Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.


# Electrochemical One-pot Synthesis of Five-membered Azaheterocycles via [4+1] Cyclization

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## Table of Contents

General Information .....S1

Conditions Screening of Electrochemical One-pot Synthesis of 1,3,4-Oxadiazoles .....S1

Conditions Screening of Electrochemical One-pot Synthesis of 1,3,4-Oxadiazoles from 26 .....S5

Conditions Screening of Electrochemical One-pot Synthesis of 1,3,4-thiadiazoles .....S6

Procedure for the Synthesis of 3 - 23, 25, 34 - 42 .....S7

Procedure for the Synthesis of 5, 27- 33 from substituted toluenes .....S7

Procedure for the Synthesis of 1,3,4-thiadiazoles 44-49 .....S8

Procedure for the Synthesis of imidazoles 51-56 .....S8

Procedure for the Synthesis of oxazolines 58-63 .....S8

Procedure for the Synthesis of oxazolines (*S*)-63 .....S9

Gram scale synthesis of 3 .....S10

Mechanism study .....S10

Characterization data for the electrolysis products .....S13

Reference .....S28

$^{1}H$, $^{13}C$ and $^{19}F$ Spectra of all products .....S30

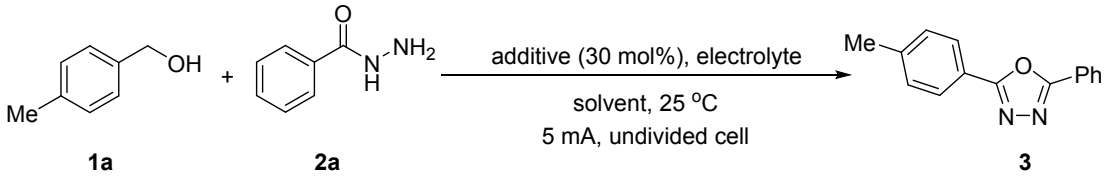
## General Information

All reactions were carried out under atmospheric conditions unless otherwise stated. Reagents and solvents were purchased commercially and used directly as received. $^{1}H$, $^{13}C$ and $^{19}F$ NMR spectra were recorded on a Bruker Advance 600 and Bruker Ultrashiled 500 instrument. Multiplicities were reported by use of the following abbreviations: s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, m = multiplet, br = broad. All chemical shifts are reported relative to tetramethylsilane and d-solvent peaks (77.16 ppm, chloroform; 39.60 ppm, dimethyl sulfoxide), respectively. High–resolution mass spectra (HRMS) were recorded on an Agilent LC-MS TOF mass spectrometer by electrospray positive ionization time-of-flight (ESI-TOF) reflectron experiments.

## Conditions Screening of Electrochemical One-pot Synthesis of 1,3,4-Oxadiazoles

In a simple undivided cell, *p*-tolylmethanol **1a** and benzohydrazide **2a** were added simultaneously with $LiClO_4$ or $Et_4NBr$ as the electrolyte. The electrolysis was carried out under 5 mA constant current for 10 h, but no desired product can be obtained (Table S1, entries 1,2). The simultaneous addition of the components might be detrimental since the decomposition of **2a** may occur prior to its condensation with the aldehyde, which was generated *in situ* from **1a** via anodic oxidation and its concentration may not be high enough to match condensation with **2a** during the process. Consequently, the benzohydrazide **2a** was added in one portion after the full conversion of *p*-tolylmethanol **1a** into *p*-tolualdehyde under the same conditions. To our delight, the proposed reaction can be fulfilled albeit in poor yield (Table S1, entries 3,4). Subsequently, redox mediator NHPI (*N*-hydroxyphthalimide) showed a superior activity than TEMPO and provided the desired product **3** in promising yields (entries 5, 6). An extensive evaluation of solvent revealed that the combination of $CH_3CN$ with TFE in the ratio of 2:1 was preferable (Table S1, entries 7-9). Additionally, $Et_4NBr$ (0.03 M) in combination with $Et_4NOTs$ (0.1 M) as electrolytes offered the best result (Table S1, entries 10-19). Amongst a set of representative electrodes, platinum showed the best efficiency (Table S1, entries 20-23). Moreover, similar reaction efficacy can be obtained under $N_2$ atmosphere, which indicate that oxygen may not act as the terminal oxidant (Table S1, entries 24).[1] Notably, no corresponding product was delivered without electricity, strongly suggesting its pivotal role in this transformation (Table S1, entry 25).

**Table S1.** Optimization of the conditions for the electrochemical cascade reaction[a]




**1a** **2a** **3**

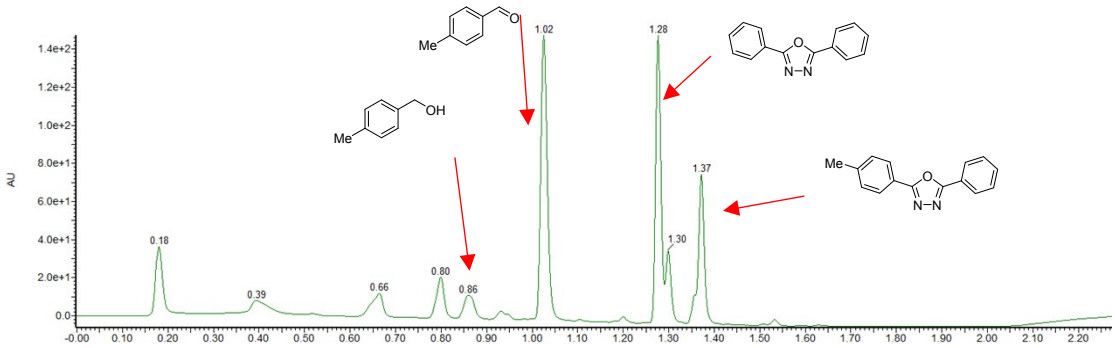
**Table S1.** Optimization of the conditions for the electrochemical cascade reaction[a]

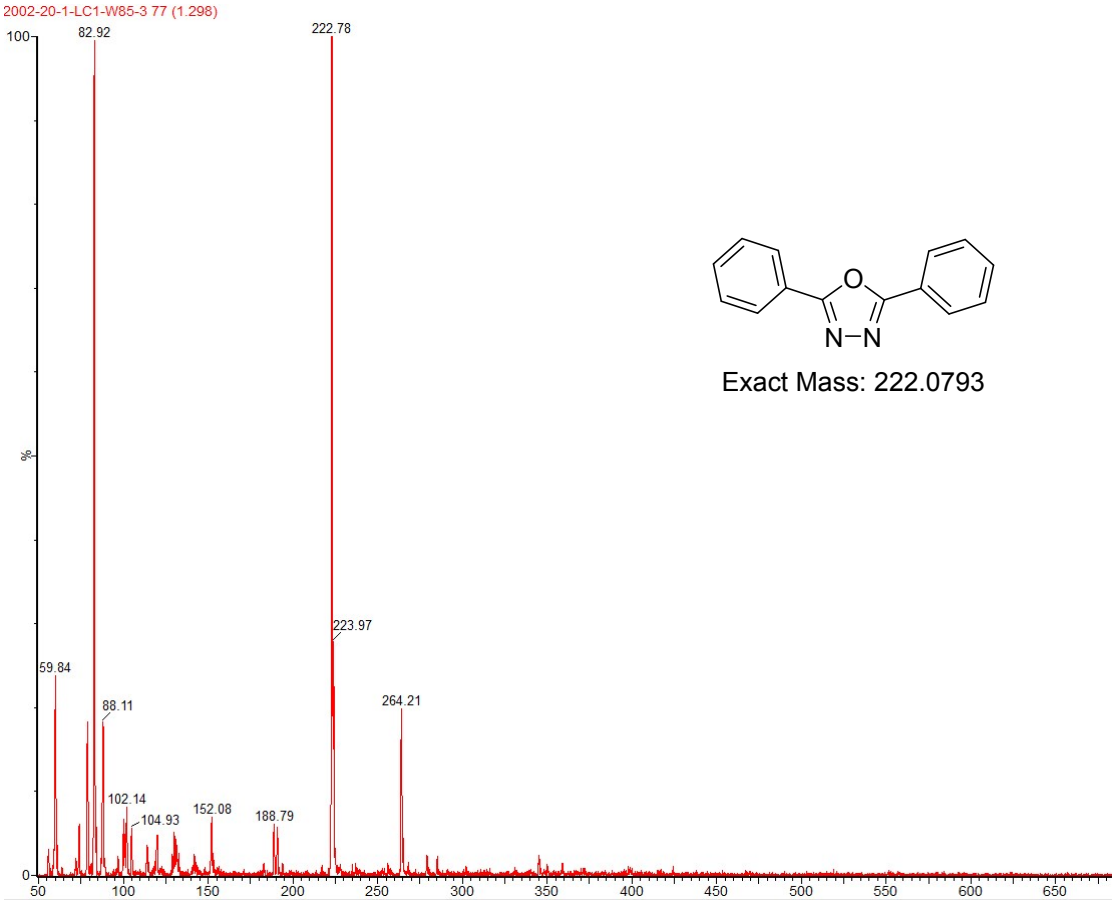
| Entry | Additive | Electrolyte | Anode/cathode | Solvent | Yield (%)[b] **3** |
|---|---|---|---|---|---|
| 1[c] | - | $Et_4NBr$ | Pt/Pt | $CH_3CN$ | n.r. |
| 2[c] | - | $LiClO_4$ | Pt/Pt | $CH_3CN$ | n.r. |
| 3 | - | $Et_4NBr$ | Pt/Pt | $CH_3CN$ | 35 |
| 4 | - | $LiClO_4$ | Pt/Pt | $CH_3CN$ | 23 |
| 5 | NHPI | $Et_4NBr$ | Pt/Pt | $CH_3CN$ | 51 |
| 6 | TEMPO | $Et_4NBr$ | Pt/Pt | $CH_3CN$ | 27 |
| 7 | NHPI | $Et_4NBr$ | Pt/Pt | TFE | 41 |
| 8 | NHPI | $Et_4NBr$ | Pt/Pt | MeOH | n.r. |
| 9 | NHPI | $Et_4NBr$ | Pt/Pt | $CH_3CN$/ TFE (2:1) | 69 |
| 10 | NHPI | $Et_4NClO_4$ | Pt/Pt | $CH_3CN$/ TFE (2:1) | 55 |
| 11 | NHPI | $Et_4NBF_4$ | Pt/Pt | $CH_3CN$/ TFE (2:1) | 40 |
| 12 | NHPI | $Et_4NOTs$ | Pt/Pt | $CH_3CN$/ TFE (2:1) | 26 |
| 13 | NHPI | $Et_4NI$ | Pt/Pt | $CH_3CN$/ TFE (2:1) | 12 |
| 14 | NHPI | $LiClO_4$ | Pt/Pt | $CH_3CN$/ TFE (2:1) | 31 |
| 15 | NHPI | $Et_4NBr$ (0.1 M)/ $Et_4NOTs$ (0.1 M) | Pt/Pt | $CH_3CN$/ TFE (2:1) | 73 |
| 16 | NHPI | $Et_4NBr$ (0.05 M)/ $Et_4NOTs$ (0.1 M) | Pt/Pt | $CH_3CN$/ TFE (2:1) | 77 |
| 17 | **NHPI** | **$Et_4NBr$ (0.03 M)/ $Et_4NOTs$ (0.1 M)** | **Pt/Pt** | **$CH_3CN$/ TFE (2:1)** | **86** |
| 18[c] | NHPI | $Et_4NBr$ (0.03 M)/ $Et_4NOTs$ | Pt/Pt | $CH_3CN$/ TFE (2:1) | 32 |

| | | | | | |
|---|---|---|---|---|---|
| | | (0.1 M) | | | |
| 19 | NHPI | $Et_4NBr$ (0.01 M)/ $Et_4NOTs$ (0.1 M) | Pt/Pt | $CH_3CN$/ TFE (2:1) | 59 |
| 20 | NHPI | $Et_4NBr$ (0.03 M)/ $Et_4NOTs$ (0.1 M) | C/ C | $CH_3CN$/ TFE (2:1) | 77 |
| 21 | NHPI | $Et_4NBr$ (0.03 M)/ $Et_4NOTs$ (0.1 M) | C/ Ni | $CH_3CN$/ TFE (2:1) | 63 |
| 22 | NHPI | $Et_4NBr$ (0.03 M)/ $Et_4NOTs$ (0.1 M) | Pt/ Ni | $CH_3CN$/ TFE (2:1) | 79 |
| 23 | NHPI | $Et_4NBr$ (0.03 M)/ $Et_4NOTs$ (0.1 M) | Pt/ Fe | $CH_3CN$/ TFE (2:1) | 61 |
| 24[d] | NHPI | $Et_4NBr$ (0.03 M)/ $Et_4NOTs$ (0.1 M) | Pt/Pt | $CH_3CN$/ TFE (2:1) | 83 |
| 25[e] | NHPI | $Et_4NBr$ (0.03 M)/ $Et_4NOTs$ (0.1 M) | Pt/Pt | $CH_3CN$/ TFE (2:1) | n.r. |

[a] Reaction conditions: under atmospheric conditions at 25 °C, *p*-tolylmethanol **1a** (0.2 mmol), additive (0.06 mmol, 30 mol%), electrolyte with solvent (3 mL) at a constant current of 5 mA for 3 h, then benzohydrazide **2a** (0.26 mmol) was added, the system was electrolyzed at a constant current of 5 mA for additional 7 h. NHPI = *N*-hydroxyphthalimide. TEMPO = 2,2,6,6-Tetramethylpiperidinooxy [b] Isolated yield. [c]**1a** and **2a** were added simultaneously. [d]Under $N_2$ atmosphere. [e]Without electricity.

**Figure S1. UPLC-MS spectra of the reaction mixture which 1a and 2a were added simultaneously at the beginning of reaction**

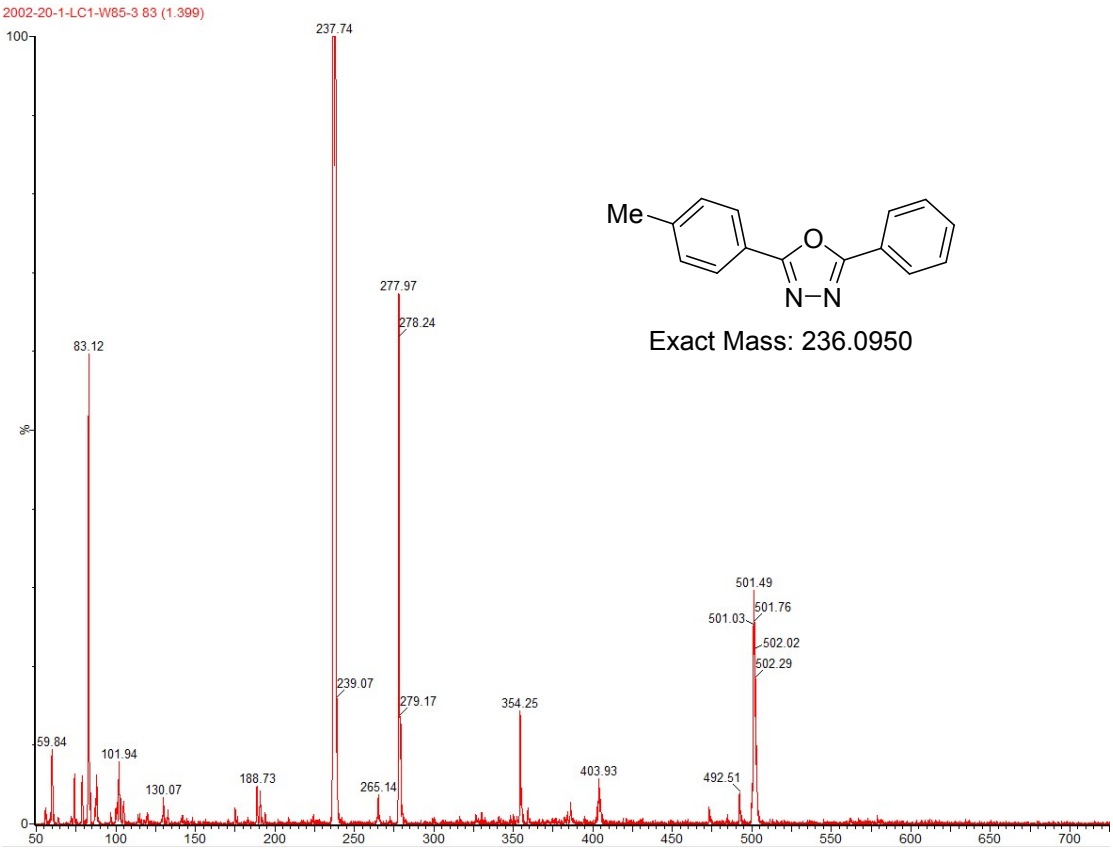




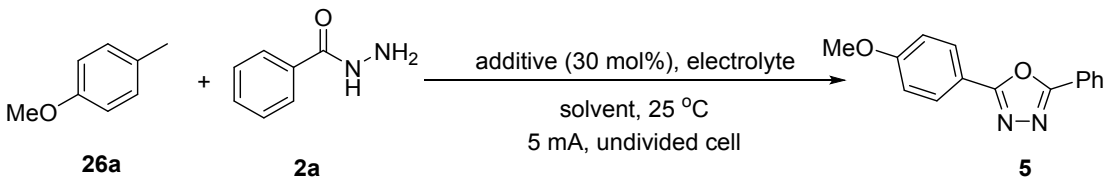


Exact Mass: 222.0793




## Conditions Screening of Electrochemical One-pot Synthesis of 1,3,4-Oxadiazoles from **26**

Under the above optimized conditions, the desired product **5** can be afforded with a low yield (Table S2, entry 1). As the solvent was changed to MeOH, although no desired product can be detected, we found the **26a** was full converted to the corresponding 4-methoxybenzaldehyde (Table S2, entry 5). This result inspired us to introduce the MeOH to the reaction system. To our delight, $Et_4NBr$ (0.1 M) as electrolyte with $CH_3CN$/MeOH (9:1) as solvent, the yield of **5** can be improved to 73% (Table S2, entry 7). Control experiments also confirmed the effect of oxygen and electricity (Table S2, entries 9, 10).




**26a** **2a** **5**

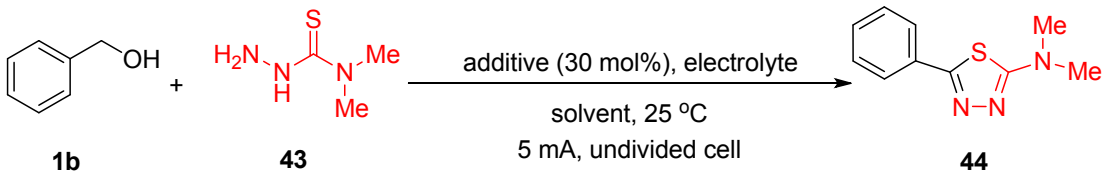
**Table S2.** Optimization of the conditions for the electrochemical cascade reaction[a]

| Entry | Additive | Electrolyte | Anode/ cathode | Solvent | Yield (%)[b] **5** |
|---|---|---|---|---|---|
| 1 | NHPI | $Et_4NBr$ (0.03 M)/ $Et_4NOTs$ (0.1 M) | Pt/Pt | $CH_3CN$/ TFE (2:1) | 19 |
| 2 | NHPI | $Et_4NOTs$ (0.1 M) | Pt/Pt | $CH_3CN$/ TFE (2:1) | 13 |
| 3 | NHPI | $Et_4NBr$ (0.1 M) | Pt/Pt | $CH_3CN$/ TFE (2:1) | 29 |
| 4 | NHPI | $Et_4NBr$ (0.1 M) | Pt/Pt | $CH_3CN$ | 45 |
| 5 | NHPI | $Et_4NBr$ (0.1 M) | Pt/Pt | MeOH | n.r. |
| 6 | NHPI | $Et_4NBr$ (0.1 M) | Pt/Pt | $CH_3CN$/MeOH (2:1) | 25 |
| 7 | **NHPI** | **$Et_4NBr$ (0.1 M)** | **Pt/Pt** | **$CH_3CN$/MeOH (9:1)** | **73** |
| 8[c] | NHPI | $Et_4NBr$ (0.1 M) | Pt/Pt | $CH_3CN$/MeOH (9:1) | 31 |
| 9[d] | NHPI | $Et_4NBr$ (0.1 M) | Pt/Pt | $CH_3CN$/MeOH (9:1) | 69 |
| 10[e] | NHPI | $Et_4NBr$ (0.1 M) | Pt/Pt | $CH_3CN$/MeOH (9:1) | n.r. |

[a] Reaction conditions: under atmospheric conditions at 25 °C, 4-methoxytoluene **26a** (0.2 mmol), additive (0.06 mmol, 30 mol%), electrolyte with solvent (3 mL) at a constant current of 5 mA for 3 h, then benzohydrazide **2a** (0.26 mmol) was added, the system was electrolyzed at a constant current of 5 mA for additional 7 h. NHPI = *N*-hydroxyphthalimide. [b] Isolated yield. [c]**26a** and **2a** were added simultaneously. [d]Under $N_2$ atmosphere. [e]Without electricity.

## Conditions Screening of Electrochemical One-pot Synthesis of 1,3,4-thiadiazoles

Based on the previous results, the optimization can be accomplished by taking $Et_4NBr$ (0.1 M) in $CH_3CN$ (3 mL) in the presence of NHPI (30 mol%) as redox mediator under constant current electrolysis. And the optimized conditions also showed good efficiency in the synthesis of imidazoles and oxazolines.




**1b** **43** **44**

**Table S3.** Optimization of the conditions for the electrochemical cascade reaction[a]

| Entry | Additive | Electrolyte | Anode/ | Solvent | Yield (%)[b] |
|---|---|---|---|---|---|

| | | | cathode | | **44** |
|---|---|---|---|---|---|
| 1 | NHPI | $Et_4NBr$ (0.03 M)/ $Et_4NOTs$ (0.1 M) | Pt/Pt | $CH_3CN$/ TFE (2:1) | 55 |
| 2 | NHPI | $Et_4NOTs$ (0.1 M) | Pt/Pt | $CH_3CN$/ TFE (2:1) | 23 |
| 3 | NHPI | $Et_4NBr$ (0.1 M) | Pt/Pt | $CH_3CN$/ TFE (2:1) | 62 |
| 4 | **NHPI** | **$Et_4NBr$ (0.1 M)** | **Pt/Pt** | **$CH_3CN$** | **87** |
| 5 | - | $Et_4NBr$ (0.1 M) | Pt/Pt | $CH_3CN$ | 16 |
| 6 | TEMPO | $Et_4NBr$ (0.1 M) | Pt/Pt | $CH_3CN$ | 17 |
| 7 | NHPI | $Et_4NBr$ (0.1 M) | Pt/Pt | MeOH | 11 |
| 8[c] | NHPI | $Et_4NBr$ (0.1 M) | Pt/Pt | $CH_3CN$ | 35 |
| 9[d] | NHPI | $Et_4NBr$ (0.1 M) | Pt/Pt | $CH_3CN$ | 83 |
| 10[e] | NHPI | $Et_4NBr$ (0.1 M) | Pt/Pt | $CH_3CN$ | n.r. |

[a] Reaction conditions: under atmospheric conditions at 25 °C, benzyl alcohol **1b** (0.2 mmol), additive (0.06 mmol, 30 mol%), electrolyte with solvent (3 mL) at a constant current of 5 mA for 3 h, then *N*, *N*-dimethylhydrazinecarbothioamide **43** (0.26 mmol) was added, the system was electrolyzed at a constant current of 5 mA for additional 7 h. NHPI = *N*-hydroxyphthalimide. [b] Isolated yield. [c]**1b** and **43** were added simultaneously. [d]Under $N_2$ atmosphere. [e]Without electricity.

## Procedure for the Synthesis of **3** - **23**, **25**, **34** - **42**

A 10-mL three-necked round-bottomed flask was charged with alcohol **1** (0.2 mmol), NHPI (0.006 mmol, 0.0098 g), $Et_4NBr$ (0.09 mmol, 0.0189 g) and $Et_4NOTs$ (0.3 mmol, 0.0903 g). The flask was equipped with two platinum plates (1 cm x 1 cm) as anode and cathode respectively, then $CH_3CN$ (2 mL) and TFE (1 mL) were added. The mixture was stirred at 25 °C and electrolyzed at a constant current of 5 mA for 3 h. Then acyl hydrazine (0.26 mmol) was added, and the constant current (5 mA) electrolysis was carried out at 25 °C for 7 h. The reaction mixture was concentrated under reduced pressure, and the residue was purified through silica gel chromatography with petroleum/ ethyl acetate as eluent.

## Procedure for the Synthesis of **5**, **27**- **33** from substituted toluenes

A 10-mL three-necked round-bottomed flask was charged with substituted toluene **26** (0.2 mmol), NHPI (0.006 mmol, 0.0098 g), $Et_4NBr$ (0.3 mmol, 0.0630 g). The flask was equipped with two platinum plates (1 cm x 1 cm) as anode and cathode respectively, then $CH_3CN$ (2.7 mL) and MeOH (0.3 mL) were added. The mixture was stirred at 25 °C and electrolyzed at a constant current of 5 mA for 3 h. Then acyl

hydrazine (0.26 mmol) was added, and the constant current (5 mA) electrolysis was carried out at 25 °C for 7 h. The reaction mixture was concentrated under reduced pressure, and the residue was purified through silica gel chromatography with petroleum/ ethyl acetate as eluent.

### Procedure for the Synthesis of 1,3,4-thiadiazoles **44-49**

A 10-mL three-necked round-bottomed flask was charged with alcohol **1** (0.2 mmol), NHPI (0.006 mmol, 0.0098 g), $Et_4NBr$ (0.3 mmol, 0.0630 g). The flask was equipped with two platinum plates (1 cm x 1 cm) as anode and cathode respectively, then $CH_3CN$ (3 mL) was added. The mixture was stirred at 25 °C and electrolyzed at a constant current of 5 mA for 3 h. Then *N*, *N*-dimethylhydrazinecarbothioamide (0.26 mmol, 0.0309 g) was added, and the constant current (5 mA) electrolysis was carried out at 25 °C for 7 h. The reaction mixture was concentrated under reduced pressure, and the residue was purified through silica gel chromatography with petroleum/ ethyl acetate as eluent.

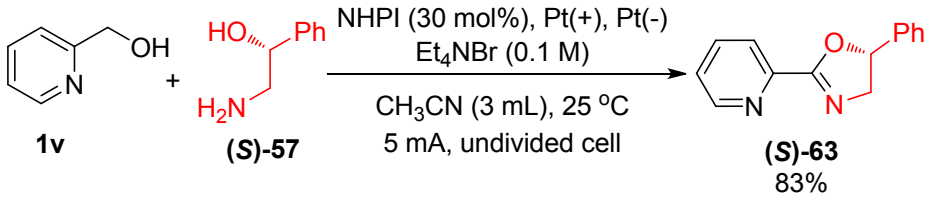
### Procedure for the Synthesis of imidazoles **51-56**

A 10-mL three-necked round-bottomed flask was charged with alcohol **1** (0.2 mmol), NHPI (0.006 mmol, 0.0098 g), $Et_4NBr$ (0.3 mmol, 0.0630 g). The flask was equipped with two platinum plates (1 cm x 1 cm) as anode and cathode respectively, then $CH_3CN$ (3 mL) was added. The mixture was stirred at 25 °C and electrolyzed at a constant current of 5 mA for 3 h. Then *N*-methylethanediamine (0.26 mmol, 0.0192 g) was added, and the constant current (5 mA) electrolysis was carried out at 25 °C for 7 h. The reaction mixture was concentrated under reduced pressure, and the residue was purified through silica gel chromatography with petroleum/ ethyl acetate as eluent.

### Procedure for the Synthesis of oxazolines **58-63**

A 10-mL three-necked round-bottomed flask was charged with alcohol **1** (0.2 mmol), NHPI (0.006 mmol, 0.0098 g), $Et_4NBr$ (0.3 mmol, 0.0630 g). The flask was equipped with two platinum plates (1 cm x 1 cm) as anode and cathode respectively, then $CH_3CN$ (3 mL) was added. The mixture was stirred at 25 °C and electrolyzed at a constant current of 5 mA for 3 h. Then 2-phenylglycinol (0.26 mmol, 0.0356 g) was added, and the constant current (5 mA) electrolysis was carried out at 25 °C for 7 h. The reaction mixture was concentrated under reduced pressure, and the residue was purified through silica gel chromatography with petroleum/ ethyl acetate as eluent.

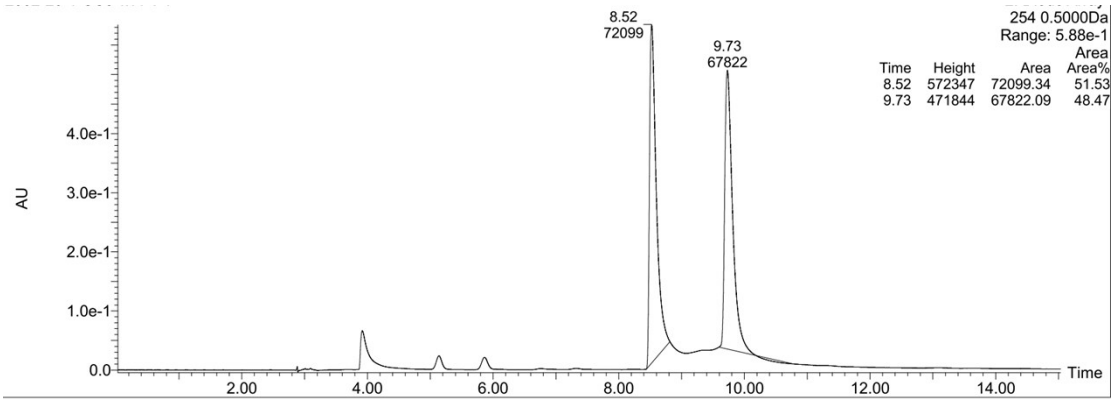
## Procedure for the Synthesis of oxazolines (*S*)-63



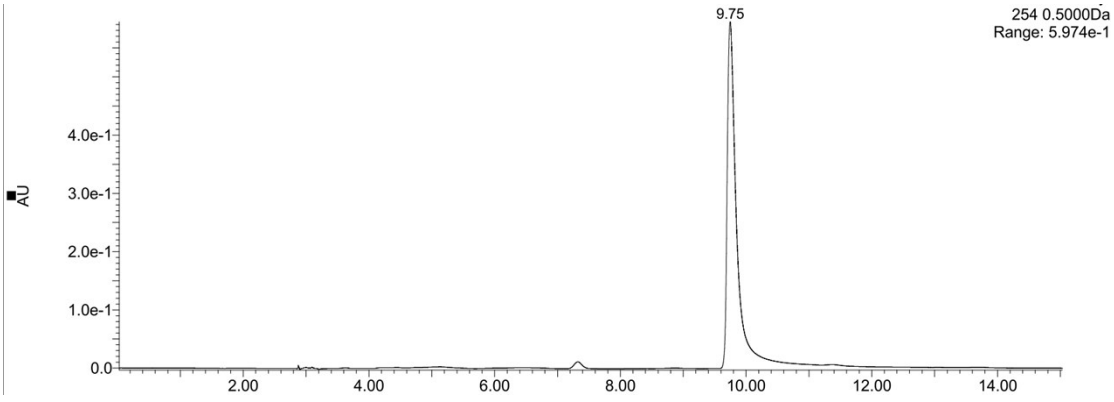

A 10-mL three-necked round-bottomed flask was charged with 2-(hydroxymethyl)pyridine **1v** (0.2 mmol, 0.0218 g), NHPI (0.006 mmol, 0.0098 g), $Et_4NBr$ (0.3 mmol, 0.0630 g). The flask was equipped with two platinum plates (1 cm x 1 cm) as anode and cathode respectively, then $CH_3CN$ (3 mL) was added. The mixture was stirred at 25 °C and electrolyzed at a constant current of 5 mA for 3 h. Then (*S*)-2-phenylglycinol (0.26 mmol, 0.0356 g) was added, and the constant current (5 mA) electrolysis was carried out at 25 °C for 7 h. The reaction mixture was concentrated under reduced pressure, and the residue was purified through silica gel chromatography with petroleum/ ethyl acetate as eluent.

## UPCC spectra of 63

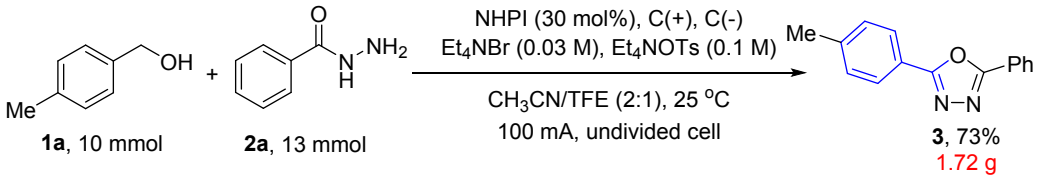
Racemic sample

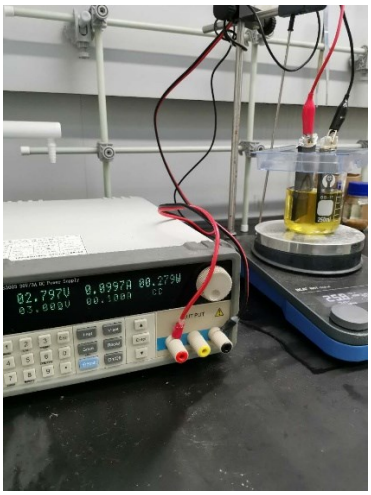


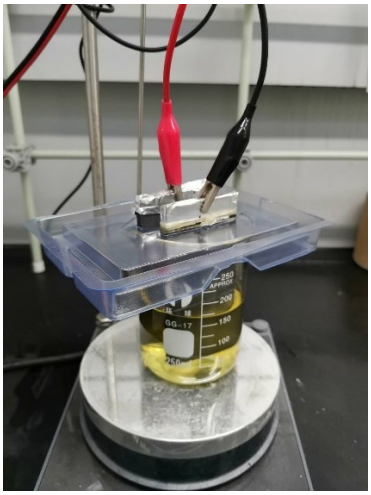

(*S*)-**63**



## Gram scale synthesis of 3



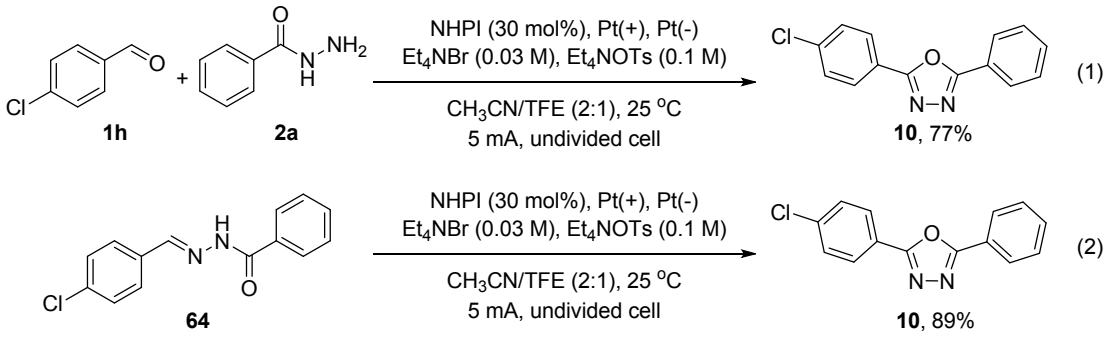


A 250-mL beaker was charged with alcohol **1a** (10 mmol, 1.22g), NHPI (0.3 mmol, 0.50 g), $Et_4NBr$ (4.5 mmol, 0.95 g) and $Et_4NOTs$ (15 mmol, 4.51 g). The flask was equipped with two graphite carbon plates (10 cm x 5 cm x 0.5cm) as anode and cathode respectively, then $CH_3CN$ (100 mL) and TFE (50 mL) were added. The mixture was stirred at 25 °C and electrolyzed at a constant current of 100 mA for 7 h. Then Benzoyl hydrazine (13 mmol, 1.77 g) was added, and the constant current (100 mA) electrolysis was carried out at 25 °C for 10 h. The reaction mixture was concentrated under reduced pressure, and the residue was purified through silica gel chromatography with petroleum/ ethyl acetate (10:1) as eluent give the final product **3** as white solid (1.72g, 73%)

## Mechanism study

### Control experiments

Treatment of **1h** with **2a** under standard conditions afforded the desired 1,3,4-oxadiazoles **10** in 77% yield (eq. 1). While acylhydrazone **64**, prepared beforehand from the condensation of aldehyde **1h** with benzohydrazine **2a**, was used as the substrate under the same condition, 89% yield of **10** was obtained (eq. 2). The above results indicate the formation of 1,3,4-oxadiazole may involve an oxidation of alcohol to aldehyde as well as an oxidative annulation of acylhydrazone.




## Cyclic voltammetry (CV) experiments

We probed the mechanism by means of a series of cyclic voltammetric (CV) analyses. As depicted in Fig. 1A, the oxidative peak of NHPI was observed at 1.76 V *vs*. Ag/AgCl (curve c) in acetonitrile and benzyl alcohol was found to be oxidized at 1.78 V *vs*. Ag/AgCl (curve b) in acetonitrile. Compared with curve b, the anodic current of the mixture of benzyl alcohol and NHPI (curve d) was greatly enhanced with similar onset potentials. Furthermore, compared to that of pure acylhydrazone **64**, the CV curve of the mixture of **64**, with NHPI in acetonitrile also demonstrated a slight increase in the catalytic current (Fig. S1B, curve e, f). The above results represent typical behavior of mediator-assisted dehydrogenation, and the NHPI undergoes anodic oxidation to afford phthalimido-*N*-oxyl (PINO), which then mediates hydrogen-atom transfer (HAT) from the substrates and accordingly leads to the absence of reduction current in CV.[2]

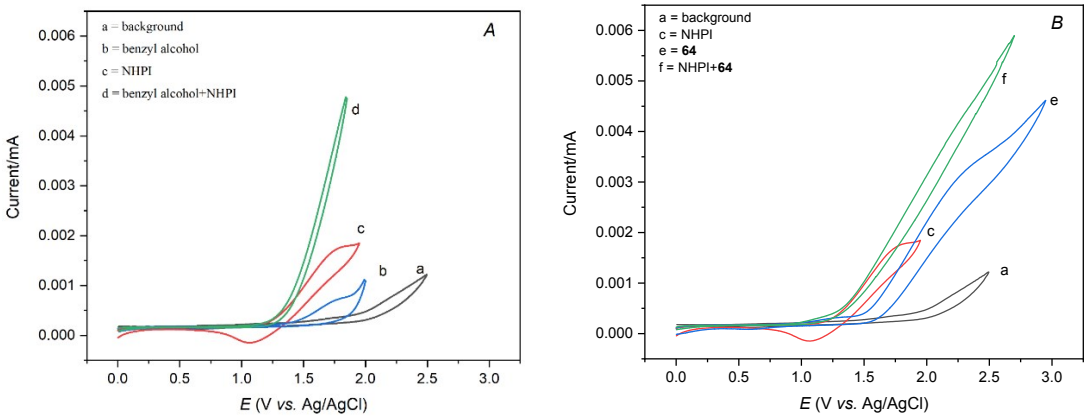
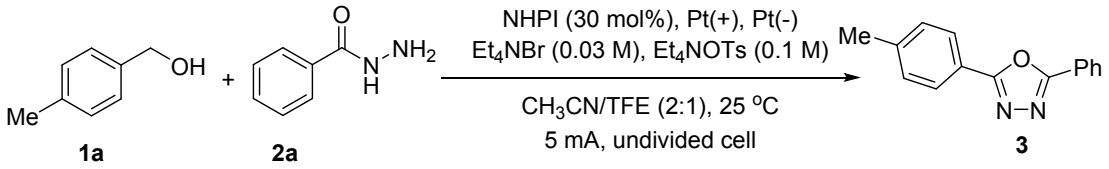
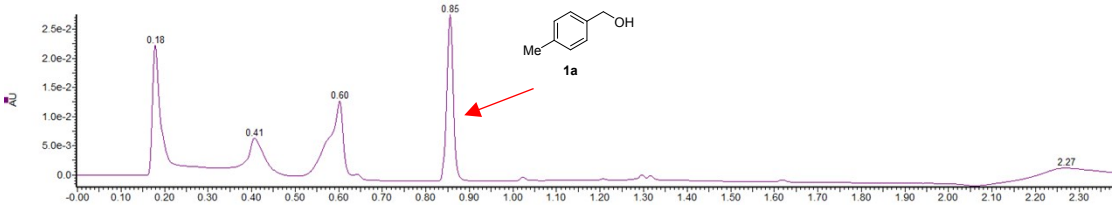



**Figure S2**. CVs (0.1 V/s) in MeCN (3 mL) and $Et_4NPF_6$ (0.1 M) of: background (**a**); benzyl alcohol (0.2 M, **b**); NHPI (0.01 M, **c**), NHPI (0.01 M) + benzyl alcohol (0.2 M) (**d**); acylhydrazone **64** (0.2 M, **e**); NHPI (0.01 M) + acylhydrazone **64** (0.2 M) (**f**). The voltammogram was obtained with Pt plates (1×1 cm) as the working and counter electrode and Ag/AgCl electrode as the reference electrode at room temperature.

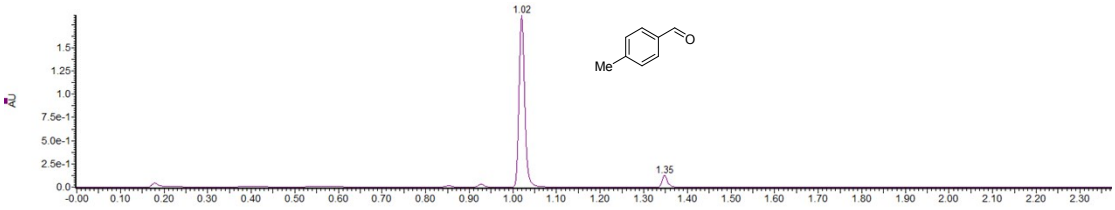
## Sample experiment via UPLC-MS



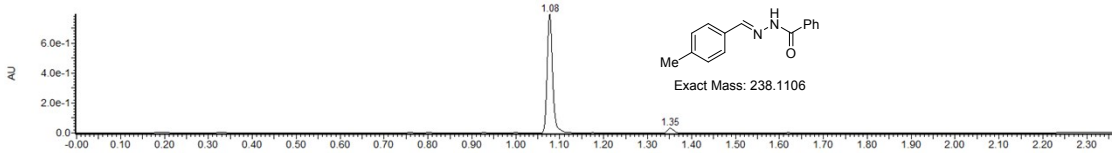

0 h

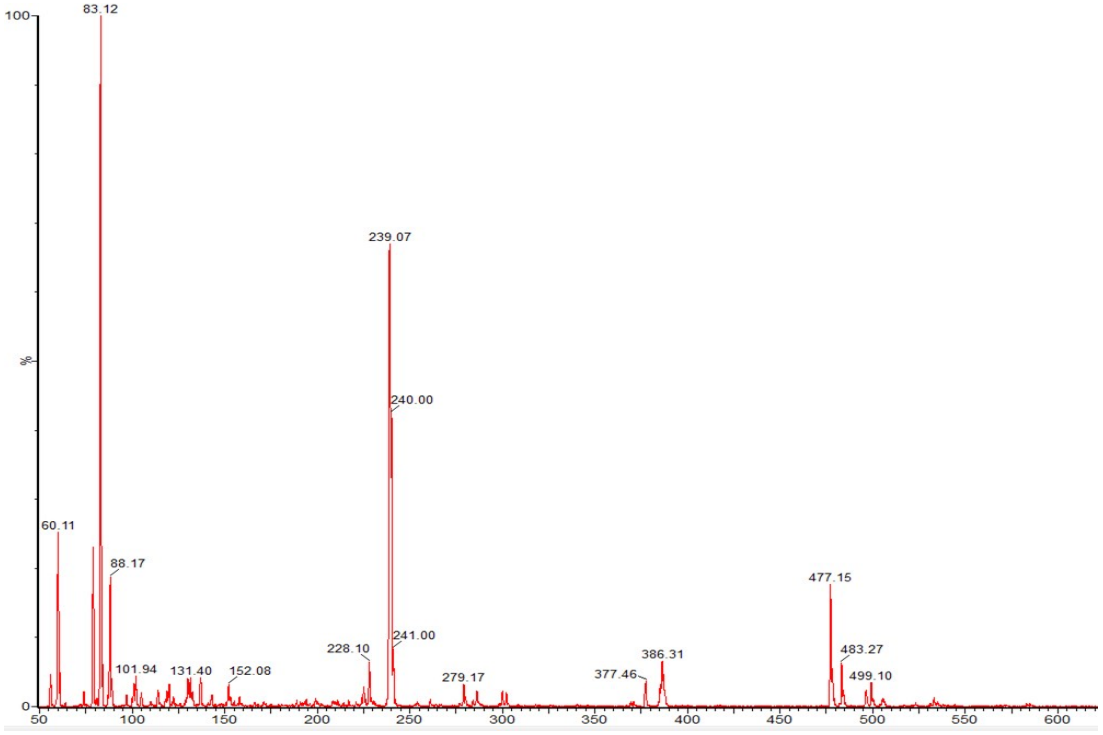



3 h

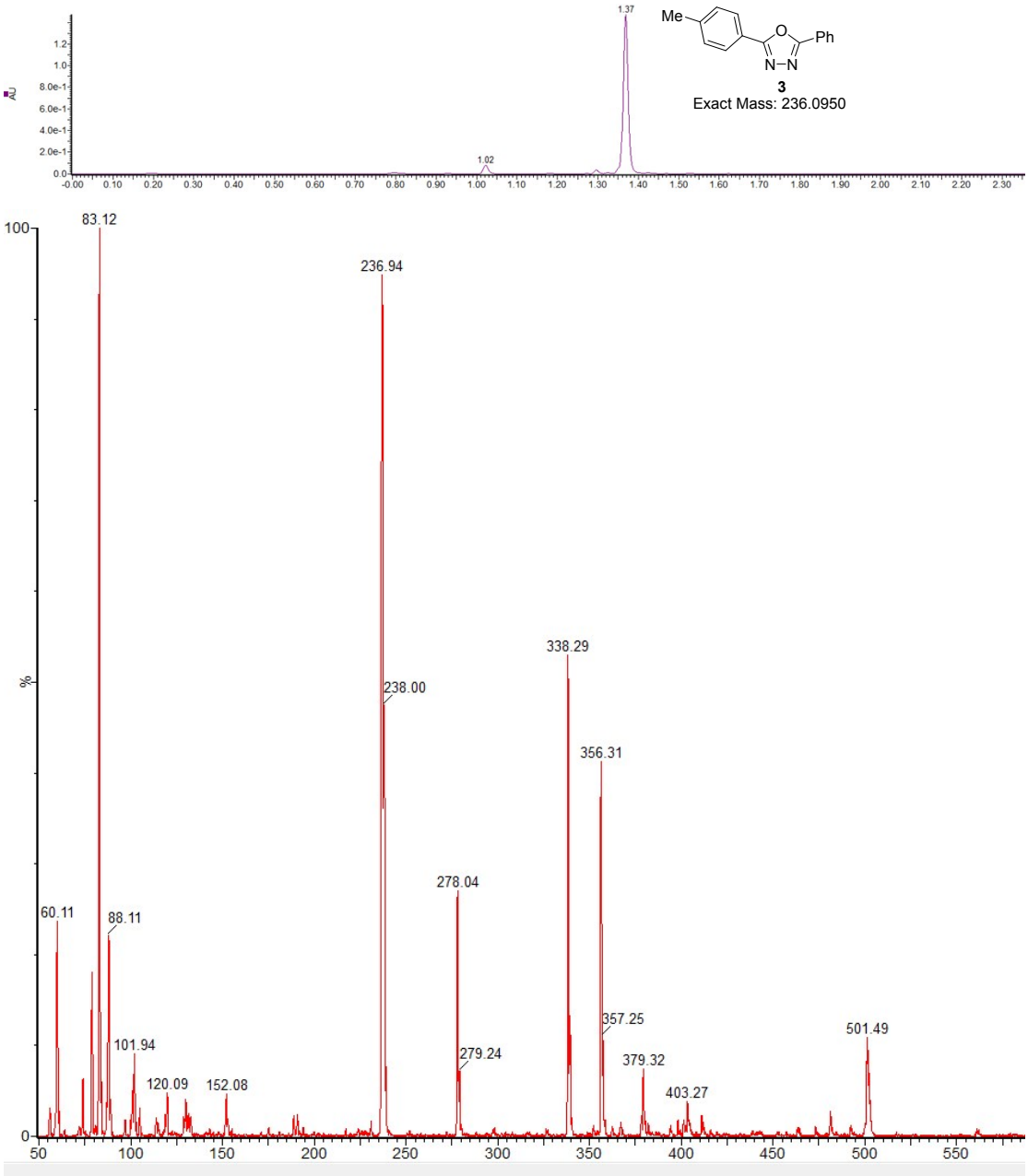



0.5 h after the addition of **2a** without the current

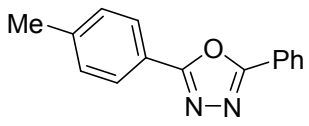


7h electrolysis after the addition of **2a**



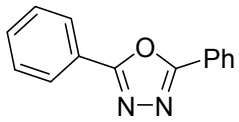

## Characterization data for the electrolysis products




**3**

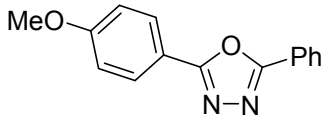
**2-phenyl-5-(*p*-tolyl)-1,3,4-oxadiazole**.[3] Yield = 86%; white solid; $^{1}$H NMR (600 MHz, Chloroform-*d*) δ 8.16 – 8.05 (m, 2H), 8.03 – 7.97 (m, 2H), 7.55 – 7.47 (m, 3H), 7.30 (d, *J* = 7.9 Hz, 2H), 2.40 (s, 3H). $^{13}$C NMR (MHz, $CDCl_3$) δ 164.7, 164.3, 142.3, 131.6,

129.8, 129.0, 126.9, 126.8, 124.0, 121.1, 21.7. M.p. 129-130 °C. HRMS (ESI) calculated for $C_{15}H_{12}N_2O$ [M+H]+: 237.1023; found: 237.1023.



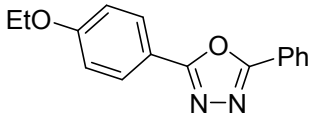

**4**

**2,5-diphenyl-1,3,4-oxadiazole**.[3] Yield = 81%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.16 – 8.13 (m, 2H), 8.12 – 8.06 (m, 2H), 7.60 – 7.50 (m, 6H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 165.5, 164.5, 138.7, 132.6, 130.2, 129.9, 128.9, 127.7, 124.5, 123.1. M.p. 132-133 °C. HRMS (ESI) calculated for $C_{14}H_{10}N_2O$ [M+H]+: 223.0866; found: 223.0871.



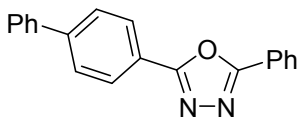

**5**

**2-(4-methoxyphenyl)-5-phenyl-1,3,4-oxadiazole**.[3] Yield = 80%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.21 – 8.08 (m, 2H), 8.01 (dd, *J* = 8.2, 2.3 Hz, 2H), 7.60 – 7.45 (m, 3H), 7.37 – 7.27 (m, 2H), 2.43 (d, *J* = 2.5 Hz, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.7, 164.3, 142.3, 131.6, 129.8, 129.0, 126.9, 124.0, 121.2, 21.7. M.p. 129-130 °C. HRMS (ESI) calculated for $C_{15}H_{12}N_2O_2$ [M+H]+: 253.0972; found: 253.0979.




**6**

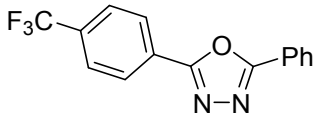
**2-(4-ethoxyphenyl)-5-phenyl-1,3,4-oxadiazole**. Yield = 73%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.17 – 8.11 (m, 2H), 8.10 – 8.02 (m, 2H), 7.54 (d, *J* = 6.4 Hz, 3H), 7.10 – 6.98 (m, 2H), 4.12 (q, *J* = 7.0 Hz, 2H), 1.47 (t, *J* = 7.0 Hz, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.6, 164.1, 161.7, 131.5, 129.0, 128.7, 126.8, 124.1, 116.2, 114.9, 63.8, 14.7. M.p. 150-151 °C. HRMS (ESI) calculated for $C_{16}H_{14}N_2O_2$ [M+H]+: 267.1129; found: 267.1132.




