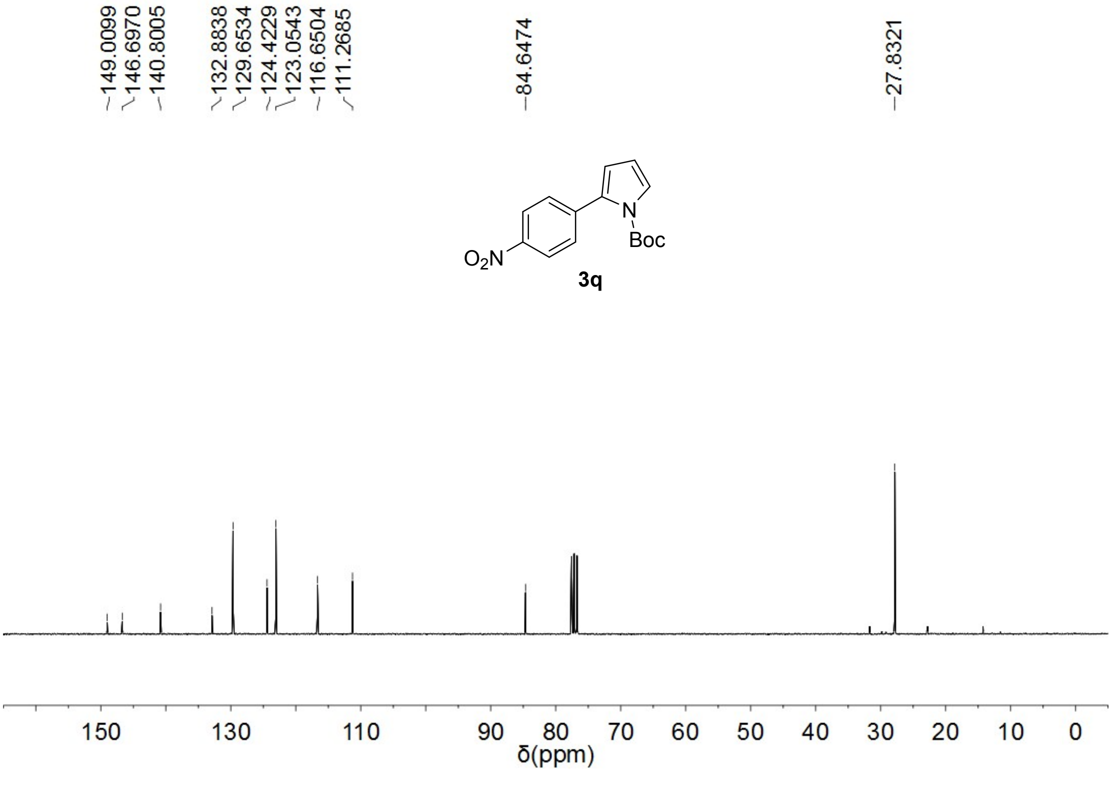
8.1994
8.1916
7.5353
7.5276
7.5209
7.5045
7.4980
7.4901
7.4145
7.4087
7.4036
7.3978
1.4345
O_2N
N
Boc
2.00
2.00
1.00
1.00
1.00
9.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
δ(ppm)

3q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140.8005
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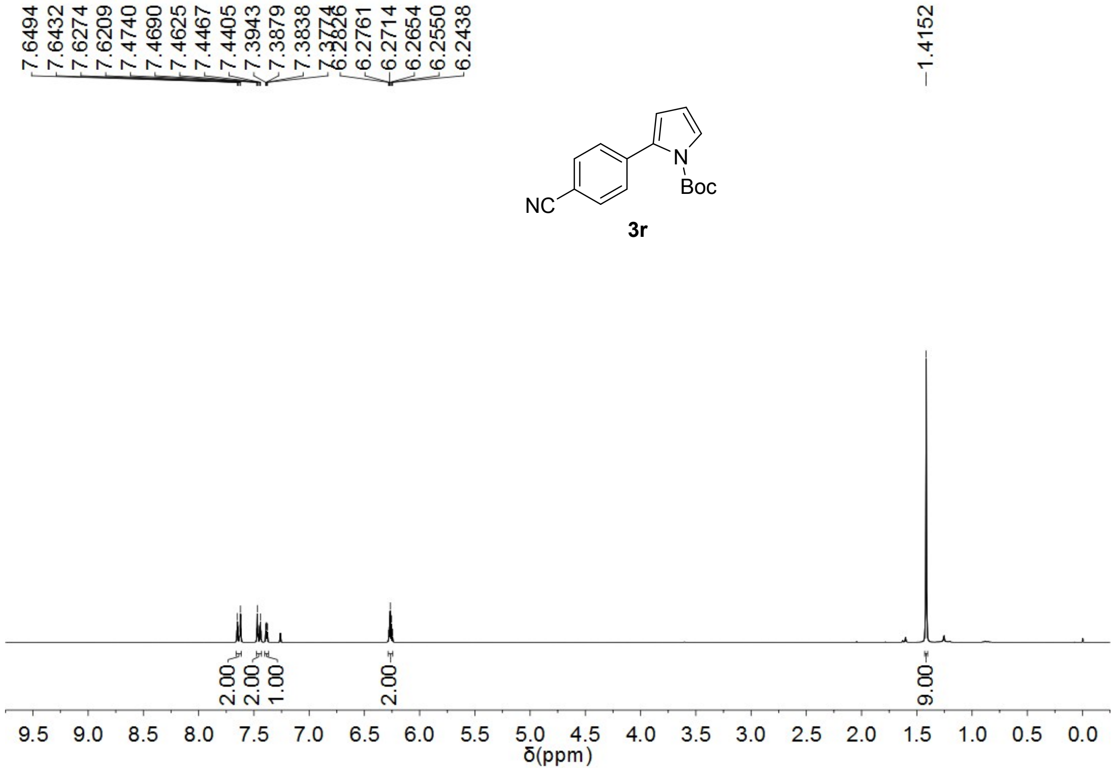
124.4229
123.0543
116.6504
111.2685
84.6474
27.8321
O_2N
N
Boc
150
130
110
90
80
70
60
50
40
30
20
10
0
δ(ppm)

3q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7.6209
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7.4405
7.3943
7.3879
7.3838
7.3774
6.2826
6.2761
6.2714
6.2654
6.2550
6.2438
1.4152
NC
N
Boc
3r
2.00
2.00
1.00
2.00
9.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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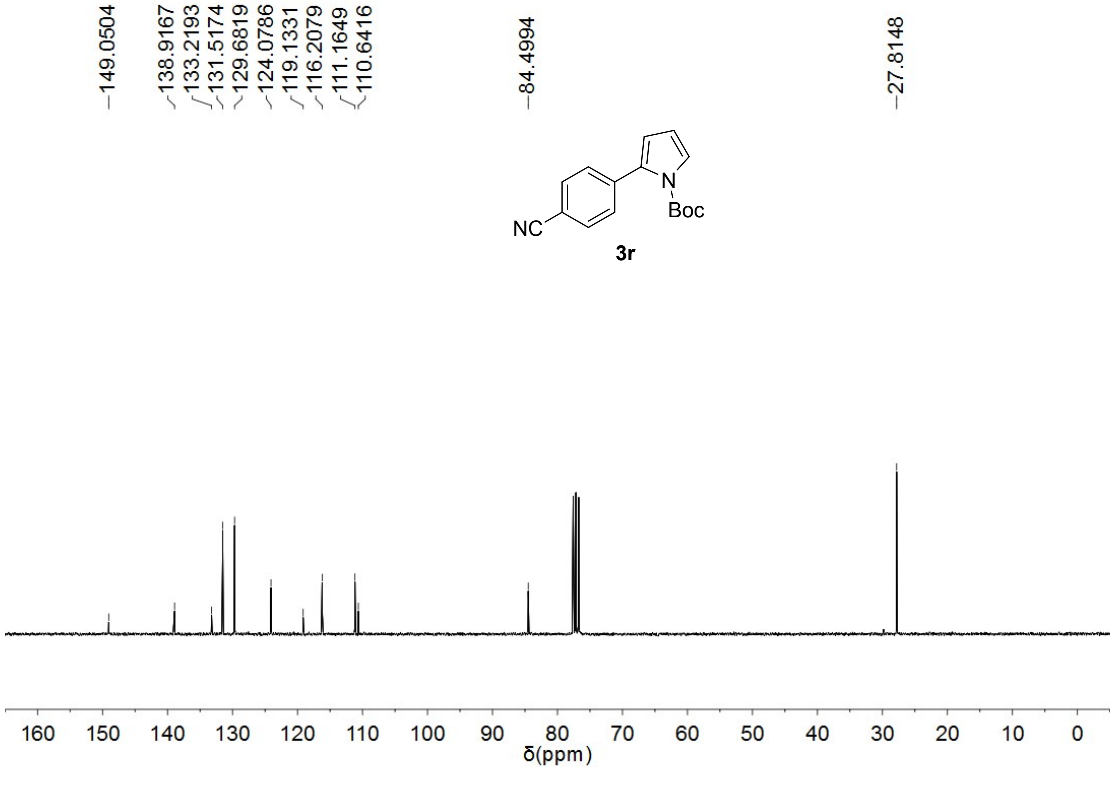
0.0
δ(ppm)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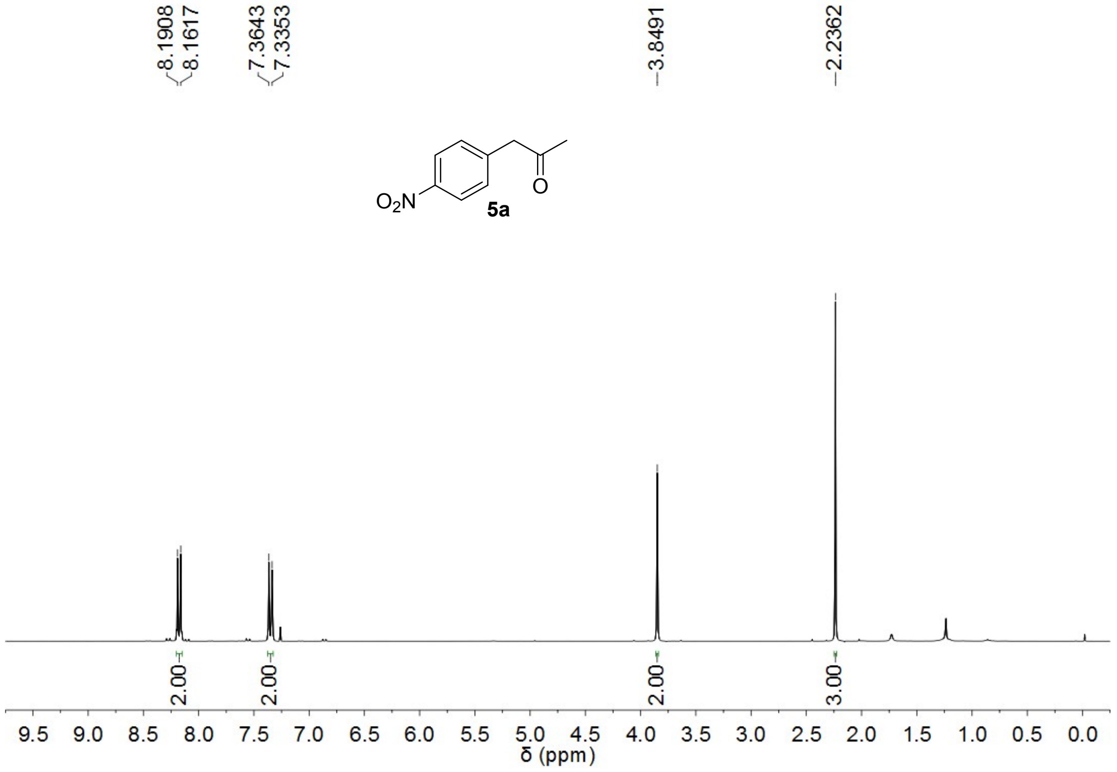
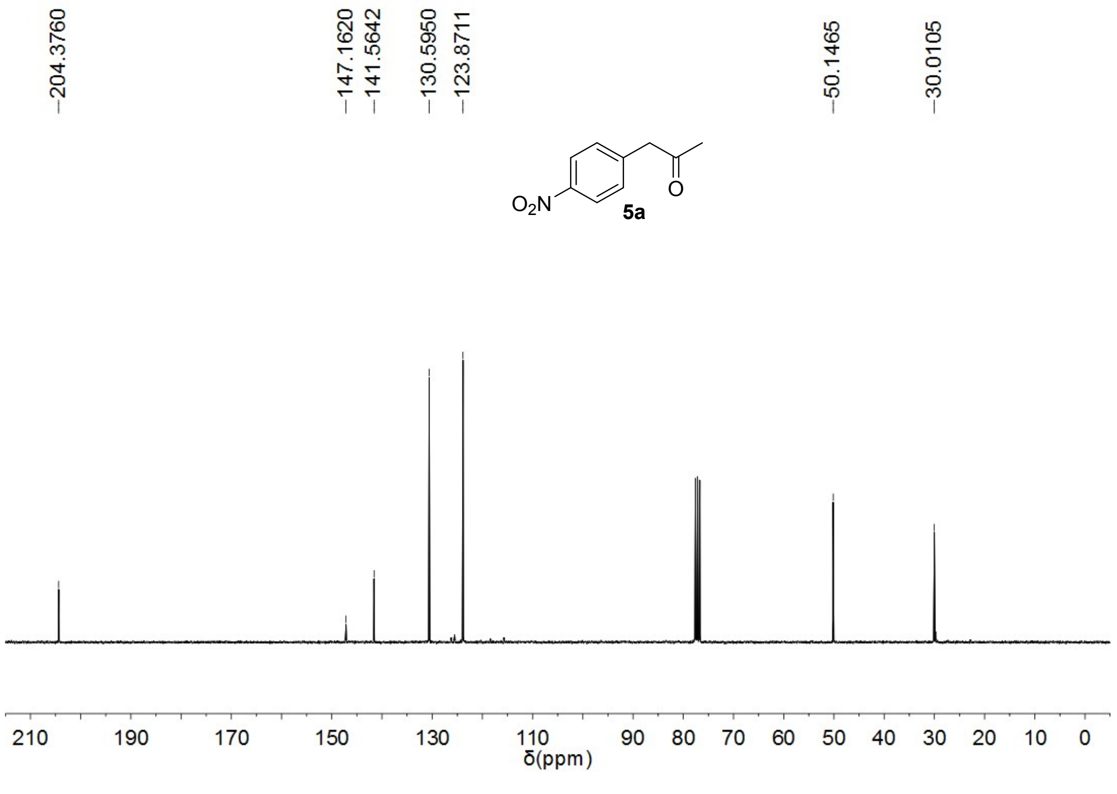
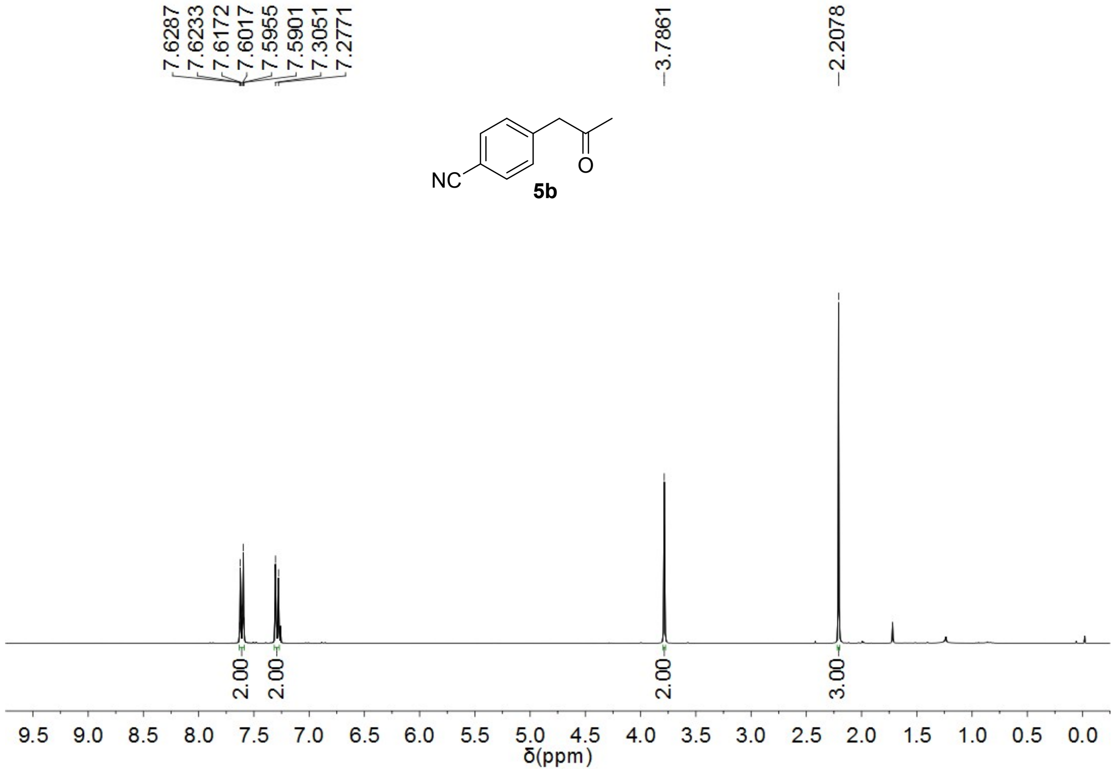
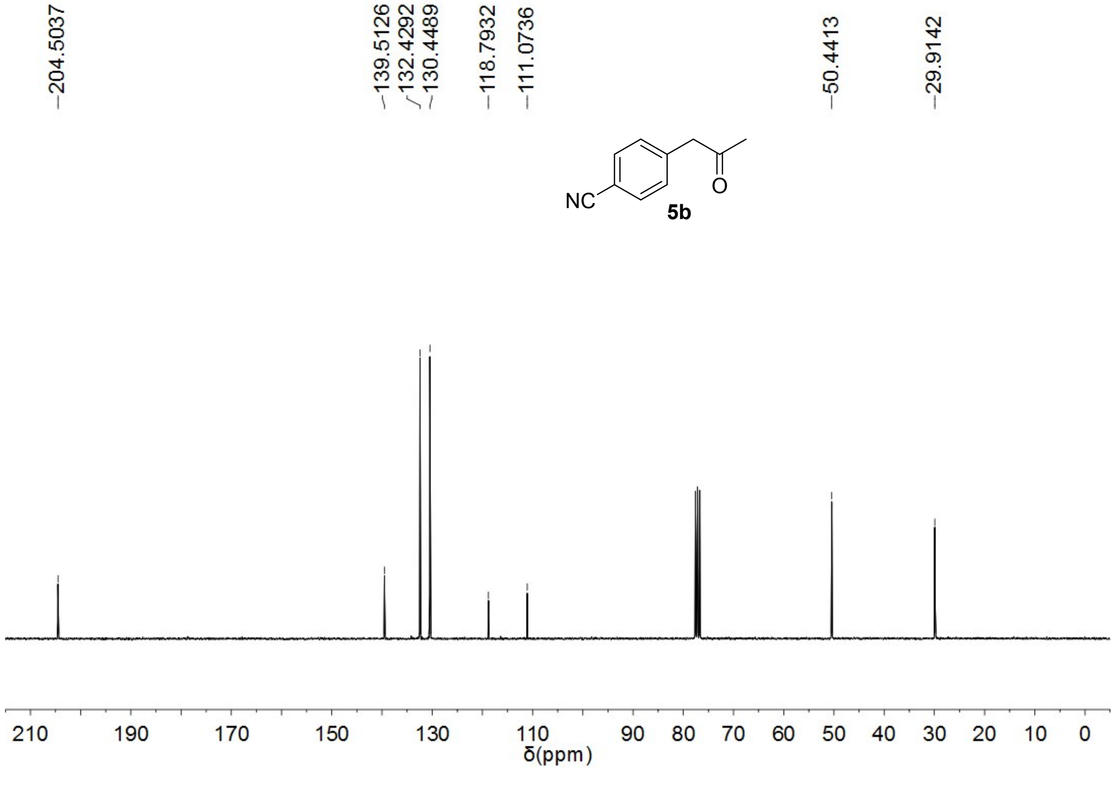
27.8148
NC
N
Boc
3r
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ(ppm)


