Electronic Supplementary Information

for

$Mn(OAc)_3$–mediated arylation–lactonization of alkenoic acids: synthesis of γ, γ-disubstituted butyrolactones

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Experimental Section

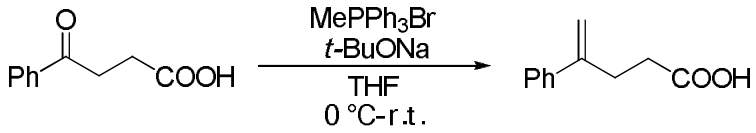
General:

All reactions were carried out under nitrogen atmosphere. All reagents were purchased and used without further purification. All new compounds were further characterized by HRMS (ESI).

Experimental procedure for the synthesis of $Mn(OAc)_3 \cdot 2H_2O$.

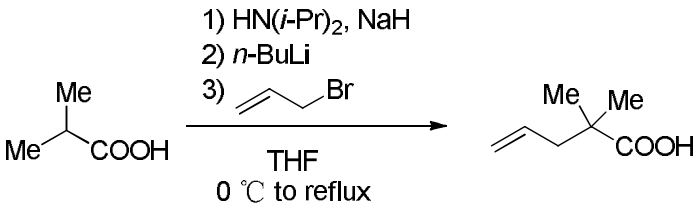
The $Mn(OAc)_3 \cdot 2H_2O$ was prepared by heating a mixture of 125 mL of acetic acid and 12 g of $Mn(OAc)_2 \cdot 4H_2O$ to reflux for 20 min, then slowly adding 2.0 g of $KMnO_4$. After refluxing for an additional 30 min, the mixture was cooled to room temperature and added a mixture of 20 mL of water. The manganic acetate was filtered off after 2 h, washed with cold acetic acid and diethyl ether, and then air dried.

Experimental procedure for the synthesis of 4-argiopent-4-enoic acid.



To a suspension of methyltriphenylphosphonium bromide (6.5 mmol) in THF (12 mL) was added sodium *t*-butoxide (13 mmol) at 0 °C. The mixture was then stirred for 30 min. 4-argio-4-oxobutanoic acid (5.0 mmol) was then added to the reaction mixture at 0 °C. The mixture was allowed to warm to room temperature, and then stirred for 16 h. After evaporation of THF, dichloromethane and 1 N NaOH were added. The aqueous layer was washed with dichloromethane. 12 N HCl was then added until pH of the aqueous layer was 2.0. The aqueous layer was then extracted with dichloromethane twice. The combined organic layer was dried over MgSO4, filtered and concentrated in vacuo. The residue was purified by silica gel column chromatography using petroleum ether–AcOEt (10:1-5:1, v/v) as the eluent to give the corresponding products. 4-Phenylpent-4-enoic acid, ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 10.7 (br s, 1 H), 7.46 – 7.43 (m, 2H), 7.39 – 7.36 (m, 2H), 7.34 – 7.30 (m, 1H), 5.37 (s, 1H), 5.15 (s, 1H), 2.89 (t, J = 7.4 Hz, 2H), 2.58 (t, J = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 179.7, 146.8, 140.6, 128.6, 127.9, 126.3, 113.2, 33.2, 30.3.

Experimental procedure for the synthesis of 2,2-dimethylpent-4-enoic acid



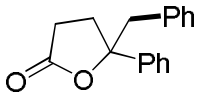
Isobutyric acid (880 mg, 10.0 mmol, 1.0 equiv) was added dropwise to a stirred suspension of NaH (60% dispersion in mineral oil, 480 mg, 12 mmol, 1.2 e quiv) and diisopropylamine (1.11g, 11 mmol, 1.1 equiv) in THF (20 mL). The resulting suspension was heated at reflux for ca 1 h and then cooled to 0°C for 15 min prior to the dropwise addition of *n*-BuLi (2.5 M in hexanes, 4.8 mL, 12 mmol, 1.2 equiv). The resulting suspension was stirred at 0°C for an additional 15 min and then at room temperature for 2 h. It was then cooled to 0°C and allyl bromide (1.20g, 10 mmol, 1.0 equiv) was added dropwise to give an off-white suspension which was stirred at 0°C for 1 h and then at room temperature overnight. The suspension was then cooled with an ice-bath and the excess of NaH was neutralized with water (20 mL). The organic layer was washed with a 1 M NaOH solution (3 x 20 mL) and the combined aqueous layers were then extracted with Et_2O (2 x 20 mL). The aqueous layer was acidified by addition of a 1 M HCl solution until pH 3 and it was then extracted with ether (3 x 30 mL). The combined organic layers were dried over MgSO4, filtered and the solvent was evaporated in vacuo. The crude product was purified by column chromatography (petroleum ether /EtOAc= 15:1) to afford 2,2-dimethylpent-4-enoic acid (282 mg, 2.20 mmol, 22%) as a light yellow oil. 2,2-Dimethylpent-4-enoic acid, ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 10.2 (br s, 1 H), 5.85 – 5.74 (m, 1 H), 5.11 - 5.08 (m, 2 H), 2.31 (d, 2H, J = 7.4 Hz), 1.22 (s, 6 H).

Experimental procedure for the arylation–lactonization of alkenoic acids

An oven-dried Schlenk tube containing $Mn(OAc)_3 \cdot 2H_2O$ (0.9 mmol), alkenoic acids (0.30 mmol) and phenylboronic acid (0.45 mmol) were evacuated and purged with nitrogen three times. CH_3COOH (2 mL) was sequentially added to the system at room temperature. The reaction mixture was heated with stirring at 60 °C for 6 hours. The reaction solution was concentrated in vacuo and then added 15 mL of saturated sodium bicarbonate solution and extracted with EtOAc (3×10 mL). The combined organic layer was dried over $MgSO_4$, filtered and concentrated in vacuo. The residue was purified by silica gel column chromatography using petroleum ether–AcOEt (15:1-5:1, v/v) as the eluent to give the corresponding products.

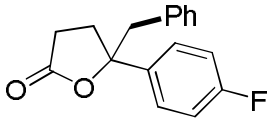
Spectral Data

*5-Benzyl-5-phenyldihydrofuran-2(3H)-one (**3a**) (CAS no: 66687-64-7).*



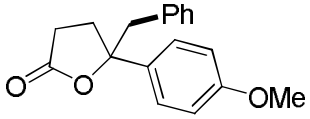
Colorless oil; 47 mg, 62% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.35 – 7.29 (m, 5H), 7.25 – 7.21 (m, 3H), 7.13 – 7.08 (m, 2H), 3.25 (d, *J* = 14.1 Hz, 1H), 3.11 (d, *J* = 14.1 Hz, 1H), 2.58 – 2.52 (m, 1H), 2.46 – 2.40 (m, 1H), 2.33 – 2.26 (m, 1H), 2.33 – 2.26 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.6, 143.6, 135.2, 130.8, 128.6, 128.4, 127.8, 127.2, 124.9, 89.1, 48.8, 33.1, 28.9. MS-ESI: *m/z* 275.0 ($[M+Na]^+$).

*5-Benzyl-5-(4-fluorophenyl)dihydrofuran-2(3H)-one (**3b**).*



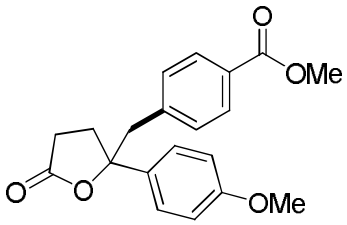
Colorless oil; 37 mg, 46% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.28 – 7.23 (m, 5H), 7.07 – 7.00 (m, 4H), 3.21 (d, *J* = 14.1 Hz, 1H), 3.10 (d, *J* = 14.0 Hz, 1H), 2.59 – 2.54 (m, 1H), 2.45 – 2.39 (m, 1H), 2.36 – 2.29 (m, 1H), 2.16 – 2.09 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.4, 162.3 (d, *J* = 247.1 Hz), 139.33 (d, *J* = 3.2 Hz), 134.9, 130.8, 128.5, 127.3, 126.8 (d, *J* = 8.1 Hz), 115.4 (d, *J* = 21.6 Hz), 88.7, 49.0, 33.3, 28.9. HRMS-ESI: *m/z* 293.0953 ($[M+Na]^+$, $C_{17}H_{15}FNaO_2^+$ calcd. 293.0948).

*5-Benzyl-5-(4-methoxyphenyl)dihydrofuran-2(3H)-one (**3c**) (CAS no: 101790-11-8).*



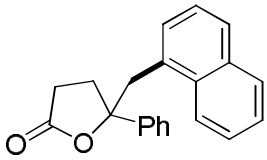
Colorless oil; 35 mg, 41% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.25 – 7.19 (m, 5H), 7.09 – 7.07 (m, 2H), 6.88 – 6.85 (m, 2H), 3.80 (s), 3.22 (d, *J* = 13.9 Hz, 1H), 3.11 (d, *J* = 14.0 Hz, 1H), 2.53 – 2.46 (m, 1H), 2.43 – 2.38 (m, 1H), 2.35 – 2.27 (m, 1H), 2.14 – 2.06 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.6, 159.2, 135.6, 135.3, 130.8, 128.4, 127.2, 126.3, 113.9, 89.1, 55.5, 49.0, 33.1, 29.0. MS-ESI: *m/z* 305.0 ($[M+Na]^+$).

*Methyl 3-((2-(4-methoxyphenyl)-5-oxotetrahydrofuran-2-yl)methyl)benzoate (**3d**).*



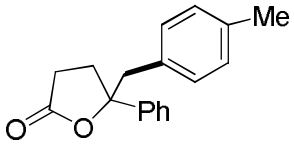
Colorless oil; 46 mg, 45% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.90 – 7.86 (m, 2H), 7.17 – 7.14 (m, 2H), 7.09 (d, *J* = 8.3 Hz, 2H), 6.84 – 6.82 (m, 2H), 3.88 (s, 3H), 3.78 (s, 3H), 3.26 (d, *J* = 13.9 Hz, 1H), 3.18 (d, *J* = 13.8 Hz, 1H), 2.51 – 2.44 (m, 2H), 2.40 – 2.32 (m, 1H), 2.28 – 2.20 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.4, 167.1, 159.3, 140.7, 134.6, 130.8, 129.5, 129.0, 126.2, 113.9, 88.8, 55.4, 52.2, 48.9, 33.7, 28.9. HRMS-ESI: *m/z* 363.1206 ($[M+Na]^+$, $C_{20}H_{20}NaO_5^+$ calcd. 363.1203).

*5-(Naphthalen-1-ylmethyl)-5-phenyldihydrofuran-2(3H)-one (**3e**)*.



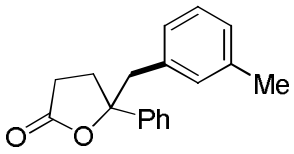
Yellow cream; 25 mg, 28% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.06 (d, *J* = 8.2 Hz, 1H), 7.90 (d, *J* = 7.8 Hz, 1H), 7.84 (d, *J* = 7.9 Hz, 1H), 7.57 – 7.35 (m, 9H), 3.77 (d, *J* = 14.7 Hz, 1H), 3.71 (d, *J* = 14.7 Hz, 1H), 2.65 – 2.59 (m, 1H), 2.50 – 2.45 (m, 1H), 2.30 – 2.23 (m, 1H), 1.96 – 1.90 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.4, 144.1, 134.0, 133.0, 131.5, 129.9, 129.0, 128.7, 128.3, 128.0, 126.3, 125.8, 125.5, 124.9, 124.1, 89.7, 44.4, 32.9, 29.1. HRMS-ESI: *m/z* 325.1201 ($[M+Na]^+$, $C_{21}H_{18}NaO_2^+$ calcd. 325.1199).

*5-(4-Methylbenzyl)-5-phenyldihydrofuran-2(3H)-one (**3f**)*.



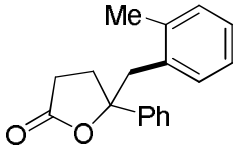
Colorless oil; 40 mg, 50% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.40 – 7.26 (m, 5H), 7.08 – 7.01 (m, 4H), 3.23 (d, *J* = 14.1 Hz, 1H), 3.07 (d, *J* = 14.1 Hz, 1H), 2.60 – 2.53 (m, 1H), 2.46 – 2.39 (m, 1H), 2.34 – 2.24 (m, 4H), 2.09 – 2.01 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.7, 143.9, 136.9, 132.1, 130.7, 129.2, 128.6, 127.8, 124.9, 89.2, 48.5, 33.0, 29.0, 21.2. HRMS-ESI: *m/z* 267.1380 ($[M+H]^+$, $C_{18}H_{19}O_2^+$ calcd. 267.1380).

