Electronic Supplementary Material (ESI) for ChemComm.

Supporting information for….

3-Nitro-Coumarins Synthesis *via* Nitrative Cyclization of Aryl Alkynoates using *tert*-Butyl Nitrite

CONTENTS

EXPERIMENTAL SECTION

Instrumentation and Chemicals: Column chromatographic purifications of the compounds were performed using silica gel (mesh 100–200 or mesh 230–400) and hexane – ethyl acetate mixtures as eluent unless otherwise specified. Solvents were commercially available and used without further purification. NMR spectra were recorded on a 400 MHz instrument at 25 °C. The chemical shift values are reported in parts per million (ppm) with respect to residual trichloromethane (7.26 ppm for ^{1}H and 77.16 ppm for ^{13}C). The peak patterns are designated as follows: s: singlet; d: doublet; t: triplet; q: quartet; m: multiplet; dd: doublet of doublets; td: triplet of doublets; br s: broad singlet. The coupling constants (*J*) are reported in hertz (Hz). High-resolution mass spectra (HR-MS) were recorded on an ESI-TOF (time of flight) mass spectrometer. Infrared spectral data are reported in wave number (cm^{-1}). FT-IR spectra were recorded after making thin layer of the compounds on the surface of KBr crystal using dichloromethane. Melting points of the compounds were determined using a digital melting point apparatus and are uncorrected.

Crystallographic Data collection

The crystals data were collected with Bruker SMART D8 goniometer equipped with an APEX CCD detector and with an INCOATEC micro source (Mo-Kα radiation, λ = 0.71073 Å). SAINT+[1] and SADABS[2] were used to integrate the intensities and to correct the absorption respectively The structure was resolved by direct methods and refined on F^2 with SHELXL-97.[3] Good quality of crystals for the compound **2f** and **2u** were obtained by the slow evaporation of chloroform. ORTEP drawing of the compounds show ellipsoid contour at the 50% probability level.

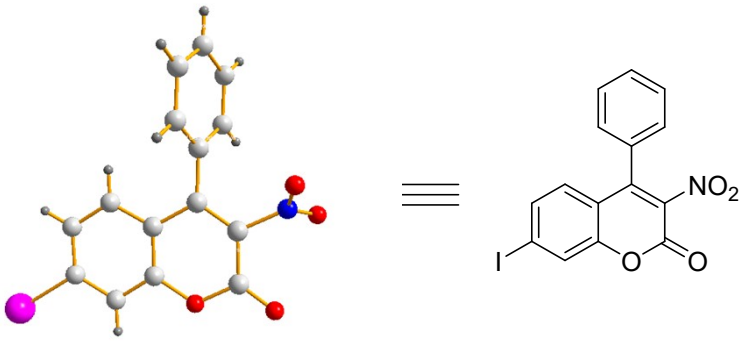


Fig. S1. Crystal structure of **2f**.

Table S1. Crystal data and structure refinement for 2f.

CCDC number	2064075
Empirical formula	$C_{15}H_8INO_4$
Formula weight	394.63
Temperature/K	100.00(10)
Crystal system	monoclinic
Space group	*P*21/c
a/Å	10.34430(10)
b/Å	15.20400(10)
c/Å	8.99570(10)
α/°	90
β/°	100.7960(10)
γ/°	90
Volume/Å^3	1389.75(2)
Z	4
ρ_{calc}g/cm^3	1.886
μ/mm^{-1}	18.249
F(000)	763
Crystal size/mm^3	0.20 × 0.20 ×0.18
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	8.702 to 150.378
Index ranges	-12 ≤ h ≤ 12, -14 ≤ k ≤ 19, -11 ≤ l ≤

	10
Reflections collected	20661
Independent reflections	2838 [R_{int} = 0.0582, R_{sigma} = 0.0235]
Data/restraints/parameters	2838/0/190
Goodness-of-fit on F^2	1.059
Final R indexes [I>=2σ (I)]	R_1 = 0.0303, wR_2 = 0.0821
Final R indexes [all data]	R_1 = 0.0305, wR_2 = 0.0823
Largest diff. peak/hole / e $Å^{-3}$	0.96/-1.82

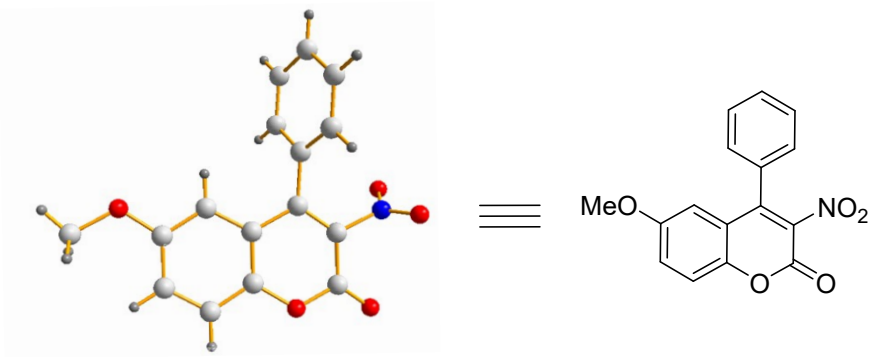


Fig. S2. Crystal structure of **2s**.

Table S2. Crystal data and structure refinement for 2s.

CCDC number	2076536
Empirical formula	$C_{16}H_{11}NO_5$
Formula weight	297.26
Temperature/K	299.3(6)
Crystal system	orthorhombic
Space group	*P*na2_1
a/Å	7.91770(10)
b/Å	16.29580(10)
c/Å	10.82590(10)
α/°	90
β/°	90

γ/°	90
Volume/Å^3	1396.81(2)
Z	4
ρ_{calc}g/cm^3	1.414
μ/mm^{-1}	0.899
F(000)	616
Crystal size/mm^3	0.20 × 0.20 ×0.18
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	4.8770 to 150.094
Index ranges	-8 ≤ h ≤ 9, -20 ≤ k ≤ 20, -13 ≤ l ≤ 13
Reflections collected	37735
Independent reflections	2873 [R_{int} = 0.0561, R_{sigma} = 0.0203]
Data/restraints/parameters	2873/1/200
Goodness-of-fit on F^2	1.072
Final R indexes [I>=2σ (I)]	R_1 = 0.0335, wR_2 = 0.0885
Final R indexes [all data]	R_1 = 0.0353, wR_2 = 0.0901
Largest diff. peak/hole / e Å^{-3}	0.10/-0.13

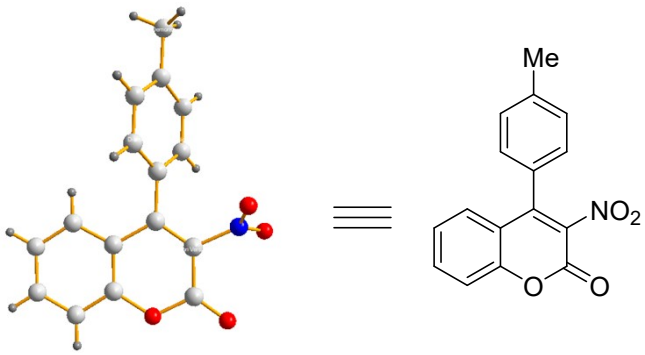


Fig. S3. Crystal structure of **2u**.

Table S3. Crystal data and structure refinement for 2u.

CCDC number	2064074
Empirical formula	$C_{16}H_{11}NO_4$
Formula weight	281.26
Temperature/K	298.84(10)
Crystal system	triclinic
Space group	*P*-1
a/Å	8.2983(8)
b/Å	9.1387(8)
c/Å	9.6145(7)
α/°	73.544(7)
β/°	72.349(8)
γ/°	82.099(8)
Volume/Å^3	665.27(11)
Z	2
ρ_{calc}g/cm^3	1.404
μ/mm^{-1}	0.852
F(000)	292
Crystal size/mm^3	0.25 × 0.21 ×0.20
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	9.988 to 150.750
Index ranges	$-10 \le h \le 10$, $-10 \le k \le 11$, $-12 \le l \le 11$
Reflections collected	9577
Independent reflections	2670 [R_{int} = 0.0479, R_{sigma} = 0.0245]
Data/restraints/parameters	2670/0/191
Goodness-of-fit on F^2	1.052
Final R indexes [I>=2σ (I)]	R_1 = 0.0708, wR_2 = 0.2122
Final R indexes [all data]	R_1 = 0.0808, wR_2 = 0.2247
Largest diff. peak/hole / e Å^{-3}	0.35/-0.29

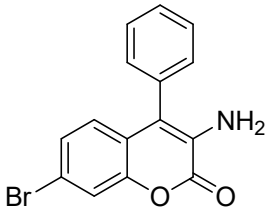
Synthesis and characterization Data

Synthetic procedure for preparation of 7-Methyl-3-nitro-4-phenyl-2H-chromen-2-one (2a). To an oven dried sealed tube charged with a magnetic stirring bar, *p*-Tolyl 3-phenylpropiolate (81 mg, 0.34 mmol, 1.0 equiv) and 3 mL DMSO were taken. The tube was fitted with a rubber septum and then it was charged with dioxygen. After that TBN (162 µL, 1.37 mmol, 4.0 equiv) was added to it in an oxygen atmosphere, the septum was then replaced by a Teflon screwcap under oxygen flow. The reaction mixture was allowed to stir at 120 ^{o}C in a preheated oil bath for 36 h. After that the reaction mixture was cooled at room temperature and washed with water. The organic layer was extracted with dichloromethane and were dried over Na_2SO_4, and concentrated under reduced pressure. The crude reaction mixture was purified by silica gel column chromatography using 5% ethyl acetate/hexane. Purification through column chromatography (in 5% ethyl acetate/hexane) yielded 67 mg of 2a (70%).

Gram scale synthesis. To an oven dried sealed tube charged with a magnetic stirring bar, 4-(*tert*-Butyl)phenyl 3-phenylpropiolate (2.0 g, 7.18 mmol, 1.0 equiv) and 25 mL DMSO were taken. The tube was fitted with a rubber septum and then it was charged with dioxygen. After that TBN (3.4 mL, 28.7 mmol, 4.0 equiv) was added to it in an oxygen atmosphere, the septum was then replaced by a Teflon screwcap under oxygen flow. The reaction mixture was allowed to stir at 120 ^{o}C in a preheated oil bath for 36 h. After that the reaction mixture was cooled at room temperature and washed with water. The organic layer was extracted with dichloromethane and were dried over Na_2SO_4, and concentrated under reduced pressure. The crude reaction mixture was purified by silica gel column chromatography using 5% ethyl acetate/hexane. Purification through column chromatography (in 5% ethyl acetate/hexane) yielded 1.37 g of **2b** (59%)

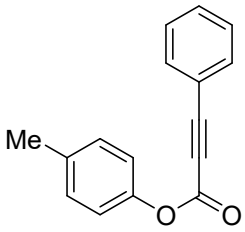
Procedure for the radical scavenger experiment with 2,2,6,6-Tetramethylpiperidin-1-yl)oxyl radical (TEMPO). To an oven dried sealed tube charged with a magnetic stirring bar, *p*-Tolyl 3-phenylpropiolate (86.5 mg, 0.37 mmol, 1.0 equiv), TEMPO (228 mg, 1.46 mmol, 3.0 equiv) and 3 mL DMSO were taken. The tube was fitted with a rubber septum and then it was charged with dioxygen. After that TBN (175 μL, 1.47 mmol, 4.0 equiv) was added to it in an oxygen atmosphere, the septum was then replaced by a Teflon screwcap under oxygen flow. The reaction mixture was allowed to stir at 120 °C in a preheated oil bath for 36 h. After 36 h reaction formation of product 2a was not observed.

Synthetic procedure of 3-Amino-7-bromo-4-phenyl-2H-chromen-2-one (7). To an oven dried sealed tube charged with a magnetic stirring bar, 7-Bromo-3-nitro-4-phenyl-2H-chromen-2-one (64.4 mg, 0.186 mmol, 1.0 equiv) was taken and dissolved in EtOH and H_2O (8:2 v/v). To it Fe powder (83.0 mg, 1.488 mmol, 8 equiv), NH_4Cl (12.0 mg, 0.223 mmol, 1.2 equiv) and the resulting mixture was stirred in a preheated oil bath at 90 °C, for 6 h.[4] Completion of reaction was confirmed by TLC and the reaction mixture was cooled at room temperature and washed with water. The organic layer was extracted with ethyl acetate and were dried over Na_2SO_4, and concentrated under reduced pressure. The crude reaction mixture was purified by silica gel column chromatography using 5% ethyl acetate/hexane as eluent to get 3-Amino-7-bromo-4-phenyl-2H-chromen-2-one as off white solid. R_f = 0.4 (10% ethyl acetate/hexane); off white solid; yield 48 mg (80%); mp: 138-140 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59-7.56 (m, 2H), 7.51 – 7.47 (m, 2H), 7.35 – 7.33 (m, 2H), 7.22 (dd, *J* = 8.4, 1.6 Hz, 1H), 6.86 (d, *J* = 8.4 Hz, 1H), 4.23 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.8, 148.7, 132.7, 129.9, 129.3, 129.2, 129.2, 127.8, 125.4, 121.6, 121.1, 119.7, 119.5; HRMS (ESI-TOF) calcd for $C_{15}H_{11}BrNO_2$ $[M+H]^+$ 315.9968, found 315.9928.

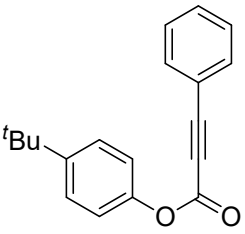


Synthesis of starting materials. Starting materials were prepared from literature known procedure.[5]

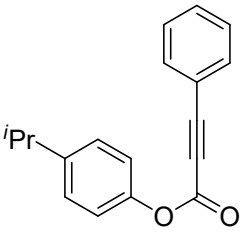
***p*-Tolyl 3-phenylpropiolate (1a).[5]** R_f = 0.6 (5% ethyl acetate/hexane); white solid; yield 677 mg (69%); mp: 63-65 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 7.2 Hz, 2H), 7.49 (t, *J* = 7.6 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.22 (d, *J* = 8.8 Hz, 2H), 7.10 – 7.08 (m, 2H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.7, 148.0, 136.3, 133.3, 131.1, 130.2, 128.8, 121.2, 119.4, 88.6, 80.4, 21.0; IR (KBr) ῡ 3004, 2234, 1722, 750 cm^{-1}.



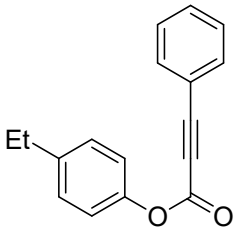
4-(*tert*-Butyl)phenyl 3-phenylpropiolate (1b).[5] R_f = 0.8 (5% ethyl acetate/hexane); white solid; yield 750 mg (60%); mp: 88-90 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 – 7.62 (m, 2H), 7.51 – 7.47 (m, 1H), 7.45 – 7.39 (m, 4H), 7.13 – 7.11 (m, 2H), 1.34 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.7, 149.4, 147.9, 133.3, 131.1, 128.8, 126.6, 120.9, 119.4, 88.7, 80.5, 34.7, 31.5; IR (KBr) ῡ 2236, 1722, 1604, 737 cm^{-1}.



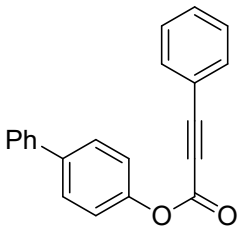
4-Isopropylphenyl 3-phenylpropiolate (1c).[5] R_f = 0.8 (5% ethyl acetate/hexane); yellowish white solid; yield 650 mg (66%); mp: 66-68 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 – 7.61 (m, 2H), 7.52 – 7.47 (m, 1H), 7.42 – 7.38 (m, 2H), 7.27 (d, *J* = 7.2 Hz, 2H), 7.12 – 7.10 (m, 2H), 2.93 (h, *J* = 6.8 Hz, 1H), 1.26 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.8, 148.2, 147.2, 133.3, 131.1, 128.8, 127.6, 121.3, 119.5, 88.7, 80.5, 33.8, 24.2; IR (KBr) ῡ 2235, 1723, 1603, 754 cm^{-1}.



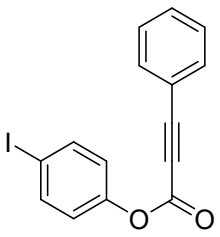
4-Ethylphenyl 3-phenylpropiolate (1d).[5] R_f = 0.7 (5% ethyl acetate/hexane); yellowish white solid; yield 585 mg (55%); mp: 48-50 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 7.2 Hz, 2H), 7.48 (t, *J* = 6.8 Hz, 1H), 7.41 (t, *J* = 6.8 Hz, 2H), 7.25 (d, *J* = 7.2 Hz, 2H), 7.12 (d, *J* = 7.6 Hz, 2H), 2.68 (q, *J* = 7.2 Hz, 2H), 1.26 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.7, 148.1, 142.5, 133.3, 131.1, 129.0, 128.8, 121.3, 119.4, 88.6, 80.4, 28.4, 15.6; IR (KBr) ῡ 2237, 1721, 1635, 758 cm^{-1}.



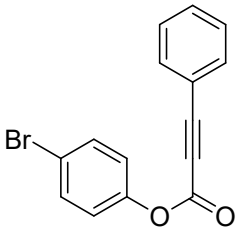
[1,1'-Biphenyl]-4-yl 3-phenylpropiolate (1e).[5] R_f = 0.6 (5% ethyl acetate/hexane); yellowish white solid; yield 560 mg (68%); mp: 140-142 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (t, *J* = 7.6 Hz, 4H), 7.58 (d, *J* = 7.6 Hz, 2H), 7.52 – 7.35 (m, 6H), 7.29 –7.26 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.6, 149.6, 140.3, 139.7, 133.4, 131.2, 129.0, 128.8, 128.5, 127.6, 127.3, 121.9, 119.4, 89.0, 80.4; IR (KBr) ῡ 2219, 1726, 1644, 760 cm^{-1}.



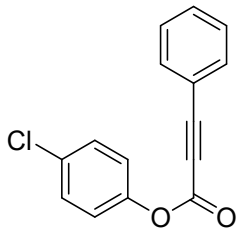
4-Iodophenyl 3-phenylpropiolate (1f).[5] R_f = 0.7 (5% ethyl acetate/hexane); white solid; yield 698 mg (65%); mp: 81-83 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 – 7.71 (m, 2H), 7.65 – 7.62 (m, 2H), 7.52 – 7.48 (m, 1H), 7.43 – 7.39 (m, 2H), 7.00 – 6.96 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.0, 150.1, 138.8, 133.4, 131.3, 128.8, 123.7, 119.2, 90.7, 89.3, 80.1; IR (KBr) ῡ 2305, 1726, 1605, 770 cm^{-1}.



4-Bromophenyl 3-phenylpropiolate (1g).[5] R_f = 0.7 (5% ethyl acetate/hexane); white solid; yield 670 mg (68%); mp: 68-70 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 –

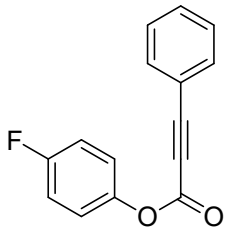


7.62 (m, 2H), 7.55 – 7.48 (m, 3H), 7.43 – 7.39 (m, 2H), 7.12 – 7.08 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.0, 149.2, 133.3, 132.7, 131.3, 128.8, 123.4, 119.7, 119.1, 89.3, 80.1; IR (KBr) ῡ 2235, 1726, 1605, 701 cm^{-1}.

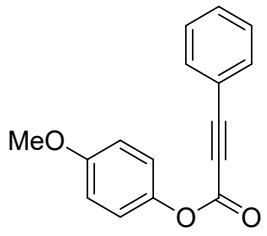


4-Chlorophenyl 3-phenylpropiolate (1h).[5] R_f = 0.7 (5% ethyl acetate/hexane); white solid; yield 850 mg (58%); mp: 63-65 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 – 7.63 (m, 2H), 7.52 – 7.48 (m, 1H) 7.43 – 7.36 (m, 4H), 7.15 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.1, 148.7, 133.4, 132.0, 131.3, 129.8, 128.9, 123.0, 119.2, 89.3, 80.1; IR (KBr) ῡ 2305, 1727, 1604, 768 cm^{-1}.

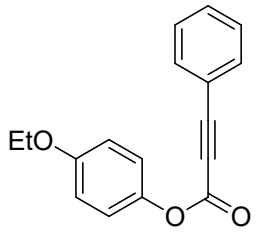
4-Fluorophenyl 3-phenylpropiolate (1i).[5] R_f = 0.6 (5% ethyl acetate/hexane); white solid; yield 620 mg (62%); mp: 54-56 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.2 Hz, 2H), 7.50 (t, *J* = 7.6 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.16 (s, 2H), 7.10 (t, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.6 (d, $^{1}J_{C\text{-}F}$ = 243.7 Hz), 152.4, 146.0 (d, $^{4}J_{C\text{-}F}$ = 2.9 Hz), 133.3, 131.3, 128.8, 123.1 (d, $^{3}J_{C\text{-}F}$ = 8.6 Hz), 119.2, 116.4 ($^{2}J_{C\text{-}F}$ = 23.5 Hz), 89.1, 80.1; IR (KBr) ῡ 2210, 1724, 1644, 741 cm^{-1}.



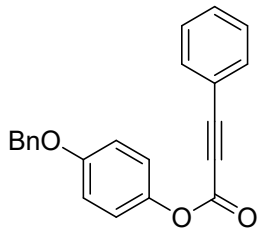
4-Methoxyphenyl 3-phenylpropiolate (1j).[5] R_f = 0.5 (5% ethyl acetate/hexane); yellow solid; yield 500 mg (55%); mp: 80-82 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.2 Hz, 2H), 7.49 (t, *J* = 7.2 Hz, 1H), 7.40 (t, *J* = 7.2 Hz, 2H), 7.12 (d, *J* = 8.8 Hz, 2H), 6.93 (d, *J* = 8.8 Hz, 2H), 3.81 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.7, 152.9, 143.7, 133.3, 131.1, 128.8, 122.3, 119.4, 114.7, 88.7, 80.4, 55.7; IR (KBr) ῡ 2235, 1724, 1639, 745 cm^{-1}.



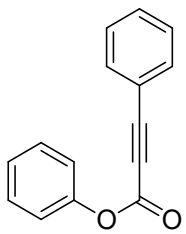
4-Ethoxyphenyl 3-phenylpropiolate (1k).[6] R_f = 0.5 (5% ethyl acetate/hexane); yellowish white solid; yield 566 mg (59%); mp: 80-82 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.2 Hz, 2H), 7.49 (t, *J* = 7.2 Hz, 1H), 7.40 (t, *J* = 7.2 Hz, 2H), 7.10 (d, *J* = 8.8 Hz, 2H), 6.91 (d, *J* = 8.4 Hz, 2H), 4.03 (q, *J* = 6.8 Hz, 2H), 1.42 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.2, 153.0, 143.6, 133.3, 131.1, 128.8, 122.3, 119.4, 115.2, 88.7, 80.4, 64.0, 14.9; IR (KBr) ῡ 2232, 1723, 1644, 769 cm^{-1}.



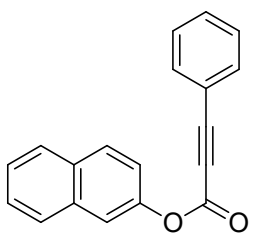
4-(Benzyloxy)phenyl 3-phenylpropiolate (1l).[7] R_f = 0.4 (5% ethyl acetate/hexane); yellowish white solid; yield 540 mg (58%); mp: 118-120 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (dd, *J* = 8.8, 1.6 Hz, 2H), 7.52 – 7.33 (m, 8H), 7.15 – 7.11 (m, 2H), 7.03 – 6.99 (m, 2H), 5.07 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.0, 152.9, 143.9, 136.8, 133.3, 131.1, 128.8, 128.8, 128.2, 127.6, 122.4, 119.4, 115.7, 88.8, 80.4, 70.5.



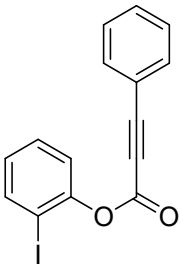
Phenyl 3-phenylpropiolate (1m).[5] R_f = 0.8 (5% ethyl acetate/hexane); white solid; yield 840 mg (72%); mp: 46-48 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 – 7.62 (m, 2H), 7.52 – 7.47 (m, 1H), 7.45 – 7.39 (m, 4H), 7.31 – 7.27 (m, 1H), 7.22 – 7.20 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.5, 150.3, 133.3, 131.2, 129.7, 128.8, 126.5, 121.6, 119.4, 88.8, 80.4; IR (KBr) ῡ 2116, 1723, 1590, 731 cm^{-1}.



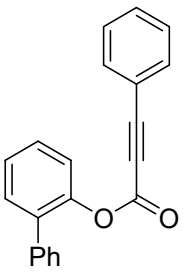
Naphthalen-2-yl 3-phenylpropiolate (1n).[8] R_f = 0.7 (5% ethyl acetate/hexane); yellowish white solid; yield 650 mg (66%); mp: 94-96 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 – 7.84 (m, 3H), 7.70 (d, *J* = 2.4 Hz, 1H), 7.67 – 7.65 (m,



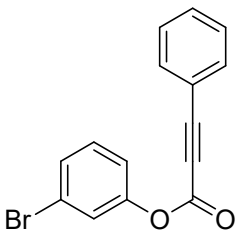
2H), 7.56 – 7.48 (m, 3H), 7.44 – 7.40 (m, 2H), 7.35 (dd, *J* = 8.8, 2.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.6, 147.8, 133.8, 133.3, 131.8, 131.2, 129.8, 128.8, 127.9, 127.9, 126.9, 126.2, 120.8, 119.32, 118.7, 89.0, 80.4; IR (KBr) ῡ 2230, 1720, 1630, 748 cm^{-1}.



2-Iodophenyl 3-phenylpropiolate (1o).[9] R_f = 0.5 (5% ethyl acetate/hexane); yellowish white solid; yield 600 mg (67%); mp: 85-87 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.67-7.65 (m, 2H), 7.52-7.48 (m, 1H), 7.44 – 7.39 (m, 3H), 7.20 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.05-7.01 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.6, 150.8, 139.9, 133.5, 131.3, 129.7, 128.8, 128.3, 123.2, 119.3, 90.3, 89.6, 80.3; IR (KBr) ῡ 2233, 1727, 1490, 739 cm^{-1}.

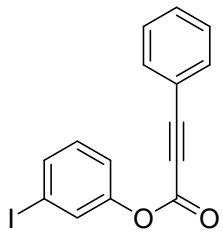


[1,1'-Biphenyl]-2-yl 3-phenylpropiolate (1p).[10] R_f = 0.7 (5% ethyl acetate/hexane); yellowish white solid; yield 549 mg (62%); mp: 93-95 °C; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.55 (d, *J* = 7.7 Hz, 2H), 7.48 – 7.40 (m, 7H), 7.39-7.35 (m, 4H), 7.24 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.5, 147.3, 137.2, 135.1, 133.3, 131.3, 131.1, 129.1, 128.8, 128.7, 128.6, 127.8, 127.1, 122.9, 119.4, 88.8, 80.2; IR (KBr) ῡ 3052, 2205, 1723, 748 cm^{-1}.

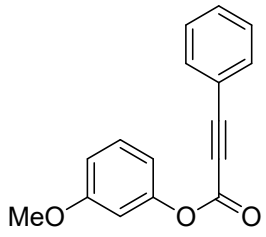


3-Bromophenyl 3-phenylpropiolate (1q).[11] R_f = 0.7 (5% ethyl acetate/hexane); white solid; yield 652 mg (51%); mp: 54-56 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 7.2 Hz, 2H), 7.50 (t, *J* = 7.2 Hz, 1H), 7.44 – 7.40 (m, 4H), 7.30 (d, *J* = 8.0 Hz, 1H), 7.16 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.9, 150.6, 133.4, 131.4, 130.8, 129.7, 128.8, 125.1, 122.6, 120.5, 119.2, 89.4, 80.0;. IR (KBr) ῡ 3053, 2211, 1726, 1585, 737 cm^{-1}.

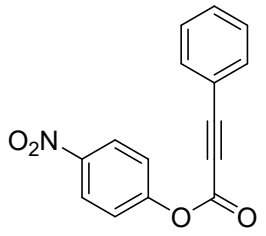
3-Iodophenyl 3-phenylpropiolate (1r).[12] R_f = 0.6 (5% ethyl acetate/hexane); white solid; yield 422 mg (62%); mp: 61-63 °C; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.64-7.61 (m, 3H), 7.58 (s, 1H), 7.50 (t, *J* = 7.7 Hz, 1H), 7.41 (t, *J* = 7.7 Hz, 2H), 7.20 – 7.19 (m, 1H), 7.15-7.13 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.0, 150.4, 135.7, 133.4, 131.4, 131.0, 130.8, 128.9, 121.2, 119.2, 93.7, 89.4, 80.0; IR (KBr) ῡ 2237, 1724, 1638, 748 cm^{-1}.



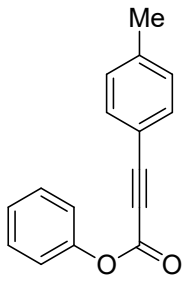
3-Methoxyphenyl 3-phenylpropiolate (1s).[11] R_f = 0.6 (5% ethyl acetate/hexane); colorless liquid; yield 729 mg (61%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 – 7.61 (m, 2H), 7.52 – 7.46 (m, 1H), 7.41 (t, *J* = 7.4 Hz, 2H), 7.32 (t, *J* = 8.2 Hz, 1H), 6.86 – 6.78 (m, 2H), 6.75 (t, *J* = 2.2 Hz, 1H), 3.82 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.7, 152.4, 151.2, 133.3, 131.2, 130.1, 128.8, 119.4, 113.8, 112.4, 107.6, 88.8, 80.4, 55.6; IR (KBr) ῡ 2216, 1723, 1610, 763 cm^{-1}.



4-Nitrophenyl 3-phenylpropiolate (1t).[11] R_f = 0.4 (5% ethyl acetate/hexane); yellowish white solid; yield 652 mg (63%); mp: 96-98 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (d, *J* = 9.2 Hz, 2H), 7.67 – 7.65 (m, 2H), 7.55 – 7.51 (m, 1H), 7.45 – 7.38 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 154.8, 151.2, 145.8, 133.5, 131.6, 128.9, 125.5, 122.5, 118.9, 90.2, 79.7; IR (KBr) ῡ 2304, 1732, 1616, 764 cm^{-1}.

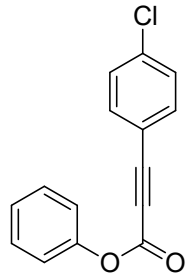


Phenyl 3-(*p*-tolyl)propiolate (1u).[13] R_f = 0.6 (5% ethyl acetate/hexane); yellow solid; yield 315 mg (51%); mp: 79-81 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.0 Hz, 2H), 7.44 – 7.40 (m, 2H), 7.29 (d, *J* = 7.2 Hz, 1H), 7.21 (t, *J* = 7.4 Hz,

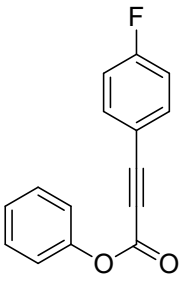


4H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.6, 150.3, 142.0, 133.3, 129.7, 129.6, 126.5, 121.6, 116.2, 89.5, 80.1, 21.9; IR (KBr) ῡ 2236, 1723, 1636, 779 cm^{-1}.

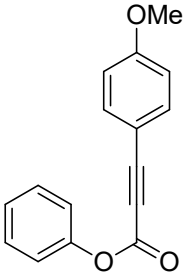
Phenyl 3-(4-chlorophenyl)propiolate (1v).[13] R_f = 0.8 (5% ethyl acetate/hexane); yellowish white solid; yield 249 mg (55%); mp: 94-96 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 – 7.55 (m, 2H), 7.45 – 7.38 (m, 4H), 7.31 – 7.28 (m, 1H), 7.20 – 7.18 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.3, 150.2, 137.7, 134.5, 129.8, 129.3, 126.6, 121.6, 117.9, 87.5, 81.2; IR (KBr) ῡ 2242, 1730, 1650, 758 cm^{-1}.



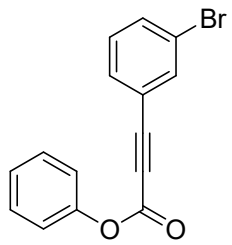
Phenyl 3-(4-fluorophenyl)propiolate (1w).[13] R_f = 0.6 (5% ethyl acetate/hexane); white solid; yield 398 mg (51%); mp: 80-82 ^{o}C; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.66 – 7.60 (m, 2H), 7.42 (t, J = 7.7 Hz, 2H), 7.28 (t, J = 7.7 Hz, 1H), 7.19 (d, J = 8.4 Hz, 2H), 7.11 (t, J = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.3 (d, $^{1}J_{C\text{-}F}$ = 252.4 Hz), 152.4, 150.2, 135.7 (d, $^{3}J_{C\text{-}F}$ = 8.9 Hz), 129.8, 126.6, 121.6, 116.4 (d, $^{2}J_{C\text{-}F}$ = 22.2 Hz), 115.5 (d, $^{4}J_{C\text{-}F}$ = 3.5 Hz), 87.8, 80.3; IR (KBr) ῡ 2214, 1726, 1644, 752 cm^{-1}.



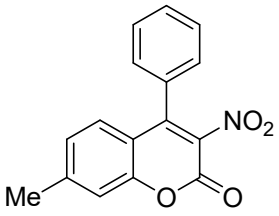
Phenyl 3-(4-methoxyphenyl)propiolate (1x).[13] R_f = 0.6 (5% ethyl acetate/hexane); yellowish white solid; yield 527 mg (44%); mp: 70-72 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 – 7.56 (m, 2H), 7.44 – 7.39 (m, 2H), 7.30 – 7.27 (m, 1H), 7.21 – 7.18 (m, 2H), 6.93 – 6.89 (m, 2H), 3.85 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.0 , 152.8, 150.4, 135.4, 129.7, 126.5, 121.7, 114.6, 111.2, 89.9, 80.0, 55.6; IR (KBr) ῡ 2215, 1725, 1609, 763 cm^{-1}.



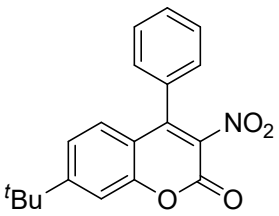
Phenyl 3-(3-bromophenyl)propiolate (1y).[13] R_f = 0.7 (5% ethyl acetate/hexane); yellow solid; yield 106 mg (51%); mp: 60-62 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (s, 1H), 7.58 (d, *J* = 8.4 Hz, 1H), 7.51 (d, *J* = 8.4 Hz, 1H), 7.38 (t, *J* = 8.0 Hz, 2H), 7.25 (d, *J* = 7.6 Hz, 2H), 7.16 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.1, 150.2, 135.9, 134.4, 131.8, 130.3 129.8, 126.7, 122.6, 121.5, 121.4, 86.6, 81.2; IR (KBr) ῡ 2236, 1728, 1648, 766 cm^{-1}.

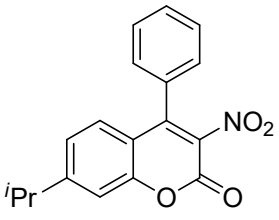


7-Methyl-3-nitro-4-phenyl-2H-chromen-2-one (2a). R_f = 0.5 (10% ethyl acetate/hexane); yellow solid; yield 67 mg (70%); mp: 185-187 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 – 7.52 (m, 3H), 7.37 (dd, *J* = 7.2, 1.6 Hz, 2H), 7.28 (s, 1H), 7.17 (d, *J* = 8.4 Hz, 1H), 7.12 (d, *J* = 8.8 Hz, 1H), 2.50 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.8, 153.1, 147.4, 146.3, 136.1, 130.8, 129.3, 129.2, 129.0, 128.0, 127.0, 117.7, 115.5, 22.0; IR (KBr) ῡ 1735, 1616, 1540, 1462, 741 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{16}H_{12}NO_4$ $[M+H]^+$ 282.0761, found 282.0763.



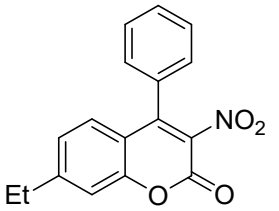
7-(*tert*-Butyl)-3-nitro-4-phenyl-2H-chromen-2-one (2b). R_f = 0.4 (10% ethyl acetate/hexane); yellow liquid; yield 69 mg (81%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 – 7.51 (m, 3H), 7.47 (d, *J* = 2.0 Hz, 1H), 7.39 – 7.34 (m, 3H), 7.23 (d, *J* = 8.4 H, 1H), 1.36 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.5, 153.9, 153.1, 147.3, 136.2, 130.8, 129.3, 129.2, 128.9, 128.0, 123.4, 115.4, 114.4, 35.7, 31.0; IR (KBr) ῡ 1735, 1635, 1540, 1457, 756 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{19}H_{18}NO_4$ $[M+H]^+$ 324.1230, found 324.1230.



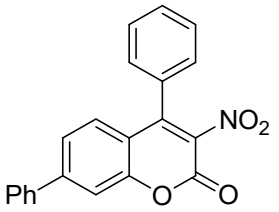


7-Isopropyl-3-nitro-4-phenyl-2H-chromen-2-one (2c). R_f = 0.5 (10% ethyl acetate/hexane); yellow solid; yield 74 mg (72%); mp: 138-140 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 – 7.51 (m, 3H), 7.38 – 7.36 (m, 2H), 7.32 (d, *J* = 0.8 Hz, 1H), 7.23 – 7.17 (m, 2H), 3.03 (h, *J* = 6.8 Hz, 1H), 1.30 (d, *J* = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.1, 153.9, 153.3, 147.4, 136.1, 130.8, 129.3, 129.3, 129.2, 128.1, 124.5, 115.8, 115.2, 34.5, 23.6; IR (KBr) ῡ 1738, 1611, 1541, 1421, 744 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{18}H_{15}NNaO_4$ $[M+Na]^+$ 332.0893, found 332.0915.

7-Ethyl-3-nitro-4-phenyl-2H-chromen-2-one (2d). R_f = 0.4 (10% ethyl acetate/hexane); light yellow solid; yield 64 mg (68%); mp: 172-175 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 – 7.51 (m, 3H), 7.39 – 7.36 (m, 2H), 7.30 (d, *J* = 0.8 Hz, 1H), 7.20 (d, *J* = 8.4 Hz, 1H), 7.15 (dd, *J* = 8.4, 1.6 Hz, 1H), 2.79 (q, *J* = 7.6 Hz, 2H), 1.29 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.9, 153.3, 152.5, 147.4, 136.1, 130.8, 129.3, 129.2, 129.1, 128.1, 125.9, 116.5, 115.7, 29.2, 15.1; IR (KBr) ῡ 1738, 1611, 1542, 1421, 760 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{17}H_{13}NNaO_4$ $[M+Na]^+$ 318.0737, found 318.0733.



3-Nitro-4,7-diphenyl-2H-chromen-2-one (2e). R_f = 0.3 (10% ethyl acetate/hexane); yellow solid; yield 64 mg (71%); mp: 182-185 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (s, 1H), 7.64 (d, *J* = 6.8 Hz, 2H), 7.58 – 7.47 (m, 7H), 7.42 (d, *J* = 4.8 Hz, 2H), 7.36 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.7, 153.5, 147.6, 147.2, 138.4, 130.9, 129.7 129.5, 129.4, 129.4, 129.1, 129.1, 128.1, 127.5, 124.5, 116.8, 115.6; IR (KBr) ῡ 1739, 1616, 1543, 1421, 760 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{21}H_{14}NO_4$ $[M+H]^+$ 344.0917, found 344.0920.

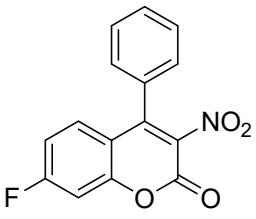


7-Iodo-3-nitro-4-phenyl-2H-chromen-2-one (2f). R_f = 0.4 (10% ethyl acetate/hexane); yellow solid; yield 37 mg (62%); mp: 220-222 °C; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.87 (s, 1H), 7.64 (dd, *J* = 8.4, 0.7 Hz, 1H), 7.59 – 7.54 (m, 3H), 7.36 (d, *J* = 7.0 Hz, 2H), 6.98 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.8, 152.5, 146.9, 136.8, 135.1, 131.2, 130.0, 129.5, 128.4, 128.0, 126.8, 117.6, 100.8; IR (KBr) ῡ 1745, 1591, 1546, 1421, 760 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{15}H_9INO_4$ $[M+H]^+$ 393.9571, found 393.9557.

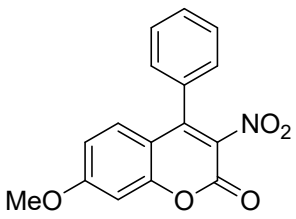
7-Bromo-3-nitro-4-phenyl-2H-chromen-2-one (2g). R_f = 0.3 (10% ethyl acetate/hexane); yellow solid; yield 45 mg (63%); mp: 190-192 °C; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.67 (s, 1H), 7.59 – 7.55 (m, 3H), 7.44 (dd, *J* = 8.4, 1.4 Hz, 1H), 7.36 (d, *J* = 7.0 Hz, 2H), 7.16 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.0, 152.9, 146.8, 136.6, 131.2, 130.2, 129.5, 129.3, 128.9, 128.5 128.0, 120.9, 117.1; IR (KBr) ῡ 1745, 1617, 1597, 1545, 1458, 763 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{15}H_8BrNNaO_4$ $[M+Na]^+$ 367.9529 , found 367.9515.