8.1908
8.1617
7.3643
7.3353
3.8491
2.2362
O2N
O
5a
2.00
2.00
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ (ppm)


204.3760
147.1620
141.5642
130.5950
123.8711
50.1465
30.0105
O2N
O
5a
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
δ(ppm)


7.6287
7.6233
7.6172
7.6017
7.5955
7.5901
7.3051
7.2771
3.7861
2.2078
NC
O
5b
2.00
2.00
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ(ppm)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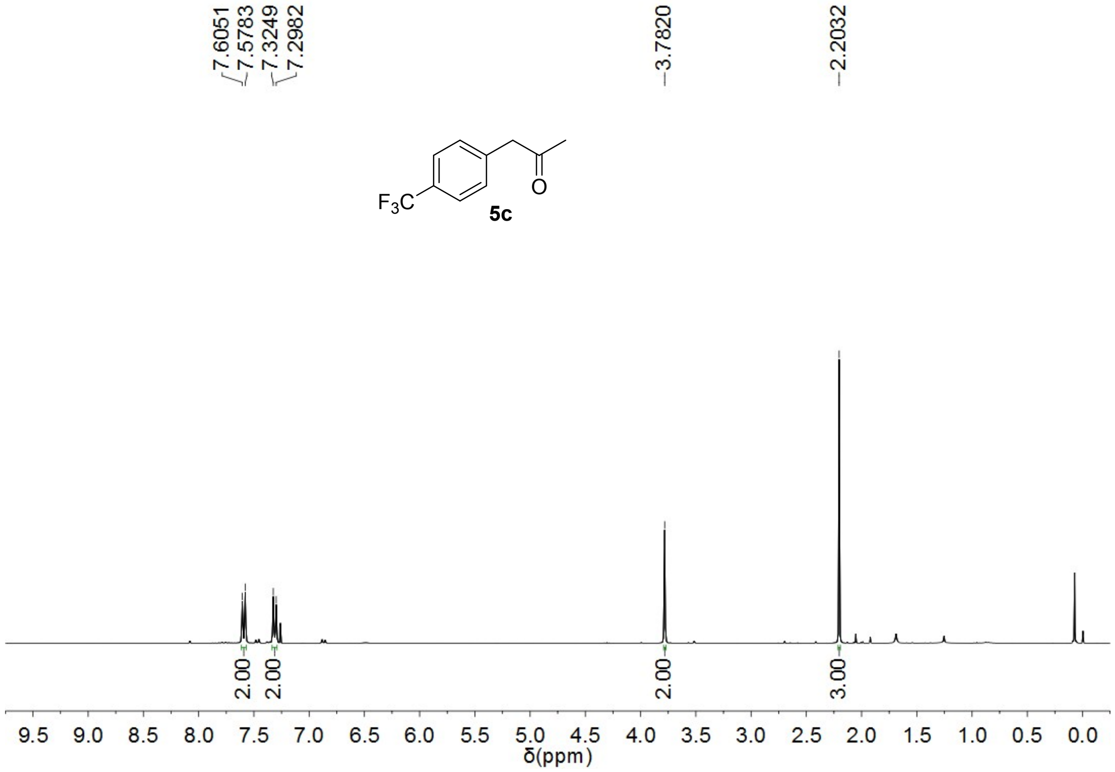
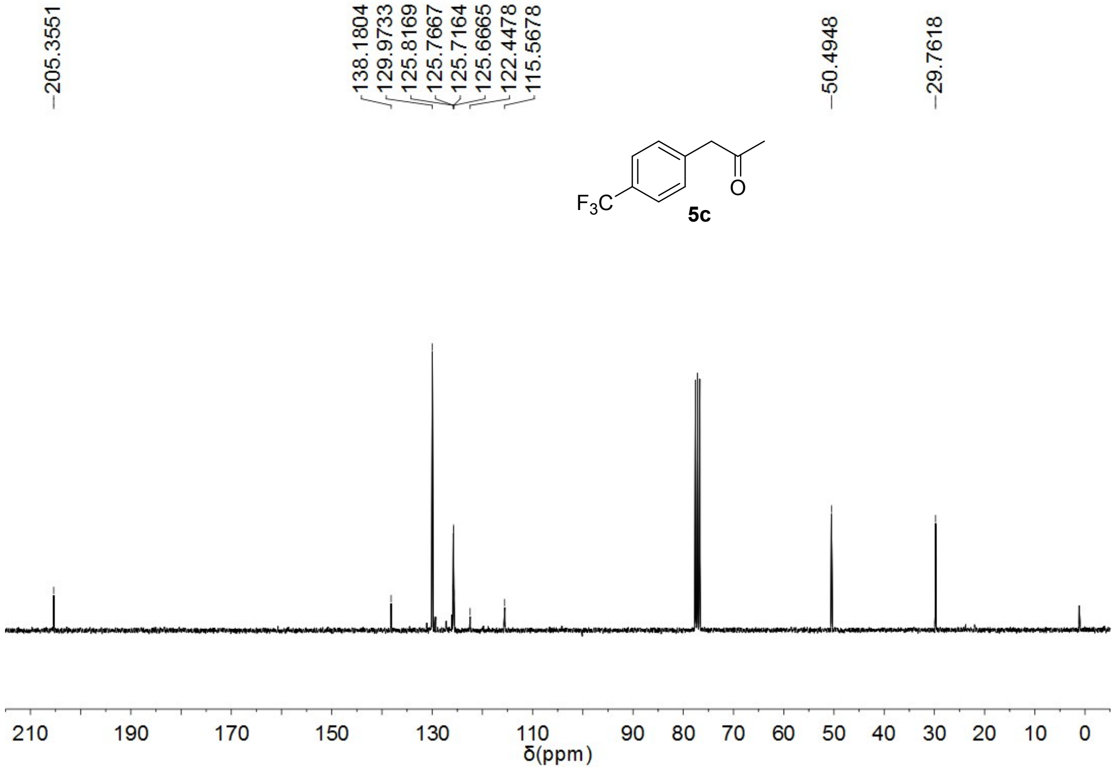
132.4292
130.4489
118.7932
111.0736
50.4413
29.9142
NC
O
5b
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
δ(ppm)


7.6051
7.5783
7.3249
7.2982
3.7820
2.2032
F3C
O
5c
2.00
2.00
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ(ppm)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138.1804
129.9733
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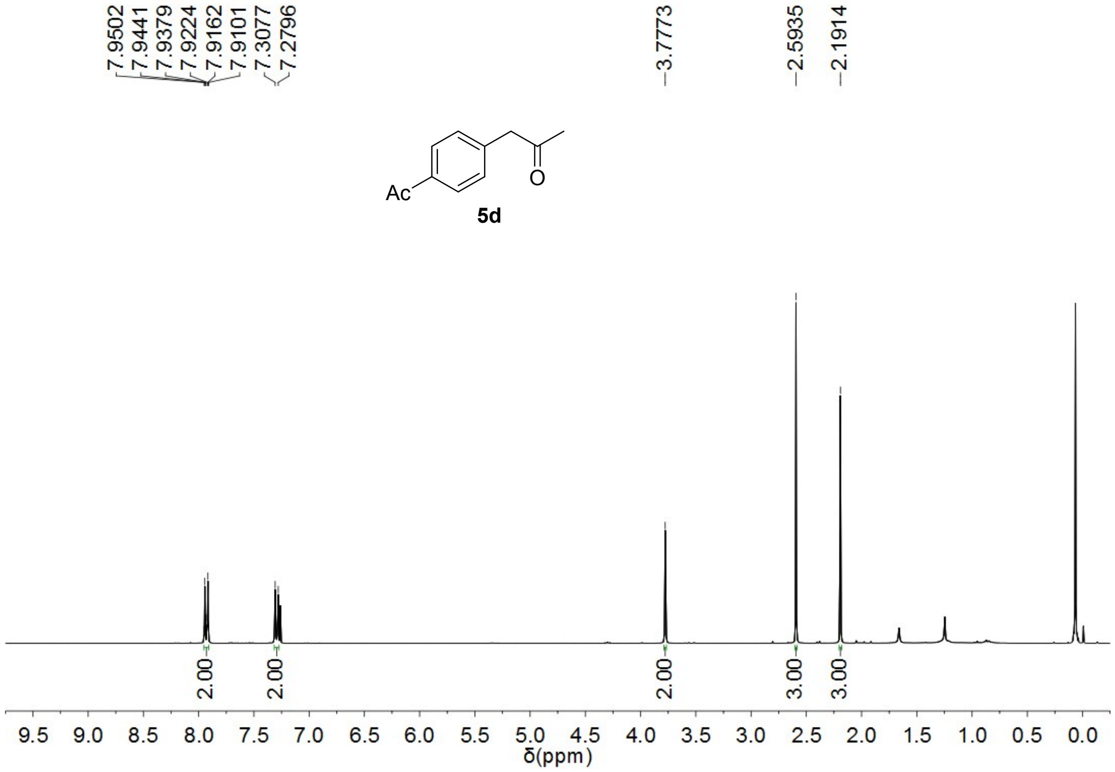
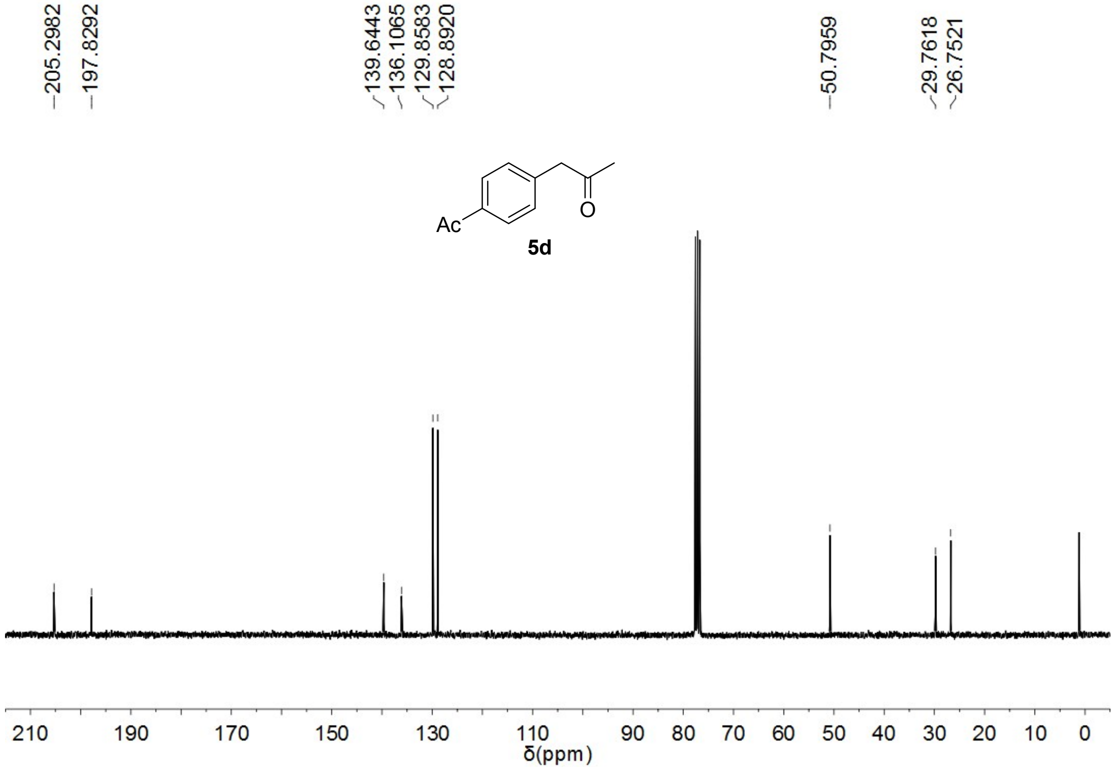
125.7667
125.7164
125.6665
122.4478
115.5678
50.4948
29.7618
F3C
O
5c
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
δ(ppm)


