# Supporting Information

## NHC-Palladium-catalyzed ionic liquids-accelerated regioselective oxyarylation of alkynes with diaryl ethers

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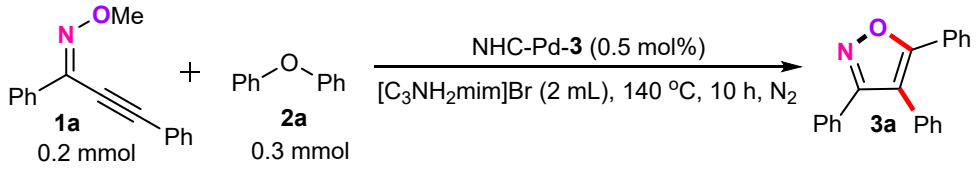
## General methods

Melting points were measured using a melting point instrument and are uncorrected. $^{1}H$ and $^{13}C$ NMR spectra were recorded on a 400 MHz NMR spectrometer. The chemical shifts are referenced to signals at 7.24 and 77.0 ppm, respectively, and chloroform was used as a solvent with TMS as the internal standard. IR spectra were obtained with an infrared spectrometer on either potassium bromide pellets or liquid films between two potassium bromide pellets. GC-MS data were obtained using electron ionization. HRMS was carried out on a high-resolution mass spectrometer (LCMS-IT-TOF). TLC was performed using commercially available 100-400 mesh silica gel plates ($GF_{254}$). Unless otherwise noted, purchased chemicals were used without further purification.

## Typical procedure for the preparation of 4-arylisoxazoles

A mixture of IPr-Pd-Im-$Cl_2$ (**Pd-3**) (0.10 mol %), ionic liquid [$C_3NH_2$mim]Br (2 mL) was added to an Schlenk tube equipped with a stir-bar. A balloon filled with $N_2$ was connected to the Schlenk tube via the side tube and purged 3 times. Then, *O*-methyl oxime ethers (**1**, 0.2 mmol), and diaryl ethers (**2**, 0.3 mmol) were quickly added to the tube under $N_2$ atmosphere and stirred at 140 $^{o}$C for 10 h. After the reaction was finished, the $N_2$ gas was released carefully and the reaction was quenched by water and extracted with $CH_2Cl_2$ three times. The combined organic layers were dried over anhydrous $Na_2SO_4$ and evaporated under vacuum. The residue was purified by flash column chromatography on silica gel (hexanes/ethyl acetate) to afford the desired products.

## General procedure for the recycling experiments [a]



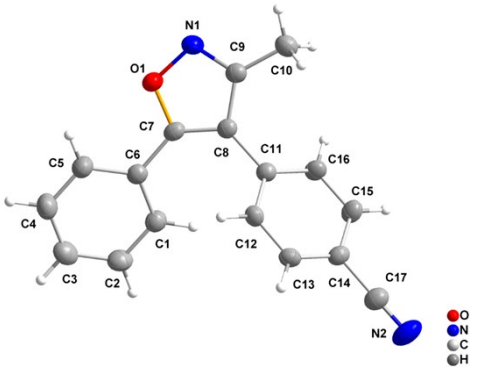

| Run | GC yield of **3a** [b] |
|---|---|
| 1 | 87% |
| 2 | 87% |
| 3 | 87% |
| 4 | 85% |
| 5 | 85% |
| 6 | 82% |
| 7 | 82% |
| 8 | 79% |
| 9 | 67% |
| 10 [c] | 86% |

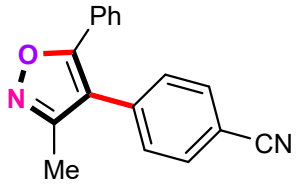
[a] All runs were performed with **1a** (0.20 mmol), **2a** (0.3 mmol), NHC-Pd-**3** (0.10 mol %), $[C_3NH_2mim]Br$ (2 mL) at 140 °C for 10 h. [b] Determined by GC using dodecane as the internal standard. [c] 1 mL of $[C_3NH_2mim]Br$ was added.

After each run, the reaction mixture was cooled to room temperature and extractions were carried out using ethyl ether (3 × 5 mL) from the ionic liquid phase. Under these conditions, neither the ionic liquid nor catalyst was extracted. Upon extractions, the catalytic system was then treated under vacuum in order to remove the volatiles. The corresponding amounts of *O*-methyl oxime ether (**1a**) and diphenyl ether (**2a**) were then added for starting a new run. The results shown that the catalytic system could be typically recovered and reused for subsequent reactions for eight times with no appreciable decrease in yields. However, after eight runs, the yield of **3a** sharply decreased from 79% to 67%. Gratifyingly, when added 1 mL of $[C_3NH_2mim]Br$ to the catalytic system, the recovered IL phase exhibited the activity as well as the fresh one.

## X-ray Crystallographic analysis for product 4j

The CCDC number of the compound **4j** is 2046761.



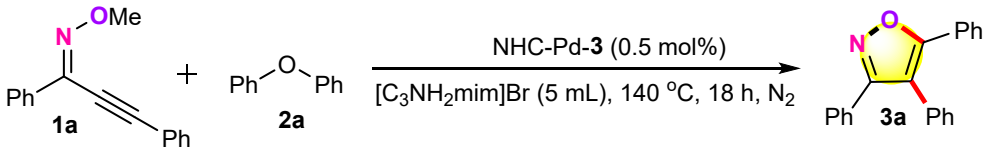




**4j**

Crystal Data and Structure Refinement for Product **4j**

| | |
|---|---|
| Empirical formula | $C_{17}H_{12}N_2O$ |
| Formula weight | 260.29 |
| Temperature | 150(10) K |
| Wavelength | 0.71073 Å |
| Crystal system | monoclinic |
| Space group | $P2_1/c$ |
| Unit cell dimensions | a = 12.0038(6) Å, α= 90.00° |
| | b= 12.5724(6) Å, β= 100.273(2)° |
| | c= 8.9600(4) Å, γ= 90.00° |
| Density (calculated) | 1.299 |
| Absorption coefficient | 0.083 |
| F(000) | 544.0 |
| Crystal size | 0.16 × 0.12 × 0.08 |
| Theta range for data collection | 4.732 to 52.8 |
| Index ranges | -15⩽h⩽15, -15 ⩽k⩽ 15, -11 ⩽l⩽ 11 |
| Reflections collected | 13015 |
| Independent reflections | 2687 |
| Completeness to theta = 29.55° | 98.40% |
| Absorption correction | multi-scan |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data/restraints/parameters | 2687/0/182 |
| Goodness-of-fit on $F^2$ | 1.065 |
| Final R indices [I>2sigma(I)] | R1 = 0.0399, wR2 = 0.0875 |
| R indices (all data) | R1 = 0.0601, wR2 = 0.1011 |

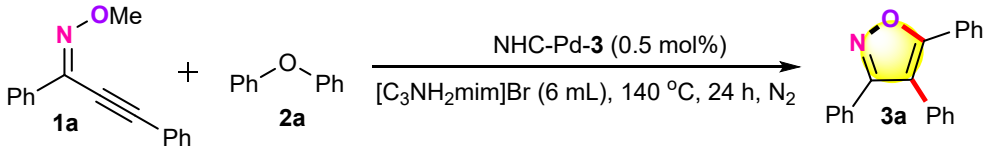
**Procedure for the Gram-scale synthesis of 3a**




5 mmol

83% (1.23g)

A mixture of IPr-Pd-Im-$Cl_2$ (**Pd-3**) (0.5 mol %), ionic liquid [$C_3NH_2$mim]Br (5 mL) was added to an Schlenk tube equipped with a stir-bar. A balloon filled with $N_2$ was connected to the Schlenk tube via the side tube and purged 3 times. Then, 1,3-diphenylprop-2-yn-1-one *O*-methyl oxime (**1a**, 5 mmol), and diphenyl ether (**2a**, 7.5 mmol) were quickly added to the tube under $N_2$ atmosphere and stirred at 140 °C for 18 h. After the reaction was finished, the $N_2$ gas was released carefully and the reaction was quenched by water and extracted with $CH_2Cl_2$ three times. The combined organic layers were dried over anhydrous $Na_2SO_4$ and evaporated under vacuum. The residue was purified by flash column chromatography on silica gel (hexanes/ethyl acetate: 100:1) to afford the desired product **3a** in 83% yield (1.23g).




8 mmol

78% (1.85g)

A mixture of IPr-Pd-Im-$Cl_2$ (**Pd-3**) (0.5 mol %), ionic liquid [$C_3NH_2$mim]Br (6 mL) was added to an Schlenk tube equipped with a stir-bar. A balloon filled with $N_2$ was connected to the Schlenk tube via the side tube and purged 3 times. Then, 1,3-diphenylprop-2-yn-1-one *O*-methyl oxime (**1a**, 8 mmol), and diphenyl ether (**2a**, 12 mmol) were quickly added to the tube under $N_2$ atmosphere and stirred at 140 °C for 24 h. After the reaction was finished, the $N_2$ gas was released carefully and the reaction was quenched by water and extracted with $CH_2Cl_2$ three times. The combined organic layers were dried over anhydrous $Na_2SO_4$ and evaporated under vacuum. The residue was purified by flash column chromatography on silica gel (hexanes/ethyl acetate: 100:1) to afford the desired product **3a** in 78% yield (1.85g).

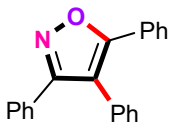
**Procedure for the synthesis of COX-2 inhibitor Parecoxib 8** [1]

Chlorosulfonic acid (0.2 mmol) was cooled in ice bath, and 5-methyl-3,4-diphenylisoxazole (**7**, 0.2 mmol) was added at the 0 °C. After that, the ice bath was removed and the mixture was stirred at room temperature for 24 h. The above mixture was then diluted with $CH_2Cl_2$ (3 × 10 mL), then the ice water (3 × 10 mL) was added slowly. The combined organic layers were dried over anhydrous $Na_2SO_4$, evaporated under vacuum, and obtained as a yellow mixture. Then, the unpurified mixture was dissolved with $CH_2Cl_2$ (5 mL), cooled to 5-10 °C, and added $NH_4OH$ (5 mL) and stirred at 15 °C for 30 min. After the reaction was finished, the reaction was quenched by water and extracted with $CH_2Cl_2$ three times. The combined organic layers were dried over anhydrous $Na_2SO_4$ and evaporated under vacuum. The residue was purified by flash column chromatography on silica gel (hexanes/ethyl acetate: 2:3) to afford the desired product **8**.

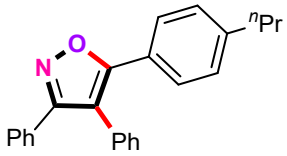
**References**

[1] J. J. Talley, D. L. Brown, J. S. Carter, M. J. Graneto, C. M. Koboldt, J. L. Masferrer, W. E. Perkins, R. S. Rogers, A. F. Shaffer, Y. Y. Zhang, B. S. Zweifel, K. Seibert, *J. Med. Chem*. **2000**, *43*, 775-777.

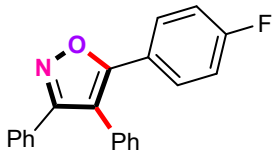
## Characterization data for all products



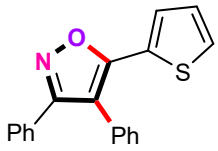

3,4,5-Triphenylisoxazole (**3a**): Yield: 81% as a white solid; mp = 197.4 - 198.7 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.58 - 7.52 (m, 2H), 7.46 - 7.41 (m, 2H), 7.41 - 7.37 (m, 3H), 7.37 - 7.31 (m, 4H), 7.31 - 7.22 (m, 4H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 165.6, 162.2, 130.6, 130.5, 129.8, 129.4, 129.1, 129.0, 128.7, 128.5, 128.4, 128.2, 127.9, 127.0, 115.3; $\nu_{max}$(KBr)/cm$^{-1}$ 3060, 2928, 1624, 1585, 1453, 1426, 754; MS (EI) m/z 77, 105, 165, 269, 297; HRMS-ESI (m/z): calcd for $C_{21}H_{16}NO$, $[M + H]^+$: 298.1226, found 298.1223.



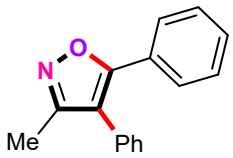

3,4-Diphenyl-5-(4-propylphenyl)isoxazole (**3b**): Yield: 80% as a yellow solid; mp = 128.5 - 129.7 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.56 - 7.46 (m, 4H), 7.45 - 7.36 (m, 4H), 7.37 - 7.29 (m, 4H), 7.25 - 7.13 (m, 2H), 2.74 - 2.41 (m, 2H), 1.74 - 1.54 (m, 2H), 0.97 (t, $J$ = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 165.8, 162.2, 144.8, 130.9, 130.6, 129.4, 129.2, 129.1, 128.8, 128.5, 128.4, 128.2, 126.8, 125.4, 114.7, 37.9, 24.2, 13.8 ppm; $\nu_{max}$(KBr)/cm$^{-1}$ 3042, 2936, 1640, 1568, 1445, 1256, 752; MS (EI) m/z 91, 147, 178, 222, 310, 339; HRMS-ESI (m/z): calcd for $C_{24}H_{22}NO$, $[M+H]^+$: 340.1696, found 340.1690.



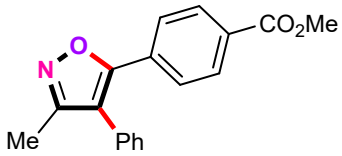

5-(4-Fluorophenyl)-3,4-diphenylisoxazole (**3c**): Yield: 75% as a yellow solid; mp =185.2 - 186.8 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.62 - 7.53 (m, 2H), 7.50 - 7.40 (m, 5H), 7.39 - 7.25 (m, 5H), 7.12 - 7.01 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 164.7, 163.5 (d, $J$ = 241.5 Hz), 162.2, 130.5, 130.4, 129.5, 129.2, 129.0, 128.9, 128.8, 128.5, 128.4, 128.3, 124.2 (d, $J$ = 3.5 Hz), 115.9 (d, $J$ = 22.0 Hz); $\nu_{max}$(KBr)/cm$^{-1}$ 3046, 2924, 1626, 1582, 1450, 754; MS (EI) m/z 89, 123, 198, 287, 315; HRMS-ESI (m/z): calcd for $C_{21}H_{15}FNO$, $[M + H]^+$: 316.1132, found 316.1128.



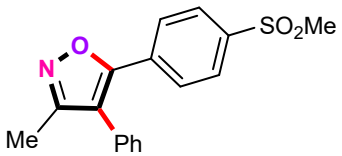

3,4-Diphenyl-5-(thiophen-2-yl)isoxazole (**3d**): Yield: 85% as a yellow solid; mp = 153.8 - 154.2 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.58 - 7.54 (m, 1H), 7.53 - 7.49 (m, 2H), 7.48 - 7.44 (m, 3H), 7.42 - 7.32 (m, 5H), 7.30 (dd, $J$ = 5.2, 2.8 Hz, 1H), 7.22 (dd, $J$ = 5.2, 1.2 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 162.6, 161.9, 130.6, 130.5, 129.5, 129.2, 129.0, 128.8, 128.5, 128.4, 128.3, 126.3, 125.6, 124.9, 114.5; $\nu_{max}$(KBr)/cm$^{-1}$ 3056, 2933, 1622, 1580, 1458, 1416, 758; MS (EI) m/z 77, 111, 171, 186, 275, 303; HRMS-ESI (m/z): calcd for $C_{19}H_{14}NOS$, $[M + H]^+$: 304.0791, found 304.0785.




3-Methyl-4,5-diphenylisoxazole (**3e**): Yield: 71% as a yellow oil; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.57 (d, $J$ = 7.6 Hz, 2H), 7.54 - 7.42 (m, 3H), 7.35 (dt, $J$ = 12.4, 6.2 Hz, 5H), 2.28 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 164.3, 160.2, 130.7, 129.9, 129.7, 129.1, 128.7, 128.1, 127.9, 126.9, 116.2, 10.7 ppm; $\nu_{max}$(KBr)/cm$^{-1}$ 3036, 2921, 1635, 1534, 1428, 1246, 754; MS (EI) m/z 77, 104, 165, 206, 235; HRMS-ESI (m/z): calcd for $C_{16}H_{14}NO$, $[M+H]^+$: 236.1070, found 236.1064.

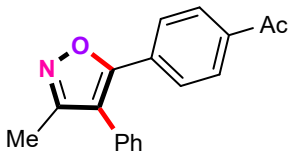



Methyl 4-(3-methyl-4-phenylisoxazol-5-yl)benzoate (**3f**): Yield: 57% as a yellow solid; mp = 111.3 - 112.6 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 8.13 (d, $J$ = 8.2 Hz, 2H), 7.54 - 7.50 (m, 2H), 7.42 (t, $J$ = 7.2 Hz, 3H), 7.38 - 7.31 (m, 2H), 3.98 (s, 3H), 2.29 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 166.7, 164.9, 159.7, 135.6, 130.3, 130.0, 129.9, 129.0, 128.8, 128.3, 127.5, 127.0, 115.3, 52.3, 10.7 ppm; $\nu_{max}$(KBr)/cm$^{-1}$ 3043, 2930, 1644, 1556, 1423, 1256, 747; MS (EI) m/z 77, 104, 165, 188, 264, 293; HRMS-ESI (m/z): calcd for $C_{18}H_{16}NO_3$, $[M+H]^+$: 294.1125, found 294.1118.

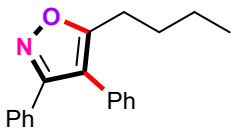



3-Methyl-5-(4-(methylsulfonyl)phenyl)-4-phenylisoxazole (**3g**): Yield: 62% as a yellow solid; mp = 136.1 - 137.7 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 8.03 (d, $J$ = 8.4 Hz, 2H), 7.67 - 7.47 (m, 4H), 7.46 - 7.33 (m, 3H), 3.16 (s, 3H), 2.30 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 165.5, 159.4, 140.2,

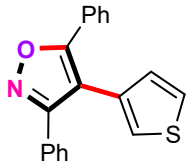
136.8, 130.8, 130.4, 128.9, 128.2, 127.2, 127.1, 114.5, 44.4, 10.8 ppm; $\nu_{max}$(KBr)/cm$^{-1}$ 3047, 2926, 1646, 1556, 1421, 1245, 763; MS (EI) m/z 77, 133, 191, 207, 281, 313; HRMS-ESI (m/z): calcd for $C_{17}H_{16}NO_3S$, $[M+H]^+$: 314.0845, found 314.0838.



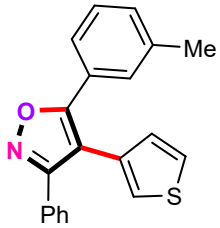

1-(4-(3-Methyl-4-phenylisoxazol-5-yl)phenyl)ethan-1-one (**3h**): Yield: 62% as a yellow oil; $^1$H NMR (400 MHz, $CDCl_3$) $\delta$ 8.05 (d, $J$ = 8.0 Hz, 2H), 7.53 (d, $J$ = 7.2 Hz, 2H), 7.43 (d, $J$ = 8.0 Hz, 2H), 7.36 (dd, $J$ = 13.4, 6.2 Hz, 3H), 2.67 (s, 3H), 2.29 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 197.6, 165.0, 159.7, 136.6, 135.8, 130.1, 129.1, 128.8, 127.5, 127.0, 115.2, 26.7, 10.7 ppm; $\nu_{max}$(KBr)/cm$^{-1}$ 3056, 2930, 1638, 1566, 1452, 1256, 764; MS (EI) m/z 77, 105, 165, 207, 234, 262, 277; HRMS-ESI (m/z): calcd for $C_{18}H_{16}NO_2$, $[M+H]^+$: 278.1176, found 278.1170.



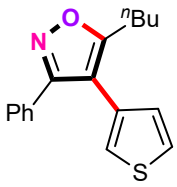

5-Butyl-3,4-diphenylisoxazole (**3i**): Yield: 86% as a yellow oil; $^1$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.51 - 7.45 (m, 2H), 7.43 - 7.36 (m, 4H), 7.36 - 7.30 (m, 2H), 7.26 - 7.18 (m, 2H), 2.97 - 2.65 (m, 2H), 1.97 - 1.64 (m, 2H), 1.48 - 1.36 (m, 2H), 0.93 (t, $J$ = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 170.4, 161.0, 134.8, 130.6, 130.0, 129.3, 128.7, 128.4, 127.7, 115.5, 83.8, 29.8, 24.9, 22.3, 13.7 ppm; $\nu_{max}$(KBr)/cm$^{-1}$ 3046, 2933, 1658, 1523, 1454, 1256, 754; MS (EI) m/z 77, 158, 243, 285, 327; HRMS-ESI (m/z): calcd for $C_{19}H_{20}NO$, $[M+H]^+$: 278.1539, found 278.1534.



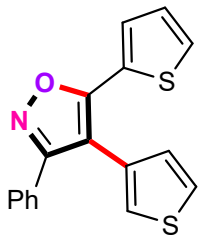

3,5-Diphenyl-4-(thiophen-3-yl)isoxazole (**3j**): Yield: 80% as a white solid; mp = 179.0 - 179.8 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.64 - 7.57 (m, 2H), 7.50 - 7.43 (m, 2H), 7.41 (dd, $J$ = 4.8, 3.2 Hz, 1H), 7.39 - 7.30 (m, 6H), 7.16 (d, $J$ = 4.8 Hz, 1H), 6.97 (d, $J$ = 4.8 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 165.8, 162.4, 130.1, 129.9, 129.5, 129.0, 129.0, 128.7, 128.5, 128.3, 127.9, 126.9, 126.7, 125.4, 110.3 ppm; $\nu_{max}$(KBr)/cm$^{-1}$ 3054, 2930, 1646, 1534, 1436, 753; MS (EI) m/z 77, 105, 171, 275, 303; HRMS-ESI (m/z): calcd for $C_{19}H_{14}NOS$, $[M+H]^+$: 304.0791, found 304.0785.



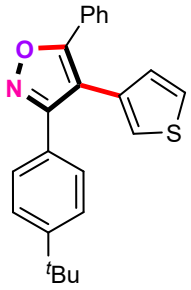

3-Phenyl-4-(thiophen-3-yl)-5-(m-tolyl)isoxazole (**3k**): Yield: 84% as a white solid; mp = 130.5 - 132.2 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.64 - 7.48 (m, 3H), 7.48 - 7.35 (m, 5H), 7.32 - 7.16 (m, 3H), 7.01 (d, $J$ = 4.8 Hz, 1H), 2.37 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 166.1, 162.3, 138.5, 130.7, 130.2, 129.5, 129.1, 129.0, 128.6, 128.5, 128.4, 127.8, 127.6, 126.6, 125.4, 124.0, 110.2, 21.5 ppm; $\nu_{max}$(KBr)/cm$^{-1}$ 3046, 2927, 1648, 1532, 1458, 1266, 760; MS (EI) m/z 91, 119, 194, 289, 317; HRMS-ESI (m/z): calcd for $C_{20}H_{16}NOS$, $[M+H]^+$: 318.0947, found 318.0942.



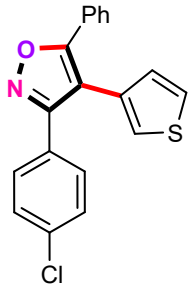

5-Butyl-3-phenyl-4-(thiophen-3-yl)isoxazole (**3l**): Yield: 64% as a yellow solid; mp = 68.2 - 69.5 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.50 (d, $J$ = 7.8 Hz, 2H), 7.45 - 7.32 (m, 4H), 7.15 (d, $J$ = 1.8 Hz, 1H), 6.90 (d, $J$ = 4.8 Hz, 1H), 2.83 (t, $J$ = 7.6 Hz, 2H), 1.91 - 1.66 (m, 2H), 1.51 - 1.31 (m, 2H), 0.94 (t, $J$ = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 170.5, 161.1, 130.2, 129.4), 129.3, 128.7, 128.5, 128.4, 126.0, 124.0, 110.7, 29.8, 25.7, 22.4, 13.7 ppm; $\nu_{max}$(KBr)/cm$^{-1}$ 3044, 2932, 1638, 1566, 1449, 1257, 752; MS (EI) m/z 77, 95, 171, 199, 250, 283; HRMS-ESI (m/z): calcd for $C_{17}H_{18}NOS$, $[M+H]^+$: 284.1104, found 284.1099.



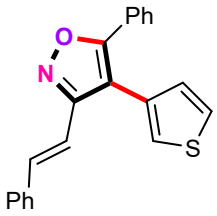

3-Phenyl-5-(thiophen-2-yl)-4-(thiophen-3-yl)isoxazole (**3m**): Yield: 76% as a white solid; mp = 140.3 - 141.4 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.60 (d, $J$ = 1.8 Hz, 1H), 7.53 (d, $J$ = 8.0 Hz, 2H), 7.49 (d, $J$ = 2.0 Hz, 1H), 7.44 - 7.31 (m, 4H), 7.26 (d, $J$ = 5.2 Hz, 2H), 7.05 (d, $J$ = 4.6 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 162.8, 162.0, 130.0, 129.6, 129.1, 129.0, 128.8, 128.5, 128.2, 126.8, 126.4, 125.7, 125.6, 125.0, 109.4 ppm; $\nu_{max}$(KBr)/cm$^{-1}$ 3046, 2936, 1568, 1452, 1256, 753; MS (EI) m/z 77, 111, 186, 280, 309; HRMS-ESI (m/z): calcd for $C_{17}H_{12}NOS_2$, $[M+H]^+$: 310.0355, found 310.0350.



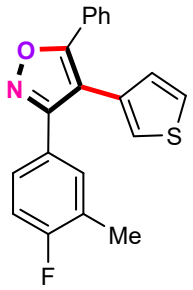

3-(4-(*tert*-Butyl)phenyl)-5-phenyl-4-(thiophen-3-yl)isoxazole (**3n**): Yield: 82% as a yellow solid; mp = 145.5 - 146.8 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.58 (d, $J$ = 8.8 Hz, 2H), 7.54 -7.49 (m, 2H), 7.48 - 7.39 (m, 4H), 7.36 (dd, $J$ = 8.0, 6.0 Hz, 2H), 7.22 (dd, $J$ = 3.0, 1.2 Hz, 1H), 7.10 - 6.98 (m, 1H), 1.36 (s, 9H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 166.0, 162.3, 153.3, 130.3, 129.5, 129.2, 128.5, 128.3, 126.6, 126.5, 125.7, 125.4, 125.1, 109.7, 34.9, 31.2 ppm; $\nu_{max}$(KBr)/cm$^{-1}$ 3044, 2932, 1566, 1452, 1258, 756; MS (EI) m/z 77, 118, 161, 236, 344, 359; HRMS-ESI (m/z): calcd for $C_{23}H_{22}NOS$, $[M+H]^+$: 360.1417, found 360.1410.



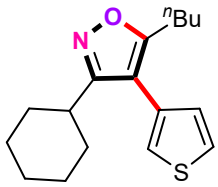

3-(4-Chlorophenyl)-5-phenyl-4-(thiophen-3-yl)isoxazole (**3o**): Yield: 81% as a yellow solid; mp = 170.1 - 171.2 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.62 (d, $J$ = 8.0 Hz, 2H), 7.50 - 7.37 (m, 6H), 7.34 (d, $J$ = 8.4 Hz, 2H), 7.26 - 7.19 (m, 1H), 7.00 (d, $J$ = 4.8 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 166.1, 161.4, 135.7, 130.1, 129.8, 129.6, 128.9, 128.8, 128.8, 127.7, 127.5, 127.0, 126.9, 125.5, 110.1 ppm; $\nu_{max}$(KBr)/cm$^{-1}$ 3045, 2928, 1637, 1565, 1416, 746; MS (EI) m/z 77, 105, 171, 274, 309, 337; HRMS-ESI (m/z): calcd for $C_{19}H_{13}ClNOS$, $[M-H]^+$: 336.0255, found 336.0258.



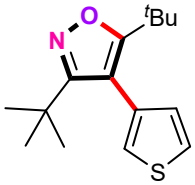

5-Phenyl-3-styryl-4-(thiophen-3-yl)isoxazole (**3p**): Yield: 64% as a yellow solid; mp = 128.8 - 130.5 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.63 (dt, $J$ = 8.0, 2.8 Hz, 2H), 7.53 (dd, $J$ = 5.0, 3.0 Hz, 1H), 7.48 - 7.43 (m, 2H), 7.40 (ddd, $J$ = 6.8, 4.4, 1.6 Hz, 3H), 7.38 - 7.33 (m, 3H), 7.33 - 7.25 (m, 2H), 7.13 (dd, $J$ = 5.0, 1.2 Hz, 1H), 6.91 (d, $J$ = 16.6 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 165.4, 160.2, 136.1, 135.8, 130.0, 129.9, 128.9, 128.8, 128.7, 128.7, 127.7, 127.1, 126.9, 126.8, 125.3, 114.7, 110.4 ppm; $\nu_{max}$(KBr)/cm$^{-1}$ 3046, 2930, 1646, 1578, 1456, 754; MS (EI) m/z 77, 105, 171, 224, 296, 329; HRMS-ESI (m/z): calcd for $C_{21}H_{16}NOS$, $[M+H]^+$: 330.0947, found 330.0939.



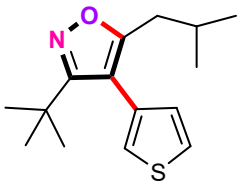

3-(4-Fluoro-3-methylphenyl)-5-phenyl-4-(thiophen-3-yl)isoxazole (**3q**): Yield: 73% as a white solid; mp = 131.3 - 132.6 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.63 (d, $J$ = 8.0 Hz, 2H), 7.46 (dd, $J$ = 4.8, 3.2 Hz, 1H), 7.44 - 7.34 (m, 4H), 7.22 (dd, $J$ = 9.4, 4.2 Hz, 2H), 7.06 - 6.93 (m, 2H), 2.26 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 165.8, 162.1 (d, $J$ = 248.3 Hz), 161.6, 131.6 (d, $J$ = 5.5 Hz), 130.0, 130.0, 129.0, 128.8, 127.8, 127.5 (d, $J$ = 8.4 Hz), 126.9, 125.5, 125.3 (d, $J$ = 17.7 Hz), 124.8 (d, $J$ = 3.7 Hz), 115.2 (d, $J$ = 22.8 Hz), 110.2, 14.5 ppm; $\nu_{max}$(KBr)/cm$^{-1}$ 3054, 2933, 1658, 1606, 1544, 1424, 1256, 758; MS (EI) m/z 77, 105, 212, 307, 335; HRMS-ESI (m/z): calcd for $C_{20}H_{15}FNOS$, $[M+H]^+$: 336.0853, found 336.0845.



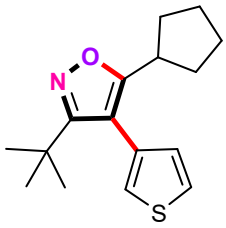

5-Butyl-3-cyclohexyl-4-(thiophen-3-yl)isoxazole (**3r**): Yield: 72% as a yellow oil; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.53 - 7.38 (m, 1H), 7.16 (d, $J$ = 1.6Hz, 1H), 7.04 (d, $J$ = 4.8 Hz, 1H), 2.86 - 2.58 (m, 3H), 1.89 (d, $J$ = 13.2 Hz, 2H), 1.78 (s, 2H), 1.73 - 1.48 (m, 5H), 1.35 (dt, $J$ = 14.8, 7.2 Hz, 2H), 1.27 (t, $J$ = 9.2 Hz, 3H), 0.89 (t, $J$ = 7.2 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 169.2, 166.3, 130.6, 128.4, 126.0, 123.5, 110.6, 35.7, 31.6, 29.8, 26.3, 25.9, 25.5, 22.3, 13.7; IR $(KBr)_{vmax}$/cm$^{-1}$: 3048, 2920, 1623, 1458, 1264, 1168, 755; MS (EI) m/z 97, 123, 165, 232, 256, 289; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{17}H_{24}NOS$, 290.1573, found 290.1567.



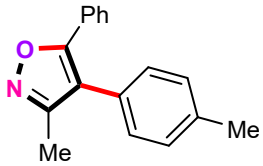

3,5-Di-*tert*-butyl-4-(thiophen-3-yl)isoxazole (**3s**): Yield: 69% as a yellow oil; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.36 (dd, $J$ = 4.8, 3.0 Hz, 1H), 7.14 (dd, $J$ = 3.0, 1.2 Hz, 1H), 6.98 (dd, $J$ = 4.8, 1.2 Hz, 1H), 1.21 (s, 9H), 1.19 (s, 9H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 176.1, 169.8, 132.1, 131.2, 126.2, 124.9, 108.3, 34.1, 33.4, 29.2, 29.1; IR $(KBr)_{vmax}$/cm$^{-1}$: 3046, 2928, 1630, 1456, 1253, 756; MS (EI) m/z 57, 123, 164, 220, 263; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{15}H_{22}NOS$, 264.1417, found 264.1411.



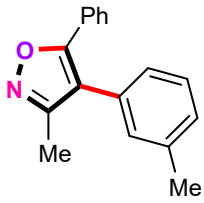
3-(*tert*-Butyl)-5-isobutyl-4-(thiophen-3-yl)isoxazole (**3t**): Yield: 78% as a yellow oil; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.37 (dd, $J$ = 4.8, 3.0 Hz, 1H), 7.12 (dd, $J$ = 3.0, 1.2 Hz, 1H), 6.96 (dd, $J$ = 4.8, 1.2 Hz, 1H), 2.37 (d, $J$ = 7.2 Hz, 2H), 1.98 (dt, $J$ = 13.6, 6.8 Hz, 1H), 1.22 (s, 9H), 0.88 (s, 3H), 0.86 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 170.1, 169.1, 131.6, 130.4, 125.7, 125.4, 111.0, 34.3, 33.2, 29.2, 27.6, 22.4; IR $(KBr)_{vmax}/cm^{-1}$: 3054, 2932, 1638, 1282, 756; MS (EI) m/z 97, 123, 164, 192, 220, 263; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{15}H_{22}NOS$, 264.1417, found, 264.1411.



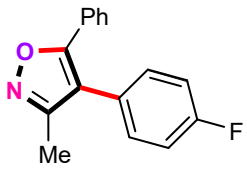
3-(*tert*-Butyl)-5-cyclopentyl-4-(thiophen-3-yl)isoxazole (**3u**): Yield: 73% as a yellow oil; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.39 (dd, $J$ = 4.8, 2.8 Hz, 1H), 7.15 (dd, $J$ = 3.2, 1.2 Hz, 1H), 6.99 (dd, $J$ = 4.8, 1.2 Hz, 1H), 2.89 (dd, $J$ = 16.0, 8.0 Hz, 1H), 2.00 - 1.68 (m, 6H), 1.69 - 1.45 (m, 2H), 1.24 (s, 9H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 173.7, 169.2, 131.7, 130.5, 125.6, 125.4, 109.4, 36.5, 33.2, 31.8, 29.2, 25.7; IR $(KBr)_{vmax}/cm^{-1}$: 3056, 2938, 1648, 1255, 756; MS (EI) m/z 60, 97, 123, 191, 260, 275; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{16}H_{22}NOS$, 276.1417, found, 276.1412.



3-Methyl-5-phenyl-4-(p-tolyl)isoxazole (**4a**): Yield: 91% as a yellow solid; mp = 92.1 - 93.0 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.58 (d, $J$ = 7.2 Hz, 2H), 7.36 (t, $J$ = 9.4 Hz, 3H), 7.27 (d, $J$ = 7.8 Hz, 2H), 7.21 (d, $J$ = 7.8 Hz, 2H), 2.45 (s, 3H), 2.27 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 164.1, 160.3, 137.9, 129.8, 129.7, 129.6, 128.6, 128.1, 127.5, 126.8, 116.2, 21.4, 10.7; IR $(KBr)_{vmax}/cm^{-1}$: 3036, 2933, 1624, 1456, 1400, 764; MS (EI) m/z 77, 96, 177, 207, 249; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{17}H_{16}NO$, 250.1226, found 250.1221.

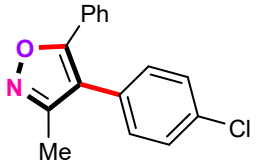



3-Methyl-5-phenyl-4-(m-tolyl)isoxazole (**4b**): Yield: 90% as a yellow oil; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.58 (d, $J$ = 7.8 Hz, 2H), 7.45 - 7.31 (m, 4H), 7.25 (d, $J$ = 7.6 Hz, 1H), 7.12 (d, $J$ = 11.6 Hz, 2H), 2.41 (s, 3H), 2.27 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 164.1, 160.2, 138.8, 130.6, 130.4, 129.6, 129.0, 128.9, 128.6, 128.0, 126.9, 126.8, 116.3, 21.5, 10.7; IR $(KBr)_{vmax}/cm^{-1}$: 3058, 2936, 1628, 1456, 1256, 756; MS (EI) m/z 77, 105, 165, 220, 249; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{17}H_{16}NO$, 250.1226, found 250.1221.

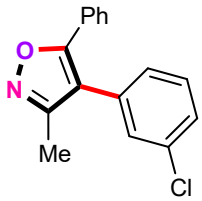



4-(4-Fluorophenyl)-3-methyl-5-phenylisoxazole (**4c**): Yield: 80% as a white solid; mp = 113.5 - 114.9 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.54 (d, $J$ = 7.6 Hz, 2H), 7.36 (p, $J$ = 6.2 Hz, 3H), 7.30 (dd, $J$ = 13.0, 4.8 Hz, 2H), 7.16 (t, $J$ = 8.4 Hz, 2H), 2.26 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$

164.5, 162.6 (d, $J$ = 247.9 Hz), 160.1, 131.6 (d, $J$ = 8.1 Hz), 129.8, 128.7, 127.7, 126.8, 126.6 (d, $J$ = 3.4 Hz), 116.2 (d, $J$ = 21.6 Hz), 115.2, 10.6; IR (KBr)$_{vmax}$/cm$^{-1}$: 3052, 2926, 1623, 1446, 1255, 754; MS (EI) m/z 77, 105, 148, 183, 224, 253; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{16}H_{13}FNO$, 254.0976, found 254.0971.

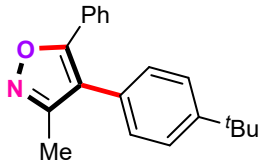



4-(4-Chlorophenyl)-3-methyl-5-phenylisoxazole (**4d**): Yield: 75% as a yellow solid; mp = 120.5 - 121.7 °C; $^1$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.54 (d, $J$ = 6.6 Hz, 2H), 7.44 (d, $J$ = 8.4 Hz, 2H), 7.37 (t, $J$ = 7.6 Hz, 3H), 7.27 (t, $J$ = 7.8 Hz, 3H), 2.26 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 164.6, 159.9, 134.2, 131.2, 129.9, 129.4, 129.1, 128.8, 127.6, 126.9, 115.0, 10.6; $\nu_{max}$(KBr)/cm$^{-1}$ 3046, 2938, 1645, 1442, 1268, 758; MS (EI) m/z 77, 105, 165, 240, 269; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{16}H_{13}ClNO$, 270.0680, found 270.0676.

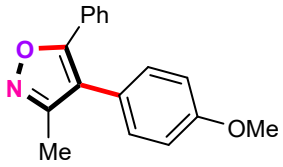



4-(3-Chlorophenyl)-3-methyl-5-phenylisoxazole (**4e**): Yield: 77% as a yellow solid; mp = 78.2 - 79.7 °C; $^1$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.58 - 7.52 (m, 2H), 7.44 - 7.35 (m, 5H), 7.33 (s, 1H), 7.23 - 7.18 (m, 1H), 2.27 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 164.8, 159.8, 134.9, 132.6, 130.4, 130.0, 129.8, 128.8, 128.4, 128.2, 127.5, 126.9, 114.9, 10.6; $\nu_{max}$(KBr)/cm$^{-1}$ 3036, 2938, 1715, 1432, 1264, 756; MS (EI) m/z 77, 105, 165, 240, 269; HRMS (ESI, m/z): $[M+H]^+$ Calcd.

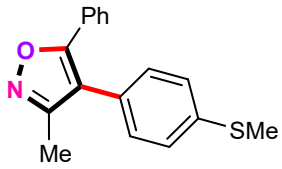
for $C_{16}H_{13}ClNO$, 270.0680, found 270.0676.



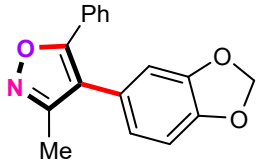

4-(4-(*tert*-Butyl)phenyl)-3-methyl-5-phenylisoxazole (**4f**): Yield: 82% as a yellow solid; mp = 66.2 - 67.7 °C; $^1$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.59 (dd, $J$ = 7.6, 2.0 Hz, 2H), 7.50 - 7.43 (m, 2H), 7.40 - 7.33 (m, 3H), 7.27 - 7.21 (m, 2H), 2.28 (s, 3H), 1.40 (s, 9H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 164.2, 160.3, 151.0, 129.6, 129.4, 128.6, 128.1, 127.5, 126.9, 125.9, 116.1, 34.7, 31.4, 10.8; $\nu_{max}$(KBr)/cm$^{-1}$ 3036, 2930, 1715, 1556, 1448, 1262, 758; MS (EI) m/z 77, 105, 165, 233, 248, 276, 291; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{20}H_{22}NO$, 292.1696, found 292.1690.



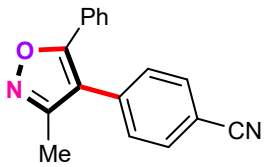

4-(4-Methoxyphenyl)-3-methyl-5-phenylisoxazole (**4g**): Yield: 80% as a yellow oil; $^1$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.58 (d, $J$ = 7.8 Hz, 2H), 7.34 (d, $J$ = 6.8 Hz, 3H), 7.23 (d, $J$ = 8.4 Hz, 2H), 7.00 (d, $J$ = 8.4 Hz, 2H), 3.88 (s, 3H), 2.26 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 164.0, 160.4, 159.4, 143.3, 131.0, 129.6, 128.6, 128.1, 126.8, 122.6, 114.6, 55.3, 10.7; $\nu_{max}$(KBr)/cm$^{-1}$ 3046, 2930, 1717, 1600, 1438, 1256, 755; MS (EI) m/z 77, 105, 153, 181, 222, 265; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{17}H_{16}NO_2$, 266.1176, found 266.1171.




3-Methyl-4-(4-(methylthio)phenyl)-5-phenylisoxazole (**4h**): Yield: 77% as a yellow solid; mp = 70.6 - 72.2 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.57 (d, $J$ = 7.2 Hz, 2H), 7.42 - 7.30 (m, 5H), 7.23 (d, $J$ = 8.0 Hz, 2H), 2.56 (s, 3H), 2.27 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 164.3, 160.2, 138.8, 130.2, 129.7, 128.7, 127.9, 127.0, 126.9, 126.6, 115.6, 15.4, 10.7; $\nu_{max}$(KBr)/cm$^{-1}$ 3048, 2928, 1682, 1445, 1246, 754; MS (EI) m/z 77, 105, 165, 238, 253, 281; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{17}H_{16}NOS$, 282.0947, found 282.0941.

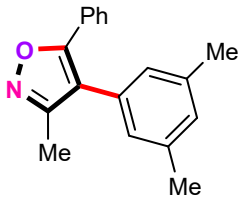



4-(Benzo[*d*][1,3]dioxol-5-yl)-3-methyl-5-phenylisoxazole (**4i**): Yield: 71% as a yellow solid; mp = 116.5 - 117.8 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.65 - 7.55 (m, 2H), 7.40 - 7.30 (m, 3H), 6.89 (d, $J$ = 8.0 Hz, 1H), 6.83 - 6.69 (m, 2H), 6.04 (s, 2H), 2.25 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 164.2, 160.3, 148.2, 147.6, 129.7, 128.7, 127.9, 126.8, 124.0, 123.5, 115.9, 110.1, 109.0, 101.4, 10.6; $\nu_{max}$(KBr)/cm$^{-1}$ 3046, 2928, 1678, 1442, 1286, 755; MS (EI) m/z 77, 105, 152, 180, 250, 279; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{17}H_{14}NO_3$, 280.0968, found 280.0965.

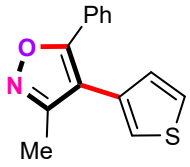



4-(3-Methyl-5-phenylisoxazol-4-yl)benzonitrile (**4j**): Yield: 65% as a yellow solid; mp = 131.3 - 131.9 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.75 (d, $J$ = 8.0 Hz, 2H), 7.49 (d, $J$ = 7.6 Hz, 2H), 7.47 -

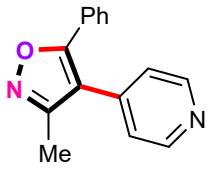
7.42 (m, 3H), 7.38 (dd, $J$ = 14.2, 6.8 Hz, 2H), 2.29 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) : $\delta$ 165.4, 159.4, 135.8, 132.9, 130.6, 130.3, 128.9, 127.2, 127.1, 118.5, 114.6, 112.0, 10.7; $\nu_{max}$(KBr)/cm$^{-1}$ 3050, 2926, 1658, 1464, 1262, 756; MS (EI) m/z 77, 105, 163, 190, 231, 260; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{17}H_{13}N_2O$, 261.1022, found 261.1017.



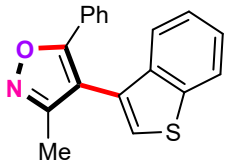

4-(3,5-Dimethylphenyl)-3-methyl-5-phenylisoxazole (**4k**): Yield: 83% as a yellow oil; $^1$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.59 (d, $J$ = 7.4 Hz, 2H), 7.41 - 7.31 (m, 3H), 7.07 (s, 1H), 6.93 (s, 2H), 2.36 (s, 6H), 2.26 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 164.0, 160.3, 138.6, 130.5, 129.8, 129.6, 128.6, 128.1, 127.5, 126.8, 116.4, 21.3, 10.7; $\nu_{max}$(KBr)/cm$^{-1}$ 3060, 2926, 1662, 1438, 1266, 759; MS (EI) m/z 77, 105, 158, 234, 263; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{18}H_{18}NO$, 264.1383, found 264.1378.



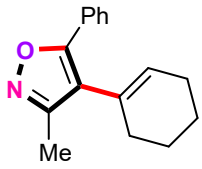

3-Methyl-5-phenyl-4-(thiophen-3-yl)isoxazole (**4l**): Yield: 85% as a yellow solid; mp = 84.5 - 85.8 °C; $^1$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.61 (d, $J$ = 7.0 Hz, 2H), 7.49 - 7.41 (m, 1H), 7.38 (d, $J$ = 6.4 Hz, 3H), 7.29 (s, 1H), 7.04 (d, $J$ = 4.8 Hz, 1H), 2.30 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 164.6, 160.2, 130.2, 129.8, 128.7, 128.5, 127.9, 126.9, 126.5, 124.4, 111.4, 10.8; $\nu_{max}$(KBr)/cm$^{-1}$ 3060, 2928, 1720, 1456, 1269, 756; MS (EI) m/z 77, 105, 136, 171, 212, 241; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{14}H_{12}NOS$, 242.0634, found 242.0630.



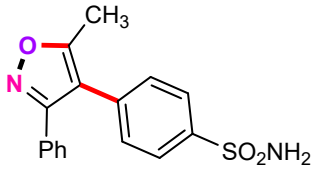

3-Methyl-5-phenyl-4-(pyridin-4-yl)isoxazole (**4m**): Yield: 52% as a yellow solid; mp = 95.7 - 97.4 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 8.70 (s, 1H), 8.61 (s, 1H), 7.70 (d, $J$ = 7.8 Hz, 1H), 7.52 (d, $J$ = 7.6 Hz, 2H), 7.48 - 7.32 (m, 4H), 2.30 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 164.2, 160.4, 143.8, 141.2, 130.5, 129.6, 129.4, 128.3, 126.7, 112.7, 10.8; $\nu_{max}$(KBr)/cm$^{-1}$ 3066, 2929, 1708, 1445, 1268, 755; MS (EI) m/z 77, 105, 166, 207, 236; HRMS (ESI, m/z): $[M-H]^+$ Calcd. for $C_{15}H_{13}N_2O$, 235.0877, found 235.0873.



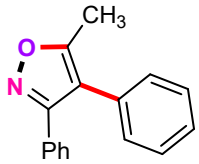

4-(Benzo[*b*]thiophen-3-yl)-3-methyl-5-phenylisoxazole (**4n**): Yield: 63% as a yellow solid; mp = 137.6 - 139.3 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.98 (d, $J$ = 8.4 Hz, 1H), 7.54 (d, $J$ = 7.6 Hz, 2H), 7.44 (dd, $J$ = 9.6, 6.8 Hz, 3H), 7.34 (d, $J$ = 7.7 Hz, 2H), 7.29 (dd, $J$ = 15.0, 7.6 Hz, 2H), 2.22 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 165.5, 161.1, 140.2, 138.0, 129.9, 128.7, 127.6, 126.6, 126.5, 125.7, 124.9, 124.7, 123.0, 122.8, 109.6, 10.6; $\nu_{max}$(KBr)/cm$^{-1}$ 3043, 2933, 1718, 1446, 1268, 758; MS (EI) m/z 77, 105, 186, 207, 262, 291; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{18}H_{14}NOS$, 292.0791, found 292.0785.



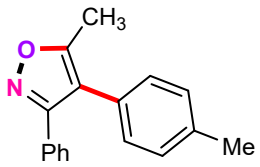

4-(Cyclohex-1-en-1-yl)-3-methyl-5-phenylisoxazole (**4o**): Yield: 66% as a yellow oil; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.79 (d, $J$ = 7.2 Hz, 2H), 7.55 - 7.35 (m, 3H), 5.82 (s, 1H), 2.29 - 2.21 (m, 5H), 2.15 - 2.07 (m, 2H), 1.79 - 1.71 (m, 4H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 163.2, 160.1, 130.3, 129.4, 128.7, 128.5, 127.9, 126.4, 118.1, 28.9, 25.6, 23.0, 21.9, 10.3; $\nu_{max}$(KBr)/cm$^{-1}$ 3063, 2928, 1649, 1434, 1260, 749; MS (EI) m/z 77, 105, 170, 196, 207, 239; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{16}H_{18}NO$, 240.1383, found 240.1378.



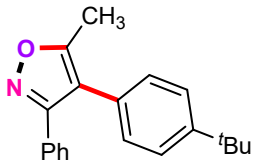

4-(5-Methyl-3-phenylisoxazol-4-yl)benzenesulfonamide (**8**): Yield: 77% as a yellow solid; mp = 158.2 - 159.7 °C; $^{1}$H NMR (400 MHz, DMSO) $\delta$ 7.92 - 7.81 (m, 2H), 7.52 - 7.40 (m, 7H), 7.39 - 7.28 (m, 2H), 3.42 (s, 2H), 2.47 (s, 3H); $^{13}$C NMR (100 MHz, DMSO): $\delta$ 168.1, 161.2, 143.7, 133.8, 130.5, 130.3, 129.3, 128.8, 128.7, 126.6, 114.7, 11.8; $\nu_{max}$(KBr)/cm$^{-1}$ 3341, 3036, 2928, 1722, 1586, 1454, 1269, 758; MS (EI) m/z 77, 103, 191, 272, 299, 314; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{16}H_{15}N_2O_3S$, 315.0798, found 315.0792.



