Supporting Information（SI）

Synthesis of 2-Alkenylfurans *via* Ag(I)-Catalyzed Tandem Cyclization/Cross-Coupling Reaction of Enynones with Iodonium Ylides

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Content

The X-Ray Crystal Structure of **3da**

Important Crystal Data for 3da. Crystallographic data have been deposited with the Cambridge Crystallographic Data Centre; deposition no.CCDC-1953177.

Bond precision: C-C = 0.0019 A Wavelength=1.54184

Cell:	a=12.1107(2)	b=12.1107(2)	c=52.4973(14)
	alpha=90	beta=90	gamma=90
Temperature:	293 K		

	Calculated	Reported
Volume	7699.7(3)	7699.7(3)
Space group	I 41/a	I 41/a
Hall group	-I 4ad	-I 4ad
Moiety formula	C21 H20 O7	C21 H20 O7
Sum formula	C21 H20 O7	C21 H20 O7
Mr	384.37	384.37
Dx,g cm-3	1.326	1.326
Z	16	16
Mu (mm-1)	0.837	0.837
F000	3232.0	3232.0
F000'	3243.25	
h,k,lmax	14,14,62	14,14,62
Nref	3419	3419
Tmin,Tmax	0.817,0.920	0.174,1.000
Tmin'	0.741	

Correction method= # Reported T Limits: Tmin=0.174 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 1.000 Theta(max)= 66.580

R(reflections)= 0.0431(3204) wR2(reflections)= 0.1122(3419)

S = 1.069 Npar= 258

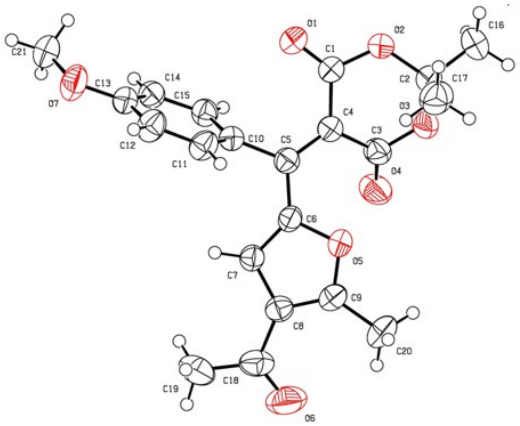


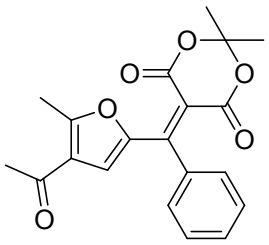
Figure S1 X-ray crystal structure of product **3da**

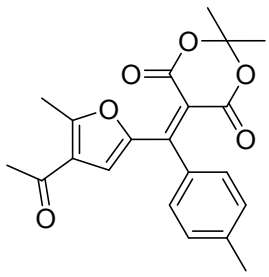
Experimental section

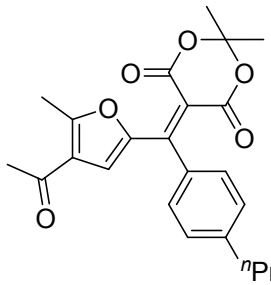
General Methods. All reactions were carried out in a round flask with magnetic stirring. Unless otherwise noted, all reagents were purchased from Aladdin, Energy Chemical for direct use, or prepared as described in the literature. DCM was dried over CaH before use. For chromatographic purification, 200-300 mesh silica gel (Qingdao, China) was employed. For thin layer chromatography (TLC) analysis, Merck 25 TLC aluminium sheets (silica gel 60 GF254, 0.25 mm) were used. ^{1}H NMR and ^{13}C NMR were recorded on Bruker ARX 500 MHz spectrometer in $CDCl_3$ solution and the chemical shift were reported in parts per million (δ) relative to internal standard TMS (0 ppm) and coupling constants are reported as Hertz (Hz). Splitting patterns are designated as singlet (s), broad singlet (bs), doublet (d), triplet (t). Splitting patterns that could not be interpreted or easily visualized are designated as multiple (m). HRMS were performed on Thermo Scientific LTQ Qrbitrap XL mass spectrometer (ESI). Enynones **1,** and iodonium ylides **2,** were known compounds and prepared according to the literature procedures.

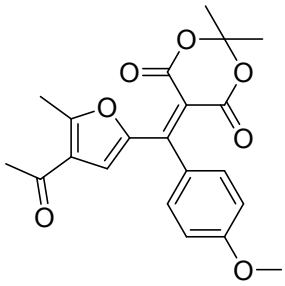
General Procedures. ***Synthesis of 3aa.*** Dissolve 3-(3-phenylprop-2-yn-1-ylidene)pentane-2,4-dione **1a** (0.2 mmol, 42 mg) and 2,2-dimethyl-4-oxo-5 (phenyliodonio)-4*H*-1,3-dioxin-6-olate **2a** (0.24 mmol, 83 mg) with DCM (1 mL). The mixture were syringed dropwise into a vigorous stirring mixture of $AgBF_4$ (0.02 mmol, 5 mg) in DCM (1 mL) at -40 ^{o}C under air atmosphere. After stirring for 20 minutes, the reaction mixture was concentrated in vacuum, and the residue was purified by flash chromatography (eluted with petroleum ether/ethyl acetate = 3:1), providing product **3aa**.

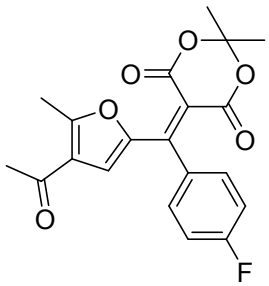
1 gram scale for one representative example: Synthesis of 3aa. Dissolve 3-(3-phenylprop-2-yn-1-ylidene)pentane-2,4-dione **1a** (2.41 mmol, 511 mg) and 2,2-dimethyl-4-oxo-5 (phenyliodonio)-4*H*-1,3-dioxin-6-olate **2a** (2.89 mmol, 1 g) with DCM (5 mL). The mixture were syringed dropwise into a vigorous stirring mixture of $AgBF_4$ (0.24 mmol, 46 mg) in DCM (5 mL) at -40 ^{o}C under air atmosphere. After stirring for 60 minutes, the reaction mixture was concentrated in vacuum, and the residue was purified by flash chromatography (eluted with petroleum ether/ethyl acetate = 3:1), providing **3aa** (682 mg, 80%).

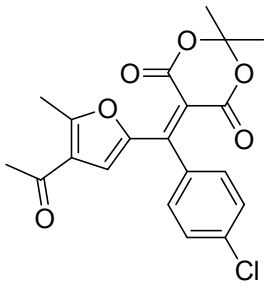
5-((4-acetyl-5-methylfuran-2-yl)(phenyl)methylene)-2,2-dimethyl-1, 3-dioxane-4,6-dione (3aa).  Yellow solid (66 mg, 94%), R_f = 0.38 (PE : EA = 3 : 1), mp 130-132 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.59-7.39 (m, 5H), 6.95 (s, 1H), 2.69 (s, 3H), 2.39 (s, 3H), 1.88 (s, 6H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 193.0, 164.2, 161.4, 160.1, 153.6, 149.6, 135.9, 131.2, 130.9, 127.9, 124.7, 124.6, 111.7, 104.0, 29.0, 27.3, 15.0. HRMS (ESI) $[C_{20}H_{18}O_6 + Na]^+$ m/z calcd for 377.0996, found 377.0995.

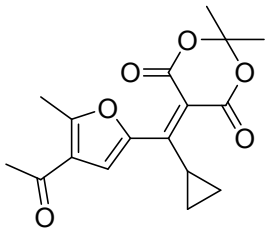
5-((4-acetyl-5-methylfuran-2-yl)(p-tolyl)methylene)-2,2-dimethyl-1,3-dioxane-4,6-dione (3ba).  Yellow solid (57 mg, 78%), R_f = 0.22 (PE : EA = 3 : 1), mp 139-141 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.34-7.27 (m, 4H), 6.91 (s, 1H), 2.70 (s, 3H), 2.48 (s, 3H), 2.40 (s, 3H) 1.88 (s, 6H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 193.0, 164.2, 161.8, 160.2, 154.5, 150.0, 142.4, 133.0, 131.7, 128.6, 124.6, 124.6, 111.0, 103.9, 29.0, 27.2, 21.7, 15.0. HRMS (ESI) $[C_{21}H_{20}O_6 + Na]^+$ m/z calcd for 391.1152, found 391.1151.

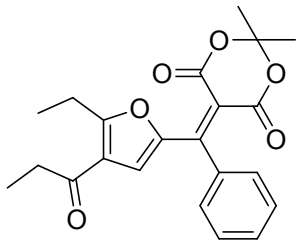
5-((4-acetyl-5-methylfuran-2-yl)(4-propylphenyl)methylene)-2,2-dimethyl-1,3-dioxane-4,6-dione (3ca).  Yellow solid (61 mg, 78%), R_f = 0.63 (PE : EA = 1 : 1), mp 137-139 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.36-7.27 (m, 4H), 6.91 (s, 1H), 2.70 (t, J = 7.5 Hz, 2H), 2.70 (s, 3H), 2.40 (s, 3H), 2.40 (s, 3H) 1.87 (s, 6H), 1.76-1.71 (m, 2H), 1.01 (t, J = 7.5 Hz, 3H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 193.0, 164.1, 161.8, 160.2, 154.5, 150.1, 147.1, 133.1, 131.75, 127.9, 124.7, 124.5, 111.0, 103.8, 38.1, 29.0, 27.2, 24.1, 15.0, 14.0. HRMS (ESI) $[C_{23}H_{24}O_6 + Na]^+$ m/z calcd for 419.1465, found 419.1464.

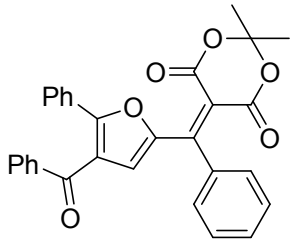
5-((4-acetyl-5-methylfuran-2-yl)(4-methoxyphenyl)methylene)-2,2-dimethyl-1,3-dioxane-4,6-dione (3da).  Yellow solid (61 mg, 80%), R_f = 0.50 (PE : EA = 1 : 1), mp 135-136 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.46-6.96 (m, 4H), 6.86 (s, 1H), 3.91 (s, 3H), 2.70 (s, 3H), 2.39 (s, 3H), 1.86 (s, 6H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 193.0, 164.2, 163.3, 162.2, 160.4, 155.0, 150.5, 134.8, 127.8, 124.7, 124.4, 113.3, 109.5, 103.7, 55.5, 29.0, 27.2, 15.0. HRMS (ESI) $[C_{21}H_{20}O_7 + Na]^+$ m/z calcd for 407.1101, found 407.1100.

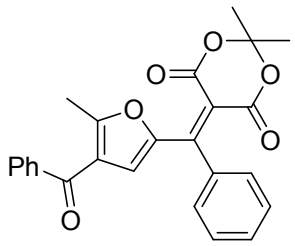
5-((4-acetyl-5-methylfuran-2-yl)(4-fluorophenyl)methylene)-2,2-dimethyl-1,3-dioxane-4,6-dione (3ea).  Yellow solid (61 mg, 82%), R_f = 0.20 (PE : EA = 3 : 1), mp 145-146 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.35-7.07 (m, 4H), 6.83 (s, 1H), 2.60 (s, 3H), 2.30 (s, 3H), 1.78 (s, 6H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 192.9, 165.7, 164.4, 163.7, 161.4, 160.1, 152.6, 149.6, 133.7, 133.6, 131.7, 131.6, 124.8, 124.7, 115.2, 115.1, 111.6, 104.0, 29.0, 27.2, 15.0. HRMS (ESI) $[C_{20}H_{17}FO_6 + Na]^+$ m/z calcd for 395.0901, found 395.0900.

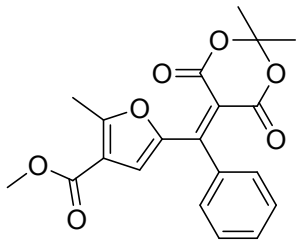
5-((4-acetyl-5-methylfuran-2-yl)(4-chlorophenyl)methylene)-2,2-dimethyl-1,3-dioxane-4,6-dione (3fa).  Yellow solid (60 mg, 78%), R_f = 0.24 (PE : EA = 3 : 1), mp 150-151 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.44 (d, J = 8.5 Hz, 2H), 7.34 (d, J = 8.5 Hz, 2H), 6.96 (s, 1H), 2.68 (s, 3H), 2.39 (s, 3H), 1.86 (s, 6H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 192.9, 164.5, 161.2, 160.0, 152.2, 149.3, 137.6, 134.2, 132.4, 128.2, 124.8, 124.7, 111.8, 104.1, 29.0, 27.3, 15.0. HRMS (ESI) $[C_{20}H_{17}ClO_6 + Na]^+$ m/z calcd for 411.0606, found 411.0605.

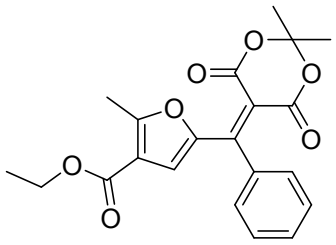
5-((4-acetyl-5-methylfuran-2-yl)(cyclopropyl)methylene)-2,2-dimethyl-1,3-dioxane-4,6-dione (3ga).  Yellow solid (51 mg, 81%), R_f = 0.30 (PE : EA = 3 : 1), mp 128-130 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 6.85 (s, 1H), 3.29-3.25 (m, 1H), 2.58 (s, 3H), 2.42 (s, 3H), 1.81 (s, 6H), 1.27-1.23 (m, 2H), 1.00-0.97 (m, 2H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 193.1, 163.1, 161.2, 160.8, 160.7, 144.5, 123.4, 117.3, 115.1, 104.0, 29.1, 27.1, 16.0, 14.5, 10.2. HRMS (ESI) $[C_{17}H_{18}O_6 + Na]^+$ m/z calcd for 341.0996, found 341.0995.

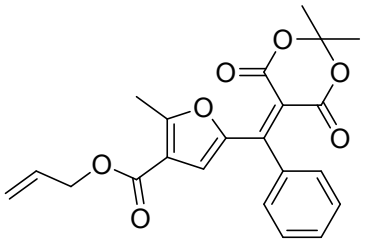
5-((5-ethyl-4-propionylfuran-2-yl)(phenyl)methylene)-2,2-dimethyl-1,3-dioxane-4,6-dione (3ha).  Yellow solid (55 mg, 73%), R_f = 0.70 (PE : EA = 1 : 1), mp 128-130 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.59-7.42 (m, 5H), 6.93 (s, 1H), 3.14-3.12 (q, J = 2.5 Hz, 4H), 2.72-2.70 (q, J = 2.5 Hz, 4H) 1.88 (s, 6H), 1.31-1.28 (m, 3H), 1.16-1.12 (m, 3H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 195.9, 169.0, 161.6, 160.1, 153.5, 149.6, 135.8, 131.2, 131.1, 127.8, 124.4, 123.4, 111.5, 103.9, 34.4, 27.2, 22.4, 11.6, 7.6. HRMS (ESI) $[C_{22}H_{22}O_6 + Na]^+$ m/z calcd for 405.1309, found 405.1308.

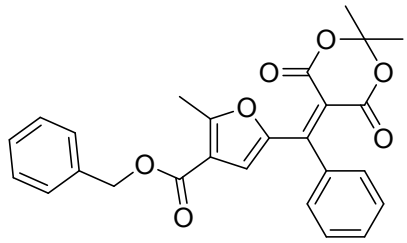
5-((4-benzoyl-5-phenylfuran-2-yl)(phenyl)methylene)-2,2-dimethyl-1,3-dioxane-4,6-dione (3ia). 
Yellow solid (79 mg, 83%), R_f = 0.49 (PE : EA = 2 : 1), mp 146-148 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.86-7.74 (m, 4H), 7.57-7.49 (m, 6H), 7.42-7.35 (m, 5H) 6.95 (s, 1H), 1.92 (s, 6H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 190.5, 161.7, 160.3, 160.1, 152.9, 150.2, 137.0, 135.8, 133.6, 131.2, 131.0, 130.8, 129.8, 128.8, 128.6, 128.4, 128.3, 127.8, 127.6, 124.0, 112.0, 104.1, 27.3. HRMS (ESI) $[C_{30}H_{22}O_6 + Na]^+$ m/z calcd for 501.1309, found 501.1308.

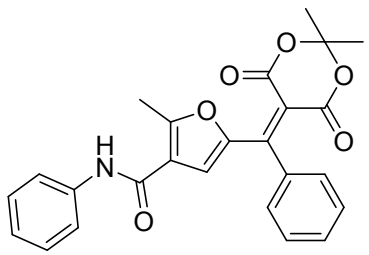
5-((4-benzoyl-5-methylfuran-2-yl)(phenyl)methylene)-2,2-dimethyl-1,3-dioxane-4,6-dione (3ja). 
Yellow solid (63 mg, 75%), R_f = 0.49 (PE : EA = 2 : 1), mp 139-140 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.96-7.94 (m, 2H), 7.61-7.58(m, 1H), 7.53-7.49 (m, 5H), 7.47-7.46 (m, 2H), 7.03 (s, 1H), 2.42 (s, 3H), 1.88 (s, 6H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 193.0, 161.6, 161.1, 160.0, 153.0, 150.2, 135.7, 131.3, 131.2, 131.0, 129.3, 128.6, 128.6, 127.9, 126.4, 125.1, 112.2, 104.1, 29.8, 27.2. HRMS (ESI) $[C_{25}H_{20}O_6$+ Na]$^+$ m/z calcd for 439.1152, found 439.1151.

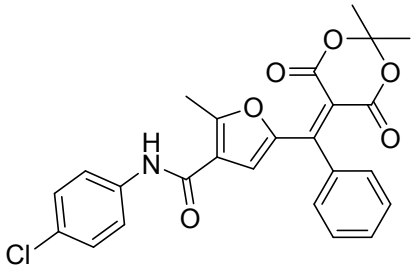
methyl 5-((2,2-dimethyl-4,6-dioxo-1,3-dioxan-5-ylidene)(phenyl)methyl)-2-methylfuran-3-carboxylate (3ka). 
Yellow solid (63 mg, 85%), R_f = 0.50 (PE : EA = 2 : 1), mp 139-142 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.56-7.39 (m, 5H), 6.90 (s, 1H), 3.83 (s, 3H), 2.70 (s, 3H), 1.87 (s, 6H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 164.8, 163.0, 161.5, 160.0, 153.5, 149.9, 135.8, 131.1, 131.0, 127.8, 125.4, 117.3, 111.4, 103.9, 51.8, 27.3, 14.4. HRMS (ESI) $[C_{20}H_{18}O_7$+ Na]$^+$ m/z calcd for 393.0945, found 393.0944.