**7**

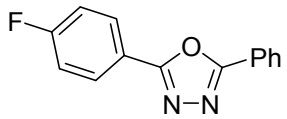
**2-([1,1'-biphenyl]-4-yl)-5-phenyl-1,3,4-oxadiazole**.[3] Yield = 87%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.21 – 8.17 (m, 2H), 8.17 – 8.12 (m, 2H), 7.77 –

7.70 (m, 2H), 7.67 – 7.62 (m, 2H), 7.57 – 7.51 (m, 3H), 7.50 – 7.44 (m, 2H), 7.43 – 7.38 (m, 1H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.6, 164.5, 144.4, 139.8, 131.7, 129.1, 128.9, 128.2, 127.7, 127.3, 127.1, 126.9, 123.9, 122.7. M.p. 169-170 °C. HRMS (ESI) calculated for $C_{20}H_{14}N_2O$ [M+H]+: 299.1179; found: 299.1170.



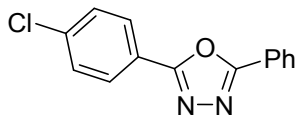

**8**

**2-phenyl-5-(4-(trifluoromethyl)phenyl)-1,3,4-oxadiazole**.[3] Yield = 88%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.19 – 8.05 (m, 4H), 7.57 – 7.47 (m, 3H), 7.21 (t, *J* = 8.6 Hz, 2H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.7 (d, *J* = 280.8 Hz), 164.6, 163.9, 131.8, 129.2, 129.2, 129.1, 126.9, 123.8, 120.2 (q, *J* = 3.0 Hz), 116.5, 116.3. $^{19}$F NMR (565 MHz, $CDCl_3$) δ -106.80. M.p. 160-162 °C. HRMS (ESI) calculated for $C_{15}H_9F_3N_2O$ [M+H]+: 291.0740; found: 291.0751.



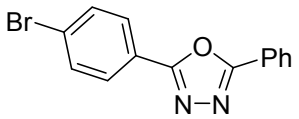

**9**

**2-(4-fluorophenyl)-5-phenyl-1,3,4-oxadiazole**.[3] Yield = 71%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.37 – 7.98 (m, 4H), 7.77 – 7.45 (m, 3H), 7.24 – 7.19 (m, 2H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.7 (d, *J* = 250 Hz), 164.5, 163.7, 131.7, 129.2 (d, *J* = 9 Hz), 129.0, 126.9, 123.7, 120.2 (d, *J* = 3 Hz), 116.3 (d, *J* = 22.5 Hz). $^{19}$F NMR (565 MHz, $CDCl_3$) δ -106.81. M.p. 150-151 °C. HRMS (ESI) calculated for $C_{14}H_9FN_2O$ [M+H]+: 241.0772; found: 241.0778.



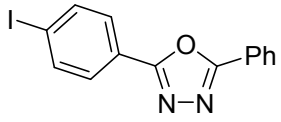

**10**

**2-(4-chlorophenyl)-5-phenyl-1,3,4-oxadiazole**.[3] Yield = 75%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.16 – 8.13 (m, 2H), 8.12 – 8.06 (m, 2H), 7.60 – 7.50 (m, 5H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 165.5, 164.5, 138.7, 132.6, 130.2, 129.9, 128.9, 127.7, 124.5, 123.1. M.p. 155-156 °C. HRMS (ESI) calculated for $C_{14}H_9ClN_2O$ [M+H]+: 257.0477; found: 257.0483.



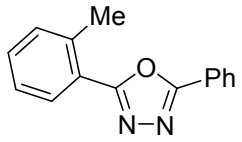

**11**

**2-(4-bromophenyl)-5-phenyl-1,3,4-oxadiazole**.[4] Yield = 66%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.15 (dq, *J* = 6.1, 1.6 Hz, 2H), 8.09 – 7.99 (m, 2H), 7.76 – 7.68 (m, 2H), 7.57 (m, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.8, 163.9, 132.5, 131.9, 129.1, 128.32, 126.9, 126.4, 123.7, 122.8. M.p. 143-145 °C. HRMS (ESI) calculated for $C_{14}H_9BrN_2O$ [M+H]+: 300.9972; found: 300.9962.



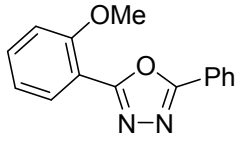

**12**

**2-(4-iodophenyl)-5-phenyl-1,3,4-oxadiazole**.[3] Yield = 62%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.12 (dt, *J* = 6.7, 1.7 Hz, 2H), 7.92 – 7.86 (m, 2H), 7.85 (d, *J* = 8.7 Hz, 2H), 7.54 (m, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.7, 164.0, 138.3, 131.9, 129.1, 128.2, 126.9, 123.7, 123.3, 98.6. M.p. 165-156 °C. HRMS (ESI) calculated for $C_{14}H_9IN_2O$ [M+H]+: 348.9833; found: 348.9830.



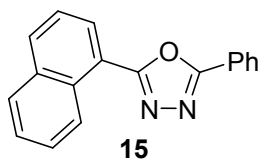

**13**

**2-phenyl-5-(o-tolyl)-1,3,4-oxadiazole**.[4] Yield = 70%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.51 – 8.46 (m, 2H), 8.39 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.93 – 7.85 (m, 3H), 7.78 (td, *J* = 7.5, 1.4 Hz, 1H), 7.74 – 7.67 (m, 2H), 3.12 (s, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.9, 164.2, 138.5, 131.8, 131.70, 131.2, 129.1, 128.9, 126.9, 126.2, 124.0, 123.0, 22.1. M.p. 1113-1114 °C. HRMS (ESI) calculated for $C_{15}H_{12}N_2O$ [M+H]+: 237.1023; found: 237.1025.



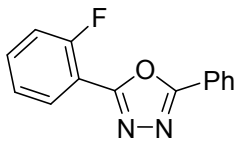

**14**

**2-(2-methoxyphenyl)-5-phenyl-1,3,4-oxadiazole**.[3] Yield = 63%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.11 (dd, *J* = 7.7, 2.0 Hz, 2H), 7.99 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.59 – 7.44 (m, 4H), 7.11 – 6.99 (m, 2H), 3.96 (s, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.3, 163.3, 157.9, 133.1, 131.5, 130.4, 128.9, 126.9, 124.1, 120.7, 113.1, 112.0, 56.0. M.p. 90-91 °C. HRMS (ESI) calculated for $C_{15}H_{12}N_2O_2$ [M+H]+: 253.0972; found: 253.0977.



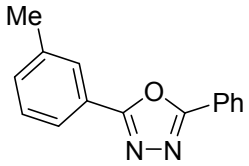

**15**

**2-(naphthalen-1-yl)-5-phenyl-1,3,4-oxadiazole**.[3] Yield = 72%; white solid; $^1$H NMR (500 MHz, Chloroform-*d*) δ 9.30 (d, *J* = 8.7 Hz, 1H), 8.25 (d, *J* = 7.3 Hz, 1H), 8.19 (dt, *J* = 7.3, 2.1 Hz, 2H), 8.02 (d, *J* = 8.2 Hz, 1H), 7.92 (d, *J* = 8.2 Hz, 1H), 7.70 (t, *J* = 7.8 Hz, 1H), 7.64 – 7.50 (m, 5H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 164.6, 164.2, 133.9, 132.59, 131.8, 130.1, 129.1, 128.7, 128.3, 128.2, 127.0, 126.7, 126.3, 124.9, 123.9, 120.5. M.p. 116-117 °C. HRMS (ESI) calculated for $C_{18}H_{12}N_2O$ [M+H]+: 273.1023; found: 273.1025.



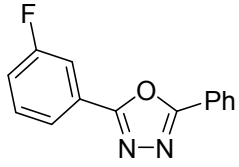

**16**

**2-(2-fluorophenyl)-5-phenyl-1,3,4-oxadiazole**.[5] Yield = 65%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.16 (m, 3H), 7.59 – 7.50 (m, 4H), 7.32 (td, *J* = 7.6, 1.1 Hz, 1H), 7.27 (ddd, *J* = 10.5, 8.3, 1.2 Hz, 1H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.9 (d, *J* = 1.5 Hz), 161.4 (d, *J* = 6 Hz), 160.0 (d, *J* = 256 Hz), 133.5 9 (d, *J* = 9 Hz), 131.9, 129.8 (d, *J* = 1.5 Hz), 129.1, 127.0, 124.7 (d, *J* = 4.5 Hz), 123.8, 117.0 (d, *J* = 19.5 Hz), 112.4 (d, *J* = 12 Hz). $^{19}$F NMR (565 MHz, $CDCl_3$) δ -110.1. M.p. 123-125 °C. HRMS (ESI) calculated for $C_{14}H_9FN_2O$ [M+H]+: 241.0772; found: 241.0779.




**17**

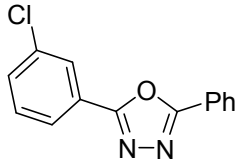
**2-phenyl-5-(m-tolyl)-1,3,4-oxadiazole**.[4] Yield = 86%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.16 – 8.11 (m, 2H), 7.96 (s, 1H), 7.93 (d, *J* = 7.7 Hz, 1H), 7.57 – 7.50 (m, 3H), 7.41 (t, *J* = 7.6 Hz, 1H), 7.35 (d, *J* = 7.6 Hz, 1H), 2.45 (s, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.7, 164.5, 138.9, 132.5, 131.6, 129.1, 128.9, 127.4, 126.9, 124.1, 123.9, 123.8, 21.3. M.p. 111-112 °C. HRMS (ESI) calculated for $C_{15}H_{12}N_2O$ [M+H]+: 237.1023; found: 237.1028.




**18**

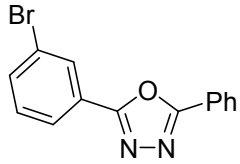
**2-(3-fluorophenyl)-5-phenyl-1,3,4-oxadiazole**.[3] Yield = 67%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.13 – 8.09 (m, 2H), 7.92 (dt, *J* = 7.7, 1.2 Hz, 1H), 7.81

(ddd, $J$ = 9.1, 2.5, 1.5 Hz, 1H), 7.58 – 7.46 (m, 4H), 7.26 – 7.21 (m, 1H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.7, 163.4 (d, $J$ = 3 Hz), 162.8 (d, $J$ = 246 Hz), 131.8, 130.8 (d, $J$ = 7.5 Hz), 129.0, 126.90, 125.7 (d, $J$ = 9 Hz), 123.6, 122.6 (d, $J$ = 3 Hz), 118.7 (d, $J$ = 22.5 Hz), 113.8 (d, $J$ = 24 Hz). $^{19}$F NMR (565 MHz, $CDCl_3$) δ -111.09. M.p. 128-129 $^{o}$C. HRMS (ESI) calculated for $C_{14}H_9FN_2O$ [M+H]+: 241.0772; found: 241.0775.



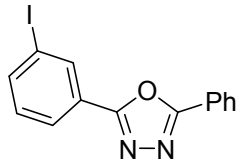

**19**

**2-(3-chlorophenyl)-5-phenyl-1,3,4-oxadiazole**.[3] Yield = 70%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.13 – 8.09 (m, 2H), 8.08 (t, $J$ = 1.9 Hz, 1H), 8.00 (dt, $J$ = 7.6, 1.4 Hz, 1H), 7.56 – 7.47 (m, 4H), 7.45 (t, $J$ = 7.8 Hz, 1H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.8, 163.3, 135.2, 131.9, 131.7, 130.5, 129.1, 126.9, 126.8, 125.5, 124.9, 123.6. M.p. 129-130 $^{o}$C. HRMS (ESI) calculated for $C_{14}H_9ClN_2O$ [M+H]+: 257.0477; found: 257.0480.



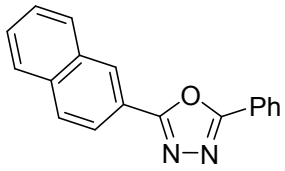

**20**

**2-(3-bromophenyl)-5-phenyl-1,3,4-oxadiazole**.[3] Yield = 76%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.25 (t, $J$ = 1.8 Hz, 1H), 8.18 – 8.10 (m, 2H), 8.07 (dt, $J$ = 7.8, 1.3 Hz, 1H), 7.66 (ddd, $J$ = 8.0, 2.0, 1.0 Hz, 1H), 7.53 (m, 3H), 7.40 (t, $J$ = 7.9 Hz, 1H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.8, 163.2, 134.6, 131.9, 130.7, 129.7, 129.1, 127.0, 125.8, 125.5, 123.6, 123.1. M.p. 112-113 $^{o}$C.HRMS (ESI) calculated for $C_{14}H_9BrN_2O$ [M+H]+: 300.9972; found: 300.9979.



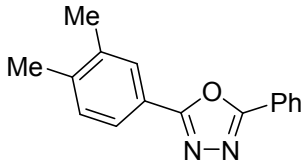

**21**

**2-(3-iodophenyl)-5-phenyl-1,3,4-oxadiazole**. Yield = 72%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.46 (t, $J$ = 1.7 Hz, 1H), 8.12 (m, 3H), 7.87 (dt, $J$ = 7.9, 1.3 Hz, 1H), 7.54 (qd, $J$ = 8.7, 7.8, 3.6 Hz, 3H), 7.27 (t, $J$ = 7.9 Hz, 1H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.8, 163.0, 140.6, 135.5, 131.9, 130.7, 129.1, 126.9, 126.0, 125.7, 123.6, 94.5. M.p. 150-151 $^{o}$C. HRMS (ESI) calculated for $C_{14}H_9IN_2O$ [M+H]+: 348.9833; found: 348.9837.



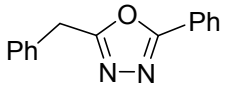

**22**

**2-(naphthalen-2-yl)-5-phenyl-1,3,4-oxadiazole**.[3] Yield = 78%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.58 (d, *J* = 1.7 Hz, 1H), 8.19 (m, 3H), 7.96 (dd, *J* = 7.2, 3.0 Hz, 2H), 7.90 – 7.86 (m, 1H), 7.62 – 7.53 (m, 5H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.7, 164.6, 134.7, 132.8, 131.7, 129.1, 129.0, 128.8, 127.9, 127.3, 127.1, 126.9, 123.9, 123.2, 121.1. M.p. 112-113 °C. HRMS (ESI) calculated for $C_{18}H_{12}N_2O$ [M+H]+: 273.1023; found: 273.1025.



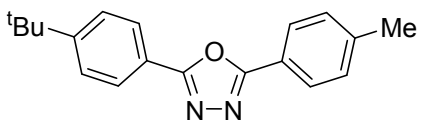

**23**

**2-(3,4-dimethylphenyl)-5-phenyl-1,3,4-oxadiazole**.[3] Yield = 73%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.17 – 8.10 (m, 2H), 7.92 (d, *J* = 1.9 Hz, 1H), 7.85 (dd, *J* = 7.8, 1.9 Hz, 1H), 7.54 (m, 3H), 7.31 – 7.23 (m, 1H), 2.36 (s, 3H), 2.34 (s, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.8, 164.2, 141.0, 137.6, 131.6, 130.3, 129.0, 127.9, 126.9, 124.4, 124.1, 121.3, 20.0, 19.7. M.p. 113-115 °C.HRMS (ESI) calculated for $C_{16}H_{14}N_2O$ [M+H]+: 251.1179; found: 251.1183.




**25**

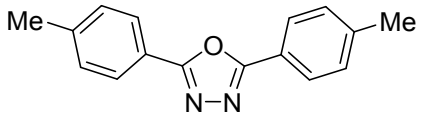
**2-benzyl-5-phenyl-1,3,4-oxadiazole**.[6] Yield =62%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.15 – 7.91 (m, 2H), 7.57 – 7.46 (m, 3H), 7.38 (d, *J* = 5.9 Hz, 4H), 7.34 – 7.31 (m, 1H), 4.30 (s, 2H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 165.3, 165.2, 133.9, 131.6, 129.0, 128.9, 128.8, 127.5, 126.8, 123.8, 31.9. M.p. 97-98 °C. HRMS (ESI) calculated for $C_{15}H_{12}N_2O$ [M+H]+: 237.1022; found: 237.1028.




**27**

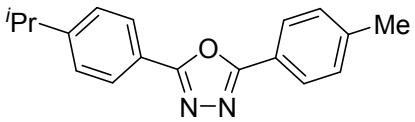
**2-(4-(*tert*-butyl)phenyl)-5-(*p*-tolyl)-1,3,4-oxadiazole**.[7] Yield = 61%; white solid; $^1$H

NMR (500 MHz, Chloroform-*d*) δ 8.06 (d, *J* = 8.4 Hz, 2H), 8.04 – 7.99 (m, 2H), 7.54 (dd, *J* = 8.6, 2.4 Hz, 2H), 7.31 (dd, *J* = 7.9, 4.3 Hz, 2H), 2.41 (s, 3H), 1.37 (s, 9H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 164.5, 164.3, 155.2, 142.1, 129.7, 126.8, 126.7, 125.9, 121.2, 121.21, 35.1, 31.1, 21.6. M.p. 129-130 °C. HRMS (ESI) calculated for $C_{19}H_{20}N_2O$ [M+H]+: 293.1648; found: 293.1648.



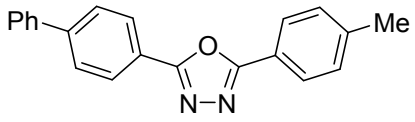

**28**

**2,5-di-*p*-tolyl-1,3,4-oxadiazole**. Yield = 53%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.01 (d, *J* = 8.0 Hz, 4H), 7.32 (d, *J* = 7.8 Hz, 4H), 2.42 (s, 6H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.5, 142.2, 129.7, 126.8, 121.2, 21.6. M.p. 169-173 °C. HRMS (ESI) calculated for $C_{16}H_{14}N_2O$ [M+H]+: 251.1179; found: 251.1181.



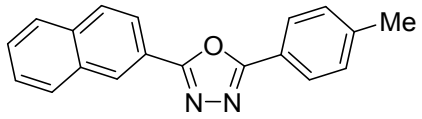

**29**

**2-([1,1'-biphenyl]-4-yl)-5-(*p*-tolyl)-1,3,4-oxadiazole**. Yield = 3%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.09 – 8.04 (m, 1H), 8.05 – 8.01 (m, 2H), 7.39 (dd, *J* = 8.4, 2.6 Hz, 2H), 7.33 (dd, *J* = 8.5, 4.5 Hz, 2H), 3.00 (tt, *J* = 7.6, 3.7 Hz, 1H), 2.44 (m, 3H), 1.31 (m, 6H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.5, 164.5, 152.9, 142.2, 129.8, 127.2, 127.0, 126.8, 121.6, 121.3, 34.3, 23.7, 21.7. M.p. 153-156 °C. HRMS (ESI) calculated for $C_{18}H_{18}N_2O$ [M+H]+: 279.1492; found: 279.1495.



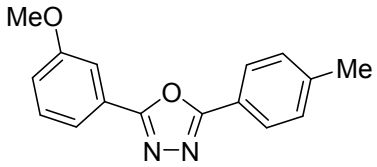

**30**

**2-([1,1'-biphenyl]-4-yl)-5-(*p*-tolyl)-1,3,4-oxadiazole**. Yield = 31%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.20 (d, *J* = 8.4 Hz, 2H), 8.05 (d, *J* = 8.1 Hz, 2H), 7.76 (d, *J* = 8.4 Hz, 2H), 7.67 (d, *J* = 8.3 Hz, 2H), 7.50 (t, *J* = 7.6 Hz, 2H), 7.42 (t, *J* = 7.4 Hz, 1H), 7.35 (d, *J* = 7.9 Hz, 2H), 2.46 (s, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.7, 164.2, 144.3, 142.3, 139.8, 129.8, 129.0, 128.2, 127.6, 127.3, 127.2, 126.9, 122.7, 121.2, 21.7. M.p. 171-172 °C. HRMS (ESI) calculated for $C_{21}H_{16}N_2O$ [M+H]+: 313.1335; found: 313.1330.



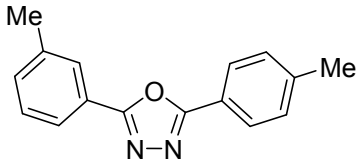

**31**

**2-(naphthalen-2-yl)-5-(*p*-tolyl)-1,3,4-oxadiazole**.[15] Yield = 65%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.61 (s, 1H), 8.21 (d, *J* = 8.6 Hz, 1H), 8.09 (d, *J* = 8.1 Hz, 2H), 7.98 (d, *J* = 8.3 Hz, 2H), 7.90 (d, *J* = 8.9 Hz, 1H), 7.63 – 7.54 (m, 2H), 7.36 (d, *J* = 7.9 Hz, 2H), 2.46 (s, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.8, 164.5, 142.3, 134.6, 132.8, 129.8, 129.0, 128.8, 127.9, 127.9, 127.2, 127.1, 126.9, 123.3, 121.3, 121.2, 21.7. M.p. 151-153 °C. HRMS (ESI) calculated for $C_{19}H_{14}N_2O$ [M+H]+: 287.1179; found: 287.1185.



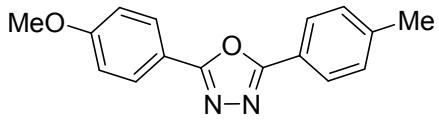

**32**

**2-(3-methoxyphenyl)-5-(*p*-tolyl)-1,3,4-oxadiazole**.[2] Yield = 37%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.03 (d, *J* = 8.1 Hz, 2H), 7.72 (d, *J* = 7.6 Hz, 1H), 7.68 (s, 1H), 7.45 (t, *J* = 8.0 Hz, 1H), 7.35 (d, *J* = 7.8 Hz, 2H), 7.10 (d, *J* = 8.3 Hz, 1H), 3.92 (s, 3H), 2.45 (s, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.7, 164.3, 159.9, 142.3, 130.2, 129.8, 126.9, 125.1, 121.1, 119.3, 118.1, 111.6, 55.5, 21.7. M.p. 95-96 °C. HRMS (ESI) calculated for $C_{16}H_{14}N_2O_2$ [M+H]+: 267.1128; found: 267.1133.



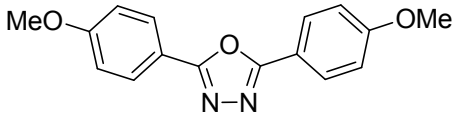

**33**

**2-(3-methoxyphenyl)-5-(*p*-tolyl)-1,3,4-oxadiazole**.[5] Yield = 23%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 8.03 (d, *J* = 8.0 Hz, 2H), 7.96 (s, 1H), 7.93 (d, *J* = 7.7 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 1H), 7.38 – 7.31 (m, 3H), 2.46 (s, 3H), 2.44 (s, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.6, 164.5, 142.2, 138.9, 132.5, 129.8, 128.9, 127.3, 126.9, 124.0, 123.9, 121.19, 2.66, 21.3. M.p. 83-85 °C. HRMS (ESI) calculated for $C_{16}H_{14}N_2O$ [M+H]+: 251.1179; found: 251.1175.



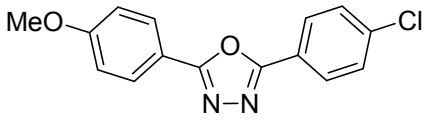

**34**

**2-(4-methoxyphenyl)-5-(p-tolyl)-1,3,4-oxadiazole**.[3] Yield = 83%; white solid; $^{1}$H NMR (600 MHz, DMSO-$d_6$) δ 8.07 – 7.99 (m, 2H), 7.96 (d, $J$ = 8.0 Hz, 2H), 7.39 (d, $J$ = 7.9 Hz, 2H), 7.22 – 7.08 (m, 2H), 3.84 (s, 3H), 2.38 (s, 3H). $^{13}$C NMR (151 MHz, DMSO) δ 164.1, 164.1, 162.5, 142.4, 130.3, 128.9, 126.9, 121.2, 116.2, 115.3, 55.9, 21.6. M.p. 135-136 $^{o}$C. HRMS (ESI) calculated for $C_{16}H_{14}N_2O_2$ [M+H]+: 267.1129; found: 267.1135.



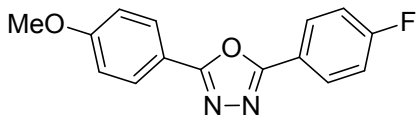

**35**

**2,5-bis(4-methoxyphenyl)-1,3,4-oxadiazole**. [3]Yield = 80%; white solid; $^{1}$H NMR (600 MHz, Chloroform-*d*) δ 8.04 (d, $J$ = 8.9 Hz, 4H), 7.01 (d, $J$ = 8.9 Hz, 4H), 3.87 (s, 6H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.0, 162.2, 128.5, 116.6, 114.5, 55.5. M.p. 173-175 $^{o}$C. HRMS (ESI) calculated for $C_{16}H_{14}N_2O_3$ [M+H]+: 283.1078; found: 283.1085.



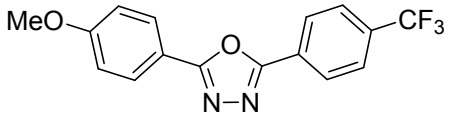

**36**

**2-(4-chlorophenyl)-5-(4-methoxyphenyl)-1,3,4-oxadiazole**.[3] Yield = 77%; white solid; $^{1}$H NMR (600 MHz, Chloroform-*d*) δ 8.08 – 8.00 (m, 4H), 7.53 – 7.44 (m, 2H), 7.05 – 6.96 (m, 2H), 3.87 (s, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.6, 163.3, 162.5, 137.7, 129.4, 128.7, 128.0, 122.6, 116.2, 114.5, 55.5. M.p. 163-164 $^{o}$C. HRMS (ESI) calculated for $C_{15}H_{11}ClN_2O_2$ [M+H]+: 287.0582; found: 287.0588.



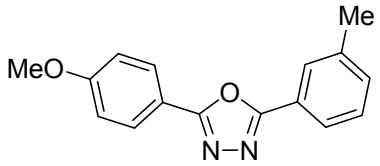

**37**

**2-(4-fluorophenyl)-5-(4-methoxyphenyl)-1,3,4-oxadiazole**.[3] Yield = 87%; $^{1}$H NMR (600 MHz, Chloroform-*d*) δ 8.15 – 8.08 (m, 2H), 8.07 – 8.02 (m, 2H), 7.21 (t, $J$ = 8.6 Hz, 2H), 7.02 (d, $J$ = 8.8 Hz, 2H), 3.88 (s, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.7 (d, $J$ = 252.2 Hz), 163.9 (d, $J$ = 187.2 Hz), 162.3, 129.0 (d, $J$ = 9.1 Hz), 128.6, 120.43, 116.4, 116.3, 116.3, 114.5, 55.5. $^{19}$F NMR (565 MHz, $CDCl_3$) δ -107.20. M.p. 172-173 $^{o}$C. HRMS (ESI) calculated for $C_{15}H_{11}FN_2O_2$ [M+H]+: 271.0878; found: 271.0882.



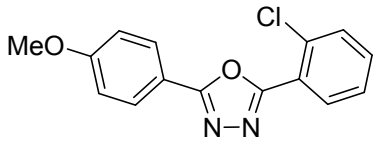

**38**

**2-(4-methoxyphenyl)-5-(4-(trifluoromethyl)phenyl)-1,3,4-oxadiazole**.[8] Yield = 73%; white solid; $^{1}$H NMR (600 MHz, Chloroform-*d*) δ 8.28 – 8.21 (m, 2H), 8.13 – 8.04 (m, 2H), 7.78 (d, *J* = 8.2 Hz, 2H), 7.08 – 6.95 (m, 2H), 3.89 (s, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 165.1, 162.9, 162.6, 133.1 (q, *J* = 33.2 Hz), 128.8, 127.3, 127.1, 126.0 (q, *J* = 3.0 Hz), 123.6 (q, *J* = 273.3 Hz), 116.0, 114.6, 55.5. $^{19}$F NMR (565 MHz, $CDCl_3$) δ -63.06. M.p. 176-177 °C. HRMS (ESI) calculated for $C_{16}H_{11}F_3N_2O_2$ [M+H]+: 321.0846; found: 321.0850.



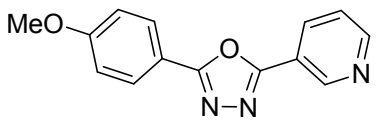

**39**

**2-(4-methoxyphenyl)-5-(m-tolyl)-1,3,4-oxadiazole**.[3] Yield = 79%; white solid; $^{1}$H NMR (600 MHz, Chloroform-*d*) δ 8.09 – 8.01 (m, 2H), 7.92 (s, 1H), 7.89 (d, *J* = 7.8 Hz, 1H), 7.38 (t, *J* = 7.6 Hz, 1H), 7.32 (d, *J* = 7.6 Hz, 1H), 7.03 – 6.98 (m, 2H), 3.86 (s, 3H), 2.43 (s, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.4, 164.2, 162.3, 138.9, 132.3, 128.9, 128.6, 127.3, 123.9, 123.9, 116.5, 114.5, 55.5, 21.3. M.p. 129-130 °C. HRMS (ESI) calculated for $C_{16}H_{14}N_2O_2$ [M+H]+: 267.1129; found: 267.1135.




**40**

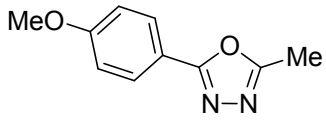
**2-(2-chlorophenyl)-5-(4-methoxyphenyl)-1,3,4-oxadiazole**. Yield = 62%; white solid; $^{1}$H NMR (600 MHz, Chloroform-*d*) δ 8.05 (t, *J* = 1.8 Hz, 1H), 8.05 – 8.00 (m, 2H), 7.97 (dt, *J* = 7.5, 1.5 Hz, 1H), 7.50 – 7.45 (m, 1H), 7.43 (t, *J* = 7.8 Hz, 1H), 7.03 – 6.96 (m, 2H), 3.85 (s, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.7, 162.9, 162.5, 135.1, 131.5, 130.3, 128.7, 126.7, 125.7, 124.8, 116.1, 114.5, 55.5. M.p. 125-126 °C. HRMS (ESI) calculated for $C_{15}H_{11}ClN_2O_2$ [M+H]+: 287.0582; found: 287.0588.




**41**

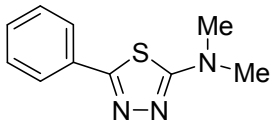
**2-(4-methoxyphenyl)-5-(pyridin-3-yl)-1,3,4-oxadiazole**.[9] Yield = 85%; white solid;

$^1$H NMR (600 MHz, DMSO-$d_6$) δ 8.12 (t, $J$ = 1.9 Hz, 1H), 8.11 – 8.01 (m, 3H), 7.70 (ddd, $J$ = 8.1, 2.1, 1.0 Hz, 1H), 7.64 (t, $J$ = 7.9 Hz, 1H), 7.21 – 7.08 (m, 2H), 3.86 (s, 3H). $^{13}$C NMR (151 MHz, DMSO) δ 164.7, 162.8, 162.7, 134.5, 132.1, 131.8, 129.2, 126.5, 125.8, 125.6, 115.9, 115.3, 56.0. M.p. 145-146 °C. HRMS (ESI) calculated for $C_{14}H_{11}N_3O_2$ [M+H]+: 254.0925; found: 254.0930.



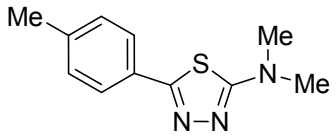

**42**

**2-(4-methoxyphenyl)-5-methyl-1,3,4-oxadiazole**.[3] Yield = 87%; white solid; $^1$H NMR (600 MHz, Chloroform-*d*) δ 7.91 (d, $J$ = 8.8 Hz, 2H), 7.14 – 6.78 (m, 2H), 3.83 (s, 3H), 2.55 (s, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.7, 163.1, 162.1, 128.3, 116.5, 114.3, 55.3, 11.0. M.p. 71-72 °C. HRMS (ESI) calculated for $C_{10}H_{10}N_2O_2$ [M+H]+: 191.0816; found: 191.0821.



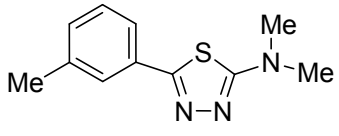

**44**

***N,N*-dimethyl-5-phenyl-1,3,4-thiadiazol-2-amine**.[10] Yield = 87%; white solid; $^1$H NMR (500 MHz, Chloroform-*d*) δ 7.87 – 7.75 (m, 2H), 7.47 – 7.31 (m, 3H), 3.32 – 3.13 (m, 6H). 13C NMR (126 MHz, CDCl3) δ 171.6, 157.8, 131.3, 129.5, 128.8, 126.6, 41.5. M.p. 101-102 °C. HRMS (ESI) calculated for $C_{10}H_{11}N_3S$ [M+H]+: 206.0746; found: 206.0745.



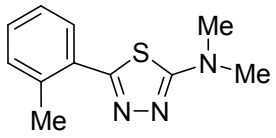

**45**

***N,N*-dimethyl-5-(p-tolyl)-1,3,4-thiadiazol-2-amine**.[11] Yield = 85%; white solid; $^1$H NMR (500 MHz, Chloroform-*d*) δ 7.70 (d, $J$ = 7.7 Hz, 2H), 7.23 (d, $J$ = 7.8 Hz, 2H), 3.20 (d, $J$ = 1.7 Hz, 6H), 2.39 (s, 3H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 171.3, 158.1, 139.7, 129.5, 128.6, 126.6, 41.5, 21.3. M.p. 131-132 °C. HRMS (ESI) calculated for $C_{11}H_{13}N_3S$ [M+H]+: 220.0903; found: 220.0900.



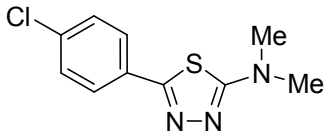

**46**

***N,N*-dimethyl-5-(m-tolyl)-1,3,4-thiadiazol-2-amine**.[11] Yield = 79%; white solid; $^1$H NMR (500 MHz, Chloroform-*d*) δ 7.66 (s, 1H), 7.61 (d, *J* = 7.8 Hz, 1H), 7.37 – 7.25 (m, 1H), 7.21 (d, *J* = 7.7 Hz, 1H), 3.22 (s, 6H), 2.41 (s, 3H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 171.5, 158.1, 138.6, 131.2, 130.3, 128.7, 127.3, 123.9, 41.5, 21.3. M.p. 90-92 $^o$C. HRMS (ESI) calculated for $C_{11}H_{13}N_3S$ [M+H]+: 220.0903; found: 220.0909.



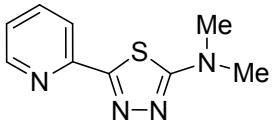

**47**

***N,N*-dimethyl-5-(o-tolyl)-1,3,4-thiadiazol-2-amine**.[11] Yield = 71%; white solid; $^1$H NMR (500 MHz, Chloroform-*d*) δ 7.55 (d, *J* = 7.5 Hz, 1H), 7.33 – 7.16 (m, 3H), 3.22 (s, 6H), 2.60 (s, 3H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 172.0, 157.2, 136.9, 131.4, 130.1, 130.0, 129.2, 125.9, 41.5, 21.6. M.p. 52-53 $^o$C. HRMS (ESI) calculated for $C_{11}H_{13}N_3S$ [M+H]+: 220.0903; found: 220.0903.



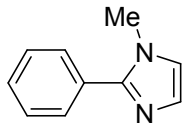

**48**

**5-(4-chlorophenyl)-*N,N*-dimethyl-1,3,4-thiadiazol-2-amine**.[11] Yield = 63%; white solid; $^1$H NMR (500 MHz, Chloroform-*d*) δ 7.85 – 7.67 (m, 2H), 7.41 – 7.32 (m, 2H), 3.20 (d, *J* = 1.5 Hz, 6H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 171.7, 156.5, 135.3, 129.8, 129.1, 127.7, 41.5. M.p. 160-161 $^o$C. HRMS (ESI) calculated for $C_{10}H_{10}ClN_3S$ [M+H]+: 240.0357; found: 240.0355.



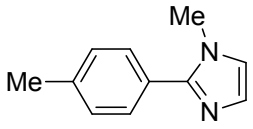

**49**

***N,N*-dimethyl-5-(pyridin-2-yl)-1,3,4-thiadiazol-2-amine**. Yield = 76%; white solid; $^1$H NMR (500 MHz, Chloroform-*d*) δ 8.54 (d, *J* = 4.8 Hz, 1H), 8.17 (d, *J* = 8.0 Hz, 1H), 7.74 (t, *J* = 7.7 Hz, 1H), 7.28 – 7.21 (m, 1H), 3.21 (s, 6H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 172.97, 159.7, 150.2, 149.2, 136.7, 123.7, 119.6, 41.3. M.p. 79-80 $^o$C. HRMS (ESI) calculated for $C_9H_{10}N_4S$ [M+H]+: 207.0699; found: 207.0695.



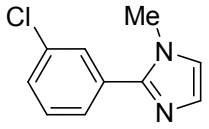

**51**

**1-methyl-2-phenyl-1H-imidazole**.[12] Yield = 82%; colorless oil; $^1$H NMR (500 MHz, Chloroform-*d*) δ 7.68 – 7.61 (m, 2H), 7.50 – 7.43 (m, 2H), 7.43 (m, 1H), 7.13 (s, 1H), 6.98 (s, 1H), 3.75 (s, 3H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 147.8, 130.5, 128.6, 128.5, 128.2, 127.4, 122.2, 34.4. HRMS (ESI) calculated for $C_{10}H_{10}N_2$ [M+H]+: 159.0917; found: 159.0915.



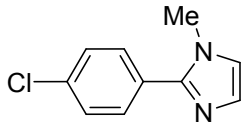

**52**

**1-methyl-2-(*p*-tolyl)-1H-imidazole**.[13] Yield = 80%; colorless oil; $^1$H NMR (500 MHz, Chloroform-*d*) δ 7.67 – 7.42 (m, 2H), 7.28 (d, *J* = 7.0 Hz, 2H), 7.10 (d, *J* = 1.8 Hz, 1H), 6.96 (d, *J* = 1.6 Hz, 1H), 3.75 (d, *J* = 1.4 Hz, 3H), 2.42 (s, 3H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 147.9, 138.5, 129.1, 128.5, 128.2, 127.7, 122.1, 34.4, 21.3. HRMS (ESI) calculated for $C_{11}H_{12}N_2$ [M+H]+: 173.1073; found: 173.1073.



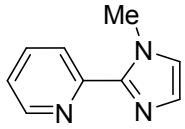

**53**

**2-(3-chlorophenyl)-1-methyl-1H-imidazole**.[14] Yield = 73%; colorless oil; $^1$H NMR (500 MHz, Chloroform-*d*) δ 7.64 (s, 1H), 7.54 – 7.48 (m, 1H), 7.38 (d, *J* = 4.6 Hz, 2H), 7.11 (s, 1H), 6.97 (s, 1H), 3.75 (s, 3H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 146.3, 134.5, 132.3, 129.7, 128.6, 128.6, 126.6, 122.8, 34.5. HRMS (ESI) calculated for $C_{10}H_9ClN_2$ [M+H]+: 193.0527; found: 193.0525.



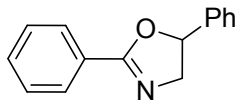

**55**

**2-(4-chlorophenyl)-1-methyl-1H-imidazole**.[15] Yield = 69%; colorless oil; $^1$H NMR (500 MHz, Chloroform-*d*) δ 7.58 (d, *J* = 8.1 Hz, 2H), 7.44 (d, *J* = 8.0 Hz, 2H), 7.14 (s, 1H), 6.98 (s, 1H), 3.74 (s, 3H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 146.6, 134.7, 129.8, 129.0, 128.7, 128.5, 122.6, 34.5. HRMS (ESI) calculated for $C_{10}H_9ClN_2$ [M+H]+: 193.0527; found: 193.0530.



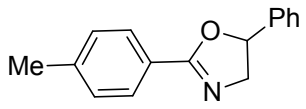
**56**

**2-(1-methyl-1H-imidazol-2-yl)pyridine**.[16] Yield = 77%; colorless oil; $^1$H NMR (500 MHz, Chloroform-*d*) δ 8.60 (d, *J* = 4.8 Hz, 1H), 8.25 – 8.06 (m, 1H), 7.77 (td, *J* = 7.7, 1.6 Hz, 1H), 7.27 – 7.20 (m, 1H), 7.14 (s, 1H), 6.99 (s, 1H), 4.15 (s,3H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 150.7, 148.2, 144.9, 136.5, 128.1, 124.3, 122.6, 122.3, 36.3. HRMS (ESI) calculated for $C_9H_9N_3$ [M+H]+: 160.0869; found: 160.0870.



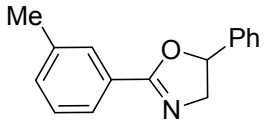
**58**

**2,5-diphenyl-4,5-dihydrooxazole**.[17] Yield = 85%; colorless oil; $^1$H NMR (500 MHz, Chloroform-*d*) δ 8.11 – 8.03 (m, 2H), 7.56 – 7.49 (m, 1H), 7.50 – 7.43 (m, 2H), 7.45 – 7.32 (m, 5H), 5.69 (dd, *J* = 10.2, 7.9 Hz, 1H), 4.51 (dd, *J* = 14.8, 10.2 Hz, 1H), 4.03 (dd, *J* = 14.8, 7.9 Hz, 1H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 164.0, 141.1, 131.5, 128.8, 128.4, 128.3, 128.3, 127.6, 125.7, 81.1, 63.2. HRMS (ESI) calculated for $C_{15}H_{13}NO$ [M+H]+: 224.1070; found: 224.1075.



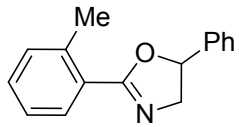
**59**

**5-phenyl-2-(*p*-tolyl)-4,5-dihydrooxazole**.[18] Yield = 81%; colorless oil; $^1$H NMR (600 MHz, Chloroform-*d*) δ 7.96 (s, 2H), 7.38 (td, *J* = 12.3, 10.5, 7.2 Hz, 5H), 7.31 – 7.23 (m, 2H), 5.67 (dd, *J* = 10.2, 7.9 Hz, 1H), 4.50 (dd, *J* = 14.7, 10.1 Hz, 1H), 4.01 (dd, *J* = 14.6, 7.8 Hz, 1H), 2.43 (s, 3H). $^{13}$C NMR (151 MHz, $CDCl_3$) δ 164.1, 141.9, 141.2, 129.2, 128.8, 128.3, 128.3, 125.7, 124.9, 80.9, 63.2, 21.6. HRMS (ESI) calculated for $C_{16}H_{15}NO$ [M+H]+: 238.1226; found: 238.1227.



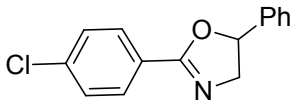
**60**

**5-phenyl-2-(*m*-tolyl)-4,5-dihydrooxazole**. Yield = 77%; colorless oil; $^1$H NMR (500 MHz, Chloroform-*d*) δ 7.89 (s, 1H), 7.88 – 7.83 (m, 1H), 7.38 (m, 7H), 5.68 (dd, *J* = 10.0, 7.8 Hz, 1H), 4.50 (dd, *J* = 14.8, 10.1 Hz, 1H), 4.02 (dd, *J* = 14.7, 7.9 Hz, 1H), 2.42 (s, 3H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 164.2, 141.1 138.1, 132.2, 128.8, 128.8, 128.3, 128.2, 127.5, 125.7, 125.4, 81.0, 63.2, 21.3. HRMS (ESI) calculated for $C_{16}H_{15}NO$ [M+H]+: 238.1226; found: 238.1226.



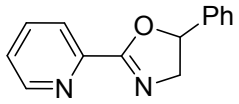

**61**

**5-phenyl-2-(*o*-tolyl)-4,5-dihydrooxazole**. Yield = 53%; colorless oil; $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.93 (d, *J* = 7.7 Hz, 1H), 7.40 (m, 6H), 7.33 – 7.20 (m, 2H), 5.64 (dd, *J* = 10.2, 8.1 Hz, 1H), 4.55 (ddd, *J* = 14.8, 10.2, 1.6 Hz, 1H), 4.06 (ddd, *J* = 14.8, 8.1, 1.6 Hz, 1H), 2.68 (s, 3H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 164.4, 141.3, 138.9, 131.3, 130.6, 129.9, 128.8, 128.2, 127.0, 125.7, 125.6, 80.2, 63.5, 21.9. HRMS (ESI) calculated for $C_{16}H_{15}NO$ [M+H]+: 238.1226; found: 238.1227.




**62**

**2-(4-chlorophenyl)-5-phenyl-4,5-dihydrooxazole**.[17] Yield = 71%; colorless oil; $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 7.98 (d, *J* = 8.1 Hz, 2H), 7.42 (m, 4H), 7.36 (m, 3H), 5.68 (dd, *J* = 10.1, 8.1 Hz, 1H), 4.50 (dd, *J* = 14.9, 10.1 Hz, 1H), 4.02 (dd, *J* = 14.8, 8.0 Hz, 1H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 163.1, 140.8, 137.6, 129.7, 128.8, 128.7, 128.4, 126.2, 125.7, 81.2, 63.2. HRMS (ESI) calculated for $C_{15}H_{12}ClNO$ [M+H]+: 258.0680; found: 258.0685.




**63**

**5-phenyl-2-(pyridin-2-yl)-4,5-dihydrooxazole**.[19] Yield = 80%; white solid; $^{1}$H NMR (500 MHz, Chloroform-*d*) δ 8.76 (d, *J* = 4.7 Hz, 1H), 8.10 (d, *J* = 7.9 Hz, 1H), 7.82 (t, *J* = 7.8 Hz, 1H), 7.43 (dd, *J* = 7.4, 5.0 Hz, 1H), 7.39 (m, 4H), 7.35 (q, *J* = 4.4 Hz, 1H), 5.77 (dd, *J* = 10.2, 8.2 Hz, 1H), 4.56 (dd, *J* = 15.2, 10.2 Hz, 1H), 4.10 (dd, *J* = 15.2, 8.2 Hz, 1H). $^{13}$C NMR (126 MHz, $CDCl_3$) δ 163.2, 149.8, 146.6, 140.5, 136.7, 128.8, 128.4, 125.9, 125.6, 123.9, 81.8, 63.2. M.p. 89-91 °C. HRMS (ESI) calculated for $C_{14}H_{12}N_2O$ [M+H]+: 225.1022; found: 225.1022.

