# *Supporting Information*

# Direct synthesis of fluorene-based spirolactones through a $BF_3$-promoted spiroannulation of α-keto acids and *o*-alkynyl biaryls

Qingting Song,[a] Zhen Zhang,[a] Xiaoyu Xie,[a] Guang-Zhu Ding,*[a] Pinhua Li,[a] Lei Wang,[a,b,c] and Tao Miao*,[a]

[a]Key Laboratory of Green and Precise Synthetic Chemistry and Application, Ministry of Education; Department of Chemistry, Huaibei Normal University, Huaibei, Anhui 235000, P. R. China, E-mail: taomiao@chnu.edu.cn, dinggz@chnu.edu.cn

[b]Advanced Research Institute and Department of Chemistry, Taizhou University, Taizhou, Zhejiang 318000, P. R. China

[c]State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, Shanghai 200032, P. R. China

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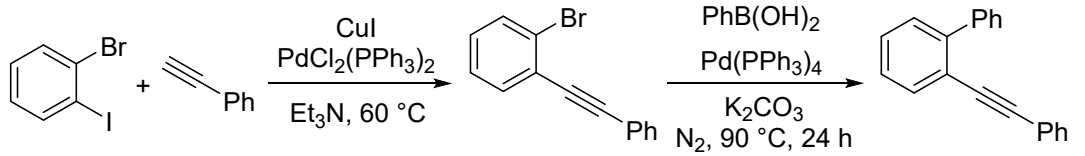
# 1. General information

Unless otherwise specified, the chemicals and solvents are purchased from commercial suppliers without further purification. All experiments were monitored by analytical thin layer chromatography (TLC) which was performed on pre-coated silica gel plates. Products were purified by flash chromatography on 100–200 mesh silica gels, $SiO_2$. $^1H$ NMR and $^{13}C$ NMR spectra were measured on a Bruker Avance NMR spectrometer (600 MHz) in $CDCl_3$ or DMSO-$d_6$ as solvent and recorded in ppm relative to internal standard tetramethylsilane. $^1H$ NMR data are reported as follows: δ, chemical shift; coupling constants (*J* are given in Hertz, Hz) and integration. Abbreviations to denote the multiplicity of a particular signal were s (singlet), d (doublet), t (triplet), q (quartet), br (broad singlet) and m (multiplet). High resolution mass spectroscopic data of the products were obtained on an Agilent Technologies 6540 UHD Accurate-Mass Q-TOF LC/MS using ESI.

# 2. Experimental section

## 2.1 General procedure for the synthesis of *o*-alkynyl biaryls (2)



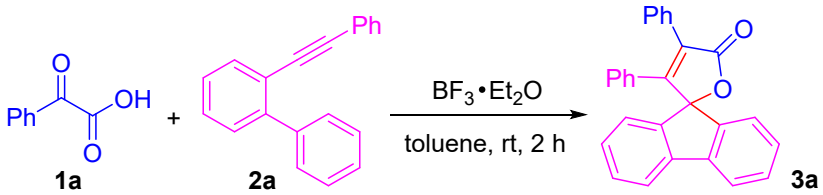

All *o*-alkynyl biaryls (**2**) were synthesized on the basis of reported method [See: Milián, A.; García-García, P.; Pérez-Redondo, A.; Sanz, R.; Vaquero, J. J.; Fernández-Rodríguez, M. A. *Org. Lett.* **2020**, *22*, 8464−8469]. A typical experimental procedure for the synthesis of 2-(phenylethynyl)-1,1'-biphenyl (**2a**) involves a Sonogashira and Suzuki couplings:

**Step 1**: 1-Bromo-2-iodobenzene (1.42 g, 5 mmol), phenylacetylene (0.62 g, 6 mmol), $PdCl_2(PPh_3)_2$ (140 mg, 0.04 equiv), CuI (47.6 mg, 0.05 equiv) were added into a 50 mL round bottom flask with 20 mL of $Et_3N$. The resulting mixture was evacuated and backfilled with nitrogen for 3 times. Then, the mixture was stirred, and was heated by an oil bath at 60 ℃ under nitrogen atmosphere. Upon completion (monitored by TLC), the mixture was cooled to room temperature, and ethyl acetate and distilled water were added. The aqueous layer was extracted with ethyl acetate for 3 times (20 mL×3). Then the organic phase was combined and dried over anhydrous $MgSO_4$. The organic layer removed in vacuo and the residue was purified by column

chromatography on silica gel by using a mixture of petroleum ether/ethyl acetate as an eluent to provide 1-bromo-2-(phenylethynyl)benzene in 81% yield (1.04 g).

**Step 2**: 1-Bromo-2-(phenylethynyl)benzene (1.30 g, 5 mmol) obtained in the step 1, phenylboronic acid (0.85 g, 7 mmol), $Pd(PPh_3)_4$ (57.8 mg, 0.01 equiv.) and $K_2CO_3$ (1.73 g, 2.5 equiv.) were added to a round bottom flask with mixture of toluene and methanol (3/1). The Schlenk flask was evacuated and backfilled with nitrogen for 3 times. Then, the mixture was stirred, and was heated by an oil bath at 90℃ for 24 h under nitrogen atmosphere. Upon completion (monitored by TLC), the mixture was cooled to room temperature, and ethyl acetate and distilled water were added. The aqueous layer was extracted with ethyl acetate for 3 times (20 mL×3). Then the organic phase was combined and dried over anhydrous $MgSO_4$. The organic layer removed in vacuo and the residue was purified by column chromatography on silica gel by using petroleum ether as an eluent to provide 2-(phenylethynyl)-1,1'-biphenyl in 86% yield (1.10 g).

## 2.2 General procedure for the synthesis of 3




**Typical procedure for the synthesis of 3a in 0.25 mmol scale:**

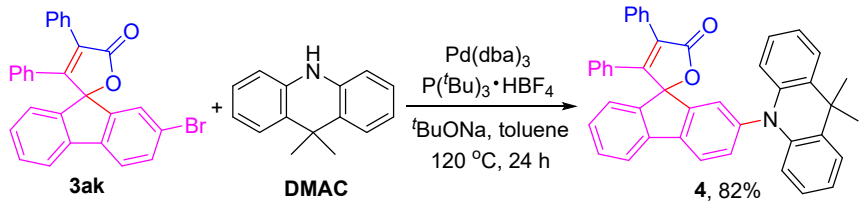
A Schlenk tube (10 mL) equipped with magnetic stir bar was charged with 2-Oxo-2-phenylacetic acid (**1a**, 60.1 mg, 0.40 mmol), 2-(phenylethynyl)-1,1'-biphenyl (**2a**, 63.6 mg, 0.25 mmol), $BF_3$•$Et_2O$ (71.0 mg, 0.50 mmol) and toluene (2 mL). Then, the mixture was stirred at room temperature for 2 h. After the reaction was completed, the solvent was removed in vacuo, and the resulting crude mixture was purified by flash chromatography on silica gel (petroleum ether/EtOAc= 9:1) to provide target product (**3a**, 92.7 mg) in 96% yield.

**Typical procedure for the synthesis of 3a in 2.5 mmol scale:**

A Schlenk tube (100 mL) equipped with magnetic stir bar was charged with 2-Oxo-2-phenylacetic acid (**1a**, 0.60 g, 4.0 mmol), 2-(phenylethynyl)-1,1'-biphenyl (**2a**, 0.64 g, 2.5 mmol), $BF_3$•$Et_2O$ (0.71 g, 5.0 mmol) and toluene (20 mL). Then, the mixture was stirred at room temperature for 2 h. After the reaction was completed, the solvent

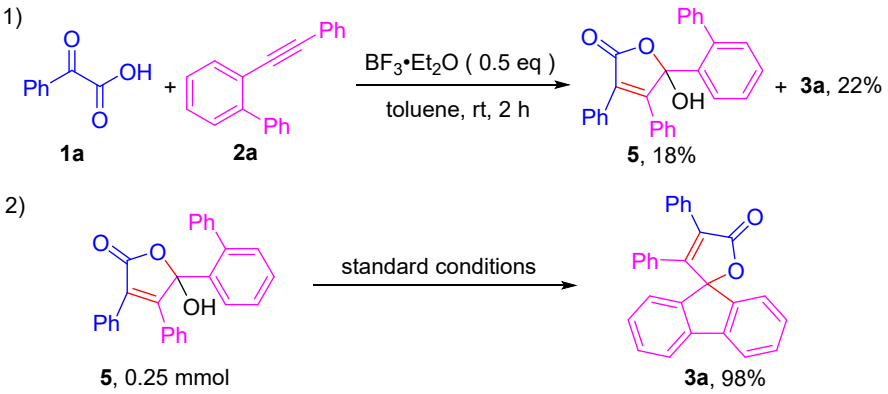
was removed in vacuo, and the resulting crude mixture was purified by flash chromatography on silica gel (petroleum ether/EtOAc= 9:1) to provide target product (**3a**, 0.89 g) in 92% yield.

### 2.3 General procedure for the synthesis of 4




To a solution of 2-bromo-3',4'-diphenyl-5'H-spiro[fluorene-9,2'-furan]-5'-one (**3ak**, 174.0 mg, 0.375 mmol) in toluene (5 mL) was added $Pd_2(dba)_3$ (12 mg, 0.05 equiv.), *t*BuONa (60 mg, 2.5 equiv.), $(tBu)_3P{\cdot}HBF_4$ (11 mg, 0.15 equiv.) and 9,9-dimethyl-9,10-dihydroacridine (DMAC, 52.3 mg, 0.25 mmol). The reaction mixture was evacuated and backfilled with nitrogen for 3 times, and was refluxed under nitrogen for 24 h. After cooling to room temperature, the reaction mixture was extracted with ethyl acetate and brine, and dried over anhydrous $MgSO_4$. Then, the solvent was removed in vacuo, and the residue was purified by flash chromatography on silica gel (petroleum ether/EtOAc= 15:1) to provide target product (**4**, 121.6 mg) in 82% yield.

## 3. The mechanism experiment




### 3.1 General procedure for the synthesis of 5

A Schlenk tube (10 mL) equipped with magnetic stir bar was charged with 2-Oxo-2-phenylacetic acid (**1a**, 60.1 mg, 0.40 mmol), 2-(phenylethynyl)-1,1'-biphenyl (**2a**, 63.6 mg, 0.25 mmol), $BF_3{\bullet}Et_2O$ (18.0 mg, 0.125 mmol) and toluene (2 mL). Then, the mixture was stirred at room temperature for 2 h. After the reaction was completed, the

solvent was removed in vacuo, and the resulting crude mixture was purified by flash chromatography on silica gel (petroleum ether/EtOAc= 9:1) to provide compound **5** (18.2 mg) in 18% yield and the product **3a** (21.3 mg) in 22% yield.

### 3.2 General procedure for the transformation of 5 to 3a

A Schlenk tube (10 mL) equipped with magnetic stir bar was charged with compound **5** (101.0 mg, 0.25 mmol), $BF_3$•$Et_2O$ (71.0 mg, 0.50 mmol) and toluene (2 mL). Then, the mixture was stirred at room temperature for 2 h. After the reaction was completed, the solvent was removed in vacuo, and the resulting crude mixture was purified by flash chromatography on silica gel (petroleum ether/EtOAc= 9:1) to provide product **3a** (94.7 mg) in 98% yield.

## 4. X-ray structural analysis of 3a and 5

In order to determine the absolute configuration of product **3** and **5**, the single crystals of **3a** and **5** were cultured in 25 mL vials by slow evaporation technique. The purified product **3a** (20 mg) and **5** (20 mg) were dissolved in a mixed solvent of $CH_2Cl_2$ and petroleum ether (1/1) at room temperature, respectively. After a few days, the crystals of **3a** and **5** appeared and were collected for X-ray diffraction analysis. The crystal structure was determined by Bruker D8 QUESTX-ray single crystal diffractometer. The CCDC number of **3a** is 2190445, and **5** is 2194522.

## checkCIF/PLATON report

You have not supplied any structure factors. As a result the full set of tests cannot be run.

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No syntax errors found. CIF dictionary Interpreting this report

## Datablock: t

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Temperature:      273 K

                 Calculated                Reported
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Hall group       -P 2yn                    -P 2yn
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Sum formula      C28 H18 O2                C1.84 H1.18 O0.13
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Dx,g cm-3        1.241                     1.241
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F000'            808.36
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Nref             4759                      4742
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The following ALERTS were generated. Each ALERT has the format
**test-name_ALERT_alert-type_alert-level.**
Click on the hyperlinks for more details of the test.

**Alert level B**

PLAT230_ALERT_2_B Hirshfeld Test Diff for C12 --C13 . 7.9 s.u.

**Alert level C**

PLAT230_ALERT_2_C Hirshfeld Test Diff for C7 --C8 . 5.3 s.u.

**Alert level G**

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FORMU01_ALERT_1_G  There is a discrepancy between the atom counts in the
           _chemical_formula_sum and _chemical_formula_moiety. This is
           usually due to the moiety formula being in the wrong format.
           Atom count from _chemical_formula_sum:   C1.84 H1.18 O0.13
           Atom count from _chemical_formula_moiety:C1.96 H1.26 O0.14
CELLZ01_ALERT_1_G Difference between formula and atom_site contents detected.
CELLZ01_ALERT_1_G ALERT: check formula stoichiometry or atom site occupancies.
          From the CIF: _cell_formula_units_Z   61
          From the CIF: _chemical_formula_sum  C1.84 H1.18 O0.13
          TEST: Compare cell contents of formula and atom_site data

          atom    Z*formula  cif sites diff
          C        112.24    112.00    0.24
          H         71.98     72.00   -0.02
          O          7.93      8.00   -0.07
PLAT042_ALERT_1_G Calc. and Reported Moiety Formula Strings Differ     Please Check
PLAT045_ALERT_1_G Calculated and Reported Z Differ by a Factor ...       0.07 Check
PLAT072_ALERT_2_G SHELXL First  Parameter in WGHT  Unusually Large       0.10 Report
PLAT199_ALERT_1_G Reported _cell_measurement_temperature ..... (K)        273 Check
PLAT200_ALERT_1_G Reported   _diffrn_ambient_temperature ..... (K)        273 Check
PLAT398_ALERT_2_G Deviating  C-O-C    Angle From 120 for O1             109.7 Degree
PLAT933_ALERT_2_G Number of OMIT Records in Embedded .res File ...          3 Note
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   0 ALERT level A = Most likely a serious problem - resolve or explain
   1 ALERT level B = A potentially serious problem, consider carefully
   1 ALERT level C = Check. Ensure it is not caused by an omission or oversight
  10 ALERT level G = General information/check it is not something unexpected

   7 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   5 ALERT type 2 Indicator that the structure model may be wrong or deficient
   0 ALERT type 3 Indicator that the structure quality may be low
   0 ALERT type 4 Improvement, methodology, query or suggestion
   0 ALERT type 5 Informative message, check
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

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A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

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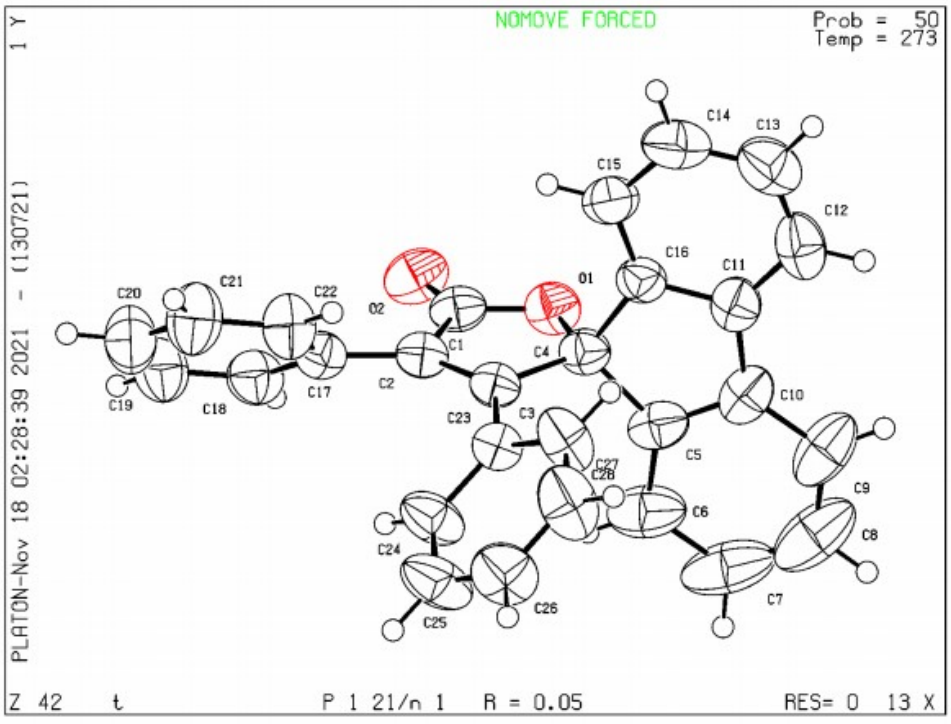
**PLATON version of 13/07/2021; check.def file version of 13/07/2021**

**Datablock t** - ellipsoid plot



## checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 2

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No syntax errors found. CIF dictionary Interpreting this report

## Datablock: 2

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Temperature:   296 K
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| Hall group | -P 1 | -P 1 |
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| Sum formula | C28 H20 O3 | C56 H40 O6 |
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| Z | 4 | 2 |
| Mu (mm-1) | 0.080 | 0.080 |
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| F000' | 848.40 | |
| h,k,lmax | 12,14,22 | 12,14,22 |
| Nref | 7582 | 7524 |
| Tmin,Tmax | 0.979,0.982 | |
| Tmin' | 0.979 | |

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Correction method= Not given

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                                                 0.1182( 7524)
S = 0.872                 Npar= 561
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The following ALERTS were generated. Each ALERT has the format
**test-name_ALERT_alert-type_alert-level.**
Click on the hyperlinks for more details of the test.

### Alert level B

PLAT601_ALERT_2_B Unit Cell Contains Solvent Accessible VOIDS of . 110 Ang**3

### Alert level C

PLAT052_ALERT_1_C Info on Absorption Correction Method Not Given Please Do !
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PLAT230_ALERT_2_C Hirshfeld Test Diff for O5 --C36 . 5.1 s.u.
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PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ..... 2.257 Check
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PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.595 49 Report

### Alert level G

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PLAT398_ALERT_2_G Deviating C-O-C Angle From 120 for O2 . 109.4 Degree
PLAT398_ALERT_2_G Deviating C-O-C Angle From 120 for O5 . 109.2 Degree
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PLAT793_ALERT_4_G Model has Chirality at C44 (Centro SPGR) R Verify
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PLAT961_ALERT_5_G Dataset Contains no Negative Intensities ..... Please Check
PLAT965_ALERT_2_G The SHELXL WEIGHT Optimisation has not Converged Please Check
PLAT967_ALERT_5_G Note: Two-Theta Cutoff Value in Embedded .res .. 50.0 Degree
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 1 Info

0 **ALERT level A** = Most likely a serious problem - resolve or explain
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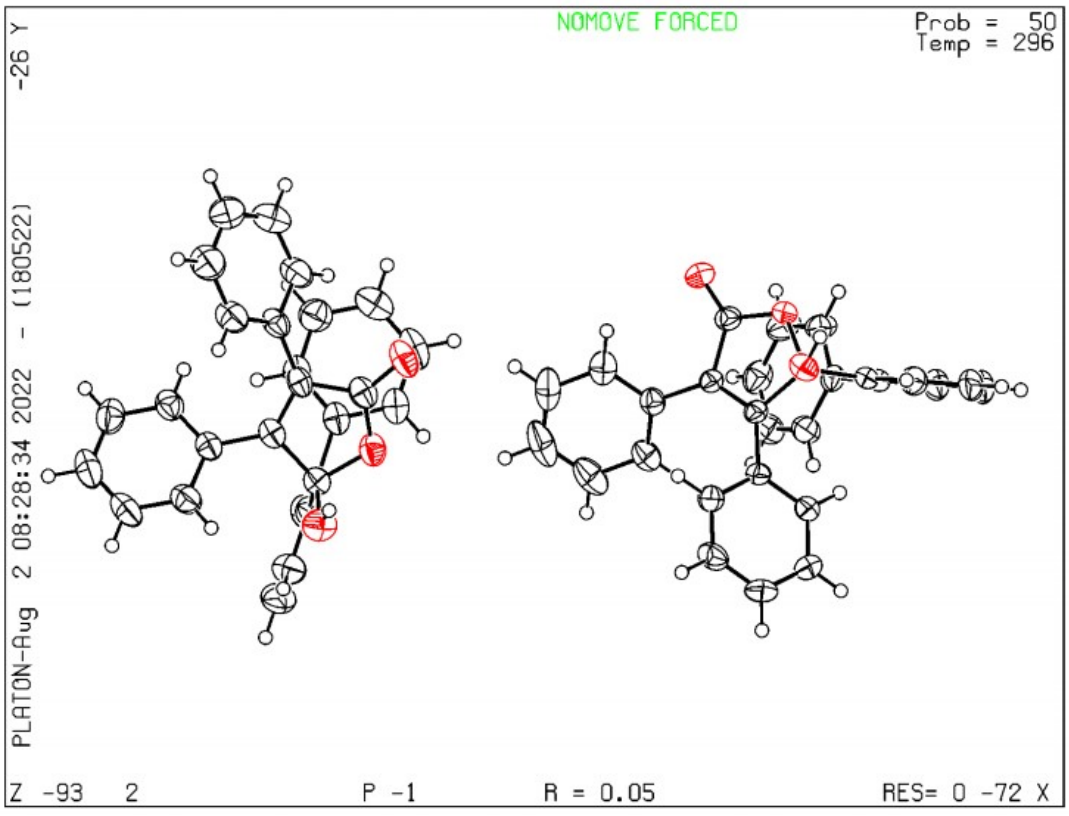
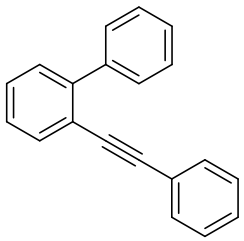
**PLATON version of 18/05/2022; check.def file version of 17/05/2022**

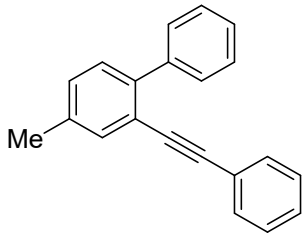
**Datablock 2** - ellipsoid plot



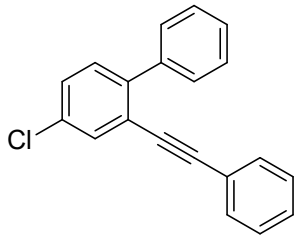
## 5. Characterization data for *o*-alkynyl biaryls (2) and all products



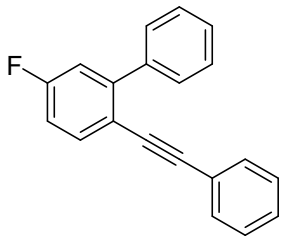
**2-(Phenylethynyl)-1,1'-biphenyl (2a)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford an orange liquid (1.10 g, 86% yield); $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.67–7.66 (m, 2H), 7.65–7.64 (m, 1H), 7.45 (t, $J$ = 7.8 Hz, 2H), 7.43–7.41 (m, 1H), 7.39–7.37 (m, 2H), 7.33–7.30 (m, 3H), 7.27–7.26 (m, 3H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 143.9, 140.5, 132.8, 131.3, 129.5, 129.4, 128.5, 128.2, 128.1, 127.9, 127.4, 127.0, 123.4, 121.6, 92.2, 89.4. HRMS (ESI) *m/z*: [M + H]$^{+}$ calcd for $C_{20}H_{15}$, 255.1168; found, 255.1168.



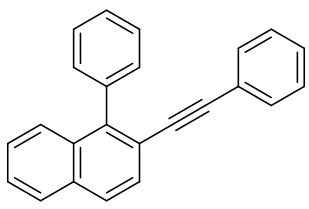

**4-Methyl-2-(phenylethynyl)-1,1'-biphenyl (2b)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford a yellow liquid (1.13 g, 84% yield); $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.65 (d, $J$ = 7.2 Hz, 2H), 7.47 (s, 1H), 7.43 (t, $J$ = 7.8 Hz, 2H), 7.36 (t, $J$ = 7.2 Hz, 1H), 7.33–7.31 (m, 3H), 7.28–7.26 (m, 3H), 7.20 (d, $J$ = 7.8 Hz, 1H), 2.38 (s, 3H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 141.1, 140.5, 136.8, 133.3, 131.3, 129.5, 129.4, 128.2, 128.0, 127.8, 127.2, 123.5, 121.3, 91.8, 89.6, 20.8. HRMS (ESI) *m/z*: [M + H]$^{+}$ calcd for $C_{21}H_{17}^{+}$, 269.1325; found, 269.1329.



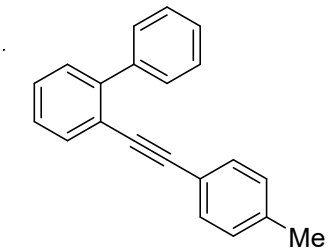

**4-Chloro-2-(phenylethynyl)-1,1'-biphenyl (2c)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford an orange liquid (1.17 g, 81% yield); $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.62–7.61 (m, 3H), 7.44 (t, $J$ = 7.5 Hz, 2H), 7.40–7.37 (m, 1H), 7.35–7.34 (m, 2H), 7.32–7.30 (m, 2H), 7.29–7.26 (m, 3H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 142.3, 139.4, 132.8, 132.3, 131.4, 130.6, 129.2, 128.6, 128.4, 128.3, 128.0, 127.7, 123.2, 122.9, 93.2, 88.1. HRMS (ESI) *m/z*: [M + H]$^{+}$ calcd for $C_{20}H_{14}Cl^{+}$, 289.0779; found, 289.0776.



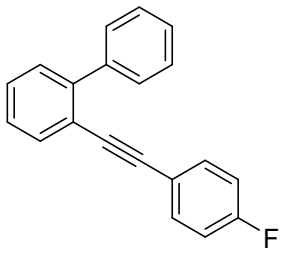

**4-Fluoro-2-(phenylethynyl)-1,1'-biphenyl (2d)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford an orange liquid (1.06 g, 78% yield); $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.66–7.64 (m, 2H), 7.62–7.60 (m, 1H), 7.46 (t, $J$ = 7.5 Hz, 2H), 7.40 (t, $J$ = 7.5 Hz, 1H), 7.31–7.26 (m, 5H), 7.15–7.12 (m, 1H), 7.04–7.01 (m, 1H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 162.4 (C–F, $1J_{C–F}$ = 248.7 Hz), 146.1 (C–F, $3J_{C–F}$ = 8.3 Hz), 139.5, 134.6 (C–F, $3J_{C–F}$ = 8.4 Hz), 131.3, 129.2, 128.3, 128.1, 128.01, 127.97, 123.3, 117.7(C–F, $4J_{C–F}$ = 3.2 Hz), 116.5 (C–F, $2J_{C–F}$ = 22.7 Hz), 114.3 (C–F, $2J_{C–F}$ = 21.8 Hz), 91.8, 88.4; $^{19}$F{$^{1}$H} NMR (565 MHz, $CDCl_3$): $\delta$ –111.0. HRMS (ESI) *m/z*: [M + H]$^{+}$ calcd for $C_{20}H_{14}F^{+}$, 273.1074; found, 273.1075.



**1-Phenyl-2-(phenylethynyl)naphthalene (2e)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford a yellow liquid (1.22 g, 80% yield); $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.84 (d, $J$ = 8.4 Hz, 1H), 7.81 (d, $J$ = 9.0 Hz, 1H), 7.66 (t, $J$ = 9.0 Hz, 2H), 7.53–7.45 (m, 6H), 7.38 (t, $J$ = 7.5 Hz, 1H), 7.23–7.21 (m, 3H), 7.18–7.16 (m, 2H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 143.1, 138.9, 133.1, 132.2, 131.4, 130.7, 128.3, 128.2, 128.00, 127.98, 127.9, 127.5, 126.7, 126.5, 126.3, 123.4, 120.1, 93.2, 90.0. HRMS (ESI) *m/z*: [M + H]$^{+}$ calcd for $C_{24}H_{17}^{+}$, 305.1325; found, 305.1322.

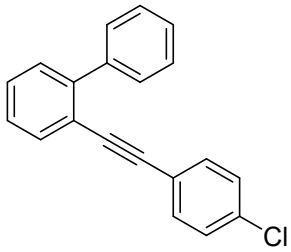



**2-(*p*-Tolylethynyl)-1,1'-biphenyl (2f)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford an orange liquid (1.13 g, 84% yield); $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.67–7.66 (m, 2H), 7.63–7.62 (m, 1H), 7.44 (t, $J$ = 7.5 Hz, 2H), 7.42–7.41 (m, 1H), 7.38–7.35 (m, 2H), 7.32–7.30 (m, 1H), 7.22 (d, $J$ = 8.4 Hz, 2H), 7.08 (d, $J$ = 7.8 Hz, 2H), 2.32 (s, 3H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 143.8, 140.6, 138.2, 132.7, 131.2, 129.42, 129.38, 129.0, 128.3, 127.8, 127.4, 127.0, 121.8, 120.4, 92.4, 88.7, 21.5. HRMS (ESI) *m/z*: [M + H]$^{+}$ calcd for $C_{21}H_{17}^{+}$, 269.1325; found, 269.1328.

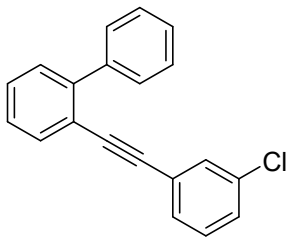



**2-((4-Fluorophenyl)ethynyl)-1,1'-biphenyl (2g)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford a yellow liquid (1.12 g, 82%

yield); $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.65–7.63 (m, 2H), 7.62 (d, $J$ = 7.8 Hz, 1H), 7.44 (t, $J$ = 7.5 Hz, 2H), 7.42–7.40 (m, 1H), 7.38 (t, $J$ = 7.2 Hz, 2H), 7.32–7.30 (m, 1H), 7.29–7.26 (m, 2H), 6.96 (t, $J$ = 8.7 Hz, 2H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 162.4 (C–F, $1J_{C–F}$ = 248.0 Hz), 143.9, 140.5, 133.2 (C–F, $3J_{C–F}$ = 8.4 Hz), 132.7, 129.5, 129.4, 128.5, 127.9, 127.5, 127.1, 121.4, 119.5 (C–F, $4J_{C–F}$ = 3.3 Hz), 115.5 (C–F, $2J_{C–F}$ = 21.9 Hz), 91.1, 89.0; $^{19}$F{$^1$H} NMR (565 MHz, $CDCl_3$): $\delta$ –111.0. HRMS (ESI) *m/z*: $[M + H]^+$ calcd for $C_{20}H_{14}F^+$, 273.1074; found, 273.1076.

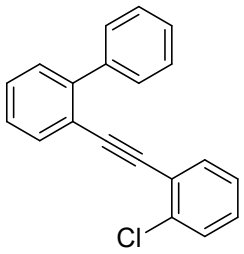



**2-((4-Chlorophenyl)ethynyl)-1,1'-biphenyl (2h)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford a yellow liquid (1.17 g, 81% yield); $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.63 (t, $J$ = 7.2 Hz, 3H), 7.45–7.41 (m, 3H), 7.40–7.37 (m, 2H), 7.33–7.30 (m, 1H), 7.24–7.21 (m, 4H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 144.0, 140.5, 134.1, 132.8, 132.5, 129.5, 129.3, 128.7, 128.6, 127.9, 127.5, 127.1, 121.9, 121.2, 91.0, 90.3. HRMS (ESI) *m/z*: $[M + H]^+$ calcd for $C_{20}H_{14}Cl^+$, 289.0779; found, 289.0780.

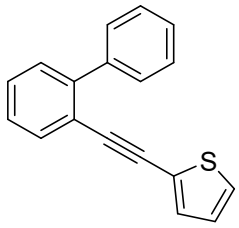



**2-((3-Chlorophenyl)ethynyl)-1,1'-biphenyl (2i)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford an orange liquid (1.22 g, 85% yield); $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.64–7.63 (m, 3H), 7.46 (t, $J$ = 7.5 Hz, 2H), 7.44–7.39 (m, 3H), 7.34–7.33 (m, 1H), 7.29 (s, 1H), 7.25–7.23 (m, 1H), 7.21–7.17 (m, 2H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 144.1, 140.4, 134.0, 132.9, 131.1,

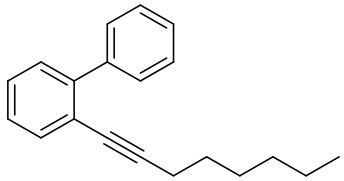
129.5, 129.44, 129.43, 129.3, 128.9, 128.3, 127.9, 127.6, 127.1, 125.1, 121.1, 90.7, 90.6. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{14}Cl^+$, 289.0779; found, 289.0782.



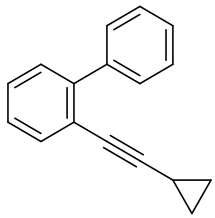

**2-((2-Chlorophenyl)ethynyl)-1,1'-biphenyl (2j)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford a yellow liquid (1.15 g, 80% yield); $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.71–7.69 (m, 1H), 7.68–7.66 (m, 2H), 7.45–7.40 (m, 4H), 7.39–7.37 (m, 1H), 7.36–7.31 (m, 3H), 7.20–7.17 (m, 1H), 7.16–7.13 (m, 1H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$): $\delta$ 143.9, 140.3, 135.7, 133.3, 133.2, 129.5, 129.4, 129.2, 129.1, 128.9, 128.0, 127.5, 127.0, 126.3, 123.4, 121.2, 94.3, 88.9. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{14}Cl^+$, 289.0779; found, 289.0778.



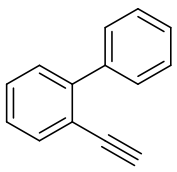

**2-([1,1'-Biphenyl]-2-ylethynyl)thiophene (2k)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford an orange liquid (1.14 g, 88% yield); $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.64–7.63 (m, 2H), 7.61–7.60 (m, 1H), 7.44 (t, $J$ = 7.5 Hz, 2H), 7.43–7.41 (m, 1H), 7.39–7.36 (m, 2H), 7.32–7.30 (m, 1H), 7.22–7.21 (m, 1H), 7.09–7.08 (m, 1H), 6.94–6.93 (m, 1H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$): $\delta$ 143.7, 140.3, 132.5, 131.5, 129.5, 129.3, 128.6, 127.9, 127.5, 127.1, 127.03, 126.97, 123.5, 121.3, 93.1, 85.6. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{18}H_{13}S^+$, 261.0732; found, 261.0731.



**2-(Oct-1-yn-1-yl)-1,1'-biphenyl (2l)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford an orange liquid (0.98 g, 75% yield); $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.59–7.58 (m, 2H), 7.51–7.50 (m, 1H), 7.39 (t, $J$ = 7.5 Hz, 2H), 7.35–7.30 (m, 3H), 7.26–7.24 (m, 1H), 2.28 (t, $J$ = 7.2 Hz, 2H), 1.48–1.43 (m, 2H), 1.31–1.25(m, 4H), 1.24–1.19 (m, 2H), 0.88 (t, $J$ = 7.2 Hz, 3H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 143.6, 140.8, 133.0, 129.4, 129.3, 127.7, 127.6, 127.1, 126.9, 122.4, 93.5, 80.1, 31.4, 28.5, 28.4, 22.5, 19.5, 14.1. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{23}^+$, 263.1794; found, 263.1795.

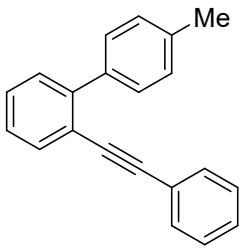


**2-(Cyclopropylethynyl)-1,1'-biphenyl (2m)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford a yellow liquid (0.90 g, 82% yield); $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.55 (d, $J$ = 7.8 Hz, 2H), 7.48 (d, $J$ = 7.8 Hz, 1H), 7.39 (t, $J$ = 7.5 Hz, 2H), 7.34–7.32 (m, 2H), 7.30–7.28 (m, 1H), 7.24–7.21 (m, 1H), 1.32–1.28 (m, 1H), 0.75–0.72 (m, 2H), 0.62–0.59 (m, 2H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 143.6, 140.7, 132.8, 129.3, 129.2, 127.7, 127.6, 127.2, 126.9, 122.2, 96.4, 75.2, 8.3, 0.3. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{17}H_{15}^+$, 219.1168; found, 219.1167.

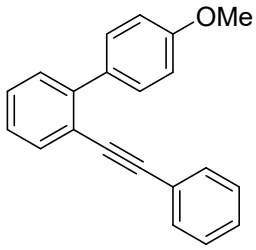


**2-Ethynyl-1,1'-biphenyl (2n)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford a yellow liquid (0.72 g, 81% yield); $^{1}$H NMR (600 MHz,

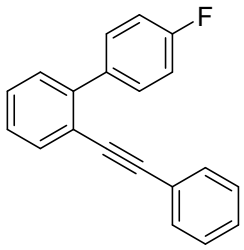
$CDCl_3$): $\delta$ 7.62–7.61 (m, 1H), 7.60–7.58 (m, 2H), 7.44–7.41 (m, 2H), 7.40–7.39 (m, 1H), 7.38–7.36 (m, 2H), 7.31–7.28 (m, 1H), 3.04 (s, 1H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$): $\delta$ 144.4, 140.2, 133.8, 129.6, 129.2, 128.9, 128.0, 127.5, 127.0, 120.4, 83.1, 80.1. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{14}H_{11}^+$, 179.0855; found, 179.0857.



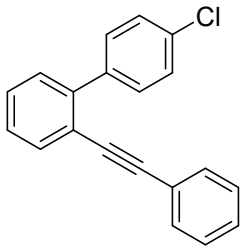

**4'-Methyl-2-(phenylethynyl)-1,1'-biphenyl (2o)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford a yellow liquid (1.12 g, 83% yield); $^1H$ NMR (600 MHz, $CDCl_3$): $\delta$ 7.63 (d, $J$ = 7.8 Hz, 1H), 7.58–7.57 (m, 2H), 7.40 (d, $J$ = 7.8 Hz, 1H), 7.37–7.34 (m, 3H), 7.30–7.24 (m, 6H), 2.41 (s, 3H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$): $\delta$ 143.7, 137.6, 137.1, 132.9, 131.3, 129.4, 129.2, 128.6, 128.5, 128.2, 128.0, 126.8, 123.5, 121.4, 92.1, 89.5, 21.2. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{21}H_{17}^+$, 269.1325; found, 169.1323.



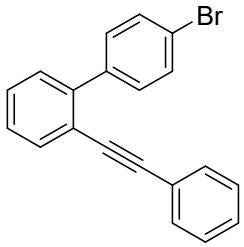

**4'-Methoxy-2-(phenylethynyl)-1,1'-biphenyl (2p)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford an orange liquid (1.15 g, 81% yield); $^1H$ NMR (600 MHz, $CDCl_3$): $\delta$ 7.63–7.61 (m, 3H), 7.40–7.39 (m, 1H), 7.37–7.35 (m, 3H), 7.30–7.27 (m, 4H), 6.99 (d, $J$ = 9.0 Hz, 2H), 3.86 (s, 3H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$): $\delta$ 159.1, 143.4, 133.0, 132.9, 131.3, 130.5, 129.3, 128.5, 128.2, 128.0, 126.6, 123.5, 121.4, 113.3, 92.1, 89.6, 55.3. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{21}H_{17}O^+$, 285.1274; found, 285.1278.



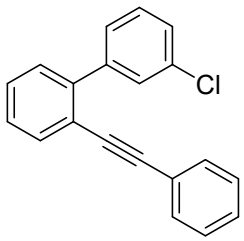

**4'-fluoro-2-(phenylethynyl)-1,1'-biphenyl (2q).** Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford a yellow liquid (1.13 g, 83% yield); $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.65–7.61 (m, 3H), 7.39 (d, $J$ = 4.2 Hz, 2H), 7.34–7.32 (m, 3H), 7.30–7.29 (m, 3H), 7.14 (t, $J$ = 8.7 Hz, 2H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 162.4 (C–F, $1J_{C–F}$ = 245.0 Hz), 142.8, 136.6 (C–F, $4J_{C–F}$ = 3.3 Hz), 132.9, 131.3, 131.0 (C–F, $3J_{C–F}$ = 8.4 Hz), 129.4, 128.6, 128.3, 128.2, 127.2, 123.3, 121.6, 114.8 (C–F, $2J_{C–F}$ = 20.9 Hz), 92.4, 89.1; $^{19}$F{$^{1}$H} NMR (565 MHz, $CDCl_3$): $\delta$ -115.1. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{14}F^+$, 273.1074; found, 273.1076.



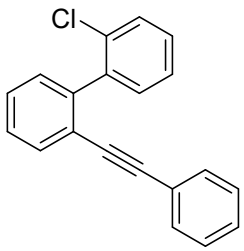

**4'-Chloro-2-(phenylethynyl)-1,1'-biphenyll (2r)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford a yellow liquid (1.16 g, 80% yield); $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.64 (d, $J$ = 7.8 Hz, 1H), 7.60 (d, $J$ = 8.4 Hz, 2H), 7.42 (d, $J$ = 9.0 Hz, 2H), 7.38–7.37 (m, 2H), 7.34–7.29 (m, 6H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 142.5, 139.0, 133.5, 133.0, 131.3, 130.7, 129.3, 128.6, 128.32, 128.26, 128.1, 127.4, 123.2, 121.5, 92.5, 88.9. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{14}Cl^+$, 289.0779; found, 289.0784.




**4'-Bromo-2-(phenylethynyl)-1,1'-biphenyl (2s)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford a yellow liquid (1.26 g, 76% yield); $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.64–7.62 (m, 1H), 7.57–7.56 (m, 2H), 7.54–7.52 (m, 2H), 7.37–7.36 (m, 3H), 7.34–7.31 (m, 3H), 7.30–7.28 (m, 2H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 142.5, 139.4, 133.0, 131.3, 131.0, 129.2, 128.6, 128.32, 128.27, 127.4, 123.2, 121.7, 121.4, 92.6, 88.9.

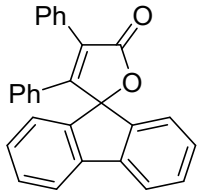



**3'-Chloro-2-(phenylethynyl)-1,1'-biphenyl (2t)**. Following the general procedure for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford a yellow liquid (1.24 g, 86% yield); $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.72 (s, 1H), 7.64 (d, $J$ = 7.8 Hz, 1H), 7.52–7.50 (m, 1H), 7.40–7.36 (m, 6H), 7.35–7.32 (m, 1H), 7.30–7.28 (m, 3H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 142.2, 133.7, 132.9, 131.4, 129.5, 129.3, 129.2, 128.6, 128.29, 128.26, 127.57, 127.55, 127.5, 123.2, 121.6, 92.8, 88.8. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{14}Cl^+$, 289.0779; found, 289.0777.

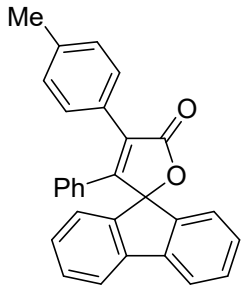



**2-Chloro-2'-(phenylethynyl)-1,1'-biphenyl (2u)**. Following the general procedure

for the synthesis of **2**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether) to afford an orange liquid (1.18 g, 82% yield); $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.64–7.63 (m, 1H), 7.51–7.50 (m, 1H), 7.41–7.37 (m, 3H), 7.36–7.32 (m, 3H), 7.25–7.22 (m, 3H), 7.18–7.16 (m, 2H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 142.0, 139.6, 133.5, 131.8, 131.7, 131.4, 129.7, 129.4, 128.9, 128.2, 128.1, 127.9, 127.7, 126.2, 123.2, 123.0, 92.7, 88.4. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{14}Cl^+$, 289.0779; found, 289.0778.

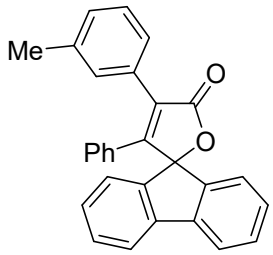



**3',4'-Diphenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3a).** Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (92.7 mg, 96% yield), mp 163.7–165.5 °C; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.57 (d, $J$ = 7.8 Hz, 2H), 7.54–7.53 (m, 2H), 7.39–7.35 (m, 4H), 7.32–7.31 (m, 3H), 7.28–7.25 (m, 2H), 7.04 (t, $J$ = 7.5 Hz, 1H), 6.92 (t, $J$ = 7.8 Hz, 2H), 6.66–6.64 (m, 2H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 172.2, 160.3, 141.3, 140.7, 130.7, 130.4, 129.7, 129.4, 129.3, 128.8, 128.4, 128.2, 127.8, 127.7, 124.1, 120.6, 92.7. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{19}O_2^+$, 387.1380; found, 387.1376.

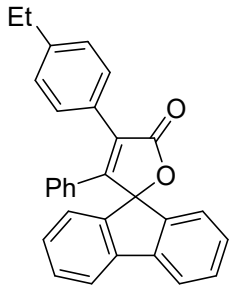



**3'-Phenyl-4'-(*p*-tolyl)-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3b)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (94.1 mg, 94% yield), mp 193.2–194.8 °C; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.58 (d, $J$ = 7.2 Hz, 2H), 7.43 (d, $J$ = 7.8 Hz, 2H), 7.39–7.36 (m, 4H), 7.27 (t, $J$ = 7.2 Hz, 2H),

7.12 (d, $J$ = 7.8 Hz, 2H), 7.05 (t, $J$ = 7.5 Hz, 1H), 6.94 (t, $J$ = 7.8 Hz, 2H), 6.66 (d, $J$ = 7.2 Hz, 2H), 2.33 (s, 3H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$): $\delta$ 172.4, 159.4, 141.3, 140.8, 138.8, 131.0, 130.4, 129.3, 129.2, 129.1, 128.4, 128.2, 127.74, 127.69, 126.8, 124.1, 120.6, 92.6, 21.3. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{29}H_{21}O_2^+$, 401.1536; found, 401.1534.

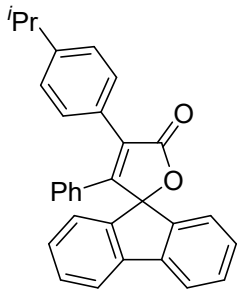



**3'-Phenyl-4'-(*m*-tolyl)-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3c)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (82.1 mg, 82% yield), mp 156.4–157.8 °C; $^1H$ NMR (600 MHz, $CDCl_3$): $\delta$ 7.60 (d, $J$ = 7.2 Hz, 2H), 7.41–7.38 (m, 5H), 7.29–7.27 (m, 2H), 7.25–7.24 (m, 1H), 7.19 (t, $J$ = 7.5 Hz, 1H), 7.14 (d, $J$ = 7.2 Hz, 1H), 7.06 (t, $J$ = 7.5 Hz, 1H), 6.94 (t, $J$ = 7.8 Hz, 2H), 6.66 (d, $J$ = 7.2 Hz, 2H), 2.30 (s, 3H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$): $\delta$ 172.3, 160.0, 141.3, 140.8, 138.1, 130.9, 130.4, 130.0, 129.7, 129.6, 129.3, 128.4, 128.3, 128.2, 128.0, 126.6, 124.1, 92.6, 21.4. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{29}H_{21}O_2^+$, 401.1536; found, 401.1539.

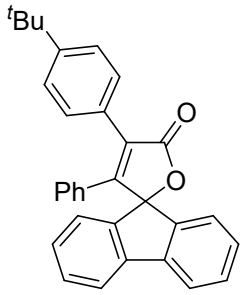



**4'-(4-Ethylphenyl)-3'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3d)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (93.3 mg, 90% yield), mp 180.5–181.3 °C; $^1H$ NMR (600 MHz, $CDCl_3$): $\delta$ 7.59 (d, $J$ = 7.8 Hz, 2H), 7.46 (d, $J$ = 7.8 Hz, 2H), 7.38 (t, $J$ = 7.5 Hz, 4H), 7.29–

7.27 (m, 2H), 7.16 (d, $J$ = 8.4 Hz, 2H), 7.07 (t, $J$ = 7.5 Hz, 1H), 6.95 (t, $J$ = 7.8 Hz, 2H), 6.66 (d, $J$ = 8.4 Hz, 2H), 2.64 (q, $J$ = 7.6 Hz, 2H), 1.22 (t, $J$ = 7.5 Hz, 3H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$): $\delta$ 172.4, 159.5, 145.1, 141.3, 140.8, 131.0, 130.4, 129.4, 129.2, 128.4, 128.3, 127.9, 127.8, 127.7, 127.0, 124.1, 120.6, 92.7, 28.7, 15.2. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{30}H_{23}O_2^+$, 415.1693; found, 415.1695.

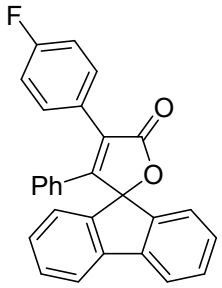



**4'-(4-Isopropylphenyl)-3'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3e)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (91.1 mg, 85% yield), mp 176.8–178.5 °C; $^1H$ NMR (600 MHz, $CDCl_3$): $\delta$ 7.59 (d, $J$ = 7.8 Hz, 2H), 7.49–7.47 (m, 2H), 7.38 (t, $J$ = 7.5 Hz, 4H), 7.28 (t, $J$ = 7.5 Hz, 2H), 7.18 (d, $J$ = 7.8 Hz, 2H), 7.07 (t, $J$ = 7.5 Hz, 1H), 6.95 (t, $J$ = 7.8 Hz, 2H), 6.67 (d, $J$ = 7.8 Hz, 2H), 2.91–2.87 (m, 1H), 1.23 (d, $J$ = 7.2 Hz, 6H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$): $\delta$ 172.5, 159.5, 149.7, 141.3, 140.8, 131.1, 130.4, 129.4, 129.2, 128.4, 128.3, 127.8, 127.6, 127.1, 126.5, 124.1, 120.6, 92.7, 33.9, 23.8. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{31}H_{25}O_2^+$, 429.1849; found, 429.1849.