*5-(3-Methylbenzyl)-5-phenyldihydrofuran-2(3H)-one (**3g**)*.



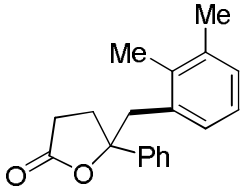
Colorless oil; 42 mg, 53% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.39 – 7.26 (m, 5H), 7.14 (t, *J* = 7.5 Hz, 1H), 7.05 (d, *J* = 7.6 Hz, 1H), 6.94 – 6.90 (m, 2H), 3.23 (d, *J* = 14.1 Hz, 1H), 3.07 (d, *J* = 14.0 Hz, 1H), 2.61 – 2.53 (m, 1H), 2.47 – 2.40 (m, 1H), 2.35 – 2.23 (m, 4H), 2.10 – 2.01 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.7, 143.9, 138.0, 135.1, 131.7, 128.6, 128.3, 128.0, 127.8, 124.9, 89.1, 48.8, 33.0, 29.0, 21.5. HRMS-ESI: *m/z* 267.1384 ($[M+H]^+$, $C_{18}H_{19}O_2^+$ calcd. 267.1380).

*5-(2-Methylbenzyl)-5-phenyldihydrofuran-2(3H)-one (**3h**)*.



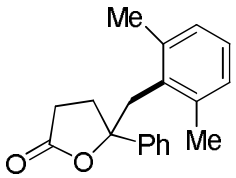
Colorless oil. 35 mg, 44% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.38 – 7.29 (m, 5H), 7.18 – 7.09 (m, 4H), 3.28 (d, *J* = 14.5 Hz, 1H), 3.19 (d, *J* = 14.4 Hz, 1H), 2.62 – 2.55 (m, 1H), 2.52 – 2.45 (m, 1H), 2.36 – 2.27 (m, 1H), 2.20 – 2.12 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.4, 143.8, 137.9, 133.7, 131.7, 130.8, 128.6, 127.9, 127.4, 125.9, 124.9, 89.8, 45.0, 33.1, 28.9, 20.1. HRMS-ESI: *m/z* 289.1202 ($[M+Na]^+$, $C_{18}H_{19}O_2^+$ calcd. 289.1199).

*5-(2,3-Dimethylbenzyl)-5-phenyldihydrofuran-2(3H)-one (**3i**)*.



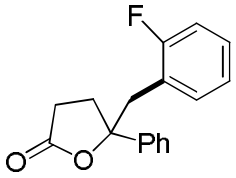
White solid; mp 111-112 ^{o}C; 31 mg, 37% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.34 – 7.25 (m, 5H), 7.05 – 6.95 (m, 3H), 3.30 (d, *J* = 14.5 Hz, 1H), 3.19 (d, *J* = 14.5 Hz, 1H), 2.60 – 2.53 (m, 1H), 2.45 – 2.38 (m, 1H), 2.29 – 2.21 (m, 4H), 2.09 – 2.00 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.4, 144.2, 137.5, 136.6, 133.5, 129.6, 129.1, 128.6, 127.8, 125.4, 124.9, 89.8, 45.6, 33.0, 28.9, 21.1, 16.1. HRMS-ESI: *m/z* 303.1357 ($[M+Na]^+$, $C_{19}H_{20}NaO_2^+$ calcd. 303.1356).

*5-(2,6-Dimethylbenzyl)-5-phenyldihydrofuran-2(3H)-one**(**3j**)*



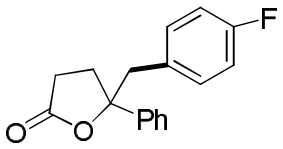
Colorless oil. 18 mg, 22% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.47 – 7.40 (m, 4H), 7.37 – 7.34 (m, 1H), 7.12 – 7.04 (m, 3H), 3.42 (d, *J* = 15.1 Hz, 1H), 3.25 (d, *J* = 15.1 Hz, 1H), 2.53 – 2.44 (m, 2H), 2,35 (s, 6H), 2.30 – 2.13 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.1, 144.4, 138.6, 132.8, 128.8, 128.0, 127.2, 125.0, 90.8, 41.3, 32.6, 28.9, 21.3. HRMS-ESI: *m/z* 303.1360 ($[M+Na]^+$, $C_{19}H_{20}NaO_2^+$ calcd. 303.1356).

*5-(2-Fluorobenzyl)-5-phenyldihydrofuran-2(3H)-one (CSA no: 1638127-88-4) (**3k**).*



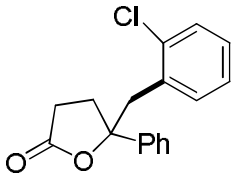
Colorless oil. 51 mg, 63% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.42 – 7.29 (m, 5H), 7.26 – 7.21 (m, 1H), 7.13 – 7.09 (m, 1H), 7.05 – 7.00 (m, 2H), 3.35 (d, *J* = 14.2 Hz, 1H), 3.23 (d, *J* = 14.2 Hz, 1H), 2.63 – 2.55 (m, 1H), 2.52 – 2.45 (m, 1H), 2.42 – 2.34 (m, 1H), 2.29 – 2.21 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.4, 161.5 (d, J = 245.4 Hz), 143.3, 133.23 (d, J = 3.9 Hz), 129.2 (d, J = 8.2 Hz), 128.6, 128.0, 124.9, 124.17 (d, J = 3.4 Hz), 122.3 (d, J = 15.2 Hz), 115.35 (d, J = 22.7 Hz), 89.0, 40.7, 33.2, 28.9. MS-ESI: *m/z* 293.0 ($[M+Na]^+$).

*5-(4-Fluorobenzyl)-5-phenyldihydrofuran-2(3H)-one (**3l**).*



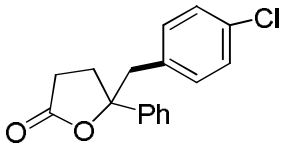
Colorless oil; 47 mg, 60% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.35 – 7.26 (m, 5H), 7.04 – 7.00 (m, 2H), 6.92 – 6.88 (m, 2H), 3.21 (d, *J* = 14.2 Hz, 1H), 3.11 (d, *J* = 14.2 Hz, 1H), 2.57 – 2.44 (m, 2H), 2.39 – 2.30 (m, 1H), 2.23 – 2.15 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.4, 162.1 (d, *J* = 245.2 Hz), 143.1, 132.24 (d, *J* = 8.0 Hz), 130.9 (d, *J* = 3.5 Hz), 128.6, 127.9, 124.9, 115.2 (d, *J* = 21.2 Hz), 88.9, 47.9, 33.4, 28.8. HRMS-ESI: *m/z* 271.1130 ($[M+H]^+$, $C_{17}H_{16}FO_2^+$ calcd. 271.1129).

*5-(2-Chlorobenzyl)-5-phenyldihydrofuran-2(3H)-one(**3m**)*.



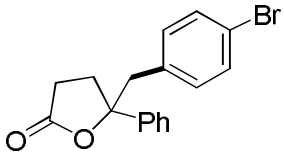
Colorless oil. 52 mg, 61% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.45 – 7.31 (m, 6H), 7.25 – 7.15 (m, 3H), 3.56 (d, *J* = 14.5 Hz, 1H), 3.32 (d, *J* = 14.5 Hz, 1H), 2.64 – 2.56 (m, 1H), 2.51 – 2.44 (m, 1H), 2.40 – 2.32 (m, 1H), 2.25 – 2.17 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.5, 143.6, 135.3, 133.3, 133.2, 129.7, 128.8, 128.7, 128.0, 127.0, 124.9, 89.3, 44.4, 32.9, 29.0. HRMS-ESI: *m/z* 287.0836 ($[M+H]^+$, $C_{17}H_{16}ClO_2^+$ calcd. 287.0833).

*5-(4-Chlorobenzyl)-5-phenyldihydrofuran-2(3H)-one (**n**)*.



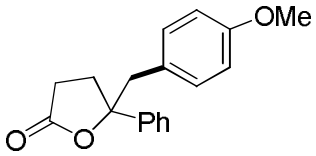
Colorless oil; 54 mg, 63% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.38 – 7.28 (m, 5H), 7.22 – 7.20 (m, 2H), 7.02 – 7.00 (m, 2H), 3.23 (d, *J* = 14.2 Hz, 1H), 3.14 (d, *J* = 14.1 Hz, 1H), 2.58 – 2.47 (m, 2H), 2.43 – 2.34 (m, 1H), 2.29 – 2.21 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.3, 142.9, 133.7, 133.2, 132.1, 128.6, 128.5, 128.0, 124.9, 88.8, 48.1, 33.5, 28.8. HRMS-ESI: *m/z* 287.0834 ($[M+H]^+$, $C_{17}H_{16}ClO_2^+$ calcd. 287.0833).

*5-(4-Bromobenzyl)-5-phenyldihydrofuran-2(3H)-one (**3o**)*.



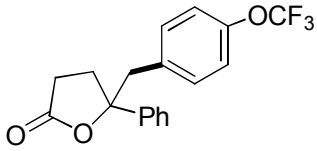
Yellow cream; 63 mg, 63% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.38 – 7.35 (m, 4H), 7.33 – 7.28 (m, 3H), 6.96 – 6.93 (m, 2H), 3.22 (d, *J* = 14.1 Hz, 1H), 3.13 (d, *J* = 14.0 Hz, 1H), 2.59 – 2.47 (m, 2H), 2.43 – 2.35 (m, 1H), 2.30 – 2.22 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.5, 142.8, 134.1, 132.4, 131.5, 128.7, 128.0, 124.9, 121.4, 88.9, 48.2, 33.5, 28.8. HRMS-ESI: *m/z* 353.0151 ($[M+Na]^+$, $C_{17}H_{15}BrNaO_2^+$ calcd. 353.0148).

*5-(4-Methoxybenzyl)-5-phenyldihydrofuran-2(3H)-one (**3p**)*.



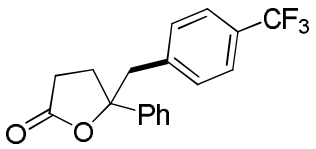
Colorless oil; 36 mg, 42% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.37 – 7.23 (m, 5H), 7.06 – 6.99 (m, 2H), 6.80 – 6.76 (m, 2H), 3,76 (s, 3H), 3.19 (d, *J* = 14.2 Hz, 1H), 3.04 (d, *J* = 14.2 Hz, 1H), 2.58 – 2.51 (m, 1H), 2.465 – 2.38 (m, 1H), 2.34 – 2.26 (m, 1H), 2.11 – 2.02 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.7, 158.9, 143.8, 131.8, 128.6, 127.8, 127.2, 124.9, 113.9, 89.2, 55.3, 48.0, 33.0, 29.0. HRMS-ESI: *m/z* 305.1147 ($[M+Na]^+$, $C_{18}H_{18}NaO_3^+$ calcd. 305.1148).

*5-Phenyl-5-(4-(trifluoromethoxy)benzyl)dihydrofuran-2(3H)-one (**3q**)*.



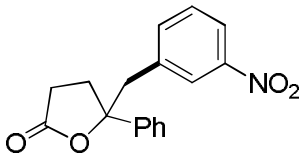
Colorless oil; 55 mg, 55% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.35 – 7.26 (m, 5H), 7.14 – 7.03 (m, 4H), 3.24 (d, *J* = 14.1 Hz, 1H), 3.15 (d, *J* = 14.1 Hz, 1H), 2.56 – 2.46 (m, 2H), 2.41 – 2.32 (m, 1H), 2.28 – 2.20 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.3, 148.5, 142.8, 134.0, 132.1, 128.6, 128.0, 124.9, 120.7, 88.8, 48.0, 33.6, 28.8. HRMS-ESI: *m/z* 337.1049 ($[M+H]^+$, $C_{18}H_{16}F_3O_3^+$ calcd. 337.1046).

*5-Phenyl-5-(4-(trifluoromethyl)benzyl)dihydrofuran-2(3H)-one (**3r**)*.



