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Supporting Information

Synthesis of β-nitrostyrenes in the Presence of Sulfated Zirconia and Secondary Amines

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1. General methods

Commercially available reagents and solvents were used as received. Column chromatography was performed on Kiesel gel silica gel 60 (230-400 mesh). Melting points were determined using a Fisher-Johns apparatus and are uncorrected. The NMR spectra were obtained using Bruker Ascend-400 (400 MHz) and Bruker Avance DMX-500 (500 MHz) spectrometers. Chemical shifts (δ) are given in ppm and coupling constants *J* are given in hertz (Hz). Mass spectra (MS) were recorded on a GC-MS (Agilent Technologies 6890N, Detector 5973), in the chemical ionization mode using methane UAP grade as ionization gas. Microwave irradiation experiments were performed using a Discover System (CEM Corporation) single-mode microwave with standard sealed microwave glass vials. The nitrogen adsorption-desorption isotherm of sulfated zirconia was obtained at –196 °C on Micromeritics ASAP 2020 equipment. Powder X-ray diffraction (XRD) was performed using a Philips X'Pert Instrument with Cu-Kα radiation (45kV, 40 mA). Chemical composition of SZ was recorded by an EDX detector on a Zeiss SUPRA VP instrument. All nitrostyrenes **2a-m** are known compounds.

2. Experimental procedures

Synthesis and Characterization of Sulfated Zirconia (SZ)

Sulfated zirconia was synthesized according to our previously reported method.[1] Figure 1 shows XRD plot corresponding to the crystalline zirconia tetragonal as the predominantly phase which is given by reflections in 2θ = 30.18° as well as peaks in 34.616°, 35.283°, 43.002°, 50.214°, 50.770°, 59.291°, 60.187°, 62.724°, 72.894°, 74.617° and 81.768°. The nitrogen adsorption-desorption isotherm of SZ showed a profile corresponding to type IV of the IUPAC classification, typical for mesoporous materials (Figure 2). The SZ surface area, pore volume, and pore size were 90.75 $m^2 \cdot g^{-1}$, 0.12 $cm^3 \cdot g^{-1}$, and 52.7 Å, respectively (Table 1). SEM-EDX analysis revealed the presence of sulphur in the zirconia structure, as shown in the emission spectra of Figure 3 and elemental composition (Table 2). Once the sulfated zirconia was recycled is evident the monoclinic phase formation according with the intensification of peak in 2θ = 28° and the peak formation in 2θ = 32° (Figure 4 and 5), even though the sulphur content did not change significantly after the fourth reuse (Table 4).

a) Characterization of synthesised SZ

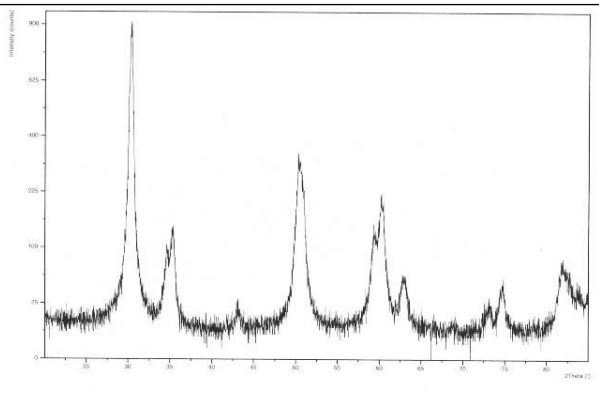


Figure 1. XRD pattern of synthesized SZ

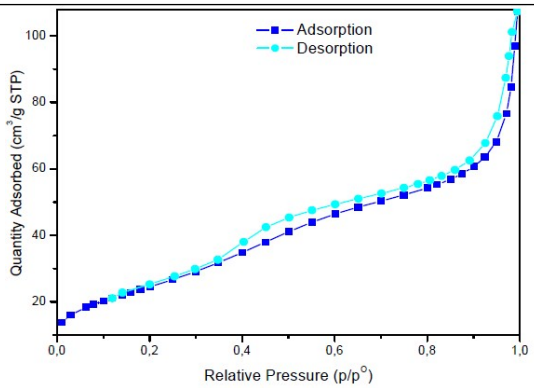


Figure 2. Nitrogen adsorption-desorption isotherm of synthesised SZ

Table 1. Textural properties of synthesised SZ

BET Area (m^2/g)	Pore Volume (cm^3/g)	Pore Size (Å)
90.75	0.12	52.73

Table 2. Composition of synthesised SZ

Element	**Weight%**
C	11.07
O	27.31
S	0.32
Zr	61.30
Total	100.00

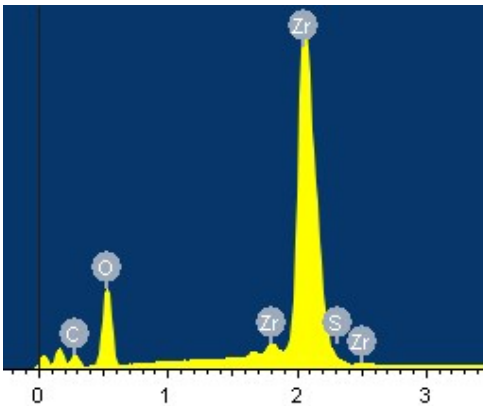


Figure 3. SEM-EDX spectra of synthesised SZ

b) Characterization of recovered SZ

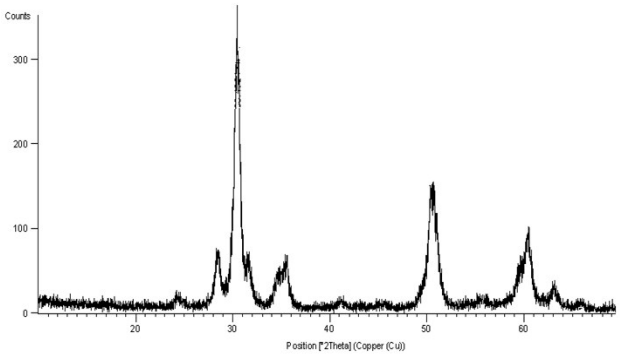


Figure 4. XRD pattern of recovered SZ

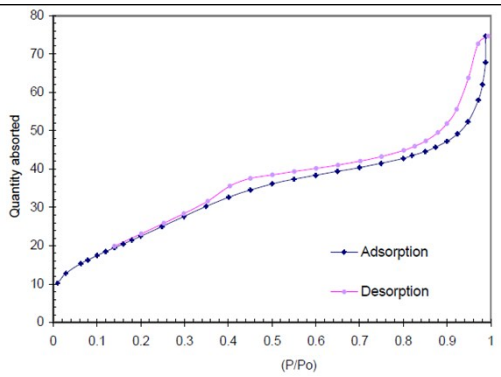


Figure 5. Nitrogen adsorption-desorption isotherm of recovered SZ

Table 3. Textural properties of recovered SZ

BET Area (m^2/g)	Pore Volume (cm^3/g)	Pore Size (Å)
86.15	0.089	41.62

Table 4. Composition of recovered SZ

Element	Weight%
C	23.36
O	26.71
S	0.37
Zr	49.56
Totals	100.00

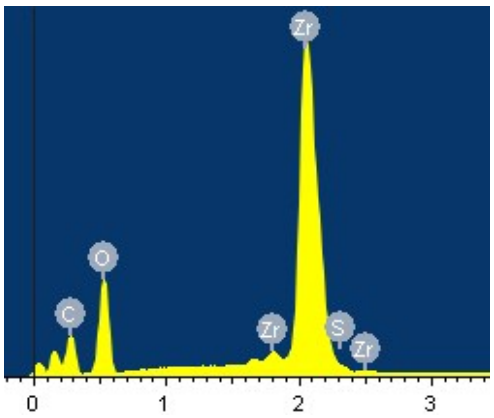


Figure 6. SEM-EDX spectra of recovered SZ

Identification of the Brønsted and Lewis acids sites present in synthesised SZ

The quantification of acids sites presents in the material was determined by FT-IR (Nicolet 750 Spectrometer). The sample was passed into thin wafers (10 mg cm^{-2}) and pre-treated in a quartz cell under vacuum (10^{-6}mbar) at 450 °C during 4h. Then the sample was cooled at room temperature and expose to pyridine (1 μL) (P_{eq} = 2-3 mbar). The pyridine excess was removed under vacuum (1 h). Finally, the spectra were recorded at 50-400 °C (Figure 7) and the concentration of Brønsted and Lewis acids sites (Table 5) were calculated by the following equation[2]:

$n_i = A_i a_c / \varepsilon_i m$

n_i is the amount of type i acid sites (μmmol g^{-1})

A_i is the integrated absorbance in cm^{-1}

a_c is the cross-sectional area in square centimeter of the wafer

ε_i is the integrated molar extinction coefficient in cm μmmol^{-1}

m is the mass of the sample

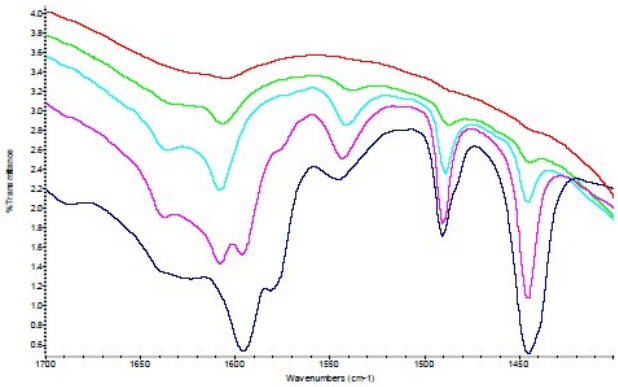


Fig. 7 FT-IR spectra of SZ after pyridine desorption at different temperatures: a) dark blue 50 °C, purple 100 °C, turquoise 200 °C, green 300 °C and red 400 °C

Table 5. The nitrogen adsorption–desorption analysis parameters of materials

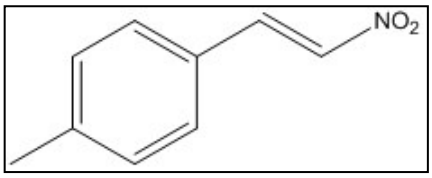
Entry	Parameters	Synthesised SZ	Recovered SZ
1	S_{BET} ($m^2 \cdot g^{-1}$)	90.75	86.15
2	Pore volume ($cm^3 \cdot g^{-1}$)	0.12	0.089
3	Pore size (Å)	52.73	41.62

General procedure for the synthesis of β-nitrostyrenes 2a-m

A mixture of SZ (50 mg) and amine (0.1 mmol, 10 µL) in dry toluene (1mL) was placed in a microwave tube having a magnetic stirrer. Subsequently, aromatic aldehyde (1 mmol) and nitromethane (3 mmol, 0.15 mL) were added to the mixture, which was heated under microwave irradiation (30 W, 110 °C) during 30 minutes. Then, SZ was removed by centrifugation and washed with CH_2Cl_2 (5x5mL). The combined organic extracts were evaporated, giving the corresponding β-nitrostyrene, which was purified by column chromatography (CH_2Cl_2 or hexanes-EtOAc 1:1) and recrystallization (CH_2Cl_2-hexanes, 1:2).

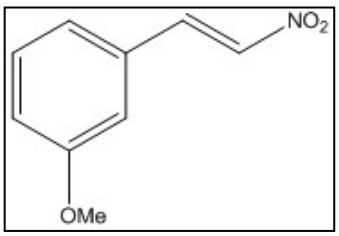
3. Characterization data

(*E*)-1-methyl-4-(2-nitrovinyl)benzene (2a)



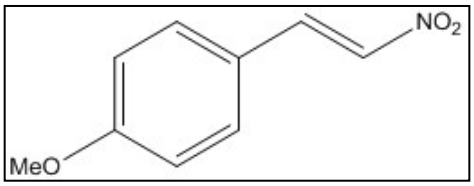
The compound **2a** was prepared from **1a** (0.1 mL, 1 mmol) and nitromethane (0.15 mL, 3mmol) according to General Procedure. Yield: 137 mg (84%); light yellow solid; mp = 99-101°C (Lit.[3] mp=102°C). ^{1}H NMR (400 MHz, DMSO-d_6): δ = 2.36 (s, 3H, CH_3), 7.29 (d, *J* = 8.0 Hz, 2H, ArH), 7.74 (d, *J* = 8.0 Hz, 2H, ArH), 8.08 (d, *J* = 13.6 Hz, 1H, =CH), 8.17 (d, *J* = 13.6 Hz, 1H, =CH). ^{13}C NMR (100.6 MHz, DMSO-d_6): δ = 21.6 (CH_3), 128.0 (C_{ipso}), 130.28(2xArCH), 130.32 (2xArCH), 137.7 (=CH), 139.8 (=CH), 143.0 (C_{ipso}). MS (CI) ($C_9H_9NO_2$): *m/z* =204 $[M+41]^+$, 192 $[M+29]^+$, 164 $[M+1]^+$, 149, 121.