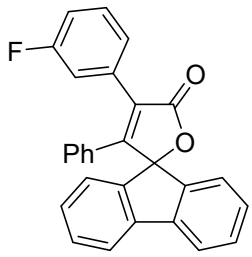



**4'-(4-(*tert*-Butyl)phenyl)-3'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3f)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (95.1 mg, 86% yield), mp 209.2–210.6 °C; $^1H$ NMR (600 MHz, $CDCl_3$):

$\delta$ 7.59 (d, $J$ = 7.8 Hz, 2H), 7.49 (d, $J$ = 8.4 Hz, 2H), 7.40–7.38 (m, 4H), 7.34 (d, $J$ = 8.4 Hz, 2H), 7.28 (t, $J$ = 7.5Hz, 2H), 7.08 (t, $J$ = 7.5 Hz, 1H), 6.96 (t, $J$ = 7.8 Hz, 2H), 6.67 (d, $J$ = 7.8Hz, 2H), 1.30 (s, 9H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$): $\delta$ 172.5, 159.5, 152.0, 141.3, 140.8, 131.1, 130.4, 129.2, 129.1, 128.4, 128.3, 127.8, 127.5, 126.7, 125.4, 124.2, 120.6, 92.7, 34.7, 31.2. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{32}H_{27}O_2^+$, 443.2006; found, 443.2009.

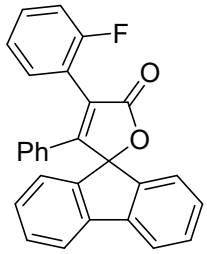



**4'-(4-Fluorophenyl)-3'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3g)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (96.1 mg, 95% yield), mp 195.9–197.5 °C; $^1H$ NMR (600 MHz, $CDCl_3$): $\delta$ 7.60–7.59 (m, 2H), 7.54–7.52 (m, 2H), 7.41–7.38 (m, 4H), 7.30–7.27 (m, 2H), 7.09 (t, $J$ = 7.5 Hz, 1H), 7.01 (t, $J$ = 9.0 Hz, 2H), 6.97 (t, $J$ = 7.8 Hz, 2H), 6.64–6.63 (m, 2H); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$): $\delta$ 172.2, 162.8 (C–F, $1J_{C–F}$ = 248.1 Hz), 160.3, 141.3, 140.5, 131.4 (C–F, $3J_{C–F}$ = 8.3 Hz), 130.7, 130.5, 129.5, 128.4, 127.7, 126.7, 125.8 (C–F, $4J_{C–F}$ = 3.2 Hz), 124.1, 120.7, 115.5 (C–F, $2J_{C–F}$ = 21.5 Hz), 92.8; $^{19}F\{^1H\}$ NMR (565 MHz, $CDCl_3$): $\delta$ -115.5. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}FO_2^+$, 405.1285; found, 405.1286.

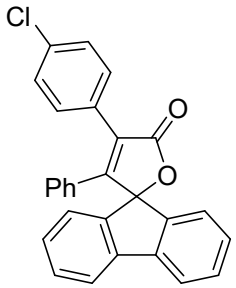



**4'-(3-Fluorophenyl)-3'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3h)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford

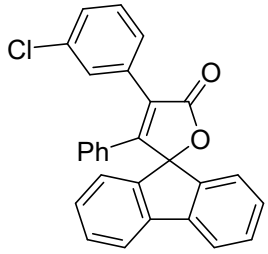
a white solid (83.9 mg, 83% yield), mp 160.2–161.6 °C; $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.59 (d, $J$ = 7.8 Hz, 2H), 7.39 (t, $J$ = 6.6 Hz, 4H), 7.31–7.25 (m, 5H), 7.09 (t, $J$ = 7.5 Hz, 1H), 7.04–7.01 (m, 1H), 6.97 (t, $J$ = 7.8 Hz, 2H), 6.64 (d, $J$ = 7.2 Hz, 2H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 171.8, 162.5 (C–F, $1J_{C–F}$ = 244.8 Hz), 161.6, 141.3, 140.4, 131.8 (C–F, $3J_{C–F}$ = 8.4 Hz), 130.6, 130.4, 130.0, 129.9 (C–F, $3J_{C–F}$ = 8.6 Hz), 128.4, 127.6, 126.6, 126.5, 125.2 (C–F, $4J_{C–F}$ = 3.0 Hz), 124.1, 120.7, 116.4 (C–F, $2J_{C–F}$ = 22.8 Hz), 115.8 (C–F, $2J_{C–F}$ = 20.9 Hz), 92.8; $^{19}$F{$^{1}$H} NMR (565 MHz, $CDCl_3$): $\delta$ -112.3. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}FO_2^+$, 405.1285; found, 405.1285.



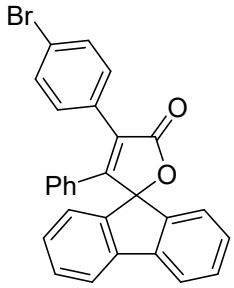

**4'-(2-Fluorophenyl)-3'-phenyl-5'H-spiro[fluorene-9,2'-furan]-5'-one (3i)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (91.0 mg, 90% yield), mp 176.7–178.3 °C; $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.65 (d, $J$ = 7.8 Hz, 2H), 7.57–7.54 (m, 1H), 7.42–7.35 (m, 5H), 7.29 (t, $J$ = 7.2 Hz, 2H), 7.24–7.23 (m, 1H), 7.08–7.05 (m, 2H), 6.92 (t, $J$ = 7.8 Hz, 2H), 6.67 (d, $J$ = 7.2 Hz, 2H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 171.6, 162.5, 160.0 (C–F, $1J_{C–F}$ = 249.0 Hz), 141.3, 140.9, 131.61, 131.59, 130.9 (C–F, $3J_{C–F}$ = 8.3 Hz), 130.61, 130.59, 129.8, 128.6, 128.2, 127.3, 124.4 (C–F, $4J_{C–F}$ = 3.3 Hz), 124.2, 123.5, 120.7, 118.5 (C–F, $2J_{C–F}$ = 15.2 Hz), 116.0 (C–F, $2J_{C–F}$ = 21.3 Hz), 93.1; $^{19}$F{$^{1}$H} NMR (565 MHz, $CDCl_3$): $\delta$ -111.6. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}FO_2^+$, 405.1285; found, 405.1286.



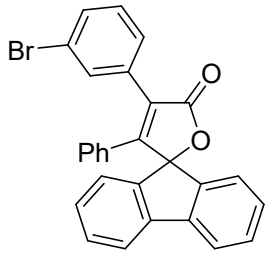

**4'-(4-Chlorophenyl)-3'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3j)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (89.4 mg, 85% yield), mp 186.4–188.1 °C; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.60 (d, $J$ = 7.2 Hz, 2H), 7.49 (dd, $J$ = 1.8 Hz, 8.4 Hz, 2H), 7.39 (t, $J$ = 8.1 Hz, 4H), 7.30–7.27 (m, 4H), 7.09 (t, $J$ = 7.5 Hz, 1H), 6.97 (t, $J$ = 8.1 Hz, 2H), 6.63 (d, $J$ = 7.8 Hz, 2H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 172.0, 160.9, 141.3, 140.4, 134.9, 130.8, 130.6, 129.6, 128.7, 128.5, 128.4, 128.2, 127.6, 126.6, 124.1, 120.7, 92.8. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{28}H_{18}ClO_2^+$, 421.0990; found, 421.0990.



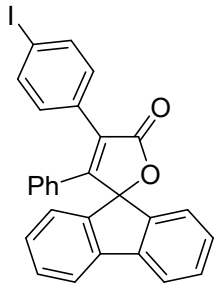

**4'-(3-Chlorophenyl)-3'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3k)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (92.6 mg, 88% yield), mp 176.1–177.5 °C; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.61 (d, $J$ = 7.8 Hz, 2H), 7.57 (s, 1H), 7.42–7.37 (m, 5H), 7.31–7.29 (m, 3H), 7.25 (t, $J$ = 3.9 Hz, 1H), 7.10 (t, $J$ = 7.5 Hz, 1H), 6.98 (t, $J$ = 7.8 Hz, 2H), 6.64 (d, $J$ = 7.2 Hz, 2H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 171.8, 161.7, 141.4, 140.4, 134.4, 131.6, 130.6, 130.3, 129.68, 129.67, 129.5, 129.0, 128.47, 128.45, 127.68, 127.66, 126.5, 124.1, 120.7, 92.8. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}ClO_2^+$, 421.0990; found, 421.0992.



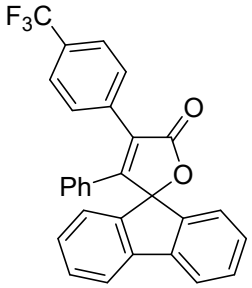

**4'-(4-Bromophenyl)-3'-phenyl-5′*H*-spiro[fluorene-9,2'-furan]-5'-one (3l)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (96.6 mg, 83% yield), mp 196.5–198.3 °C; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.60 (d, $J$ = 7.8 Hz, 2H), 7.45 (d, $J$ = 9.0 Hz, 2H), 7.42–7.38 (m, 6H), 7.29 (t, $J$ = 7.5 Hz, 2H), 7.10 (t, $J$ = 7.5 Hz, 1H), 6.97 (t, $J$ = 8.1 Hz, 2H), 6.64–6.62 (m, 2H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 171.9, 161.0, 141.3, 140.4, 131.7, 131.0, 130.6, 130.5, 129.6, 128.7, 128.5, 128.4, 127.6, 126.7, 124.1, 123.2, 120.7, 92.8. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}BrO_2^+$, 465.0485; found, 465.0487.



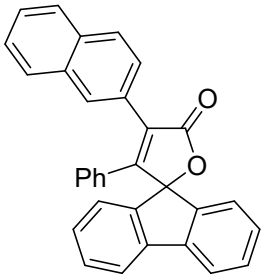

**4'-(3-Bromophenyl)-3'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3m)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (98.9 mg, 85% yield), mp 176.9–178.7 °C; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.73–7.72 (m, 1H), 7.61 (d, $J$ = 7.8 Hz, 2H), 7.47–7.45 (m, 1H), 7.42–7.38 (m, 5H), 7.31–7.29 (m, 2H), 7.18 (t, $J$ = 8.1 Hz, 1H), 7.11 (t, $J$ = 7.5 Hz, 1H), 6.98 (t, $J$ = 7.8 Hz, 2H), 6.64 (d, $J$ = 7.2 Hz, 2H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 171.7, 161.7, 141.4, 140.4, 132.3, 131.9, 131.8, 130.6, 130.3, 129.9, 129.7, 128.47, 128.46, 128.1, 127.7, 126.4, 124.1, 122.4, 120.7, 92.8. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}BrO_2^+$, 465.0485; found, 465.0486.



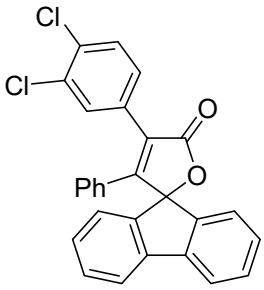

**4'-(4-Iodophenyl)-3'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3n)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a yellow solid (103.8 mg, 81% yield), mp 220.6–222.2 ℃; $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.66 (d, $J$ = 8.4 Hz, 2H), 7.61 (d, $J$ = 7.8 Hz, 2H), 7.41–7.37 (m, 4H), 7.30–7.27 (m, 4H), 7.10 (t, $J$ = 7.5 Hz, 1H), 6.97 (t, $J$ = 7.8 Hz, 2H), 6.63 (d, $J$ = 7.2 Hz, 2H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 171.8, 161.1, 141.4, 140.4, 137.6, 131.1, 130.6, 129.6, 129.2, 128.5, 128.4, 127.6, 126.8, 124.1, 120.7, 95.2, 92.9. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}IO_2^+$, 513.0346; found, 513.0346.



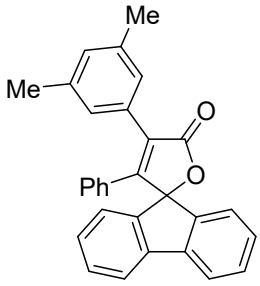

**3'-Phenyl-4'-(4-(trifluoromethyl)phenyl)-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3o).** Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (90.9 mg, 80% yield), mp 176.4–177.9 ℃; $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.66 (d, $J$ = 7.8 Hz, 2H), 7.62–7.58 (m, 4H), 7.42–7.39 (m, 4H), 7.31–7.29 (m, 2H), 7.11 (t, $J$ = 7.2 Hz, 1H), 6.98 (t, $J$ = 7.8 Hz, 2H), 6.63 (d, $J$ = 7.2 Hz, 2H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 171.7, 162.5, 141.4, 140.3, 133.4, 130.7, 130.6 (C–F, $2J_{C–F}$ = 33.7 Hz), 129.84, 129.77, 128.54, 128.48, 127.6, 126.5, 125.3 (C–F, $4J_{C–F}$ = 3.6Hz), 124.1, 123.9(C–F, $1J_{C–F}$ = 270.4 Hz), 120.8, 93.0; $^{19}$F{$^{1}$H} NMR (565 MHz, $CDCl_3$): $\delta$ -62.8. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{29}H_{18}F_3O_2^+$, 455.1253; found, 455.1254.



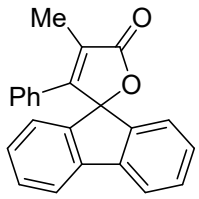

**4'-(Naphthalen-2-yl)-3'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3p)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (89.5 mg, 82% yield), mp 217.2–218.5 °C; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 8.28 (s, 1H), 7.84–7.82 (m, 1H), 7.78–7.77 (m, 1H), 7.70 (d, $J$ = 8.4 Hz, 1H), 7.60 (d, $J$ = 7.8 Hz, 2H), 7.49–7.45 (m, 2H), 7.43 (d, $J$ = 7.8 Hz, 2H), 7.40–7.38 (m, 3H), 7.30–7.27 (m, 2H), 7.06 (t, $J$ = 7.5 Hz, 1H), 6.92 (t, $J$ = 8.1 Hz, 2H), 6.69 (d, $J$ = 7.2 Hz, 2H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 172.3, 160.4, 141.3, 140.8, 133.1, 130.8, 130.5, 129.50, 129.46, 128.6, 128.4, 128.3, 127.9, 127.7, 127.6, 127.2, 126.8, 126.5, 126.3, 124.2, 120.7, 92.7. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{32}H_{21}FO_2^+$, 437.1536; found, 437.1533.



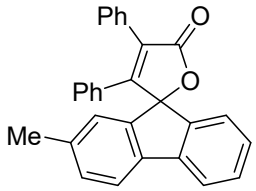

**4'-(3,4-Dichlorophenyl)-3'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3q)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (91.1 mg, 80% yield), mp 186.2–187.8 °C; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.71 (d, $J$ = 2.4 Hz, 1H), 7.61 (d, $J$ = 7.8 Hz, 2H), 7.42–7.36 (m, 5H), 7.32–7.29 (m, 3H), 7.13 (t, $J$ = 7.5 Hz, 1H), 7.00 (t, $J$ = 7.8 Hz, 2H), 6.63 (d, $J$ = 7.8 Hz, 2H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 171.5, 162.1, 141.4, 140.2, 133.1, 132.7, 131.2, 130.7, 130.4, 130.2, 129.8, 129.7, 128.7, 128.6, 128.5, 127.6, 125.5, 124.1, 120.8, 92.9. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{17}Cl_2O_2^+$, 455.0600; found, 455.0597.



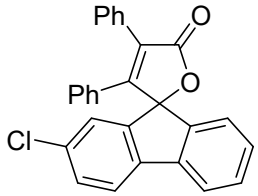

**4'-(3,5-Dimethylphenyl)-3'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3r)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (86.0 mg, 83% yield), mp 194.7–196.2 ℃; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.61 (d, $J$ = 7.2 Hz, 2H), 7.40–7.38 (m, 4H), 7.28 (t, $J$ = 7.5 Hz, 2H), 7.13 (s, 2H), 7.06 (t, $J$ = 7.2 Hz, 1H), 6.97–6.93 (m, 3H), 6.66 (d, $J$ = 7.2 Hz, 2H), 2.24 (s, 6H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 172.4, 159.7, 141.3, 140.9, 137.9, 130.9, 130.5, 130.4, 129.7, 129.2, 128.4, 128.20, 128.15, 127.8, 127.2, 124.1, 120.6, 92.6, 21.2. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{30}H_{23}O_2^+$, 415.1693; found, 415.1695.



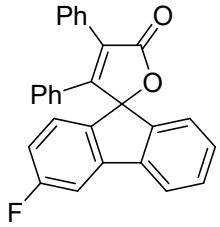

**4'-Methyl-3'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3s)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (36.5 mg, 45% yield), mp 167.3–168.8 ℃; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.59 (d, $J$ = 7.8 Hz, 2H), 7.39–7.36 (m, 2H), 7.29 (d, $J$ = 7.2 Hz, 2H), 7.27–7.24 (m, 2H), 7.14 (t, $J$ = 7.5 Hz, 1H), 7.08 (t, $J$ = 7.5 Hz, 2H), 6.77–6.75 (m, 2H), 2.18 (s, 3H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 174.3, 159.3, 141.2, 141.0, 130.8, 130.3, 129.3, 128.3, 127.5 , 125.6, 124.0, 120.6, 93.1, 10.6. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{23}H_{17}O_2^+$, 325.1223; found, 325.1220.



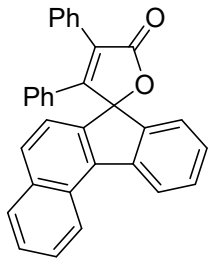

**2-Methyl-3',4'-diphenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3t)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (95.1 mg, 95% yield), mp 195.1–196.7 ℃; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.55–7.54 (m, 3H), 7.48 (d, $J$ = 8.4 Hz, 1H), 7.37–7.35 (m, 2H), 7.33–7.32 (m, 3H), 7.25–7.22 (m, 1H), 7.19 (d, $J$ = 6.6 Hz, 2H), 7.07 (t, $J$ = 7.5 Hz, 1H), 6.95 (t, $J$ = 7.8 Hz, 2H), 6.68–6.66 (m, 2H), 2.36 (s, 3H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 172.3, 160.4, 141.5, 140.9, 140.6, 138.7, 138.6, 131.2, 130.8, 130.4, 129.9, 129.5, 129.3, 128.8, 128.4, 128.3, 127.9, 127.8, 127.7, 124.7, 124.1, 120.4, 120.3, 92.7, 21.5. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{29}H_{21}O_2^+$, 401.1536; found, 401.1540.




**2-Chloro-3',4'-diphenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3u)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (84.2 mg, 80% yield), mp 205.4–206.9 ℃; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.58 (d, $J$ = 7.8 Hz, 1H), 7.54–7.51 (m, 3H), 7.43–7.40 (m, 2H), 7.37–7.35 (m, 2H), 7.34–7.31 (m, 4H), 7.11 (t, $J$ = 7.5 Hz, 1H), 6.98 (t, $J$ = 7.8 Hz, 2H), 6.66 (d, $J$ = 7.2 Hz, 2H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 171.9, 159.6, 142.7, 140.7, 140.3, 139.8, 134.2, 130.7, 130.5, 129.6, 129.53, 129.49, 129.0, 128.7, 128.47, 128.45, 128.1, 127.8, 124.5, 124.3, 121.6, 120.8, 92.1. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}ClO_2^+$, 421.0990; found, 421.0992.

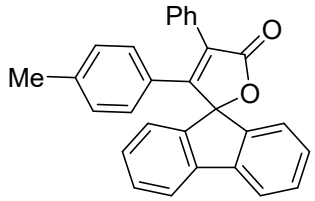



**3-Fluoro-3',4'-diphenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3v)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (87.0 mg, 86% yield), mp 208.4–210.2 °C; $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.58 (d, $J$ = 7.8 Hz, 1H), 7.53–7.52 (m, 2H), 7.44–7.40 (m, 2H), 7.36–7.32 (m, 5H), 7.29–7.27 (m, 1H), 7.10 (t, $J$ = 7.5 Hz, 1H), 6.99–6.95 (m, 3H), 6.65 (d, $J$ = 7.2 Hz, 2H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 172.0, 164.6 (C–F, $1J_{C–F}$ = 247.2 Hz), 159.9, 143.7 (C–F, $3J_{C–F}$ = 9.5 Hz), 141.6, 140.2 (C–F, $4J_{C–F}$ = 2.6 Hz), 136.2 (C–F, $4J_{C–F}$ = 2.1 Hz), 130.7, 130.6, 129.6, 129.5, 129.1, 128.9, 128.45, 128.40, 127.9, 127.7, 125.6 (C–F, $3J_{C–F}$ = 9.5 Hz), 124.2, 121.0, 115.2 (C–F, $2J_{C–F}$ = 23.6 Hz), 108.2 (C–F, $2J_{C–F}$ = 23.6 Hz), 92.0; $^{19}$F{$^{1}$H} NMR (565 MHz, $CDCl_3$): $\delta$ -110.2. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}FO_2^+$, 405.1285; found, 405.1288.

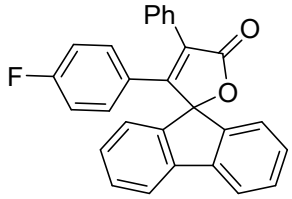



**3',4'-Diphenyl-5'*H*-spiro[benzo[*c*]fluorene-7,2'-furan]-5'-one (3w)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (103.7 mg, 95% yield), mp 203.8–205.2 °C; $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 8.57 (d, $J$ = 8.4 Hz, 1H), 8.18 (d, $J$ = 7.2 Hz, 1H), 7.90 (d, $J$ = 8.4 Hz, 1H), 7.82 (d, $J$ = 8.4 Hz, 1H), 7.63–7.60 (m, 1H), 7.56–7.53 (m, 3H), 7.51–7.47 (m, 3H), 7.34–7.32 (m, 4H), 7.02 (t, $J$ = 7.2 Hz, 1H), 6.89 (t, $J$ = 8.1 Hz, 2H), 6.64 (d, $J$ = 7.8 Hz, 2H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 172.3, 160.3, 142.4, 141.6, 139.1, 137.1, 135.3, 130.7, 130.6, 129.8, 129.7, 129.5, 129.42, 129.37, 129.2, 128.9, 128.44, 128.39, 128.3, 127.7, 127.3, 126.6, 124.1, 124.0, 123.7, 120.7, 92.7. HRMS (ESI) m/z: $[M+H]^+$ calcd for

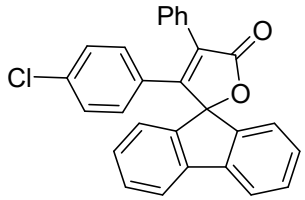
$C_{32}H_{21}O_2^+$, 437.1536; found, 437.1539.



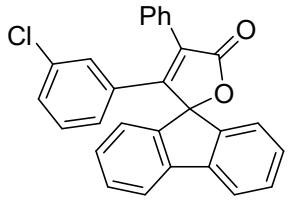

**4'-Phenyl-3'-(*p*-tolyl)-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3x)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (88.1 mg, 88% yield), mp 164.5–166.1 ℃; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.61 (d, $J$ = 7.8 Hz, 2H), 7.55–7.53 (m, 2H), 7.40–7.38 (m, 4H), 7.34–7.33 (m, 3H), 7.28 (t, $J$ = 7.5 Hz, 2H), 6.74 (d, $J$ = 7.8 Hz, 2H), 6.55 (d, $J$ = 8.4 Hz, 2H), 2.10 (s, 3H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 172.4, 160.3, 141.3, 141.0, 139.6, 130.4, 130.1, 129.5, 129.1, 128.7, 128.4, 127.8, 127.7, 127.4, 124.2, 120.6, 92.6, 21.2. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{29}H_{21}O_2^+$, 401.1536; found, 401.1540.



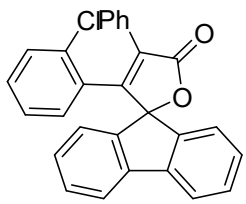

**3'-(4-Fluorophenyl)-4'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3y)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (97.1 mg, 96% yield), mp 188.7–190.3 ℃; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.63 (d, $J$ = 7.8 Hz, 2H), 7.53–7.51 (m, 2H), 7.43–7.40 (m, 2H), 7.38 (d, $J$ = 7.8 Hz, 2H), 7.35 (t, $J$ = 3.3 Hz, 3H), 7.31–7.29 (m, 2H), 6.65–6.64 (m, 4H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 172.1, 162.9 (C–F, $1J_{C–F}$ = 249.2 Hz), 159.1, 141.3, 140.6, 130.6, 129.9 (C–F, $3J_{C–F}$ = 8.4 Hz), 129.6, 129.5, 129.0, 128.54, 128.52, 128.0, 126.8 (C–F, $4J_{C–F}$ = 3.3 Hz), 124.1, 120.8, 115.6 (C–F, $2J_{C–F}$ = 21.6 Hz), 92.6; $^{19}$F{$^1$H} NMR (565 MHz, $CDCl_3$): $\delta$ -110.2. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}FO_2^+$, 405.1285; found, 405.1285.



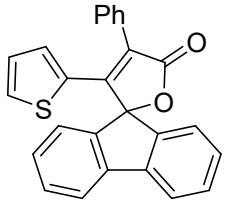

**3'-(4-Chlorophenyl)-4'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3z)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (87.3 mg, 83% yield), mp 236.4–238.1 °C; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.63 (d, $J$ = 7.8 Hz, 2H), 7.52–7.51 (m, 2H), 7.42 (t, $J$ = 7.5 Hz, 2H), 7.38 (d, $J$ = 7.2 Hz, 2H), 7.36–7.35 (m, 3H), 7.30 (t, $J$ = 7.5 Hz, 2H), 6.93 (d, $J$ = 8.4 Hz, 2H), 6.58 (d, $J$ = 9.0 Hz, 2H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 171.9, 158.9, 141.3, 140.5, 135.5, 130.7, 129.5, 129.2, 129.1, 128.7, 128.6, 128.5, 124.1, 120.8, 92.5. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}ClO_2^+$, 421.0990; found, 421.0994.



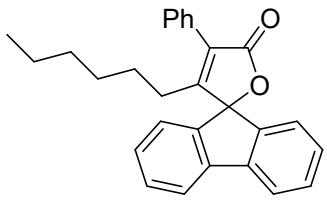

**3'-(3-Chlorophenyl)-4'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3aa)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (63.1 mg, 60% yield), mp 218.7–220.5 °C; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.62 (d, $J$ = 7.8 Hz, 2H), 7.52–7.51 (m, 2H), 7.42–7.38 (m, 4H), 7.35–7.34 (m, 3H), 7.32–7.29 (m, 2H), 7.05–7.04 (m, 1H), 6.88 (t, $J$ = 8.1 Hz, 1H), 6.59 (t, $J$ = 1.8 Hz, 1H), 6.53 (d, $J$ = 7.8 Hz, 1H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 171.9, 158.6, 141.3, 140.2, 134.2, 132.6, 130.7, 129.7, 129.5, 129.4, 129.2, 129.1, 128.6, 128.53, 128.52, 127.7, 126.0, 124.1, 120.8, 92.6. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}ClO_2^+$, 421.0990; found, 421.0994.



**3'-(2-chlorophenyl)-4'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3ab).** Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (53.7 mg, 51% yield), mp 212.2–213.6 °C; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.60 (s, 1H), 7.55–7.47 (m, 6H), 7.41 (s, 1H), 7.33–7.27 (m, 4H), 7.24–7.21 (m, 1H), 7.07–7.03 (m, 2H), 6.88–6.85 (m, 1H), 6.47–6.45 (m, 1H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 171.8, 156.4, 141.9, 140.8, 140.3, 139.5, 132.1, 130.7, 130.3, 130.24, 130.19, 130.1, 129.7, 129.0, 128.4, 128.33, 128.31, 127.8, 126.3, 125.2, 124.2, 120.8, 120.2, 93.2. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}ClO_2^+$, 421.0990; found, 421.0992.

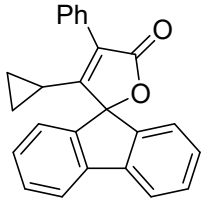



**4'-Phenyl-3'-(thiophen-2-yl)-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3ac)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a yellow solid (60.8 mg, 62% yield), mp 176.3–177.8 °C; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.86–7.83 (m, 3H), 7.55–7.54 (m, 2H), 7.52–7.49 (m, 1H), 7.47–7.43 (m, 2H), 7.41–7.38 (m, 4H), 7.19–7.16 (m, 1H), 7.15–7.14 (m, 1H), 6.75–6.74 (m, 1H), 6.663–6.655 (m, 1H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 171.4, 158.9, 143.1, 135.7, 134.4, 132.0, 131.6, 129.6, 129.2, 129.09, 129.07, 128.6, 128.3, 127.7, 126.9, 126.8, 126.7, 126.6, 125.5, 124.9, 124.5, 122.8, 84.8. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{26}H_{17}O_2S^+$, 393.0944; found, 393.0940.

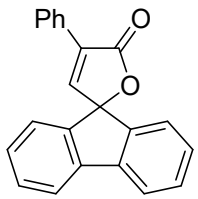



**3'-hexyl-4'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3ad).** Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 15:1) to afford a yellow

liquid (74.0 mg, 75% yield); $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.71 (d, $J$ = 7.8 Hz, 2H), 7.61 (d, $J$ = 7.2 Hz, 2H), 7.49–7.44 (m, 4H), 7.41 (t, $J$ = 7.5 Hz, 1H), 7.31–7.28 (m, 4H), 1.99 (t, $J$ = 7.5 Hz, 2H), 1.00–0.95 (m, 2H), 0.91–0.89 (m, 4H), 0.85–0.81 (m, 2H), 0.69 (t, $J$ = 7.2 Hz, 3H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 172.9, 164.9, 141.2, 140.9, 130.4, 130.3, 128.9, 128.6, 128.5, 128.2, 127.7, 124.1, 120.6, 93.1, 30.7, 29.0, 27.5, 26.5, 22.0, 13.8. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{27}O_2^+$, 395.2006; found, 395.2003.

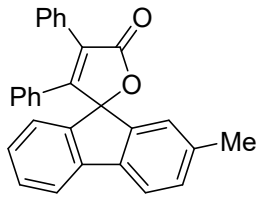



**3'-Cyclopropyl-4'-phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3ae).** Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (61.3 mg, 70% yield), mp 168.5–169.7 °C; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.71–7.69 (m, 4H), 7.49 (t, $J$ = 7.8 Hz, 2H), 7.47–7.44 (m, 2H), 7.41 (t, $J$ = 7.5 Hz, 1H), 7.33–7.30 (m, 4H), 1.60–1.55 (m, 1H), 0.50–0.47 (m, 2H), 0.16–0.14 (m, 2H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 172.4, 164.1, 141.5, 140.9, 130.5, 130.2, 129.6, 128.6, 128.4, 128.1, 124.2, 120.7, 91.9, 10.4, 8.3. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{25}H_{19}O_2^+$, 351.1380; found, 351.1379.

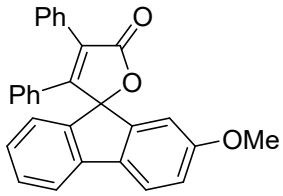



**4'-Phenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3af)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (62.8 mg, 81% yield), mp 165.4–166.8 °C; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.98–7.96 (m, 2H), 7.70 (d, $J$ = 7.2 Hz, 2H), 7.46–7.45 (m, 2H), 7.44–7.40 (m, 3H), 7.35 (s, 1H), 7.30–7.27 (m, 4H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 171.9, 147.8, 140.7, 140.0, 131.1, 130.6, 129.6, 129.2, 128.8, 128.3, 127.2, 124.4, 120.7, 90.5. HRMS (ESI) m/z:

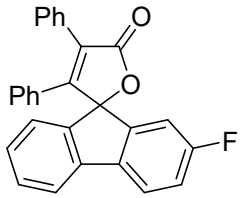
$[M+H]^+$ calcd for $C_{22}H_{15}O_2^+$, 311.1067; found, 311.1068.



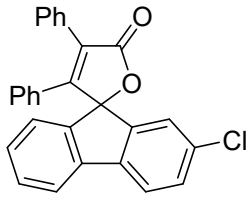

**2-Methyl-3',4'-diphenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3ag)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (91.1 mg, 91% yield), mp 171.1–172.4 °C; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.56–7.54 (m, 3H), 7.48 (d, $J$ = 8.4 Hz, 1H), 7.37–7.35 (m, 2H), 7.33 (t, $J$ = 3.0 Hz, 3H), 7.24 (t, $J$ = 7.5 Hz, 1H), 7.20 (d, $J$ = 5.4 Hz, 2H), 7.07 (t, $J$ = 7.5 Hz, 1H), 6.95 (t, $J$ = 7.8 Hz, 2H), 6.67 (d, $J$ = 7.2 Hz, 2H), 2.36 (s, 3H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 172.3, 160.4, 141.5, 141.0, 140.6, 138.7, 138.6, 131.2, 130.8, 130.4, 129.9, 129.5, 129.3, 128,8, 128.4, 128.3, 127.9, 127.8, 127.7, 124.7, 124.1, 120.4, 120.3, 92.7, 21.5. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{29}H_{21}O_2^+$, 401.1536; found, 401.1537.



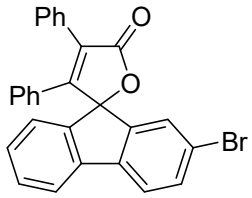

**2-Methoxy-3',4'-diphenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3ah)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (93.7 mg, 90% yield), mp 191.4–193.2 °C; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.54–7.53 (m, 2H), 7.51–7.50 (m, 2H), 7.37–7.33 (m, 5H), 7.22–7.20 (m, 1H), 7.09 (t, $J$ = 7.5 Hz, 1H), 6.97 (t, $J$ = 8.1 Hz, 2H), 6.94–6.92 (m, 2H), 6.69–6.67 (m, 2H), 3.80 (s, 3H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 172.3, 160.38, 160.37, 142.6, 141.4, 140.3, 134.1, 130.8, 130.5, 129.8, 129.5, 129.4, 128.8, 128.4, 128.3, 127.8, 127.7, 127.2, 124.0, 121.6, 119.8, 116.2, 109.8, 92.5, 55.7. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{29}H_{21}O_3^+$, 417.1485; found, 417.1489.



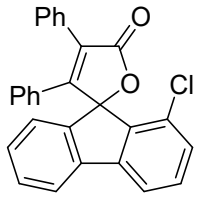

**2-Fluoro-3',4'-diphenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3ai)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (94.0 mg, 93% yield), mp 185.8–187.5 ℃; $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.56–7.52 (m, 4H), 7.41–7.39 (m, 2H), 7.334–7.325 (m, 3H), 7.29 (t, $J$ = 7.5 Hz, 1H), 7.11–7.06 (m, 3H), 6.97 (t, $J$ = 8.1 Hz, 2H), 6.66 (d, $J$ = 7.2 Hz, 2H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 171.9, 163.0 (C–F, $1J_{C–F}$ = 247.5 Hz), 159.7, 143.1 (C–F, $3J_{C–F}$ = 8.0 Hz), 140.7, 140.5, 137.3 (C–F, $4J_{C–F}$ = 2.3 Hz), 130.7, 130.5, 129.52, 129.46, 129.0, 128.5, 128.4, 128.1, 128.0, 127.7, 124.2, 121.8 (C–F, $3J_{C–F}$ = 8.4 Hz), 120.4, 117.5 (C–F, $2J_{C–F}$ = 22.8 Hz), 111.8 (C–F, $2J_{C–F}$ = 23.7 Hz), 92.1 (C–F, $4J_{C–F}$ = 1.5 Hz); $^{19}$F{$^{1}$H} NMR (565 MHz, $CDCl_3$): $\delta$ -112.1. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}FO_2^+$, 405.1285; found, 405.1285.



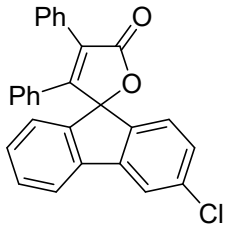

**2-Chloro-3',4'-diphenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3aj)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (92.6 mg, 88% yield), mp 163.2–164.7 ℃; $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.59 (d, $J$ = 7.8 Hz, 1H), 7.54–7.53 (m, 3H), 7.44–7.40 (m, 2H), 7.38–7.37 (m, 2H), 7.34–7.31 (m, 4H), 7.12 (t, $J$ = 7.5 Hz, 1H), 6.99 (t, $J$ = 7.8 Hz, 2H), 6.66 (d, $J$ = 7.8 Hz, 2H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 171.9, 159.6, 142.7, 140.7, 140.3, 139.9, 134.2, 130.7, 130.5, 129.6, 129.54, 129.50, 129.0, 128.8, 128.48, 128.46, 128.1, 124.6, 124.3, 121.7, 120.8, 92.2. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}ClO_2^+$, 421.0990; found, 421.0987.



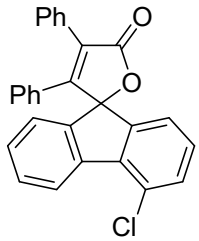

**2-Bromo-3',4'-diphenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3ak)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (98.9 mg, 85% yield), mp 208.6–209.9 °C; $^1$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.58 (d, $J$ = 7.8 Hz, 1H), 7.54–7.50 (m, 4H), 7.46–7.45 (m, 1H), 7.42–7.39 (m, 2H), 7.35–7.31 (m, 4H), 7.10 (t, $J$ = 7.5 Hz, 1H), 6.98 (t, $J$ = 7.8 Hz, 2H), 6.67–6.65 (m, 2H); $^{13}$C{$^1$H} NMR (150 MHz, $CDCl_3$): $\delta$ 171.9, 159.5, 142.9, 140.5, 140.3, 133.6, 130.7, 130.5, 129.6, 129.50, 129.48, 129.0, 128.9, 128.46, 128.45, 128.1, 127.8, 127.4, 124.2, 122.03, 121.99, 120.8, 92.1. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}BrO_2^+$, 465.0485; found, 465.0489.



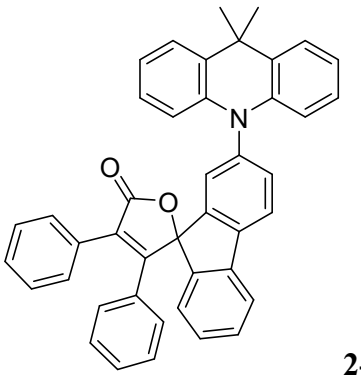

**1-Chloro-3',4'-diphenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3al)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (53.7 mg, 51% yield), mp 205.2–206.9 °C; $^1$H NMR (600 MHz, DMSO): $\delta$ 7.91 (d, $J$ = 7.8 Hz, 1H), 7.86 (d, $J$ = 7.8 Hz, 1H), 7.69 (d, $J$ = 7.8 Hz, 1H), 7.57–7.53 (m, 2H), 7.47 (t, $J$ = 7.5 Hz, 1H), 7.43–7.39 (m, 6H), 7.17 (t, $J$ = 7.2 Hz, 1H), 7.08 (t, $J$ = 7.8 Hz, 2H), 6.64 (d, $J$ = 7.2 Hz, 2H); $^{13}$C{$^1$H} NMR (150 MHz, DMSO): $\delta$ 171.2, 157.7, 143.6, 140.1, 139.4, 135.7, 133.1, 131.2, 131.1, 130.3, 129.8, 129.71, 129.69, 129.6, 129.5, 129.1, 129.0, 128.6, 128.5, 127.1, 124.4, 121.7, 120.1, 92.2. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}ClO_2^+$, 421.0990; found, 421.0986.



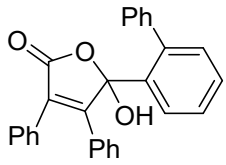

**3-Chloro-3',4'-diphenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3al').** Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (21.0 mg, 20% yield), mp 215.7–217.5 ℃; $^{1}$H NMR (600 MHz, DMSO): $\delta$ 8.02 (d, $J$ = 1.8 Hz, 1H), 7.93 (d, $J$ = 7.8 Hz, 1H), 7.71 (d, $J$ = 7.8 Hz, 1H), 7.68 (d, $J$ = 7.2 Hz, 1H), 7.55–7.52 (m, 1H), 7.49–7.47 (m, 2H), 7.46–7.42 (m, 2H), 7.39–7.38 (m, 3H), 7.15 (t, $J$ = 7.5 Hz, 1H), 7.07 (t, $J$ = 7.8 Hz, 2H), 6.64 (d, $J$ = 7.2 Hz, 2H); $^{13}$C{$^{1}$H} NMR (150 MHz, DMSO): $\delta$ 171.2, 158.8, 142.9, 140.7, 139.5, 139.0, 135.7, 131.0, 130.4, 129.7, 129.6, 129.5, 128.9, 128.5, 128.4, 128.3, 127.4, 126.3, 124.7, 121.8, 121.5, 91.6. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}ClO_2^+$, 421.0990; found, 421.0987.




**4-Chloro-3',4'-diphenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (3am)**. Following the general procedure for the synthesis of **3**, the crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9:1) to afford a white solid (69.4 mg, 66% yield), mp 188.2–189.8 ℃; $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 8.27 (d, $J$ = 7.8 Hz, 1H), 7.53–7.52 (m, 2H), 7.47–7.45 (m, 1H), 7.43 (d, $J$ = 7.2 Hz, 1H), 7.38–7.33 (m, 5H), 7,30 (d, $J$ = 7.8 Hz, 1H), 7.21 (t, $J$ = 7.5 Hz, 1H), 7.10 (t, $J$ = 7.5 Hz, 1H), 6.98 (t, $J$ = 7.8 Hz, 2H), 6.64 (d, $J$ = 7.8 Hz, 2H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 172.0, 159.7, 143.2, 140.7, 140.0, 138.1, 132.0, 130.7, 130.6, 129.6, 129.54, 129.47, 129.01, 128.96, 128.9, 128.5, 128.1, 127.7, 124.4, 124.0, 122.5, 92.2. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{18}ClO_2^+$, 421.0990; found, 421.0993.



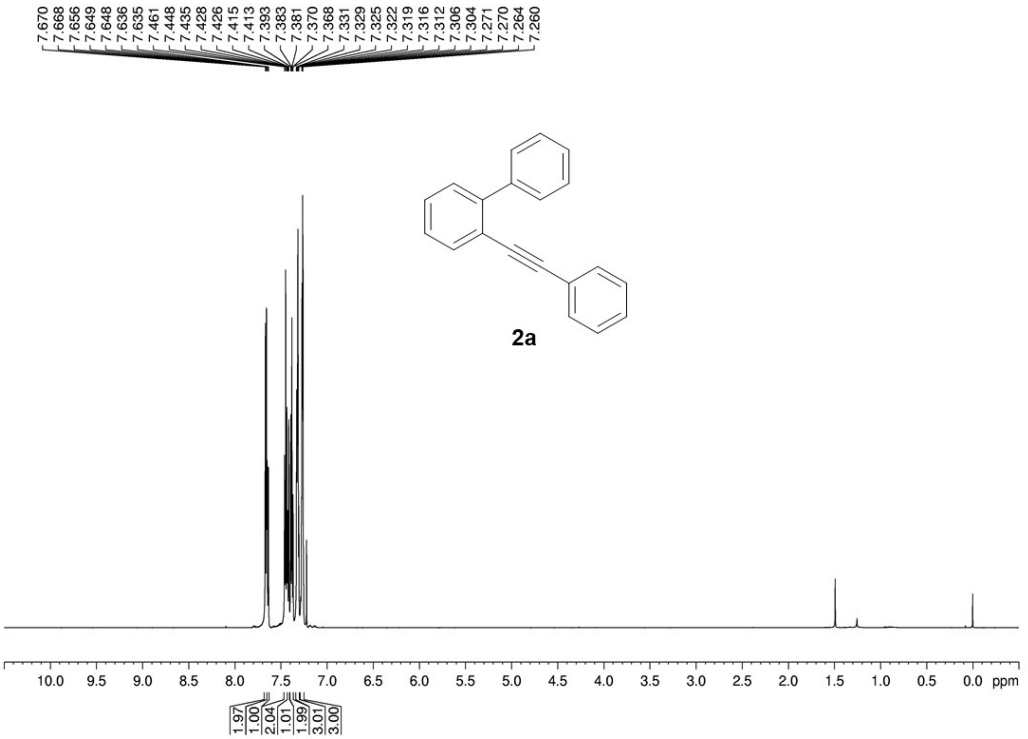
**2-(9,9-Dimethylacridin-10(9*H*)-yl)-3',4'-diphenyl-5'*H*-spiro[fluorene-9,2'-furan]-5'-one (4)**. White solid, mp 212.6–213.8 °C; $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.82 (d, $J$ = 7.8 Hz, 1H), 7.73 (d, $J$ = 7.8 Hz, 1H), 7.54 (t, $J$ = 7.5 Hz, 2H), 7.48–7.47 (m, 2H), 7.46–7.42 (m, 3H), 7.33 (d, $J$ = 1.8 Hz, 1H), 7.30–7.26 (m, 4H), 7.20 (t, $J$ = 7.5 Hz, 1H), 7.06 (t, $J$ = 7.8 Hz, 2H), 6.92–6.87 (m, 4H), 6.69 (d, $J$ = 7.8 Hz, 2H), 6.06–6.04 (m, 2H), 1.67 (s, 6H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 172.0, 160.1, 143.6, 141.3, 141.2, 141.1, 140.7, 140.6, 133.5, 130.9, 129.9, 129.43, 129.40, 129.3, 129.0, 128.9, 128.43, 128.38, 127.91, 127.89, 127.7, 126.4, 125.4, 124.5, 123.0, 121.0, 120.7, 113.8, 92.3, 35.9, 31.4. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{43}H_{32}NO_2^+$, 594.2428; found, 594.2429.

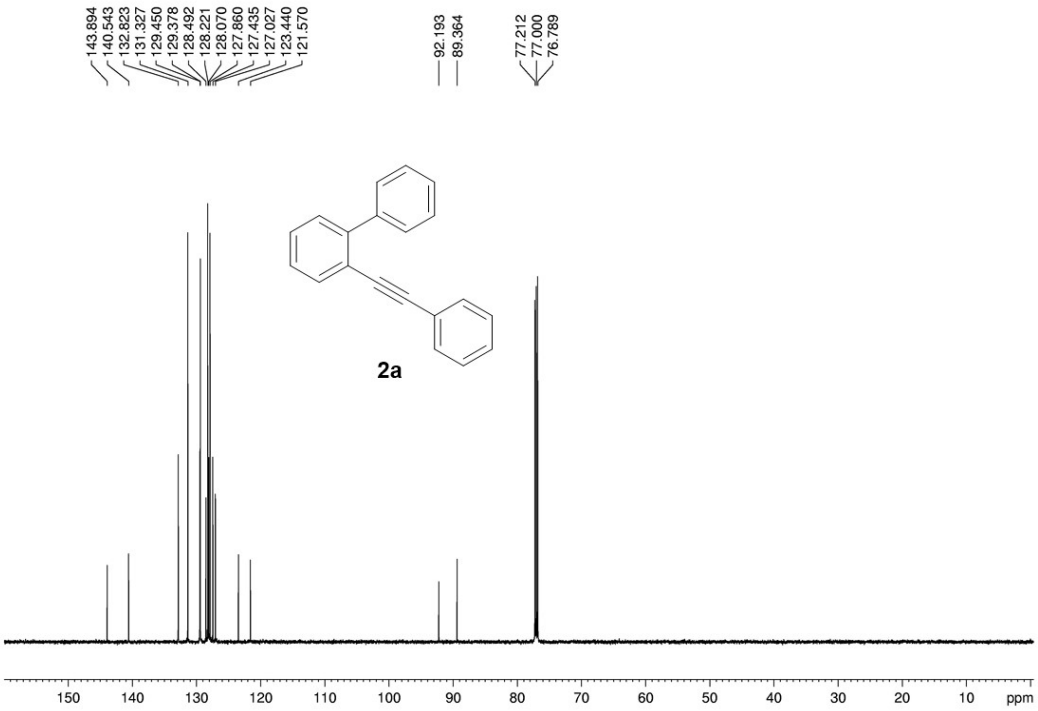


**5-([1,1'-Biphenyl]-2-yl)-5-hydroxy-3,4-diphenylfuran-2(5*H*)-one (5)**. White solid mp 168.7– 170.2 °C; $^{1}$H NMR (600 MHz, $CDCl_3$): $\delta$ 7.96–7.94 (m, 1H), 7.40–7.30 (m, 5H), 7.25–7.20 (m, 5H), 7.17 (t, $J$ = 7.5 Hz, 2H), 7.13 (t, $J$ = 7.5 Hz, 2H), 7.03–7.02 (m, 1H), 6.97 (d, $J$ = 7.8 Hz, 2H), 6.87 (s, 1H), 4.55 (s, 1H); $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$): $\delta$ 170.8, 157.5, 141.2, 140.6, 133.5, 132.0, 130.0, 129.7, 129.6, 129.35, 129.28, 128.8, 128.6, 128.15, 128.12, 127.9, 127.8, 127.5, 105.1. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{21}O_3^+$, 405.1485; found, 405.1483.

