Supporting Information

I_2/TBHP mediated oxidative cascade cyclization of vinyl azide and benzylamine to construct 2,5-disubstituted oxazoles

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1. Experimental Section

1.1. General Information

All starting materials and commercial reagents were purchased from Alfa Aesar, Sigma Aldrich, Spectrochem, TCI. Thin Layer Chromatography plates were visualized by exposure to ultraviolet light (UV) with 254 nm of wavelength and then further analyzed by using iodine chamber. Thin-layer chromatography was performed using pre-coated plates. Column chromatography was performed in 120 to 200 mesh size silica gel. The reactions were carried out in a round bottom flask and sealed tube. All NMR spectra of the synthesized products were recorded by Bruker Advanced 400 spectrometer (^{1}H NMR at 400 MHz and ^{13}C NMR at 100 MHz). Chemical shifts for ^{1}H NMR spectra have been reported in parts per million (ppm) from tetramethyl silane with the solvent resonance as the internal standard ($CDCl_3$: δ 7.26 ppm). Similarly, ^{13}C NMR spectra have been reported in parts per million (ppm) from tetramethyl silane with the solvent as the internal standard ($CDCl_3$: δ 77.00 ppm). The ^{1}H NMR and ^{13}C NMR of the known products were compared with literature reports.

1.2. General procedure for the synthesis of 2,5-disubstituted oxazoles (3): In a sealed tube, a mixture of vinyl azide (**1**, 1.0 mmol), and benzylamine (**2**, 3.0 mmol), I_2 (1.0 equiv.) and TBHP (3.0 equiv.) was taken in DMSO (2.0 mL). After that, the reaction mixture was stirred at 80 ºC for 2 h under open air. After completion of the reaction monitoring by TLC, the resulting solution was then cooled to room temperature and the mixture was diluted with ethyl acetate (15 mL) and washed with water (10 mL). Then the crude was extracted with ethyl acetate (3×5 mL), and dried over anhydrous Na_2SO_4 and concentrated in vacuo. Later, the crude product was collected and purified by column chromatography on silica gel (100-200 mesh) using petroleum ether/ethyl acetate (20:1) to get the desired product **3**.

2. HRMS data and spectra:

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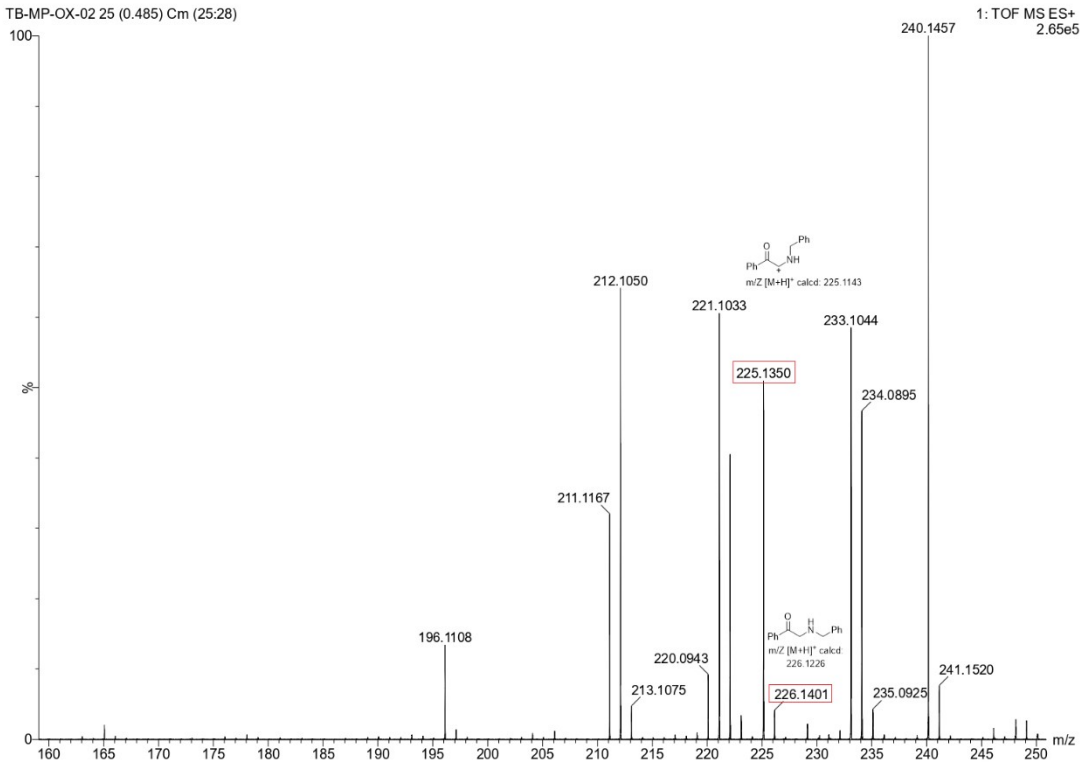

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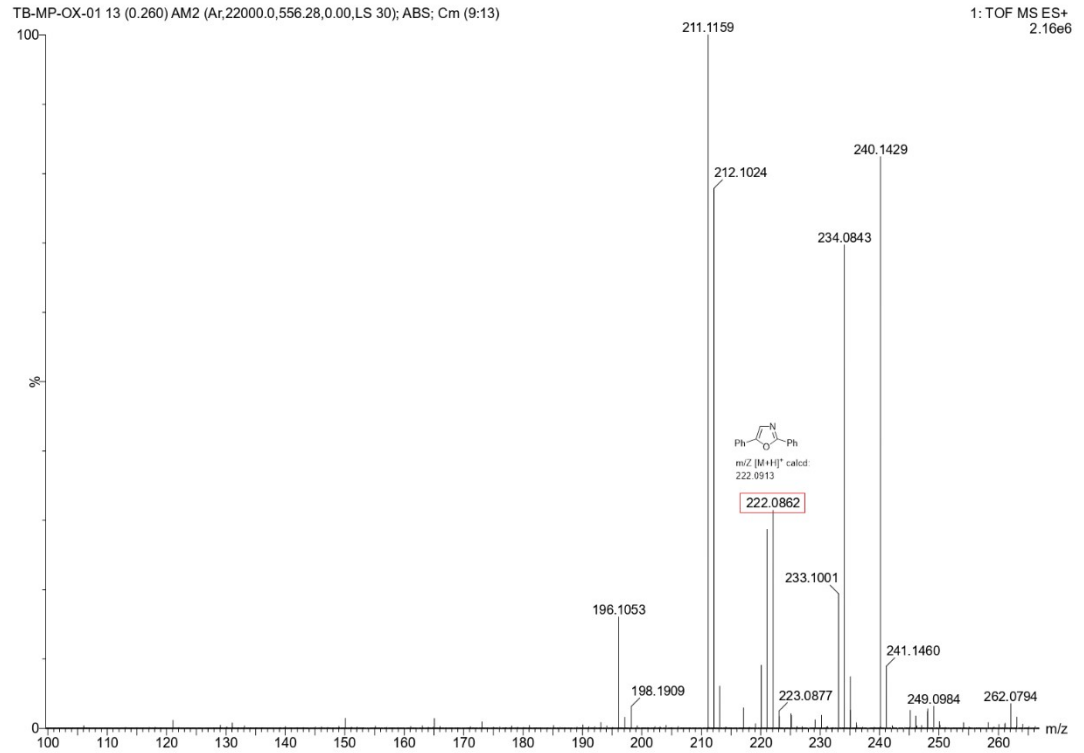


3. ^{1}H and ^{13}C data of the compounds:

2,5-diphenyloxazole (3a)[1]

Yield: 82%; white solid; m.p. 64 - 68 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (dd, *J* = 7.8, 1.8 Hz, 2H), 7.78 – 7.70 (m, 2H), 7.47 (dd, *J* = 12.6, 4.8 Hz, 6H), 7.36 (t, *J* = 7.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.80, 151.79, 130.26, 129.14, 128.83, 127.91, 126.26, 125.89, 125.44, 124.08, 122.85.

2-(4-fluorophenyl)-5-phenyloxazole (3b)[1]

Yield: 78%; white solid; m.p. 87 - 91 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (dd, *J* = 7.8, 5.7 Hz, 2H), 7.64 (d, *J* = 7.9 Hz, 2H), 7.40 – 7.35 (m, 3H), 7.28 (d, *J* = 7.2 Hz, 1H), 7.10 (t, *J* = 8.4 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.0 (d, *J* = 251.1 Hz), 159.3, 150.3, 127.9(2C), 127.4 (d, *J* = 7.5 Hz) 127.3, 126.9, 124.4, 123.2 (2C), 122.8 (d, *J* = 3.3 Hz), 122.4, 115.1, 114.9. HRMS (ESI): m/z for $C_{15}H_{11}FNO$ $(M + H)^+$: calcd: 240.0819, found: 240.0834.

2-(2-fluorophenyl)-5-phenyloxazole (3c)[1]

Yield: 71%; white solid; m.p. 99 - 103 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (t, *J* = 7.6 Hz, 1H), 7.74 (d, *J* = 7.8 Hz, 2H), 7.51 (s, 1H), 7.45 (t, *J* = 7.4 Hz, 3H), 7.35 (t, *J* = 7.4 Hz, 1H), 7.26 (d, *J* = 6.7 Hz, 1H), 7.22 (d, *J* = 8.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.06 (d, *J* = 257.3 Hz), 157.55, 151.56, 131.88 (d, *J* = 8.4 Hz), 129.36 (d, *J* = 1.7 Hz), 128.97 (2C), 128.63, 127.83, 124.40, 124.36 (2C), 123.41, 116.94 (d, *J* = 21.4 Hz), 115.75 (d, *J* = 10.9 Hz).

2-(4-chlorophenyl)-5-phenyloxazole (3d)[1]

Yield: 75%; white solid; m.p. 96 - 99 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.1 Hz, 2H), 7.65 (d, *J* = 7.6 Hz, 2H), 7.39 (d, *J* = 8.7 Hz, 5H), 7.29 (d, *J* = 7.3 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.2, 150.5, 135.4, 128.1, 128.9, 127.6, 126.8, 126.5, 124.9, 123.2, 122.5. HRMS (ESI): m/z for $C_{15}H_{11}ClNO$ $(M + H)^+$: calcd: 256.0524, found: 308.0542.

5-phenyl-2-(4-(trifluoromethyl)phenyl)oxazole (3e)[1]

Yield: 56%; white solid; m.p. 107 - 110 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (d, *J* = 8.1 Hz, 2H), 7.75 (d, *J* = 8.1 Hz, 4H), 7.51 – 7.43 (m, 3H), 7.38 (t, *J* = 7.4 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.1, 152.1, 132.0, 130.5 (d, *J* = 3.3 Hz), 129.0 (2C), 128.9, 127.6, 126.5 (2C), 125.9 (d, *J* = 3.9 Hz), 124.4 (2C), 123.8, 123.7 (dd, *J* = 278.3, 131.9 Hz), 106.2 (d, *J* = 2.1 Hz).

2-(3-fluoro-5-(trifluoromethyl)phenyl)-5-phenyloxazole (3f)

Yield: 53%; white gummy; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (s, 1H) 7.98 (d, *J* = 8.4 Hz, 1H), 7.74 (d, *J* = 7.7 Hz, 2H), 7.52 – 7.44 (m, 3H), 7.40 (dd, *J* = 13.9, 7.0 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.7 (d, *J* = 249.4 Hz), 158.6 (d, *J* = 3.5 Hz), 152.4, 130.4 (d, *J* = 8.7 Hz), 129.1 (2C), 128.9, 128.5, 127.4, 124.5 (2C), 123.8, 123.3 (dd, *J* = 270.0, 75.0 Hz), 118.9 (t, *J* = 3.7 Hz), 116.4 (d, *J* = 24.1 Hz), 114.3 (dq, *J* = 11.2, 3.9 Hz); HRMS (ESI): m/z for $C_{16}H_{10}F_4NO$ $(M + H)^+$: calcd: 308.0693, found: 308.0690.

5-(4-fluorophenyl)-2-phenyloxazole (3g)[1]

Yield: 72%; white solid; m.p. 89 - 93 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (d, *J* = 6.6 Hz, 2H), 7.72 – 7.67 (m, 2H), 7.48 (d, *J* = 5.6 Hz, 3H), 7.38 (s, 1H), 7.14 (t, *J* = 8.1 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.7 (d, *J* = 248.9 Hz), 161.2, 150.5, 134.8, 131.6 (d, *J* = 9.5 Hz), 130.4, 128.8 (2C), 127.3, 126.3 (2C), 126.1 (d, *J* = 8.2 Hz), 124.4 (d, *J* = 3.4 Hz), 123.1, 116.1 (d, *J* = 22.1 Hz).

5-(4-chlorophenyl)-2-phenyloxazole (3h)[1]

Yield: 77%; white solid; m.p. 98 - 101 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 – 8.08 (m, 2H), 7.66 (d, *J* = 8.4 Hz, 2H), 7.49 (d, *J* = 5.5 Hz, 3H), 7.44 (d, *J* = 2.7 Hz, 2H), 7.41(s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.4, 150.3, 134.2, 130.5, 129.2 (2C), 128.9 (2C), 127.3, 126.5, 126.3 (2C), 125.4 (2C), 123.8.

5-(4-chlorophenyl)-2-(4-fluorophenyl)oxazole (3i)[1]

Yield: 65%; yellow solid; m.p. 84 - 88 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 – 7.94 (m, 2H), 7.58 (d, *J* = 8.0 Hz, 2H), 7.37 (t, *J* = 9.2 Hz, 3H), 7.11 (t, *J* = 8.3 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.7, 150.4, 134.0, 129.3, 128.5 (d, *J* = 8.6 Hz), 126.4, 125.4, 123.83, 116.2, 115.99.

5-(4-chlorophenyl)-2-(4-(trifluoromethyl)phenyl)oxazole (3j)

Yield: 54%; yellow solid; m.p. 91 - 95 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (d, *J* = 7.8 Hz, 2H), 7.75 (d, *J* = 7.8 Hz, 2H), 7.67 (d, *J* = 8.5 Hz, 2H), 7.51 – 7.40 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.13, 144.58, 134.70, 130.37, 129.35, 126.54 (2C), 126.12, 125.92 (dd, *J* = 7.8, 4.0 Hz), 125.61 (2C), 125.19 (d, *J* = 2.0 Hz), 124.16, 123.20 (dd, J = 279.5, 130.7 Hz), 122.47; HRMS (ESI): m/z for $C_{16}H_{10}ClF_3NO$ $(M + H)^+$: calcd: 324.0398, found: 324.0392.

5-(4-chlorophenyl)-2-(3-fluoro-5-(trifluoromethyl)phenyl)oxazole (3k)

Yield: 51%; yellow gummy; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (s, 1H), 7.97 (d, *J* = 8.8 Hz, 1H), 7.67 (d, *J* = 8.6 Hz, 2H), 7.45 (dd, *J* = 18.2, 9.5 Hz, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.7 (d, *J* = 249.8 Hz), 151.4, 134.9, 131.5, 130.2 (d, *J* = 8.6 Hz), 129.4 (2C), 128.9, 125.8, 125.7 (2C), 124.1, 123.8 (dd, *J* = 284.3, 145.8 Hz), 119.0 – 118.8 (m), 116.5 (d, *J* = 24.8 Hz), 114.5 (dd, *J* = 24.5, 3.7 Hz); HRMS (ESI): m/z for $C_{16}H_9ClF_4NO$ $(M + H)^+$: calcd: 342.0303, found: 342.0300.

5-(3-chlorophenyl)-2-phenyloxazole (3l)[2]

Yield: 70%; white gummy; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 – 8.08 (m, 2H), 7.71 (s, 1H), 7.59 (d, *J* = 7.6 Hz, 1H), 7.52 – 7.45 (m, 4H), 7.38 (t, *J* = 7.7 Hz, 1H), 7.31 (d, *J* = 7.9 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.6, 149.9, 135.0, 130.6, 130.3, 129.7, 128.9 (2C), 128.4, 127.2, 126.4 (2C), 124.4, 124.2, 122.2.

5-(3-chlorophenyl)-2-(2-fluorophenyl)oxazole (3m)

Yield: 61%; white gummy; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (t, *J* = 7.5 Hz, 1H), 7.72 (s, 1H), 7.62 (d, *J* = 7.6 Hz, 1H), 7.53 (s, 1H), 7.47 (dd, *J* = 11.7, 5.9 Hz, 1H), 7.39 (t, *J* = 7.8 Hz, 1H), 7.35 – 7.27 (m, 2H), 7.23 (d, *J* = 10.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.10 (d, *J* = 257.5 Hz), 135.06, 132.19 (d, *J* = 8.6 Hz), 130.30 (2C), 129.47 – 129.40 (m), 128.58 (2C), 124.45 (d, *J* = 3.7 Hz), 124.31 (d, *J* = 3.9 Hz) (2C), 122.39 (2C), 116.99 (d, *J* = 21.3 Hz), 115.53. HRMS (ESI): m/z for $C_{15}H_{10}ClFNO$ $(M + H)^+$: calcd: 274.0357, found: 274.0361.

2-phenyl-5-(p-tolyl)oxazole (3n)[3]

Yield: 56%; white solid. m.p. 73-76 °C; 1H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.0 Hz, 2H), 7.70 – 7.60 (m, 2H), 7.55 (t, *J* = 7.7 Hz, 2H), 7.48 (s, 1H), 7.29 (s, 2H), 2.44 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.3, 151.1 138.5, 130.4, 130.3, 128.8, 128.7, 128.3, 127.2, 126.1, 122.8, 19.8.

2-(2-fluorophenyl)-5-(p-tolyl)oxazole (3o)

Yield: 49%; white gummy; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (td, *J* = 7.6, 1.7 Hz, 1H), 7.64 (d, *J* = 8.2 Hz, 2H), 7.46 (s, 1H), 7.30 – 7.20 (m, 4H), 2.40 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.01 (d, *J* = 257.1 Hz), 157.18, 151.75, 138.71, 131.75 (d, *J* = 8.4 Hz), 129.65 (2C), 129.30 (d, *J* = 1.8 Hz), 125.08, 124.37, 124.33 (2C), 122.78, 116.92 (d, *J* = 21.4 Hz),

115.83 (d, *J* = 10.8 Hz), 21.40; HRMS (ESI): m/z for $C_{16}H_{13}FNO$ $(M + H)^+$: calcd: 254.0976, found: 254.0976.

2-(4-fluorophenyl)-5-(p-tolyl)oxazole (3p)

Yield: 53%; white solid. m.p. 85-89 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 8.14 – 8.06 (m, 2H), 7.61 (d, *J* = 8.2 Hz, 2H), 7.38 (s, 1H), 7.26 (d, *J* = 7.4 Hz, 2H), 7.17 (t, *J* = 8.7 Hz, 2H), 2.40 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.99 (d, *J* = 250.8 Hz), 160.03, 151.58, 138.60, 129.65 (2C), 128.32 (d, *J* = 8.6 Hz), 125.18, 124.17 (2C), 123.94 (d, *J* = 3.3 Hz), 122.74,116.00 (d, *J* = 22.2 Hz), 21.38; HRMS (ESI): m/z for $C_{16}H_{13}FNO$ $(M + H)^+$: calcd: 254.0976, found: 254.0988.

5-(4-(tert-butyl)phenyl)-2-(2-fluorophenyl)oxazole (3q)

Yield: 50%; white gummy; ^1H NMR (400 MHz, $CDCl_3$) δ 8.11 (td, *J* = 7.6, 1.7 Hz, 1H), 7.68 (d, *J* = 8.5 Hz, 2H), 7.48 (d, *J* = 8.6 Hz, 3H), 7.30 – 7.20 (m, 3H), 1.36 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.00 (d, *J* = 257.2 Hz), 157.22 (d, *J* = 5.0 Hz), 151.93, 151.70, 131.74 (d, *J* = 8.4 Hz), 131.25, 129.29 (d, *J* = 1.7 Hz), 125.89 (2C), 125.58 (d, *J* = 3.9 Hz), 125.04, 124.35 (d, *J* = 3.7 Hz), 124.20 (2C), 122.87, 120.68, 116.91 (d, *J* = 21.4 Hz), 31.23, 29.71; HRMS (ESI): m/z for $C_{19}H_{19}FNO$ $(M + H)^+$: calcd: 296.1445, found: 296.1454.

5-(4-(tert-butyl)phenyl)-2-(4-chlorophenyl)oxazole (3r)

Yield: 52%; white gummy; ^1H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.6 Hz, 2H), 7.65 (d, *J* = 8.4 Hz, 2H), 7.47 (dd, *J* = 8.4, 6.3 Hz, 4H), 7.40 (s, 1H), 1.36 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 13C NMR (101 MHz, CDCl3) δ 159.98, 151.98, 151.75, 136.31, 129.50, 129.15, 127.50, 125.92, 125.46, 124.12, 122.97, 31.23, 29.71; HRMS (ESI): m/z for $C_{19}H_{19}ClNO$ $(M + H)^+$: calcd: 312.1150, found: 312.1159.

