Electronic Supplementary Material (ESI) for Nanoscale.

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Electro- and photoactivation of silver–iron oxide particles as magnetically recyclable catalysts for cross-coupling: Electronic Supplementary Information

Synthesis of colloidal Fe_3O_4: $FeCl_3$ (648 mg, 4 mmol) and $FeCl_2{\cdot}4\ H_2O$ (398 mg, 2 mmol) were dissolved in 5 mL of deaerated, deionized water, then added dropwise over a period of 15 min to a 20-mL solution of 28% NH_4OH in a glass test tube, immersed in an ultrasonic water bath. A black precipitate was formed immediately upon mixing. The Fe_3O_4 suspension remained immersed in the ultrasonic bath for another 2 minutes, then removed and agitated by vortex mixing to generate a homogeneous dispersion. Colloidal Fe_3O_4 was collected by holding a NeFeB magnet against the side of the reaction tube for up to 15 minutes; the supernatant was then decanted, and the magnetic Fe_3O_4 was redispersed in deionized water. This process was repeated several times to remove residual salts. The mass of magnetically active Fe_3O_4 could be obtained by drying the precipitated colloids in a tared vial, but were otherwise used as freshly prepared aqueous dispersions at a concentration of 10 mg/mL.

Conditioning of colloidal Fe_3O_4: 5-kDa PEG (40 mg) was dissolved in 2.0 mL of deaerated, deionized water and allowed to sit for 10 min, then treated with a 2.0-mL dispersion of colloidal Fe_3O_4 (20 mg) and agitated with a vortex mixer before resting for 1 h. A 1.0-mL aliquot of PEG-coated Fe_3O_4 (5 mg) was then mixed with 20 mL of aqueous L-histidine (1 mg/mL) adjusted to pH 5–6, and allowed to sit for another hour before washing twice with water.

Additional materials characterization of colloidal SIO

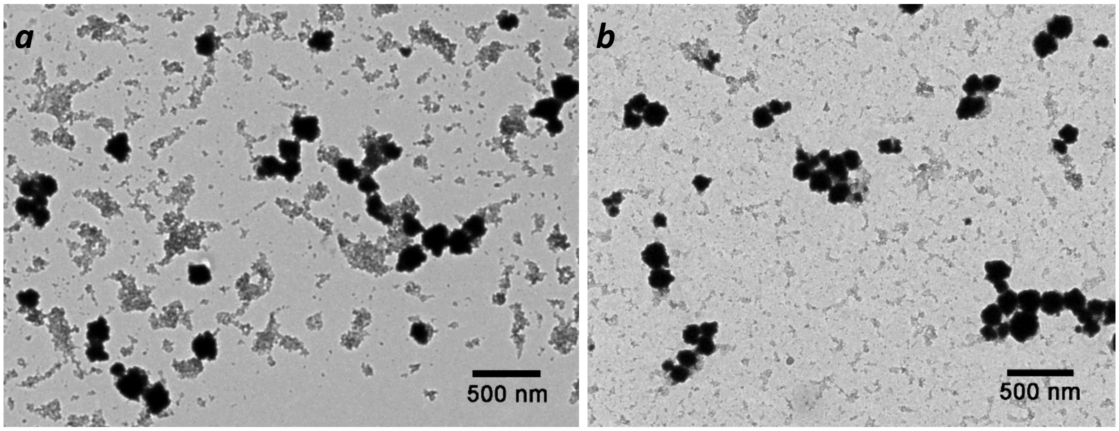
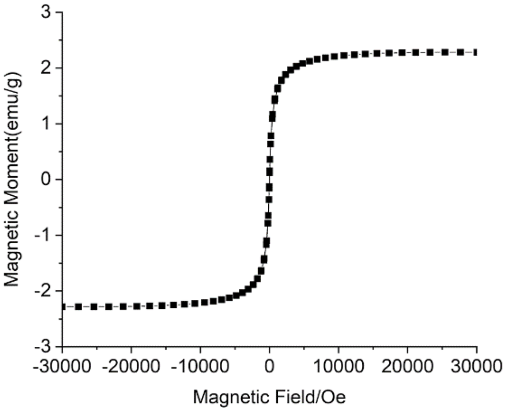


Figure S1. (a) Brightfield TEM image of freshly prepared colloidal SIO (with residual iron oxide), collected by magnetic precipitation. (b) Colloidal SIO after five uses (with residual iron oxide), collected by magnetic precipitation.



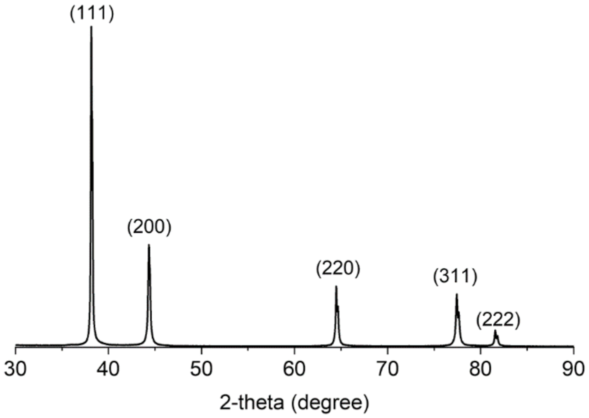


Figure S2 (*left*). Magnetization curve of SIO (powder sample, acquired at 298 K); M_S = 2.3 emu/g.
Figure S3 (*right*). Powder X-ray diffraction of freshly prepared SIO, with peaks assigned to fcc-Ag (JCPDS 04-0783).

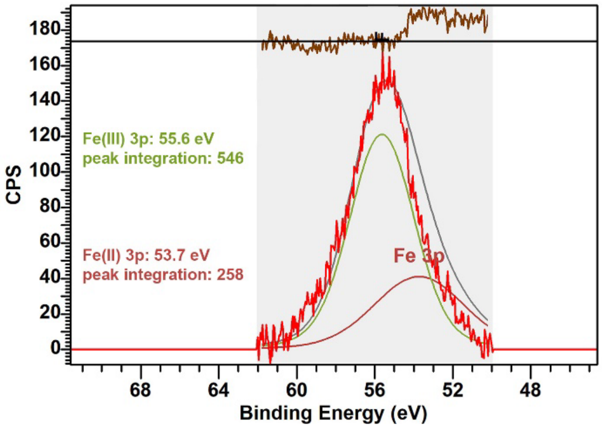


Figure S4. X-ray photoelectron spectroscopy (XPS) analysis of the Fe 3p region of freshly prepared SIO; peak deconvolution reveals a Fe(III):Fe(II) ratio of 2.1, characteristic of Fe_3O_4.[1]

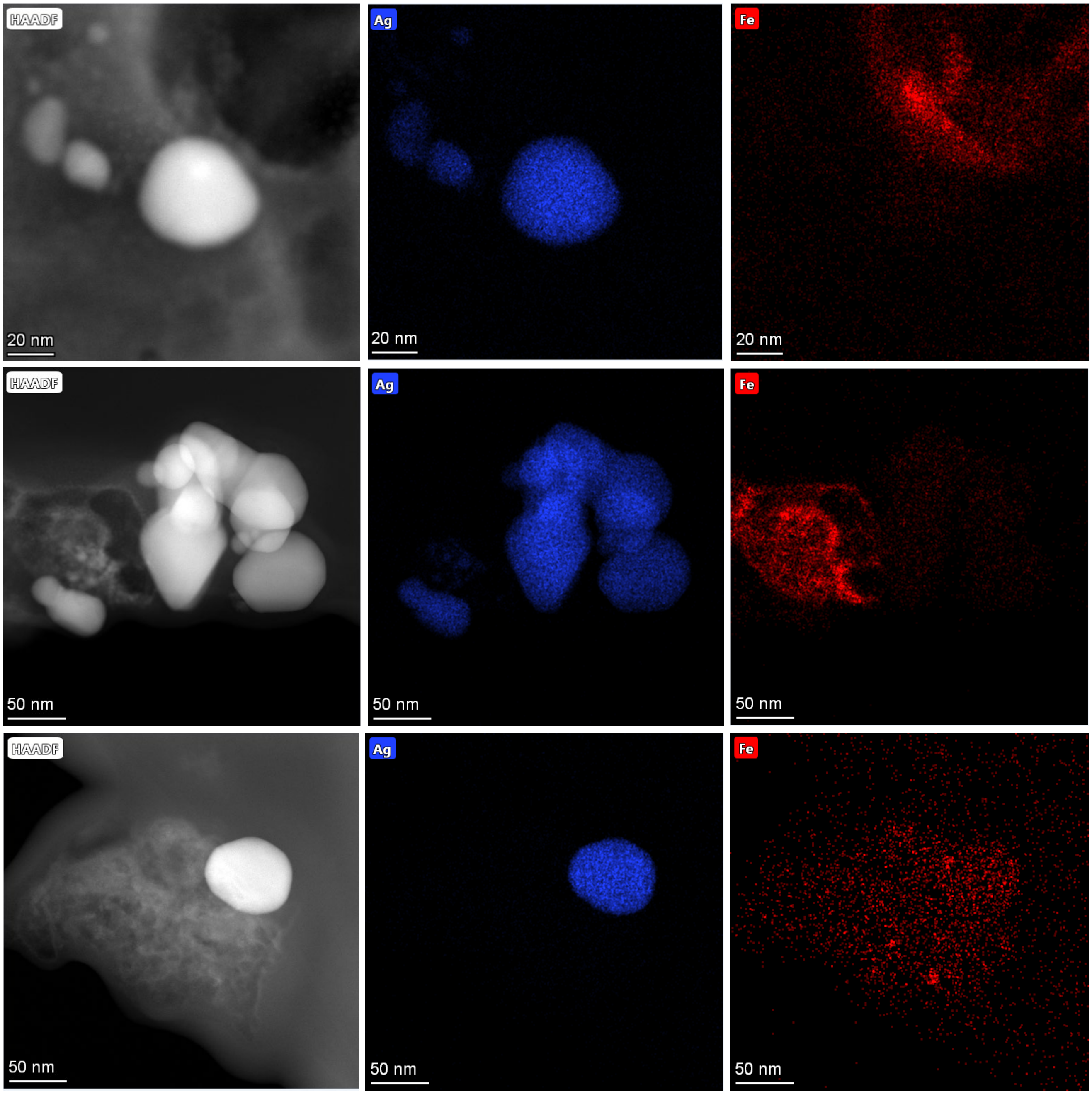


Figure S5. HAADF and EDS images with elemental mapping for Ag (Lα_1: 2.9846 keV), Fe (Kα_1: 6.4031 keV, Kα_2: 6.3895 keV) for non-magnetic residues recovered by centrifugation after a SIO-catalyzed cross-coupling reaction. The interface between Ag nanoparticles and residual iron oxide was greatly reduced in the non-magnetic solute, and no catalytic activity was observed.

Table S1. Reaction cycling of SIO-mediated cross coupling with constant light irradiation

Sample	**SIO mass (mg)**	**Yield of 2a (%)**[b]
Colloidal SIO (prior to reaction)	6.3	--
Rxn cycle 1[a]	--	69
Rxn cycle 2	--	69
Rxn cycle 3	--	64
Rxn cycle 4	--	62
Rxn cycle 5	3.9	59

[a] Standard condition: SIO (6.3 mg), **1a** (2.5 mmol), furan (25 mmol), DMSO (1 mL), 300-W sunlamp, 1 h. [b] Based on NMR (±5%).

Table S2. AAS analysis of SIO and supernatant (no added base)

Sample	**Ag, diluted (ppm)**	**Fe, diluted (ppm)**	**Ag, total mass (mg)**	**Fe, total mass (mg)**
Colloidal SIO, freshly prepared	10.6[a]	3.1[b]	12.7	0.50
Colloidal SIO, after 5X recycling	2.3[a]	3.7[c]	2.80	0.22
Rxn cycle 1: Supernatant	4.9[d]	7.0[e]	1.47	0.040
Rxn cycle 1: Non-magnetic ppt.	--	1.1[f]	--	0.004
Rxn cycle 2: Supernatant	5.0[d]	5.9[e]	1.51	0.035
Rxn cycle 2: Non-magnetic ppt.	--	1.8[f]	--	0.007
Rxn cycle 3: Supernatant	5.6[d]	6.7[e]	1.69	0.040
Rxn cycle 3: Non-magnetic ppt.	--	0.8[f]	--	0.003
Rxn cycle 4: Supernatant	5.5[d]	5.9[e]	1.65	0.035
Rxn cycle 4: Non-magnetic ppt.	--	1.0[f]	--	0.004
Rxn cycle 5: Supernatant	4.3[d]	4.0[e]	1.28	0.024
Rxn cycle 5: Non-magnetic ppt.	--	1.4[f]	--	0.006

[a] 1200X dilution. [b] 160X dilution. [c] 60X dilution. [d] 300X dilution. [e] 6X dilution. [f] Resuspended in 4 mL water.

Table S3. AAS analysis of SIO and supernatant (with added Et_3N)

Sample	**Ag, diluted (ppm)**	**Fe, diluted (ppm)**	**Ag, total mass (mg)**	**Fe, total mass (mg)**
Colloidal SIO, freshly prepared	7.5[a]	3.6[b]	9.05	0.60
Colloidal SIO, after 5X recycling	7.4[a]	0.9[c]	8.90	0.06

[a] 1200X dilution. [b] 160X dilution. [c] 60X dilution.