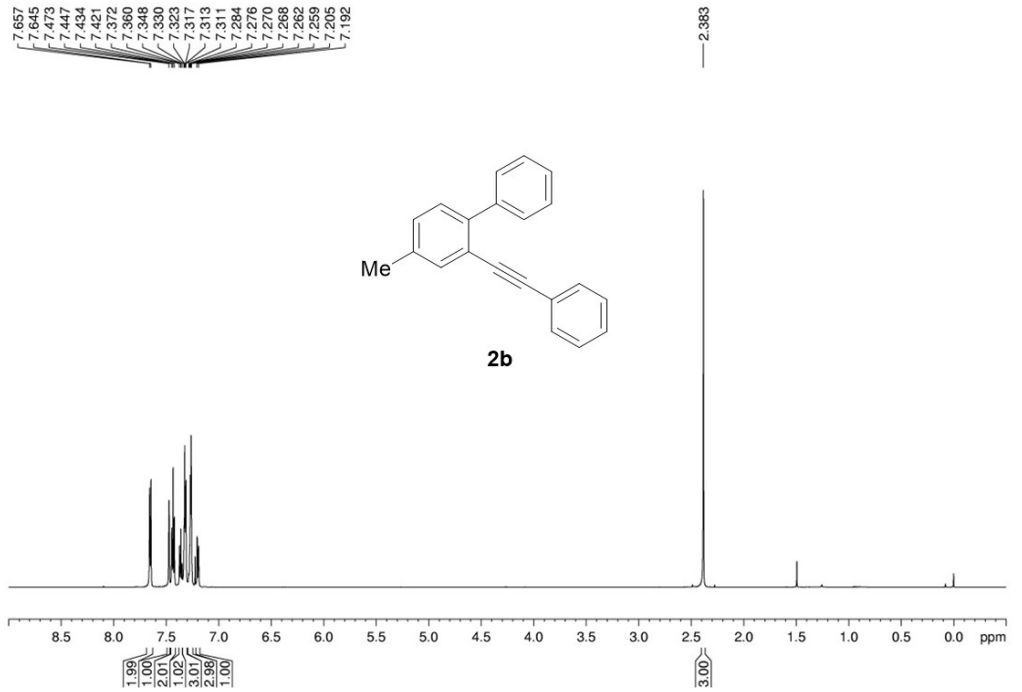
## 6. $^{1}$H and $^{13}$C NMR spectra of *o*-alkynyl biaryls (2) and all products

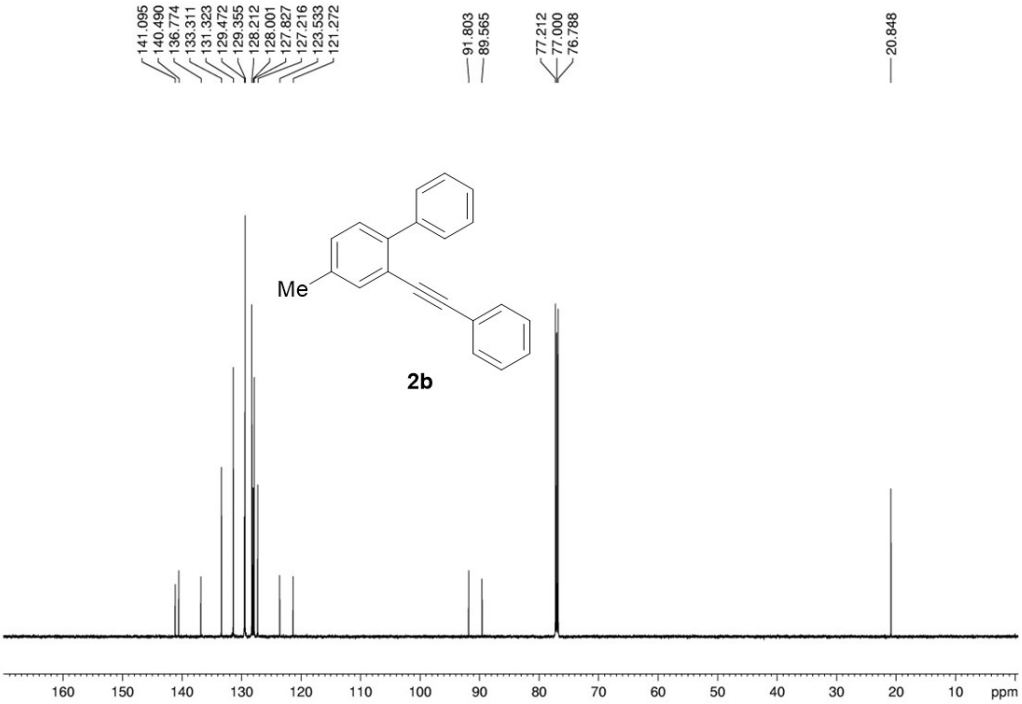
$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **2a**



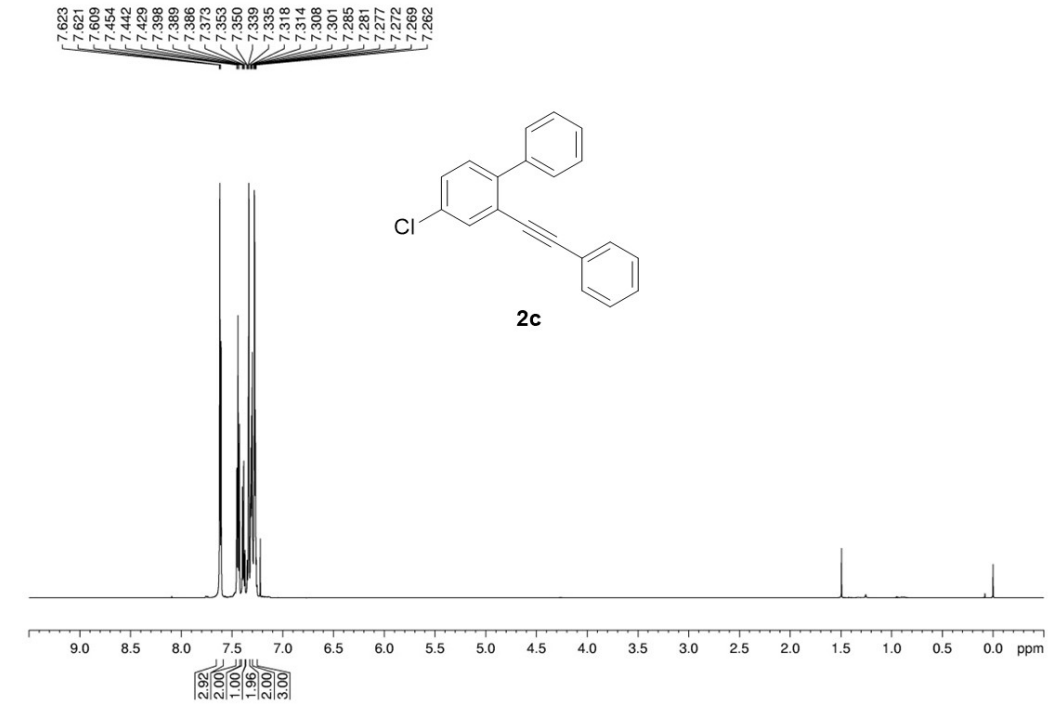



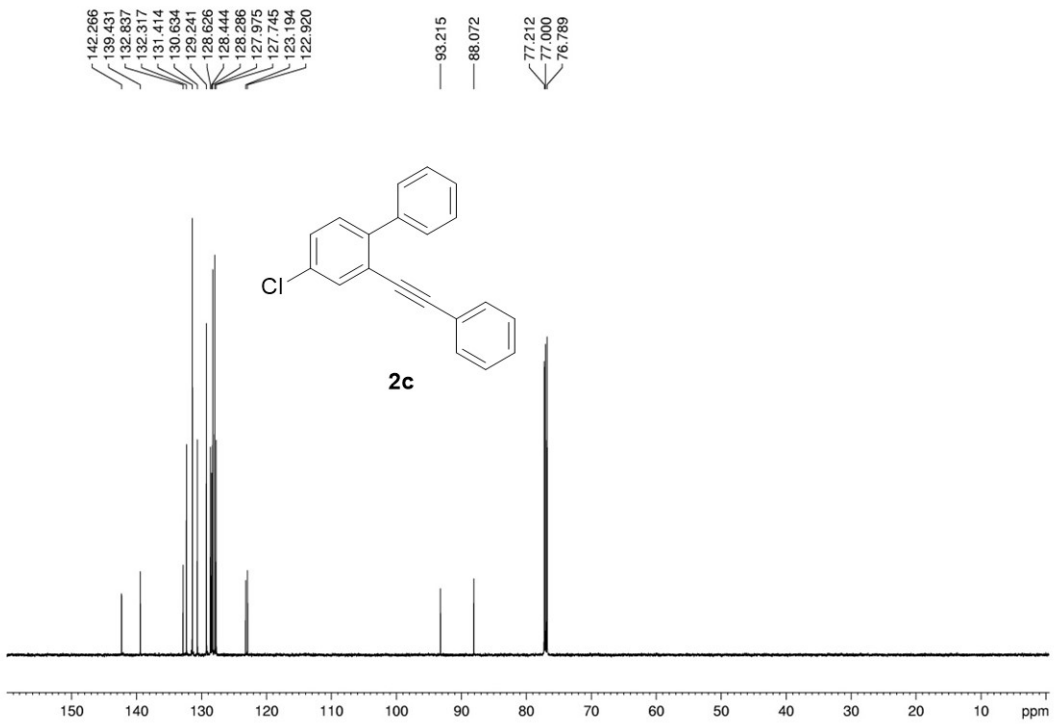
$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **2b**



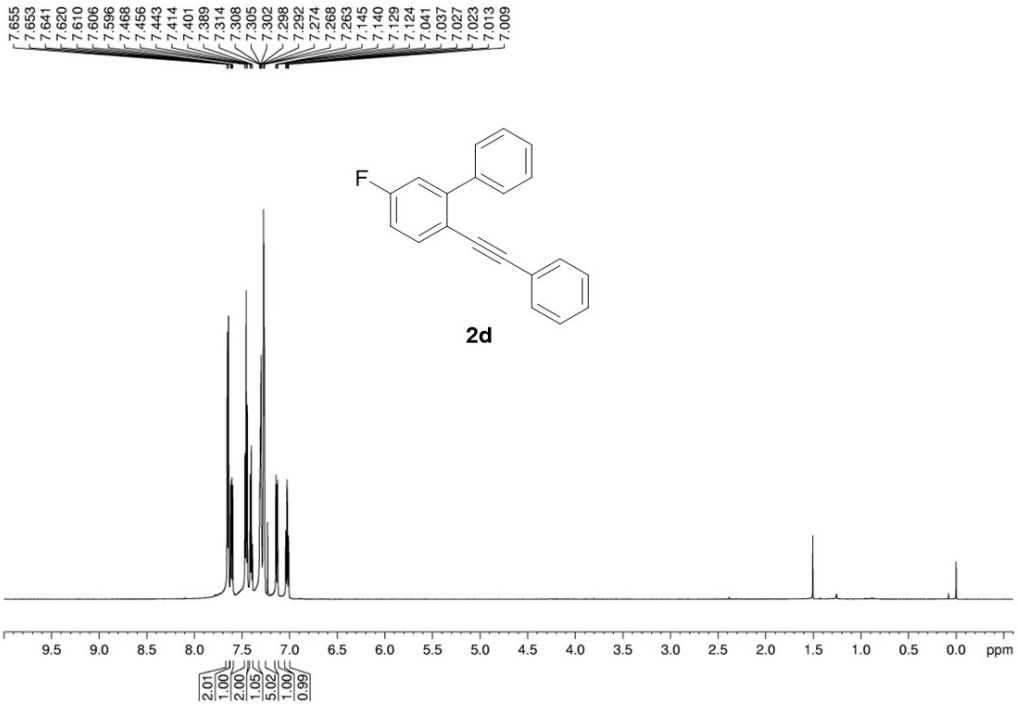



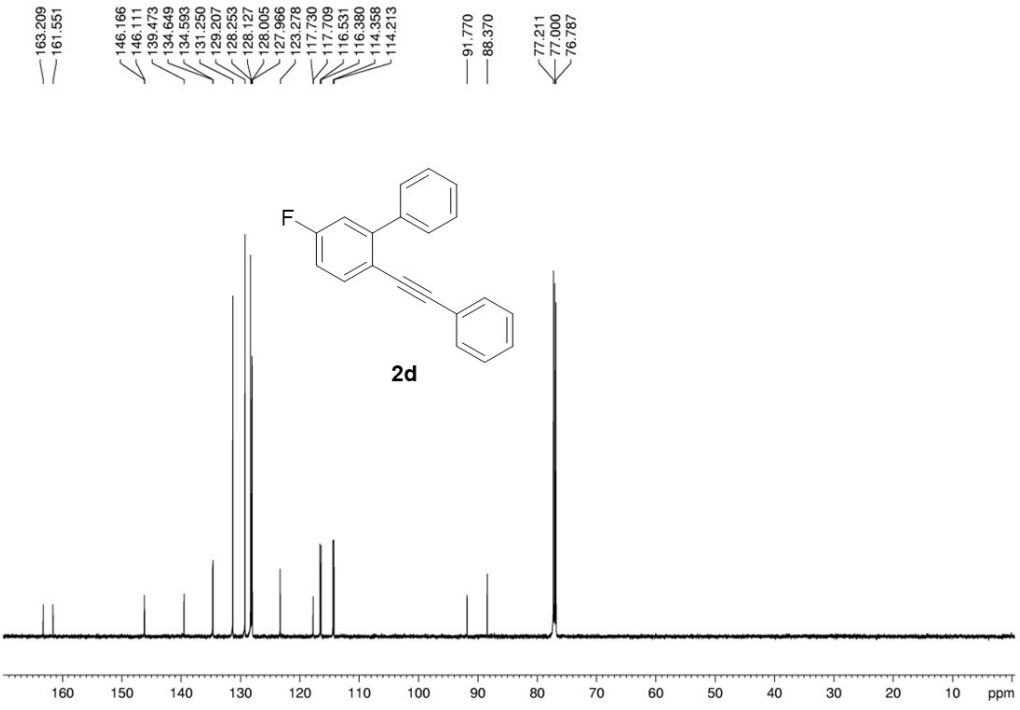
$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **2c**

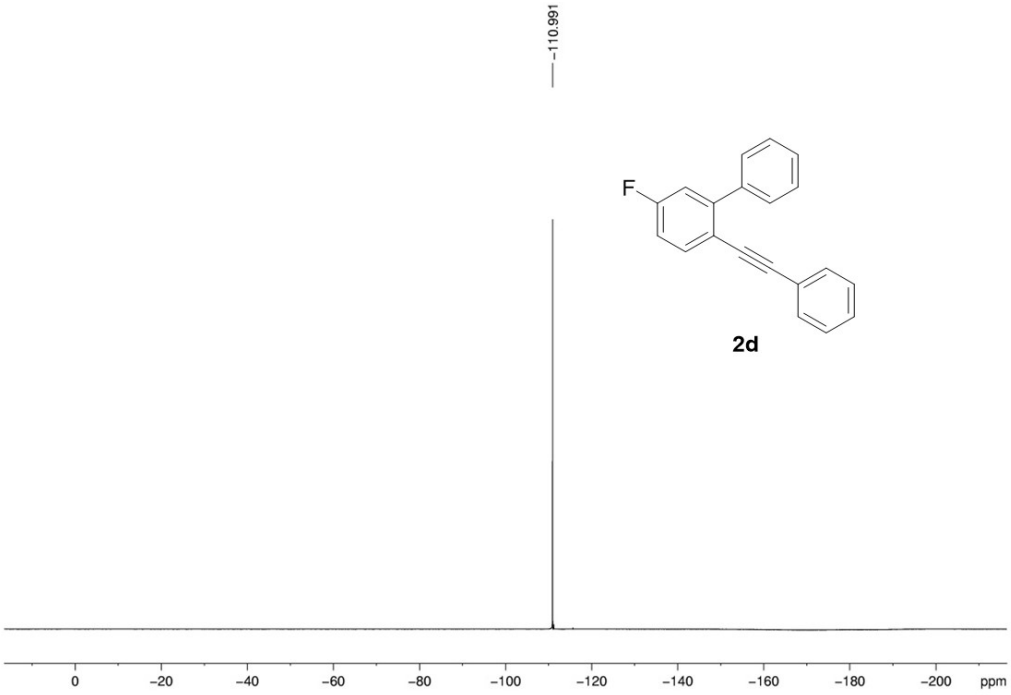


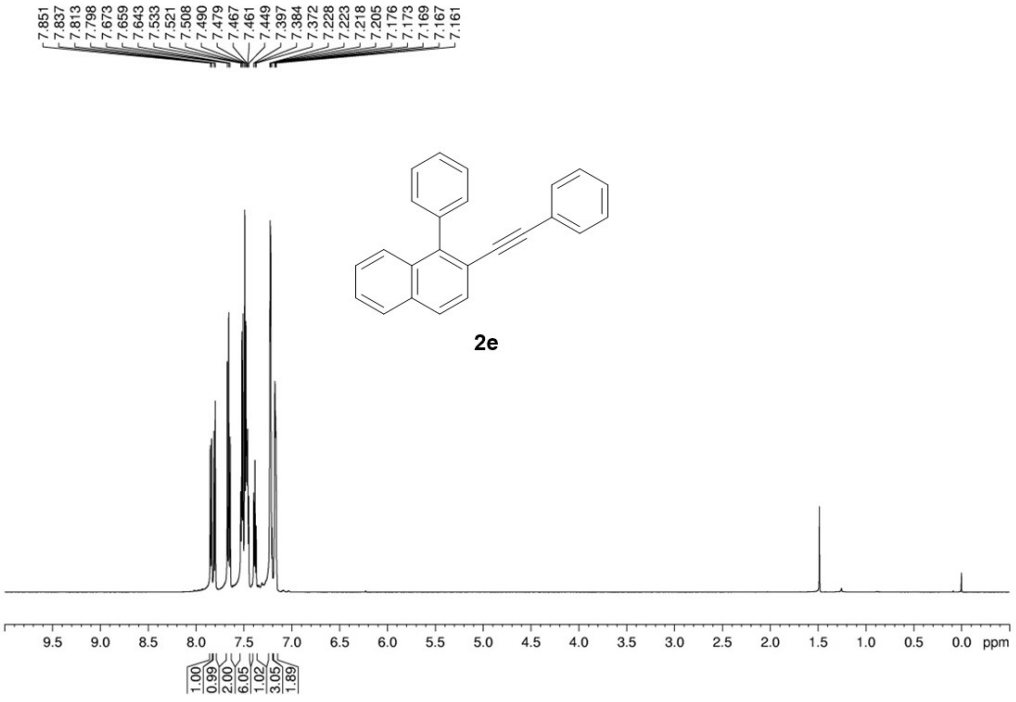
$^{1}H$ NMR (600 MHz, $CDCl_3$), $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) and $^{19}F\{^{1}H\}$ NMR (565 MHz, $CDCl_3$) of **2d**

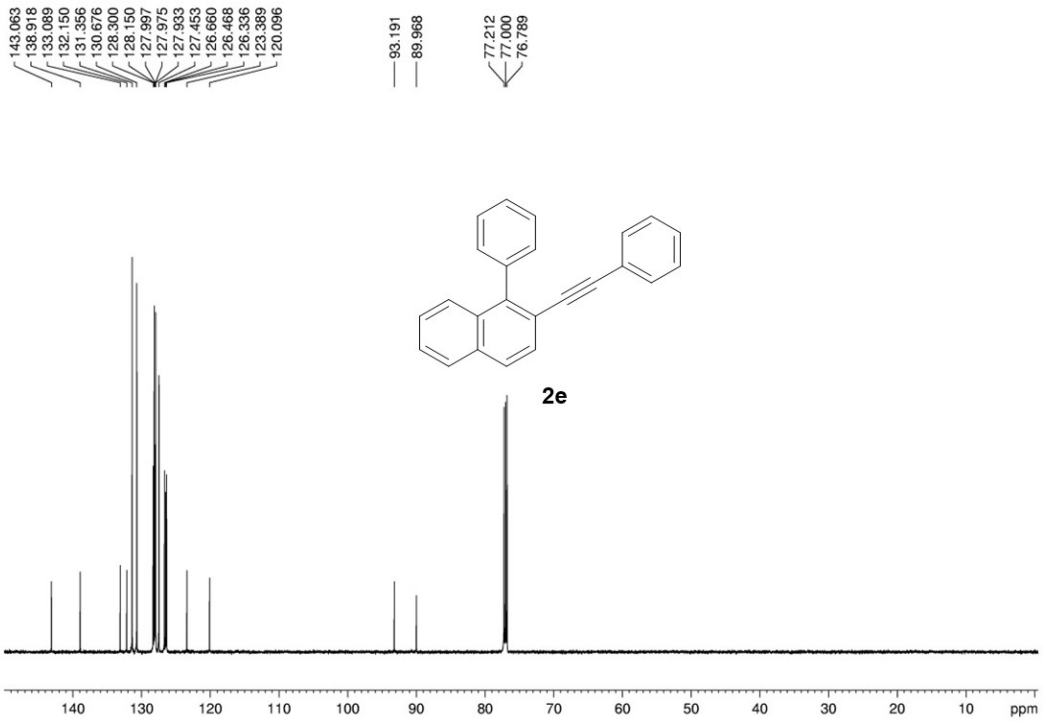


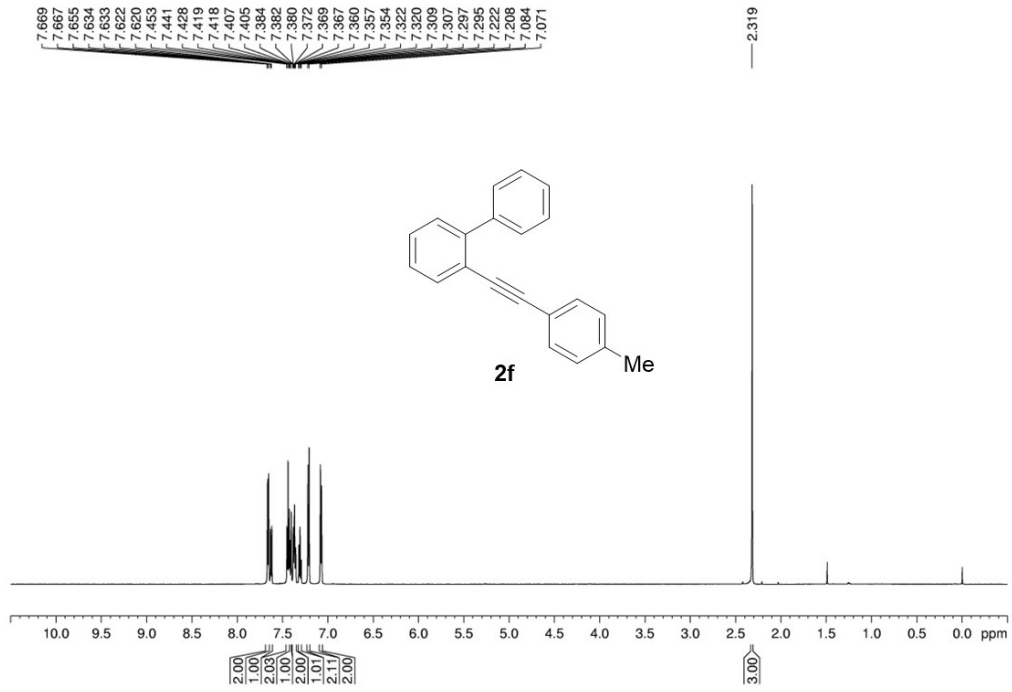


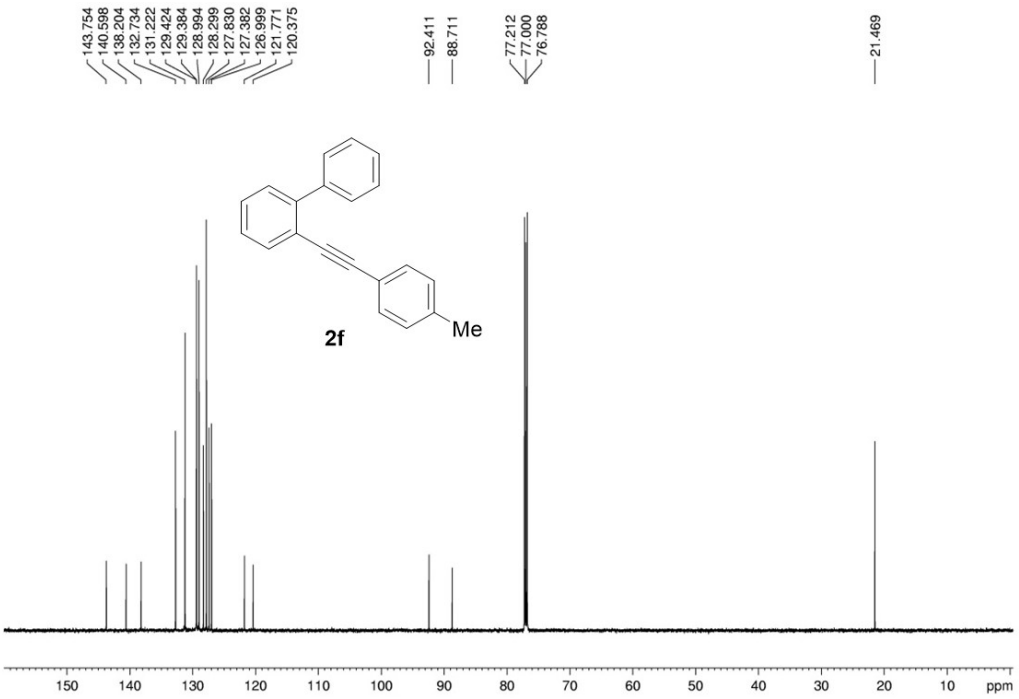

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **2e**



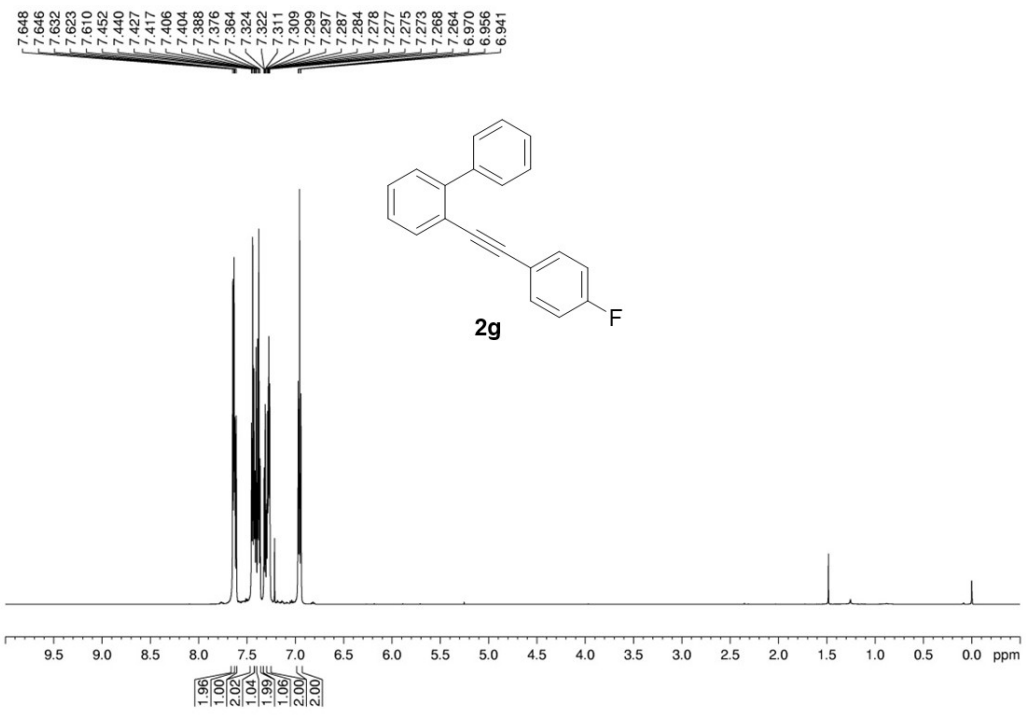


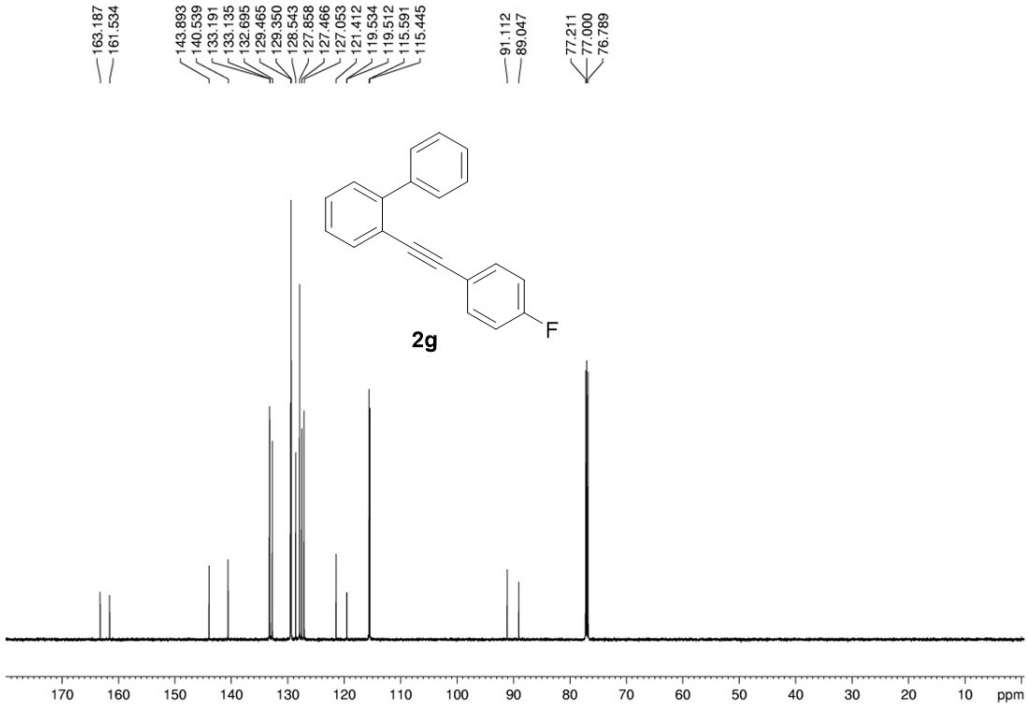

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **2f**

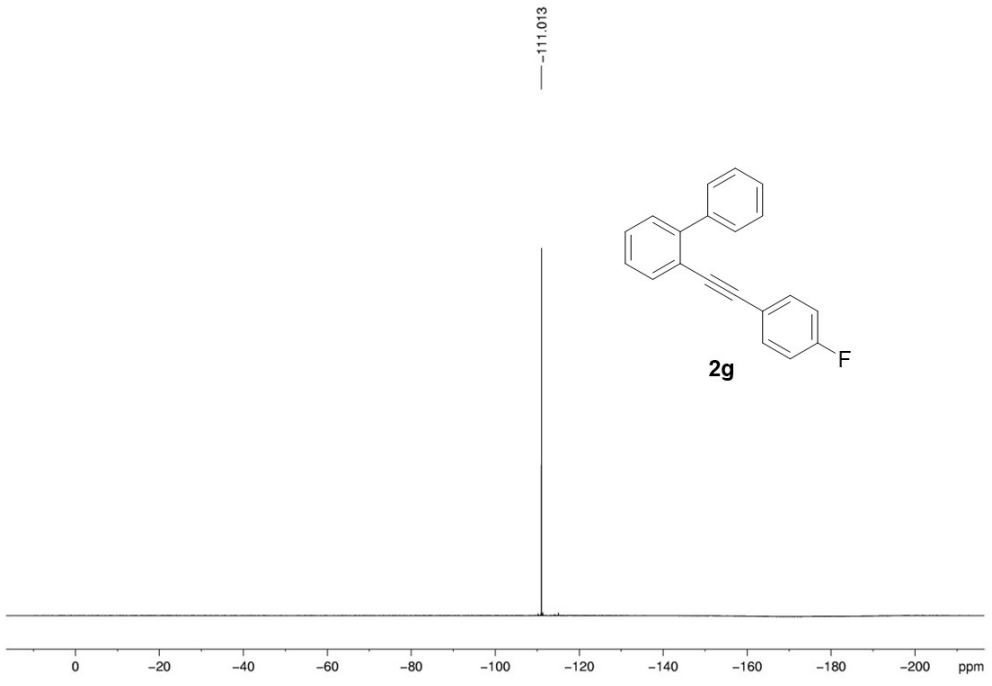




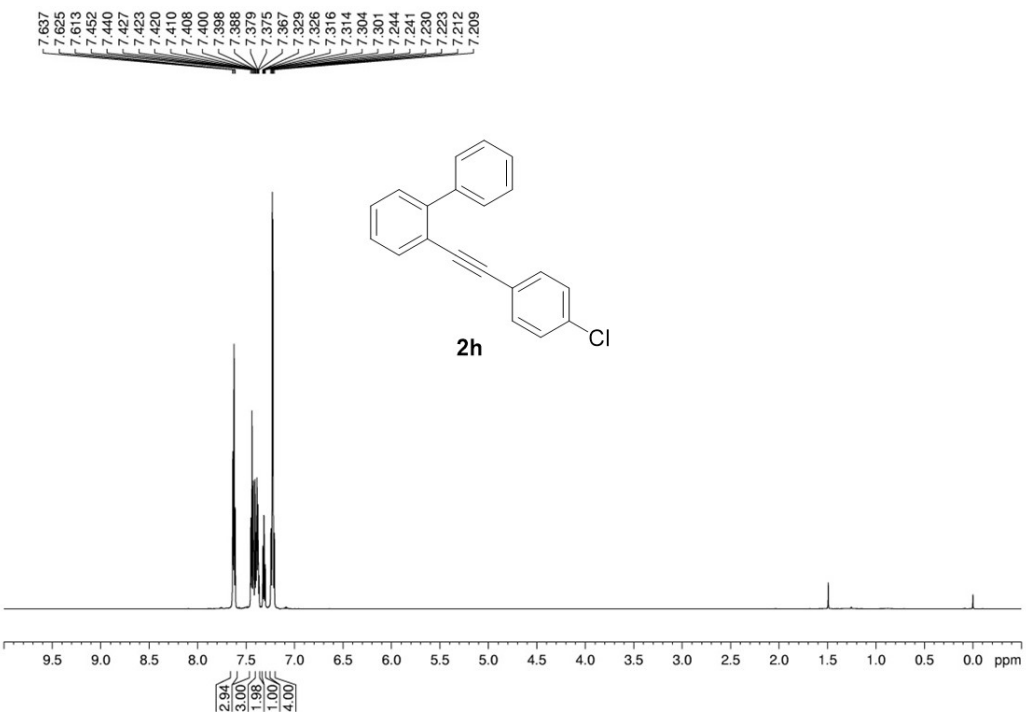

$^{1}$H NMR (600 MHz, $CDCl_3$), $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) and $^{19}F\{^{1}H\}$ NMR (565 MHz, $CDCl_3$) of **2g**

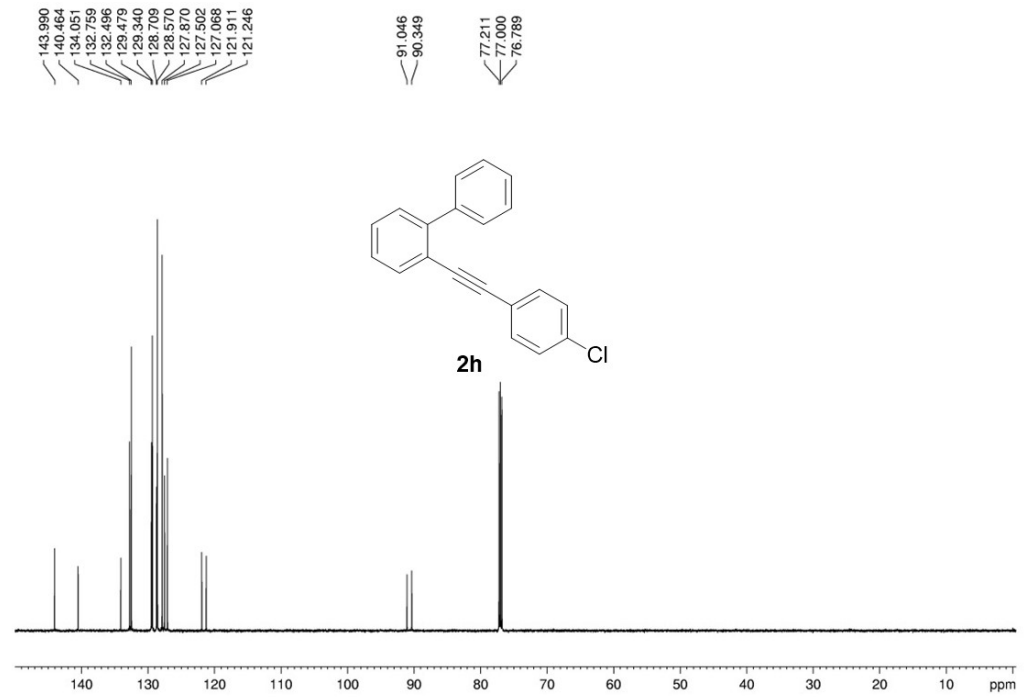



163.187
161.534
143.893
140.539
133.191
133.135
132.695
129.465
129.350
128.543
127.858
127.466
127.053
121.412
119.534
119.512
115.591
115.445
91.112
89.047
77.211
77.000
76.789
F
2g
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

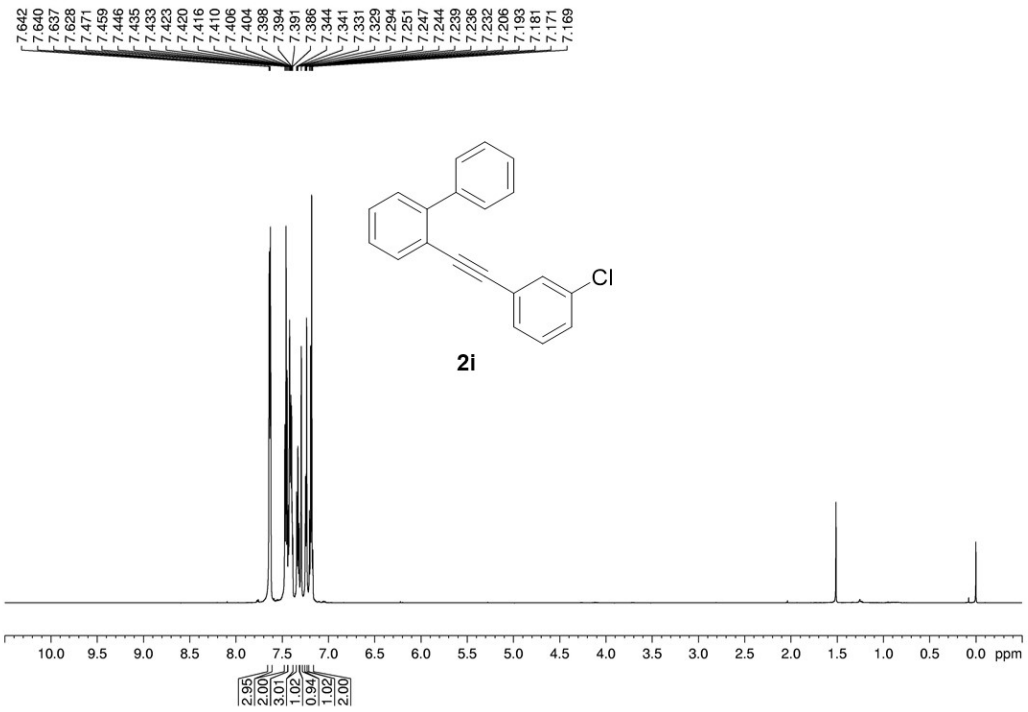

-111.013
F
2g
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
ppm

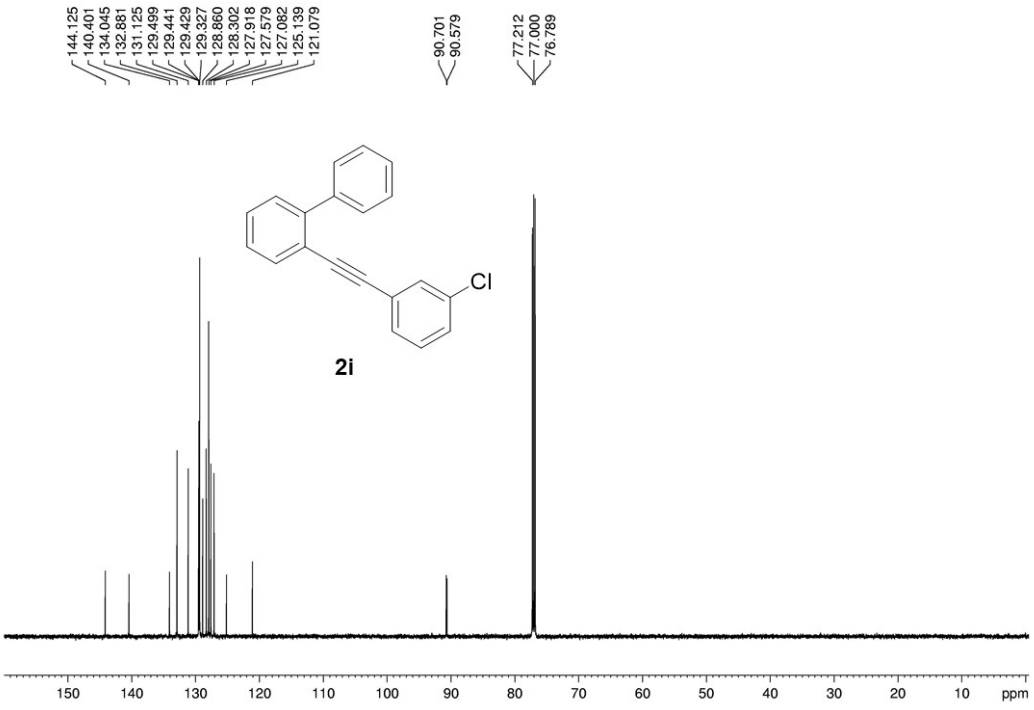
$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **2h**



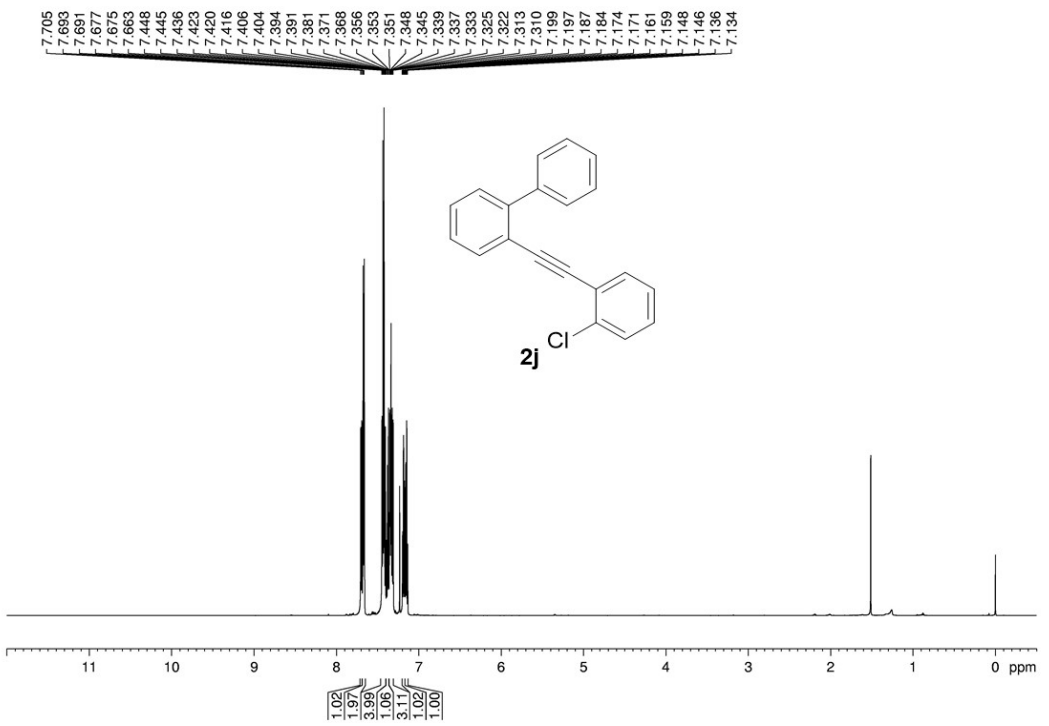



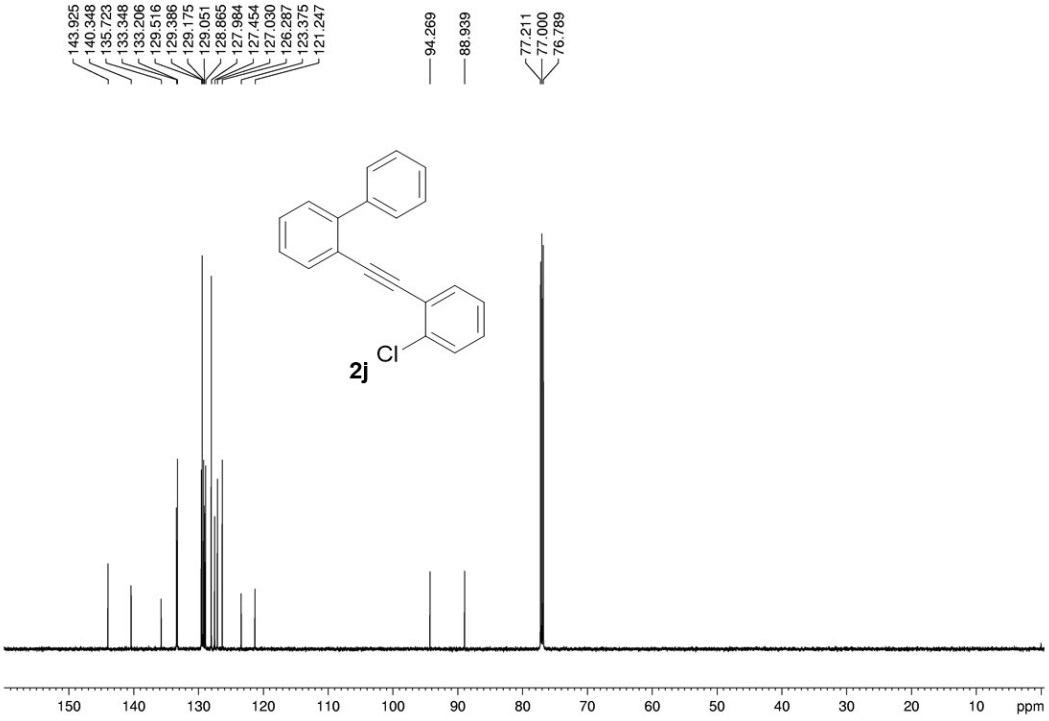
$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **2i**



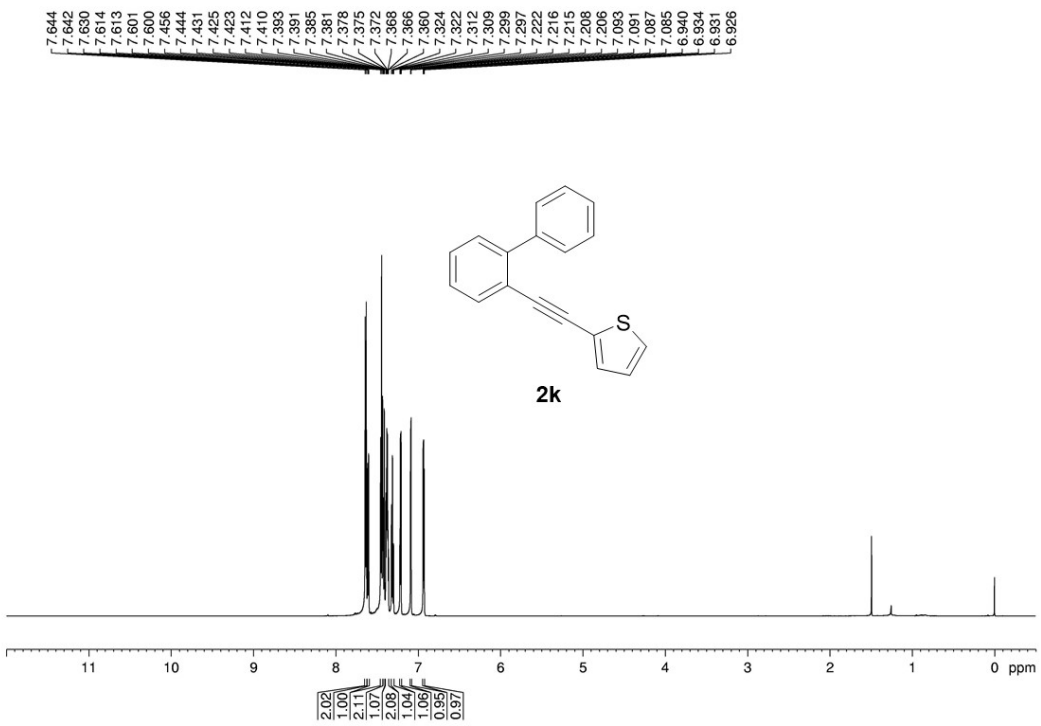



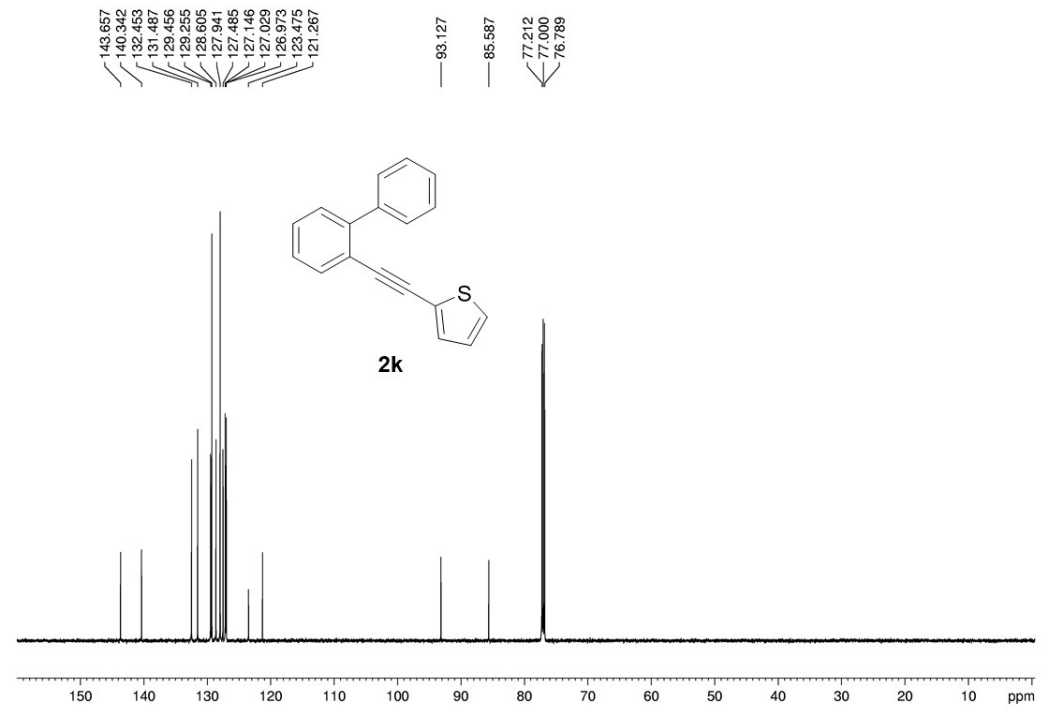
$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **2j**