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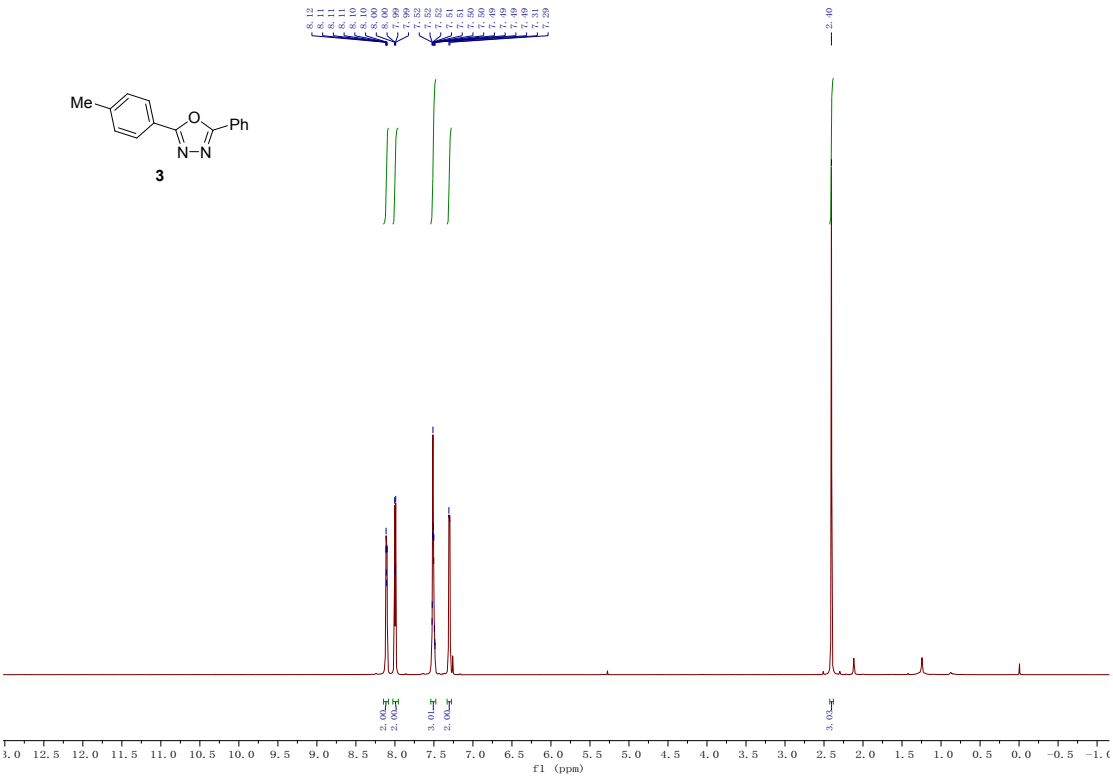
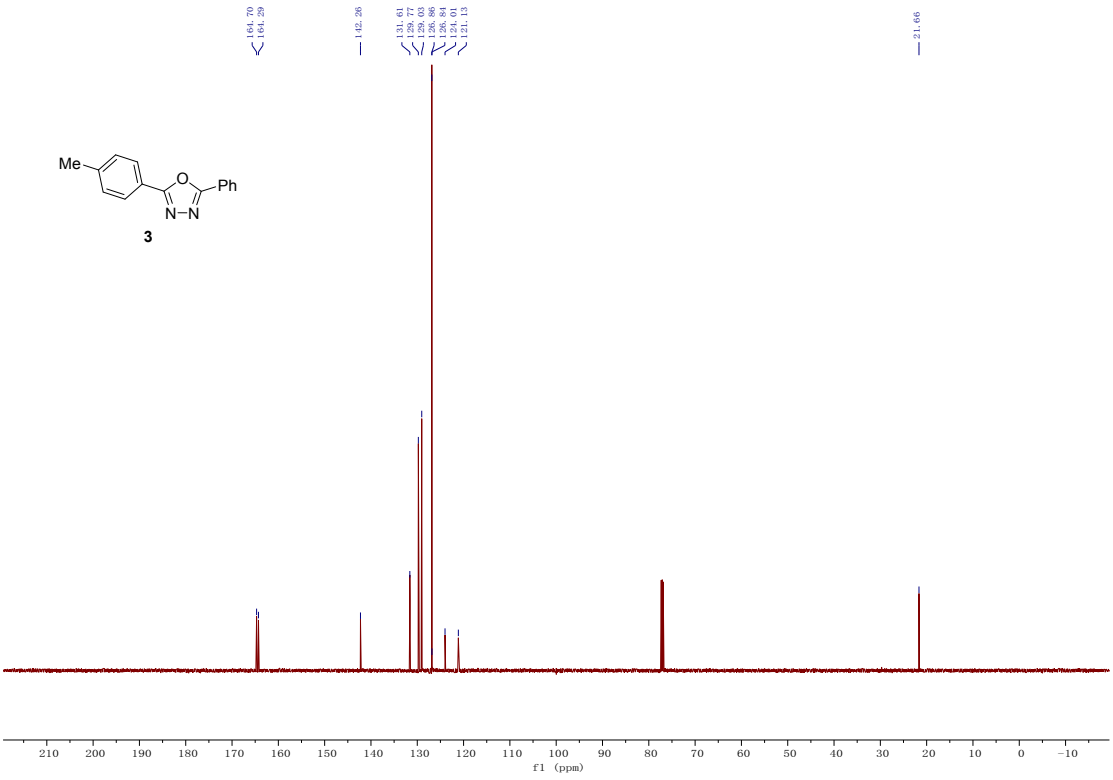
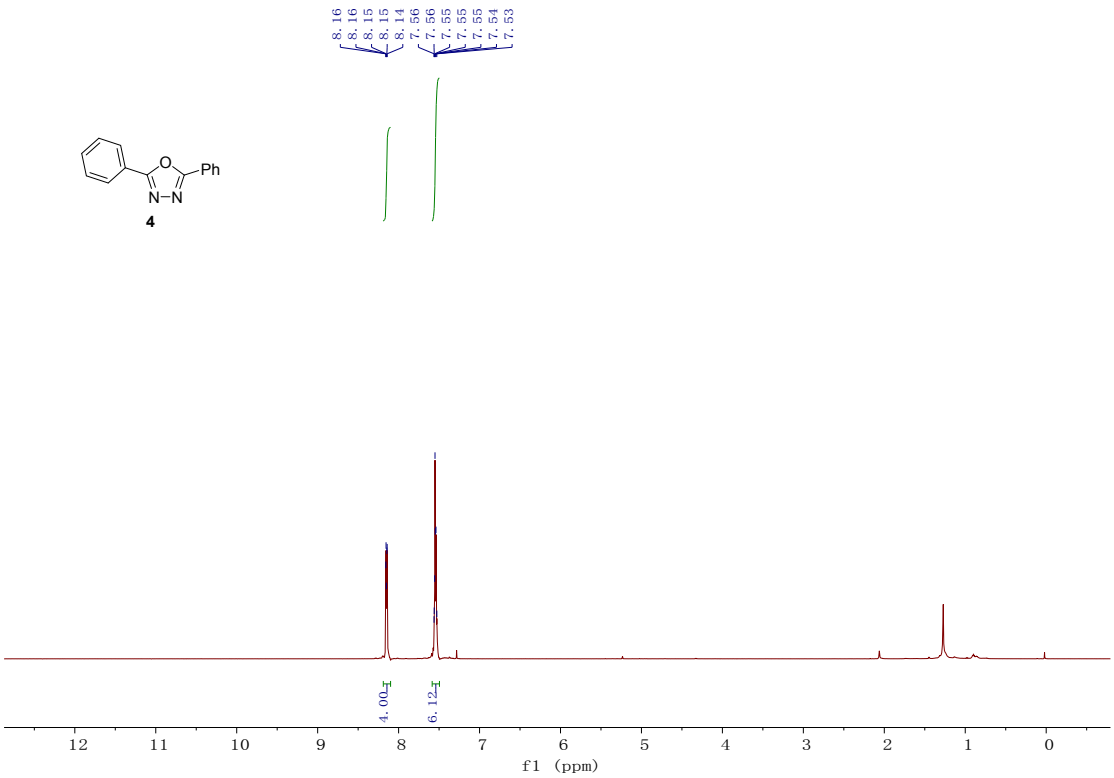
## $^{1}H$, $^{13}C$ and $^{19}F$ Spectra of all products

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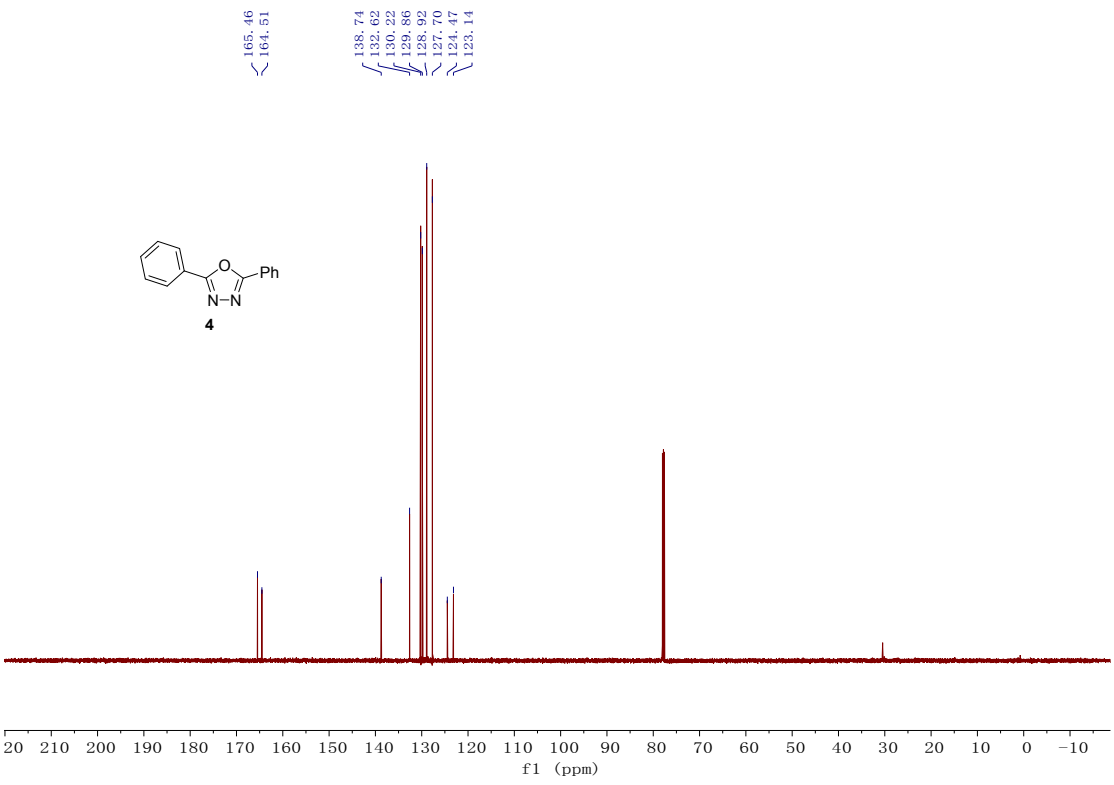
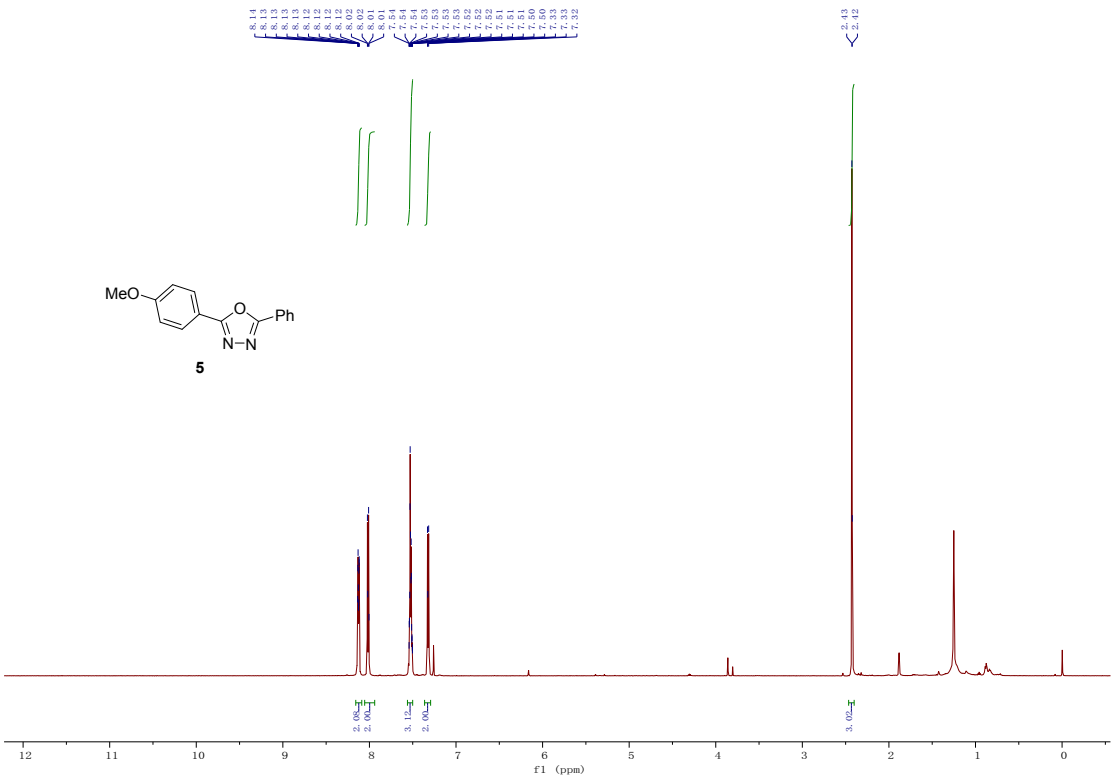
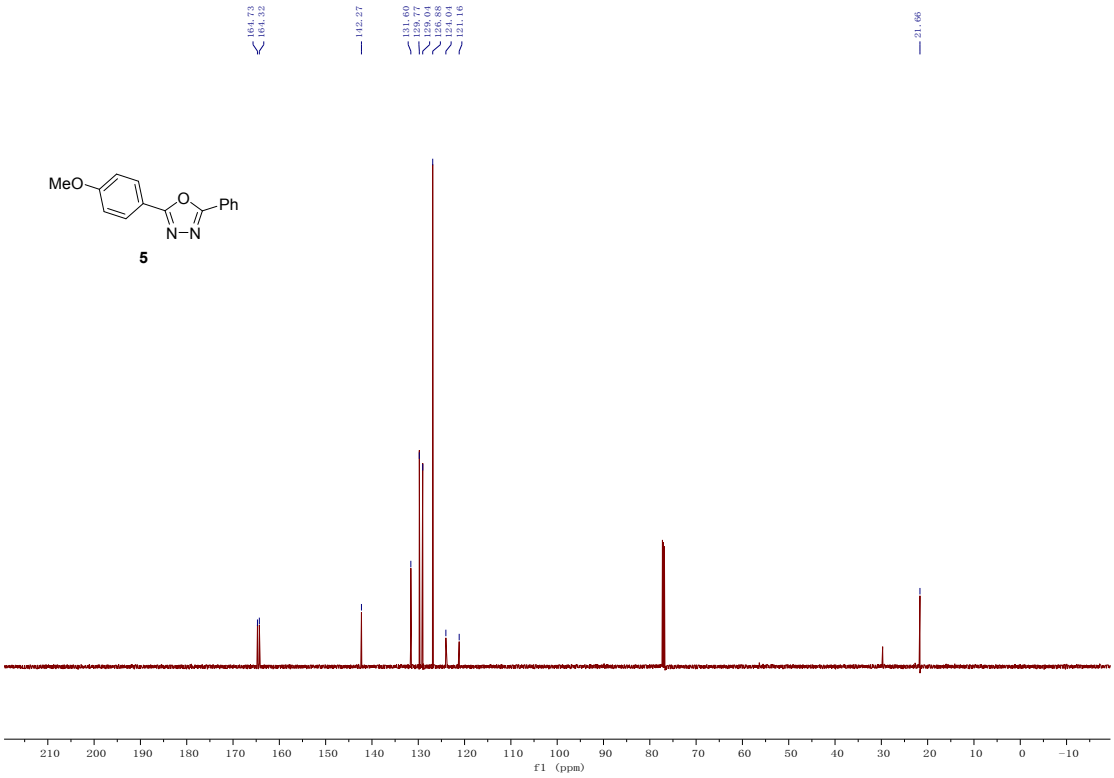
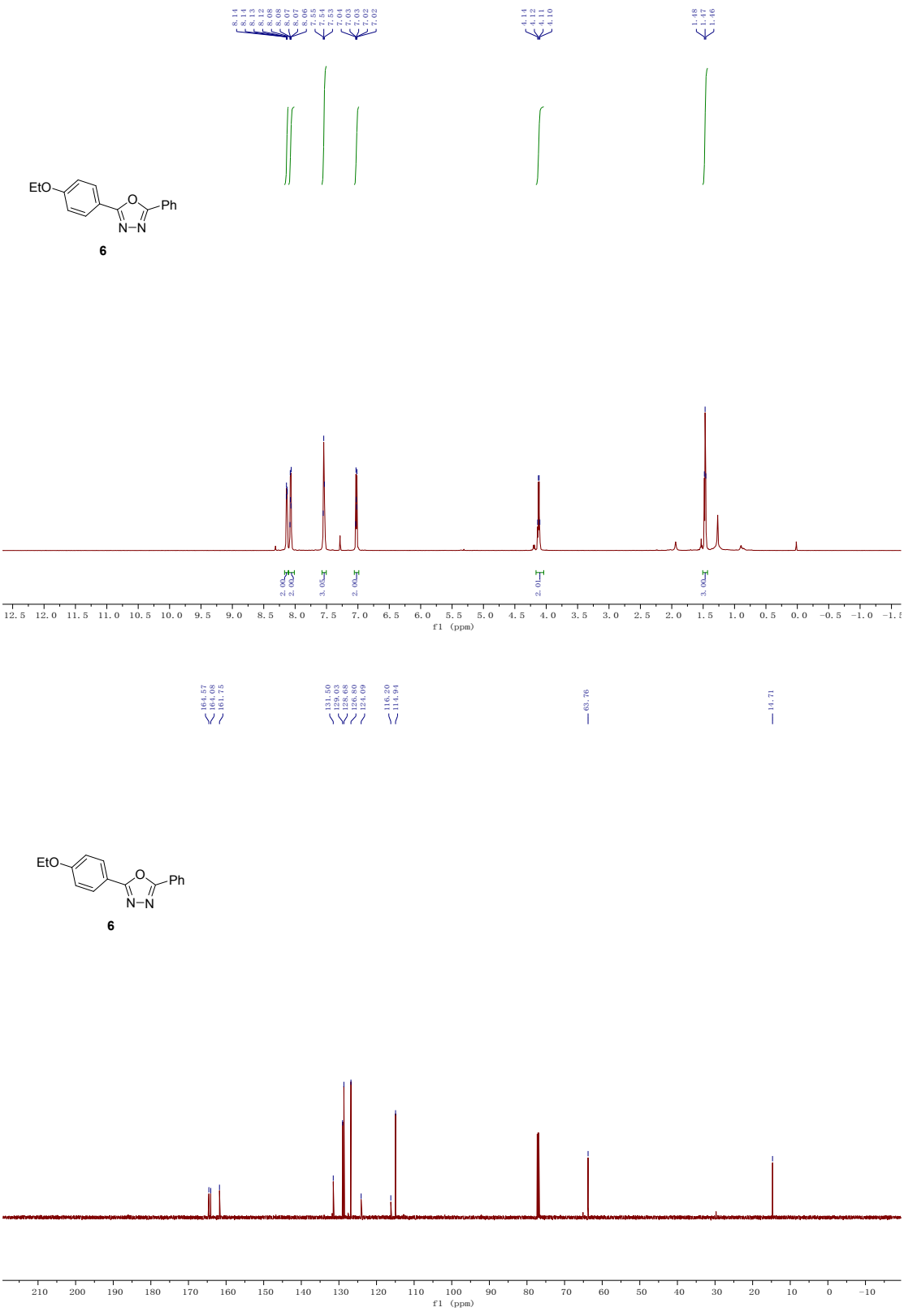
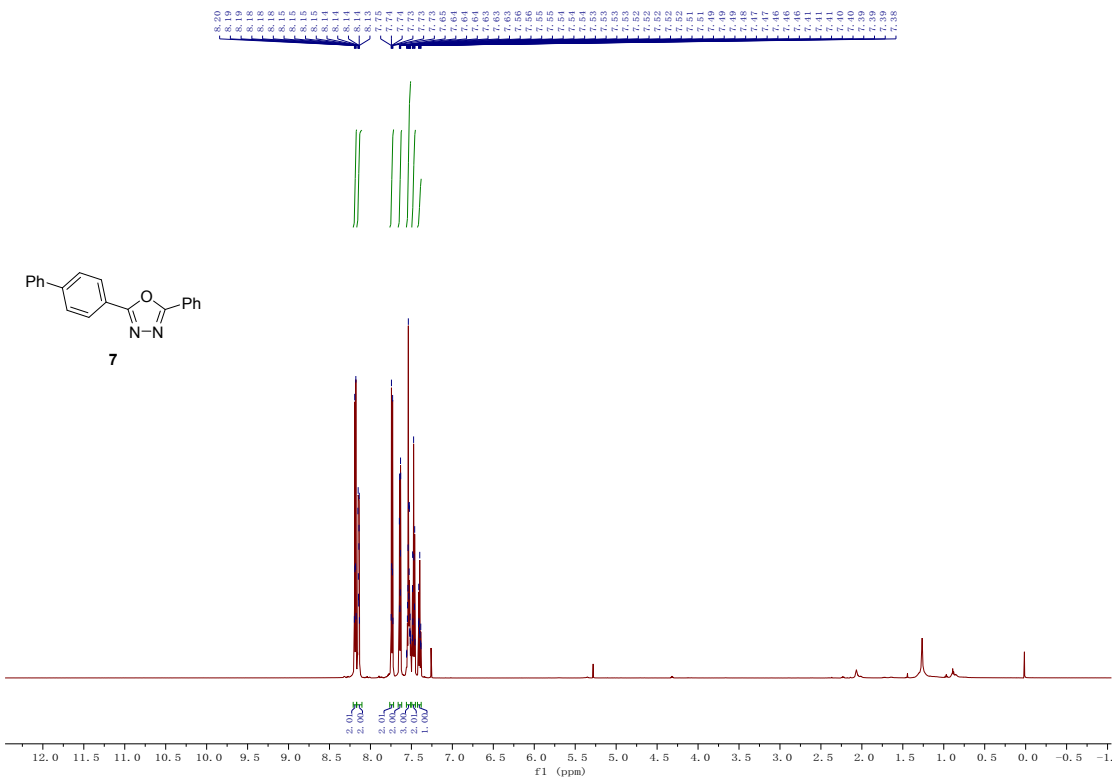
6
5
4
3
2
1
0
f1 (ppm)

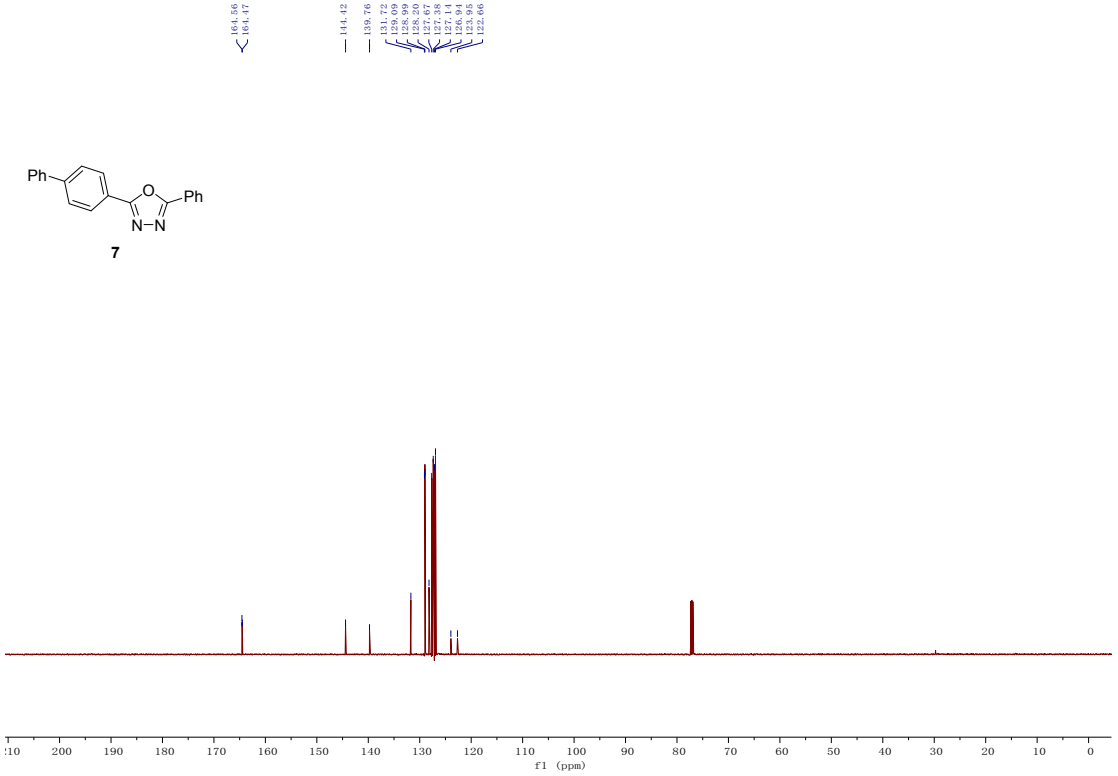

165.46
164.51
138.74
132.62
130.22
129.86
128.92
127.70
124.47
123.14
Ph
O
N–N
4
20
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

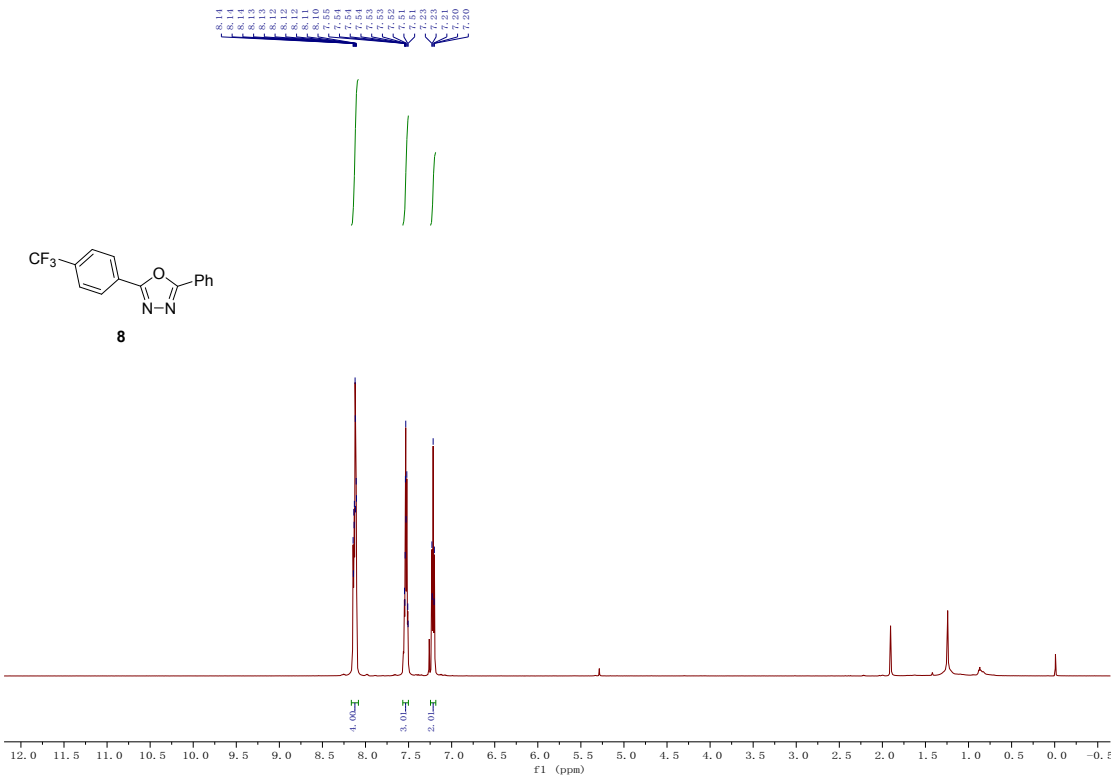

MeO
O
Ph
N–N
5
f1 (ppm)

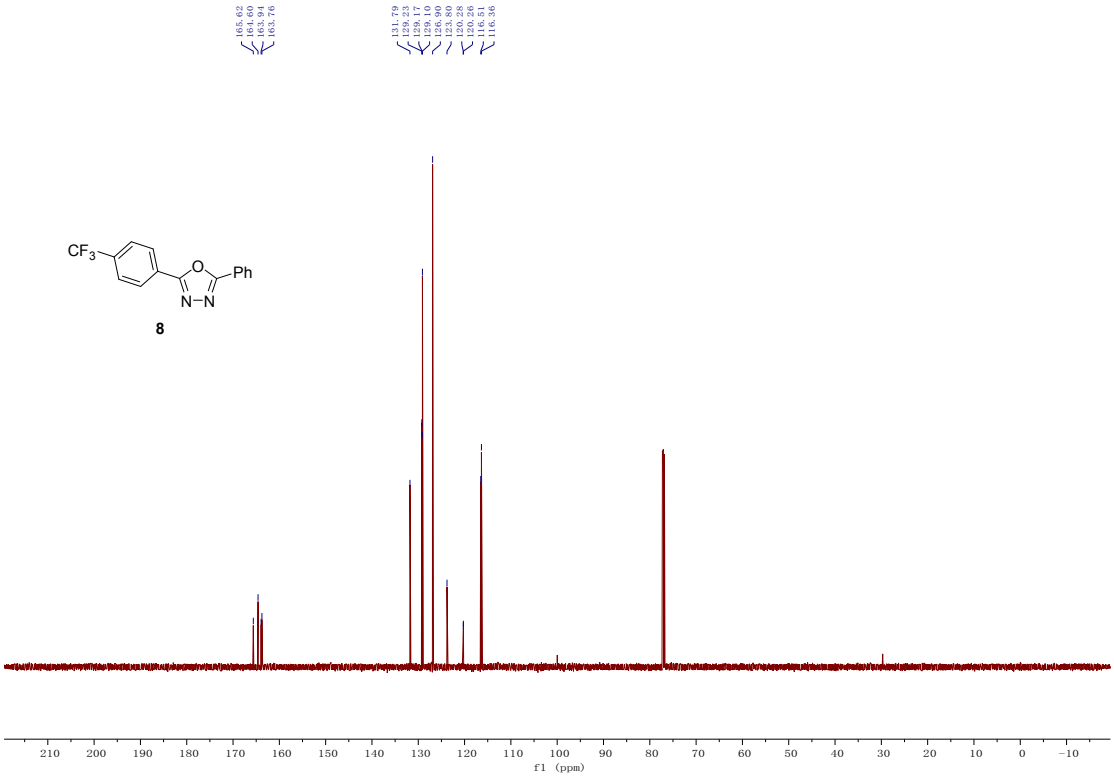

MeO
O
Ph
N–N
5
f1 (ppm)

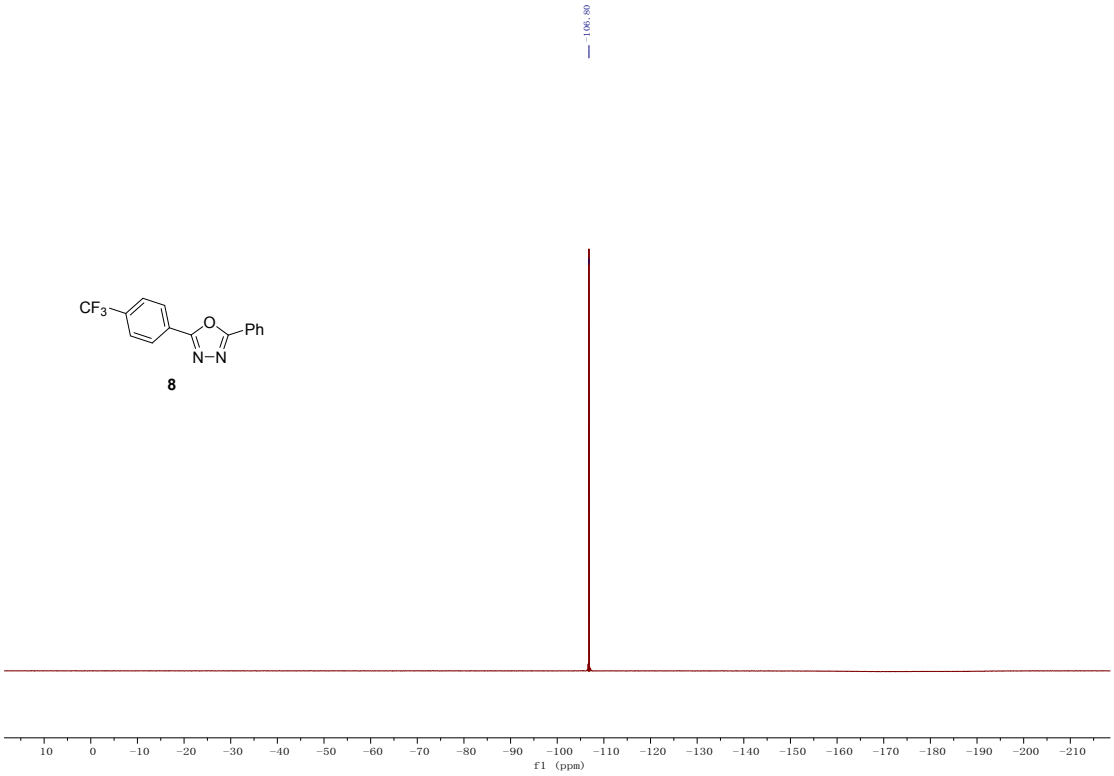

8.14
8.14
8.13
8.13
8.08
8.08
8.07
8.07
8.06
7.55
7.54
7.53
7.04
7.03
7.02
7.02
7.02
4.14
4.12
4.11
4.10
1.48
1.47
1.46
EtO
O
Ph
N–N
6
2.00
2.00
3.05
2.00
2.01
3.00
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0
f1 (ppm)
164.57
164.08
161.75
131.50
129.03
128.68
126.89
124.09
116.20
114.94
63.76
14.71
EtO
O
Ph
N–N
6
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)

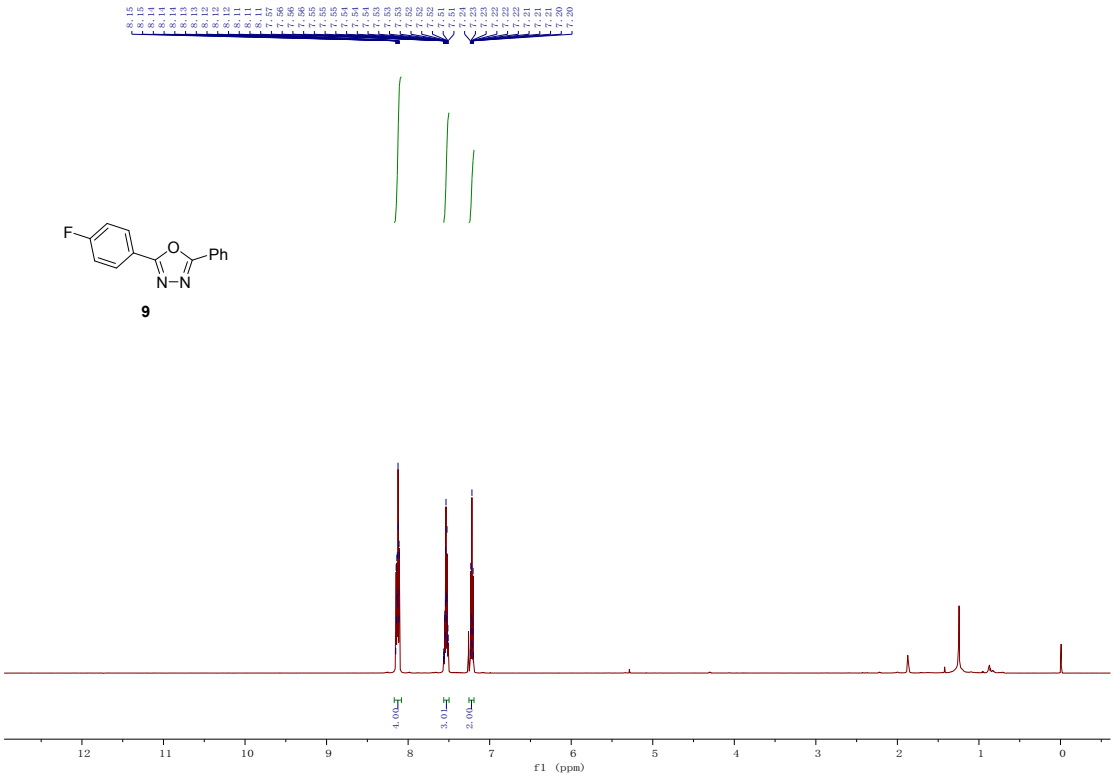

Ph
Ph
O
N–N
7
f1 (ppm)

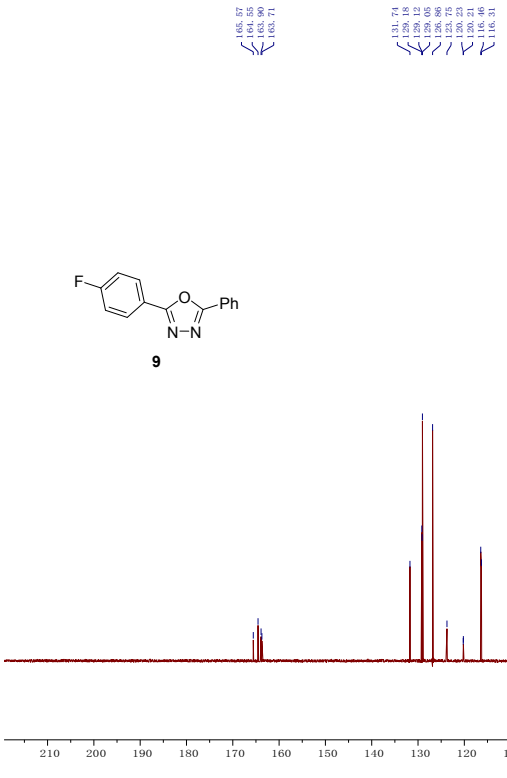

Ph
Ph
O
N–N
7
f1 (ppm)

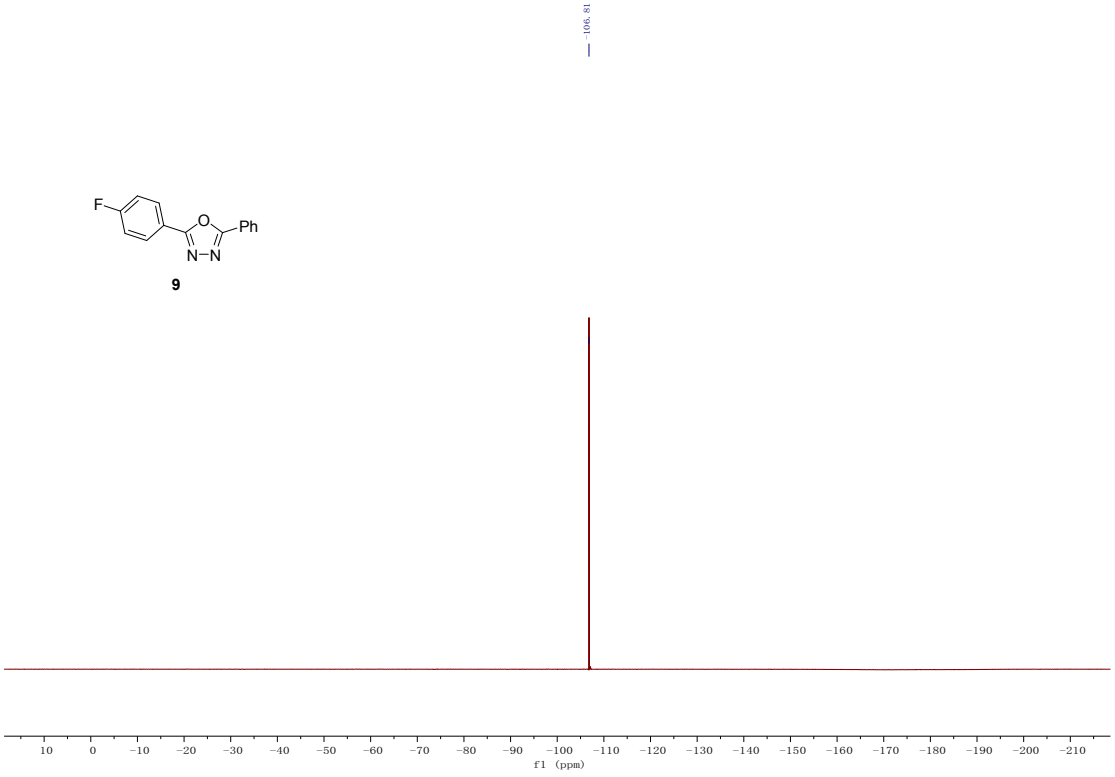

8.14
8.14
8.14
8.13
8.13
8.12
8.12
8.12
8.11
8.10
7.55
7.54
7.54
7.54
7.53
7.53
7.52
7.51
7.51
7.23
7.23
7.21
7.20
7.20
CF3
O
Ph
N–N
8
4.00
3.01
2.01
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

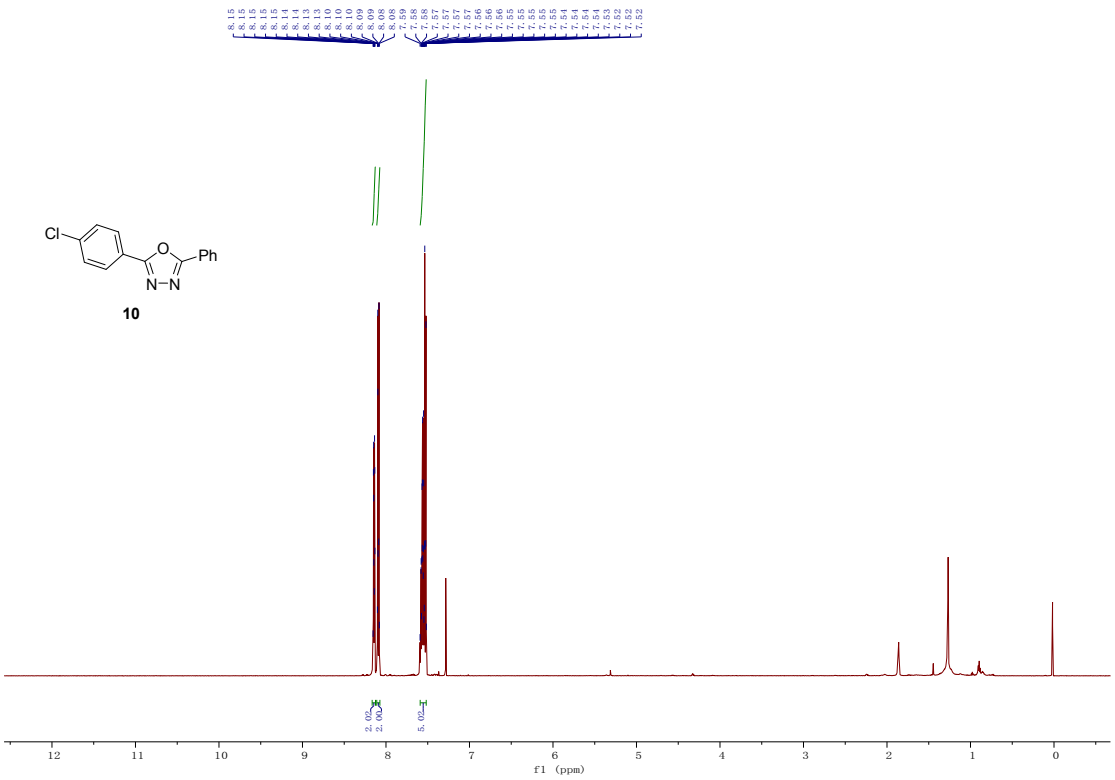

165.62
164.69
163.94
163.76
131.79
129.23
129.17
129.10
126.90
123.80
120.28
120.26
116.51
116.36
CF3
O
Ph
N–N
8
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

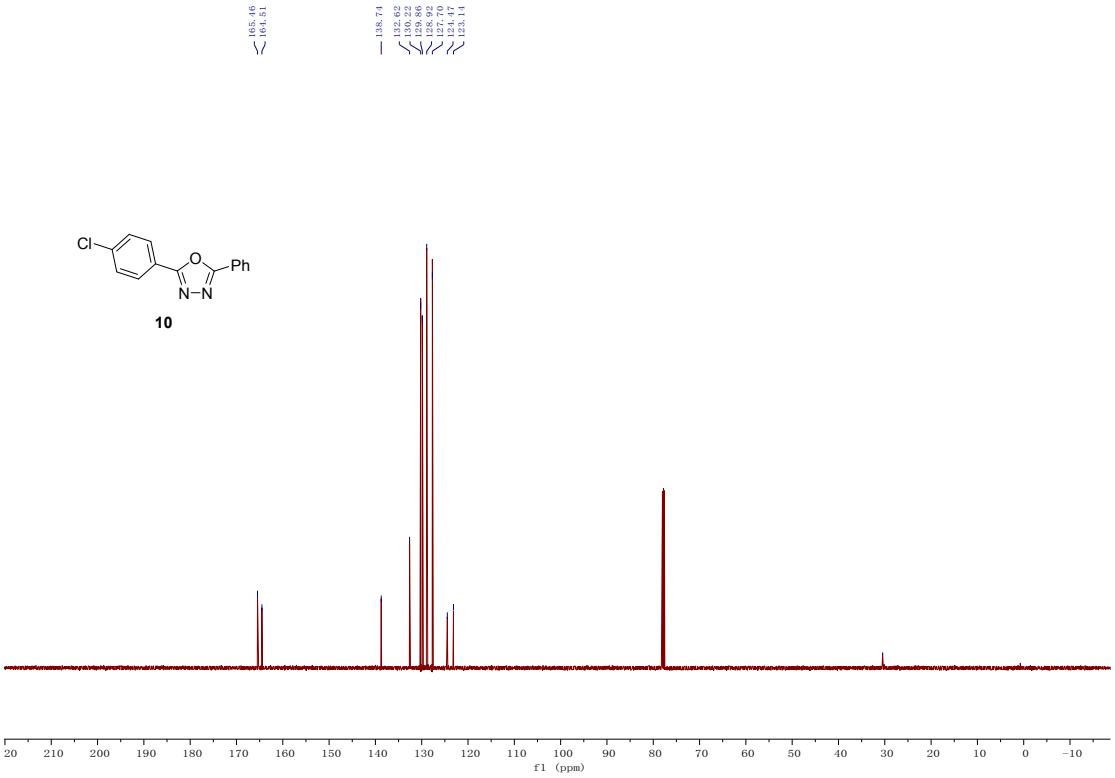

-106.80
CF3
O
Ph
N–N
8
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)

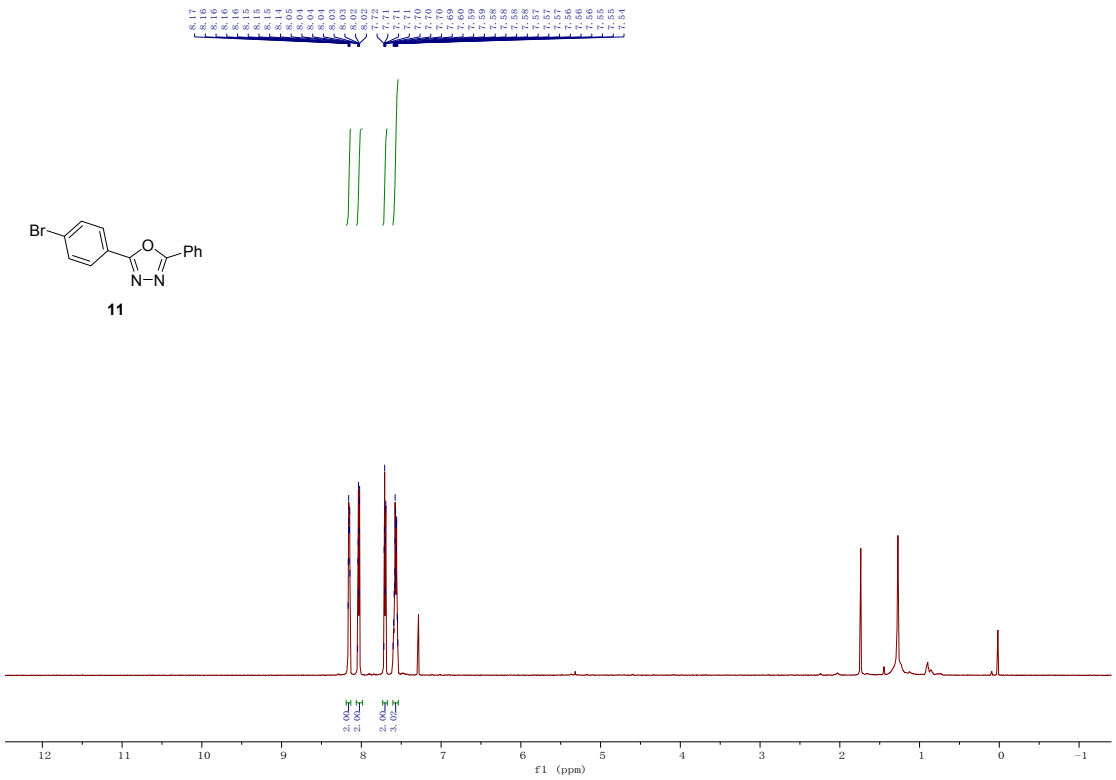

F
O
Ph
N–N
9
4.00
3.01
2.01
12 11 10 9 8 7 6 5 4 3 2 1 0
f1 (ppm)

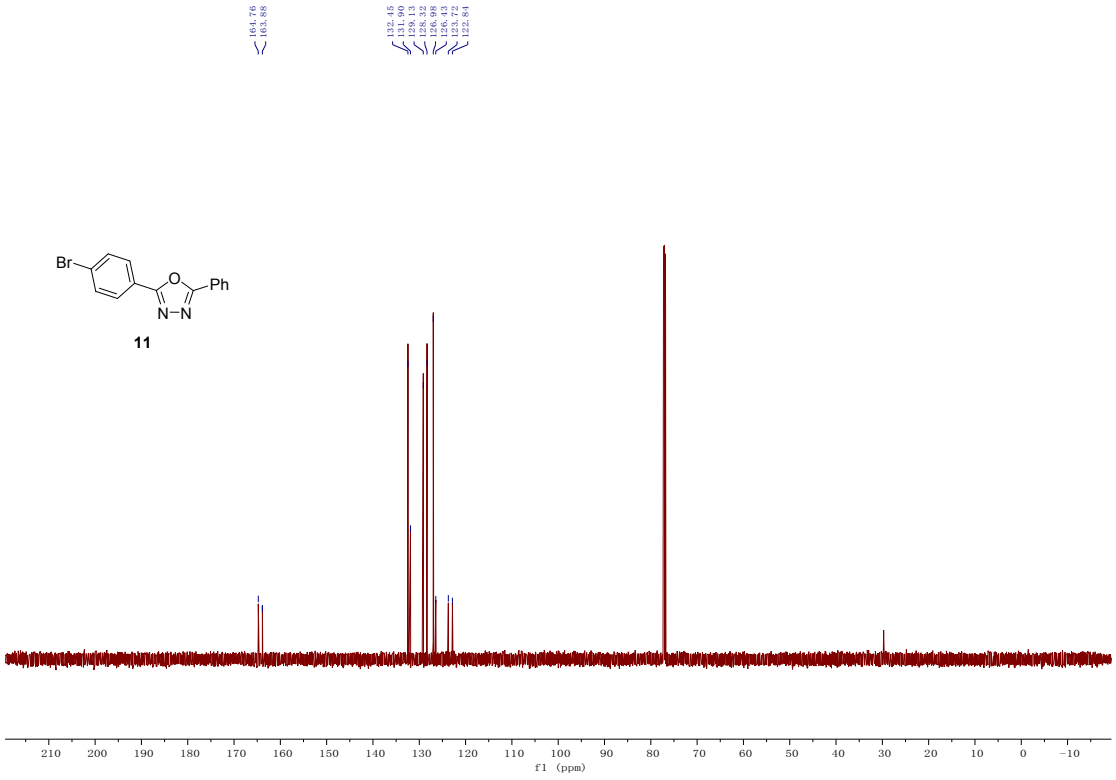

165.57
164.55
163.90
163.71
131.74
129.18
129.12
129.05
126.86
125.75
120.23
120.21
116.46
116.31
F
O
Ph
N–N
9
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

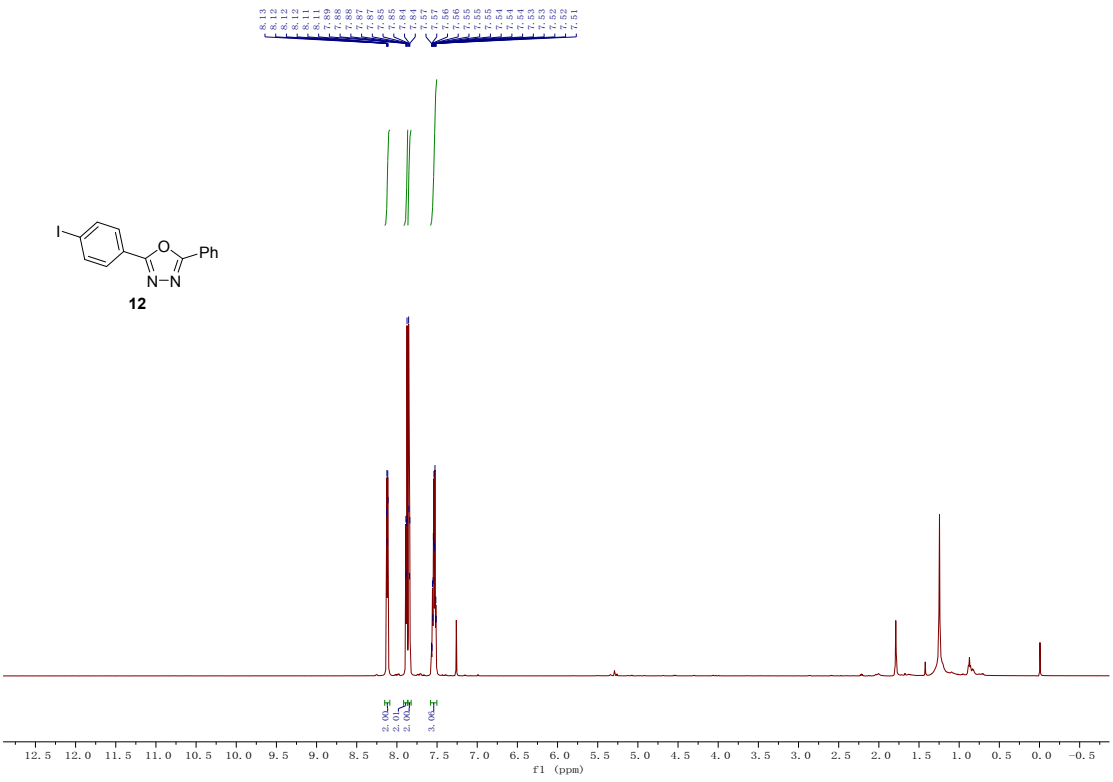

−106.81
F
O
Ph
N–N
9
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)

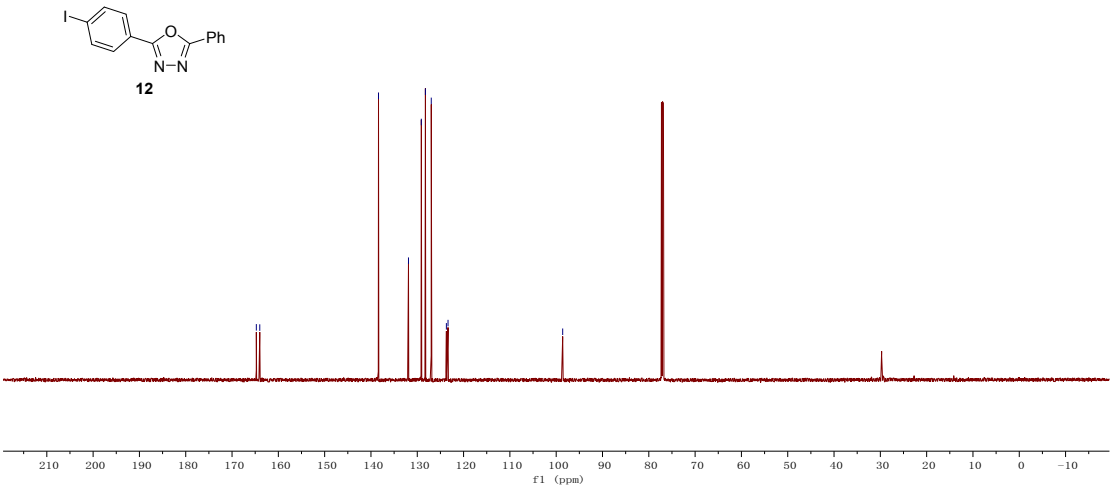

Cl
O
Ph
N–N
10
f1 (ppm)

