Supporting information

Highly Enantioselective Synthesis of Chiral Succinic anhydrides *via* Rh/Bisphosphine-Thiourea-Catalyzed Asymmetric Hydrogenation

Zhengyu Han,[a] Rui Wang,[a] Guoxian Gu,[b] Xiu-Qin Dong,*[a] Xumu Zhang* [b,a]

[a] *College of Chemistry and Molecular Sciences, Wuhan University, Wuhan, Hubei 430072, P. R. China.*

[b] *Department of Chemistry, South University of Science and Technology of China, Shenzhen, 518000, P.R. China.*

E-mail address: xumu@whu.edu.cn, xiuqindong@ whu.edu.cn.

Content

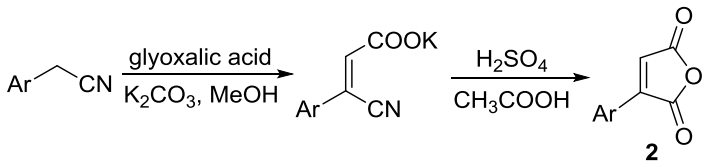
I. General Remarks

All the reactions dealing with air- or moisture-sensitive compounds were carried out in a dry reaction vessel under a positive pressure of nitrogen or in the nitrogen-filled glovebox. Unless otherwise noted, all reagents and solvents were purchased from commercial suppliers without further purification. Anhydrous solvents were purchased from Sigma-Aldrich and transferred by syringe. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker ADVANCE III (400 MHz) spectrometer with $CDCl_3$ or $(CD_3)_2CO$ as the solvent and tetramethylsilane (TMS) as the internal standard. Chemical shifts are reported in parts per million (ppm, δ scale) downfield from TMS at 0.00 ppm and referenced to the $CDCl_3$ at 7.26 ppm (for ^{1}H NMR) or 77.0 ppm (for ^{13}C NMR) or to $(CD_3)_2CO$ at 2.05 ppm (for ^{1}H NMR) or 29.8 ppm, 206.3 ppm (for ^{13}C NMR). Data are reported as: multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant in hertz (Hz) and signal area integration in natural numbers. ^{13}C NMR analyses were run with decoupling. Enantiomeric excess values were determined by Daicel chiral column on an Agilent 1260 Series HPLC instrument. Optical rotations $[\alpha]_D$ were measured on a PERKIN ELMER polarimeter 343 instrucment.

All the 3-substituted maleic anhydrides were prepared according the literature.[1] The absolute configuration of products **2a** and **2l** were determined by comparison of analytical data with the literature (HPLC spectra, optical rotation).[2] The absolute configuration of others were assigned by analogy.

II. General Procedure for the Synthesis of the substrates

Method A: [1a]



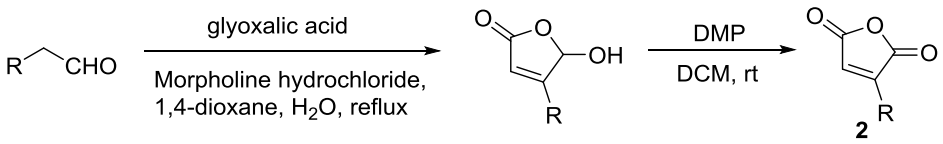
Condensation of arylacetonitriles with glyoxylic acid. A mixture of 20 mmol of the appropriate arylacetonitrile, 25 mmol of glyoxylicacid hydrate, and 50 mmol of potassium carbonate in 40 mL of methanol was stirred at room temperature or reflux for 3-24 h. The resulting thick solid precipitate was filtered and washed with dichloromethane. This solid was suspended in 100 mL of cold water,

stirred overnight, and then filtered and air dried to provide the corresponding potassium (2)-3-aryl-3-cyanopropenoate.

The potassium (*Z*)-3-aryl-3-cyanopropenoates (10 mmol) were dissolved in ca. 25 mL of 88% formic acid containing 1.5 mL of concentrated sulfuric acid. These mixtures were heated at reflux for 2-3 h and then cooled and poured into ice water. The resulting solids were filtered, washed with water, and air dried to provide the arylmaleic anhydrides.

Substrates **1a-1j** were prepared through method A.

Method B: [1b]

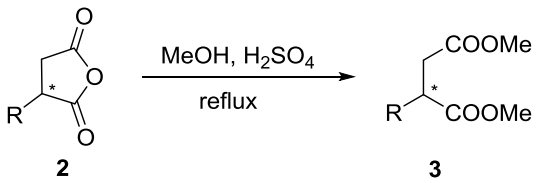


Solid morpholine hydrochloride (3.4 g, 27.5 mmol) was added to a solution of glyoxylic acid monohydrate (2.3 g, 25 mmol) in 20 mL of dioxane. To this suspension was added dropwise 3 mL of H_2O, after which all of the solid material was dissolved. A solution of appropriate alkylaldehyde (26.25 mmol) in 5 mL of dioxane was added via syringe, and the colorless solution was stirred at ambient temperature for 3 h and then heated to 100 ℃ and stirred overnight. The reaction mixture was evaporated and extracted 3 times with Et_2O, and the combined organic layers were then washed with saturated $NaHCO_3$ (aq.) and brine, dried over $MgSO_4$, and concentrated. The residue was purified with column chromatography to obtain desired 5-hydroxy-4-aryl-2(5H)-furanone.

To a stirred solution of 5-hydroxy-4-aryl-2(5H)-furanone (2 mmol) in DCM (10 mL) was added Dess−Martin periodinane (4 mmol) in one portion and the mixture was stirred for further 3 h in room temperature. After TLC shows starting material has disappeared, the reaction mixture was concentrated under reduced pressure, and the resulting residue was separated by column chromatography (hexane:EtOAc = 10:1) to give the desired maleic anhydrides.

Substrates **1k-1n** were prepared through method B.

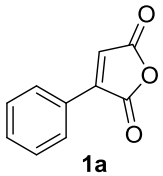
Esterification: [3]



A solution of succinic anhydride **2** (0.1 mmol) in dry methanol (1 mL) containing concentrated sulfuric acid (1 drops) was heated at reflux under anhydrous conditions. After 12 h, the solution was concentrated to near dryness on a rotary vacuum evaporator. The residue was dissolved in chloroform and the solution was washed successively with saturated $NaHCO_3$ and water before being dried over anhydrous $MgSO_4$. Removal of solvent under reduced pressure afforded dimethyl succinate as a colorless oil that was sufficiently pure to be used directly in the next step without further purification.

The configuration of **2a** and **2l** was determined as by comparing the optical rotation data with the reported by the literature after esterification of the anhydride.[2a, 2b] The configuration of **2j** and **2k** was determined by comparing the optical rotation data with the reported by the literature directly.[2a]

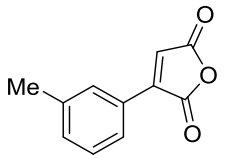
2-phenylmaleic anhydride **1a**



1a

White solid; 1.3 g, 38% total yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.00-7.97 (m, 2H), 7.60-7.50 (m, 3H), 7.01 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 164.6, 163.7, 146.9, 132.8, 129.4, 129.1, 126.8, 124.5. HRMS (ESI): [M+H$^+$] Calc. 175.0395, found 175.0390.

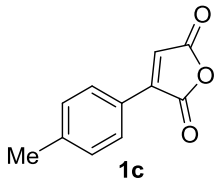
2-(3-methylphenyl)-maleic anhydride **1b**



1b

White solid, 410 mg, 22% total yield (10 mmol scale); ^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ (ppm) 7.79-7.77 (m, 2H), 7.43-7.37 (m, 2H), 6.98 (s, 1H), 2.43 (s, 3H); ^{13}C NMR (101 MHz, $(CD_3)_2CO$): δ (ppm) 168.2, 165.3, 149.6, 138.7, 133.8, 131.1, 128.9, 127.3, 124.0, 116.3, 20.5. HRMS (ESI): [M+H$^+$] Calc. 189.0552, found 189.0545.

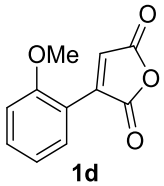
2-(4-methylphenyl)-maleic anhydride **1c**



1c

White solid; 180 mg, 10% total yield (10 mmol scale); ^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ (ppm) 8.01 (d, *J* = 8.0Hz, 2H), 7.45 (s, 1H), 7.39 (d, *J* = 8.0 Hz, 2H), 2.42 (s, 3H); ^{13}C NMR (101 MHz, $(CD_3)_2CO$): δ (ppm) 165.4, 164.4, 146.4, 143.1, 129.8, 129.0, 125.0, 124.6, 20.7. HRMS (ESI): [M+H$^+$] Calc. 189.0552, found 189.0545.

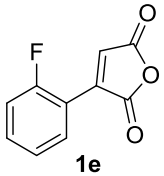
2-(2-methoxyphenyl)-maleic anhydride **1d**



1d

Yellow solid; 210 mg, 11% total yield (10 mmol scale); ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.35-8.33 (m, 1H), 7.54-7.50 (m, 1H), 7.39 (s, 1H), 7.12-7.08 (m, 1H), 7.04-7.02 (m, 1H), 3.97 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 165.6, 165.1, 160.0, 141.9, 133.8, 131.7, 127.6, 121.1, 116.2, 111.2, 55.8. HRMS (ESI): [M+H$^+$] Calc. 205.0501, found 204.0495.

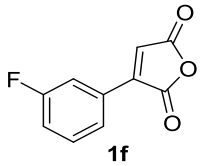
2-(2-fluorophenyl)-maleic anhydride **1e**



1e

White yellow solid, mp 95-99 °C; 580 mg, 15% total yield; ^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ (ppm) 8.27-8.23 (m, 1H), 7.72-7.67 (m, 1H), 7.47-7.38 (m, 2H), 7.45 (s, 1H); ^{13}C NMR (101 MHz, $(CD_3)_2CO$): δ (ppm) 165.0, 164.3, 161.8 (d, *J* =253.0 Hz), 140.5, 134.1 (d, *J* = 10.1 Hz), 131.2 (d, *J* = 2.0 Hz), 129.4 (d, *J* = 13.1 Hz), 124.9 (d, *J* = 4.0 Hz), 116.4 (d, *J* = 22.2 Hz), 115.9 (d, *J* = 11.1 Hz). HRMS (ESI): [M+H$^+$] Calc. 193.0301, found 193.0295.

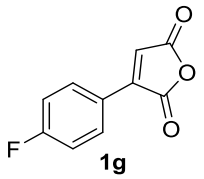
2-(3-fluorophenyl)-maleic anhydride **1f**



1f

Light yellow solid, mp 88-91 °C; 930 mg, 24% total yield; ^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ (ppm) 7.84-7.81 (m, 1H), 7.77-7.73 (m, 1H), 7.53-7.48 (m, 1H), 7.51 (s, 1H), 7.03-7.25 (m, 1H); ^{13}C NMR (101 MHz, $(CD_3)_2CO$): δ (ppm) 165.0, 164.0, 162.7 (d, *J* = 246.4 Hz), 145.2 (d, *J* = 3.0 Hz), 131.2 (d, *J* = 8.0 Hz), 129.7 (d, *J* = 9.0 Hz), 127.3, 125.2 (d, *J* = 3.0 Hz), 118.9 (d, *J* = 21.2 Hz), 115.6 (d, *J* = 23.2 Hz). HRMS (ESI): [M+H$^+$] Calc. 193.0301, found 193.0295.

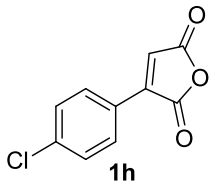
2-(4-fluorophenyl)-maleic anhydride **1g**



1g

White solid; 580 mg, 15% total yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.05-8.01 (m, 2H), 7.24-7.20 (m, 2H), 6.97 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 165.3 (d, *J* = 254.0 Hz), 164.6, 163.5, 145.7, 131.5 (d, *J* = 10.0 Hz), 123.9 (d, *J* = 2.0 Hz), 123.1 (d, *J* = 4.0 Hz), 116.9 (d, *J* = 22.2 Hz). HRMS (ESI): [M-H$^+$] Calc. 193.0301, found 193.0295.

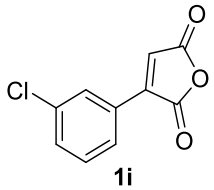
2-(4-chlorophenyl)-maleic anhydride **1h**



1h

Light yellow solid; 1.8 g, 43% total yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.95-7.93 (m, 2H), 7.52-7.50 (m, 2H), 7.01 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 164.4, 163.4, 145.7, 139.4, 130.3, 129.8, 125.2, 124.7. HRMS (ESI): [M+H$^+$] Calc. 209.0005, found 209.0000.

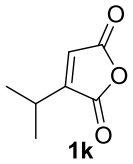
2-(3-chlorophenyl)-maleic anhydride **1i**



1i

White solid, mp 89-94 °C; 500 mg, 24% total yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.97 (s, 1H), 7.89-7.87 (m, 1H), 7.57-7.54 (m, 1H), 7.49-7.46 (m, 1H), 7.05 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 164.1, 163.2, 145.6, 135.6, 132.7, 130.7, 128.9, 128.3, 127.1, 125.7. HRMS (ESI): [M+H$^+$] Calc. 209.0005, found 209.0000.

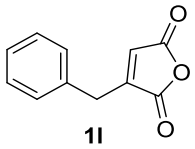
N-phenylmethyl-2-phenyl-maleic anhydride **1k**



1k

Colorless liquid; 310 mg, 9% total yield; ^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ (ppm) 6.55 (s, 1H), 2.91-2.84 (m, 1H), 1.26 (d, *J* = 8.0 Hz, 6H); ^{13}C NMR (101 MHz, $(CD_3)_2CO$): δ (ppm) 165.3, 164.1, 159.3, 127.1, 26.6, 20.5. HRMS (ESI): [M+H$^+$] Calc. 141.0552, found 141.0545.

N-phenyl-2-phenyl-maleic anhydride **1l**



1l

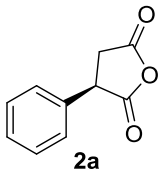
White solid; 550mg, 24% total yield (10 mmol scale); mp 87-91 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.38-7.33 (m, 3H), 7.24-7.22 (m, 2H), 6.42-6.41 (m, 1H), 2.82 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$): δ (ppm) 165.6, 163.6, 152.8, 134.3, 129.5, 129.3, 129.0, 127.8, 32.4. HRMS (ESI): [M+H$^+$] Calc. 189.0552, found 189.0546.

III. General Procedure for Asymmetric Hydrogenation of Chiral Maleic Anhydrides

In the nitrogen-filled glovebox, a solution of ZhaoPhos (1.0 eq.) and $Rh(NBD)_2BF_4$ (3.7 mg, 0.005 mmol) in 1.0 mL anhydrous ethyl acetate (EA) was stirred at room temperature for 40 min.

100 uL of the resulting solution transferred by syringe into a vial charged with **1** (0.1 mmol) in 0.9 mL anhydrous EA. The vials were transferred to an autoclave, which was then charged with 1 atm of H_2 and stirred at room temperature for 30min. The solution was passed through short column of silica gel (about 4 cm) to remove the metal complex and concentrated. The product was analyzed by NMR spectroscopy for conversion and chiral HPLC or GC for ee values.

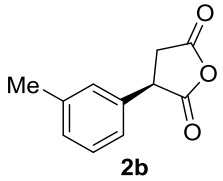
(*R*)-3-phenylsuccinic anhydride **2a**



2a

White solid; >99% conversion; 17.1 mg, 98% yield; 97% ee; $[\alpha]_D^{20}$ = -76.09 (c = 1.0, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 98:2; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 46.8 min (major), 52.8 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.44-7.36 (m, 3H), 7.28-7.25 (m, 2H), 4.37-4.33 (m, 1H), 3.51-3.44 (m, 1H), 3.16-3.10 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 170.5, 168.4, 133.5, 128.5, 127.7, 126.2, 45.5, 35.7. HRMS (ESI): [M+H$^+$] Calc. 177.0552, found 177.0546.

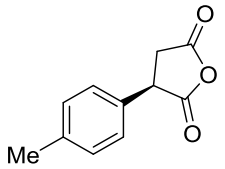
(*R*)-3-(2-methoxyphenyl)-succinic anhydride **2b**



2b

Colorless liquid; >99% conversion; 18.1 mg, 95% yield; 96% ee; $[\alpha]_D^{20}$ = -65.70 (c = 1.0, EA); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 97:3; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 65.8 min (major), 75.2 min (minor). ^{1}H NMR (400 MHz, $(CD_3)_2CO$) δ = 7.31-7.28 (m, 2H), 7.24-7.23 (m, 1H), 7.19-7.17 (m, 1H), 4.65-4.60 (m, 1H), 3.62-3.55 (m, 1H), 3.30-3.24 m, 1H), 2.34 (s, 3H); ^{13}C NMR (101 MHz, $(CD_3)_2CO$) δ = 172.9, 170.5, 138.6, 136.0, 128.9, 128.7, 128.6, 125.1, 46.6, 36.5, 20.5. HRMS (ESI): [M+H$^+$] Calc. 189.0552, found 189.0546.

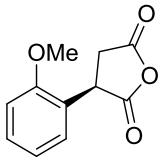
(*R*)-3-(3-methoxyphenyl)-succinic anhydride **2c**



2c

White solid, mp 153-157 °C; >99% conversion; 18.6 mg, 98% yield; >99% ee; $[\alpha]_D^{20}$ = -60.60 (c = 1.0, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AS-H column, hexane: isopropanol = 95:5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 70.4 min (major), 83.8 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.23-7.21 (m, 2H), 7.16-7.12 (m, 2H) 4.32-4.28 (m, 1H), 3.48-3.40 (m, 1H), 3.13-3.06 m, 1H), 2.36 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 170.8, 168.6, 137.6, 130.6, 129.1, 126.1, 45.2, 35.7, 20.1. HRMS (ESI): [M+H$^+$] Calc. 189.0552, found 189.0546.

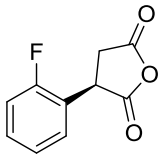
(*R*)-3-(4-methoxyphenyl)-succinic anhydride **2d**



2d

Yellow solid; >99% conversion; 20.4 mg, 99% yield; 96% ee; $[\alpha]_D^{20}$ = -81.93 (c = 1.0, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 90:10; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 19.0 min (major), 24.6 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.38-7.33 (m, 1H), 7.21-7.19 (m, 1H), 6.99-6.92 (m, 2H), 4.16-4.12 (m, 1H), 3.83 (s, 1H), 3.33-3.25 (m, 1H), 3.05-2.99 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 171.7, 169.6, 155.4, 129.9, 129.3, 123.2, 120.2, 110.3, 54.5, 43.6, 34.9. HRMS (ESI): [M+H$^+$] Calc. 207.0652, found 207.0648.

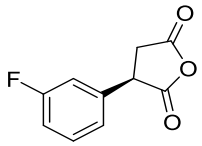
(*R*)-3-(2-fluorophenyl)-succinic anhydride **2e**



2e

Light yellow solid, mp 85-89 °C; >99% conversion; 18.5 mg, 95% yield; >99% ee; $[\alpha]_D^{25}$ = -33.40 (c = 1.0, EA); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 98:2; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 45.9 min (major), 55.8 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.41-7.36 (m, 1H), 7.30-7.26 (m, 1H), 7.21-7.13 (m, 2H), 4.41-4.36 (m, 1H), 3.50-3.42 (m, 1H), 3.11-3.05 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 170.1, 168.1, 159.4 (d, *J* = 247.5 Hz), 129.9 (d, *J* = 11.1 Hz), 129.2 (d, *J* = 4.0 Hz), 124.0 (d, *J* = 4.0 Hz), 121.6 (d, J_{cf}=14.1 Hz), 115.3 (d, *J* = 20.2 Hz), 41.4 (d, *J* = 1.0 Hz), 35.2 (d, *J* = 2.0 Hz). HRMS (ESI): [M+H$^+$] Calc. 195.0457, found 195.0453.

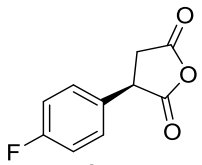
(*R*)-3-(3-fluorophenyl)-succinic anhydride **2f**



2f

White solid; >99% conversion; 18.2 mg, 94% yield; 95% ee; $[\alpha]_D^{20}$ = -52.04 (c = 1.0, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 93:7; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R =27.9 min (minor), 34.5 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.43-7.38 (m, 1H), 7.11-7.01 (m, 3H), 4.38-4.34 (m, 1H), 3.53-3.45 (m, 1H), 3.16-3.10 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.9, 167.9, 162.0 (d, *J* = 249.5 Hz), 135.5 (d, *J* = 7.0 Hz), 130.2 (d, *J* = 8.0 Hz), 121.9 (d, *J* = 3.0 Hz), 114.9 (d, *J* = 20.2 Hz), 113.8 (d, *J* = 23.2 Hz), 45.0 (d, *J* = 2.0 Hz), 35.4. HRMS (ESI): [M+H$^+$] Calc. 195.0457, found 195.0453.

(*R*)-3-(4-fluorophenyl)-succinic anhydride **2g**

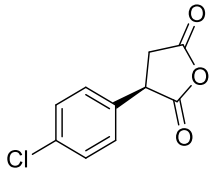


2g

Light yellow solid, mp 159-163 °C; >99% conversion; 18.6 mg, 96% yield; 98% ee; $[\alpha]_D^{20}$ = -86.90 (c = 1.0, $CHCl_3$); The enantiomeric excess of 4g was determined by corresponding di-methyl ester **3g**: HPLC on Chiralpak OJ-H column, hexane: isopropanol = 90:10; flow rate = 1.0 mL/min;

UV detection at 230 nm; t_R = 14.9 min (major), 16.2 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.29-7.24 (m, 2H), 7.14-7.09 (m, 2H), 4.37-4.33 (m, 1H), 3.52-3.44 (m, 1H), 3.14-3.07 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 170.4, 168.1, 161.7 (d, *J* = 248 Hz), 129.2 (d, *J* = 3.0 Hz), 128.1 (d, *J* = 8.0 Hz), 115.5 (d, *J* = 22.0 Hz), 44.8, 35.6. HRMS (ESI): [M+H$^+$] Calc. 195.0457, found 195.0453.

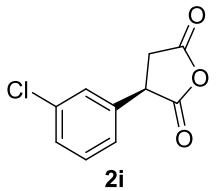
(*R*)-3-(4-chlorophenyl)-succinic anhydride **2h**



2h

Light yellow solid; >99% conversion; 20.4 mg, 98% yield; 93% ee; $[\alpha]_D^{20}$ = -72.60 (c = 1.0, $CHCl_3$); The enantiomeric excess of 4h was determined by corresponding methyl-ester 4h: HPLC on Chiralpak OJ-H column, hexane: isopropanol = 98:2; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 21.6 min (major), 24.9 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.41-7.38 (m, 2H), 7.24-7.21 (m, 2H), 4.36-4.32 (m, 1H), 3.51-3.44 (m, 1H), 3.13-3.06 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 170.2, 168.0, 133.8, 131.8, 128.7, 127.7, 44.8, 35.4. HRMS (ESI): [M-H$^+$] Calc. 209.0005, found 209.0004.

(*R*)-3-(4-bromophenyl)-succinic anhydride **2i**

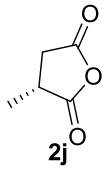


2i

White solid; >99% conversion; 20.7 mg, 97% yield; 93% ee; $[\alpha]_D^{20}$ = -59.10 (c = 1.0, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 93:7; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 27.4 min (major), 33.3 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.37-7.29 (m, 2H), 7.19 (d, *J* = 4.0 Hz, 1H), 7.17-7.16 (m, 1H), 4.36-4.32 (m, 1H), 3.52-3.44 (m, 1H), 3.15-3.09 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.9, 167.9, 135.2, 134.3, 129.8, 128.0, 126.7, 124.4, 45.0, 35.4. HRMS (ESI): [M-H$^+$] Calc.

209.0005, found 209.0004.

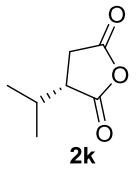
(*R*)-3-methyl-succinic anhydride **2j**



2j

Colorless liquid; >99% conversion; 11.3 mg, 99% yield; 96% ee; $[\alpha]_D^{20}$ = -36.60 (c = 1.0, $CHCl_3$); The enantiomeric excess was determined by GC on Chiralpak β-dex225 column, 100 ℃, 0 min, 10 ℃/min, 150 ℃, 25 min; t_R = 18.5 min (minor), 19.2 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 3.23-3.13 (m, 2H), 2.67-2.59 (m, 1H), 1.46 (d, *J* = 4.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 173.2, 178.8, 35.0, 34.6, 15.2. HRMS (ESI): [M+H$^+$] Calc. 115.0395, found 115.0392.

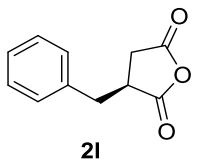
(*S*)- 3-isopropyl-succinic anhydride **2k**



2k

Colorless liquid; >99% conversion; 13.7 mg, 97% yield; 95% ee; $[\alpha]_D^{20}$ = 23.70 (c =1.0, $CHCl_3$); The enantiomeric excess was determined by GC on Chiralpak β-dex120 column, 100 ℃, 0 min, 2 ℃/min, 120 ℃, 25 min; t_R = 28.3 min (minor), 28.9 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 3.11-3.06 (m, 1H), 3.02-2.95 (m, 1H), 2.77-2.71 (m, 1H), 2.33-2.29 (m, 1H), 1.05 (d, *J* = 8.0 Hz, 3H), 1.00 (d, *J* = 8.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 171.8, 169.3, 45.7, 29.6, 28.2, 18.7, 17.0. HRMS (ESI): [M+H$^+$] Calc. 143.0708, found 143.0702.

(S)-3-benzyl-succinic anhydride **2l**



2l

White solid; >99% conversion; 18.3 mg, 96% yield; 90% ee; $[\alpha]_D^{20}$ = 42.70 (c = 1.0, $CHCl_3$)

(4l: $[\alpha]_D^{20}$ = +82.33 (c = 1.0, $CHCl_3$)); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 90:10; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 15.7 min (major), 17.2 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.37-7.27 (m, 3H), 7.19-7.17 (m, 2H), 3.48-3.42 (m, 1H), 3.27-3.22 (m, 1H), 3.05-2.92 (m, 2H), 2.75-2.69 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 172.1, 168.6, 134.8, 128.2, 127.9, 126.6, 41.1, 35.0, 31.9. HRMS (ESI): [M+H$^+$] Calc. 191.0708, found 191.0701.