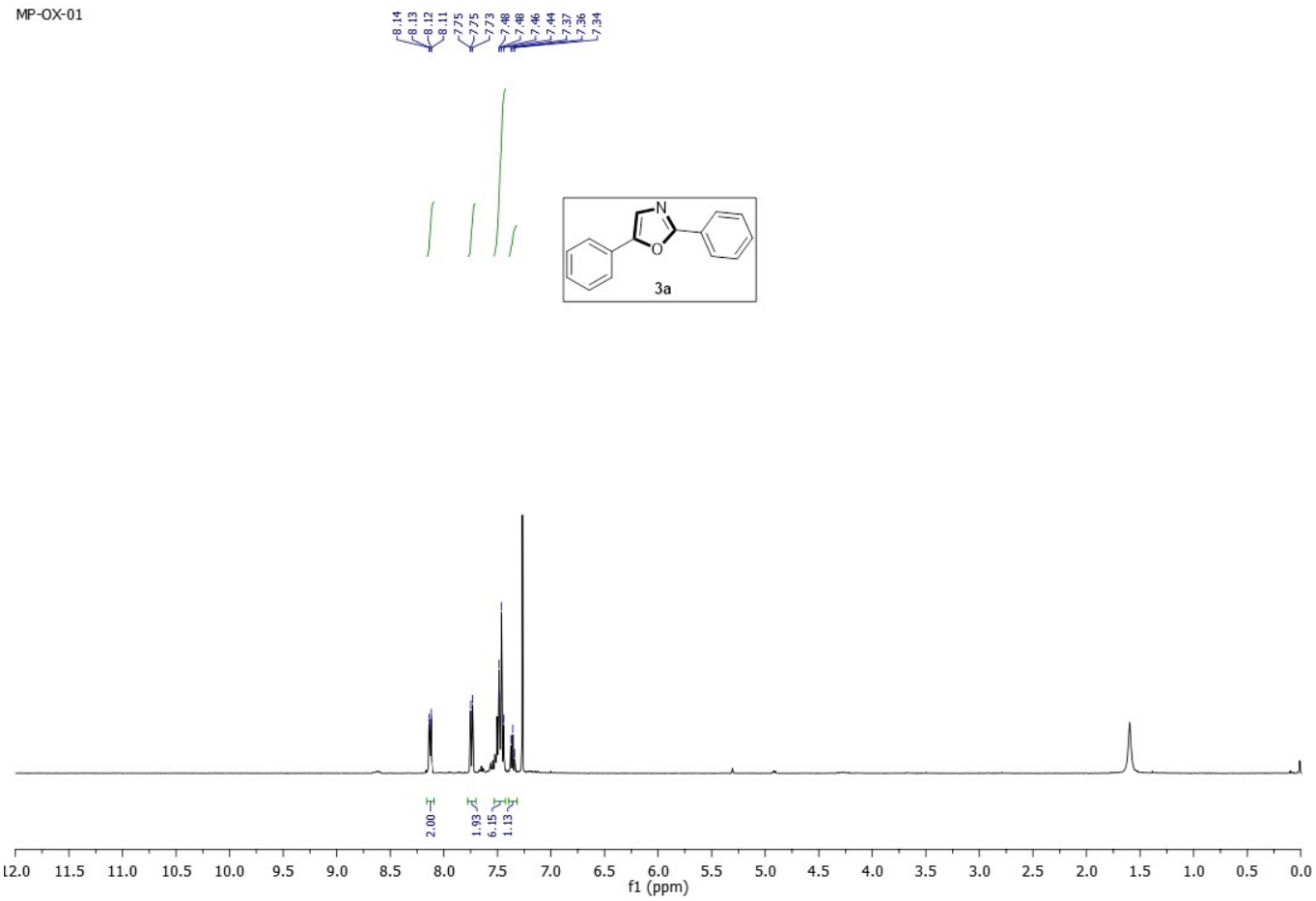
2-(4-methoxyphenyl)-5-phenyloxazole (3s)[4]

^1H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 7.9 Hz, 2H), 7.70 (d, *J* = 7.3 Hz, 2H), 7.50 – 7.29 (m, 4H), 7.00 (d, *J* = 7.9 Hz, 2H), 3.87 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.39, 161.30, 150.75, 128.91, 128.22, 127.98, 124.07, 123.29, 120.32, 114.27, 55.41.

4. References

1. S. Samanta, R. R. Donthiri, M. Dinda and S. Adimurthy, *RSC Adv.,* 2015, **5**, 66718.
2. X. Yang, X. Guo, M. Qin, X. Yuan, H. Jing and B. Chen, *Org. Biomol. Chem*., 2018, **16**, 3104.
3. H. Jiang, H. Huang, H. Cao and C. Qi, *Org. Lett*., 2010, **12**, 5561.
4. C. W. Cheung and S. L. Buchwald, *J. Org. Chem*., 2012, **77**, 7526.

4. Copies of ^{1}H and ^{13}C NMR Spectra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7.36
7.34
N
O
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6.15
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6.0
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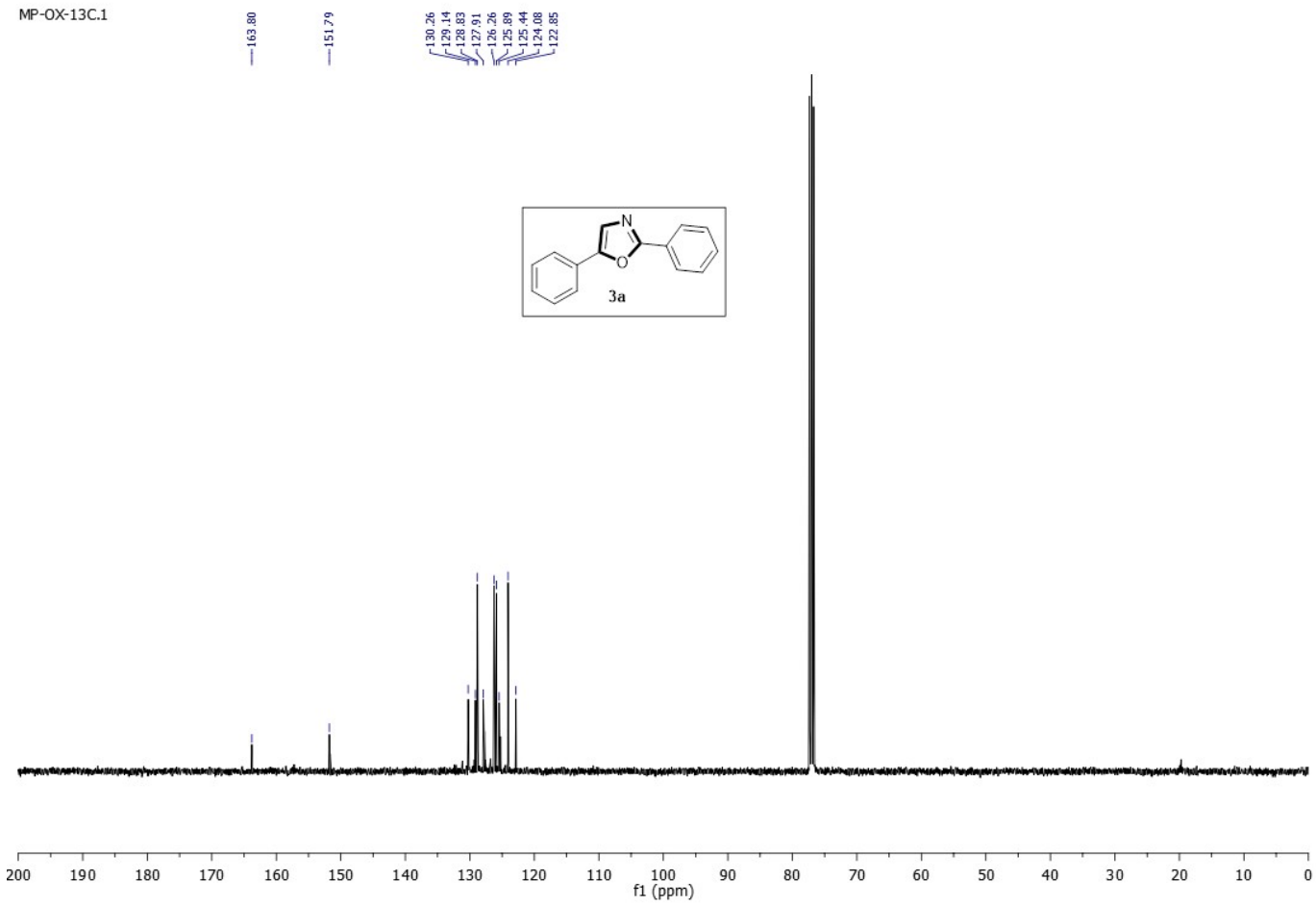
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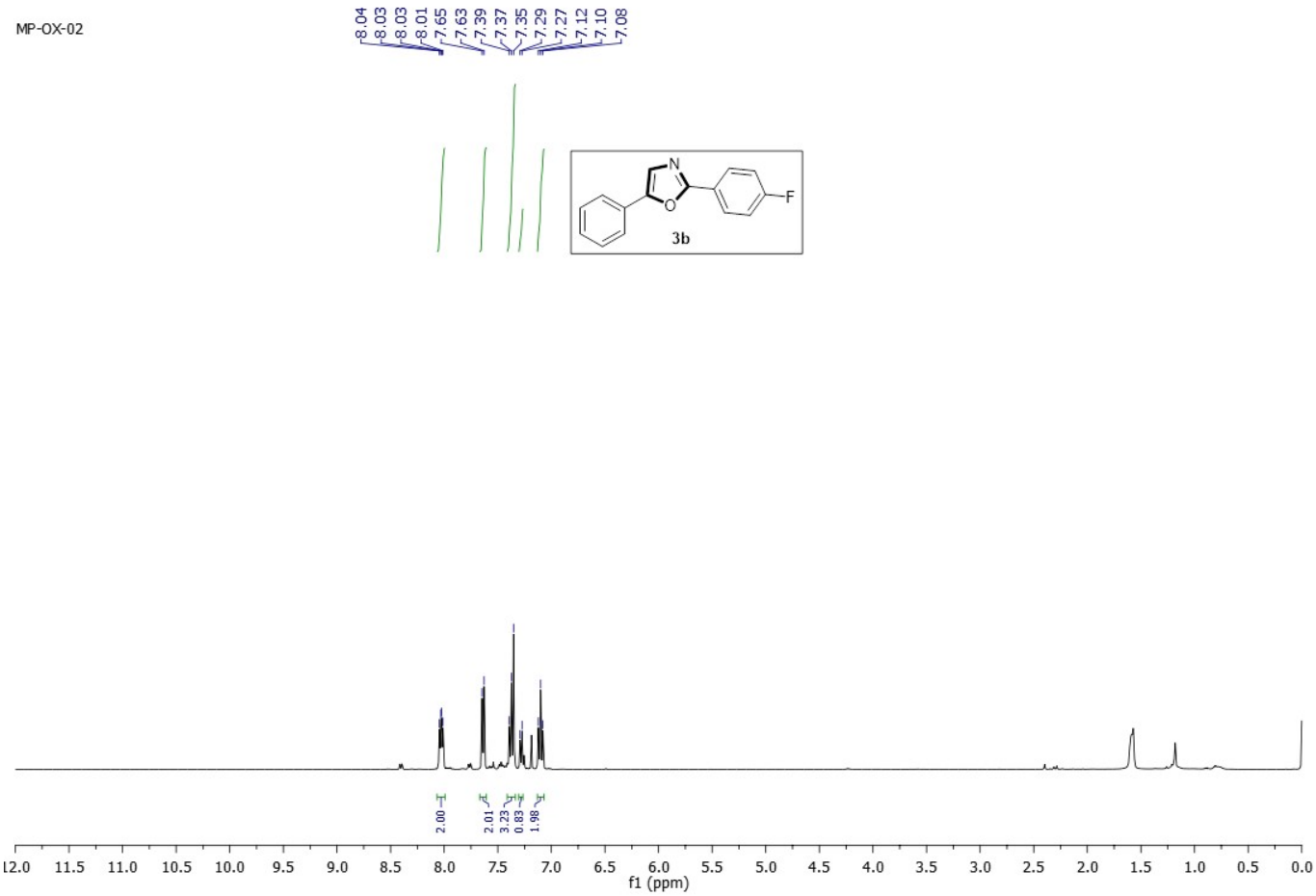
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7.10
7.08
N
O
F
3b
2.00
2.01
3.23
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1.98
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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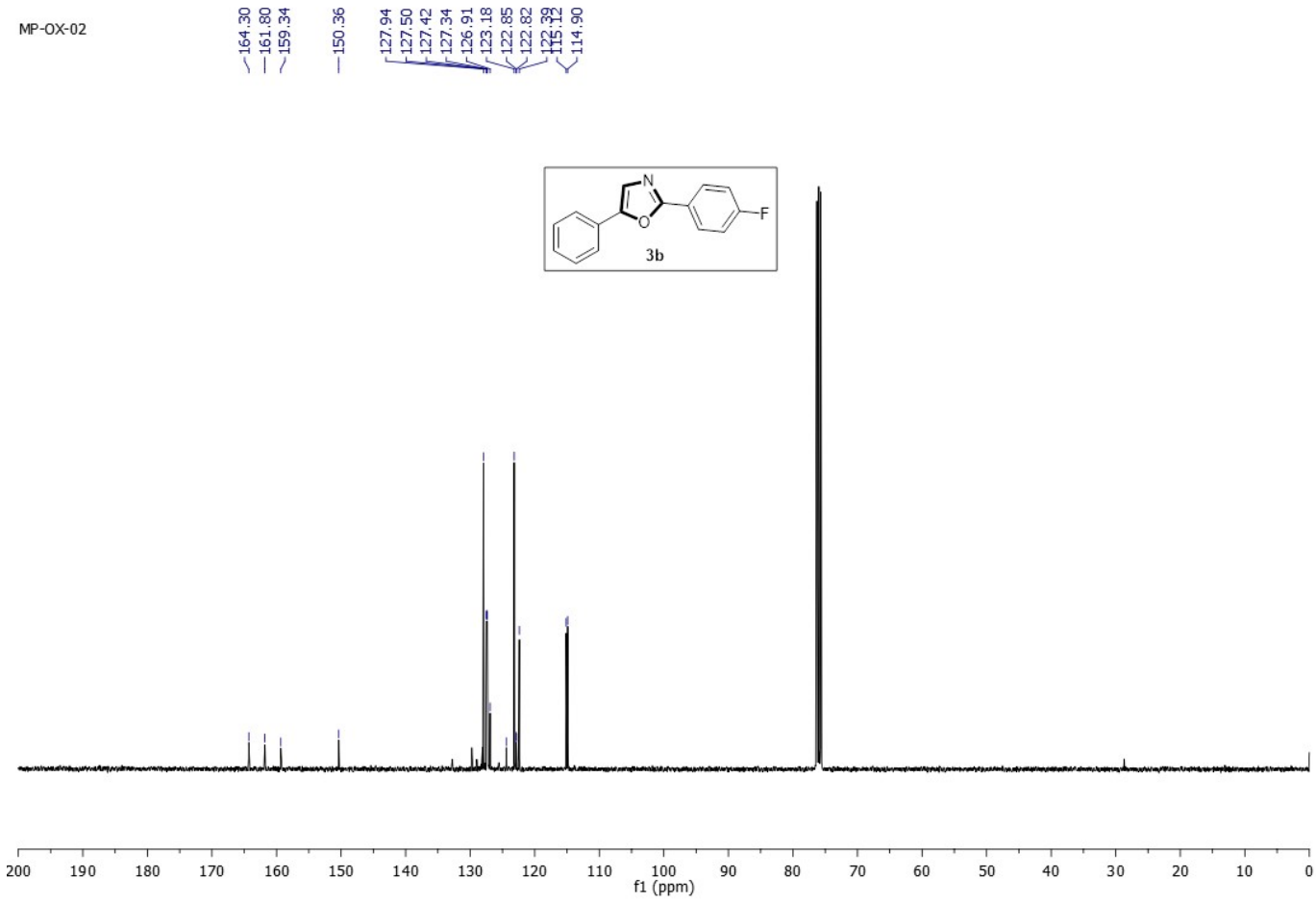
f1 (ppm)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190
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20
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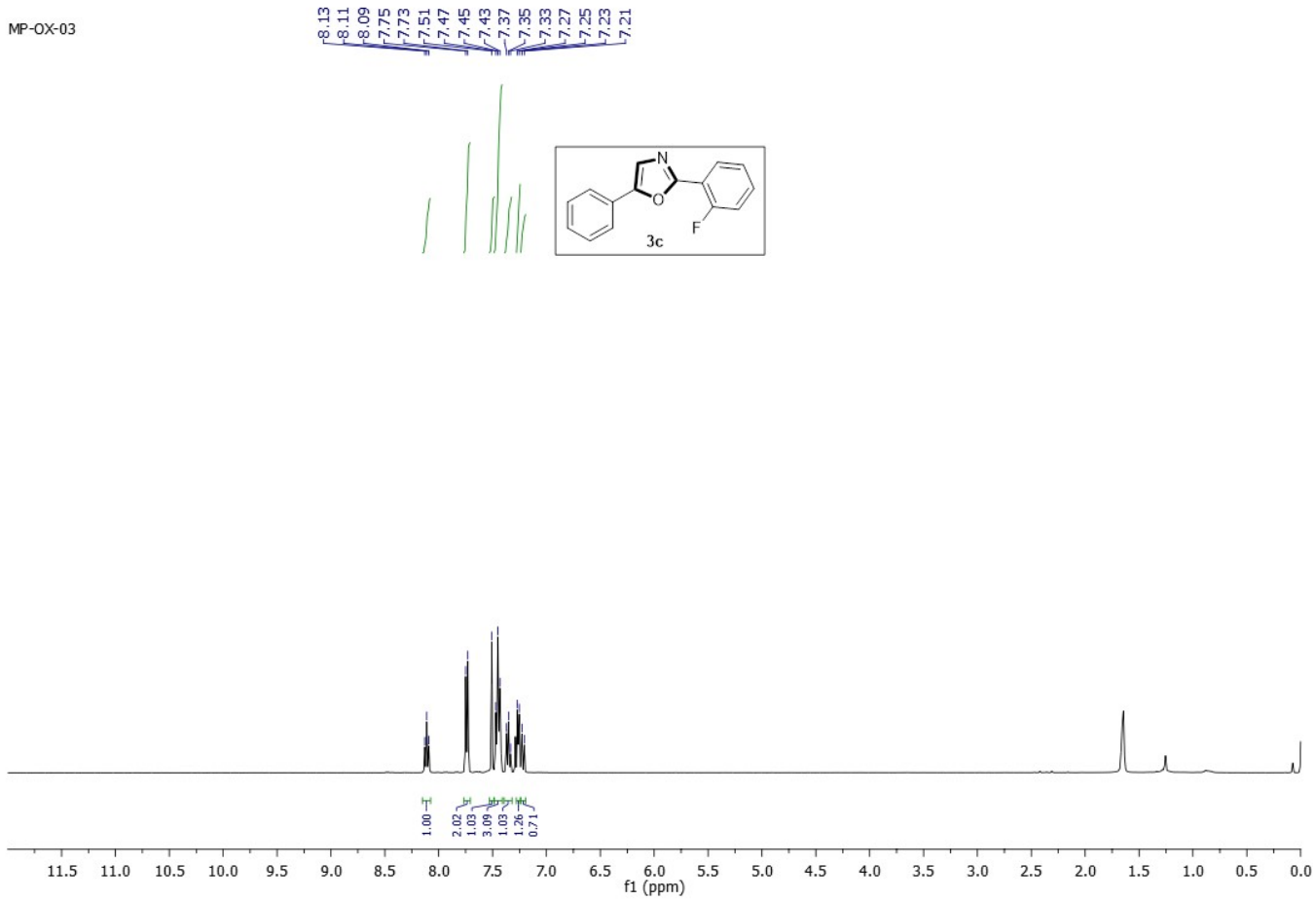
f1 (ppm)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7.25
7.23
7.21
N
O
F
3c
1.00
2.02
1.03
3.09
1.03
1.26
0.71
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