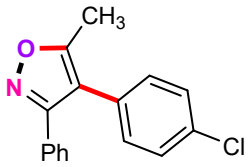

5-Methyl-3,4-diphenylisoxazole (**9a**): Yield: 90% as a yellow solid; mp = 95.1 - 96.0 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.50 - 7.45 (m, 2H), 7.42 - 7.31 (m, 6H), 7.25 - 7.17 (m, 2H), 2.48 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 166.6, 161.2, 130.4, 129.8, 129.4, 129.2, 128.7, 128.5, 127.7, 115.8, 11.6; $\nu_{max}$(KBr)/cm$^{-1}$ 3044, 2935, 1720, 1568, 1444, 1264, 757; MS (EI) m/z 77, 89, 165, 193, 220, 235; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{16}H_{14}NO$, 236.1070, found 236.1066.



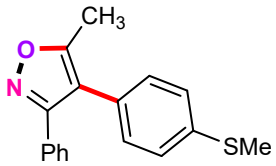

5-Methyl-3-phenyl-4-(*p*-tolyl)isoxazole (**9b**): Yield: 88% as a yellow oil; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.49 (d, $J$ = 7.2 Hz, 2H), 7.42 - 7.31 (m, 3H), 7.21 (d, $J$ = 7.8 Hz, 2H), 7.10 (d, $J$ = 8.0 Hz, 2H), 2.47 (s, 3H), 2.41 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 166.5, 161.2, 137.4, 129.7, 129.5, 129.3, 128.5, 127.3, 115.7, 21.3, 11.6; $\nu_{max}$(KBr)/cm$^{-1}$ 3054, 2932, 1717, 1558, 1446, 1260, 758; MS (EI) m/z 77, 105, 165, 207, 234, 249; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{17}H_{16}NO$, 250.1226, found 250.1221.



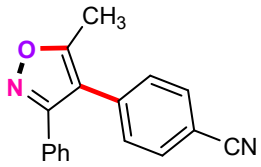
4-(4-(*tert*-Butyl)phenyl)-5-methyl-3-phenylisoxazole (**9c**): Yield: 91% as a yellow oil; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.51 - 7.46 (m, 2H), 7.43 - 7.31 (m, 5H), 7.19 - 7.08 (m, 2H), 2.48 (s, 3H), 1.37 (s, 9H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 166.5, 161.3, 150.6, 129.4, 129.3, 129.2, 128.5, 128.4, 127.2, 125.6, 115.6, 34.6, 31.3, 11.7; $\nu_{max}$(KBr)/cm$^{-1}$ 3056, 2938, 1722, 1566, 1446, 1266, 754; MS (EI) m/z 77, 115, 234, 276, 291; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{22}H_{20}NO$, 292.1696, found 292.1691.



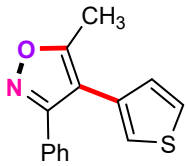

4-(4-Chlorophenyl)-5-methyl-3-phenylisoxazole (**9d**): Yield: 75% as a yellow oil; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.48 - 7.42 (m, 2H), 7.42 - 7.32 (m, 5H), 7.20 - 7.10 (m, 2H), 2.47 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 166.7, 161.1, 133.8, 131.1, 129.5, 129.0, 128.9, 128.8, 128.6, 128.4, 114.8, 11.6; $\nu_{max}$(KBr)/cm$^{-1}$ 3060, 2934, 1720, 1560, 1446, 1264, 755; MS (EI) m/z 77, 123, 165, 227, 254, 269; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{16}H_{13}ClNO$, 270.0680, found 270.0676.



5-Methyl-4-(4-(methylthio)phenyl)-3-phenylisoxazole (**9e**): Yield: 81% as a yellow solid; mp = 75.4 - 76.7 °C; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.47 (d, $J$ = 6.8 Hz, 2H), 7.42 - 7.32 (m, 3H), 7.26 (d, $J$ = 8.2 Hz, 2H), 7.11 (d, $J$ = 8.2 Hz, 2H), 2.52 (s, 3H), 2.47 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 166.6, 161.1, 138.2, 130.2, 129.4, 129.1, 128.5, 128.4, 126.9, 126.4, 115.2, 15.5, 11.6; $\nu_{max}$(KBr)/cm$^{-1}$ 3036, 2924, 1716, 1566, 1448, 1260, 758; MS (EI) m/z 77, 135, 150, 239, 266, 281; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{17}H_{16}NOS$, 282.0947, found 282.0943.

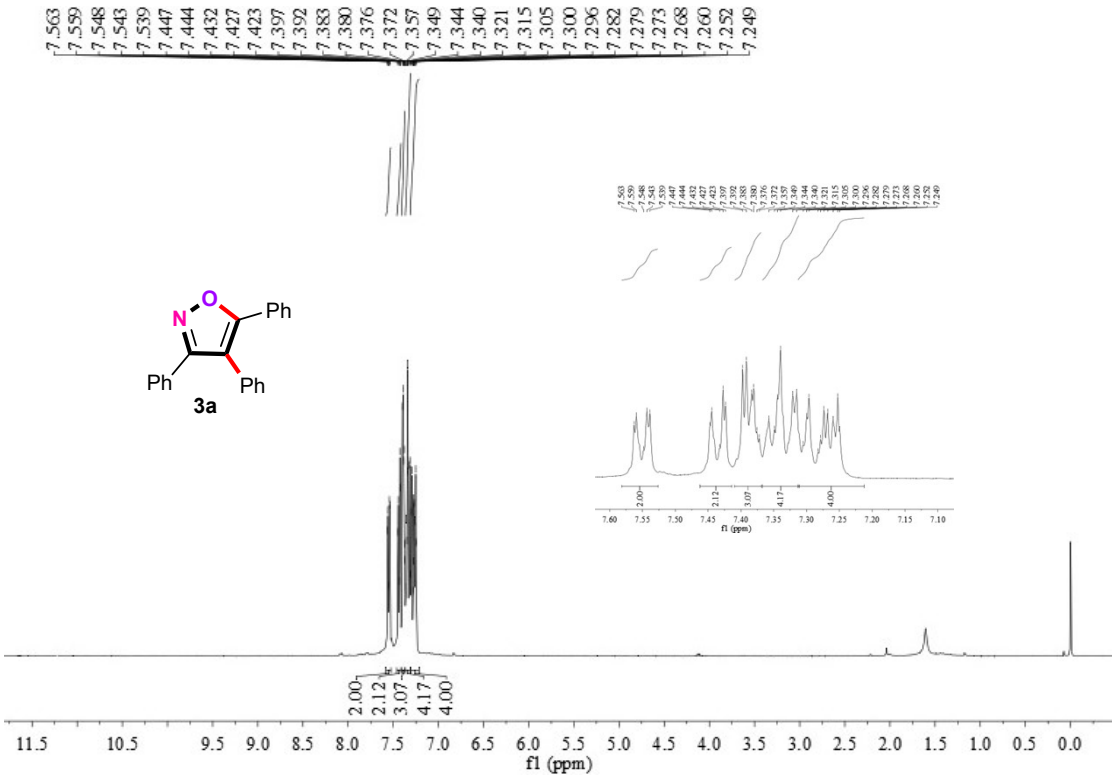



4-(5-Methyl-3-phenylisoxazol-4-yl)benzonitrile (**9f**): Yield: 70% as a yellow oil; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.71 - 7.64 (m, 2H), 7.48 - 7.34 (m, 5H), 7.33 - 7.29 (m, 2H), 2.51 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 167.3, 161.1, 135.5, 132.5, 130.3, 129.8, 128.8, 128.5, 128.4, 118.5, 114.5, 111.5, 11.8; $\nu_{max}$(KBr)/cm$^{-1}$ 3044, 2928, 2223, 1717, 1566, 1445, 1262, 756; MS (EI) m/z 77, 114, 190, 218, 245, 260; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{17}H_{13}N_2O$, 261.1022, found 261.1018.



5-Methyl-3-phenyl-4-(thiophen-3-yl)isoxazole (**9g**): Yield: 73% as a yellow oil; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.56 - 7.48 (m, 2H), 7.46 - 7.33 (m, 4H), 7.15 (dd, $J$ = 4.8, 1.2 Hz, 1H), 6.90 (dd, $J$ = 5.0, 1.2 Hz, 1H), 2.50 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 166.6, 161.3, 130.2, 129.5, 129.2, 128.5, 128.4, 128.4, 126.0, 123.9, 111.1, 11.8; $\nu_{max}$(KBr)/cm$^{-1}$ 3034, 2932, 1715, 1547, 1412, 1268, 756; MS (EI) m/z 77, 95, 171, 199, 226, 241; HRMS (ESI, m/z): $[M+H]^+$ Calcd. for $C_{14}H_{12}NOS$, 242.0634, found 242.0629.

## $^{1}$H and $^{13}$C NMR spectra of all compounds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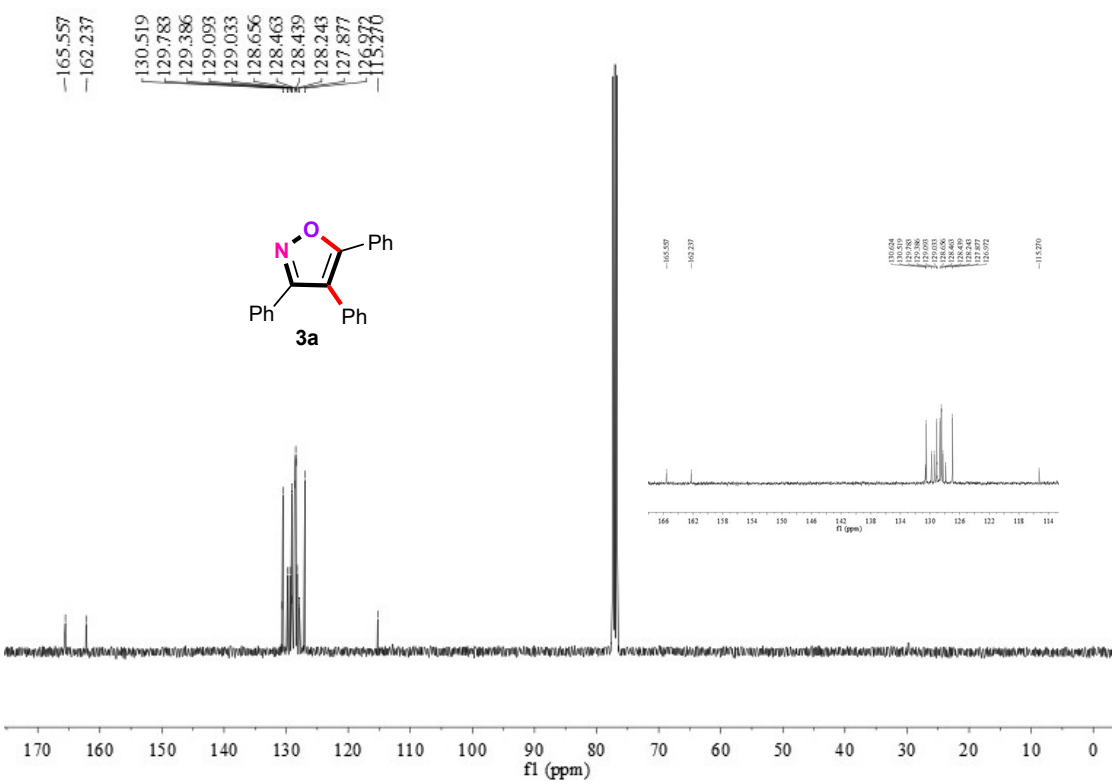
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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128.656
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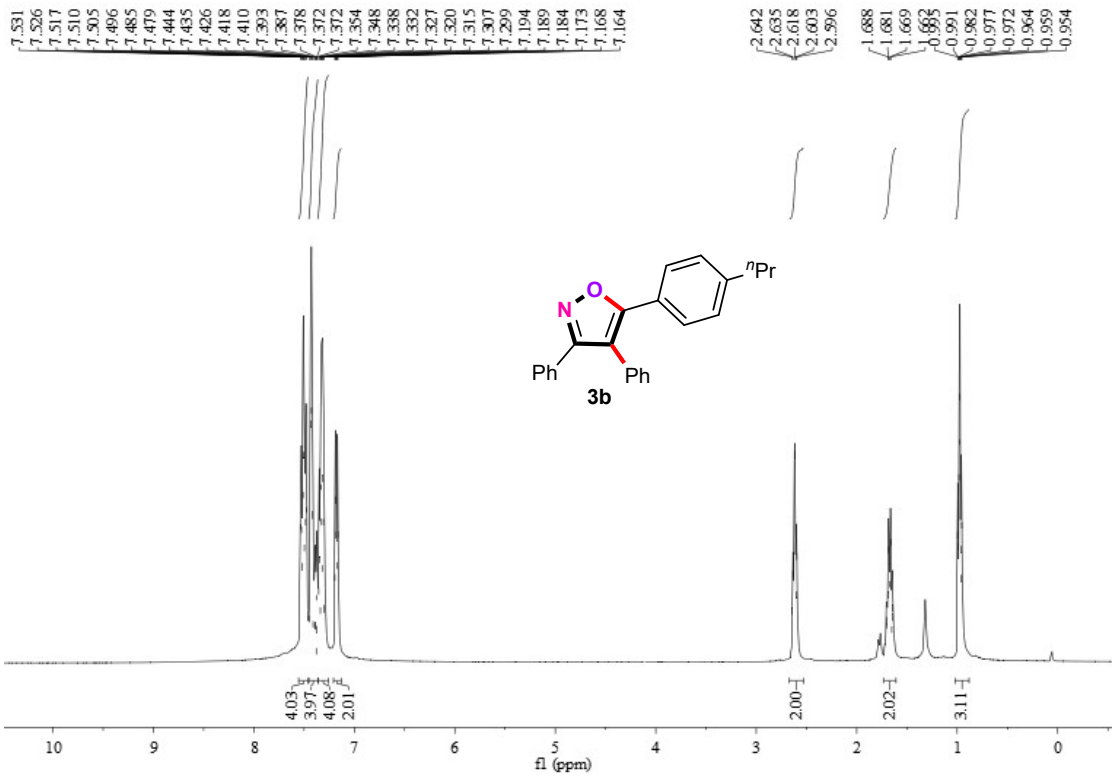
128.243
127.877
126.972
115.270
Ph
Ph
Ph
3a
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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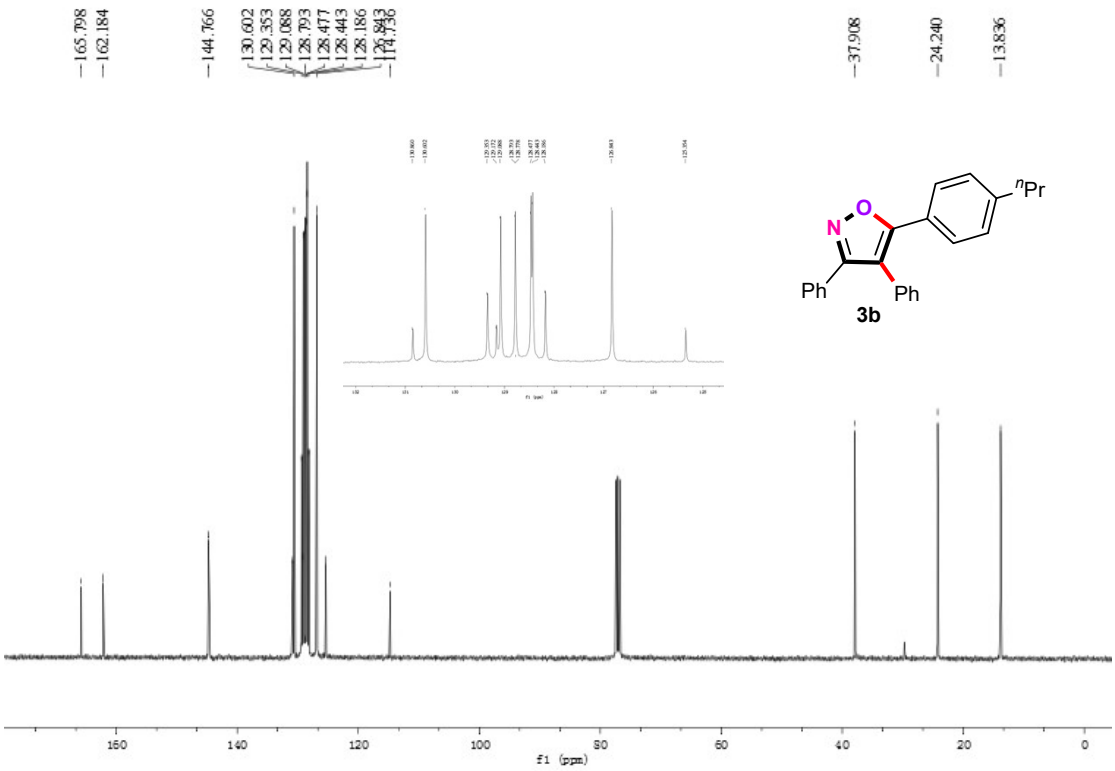
7.410
7.393
7.387
7.378
7.372
7.372
7.354
7.348
7.338
7.332
7.327
7.320
7.315
7.307
7.299
7.194
7.189
7.184
7.173
7.168
7.164
2.642
2.635
2.618
2.603
2.596
1.688
1.681
1.669
0.991
0.982
0.977
0.972
0.964
0.959
0.954
nPr
N
O
Ph
Ph
3b
4.03
3.97
4.08
2.01
2.00
2.02
3.11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)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37.908
24.240
13.836
nPr
N
O
Ph
Ph
3b
160
140
120
100
80
60
40
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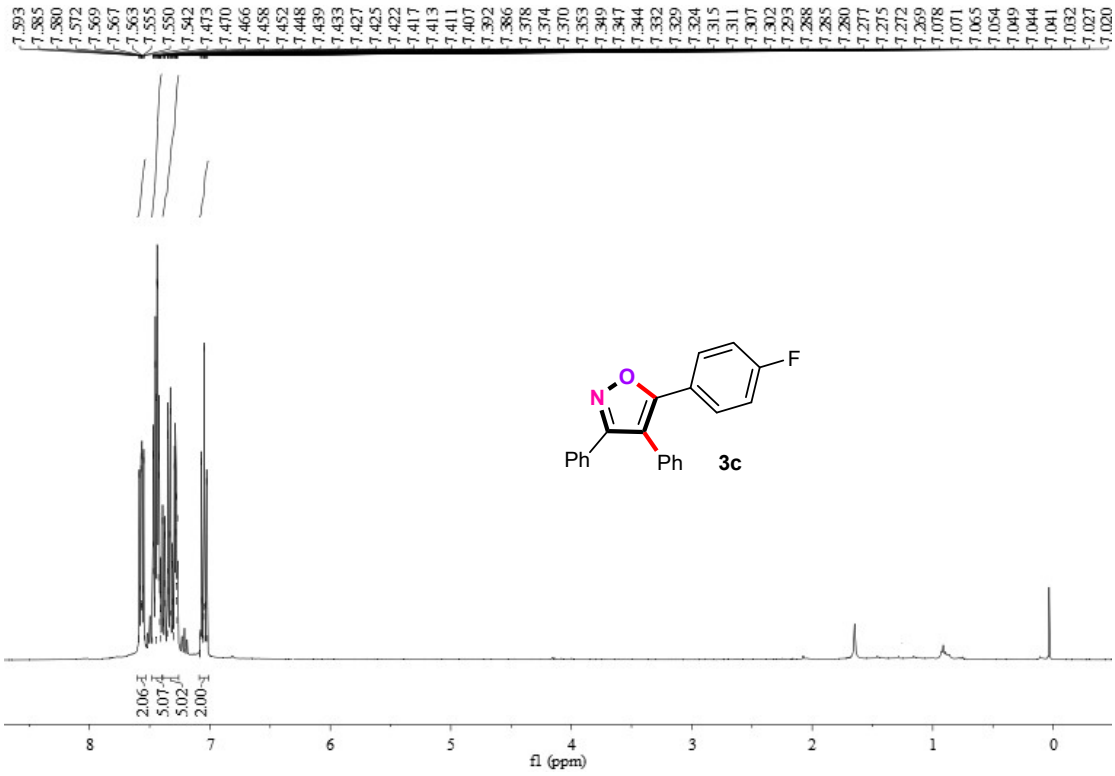
0
f1 (ppm)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7.555
7.550
7.542
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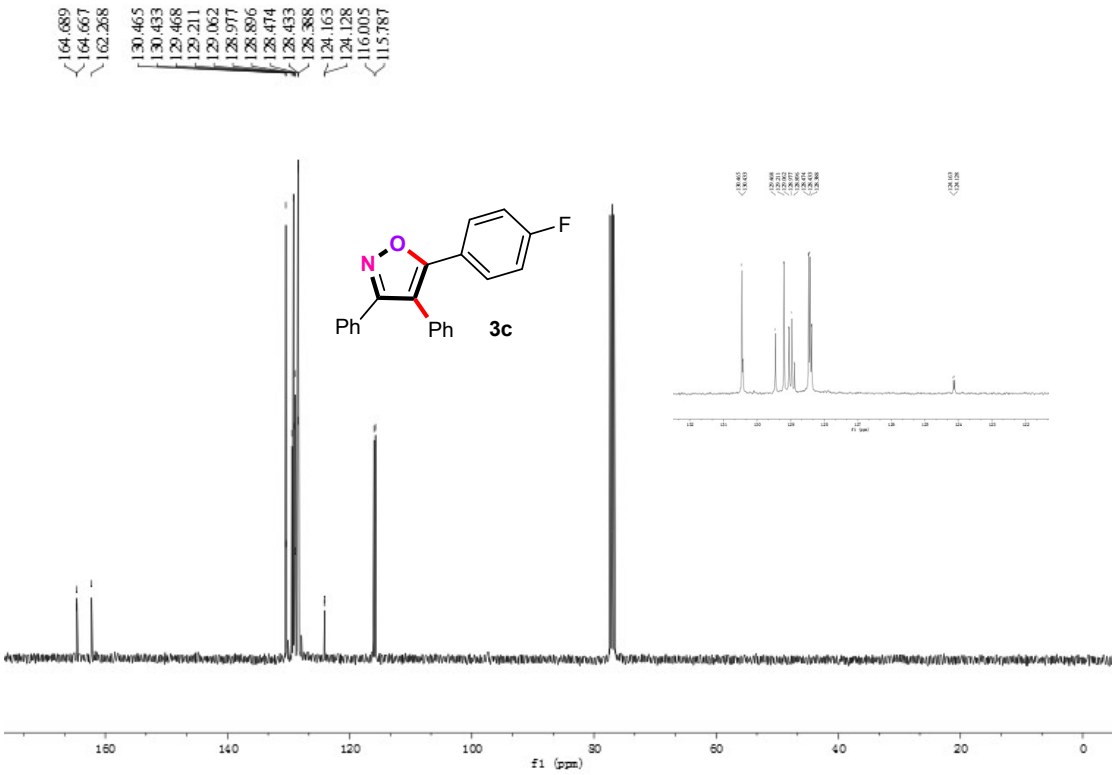
7.466
7.458
7.452
7.448
7.439
7.433
7.427
7.425
7.422
7.417
7.413
7.411
7.407
7.392
7.386
7.378
7.374
7.370
7.353
7.349
7.347
7.344
7.332
7.329
7.324
7.315
7.311
7.307
7.302
7.293
7.288
7.285
7.280
7.277
7.275
7.272
7.269
7.078
7.071
7.065
7.054
7.049
7.044
7.041
7.032
7.027
7.020
2.06
5.07
5.02
2.00
8
7
6
5
4
3
2
1
0
f1 (ppm)
N
O
F
Ph
Ph
3c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130.433
129.468
129.211
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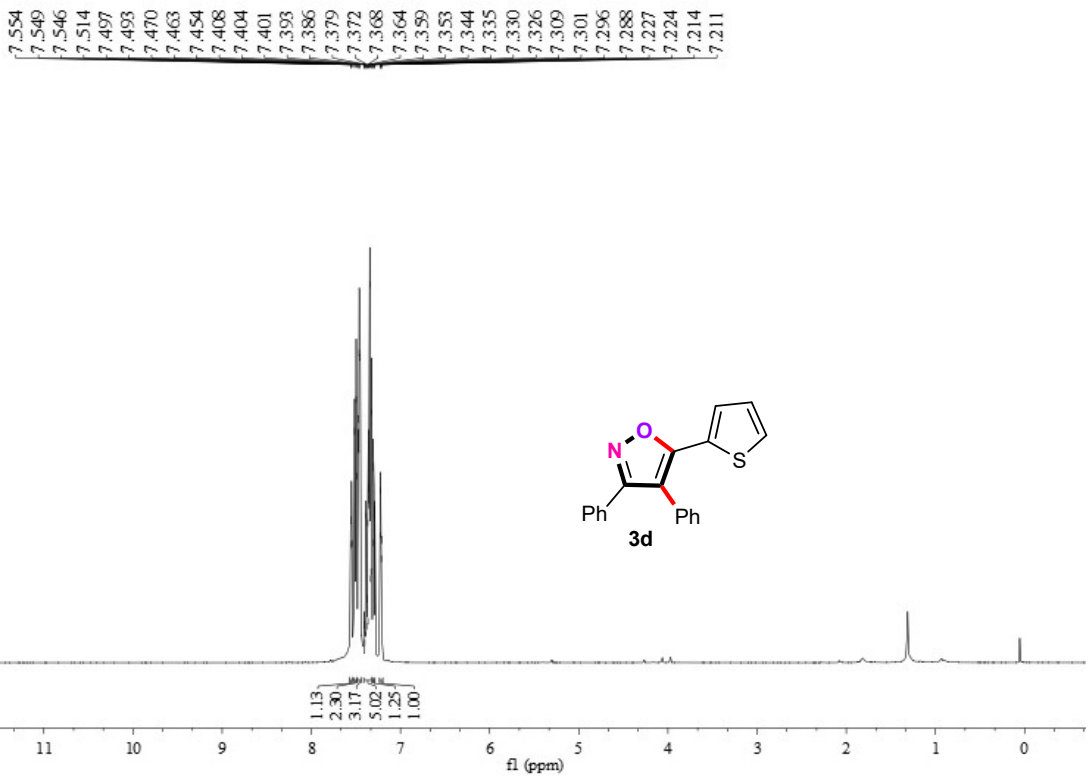
128.433
128.368
124.163
124.128
116.005
115.787
160
140
120
100
80
60
40
20
0
f1 (ppm)
N
O
F
Ph
Ph
3c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7.546
7.514
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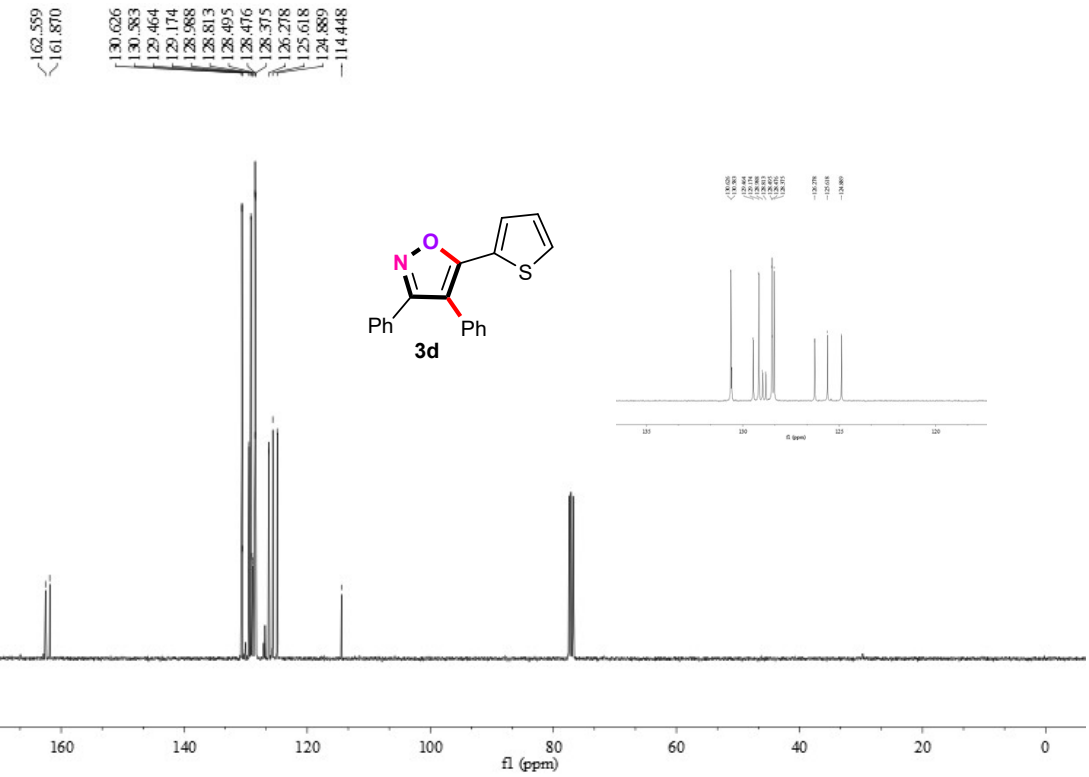
7.493
7.470
7.463
7.454
7.408
7.404
7.401
7.393
7.386
7.379
7.372
7.368
7.364
7.359
7.353
7.344
7.335
7.330
7.326
7.309
7.301
7.296
7.288
7.227
7.224
7.214
7.211
1.13
2.30
3.17
5.02
1.25
1.00
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)
N
O
S
Ph
Ph
3d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130.626
130.383
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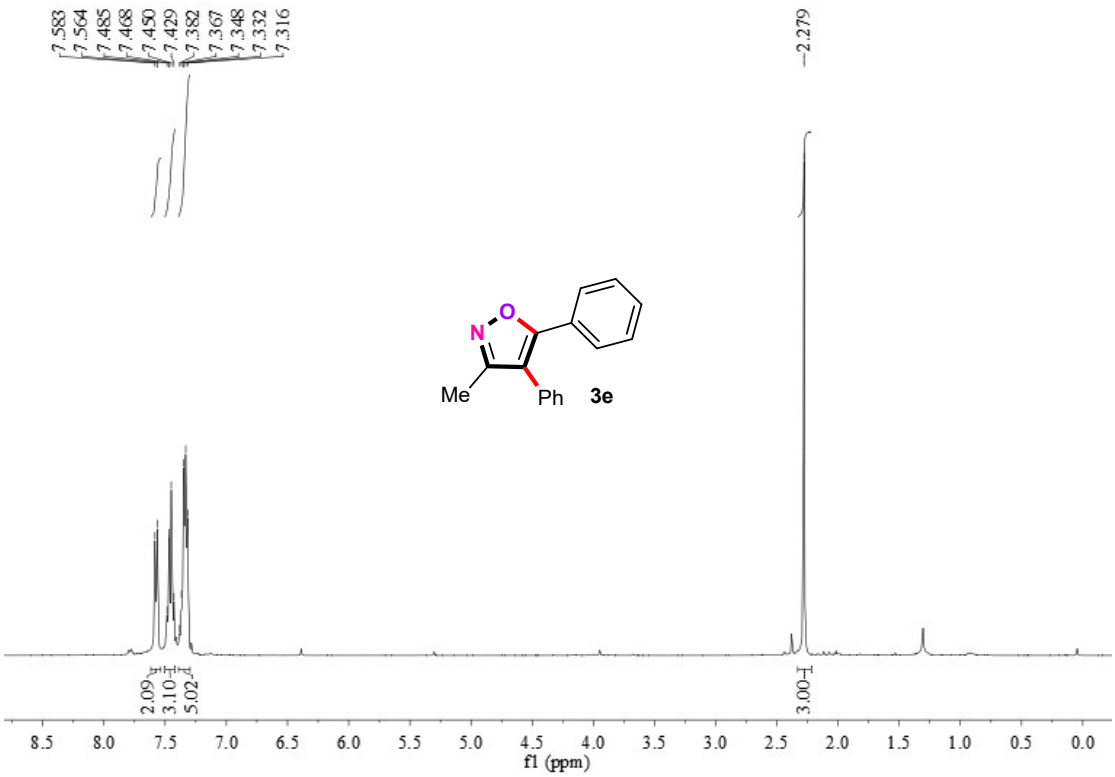
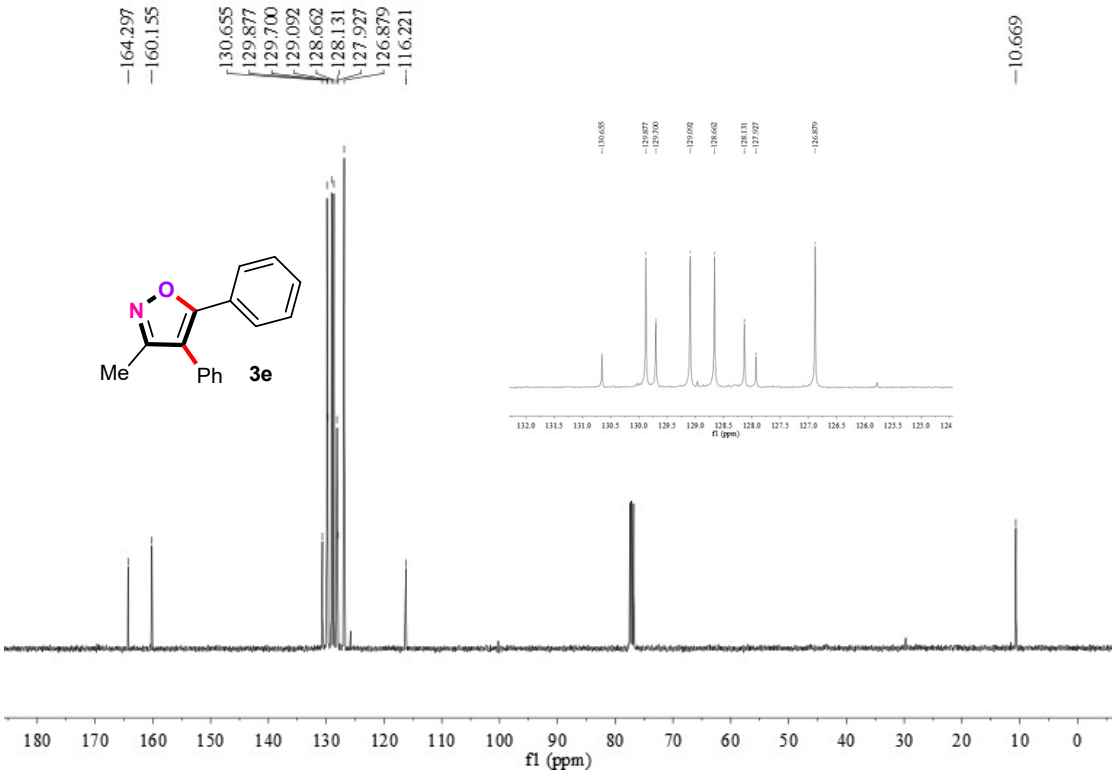
129.174
128.988
128.813
128.495
128.476
128.375
126.278
125.618
124.889
114.448
160
140
120
100
80
60
40
20
0
f1 (ppm)
N
O
S
Ph
Ph
3d