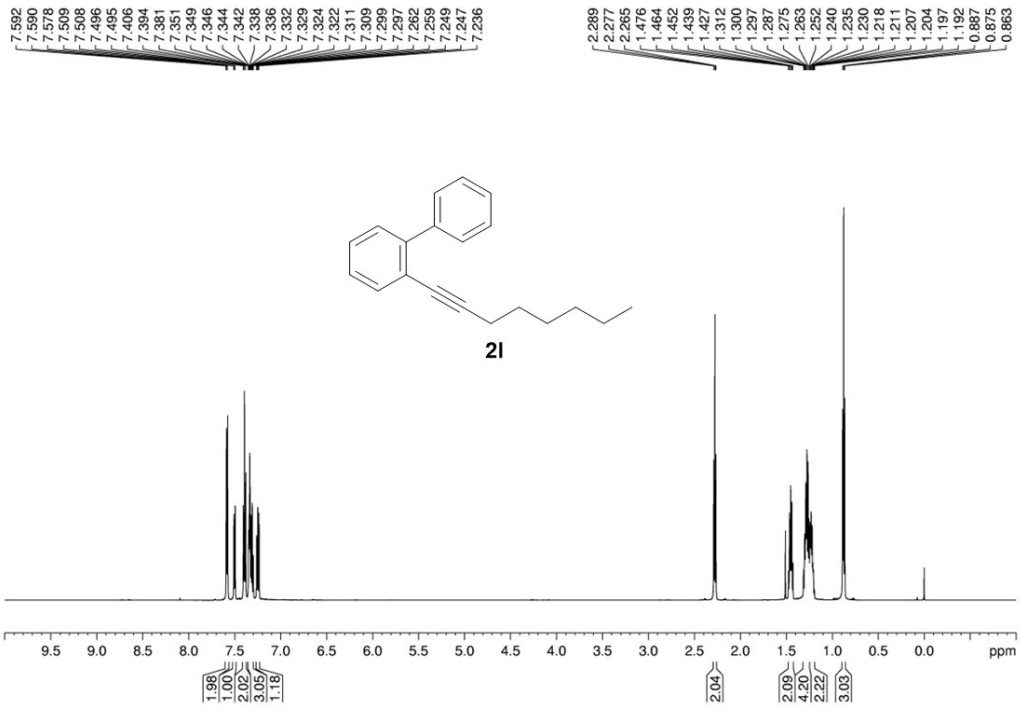



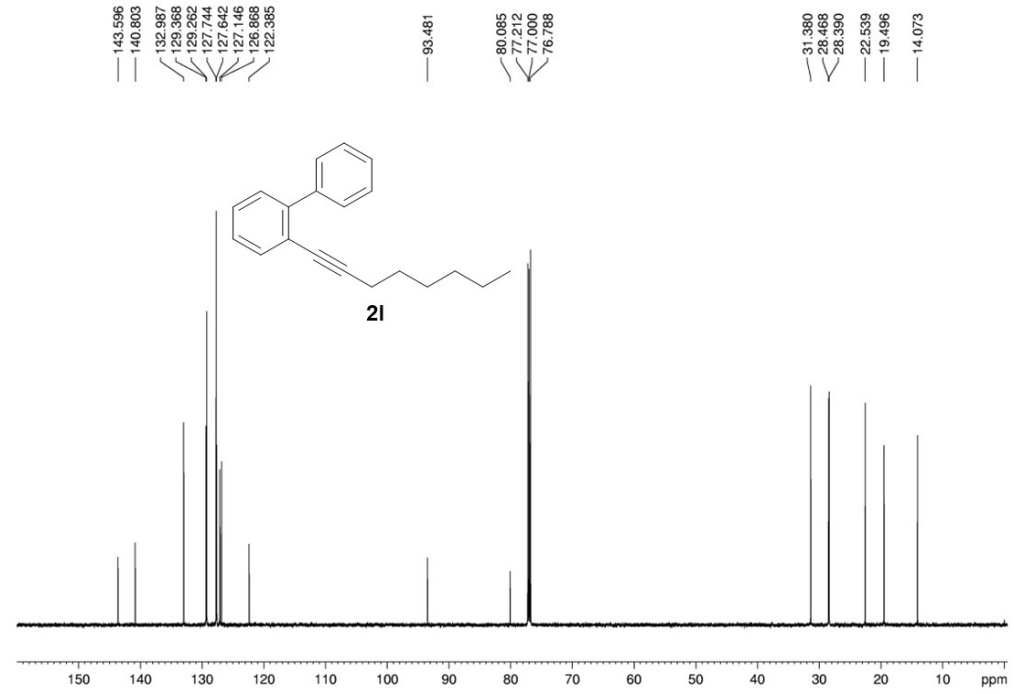
$^{1}H$ NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **2k**



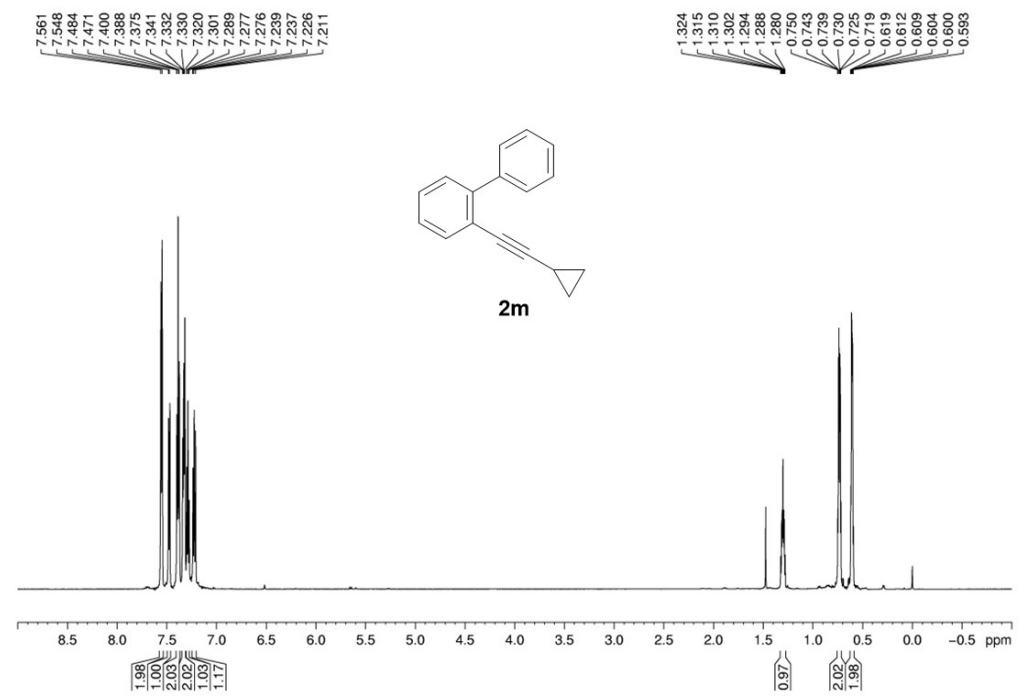



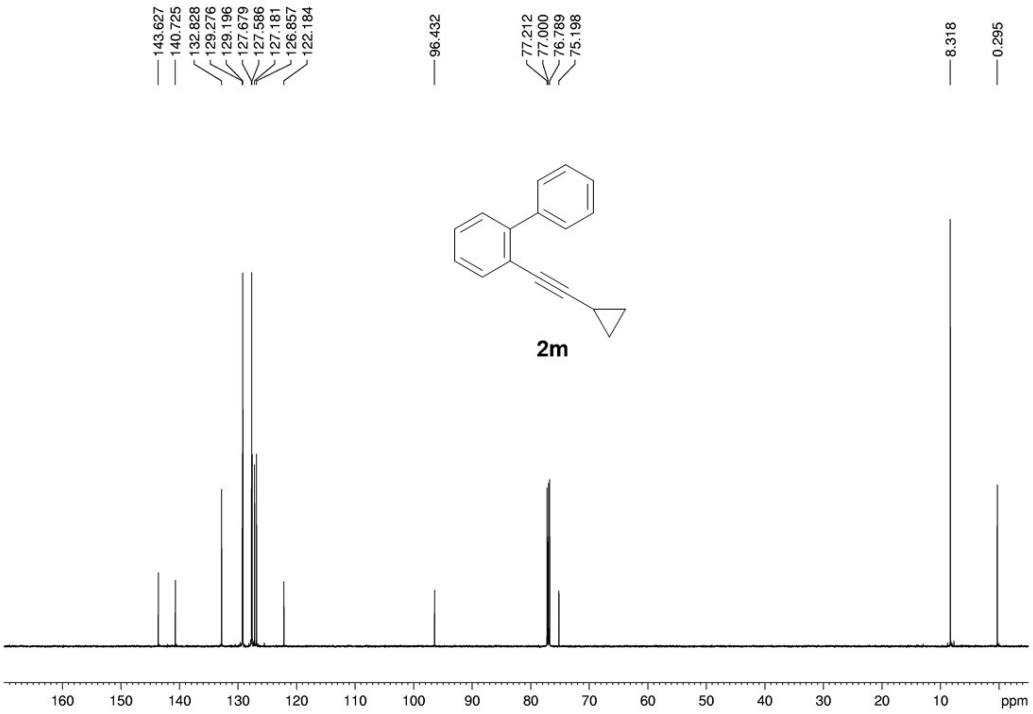
$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}$C{$^{1}$H} NMR (150 MHz, $CDCl_3$) of **2l**



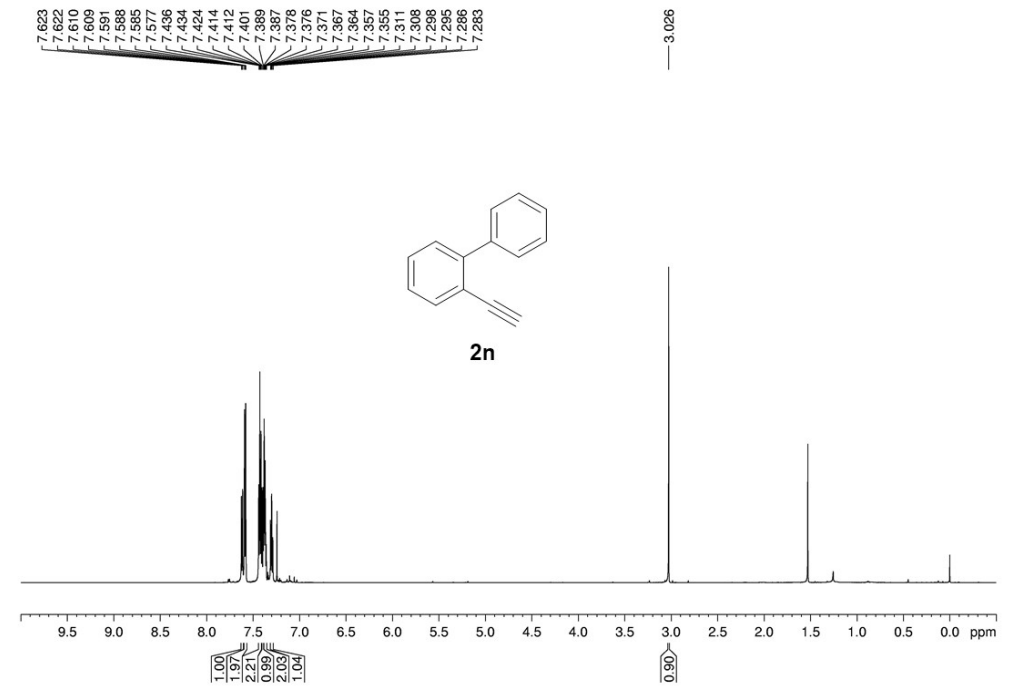



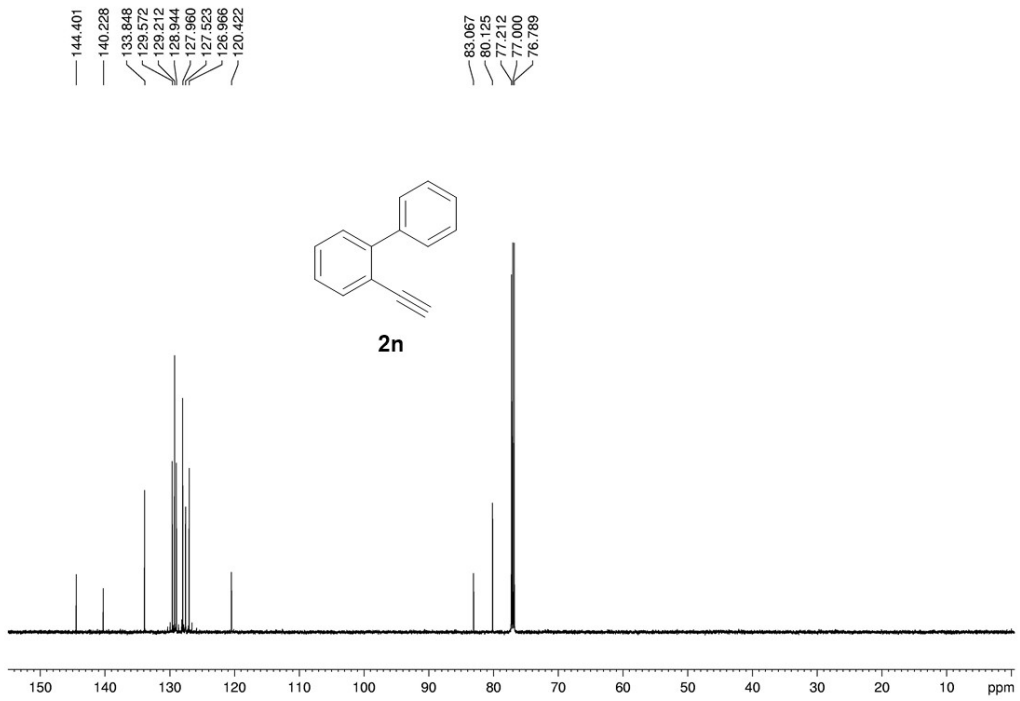
$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **2m**



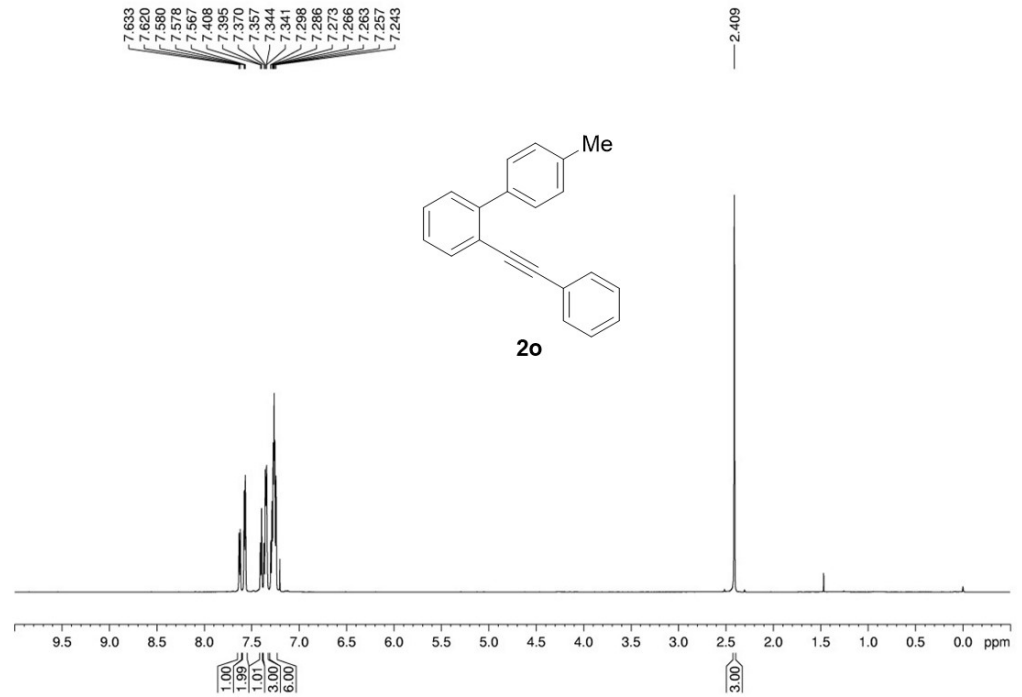



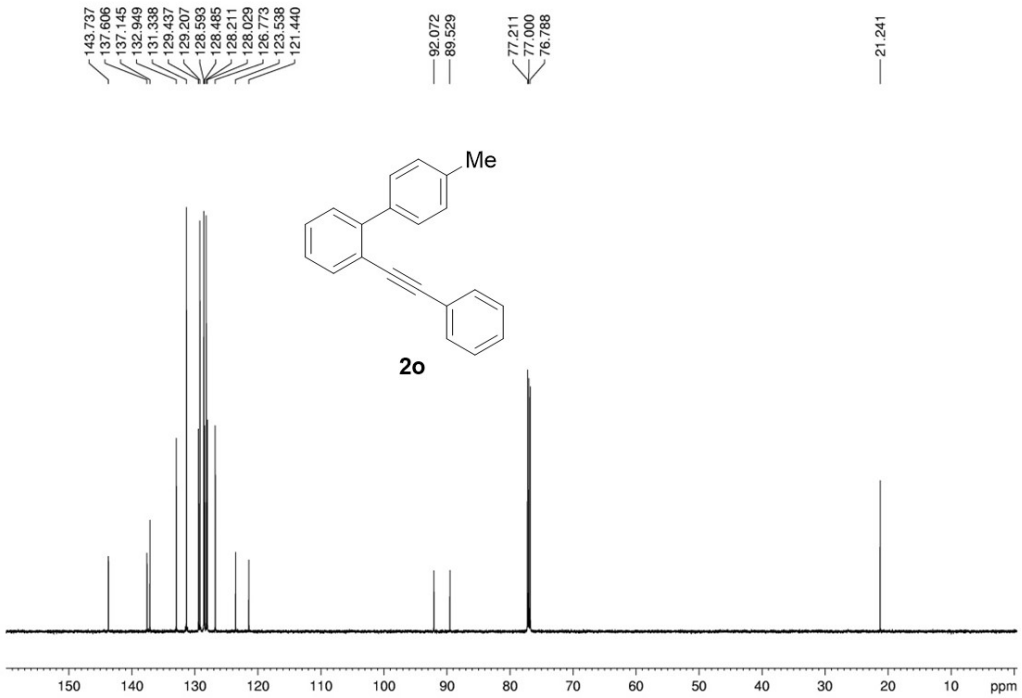
$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **2n**



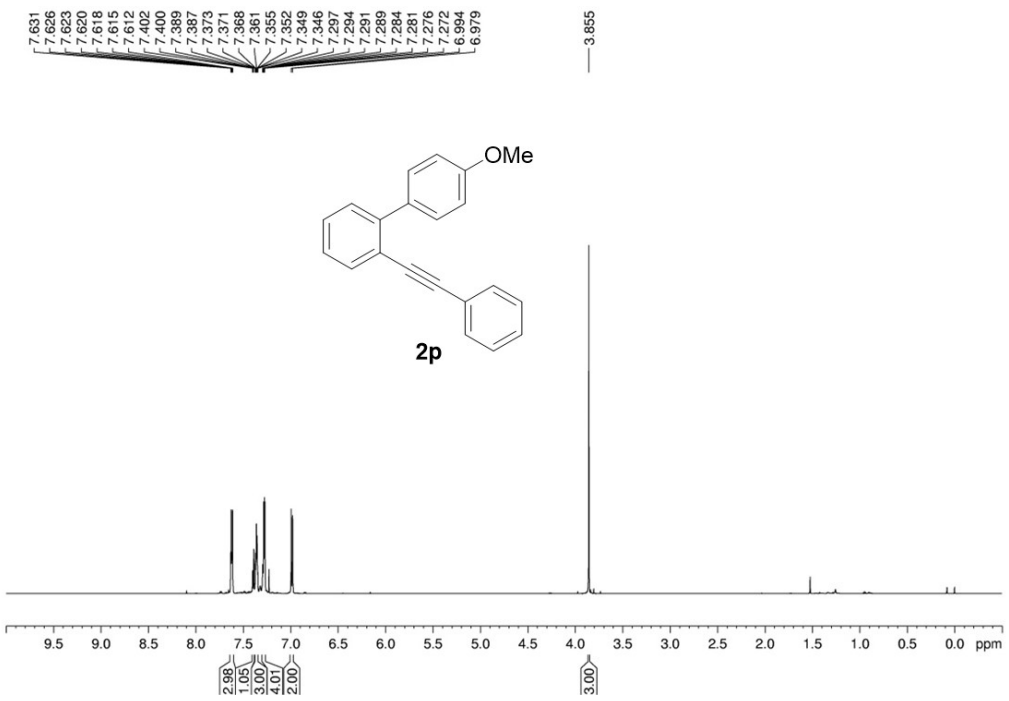



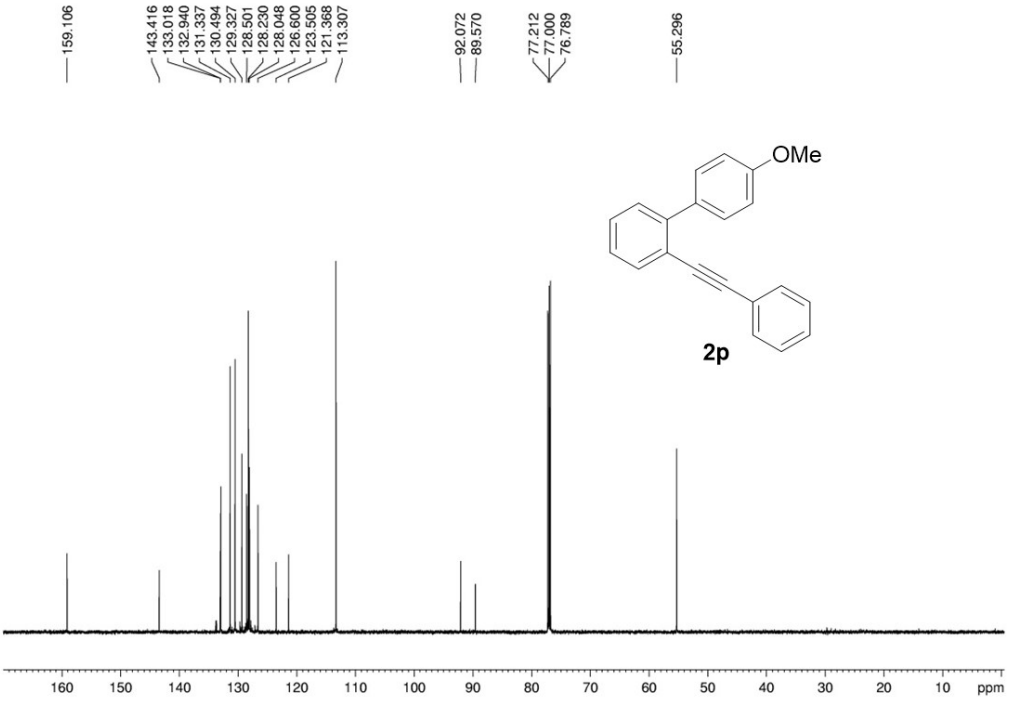
$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **2o**



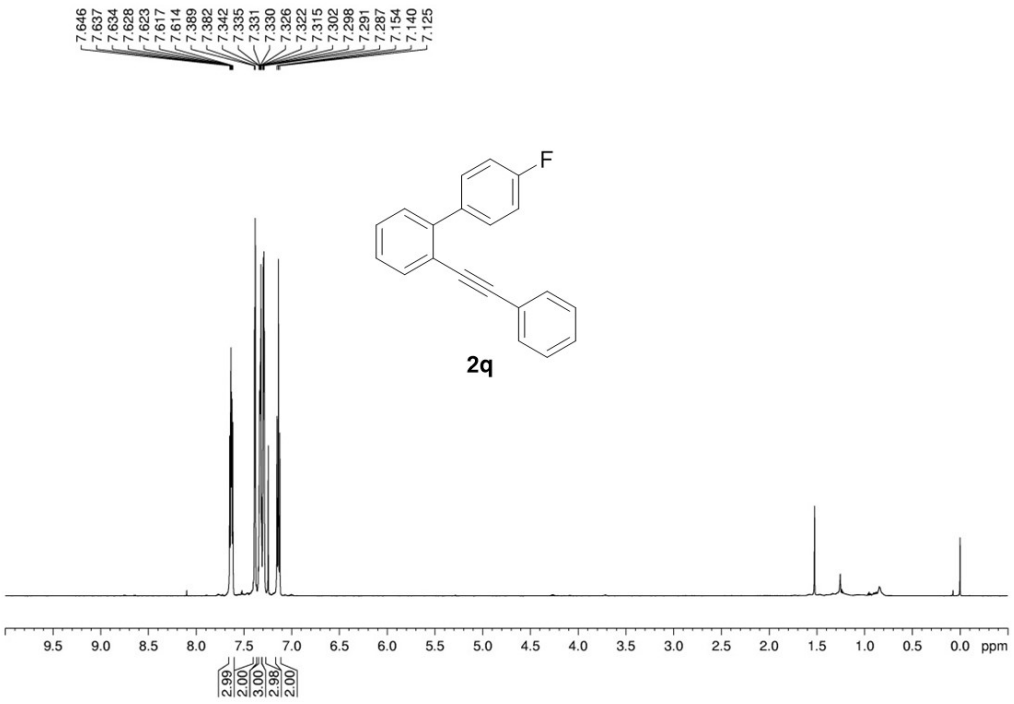



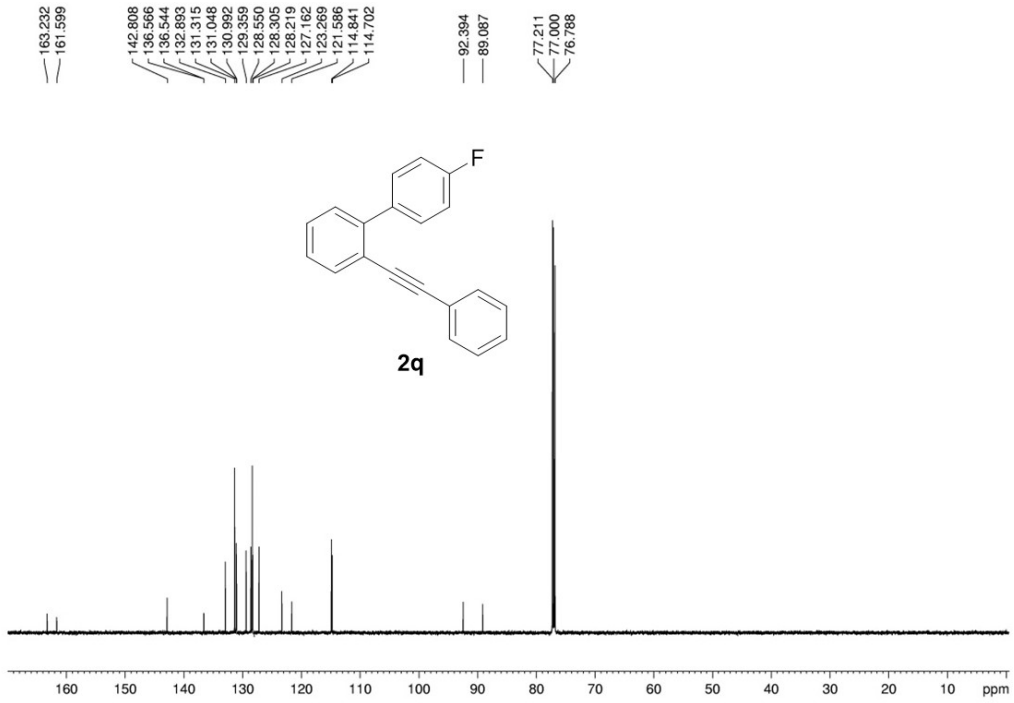
$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **2p**

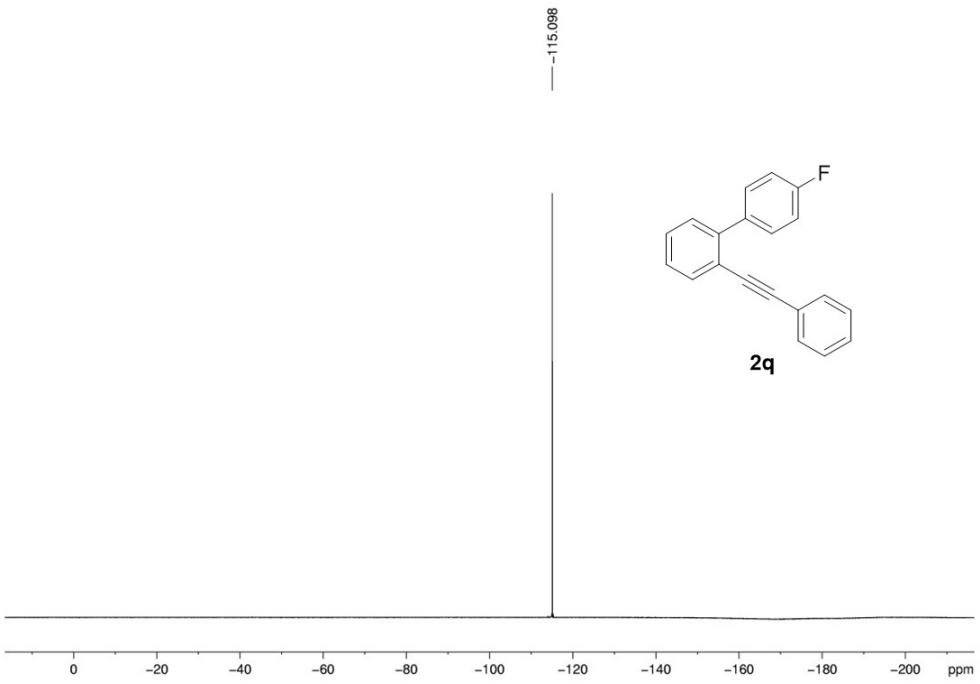


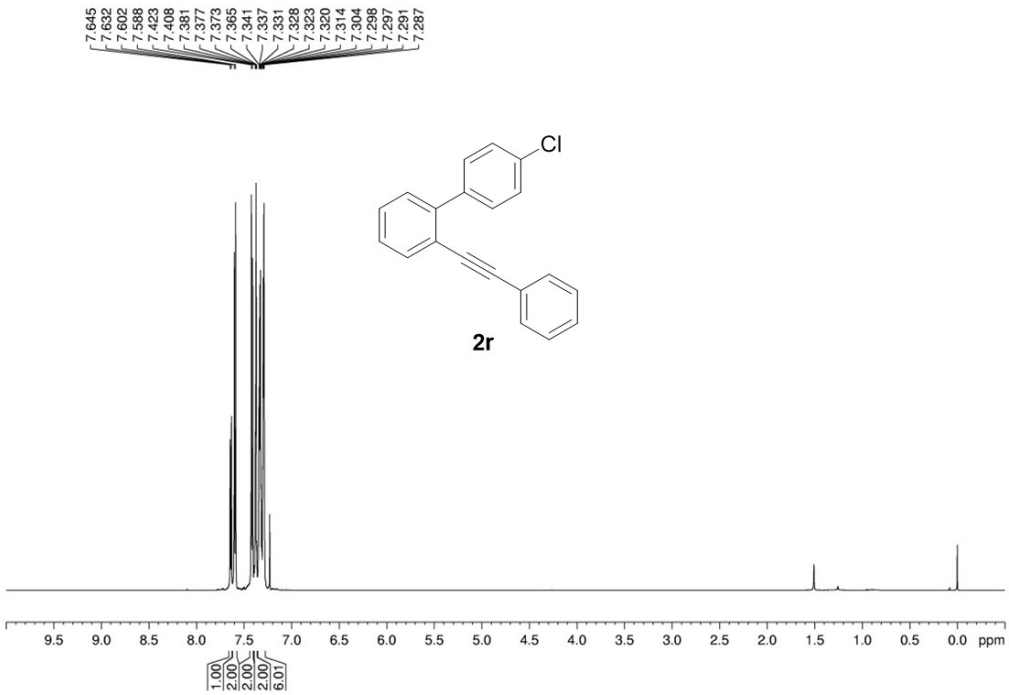
$^{1}$H NMR (600 MHz, $CDCl_3$), $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) and $^{19}F\{^{1}H\}$ NMR (565 MHz, $CDCl_3$) of **2q**