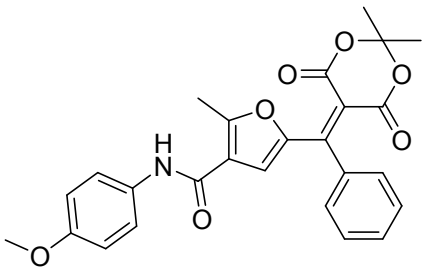
ethyl 5-((2,2-dimethyl-4,6-dioxo-1,3-dioxan-5-ylidene)(phenyl)methyl)-2-methylfuran-3-carboxylate (3la). 
Yellow solid (58 mg, 76%), R_f = 0.73 (PE : EA = 1 : 1), mp 137-140 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.58-7.39 (m, 5H), 6.90 (s, 1H), 4.30 (q, J = 6.5 Hz, 2H), 2.70 (s, 3H), 1.87 (s, 6H) 1.33 (t, J = 7.5 Hz, 3H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 164.7, 162.6, 161.5, 160.1, 153.7, 149.9, 135.8, 131.2, 131.1, 127.8, 125.4, 117.7, 111.4, 103.9, 60.8, 27.3, 14.5, 14.3. HRMS (ESI) $[C_{21}H_{20}O_7$+ Na]$^+$ m/z calcd for 407.1101, found 407.1100.

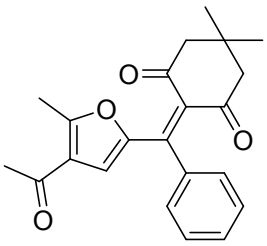
allyl 5-((2,2-dimethyl-4,6-dioxo-1,3-dioxan-5-ylidene)(phenyl)methyl)-2-methylfuran-3-carboxylate. (3ma).  Yellow solid (58 mg, 74%), R_f = 0.27 (PE : EA = 2 : 1), mp 135-137 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.55-7.39 (m, 5H), 6.91 (s, 1H), 6.00-5.92 (m, 1H), 5.37-5.26 (m, 2H), 4.74-4.73 (m, 2H), 2.70 (s,3H), 1.87 (s, 6H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 164.9, 162.2, 161.5, 160.0, 153.6, 149.9, 135.8, 131. 9, 131.2, 131.1, 127.8, 125.3, 118.7, 117.4, 111.5, 103.9, 65.4, 27.3, 14.5. HRMS (ESI) $[C_{22}H_{20}O_7 + Na]^+$ m/z calcd for 419.1101, found 419.1100.

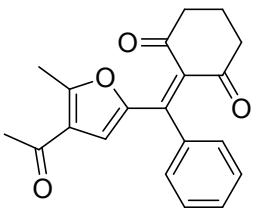
benzyl 5-((2,2-dimethyl-4,6-dioxo-1,3-dioxan-5-ylidene)(phenyl)methyl)-2-methylfuran-3-carboxylate (3na).  Yellow solid (65 mg, 73%), R_f = 0.45 (PE : EA = 2 : 1) mp 147-148 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.58-7.55 (m, 1H), 7.47 (t, *J* = 7.5 Hz, 2H), 7.42-7.37 (m, 7H), 6.94 (s, 1H), 5.30 (s, 2H), 2.71 (s, 3H), 1.89 (s, 6H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 165.0, 162.4, 161.5, 160.0, 153.7, 150.0, 135.8, 135.6, 131.2, 131.1, 128.7, 128.4, 128.4, 127.8, 125.2, 117.4, 111.6, 103.9, 66.5, 27.3, 14.6. HRMS (ESI) $[C_{26}H_{22}O_7 + Na]^+$ m/z calcd for 469.1258, found 469.1257.

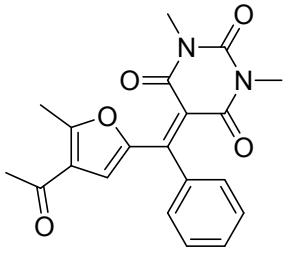
5-((2,2-dimethyl-4,6-dioxo-1,3-dioxan-5-ylidene)(phenyl)methyl)-2-methyl-*N*-phenylfuran-3-carboxamide (3oa).  Yellow solid (62 mg, 72%), R_f = 0.44 (PE : EA = 1 : 1), mp 150-152 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 8.04 (s, 1H), 7.50 (t, *J* = 8.0 Hz, 3H), 7.42 (t, *J* = 8.0 Hz, 2H), 7.31 (t, *J* = 8.0 Hz, 4H), 7.11 (q, *J* = 8.0 Hz, 2H), 2.61 (s, 3H), 1.90 (s, 6H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 164.4, 161.5, 160.6, 160.5, 153.8, 149.2, 137.6, 136.1, 131.1, 130.7, 128.9, 127.9, 124.6, 123.4, 120.4, 120.2, 111.4, 104.1, 27.3, 14.4. HRMS (ESI) $[C_{25}H_{21}NO_6 + Na]^+$ m/z calcd for 454.1261, found 454.1260.

***N*-(4-chlorophenyl)-5-((2,2-dimethyl-4,6-dioxo-1,3-dioxan-5-ylidene)(phenyl)methyl)-2-methylfuran-3-carboxamide (3pa).**  Yellow solid (68 mg, 73%), R_f = 0.63 (PE : EA = 1 : 1), mp 160-161 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 8.08 (s, 1H), 7.48-7.45 (m, 3H), 7.40 (t, *J* = 7.5 Hz, 2H), 7.27-7,25 (m, 5H), 2.61 (s, 3H), 1.91 (s, 6H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 164.6, 161.3, 160.9, 160.5, 153.8, 148.9, 136.3, 136.3, 131.1, 130.4, 129.4, 128.8, 128.0, 123.3, 121.6, 119.8, 111.8, 104.2, 27.3, 14.4. HRMS (ESI) $[C_{25}H_{20}ClNO_6 + Na]^+$ m/z calcd for 488.0871, found 488.0870.

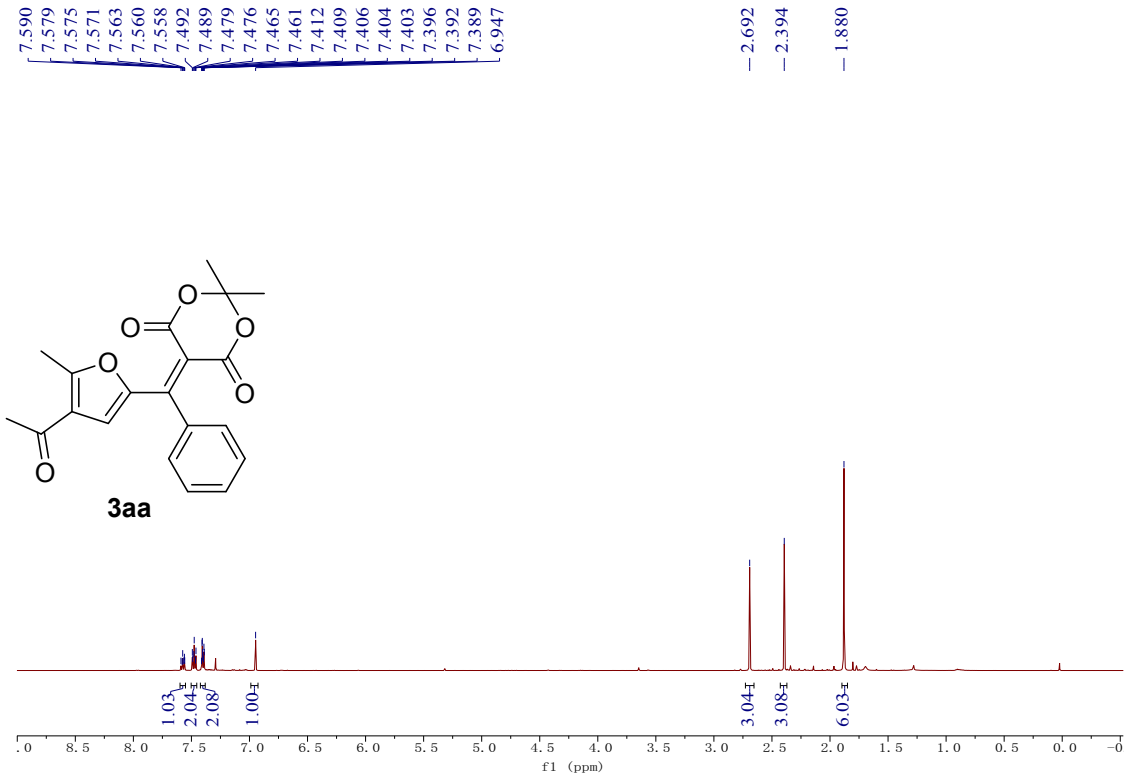
5-((2,2-dimethyl-4,6-dioxo-1,3-dioxan-5-ylidene)(phenyl)methyl)-*N*-(4-methoxyphenyl)-2-methylfuran-3-carboxamide (3qa). 
Yellow solid (68 mg, 74%), R_f = 0.37 (PE : EA = 1 : 1), mp 146-148 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.97 (s, 1H), 7.51-7.47 (m, 1H), 7.42-7.38 (m, 4H), 7.06 (s, 1H), 6.84-6.81 (m, 2H), 3.79 (s, 3H), 2.61 (s, 3H), 1.88 (s, 6H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 164.2, 161.5, 160.6, 160.5, 156.6, 153.8, 149.2, 136.2, 131.1, 130.7, 130.6, 127.8, 123.5, 122.3, 120.2, 114.0, 111.2, 104.0, 55.5, 27.3, 14.4. HRMS (ESI) $[C_{26}H_{23}NO_7 + Na]^+$ m/z calcd for 484.1367, found 484.1365.

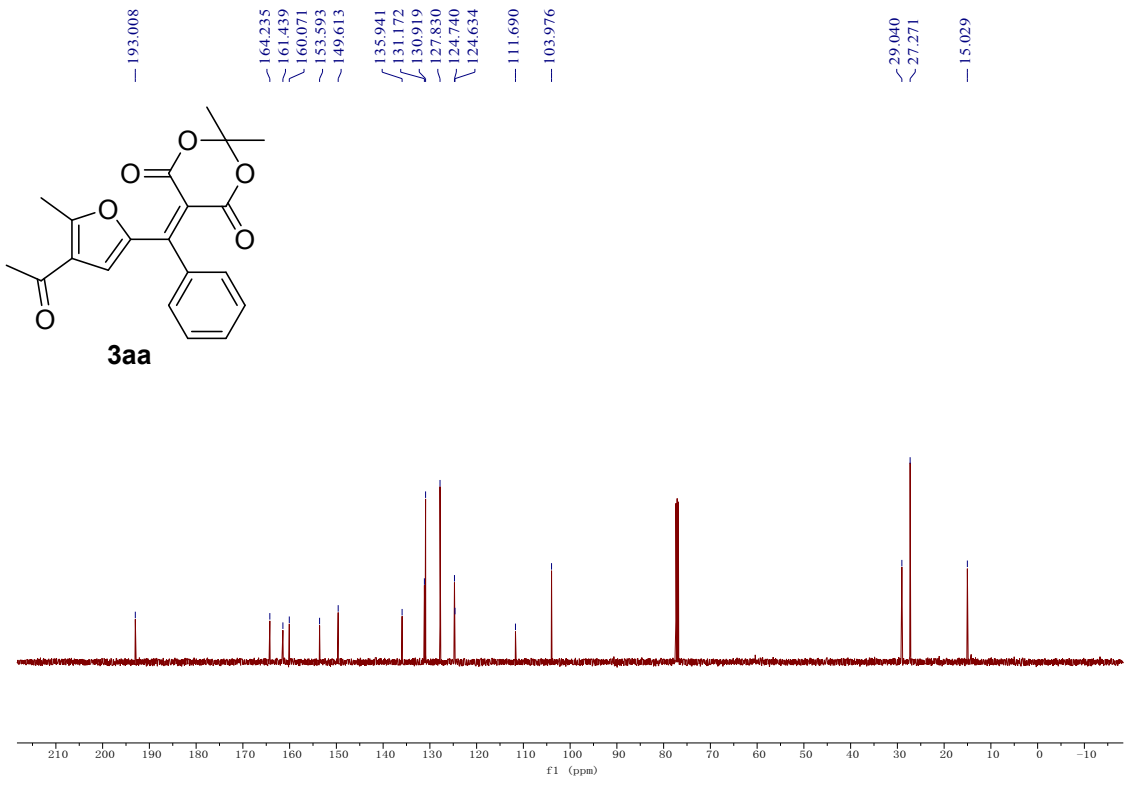
2-((4-acetyl-5-methylfuran-2-yl)(phenyl)methylene)-5,5-dimethylcyclohexane-1,3-dione (3ab). 
Yellow solid (63 mg, 90%), R_f = 0.31 (PE : EA = 3 : 1), mp 157-159 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.50-7.47 (m, 1H), 7.42-7.39 (m, 2H), 7.30-7.29 (m, 2H), 6.76 (s, 1H), 2.75 (s, 2H), 2.62 (s, 3H), 2.55 (s, 2H), 2.37 (s, 3H), 1.16 (s, 6H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 198.6, 196.4, 193.4, 162.3, 150.5, 145.3, 137.3, 133.7, 130.8, 130. 0, 127.7, 123.3, 122.1, 55.2, 54.7, 30.2, 29.0, 28.8, 14.8. HRMS (ESI) $[C_{22}H_{22}O_4 + H]^+$ m/z calcd for 351.1591, found 351.1590.

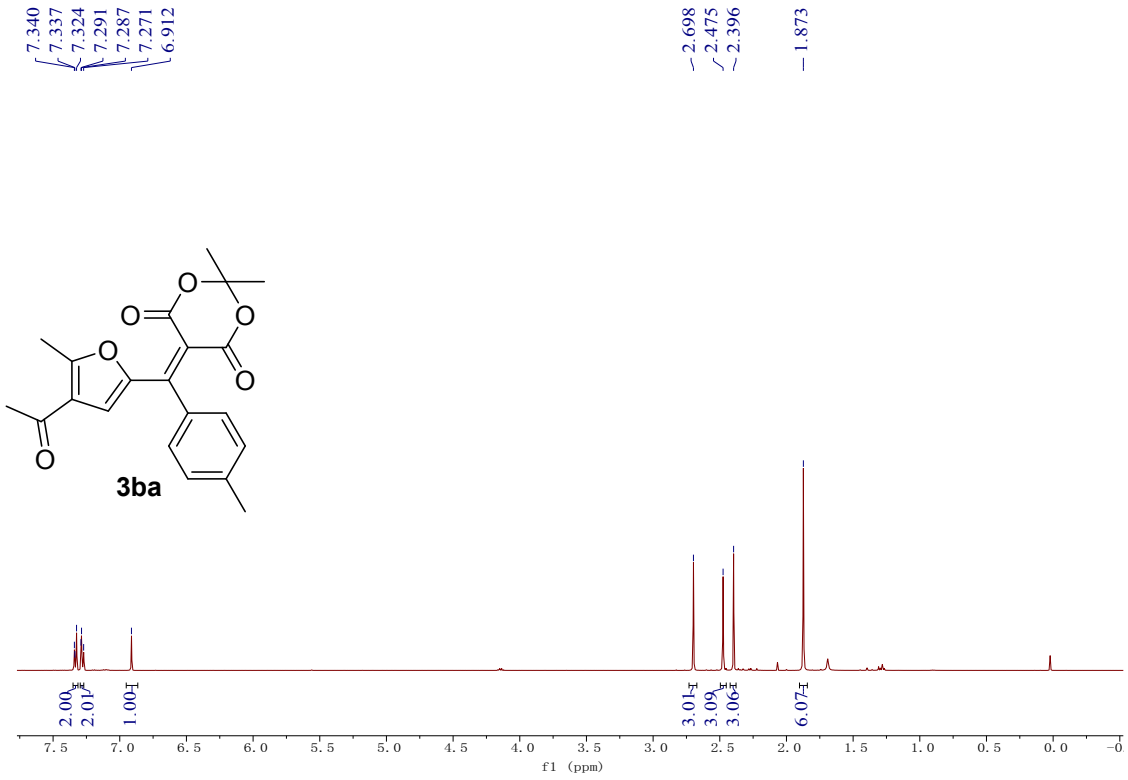
2-((4-acetyl-5-methylfuran-2-yl)(phenyl)methylene)cyclohexane-1,3-dione (3ac). 
Yellow solid (51 mg, 80%), R_f = 0.27 (PE : EA = 3 : 1), mp 148-150 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.51-7.48 (m, 1H), 7.43-7.40 (m, 2H), 7.31-7.29 (m, 2H), 6.76 (s, 1H), 2.82 (t, *J* = 6.5 Hz, 2H), 2.63 (s, 3H), 2.59 (t, *J* = 6.5 Hz, 2H), 2.37 (s, 3H), 2.16-2.10 (m, 2H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 199.6, 196.9, 193.4, 162.4, 150.4, 145.7, 137.2, 134.6, 130.7, 130.0, 127.7, 124.0, 122.2, 40.9, 40.2, 29.0, 17.9, 14.8. HRMS (ESI) $[C_{20}H_{18}O_4 + H]^+$ m/z calcd for 323.1278, found 323.1277.

5-((4-acetyl-5-methylfuran-2-yl)(phenyl)methylene)-1,3-dimethylpyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (3ac). 
Yellow solid (63 mg, 86%), R_f = 0.47 (PE : EA = 2 : 1), mp 149-150 °C. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.62-7.59 (m, 1H), 7.47 (t, *J* = 7.5 Hz, 2H), 7.39-7.29 (d, *J* = 7.5 Hz, 2H), 6.80 (s, 1H), 3.40 (s, 3H), 3.62 (s, 3H), 2.71 (s, 3H), 2.38 (s, 3H). ^{13}C NMR (125.8 MHz, $CDCl_3$, ppm) δ 193.0, 164.6, 161.1, 160.9, 157.7, 151.5, 151.4, 138.6, 131.8, 131.6, 128.0, 125.0, 124.8, 113.7, 29.0, 28.8, 28.7, 15.1. HRMS (ESI) $[C_{20}H_{18}N_2O_5 + H]^+$ m/z calcd for 367.1288, found 367.1288.

