

Pd-Catalyzed Sequential Hydroarylation and Olefination Reactions of 3-Allylchromones

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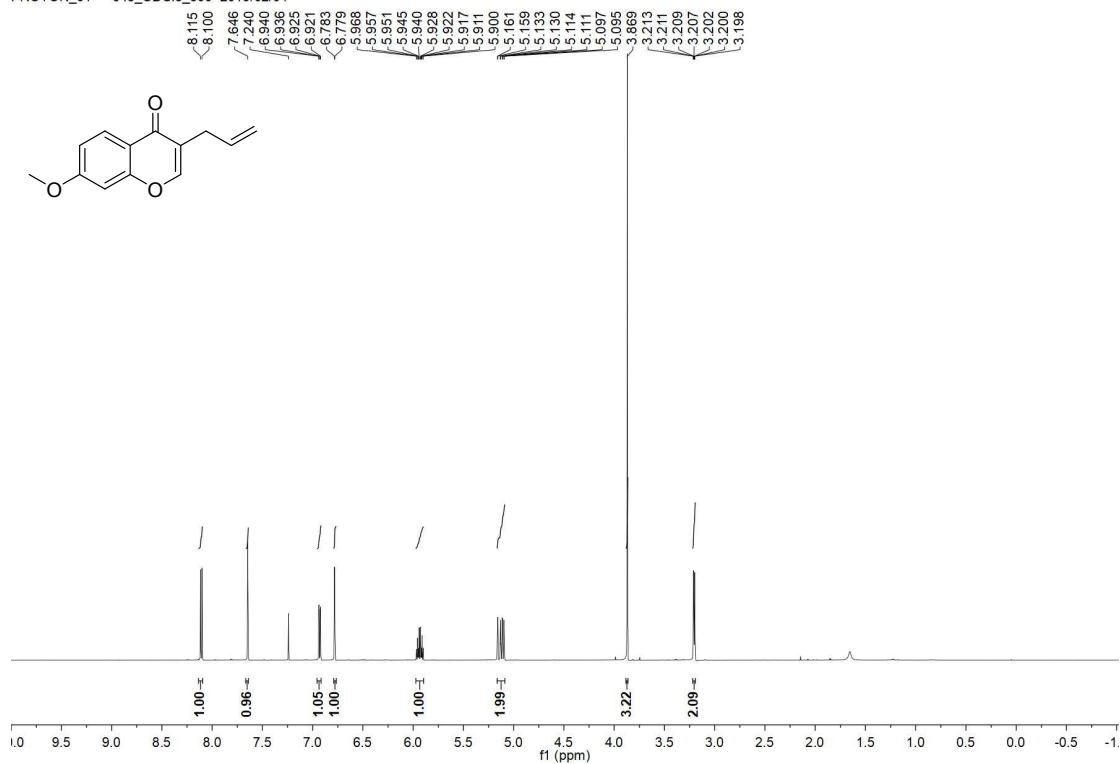
Supporting Information

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3. X-ray data of 2c	S64

3-Allyl-7-methoxy-4H-chromen-4-one (1a)

¹H NMR

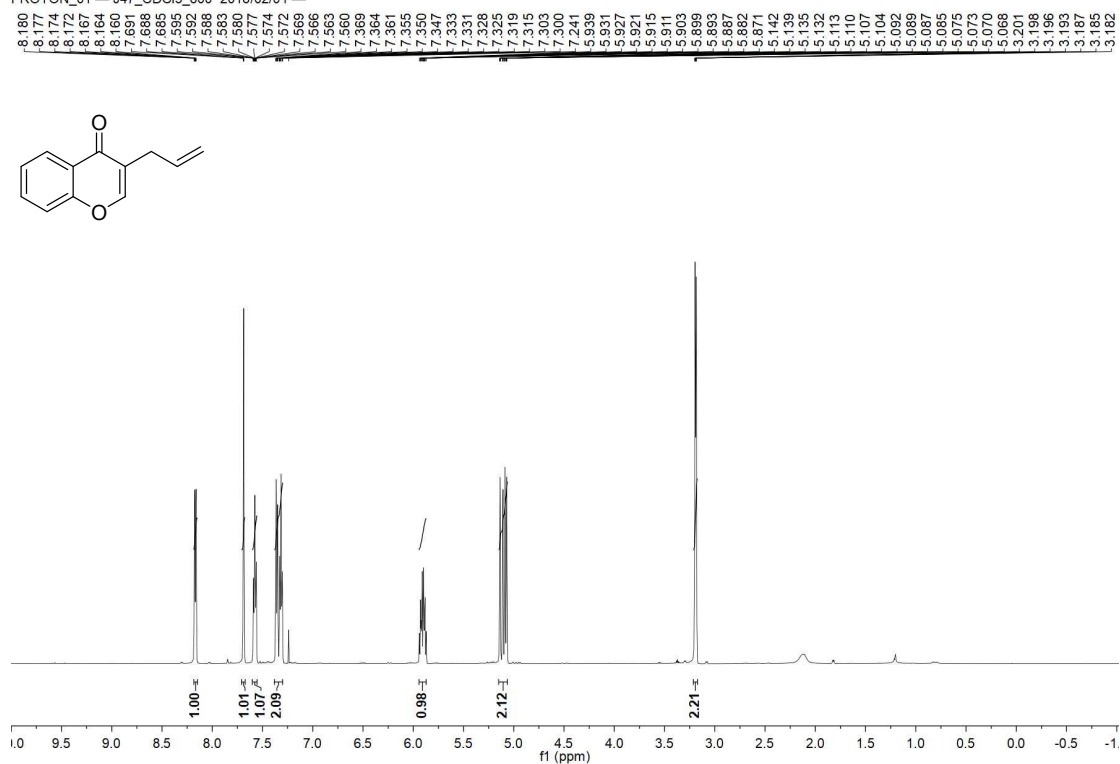
PROTON_01 — J45_CDCl3_600 2018/02/01 —



3-Allyl-4H-chromen-4-one (1b)

¹H NMR

PROTON_01 — J47_CDCl3_600 2018/02/01 —

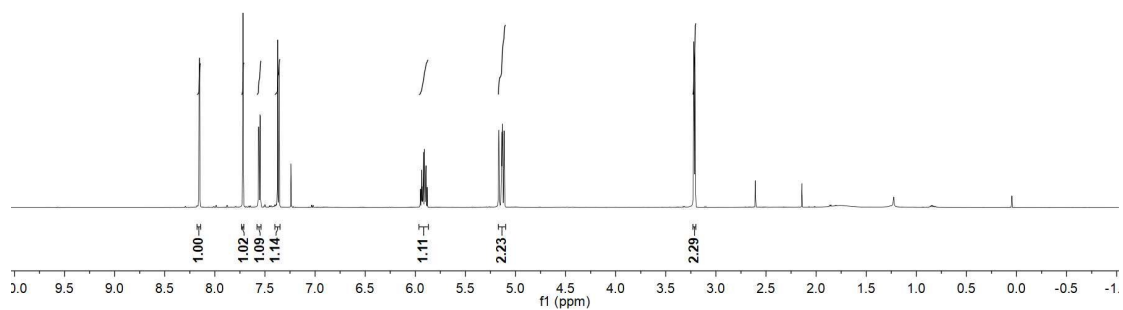
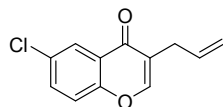


3-Allyl-6-chloro-4H-chromen-4-one (**1d**)

¹H NMR

PROTON_01 — J42_CDCl3_600 2018/02/01 —

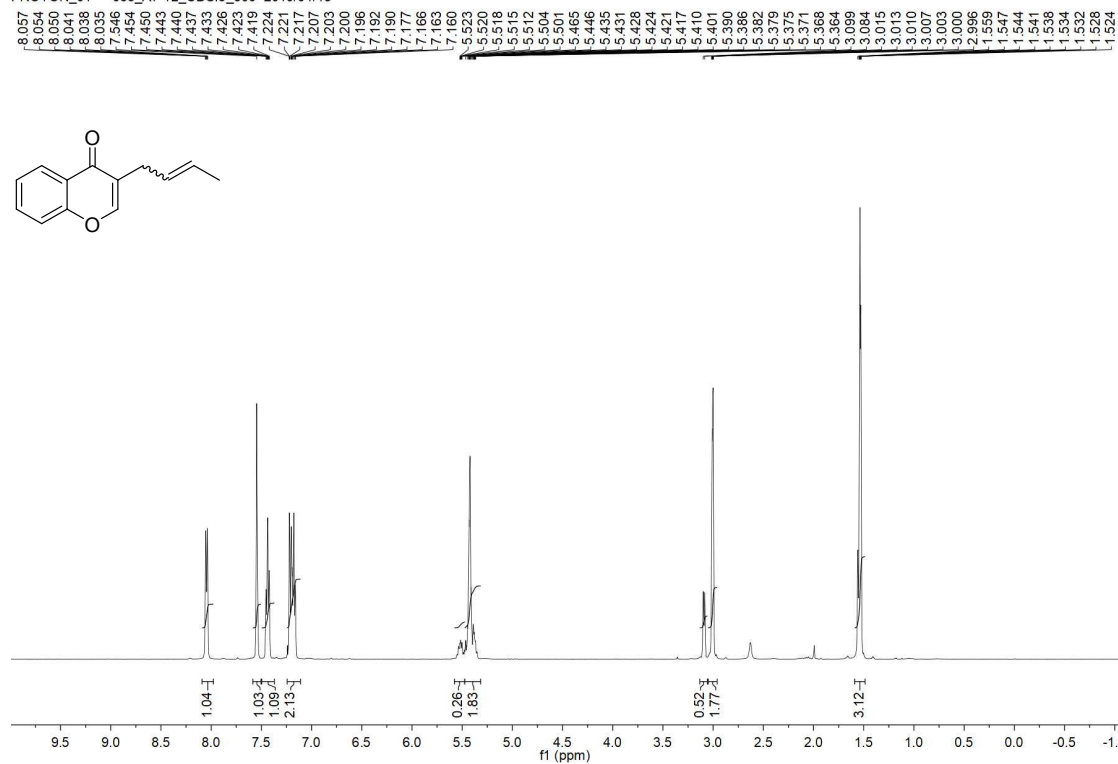
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5.139
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5.126
5.116
5.114
5.111
5.109
3.223
3.221
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3.216
3.212
3.209
3.207
3.205



(E)-3-(But-2-en-1-yl)-4H-chromen-4-one (**3a**)

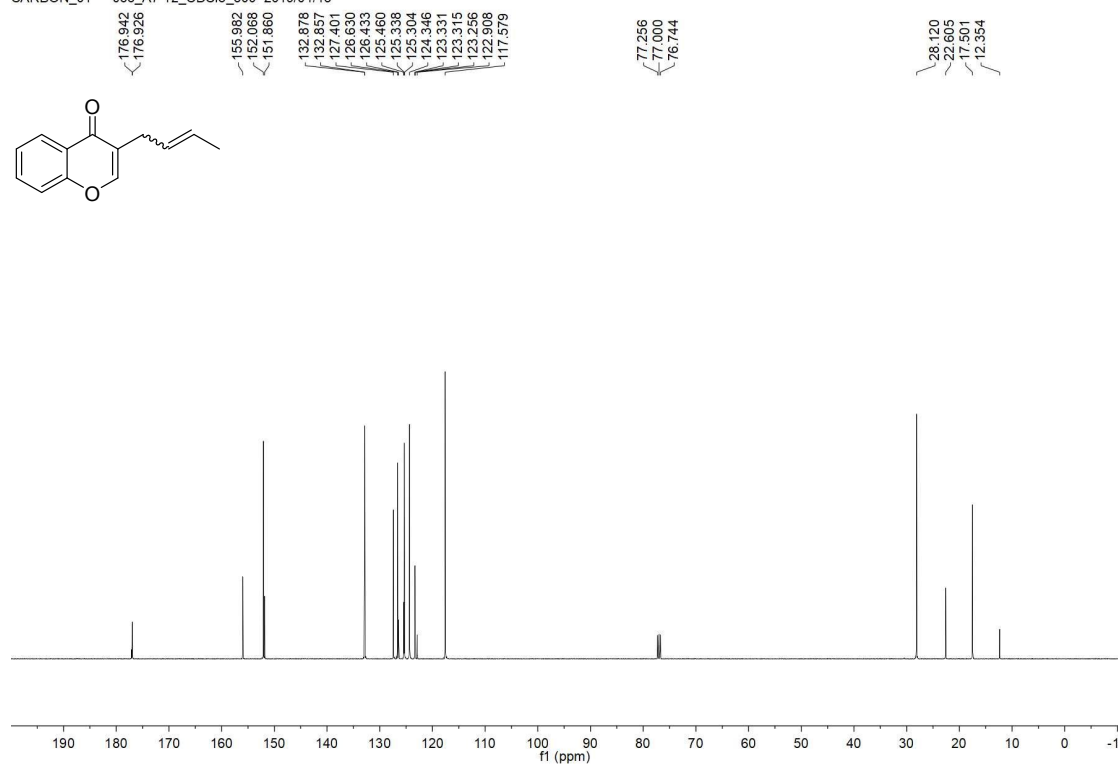
¹H NMR

PROTON_01 — J66_A7-12_CDCl3_500 2019/01/15 —



¹³C NMR

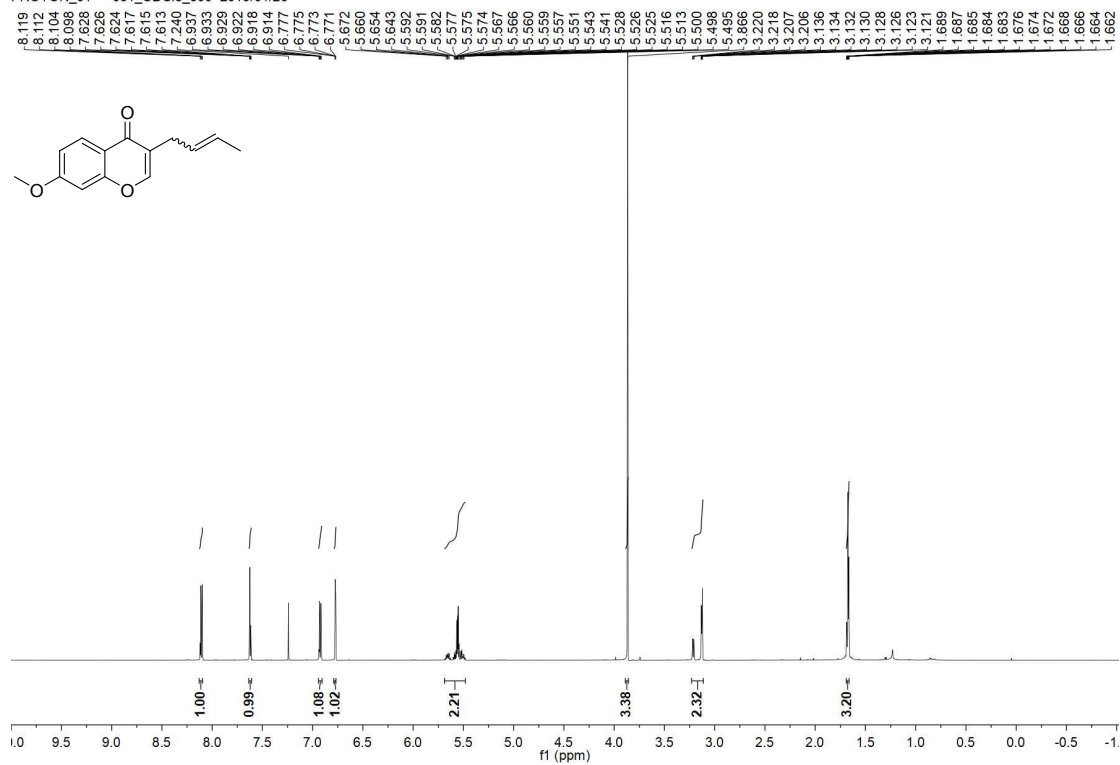
CARBON_01 — J66_A7-12_CDCl3_500 2019/01/15 —



3-(But-2-en-1-yl)-7-methoxy-4H-chromen-4-one (3b)

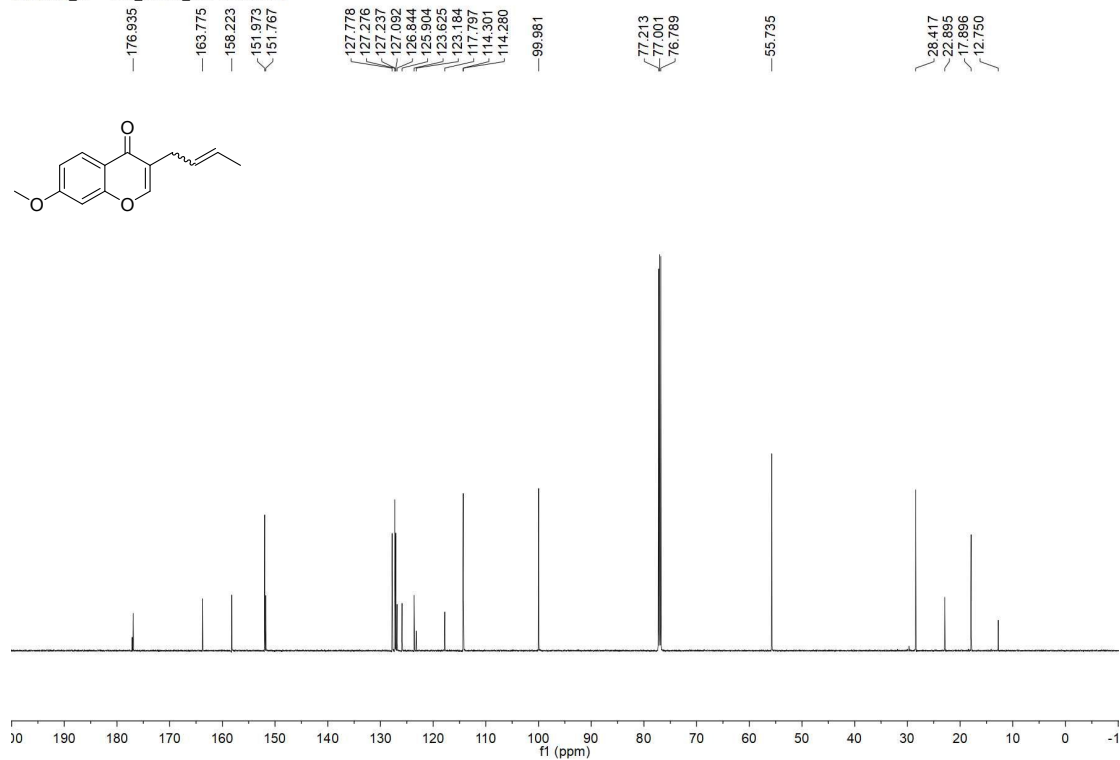
¹H NMR

PROTON_01 — J61_CDCl3_600 2018/01/29 —



¹³C NMR

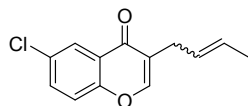
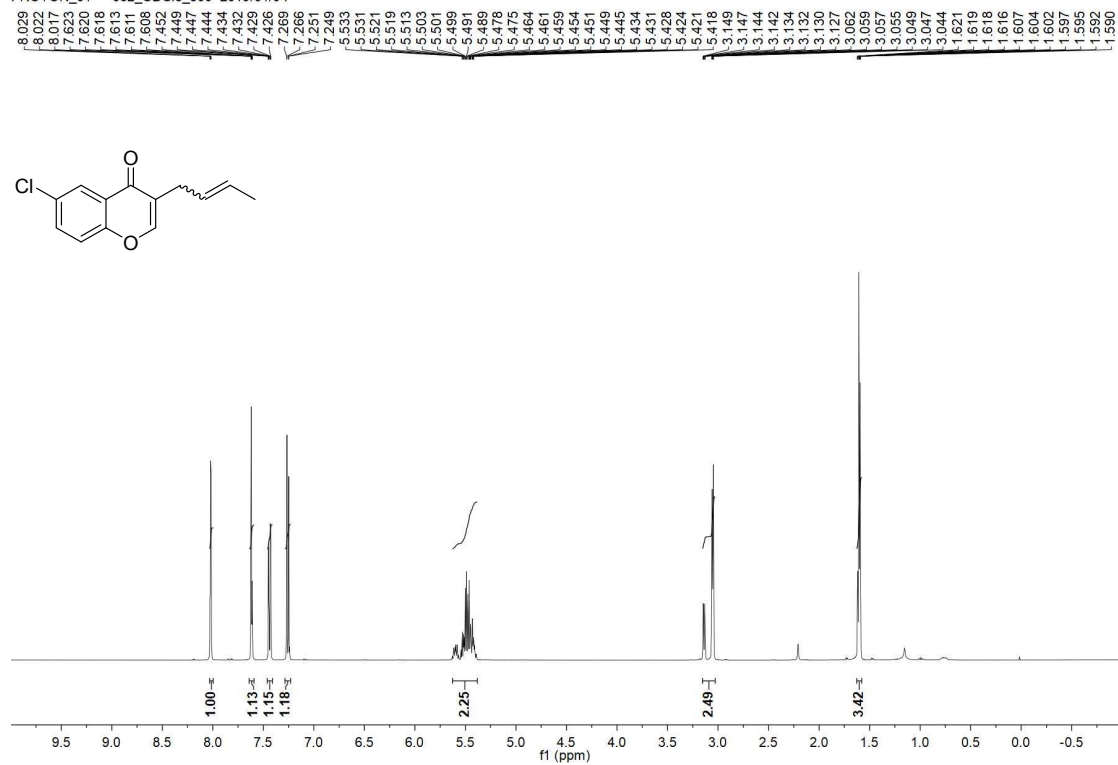
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3-(But-2-en-1-yl)-6-chloro-4H-chromen-4-one (3c)

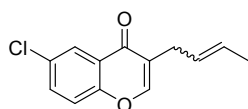
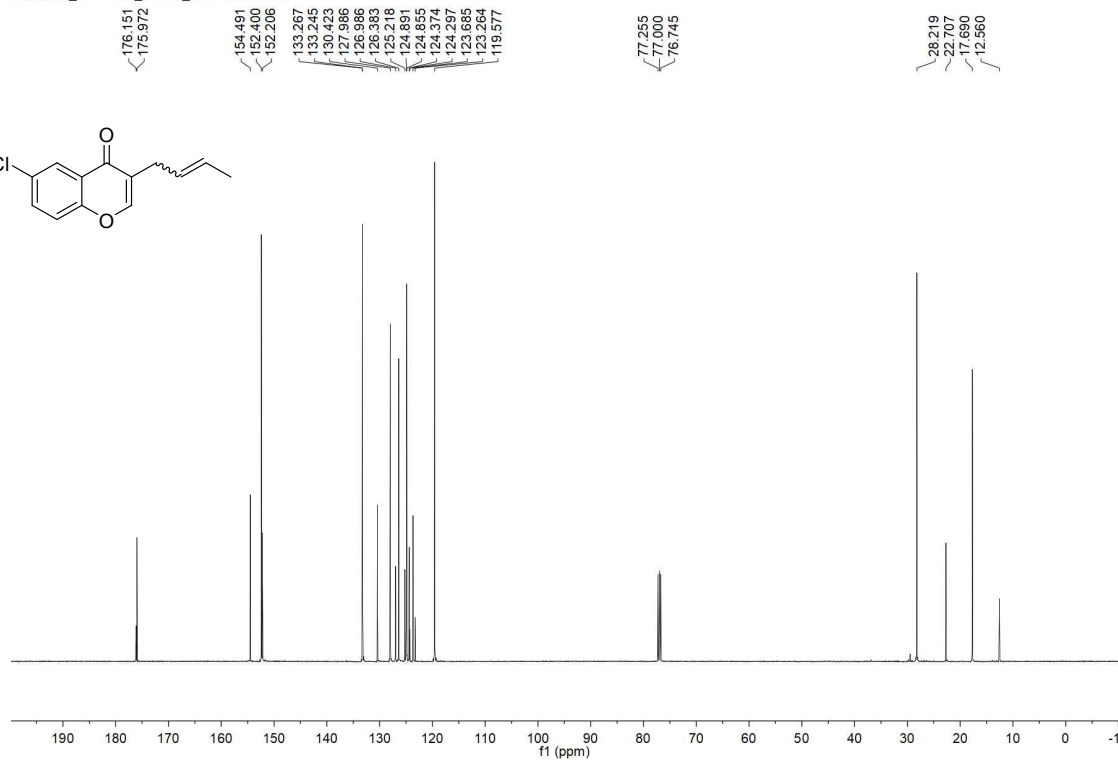
¹H NMR

PROTON_01 — J62_CDCl3_500 2019/01/04 —



¹³C NMR

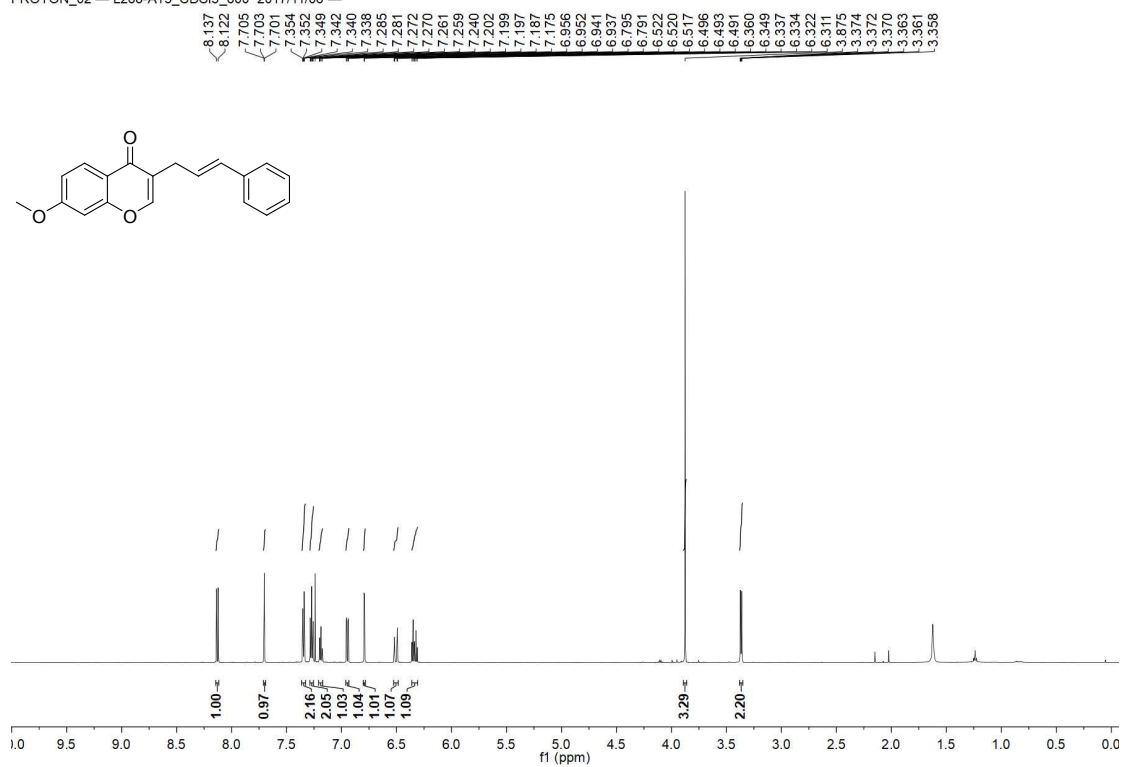
CARBON_01 — J62_CDCl3_500 2019/01/04 —



3-Cinnamyl-7-methoxy-4H-chromen-4-one (5a)

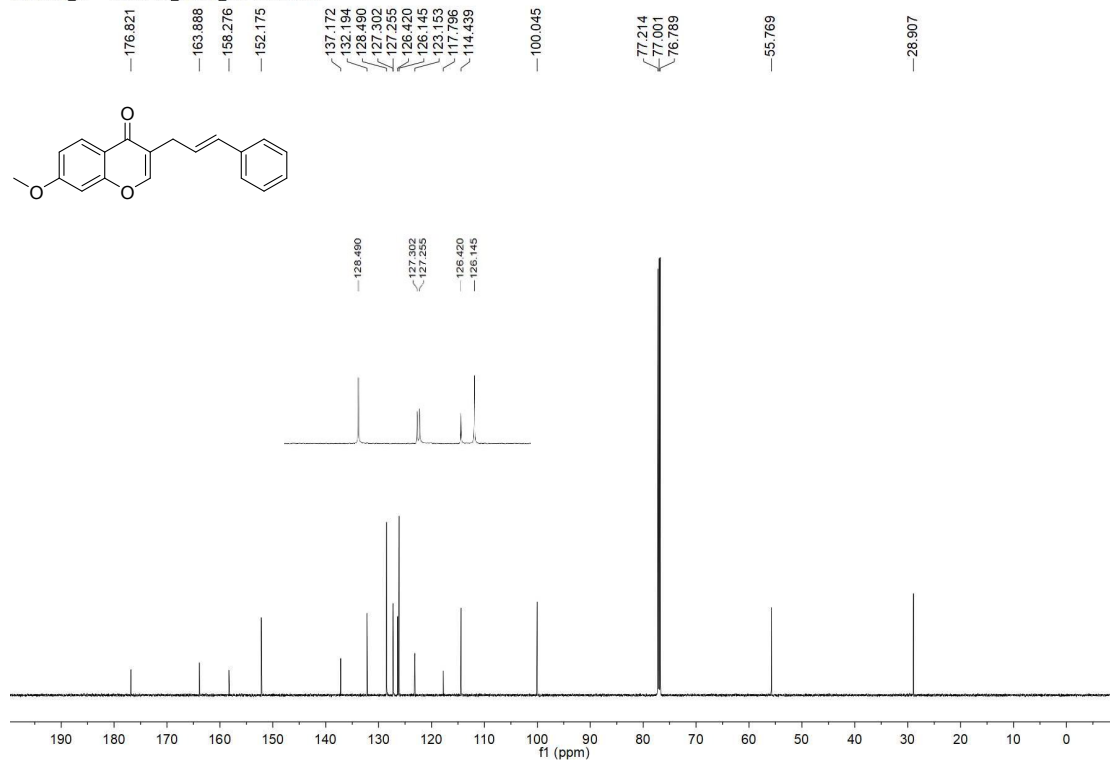
¹H NMR

PROTON_02 — L268-A15_CDCl3_600 2017/11/06 —



¹³C NMR

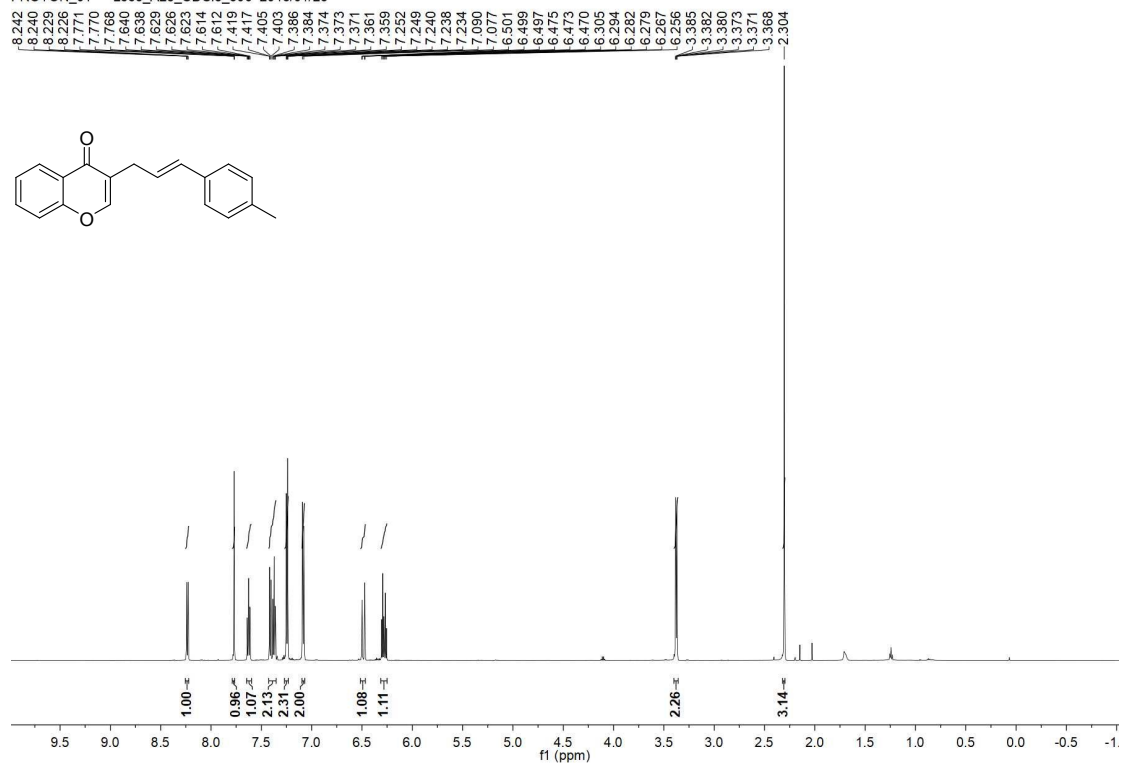
CARBON_01 — L268-A15_CDCl3_600 2017/11/06 —



(E)-3-(3-(p-Tolyl)allyl)-4H-chromen-4-one (5b)

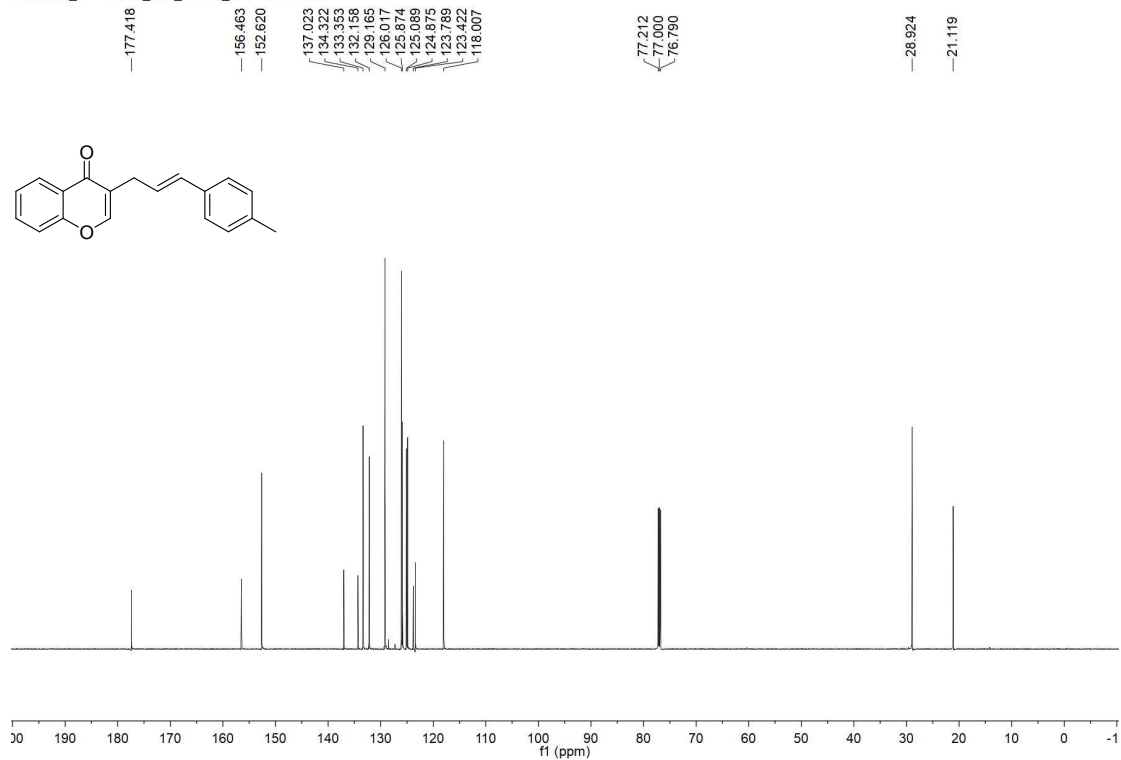
¹H NMR

PROTON_01 — L366_A25_CDCl3_600 2018/01/26 —



¹³C NMR

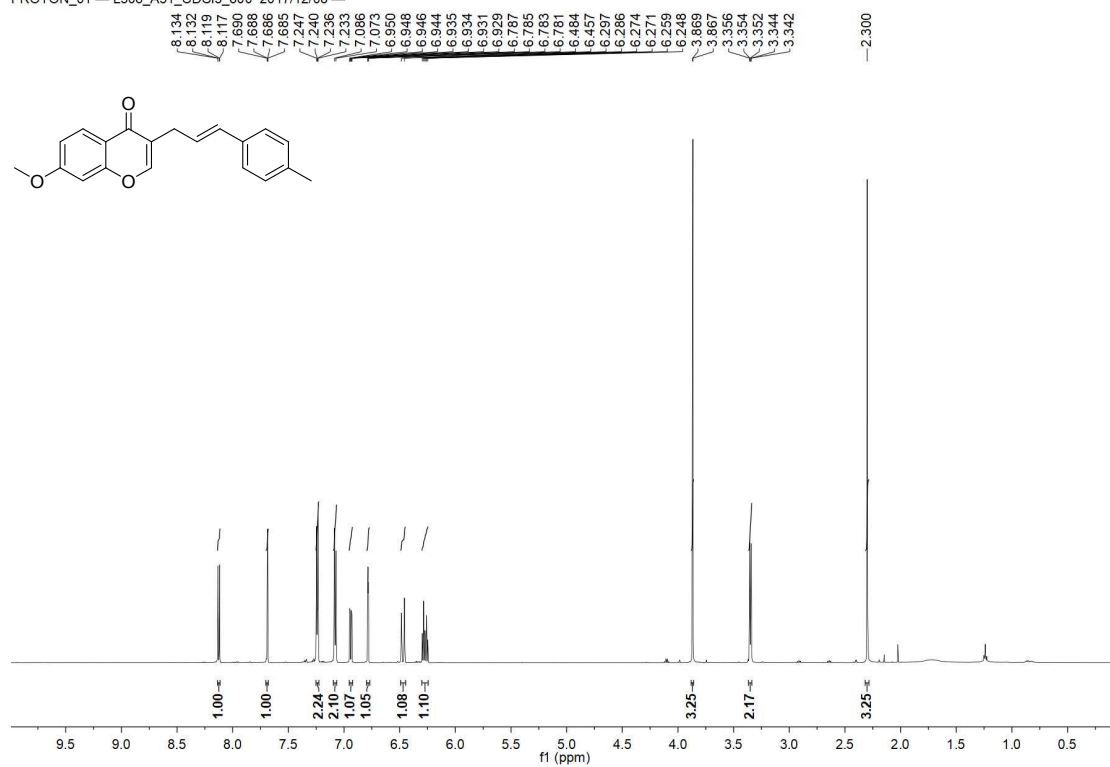
CARBON_01 — L366_A25_CDCl3_600 2018/01/26 —



(E)-7-Methoxy-3-(3-(*p*-tolyl)allyl)-4*H*-chromen-4-one (**5c**)