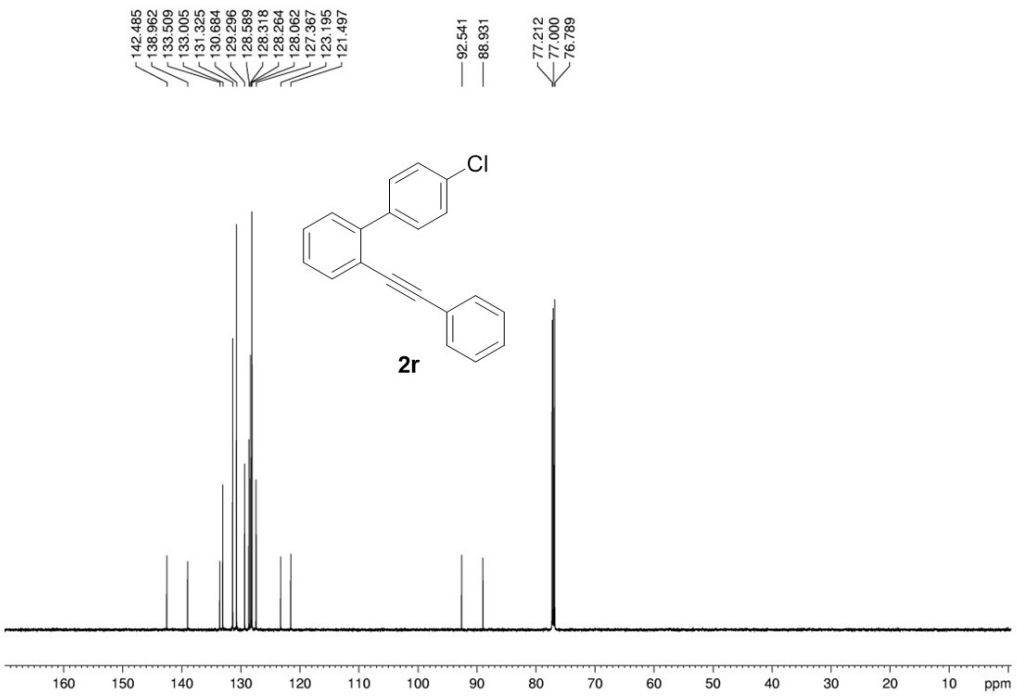


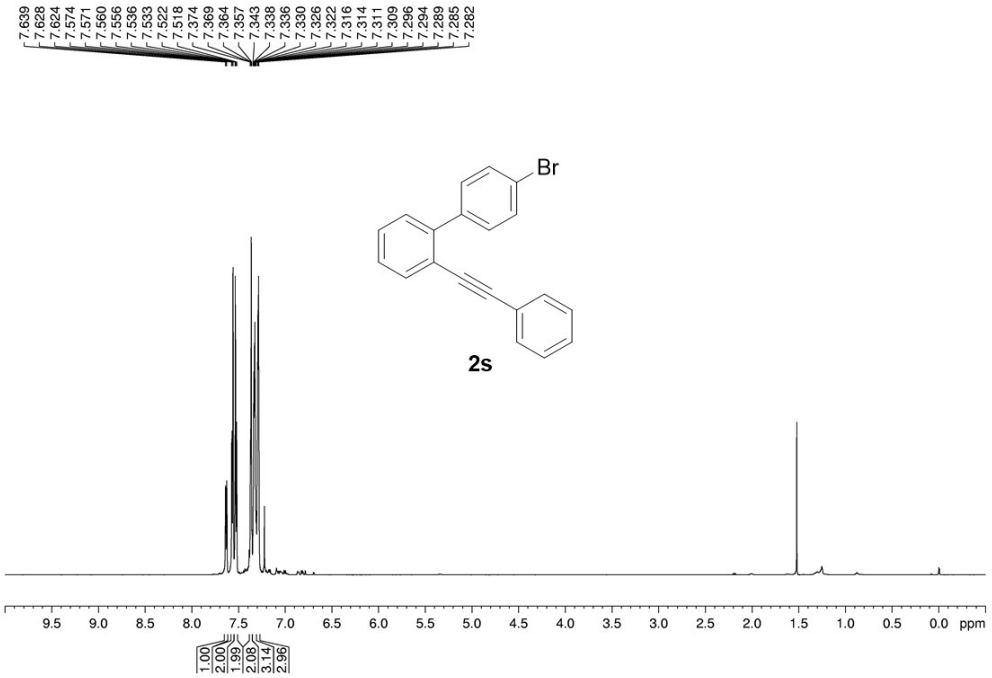


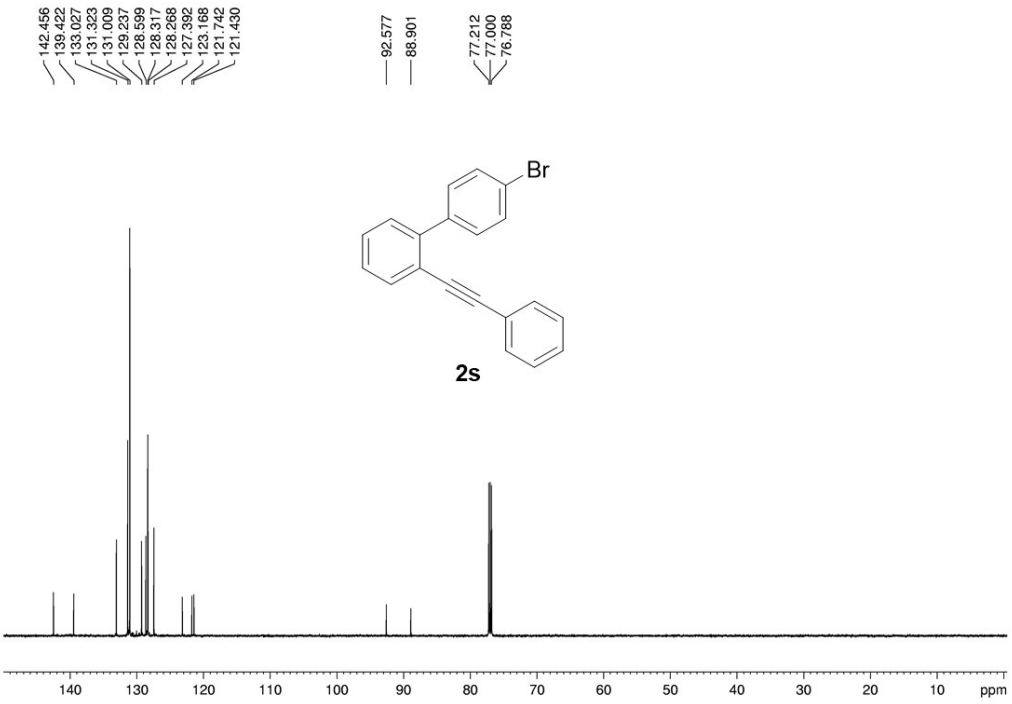

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **2r**



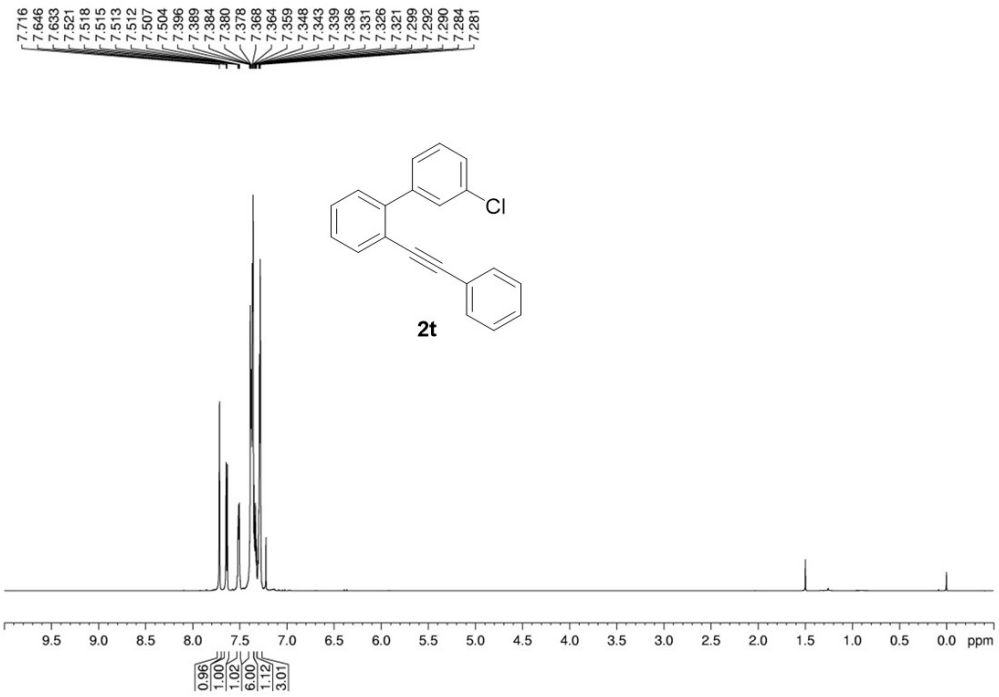


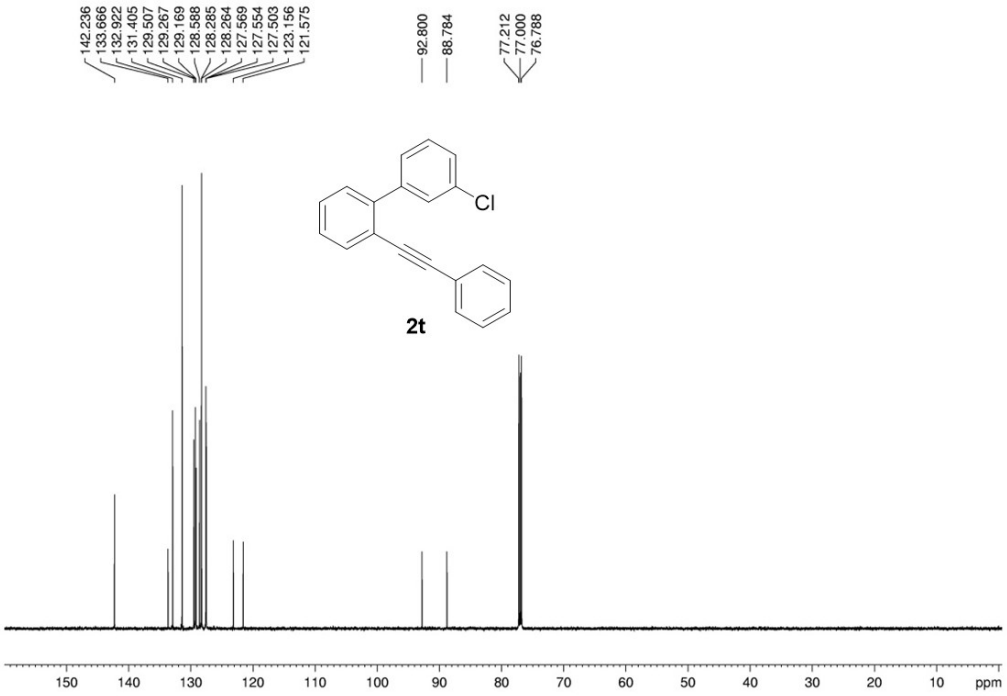

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **2s**



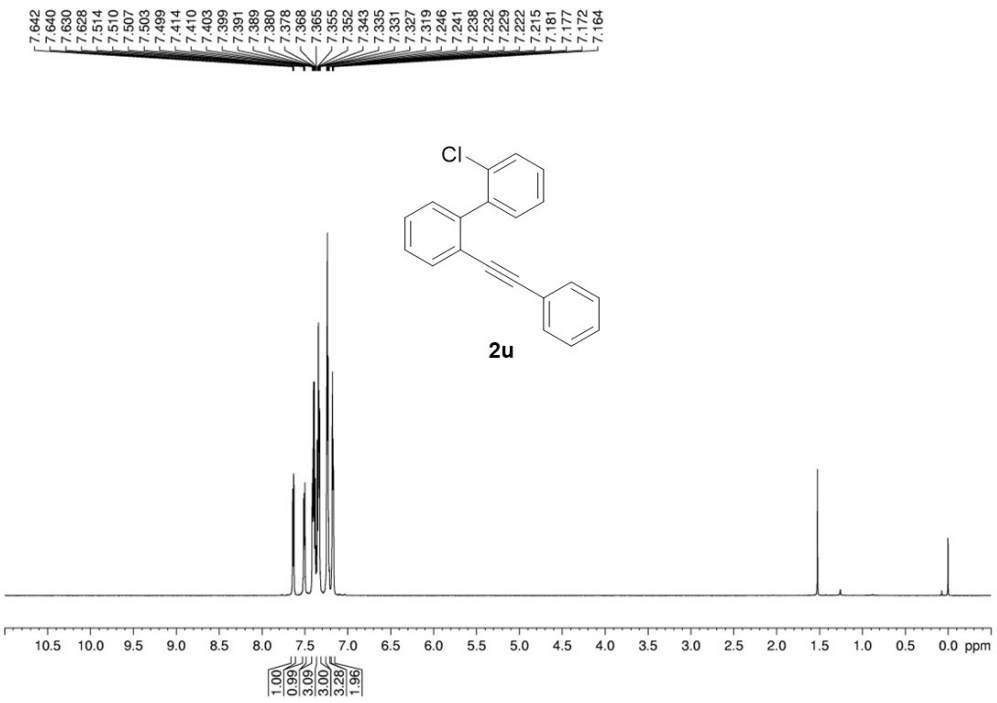


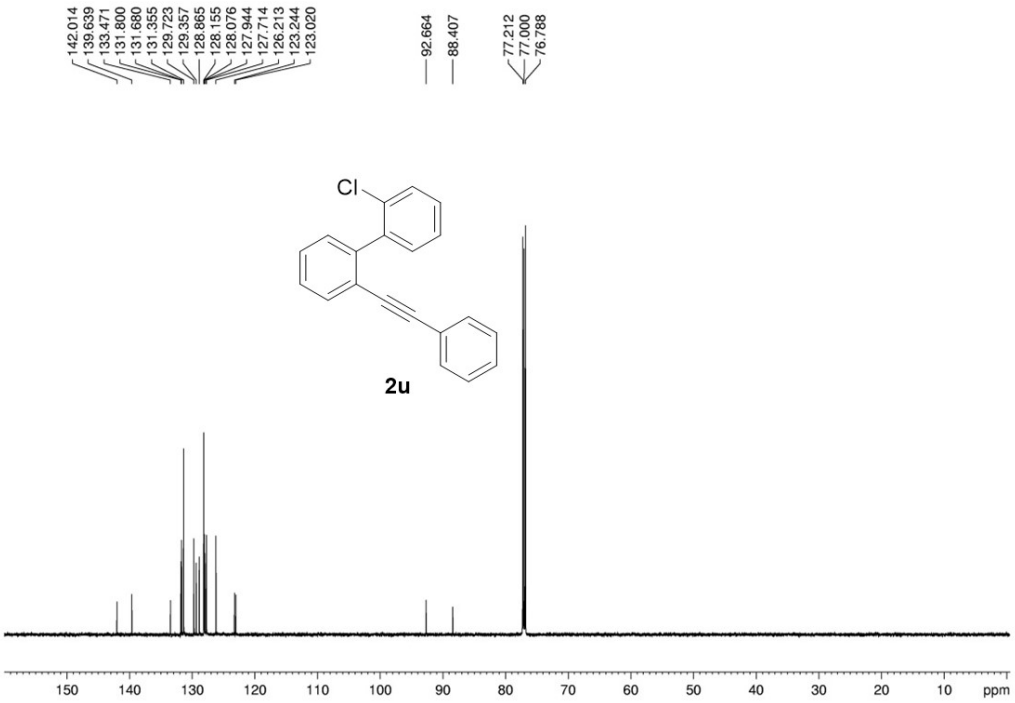

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **2t**



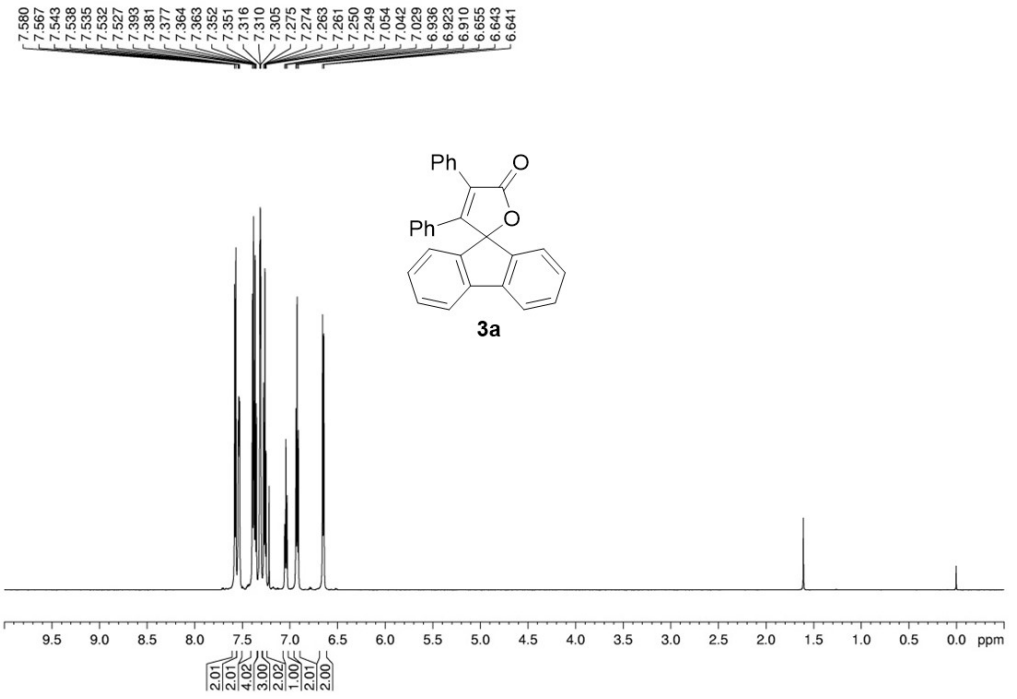


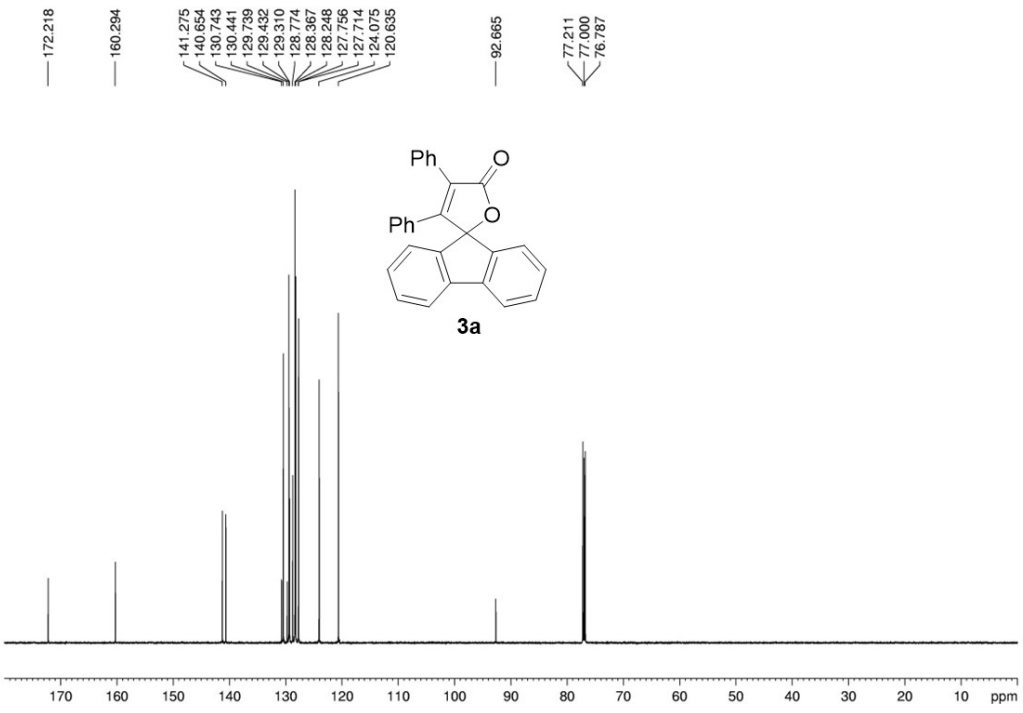

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **2u**



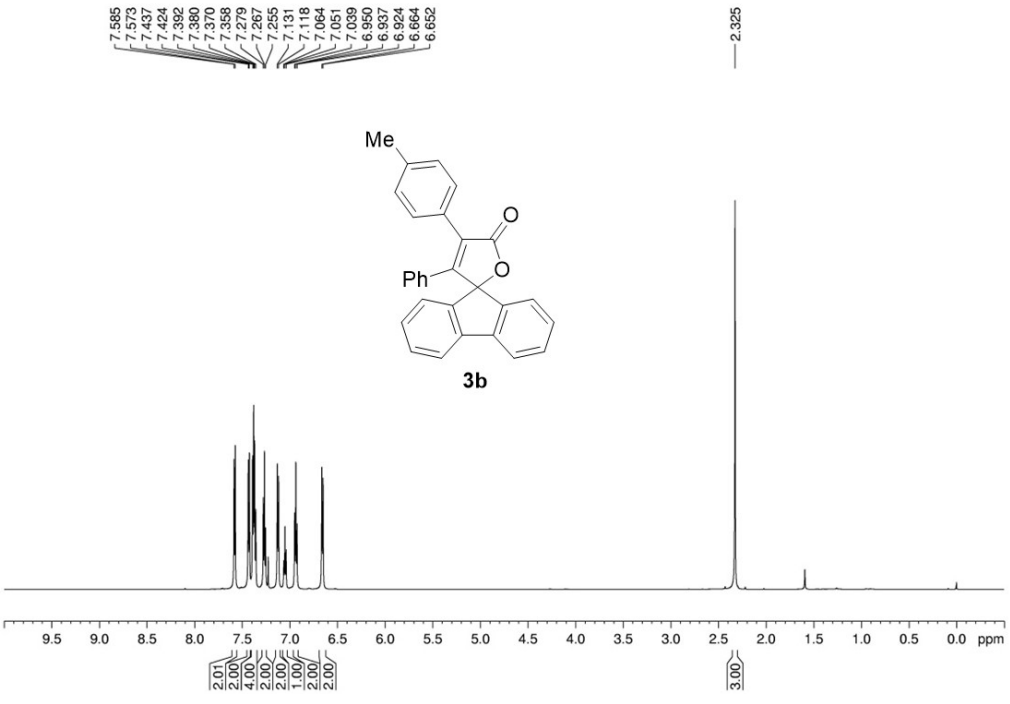


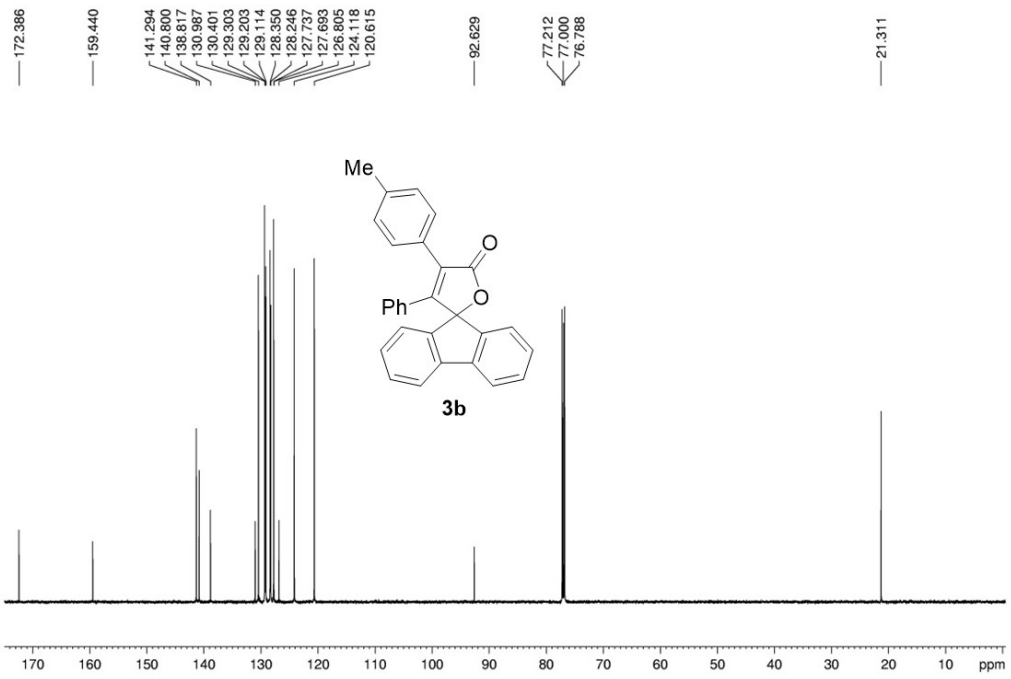

$^{1}H$ NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3a**



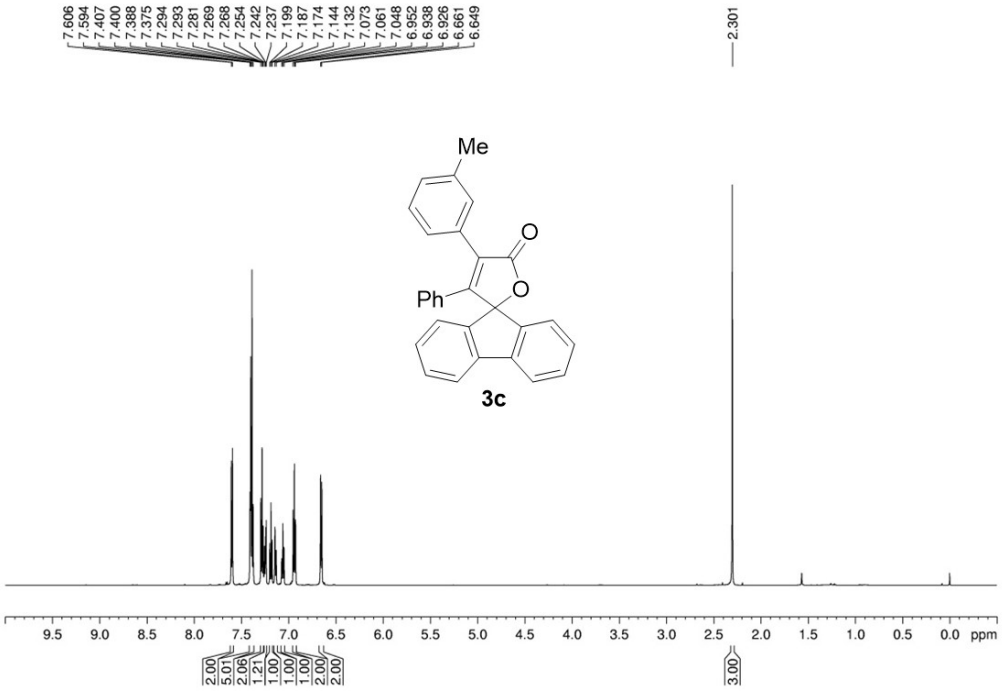


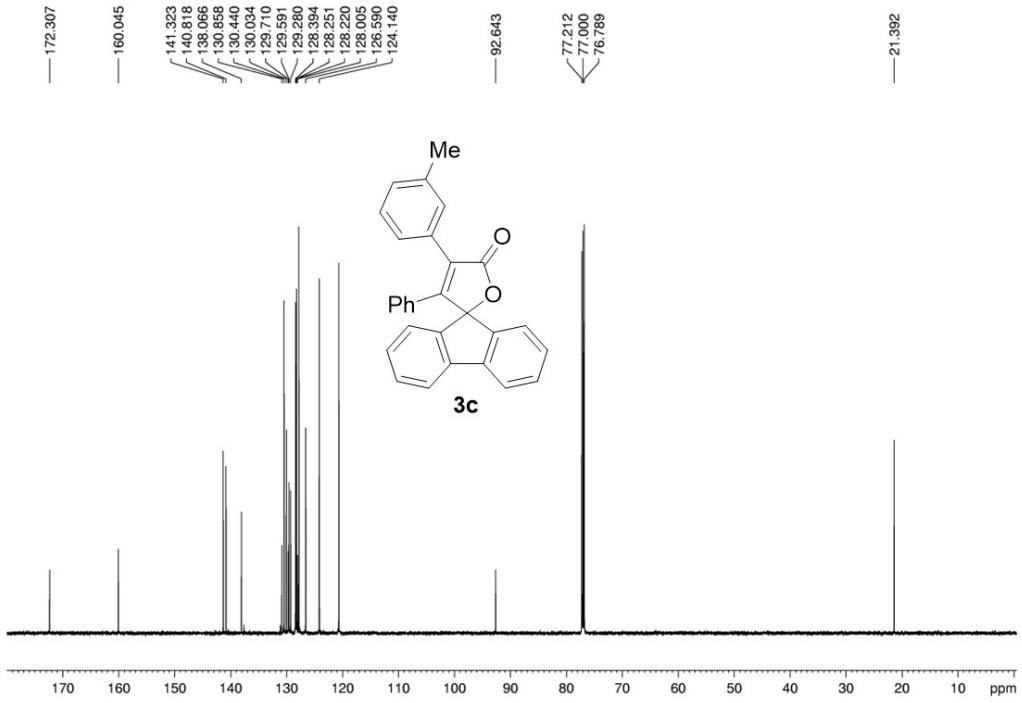

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3b**



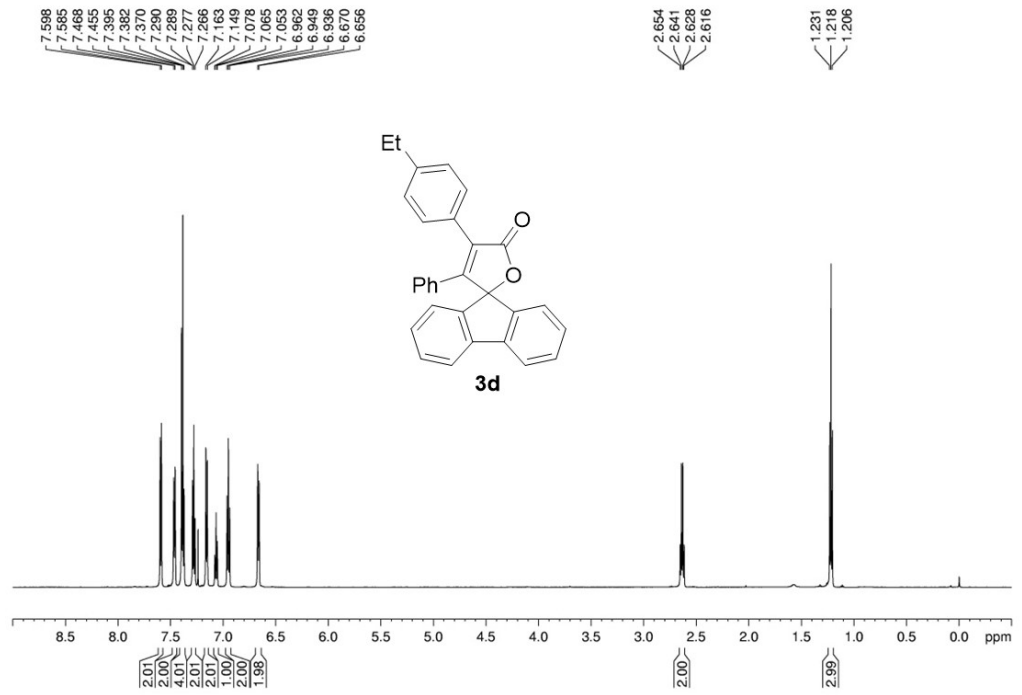


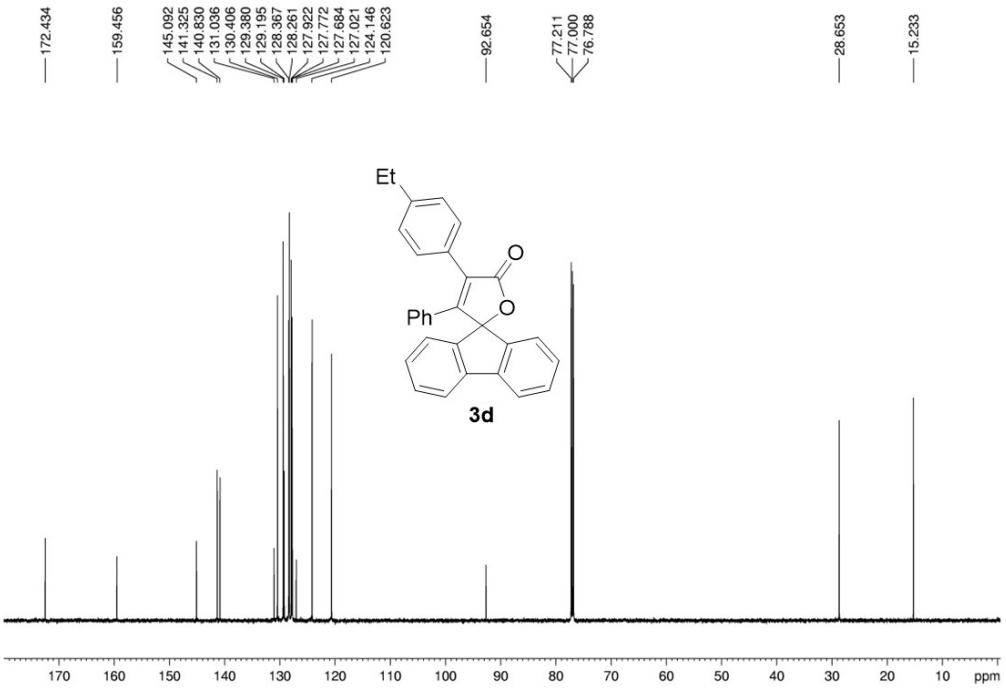

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3c**



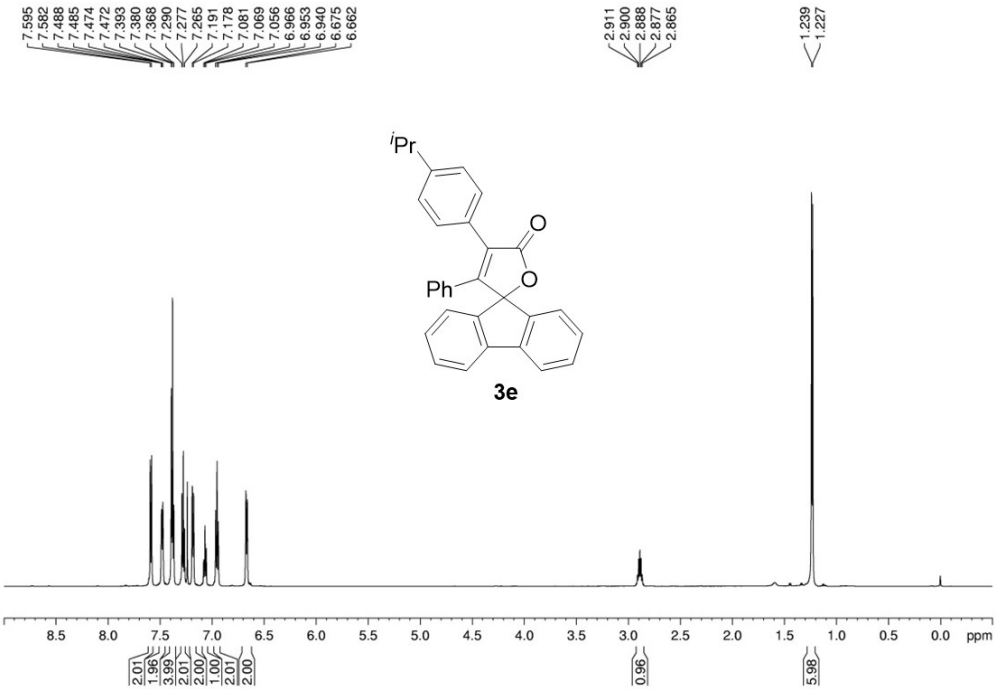


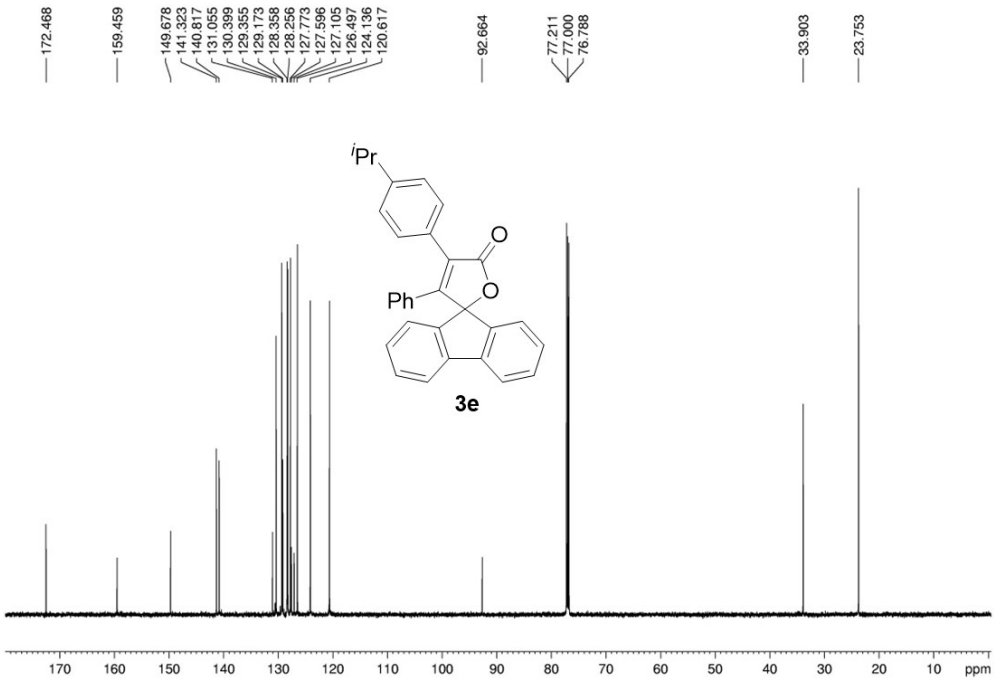

$^{1}H$ NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3d**



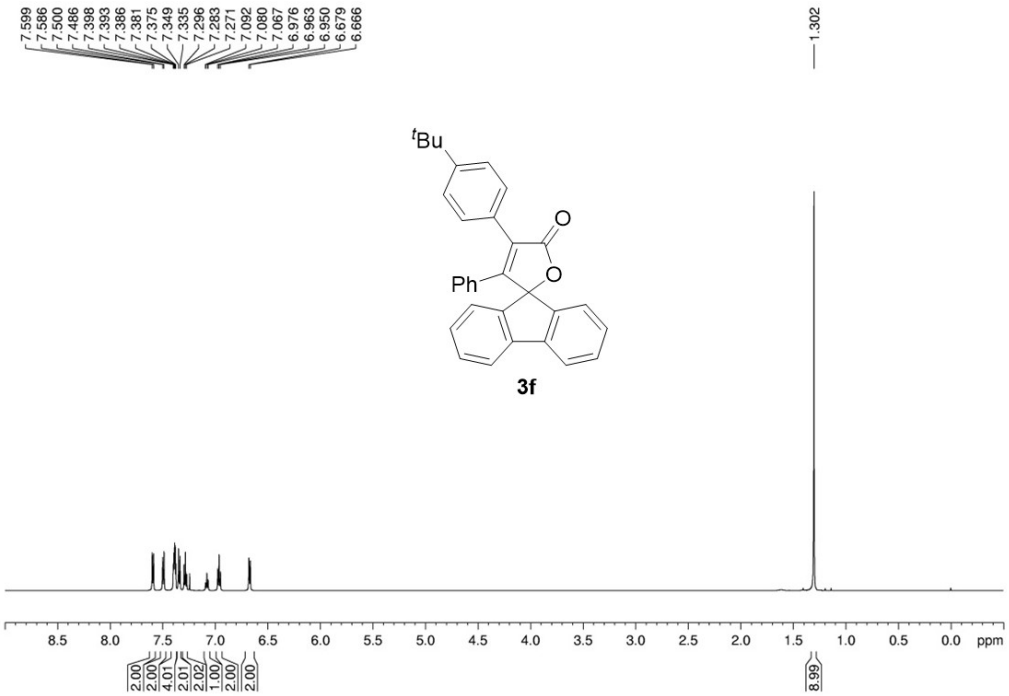


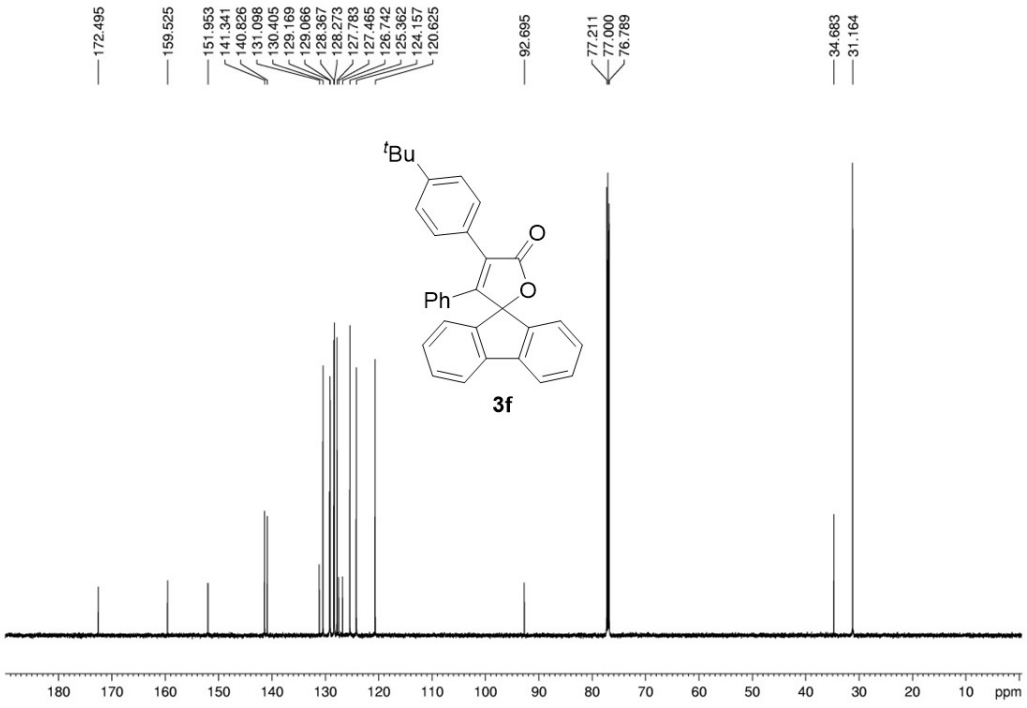

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3e**



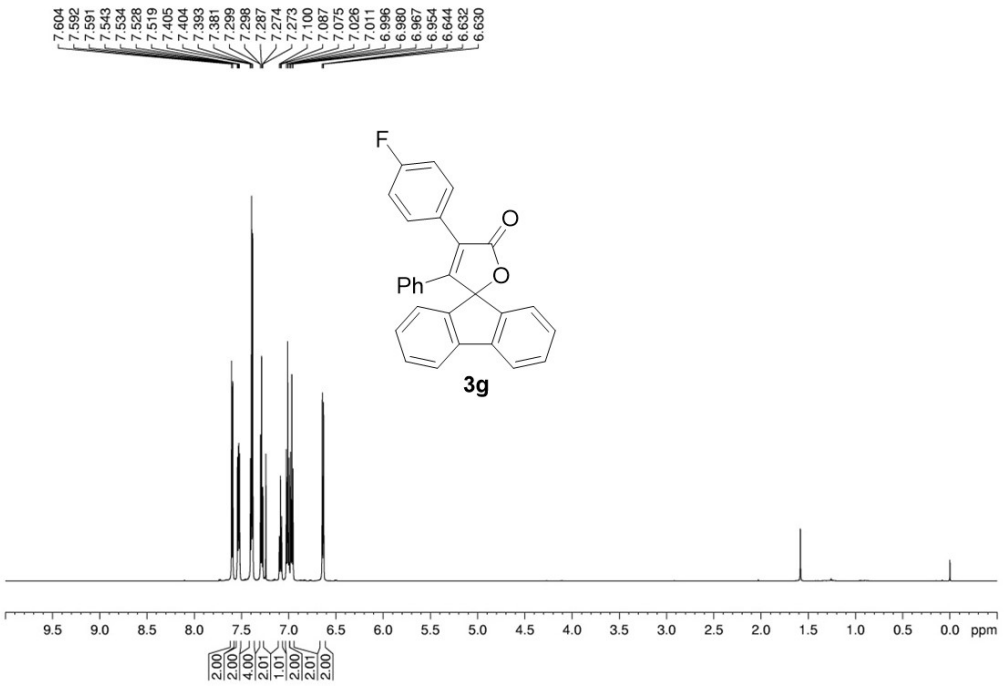


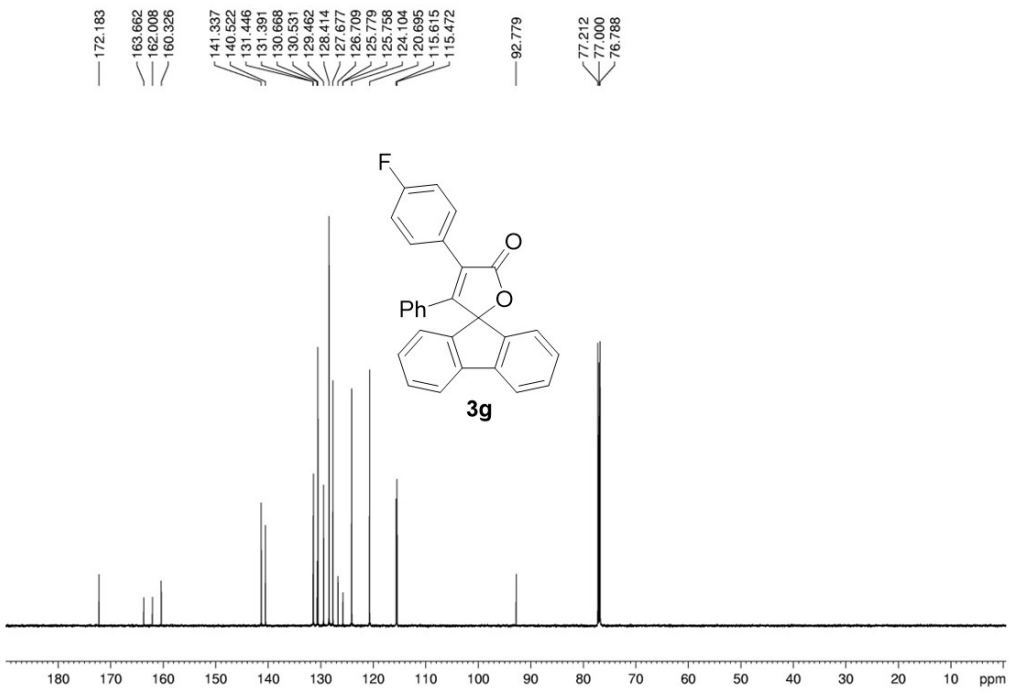

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3f**

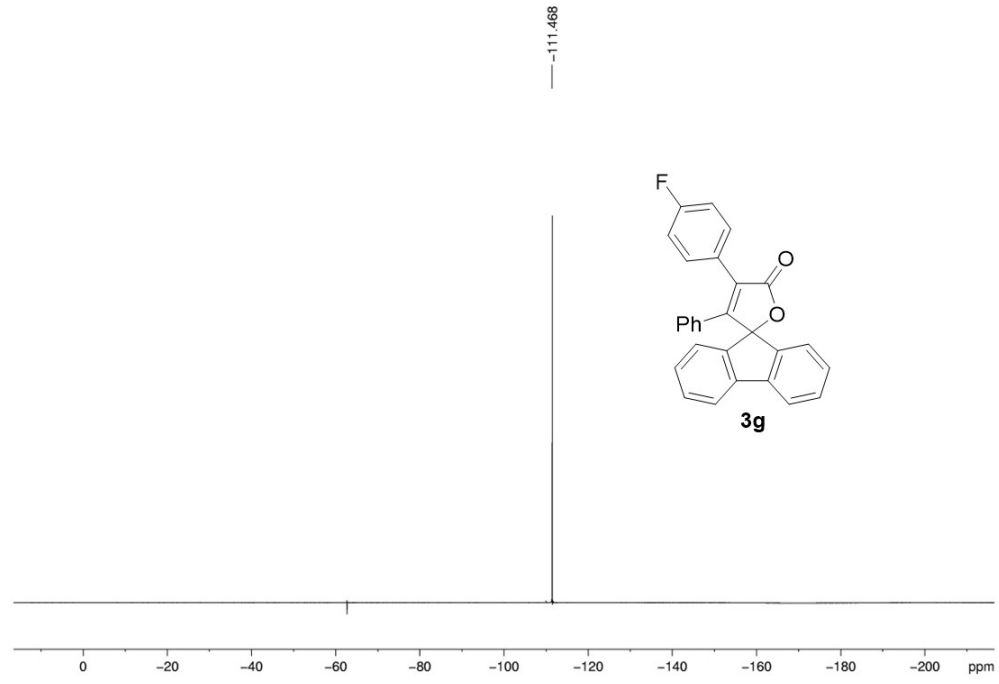




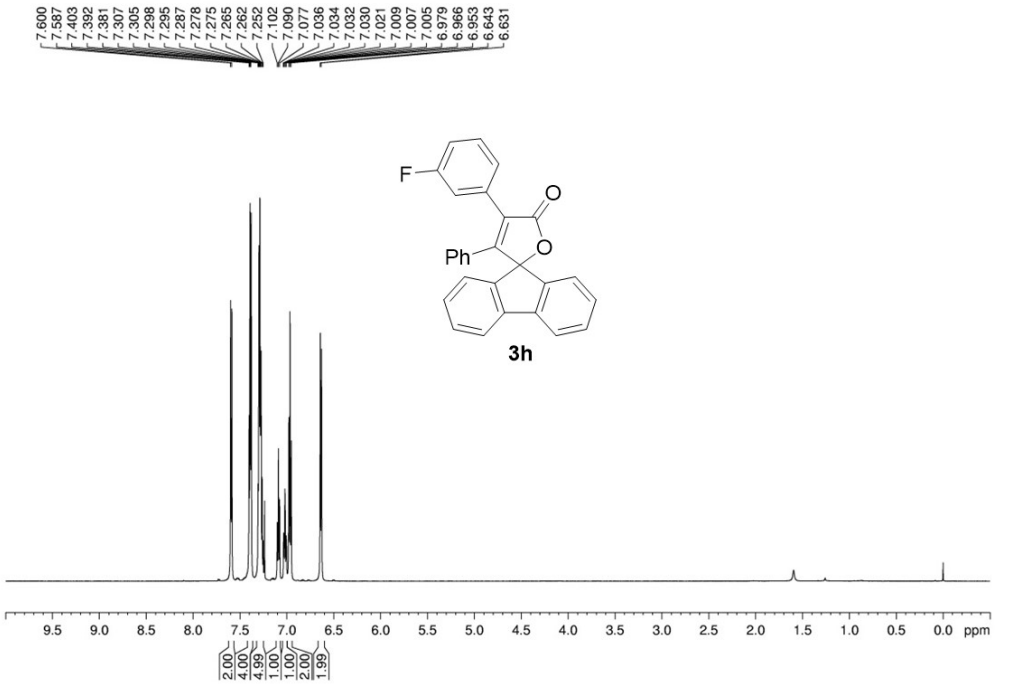

$^{1}H$ NMR (600 MHz, $CDCl_3$), $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) and $^{19}F\{^{1}H\}$ NMR (565 MHz, $CDCl_3$) of **3g**

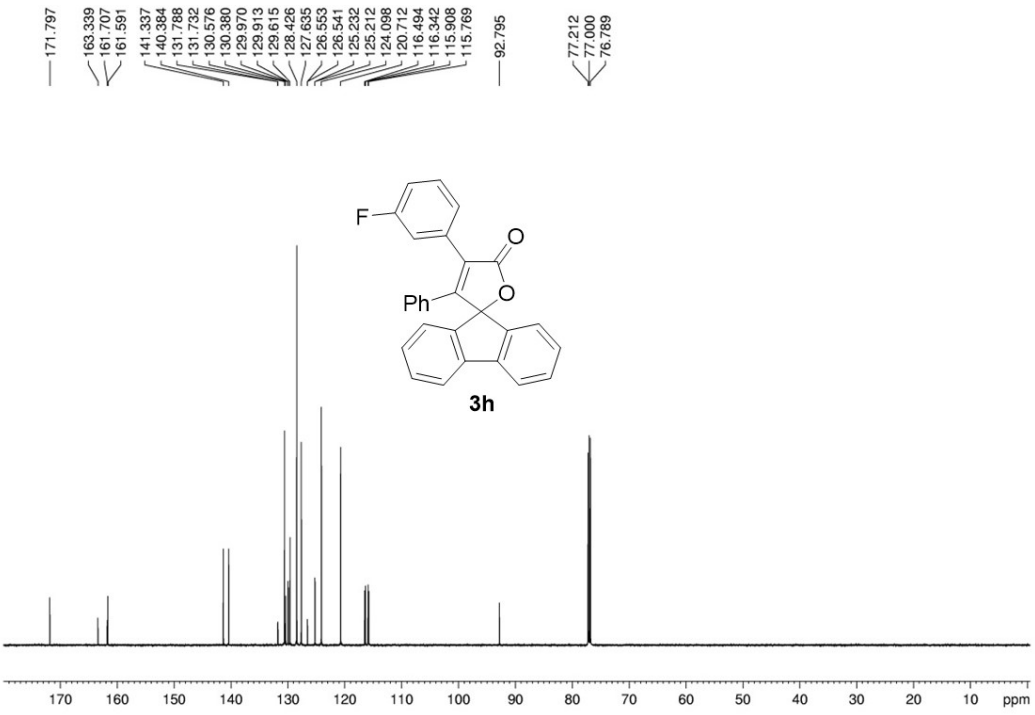


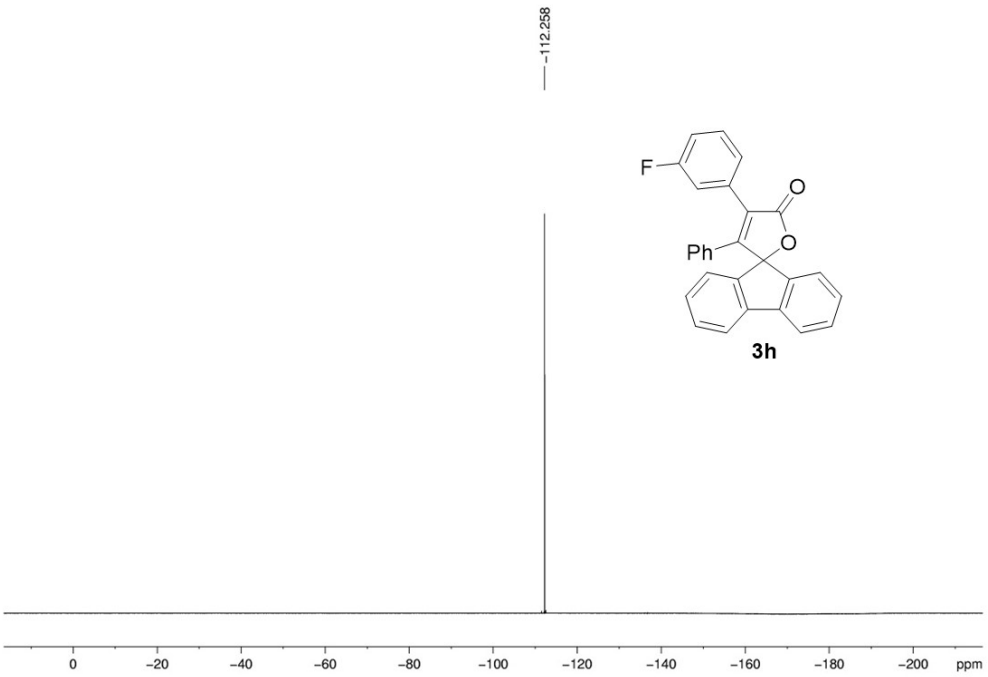

172.183
163.662
162.008
160.326
141.337
140.522
131.446
131.391
130.668
130.531
129.462
128.414
127.677
126.709
125.779
125.758
124.104
120.695
115.615
115.472
92.779
77.212
77.000
76.788
F
O
O
Ph
3g
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


-111.468
F
O
O
Ph
3g
0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 ppm

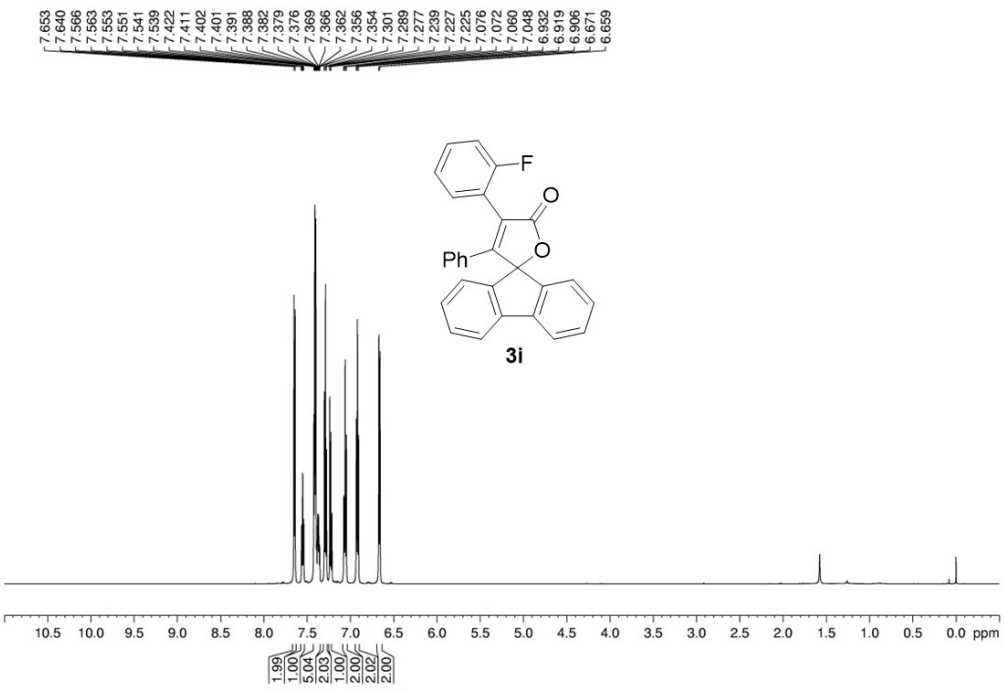
$^{1}H$ NMR (600 MHz, $CDCl_3$), $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) and $^{19}F\{^{1}H\}$ NMR (565 MHz, $CDCl_3$) of **3h**

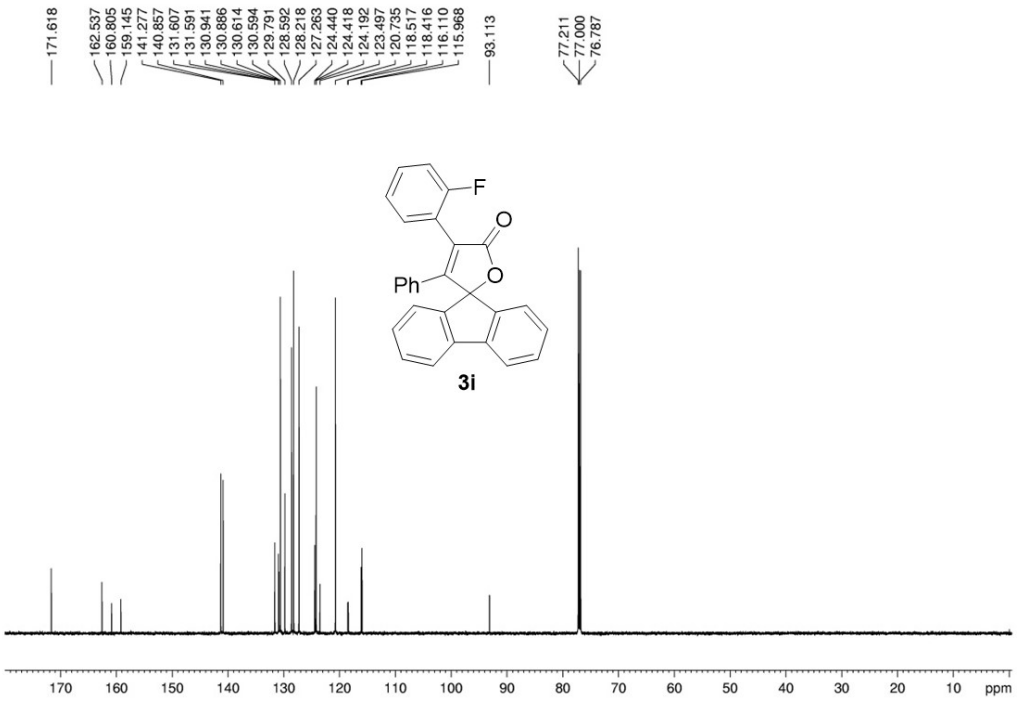


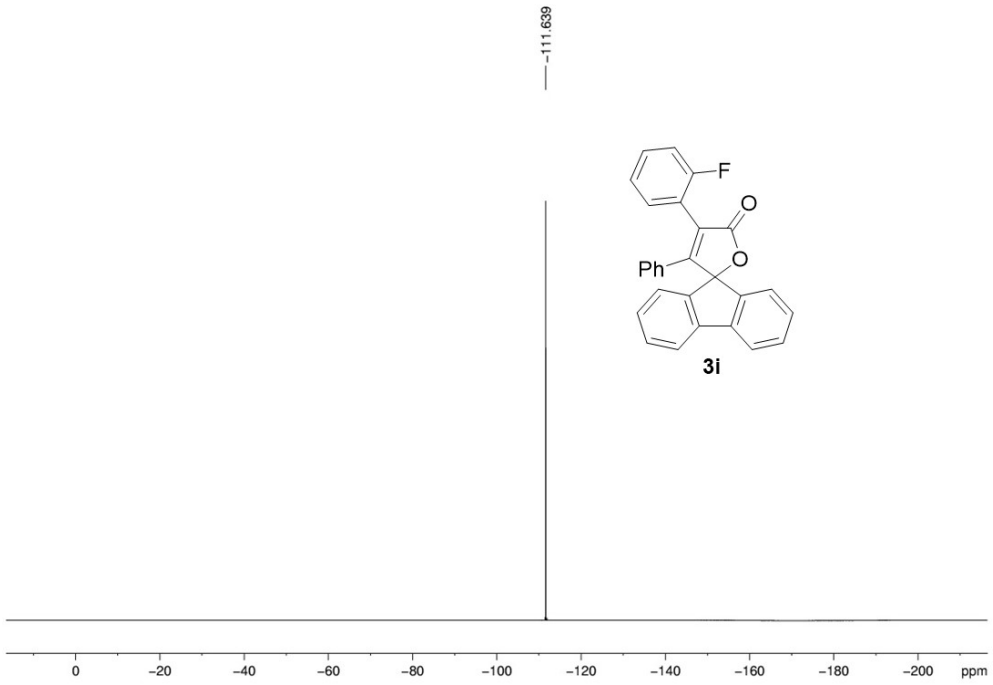





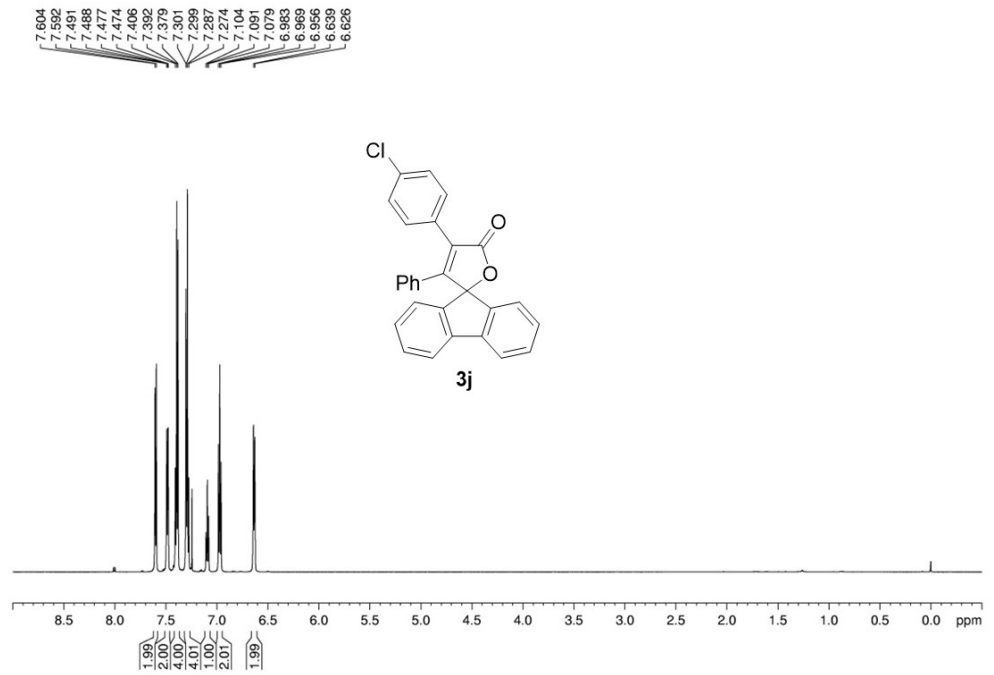

$^{1}$H NMR (600 MHz, $CDCl_3$), $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) and $^{19}F\{^{1}H\}$ NMR (565 MHz, $CDCl_3$) of **3i**

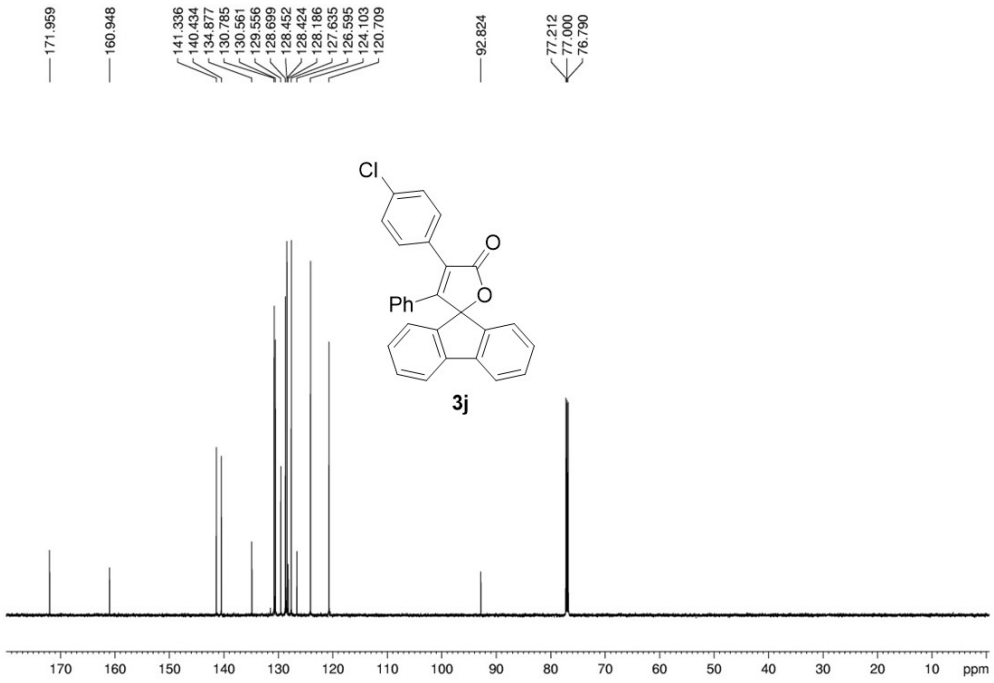



171.618
162.537
160.805
159.145
141.277
140.857
131.607
131.591
130.941
130.886
130.614
130.594
129.791
128.592
128.218
127.263
124.440
124.418
124.192
123.497
120.735
118.517
118.416
116.110
115.968
93.113
77.211
77.000
76.787
F
O
Ph
O
3i
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