^{1}H and ^{13}C NMR spectra of 3aa-3ra, 3ab-3ad



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7.291
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7.271
6.912
2.698
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1.873
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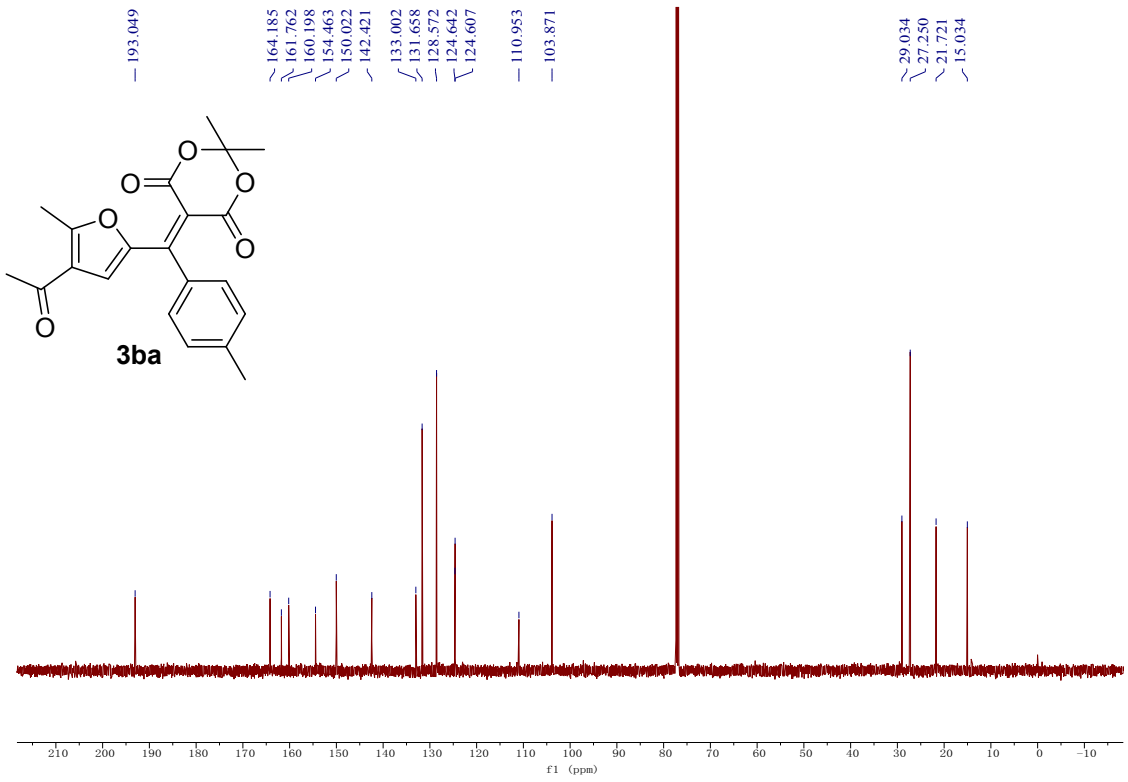
3.01
3.09
3.06
6.07
f1 (ppm)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142.421
133.002
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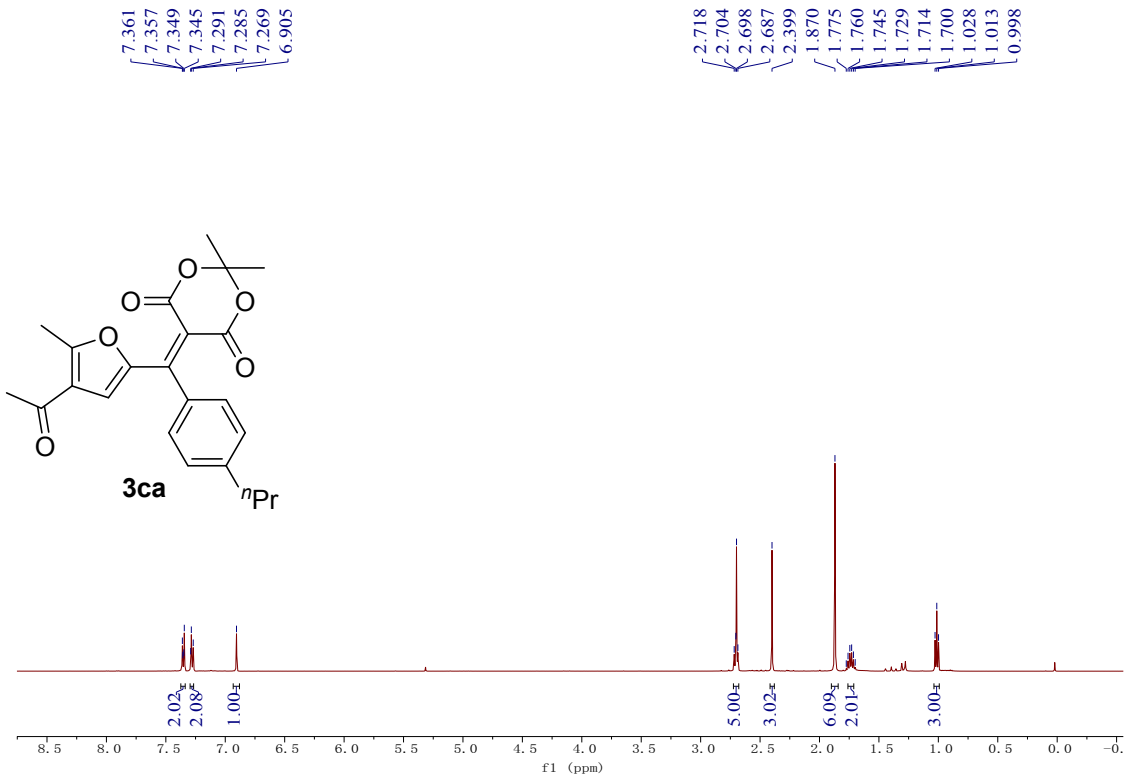
124.607
110.953
103.871
29.034
27.250
21.721
15.034
3ba
f1 (ppm)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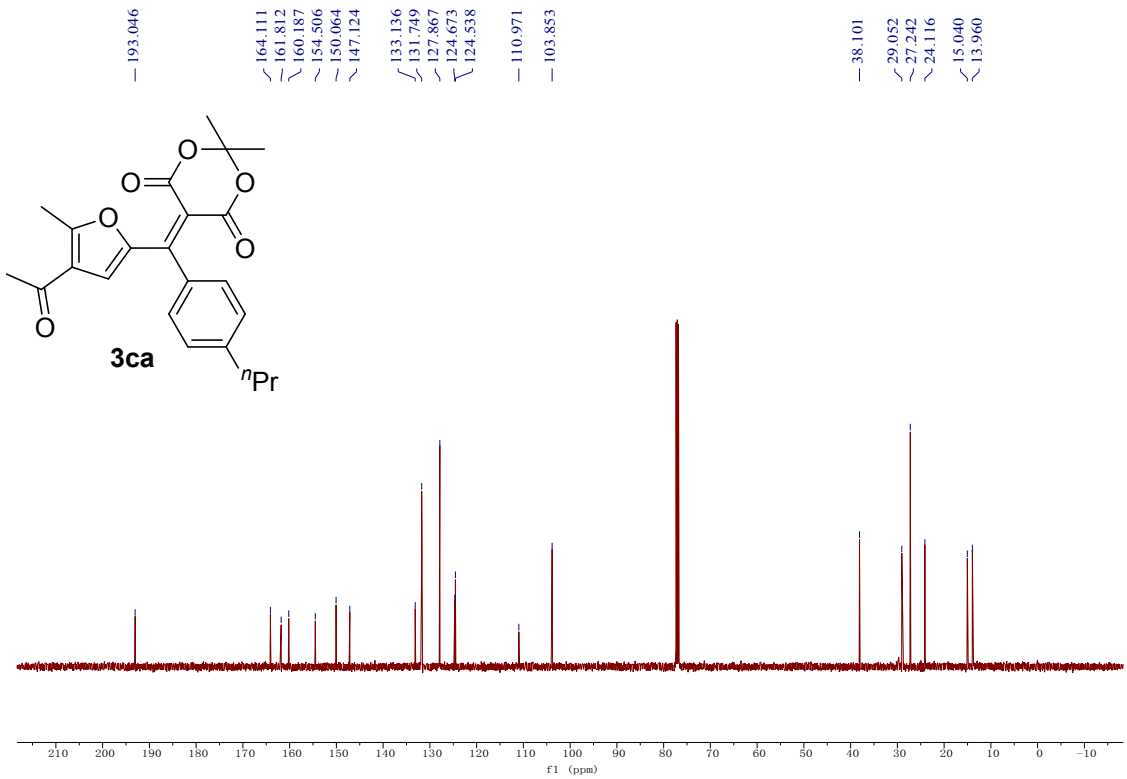
7.345
7.291
7.285
7.269
6.905
2.718
2.704
2.698
2.687
2.399
1.870
1.775
1.760
1.745
1.729
1.714
1.700
1.028
1.013
0.998
3ca
nPr
2.02
2.08
1.00
5.00
3.02
6.09
2.01
3.00
f1 (ppm)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161.812
160.187
154.506
150.064
147.124
133.136
131.749
127.867
124.673
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103.853
38.101
29.052
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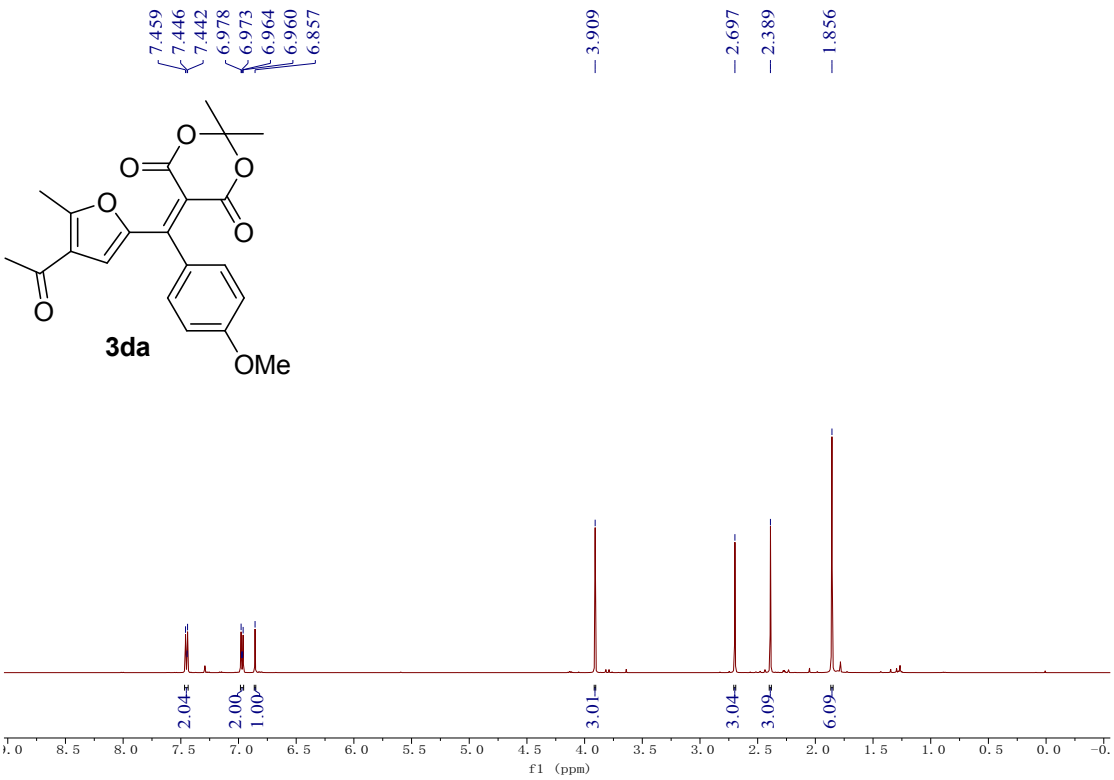
24.116
15.040
13.960
3ca
nPr
f1 (ppm)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7.442
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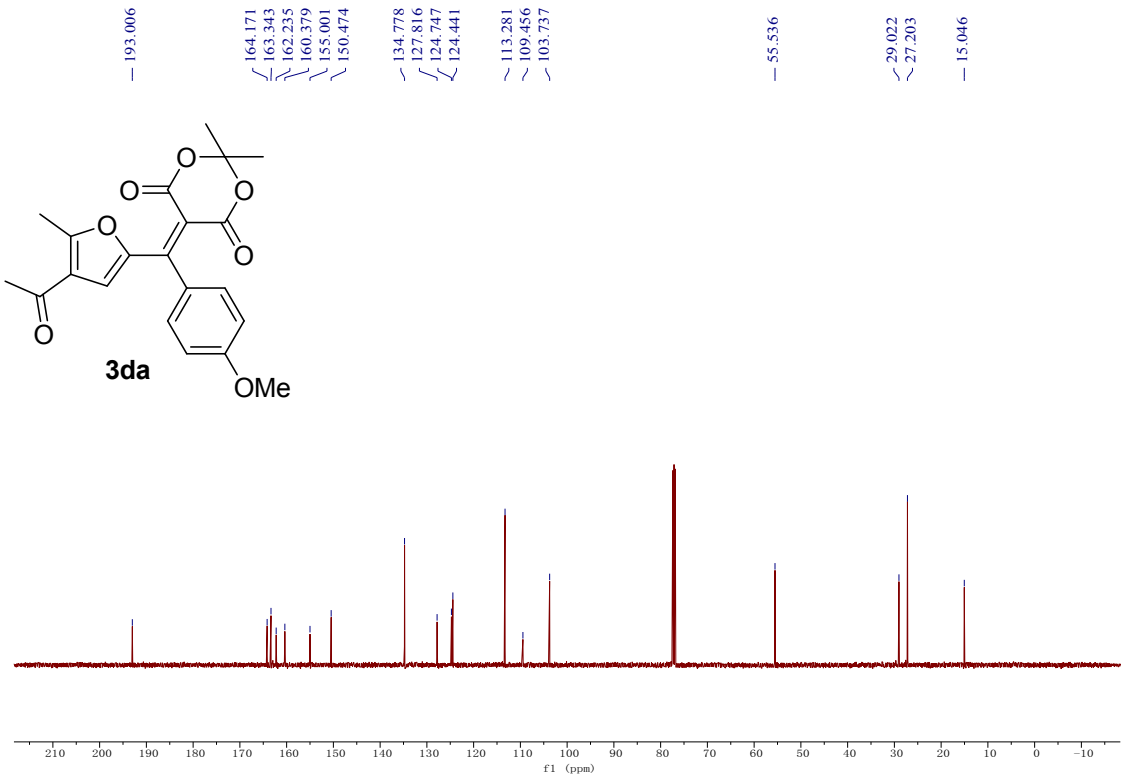
6.973
6.964
6.960
6.857
3.909
2.697
2.389
1.856
3da
OMe
2.04
2.00
1.00
3.01
3.04
3.09
6.09
f1 (ppm)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163.343
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155.001
150.474
134.778
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109.456
103.737
55.536
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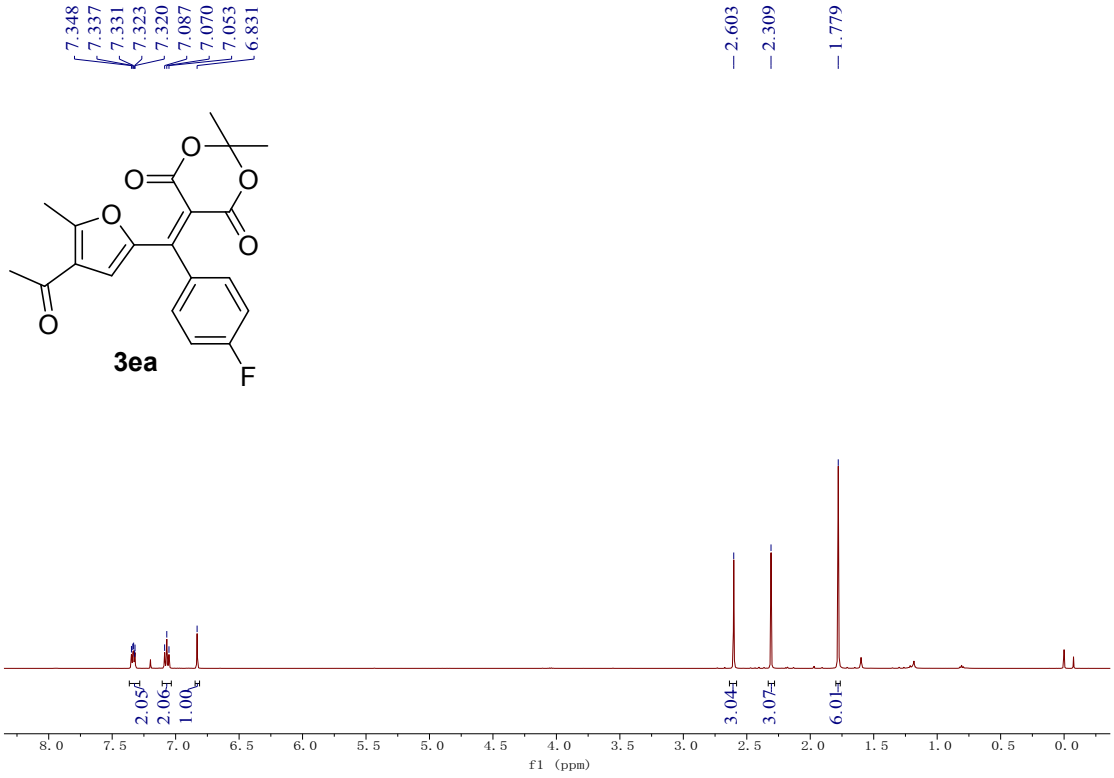
27.203
15.046
3da
OMe
f1 (ppm)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7.323
7.320
7.087
7.070
7.053
6.831
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2.309
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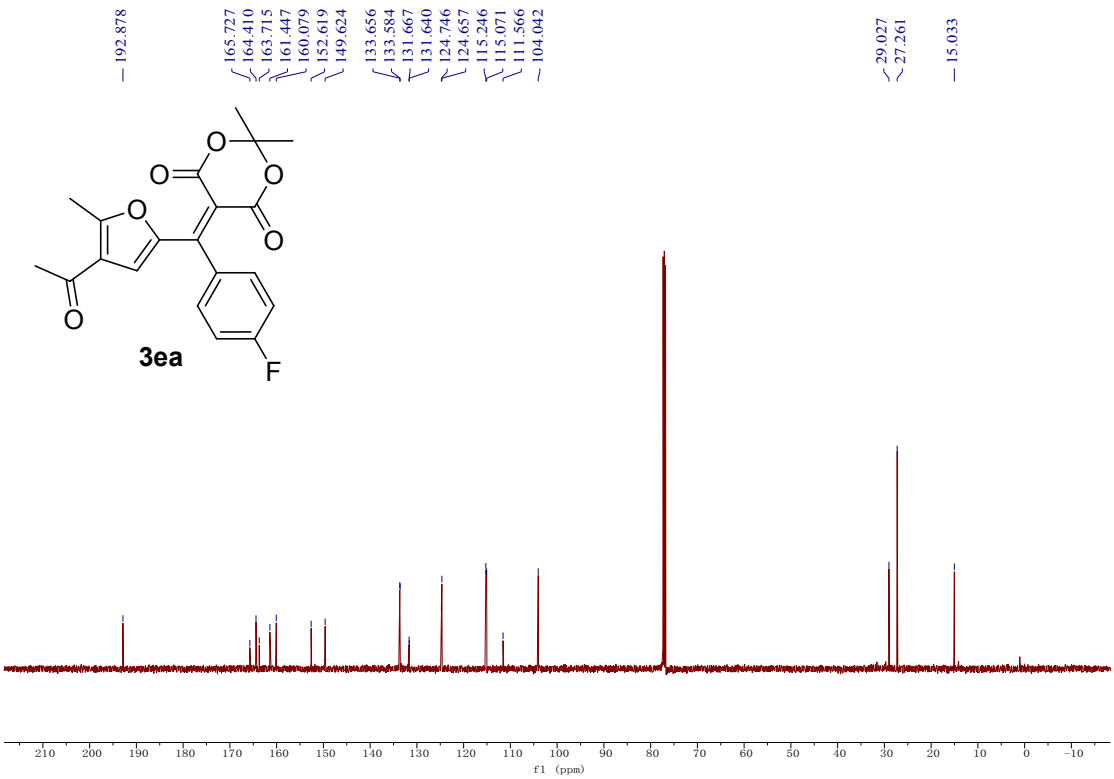
3ea
2.05
2.06
1.00
3.04
3.07
6.01
f1 (ppm)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149.624
133.656
133.584