Colorless oil; 59 mg, 61% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.45 (d, *J* = 8.0 Hz, 2H), 7.36 – 7.26 (m, 5H), 7.15 (d, *J* = 8.0 Hz, 2H), 3.30 (d, *J* = 14.0 Hz, 1H), 3.22 (d, *J* = 14.0 Hz, 1H), 2.55 – 2.51 (m, 2H), 2.43 – 2.23 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.3, 142.6, 139.3, 131.1, 128.7, 128.1, 125.2 (q, *J* = 7.5Hz), 124.9, 88.7, 48.5, 33.8, 28.7. HRMS-ESI: *m/z* 343.0910 ($[M+Na]^+$, $C_{18}H_{15}F_3NaO_2^+$ calcd. 343.0916).

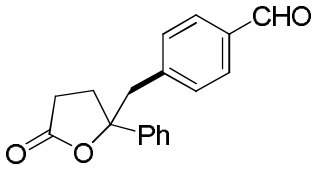
*5-(3-Nitrobenzyl)-5-phenyldihydrofuran-2(3H)-one (**3s**)*.



Yellow oil; 53 mg, 60% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.06 – 8.03 (m, 1H), 7.82 (s,

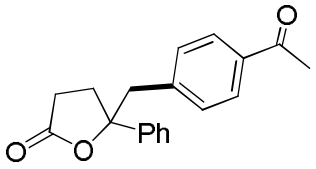
1H), 7.39 – 7.29 (m, 7H), 3.37 (d, *J* = 14.2 Hz, 1H), 3.34 (d, *J* = 14.2 Hz, 1H), 2.66 – 2.55 (m, 2H), 2.48 – 2.41 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.2, 148.0, 141.8, 137.2, 136.9, 129.1, 128.8, 128.3, 125.3, 124.9, 122.2, 88.6, 48.2, 34.2, 28.6. HRMS-ESI: *m/z* 320.0889 ($[M+Na]^+$, $C_{17}H_{15}NNaO_4^+$ calcd. 320.0893).

*4-((5-Oxo-2-phenyltetrahydrofuran-2-yl)methyl)benzaldehyde (**3t**)*.



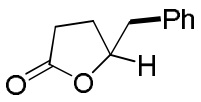
Colorless oil; 48 mg, 57% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 9.94 (s, 1H), 7.72 – 7.70 (m, 2H), 7.35 – 7.25 (m, 5H), 7.20 (d, J = 8.1 Hz, 2H), 3.33 (d, *J* = 13.9 Hz, 1H), 3.26 (d, *J* = 13.9 Hz, 1H), 2.58 – 2.53 (m, 2H), 2.44 – 2.25 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 192.1, 176.2, 142.5, 142.3, 135.3, 131.4, 129.6, 128.7, 128.1, 124.9, 88.7, 48.8, 33.9, 28.7. HRMS-ESI: *m/z* 303.0992 ($[M+Na]^+$, $C_{18}H_{16}NaO_3^+$ calcd. 303.0992).

*5-(4-Acetylbenzyl)-5-phenyldihydrofuran-2(3H)-one (**3u**)*.



White solid; mp 124-125 ^{o}C; 53 mg, 60% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.81 (d, J = 8.2 Hz, 2H), 7.36 – 7.27 (m, 5H), 7.15 (d, J = 8.2 Hz, 2H), 3.31 (d, *J* = 13.9 Hz, 1H), 3.22 (d, *J* = 13.9 Hz, 1H), 2,56 (s, 3H), 2.55 – 2.49 (m, 2H), 2.42 – 2.33 (m, 1H), 2.28 – 2.20 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 198.0, 176.2, 142.8, 140.8, 136.1, 131.0, 128.7, 128.4, 128.1, 124.9, 88.8, 48.7, 33.8, 28.8, 26.7. HRMS-ESI: *m/z* 317.1153 ($[M+Na]^+$, $C_{19}H_{18}NaO_3^+$ calcd. 317.1148).

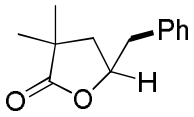
*5-Benzyldihydrofuran-2(3H)-one (**3v**) (CAS no: 21175-42-8)*.



Colorless oil. 14 mg, 26% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.34 – 7.30 (m, 2H), 7.27 – 7.22 (m, 3H), 4.76 – 4.70 (m, 1H), 3.06 (m, 1H), 2.92 (m, 1H), 2.50 – 2.33 (m, 2H), 2.29 – 2.20 (m, 1H), 2.00 – 1.90 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 177.2, 136.1, 129.6, 128.8,

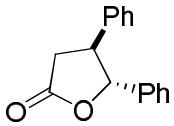
127.2, 81.0, 41.5, 28.8, 27.3. MS-ESI: *m/z* 199.0 ($[M+Na]^+$).

*5-Benzyl-3,3-dimethyldihydrofuran-2(3H)-one (**3w**) (CAS no: 1251739-93-1)*



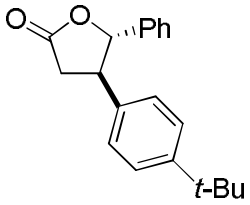
Colorless oil. 20 mg, 32% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.33 – 7.30 (m, 2H), 7.27 – 7.22 (m, 3H), 4.67 – 4.60 (m, 1H), 3.13 – 3.08 (m, 1H), 2.90 – 2.85 (m, 1H), 2.10 – 2.06 (m, 1H), 1.85 – 1.79 (m, 1H), 1.23 (d, *J* = 4.1 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 181.9, 136.4, 129.6, 128.8, 127.1, 77.5, 43.2, 41.9, 40.6, 25.2, 24.6. MS-ESI: *m/z* 227.1 ($[M+Na]^+$).

*4,5-Diphenyldihydrofuran-2(3H)-one (**3x**) (CAS no: 20453-83-2).*



White solid. 17 mg, 24% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.38 – 7.29 (m, 6H), 7.23 – 7.18 (m, 4H), 5.43 (d, *J* = 8.5 Hz, 1H), 3.64 – 3.57 (m, 1H), 3.10 – 3.04 (m, 1H), 2.96 – 2.89 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 175.5, 138.2, 138.0, 129.3, 128.8, 128.1, 127.5, 125.8, 87.6, 50.7, 37.3. MS-ESI: *m/z* 261.0 ($[M+Na]^+$).

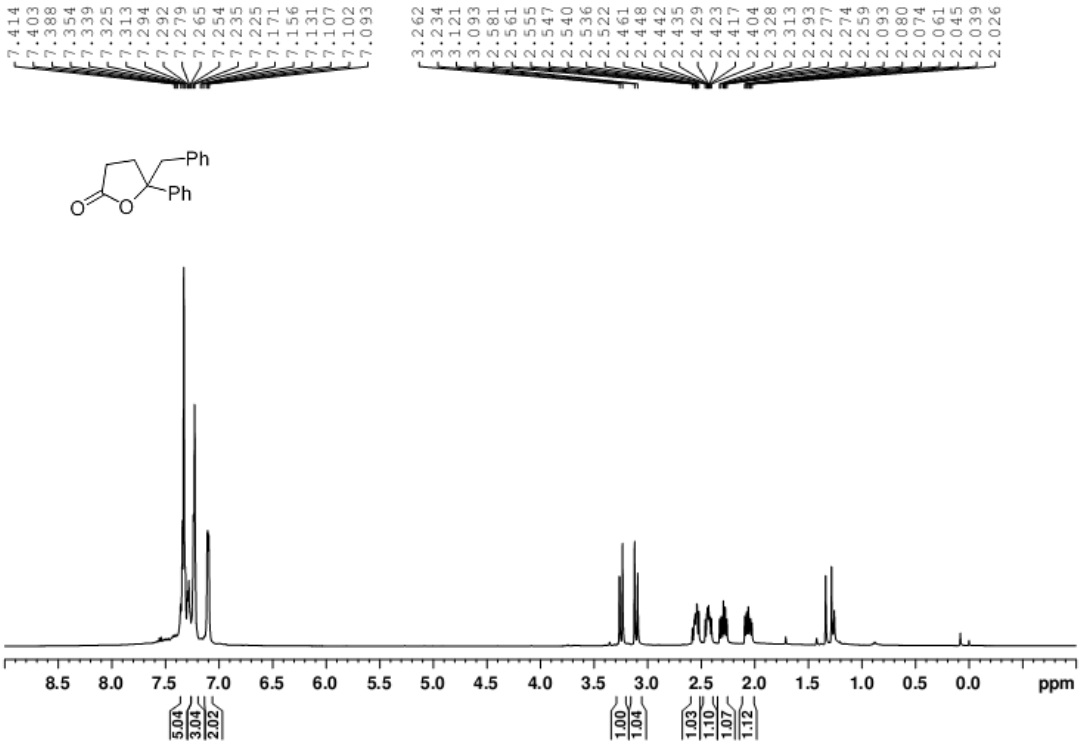
*4-(4-(tert-Butyl)phenyl)-5-phenyldihydrofuran-2(3H)-one (**3y**)*



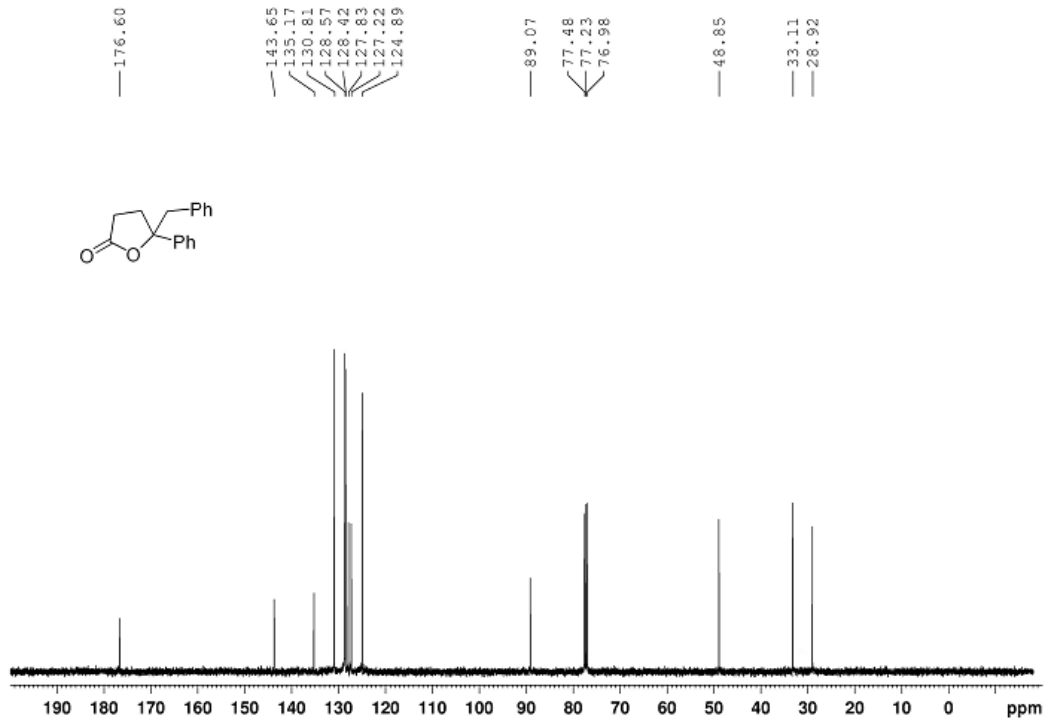
Yellow solid. 28 mg, 30% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.37 – 7.34 (m, 4H), 7.30 (d, *J* = 7.0 Hz, 1H), 7.20 (d, *J* = 7.4 Hz, 2H), 7.14 (d, *J* = 8.3 Hz, 2H), 5.44 (d, *J* = 8.3 Hz, 1H), 3.64 – 3.57 (m, 1H), 3.10 – 3.04 (m, 1H), 2.96 – 2.89 (m, 1H), 1.30 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 175.6, 151.9, 138.6, 134.9, 129.3, 128.0, 127.5, 125.8, 125.6, 87.5, 50.3 37.5, 34.8, 31.5. HRMS-ESI: *m/z* 317.1516 ($[M+Na]^+$, $C_{20}H_{22}NaO_2^+$ calcd. 317.1517).