(*E*)-1-methoxy-3-(2-nitrovinyl)benzene (2b)



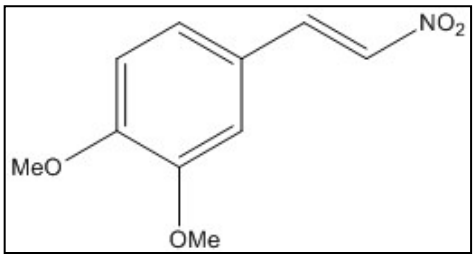
The compound **2b** was prepared from **1b** (136.15 mg, 1 mmol) and nitromethane (0.15 mL, 3mmol) according to General Procedure. Yield: 150 mg (84%); light yellow solid; mp = 91-95°C (Lit.[3] mp=93-94°C). ^{1}H NMR (500 MHz, DMSO-d_6): δ = 3.81 (s, 3H, OCH_3), 7.08-7.11 (m, 1H, ArH), 7.37-7.42 (m, 2H, ArH), 7.45-7.47 (m, 1H, ArH), 8.09 (d, *J* = 13.6 Hz, 1H, =CH), 8.26 (d, *J* = 13.6 Hz, 1H, =CH). ^{13}C NMR (125.7 MHz, DMSO-d_6): δ = 55.8 (OCH_3), 114.5 (ArCH), 118.8(ArCH), 123.1 (ArCH), 130.6 (ArCH), 132.1 (C_{ipso}), 138.8 (=CH), 139.6 (=CH), 160.1 (C_{ipso}). MS (CI) ($C_9H_9NO_3$): *m/z* = 220 $[M+41]^+$, 208 $[M+29]^+$, 180 $[M+1]^+$, 165, 152, 137.

(*E*)-1-methoxy-4-(2-nitrovinyl)benzene (2c)



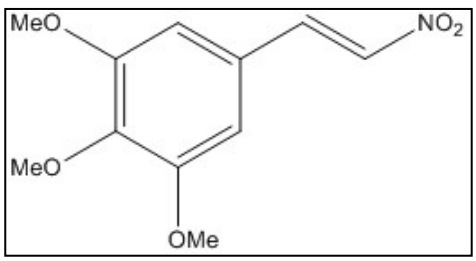
The compound **2c** was prepared from **1c** (136.15 mg, 1 mmol) and nitromethane (0.15 mL, 3mmol) according to General Procedure. Yield: 131 mg (73%); light yellow solid; mp = 84-86°C (Lit.[4] mp = 86-87°C). ^{1}H NMR (400 MHz, DMSO-d_6):δ = 3.83 (s, 3H, OCH_3), 7.05 (d, *J* = 8.8 Hz, 2H, ArH), 7.83 (d, *J* = 8.8 Hz, 2H, ArH), 8.09 (d, *J* = 13.5 Hz, 1H, =CH), 8.13 (d, *J* = 13.5 Hz, 1H,=CH). ^{13}C NMR (100.6 MHz, DMSO-d_6): δ = 56.0 (OCH_3), 115.3 (2xArCH), 123.2 (C_{ipso}), 132.4 (2xArCH), 136.2 (=CH), 139.8 (=CH), 163.0 (C_{ipso}). MS (CI) ($C_9H_9NO_3$): *m/z* = 220 $[M+41]^+$, 208 $[M+29]^+$, 180 $[M+1]^+$, 165, 150, 137.

(*E*)-1,2-dimethoxy-4-(2-nitrovinyl)benzene (2d)



The compound **2d** was prepared from **1d** (166.18 mg, 1 mmol) and nitromethane (0.15 mL, 3mmol) according to General Procedure. Yield: 130 mg (62%); yellow solid; mp = 140-142°C (Lit.[3] mp = 140-141°C). ^{1}H NMR (400 MHz, DMSO-d_6): δ = 3.82 (s, 3 H, OCH_3), 3.83 (s, 3 H, OCH_3), 7.06 (d, *J* = 8.3 Hz, 1H, ArH), 7.43 (dd, *J* = 1.8, 8.3 Hz, 1H, ArH), 7.49(d, *J* = 1.8 Hz, 1H, ArH), 8.07 (d, *J*= 13.5 Hz, 1H, =CH), 8.21 (d, *J* = 13.5 Hz, 1H, =CH). ^{13}C NMR (100.6 MHz, DMSO-d_6): δ = 56.2 (OCH_3), 56.3 (OCH_3), 111.8 (ArCH), 112.2 (ArCH), 123.4 (C_{ipso}), 126.1 (ArCH), 136.4 (=CH), 140.3 (=CH), 149.7 (C_{ipso}), 153.0 (C_{ipso}). MS (CI) ($C_{10}H_{11}NO_4$): *m/z* = 250 $[M+41]^+$, 238 $[M+29]^+$, 210 $[M+1]^+$, 194, 178, 167.

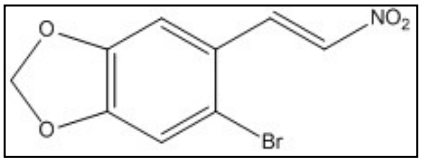
(*E*)-1,2,3-trimethoxy-5-(2-nitrovinyl)benzene (2e)



The compound **2e** was prepared from **1e** (196.20 mg, 1 mmol) and nitromethane (0.15 mL, 3mmol) according to General Procedure. Yield: 174 mg (73%), yellow solid; mp = 114-116°C (Lit.[2] mp=120°C). ^{1}H NMR (400 MHz, DMSO-d_6): δ = 3.73 (s, 3H, OCH_3), 3.83 (s, 6H, OCH_3), 7.24 (s, 2H, ArH), 8.06 (d, *J* = 13.5 Hz, 1H, =CH), 8.28 (d, *J* = 13.5, 1H, =CH). ^{13}C NMR(100.6 MHz, DMSO-d_6): δ = 56.7 (2xOCH_3), 60.7 (OCH_3), 108.2 (2xArCH), 126.1 (C_{ipso}), 137.9 (=CH), 140.1 (=CH),

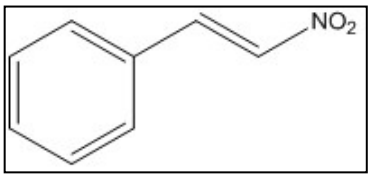
141.5 (C_{ipso}), 153.6 (2xC_{ipso}). MS (CI) ($C_{11}H_{13}NO_5$): *m/z* = 280 $[M+41]^+$, 268 $[M+29]^+$, 240 $[M+1]^+$, 224.

(*E*)-5-bromo-6-(2-nitrovinyl)benzo[*d*][1,3]dioxole (2f)



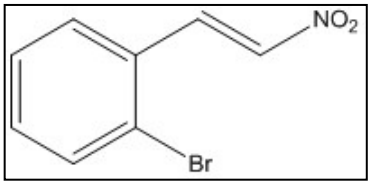
The compound **2f** was prepared from **1f** (229.03 mg, 1 mmol) and nitromethane (0.15 mL, 3mmol) according to General Procedure. Yield: 176 mg (65%); light yellow solid; mp = 156-158°C. ^{1}H NMR (400 MHz, DMSO-d_6): δ = 6.19 (s, 2H, OCH_2O), 7.41 (s, 1 H, ArH), 7.67 (s, 1 H, ArH), 8.19 (s, 2H, =CH). ^{13}C NMR (100.6 MHz, DMSO-d_6): δ = 103.6 (OCH_2O), 108.0 (ArCH), 113.6 (ArCH), 120.1 (C_{ipso}), 123.1 (C_{ipso}), 137.2 (=CH), 138.9 (=CH), 148.6 (C_{ipso}), 152.2 (C_{ipso}). MS (CI) ($C_9H_6BrNO_4$): *m/z* = 314, 312 $[M+41]^+$, 302, 300 $[M+29]^+$, 274, 272 $[M+1]^+$, 259, 257, 244, 242, 231, 229, 228, 226, 193, 176, 162.

(*E*)-1-(2-nitrovinyl)benzene (2g)



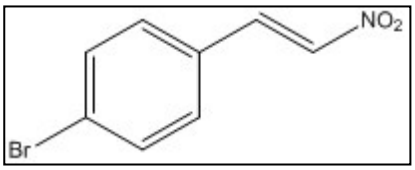
The compound **2g** was prepared from **1g** (0.1 mL, 1 mmol) and nitromethane (0.15 mL, 3mmol) according to General Procedure. Yield: 104 mg (70%); light yellow solid; mp = 55-57°C (Lit.2 mp = 56°C). ^{1}H NMR (500 MHz, DMSO-d_6): δ = 7.46-7.50 (m, 2H, ArH), 7.52-7.56 (m, 1H, ArH), 7.84-7.88 (m, 2H, ArH), 8.13 (d, *J* = 13.6 Hz, 1H, =CH), 8.22 (d, *J* = 13.6 Hz, 1H, =CH). ^{13}C NMR (125.7 MHz, DMSO-d_6): δ = 129.6 (2xArCH), 130.2 (2xArCH), 130.8 (C_{ipso}), 132.5 (ArCH), 138.5 (=CH), 139.7 (=CH). MS (CI) ($C_8H_7NO_2$): *m/z* = 190 $[M+41]^+$, 178 $[M+29]^+$, 150 $[M+1]^+$, 135, 107.

(*E*)-1-bromo-2-(2-nitrovinyl)benzene (2h)



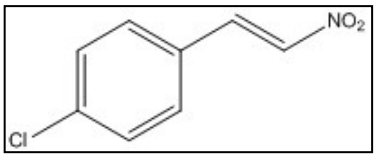
The compound **2h** was prepared from **1h** (0.11 mL, 1 mmol) and nitromethane (0.15 mL, 3mmol) according to General Procedure. Yield: 192 mg (84%); light yellow solid; mp = 82-84°C (Lit.2 mp = 86°C). ^{1}H NMR (500 MHz, DMSO-d_6): δ = 7.44-7.51 (m, 2H, ArH), 7.77-7.80 (m, 1H, ArH), 8.00-8.03 (m, 1H, ArH), 8.22 (d, *J* = 13.5 Hz, 1H, =CH), 8.25 (d, *J* = 13.5 Hz, 1H, =CH). ^{13}C NMR (125.7 MHz, DMSO-d_6): δ = 125.7 (C_{ipso}), 128.4 (ArCH), 129.5 (ArCH), 129.7 (C_{ipso}), 133.5 (2xArCH), 136.4 (=CH), 140.1 (=CH). MS (CI) ($C_8H_6BrNO_2$): *m/z* = 270, 268 $[M+41]^+$, 258, 256 $[M+29]^+$, 230, 228 $[M+1]^+$, 215, 213, 202, 200, 187, 185, 149, 132.

(*E*)-1-bromo-4-(2-nitrovinyl)benzene(2i)



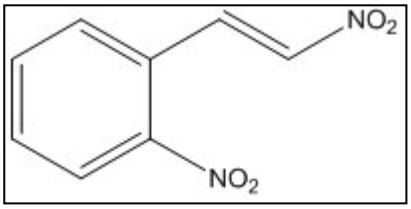
The compound **2i** was prepared from **1i** (185.02 mg, 1 mmol) and nitromethane (0.15 mL, 3mmol) according to General Procedure. Yield: 169mg (74%); yellow solid; mp = 141-143°C (Lit.[5] mp = 148-150°C). ^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.70 (d, *J* = 8.6 Hz, 2H, ArH), 7.82 (d, *J* = 8.6 Hz, 2H, ArH), 8.12 (d, *J* =13.6 Hz, 1H, =CH), 8.26 (d, *J* = 13.6 Hz, 1H, =CH). ^{13}C NMR (100.6 MHz, DMSO-d_6): δ = 126.2 (C_{ipso}), 130.1 (C_{ipso}), 132.1 (2xArCH), 132.7 (2xArCH), 138.5 (=CH), 139.1 (=CH). MS (CI) ($C_8H_6BrNO_2$): *m/z* = 270, 268 $[M+41]^+$, 258, 256 $[M+29]^+$, 230, 228 $[M+1]^+$, 215, 213, 187, 185, 150.

(*E*)-1-chloro-4-(2-nitrovinyl)benzene (2j)



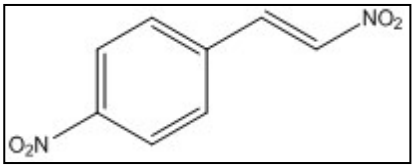
The compound **2j** was prepared from **1j** (140.57 mg, 1 mmol) and nitromethane (0.15 mL, 3mmol) according to General Procedure. Yield: 150 mg (82%); yellow solid; mp = 111-113ºC (Lit.4 mp = 113-114ºC). ^{1}H NMR (500 MHz, DMSO-d_6): δ = 7.55 (d, *J* = 8.5 Hz, 2H, ArH), 7.89 (d, *J* = 8.4 Hz, 2H, ArH), 8.13 (d, *J* = 13.6 Hz, 1H, =CH), 8.26 (d, *J* = 13.6 Hz, 1H, =CH). ^{13}C NMR (125.7 MHz, DMSO-d_6): δ = 129.7 (2xArCH), 129.8 (C_{ipso}), 131.9 (2xArCH), 137.2 (C_{ipso}), 138.4 (=CH), 139.0 (=CH). MS (CI) ($C_8H_6ClNO_2$): *m/z* = 226, 224 $[M+41]^+$, 214, 212 $[M+29]^+$, 186, 184 $[M+1]^+$, 143, 141.