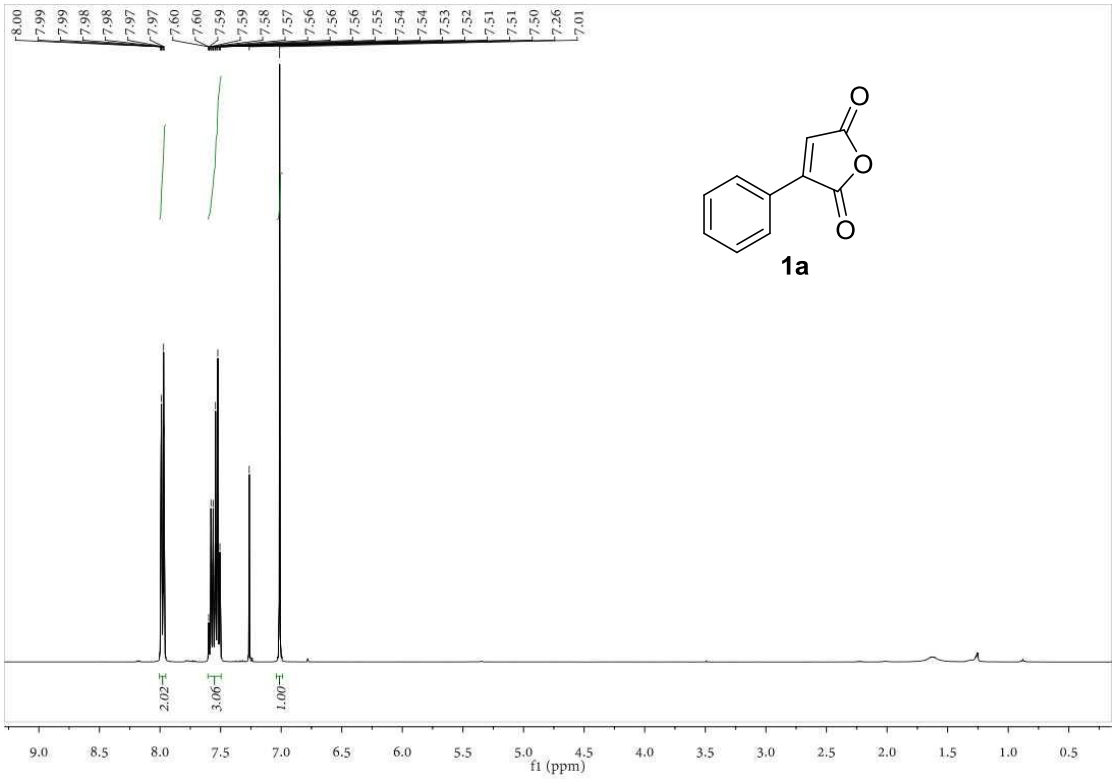
IV. Reference

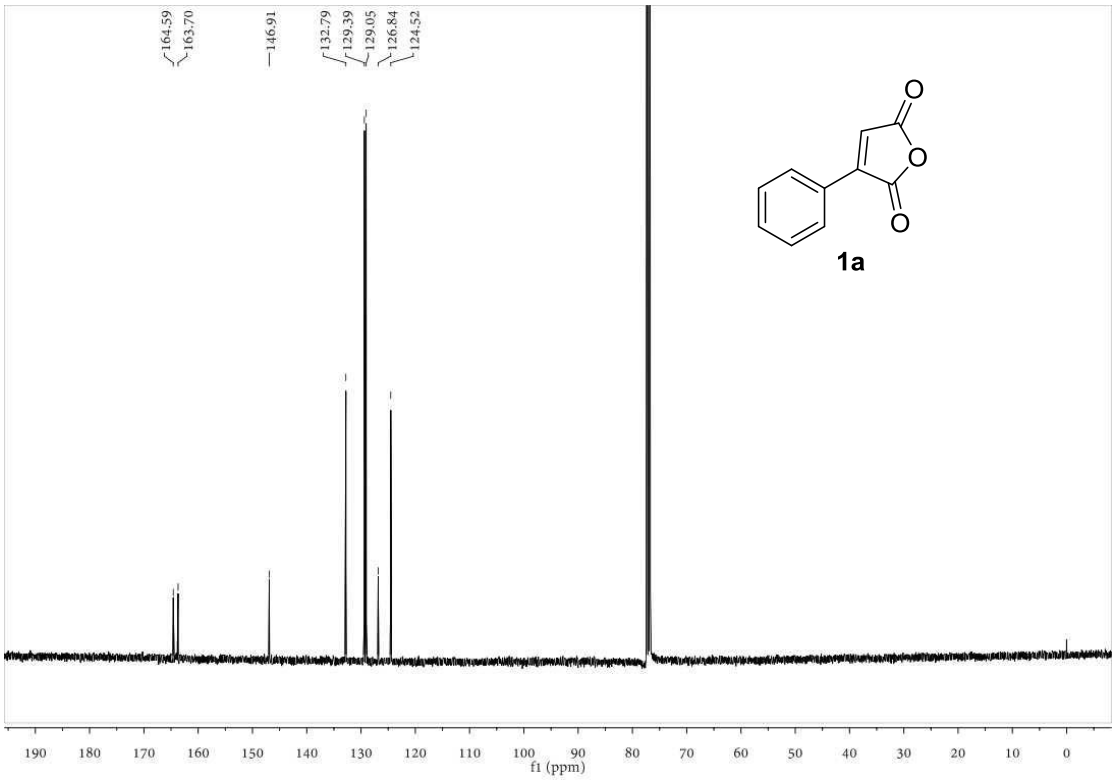
1. (a) Z. Y. Han, P. Li, Z. Zhang, C. Chen, Q. Wang, X. Q. Dong, X. Zhang, *ACS Catal.* 2016, **6**, 6214−6218. (b) Y. Chen, K. Tsao, J.W. Keillor, *J. Org. Chem.* 2015, **80**, 12182−12192.

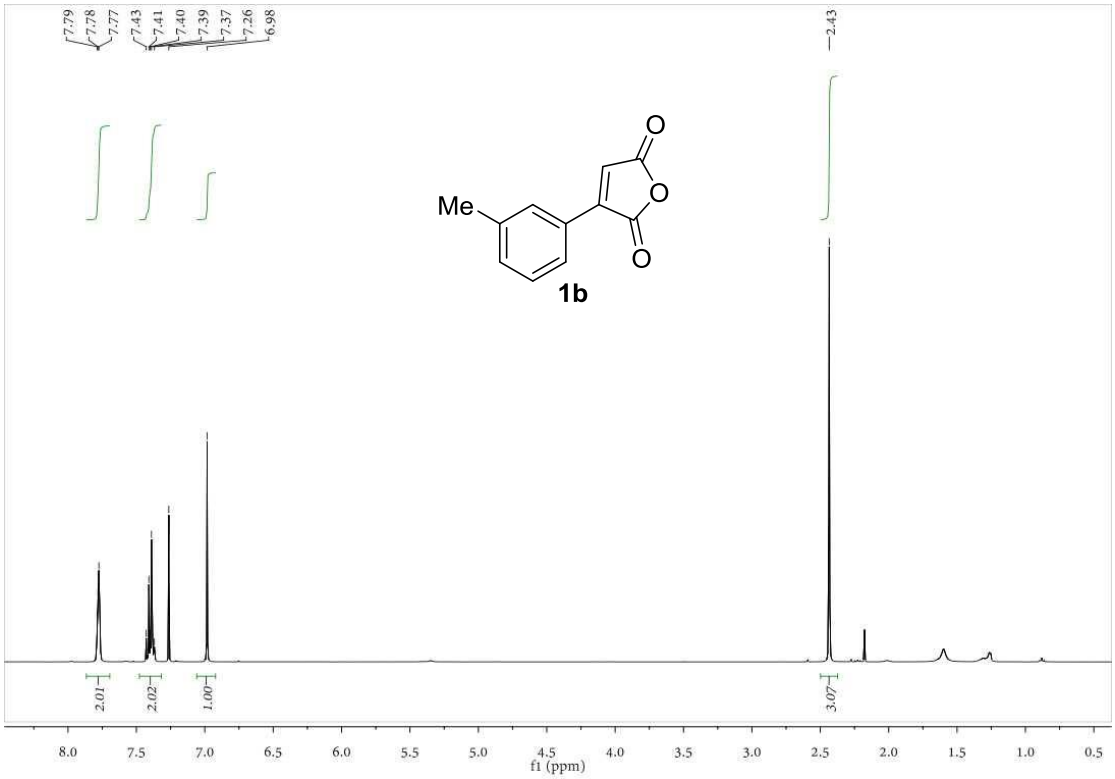
2. (a) B. Maurizio, M. Marc-Andre, P. Andreas, *Angew. Chem. Int. Ed.* 2014, **53**, 5385–5388. (b) L. F. Julio, K. Achim, S. Peter, W. Hubert, H. G. Lutz, *Chem.-Eur. J.* 2011, **17**, 14047–14062.