^{1}H NMR and ^{13}C NMR spectra

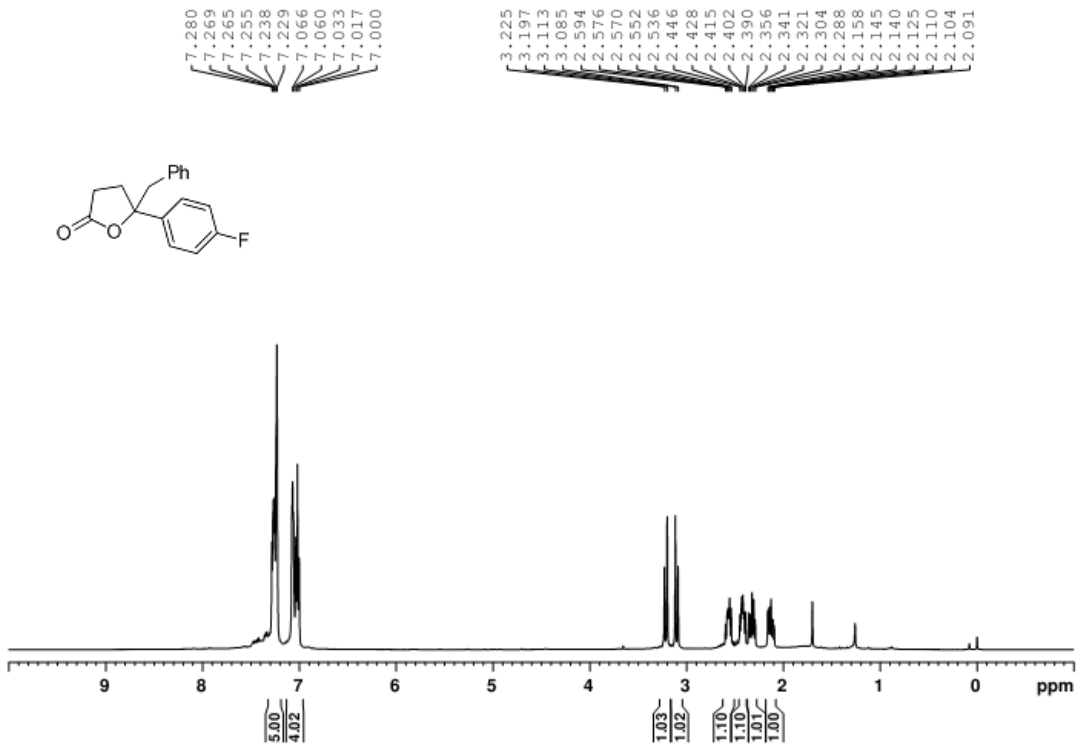
3a



3a



3b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3.113
3.085
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2.576
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2.536
2.446
2.428
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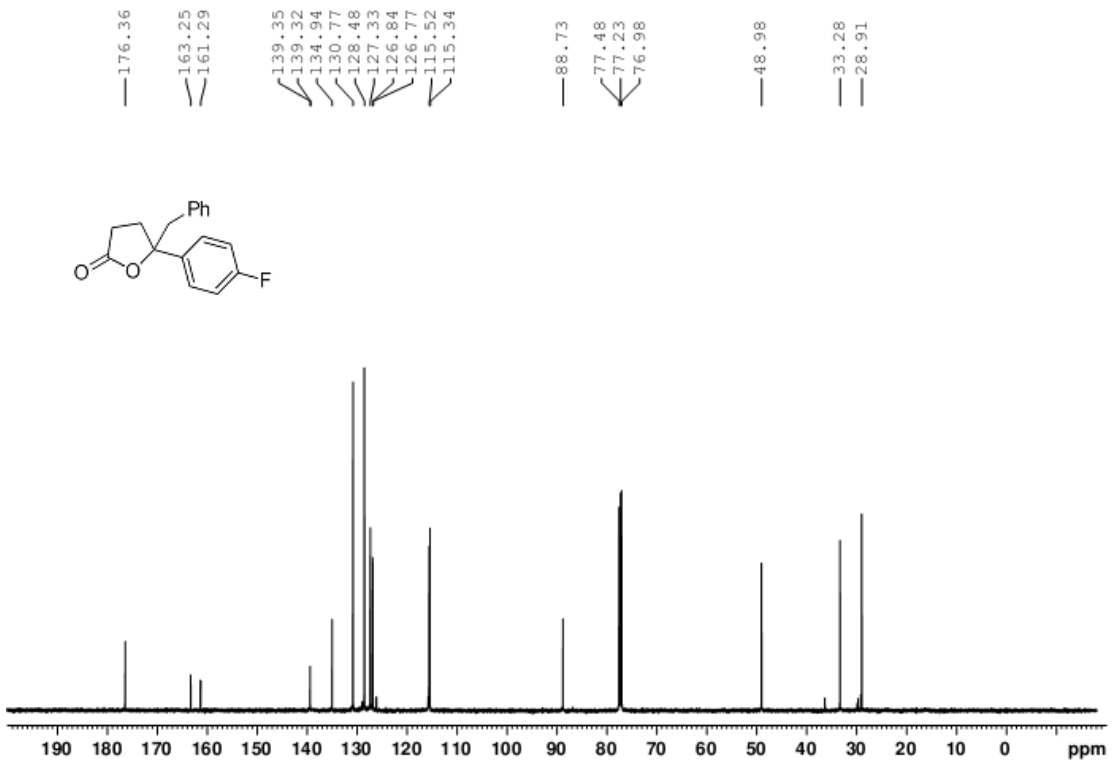
1.00

3b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50
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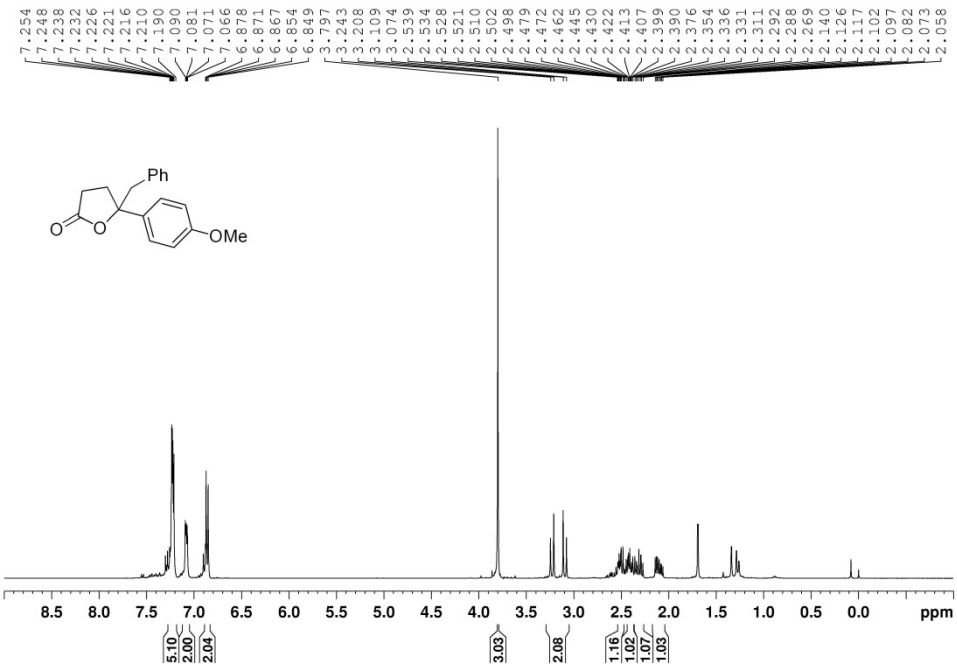
ppm

3c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OMe
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2.00
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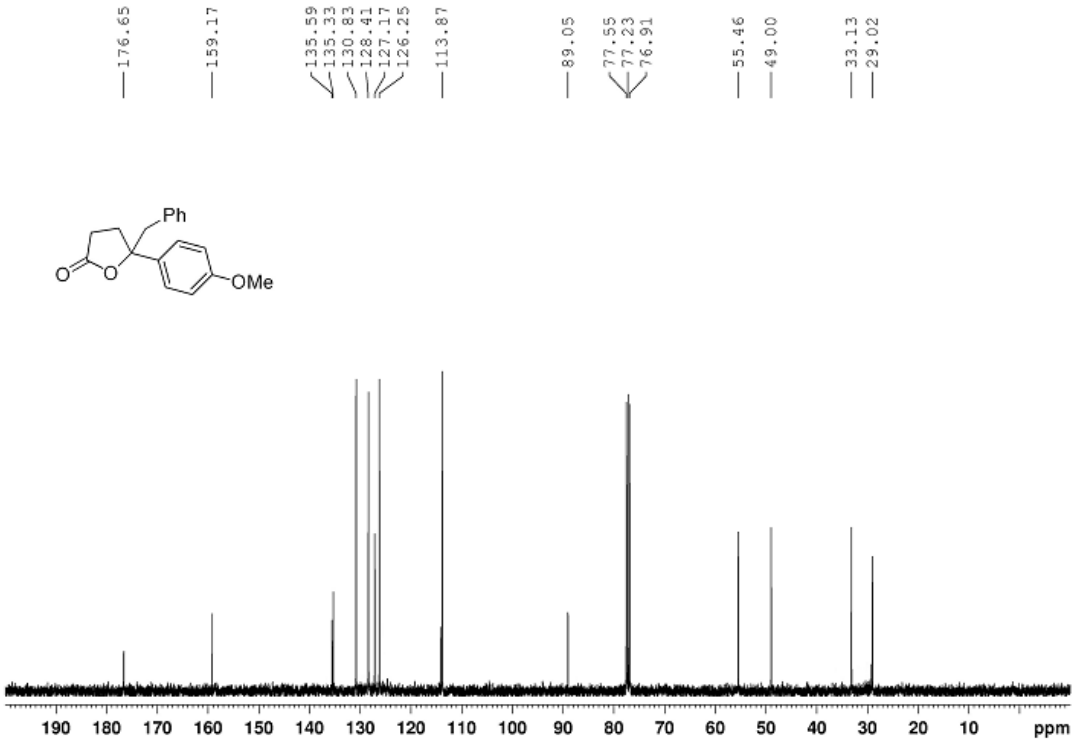
3.03
2.08
1.16
1.02
1.07
1.03