7-Chloro-3-nitro-4-phenyl-2H-chromen-2-one (2h). R_f = 0.4 (10% ethyl acetate/hexane); yellowish white solid; yield 35 mg (59%); mp: 204-206 °C; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.60-7.55 (m, 3H), 7.50 (d, *J* = 1.4 Hz, 1H), 7.36 (d, *J* = 7.0 Hz, 2H), 7.29 (dd, *J* = 8.4, 1.4 Hz, 1H), 7.24 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.1, 153.0, 146.7, 140.7, 131.2, 130.2, 129.5, 128.6, 128.0, 126.4, 117.9, 116.7; IR (KBr) ῡ 1744, 1641, 1540, 1461, 752 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{15}H_9ClNO_4$ $[M+H]^+$ 302.0215, found 302.0214.

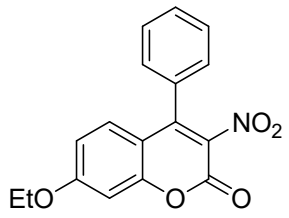
7-Fluoro-3-nitro-4-phenyl-2H-chromen-2-one (2i). R_f = 0.4 (10% ethyl acetate/hexane); light yellow solid; yield 38 mg (52%); mp: 178-180 °C; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.59 – 7.55 (m, 3H), 7.37 (d, *J* = 7.0 Hz, 2H), 7.31 (dd, *J* = 9.1, 6.3 Hz, 1H), 7.21 (dd, *J* = 8.4, 2.1 Hz, 1H), 7.07 – 7.05 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.9 (d, $^1J_{C\text{-}F}$ = 257.3 Hz), 154.3, 154.2, 153.2, 147.0, 131.3 (d, $^3J_{C\text{-}F}$ = 10.5 Hz), 131.1, 129.5, 128.8, 128.0, 114.8 (d, $^4J_{C\text{-}F}$ = 2.9 Hz), 114.1 (d, $^2J_{C\text{-}F}$ = 22.5 Hz), 105.3 (d, $^2J_{C\text{-}F}$ = 25.8 Hz); IR (KBr) ῡ 1749, 1616, 1541, 1457, 764 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{15}H_9FNO_4$ $[M+H]^+$ 286.0510, found 286.0512.



7-Methoxy-3-nitro-4-phenyl-2H-chromen-2-one (2j). R_f = 0.3 (10% ethyl acetate/hexane); yellow solid; yield 59 mg (61%); mp: 165-167 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 – 7.51 (m, 3H), 7.36 (dd, *J* = 7.4, 1.8 Hz, 2H), 7.18 (d, *J* = 8.8 Hz, 1H), 6.94 (d, *J* = 2.4 Hz, 1H), 6.86 (dd, *J* = 8.8, 2.4 Hz, 1H), 3.92 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.9, 155.1, 154.0, 148.0, 134.5, 130.8, 130.4, 129.5, 129.3, 128.0, 114.3, 111.1, 101.3, 56.3; IR (KBr) ῡ 1739, 1617, 1540, 1462, 763 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{16}H_{12}NO_5$ $[M+H]^+$ 298.0710, found 298.0716.

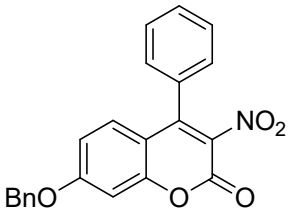


7-Ethoxy-3-nitro-4-phenyl-2H-chromen-2-one (2k). R_f = 0.3 (10% ethyl acetate/hexane); yellow solid; yield 42 mg (57%); mp: 175-177 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 – 7.50 (m, 3H), 7.37 – 7.35 (m, 2H), 7.17 (d, *J* = 9.2 Hz, 1H), 6.91 (d, *J* = 2.4 Hz, 1H), 6.84 (dd, *J* = 8.8, 2.4 Hz, 1H), 4.14 (q, *J* = 7.2 Hz, 2H), 1.48 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.3, 155.2, 154.1, 148.1, 134.4, 130.7, 130.4, 129.5, 129.3, 128.0, 114.6, 110.9,

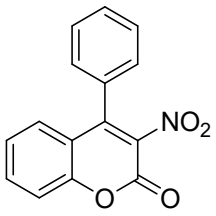


101.7, 64.9, 14.6; IR (KBr) ῡ 1739, 1610, 1542, 1421, 756 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{17}H_{13}NNaO_5$ $[M+Na]^+$ 334.0686, found 334.0692.

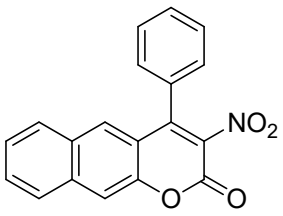
7-(Benzyloxy)-3-nitro-4-phenyl-2H-chromen-2-one (2l). R_f = 0.3 (10% ethyl acetate/hexane); yellow solid; yield 42 mg (56%); mp: 192-194 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.55 – 7.50 (m, 3H), 7.43 – 7.34 (m, 7H), 7.19 (d, *J* = 9.2 Hz, 1H), 7.00 (d, *J* = 2.4 Hz, 1H), 6.93 (dd, *J* = 9.2, 2.4 Hz, 1H), 5.18 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.9, 155.0, 154.0, 148.0, 135.2, 134.5, 130.7, 130.5, 129.4, 129.3, 129.0, 128.8, 128.0, 127.7, 114.8, 111.3, 102.3, 71.0; IR (KBr) ῡ 1735, 1609, 1539, 1490, 734 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{22}H_{16}NO_5$ $[M+H]^+$ 374.1023, found 374.1008.



3-Nitro-4-phenyl-2H-chromen-2-one (2m).[14] R_f = 0.3 (10% ethyl acetate/hexane); yellow solid; yield 29 mg (55%); mp: 175-177 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.69 (s, 1H), 7.56 – 7.47 (m, 4H), 7.39-7.32 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.6, 153.0, 147.2, 136.8, 134.3, 130.9, 129.3, 128.9, 128.1, 125.7; 118.0, 117.6; IR (KBr) ῡ 1741, 1604, 1543, 1421, 764 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{15}H_9NNaO_4$ $[M+Na]^+$ 290.0424, found 290.0426.

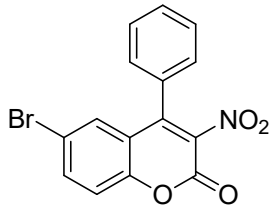


3-Nitro-4-phenyl-2H-benzo[g]chromen-2-one (2n). R_f = 0.3 (10% ethyl acetate/hexane); yellow solid; yield 58 mg (63%); mp: 172-174 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 8.59 (dd, *J* = 6.8, 2.8 Hz, 1H), 7.90 (dd, *J* = 6.4, 2.4 Hz, 1H), 7.77 – 7.70 (m, 2H), 7.68 (d, *J* = 8.8 Hz, 1H), 7.58 (dd, *J* = 5.0, 1.8 Hz, 3H), 7.44 – 7.41 (m, 2H), 7.21 (d, *J* = 8.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.6, 151.0, 148.5, 136.5, 135.9, 130.8, 130.4, 129.5, 129.4, 128.2, 128.1,

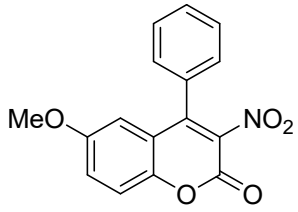


125.7, 123.1, 123.0, 122.8, 113.3; IR (KBr) ῡ 1741, 1636, 1542, 1466, 748 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{19}H_{11}NNaO_4$ $[M+Na]^+$ 340.0580, found 340.0585.

7-Bromo-3-nitro-4-phenyl-2H-chromen-2-one (2q). R_f = 0.3 (10% ethyl acetate/hexane); yellow solid; yield 41 mg (60%); mp: 180-182 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.77 (dd, *J* = 9.0, 2.2 Hz), 7.61 – 7.55 (m, 3H), 7.39 – 7.36 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.9, 151.8, 146.0, 137.3, 137.1 131.4, 131.3, 129.6, 128.2, 128.0, 119.8, 119.3, 118.7; IR (KBr) ῡ 1745, 1621, 1546, 1421, 752 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{15}H_9BrNO_4$ $[M+H]^+$ 345.9709, found 345.9709.



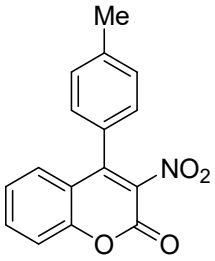
6-Iodo-3-nitro-4-phenyl-2H-chromen-2-one (2r). R_f = 0.3 (10% ethyl acetate/hexane); yellow solid; yield 52 mg (61%); mp: 212-214 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.95 (dd, *J* = 8.6, 2.2 Hz, 1H), 7.61 – 7.55 (m, 4H), 7.36 (dd, *J* = 7.8, 1.8 Hz, 2H), 7.24 (d, *J* = 8.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.9, 152.6, 145.9, 142.9, 137.4, 137.1, 131.3, 129.6, 128.2, 128.0, 120.1, 119.5, 88.8; IR (KBr) ῡ 1742, 1640, 1546, 1420, 763 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{15}H_8INNaO_4$ $[M+Na]^+$ 415.9390, found 415.9376.



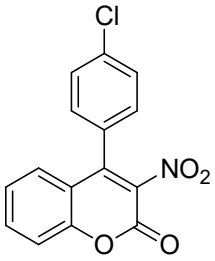
6-Methoxy-3-nitro-4-phenyl-2H-chromen-2-one (2s). R_f = 0.2 (10% ethyl acetate/hexane); yellow solid; yield 77 mg (64%); mp: 166-168 °C; ^1H NMR (700 MHz, $CDCl_3$) δ 7.57 – 7.524 (m, 3H), 7.42-7.38 (m, 3H), 7.26-7.24 (m, 1H), 6.68 (d, *J* = 2.8 Hz, 1H), 3.71 (s, 3H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 156.9, 153.7, 147.4, 146.9, 137.2, 131.0, 129.4, 129.0,

128.0, 121.5, 118.6, 118.6, 111.7, 56.0; IR (KBr) ῡ 1737, 1634, 1570, 1543, 1421, 752 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{16}H_{12}NO_5$ $[M+H]^+$ 298.0710, found 298.0707.

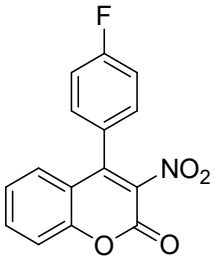
3-Nitro-4-(*p*-tolyl)-2H-chromen-2-one (2u). R_f = 0.3 (10% ethyl acetate/hexane); light yellow solid; yield 53 mg (68%); mp: 162-164 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.70 – 7.66 (m, 1H), 7.47 (d, *J* = 8.4 Hz, 1H), 7.38 – 7.32 (m, 4H), 7.30 – 7.28 (m, 2H), 2.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.7, 152.9, 147.4, 141.4, 136.7, 134.2, 130.0, 129.4, 128.0, 125.9, 125.6, 118.1, 117.5, 21.6; IR (KBr) ῡ 1741, 1636, 1543, 1466, 752 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{16}H_{11}NNaO_4$ $[M+Na]^+$ 304.0580, found 304.0579.



4-(4-Chlorophenyl)-3-nitro-2H-chromen-2-one (2v). R_f = 0.6 (10% ethyl acetate/hexane); light yellow solid; yield 59 mg (63%); mp: 182-184 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.73 – 7.69 (m, 1H), 7.54 (d, *J* = 8.4 Hz, 2H), 7.49 (d, *J* = 8.0 Hz, 1H), 7.37 – 7.32 (m, 3H), 7.29 (d, *J* = 1.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.3, 153.0, 146.1, 137.4, 136.9, 134.6, 129.8, 129.5, 129.0, 127.2, 125.9, 117.8, 117.7; IR (KBr) ῡ 1748, 1616, 1546, 1421, 740 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{15}H_8ClNNaO_4$ $[M+Na]^+$ 324.0034, found 324.0039.

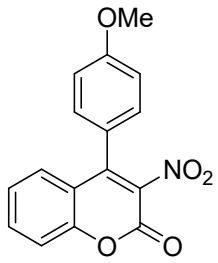


4-(4-Fluorophenyl)-3-nitro-2H-chromen-2-one (2w). R_f = 0.2 (10% ethyl acetate/hexane); yellow solid; yield 48 mg (56%); mp: 172-174 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.71 (t, *J* = 7.6 Hz, 1H), 7.49 (d, *J* = 8.0 Hz, 1H), 7.40 – 7.33 (m, 3H), 7.30 – 7.24 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1 (d, $^1J_{C\text{-}F}$ = 251.0 Hz), 153.4, 153.0, 146.2, 137.0, 134.5, 130.4 (d, $^3J_{C\text{-}F}$ = 8.7 Hz),



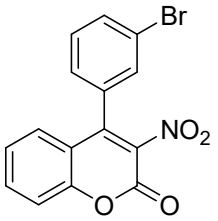
129.0, 125.8, 124.8 (d, $^{4}J_{C\text{-}F}$ = 3.6 Hz), 117.9, 117.7, 116.8 (d, $^{2}J_{C\text{-}F}$ = 22.1 Hz); IR (KBr) ῡ 1745, 1598, 1546, 1421, 748 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{15}H_9FNO_4$ $[M+H]^+$ 286.0510, found 286.0511.

4-(4-Methoxyphenyl)-3-nitro-2H-chromen-2-one (2x). R_f = 0.2 (10% ethyl acetate/hexane);



yellow solid; yield 51 mg (60%); mp: 172-174 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 – 7.66 (m, 1H), 7.47 (dd, *J* = 8.4, 0.8 Hz, 1H), 7.40 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.35 – 7.30 (m, 3H), 7.07 – 7.03 (m, 2H), 3.88 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.7, 153.7, 153.0, 147.0, 136.9, 134.1, 129.8, 129.3, 125.6, 120.8, 118.3, 117.6, 114.9, 55.6; IR (KBr) ῡ 1738, 1624, 1570, 1537, 756 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{16}H_{12}NO_5$ $[M+H]^+$ 298.0710, found 298.0692.

4-(3-Bromophenyl)-3-nitro-2H-chromen-2-one (2y). R_f = 0.2 (10% ethyl acetate/hexane);



yellow solid; yield 52 mg (58%); mp: 182-184 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 – 7.69 (m, 2H)), 7.54 (dd, *J* = 2.8, 1.6 Hz, 1H), 7.51 – 7.48 (m, 1H), 7.44 (t, *J* = 8.0 Hz, 1H), 7.37 – 7.32 (m, 2H), 7.27 – 7.25 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.2, 153.0, 145.5 136.9, 134.6, 134.1, 131.0, 130.9, 130.8, 129.0, 126.8, 125.9, 123.4, 117.8, 117.7; IR (KBr) ῡ 1746, 1624, 1545, 1421, 752 cm^{-1}; HRMS (ESI-TOF) calcd for $C_{15}H_8BrNNaO_4$ $[M+Na]^+$ 367.9529, found 367.9551.

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NMR SPECTRA

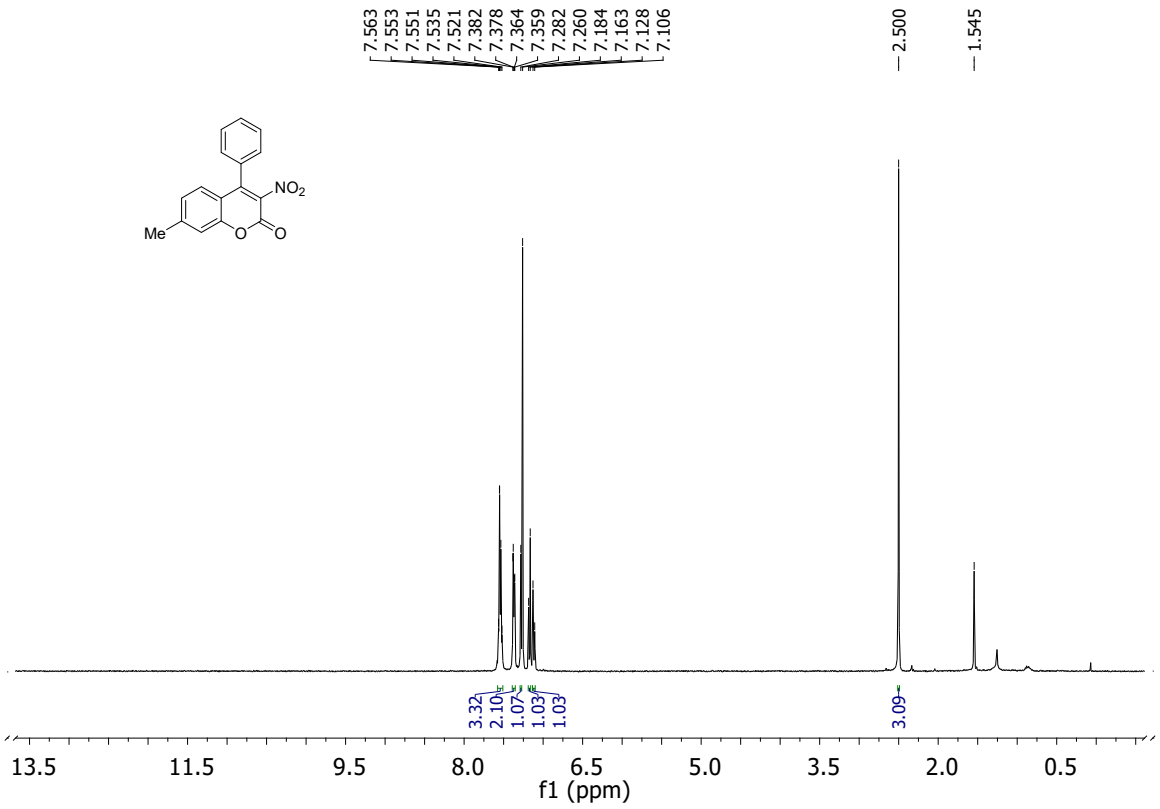

Fig. S4. ^{1}H NMR of 7-Methyl-3-nitro-4-phenyl-2H-chromen-2-one **(2a).**

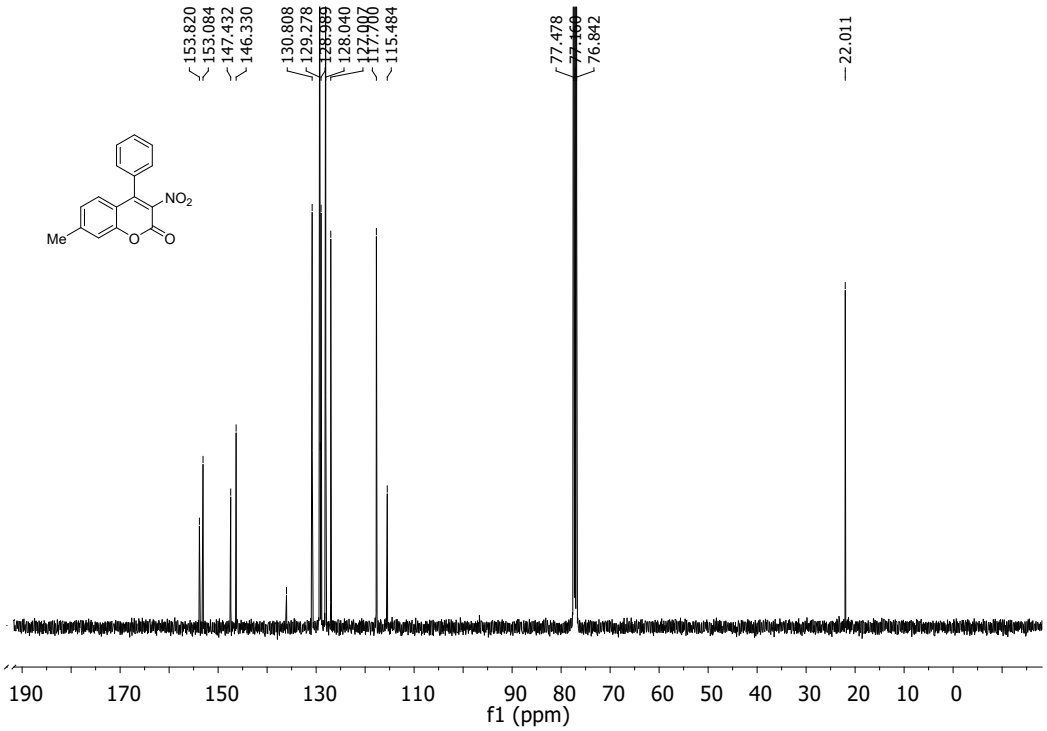

Fig. S5. ^{13}C NMR of 7-Methyl-3-nitro-4-phenyl-2H-chromen-2-one **(2a).**

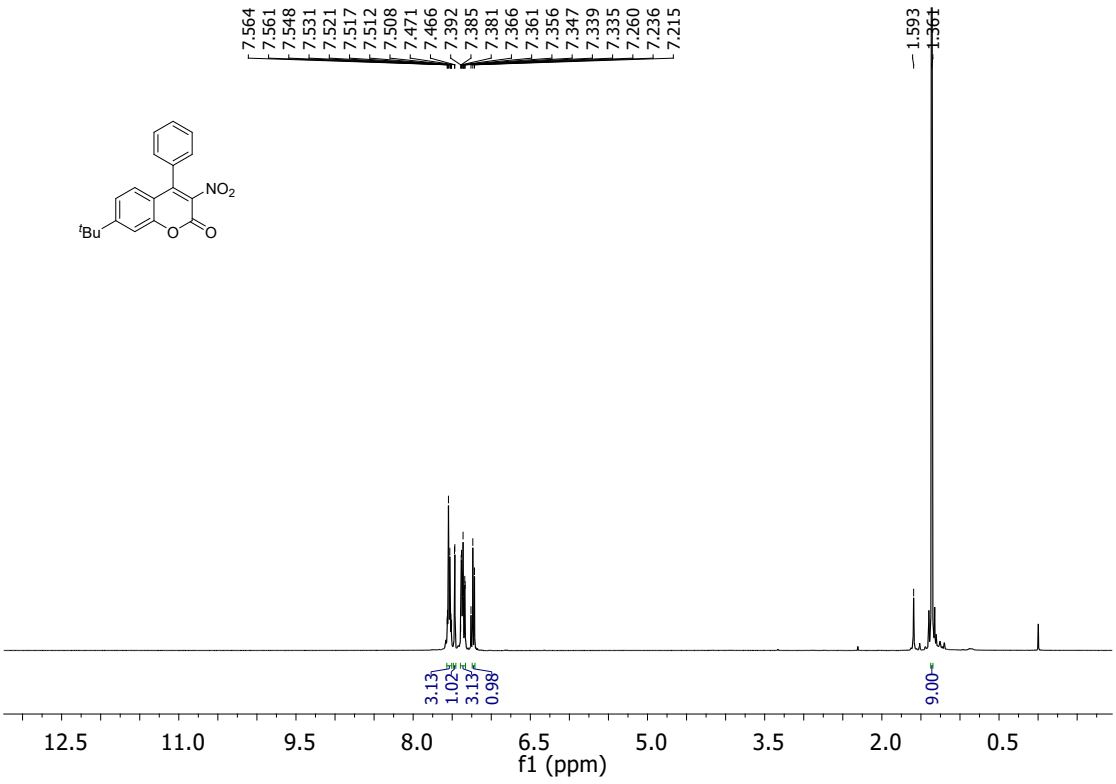

Fig. S6. ^{1}H NMR of 7-(*tert*-Butyl)-3-nitro-4-phenyl-2H-chromen-2-one **(2b).**

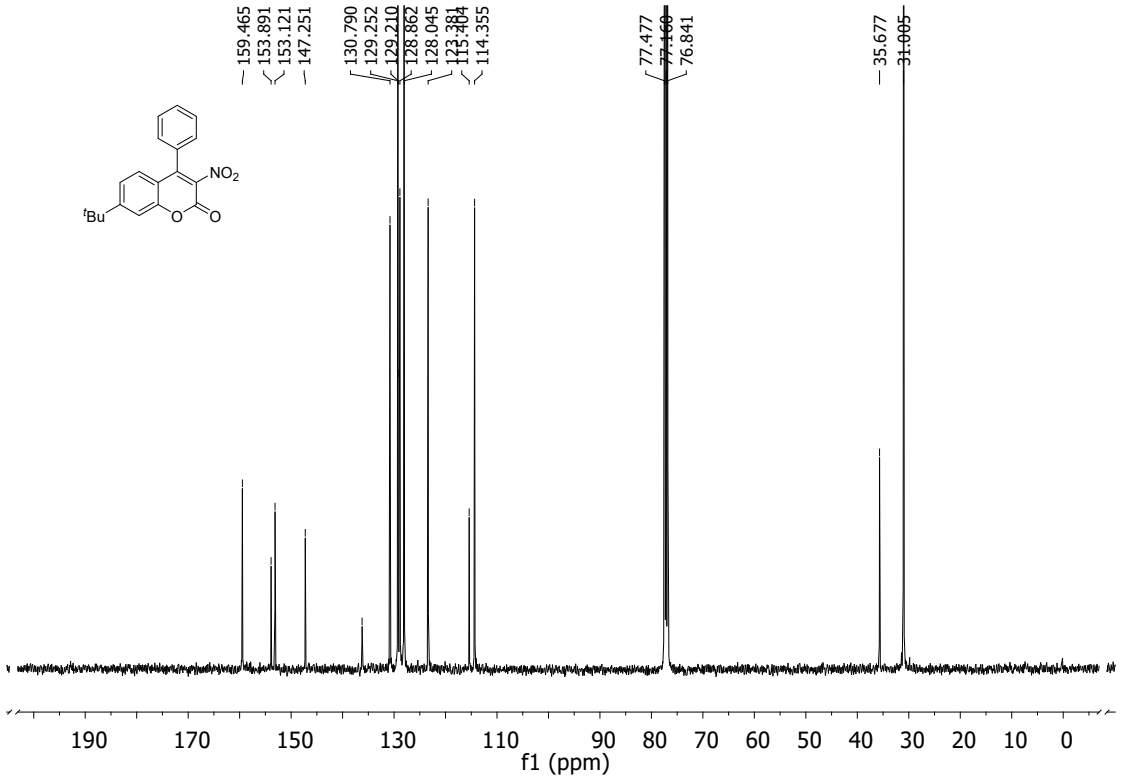

Fig. S7. ^{13}C NMR of 7-(*tert*-Butyl)-3-nitro-4-phenyl-2H-chromen-2-one **(2b).**