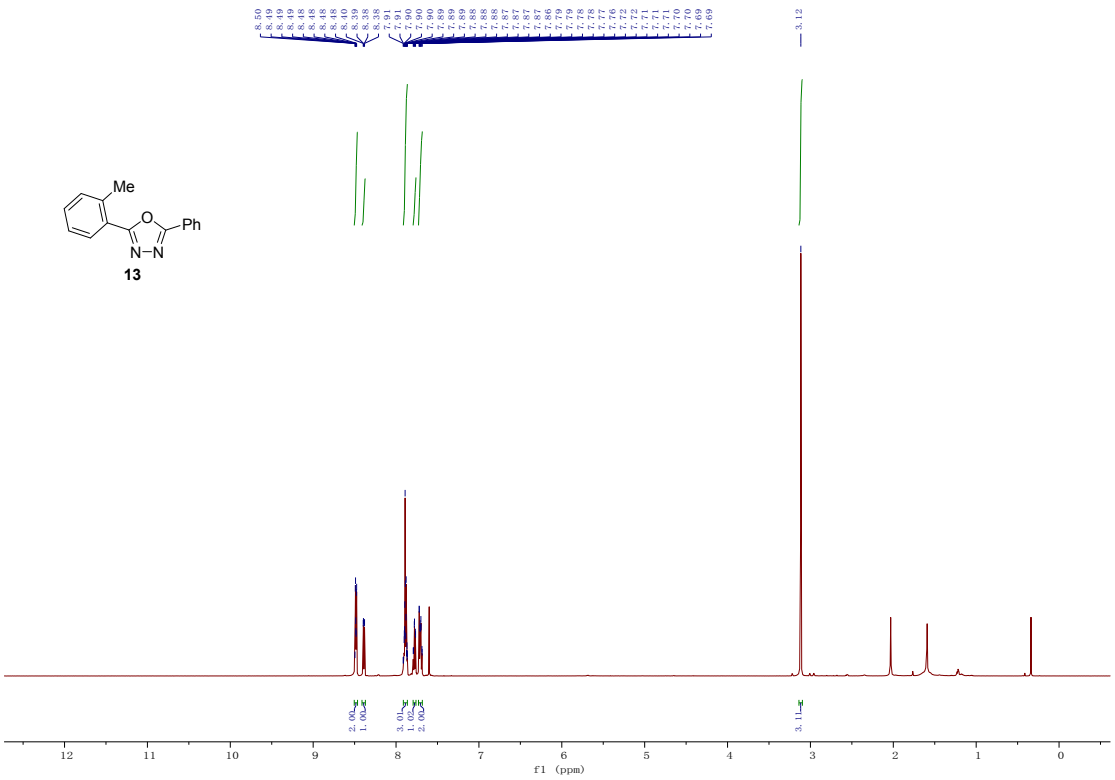

Cl
O
Ph
N–N
10
f1 (ppm)

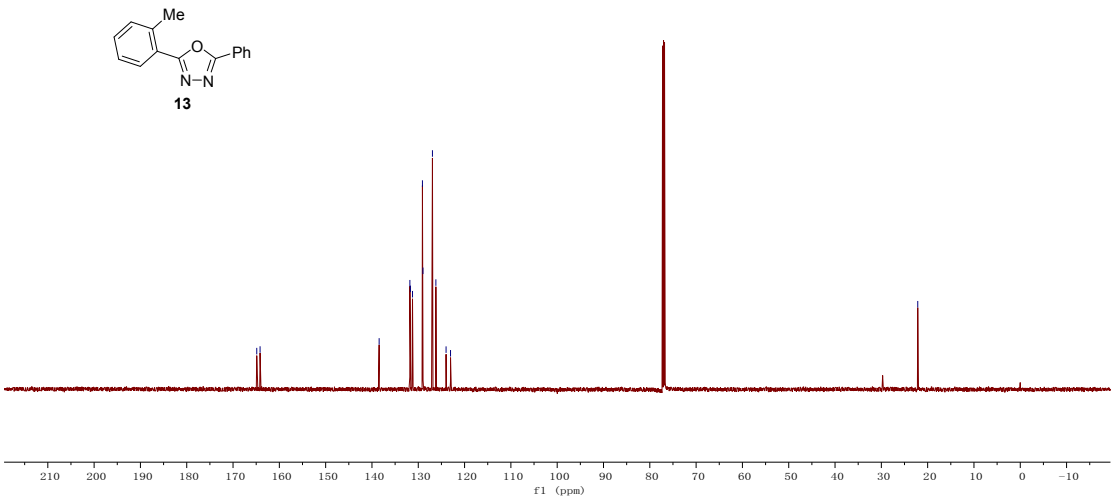

Br
O
Ph
N–N
11
f1 (ppm)

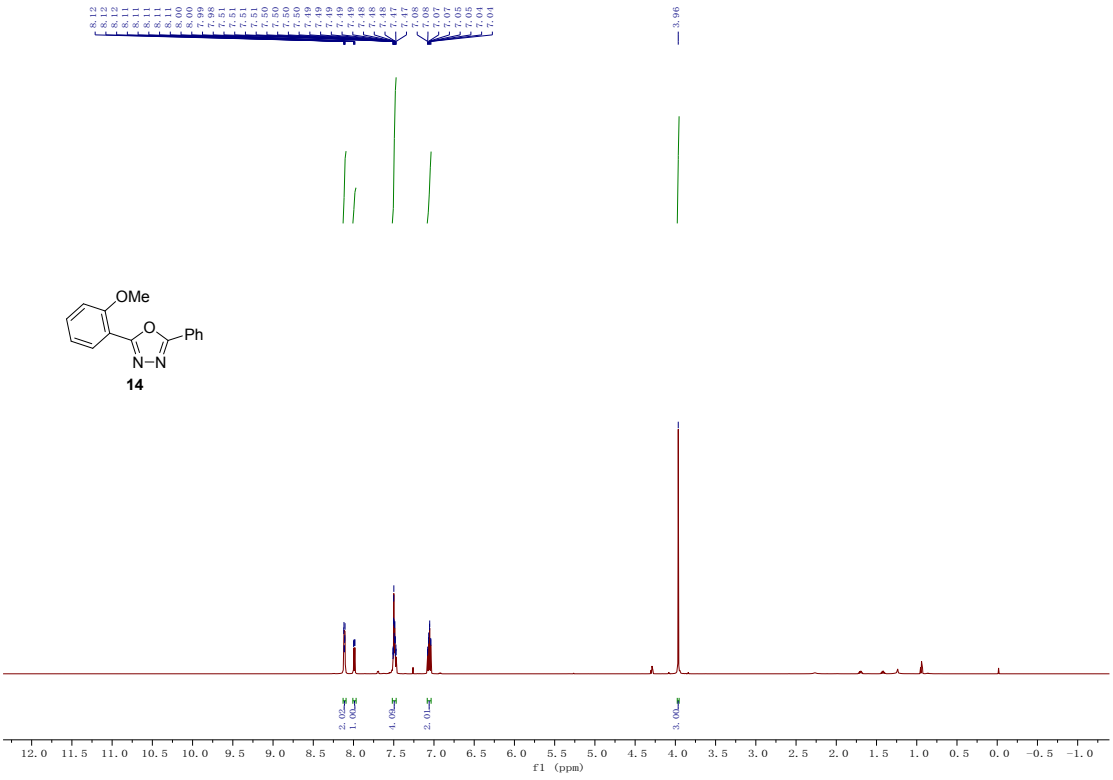

Br
O
Ph
N–N
11
f1 (ppm)

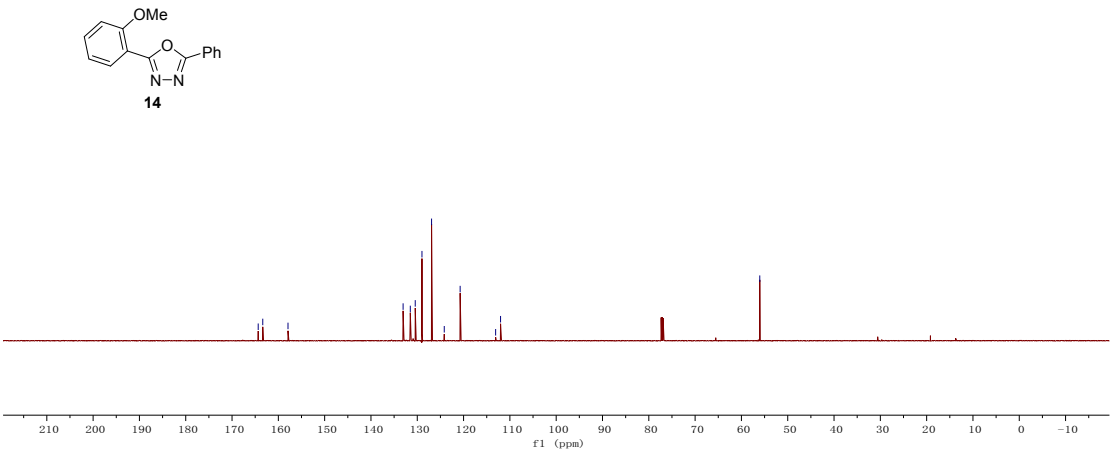

I
O
Ph
N–N
12
f1 (ppm)

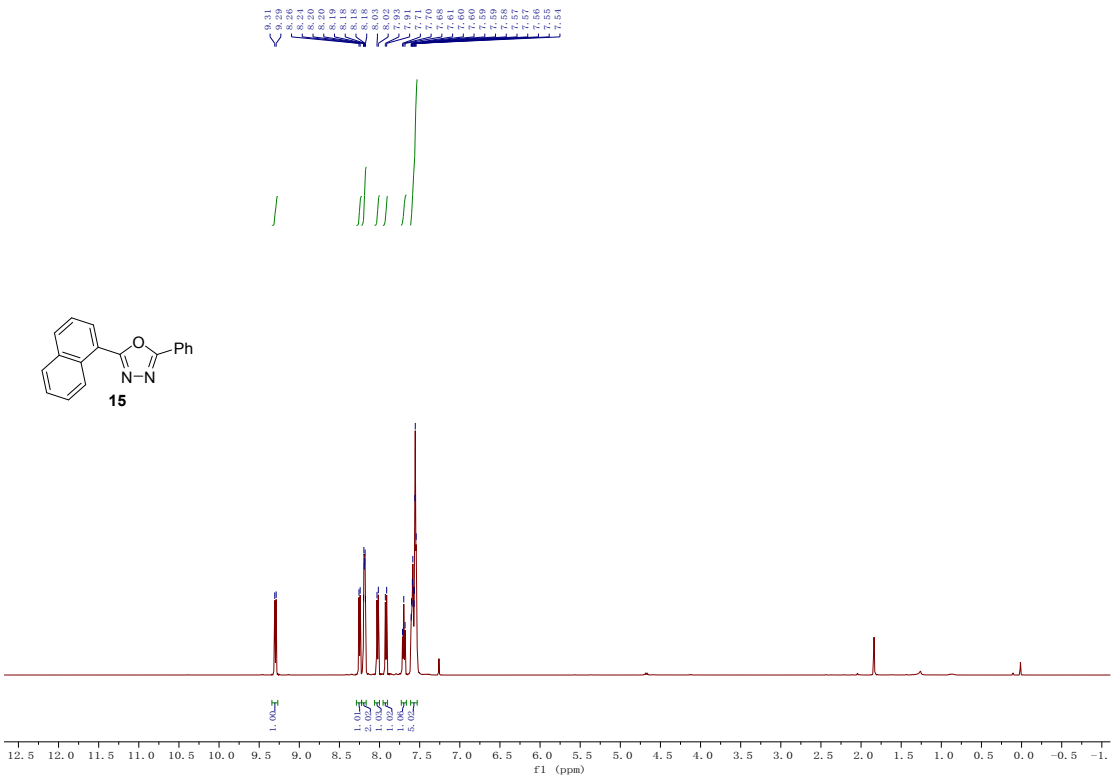

I
O
Ph
N–N
12
f1 (ppm)

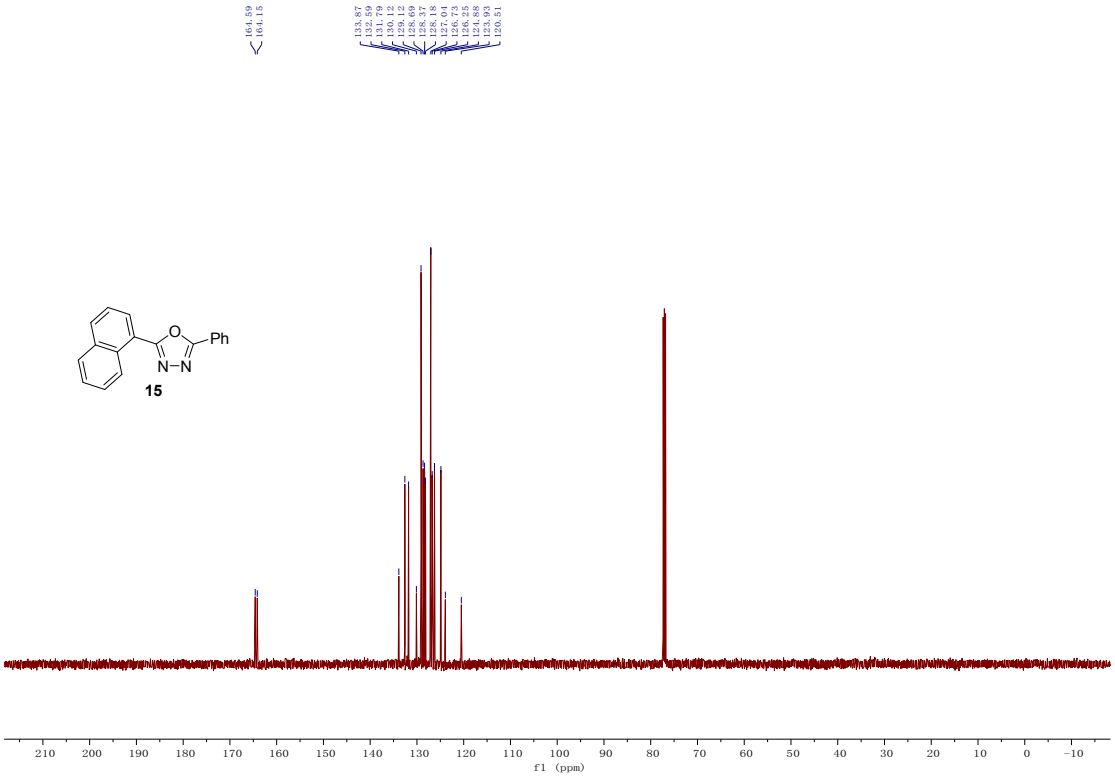

Me
O
Ph
N–N
13
f1 (ppm)

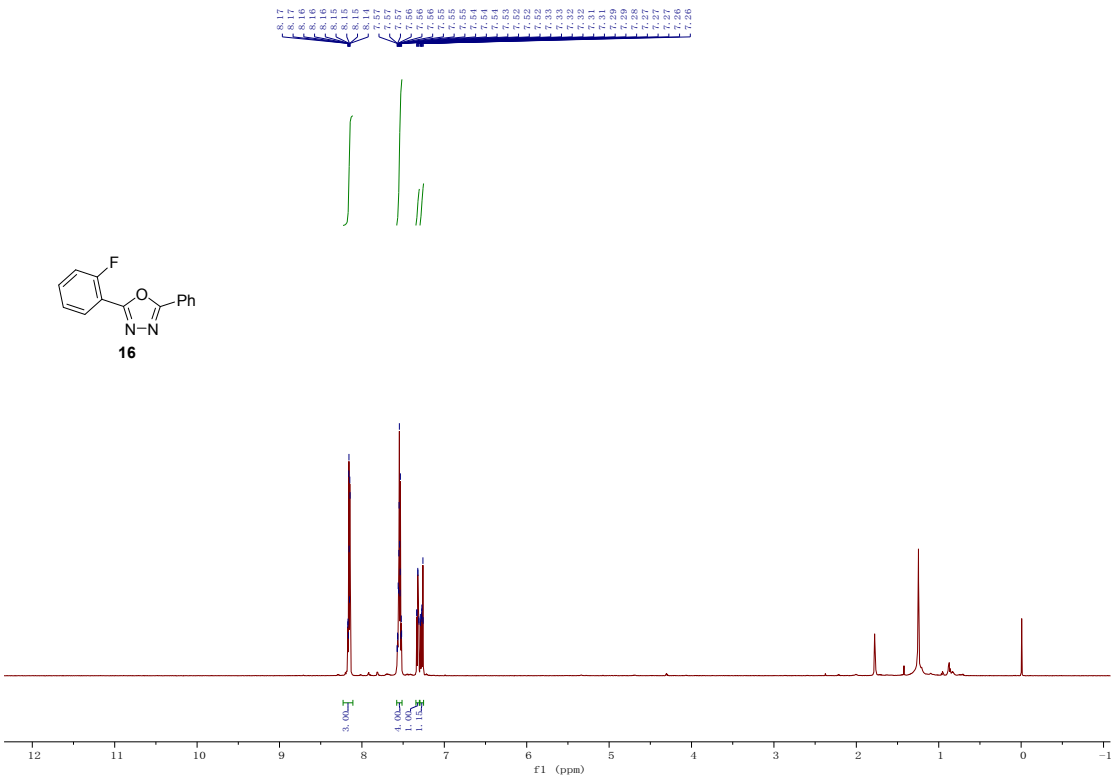

Me
O
Ph
N–N
13
f1 (ppm)

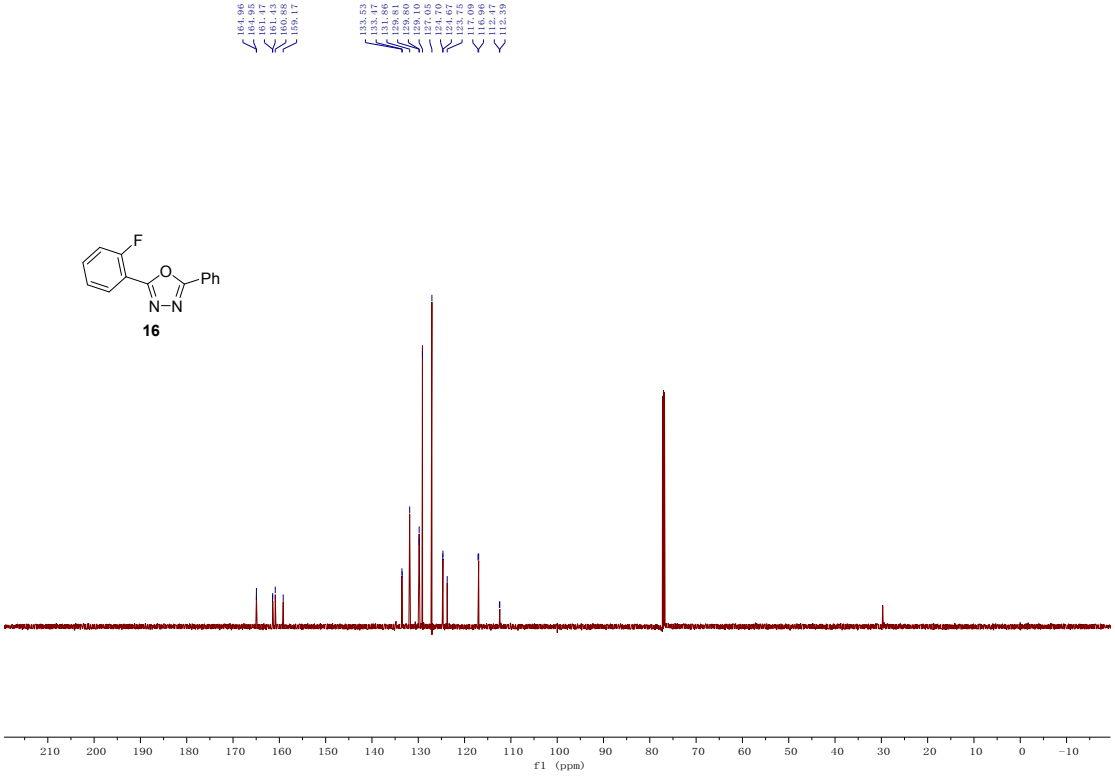

OMe
O
Ph
N–N
14
f1 (ppm)

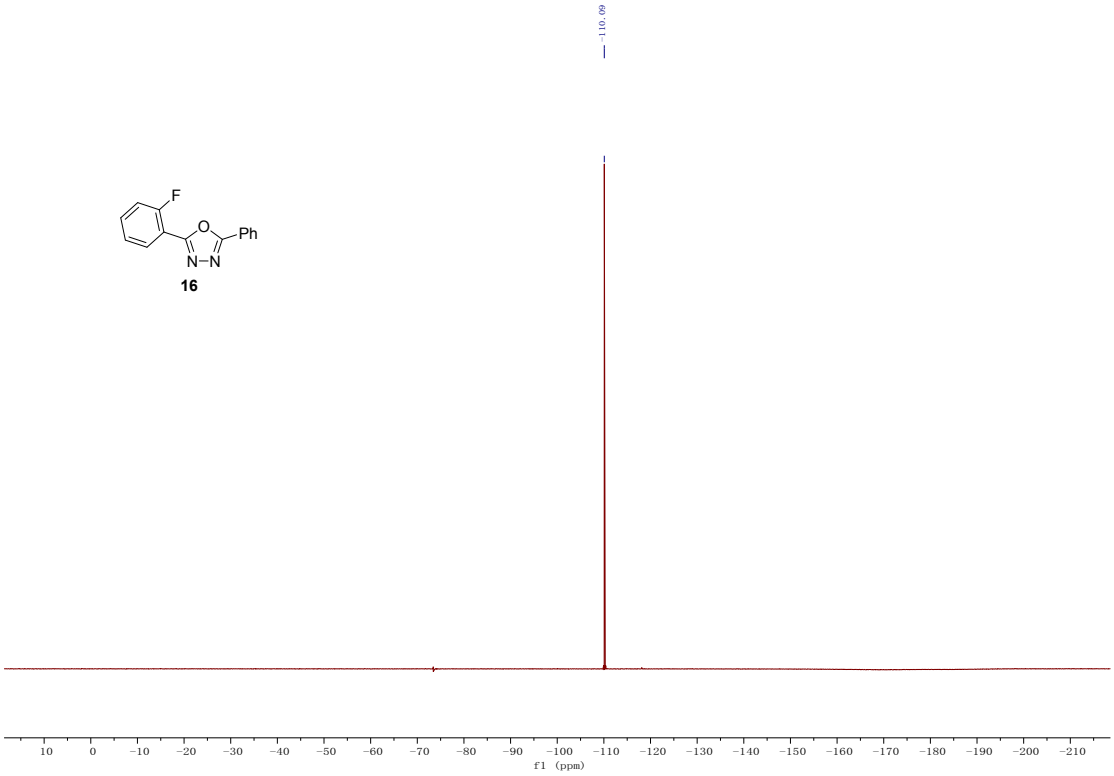

OMe
O
Ph
N–N
14
f1 (ppm)

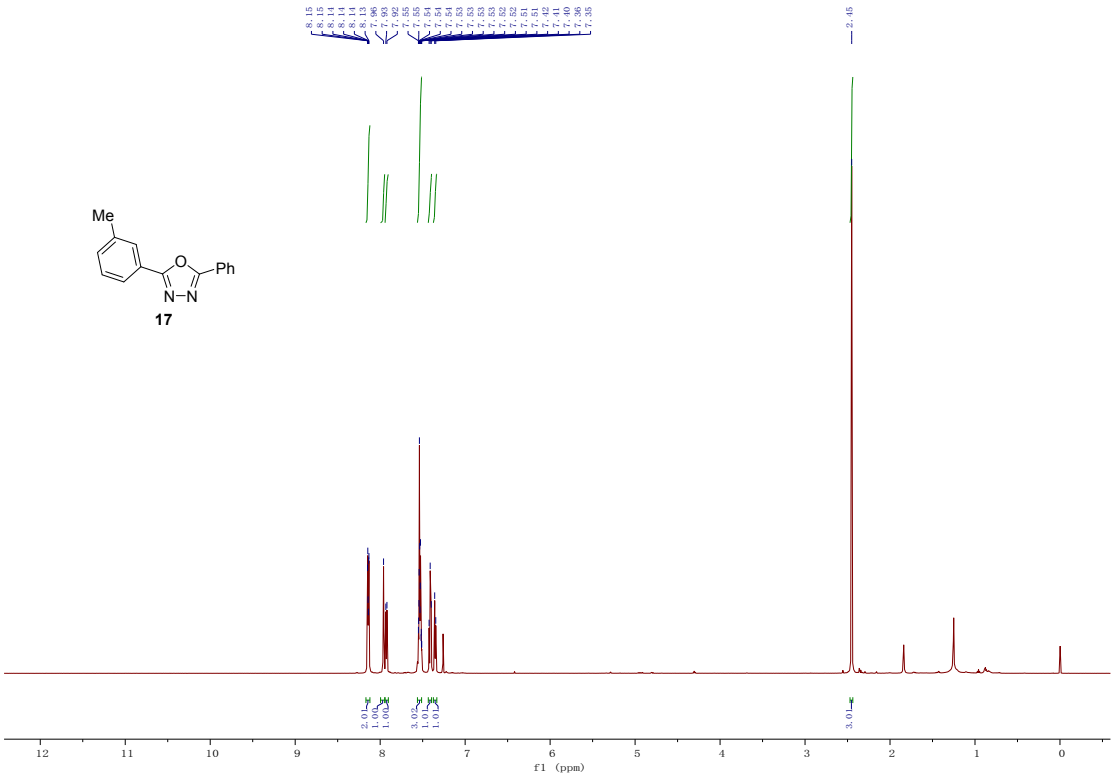

8.31
8.29
8.26
8.24
8.20
8.20
8.19
8.18
8.18
8.18
8.03
8.02
7.93
7.91
7.71
7.70
7.68
7.61
7.60
7.60
7.59
7.59
7.58
7.57
7.57
7.56
7.55
7.54
O
Ph
N–N
15
1.00
1.01
2.02
1.03
1.02
1.06
5.02
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.
f1 (ppm)

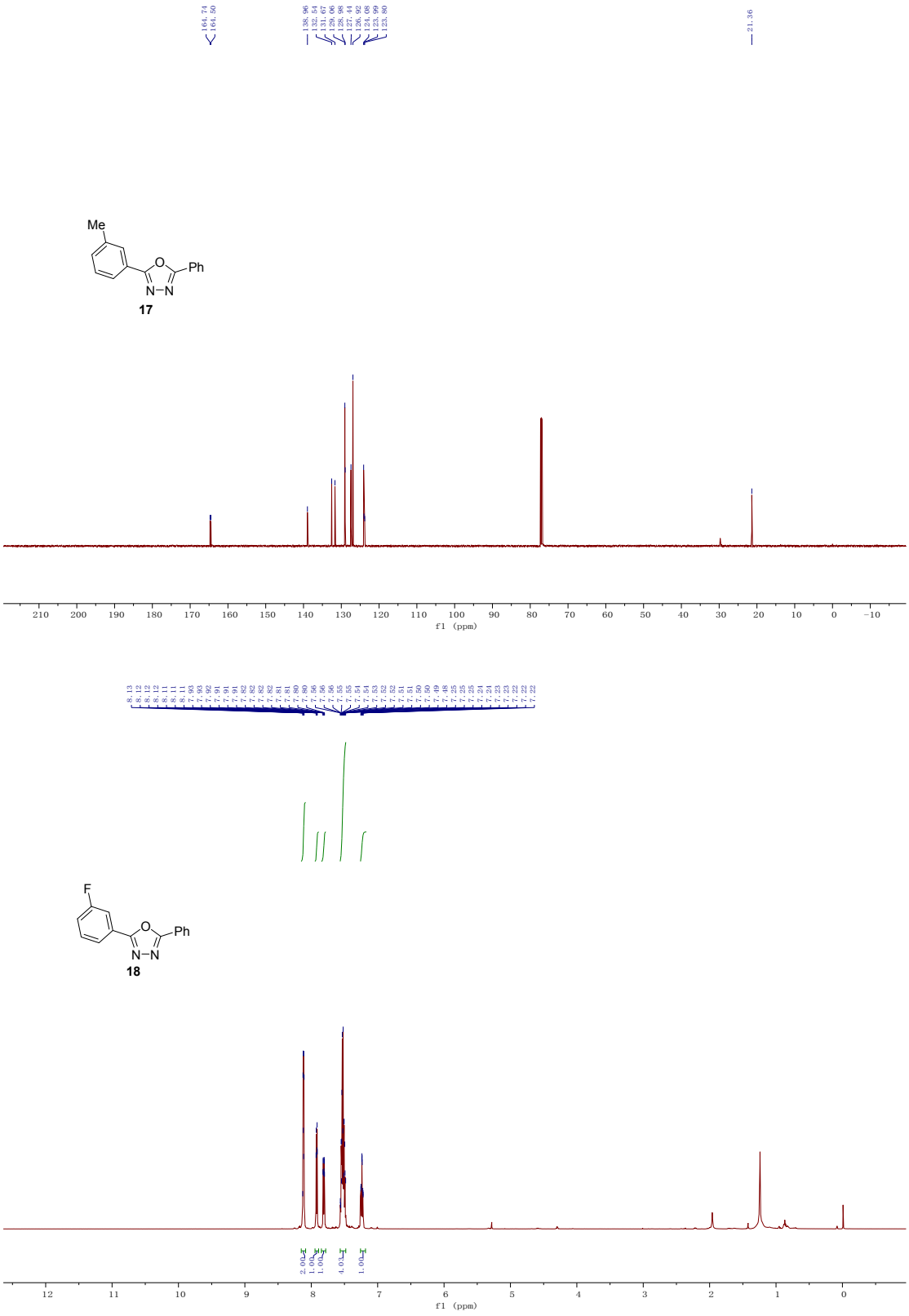

164.59
164.15
133.87
132.59
131.79
130.12
129.12
128.69
128.37
128.18
127.04
126.73
126.25
124.86
123.93
120.51
O
Ph
N–N
15
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


F
O
Ph
N–N
16
f1 (ppm)


F
O
Ph
N–N
16
f1 (ppm)


F
O
Ph
N–N
16
-110.09
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)


Me
O
Ph
N–N
17
8.15 8.15 8.14 8.14 8.14 8.13 7.96 7.93 7.92 7.55 7.55 7.54 7.54 7.54 7.53 7.53 7.53 7.53 7.52 7.51 7.51 7.42 7.41 7.40 7.36 7.35
2.45
2.01 1.00 1.02 3.02 1.01 1.01 3.01
12 11 10 9 8 7 6 5 4 3 2 1 0
f1 (ppm)


164.71
164.50
138.96
132.54
131.67
129.05
128.98
127.44
126.92
124.68
123.99
123.80
21.36
Me
O
Ph
N–N
17
f1 (ppm)
8.13
8.12
8.12
8.12
8.11
8.11
8.11
7.93
7.92
7.91
7.91
7.91
7.82
7.82
7.82
7.82
7.81
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7.81
7.80
7.80
7.56
7.56
7.56
7.55
7.55
7.54
7.54
7.53
7.52
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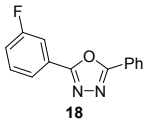
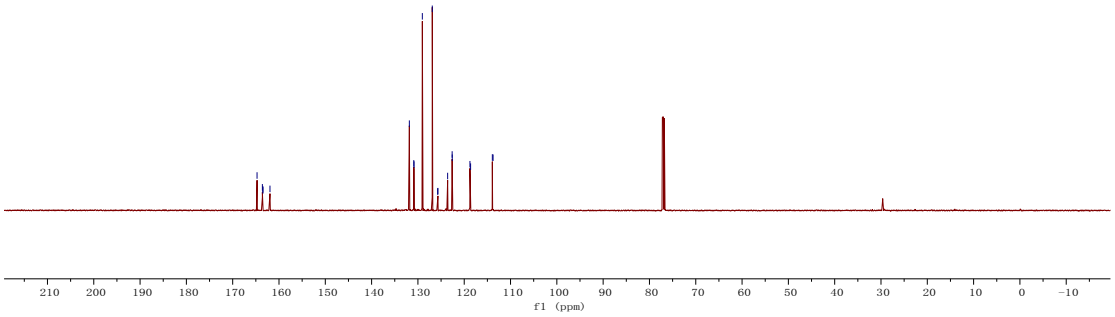
7.51
7.51
7.50
7.50
7.49
7.49
7.25
7.25
7.25
7.24
7.24
7.23
7.23
7.22
7.22
7.22
F
O
Ph
N–N
18
2.00
1.00
1.00
4.03
1.00
f1 (ppm)

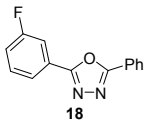

164.74
163.59
163.49
163.47
161.95
131.85
130.89
130.84
129.05
126.90
125.74
125.68
123.58
122.59
118.77
118.62
113.93
113.77


F
O
Ph
N–N

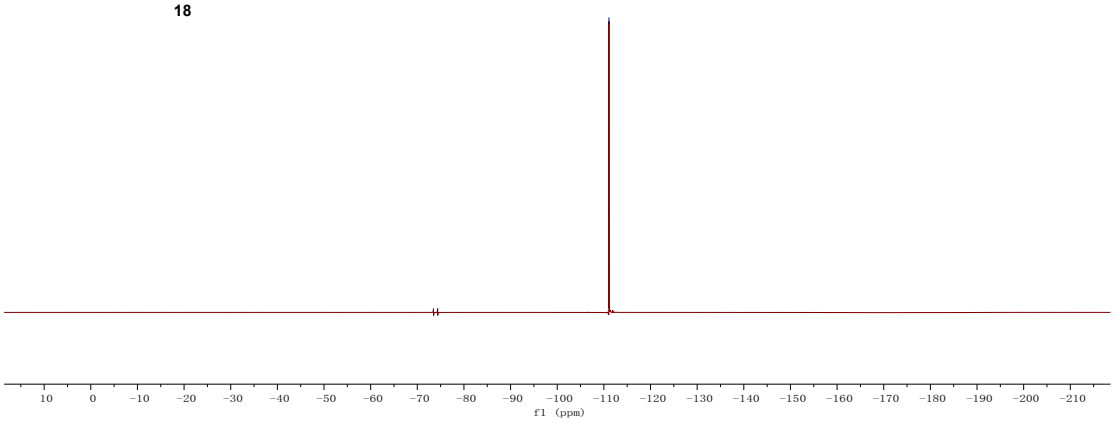
**18**

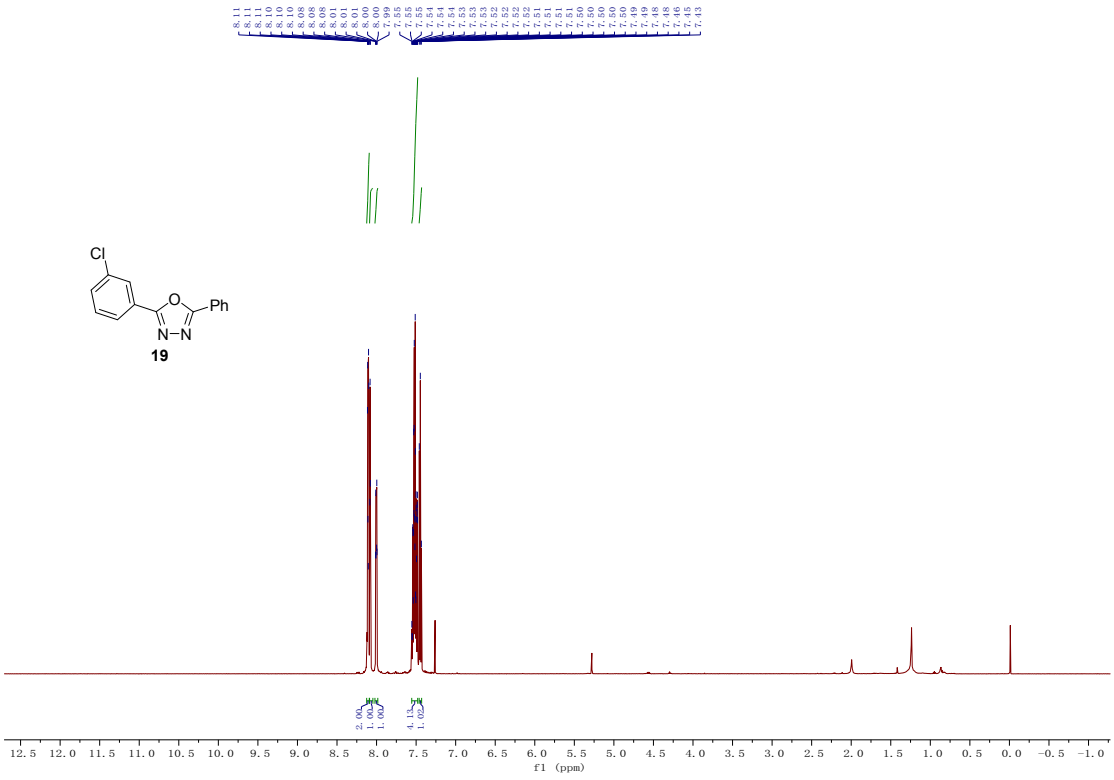

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

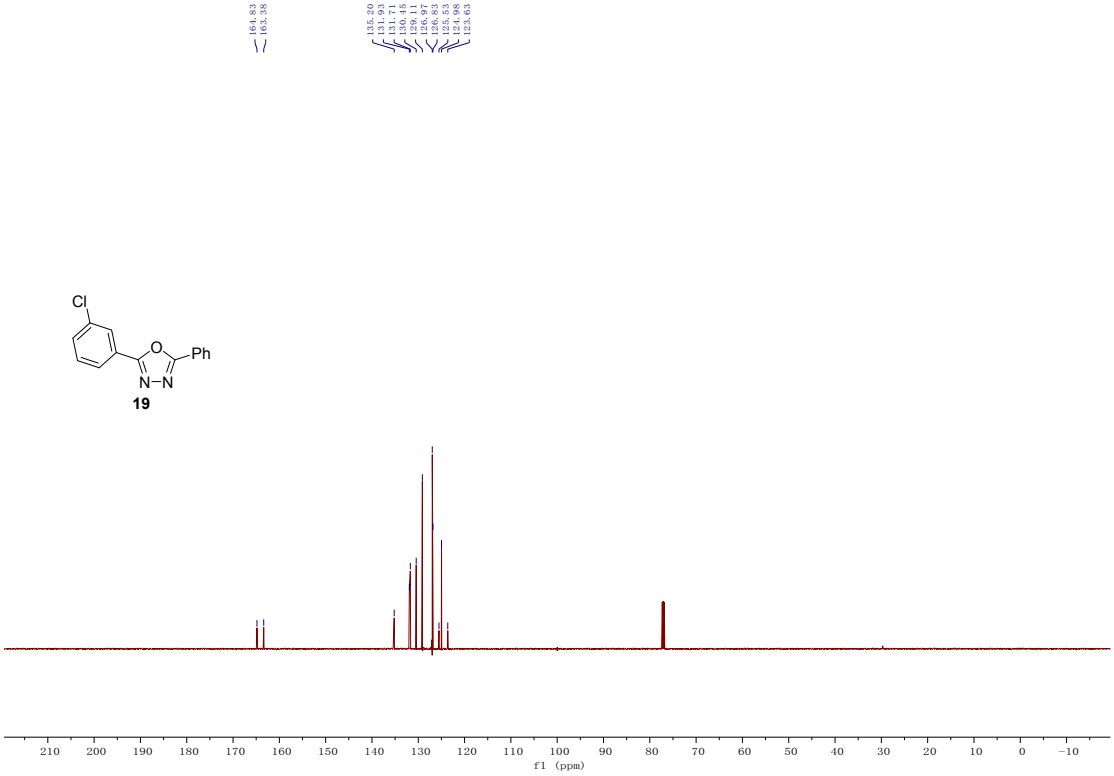

-111.09

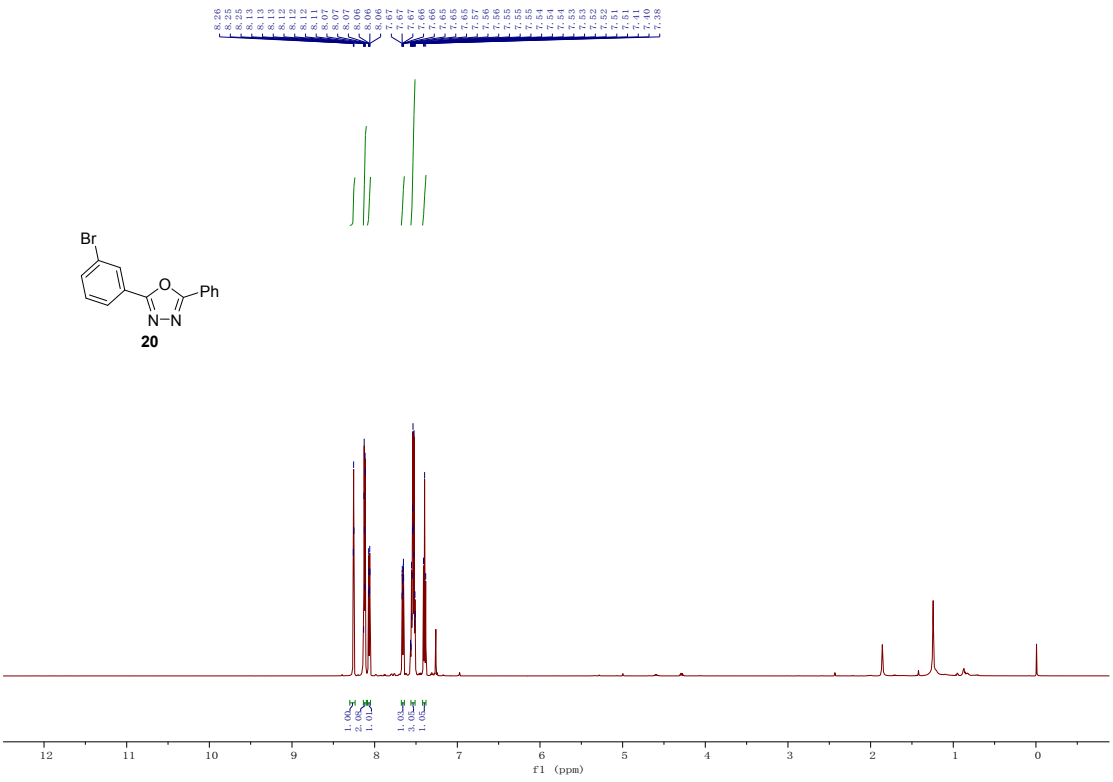

F
O
Ph
N–N

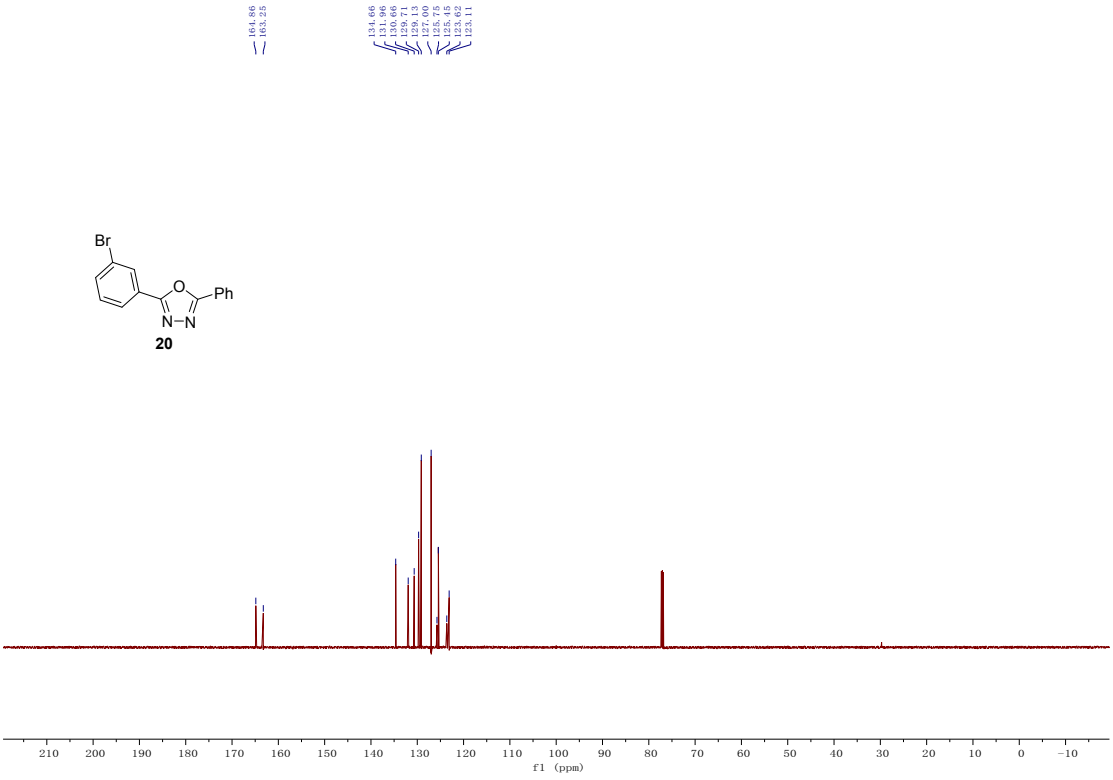
**18**

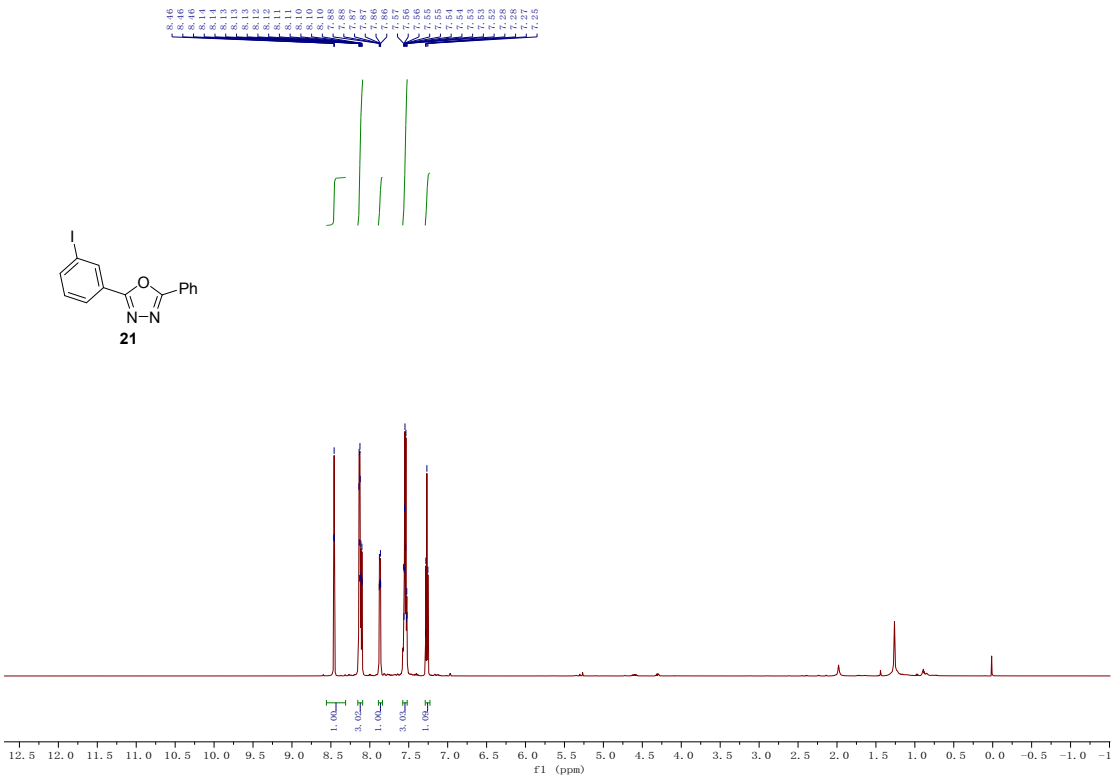

10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)

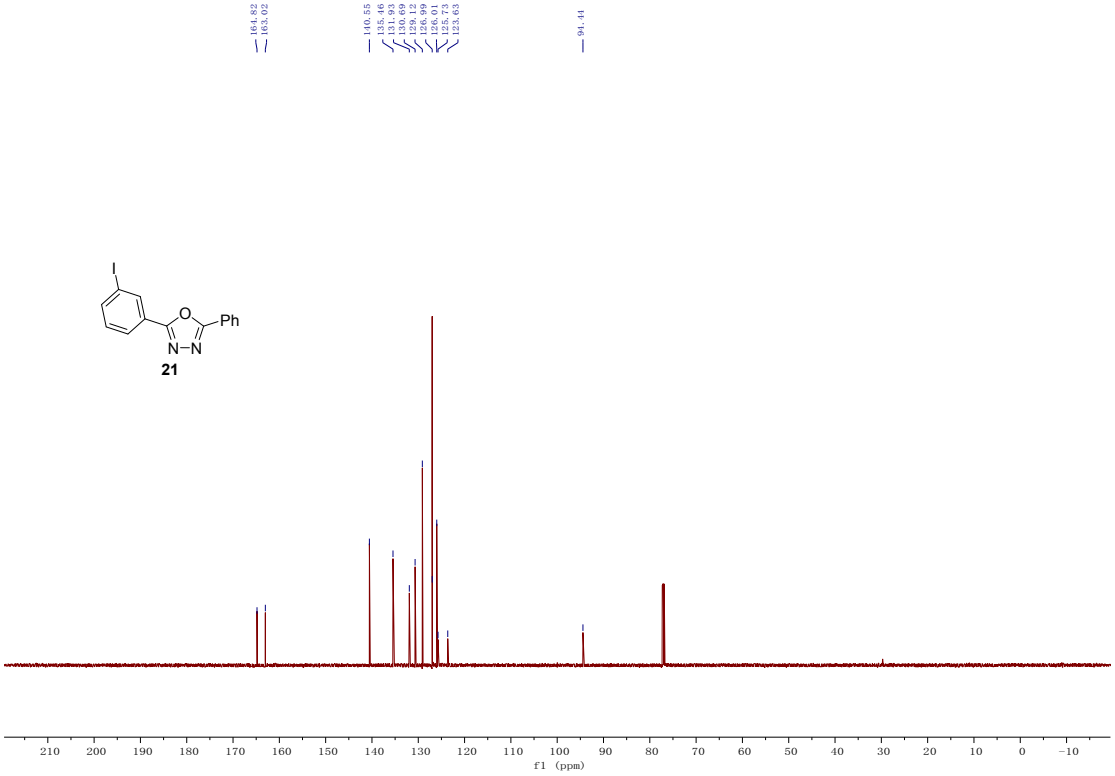

Cl
O
Ph
N–N
19
f1 (ppm)

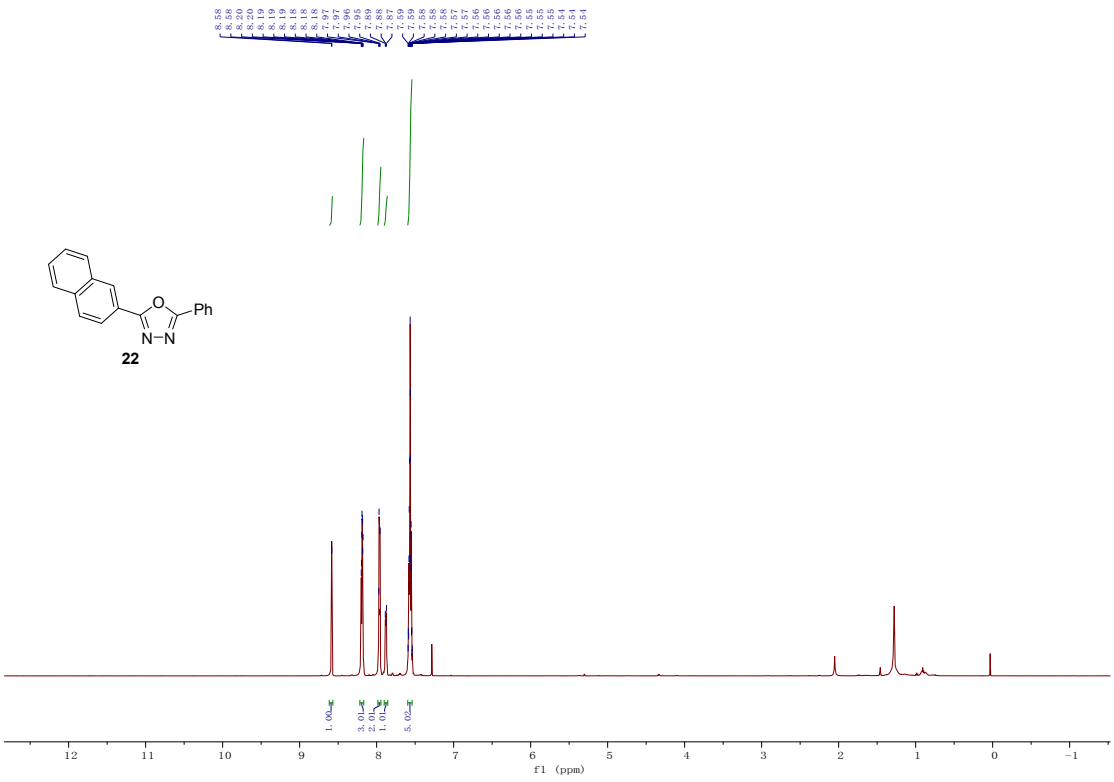

Cl
O
Ph
N–N
19
f1 (ppm)