7.9502
7.9441
7.9379
7.9224
7.9162
7.9101
7.3077
7.2796
3.7773
2.5935
2.1914
Ac
O
5d
2.00
2.00
2.00
3.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ(ppm)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Ac
O
5d
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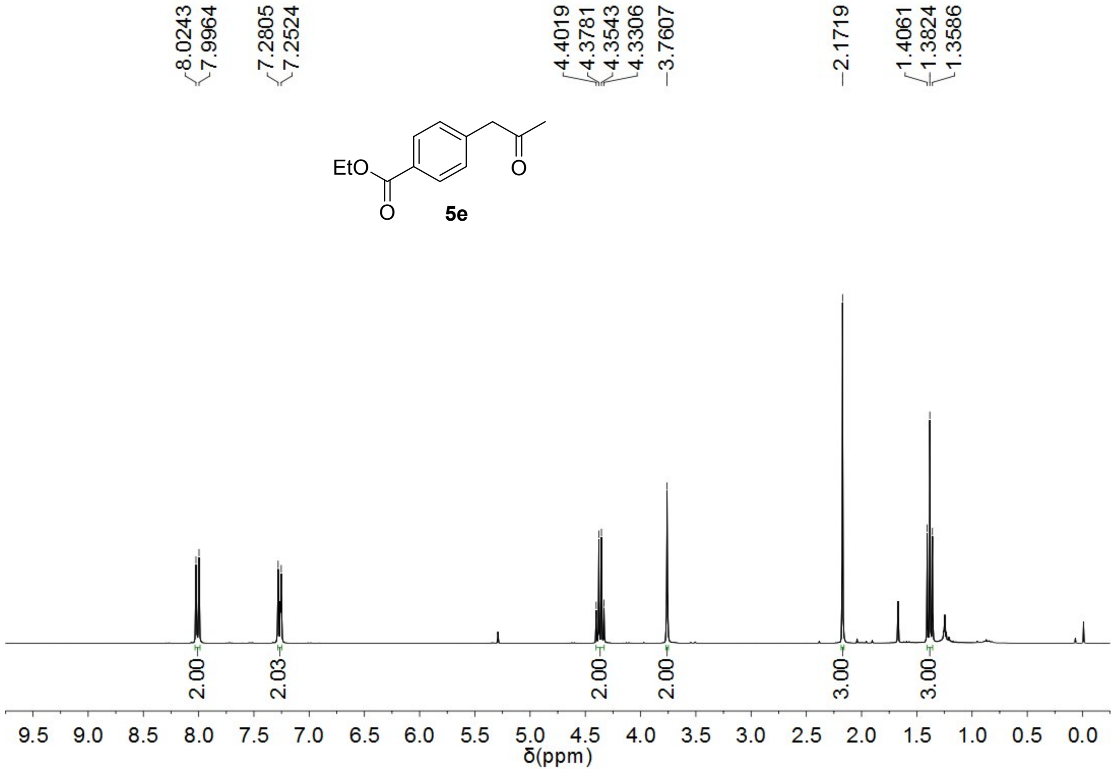
δ(ppm)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7.2524
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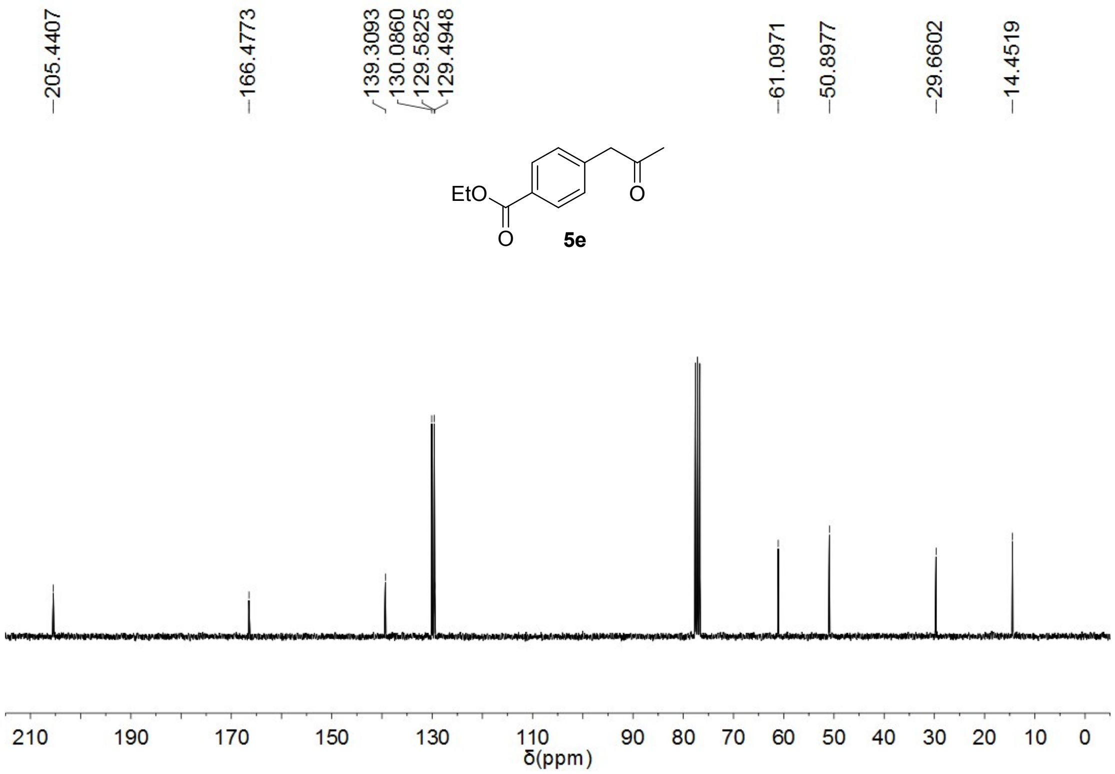
4.3781
4.3543
4.3306
3.7607
2.1719
1.4061
1.3824
1.3586
EtO
O
O
5e
2.00
2.03
2.00
2.00
3.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ(ppm)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129.4948
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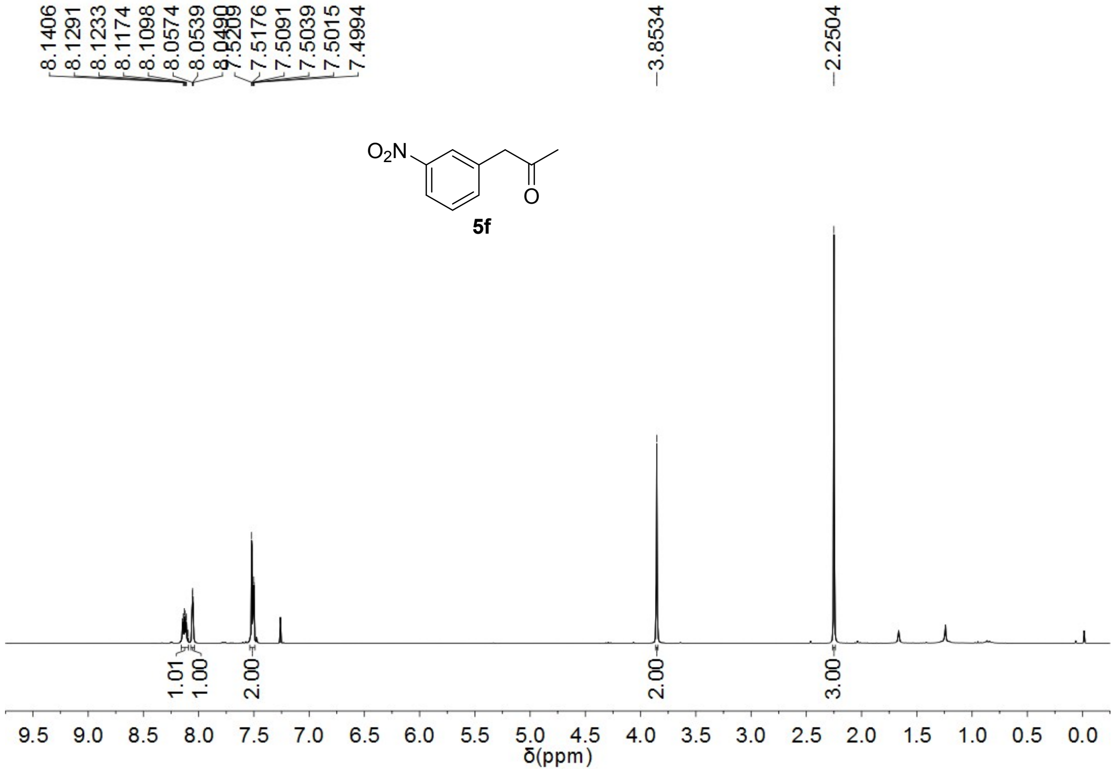
EtO
O
O
5e
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
δ(ppm)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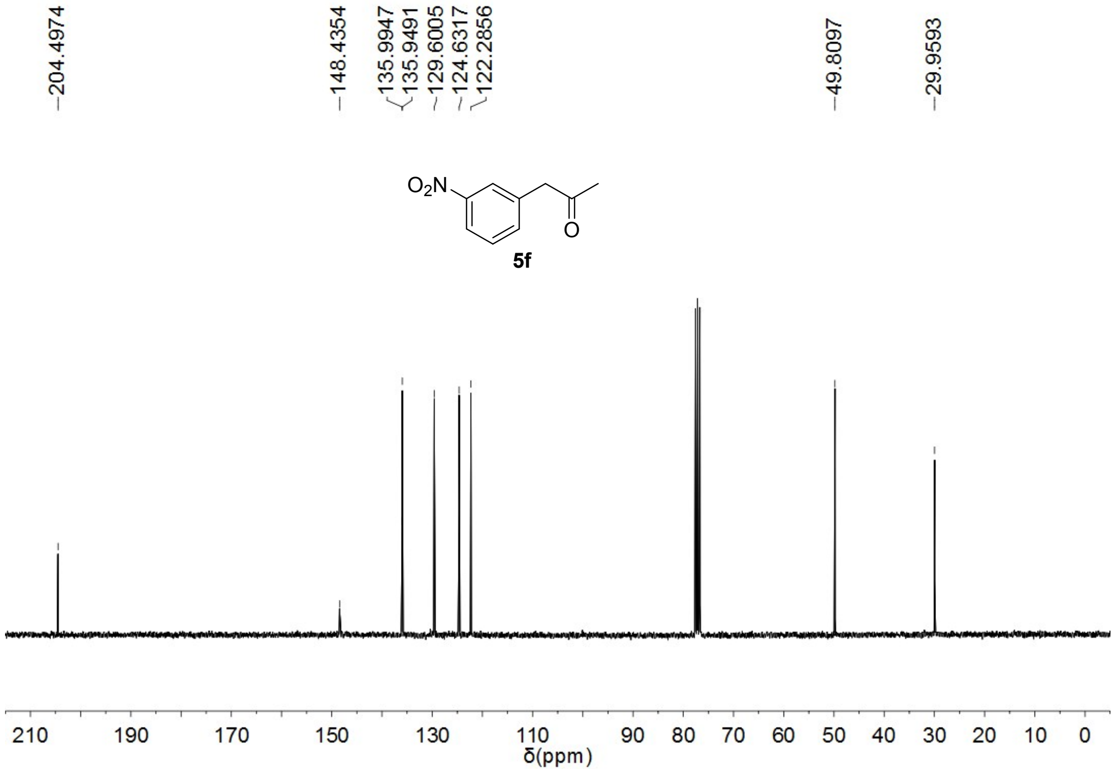
7.5039
7.5015
7.4994
3.8534
2.2504
O_2N
O
5f
1.01
1.00
2.00
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ(ppm)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29.9593
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O
5f
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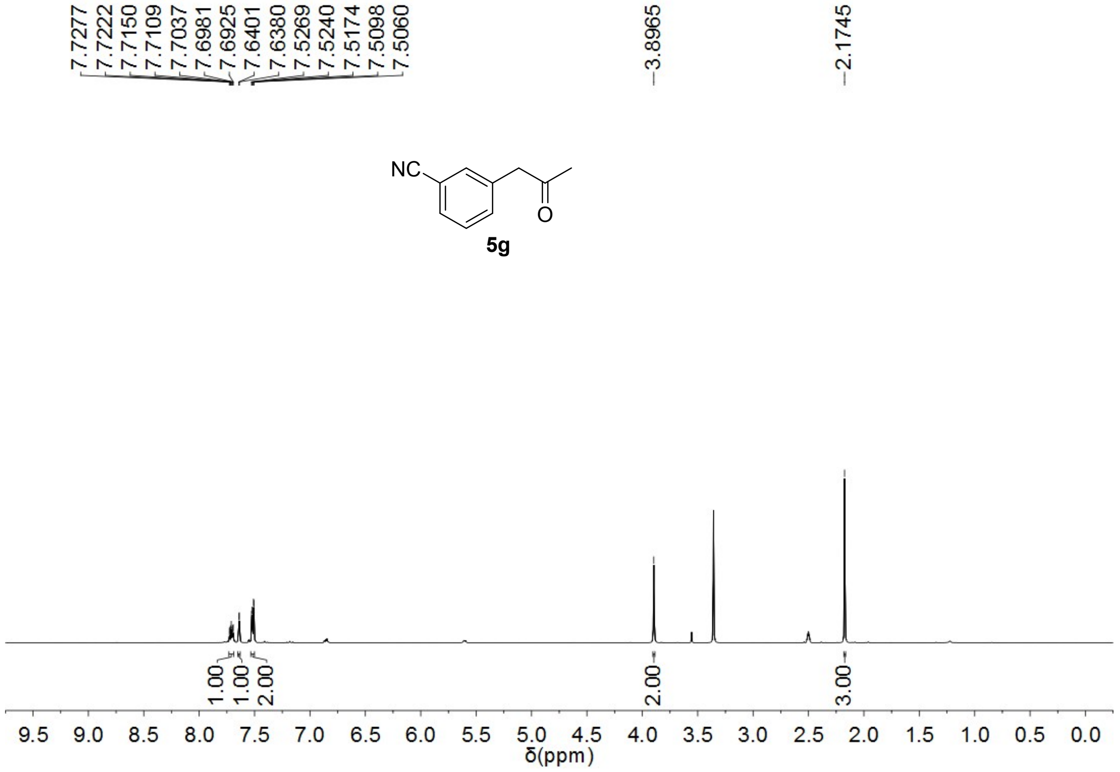
δ(ppm)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7.6380
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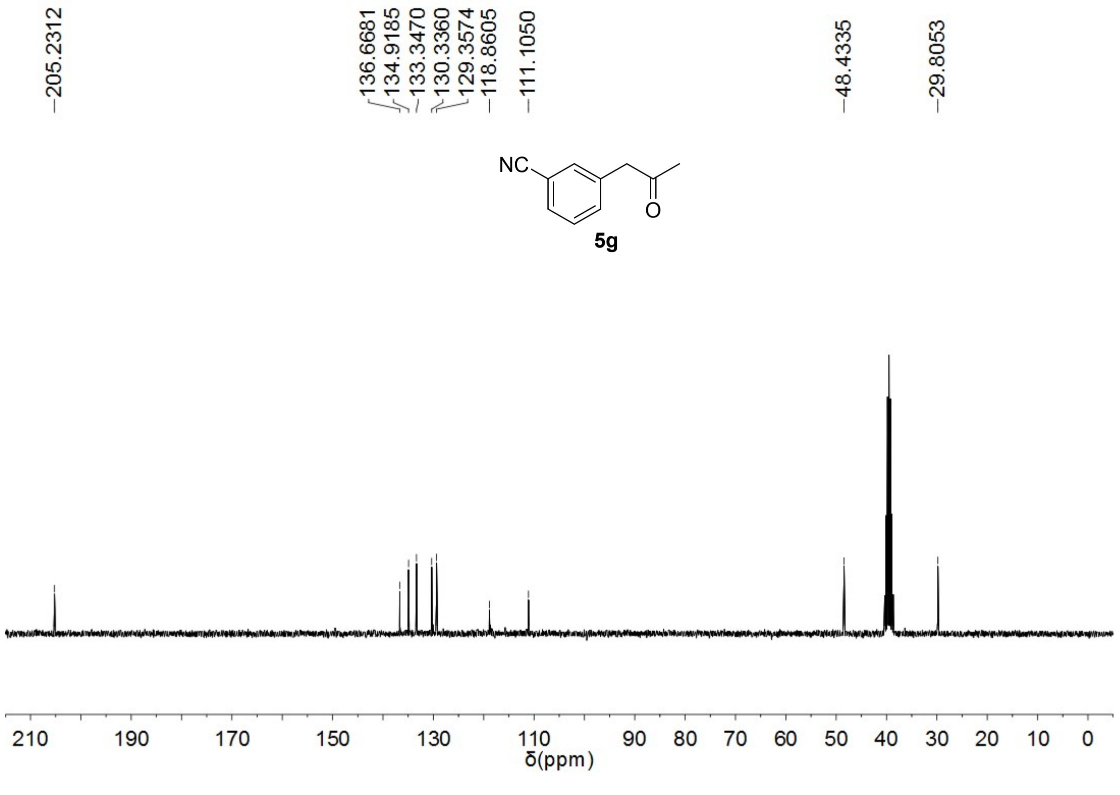
7.5240
7.5174
7.5098
7.5060
3.8965
2.1745
NC
O
5g
1.00
1.00
2.00
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ(ppm)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129.3574
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O
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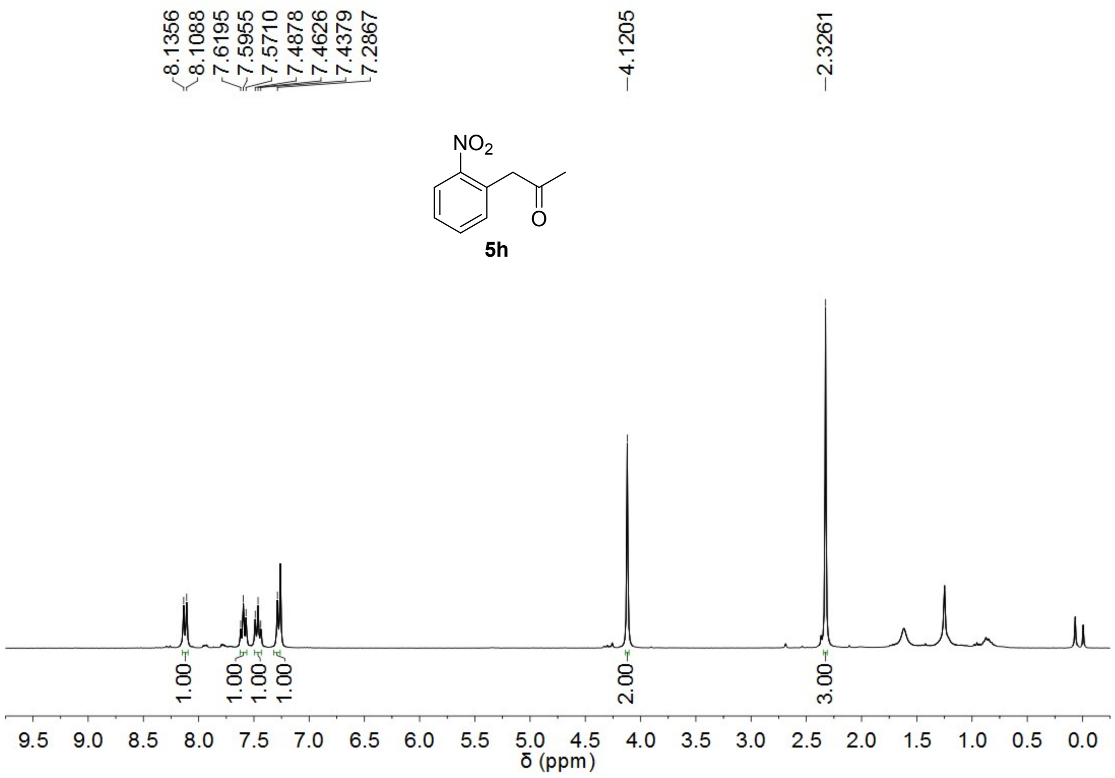
δ(ppm)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7.4626
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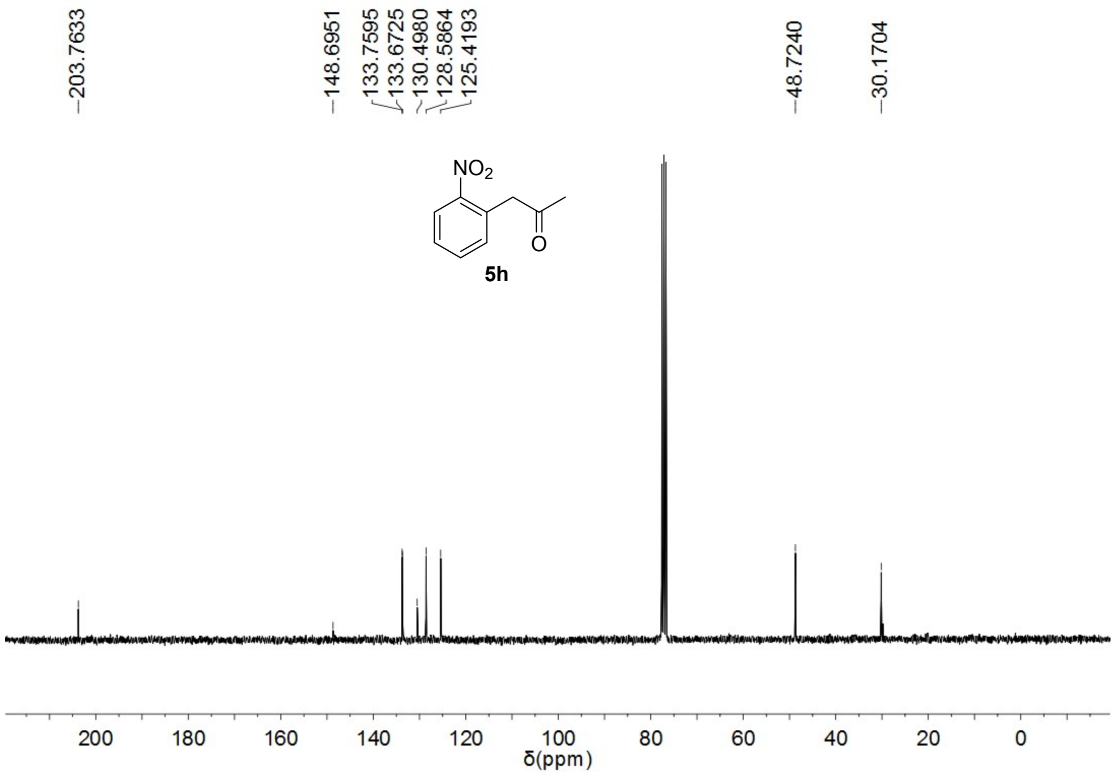
7.2867
4.1205
2.3261
NO2
O
5h
1.00
1.00
1.00
1.00
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ (ppm)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133.6725