3. G. M. Strunz, C. M. Yu, L. Ya, *Canadian Journal of Chemistry*, 1990, **68**(5), 782-6.

V. NMR Spectra



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O
O
1b
2.01
2.02
1.00
3.07
8.0
7.5
7.0
6.5
6.0
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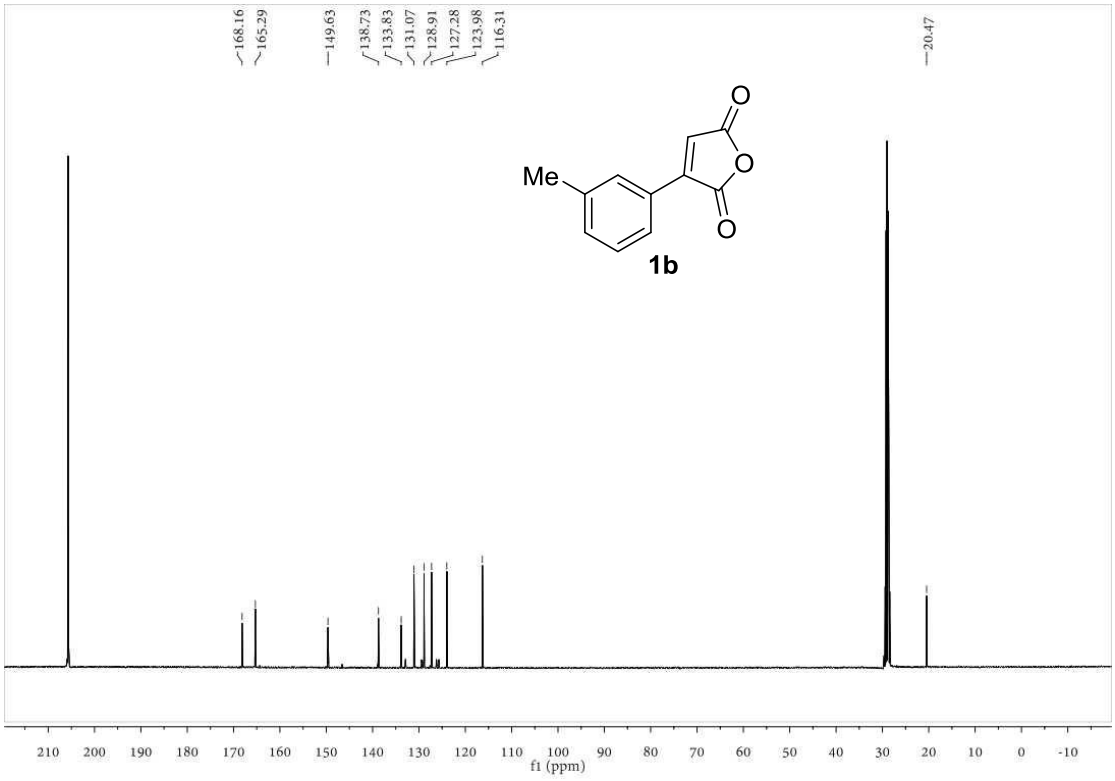
f1 (ppm)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O
O
1b
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200
190
180
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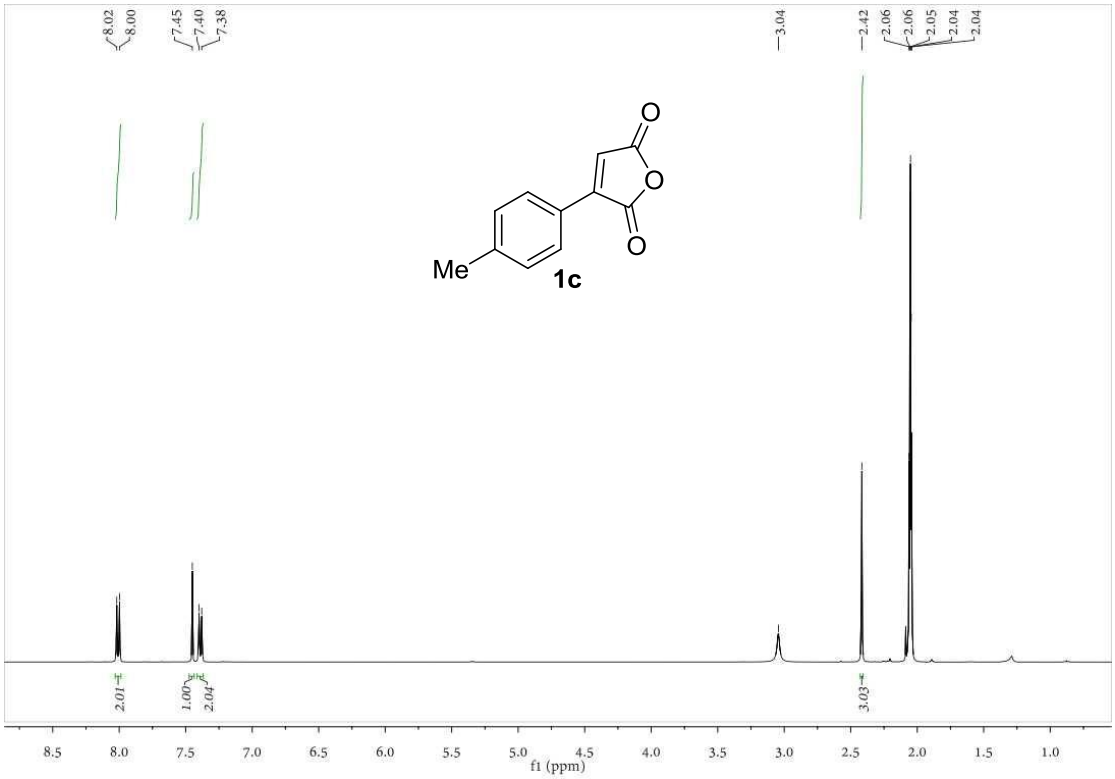
f1 (ppm)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1.00
2.04
3.03
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7.0
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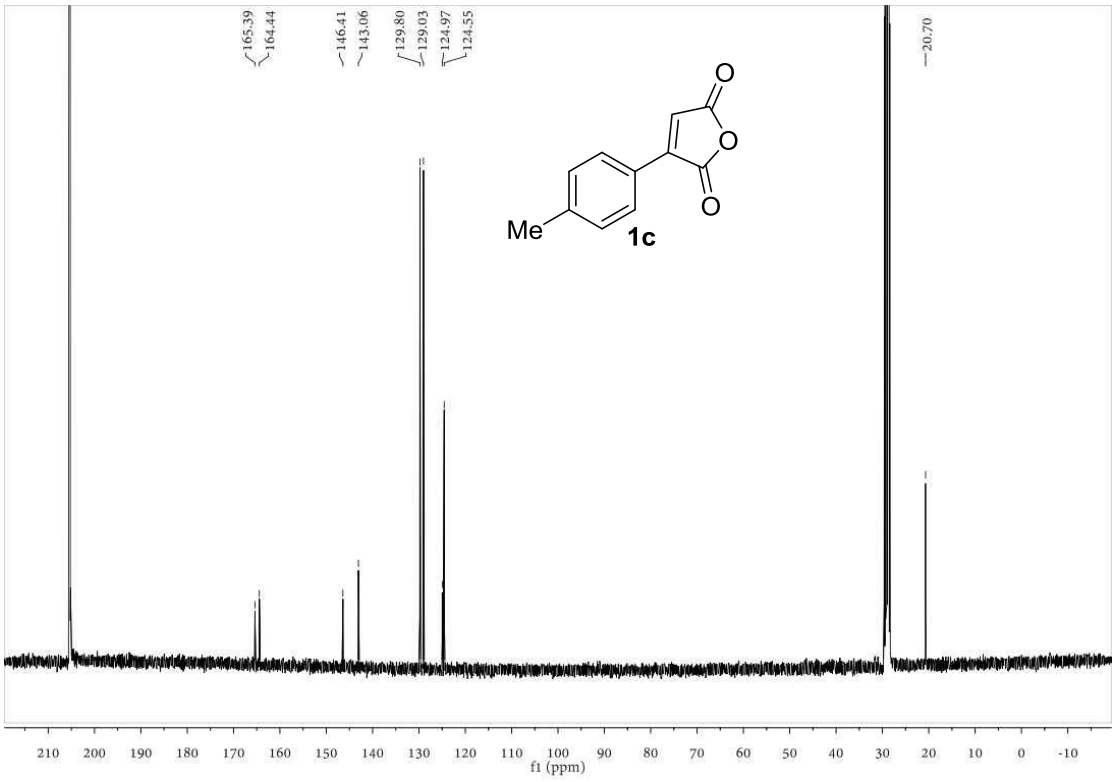
f1 (ppm)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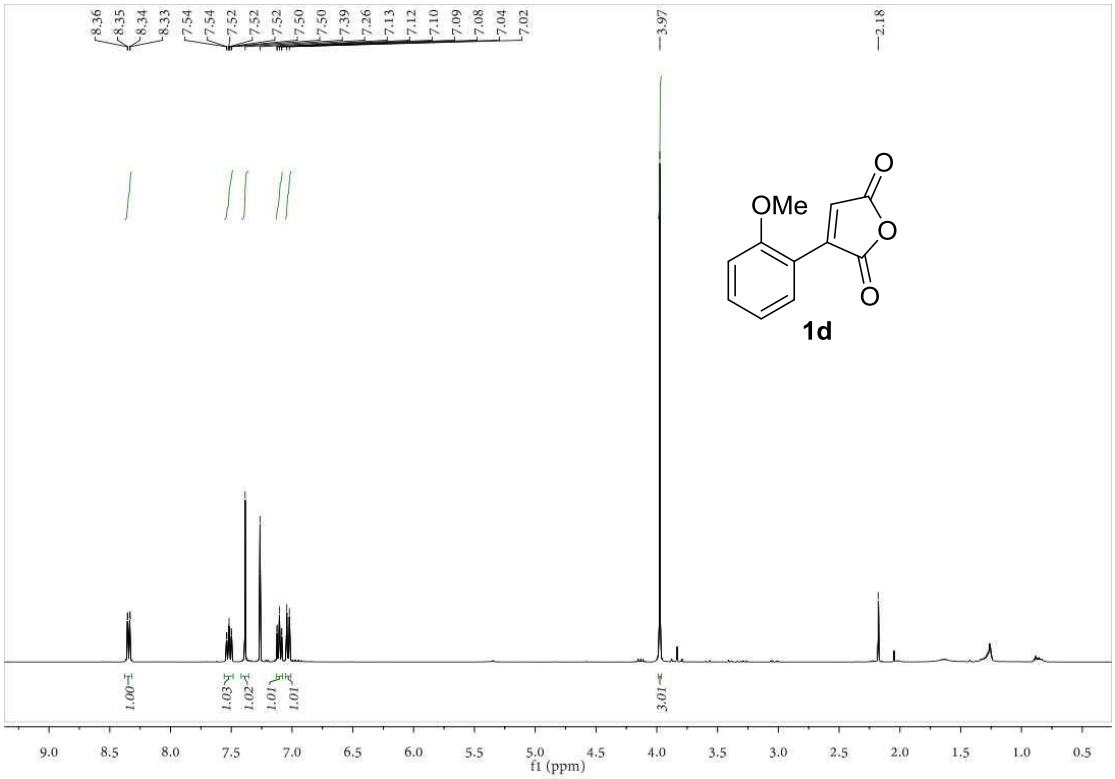
-10
f1 (ppm)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7.10
7.09
7.08
7.04
7.02
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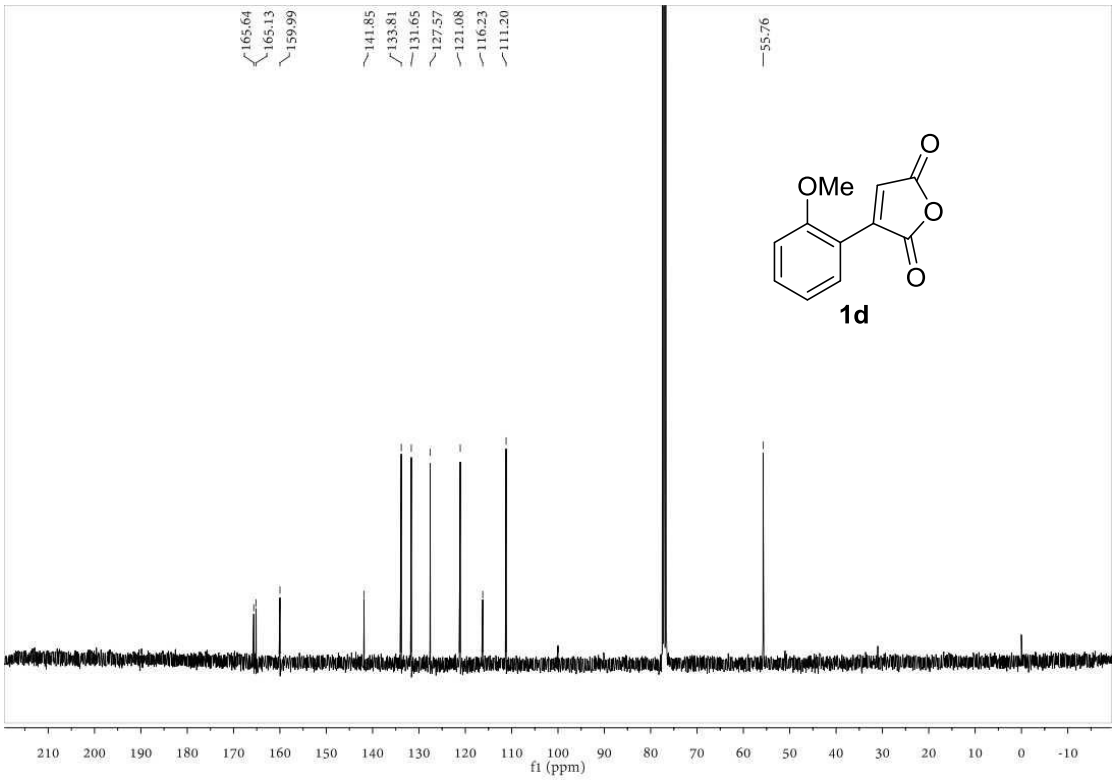
1.01
3.01
f1 (ppm)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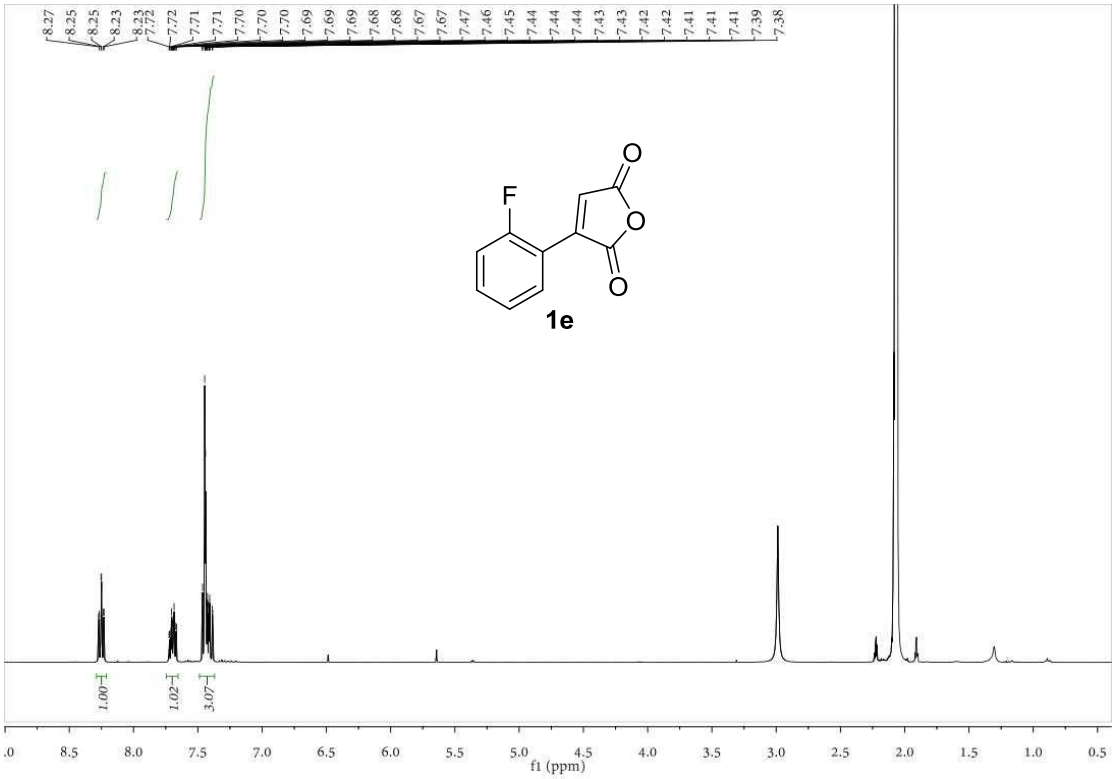
1d
f1 (ppm)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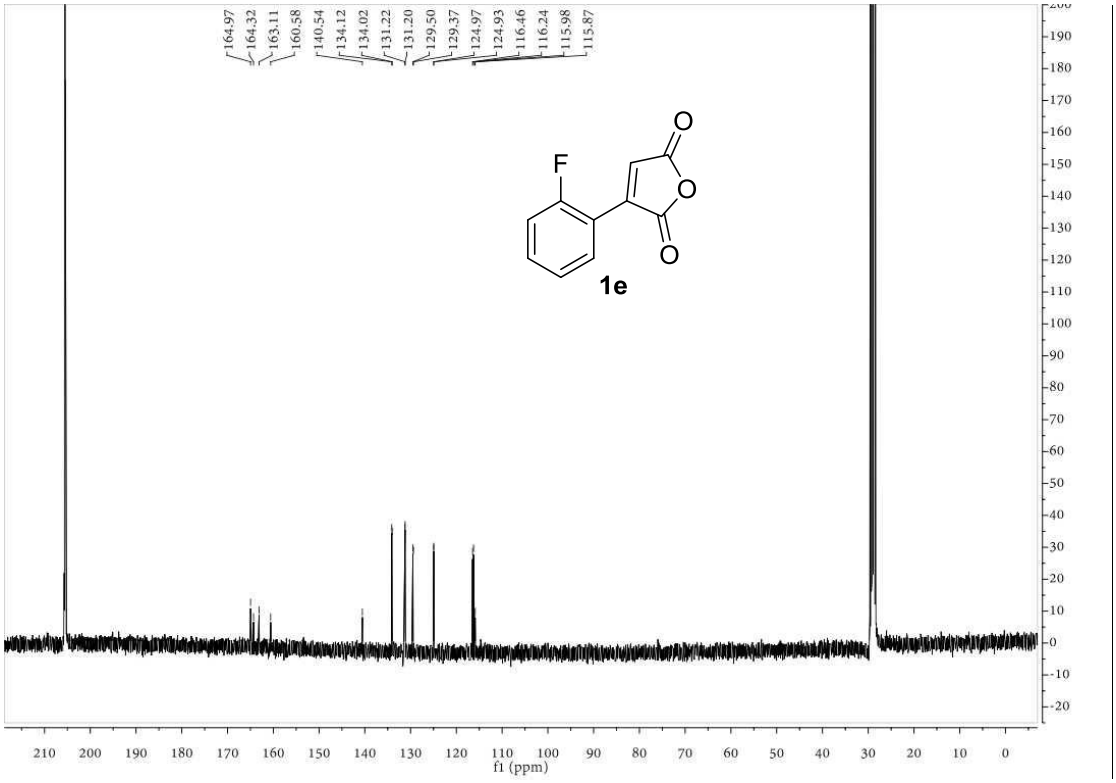
3.07
f1 (ppm)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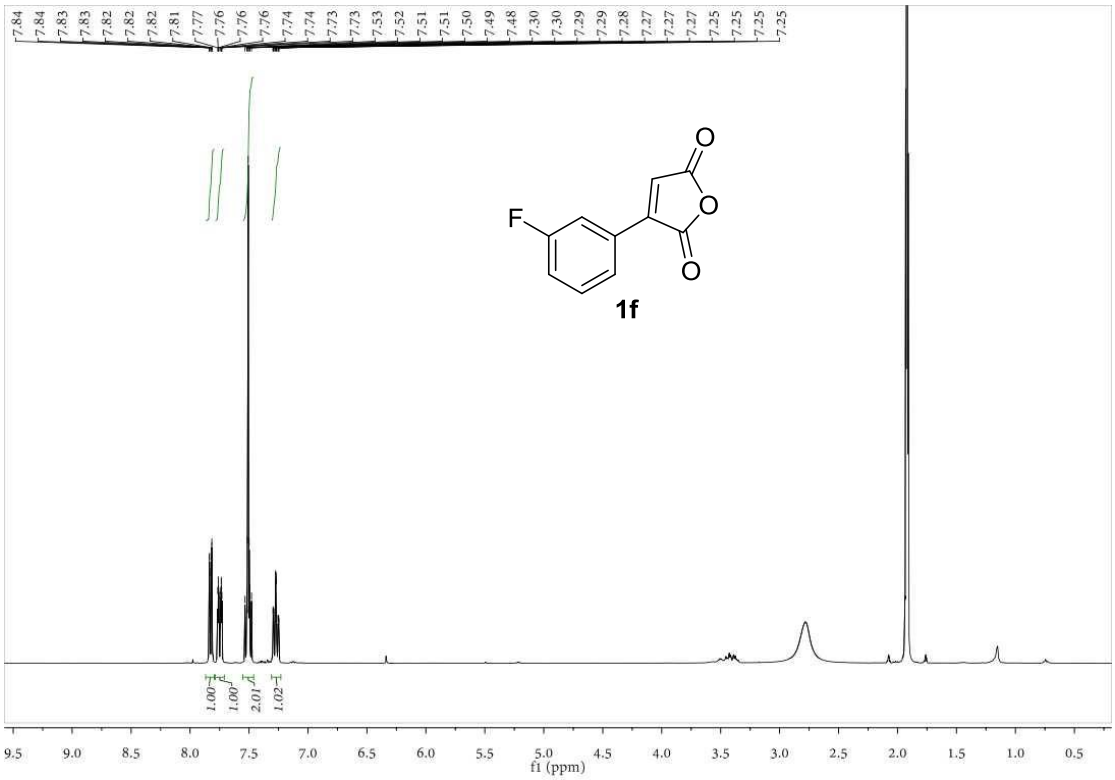
1e
f1 (ppm)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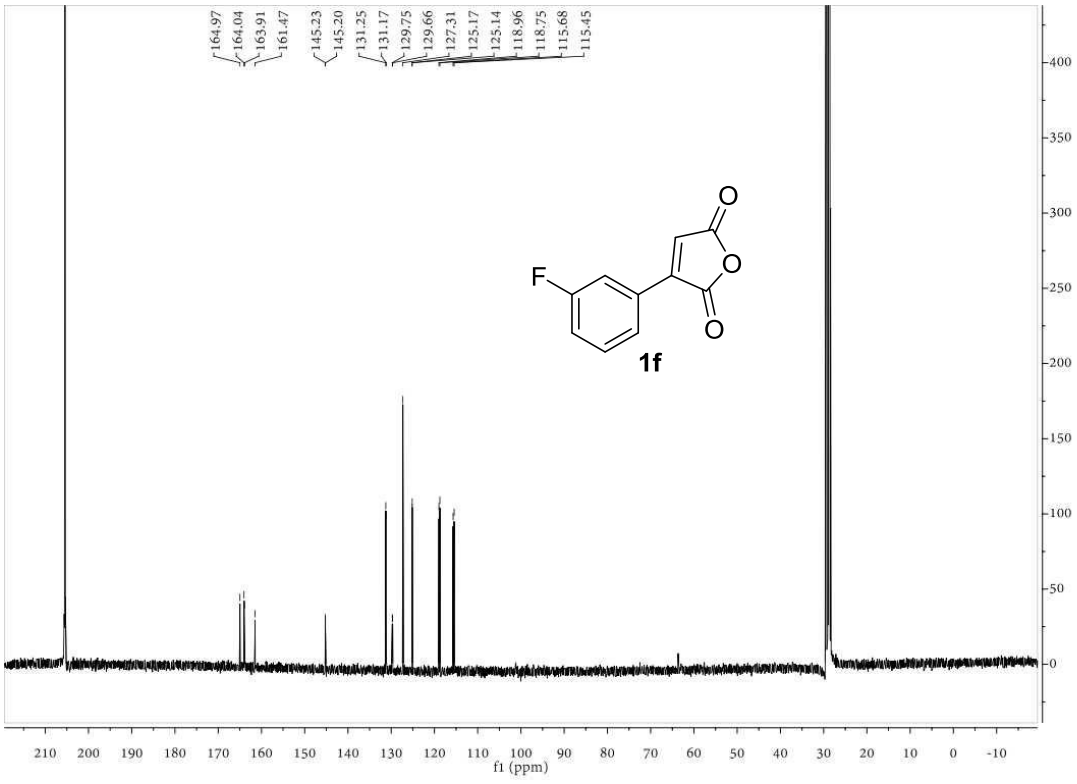
O
O
O
F
1f
1.00 1.00 2.01 1.02
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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O
O
F
1f
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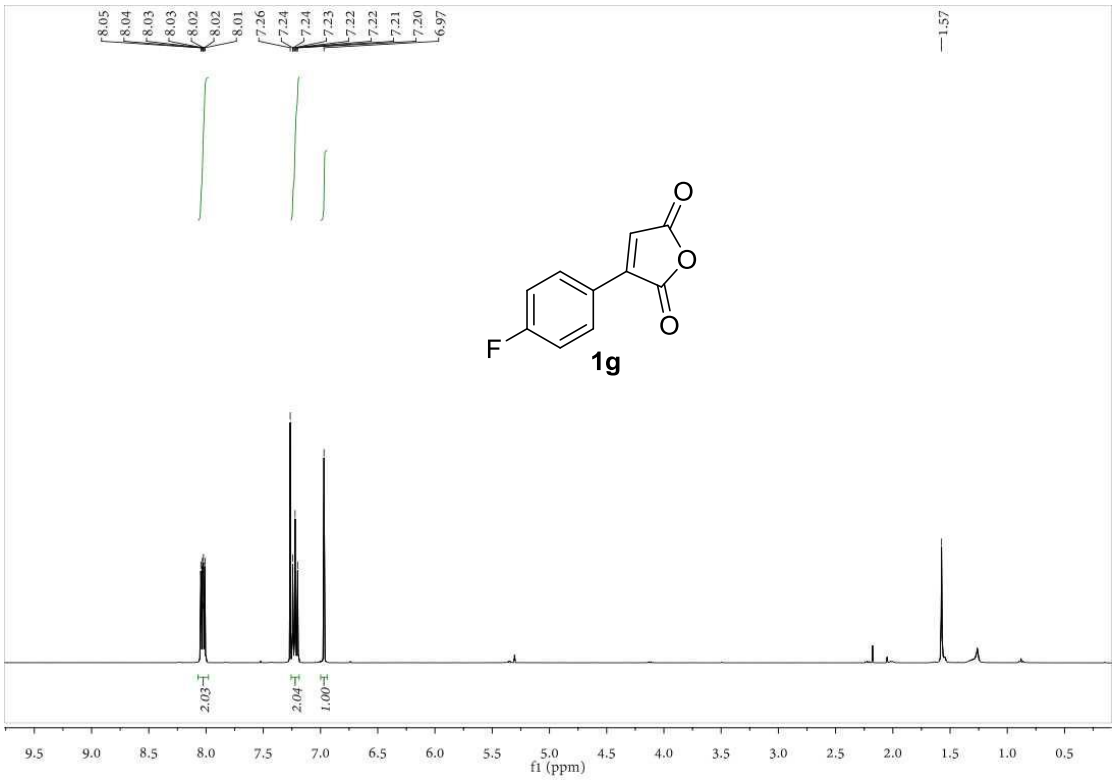
f1 (ppm)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1.00
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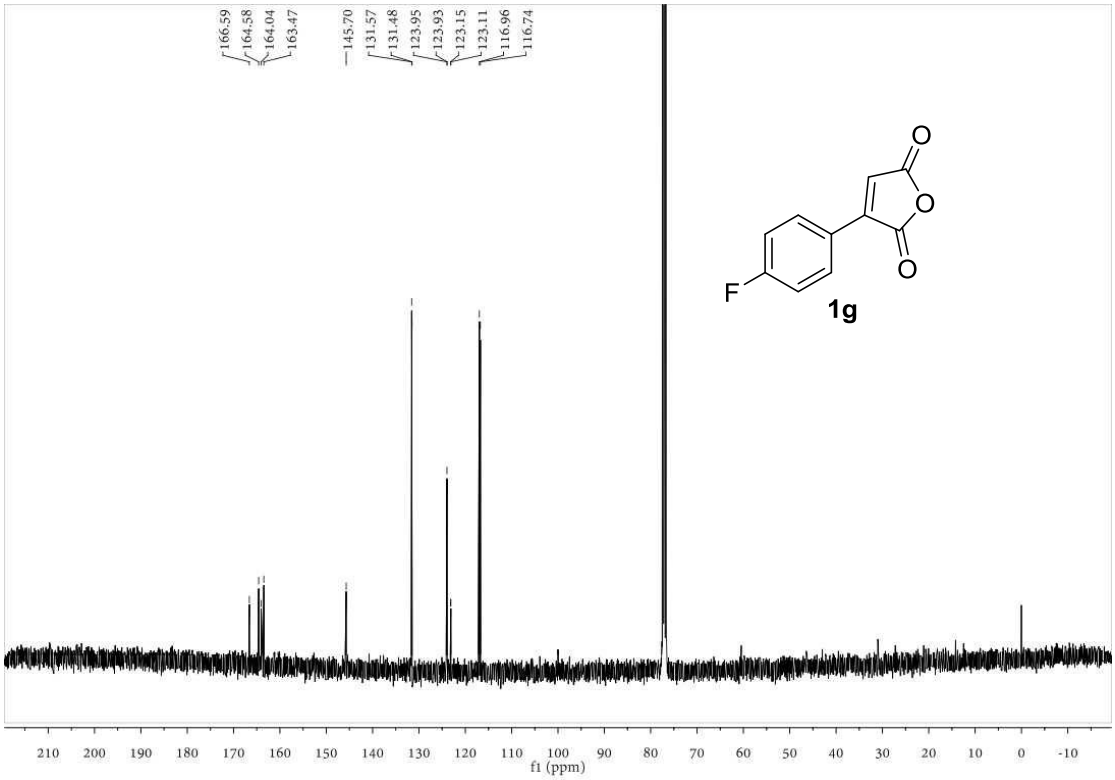
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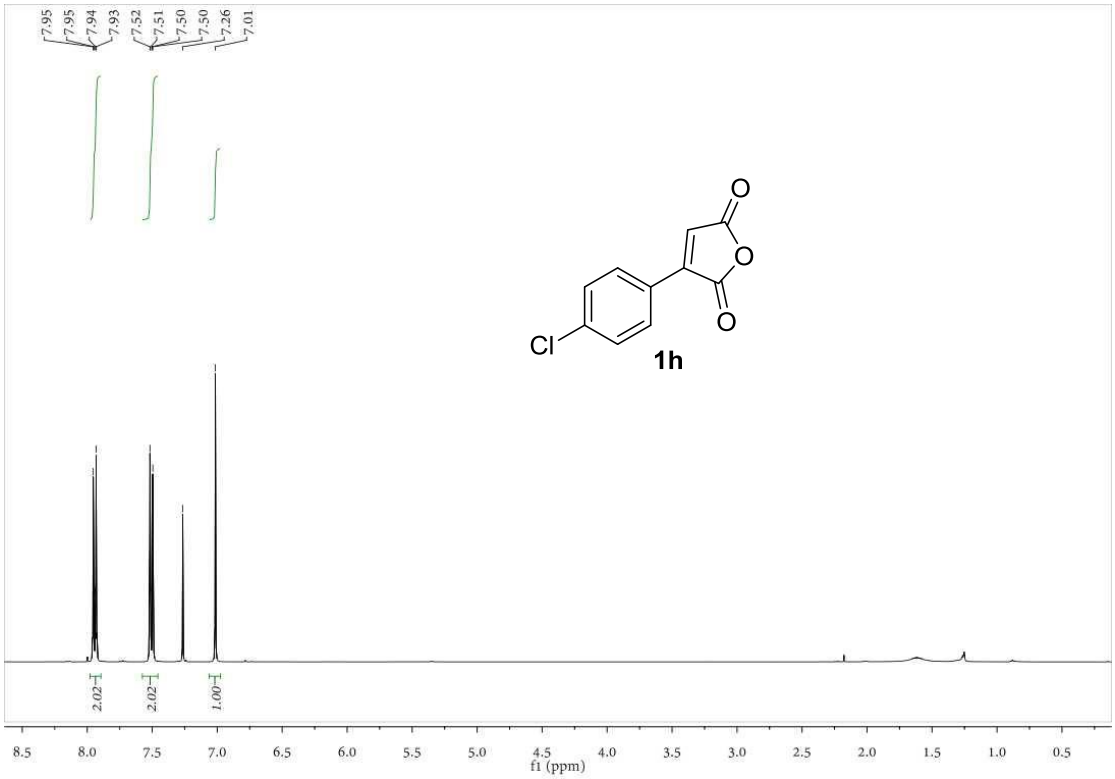
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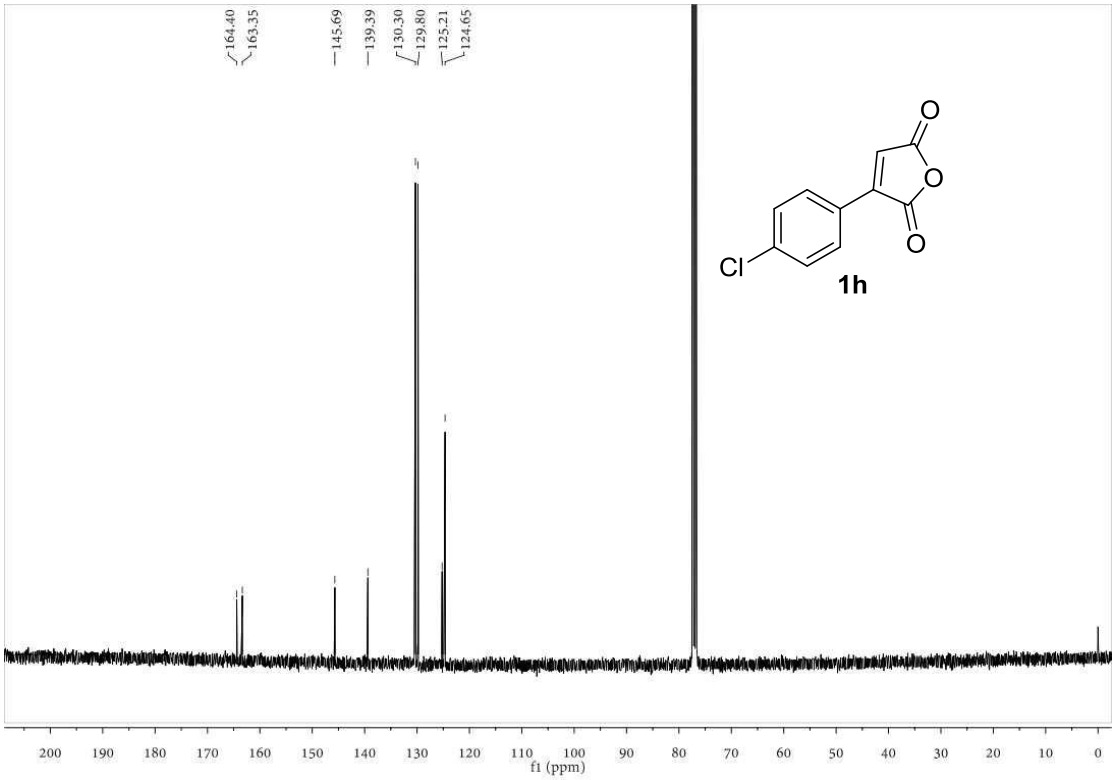
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70
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50
40
30
20
10
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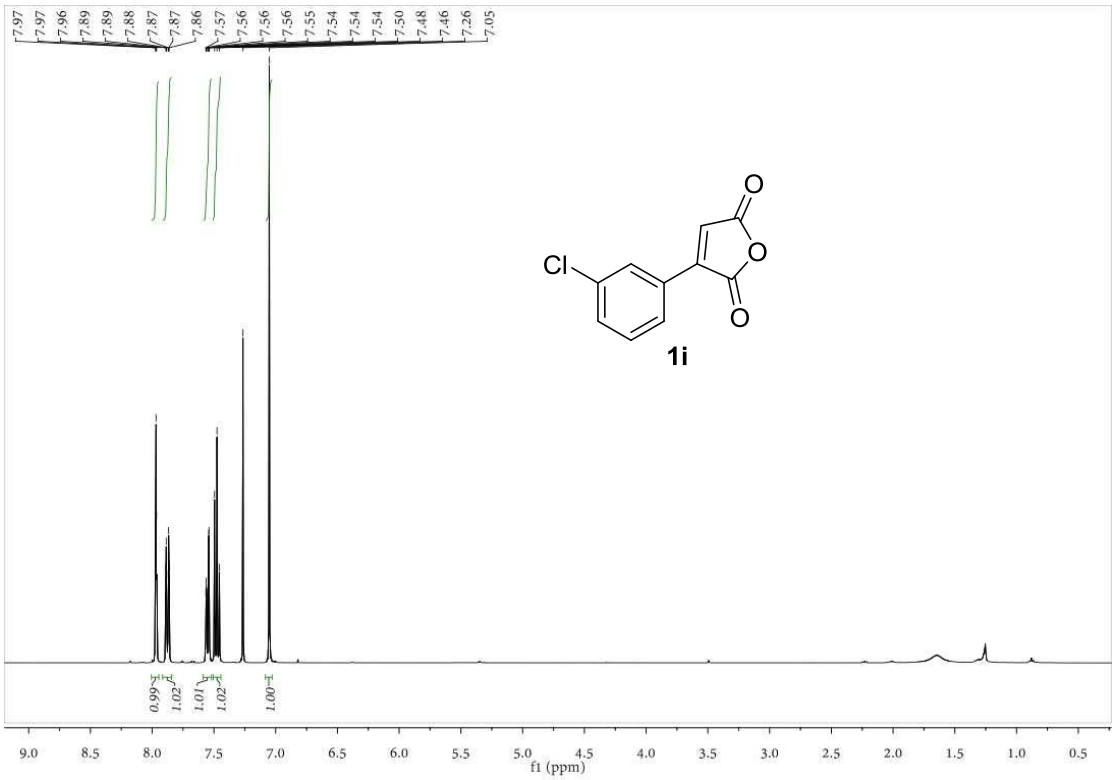
f1 (ppm)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7.87
7.87
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7.57
7.56
7.56
7.56
7.55
7.54
7.54
7.54
7.50
7.48
7.46
7.26
7.05
0.99
1.02
1.01
1.02
1.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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2.5
2.0
1.5
1.0
0.5
f1 (ppm)
Cl
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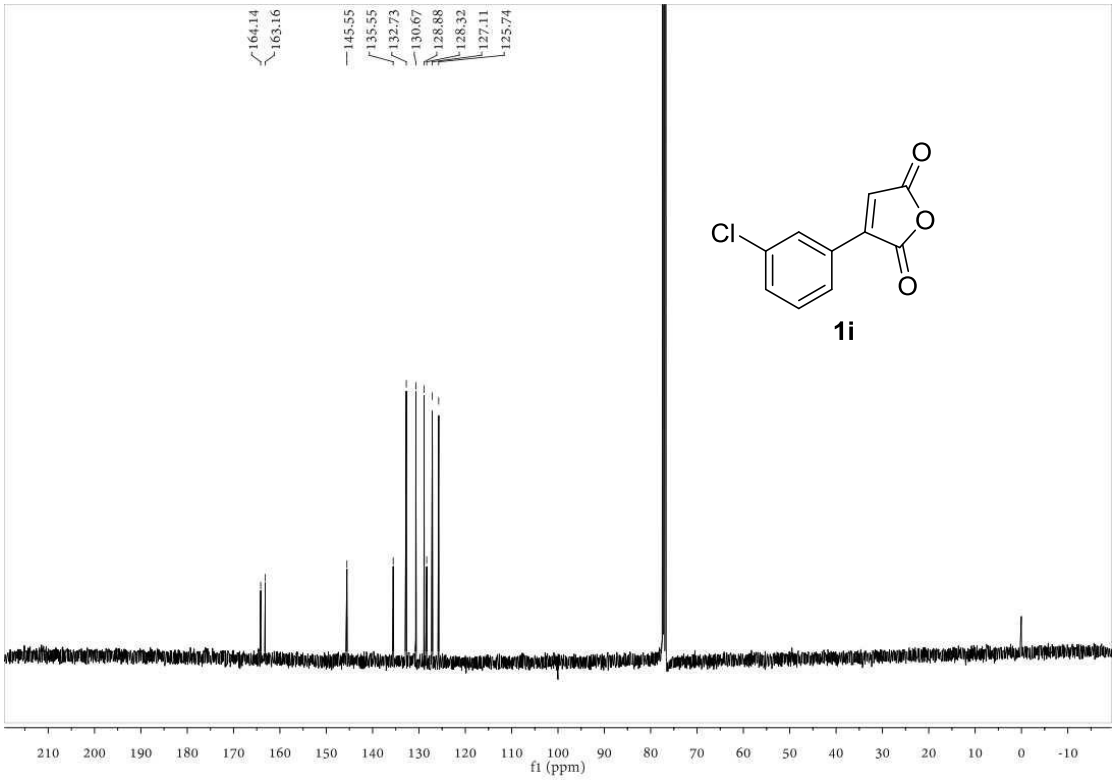
O
O
1i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128.32
127.11
125.74
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200
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180
170
160
150
140
130
120
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60
50
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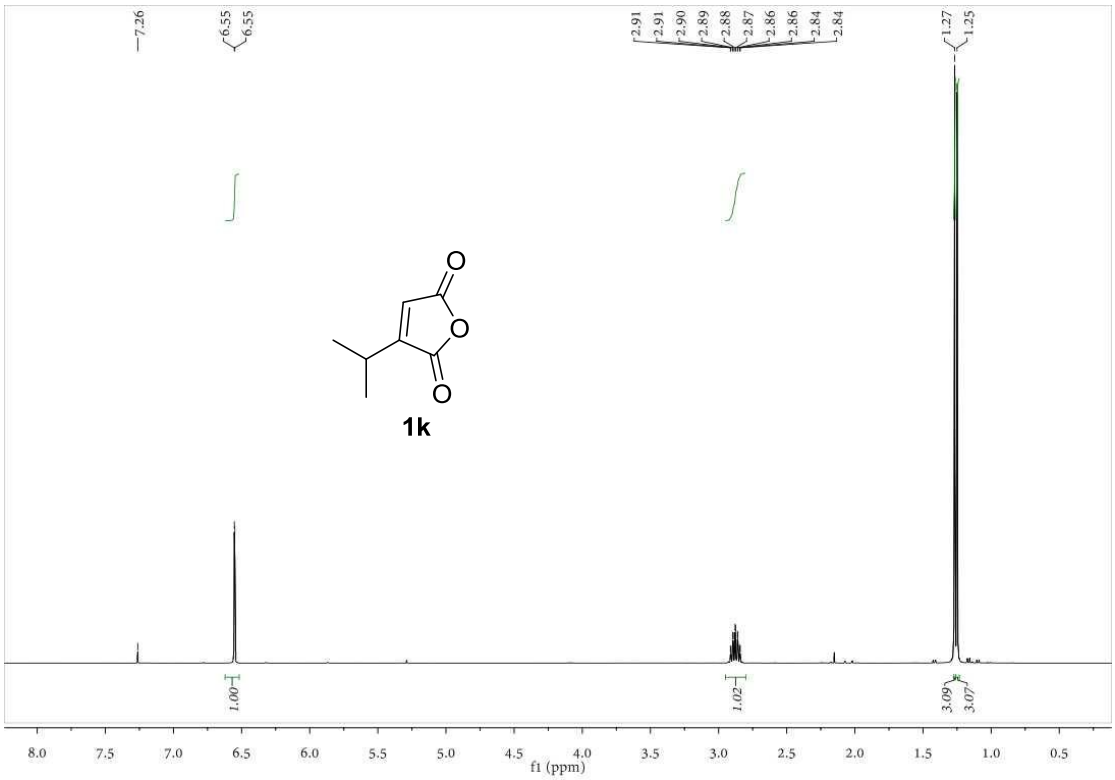
30
20
10
0
-10
f1 (ppm)
Cl
O
O
O
1i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2.88
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1.25
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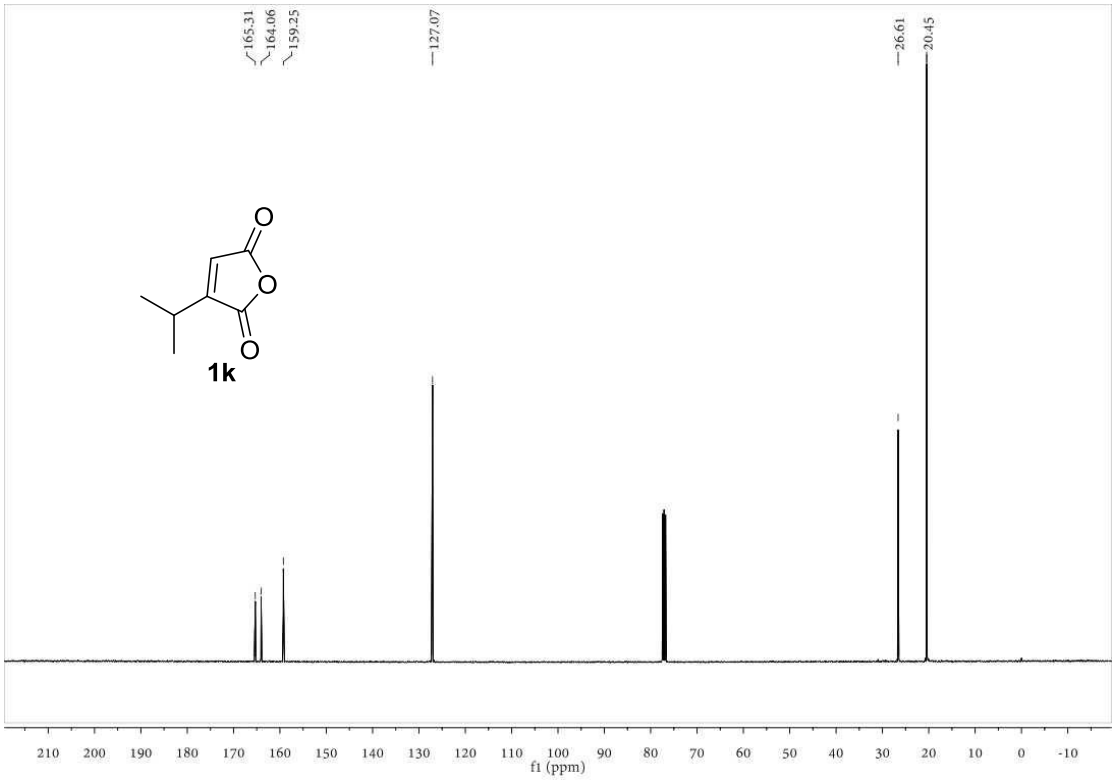
1.02
3.09
3.07
f1 (ppm)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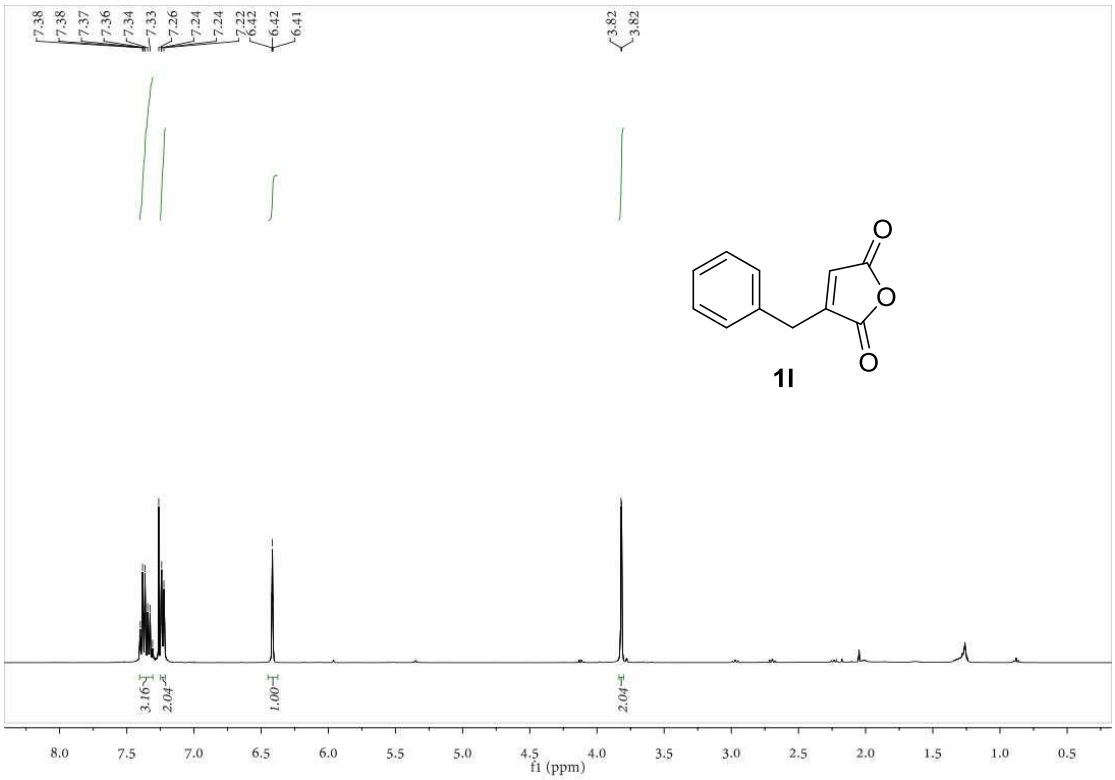
1k
f1 (ppm)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6.42
6.41
3.82
3.82
O
O
O
1l
3.16
2.04
1.00
2.04
8.0
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7.0
6.5
6.0
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3.0
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2.0
1.5
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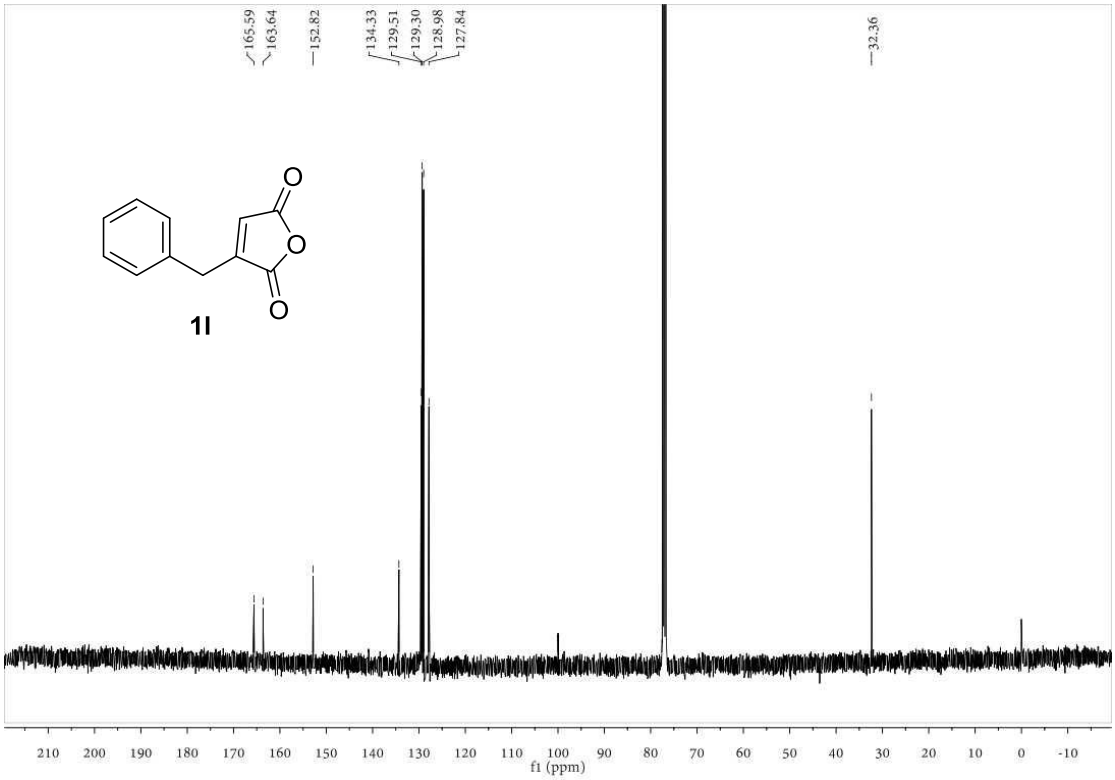
0.5
f1 (ppm)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152.82
134.33
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128.98
127.84
32.36
O
O
O
1l
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200
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180
170
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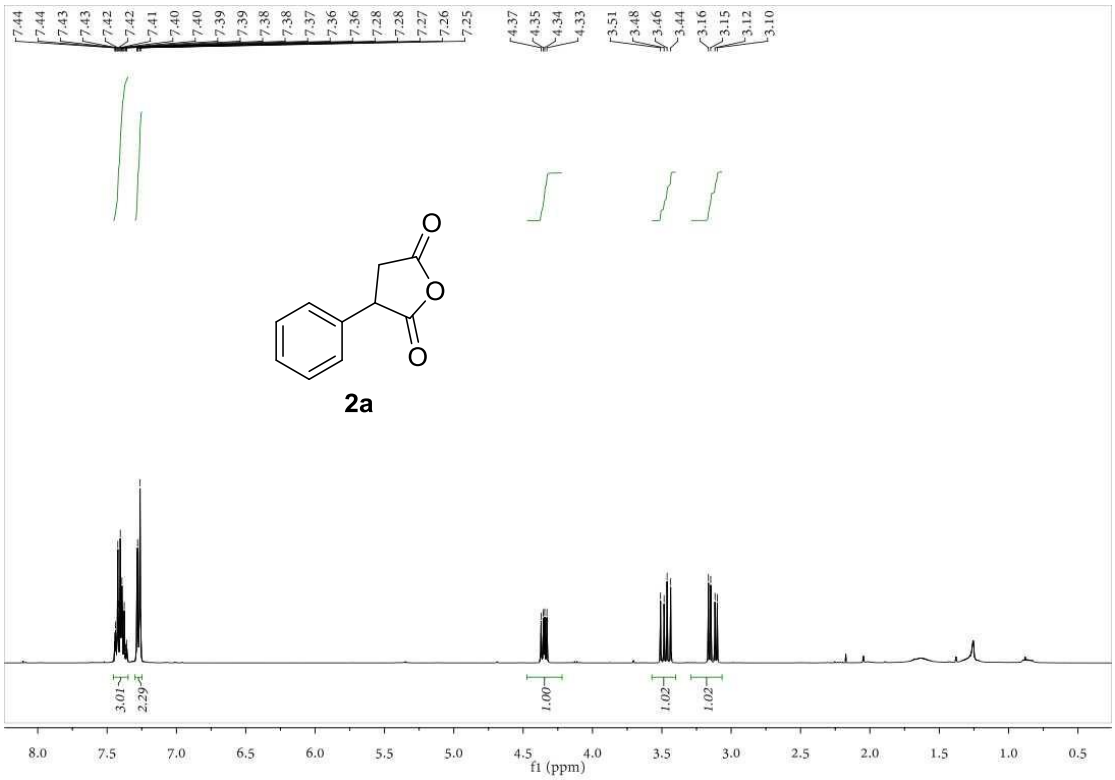
f1 (ppm)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7.37
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7.28
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7.27
7.26
7.25
4.37
4.35
4.34
4.33
3.51
3.48
3.46
3.44
3.16
3.15
3.12
3.10
O
O
O
2a
3.01
2.29
1.00
1.02
1.02
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7.5
7.0
6.5
6.0
5.5
5.0
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3.0
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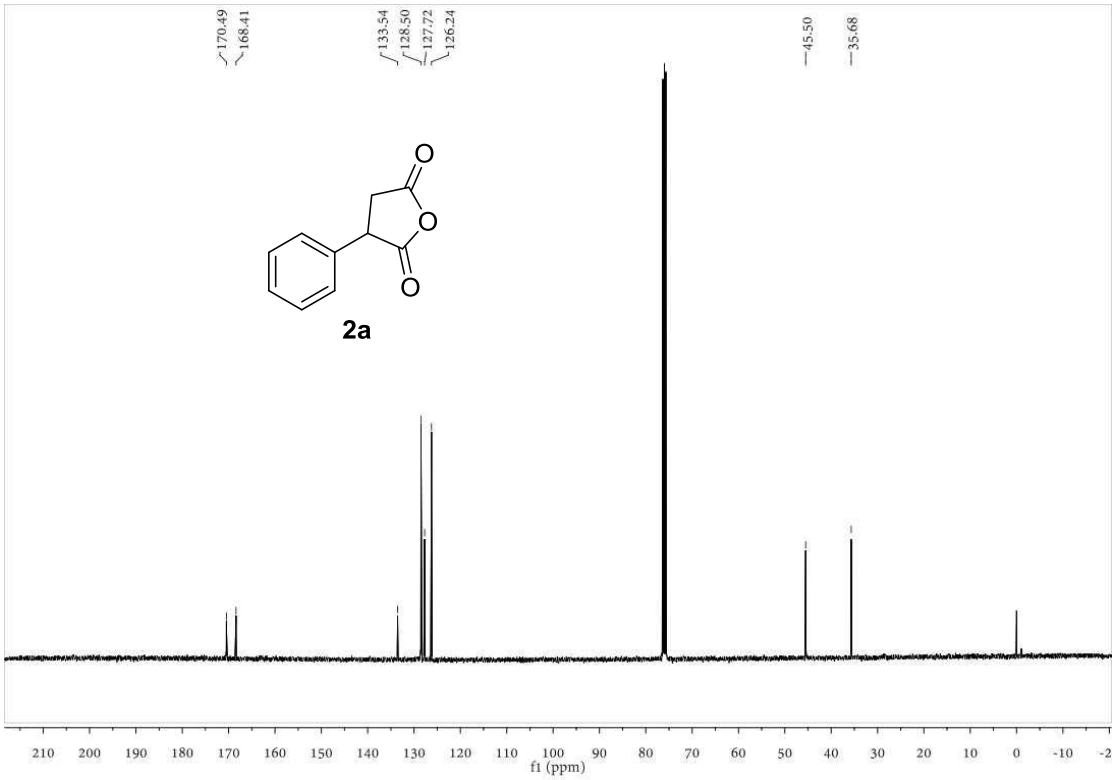
2.0
1.5
1.0
0.5
f1 (ppm)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126.24
45.50
35.68
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O
O
2a
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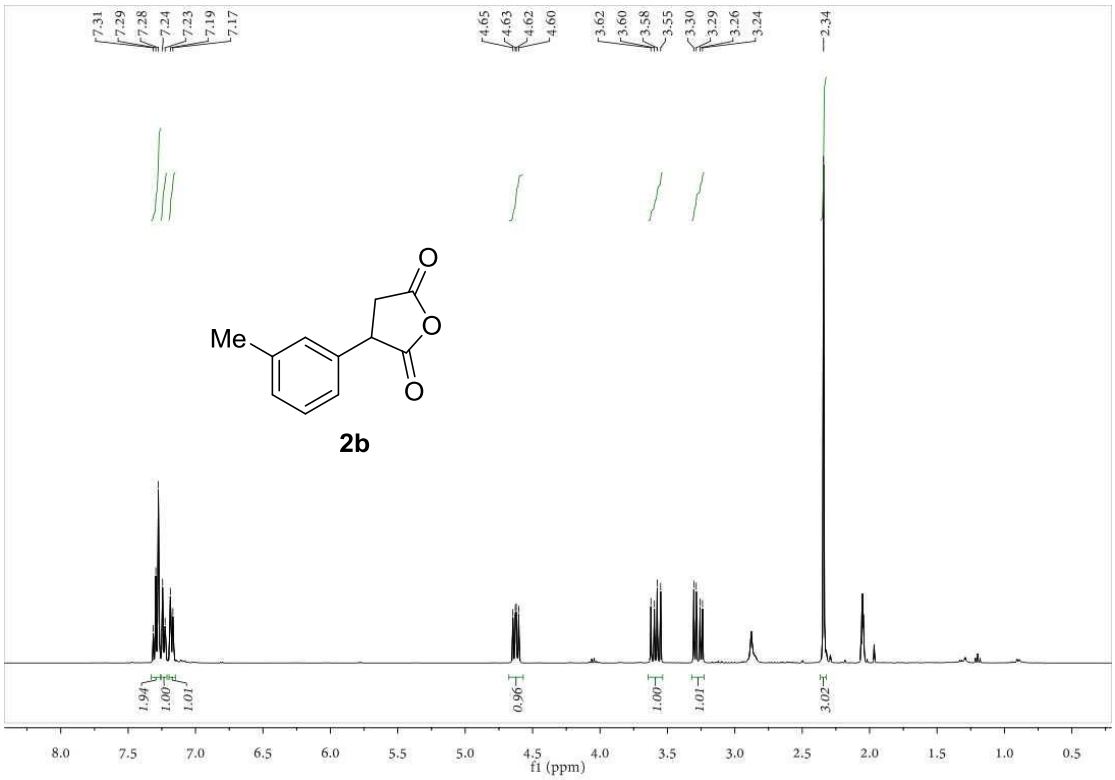
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-2
f1 (ppm)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4.65
4.63
4.62
4.60
3.62
3.60
3.58
3.55
3.30
3.29
3.26
3.24
2.34
Me
O
O
O
2b
1.94
1.00
1.01
0.96
1.00
1.01
3.02
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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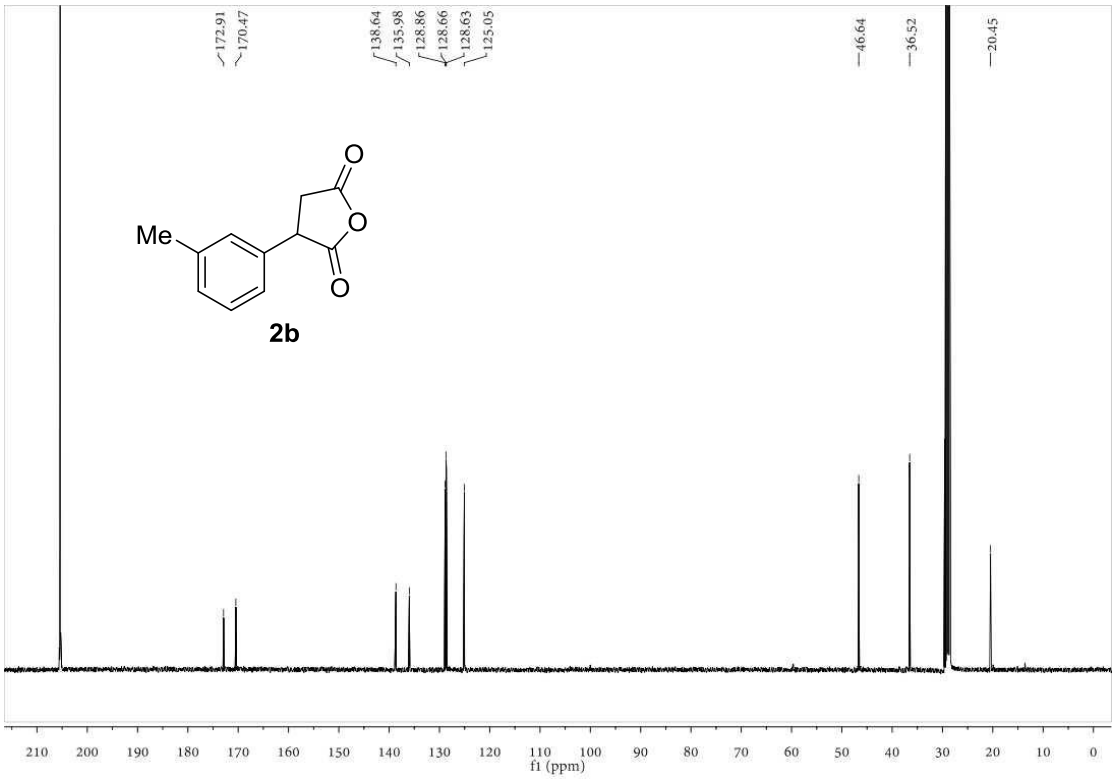