7.583
7.564
7.485
7.468
7.450
7.429
7.382
7.367
7.348
7.332
7.316
2.279
N
O
Me
Ph
3e
2.09
3.10
5.02
3.00
f1 (ppm)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130.655
129.877
129.700
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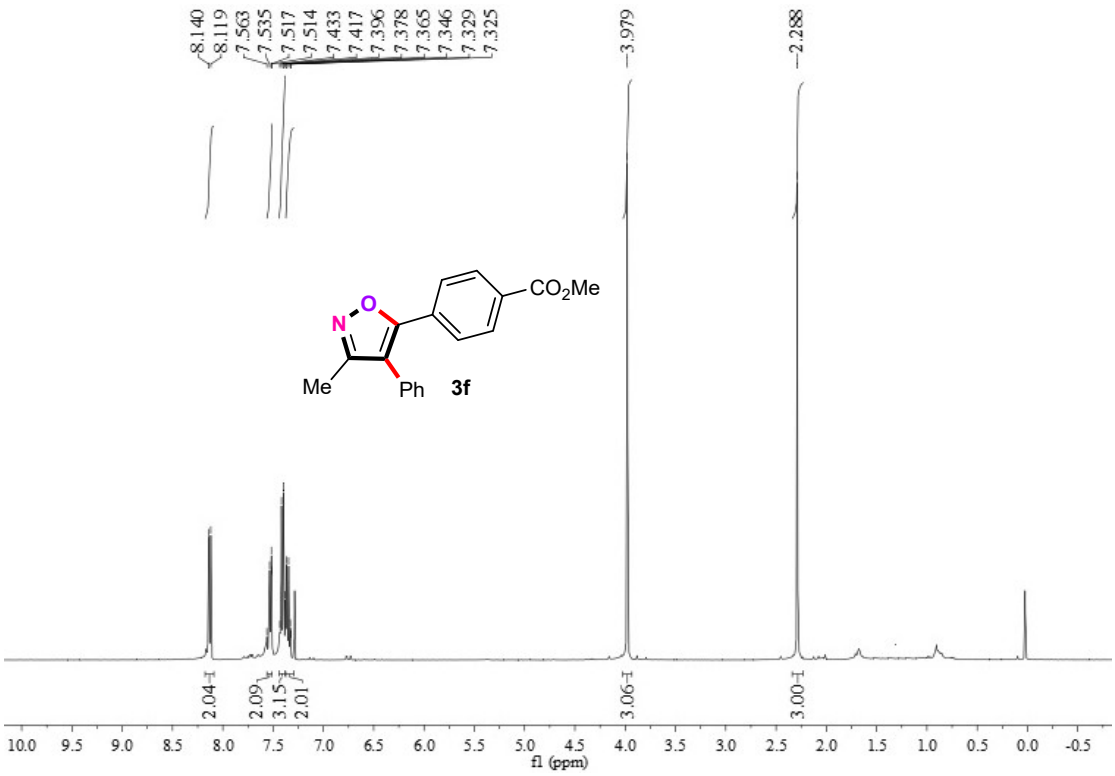
128.131
127.927
126.879
116.221
10.669
N
O
Me
Ph
3e
f1 (ppm)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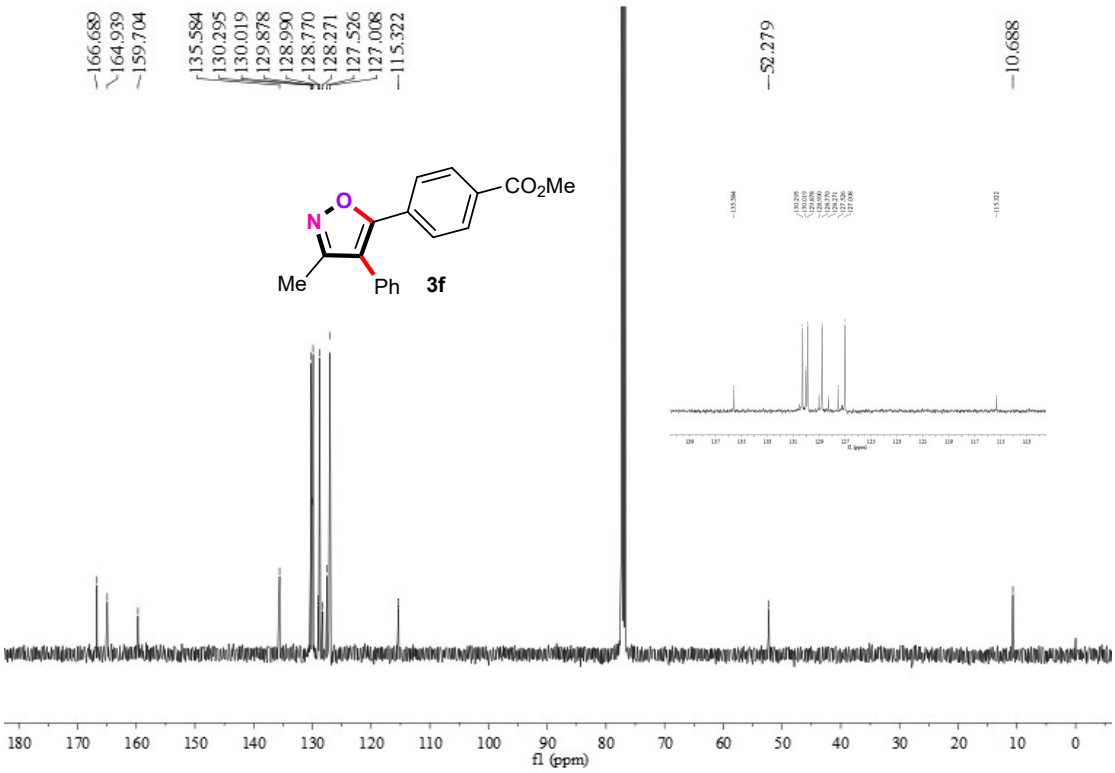
8.119
7.563
7.535
7.517
7.514
7.433
7.417
7.396
7.378
7.365
7.346
7.329
7.325
3.979
2.288
CO2Me
N
O
Me
Ph
3f
2.04
2.09
3.15
2.01
3.06
3.00
f1 (ppm)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130.019
129.878
128.990
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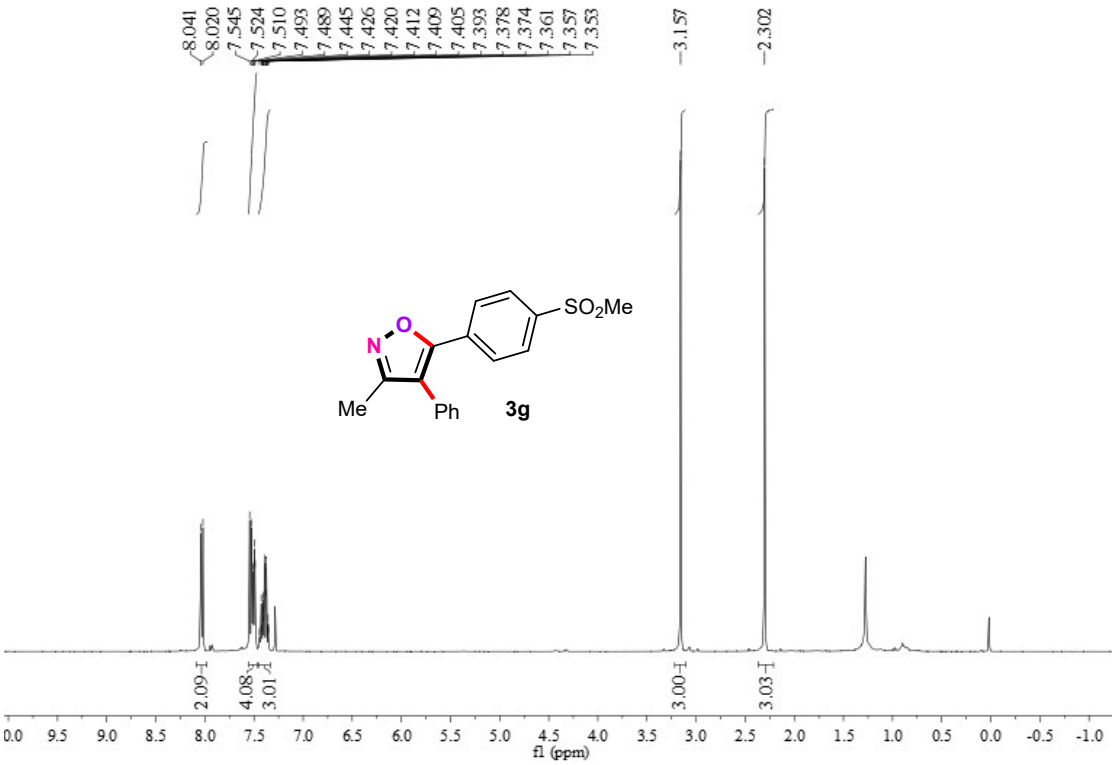
127.008
115.322
52.279
10.688
CO2Me
N
O
Me
Ph
3f
f1 (ppm)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8.020
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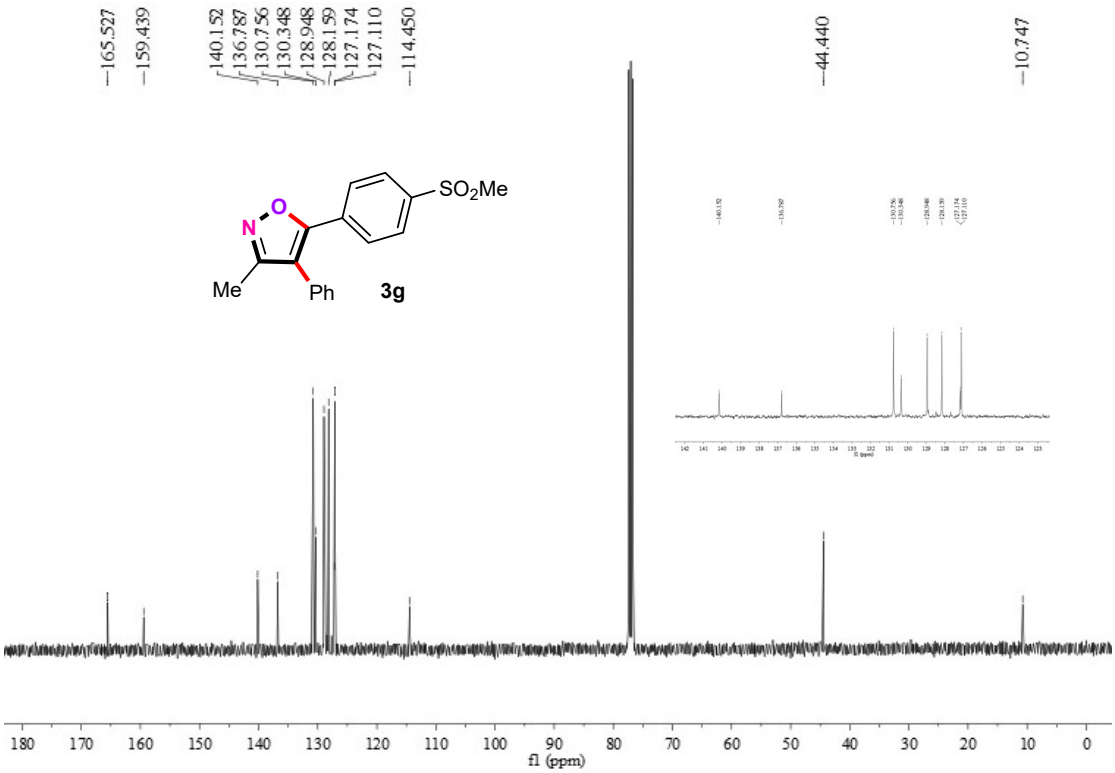
7.524
7.510
7.493
7.489
7.445
7.426
7.420
7.412
7.409
7.405
7.393
7.378
7.374
7.361
7.357
7.353
3.157
2.302
SO2Me
N
O
Me
Ph
3g
2.09
4.08
3.01
3.00
3.03
f1 (ppm)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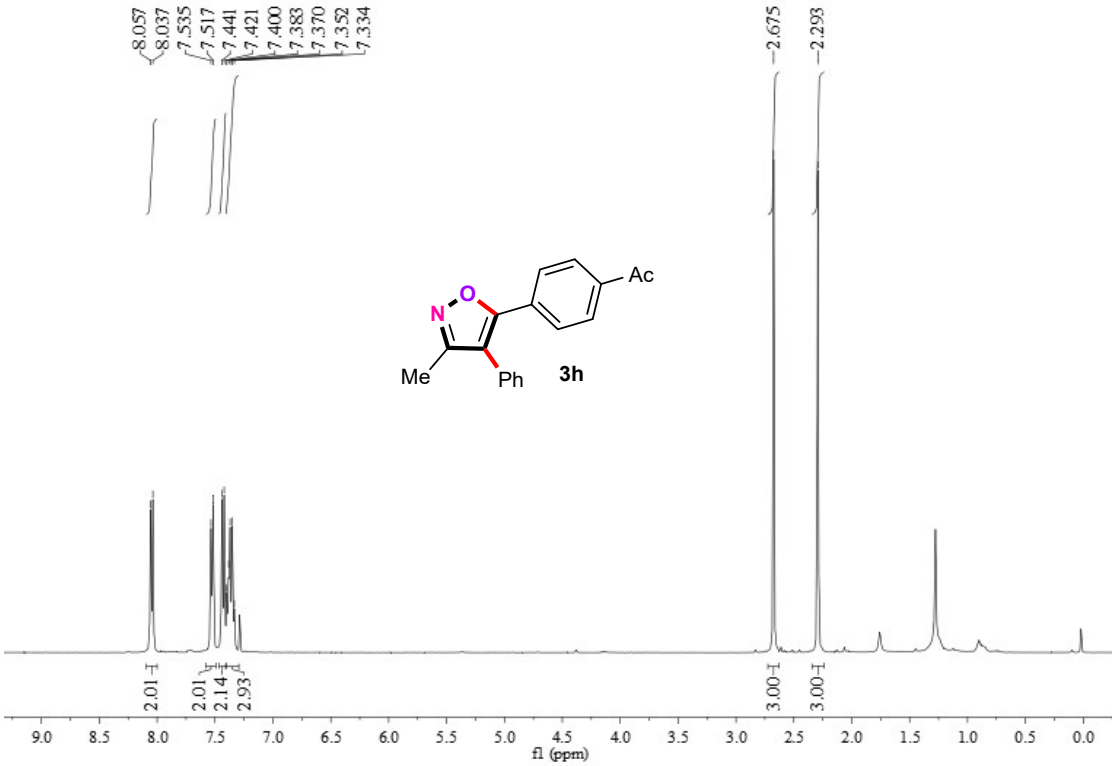
159.439
140.152
136.787
130.756
130.348
128.948
128.159
127.174
127.110
114.450
44.440
10.747
SO2Me
N
O
Me
Ph
3g
f1 (ppm)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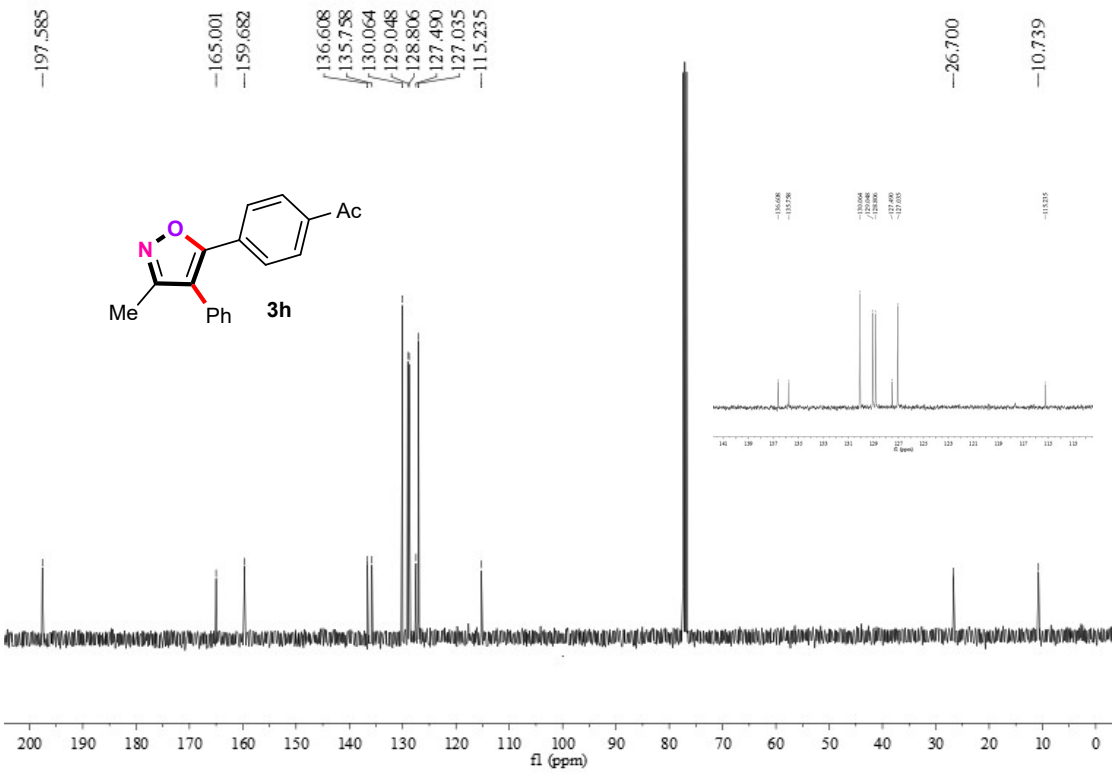
8.037
7.535
7.517
7.441
7.421
7.400
7.383
7.370
7.352
7.334
2.675
2.293
Ac
N
O
Me
Ph
3h
2.01
2.01
2.14
2.93
3.00
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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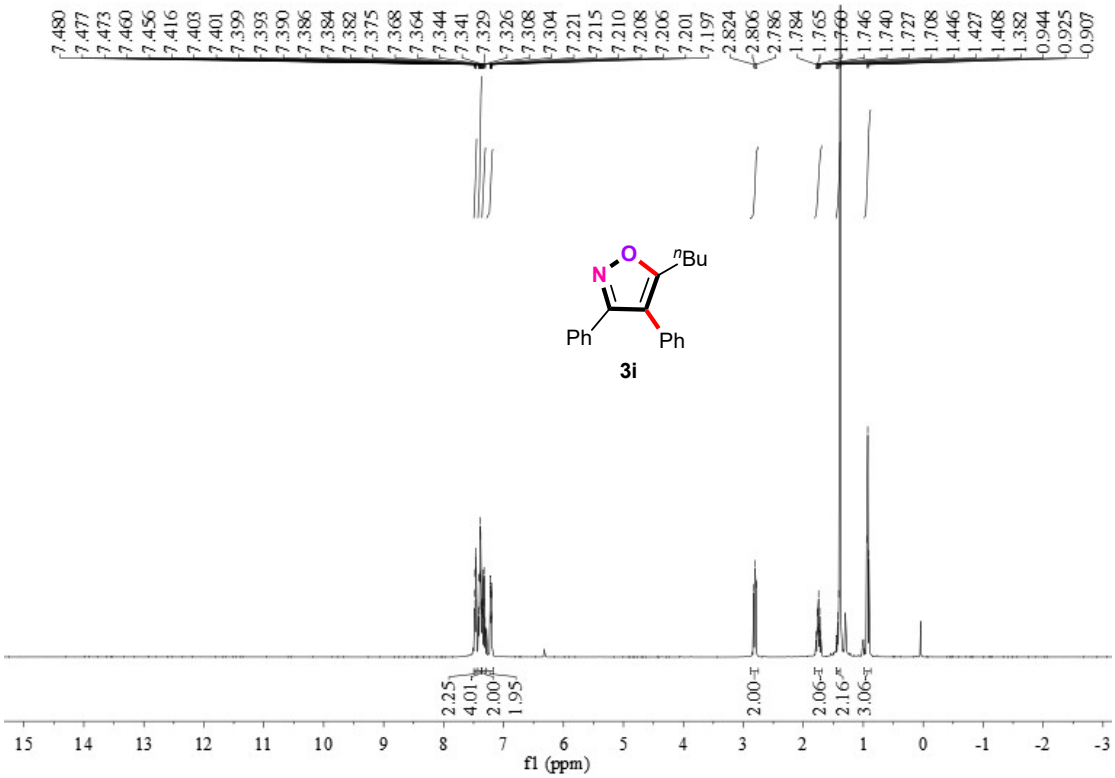
135.758
130.064
129.048
128.806
127.490
127.035
115.235
26.700
10.739
Ac
N
O
Me
Ph
3h
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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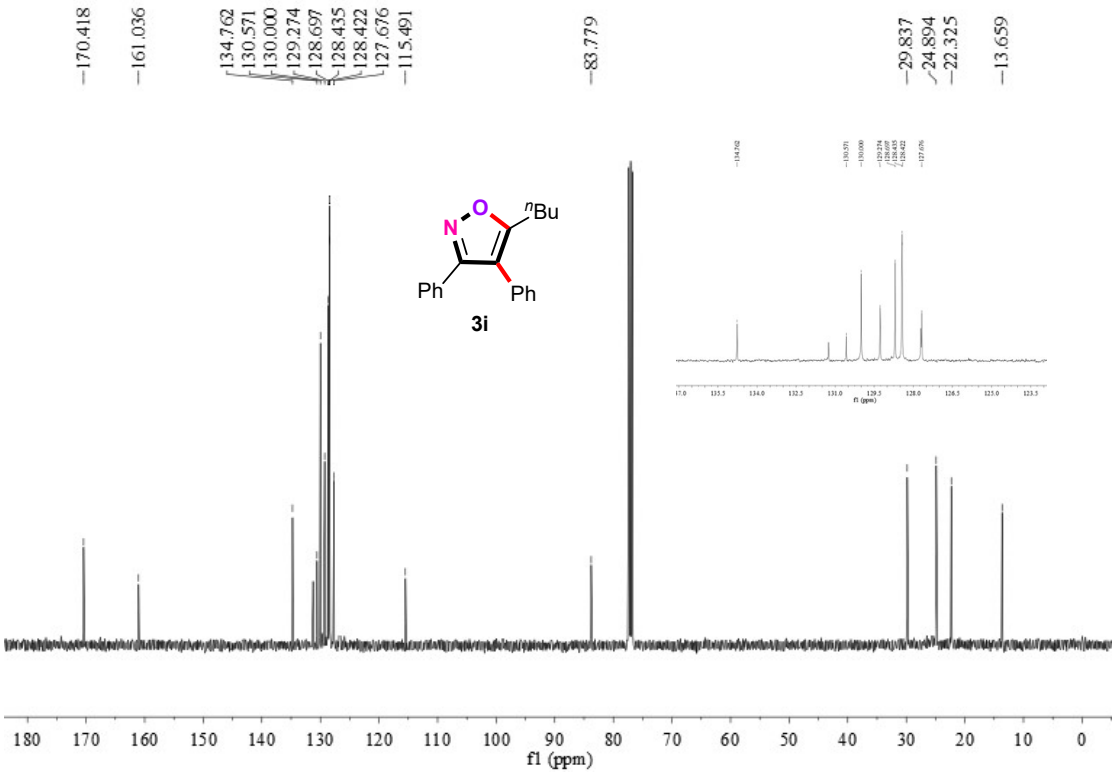
7.401
7.399
7.393
7.390
7.386
7.384
7.382
7.375
7.368
7.364
7.344
7.341
7.329
7.326
7.308
7.304
7.221
7.215
7.210
7.208
7.206
7.201
7.197
2.824
2.806
2.786
1.784
1.765
1.760
1.746
1.740
1.727
1.708
1.446
1.427
1.408
1.382
0.944
0.925
0.907
O
N
nBu
Ph
Ph
3i
2.25
4.01
2.00
1.95
2.00
2.06
2.16
3.06
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)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129.274
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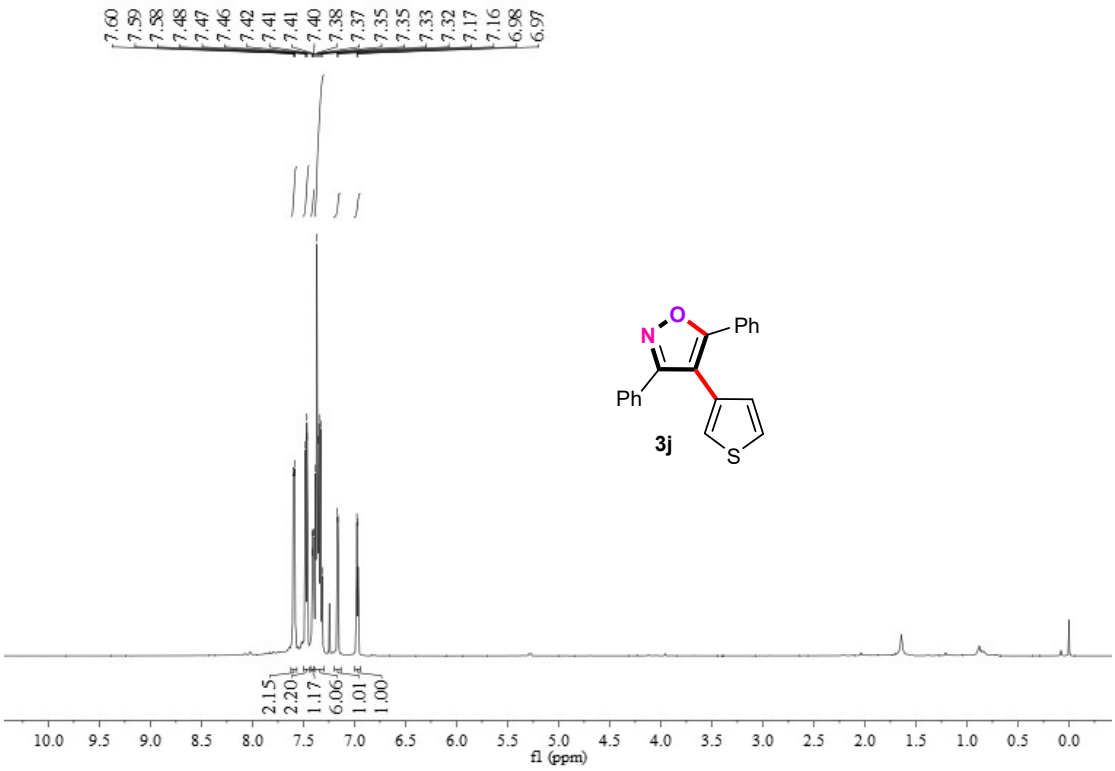
128.435
128.422
127.676
115.491
83.779
29.837
24.894
22.325
13.659
O
N
nBu
Ph
Ph
3i
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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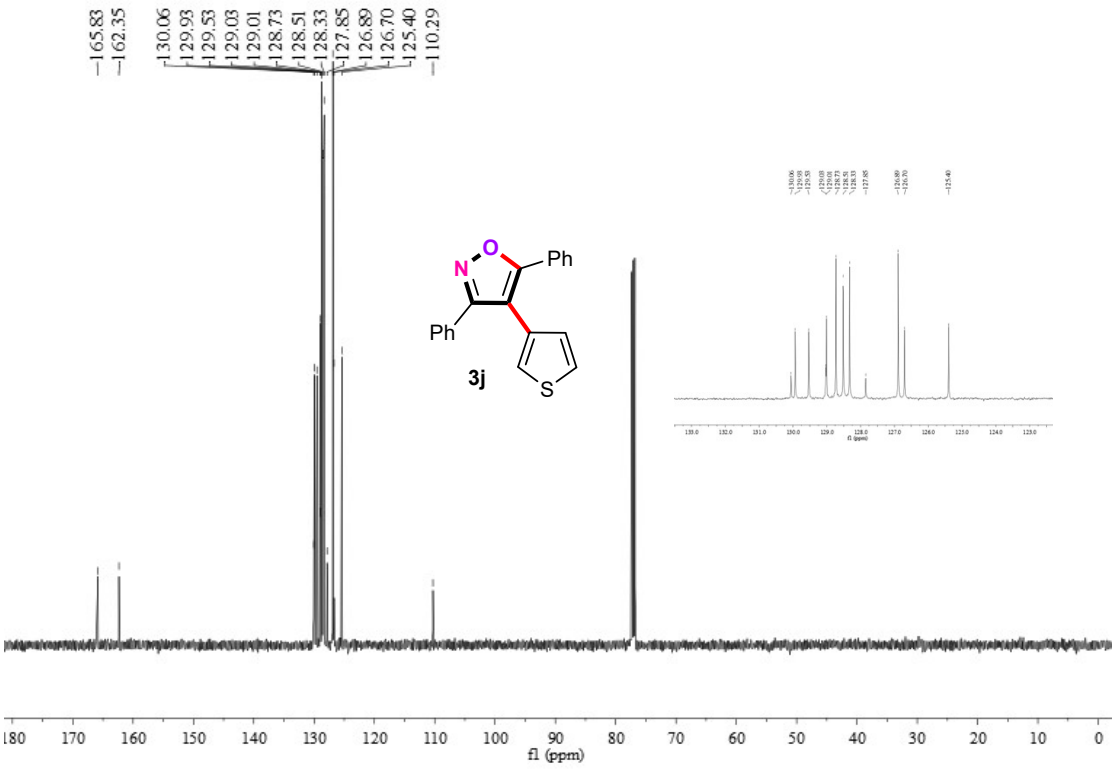
7.41
7.41
7.40
7.38
7.37
7.35
7.35
7.33
7.32
7.17
7.16
6.98
6.97
2.15
2.20
1.17
6.06
1.01
1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
N
O
Ph
Ph
S
3j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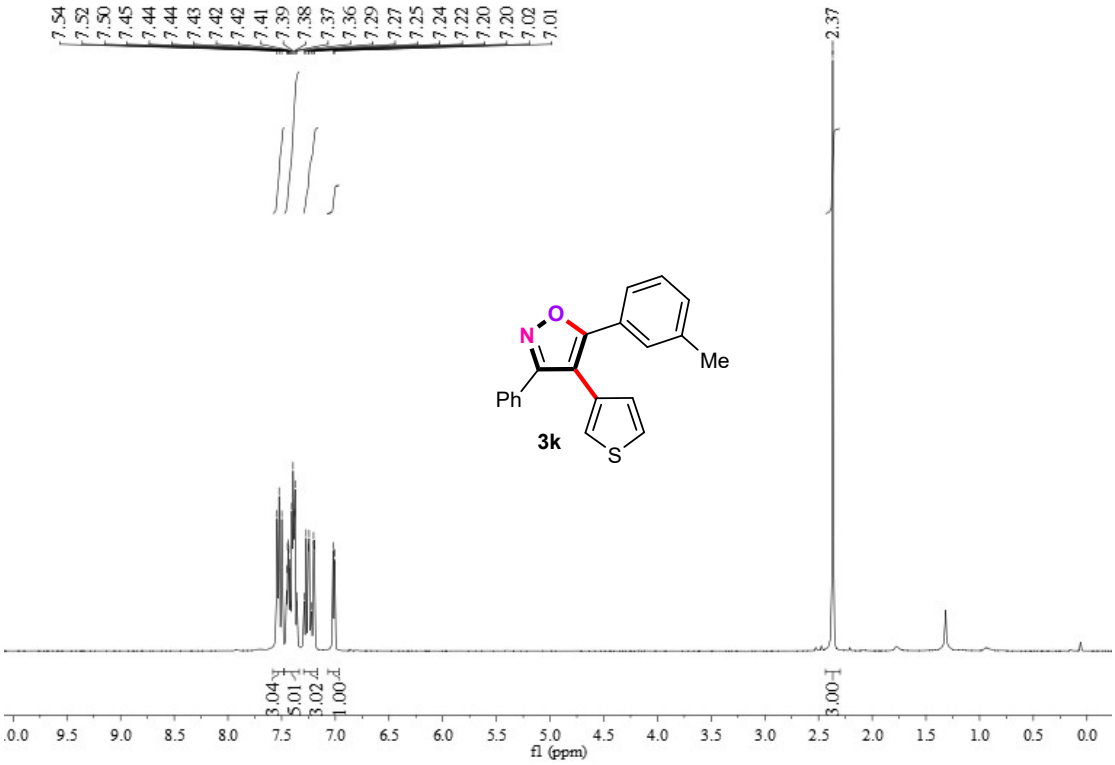
129.53
129.03
129.01
128.73
128.51
128.33
127.85
126.89
126.70
125.40
110.29
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
N
O
Ph
Ph
S
3j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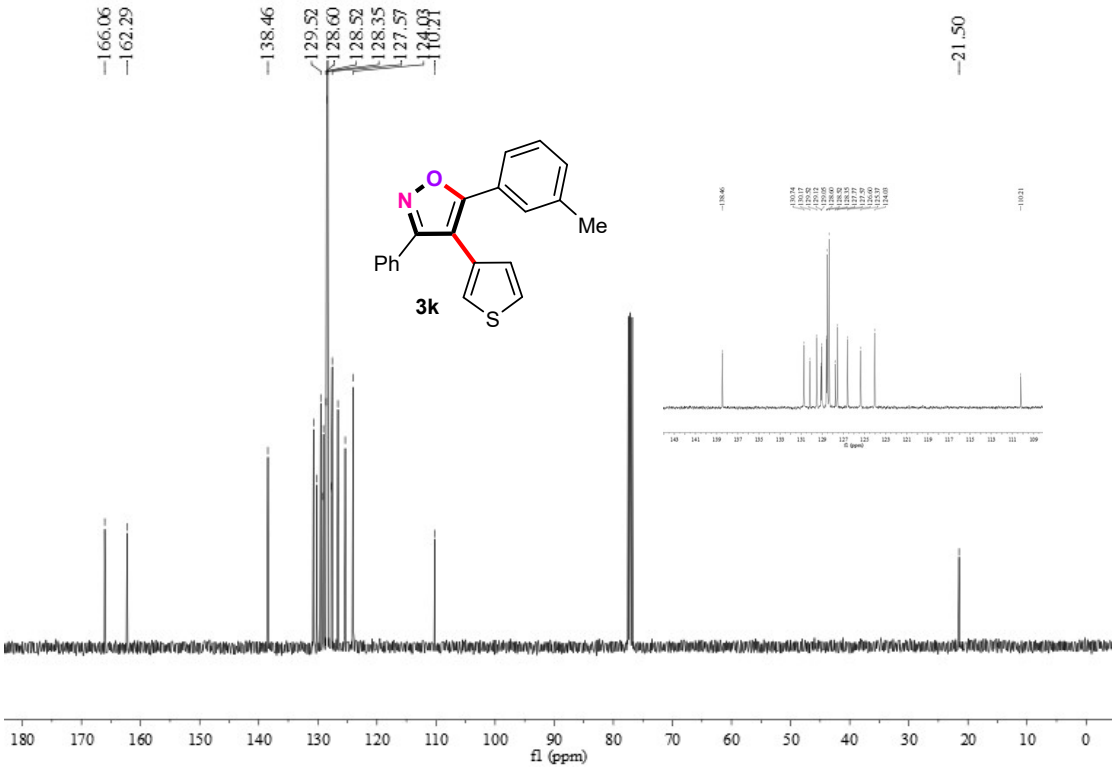
7.44
7.43
7.42
7.42
7.41
7.39
7.38
7.37
7.36
7.29
7.27
7.25
7.24
7.22
7.20
7.20
7.02
7.01
2.37
O
N
Me
Ph
S
3k
3.04
5.01
3.02
1.00
3.00
f1 (ppm)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129.52
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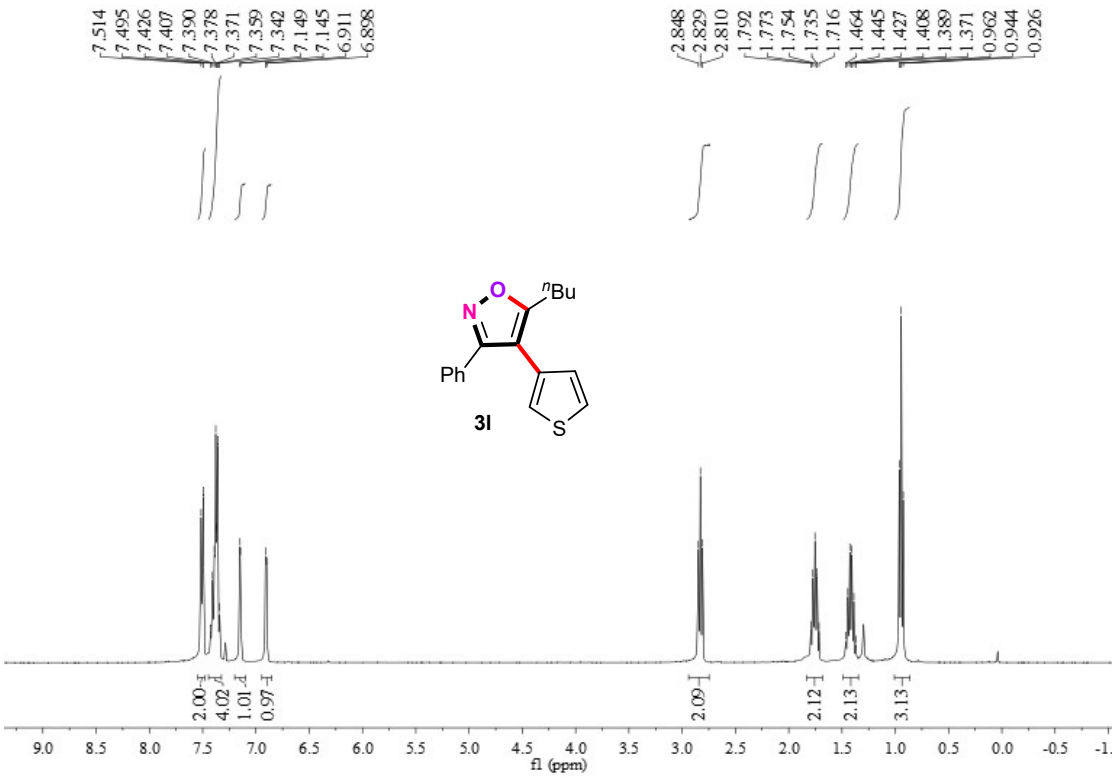
128.52
128.35
127.57
124.03
110.21
21.50
O
N
Me
Ph
S
3k
f1 (ppm)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7.378
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7.359
7.342
7.149
7.145
6.911
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2.829
2.810
1.792
1.773
1.754
1.735
1.716
1.464
1.445
1.427
1.408
1.389
1.371
0.962
0.944
0.926
nBu
N
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Ph
S
3l
2.00
4.02
1.01
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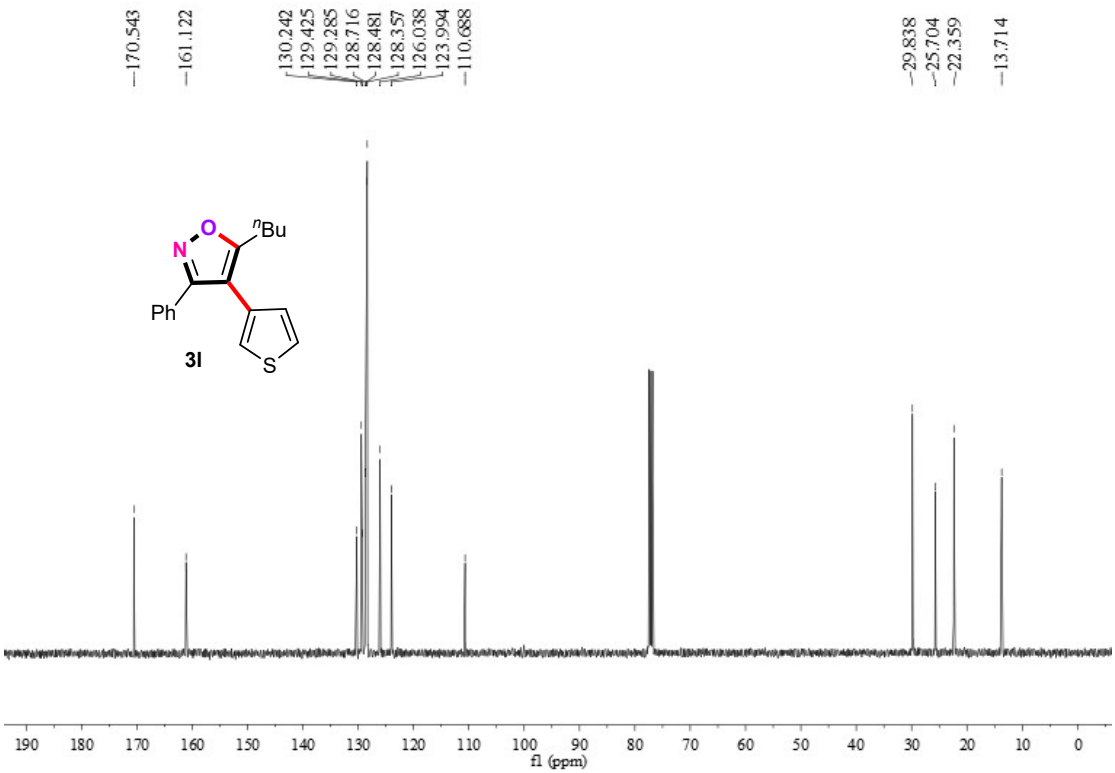
2.09
2.12
2.13
3.13
f1 (ppm)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130.242
129.425
129.285
128.716
128.481
128.357
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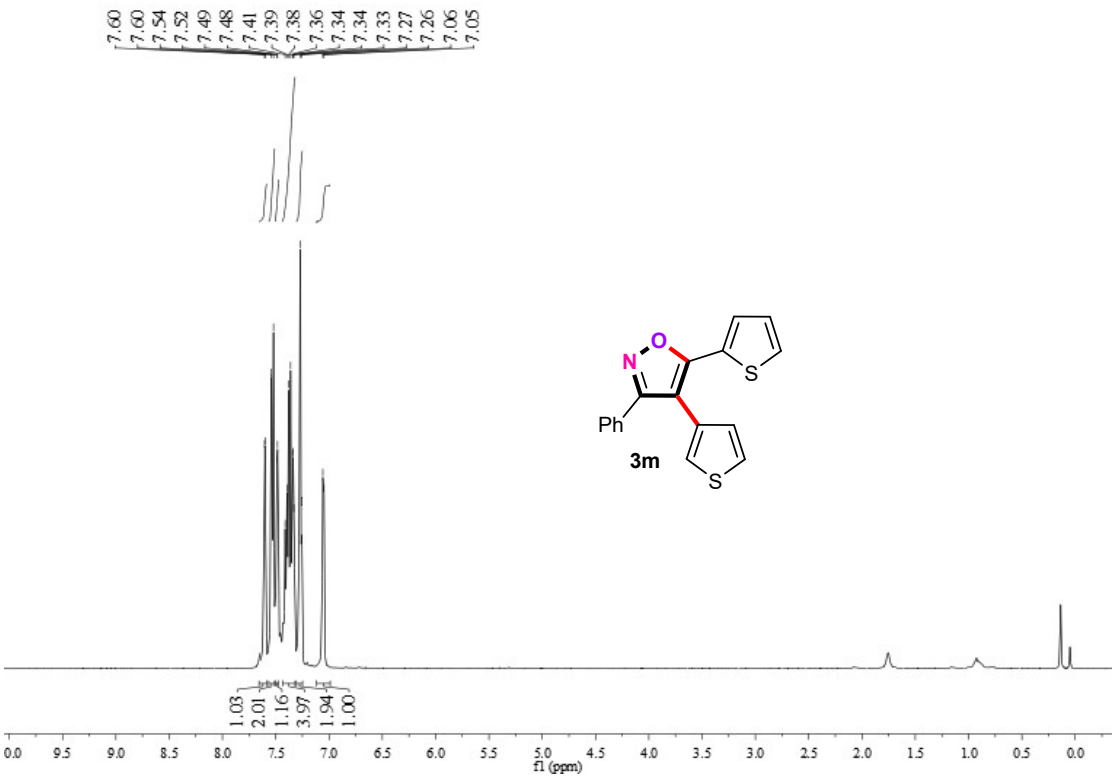
110.688
29.838
25.704
22.359
13.714
nBu
N
O
Ph
S
3l
f1 (ppm)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7.60
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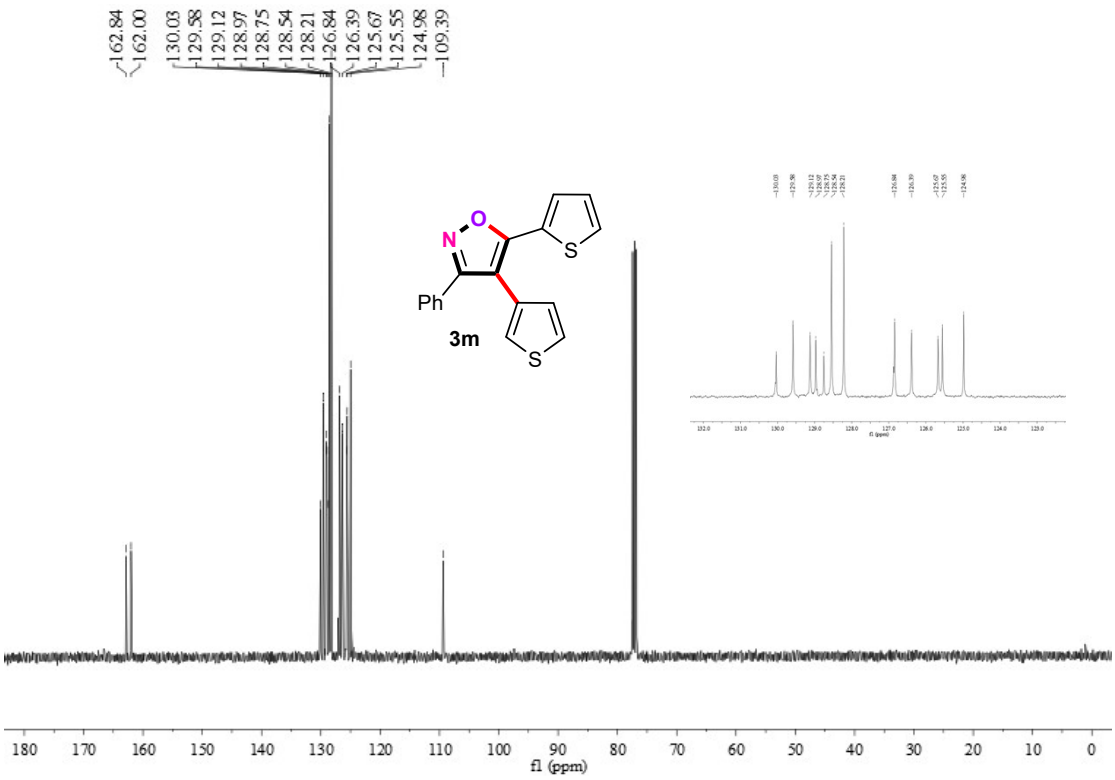
7.52
7.49
7.48
7.41
7.39
7.38
7.36
7.34
7.34
7.33
7.27
7.26
7.06
7.05
1.03
2.01
1.16
3.97
1.94
1.00
f1 (ppm)
N
O
S
Ph
S
3m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128.97
128.75
128.54
128.21
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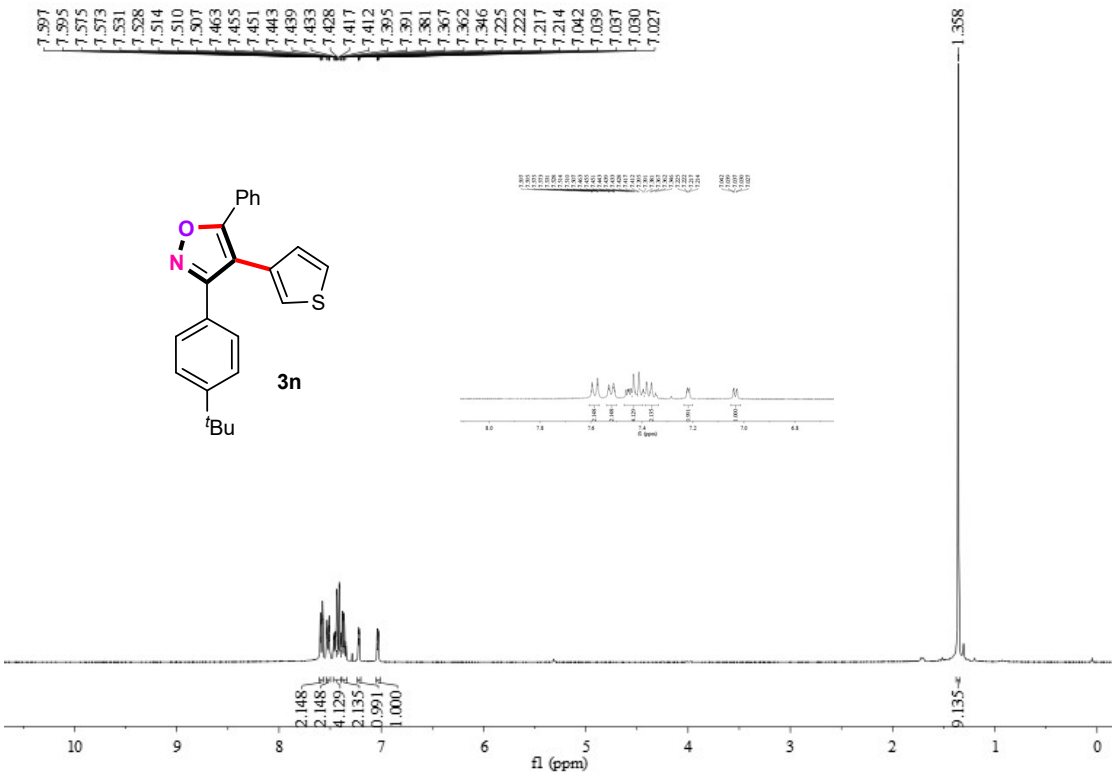
126.39
125.67
125.55
124.98
109.39
f1 (ppm)
N
O
S
Ph
S
3m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7.510
7.507
7.463
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7.443
7.439
7.433
7.428
7.417
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7.367
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7.346
7.225
7.222
7.217
7.214
7.042
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7.037
7.030
7.027
1.358
Ph
O
N
S
3n
tBu
2.148
2.148
4.129
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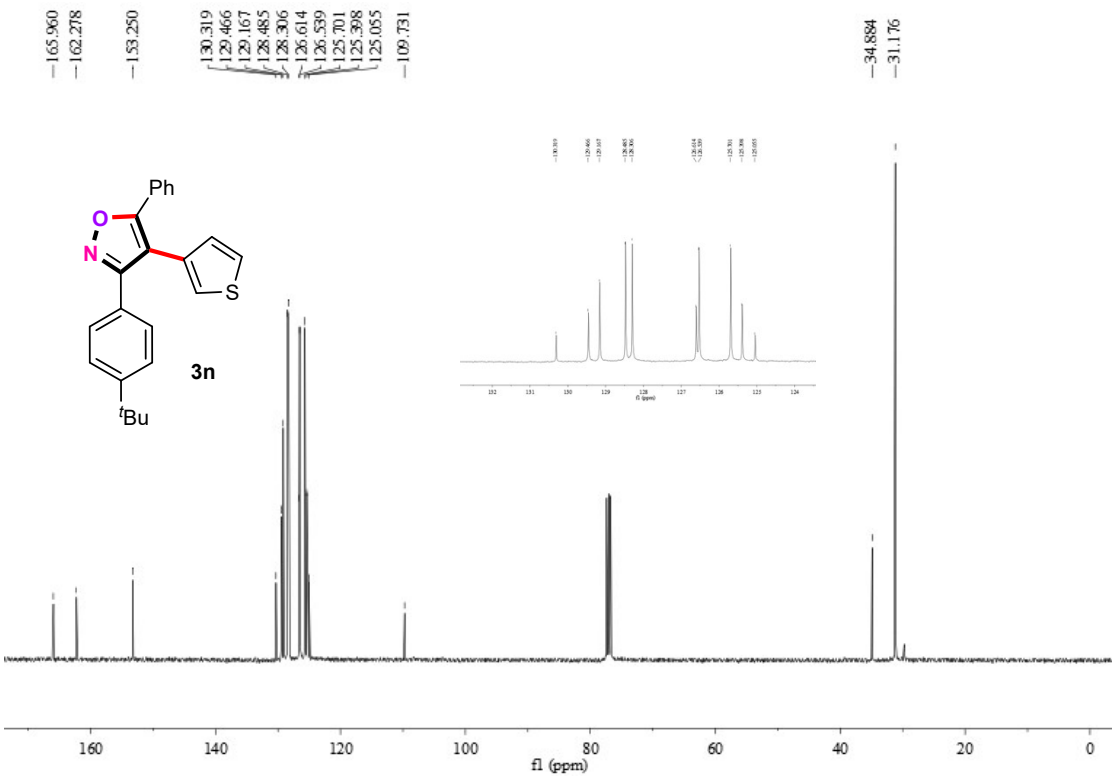
9.135
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)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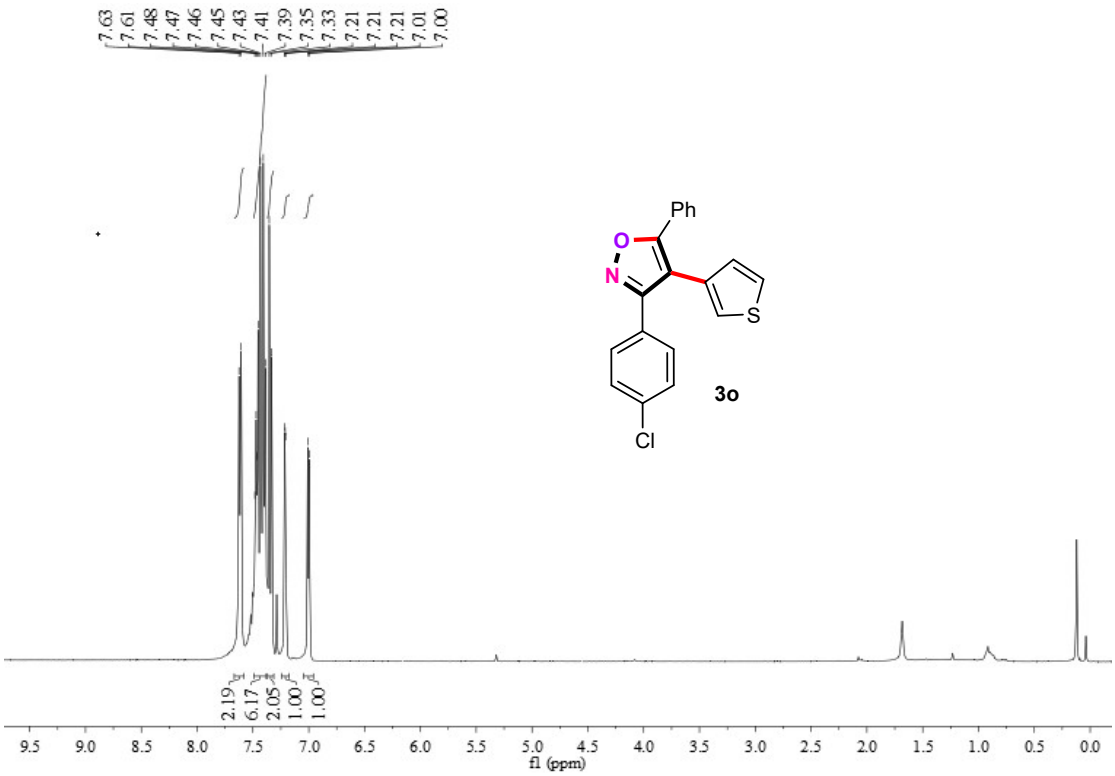
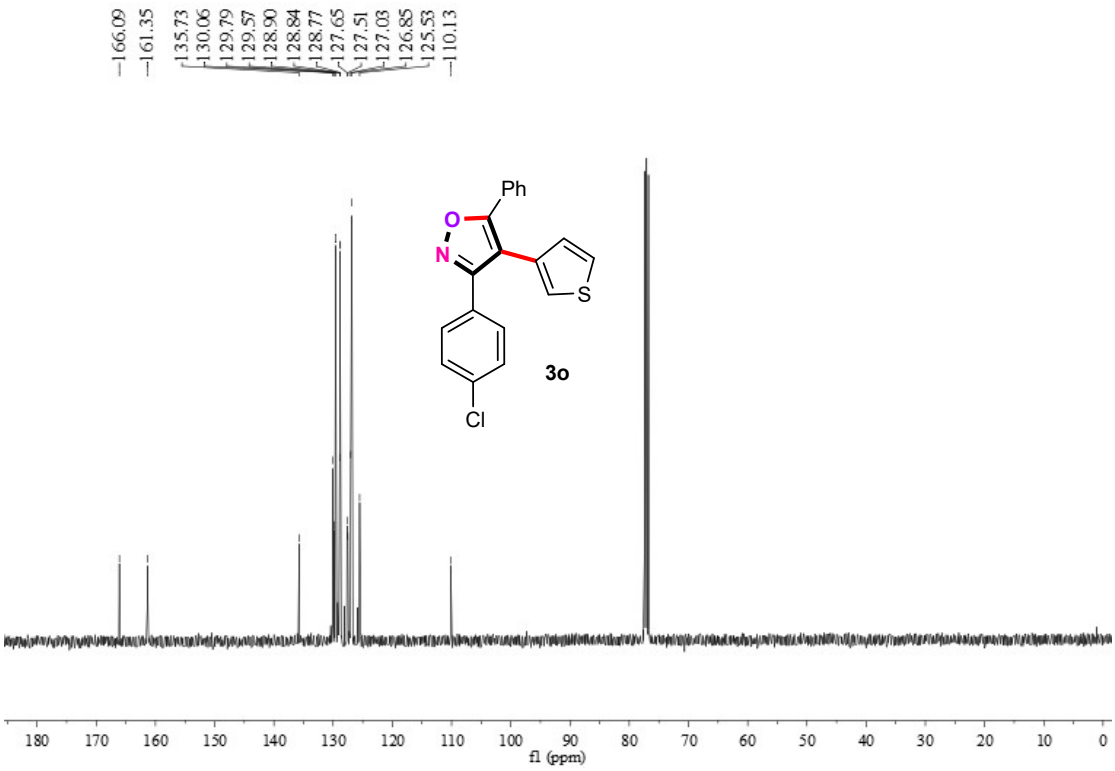
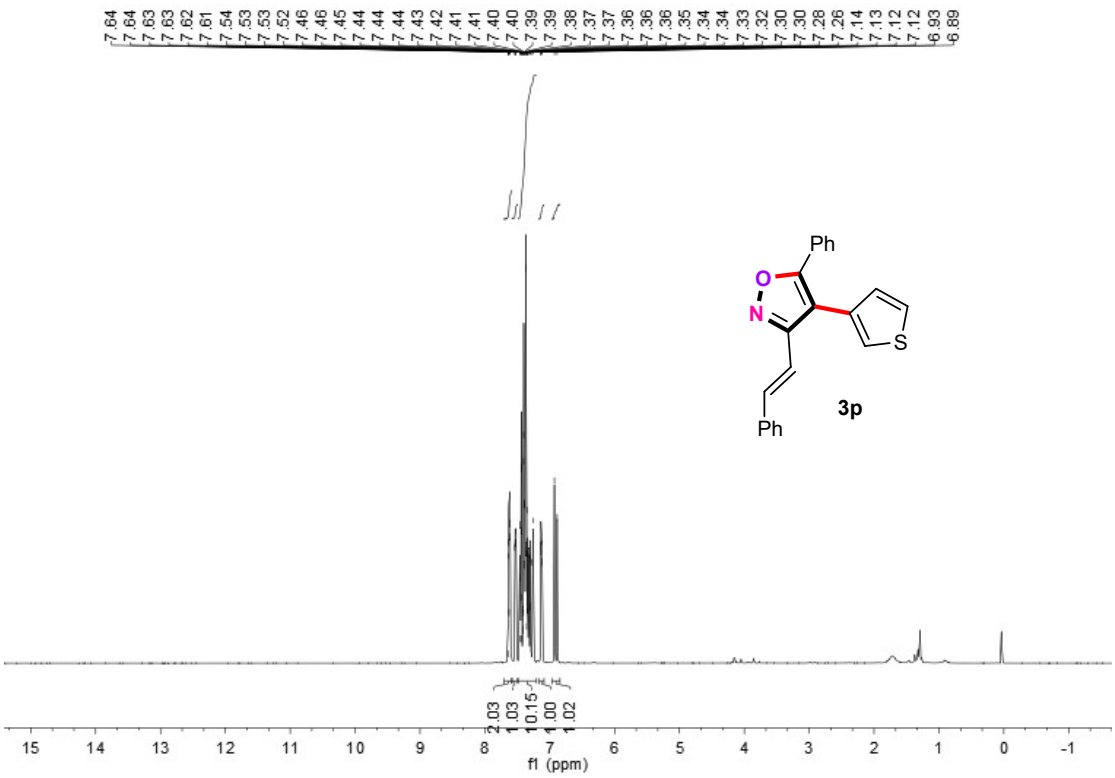
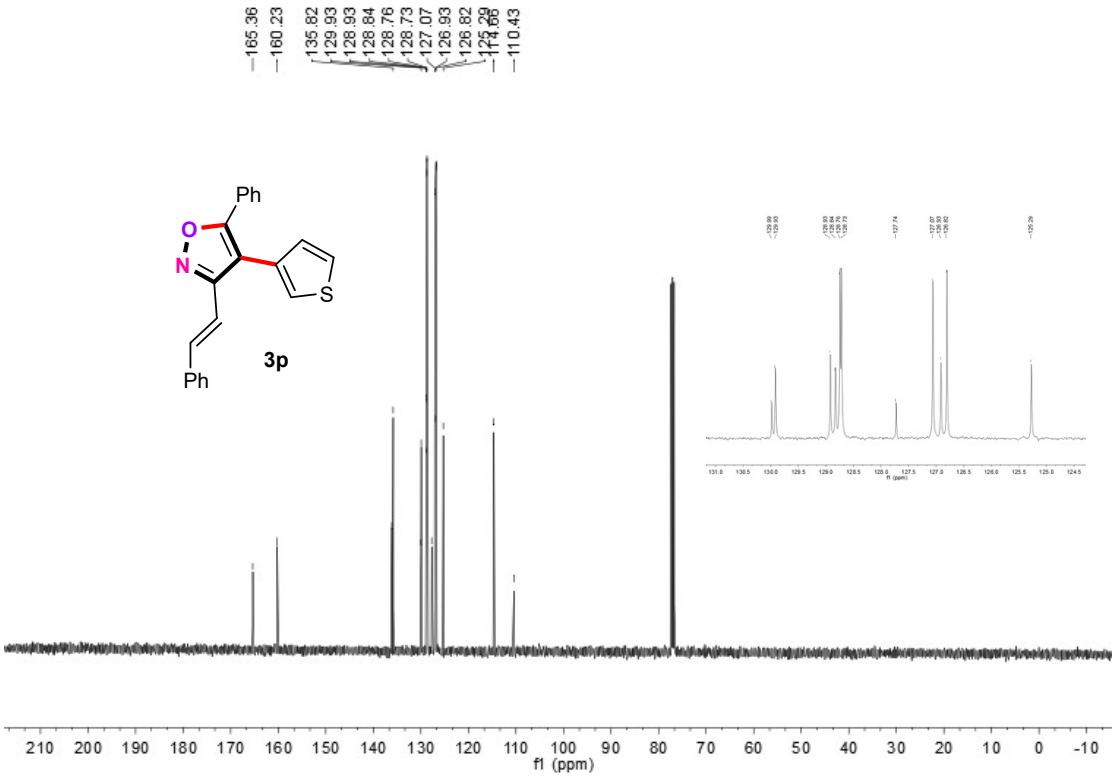
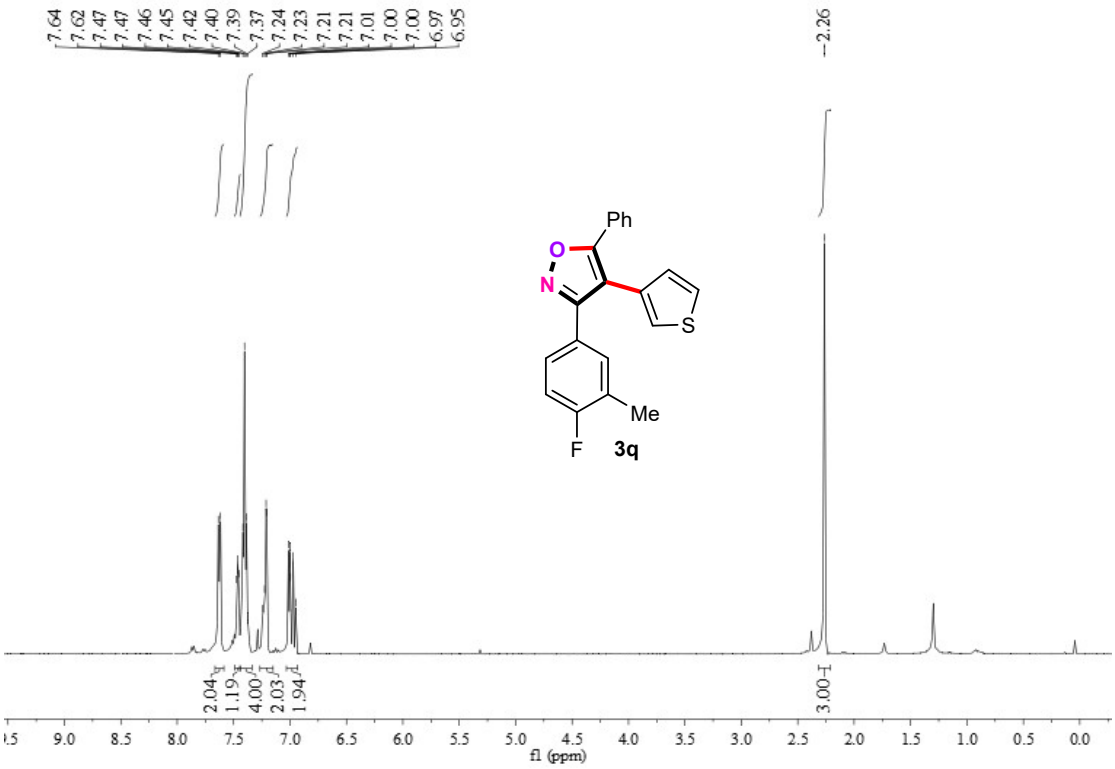
128.306
126.614
126.539
125.701
125.398
125.055
109.731
34.884
31.176
Ph
O
N
S
3n
tBu
160
140
120
100
80
60
40
20
0
f1 (ppm)

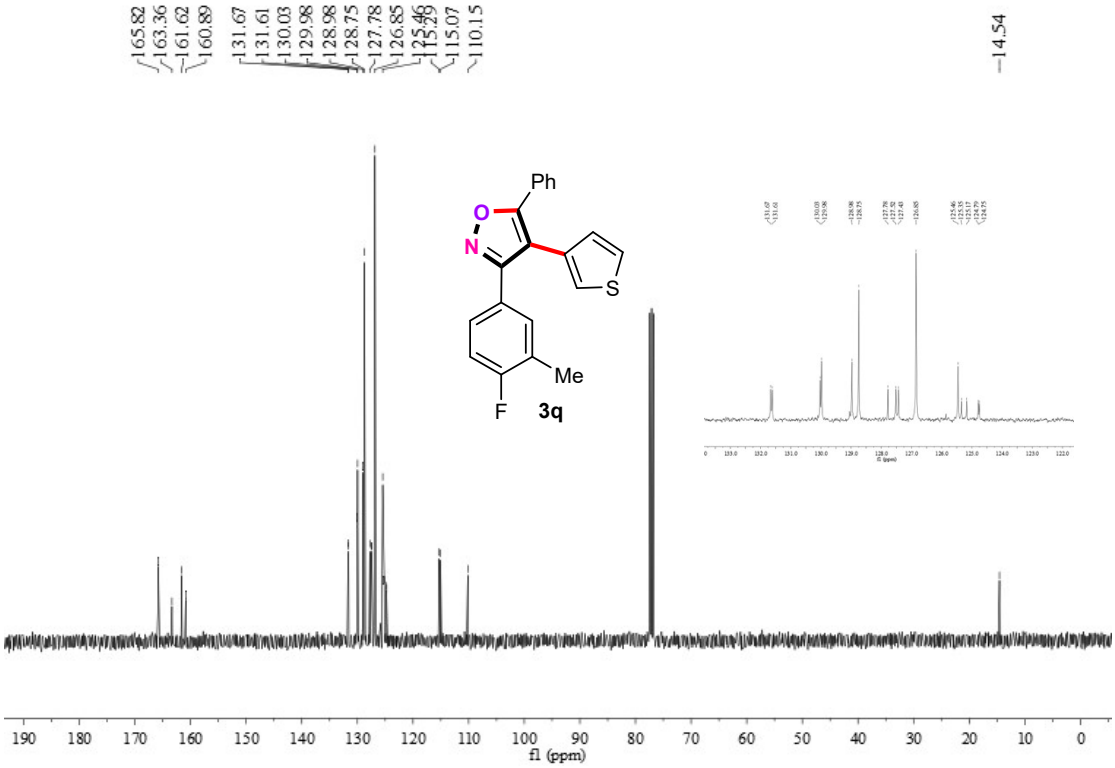

7.63
7.61
7.48
7.47
7.46
7.45
7.43
7.41
7.39
7.35
7.33
7.21
7.21
7.21
7.01
7.00
2.19
6.17
2.05
1.00
1.00
Ph
O
N
S
Cl
3o
f1 (ppm)