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131.640
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124.657
115.246
115.071
111.566
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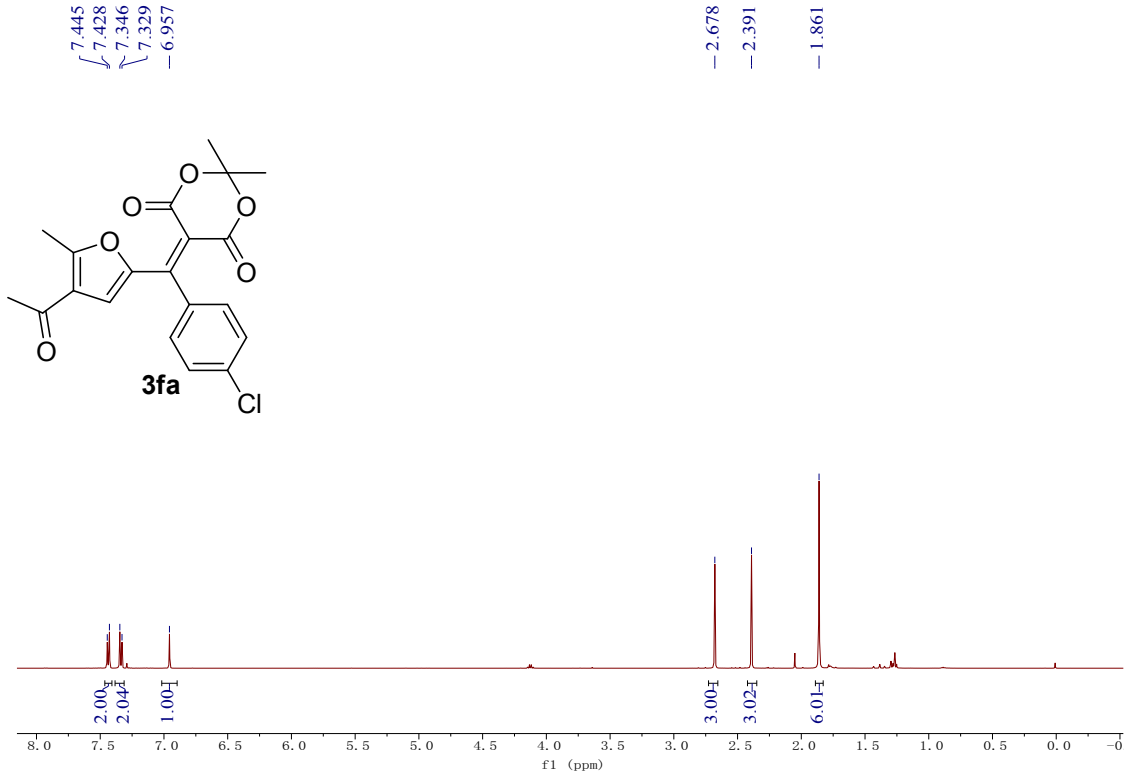
29.027
27.261
15.033
3ea
f1 (ppm)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Cl
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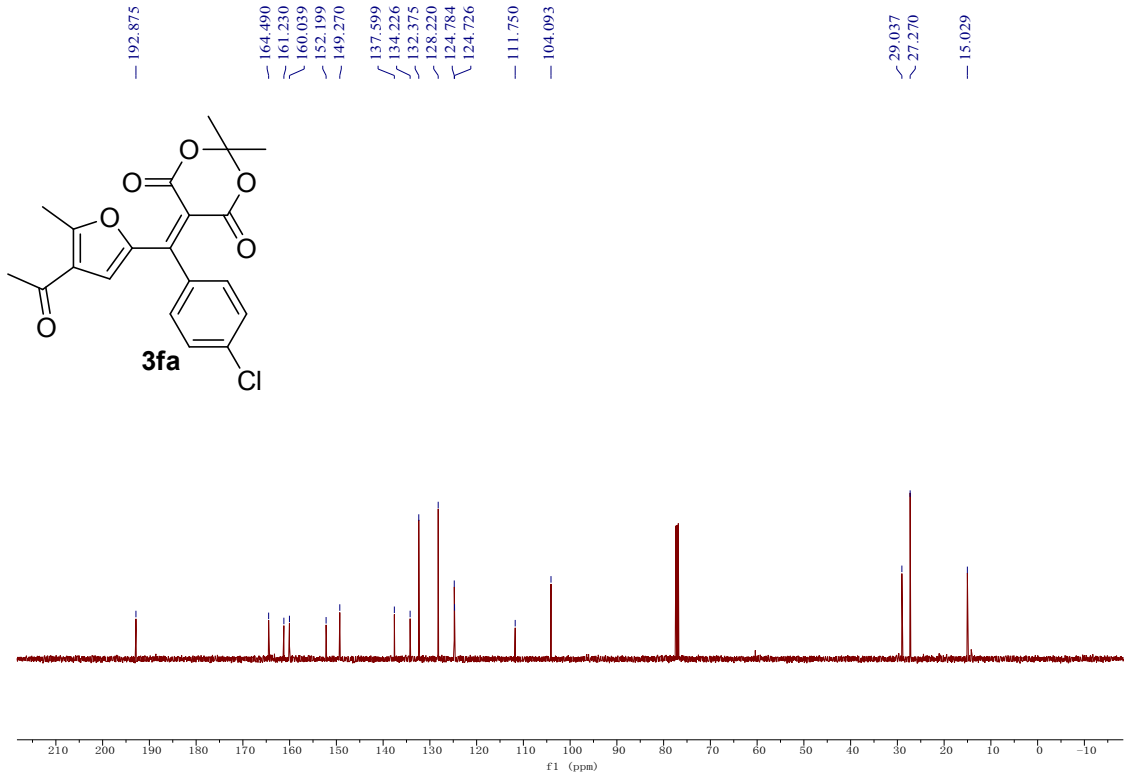
1.00
3.00
3.02
6.01
f1 (ppm)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149.270
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111.750
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27.270
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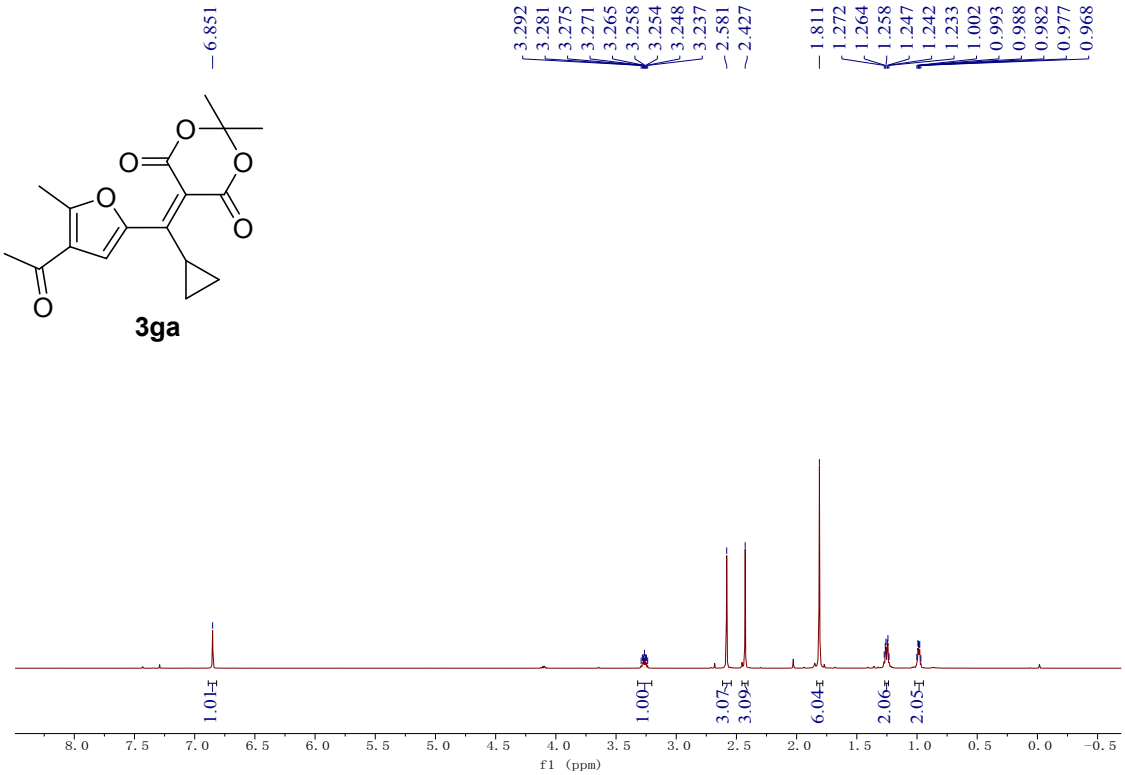
3fa
Cl
f1 (ppm)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3.265
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3.237
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1.811
1.272
1.264
1.258
1.247
1.242
1.233
1.002
0.993
0.988
0.982
0.977
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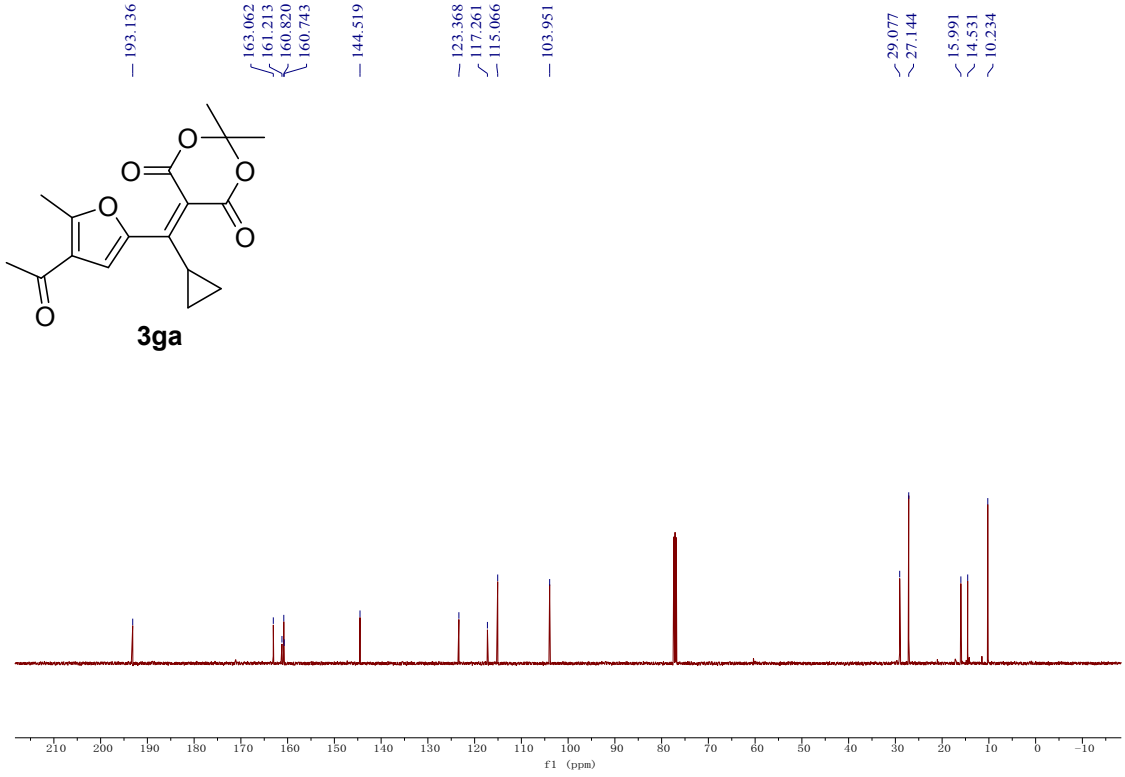
1.00
3.07
3.09
6.04
2.06
2.05
f1 (ppm)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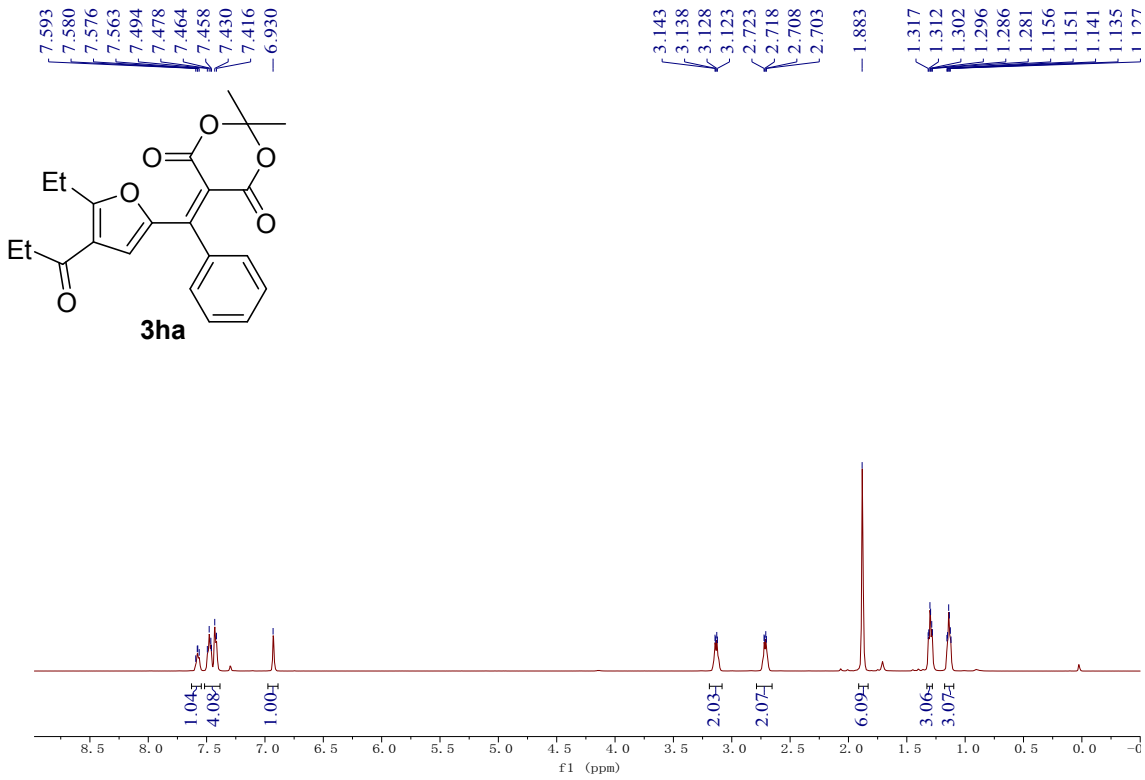
3ga
f1 (ppm)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3.128
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2.718
2.708
2.703
1.883
1.317
1.312
1.302
1.296
1.286
1.281
1.156
1.151
1.141
1.135
Et
Et
O
O
O
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4.08
1.00
2.03
2.07
6.09
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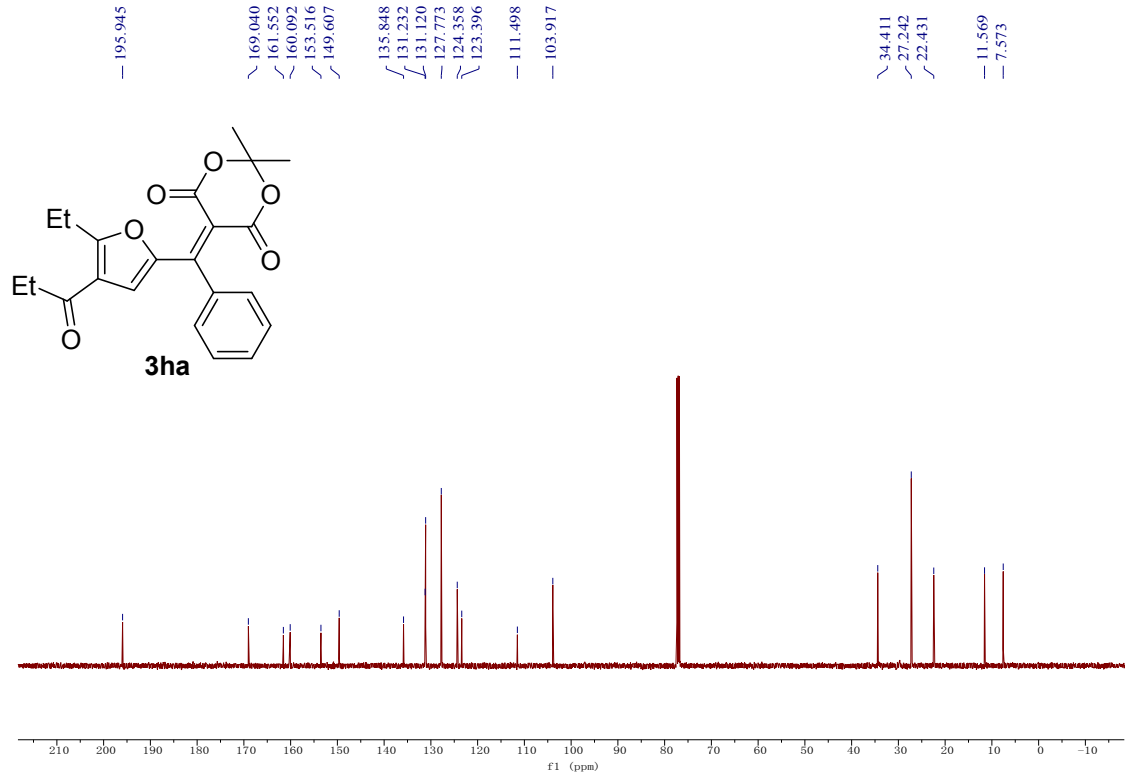
3.07
f1 (ppm)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O
O
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O
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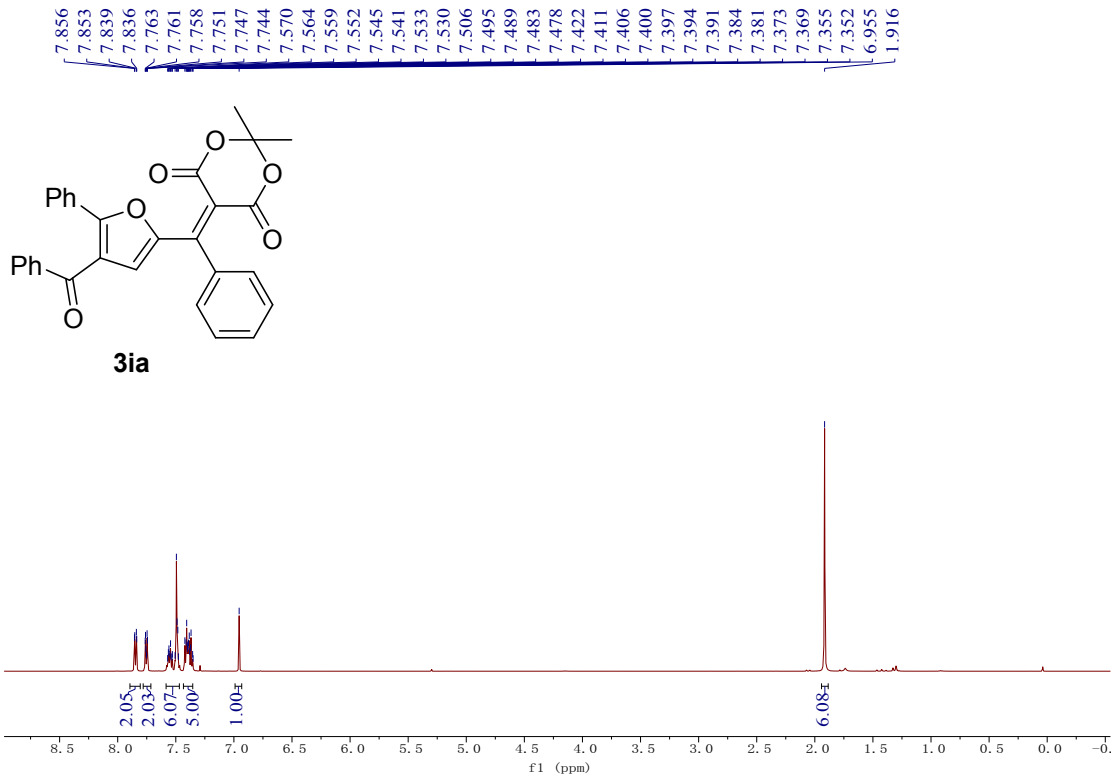
f1 (ppm)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7.394
7.391
7.384
7.381
7.373
7.369
7.355
7.352
6.955
1.916
Ph
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Ph
O
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2.03
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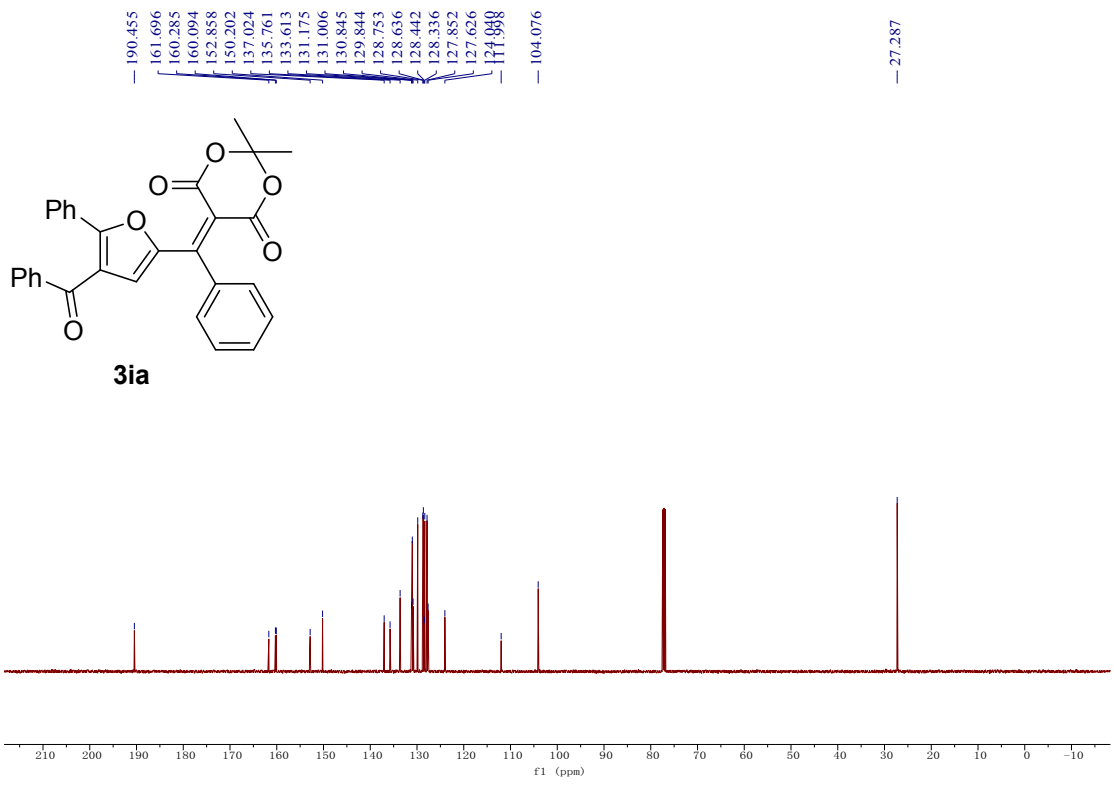
5.00
1.00
6.08
f1 (ppm)