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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128.66
128.63
125.05
46.64
36.52
20.45
Me
O
O
O
2b
210
200
190
180
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130
120
110
100
90
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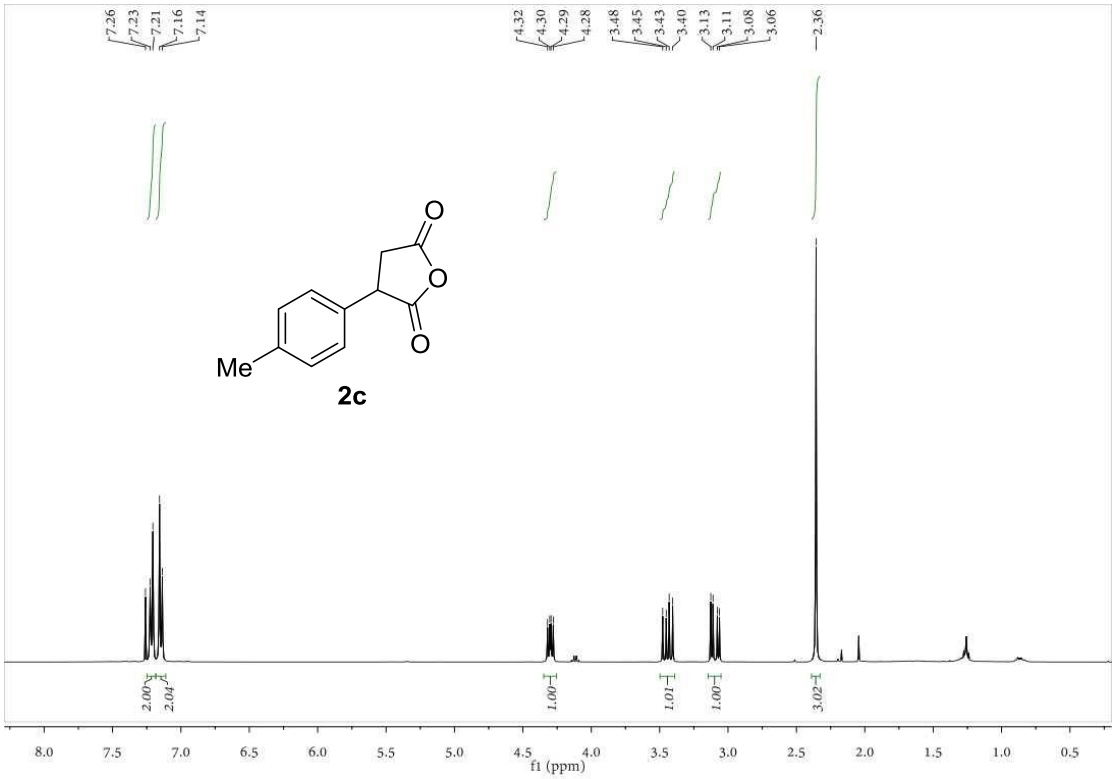
50
40
30
20
10
0
f1 (ppm)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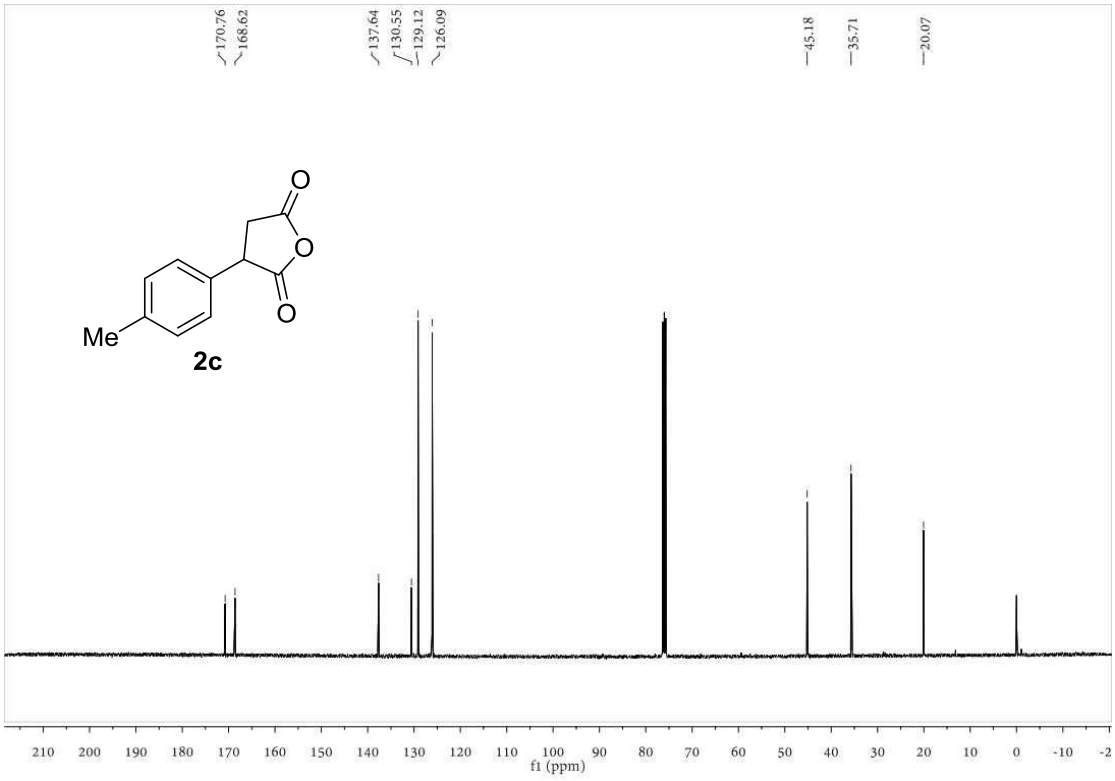
4.28
3.48
3.45
3.43
3.40
3.13
3.11
3.08
3.06
2.36
O
O
O
Me
2c
2.00
2.04
1.00
1.01
1.00
3.02
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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129.12
126.09
45.18
35.71
20.07
O
O
O
Me
2c
210
200
190
180
170
160
150
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100
90
80
70
60
50
40
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20
10
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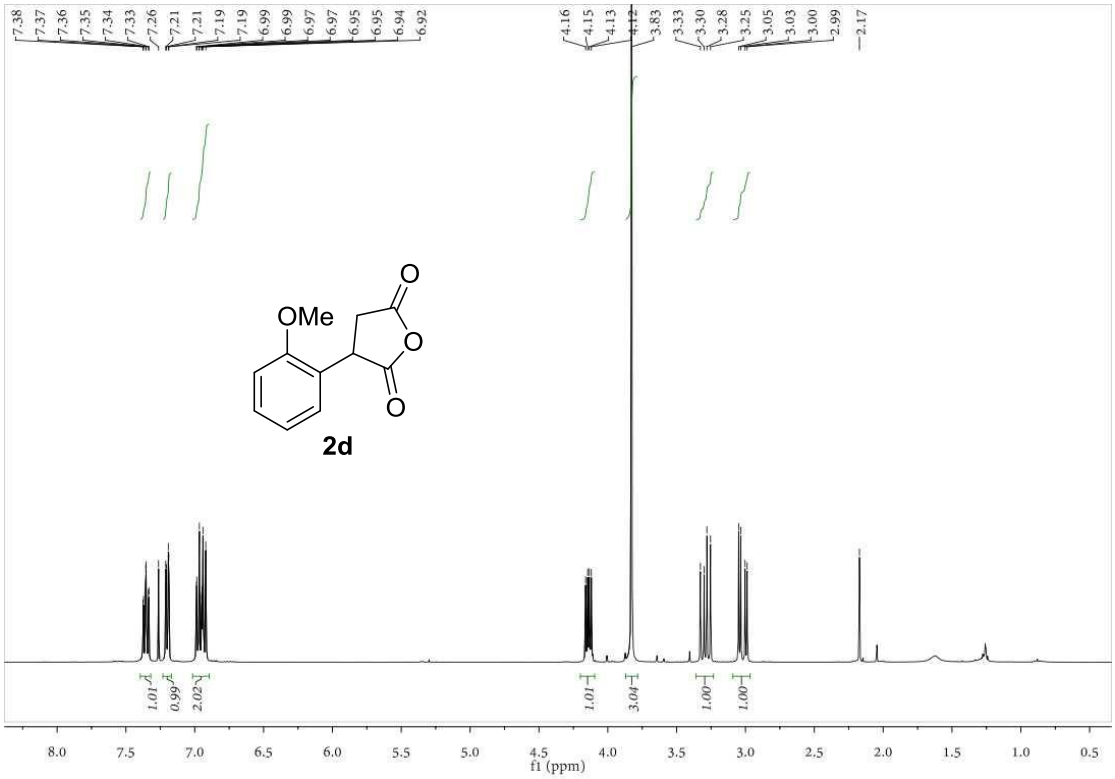
-10
-2
f1 (ppm)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7.21
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7.19
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6.99
6.97
6.97
6.95
6.95
6.94
6.92
4.16
4.15
4.13
4.12
3.83
3.33
3.30
3.28
3.25
3.05
3.03
3.00
2.99
2.17
OMe
O
O
O
2d
1.01
0.99
2.02
1.01
3.04
1.00
1.00
8.0
7.5
7.0
6.5
6.0
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3.0
2.5
2.0
1.5
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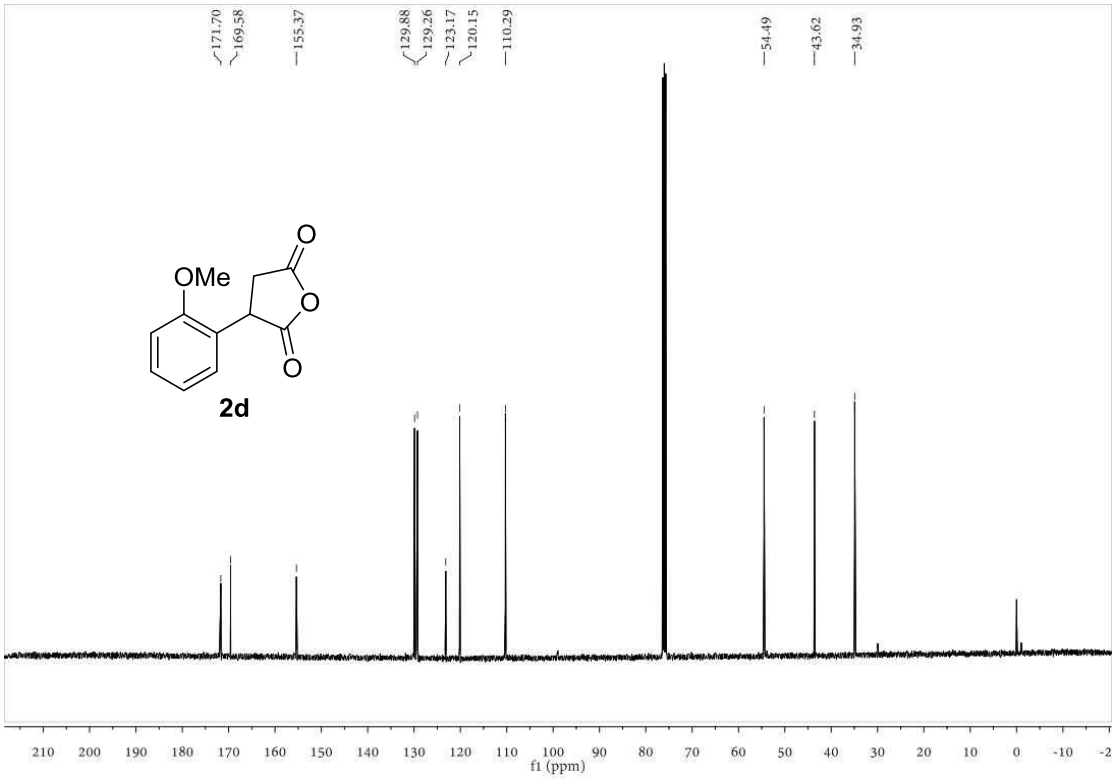
f1 (ppm)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129.88
129.26
123.17
120.15
110.29
54.49
43.62
34.93
OMe
O
O
O
2d
210
200
190
180
170
160
150
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130
120
110
100
90
80
70
60
50
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20
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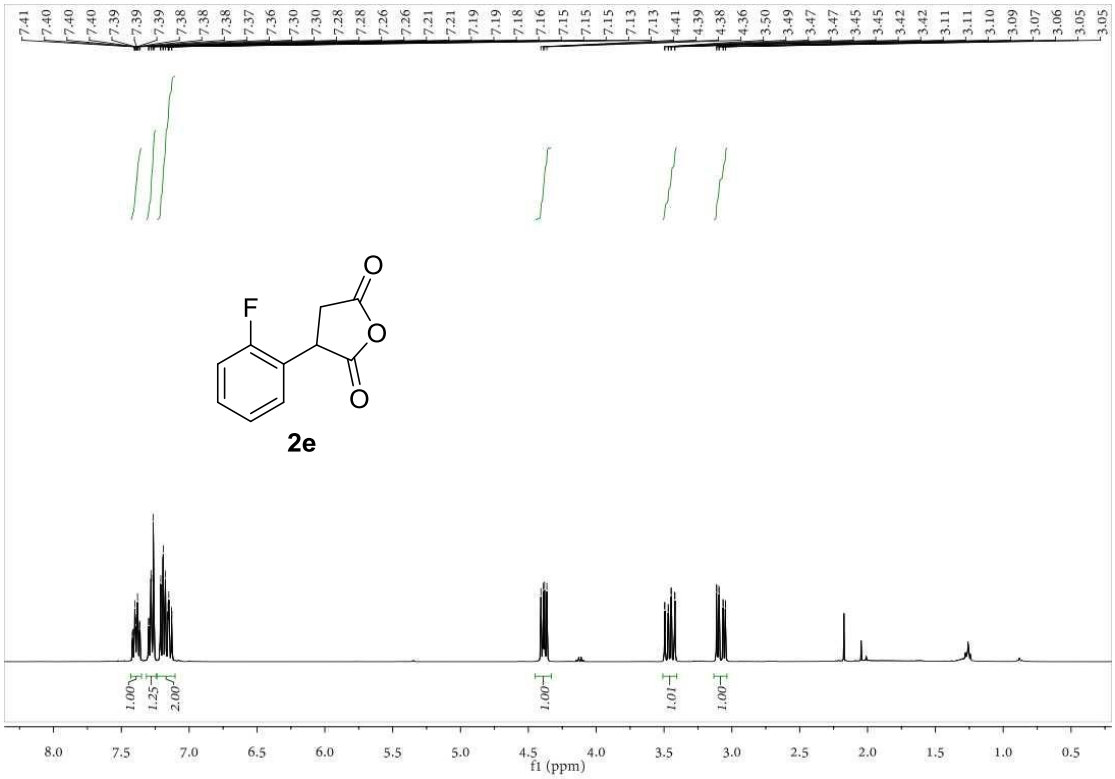
0
-10
-2
f1 (ppm)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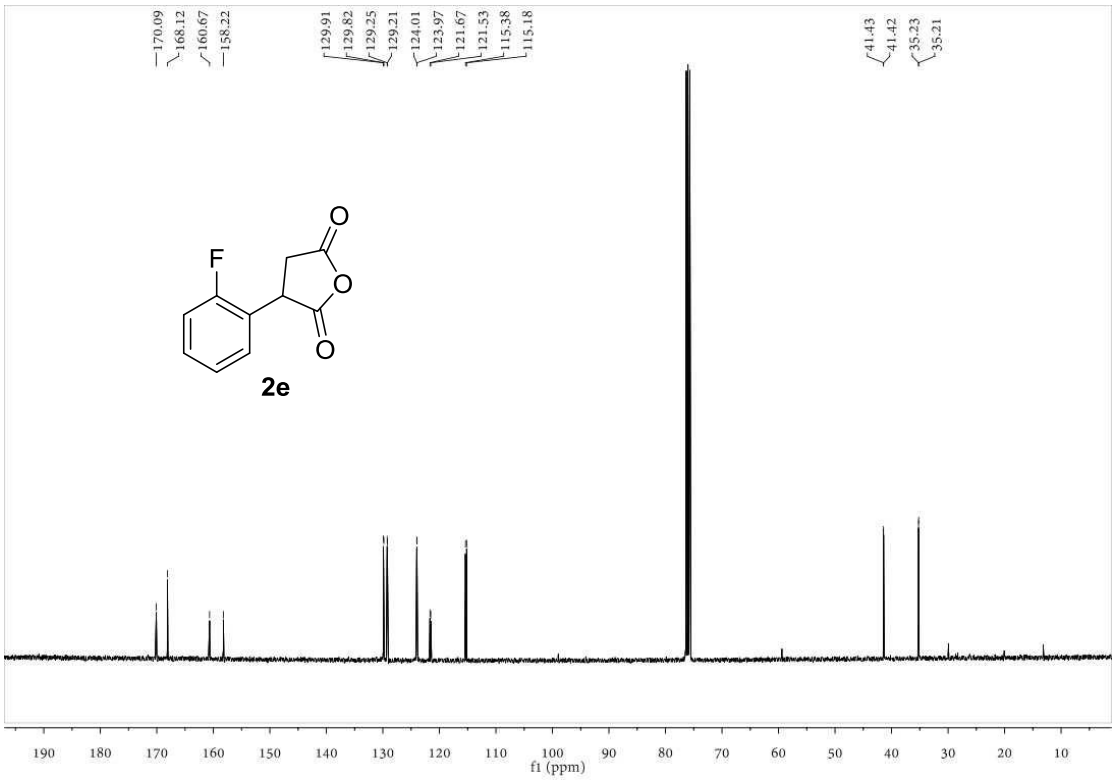
F
O
O
O
2e
1.00 1.25 2.00 1.00 1.01 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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F
O
O
O
2e
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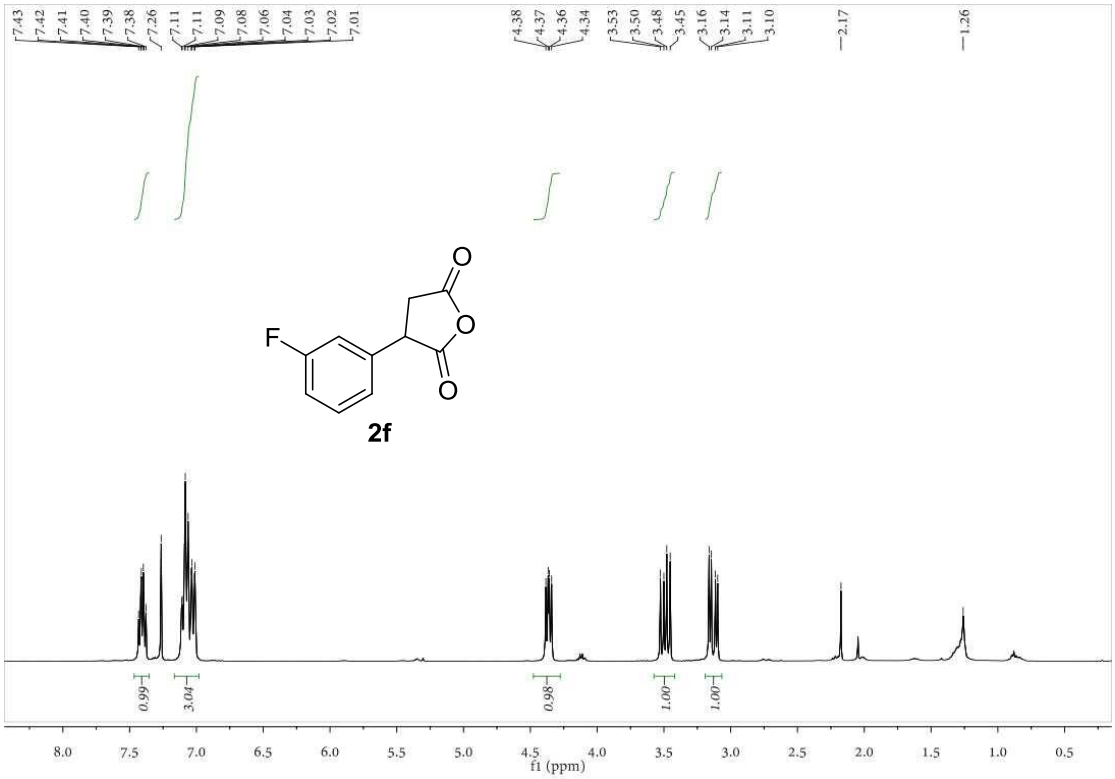
f1 (ppm)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7.04
7.03
7.02
7.01
4.38
4.37
4.36
4.34
3.53
3.50
3.48
3.45
3.16
3.14
3.11
3.10
2.17
1.26
O
O
O
F
O
2f
0.99
3.04
0.98
1.00
1.00
8.0
7.5
7.0
6.5
6.0
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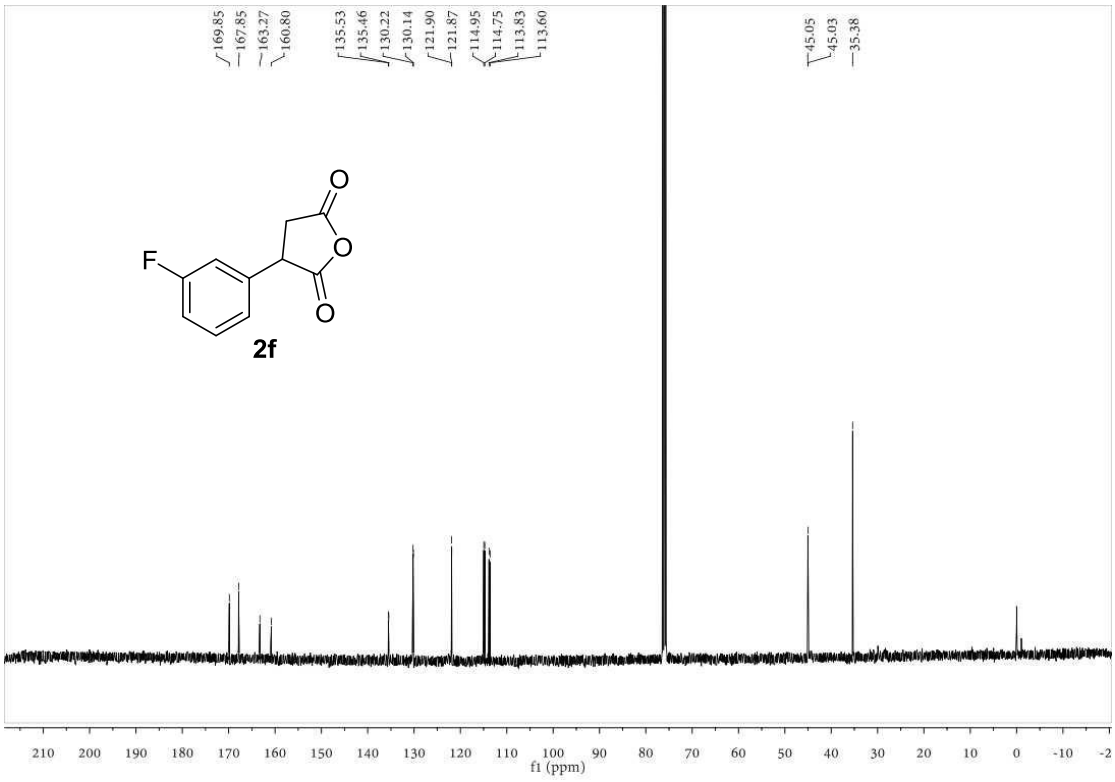
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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130.14
121.90
121.87
114.95
114.75
113.83
113.60
45.05
45.03
35.38
O
O
O
F
O
2f
210
200
190
180
170
160
150
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130
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100
90
80
70
60
50
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30
20
10
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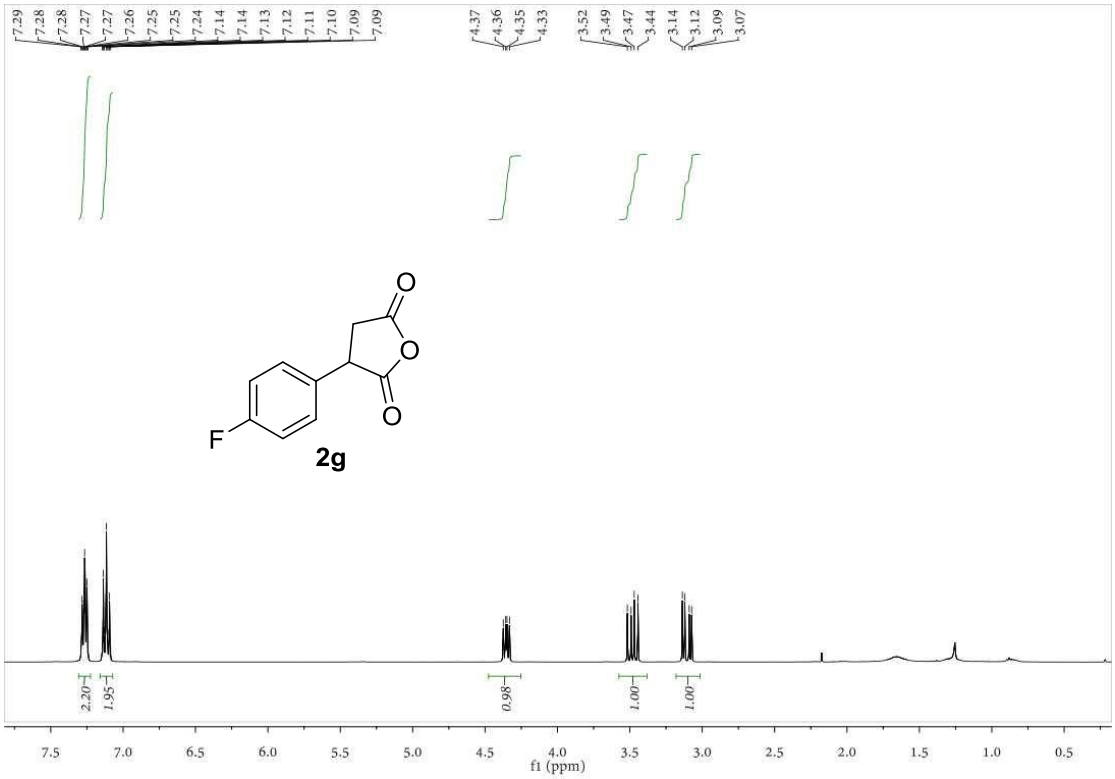
-10
-2
f1 (ppm)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7.12
7.11
7.10
7.09
7.09
4.37
4.36
4.35
4.33
3.52
3.49
3.47
3.44
3.14
3.12
3.09
3.07
O
O
O
F
2g
2.20
1.95
0.98
1.00
1.00
7.5
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6.5
6.0
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2.5
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1.5
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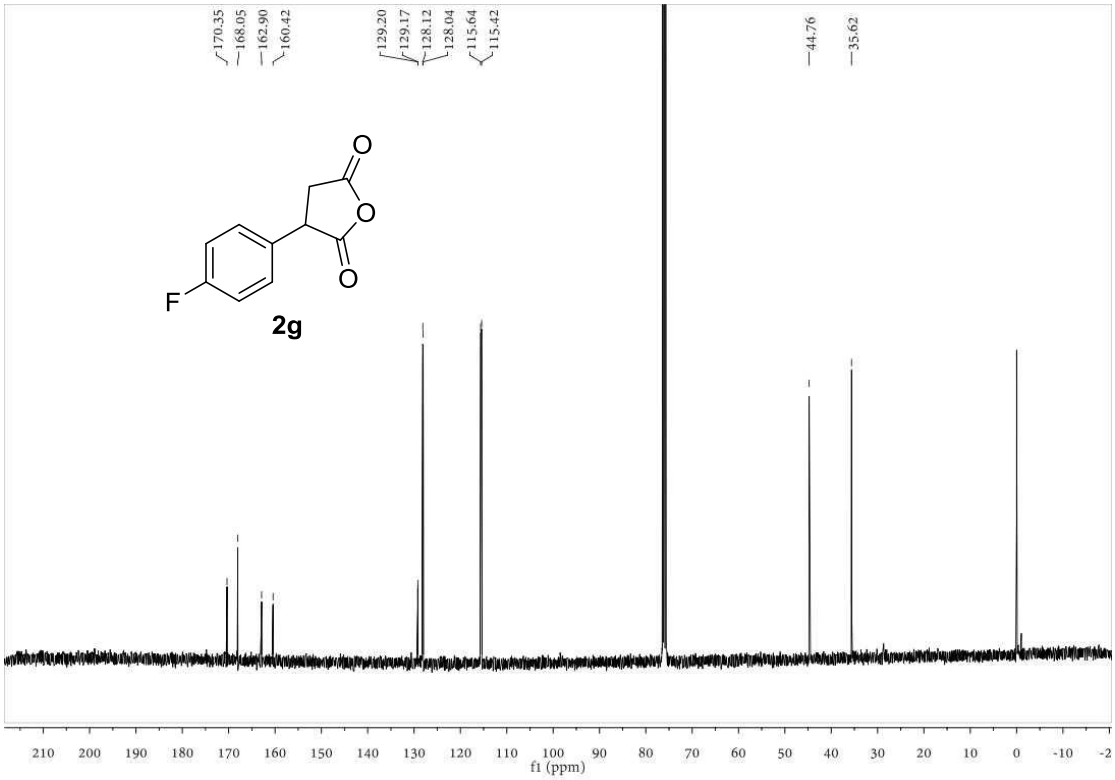
f1 (ppm)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128.04
115.64
115.42
44.76
35.62
O
O
O
F
2g
210
200
190
180
170
160
150
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130
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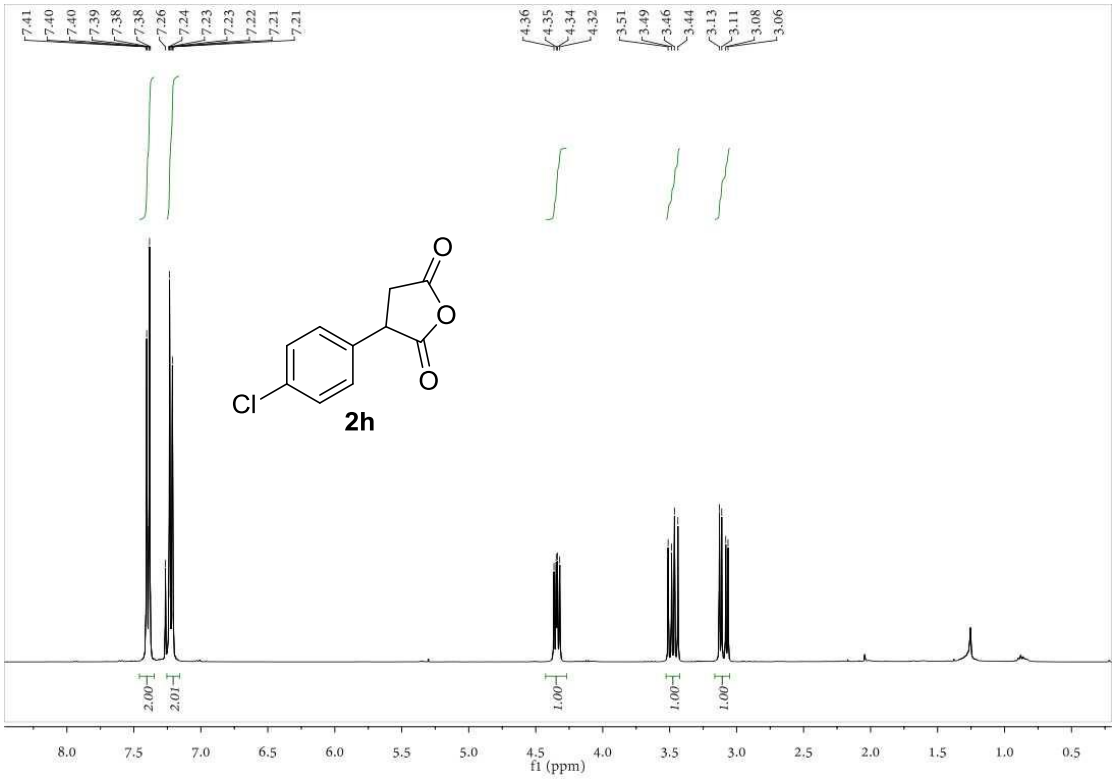
100
90
80
70
60
50
40
30
20
10
0
-10
-2
f1 (ppm)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4.35
4.34
4.32
3.51
3.49
3.46
3.44
3.13
3.11
3.08
3.06
O
O
O
Cl
2h
2.00
2.01
1.00
1.00
1.00
8.0
7.5
7.0
6.5
6.0
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2.5
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1.5
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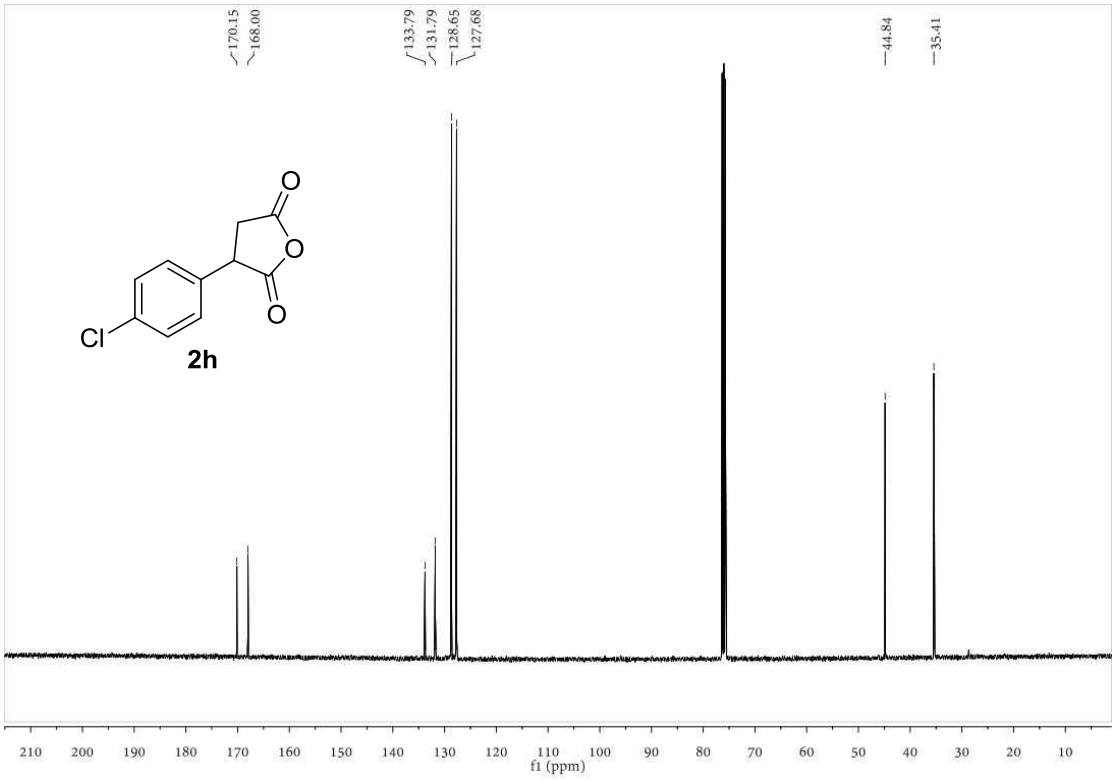
f1 (ppm)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44.84
35.41
O
O
O
Cl
2h
210
200
190
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140
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120
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100
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80
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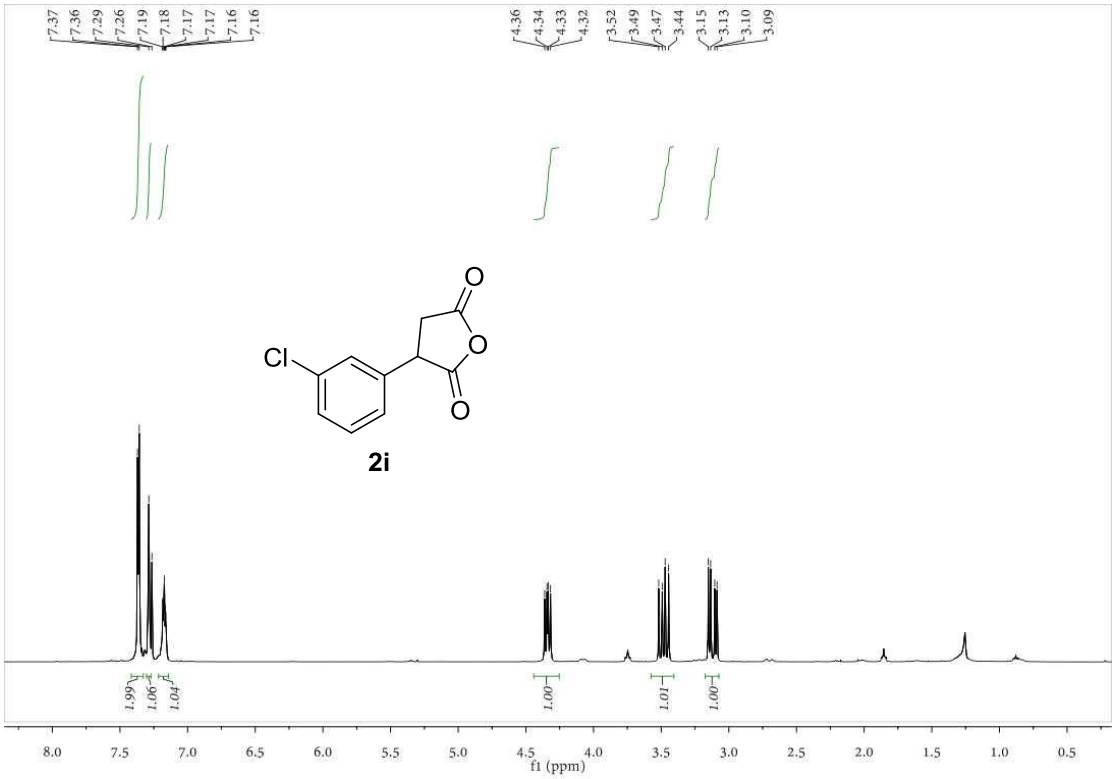
60
50
40
30
20
10
f1 (ppm)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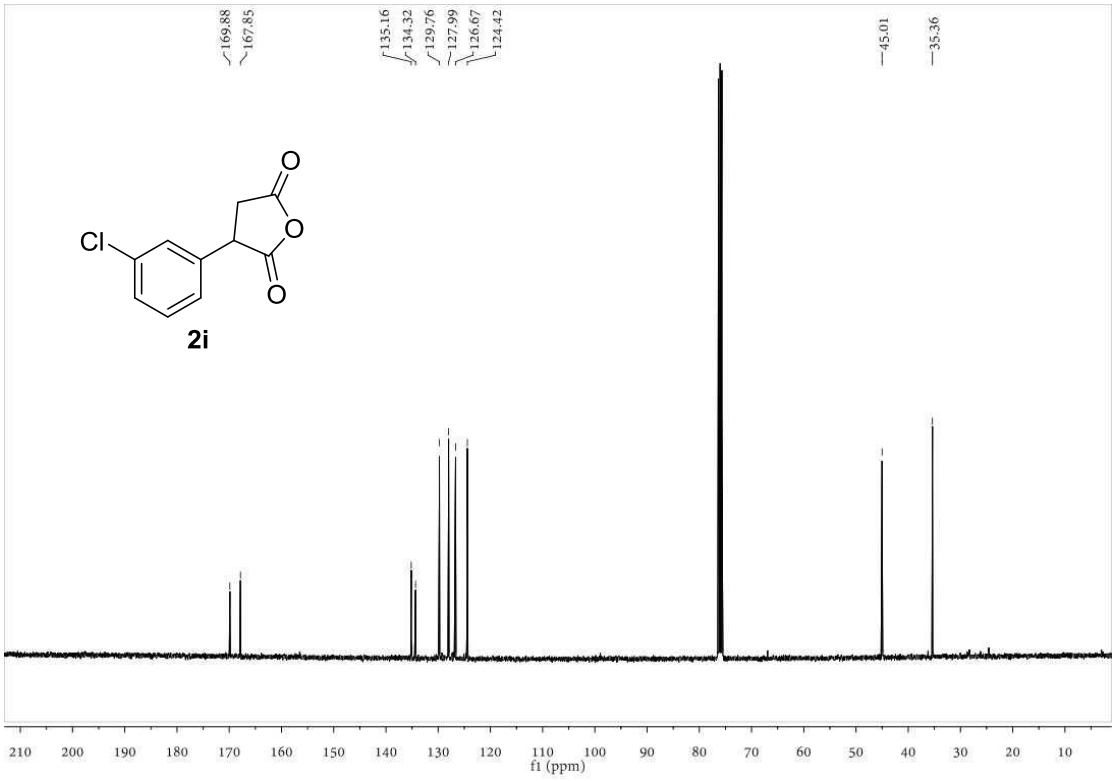
7.16
4.36
4.34
4.33
4.32
3.52
3.49
3.47
3.44
3.15
3.13
3.10
3.09
Cl
O
O
O
2i
1.99
1.06
1.04
1.00
1.01
1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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124.42
45.01
35.36
Cl
O
O
O
2i
210
200
190
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100
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80
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60
50
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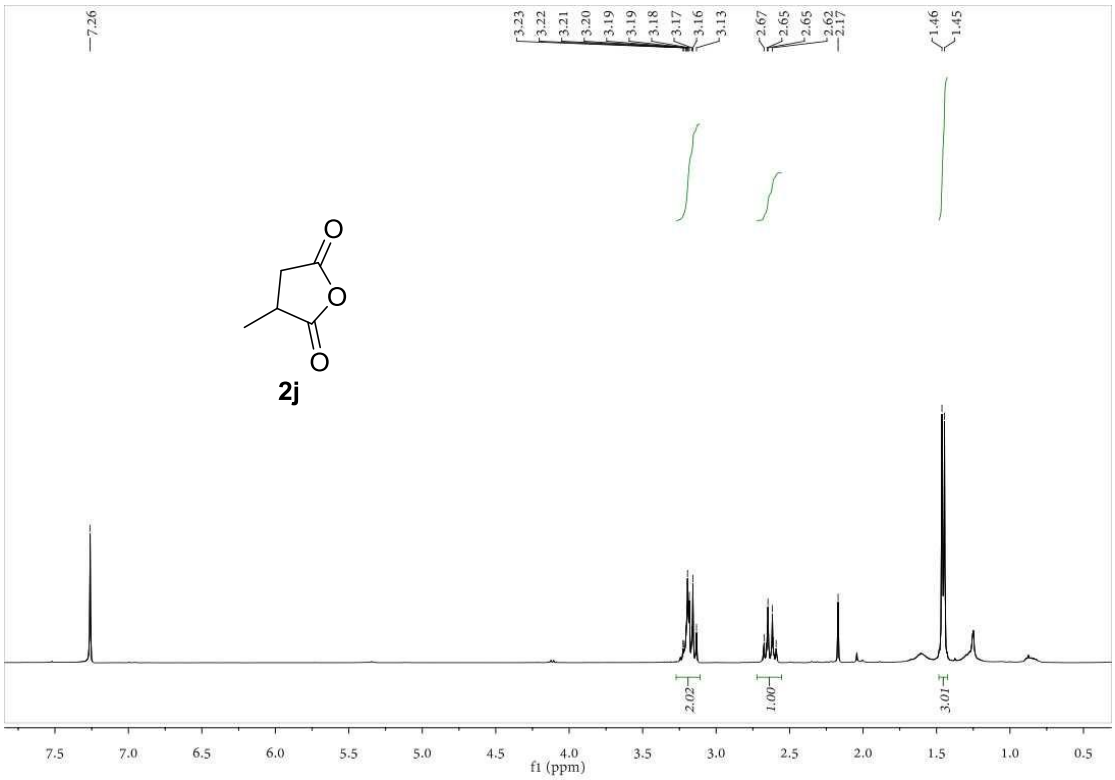
30
20
10
f1 (ppm)