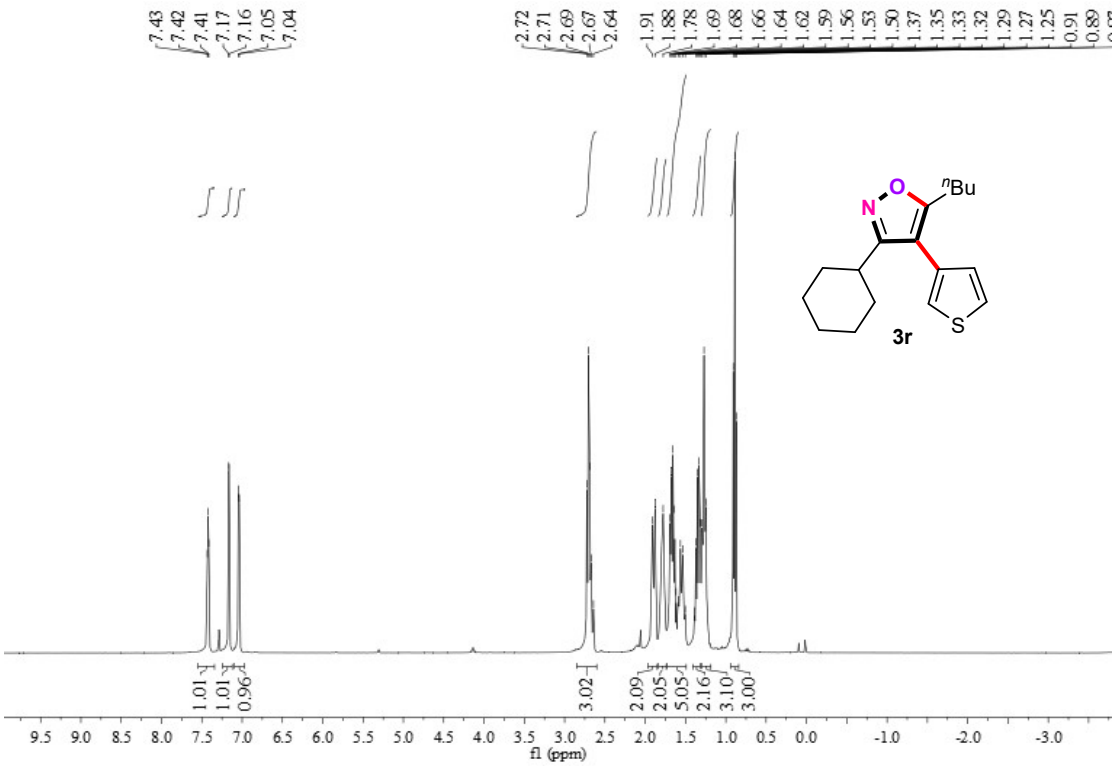

166.09
161.35
135.73
130.06
129.79
129.57
128.90
128.84
128.77
127.65
127.51
127.03
126.85
125.53
110.13
Ph
O
N
S
Cl
3o
f1 (ppm)

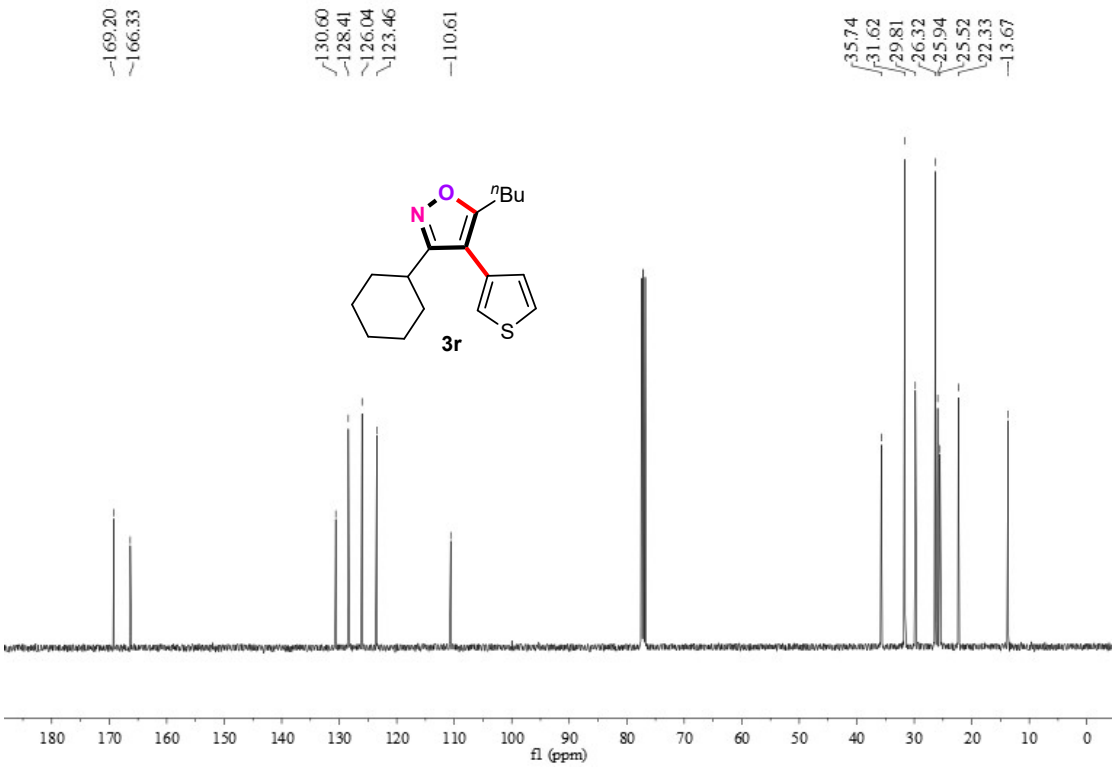

7.64 7.64 7.63 7.63 7.62 7.61 7.54 7.53 7.53 7.52 7.46 7.46 7.45 7.44 7.44 7.44 7.43 7.42 7.41 7.41 7.40 7.40 7.39 7.39 7.38 7.37 7.37 7.36 7.36 7.36 7.35 7.34 7.34 7.33 7.32 7.30 7.30 7.28 7.26 7.14 7.13 7.12 7.12 6.93 6.89
Ph
O
N
S
Ph
3p
2.03 1.03 10.15 1.00 1.02
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)

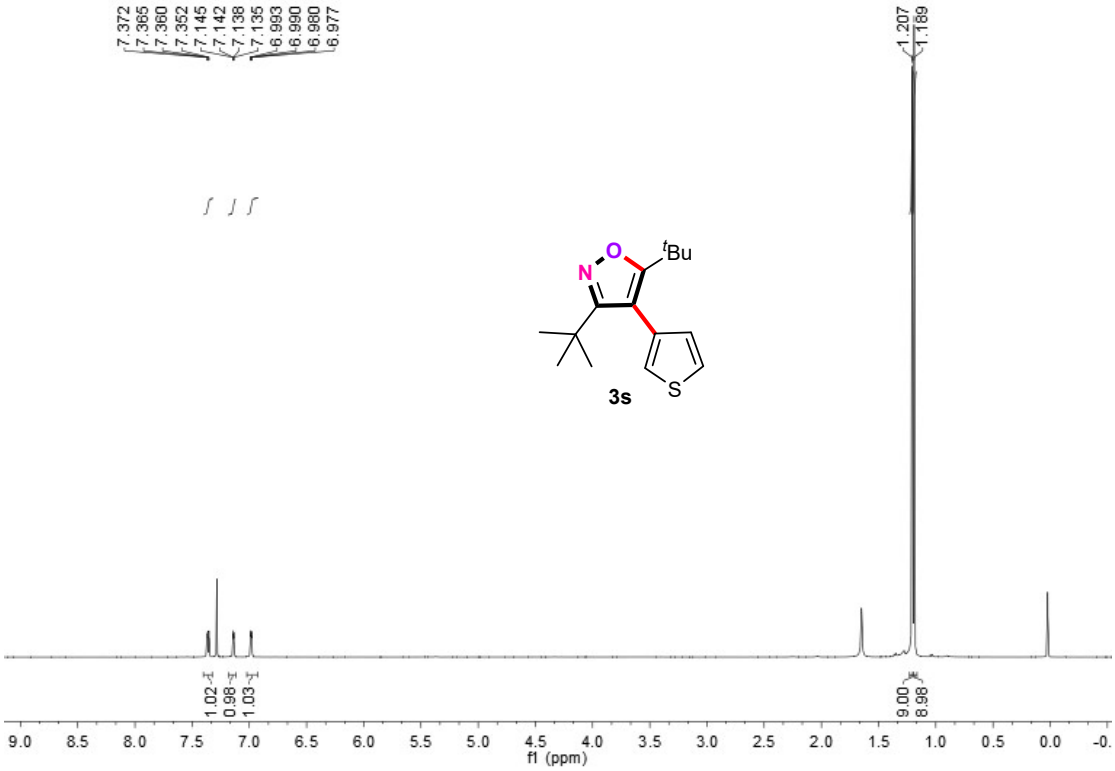

165.36 160.23 135.82 129.93 128.93 128.84 128.76 128.73 127.07 126.93 126.82 125.29 114.66 110.43
Ph
O
N
S
Ph
3p
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

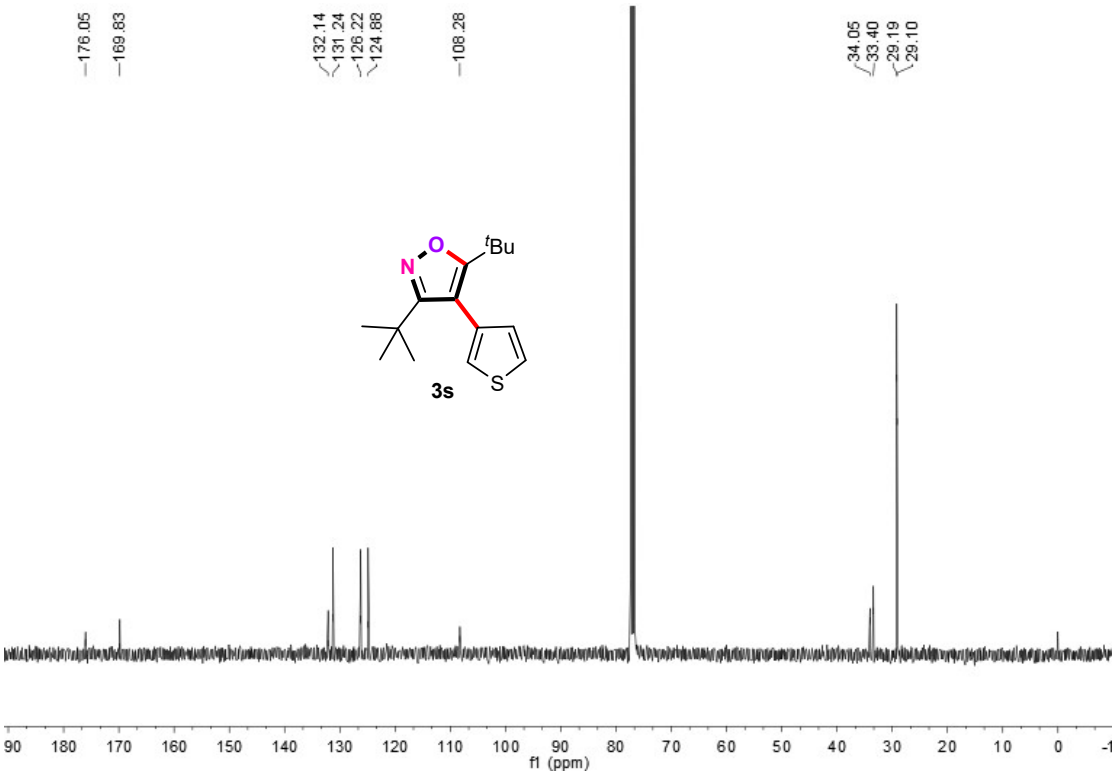

7.64
7.62
7.47
7.47
7.46
7.45
7.42
7.40
7.39
7.37
7.24
7.23
7.21
7.21
7.01
7.00
7.00
6.97
6.95
2.26
Ph
O
N
S
Me
F
3q
2.04
1.19
4.00
2.03
1.94
3.00
fl (ppm)

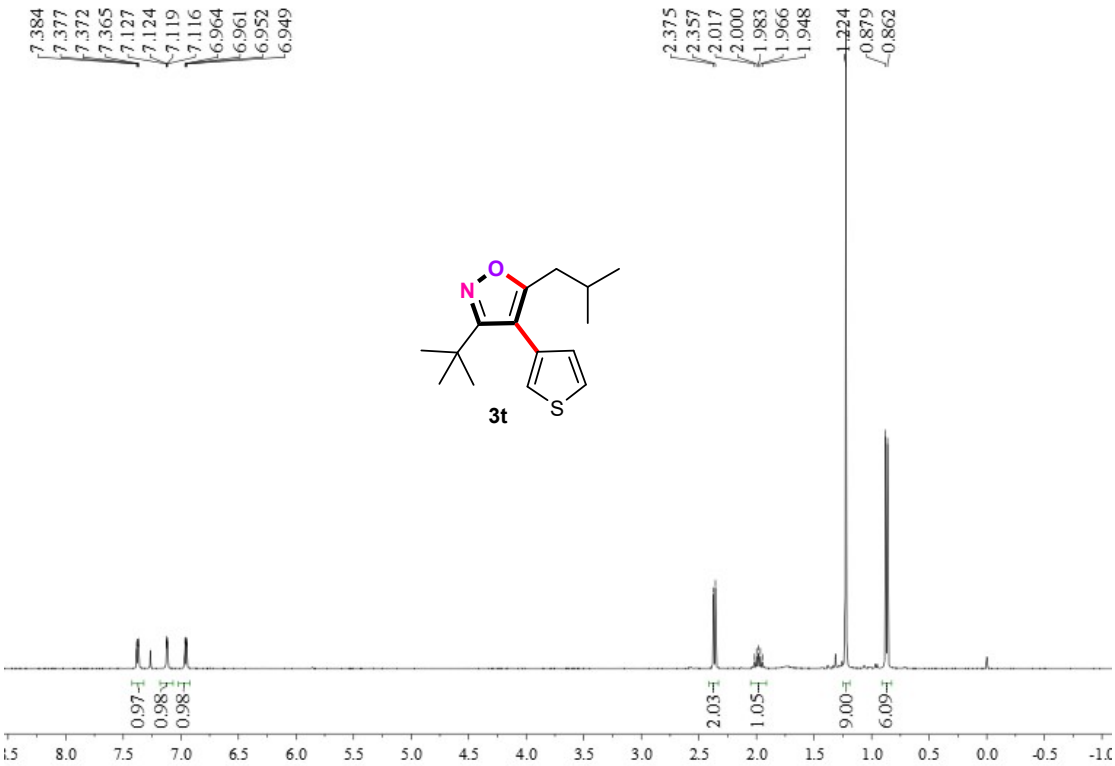

165.82
163.36
161.62
160.89
131.67
131.61
130.03
129.98
128.98
128.75
127.78
126.85
125.46
125.29
115.07
110.15
14.54
Ph
O
N
S
Me
F
3q
fl (ppm)

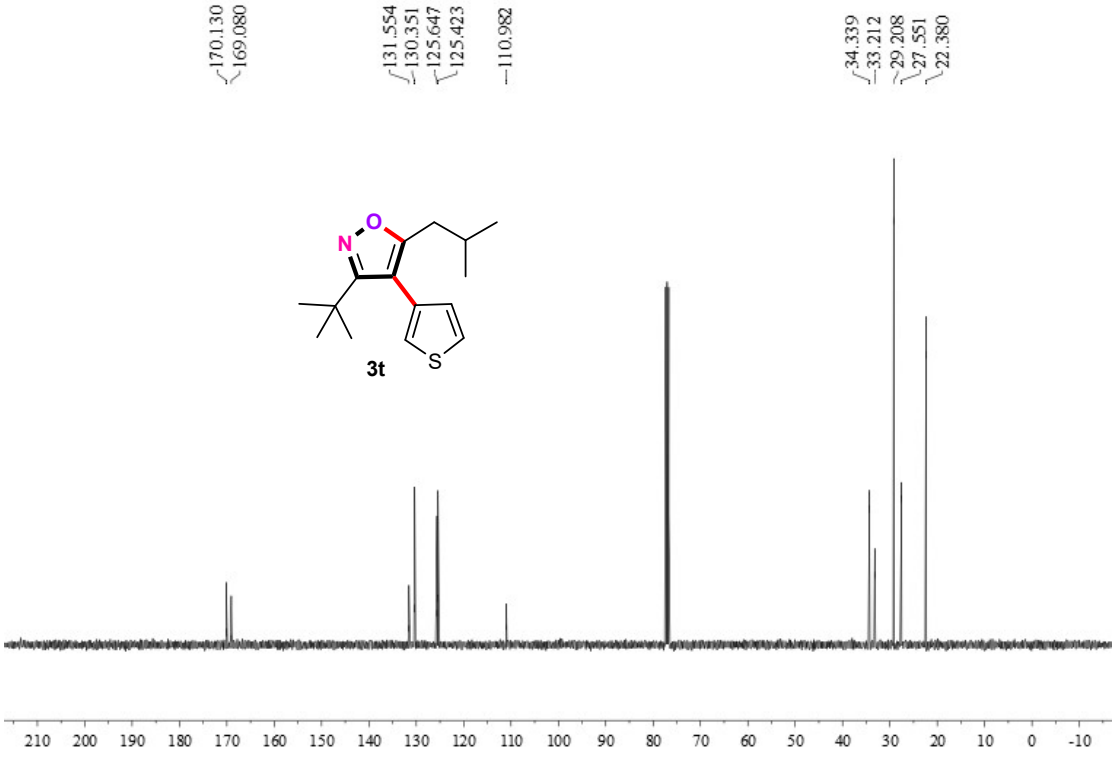

7.43
7.42
7.41
7.17
7.16
7.05
7.04
2.72
2.71
2.69
2.67
2.64
1.91
1.88
1.78
1.69
1.68
1.66
1.64
1.62
1.59
1.56
1.53
1.50
1.37
1.35
1.33
1.32
1.29
1.27
1.25
0.91
0.89
nBu
O
N
S
3r
1.01
1.01
0.96
3.02
2.09
2.05
5.05
2.16
3.10
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -1.0 -2.0 -3.0
f1 (ppm)

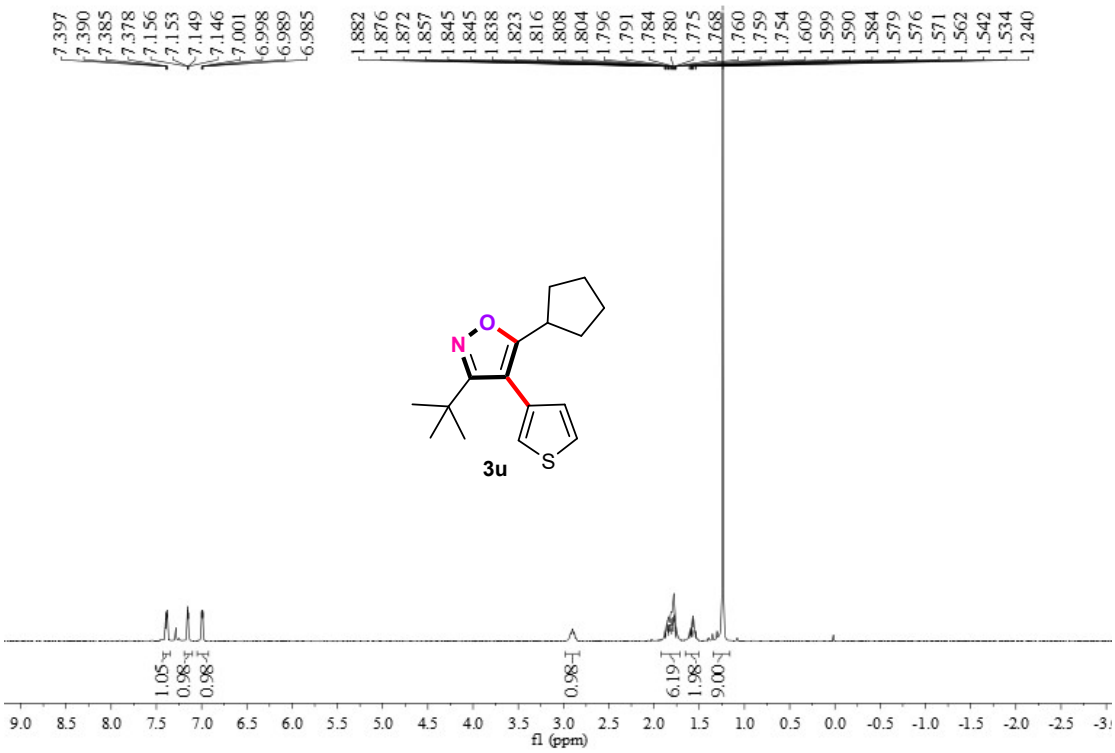

169.20
166.33
130.60
128.41
126.04
123.46
110.61
35.74
31.62
29.81
26.32
25.94
25.52
22.33
13.67
nBu
O
N
S
3r
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

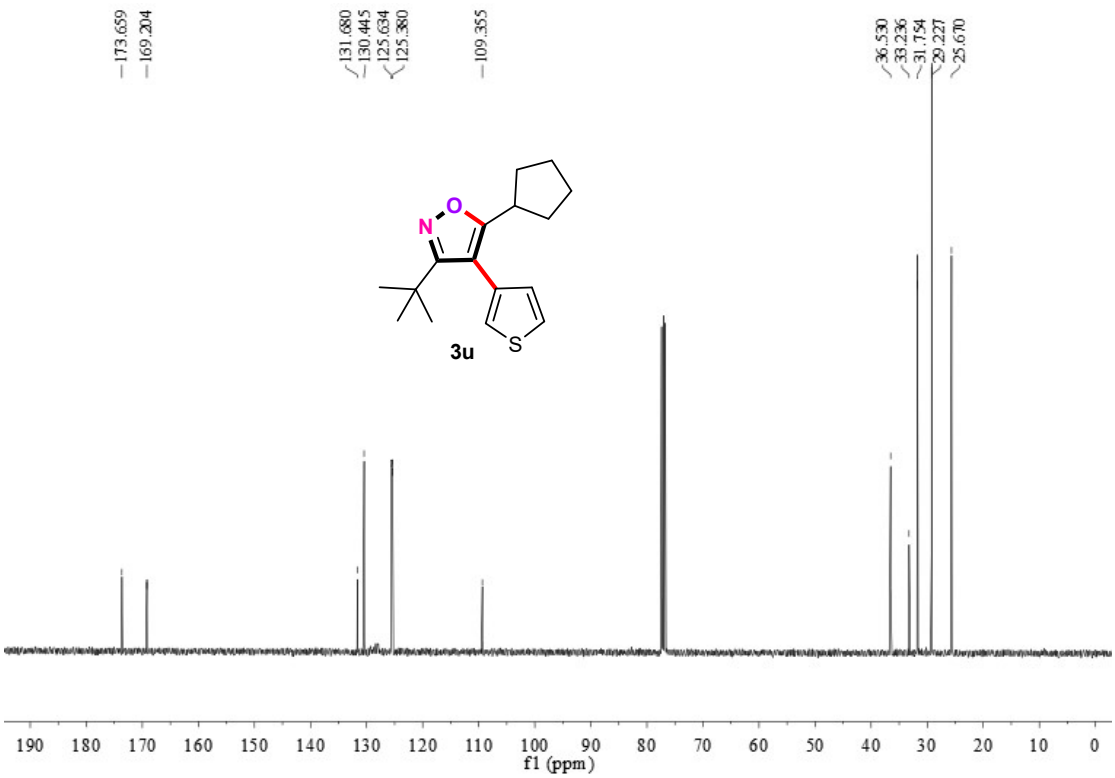

7.372
7.365
7.360
7.352
7.145
7.142
7.138
7.135
6.993
6.990
6.980
6.977
1.207
1.189
tBu
N
O
S
3s
1.02
0.98
1.03
9.00
8.98
f1 (ppm)


176.05
169.83
132.14
131.24
126.22
124.88
108.28
34.05
33.40
29.19
29.10
tBu
N
O
S
3s
f1 (ppm)

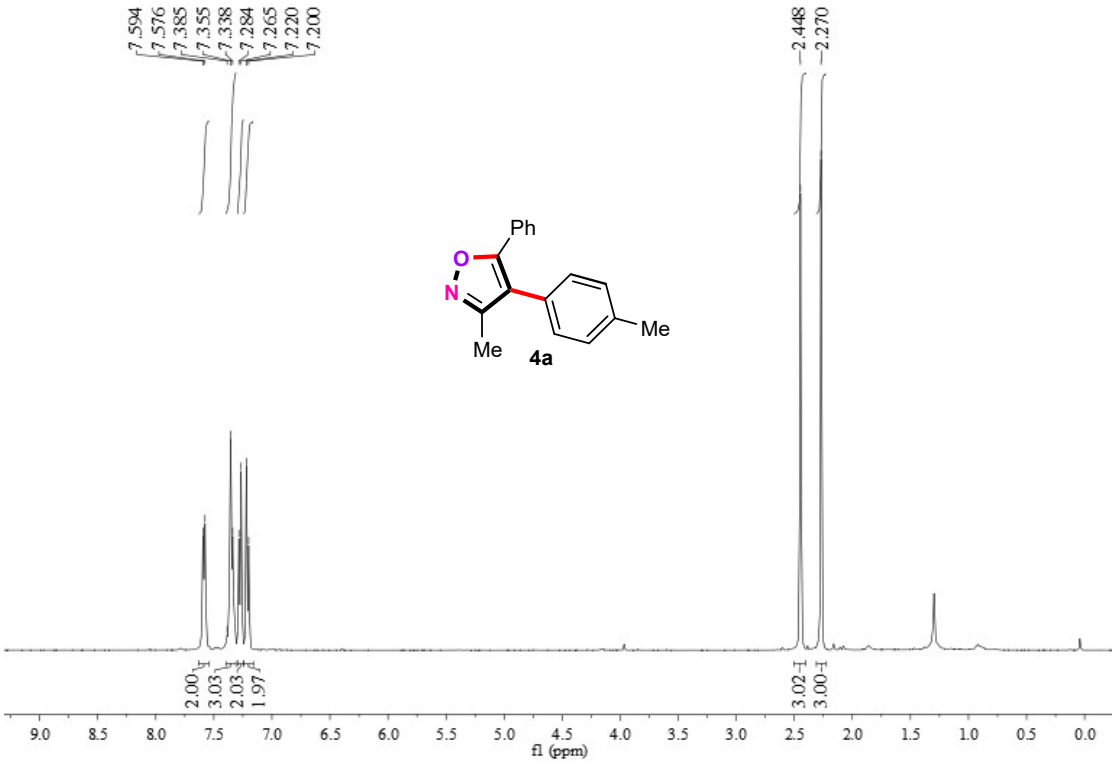

7.384
7.377
7.372
7.365
7.127
7.124
7.119
7.116
6.964
6.961
6.952
6.949
2.375
2.357
2.017
2.000
1.983
1.966
1.948
1.224
0.879
0.862
3t
0.97
0.98
0.98
2.03
1.05
9.00
6.09

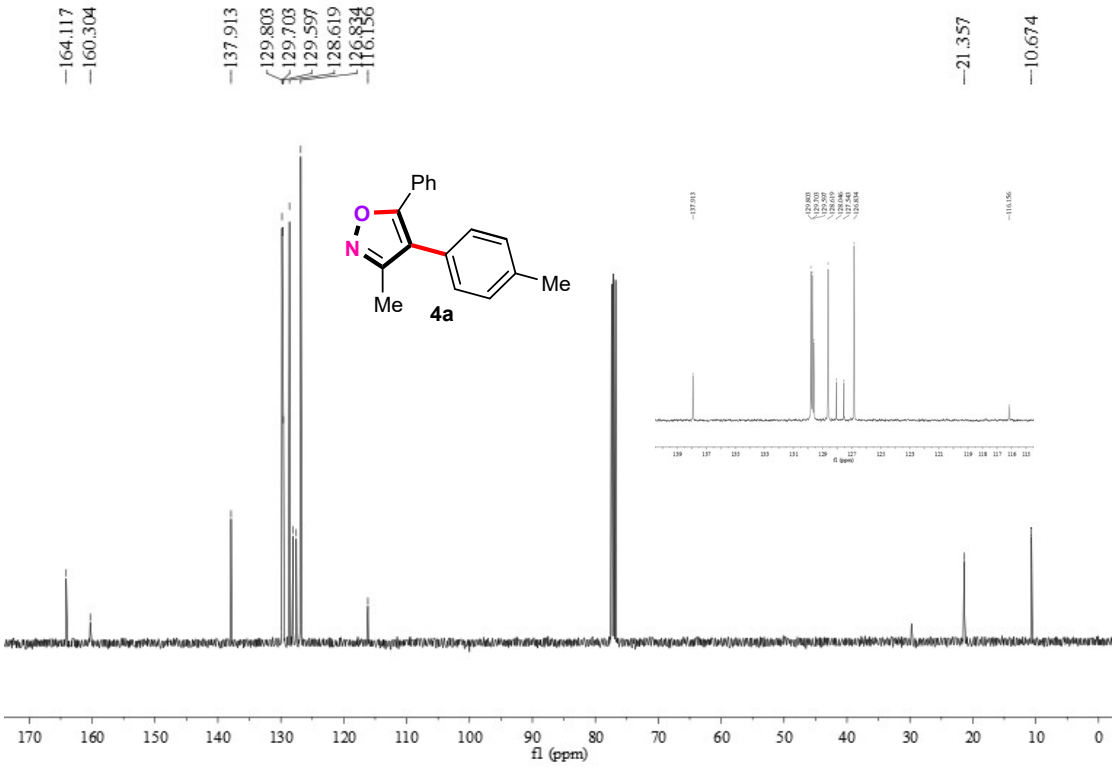

170.130
169.080
131.554
130.351
125.647
125.423
110.982
34.339
33.212
29.208
27.551
22.380
3t

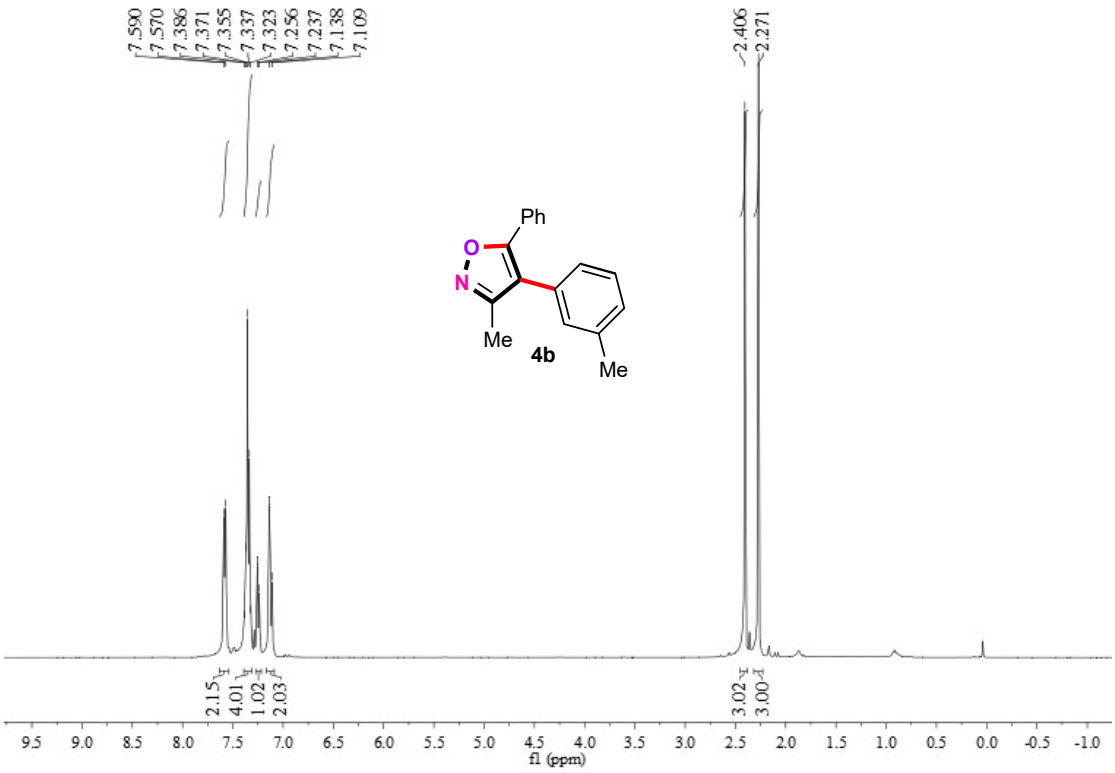


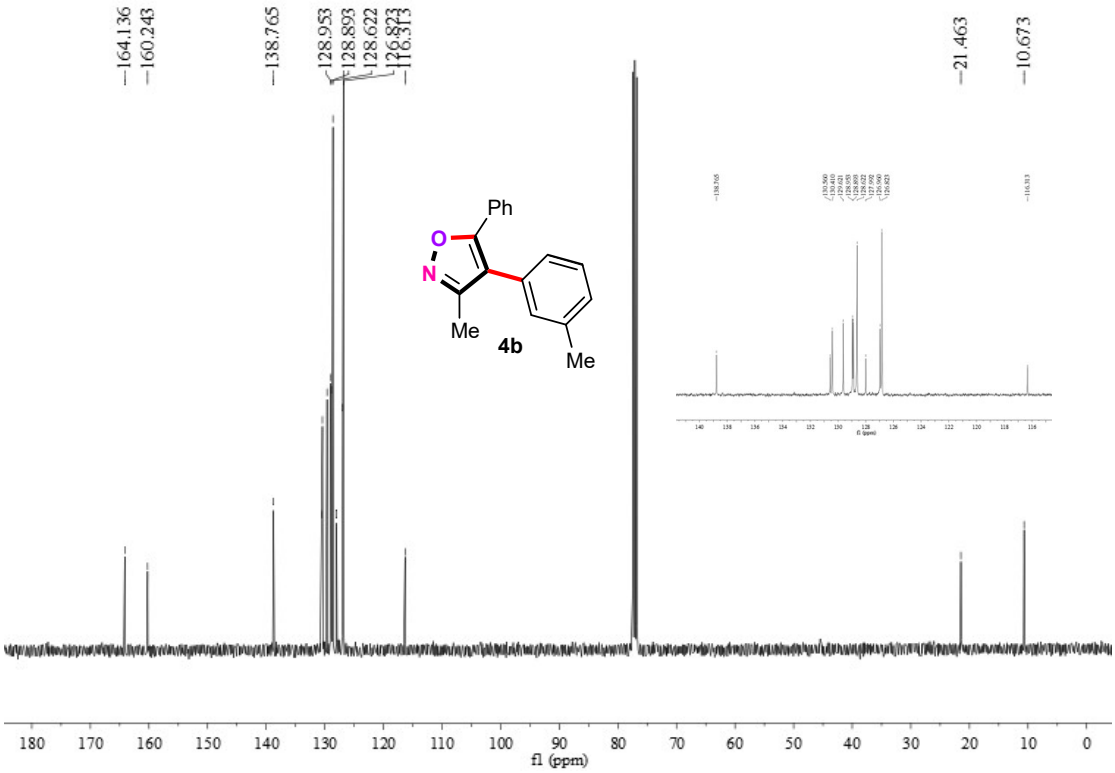



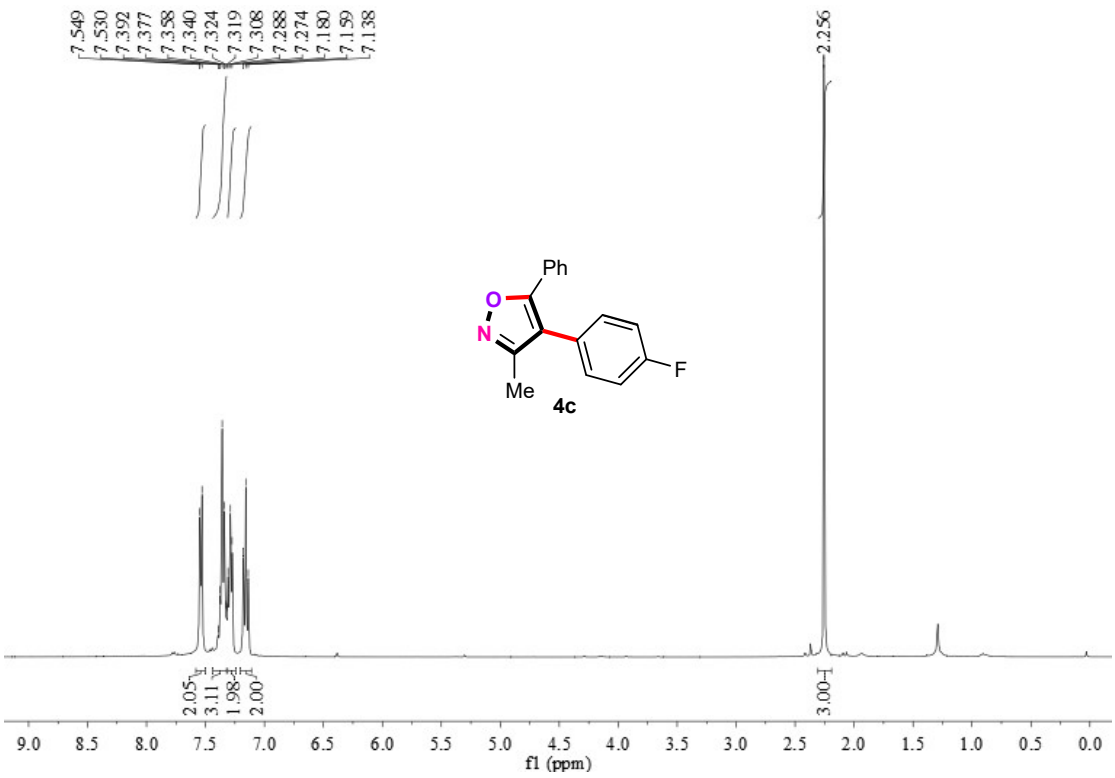

**$^{1}$H and $^{13}$C NMR spectra of compounds 4**

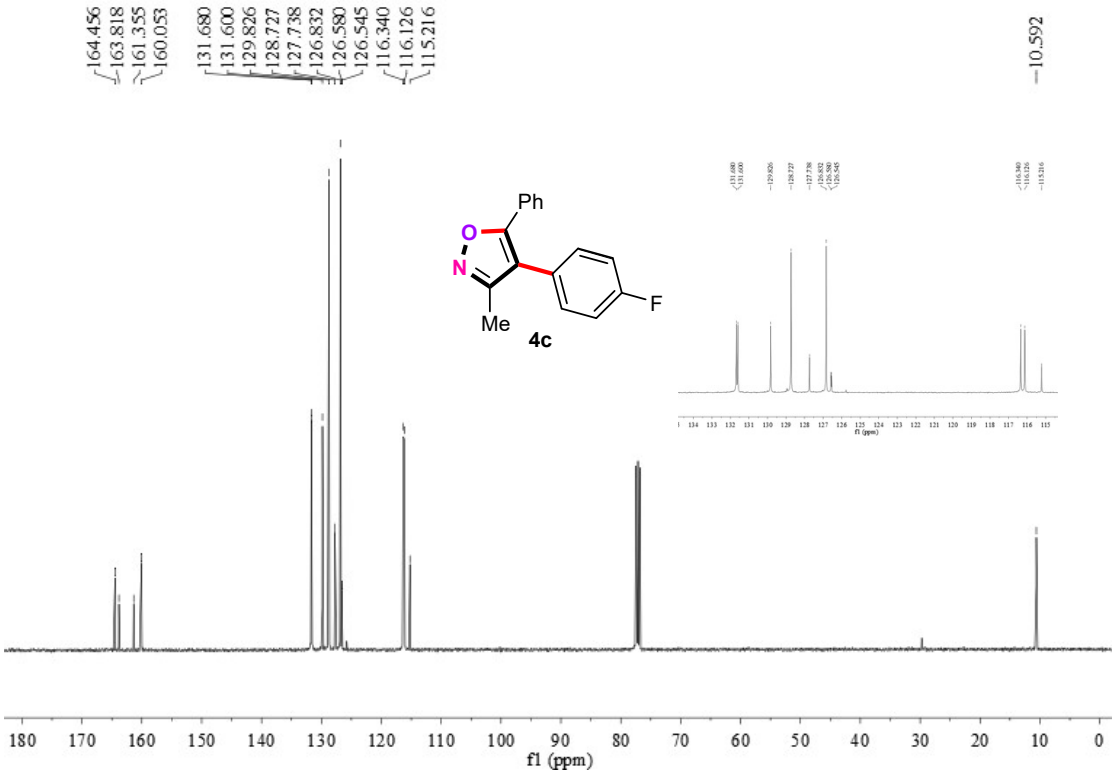

7.594
7.576
7.385
7.355
7.338
7.284
7.265
7.220
7.200
2.448
2.270
Ph
O
N
Me
Me
4a
2.00
3.03
2.03
1.97
3.02
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

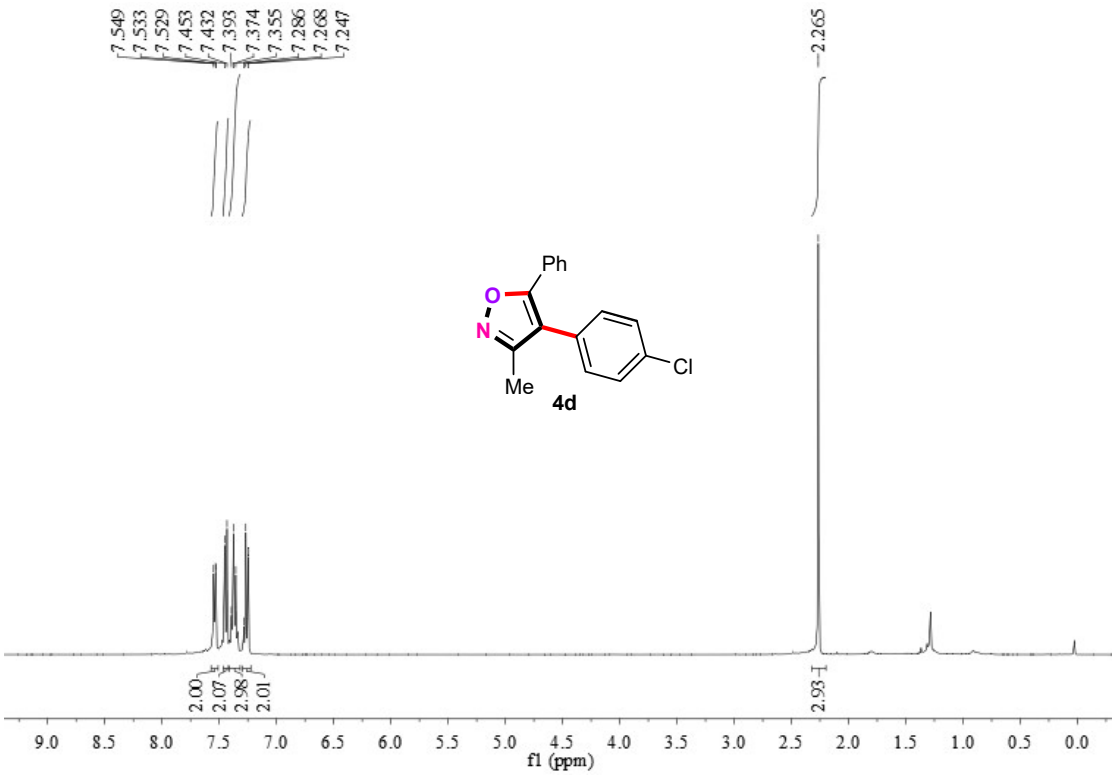

164.117
160.304
137.913
129.803
129.703
129.597
128.619
126.834
116.156
21.357
10.674
Ph
O
N
Me
Me
4a
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

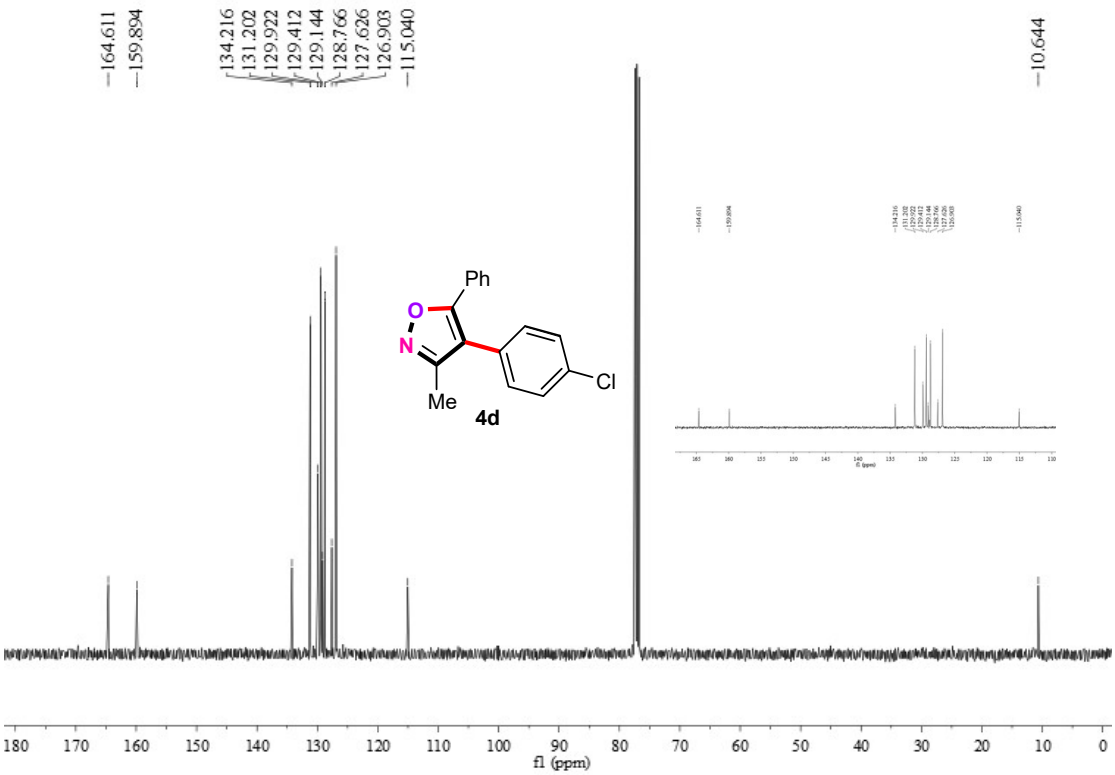

7.590
7.570
7.386
7.371
7.355
7.337
7.323
7.256
7.237
7.138
7.109
2.406
2.271
Ph
O
N
Me
4b
Me
2.15
4.01
1.02
2.03
3.02
3.00
f1 (ppm)

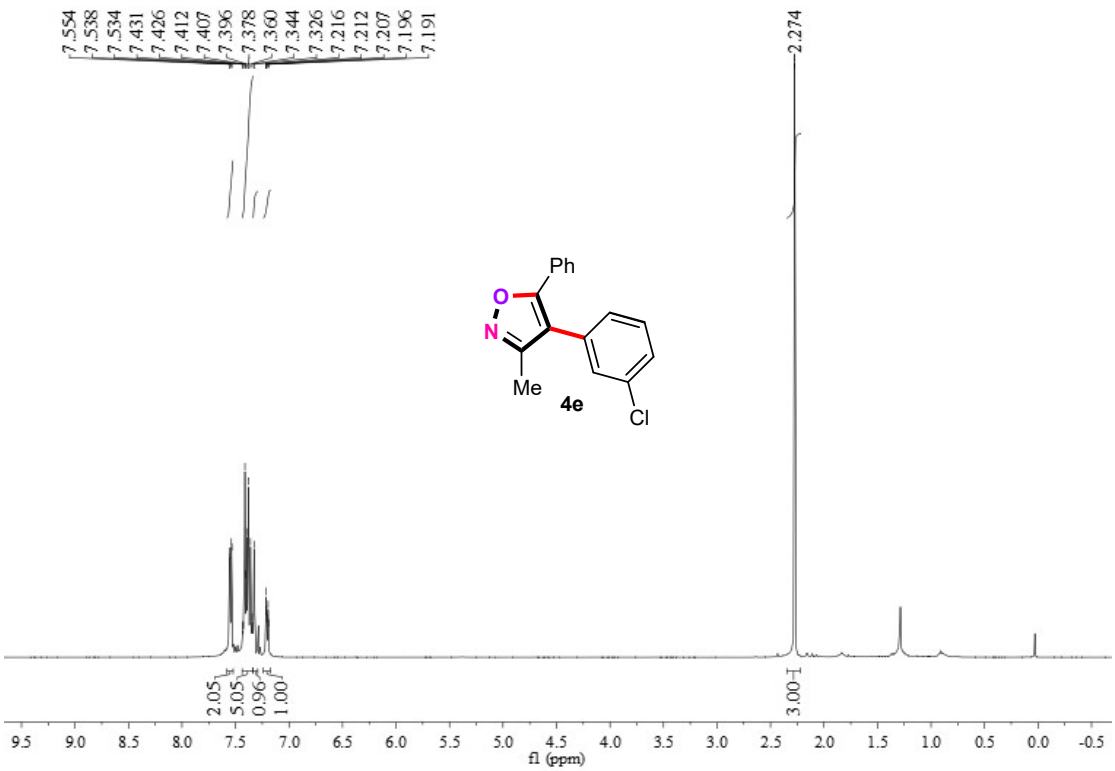

164.136
160.243
138.765
128.953
128.893
128.622
126.823
116.313
21.463
10.673
Ph
O
N
Me
4b
Me
f1 (ppm)