3ia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128.753
128.636
128.442
128.336
127.852
127.626
124.040
111.998
104.076
27.287
Ph
O
O
O
O
Ph
O
O
f1 (ppm)

3ia


7.964
7.961
7.957
7.952
7.948
7.944
7.614
7.604
7.600
7.596
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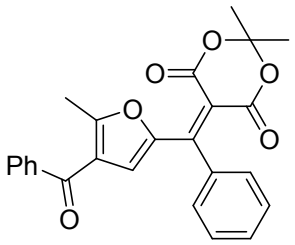
1.875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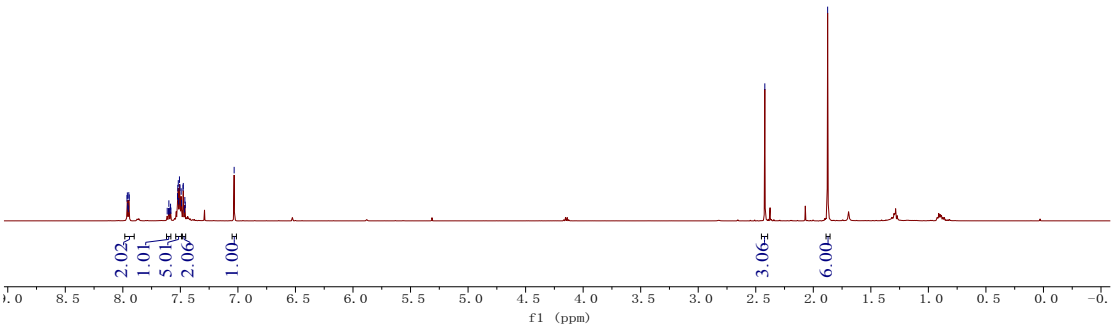
Ph
O

3ja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f1 (ppm)


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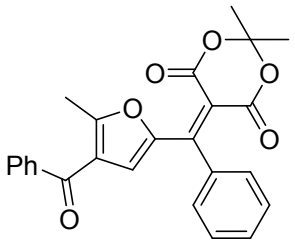
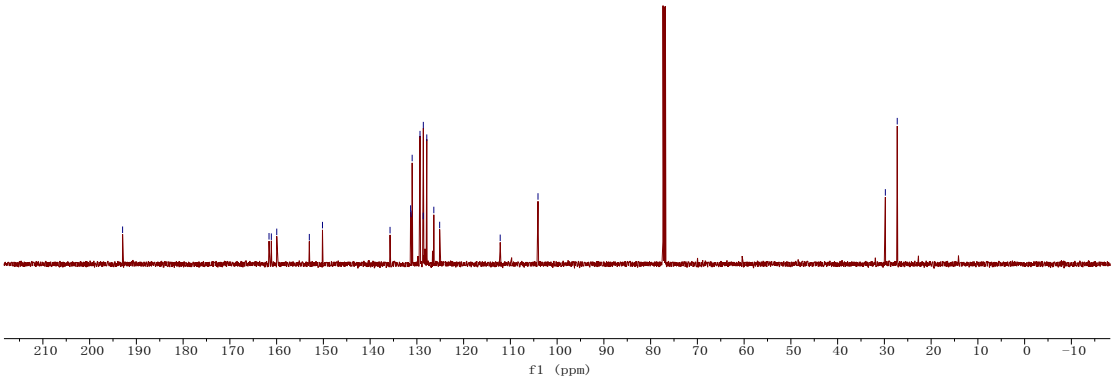
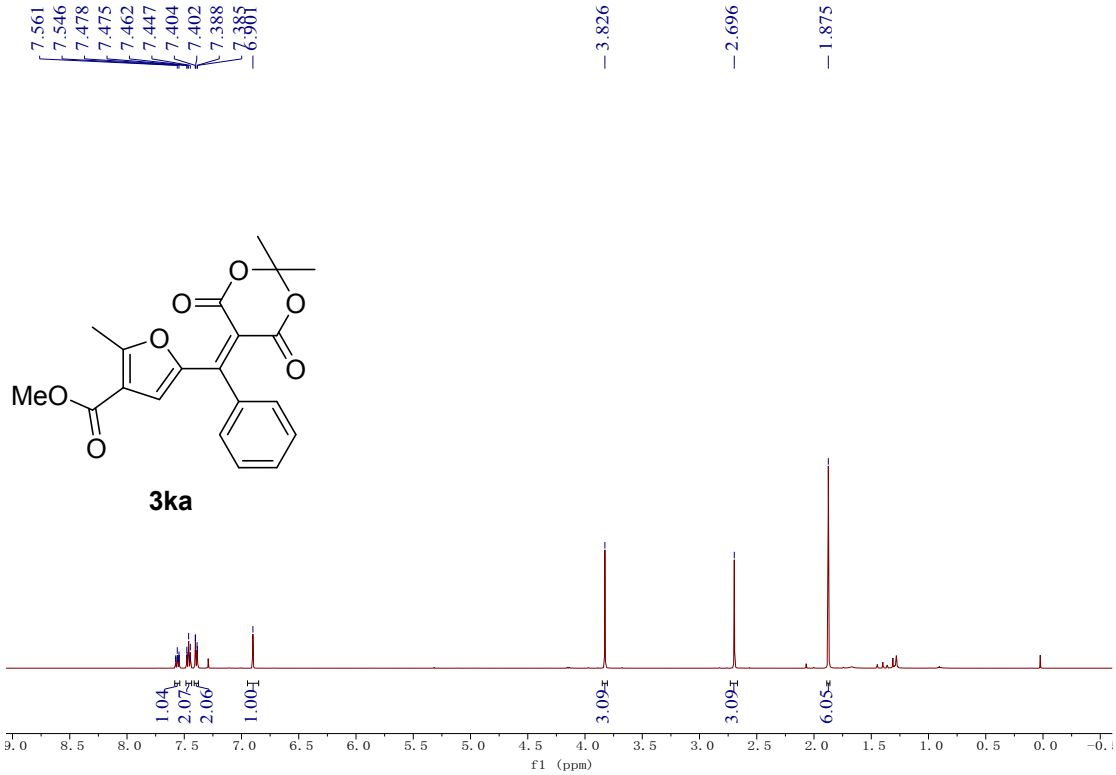
128.640
128.621
127.881
126.376
125.125
112.176
104.091
29.792
27.250