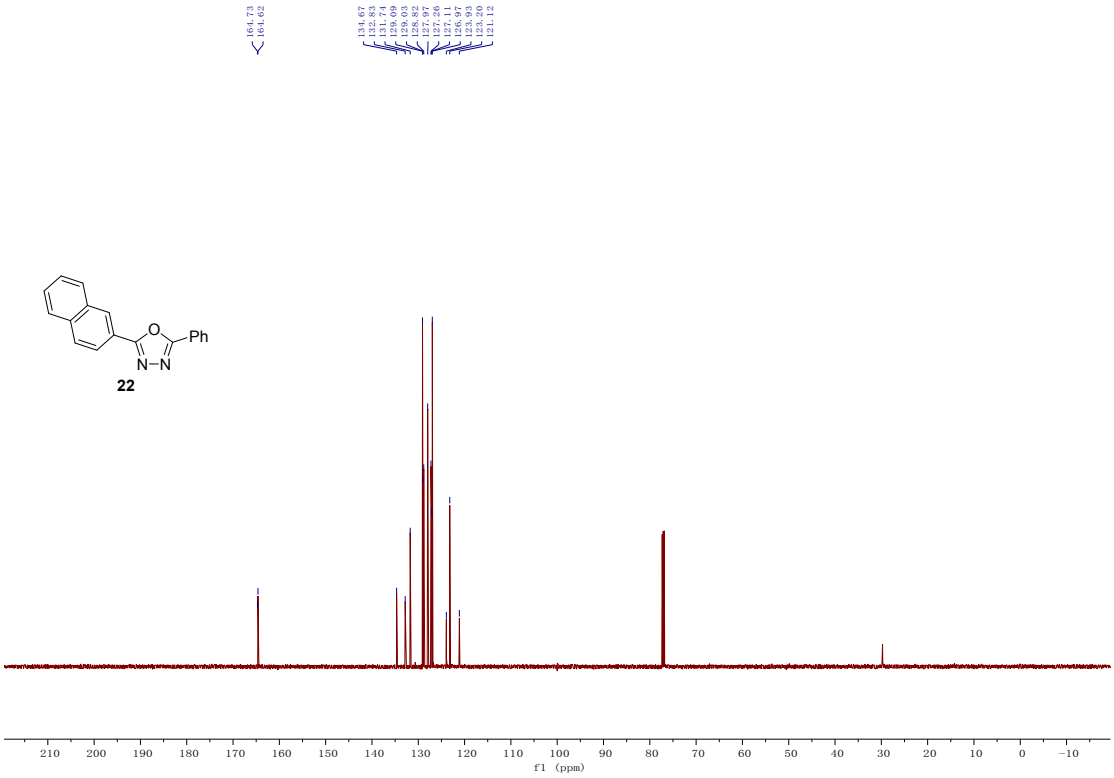

Br
O
Ph
N–N
20
f1 (ppm)

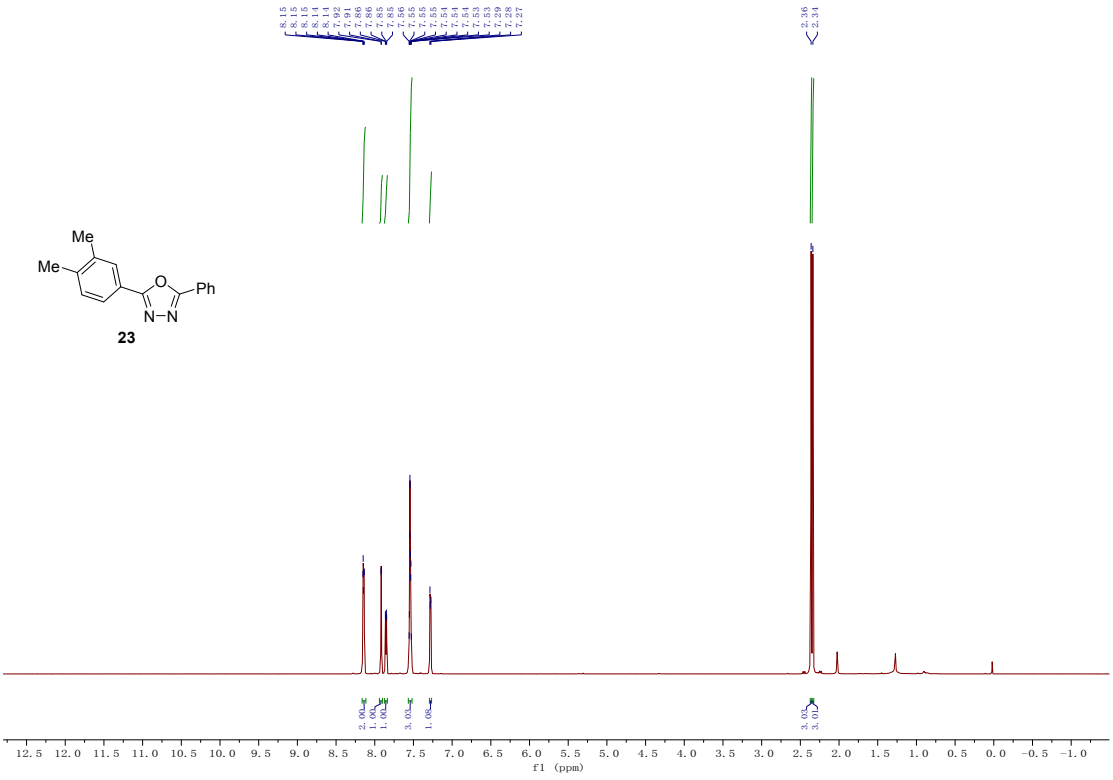

Br
O
Ph
N–N
20
f1 (ppm)

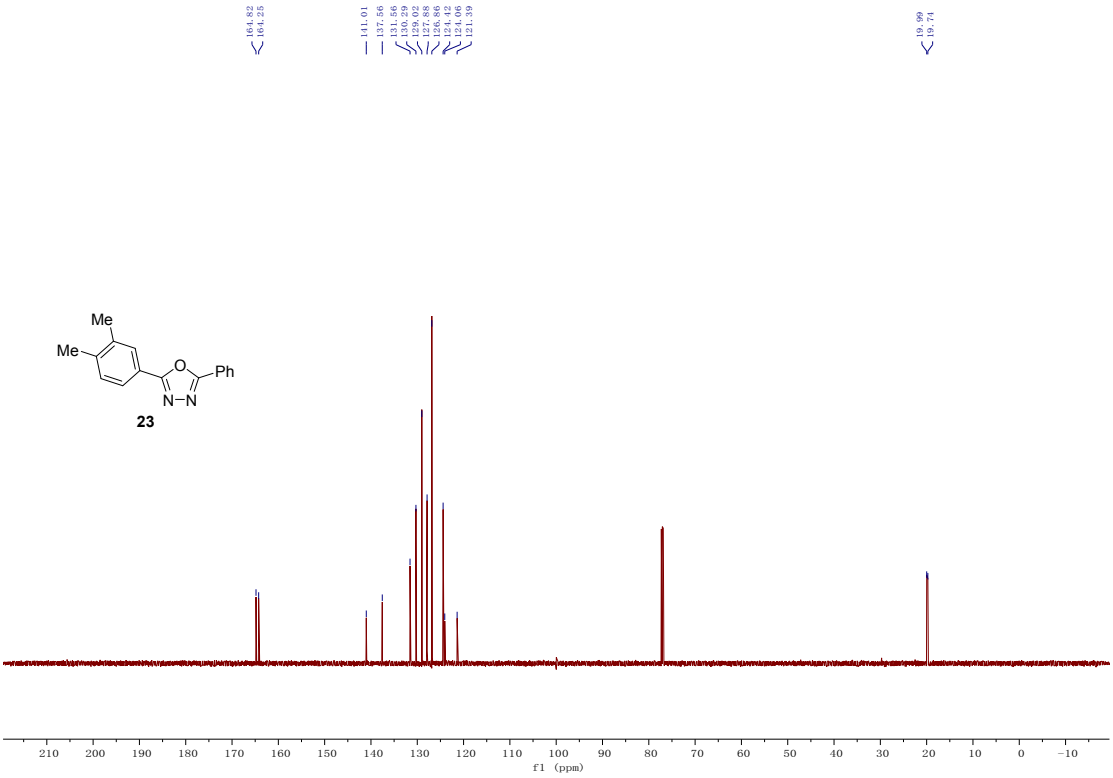

I
O
Ph
N–N
21
f1 (ppm)

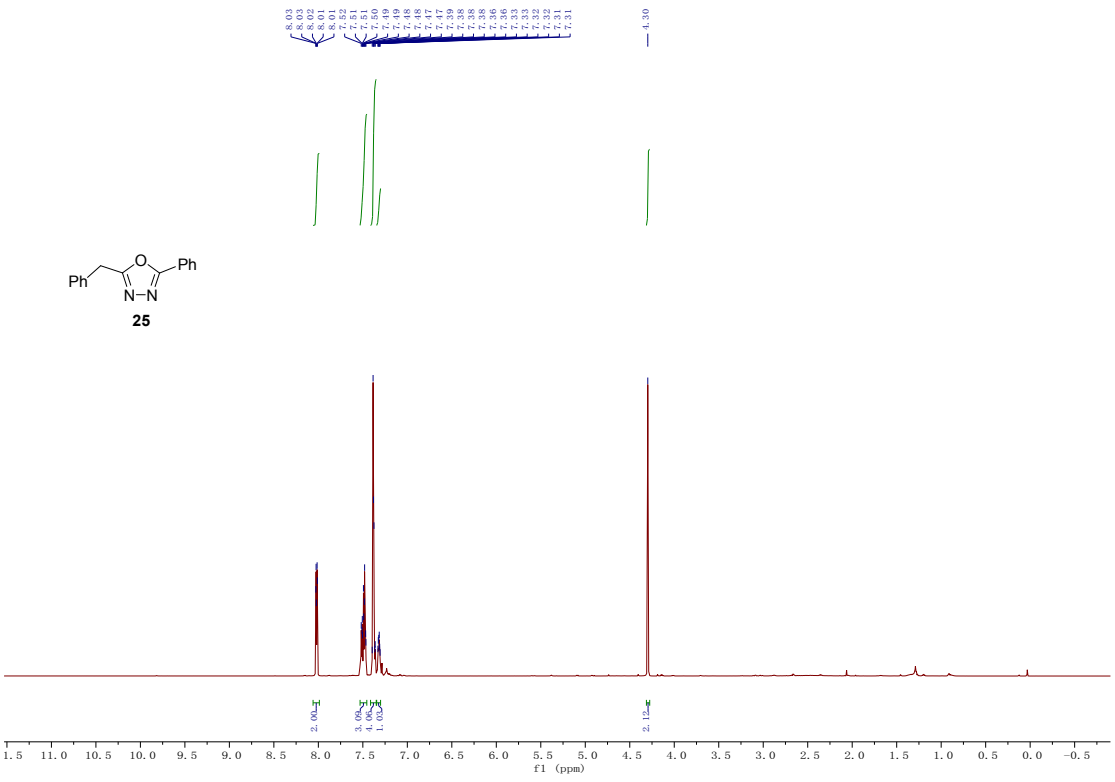

I
O
Ph
N–N
21
f1 (ppm)

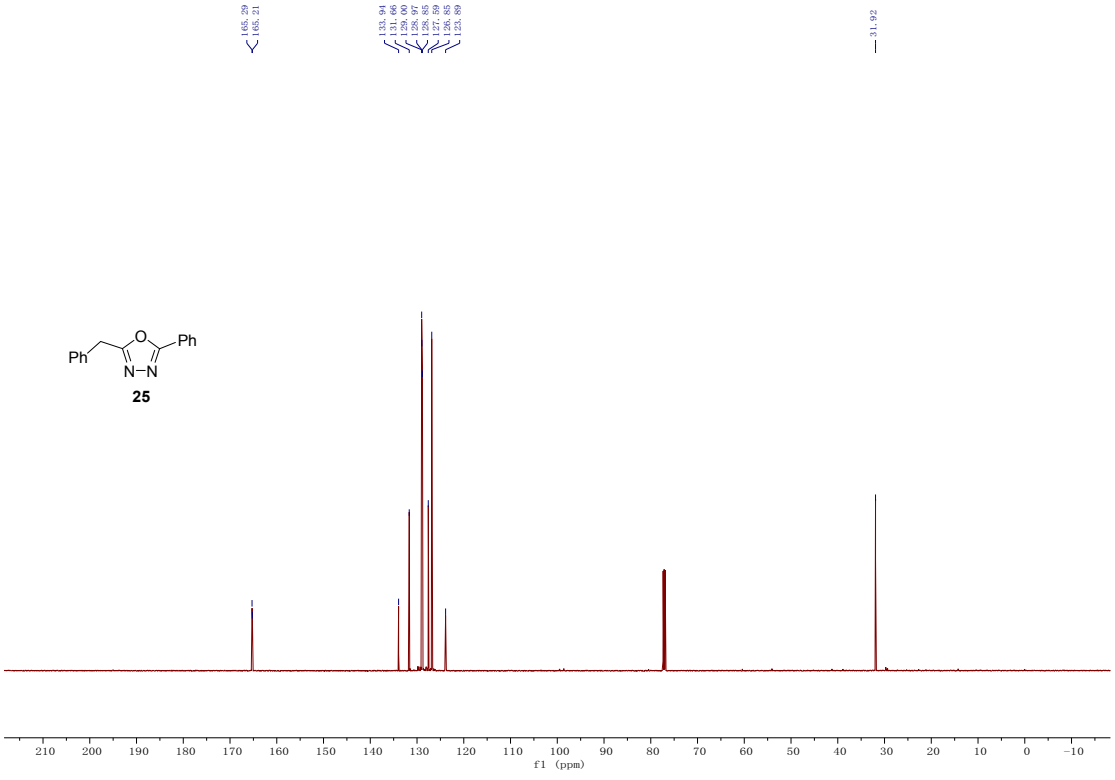

Ph
N–N
O
22
f1 (ppm)

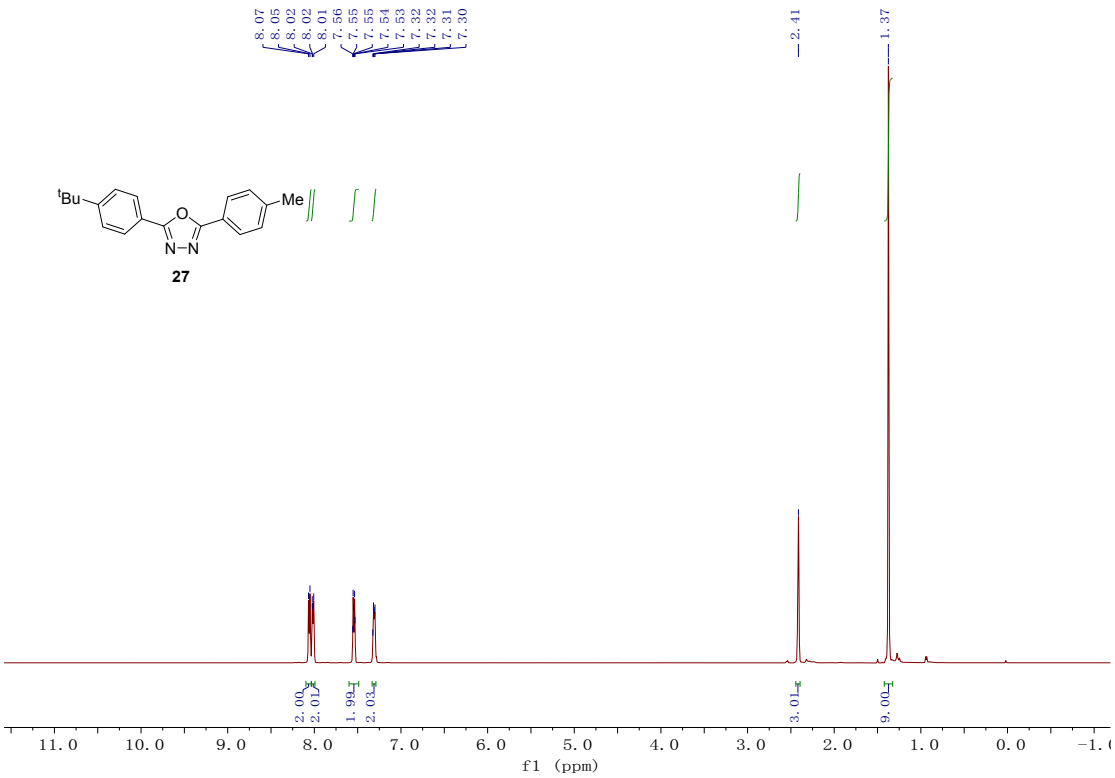

Ph
N–N
O
22
f1 (ppm)

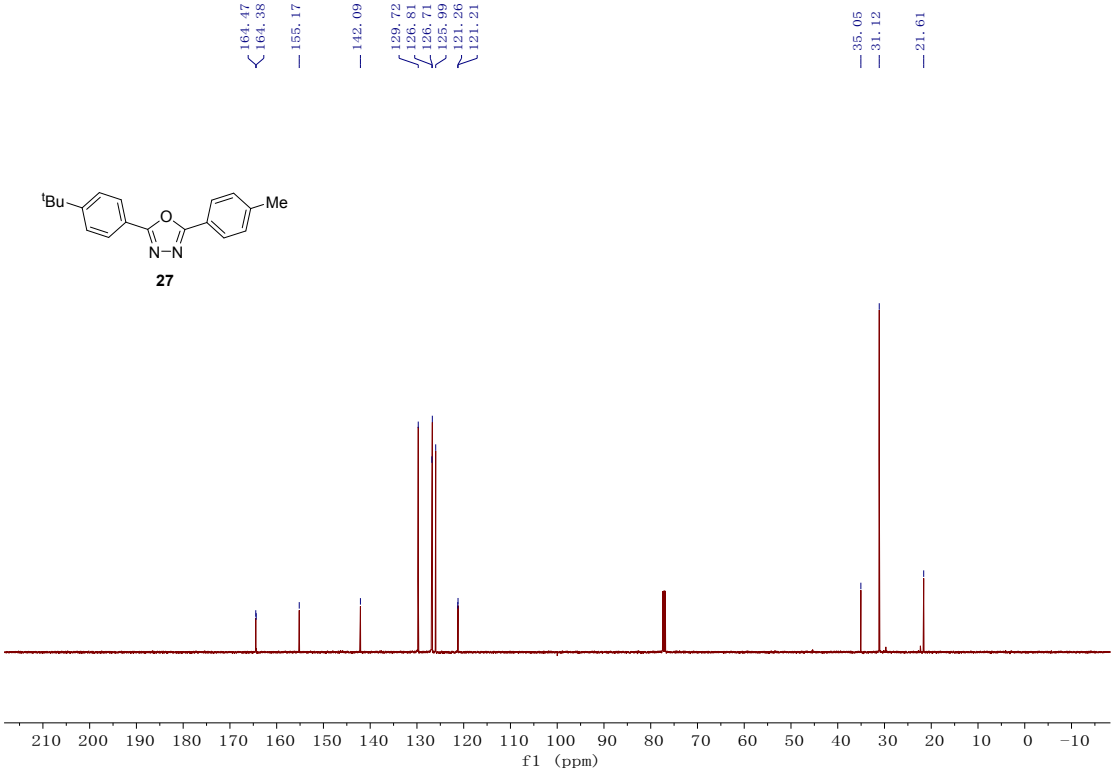

Me
Me
O
Ph
N–N
23
8.15
8.15
8.15
8.14
8.14
7.92
7.91
7.86
7.86
7.85
7.85
7.56
7.55
7.55
7.55
7.54
7.54
7.54
7.53
7.53
7.29
7.28
7.27
2.36
2.34
2.00
1.00
1.00
3.02
1.00
3.03
3.01
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

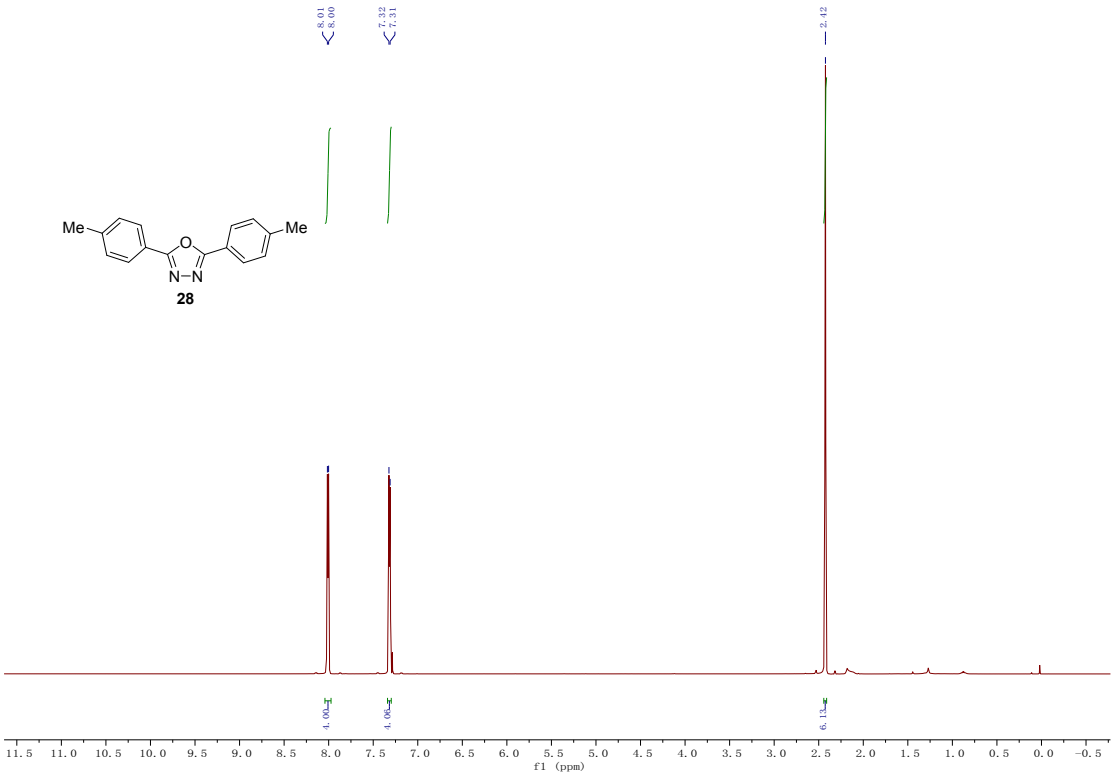

164.82
164.25
141.01
137.56
131.56
130.29
129.02
127.88
126.86
124.47
124.06
121.39
19.99
19.74
Me
Me
O
Ph
N–N
23
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

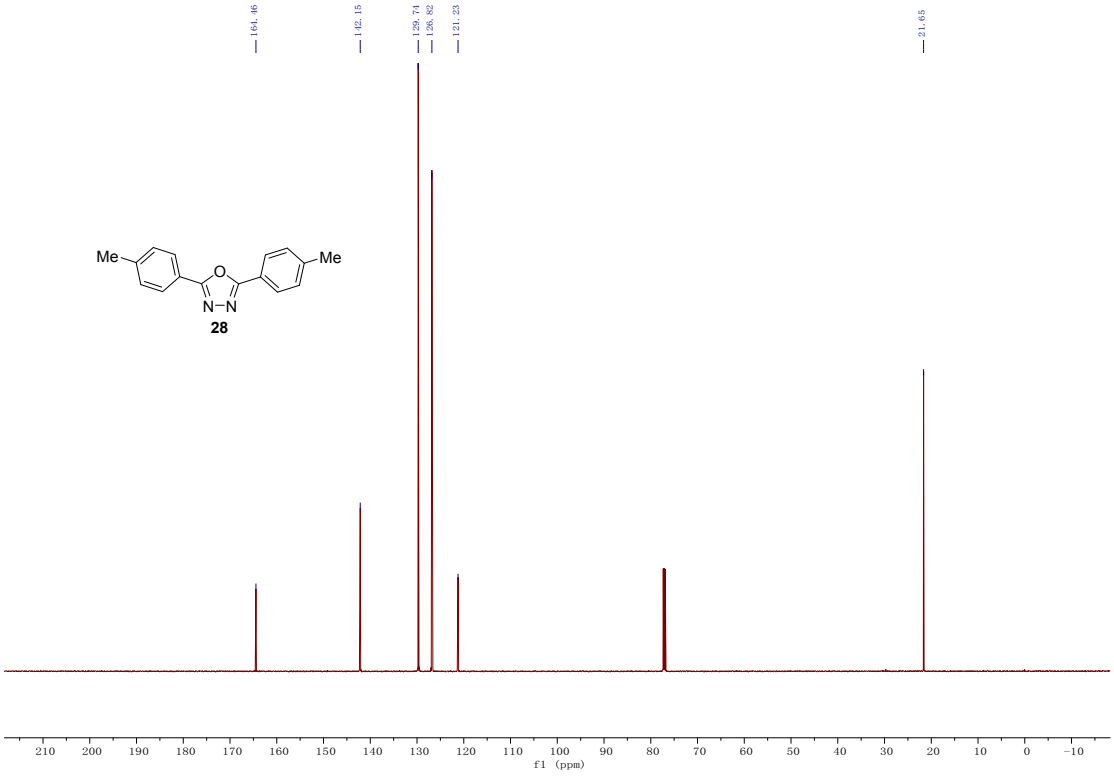

8.03
8.03
8.02
8.01
8.01
7.52
7.51
7.51
7.50
7.49
7.49
7.48
7.48
7.47
7.47
7.39
7.39
7.38
7.38
7.36
7.36
7.33
7.32
7.32
7.31
7.31
4.30
Ph
Ph
O
N–N
25
2.00
3.09
4.06
1.03
2.12
f1 (ppm)

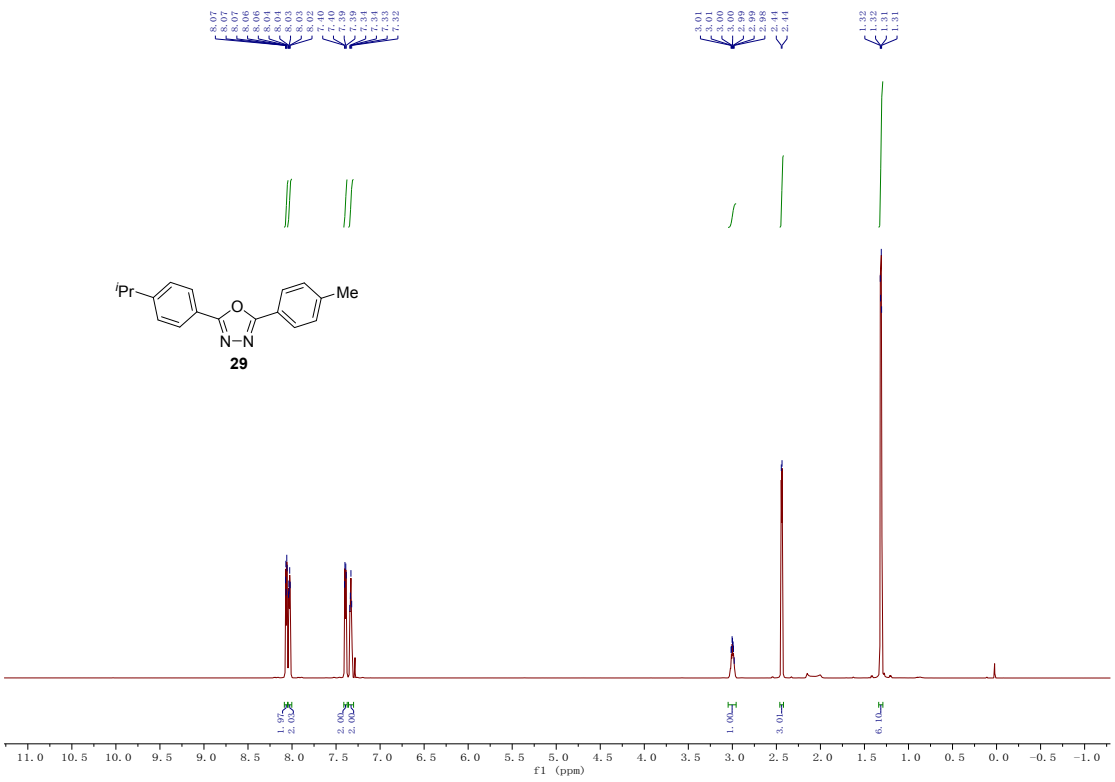

165.29
165.21
133.94
131.66
129.00
128.97
128.85
127.89
127.57
126.85
123.89
31.92
Ph
Ph
O
N–N
25
f1 (ppm)

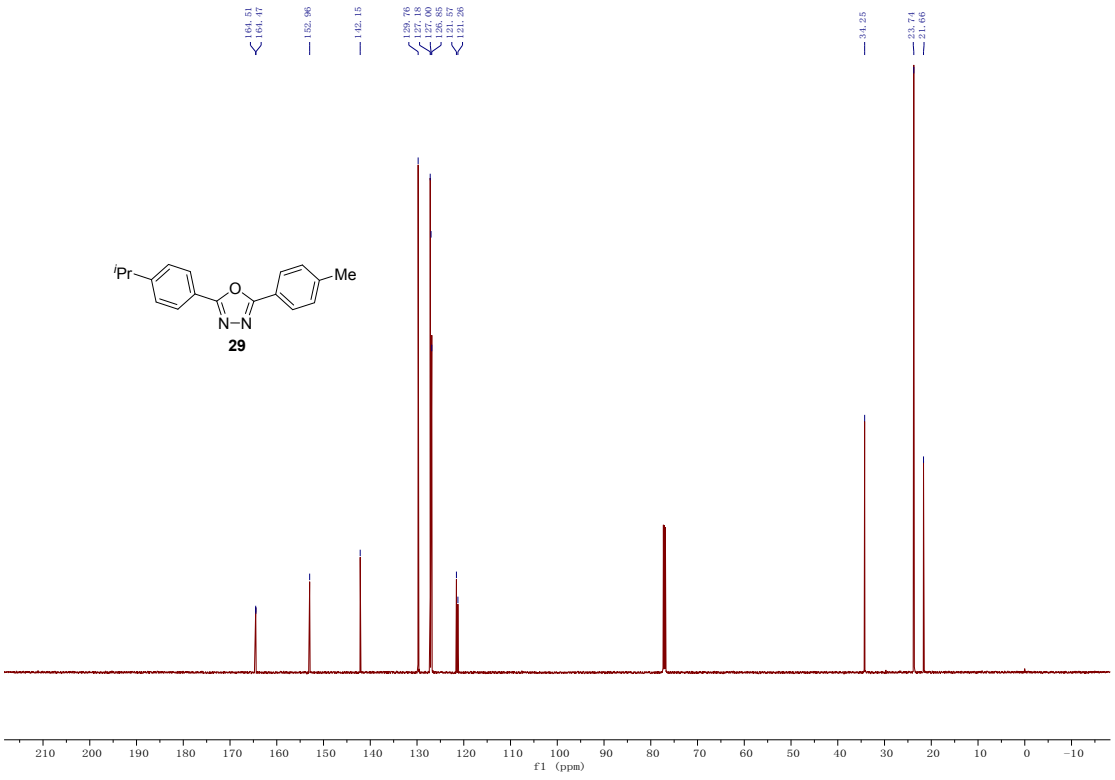

8.07
8.05
8.02
8.02
8.01
7.56
7.55
7.55
7.54
7.53
7.32
7.32
7.31
7.30
2.41
1.37
tBu
Me
O
N–N
27
2.00
2.01
1.99
2.03
3.01
9.00
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

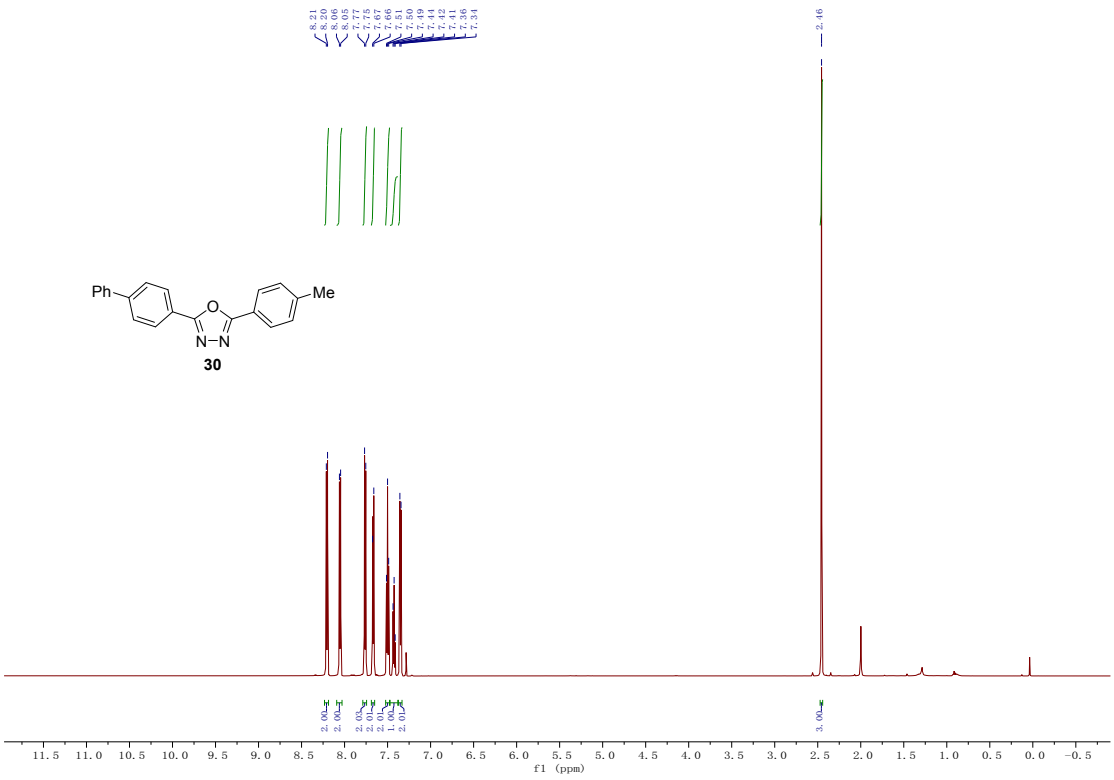

164.47
164.38
155.17
142.09
129.72
126.81
126.71
125.99
121.26
121.21
35.05
31.12
21.61
tBu
Me
O
N–N
27
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

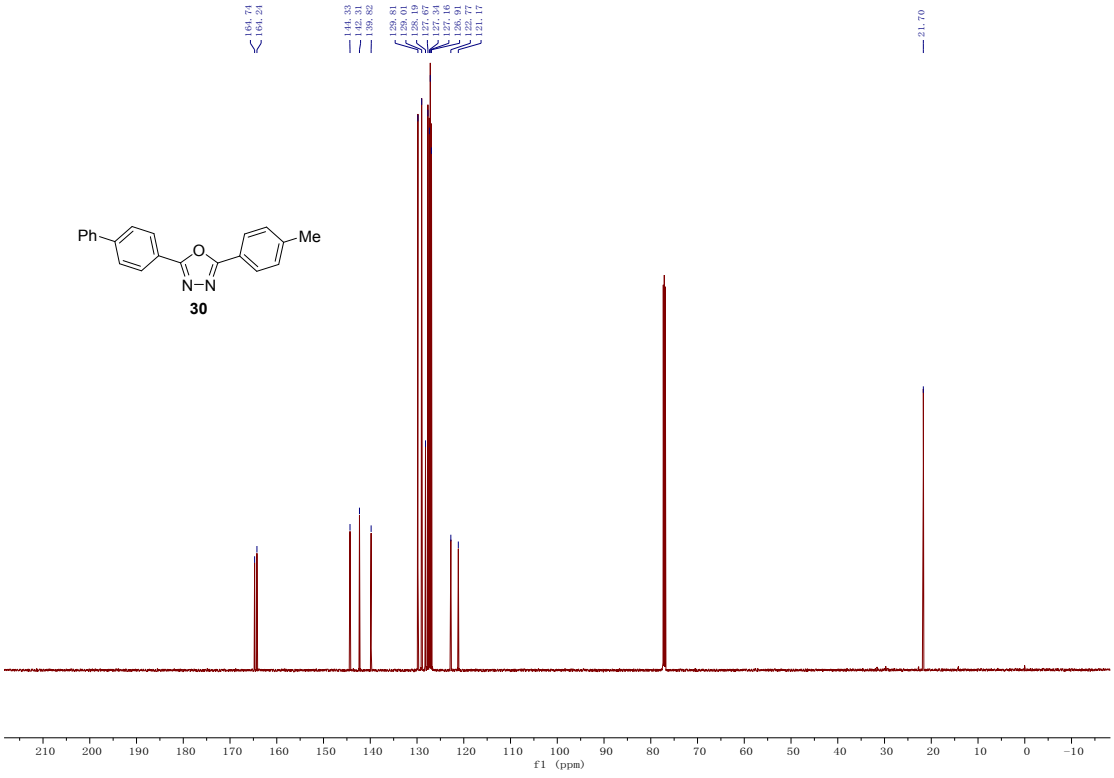

8.01
8.00
7.32
7.31
2.42
Me
Me
O
N–N
28
4.00
4.00
6.13
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5
f1 (ppm)

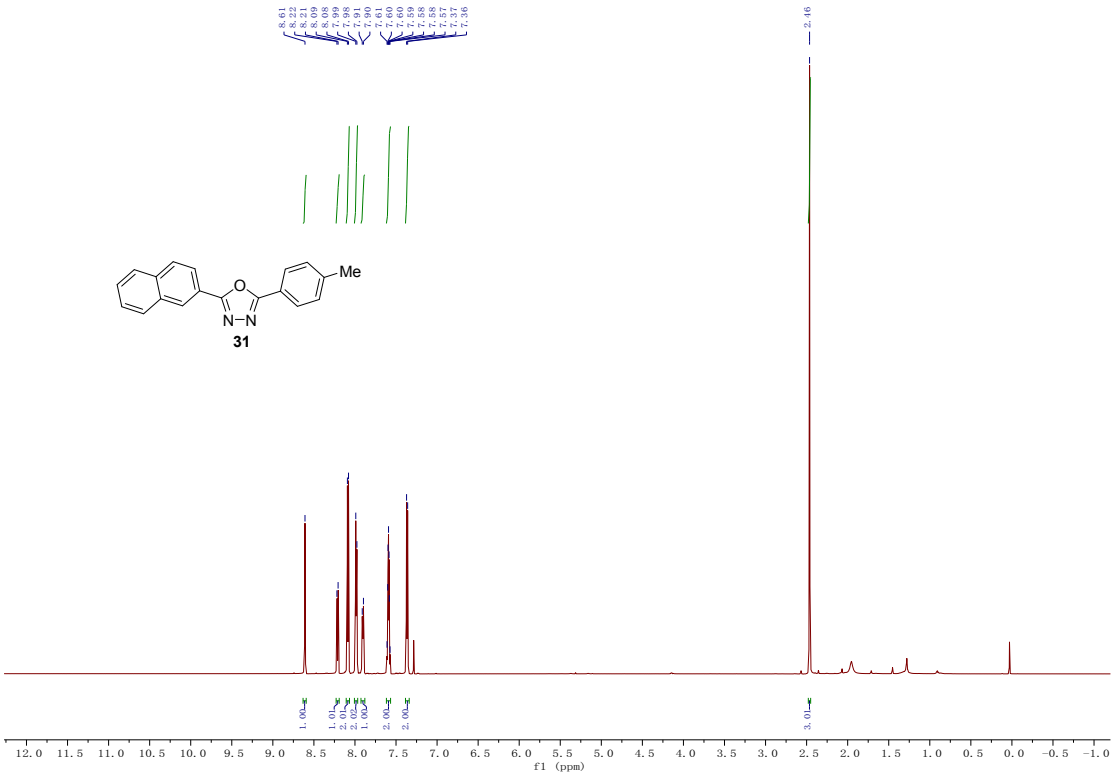

164.46
142.15
129.74
126.82
121.23
21.65
Me
Me
O
N–N
28
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

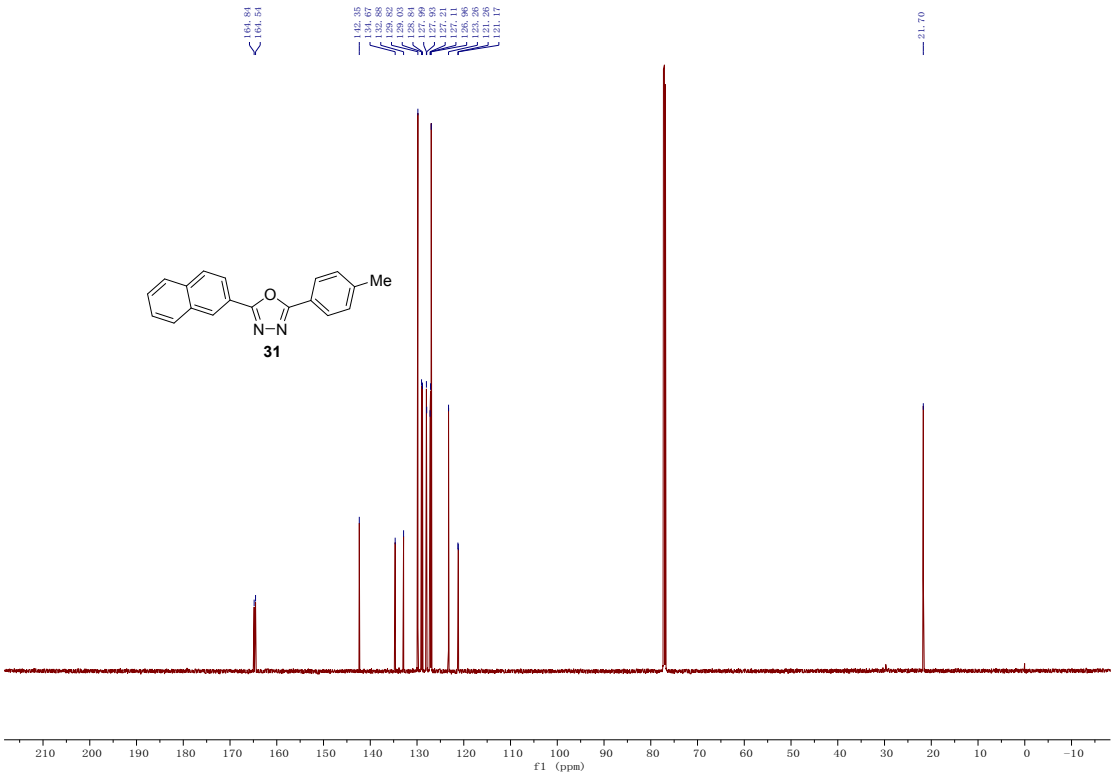

8.07
8.07
8.07
8.06
8.06
8.04
8.04
8.03
8.03
7.40
7.40
7.39
7.39
7.34
7.34
7.33
7.32
3.01
3.01
3.00
2.99
2.99
2.98
2.44
2.44
1.32
1.32
1.31
1.31
1.97
2.03
2.00
2.00
1.00
3.01
6.10
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)
iPr
Me
O
N–N
29

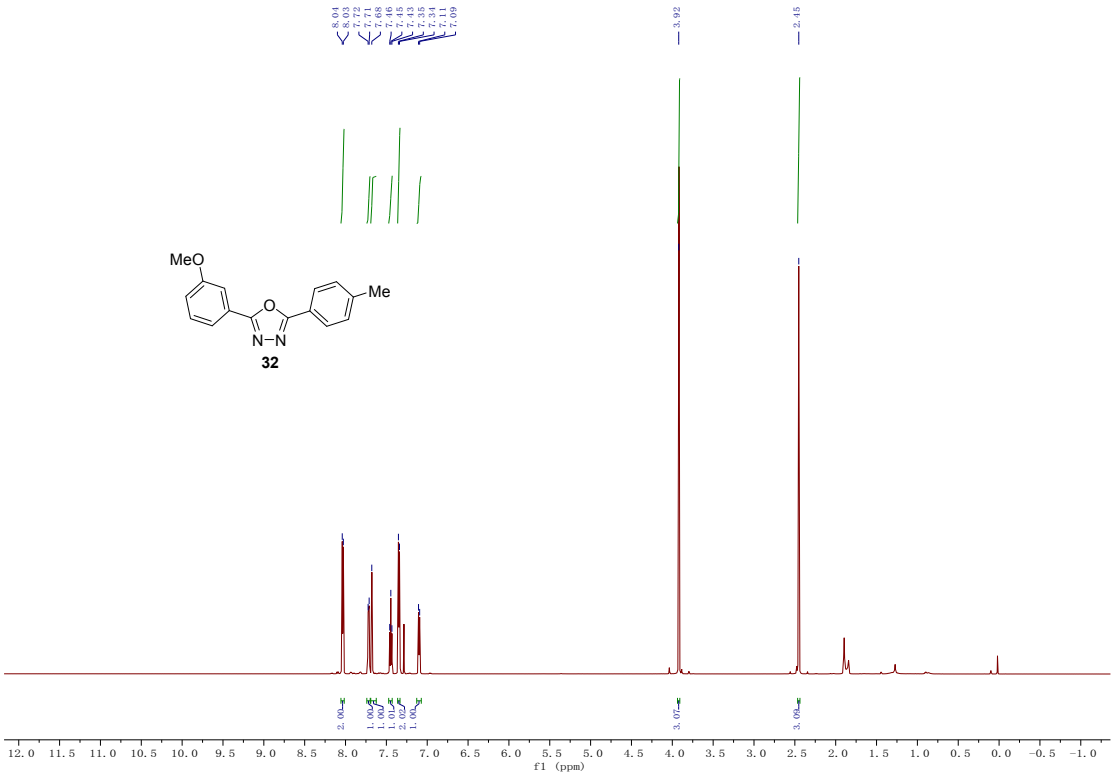

164.51
164.47
152.96
142.15
129.76
127.18
127.00
126.85
121.57
121.26
34.25
23.74
21.66
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
iPr
Me
O
N–N
29

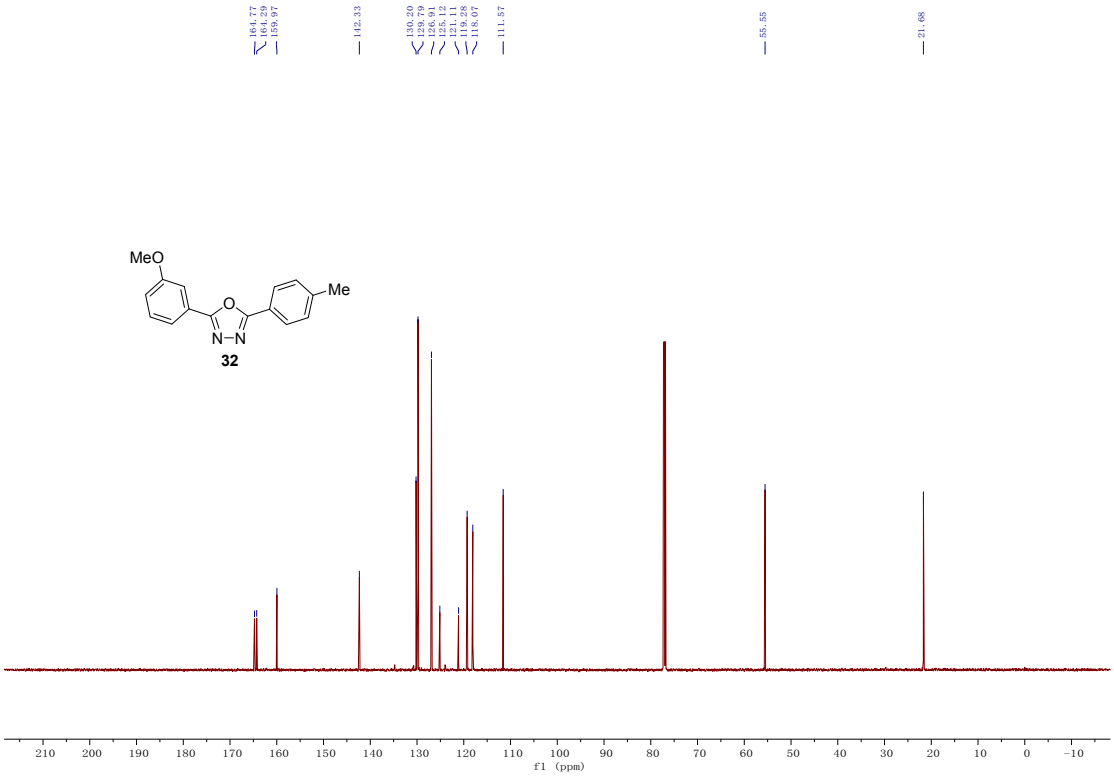

8.21
8.20
8.06
8.05
7.77
7.75
7.67
7.66
7.51
7.50
7.49
7.44
7.42
7.41
7.36
7.34
2.46
Ph
Me
O
N–N
30
2.00
2.02
2.03
2.01
1.00
2.01
3.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5
f1 (ppm)

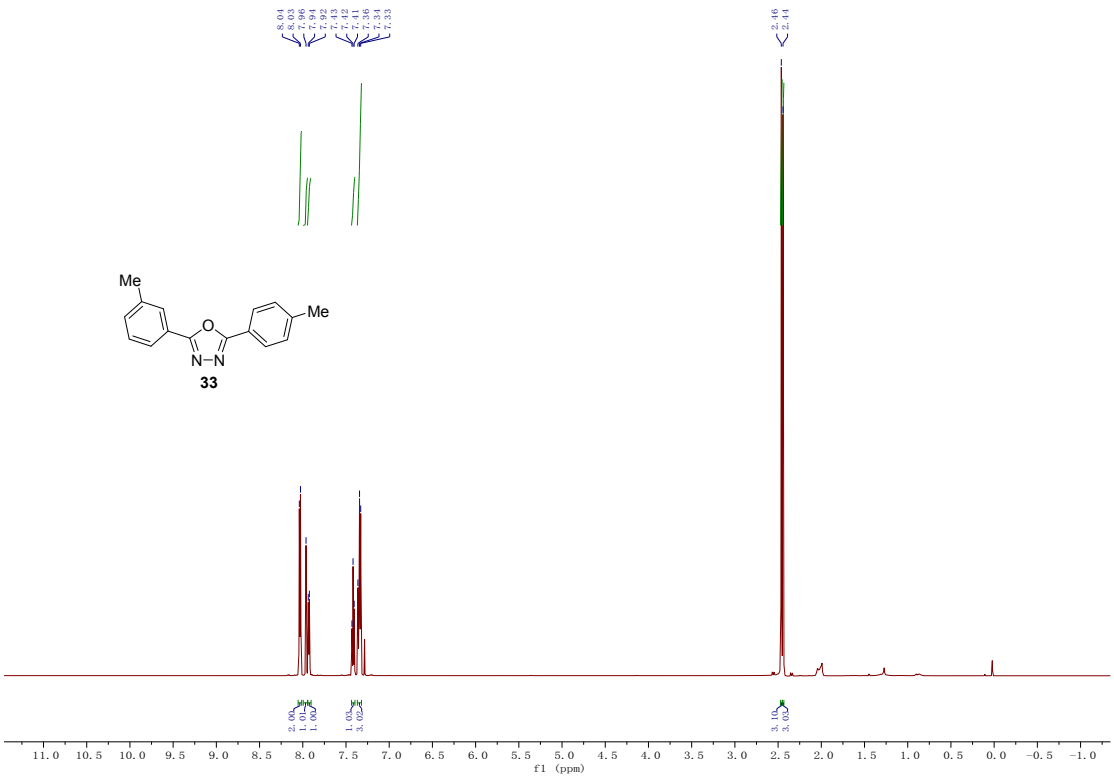

164.74
164.24
144.33
142.31
139.82
129.81
129.01
128.19
127.67
127.34
127.16
126.91
122.77
121.17
21.70
Ph
Me
O
N–N
30
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