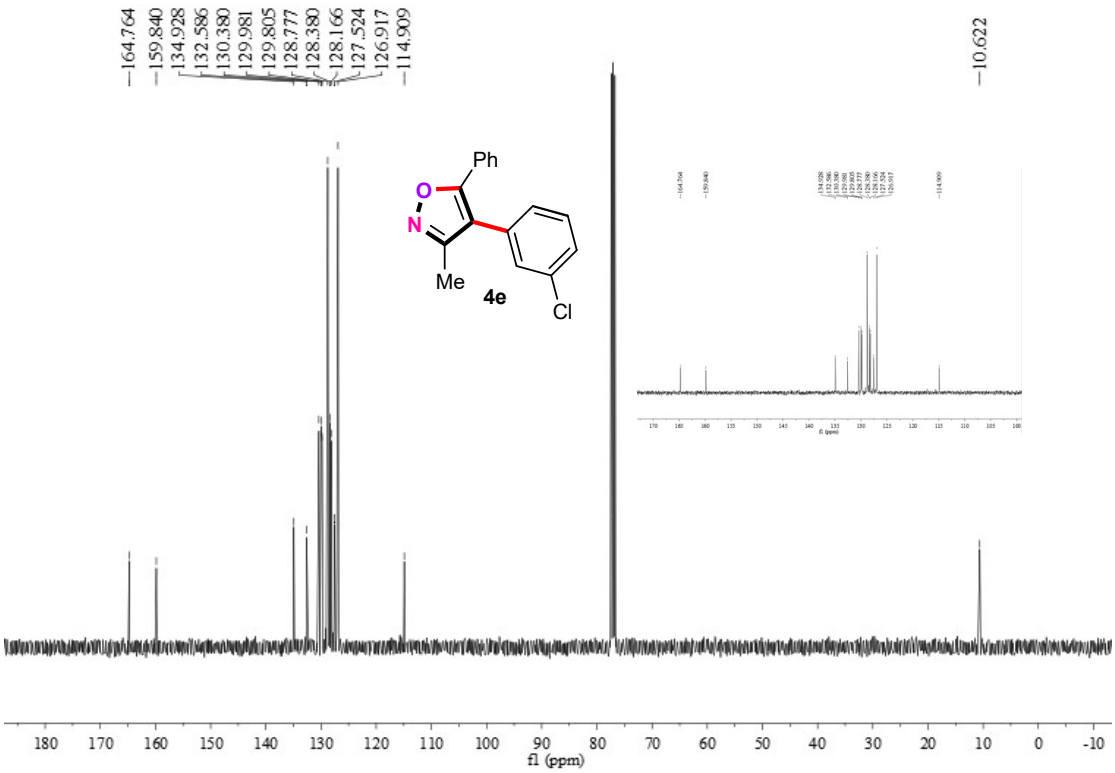

7.549
7.530
7.392
7.377
7.358
7.340
7.324
7.319
7.308
7.288
7.274
7.180
7.159
7.138
2.256
Ph
O
N
Me
F
4c
2.05
3.11
1.98
2.00
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

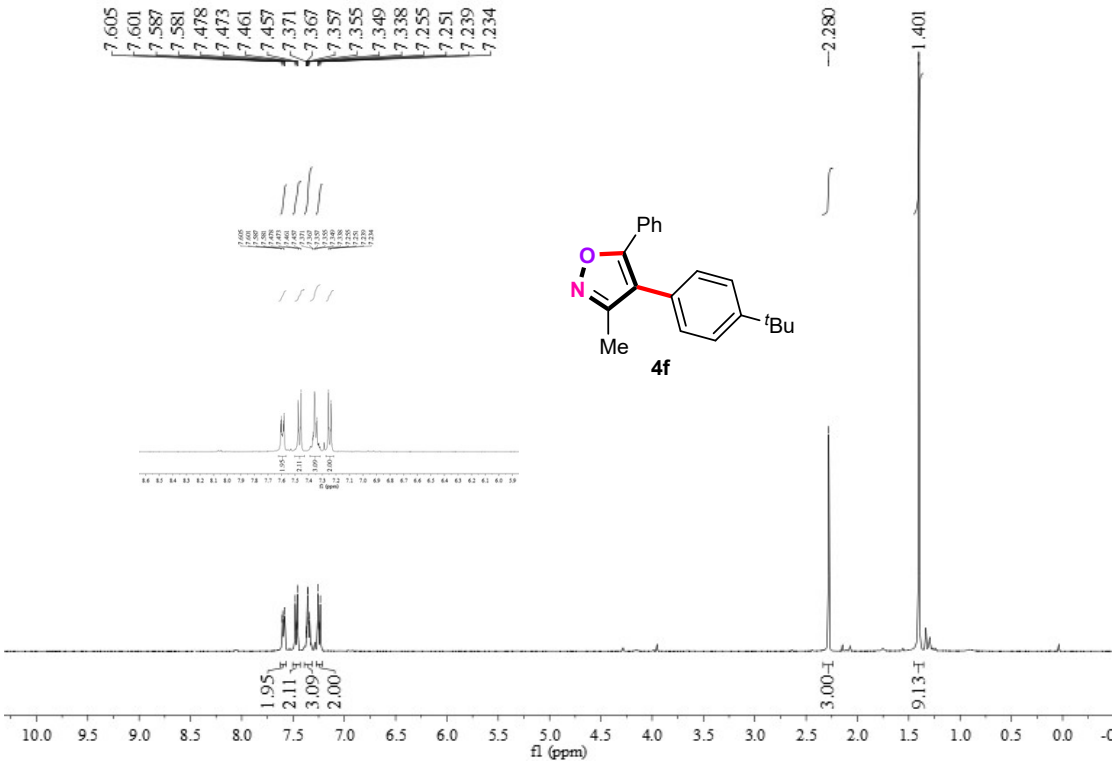

164.456
163.818
161.355
160.053
131.680
131.600
129.826
128.727
127.738
126.832
126.580
126.545
116.340
116.126
115.216
10.592
Ph
O
N
Me
F
4c
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

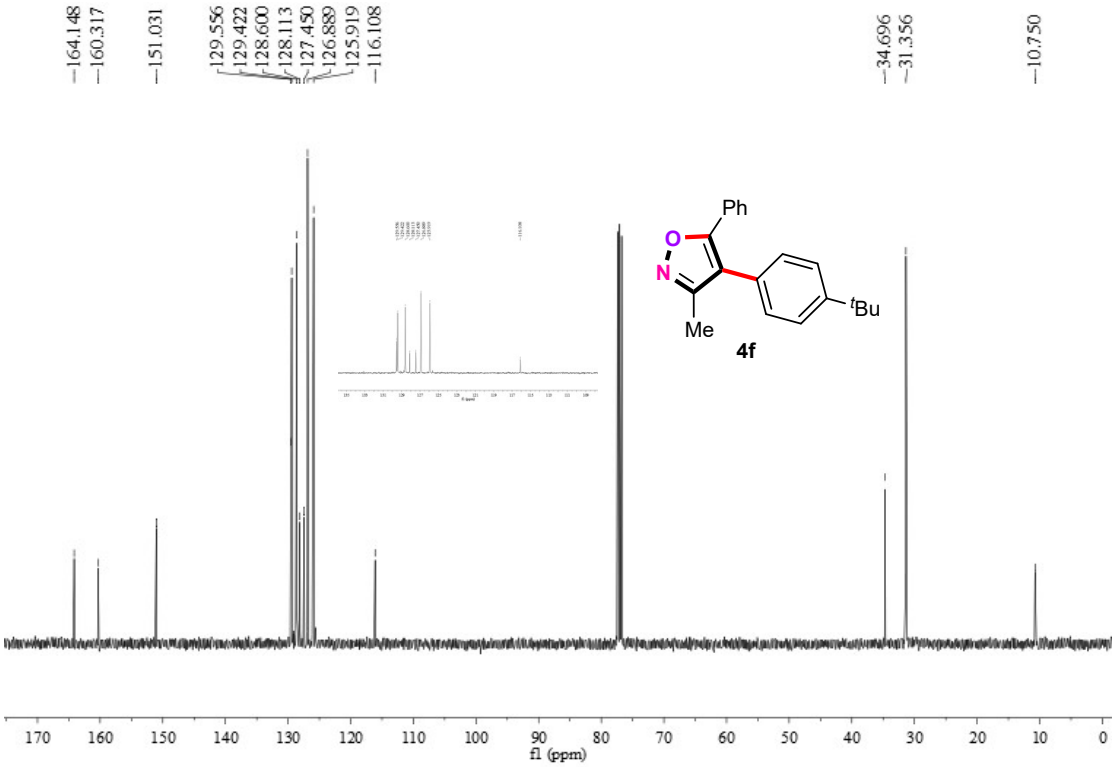

7.549
7.533
7.529
7.453
7.432
7.393
7.374
7.355
7.286
7.268
7.247
2.265
Ph
O
N
Me
Cl
4d
2.00
2.07
2.08
2.01
2.93
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

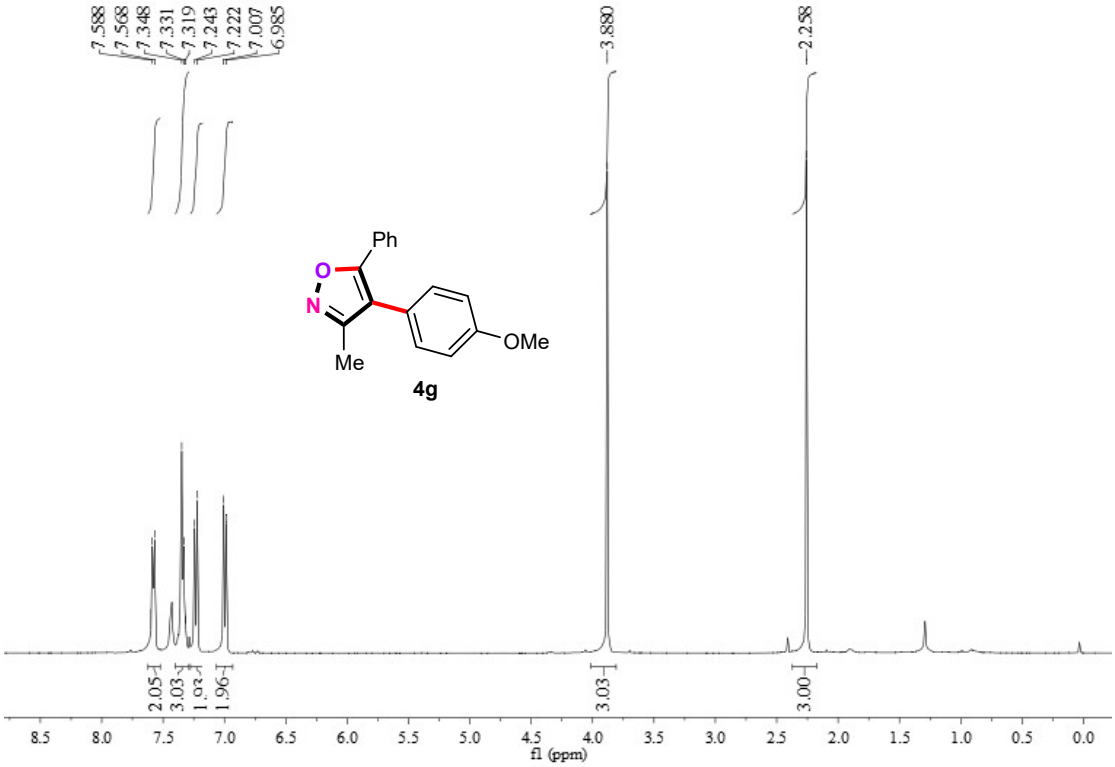

164.611
159.894
134.216
131.202
129.922
129.412
129.144
128.766
127.626
126.903
115.040
10.644
Ph
O
N
Me
Cl
4d
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

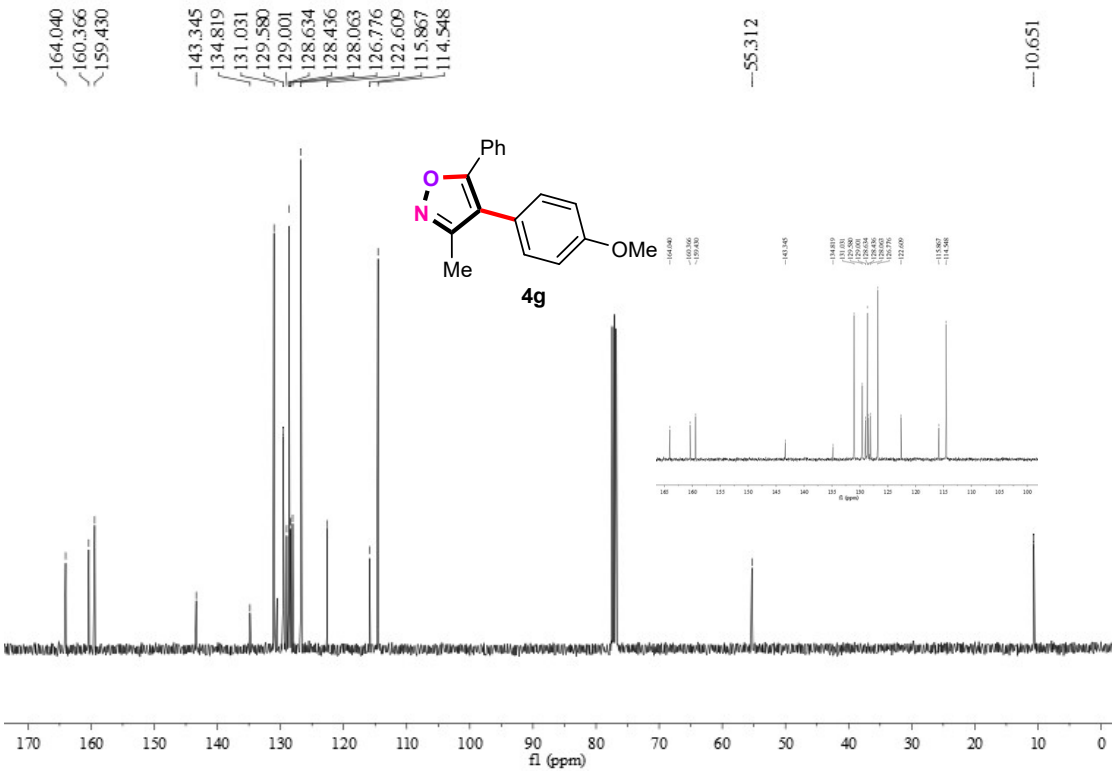

7.554
7.538
7.534
7.431
7.426
7.412
7.407
7.396
7.378
7.360
7.344
7.326
7.216
7.212
7.207
7.196
7.191
2.274
Ph
O
N
Me
4e
Cl
2.05
5.05
0.96
1.00
3.00
fl (ppm)


164.764
159.840
134.928
132.586
130.380
129.981
129.805
128.777
128.380
128.166
127.524
126.917
114.909
10.622
Ph
O
N
Me
4e
Cl
fl (ppm)


7.605
7.601
7.587
7.581
7.478
7.473
7.461
7.457
7.371
7.367
7.357
7.355
7.349
7.338
7.255
7.251
7.239
7.234
2.280
1.401
Ph
O
N
Me
tBu
4f
1.95
2.11
3.09
2.00
3.00
9.13
fl (ppm)


164.148
160.317
151.031
129.556
129.422
128.600
128.113
127.450
126.889
125.919
116.108
34.696
31.356
10.750
Ph
O
N
Me
tBu
4f
fl (ppm)


7.588
7.568
7.348
7.331
7.319
7.243
7.222
7.007
6.985
3.880
2.258
Ph
O
N
Me
OMe
4g
2.05
3.03
1.96
3.03
3.00
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
0.5
0.0
f1 (ppm)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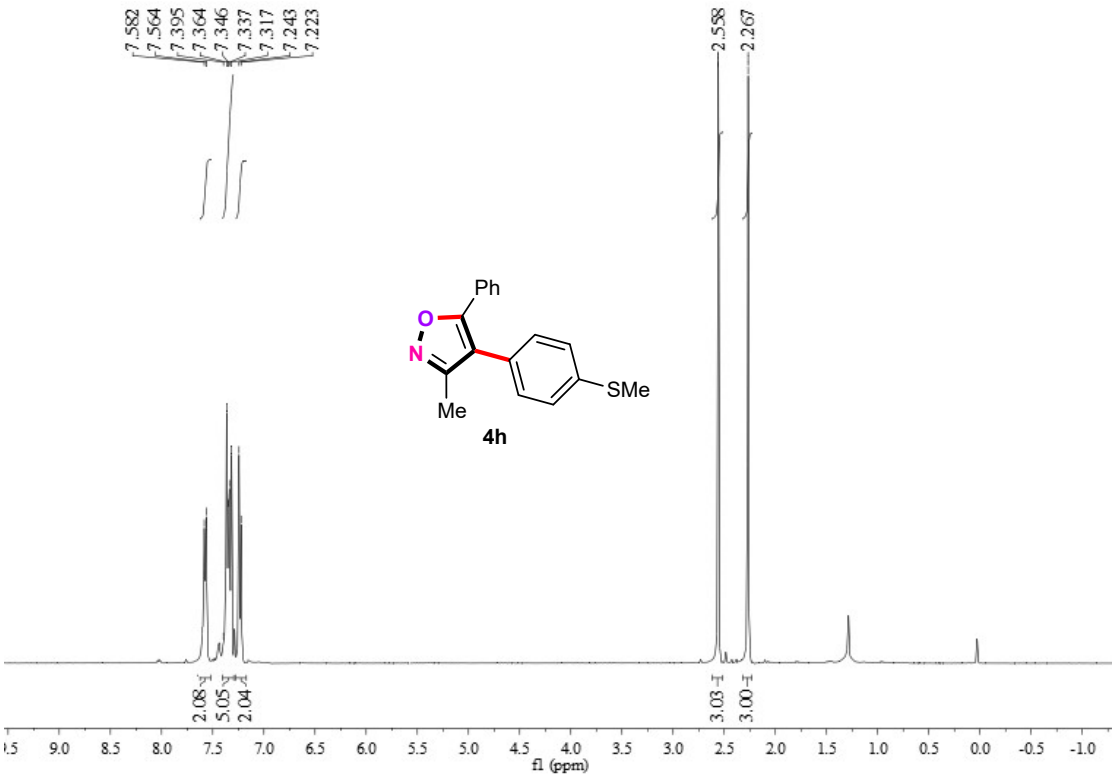
160.366
159.430
143.345
134.819
131.031
129.580
129.001
128.634
128.436
128.063
126.776
122.609
115.867
114.548
55.312
10.651
Ph
O
N
Me
OMe
4g
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.564
7.395
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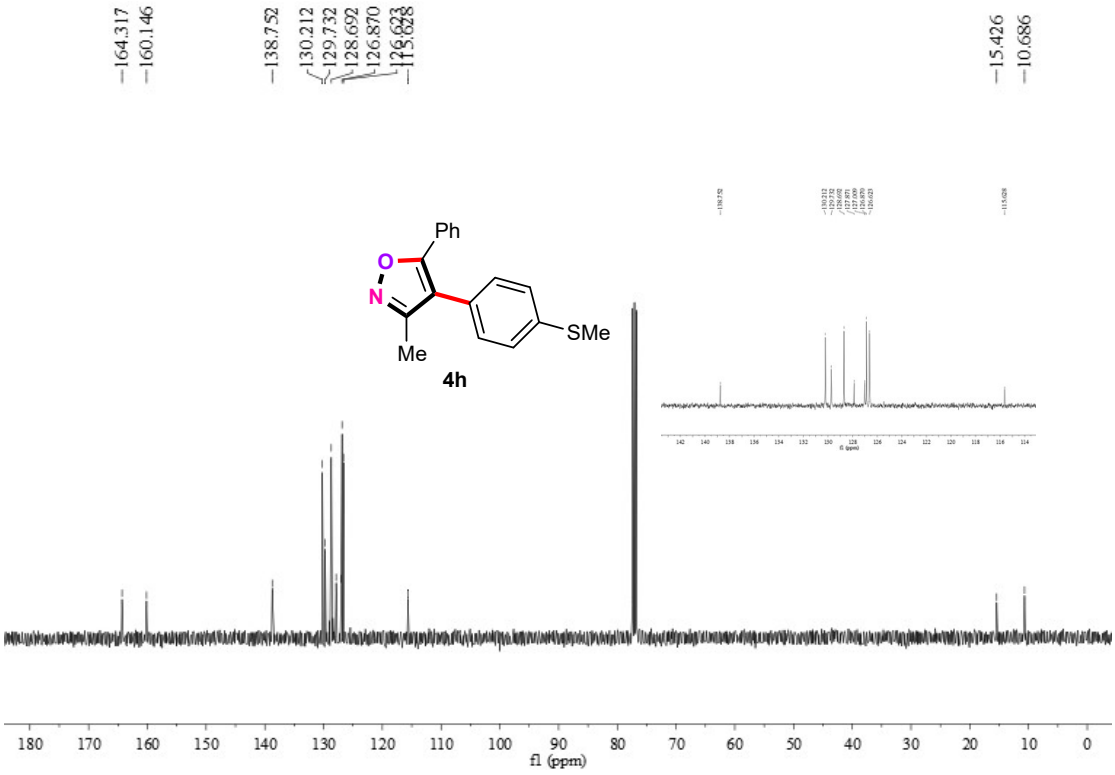
7.346
7.337
7.317
7.243
7.223
2.588
2.267
Ph
O
N
Me
SMe
4h
2.08
5.05
2.04
3.03
3.00
f1 (ppm)


164.317
160.146
138.752
130.212
129.732
128.692
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126.623
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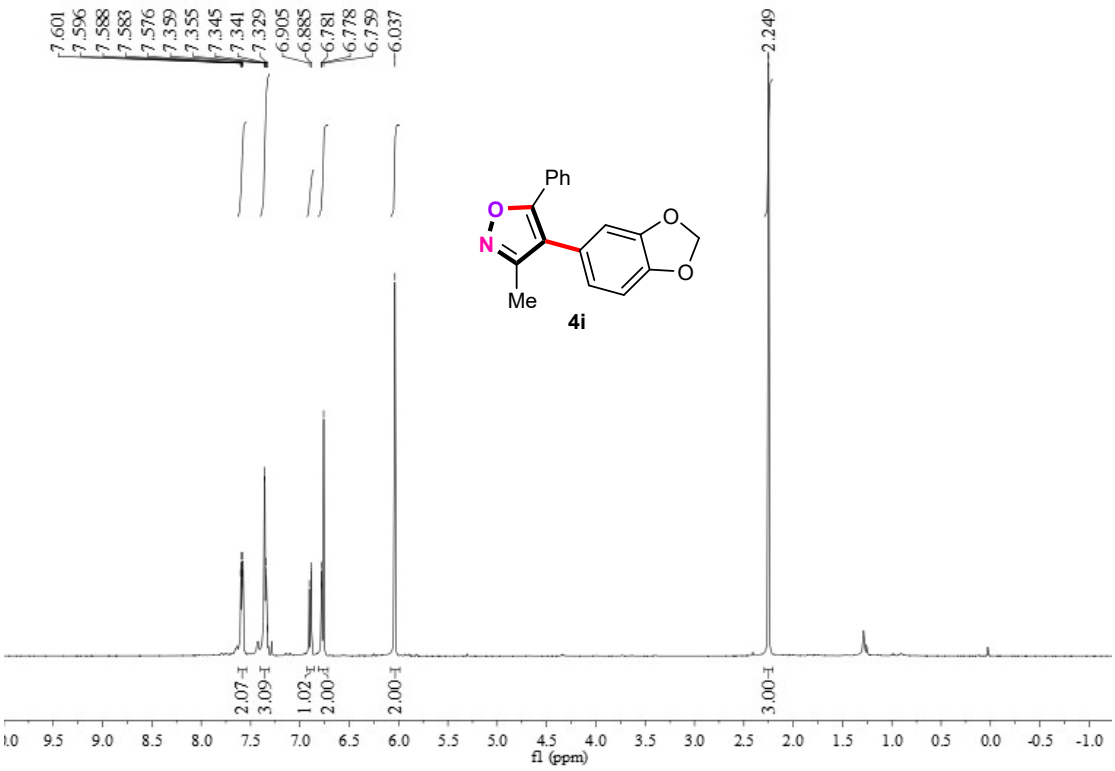
15.426
10.686
Ph
O
N
Me
SMe
4h
f1 (ppm)


7.601
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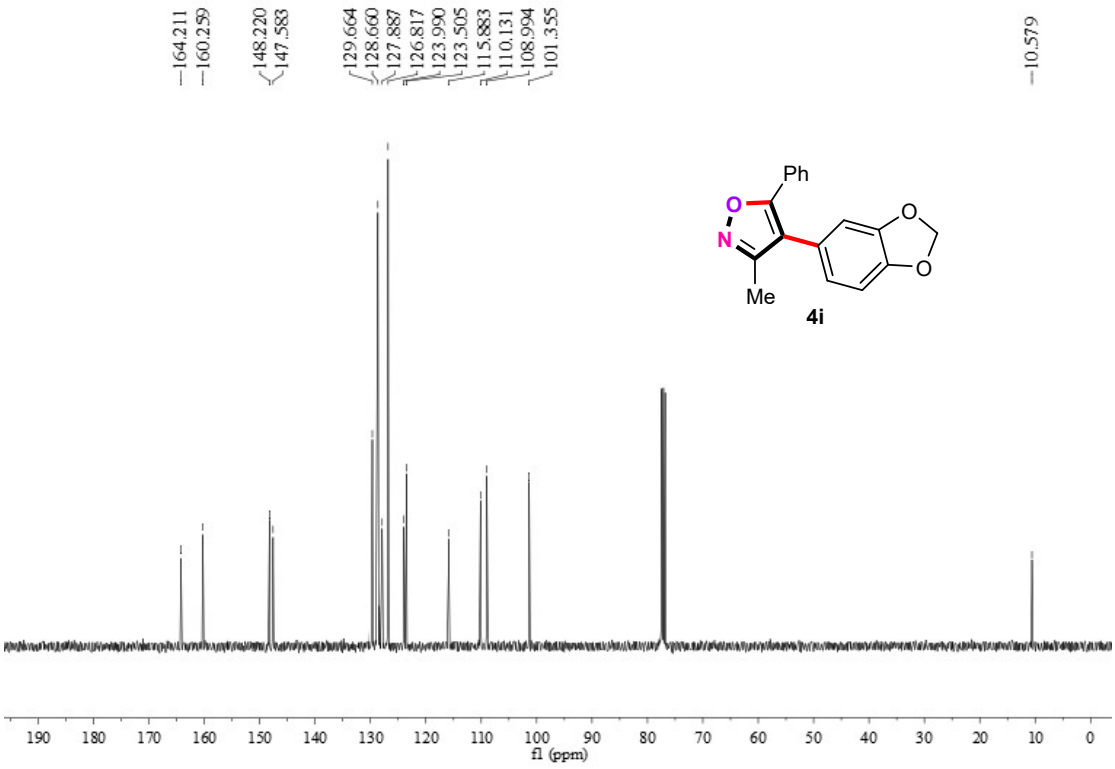
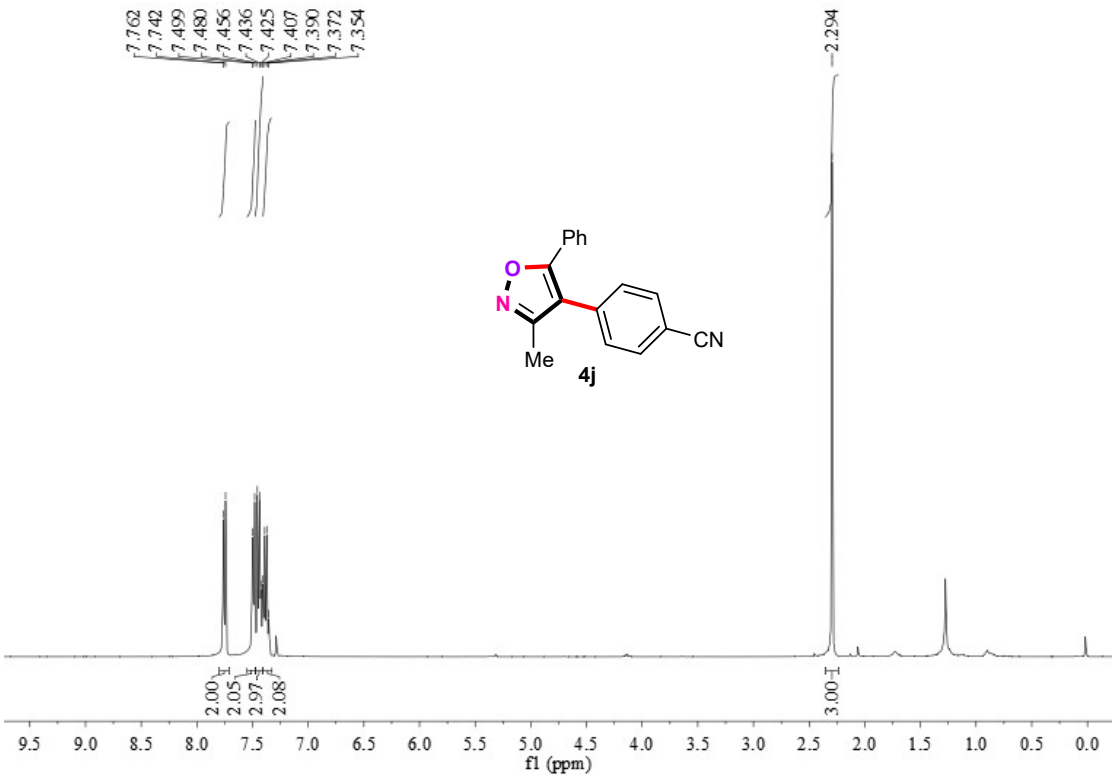
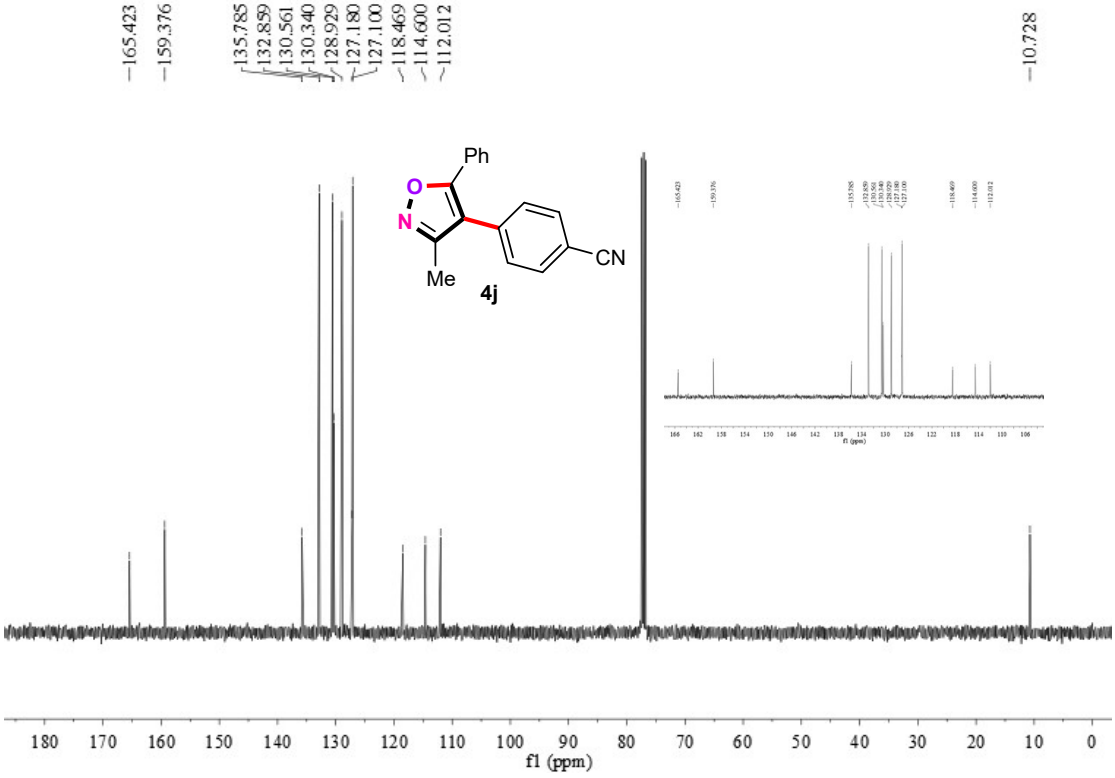
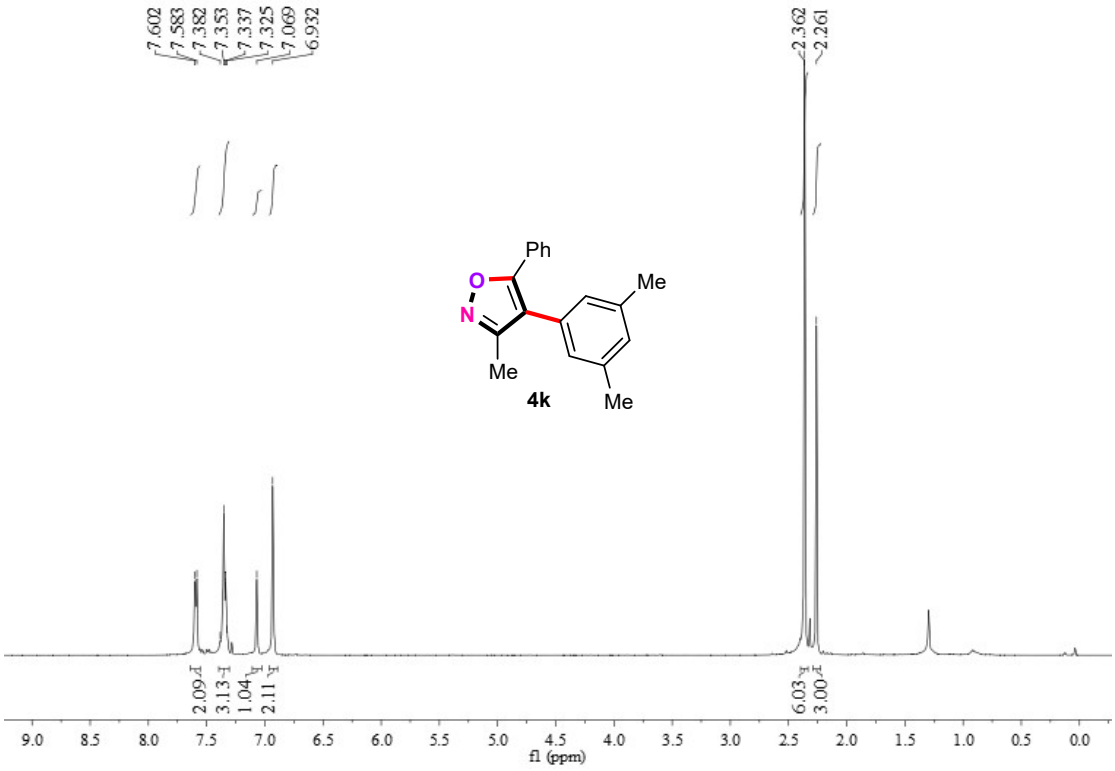
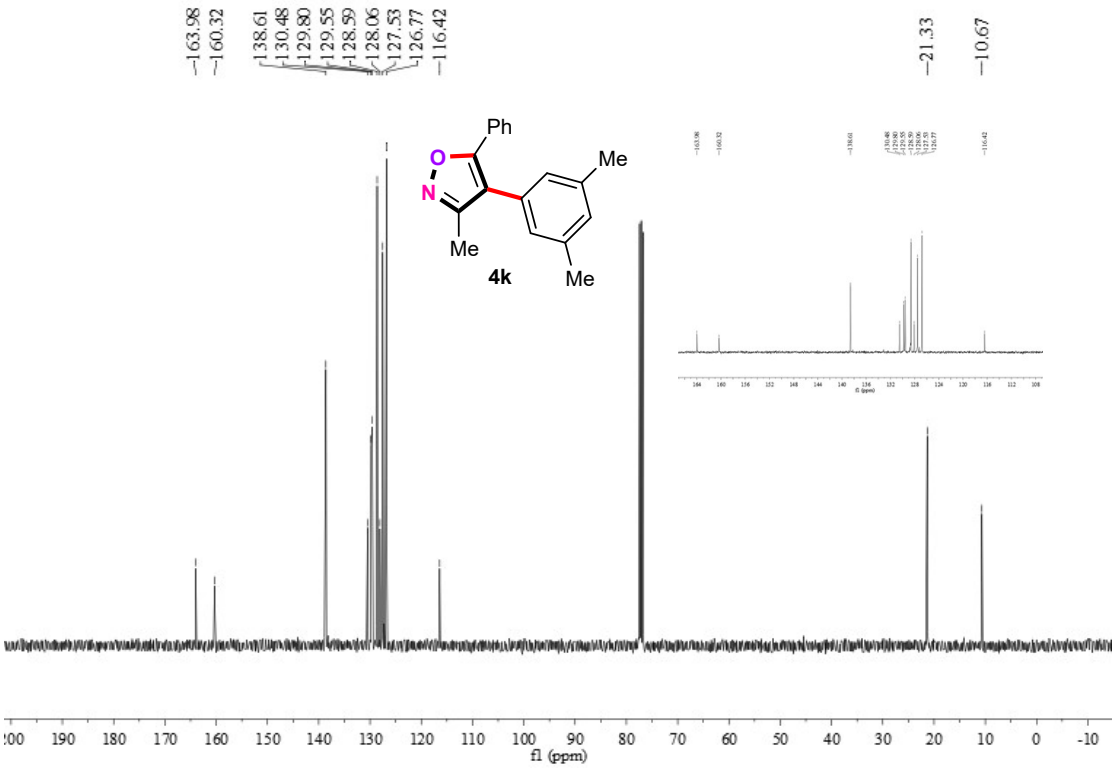
7.588
7.583
7.576
7.359
7.355
7.345
7.341
7.329
6.905
6.885
6.781
6.778
6.759
6.037
2.249
Ph
O
N
Me
O
O
4i
2.07
3.09
1.02
2.00
2.00
3.00
f1 (ppm)

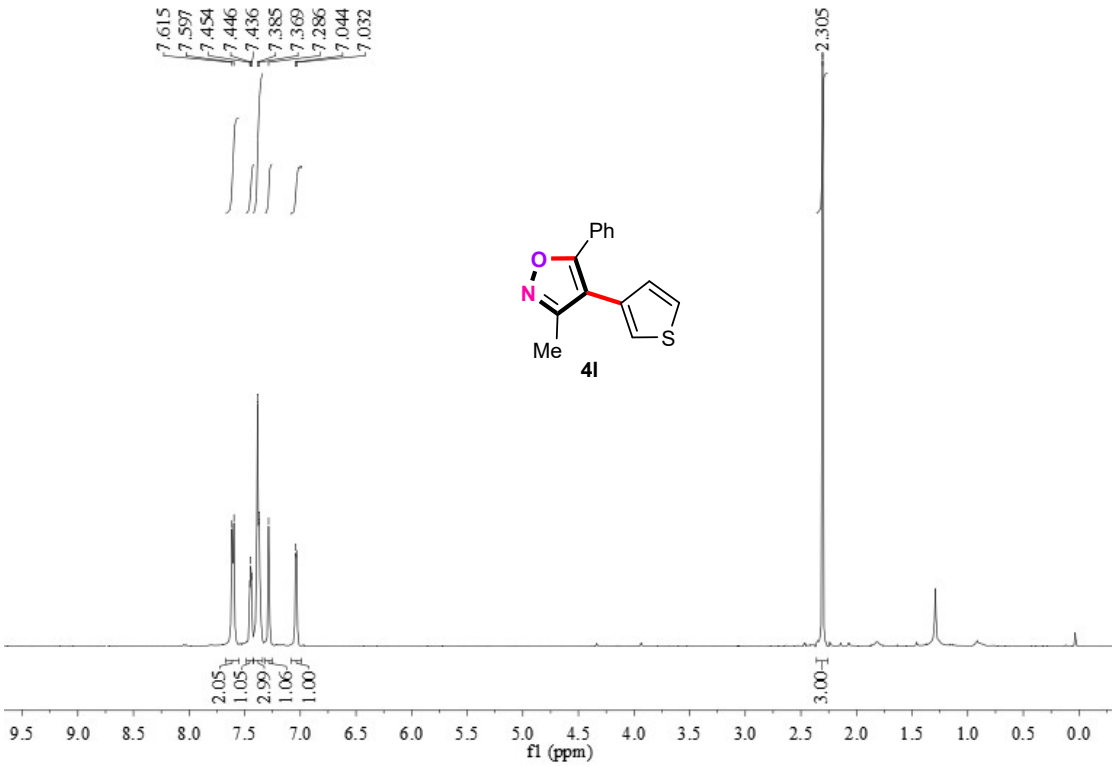

164.211
160.259
148.220
147.583
129.664
128.660
127.887
126.817
123.990
123.505
115.883
110.131
108.994
101.355
10.579
Ph
O
N
Me
O
O
4i
f1 (ppm)

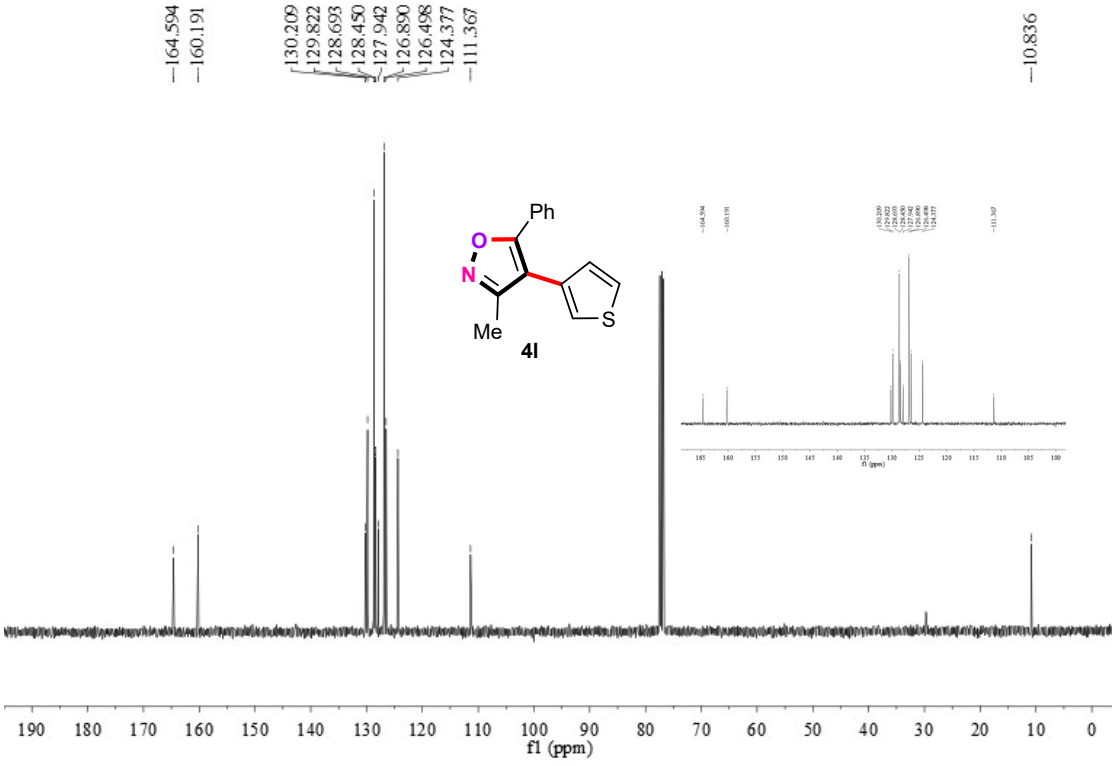

7.762
7.742
7.499
7.480
7.456
7.436
7.425
7.407
7.390
7.372
7.354
2.294
Ph
O
N
Me
CN
4j
2.00
2.05
2.97
2.08
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

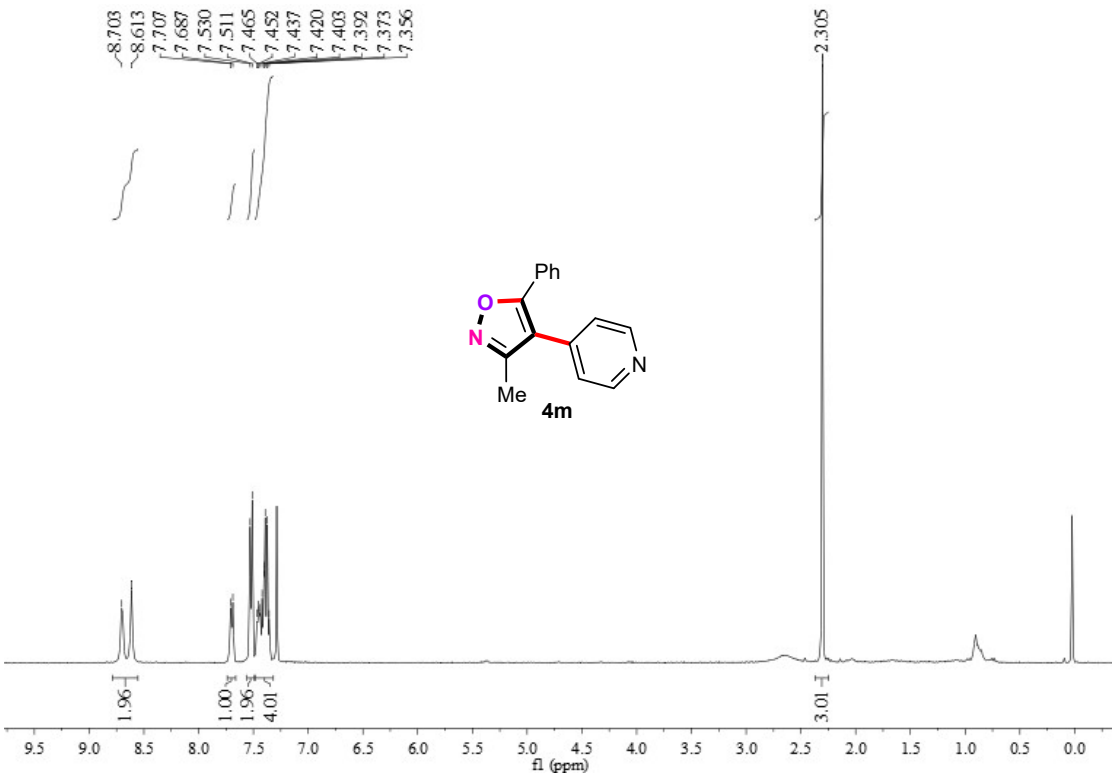

165.423
159.376
135.785
132.859
130.561
130.340
128.929
127.180
127.100
118.469
114.600
112.012
10.728
Ph
O
N
Me
CN
4j
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

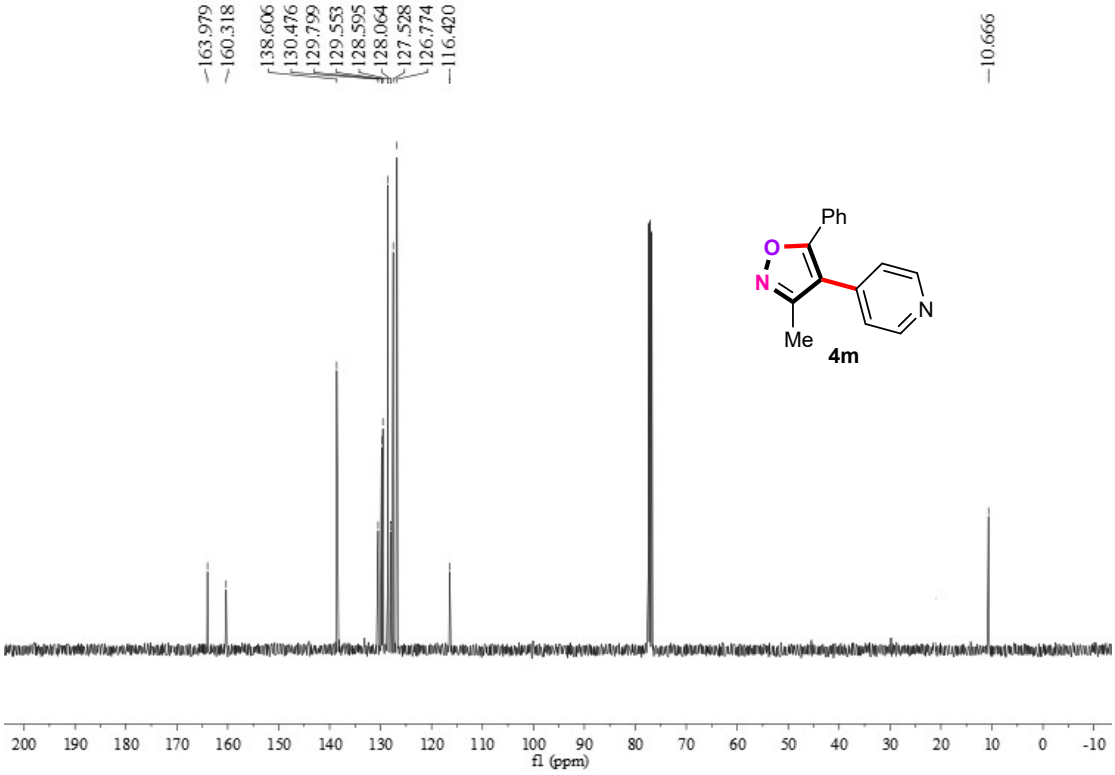

7.602
7.583
7.382
7.353
7.337
7.325
7.069
6.932
2.362
2.261
Ph
O
N
Me
Me
Me
4k
2.09
3.13
1.04
2.11
6.03
3.00
fl (ppm)

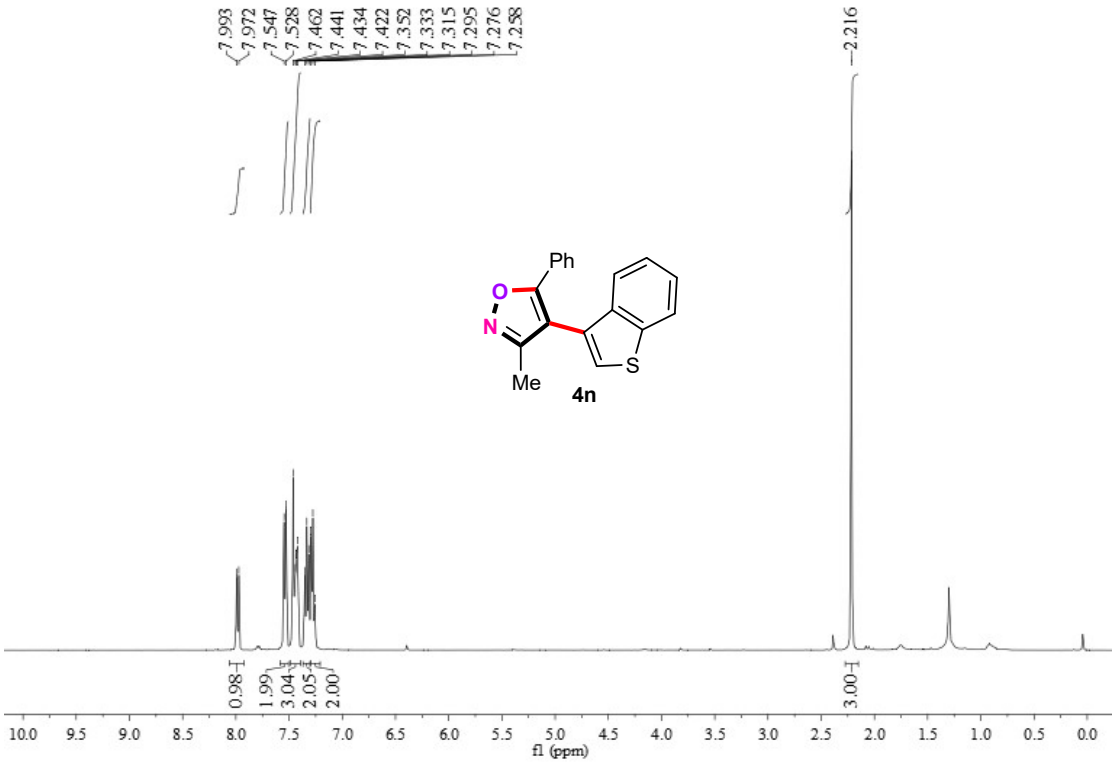

163.98
160.32
138.61
130.48
129.80
129.55
128.59
128.06
127.53
126.77
116.42
21.33
10.67
Ph
O
N
Me
Me
Me
4k
fl (ppm)