(*E*)-1-nitro-2-(2-nitrovinyl)benzene (2k)



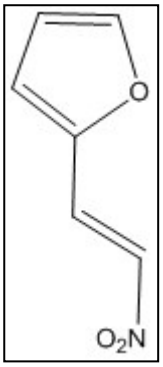
The compound **2k** was prepared from **1k** (151.12 mg, 1 mmol) and nitromethane (0.15 mL, 3mmol) according to General Procedure. Yield: 78 mg (40%); orange solid; mp = 103-105ºC (Lit.[6] mp = 103-105°C). ^{1}H NMR(500 MHz, DMSO-d_6): δ = 7.78 (td, *J* = 1.4, 7.9 Hz, 1H, ArH), 7.86 (td, *J* = 0.8, 7.5 Hz, 1H, ArH), 7.97 (dd, *J* = 1.4, 7.7 Hz, 1H, ArH), 8.12 (d, *J* = 13.5, 1H, =CH), 8.20 (dd, *J* = 1.2, 8.1, 1H, ArH), 8.43 (d, *J* =13.5 Hz, 1H, =CH). ^{13}C NMR (125.7 MHz, DMSO-d_6): δ = 125.7 (ArCH), 126.2 (C_{ipso}), 130.7 (ArCH), 132.7 (ArCH), 134.7 (ArCH), 135.9 (=CH), 140.9(=CH), 148.9 (C_{ipso}). MS (CI) ($C_8H_6N_2O_4$): *m/z* = 235 $[M+41]^+$, 223 $[M+29]^+$, 195 $[M+1]^+$, 148, 134, 120, 92.

(*E*)-1-nitro-4-(2-nitrovinyl)benzene (2l)



The compound **2l** was prepared from **1l** (151.12 mg, 1 mmol) and nitromethane (0.15 mL, 3mmol) according to General Procedure. Yield: 138 mg (71%); orange solid; mp = 196-198°C (Lit.[5] mp = 189-192°C). ^{1}H NMR (500 MHz, DMSO-d_6): δ = 8.11 (d, *J* = 8.9 Hz, 2H, ArH), 8.23 (d, *J* = 13.7 Hz, 1H, =CH), 8.29 (d, *J* = 8.8 Hz, 2H, ArH), 8.36 (d, *J* = 13.7 Hz, 1H, =CH). ^{13}C NMR (125.7 MHz, DMSO-d_6): δ = 124.5 (2xArCH), 131.3 (2xArCH), 137.0 (=CH), 137.3 (C_{ipso}), 141.4 (=CH), 149.3 (C_{ipso}). MS (CI) ($C_8H_6N_2O_4$): *m/z* = 235 $[M+41]^+$, 223 $[M+29]^+$, 195 $[M+1]^+$, 165, 152.

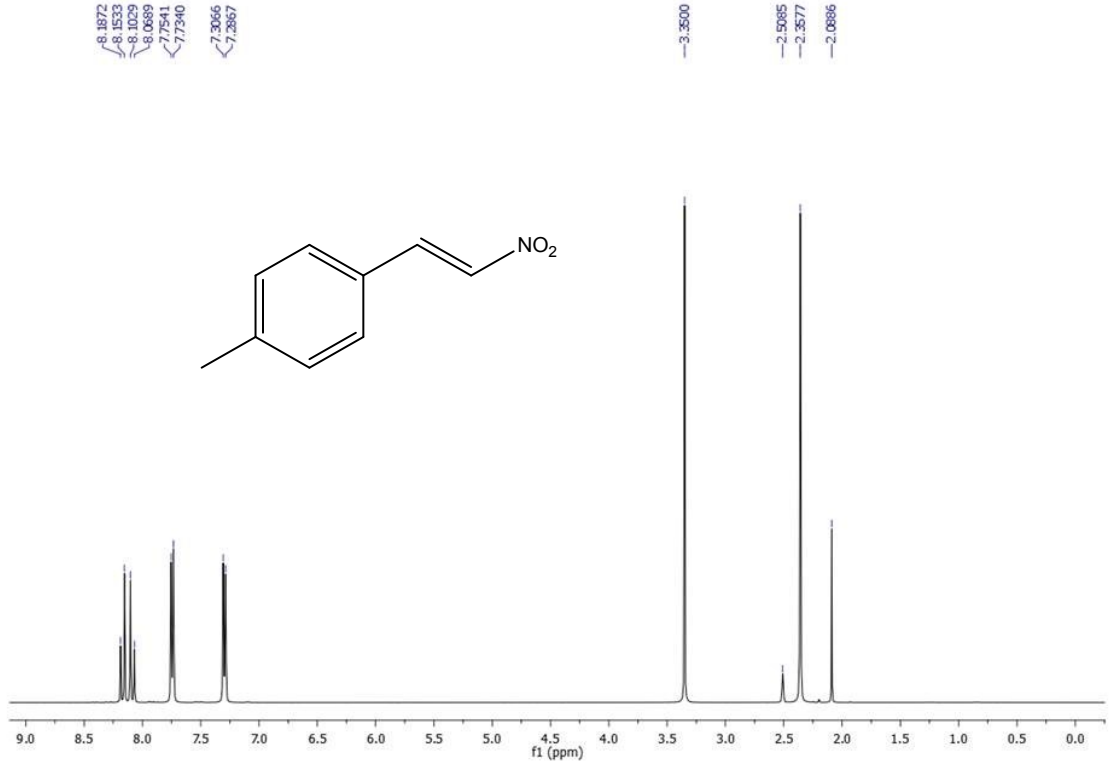
(*E*)-2-(2-nitrovinyl)furan (2m)

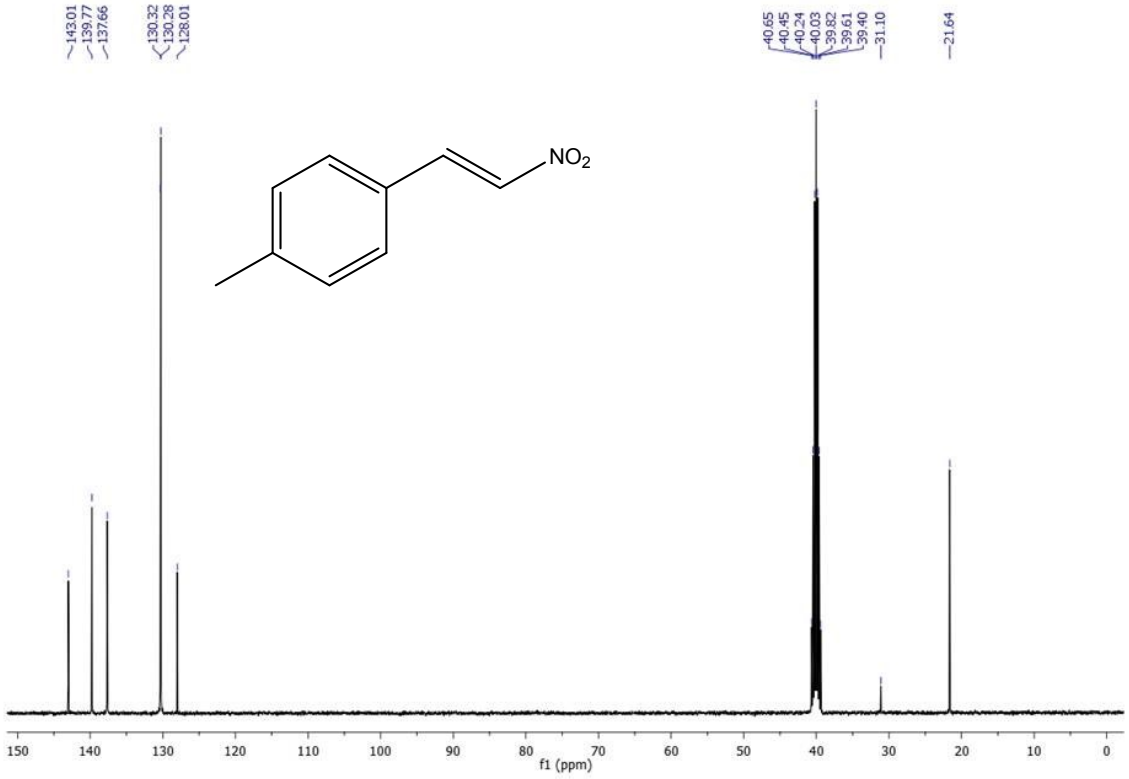


The compound **2m** was prepared from **1m** (0.08 mL, 1 mmol) and nitromethane (0.15 mL, 3mmol) according to General Procedure. Yield: 122 mg (88%); light yellow solid; mp = 71-73°C (Lit.[2] mp = 72°C). ^{1}H NMR (400 MHz, DMSO-d_6): δ = 6.76 (dd, *J* = 1.8, 3.5 Hz, 1H, ArH), 7.29 (d, *J* = 3.5 Hz, 1H, ArH), 7.75 (d, *J* = 13.3 Hz, 1H, =CH), 8.01 (d, *J* = 1.3 Hz, 1H, ArH), 8.02 (d, *J* = 13.3Hz, 1H, =CH). ^{13}C NMR (100.6 MHz, DMSO-d_6): δ = 114.2 (ArCH), 121.6 (ArCH), 126.6 (=CH), 134.9 (=CH), 146.9 (C_{ipso}), 148.6 (ArCH). MS (CI) ($C_6H_5NO_3$): *m/z* = 180 $[M+41]^+$, 168 $[M+29]^+$, 140 $[M+1]^+$, 97.

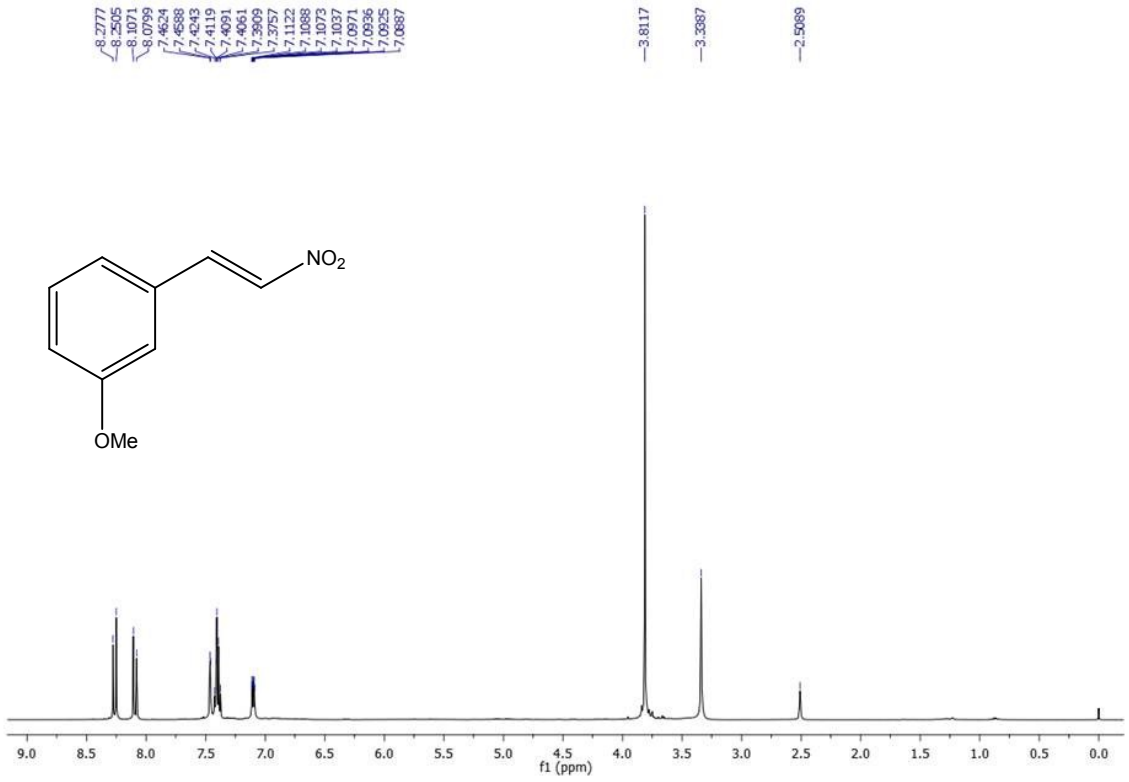
4. ^{1}H and ^{13}C NMR spectra for compounds 2a-2m