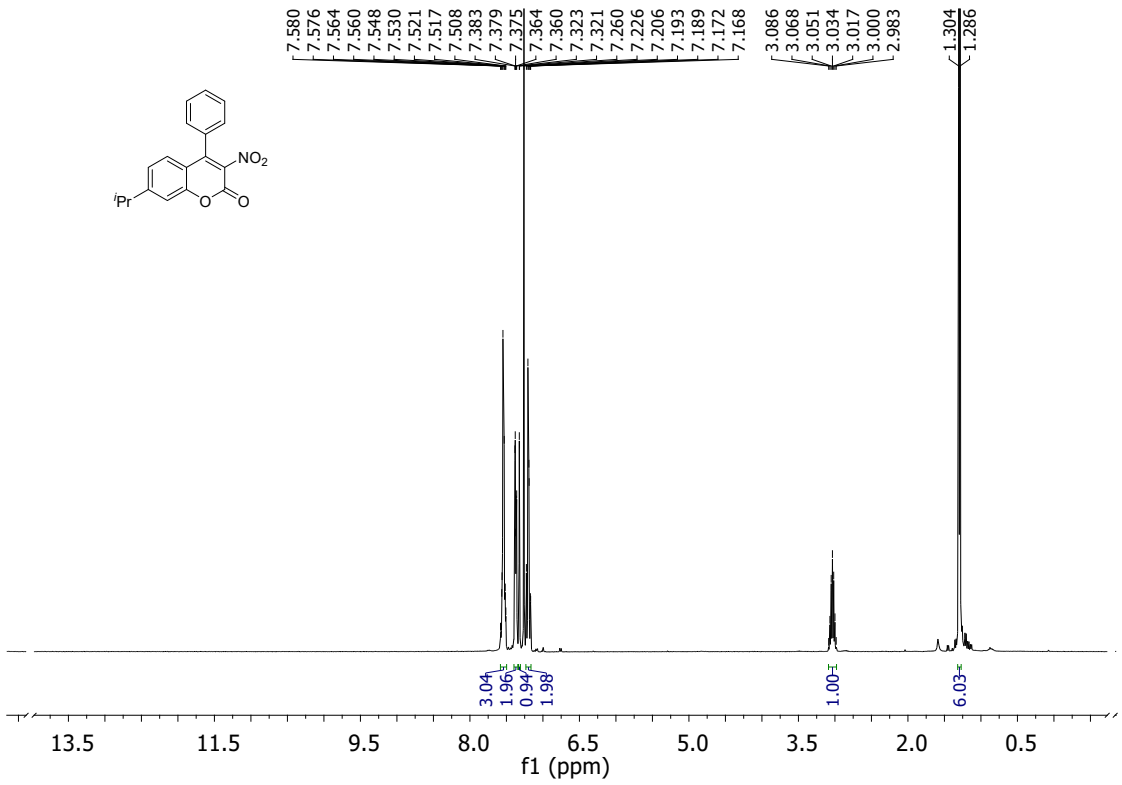

Fig. S8. ^{1}H NMR of 7-Isopropyl-3-nitro-4-phenyl-2H-chromen-2-one **(2c).**

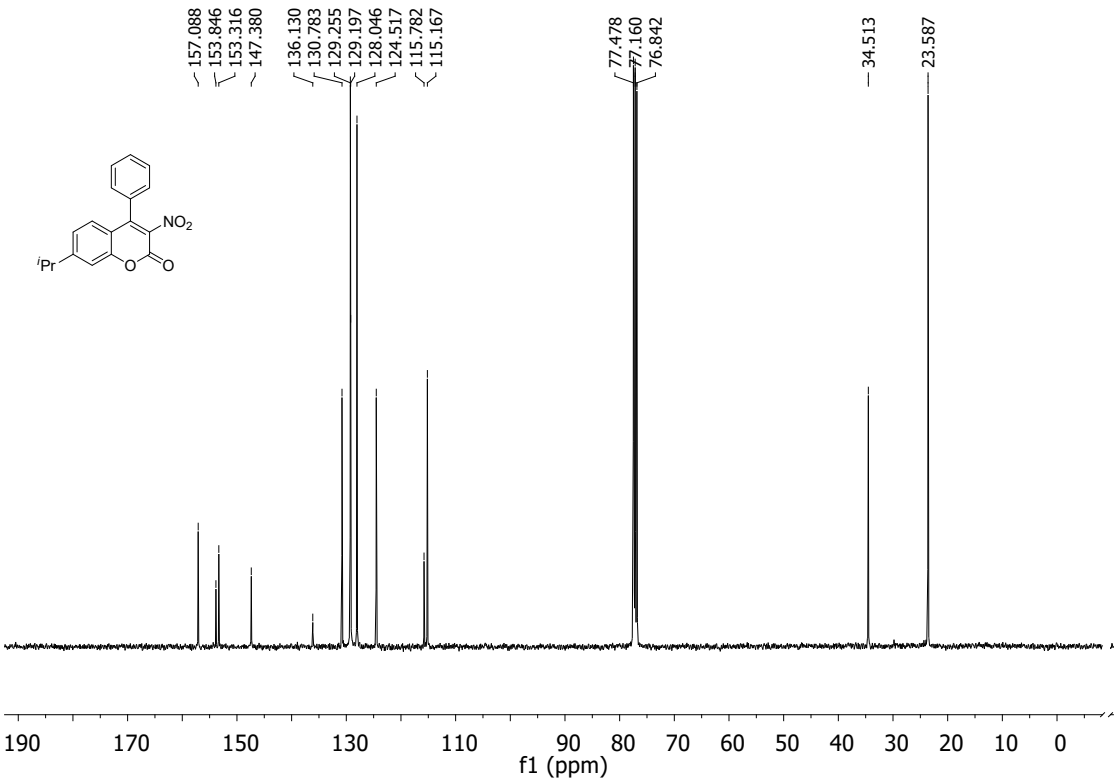

Fig. S9. ^{13}C NMR of 7-Isopropyl-3-nitro-4-phenyl-2H-chromen-2-one **(2c).**

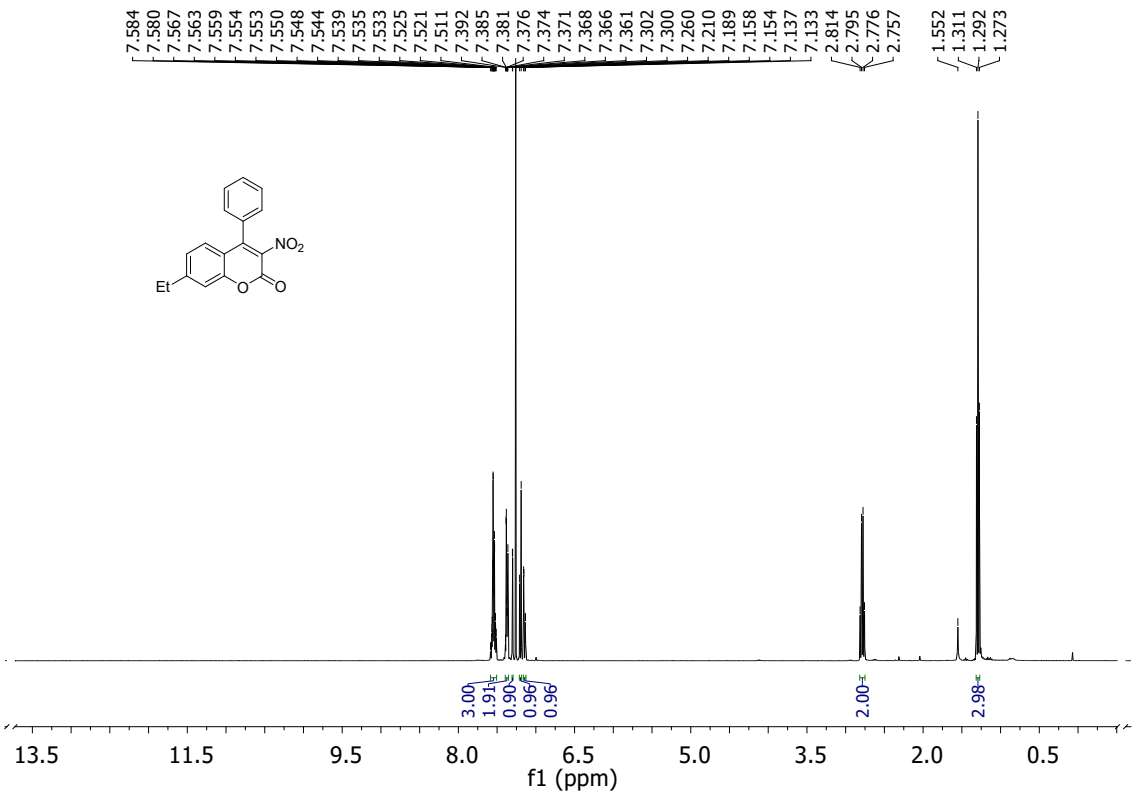

Fig. S10. ^{1}H NMR of 7-Ethyl-3-nitro-4-phenyl-2H-chromen-2-one **(2d).**

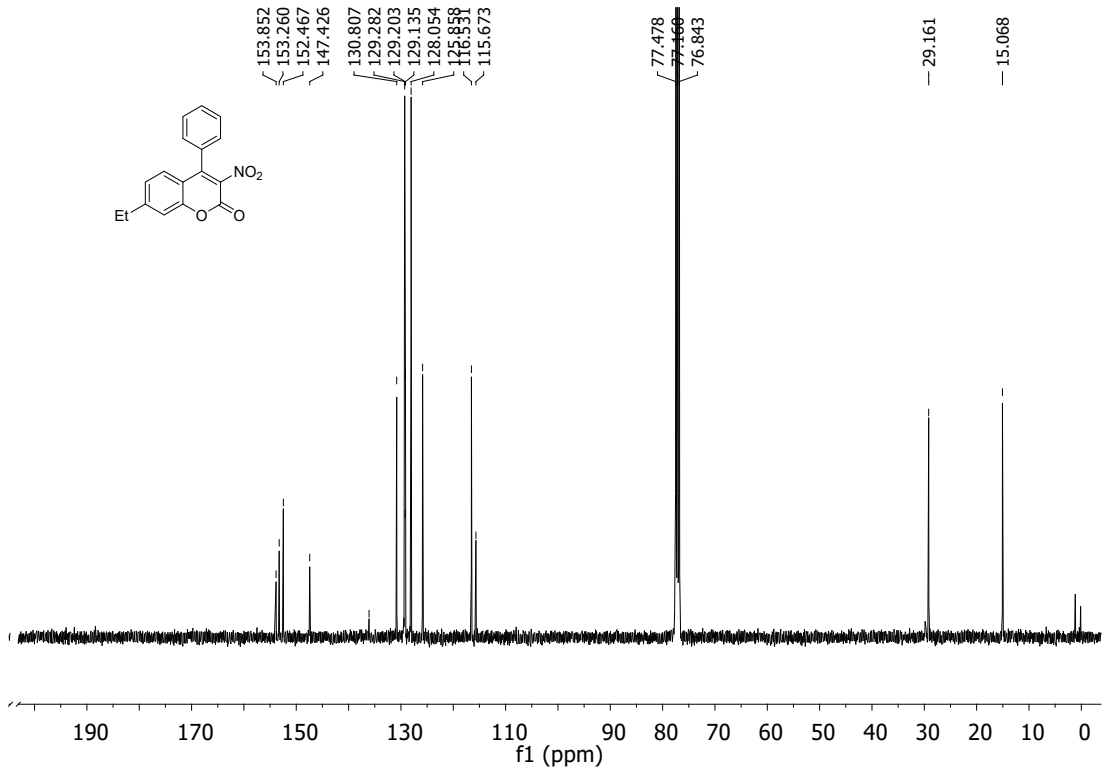

Fig. S11. ^{13}C NMR of 7-Ethyl-3-nitro-4-phenyl-2H-chromen-2-one **(2d).**

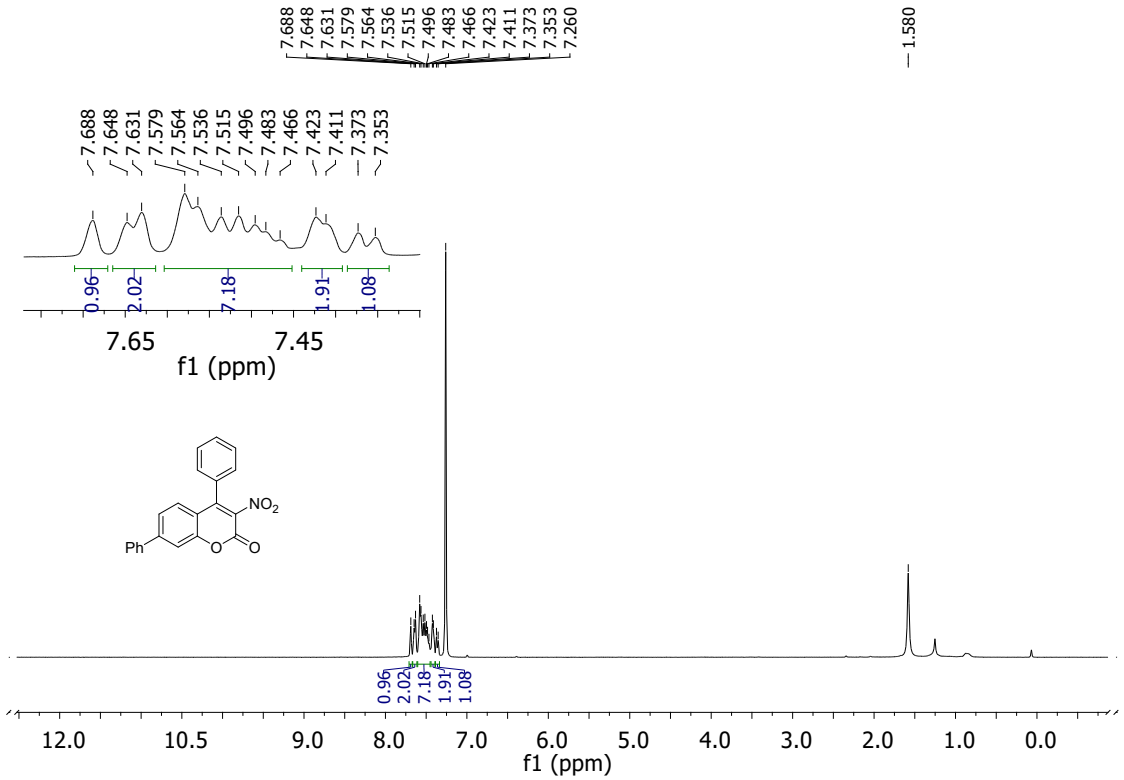

Fig. S12. ^{1}H NMR of 3-Nitro-4,7-diphenyl-2H-chromen-2-one **(2e).**

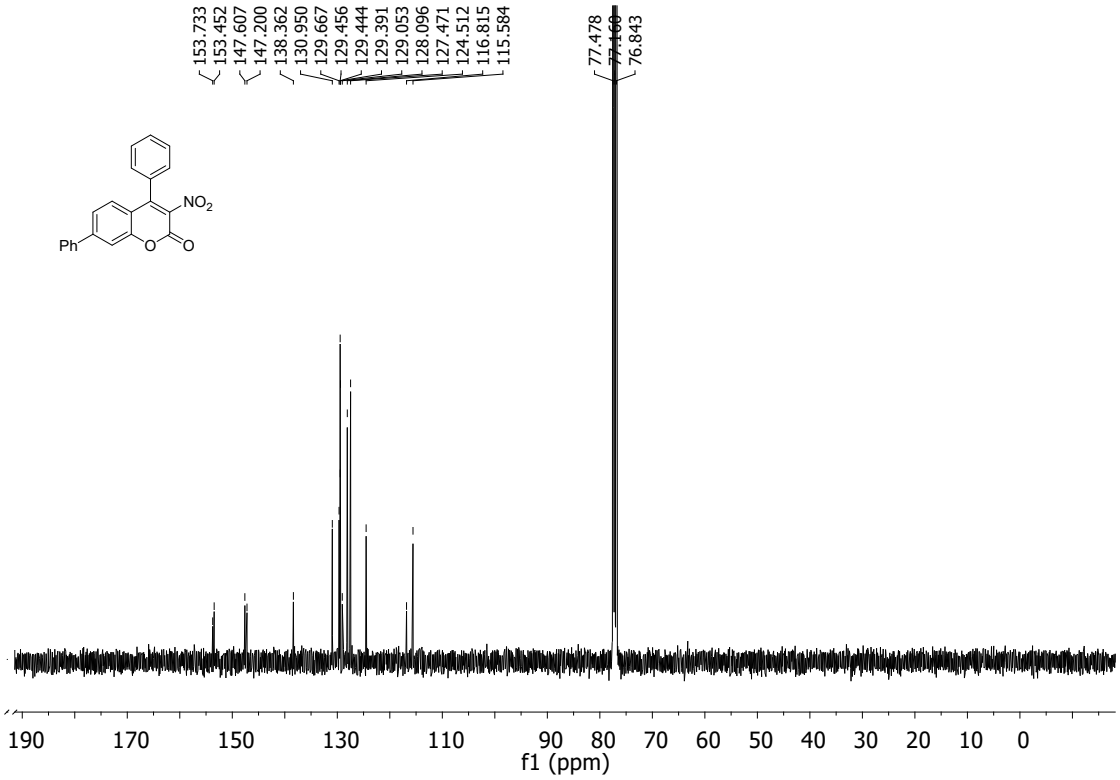

Fig. S13. ^{13}C NMR of 3-Nitro-4,7-diphenyl-2H-chromen-2-one **(2e).**

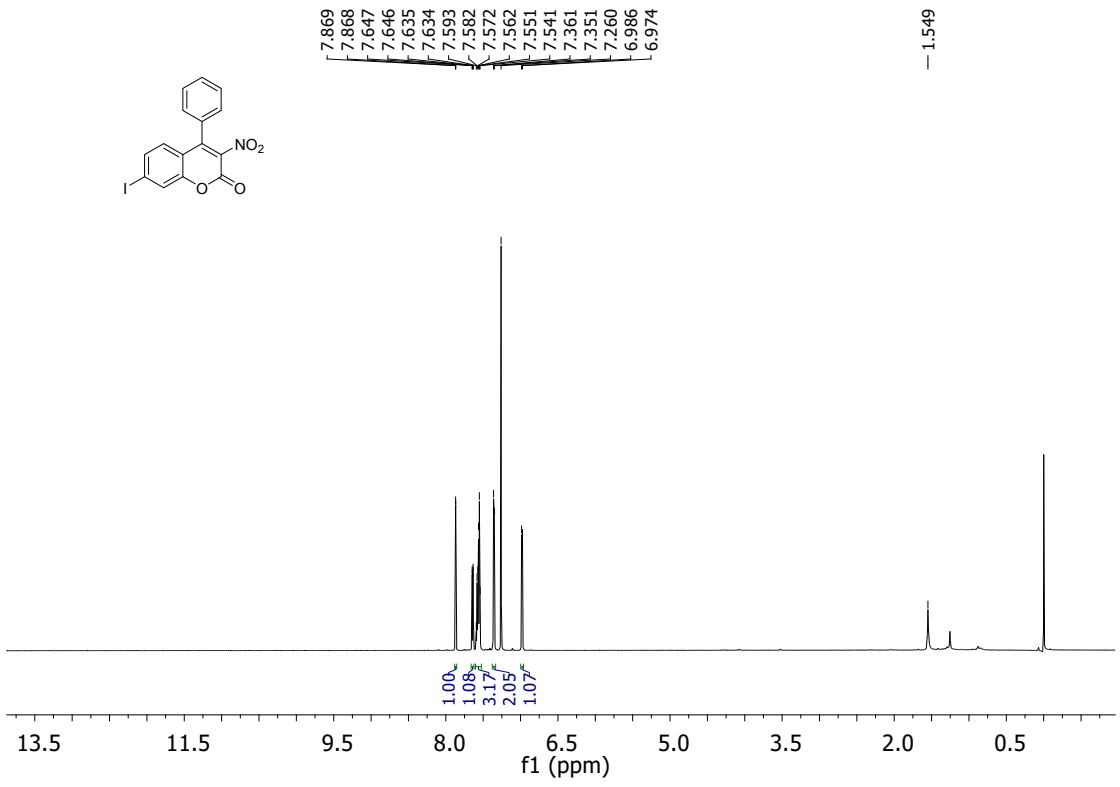

Fig. S14. ^{1}H NMR of 7-Iodo-3-nitro-4-phenyl-2H-chromen-2-one **(2f).**

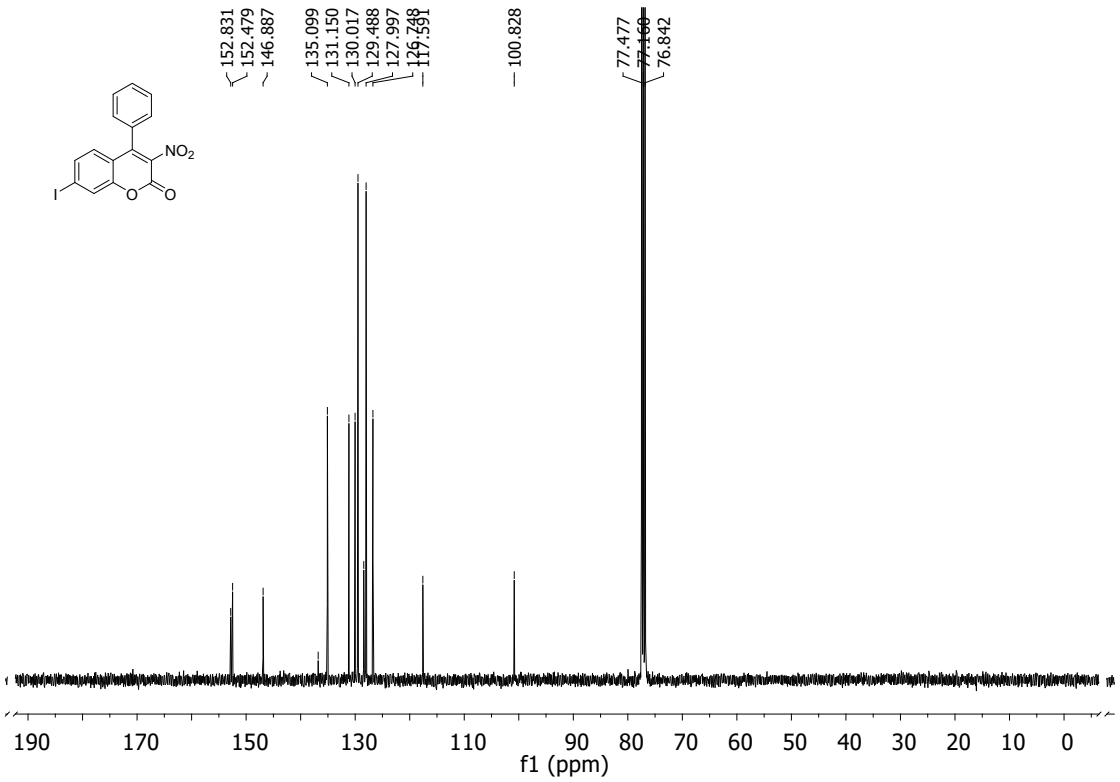

Fig. S15. ^{13}C NMR of 7-Iodo-3-nitro-4-phenyl-2H-chromen-2-one **(2f).**

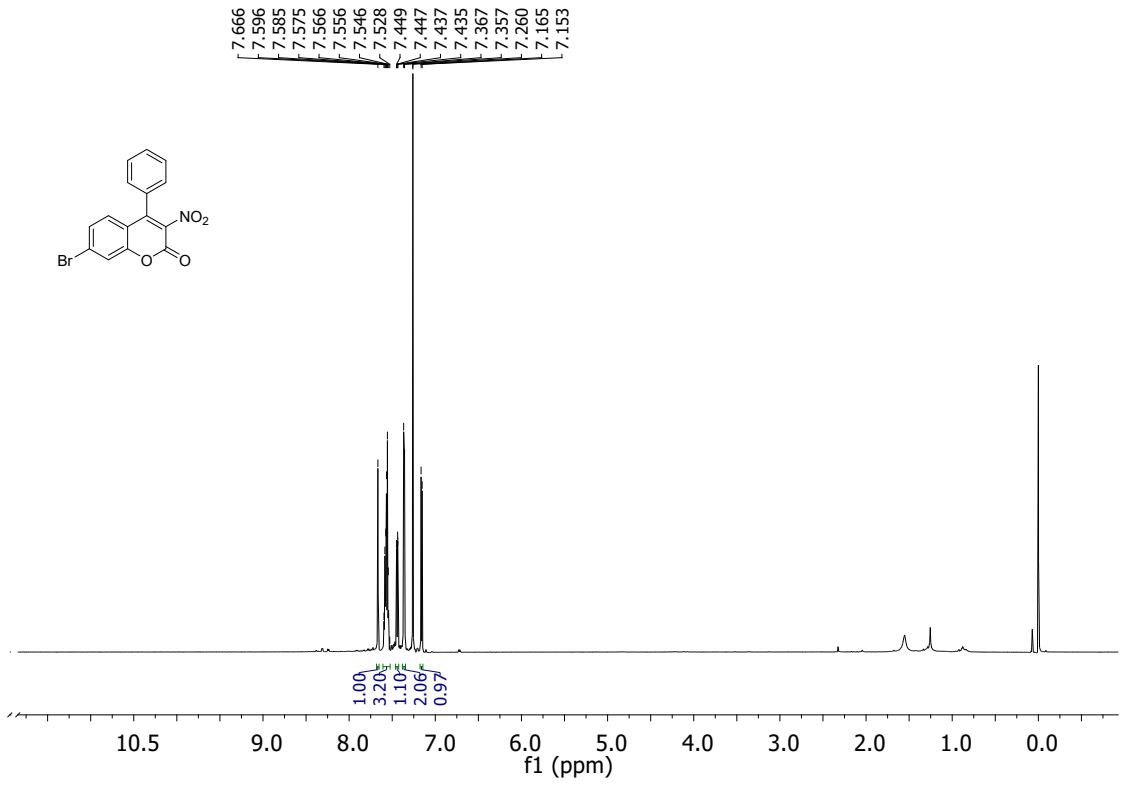

Fig. S16. ^{1}H NMR of 7-Bromo-3-nitro-4-phenyl-2H-chromen-2-one **(2g).**

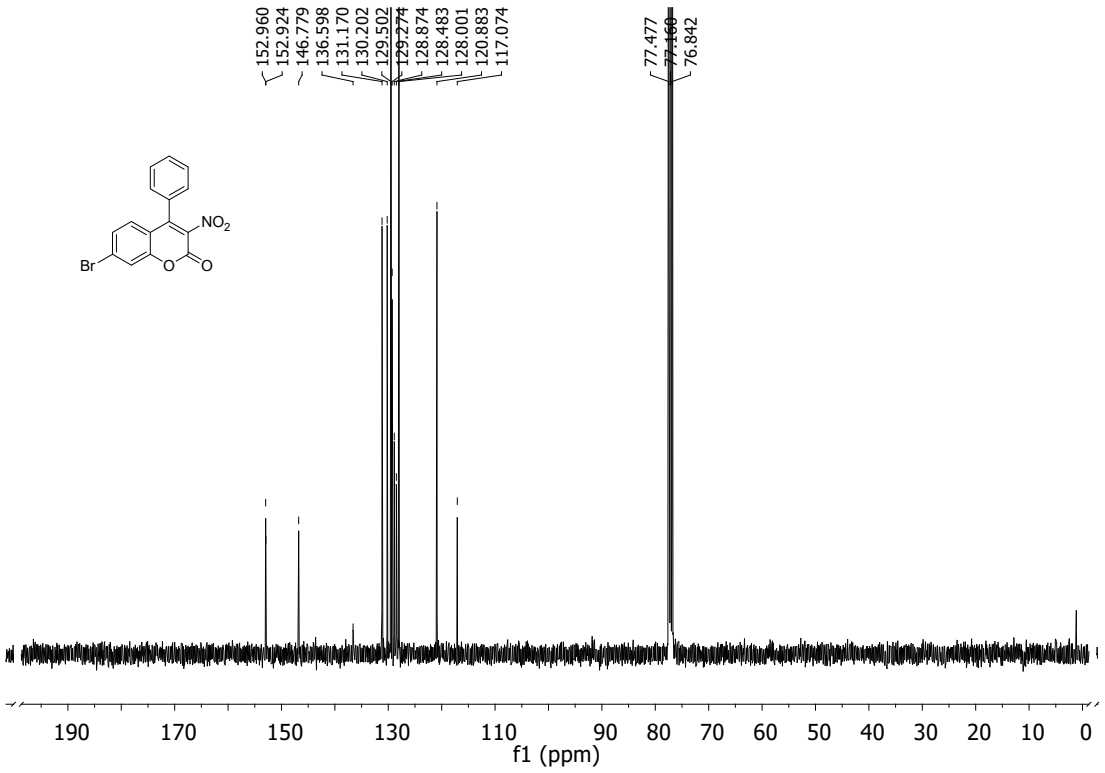

Fig. S17. ^{13}C NMR of 7-Bromo-3-nitro-4-phenyl-2H-chromen-2-one **(2g).**

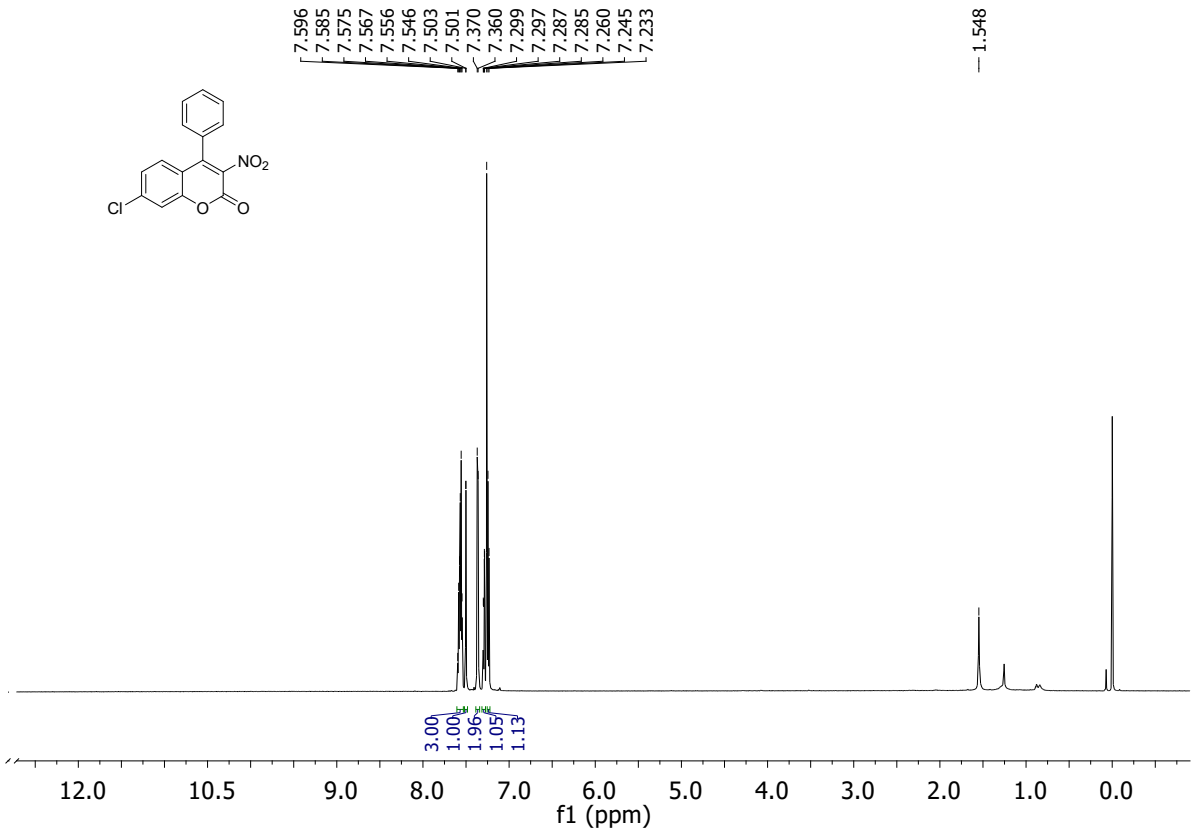

Fig. S18. [1]H NMR of 7-Chloro-3-nitro-4-phenyl-2H-chromen-2-one **(2h).**

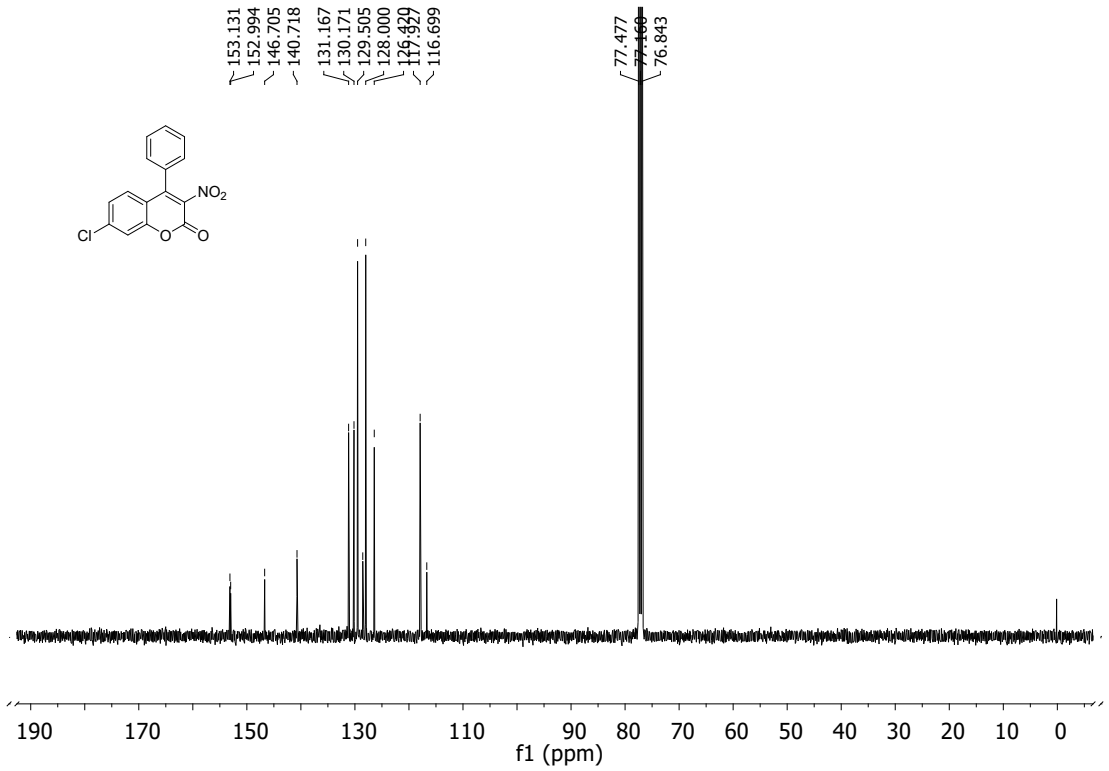

Fig. S19. [13]C NMR of 7-Chloro-3-nitro-4-phenyl-2H-chromen-2-one **(2h).**

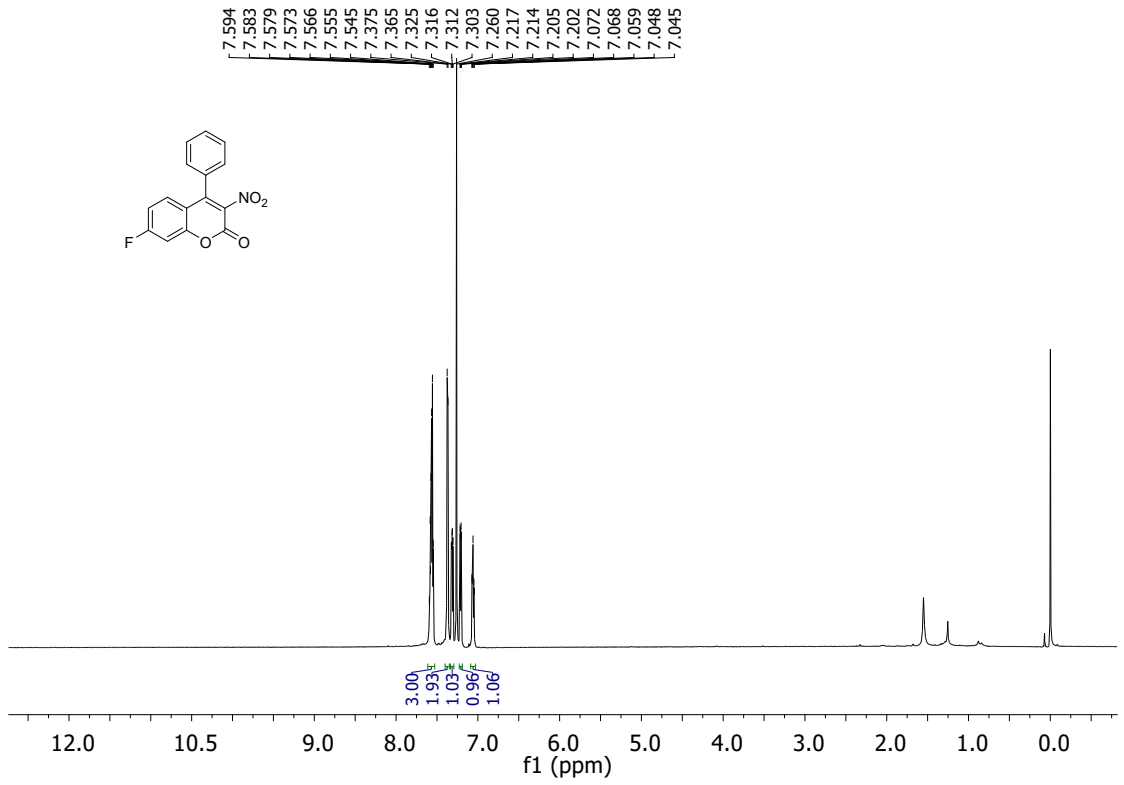

Fig. S20. ^{1}H NMR of 7-Fluoro-3-nitro-4-phenyl-2H-chromen-2-one **(2i).**

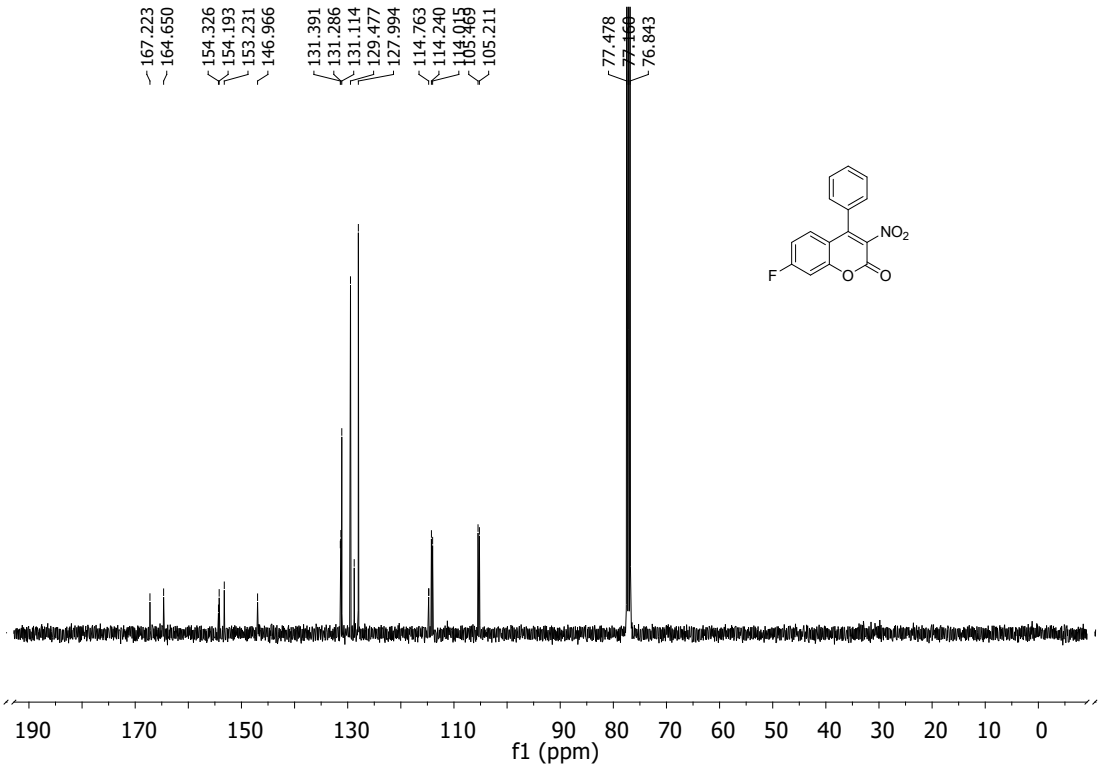

Fig. S21. ^{13}C NMR of 7-Fluoro-3-nitro-4-phenyl-2H-chromen-2-one **(2i).**

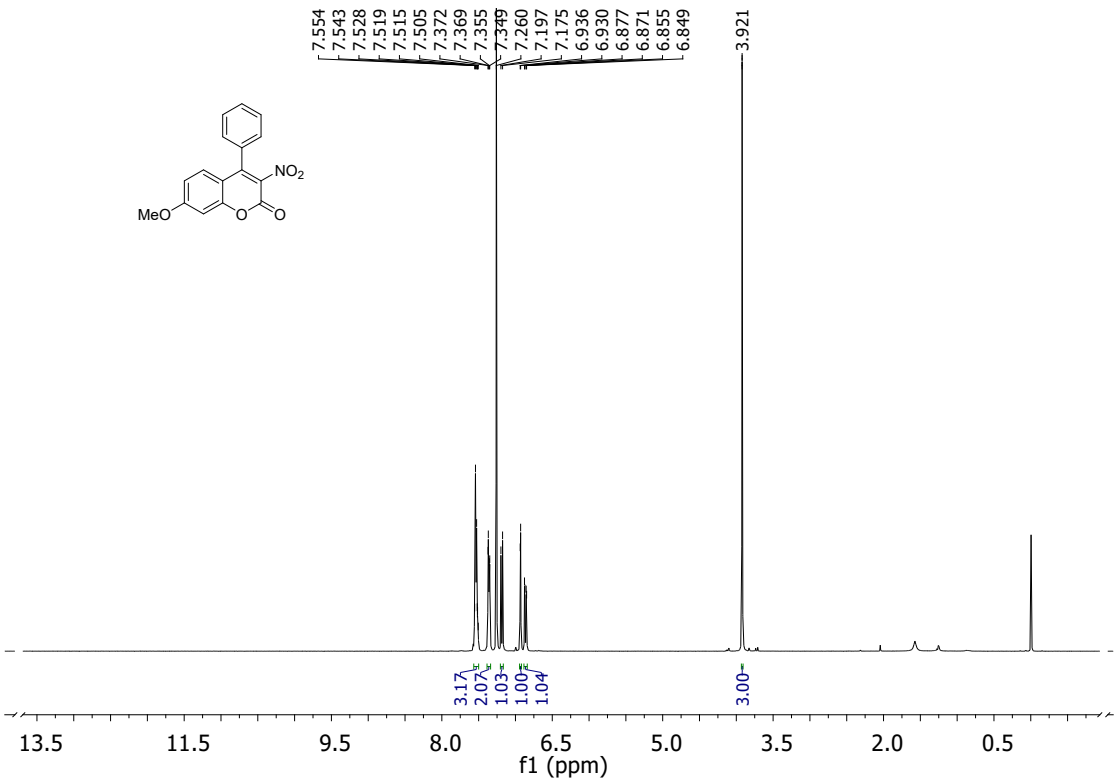

Fig. S22. [1]H NMR of 7-Methoxy-3-nitro-4-phenyl-2H-chromen-2-one **(2j).**

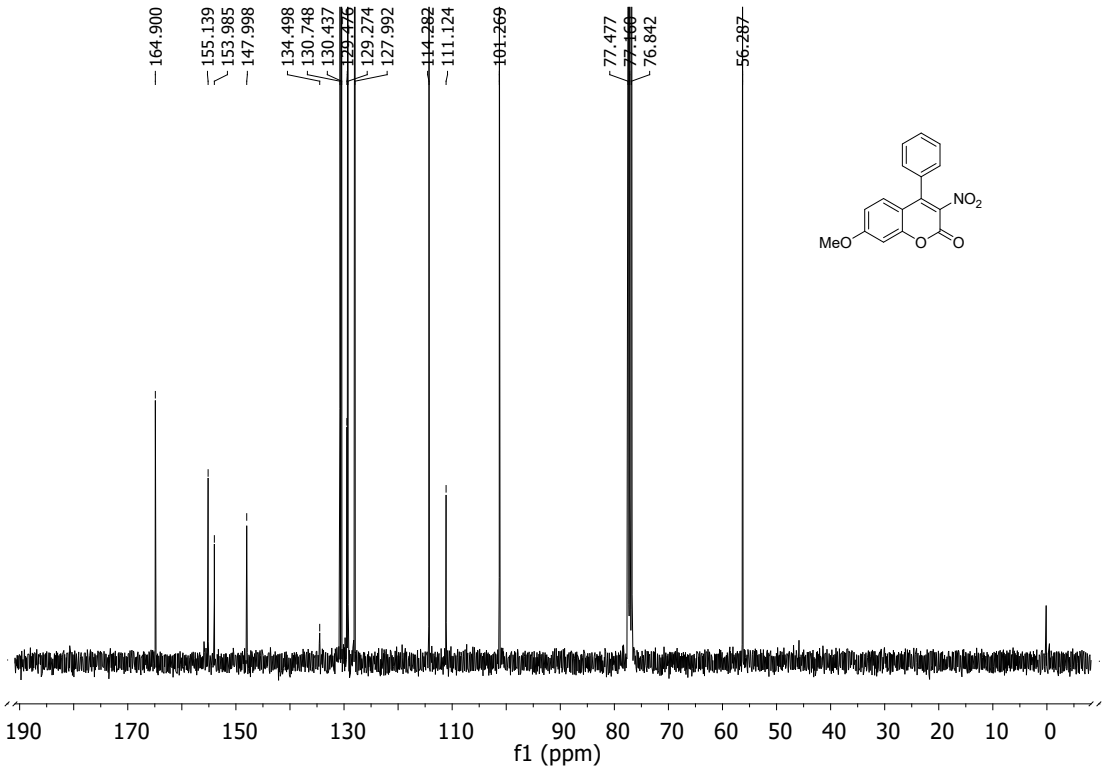

Fig. S23. [13]C NMR of 7-Methoxy-3-nitro-4-phenyl-2H-chromen-2-one **(2j).**

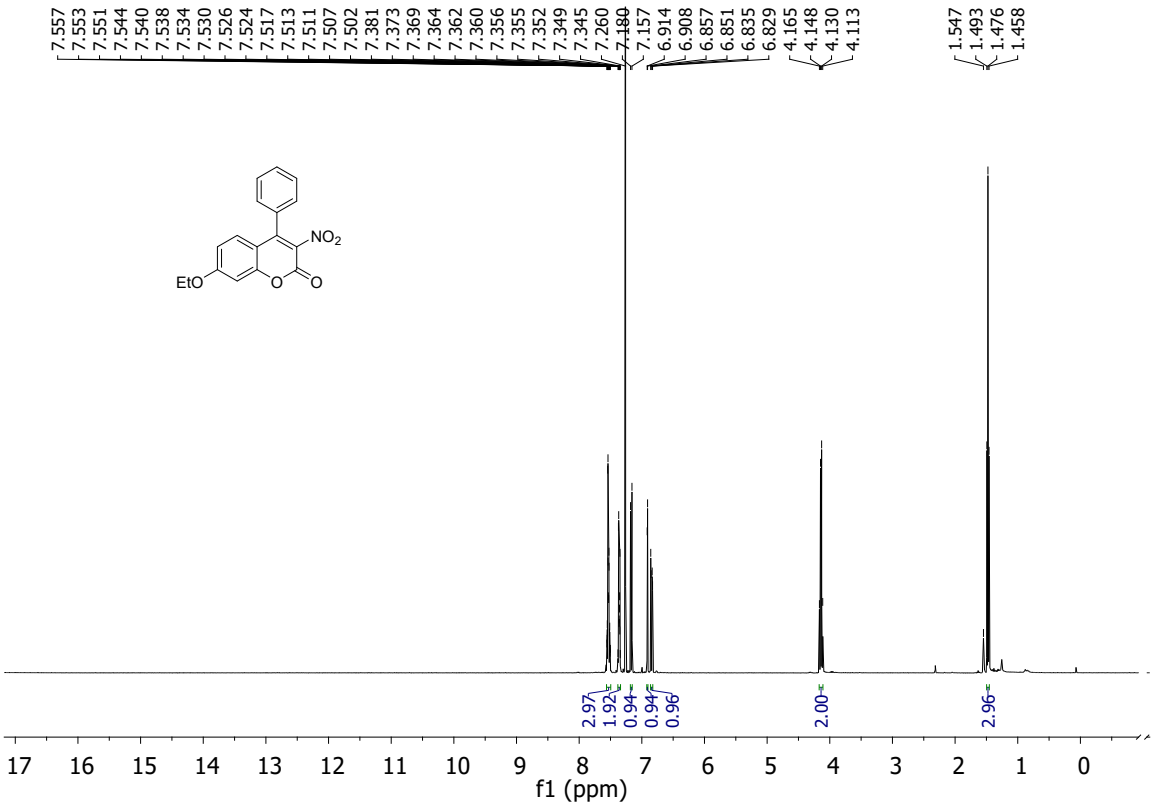

Fig. S24. [1]H NMR of 7-Ethoxy-3-nitro-4-phenyl-2H-chromen-2-one **(2k).**

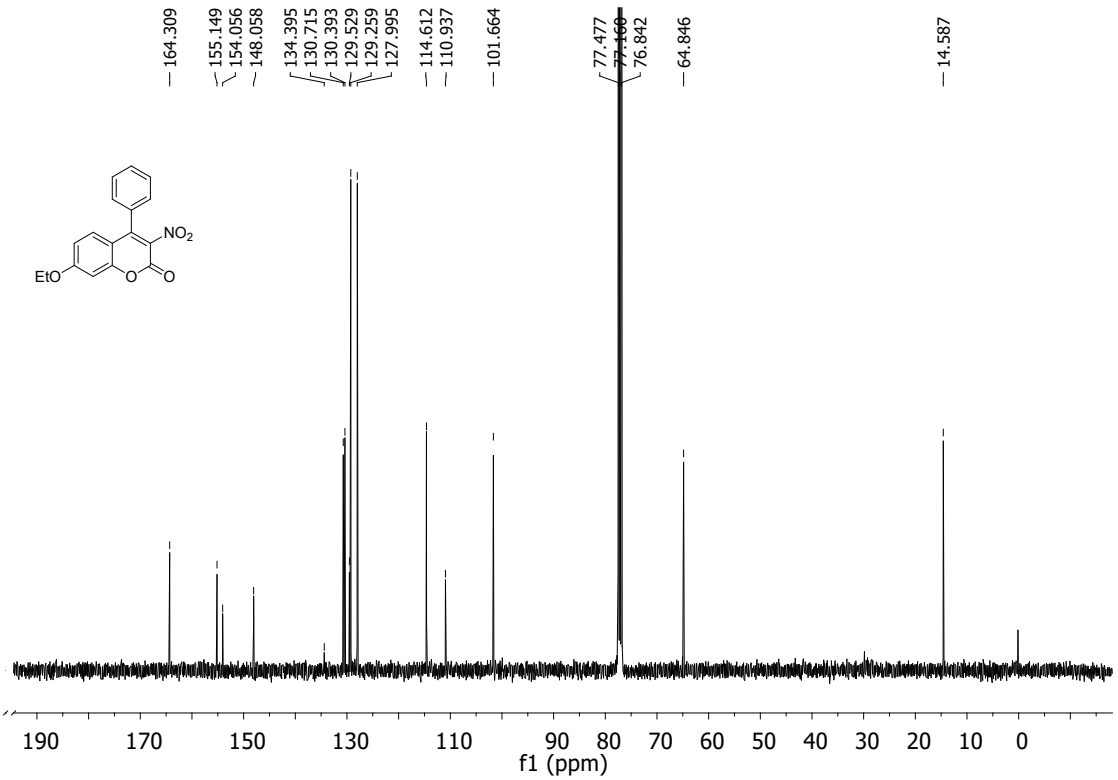