MP-OX-03

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158.78
157.55
157.51
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151.54
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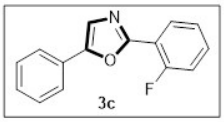
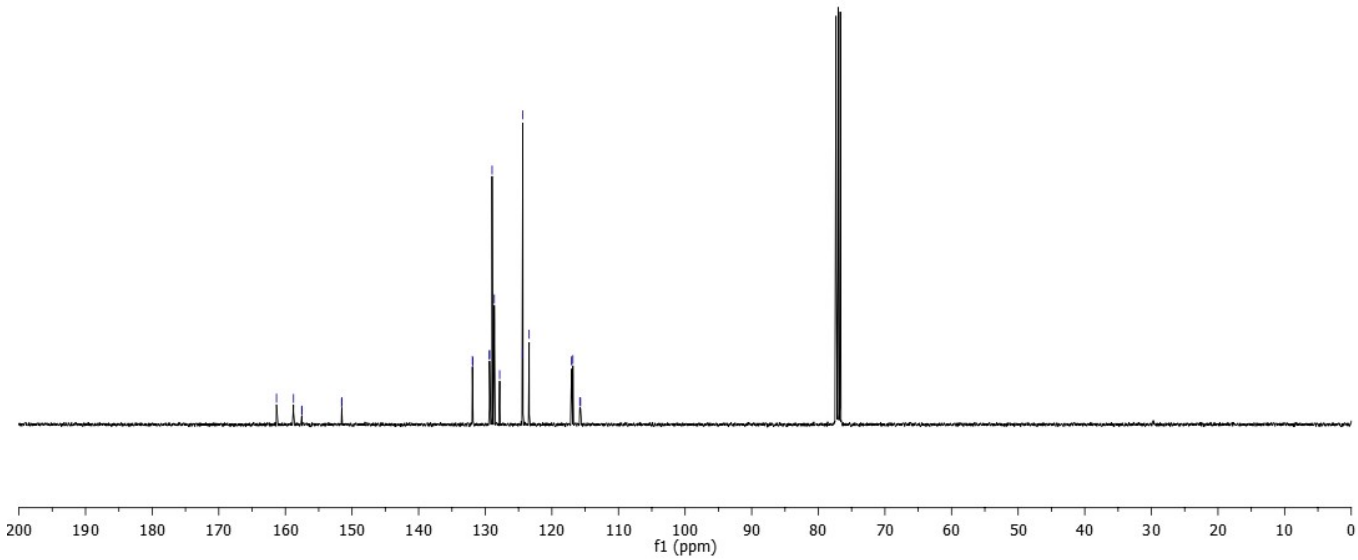
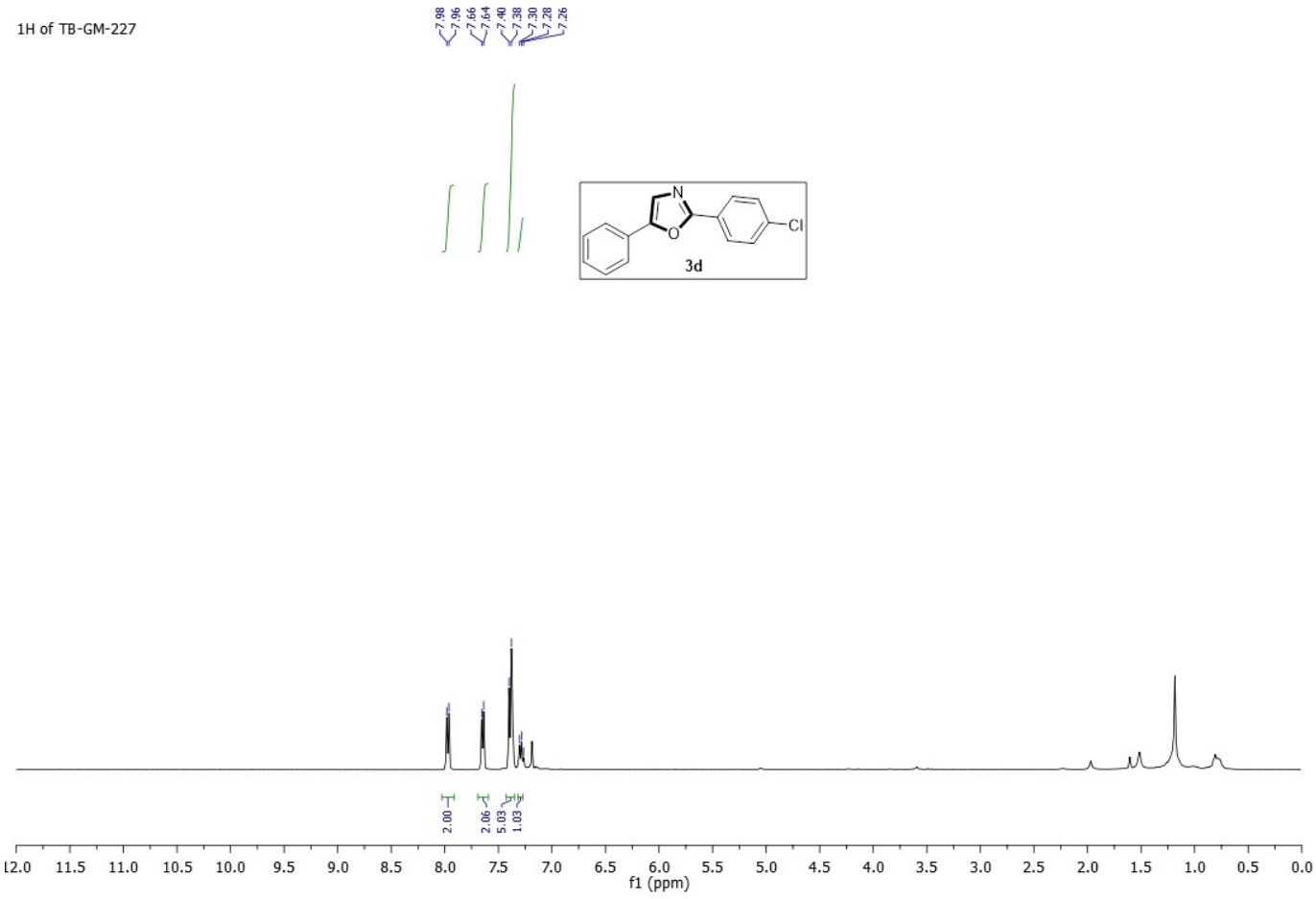
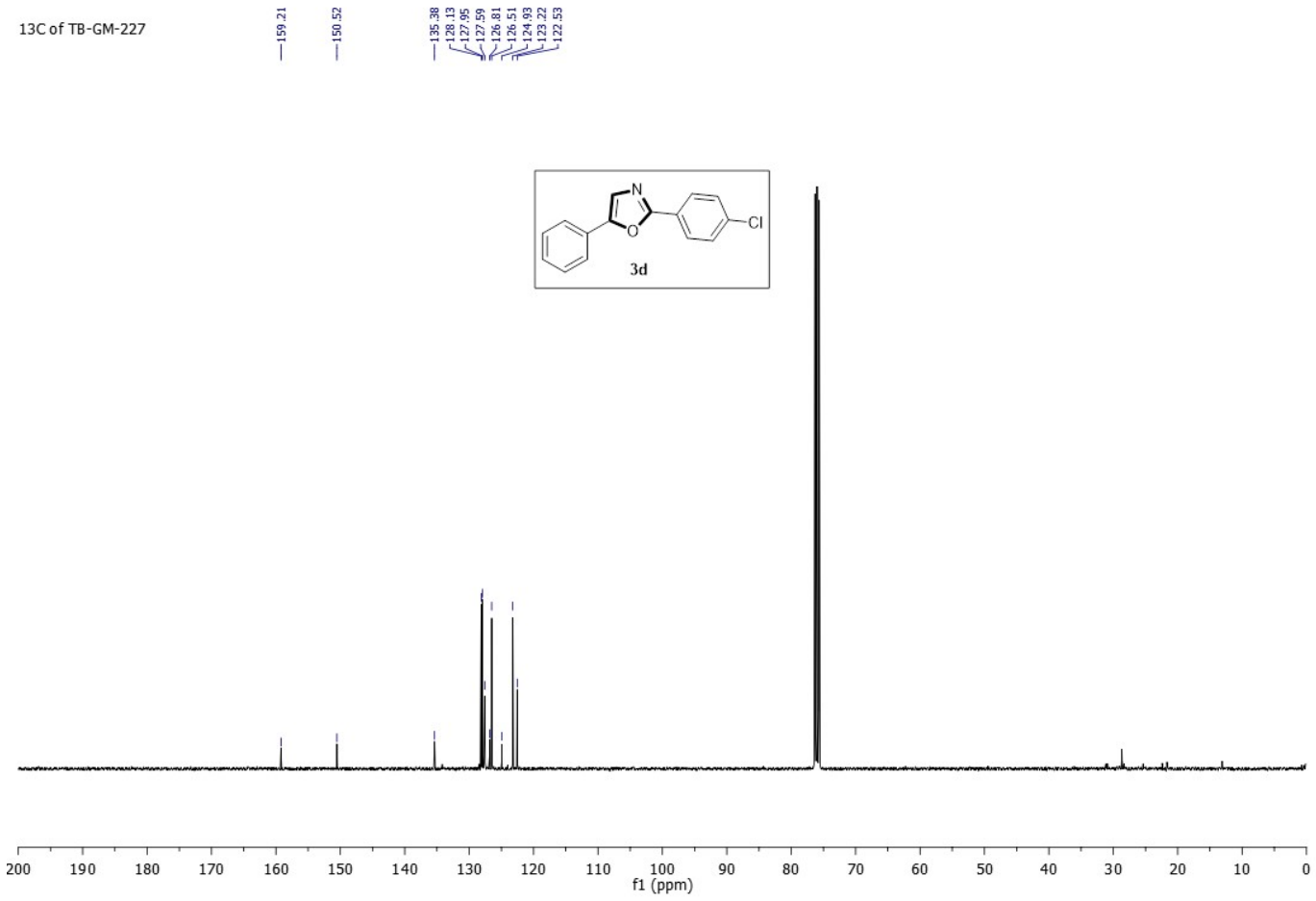
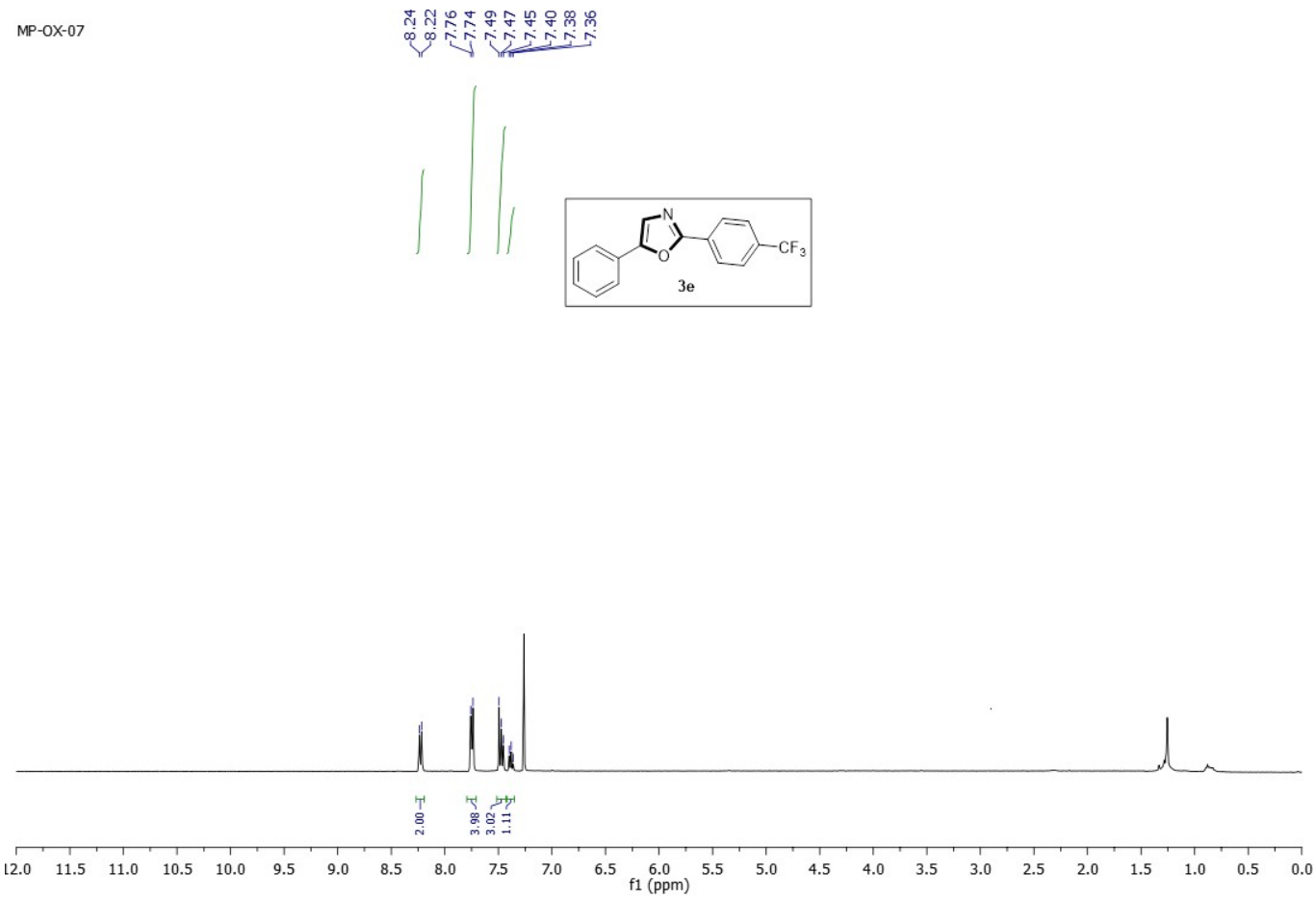


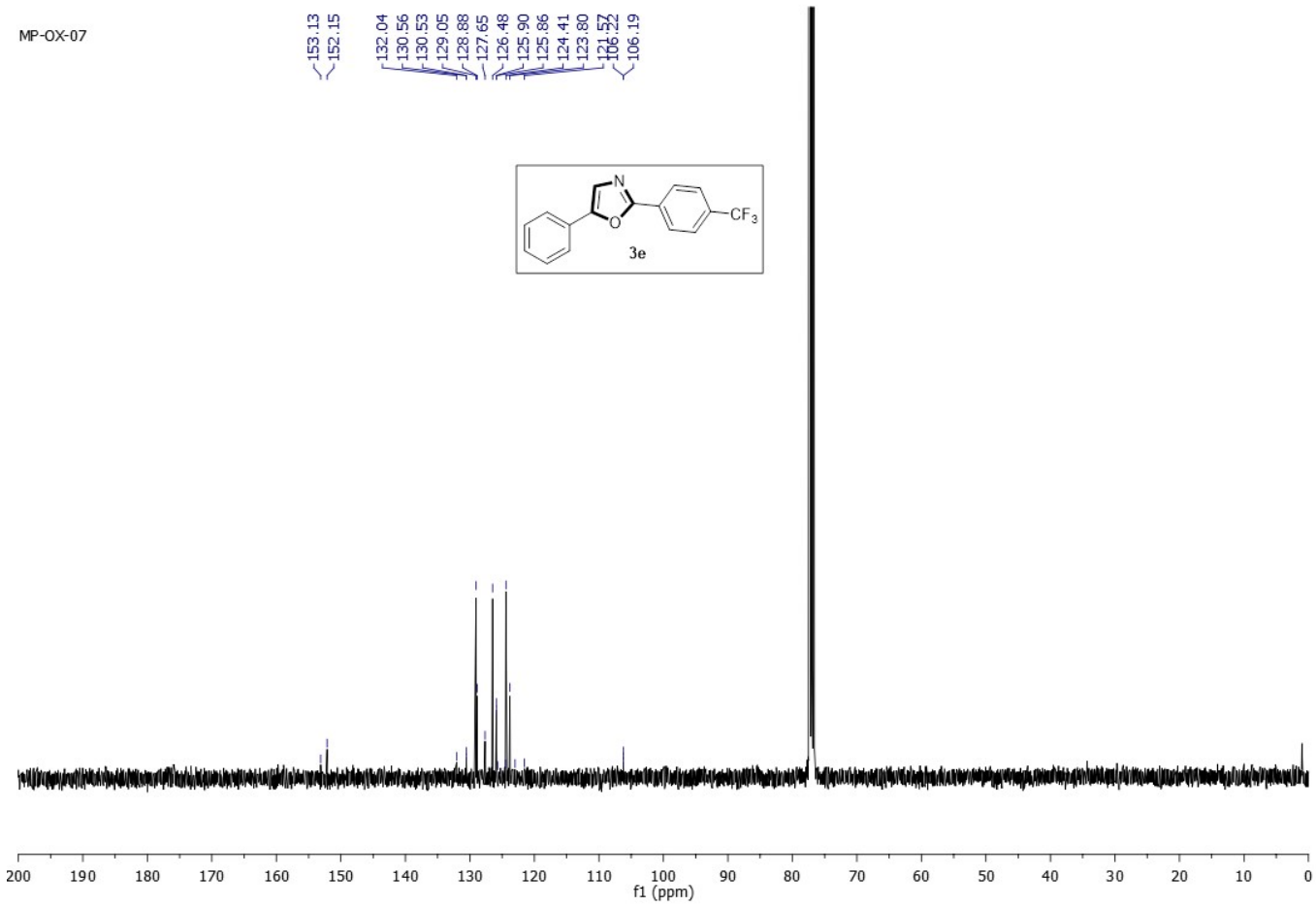
3c

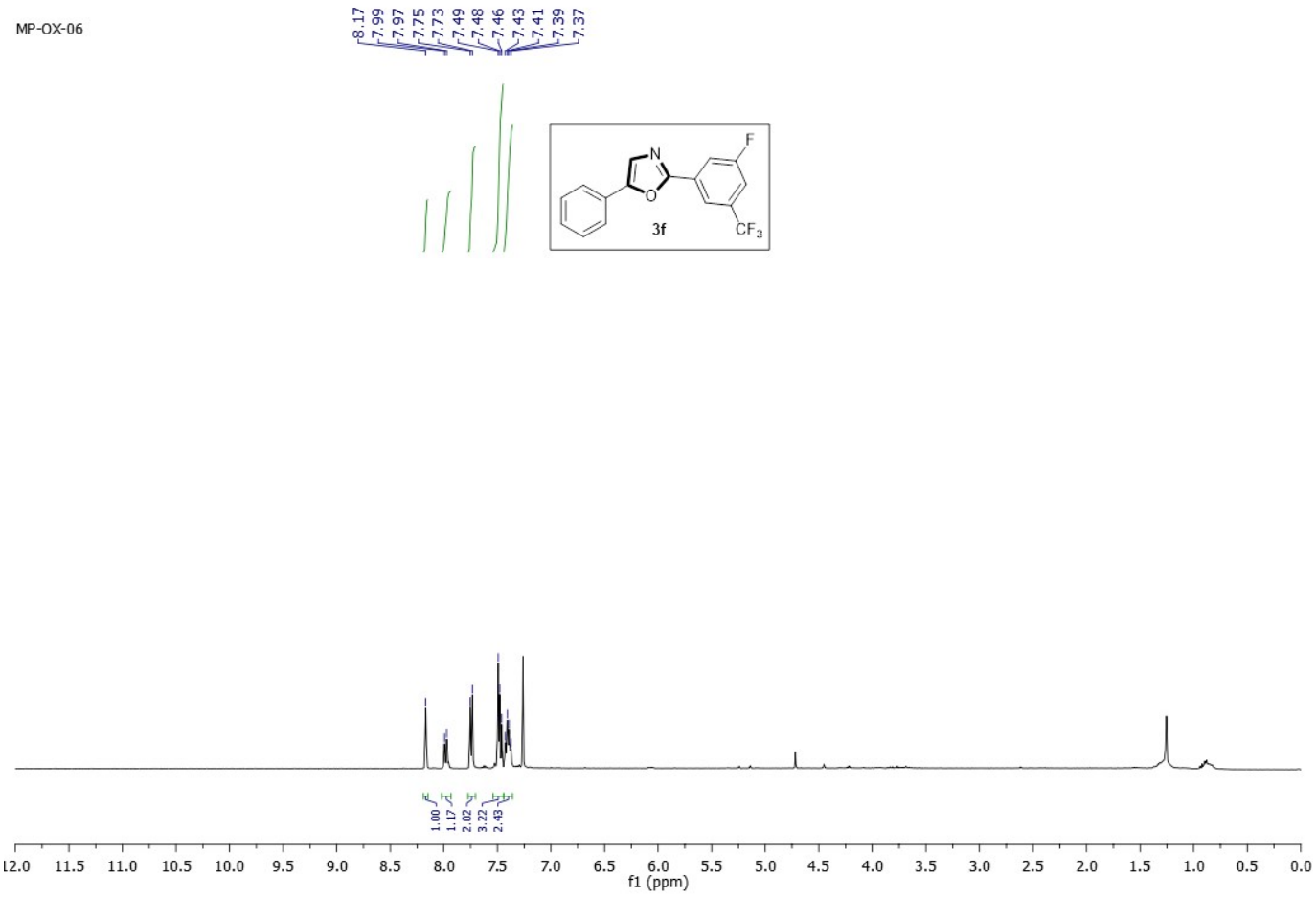


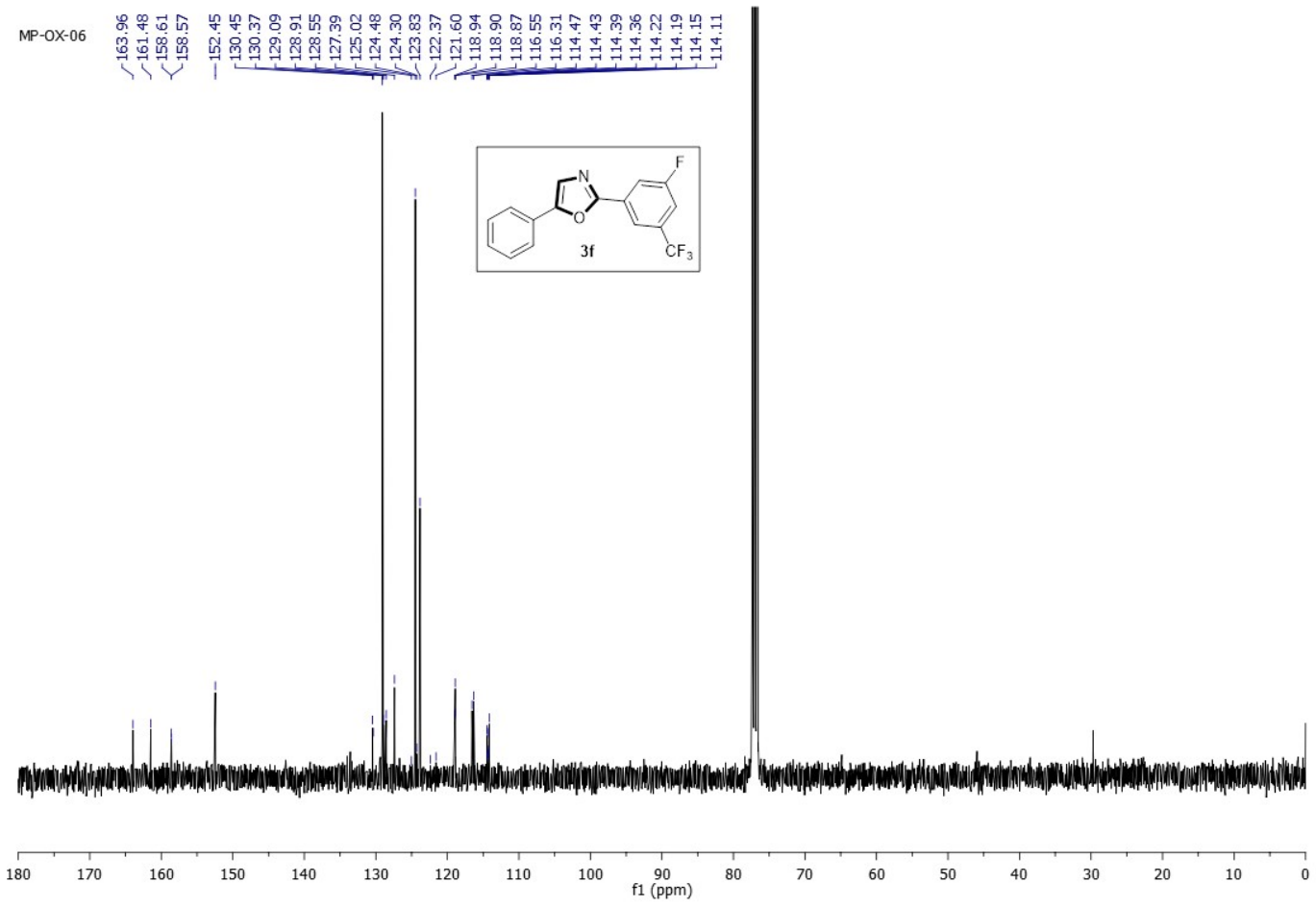

1H of TB-GM-227
7.98
7.96
7.66
7.64
7.40
7.38
7.30
7.28
7.26
N
O
Cl
3d
2.00
2.06
5.03
1.03
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