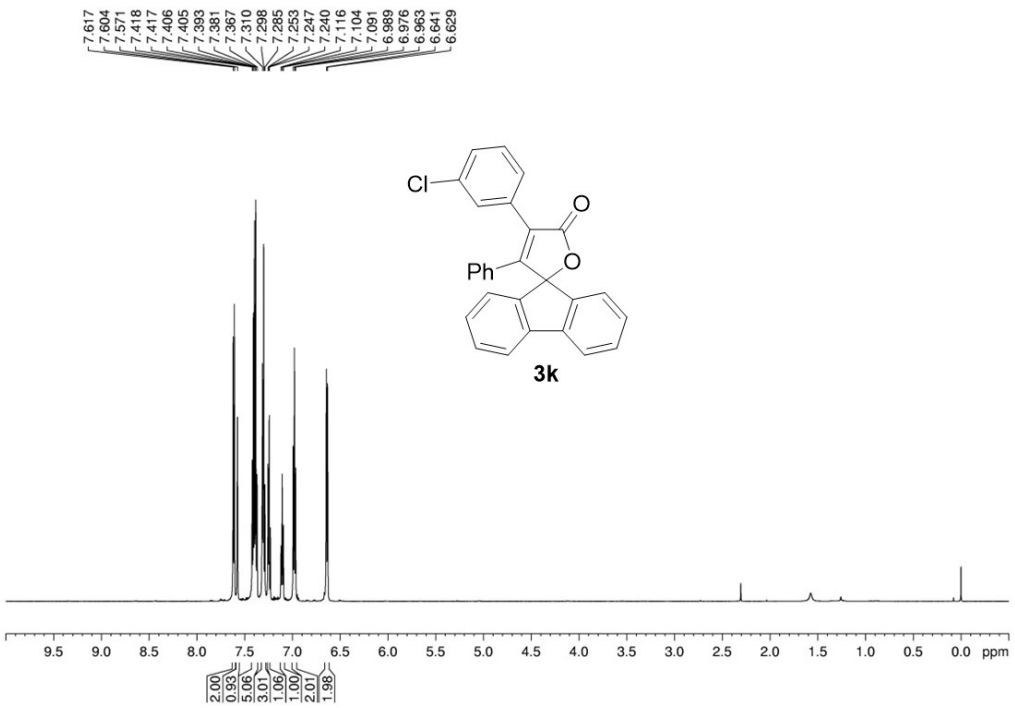

-111.639
F
O
Ph
O
3i
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
ppm

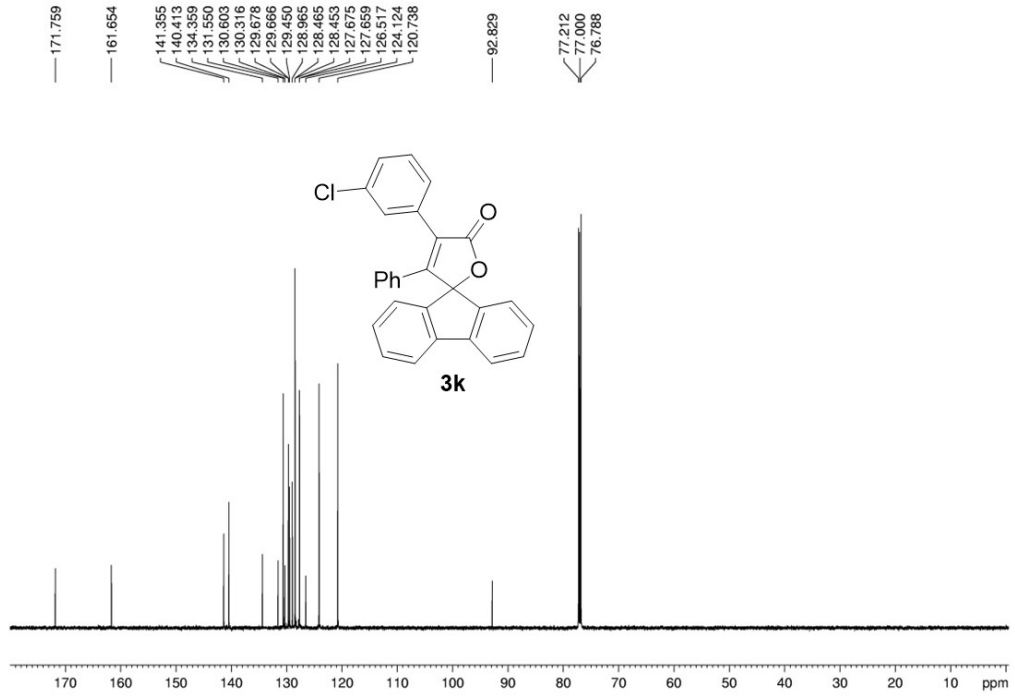
$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3j**



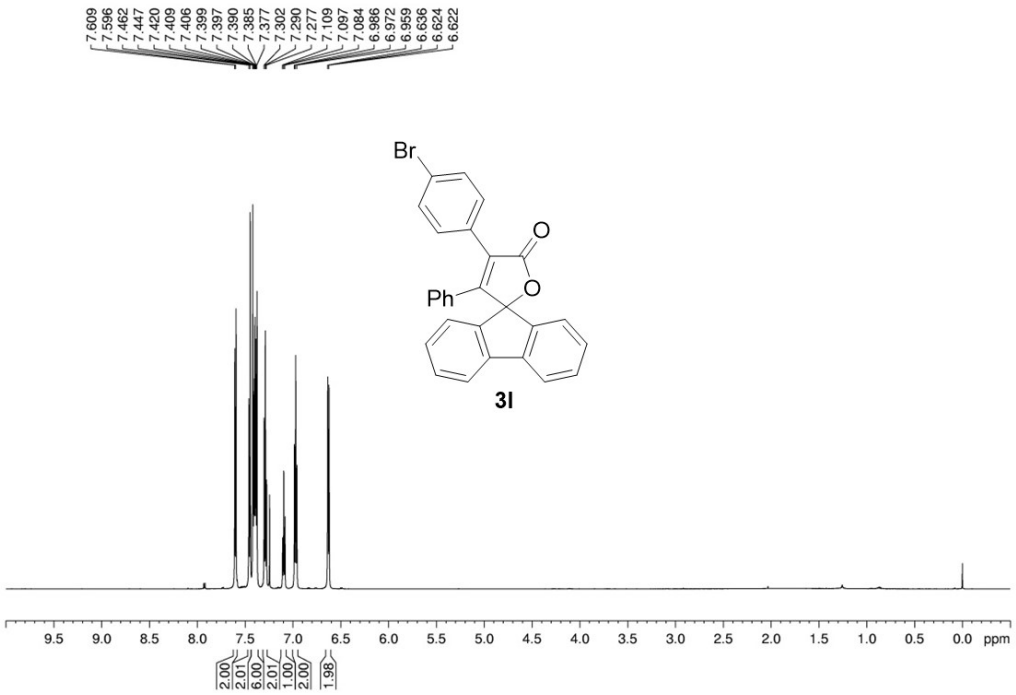



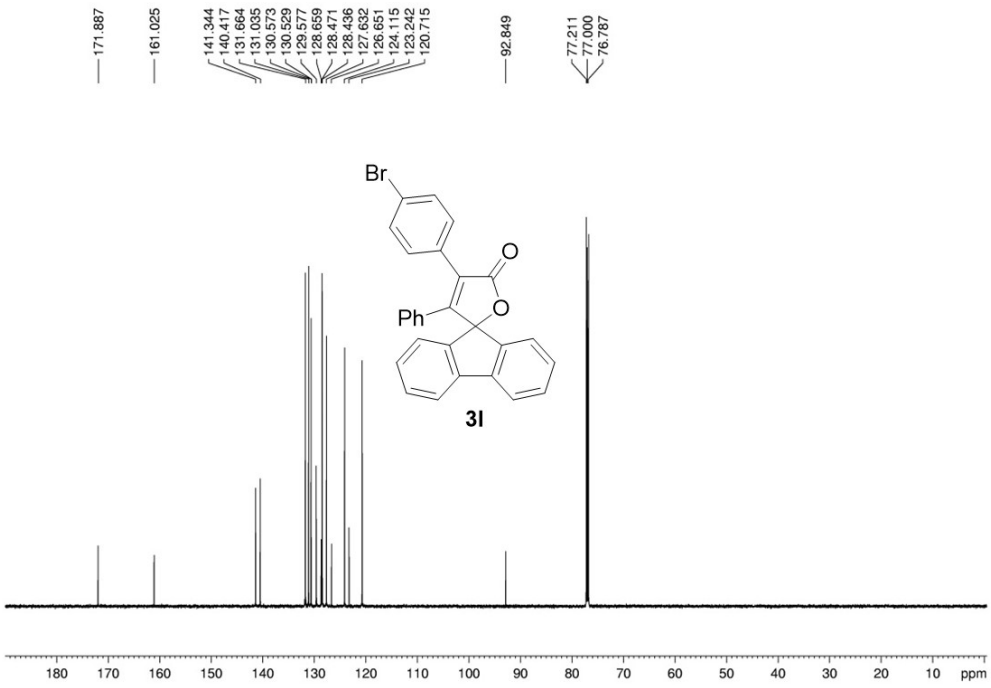
$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3k**



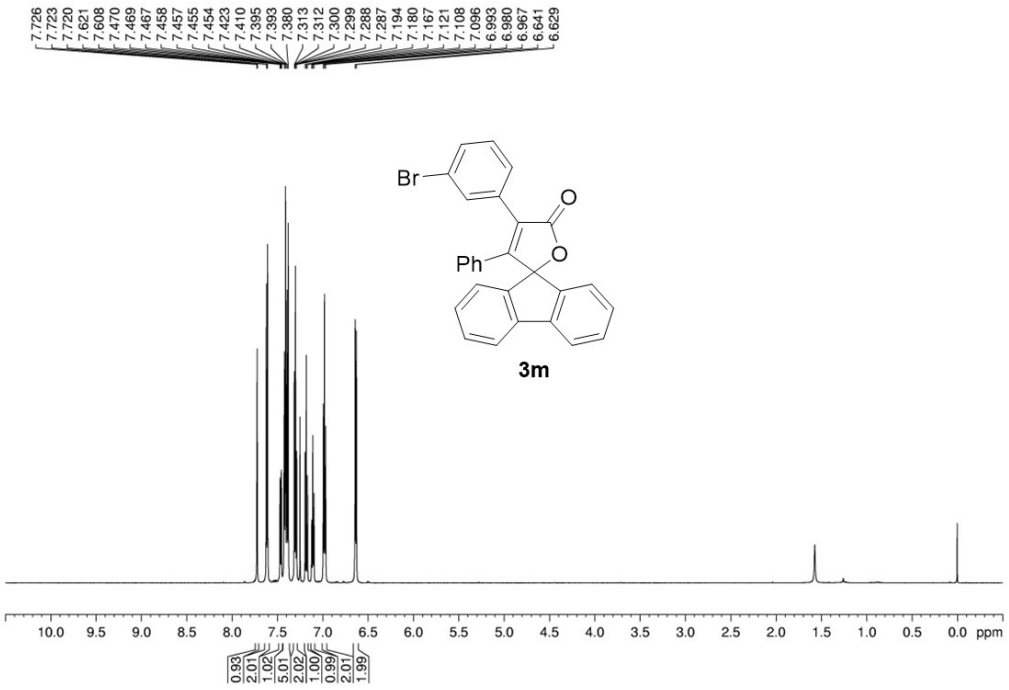



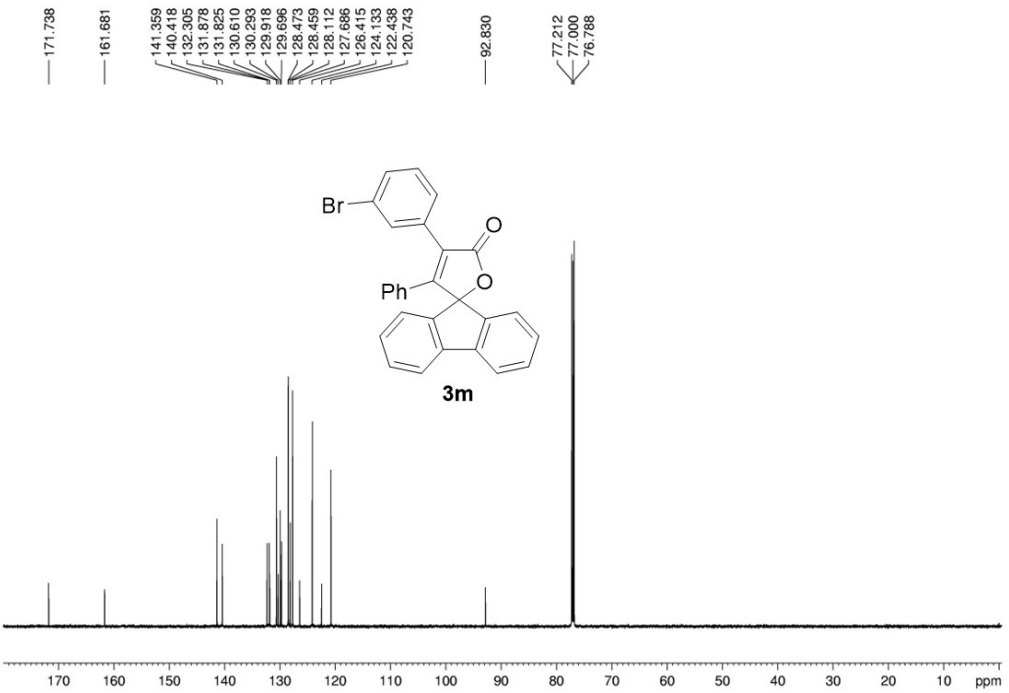
$^{1}H$ NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3l**



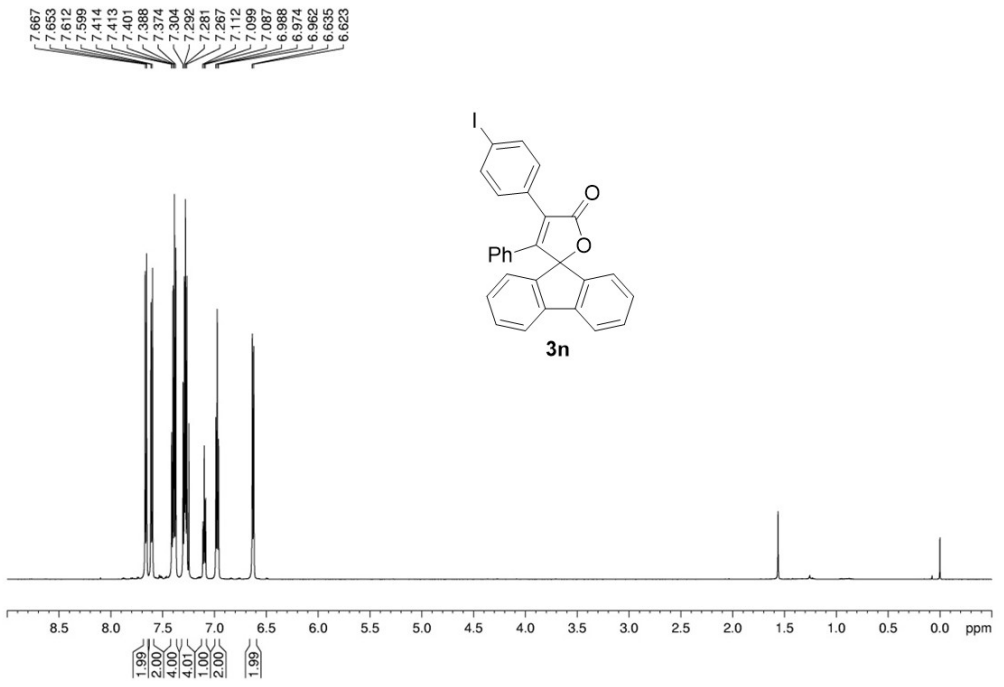



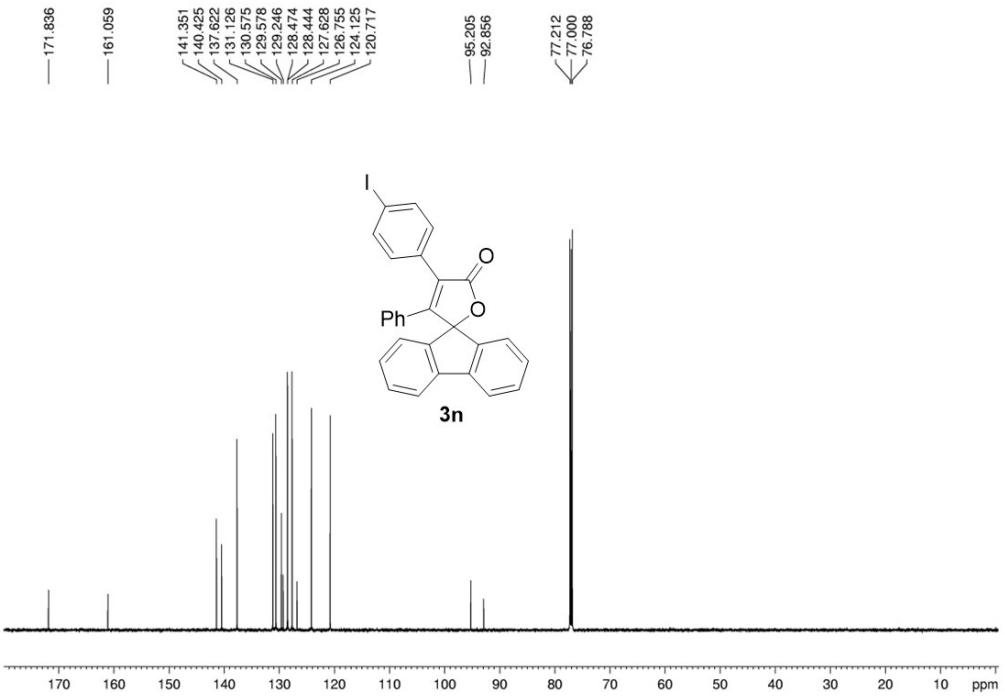
$^{1}H$ NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3m**



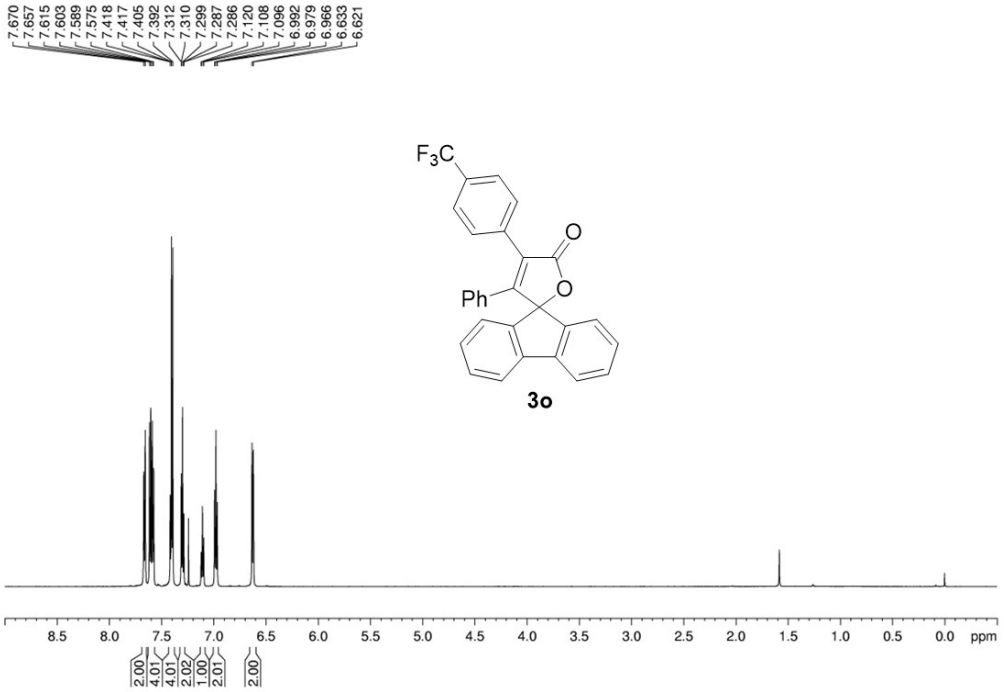



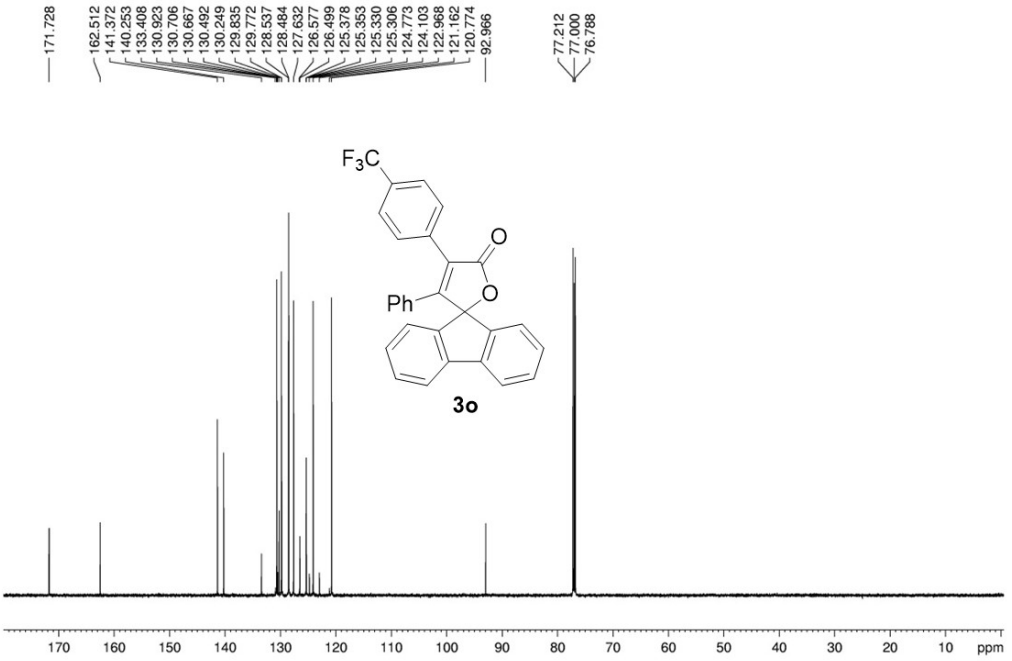
$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3n**

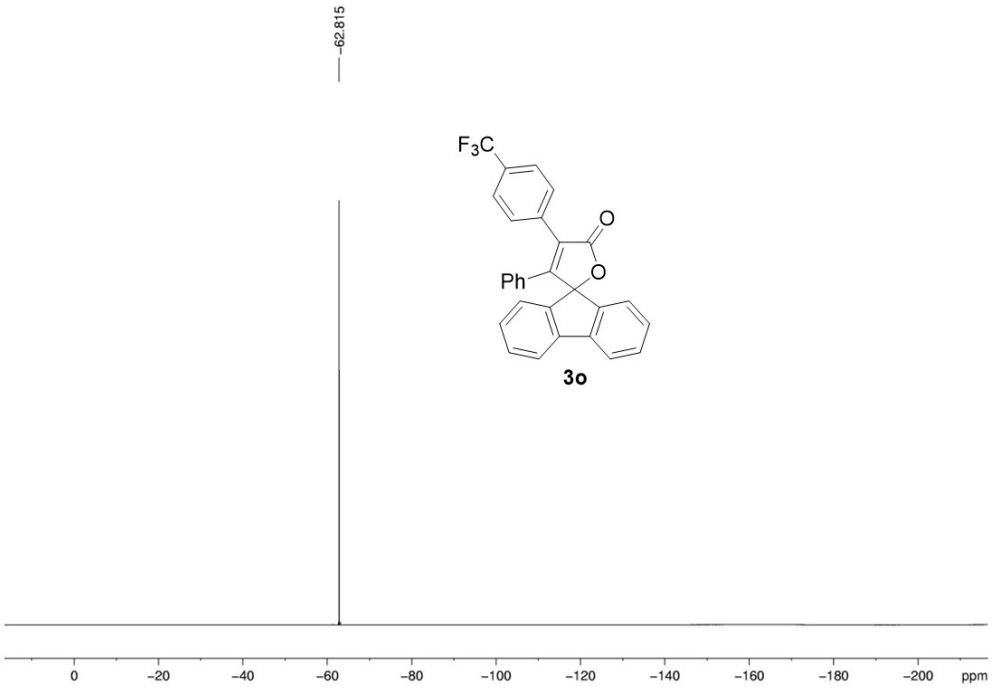


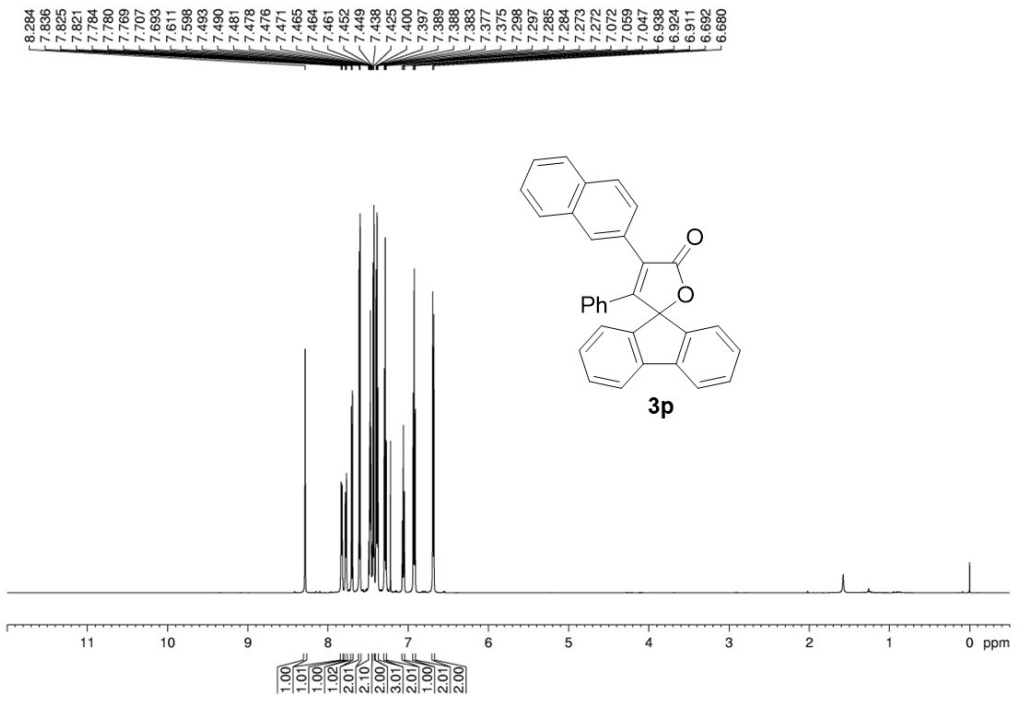
$^{1}H$ NMR (600 MHz, $CDCl_3$), $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) and $^{19}F\{^{1}H\}$ NMR (565 MHz, $CDCl_3$) of **3o**

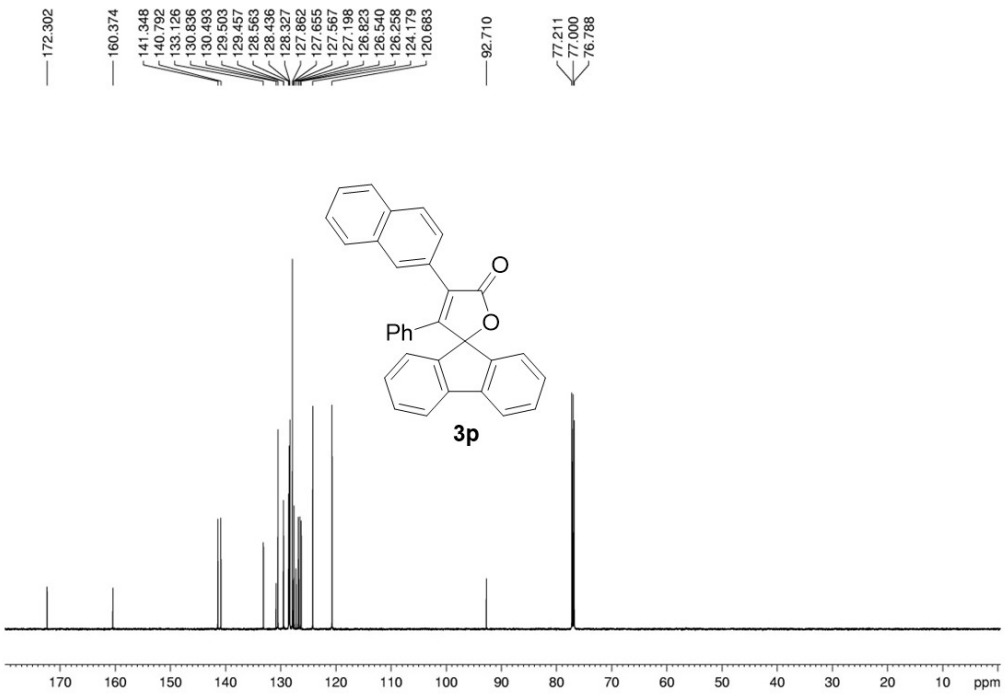


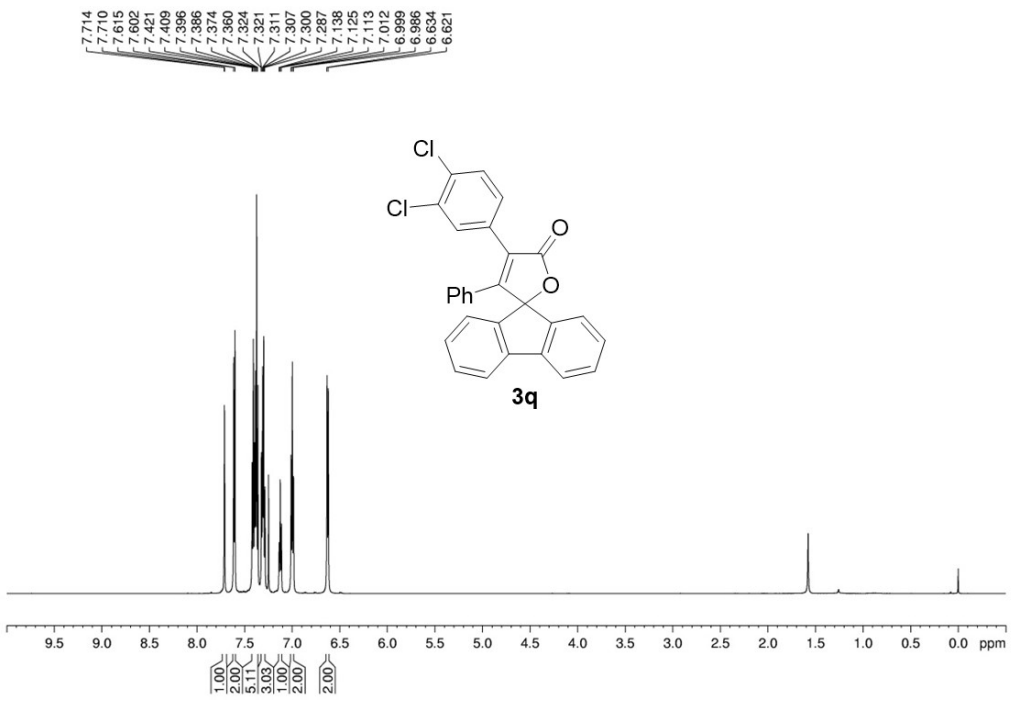


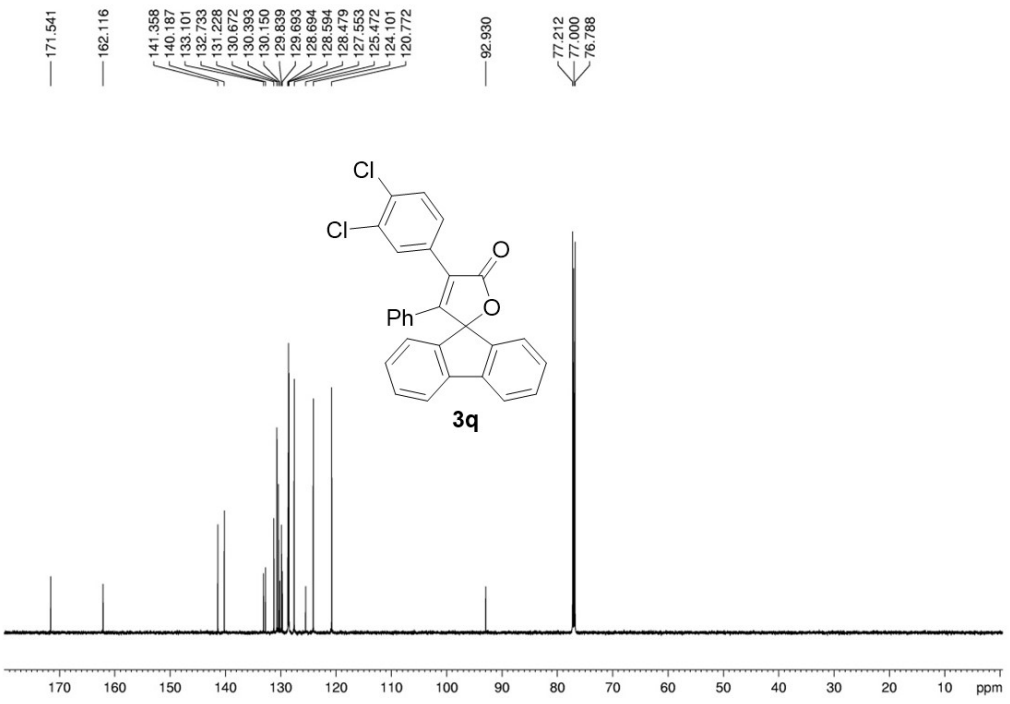

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3p**



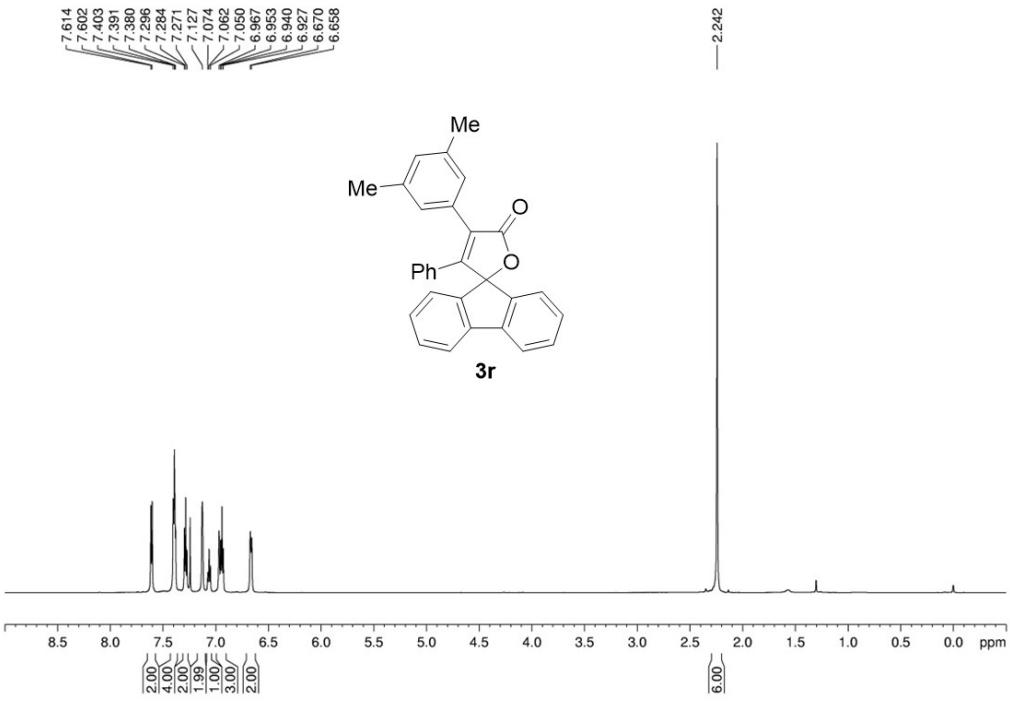


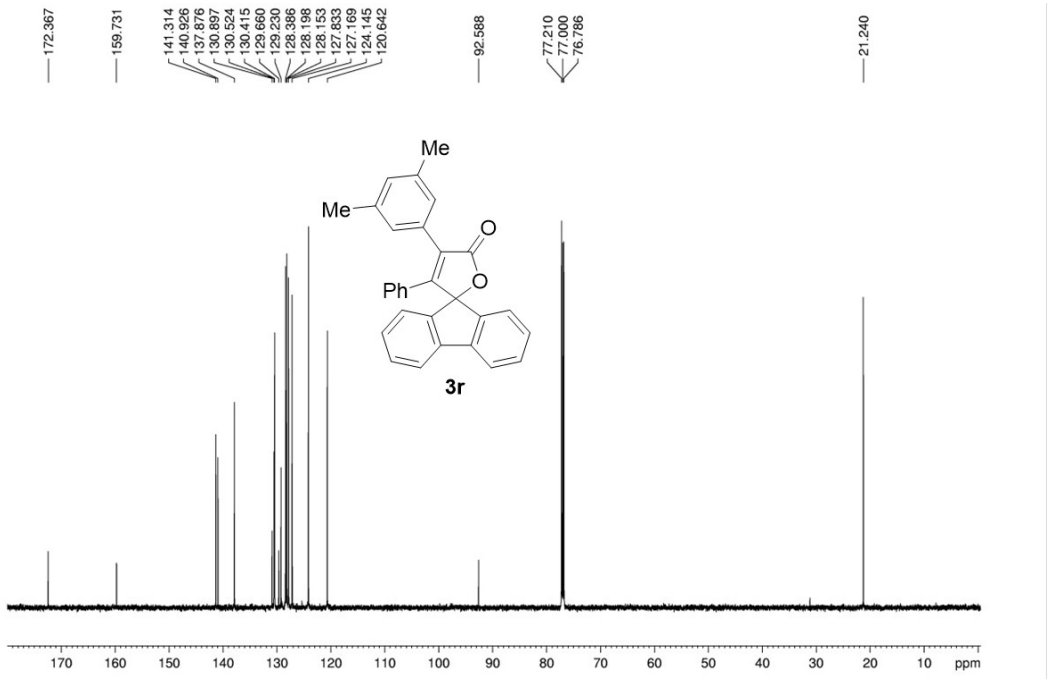

$^{1}H$ NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3q**



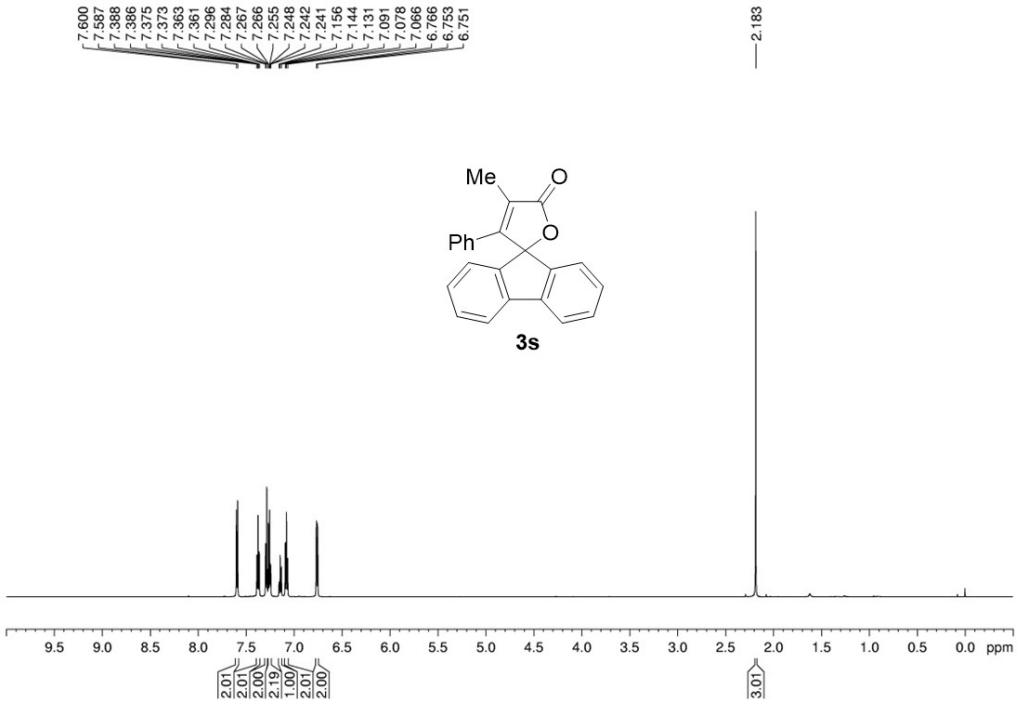


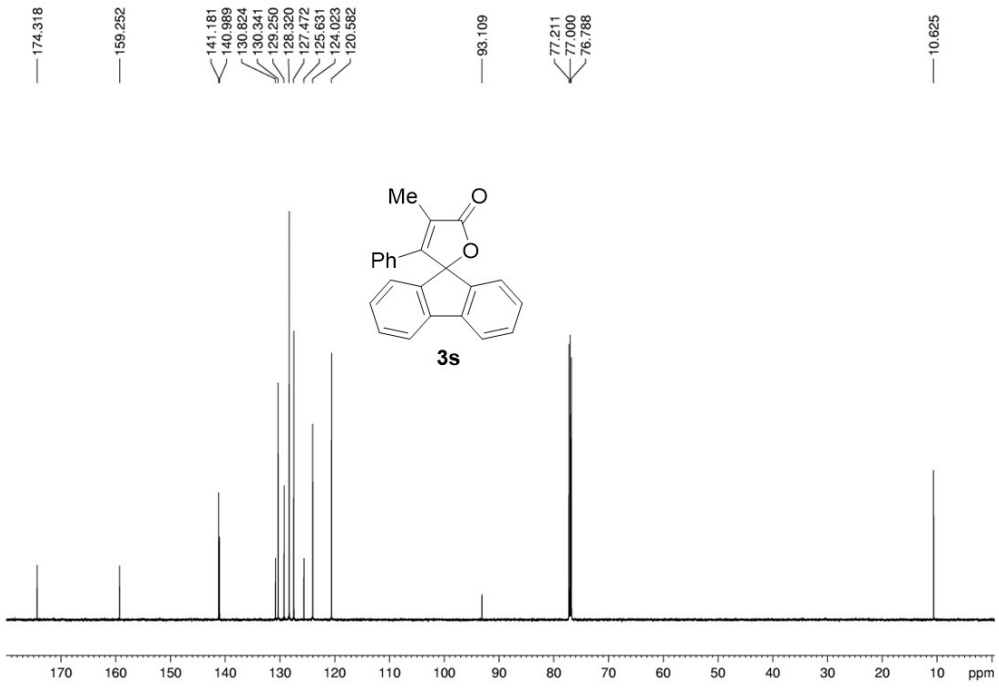

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3r**



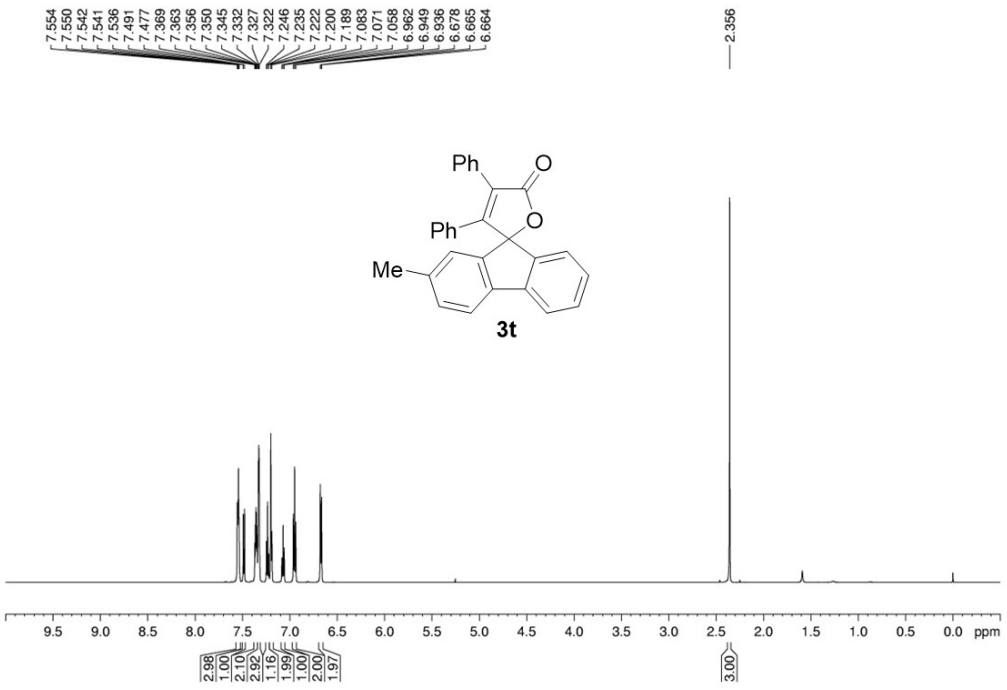


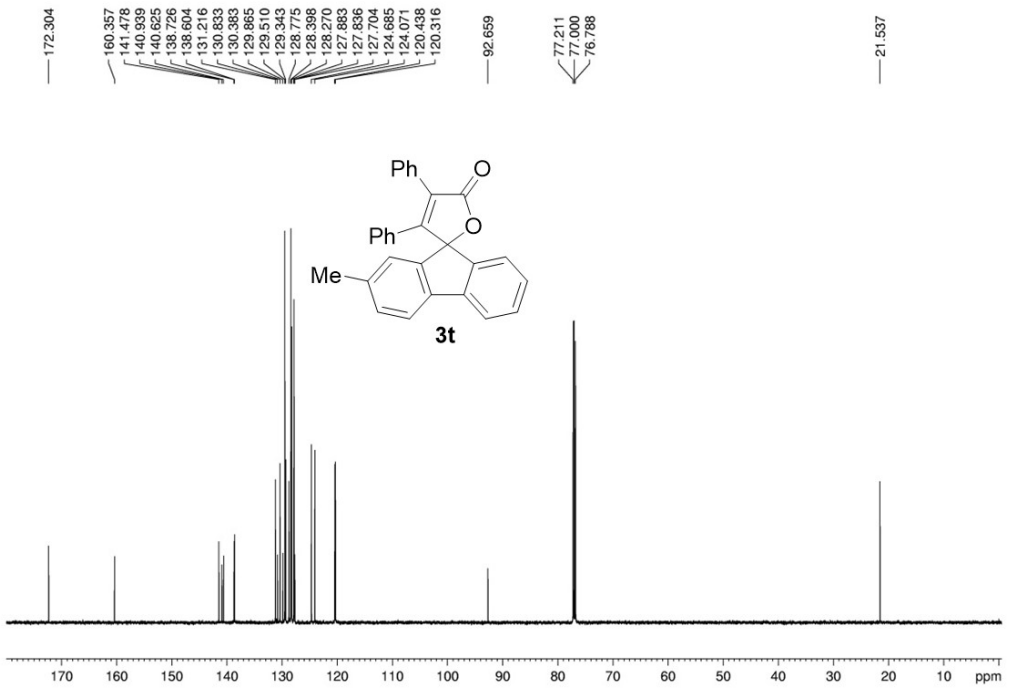

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3s**



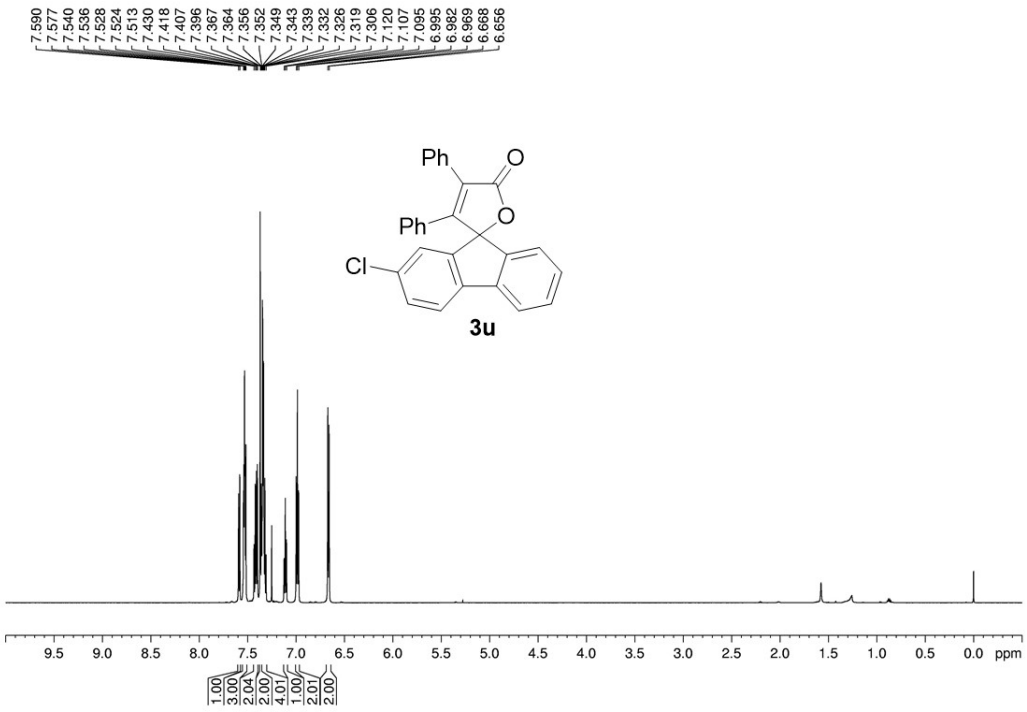


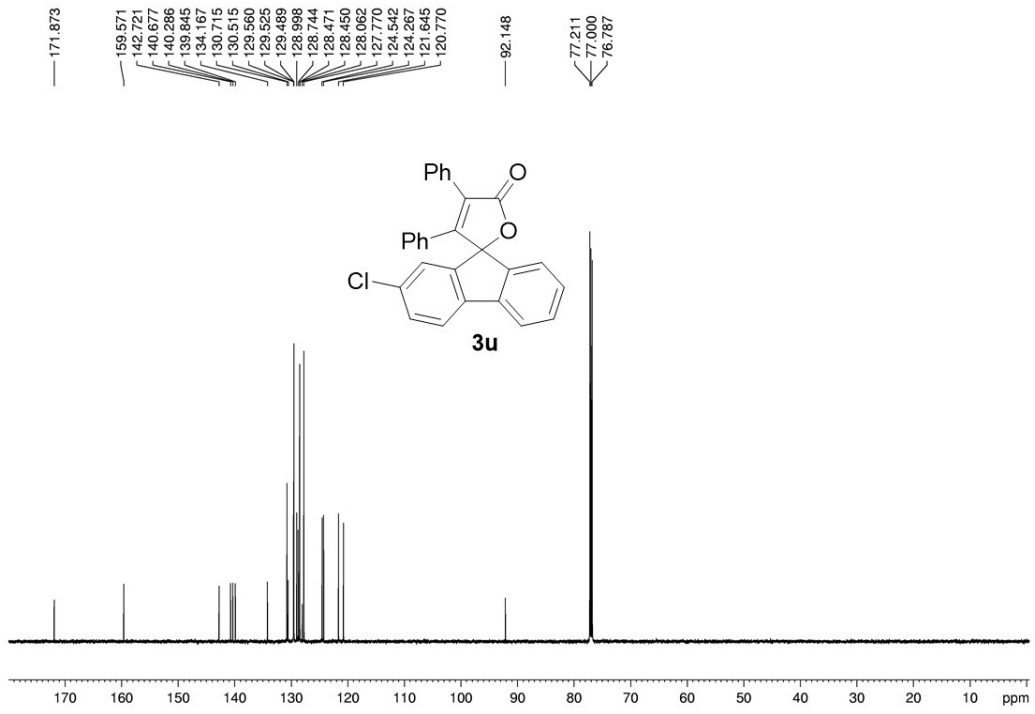

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3t**



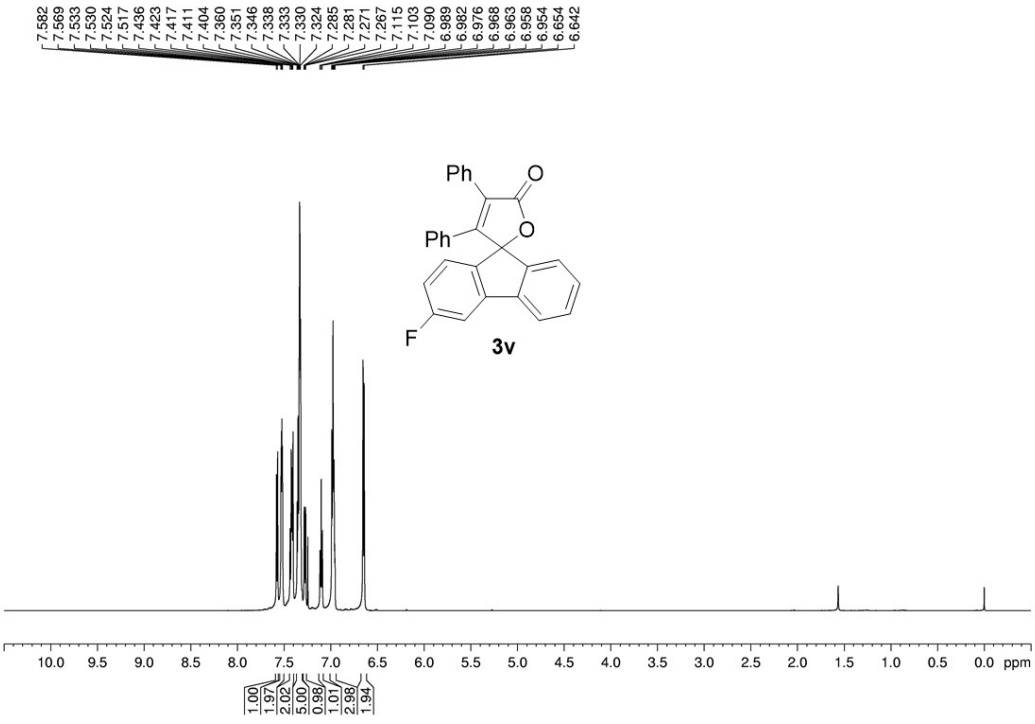


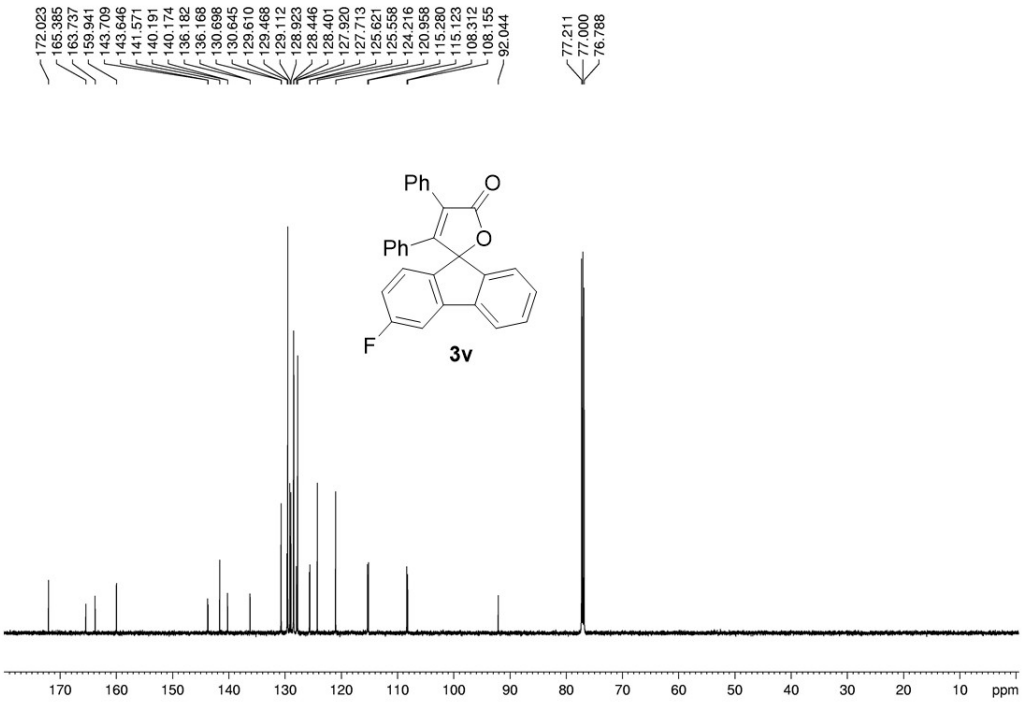

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3u**






$^{1}$H NMR (600 MHz, $CDCl_3$), $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) and $^{19}F\{^{1}H\}$ NMR (565 MHz, $CDCl_3$) of **3v**