O
O
O
O
O
Ph
O

3ja


f1 (ppm)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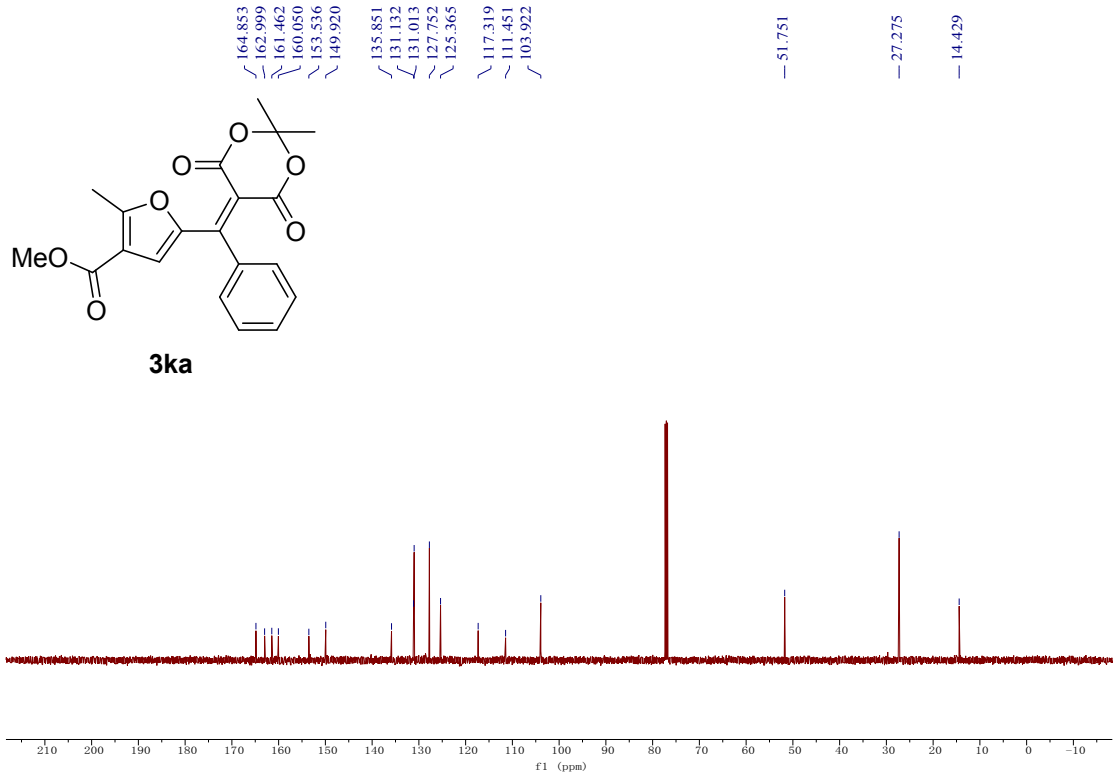
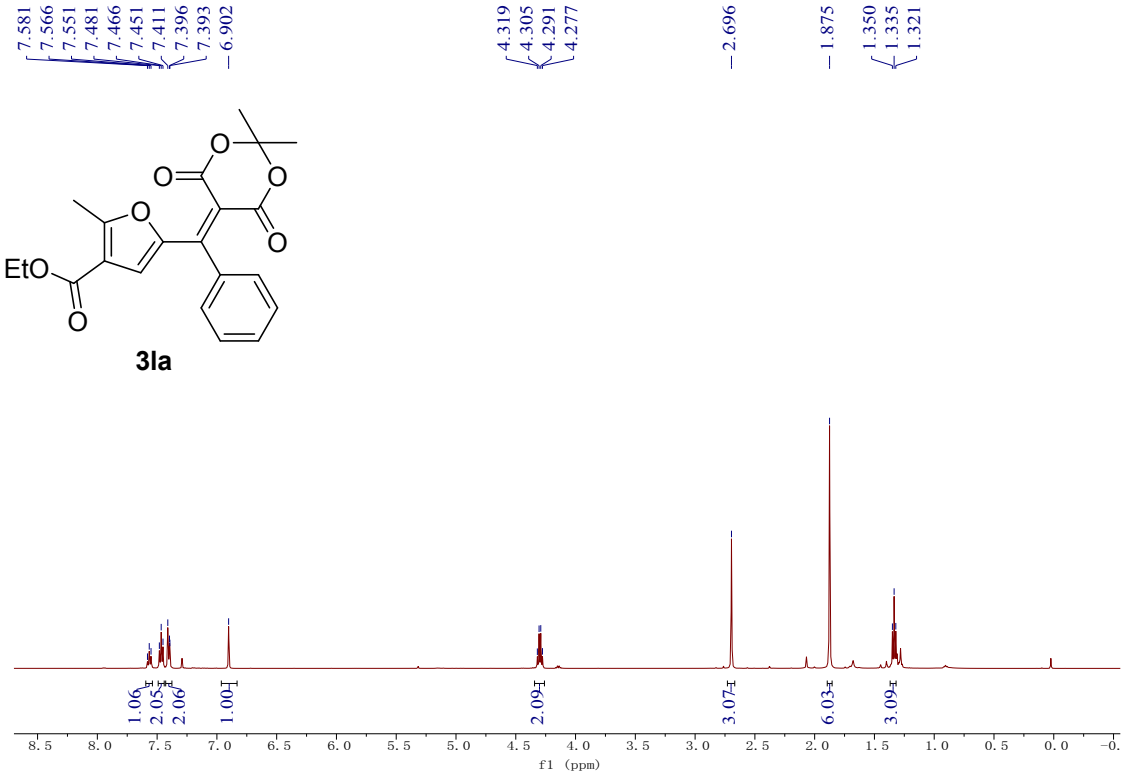
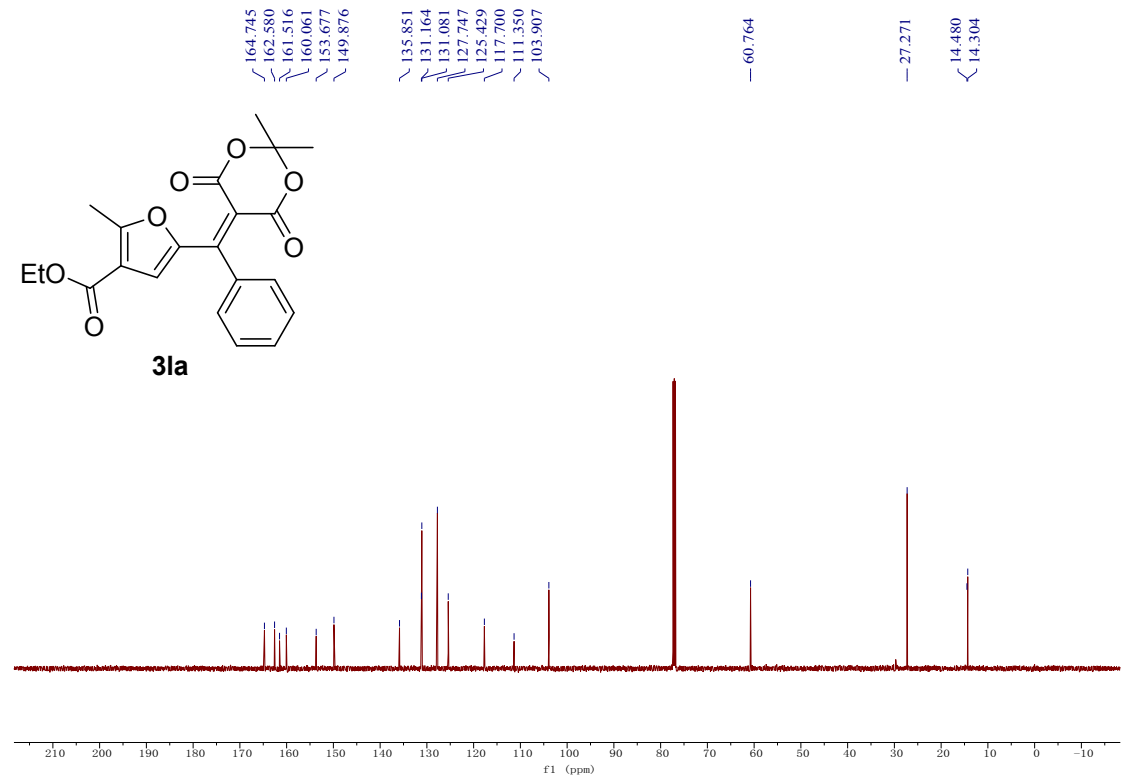
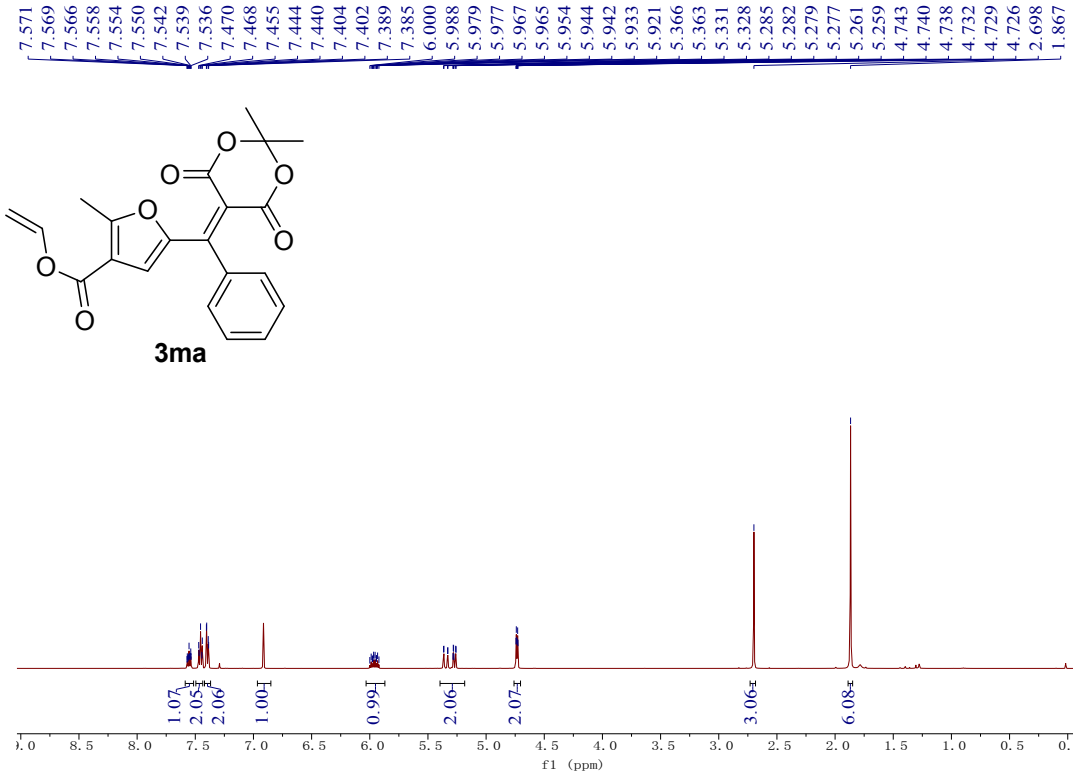
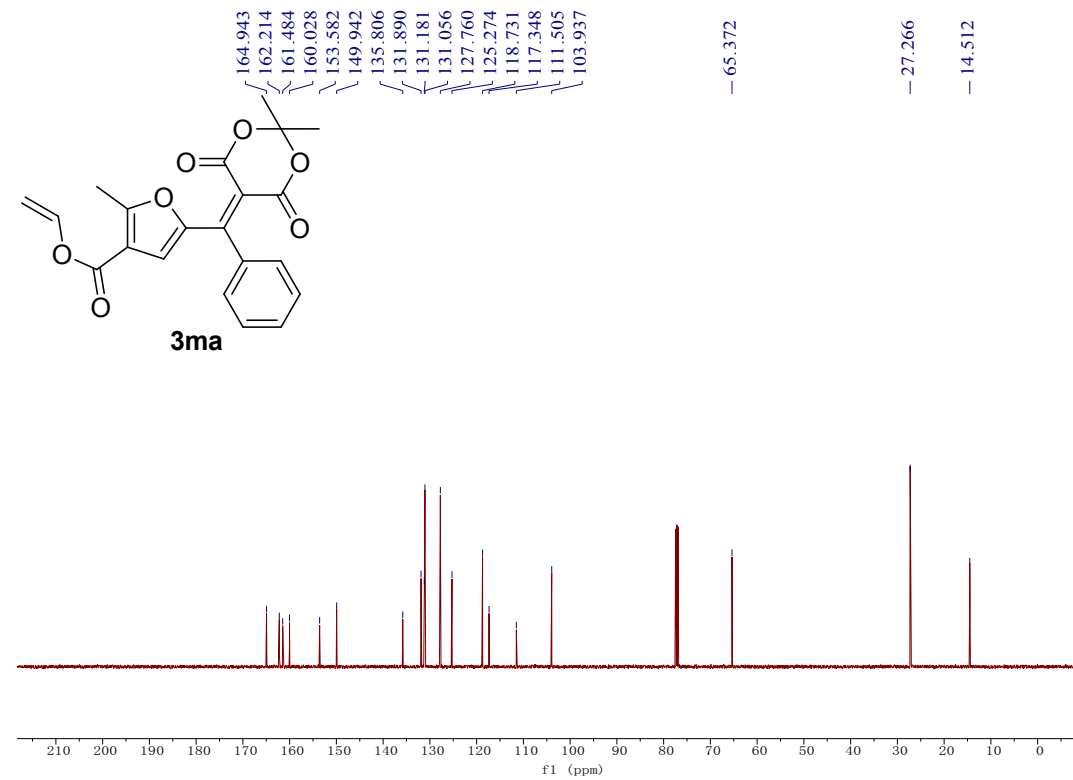
7.546
7.478
7.475
7.462
7.447
7.404
7.402
7.388
7.385
6.901
3.826
2.696
1.875
MeO
O
O
O
O
O
O
3ka
1.04
2.07
2.06
1.00
3.09
3.09
6.05
f1 (ppm)

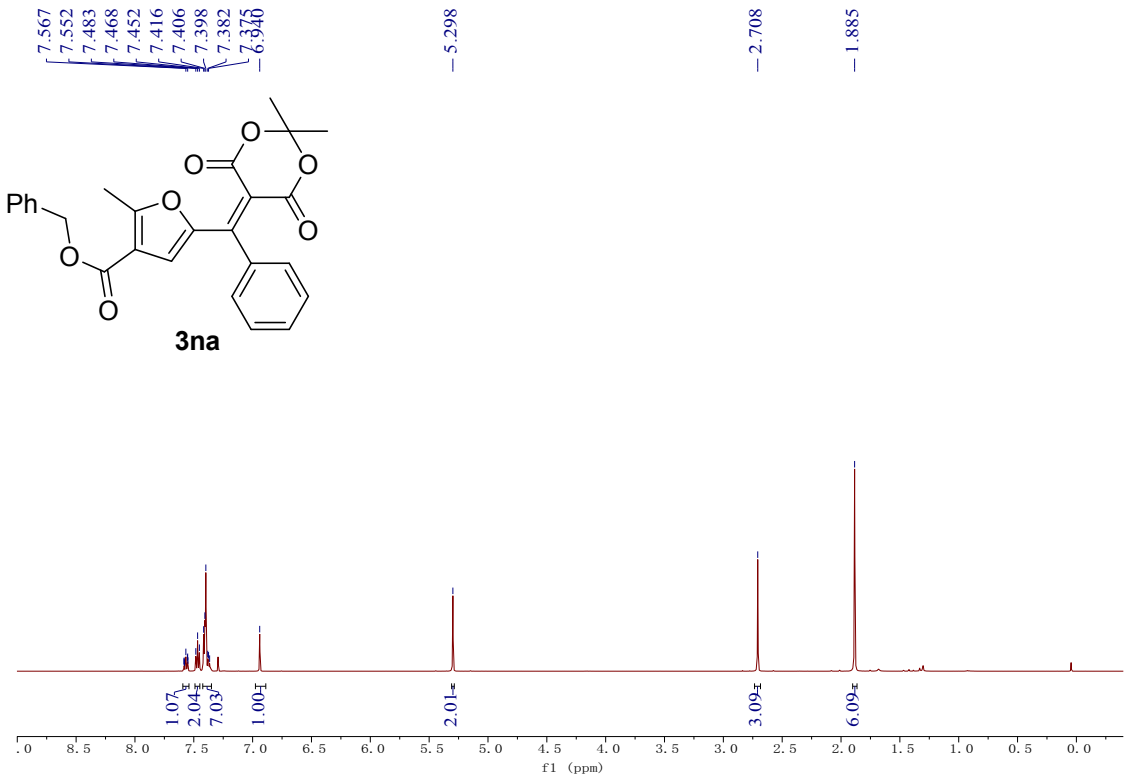

164.853
162.999
161.462
160.050
153.536
149.920
135.851
131.132
131.013
127.752
125.365
117.319
111.451
103.922
51.751
27.275
14.429
MeO
O
O
O
O
O
O
3ka
f1 (ppm)

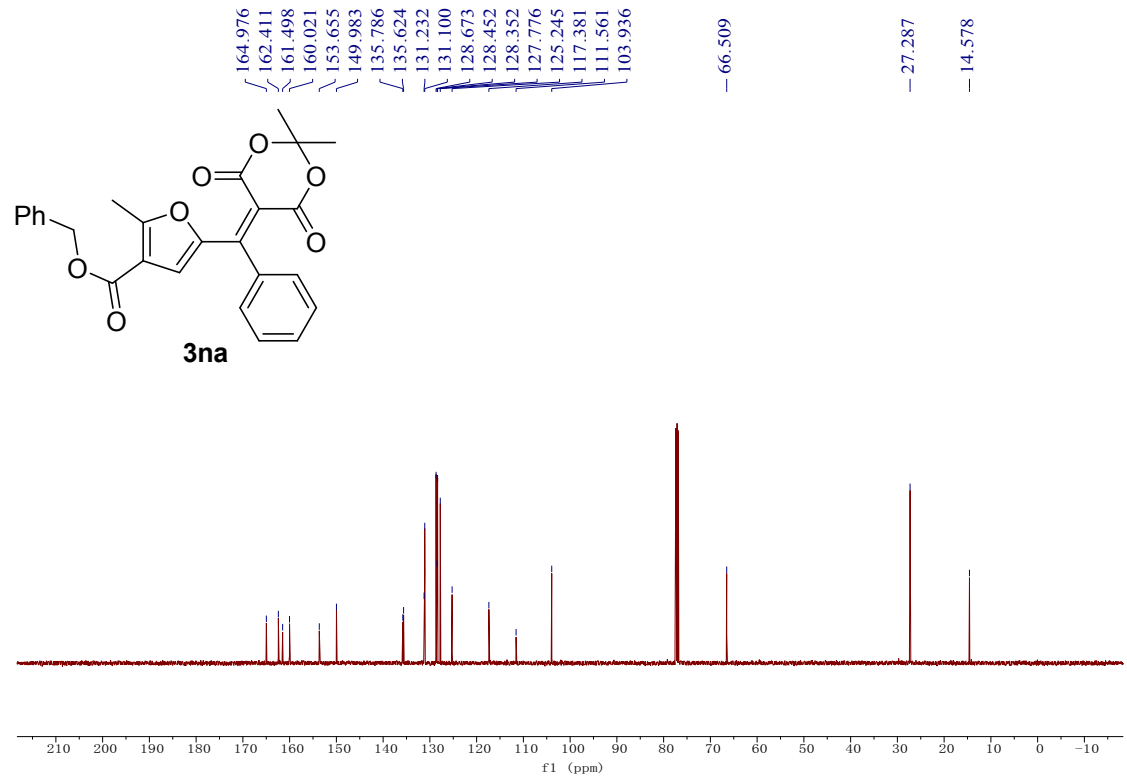

7.581
7.566
7.551
7.481
7.466
7.451
7.411
7.396
7.393
6.902
4.319
4.305
4.291
4.277
2.696
1.875
1.350
1.335
1.321
EtO
O
3la
1.06
2.05
2.06
1.00
2.09
3.07
6.03
3.09
f1 (ppm)

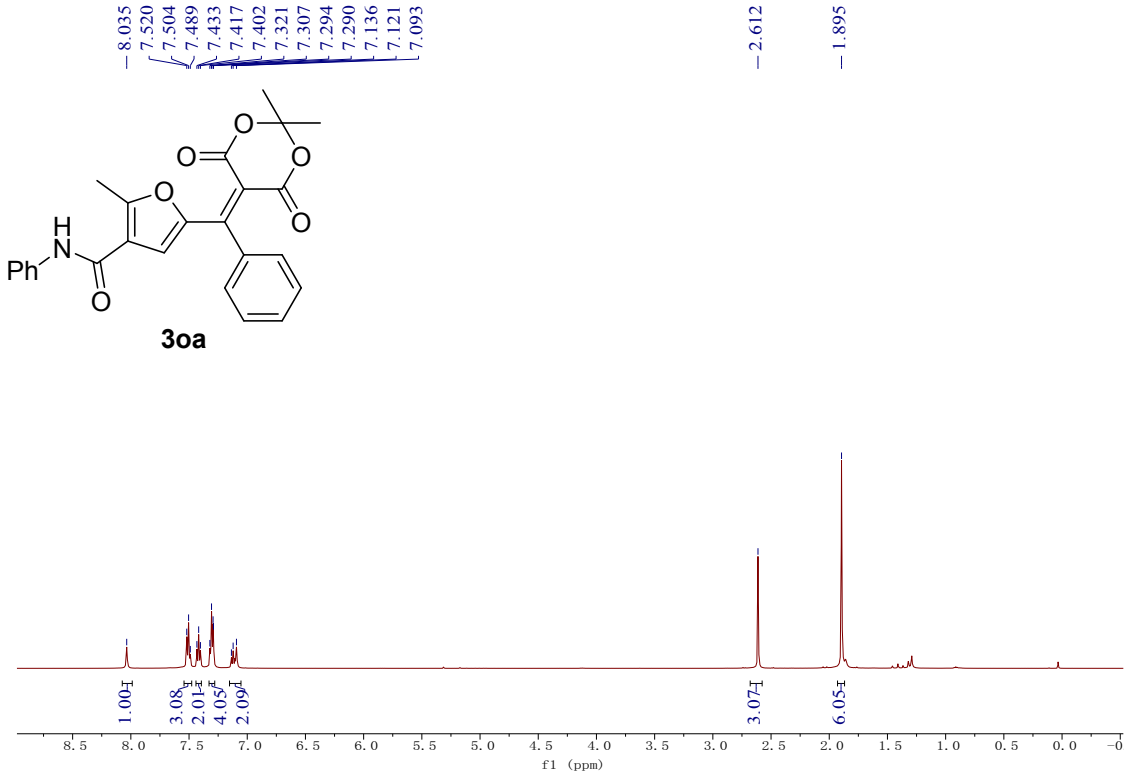

164.745
162.580
161.516
160.061
153.677
149.876
135.851
131.164
131.081
127.747
125.429
117.700
111.350
103.907
60.764
27.271
14.480
14.304
EtO
O
3la
f1 (ppm)