7.26
3.23
3.22
3.21
3.20
3.19
3.19
3.18
3.17
3.16
3.13
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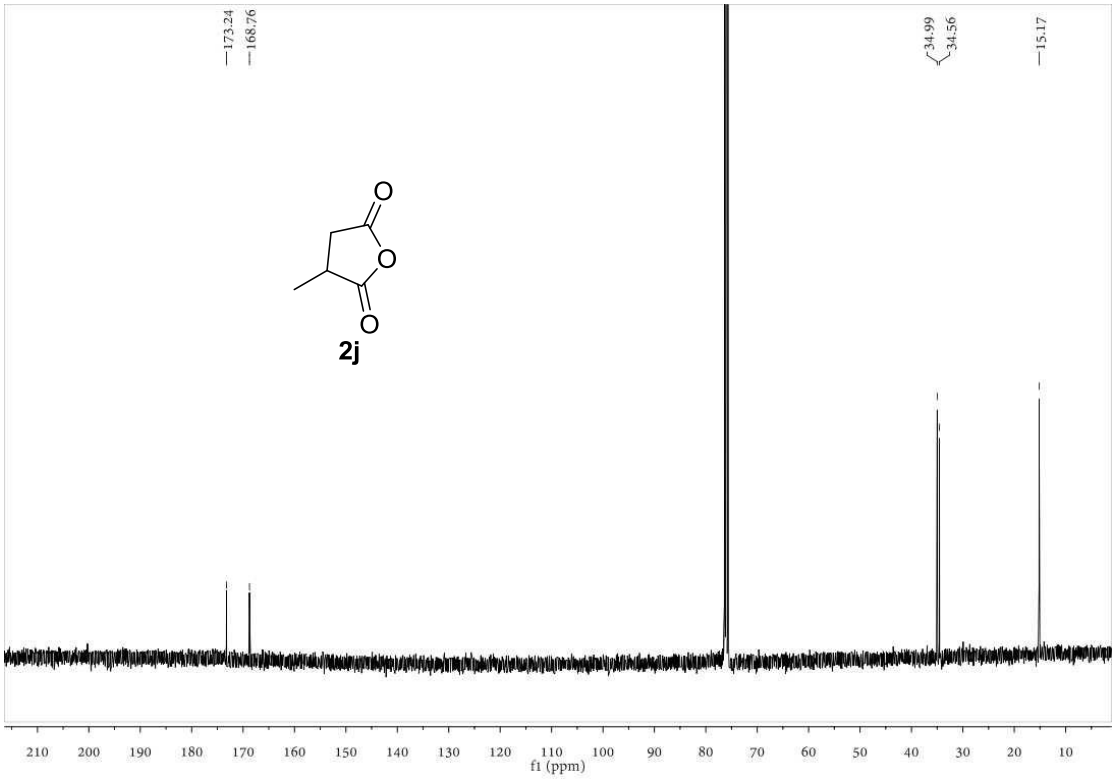
2.65
2.62
2.17
1.46
1.45
2j
2.02
1.00
3.01
f1 (ppm)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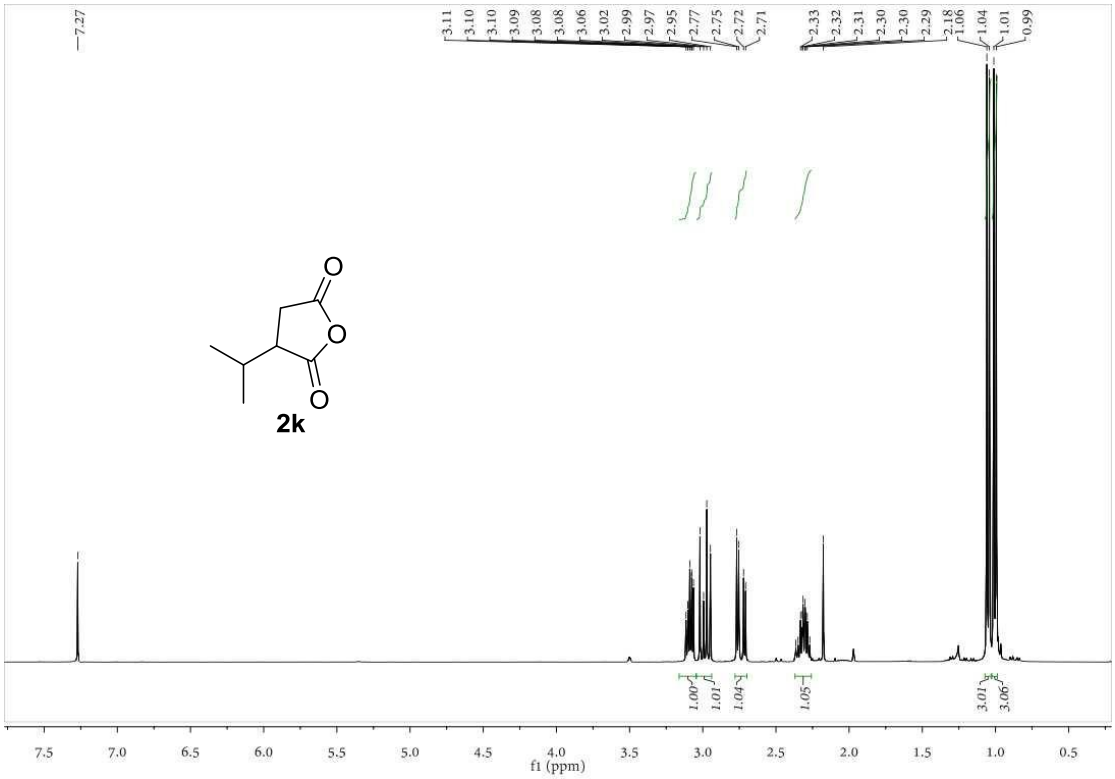
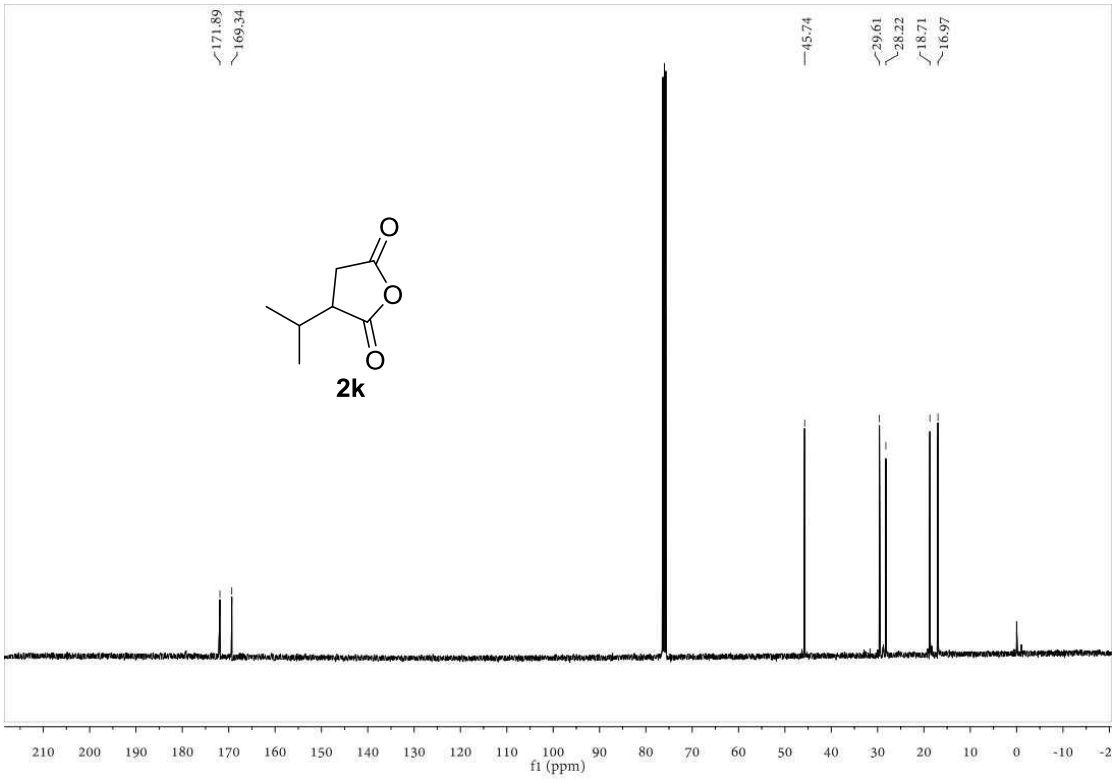
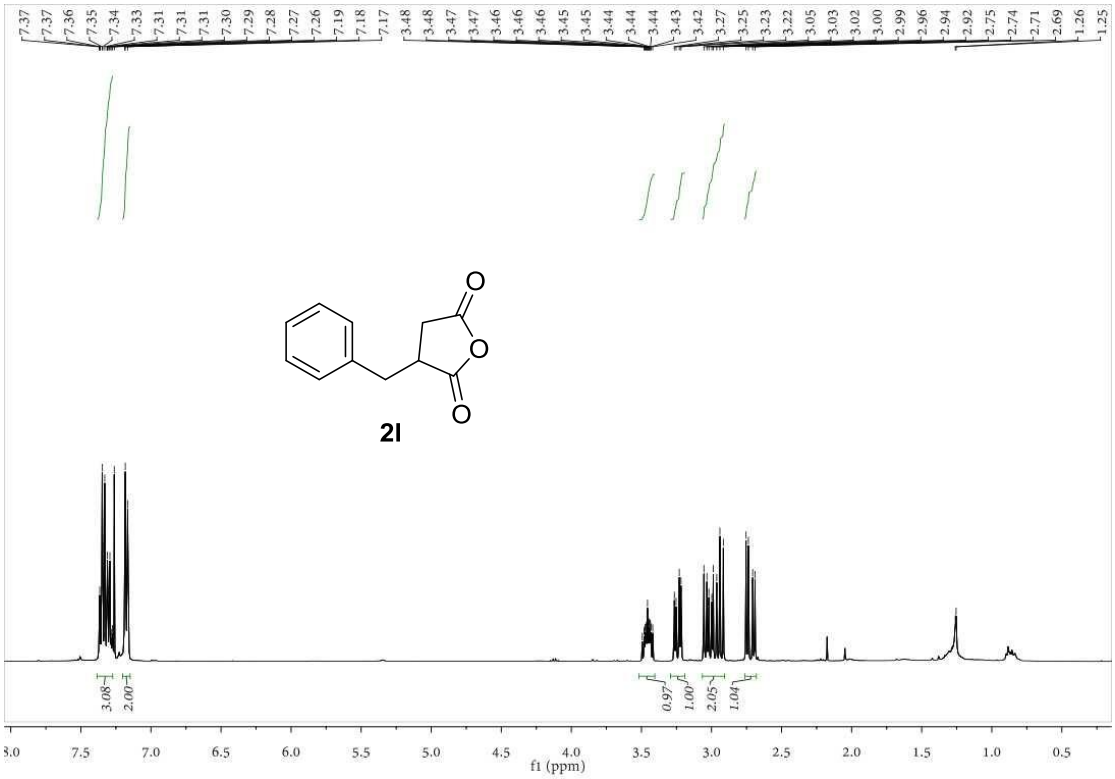
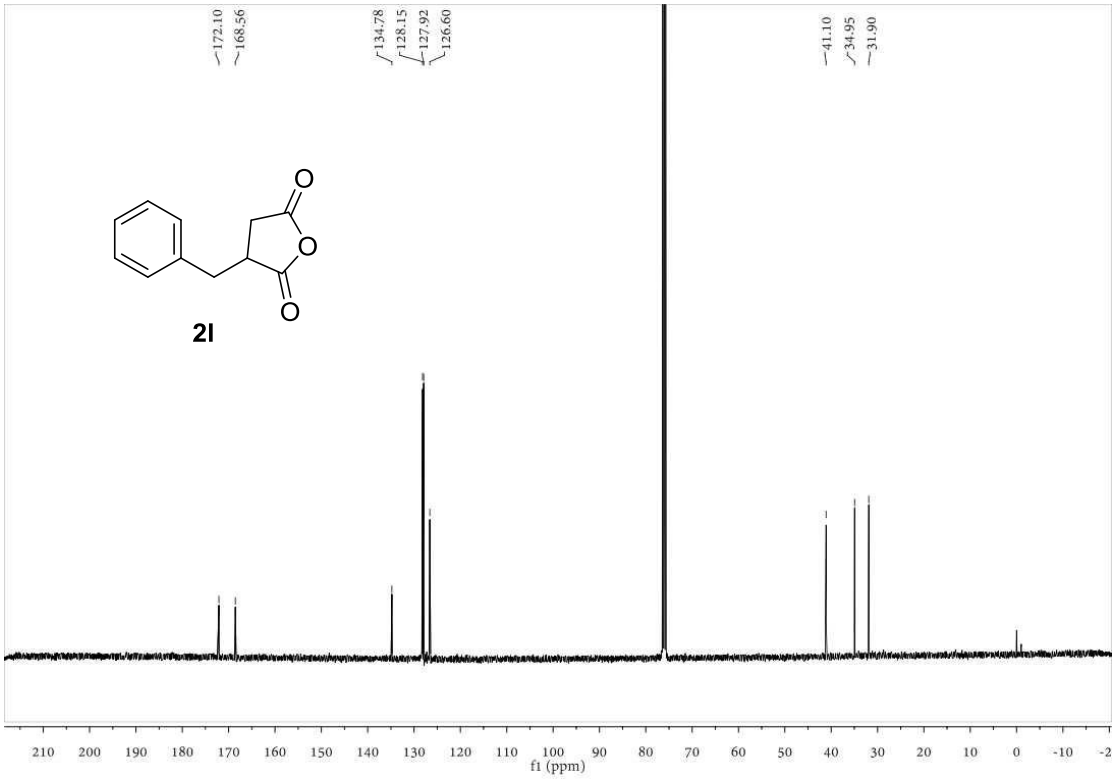
168.76
34.99
34.56
15.17
2j
f1 (ppm)