Fig. S25. ^{13}C NMR of 7-Ethoxy-3-nitro-4-phenyl-2H-chromen-2-one **(2k).**

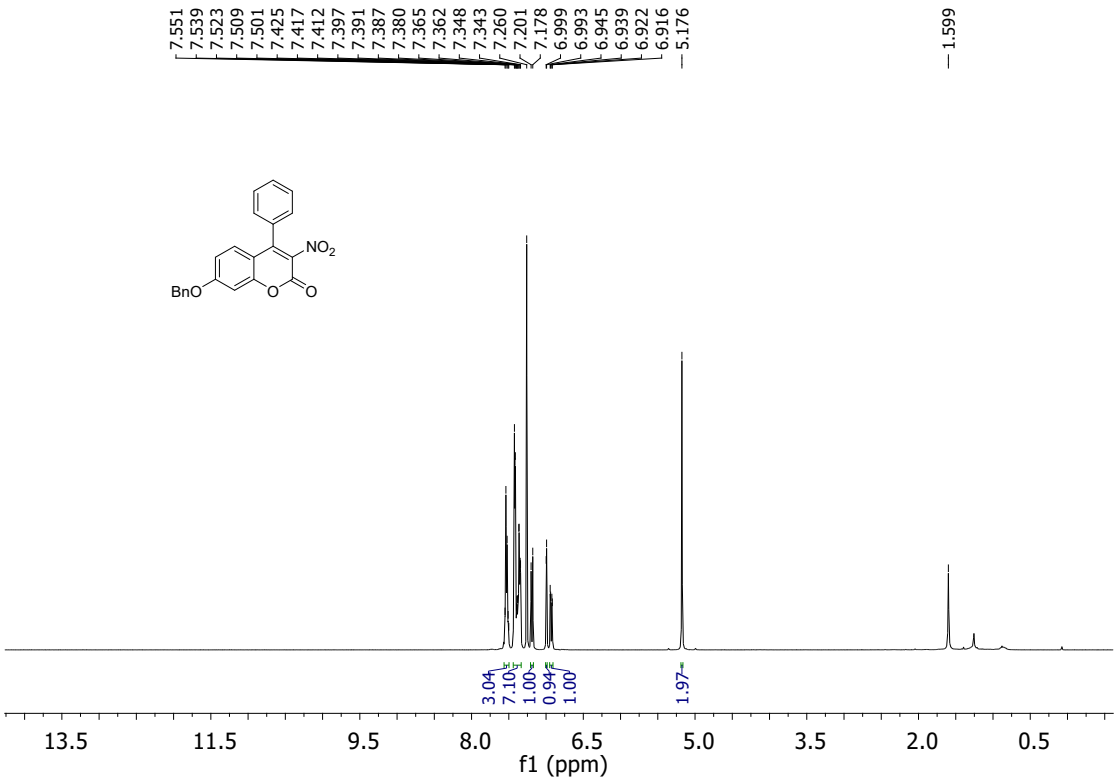

Fig. S26. ^{1}H NMR of 7-Benzyloxy-3-nitro-4-phenyl-2H-chromen-2-one **(2l).**

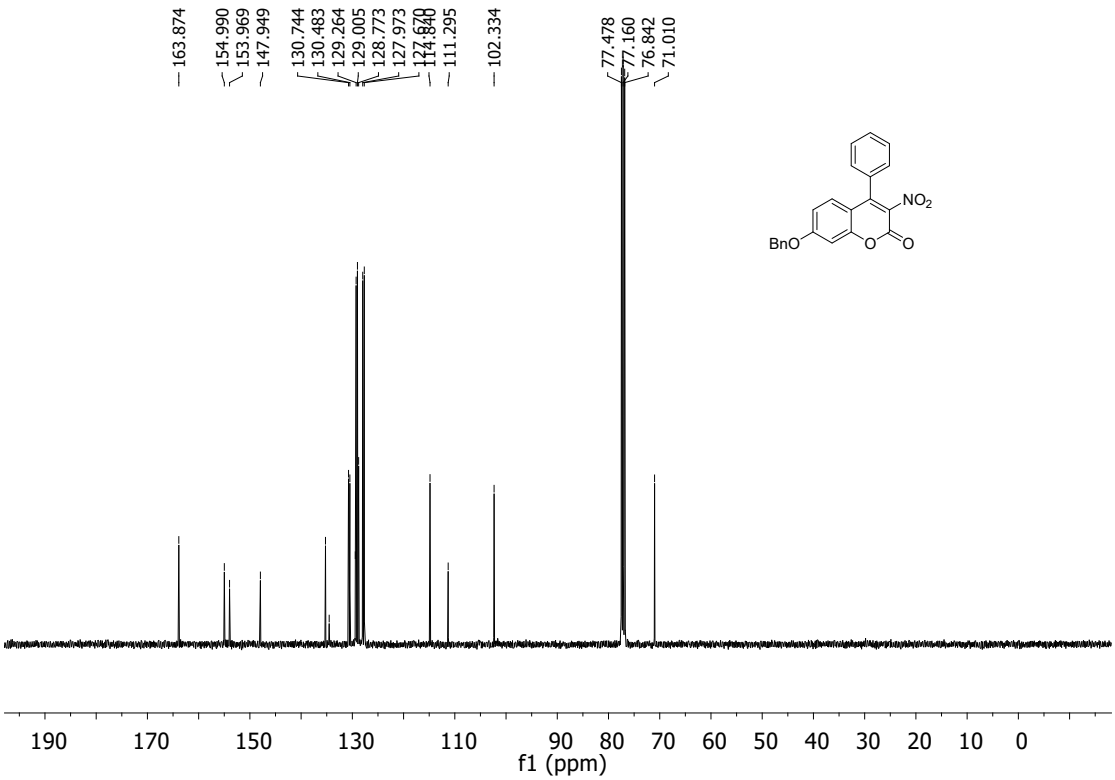

Fig. S27. ^{13}C NMR of 7-Benzyloxy-3-nitro-4-phenyl-2H-chromen-2-one **(2l).**

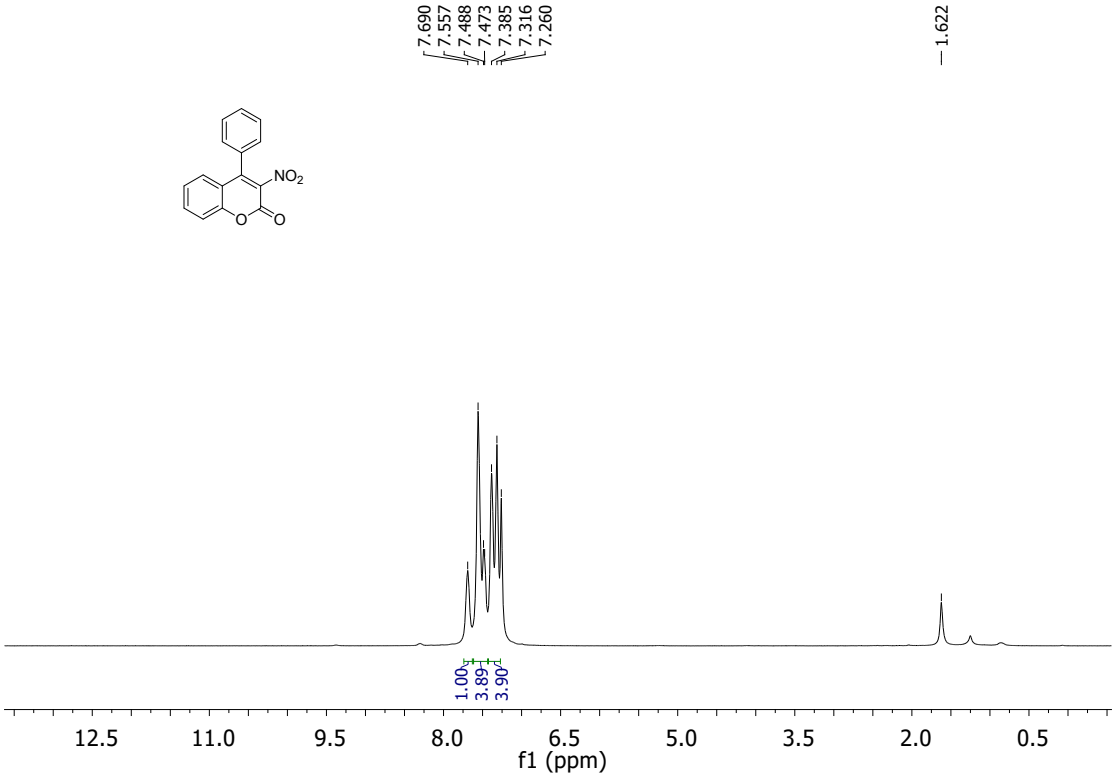

Fig. S28. ^{1}H NMR of 3-Nitro-4-phenyl-2H-chromen-2-one **(2m).**

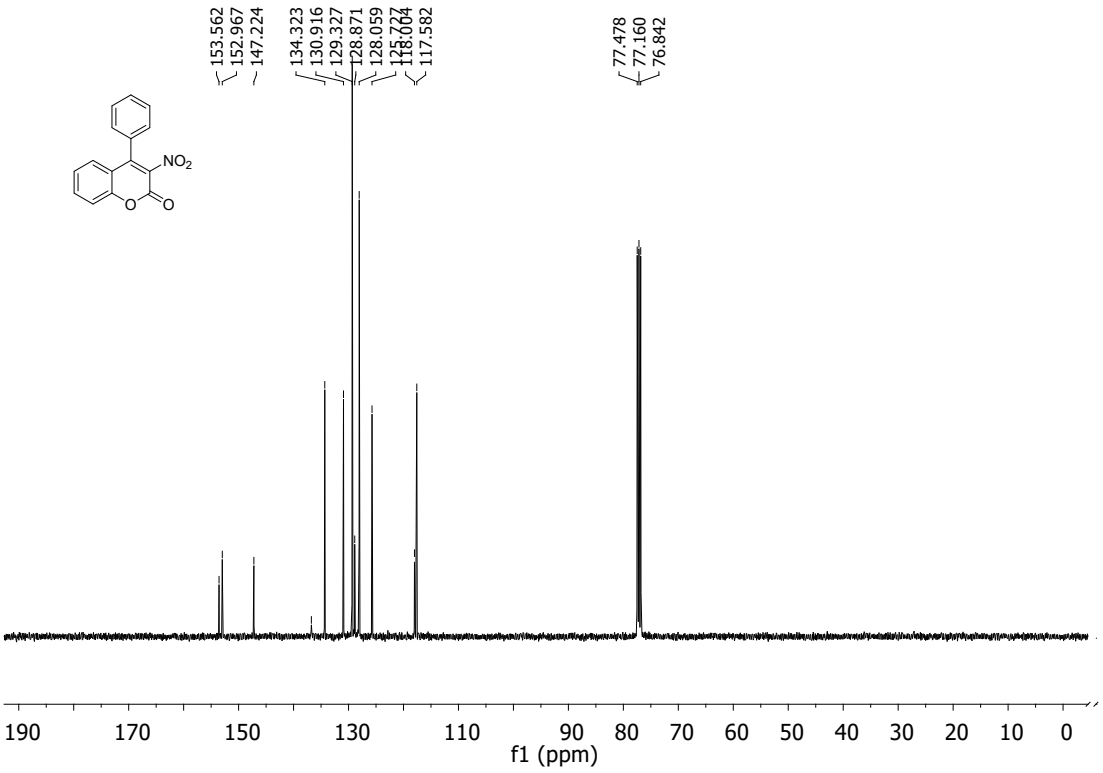

Fig. S29. ^{13}C NMR of 3-Nitro-4-phenyl-2H-chromen-2-one **(2m).**

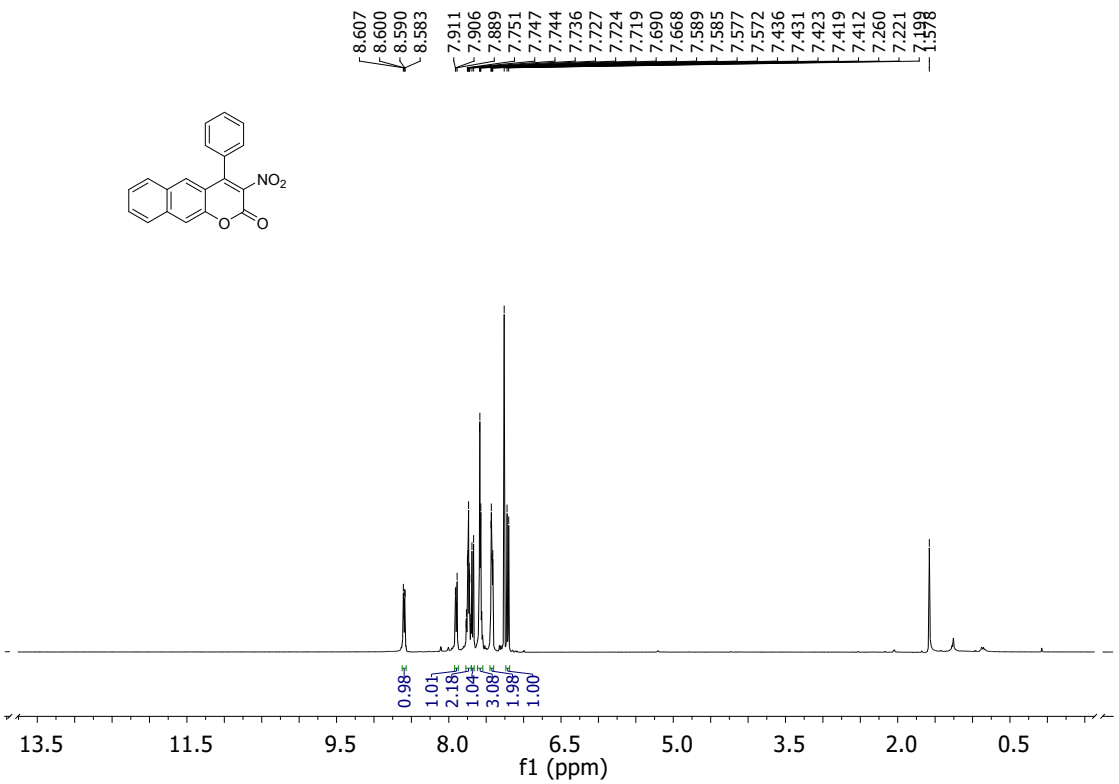

Fig. S30. ^{1}H NMR of 3-Nitro-4-phenyl-2H-benzo[g]chromen-2-one **(2n).**

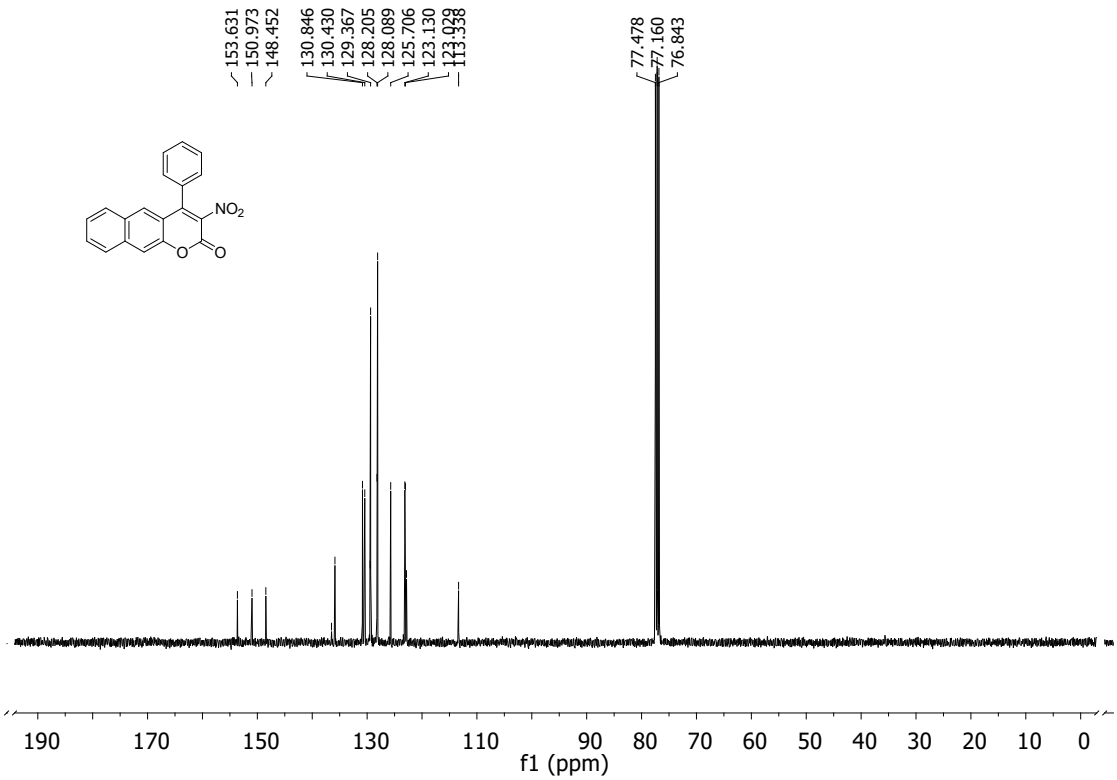

Fig. S31. ^{13}C NMR of 3-Nitro-4-phenyl-2H-benzo[g]chromen-2-one **(2n).**

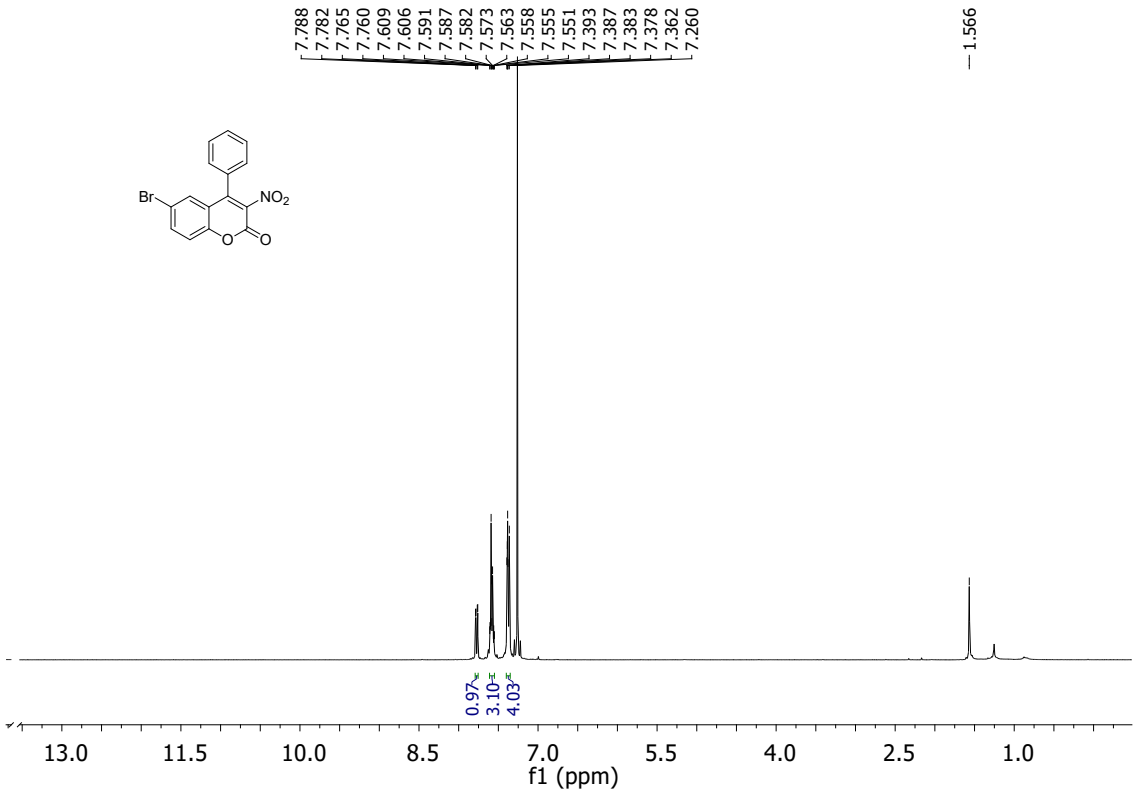

Fig. S32. ^{1}H NMR of 6-Bromo-3-nitro-4-phenyl-2H-chromen-2-one **(2q).**

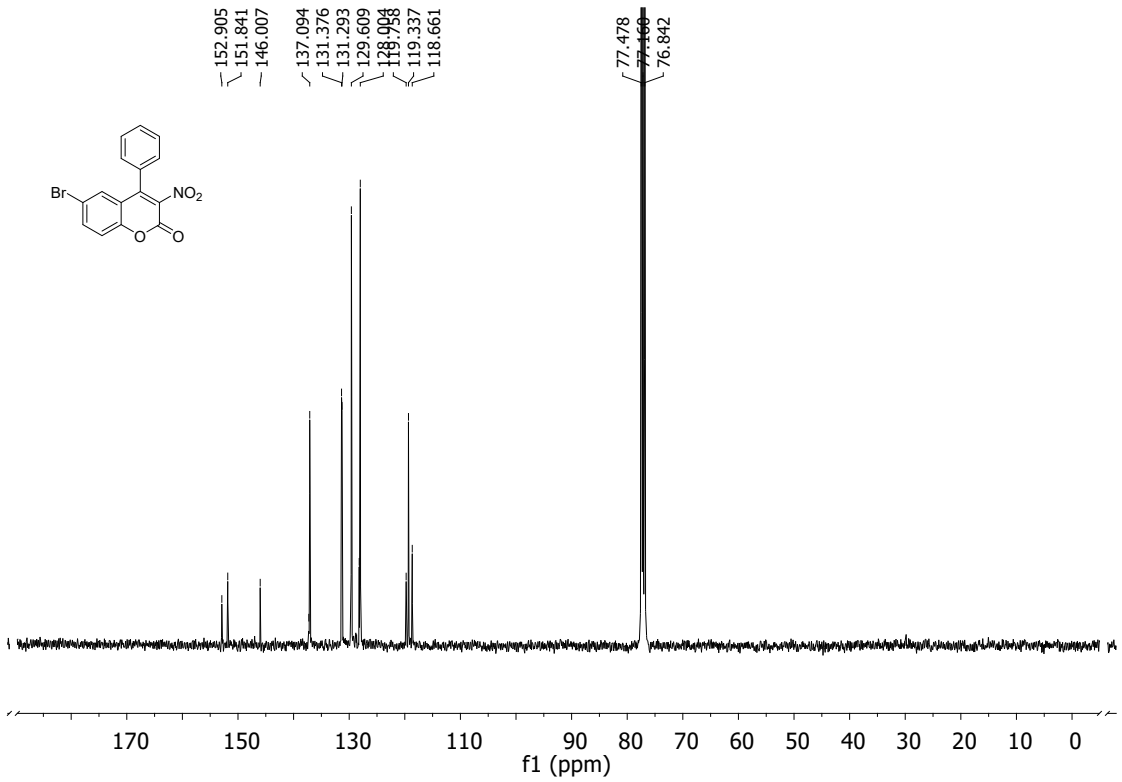

Fig. S33. ^{13}C NMR of 6-Bromo-3-nitro-4-phenyl-2H-chromen-2-one **(2q).**

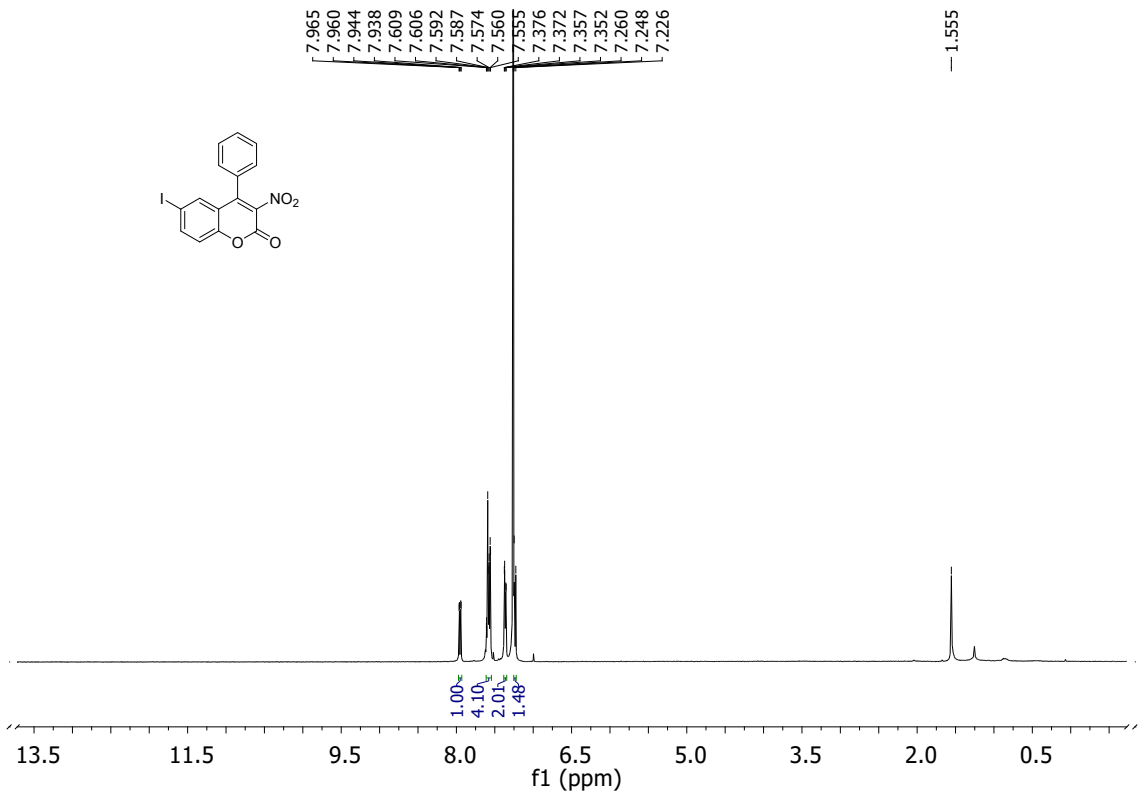

Fig. S34. ^{1}H NMR of 6-Iodo-3-nitro-4-phenyl-2H-chromen-2-one **(2r).**

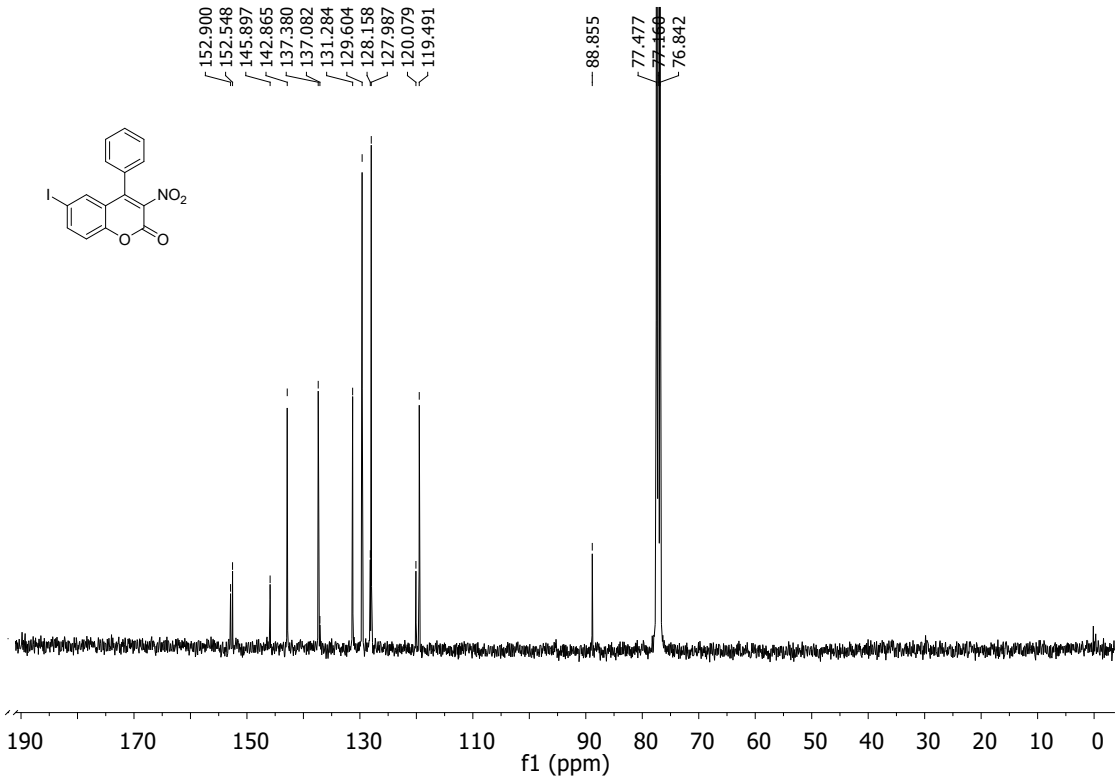

Fig. S35. ^{13}C NMR of 6-Iodo-3-nitro-4-phenyl-2H-chromen-2-one **(2r).**

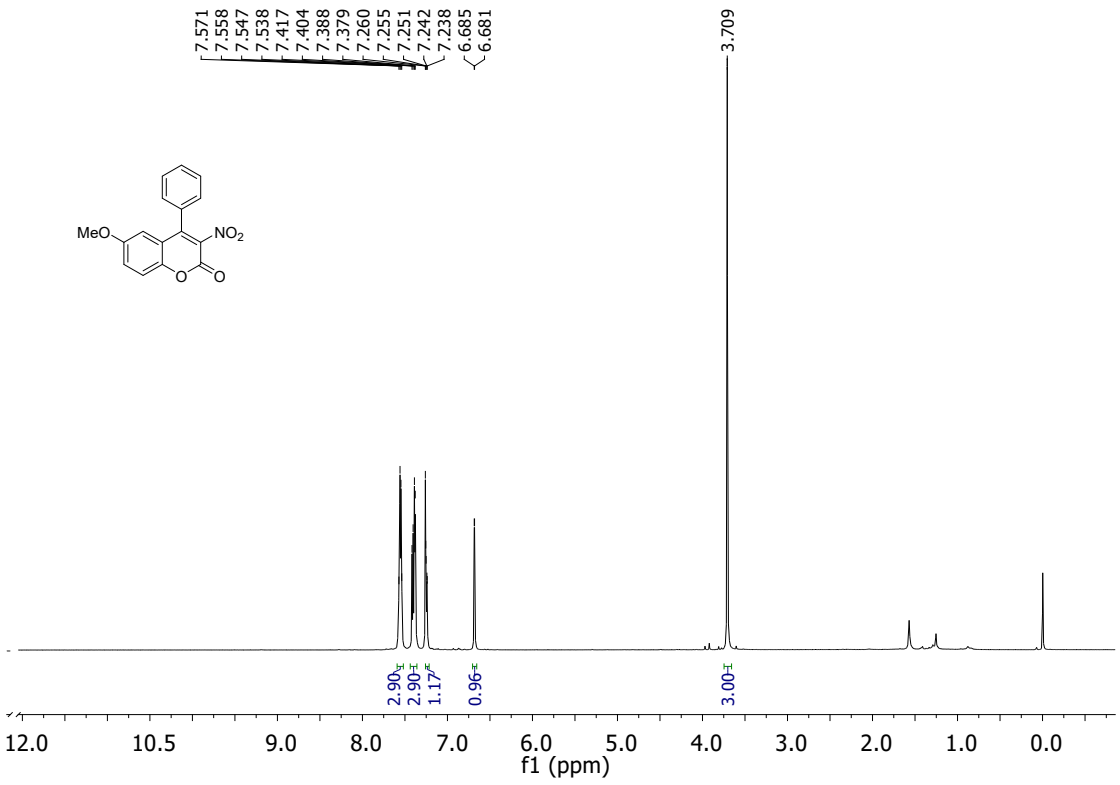

Fig. S36. ^{1}H NMR of 6-Methoxy-3-nitro-4-phenyl-2H-chromen-2-one **(2s).**

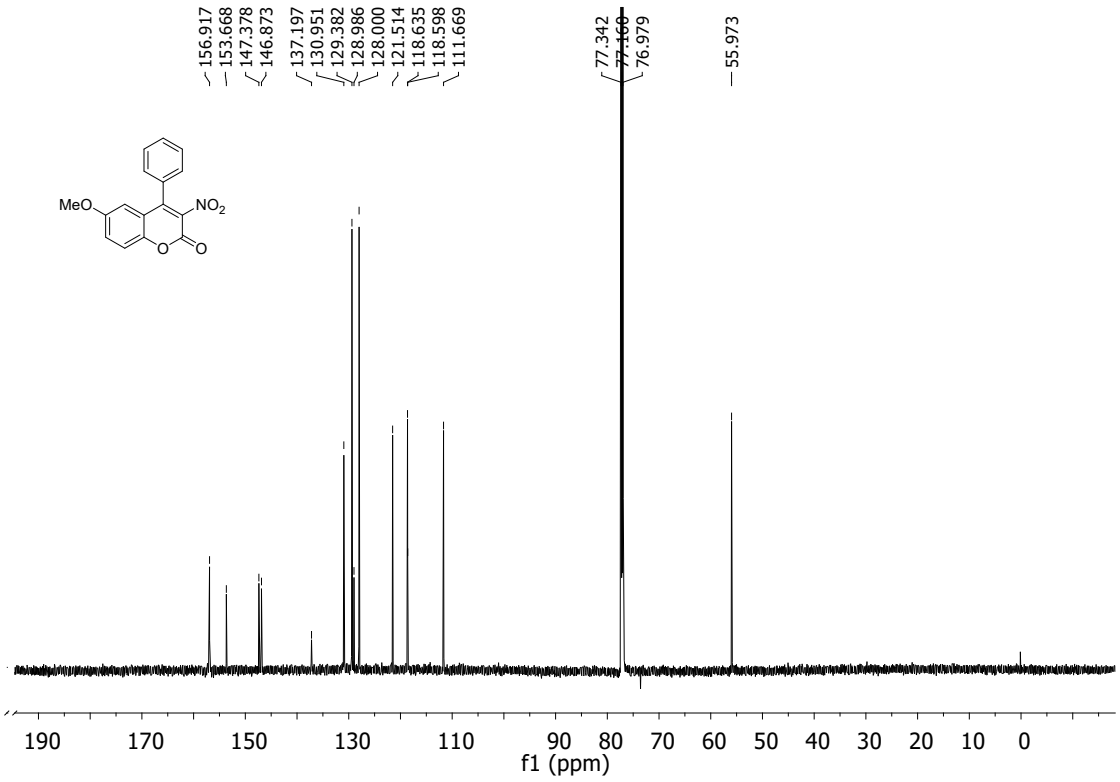

Fig. S37. ^{13}C NMR of 6-Methoxy-3-nitro-4-phenyl-2H-chromen-2-one **(2s).**

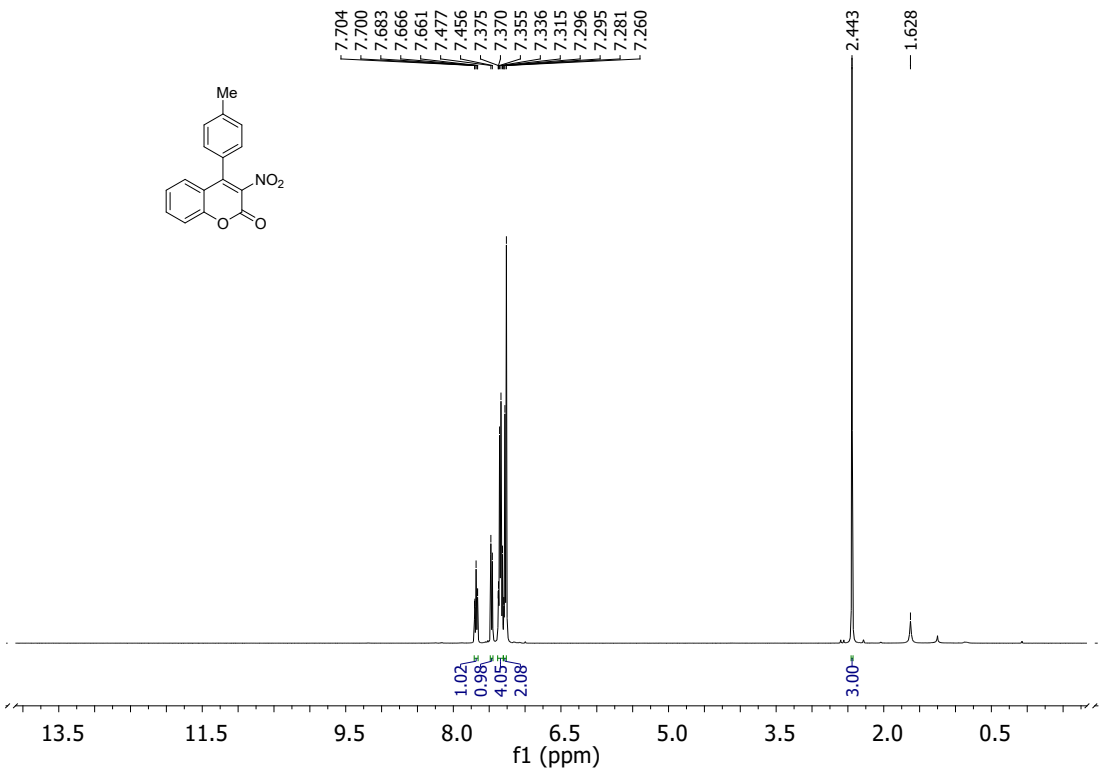

Fig. S38. [1]H NMR of 3-Nitro-4-(*p*-tolyl)-2H-chromen-2-one **(2u).**

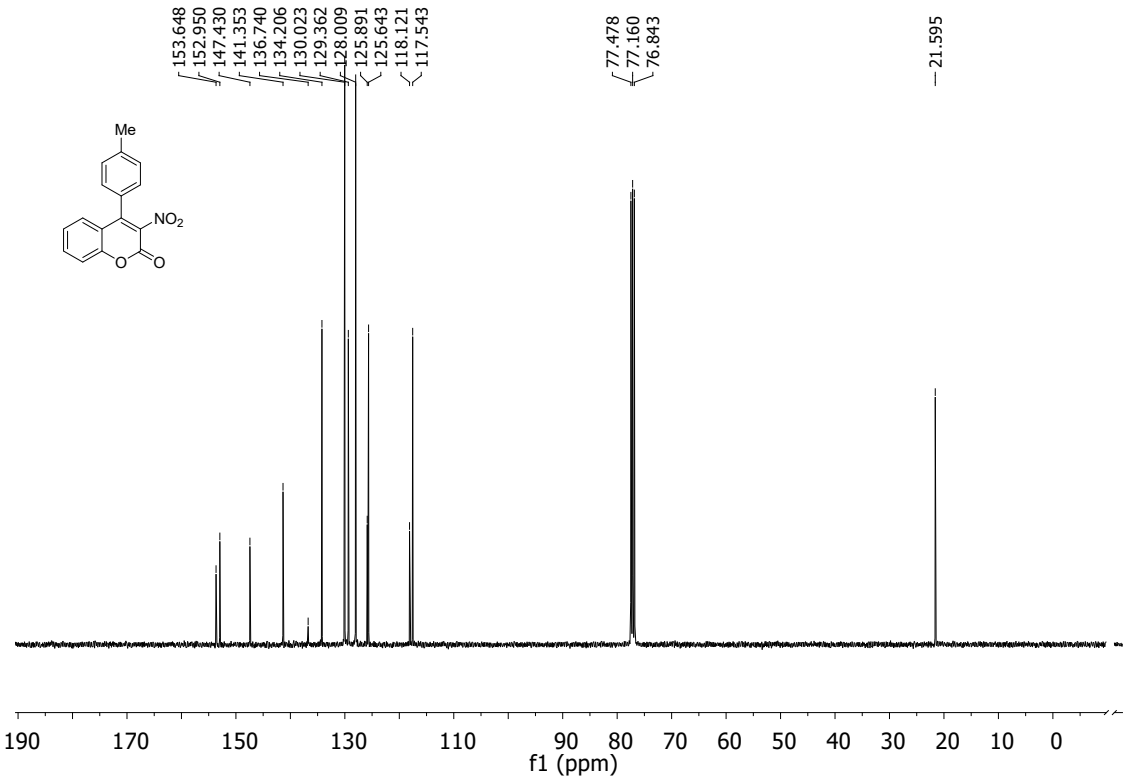

Fig. S39. [13]C NMR of 3-Nitro-4-(*p*-tolyl)-2H-chromen-2-one **(2u).**

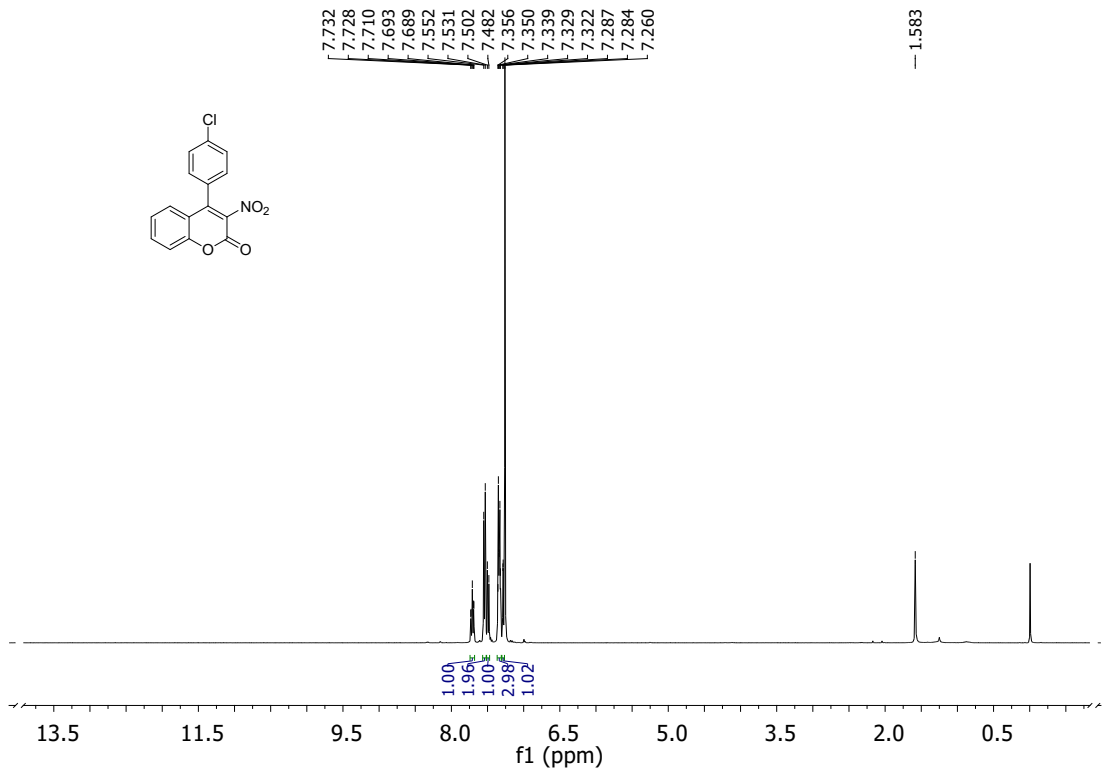

Fig. S40. [1]H NMR of 4-(4-Chlorophenyl)-3-nitro-2H-chromen-2-one **(2v).**

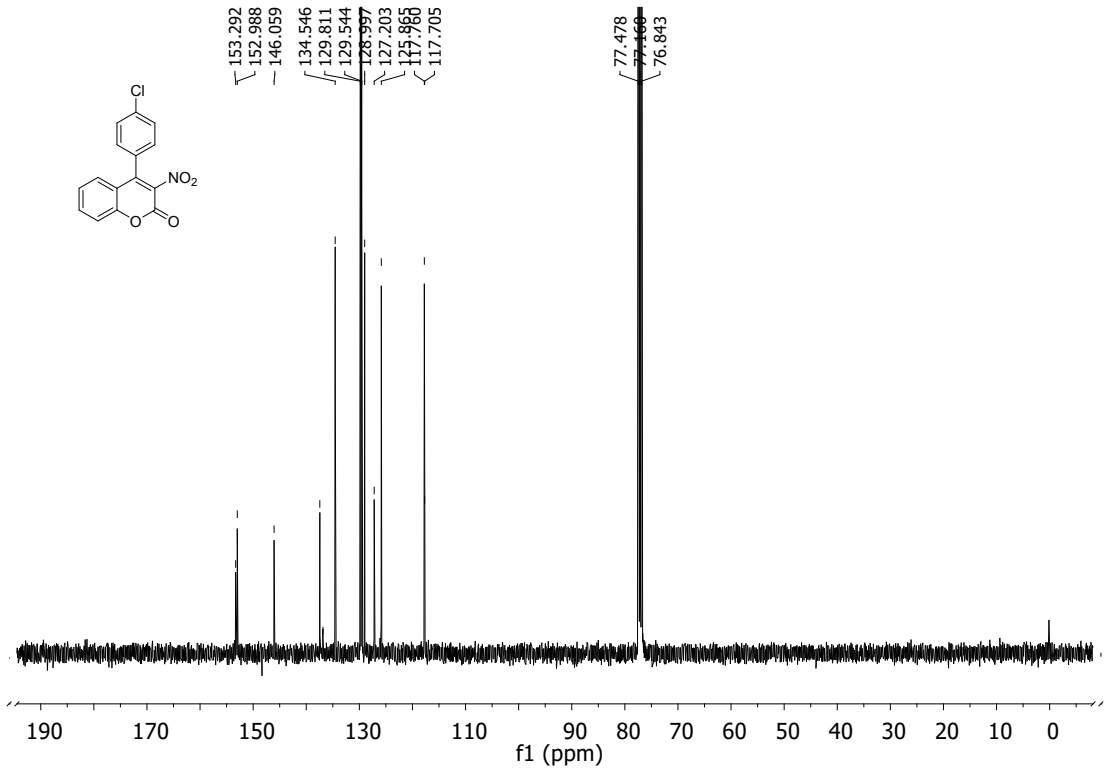

Fig. S41. [13]C NMR of 4-(4-Chlorophenyl)-3-nitro-2H-chromen-2-one **(2v).**

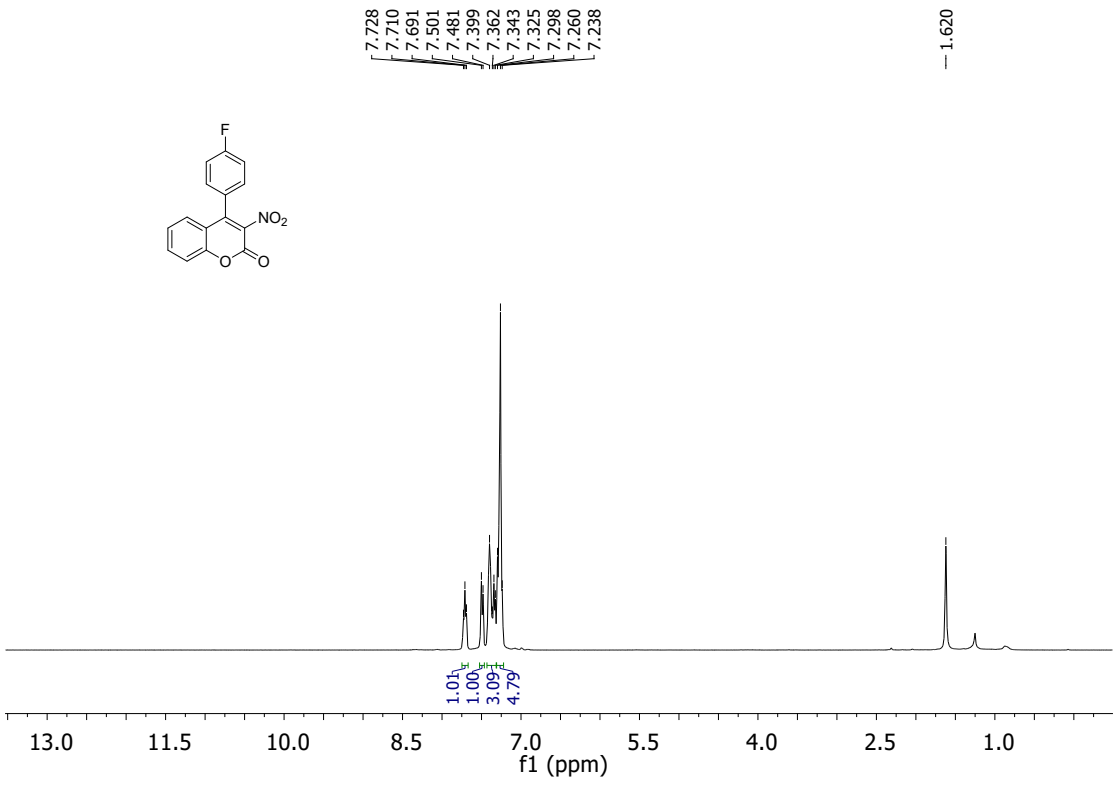

Fig. S42. [1]H NMR of 4-(4-Fluorophenyl)-3-nitro-2H-chromen-2-one (**SS 441**) **(2w).**