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125.4193
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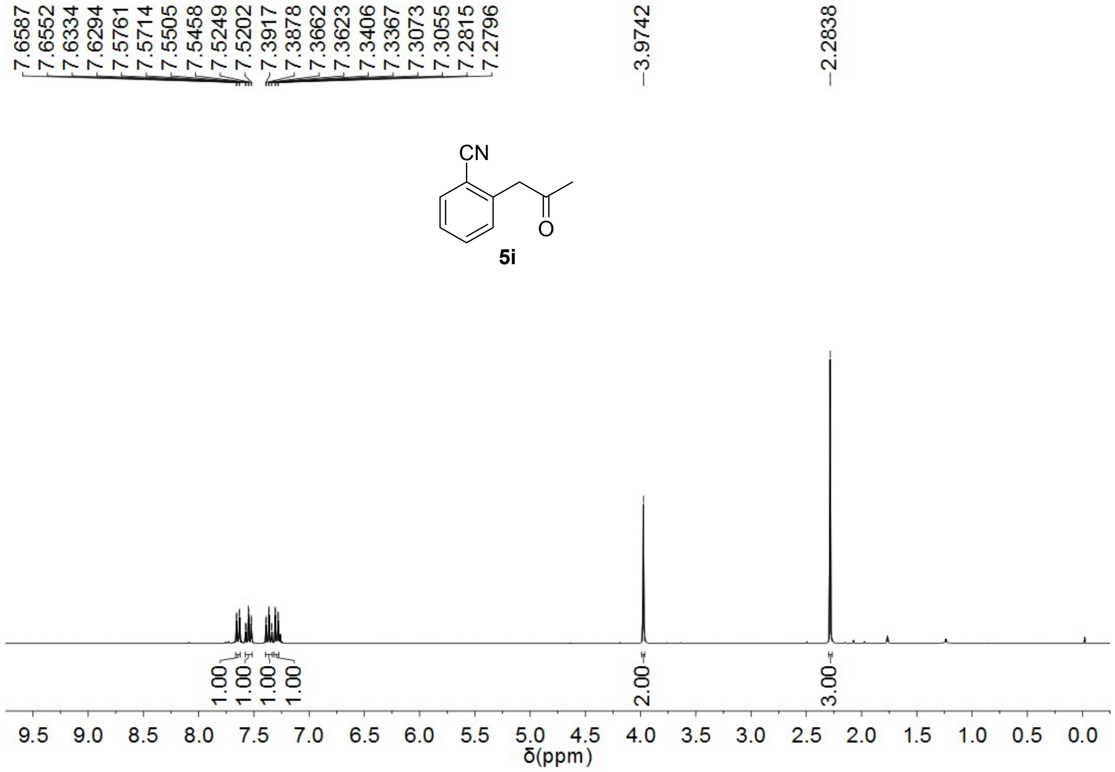
30.1704
NO2
O
5h
200 180 160 140 120 100 80 60 40 20 0
δ(ppm)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7.3878
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7.3623
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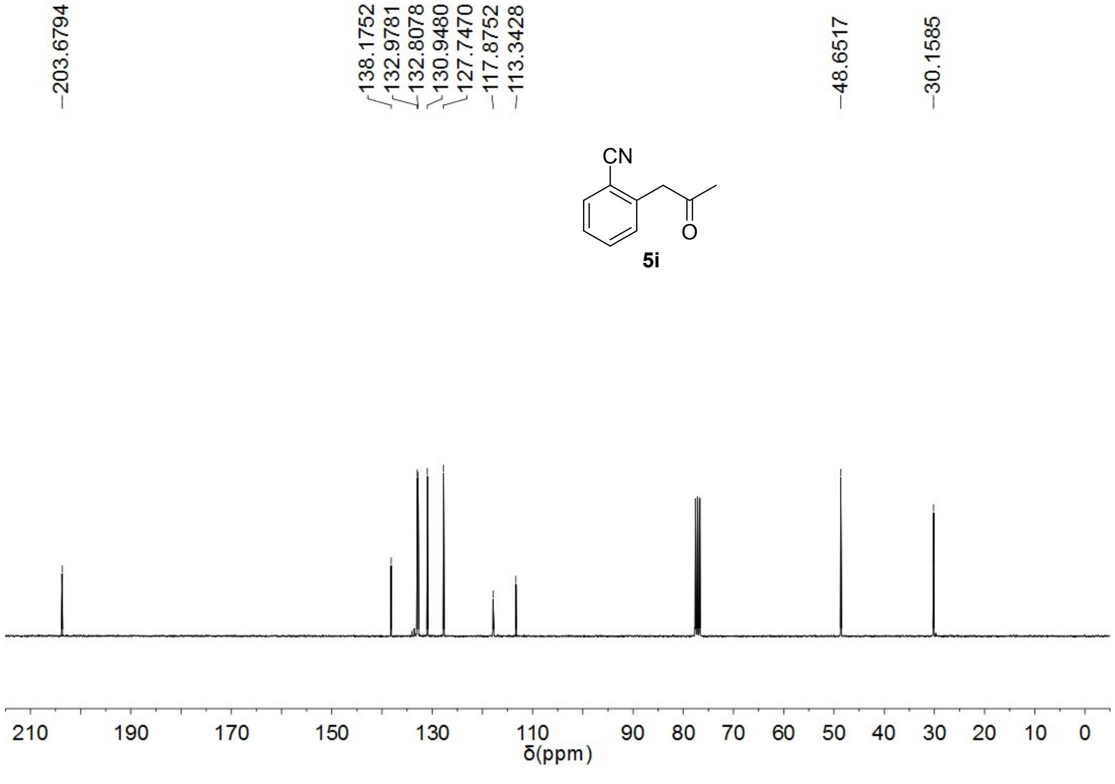
7.3367
7.3073
7.3055
7.2815
7.2796
3.9742
2.2838
CN
O
5i
1.00
1.00
1.00
1.00
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ(ppm)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CN
O
5i
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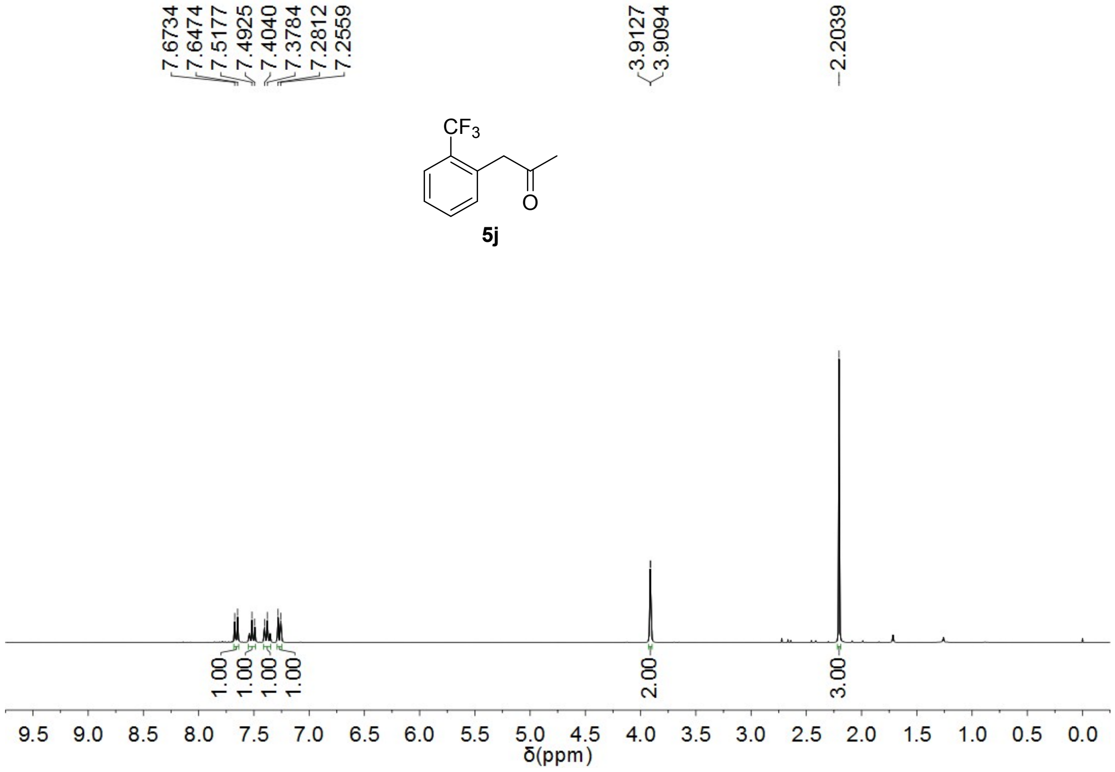
δ(ppm)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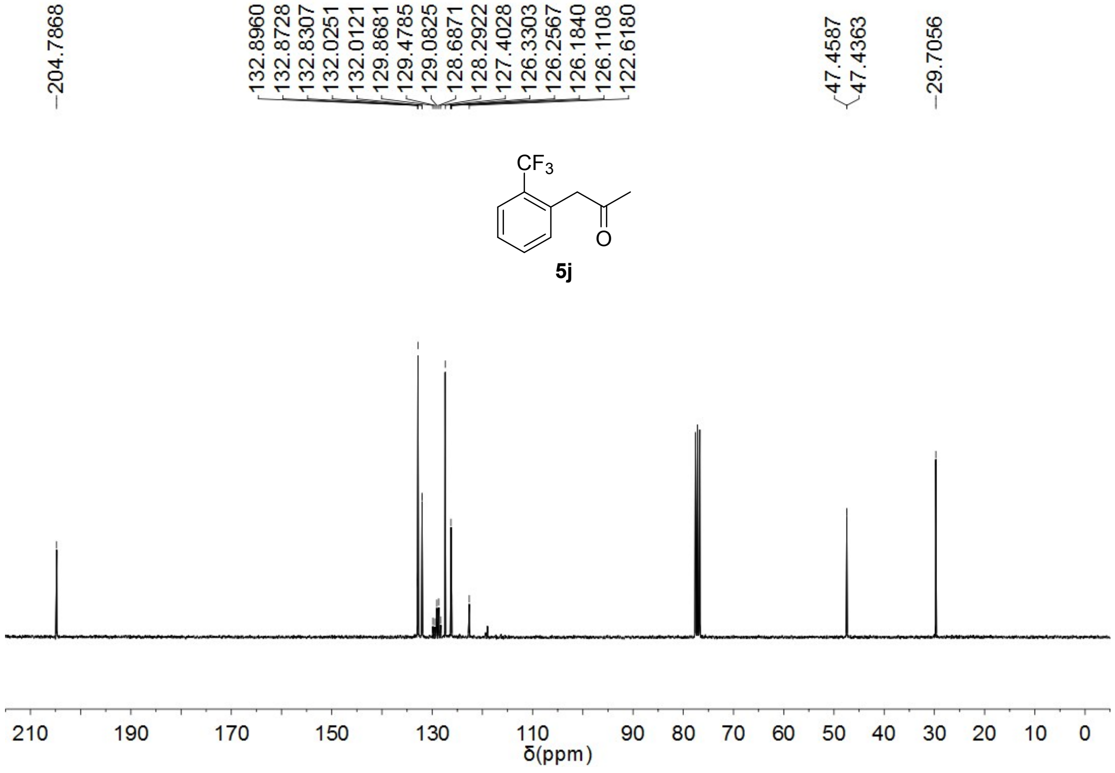
2.2039
CF3
O
5j
1.00
1.00
1.00
1.00
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ(ppm)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126.1840
126.1108
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CF3
O
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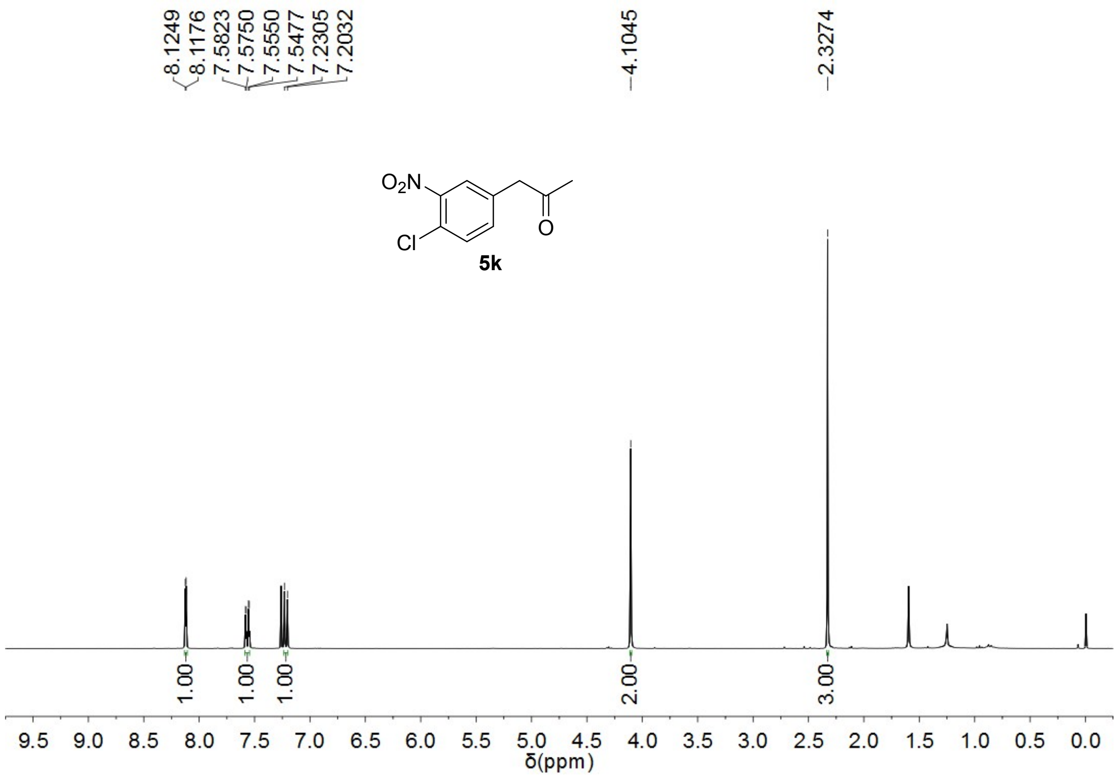
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
δ(ppm)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7.2305
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4.1045
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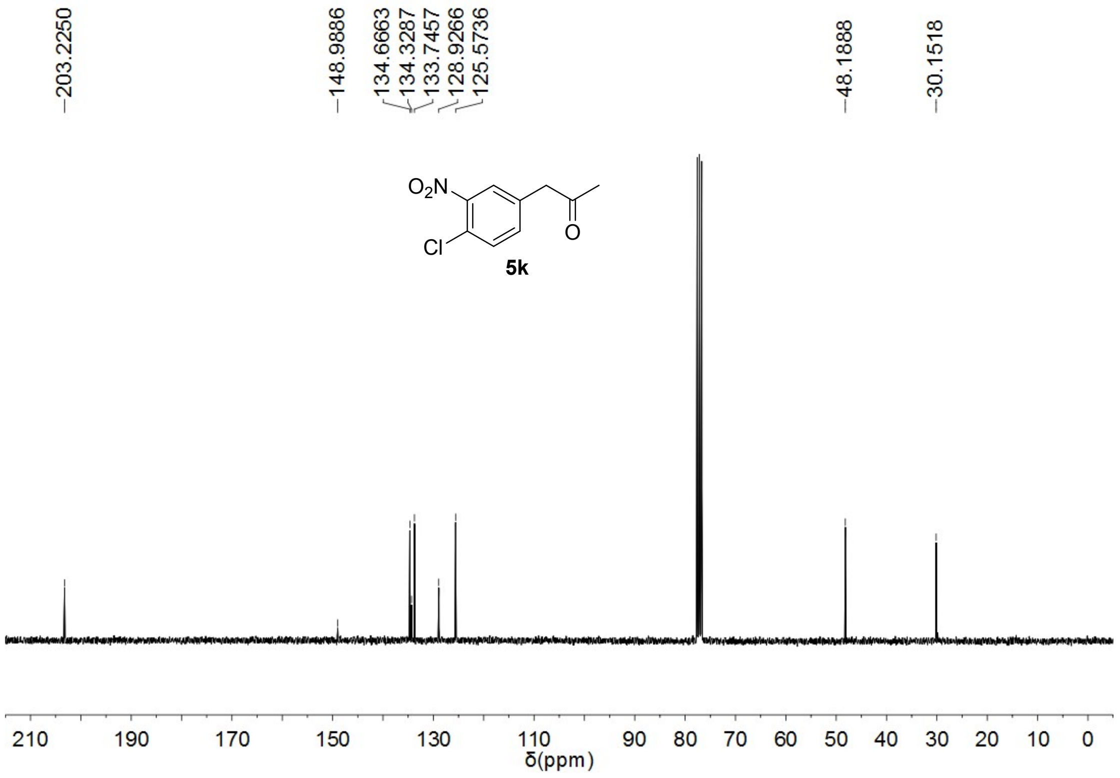
O_2N
Cl
O
5k
1.00
1.00
1.00
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ(ppm)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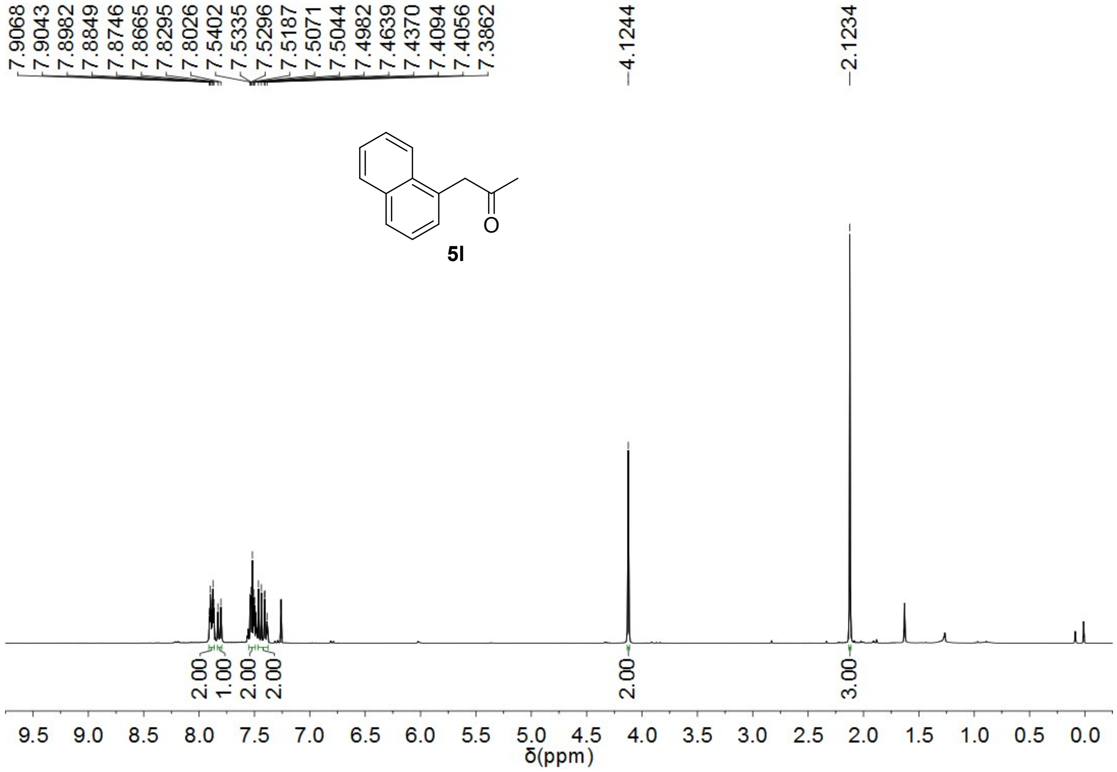
Cl
O
5k
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
δ(ppm)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7.5071
7.5044
7.4982
7.4639
7.4370
7.4094
7.4056
7.3862
4.1244
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O
5l
2.00
1.00
2.00
2.00
2.00
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
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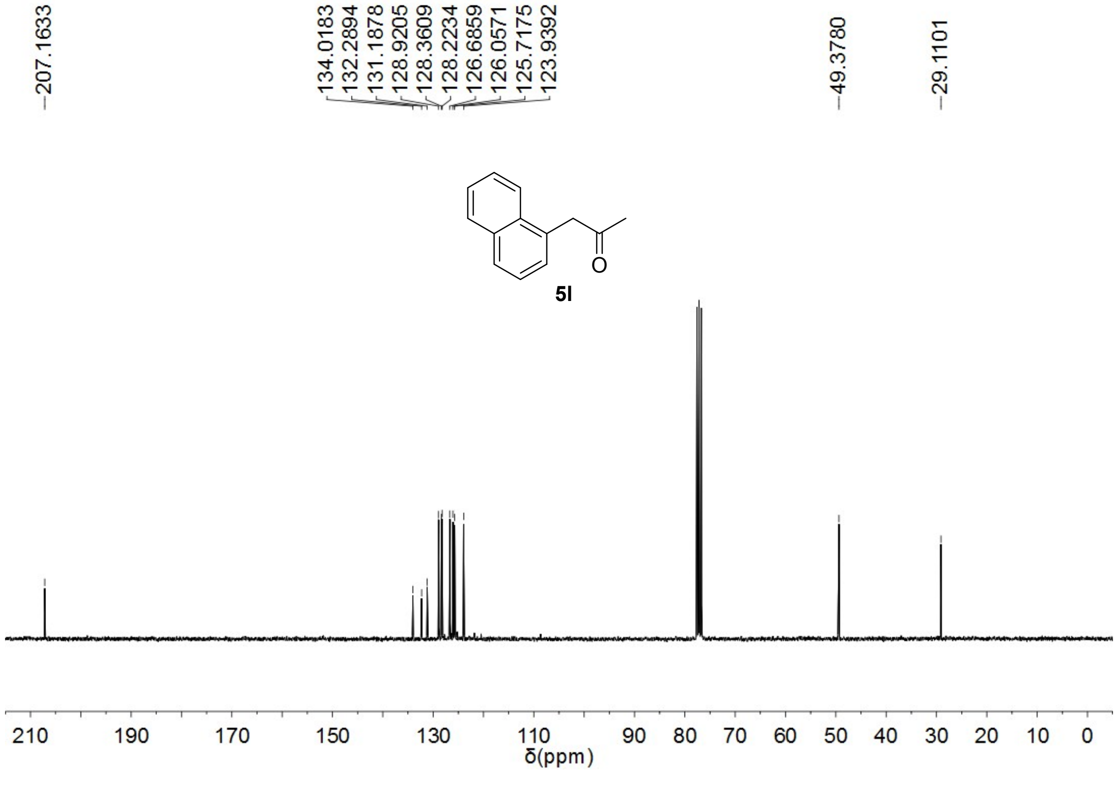
δ(ppm)