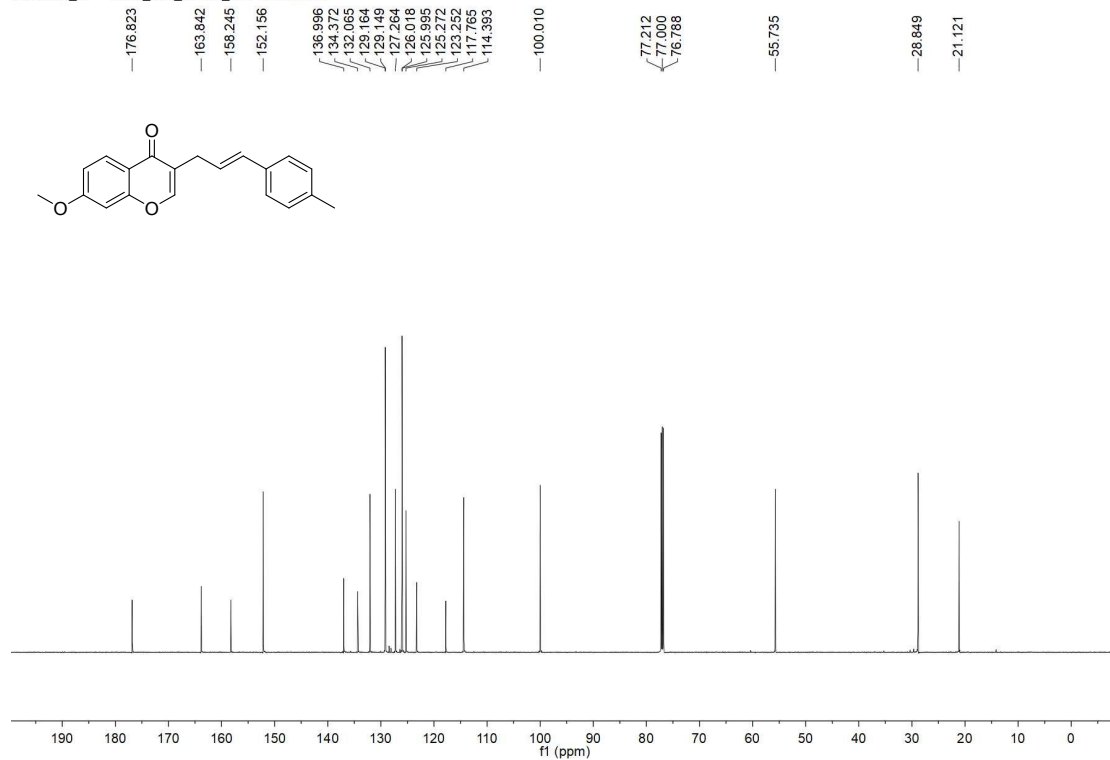
¹H NMR

PROTON_01 — L306_A51_CDCl3_600 2017/12/08 —



¹³C NMR

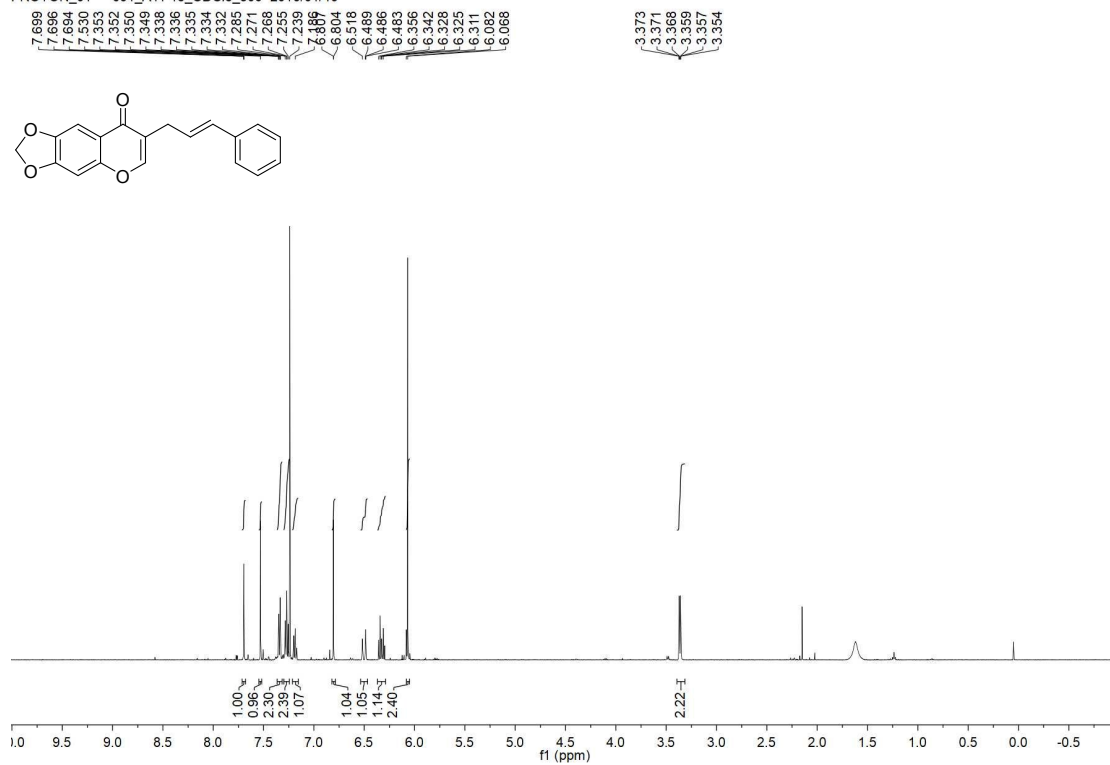
CARBON_01 — L306_A51_CDCl3_600 2017/12/08 —



(E)-7-(3-Phenylprop-1-en-1-yl)-8H-[1,3]dioxolo[4,5-g]chromen-8-one (**5f**)

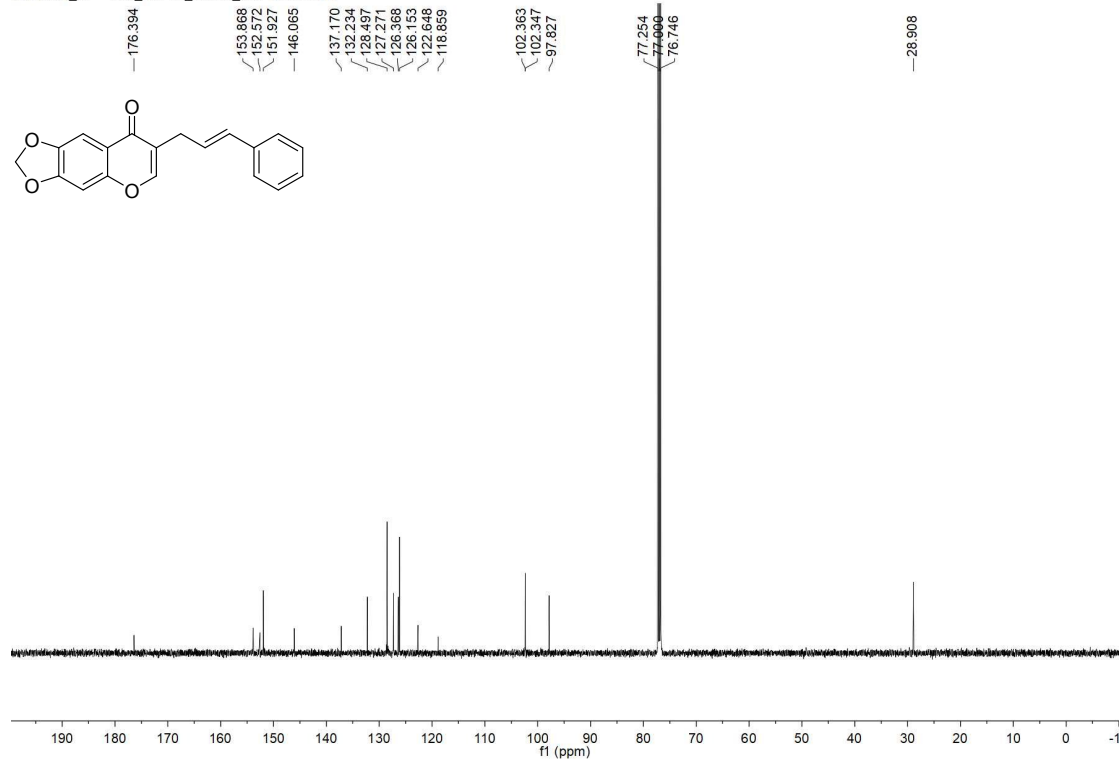
¹H NMR

PROTON_01 — J91_A11-15_CDCl3_500 2019/01/19 —



¹³C NMR

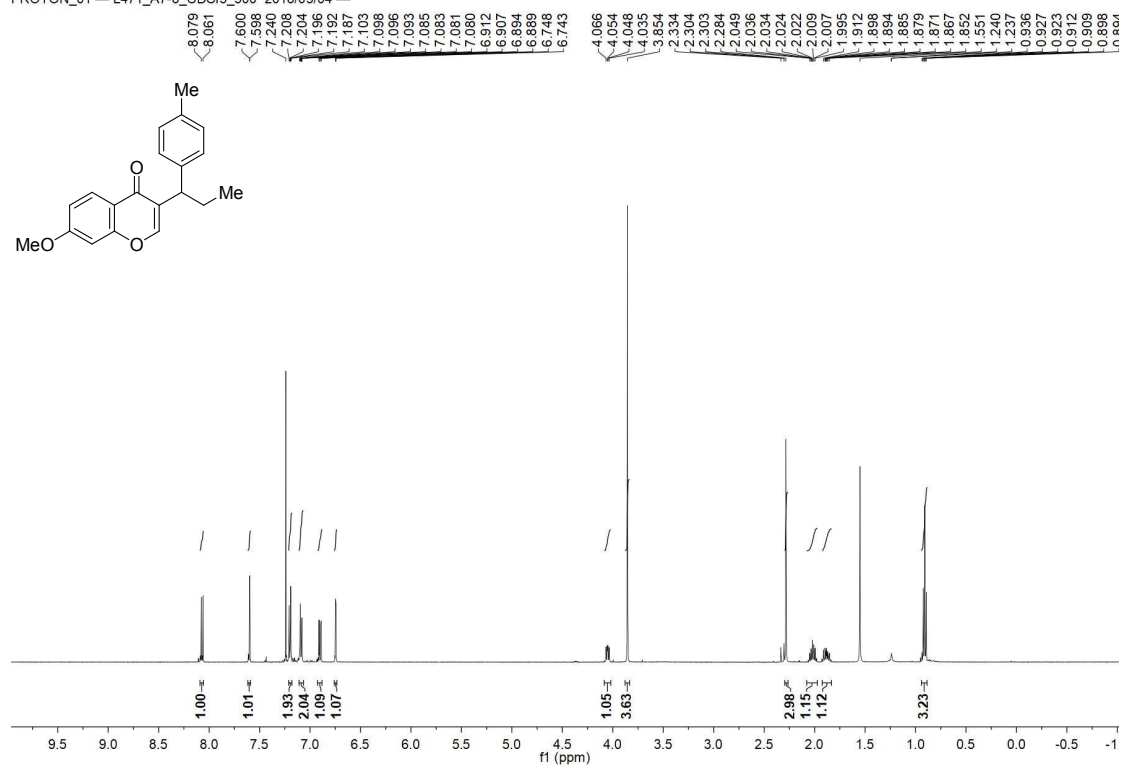
CARBON_01 — J91_A11-15_CDCl3_500 2019/01/19 —



7-Methoxy-3-(1-(p-tolyl)propyl)-4H-chromen-4-one (2a)

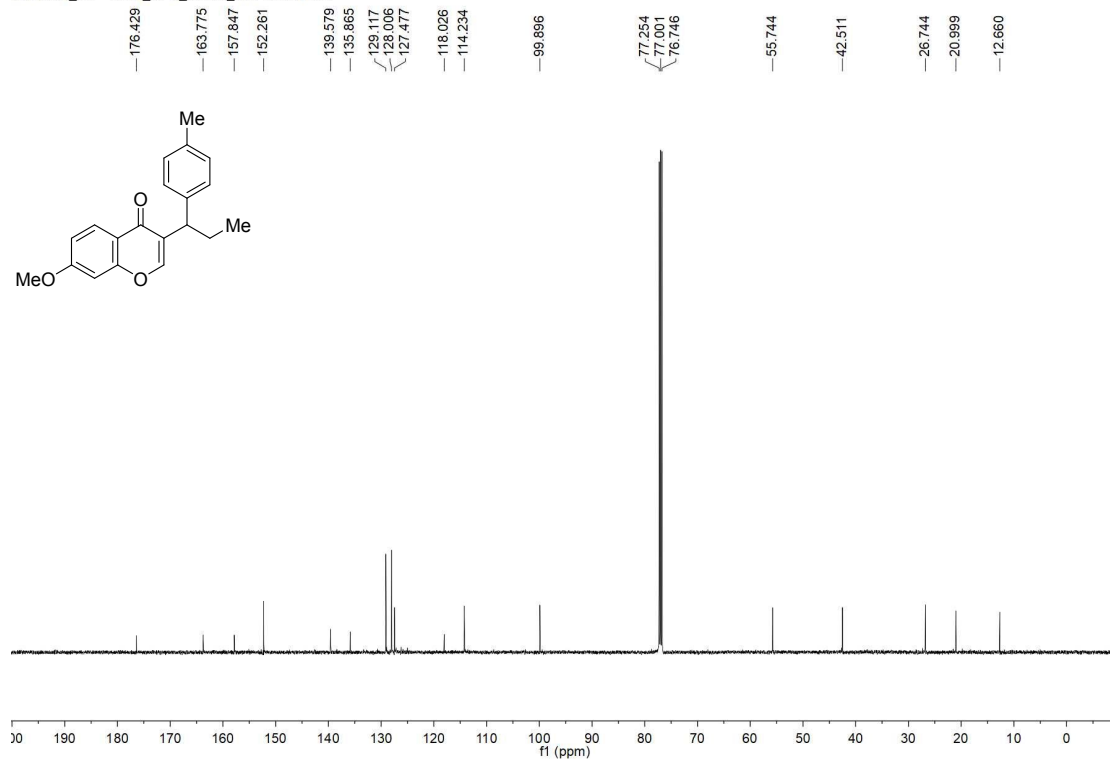
¹H NMR

PROTON_01 — L471_A7-8_CDCl3_500 2018/05/04 —



¹³C NMR

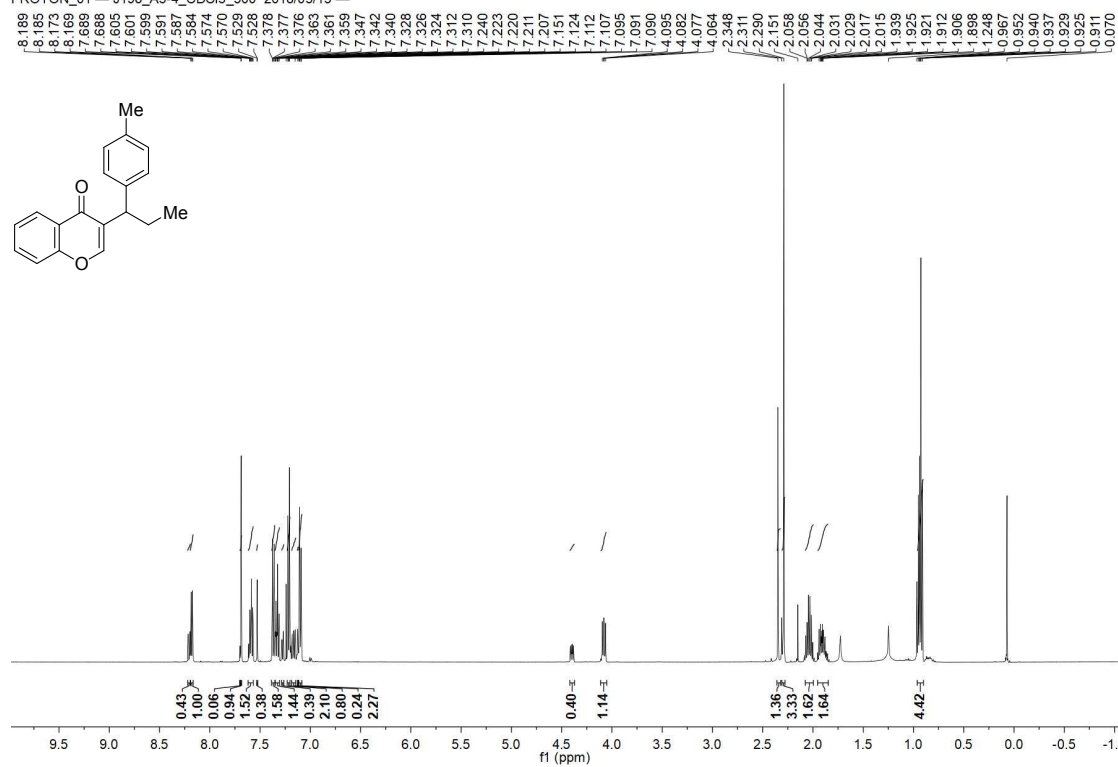
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3-(1-(p-Tolyl)propyl)-4H-chromen-4-one (2b)

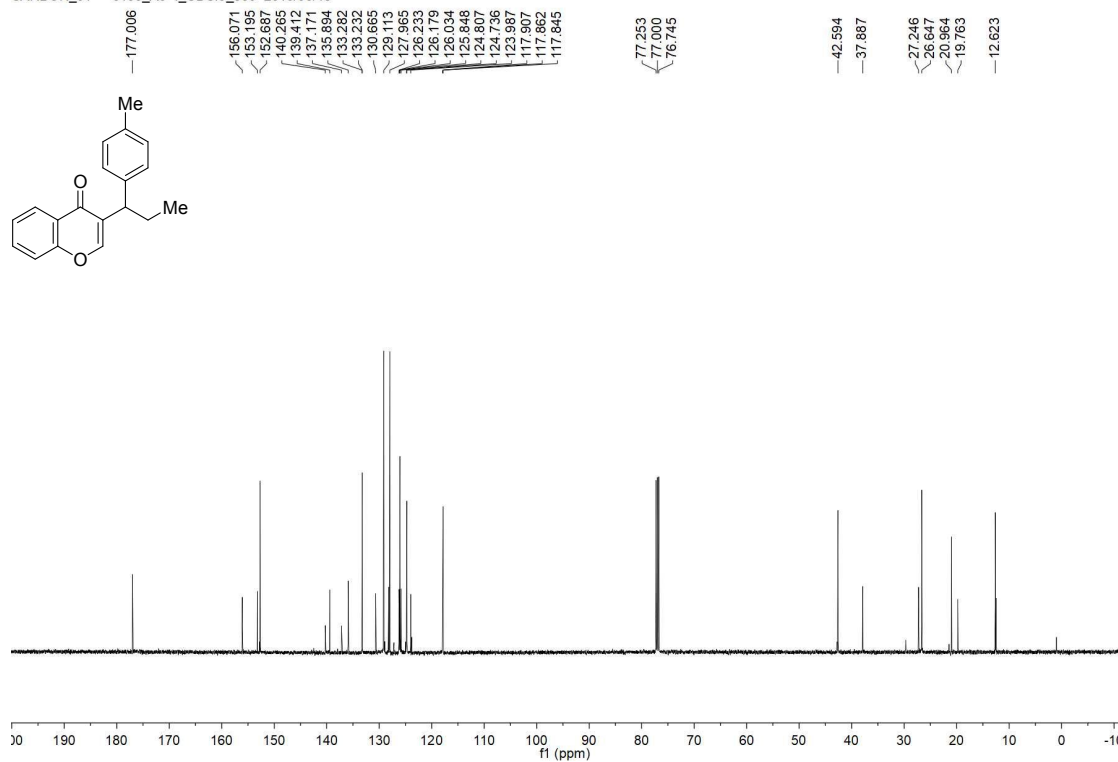
¹H NMR

PROTON_01 — J198_A3-4_CDCl3_500 2018/05/15 —



¹³C NMR

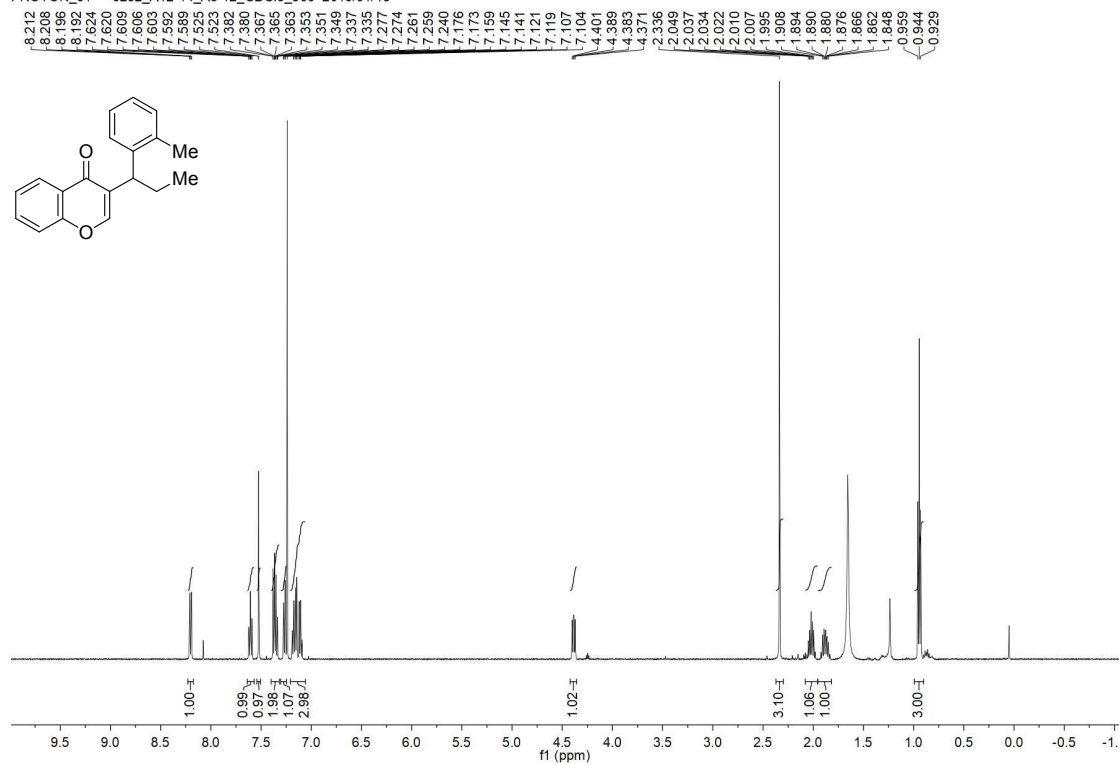
CARBON_01 — J198_A3-4_CDCl3_500 2018/05/15 —



3-(1-(*o*-Tolyl)propyl)-4H-chromen-4-one (2b')

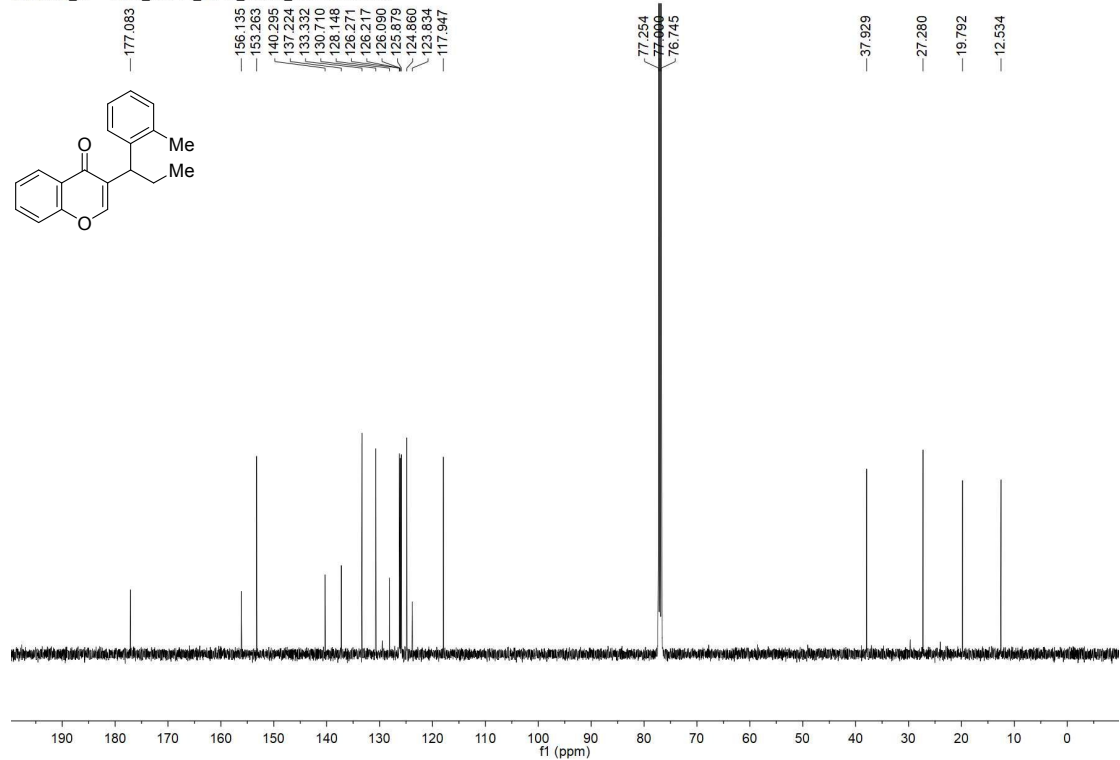
¹H NMR

PROTON_01 — J232_A12-14_A8-12_CDCl3_500 2019/01/15 —



¹³C NMR

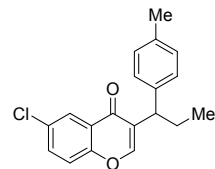
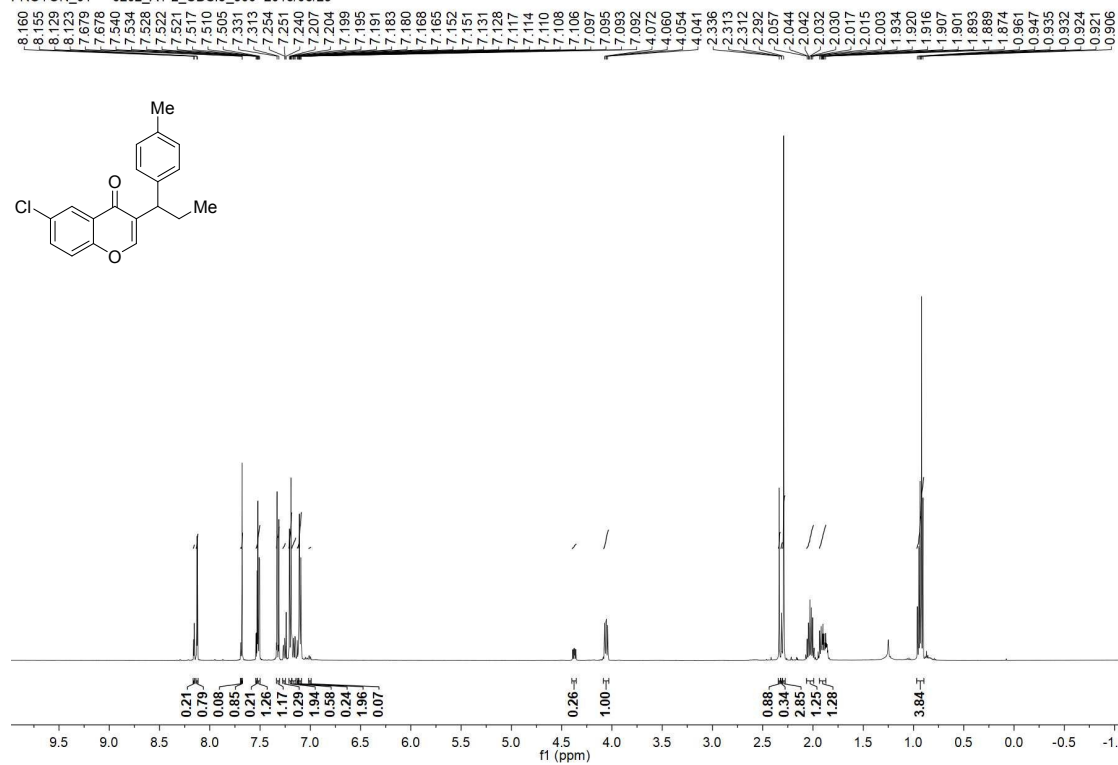
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6-Chloro-3-(1-(p-tolyl)propyl)-4H-chromen-4-one (2d)

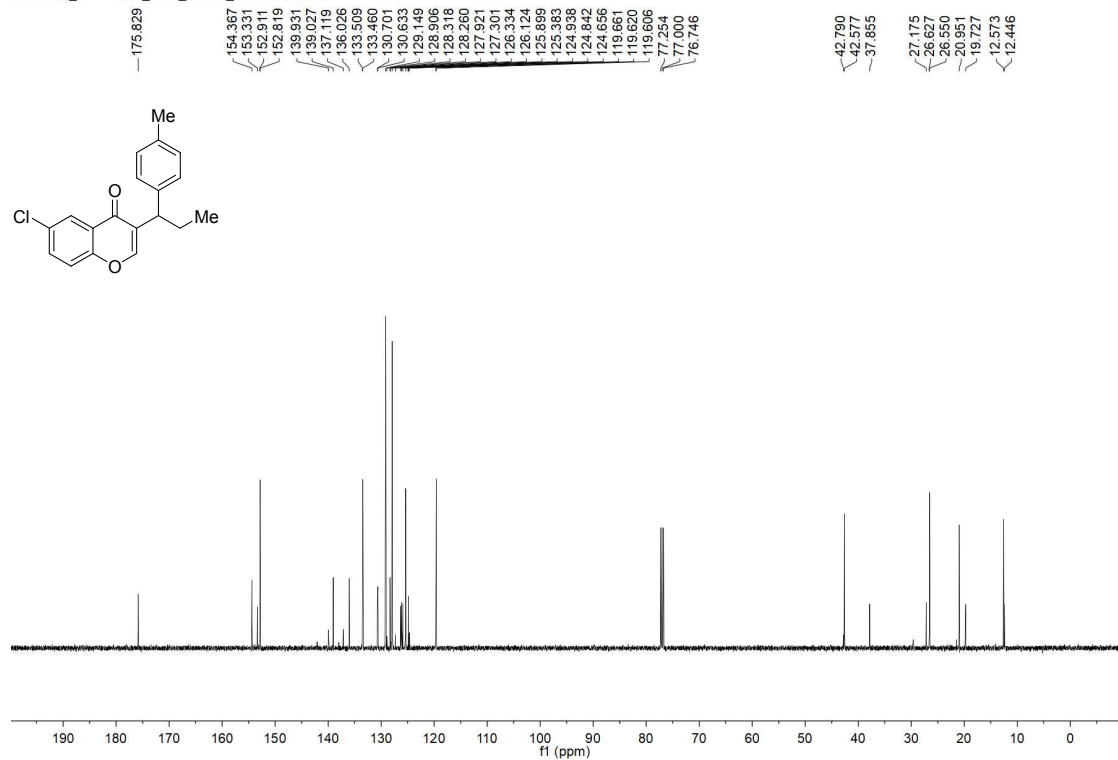
¹H NMR

PROTON_01 — J202_A1-2_CDCl3_500 2018/05/25 —



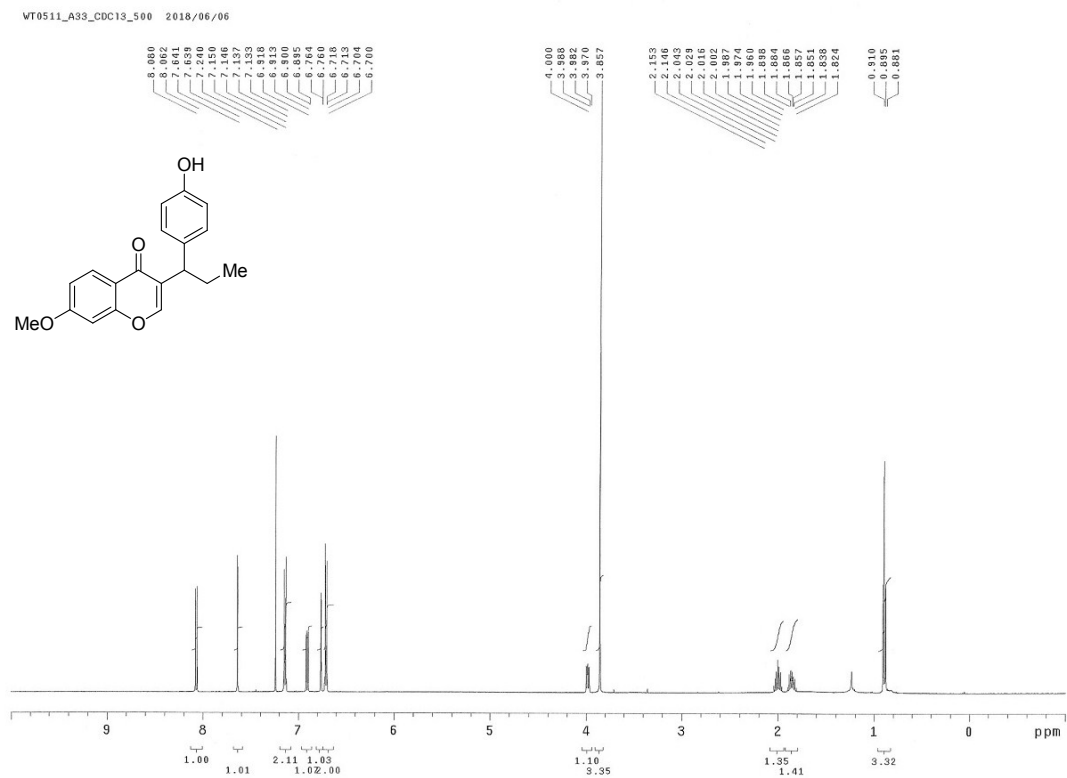
¹³C NMR

CARBON_01 — J202_A1-2_CDCl3_500 2018/05/25 —

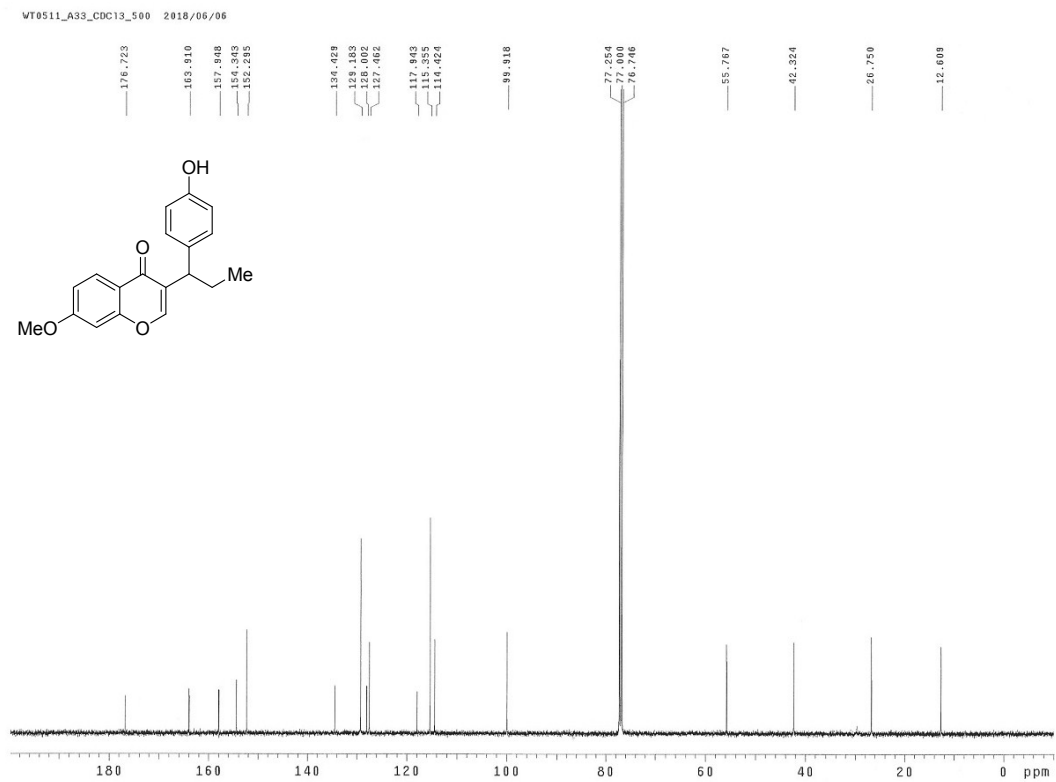


3-(1-(4-Hydroxyphenyl)propyl)-7-methoxy-4H-chromen-4-one (2e).

¹H NMR

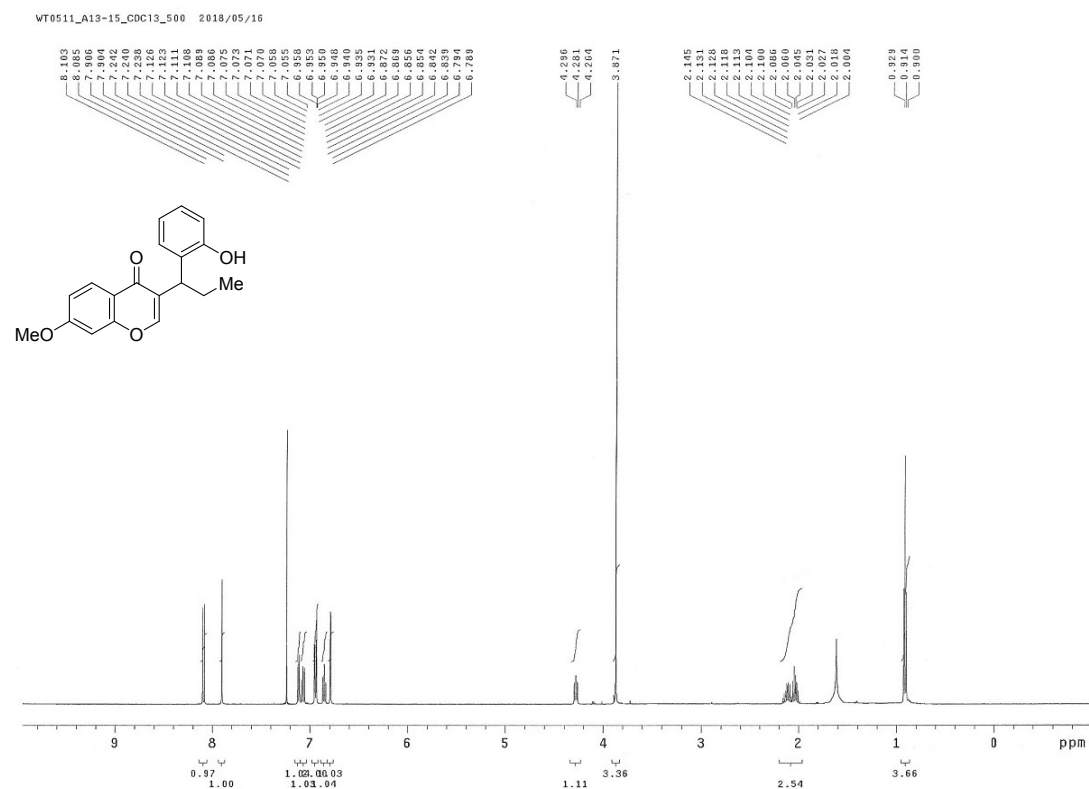


¹³C NMR

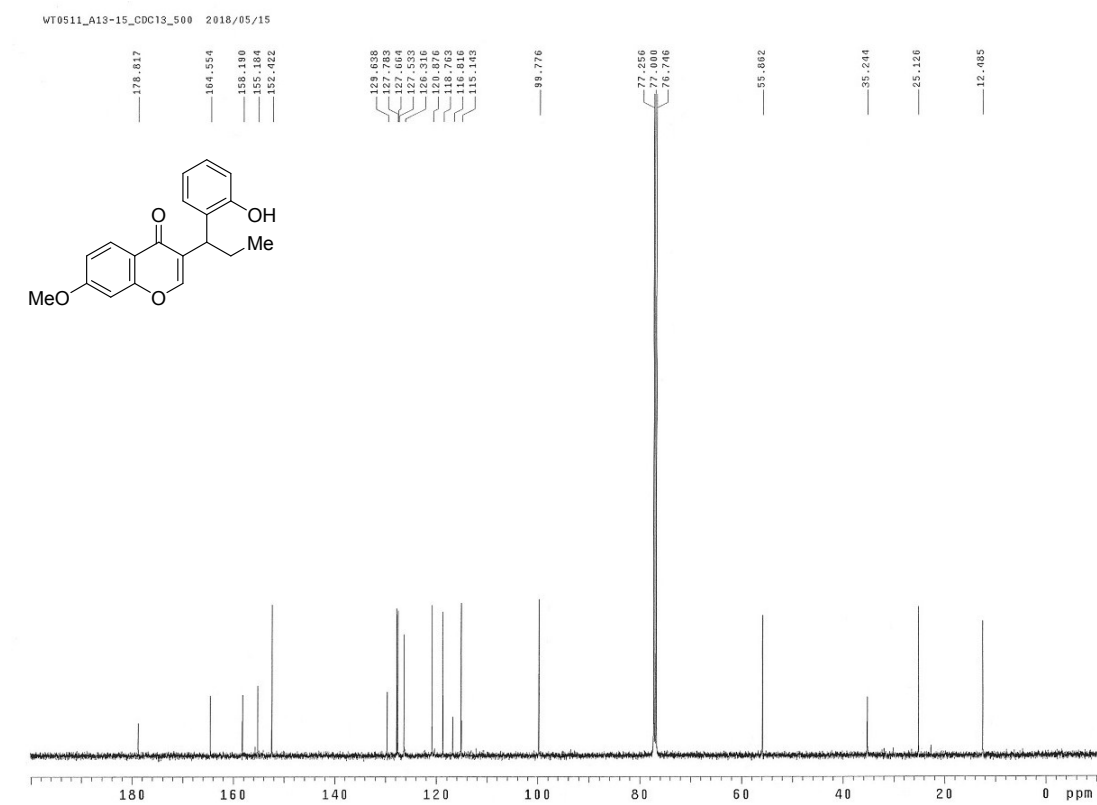


3-(1-(2-Hydroxyphenyl)propyl)-7-methoxy-4H-chromen-4-one (2e').