13C of TB-GM-227
159.21
150.52
135.38
128.13
127.95
127.59
126.81
126.51
124.93
123.22
122.53
N
O
Cl
3d
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

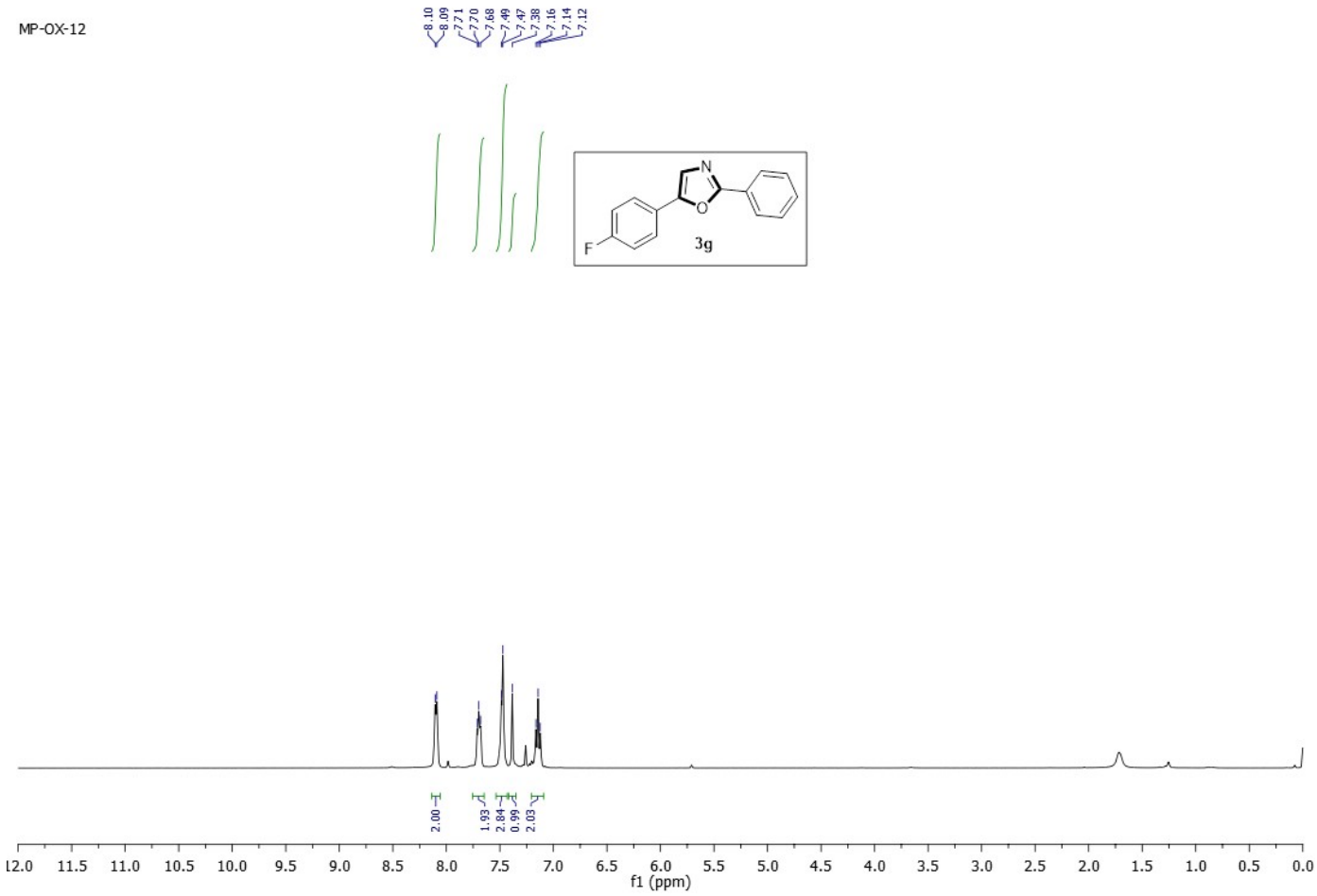

MP-OX-07
8.24
8.22
7.76
7.74
7.49
7.47
7.45
7.40
7.38
7.36
N
O
CF3
3e
2.00
3.98
3.02
1.11
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

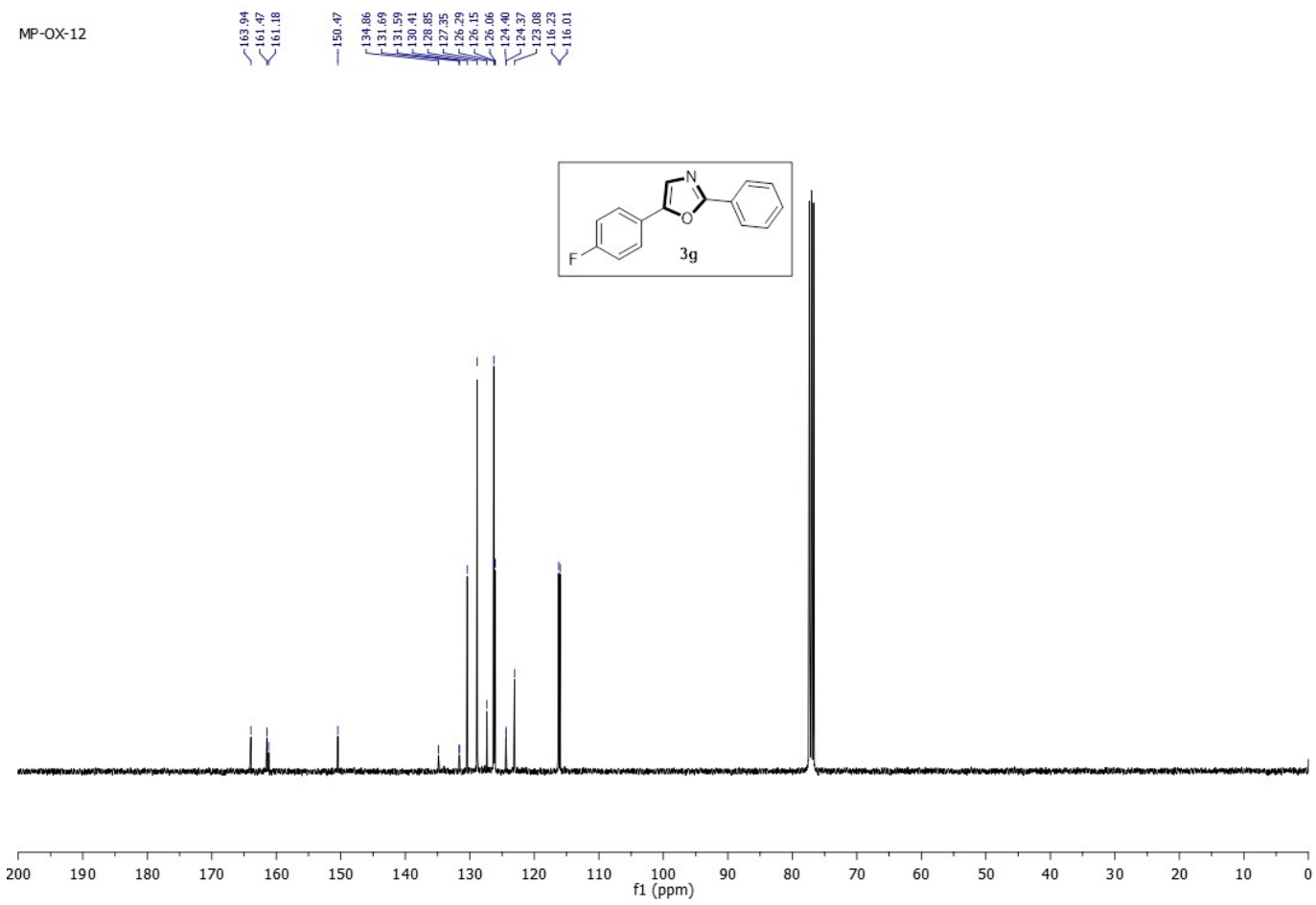

MP-OX-07
153.13
152.15
132.04
130.56
130.53
129.05
128.88
127.65
126.48
125.90
125.86
124.41
123.80
121.57
106.22
106.19
N
O
CF3
3e
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

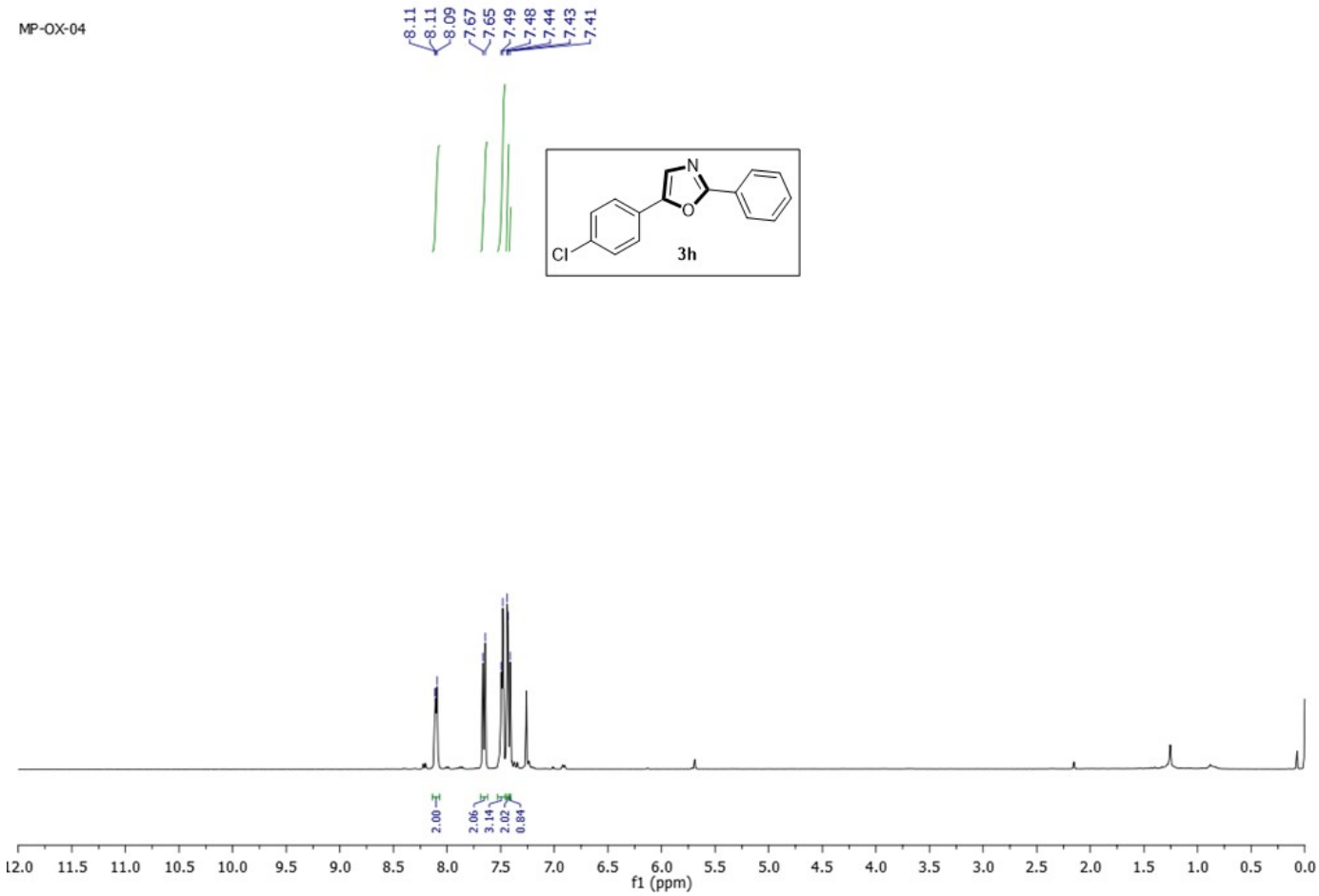

MP-OX-06
8.17
7.99
7.97
7.75
7.73
7.49
7.48
7.46
7.43
7.41
7.39
7.37
N
O
F
CF3
3f
1.00
1.17
2.02
3.22
2.43
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

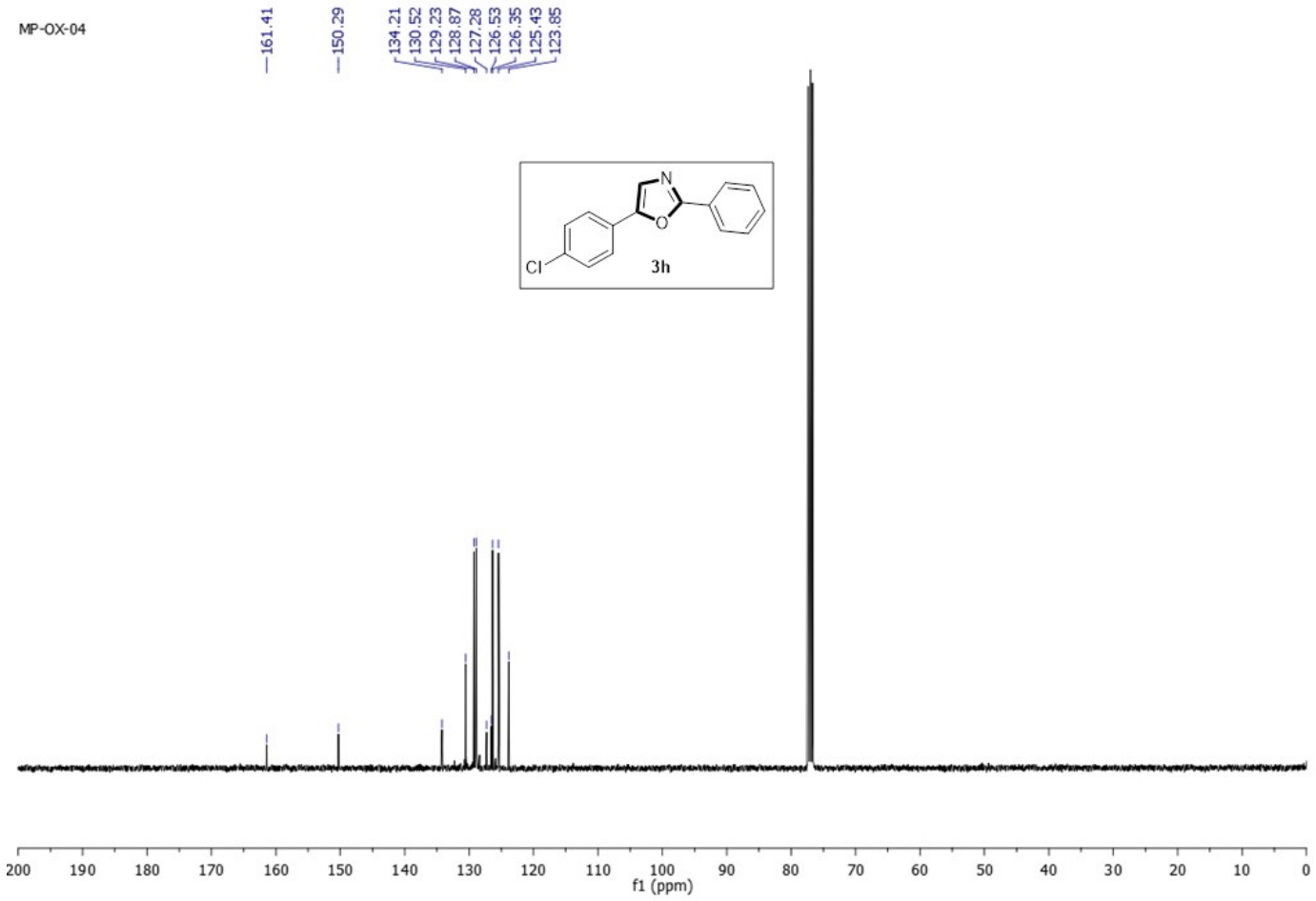

MP-OX-06
163.96
161.48
158.61
158.57
152.45
130.45
130.37
129.09
128.91
128.55
127.39
125.02
124.48
124.30
123.83
122.37
121.60
118.94
118.90
118.87
116.55
116.31
114.47
114.43
114.39
114.36
114.22
114.19
114.15
114.11
N
O
F
CF3
3f
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

MP-OX-12

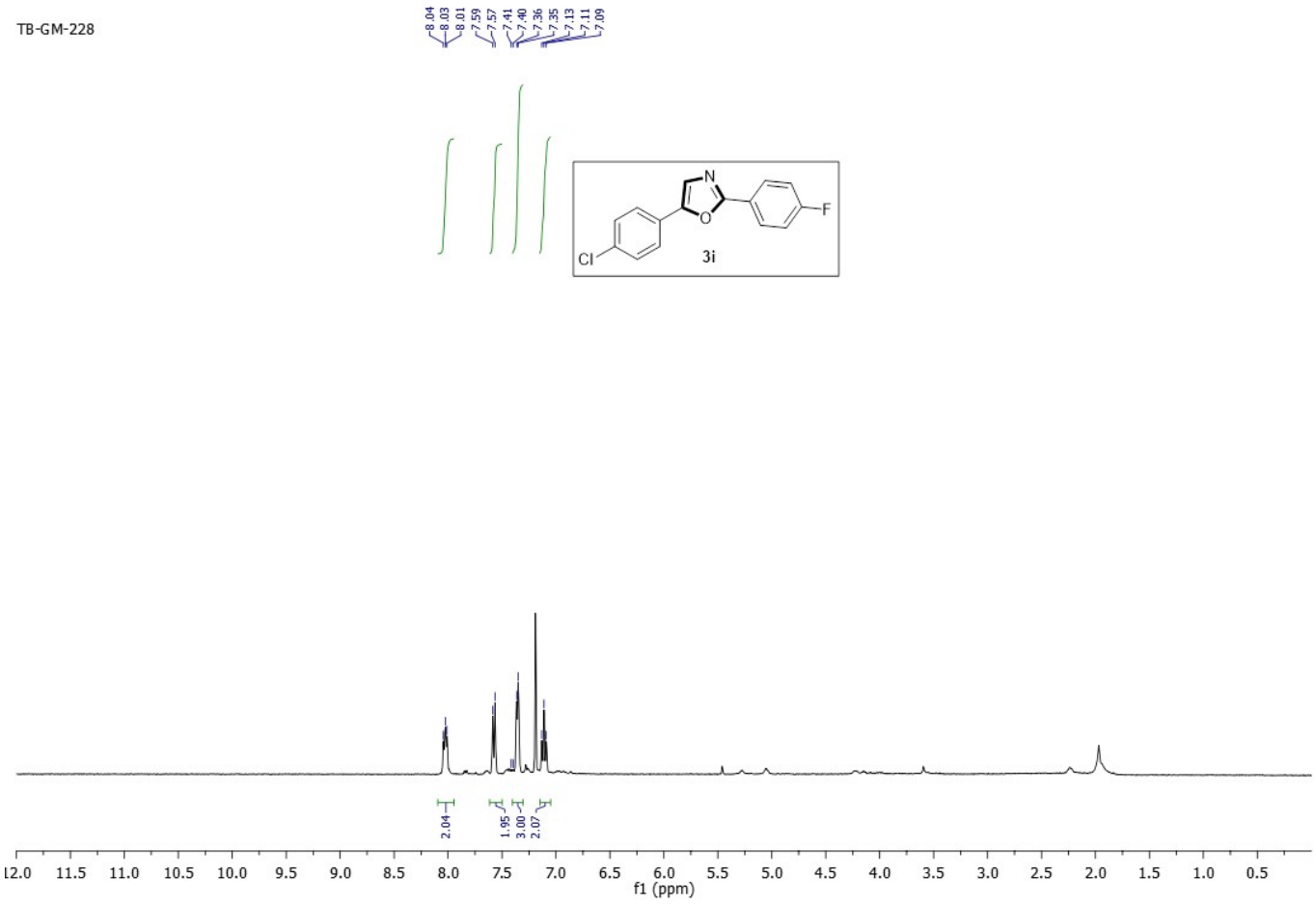

8.10
8.09
7.71
7.70
7.68
7.49
7.47
7.38
7.16
7.14
7.12
N
O
F
3g
2.00
1.93
2.84
0.99
2.03
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