Table S4. Reaction yields and SIO mass recovery in 90% aqueous DMSO

Sample	**SIO mass (mg)**	**Yield of 2a (%)**[b]
Colloidal SIO (prior to reaction)	15	--
Rxn cycle 1[a]	--	64
Rxn cycle 2	--	59
Rxn cycle 3	--	61
Rxn cycle 4	--	62
Rxn cycle 5	9.0	56

[a] Standard condition: SIO (15 mg), **1a** (2.5 mmol, 1 equiv), furan (25 mmol), DMSO (1 mL), 1.5 h, RT [b] Based on NMR (±5%).

Synthesis of diazonium salts: In a typical reaction, substituted aniline (5 mmol) was dissolved in ethanol (4 mL) in a 25-mL round-bottomed flask, followed by the careful addition of 48% aqueous HBF_4 (1.77 mL). The reaction mixture was cooled to 0 °C and treated dropwise with 5 M aqueous $NaNO_2$ (5 mmol), then allowed to stir for 2 h at 0 °C. The reaction mixture was diluted with ethyl ether (20 mL) to precipitate the product, which was filtered and washed with fresh ether, redissolved in minimal acetone, then precipitated again by ether. This procedure was repeated 3–5 times to ensure a clean diazonium salt, which was dried overnight under reduced pressure.

Chemical characterization data

^{1}H NMR, ^{13}C NMR, and APCI-MS data are provided for all reaction products. Previously synthesized products have been checked against published data; novel products include characterization by high-resolution APCI-MS.

2-(2-Nitrophenyl)furan (**2a**):[2] Obtained in 70% isolated yield as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (1H, dd, J = 1.4, 7.9 Hz). 7.66 (1H, dd, J = 1.3, 8.1 Hz) 7.55 (1H, dt, J = 1.3, 7.7 Hz), 7.50 (1H, dd, J = 0.8, 1.8 Hz), 7.39 (1H, dt, J = 1.4, 7.7 Hz), 6.66 (1H, dd, J = 0.8, 3.4 Hz), 6.49 (1H, dd. J = 1.8, 3.4 Hz).^{13}C NMR (100 MHz, $CDCl_3$): δ 148.30, 147.41, 143.70, 131.79, 128.77, 128.19, 124.00, 123.73, 111.79, 109.61. APCI-MS: *m/z* 190.00 $[M+H]^+$.

2-(4-Chloro-2-nitrophenyl)furan (2b):[3] Obtained in 72% NMR yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (1H, d, J = 8.5 Hz), 7.66 (1H, d, J = 2.2 Hz), 7.53 (1H, dd, J = 2.0, 8.5 Hz), 7.51 (1H, dd, J = 0.7, 1.8 Hz), 6.66 (1H, dd, J = 0.7, 3.5 Hz), 6.49 (1H, dd, J = 1.8, 3.5 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.37, 147.25, 144.02, 133.73, 131.98, 129.72, 123.95, 122.50, 112.00, 110.19. APCI-MS: *m/z* 224.01 and 226.01 $[M+H]^+$ for $C_{10}H_7(^{35}Cl)NO_3$ and $C_{10}H_7(^{37}Cl)NO_3$, respectively.

2-(2-Chloro-4-nitrophenyl)furan (2c): Obtained in 65% NMR yield, and 60% isolated yield as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 (1H, d, J = 2.4 Hz), 8.12 (1H, dd, J = 2.3, 8.9 Hz), 8.02 (1H, d, J = 8.8 Hz), 7.60 (1H, dd, J = 0.7, 1.8 Hz), 7.40 (1H, dd, J = 0.7, 3.6 Hz), 6.60 (1H, dd, J = 1.8, 3.6 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.36, 145.95, 144.05, 134.57, 129.94, 127.70, 126.22, 121.95, 114.55, 112.55. APCI-HRMS: *m/z* calcd $[M+H]^+$ 226.0084 for $C_{10}H_7(^{37}Cl)NO_3$; found *m/z* 226.0076.

2-(2,6-Dibromophenyl)furan (2d): Obtained in 72% NMR yield; isolated by silica gel chromatography as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (2H, d, J = 8.0 Hz), 7.58 (1H, dd, J = 0.8, 1.9 Hz)

7.10 (1H, t, J = 8.1 Hz), 6.55 (1H, dd, J =1.8, 3.3 Hz), 6.51 (1H, dd, J = 0.8, 3.3 Hz).^{13}C NMR (100 MHz, $CDCl_3$) δ 150.96, 142.26, 133.62, 131.83, 131.19, 126.15, 110.80, 110.47. APCI-HRMS: *m/z* calcd $[M+H]^+$ 304.8824 for $C_{10}H_7(^{81}Br)_2O$; found 304.8817.

2-(4-Iodo-2-nitrophenyl)furan (2e):[4] obtained in 74% NMR yield**.** ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (1H, d, J = 1.8 Hz), 7.87 (1H, dd, J = 1.8, 8.3 Hz), 7.51 (1H, dd, J = 0.7, 1.8 Hz), 7.44 (1H, d, J = 8.3 Hz), 6.68 (1H, dd, J = 0.7, 3.5 Hz), 6.49 (1H, dd, J = 1.8, 3.5 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.33, 147.27, 144.09, 140.71, 132.25, 129.74, 123.35, 112.05, 110.35, 91.47. APCI-MS: *m/z* 316.00 $[M+H]^+$.

2-(4-Nitrophenyl)furan (**2f**):[2] Obtained in 60% NMR yield, isolated by silica gel chromatography as a yellow solid in 53% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.22 (2H, d, J = 9.0 Hz), 7.76 (2H, d, J = 9.1 Hz), 7.56 (1H, dd, 0.7, 1.8 Hz), 6.87 (1H, dd, J = 0.7, 3.5 Hz), 6.54 (1H, dd, J = 1.8, 3.5 Hz).^{13}C NMR (100 MHz, $CDCl_3$) δ 151.65, 146.32, 14411, 136.36, 124.23, 123.85, 112.39, 108.95. APCI-MS: *m/z* 190.08 $[M+H]^+$.

2-(4-Chlorophenyl)furan (2g):[2] Obtained in 47% isolated yield as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (2H, d, J = 8.7 Hz), 7.47 (1H, dd, J = 0.8, 1.8 Hz), 7.35 (2H, d, J = 8.7 Hz), 6.64 (1H, dd, J = 0.8, 3.4 Hz), 6.47 (1H, dd, J = 1.8, 3.4 Hz).^{13}C NMR (100 MHz, $CDCl_3$) δ 152.92, 142.30, 132.93, 129.28, 128.94, 124.32, 111.75, 105.39. APCI-MS: *m/z* 181.08 $[M+H]^+$ for $C_{10}H_8(^{37}Cl)O$.

2-(4-Bromophenyl)furan (2h):[2] Obtained in 56% NMR yield, isolated by silica gel chromatography as a white solid in 42% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47–7.55 (4H, m), 7.48 (1H, dd, J = 0.8, 1.8 Hz), 6.65 (1H, dd, J = 0.8, 3.4 Hz), 6.48 (1H ,dd, J = 1.8, 3.4 Hz).^{13}C NMR (100 MHz, $CDCl_3$) δ 152.85, 142.29, 131.71, 129.70, 125.20, 120.97, 111.71, 105.45. APCI-MS: *m/z* 226.08 $[M+H]^+$ $C_{10}H_8(^{81}Br)O$.

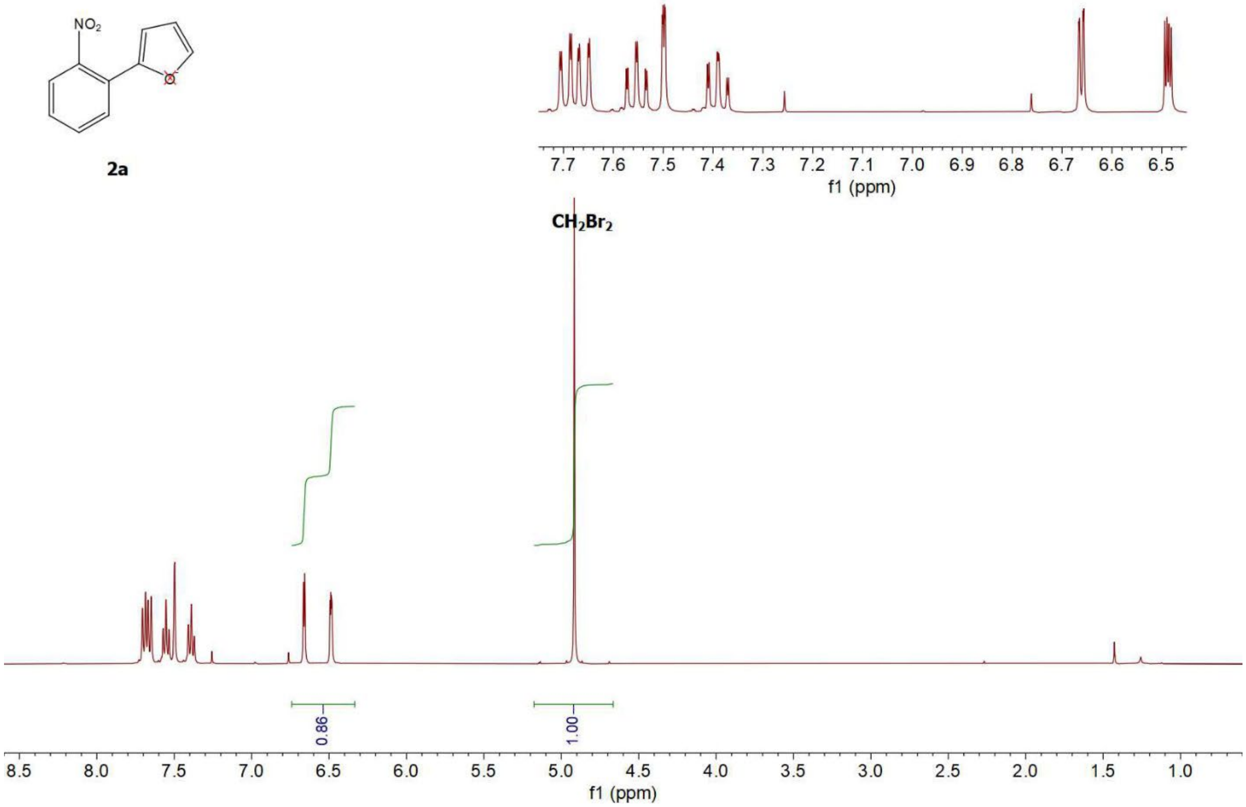
Ethyl 2-(furan-2-yl)benzoate (2i): Obtained in 42% NMR yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (1H, d, J = 7.6 Hz), 7.59 (1H, d, J = 7.8 Hz), 7.44–7.52 (2H, m), 7.35 (1H, d, J = 6.9 Hz), 6.56 (1H, d, J = 3.4 Hz), 6.47 (1H, dd, J = 1.8, 3.6 Hz), 4.29 (2H, q, J = 7.1 Hz), 1.25 (3H, t, J = 7.2 Hz).^{13}C NMR (100 MHz, $CDCl_3$) δ 169.10, 152.57, 142.51, 130.74, 130.50, 129.74, 129.13, 128.16, 127.64, 111.45, 107.82, 61.27, 14.08. APCI-MS: *m/z* 217.08 $[M+H]^+$.

2-Nitrophenylthiophene (3a):[5] Obtained with 3-nitrophenylthiophene as a 93:7 mixture in 57% combined yield, isolated as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (1H, dd, J = 1.2, 8.1 Hz), 7.57 (1H, dd, J = 1.3, 6.3 Hz), 7.56 (1H, d, 2.6 Hz) 7.46 (1H, ddd, J = 2.6, 6.3, 8.0 Hz), 7.41 (1H, dd, J = 1.5, 4.9 Hz), 7.11–7.05 (2H, m). ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.42, 137.08, 131.84, 128.55, 128.31, 127.71, 127.11, 127.09, 123.79. APCI-MS: *m/z* 206.00 $[M+H]^+$.

2-(2-Nitrophenyl)benzofuran (4a):[6] Obtained in 35% isolated yield as a yellow oil. ^{1}H NMR (800 MHz, $CDCl_3$) δ 7.87 (1H, d, J = 7.8 Hz), 7.78 (1H, d, J = 8.1 Hz), 7.65 (1H, t, 7.5 Hz), 7.62 (1H, d, 7.7 Hz), 7.51 (2H, m), 7.34 (1H, t, J = 7.8 Hz), 7.27 (1H, t, J = 7.3 Hz), 7.02 (1H, s). ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.20, 150.43, 148.21, 132.03, 129.94, 129.32, 128.47, 125.33, 124.21, 124.04, 123.26, 121.49, 111.43, 106.03. ACPI-MS 240.00 $[M+H]^+$.

***N*-Butoxycarbonyl-2-(2-nitrophenyl)pyrrole (5a):**[4] Obtained in 67% isolated yield as a red oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.11 (1H, d, J = 8.0 Hz), 7.62 (1H, t, J = 7.1 Hz) 7.50 (1H, t, J = 7.1 Hz), 7.46 (1H, d, J =7.5 Hz), 7.11 (1H, dd, J = 1.6, 3.2 Hz), 6.27 (1H, t, J = 3.2 Hz), 6.20 (1H, dd, J = 1.8, 3.3 Hz).^{13}C NMR (100 MHz, $CDCl_3$) δ 148.80, 148.46, 132.81, 132.68, 130.03, 129.47, 128.57, 124.30, 122.56, 114.77, 110.80, 84.00, 27.47. APCI-MS: *m/z* 289.08 $[M+H]^+$.