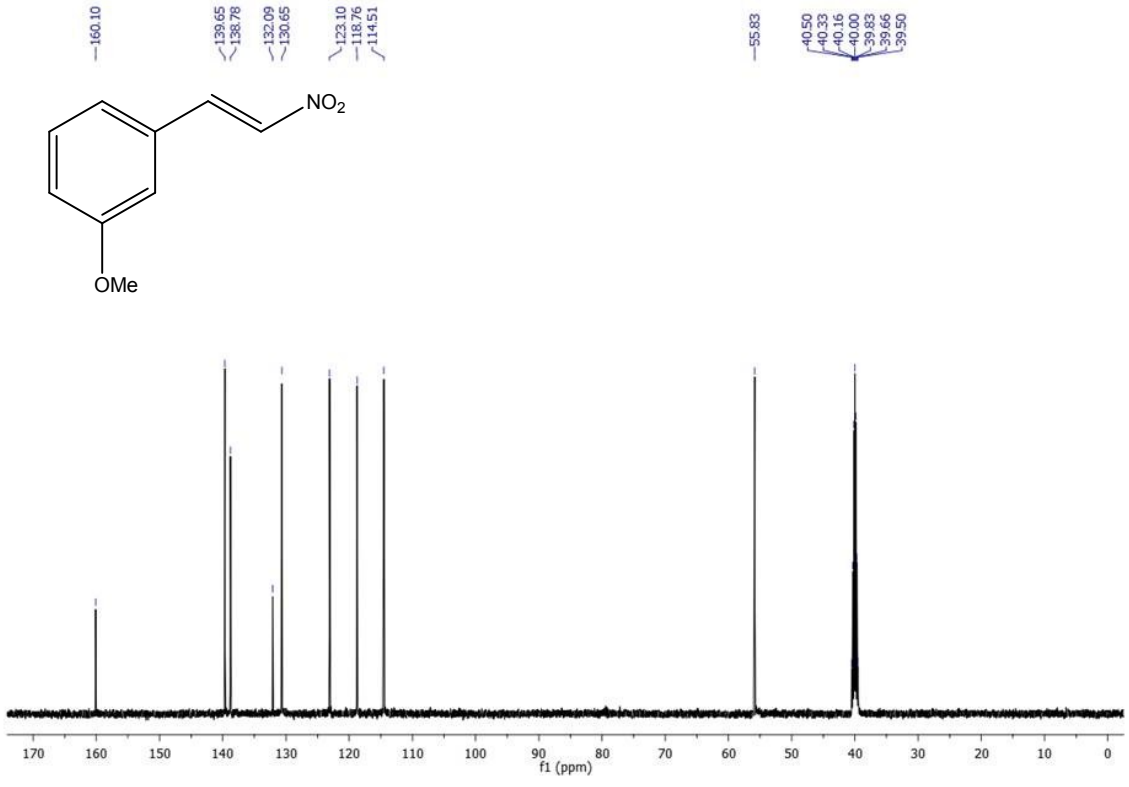
^{1}H NMR and ^{13}C NMR for compound 2a



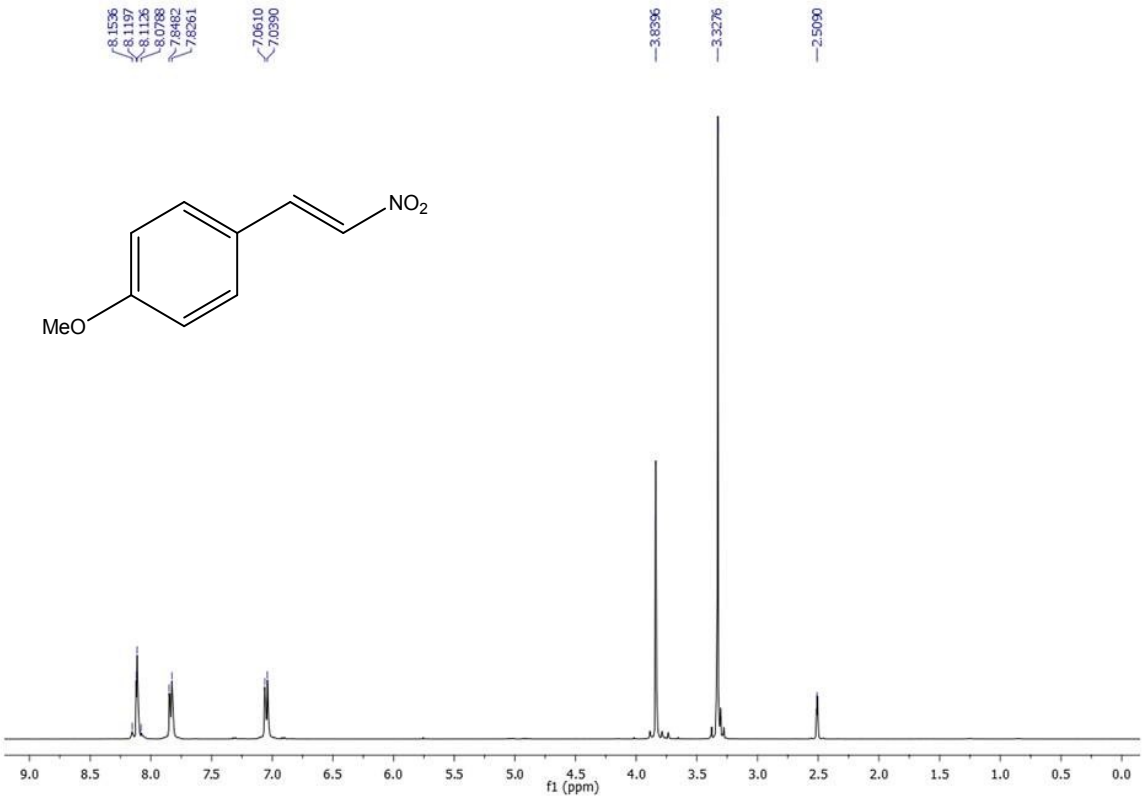


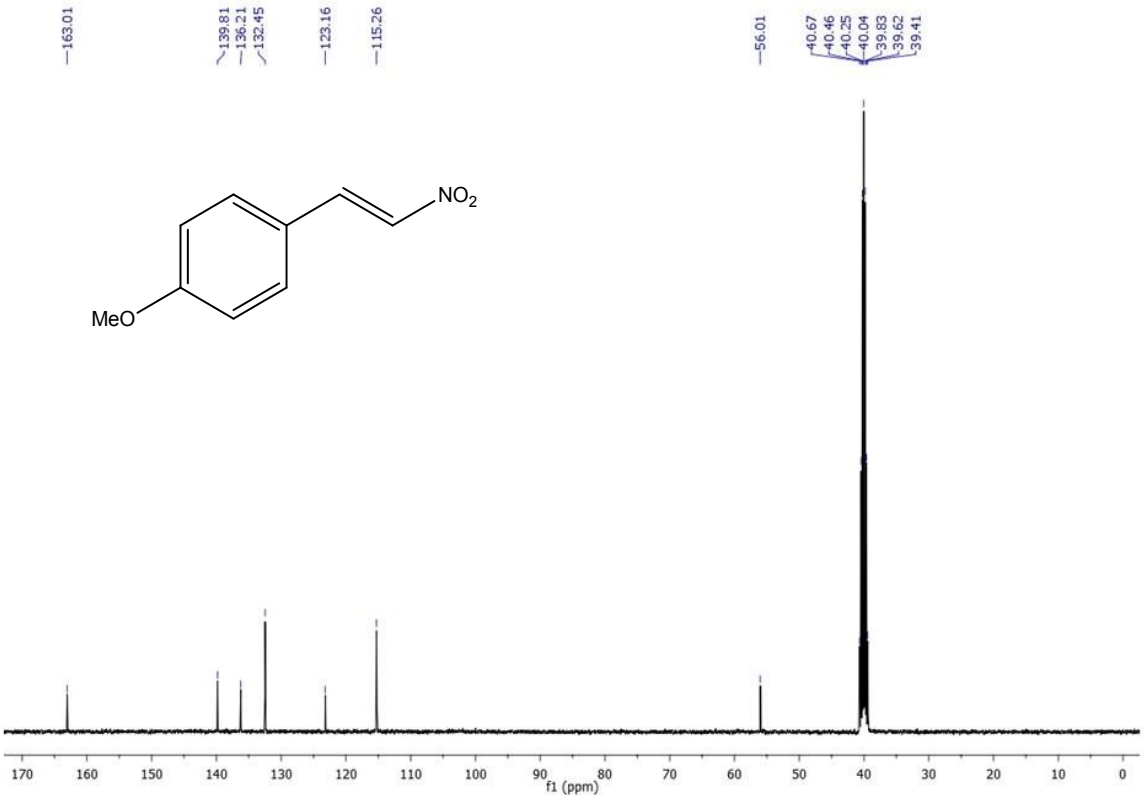
^{1}H NMR and ^{13}C NMR for compound 2b



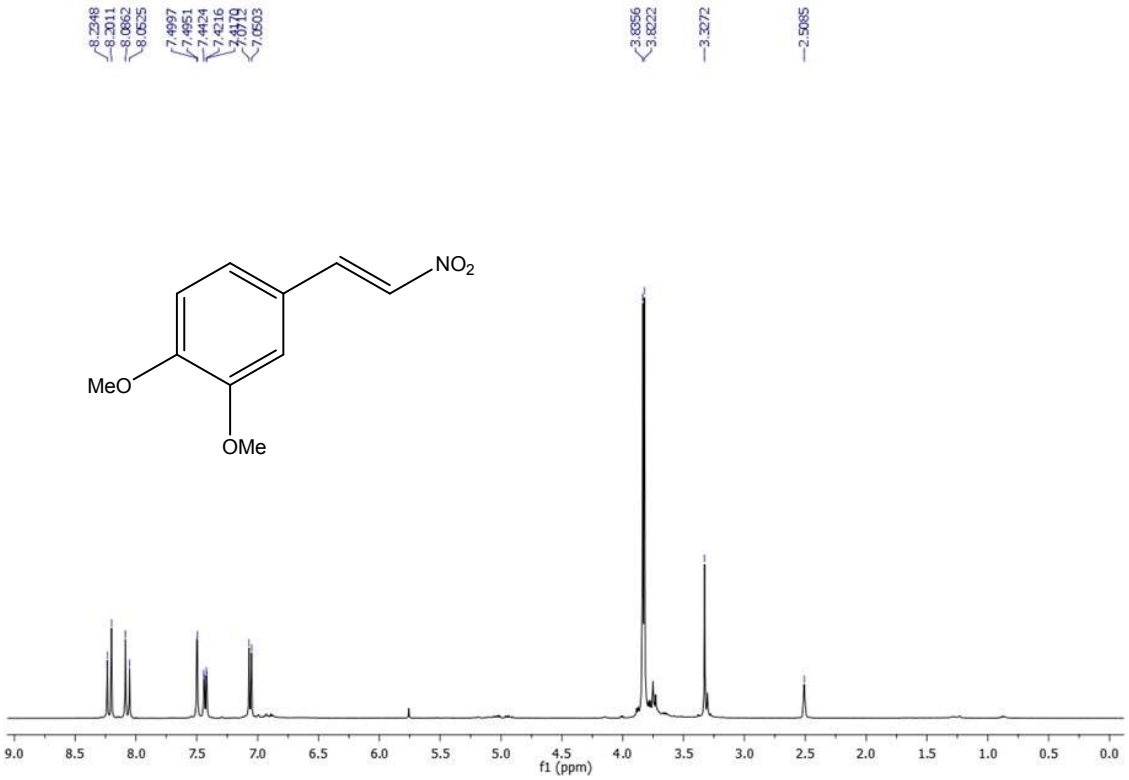


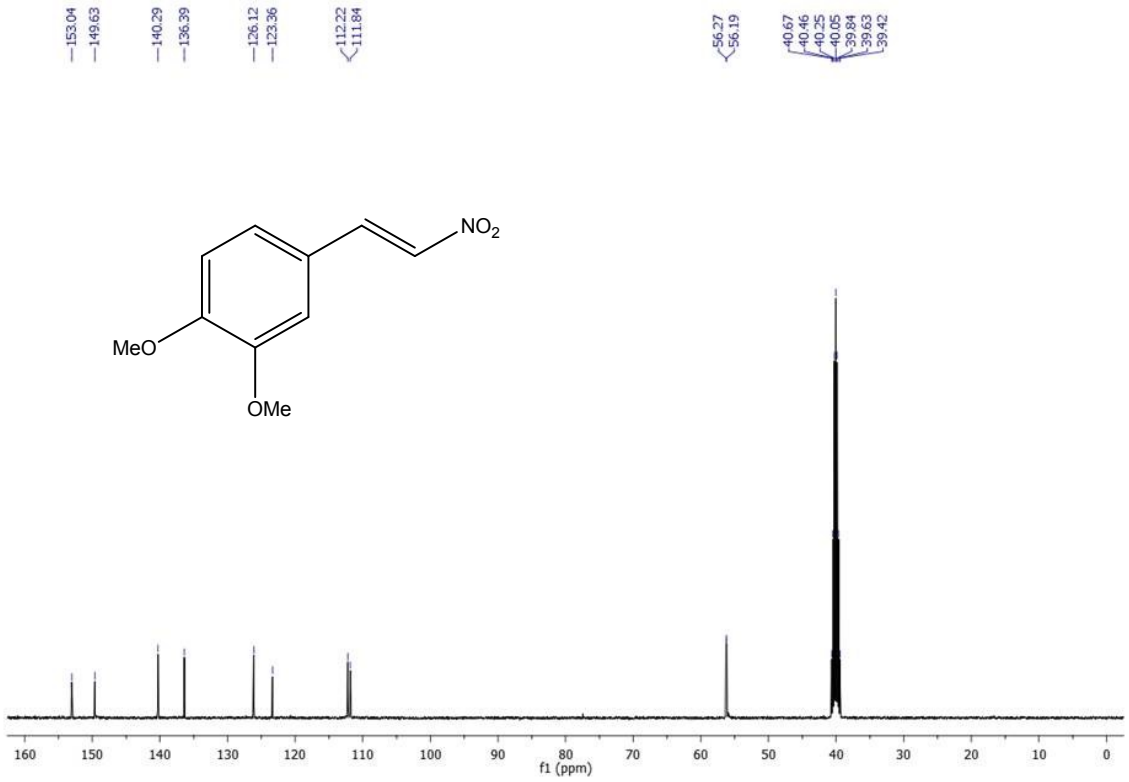
^{1}H NMR and ^{13}C NMR for compound 2c