7.27
3.11
3.10
3.10
3.09
3.08
3.08
3.06
3.02
2.99
2.97
2.95
2.77
2.75
2.72
2.71
2.33
2.32
2.31
2.30
2.30
2.29
2.18
1.06
1.04
1.01
0.99
1.00
1.01
1.04
1.05
3.01
3.06
O
O
O
O
2k
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
f1 (ppm)

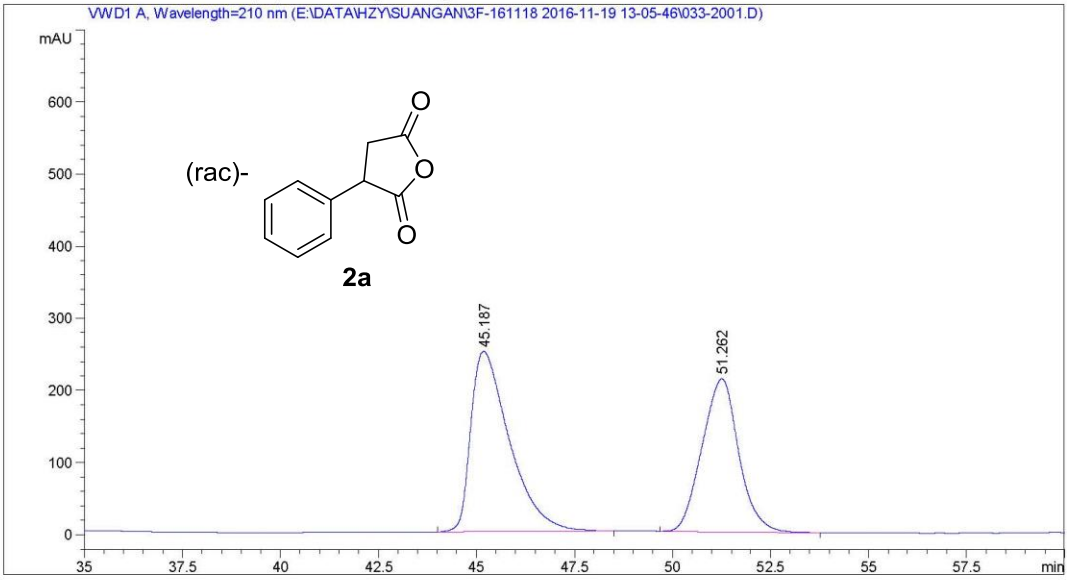

171.89
169.34
45.74
29.61
28.22
18.71
16.97
O
O
O
O
2k
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-2
f1 (ppm)


7.37 7.37 7.36 7.35 7.34 7.33 7.31 7.31 7.31 7.30 7.29 7.28 7.27 7.26 7.19 7.18 7.17
3.48 3.48 3.47 3.47 3.46 3.46 3.46 3.45 3.45 3.44 3.44 3.44 3.43 3.42 3.27 3.25 3.23 3.22 3.05 3.03 3.02 3.00 2.99 2.96 2.94 2.92 2.75 2.74 2.71 2.69 1.26 1.25
O
O
O
2l
3.08
2.00
0.97
1.00
2.05
1.04
f1 (ppm)


172.10
168.56
134.78
128.15
127.92
126.60
41.10
34.95
31.90
O
O
O
2l
f1 (ppm)

VI. HPLC Spectra

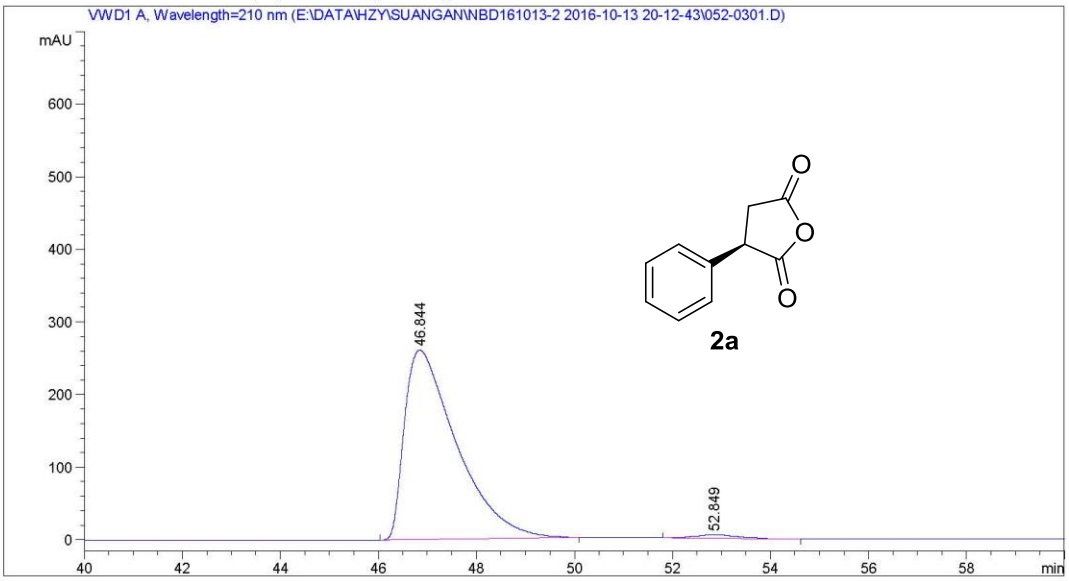
```
Data File E:\DATA\HZY\SUANGAN\3F-161118 2016-11-19 13-05-46\033-2001.D
Sample Name: H-STAND-RAC-2

    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :  20
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 33
    Injection Date  : 11/20/2016 3:32:00 AM               Inj :   1
                                                   Inj Volume : 2.000 µl
    Acq. Method     : E:\DATA\HZY\SUANGAN\3F-161118 2016-11-19 13-05-46\VWD-AD(1-6)-98-2-1ML-2UL-
                      210NM-70MIN.M
    Last changed    : 11/19/2016 9:29:56 PM by SYSTEM
    Analysis Method : E:\DATA\HZY\SUANGAN\3F-161118 2016-11-19 13-05-46\VWD-AD(1-6)-98-2-1ML-2UL-
                      210NM-70MIN.M (Sequence Method)
    Last changed    : 11/20/2016 8:42:59 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  45.187 BB    1.0309 1.70889e4   249.87816  55.3176
       2  51.262 BB    1.0065 1.38034e4   212.30431  44.6824

    Totals :                  3.08924e4   462.18246
```

Data File E:\DATA\HZY\SUANGAN\NBD161013-2 2016-10-13 20-12-43\052-0301.D
Sample Name: nbd1013-2

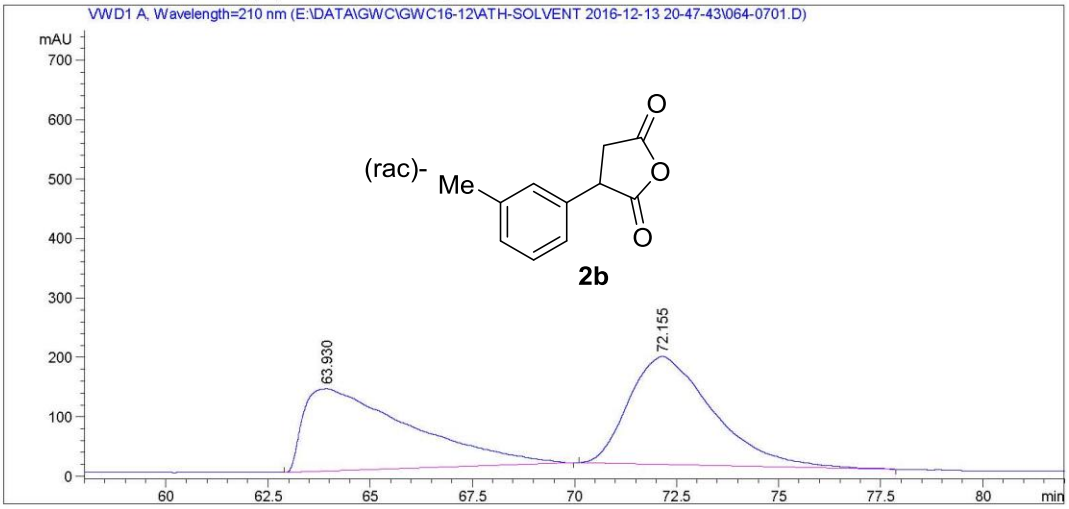
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   3
Acq. Instrument : 1260HPLC-VWD                    Location : Vial 52
Injection Date  : 10/13/2016 9:30:10 PM               Inj :   1
                                                  Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\HZY\SUANGAN\NBD161013-2 2016-10-13 20-12-43\VWD-AD(1-6)-98-2-1ML-
                  2UL-210NM-70MIN.M
Last changed    : 10/13/2016 8:12:44 PM by SYSTEM
Analysis Method : E:\DATA\HZY\SUANGAN\NBD161013-2 2016-10-13 20-12-43\VWD-AD(1-6)-98-2-1ML-
                  2UL-210NM-70MIN.M (Sequence Method)
Last changed    : 11/19/2016 7:17:50 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  46.844 BB    1.1194 1.92409e4   260.90070  98.4577
   2  52.849 BB    0.8632  301.40305    4.70622   1.5423

Totals :                  1.95423e4   265.60691
```

Data File E:\DATA\GWC\GWC16-12\ATH-SOLVENT 2016-12-13 20-47-43\064-0701.D
Sample Name: 3ME-RAC

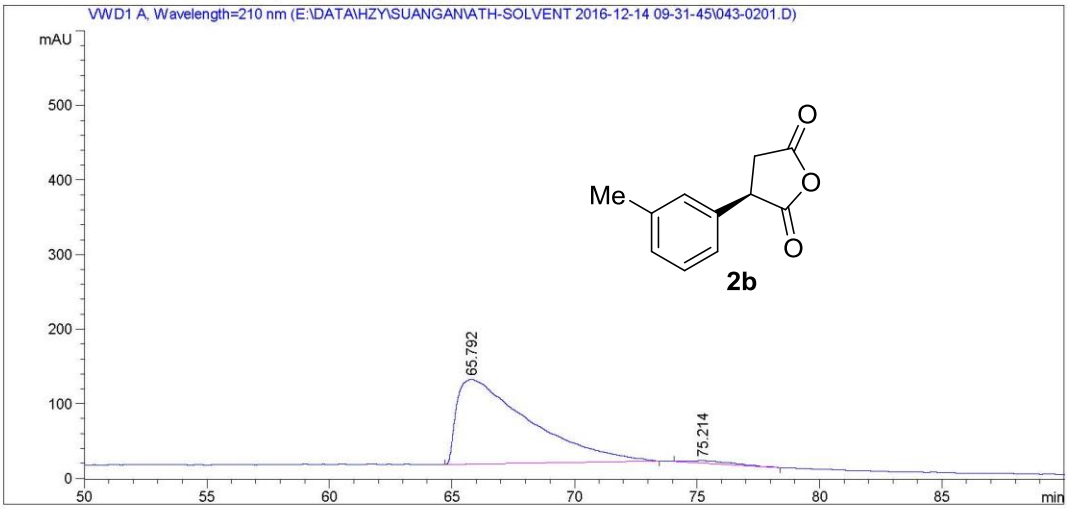
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   7
Acq. Instrument : 1260HPLC-VWD                     Location : Vial 64
Injection Date  : 12/13/2016 11:40:18 PM               Inj :   1
                                                  Inj Volume : 10.000 µl
Acq. Method     : E:\DATA\GWC\GWC16-12\ATH-SOLVENT 2016-12-13 20-47-43\VWD-AD(1-6)-97-3-1ML-
                  10UL-85MIN.M
Last changed    : 12/13/2016 10:01:33 PM by SYSTEM
Analysis Method : E:\DATA\GWC\GWC16-12\ATH-SOLVENT 2016-12-13 20-47-43\VWD-AD(1-6)-97-3-1ML-
                  10UL-85MIN.M (Sequence Method)
Last changed    : 12/14/2016 9:15:55 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  63.930 BB    2.3854 2.51050e4   138.58795  49.3451
   2  72.155 BB    1.8682 2.57714e4   181.43526  50.6549

Totals :                  5.08764e4   320.02321


==========================================================================
                          *** End of Report ***
```

Data File E:\DATA\HZY\SUANGAN\ATH-SOLVENT 2016-12-14 09-31-45\043-0201.D
Sample Name: 3ME

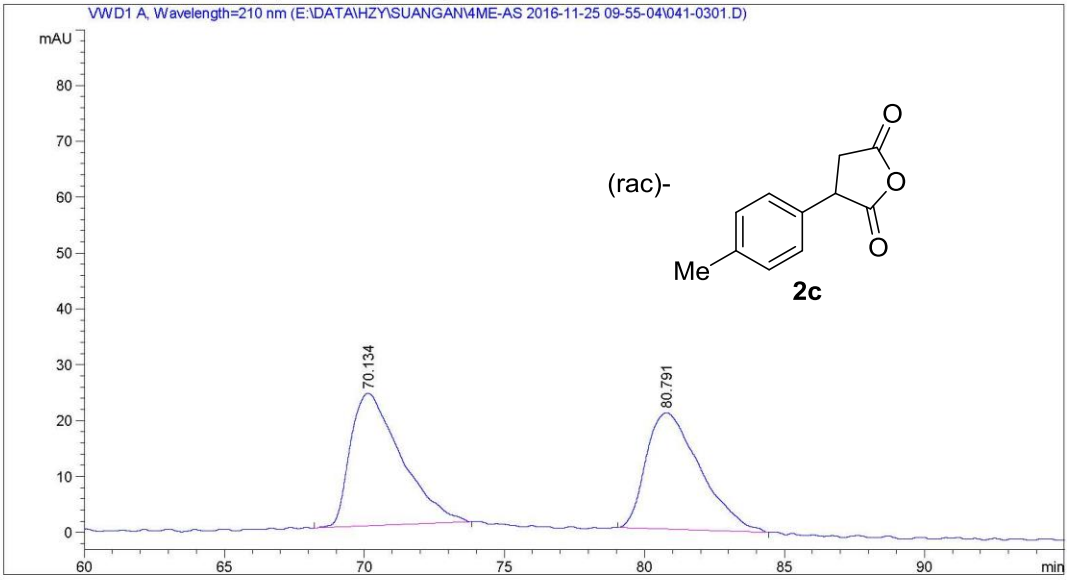
```
=====================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260HPLC-VWD                   Location : Vial 43
Injection Date  : 12/14/2016 9:42:24 AM               Inj :   1
                                                Inj Volume : 10.000 µl
Acq. Method     : E:\DATA\HZY\SUANGAN\ATH-SOLVENT 2016-12-14 09-31-45\VWD-AD(1-6)-97-3-1ML-
                  10UL-85MIN.M
Last changed    : 12/14/2016 11:03:58 AM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\HZY\SUANGAN\ATH-SOLVENT 2016-12-14 09-31-45\VWD-AD(1-6)-97-3-1ML-
                  10UL-85MIN.M (Sequence Method)
Last changed    : 12/14/2016 11:14:50 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  65.792 BB    2.6866 2.33380e4   113.46952  98.0013
   2  75.214 BB    1.6377  475.97626    3.49540   1.9987

Totals :                  2.38140e4   116.96493

=====================================================================
```