3c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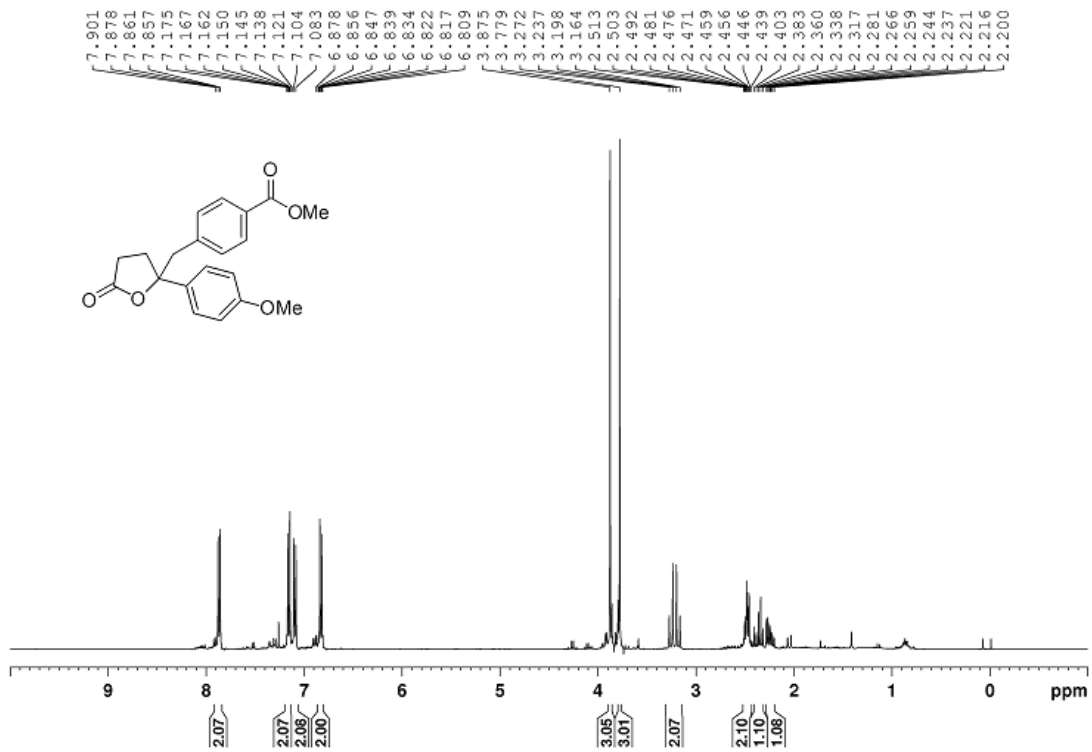
3d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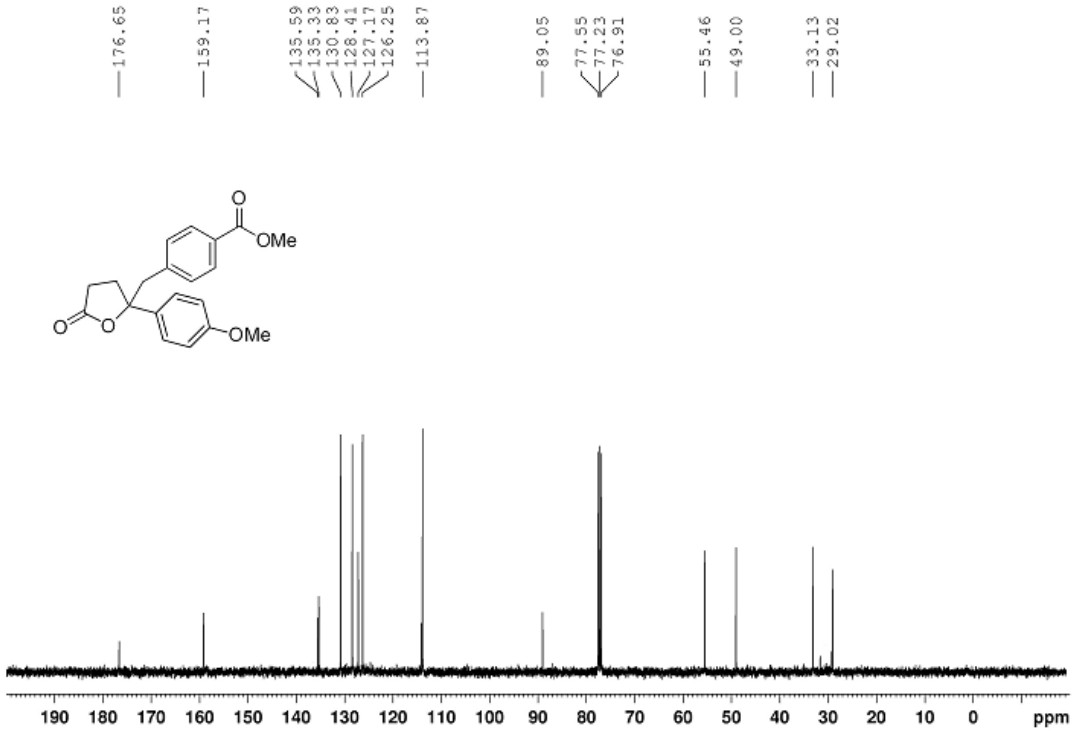
1.08

3d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OMe
190
180
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50
40
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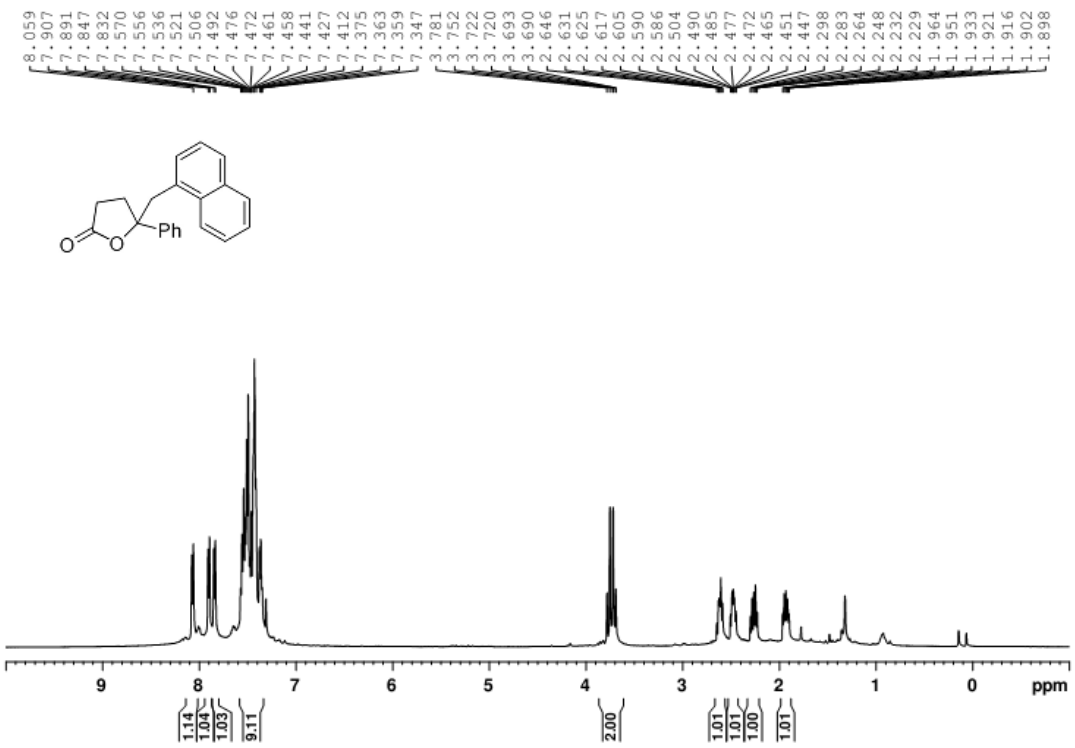
ppm

3e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Ph
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5
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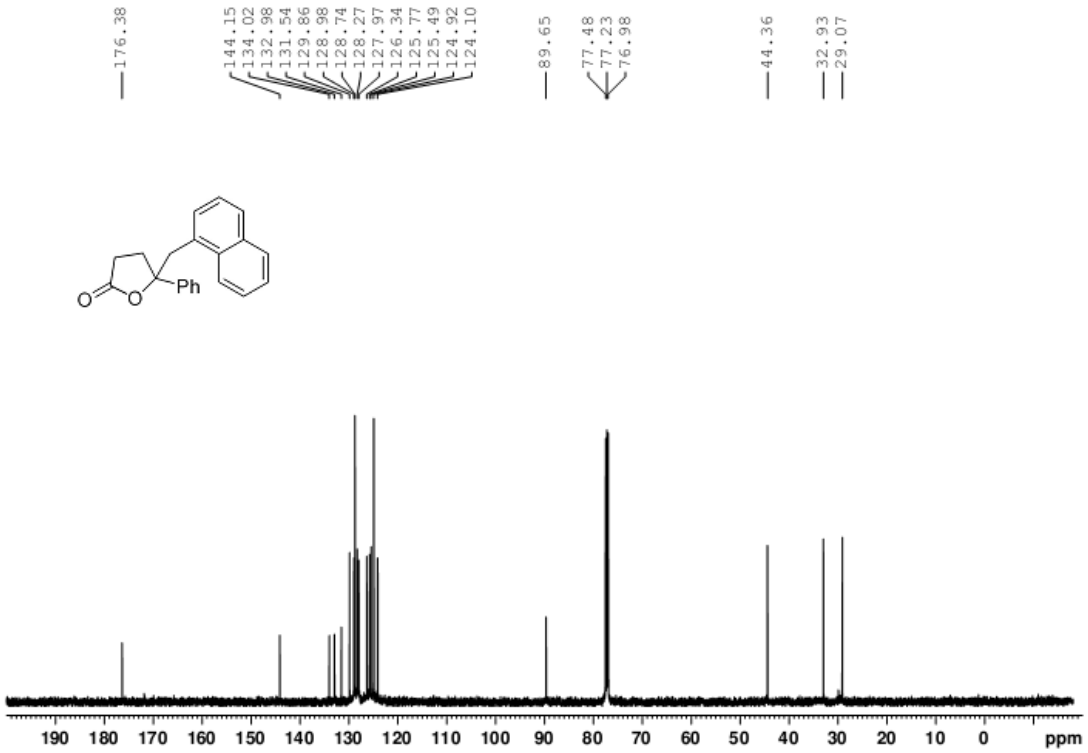
1.01
1.01
1.00
1.01