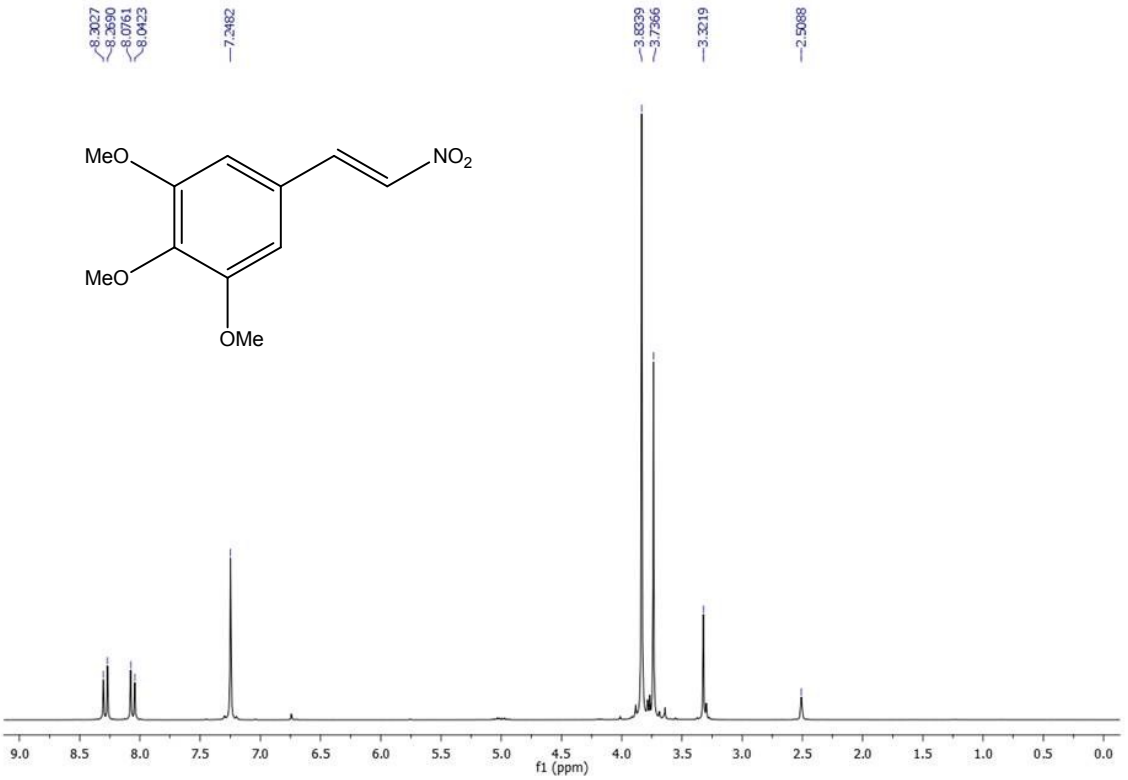


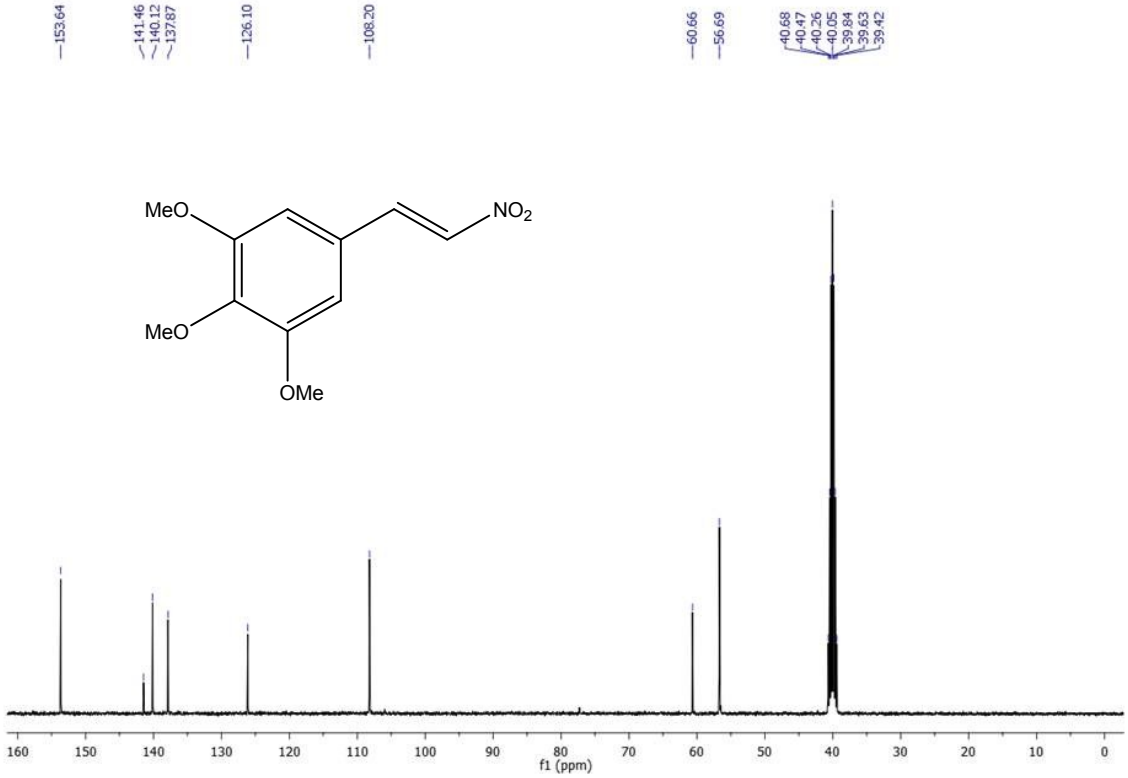
^{1}H NMR and ^{13}C NMR for compound 2d



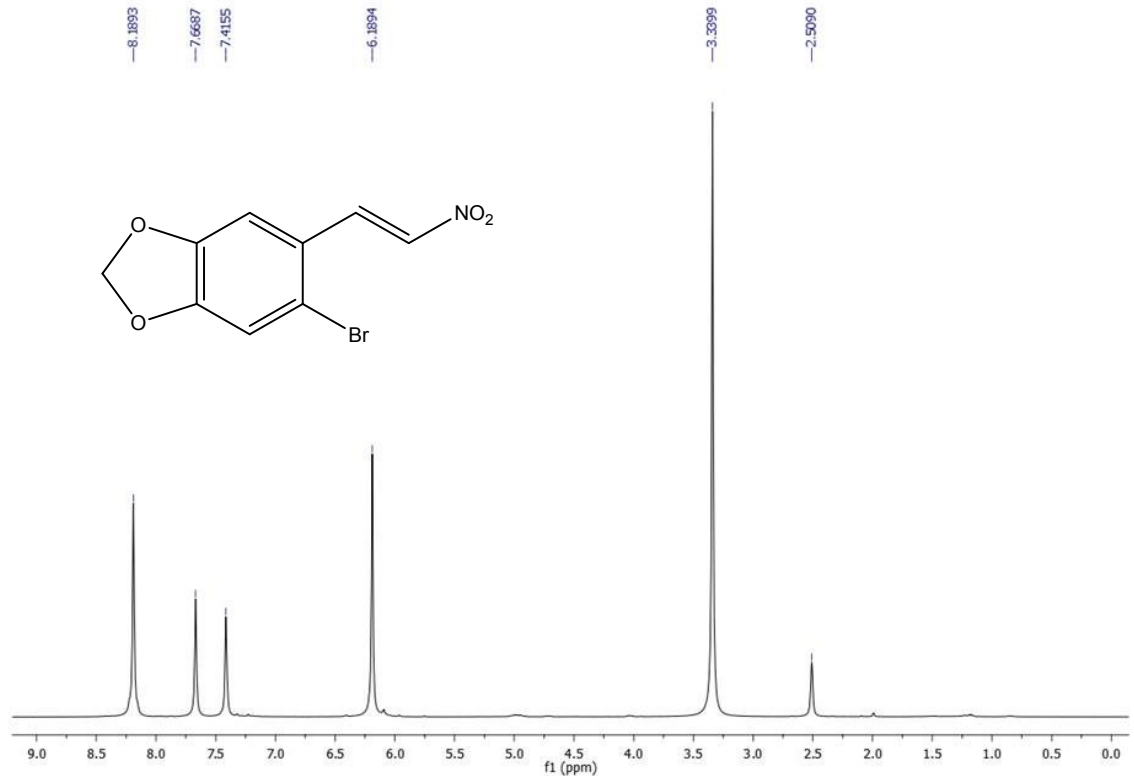


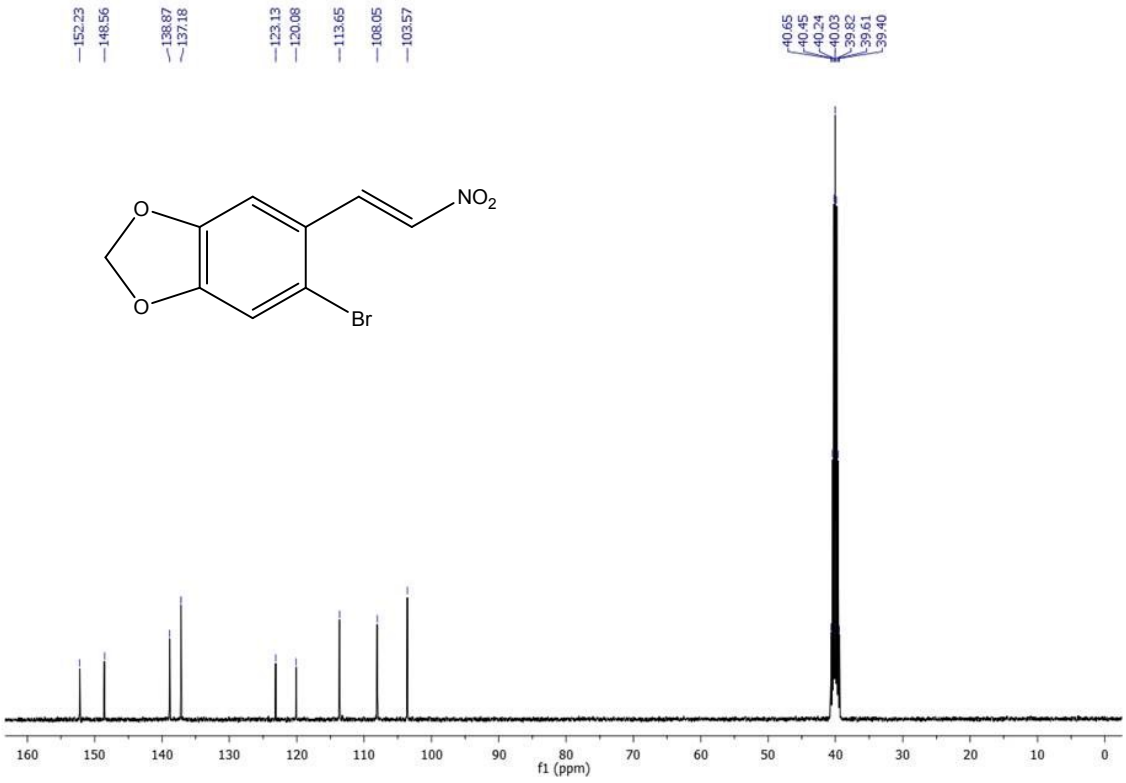
^{1}H NMR and ^{13}C NMR for compound 2e