Data File E:\DATA\HZY\SUANGAN\4ME-AS 2016-11-25 09-55-04\041-0301.D
Sample Name: 4ME-RAC

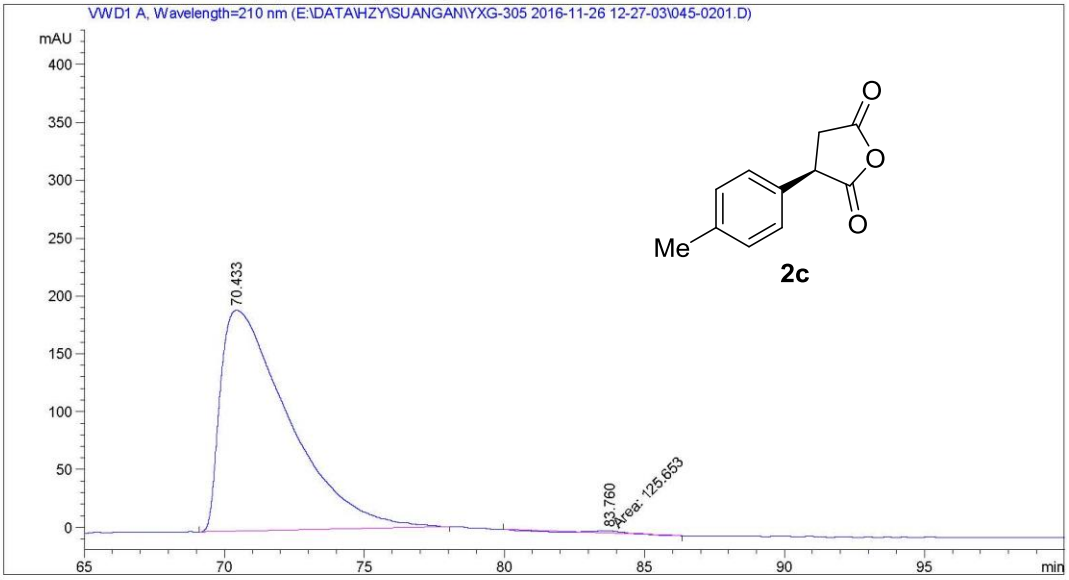
```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260HPLC-VWD                   Location : Vial 41
Injection Date  : 11/25/2016 11:51:34 AM             Inj :   1
                                                Inj Volume : 10.000 µl
Acq. Method     : E:\DATA\HZY\SUANGAN\4ME-AS 2016-11-25 09-55-04\VWD-AS(1-2)-95-5-1-10UL-
                  210NM-85MIN.M
Last changed    : 11/25/2016 11:36:33 AM by SYSTEM
Analysis Method : E:\DATA\HZY\SUANGAN\4ME-AS 2016-11-25 09-55-04\VWD-AS(1-2)-95-5-1-10UL-
                  210NM-85MIN.M (Sequence Method)
Last changed    : 11/25/2016 4:04:58 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  70.134 BB    1.7711 2983.73730   23.72738  51.6496
   2  80.791 BB    1.8465 2793.14331   20.85506  48.3504

Totals :                  5776.88062   44.58245
```

Data File E:\DATA\HZY\SUANGAN\YXG-305 2016-11-26 12-27-03\045-0201.D
Sample Name: 4ME-EE

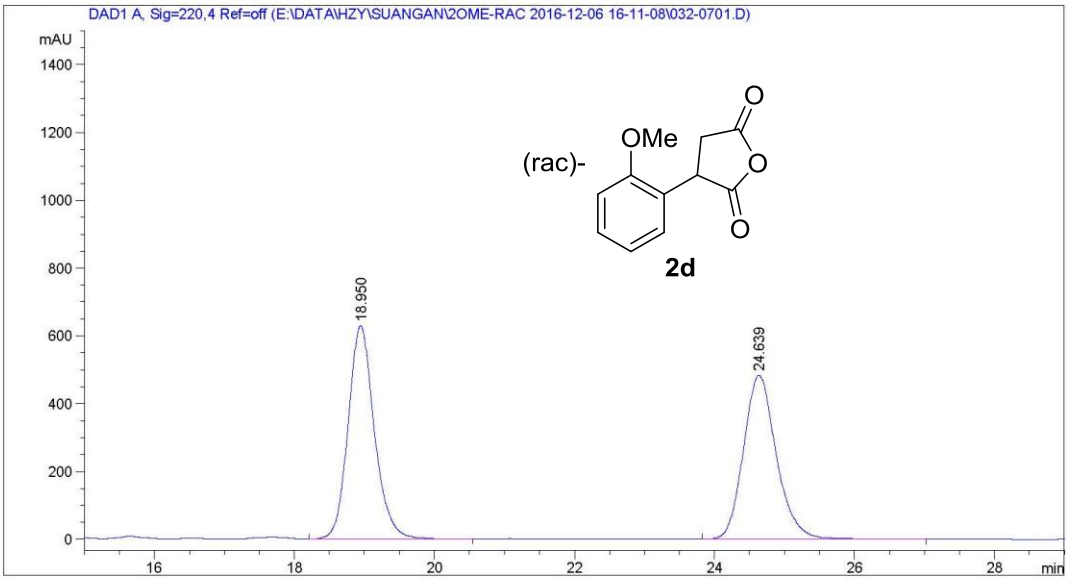
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   2
Acq. Instrument : 1260HPLC-VWD                    Location : Vial 45
Injection Date  : 11/26/2016 12:50:01 PM              Inj :   1
                                                  Inj Volume : 10.000 µl
Acq. Method     : E:\DATA\HZY\SUANGAN\YXG-305 2016-11-26 12-27-03\VWD-AS(1-2)-95-5-1-10UL-
                  210NM-100MIN.M
Last changed    : 11/26/2016 12:27:05 PM by SYSTEM
Analysis Method : E:\DATA\HZY\SUANGAN\YXG-305 2016-11-26 12-27-03\VWD-AS(1-2)-95-5-1-10UL-
                  210NM-100MIN.M (Sequence Method)
Last changed    : 1/13/2017 10:47:13 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  70.433 BB    2.4415 3.25218e4   191.00113  99.6151
   2  83.760 MM    1.2993  125.65295    1.61180   0.3849

Totals :                  3.26474e4   192.61293
```

Data File E:\DATA\HZY\SUANGAN\2OME-RAC 2016-12-06 16-11-08\032-0701.D
Sample Name: 2OME-PDC-90

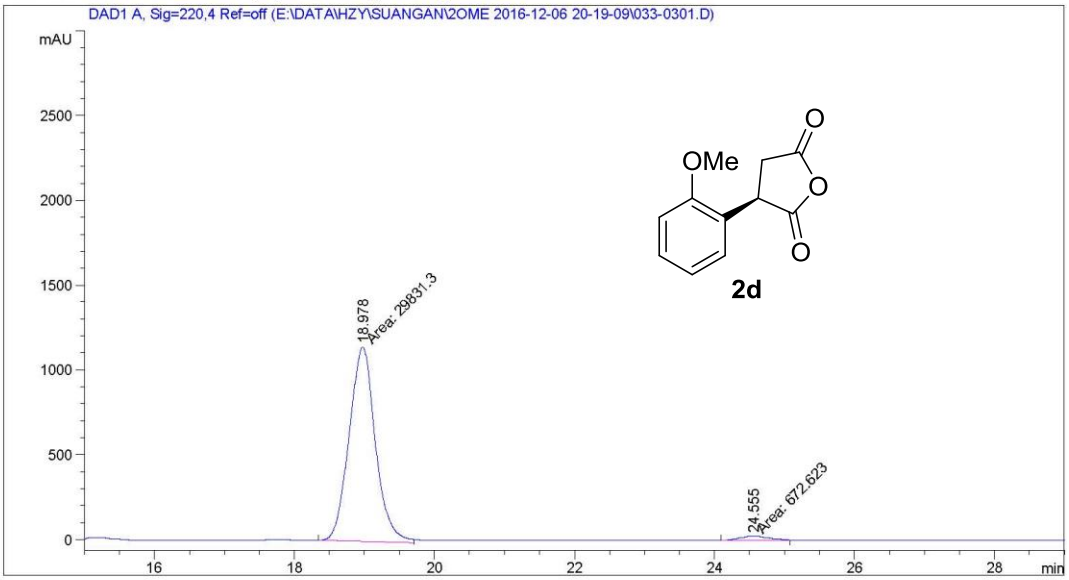
```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   7
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 32
Injection Date  : 12/6/2016 7:18:18 PM               Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\HZY\SUANGAN\2OME-RAC 2016-12-06 16-11-08\DAD-AD(1-2)-950-10-1.0ML-5
                  -ALL-60MIN.M
Last changed    : 12/6/2016 7:21:34 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\HZY\SUANGAN\2OME-RAC 2016-12-06 16-11-08\DAD-AD(1-2)-950-10-1.0ML-5
                  -ALL-60MIN.M (Sequence Method)
Last changed    : 12/6/2016 9:32:27 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=220,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  18.950 BB    0.3952 1.63472e4   629.07904  50.9966
   2  24.639 BB    0.4957 1.57083e4   482.61929  49.0034

Totals :                  3.20555e4  1111.69833
```

Data File E:\DATA\HZY\SUANGAN\2OME 2016-12-06 20-19-09\033-0301.D
Sample Name: 2OME-EE

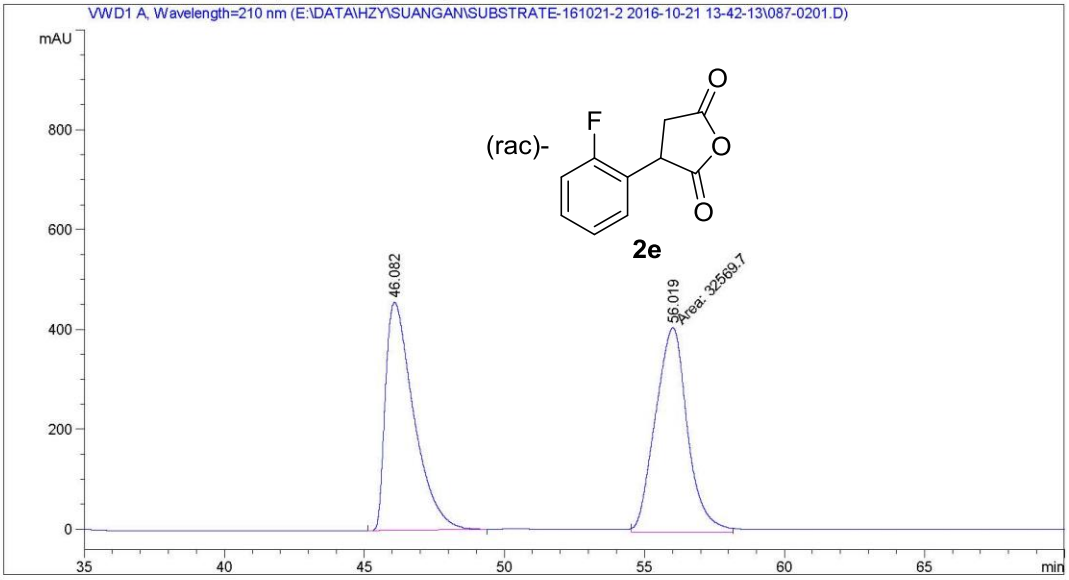
```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 33
Injection Date  : 12/6/2016 9:01:57 PM               Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\HZY\SUANGAN\2OME 2016-12-06 20-19-09\DAD-AD(1-2)-950-10-1.0ML-5-ALL
                  -60MIN.M
Last changed    : 12/6/2016 9:30:24 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\HZY\SUANGAN\2OME 2016-12-06 20-19-09\DAD-AD(1-2)-950-10-1.0ML-5-ALL
                  -60MIN.M (Sequence Method)
Last changed    : 12/6/2016 9:35:44 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=220,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  18.978 MM    0.4341 2.98313e4  1145.43665  97.7950
   2  24.555 MM    0.4879  672.62268   22.97658   2.2050

Totals :                  3.05040e4  1168.41322
```

Data File E:\DATA\HZY\SUANGAN\SUBSTRATE-161021-2 2016-10-21 13-42-13\087-0201.D
Sample Name: H-7-RAC

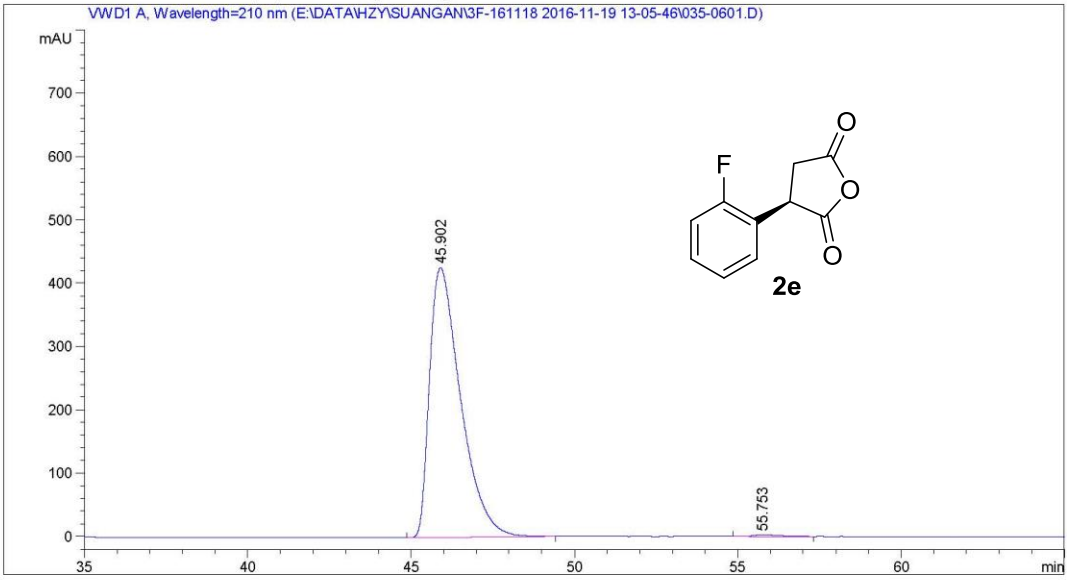
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   2
Acq. Instrument : 1260HPLC-VWD                    Location : Vial 87
Injection Date  : 10/21/2016 1:53:46 PM               Inj :   1
                                                 Inj Volume : 10.000 µl
Acq. Method     : E:\DATA\HZY\SUANGAN\SUBSTRATE-161021-2 2016-10-21 13-42-13\VWD-AD(1-6)-98-2
                  -1ML-85MIN-10UL-210NM.M
Last changed    : 10/21/2016 1:42:14 PM by SYSTEM
Analysis Method : E:\DATA\HZY\SUANGAN\SUBSTRATE-161021-2 2016-10-21 13-42-13\VWD-AD(1-6)-98-2
                  -1ML-85MIN-10UL-210NM.M (Sequence Method)
Last changed    : 11/19/2016 6:56:06 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  46.082 BB    1.0640 3.20332e4   456.87238  49.5847
   2  56.019 MM    1.3273 3.25697e4   408.98584  50.4153

Totals :                 6.46029e4   865.85822
```

Data File E:\DATA\HZY\SUANGAN\3F-161118 2016-11-19 13-05-46\035-0601.D
Sample Name: 2F

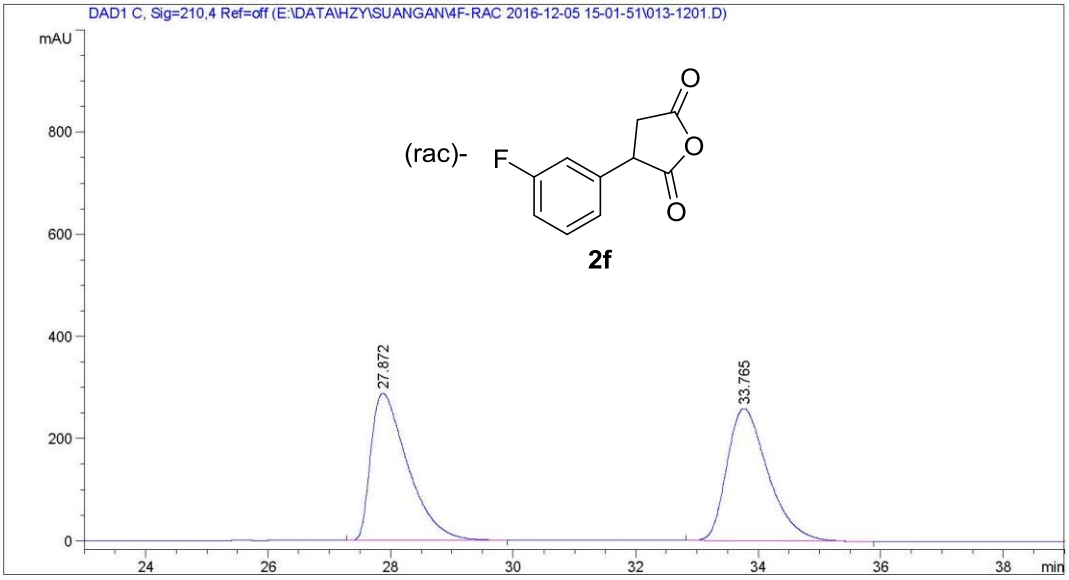
```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   6
Acq. Instrument : 1260HPLC-VWD                   Location : Vial 35
Injection Date  : 11/19/2016 4:39:51 PM               Inj :   1
                                                Inj Volume : 10.000 µl
Acq. Method     : E:\DATA\HZY\SUANGAN\3F-161118 2016-11-19 13-05-46\VWD-AD(1-6)-98-2-1ML-10UL
                  -210-85MIN.M
Last changed    : 11/19/2016 1:05:46 PM by SYSTEM
Analysis Method : E:\DATA\HZY\SUANGAN\3F-161118 2016-11-19 13-05-46\VWD-AD(1-6)-98-2-1ML-10UL
                  -210-85MIN.M (Sequence Method)
Last changed    : 11/19/2016 6:57:32 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  45.902 BB    1.0039 2.81280e4   425.66272  99.4292
   2  55.753 BB    0.8410  161.47997    2.47223   0.5708

Totals :                  2.82895e4   428.13495
```

Data File E:\DATA\HZY\SUANGAN\4F-RAC 2016-12-05 15-01-51\013-1201.D
Sample Name: 3F-ER

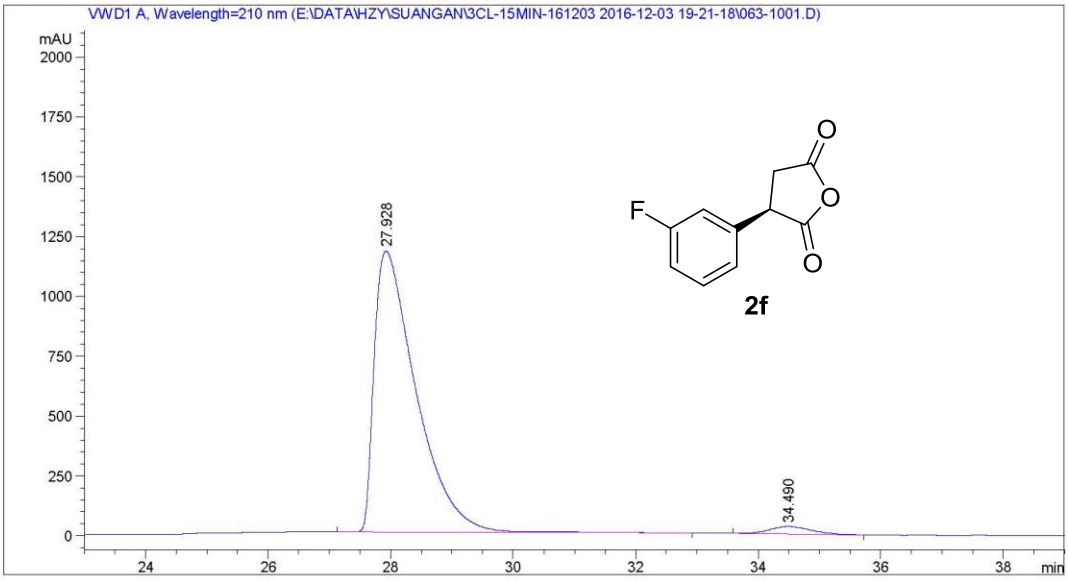
```
=====================================================================
Acq. Operator   : SYSTEM                        Seq. Line :  12
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 13
Injection Date  : 12/5/2016 9:32:26 PM               Inj :   1
                                                Inj Volume : 10.000 µl
Acq. Method     : E:\DATA\HZY\SUANGAN\4F-RAC 2016-12-05 15-01-51\DAD-AD(1-2)-93-7-1.0ML-10-
                  ALL-50MIN.M
Last changed    : 12/5/2016 5:38:50 PM by SYSTEM
Analysis Method : E:\DATA\HZY\SUANGAN\4F-RAC 2016-12-05 15-01-51\DAD-AD(1-2)-93-7-1.0ML-10-
                  ALL-50MIN.M (Sequence Method)
Last changed    : 12/6/2016 9:53:48 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  27.872 BB    0.6337 1.20300e4   287.79364  50.0087
   2  33.765 BB    0.7060 1.20258e4   258.84329  49.9913

Totals :                  2.40558e4   546.63693
```

Data File E:\DATA\HZY\SUANGAN\3CL-15MIN-161203 2016-12-03 19-21-18\063-1001.D
Sample Name: 3F

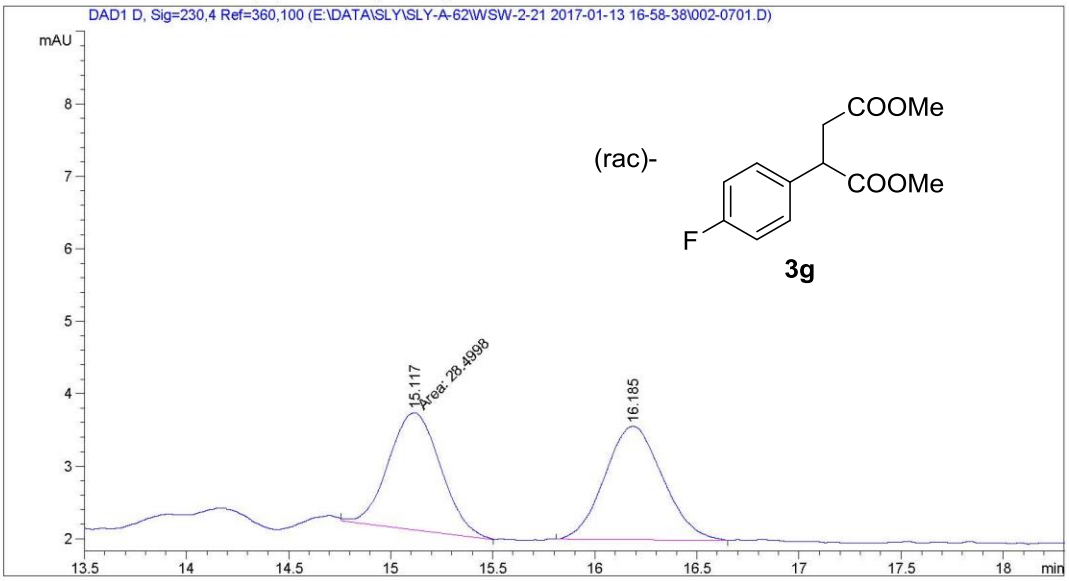
```
======================================================================
Acq. Operator   : SYSTEM                          Seq. Line :  10
Acq. Instrument : 1260HPLC-VWD                     Location : Vial 63
Injection Date  : 12/3/2016 10:37:02 PM                 Inj :   1
                                                  Inj Volume : 10.000 µl
Acq. Method     : E:\DATA\HZY\SUANGAN\3CL-15MIN-161203 2016-12-03 19-21-18\VWD-AD(1-2)-93-7-1
                  .0ML-10UL-210NM-50MIN.M
Last changed    : 12/3/2016 10:56:38 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\HZY\SUANGAN\3CL-15MIN-161203 2016-12-03 19-21-18\VWD-AD(1-2)-93-7-1
                  .0ML-10UL-210NM-50MIN.M (Sequence Method)
Last changed    : 12/6/2016 9:50:12 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  27.928 BB    0.7088 5.54647e4  1172.71887  97.4936
   2  34.490 BB    0.7224 1425.90808   30.33747   2.5064

Totals :                  5.68906e4  1203.05635
```

Data File E:\DATA\SLY\SLY-A-62\WSW-2-21 2017-01-13 16-58-38\002-0701.D
Sample Name: 4F-RAC-90

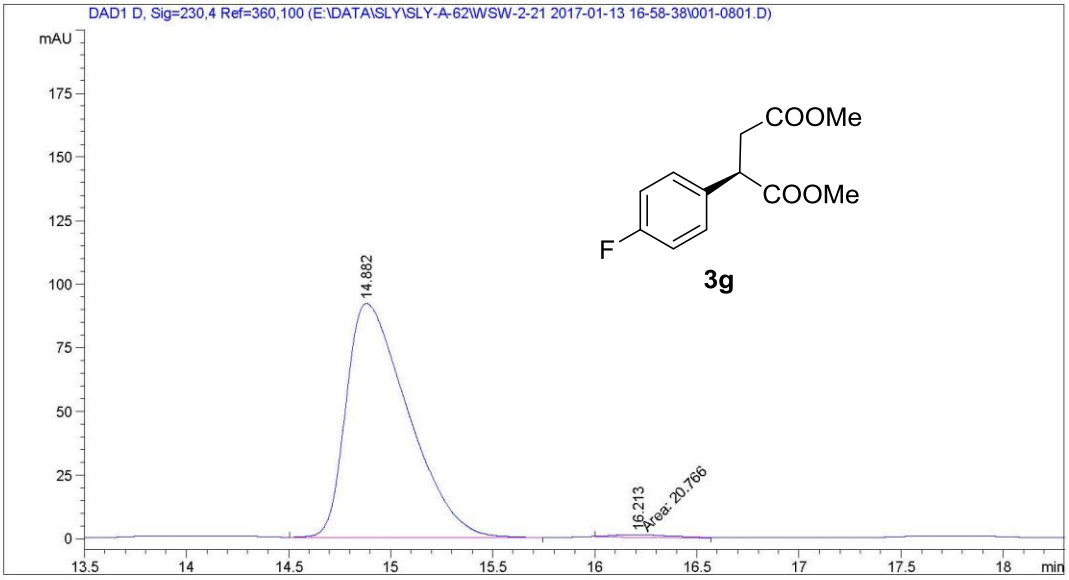
```
=====================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   7
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 2
Injection Date  : 1/13/2017 8:23:07 PM               Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\SLY\SLY-A-62\WSW-2-21 2017-01-13 16-58-38\DAD-OJ(1-2)-90-10-2UL-1ML
                  -20MIN.M
Last changed    : 1/13/2017 7:28:27 PM by SYSTEM
Analysis Method : E:\DATA\SLY\SLY-A-62\WSW-2-21 2017-01-13 16-58-38\DAD-OJ(1-2)-90-10-2UL-1ML
                  -20MIN.M (Sequence Method)
Last changed    : 1/14/2017 11:06:06 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=230,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  15.117 MM    0.2936   28.49981    1.61779  48.0550
   2  16.185 BB    0.2859   30.80683    1.56365  51.9450

Totals :                    59.30663    3.18143
```

Data File E:\DATA\SLY\SLY-A-62\WSW-2-21 2017-01-13 16-58-38\001-0801.D
Sample Name: 4F-EE-90