207.1633
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126.0571
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49.3780
29.1101
O
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110
90
80
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50
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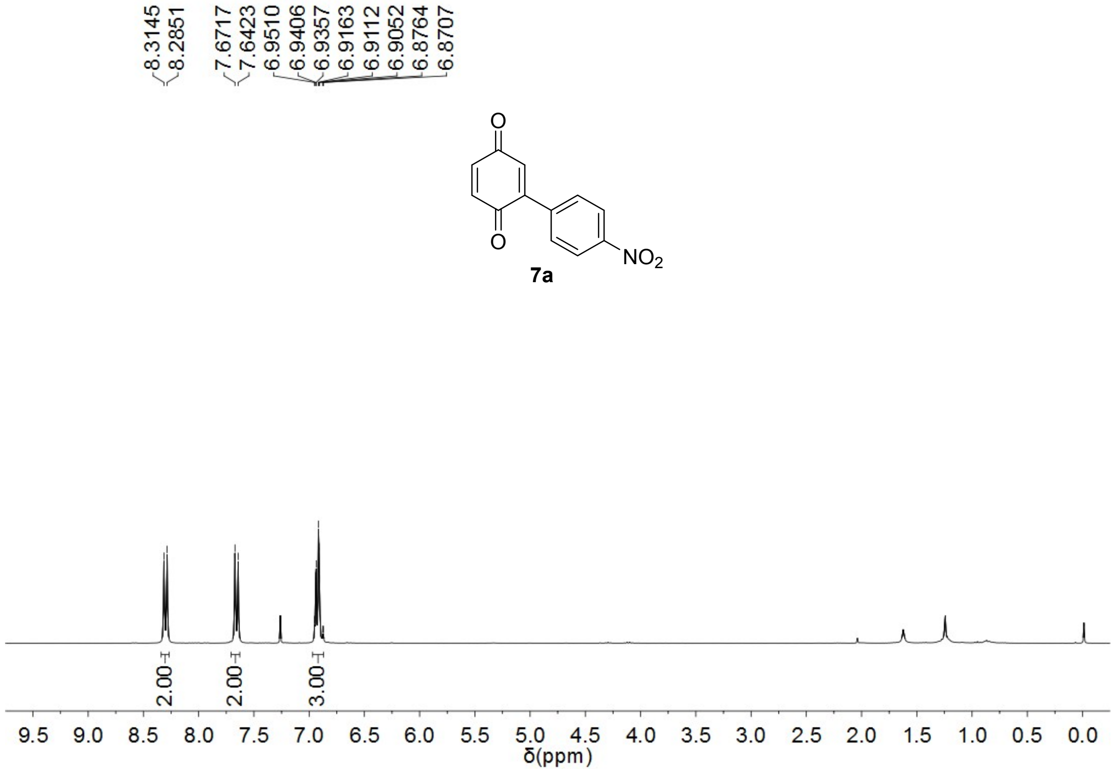
30
20
10
0
δ(ppm)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6.9112
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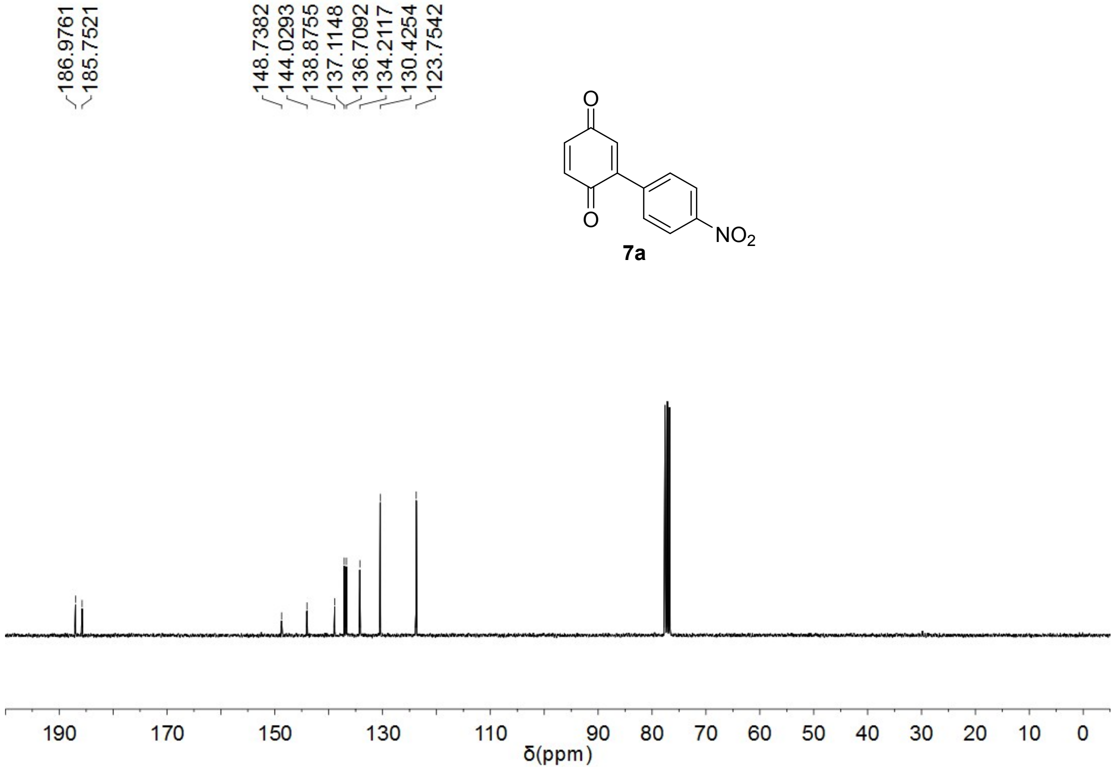
6.8764
6.8707
O
O
NO2
7a
2.00
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ(ppm)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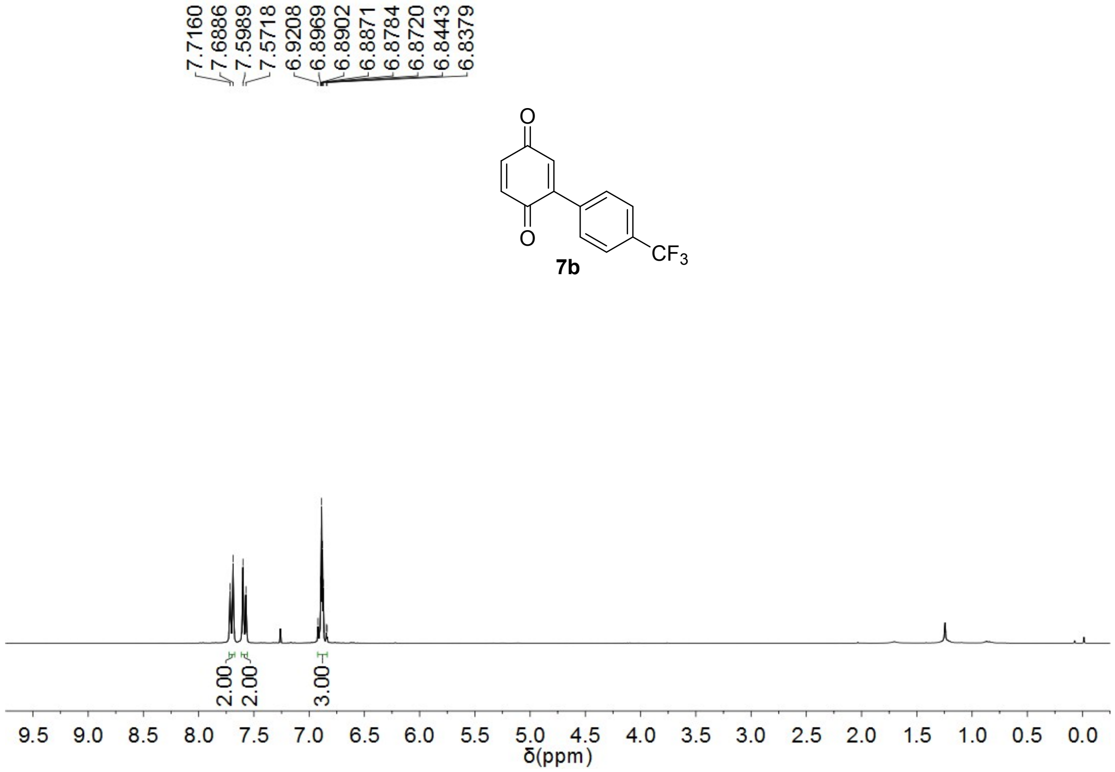
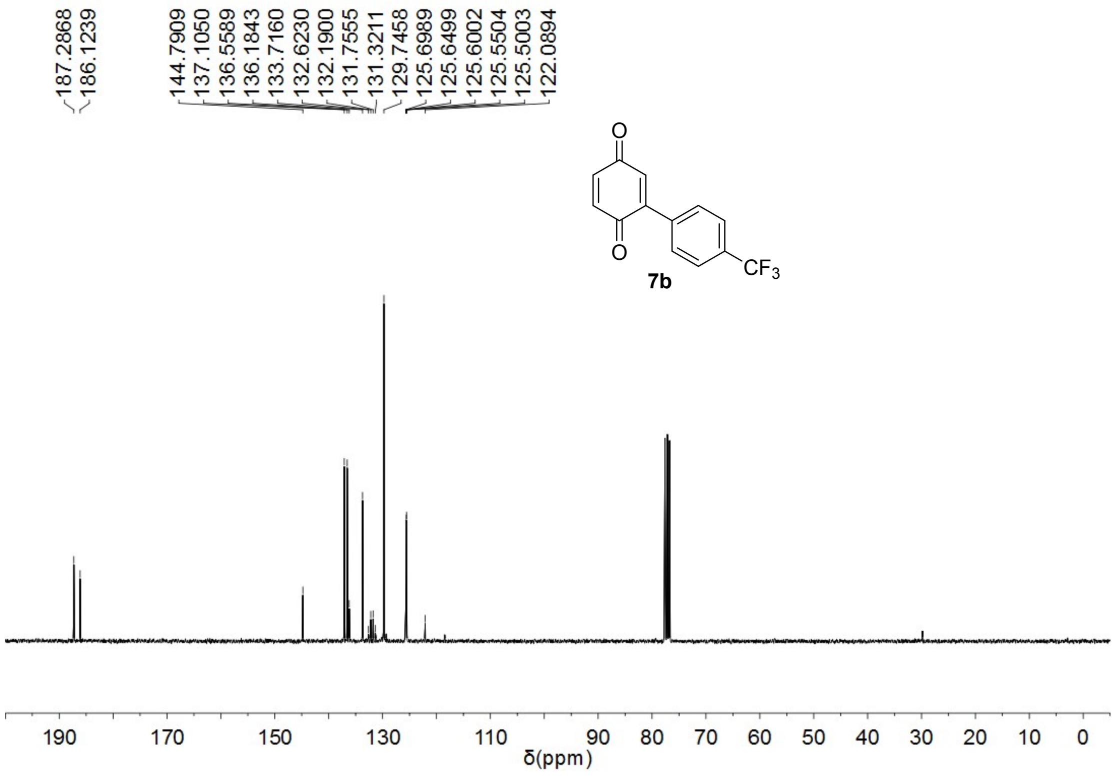
185.7521
148.7382
144.0293
138.8755
137.1148
136.7092
134.2117
130.4254
123.7542
O
O
NO2
7a
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
δ(ppm)