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Ph
O
F
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110
100
90
80
70
60
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30
20
10
ppm


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Ph
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F
3v
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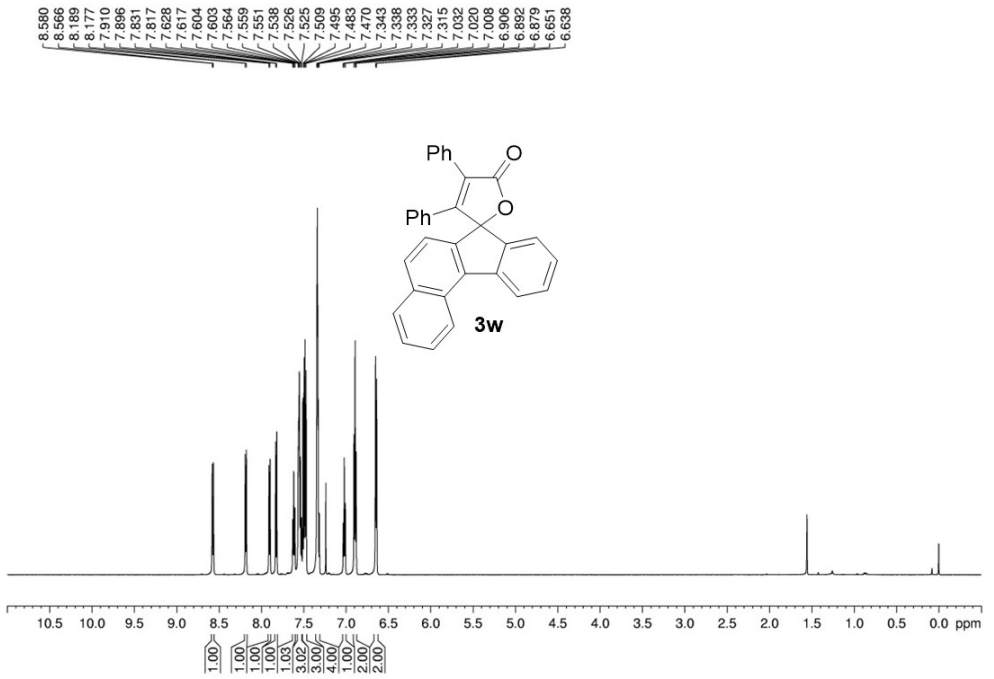
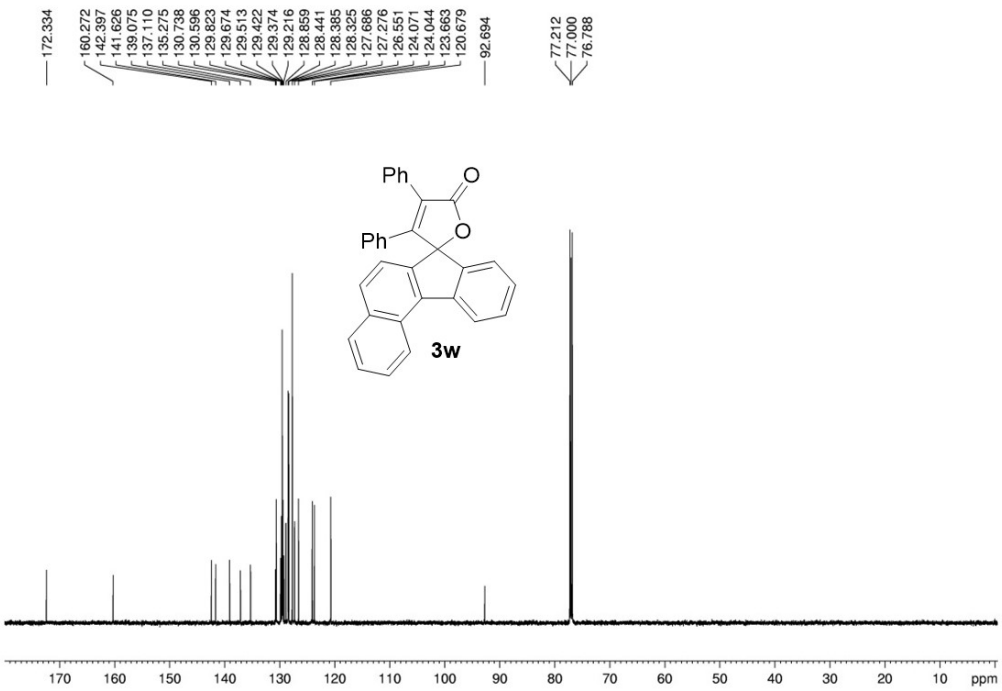
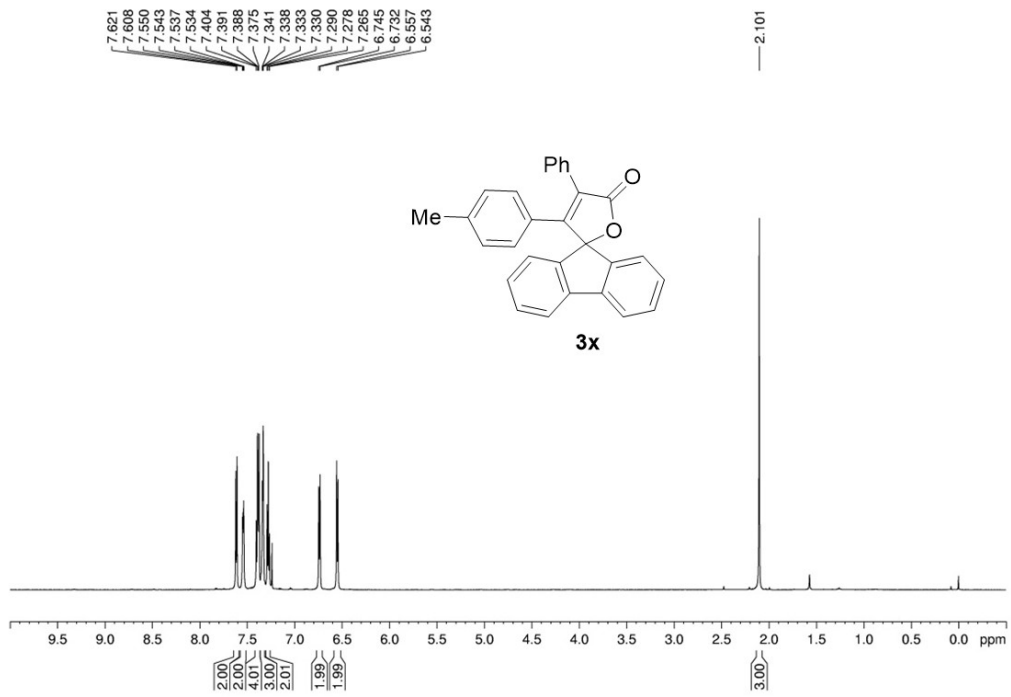
ppm

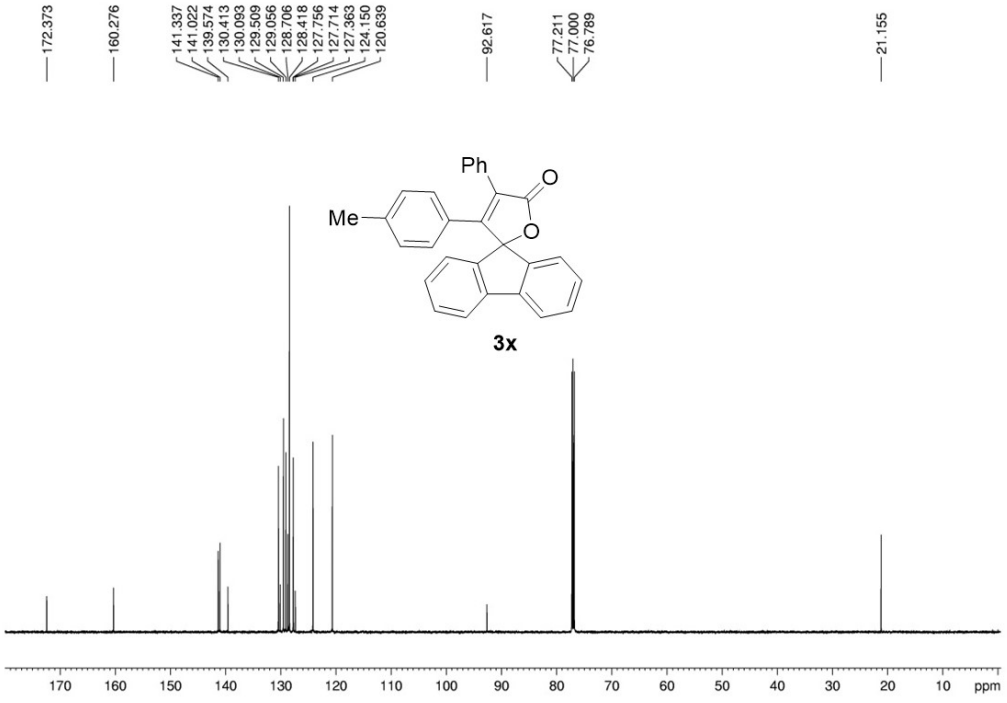
$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3w**



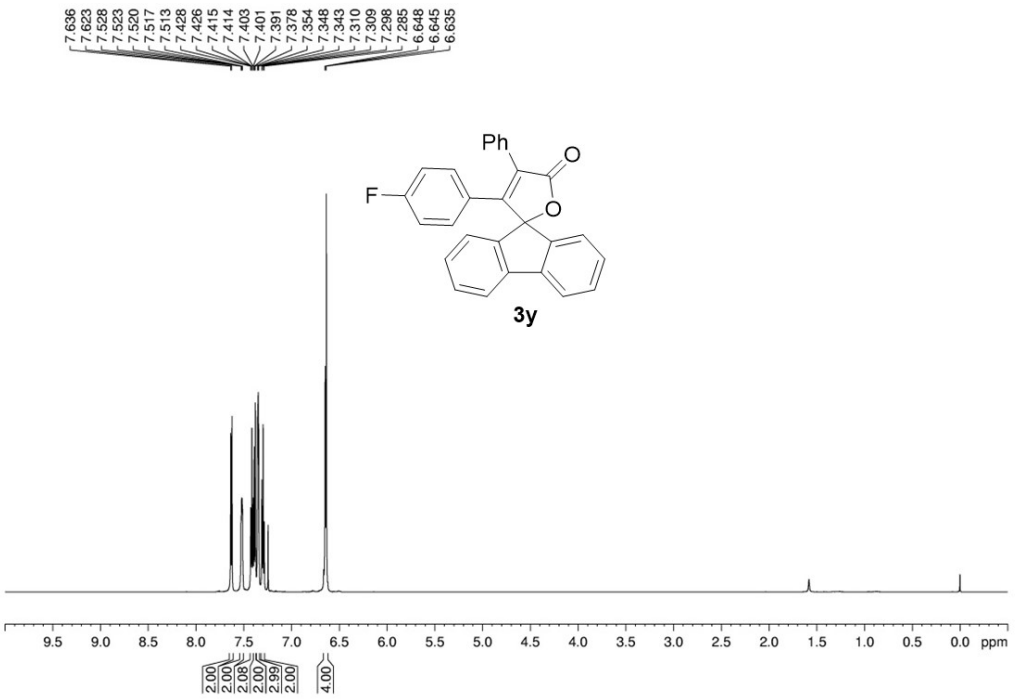



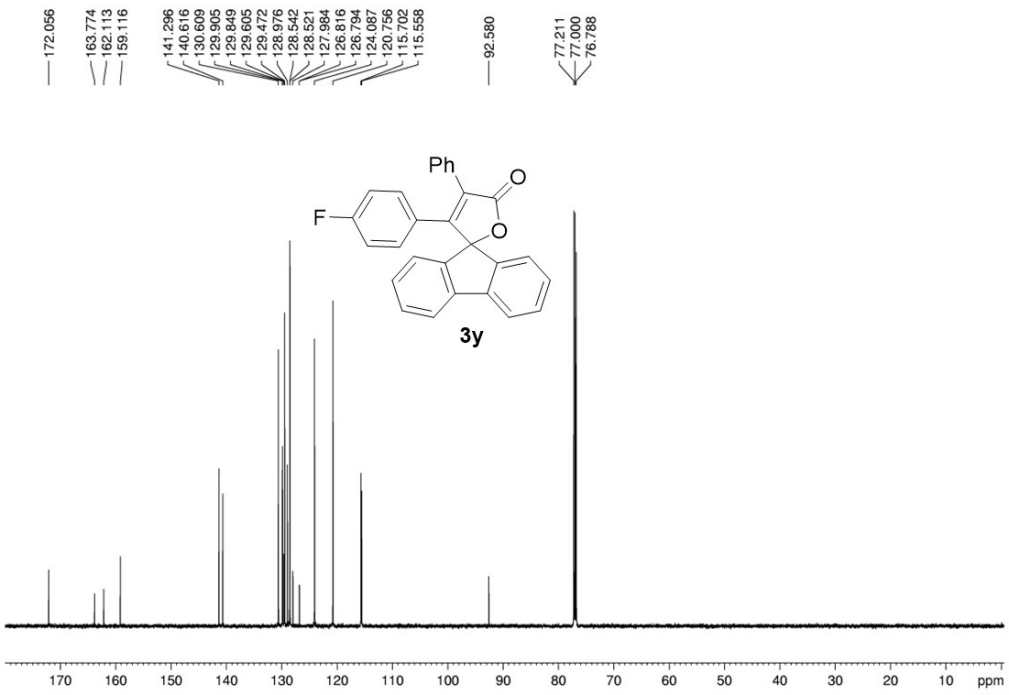
$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3x**

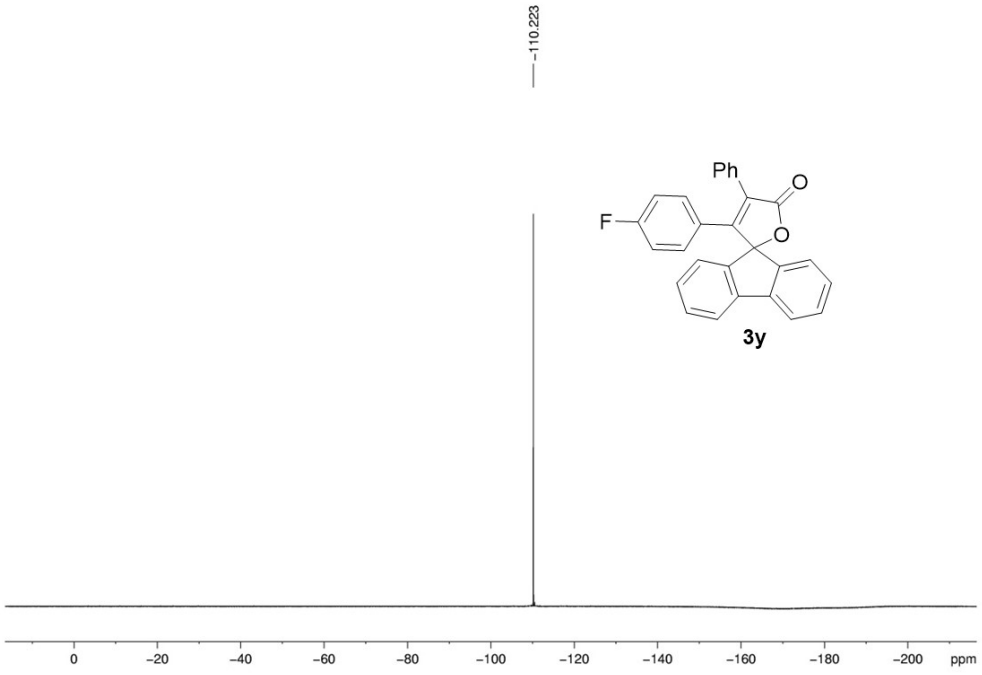


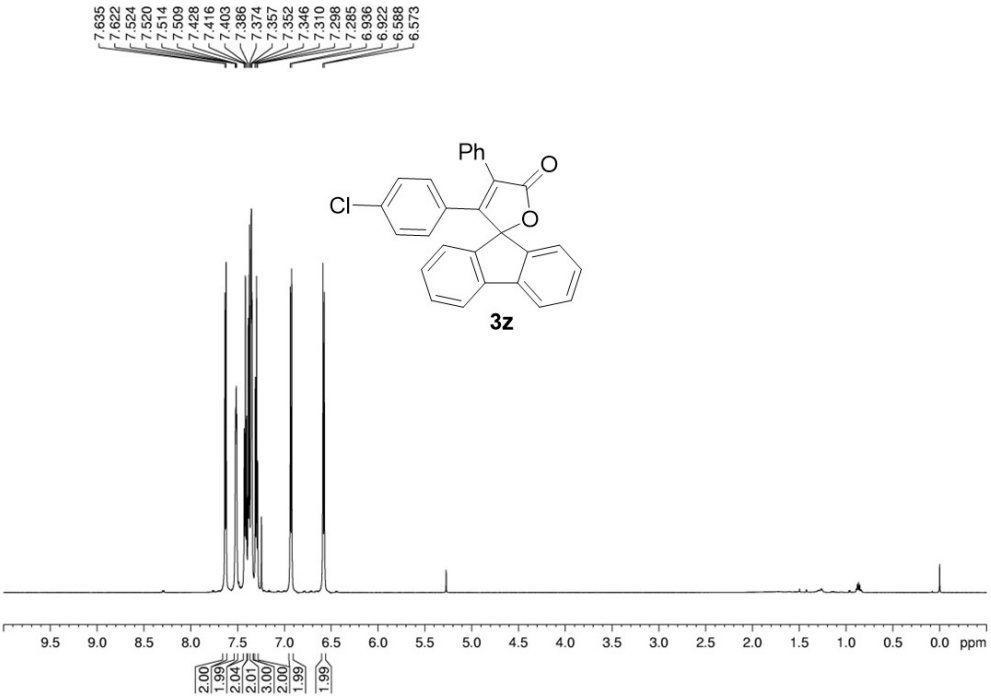
$^{1}H$ NMR (600 MHz, $CDCl_3$), $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) and $^{19}F\{^{1}H\}$ NMR (565 MHz, $CDCl_3$) of **3y**