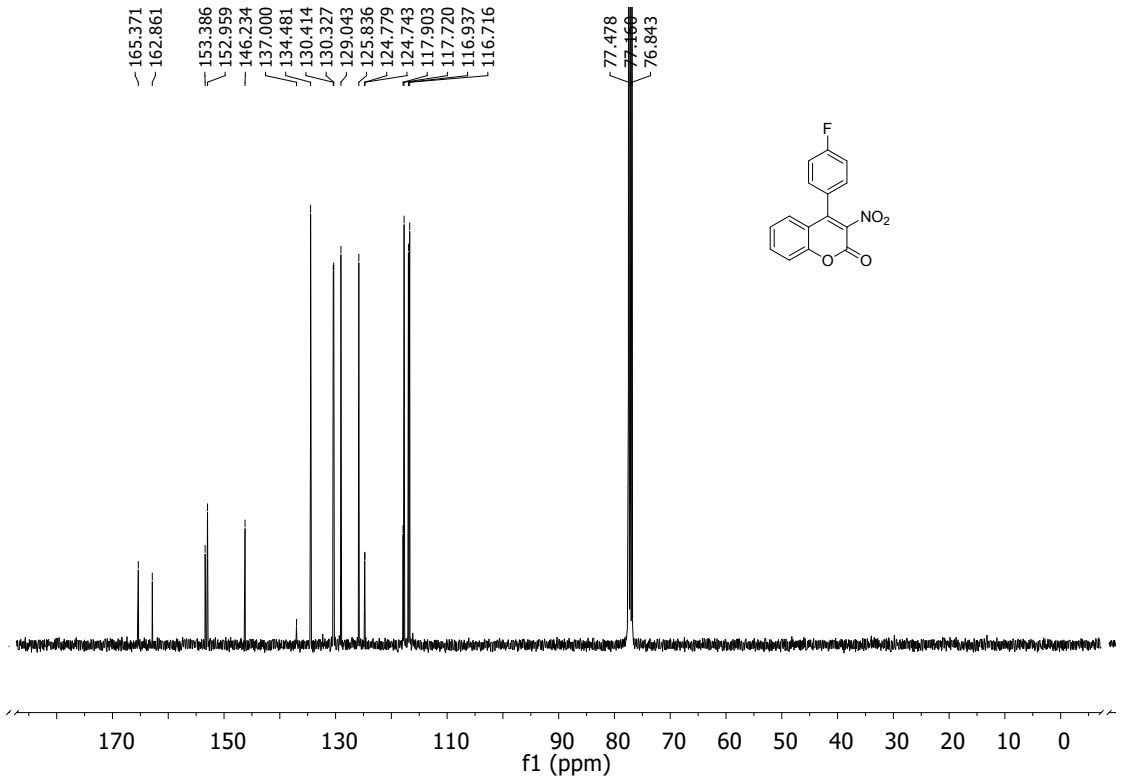

Fig. S43. [13]C NMR of 4-(4-Fluorophenyl)-3-nitro-2H-chromen-2-one **(2w).**

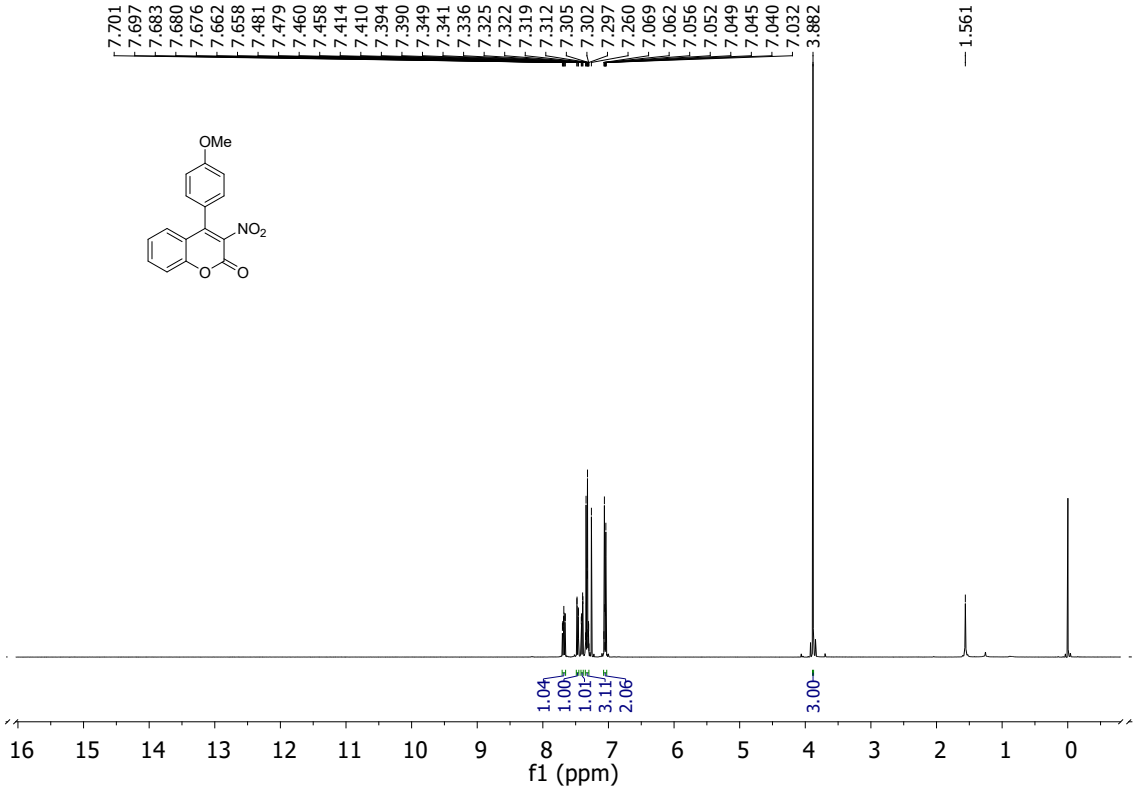

Fig. S44. [1]H NMR of 4-(4-Methoxyphenyl)-3-nitro-2H-chromen-2-one **(2x).**

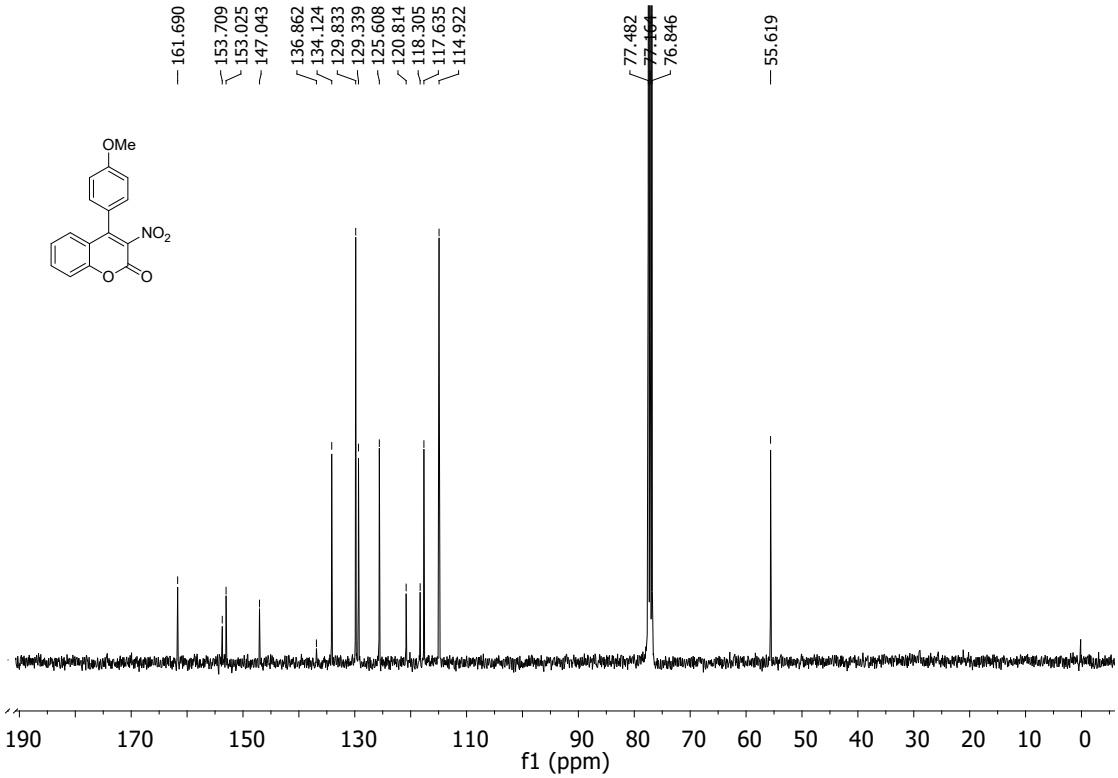

Fig. S45. ^{13}C NMR of 4-(4-Methoxyphenyl)-3-nitro-2H-chromen-2-one **(2x).**

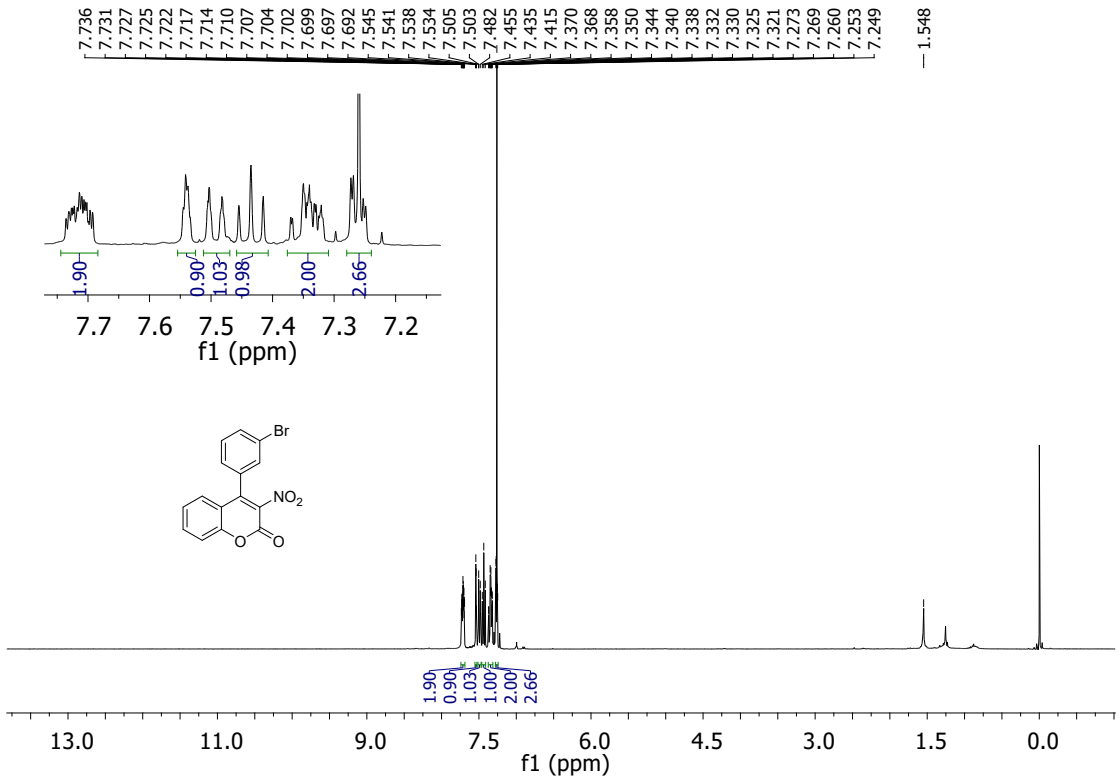

Fig. S46. ^{1}H NMR of 4-(3-Bromophenyl)-3-nitro-2H-chromen-2-one **(2y).**

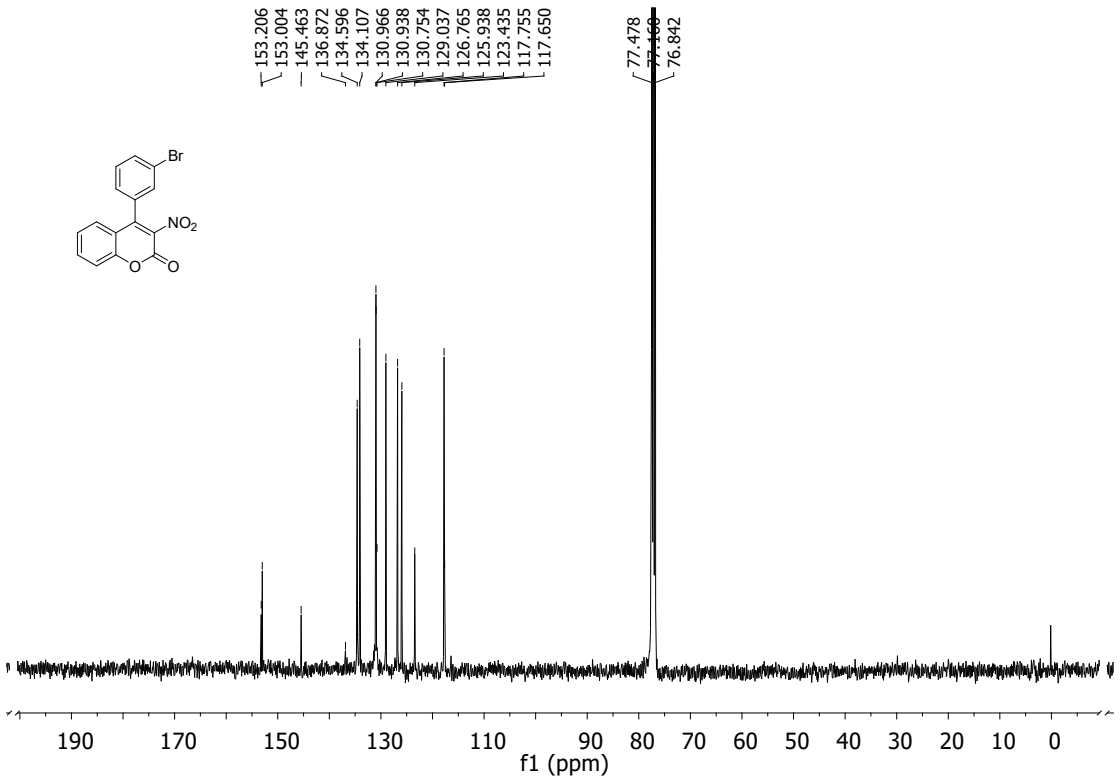

Fig. S47. ^{13}C NMR of 4-(3-Bromophenyl)-3-nitro-2H-chromen-2-one **(2y).**

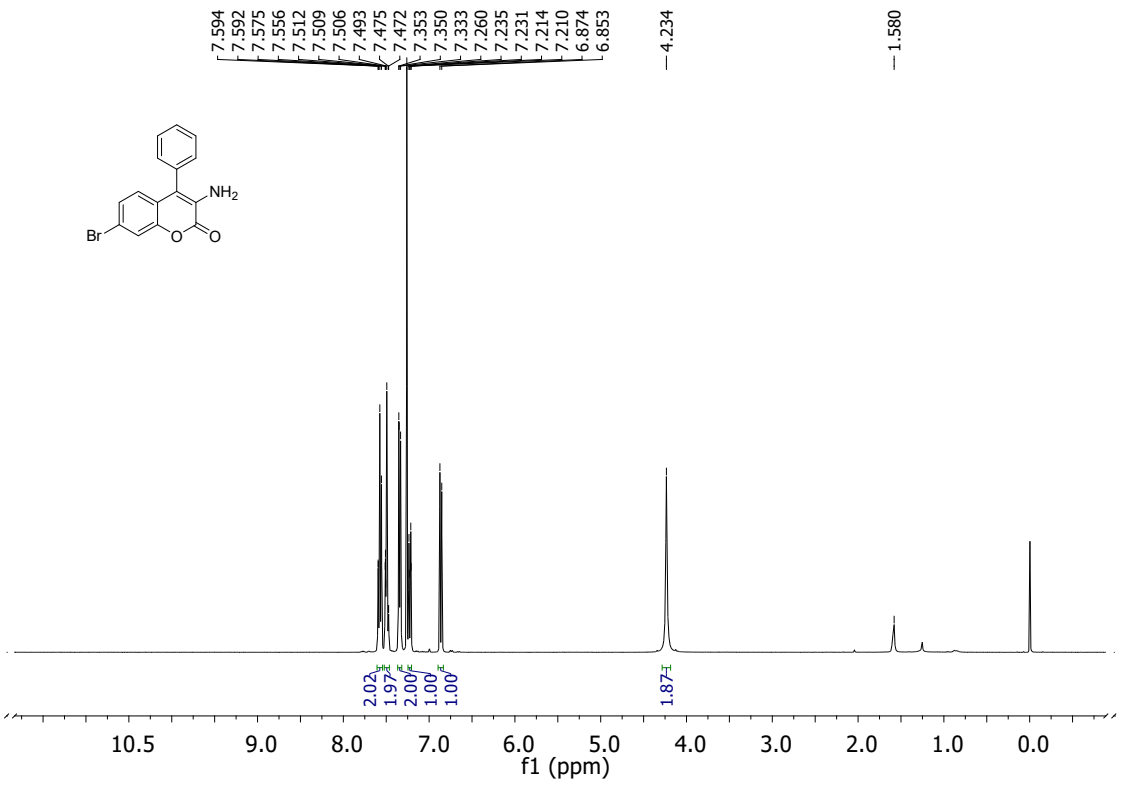

Fig. S48. [1]H NMR of 3-Amino-7-bromo-4-phenyl-2H-chromen-2-one **(7).**

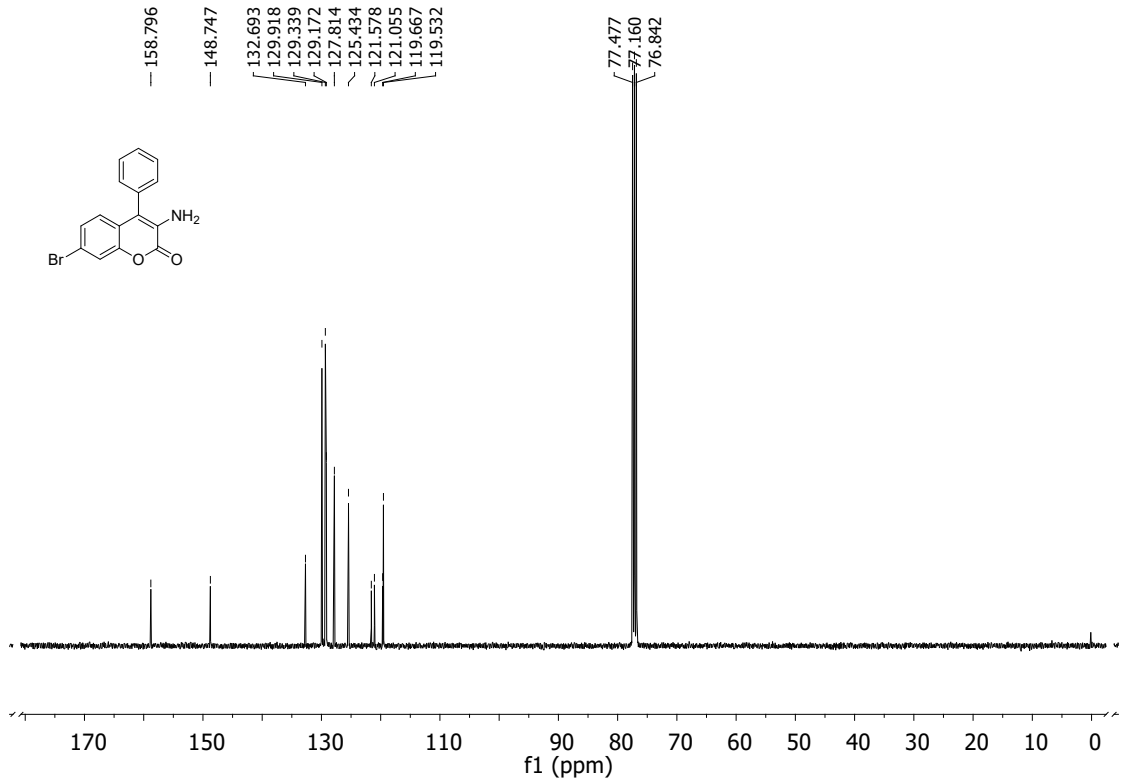

Fig. S49. [13]C NMR of 3-Amino-7-bromo-4-phenyl-2H-chromen-2-one **(7).**

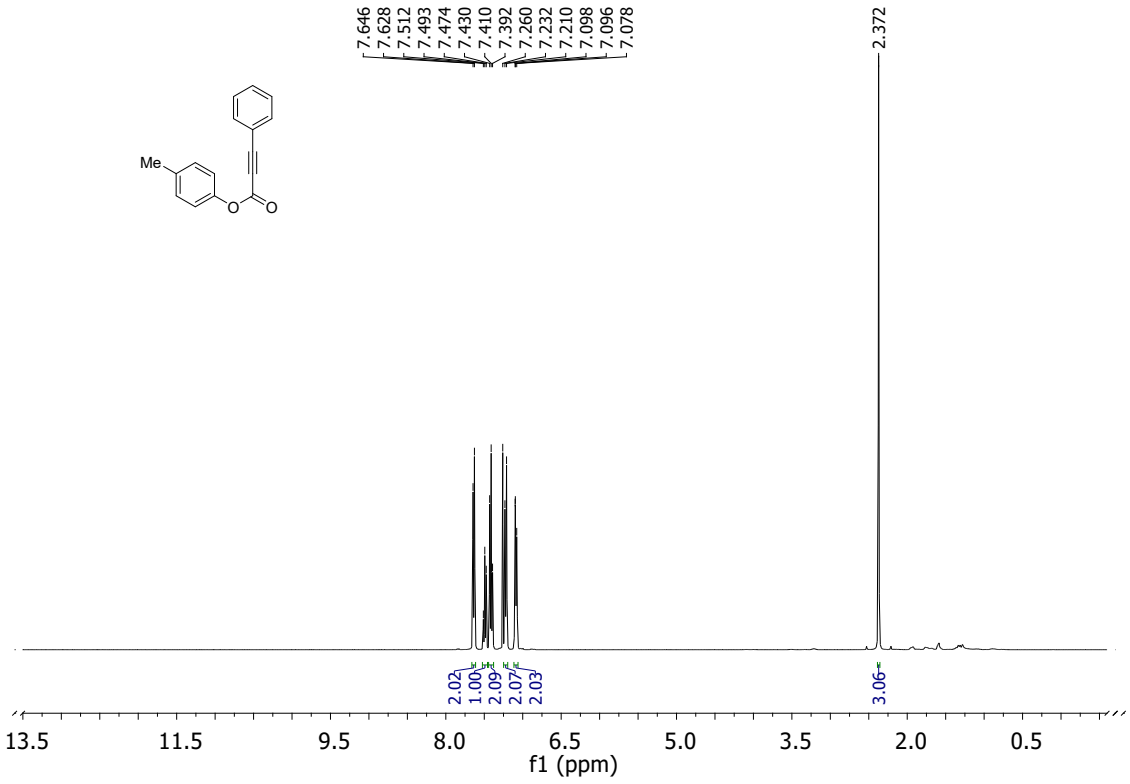

Fig. S50. [1]H NMR of *p*-tolyl 3-phenylpropiolate **(1a).**

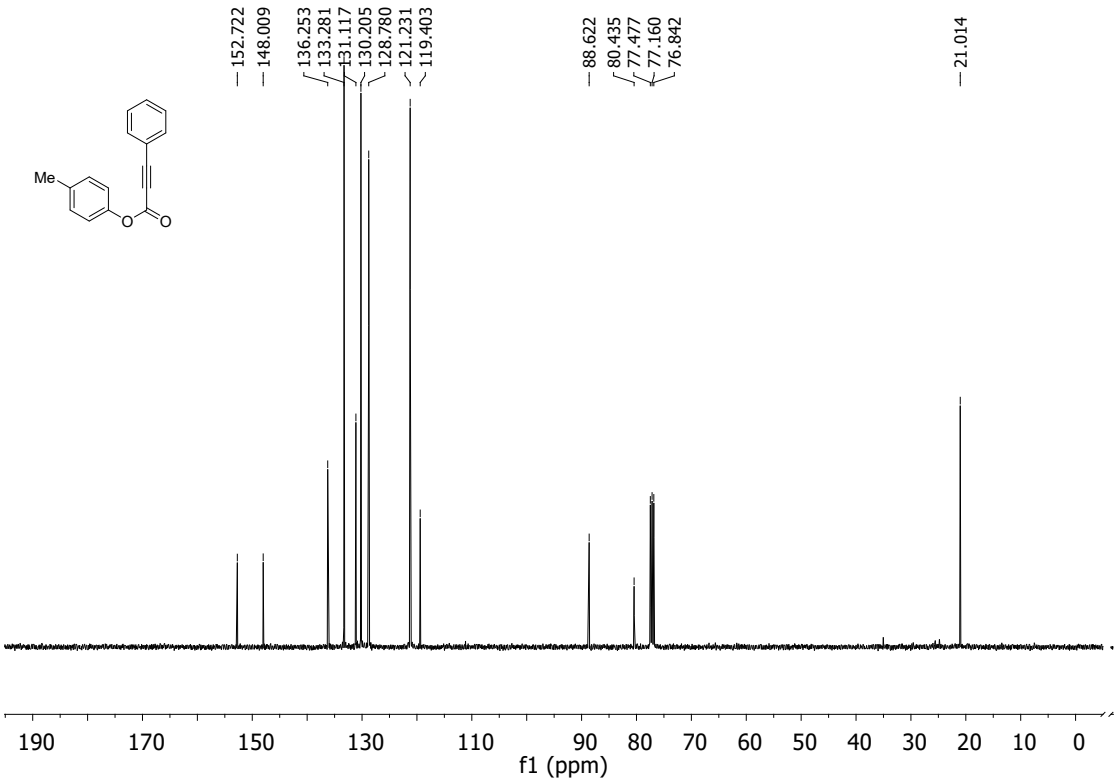

Fig. S51. ^{13}C NMR of *p*-tolyl 3-phenylpropiolate **(1a).**

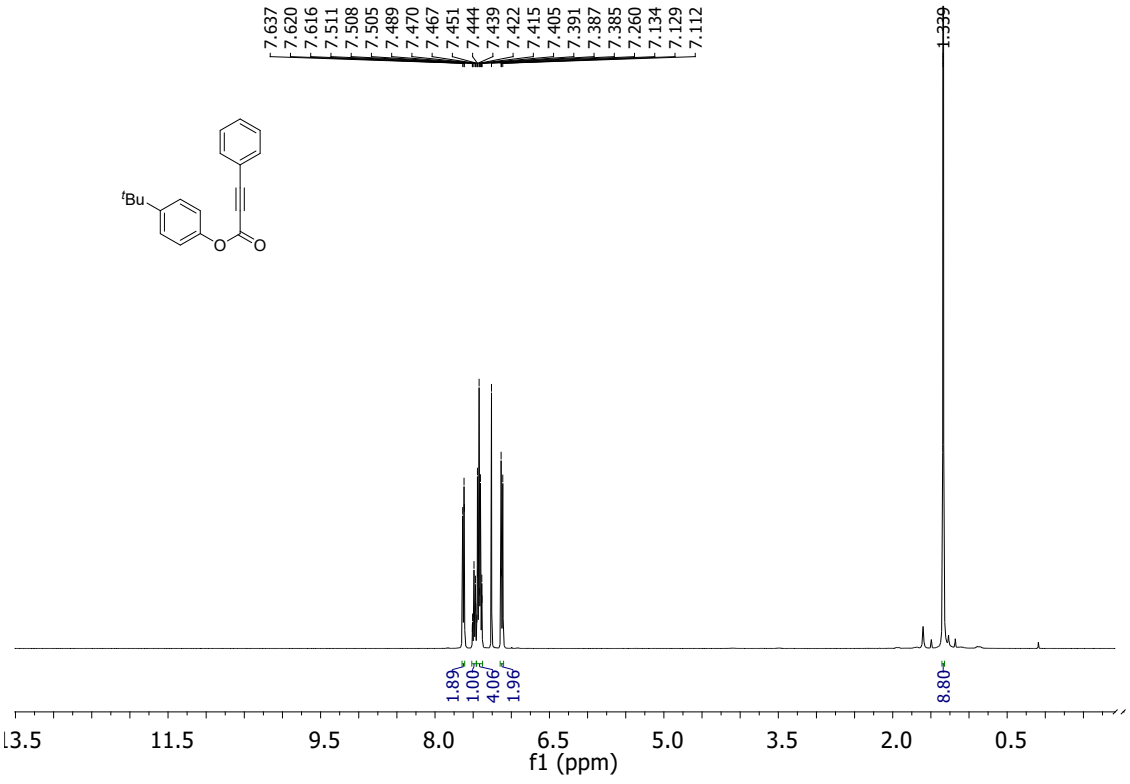

Fig. S52. ^{1}H NMR of 4-(*tert*-butyl)phenyl 3-phenylpropiolate **(1b).**

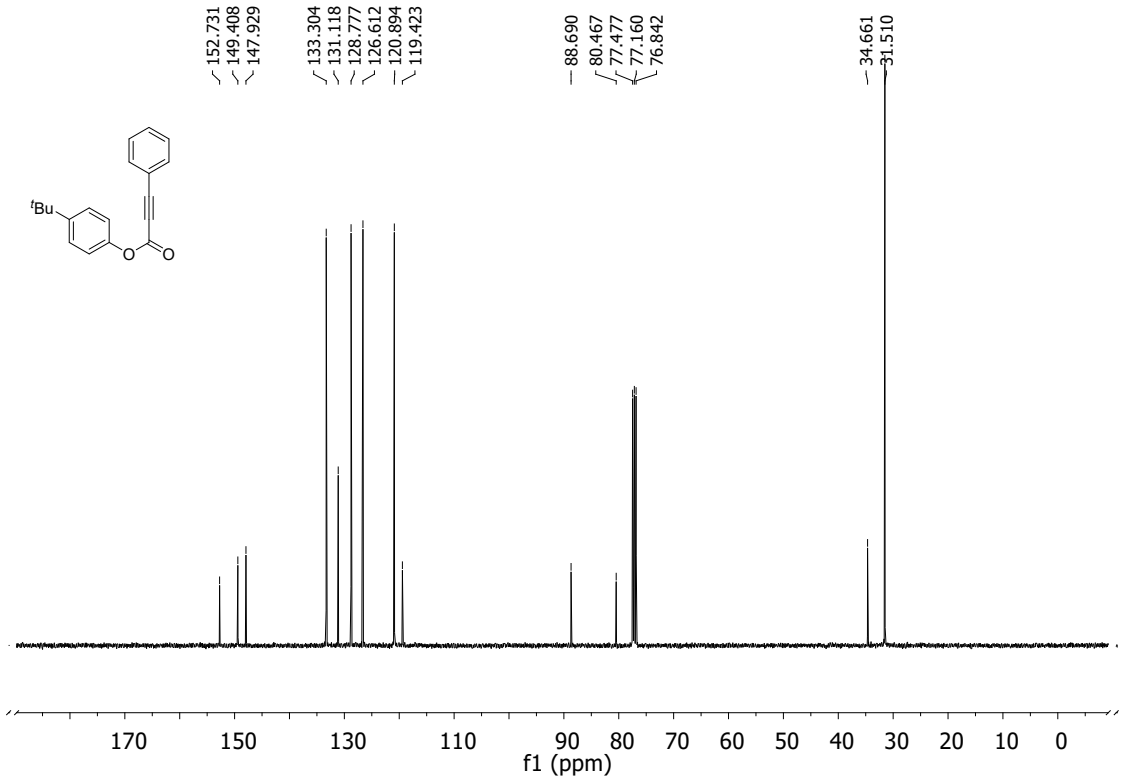

Fig. S53. ^{13}C NMR of 4-(*tert*-butyl)phenyl 3-phenylpropiolate **(SS 391) (1b).**