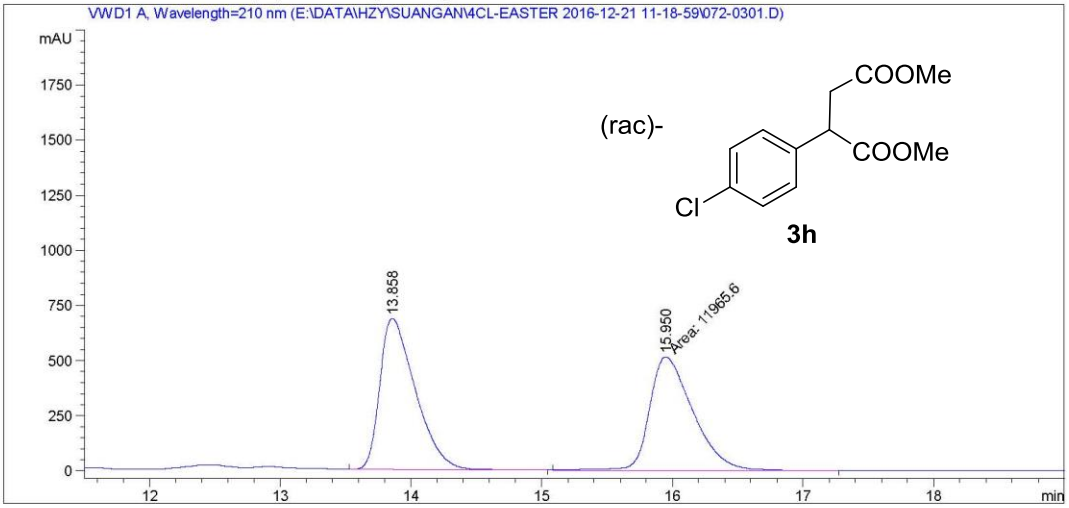
```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   8
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 1
Injection Date  : 1/13/2017 8:44:01 PM               Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\SLY\SLY-A-62\WSW-2-21 2017-01-13 16-58-38\DAD-OJ(1-2)-90-10-2UL-1ML
                  -20MIN.M
Last changed    : 1/13/2017 7:28:27 PM by SYSTEM
Analysis Method : E:\DATA\SLY\SLY-A-62\WSW-2-21 2017-01-13 16-58-38\DAD-OJ(1-2)-90-10-2UL-1ML
                  -20MIN.M (Sequence Method)
Last changed    : 1/14/2017 11:08:13 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=230,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  14.882 BB    0.3185 1922.82996   91.76545  98.9316
   2  16.213 MM    0.3546   20.76598 9.76038e-1   1.0684

Totals :                  1943.59594   92.74149
```

Data File E:\DATA\HZY\SUANGAN\4CL-EASTER 2016-12-21 11-18-59\072-0301.D
Sample Name: 4CL-EASTER-RAC

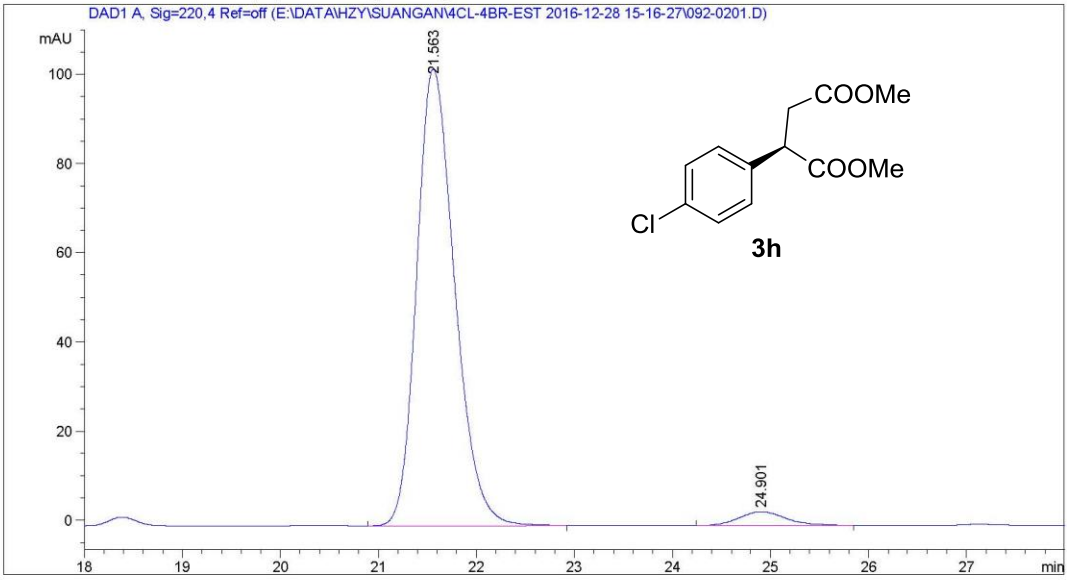
```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260HPLC-VWD                   Location : Vial 72
Injection Date  : 12/21/2016 12:31:13 PM              Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\HZY\SUANGAN\4CL-EASTER 2016-12-21 11-18-59\VWD-AD(1-6)-90-10-1ML-
                  2UL-210NM-60MIN.M
Last changed    : 12/21/2016 11:18:59 AM by SYSTEM
Analysis Method : E:\DATA\HZY\SUANGAN\4CL-EASTER 2016-12-21 11-18-59\VWD-AD(1-6)-90-10-1ML-
                  2UL-210NM-60MIN.M (Sequence Method)
Last changed    : 12/21/2016 6:27:08 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.858 BB    0.2859 1.28946e4   684.12494  51.8684
   2  15.950 MM    0.3871 1.19656e4   515.14160  48.1316

Totals :                  2.48603e4  1199.26654


======================================================================
                        *** End of Report ***
```

Data File E:\DATA\HZY\SUANGAN\4CL-4BR-EST 2016-12-28 15-16-27\092-0201.D
Sample Name: 4CL-EST

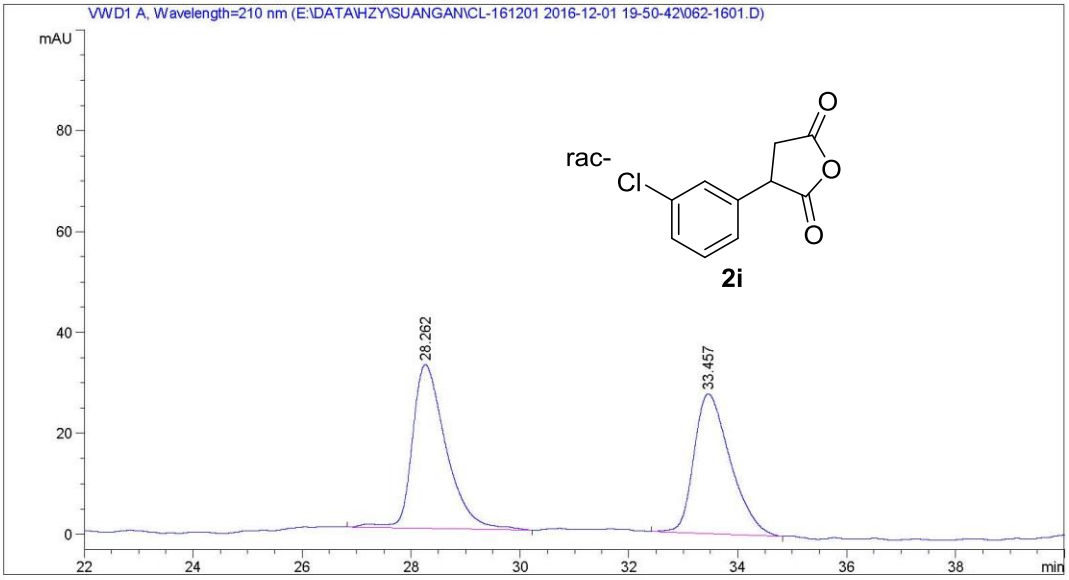
```
=====================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 92
Injection Date  : 12/28/2016 3:28:34 PM               Inj :   1
                                                Inj Volume : 1.000 µl
Acq. Method     : E:\DATA\HZY\SUANGAN\4CL-4BR-EST 2016-12-28 15-16-27\DAD-OJ(1-6)-98-2-1UL-
                  1ML-50MIN.M
Last changed    : 12/28/2016 3:16:36 PM by SYSTEM
Analysis Method : E:\DATA\HZY\SUANGAN\4CL-4BR-EST 2016-12-28 15-16-27\DAD-OJ(1-6)-98-2-1UL-
                  1ML-50MIN.M (Sequence Method)
Last changed    : 1/15/2017 9:59:01 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=220,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  21.563 BB    0.4157 2755.22803  102.46368  96.4520
   2  24.901 BB    0.4617  101.35017    3.06556   3.5480

Totals :                  2856.57819  105.52924
```

Data File E:\DATA\HZY\SUANGAN\CL-161201 2016-12-01 19-50-42\062-1601.D
Sample Name: 3CL-RAC

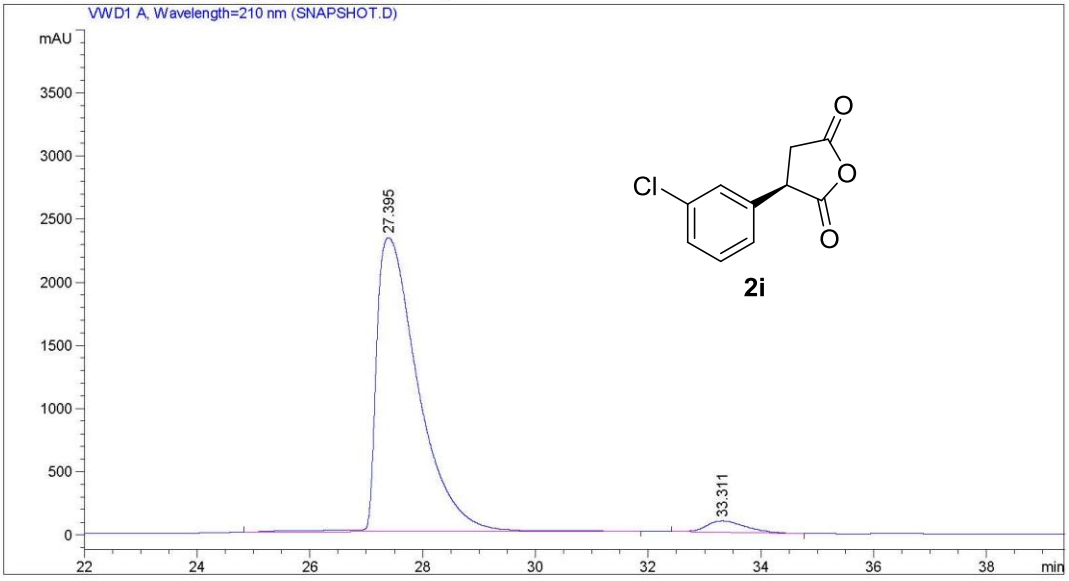
```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :  16
Acq. Instrument : 1260HPLC-VWD                   Location : Vial 62
Injection Date  : 12/2/2016 6:17:40 AM               Inj :   1
                                                Inj Volume : 10.000 µl
Acq. Method     : E:\DATA\HZY\SUANGAN\CL-161201 2016-12-01 19-50-42\VWD-AD(1-2)-93-7-1.0ML-
                  10UL-210NM-50MIN.M
Last changed    : 12/1/2016 7:50:43 PM by SYSTEM
Analysis Method : E:\DATA\HZY\SUANGAN\CL-161201 2016-12-01 19-50-42\VWD-AD(1-2)-93-7-1.0ML-
                  10UL-210NM-50MIN.M (Sequence Method)
Last changed    : 12/3/2016 9:37:21 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  28.262 BB    0.6506 1394.35962   32.42965  52.0825
   2  33.457 BB    0.7101 1282.85388   27.71011  47.9175

Totals :                  2677.21350   60.13976
```

Data File C:\CHEM32\1\DATA\SNAPSHOT.D
Sample Name:

```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   6
                                                   Location : Vial 62
Injection Date  : 12/3/2016 8:59:40 PM                Inj :   1
Acq. Method     : VWD-AD(1-2)-93-7-1.0ML-10UL-210NM-50MIN.M
Analysis Method : E:\DATA\LXX\LXX-CN-COOME\LXX-3-175 2016-12-03 08-32-19\VWD-OD(1-6)-92-8-0.
                  8ML3UL-210NM-35MIN.M (Sequence Method)
Last changed    : 12/3/2016 9:39:50 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



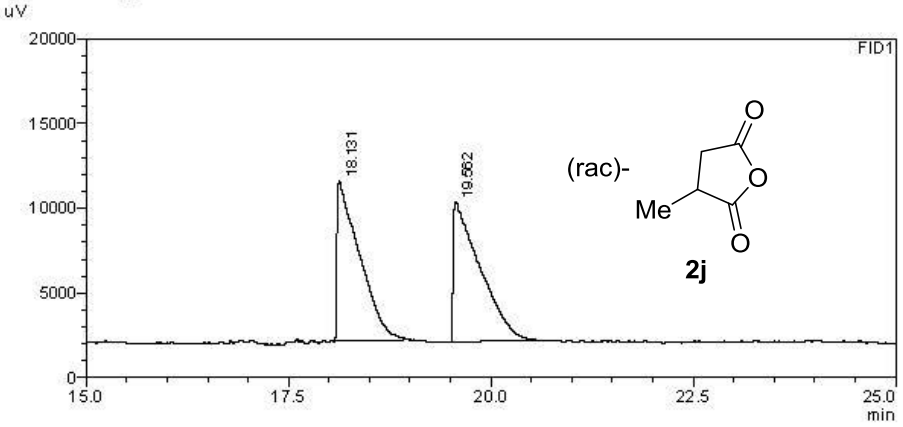

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  27.395 BB    0.7721 1.19439e5  2329.70044  96.4828
   2  33.311 BB    0.7331 4354.04248   90.85255   3.5172

Totals :                  1.23793e5  2420.55299


==========================================================================
                        *** End of Report ***
```

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: sg-me-rac		
Sample ID	:		
Data Filename	: sg-me-rac.gcd		
Method Filename	: b-dex225-250-100-150-260-30min.gcm		
Batch Filename	: sg-me.gcb		
Vial #	: 27	Sample Type	: Unknown
Injection Volume	: 1 uL		
Date Acquired	: 2016-12-27 21:19:38	Acquired by	: System Administrator
Date Processed	: 2016-12-27 21:49:43	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

FID1

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	18.131	181465	9448	49.078		M	
2	19.562	188286	8233	50.922		M	
Total		369751	17681				

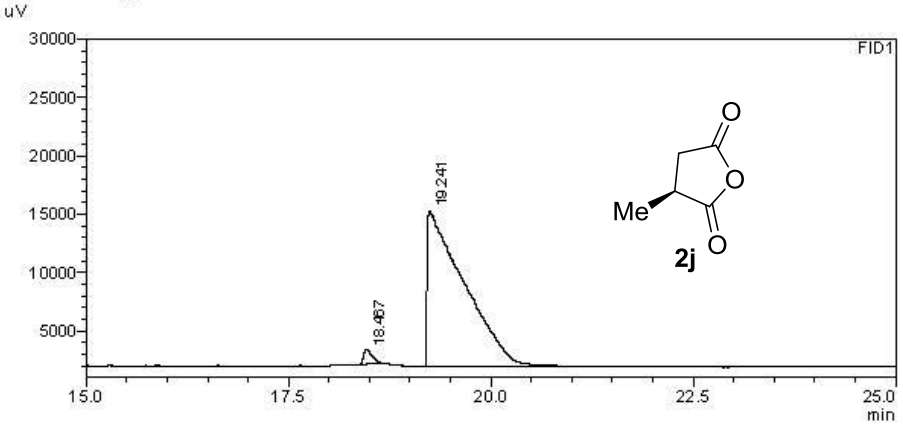
D:\DATA FILE\HZY\DATA\me\sg-me-rac.gcd

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: sg-me-2		
Sample ID	:		
Data Filename	: sg-me-2.gcd		
Method Filename	: b-dex225-250-100-150-260-30min.gcm		
Batch Filename	: sg-me-2.gcb		
Vial #	: 26	Sample Type	: Unknown
Injection Volume	: 1 uL		
Date Acquired	: 2016-12-27 22:08:50	Acquired by	: System Administrator
Date Processed	: 2016-12-27 22:38:55	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

FID1

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	18.467	9285	1258	2.163		M	
2	19.241	420072	13349	97.837		M	
Total		429357	14607				

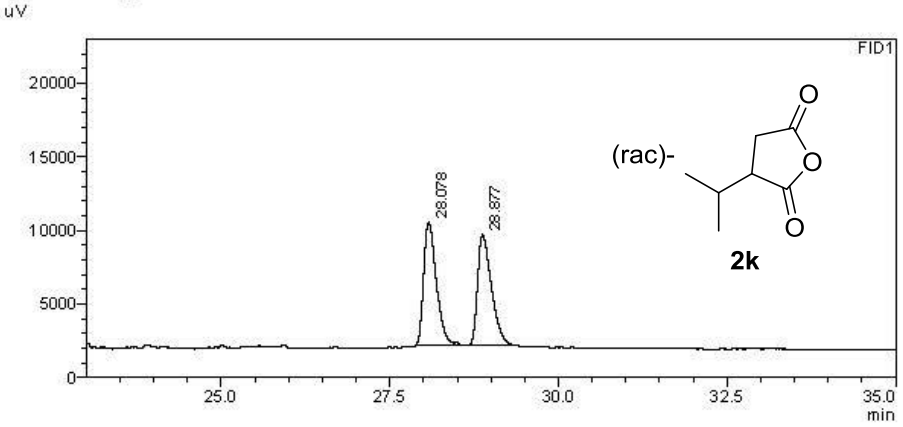
D:\DATA FILE\HZY\DATA\me\sg-me-2.gcd

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ipr-pdc		
Sample ID	:		
Data Filename	: sg-ipr-pdc-rare-1.gcd		
Method Filename	: b-dex120-250-100(0)-2-120(30)-260-40min.gcm		
Batch Filename	: hzy-ipr-pdc-rare.gcb		
Vial #	: 26	Sample Type	: Unknown
Injection Volume	: 1 uL		
Date Acquired	: 2016-12-29 12:11:30	Acquired by	: System Administrator
Date Processed	: 2016-12-29 12:51:34	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

FID1

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	28.078	109442	8392	50.552		M	
2	28.877	107050	7479	49.448		M	
Total		216492	15872				

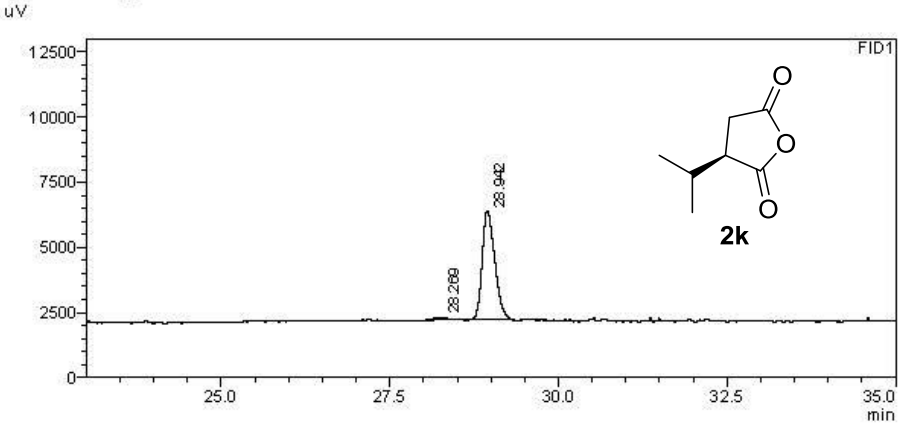
D:\DATA FILE\HZY\DATA\ipr\sg-ipr-pdc-rare-1.gcd

SHIMADZU LabSolutions **Analysis Report**

<Sample Information>

Sample Name	: ipr-ee		
Sample ID	:		
Data Filename	: sg-ipr-ee.gcd		
Method Filename	: b-dex120-250-100(0)-2-120(30)-260-40min.gcm		
Batch Filename	: hzy-ipr-2.gcb		
Vial #	: 28	Sample Type	: Unknown
Injection Volume	: 1 uL		
Date Acquired	: 2016-12-29 9:21:12	Acquired by	: System Administrator
Date Processed	: 2016-12-29 10:01:16	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

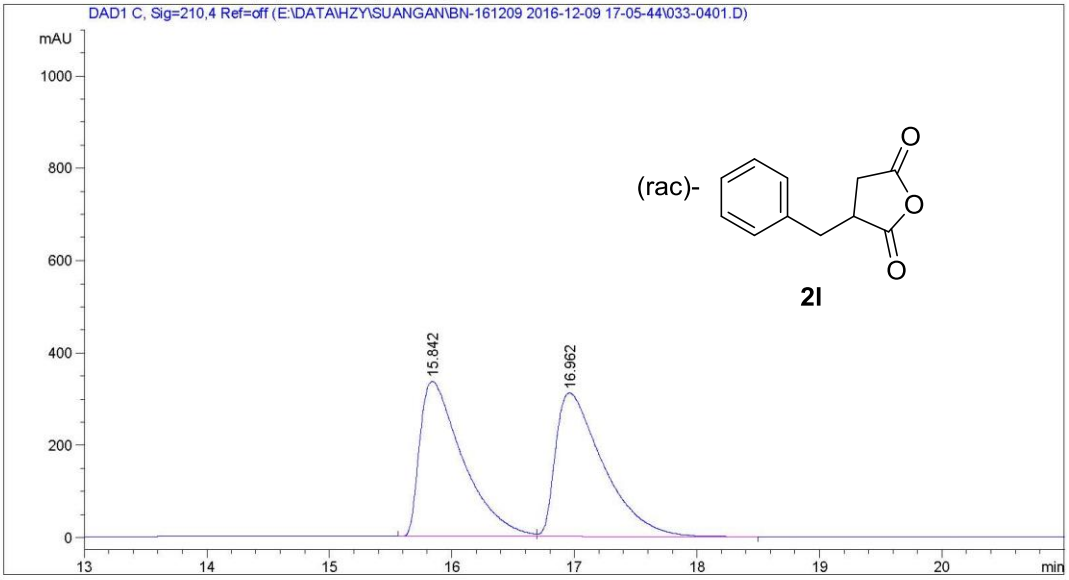
FID1

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	28.269	1369	100	2.457		M	
2	28.942	54321	4146	97.543			
Total		55689	4246				

D:\DATA FILE\HZY\DATA\ipr\sg-ipr-ee.gcd

Data File E:\DATA\HZY\SUANGAN\BN-161209 2016-12-09 17-05-44\033-0401.D
Sample Name: BN-RAC

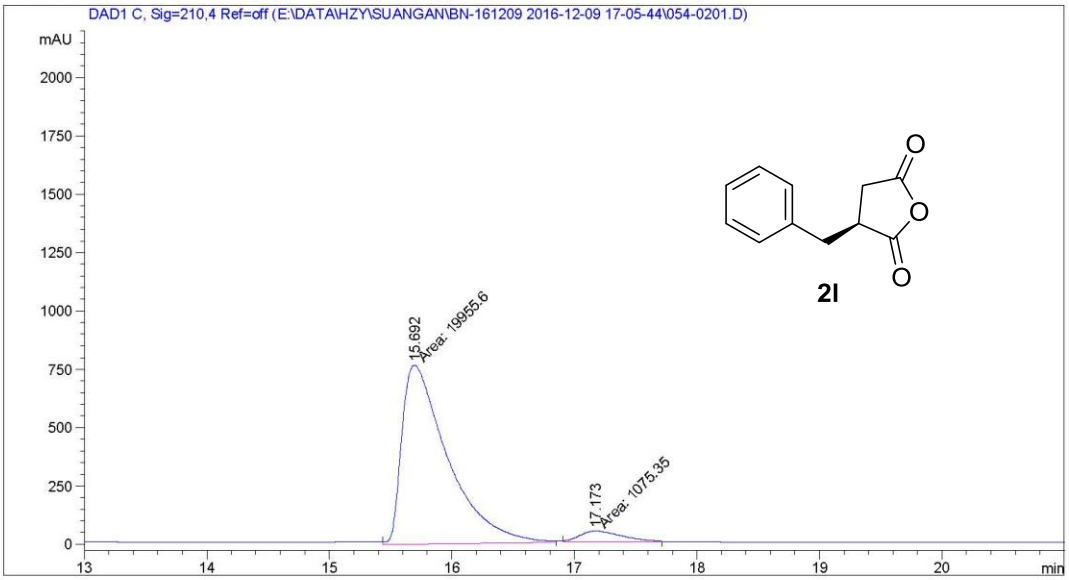
```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   4
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 33
Injection Date  : 12/9/2016 5:58:53 PM                Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\HZY\SUANGAN\BN-161209 2016-12-09 17-05-44\DAD-AD(1-2)-950-10-1.0ML-
                  5-ALL-60MIN.M
Last changed    : 12/9/2016 5:11:04 PM by SYSTEM
Analysis Method : E:\DATA\HZY\SUANGAN\BN-161209 2016-12-09 17-05-44\DAD-AD(1-2)-950-10-1.0ML-
                  5-ALL-60MIN.M (Sequence Method)
Last changed    : 12/10/2016 9:56:44 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  15.842 BV    0.3619 8166.53027  335.55035  49.3572
   2  16.962 VB    0.4002 8379.24609  311.12848  50.6428

Totals :                  1.65458e4   646.67883
```

Data File E:\DATA\HZY\SUANGAN\BN-161209 2016-12-09 17-05-44\054-0201.D
Sample Name: BN-RAC

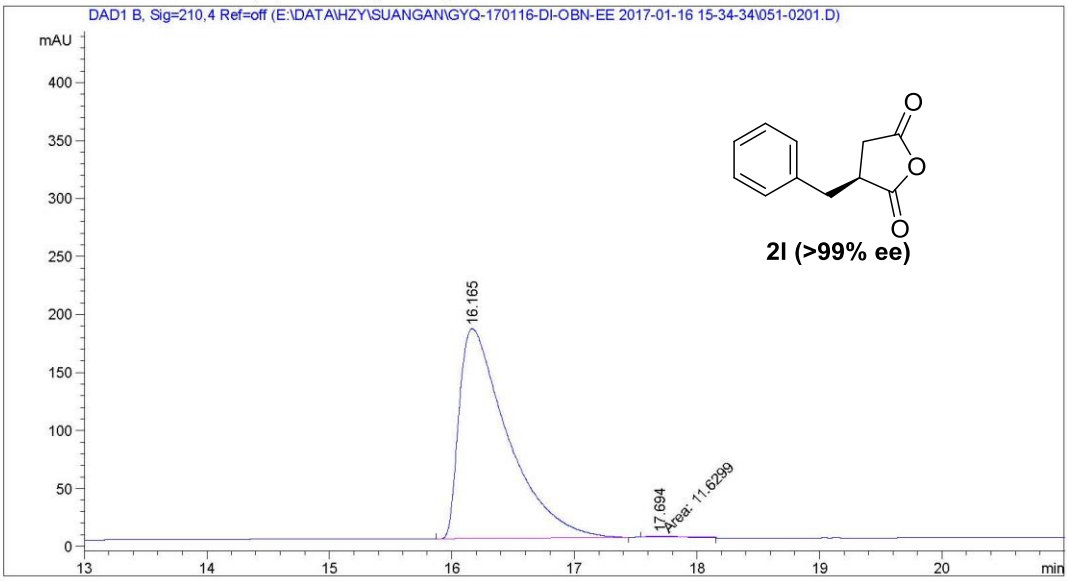
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   2
Acq. Instrument : 1260HPLC-DAD                    Location : Vial 54
Injection Date  : 12/9/2016 5:10:55 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\HZY\SUANGAN\BN-161209 2016-12-09 17-05-44\DAD-AD(1-2)-950-10-1.0ML-
                  5-ALL-60MIN.M
Last changed    : 12/9/2016 5:11:04 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\HZY\SUANGAN\BN-161209 2016-12-09 17-05-44\DAD-AD(1-2)-950-10-1.0ML-
                  5-ALL-60MIN.M (Sequence Method)
Last changed    : 12/10/2016 10:00:47 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  15.692 MM    0.4341 1.99556e4   766.11902  94.8868
   2  17.173 MM    0.3986 1075.34766   44.96540   5.1132

Totals :                  2.10310e4   811.08442
```

Data File E:\DATA\HZY\SUANGAN\GYQ-170116-DI-OBN-EE 2017-01-16 15-34-34\051-0201.D
Sample Name: BN-S

```
=====================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 51
Injection Date  : 1/16/2017 3:50:09 PM               Inj :   1
                                               Inj Volume : 10.000 µl
Acq. Method     : E:\DATA\HZY\SUANGAN\GYQ-170116-DI-OBN-EE 2017-01-16 15-34-34\DAD-AD(1-6)-90
                  -10-1ML-10UL-ALL-25MIN.M
Last changed    : 1/16/2017 3:34:34 PM by SYSTEM
Analysis Method : E:\DATA\HZY\SUANGAN\GYQ-170116-DI-OBN-EE 2017-01-16 15-34-34\DAD-AD(1-6)-90
                  -10-1ML-10UL-ALL-25MIN.M (Sequence Method)
Last changed    : 3/14/2017 9:34:29 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  16.165 BB    0.4027 4970.44580  180.83931  99.7666
   2  17.694 MM    0.2396   11.62992 8.08878e-1   0.2334

Totals :                  4982.07573  181.64819
```