2,2,6,6-Tetramethyl-1-(2-nitrophenoxy)piperidine (6a): 2-nitrobenzenediazonium tetrafluoroborate **1a** (118 mg, 0.5 mmol) and TEMPO (156 mg, 1.0 mmol) were added into a deaerated DMSO suspension containing 3 mg SIO, and stirred for 1.5 hours. The product was isolated in 13% yield (36 mg) as yellow oil. ^{1}H NMR (800 MHz, $CDCl_3$) δ 7.87 (1H, d, J = 8.6 Hz), 7.84 (1H, d, J = 8.2 Hz), 7.44 (1H, t, J = 8.5 Hz), 6.91 (1H, t, J = 7.7 Hz), 1.70 (1H, m), 1.64–1.59 (4H, m), 1.43 (1H, d.quint, J = 13.3, 3.4 Hz), 1.29 (6H, s), 1.02 (6H, s). ^{13}C NMR (200 MHz, $CDCl_3$) δ 157.11, 136.20, 133.72, 125.10, 119.49, 117.67, 61.15, 39.67, 32.17, 20.82, 16.80. ESI-HRMS: *m/z* calcd for $C_{15}H_{23}N_2O_3$ $[M+H]^+$ 279.1708; found 279.1709.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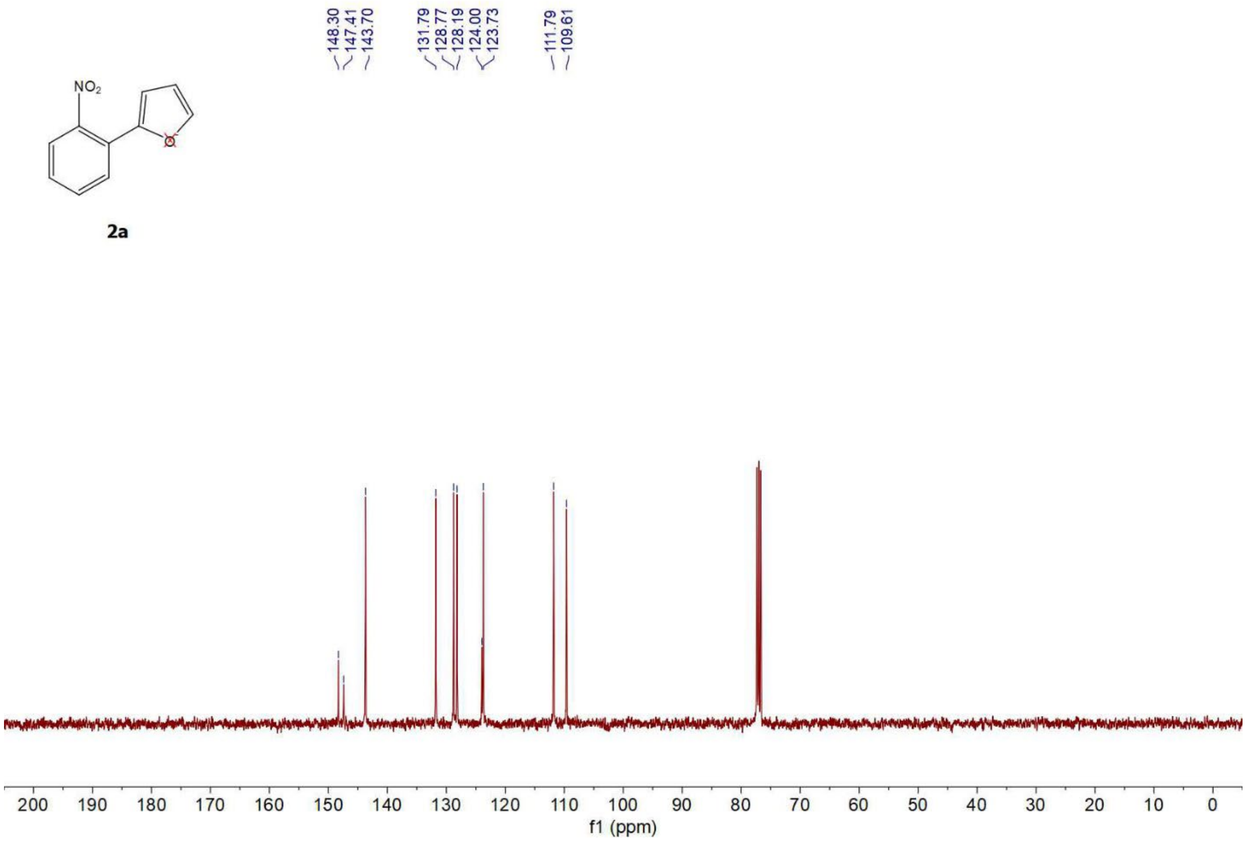
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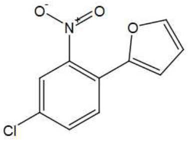
2a



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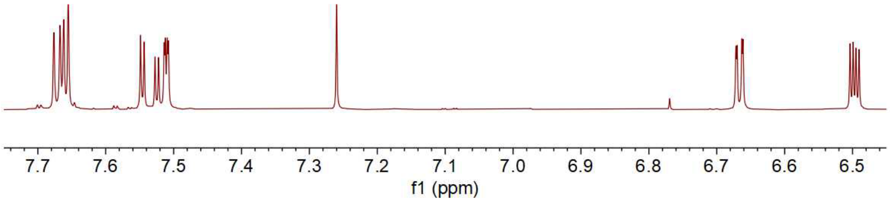
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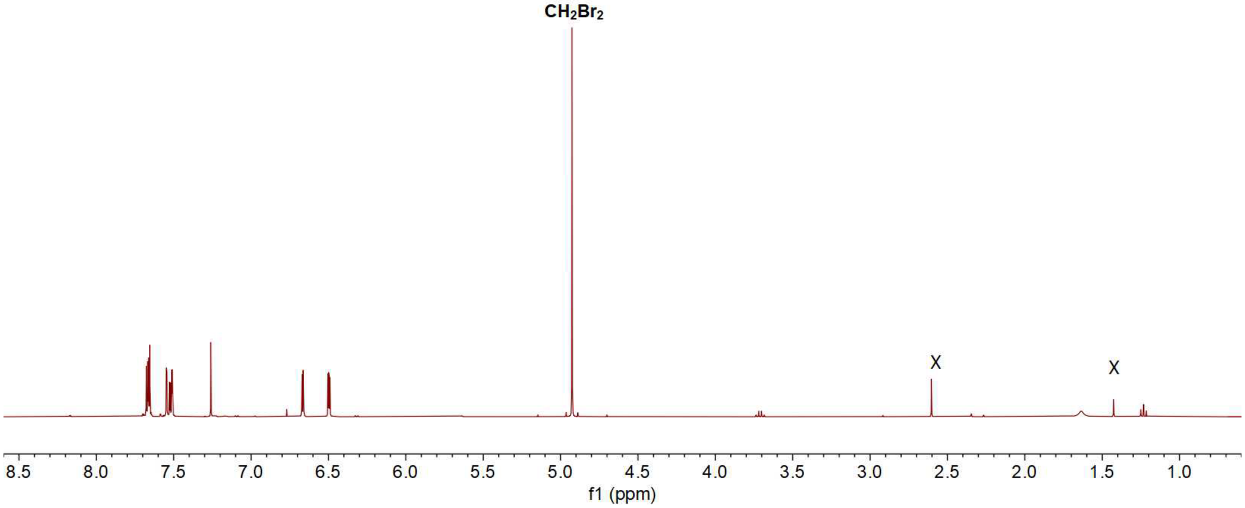
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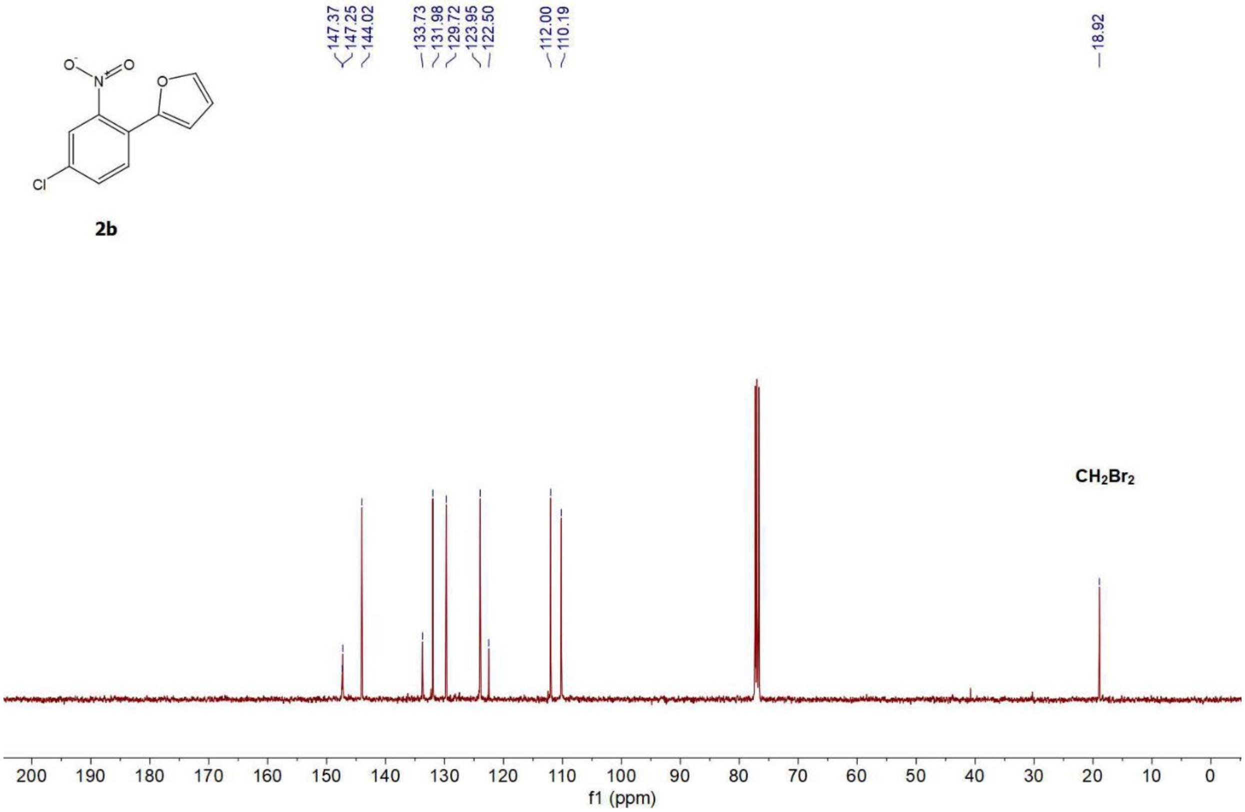