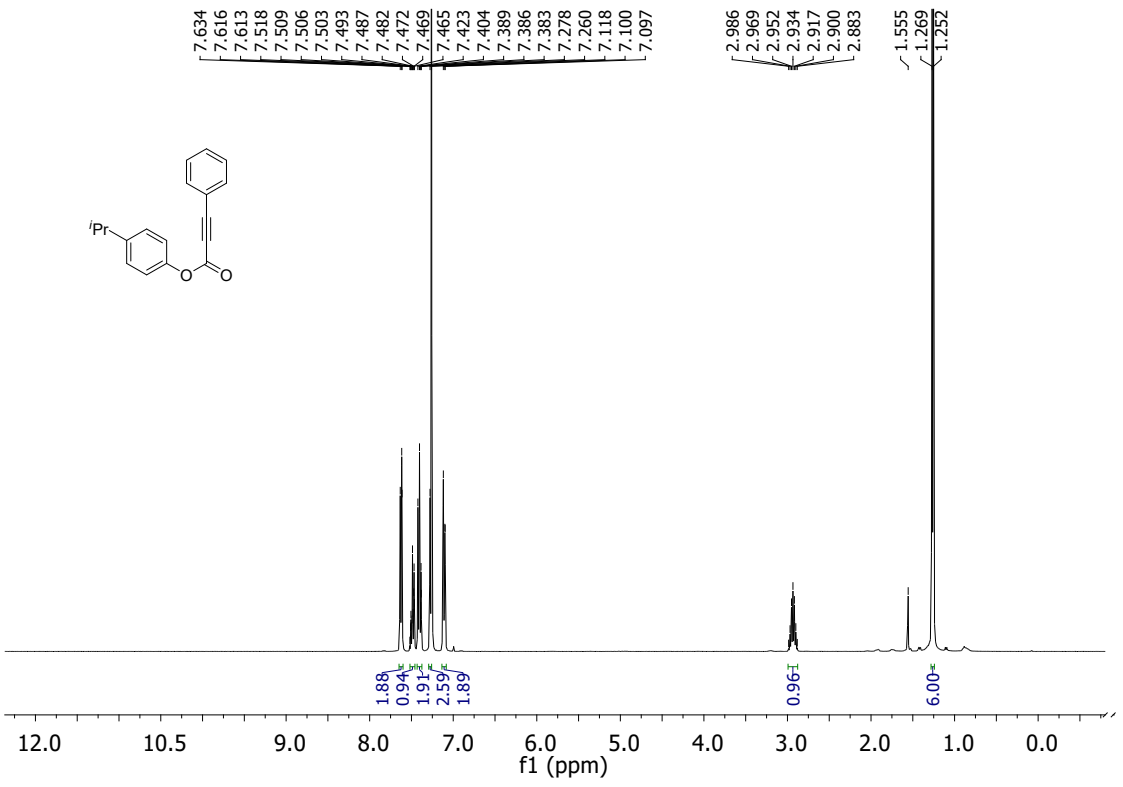

Fig. S54. ^{1}H NMR of 4-isopropylphenyl 3-phenylpropiolate **(1c).**

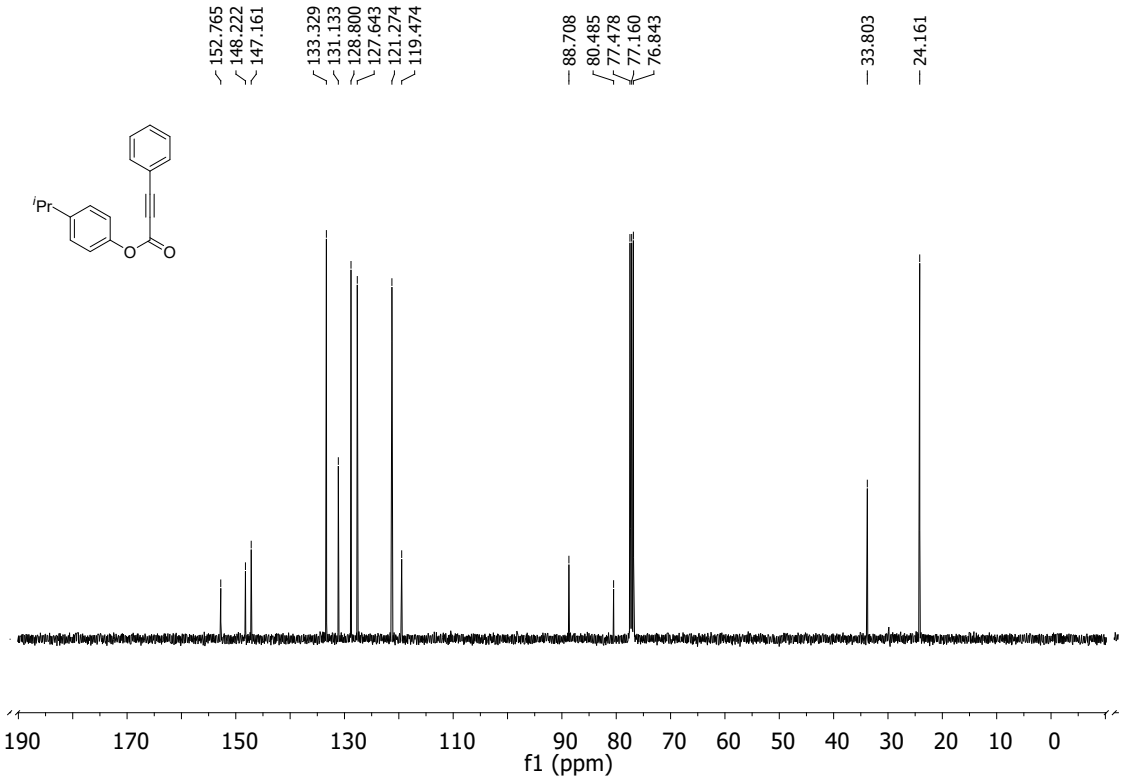

Fig. S55. ^{13}C NMR of 4-Isopropylphenyl 3-phenylpropiolate **(1c).**

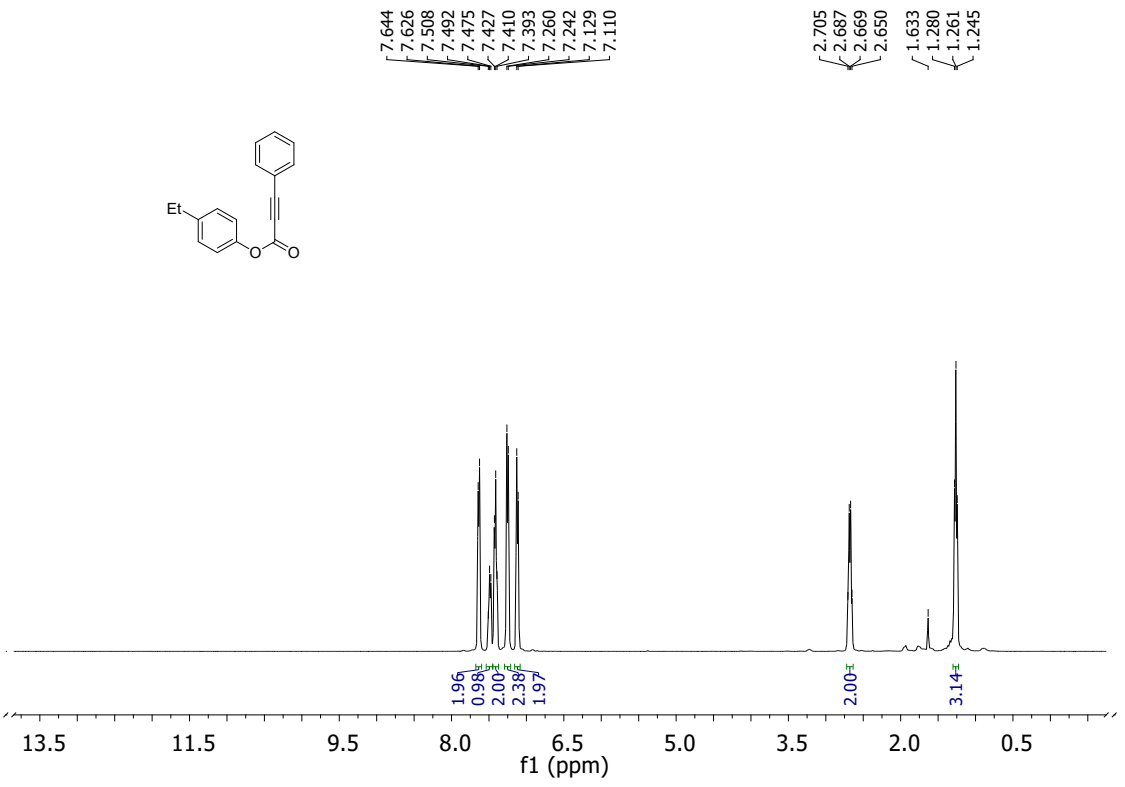

Fig. S56. ^{1}H NMR of 4-Ethylphenyl 3-phenylpropiolate **(1d).**

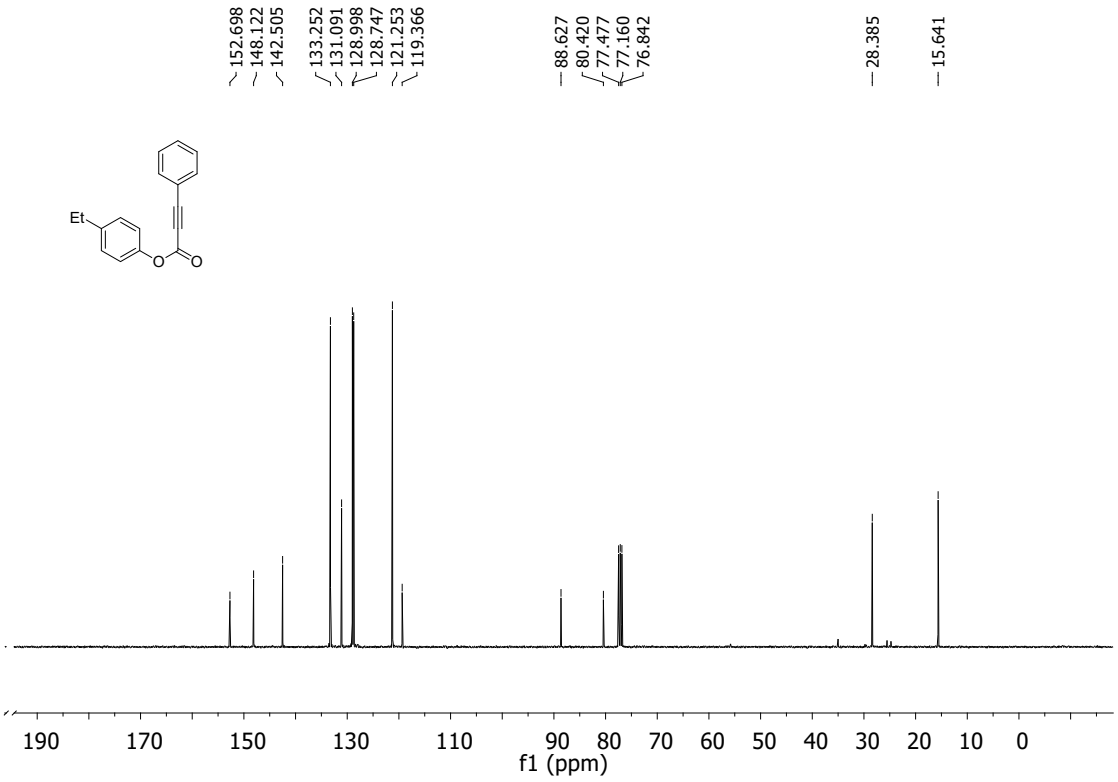

Fig. S57. ^{13}C NMR of 4-Ethylphenyl 3-phenylpropiolate **(1d).**

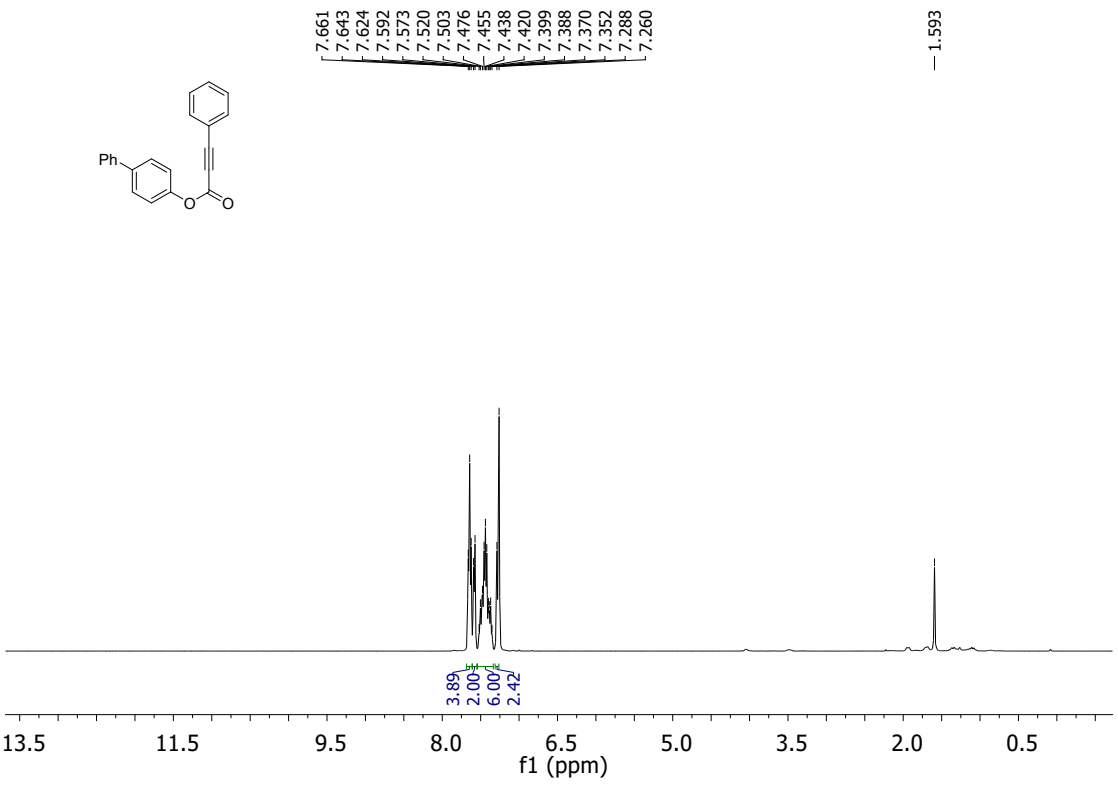

Fig. S58. ^{1}H NMR of [1,1'-Biphenyl]-4-yl 3-phenylpropiolate **(1e).**

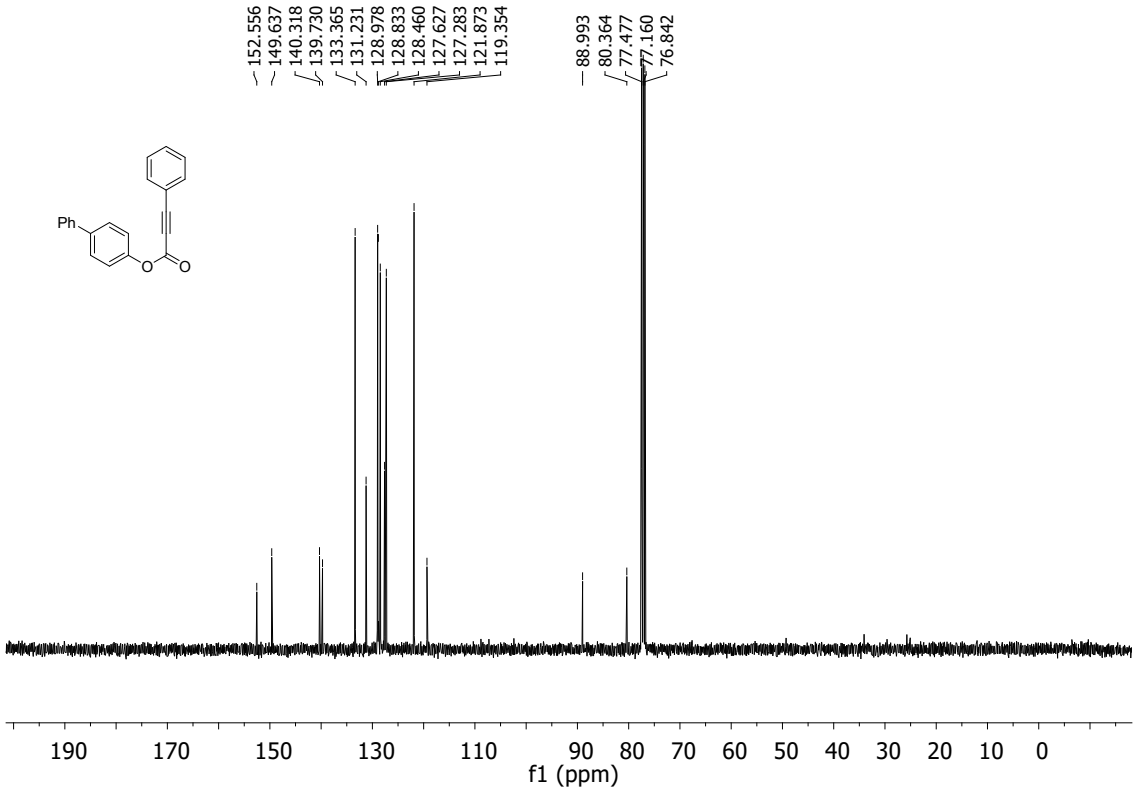

Fig. S59. ^{13}C NMR of [1,1'-Biphenyl]-4-yl 3-phenylpropiolate **(1e).**

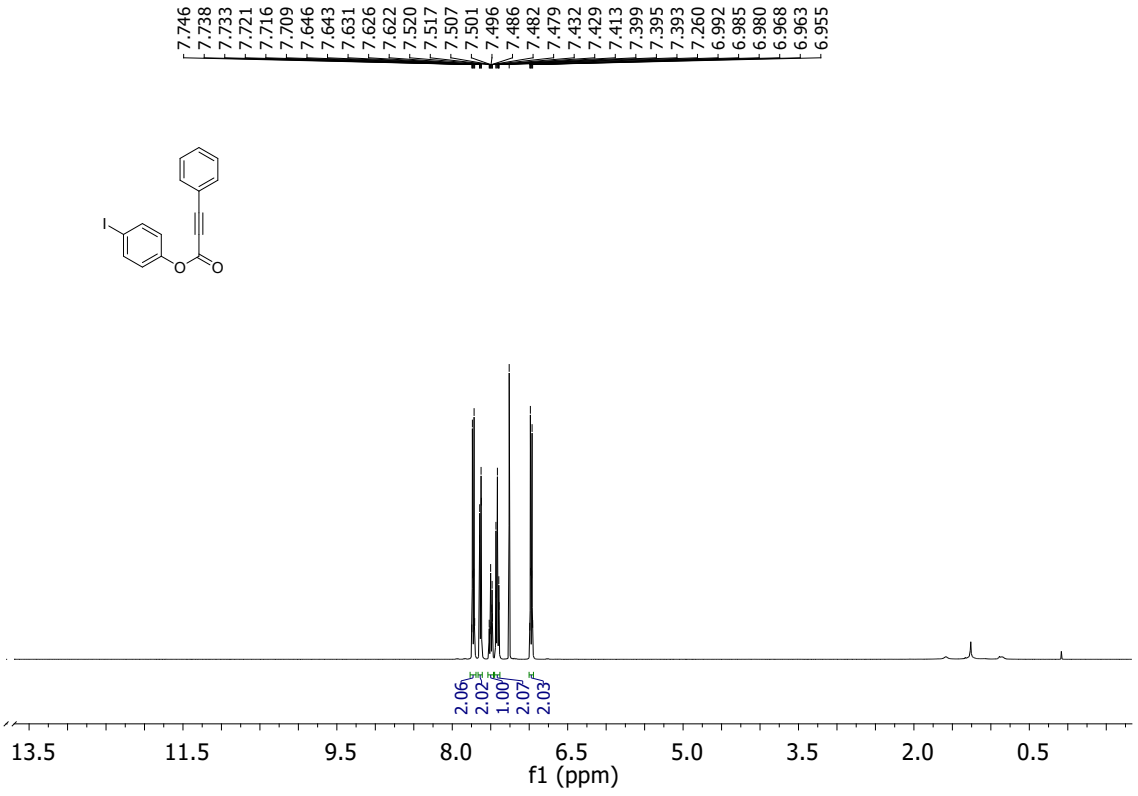

Fig. S60. ^{1}H NMR of 4-Iodophenyl 3-phenylpropiolate **(1f).**

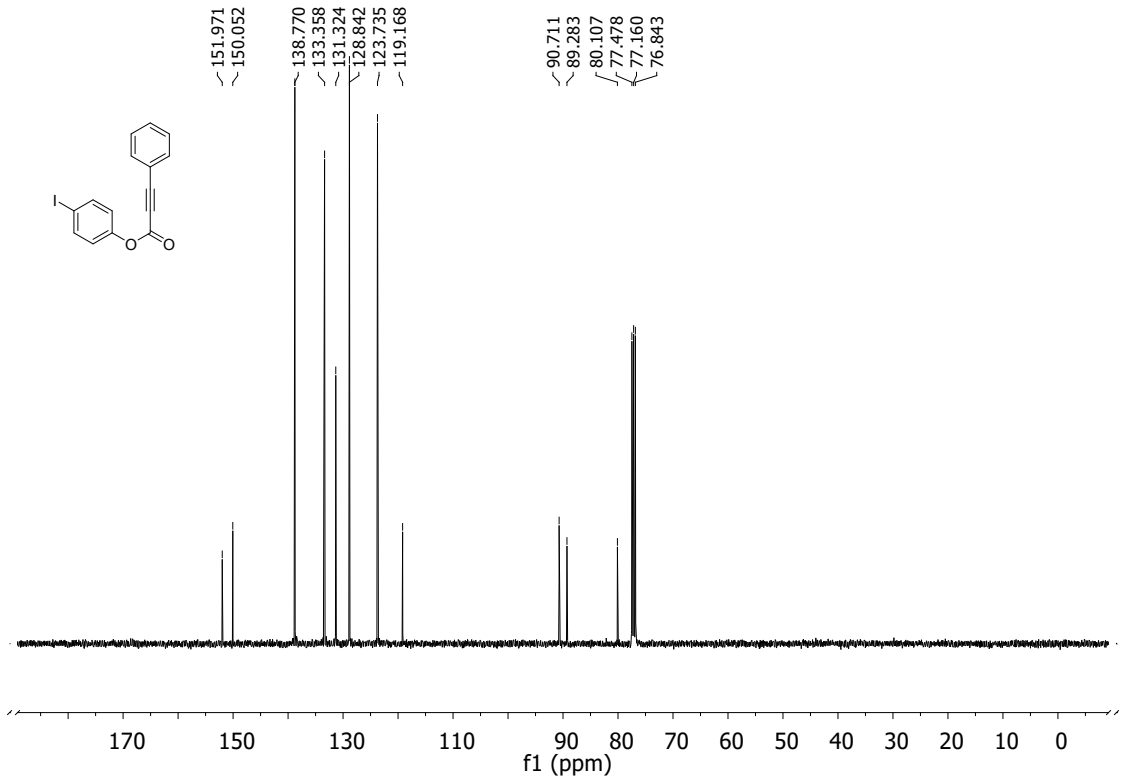

Fig. S61. ^{13}C NMR of 4-Iodophenyl 3-phenylpropiolate **(1f).**

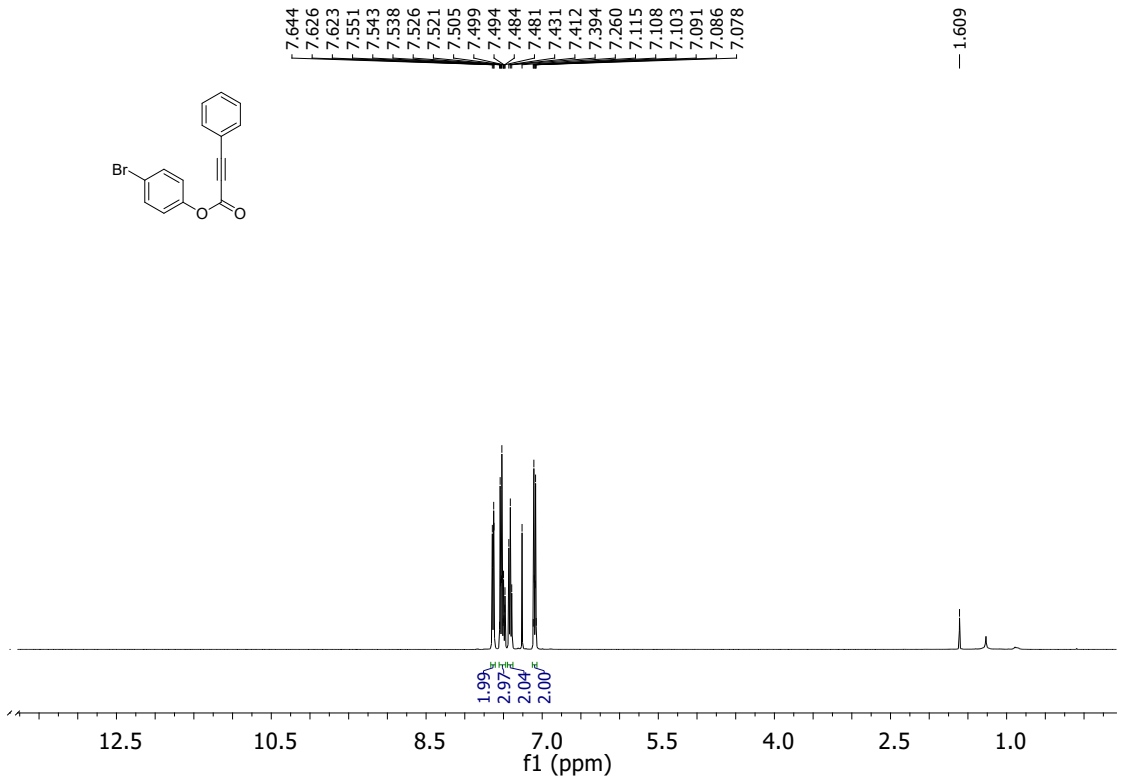

Fig. S62. ^{1}H NMR of 4-Bromophenyl 3-phenylpropiolate **(1g).**

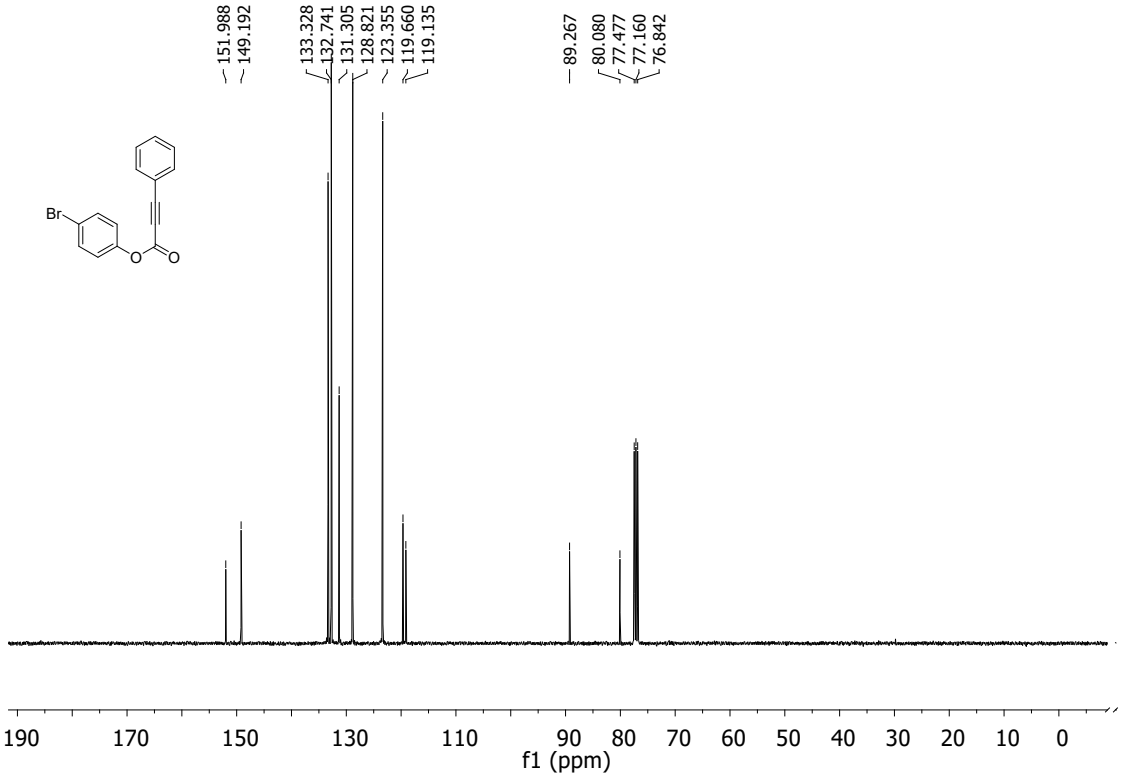

Fig. S63. ^{13}C NMR of 4-Bromophenyl 3-phenylpropiolate **(1g).**

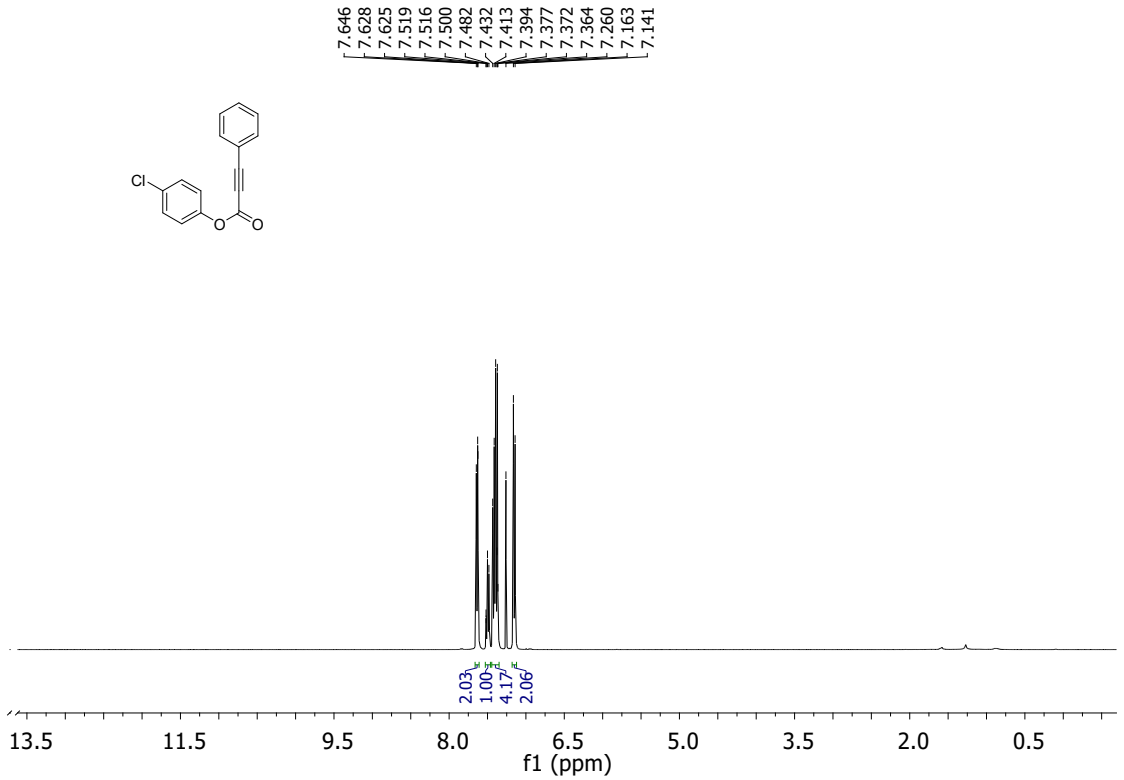

Fig. S64. [1]H NMR of 4-Chlorophenyl 3-phenylpropiolate **(1h).**

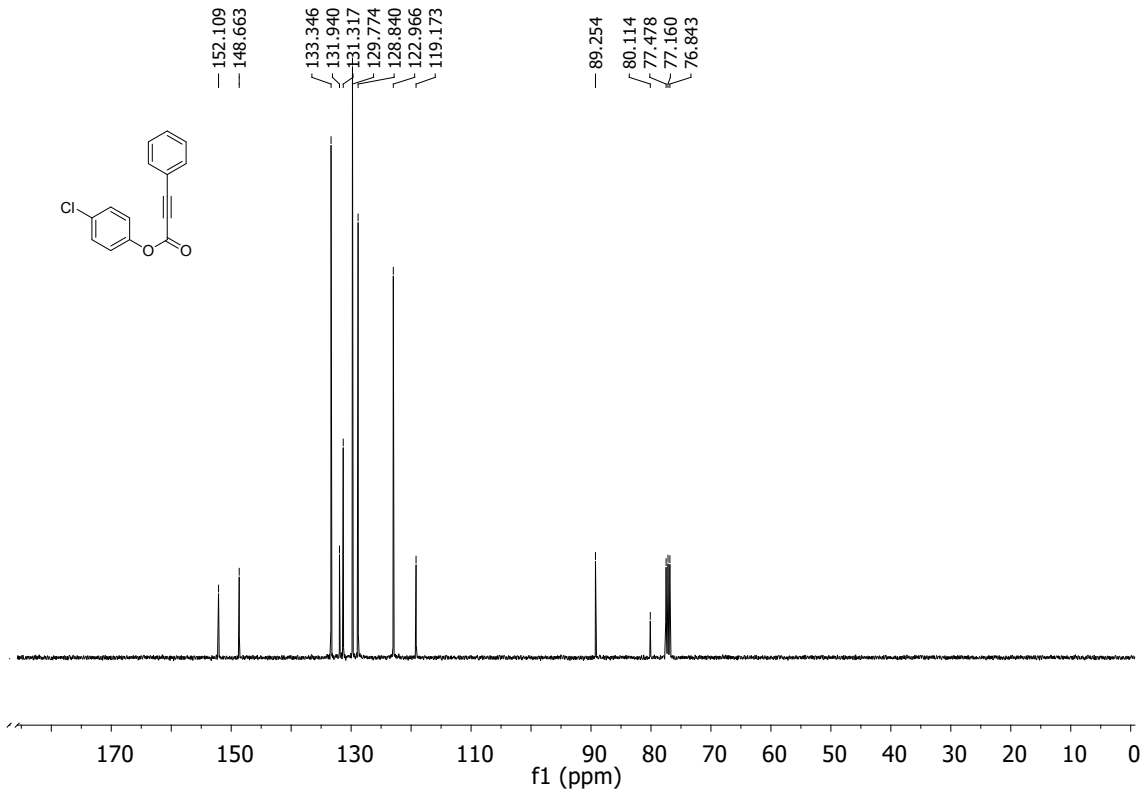

Fig. S65. [13]C NMR of 4-Chlorophenyl 3-phenylpropiolate **(1h).**

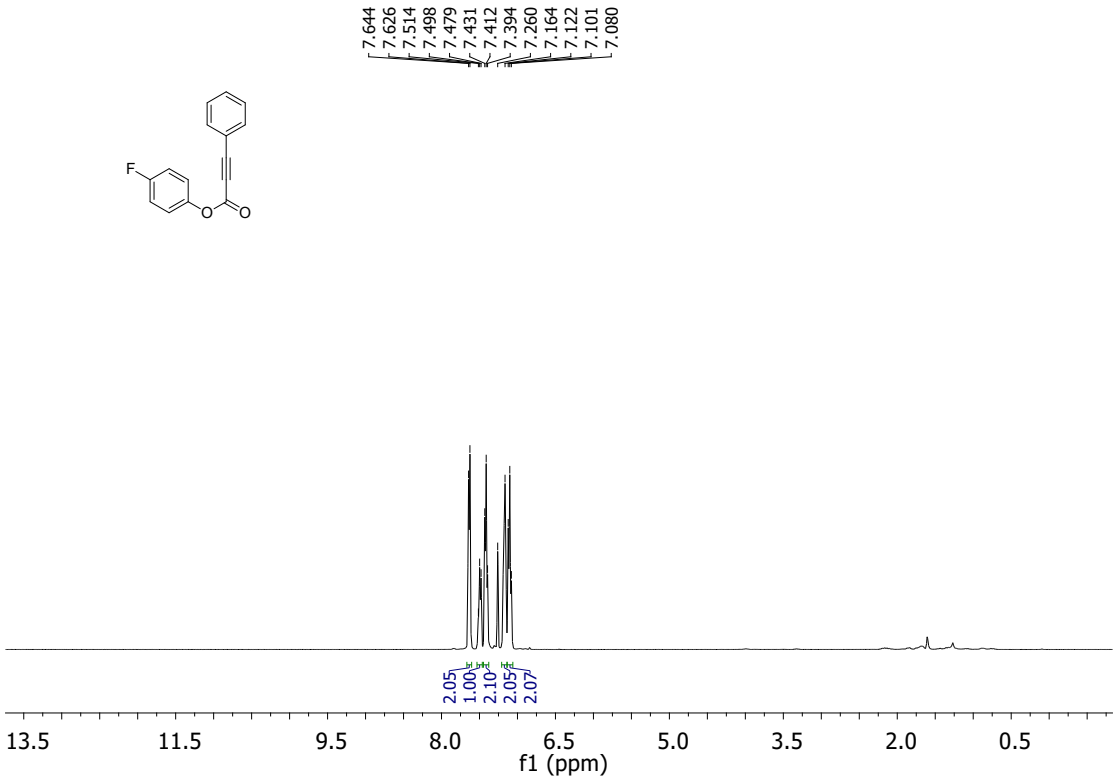

Fig. S66. ^{1}H NMR of 4-Fluorophenyl 3-phenylpropiolate **(1i).**

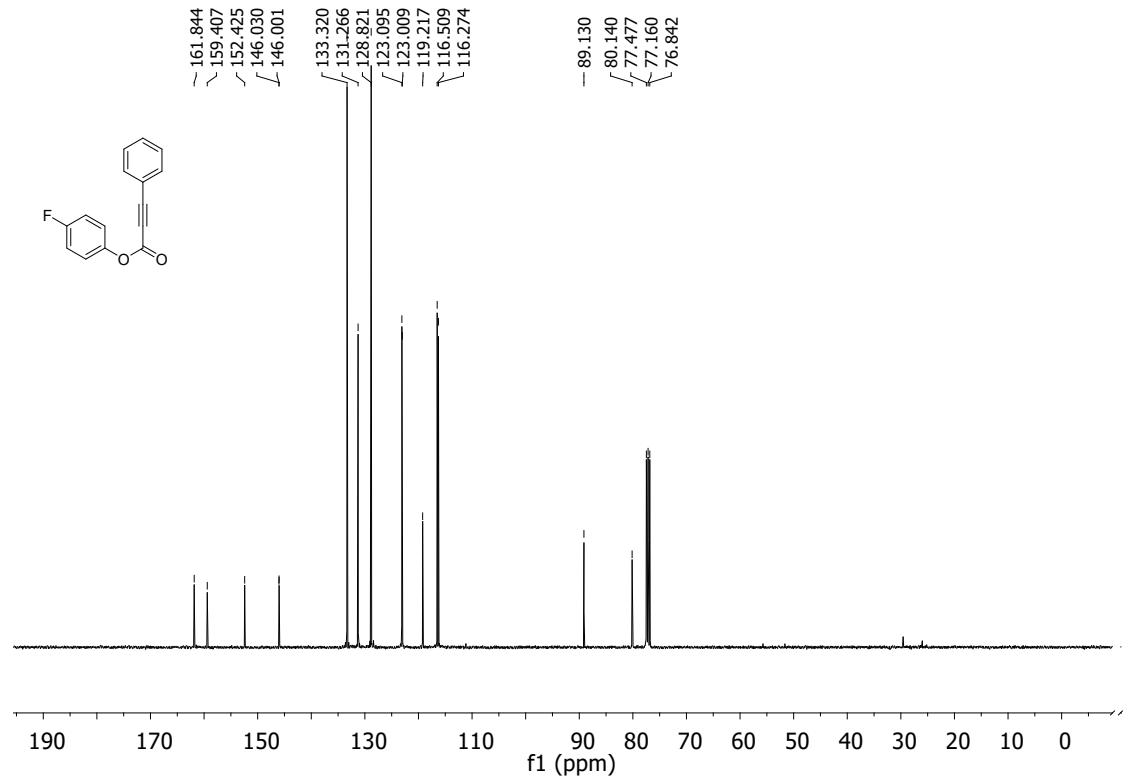

Fig. S67. ^{13}C NMR of 4-Fluorophenyl 3-phenylpropiolate **(1i).**

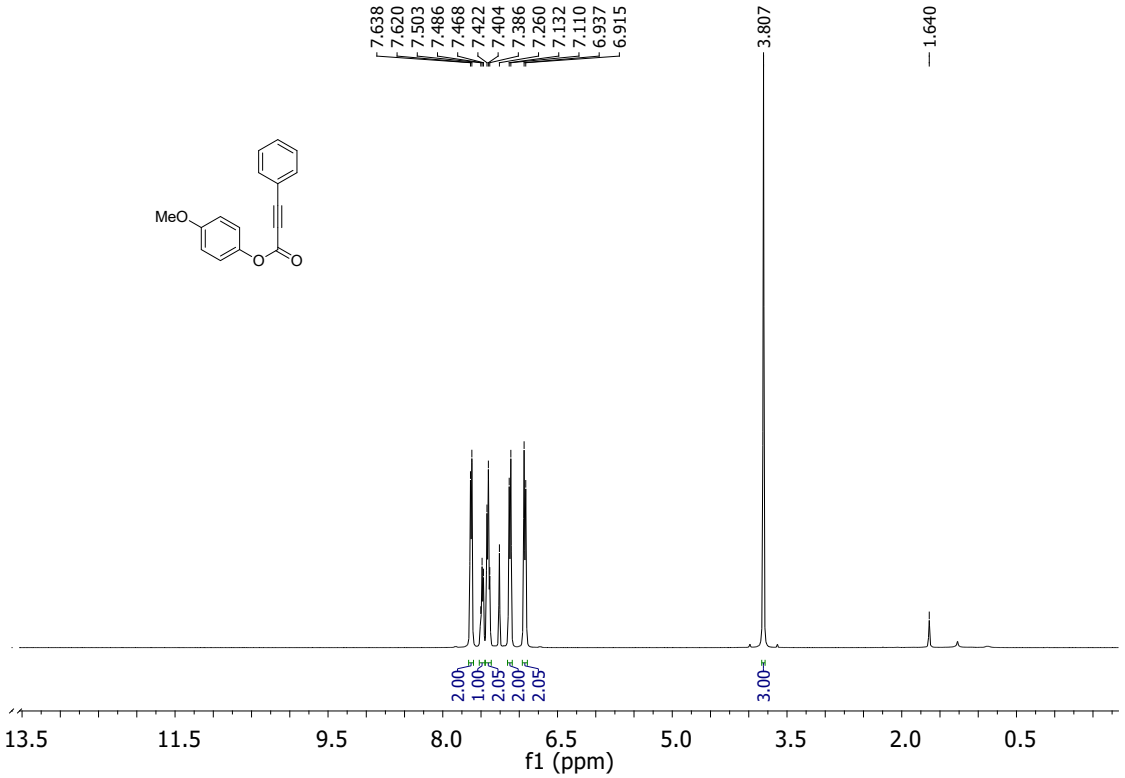

Fig. S68. [1]H NMR of 4-Methoxyphenyl 3-phenylpropiolate **(1j).**

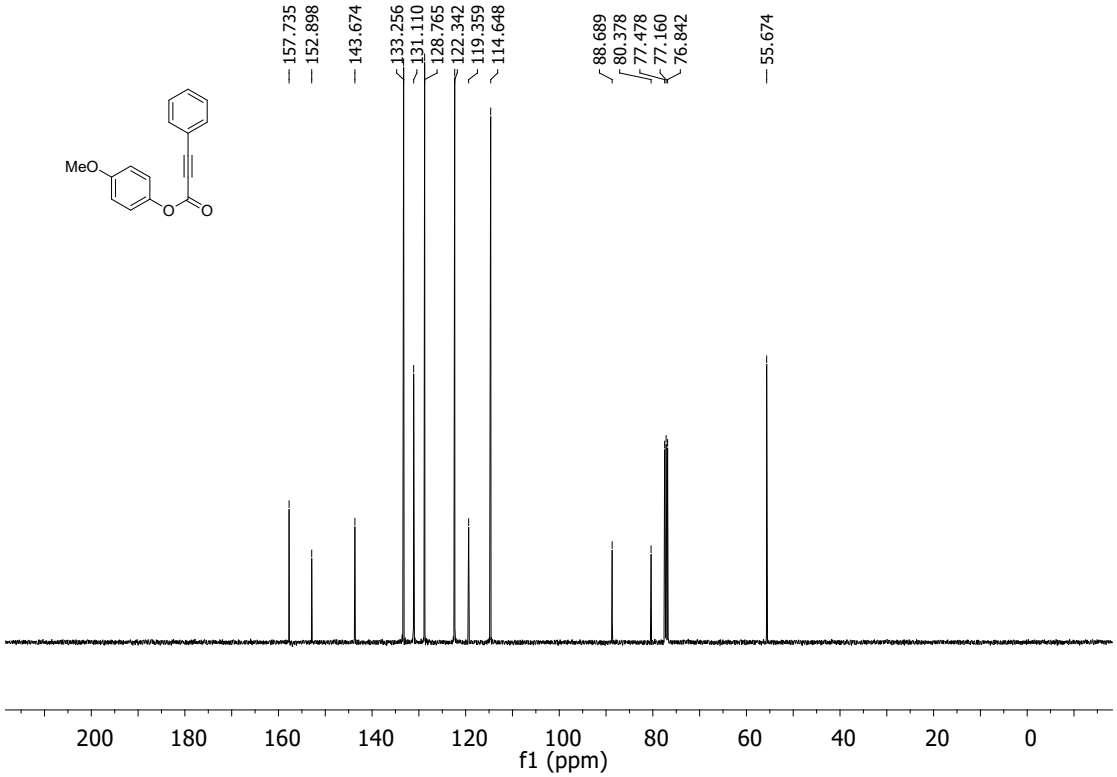