7.7160
7.6886
7.5989
7.5718
6.9208
6.8969
6.8902
6.8871
6.8784
6.8720
6.8443
6.8379
O
O
CF3
7b
2.00
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ(ppm)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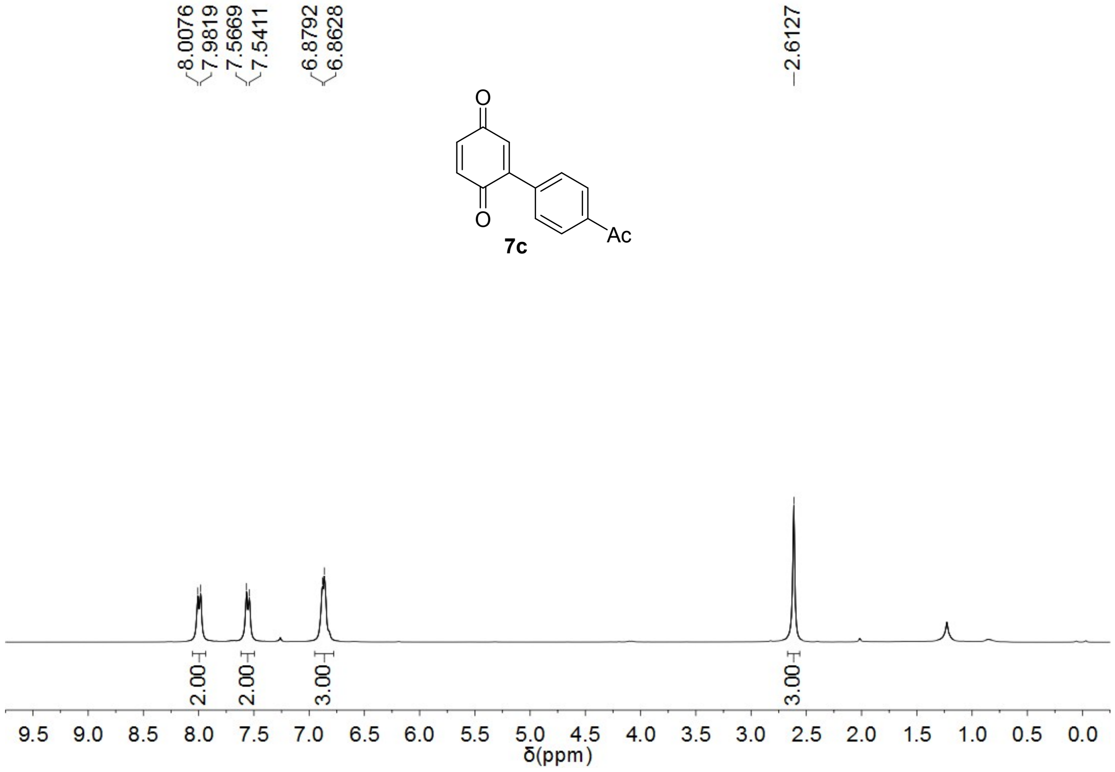
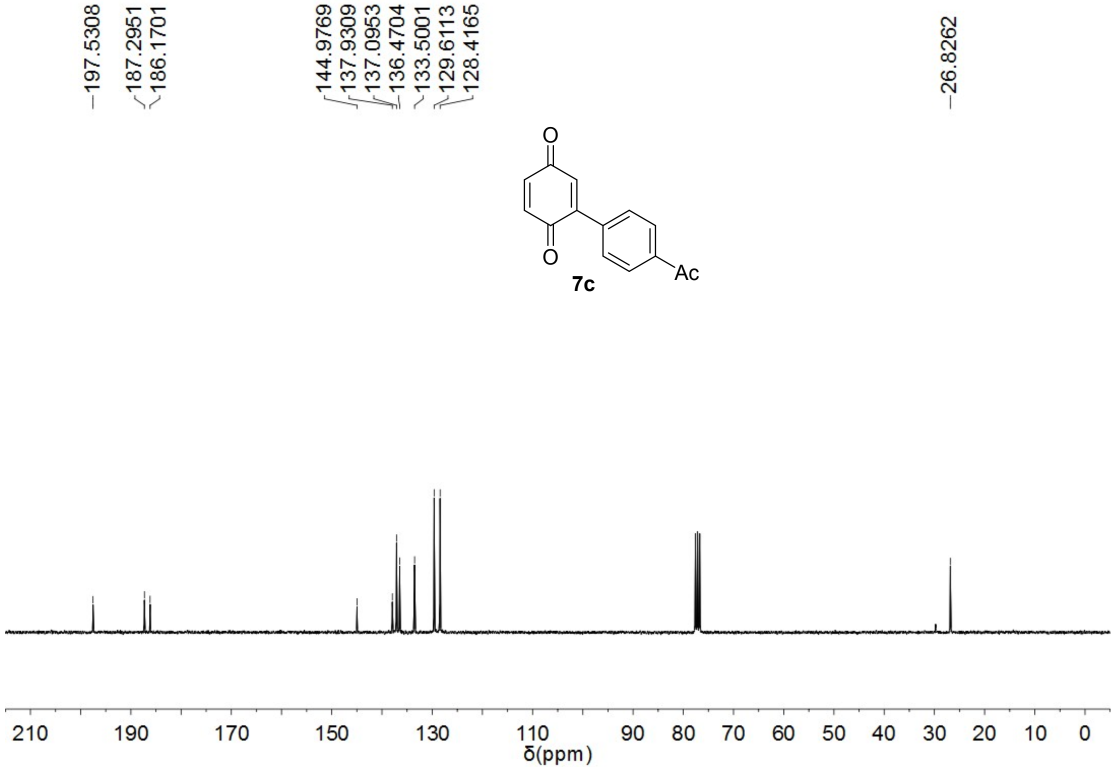
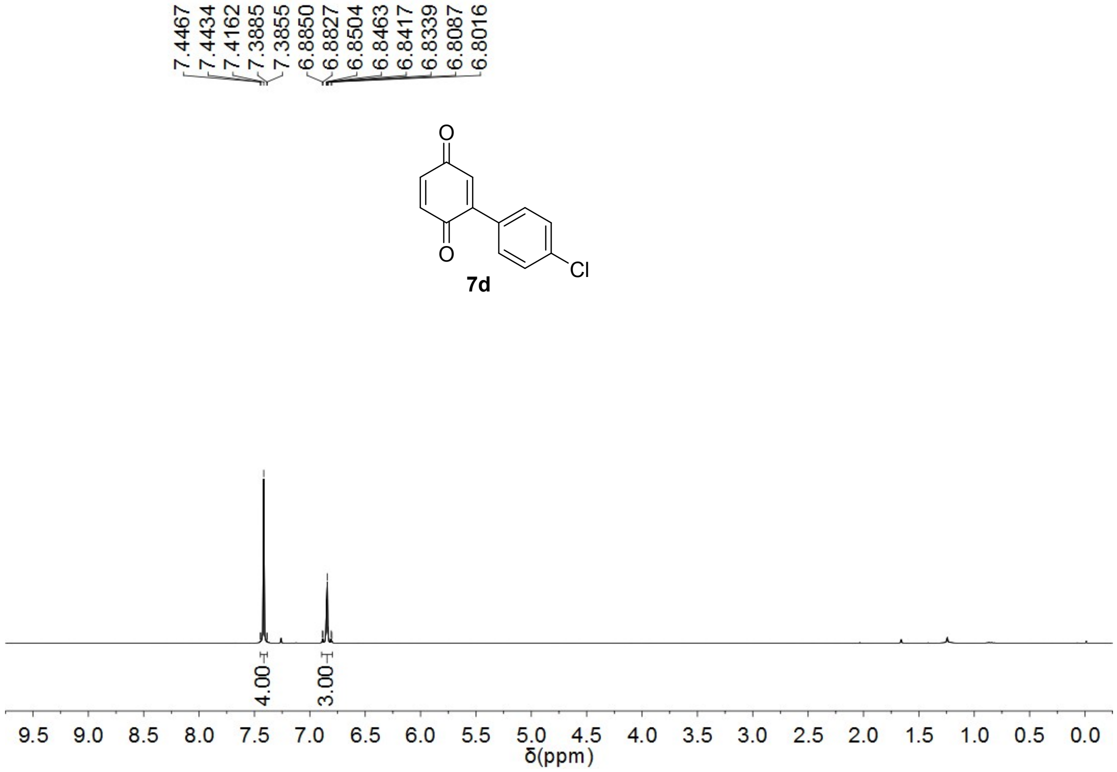
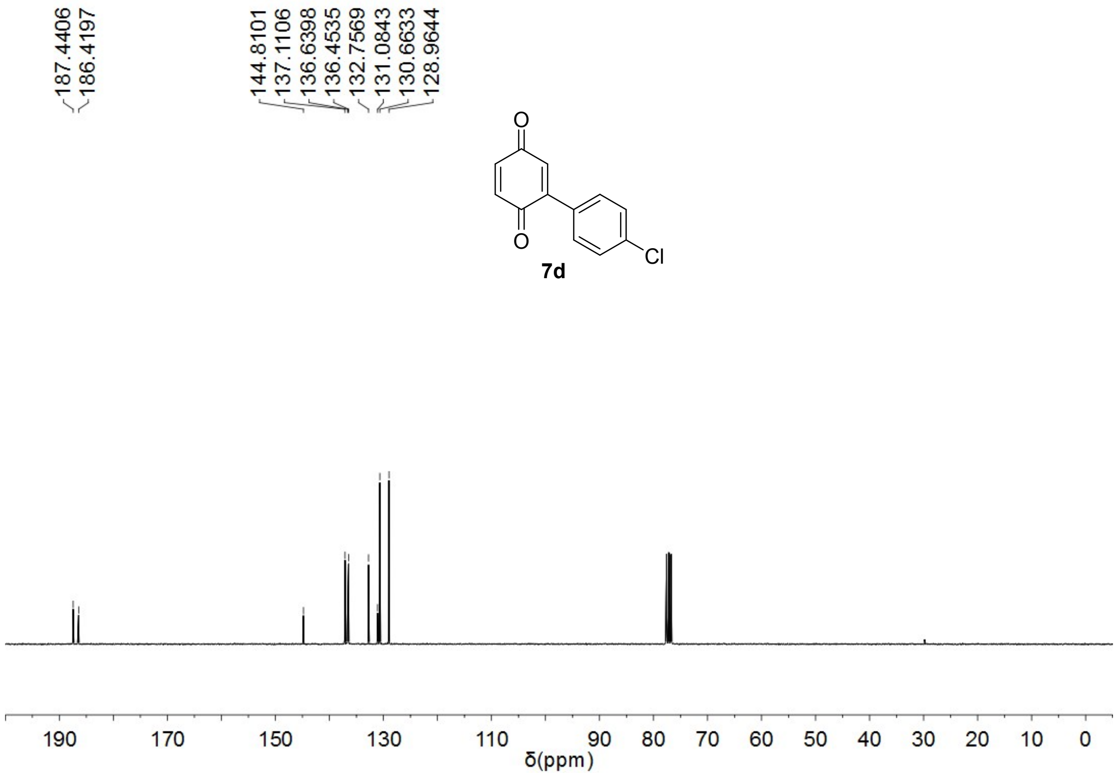
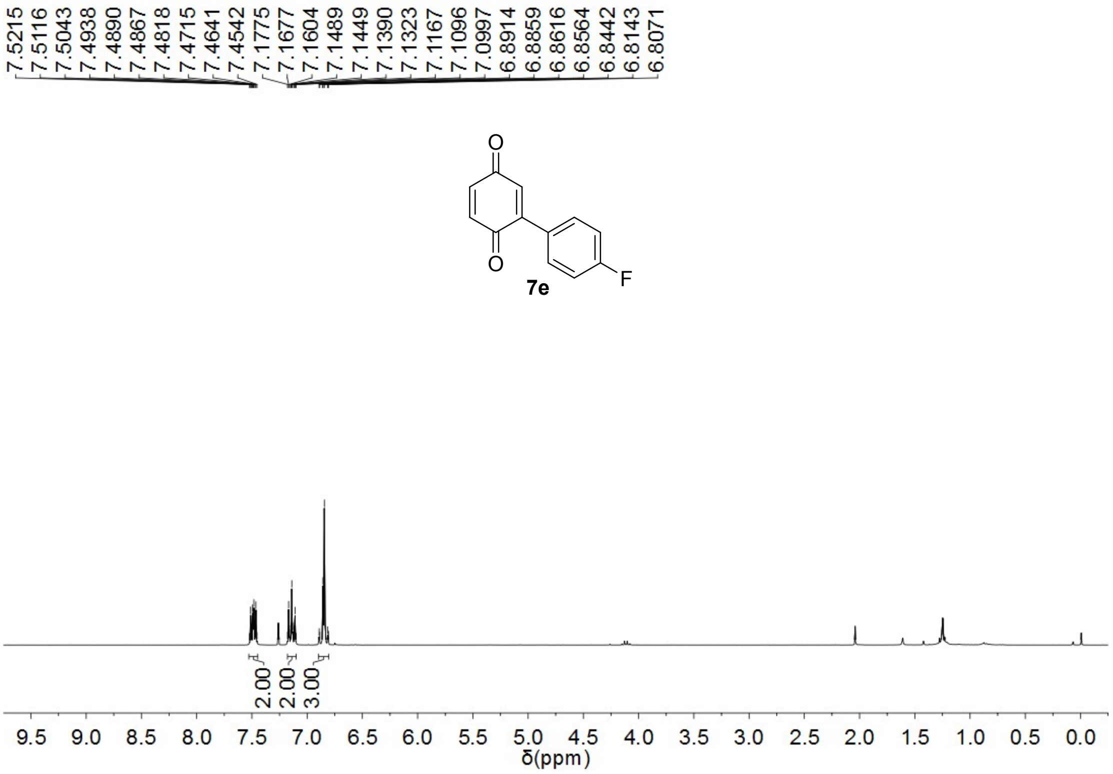
186.1239
144.7909
137.1050
136.5589
136.1843
133.7160
132.6230
132.1900
131.7555
131.3211
129.7458
125.6989
125.6499
125.6002
125.5504
125.5003
122.0894
O
O
CF3
7b
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
δ(ppm)