2b

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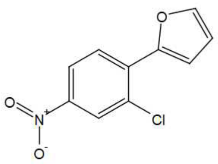
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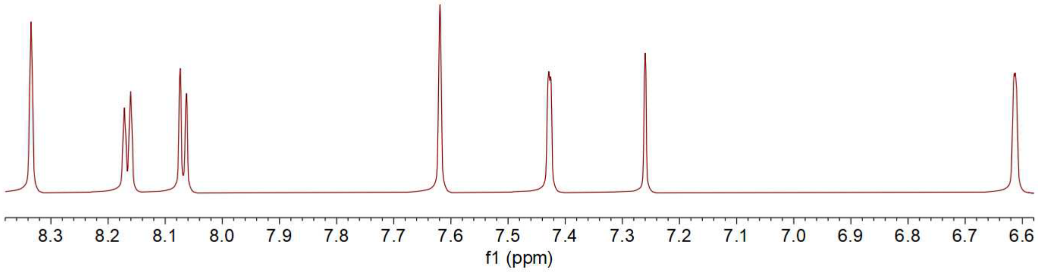
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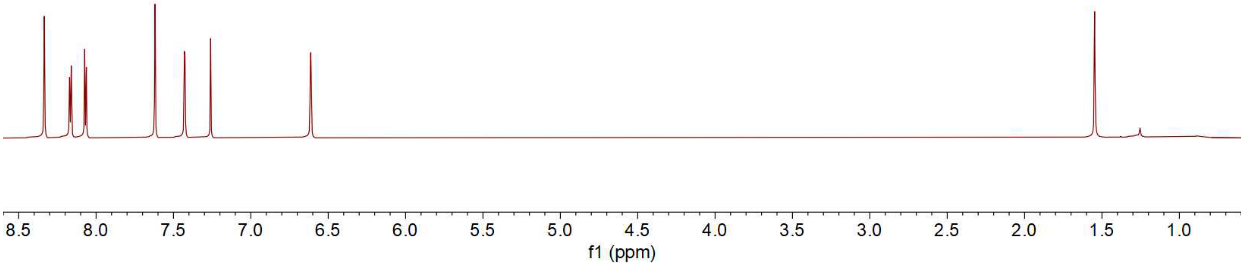
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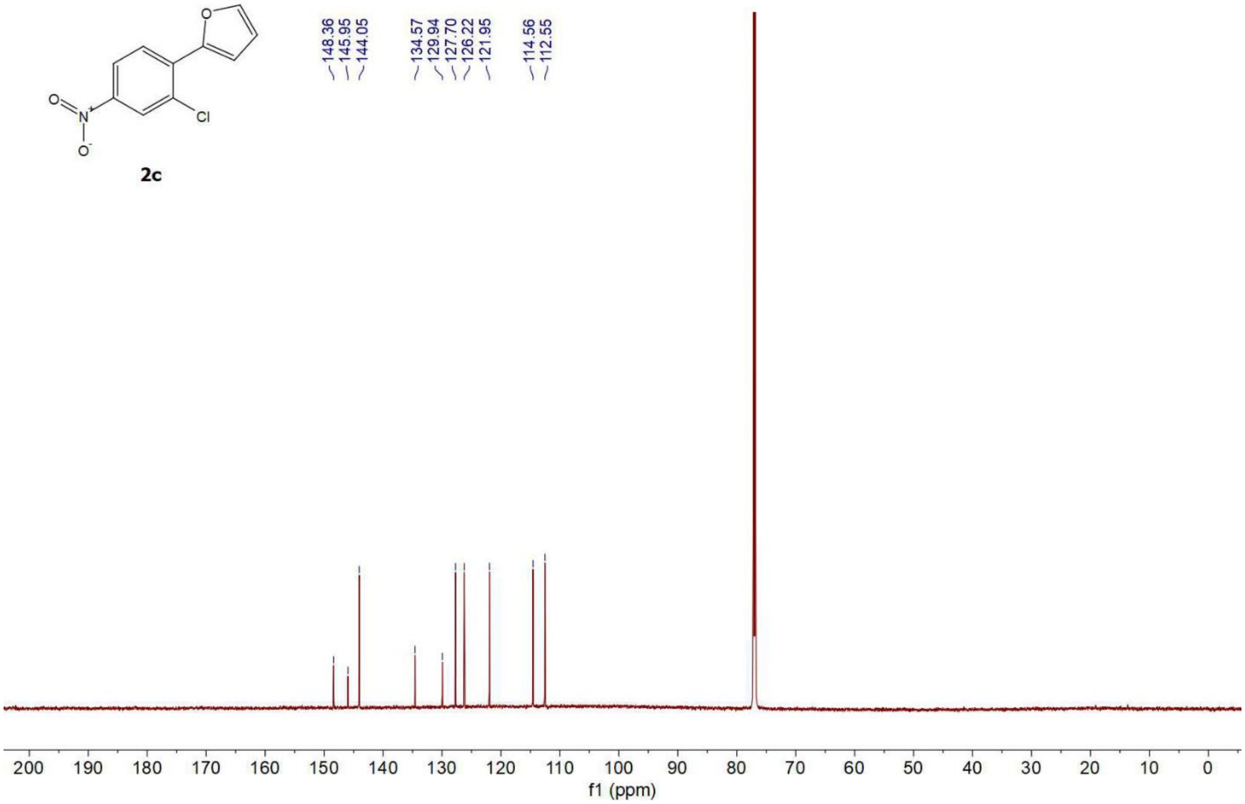
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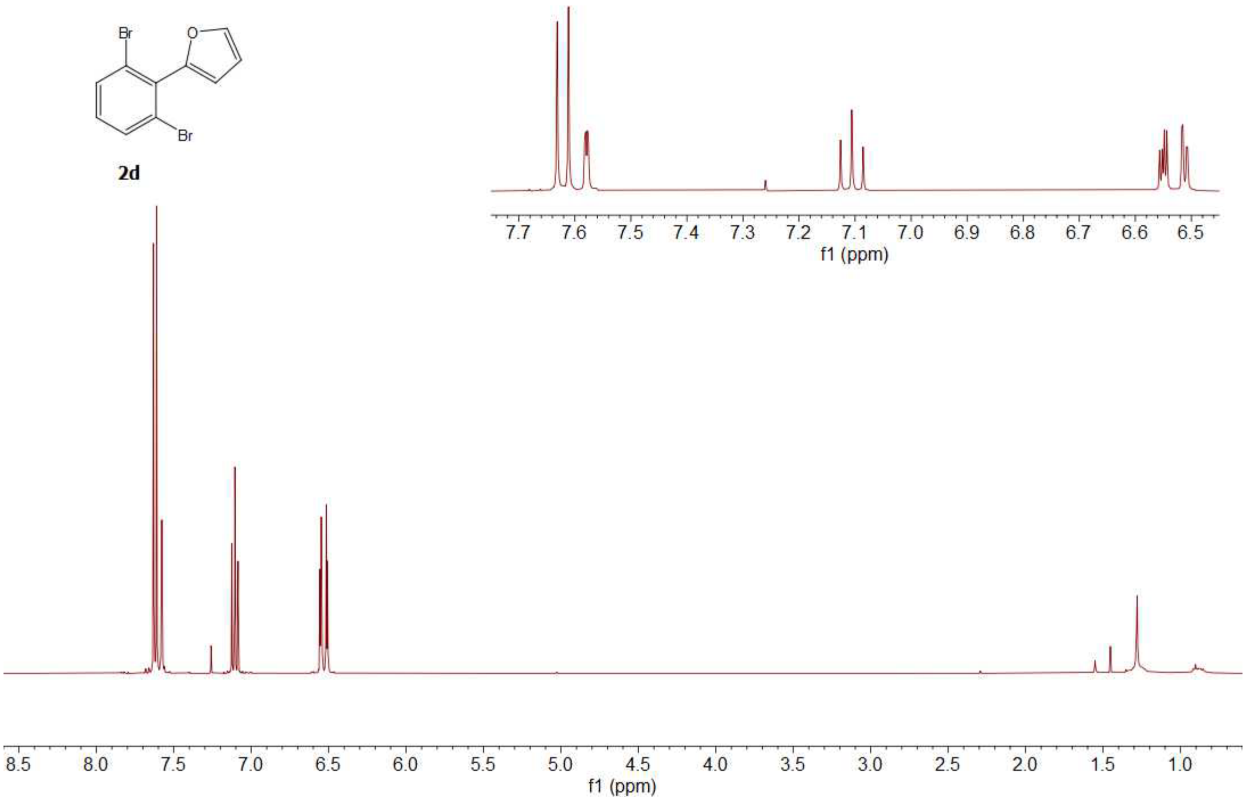
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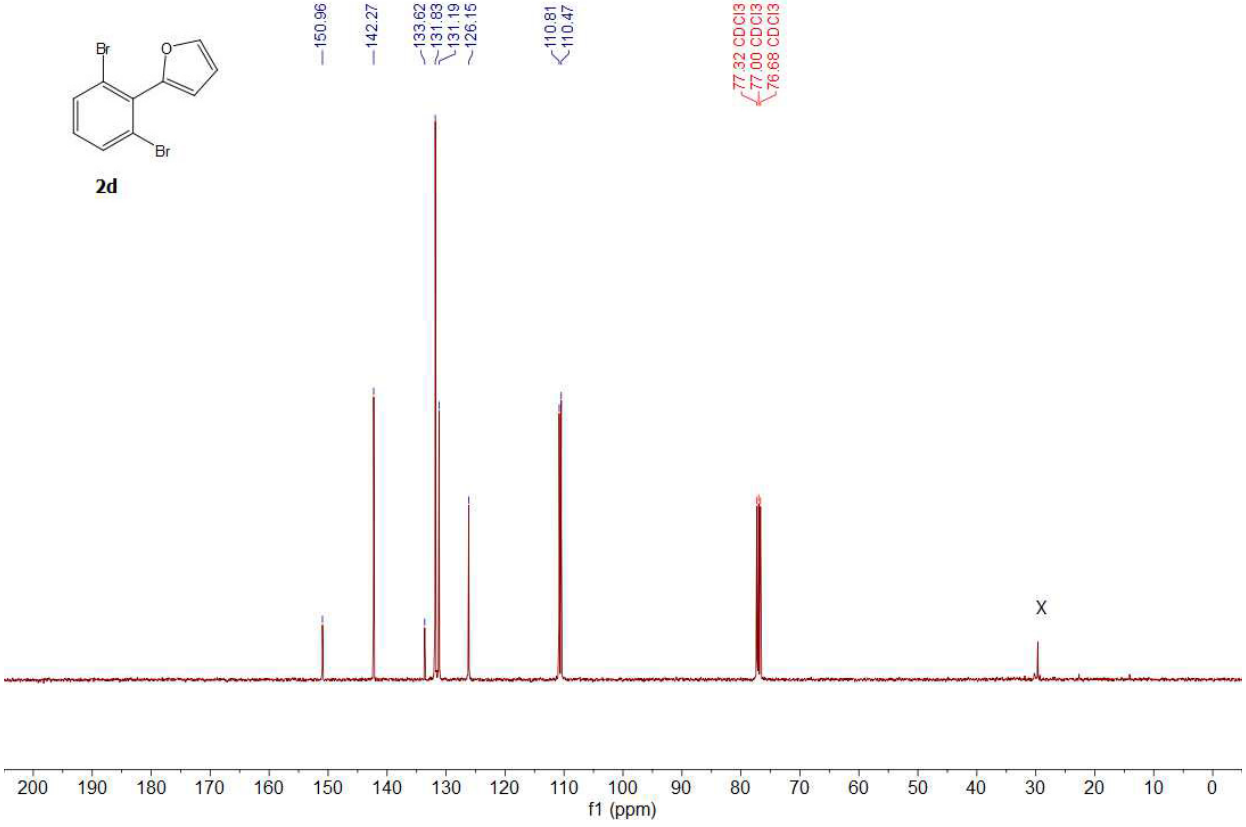
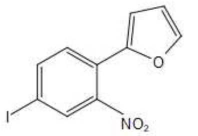
1.0
f1 (ppm)

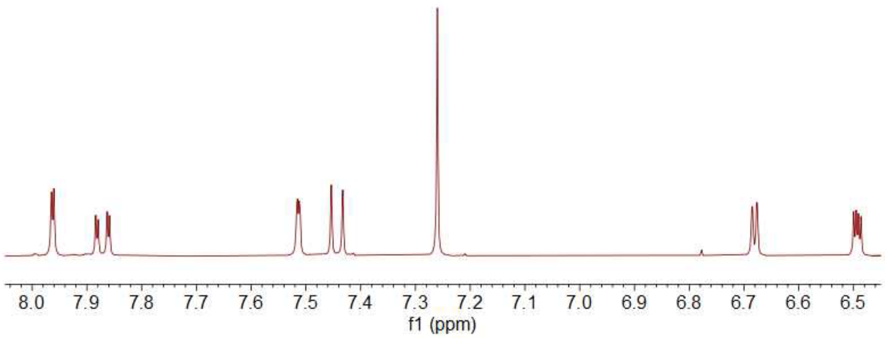

2d

Br
O
Br
150.96
142.27
133.62
131.83
131.19
126.15
110.81
110.47
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
X
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