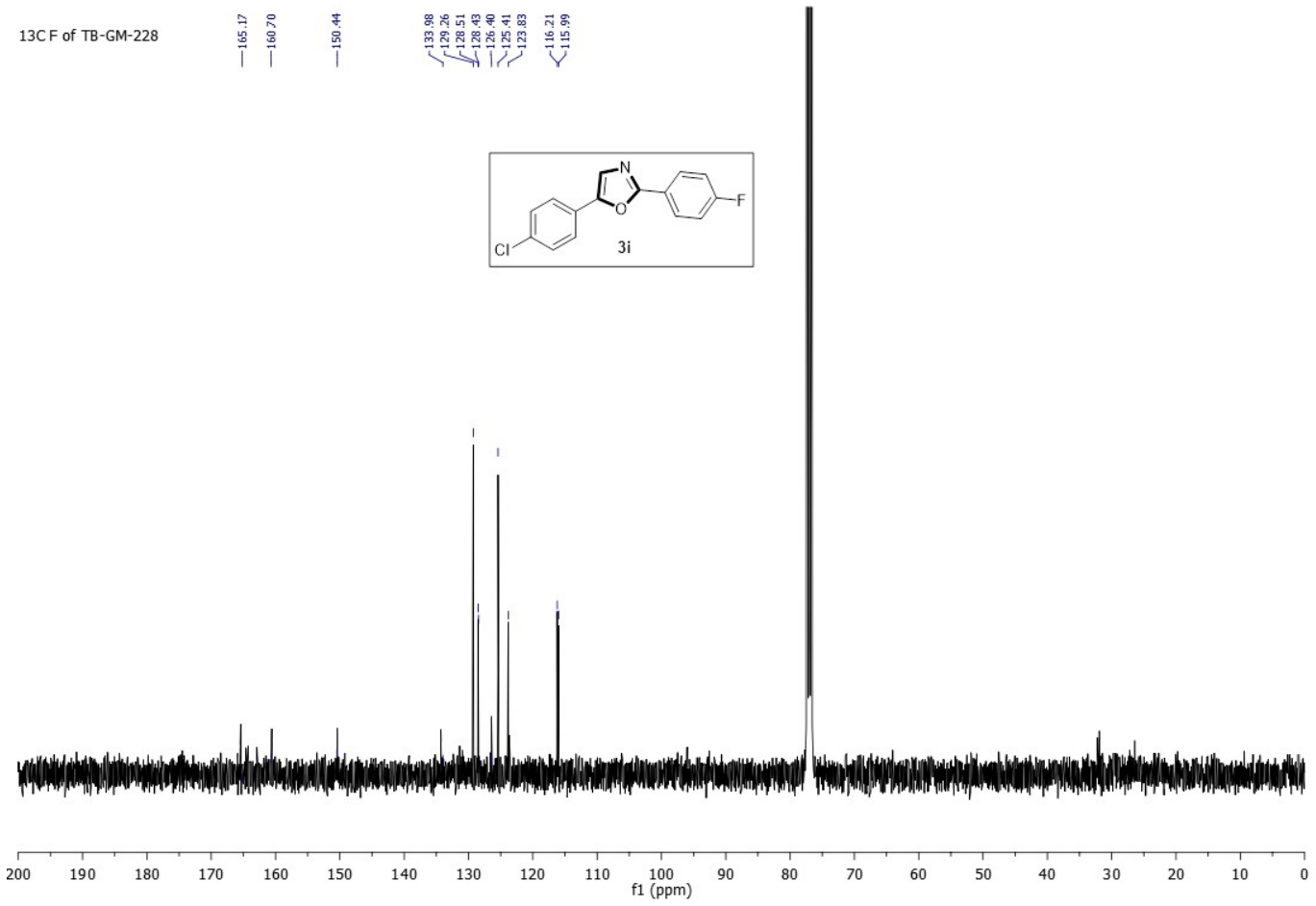

MP-OX-12
163.94
161.47
161.18
150.47
134.86
131.69
131.59
130.41
128.85
127.35
126.29
126.15
126.06
124.40
124.37
123.08
116.23
116.01
N
O
F
3g
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


MP-OX-04
8.11
8.11
8.09
7.67
7.65
7.49
7.48
7.44
7.43
7.41
N
O
Cl
3h
2.00
2.06
3.14
2.02
0.84
12.0
11.5
11.0
10.5
10.0
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
f1 (ppm)

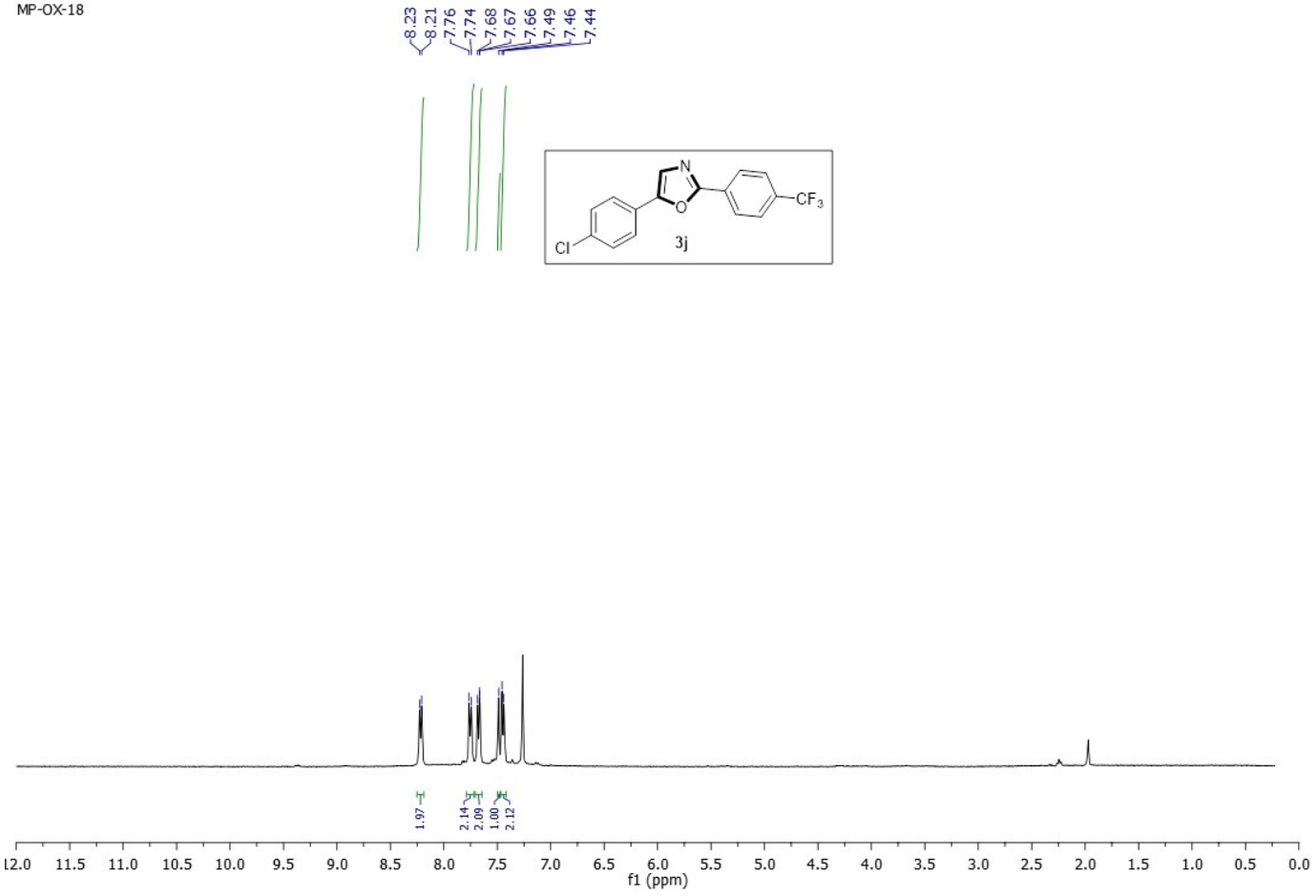

MP-OX-04
161.41
150.29
134.21
130.52
129.23
128.87
127.28
126.53
126.35
125.43
123.85
N
O
Cl
3h
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

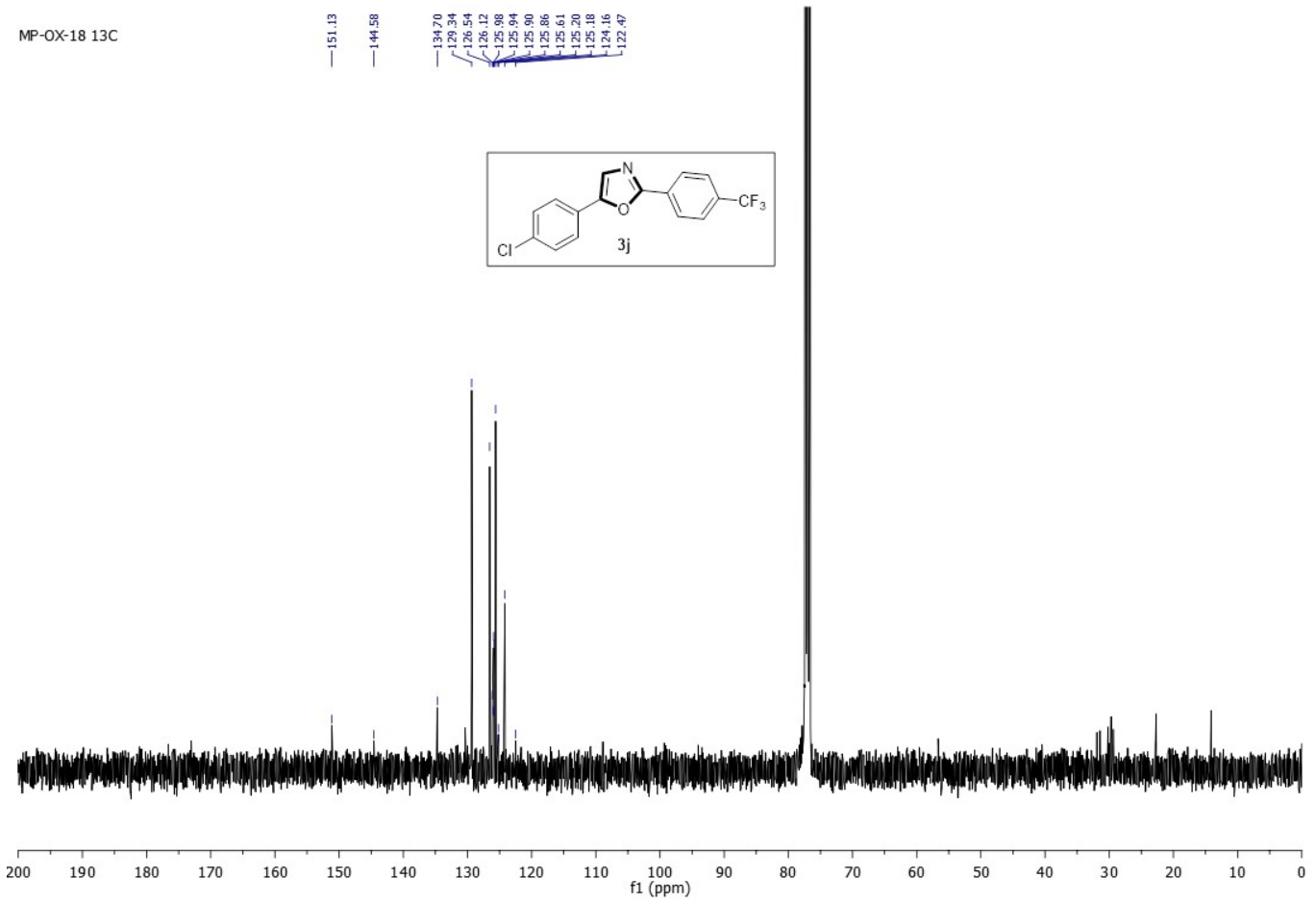
TB-GM-228

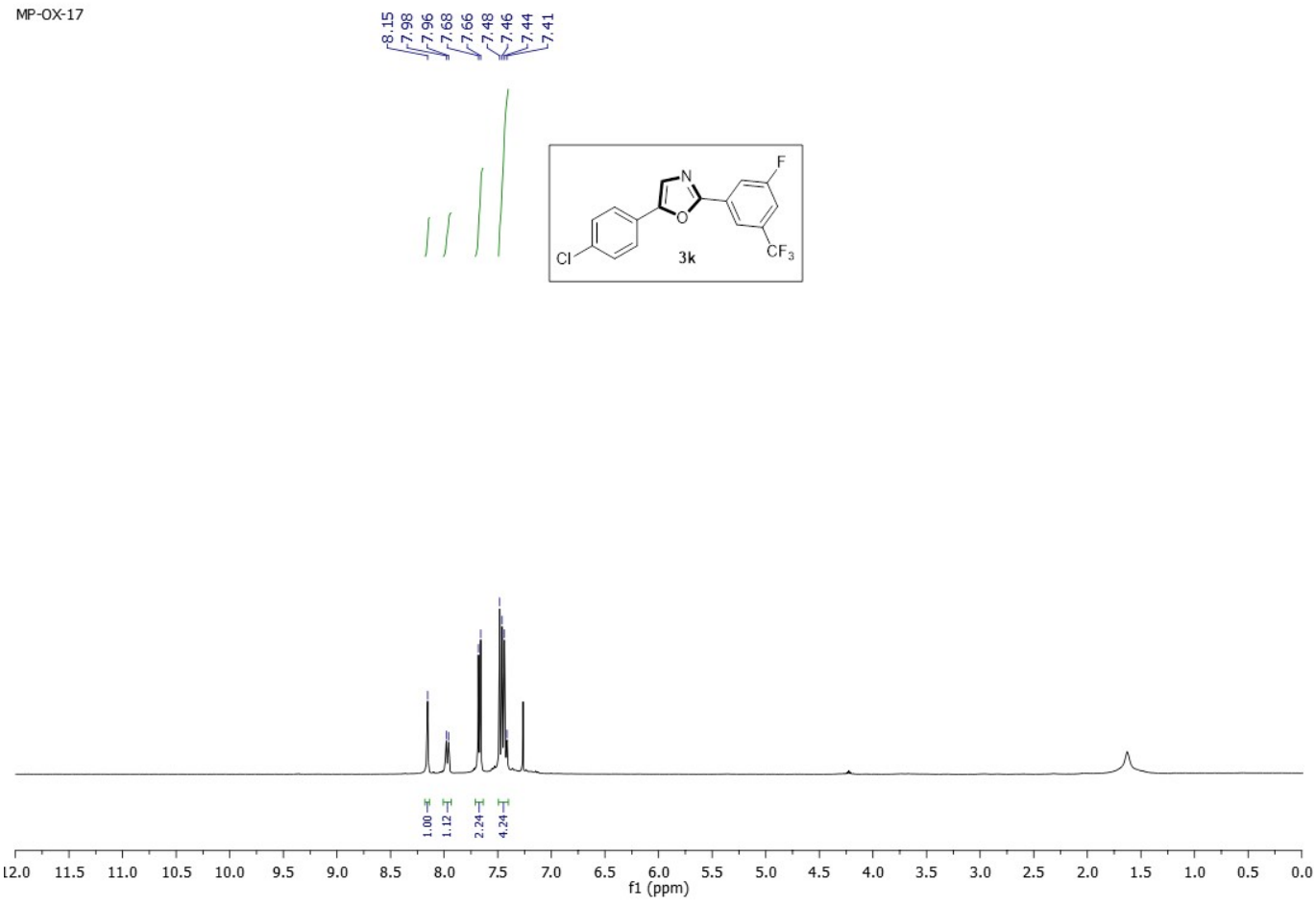

8.04
8.03
8.01
7.59
7.57
7.41
7.40
7.36
7.35
7.13
7.11
7.09
N
O
F
Cl
3i
2.04
1.95
3.00
2.07
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

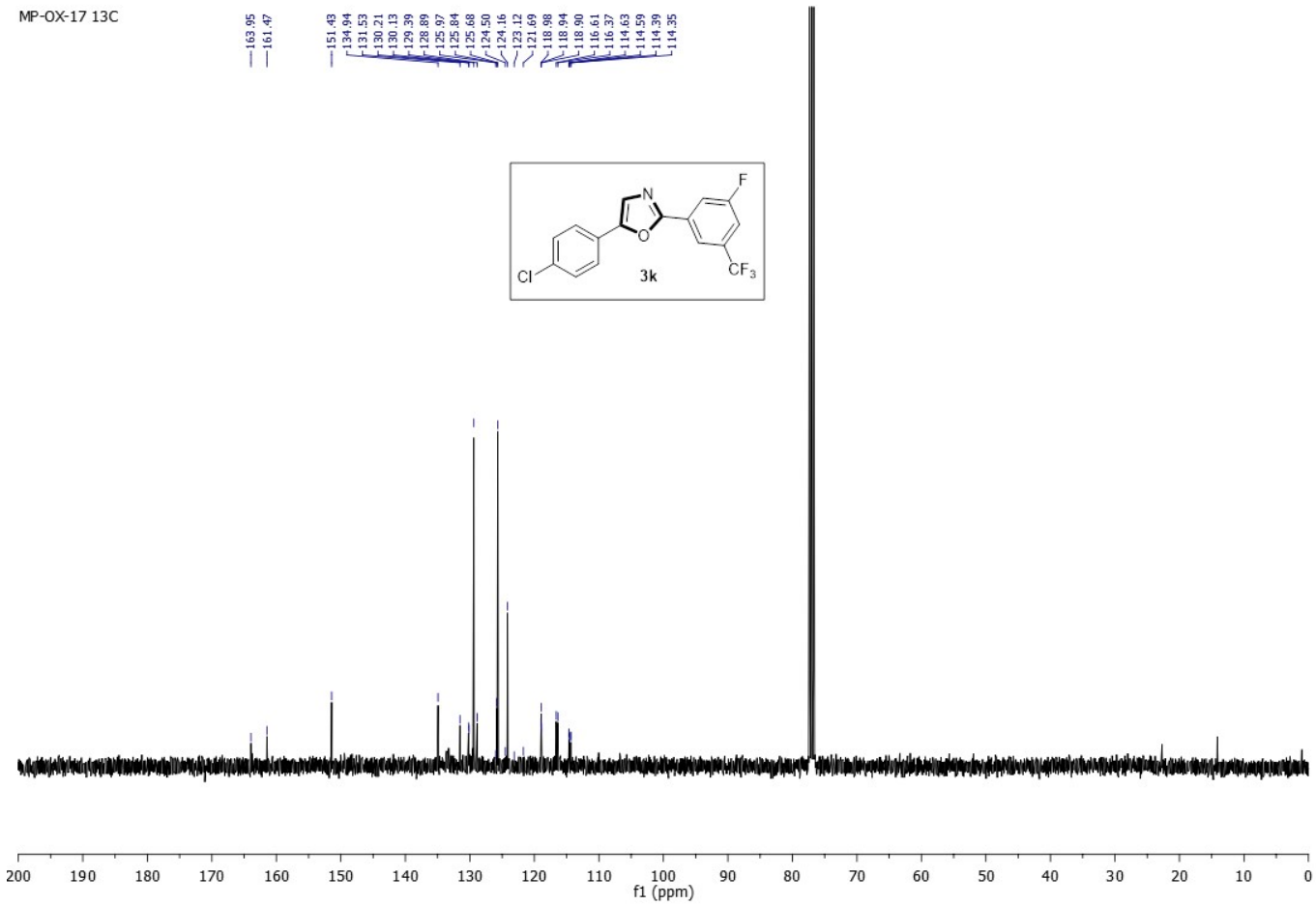

13C F of TB-GM-228
165.17
160.70
150.44
133.98
129.26
128.51
128.43
126.40
125.41
123.83
116.21
115.99
N
O
F
Cl
3i
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