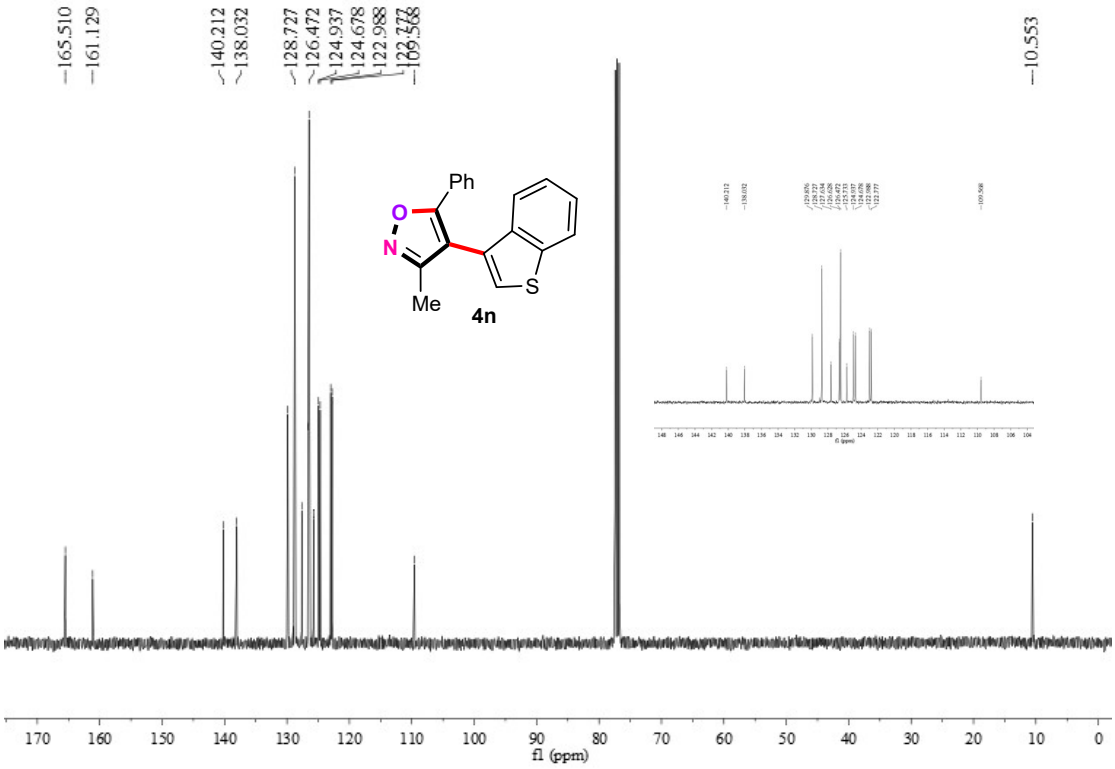

7.615
7.597
7.454
7.446
7.436
7.385
7.369
7.286
7.044
7.032
2.305
Ph
O
N
Me
S
4l
2.05
1.05
2.99
1.06
1.00
3.00
f1 (ppm)

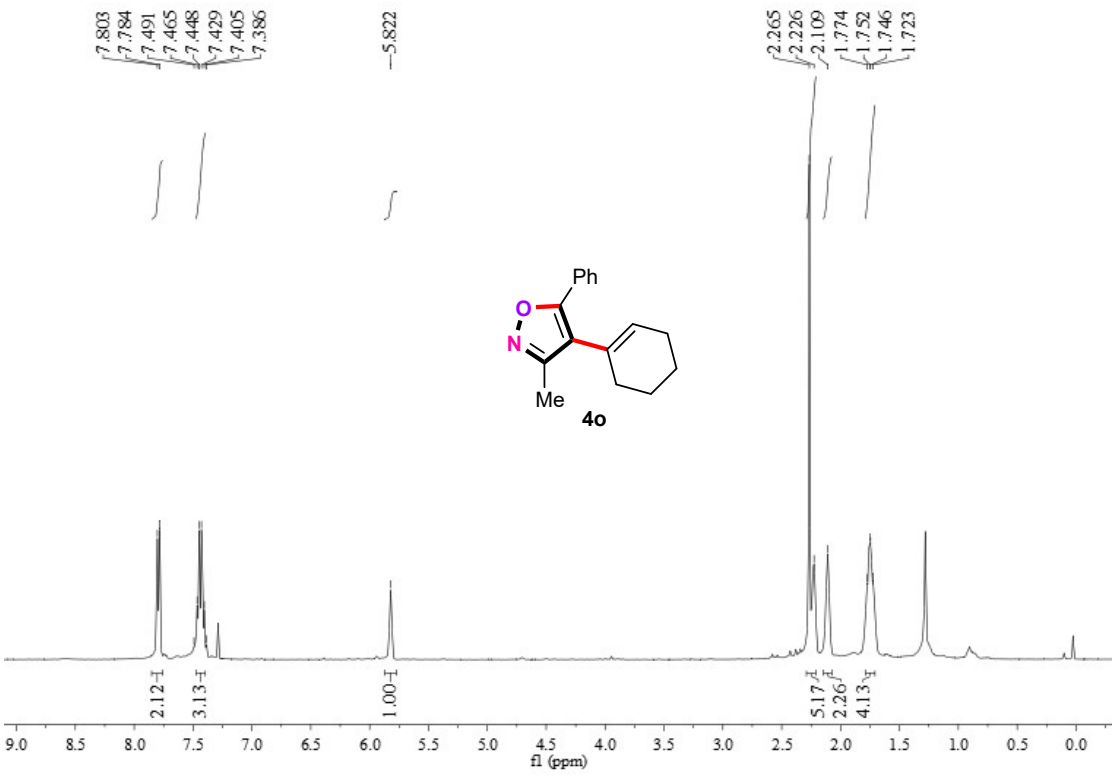

164.594
160.191
130.209
129.822
128.693
128.450
127.942
126.890
126.498
124.377
111.367
10.836
Ph
O
N
Me
S
4l
f1 (ppm)

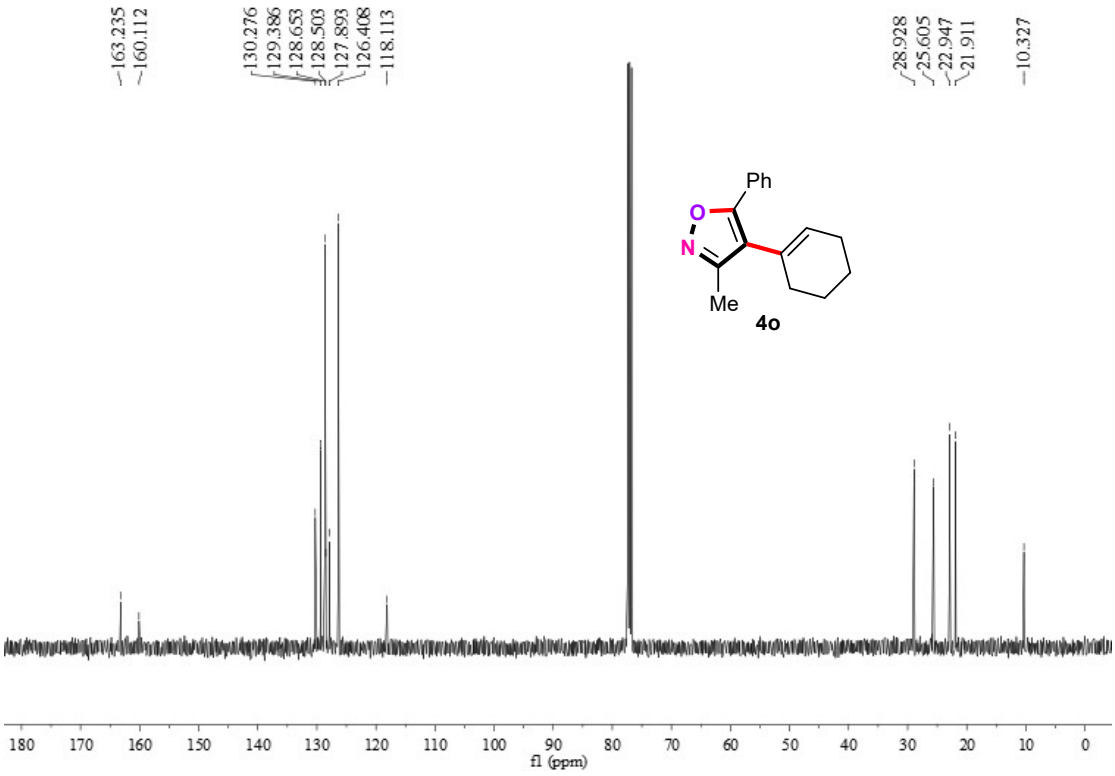

8.703
8.613
7.707
7.687
7.530
7.511
7.465
7.452
7.437
7.420
7.403
7.392
7.373
7.356
2.305
Ph
O
N
Me
N
4m
1.96
1.00
1.96
4.01
3.01
fl (ppm)


163.979
160.318
138.606
130.476
129.799
129.553
128.595
128.064
127.528
126.774
116.420
10.666
Ph
O
N
Me
N
4m
fl (ppm)

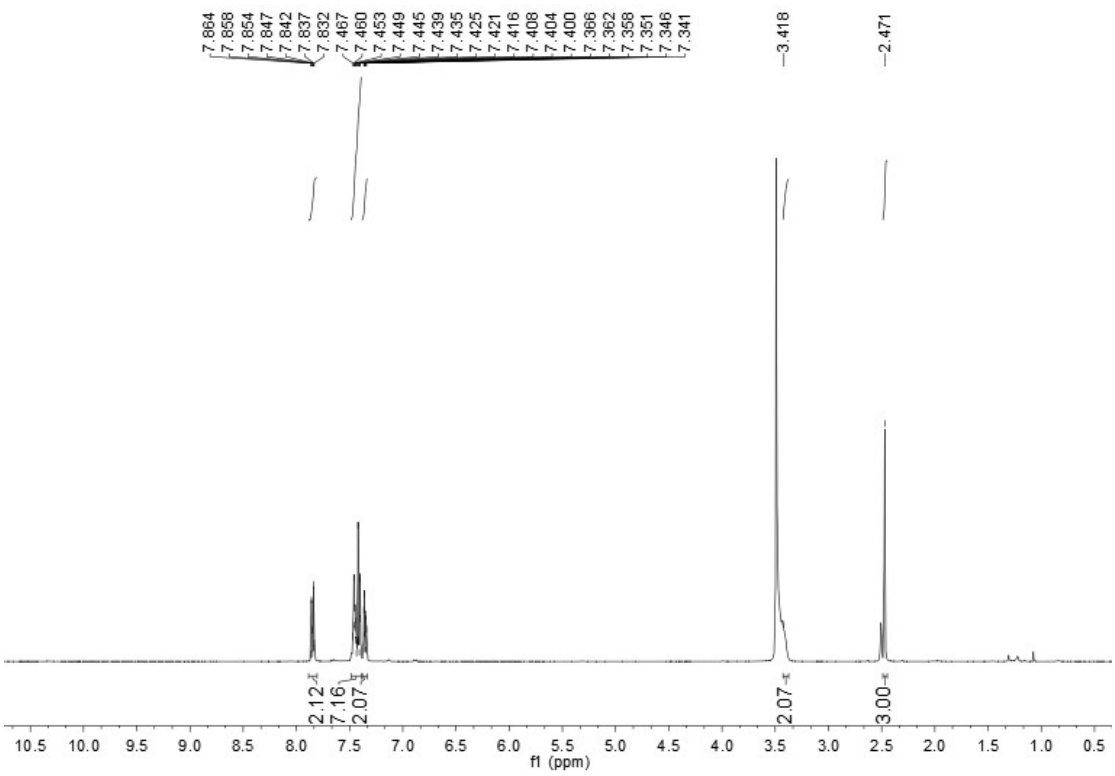

7.993
7.972
7.547
7.528
7.462
7.441
7.434
7.422
7.352
7.333
7.315
7.295
7.276
7.258
2.216
Ph
O
N
Me
S
4n
0.98
1.99
3.04
2.05
2.00
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

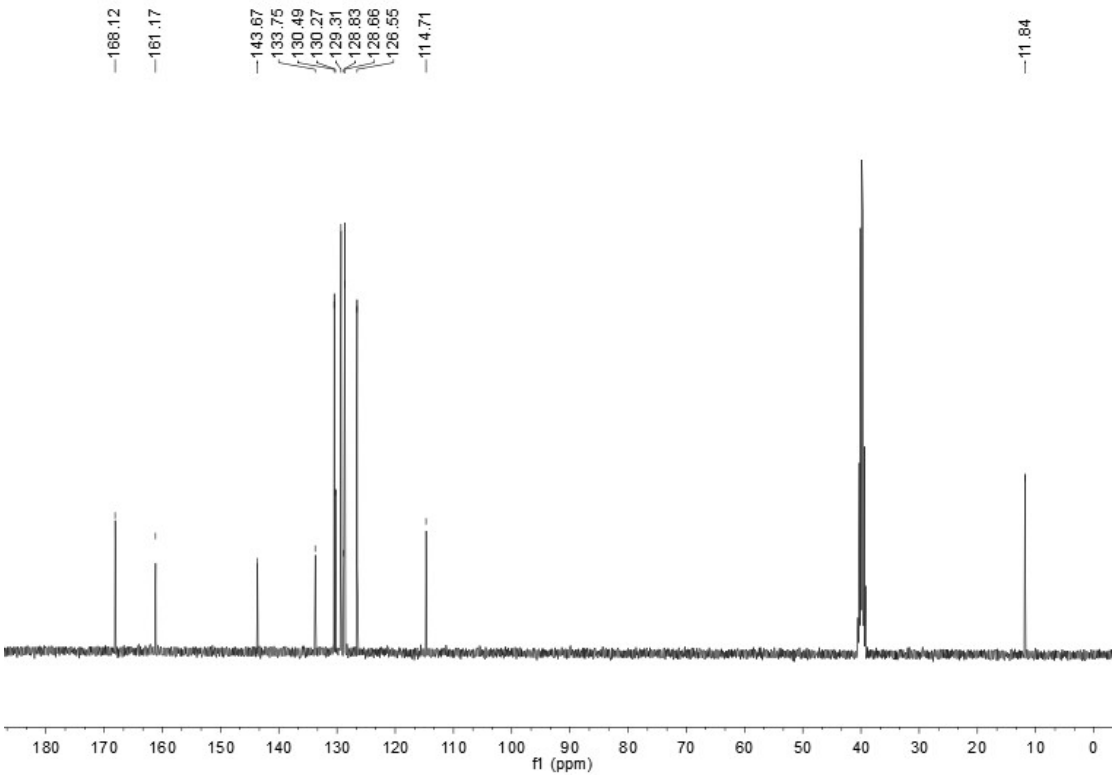

165.510
161.129
140.212
138.032
128.727
126.472
124.937
124.678
122.988
10.553
Ph
O
N
Me
S
4n
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



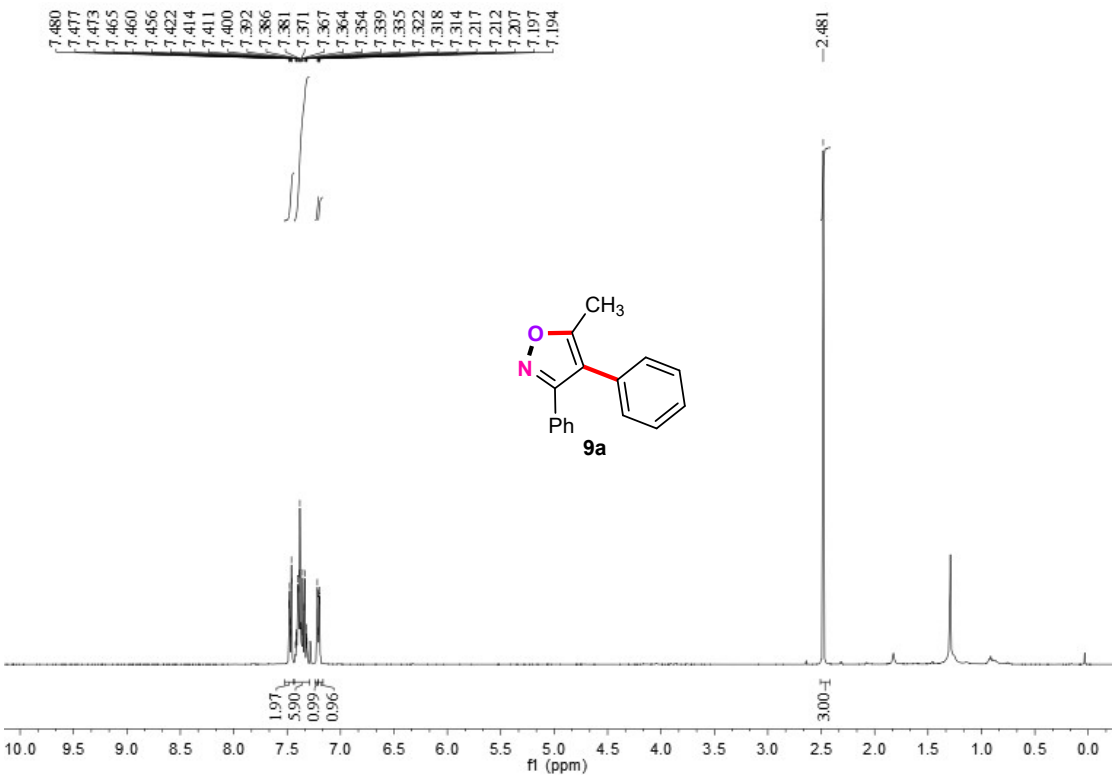



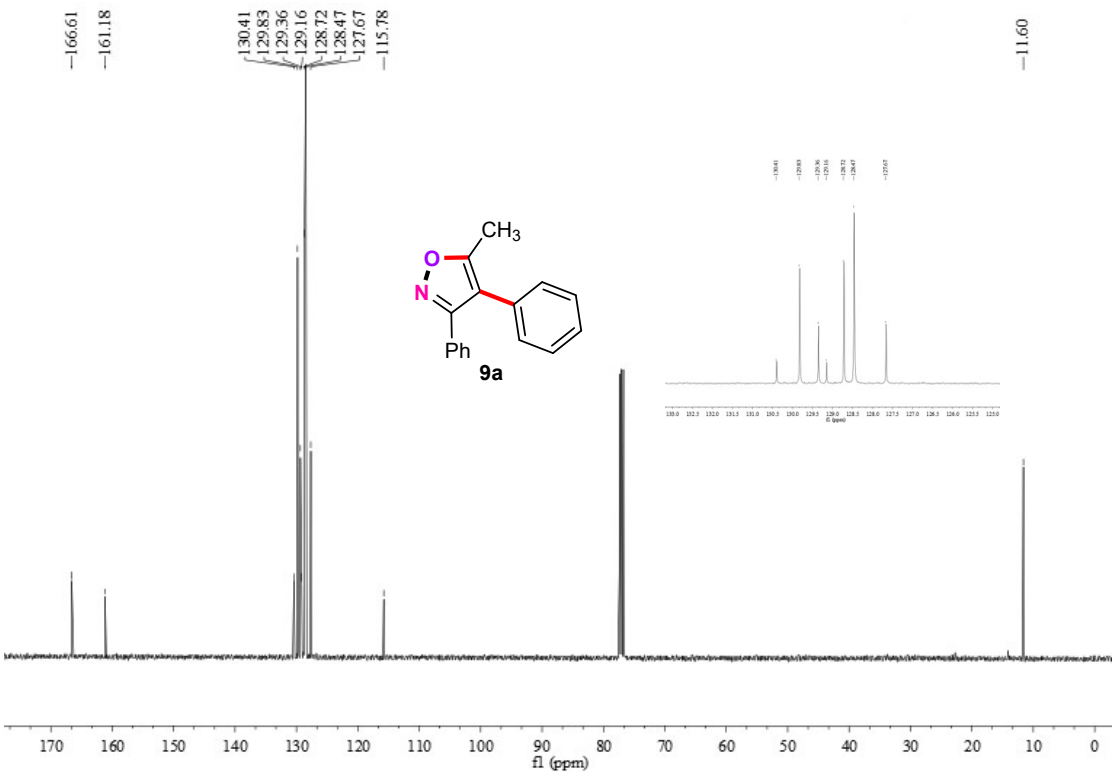

**$^{1}$H and $^{13}$C NMR spectra of compounds 8**

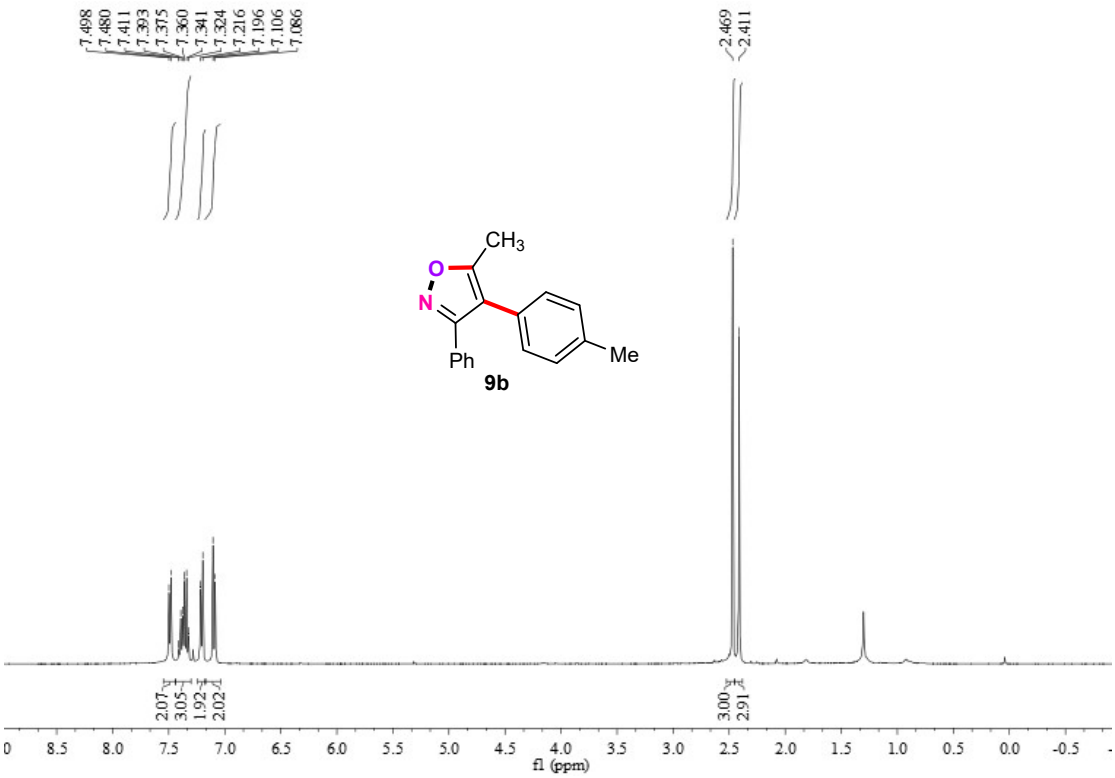


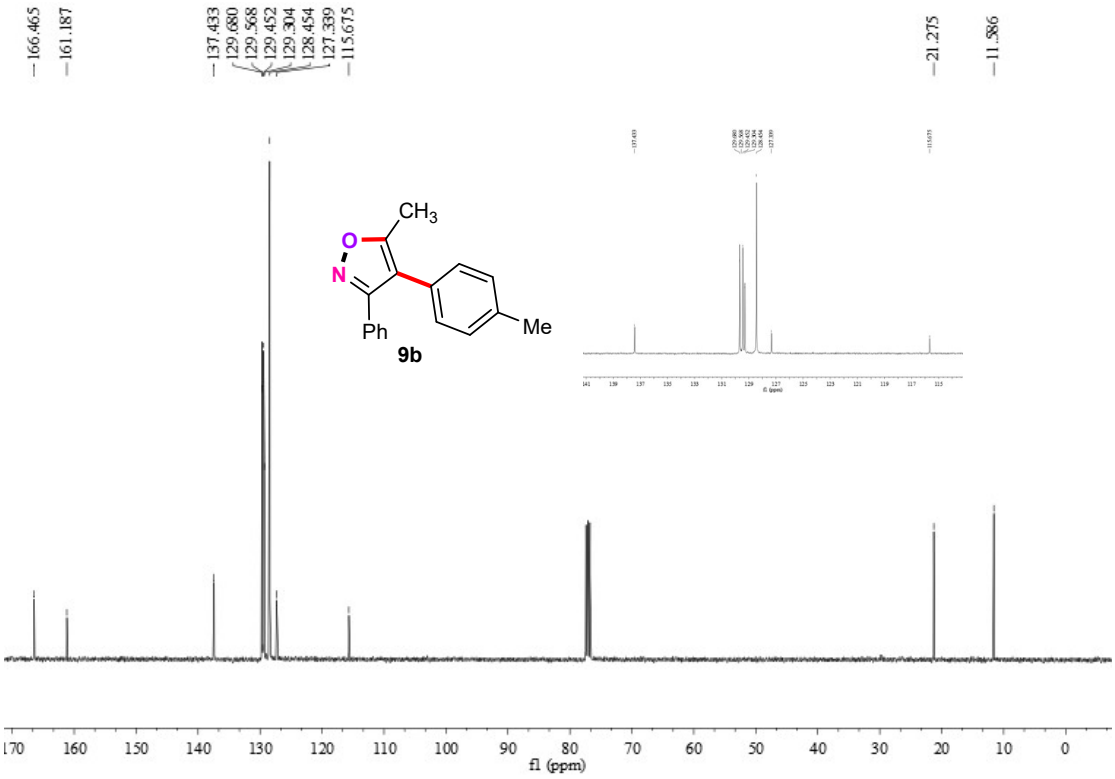



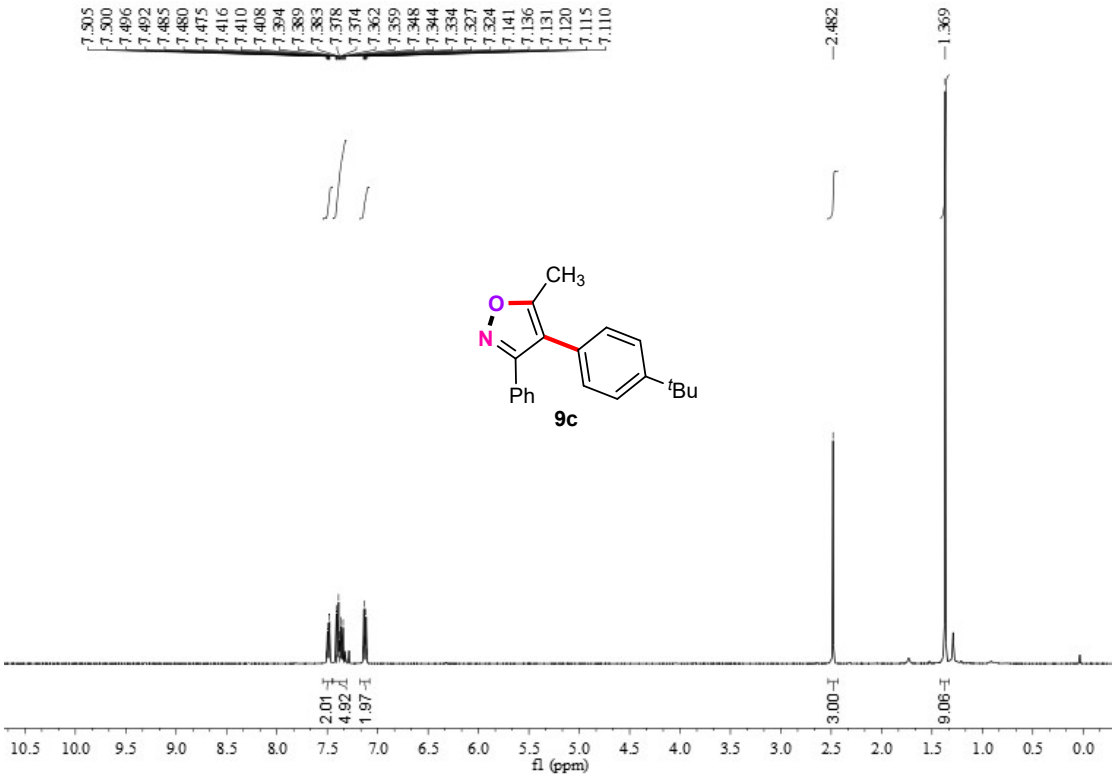

**$^{1}$H and $^{13}$C NMR spectra of compounds 9**

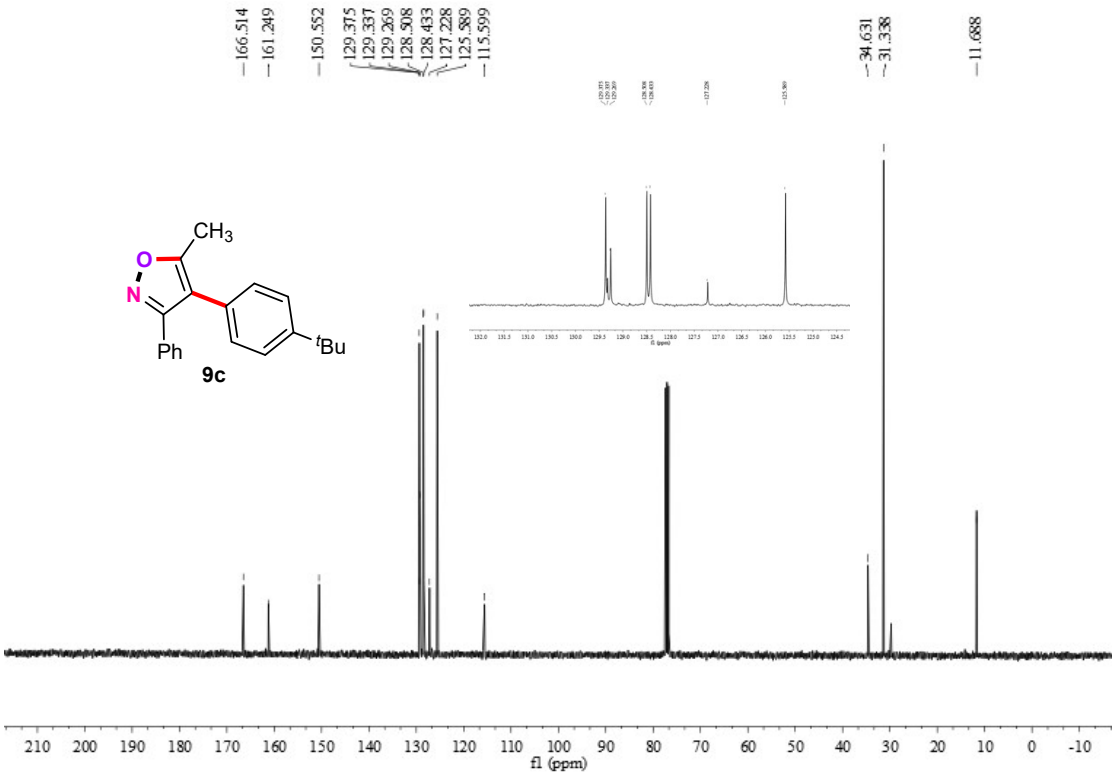

7.480
7.477
7.473
7.465
7.460
7.456
7.422
7.414
7.411
7.400
7.392
7.386
7.381
7.371
7.367
7.364
7.354
7.339
7.335
7.322
7.318
7.314
7.217
7.212
7.207
7.197
7.194
2.481
CH3
O
N
Ph
9a
1.97
5.90
0.99
0.96
3.00
f1 (ppm)

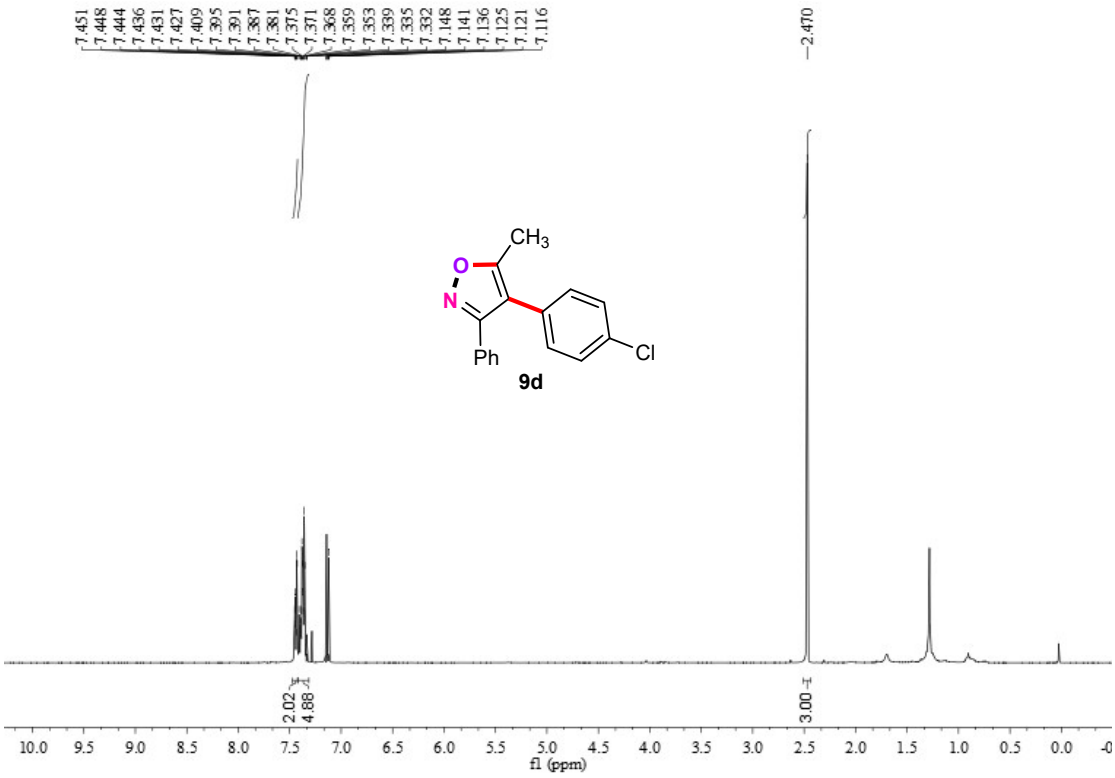

166.61
161.18
130.41
129.83
129.36
129.16
128.72
128.47
127.67
115.78
11.60
CH3
O
N
Ph
9a
f1 (ppm)

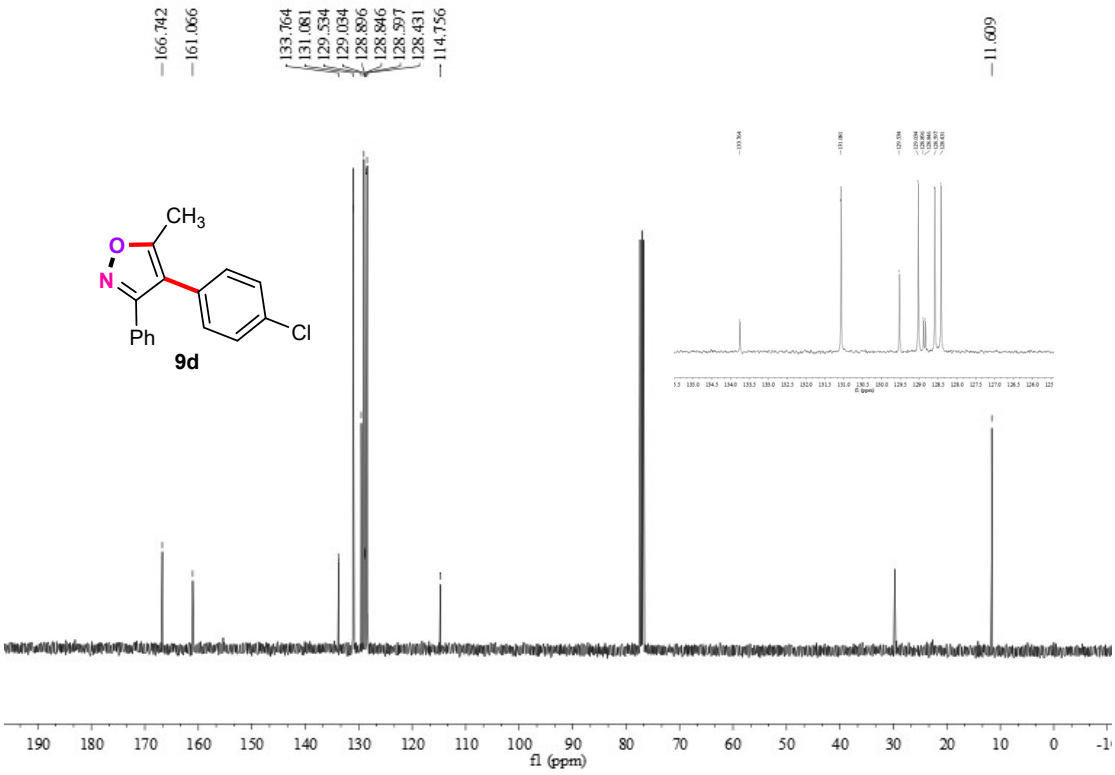

7.498
7.480
7.411
7.393
7.375
7.360
7.341
7.324
7.216
7.196
7.106
7.086
2.469
2.411
CH3
O
N
Ph
Me
9b
2.07
3.05
1.92
2.02
3.00
2.91
fl (ppm)

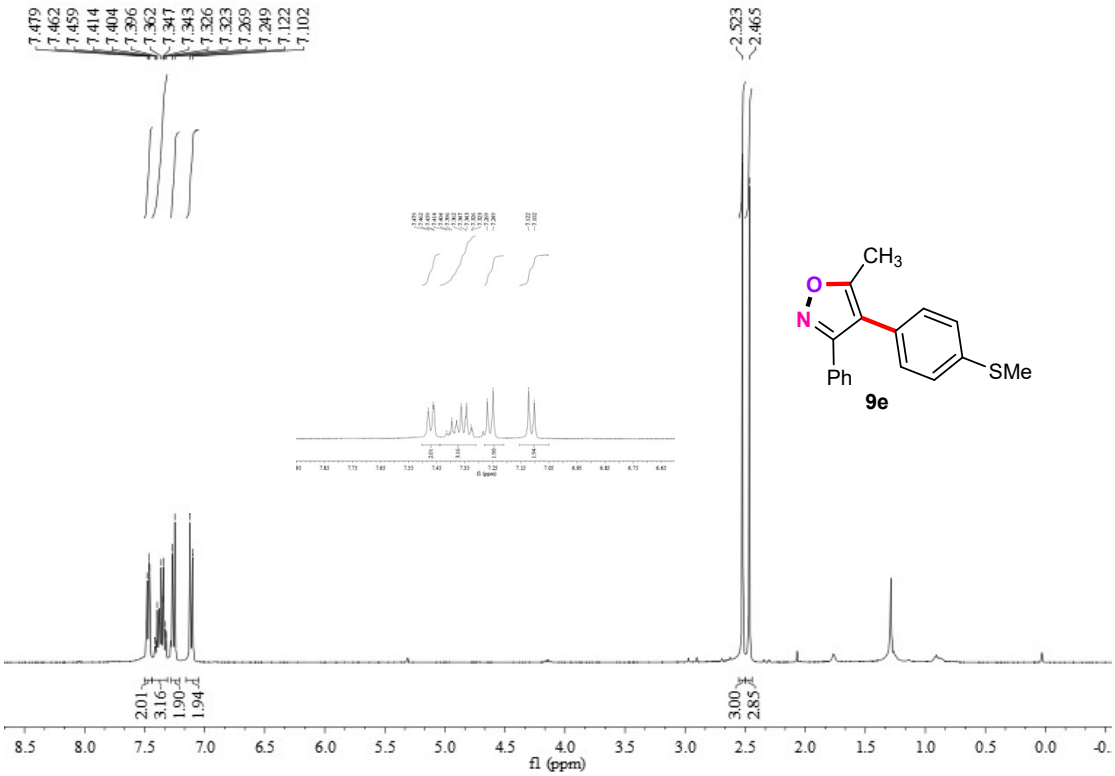

166.465
161.187
137.433
129.680
129.568
129.452
129.304
128.454
127.339
115.675
21.275
11.586
CH3
O
N
Ph
Me
9b
fl (ppm)

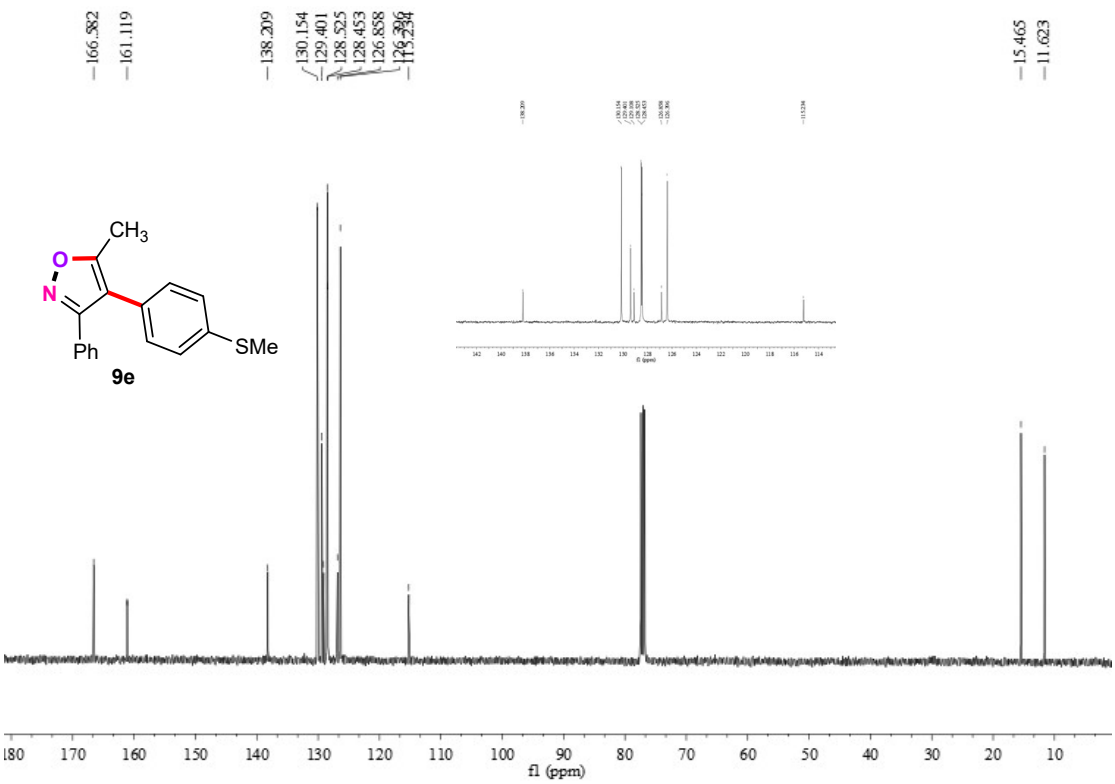

7.505
7.500
7.496
7.492
7.485
7.480
7.475
7.416
7.410
7.408
7.394
7.389
7.383
7.378
7.374
7.362
7.359
7.348
7.344
7.334
7.327
7.324
7.141
7.136
7.131
7.120
7.115
7.110
2.482
1.369
CH3
O
N
Ph
tBu
9c
2.01
4.92
1.97
3.00
9.06
fl (ppm)

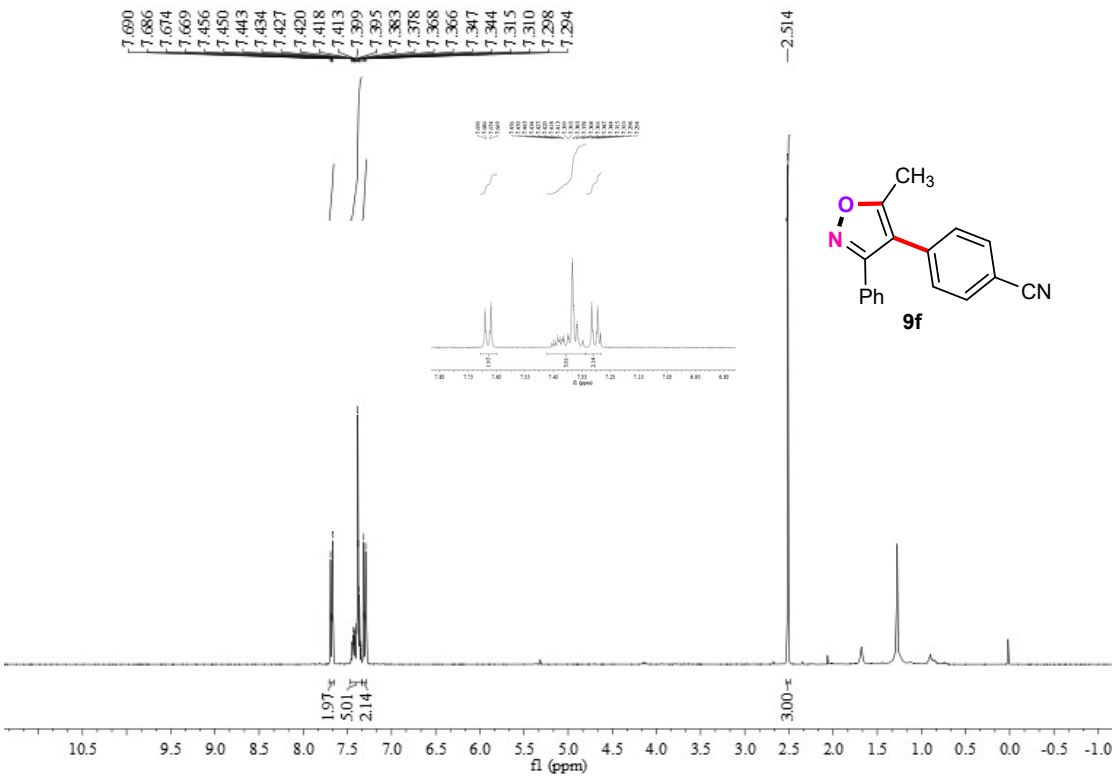

166.514
161.249
150.552
129.375
129.337
129.269
128.908
128.433
127.228
125.389
115.599
34.631
31.338
11.688
CH3
O
N
Ph
tBu
9c
fl (ppm)

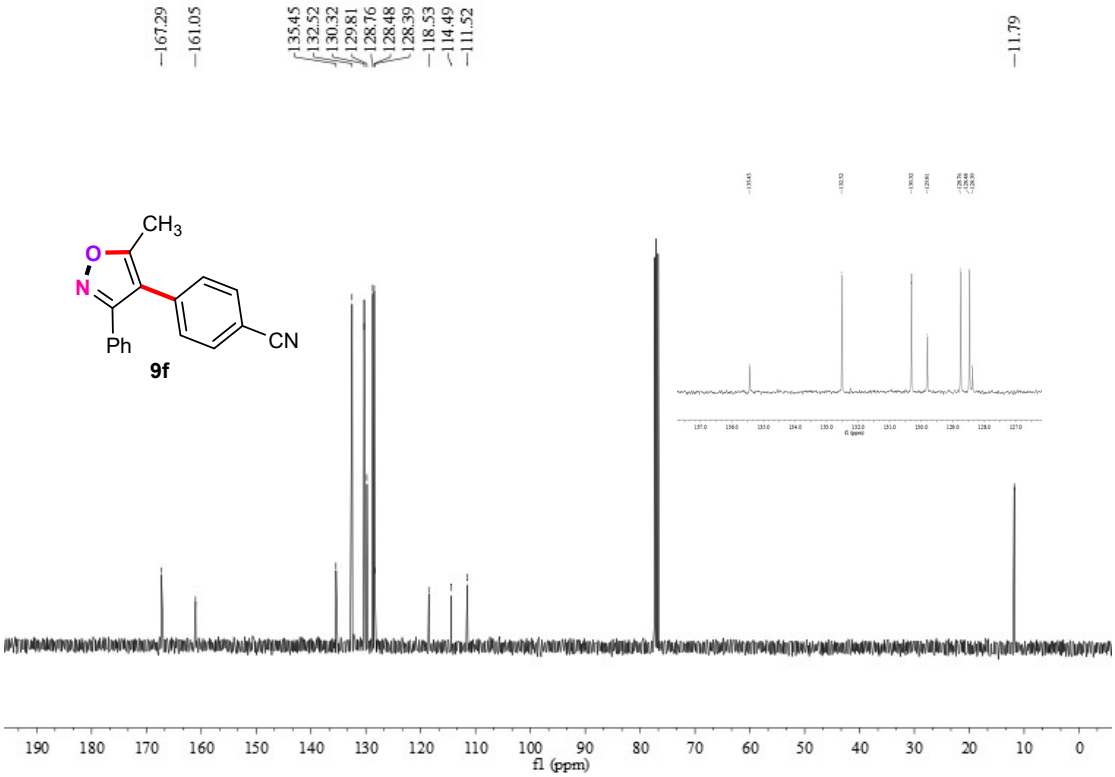

7.451
7.448
7.444
7.436
7.431
7.427
7.409
7.395
7.391
7.387
7.381
7.375
7.371
7.368
7.359
7.353
7.339
7.335
7.332
7.148
7.141
7.136
7.125
7.121
7.116
2.470
CH3
O
N
Ph
Cl
9d
2.02
4.88
3.00
fl (ppm)