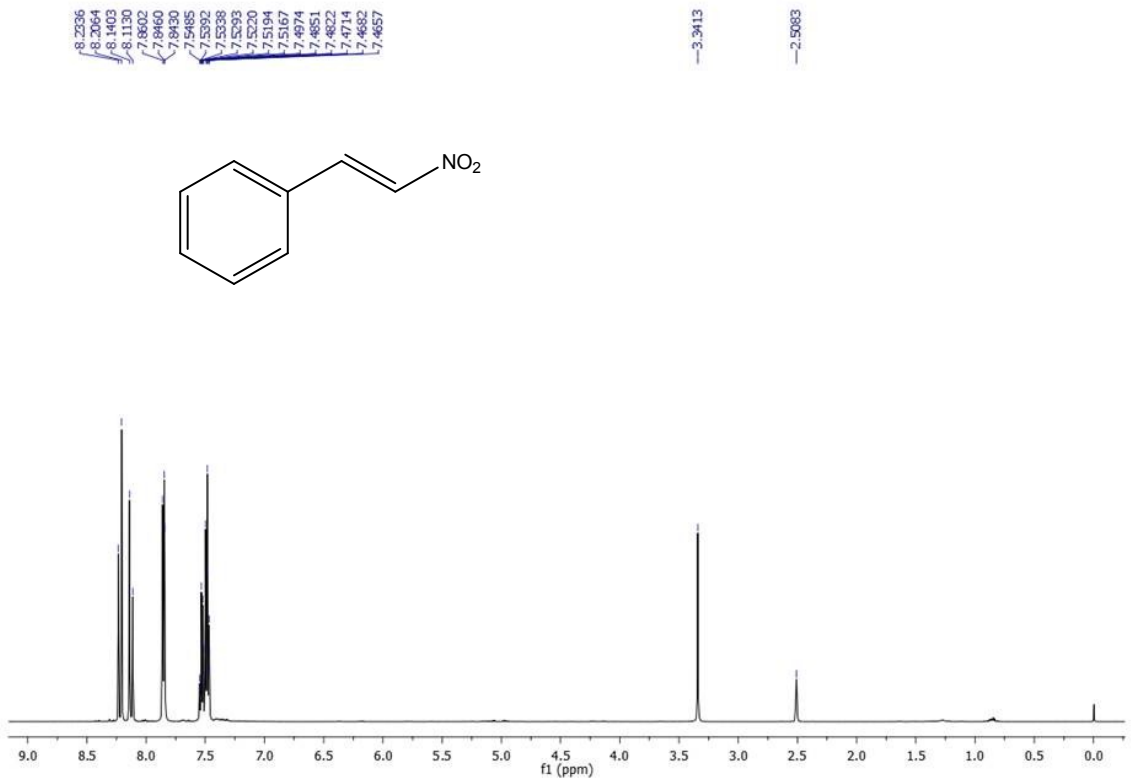


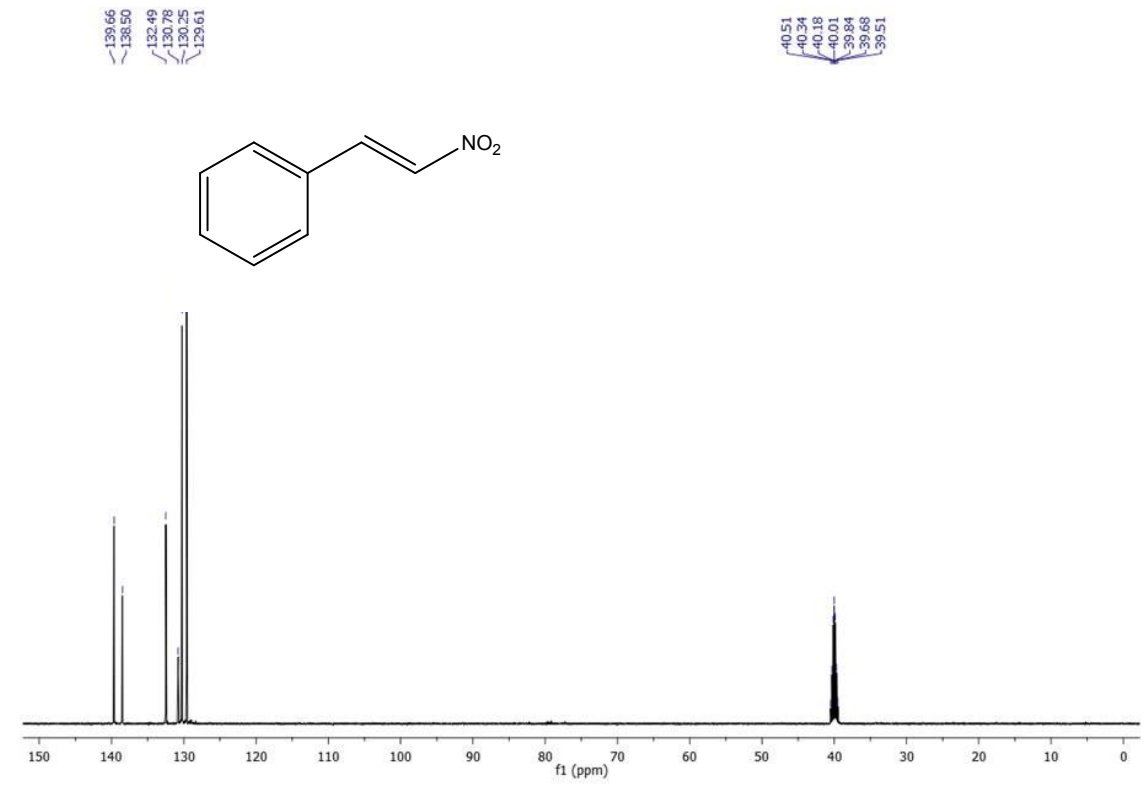
^{1}H NMR and ^{13}C NMR for compound 2f



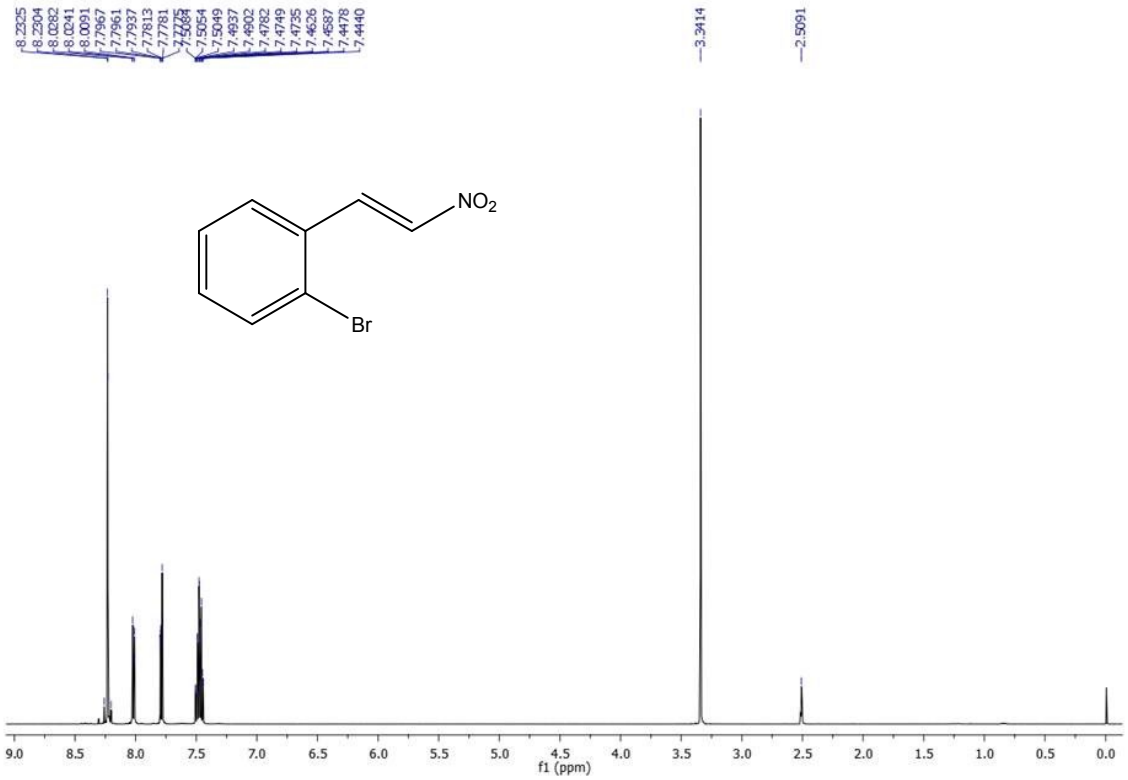


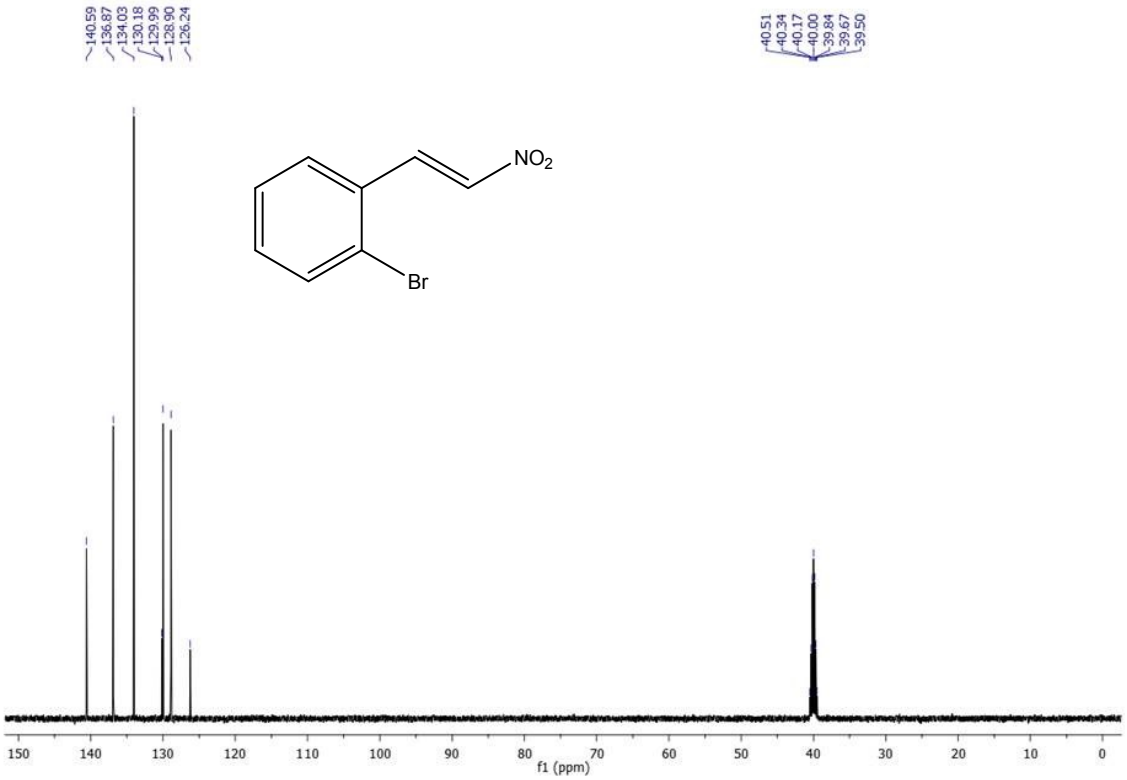
^{1}H NMR and ^{13}C NMR for compound 2g