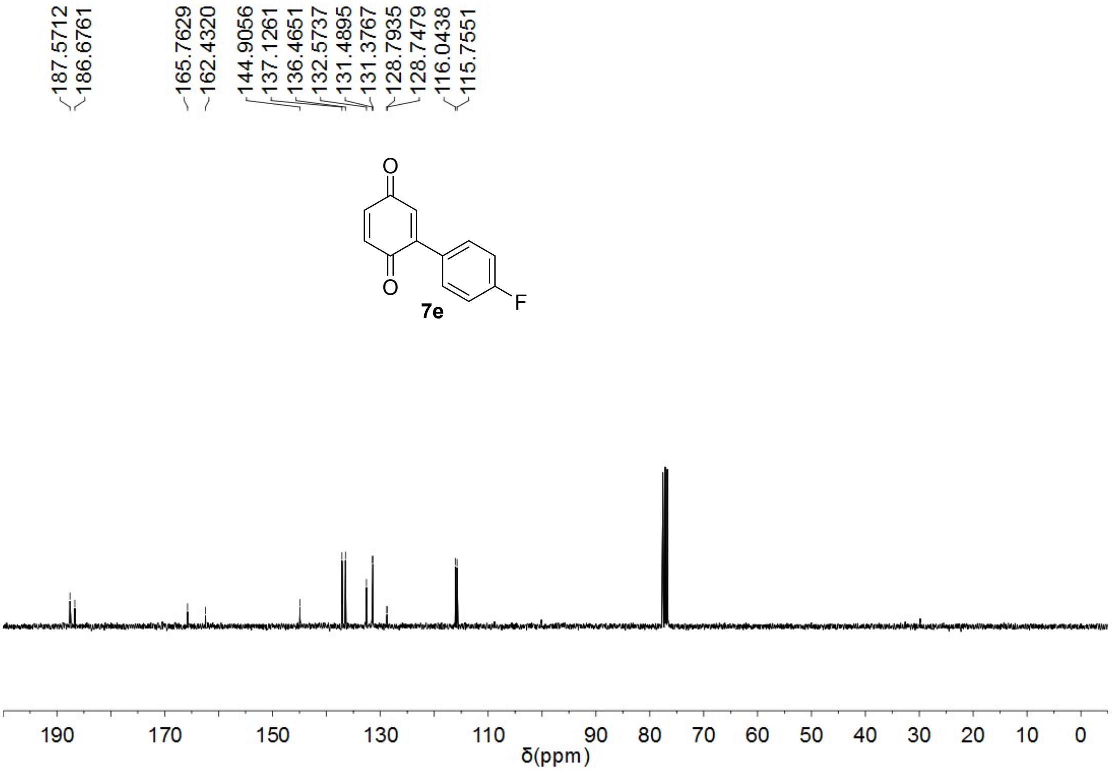

8.0076
7.9819
7.5669
7.5411
6.8792
6.8628
2.6127
O
O
Ac
7c
2.00
2.00
3.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ(ppm)

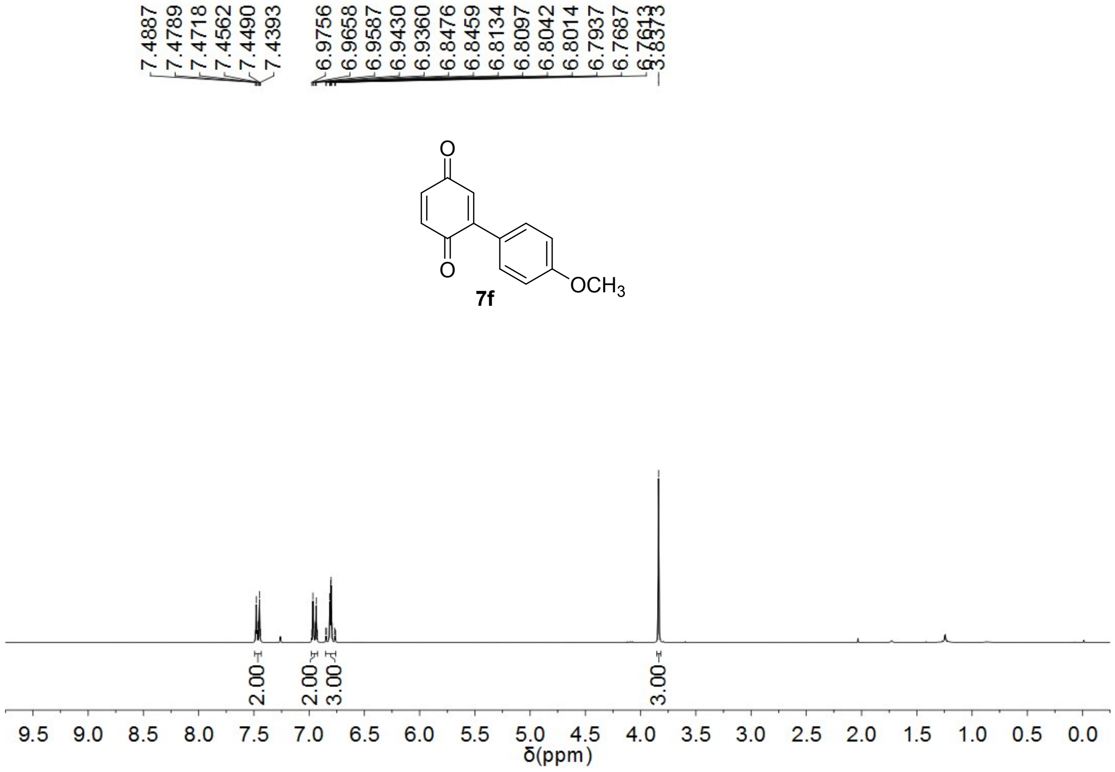

197.5308
187.2951
186.1701
144.9769
137.9309
137.0953
136.4704
133.5001
129.6113
128.4165
26.8262
O
O
Ac
7c
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
δ(ppm)

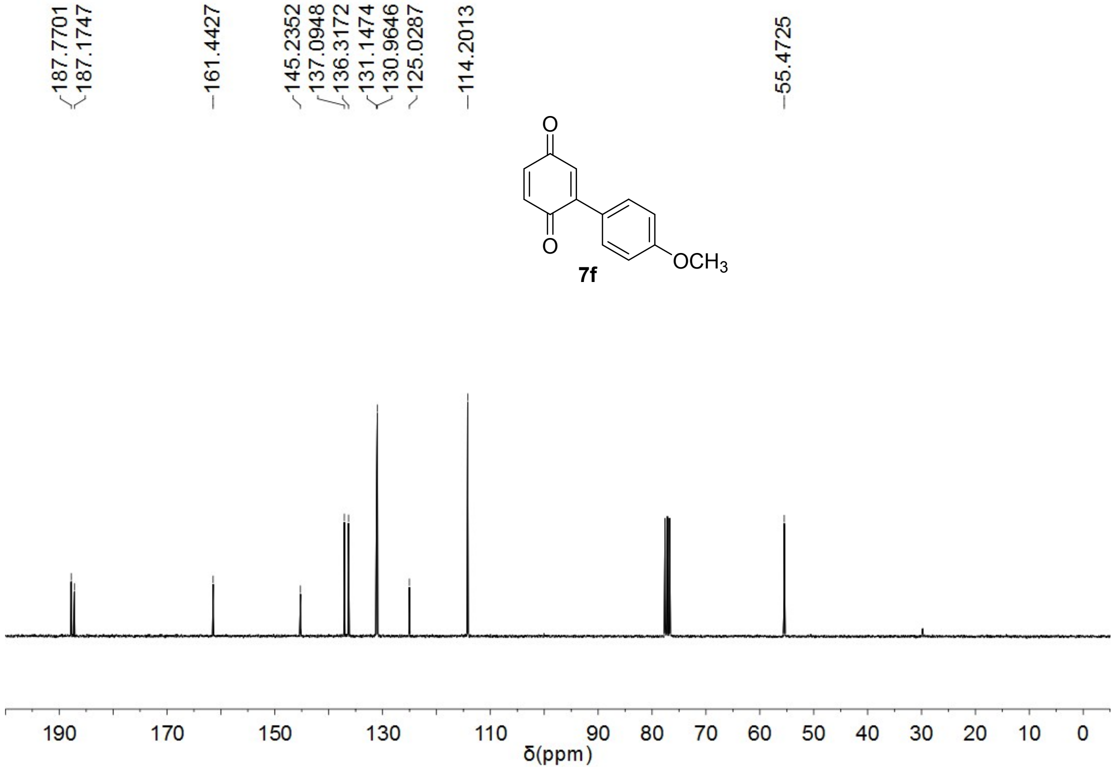

7.4467
7.4434
7.4162
7.3885
7.3855
6.8850
6.8827
6.8504
6.8463
6.8417
6.8339
6.8087
6.8016
O
O
Cl
7d
4.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ(ppm)

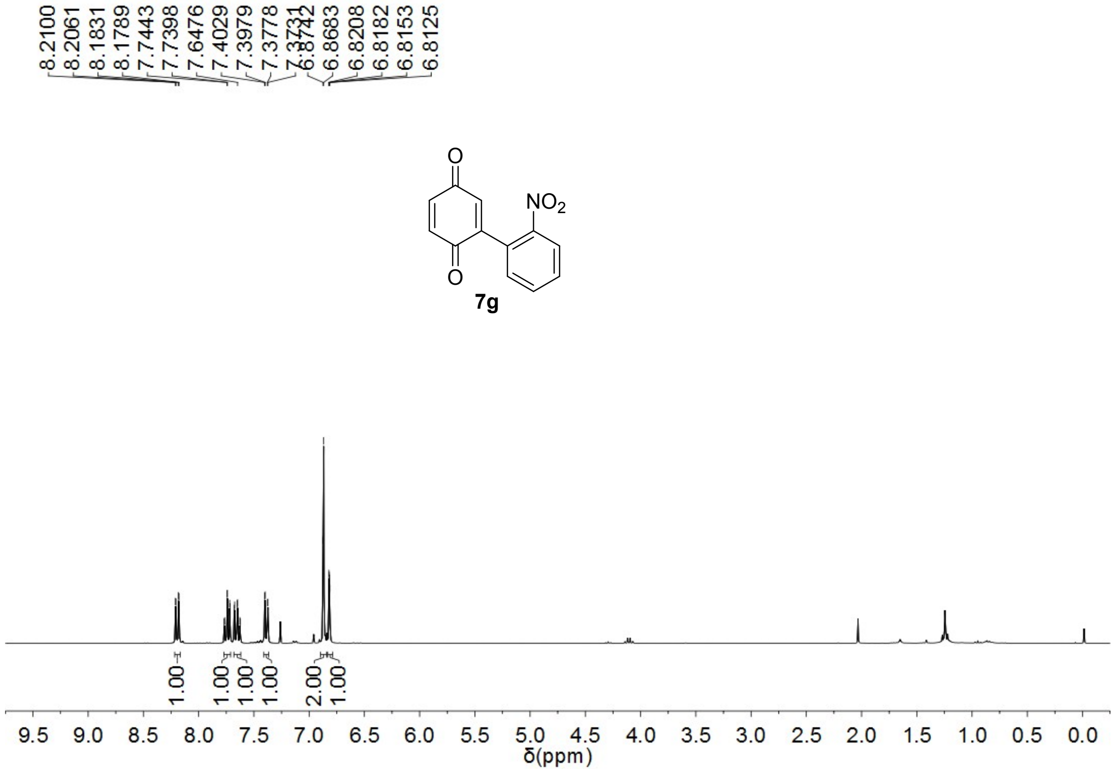

187.4406
186.4197
144.8101
137.1106
136.6398
136.4535
132.7569
131.0843
130.6633
128.9644
O
O
Cl
7d
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
δ(ppm)