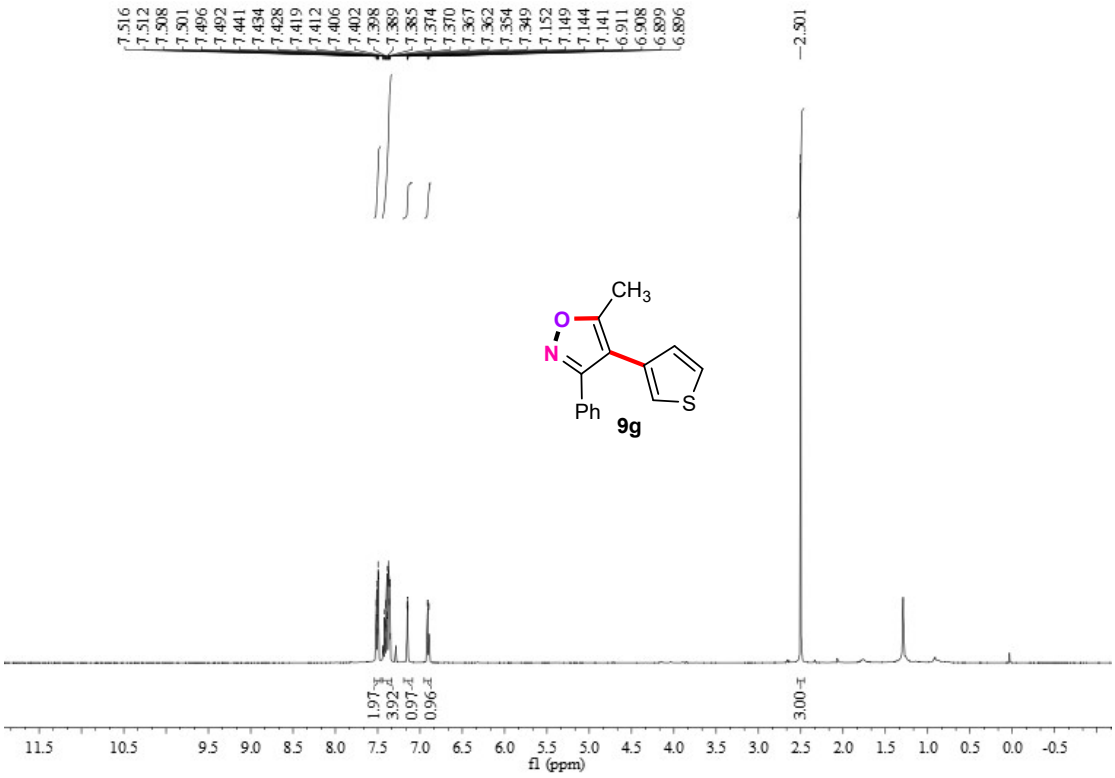

166.742
161.066
133.764
131.081
129.534
129.034
128.896
128.846
128.597
128.431
114.756
11.609
CH3
O
N
Ph
Cl
9d
fl (ppm)

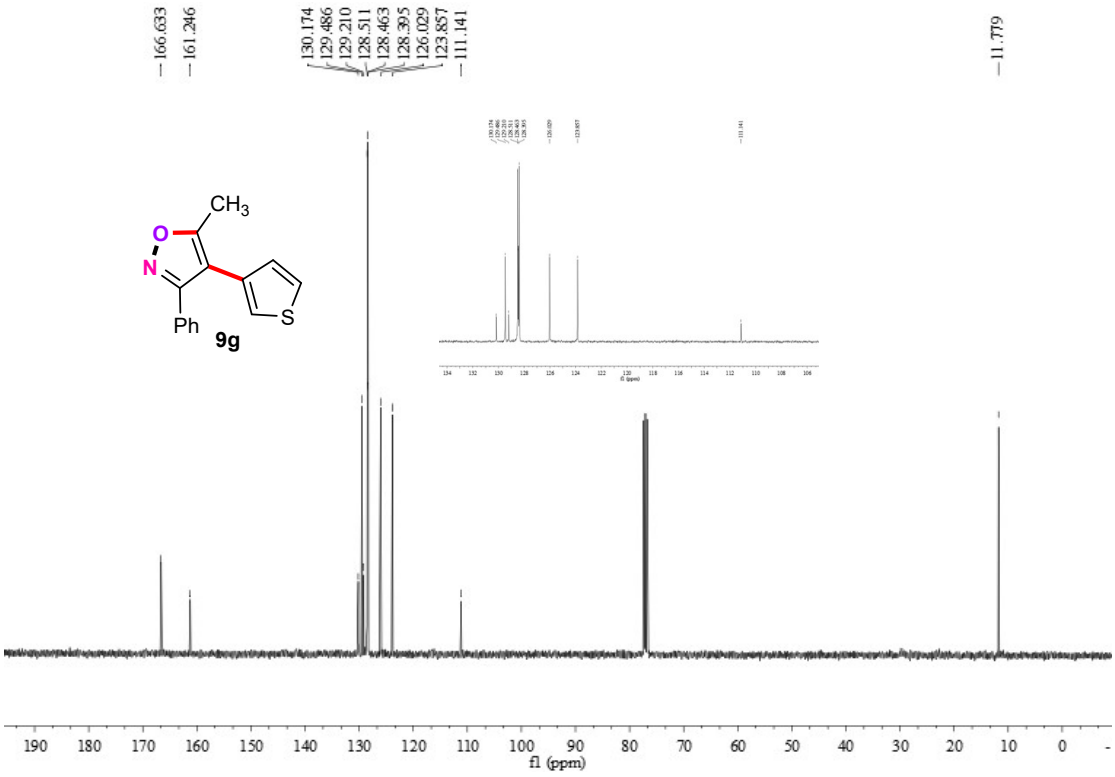

7.479
7.462
7.459
7.414
7.404
7.396
7.362
7.347
7.343
7.326
7.323
7.269
7.249
7.122
7.102
2.523
2.465
CH3
O
N
Ph
SMe
9e
2.01
3.16
1.90
1.94
3.00
2.85
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.382
161.119
138.209
130.154
129.401
128.525
128.453
126.858
115.234
15.465
11.623
CH3
O
N
Ph
SMe
9e
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


7.690
7.686
7.674
7.669
7.456
7.450
7.443
7.434
7.427
7.420
7.418
7.413
7.399
7.395
7.383
7.378
7.368
7.366
7.347
7.344
7.315
7.310
7.298
7.294
2.514
CH3
O
N
Ph
CN
9f
1.97
5.01
2.14
3.00
fl (ppm)

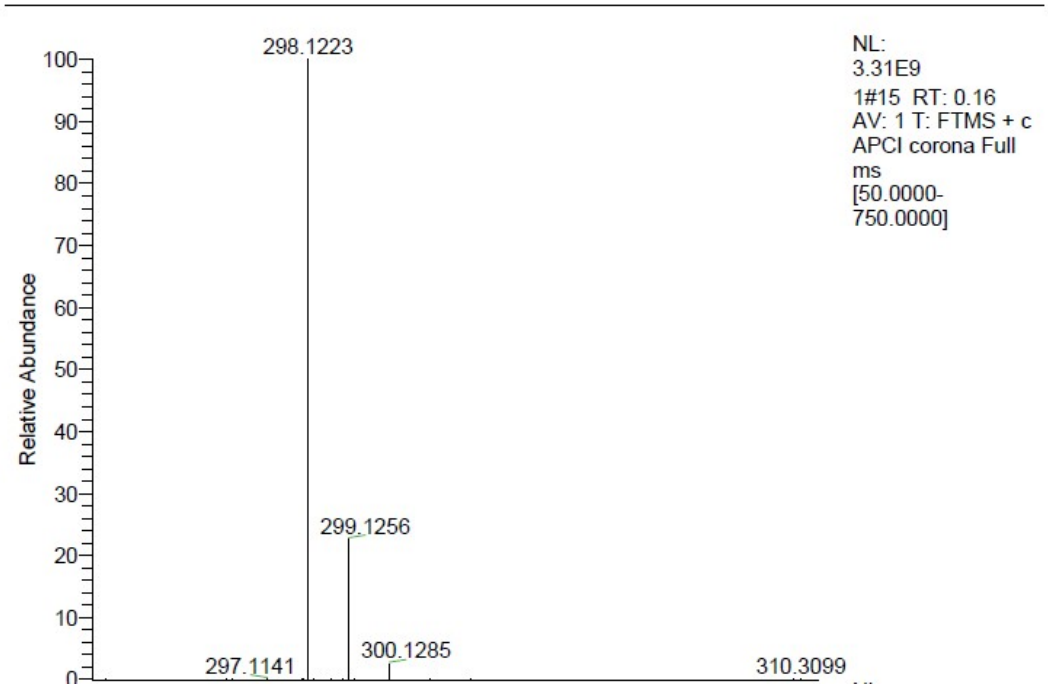

167.29
161.05
135.45
132.52
130.32
129.81
128.76
128.48
128.39
118.53
114.49
111.52
11.79
CH3
O
N
Ph
CN
9f
fl (ppm)