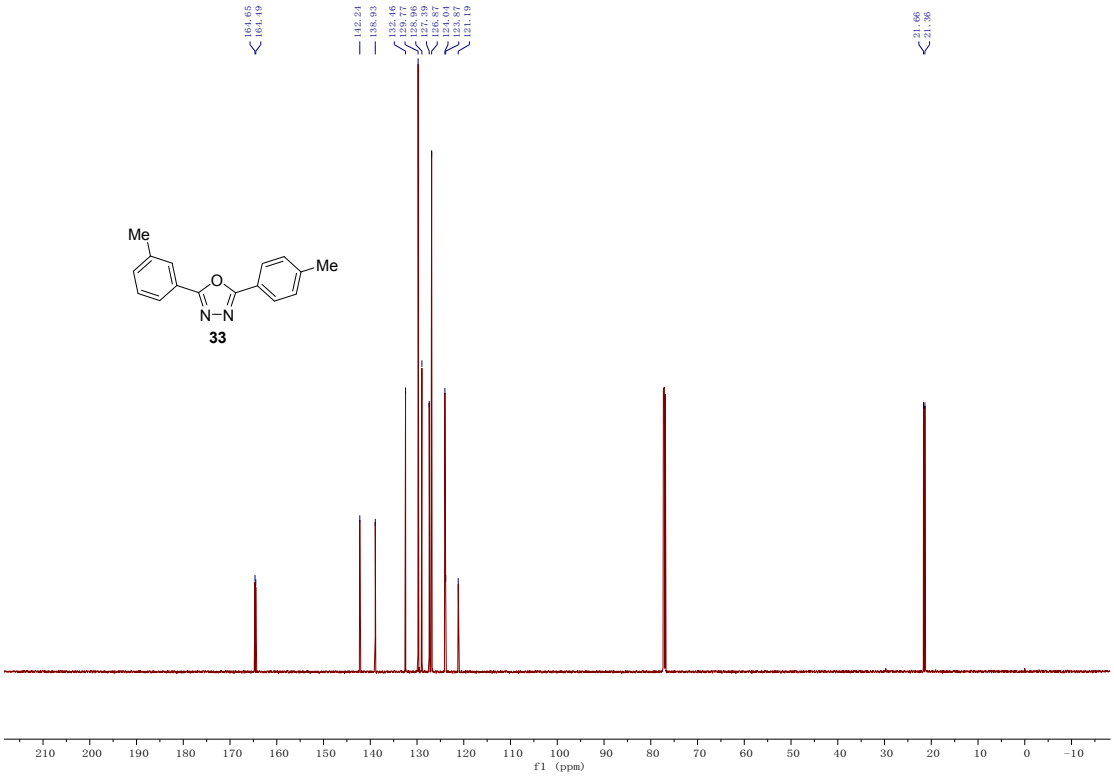

8.61
8.22
8.21
8.09
8.08
7.99
7.98
7.91
7.90
7.61
7.60
7.59
7.58
7.58
7.57
7.37
7.36
2.46
Me
O
N–N
31
1.00
1.01
2.01
2.02
1.02
2.00
2.00
3.01
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0
f1 (ppm)

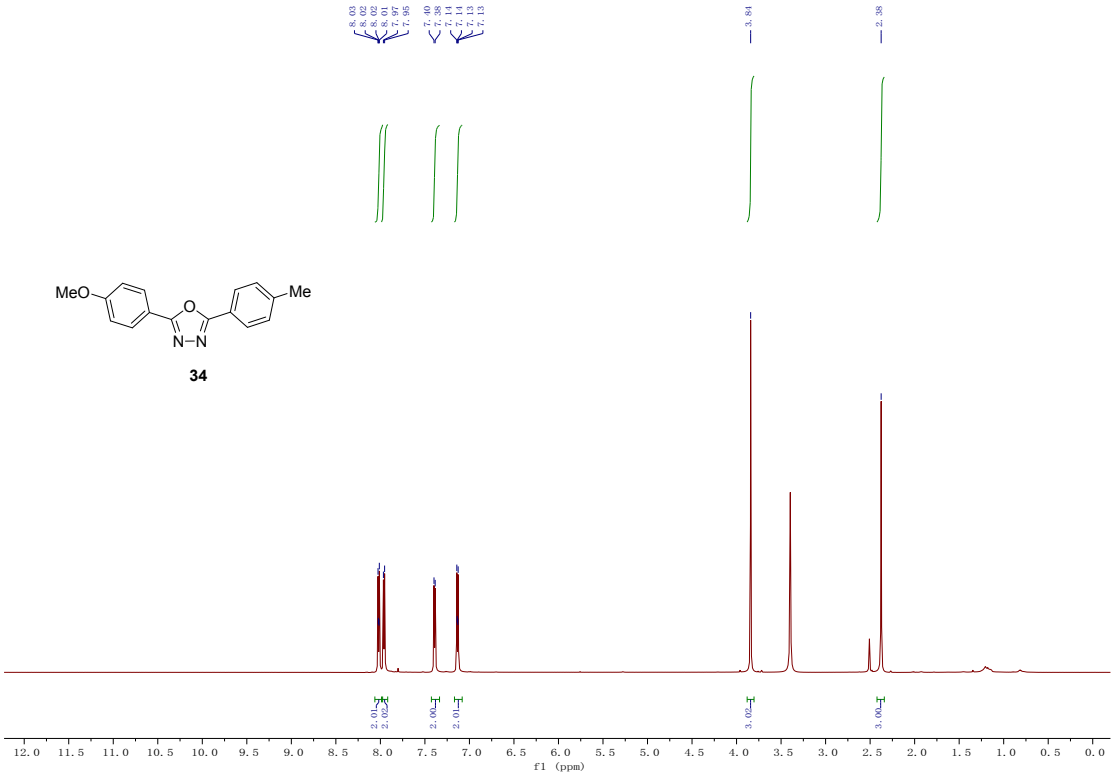

164.84
164.54
142.35
134.67
132.98
129.82
129.03
128.84
127.99
127.93
127.21
127.11
126.96
125.35
121.26
121.17
21.70
Me
O
N–N
31
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

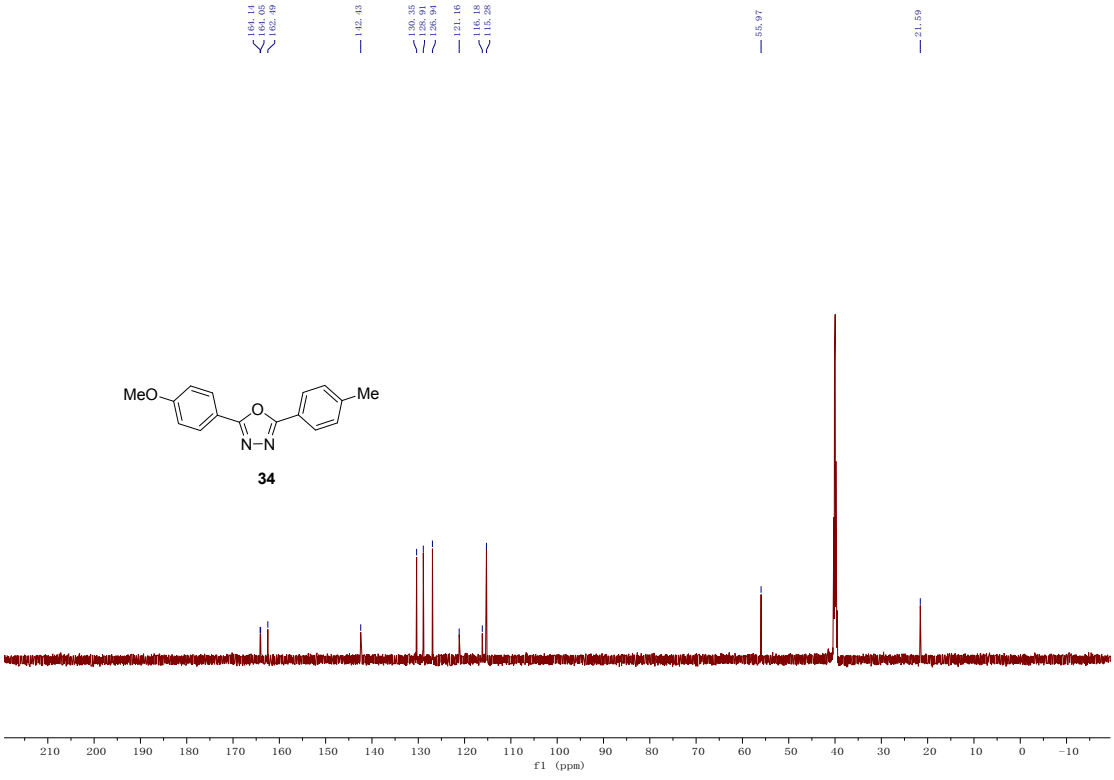

8.04
8.03
7.72
7.71
7.68
7.46
7.45
7.43
7.35
7.34
7.11
7.09
3.92
2.45
MeO
Me
O
N–N
32
2.00
1.00
1.00
2.02
1.00
3.07
3.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0
f1 (ppm)

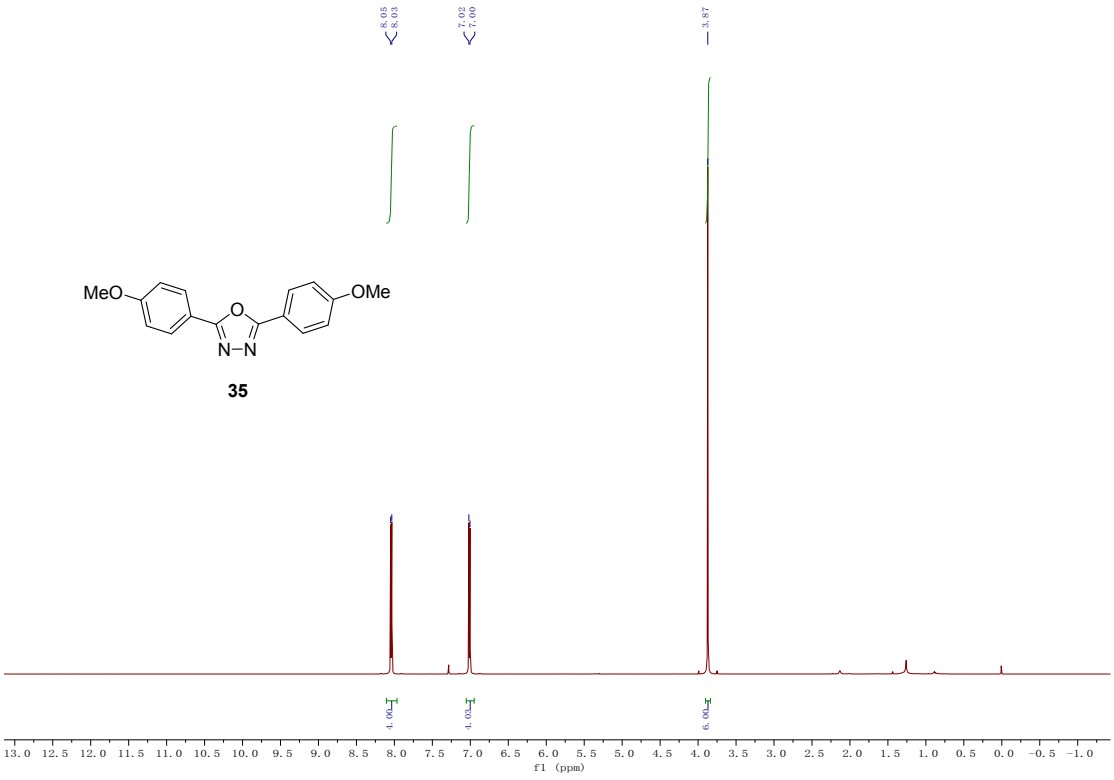

164.77
164.29
159.97
142.33
130.29
128.79
126.91
125.12
121.11
114.36
113.67
111.57
55.55
21.68
MeO
Me
O
N–N
32
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

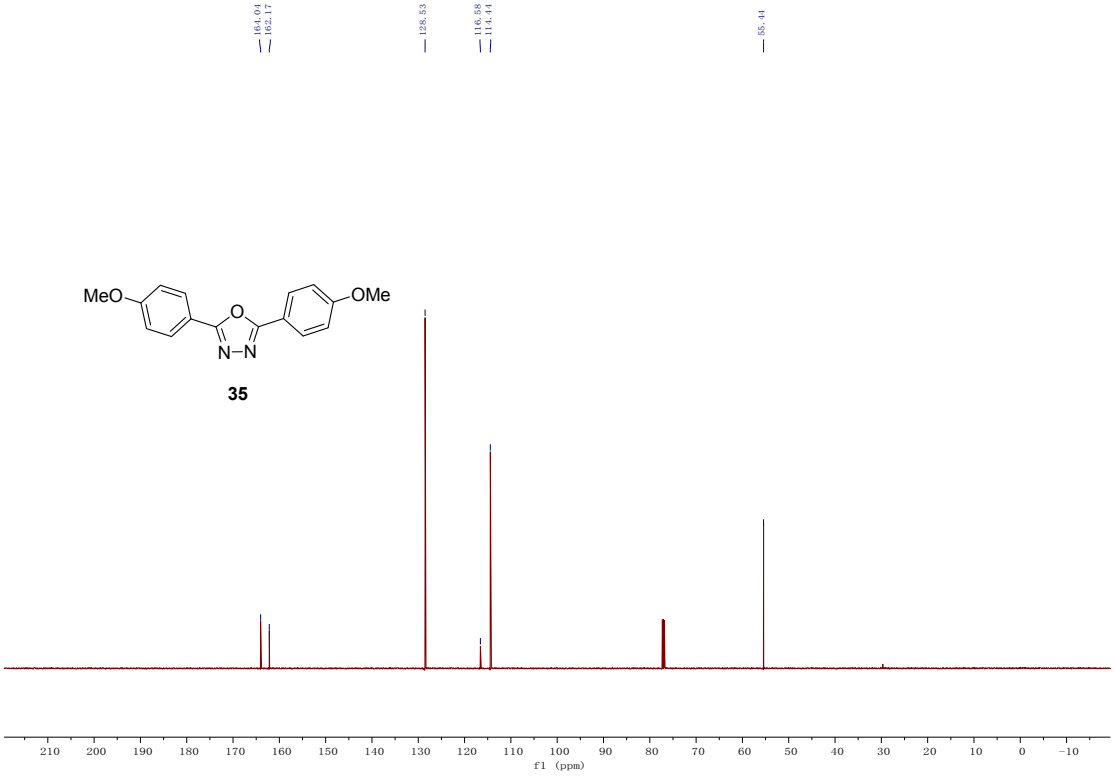

8.04
8.03
7.96
7.94
7.92
7.43
7.42
7.41
7.36
7.34
7.33
2.46
2.44
Me
Me
O
N–N
33
2.00
1.01
1.00
1.02
3.02
3.10
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0
f1 (ppm)

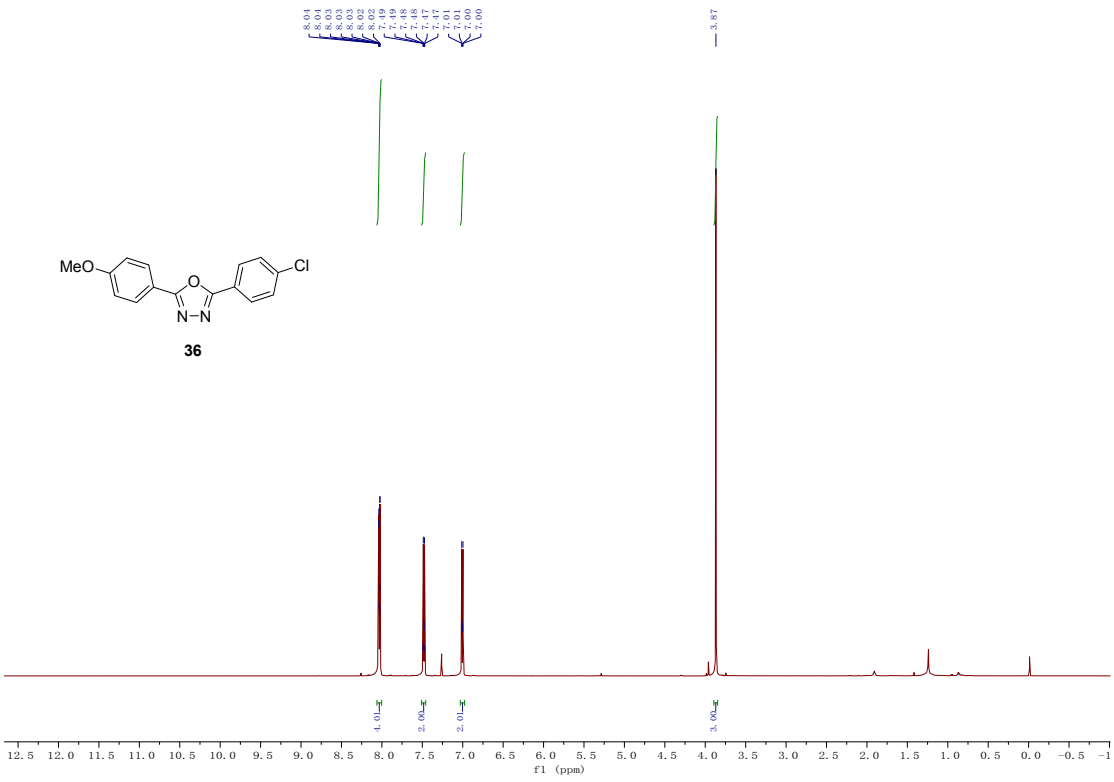

164.65
164.49
142.24
138.93
132.46
129.77
128.96
127.39
126.87
124.04
123.87
121.19
21.66
21.36
Me
Me
O
N–N
33
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

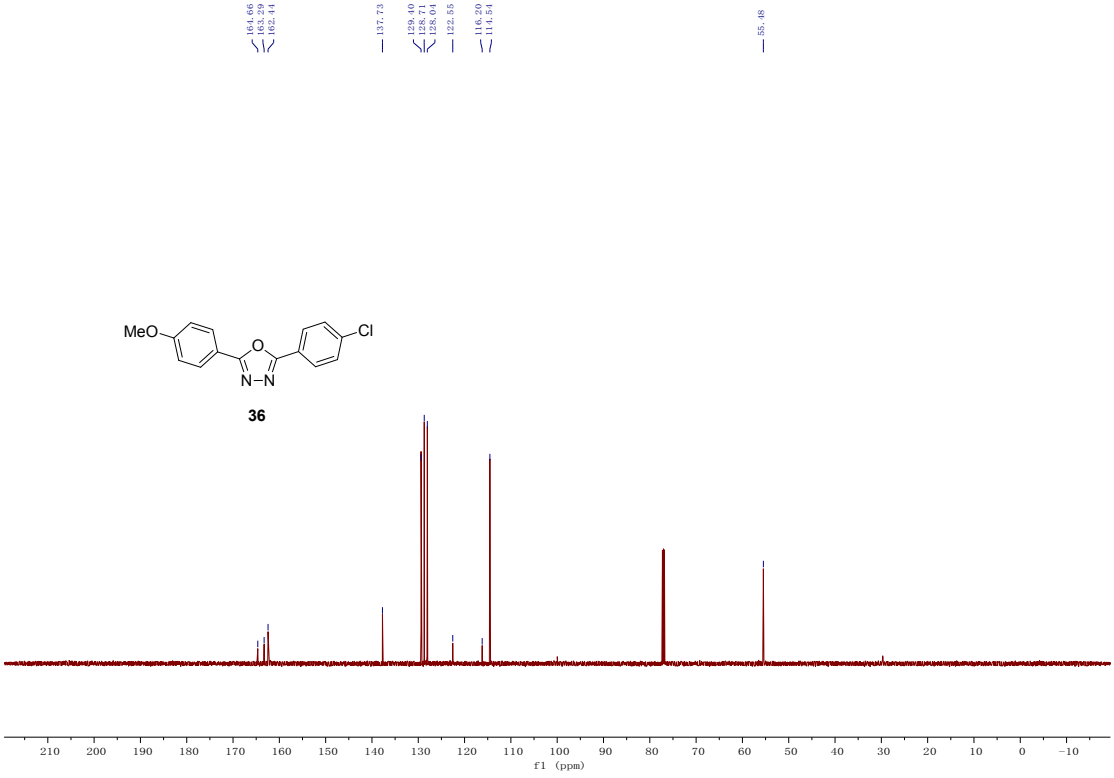

8.03
8.02
8.02
8.01
7.97
7.95
7.40
7.38
7.14
7.14
7.13
7.13
3.84
2.38
MeO
Me
O
N–N
34
2.01
2.02
2.00
2.01
3.02
3.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

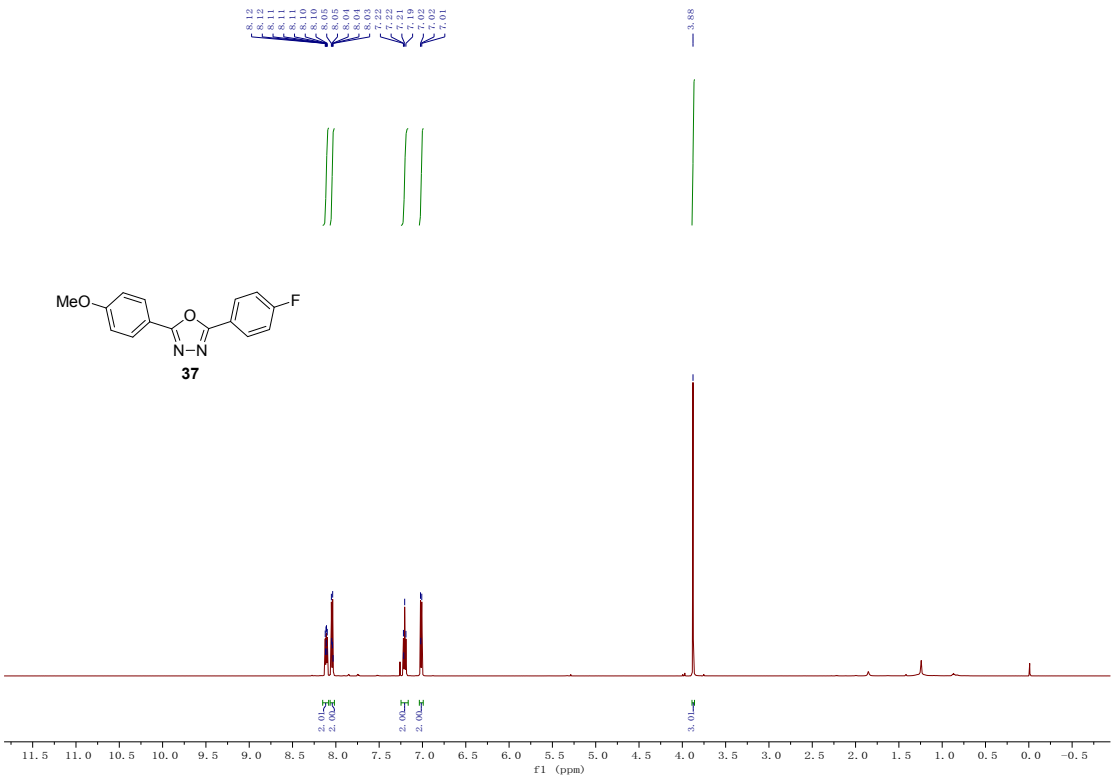

164.14
164.05
162.49
142.43
130.35
130.01
128.94
121.16
116.18
115.28
55.97
21.59
MeO
Me
O
N–N
34
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

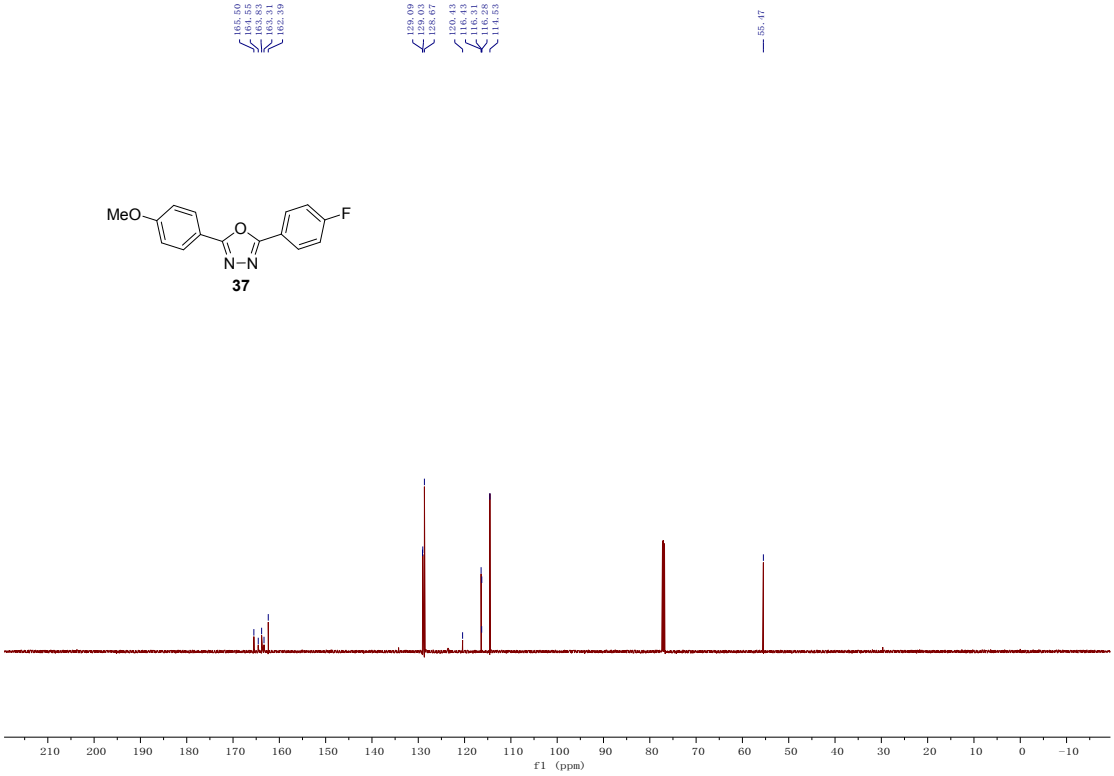

8.05
8.03
7.02
7.00
3.87
MeO
OMe
O
N–N
35
4.00
4.03
6.00
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0
f1 (ppm)

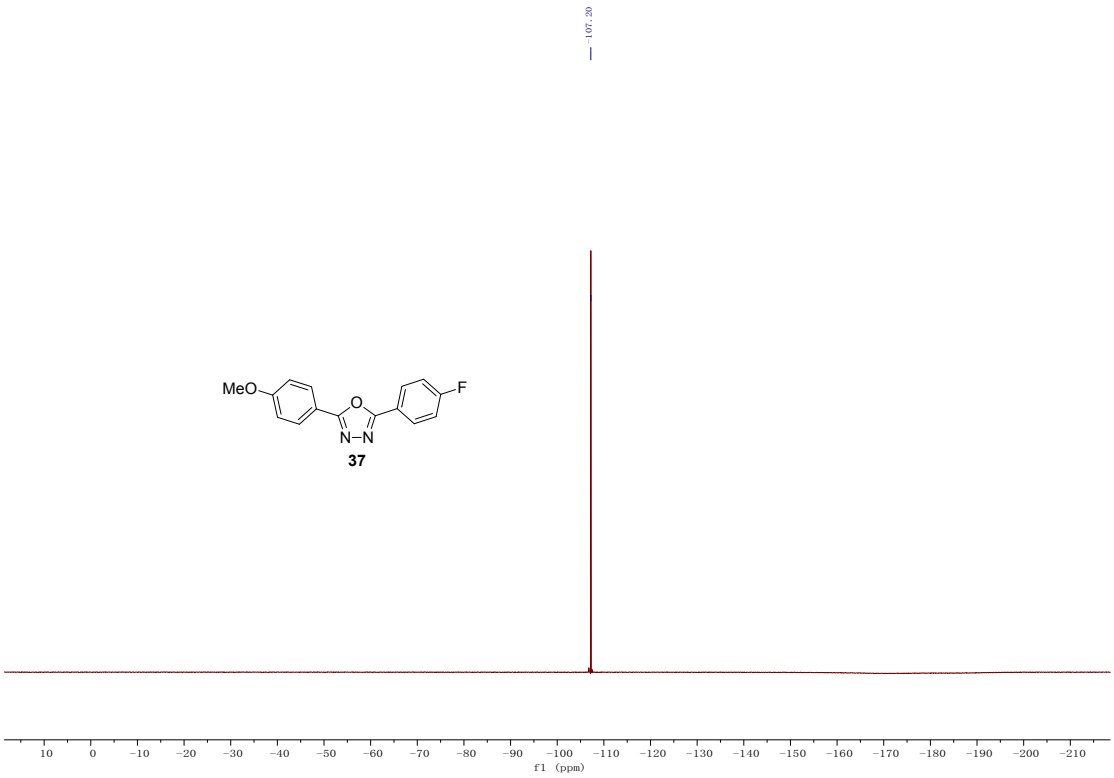

164.04
162.17
128.53
116.58
114.44
55.44
MeO
OMe
O
N–N
35
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

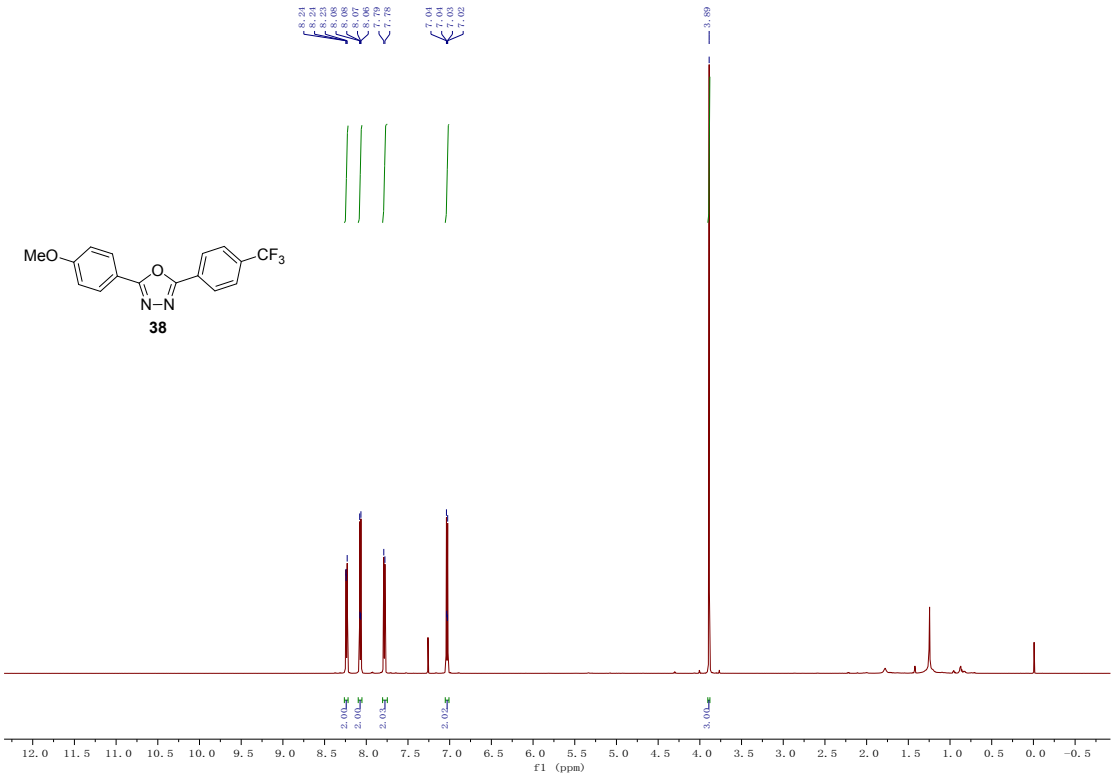

8.04
8.04
8.03
8.03
8.03
8.02
8.02
7.49
7.49
7.48
7.48
7.47
7.47
7.01
7.01
7.00
7.00
3.87
MeO
Cl
O
N–N
36
4.01
2.00
2.01
3.00
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1
f1 (ppm)

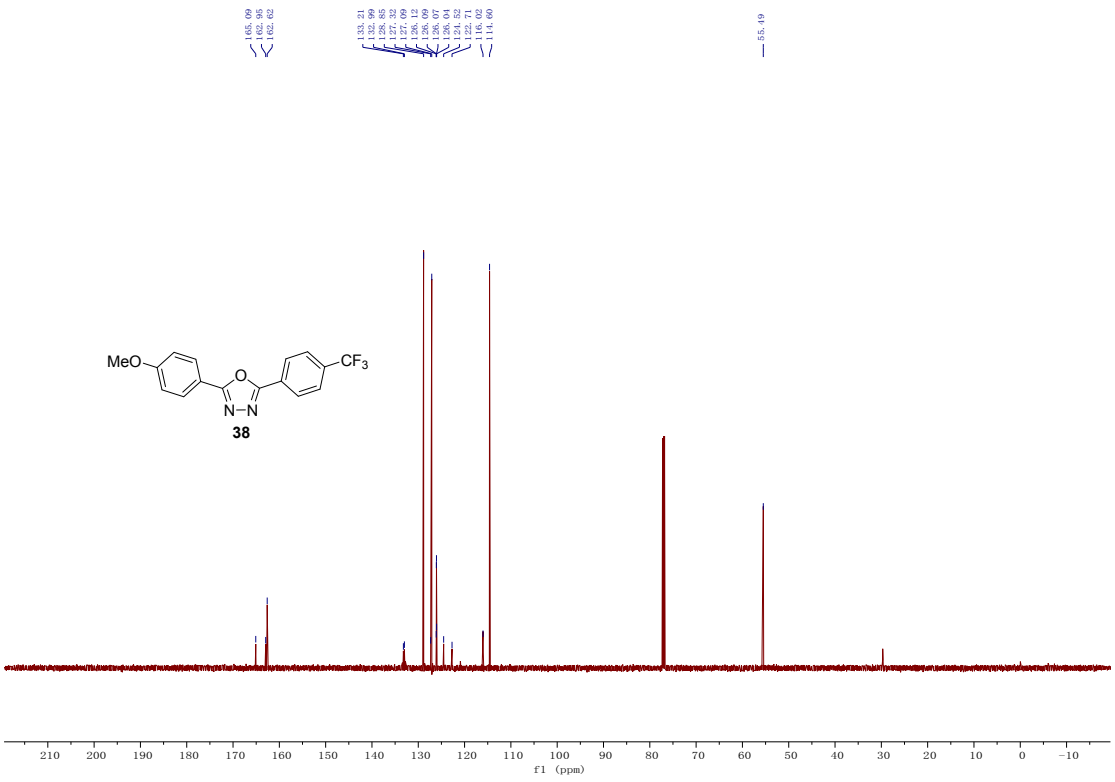

164.66
163.29
162.44
137.73
129.40
128.71
128.04
122.55
116.39
114.54
55.48
MeO
Cl
O
N–N
36
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

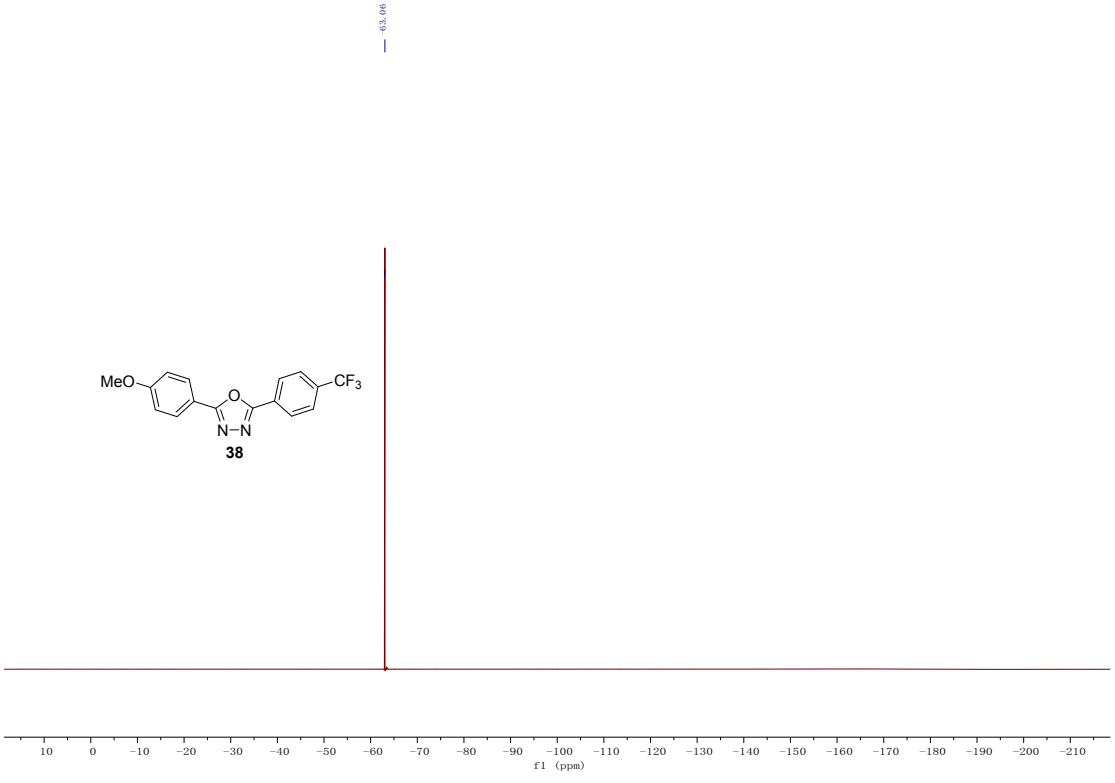

8.12
8.12
8.11
8.11
8.11
8.11
8.10
8.10
8.05
8.05
8.04
8.04
8.03
7.22
7.22
7.21
7.19
7.02
7.02
7.01
3.88
MeO
O
N–N
F
37
2.01
2.00
2.00
2.00
3.01
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5
f1 (ppm)

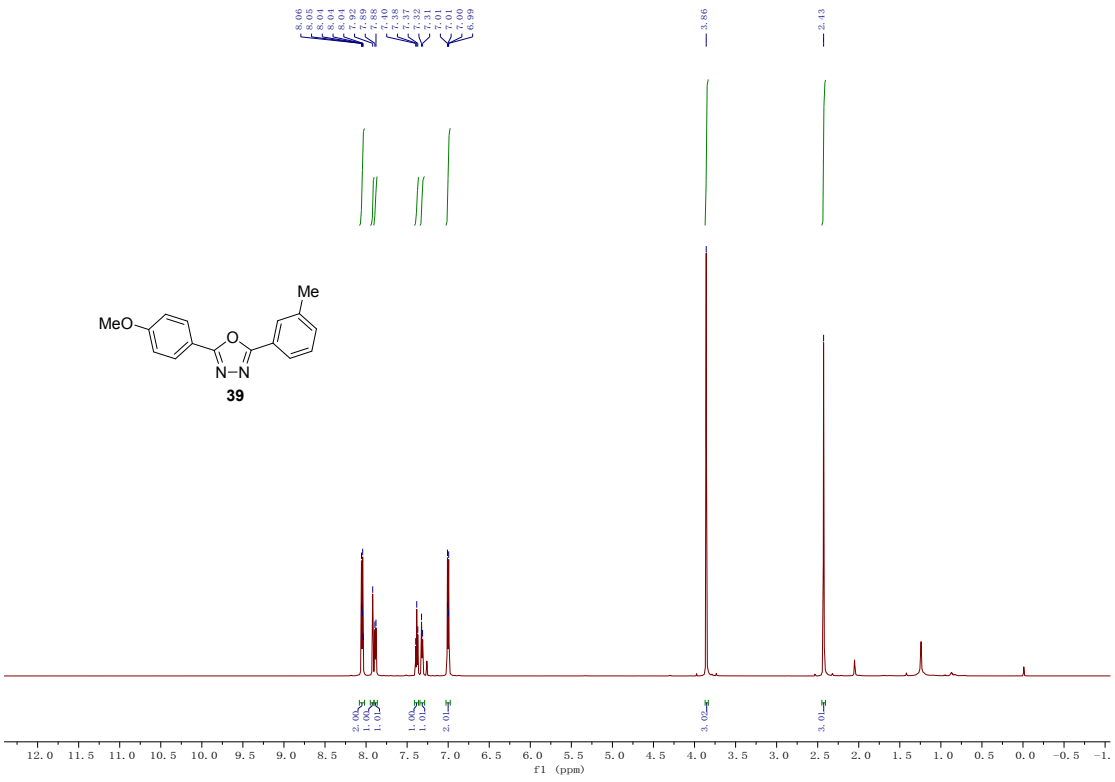

165.50
164.55
163.83
163.31
162.38
129.09
128.03
128.67
120.43
116.43
116.31
116.28
114.53
55.47
MeO
O
N–N
F
37
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

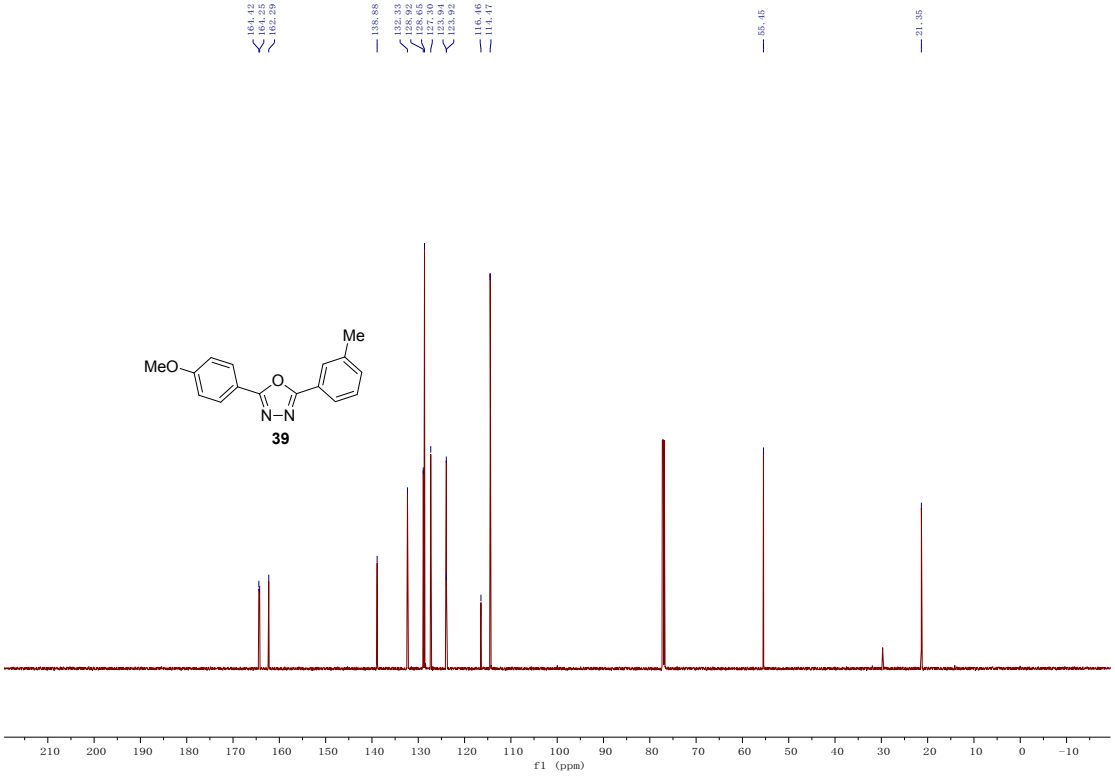

-107.20
MeO
F
O
N–N
37
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)

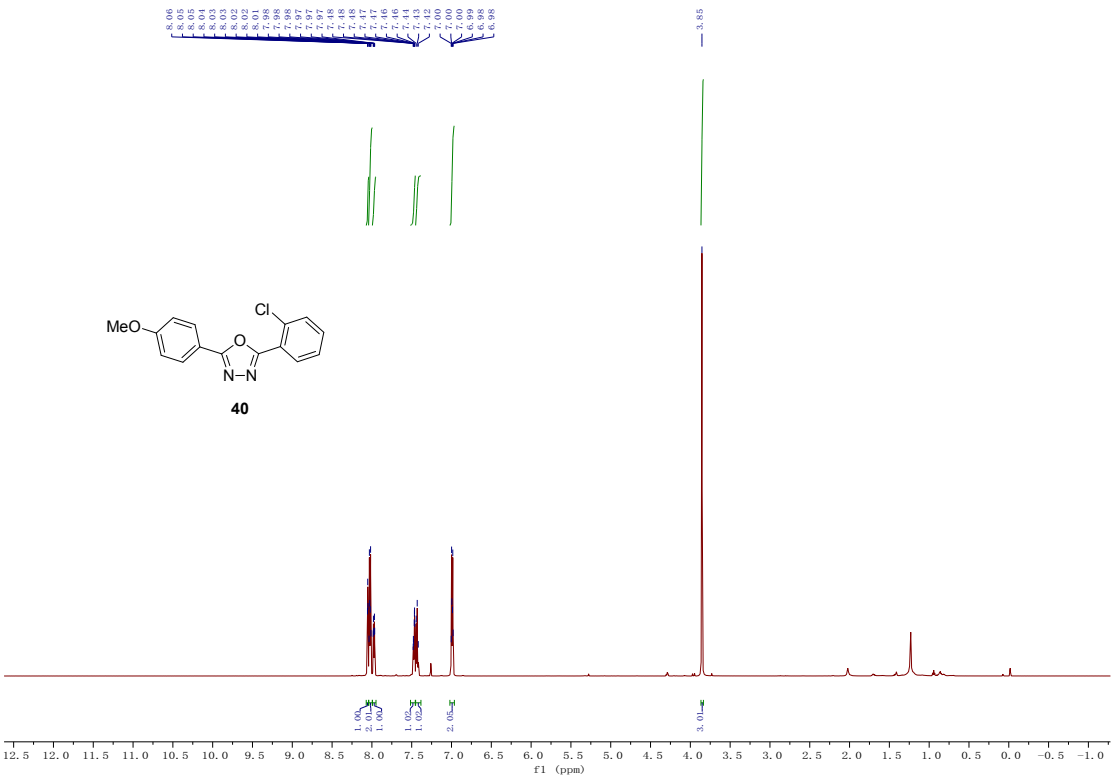

8.24
8.24
8.23
8.08
8.07
8.06
7.79
7.78
7.04
7.04
7.03
7.02
3.89
MeO
CF3
O
N–N
38
2.01
2.01
2.03
2.02
3.01
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5
f1 (ppm)

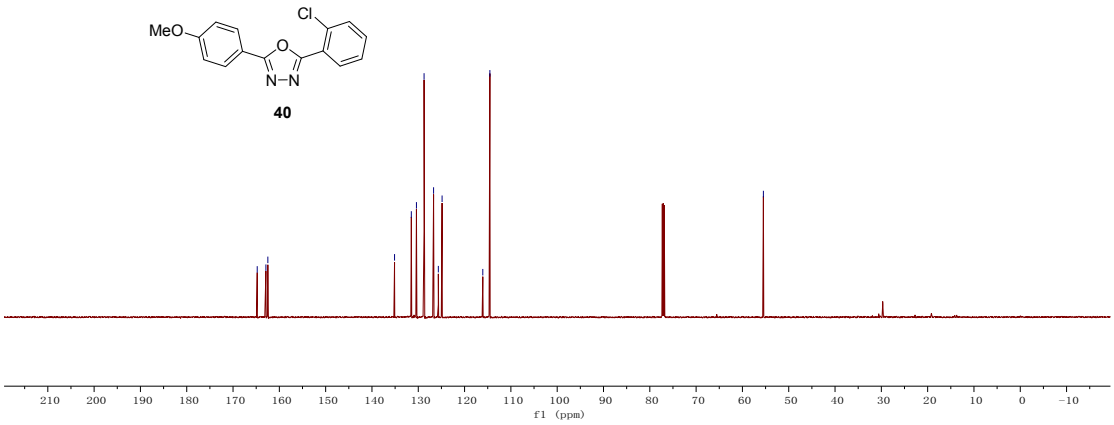

165.09
162.95
162.62
133.21
132.99
128.85
127.32
127.09
126.12
126.09
126.07
126.04
124.52
122.71
114.69
114.60
55.49
MeO
CF3
O
N–N
38
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

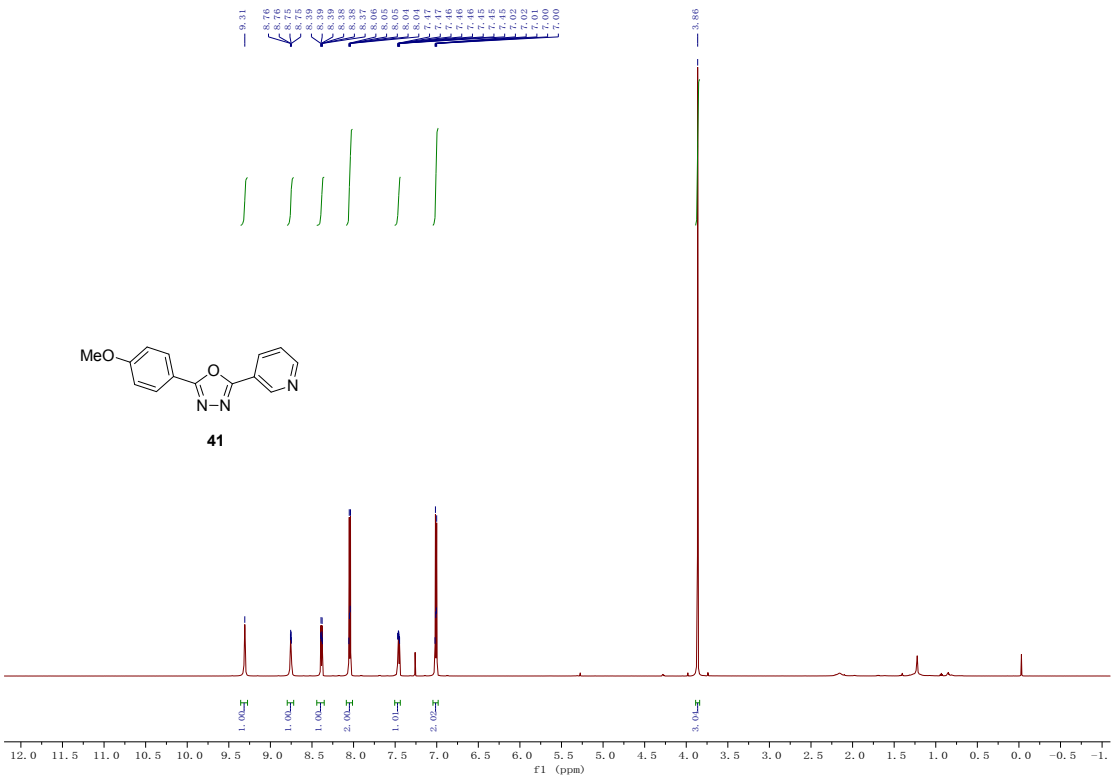

-63.06
MeO
CF3
O
N–N
38
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)