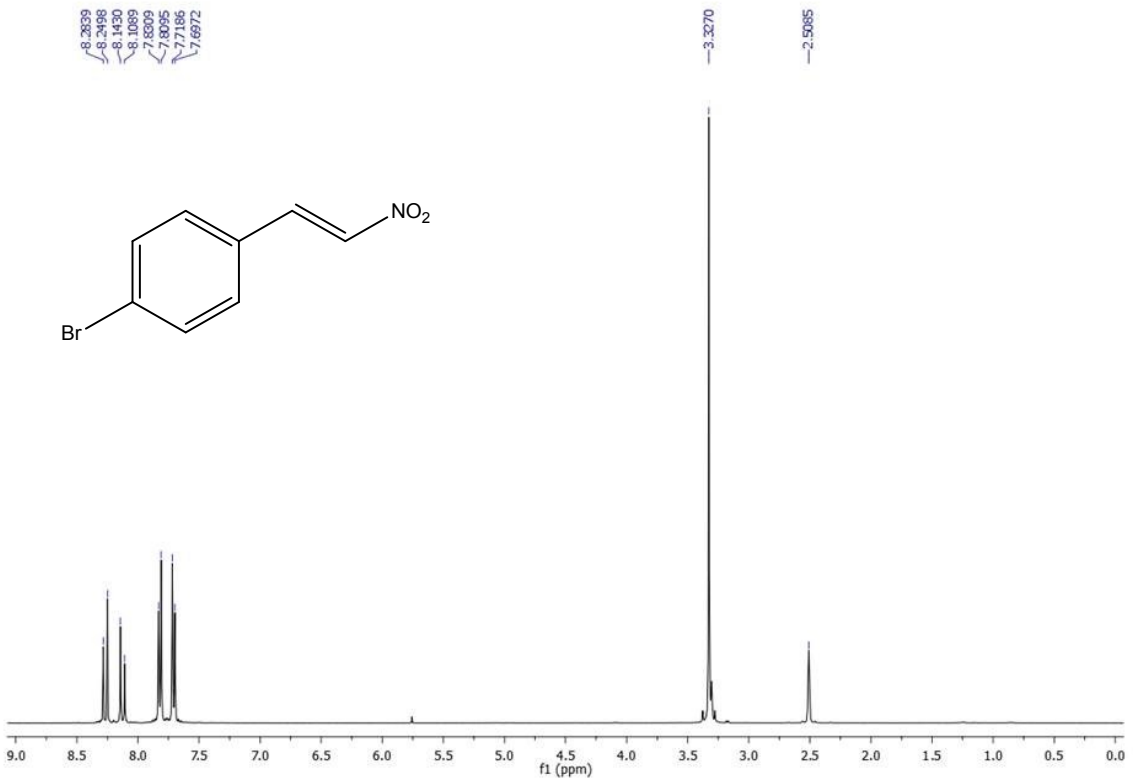


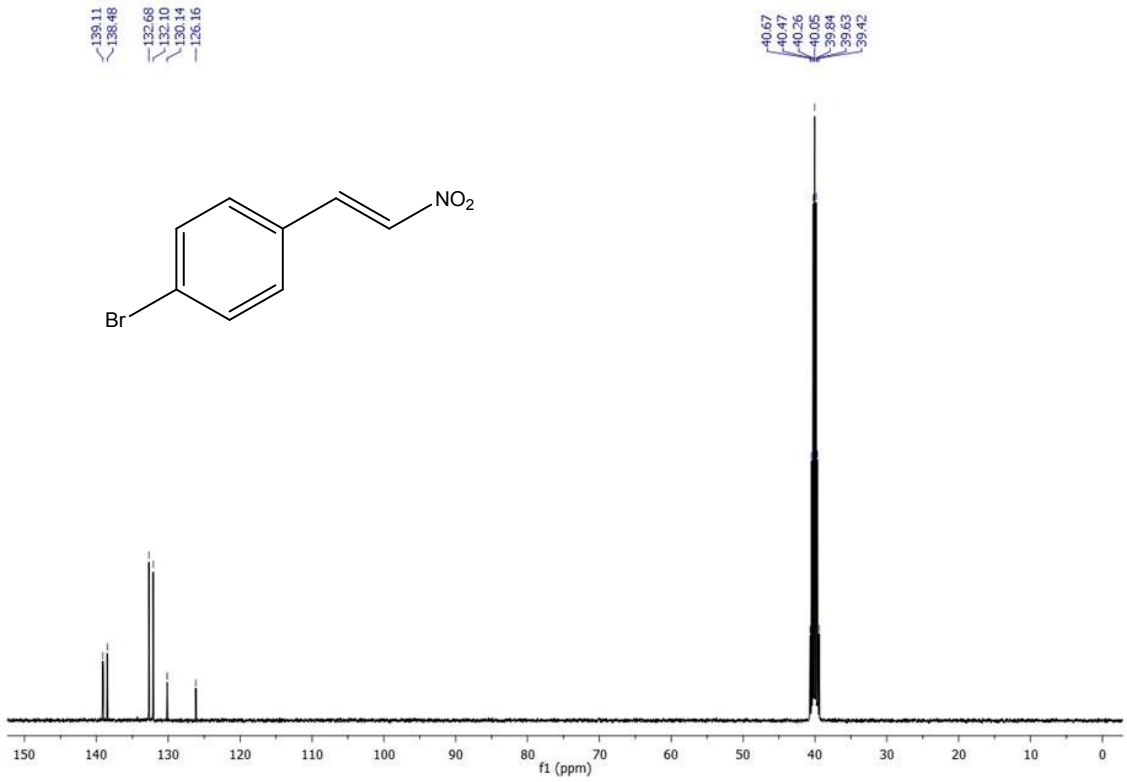
^{1}H NMR and ^{13}C NMR for compound 2h



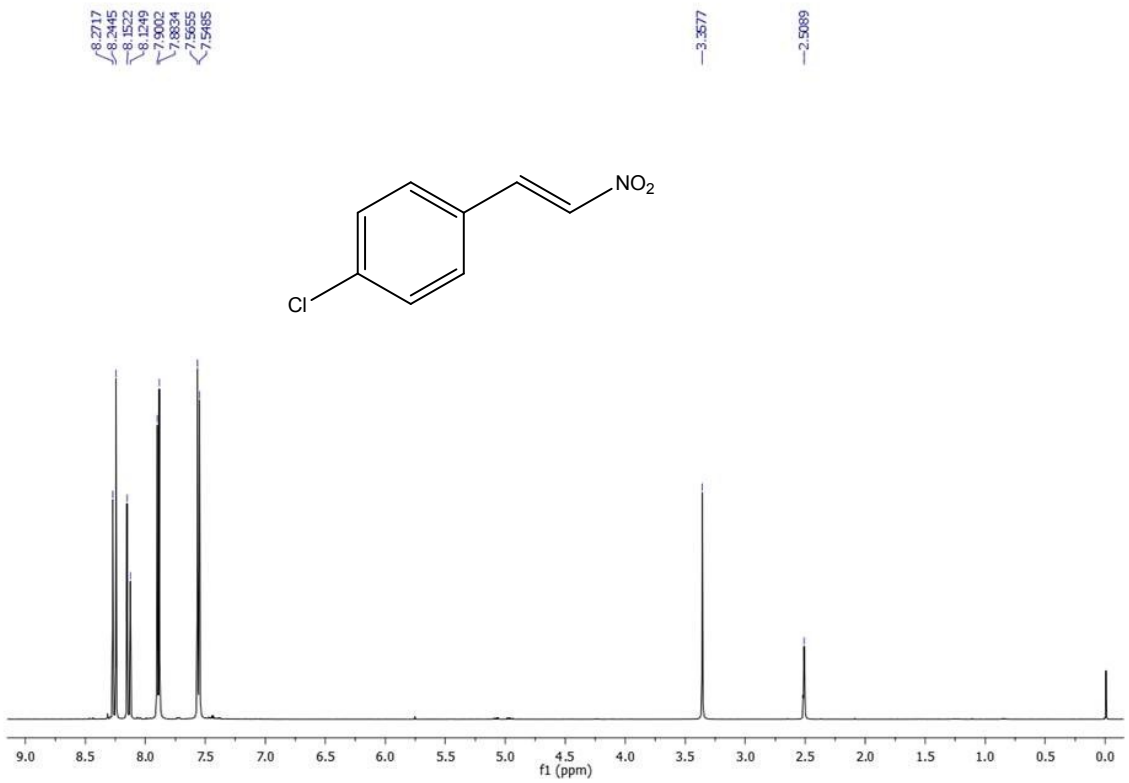


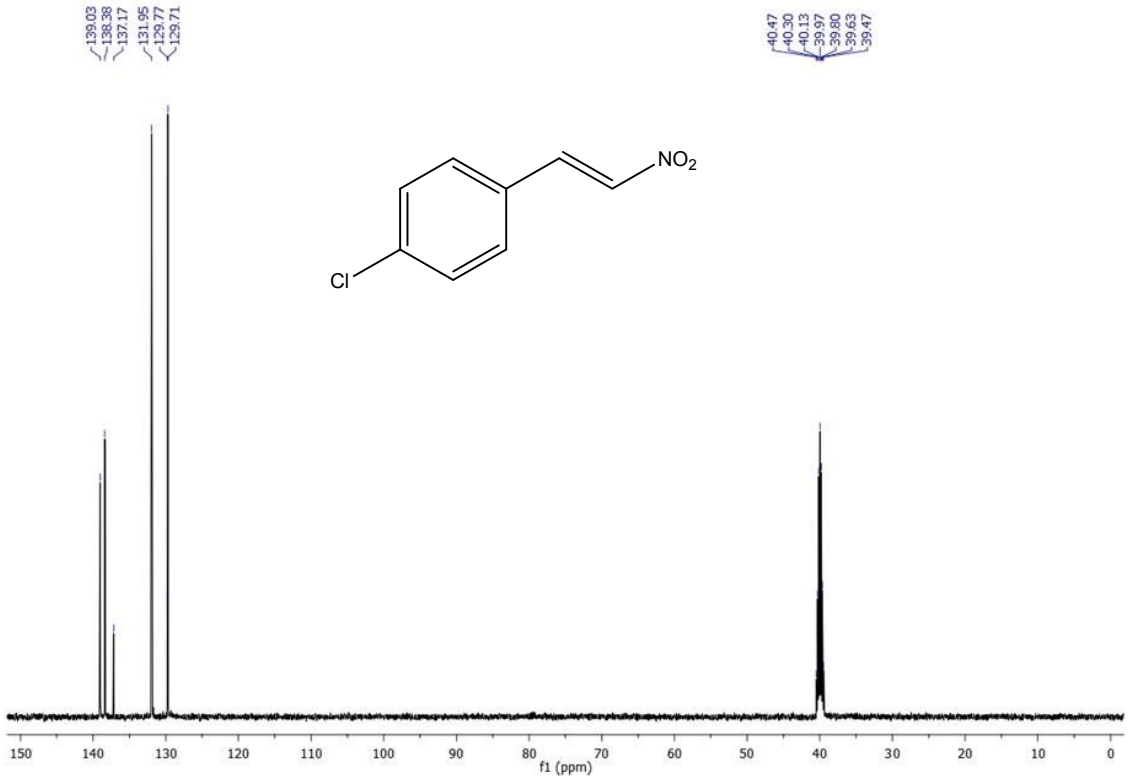
^{1}H NMR and ^{13}C NMR for compound 2i



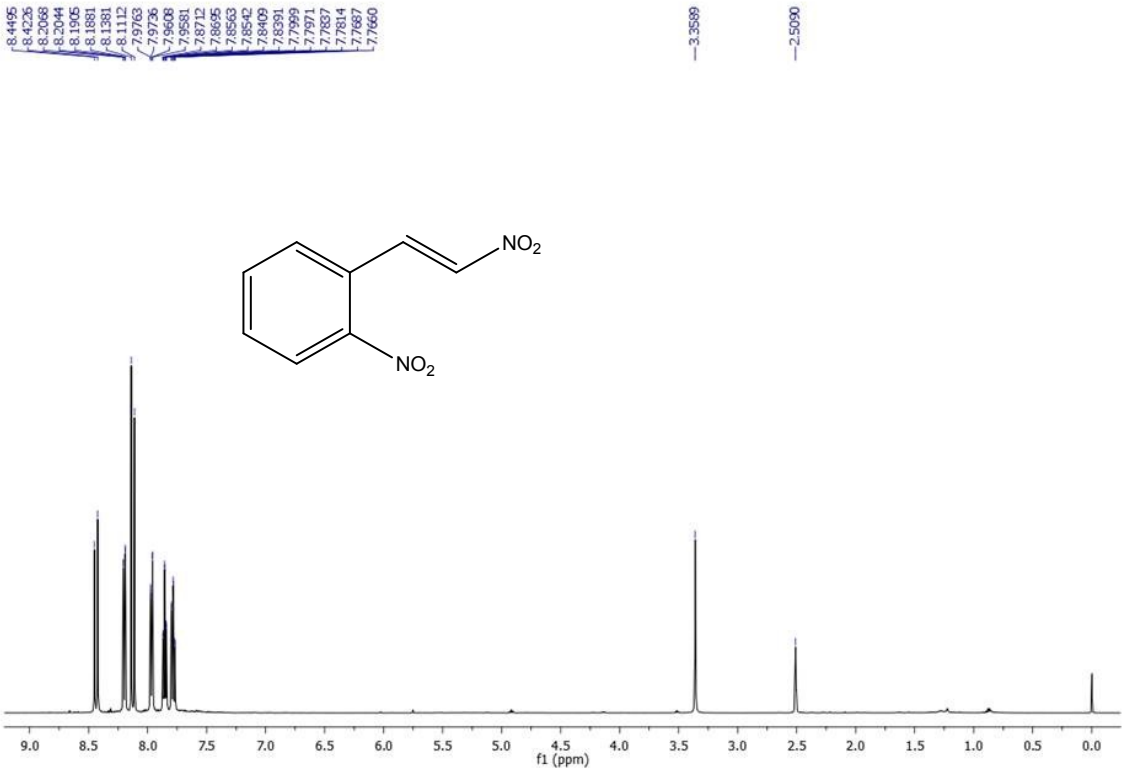


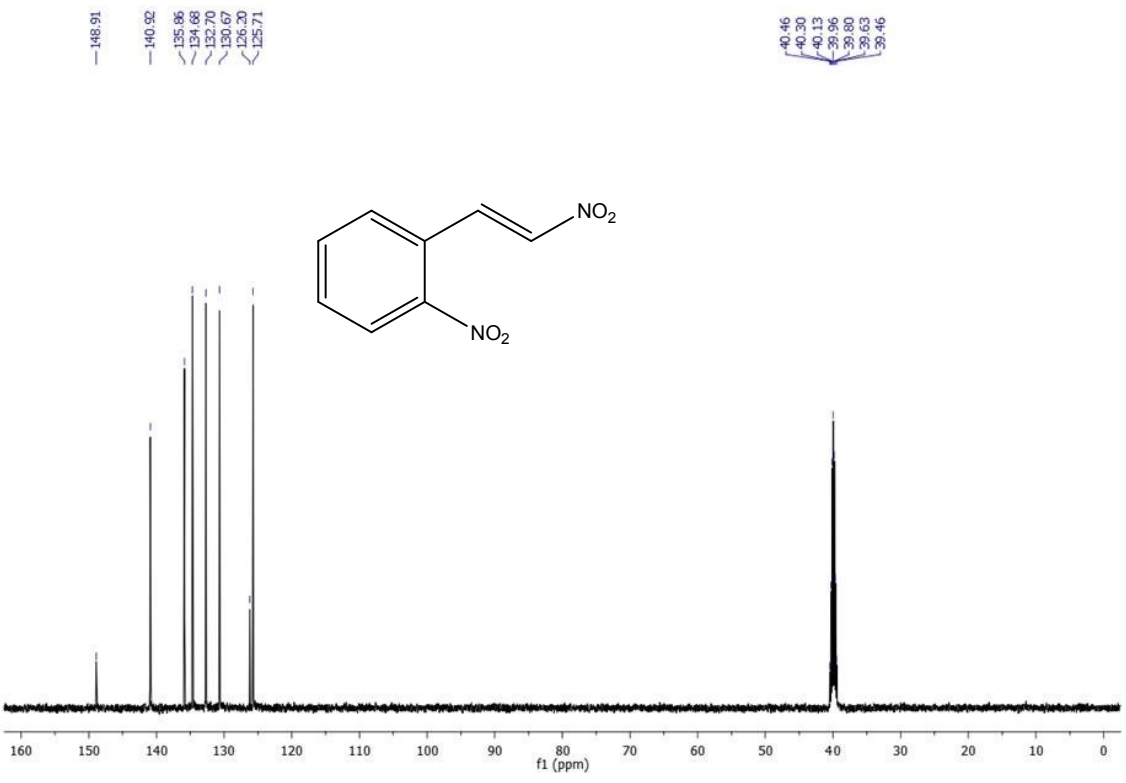
^{1}H NMR and ^{13}C NMR for compound 2j