Fig. S69. [13]C NMR of 4-Methoxyphenyl 3-phenylpropiolate **(1j).**

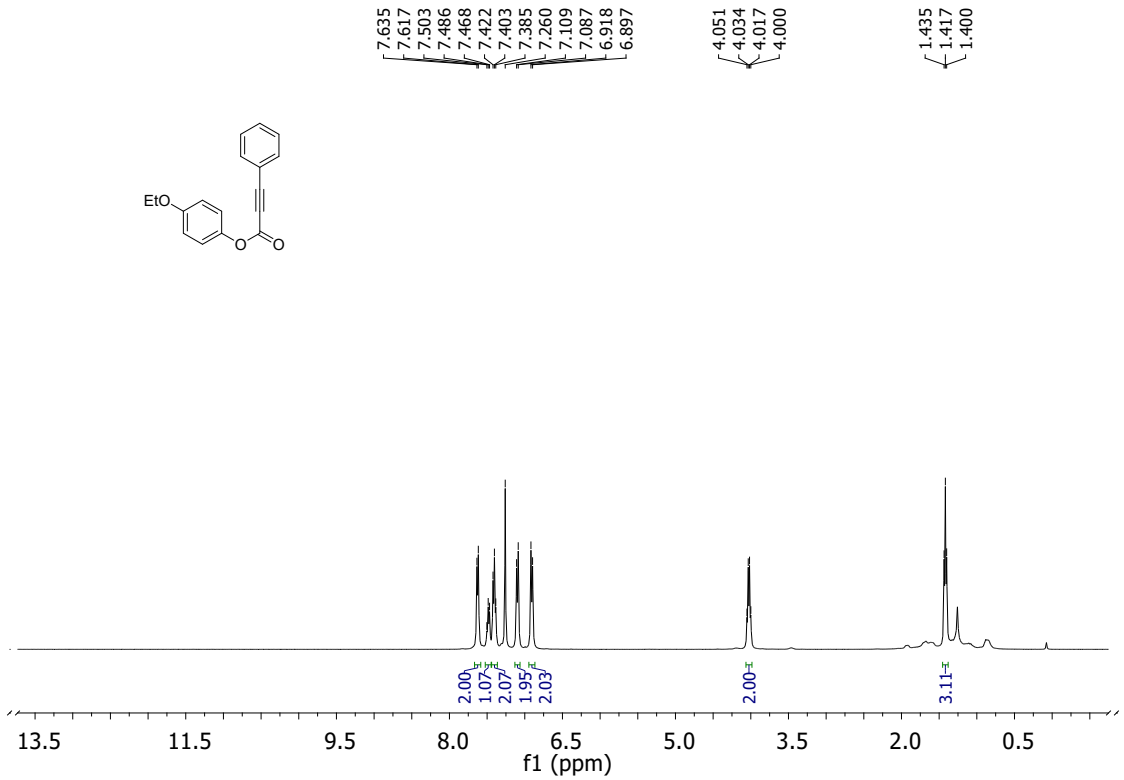

Fig. S70. [1]H NMR of 4-Ethoxyphenyl 3-phenylpropiolate **(1k).**

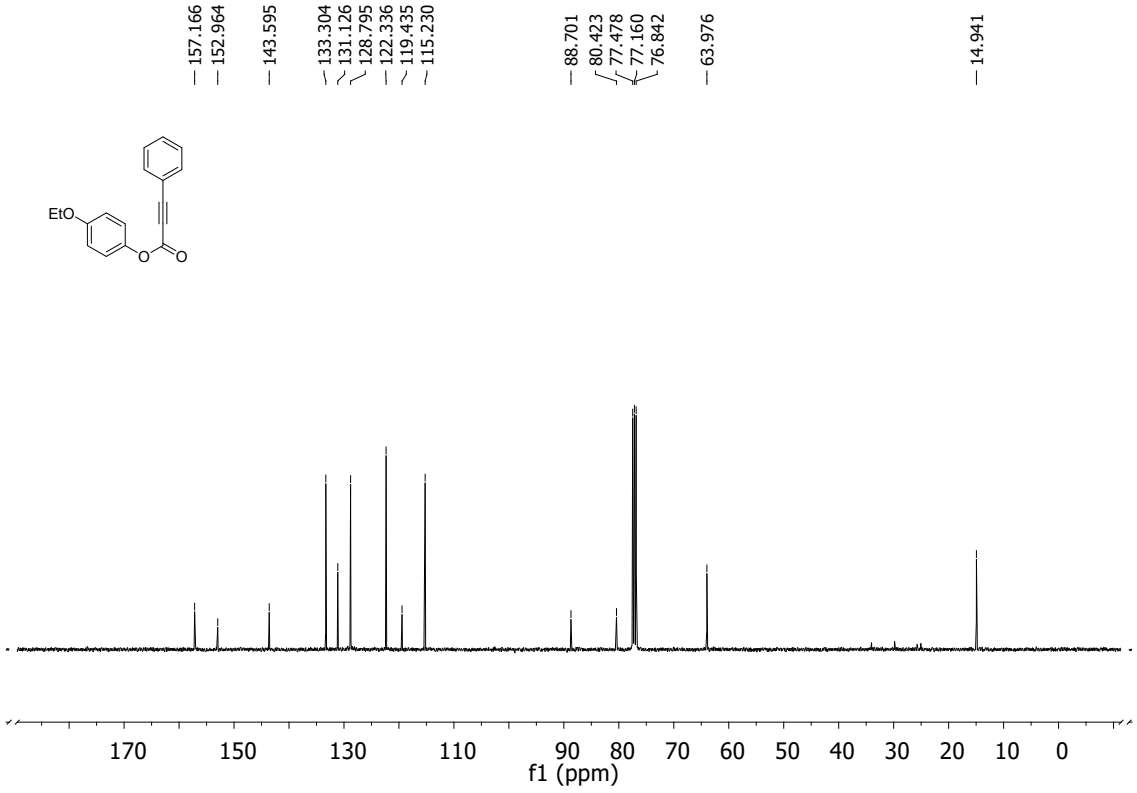

Fig. S71. [13]C NMR of 4-Ethoxyphenyl 3-phenylpropiolate **(1k).**

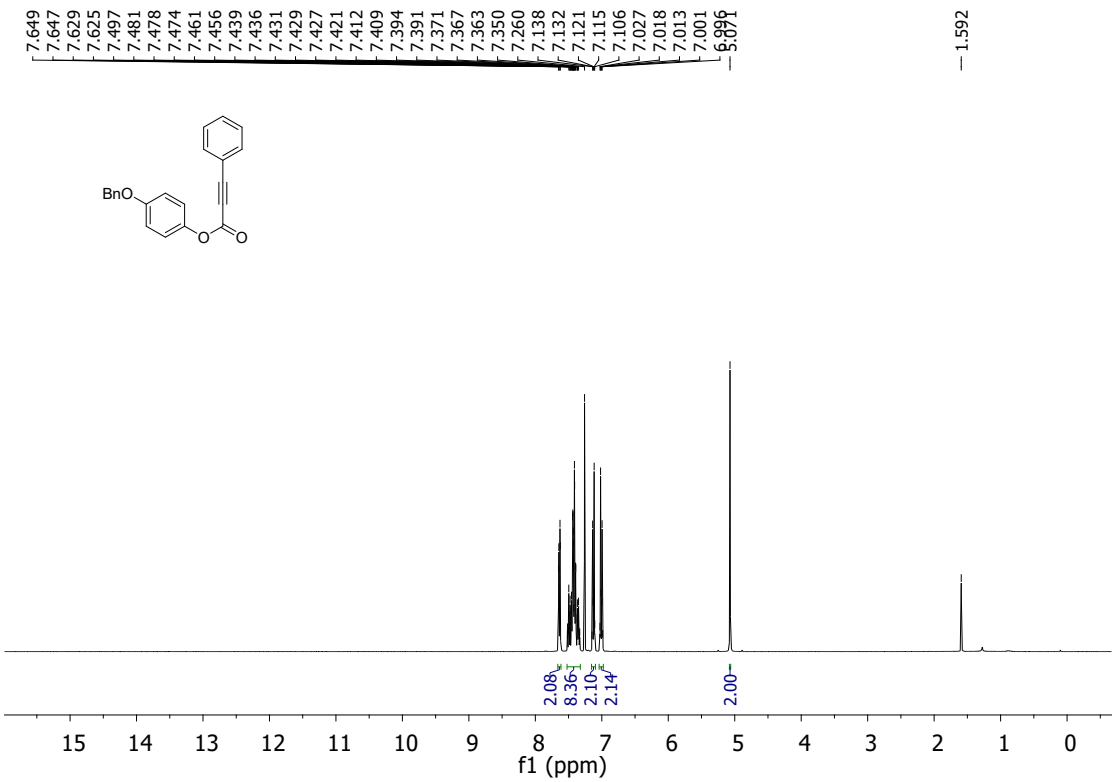

Fig. S72. [1]H NMR of 4-(Benzyloxy)phenyl 3-phenylpropiolate **(1l).**

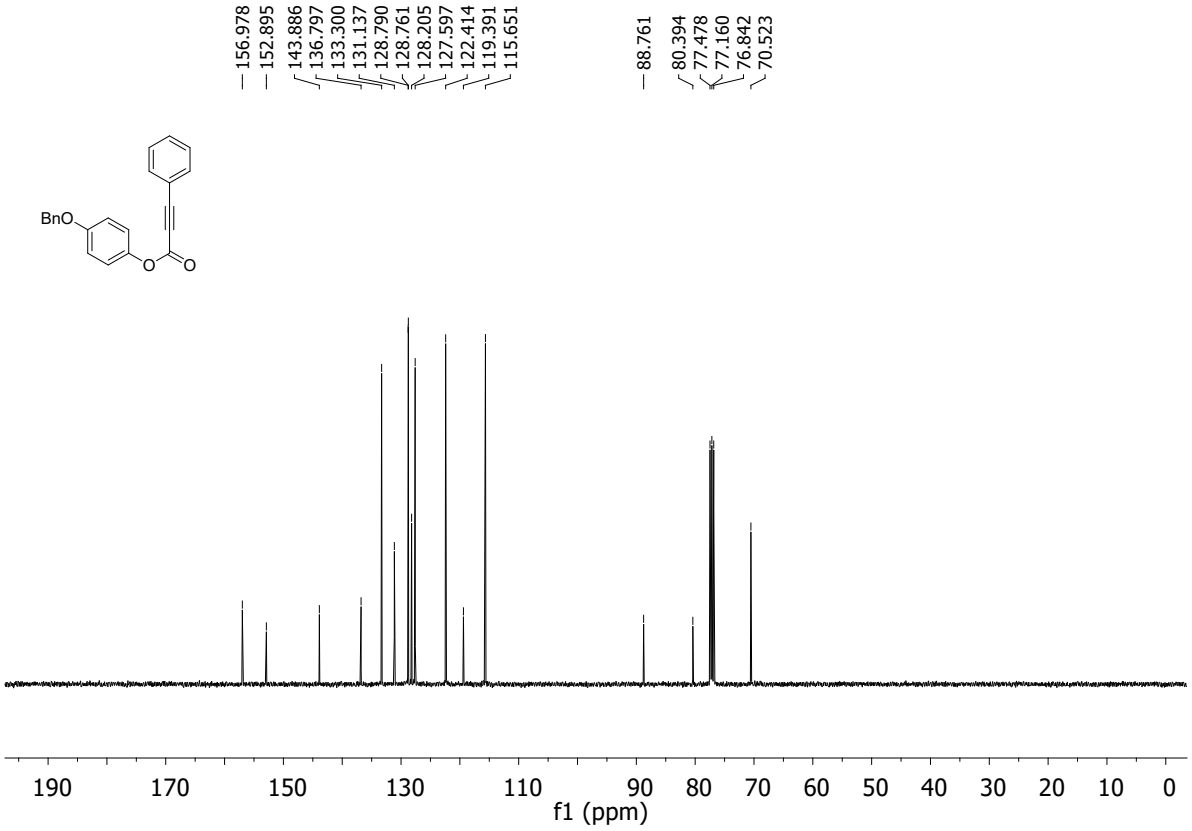

Fig. S73. [13]C NMR of 4-(Benzyloxy)phenyl 3-phenylpropiolate **(1l).**

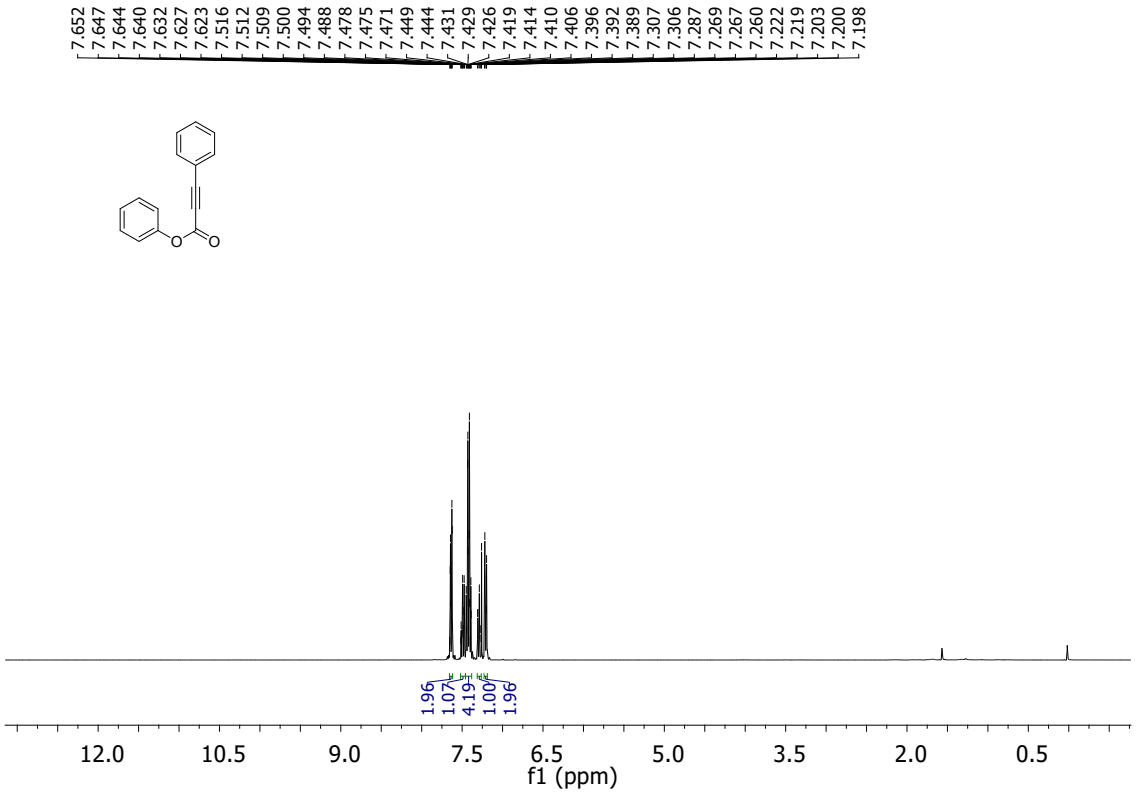

Fig. S74. ^{1}H NMR of Phenyl 3-phenylpropiolate **(1m).**

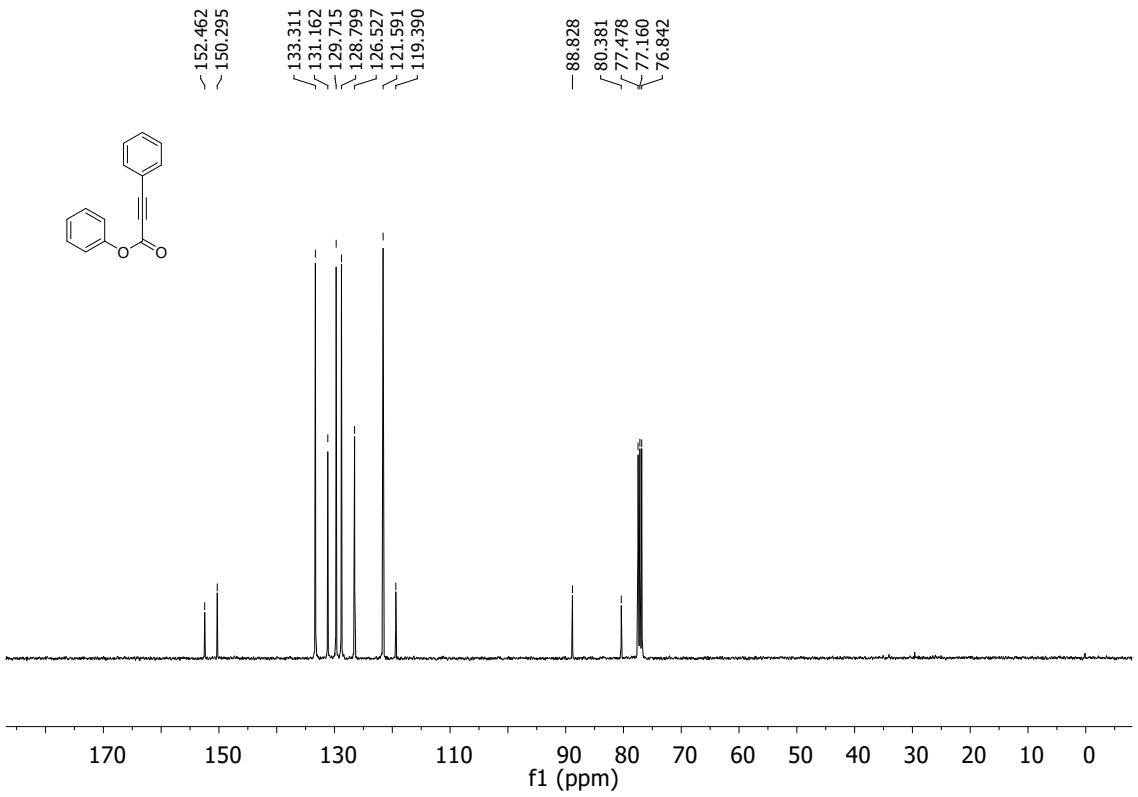

Fig. S75. ^{13}C NMR of Phenyl 3-phenylpropiolate **(1m).**

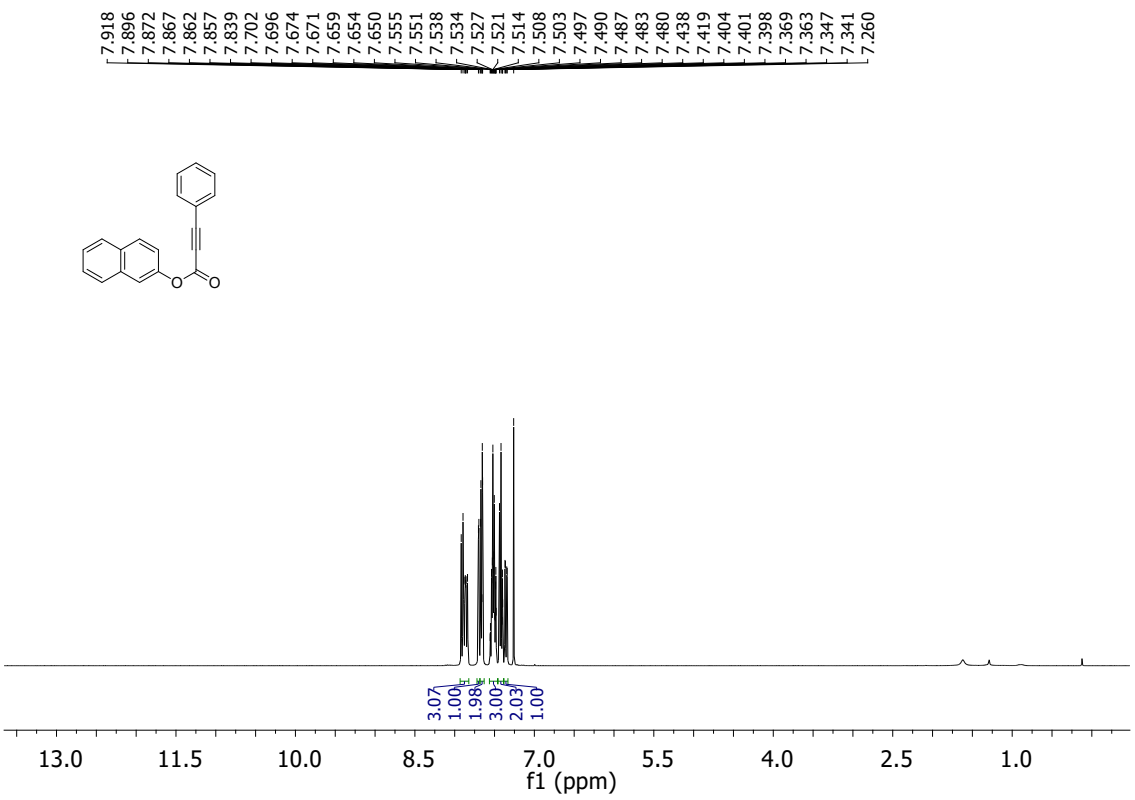

Fig. S76. [1]H NMR of Naphthalen-2-yl 3-phenylpropiolate **(1n).**

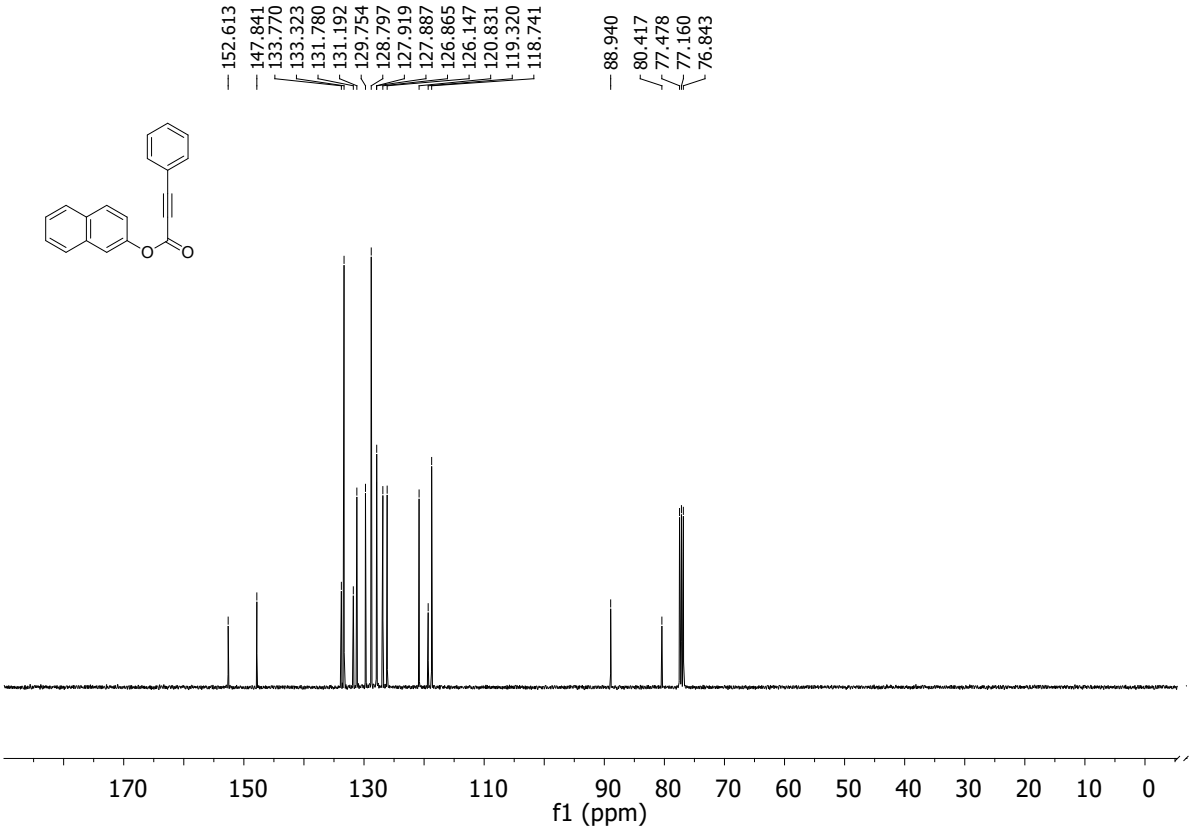

Fig. S77. [13]C NMR of Naphthalen-2-yl 3-phenylpropiolate **(1n).**

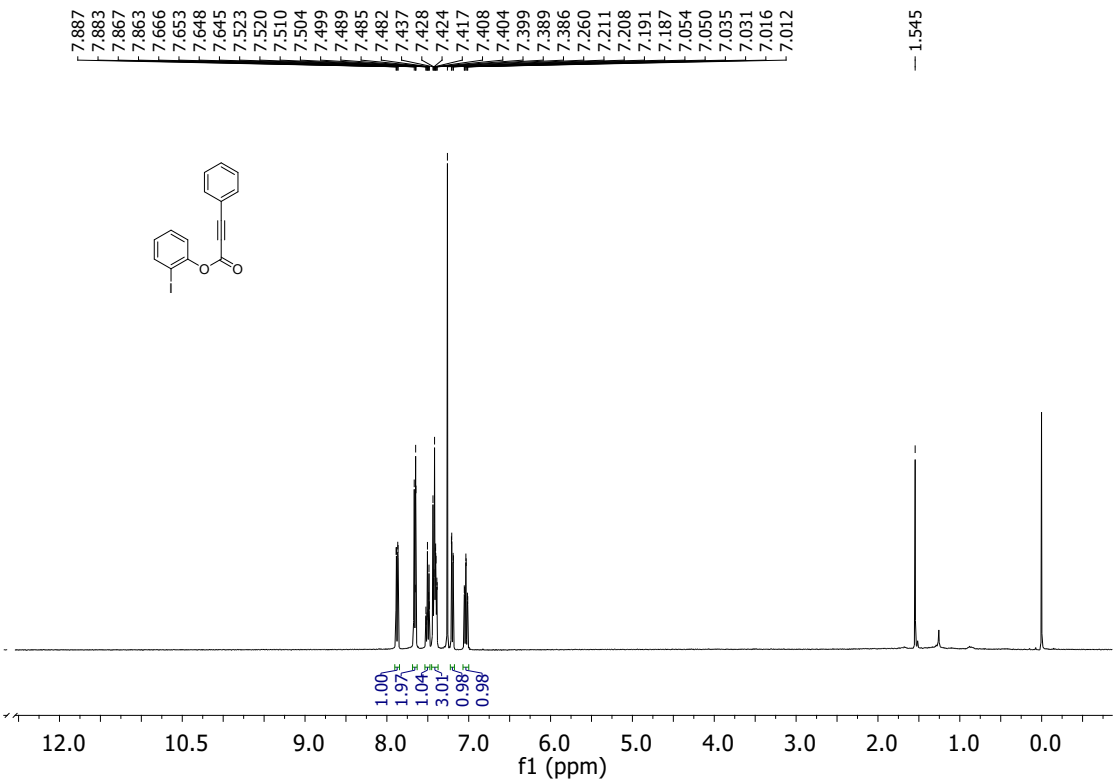

Fig. S78. [1]H NMR of 2-Iodophenyl 3-phenylpropiolate **(1o).**

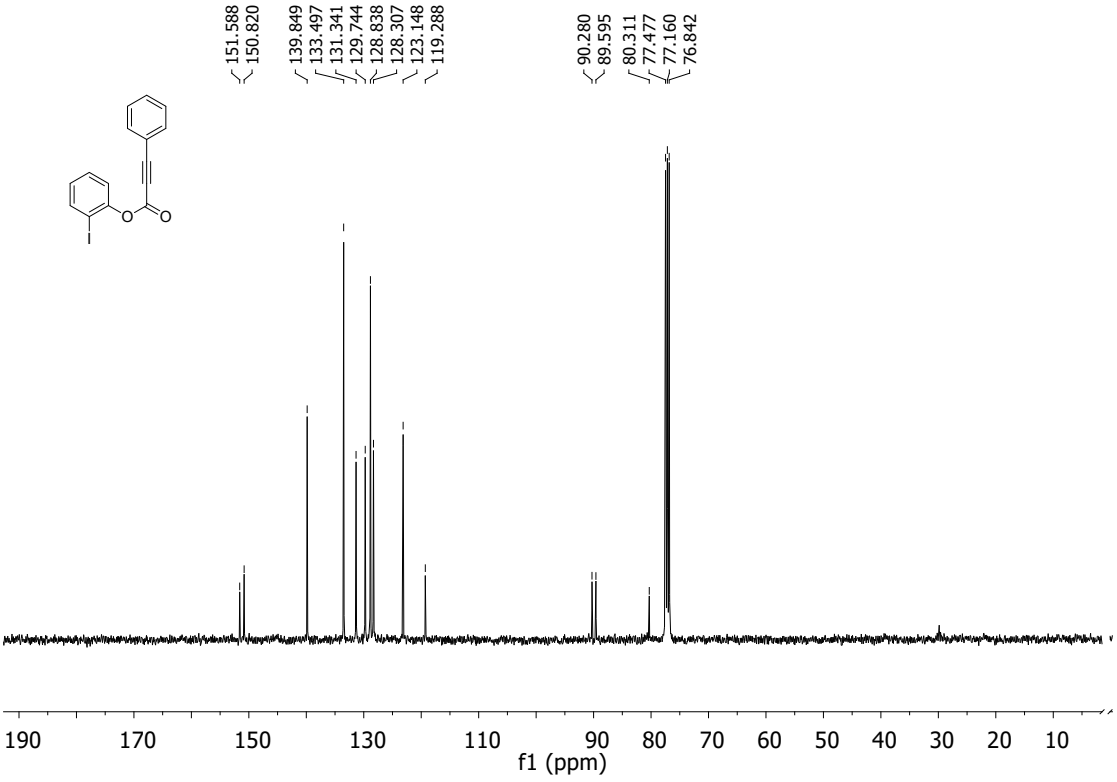

Fig. S79. [13]C NMR of 2-Iodophenyl 3-phenylpropiolate **(1o).**

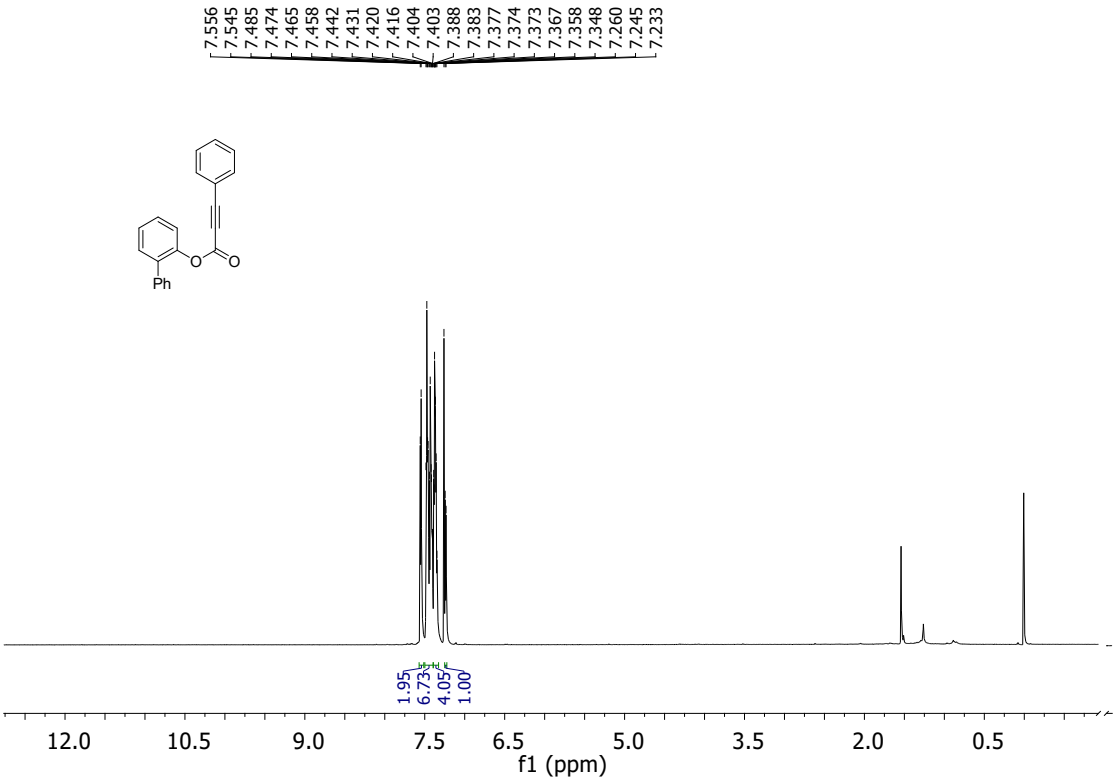

Fig. S80. ^{1}H NMR of [1,1'-Biphenyl]-2-yl 3-phenylpropiolate **(1p).**

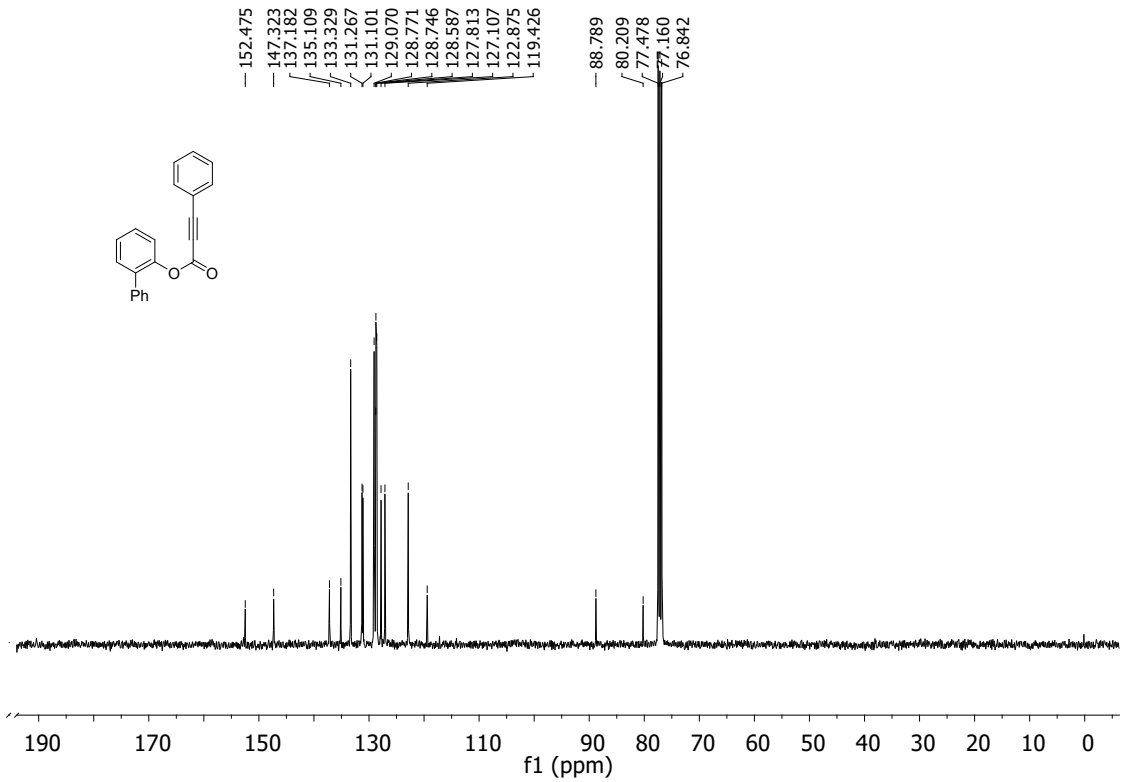

Fig. S81. ^{13}C NMR of [1,1'-Biphenyl]-2-yl 3-phenylpropiolate **(1p).**

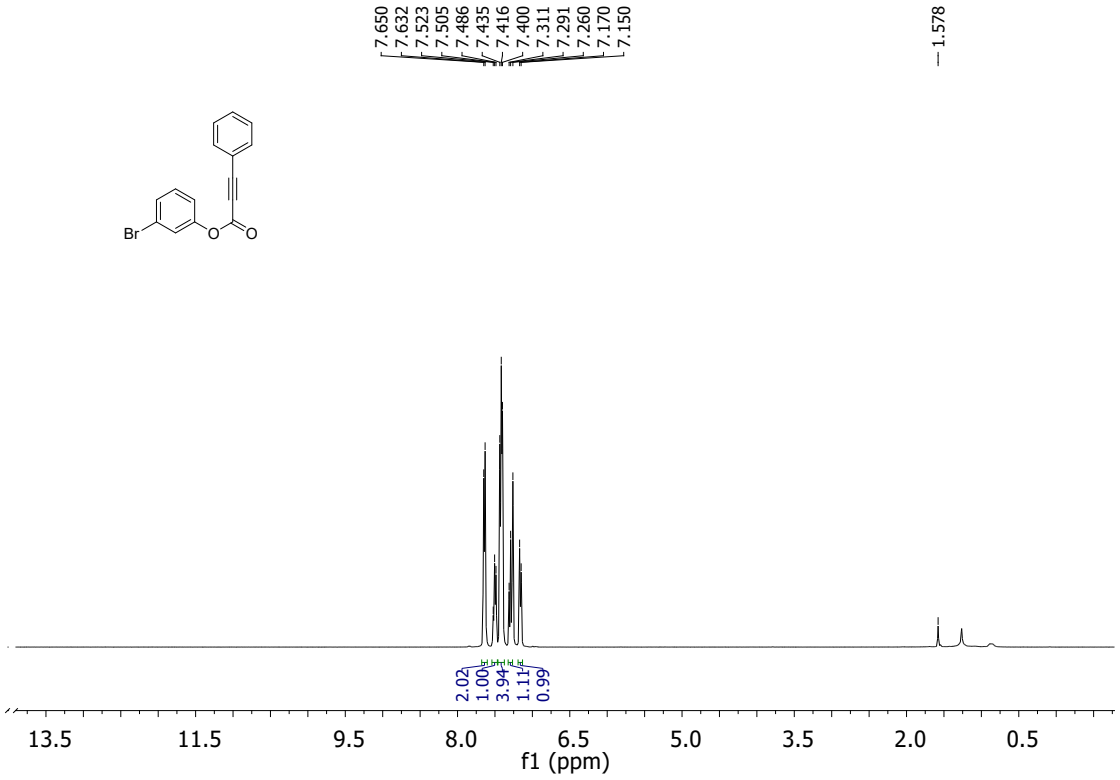

Fig. S82. ^{1}H NMR of 3-Bromophenyl 3-phenylpropiolate **(1q).**

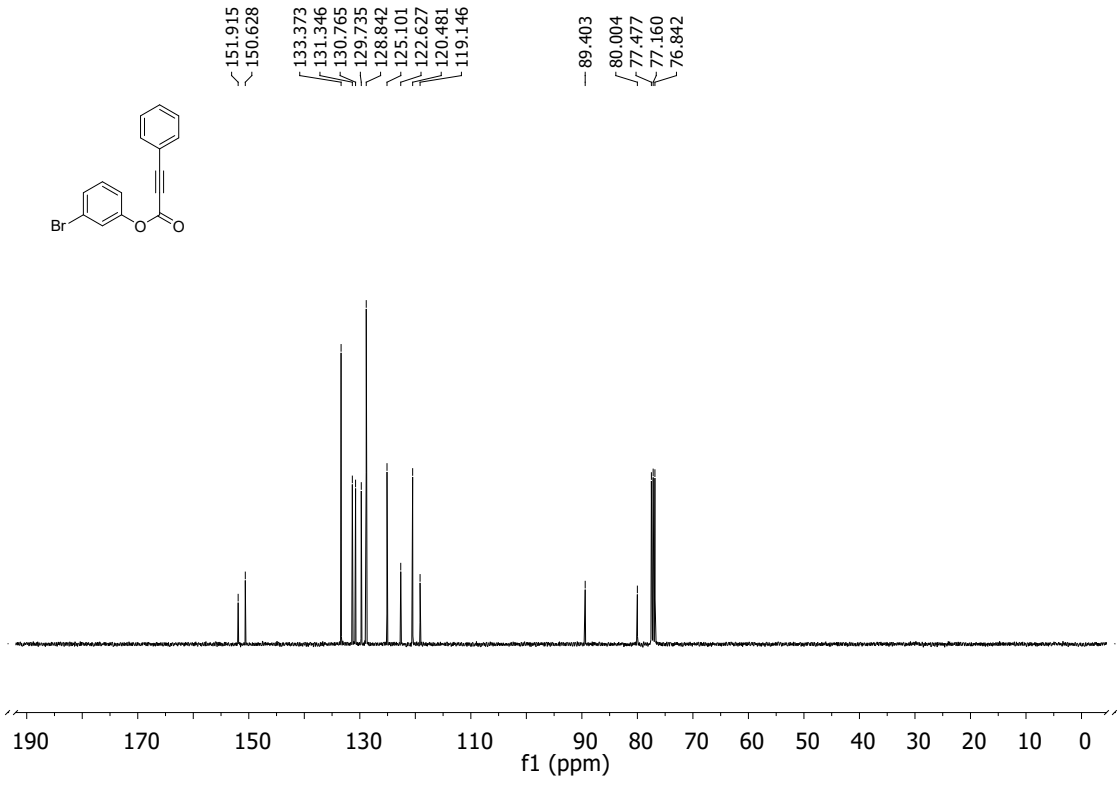

Fig. S83. ^{13}C NMR of 3-Bromophenyl 3-phenylpropiolate **(1q).**

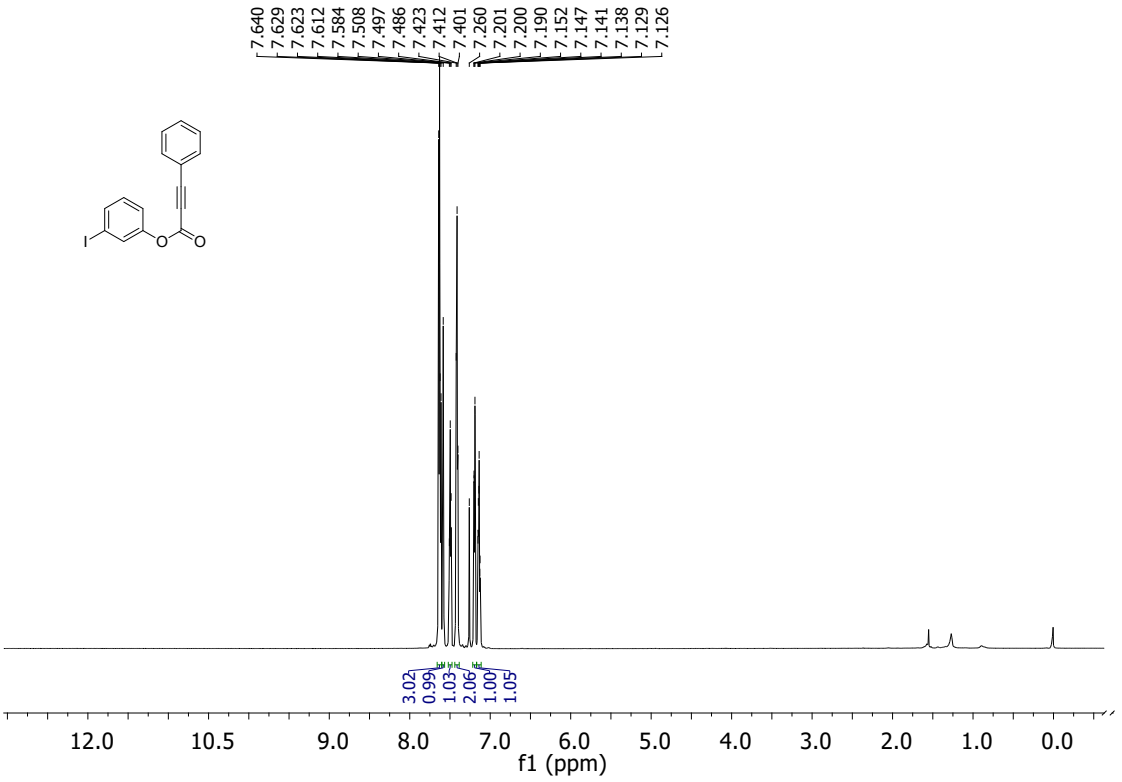