3e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128.27
127.97
126.34
125.77
125.49
124.92
124.10
89.65
77.48
77.23
76.98
44.36
32.93
29.07
Ph
O
O
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180
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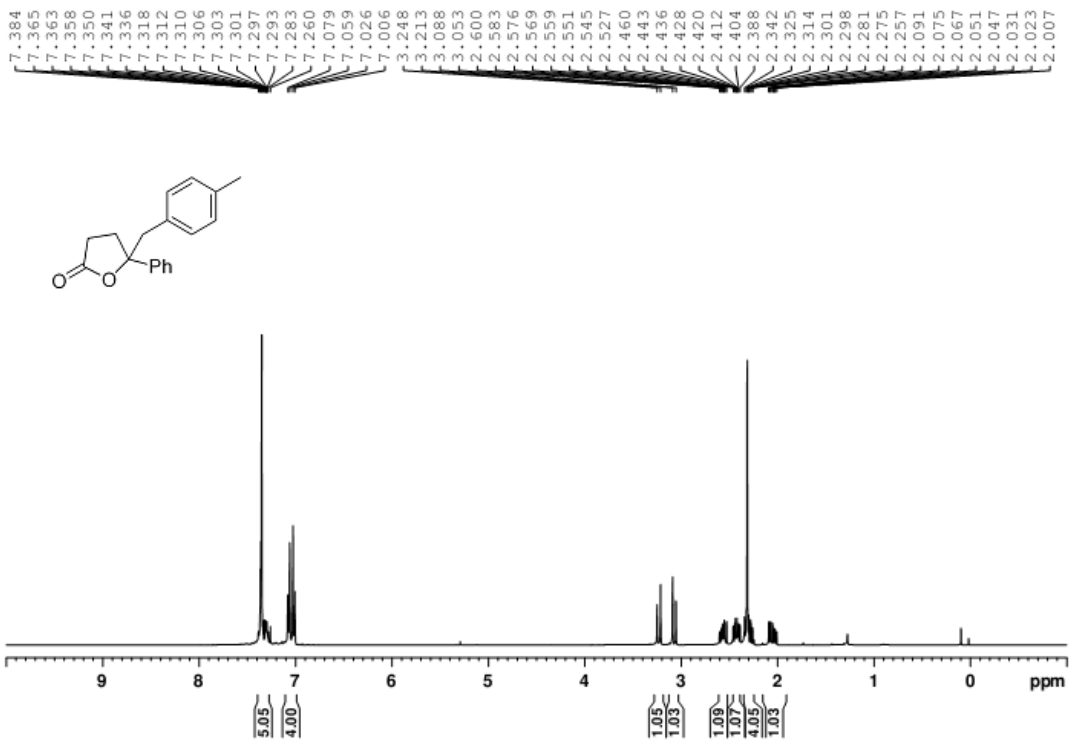
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2.412
2.404
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2.325
2.314
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2.275
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2.067
2.051
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2.031
2.023
2.007
Ph
O
O
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8
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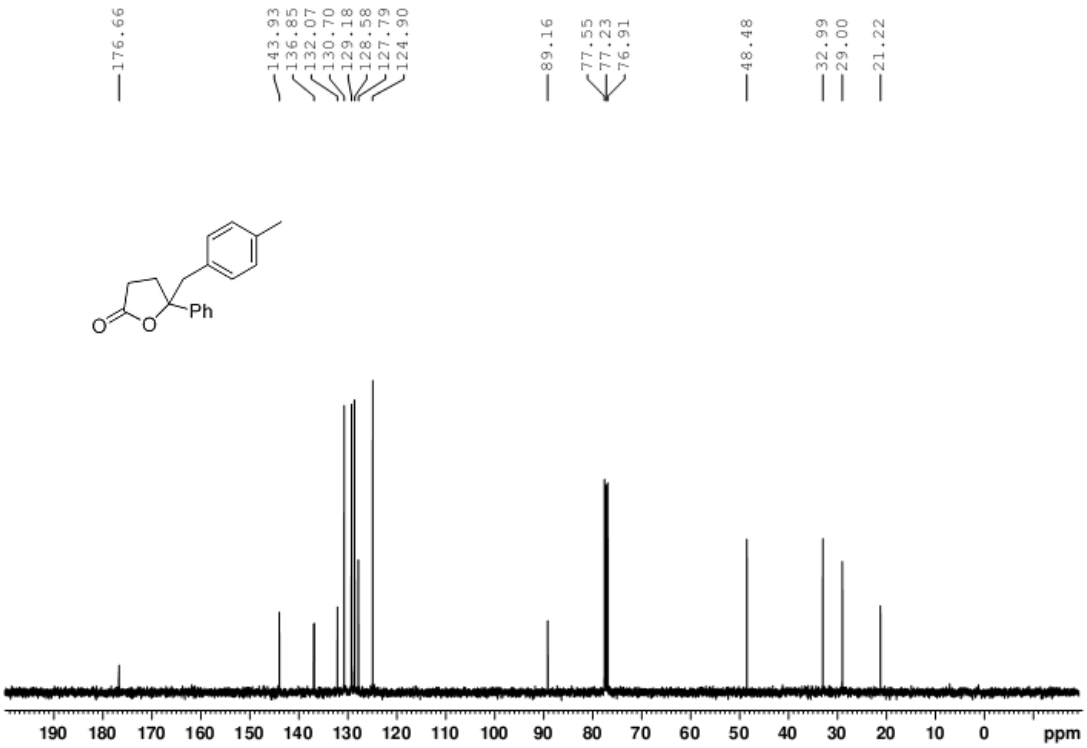
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32.99
29.00
21.22
Ph
O
O
190
180
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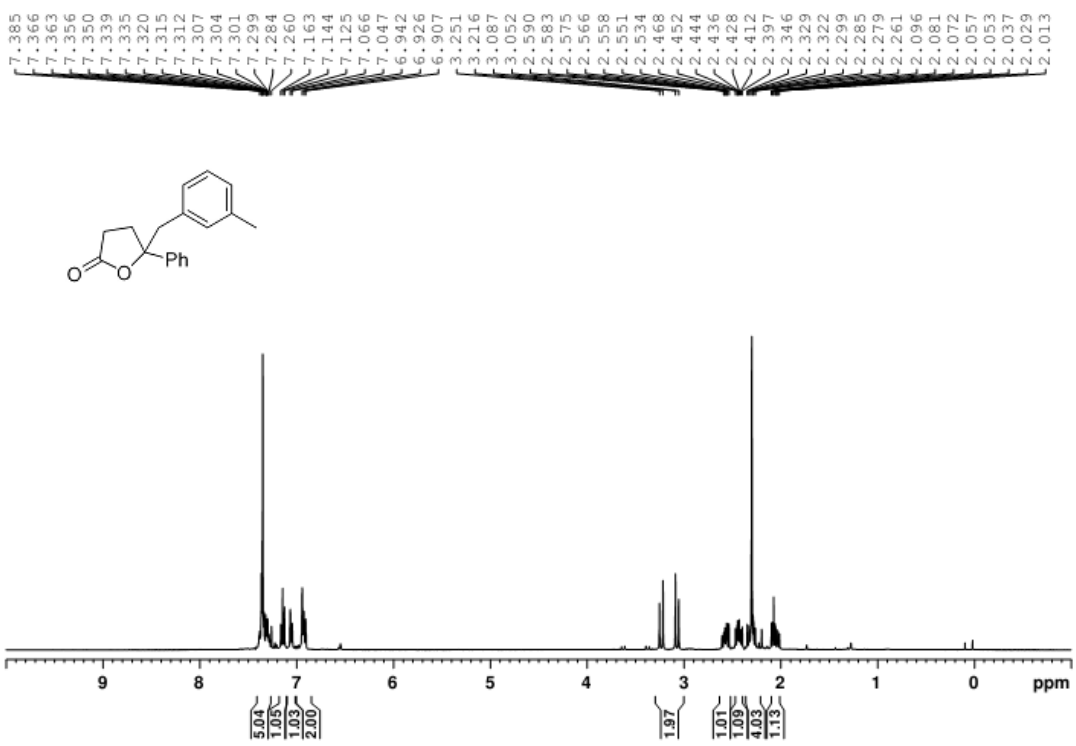
3g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2.037
2.029
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Ph
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O
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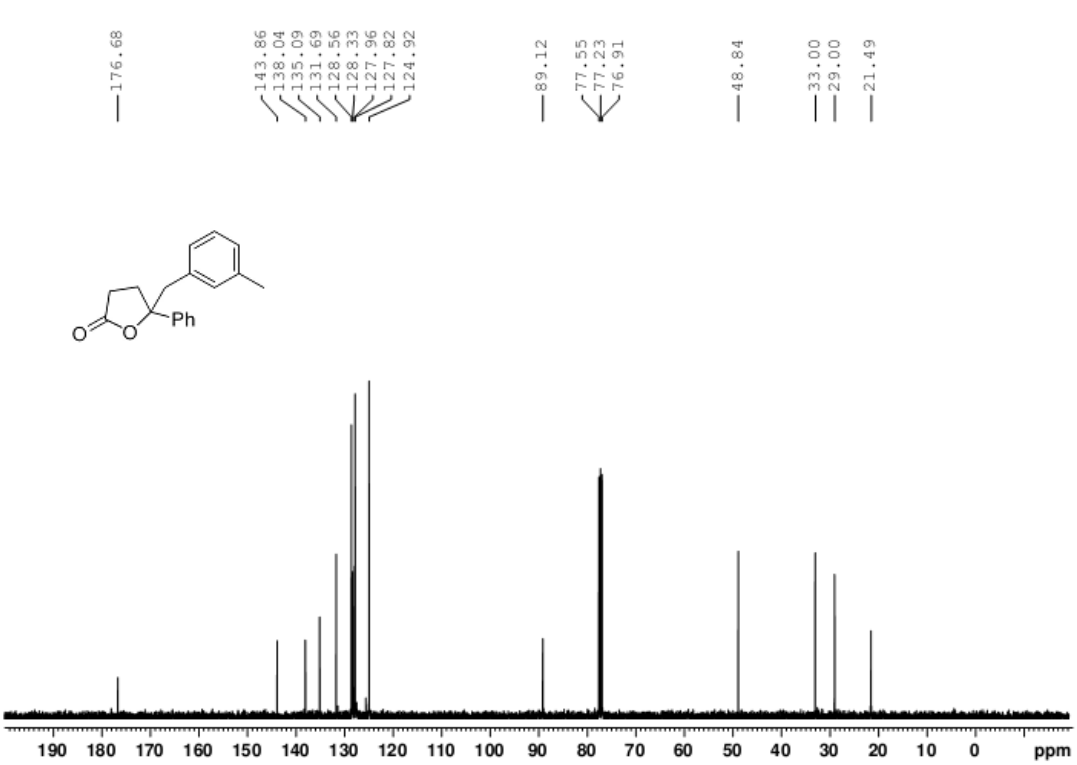
3g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29.00
21.49
Ph
O
O
190
180
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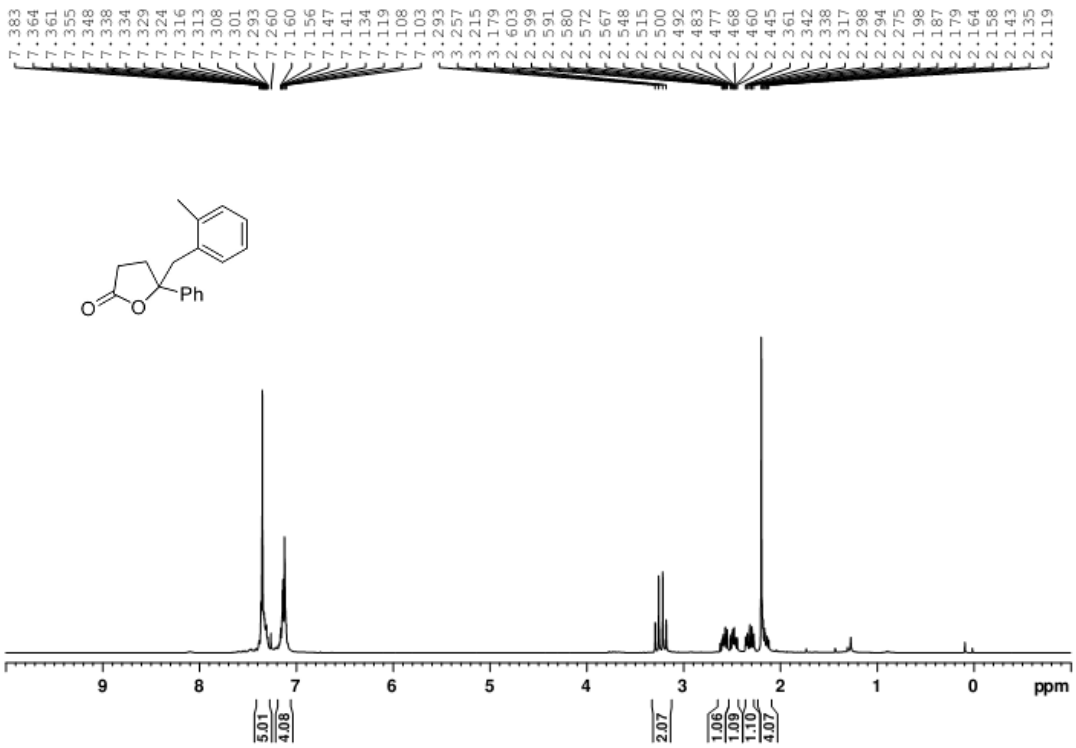
3h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2.572
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2.338
2.317
2.298
2.294
2.275
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2.179
2.164
2.158
2.143
2.135
2.119
Ph
O
O
9
8
7
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1.06
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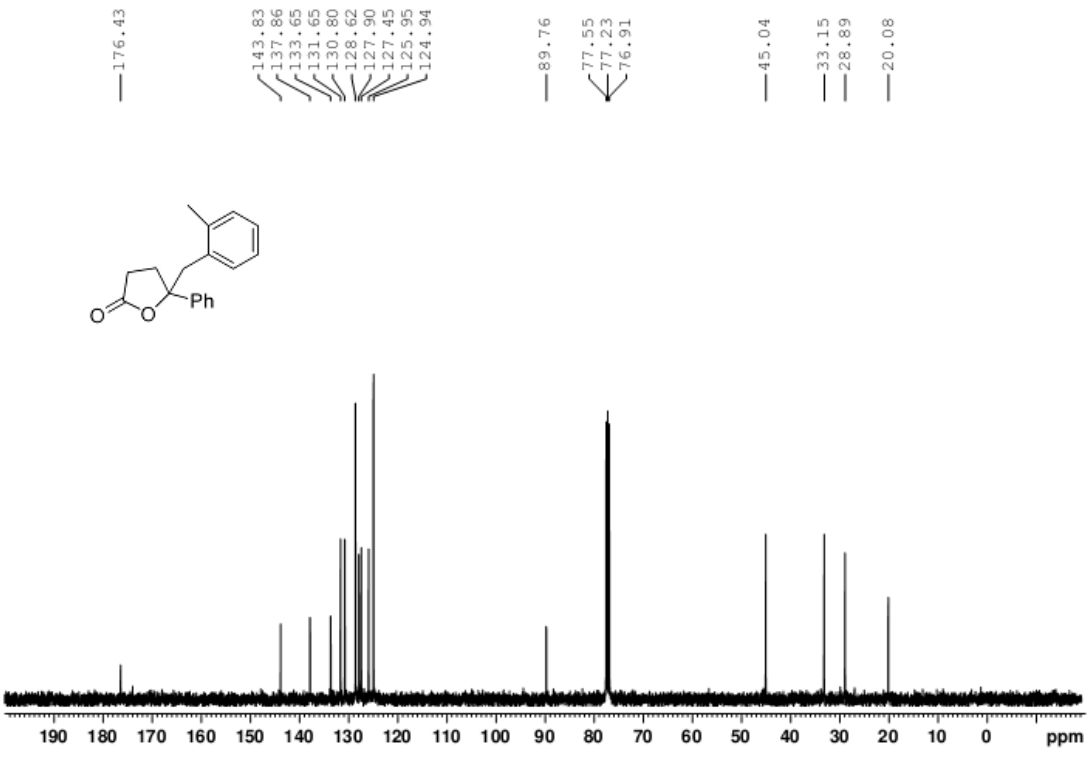
4.07