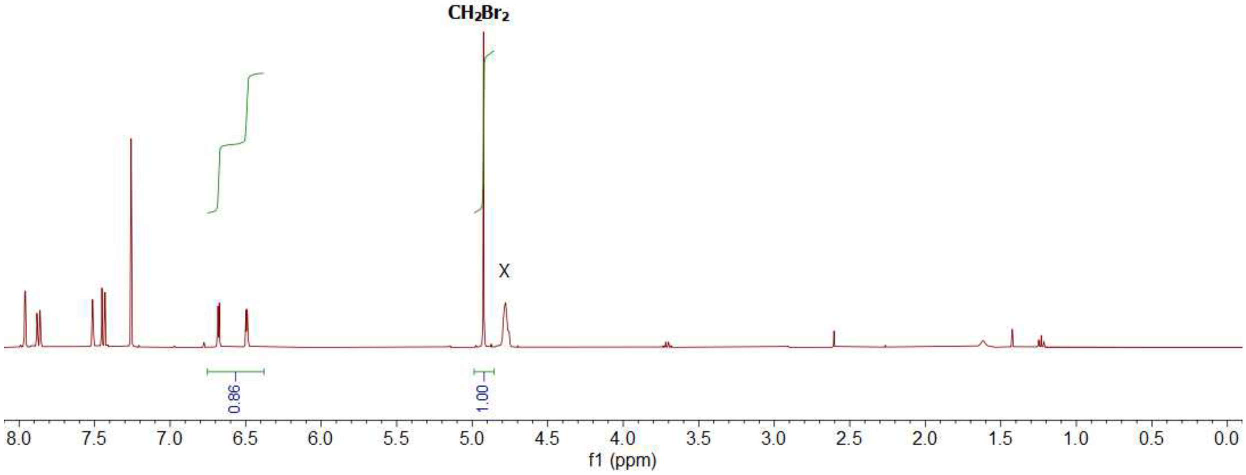


I
NO2
O

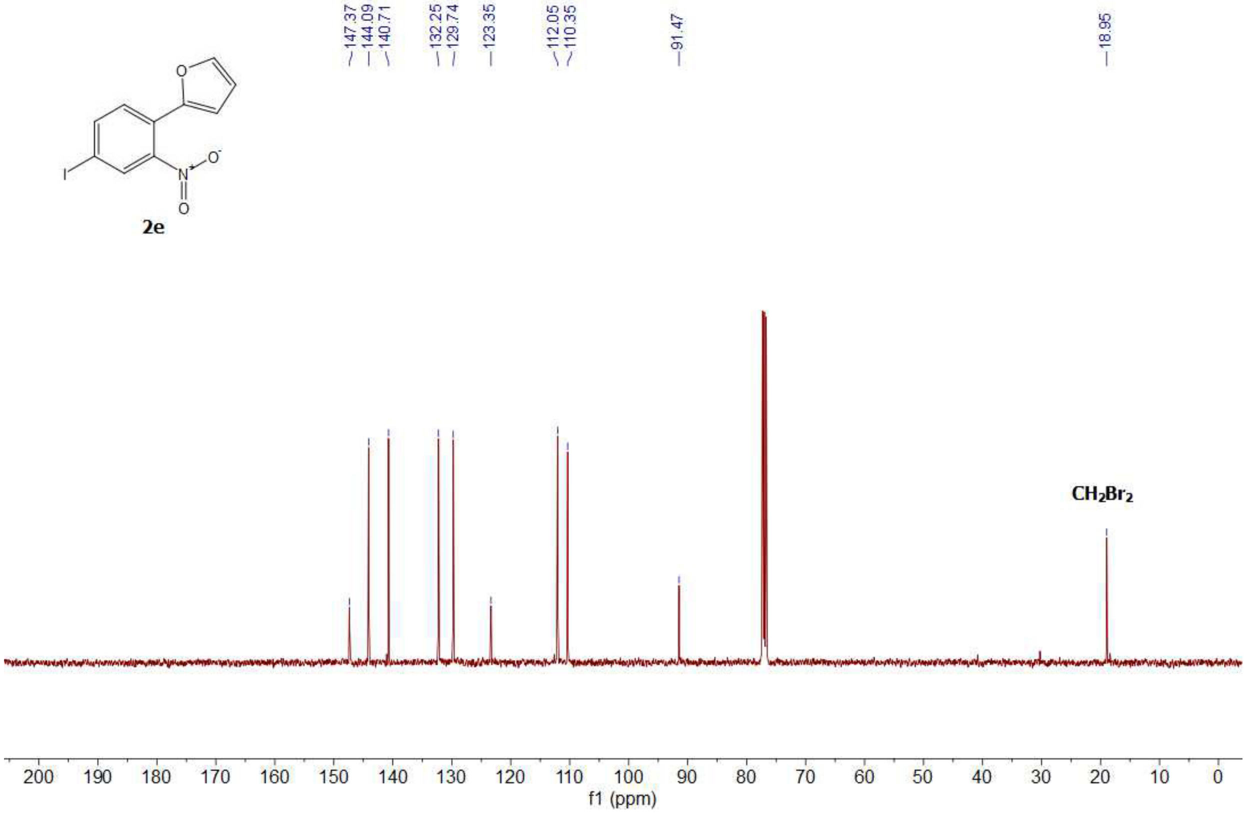
2e



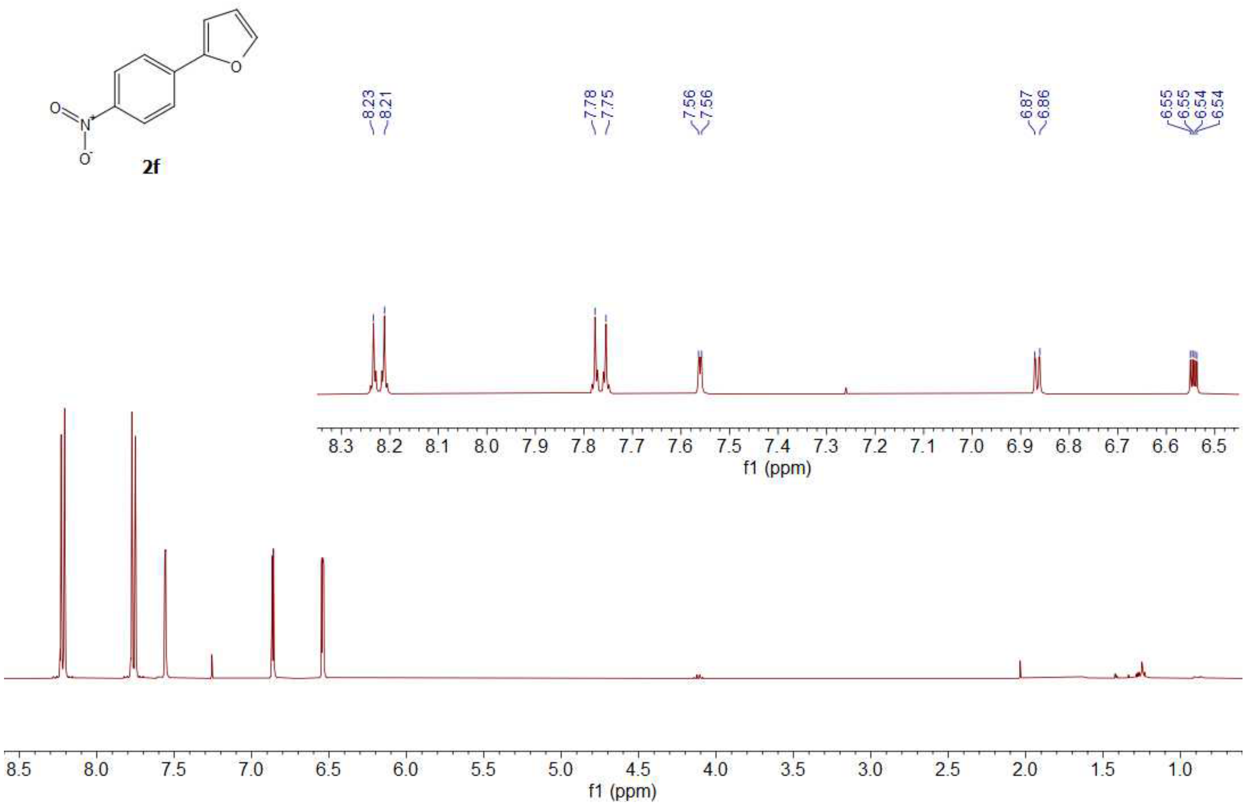
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5
f1 (ppm)



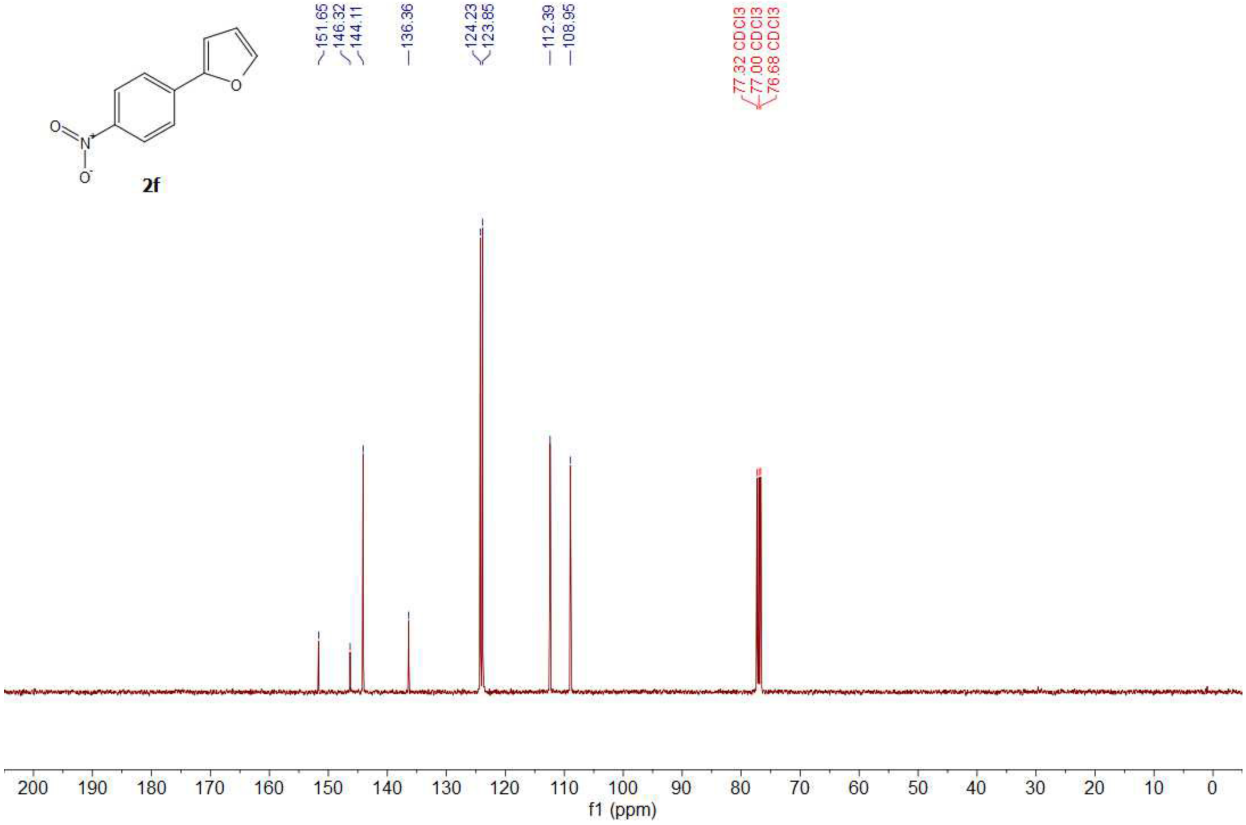
CH2Br2
X
0.86
1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)



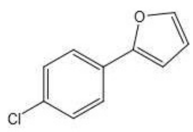
147.37
144.09
140.71
132.25
129.74
123.35
112.05
110.35
91.47
18.95
I
O
N+
O-
O
2e
CH2Br2
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
N+
O-
O
8.23
8.21
7.78
7.75
7.56
7.56
6.87
6.86
6.55
6.55
6.54
6.54
8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5
f1 (ppm)
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
f1 (ppm)

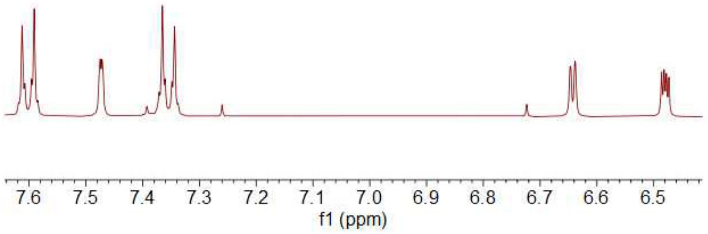
2f

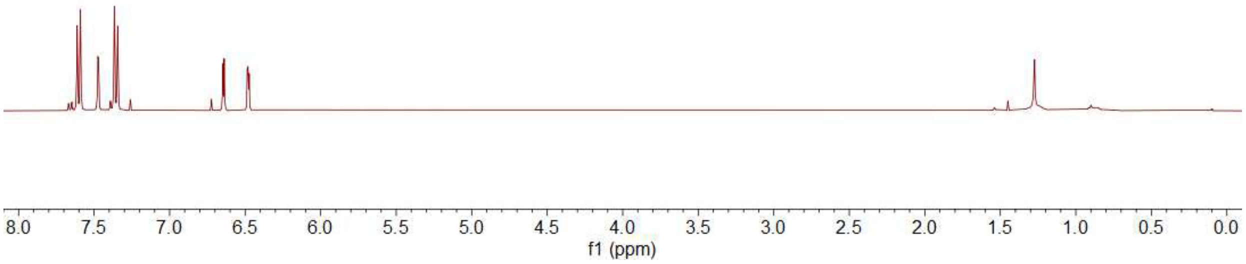

O
N+
O-
O
151.65
146.32
144.11
136.36
124.23
123.85
112.39
108.95
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