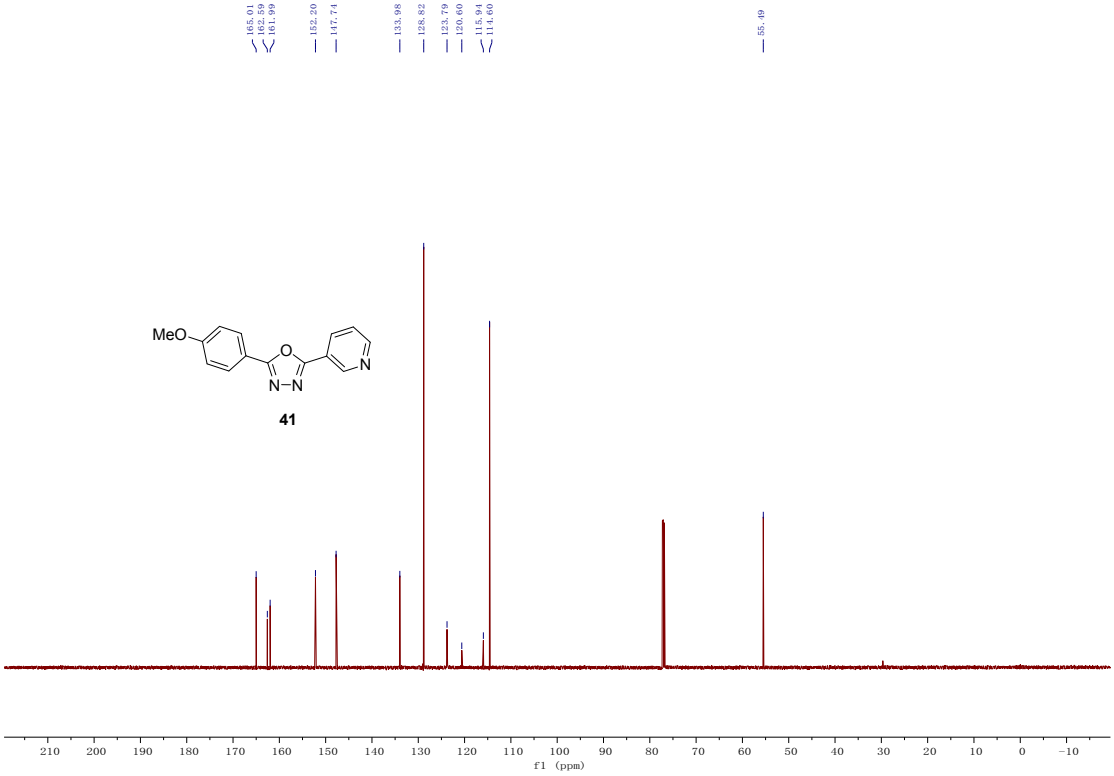

8.06
8.05
8.04
8.04
8.04
8.04
7.92
7.89
7.88
7.40
7.38
7.37
7.32
7.31
7.01
7.01
7.00
6.99
3.86
2.43
MeO
Me
O
N–N
39
2.00
1.00
1.00
1.00
2.01
3.02
3.01
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.
f1 (ppm)

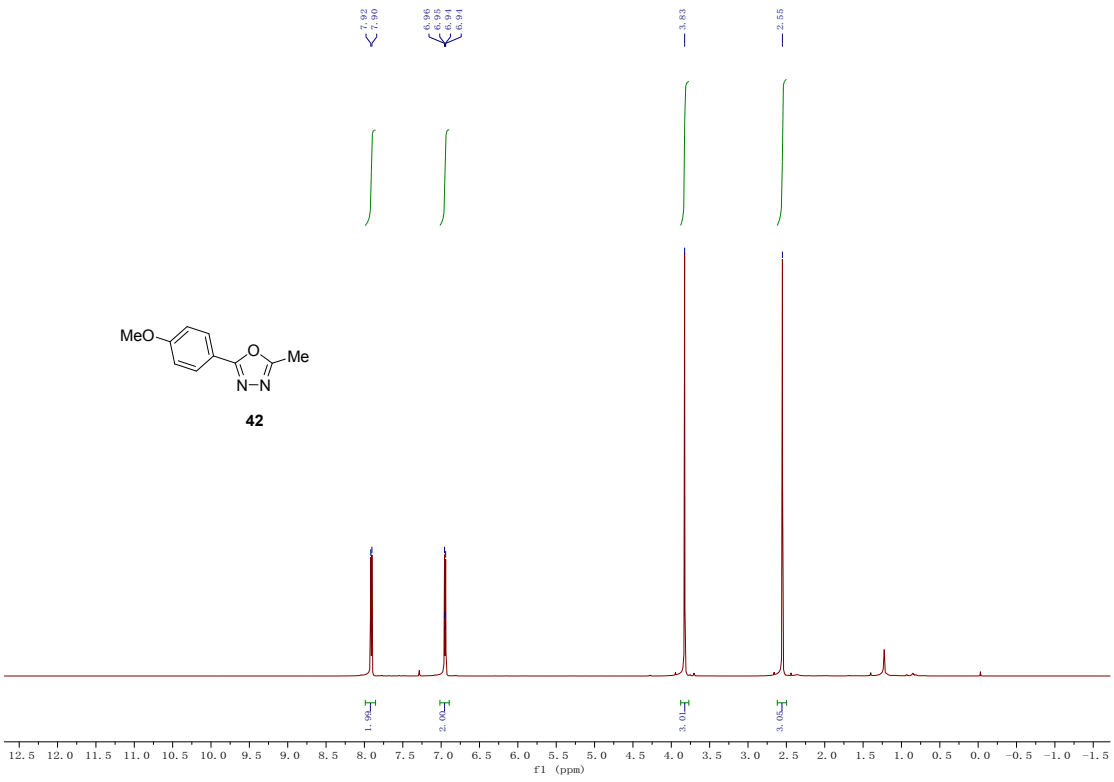

164.42
164.25
162.29
138.88
132.33
129.02
128.65
127.30
123.94
123.92
116.46
114.47
55.45
21.35
MeO
Me
O
N–N
39
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

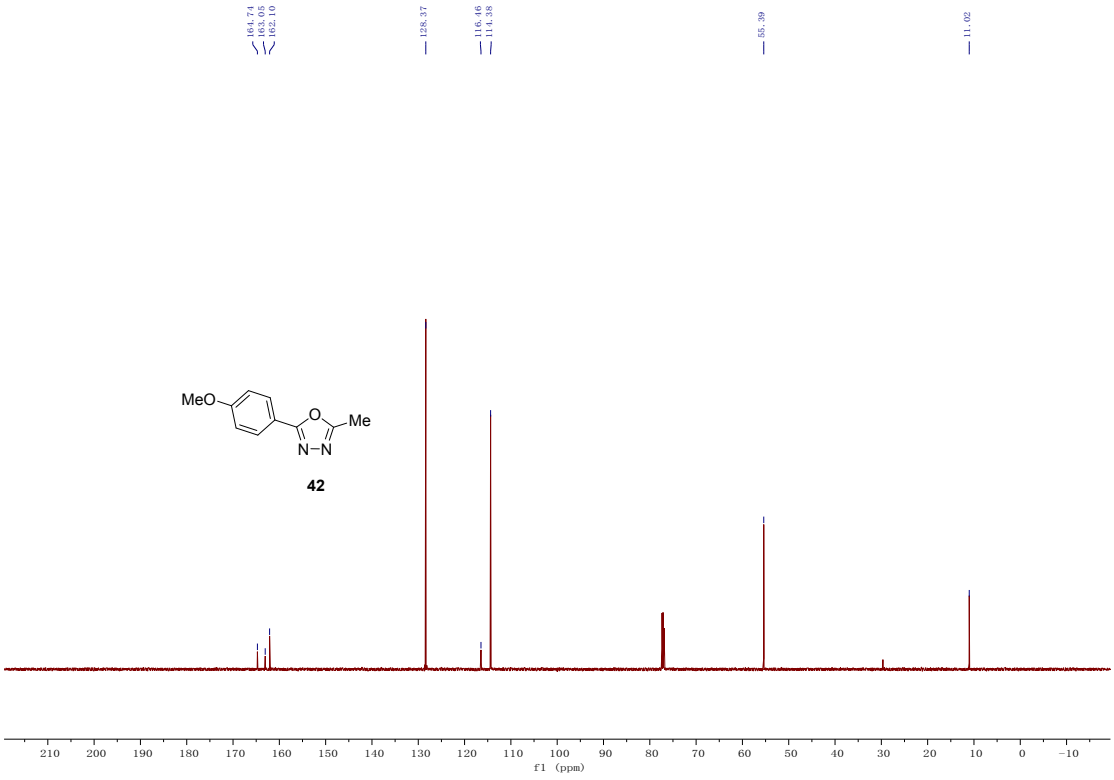

MeO
Cl
O
N–N
40
f1 (ppm)

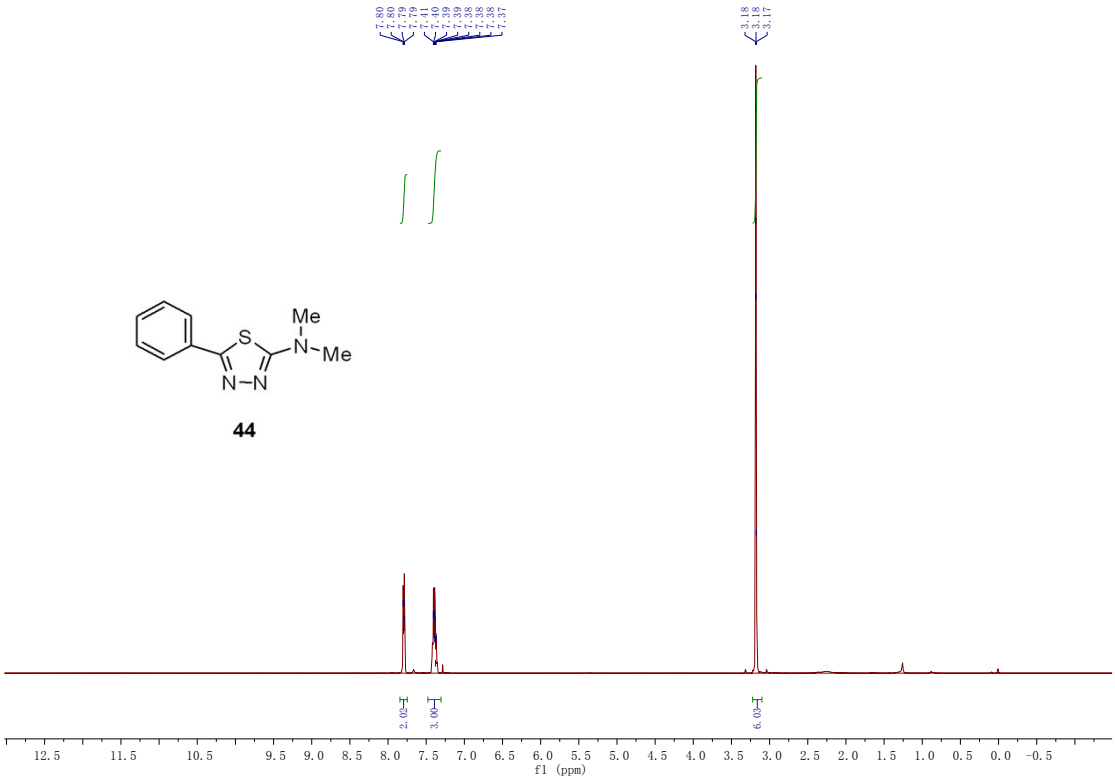

MeO
Cl
O
N–N
40
f1 (ppm)


MeO
O
N–N
N
41
f1 (ppm)

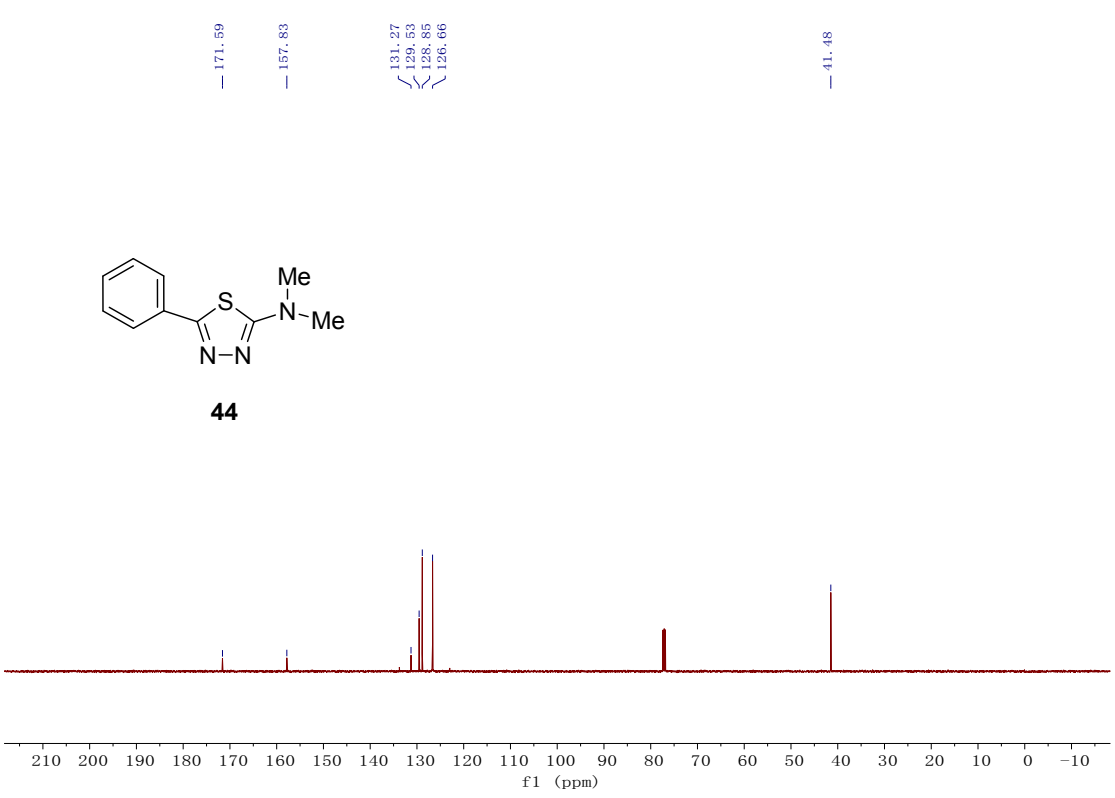

MeO
O
N–N
N
41
f1 (ppm)


7.92
7.90
6.96
6.95
6.94
6.94
3.83
2.55
MeO
O
Me
N–N
42
1.99
2.00
3.01
3.05
f1 (ppm)

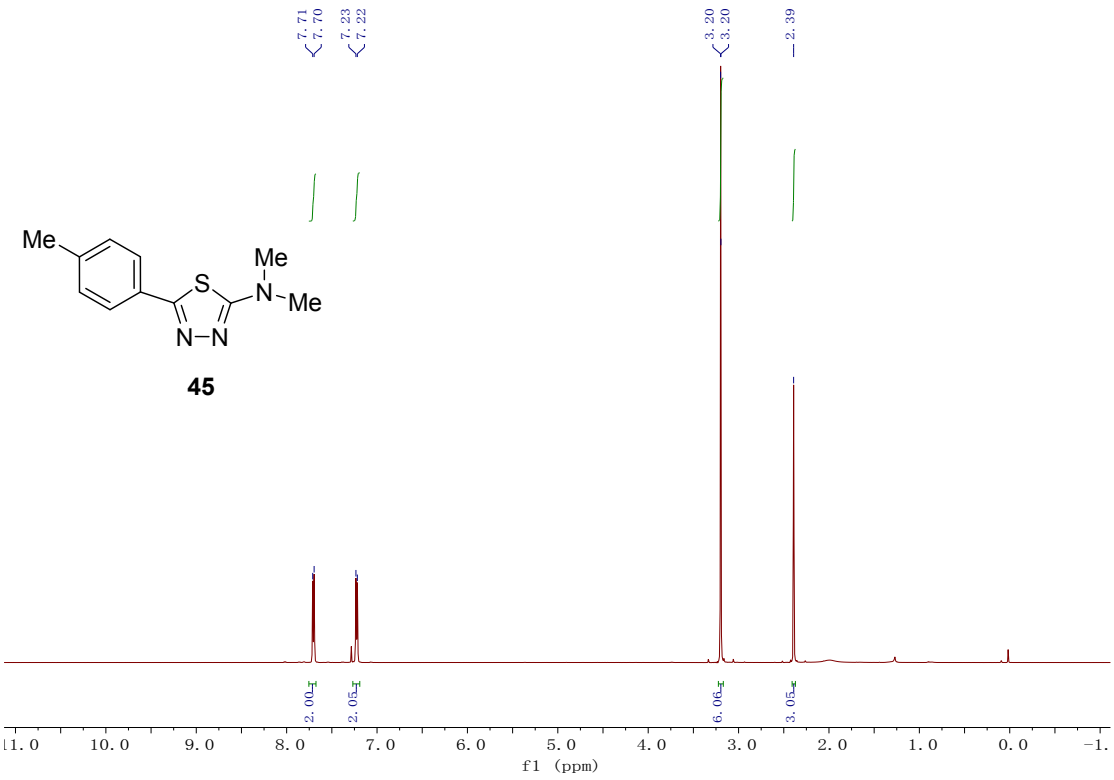

164.74
163.05
162.10
128.37
116.46
114.38
55.39
11.02
MeO
O
Me
N–N
42
f1 (ppm)

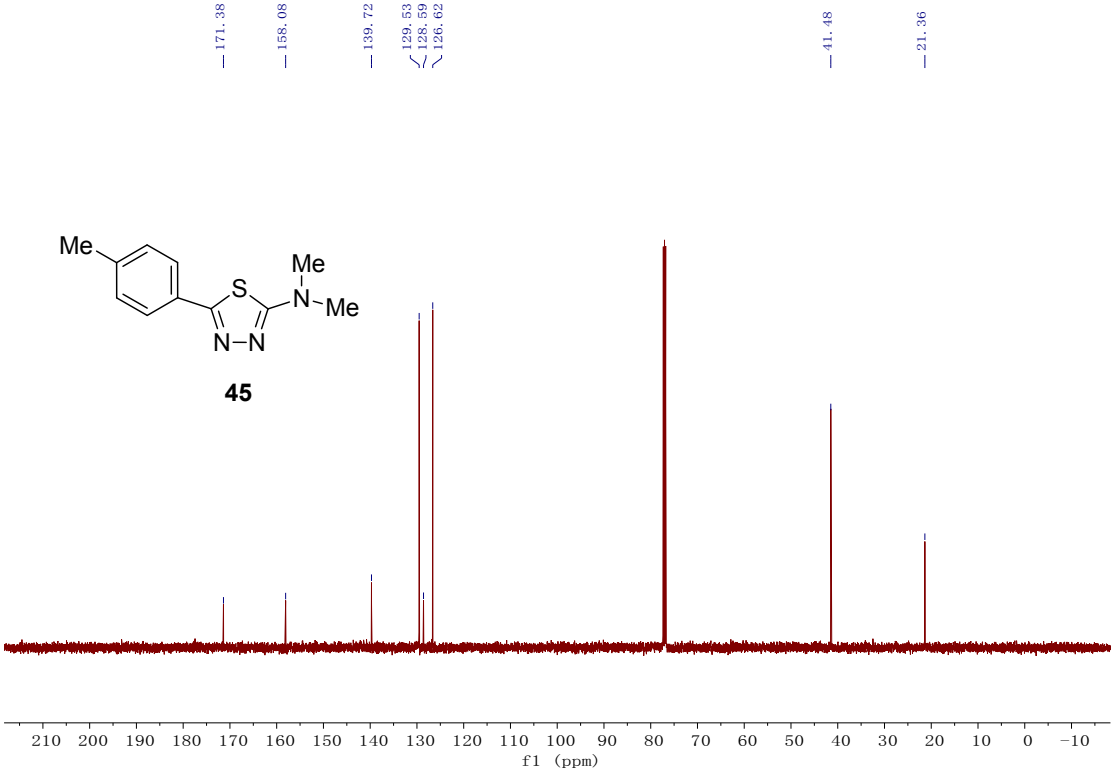

7.80
7.80
7.79
7.79
7.41
7.40
7.39
7.39
7.38
7.38
7.38
7.37
3.18
3.18
3.17
Me
N
Me
S
N
N
2.00
3.00
6.09
12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

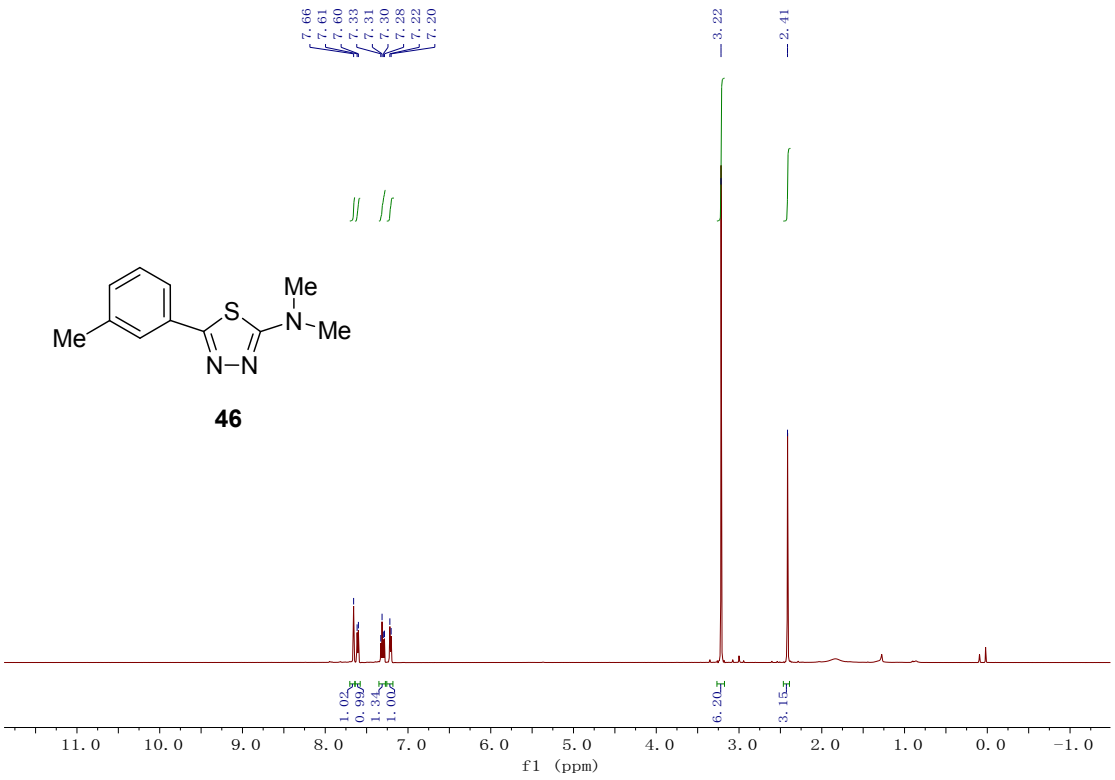
**44**


171.59
157.83
131.27
129.53
128.85
126.66
41.48
Me
N
Me
S
N
N
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)

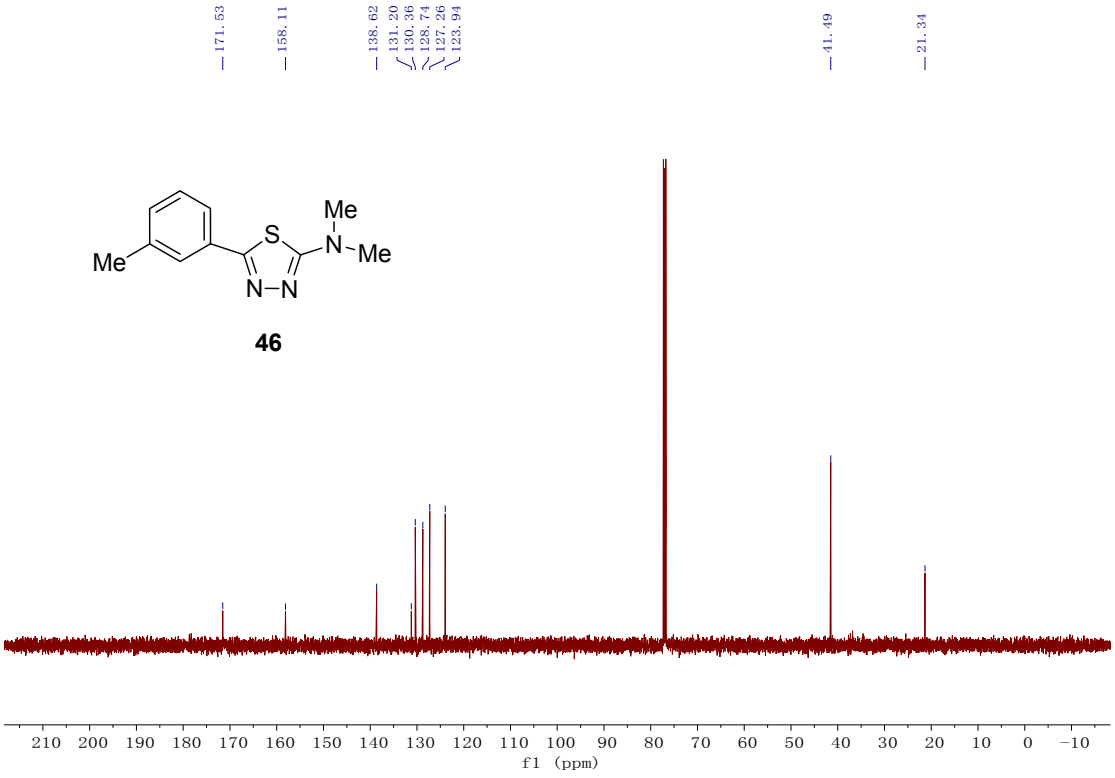
**44**


7.71
7.70
7.23
7.22
3.20
3.20
2.39
Me
S
Me
N
Me
N–N
45
2.00
2.05
6.06
3.05
f1 (ppm)

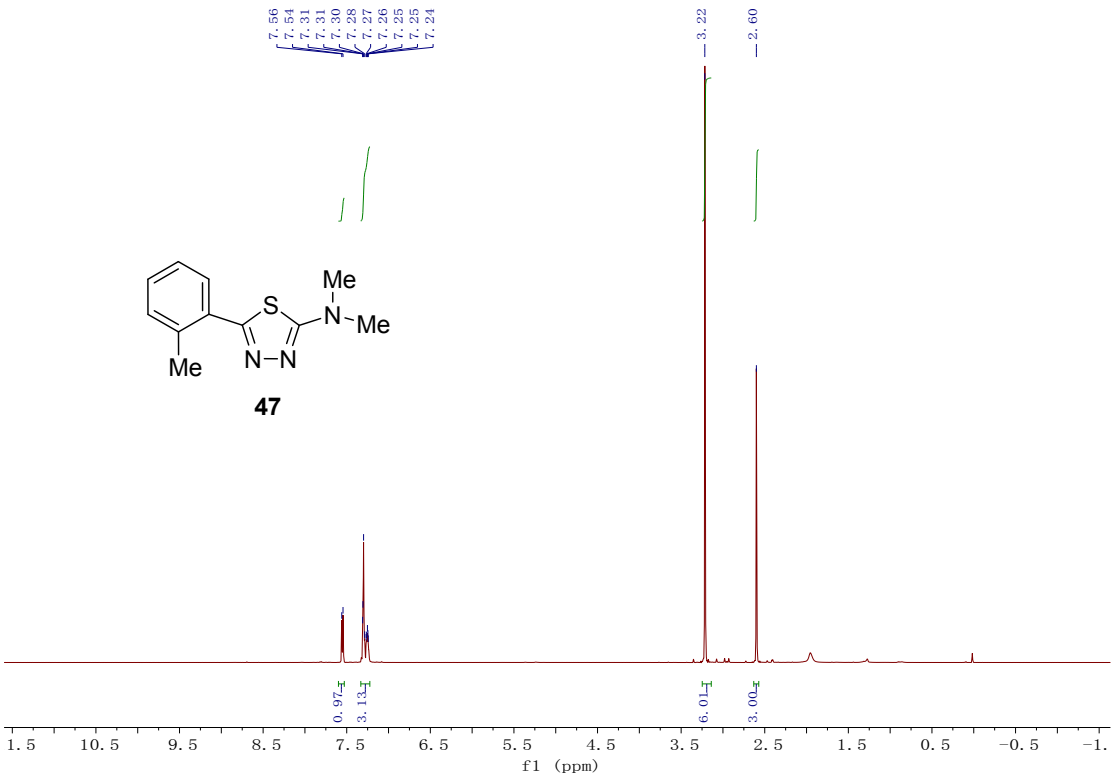

171.38
158.08
139.72
129.53
128.59
126.62
41.48
21.36
Me
S
Me
N
Me
N–N
45
f1 (ppm)


7.66
7.61
7.60
7.33
7.31
7.30
7.28
7.22
7.20
3.22
2.41
Me
S
N
Me
Me
N
N
1.02
0.99
1.34
1.00
6.20
3.15
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
−1.0
f1 (ppm)

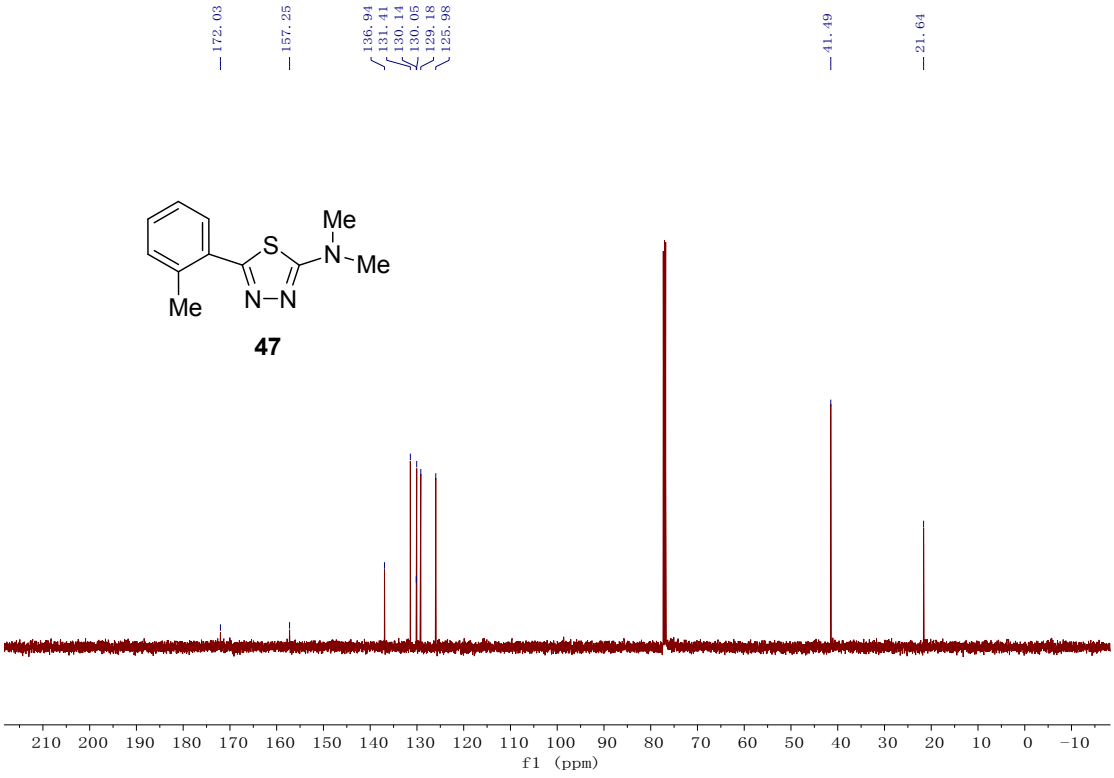
**46**


171.53
158.11
138.62
131.20
130.36
128.74
127.26
123.94
41.49
21.34
Me
S
N
Me
Me
N
N
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)

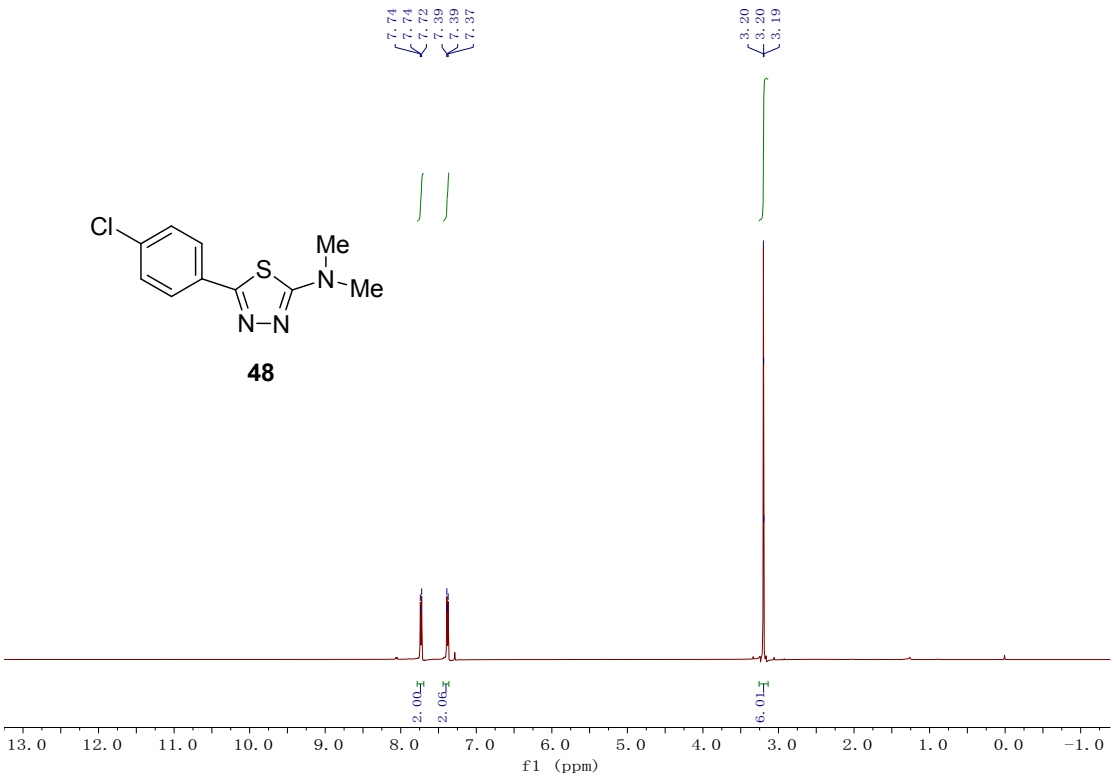
**46**


7.56
7.54
7.31
7.31
7.30
7.28
7.27
7.26
7.25
7.25
7.24
3.22
2.60
Me
S
N
Me
N–N
Me
0.97
3.13
6.01
3.00
f1 (ppm)

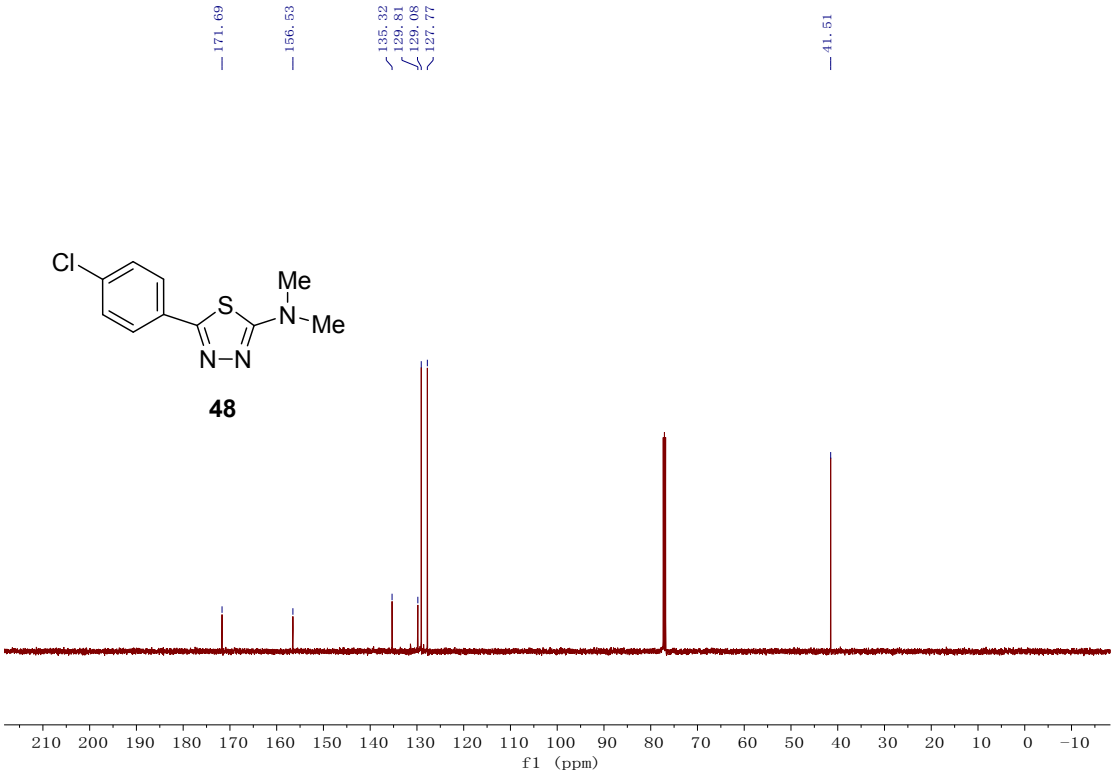
**47**


172.03
157.25
136.94
131.41
130.14
130.05
129.18
125.98
41.49
21.64
Me
S
N
Me
N–N
Me
f1 (ppm)

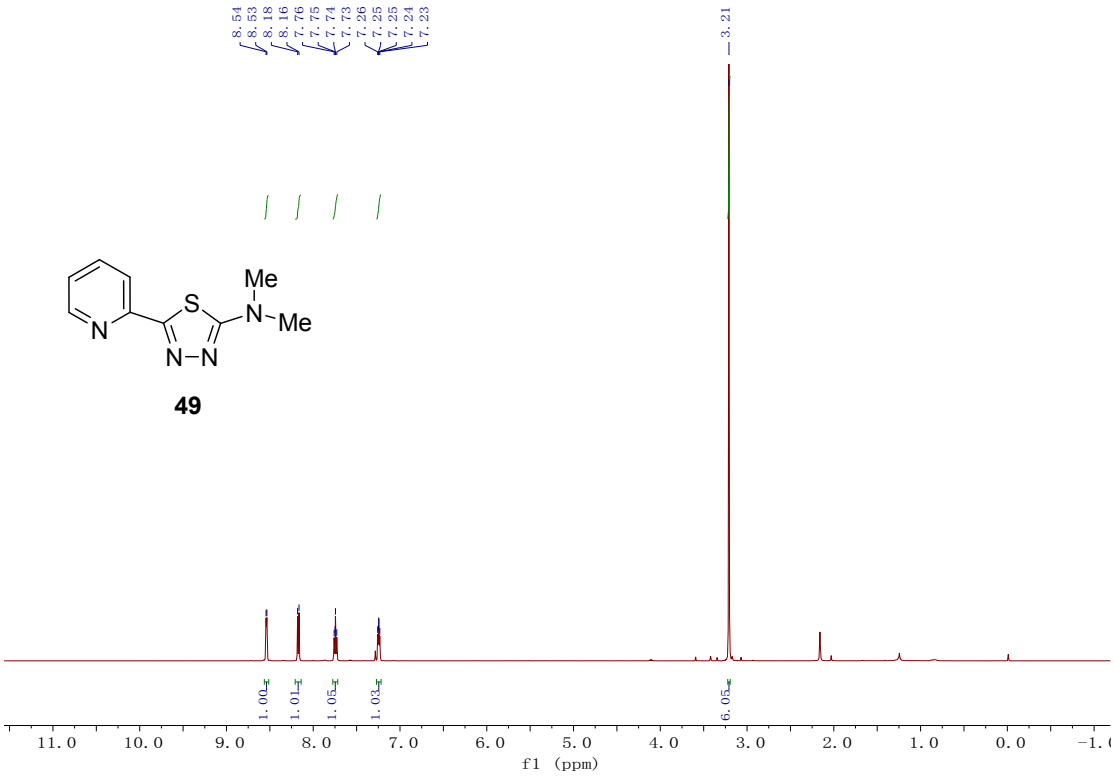
**47**


7.74
7.74
7.72
7.39
7.39
7.37
3.20
3.20
3.19
Cl
S
Me
N
Me
N–N
2.00
2.06
6.01
13.0 12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 −1.0
f1 (ppm)

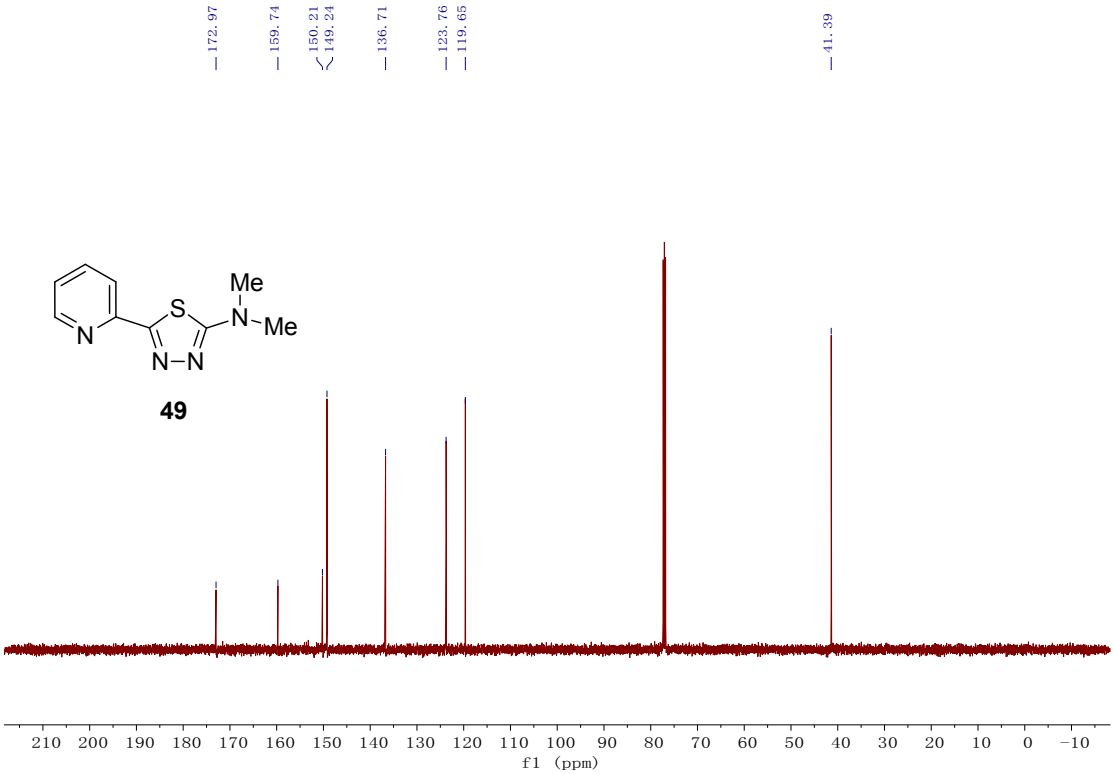
**48**


171.69
156.53
135.32
129.81
129.08
127.77
41.51
Cl
S
Me
N
Me
N–N
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)

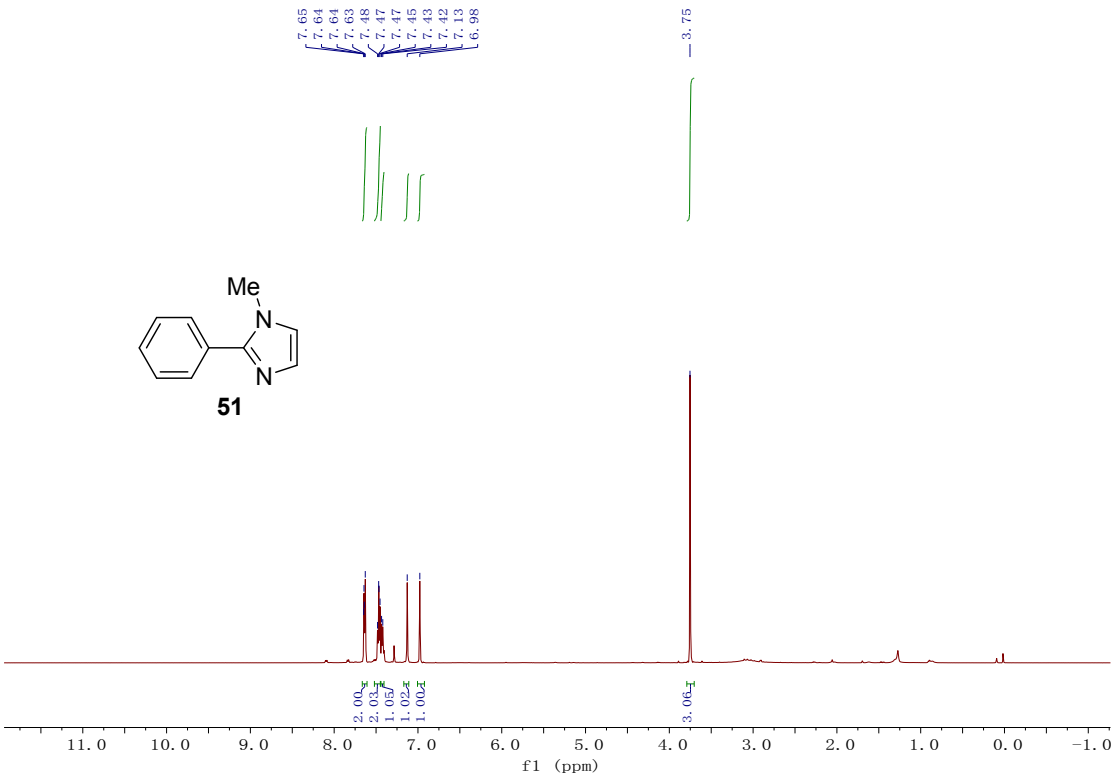
**48**

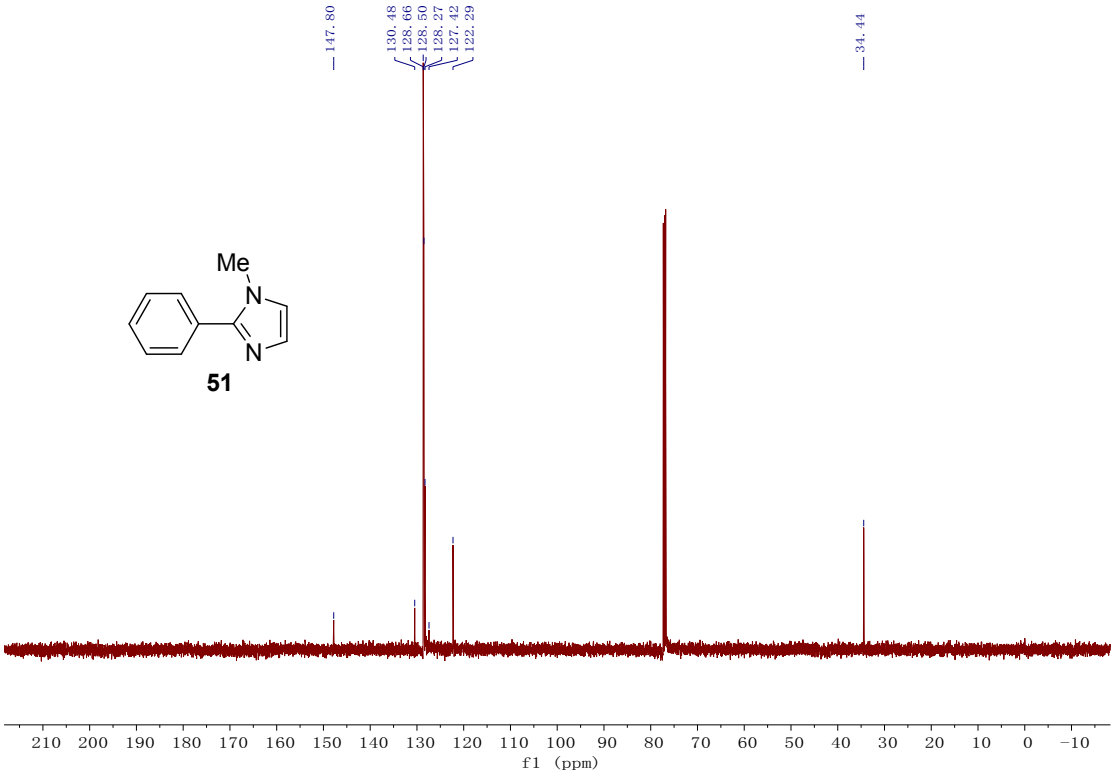

8.54
8.53
8.18
8.16
7.76
7.75
7.74
7.73
7.26
7.25
7.25
7.24
7.23
3.21
Me
N
S
N
N
N
Me
1.00
1.01
1.05
1.03
6.05
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

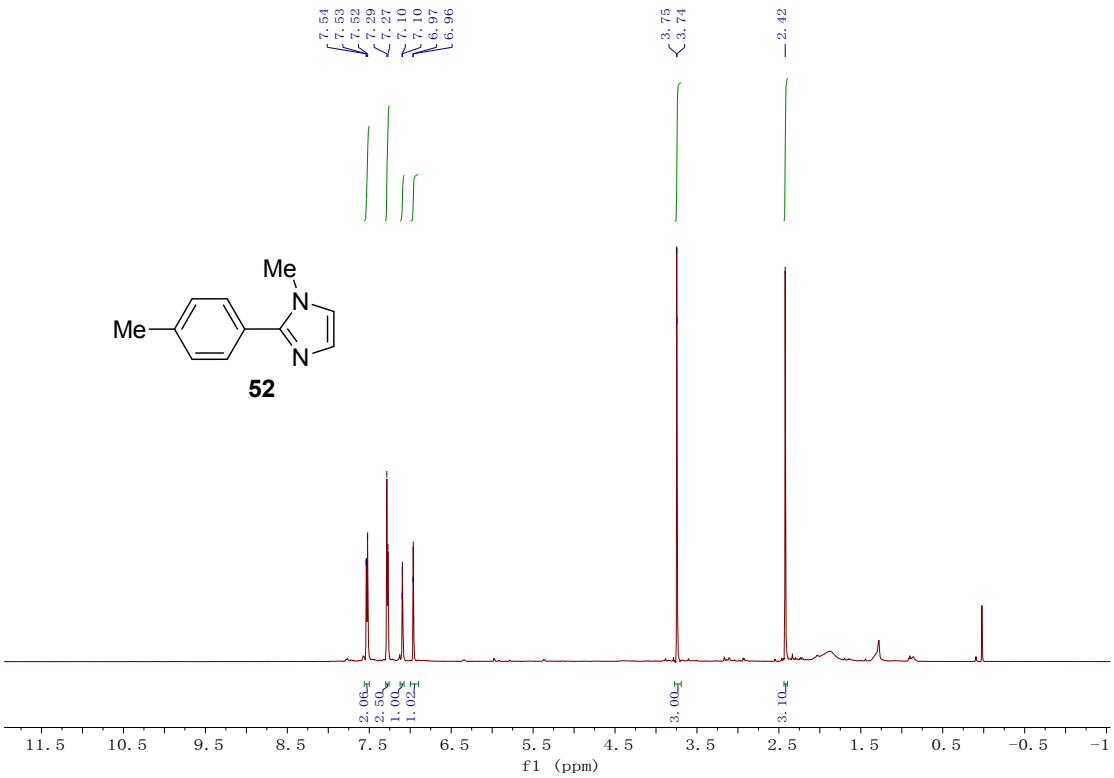
**49**

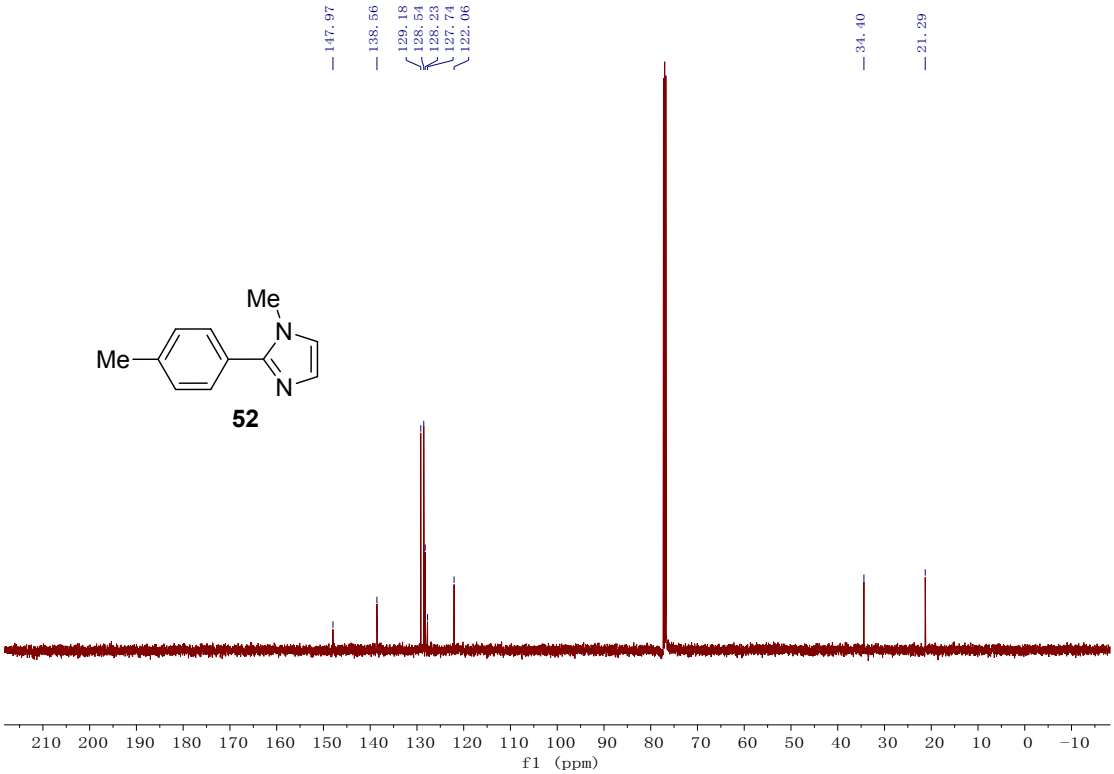

172.97
159.74
150.21
149.24
136.71
123.76
119.65
41.39
Me
N
S
N
N
N
Me
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