3h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77.23
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45.04
33.15
28.89
20.08
Ph
O
O
190
180
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10
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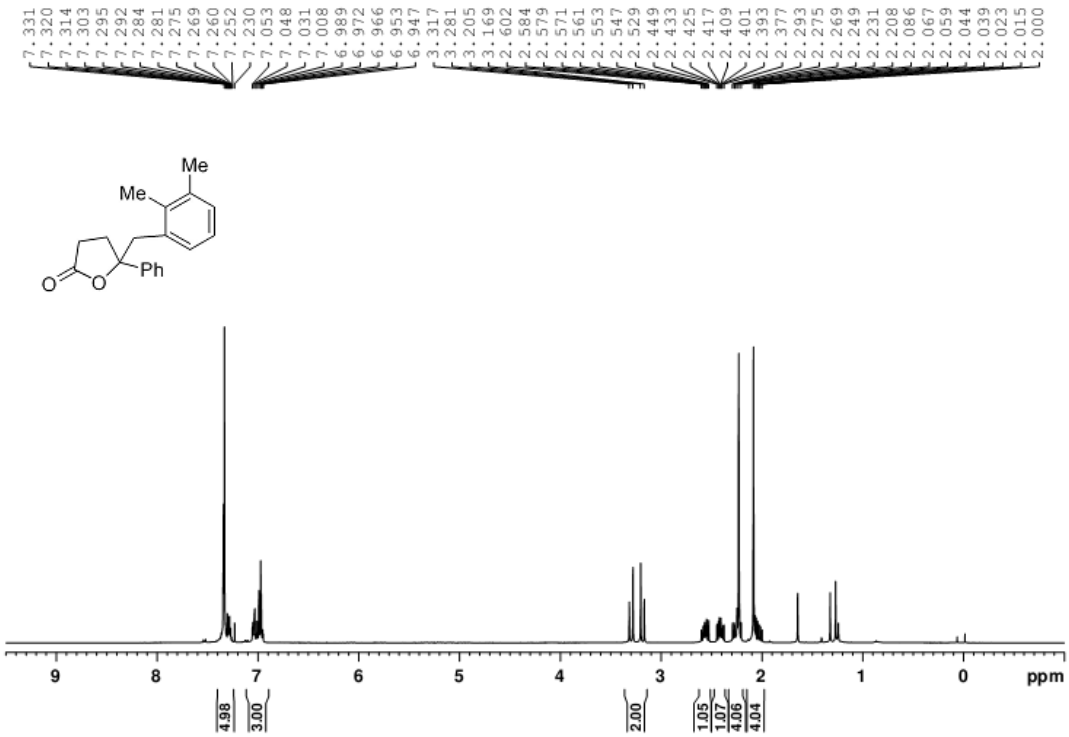
ppm

3i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7.230
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6.953
6.947
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3.205
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2.547
2.529
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2.433
2.425
2.417
2.409
2.401
2.393
2.377
2.293
2.275
2.269
2.249
2.231
2.208
2.086
2.067
2.059
2.044
2.039
2.023
2.015
2.000
Me
Me
Ph
O
O
9
8
7
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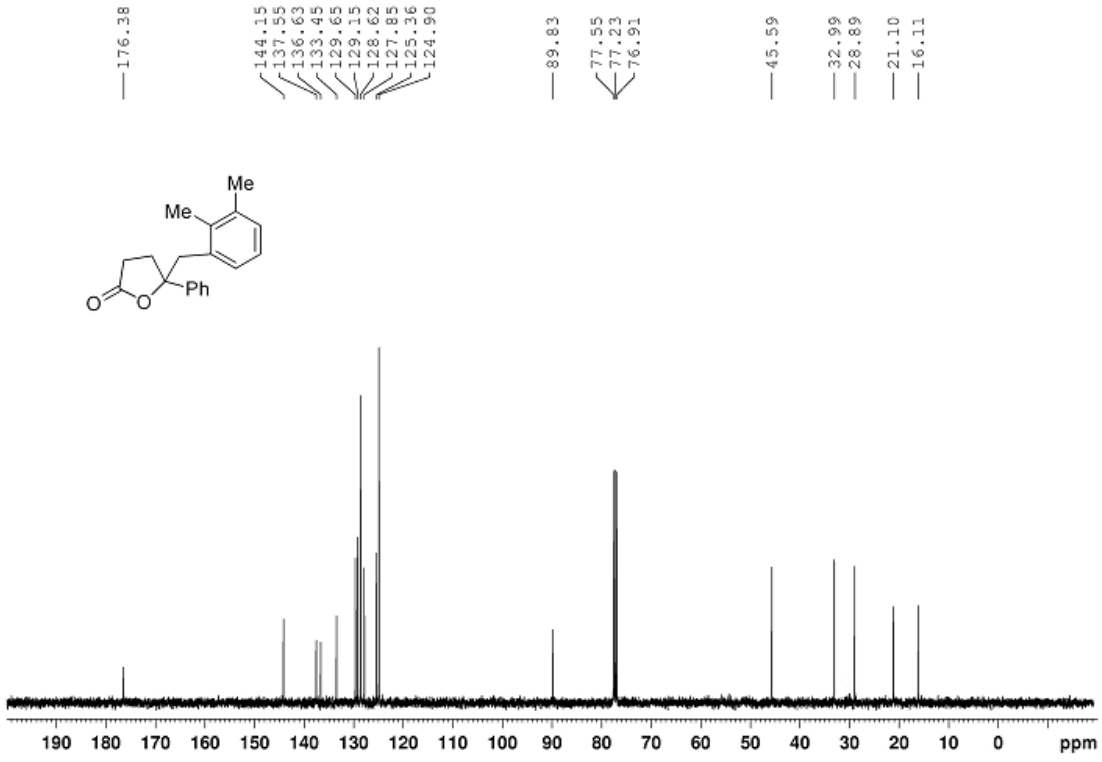
4.06
4.04

3i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77.23
76.91
45.59
32.99
28.89
21.10
16.11
Me
Me
Ph
O
O
190
180
170
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150
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130
120
110
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80
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50
40
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20
10
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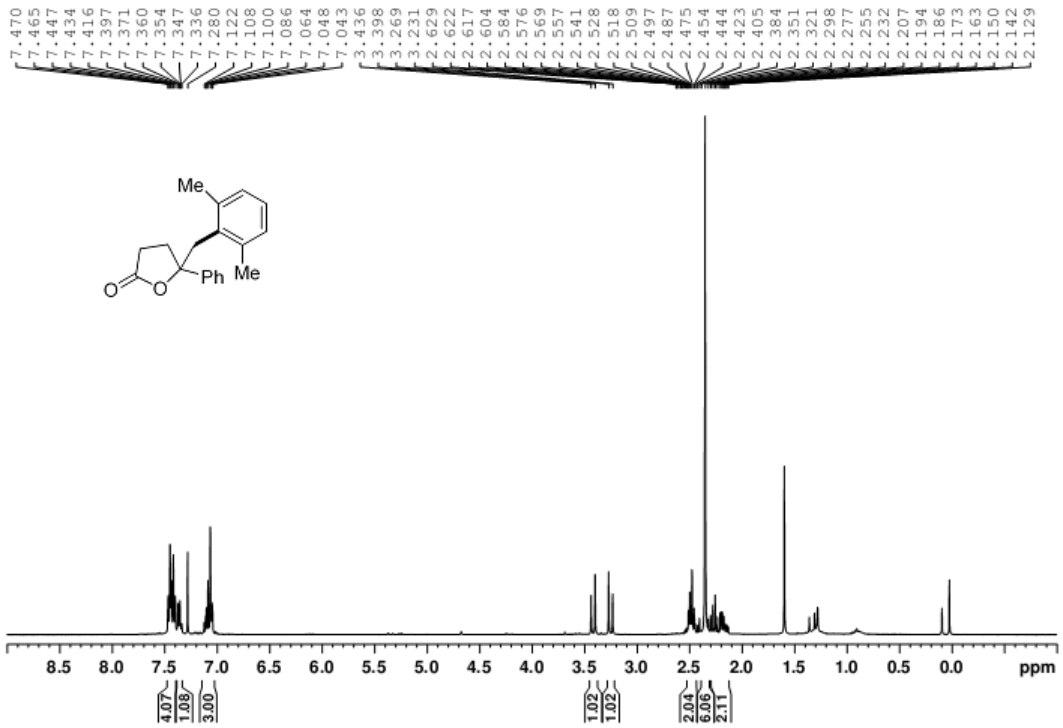
ppm

3j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7.108
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7.086
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7.048
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2.584
2.576
2.569
2.557
2.541
2.528
2.518
2.509
2.497
2.487
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2.454
2.444
2.423
2.405
2.384
2.351
2.321
2.298
2.277
2.255
2.232
2.207
2.194
2.186
2.173
2.163
2.150
2.142
2.129
Me
Ph
Me
O
O
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7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
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2.0
1.5
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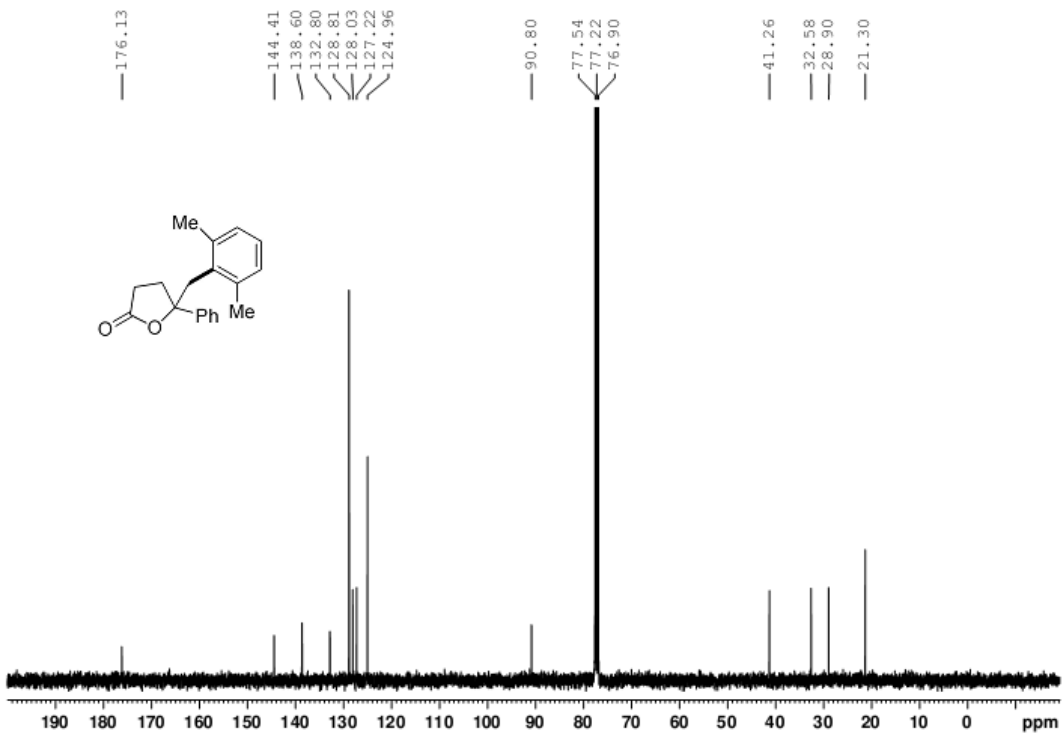
2.11