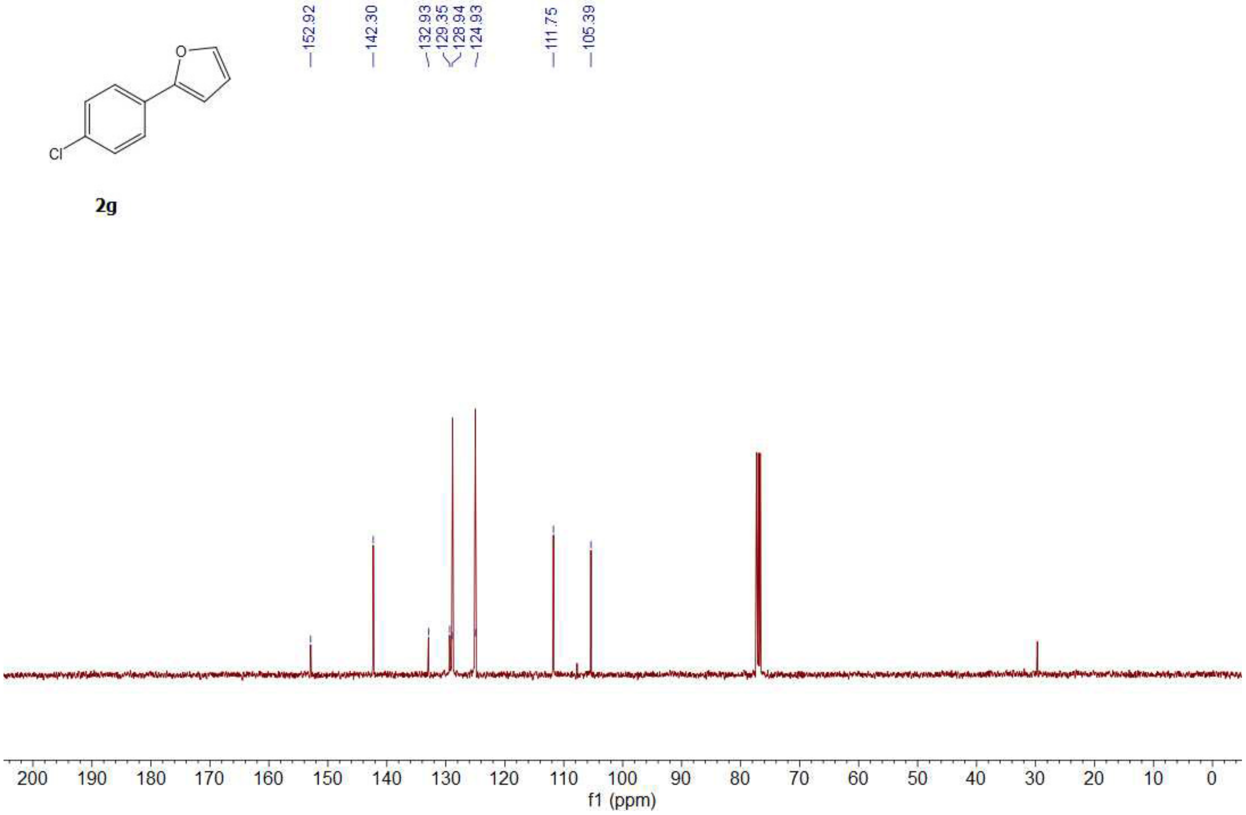
2f


O
Cl

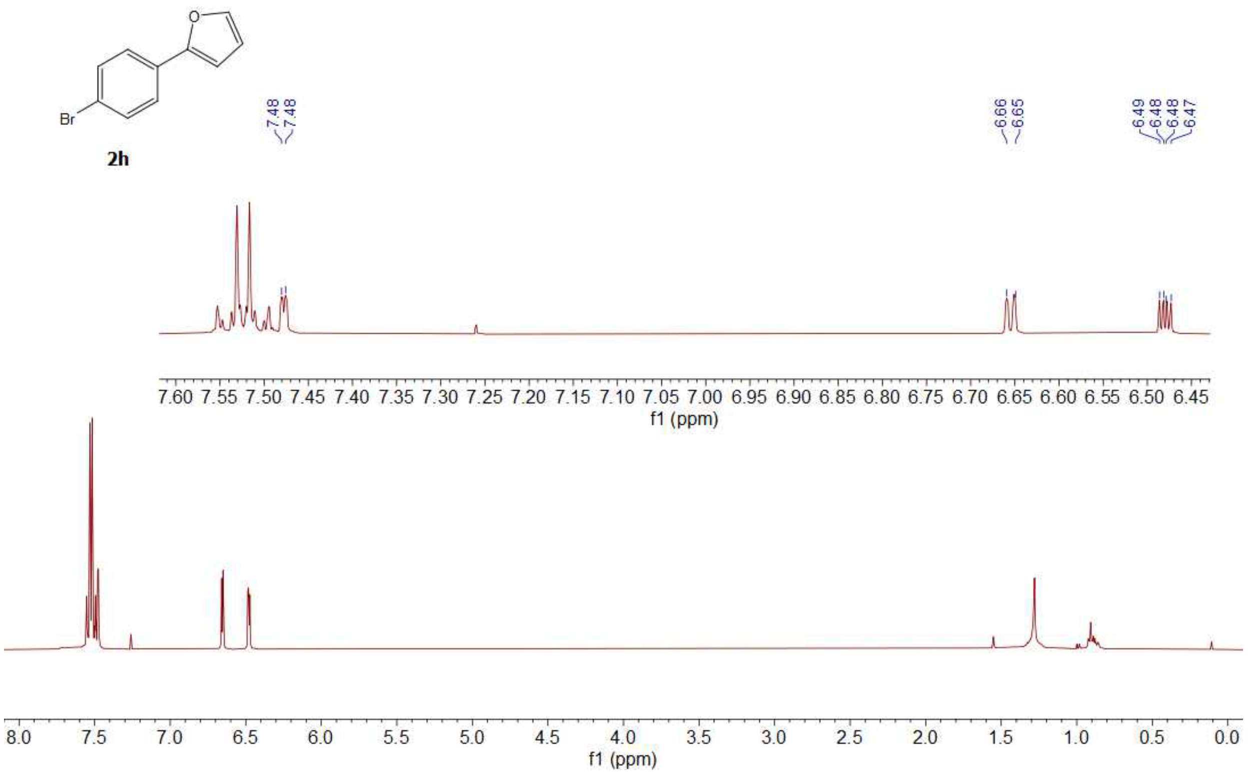
2g


7.6
7.5
7.4
7.3
7.2
7.1
7.0
6.9
6.8
6.7
6.6
6.5
f1 (ppm)

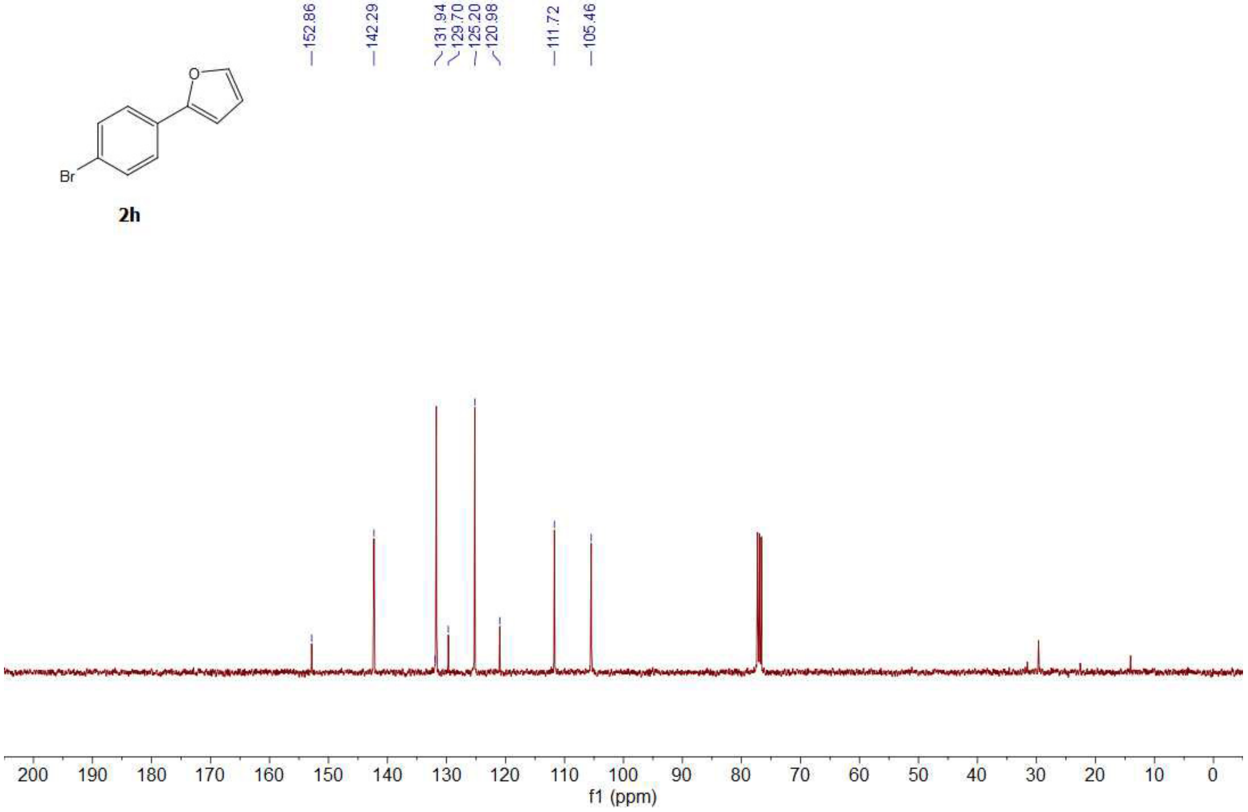

8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


O
Cl
152.92
142.30
132.93
129.35
128.94
124.93
111.75
105.39
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

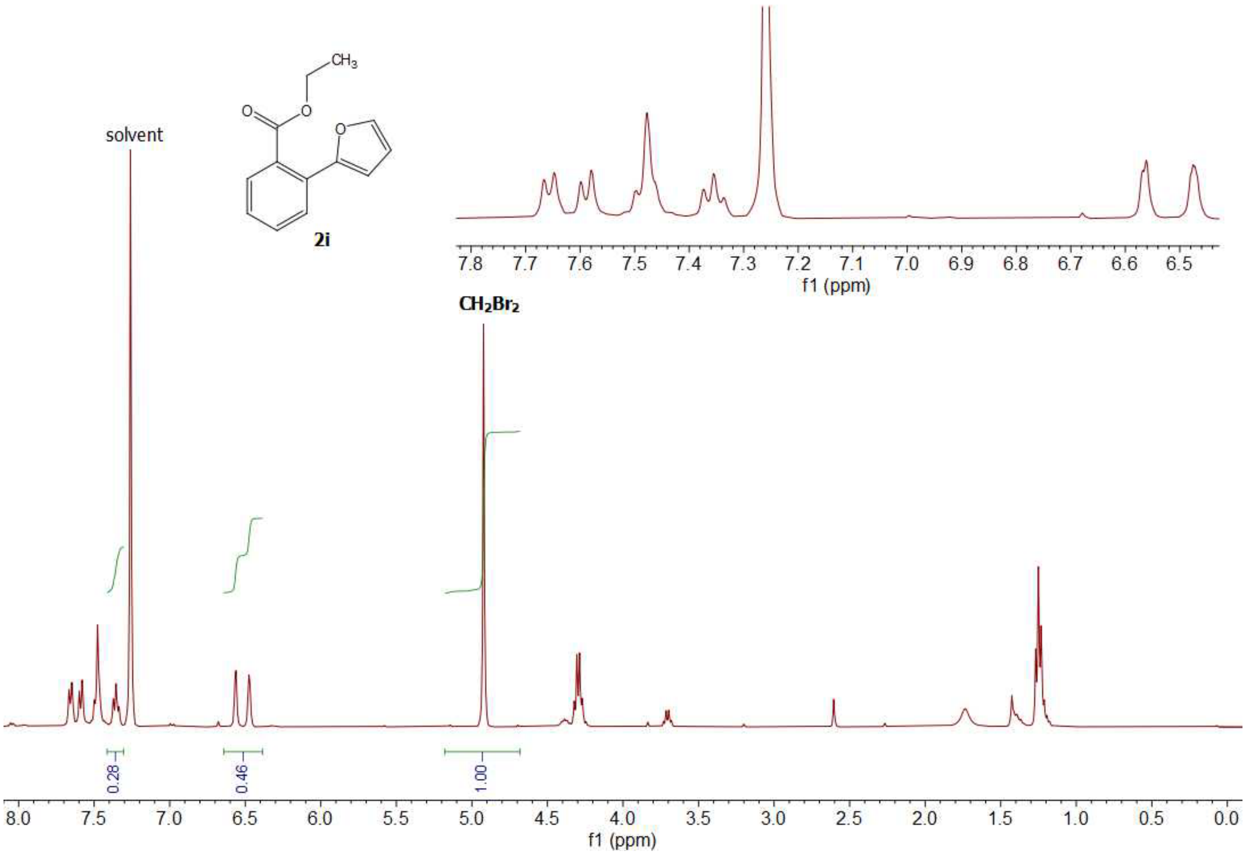
2g

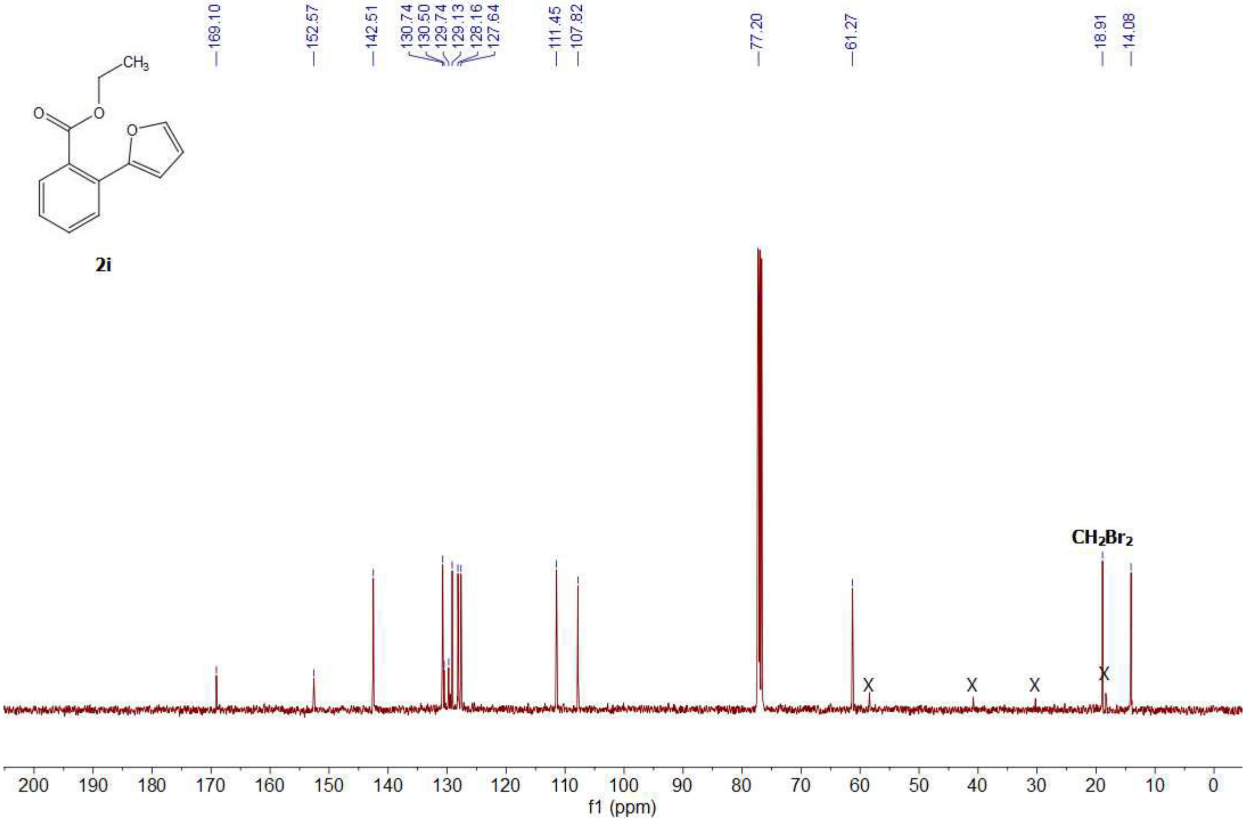

Br
O
7.48
7.48
6.66
6.65
6.49
6.48
6.48
6.47
7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80 6.75 6.70 6.65 6.60 6.55 6.50 6.45
f1 (ppm)
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