¹H NMR



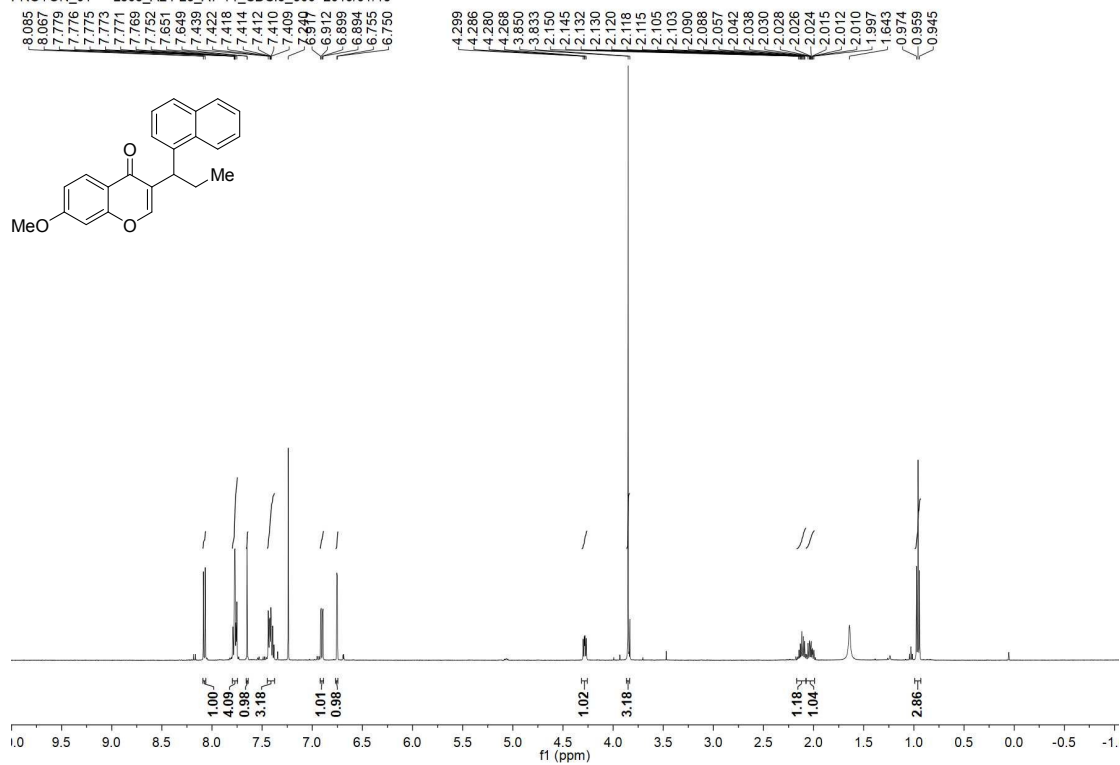
¹³C NMR



7-Methoxy-3-(1-(naphthalen-1-yl)propyl)-4H-chromen-4-one (2f)

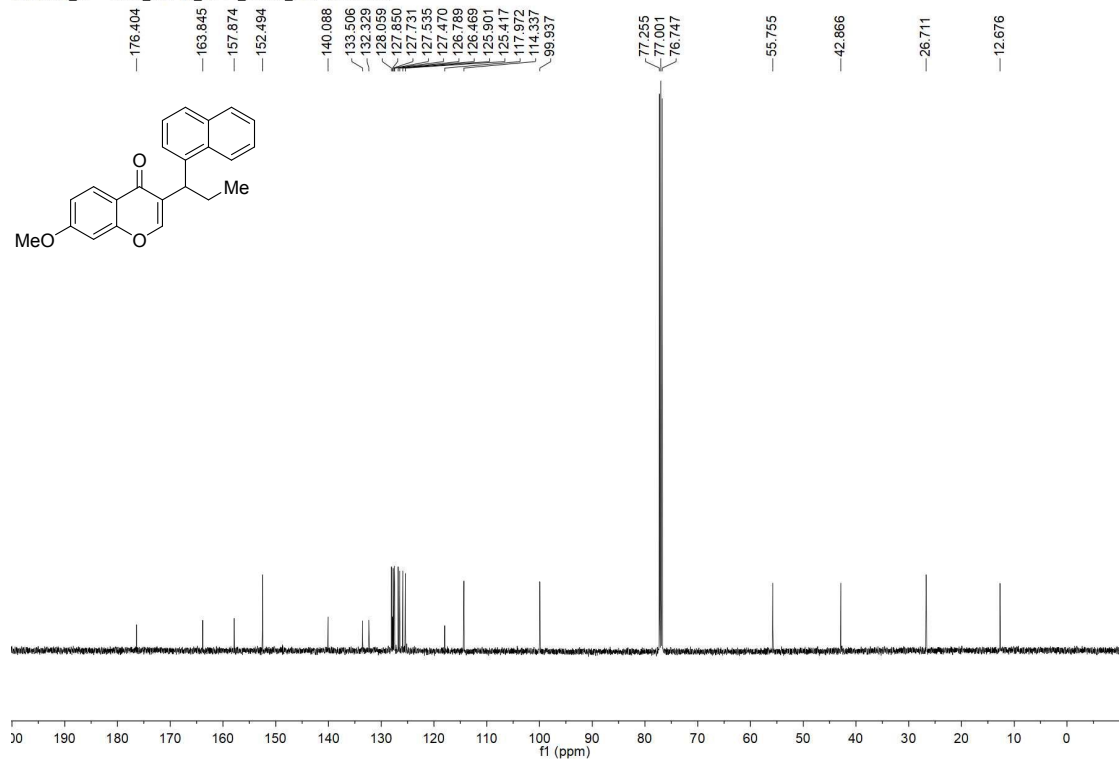
¹H NMR

PROTON_01 — L503_A24-28_A7-14_CDCl3_500 2019/01/15 —



¹³C NMR

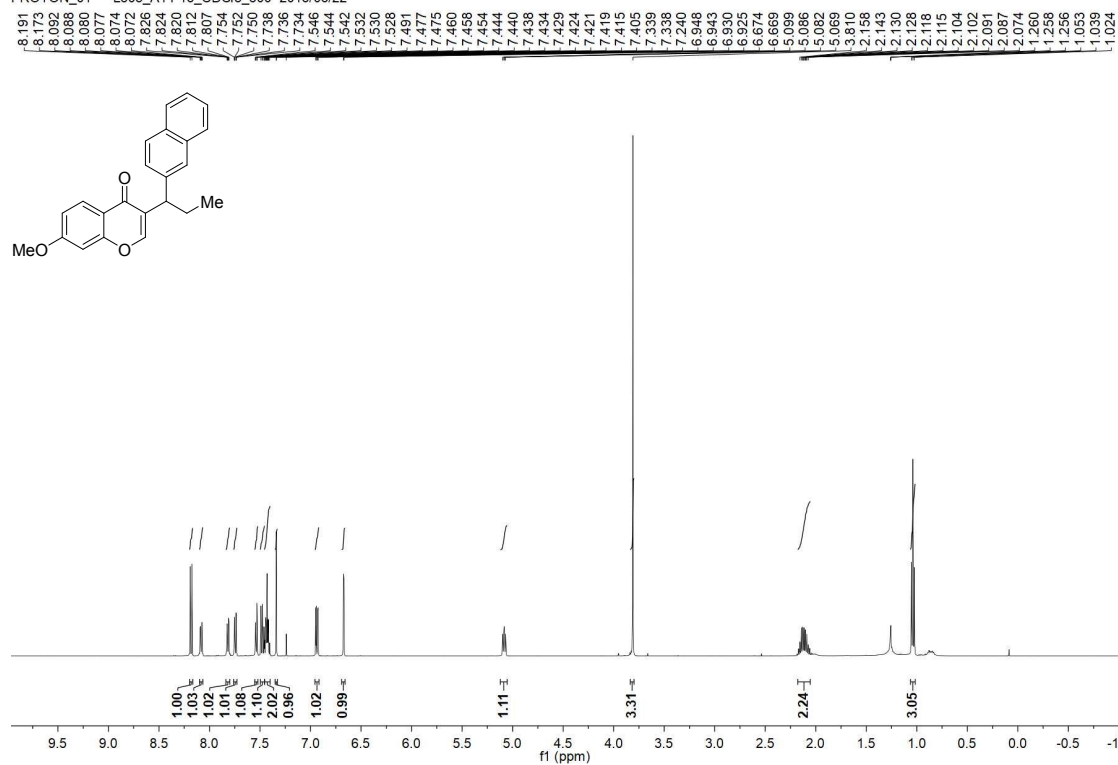
CARBON_01 — L503_A24-28_A7-14_CDCl3_500 2019/01/15 —



7-Methoxy-3-(1-(naphthalen-2-yl)propyl)-4H-chromen-4-one (2f')

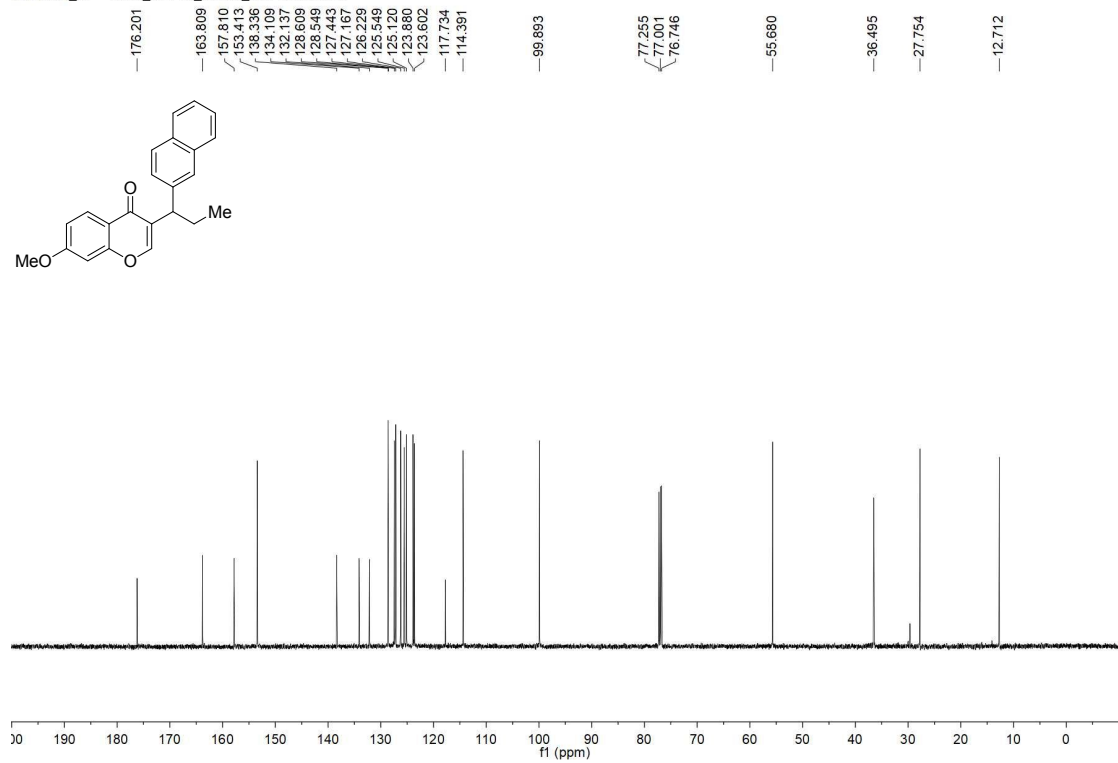
¹H NMR

PROTON_01 — L503_A14-18_CDCl3_500 2018/06/22 —



¹³C NMR

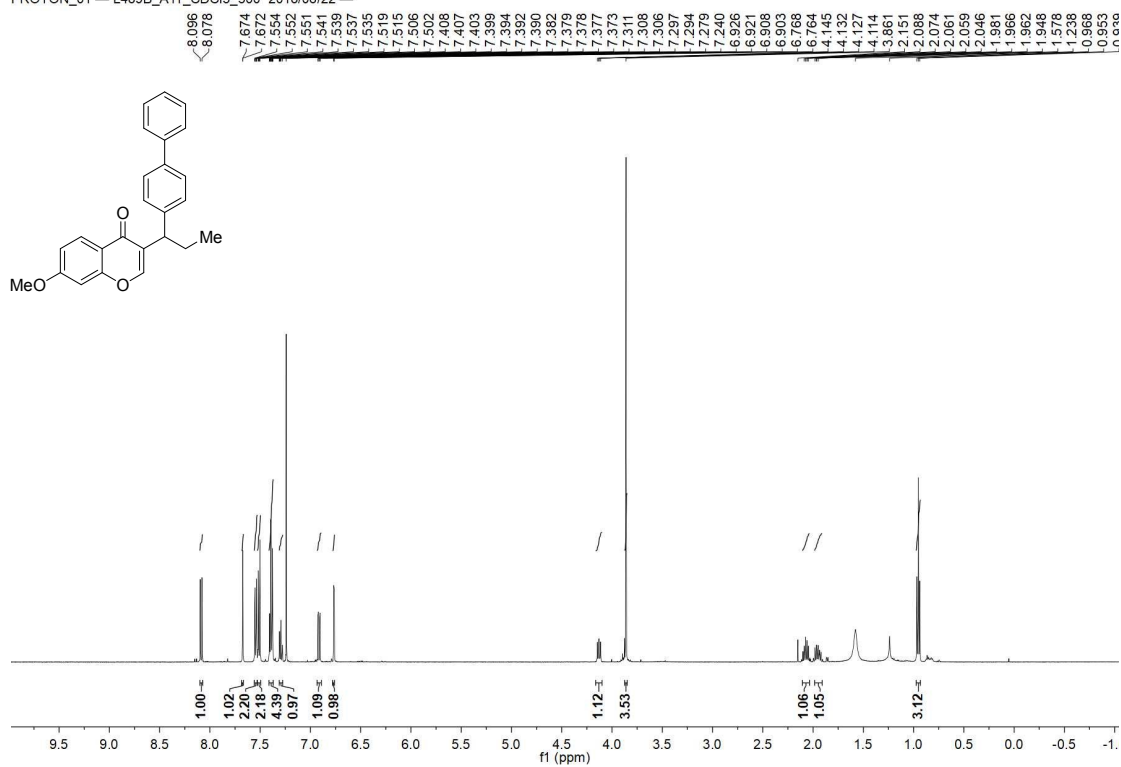
CARBON_01 — L503_A14-18_CDCl3_500 2018/06/22 —



3-(1-([1,1'-Biphenyl]-4-yl)propyl)-7-methoxy-4H-chromen-4-one (2h)

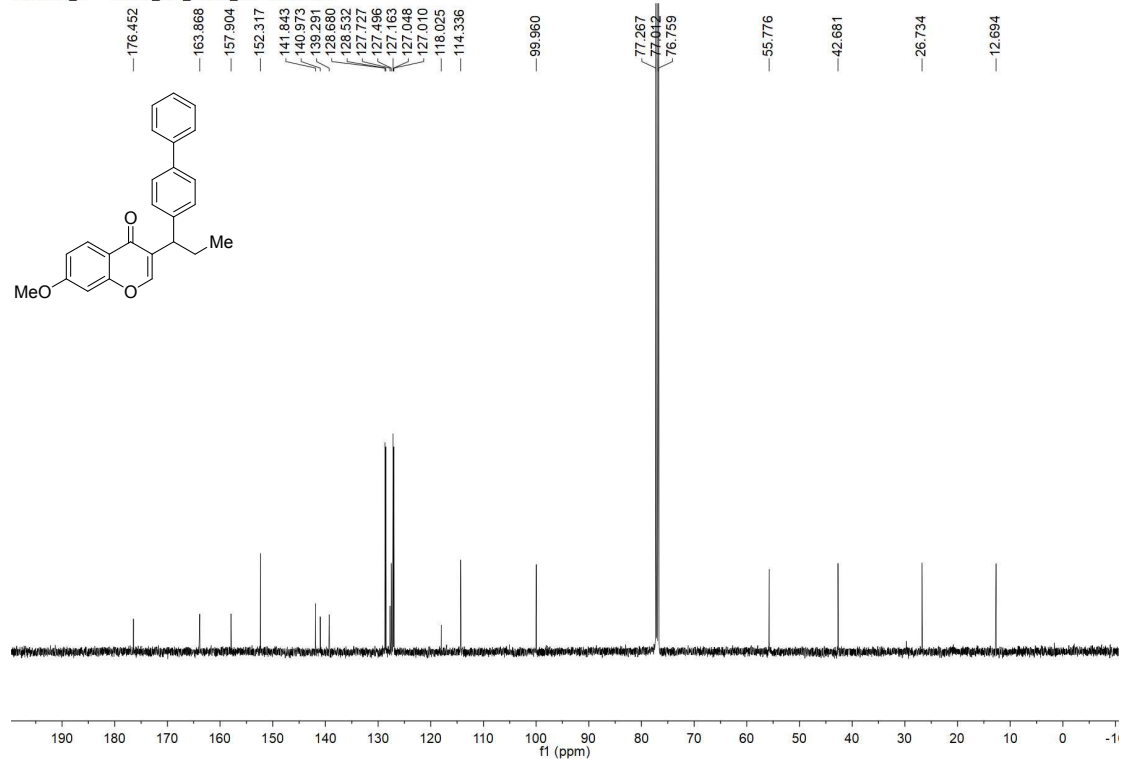
¹H NMR

PROTON_01 — L489B_A11_CDCI3_500 2018/06/22 —



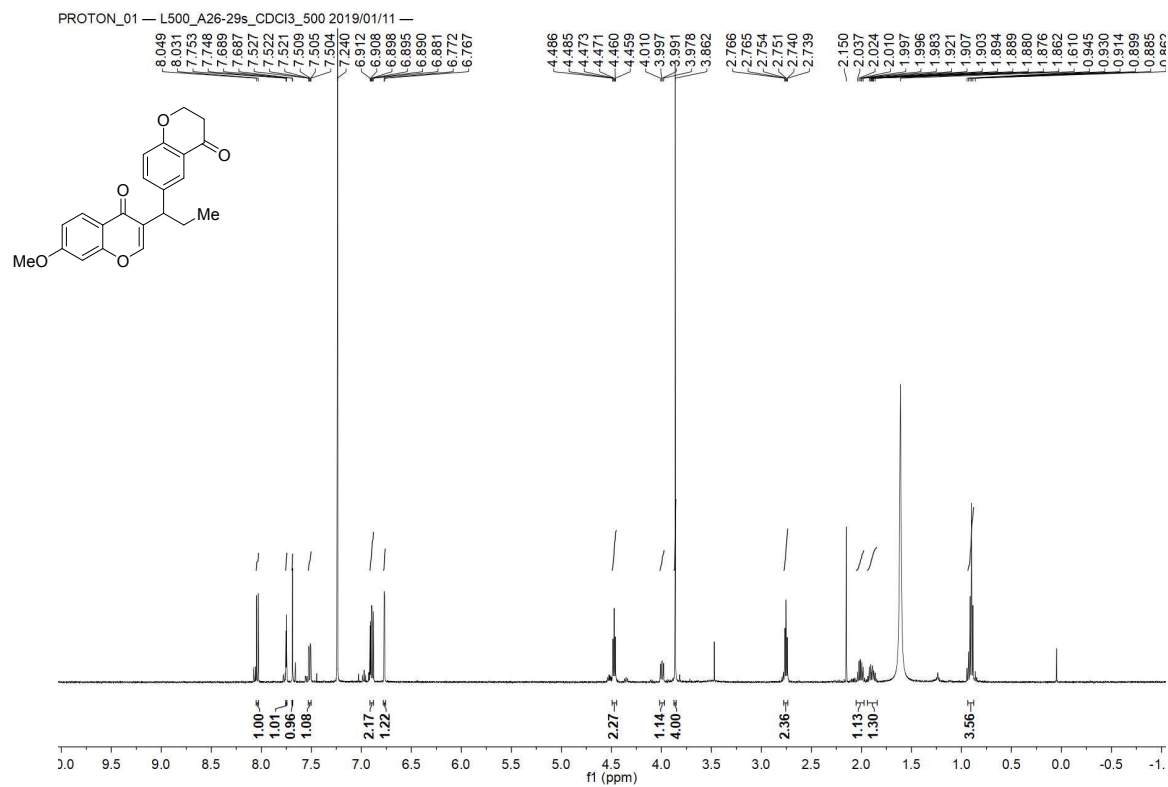
¹³C NMR

CARBON_01 — L489B_A11_CDCI3_500 2018/06/22 —

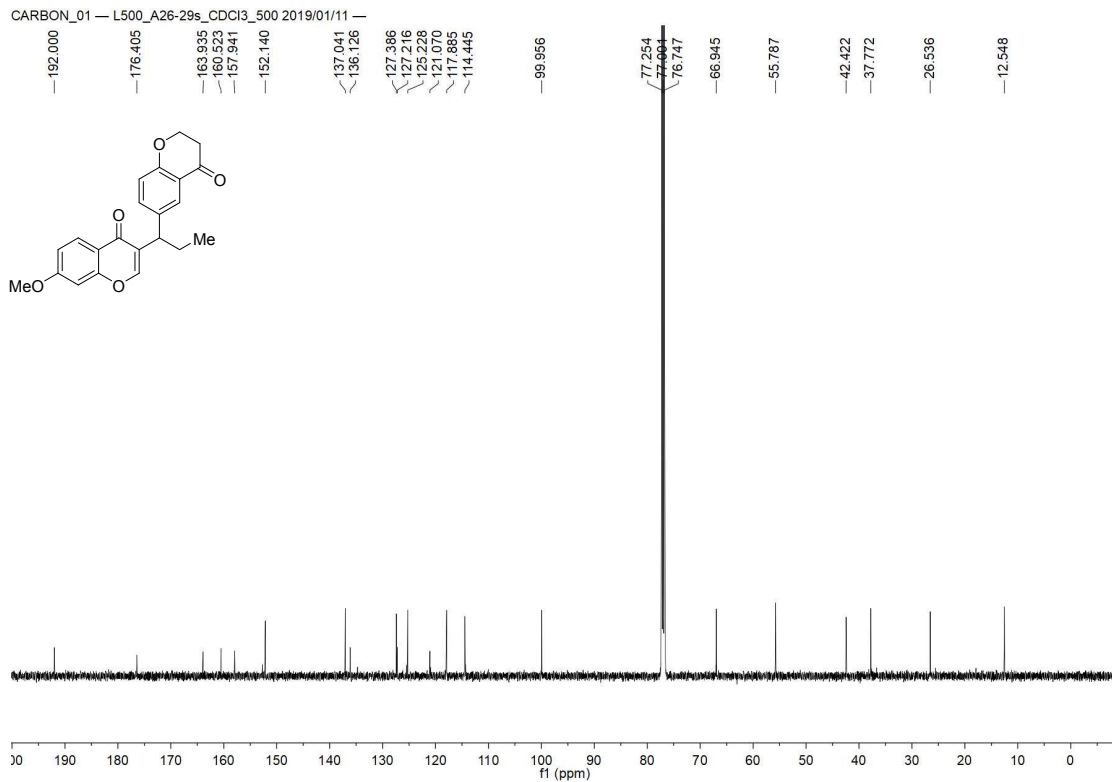


7-Methoxy-3-(1-(4-oxochroman-6-yl)propyl)-4H-chromen-4-one (**2i**)

¹H NMR



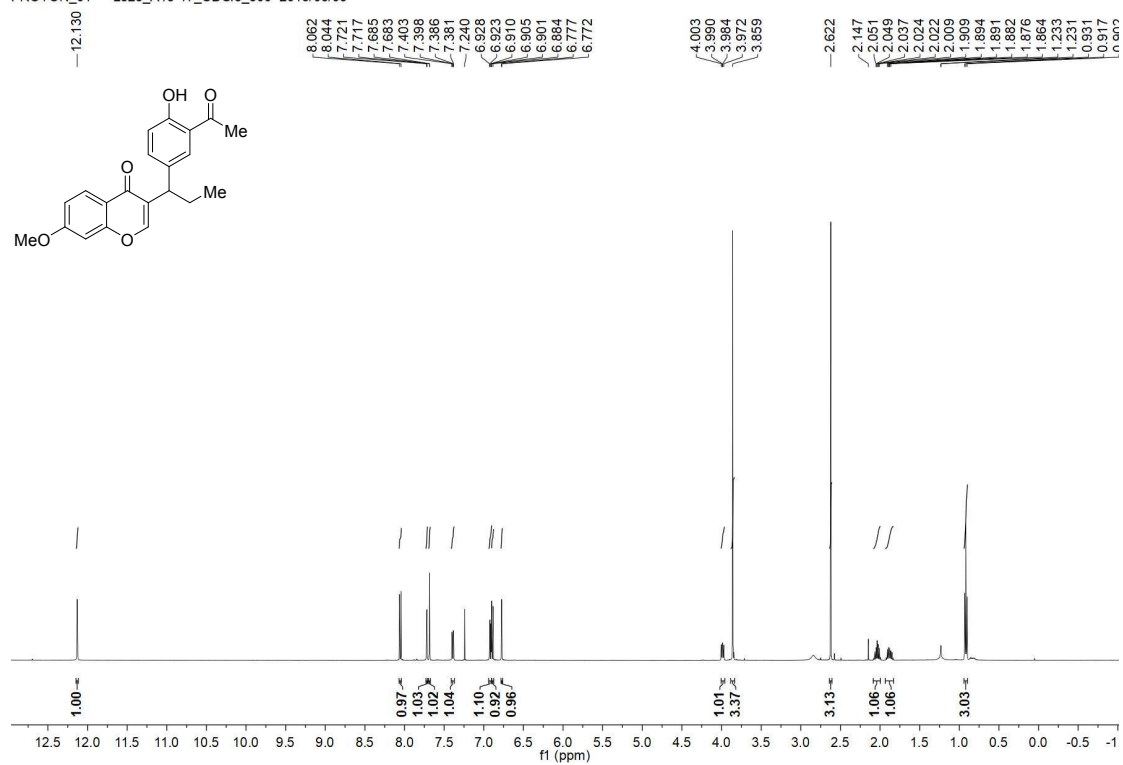
¹³C NMR



3-(1-(4-Acetyl-3-hydroxyphenyl)propyl)-7-methoxy-4H-chromen-4-one (21)

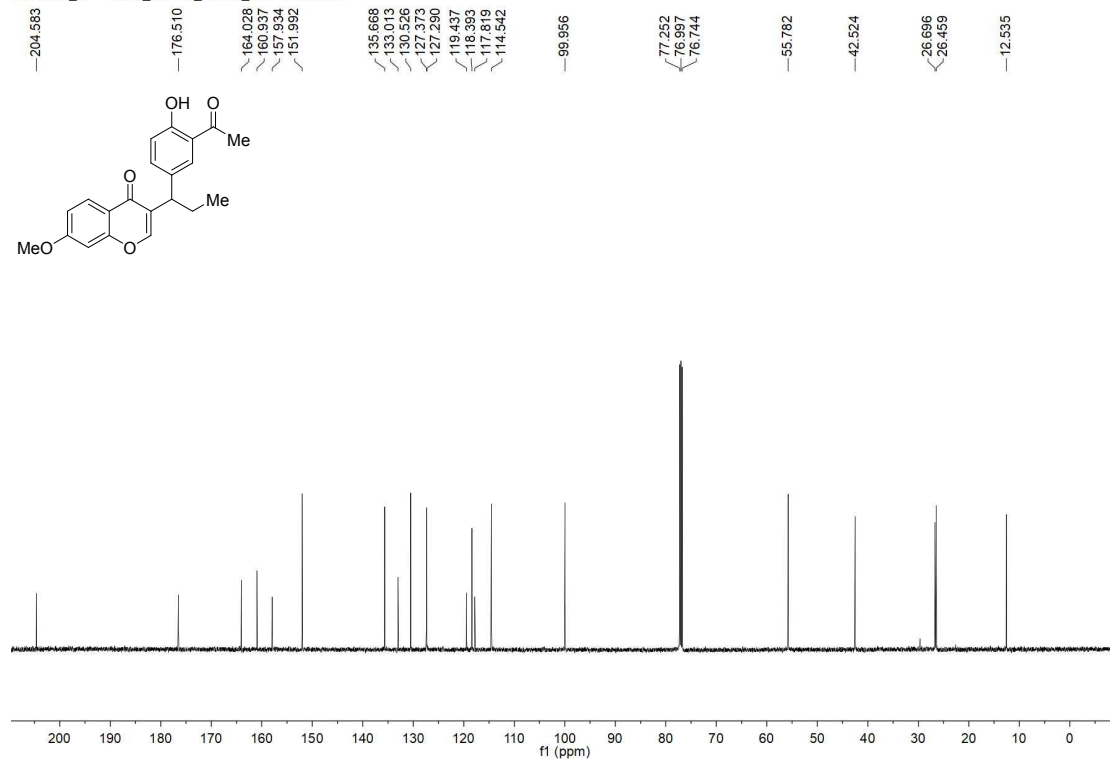
¹H NMR

PROTON_01 — L525_A13-17_CDCl3_500 2018/06/06 —



¹³C NMR

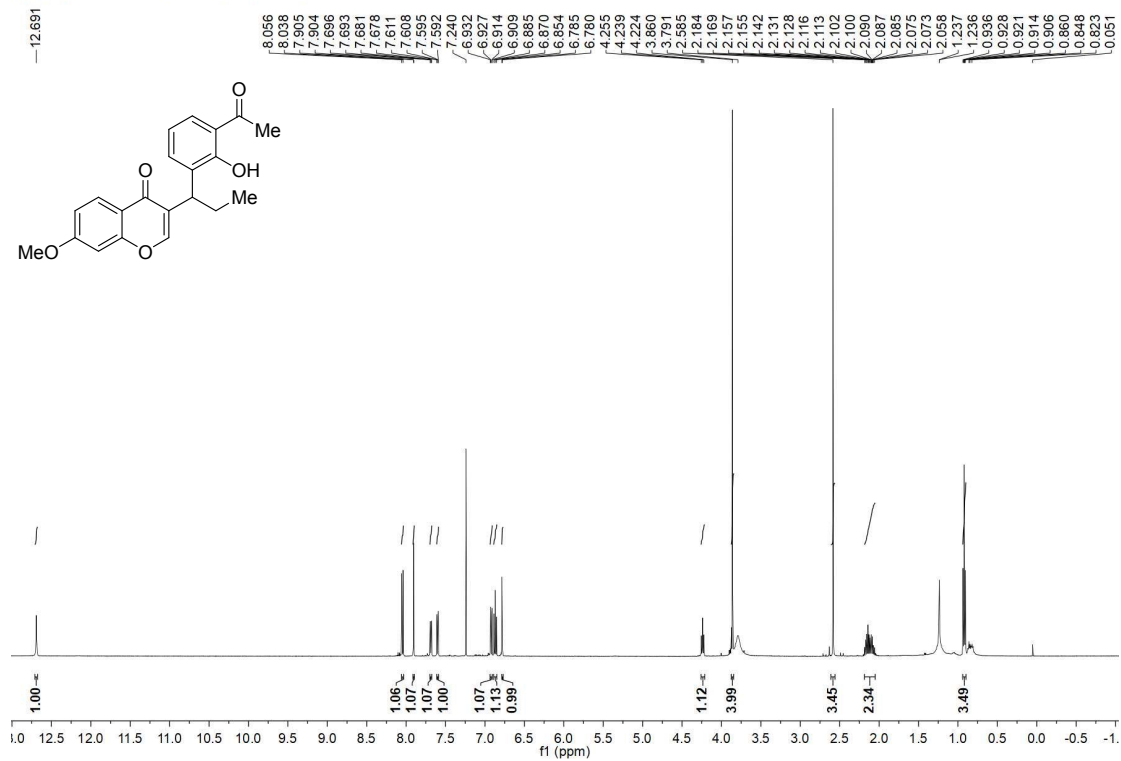
CARBON_01 — L525_A13-17_CDCl3_500 2018/06/06 —



3-(1-(3-Acetyl-2-hydroxyphenyl)propyl)-7-methoxy-4H-chromen-4-one (21')

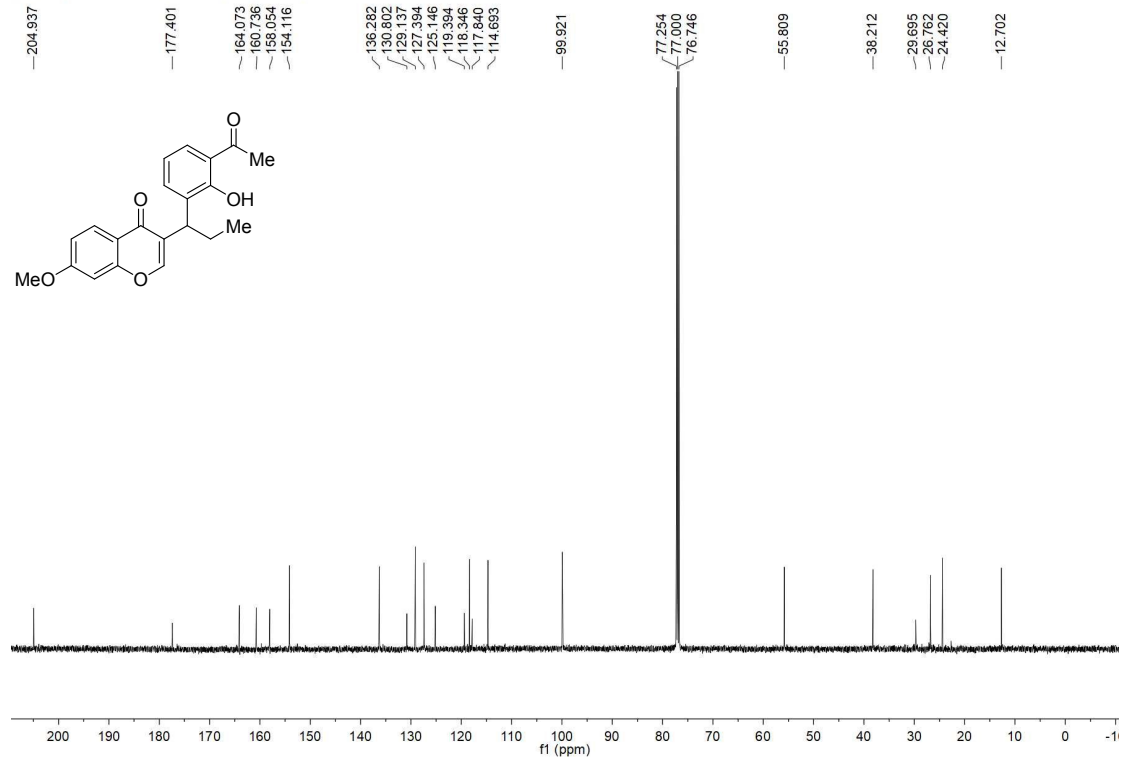
¹H NMR

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¹³C NMR

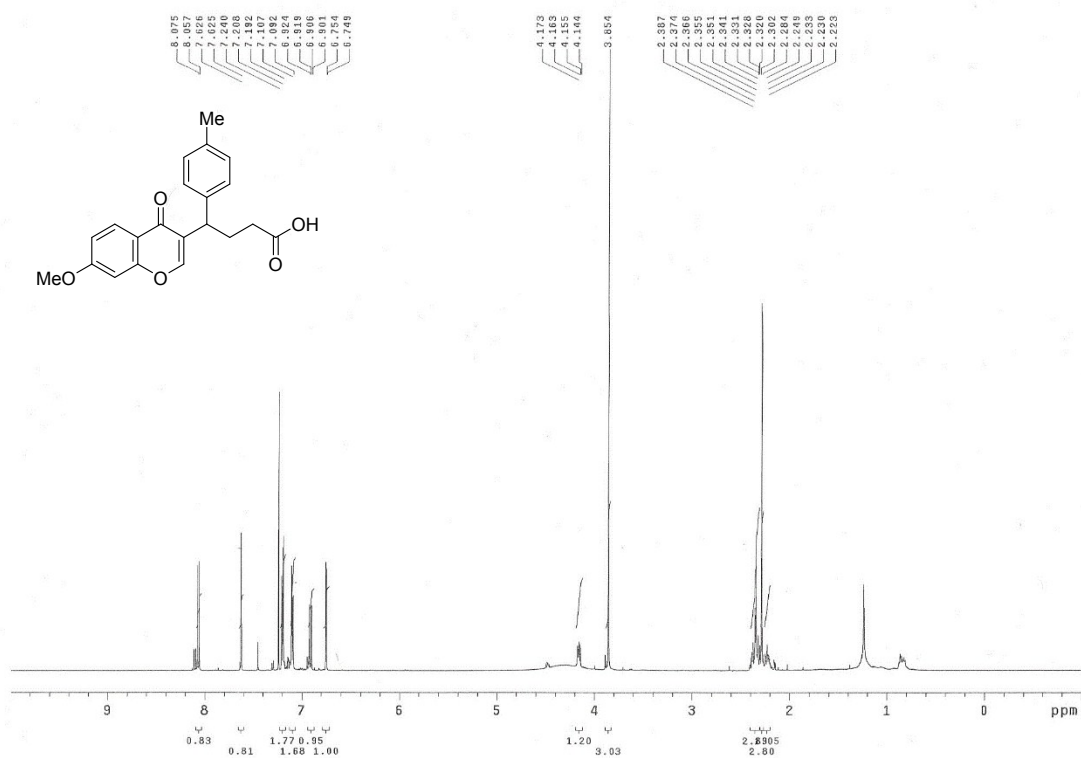
CARBON_01 — L525_A8-10_CDCl3_500 2018/06/06 —



4-(7-Methoxy-4-oxo-4H-chromen-3-yl)-4-(p-tolyl)butanoic acid (2m)