MP-OX-18

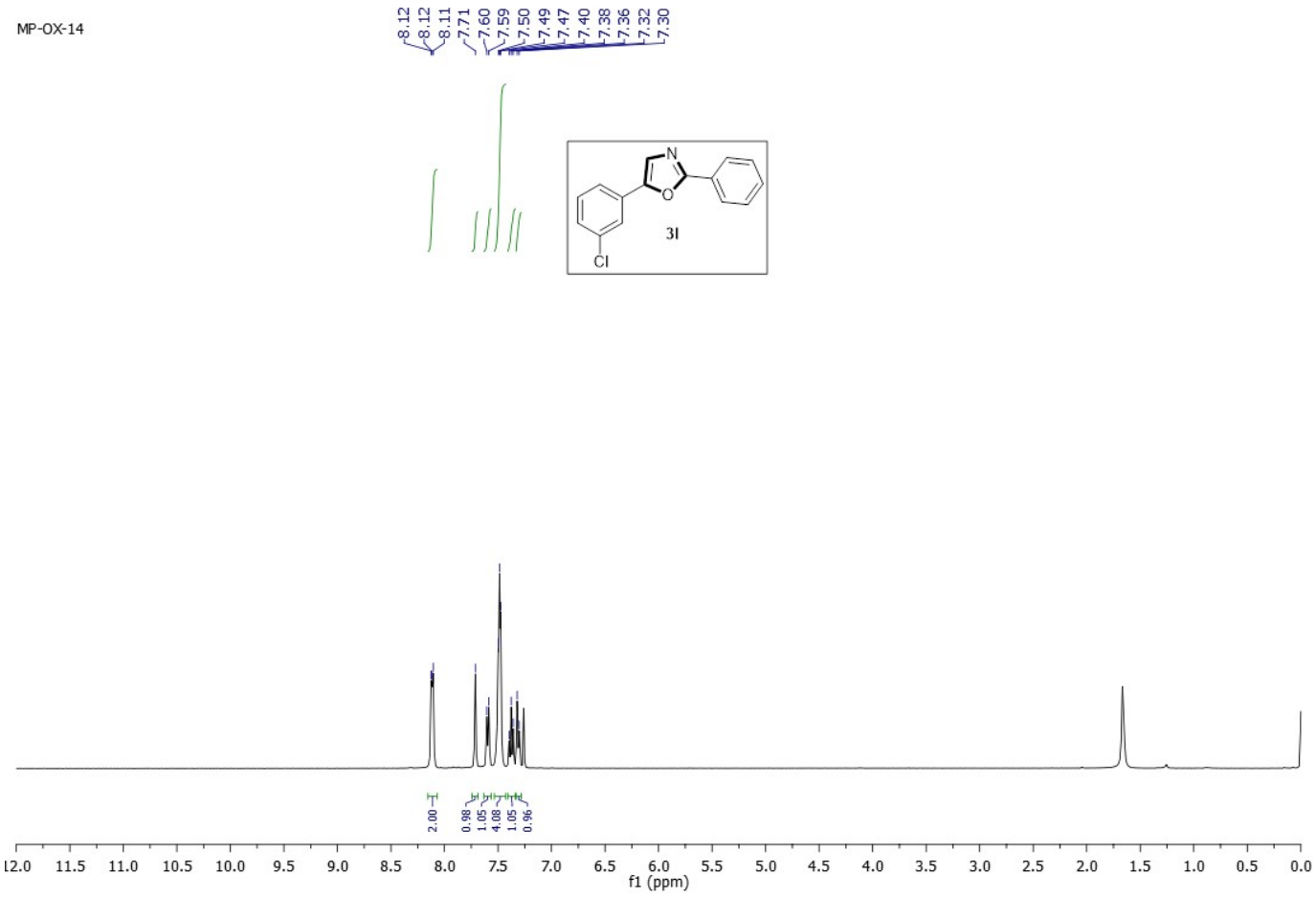

8.23
8.21
7.76
7.74
7.68
7.67
7.66
7.49
7.46
7.44
N
O
CF3
Cl
3j
1.97
2.14
2.09
1.00
2.12
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

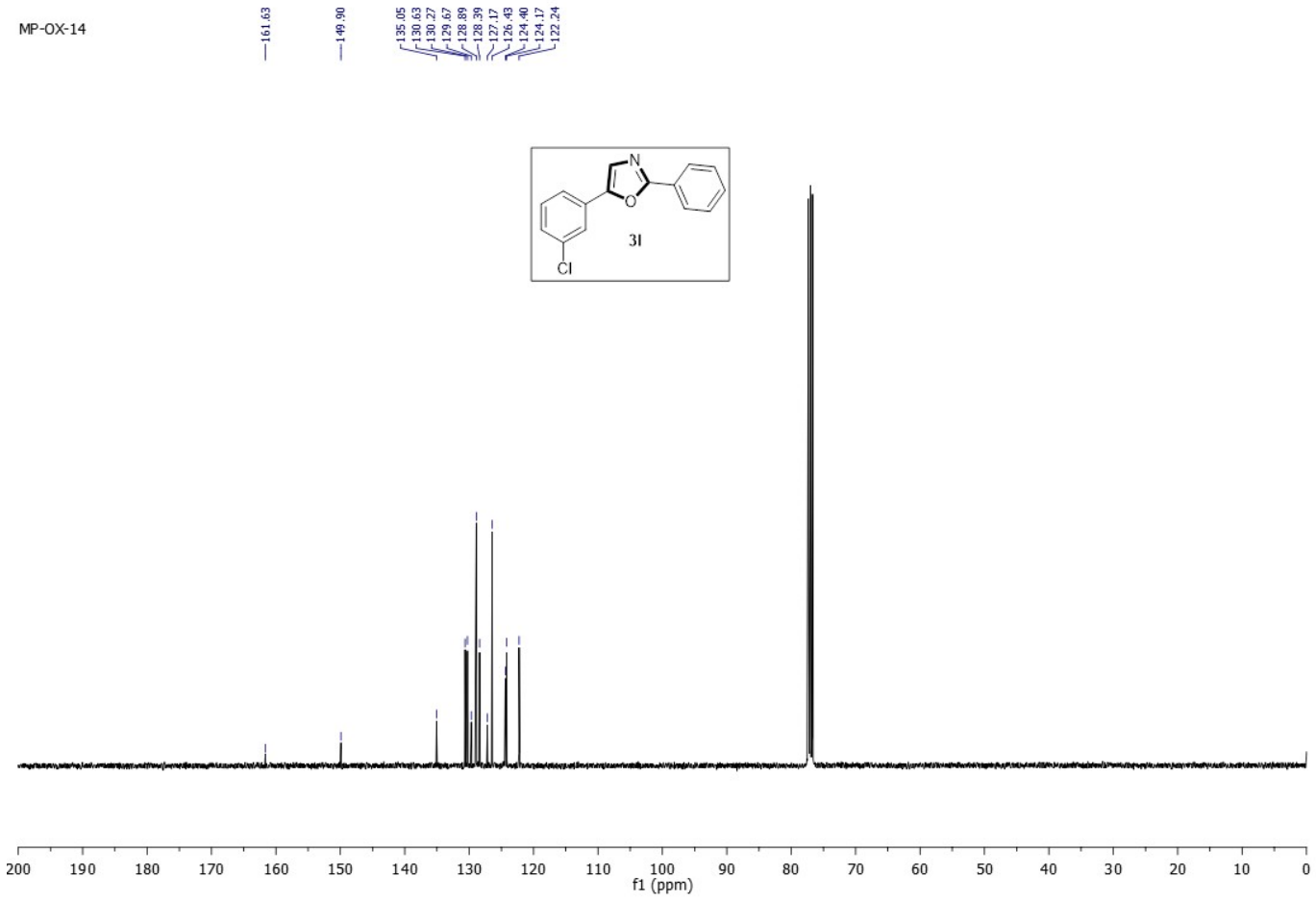

MP-OX-18 13C
151.13
144.58
134.70
129.34
126.54
126.12
125.98
125.94
125.90
125.86
125.61
125.20
125.18
124.16
122.47
N
O
CF3
Cl
3j
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

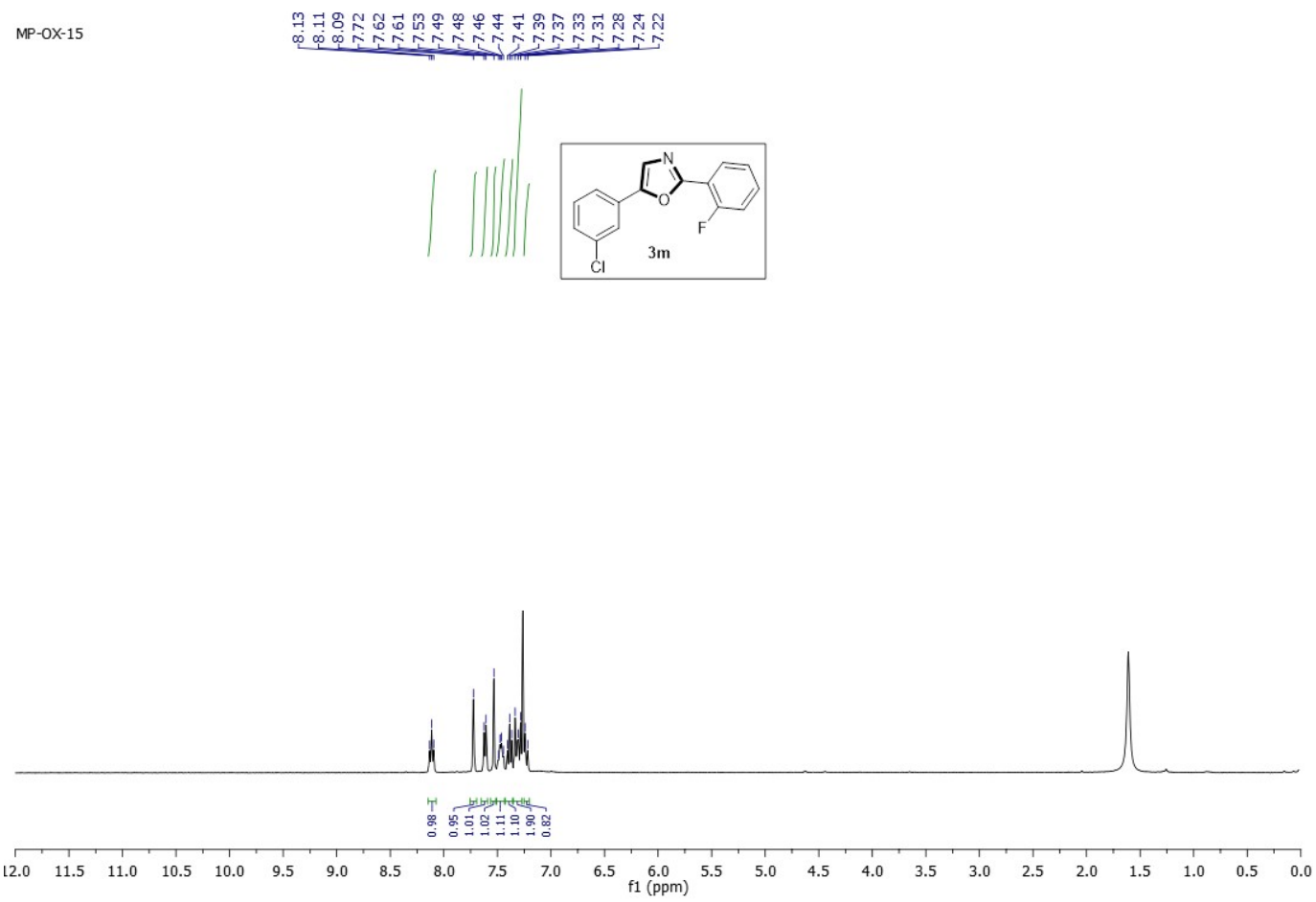

MP-OX-17
8.15
7.98
7.96
7.68
7.66
7.48
7.46
7.44
7.41
N
O
F
Cl
CF3
3k
1.00
1.12
2.24
4.24
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

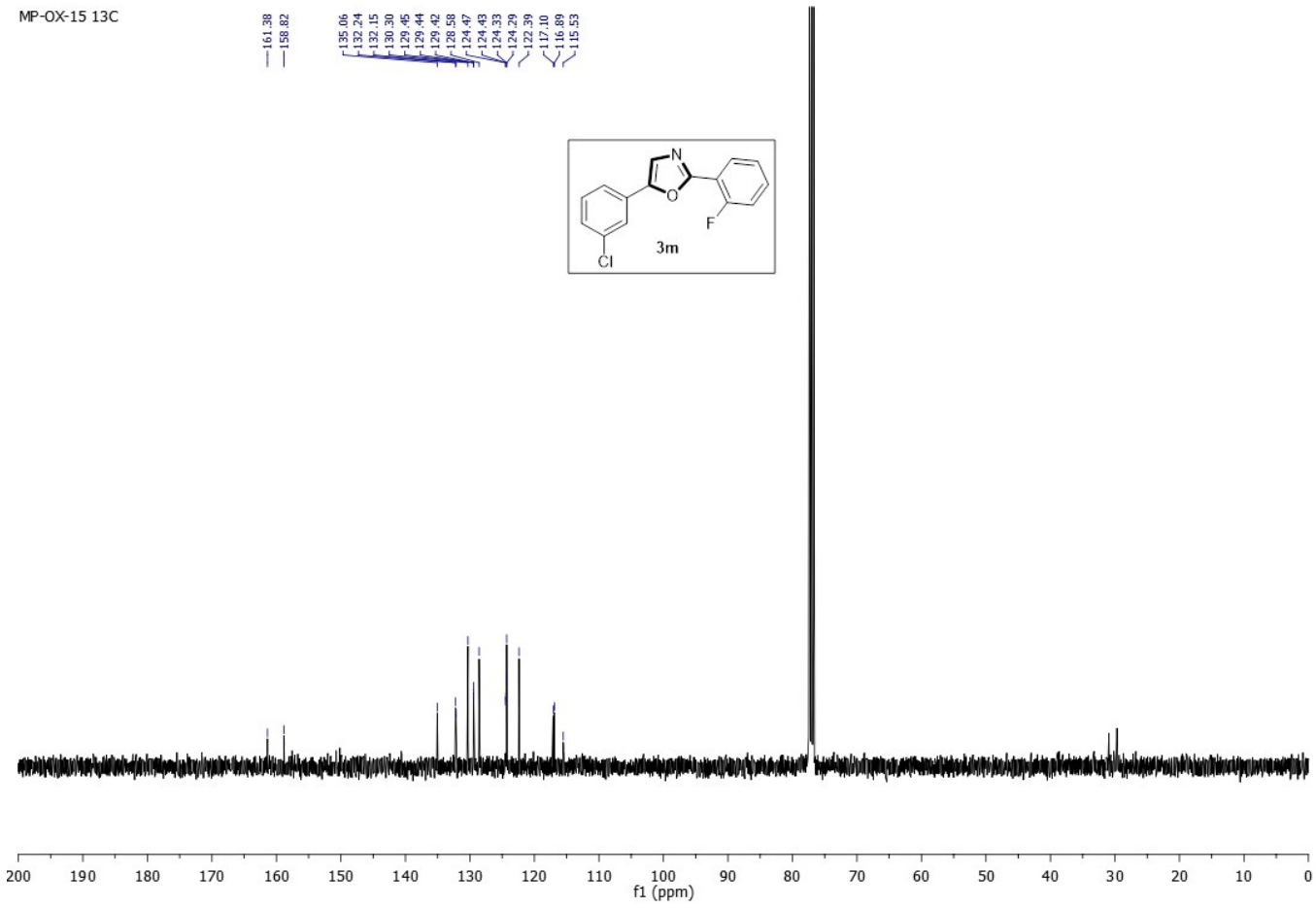

MP-OX-17 13C
163.95
161.47
151.43
134.94
131.53
130.21
130.13
129.39
128.89
125.97
125.84
125.68
124.50
124.16
123.12
121.69
118.98
118.94
118.90
116.61
116.37
114.63
114.59
114.39
114.35
N
O
F
Cl
CF3
3k
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

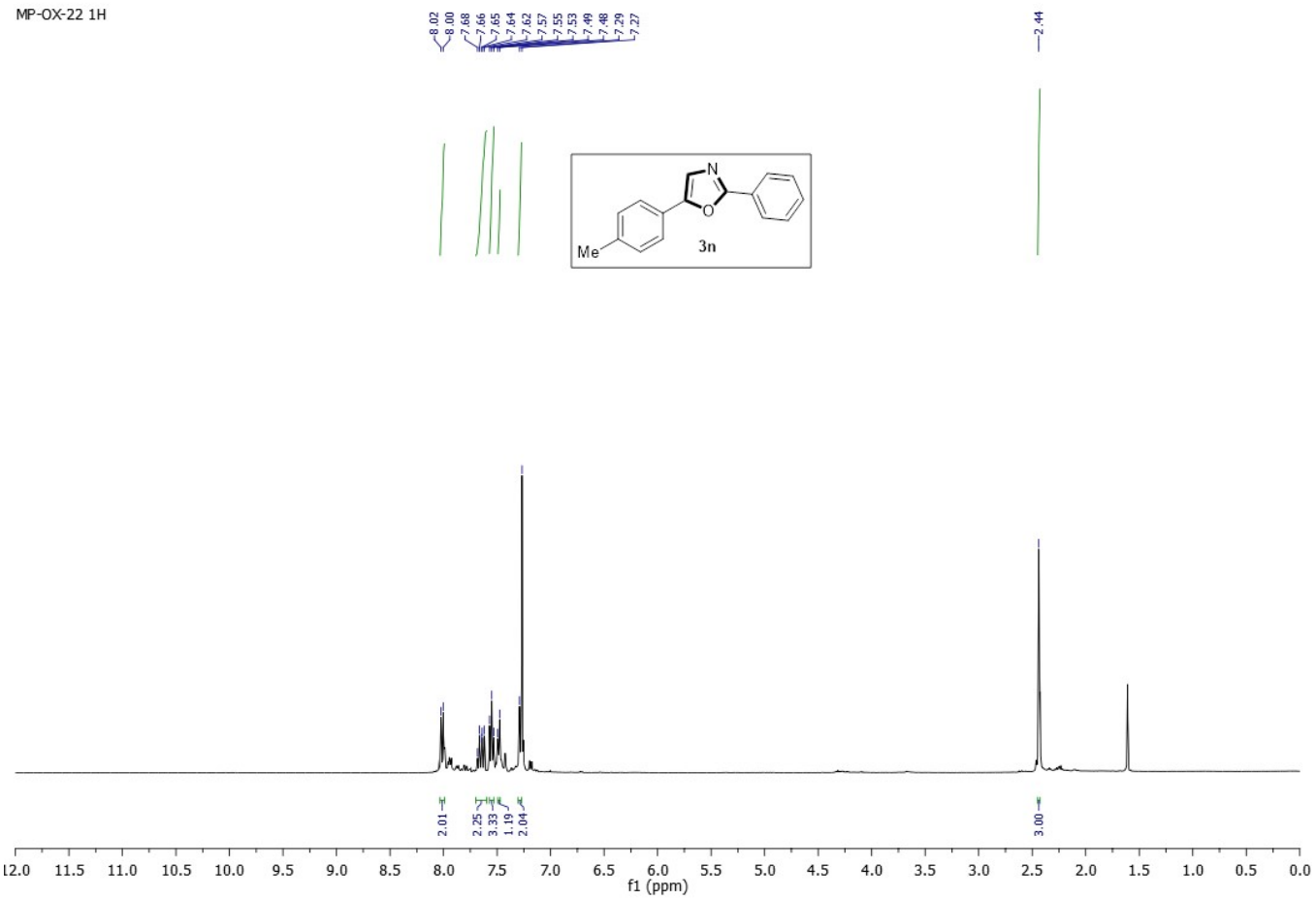
MP-OX-14

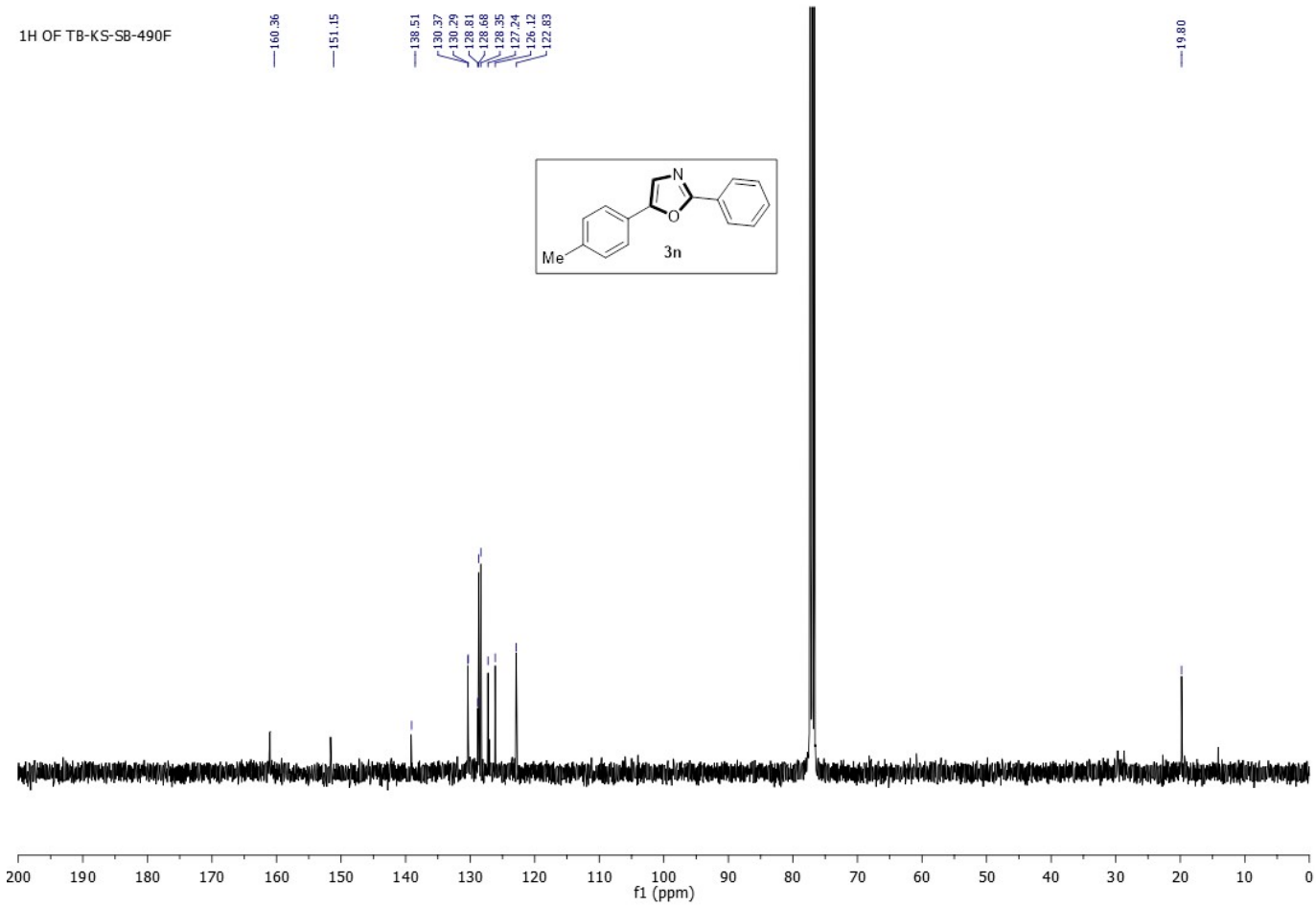

8.12
8.12
8.11
7.71
7.60
7.59
7.50
7.49
7.47
7.40
7.38
7.36
7.32
7.30
N
O
Cl
3l
2.00
0.98
1.05
4.08
1.05
0.96
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