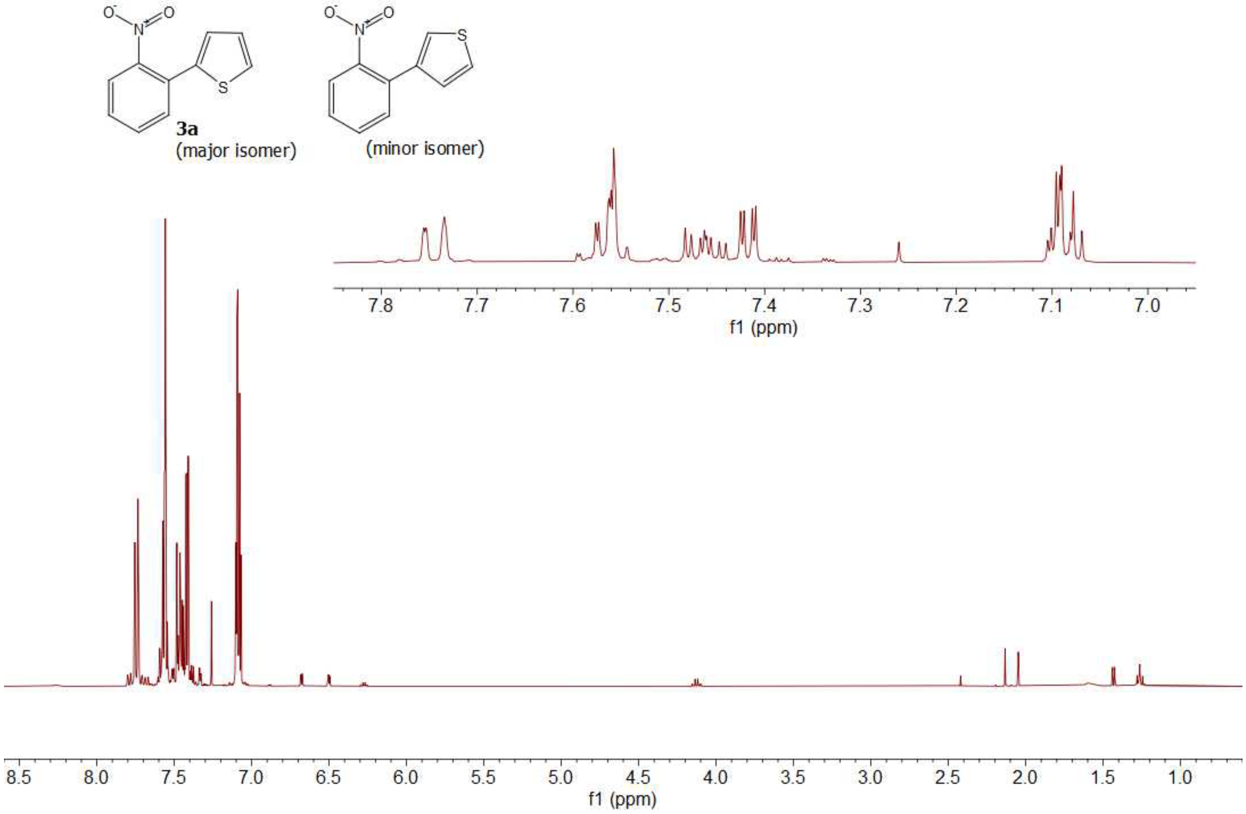
2h

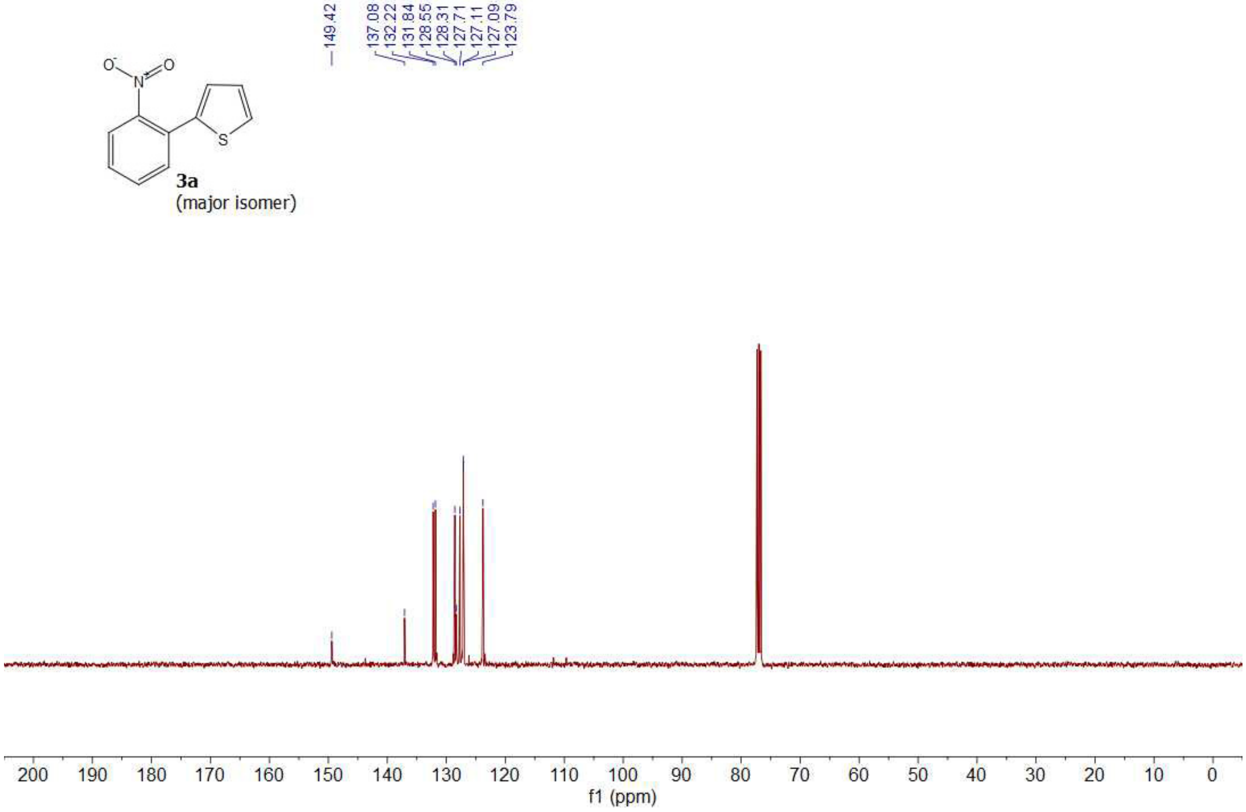

Br
O
152.86
142.29
131.94
129.70
125.20
120.98
111.72
105.46
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

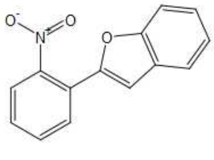
2h


solvent
CH3
O
O
O
2i
7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5
f1 (ppm)
CH2Br2
0.28
0.46
1.00
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 0.0
f1 (ppm)

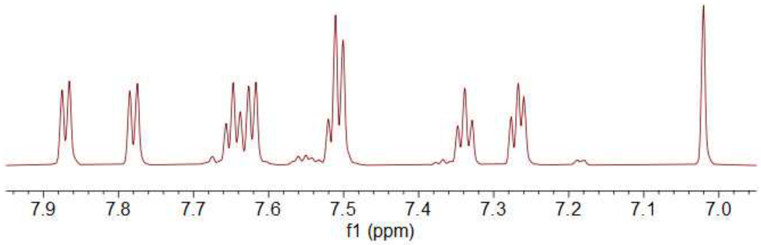

169.10
152.57
142.51
130.74
130.50
129.74
129.13
128.16
127.64
111.45
107.82
77.20
61.27
18.91
14.08
CH3
O
O
O
2i
CH2Br2
X
X
X
X
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

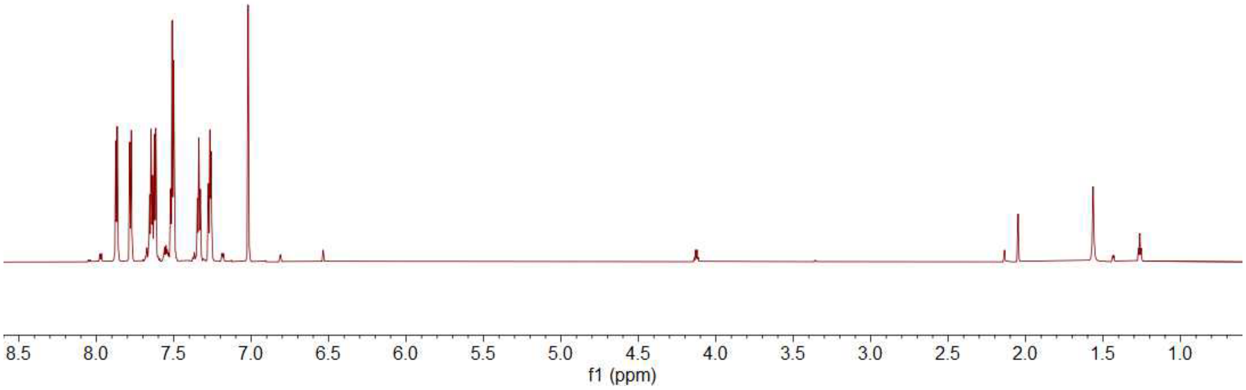

3a
(major isomer)
(minor isomer)
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
f1 (ppm)
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)

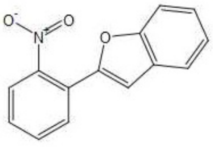

149.42
137.08
132.22
131.84
128.55
128.31
127.71
127.11
127.09
123.79
3a
(major isomer)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