7.571
7.569
7.566
7.558
7.554
7.550
7.542
7.539
7.536
7.470
7.468
7.455
7.444
7.440
7.404
7.402
7.389
7.385
6.000
5.988
5.979
5.977
5.967
5.965
5.954
5.944
5.942
5.933
5.921
5.366
5.363
5.331
5.328
5.285
5.282
5.279
5.277
5.261
5.259
4.743
4.740
4.738
4.732
4.729
4.726
2.698
1.867
3ma
1.07
2.05
2.06
1.00
0.99
2.06
2.07
3.06
6.08
f1 (ppm)

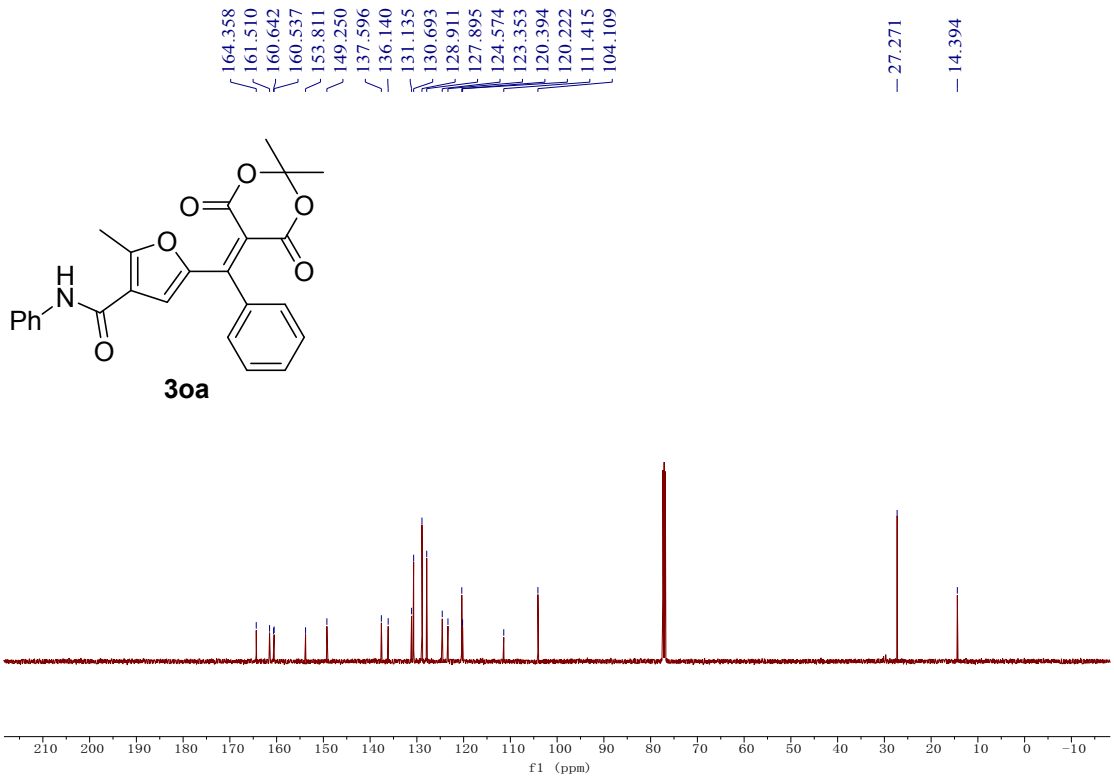

164.943
162.214
161.484
160.028
153.582
149.942
135.806
131.890
131.181
131.056
127.760
125.274
118.731
117.348
111.505
103.937
65.372
27.266
14.512
3ma
f1 (ppm)


7.567
7.552
7.483
7.468
7.452
7.416
7.406
7.398
7.382
7.375
6.940
5.298
2.708
1.885
Ph
O
O
O
O
O
O
O
O
3na
1.07
2.04
7.03
1.00
2.01
3.09
6.09
f1 (ppm)

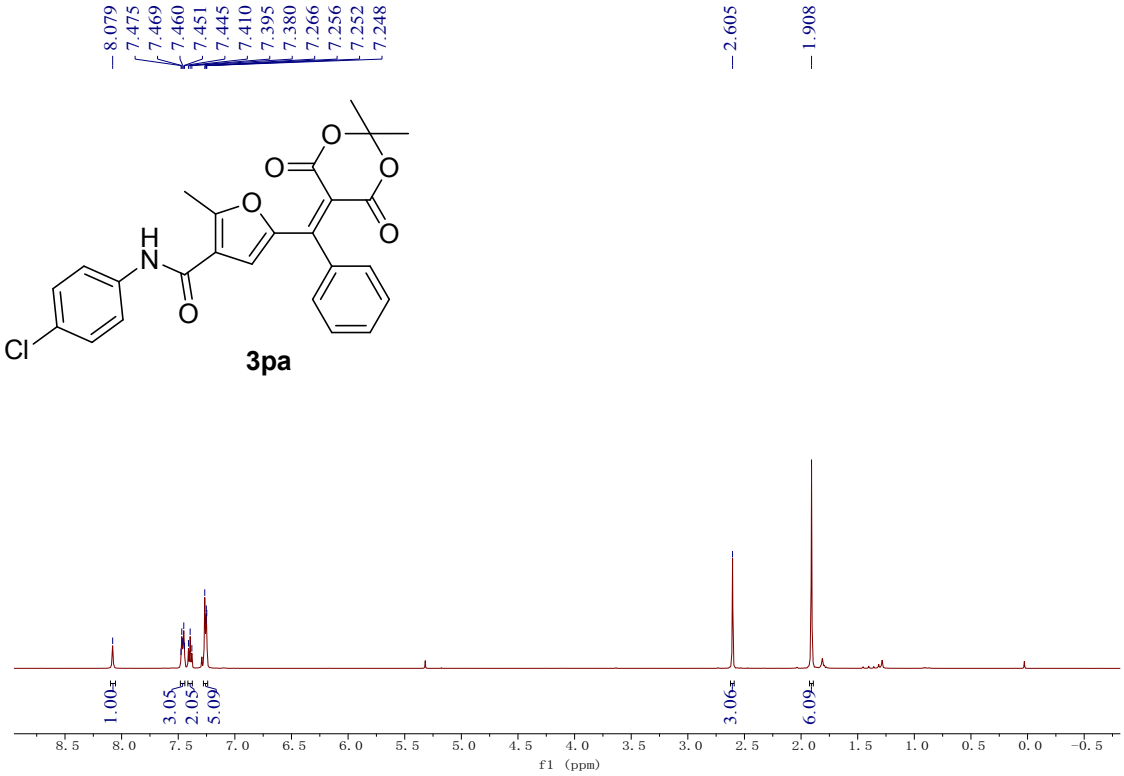

164.976
162.411
161.498
160.021
153.655
149.983
135.786
135.624
131.232
131.100
128.673
128.452
128.352
127.776
125.245
117.381
111.561
103.936
66.509
27.287
14.578
Ph
O
O
O
O
O
O
O
O
3na
f1 (ppm)

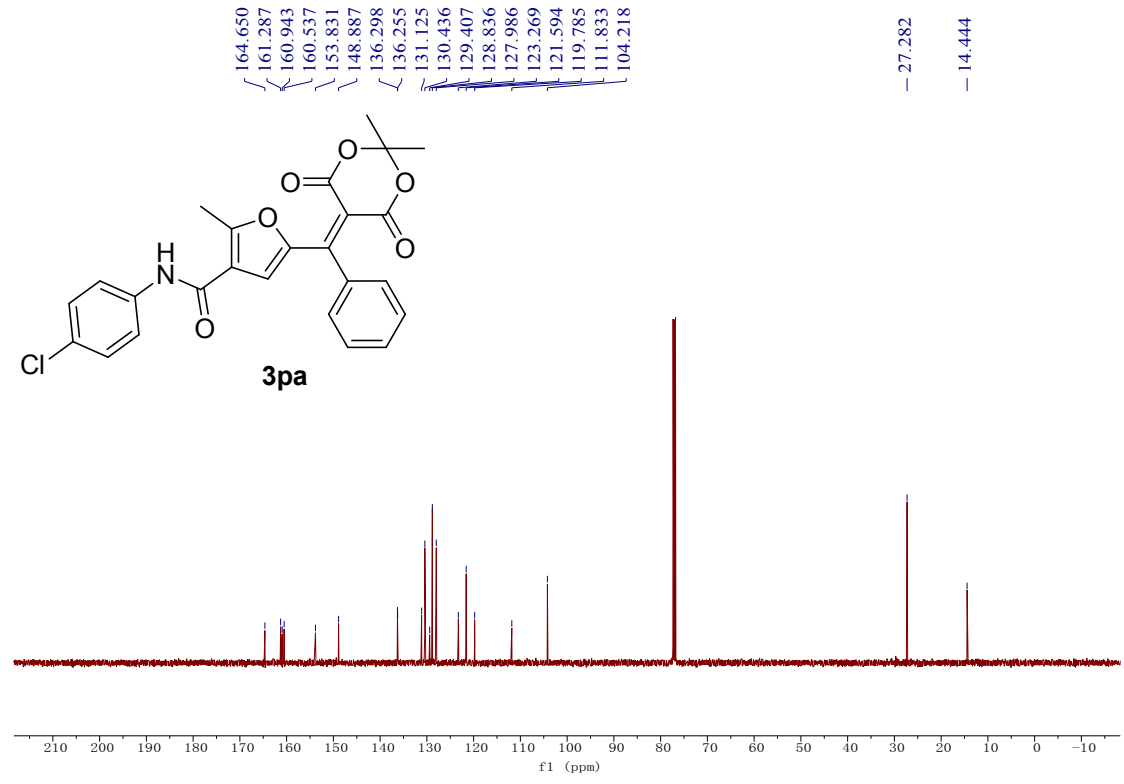

8.035
7.520
7.504
7.489
7.433
7.417
7.402
7.321
7.307
7.294
7.290
7.136
7.121
7.093
2.612
1.895
O
O
O
O
O
H
N
Ph
O
1.00
3.08
2.01
4.05
2.09
3.07
6.05
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

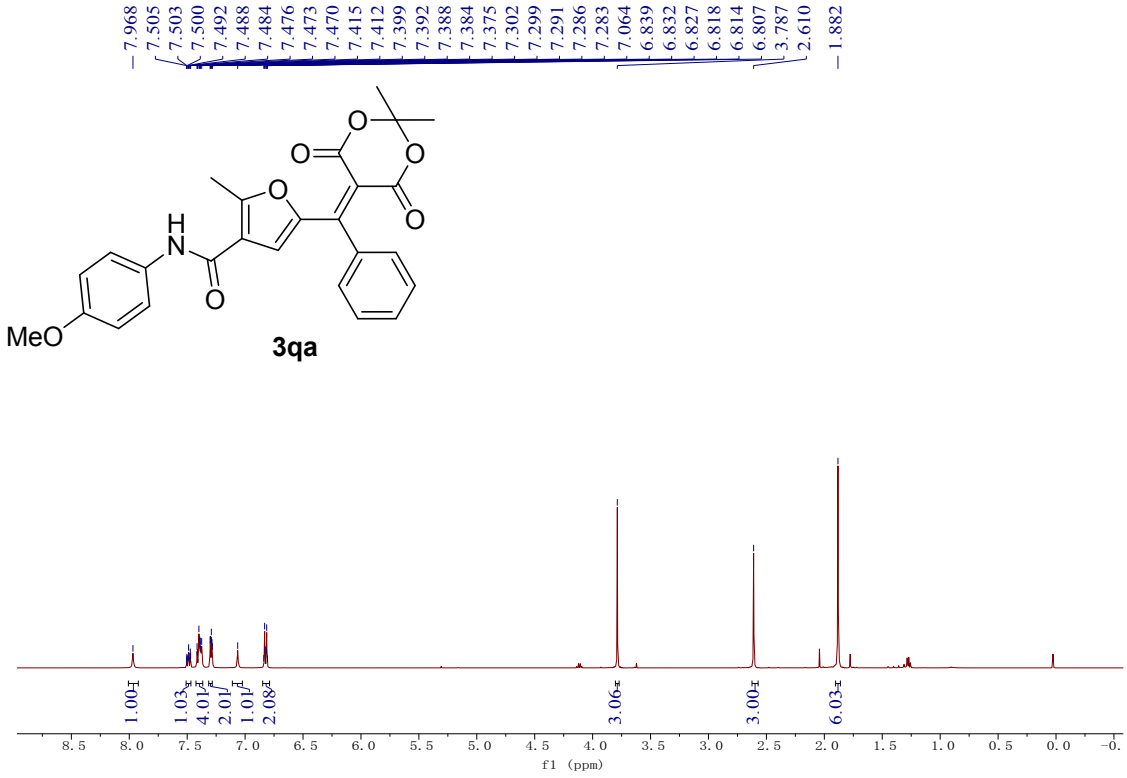
3oa


164.358
161.510
160.642
160.537
153.811
149.250
137.596
136.140
131.135
130.693
128.911
127.895
124.574
123.353
120.394
120.222
111.415
104.109
27.271
14.394
O
O
O
O
O
H
N
Ph
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

3oa


8.079
7.475
7.469
7.460
7.451
7.445
7.410
7.395
7.380
7.266
7.256
7.252
7.248
2.605
1.908
O
O
O
O
O
O
H
N
Cl
3pa
1.00
3.05
2.05
5.09
3.06
6.09
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)


164.650
161.287
160.943
160.537
153.831
148.887
136.298
136.255
131.125
130.436
129.407
128.836
127.986
123.269
121.594
119.785
111.833
104.218
27.282
14.444
O
O
O
O
O
O
H
N
Cl
3pa
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)


7.968
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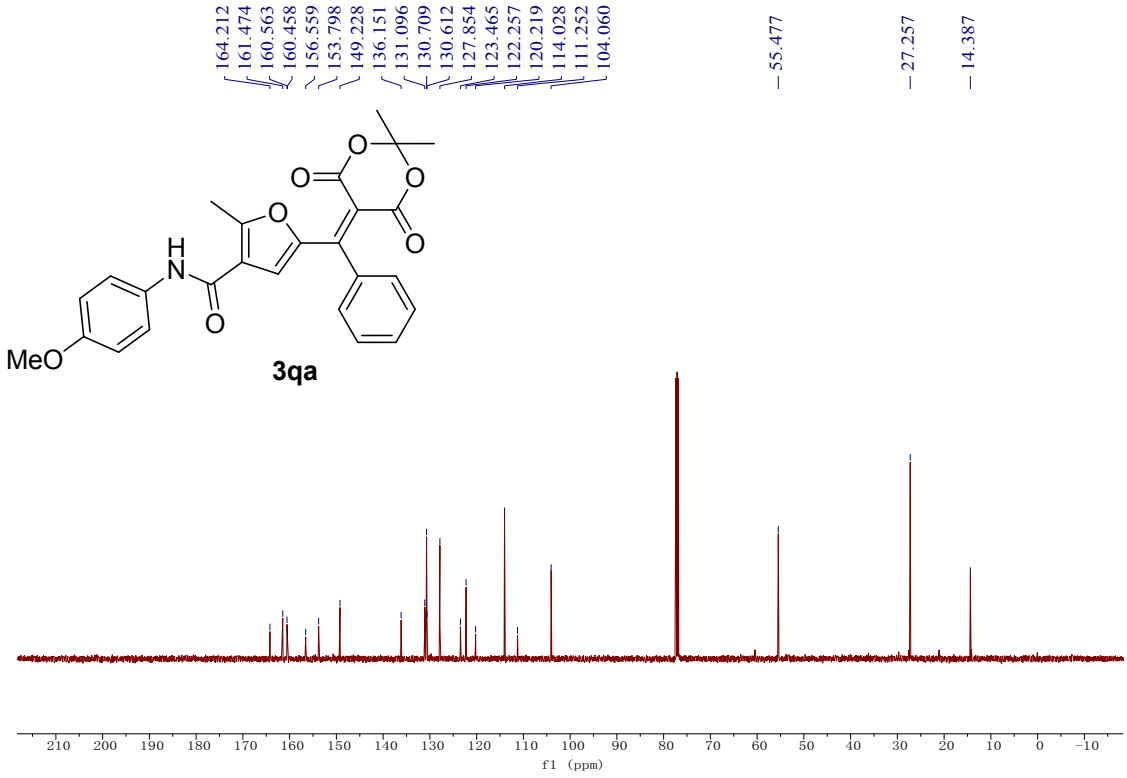
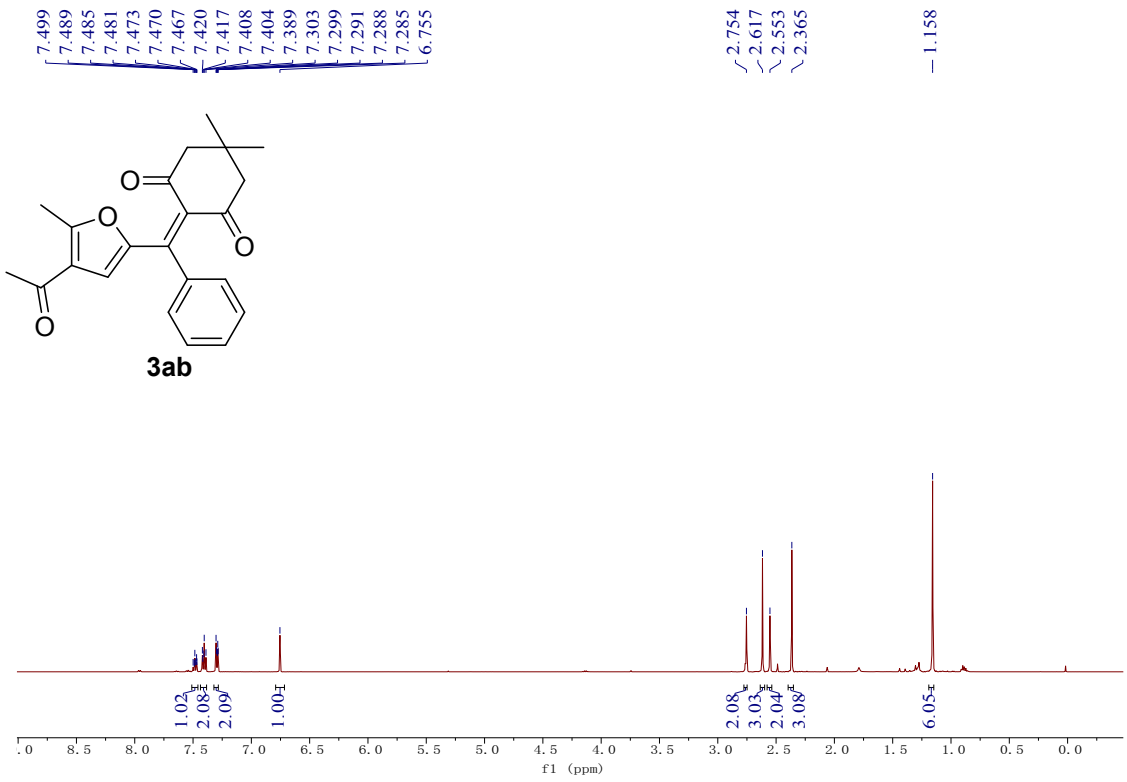
7.503
7.500
7.492
7.488
7.484
7.476
7.473
7.470
7.415
7.412
7.399
7.392
7.388
7.384
7.375
7.302
7.299
7.291
7.286
7.283
7.064
6.839
6.832
6.827
6.818
6.814
6.807
3.787
2.610
1.882
MeO
3qa
1.00
1.03
4.01
2.01
1.01
2.08
3.06
3.00
6.03
f1 (ppm)