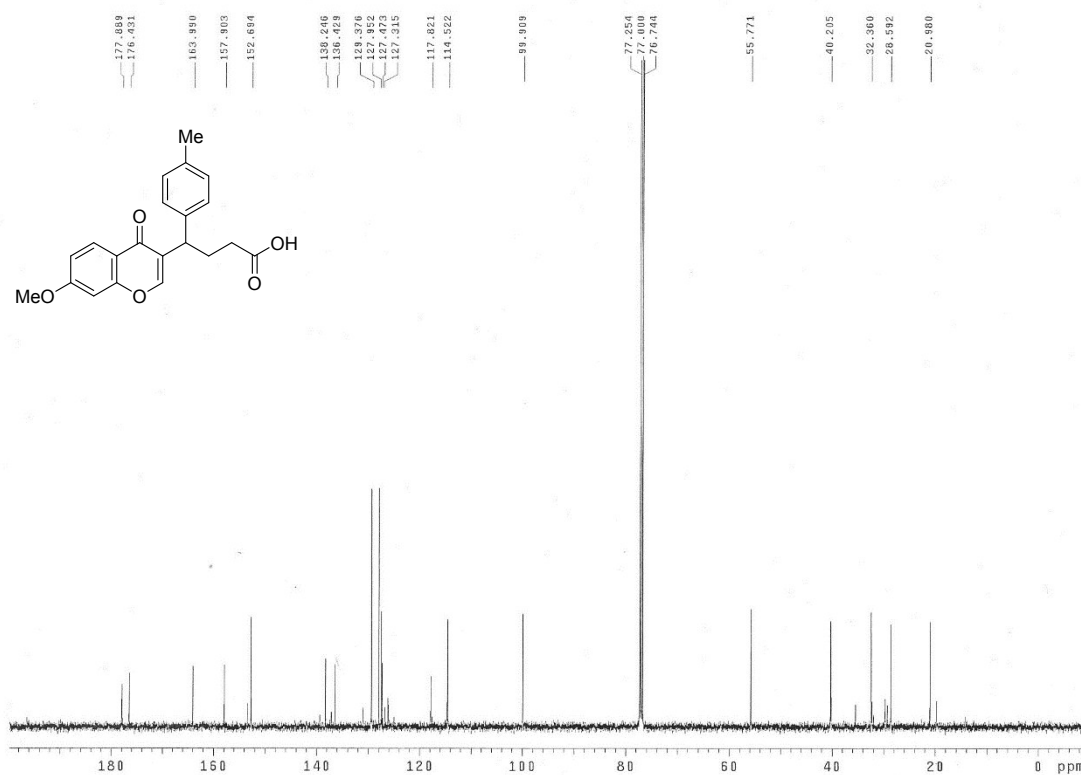
¹H NMR

L953_A23_CDCl3_500 2018/07/03



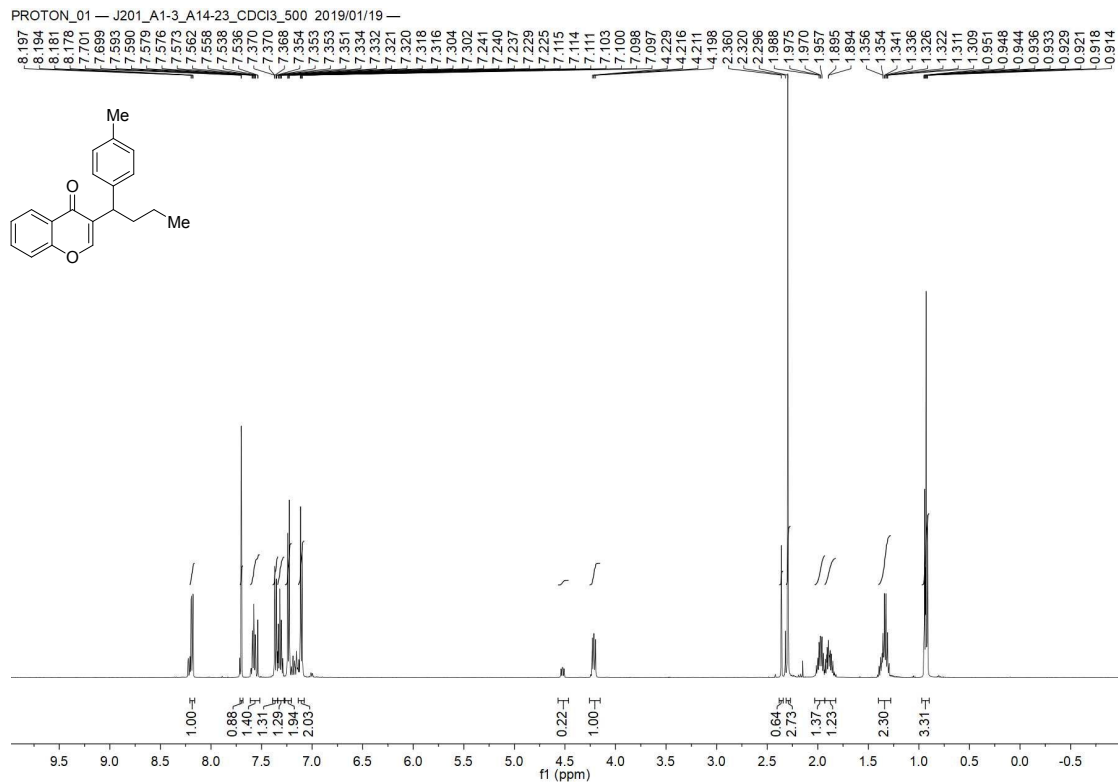
¹³C NMR

L953_A23_CDCl3_500 2018/07/03

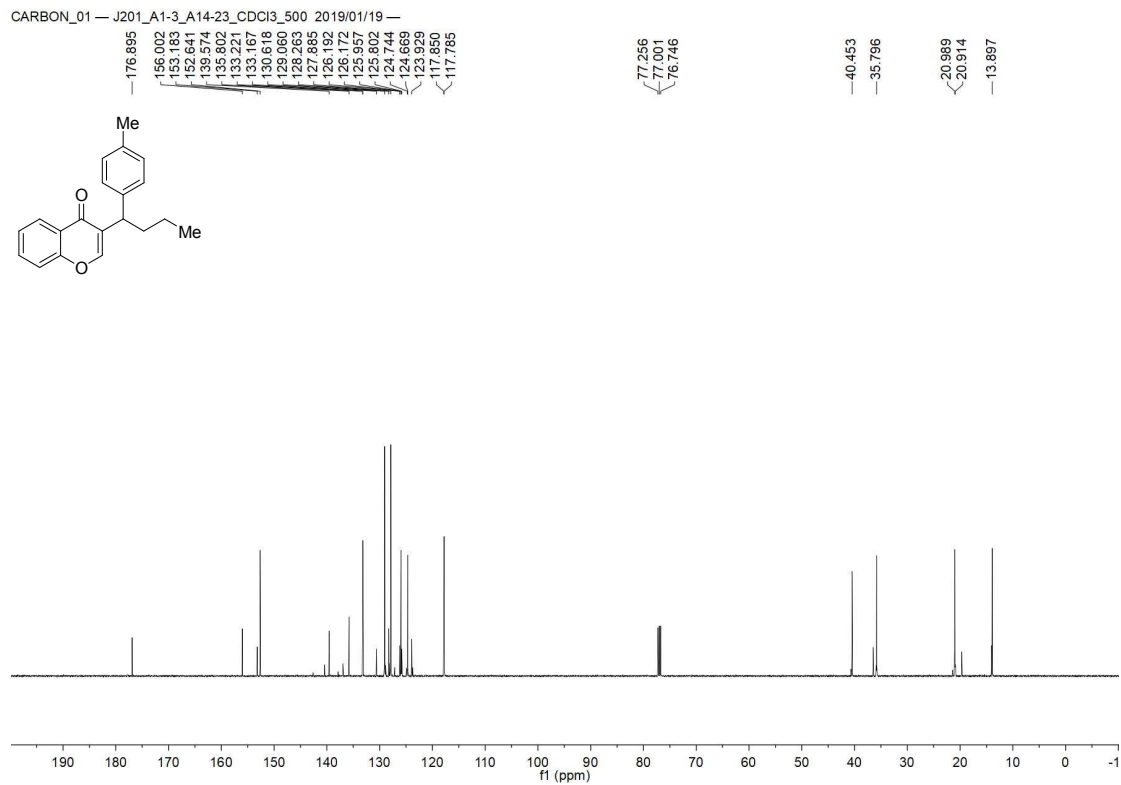


3-(1-(p-Tolyl)butyl)-4H-chromen-4-one (4a)

¹H NMR



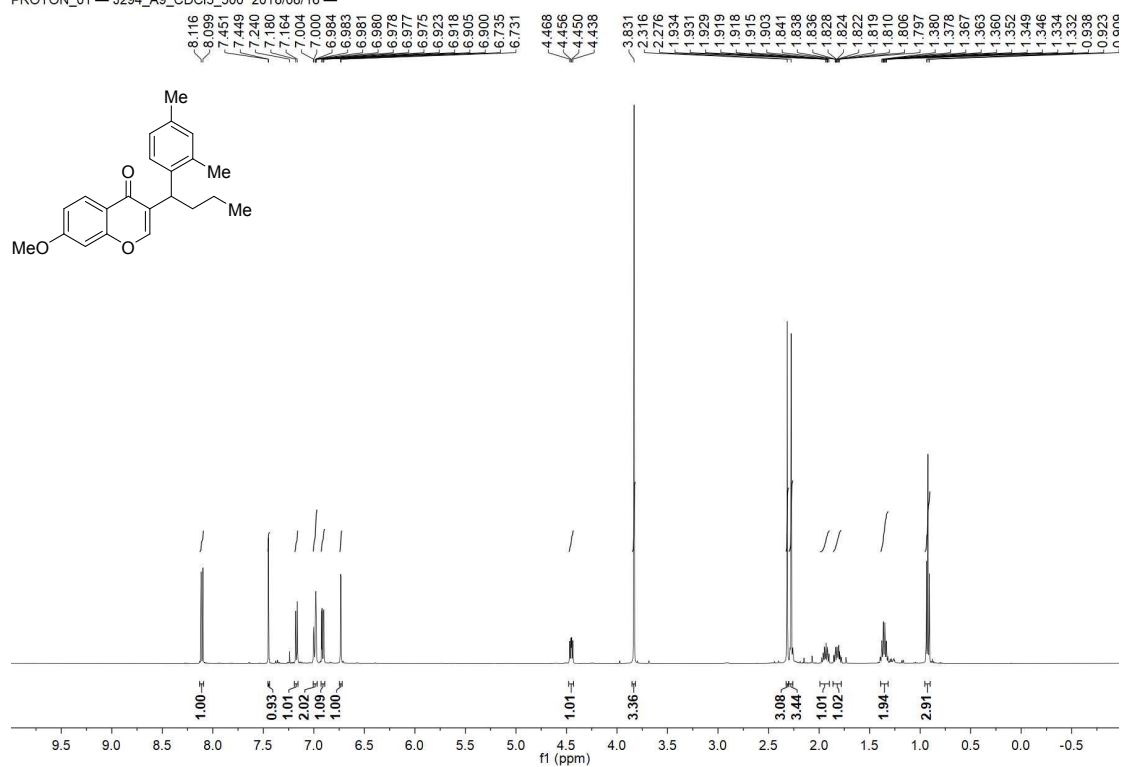
¹³C NMR



7-Methoxy-3-(1-(2,4-dimethylphenyl)butyl)-4H-chromen-4-one (**4b**)

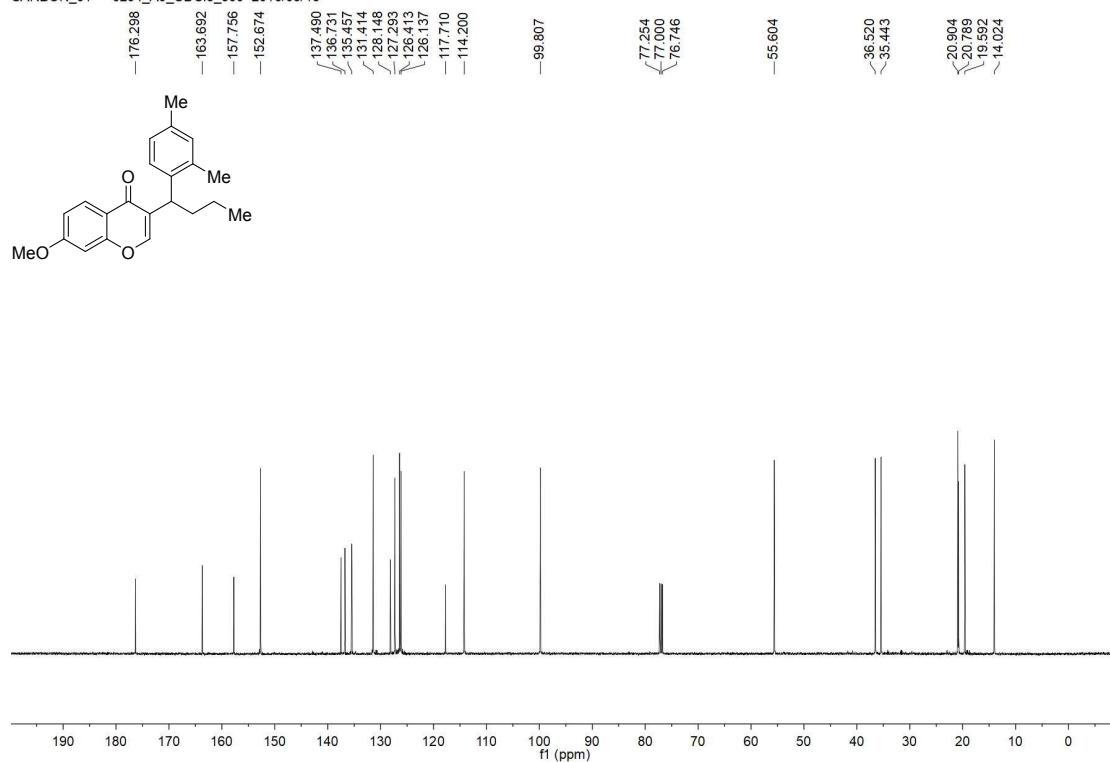
¹H NMR

PROTON_01 — J294_A9_CDCl3_500 2018/08/16 —



¹³C NMR

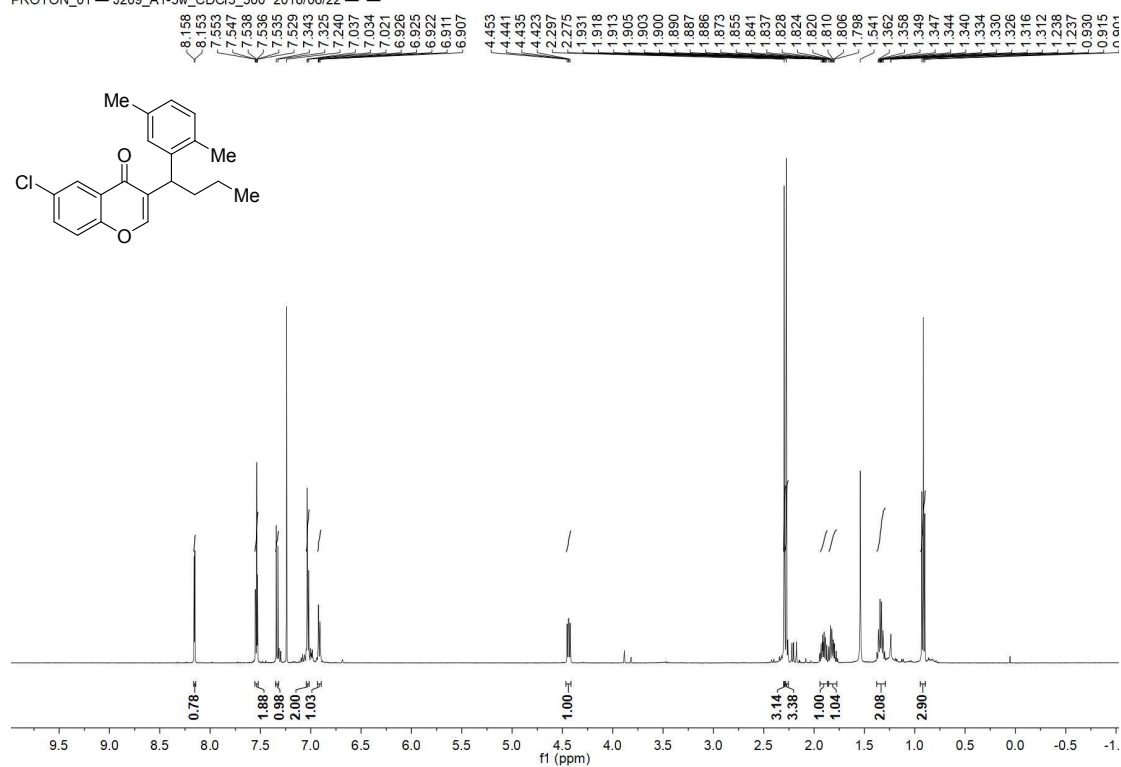
CARBON_01 — J294_A9_CDCl3_500 2018/08/16 —



6-Chloro-3-(1-(2,5-dimethylphenyl)butyl)-4H-chromen-4-one (4c)

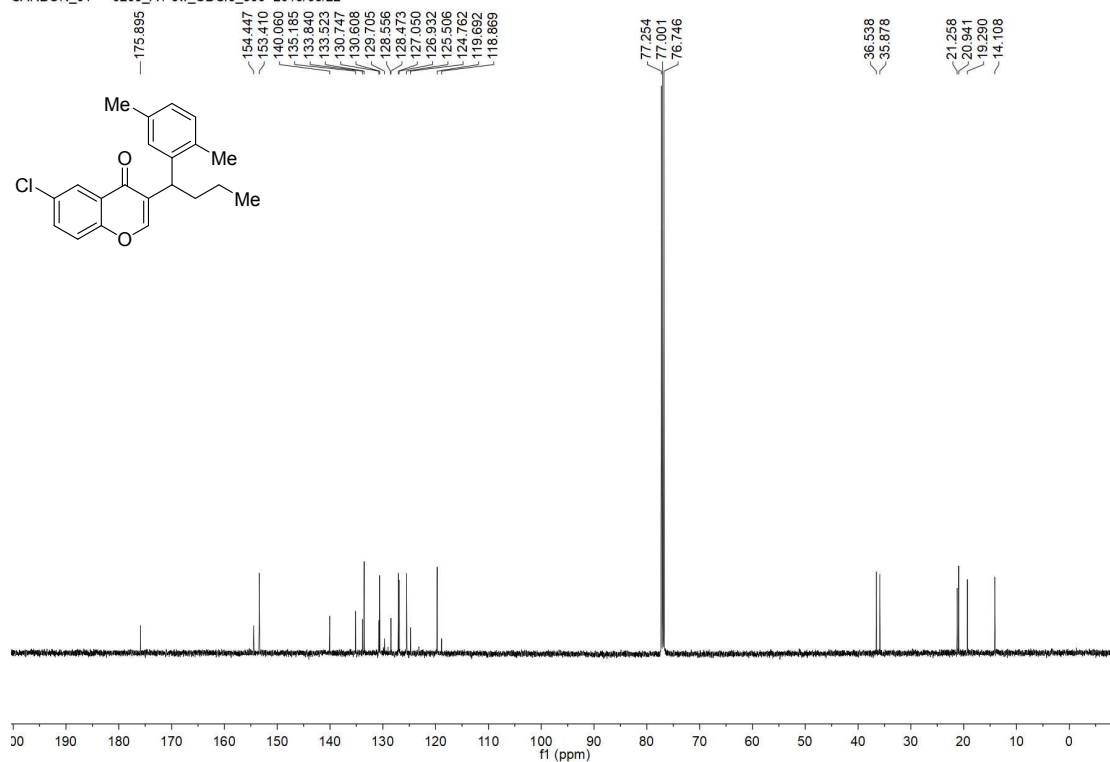
¹H NMR

PROTON_01 — J209_A1-3w_CDCl3_500 2018/06/22 —



¹³C NMR

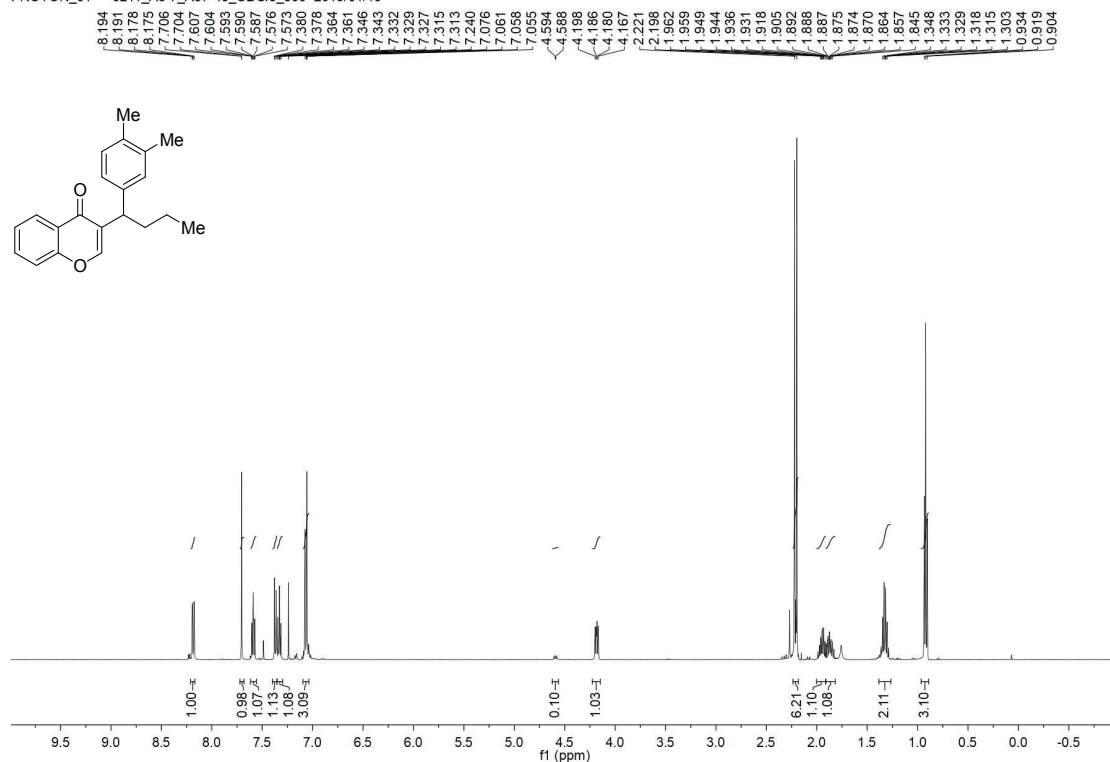
CARBON_01 — J209_A1-3w_CDCl3_500 2018/06/22 —



3-(1-(3,4-Dimethylphenyl)butyl)-4H-chromen-4-one (4d)

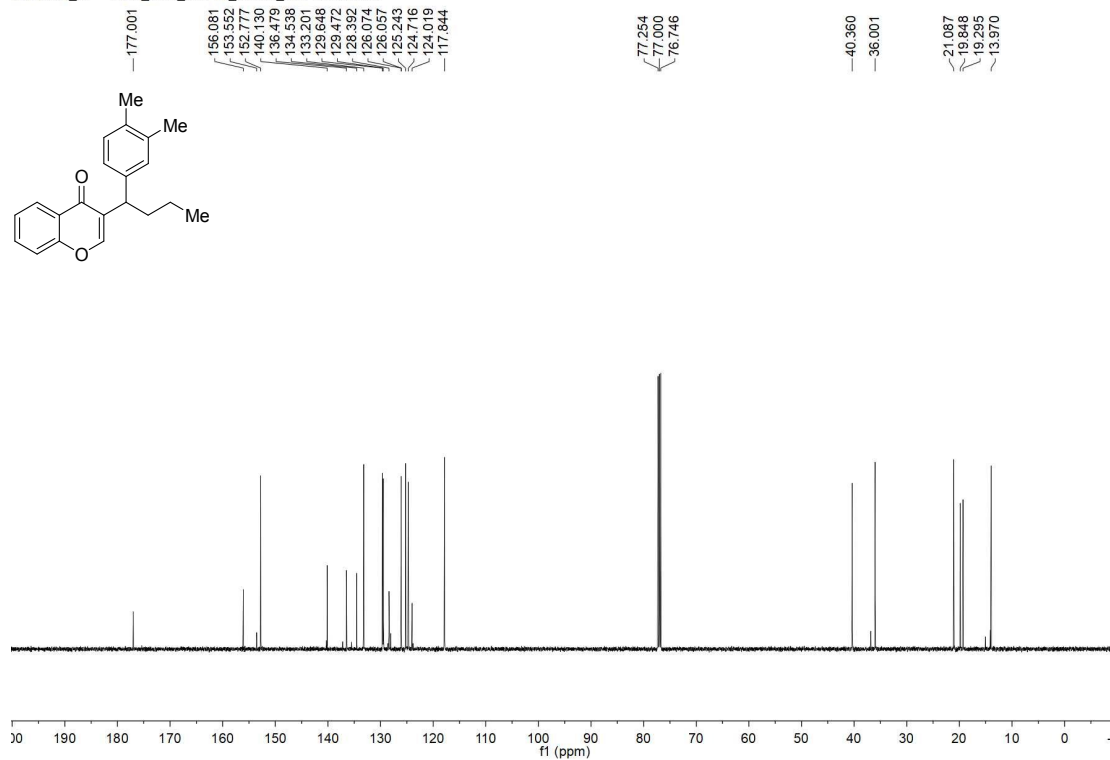
¹H NMR

PROTON_01 — J211_A6-7_A37-43_CDCl3_500 2019/01/19 —



¹³C NMR

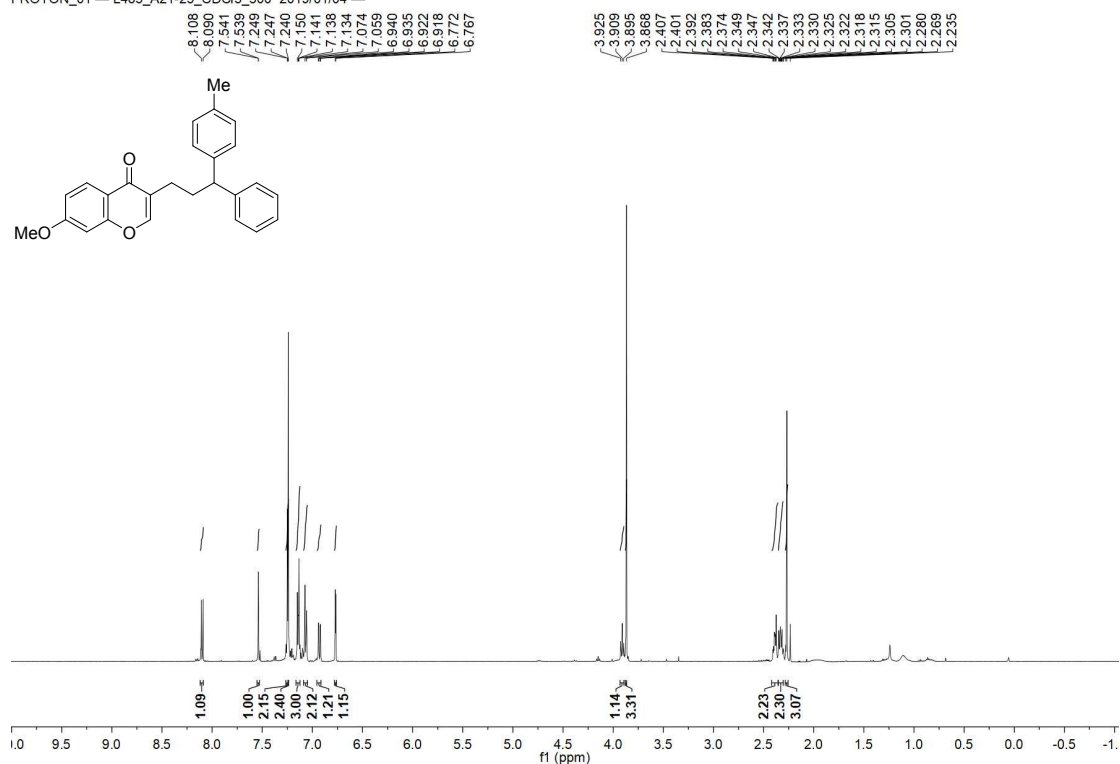
CARBON_01 — J211_A6-7_A37-43_CDCl3_500 2019/01/19 —



7-Methoxy-3-(3-phenyl-1-(p-tolyl)propyl)-4H-chromen-4-one (6a)

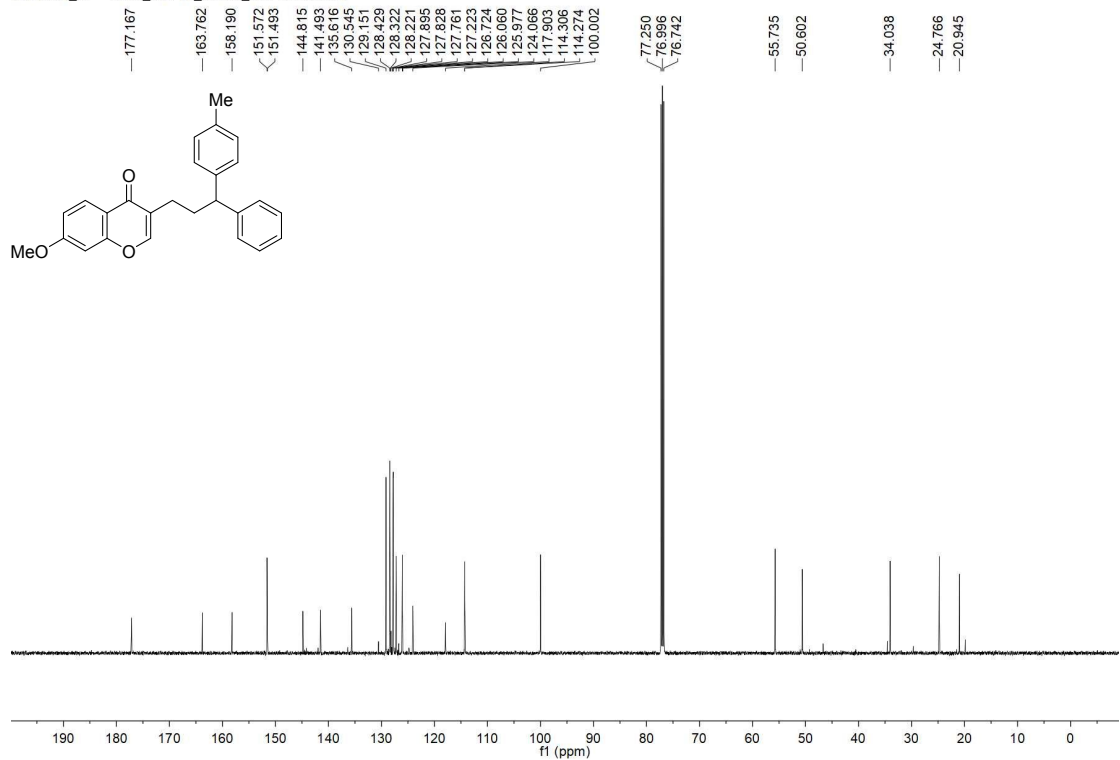
¹H NMR

PROTON_01 — L483_A21-25_CDCl3_500 2019/01/04 —



¹³C NMR

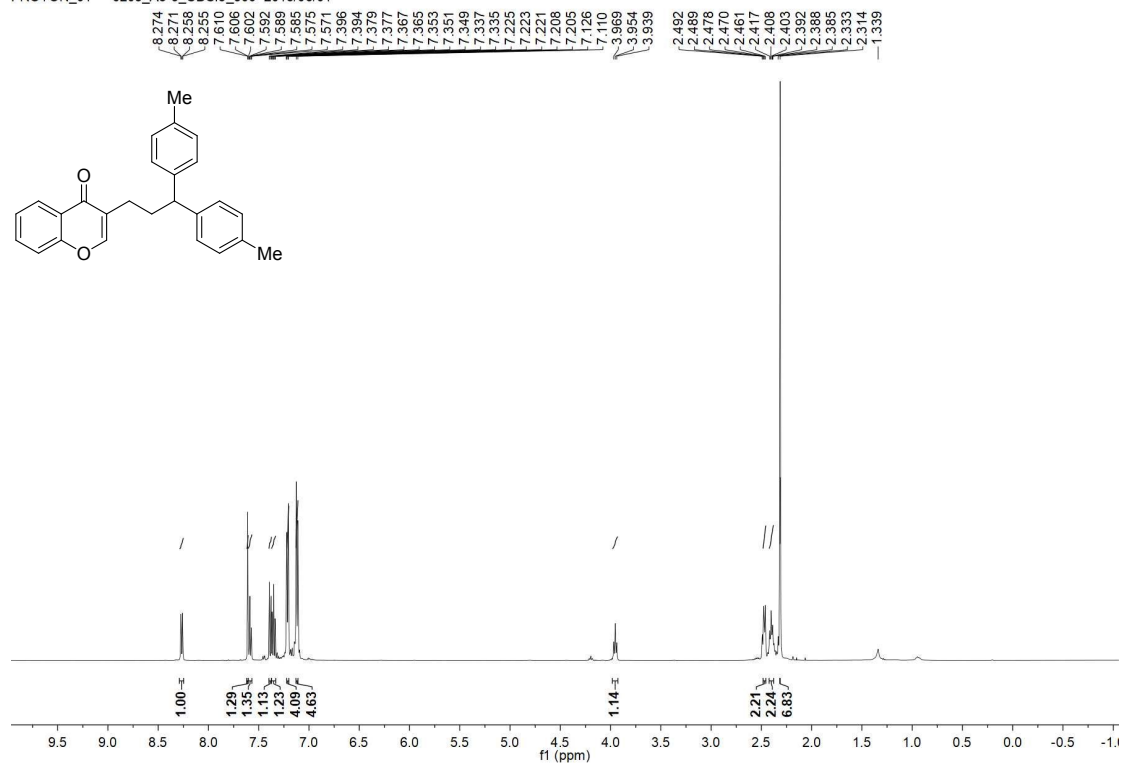
CARBON_01 — L483_A21-25_CDCl3_500 2019/01/04 —



3-(1,3-Di-p-tolylpropyl)-4H-chromen-4-one (6b)

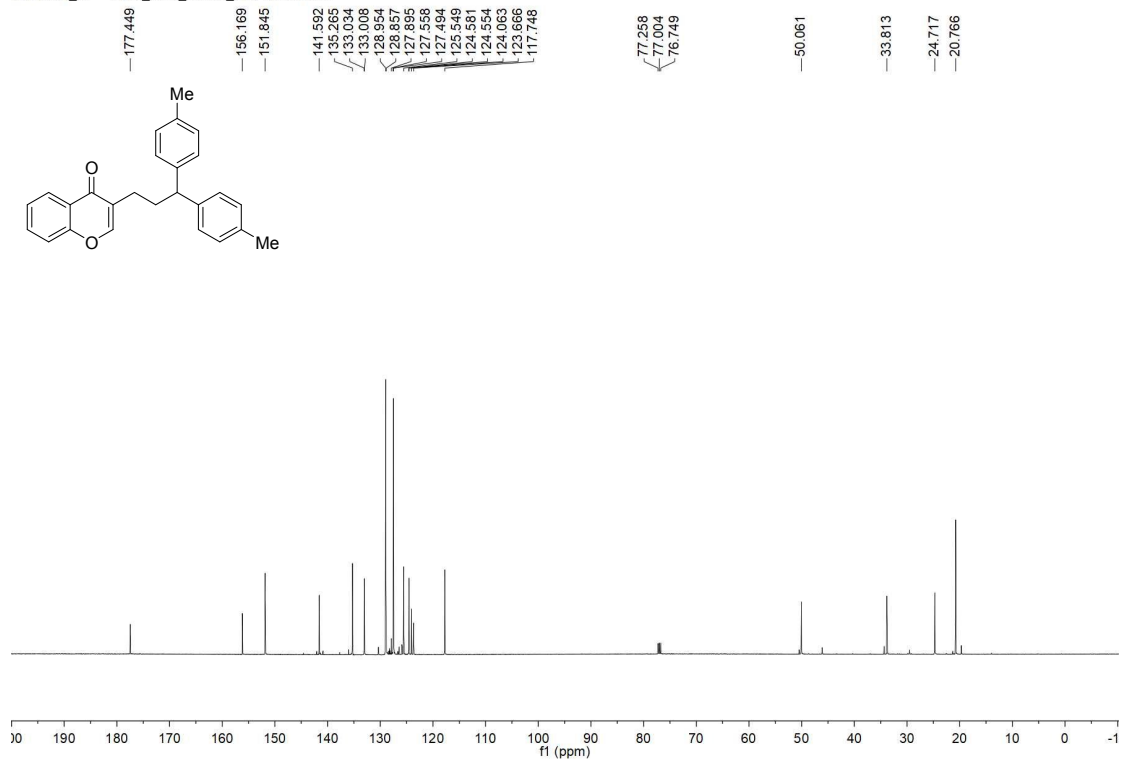
¹H NMR

PROTON_01 — J205_A3-9_CDCl3_500 2018/06/01 —



¹³C NMR

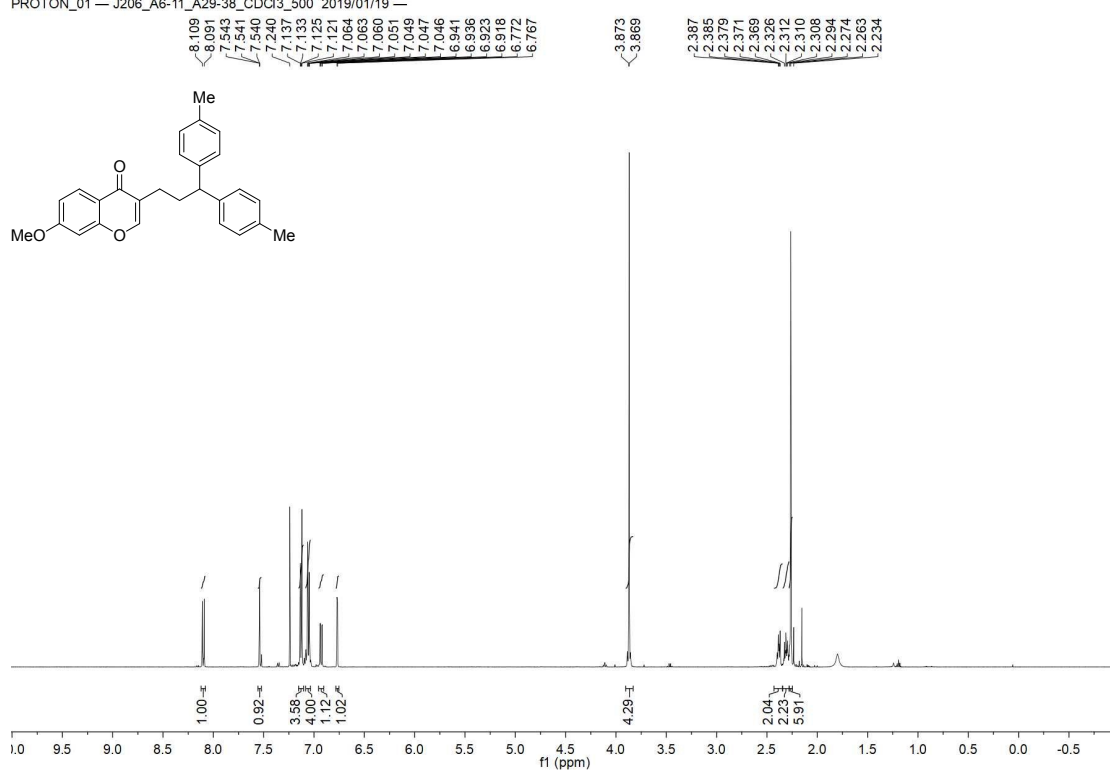
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3-(1,3-Di-p-tolylpropyl)-7-methoxy-4H-chromen-4-one (6c)

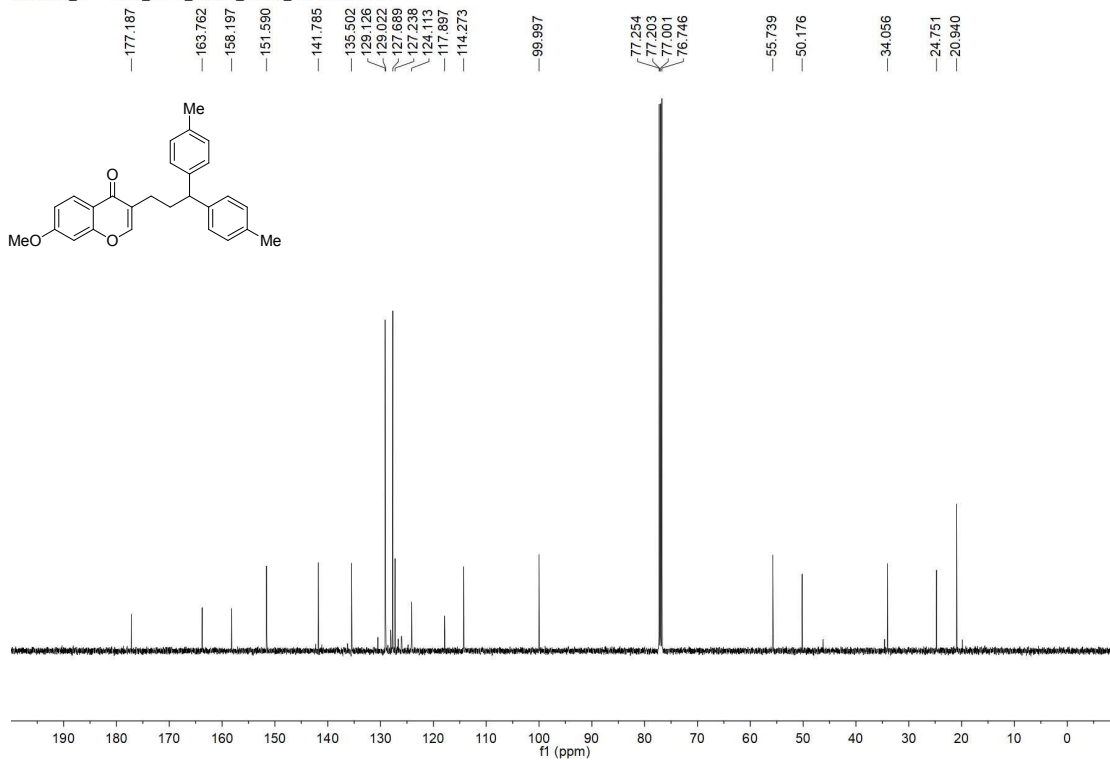
¹H NMR

PROTON_01 — J206_A6-11_A29-38_CDCl3_500 2019/01/19 —



¹³C NMR

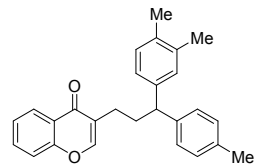
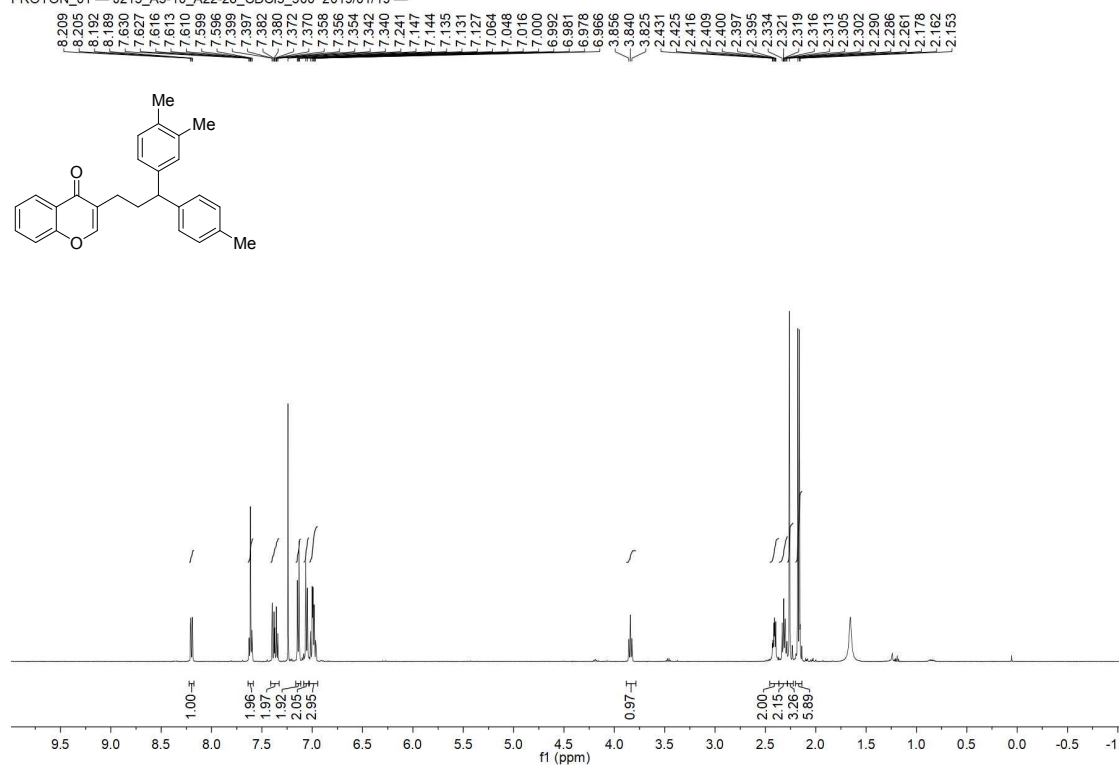
CARBON_01 — J206_A6-11_A29-38_CDCl3_500 2019/01/19 —



3-(1-(3,4-Dimethylphenyl)-3-(p-tolyl)propyl)-4H-chromen-4-one (6d)

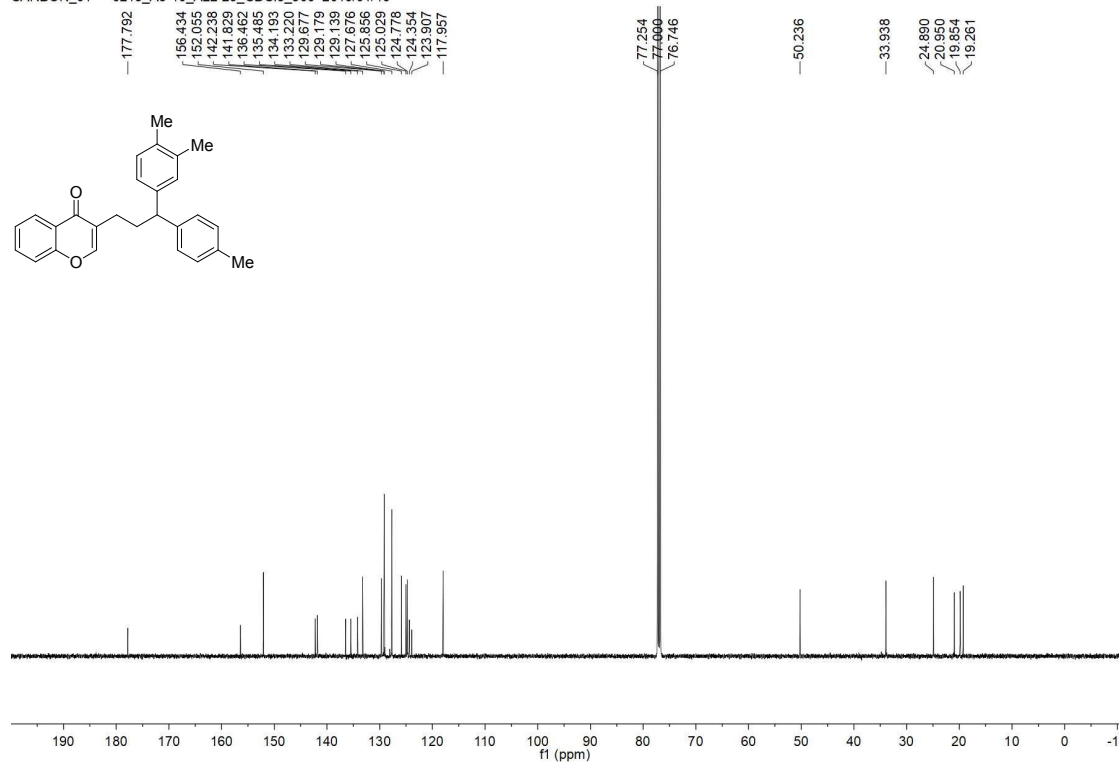
¹H NMR

PROTON_01 — J215_A3-10_A22-28_CDCl3_500_2019/01/19 —



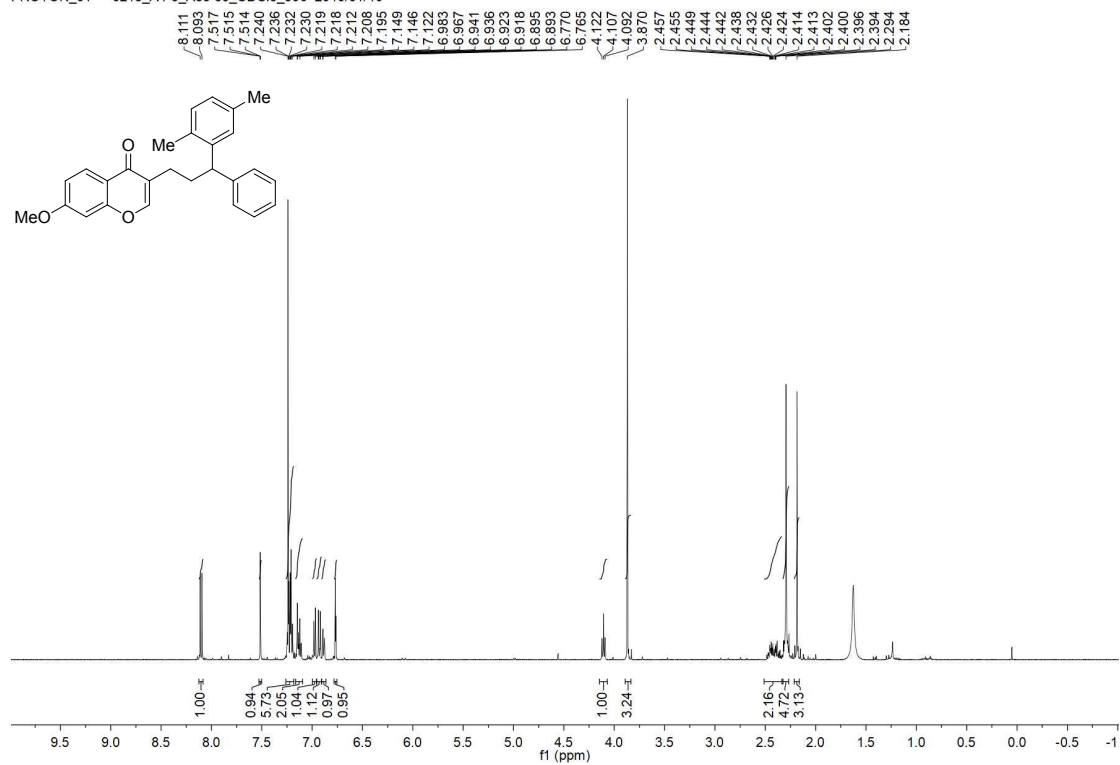
¹³C NMR

CARBON_01 — J215_A3-10_A22-28_CDCl3_500_2019/01/19 —



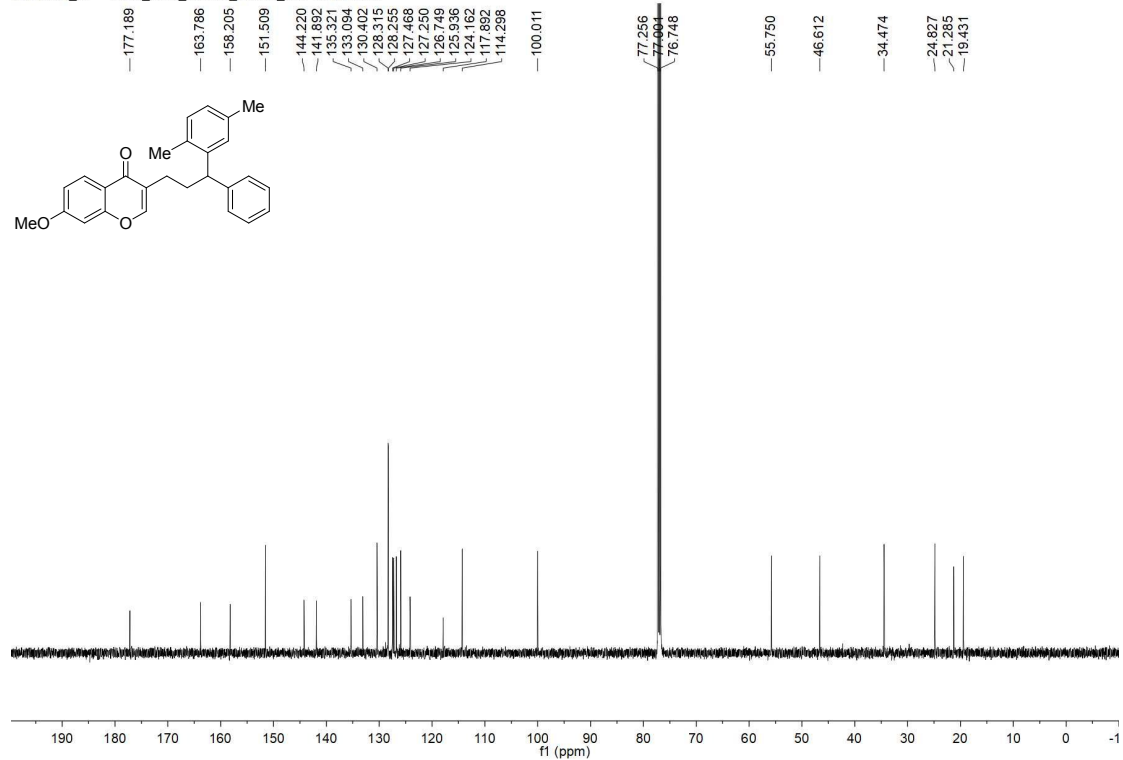
3-(1-(2,5-Dimethylphenyl)-3-phenylpropyl)-7-methoxy-4H-chromen-4-one (6e)
¹H NMR

PROTON_01 — J213_A4-9_A35-39_CDCl3_500 2019/01/10 —



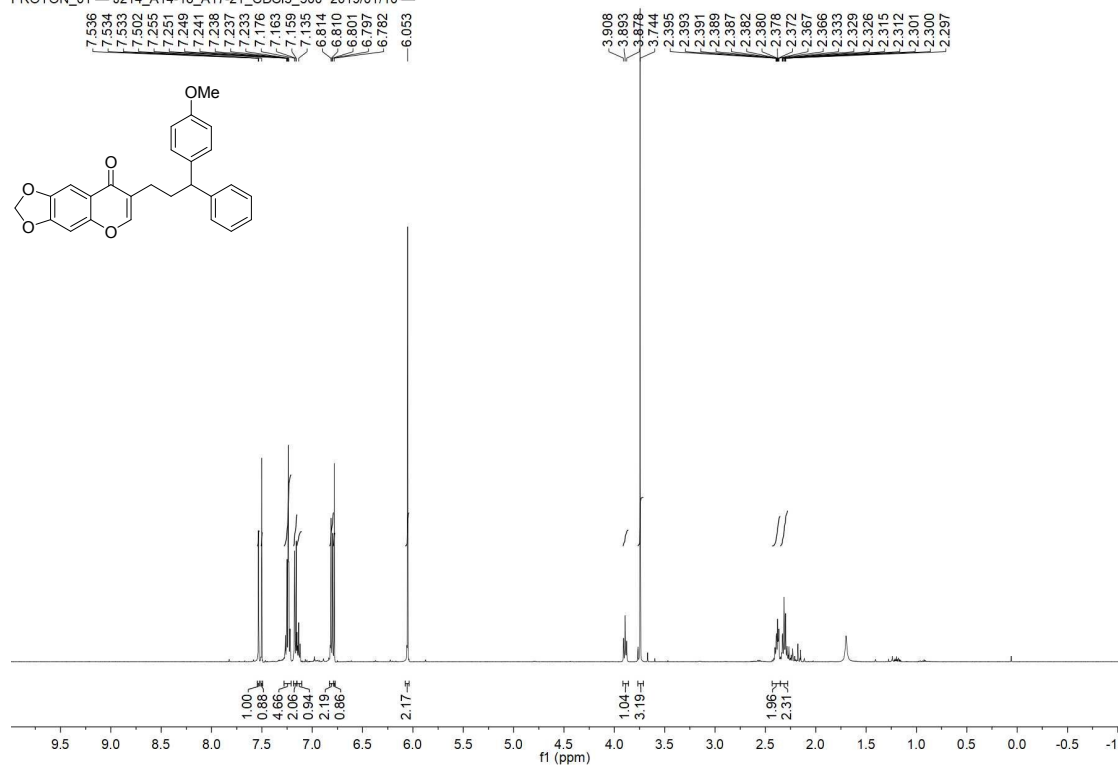
¹³C NMR

CARBON_01 — J213_A4-9_A35-39_CDCl3_500 2019/01/10 —



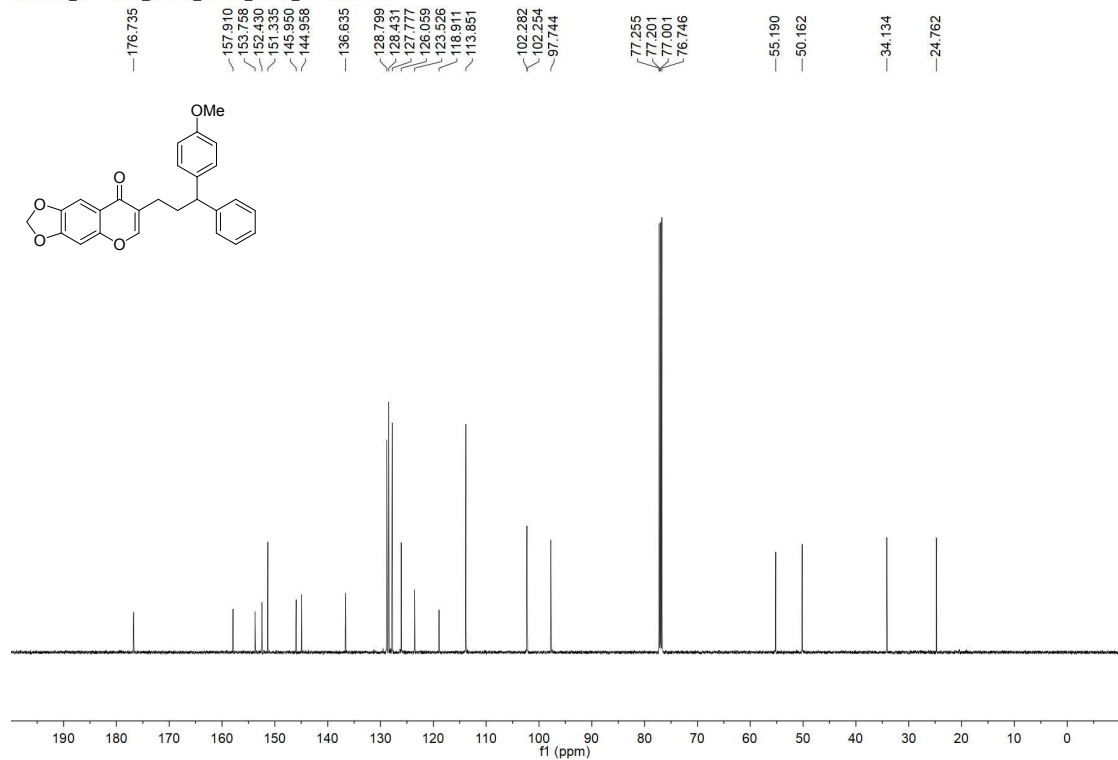
7-(1-(4-Methoxyphenyl)-3-phenylpropyl)-8H-[1,3]dioxolo[4,5-g]chromen-8-one (6f)
¹H NMR

PROTON_01 — J214_A14-18_A17-21_CDCl3_500 2019/01/10 —



¹³C NMR

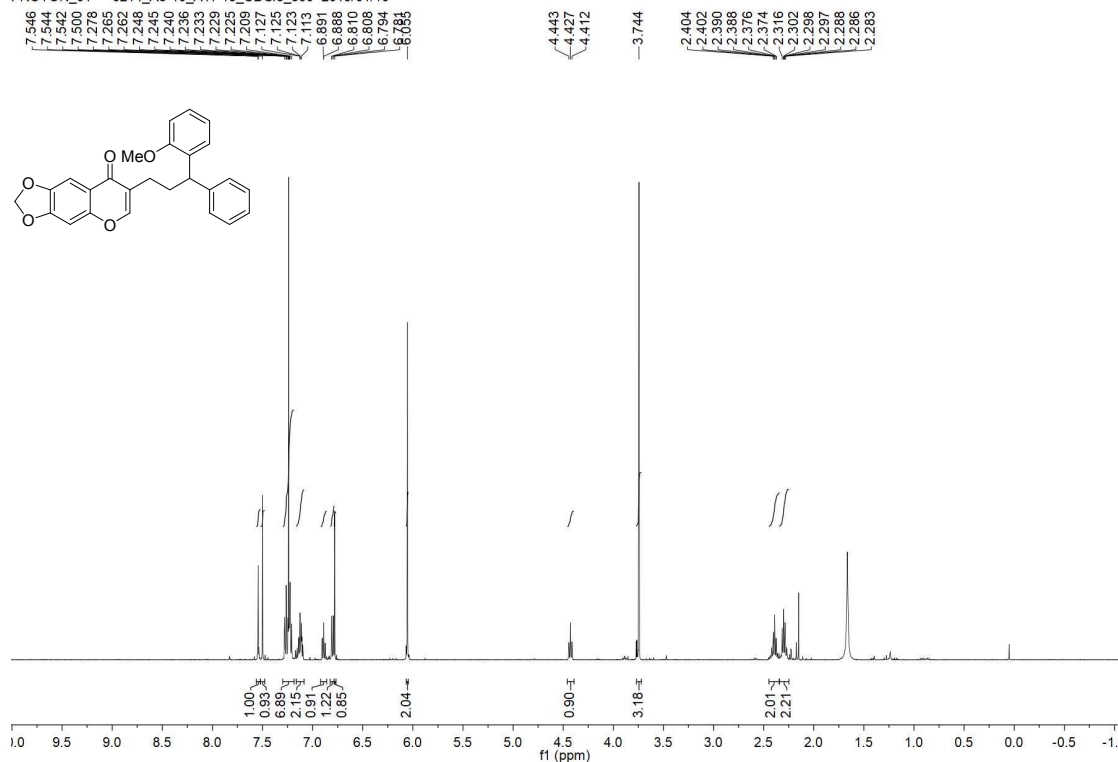
CARBON_01 — J214_A14-18_A17-21_CDCl3_500 2019/01/10 —



7-(1-(2-Methoxyphenyl)-3-phenylpropyl)-8H-[1,3]dioxolo[4,5-g]chromen-8-one (6f)

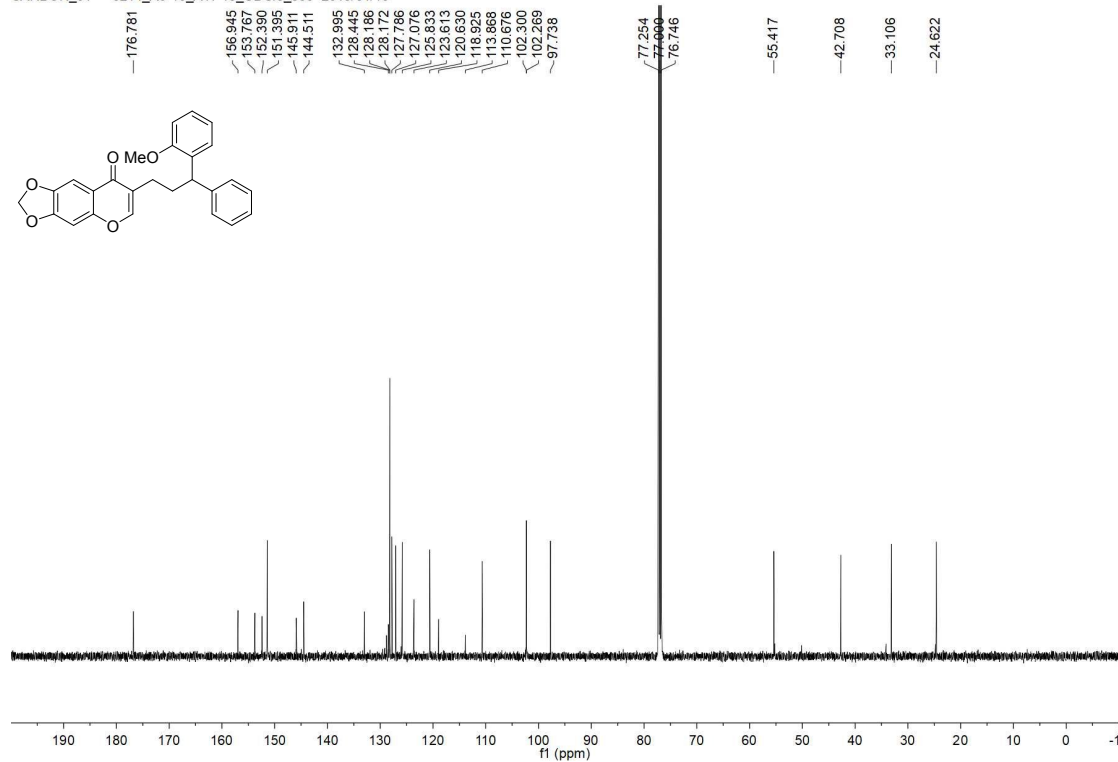
¹H NMR

PROTON_01 — J214_A8-10_A11-15_CDCl3_500 2019/01/10 —



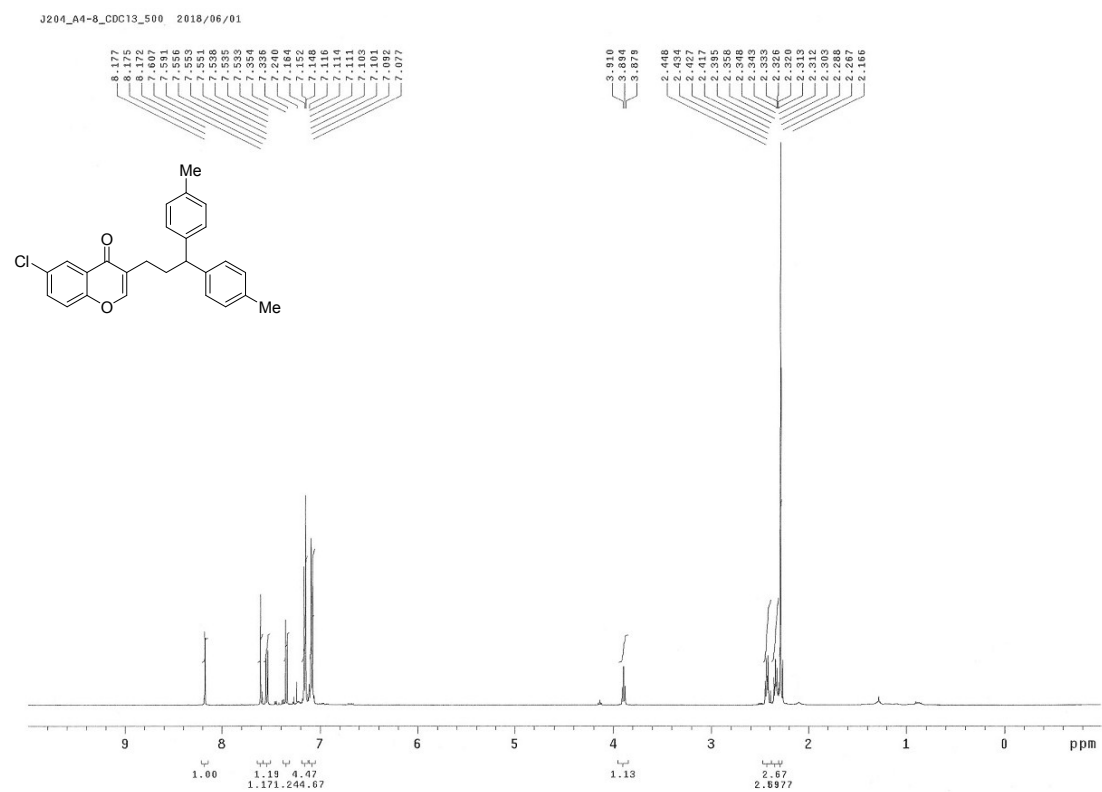
¹³C NMR

CARBON_01 — J214_A8-10_A11-15_CDCl3_500 2019/01/10 —

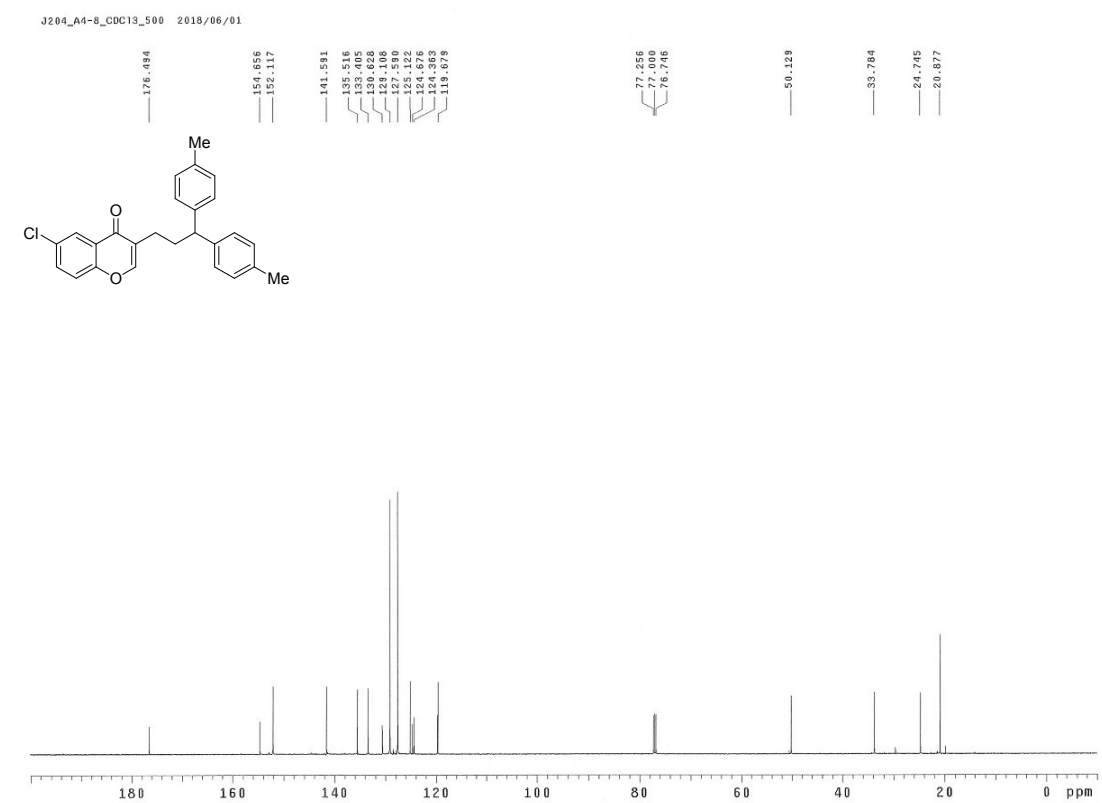


6-Chloro-3-(1,3-di-p-tolylpropyl)-4H-chromen-4-one (6g)

¹H NMR



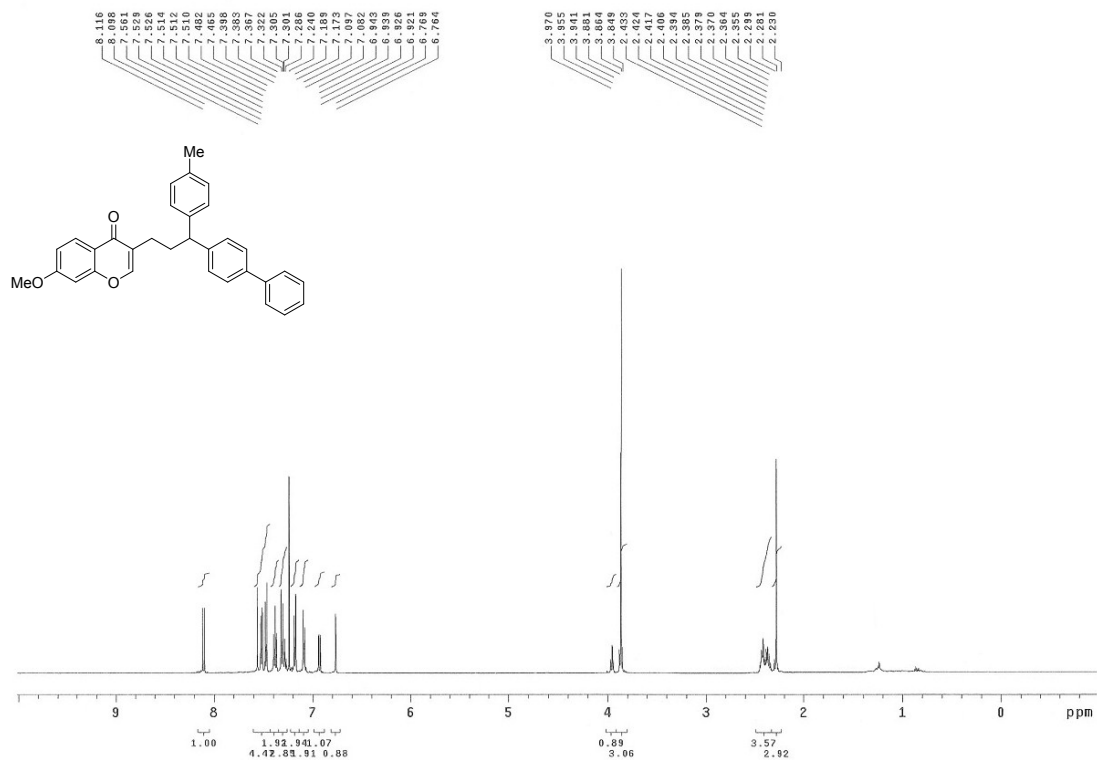
¹³C NMR



3-(1-([1,1'-Biphenyl]-4-yl)-3-(p-tolyl)propyl)-7-methoxy-4H-chromen-4-one (6h)

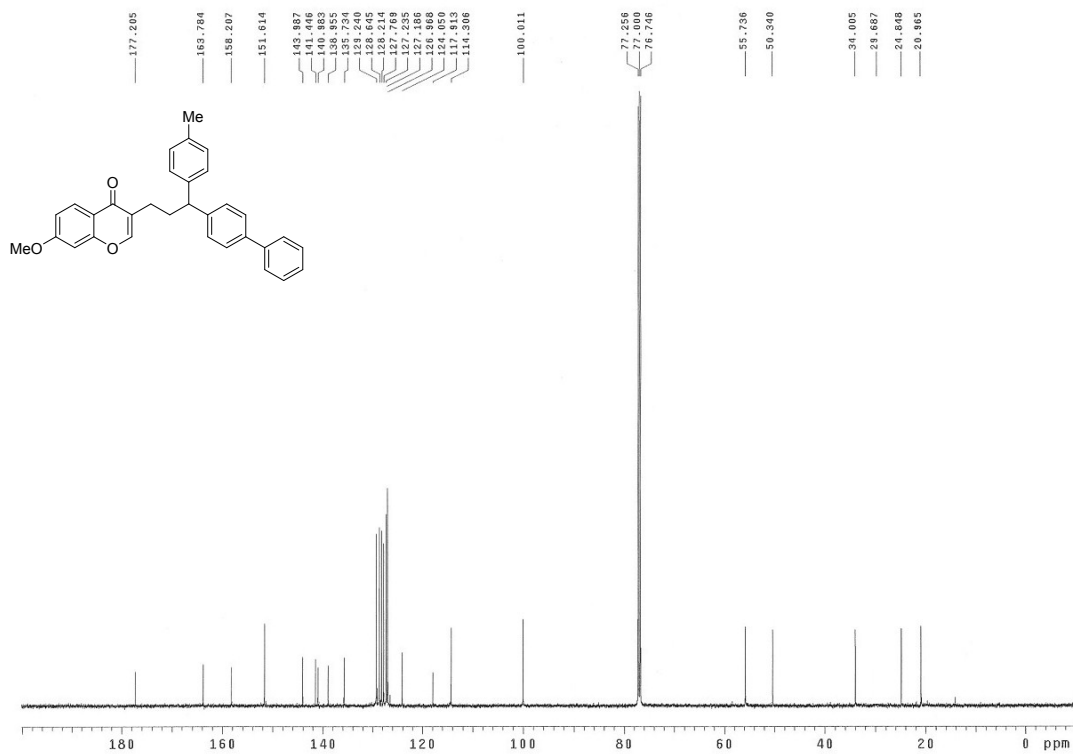
¹H NMR

J216_A7-14_CDCl3_500 2018/06/13



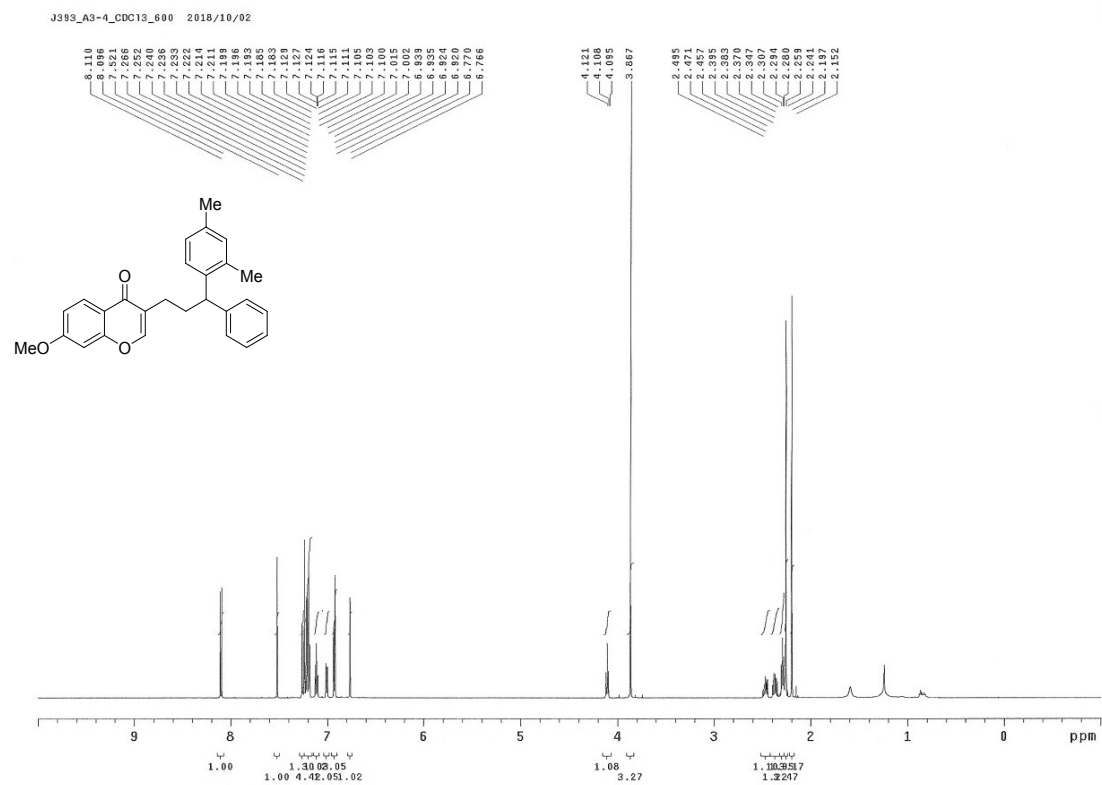
¹³C NMR

J216_A7-14_CDCl3_500 2018/06/13

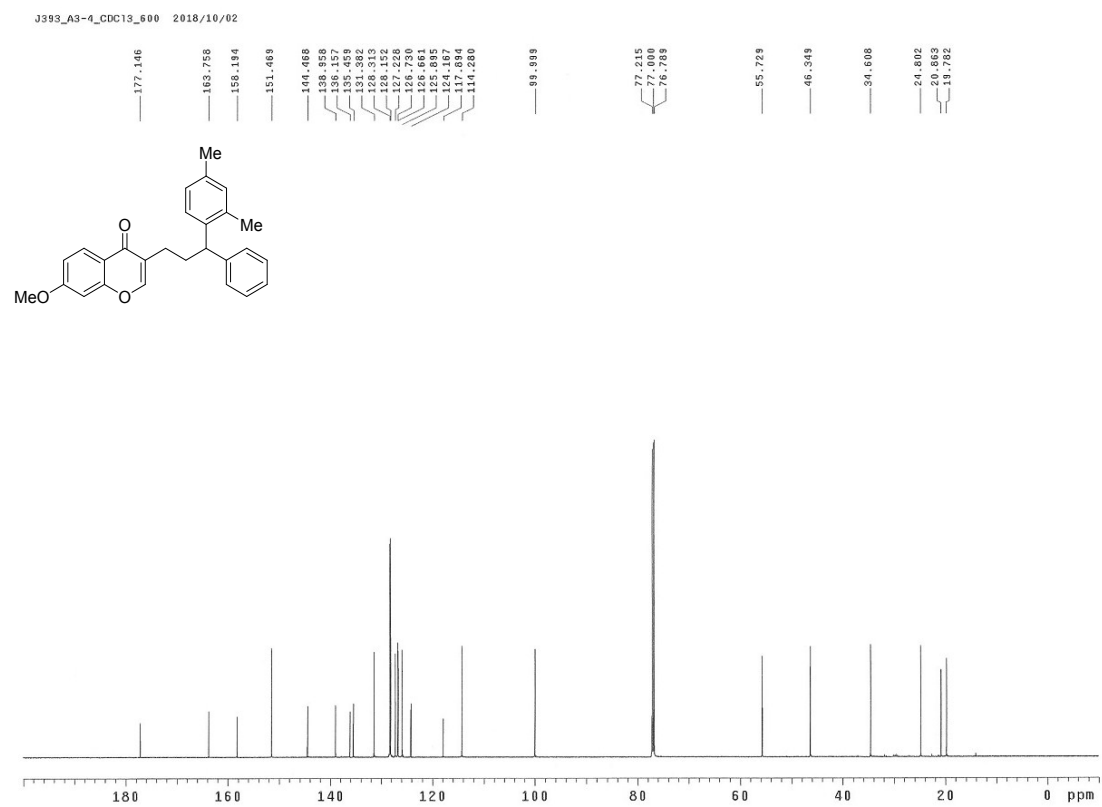


7-Methoxy-3-(3-(2,4-dimethylphenyl)-3-phenylpropyl)-4H-chromen-4-one (**6i**)

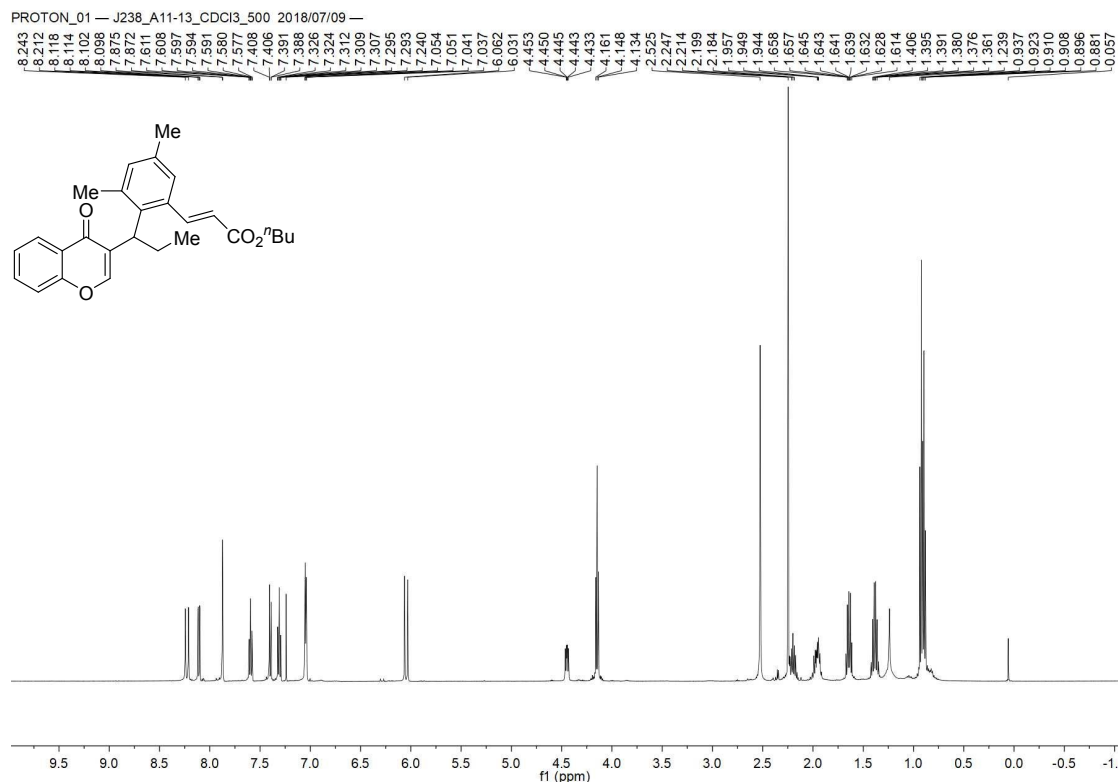
¹H NMR



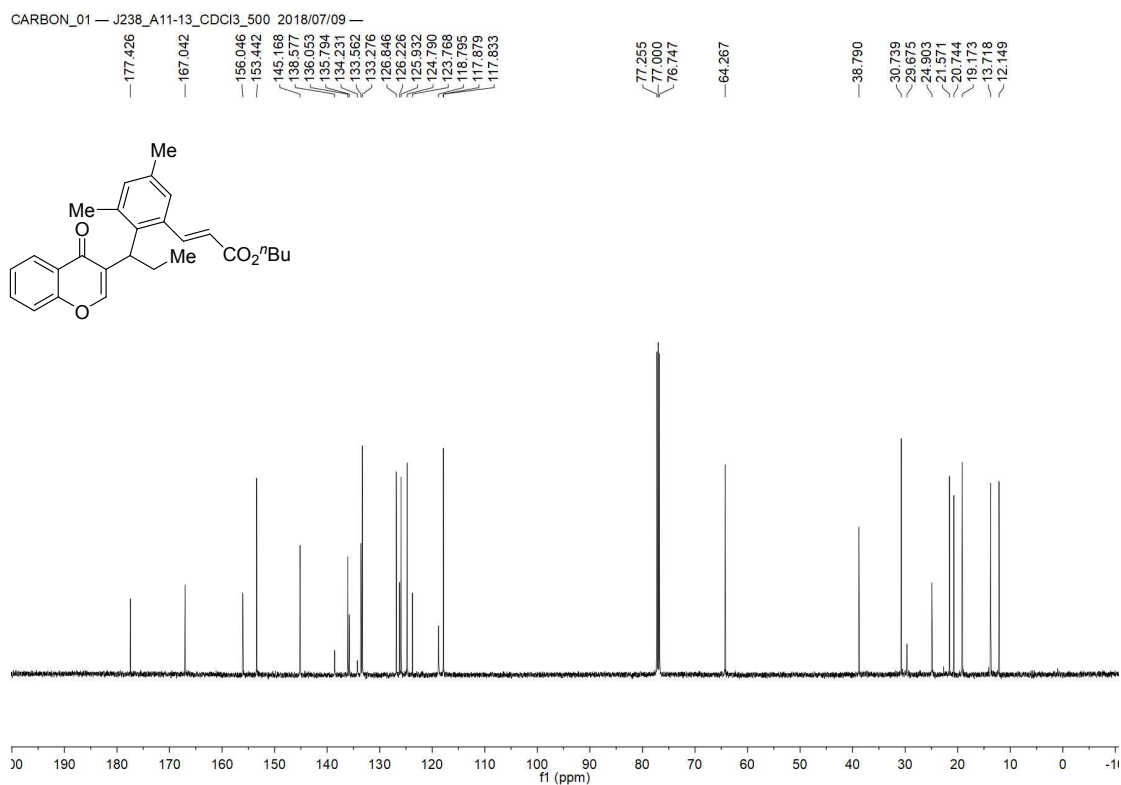
¹³C NMR



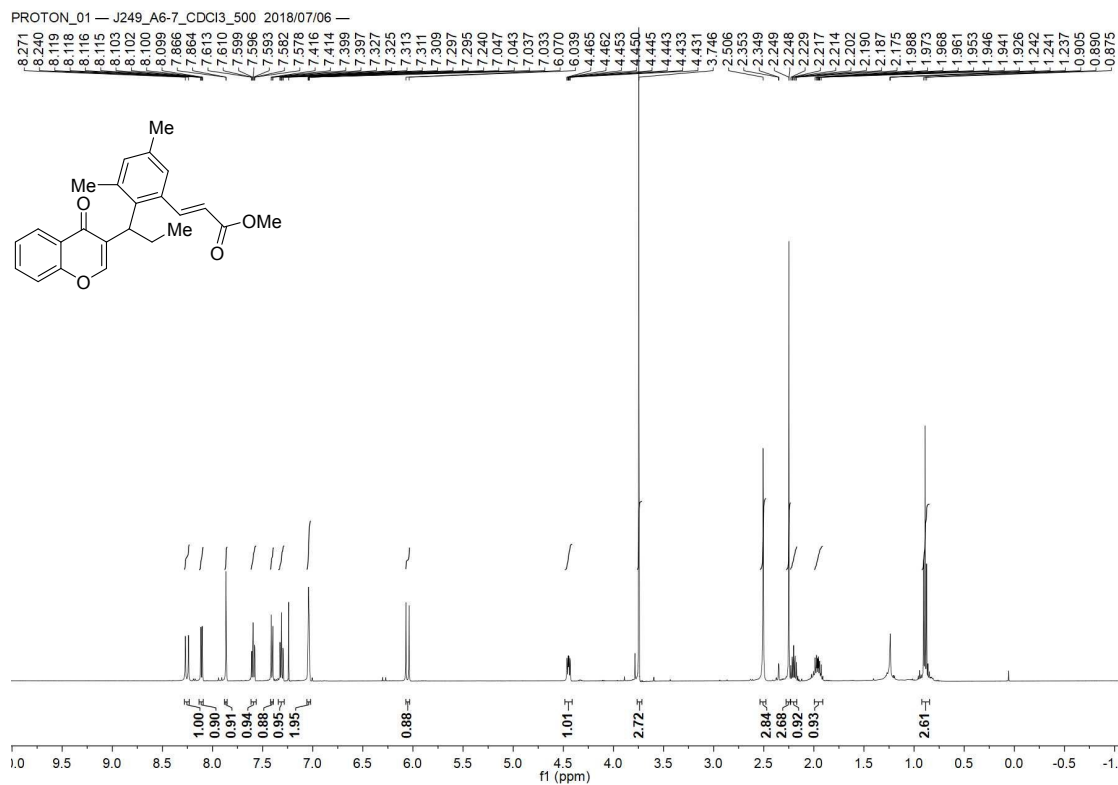
Butyl (E)-3-(3,5-dimethyl-2-(1-(4-oxo-4H-chromen-3-yl)propyl)phenyl)acrylate (7a)
¹H NMR



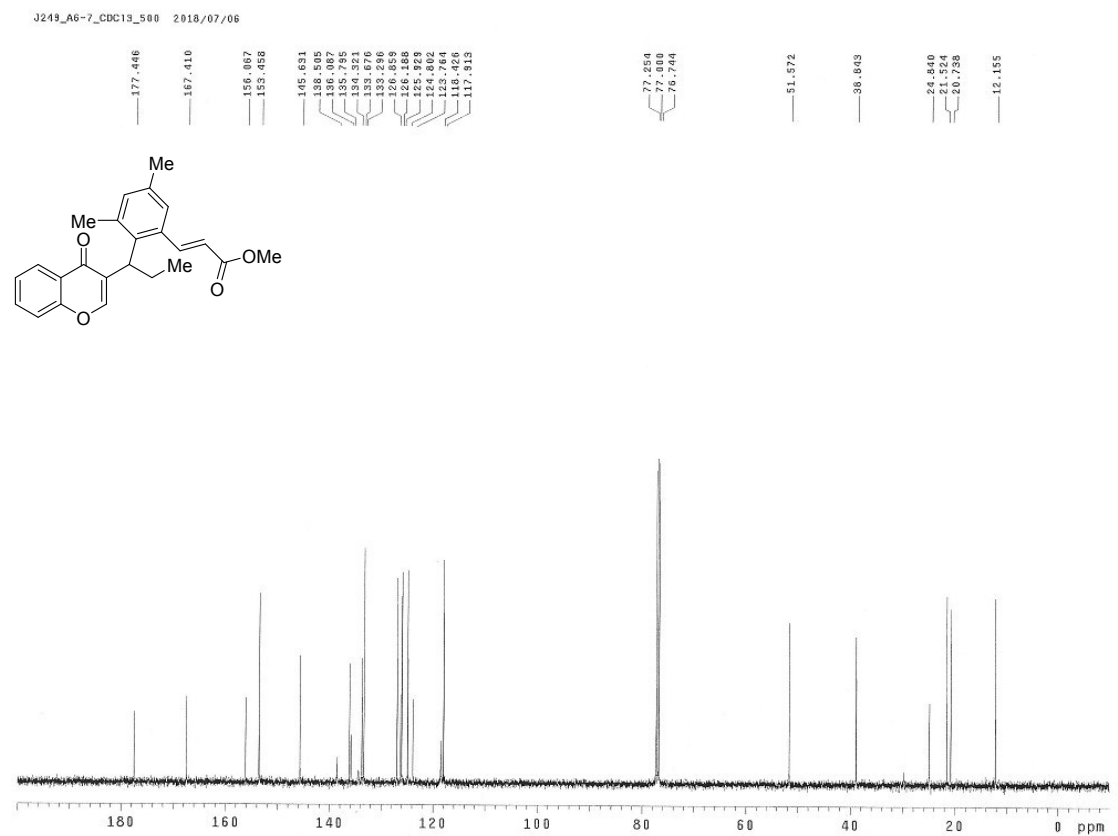
¹³C NMR



Methyl (E)-3-(3,5-dimethyl-2-(1-(4-oxo-4H-chromen-3-yl)propyl)phenyl)acrylate (7b)
¹H NMR

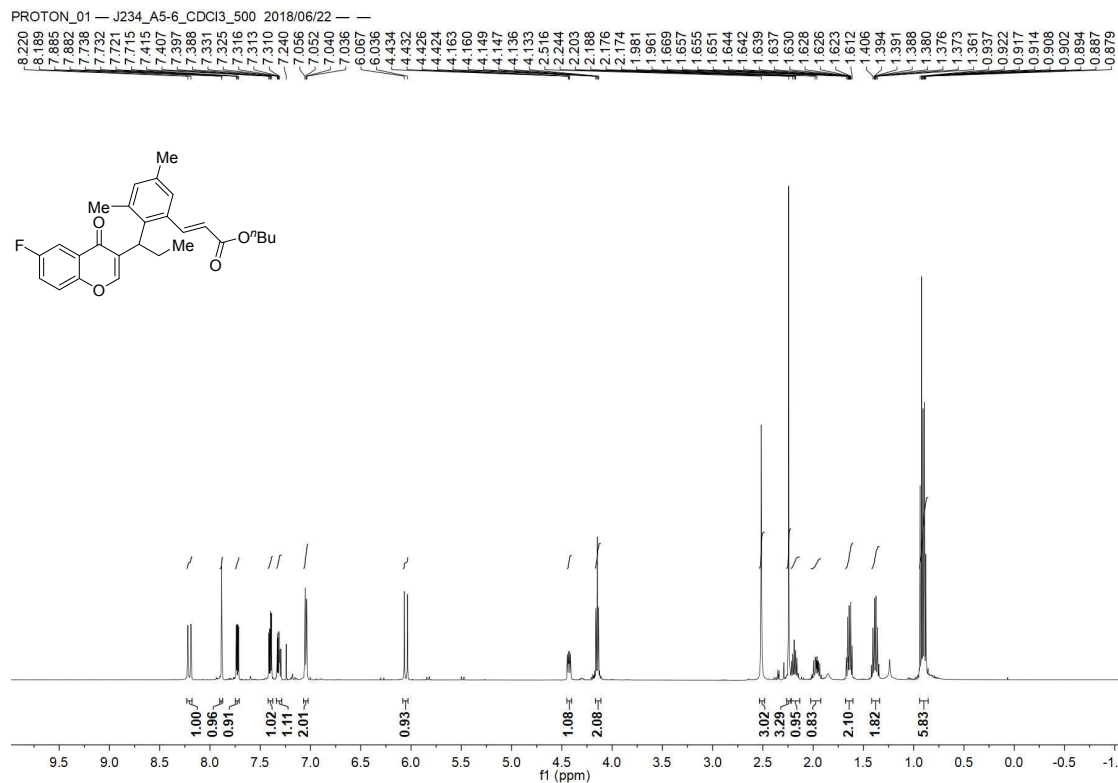


¹³C NMR

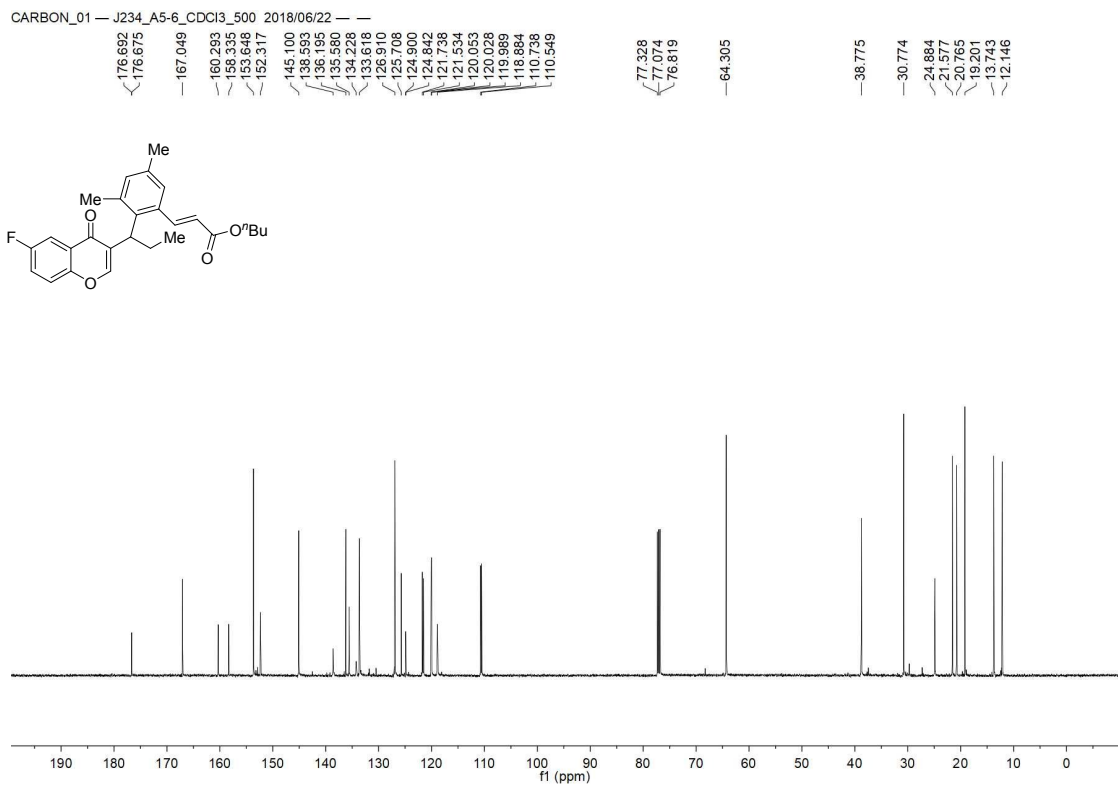


Butyl (E)-3-(2-(1-(6-fluoro-4-oxo-4H-chromen-3-yl)propyl)-3,5-dimethylphenyl)acrylate (**7c**)

¹H NMR



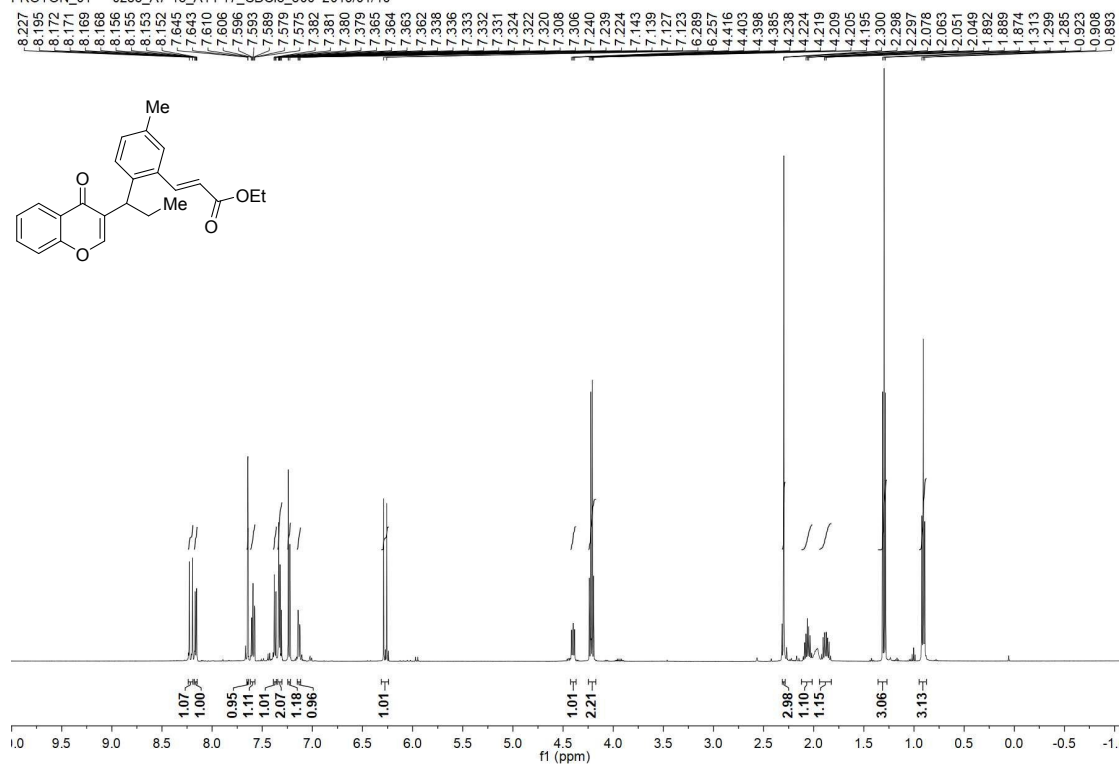
¹³C NMR



Ethyl (E)-3-(5-methyl-2-(1-(4-oxo-4H-chromen-3-yl)propyl)phenyl)acrylate (7e)

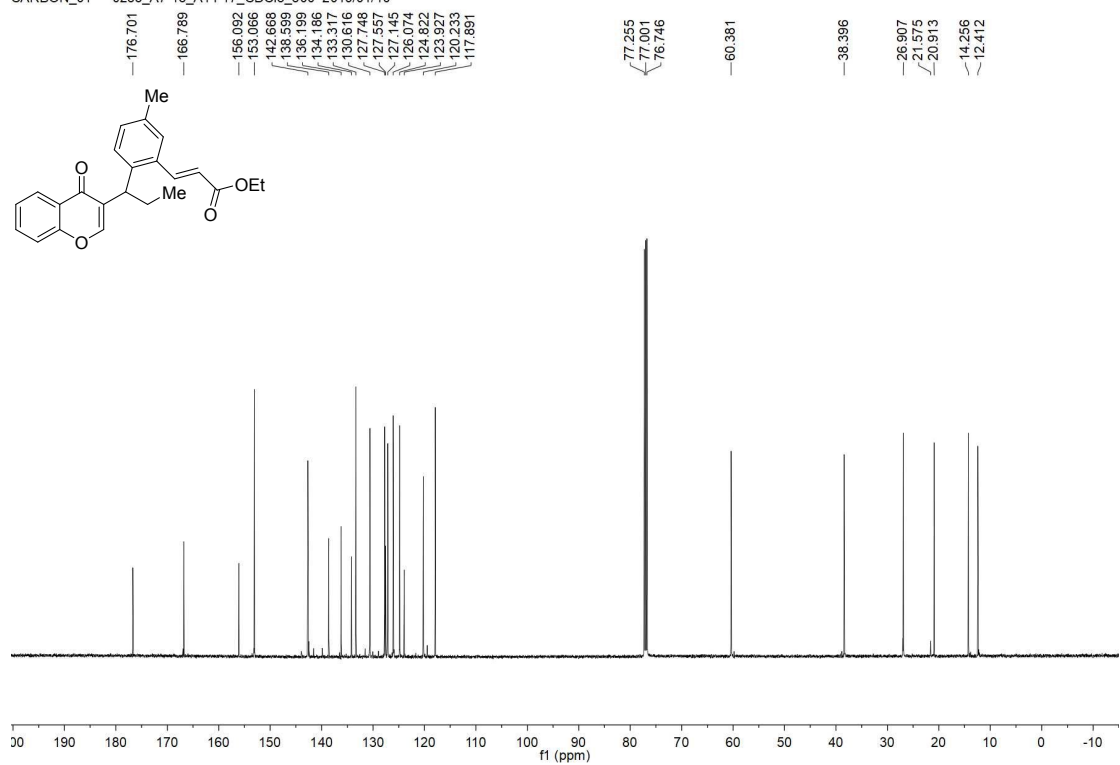
¹H NMR

PROTON_01 — J256_A7-18_A14-17_CDCl3_500 2019/01/10 —



¹³C NMR

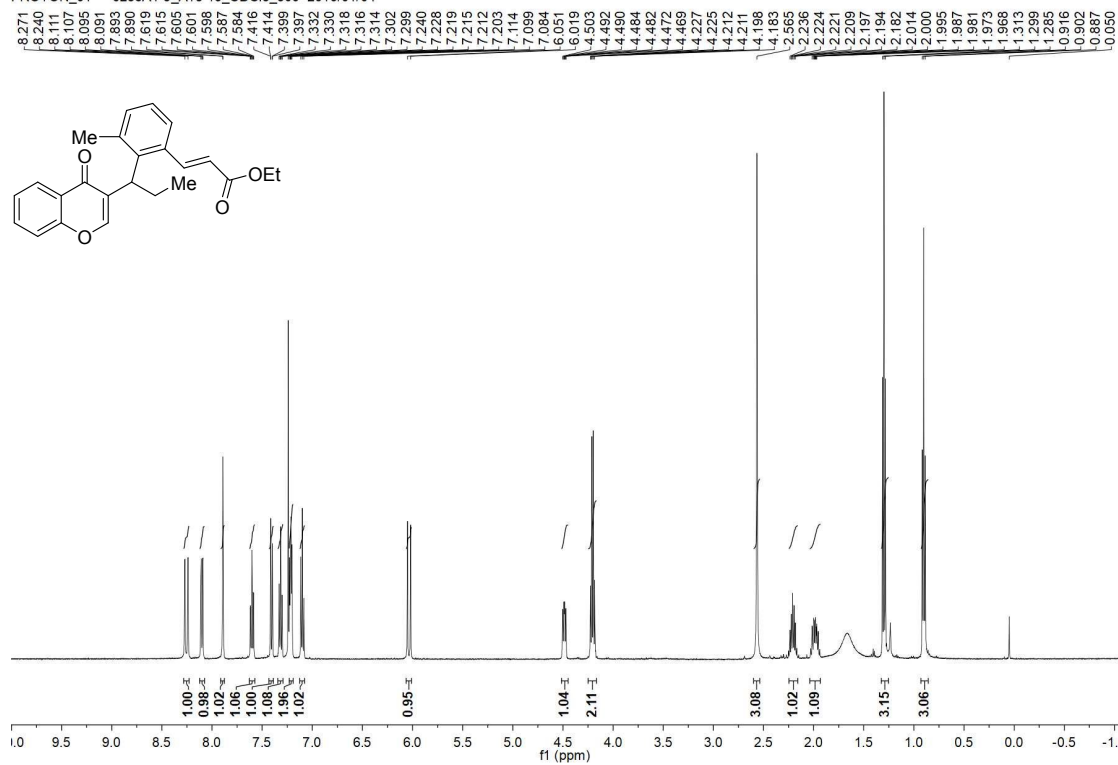
CARBON_01 — J256_A7-18_A14-17_CDCl3_500 2019/01/10 —



Ethyl (E)-3-(3-methyl-2-(1-(4-oxo-4H-chromen-3-yl)propyl)phenyl)acrylate (7e')

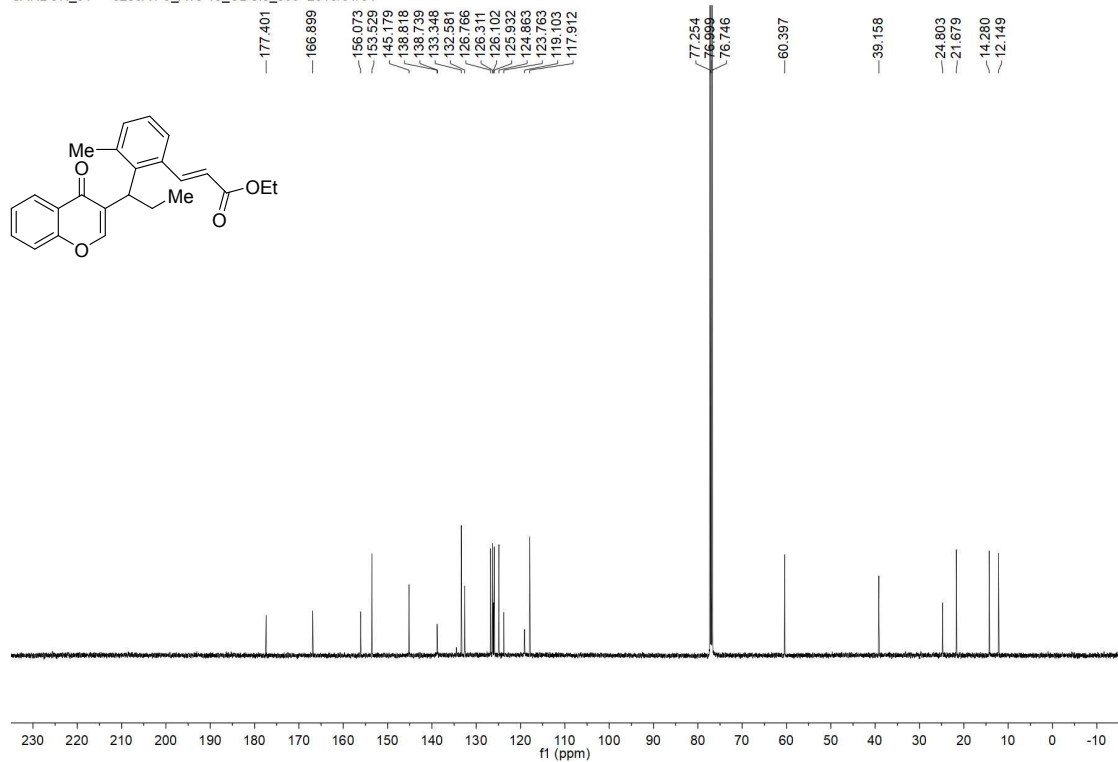
¹H NMR

PROTON_01 — J256A1-3_A15-19_CDCl3_500 2019/01/04 —



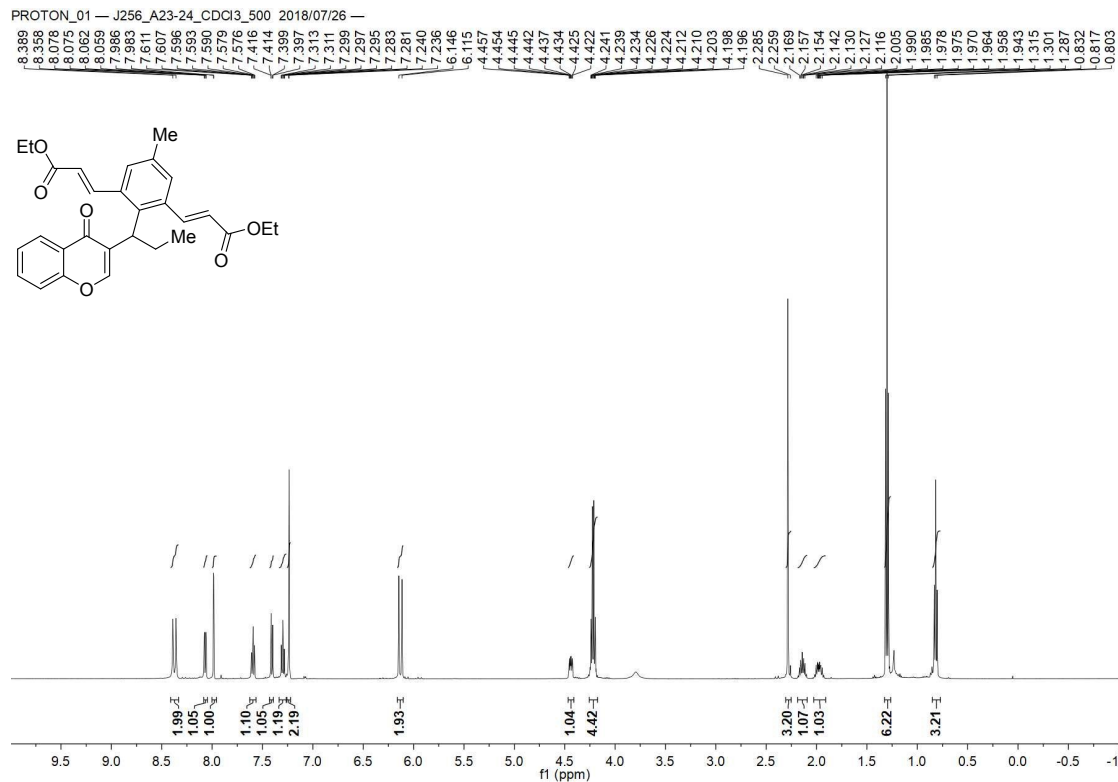
¹³C NMR

CARBON_01 — J256A1-3_A15-19_CDCl3_500 2019/01/04 —

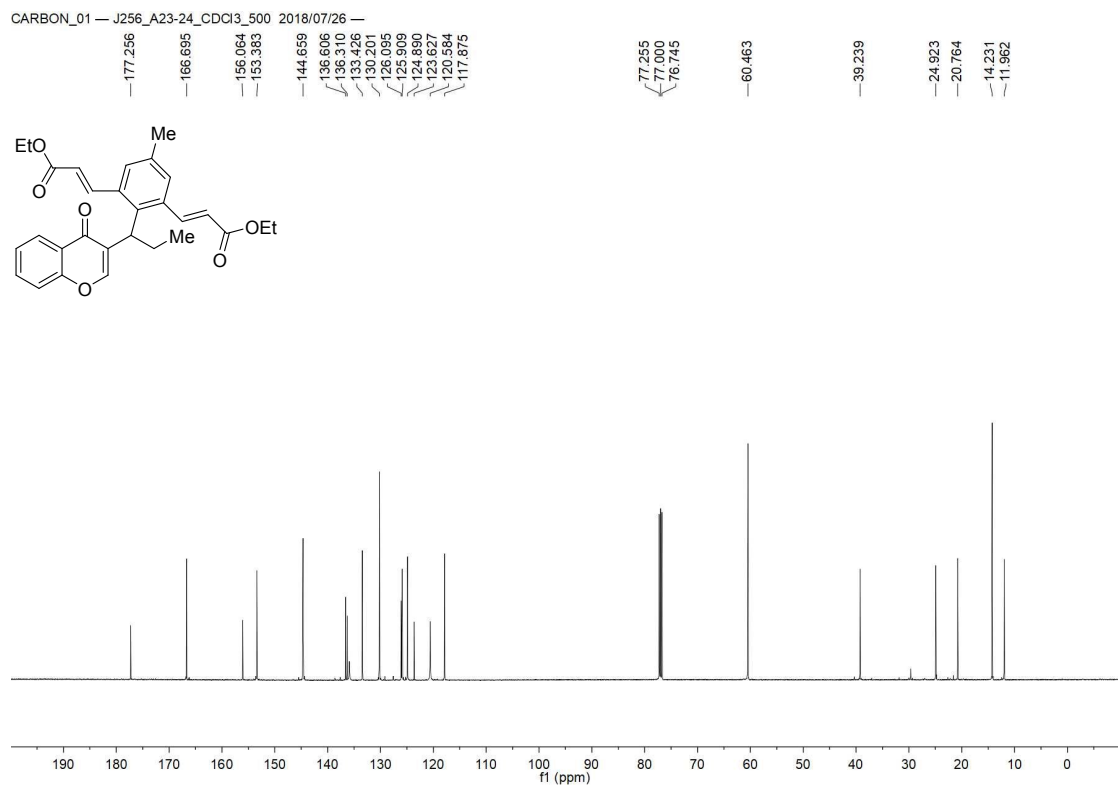


Diethyl 3,3'-(5-methyl-2-(1-(4-oxo-4H-chromen-3-yl)propyl)-1,3-phenylene)(2E,2'E)-diacrylate (7e'')

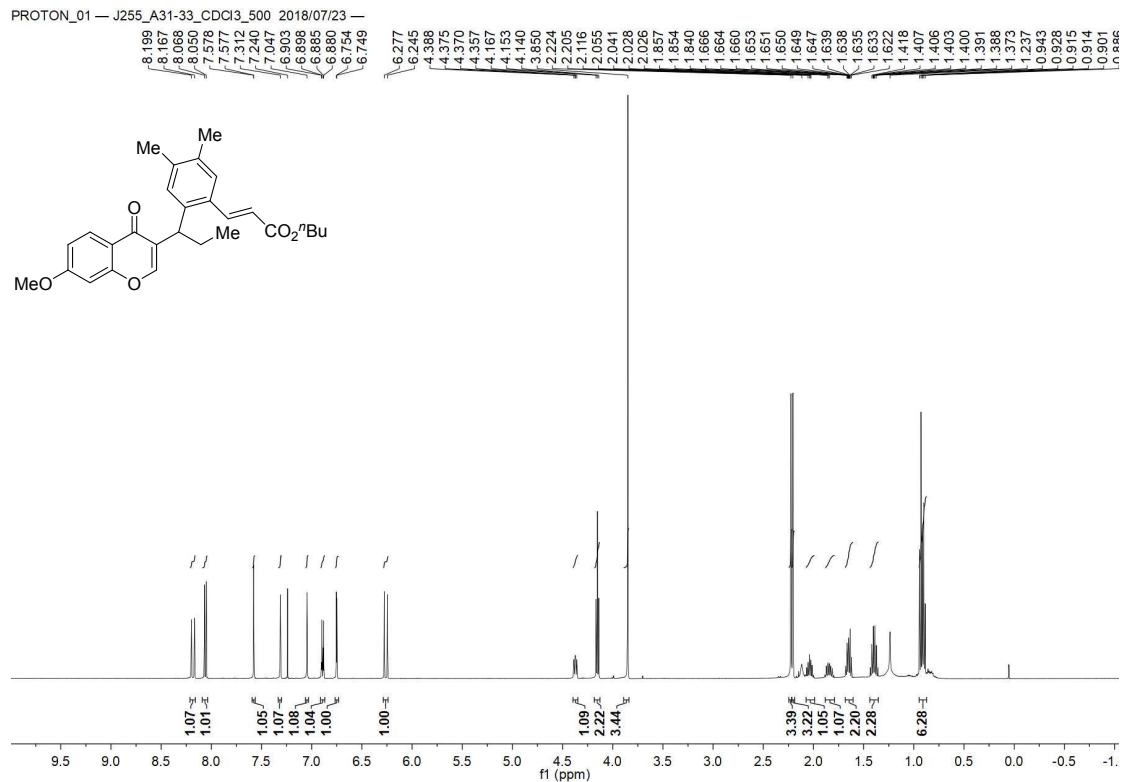
¹H NMR



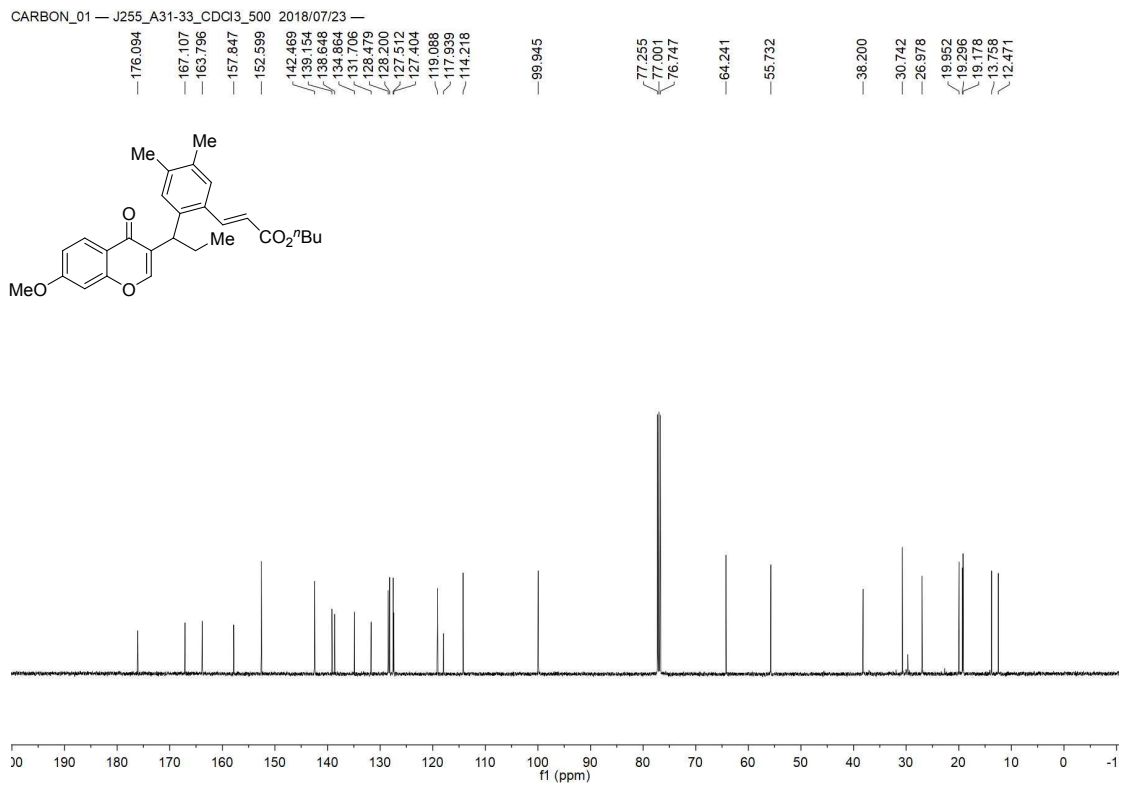
¹³C NMR



Butyl *(E)*-3-(2-(1-(7-methoxy-4-oxo-4*H*-chromen-3-yl)propyl)-4,5-dimethylphenyl)acrylate (**7f**)
¹H NMR

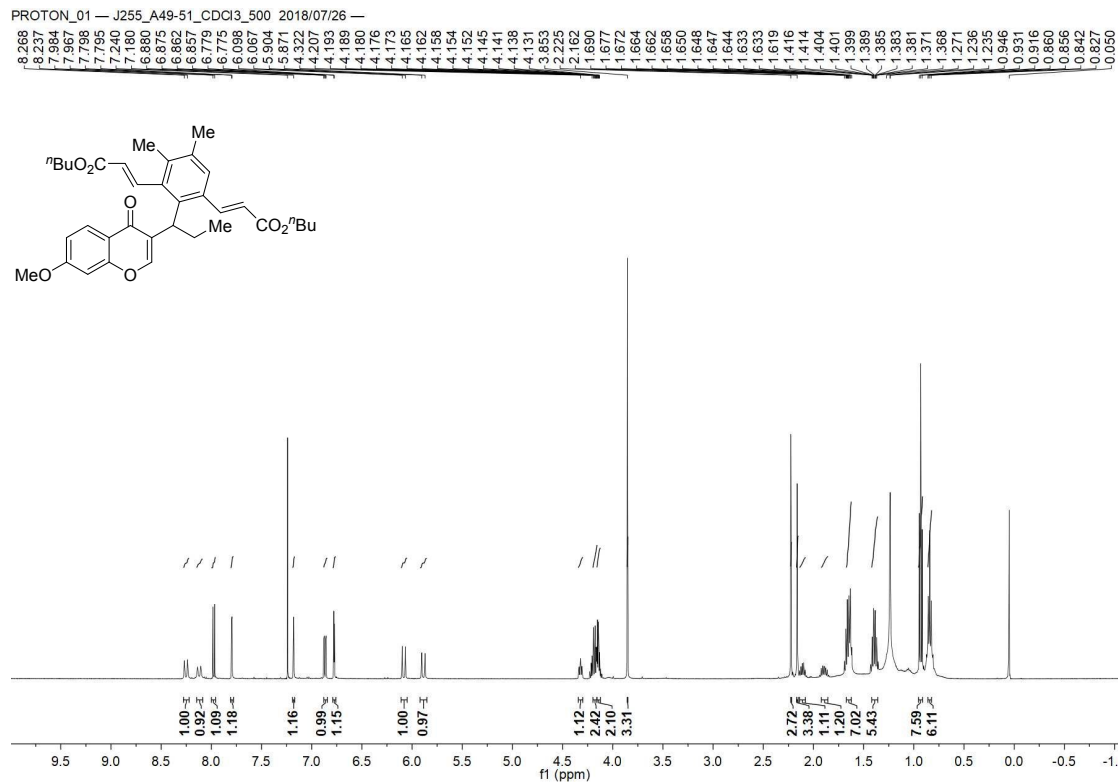


¹³C NMR

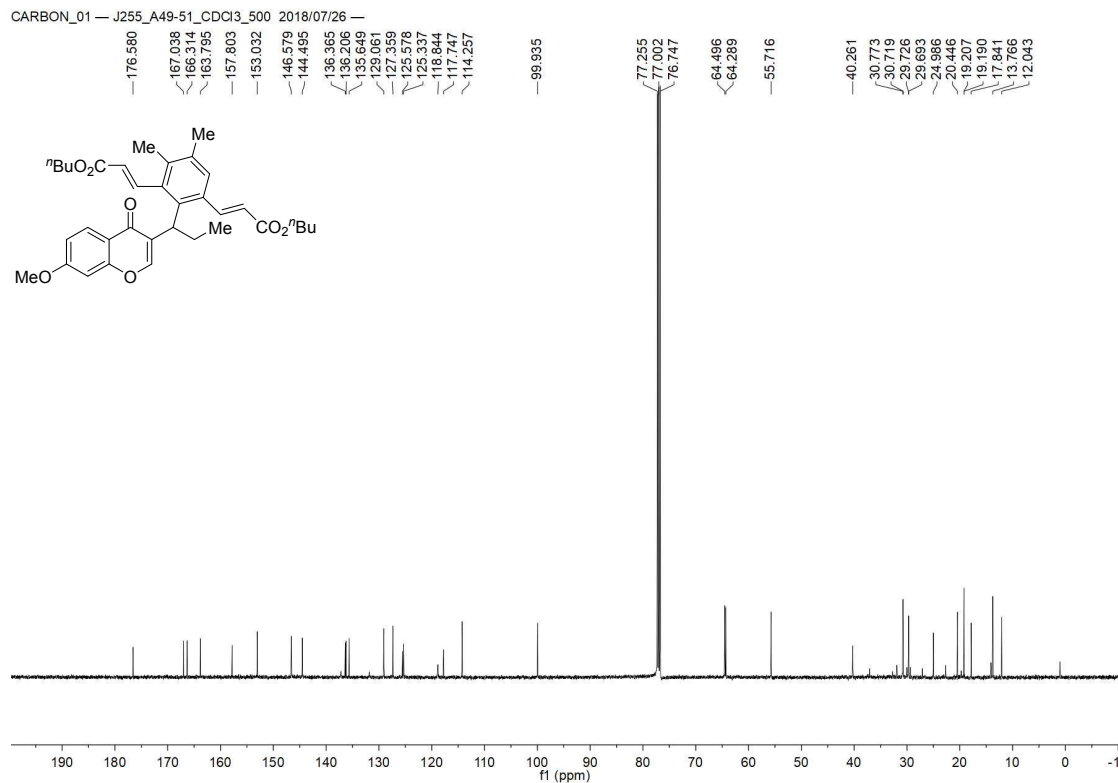


Dibutyl 3,3'-(2-(1-(7-methoxy-4-oxo-4H-chromen-3-yl)propyl)-4,5-dimethyl-1,3-phenylene)(2E,2'E)-diacrylate (7f'')

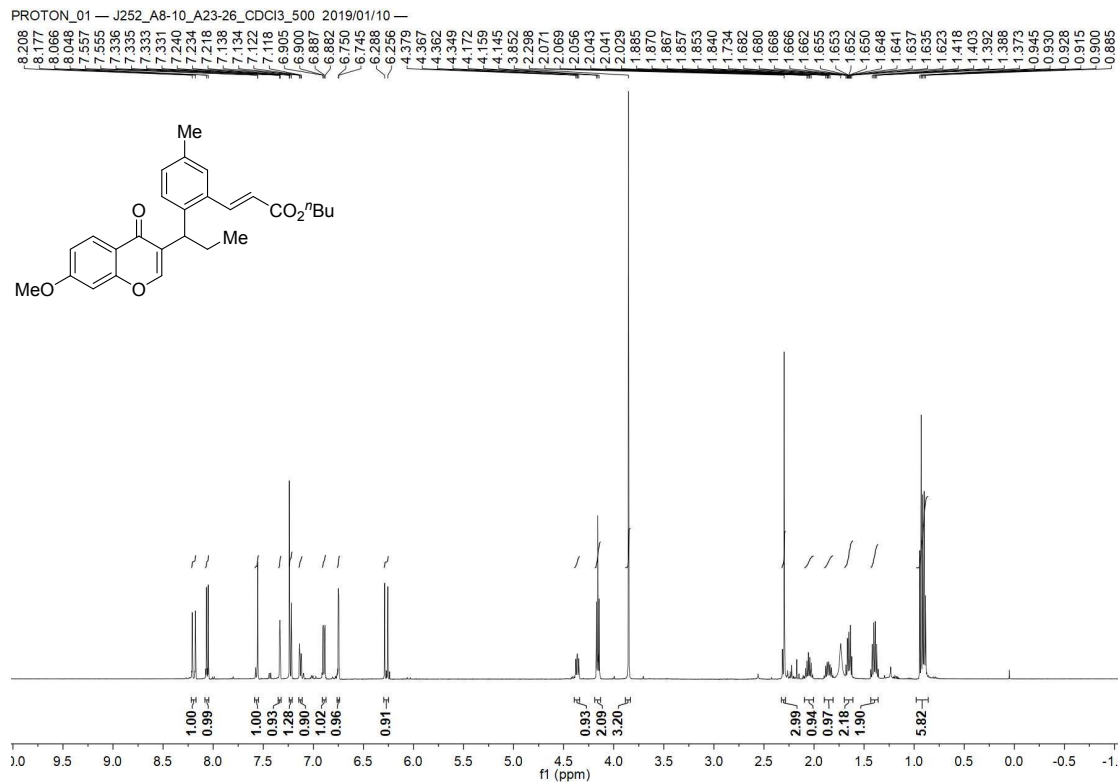
¹H NMR



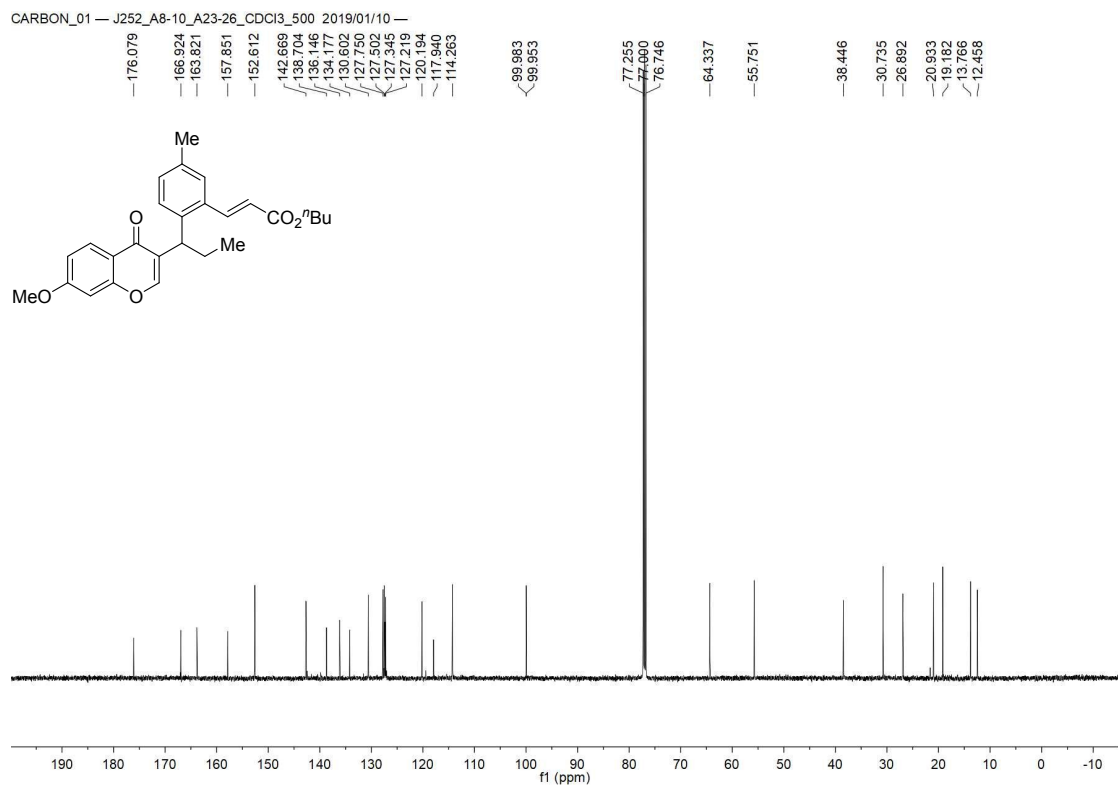
¹³C NMR



Butyl methylphenylacrylate (7g) *(E)-3-(2-(1-(7-methoxy-4-oxo-4H-chromen-3-yl)propyl)-5-methylphenyl)acrylate (7g)*
¹H NMR

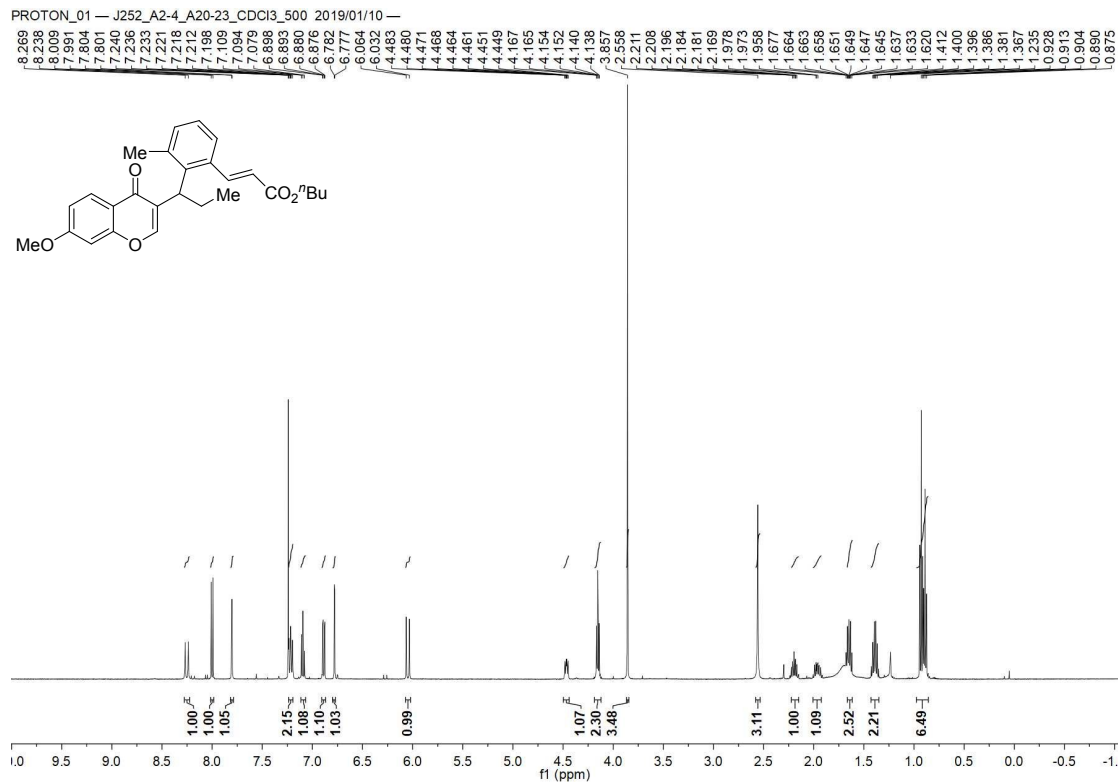


¹³C NMR

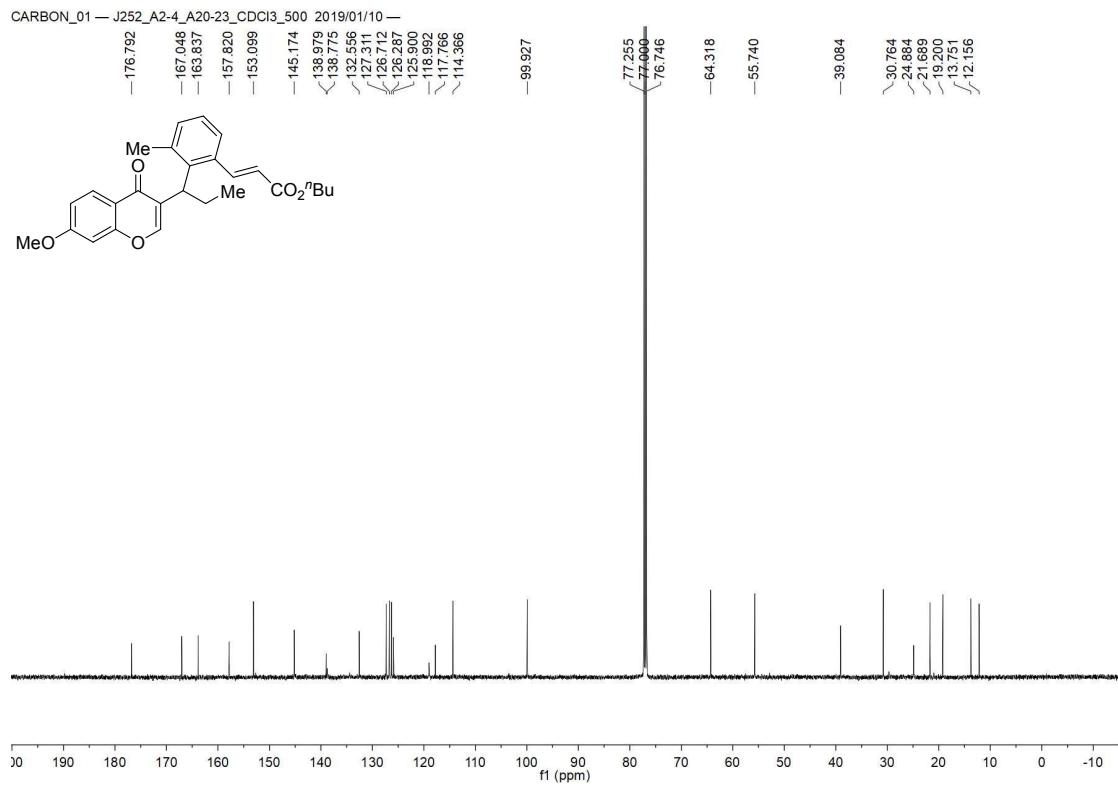


Butyl(E)-3-(2-(1-(7-methoxy-4-oxo-4H-chromen-3-yl)propyl)-3methylphenyl)acrylate
(7g')

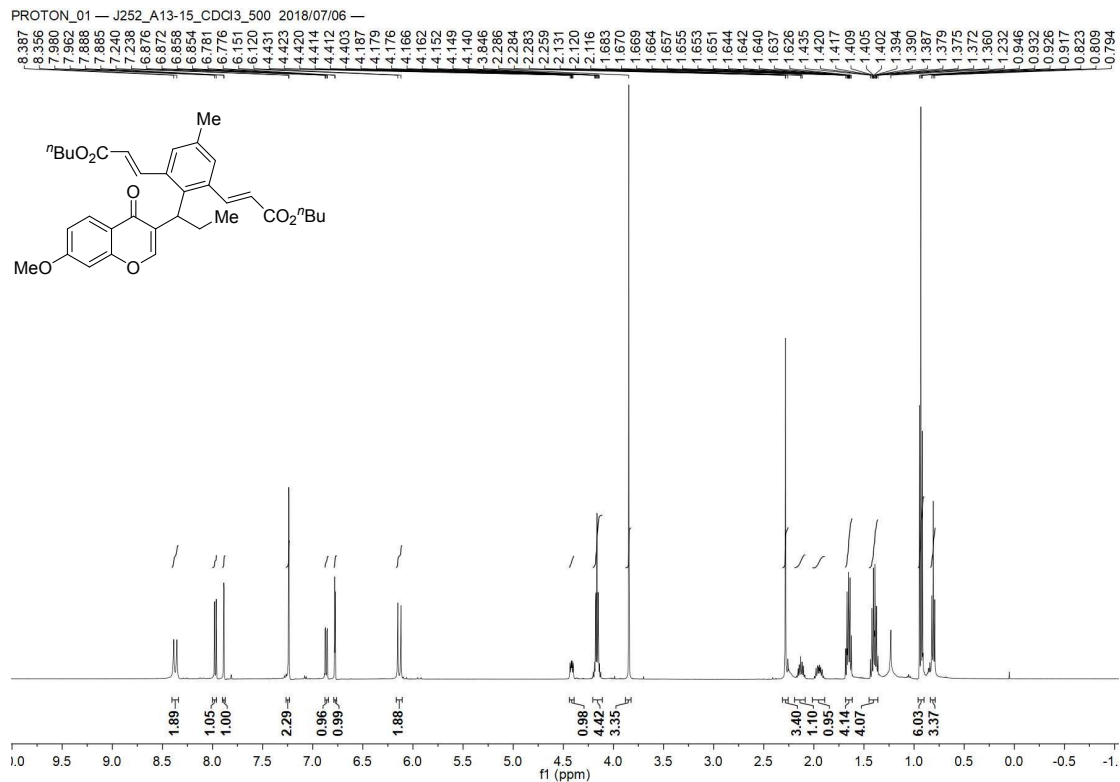
¹H NMR



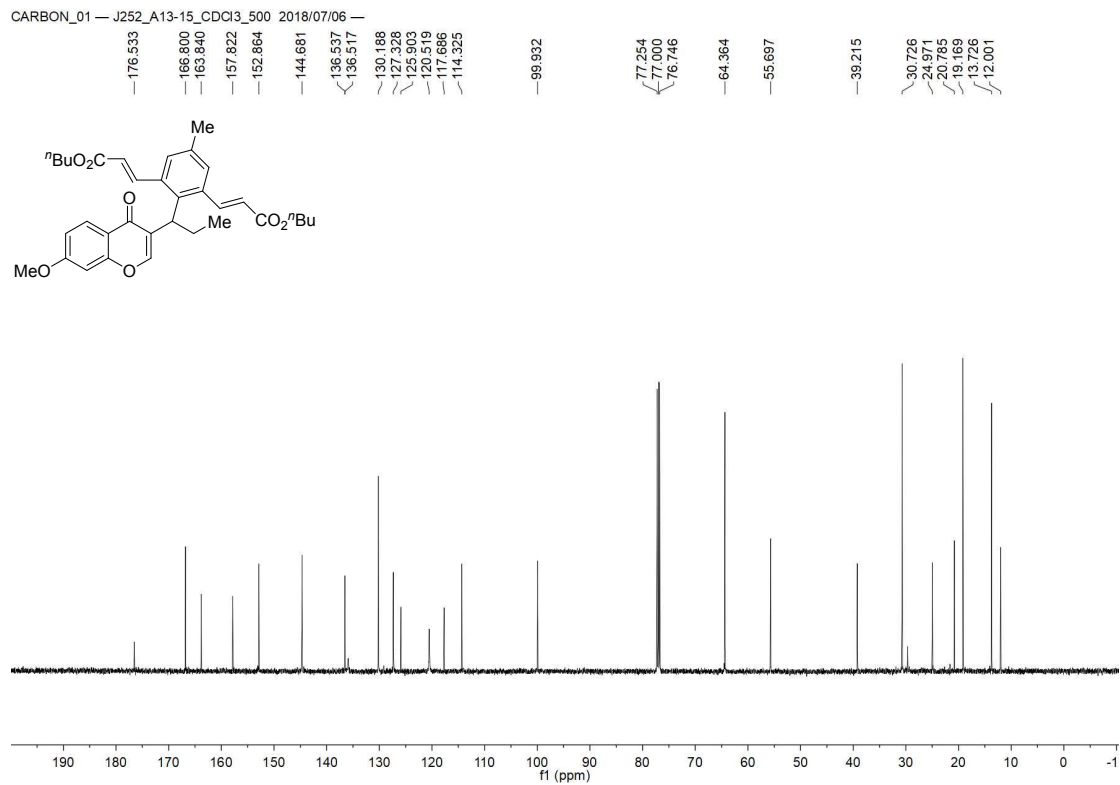
¹³C NMR



Dibutyl 3,3'-(2-(1-(7-methoxy-4-oxo-4H-chromen-3-yl)propyl)-5-methyl-1,3-phenylene)(2E,2'E)-diacrylate (7g)
¹H NMR

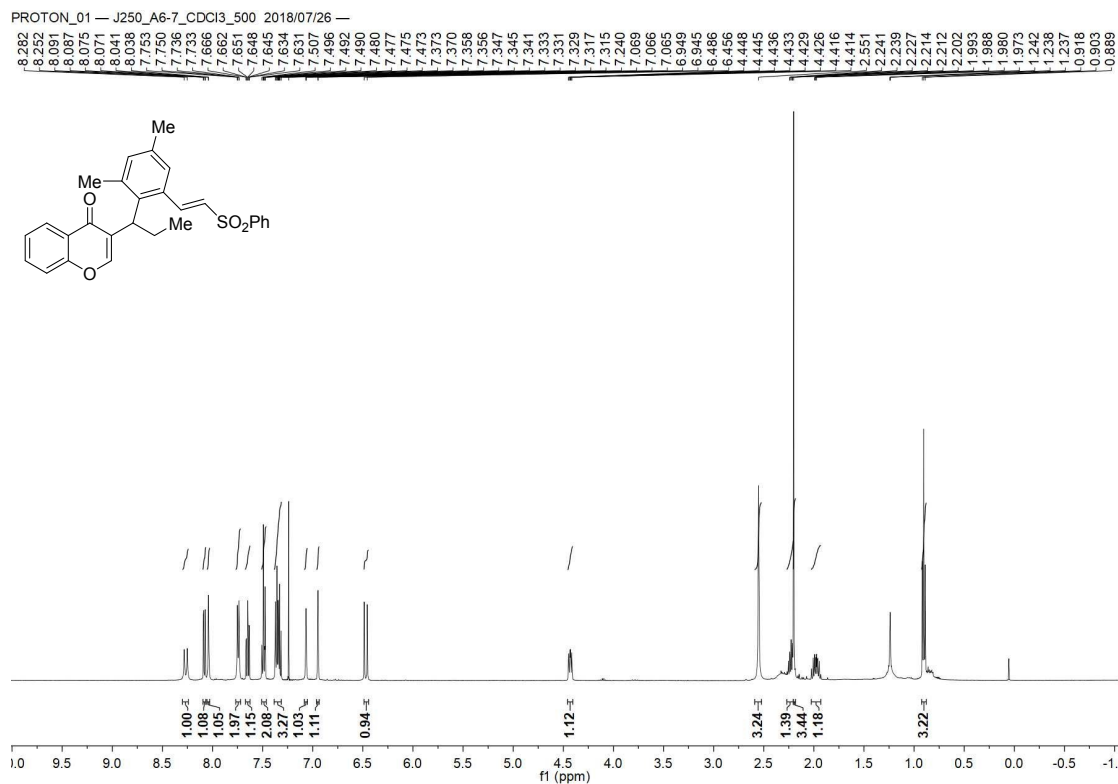


¹³C NMR

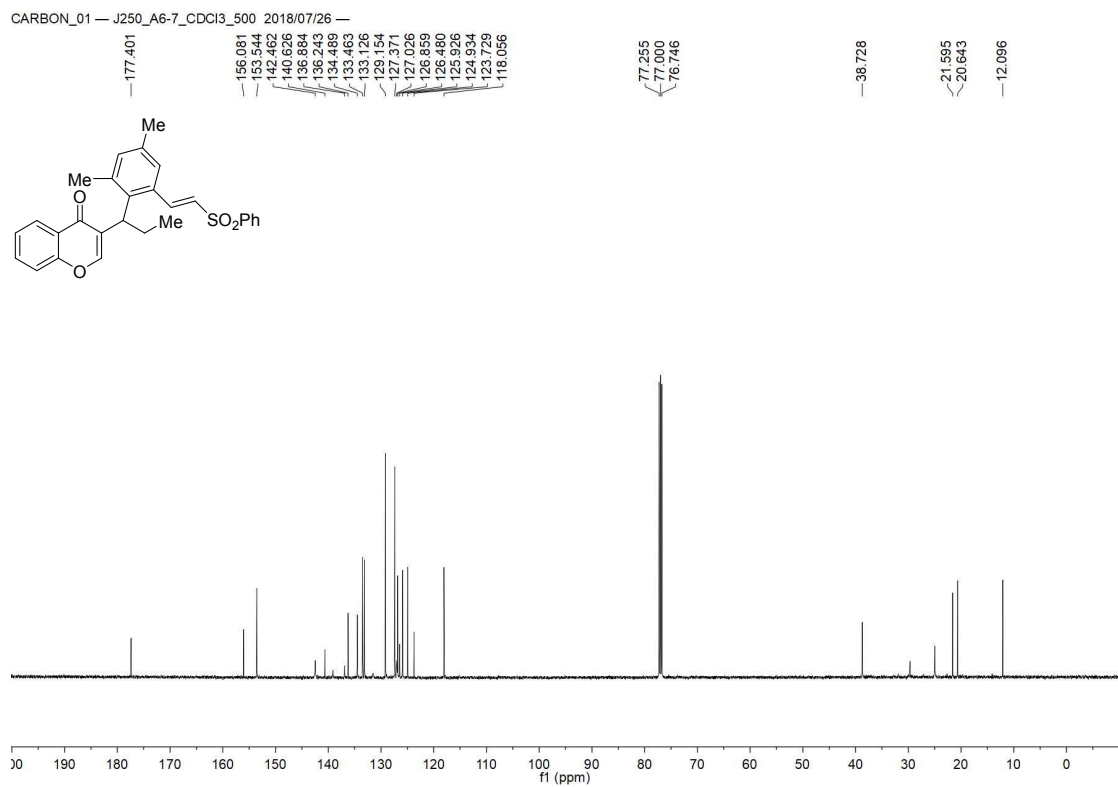


(E)-3-(1-(2,4-Dimethyl-6-(2-(phenylsulfonyl)vinyl)phenyl)propyl)-4H-chromen-4-one
(7h)

¹H NMR

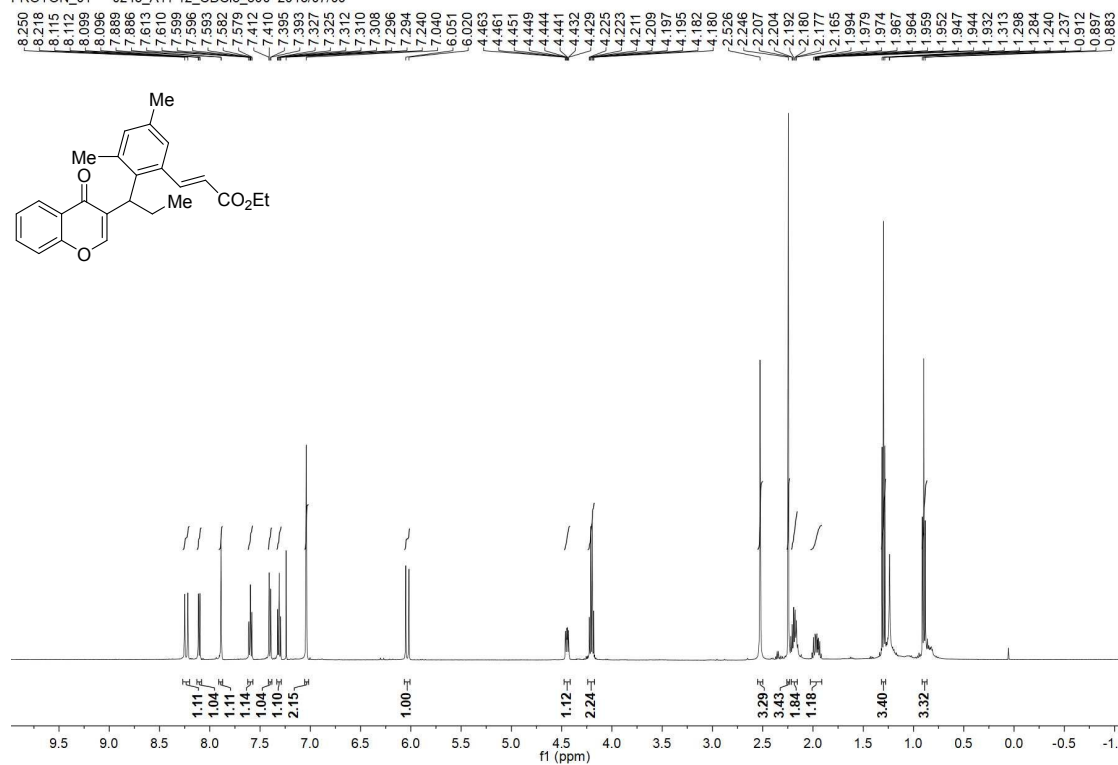


¹³C NMR



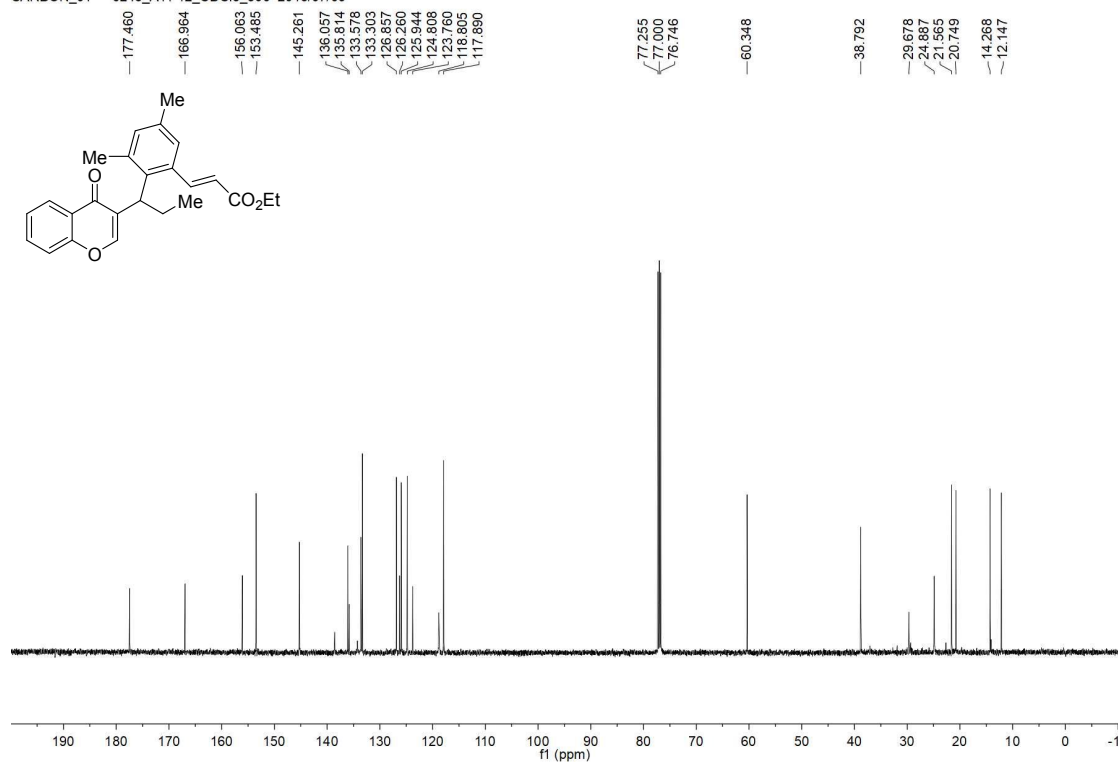
Ethyl (E)-3-(3,5-dimethyl-2-(1-(4-oxo-4H-chromen-3-yl)propyl)phenyl)acrylate (7i)
¹H NMR

PROTON_01 — J248_A11-12_CDCl3_500 2018/07/09 —



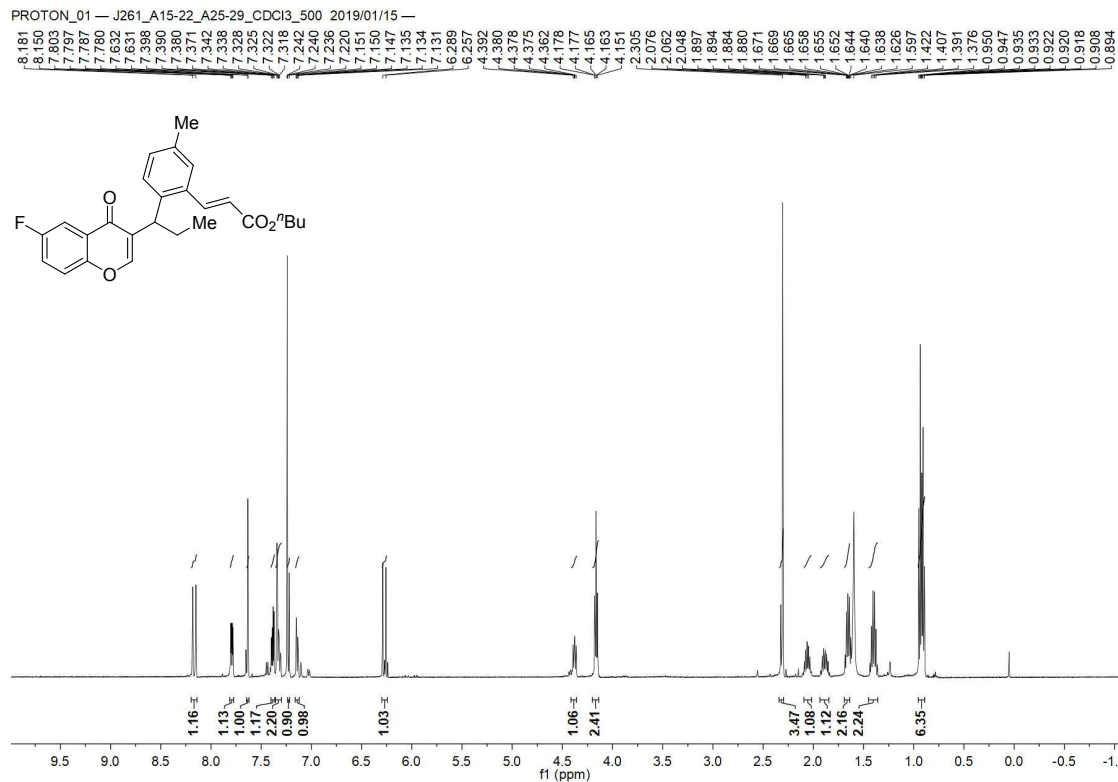
¹³C NMR

CARBON_01 — J248_A11-12_CDCl3_500 2018/07/09 —

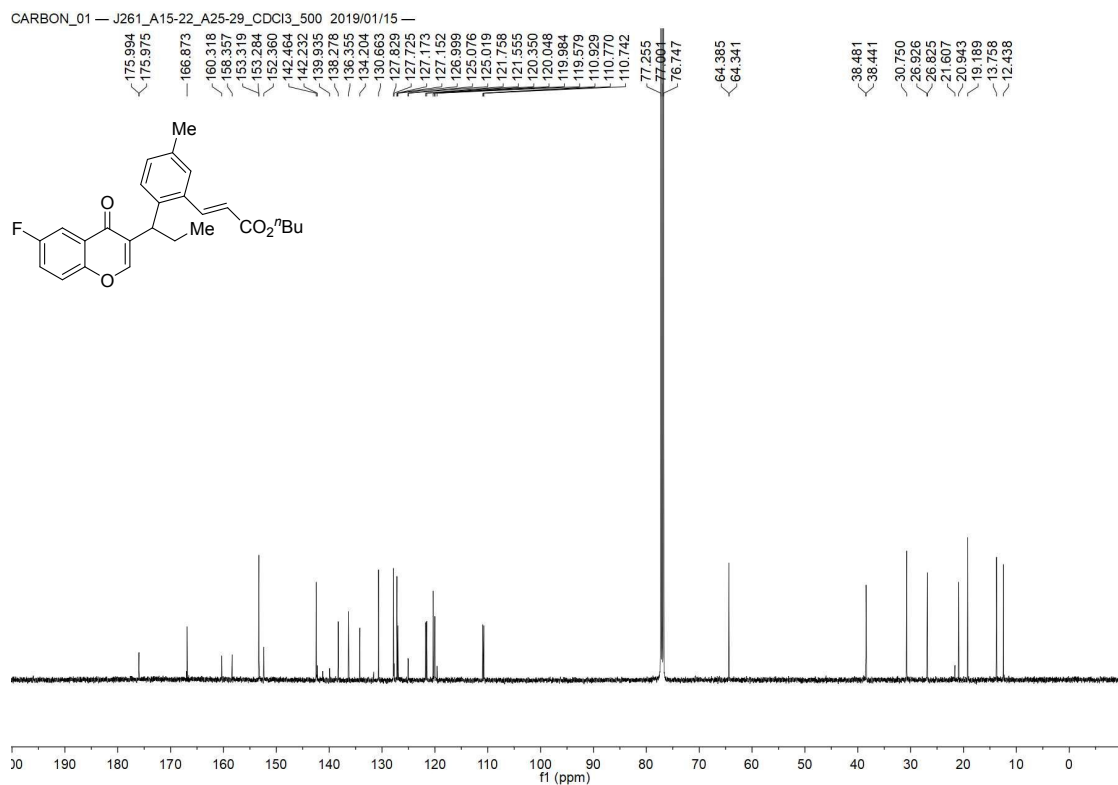


Butyl (E)-3-(2-(1-(6-fluoro-4-oxo-4H-chromen-3-yl)propyl)-5-methylphenyl)acrylate
(7j)

¹H NMR



¹³C NMR

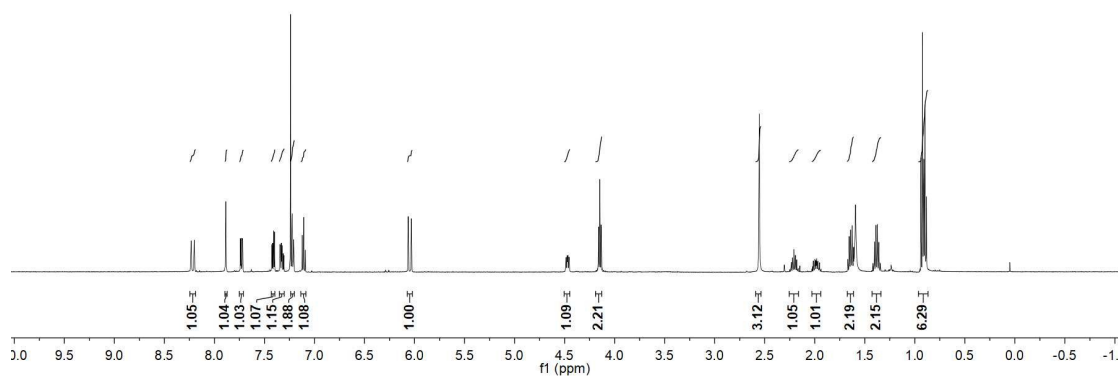
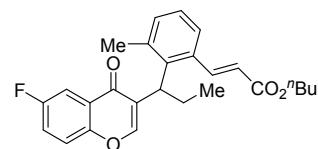


Butyl (E)-3-(2-(1-(6-fluoro-4-oxo-4H-chromen-3-yl)propyl)-3-methylphenyl)acrylate
(7j')

¹H NMR

PROTON_01 — J261_A9-11_A38-43_CDCl3_500 2019/01/22 —

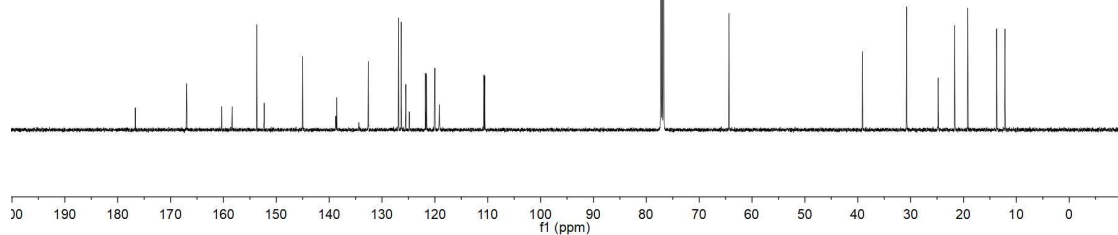
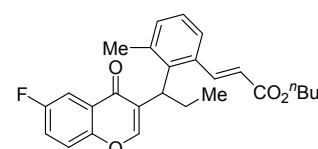
8.232, 8.201, 7.886, 7.884, 7.740, 7.734, 7.723, 7.717, 7.426, 7.408, 7.408, 7.399, 7.346, 7.340, 7.331, 7.328, 7.325, 7.321, 7.312, 7.306, 7.240, 7.237, 7.223, 7.223, 7.203, 7.192, 7.107, 7.092, 6.063, 6.031, 4.486, 4.476, 4.474, 4.469, 4.466, 4.457, 4.454, 4.162, 4.160, 4.148, 4.147, 4.135, 4.133, 2.555, 2.222, 2.208, 2.193, 1.657, 1.655, 1.644, 1.642, 1.642, 1.637, 1.630, 1.628, 1.626, 1.624, 1.612, 1.593, 1.404, 1.393, 1.389, 1.378, 1.374, 1.371, 1.359, 0.938, 0.923, 0.914, 0.909, 0.899, 0.884



¹³C NMR

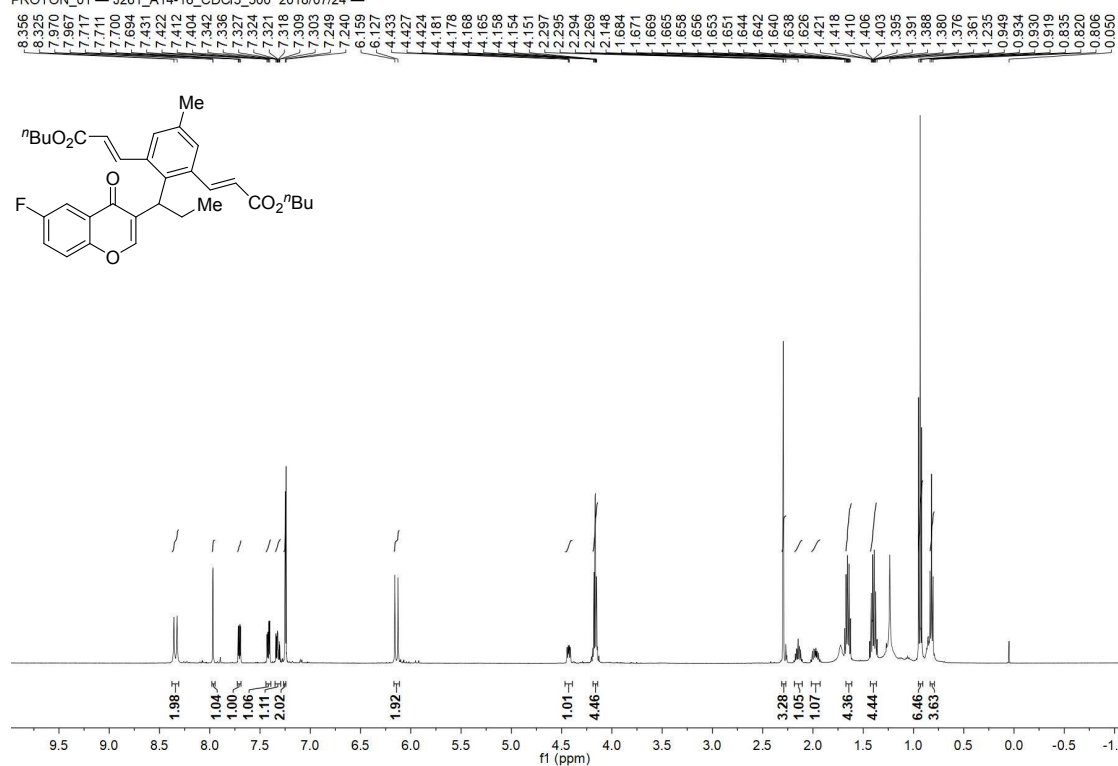
CARBON_01 — J261_A9-11_A38-43_CDCl3_500 2019/01/22 —

176.675, 176.657, 166.994, 160.316, 158.386, 153.690, 152.313, 145.005, 138.758, 138.545, 134.390, 132.607, 128.870, 128.399, 125.517, 124.872, 124.914, 121.588, 121.588, 120.087, 119.982, 119.154, 110.749, 110.561, 77.255, 77.004, 76.746, 64.353, 39.108, 30.751, 24.782, 21.669, 19.184, 13.736, 12.134



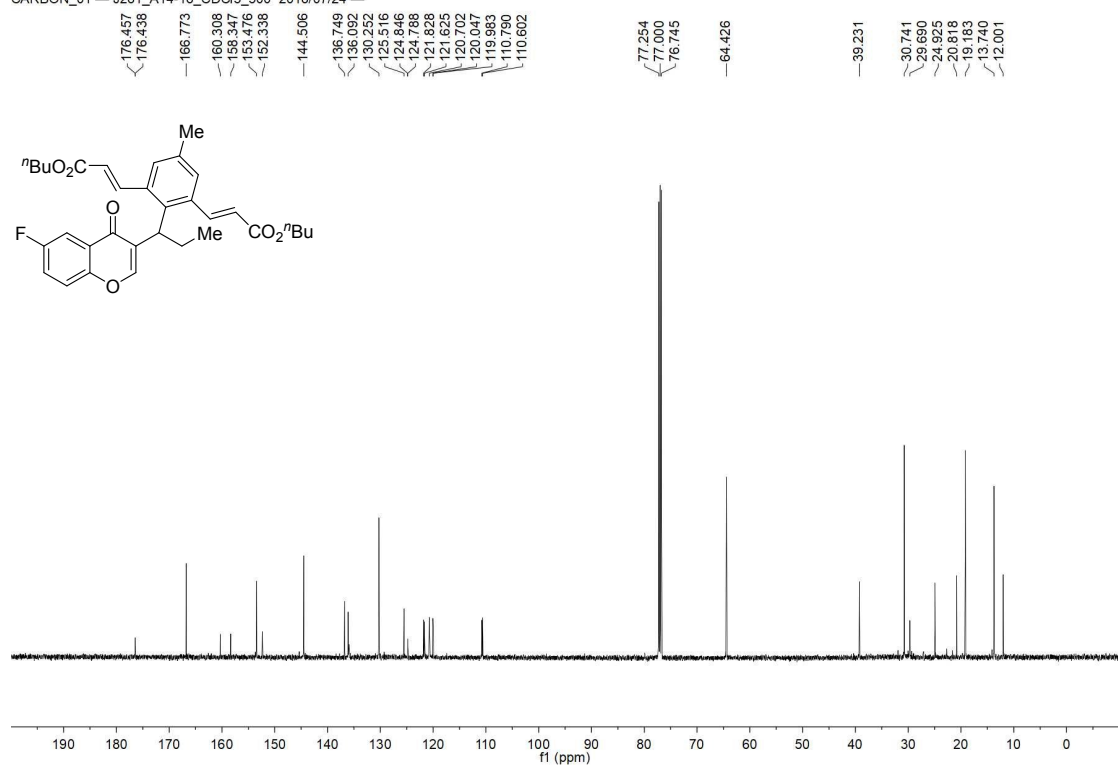
Dibutyl 3,3'-(2-(1-(6-fluoro-4-oxo-4H-chromen-3-yl)propyl)-5-methyl-1,3-phenylene)(2E,2'E)-diacrylate (7j)
¹H NMR

PROTON_01 — J261_A14-16_CDCl3_500 2018/07/24 —



¹³C NMR

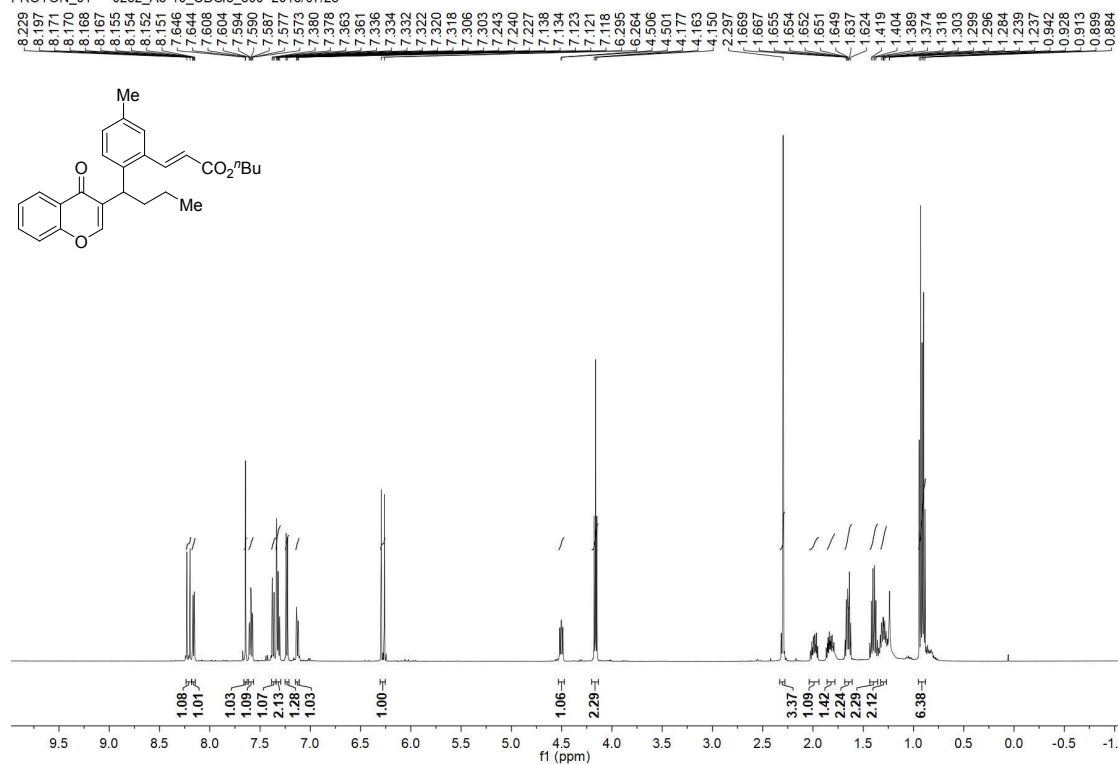
CARBON_01 — J261_A14-16_CDCl3_500 2018/07/24 —



Butyl (E)-3-(5-methyl-2-(1-(4-oxo-4H-chromen-3-yl)butyl)phenyl)acrylate (7k)

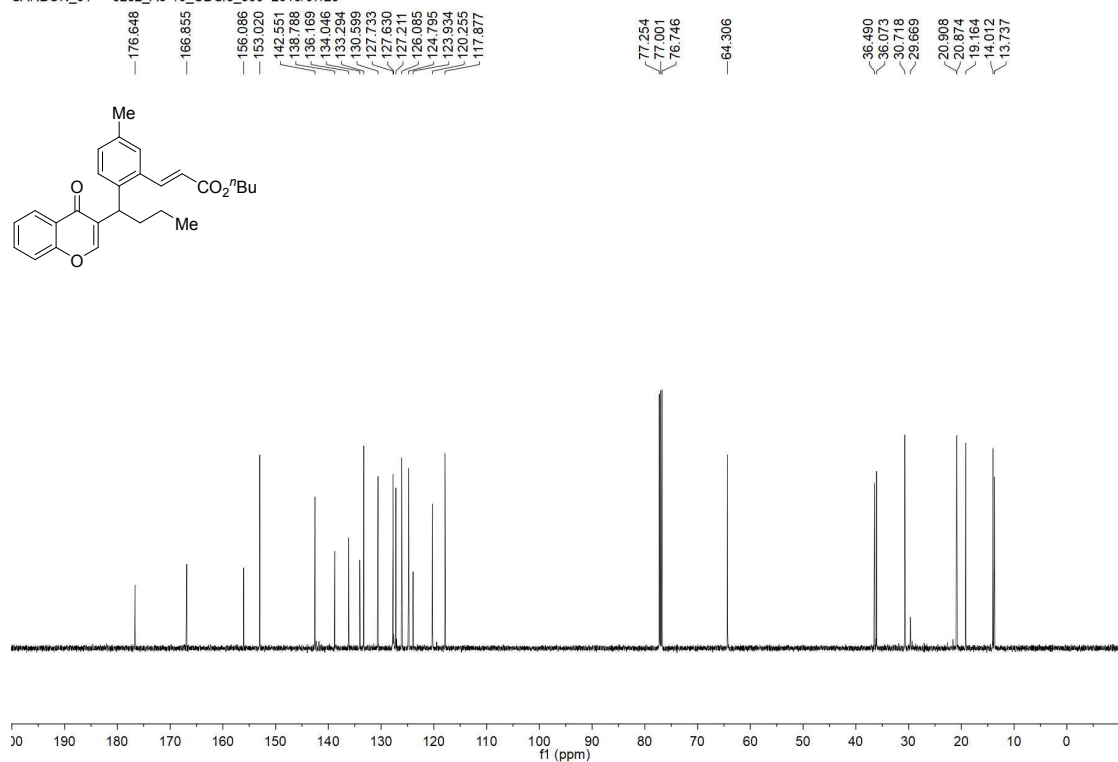
¹H NMR

PROTON_01 — J262_A8-10_CDCl3_500 2018/07/23 —



¹³C NMR

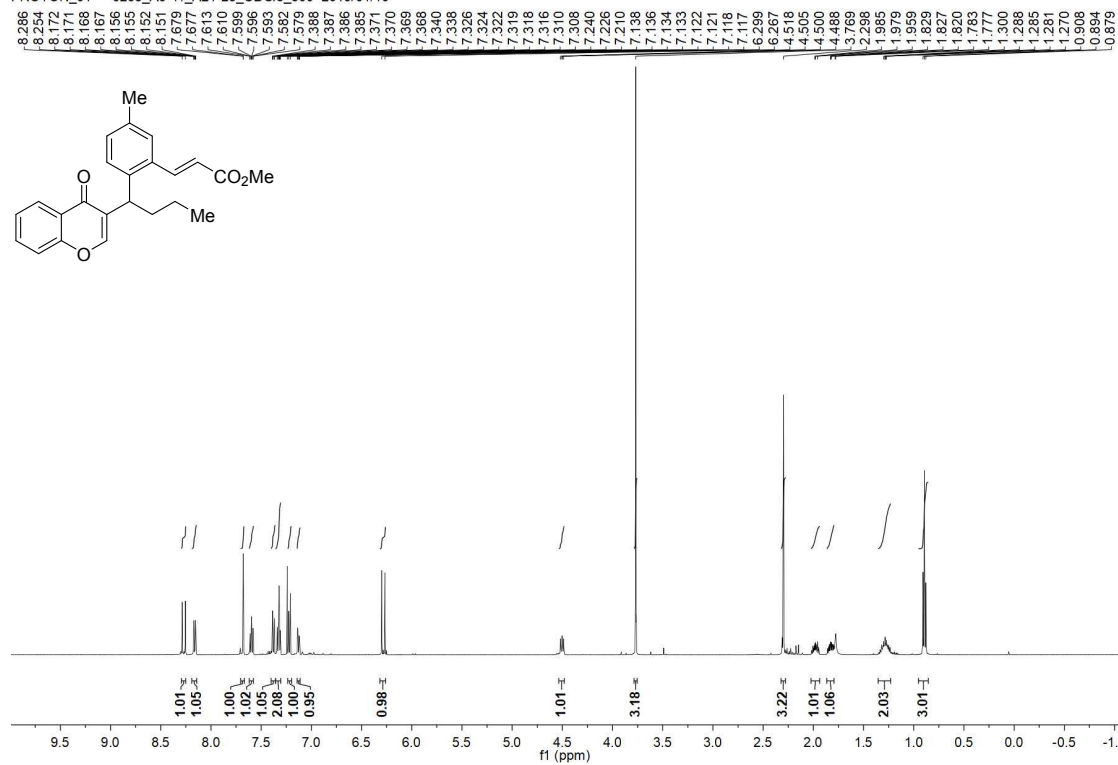
CARBON_01 — J262_A8-10_CDCl3_500 2018/07/23 —



Methyl (E)-3-(5-methyl-2-(1-(4-oxo-4H-chromen-3-yl)butyl)phenyl)acrylate (71)

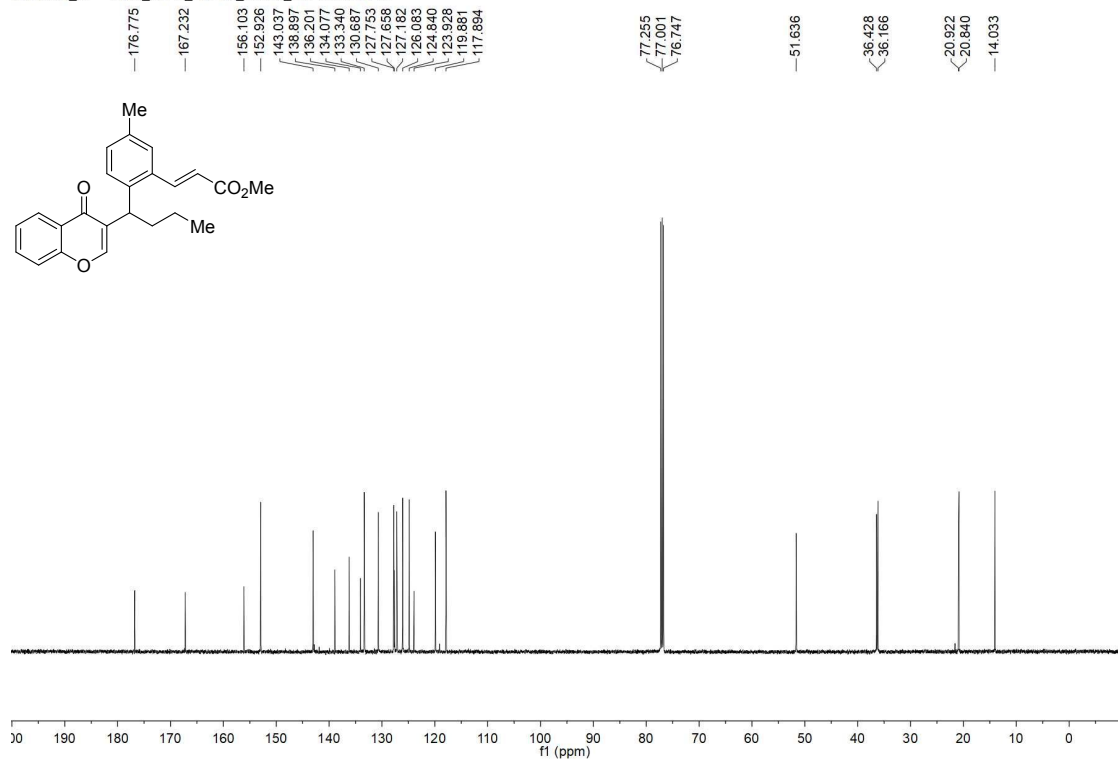
¹H NMR

PROTON_01 — J263_A9-11_A21-25_CDCl3_500 2019/01/10 —



¹³C NMR

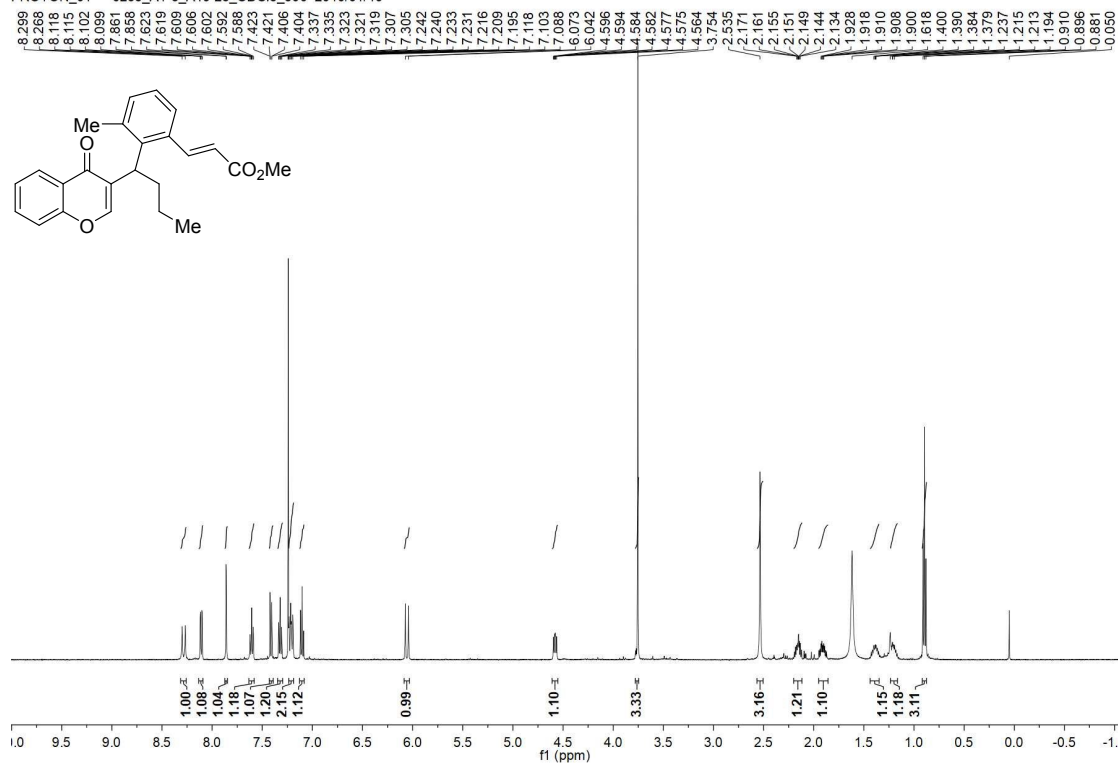
CARBON_01 — J263_A9-11_A21-25_CDCl3_500 2019/01/10 —



Methyl (E)-3-(3-methyl-2-(1-(4-oxo-4H-chromen-3-yl)butyl)phenyl)acrylate (71')

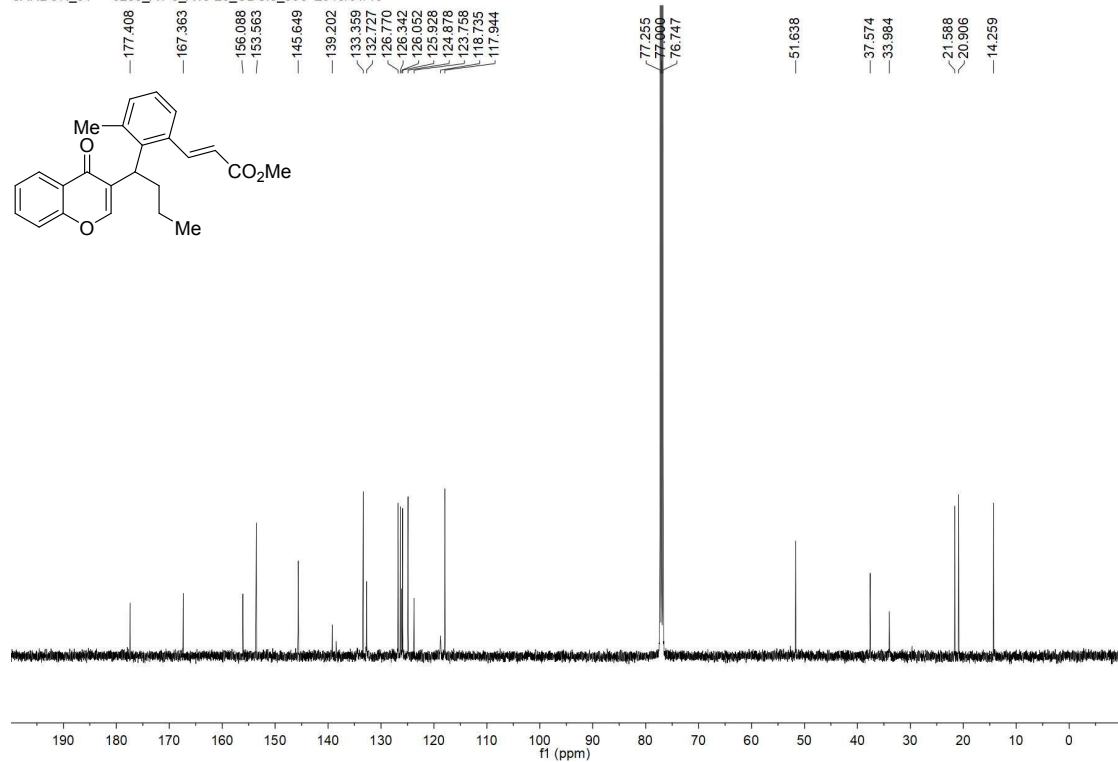
¹H NMR

PROTON_01 — J263_A1-6_A19-28_CDCl3_500 2019/01/19 —



¹³C NMR

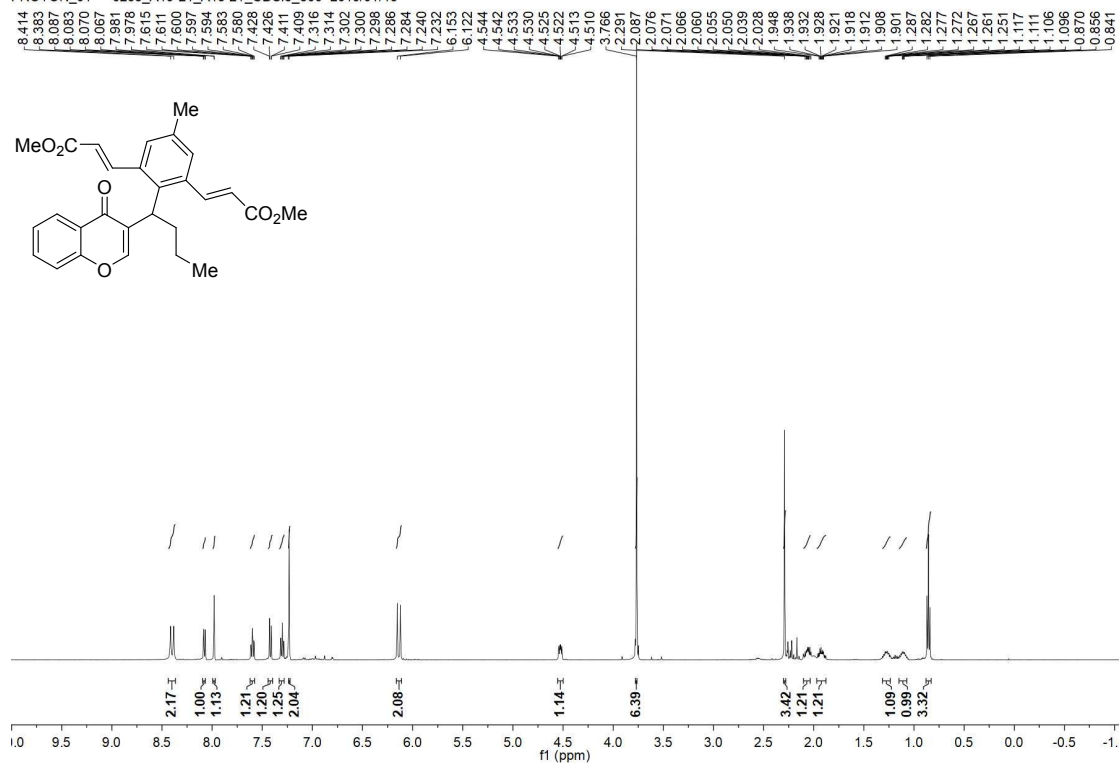
CARBON_01 — J263_A1-6_A19-28_CDCl3_500 2019/01/19 —



Methyl (E)-3-(5-methyl-2-(1-(4-oxo-4H-chromen-3-yl)butyl)phenyl)acrylate (71'')

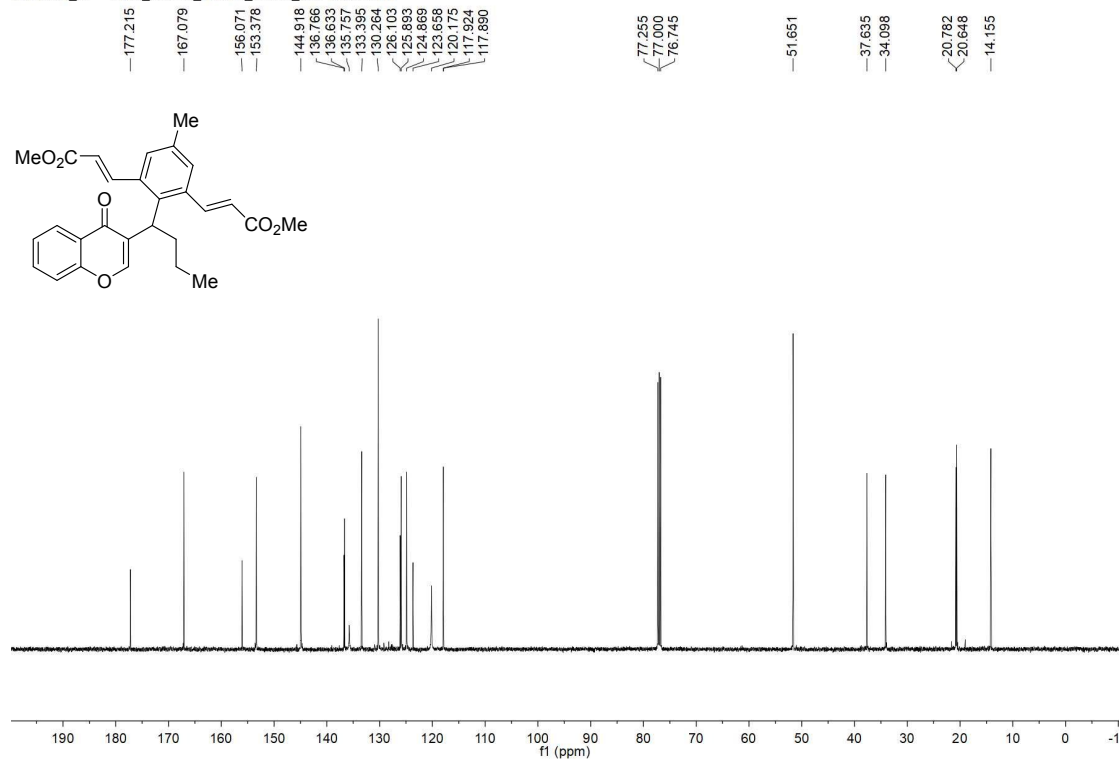
¹H NMR

PROTON_01 — J263_A19-21_A13-21_CDCl3_500 2019/01/15 —



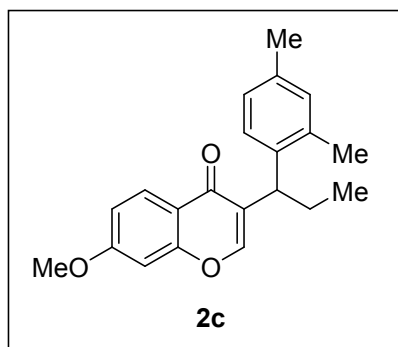
¹³C NMR

CARBON_01 — J263_A19-21_A13-21_CDCl3_500 2019/01/15 —



Single crystal X-ray diffraction

The single crystal was obtained by slow evaporation of **2c** in a solution of acetone and n-hexane. The structure was solved with the Oxford Gemini dual system dingle-crystal XRD equipped with Cryojet, and refined with the SHELXL-2014 using Least Squares minimization. The X-ray crystallographic data can be obtained free of charge from the Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif.



IC19207 in P-1

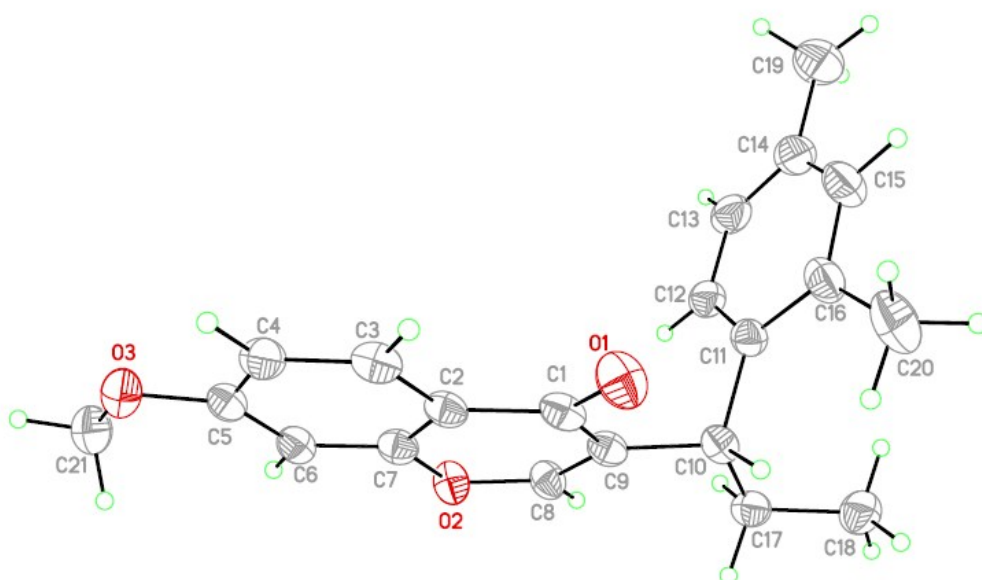


Figure S1. ORTEP plot of the crystallographic structure of **2c**. Thermal probability ellipsoids are shown at the 50% probability level.

Table S1. Crystal data and experimental details for ic19207 (**2c**).

	Crystal data	
Empirical formula	C ₂₁ H ₂₂ O ₃	
Formula weight	322.38	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 6.3862(3) Å	α = 100.358(5)°.
	b = 8.2346(4) Å	β = 97.648(5)°.
	c = 17.2255(12) Å	γ = 95.824(4)°.
Volume	875.83(9) Å ³	
Z	2	
F(000)	344	
Density (calculated)	1.222 Mg/m ³	
Wavelength	0.71073 Å	
Cell parameters reflections used	2406	
Theta range for Cell parameters	4.0070 to 28.7000°.	
Absorption coefficient	0.081 mm ⁻¹	
Temperature	150(2) K	
Crystal size	0.20 x 0.20 x 0.15 mm ³	
	Data collection	
Diffractionmeter	Xcalibur, Atlas, Gemini	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00000 and 0.95886	
No. of measured reflections	5933	
No. of independent reflections	3771 [R(int) = 0.0194]	
No. of observed [I > 2σ(I)]	2764	
Completeness to theta = 25.242°	99.2 %	
Theta range for data collection	3.152 to 27.497°.	
	Refinement	
Final R indices [I > 2σ(I)]	R1 = 0.0618, wR2 = 0.1916	
R indices (all data)	R1 = 0.0861, wR2 = 0.2156	
Goodness-of-fit on F ²	1.329	
No. of reflections	3771	
No. of parameters	217	
No. of restraints	0	
Largest diff. peak and hole	0.414 and -0.407 e.Å ⁻³	