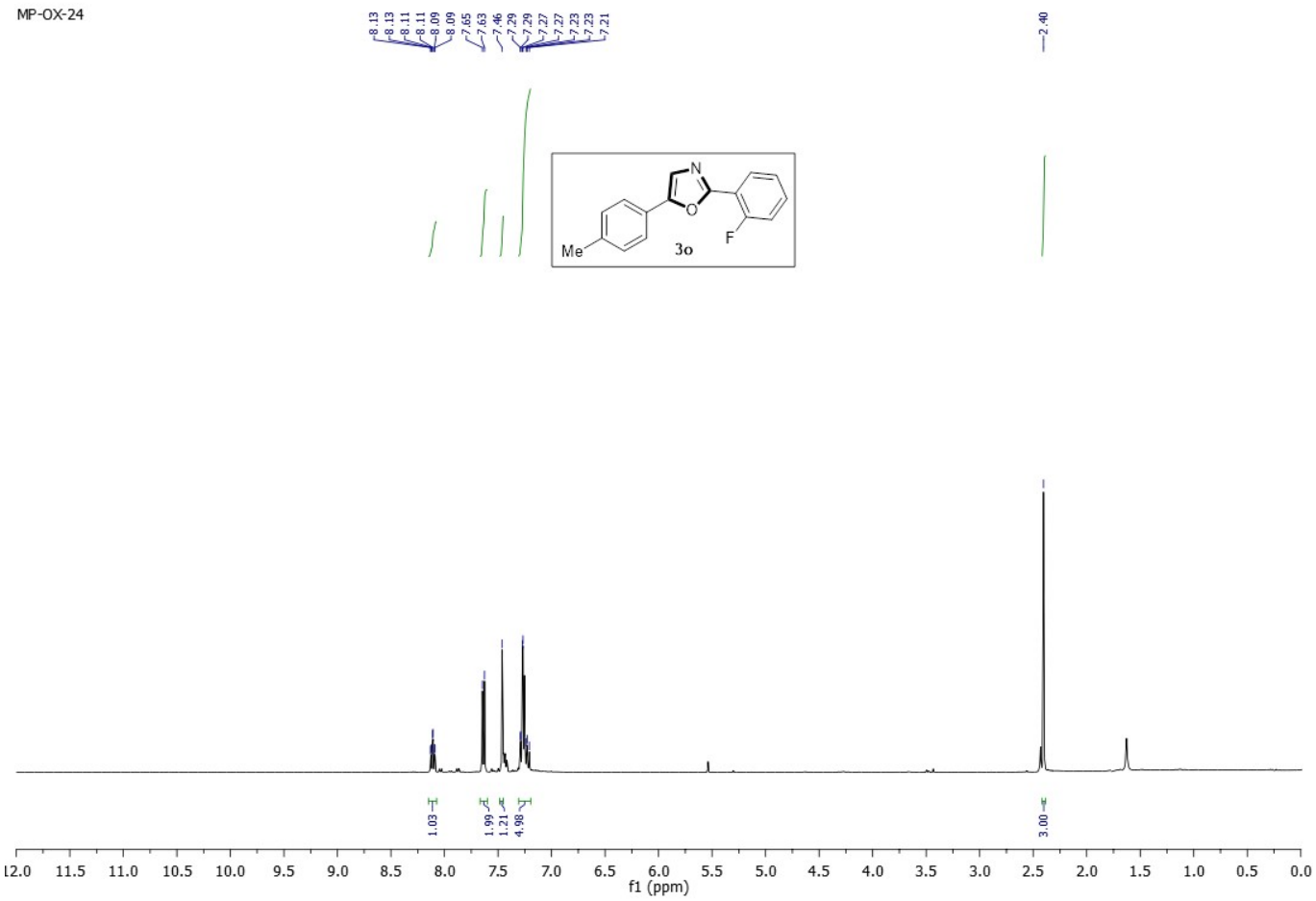

MP-OX-14
161.63
149.90
135.05
130.63
130.27
129.67
128.89
128.39
127.17
126.43
124.40
124.17
122.24
N
O
Cl
3l
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

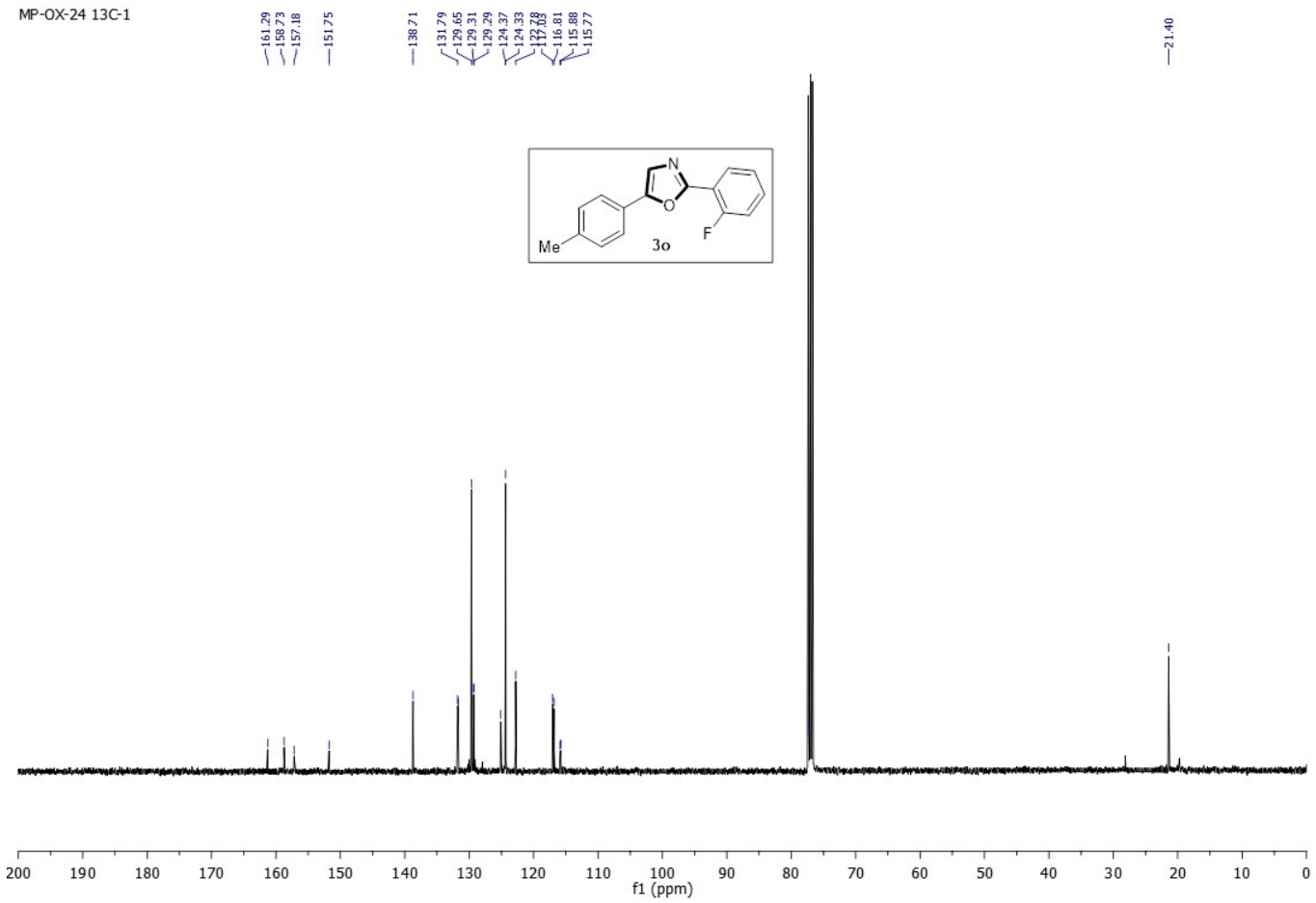

MP-OX-15
8.13
8.11
8.09
7.72
7.62
7.61
7.53
7.49
7.48
7.46
7.44
7.41
7.39
7.37
7.33
7.31
7.28
7.24
7.22
N
O
F
Cl
3m
0.98
0.95
1.01
1.02
1.11
1.10
1.90
0.82
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

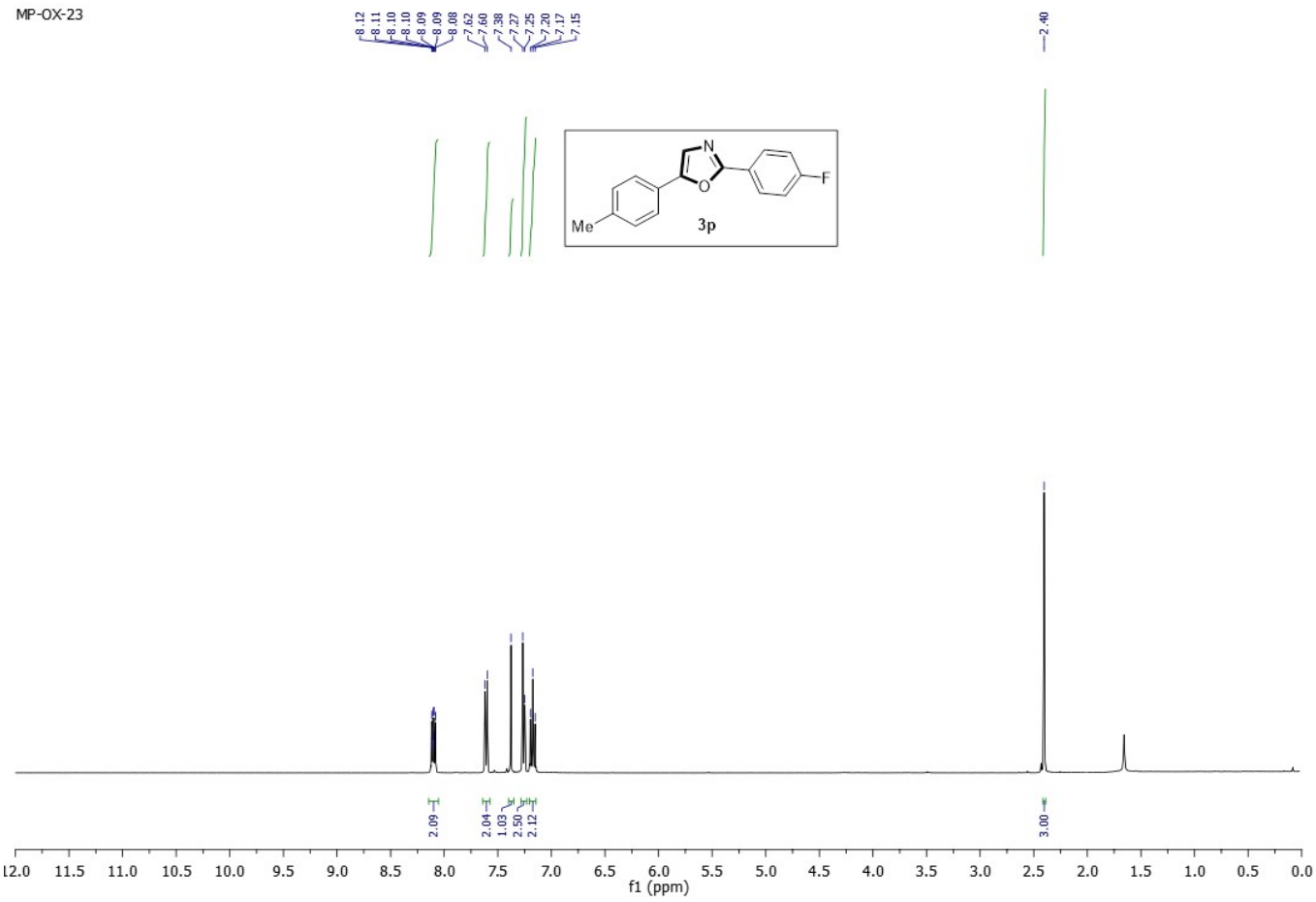

MP-OX-15 13C
161.38
158.82
135.06
132.24
132.15
130.30
129.45
129.44
129.42
128.58
124.47
124.43
124.33
124.29
122.39
117.10
116.89
115.53
N
O
F
Cl
3m
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

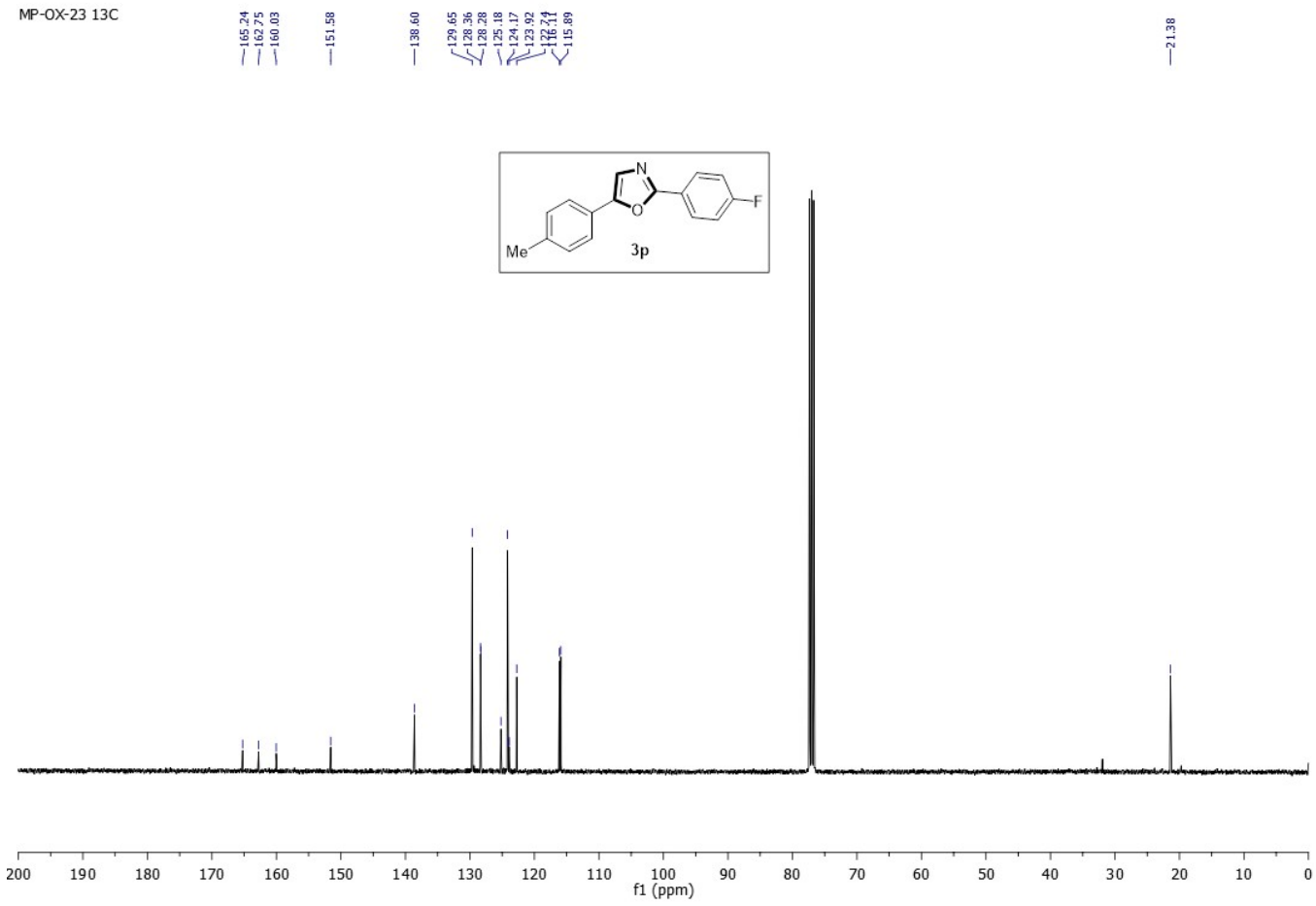

MP-OX-22 1H
8.02
8.00
7.68
7.66
7.65
7.64
7.62
7.57
7.55
7.53
7.49
7.48
7.29
7.27
2.44
N
O
Me
3n
2.01
2.25
3.33
1.19
2.04
3.00
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

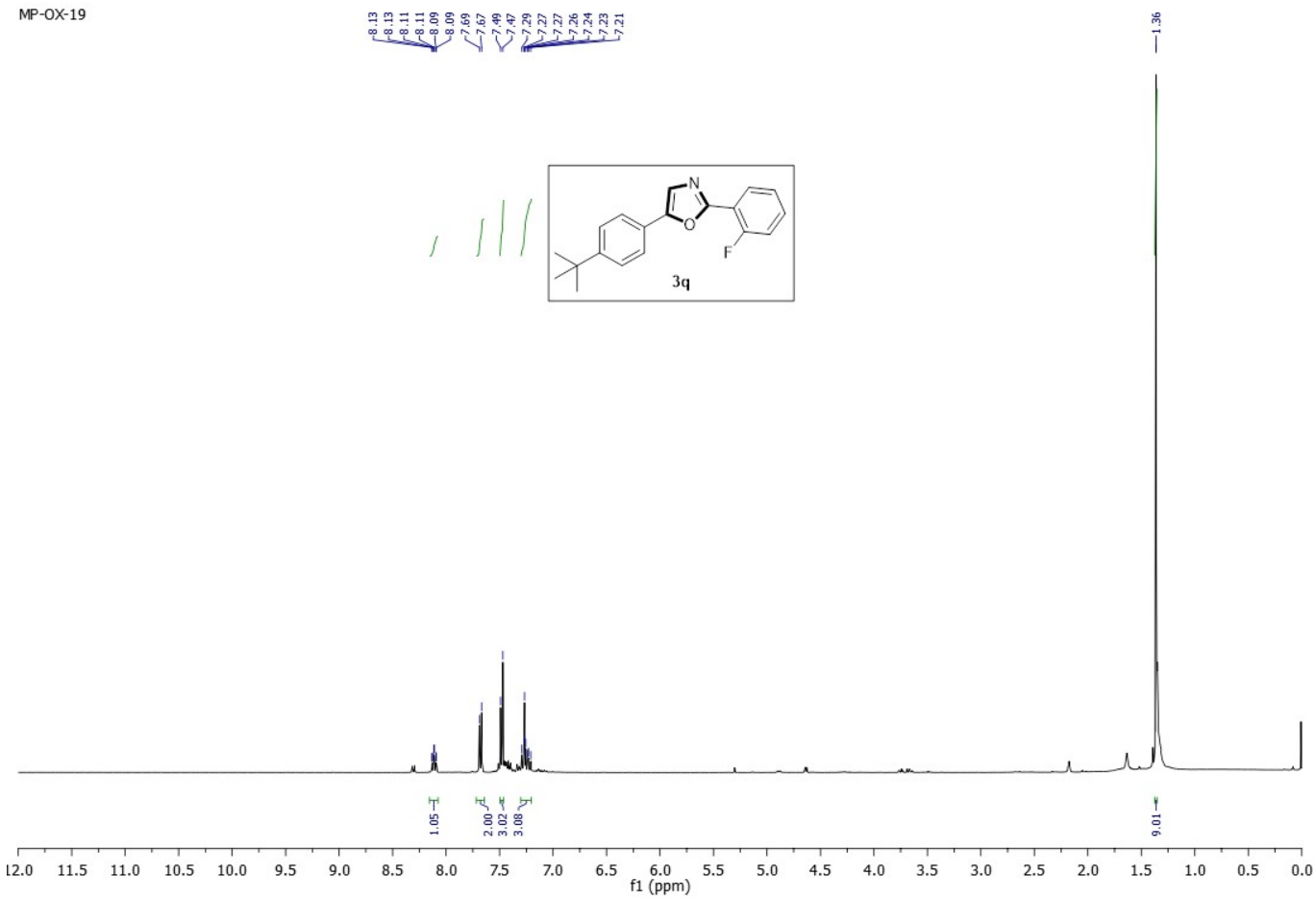

1H OF TB-KS-SB-490F
160.36
151.15
138.51
130.37
130.29
128.81
128.68
128.35
127.24
126.12
122.83
19.80
N
O
Me
3n
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