164.212
161.474
160.563
160.458
156.559
153.798
149.228
136.151
131.096
130.709
130.612
127.854
123.465
122.257
120.219
114.028
111.252
104.060
55.477
27.257
14.387
MeO
3qa
f1 (ppm)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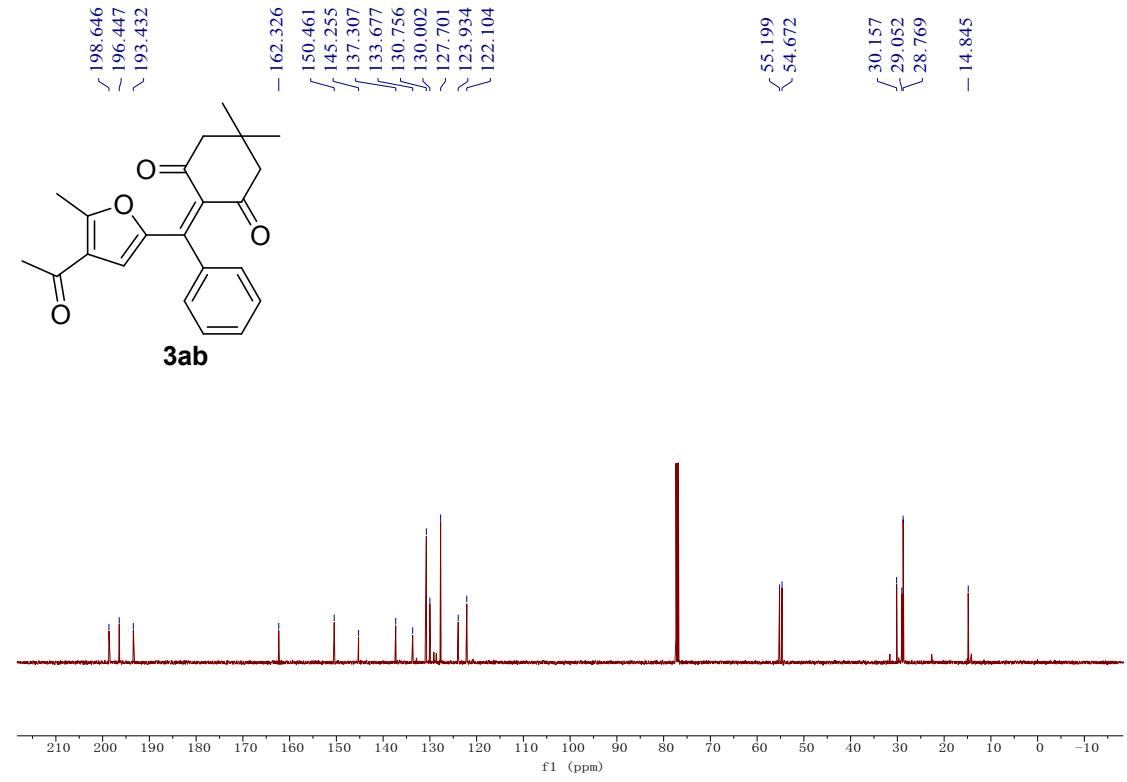
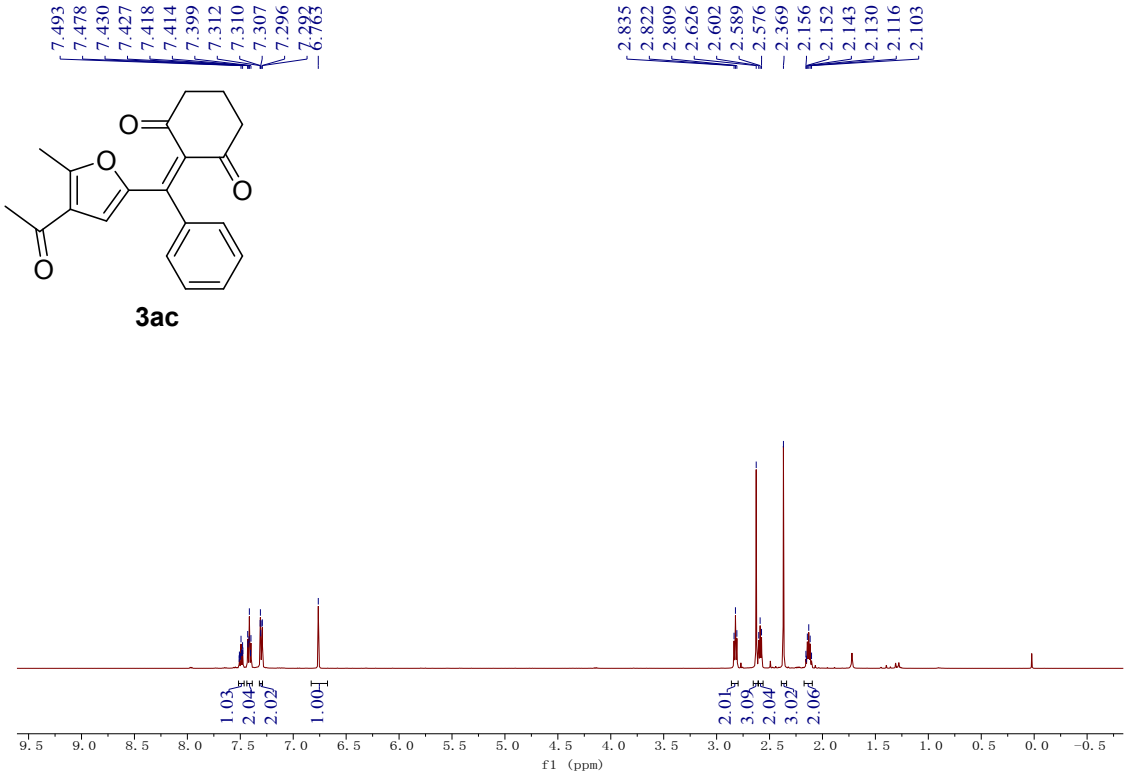
7.489
7.485
7.481
7.473
7.470
7.467
7.420
7.417
7.408
7.404
7.389
7.303
7.299
7.291
7.288
7.285
6.755
2.754
2.617
2.553
2.365
1.158
O
O
O
O
3ab
1.02
2.08
2.09
1.00
2.08
3.03
2.04
3.08
6.05
f1 (ppm)


198.646
196.447
193.432
162.326
150.461
145.255
137.307
133.677
130.756
130.002
127.701
123.934
122.104
55.199
54.672
30.157
29.052
28.769
14.845
O
O
O
O
3ab
f1 (ppm)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7.478
7.430
7.427
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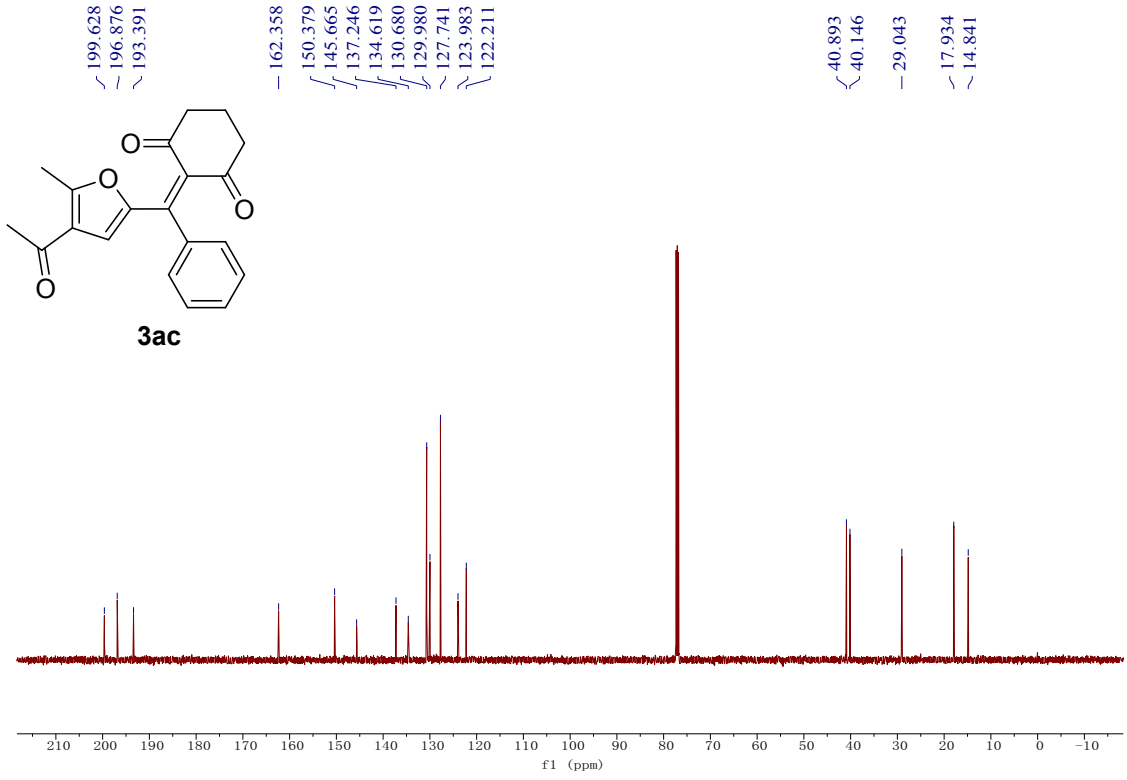
7.414
7.399
7.312
7.310
7.307
7.296
7.292
6.763
2.835
2.822
2.809
2.626
2.602
2.589
2.576
2.369
2.156
2.152
2.143
2.130
2.116
2.103
O
O
O
O
3ac
1.03
2.04
2.02
1.00
2.01
3.09
2.04
3.02
2.06
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5
f1 (ppm)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162.358
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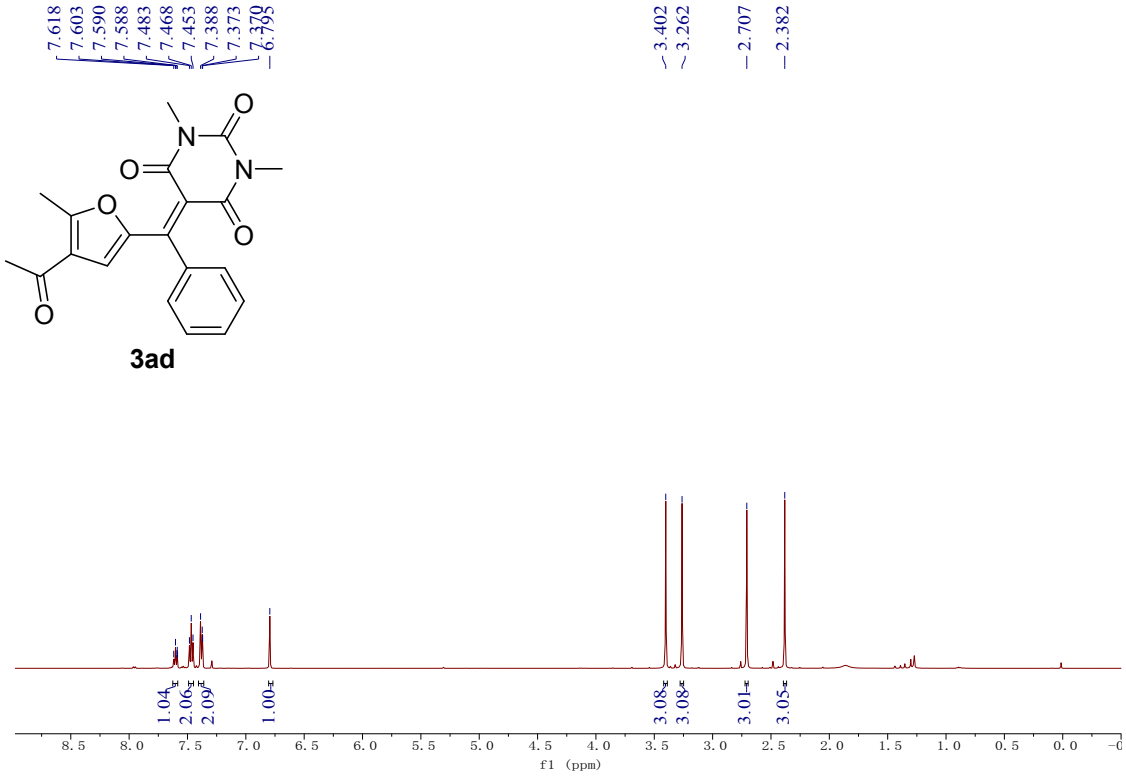
145.665
137.246
134.619
130.680
129.980
127.741
123.983
122.211
40.893
40.146
29.043
17.934
14.841
O
O
O
O
3ac
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


7.618
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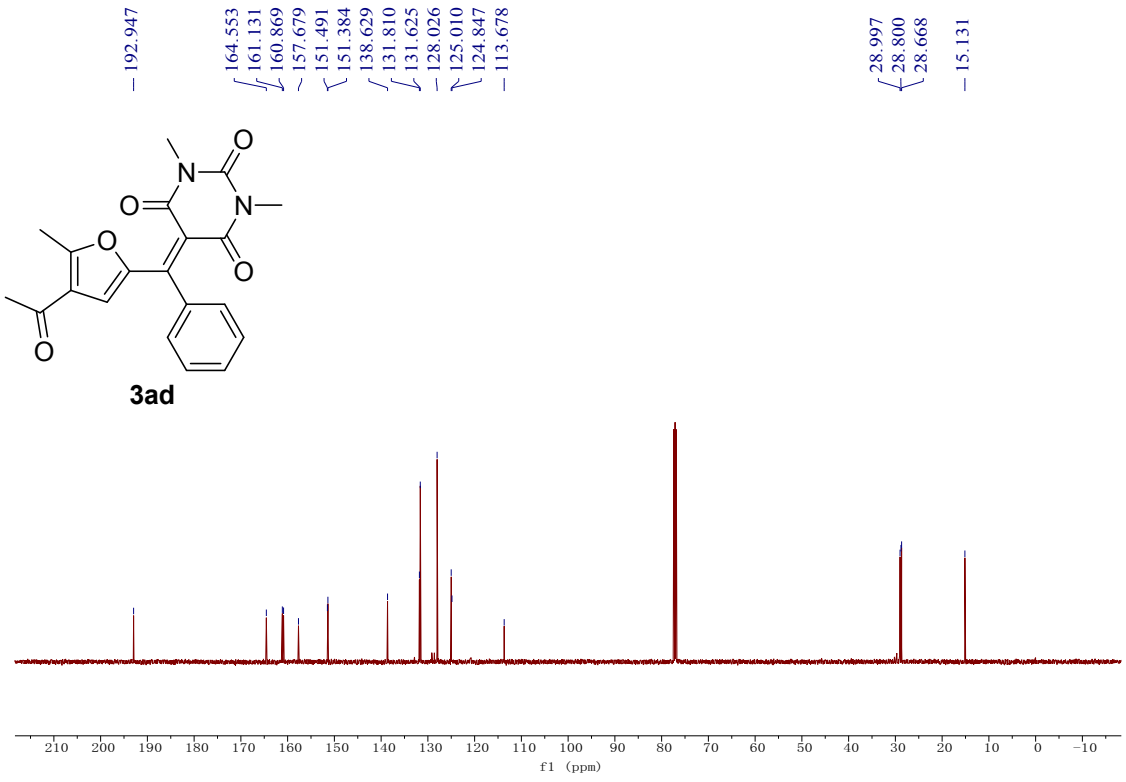
7.590
7.588
7.483
7.468
7.453
7.388
7.373
7.370
6.795
3.402
3.262
2.707
2.382
1.04
2.06
2.09
1.00
3.08
3.08
3.01
3.05
f1 (ppm)

3ad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160.869
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151.491
151.384
138.629
131.810
131.625
128.026
125.010
124.847
113.678
28.997
28.800
28.668
15.131
f1 (ppm)

3ad