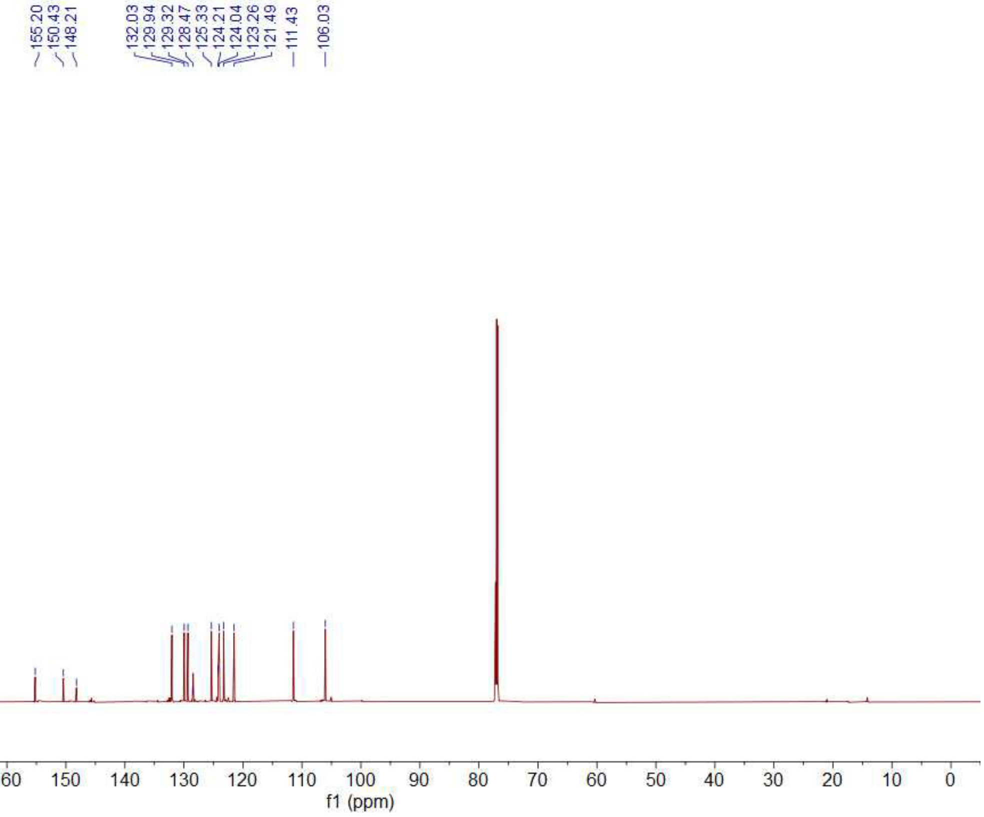
4a

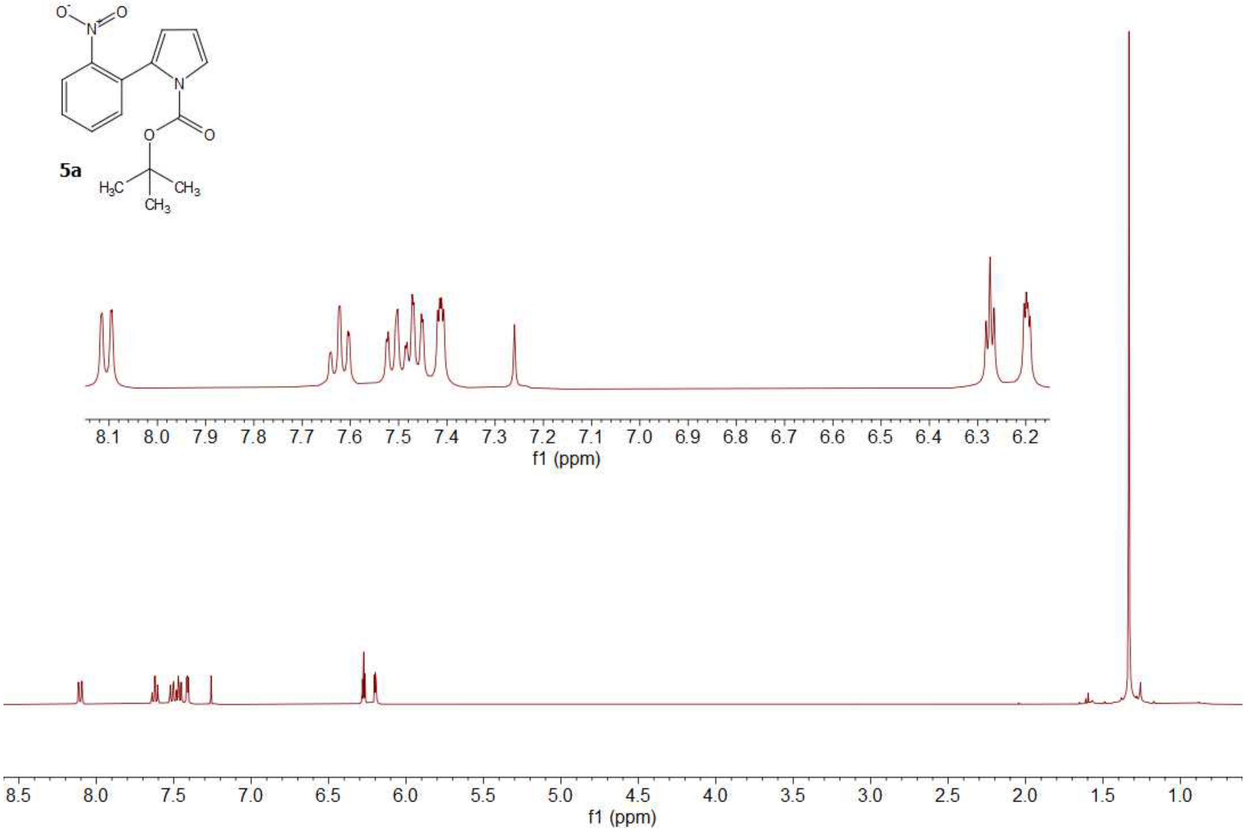

7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
f1 (ppm)


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
f1 (ppm)

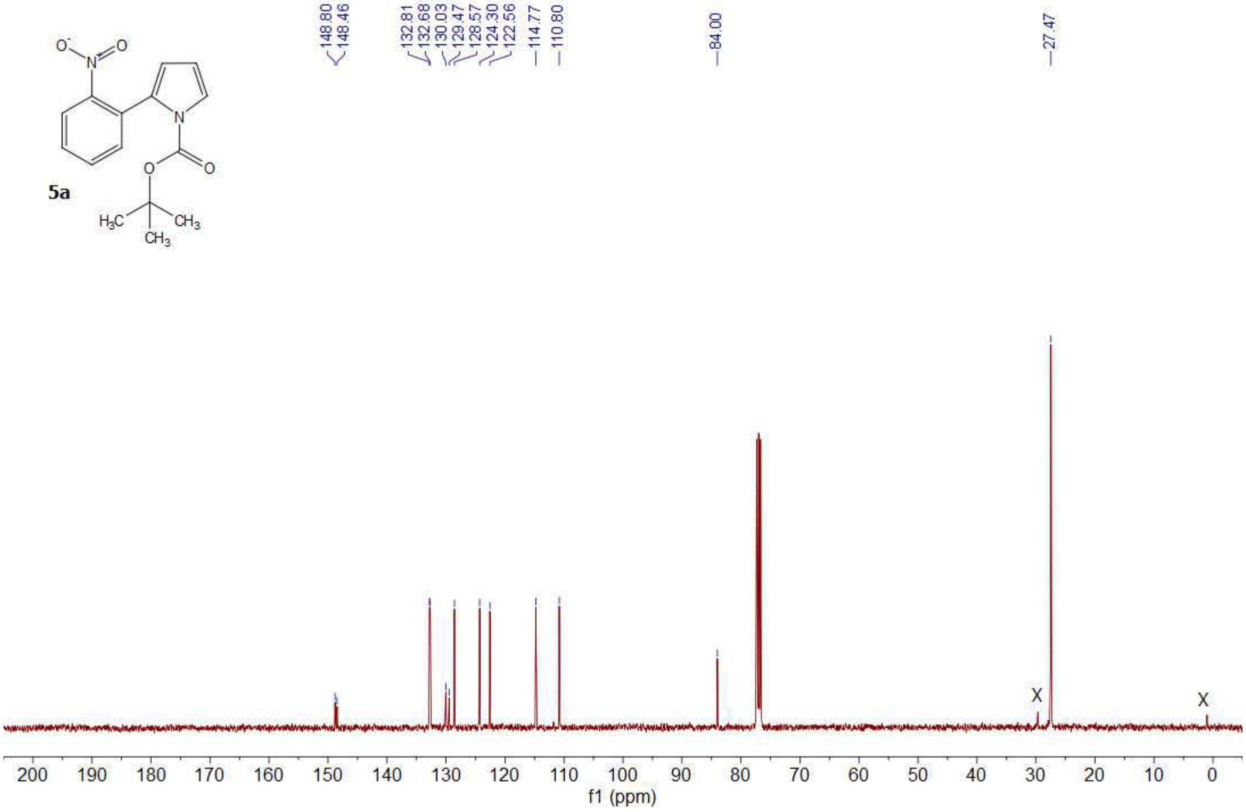


4a

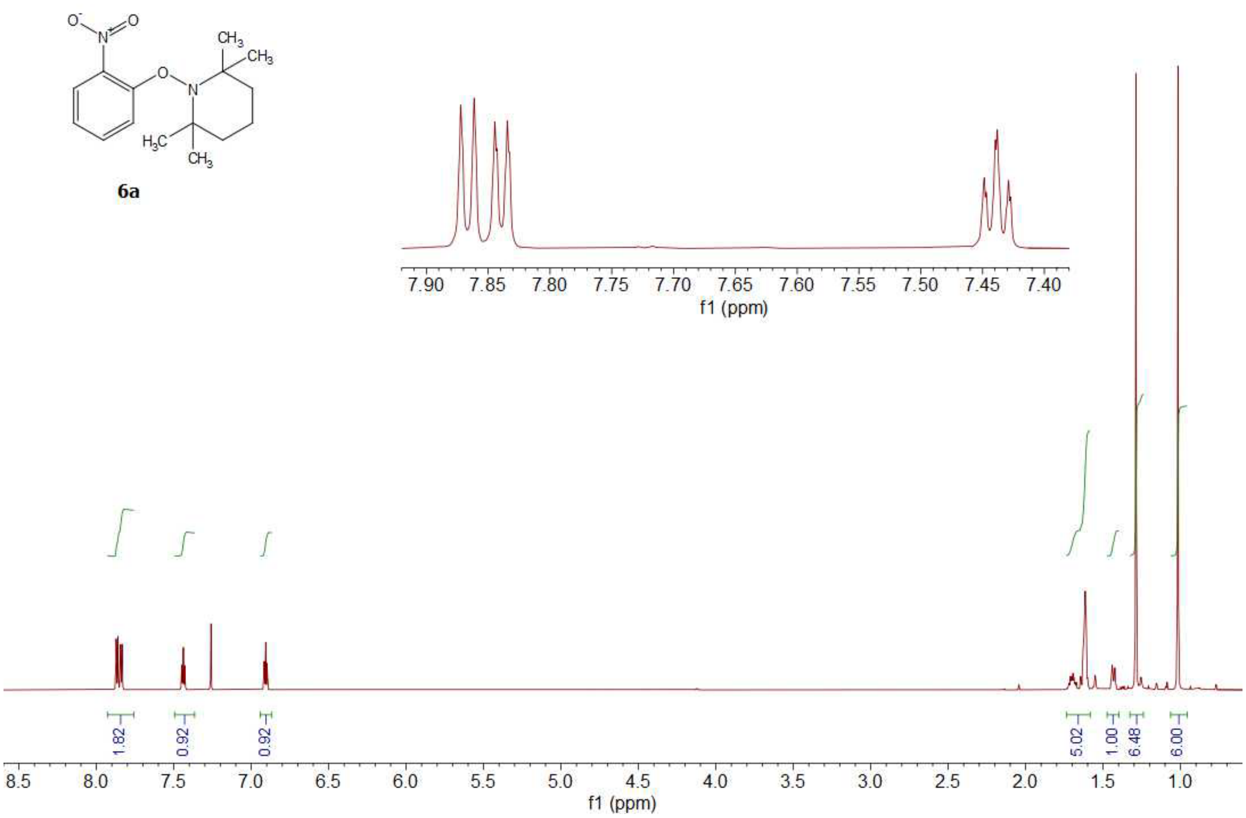

155.20
150.43
148.21
132.03
129.94
129.32
128.47
125.33
124.21
124.04
123.26
121.49
111.43
106.03
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

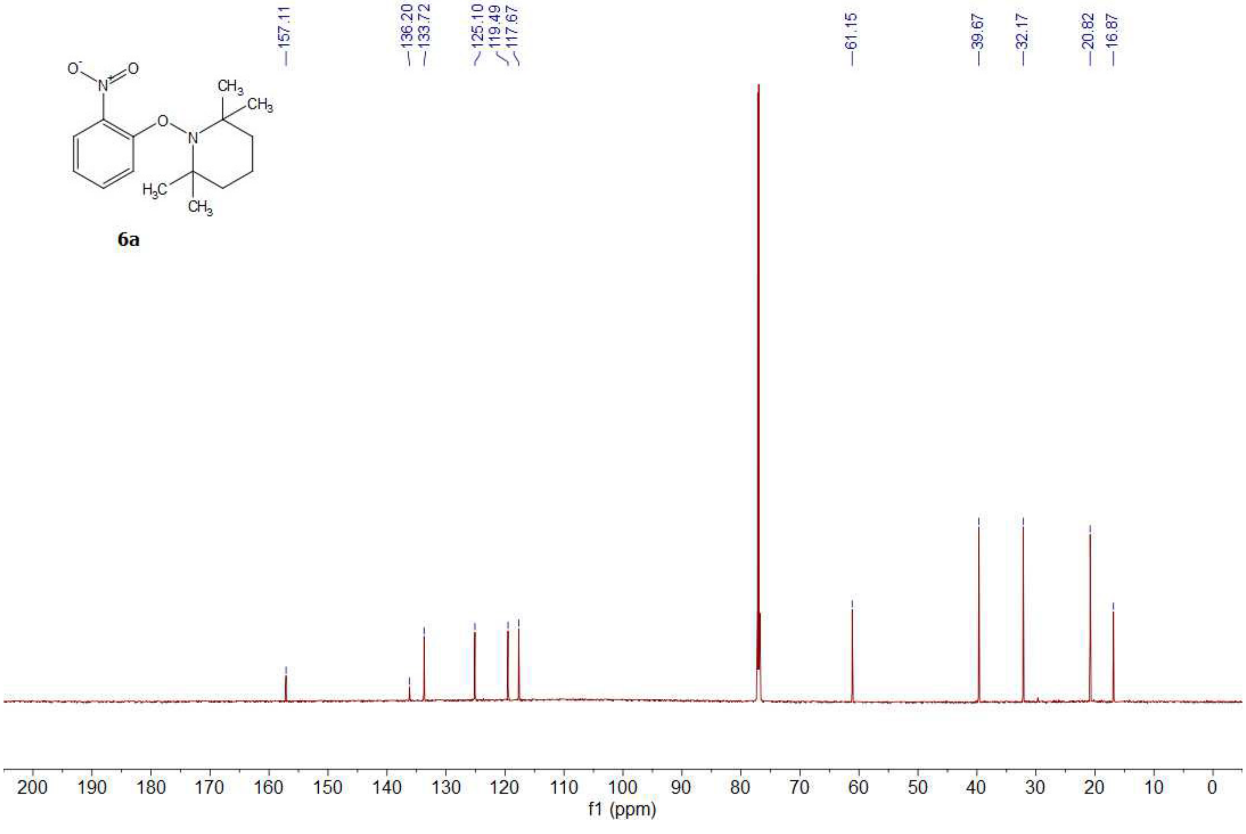

5a

8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2
f1 (ppm)
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
f1 (ppm)


5a

148.80
148.46
132.81
132.68
130.03
129.47
128.57
124.30
122.56
114.77
110.80
84.00
27.47
X
X
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


6a
O-
N+
O
CH3
CH3
H3C
CH3
7.90
7.85
7.80
7.75
7.70
7.65
7.60
7.55
7.50
7.45
7.40
f1 (ppm)
1.82
0.92
0.92
5.02
1.00
6.48
6.00
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
f1 (ppm)


157.11
136.20
133.72
125.10
119.49
117.67
61.15
39.67
32.17
20.82
16.87
6a
O-
N+
O
CH3
CH3
H3C
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

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