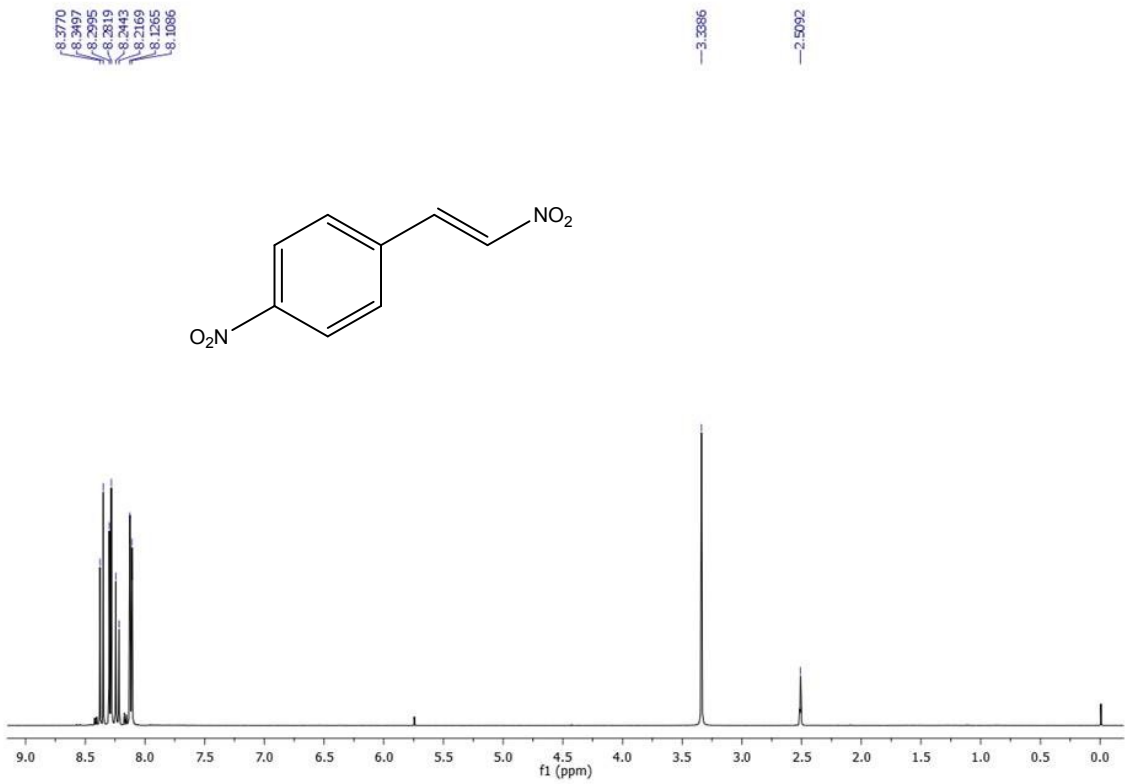


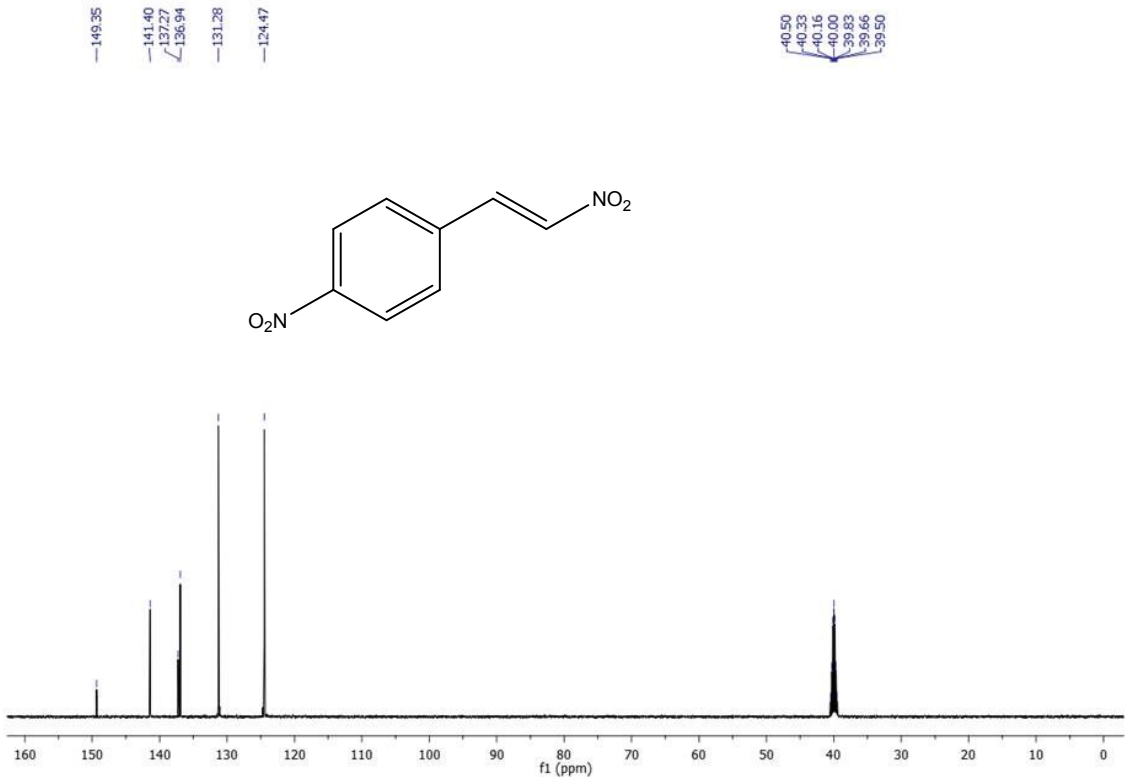
^{1}H NMR and ^{13}C NMR for compound 2k



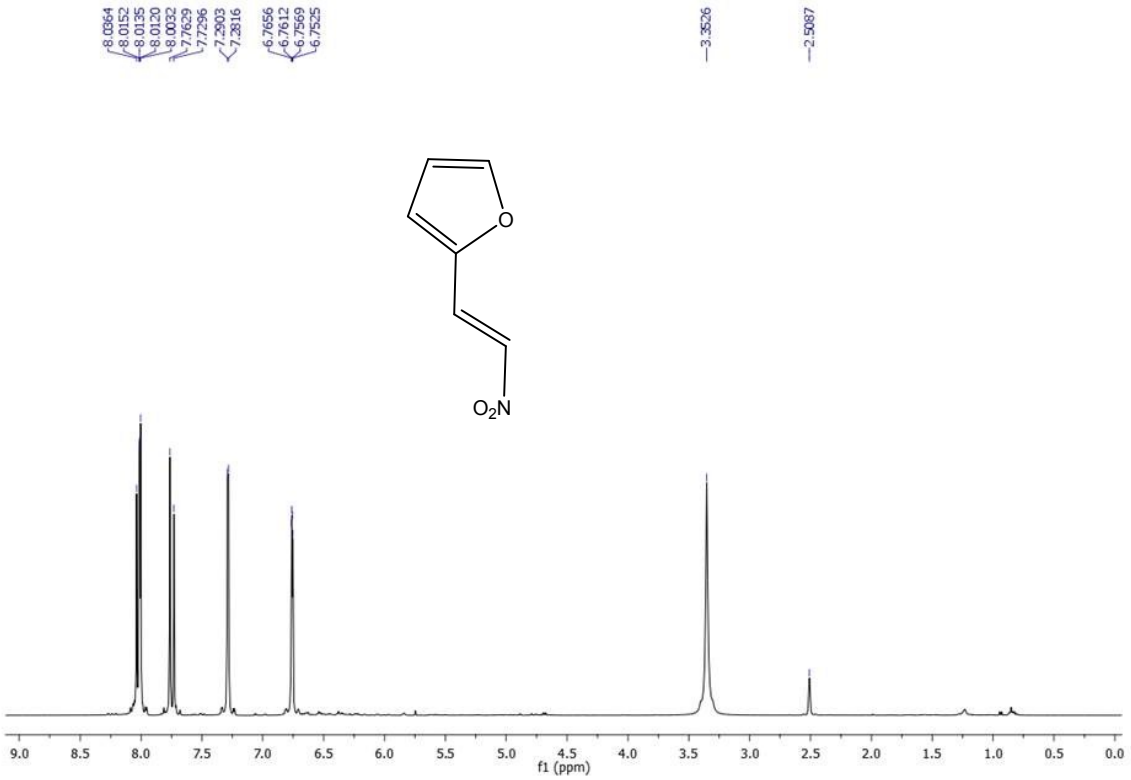


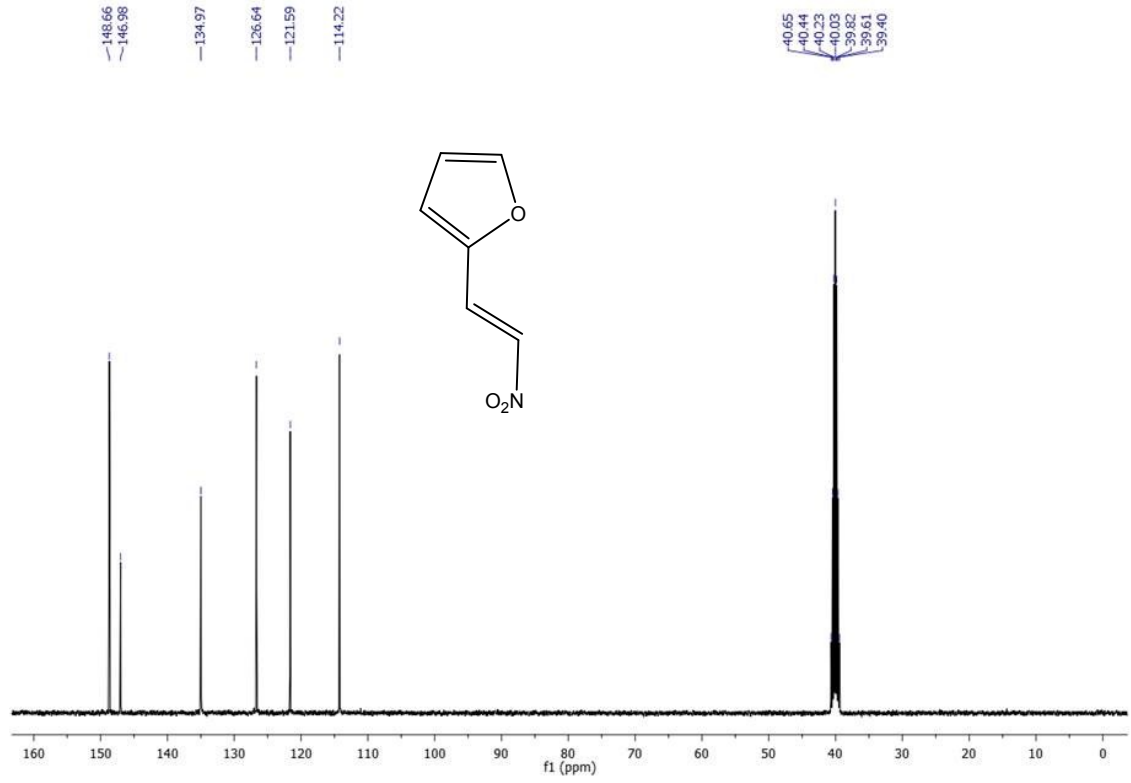
^{1}H NMR and ^{13}C NMR for compound 2l





^{1}H NMR and ^{13}C NMR for compound 2m





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