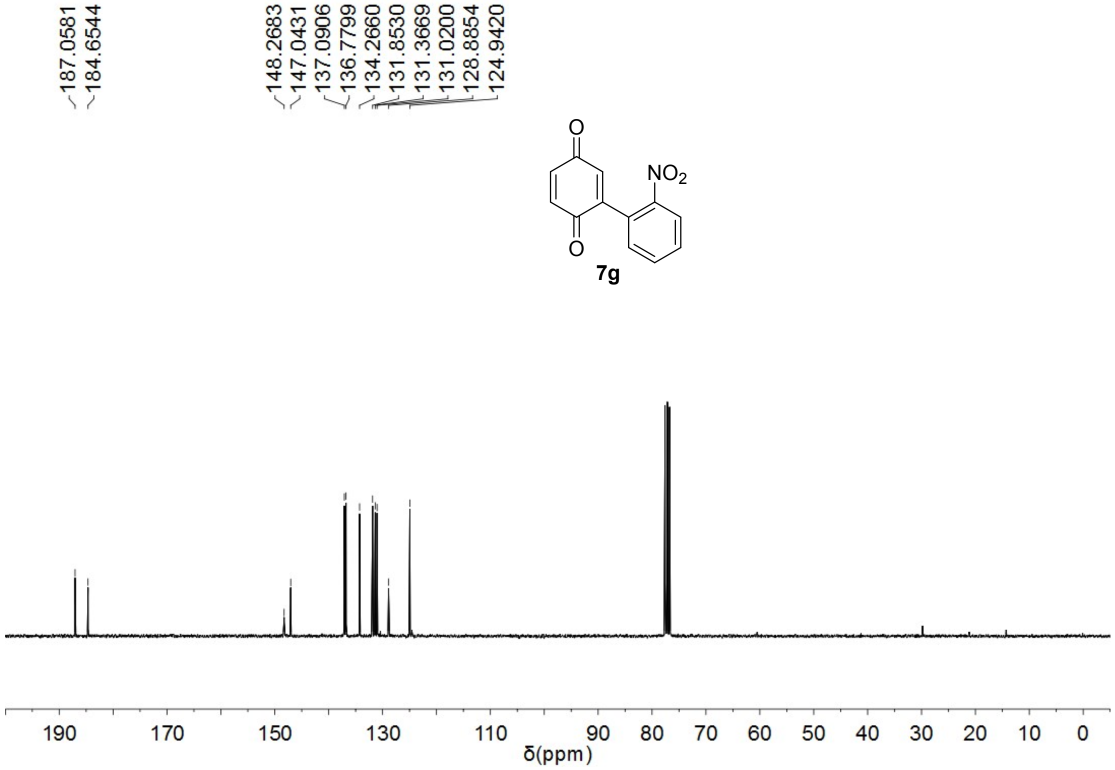

7.5215
7.5116
7.5043
7.4938
7.4890
7.4867
7.4818
7.4715
7.4641
7.4542
7.1775
7.1677
7.1604
7.1489
7.1449
7.1390
7.1323
7.1167
7.1096
7.0997
6.8914
6.8859
6.8616
6.8564
6.8442
6.8143
6.8071
O
O
F
7e
2.00
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ(ppm)

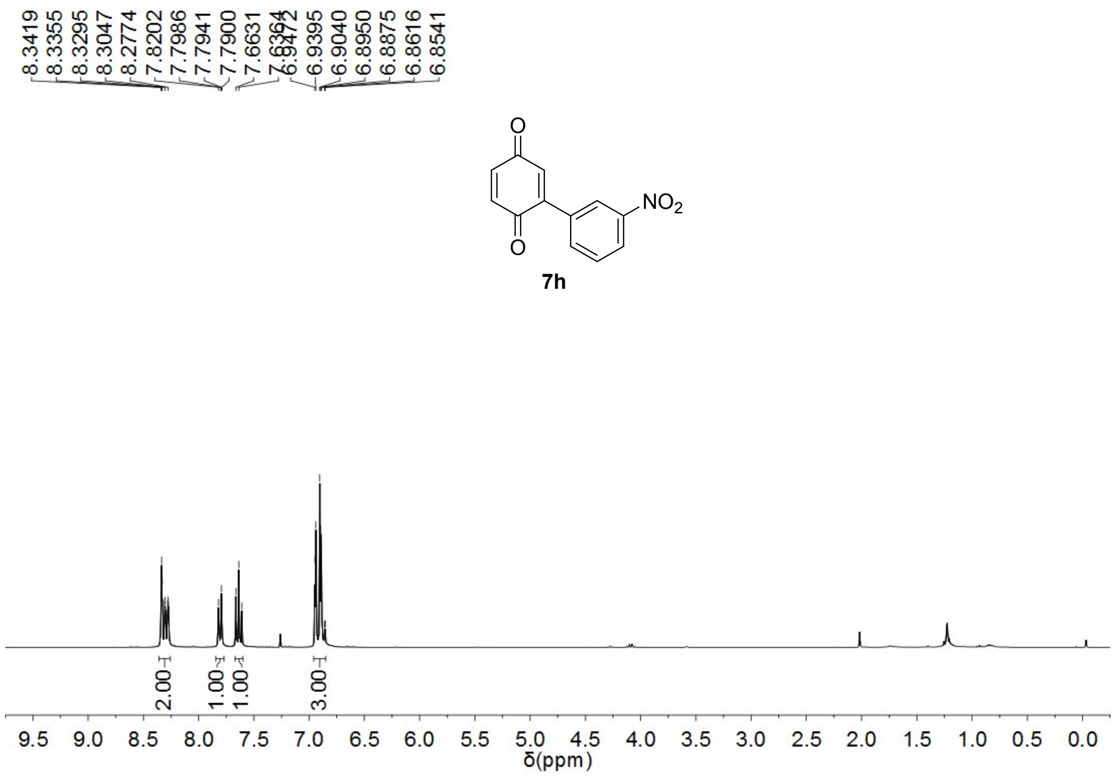

187.5712
186.6761
165.7629
162.4320
144.9056
137.1261
136.4651
132.5737
131.4895
131.3767
128.7935
128.7479
116.0438
115.7551
O
O
F
7e
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
δ(ppm)


7.4887
7.4789
7.4718
7.4562
7.4490
7.4393
6.9756
6.9658
6.9587
6.9430
6.9360
6.8476
6.8459
6.8134
6.8097
6.8042
6.8014
6.7937
6.7687
6.7613
3.8373
O
O
OCH3
7f
2.00
2.00
3.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ(ppm)

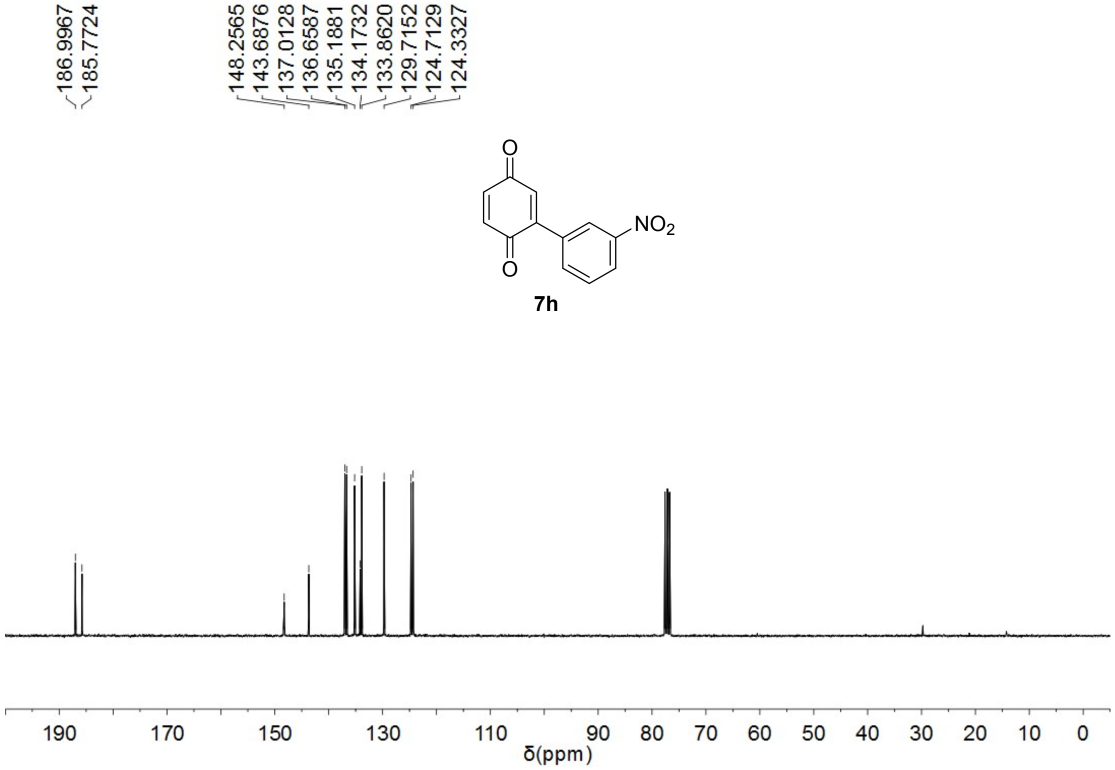

187.7701
187.1747
161.4427
145.2352
137.0948
136.3172
131.1474
130.9646
125.0287
114.2013
55.4725
O
O
OCH3
7f
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
δ(ppm)


8.2100
8.2061
8.1831
8.1789
7.7443
7.7398
7.6476
7.4029
7.3979
7.3778
7.3731
6.8742
6.8683
6.8208
6.8182
6.8153
6.8125
O
NO2
O
7g
1.00
1.00
1.00
1.00
2.00
1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
δ(ppm)


187.0581
184.6544
148.2683
147.0431
137.0906
136.7799
134.2660
131.8530
131.3669
131.0200
128.8854
124.9420
O
NO2
O
7g
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ(ppm)


8.3419
8.3355
8.3295
8.3047
8.2774
7.8202
7.7986
7.7941
7.7900
7.6631
7.6364
6.9472
6.9395
6.9040
6.8950
6.8875
6.8616
6.8541
O
O
NO2
2.00
1.00
1.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ(ppm)

7h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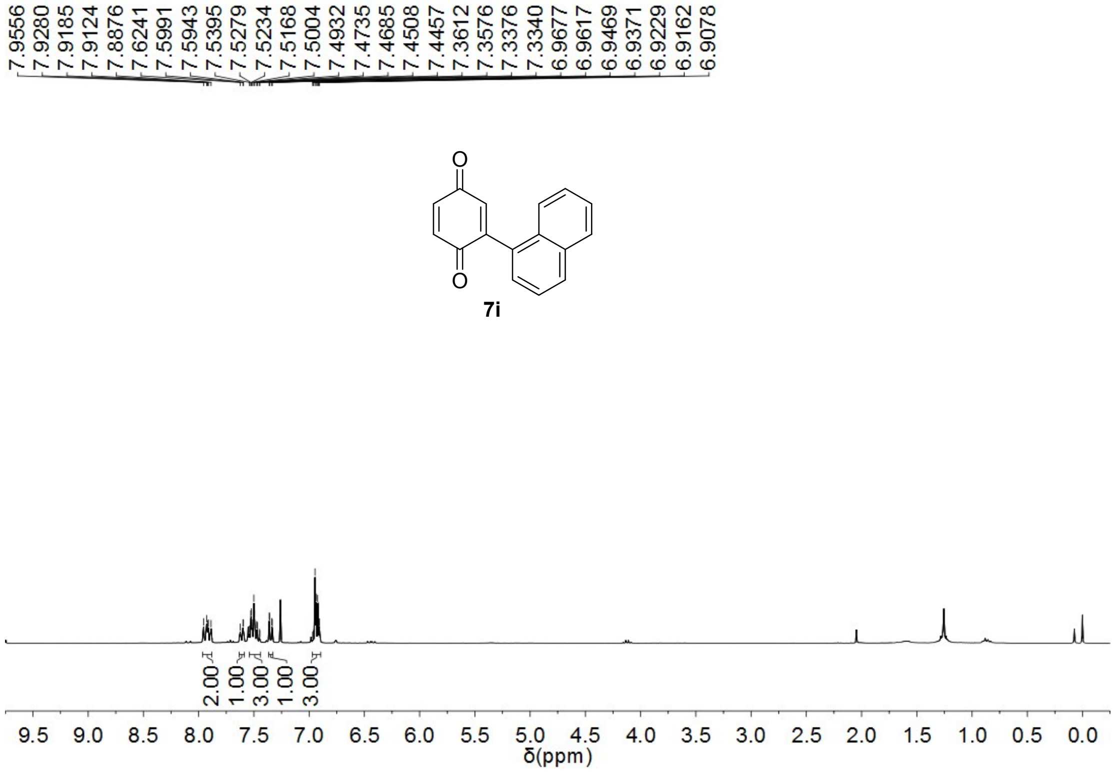
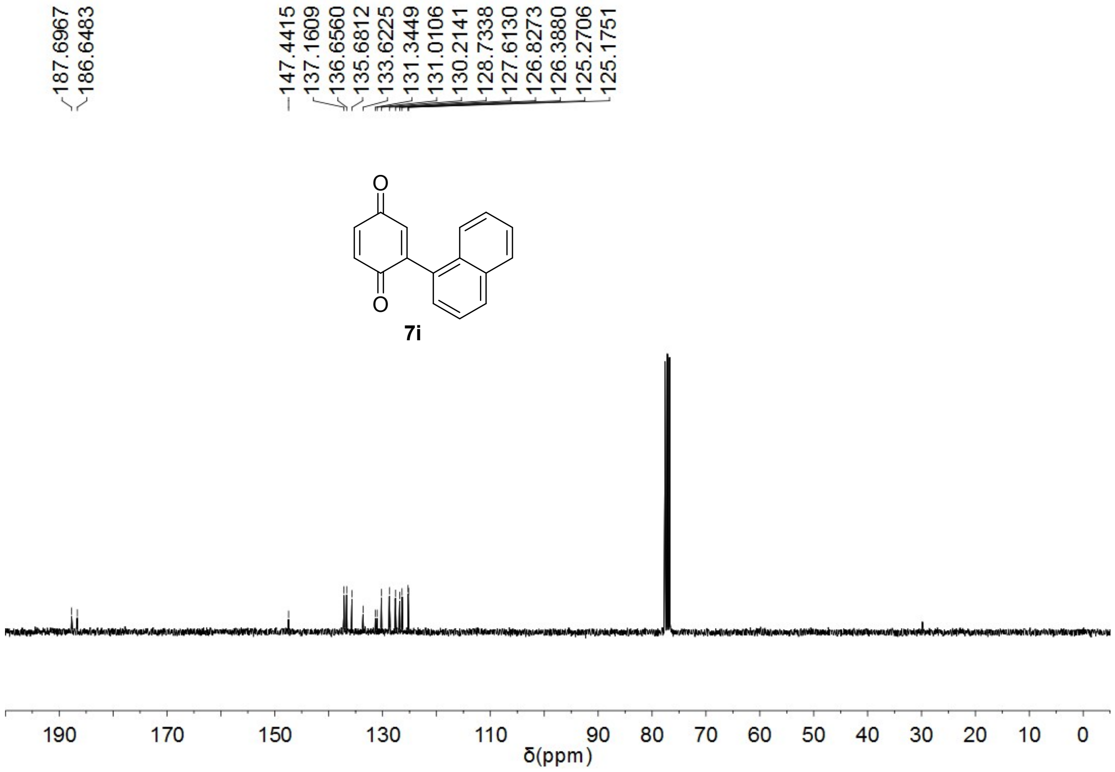
185.7724
148.2565
143.6876
137.0128
136.6587
135.1881
134.1732
133.8620
129.7152
124.7129
124.3327
O
O
NO2
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
δ(ppm)

7h


7.9556
7.9280
7.9185
7.9124
7.8876
7.6241
7.5991
7.5943
7.5395
7.5279
7.5234
7.5168
7.5004
7.4932
7.4735
7.4685
7.4508
7.4457
7.3612
7.3576
7.3376
7.3340
6.9677
6.9617
6.9469
6.9371
6.9229
6.9162
6.9078
O
O
2.00
1.00
3.00
1.00
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
δ(ppm)

7i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186.6483
147.4415
137.1609
136.6560
135.6812
133.6225
131.3449
131.0106
130.2141
128.7338
127.6130
126.8273
126.3880
125.2706
125.1751
O
O
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ(ppm)

7i