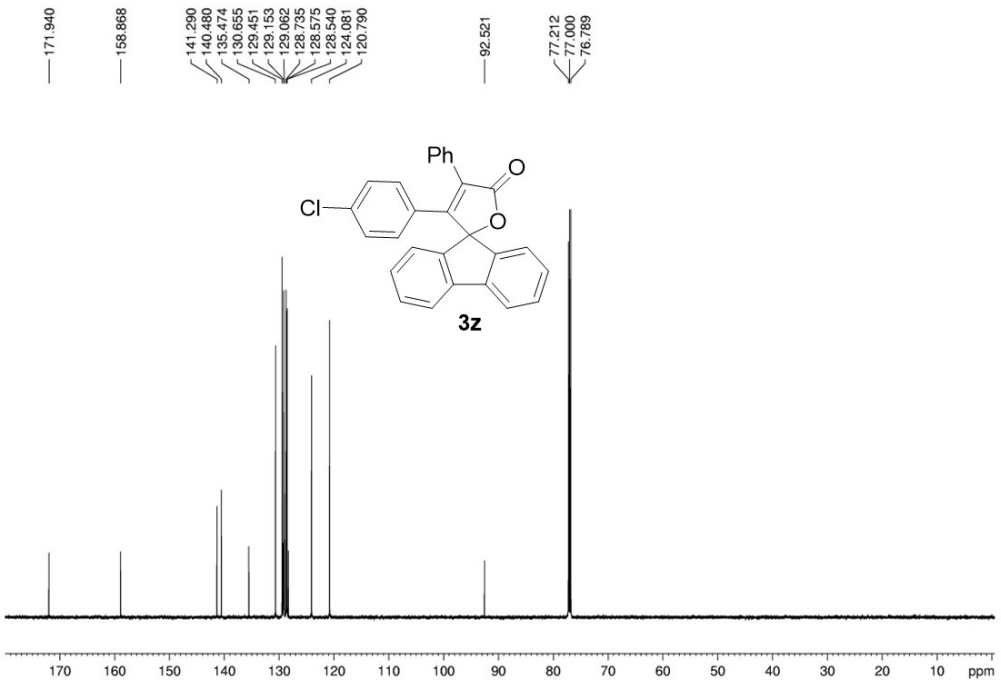


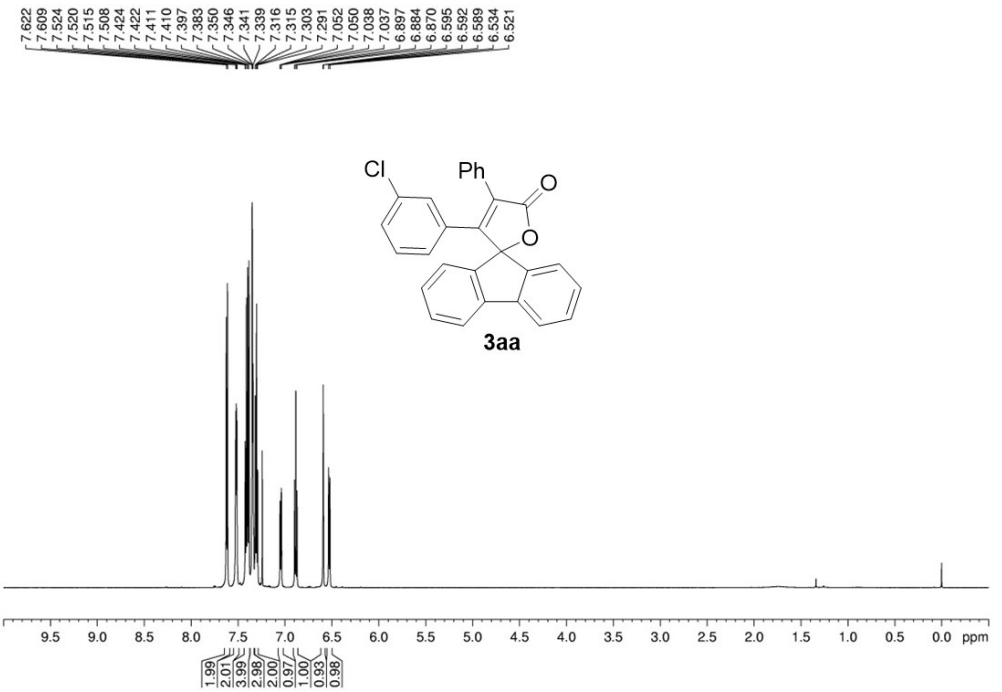


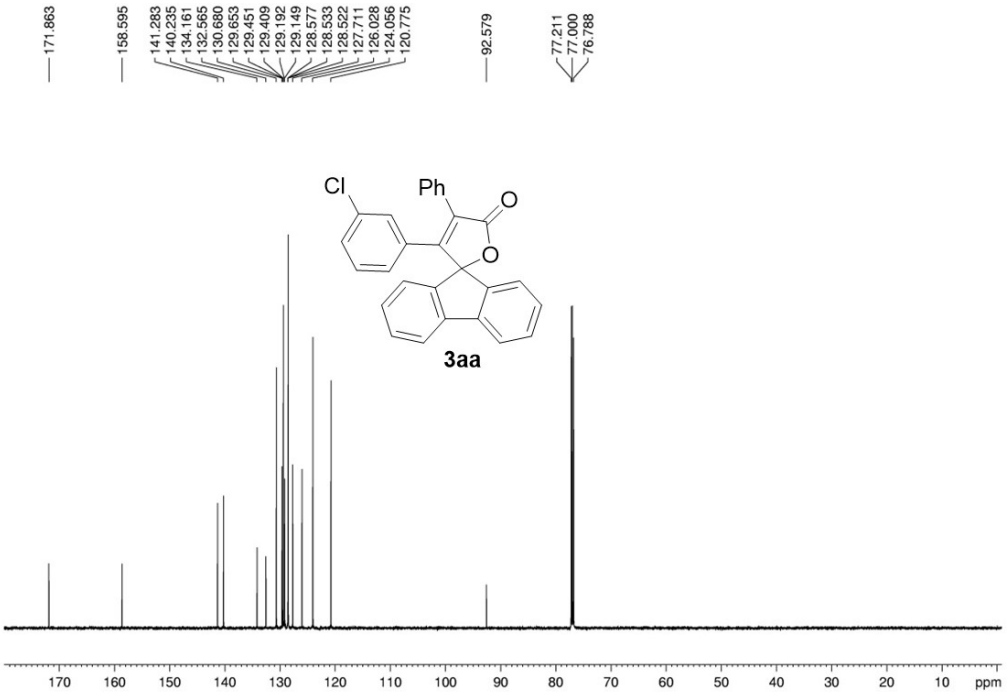

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3z**



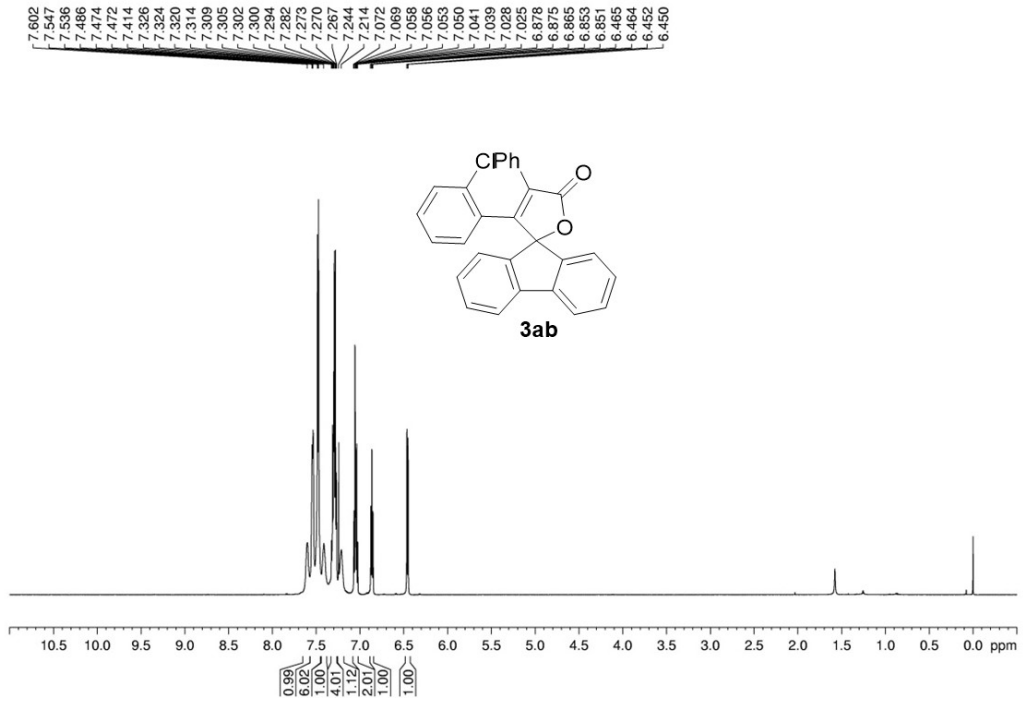


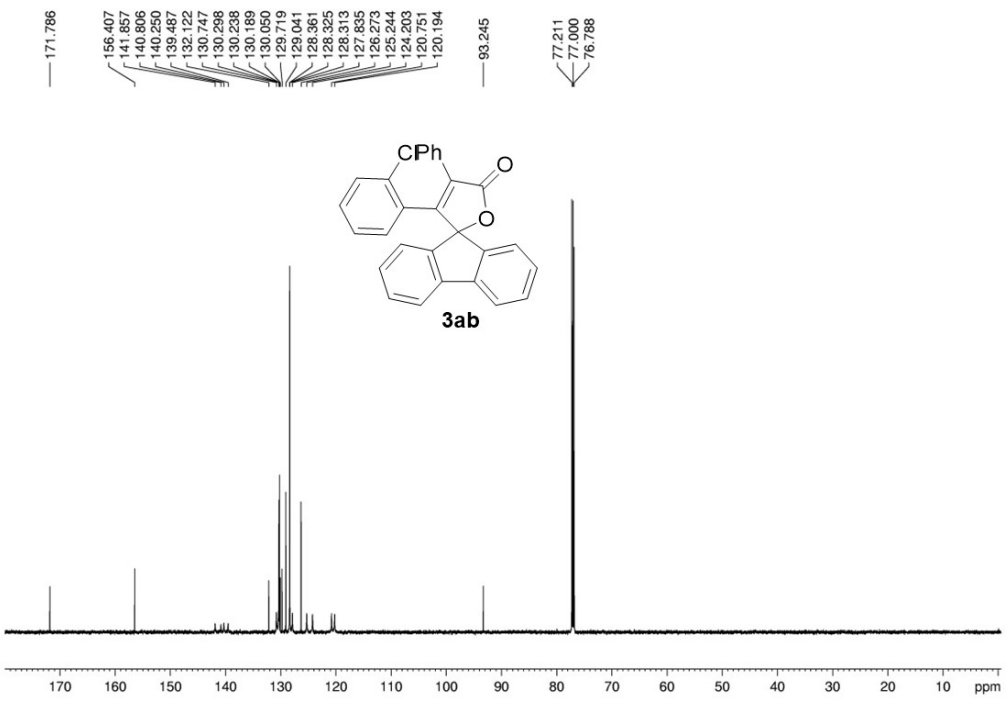

$^{1}H$ NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3aa**



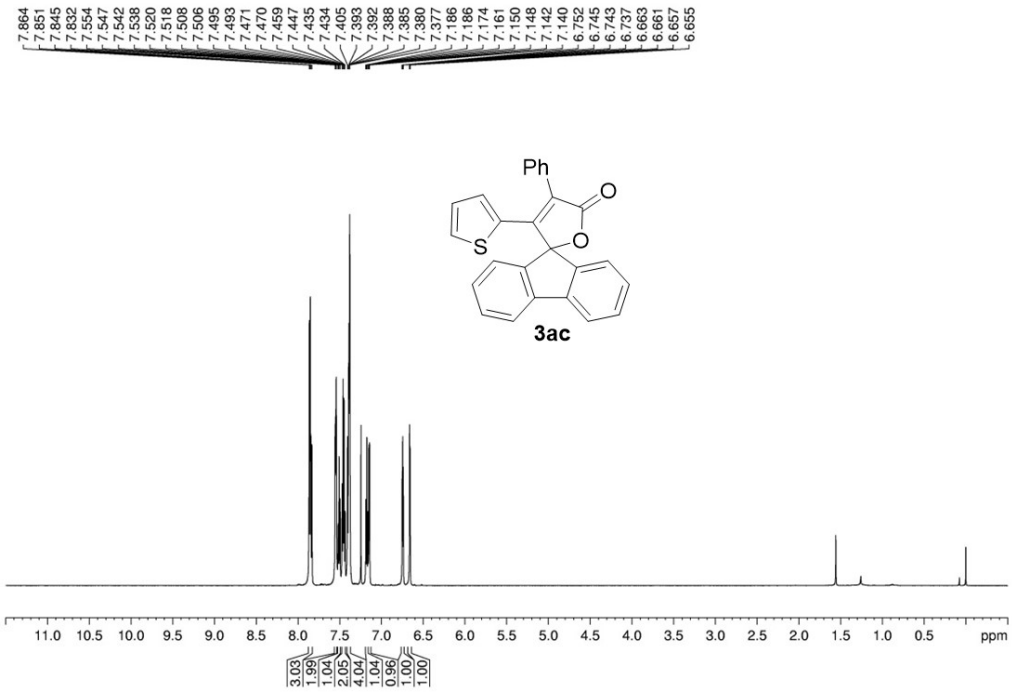


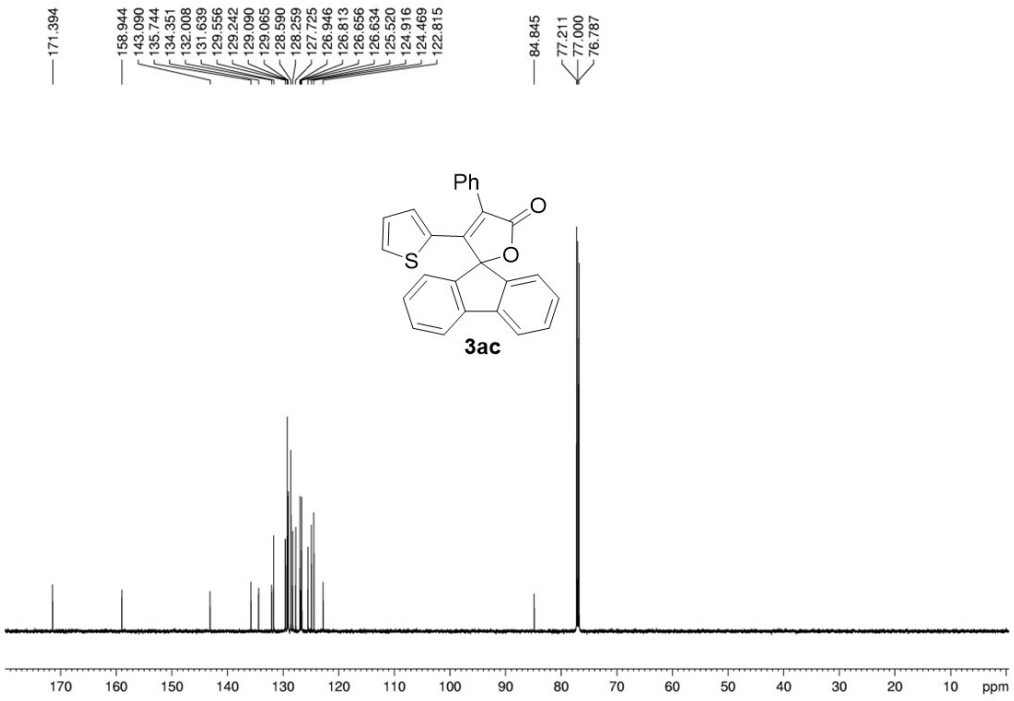

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3ab**



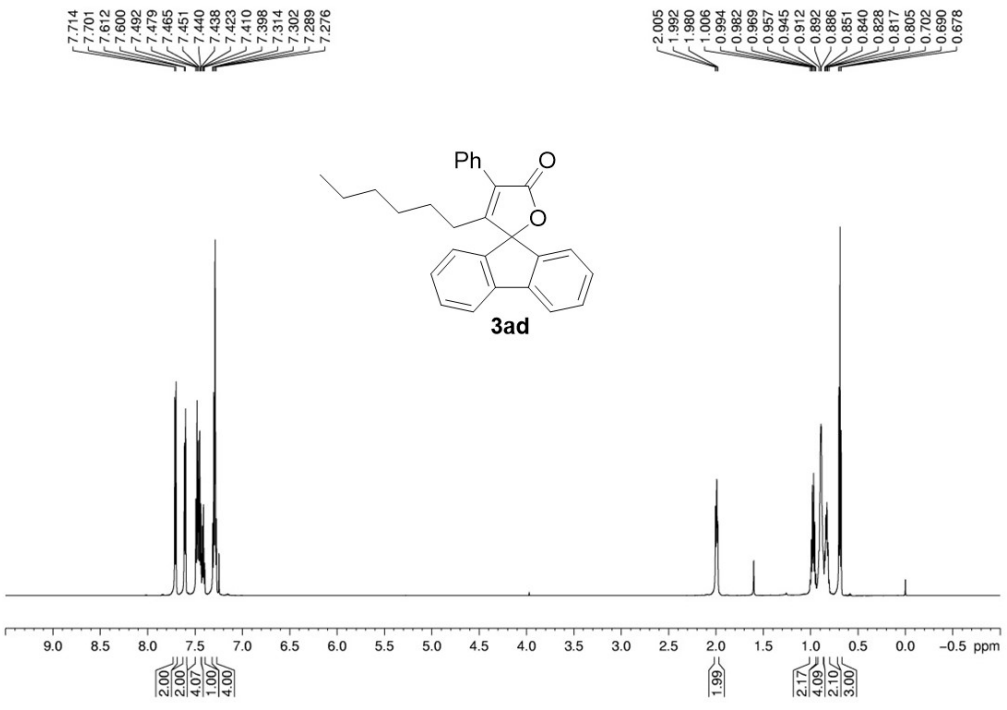


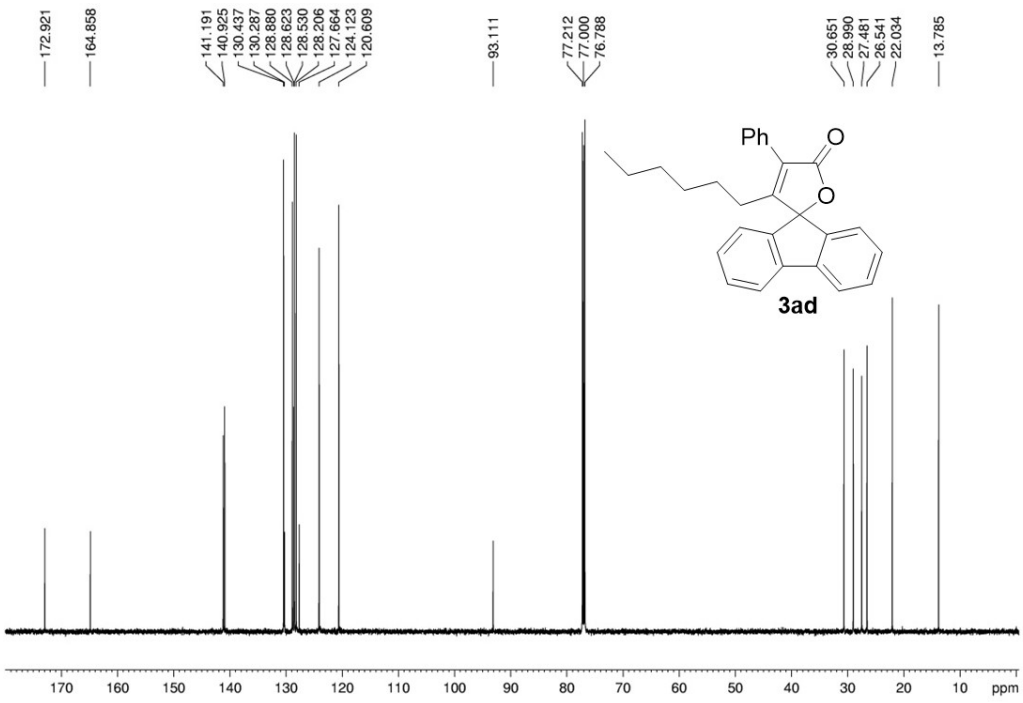

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3ac**



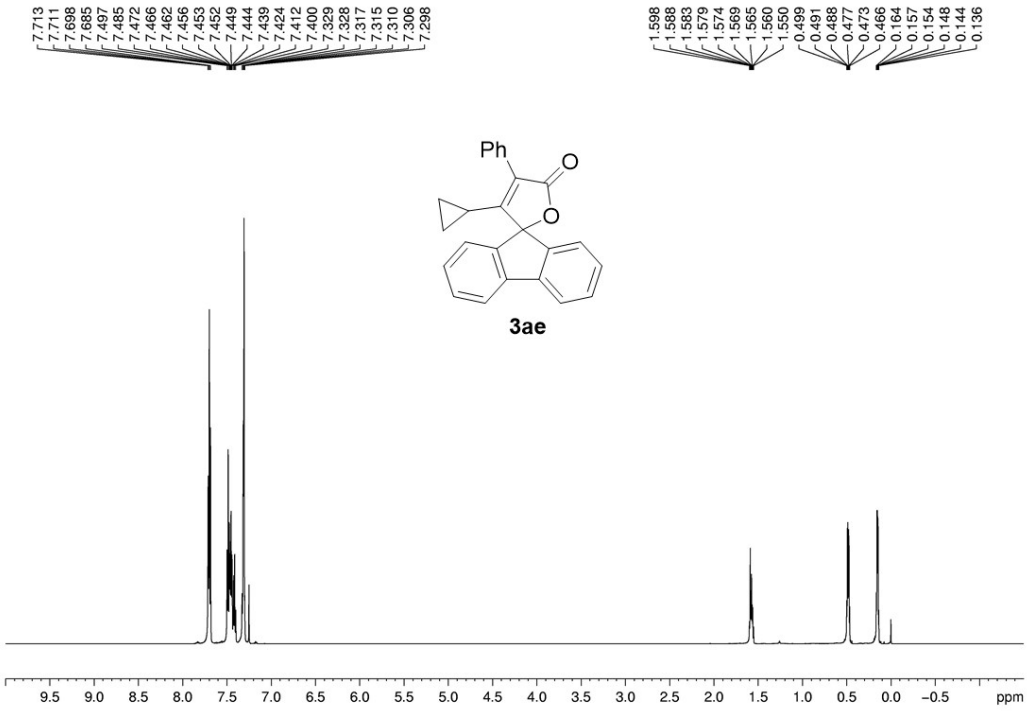


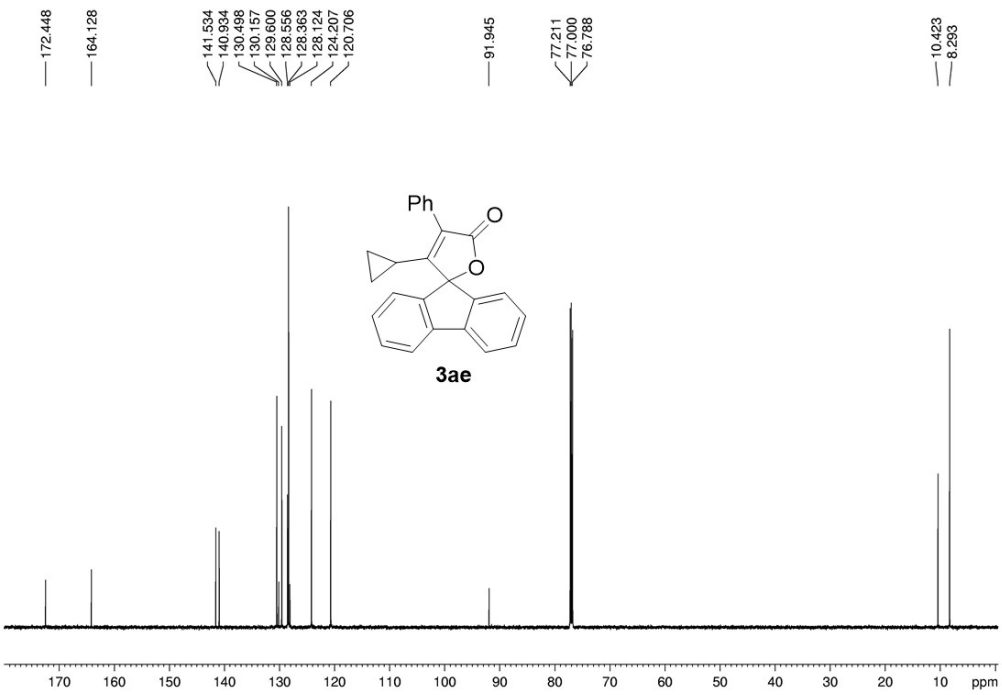

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3ad**



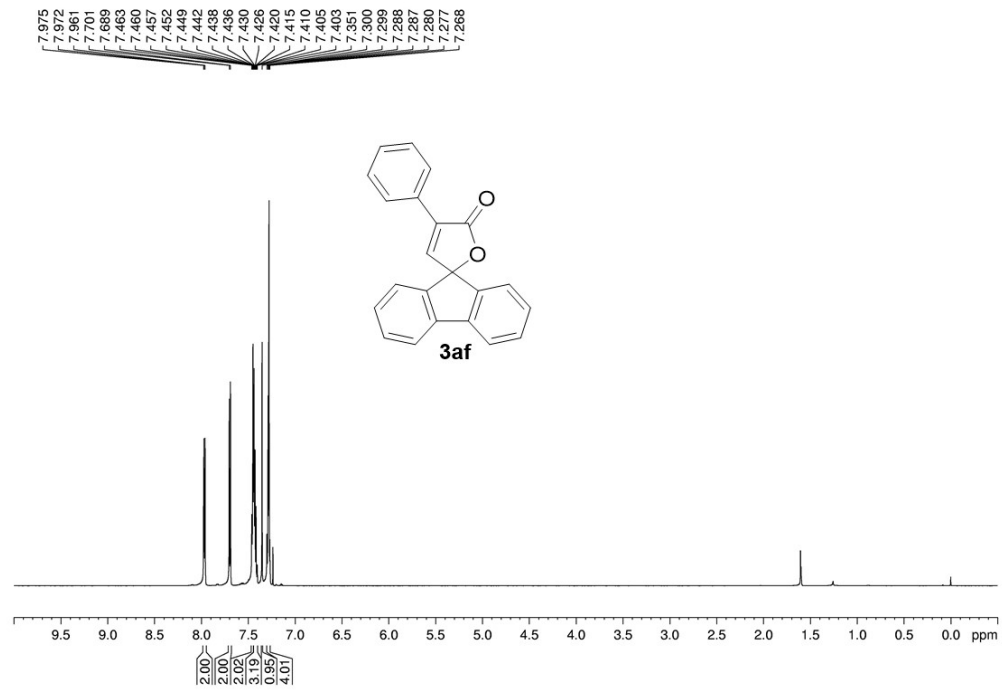


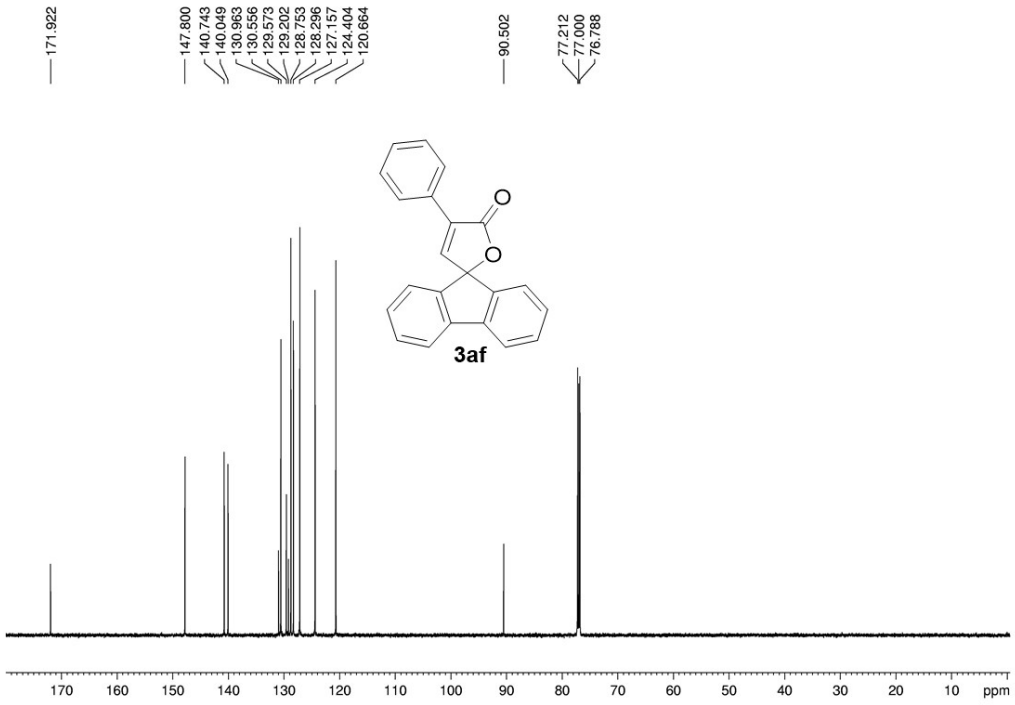

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3ae**



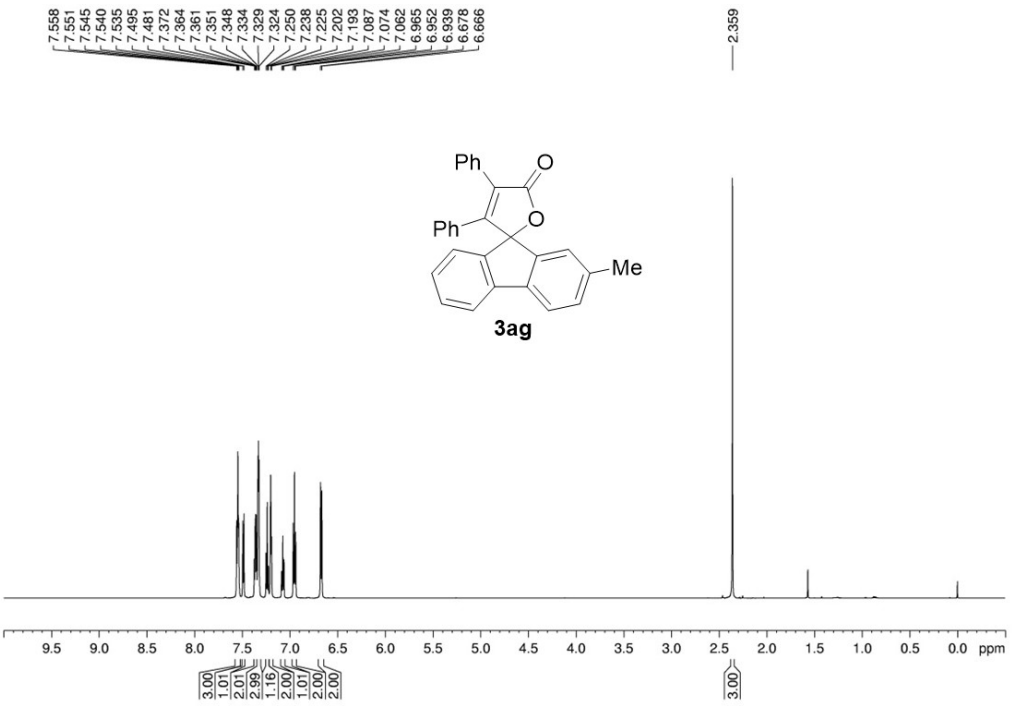


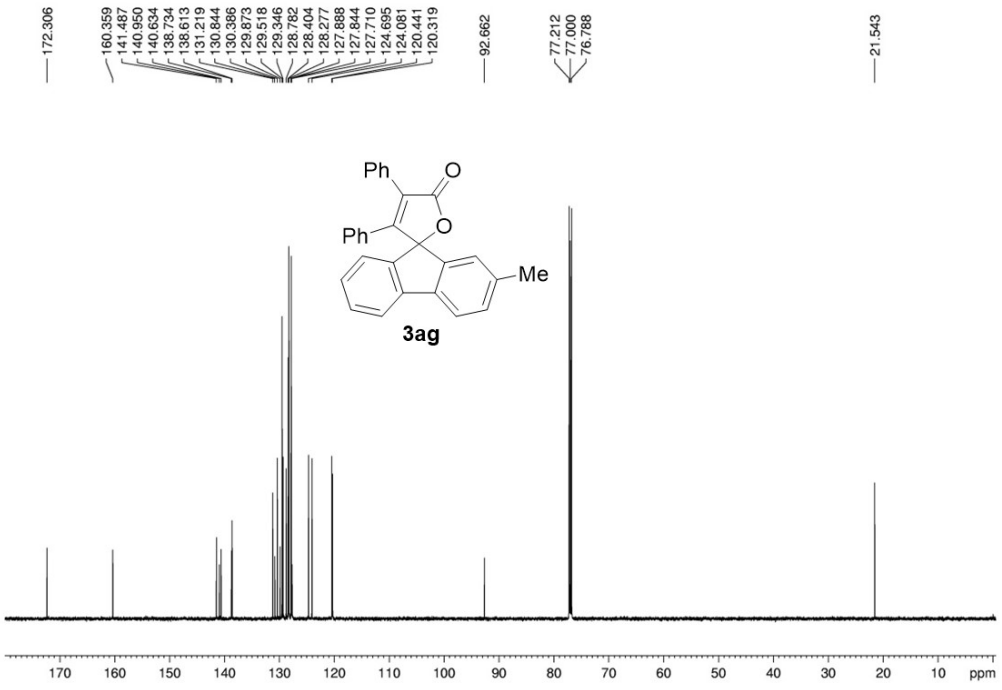

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3af**



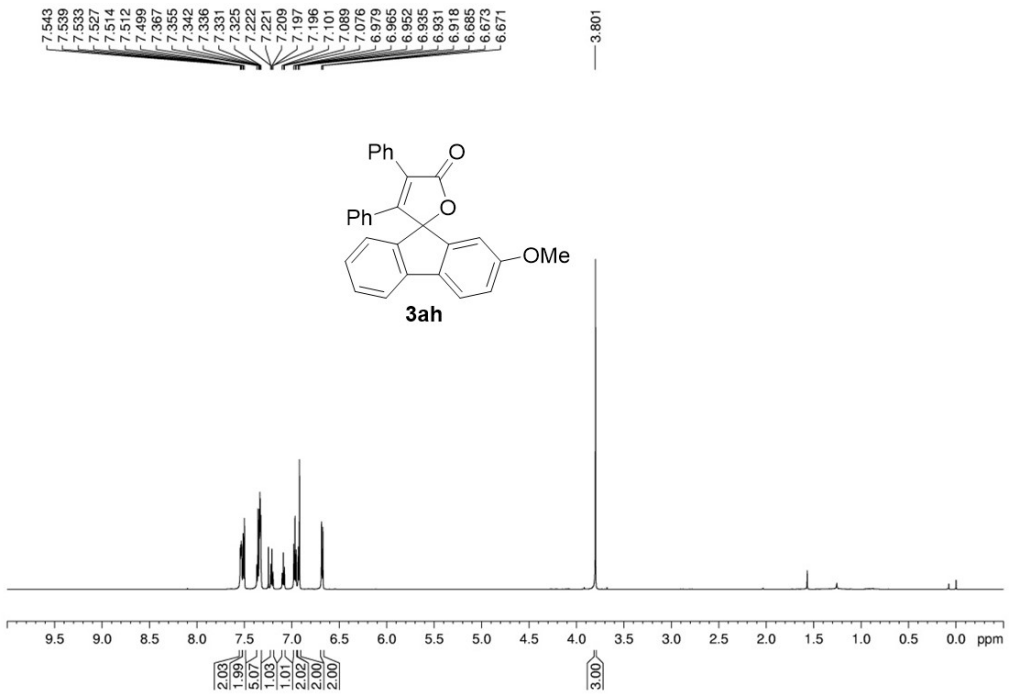


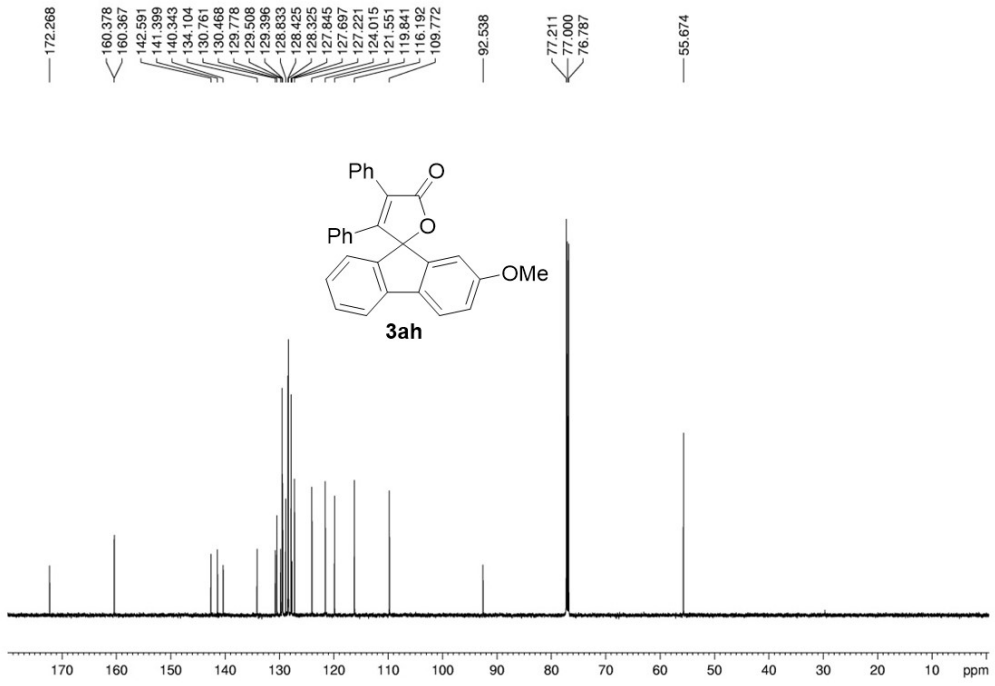

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3ag**



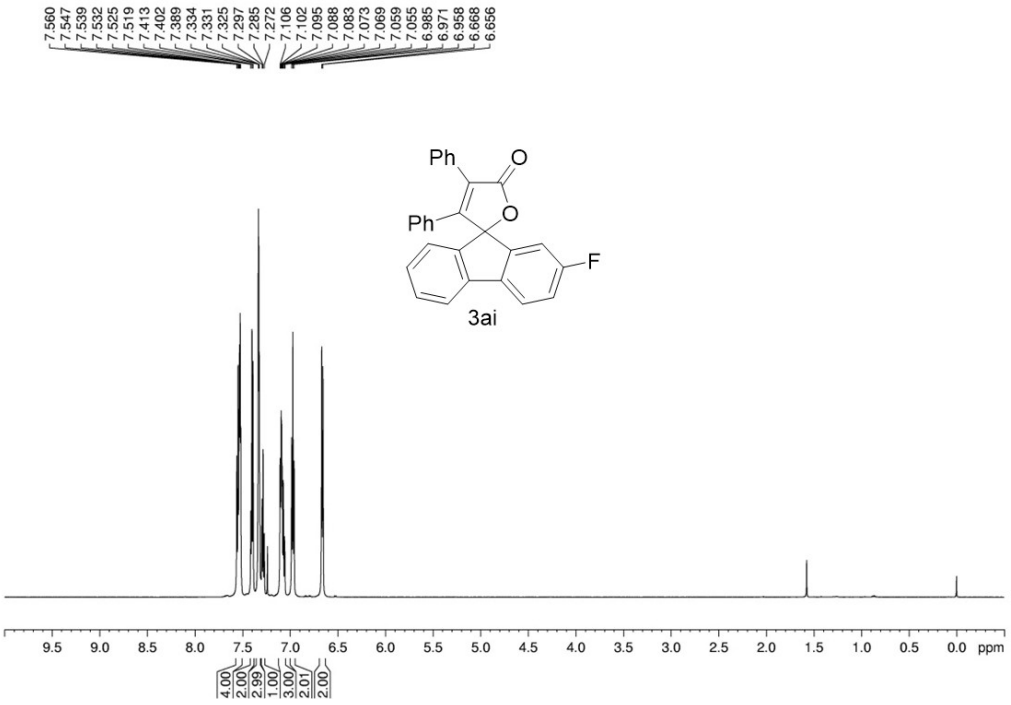


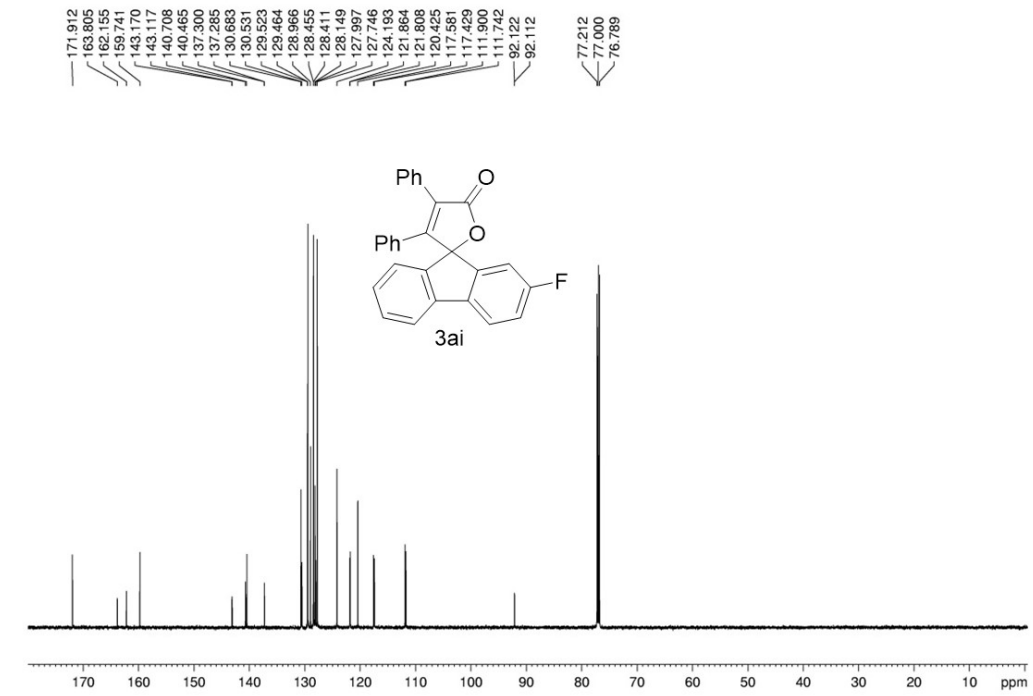

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3ah**

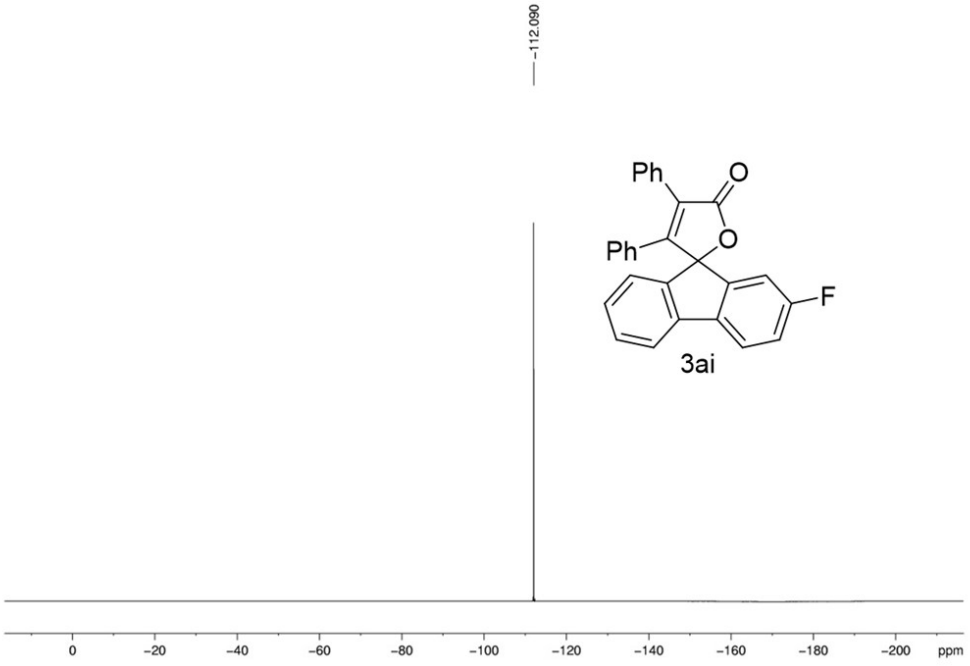




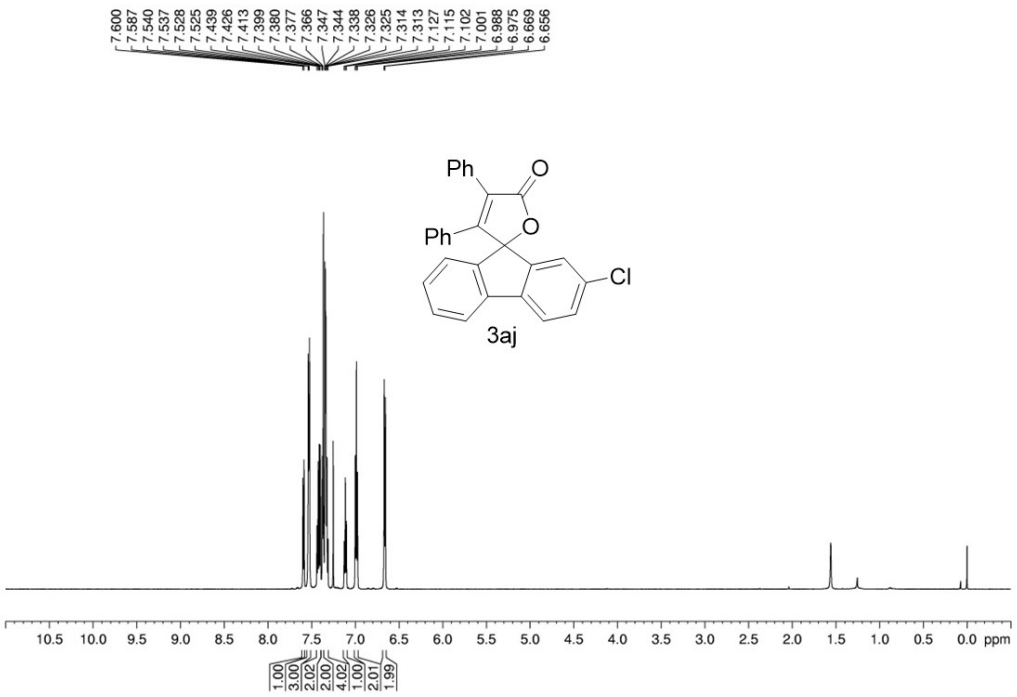

$^{1}$H NMR (600 MHz, $CDCl_3$), $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) and $^{19}F\{^{1}H\}$ NMR (565 MHz, $CDCl_3$) of **3ai**

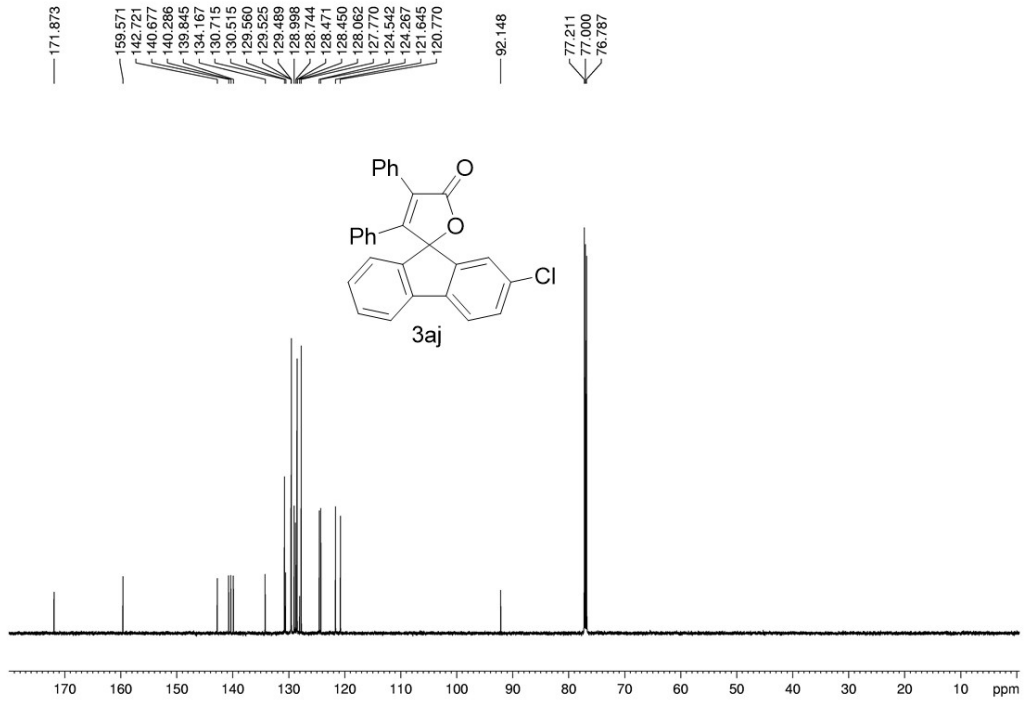




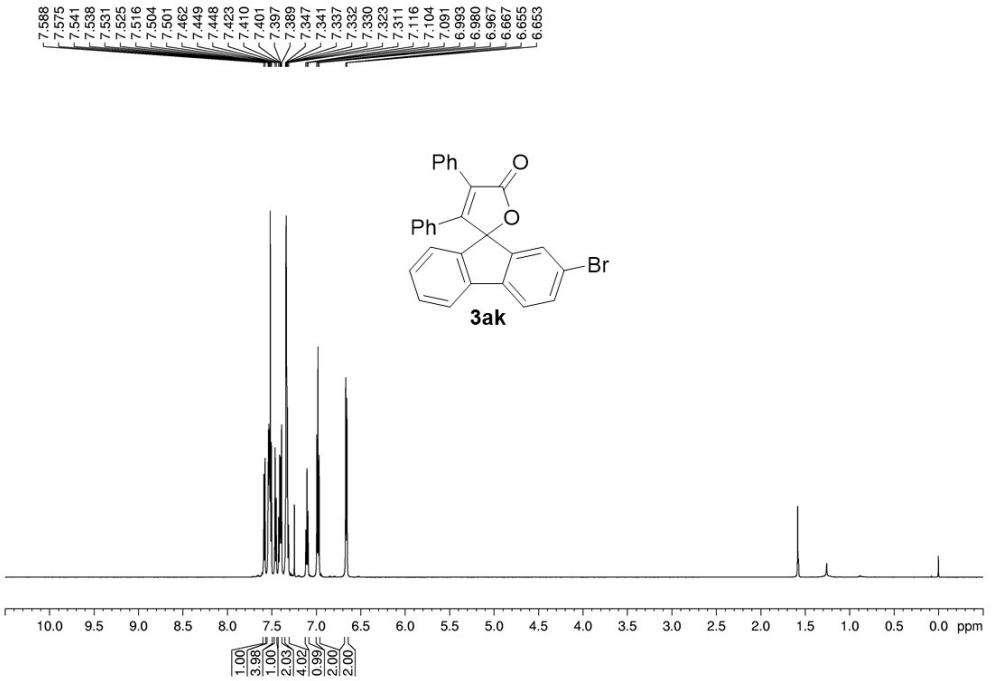



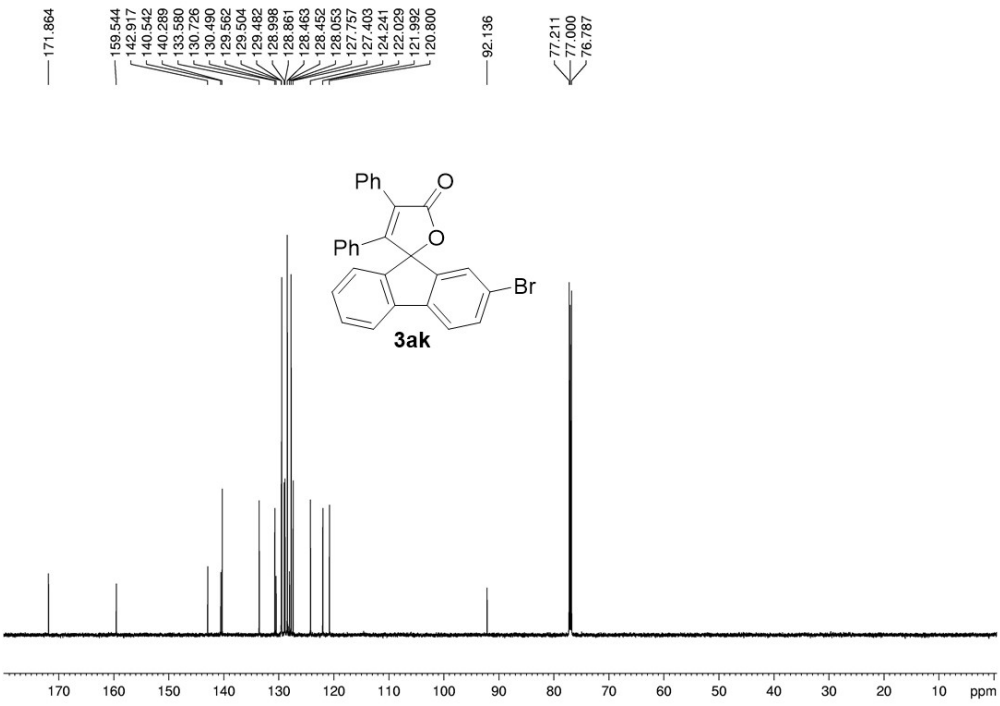

$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3aj**



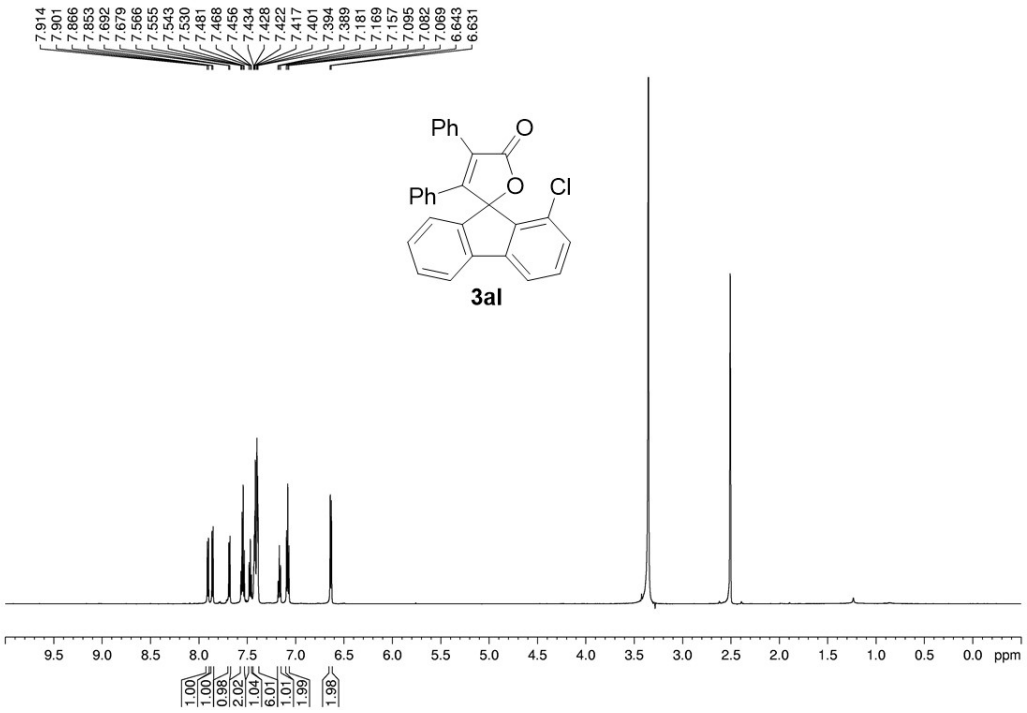



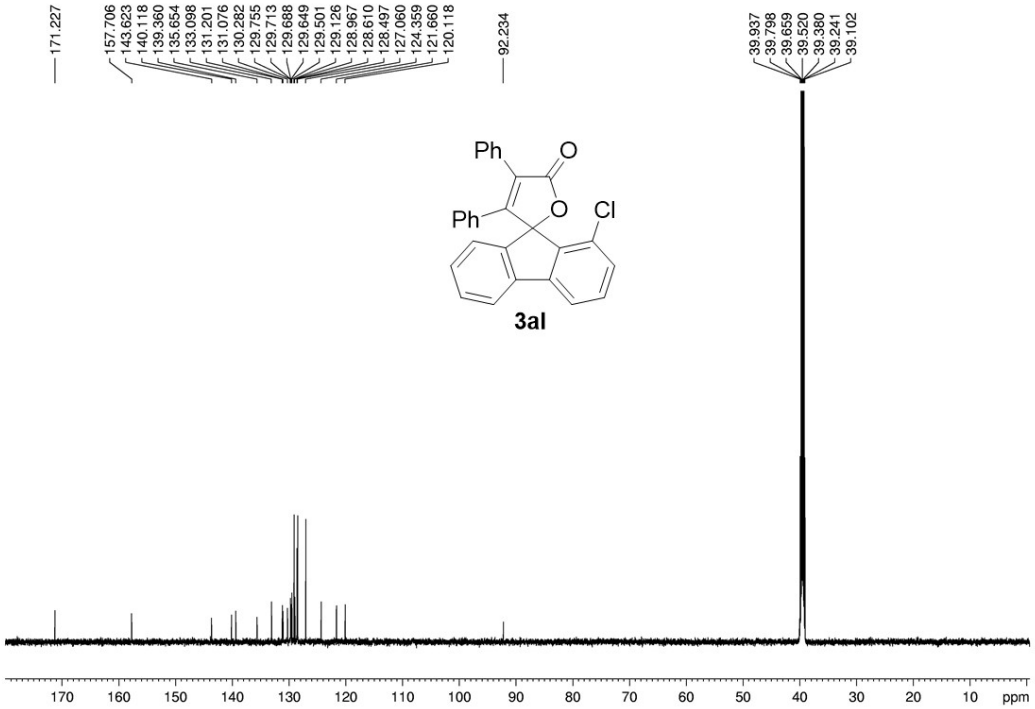

$^{1}H$ NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3ak**



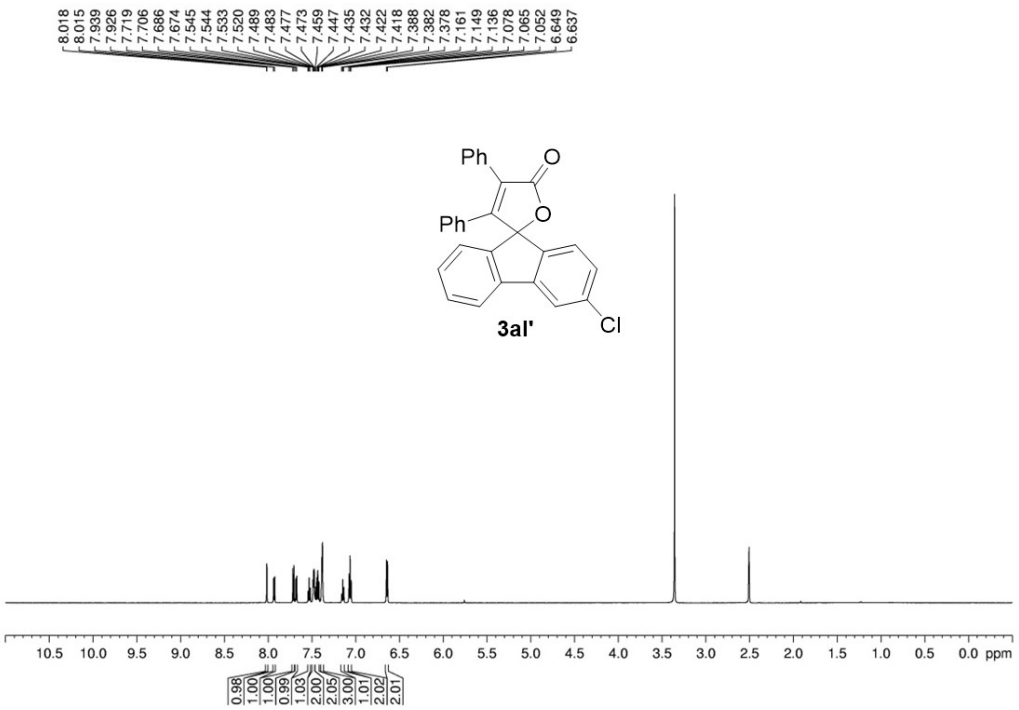



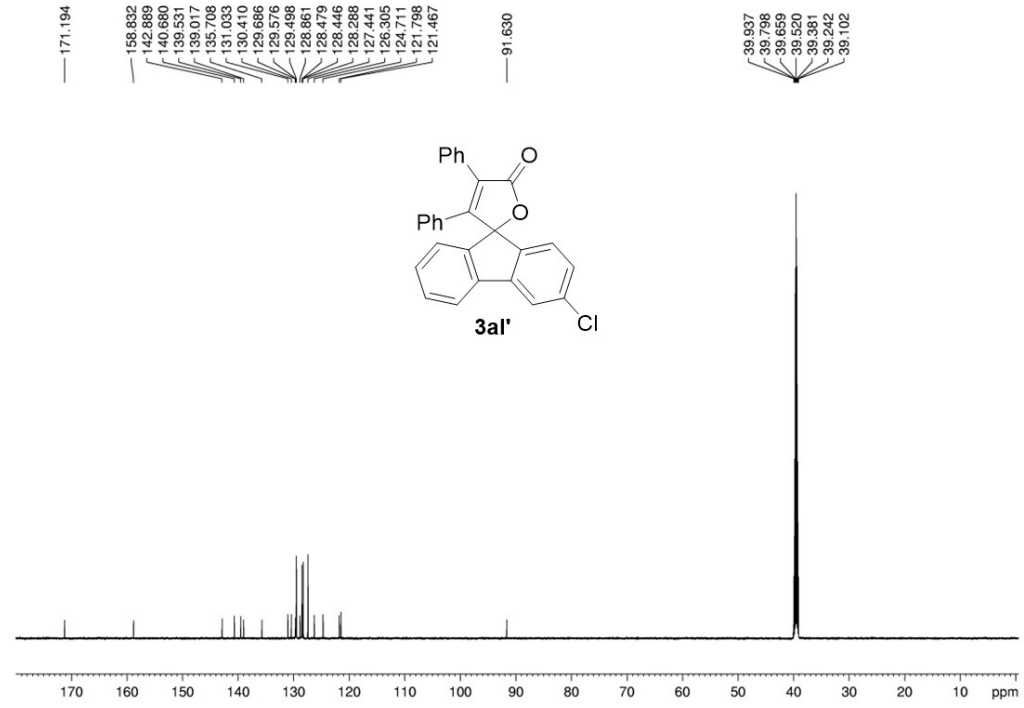

$^{1}$H NMR (600 MHz, DMSO) and $^{13}$C{$^{1}$H} NMR (150 MHz, DMSO) of **3al**



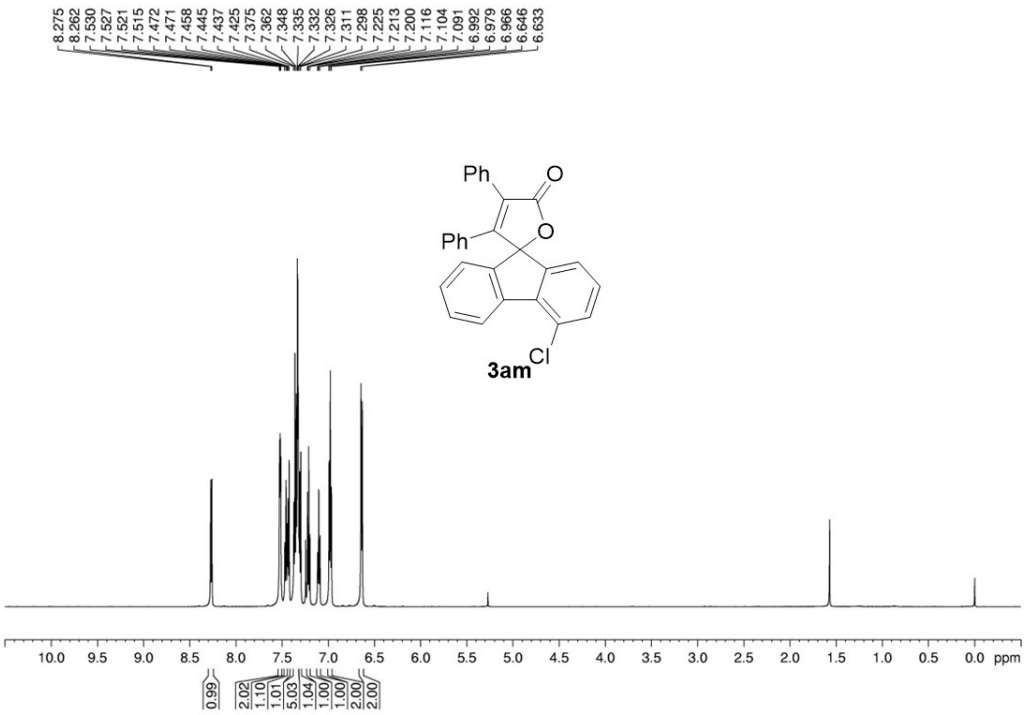



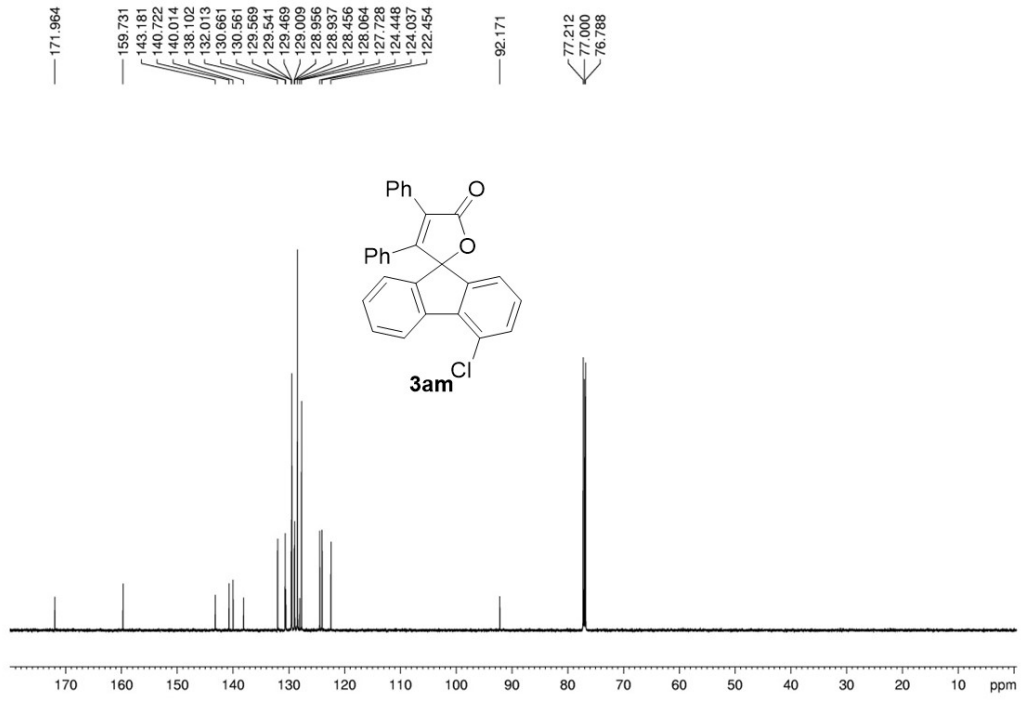

$^{1}$H NMR (600 MHz, DMSO) and $^{13}$C{$^{1}$H} NMR (150 MHz, DMSO) of **3al'**



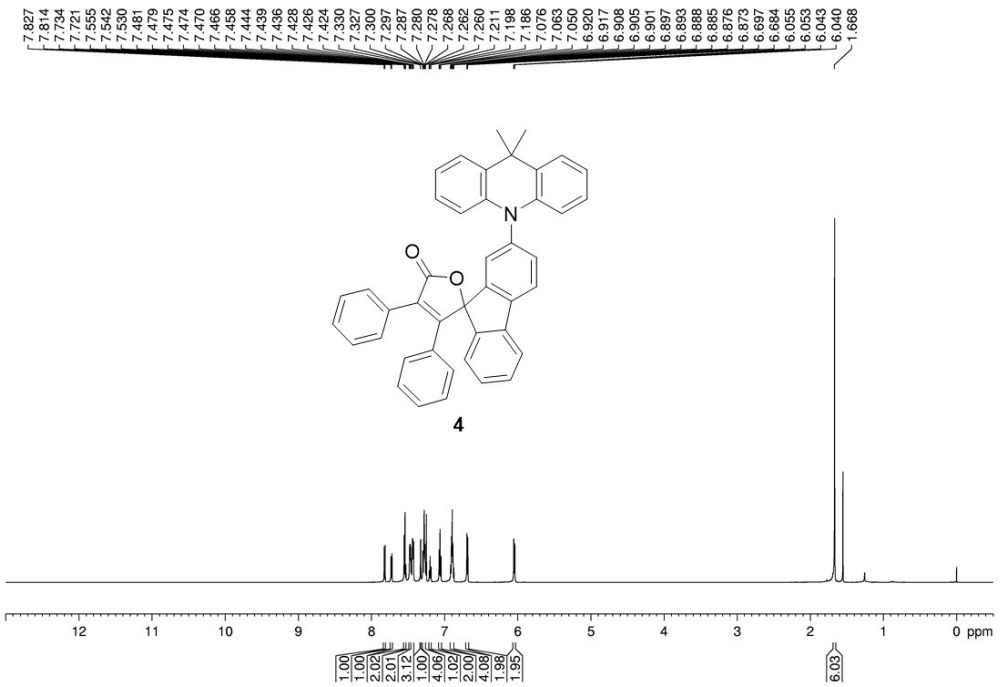



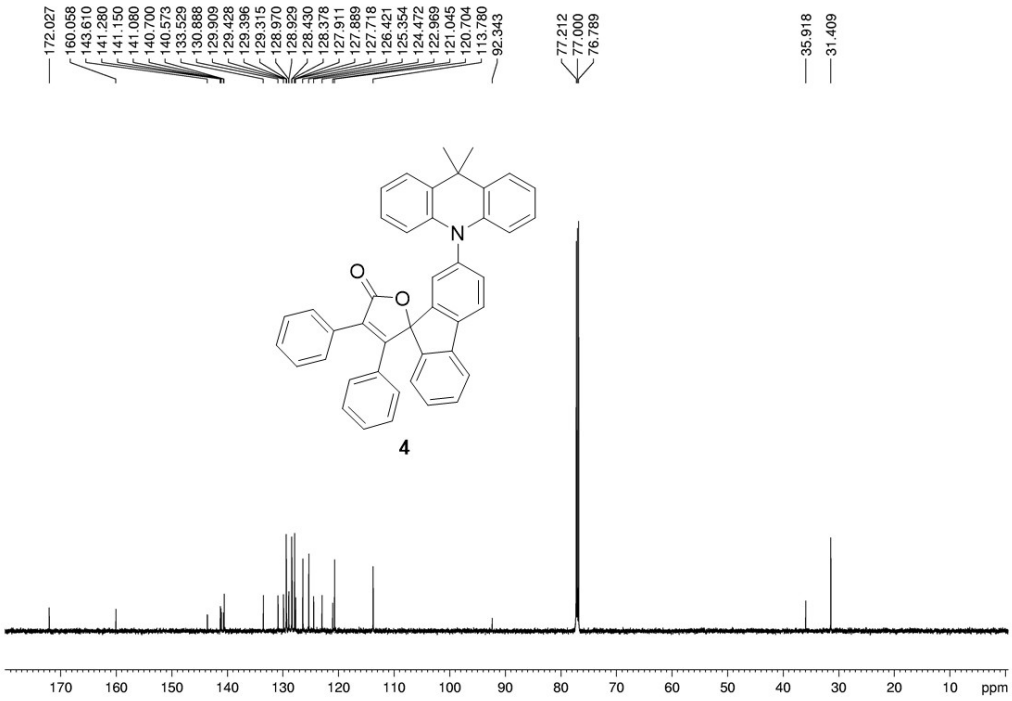

$^{1}H$ NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **3am**



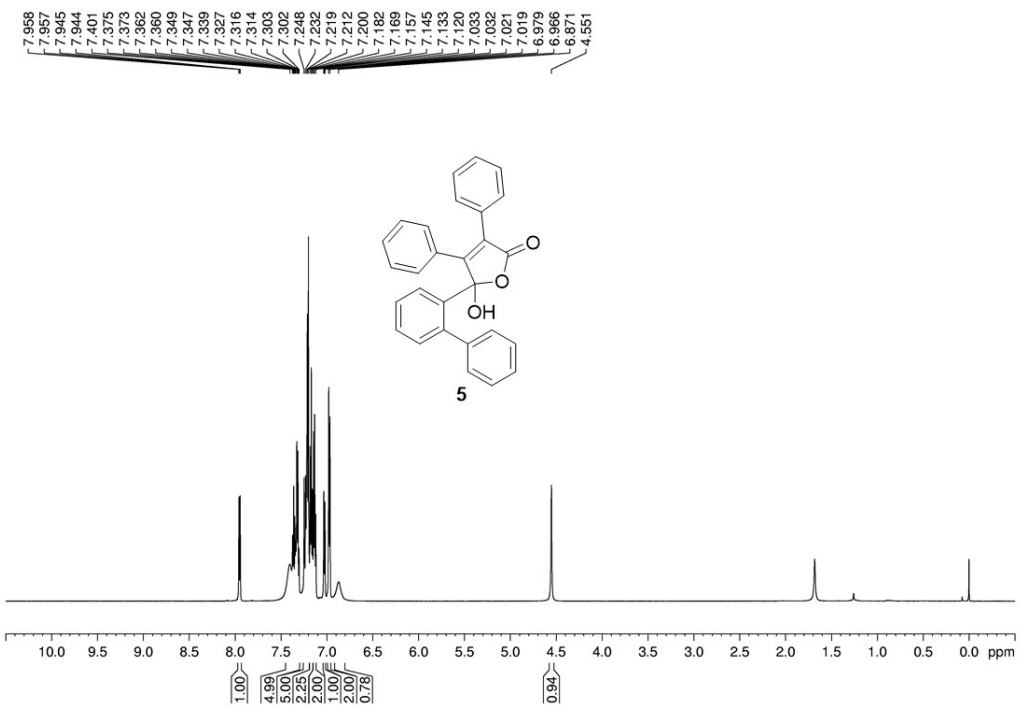




$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **4**




$^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$) of **5**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6.871
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O
OH
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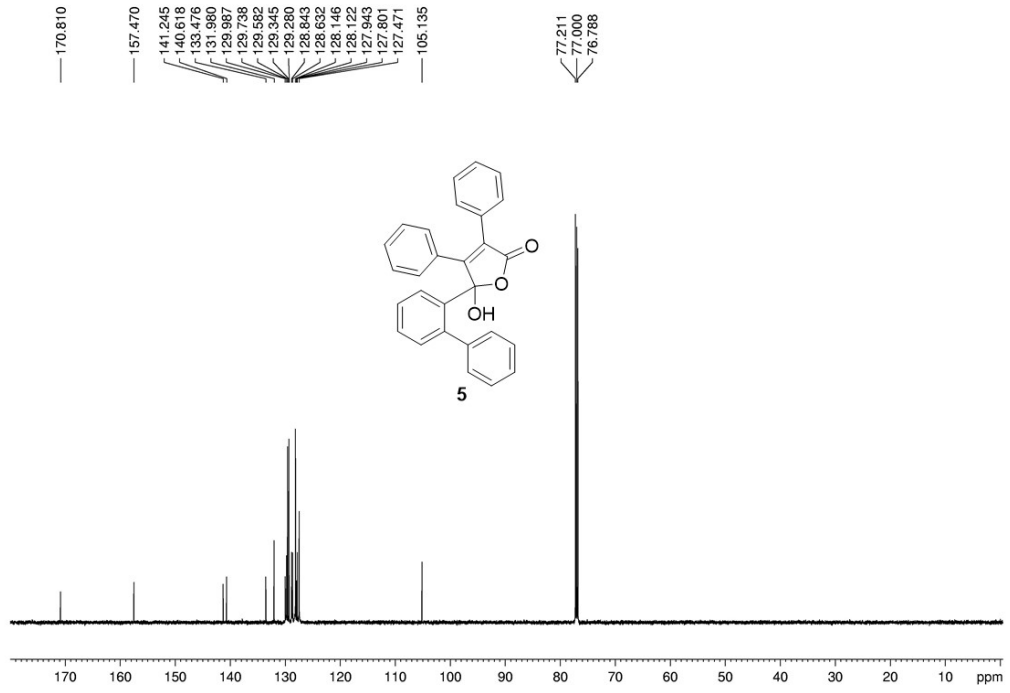
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129.345
129.280
128.843
128.632
128.146
128.122
127.943
127.801
127.471
105.135
77.211
77.000
76.788
O
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OH
5
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm