Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Synthesis of α-pyrones via gold-catalyzed cycloisomerization/[2+1] cycloaddition/rearrangement of enyne-amides and sulfur ylides

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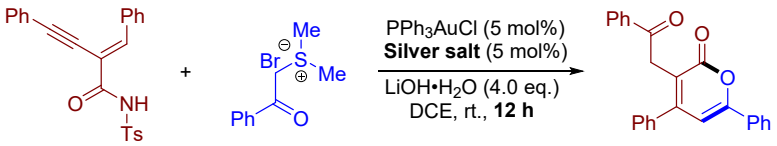
1. General Information

Commercially available materials purchased from Bidepharm or Energy Chemical was used as received. DCE was newly distilled over CaH_2, THF was distilled over sodium, and $CHCl_3$ was newly distilled over CaH_2. Other solvents were dried over 4Å molecular sieve prior use. Unless otherwise specified, all reactions were carried out under an atmosphere of nitrogen in 10 mL Schlenk tube.

^1H NMR, ^{13}C NMR spectra were measured at 400 MHz and 151 MHz in $CDCl_3$. Data for ^1H NMR spectra are reported as follows: chemical shift (ppm, referenced to TMS; s = singlet, d = doublet, t = triplet, dd = doublet of doublets, dt = doublet of triplets, m = multiplet), coupling constant (Hz), and integration. Data for ^{13}C NMR are reported in terms of chemical shift (ppm) relative to residual solvent peak ($CDCl_3$: 77.16 ppm). High-resolution mass spectrometry (HRMS) analysis was carried out using a TOF MS instrument with ESI or APCI source.

Reactions were monitored by thin layer chromatography (TLC) using silicycle pre-coated silica gel plates. Flash column chromatography was performed on silica gel 60 (particle size 200-300 mesh ASTM, purchased from Yantai, China) and eluted with petroleum ether/ethyl acetate. Ynamides were prepared according to previous literature procedures. [1,2] Various sulfur ylide salts were prepared from dimethyl sulfide and the different substituents of bromoacetophenone according to the literature.[3]

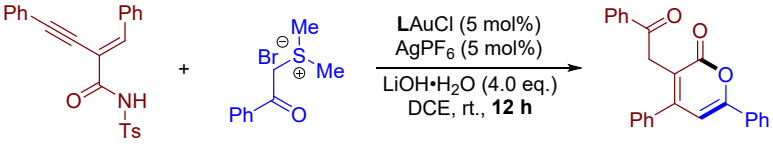
2. Some Optimization of Reaction Conditions



1a (0.2 mmol) **2a** (1.0 eq.) **3a**

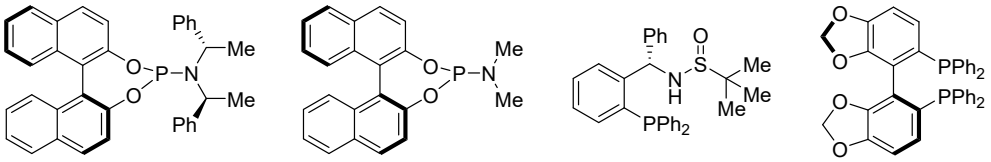
Silver Salt:

1. **$AgSbF_6$,** 54%;
2. **$AgClO_4$,** 53%
3. **AgOTf,** 61%;
4. **$AgNTf_2$,** 29%



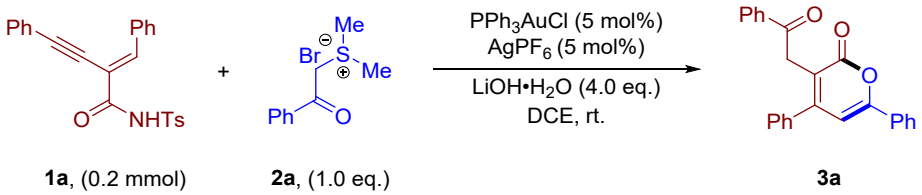
1a (0.2 mmol) **2a** (1.0 eq.) **3a**

Ligand



L1, 68% **L2**, 59% **L3**, 65% **L4**, 46%

3. General Procedure for the Synthesis of Products

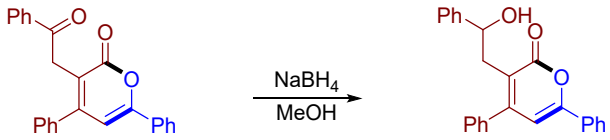


1a, (0.2 mmol) **2a**, (1.0 eq.) **3a**

In a dried and nitrogen filled Schlenk flask, a mixture of PPh_3AuCl (5.0 mg, 0.01 mmol, 5 mol %), $AgPF_6$ (2.5 mg, 0.01mmol, 5 mol %) in DCE (2 mL) was stirred at room temperature under for 15 mins to generate the gold catalyst. Ynamide **1a** (80.2 mg, 0.2 mmol) was added to the above catalyst solution under nitrogen, stirred for 0.5 h. Then sulfur ylide salt **2** (52.0 mg, 0.2 mmol) and $LiOH \cdot H_2O$ (33.6 mg, 0.8 mmol) were quickly added to the system. The resulting solution was stirred at room temperature for another 12 hours. When the reaction was completed (monitored by TLC), the mixture was directly concentrated under reduced pressure. The resulting crude residue was purified via column chromatography on silica gel, to give the pure products **3a** in 69% yield (50.5 mg).

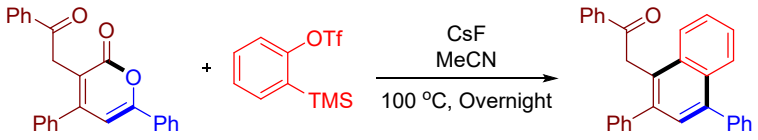
Products **3** or **4** were performed in the above procedure.

4. General Procedure for the Transformations of 3aa



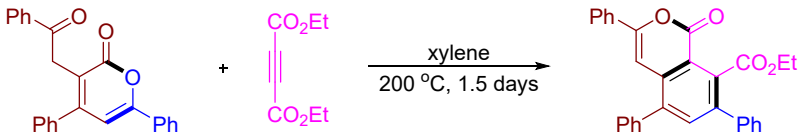
3a **5**, 82% yield

To a dry Schlenk tube with a magnetic stir bar, was added **3a** (36.6 mg, 0.10 mmol) and ultra-dry methanol (1.5 mL). Then, $NaBH_4$ (7.6 mg, 0.2 mmol) was added to the solution. The mixture was stirred at room temperature for 3 h until the **3a** was complete consumed (monitored by TLC). The mixture was directly concentrated under reduced pressure. The resulting crude residue was purified via column chromatography on silica gel (petroleum ether/ethyl acetate = 8:1) to afford the desired product **5** with 82% (30.2 mg) yield.



3a **6**, 75% yield

To a dry sealed tube with a magnetic stir bar, was added **3a** (36.6 mg, 0.10 mmol), CsF (19 mg, 0.12 mmol) and anhydrous MeCN (1.0 mL). Immediately afterwards, 2-(trimethylsilyl)phenyl trifluoromethanesulfonate (1.2 eq., 30 μL) was slowly added to the above system. After that, the reaction was stirred overnight at 100°C. After the reaction mixture was cooled to room temperature. The mixture was directly concentrated under reduced pressure. The resulting crude residue was purified via column chromatography on silica gel (petroleum ether/ethyl acetate = 100:1) to afford the desired product **6** with 75% (29.9 mg) yield.



3a **7**, 42% yield

To a dry sealed tube with a magnetic stir bar, a mixture of **3a** (36.6 mg, 0.10 mmol), diethyl but-2-ynedioate (85.0 mg, 0.50 mmol) in xylene (2 mL) was stirred at 200 °C for 1.5 days. After the reaction mixture was cooled to room temperature. The mixture was directly concentrated under reduced pressure. The resulting crude residue was purified via column chromatography on silica gel (petroleum ether/ethyl acetate = 18:1) to afford the desired product **7** with 42% (18.7 mg) yield.

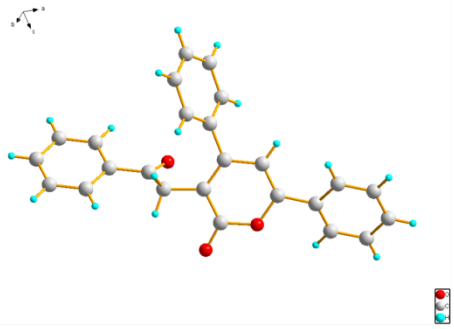
5. Reference

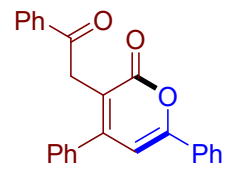
[1] R. Rossi, F. Bellina, C. Bechini, L. Mannina, P. Vergamini, *Tetrahedron* **1998**, *54*, 135.

[2] Y. Luo, K.-M. Qiu, X. Lu, K. Liu, J. Fu, H.-L. Zhu, *Bioorg. Med. Chem.* **2011**, *19*, 4730.

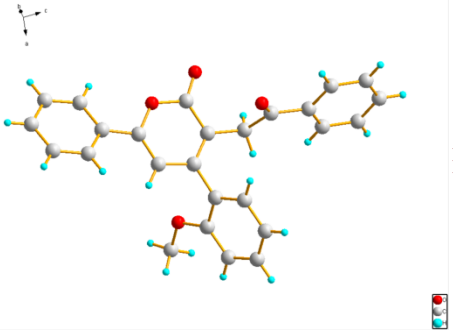
[3] P. Y. Ushakov, E. A. Khatuntseva, Y. V. Nelyubina, A. A. Tabolin, S. L. Ioffe, A. Y. Sukhorukov, *Adv. Synth. Catal.* **2019**, *361*, 5322.

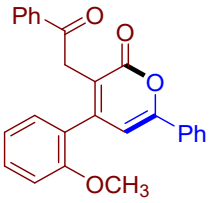
6. Crystal structures of 3a, 3e, 8 and 9



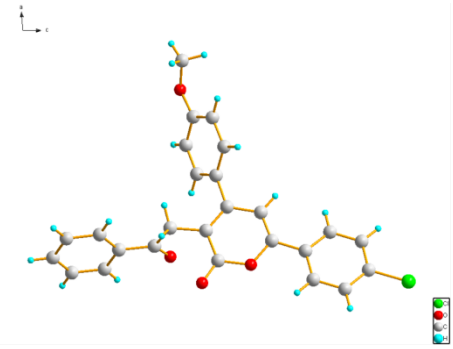


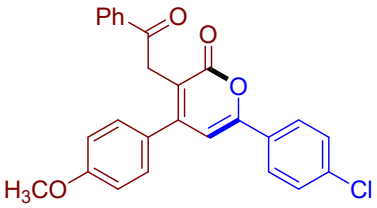
3a (CCDC: 2117411)



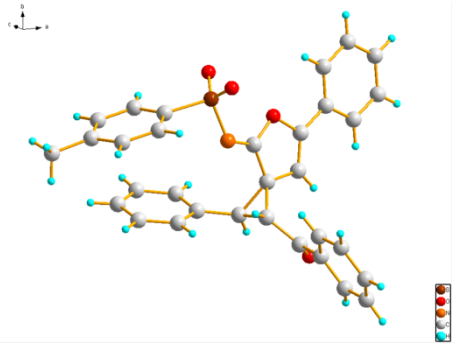


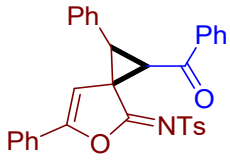
3e (CCDC: 2111301)





8 (CCDC: 2111107)





9 (CCDC: 2111140)

Crystal data and structure refinement for 3a.

CCDC number	2117411
Identification code	**3a**
Empirical formula	$C_{25}H_{18}O_3$
Formula weight	366.39
Temperature/K	160.00
Crystal system	triclinic
Space group	P-1
a/Å	9.8356(9)
b/Å	9.8517(9)
c/Å	11.1746(11)
α/°	63.911(3)
β/°	75.454(3)
γ/°	86.025(3)
Volume/Å^3	940.24(15)
Z	2
ρ_{calc}g/cm^3	1.294
μ/mm^{-1}	0.084
F(000)	384.0
Crystal size/mm^3	0.12 × 0.11 × 0.1
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.68 to 54.962
Index ranges	-12 ≤ h ≤ 12, -12 ≤ k ≤ 11, -14 ≤ l ≤ 14
Reflections collected	19071
Independent reflections	19071 [R_{int} = ?, R_{sigma} = 0.0545]
Data/restraints/parameters	19071/0/254
Goodness-of-fit on F^2	1.045
Final R indexes [I>=2σ (I)]	R_1 = 0.0552, wR_2 = 0.1474
Final R indexes [all data]	R_1 = 0.0775, wR_2 = 0.1652
Largest diff. peak/hole / e Å^{-3}	0.17/-0.15

Crystal data and structure refinement for 3e.

CCDC number	2111301
Identification code	**3e**
Empirical formula	$C_{26}H_{20}O_4$
Formula weight	396.42
Temperature/K	304.00
Crystal system	triclinic
Space group	P-1
a/Å	9.5757(4)
b/Å	10.4391(5)
c/Å	11.0084(5)
α/°	103.768(2)
β/°	99.482(2)
γ/°	104.612(2)
Volume/Å^3	1004.22(8)
Z	2
ρ_{calc}g/cm^3	1.311
μ/mm^{-1}	0.088
F(000)	416.0
Crystal size/mm^3	0.12 × 0.11 × 0.1
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.846 to 54.992
Index ranges	$-12 \leq h \leq 12$, $-13 \leq k \leq 13$, $-14 \leq l \leq 14$
Reflections collected	21021
Independent reflections	4578 [R_{int} = 0.0263, R_{sigma} = 0.0200]
Data/restraints/parameters	4578/0/272
Goodness-of-fit on F^2	1.023
Final R indexes [I>=2σ (I)]	R_1 = 0.0412, wR_2 = 0.1013
Final R indexes [all data]	R_1 = 0.0492, wR_2 = 0.1073
Largest diff. peak/hole / e Å^{-3}	0.22/-0.14

Crystal data and structure refinement for 8.

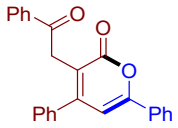
CCDC number	2111107
Identification code	**8**
Empirical formula	$C_{26}H_{19}ClO_4$
Formula weight	430.86
Temperature/K	303.00
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	11.1283(5)
b/Å	8.4980(3)
c/Å	23.7973(10)
α/°	90
β/°	94.591(2)
γ/°	90
Volume/Å^3	2243.25(16)
Z	4
ρ_{calc}g/cm^3	1.276
μ/mm^{-1}	0.200
F(000)	896.0
Crystal size/mm^3	0.13 × 0.12 × 0.11
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.092 to 54.976
Index ranges	$-14 \leq h \leq 14$, $-11 \leq k \leq 10$, $-30 \leq l \leq 30$
Reflections collected	34156
Independent reflections	5107 [R_{int} = 0.0404, R_{sigma} = 0.0246]
Data/restraints/parameters	5107/0/281
Goodness-of-fit on F^2	1.026
Final R indexes [I>=2σ (I)]	R_1 = 0.0454, wR_2 = 0.1028
Final R indexes [all data]	R_1 = 0.0687, wR_2 = 0.1179
Largest diff. peak/hole / e Å^{-3}	0.13/-0.21

Crystal data and structure refinement for 9.

CCDC number	2111140
Identification code	**9**
Empirical formula	$C_{32}H_{25}NO_4S$
Formula weight	519.59
Temperature/K	160.15
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	12.3788(8)
b/Å	21.0408(11)
c/Å	10.8677(7)
α/°	90
β/°	106.544(2)
γ/°	90
Volume/Å^3	2713.4(3)
Z	4
ρ_{calc}g/cm^3	1.272
μ/mm^{-1}	0.157
F(000)	1088.0
Crystal size/mm^3	0.12 × 0.11 × 0.1
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.814 to 54.964
Index ranges	$-15 \leq h \leq 16$, $-26 \leq k \leq 27$, $-13 \leq l \leq 14$
Reflections collected	25686
Independent reflections	6192 [R_{int} = 0.0488, R_{sigma} = 0.0405]
Data/restraints/parameters	6192/0/344
Goodness-of-fit on F^2	1.026
Final R indexes [I>=2σ (I)]	R_1 = 0.0508, wR_2 = 0.1183
Final R indexes [all data]	R_1 = 0.0864, wR_2 = 0.1401
Largest diff. peak/hole / e Å^{-3}	0.17/-0.27

7. Characterization of Products

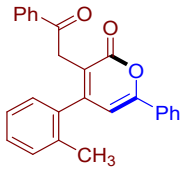
3-(2-Oxo-2-phenylethyl)-4,6-diphenyl-2H-pyran-2-one (3a)



Isolated in 69% yield (50.5 mg) as yellow solid, mp 165.2 – 168.2 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 7.5 Hz, 2H), 7.91 – 7.80 (m, 2H), 7.57 (t, *J* = 7.3 Hz, 1H), 7.50 – 7.34 (m, 10H), 6.73 (s, 1H), 4.17 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.98, 163.37, 157.89, 154.96, 137.60, 136.76, 133.40, 131.55, 130.76, 129.36, 129.04, 129.02, 128.70, 128.43, 127.54, 125.68, 118.14, 104.63, 38.43. HRMS (ESI) calcd. for $C_{25}H_{19}O_3$ [M+H$^+$]: 367.1329, found: 367.1328.

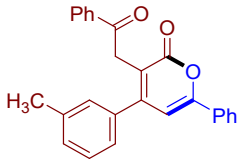
3-(2-Oxo-2-phenylethyl)-6-phenyl-4-(*o*-tolyl)-2H-pyran-2-one (3b)



Isolated in 73% yield (55.5 mg) as yellow solid, mp 145.5 – 147.1 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 – 7.80 (m, 4H), 7.55 – 7.49 (m, 1H), 7.48 – 7.37 (m, 5H), 7.31 – 7.26 (m, 2H), 7.23 – 7.13 (m, 2H), 6.61 (s, 1H), 4.15 (d, *J* = 17.1 Hz, 1H), 3.83 (d, *J* = 17.1 Hz, 1H), 2.26 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.15, 163.23, 157.82, 155.49, 136.98, 136.81, 134.69, 133.24, 131.52, 130.77, 130.75, 129.04, 128.61, 128.29, 127.53, 126.28, 125.66, 119.17, 104.47, 37.91, 19.83. HRMS (ESI) calcd. for $C_{26}H_{21}O_3$ [M+H$^+$]: 381.1485, found: 381.1463.

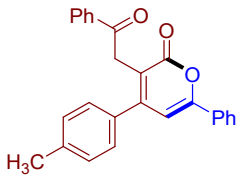
3-(2-Oxo-2-phenylethyl)-6-phenyl-4-(*m*-tolyl)-2H-pyran-2-one (3c)



Isolated in 60% yield (45.6 mg) as yellow solid, mp 165.4 – 168.6 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.5 Hz, 2H), 7.90 – 7.79 (m, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.49 – 7.38 (m, 5H), 7.29 (t, *J* = 7.7 Hz, 1H), 7.24 – 7.12 (m, 3H), 6.72 (s, 1H), 4.16 (s, 2H), 2.34 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.01, 163.39, 157.79, 155.07, 138.85, 137.58, 136.85, 133.32, 131.60, 130.70, 130.07, 129.02, 128.88, 128.68, 128.38, 128.09, 125.67, 124.60, 118.09, 104.68, 38.44, 21.53. HRMS (ESI) calcd. for $C_{26}H_{21}O_3$ [M+H$^+$]: 381.1485, found: 381.1507.

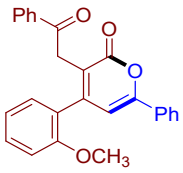
3-(2-Oxo-2-phenylethyl)-6-phenyl-4-(*p*-tolyl)-2H-pyran-2-one (3d)



Isolated in 67% yield (50.9 mg) as yellow solid, mp 157.7 – 159.1 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, J = 7.3 Hz, 2H), 7.89 – 7.80 (m, 2H), 7.56 (t, J = 7.4 Hz, 1H)., 7.49 – 7.39 (m, 5H), 7.30 – 7.18 (m, 4H), 6.72 (s, 1H), 4.18 (s, 2H), 2.37 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.04, 163.42, 157.75, 154.97, 139.49, 136.83, 134.70, 133.34, 131.63, 130.67, 129.66, 129.01, 128.68, 128.42, 127.50, 125.66, 117.93, 104.76, 38.52, 21.41. HRMS (ESI) calcd. for $C_{26}H_{21}O_3$ [M+H$^+$]: 381.1485, found: 381.1473.

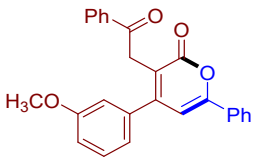
4-(2-Methoxyphenyl)-3-(2-oxo-2-phenylethyl)-6-phenyl-2H-pyran-2-one (3e)



Isolated in 56% yield (44.4 mg) as yellow solid, mp 138.0 – 142.5 ℃.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.92 (d, J = 7.6 Hz, 2H), 7.84 (d, J = 4.5 Hz, 2H), 7.54 (t, J = 7.2 Hz, 1H), 7.49 – 7.40 (m, 5H), 7.38 (t, J = 7.7 Hz, 1H), 7.23 (d, J = 7.2 Hz, 1H), 7.02 – 6.91 (m, 2H), 6.68 (s, 1H), 4.28 (d, J = 16.6 Hz, 1H), 3.84 (d, J = 16.7 Hz, 1H), 3.75 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.43, 163.40, 157.26, 155.86, 152.33, 136.94, 133.14, 131.78, 130.83, 130.48, 129.50, 128.94, 128.61, 128.28, 126.03, 125.64, 121.02, 119.60, 111.27, 105.47, 55.62, 38.60. HRMS (ESI) calcd. for $C_{26}H_{21}O_4$ [M+H$^+$]: 397.1434, found: 397.1408.

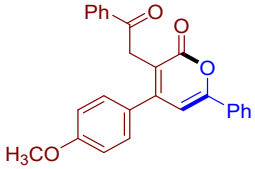
4-(3-Methoxyphenyl)-3-(2-oxo-2-phenylethyl)-6-phenyl-2H-pyran-2-one (3f)



Isolated in 61% yield (48.3mg) as yellow solid, mp 208.5 – 210.0 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, J = 7.5 Hz, 2H), 7.90 – 7.80 (m, 2H), 7.56 (t, J = 7.3 Hz, 1H), 7.50 – 7.39 (m, 5H), 7.32 (t, J = 7.8 Hz, 1H), 6.99 – 6.88 (m, 3H), 6.73 (s, 1H), 4.17 (s, 2H), 3.73 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.03, 163.33, 159.89, 157.90, 154.85, 138.92, 136.79, 133.39, 131.55, 130.75, 130.18, 129.04, 128.71, 128.41, 125.68, 119.72, 118.16, 115.24, 112.74, 104.52, 55.42, 38.50. HRMS (ESI) calcd. for $C_{26}H_{21}O_4$ [M+H$^+$]: 397.1434, found: 397.1409.

4-(4-Methoxyphenyl)-3-(2-oxo-2-phenylethyl)-6-phenyl-2H-pyran-2-one (3g)

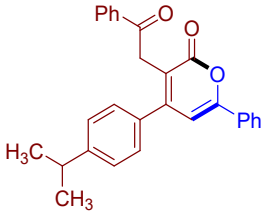


Isolated in 55% yield (43.6 mg) as yellow solid, mp 187.3 – 188.9 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, J = 7.3 Hz, 2H), 7.92 – 7.78 (m, 2H), 7.57 (t, J = 6.7 Hz, 1H)., 7.52 – 7.39 (m, 5H), 7.33 (d, J = 8.1 Hz, 2H), 6.93 (d, J = 8.1 Hz, 2H), 6.73 (s, 1H), 4.19 (s, 2H), 3.82

(s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.21, 163.49, 160.51, 157.69, 154.63, 136.86, 133.38, 131.67, 130.67, 129.84, 129.12, 129.02, 128.71, 128.45, 125.67, 117.69, 114.41, 104.83, 55.50, 38.62. HRMS (ESI) calcd. for $C_{26}H_{21}O_4$ [M+H$^+$]: 397.1434, found: 397.1427.

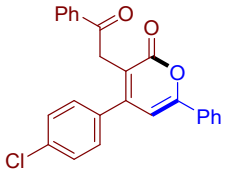
4-(4-Isopropylphenyl)-3-(2-oxo-2-phenylethyl)-6-phenyl-2H-pyran-2-one (3h)



Isolated in 58% yield (47.3 mg) as yellow solid, mp 168.4 – 168.9 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, J = 7.6 Hz, 2H), 7.91 – 7.77 (m, 2H), 7.56 (t, J = 7.1 Hz, 1H), 7.51 – 7.38 (m, 5H), 7.35 – 7.20 (m, 4H), 6.74 (s, 1H), 4.19 (s, 2H), 3.06 – 2.81 (m, 1H), 1.26 (s, 3H), 1.24 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.10, 163.46, 157.69, 154.95, 150.34, 136.85, 135.00, 133.34, 131.63, 130.65, 129.00, 128.67, 128.45, 127.62, 127.07, 125.64, 117.93, 104.81, 38.51, 34.07, 23.95. HRMS (ESI) calcd. for $C_{28}H_{25}O_3$ [M+H$^+$]: 409.1798, found: 409.1776.

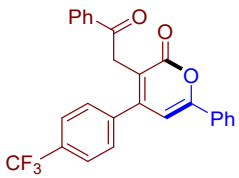
4-(4-Chlorophenyl)-3-(2-oxo-2-phenylethyl)-6-phenyl-2H-pyran-2-one (3i)



Isolated in 49% yield (39.2 mg) as yellow solid, mp 168.1 – 168.9 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, J = 7.5 Hz, 2H), 7.90 – 7.80 (m, 2H), 7.58 (t, J = 7.4 Hz, 1H), 7.51 – 7.43 (m, 5H), 7.40 (d, J = 8.4 Hz, 2H), 7.33 (d, J = 8.4 Hz, 2H), 6.68 (s, 1H), 4.14 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.88, 163.11, 158.16, 153.80, 136.65, 135.98, 135.60, 133.55, 131.41, 130.91, 129.32, 129.08, 128.98, 128.77, 128.44, 125.71, 118.37, 104.21, 38.35. HRMS (ESI) calcd. for $C_{25}H_{18}ClO_3$ [M+H$^+$]: 401.0939, found: 401.0964.

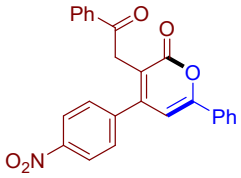
3-(2-Oxo-2-phenylethyl)-6-phenyl-4-(4-(trifluoromethyl)phenyl)-2H-pyran-2-one (3j)



Isolated in 40% yield (34.7 mg) as yellow solid, mp 168.3 – 169.3 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 – 7.94 (m, 2H), 7.89 – 7.82 (m, 2H), 7.69 (d, J = 8.1 Hz, 2H), 7.58 (t, J = 7.4 Hz, 1H), 7.53 (d, J = 8.0 Hz, 2H), 7.49 – 7.43 (m, 5H), 6.69 (s, 1H), 4.12 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.71, 162.94, 158.41, 153.56, 141.15, 136.56, 133.63, 131.49 (q, J = 32.9 Hz), 131.29, 131.03, 129.13, 128.79, 128.45, 128.06, 126.07 (q, J = 3.7 Hz), 125.73, 123.86 (q, J = 272.4 Hz), 118.70, 103.91, 38.21. HRMS (ESI) calcd. for $C_{26}H_{18}F_3O_3$ [M+H$^+$]: 435.1203, found: 435.1184.

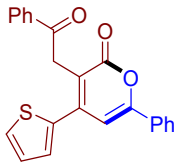
4-(4-Nitrophenyl)-3-(2-oxo-2-phenylethyl)-6-phenyl-2H-pyran-2-one (3k)



Isolated in 28% yield (23.0 mg) as brown solid, mp 222.5 – 224.3 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 – 8.26 (m, 2H), 8.00 – 7.93 (m, 2H), 7.89 – 7.83 (m, 2H), 7.63 – 7.55 (m, 3H)., 7.52 – 7.43 (m, 5H), 6.68 (s, 1H), 4.11 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.52, 162.70, 158.74, 152.78, 148.39, 143.89, 136.41, 133.78, 131.22, 131.13, 129.18, 128.86, 128.78, 128.46, 125.78, 124.32, 118.88, 103.40, 38.14. HRMS (ESI) calcd. for $C_{25}H_{18}NO_5$ [M+H$^+$]: 412.1179, found: 412.1162.

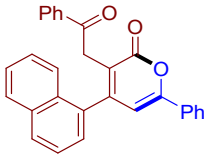
3-(2-Oxo-2-phenylethyl)-6-phenyl-4-(thiophen-2-yl)-2H-pyran-2-one (3l)



Isolated in 52% yield (38.8 mg) as brown solid, mp 139.2 – 141.3 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 7.4 Hz, 2H), 7.92 – 7.80 (m, 2H), 7.60 (t, *J* = 7.4 Hz, 1H), 7.55 – 7.39 (m, 6H), 7.26 – 7.23 (m, 1H), 7.13 – 7.03 (m, 1H), 6.85 (s, 1H), 4.45 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.60, 163.40, 157.72, 155.83, 147.19, 138.41, 136.72, 133.51, 131.49, 130.85, 129.06, 129.03, 128.79, 128.53, 128.10, 125.74, 117.36, 104.41, 39.01. HRMS (ESI) calcd. for $C_{23}H_{17}O_3S$ [M+H$^+$]: 373.0893, found: 373.0875.

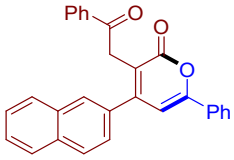
4-(Naphthalen-1-yl)-3-(2-oxo-2-phenylethyl)-6-phenyl-2H-pyran-2-one (3m)



Isolated in 42% yield (35.0 mg) as yellow solid, mp 74.2 – 75.9 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 – 7.87 (m, 2H), 7.86 – 7.82 (m, 2H), 7.82 – 7.75 (m, 3H), 7.57 – 7.50 (m, 2H), 7.49 – 7.40 (m, 6H), 7.39 – 7.32 (m, 2H), 6.73 (s, 1H), 4.22 (d, *J* = 17.1 Hz, 1H), 3.77 (d, *J* = 17.1 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.33, 163.17, 157.66, 154.33, 136.72, 134.76, 133.77, 133.22, 131.48, 130.79, 130.05, 129.47, 129.05, 128.74, 128.56, 128.27, 127.20, 126.69, 125.73, 125.58, 125.49, 125.22, 120.22, 105.30, 38.34. HRMS (ESI) calcd. for $C_{29}H_{21}O_3$ [M+H$^+$]: 417.1485, found: 417.1472.

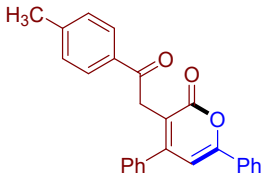
4-(Naphthalen-2-yl)-3-(2-oxo-2-phenylethyl)-6-phenyl-2H-pyran-2-one (3n)



Isolated in 41% yield (34.1 mg) as yellow solid, mp 181.7 – 183.6 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 – 7.92 (m, 2H), 7.92 – 7.84 (m, 5H), 7.84 – 7.78 (m, 1H), 7.57 – 7.49 (m, 3H), 7.49 – 7.39 (m, 6H), 6.83 (s, 1H), 4.21 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.07, 163.38, 157.96, 154.98, 136.83, 134.99, 133.39, 133.09, 131.60, 130.79, 129.07, 128.93, 128.70, 128.49, 128.42, 127.93, 127.25, 127.10, 127.05, 125.74, 124.98, 118.42, 104.74, 38.55. HRMS (ESI) calcd. for $C_{29}H_{21}O_3$ [M+H$^+$]: 417.1485, found: 417.1455.

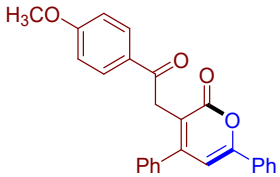
3-(2-Oxo-2-(*p*-tolyl)ethyl)-4,6-diphenyl-2H-pyran-2-one (3o)



Isolated in 54% yield (41.1 mg) as yellow solid, mp 155.7 – 157.3 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 – 7.76 (m, 4H), 7.52 – 7.31 (m, 8H), 7.30 – 7.17 (m, 2H), 6.72 (s, 1H), 4.14 (s, 2H), 2.40 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.52, 163.36, 157.80, 154.82, 144.19, 137.63, 134.28, 131.57, 130.70, 129.35, 129.30, 129.01, 128.97, 128.53, 127.54, 125.65, 118.30, 104.62, 38.30, 21.80. HRMS (ESI) calcd. for $C_{26}H_{21}O_3$ [M+H$^+$]: 381.1485, found: 381.1456.

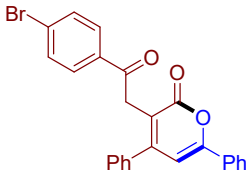
3-(2-(4-Methoxyphenyl)-2-oxoethyl)-4,6-diphenyl-2H-pyran-2-one (3p)



Isolated in 60% yield (47.5 mg) as yellow solid, mp 158.2 – 160.5 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, J = 8.8 Hz, 2H), 7.89 – 7.79 (m, 2H), 7.50 – 7.33 (m, 8H), 6.91 (d, J = 8.8 Hz, 2H), 6.72 (s, 1H), 4.11 (s, 2H), 3.85 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 195.39, 163.71, 163.37, 157.72, 154.75, 137.61, 131.55, 130.69, 130.66, 129.79, 129.27, 128.98, 128.94, 127.56, 125.62, 118.38, 113.79, 104.62, 55.58, 38.02. HRMS (ESI) calcd. for $C_{26}H_{21}O_4$ [M+H$^+$]: 397.1434, found: 397.1395.

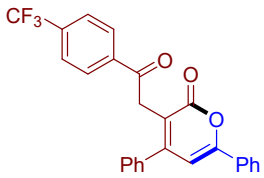
3-(2-(4-Bromophenyl)-2-oxoethyl)-4,6-diphenyl-2H-pyran-2-one (3q)



Isolated in 48% yield (42.6 mg) as yellow solid, mp 98.8 – 101.0 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 – 7.75 (m, 4H), 7.59 (d, *J* = 8.3 Hz, 2H), 7.50 – 7.40 (m, 6H), 7.39 – 7.31 (m, 2H), 6.73 (s, 1H), 4.11 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.08, 163.31, 158.03, 155.11, 137.51, 135.51, 132.02, 131.47, 130.83, 129.95, 129.44, 129.06, 128.59, 127.50, 125.70, 117.82, 104.60, 38.31. HRMS (ESI) calcd. for $C_{25}H_{18}BrO_3$ [M+H$^+$]: 445.0434, found: 445.0392.

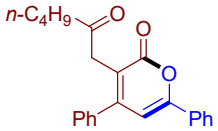
3-(2-Oxo-2-(4-(trifluoromethyl)phenyl)ethyl)-4,6-diphenyl-2H-pyran-2-one (3r)



Isolated in 50% yield (43.4 mg) as yellow solid, mp 60.9 – 62.8 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 8.1 Hz, 2H), 7.90 – 7.82 (m, 2H), 7.72 (d, *J* = 8.2 Hz, 2H), 7.48 – 7.41 (m, 6H), 7.40 – 7.35 (m, 2H), 6.75 (s, 1H), 4.16 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.28, 163.29, 158.14, 155.28, 139.50, 137.45, 134.64 (q, *J* = 32.8 Hz), 131.42, 130.90, 129.51, 129.11, 129.08, 128.75, 127.47, 125.80 (q, *J* = 3.7 Hz), 125.71, 123.70 (q, *J* = 272.7 Hz), 117.59, 104.58, 38.62. HRMS (ESI) calcd. for $C_{26}H_{18}F_3O_3$ [M+H$^+$]: 435.1203, found: 435.1189.

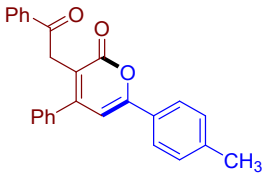
3-(2-Oxohexyl)-4,6-diphenyl-2H-pyran-2-one (3s)



Isolated in 47% yield (32.5 mg) as yellow solid, mp 51.5 – 53.1 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 – 7.79 (m, 2H), 7.50 – 7.40 (m, 6H), 7.39 – 7.32 (m, 2H), 6.69 (s, 1H), 3.56 (s, 2H), 2.53 (t, *J* = 7.4 Hz, 2H), 1.66 – 1.53 (m, 2H), 1.36 – 1.26 (m, 2H), 0.90 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 208.06, 163.39, 157.73, 154.75, 137.58, 131.51, 130.75, 129.35, 129.03, 128.95, 127.58, 125.64, 118.17, 104.56, 43.13, 41.85, 25.99, 22.39, 13.99. HRMS (ESI) calcd. for $C_{23}H_{23}O_3$ [M+H$^+$]: 347.1642, found: 347.1636.

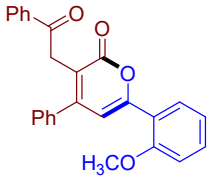
3-(2-Oxo-2-phenylethyl)-4-phenyl-6-(*p*-tolyl)-2H-pyran-2-one (4a)



Isolated in 57% yield (43.3 mg) as yellow solid, mp 174.3 – 176.5 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.93 (m, 2H), 7.78 – 7.71 (m, 2H), 7.60 – 7.51 (m, 1H), 7.48 – 7.32 (m, 7H), 7.27 – 7.23 (m, 2H), 6.68 (s, 1H), 4.15 (s, 2H), 2.40 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.03, 163.45, 158.16, 155.10, 141.20, 137.73, 136.83, 133.34, 129.76, 129.29, 128.98, 128.82, 128.68, 128.41, 127.53, 125.62, 117.58, 103.99, 38.39, 21.58. HRMS (ESI) calcd. for $C_{26}H_{21}O_3$ [M+H$^+$]: 381.1485, found: 381.1493.

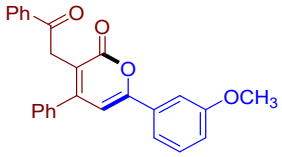
6-(2-Methoxyphenyl)-3-(2-oxo-2-phenylethyl)-4-phenyl-2H-pyran-2-one (4b)



Isolated in 55% yield (43.6 mg) as yellow solid, mp 173.9 – 175.1 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 – 7.92 (m, 3H), 7.60 – 7.52 (m, 1H), 7.49 – 7.34 (m, 8H), 7.17 (s, 1H), 7.07 (td, *J* = 7.9, 1.0 Hz, 1H), 6.99 (dd, *J* = 8.3, 0.5 Hz, 1H), 4.16 (s, 2H), 3.89 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.13, 163.66, 157.49, 155.29, 154.83, 138.12, 136.89, 133.31, 131.58, 129.24, 129.12, 128.91, 128.67, 128.43, 127.66, 121.08, 120.38, 117.82, 111.55, 109.66, 55.77, 38.39. HRMS (ESI) calcd. for $C_{26}H_{21}O_4$ [M+H$^+$]: 397.1434, found: 397.1413.

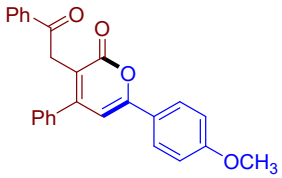
6-(3-Methoxyphenyl)-3-(2-oxo-2-phenylethyl)-4-phenyl-2H-pyran-2-one (4c)



Isolated in 62% yield (49.1 mg) as yellow solid, mp 137.1 – 138.5 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 – 7.92 (m, 2H), 7.59 – 7.53 (m, 1H), 7.48 – 7.32 (m, 10H), 6.99 (ddd, *J* = 8.1, 2.6, 1.0 Hz, 1H), 6.72 (s, 1H), 4.16 (s, 2H), 3.86 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.94, 163.30, 160.17, 157.72, 154.90, 137.59, 136.80, 133.38, 132.94, 130.06, 129.36, 129.02, 128.70, 128.41, 127.54, 118.29, 118.13, 116.95, 110.69, 104.90, 55.64, 38.43. HRMS (ESI) calcd. for $C_{26}H_{21}O_4$ [M+H$^+$]: 397.1434, found: 397.1448.

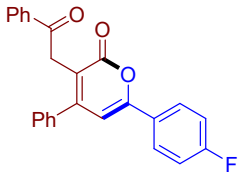
6-(4-Methoxyphenyl)-3-(2-oxo-2-phenylethyl)-4-phenyl-2H-pyran-2-one (4d)



Isolated in 58% yield (46.0 mg) as yellow solid, mp 172.6 – 173.5 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.0 Hz, 2H), 7.80 (d, *J* = 8.0 Hz, 2H), 7.61 – 7.51 (m, 1H), 7.50 – 7.31 (m, 7H), 6.95 (d, *J* = 8.1 Hz, 2H), 6.62 (s, 1H), 4.14 (s, 2H), 3.86 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.09, 163.51, 161.73, 158.06, 155.27, 137.83, 136.85, 133.32, 129.25, 128.96, 128.67, 128.40, 127.52, 127.35, 124.14, 116.81, 114.46, 103.23, 55.56, 38.35. HRMS (ESI) calcd. for $C_{26}H_{20}NaO_4$ [M+Na$^+$]: 419.1254, found: 419.1215.

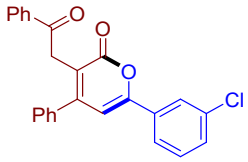
6-(4-Fluorophenyl)-3-(2-oxo-2-phenylethyl)-4-phenyl-2H-pyran-2-one (4e)



Isolated in 60% yield (46.1 mg) as yellow solid, mp 139.5 – 142.3 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 – 7.93 (m, 2H), 7.89 – 7.80 (m, 2H), 7.60 – 7.53 (m, 1H), 7.50 – 7.33 (m, 7H), 7.19 – 7.09 (m, 2H), 6.67 (s, 1H), 4.16 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.90, 164.24 (d, J = 251.9 Hz), 163.15, 156.94, 154.92, 137.51, 136.74, 133.40, 129.40, 129.03, 128.70, 128.40, 127.86, 127.79 (d, J = 8.6 Hz), 127.49, 118.06, 116.23 (d, J = 22.1 Hz), 104.37 (d, J = 0.8 Hz), 38.38. HRMS (ESI) calcd. for $C_{25}H_{18}FO_3$ [M+H$^+$]: 347.1234, found: 385.1268.

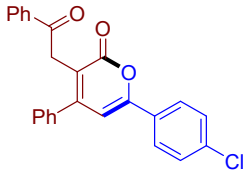
6-(3-Chlorophenyl)-3-(2-oxo-2-phenylethyl)-4-phenyl-2H-pyran-2-one (4f)



Isolated in 53% yield (42.4 mg) as yellow solid, mp 168.5 – 168.9 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 – 7.93 (m, 2H), 7.84 (d, J = 1.6 Hz, 1H), 7.73 (dt, J = 7.0, 1.6 Hz, 1H), 7.60 – 7.53 (m, 1H), 7.49 – 7.33 (m, 9H), 6.73 (s, 1H), 4.17 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.81, 162.95, 156.25, 154.68, 137.34, 136.73, 136.70, 135.27, 133.46, 133.44, 133.30, 133.29, 130.68, 130.34, 129.49, 129.08, 128.72, 128.42, 127.51, 125.70, 123.73, 119.04, 105.32, 38.45. HRMS (ESI) calcd. for $C_{25}H_{18}ClO_3$ [M+H$^+$]: 401.0939, found: 401.0903.

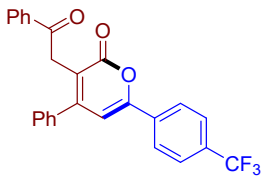
6-(4-Chlorophenyl)-3-(2-oxo-2-phenylethyl)-4-phenyl-2H-pyran-2-one (4g)



Isolated in 49% yield (39.2 mg) as yellow solid, mp 136.3 – 137.2 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, J = 7.5 Hz, 2H), 7.79 (d, J = 8.2 Hz, 2H), 7.56 (t, J = 7.0 Hz, 1H), 7.50 – 7.32 (m, 9H), 6.71 (s, 1H), 4.16 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.86, 163.06, 156.72, 154.81, 137.43, 136.87, 136.73, 133.44, 130.04, 129.45, 129.36, 129.06, 128.72, 128.42, 127.50, 126.92, 118.57, 104.81, 38.42. HRMS (ESI) calcd. for $C_{25}H_{18}ClO_3$ [M+H$^+$]: 401.0939, found: 401.0902.

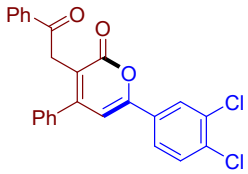
3-(2-Oxo-2-phenylethyl)-4-phenyl-6-(4-(trifluoromethyl)phenyl)-2H-pyran-2-one (4h)



Isolated in 45% yield (39.1 mg) as yellow solid, mp 142.6 – 145.1 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 7.7 Hz, 4H), 7.72 (d, *J* = 8.0 Hz, 2H), 7.58 (t, *J* = 7.0 Hz, 1H), 7.51 – 7.33 (m, 7H), 6.81 (s, 1H), 4.18 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.76, 162.85, 156.03, 154.56, 137.22, 136.66, 134.79, 133.52, 132.27 (q, *J* = 32.8 Hz), 129.56, 129.12, 128.75, 128.43, 127.51, 126.06 (q, *J* = 3.6 Hz), 125.91, 123.86 (q, *J* = 272.3 Hz), 119.63, 105.97, 38.49. HRMS (ESI) calcd. for $C_{26}H_{17}F_3NaO_3$ [M+Na$^+$]: 457.1022, found: 457.0957.

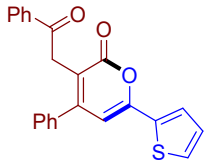
6-(3,4-Dichlorophenyl)-3-(2-oxo-2-phenylethyl)-4-phenyl-2H-pyran-2-one (4i)



Isolated in 56% yield (48.6 mg) as yellow solid, mp 168.2 – 169.1 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.92 (m, 3H), 7.68 (dd, *J* = 8.5, 2.2 Hz, 1H), 7.61 – 7.54 (m, 1H), 7.52 (d, *J* = 8.5 Hz, 1H), 7.49 – 7.39 (m, 5H), 7.39 – 7.33 (m, 2H), 6.72 (s, 1H), 4.16 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.71, 162.72, 155.30, 154.58, 137.18, 136.64, 134.91, 133.65, 133.49, 131.42, 131.10, 129.55, 129.10, 128.73, 128.40, 127.48, 127.39, 124.63, 119.30, 105.43, 38.47. HRMS (ESI) calcd. for $C_{25}H_{17}Cl_2O_3$ [M+H$^+$]: 435.0549, found: 435.0543.

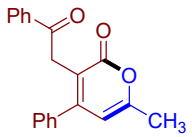
3-(2-Oxo-2-phenylethyl)-4-phenyl-6-(thiophen-2-yl)-2H-pyran-2-one (4j)



Isolated in 65% yield (48.4 mg) as yellow solid, mp 189.5 – 190.7 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.92 (m, 2H), 7.61 (dd, *J* = 3.8, 1.2 Hz, 1H), 7.59 – 7.52 (m, 1H), 7.48 – 7.38 (m, 6H), 7.38 – 7.32 (m, 2H), 7.13 – 7.08 (m, 1H), 6.55 (s, 1H), 4.13 (s, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.92, 162.69, 155.03, 153.65, 137.39, 136.77, 135.24, 133.36, 129.38, 129.01, 128.68, 128.66, 128.43, 128.38, 127.48, 127.31, 117.56, 103.70, 38.43. HRMS (ESI) calcd. for $C_{23}H_{17}O_3S$ [M+H$^+$]: 373.0893, found: 373.0887.

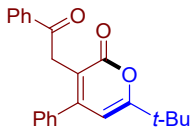
6-Methyl-3-(2-oxo-2-phenylethyl)-4-phenyl-2H-pyran-2-one (4k)



Isolated in 54% yield (32.8 mg) as yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 – 7.84 (m, 2H), 7.48 (t, *J* = 7.4 Hz, 1H), 7.37 (t, *J* = 7.6 Hz, 2H), 7.33 – 7.27 (m, 3H), 7.26 – 7.20 (m, 2H), 5.99 (s, 1H), 4.02 (s, 2H), 2.23 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.08, 164.12, 159.49, 154.89, 137.47, 136.78, 133.35, 129.24, 128.91, 128.68, 128.40, 127.46, 116.72, 106.82, 38.20, 19.93. HRMS (ESI) calcd. for $C_{20}H_{17}O_3$ [M+H$^+$]: 305.1172, found: 305.1187.

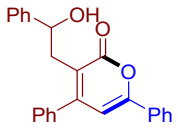
6-(*tert*-Butyl)-3-(2-oxo-2-phenylethyl)-4-phenyl-2H-pyran-2-one (4l)



Isolated in 59% yield (40.8 mg) as yellow solid, mp 98.5 – 99.7 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 – 7.91 (m, 2H), 7.59 – 7.51 (m, 1H), 7.47 – 7.41 (m, 2H), 7.41 – 7.36 (m, 3H), 7.34 – 7.30 (m, 2H), 6.08 (s, 1H), 4.09 (s, 2H), 1.32 (s, 9H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.15, 169.93, 164.00, 154.81, 137.94, 136.87, 133.31, 129.16, 128.90, 128.66, 128.40, 127.52, 116.82, 102.79, 38.21, 36.14, 28.17. HRMS (ESI) calcd. for $C_{23}H_{23}O_3$ [M+H$^+$]: 347.1642, found: 347.1652.

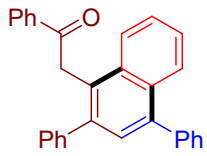
3-(2-Hydroxy-2-phenylethyl)-4,6-diphenyl-2H-pyran-2-one (5)



Isolated in 82% yield (30.2 mg) as white solid, mp 169.4 – 170.2 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 – 7.80 (m, 2H), 7.50 – 7.39 (m, 6H), 7.29 – 7.17 (m, 7H), 6.64 (s, 1H), 5.10 (dd, J = 8.4, 4.0 Hz, 1H), 3.43 (s, 1H), 2.99 (dd, J = 14.0, 8.6 Hz, 1H), 2.89 (dd, J = 14.0, 4.2 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 165.38, 157.35, 155.52, 144.50, 137.66, 131.26, 130.86, 129.09, 128.98, 128.78, 128.44, 127.85, 127.43, 125.66, 125.61, 120.76, 105.26, 73.79, 38.15. HRMS (ESI) calcd. for $C_{25}H_{21}O_3$ [M+H$^+$]: 369.1485, found: 369.1479.

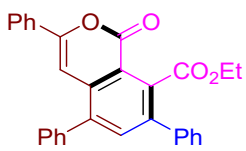
2-(2,4-Diphenylnaphthalen-1-yl)-1-phenylethan-1-one (6)



Isolated in 75% yield (29.9 mg) as white solid, mp 53.4 – 55.2 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 – 8.01 (m, 2H), 7.98 (dd, J = 8.4, 1.0 Hz, 1H), 7.74 (d, J = 8.0 Hz, 1H), 7.65 – 7.58 (m, 1H), 7.58 – 7.53 (m, 2H), 7.53 – 7.39 (m, 10H), 7.39 – 7.29 (m, 3H), 4.77 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.36, 142.11, 140.74, 140.41, 139.74, 136.85, 133.47, 133.09, 131.41, 130.38, 129.40, 129.32, 128.87, 128.45, 128.43, 128.38, 127.99, 127.42, 127.41, 127.06, 126.73, 125.74, 124.81, 40.78. HRMS (ESI) calcd. for $C_{30}H_{23}O$ [M+H$^+$]: 399.1743, found: 399.1778.

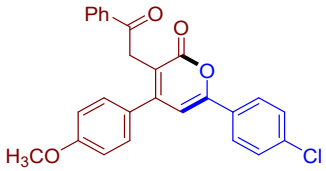
Ethyl 1-oxo-3,5,7-triphenyl-1H-isochromene-8-carboxylate (7)



Isolated in 42% yield (18.7 mg) as yellow solid, mp 157.3 – 159.9 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 – 7.72 (m, 2H), 7.70 (s, 1H), 7.57 – 7.37 (m, 13H), 7.04 (s, 1H), 4.31 (q, *J* = 7.2 Hz, 2H), 1.21 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 168.25, 160.85, 153.89, 140.14, 139.50, 138.59, 138.17, 137.41, 134.98, 134.64, 131.78, 130.31, 129.73, 129.08, 128.99, 128.97, 128.52, 128.49, 128.34, 125.48, 117.83, 99.43, 61.94, 13.96. HRMS (ESI) calcd. for $C_{30}H_{23}O_4$ [M+H$^+$]: 447.1591, found: 447.1586.

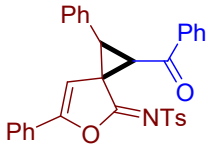
6-(4-Chlorophenyl)-4-(4-methoxyphenyl)-3-(2-oxo-2-phenylethyl)-2H-pyran-2-one (8)



Isolated in 54% yield (46.5 mg) as yellow solid, mp 185.9 – 189.5 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 – 7.95 (m, 2H), 7.82 – 7.75 (m, 2H), 7.61 – 7.54 (m, 1H), 7.50 – 7.39 (m, 4H), 7.34 – 7.28 (m, 2H), 6.97 – 6.89 (m, 2H), 6.70 (s, 1H), 4.19 (s, 2H), 3.82 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.10, 163.20, 160.57, 156.50, 154.49, 136.79, 136.77, 133.43, 130.13, 129.63, 129.33, 129.09, 128.72, 128.44, 126.90, 118.06, 114.45, 105.01, 55.50, 38.63. HRMS (ESI) calcd. for $C_{26}H_{20}ClO_4$ [M+H$^+$]: 431.1045, found: 431.1039.

N-(1-Benzoyl-2,6-diphenyl-5-oxaspiro[2.4]hept-6-en-4-ylidene)-4-methylbenzenesulfonamide (9)



Isolated in 65% yield (67.8 mg) as yellow solid, mp 165.1 – 168.2 ℃.

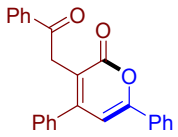
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 7.5 Hz, 2H), 7.65 (t, *J* = 7.4 Hz, 1H), 7.61 – 7.49 (m, 4H), 7.42 – 7.33 (m, 5H), 7.32 – 7.24 (m, 5H), 7.16 (d, *J* = 8.1 Hz, 2H), 6.26 (s, 1H), 4.29 (d, *J* = 8.1 Hz, 1H), 4.07 (d, *J* = 8.1 Hz, 1H), 2.39 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 194.15, 169.23, 155.16, 143.31, 138.25, 136.59, 134.37, 131.00, 130.02, 129.90, 129.16, 129.11, 128.88, 128.68, 128.38, 128.05, 127.43, 127.16, 124.91, 101.94, 46.80, 43.40, 40.15, 21.70. HRMS (ESI) calcd. for $C_{32}H_{26}NO_4S$ [M+H$^+$]: 520.1577, found: 520.1574.

The diastereomer of **9**

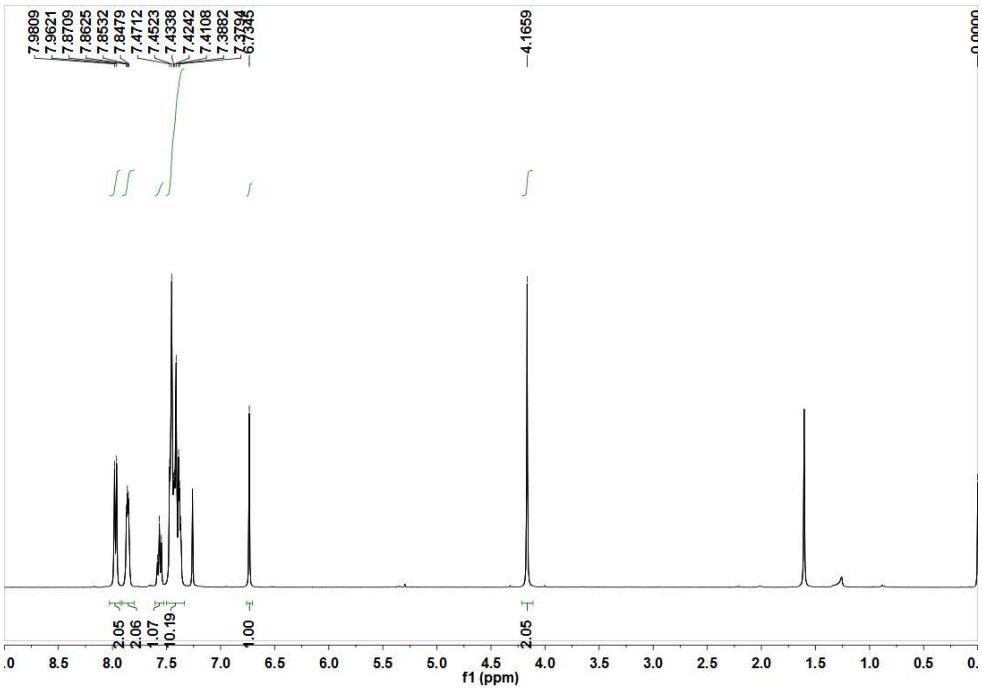
Isolated in 25% yield (26.0 mg) as yellow solid, mp 164.3 – 167.5 ℃.

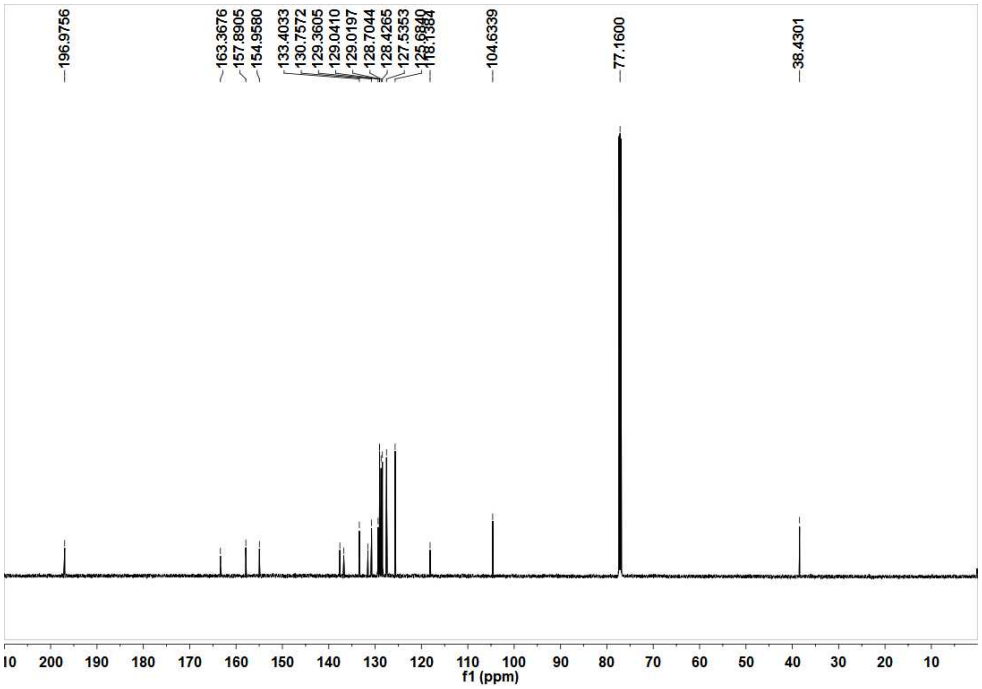
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 – 7.76 (m, 2H), 7.62 – 7.54 (m, 5H), 7.44 – 7.36 (m, 8H), 7.35 – 7.31 (m, 2H), 7.14 (d, *J* = 8.0 Hz, 2H), 5.63 (s, 1H), 4.03 (d, *J* = 8.6 Hz, 1H), 3.80 (d, *J* = 8.6 Hz, 1H), 2.40 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 189.90, 169.41, 156.20, 143.16, 138.25, 136.30, 134.28, 133.72, 130.29, 129.20, 129.07, 129.03, 128.92, 128.50, 128.35, 128.25, 127.27, 127.03, 125.12, 100.76, 44.78, 42.23, 40.06, 21.70. HRMS (ESI) calcd. for $C_{32}H_{26}NO_4S$ [M+H$^+$]: 520.1577, found: 520.1541.

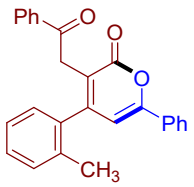
8. Spectra of the New Compounds



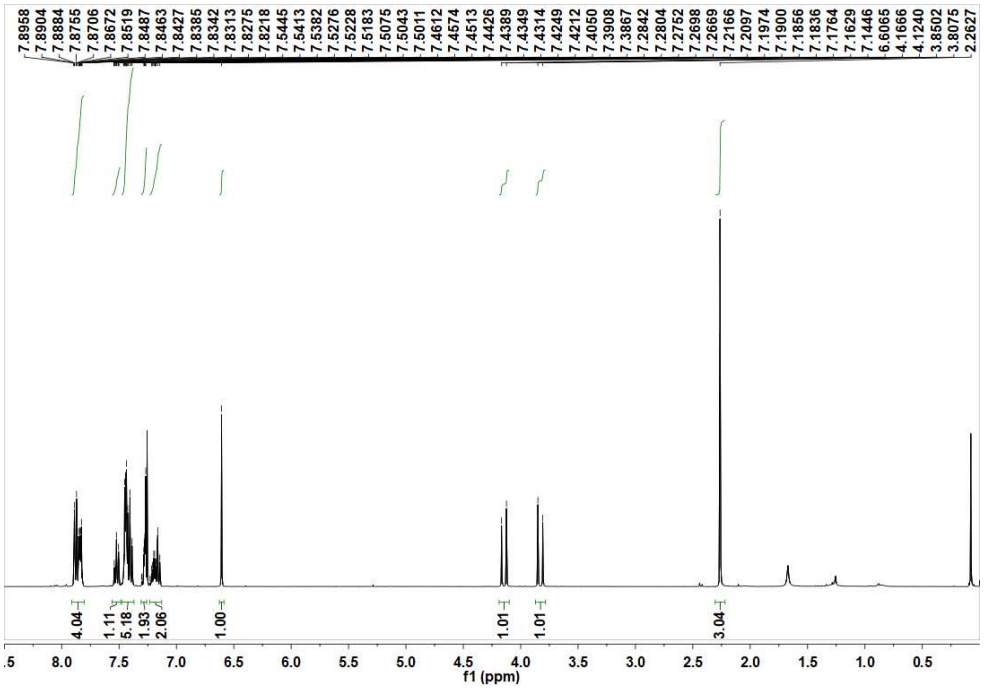
3a






Ph
O
O
O
Ph
CH3

3b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3.8075
2.2627
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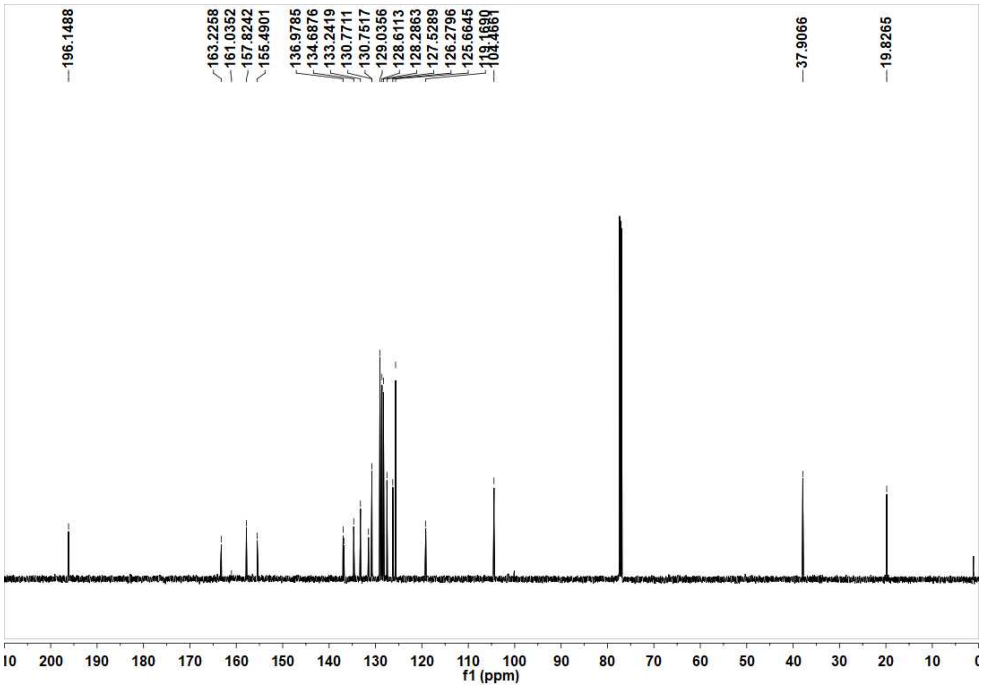
2.06
1.00
1.01
1.01
3.04
f1 (ppm)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157.8242
155.4901
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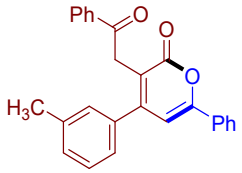
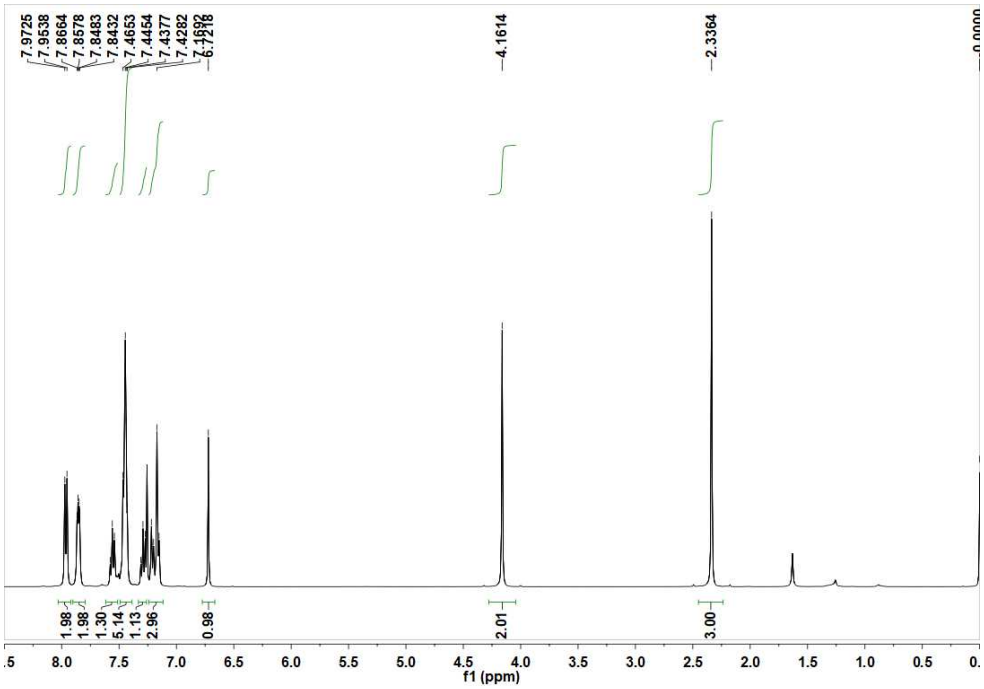
134.6876
133.2419
130.7711
130.7517
129.0356
128.6113
128.2863
127.5289
126.2796
126.6645
104.4681
37.9066
19.8265
f1 (ppm)


Ph
O
O
O
H3C
Ph

3c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7.8664
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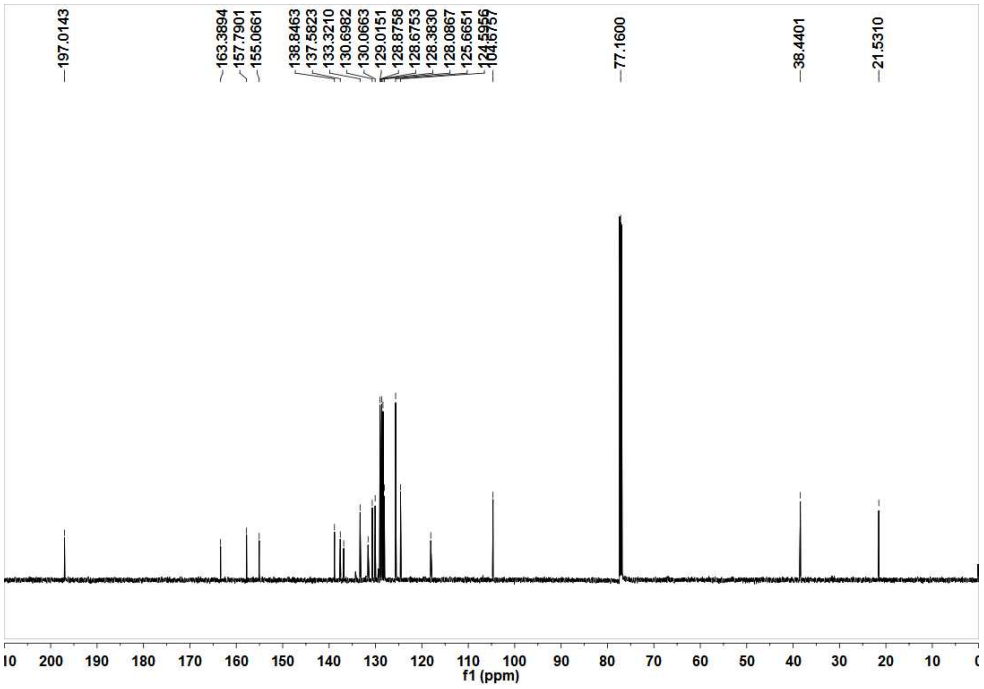
7.8483
7.8432
7.4653
7.4454
7.4377
7.4282
4.1614
2.3364
1.98
1.98
1.30
5.14
1.13
2.96
0.98
2.01
3.00
f1 (ppm)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155.0661
138.8463
137.5823
133.3210
130.6982
130.0663
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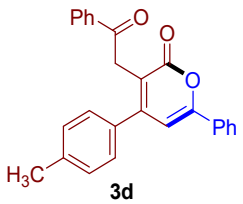
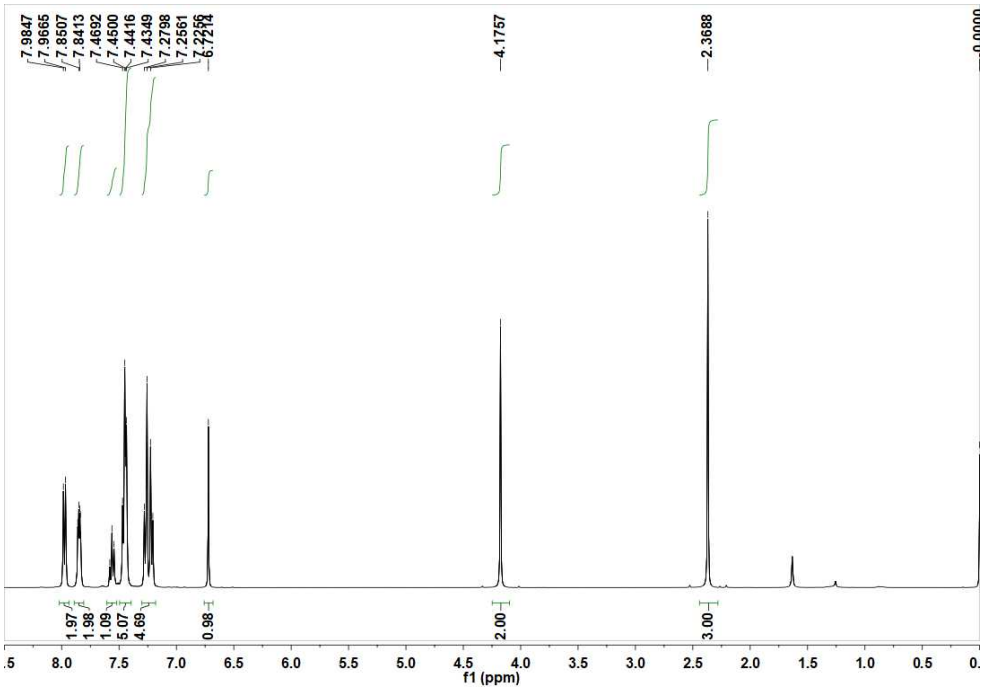
128.6753
128.3830
128.0867
125.6651
77.1600
38.4401
21.5310
f1 (ppm)


Ph
O
O
O
Ph
H_3C

3d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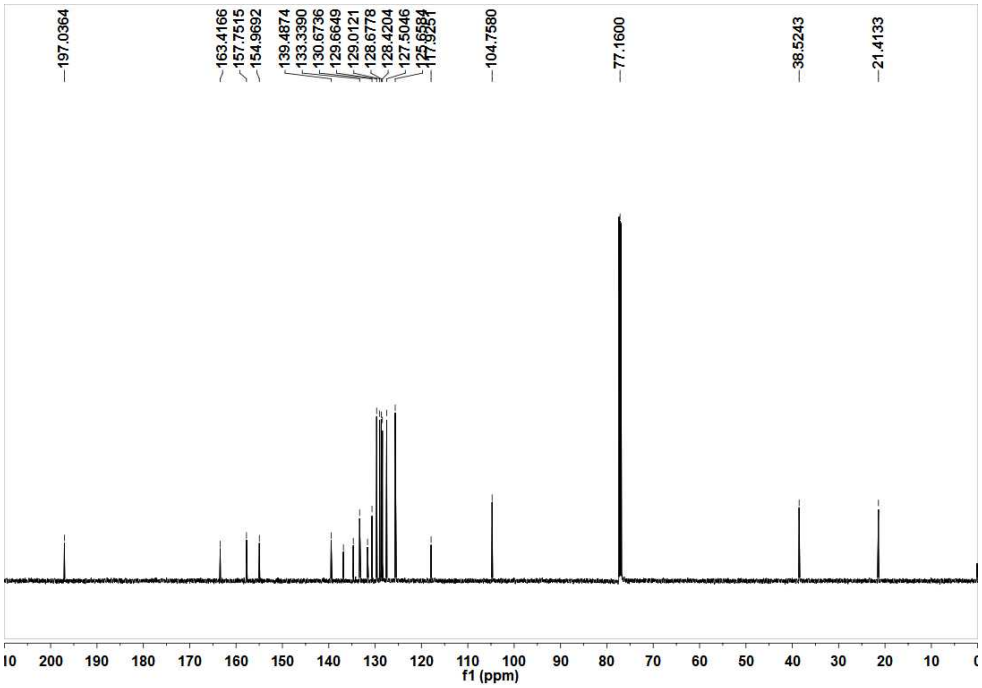
7.8507
7.8413
7.4692
7.4500
7.4416
7.4349
7.2798
7.2561
6.7214
4.1757
2.3688
1.97
1.98
1.09
5.07
4.69
0.98
2.00
3.00
f1 (ppm)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157.7515
154.9692
139.4874
133.3390
130.6736
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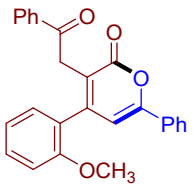
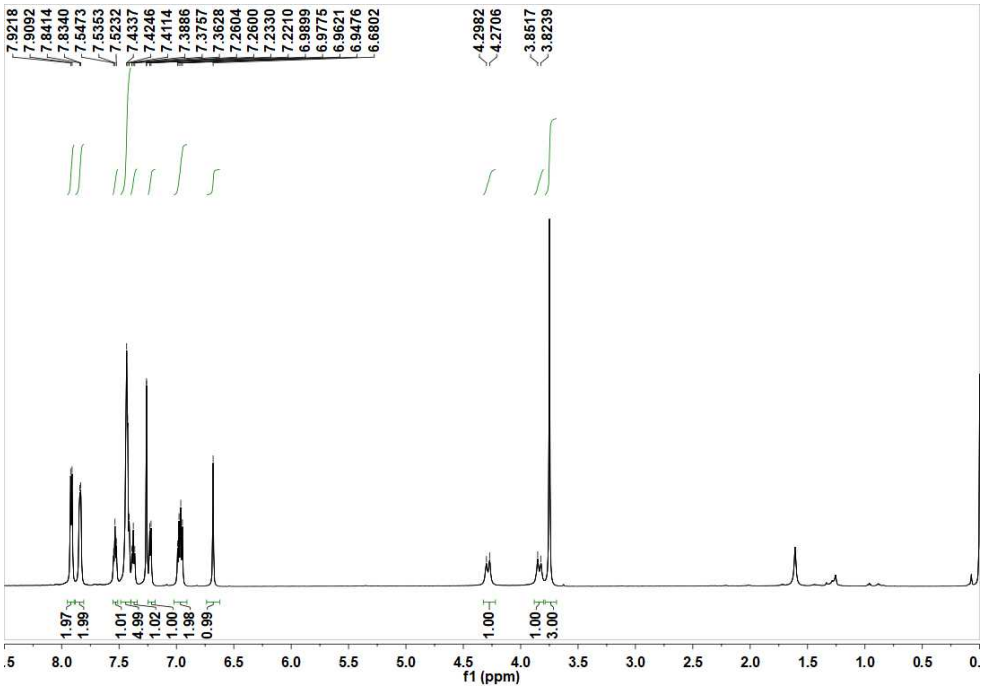
128.6778
128.4204
127.5046
104.7580
77.1600
38.5243
21.4133
f1 (ppm)


Ph
O
O
O
Ph
OCH3

3e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7.8414
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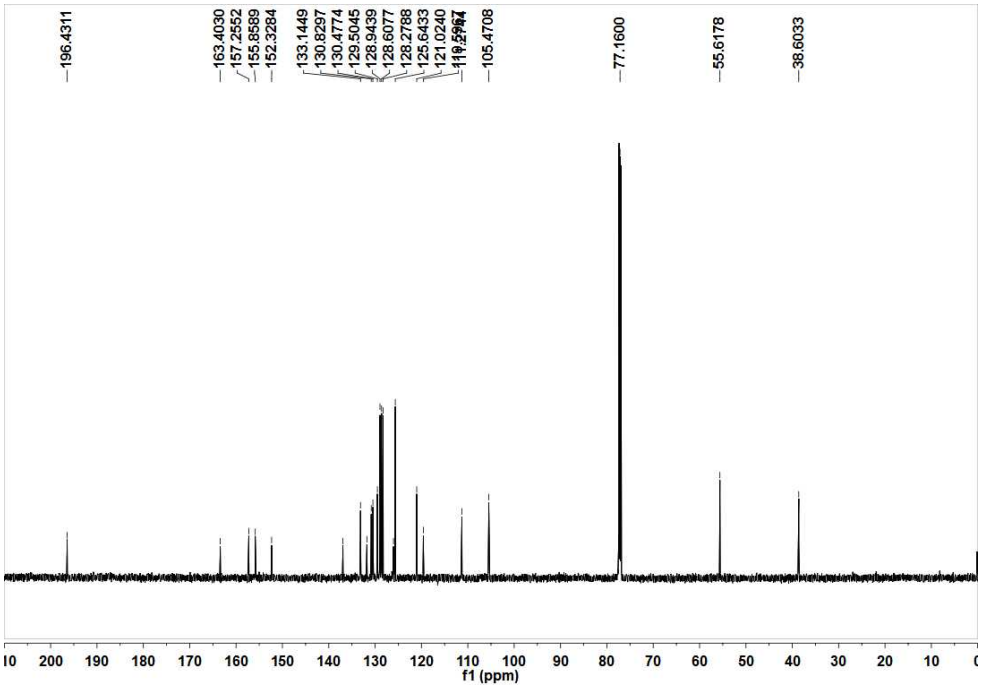
7.5473
7.5353
7.5232
7.4337
7.4246
7.4114
7.3886
7.3757
7.3628
7.2604
7.2600
7.2330
7.2210
6.9899
6.9775
6.9621
6.9476
6.6802
4.2982
4.2706
3.8517
3.8239
1.97
1.99
1.01
4.99
1.02
1.00
1.98
0.99
1.00
1.00
3.00
f1 (ppm)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157.2552
155.8589
152.3284
133.1449
130.8297
130.4774
129.5045
128.9439
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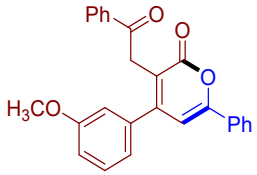
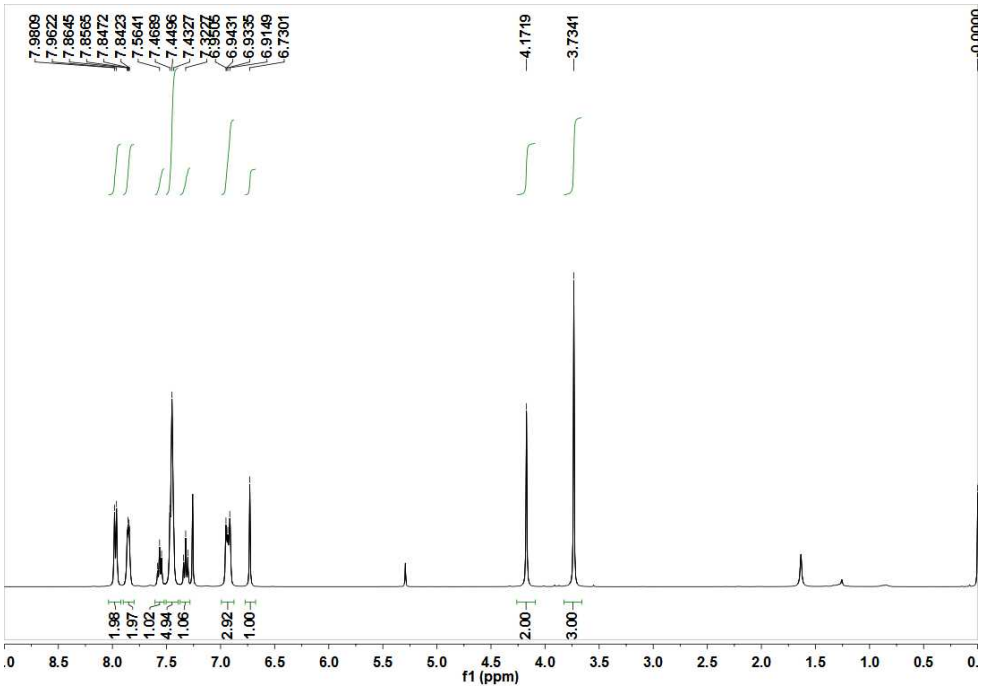
128.2788
125.6433
121.0240
105.4708
77.1600
55.6178
38.6033
f1 (ppm)


Ph
O
O
O
H_3CO
Ph

3f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7.8645
7.8565
7.8472
7.8423
7.5641
7.4689
7.4496
7.4327
6.9431
6.9335
6.9149
6.7301
4.1719
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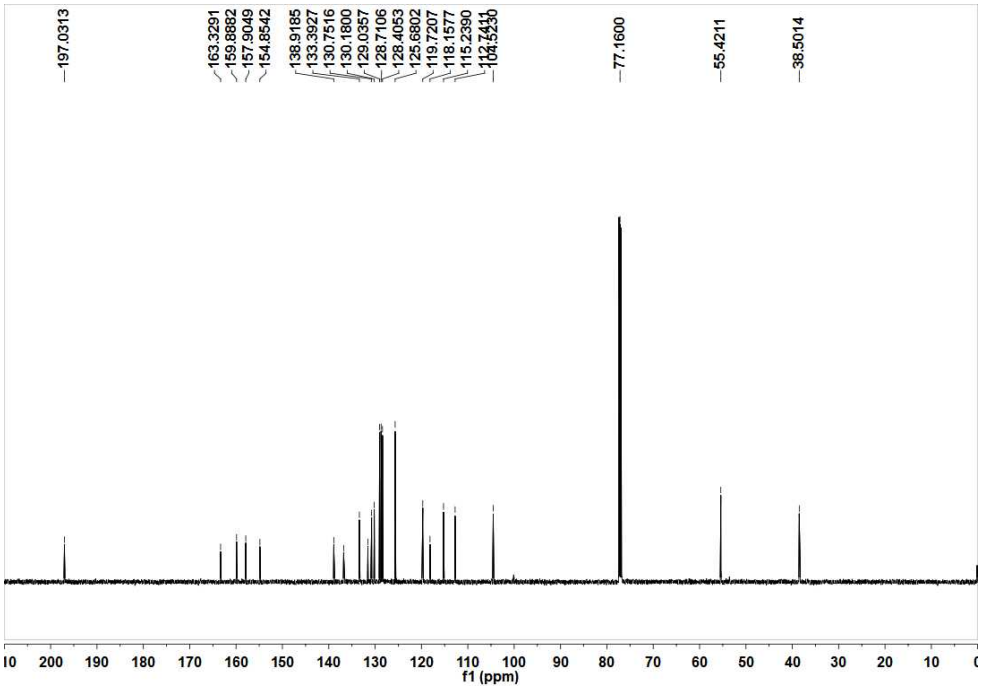
2.92
1.00
2.00
3.00
f1 (ppm)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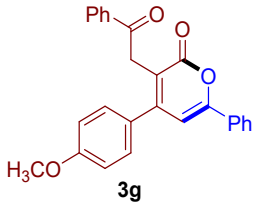
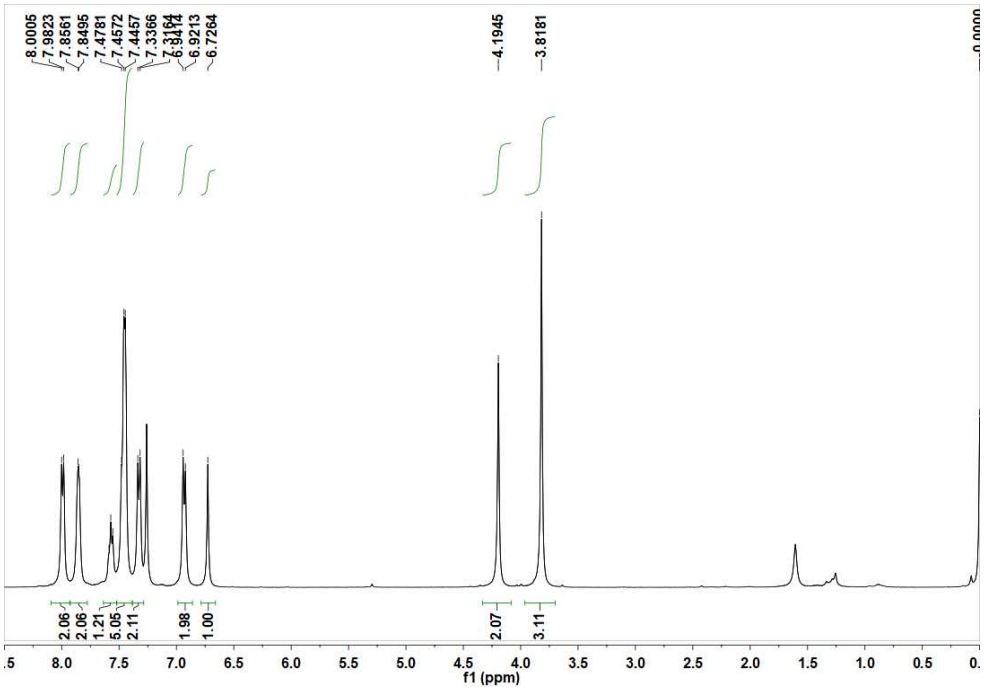
159.8882
157.9049
154.8542
138.9185
133.3927
130.7516
130.1800
129.0357
128.7106
128.4053
125.6802
119.7207
118.1577
115.2390
104.5230
77.1600
55.4211
38.5014
f1 (ppm)


Ph
O
O
O
Ph
H3CO

3g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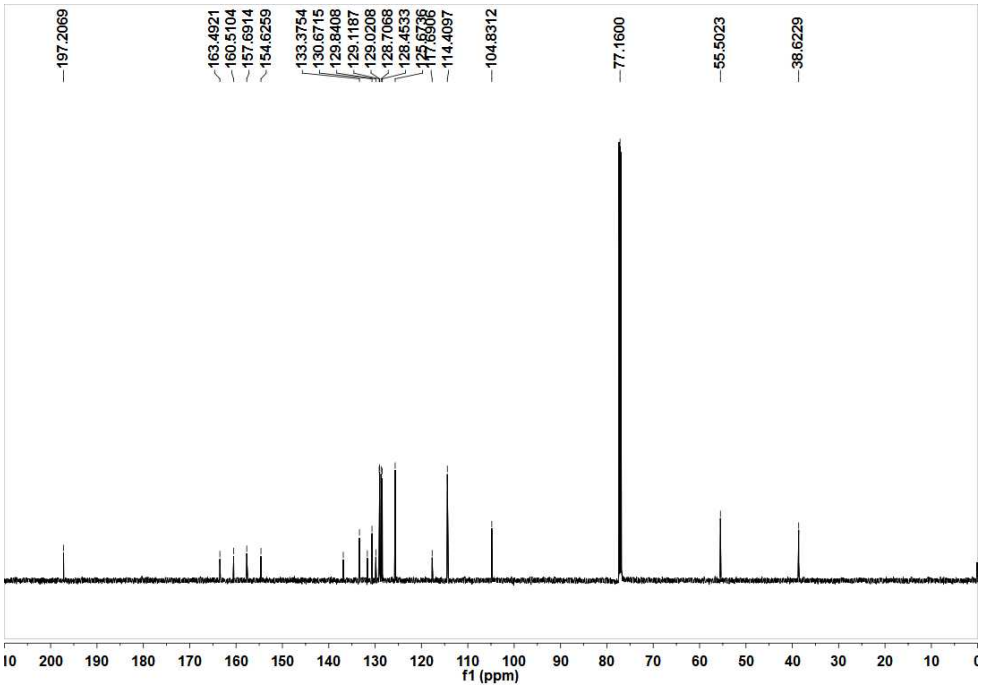
7.8661
7.8495
7.4781
7.4572
7.4457
7.3366
6.9213
6.7264
4.1945
3.8181
2.06
2.06
1.21
5.05
2.11
1.98
1.00
2.07
3.11
f1 (ppm)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160.5104
157.6914
154.6259
133.3754
130.6715
129.8408
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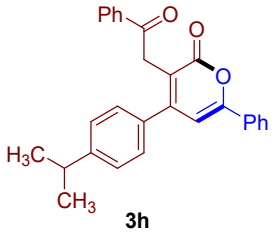
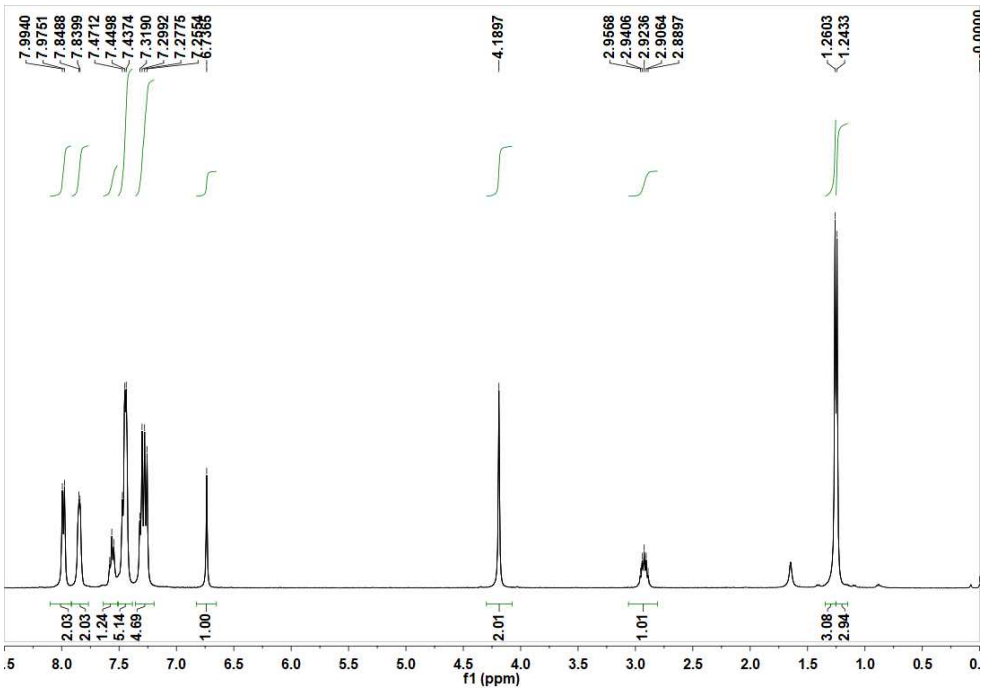
104.8312
77.1600
55.5023
38.6229
f1 (ppm)


Ph
O
O
O
Ph
H3C
CH3

3h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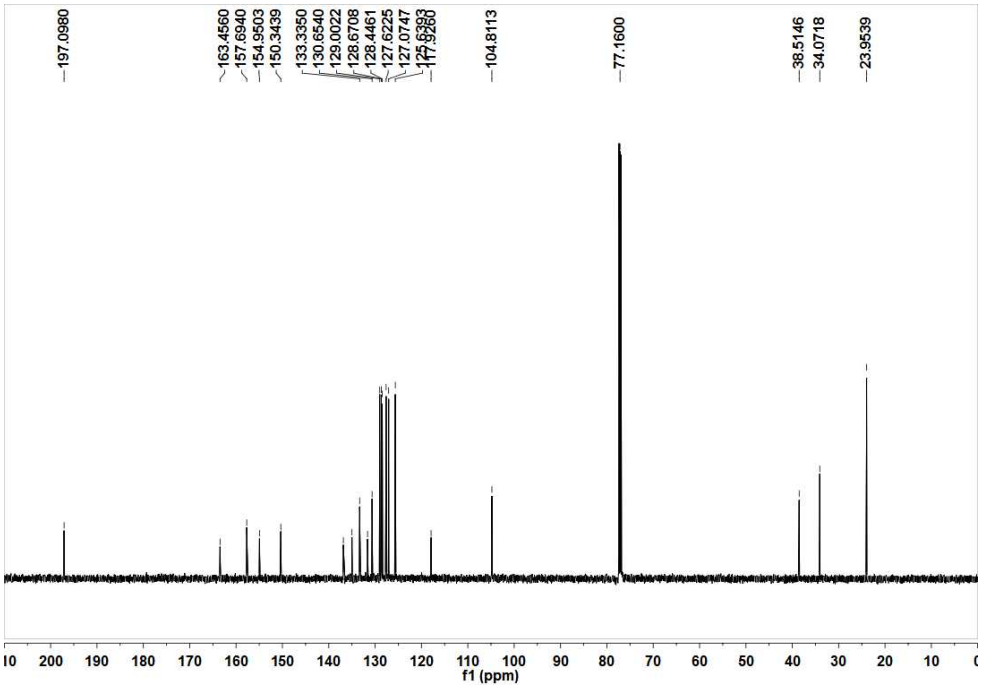
7.8488
7.8399
7.4712
7.4498
7.4374
7.3190
7.2992
7.2775
4.1897
2.9568
2.9406
2.9236
2.9064
2.8897
1.2603
1.2433
2.03
2.03
1.24
5.14
4.69
1.00
2.01
1.01
3.08
2.94
f1 (ppm)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129.0022
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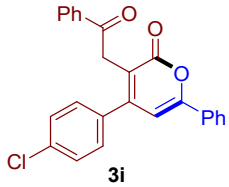
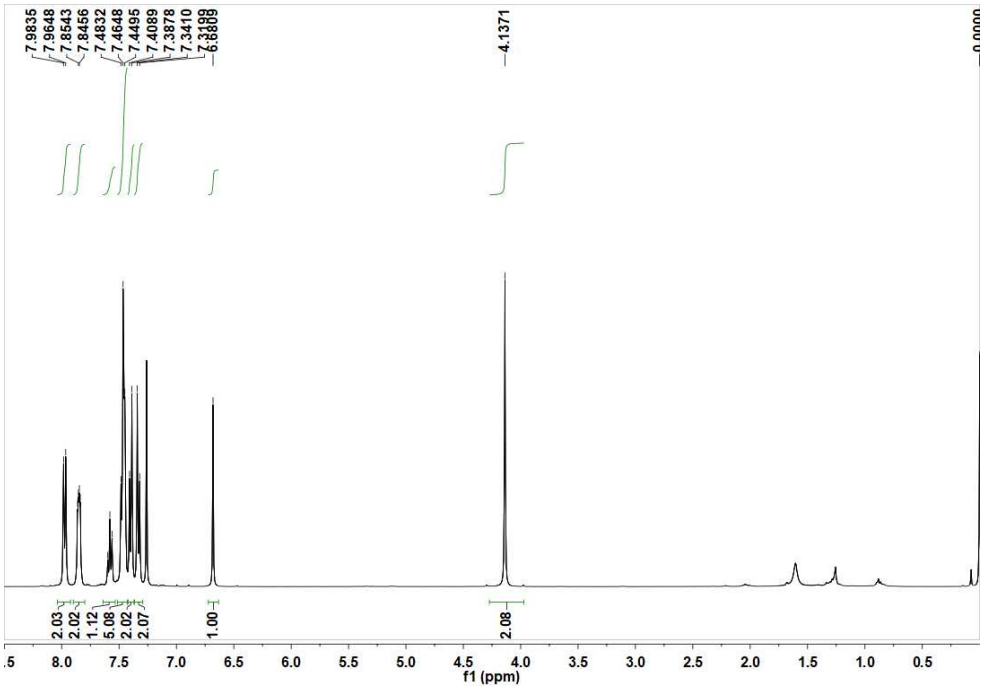
104.8113
77.1600
38.5146
34.0718
23.9539
f1 (ppm)


Ph
O
O
O
Ph
Cl

3i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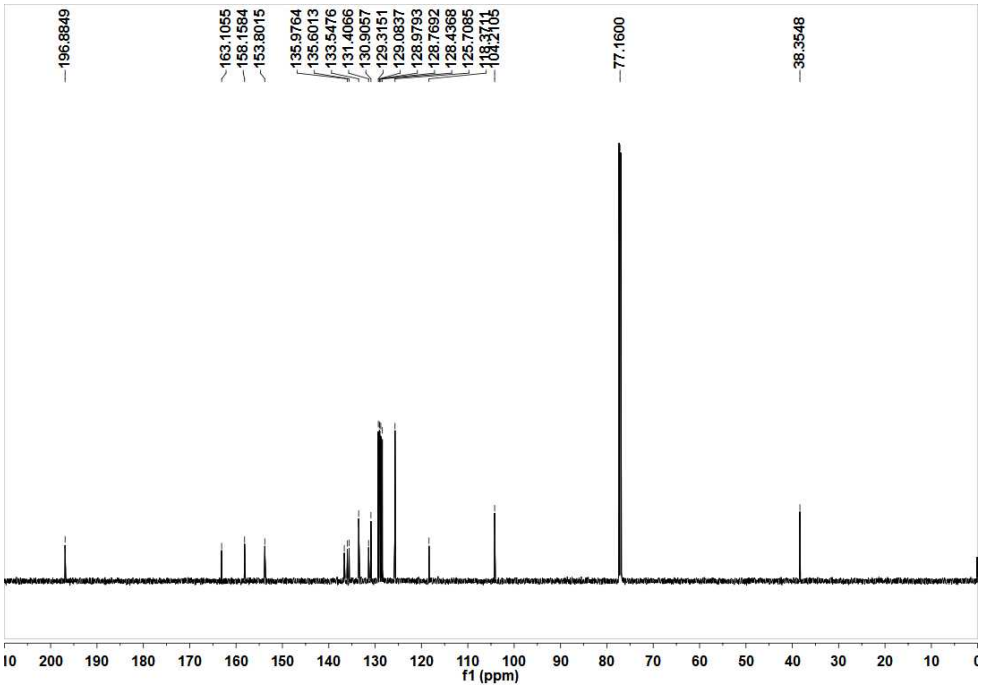
7.8456
7.4832
7.4648
7.4495
7.4089
7.3878
7.3410
6.8809
4.1371
2.03
2.02
1.12
5.08
2.02
2.07
1.00
2.08
f1 (ppm)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133.5476
131.4066
130.9057
129.3151
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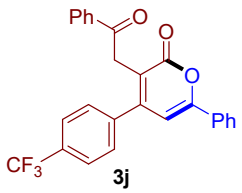
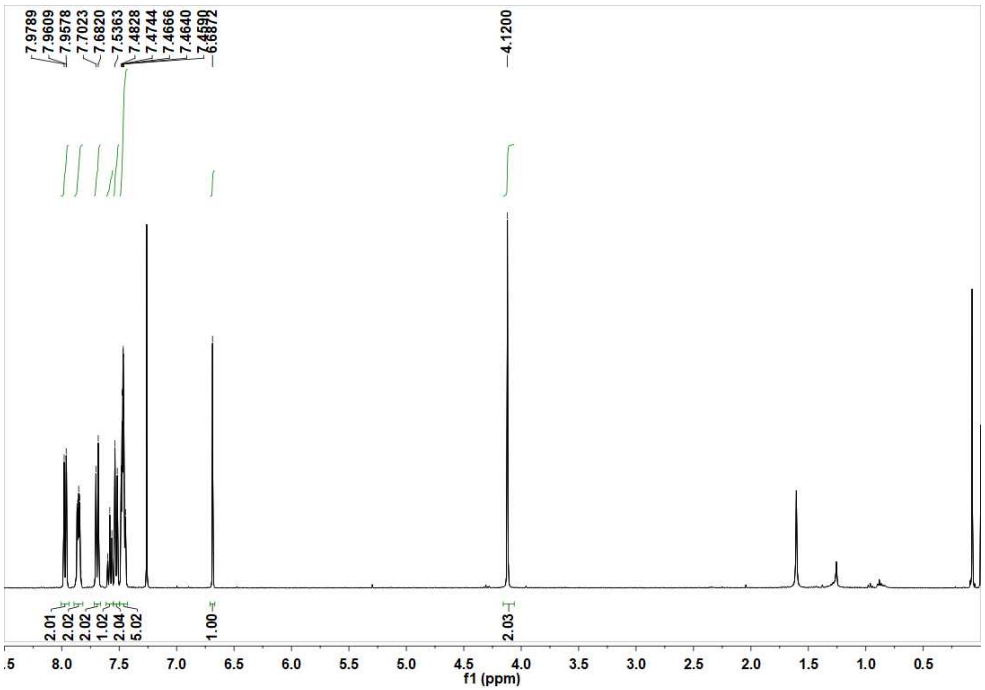
128.7692
128.4368
125.7085
118.3711
104.2105
77.1600
38.3548
f1 (ppm)


Ph
O
O
O
Ph
CF3

3j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7.6820
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7.4828
7.4744
7.4666
7.4640
6.8872
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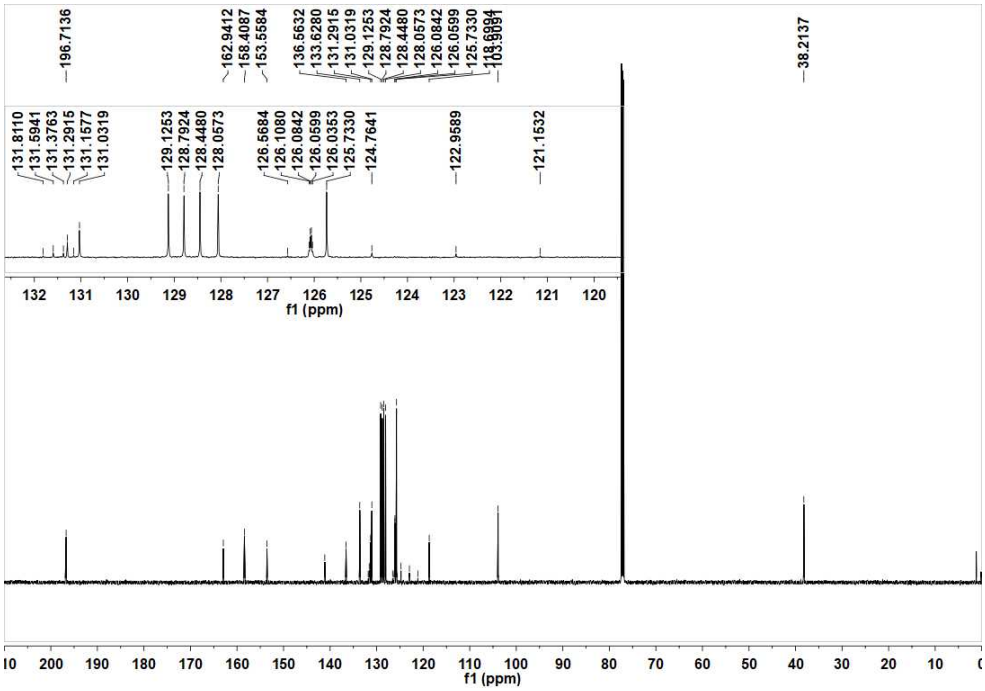
2.01
2.02
2.02
1.02
2.04
5.02
1.00
2.03
f1 (ppm)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133.6280
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129.1253
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128.0573
126.0842
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125.7330
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131.6941
131.3763
131.2915
131.1577
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129.1253
128.7924
128.4480
128.0573
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126.0599
126.0353
125.7330
124.7641
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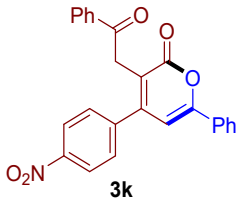
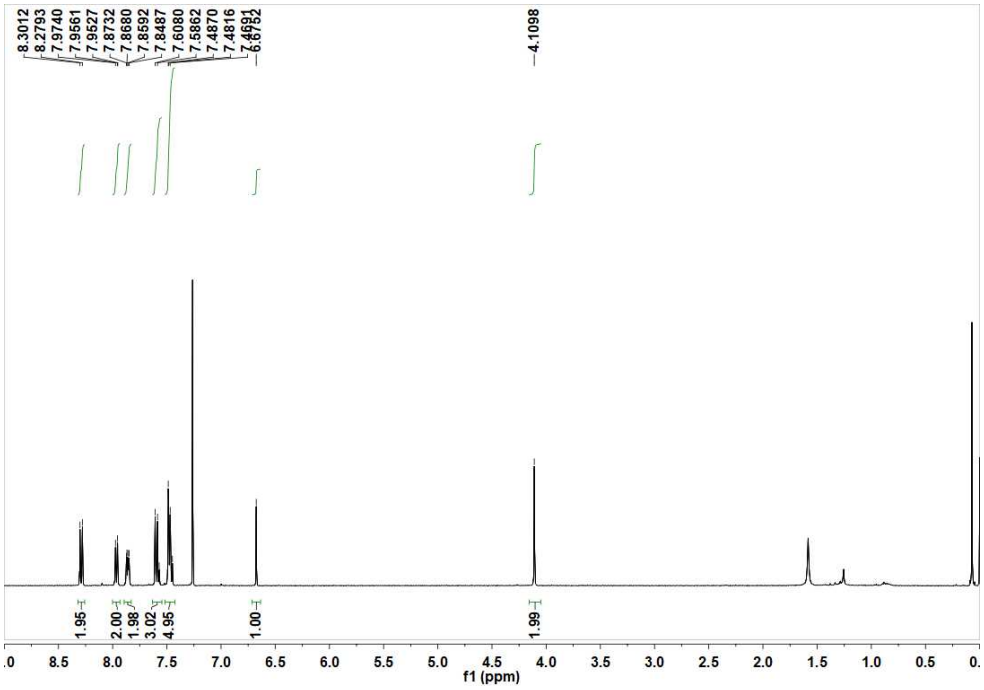
121.1532
f1 (ppm)


Ph
O
O
O
Ph
O2N

3k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7.9527
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7.8487
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7.4870
7.4816
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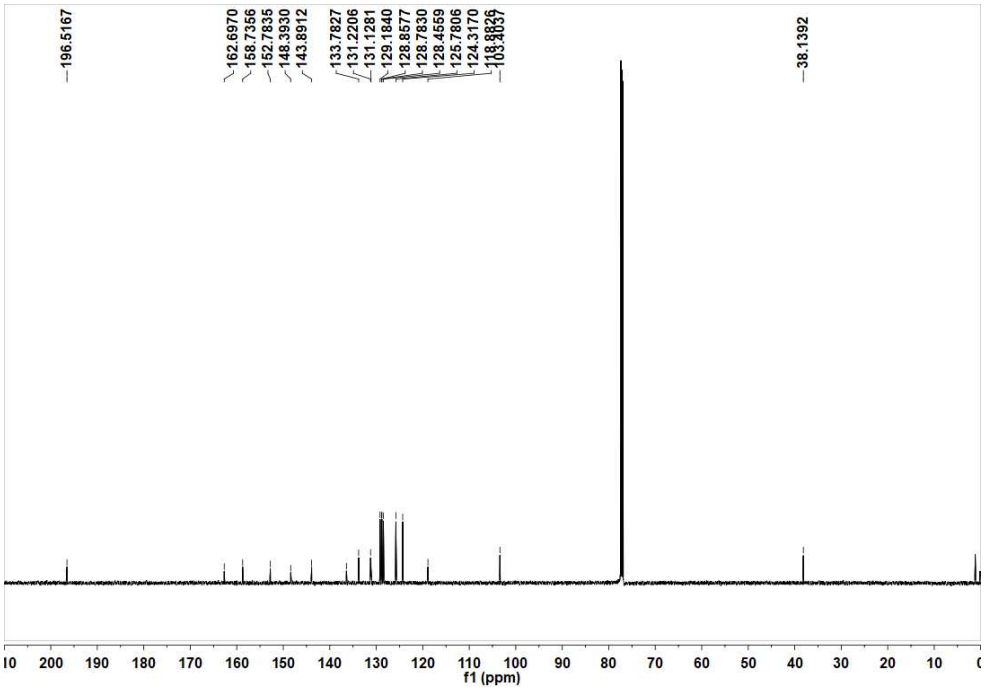
2.00
1.98
3.02
4.95
1.00
1.99
f1 (ppm)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143.8912
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128.7830
128.4559
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124.3170
118.8826
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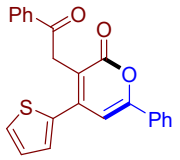
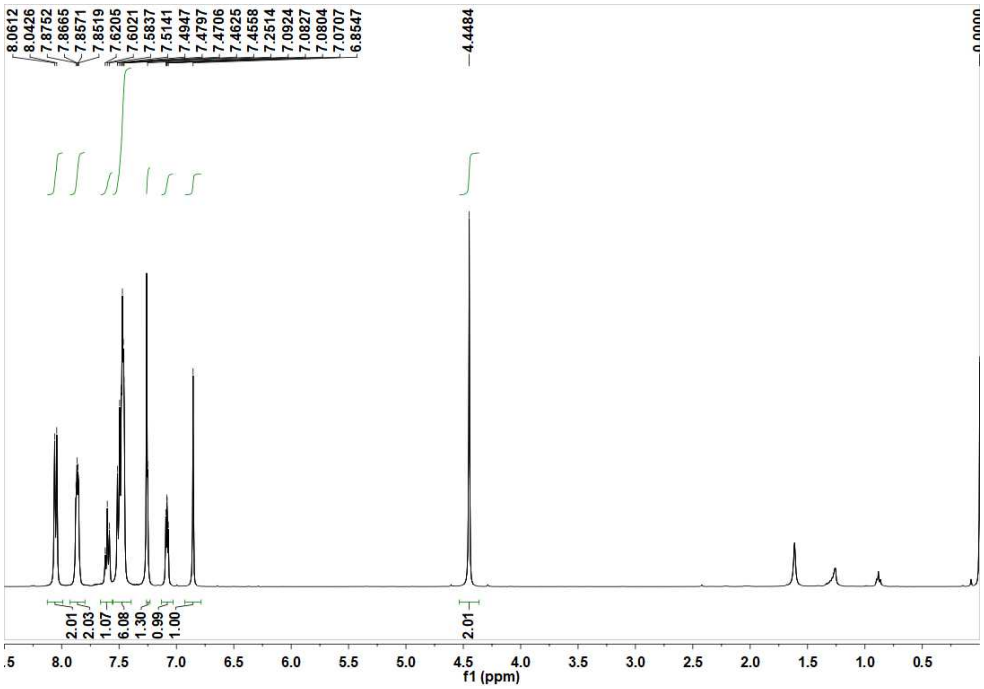
38.1392
f1 (ppm)


Ph
O
O
O
S
Ph

3l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7.4947
7.4797
7.4706
7.4625
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7.2514
7.0924
7.0827
7.0804
7.0707
6.8547
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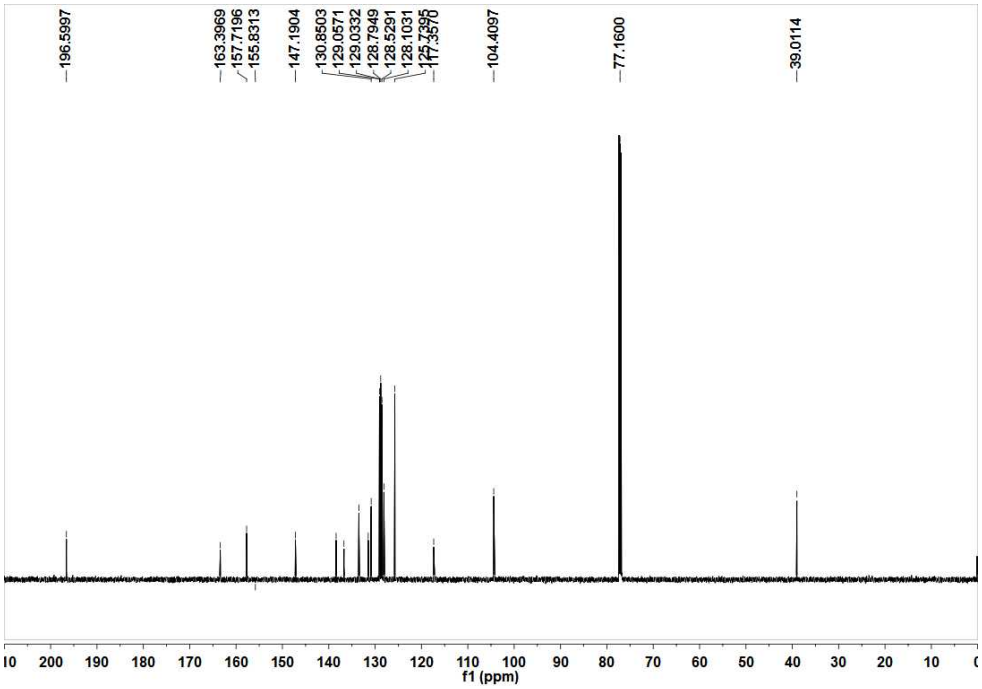
2.03
1.07
6.08
1.30
0.99
1.00
2.01
f1 (ppm)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155.8313
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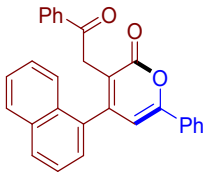
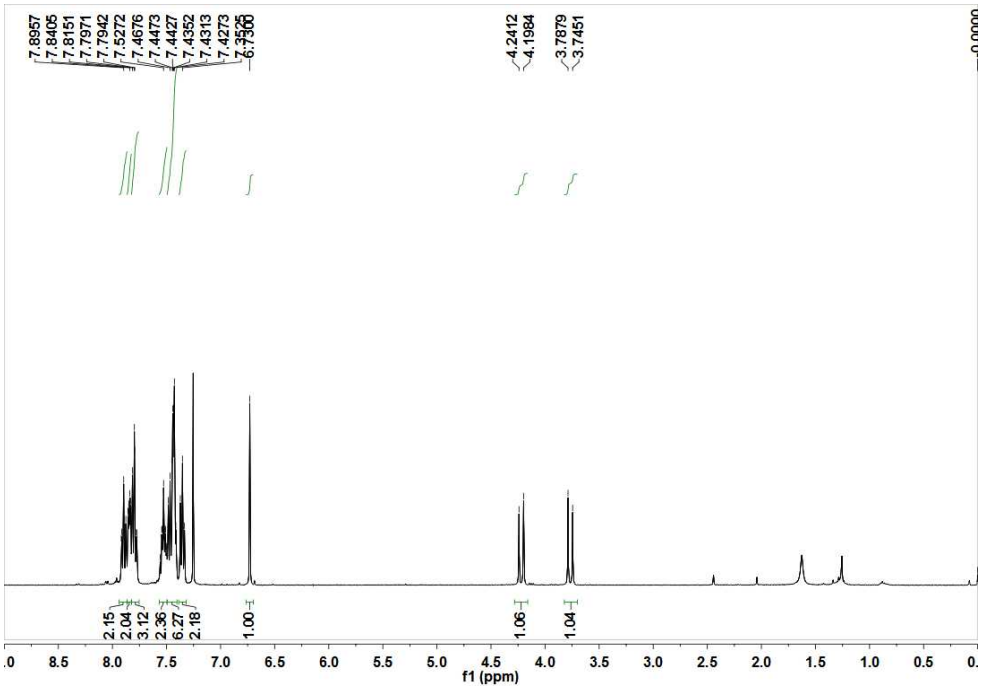
128.1031
104.4097
77.1600
39.0114
f1 (ppm)


Ph
O
O
O
Ph

3m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7.4313
7.4273
4.2412
4.1984
3.7879
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3.12
2.36
6.27
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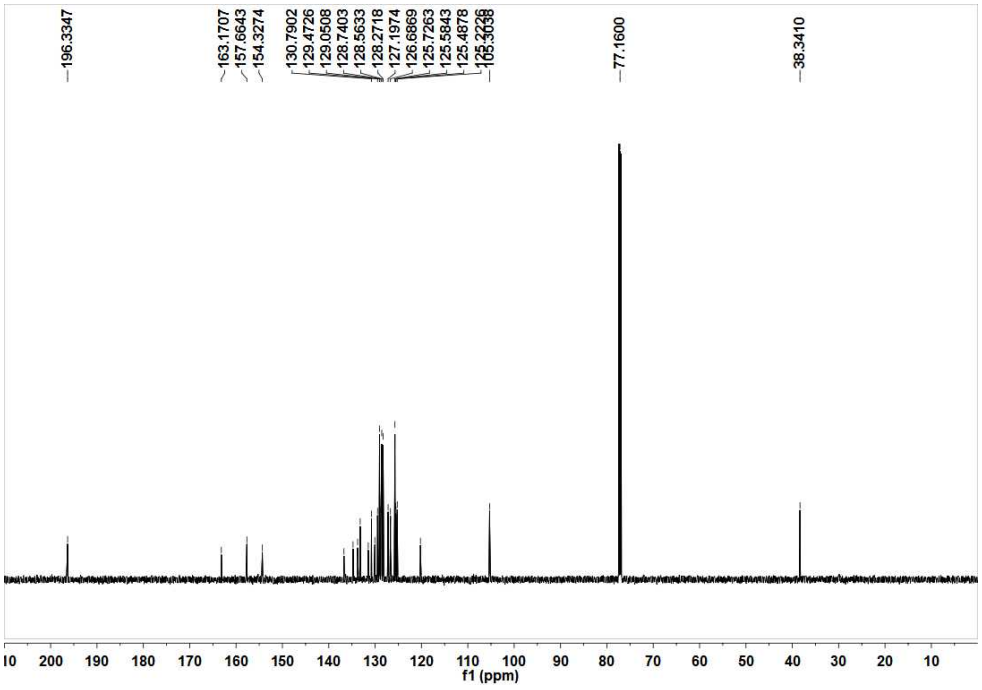
1.04
f1 (ppm)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157.6643
154.3274
130.7902
129.4726
129.0508
128.7403
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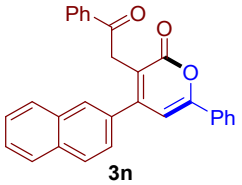
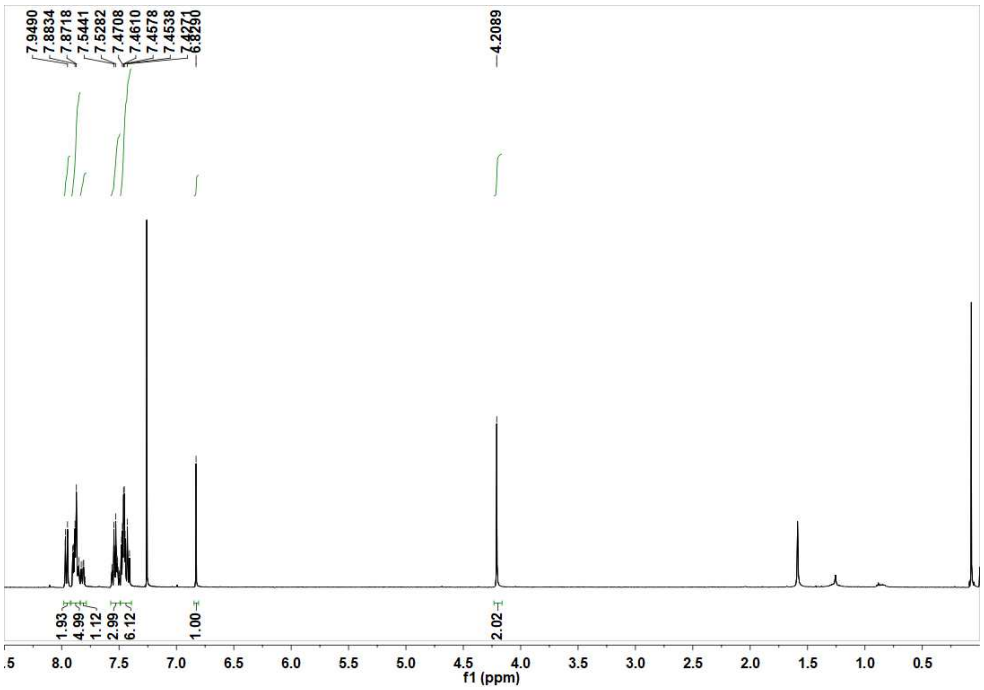
125.7263
125.5843
125.4878
77.1600
38.3410
f1 (ppm)


Ph
O
O
O
Ph

3n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7.5441
7.5282
7.4708
7.4610
7.4578
7.4538
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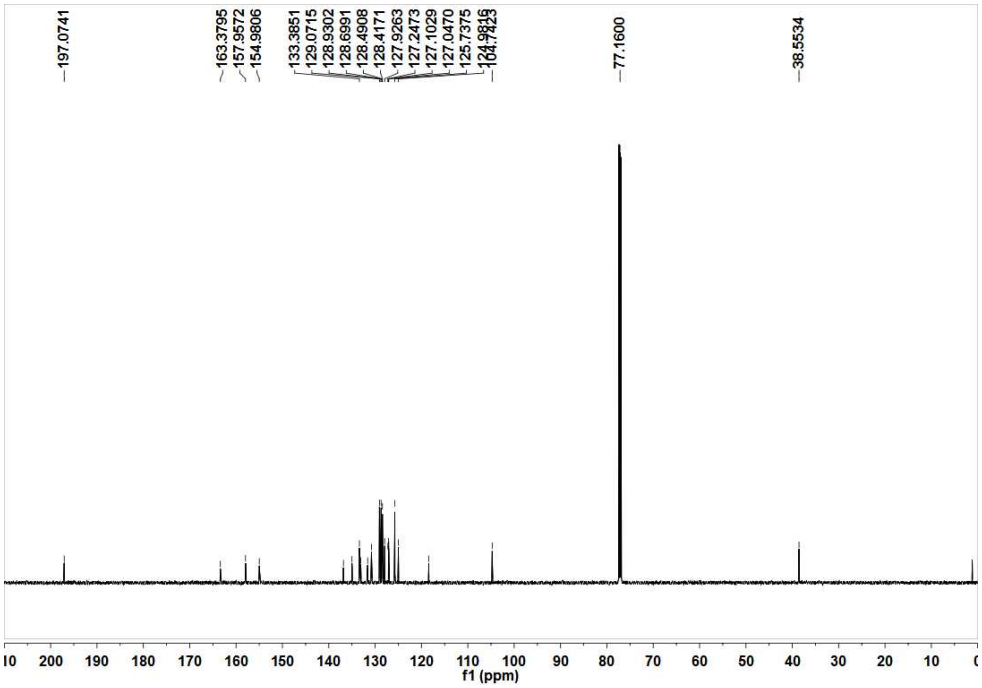
1.93
4.99
1.12
2.99
6.12
1.00
2.02
f1 (ppm)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154.9806
133.3851
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128.9302
128.6991
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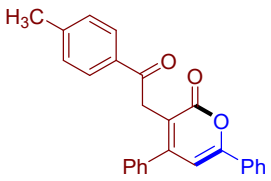
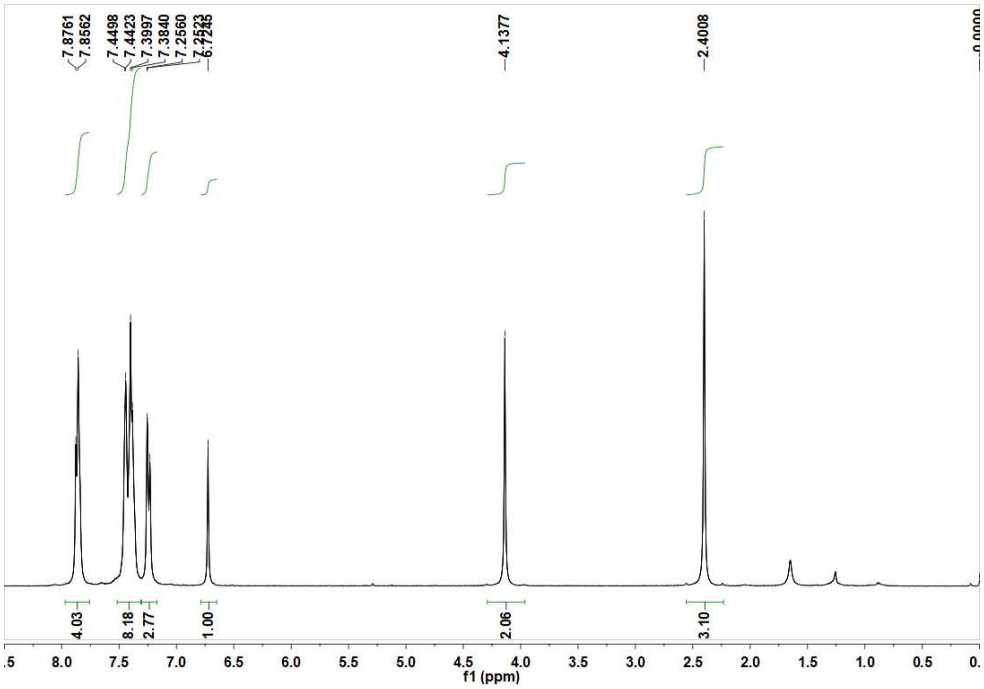
127.9263
127.2473
127.1029
127.0470
125.7375
77.1600
38.5534
f1 (ppm)


H3C
O
O
O
Ph
Ph

3o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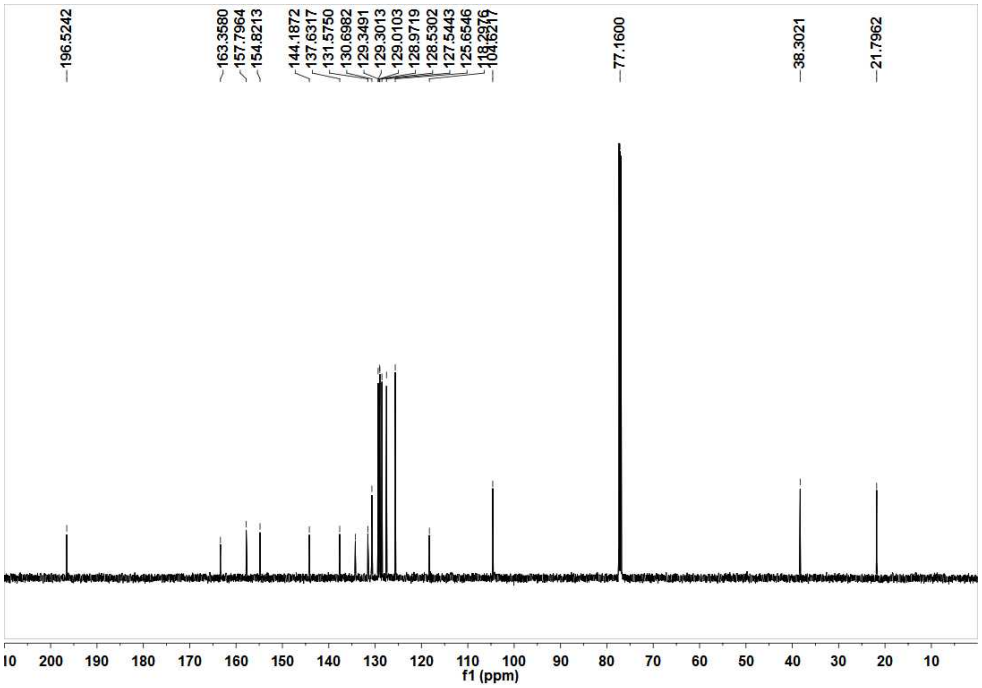
7.8562
7.4498
7.4423
7.3997
7.3840
7.2560
4.1377
2.4008
4.03
8.18
2.77
1.00
2.06
3.10
f1 (ppm)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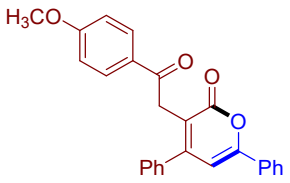
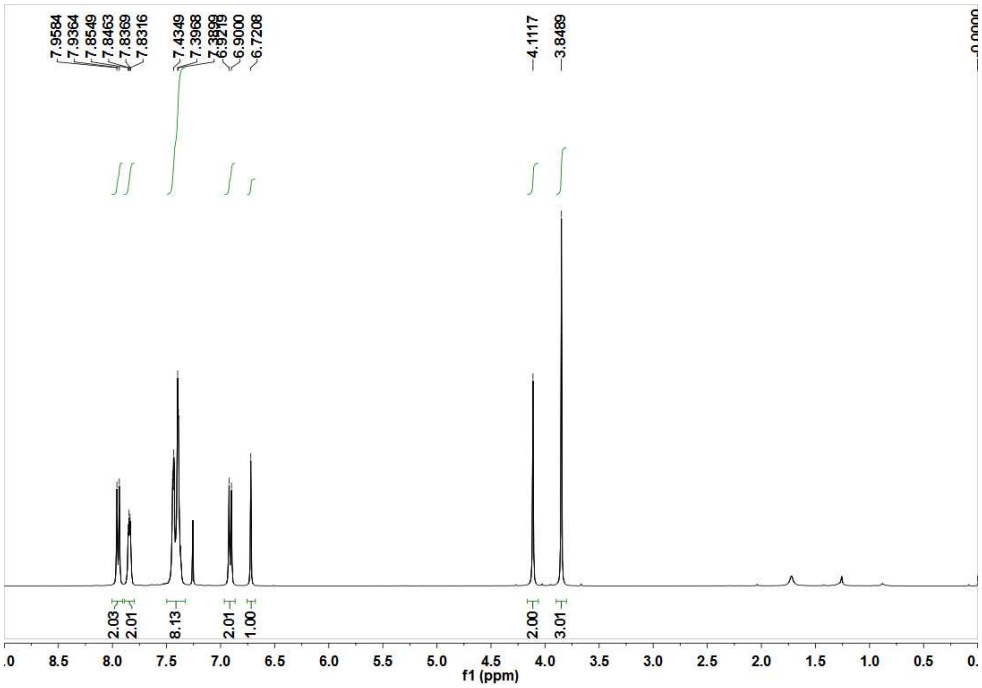
157.7964
154.8213
144.1872
137.6317
131.5750
130.6982
129.3491
129.3013
129.0103
128.9719
128.5302
127.5443
125.6546
104.8217
77.1600
38.3021
21.7962
f1 (ppm)


H3CO
O
O
O
Ph
Ph

3p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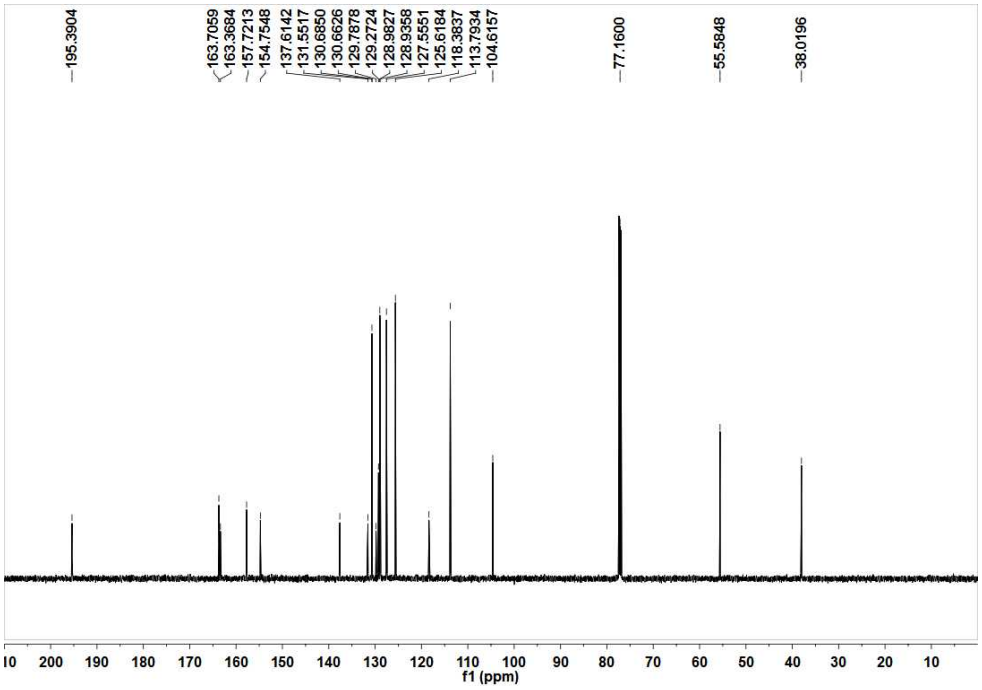
7.9364
7.8549
7.8463
7.8369
7.8316
7.4349
7.3968
6.9000
6.7208
4.1117
3.8489
2.03
2.01
8.13
2.01
1.00
2.00
3.01
f1 (ppm)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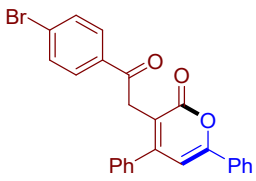
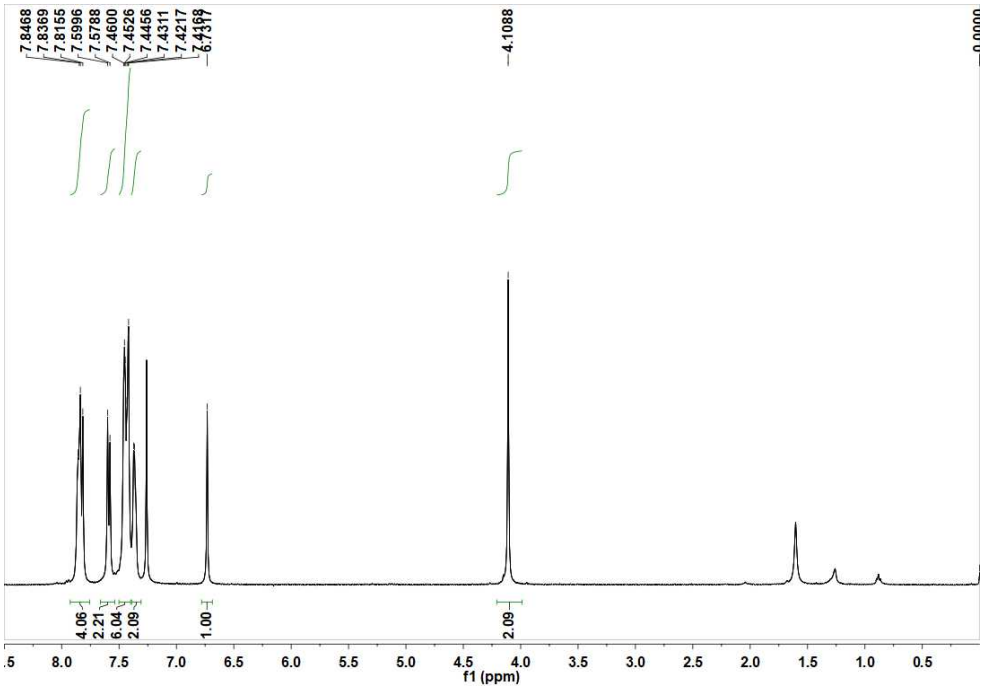
154.7548
137.6142
131.5517
130.6850
130.6626
129.7878
129.2724
128.9827
128.9358
127.5551
125.6184
118.3837
113.7934
104.6157
77.1600
55.5848
38.0196
f1 (ppm)


Br
O
O
O
Ph
Ph

3q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7.8369
7.8155
7.5996
7.5788
7.4600
7.4526
7.4466
7.4311
7.4217
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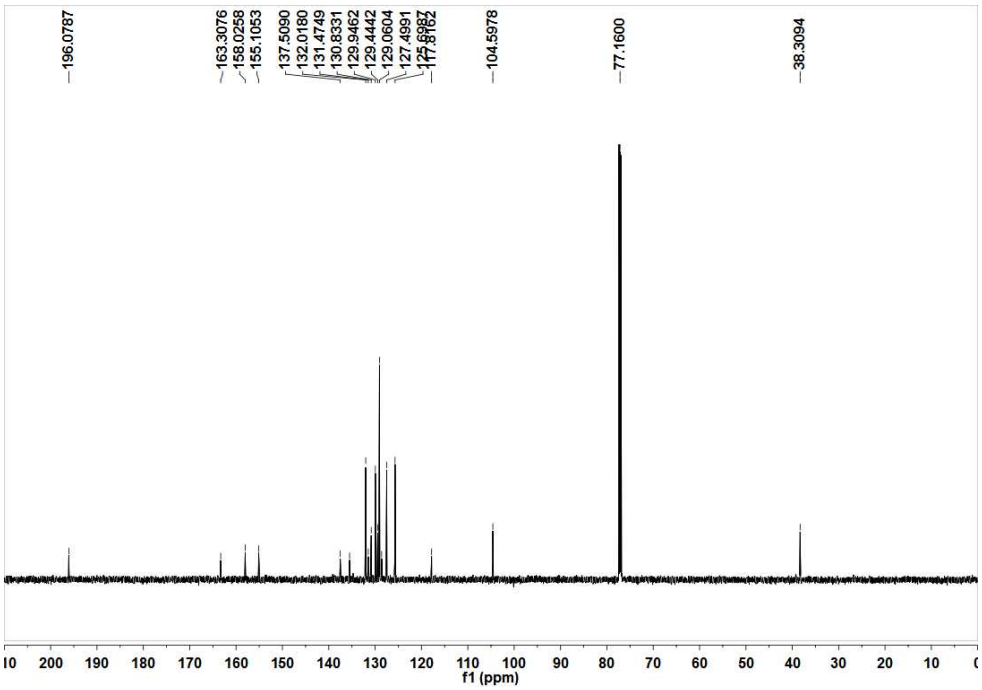
6.04
2.09
1.00
2.09
f1 (ppm)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158.0258
155.1053
137.5090
132.0180
131.4749
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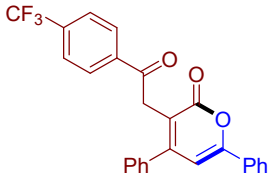
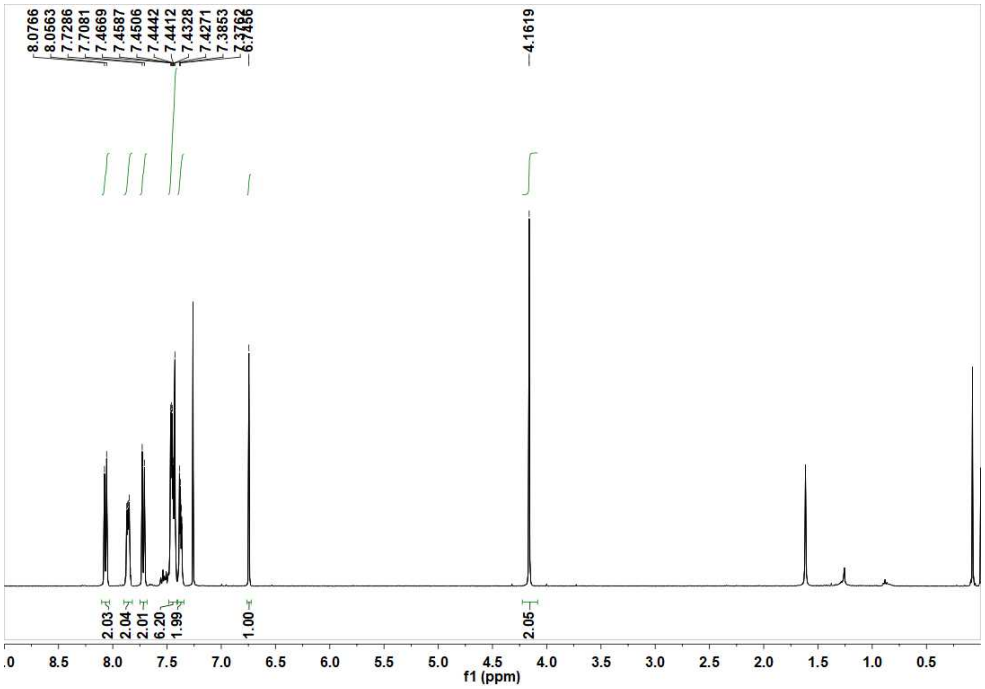
129.9462
129.4442
129.0604
127.4991
104.5978
77.1600
38.3094
f1 (ppm)


CF3
O
O
O
Ph
Ph

3r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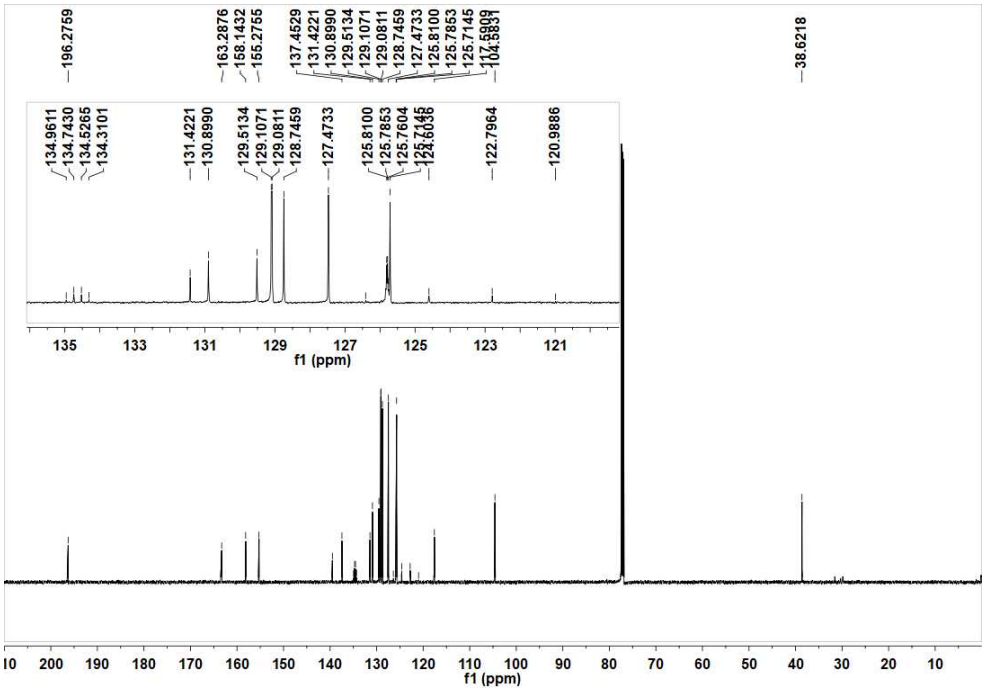
8.0563
7.7286
7.7081
7.4669
7.4587
7.4506
7.4442
7.4412
7.4328
7.4271
7.3853
7.3762
7.3456
4.1619
2.03
2.04
2.01
6.20
1.99
1.00
2.05
f1 (ppm)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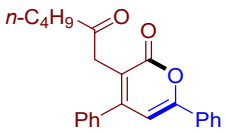
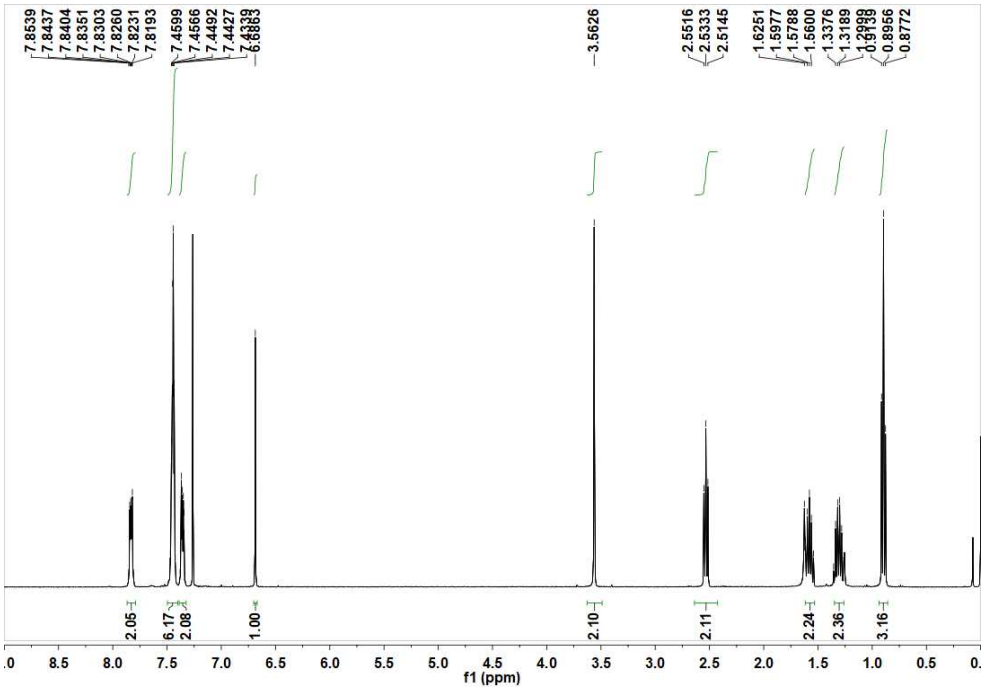
158.1432
155.2755
137.4529
131.4221
130.8990
129.5134
129.1071
129.0811
128.7459
127.4733
125.8100
125.7853
125.7145
38.6218
134.9611
134.7430
134.5265
134.3101
131.4221
130.8990
129.5134
129.1071
129.0811
128.7459
127.4733
125.8100
125.7853
125.7604
122.7964
120.9886
f1 (ppm)


n-C4H9
O
O
O
Ph
Ph

3s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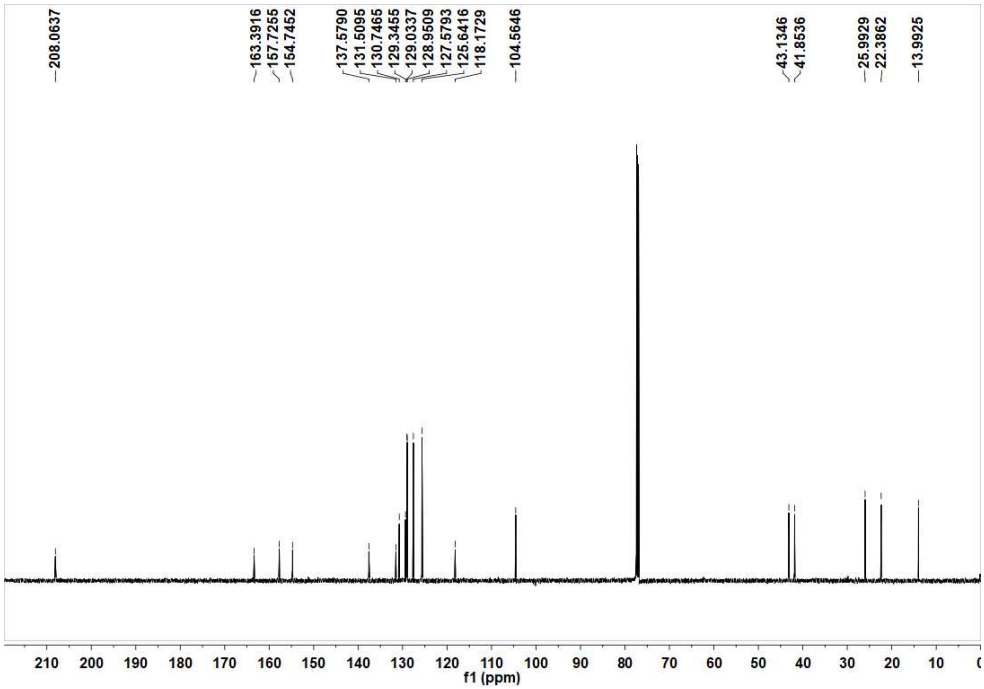
7.8404
7.8351
7.8303
7.8260
7.8231
7.8193
7.4599
7.4566
7.4492
7.4427
3.5626
2.5516
2.5333
2.5145
1.6251
1.5977
1.5788
1.5600
1.3376
1.3189
0.8956
0.8772
2.05
6.17
2.08
1.00
2.10
2.11
2.24
2.36
3.16
f1 (ppm)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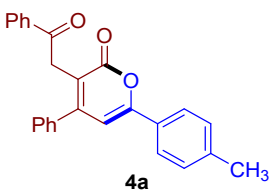
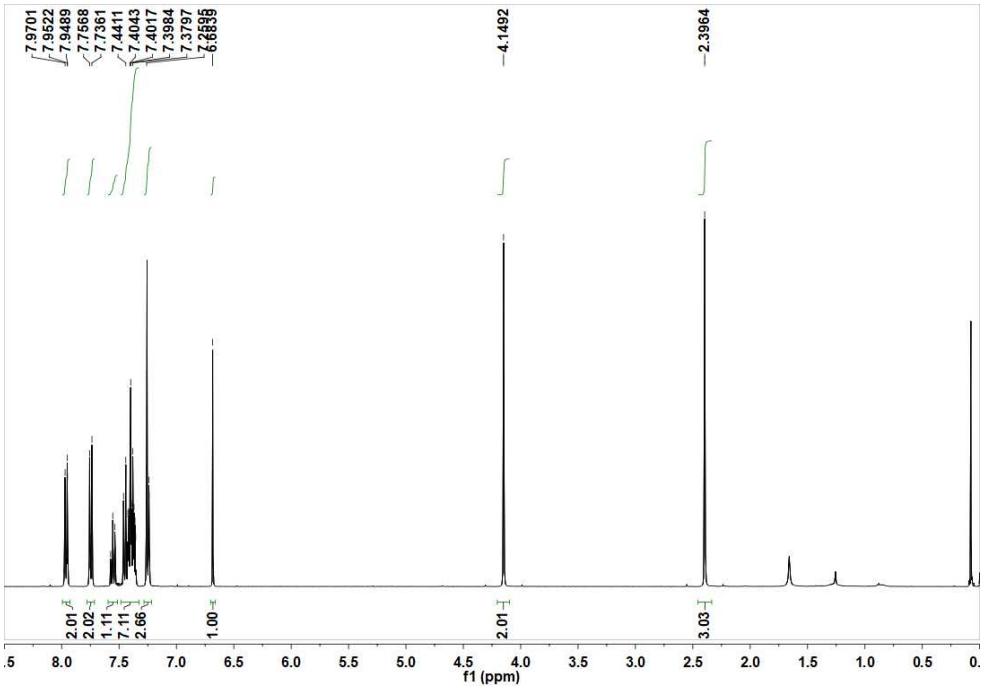
157.7255
154.7462
137.5790
131.5095
130.7465
129.3455
129.0337
128.9509
127.5793
125.6416
118.1729
104.5646
43.1346
41.8536
25.9929
22.3862
13.9925
f1 (ppm)


Ph
O
O
O
Ph
CH3

4a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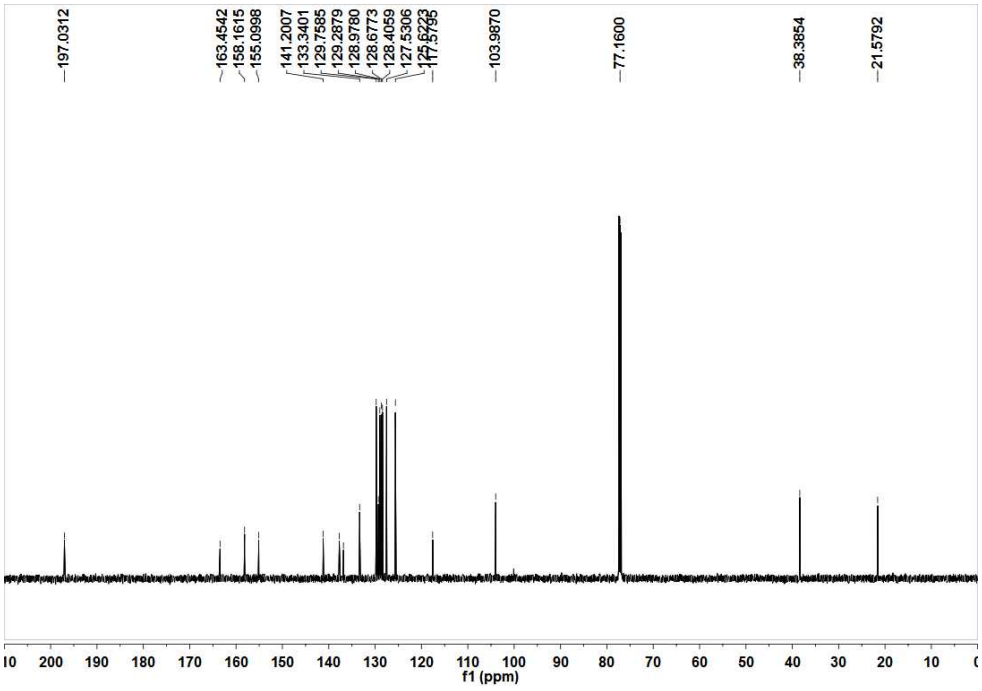
7.9522
7.9489
7.7568
7.7361
7.4411
7.4043
7.4017
7.3984
7.3797
6.6839
4.1492
2.3964
2.01
2.02
1.11
7.11
2.66
1.00
2.01
3.03
f1 (ppm)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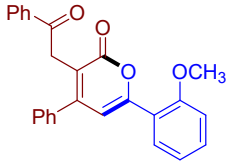
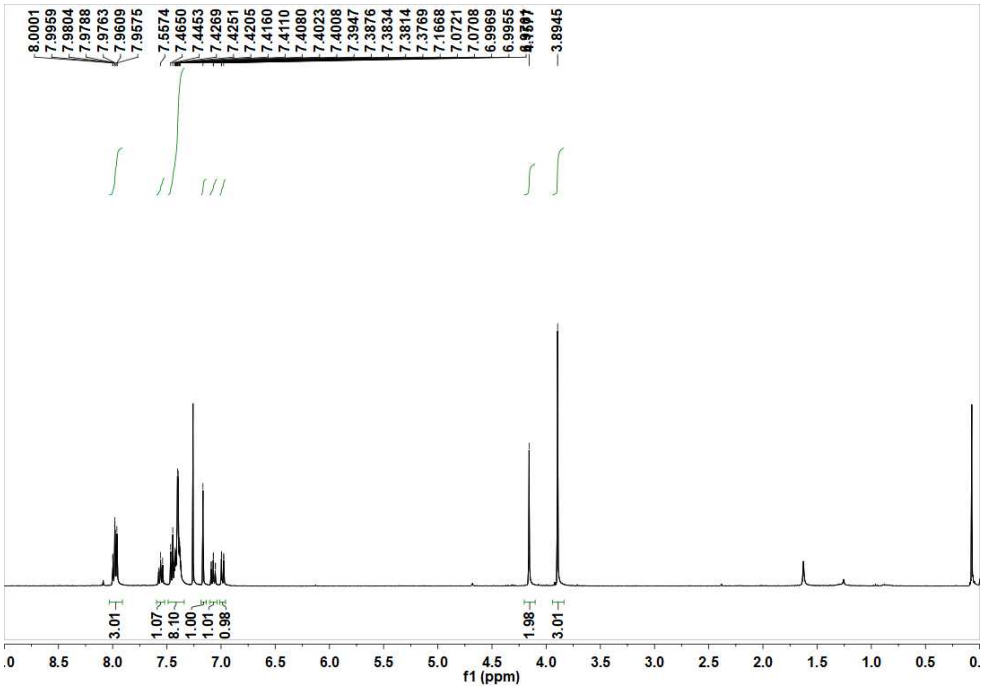
158.1615
155.0998
141.2007
133.3401
129.7585
129.2879
128.9780
128.6773
128.4059
127.5306
103.9870
77.1600
38.3854
21.5792
f1 (ppm)


Ph
O
O
O
OCH3
Ph

4b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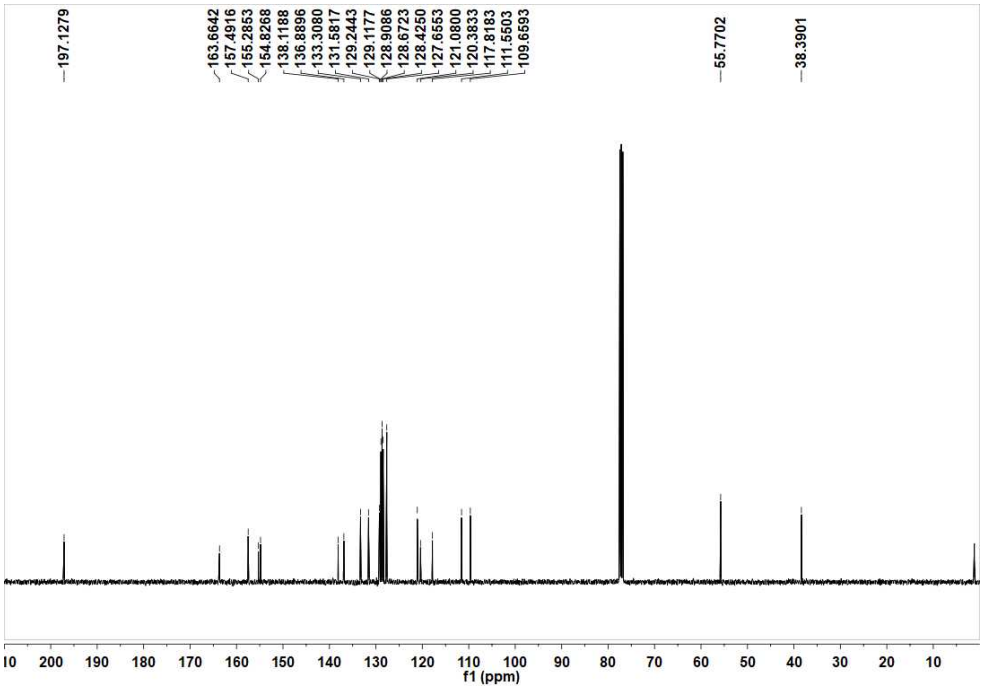
7.9804
7.9788
7.9763
7.9609
7.9575
7.5574
7.4650
7.4453
7.4269
7.4251
7.4205
7.4160
7.4110
7.4080
7.4023
7.4008
7.3947
7.3876
7.3834
7.3814
7.3769
7.1668
7.0721
7.0708
6.9969
6.9955
3.8945
3.01
1.07
8.10
1.00
1.01
0.98
1.98
3.01
f1 (ppm)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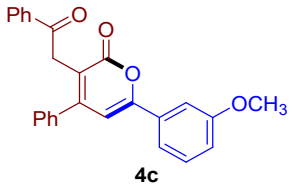
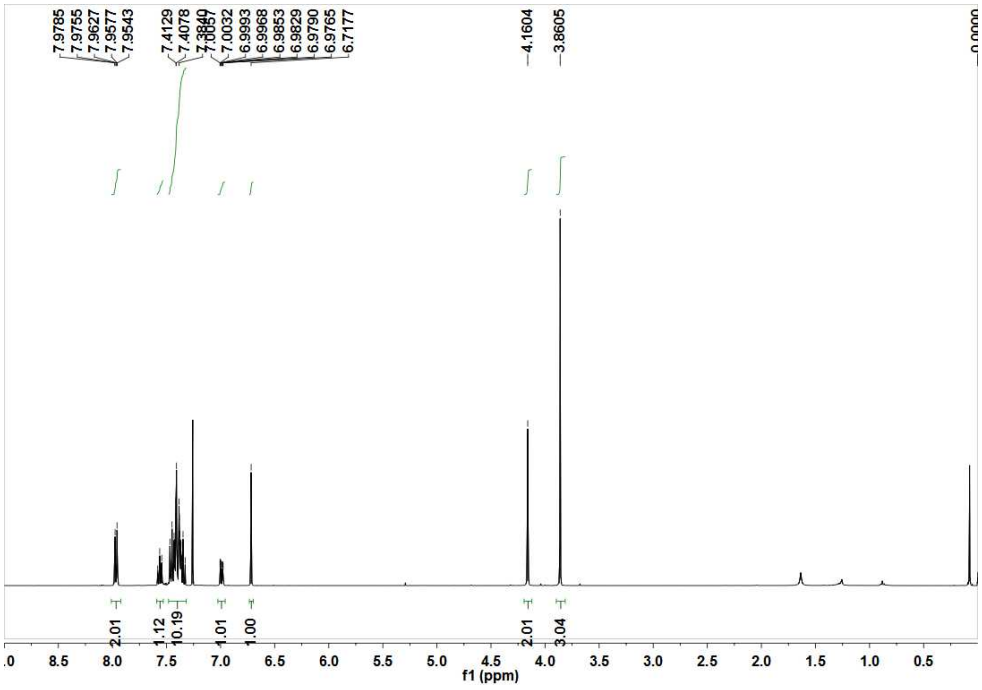
157.4916
155.2853
154.8268
138.1188
136.8896
133.3080
131.5817
129.2443
129.1177
128.9086
128.6723
128.4250
127.6553
121.0800
120.3833
117.8183
111.5503
109.6593
55.7702
38.3901
f1 (ppm)


Ph
O
O
O
Ph
OCH3

4c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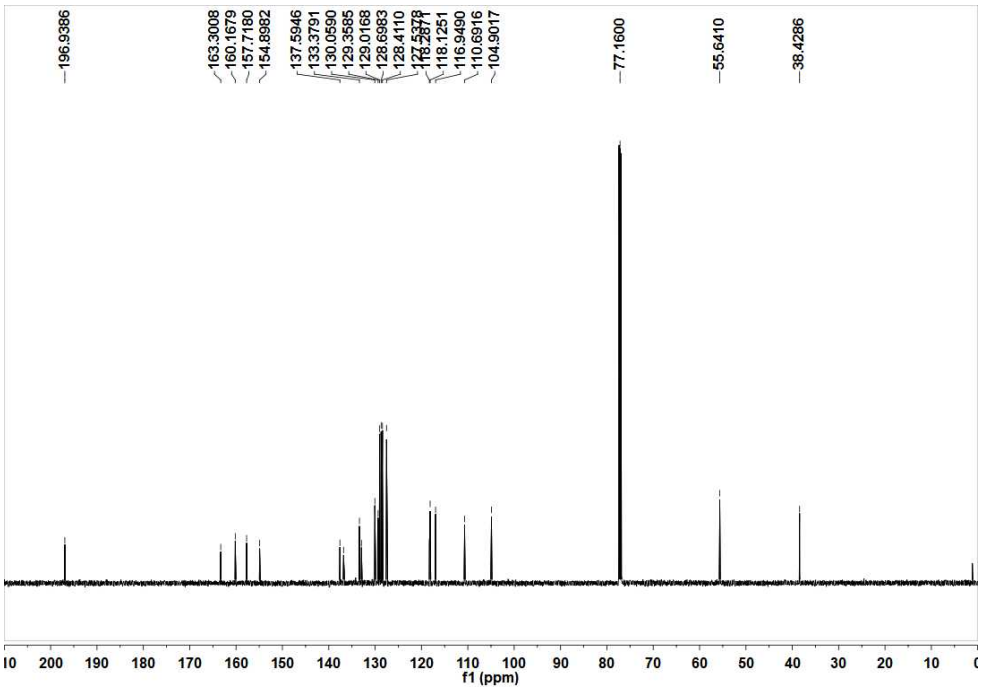
7.9627
7.9577
7.9543
7.4129
7.4078
7.0032
6.9993
6.9968
6.9853
6.9829
6.9790
6.9765
6.7177
4.1604
3.8605
2.01
1.12
10.19
1.01
1.00
2.01
3.04
f1 (ppm)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133.3791
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129.3585
129.0168
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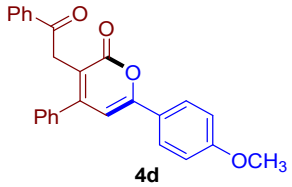
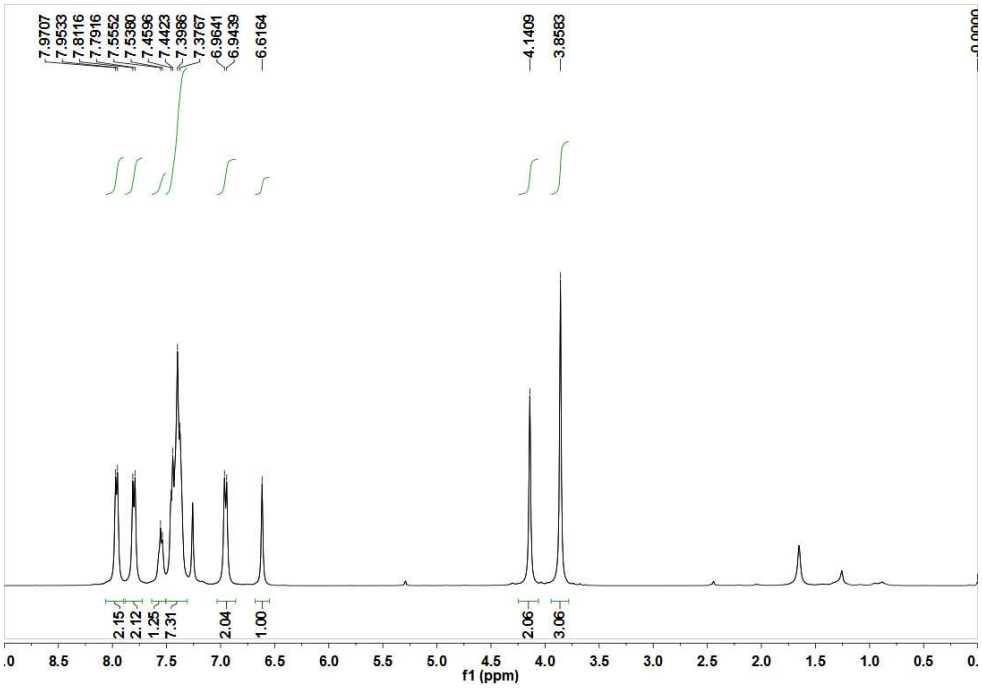
104.9017
77.1600
55.6410
38.4286
f1 (ppm)


Ph
O
O
O
Ph
OCH3

4d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7.4596
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7.3767
6.9641
6.9439
6.6164
4.1409
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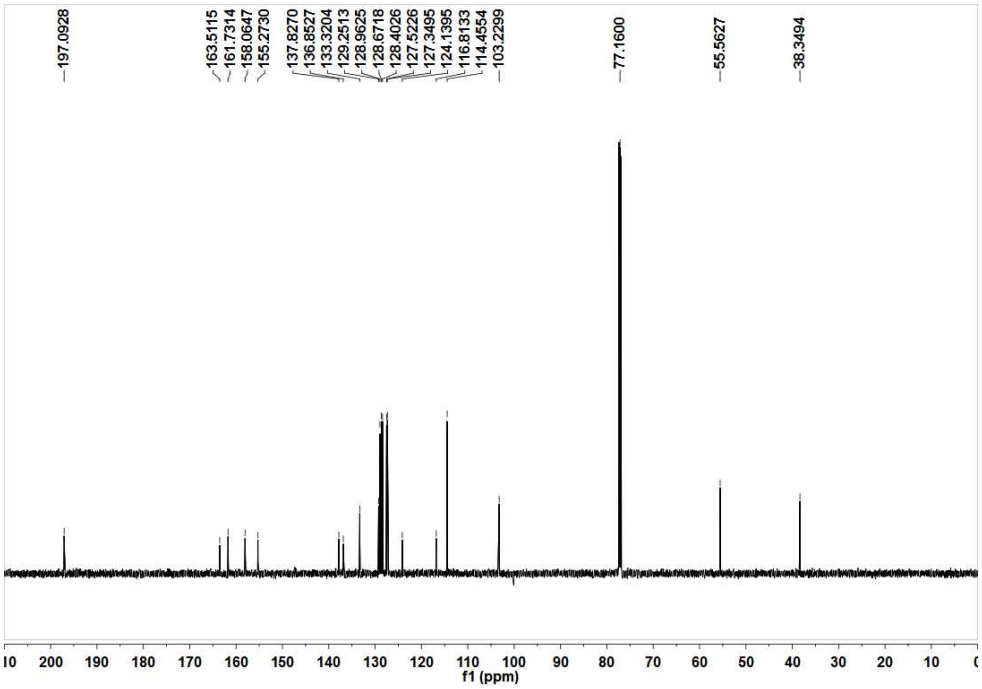
2.15
2.12
1.25
7.31
2.04
1.00
2.06
3.06
f1 (ppm)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129.2513
128.9625
128.6718
128.4026
127.5226
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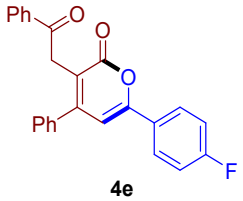
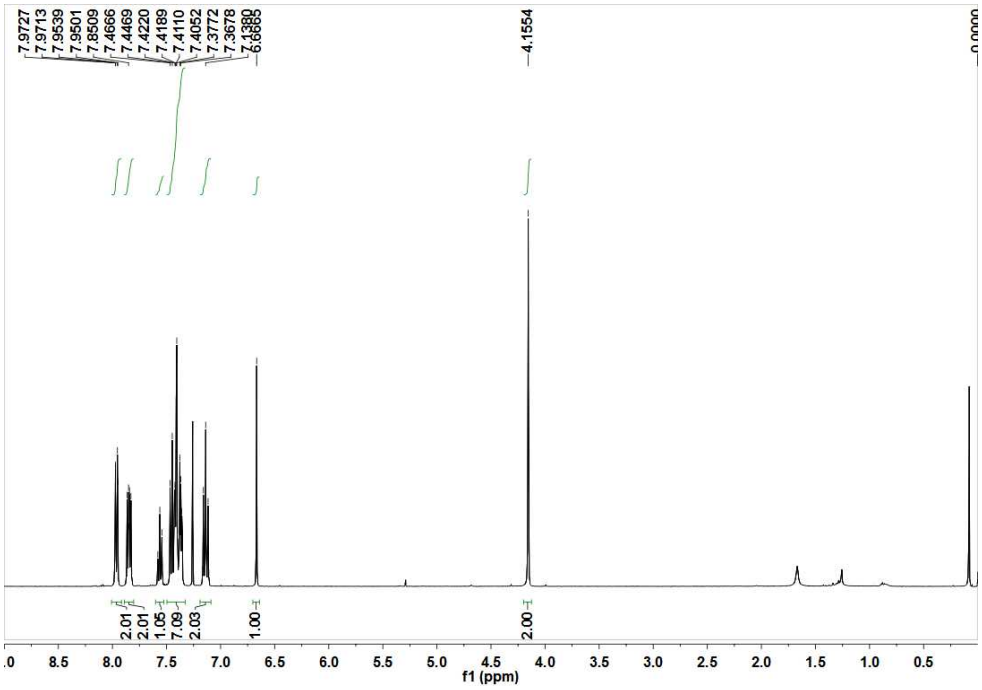
124.1395
116.8133
114.4554
103.2299
77.1600
55.5627
38.3494
f1 (ppm)


Ph
O
O
O
Ph
F

4e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7.8509
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7.4469
7.4220
7.4189
7.4110
7.4052
7.3772
7.3678
7.1380
6.8885
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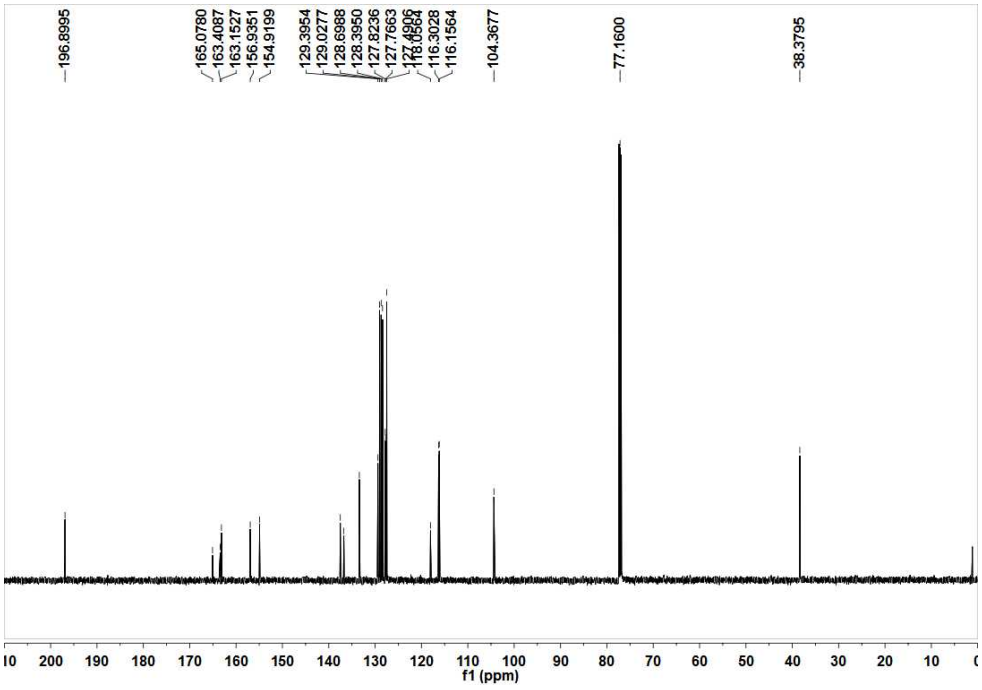
0.0000
2.01
2.01
1.05
7.09
2.03
1.00
2.00
f1 (ppm)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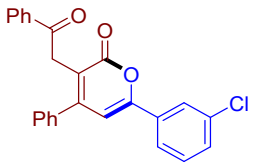
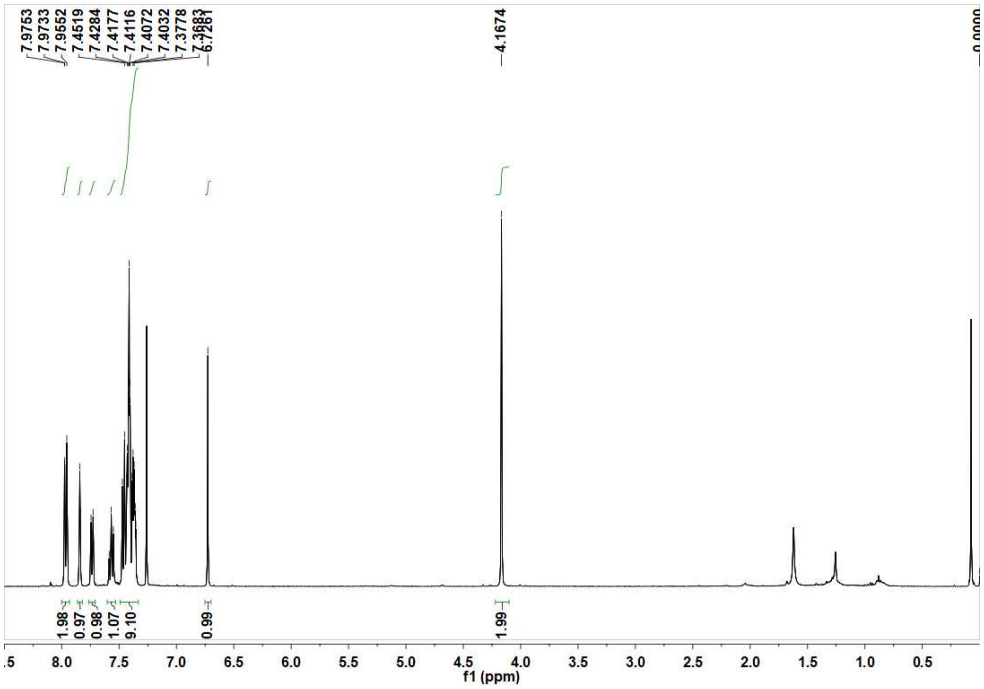
116.3028
116.1564
104.3677
77.1600
38.3795
f1 (ppm)


Ph
O
O
O
Ph
Cl

4f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7.9562
7.4519
7.4284
7.4177
7.4116
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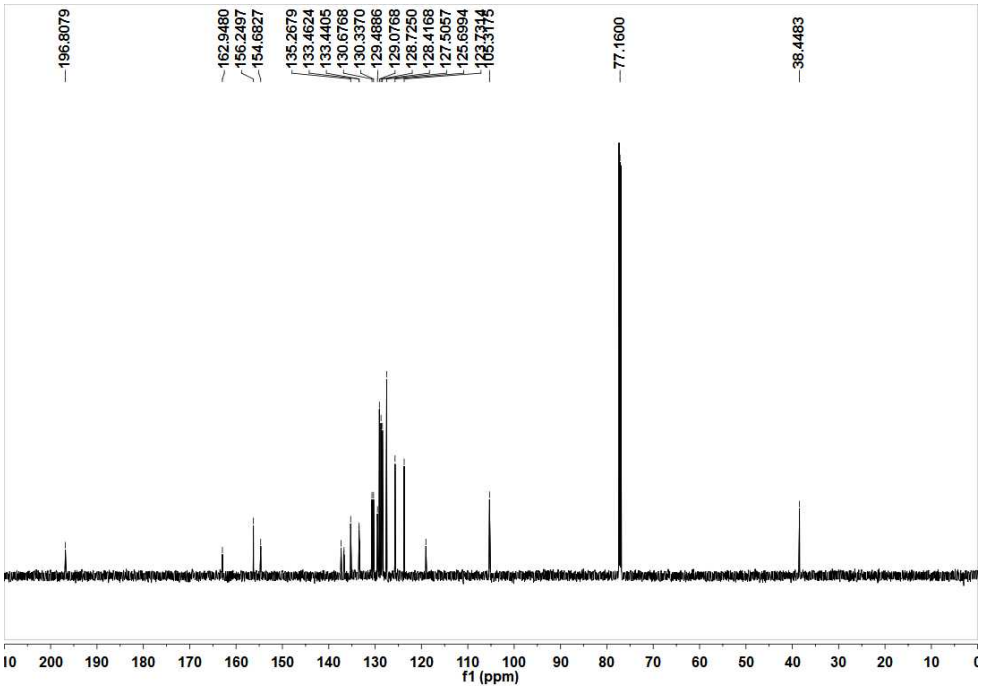
4.1674
1.98
0.97
0.98
1.07
9.10
0.99
1.99
f1 (ppm)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154.6827
135.2679
133.4624
133.4405
130.6768
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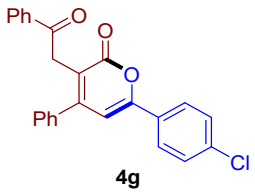
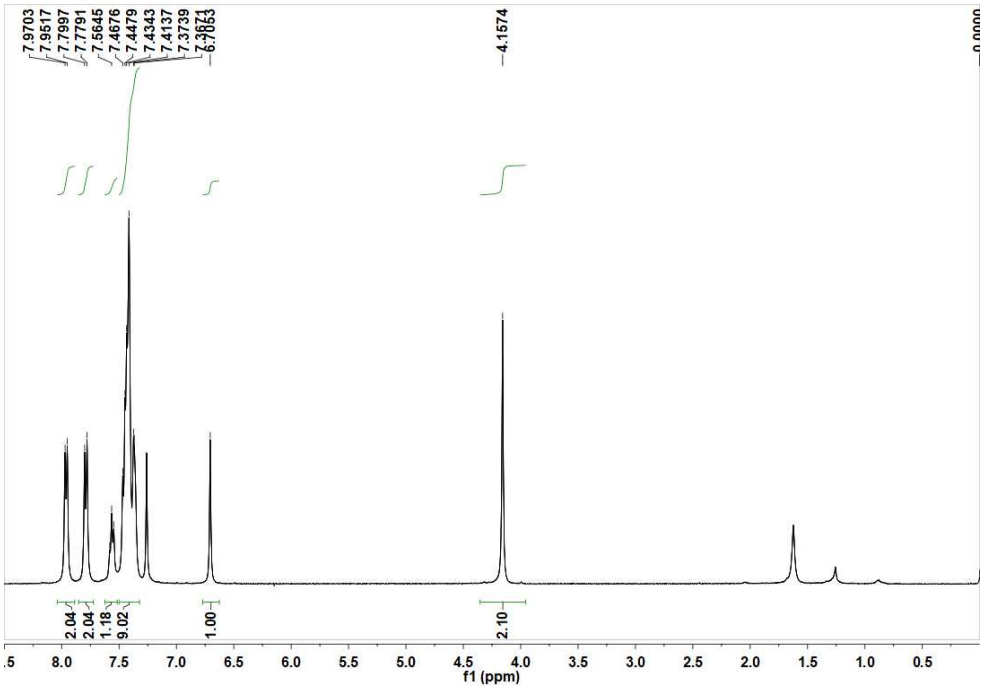
128.7250
128.4168
127.5057
125.6994
77.1600
38.4483
f1 (ppm)


Ph
O
O
O
Ph
Cl

4g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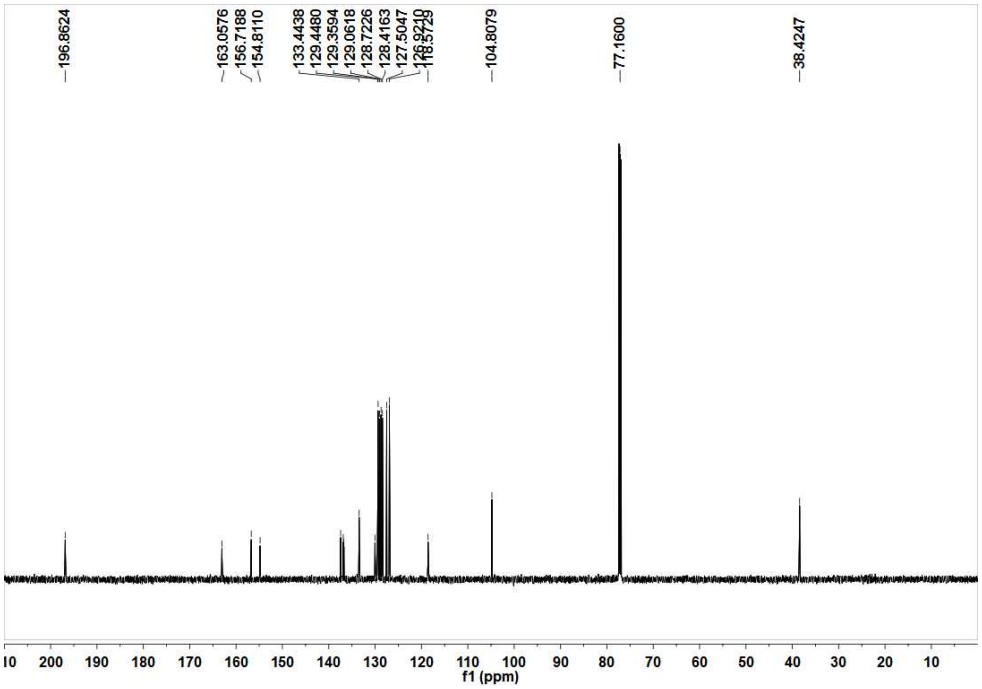
7.7997
7.7791
7.5645
7.4676
7.4479
7.4343
7.4137
7.3739
4.1574
2.04
2.04
1.18
9.02
1.00
2.10
f1 (ppm)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154.8110
133.4438
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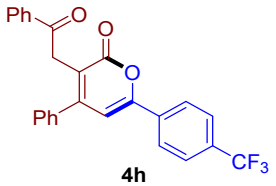
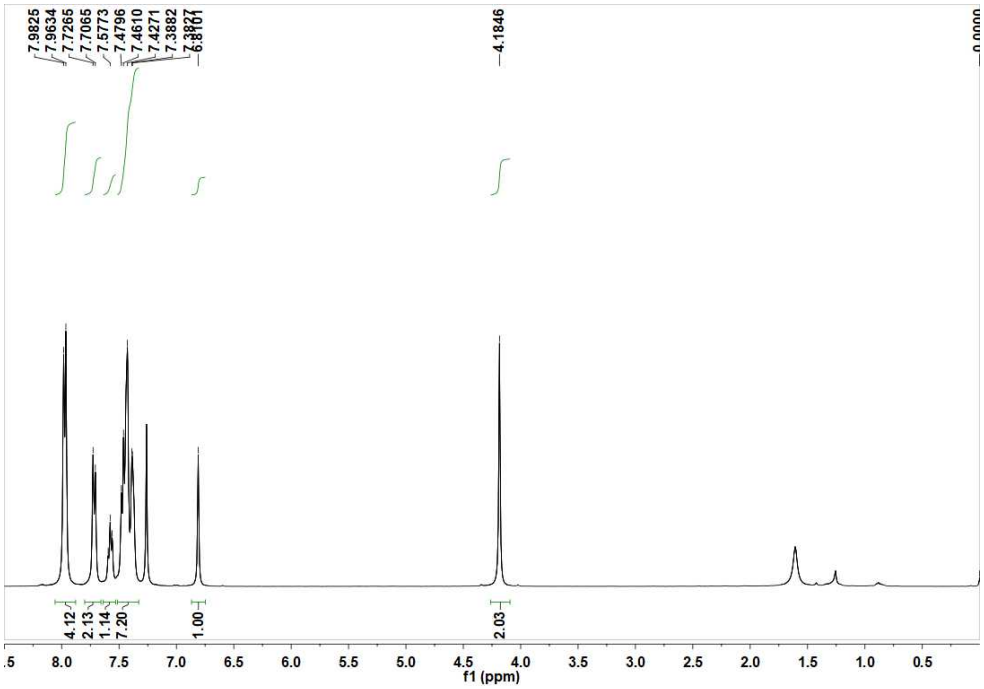
129.0618
128.7226
128.4163
127.5047
104.8079
77.1600
38.4247
f1 (ppm)


Ph
O
O
O
Ph
CF3

4h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7.7265
7.7065
7.5773
7.4796
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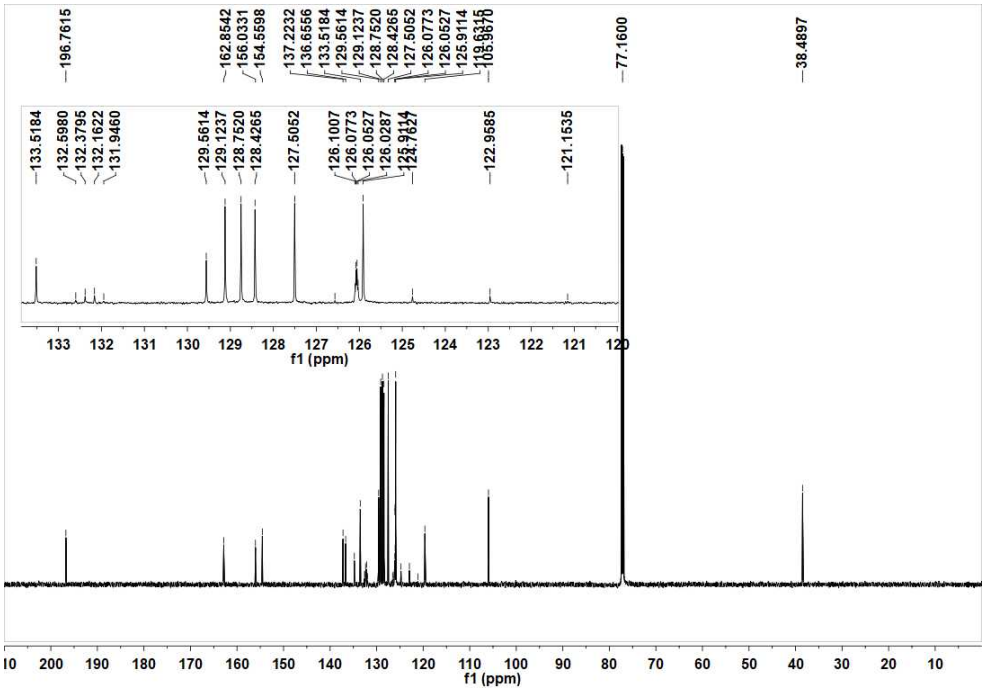
7.4271
7.3882
4.1846
4.12
2.13
1.14
7.20
1.00
2.03
f1 (ppm)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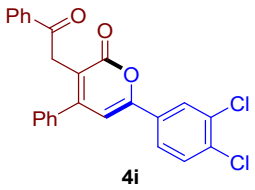
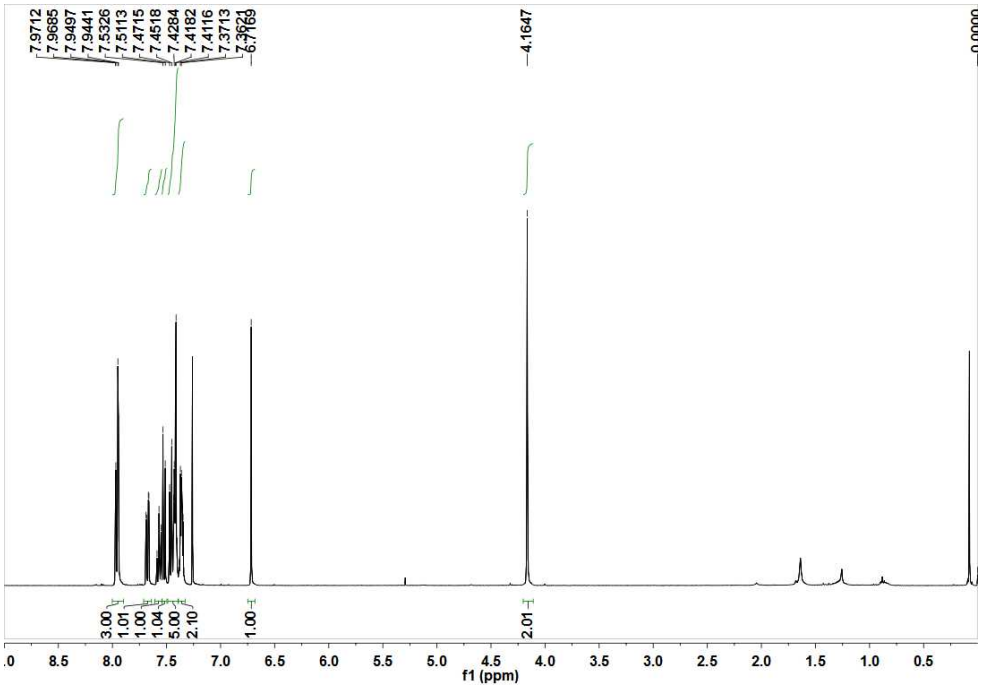
156.0331
154.5598
137.2232
136.6556
133.5184
129.5614
129.1237
128.7520
128.4265
127.5052
126.0773
126.0527
125.9114
77.1600
38.4897
133.5184
132.5980
132.3795
132.1622
131.9460
129.5614
129.1237
128.7520
128.4265
127.5052
126.1007
126.0773
126.0527
126.0287
122.9585
121.1535
f1 (ppm)


Ph
O
O
O
Ph
Cl
Cl

4i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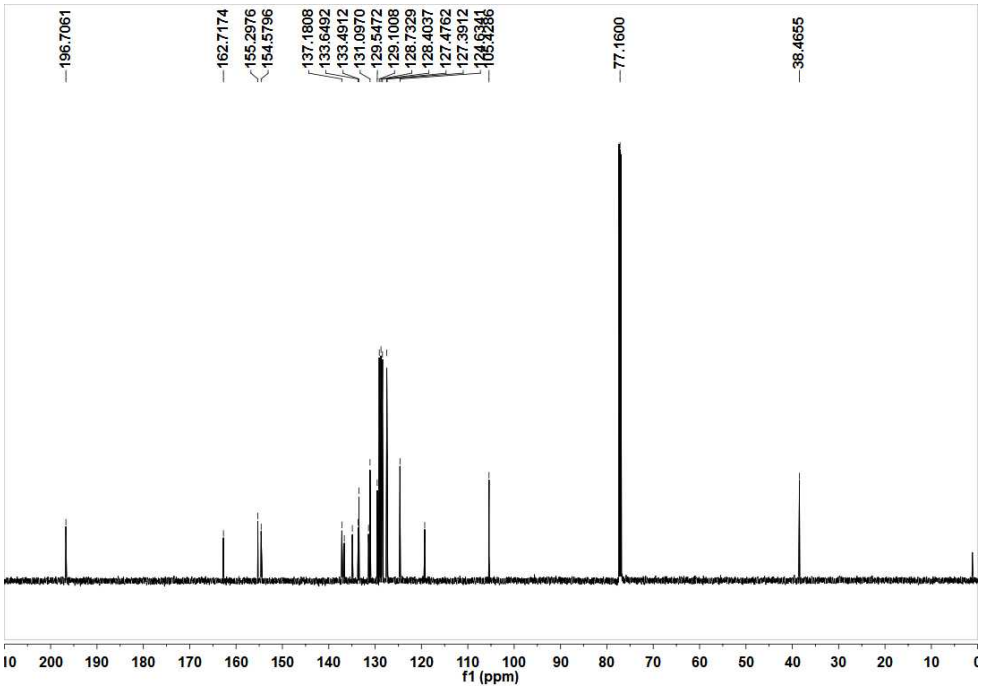
7.9685
7.9497
7.9441
7.5326
7.5113
7.4715
7.4518
7.4284
7.4182
7.4116
7.3713
4.1647
0.0000
3.00
1.01
1.00
1.04
5.00
2.10
1.00
2.01
f1 (ppm)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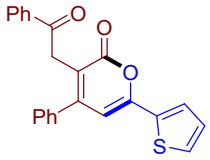
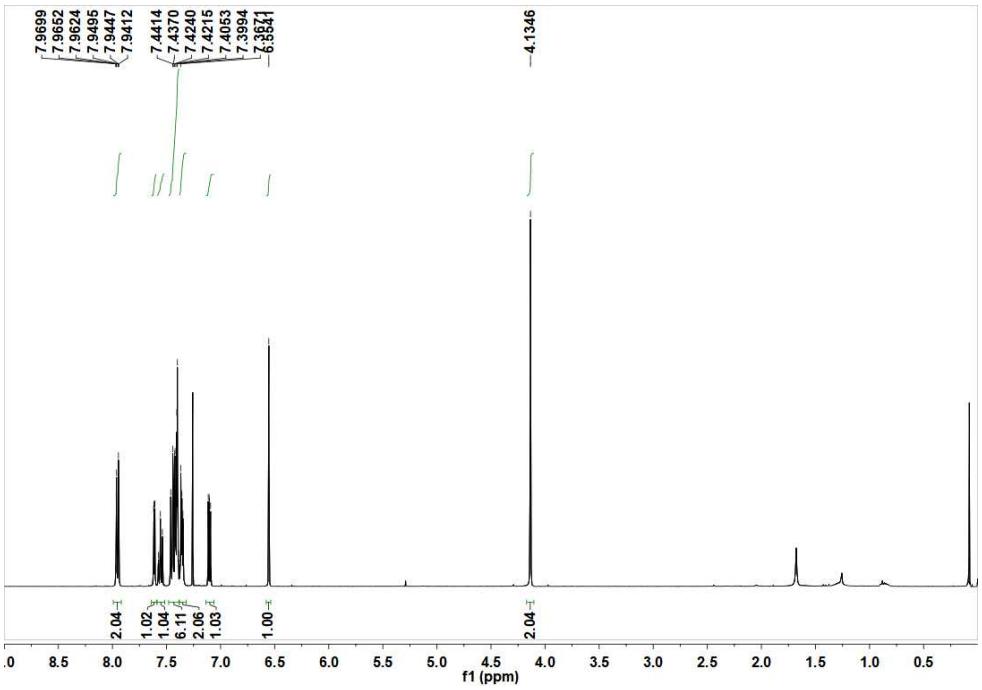
162.7174
155.2976
154.5796
137.1808
133.6492
133.4912
131.0970
129.5472
129.1008
128.7329
128.4037
127.4762
127.3912
77.1600
38.4655
f1 (ppm)


Ph
O
O
O
Ph
S

4j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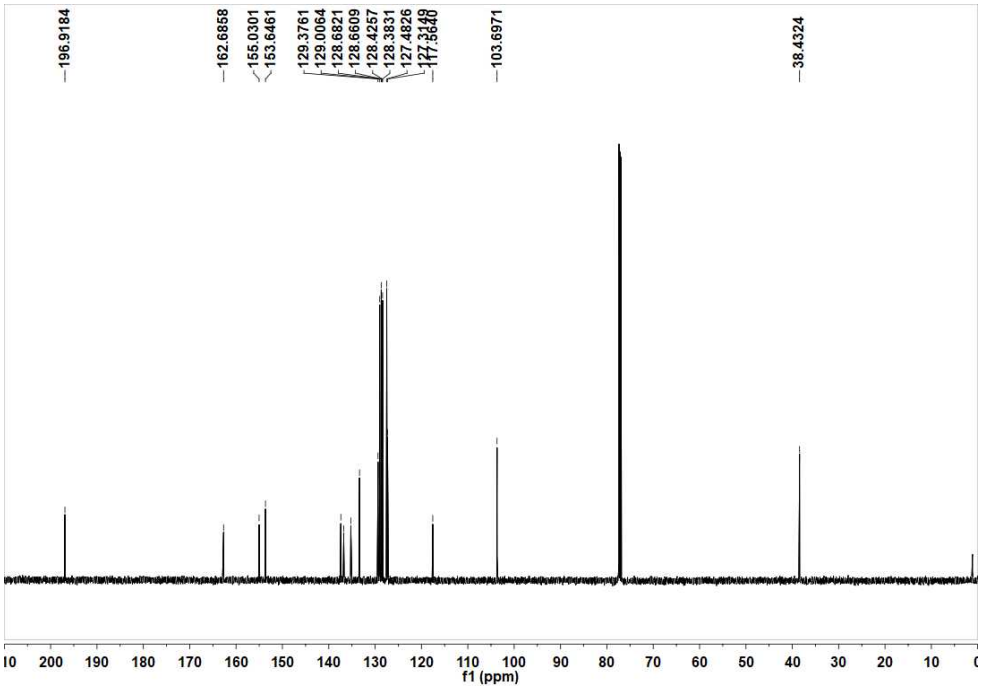
7.9652
7.9624
7.9495
7.9447
7.9412
7.4414
7.4370
7.4240
7.4215
7.4053
7.3994
6.5541
4.1346
2.04
1.02
1.04
6.11
2.06
1.03
1.00
2.04
f1 (ppm)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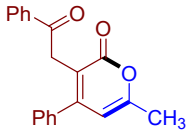
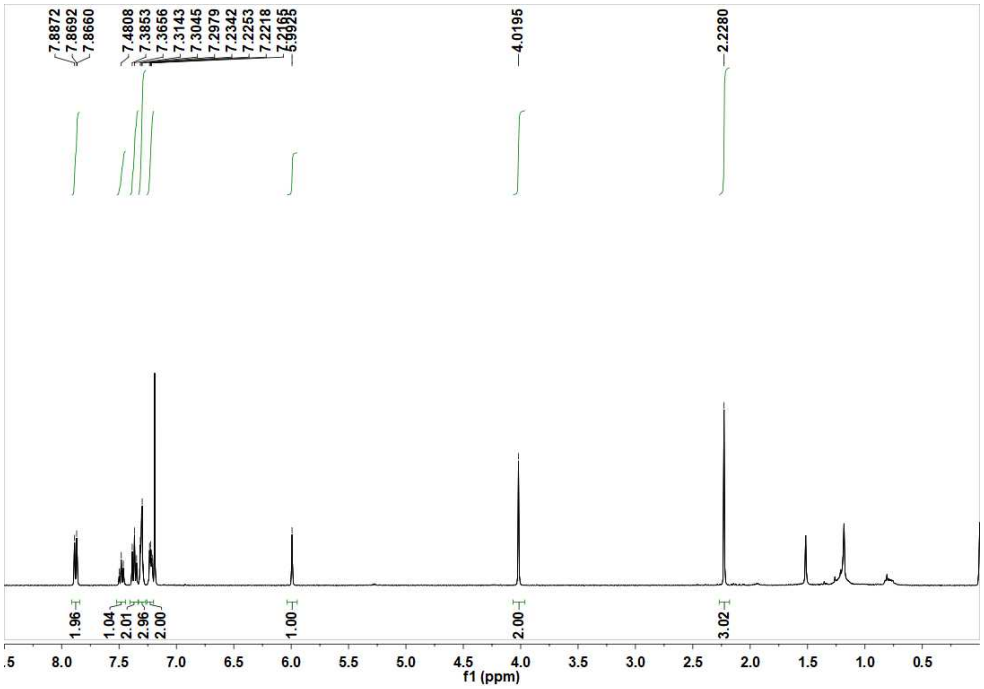
162.6858
155.0301
153.6461
129.3761
129.0064
128.6821
128.6609
128.4257
128.3831
127.4826
103.6971
38.4324
f1 (ppm)


Ph
O
O
O
Ph
CH3

4k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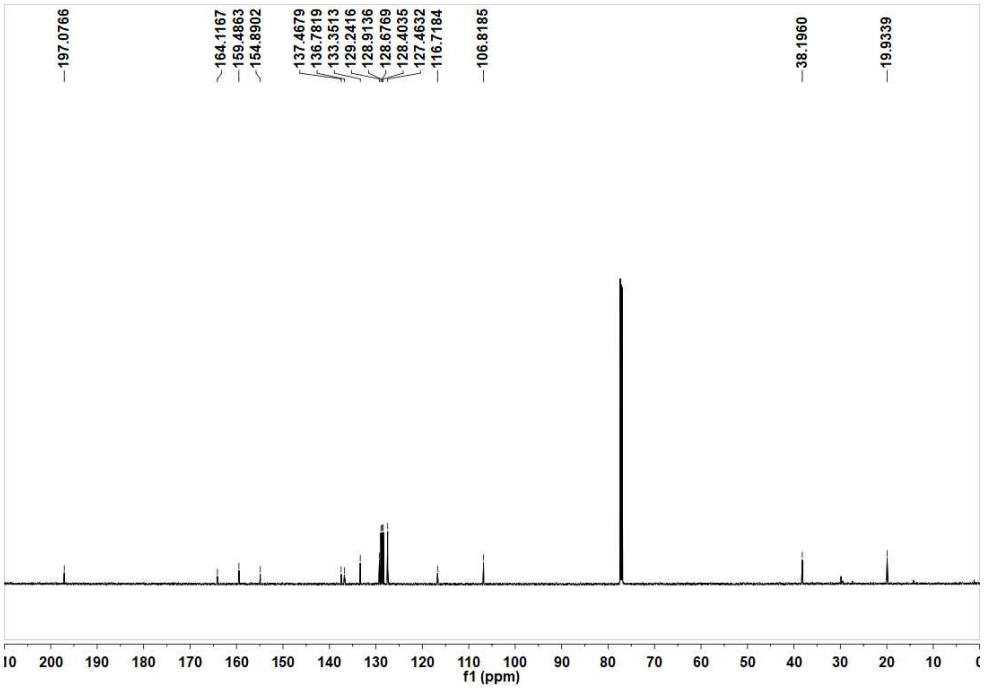
7.8660
7.4808
7.3853
7.3656
7.3143
7.3045
7.2979
7.2342
7.2253
7.2218
4.0195
2.2280
1.96
1.04
2.01
2.96
2.00
1.00
2.00
3.02
f1 (ppm)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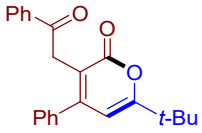
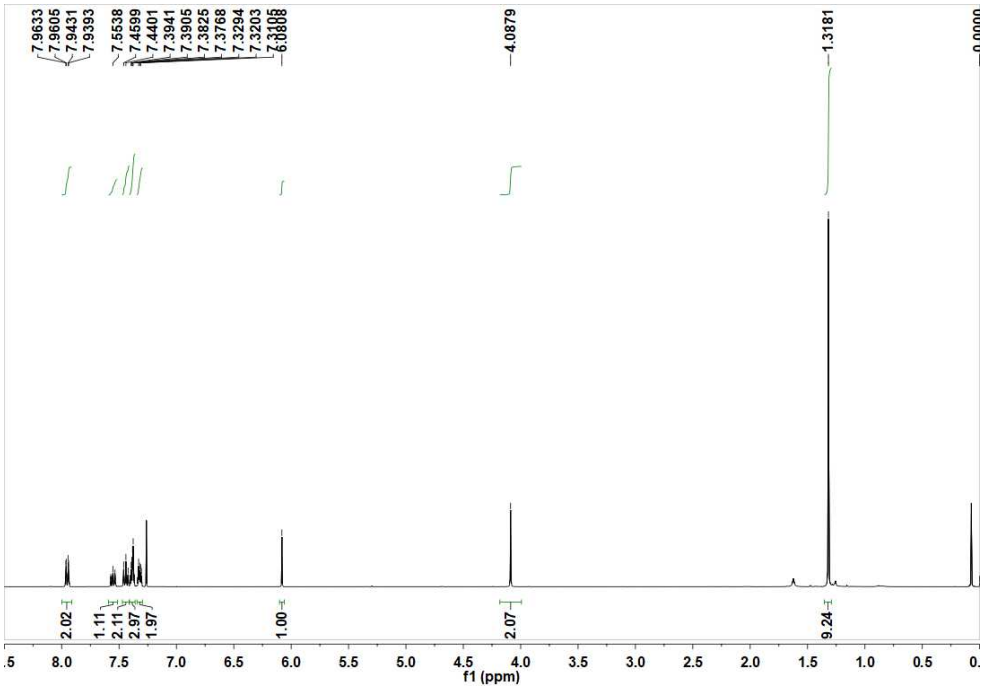
164.1167
159.4863
154.8902
137.4679
136.7819
133.3513
129.2416
128.9136
128.6769
128.4035
127.4632
116.7184
106.8185
38.1960
19.9339
f1 (ppm)


Ph
O
O
O
Ph
t-Bu

4l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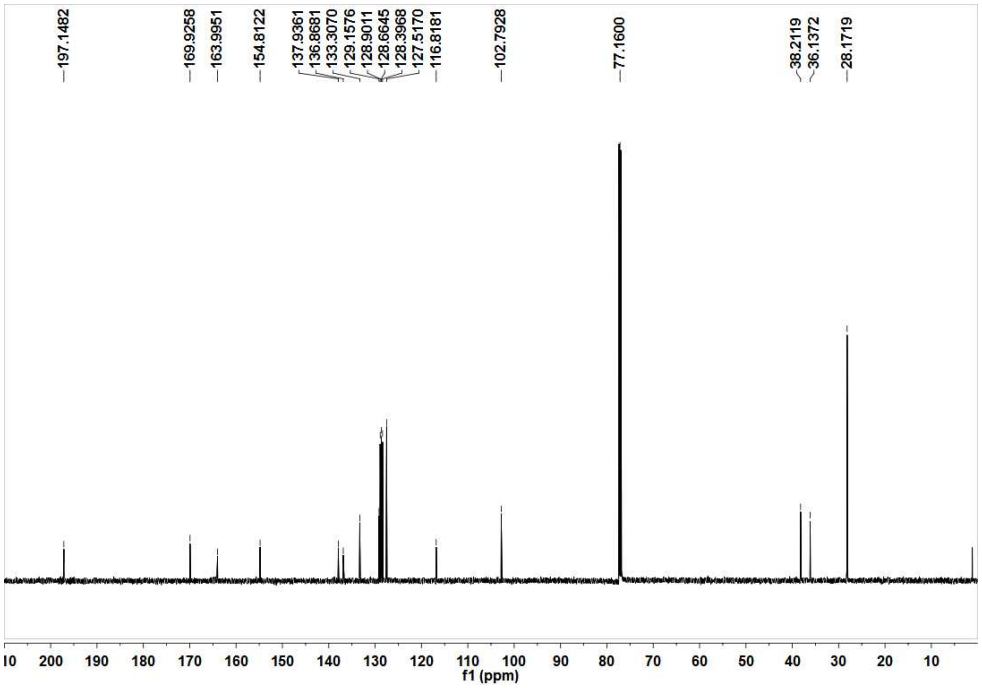
7.9605
7.9431
7.9393
7.5538
7.4599
7.4401
7.3941
7.3905
7.3825
7.3768
7.3294
7.3203
6.0808
4.0879
1.3181
2.02
1.11
2.11
2.97
1.97
1.00
2.07
9.24
f1 (ppm)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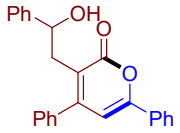
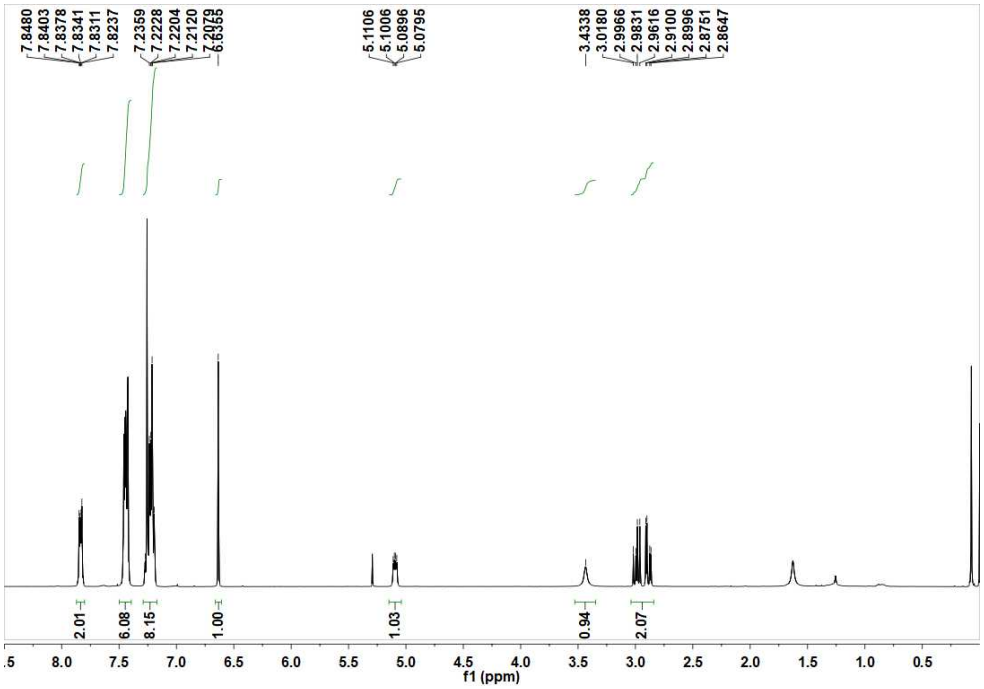
163.9951
154.8122
137.9361
136.8681
133.3070
129.1576
128.9011
128.6645
128.3968
127.5170
116.8181
102.7928
77.1600
38.2119
36.1372
28.1719
f1 (ppm)


Ph
OH
O
O
Ph
Ph

5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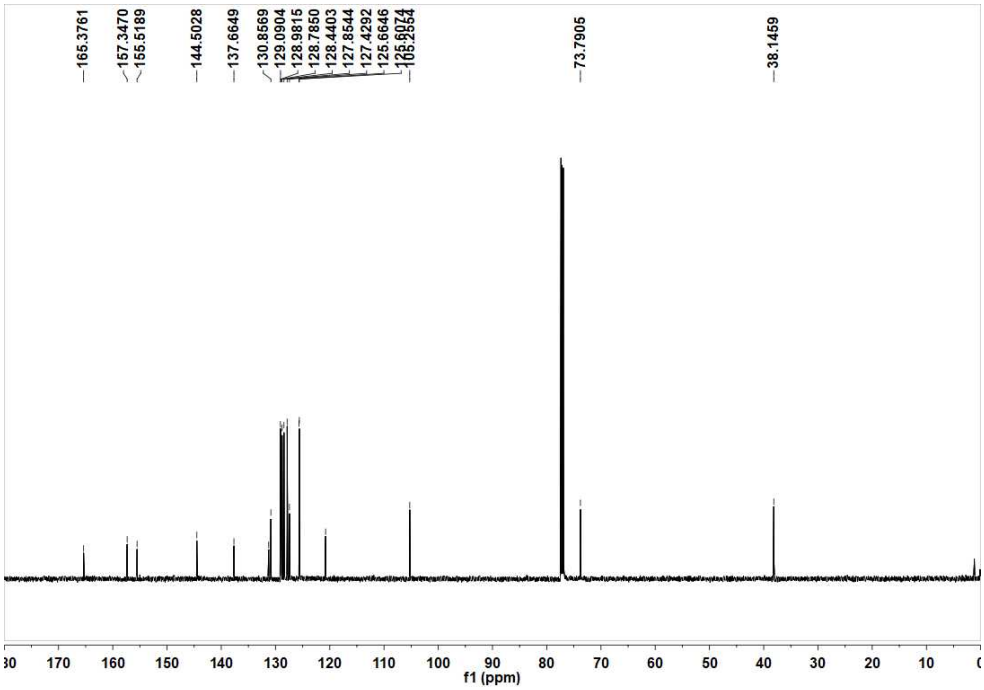
7.8403
7.8378
7.8341
7.8311
7.8237
7.2359
7.2228
7.2204
7.2120
5.1106
5.1006
5.0896
5.0795
3.4338
3.0180
2.9966
2.9831
2.9616
2.9100
2.8996
2.8751
2.8647
2.01
6.08
8.15
1.00
1.03
0.94
2.07
f1 (ppm)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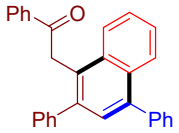
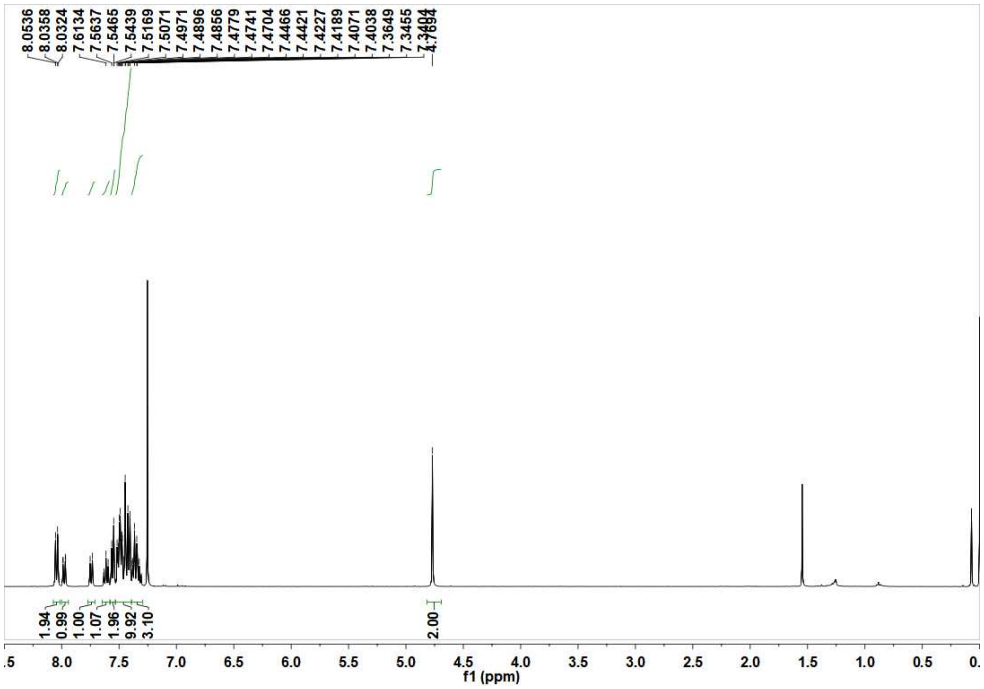
157.3470
155.5189
144.5028
137.6649
130.8569
129.0904
128.9815
128.7850
128.4403
127.8544
127.4292
125.6646
73.7905
38.1459
f1 (ppm)


Ph
O
Ph
Ph

6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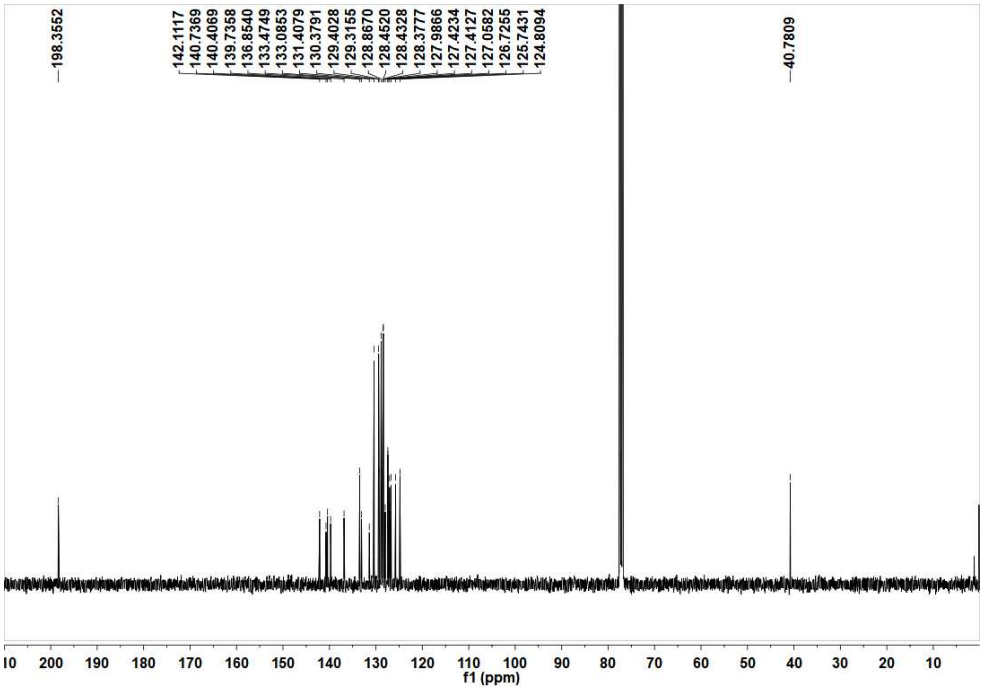
8.0358
8.0324
7.6134
7.5637
7.5465
7.5439
7.5169
7.5071
7.4971
7.4896
7.4856
7.4779
7.4741
7.4704
7.4466
7.4421
7.4227
7.4189
7.4071
7.4038
7.3649
7.3455
1.94
0.99
1.00
1.07
1.96
9.92
3.10
2.00
f1 (ppm)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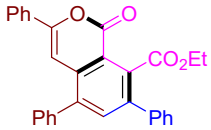
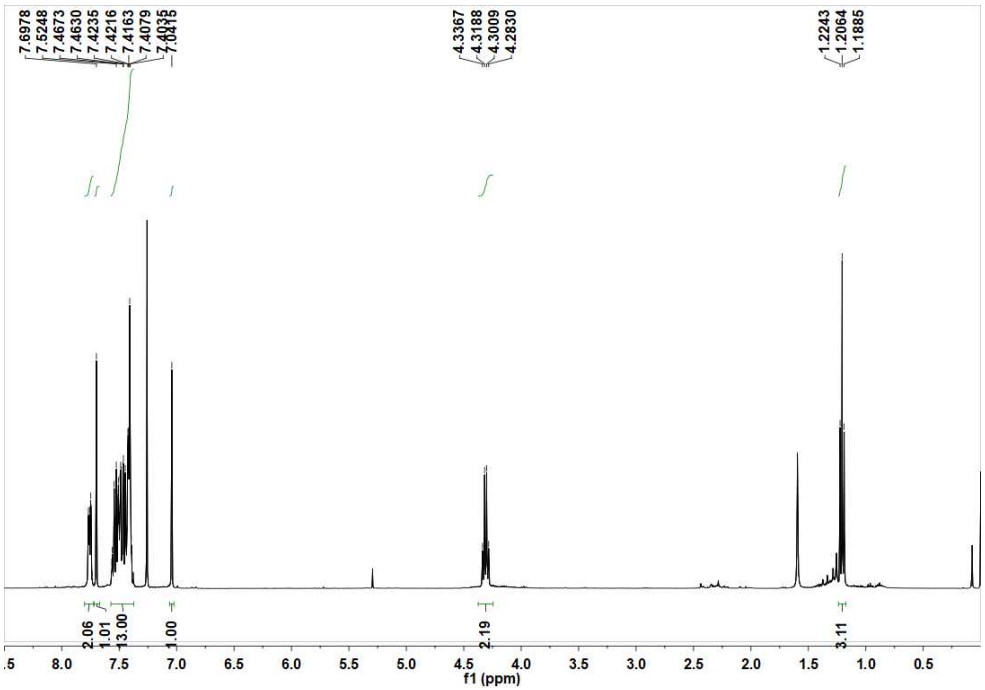
140.7369
140.4069
139.7358
136.8540
133.4749
133.0853
131.4079
130.3791
129.4028
129.3155
128.8670
128.4520
128.4328
128.3777
127.9866
127.4234
127.4127
127.0582
126.7255
125.7431
124.8094
40.7809
f1 (ppm)


Ph
O
O
CO2Et
Ph
Ph

7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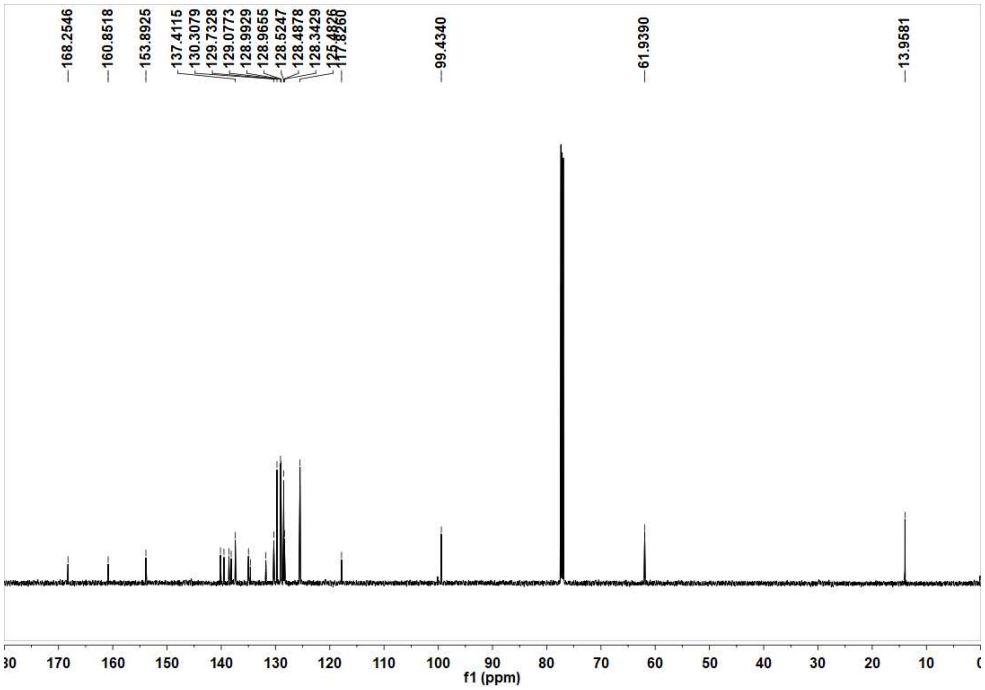
7.4630
7.4235
7.4216
7.4163
7.4079
4.3367
4.3188
4.3009
4.2830
1.2243
1.2064
1.1885
2.06
1.01
13.00
1.00
2.19
3.11
f1 (ppm)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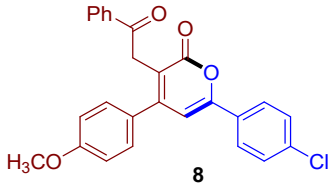
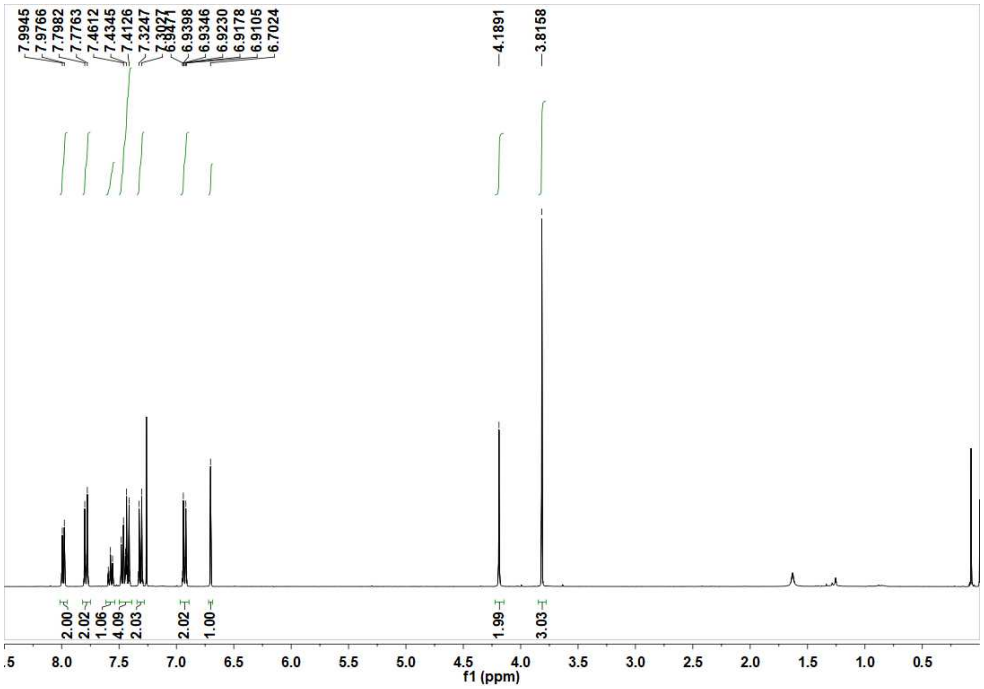
153.8925
137.4115
130.3079
129.7328
129.0773
128.9929
128.9655
128.5247
128.4878
128.3429
99.4340
61.9390
13.9581
f1 (ppm)


Ph
O
O
O
H3CO
Cl

8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7.9766
7.7982
7.7763
7.4612
7.4345
7.4126
7.3247
6.9398
6.9346
6.9230
6.9178
6.9105
6.7024
4.1891
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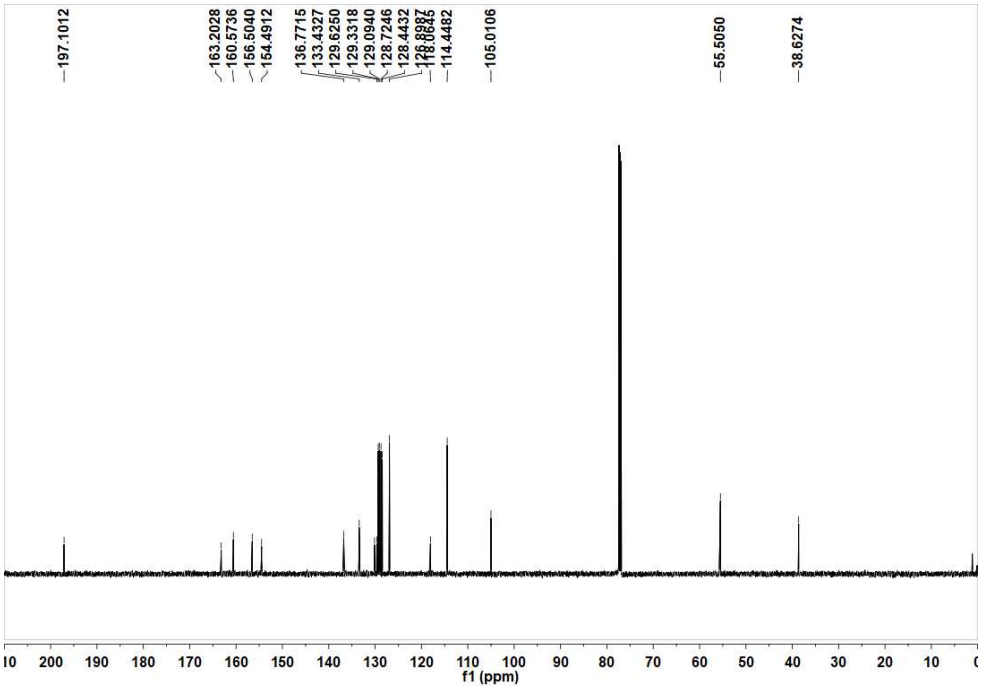
2.02
1.00
1.99
3.03
f1 (ppm)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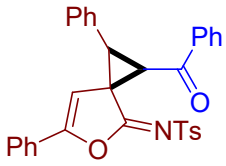
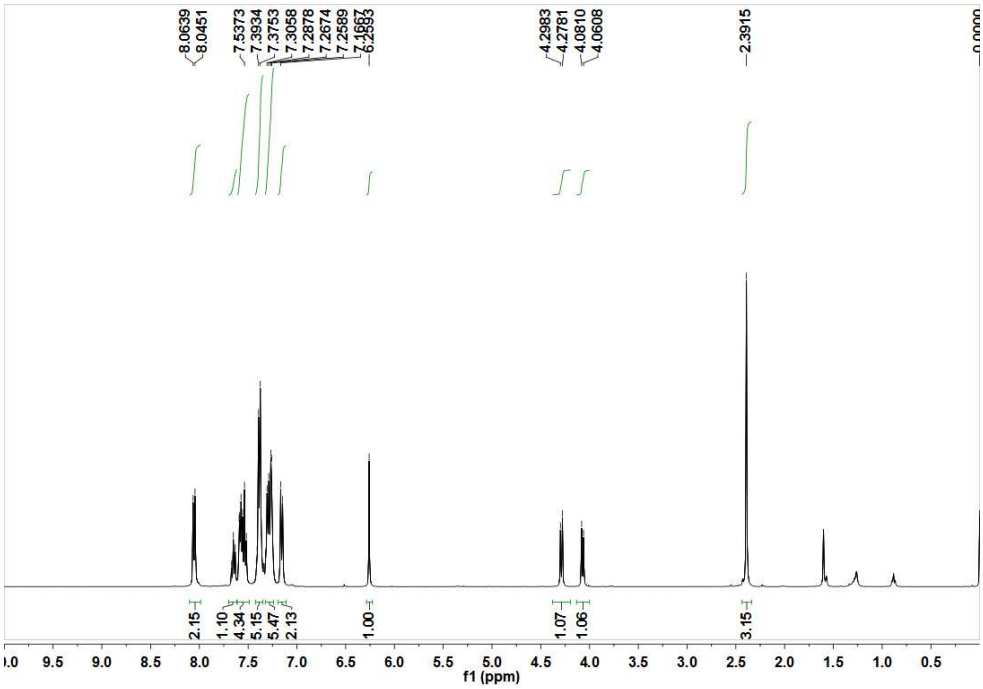
160.5736
156.5040
154.4912
136.7715
133.4327
129.6250
129.3318
129.0940
128.7246
128.4432
114.4482
105.0106
55.5050
38.6274
f1 (ppm)


Ph
Ph
O
NTs
Ph
O

9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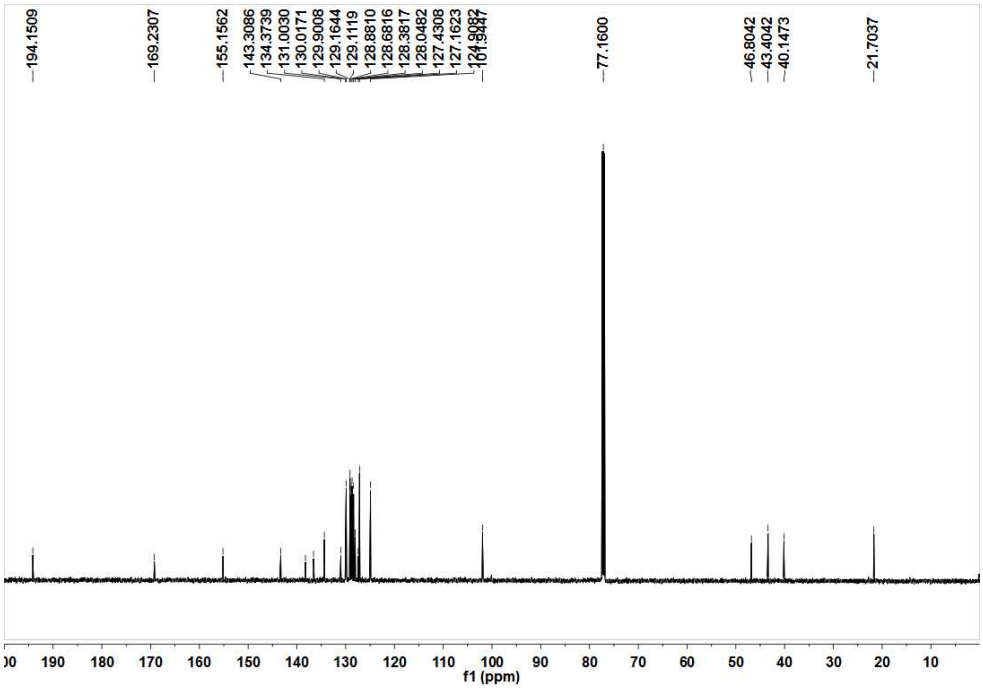
8.0451
7.5373
7.3934
7.3753
7.3058
7.2878
7.2674
7.2589
7.1667
6.2593
4.2983
4.2781
4.0810
4.0608
2.3915
2.15
1.10
4.34
5.15
5.47
2.13
1.00
1.07
1.06
3.15
f1 (ppm)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143.3086
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131.0030
130.0171
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129.1644
129.1119
128.8810
128.6816
128.3817
128.0482
127.4308
127.1623
124.9082
101.9447
77.1600
46.8042
43.4042
40.1473
21.7037
f1 (ppm)

The diastereomer of **9**