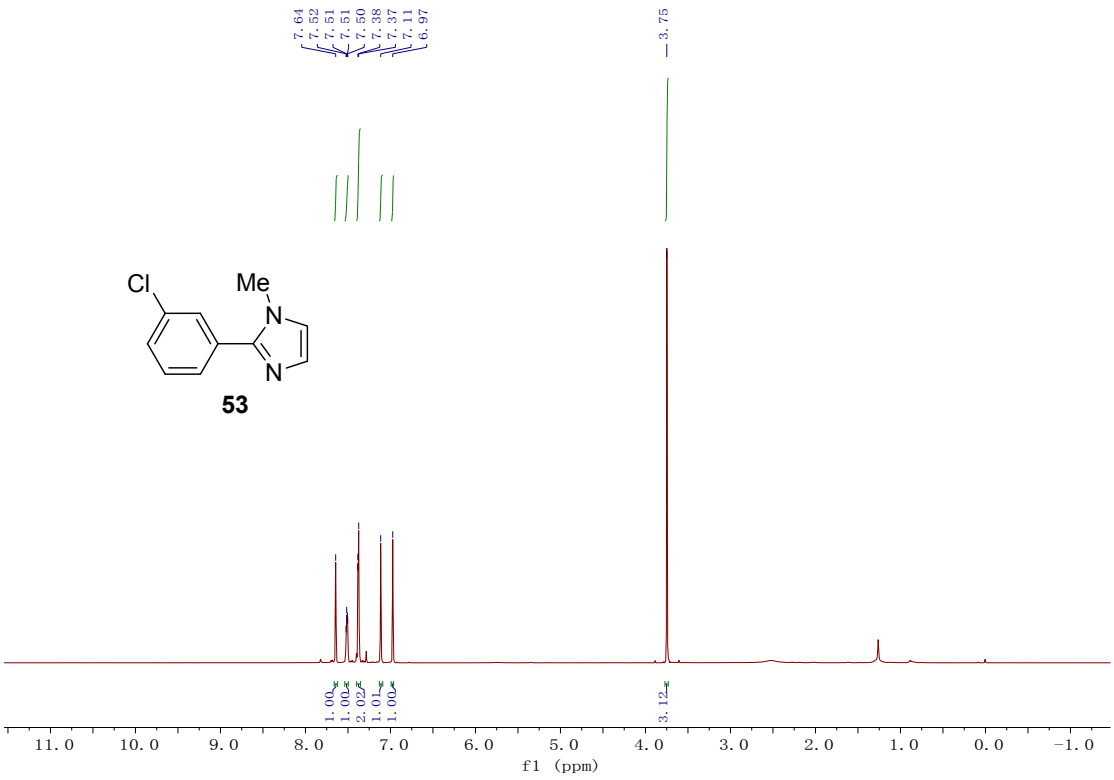
**49**

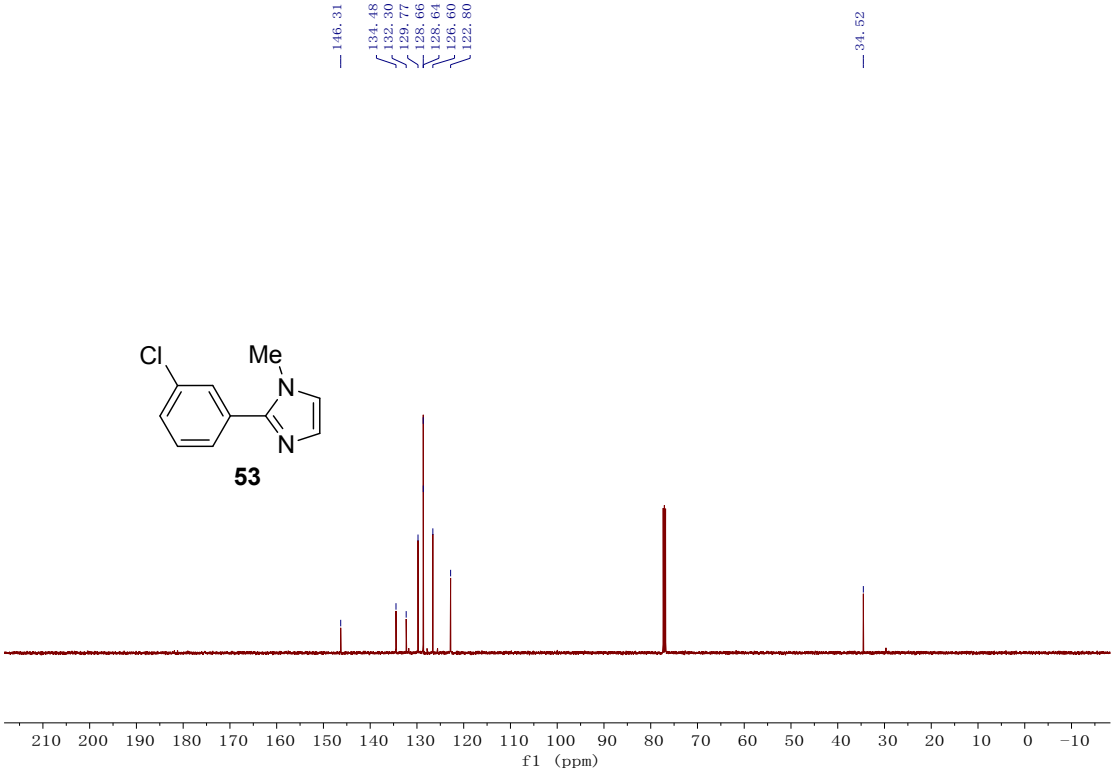

7.65
7.64
7.64
7.63
7.48
7.47
7.47
7.45
7.43
7.42
7.13
6.98
3.75
Me
N
N
51
2.00
2.03
1.05
1.02
1.00
3.06
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

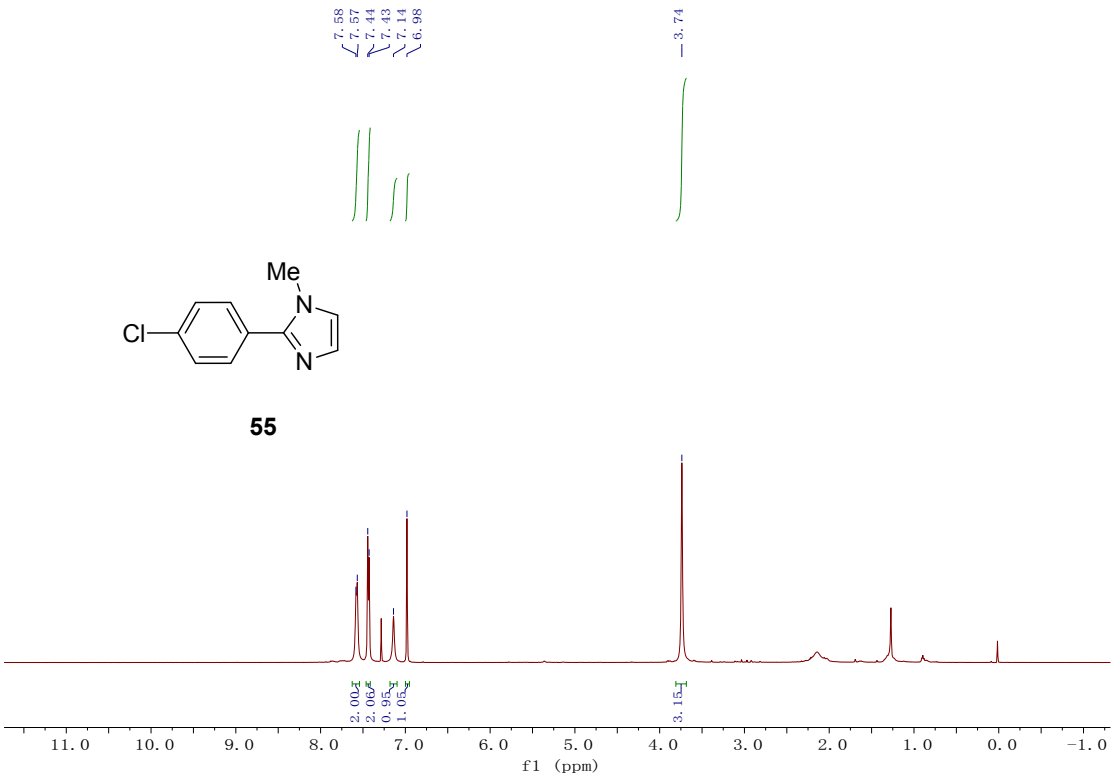

147.80
130.48
128.66
128.50
128.27
127.42
122.29
34.44
Me
N
N
51
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.54
7.53
7.52
7.29
7.27
7.10
6.97
6.96
3.75
3.74
2.42
Me
N
Me
N
52
2.06
2.50
1.00
1.02
3.00
3.10
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
−0.5
−1
f1 (ppm)

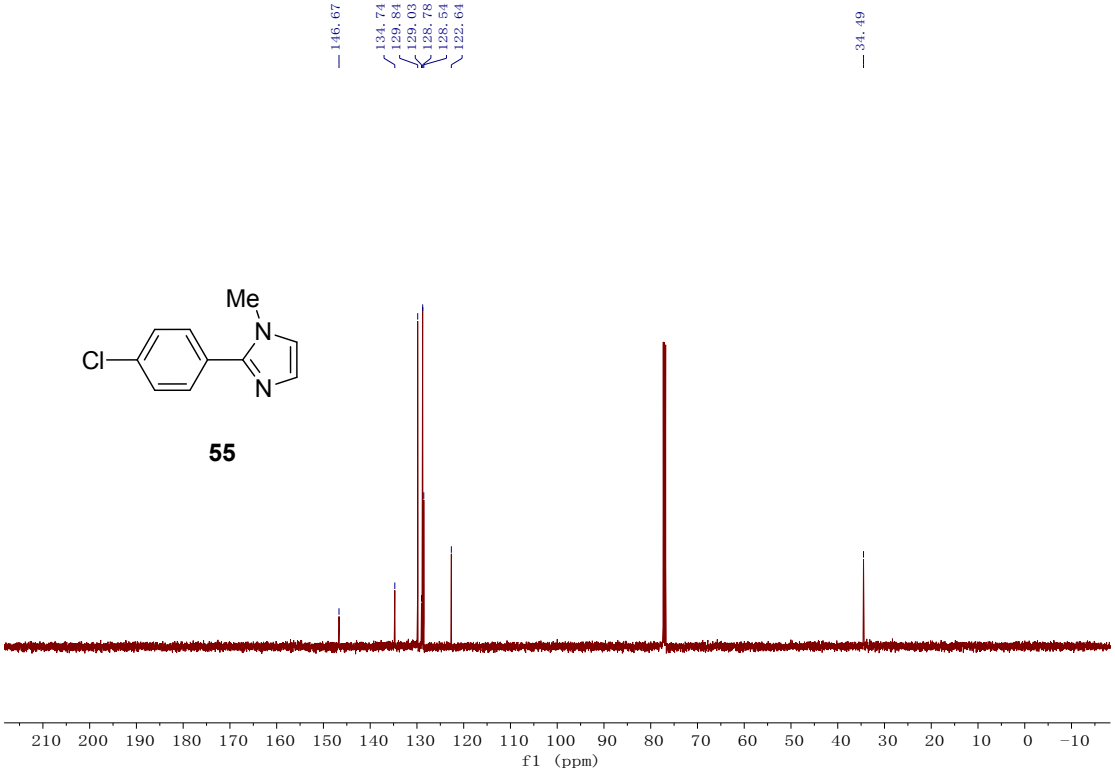

147.97
138.56
129.18
128.54
128.23
127.74
122.06
34.40
21.29
Me
N
Me
N
52
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)


7.64
7.52
7.51
7.51
7.50
7.38
7.37
7.11
6.97
3.75
Cl
Me
N
N
53
1.00
1.00
2.02
1.01
1.00
3.12
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

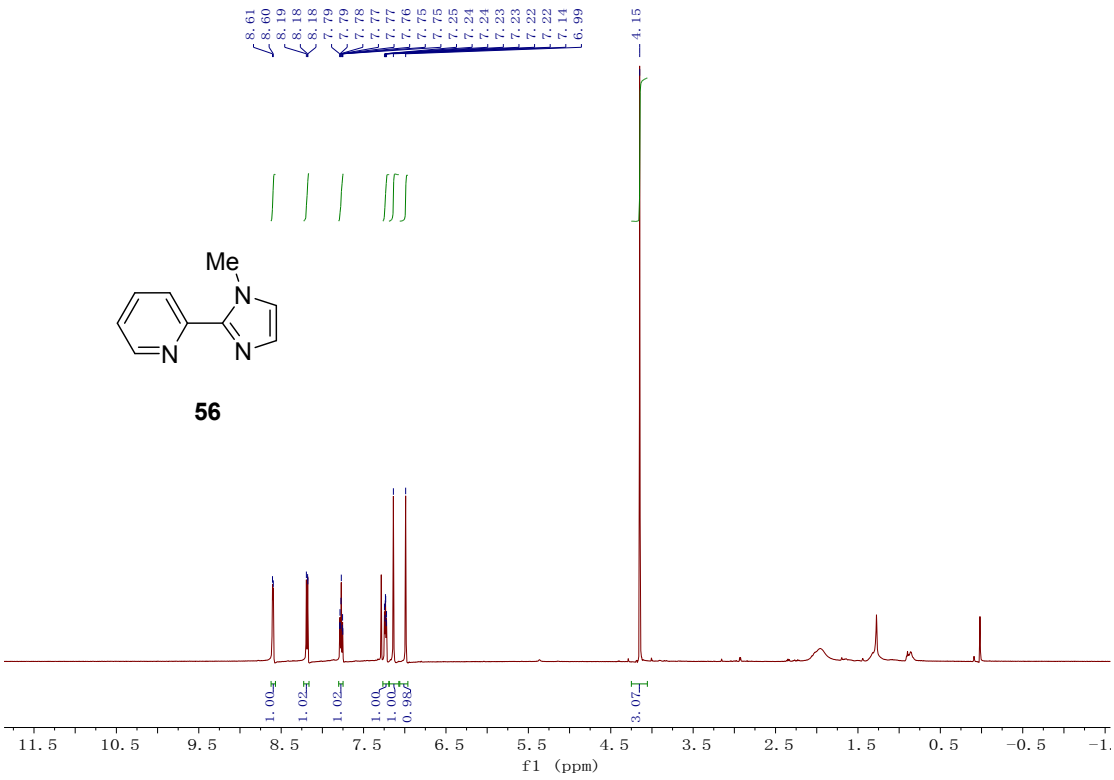

146.31
134.48
132.30
129.77
128.66
128.64
126.60
122.80
34.52
Cl
Me
N
N
53
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.58
7.57
7.44
7.43
7.14
6.98
3.74
Me
N
Cl
N
2.00
2.06
0.95
1.05
3.15
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

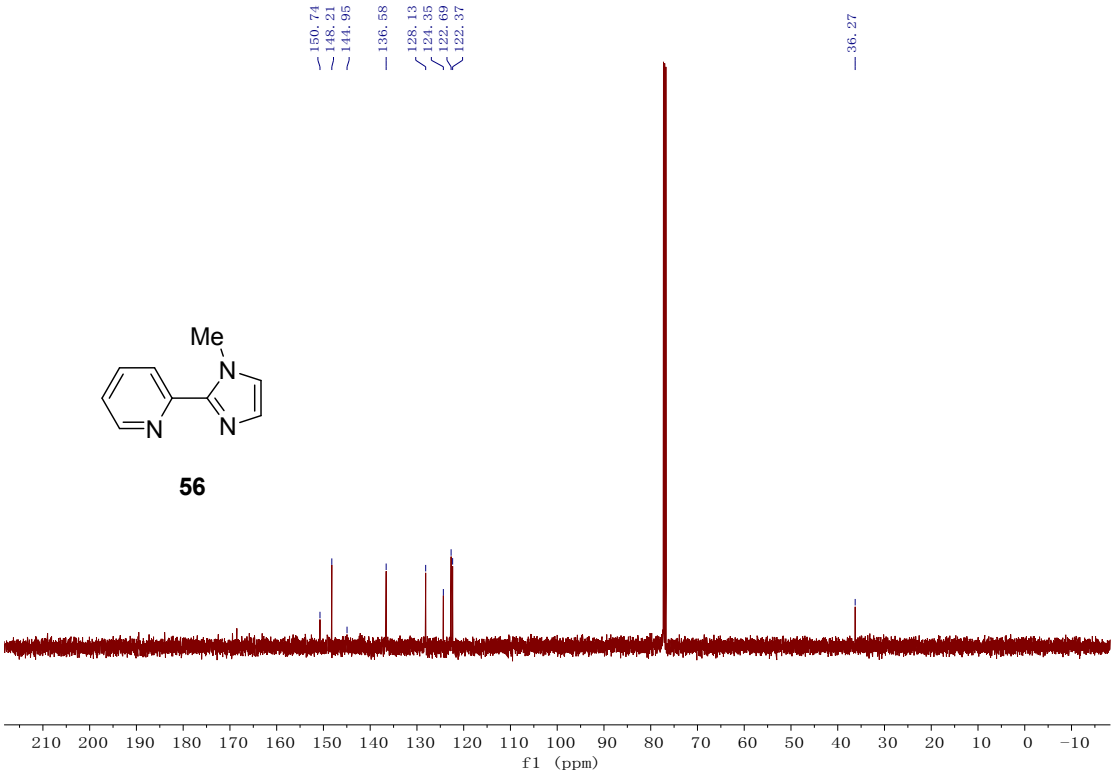
**55**


146.67
134.74
129.84
129.03
128.78
128.54
122.64
34.49
Me
N
Cl
N
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)

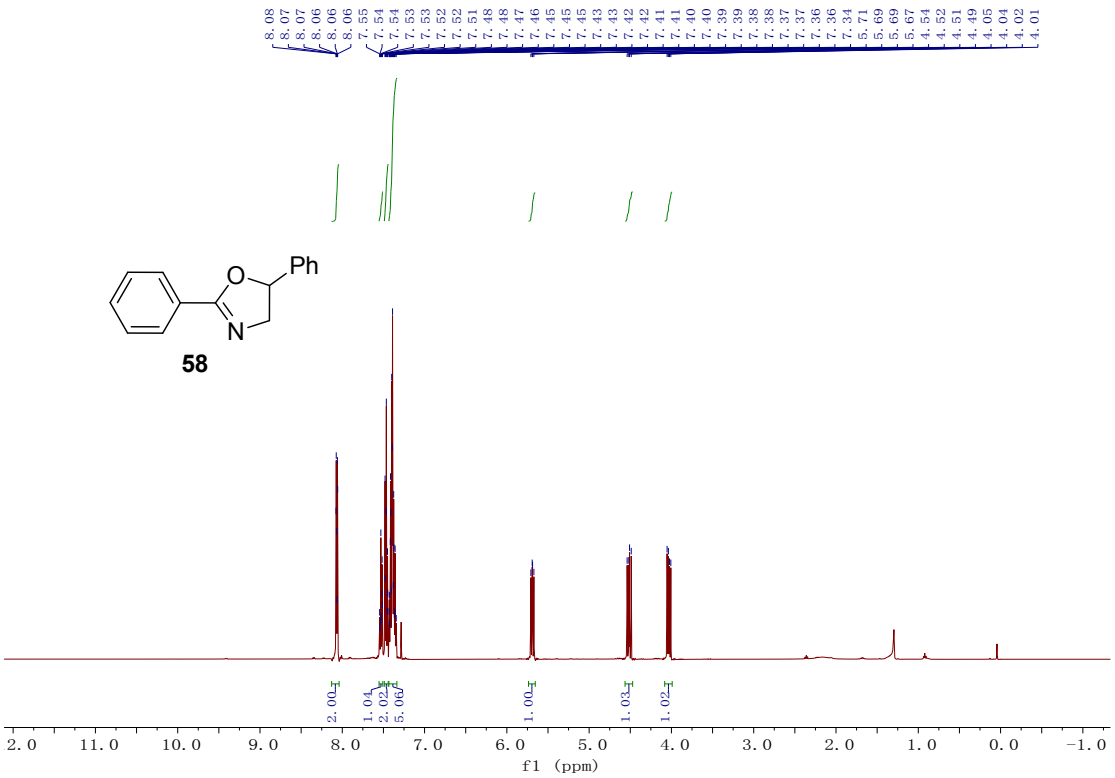
**55**

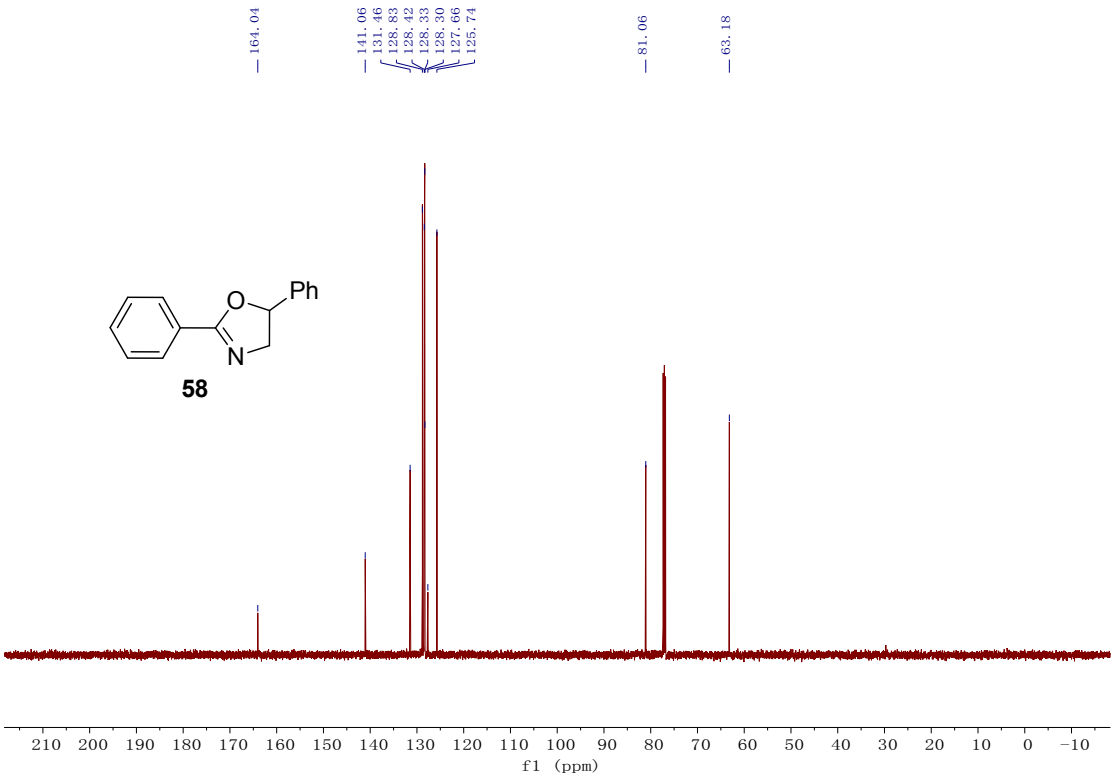

8.61
8.60
8.19
8.18
8.18
7.79
7.79
7.78
7.77
7.77
7.76
7.75
7.75
7.25
7.24
7.24
7.23
7.22
7.22
7.14
6.99
4.15
Me
N
N
N
1.00
1.02
1.02
1.00
1.00
0.98
3.07
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
−0.5
f1 (ppm)

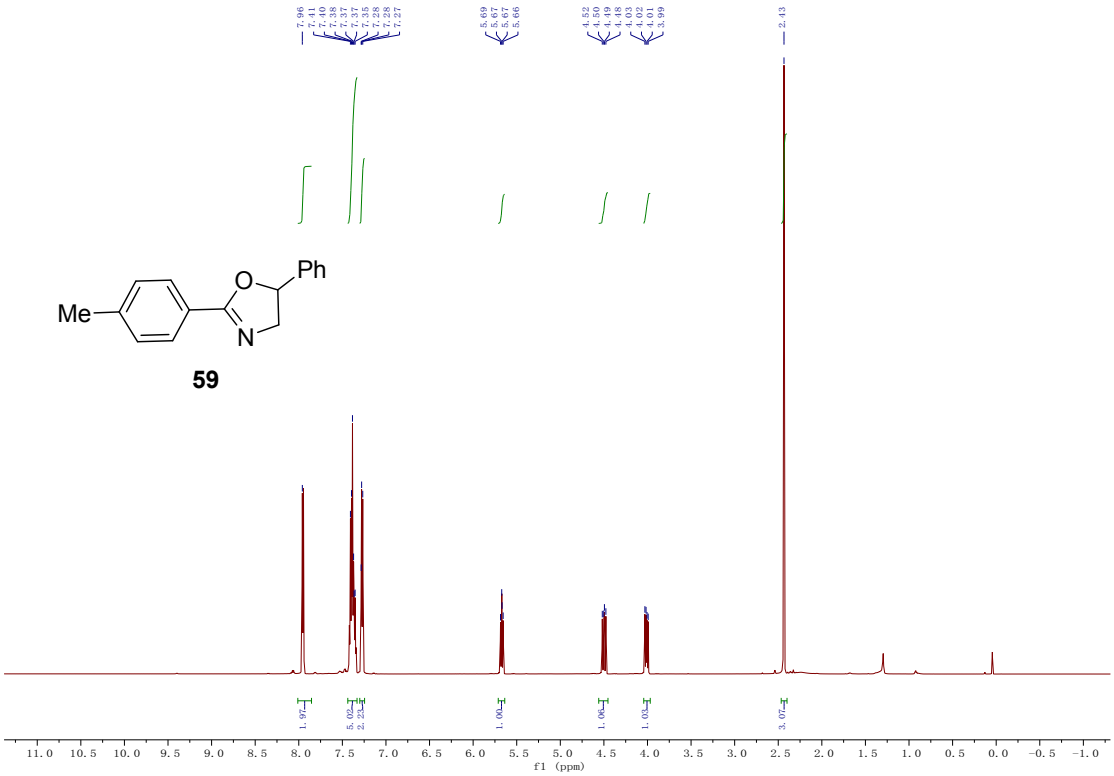
**56**

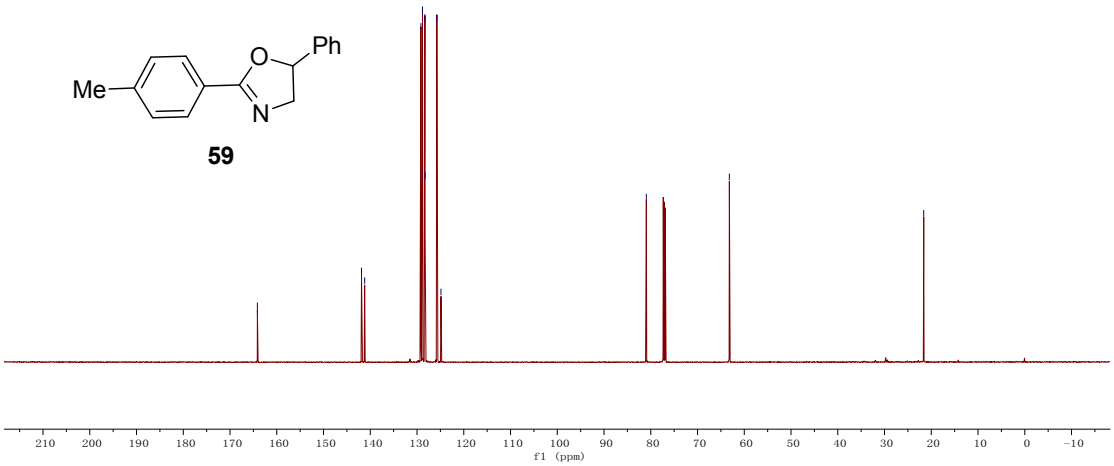

150.74
148.21
144.95
136.58
128.13
124.35
122.69
122.37
36.27
Me
N
N
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

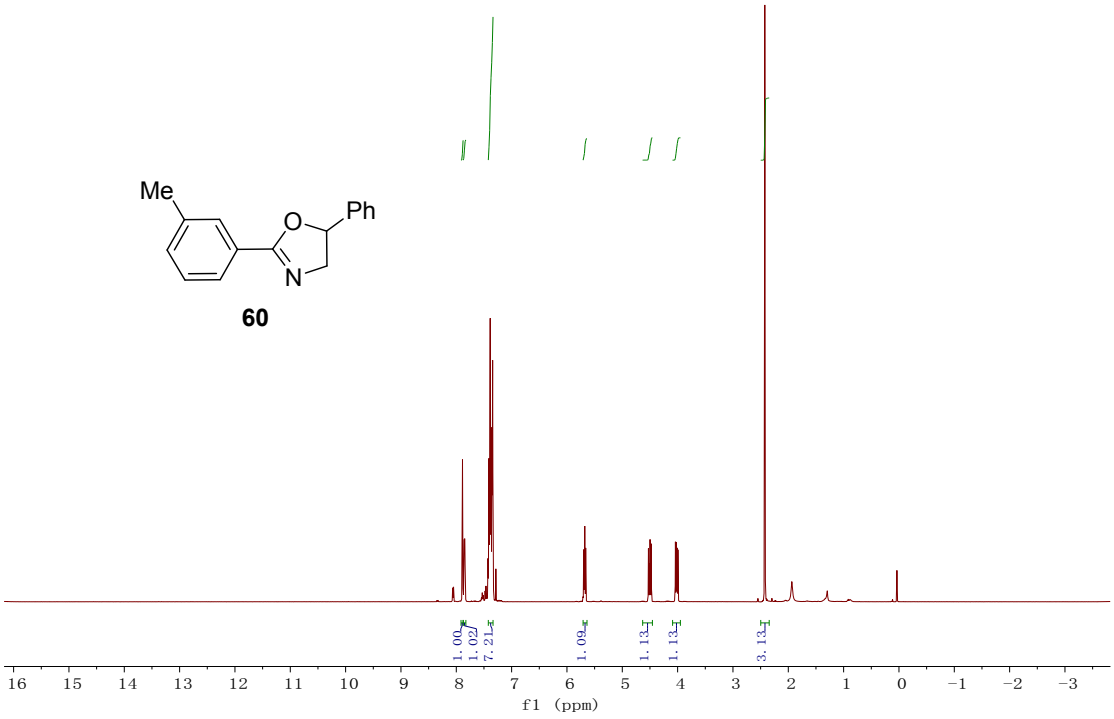
**56**

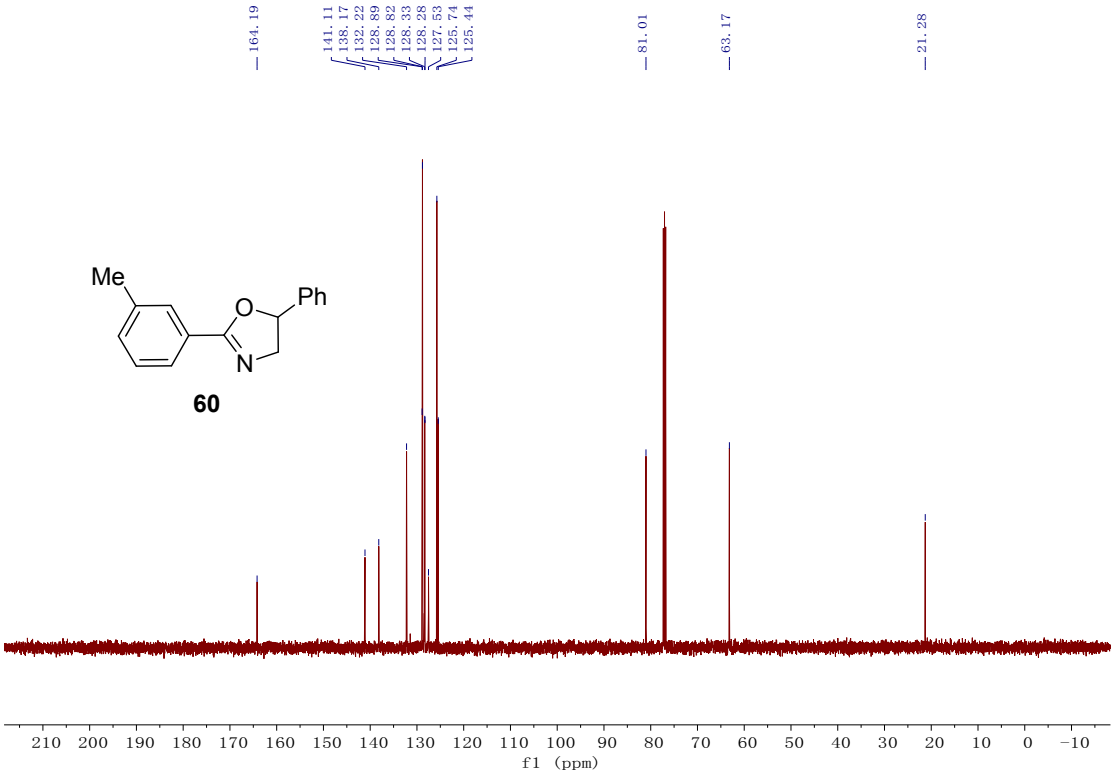

8.08 8.07 8.07 8.06 8.06 8.06 7.55 7.54 7.54 7.53 7.53 7.52 7.52 7.51 7.48 7.48 7.47 7.46 7.45 7.45 7.43 7.43 7.42 7.42 7.41 7.41 7.40 7.40 7.39 7.39 7.38 7.38 7.37 7.37 7.36 7.36 7.34 5.71 5.69 5.69 5.67 4.54 4.52 4.51 4.49 4.05 4.04 4.02 4.01
O
Ph
N
58
2.00 1.04 2.02 5.06 1.00 1.03 1.02
2.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0
f1 (ppm)

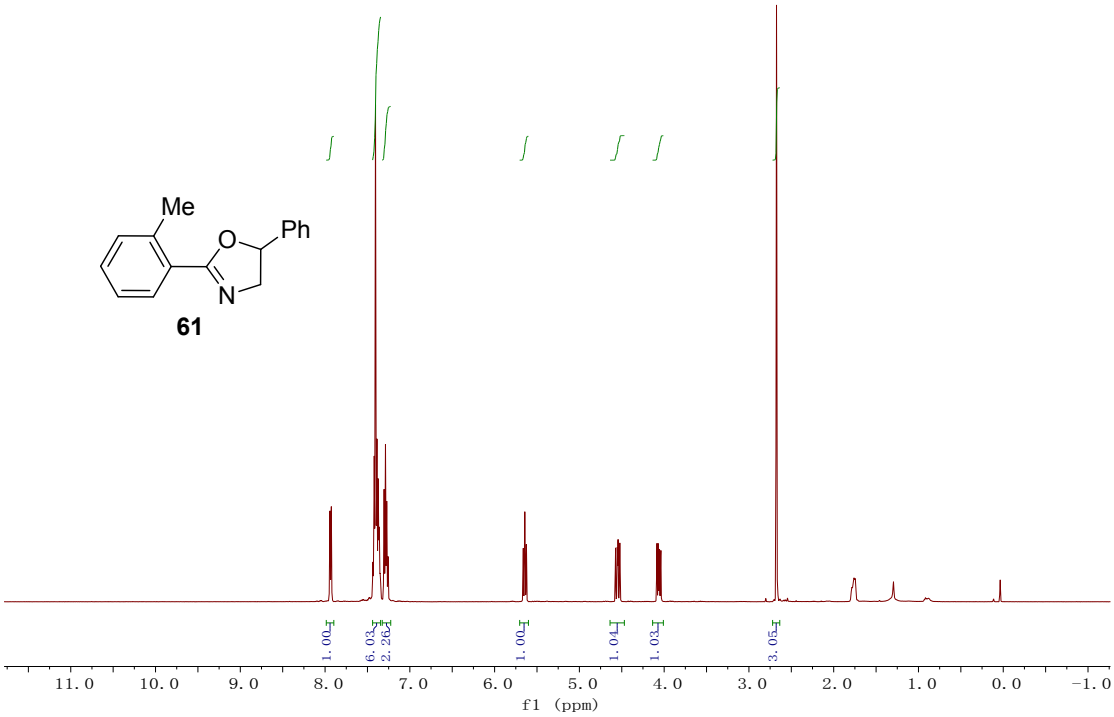

164.04
141.06 131.46 128.83 128.42 128.33 128.30 127.66 125.74
81.06
63.18
O
Ph
N
58
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

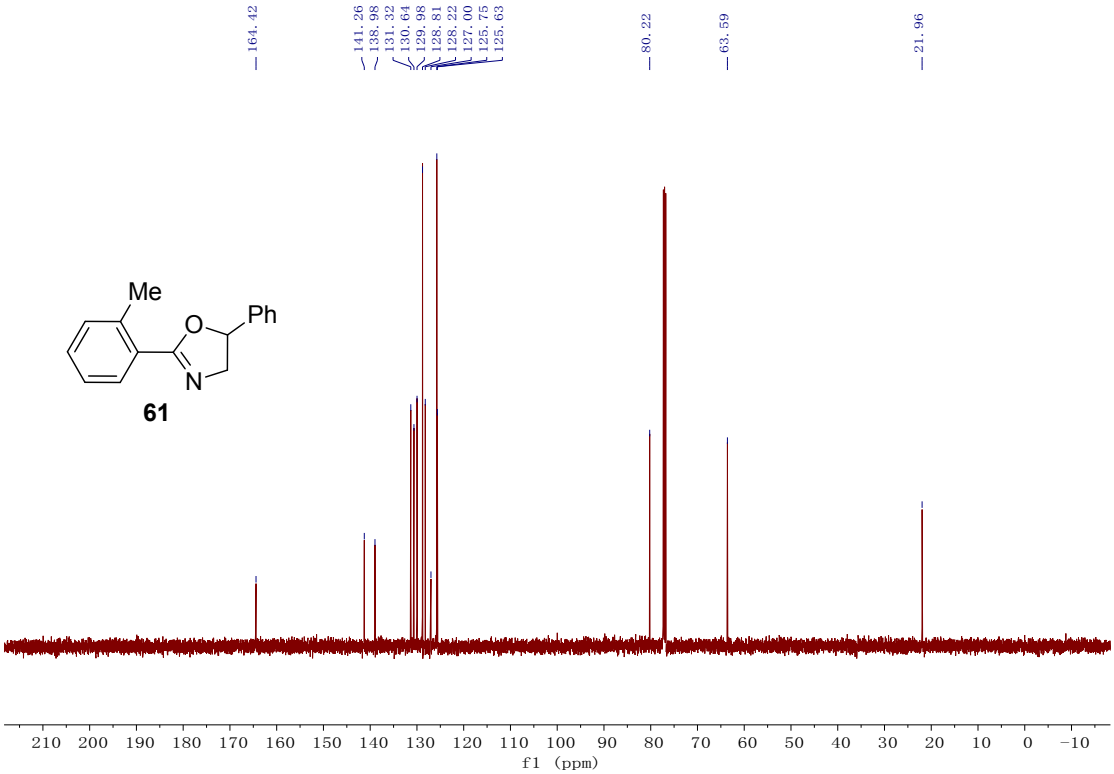

Me
O
N
Ph
59

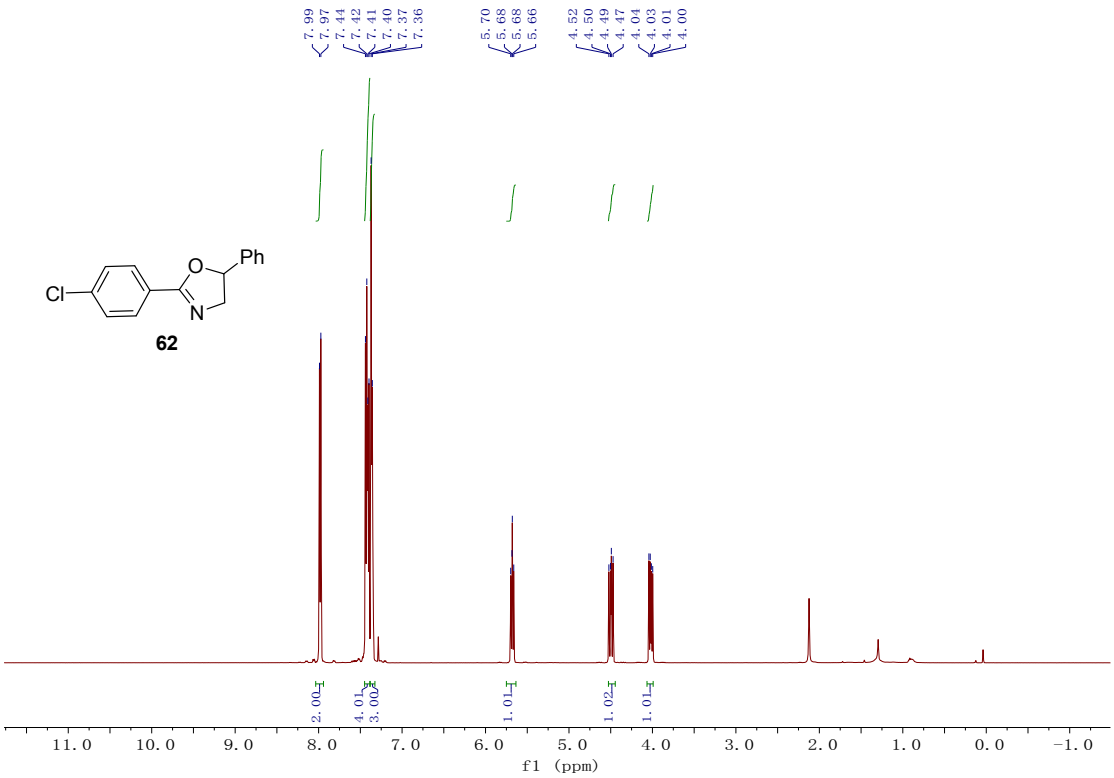

Me
O
N
Ph
59

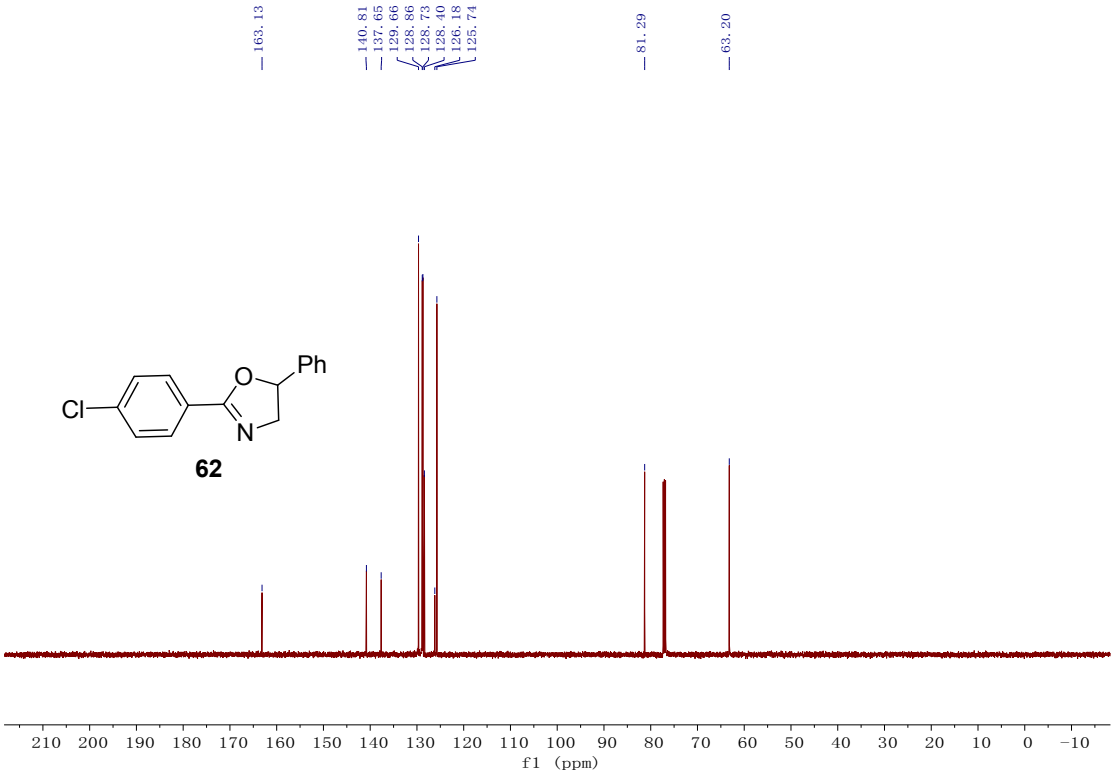

Me
O
Ph
N
60
1.00
1.02
7.21
1.09
1.13
1.13
3.13
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3
f1 (ppm)

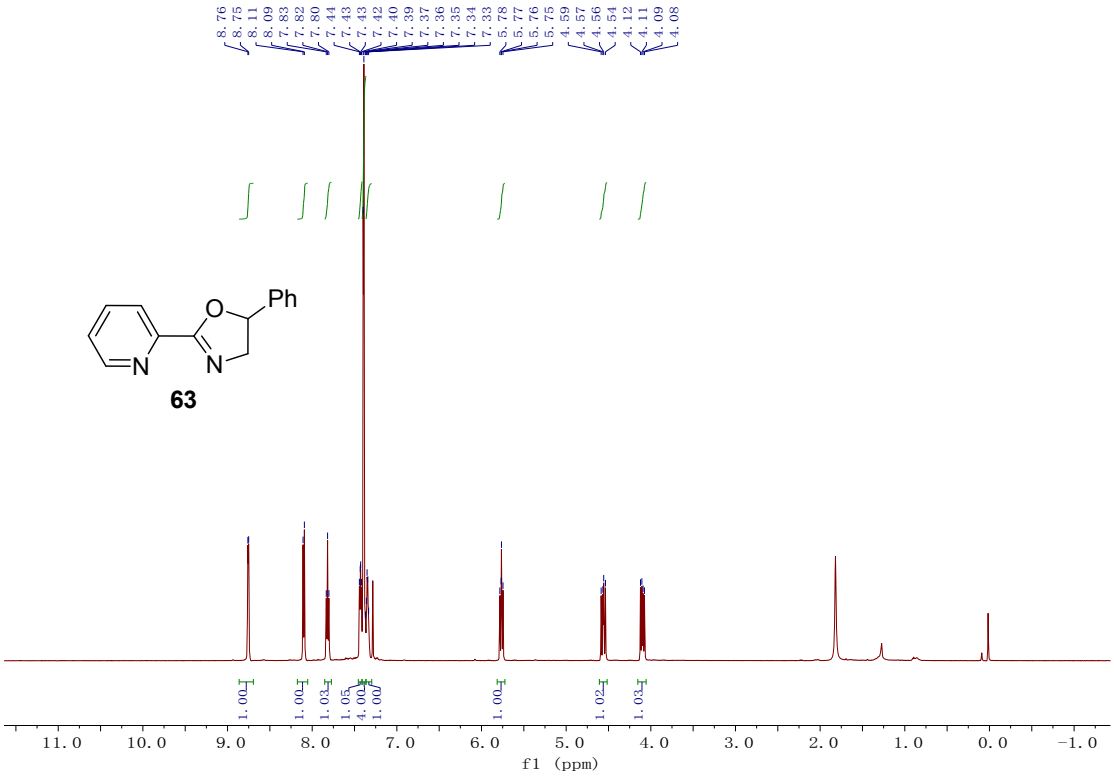

164.19
141.11
138.17
132.22
128.89
128.82
128.33
128.28
127.53
125.74
125.44
81.01
63.17
21.28
Me
O
Ph
N
60
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

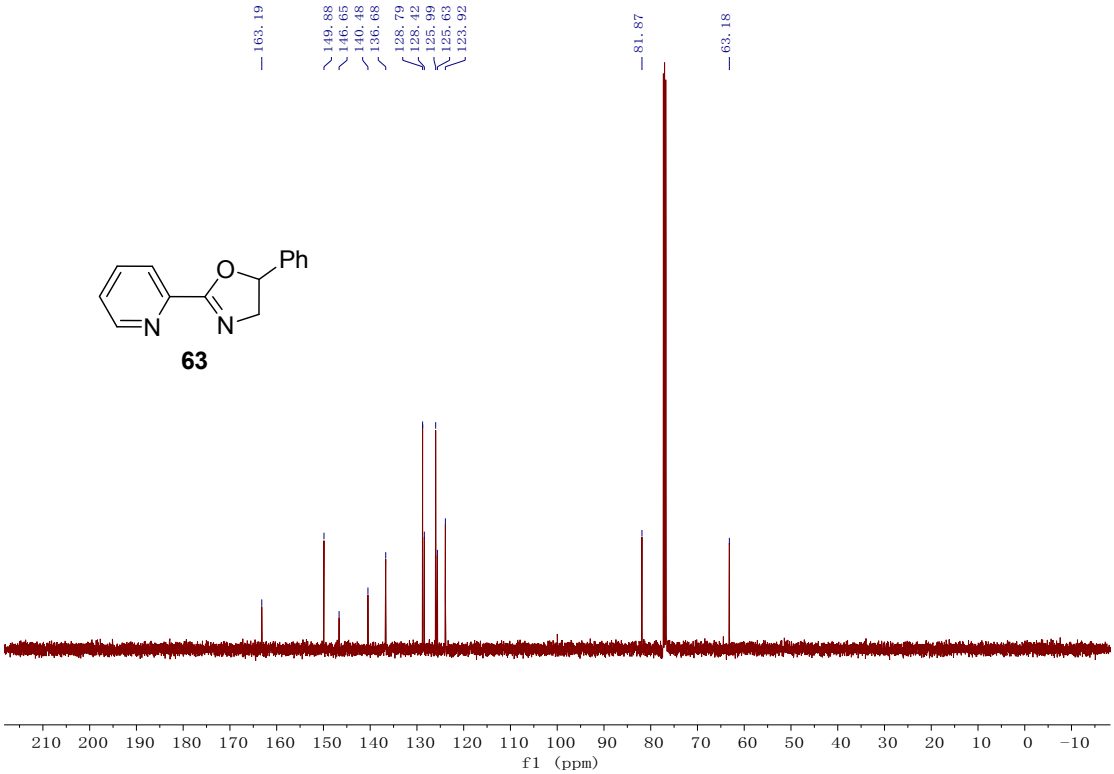

Me
O
Ph
N
61
1.00
6.03
2.26
1.00
1.04
1.03
3.05
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)


164.42
141.26
138.98
131.32
130.64
129.98
128.81
128.22
127.00
125.75
125.63
80.22
63.59
21.96
Me
O
Ph
N
61
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)


7.99
7.97
7.44
7.42
7.41
7.40
7.37
7.36
5.70
5.68
5.68
5.66
4.52
4.50
4.49
4.47
4.04
4.03
4.01
4.00
Cl
O
N
Ph
62
2.00
4.01
3.00
1.01
1.02
1.01
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)


163.13
140.81
137.65
129.66
128.86
128.73
128.40
126.18
125.74
81.29
63.20
Cl
O
N
Ph
62
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)


8.76
8.75
8.11
8.09
7.83
7.82
7.80
7.44
7.43
7.43
7.42
7.40
7.39
7.37
7.36
7.35
7.34
7.33
5.78
5.77
5.76
5.75
4.59
4.57
4.56
4.54
4.12
4.11
4.09
4.08
Ph
O
N
N
63
1.00
1.00
1.03
1.05
4.00
1.00
1.00
1.02
1.03
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)


163.19
149.88
146.65
140.48
136.68
128.79
128.42
125.99
125.63
123.92
81.87
63.18
Ph
O
N
N
63
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)