Fig. S84. ^{1}H NMR of 3-Iodophenyl 3-phenylpropiolate **(1r).**

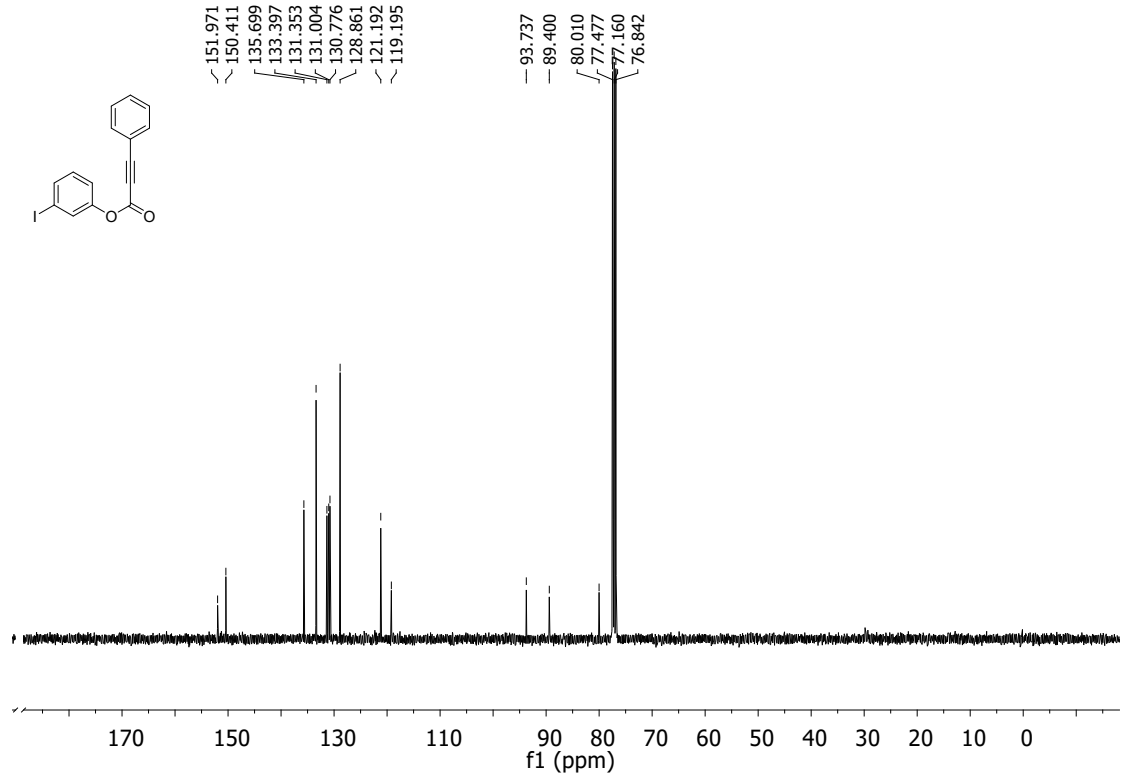

Fig. S85. ^{13}C NMR of 3-Iodophenyl 3-phenylpropiolate **(1r).**

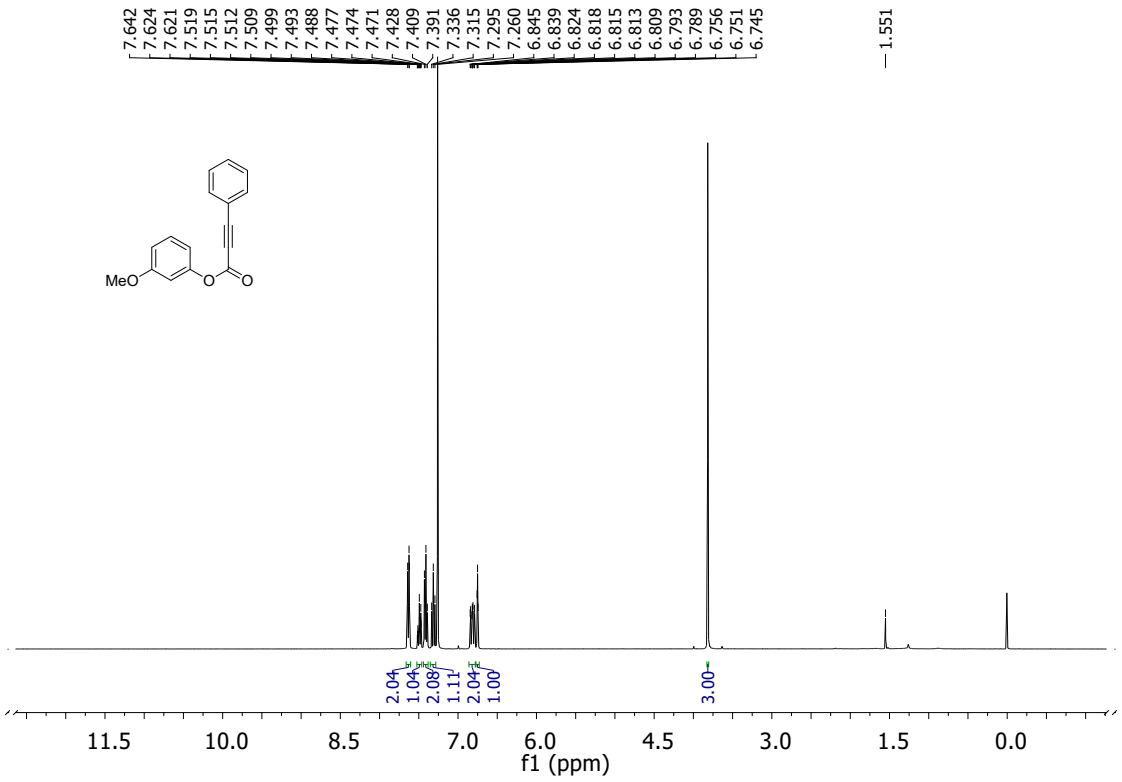

Fig. S86. [1]H NMR of 3-Methoxyphenyl 3-phenylpropiolate **(1s).**

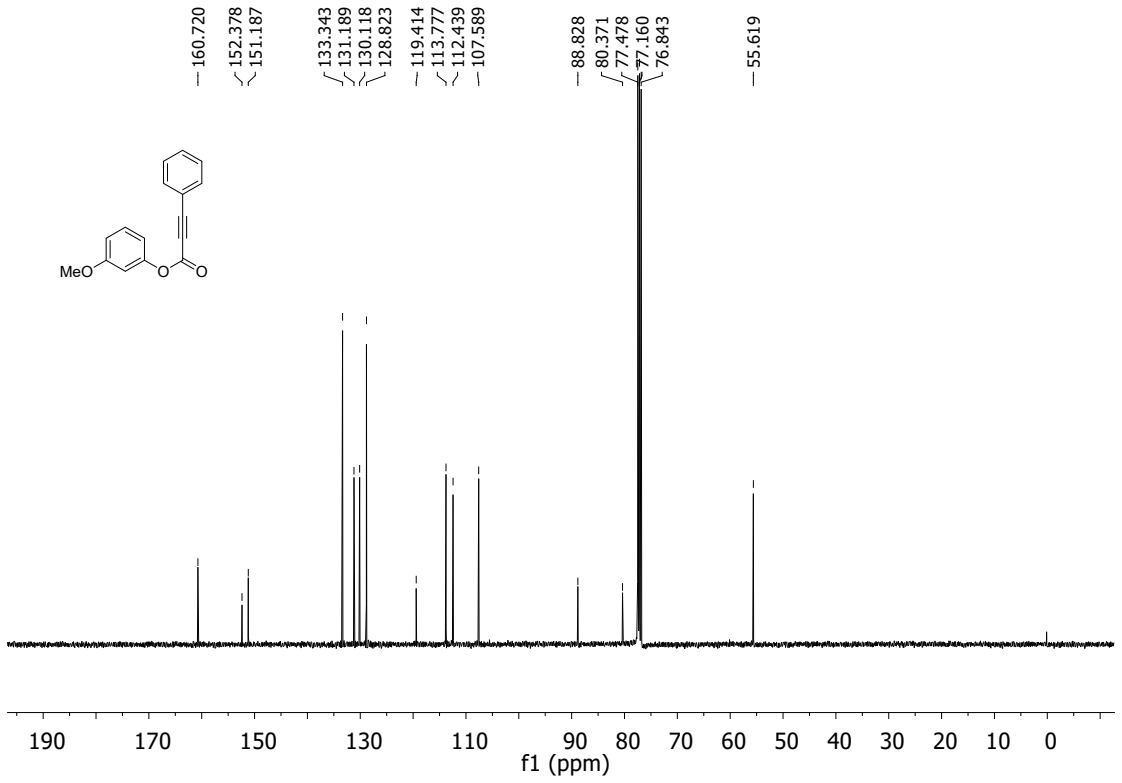

Fig. S87. [13]C NMR of 3-Methoxyphenyl 3-phenylpropiolate **(1s).**

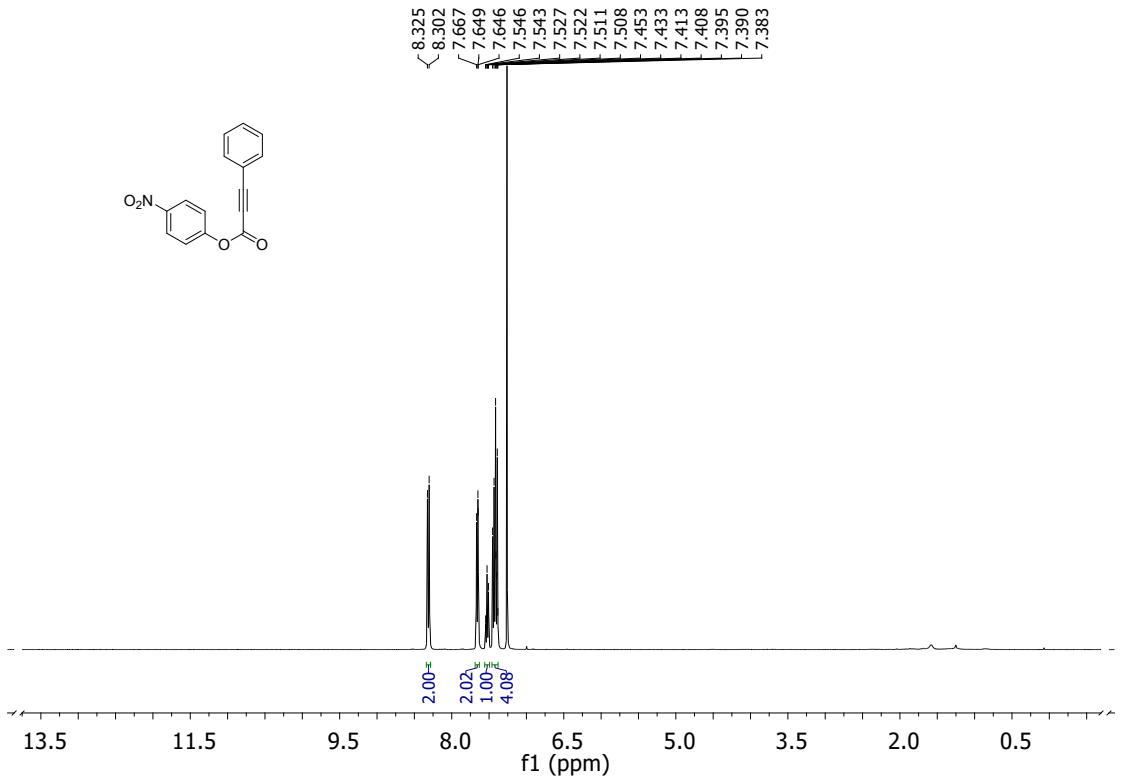

Fig. S88. ^{1}H NMR of 4-Nitrophenyl 3-phenylpropiolate **(1t).**

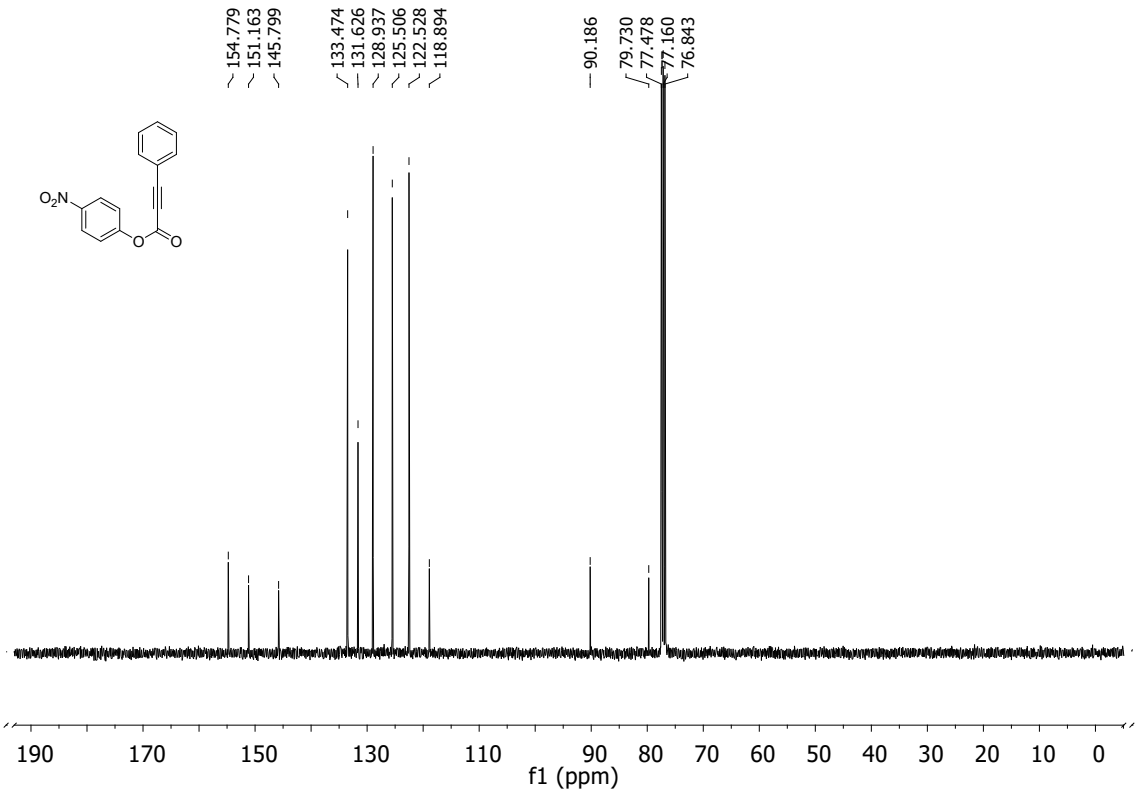

Fig. S89. ^{13}C NMR of 4-Nitrophenyl 3-phenylpropiolate **(1t).**

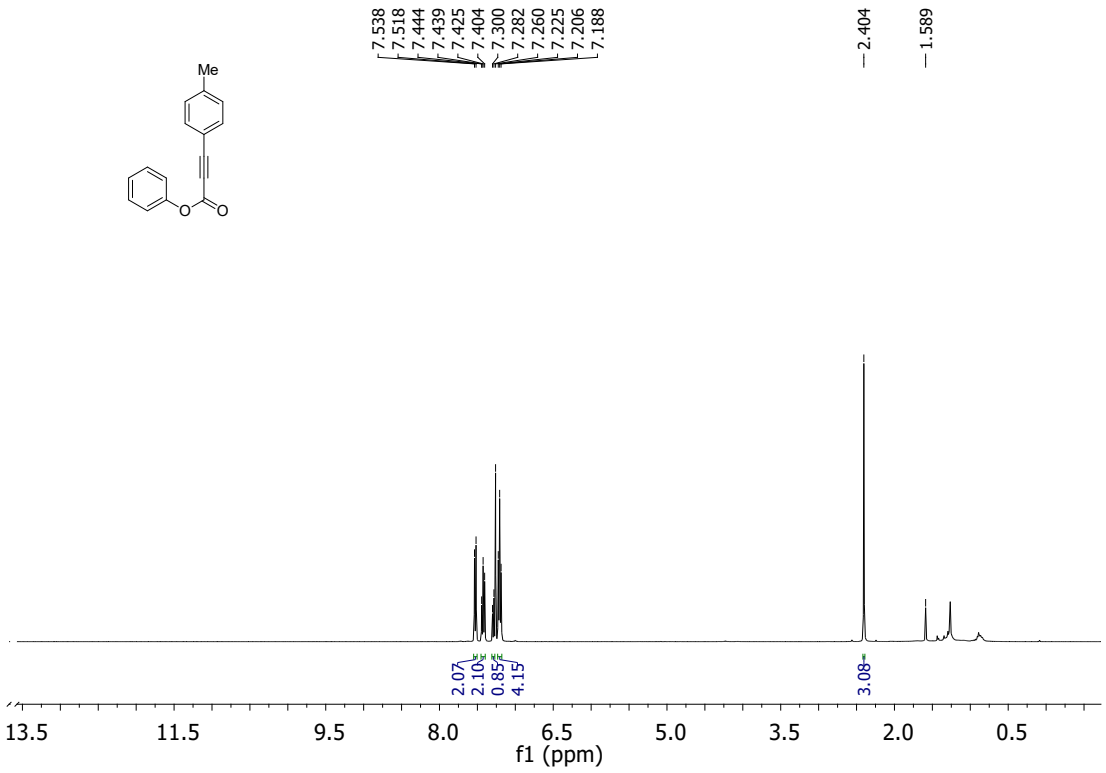

Fig. S90. ^{1}H NMR of Phenyl 3-(*p*-tolyl)propiolate **(1u).**

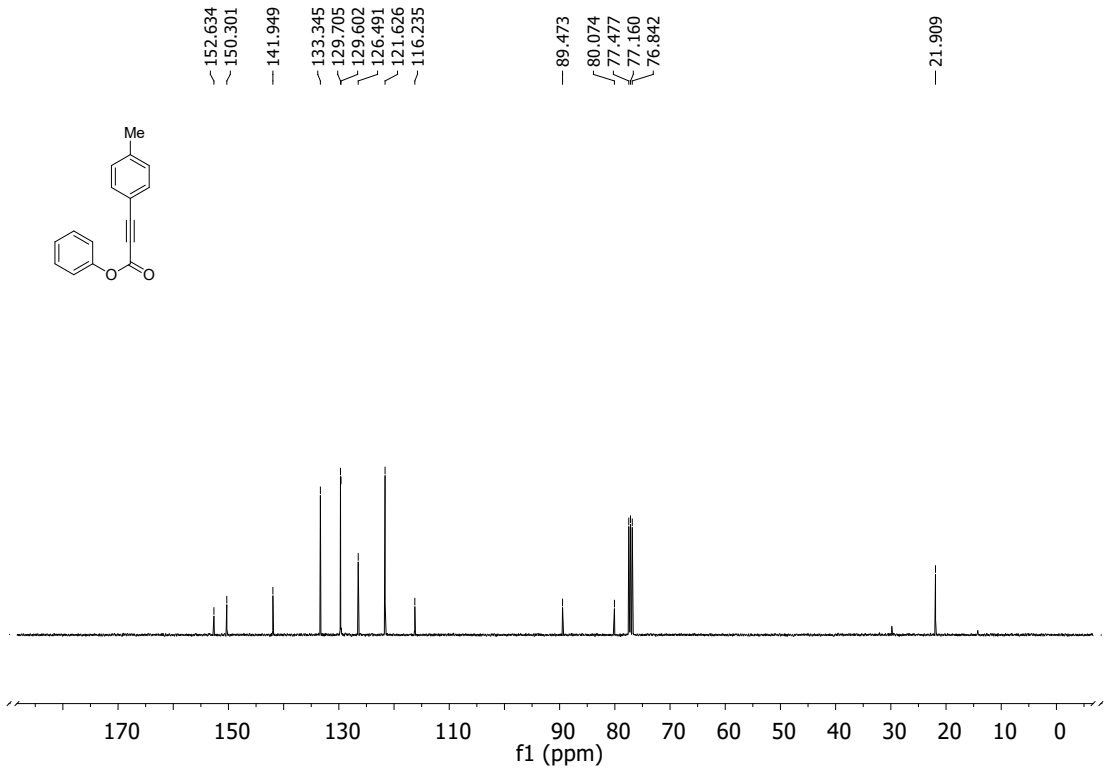

Fig. S91. ^{13}C NMR of Phenyl 3-(*p*-tolyl)propiolate **(1u).**

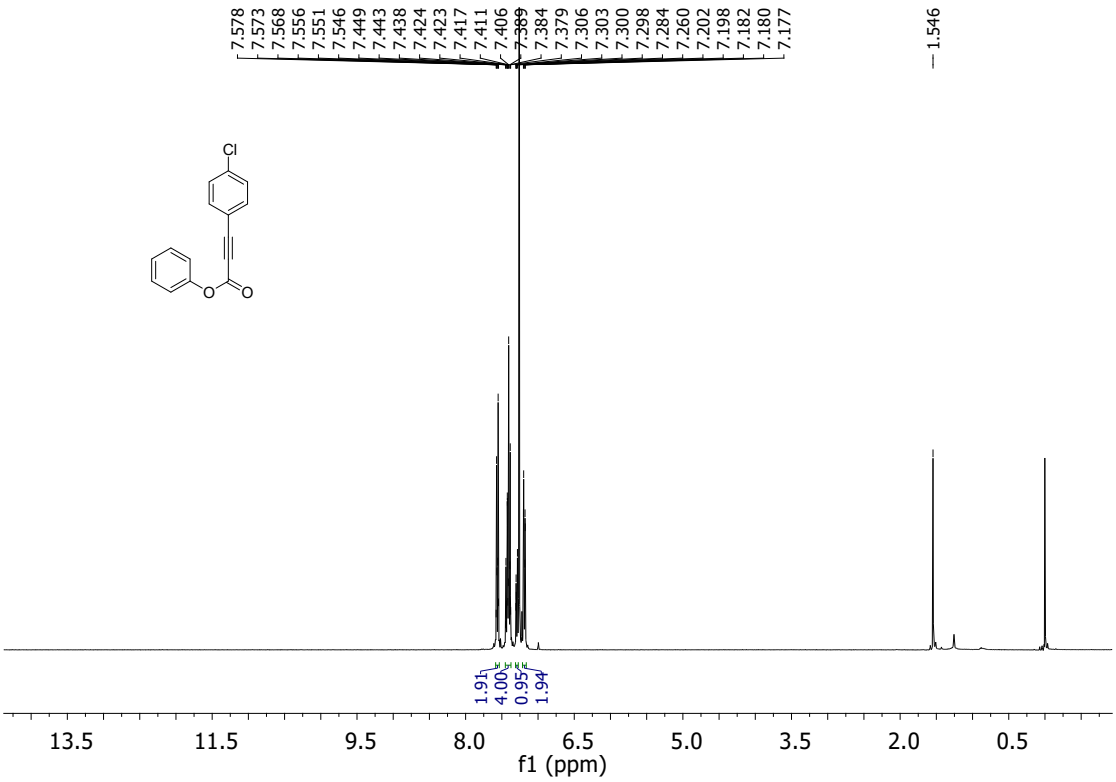

Fig. S92. [1]H NMR of Phenyl 3-(4-chlorophenyl)propiolate **(1v).**

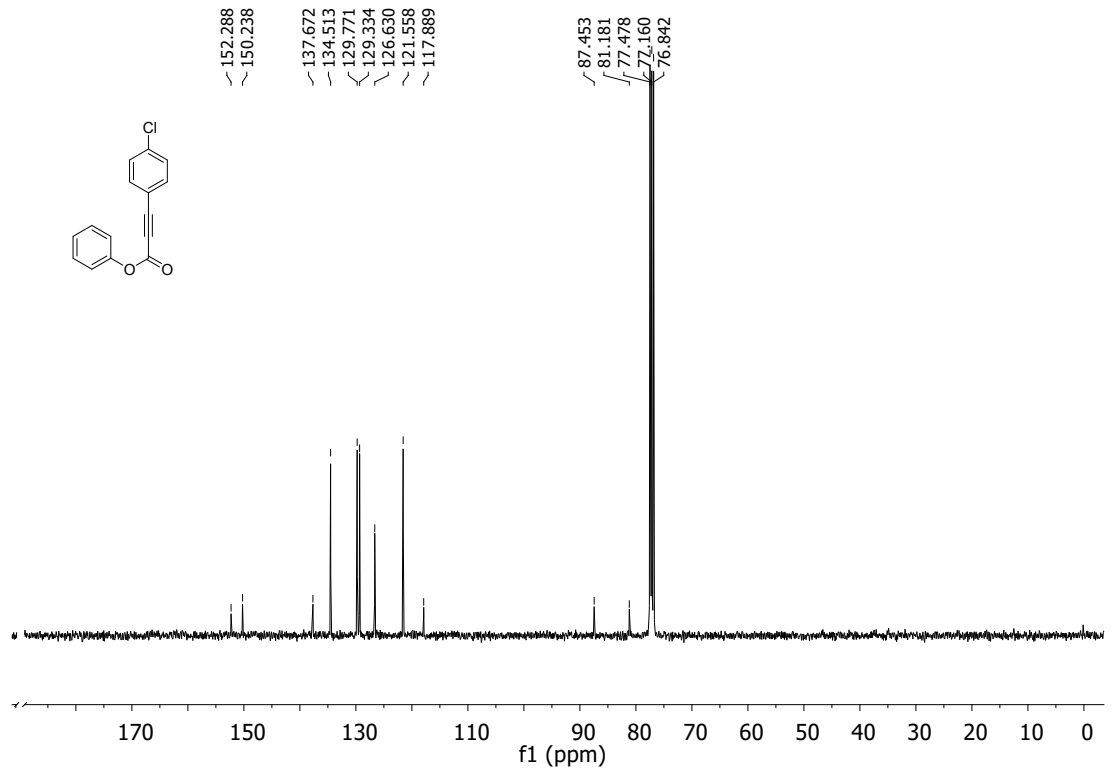

Fig. S93. [13]C NMR of Phenyl 3-(4-chlorophenyl)propiolate **(1v).**

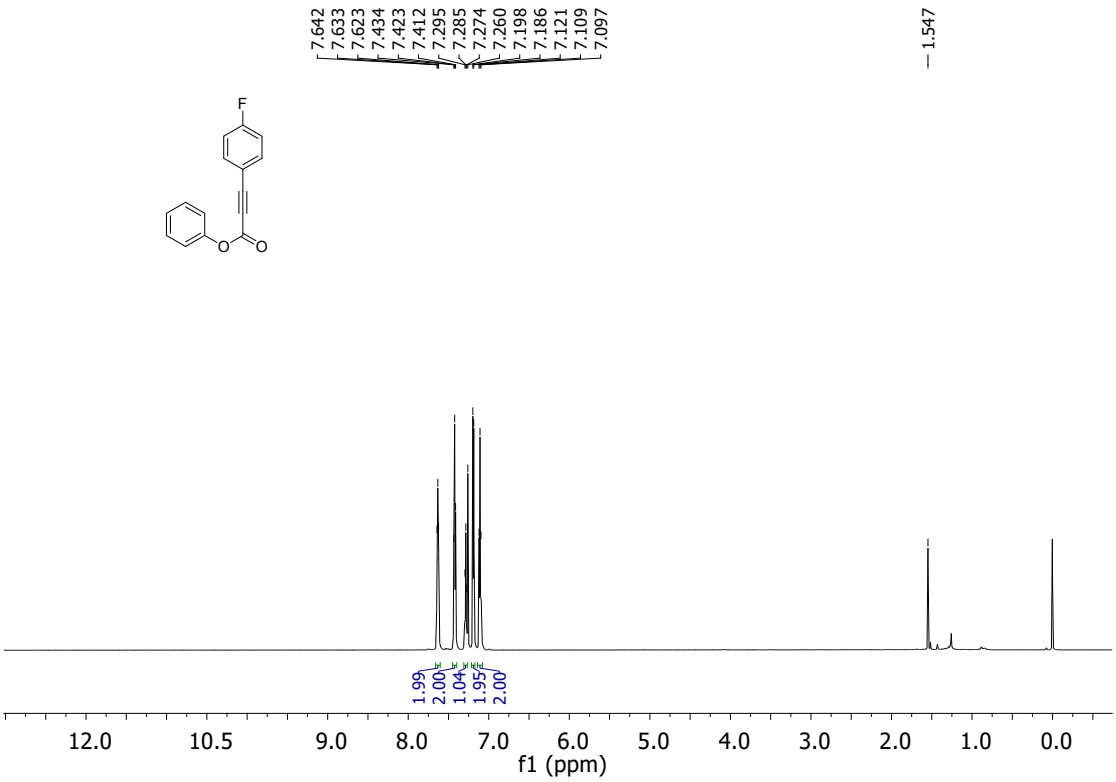

Fig. S94. ^{1}H NMR of Phenyl 3-(4-fluorophenyl)propiolate **(1w).**

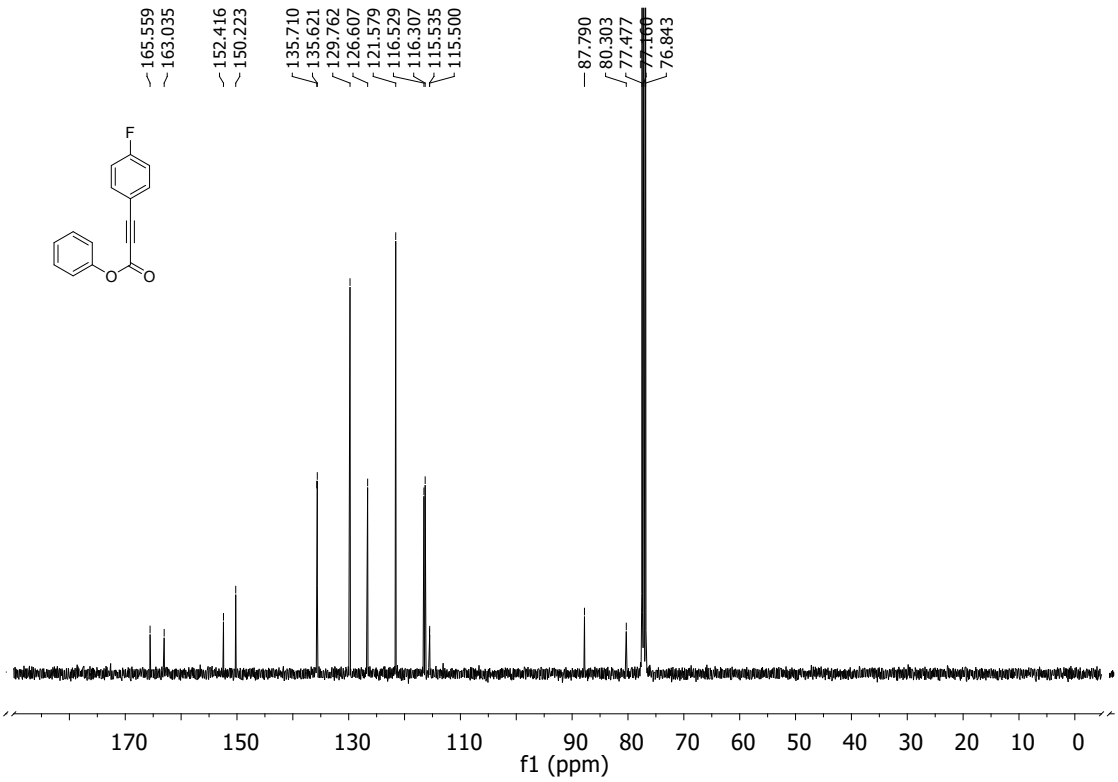

Fig. S95. ^{13}C NMR of Phenyl 3-(4-fluorophenyl)propiolate **(1w).**

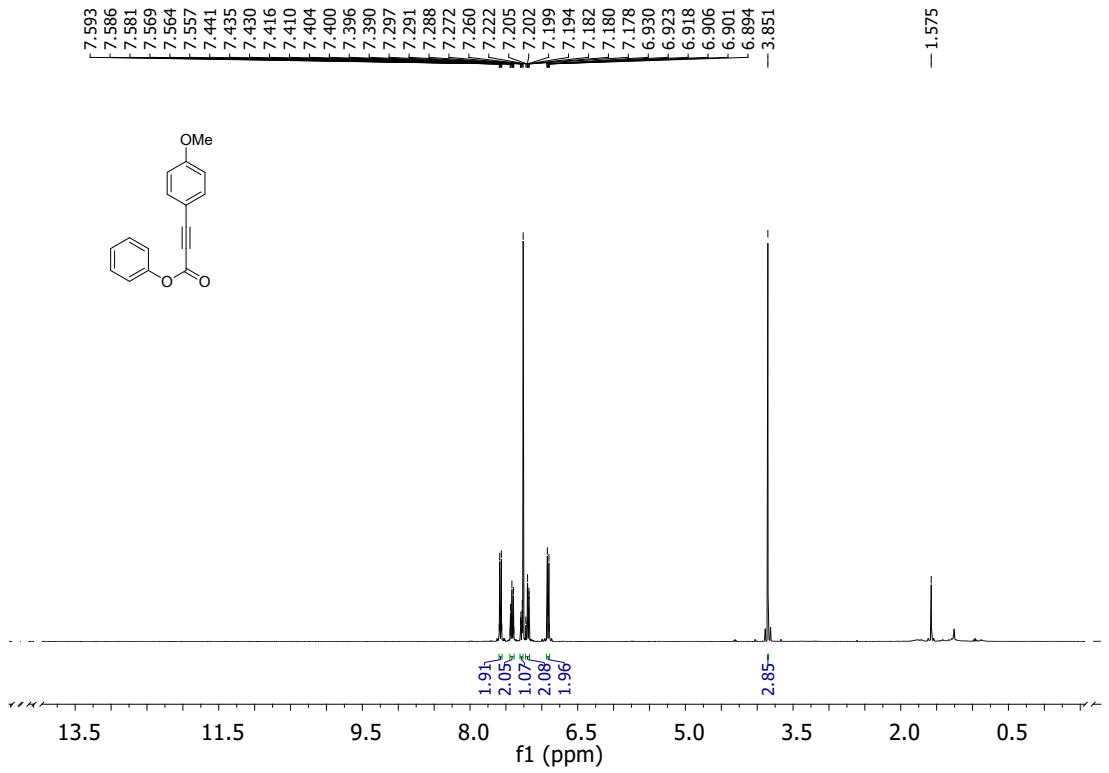

Fig. S96. ^{1}H NMR of Phenyl 3-(4-methoxyphenyl)propiolate **(1x).**

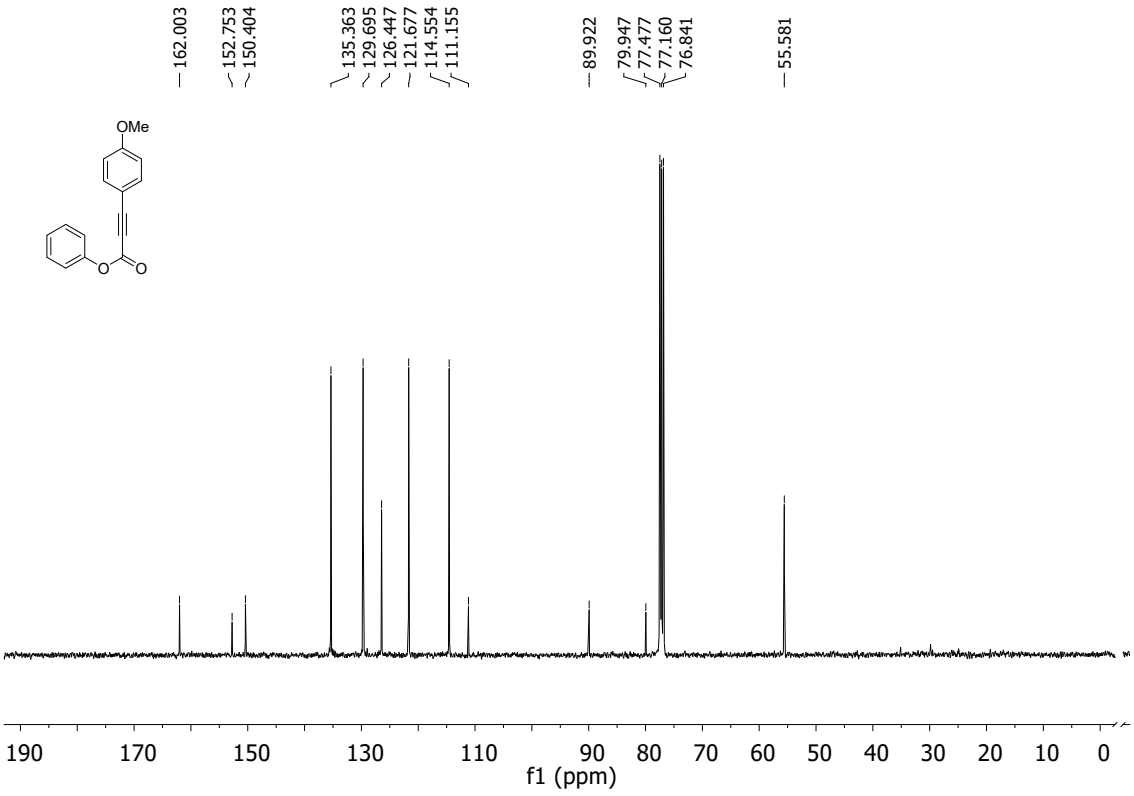

Fig. S97. ^{13}C NMR of Phenyl 3-(4-methoxyphenyl)propiolate **(1x).**

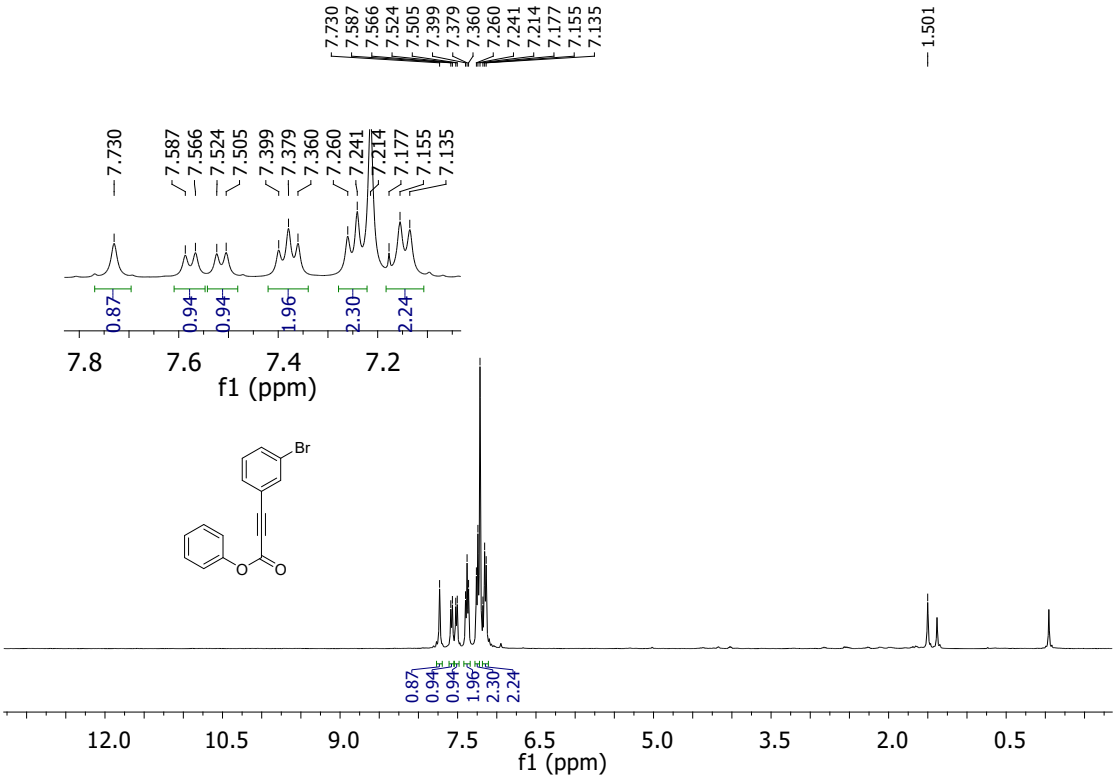

Fig. S98. [1]H NMR of Phenyl 3-(3-bromophenyl)propiolate **(1y).**

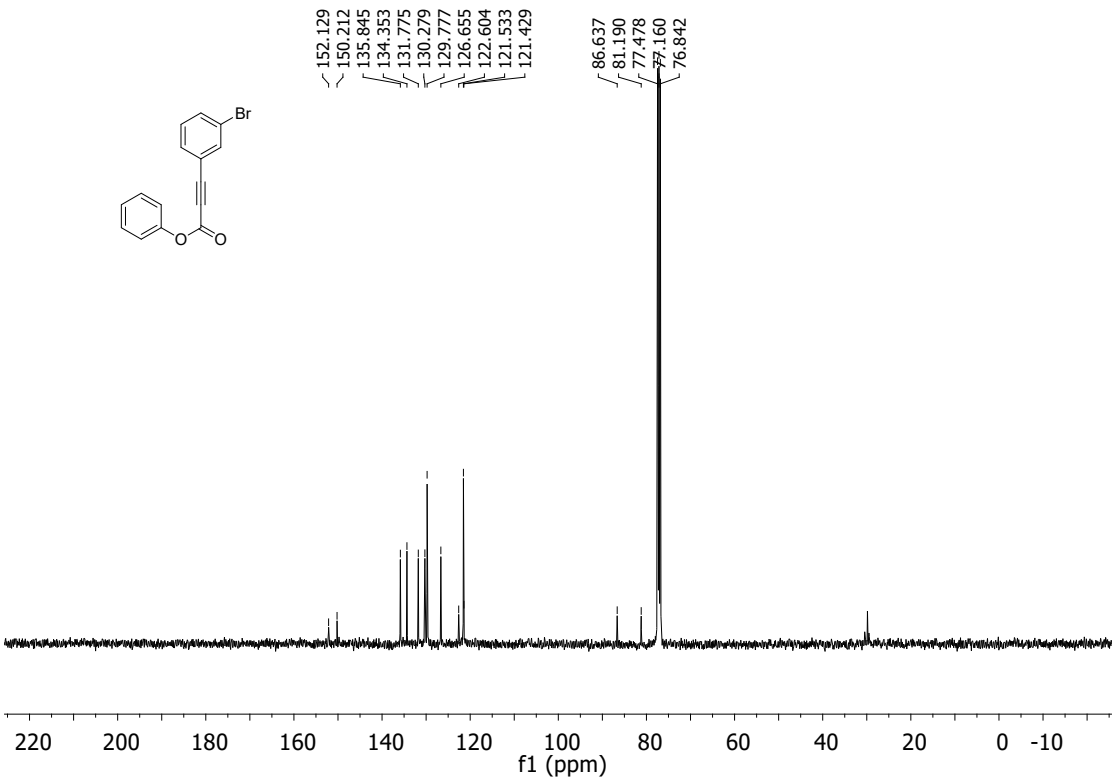

Fig. S99. [13]C NMR of Phenyl 3-(3-bromophenyl)propiolate **(1y).**