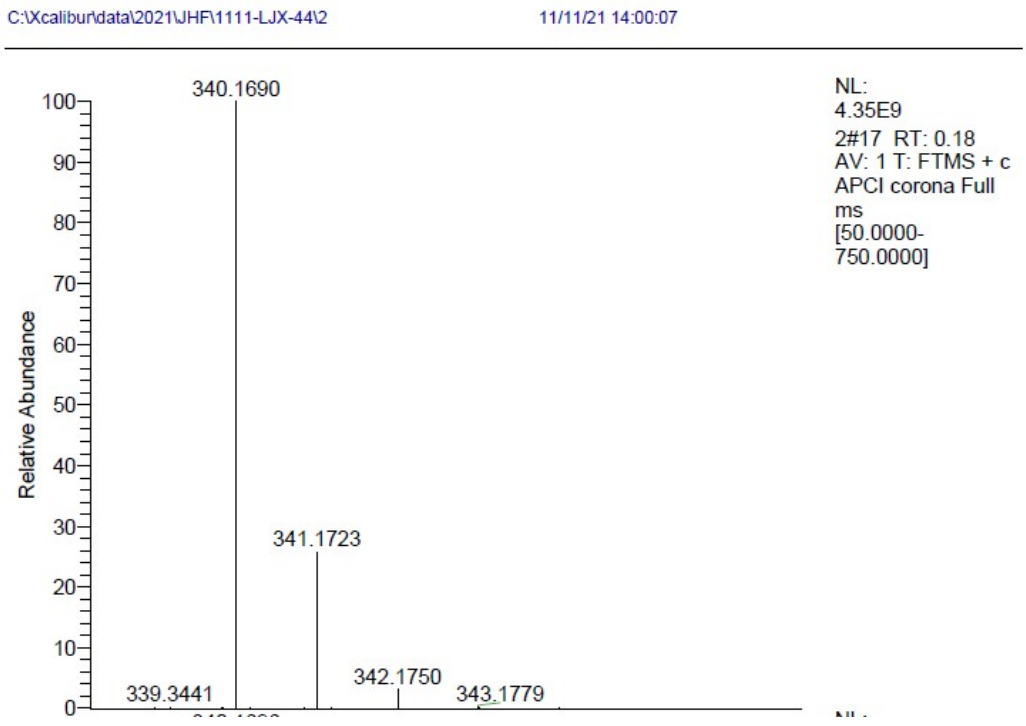




## HR-MS spectra of all compounds

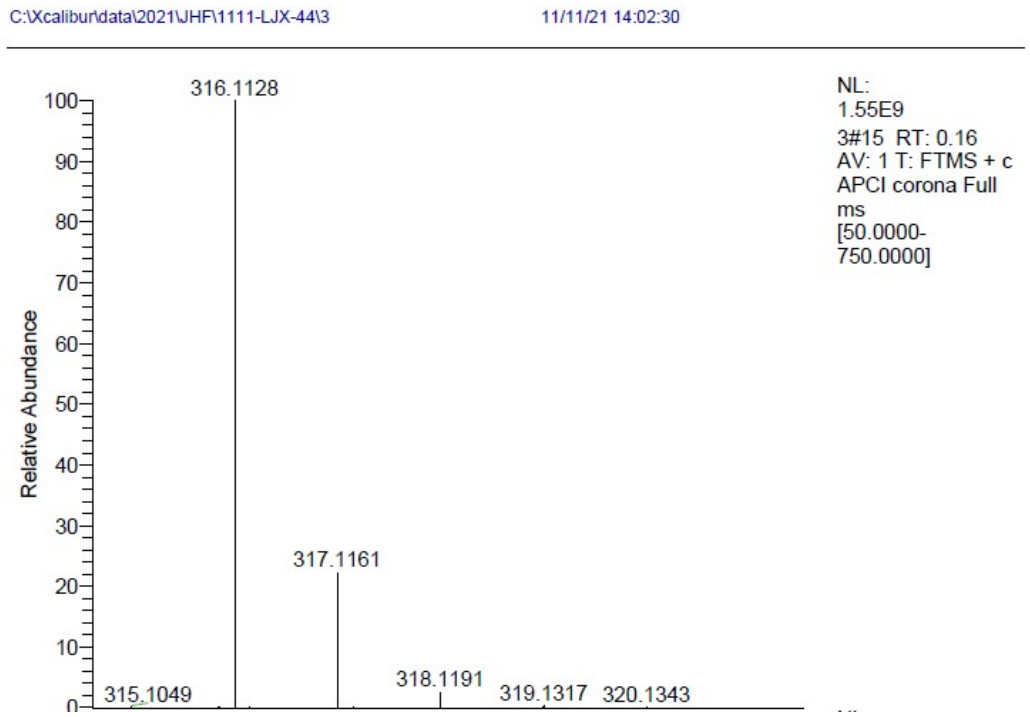
## The HR-MS spectra of 3a



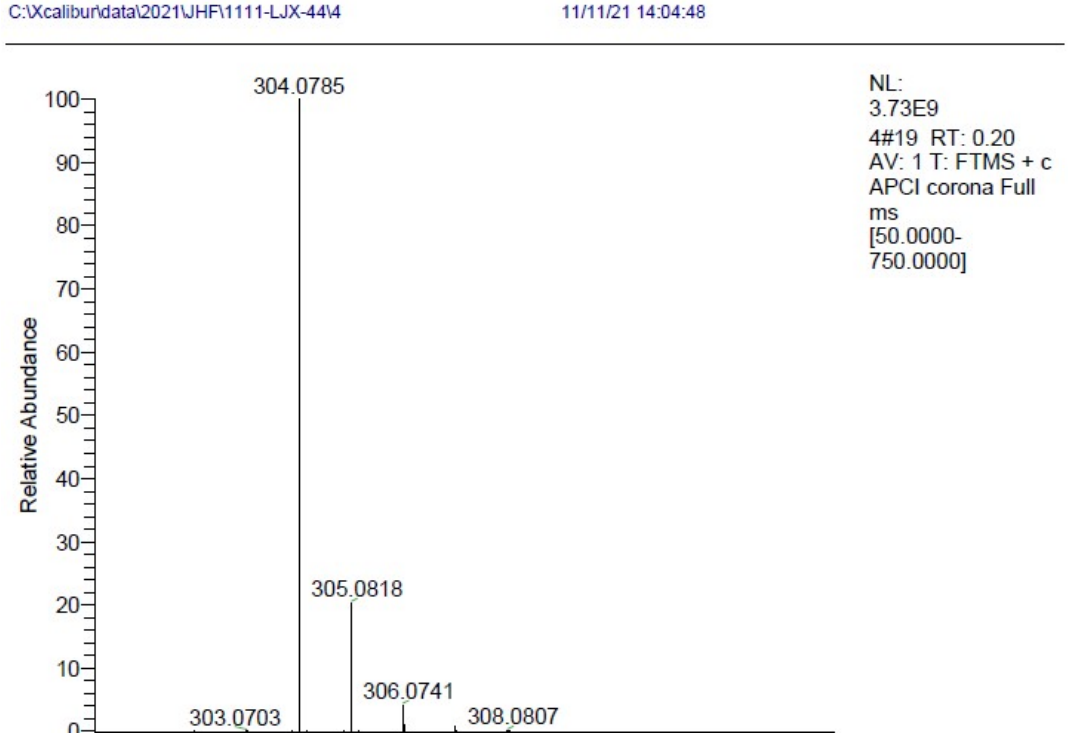

## The HR-MS spectra of 3b



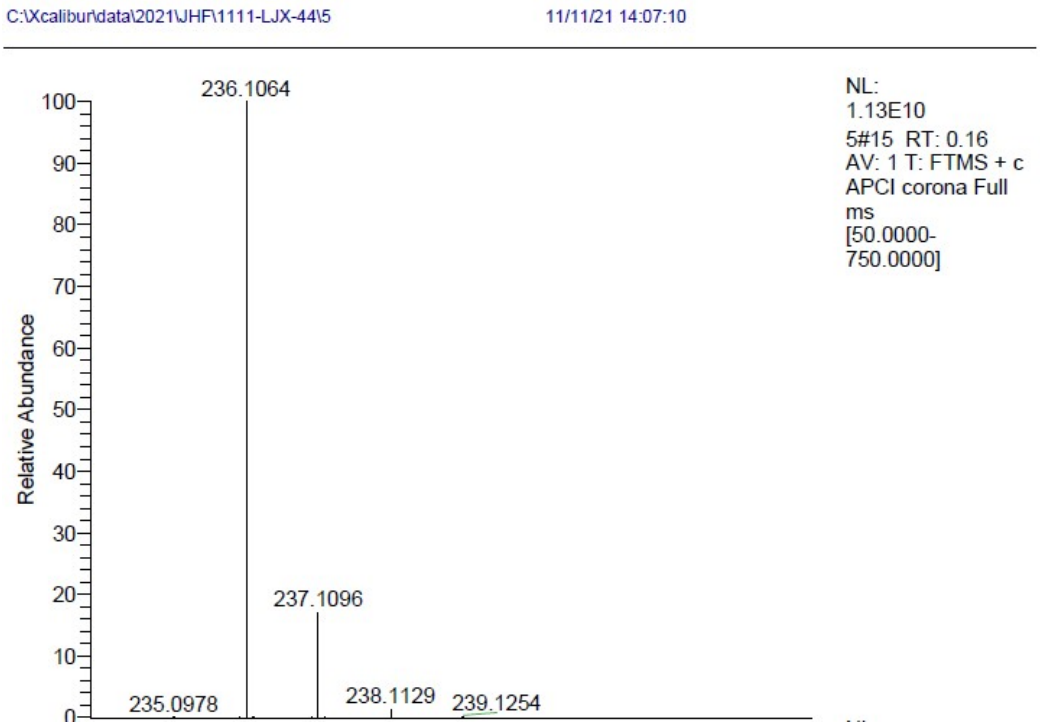
## The HR-MS spectra of 3c



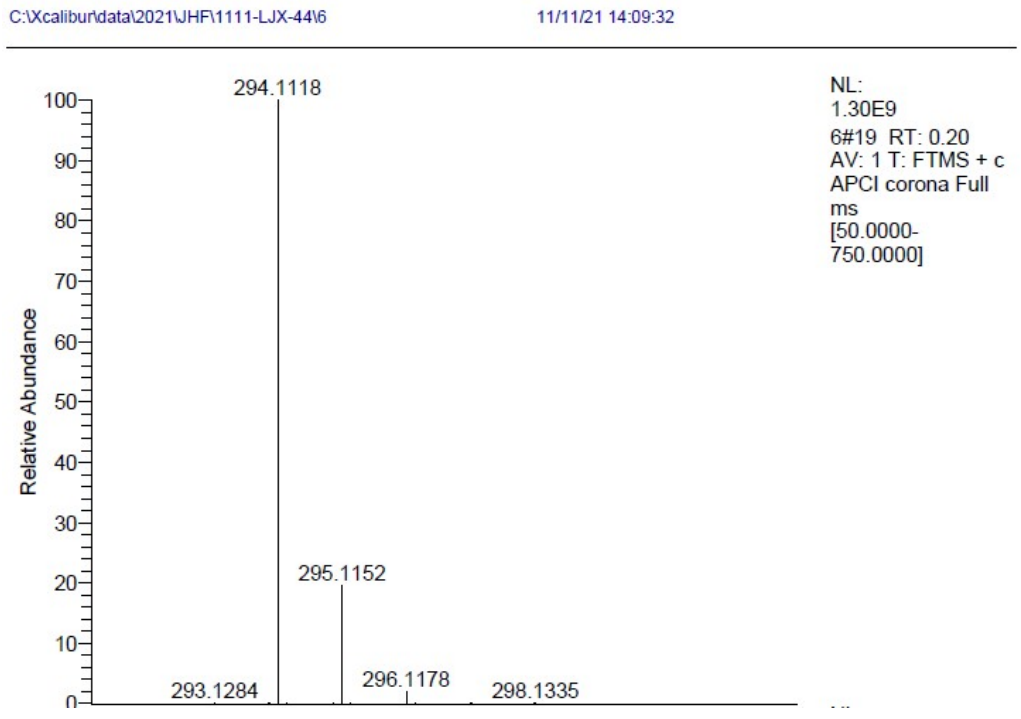

## The HR-MS spectra of 3d



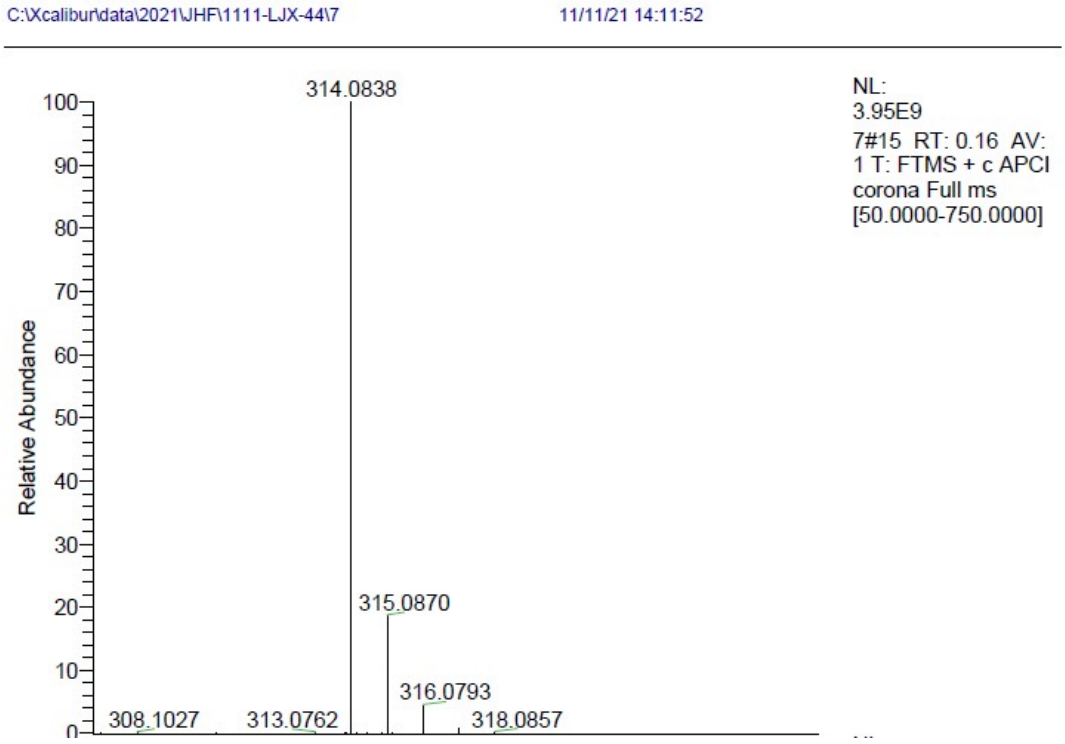
## The HR-MS spectra of 3e



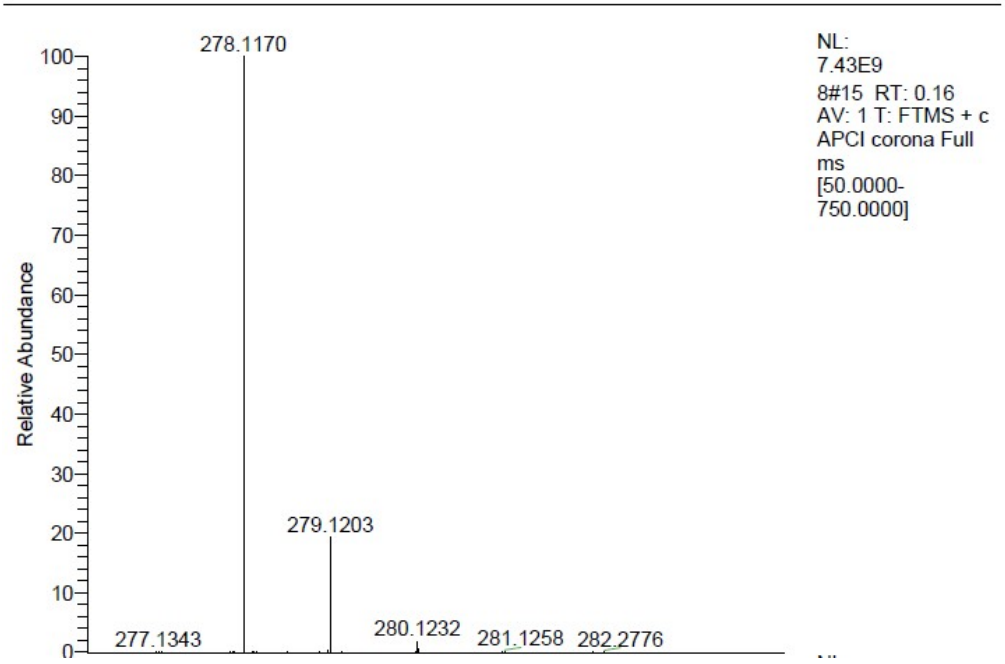

## The HR-MS spectra of 3f



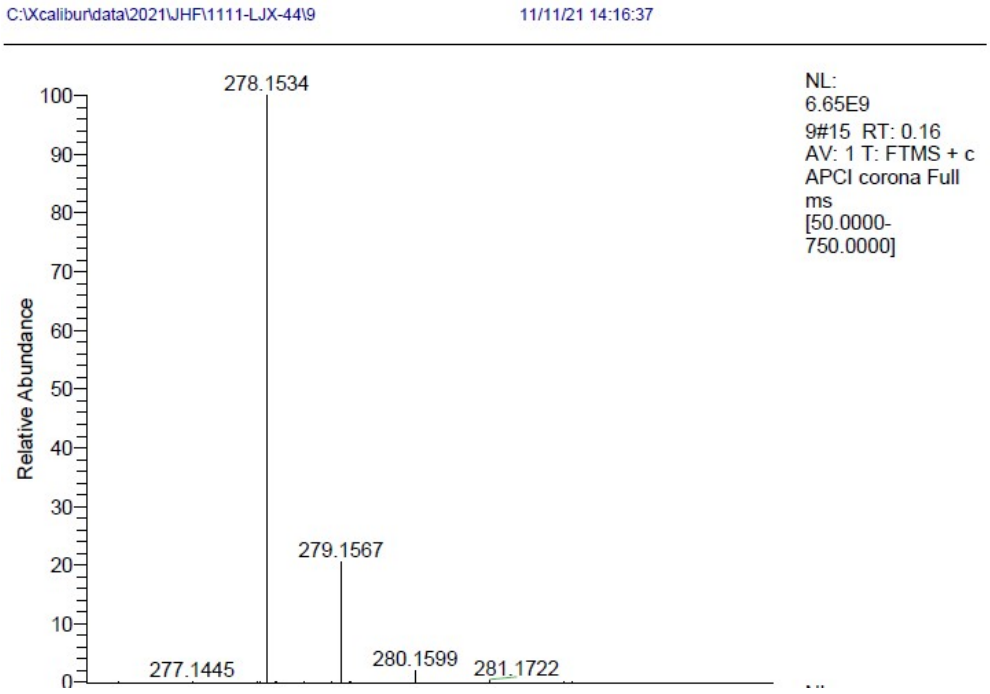
## The HR-MS spectra of 3g



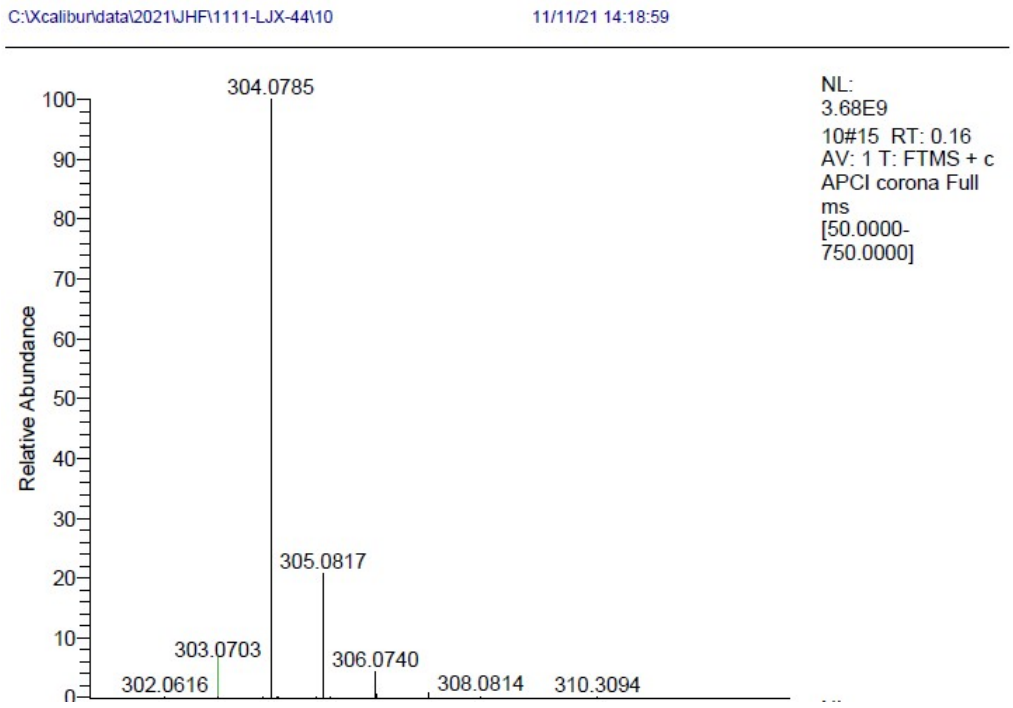

## The HR-MS spectra of 3h



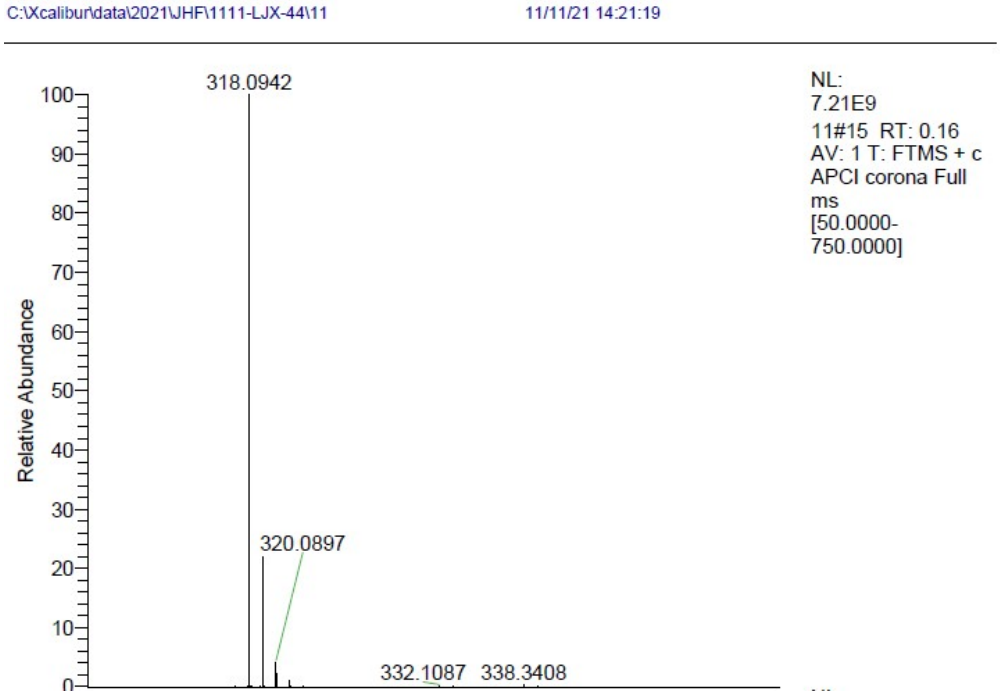
## The HR-MS spectra of 3i



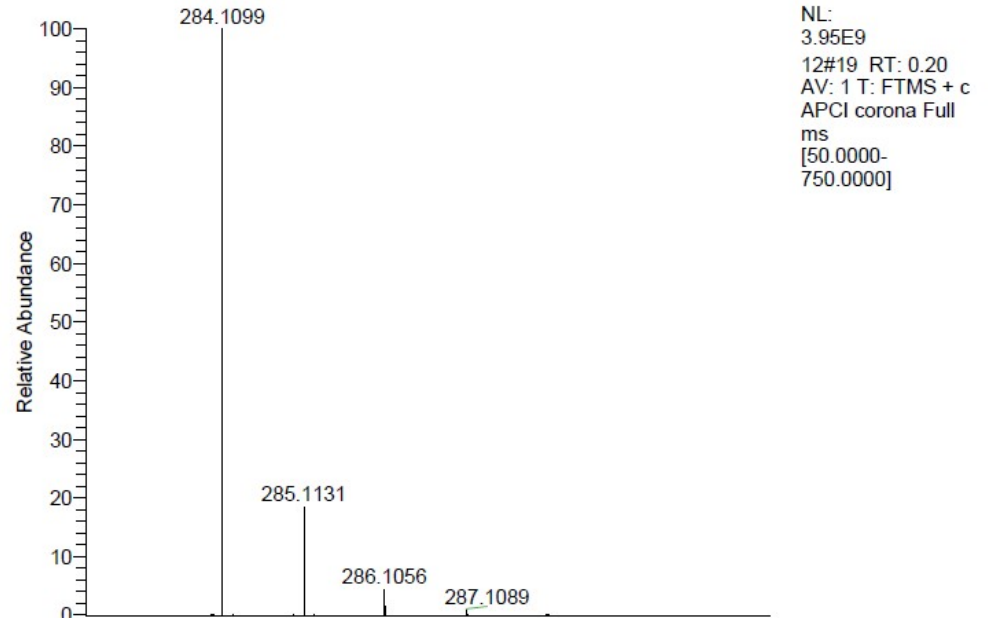

## The HR-MS spectra of 3j



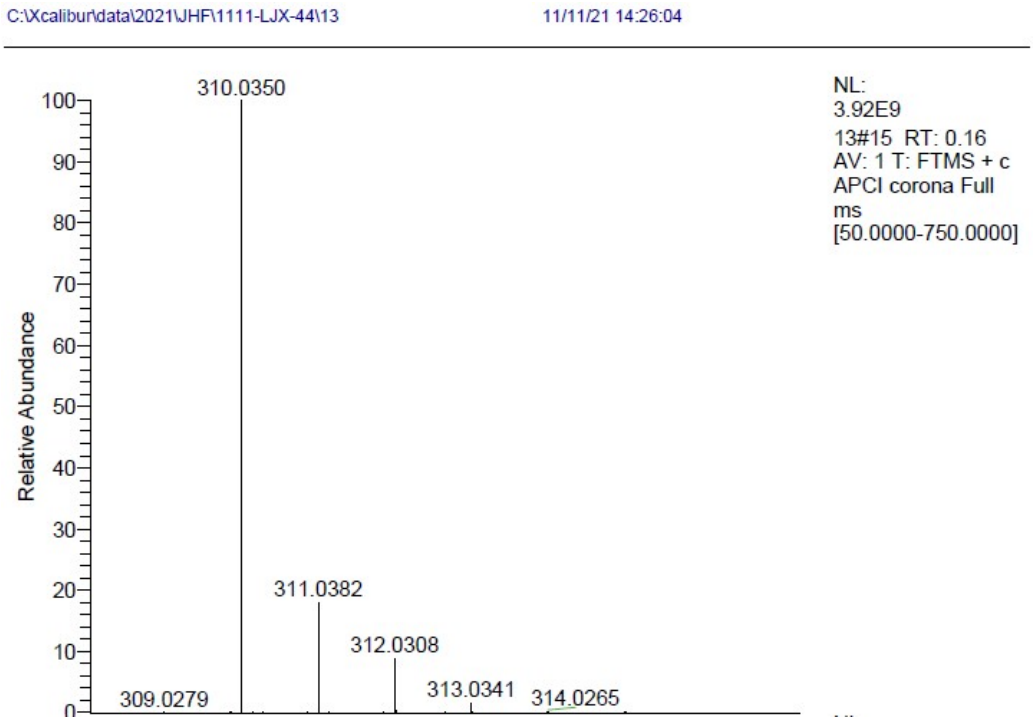
## The HR-MS spectra of 3k



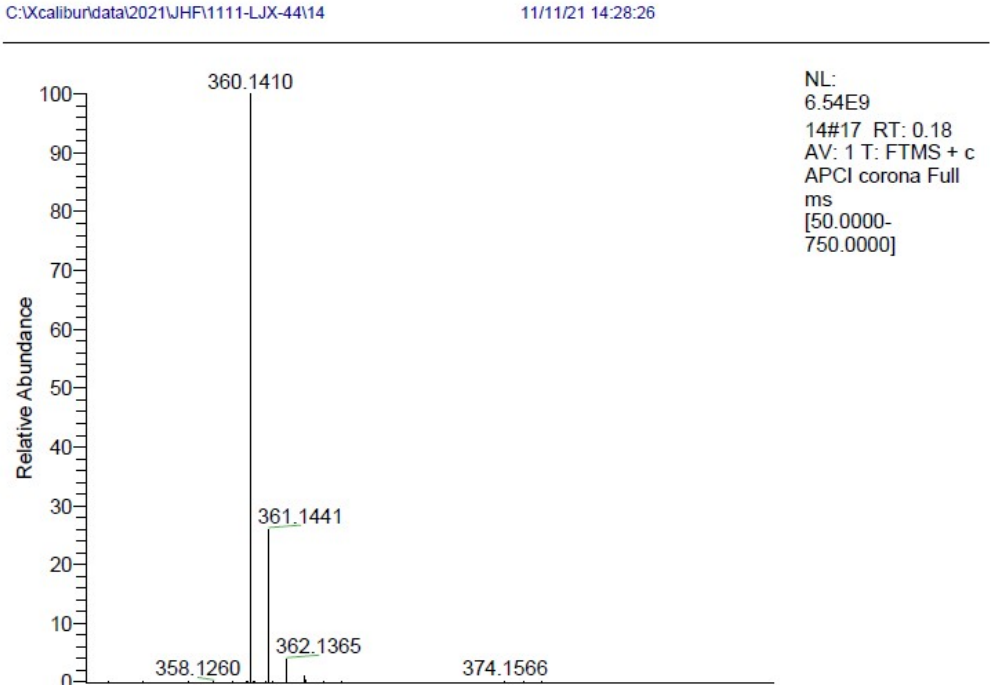

## The HR-MS spectra of 3l



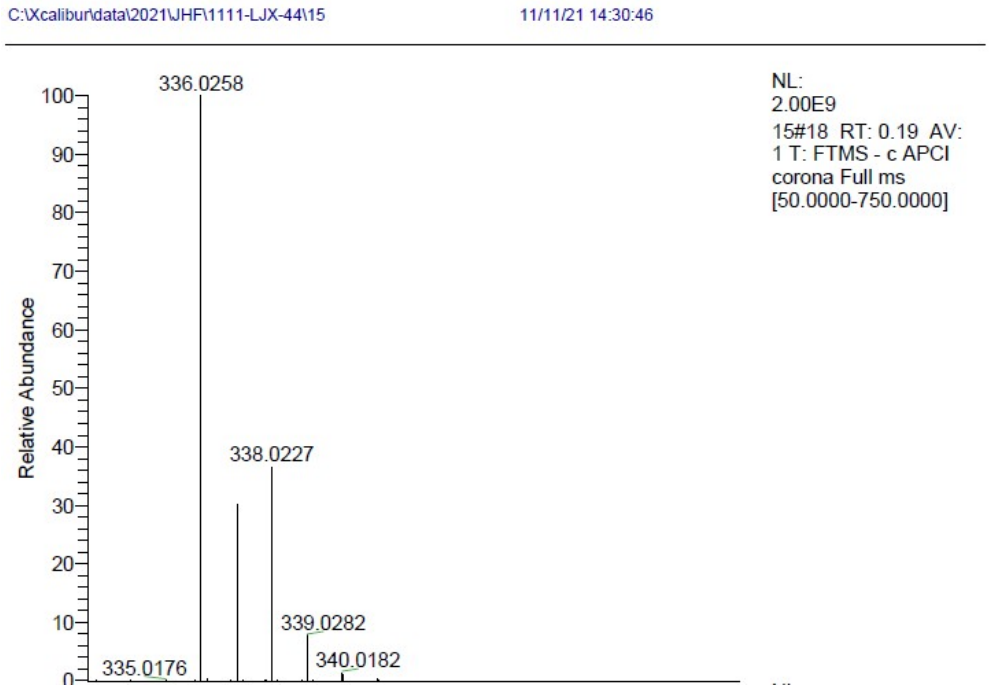
## The HR-MS spectra of 3m



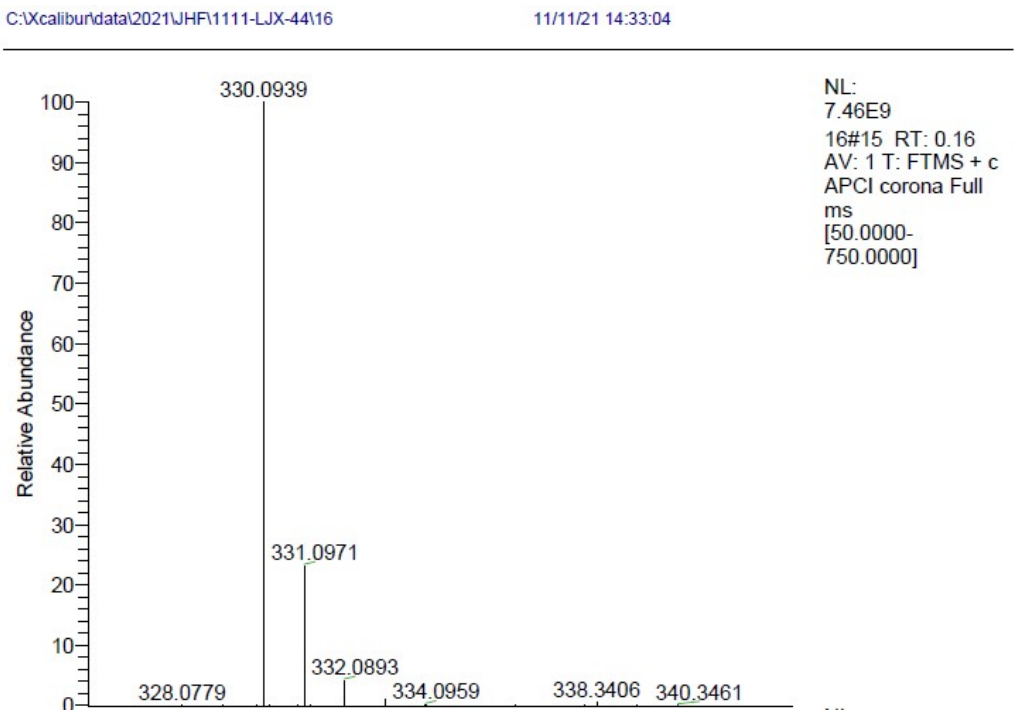

## The HR-MS spectra of 3n



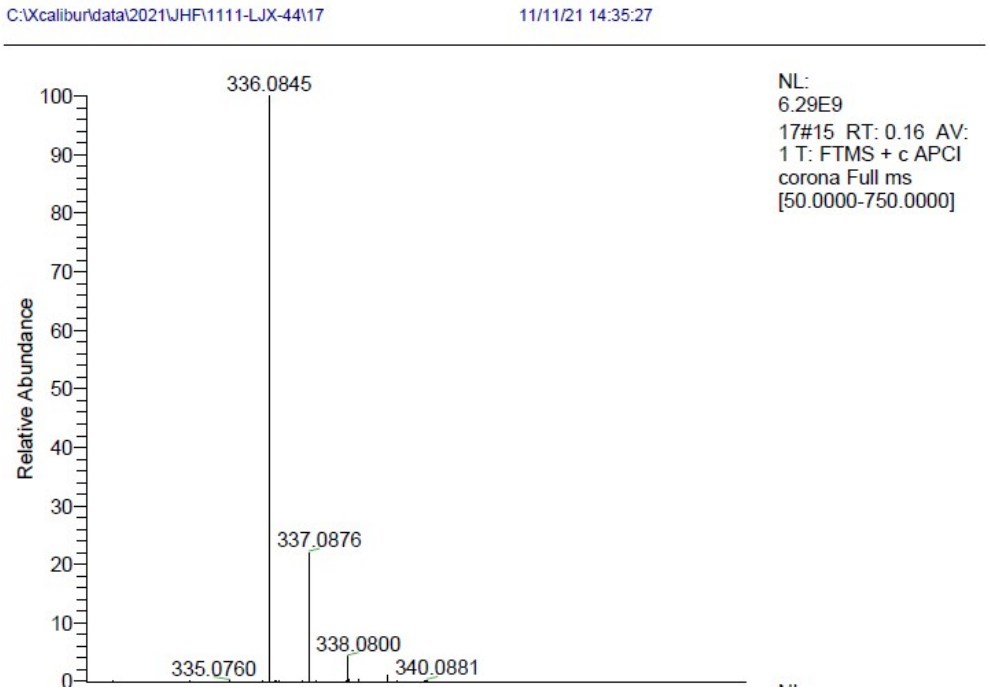
## The HR-MS spectra of 3o



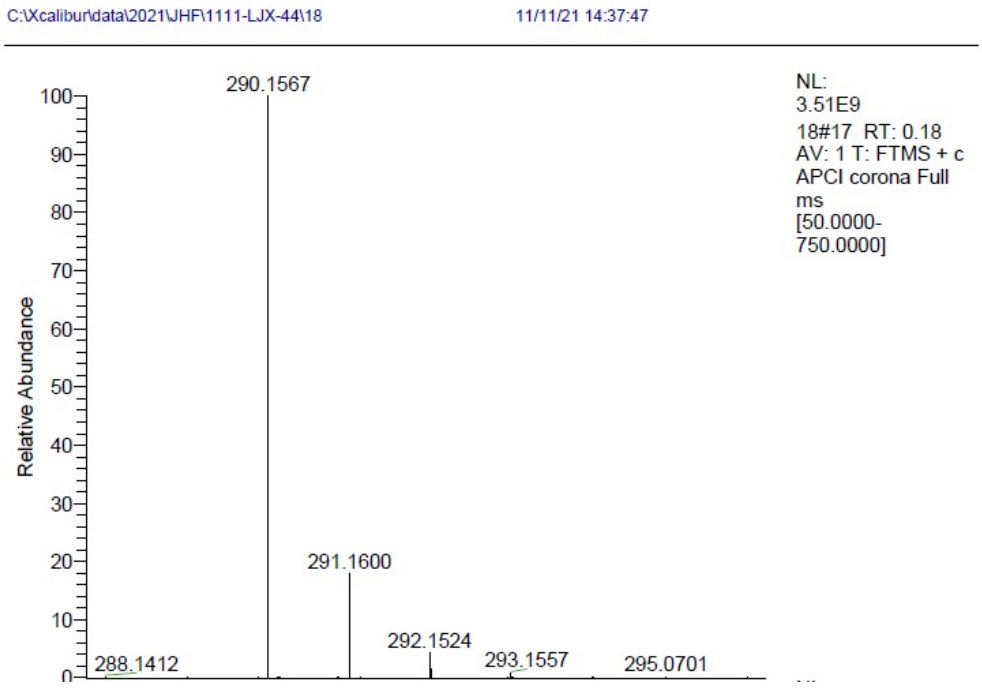

## The HR-MS spectra of 3p



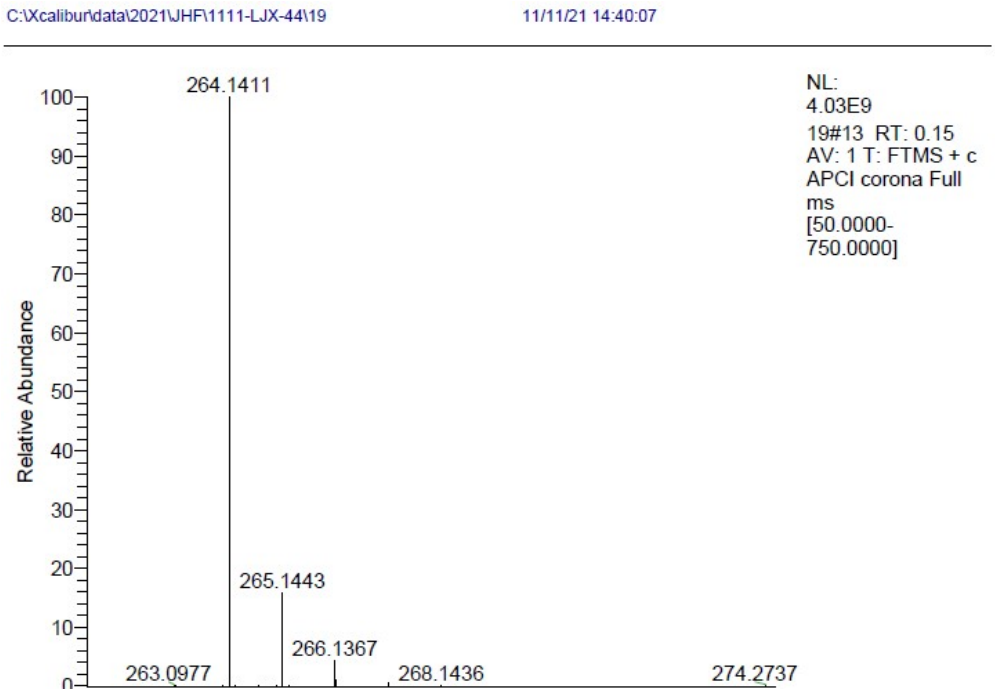
## The HR-MS spectra of 3q



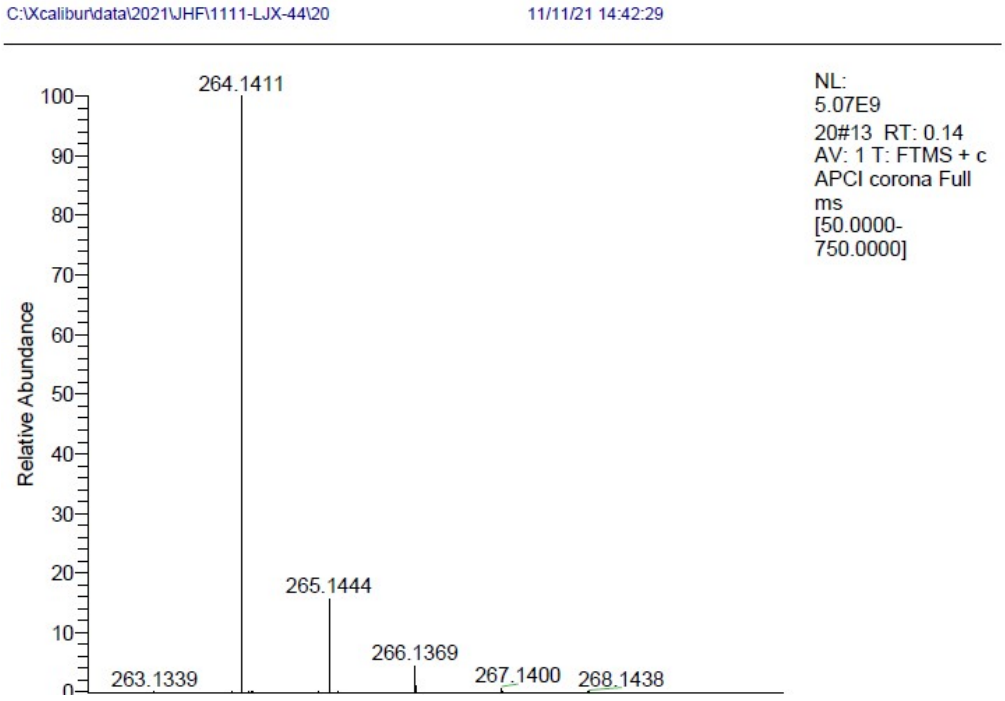

## The HR-MS spectra of 3r



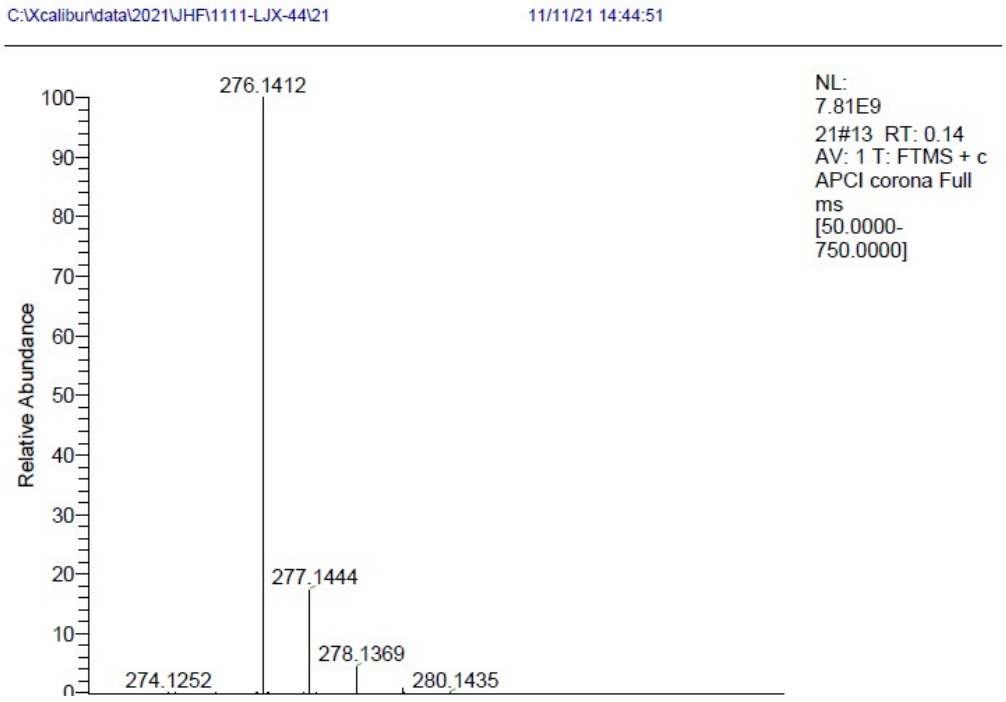
## The HR-MS spectra of 3s



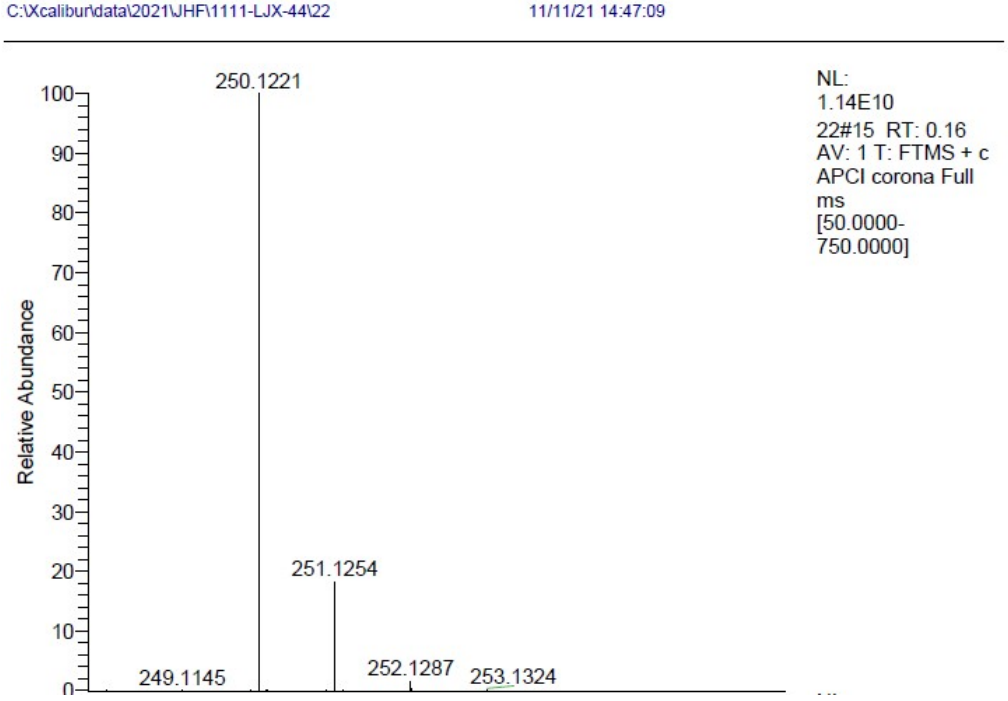

## The HR-MS spectra of 3t



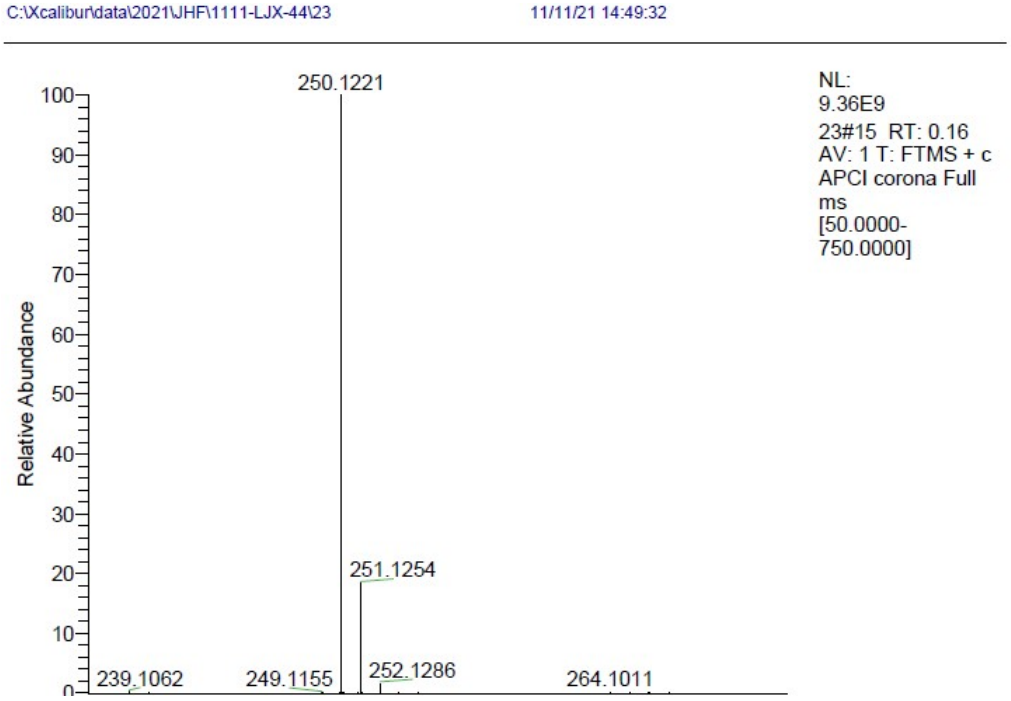
## The HR-MS spectra of 3u



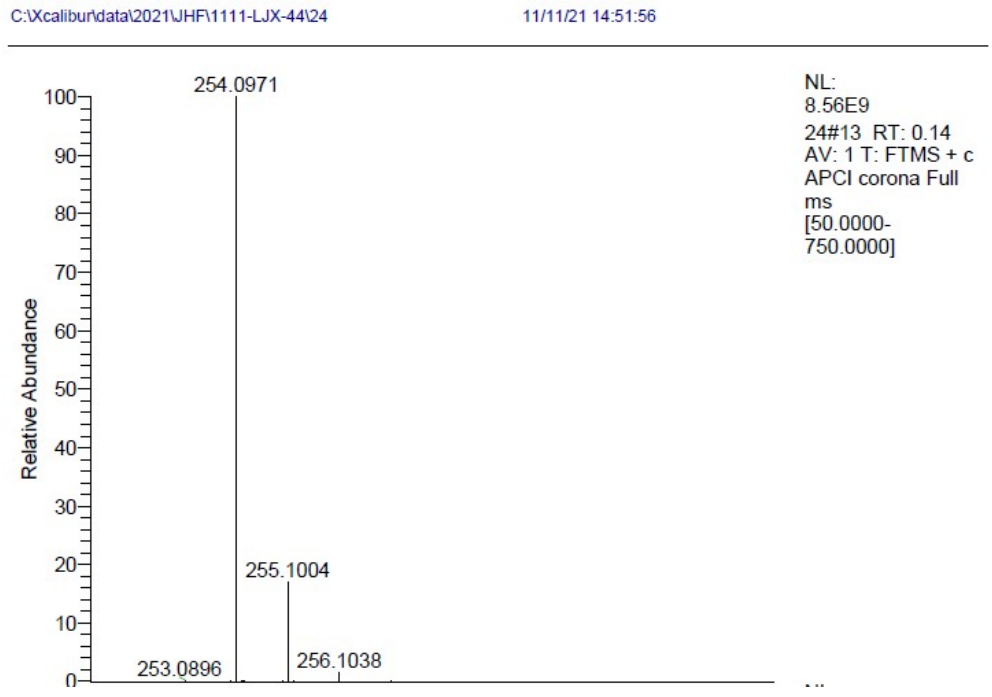

## The HR-MS spectra of 4a



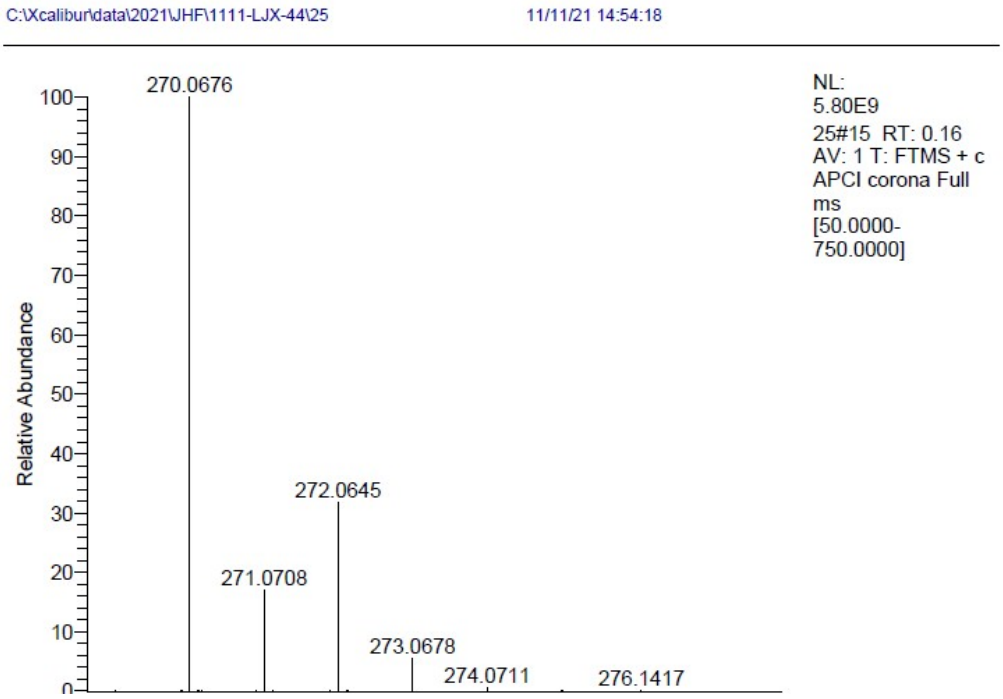
## The HR-MS spectra of 4b



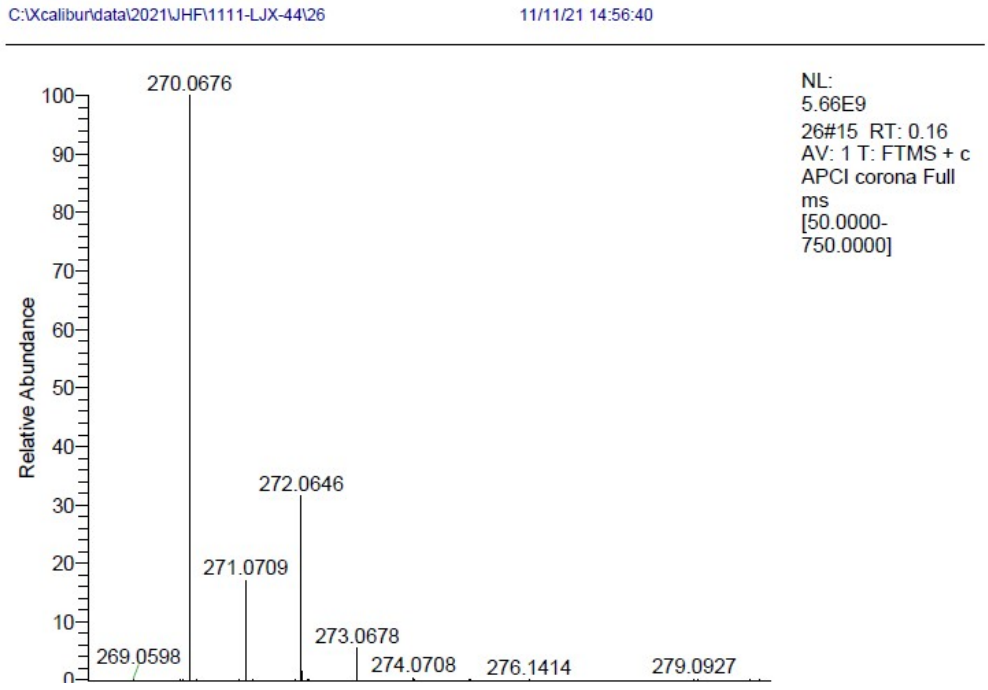

## The HR-MS spectra of 4c



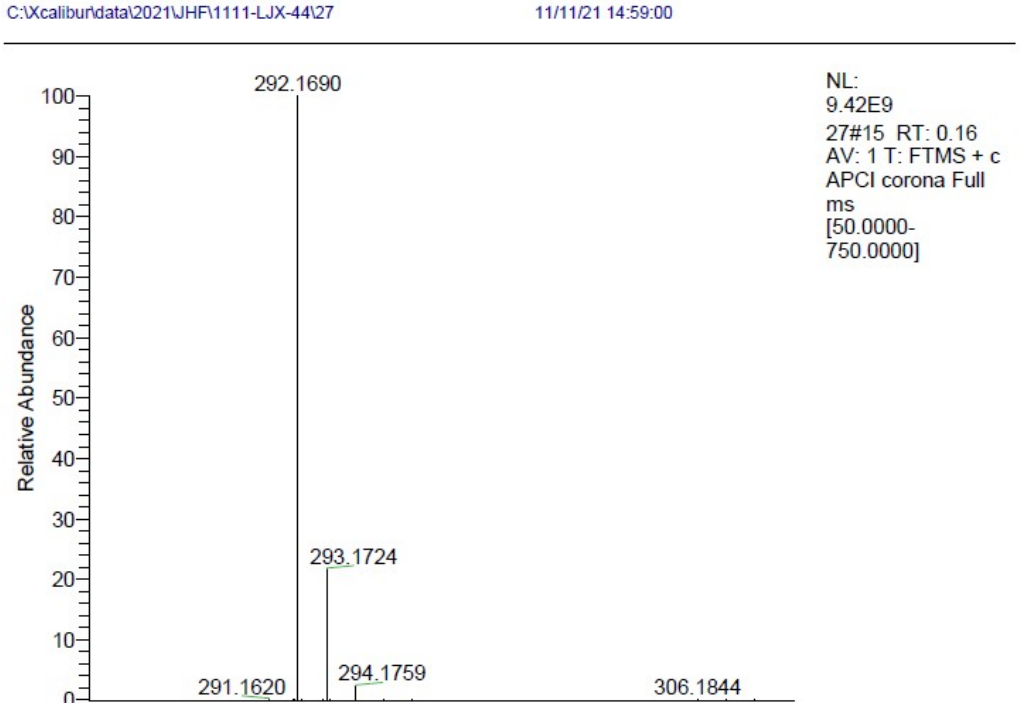
## The HR-MS spectra of 4d



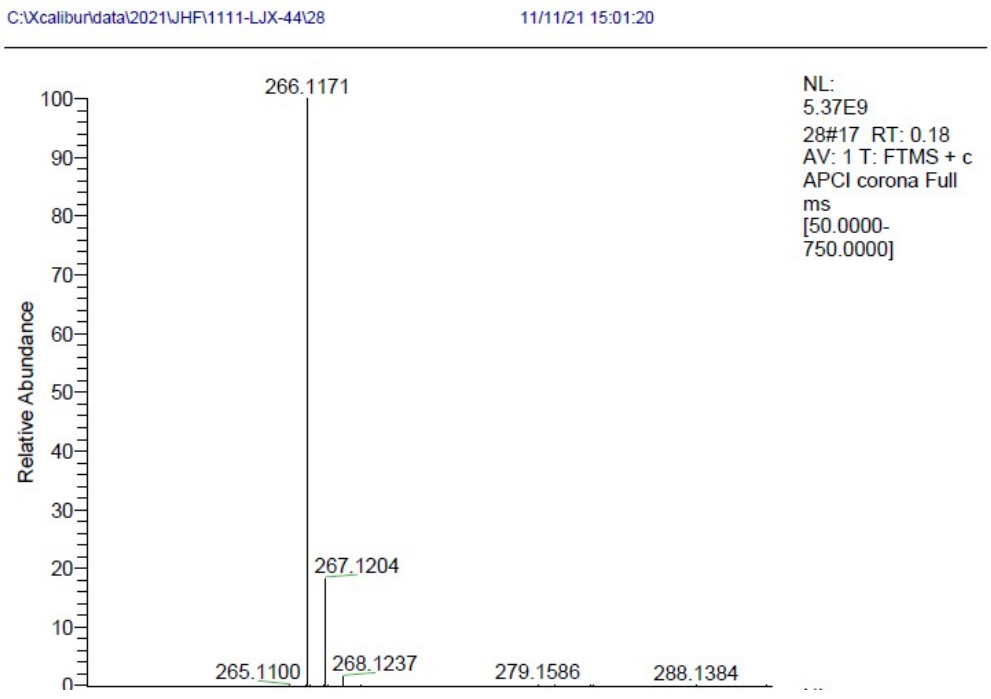

## The HR-MS spectra of 4e



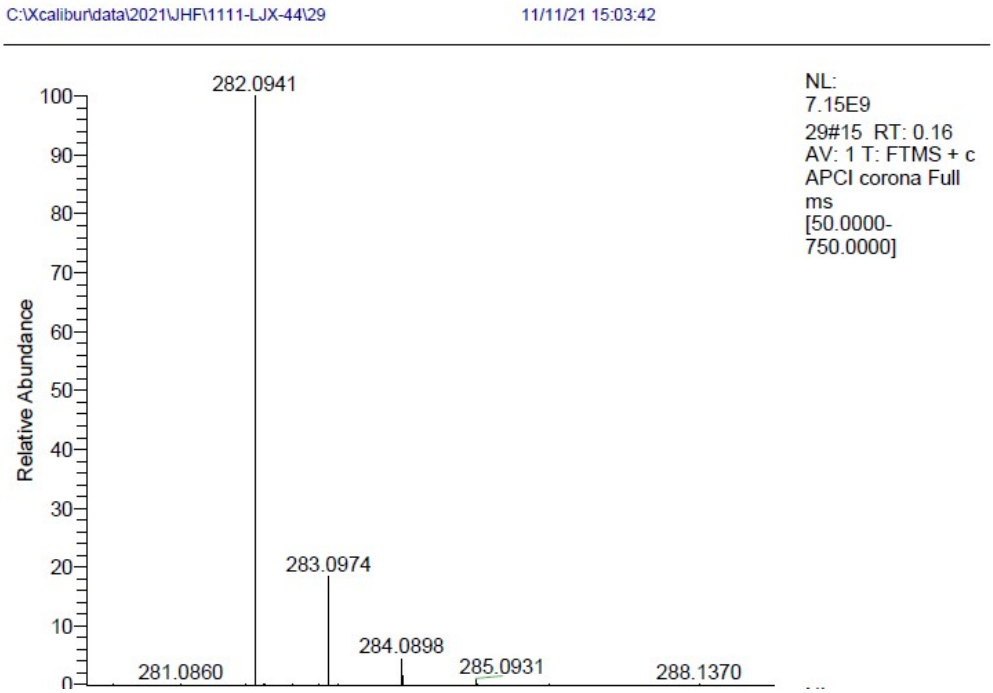
## The HR-MS spectra of 4f



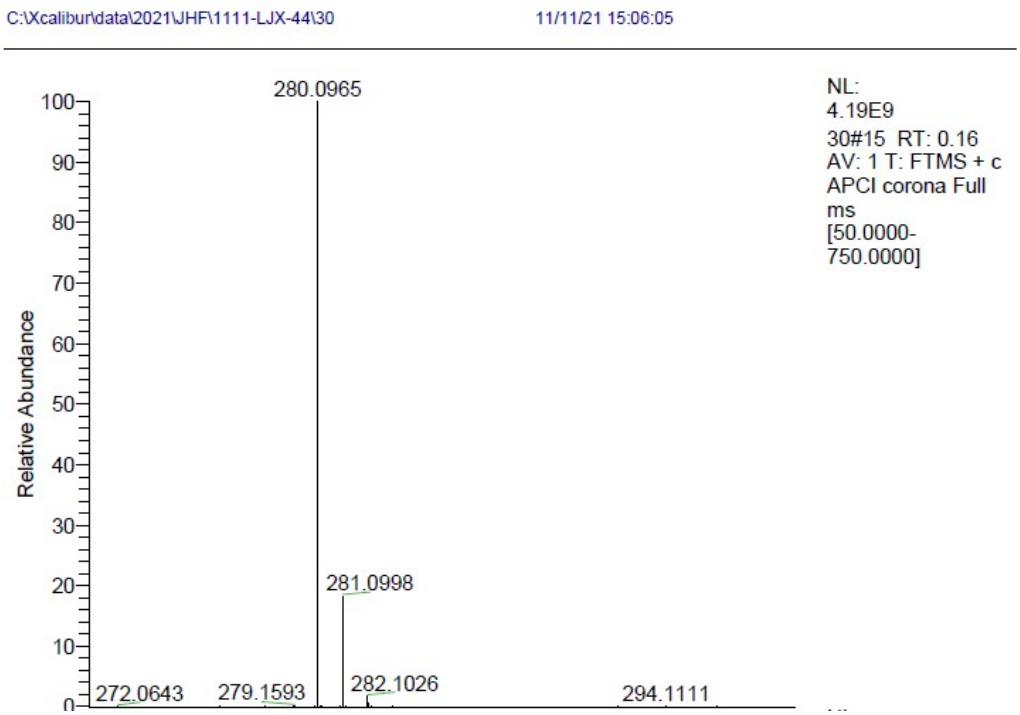

## The HR-MS spectra of 4g



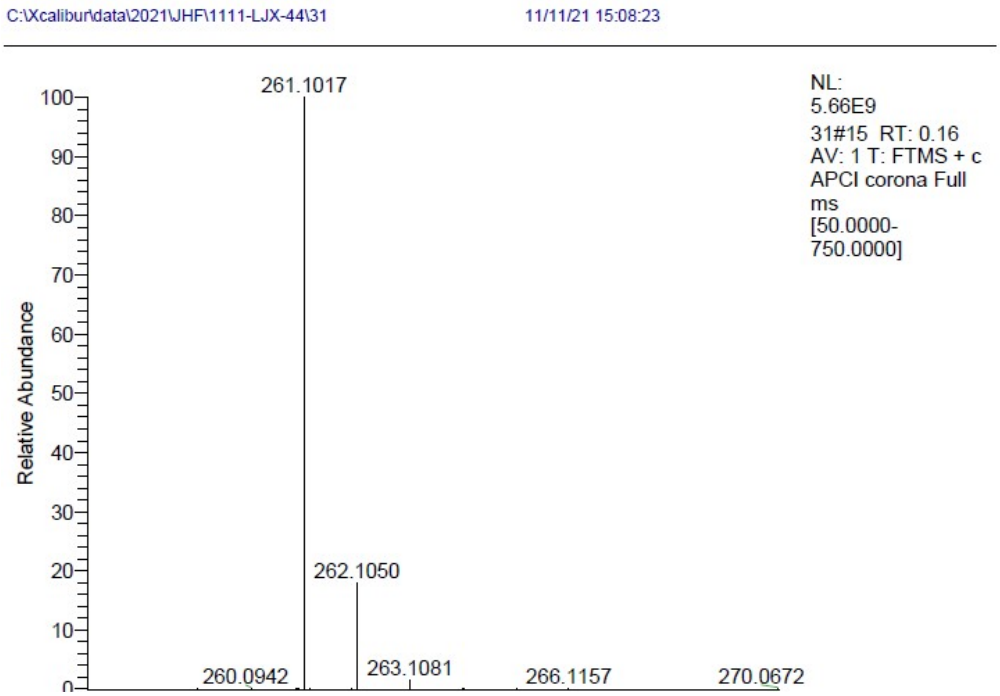
## The HR-MS spectra of 4h



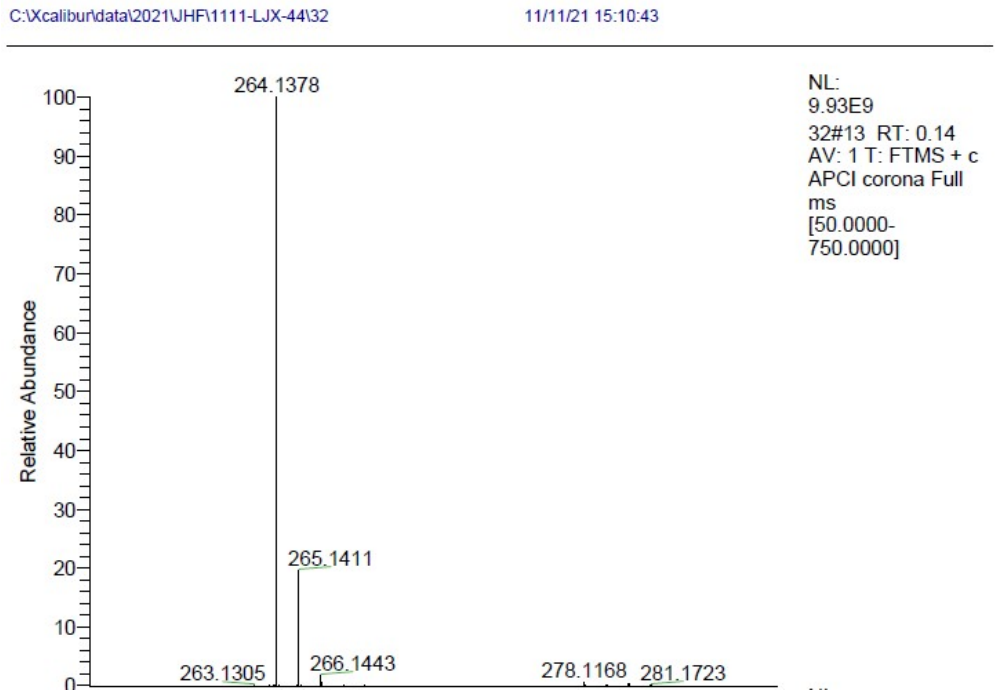

## The HR-MS spectra of 4i



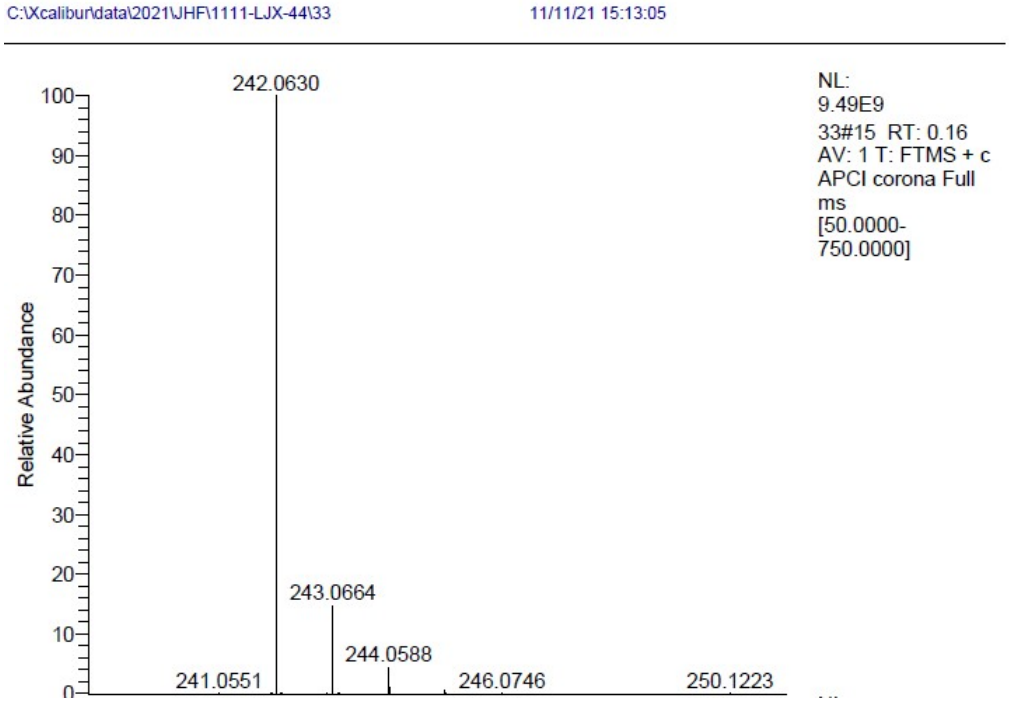
## The HR-MS spectra of 4j



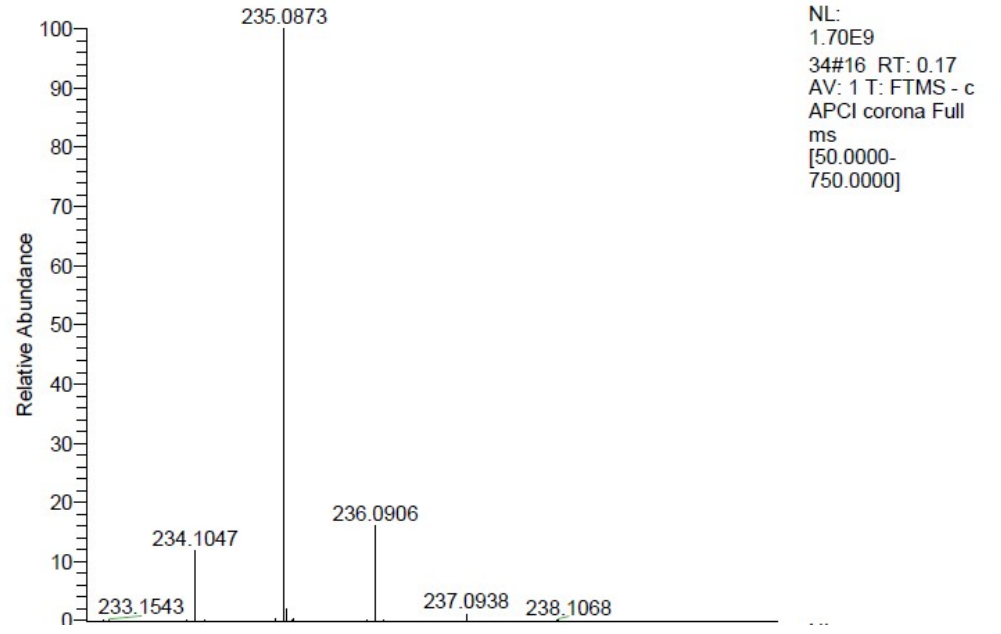

## The HR-MS spectra of 4k



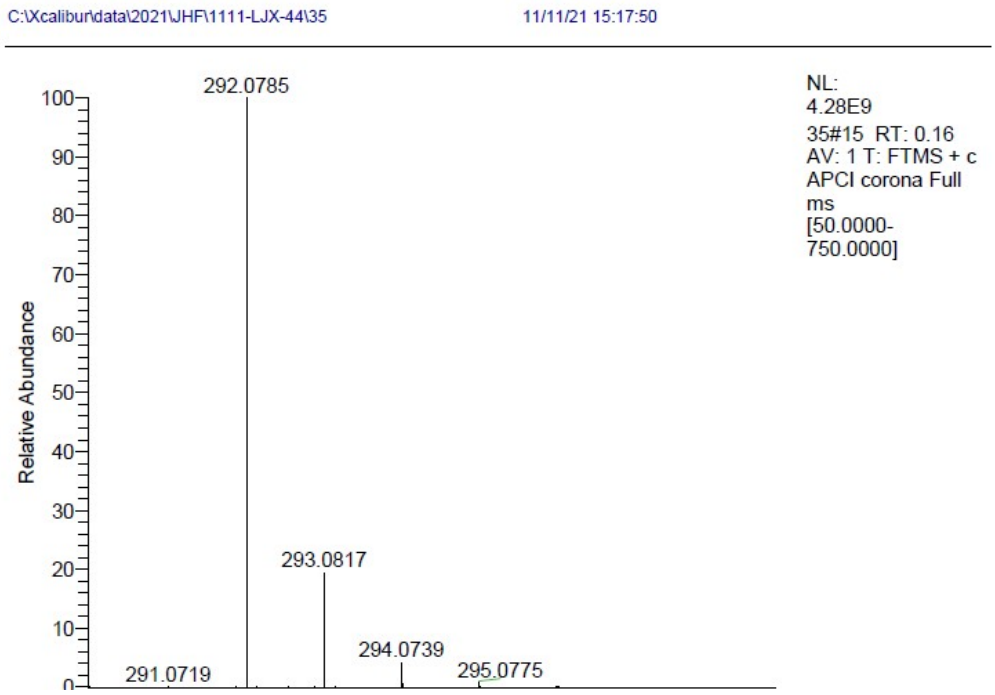
## The HR-MS spectra of 4l



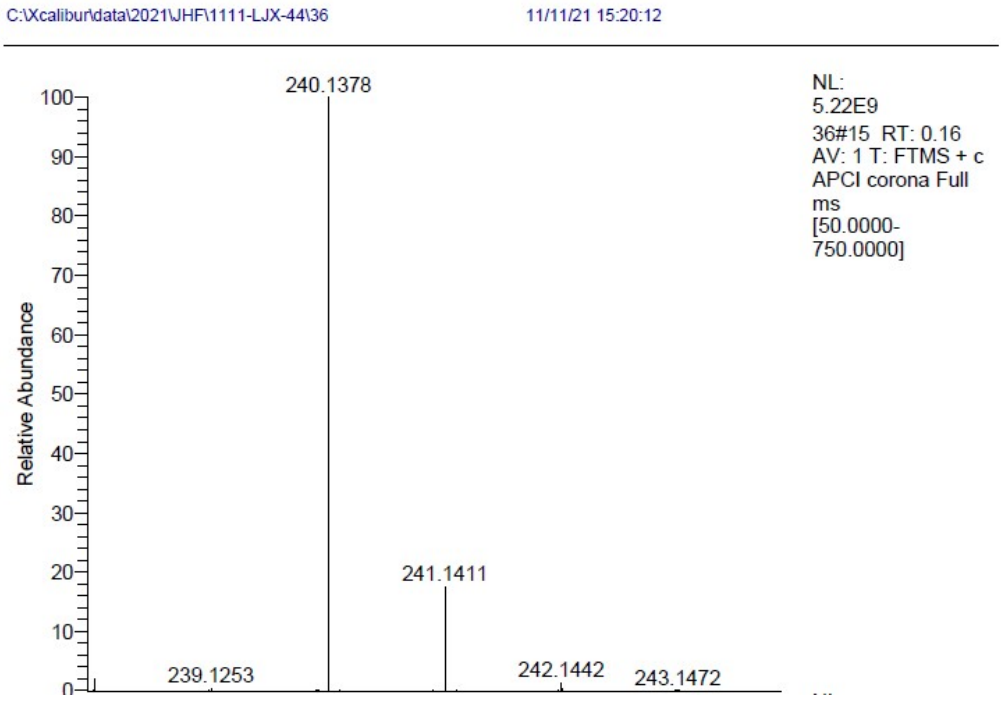

## The HR-MS spectra of 4m



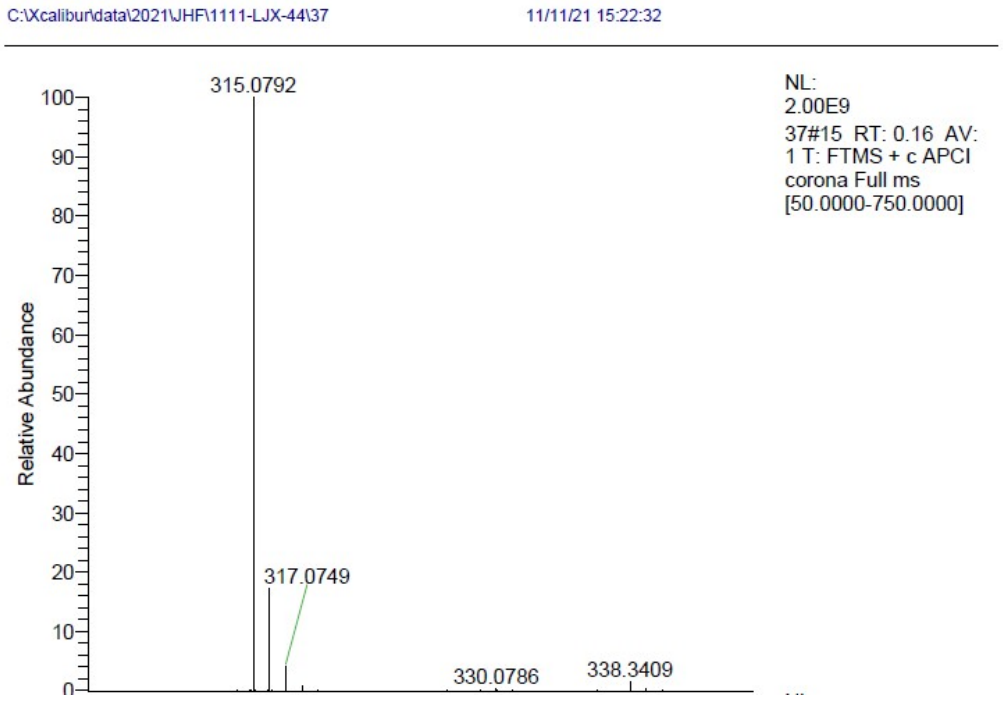
## The HR-MS spectra of 4n



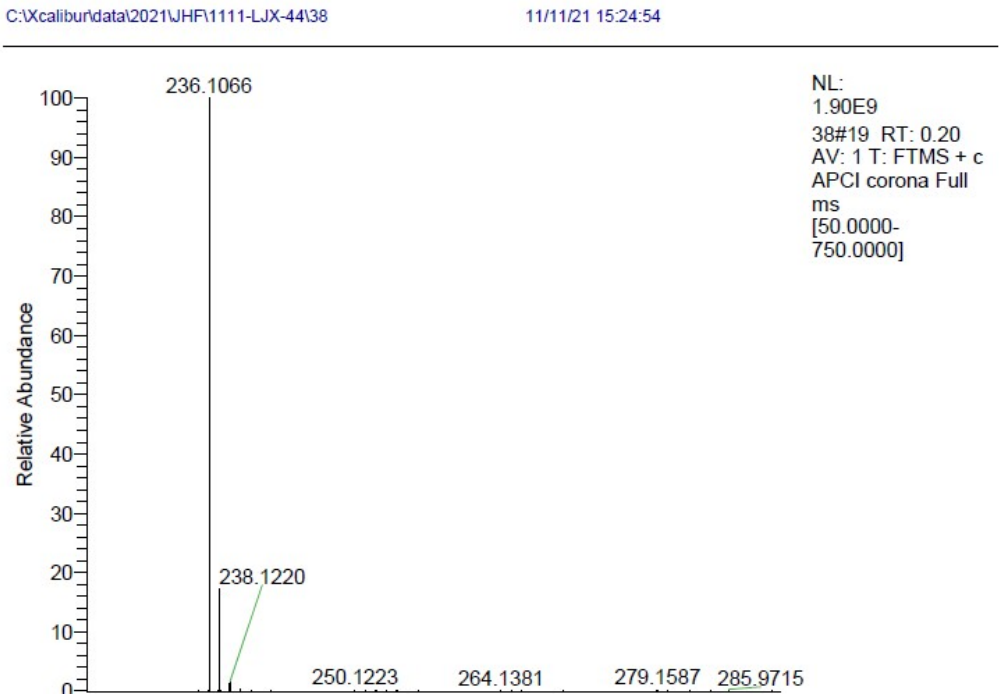

## The HR-MS spectra of 4o



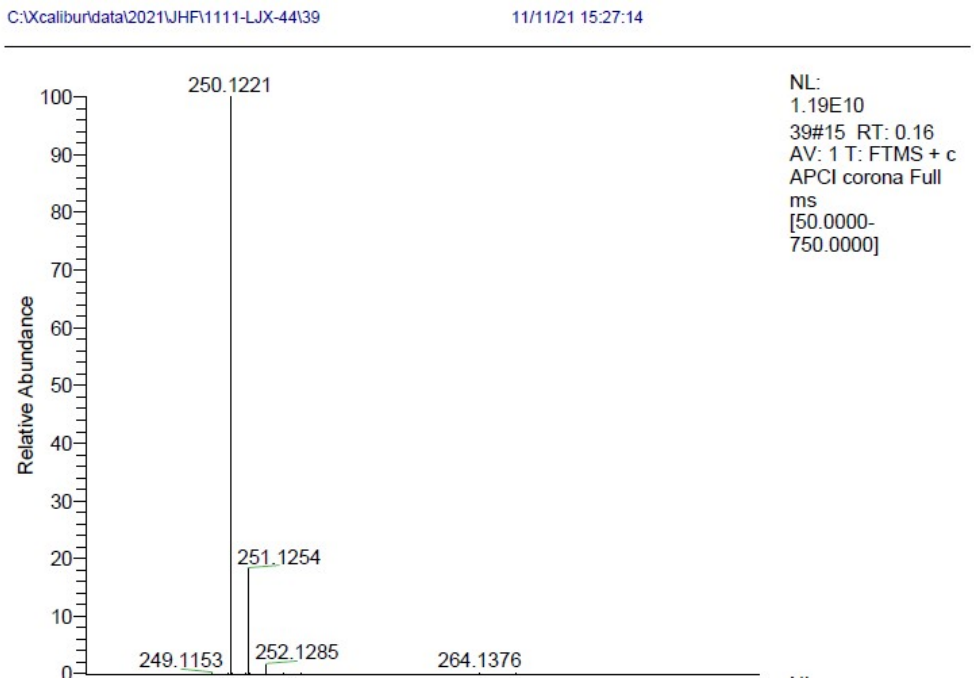
## The HR-MS spectra of 8



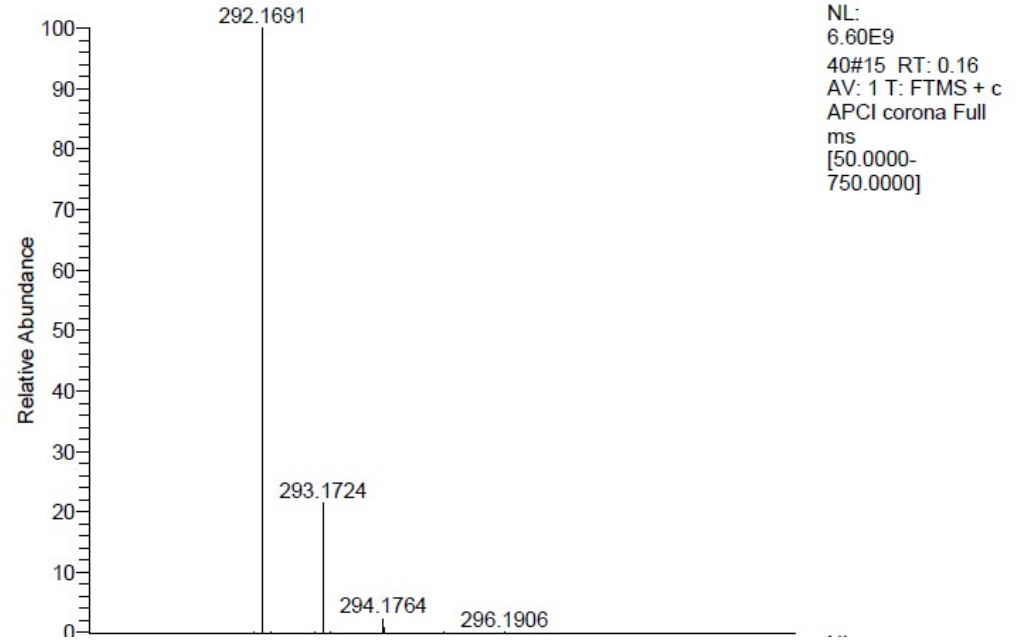

## The HR-MS spectra of 9a



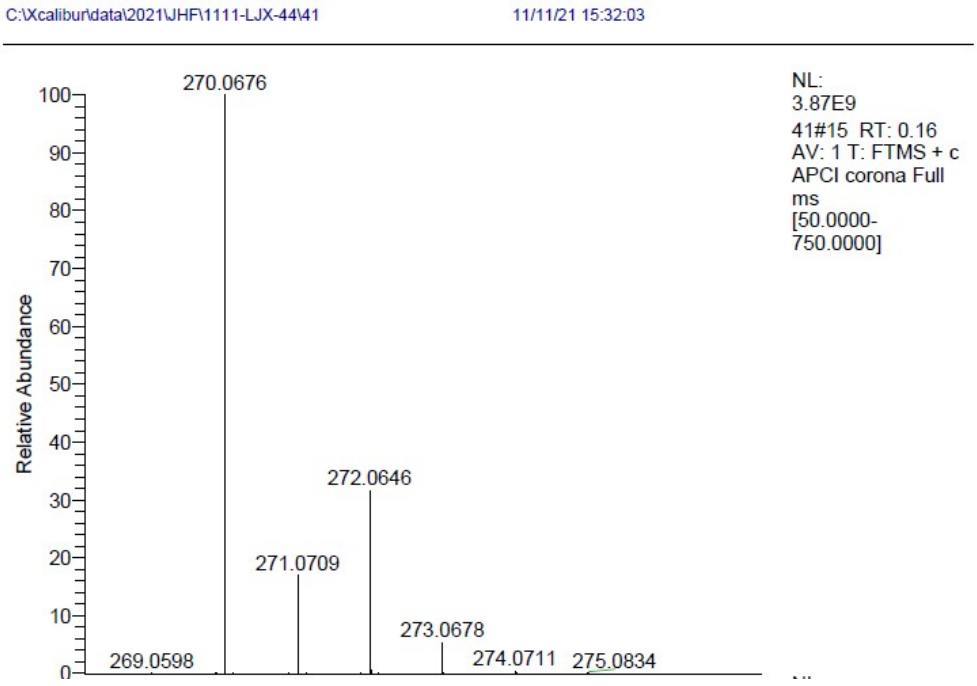
## The HR-MS spectra of 9b



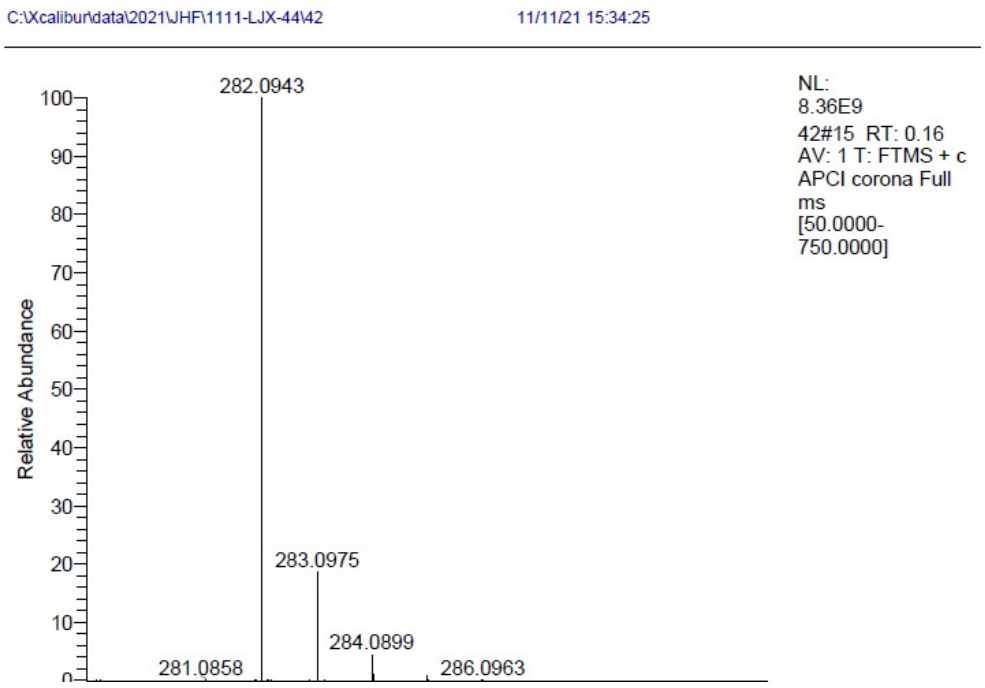

## The HR-MS spectra of 9c



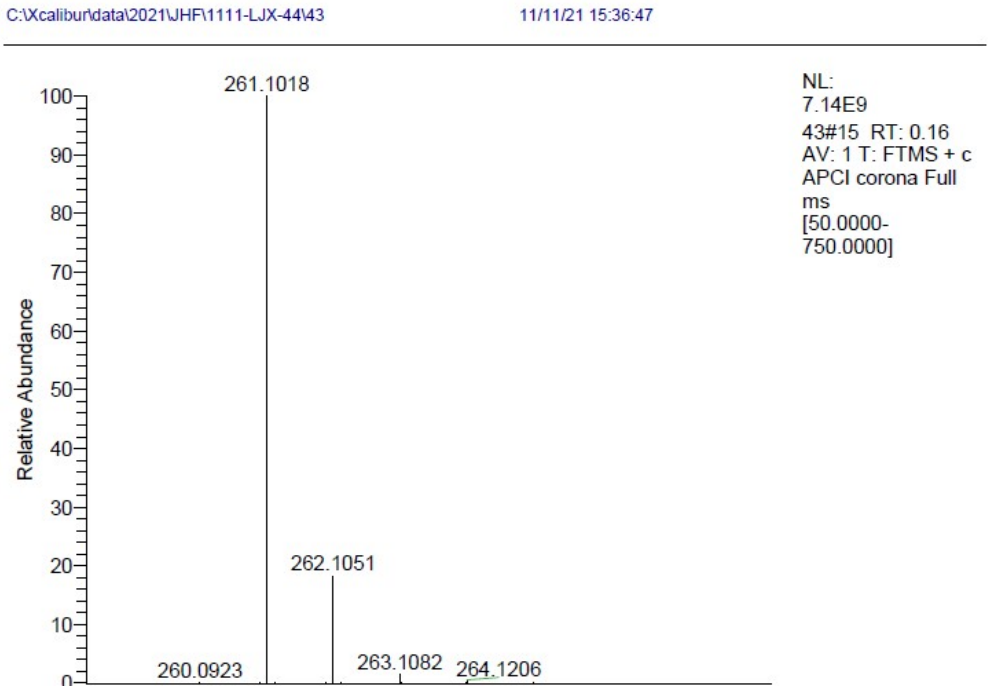
## The HR-MS spectra of 9d




## The HR-MS spectra of 9e



## The HR-MS spectra of 9f




## The HR-MS spectra of 9g