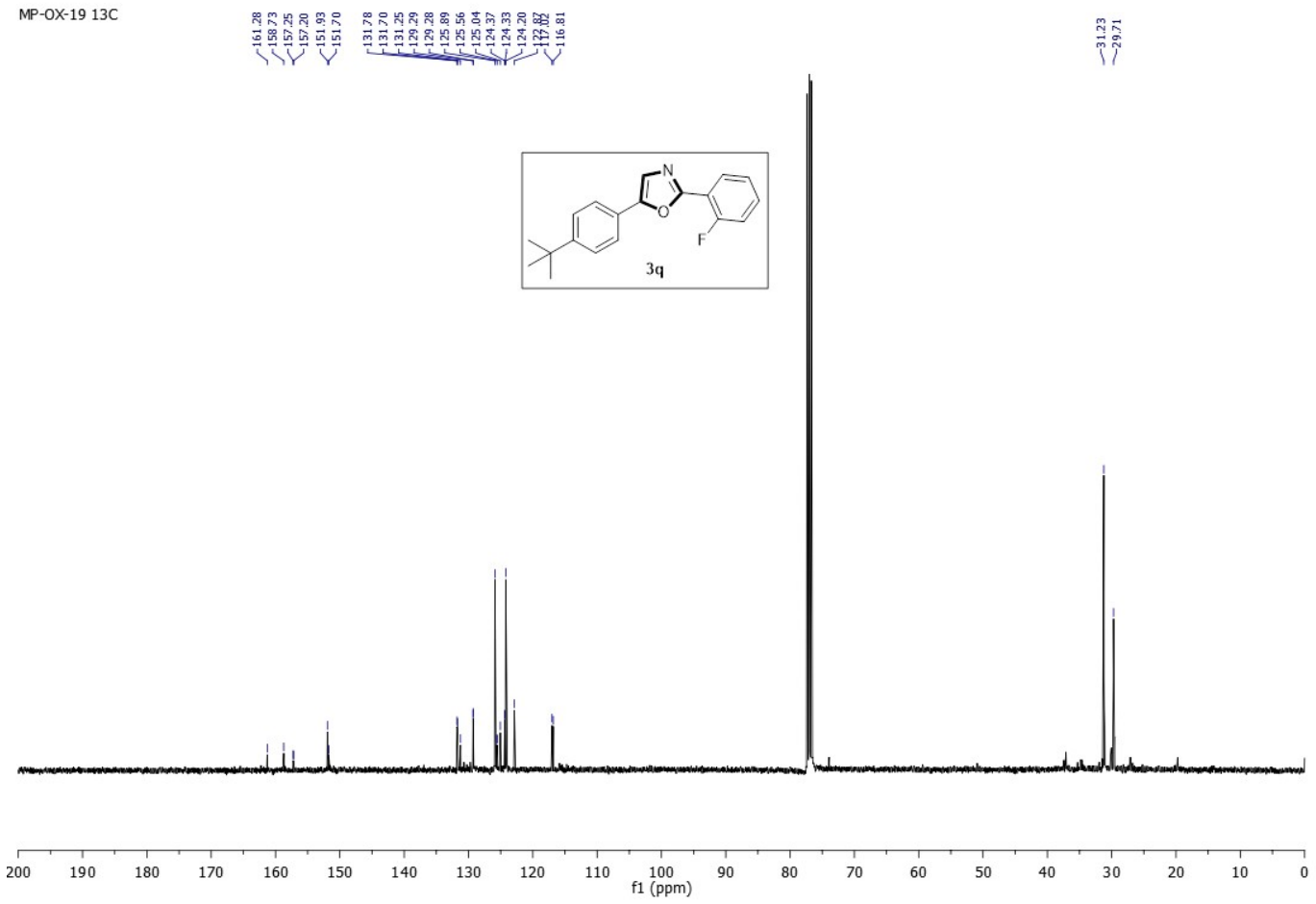

MP-OX-24
8.13
8.13
8.11
8.11
8.09
8.09
7.65
7.63
7.46
7.29
7.29
7.27
7.27
7.23
7.23
7.21
2.40
N
O
F
Me
3o
1.03
1.99
1.21
4.98
3.00
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

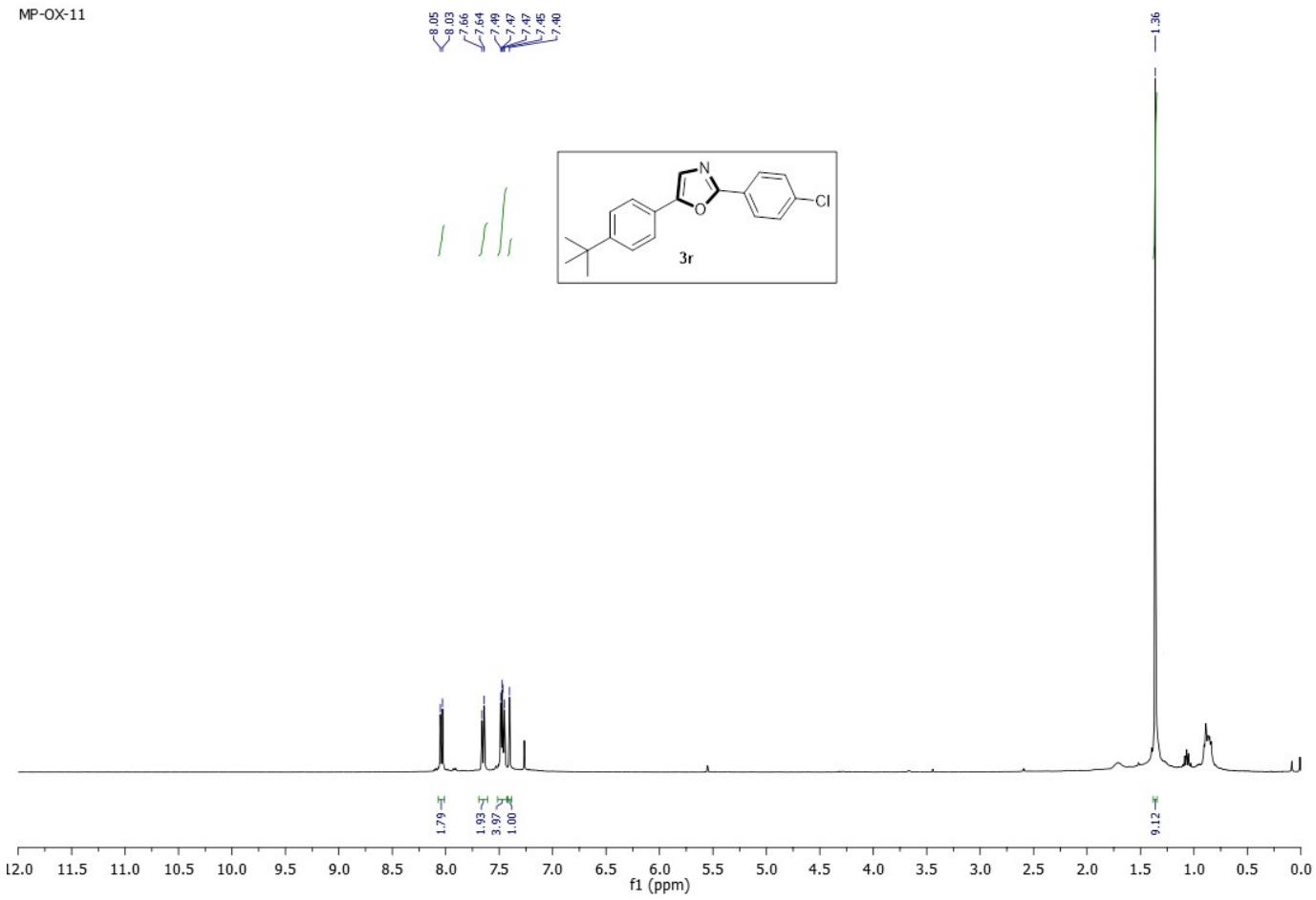

MP-OX-24 13C-1
161.29
158.73
157.18
151.75
138.71
131.79
129.65
129.31
129.29
124.37
124.33
116.81
115.88
115.77
21.40
N
O
Me
F
3o
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

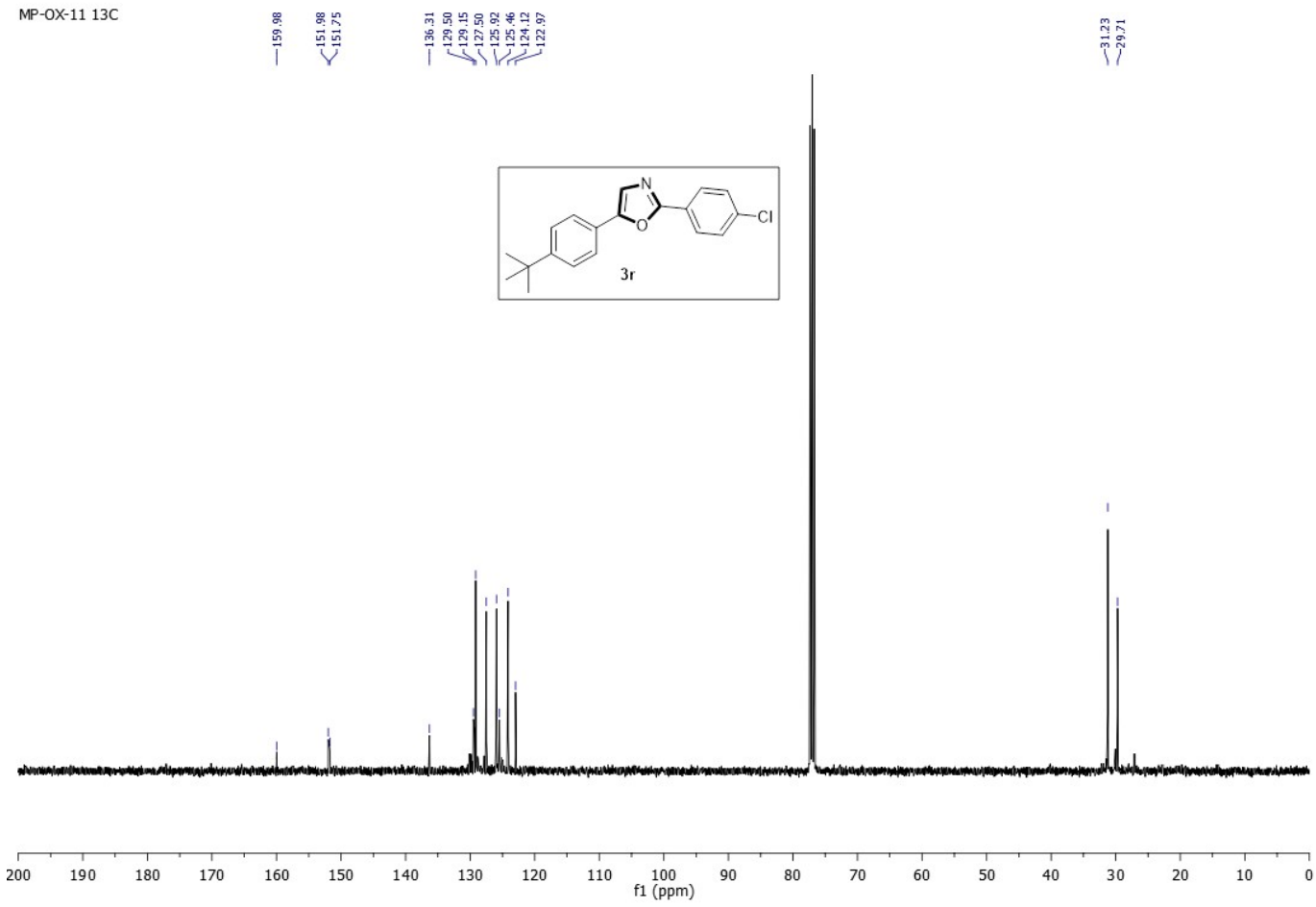

MP-OX-23
8.12
8.11
8.10
8.10
8.09
8.09
8.08
7.62
7.60
7.38
7.27
7.25
7.20
7.17
7.15
2.40
N
O
F
Me
3p
2.09
2.04
1.03
2.50
2.12
3.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

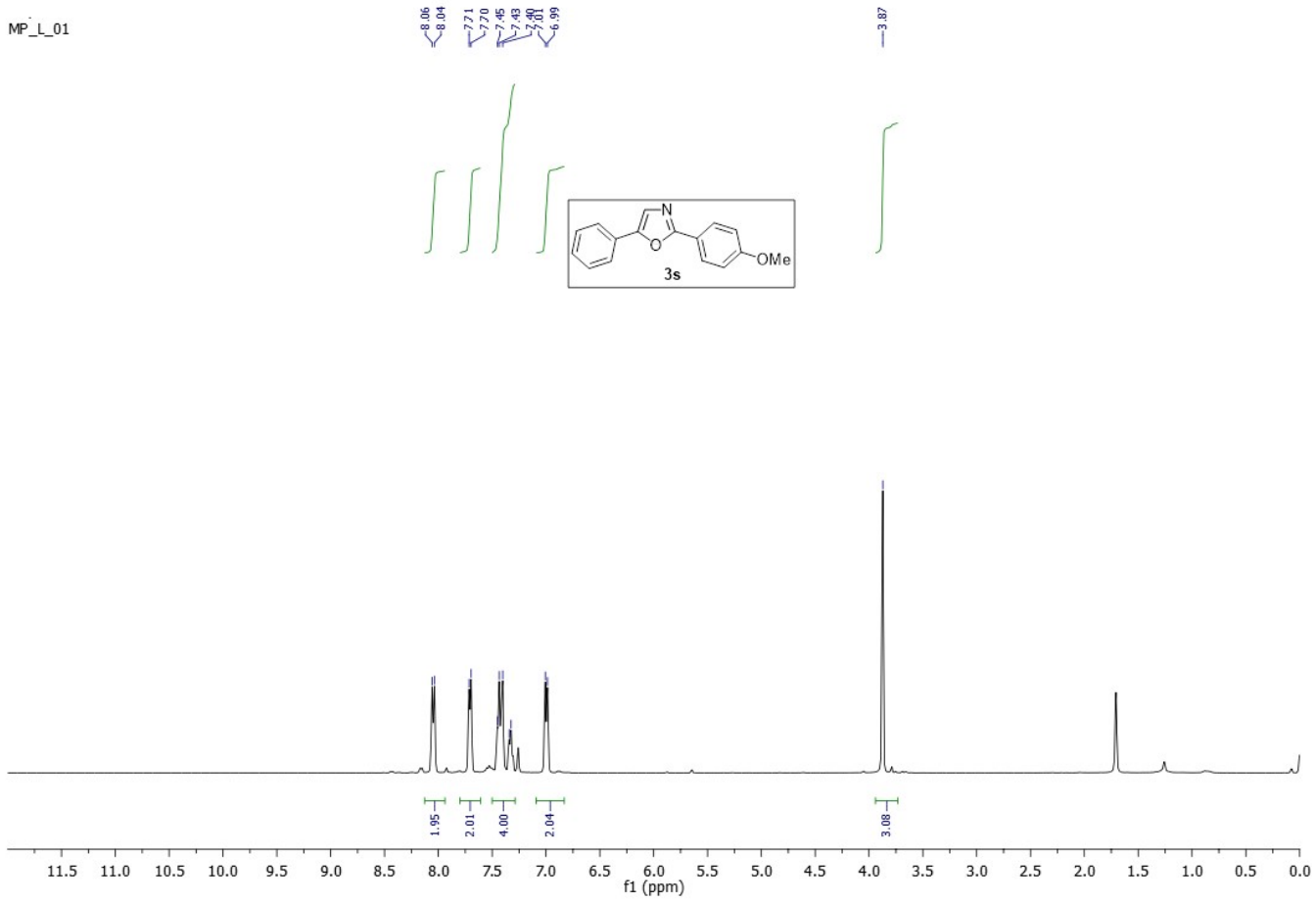

MP-OX-23 13C
165.24
162.75
160.03
151.58
138.60
129.65
128.36
128.28
125.18
124.17
123.92
122.74
116.11
115.89
21.38
N
O
F
Me
3p
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


MP-OX-19
8.13
8.13
8.11
8.11
8.09
8.09
7.69
7.67
7.49
7.47
7.29
7.27
7.27
7.26
7.24
7.23
7.21
1.36
N
O
F
3q
1.05
2.00
3.02
3.08
9.01
12.0
11.5
11.0
10.5
10.0
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
f1 (ppm)

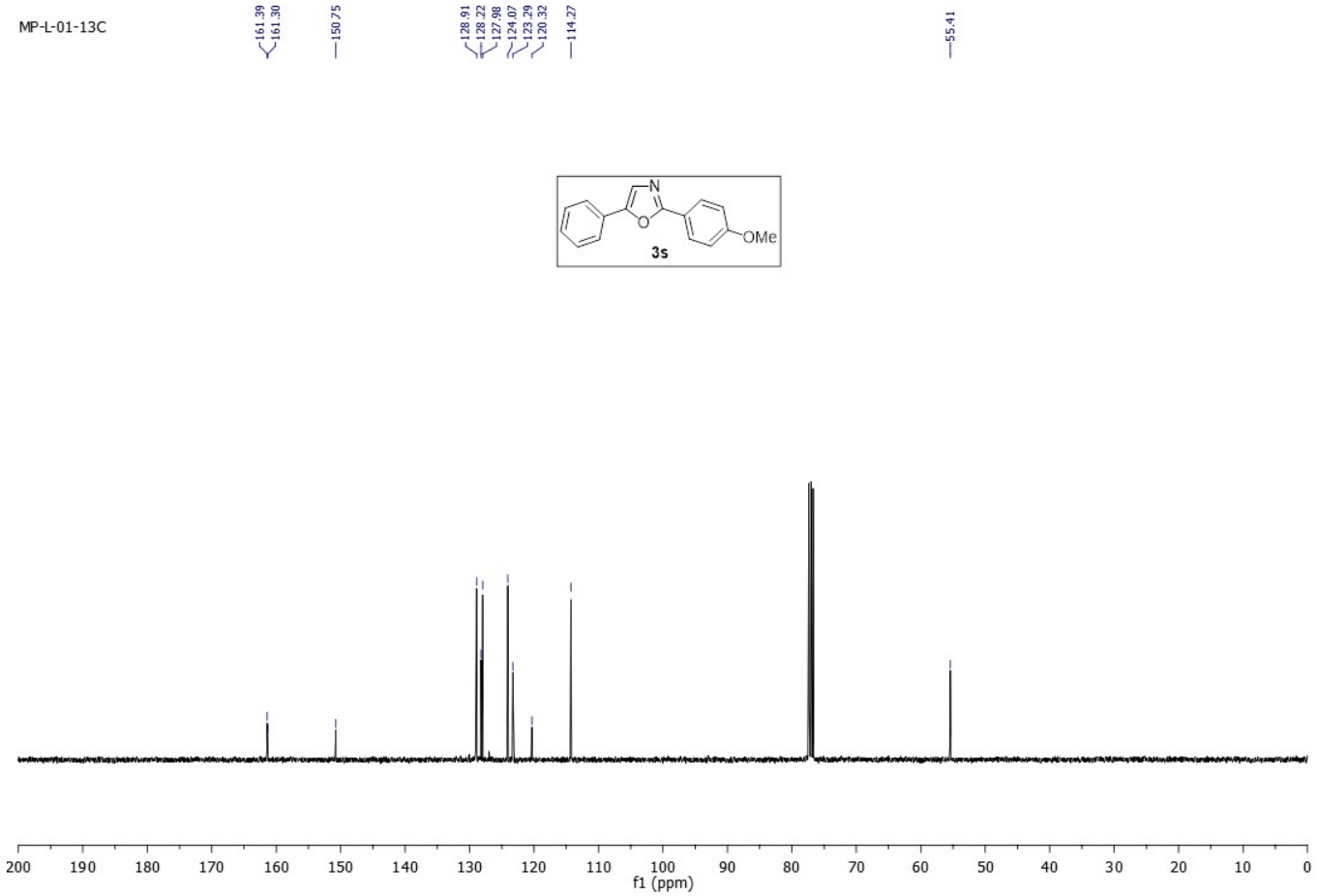

MP-OX-19 13C
161.28
158.73
157.25
157.20
151.93
151.70
131.78
131.70
131.25
129.29
129.28
125.89
125.56
125.04
124.37
124.33
124.20
116.81
31.23
29.71
N
O
F
3q
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


MP-OX-11
8.05
8.03
7.66
7.64
7.49
7.47
7.47
7.45
7.40
1.36
N
O
Cl
3r
1.79
1.93
3.97
1.00
9.12
12.0 11.5 11.0 10.5 10.0 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
f1 (ppm)


MP-OX-11 13C
159.98
151.98
151.75
136.31
129.50
129.15
127.50
125.92
125.46
124.12
122.97
31.23
29.71
N
O
Cl
3r
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


MP_L_01
8.06
8.04
7.71
7.70
7.45
7.43
7.40
7.01
6.99
3.87
N
O
OMe
3s
1.95
2.01
4.00
2.04
3.08
11.5 11.0 10.5 10.0 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
f1 (ppm)

MP-L-01-13C


161.39
161.30
150.75
128.91
128.22
127.98
124.07
123.29
120.32
114.27
55.41
N
O
OMe
3s
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)