3j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32.58
28.90
21.30
Me
Ph
Me
O
O
190
180
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50
40
30
20
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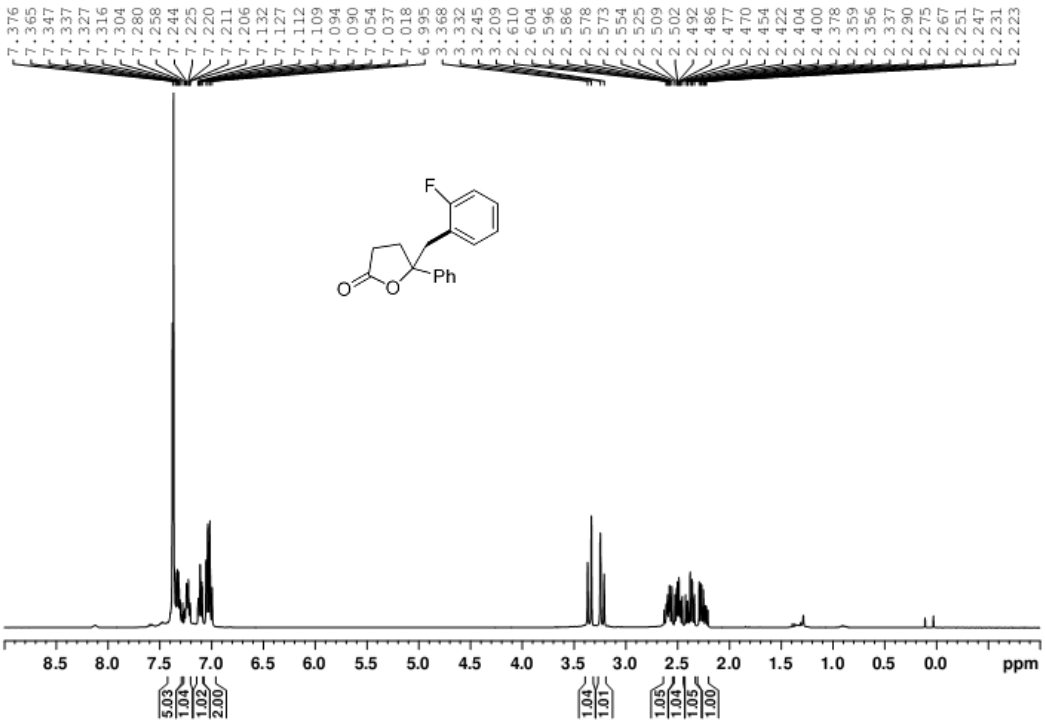
ppm

3k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7.206
7.132
7.127
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7.109
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7.090
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7.037
7.018
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2.604
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2.586
2.578
2.573
2.554
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2.509
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2.454
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2.337
2.290
2.275
2.267
2.251
2.247
2.231
2.223
F
Ph
O
O
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 ppm
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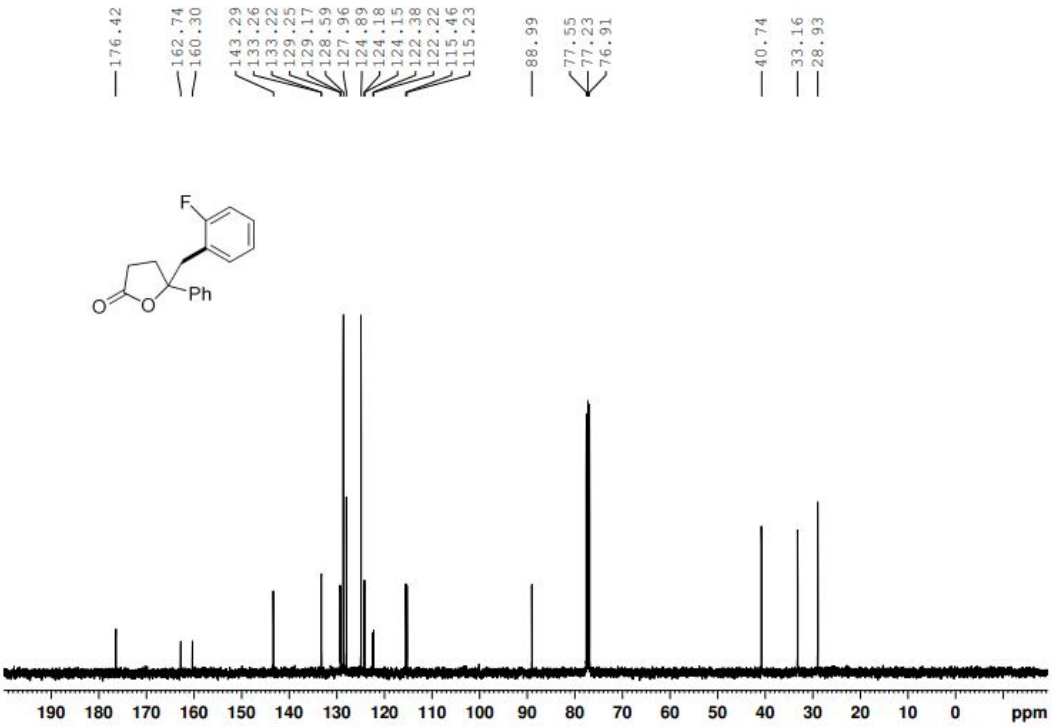
1.04
1.02
2.00
1.04
1.01
1.05
1.04
1.05
1.00

3k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122.38
122.22
115.46
115.23
88.99
77.55
77.23
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33.16
28.93
F
Ph
O
O
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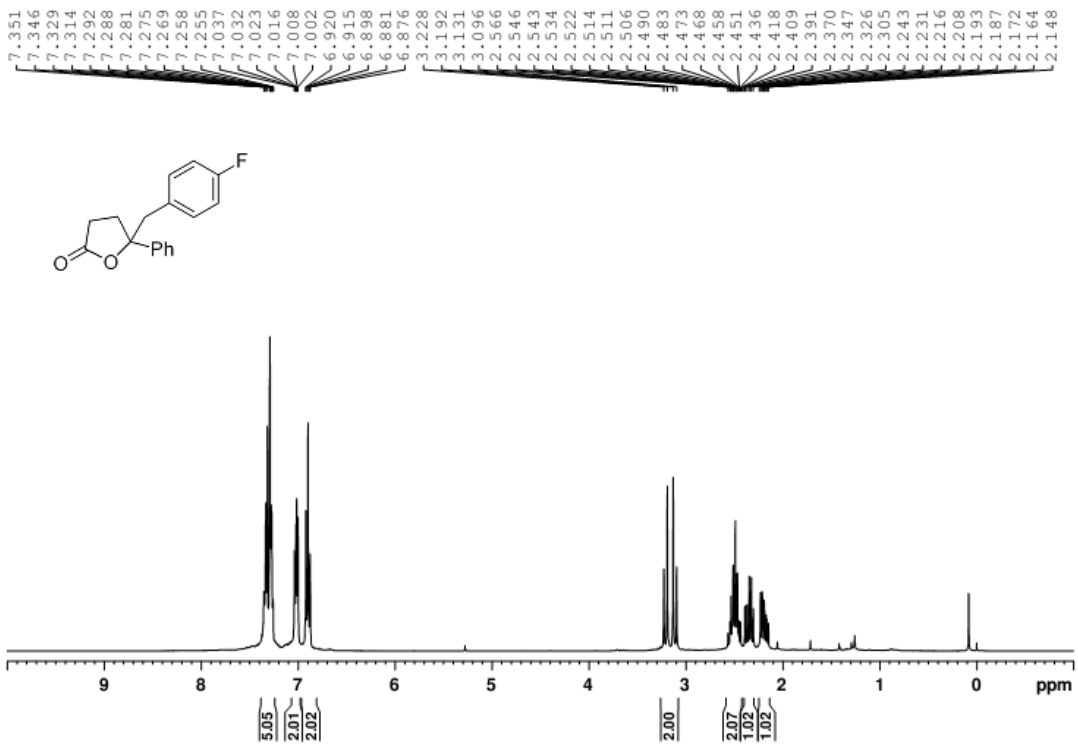
3l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7.258
7.255
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7.016
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2.370
2.347
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2.216
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2.187
2.172
2.164
2.148
F
Ph
O
O
9
8
7
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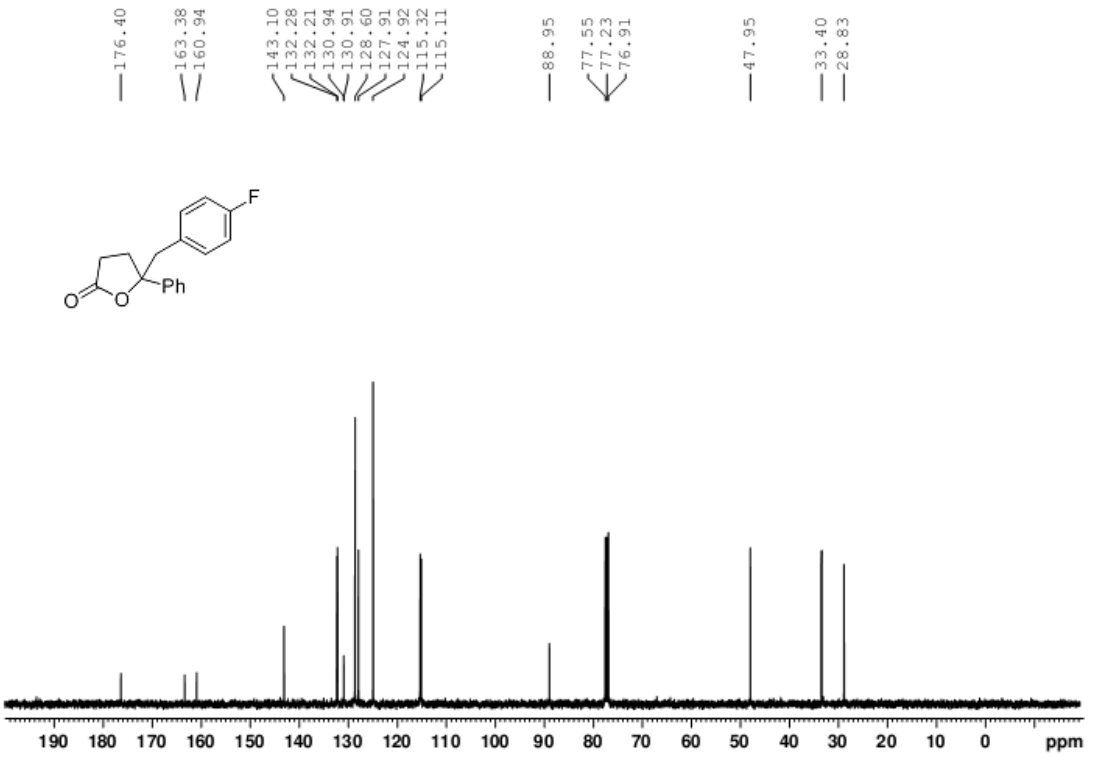
1.02

3l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115.11
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77.55
77.23
76.91
47.95
33.40
28.83
F
Ph
O
O
190
180
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130
120
110
100
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50
40
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20
10
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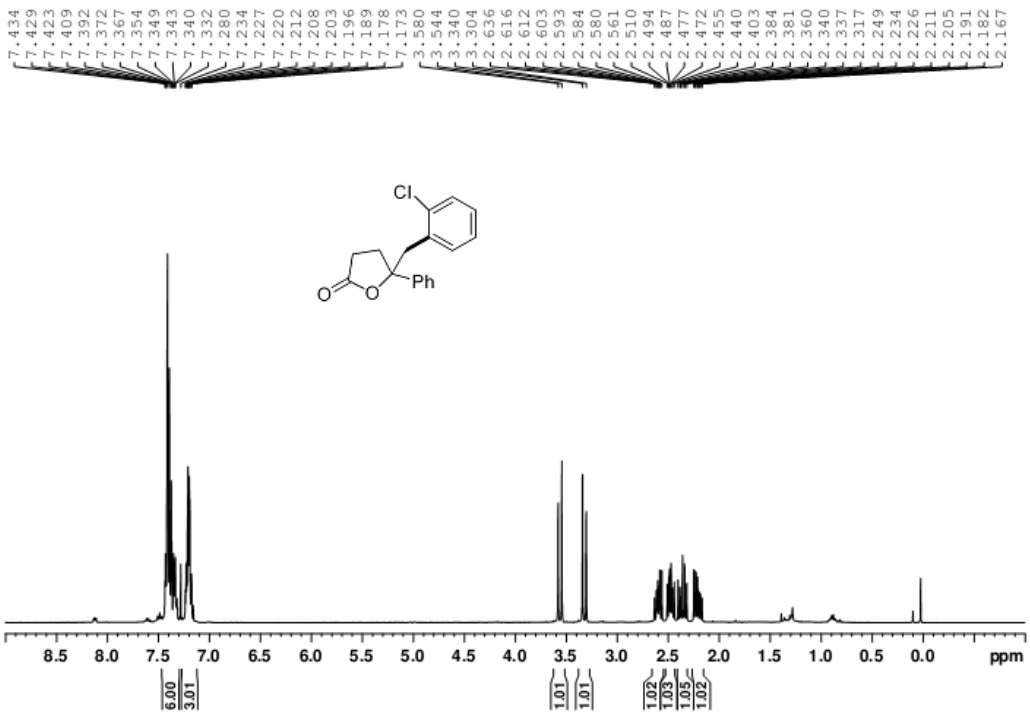
ppm

3m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7.220
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7.208
7.203
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2.603
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2.226
2.211
2.205
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2.182
2.167
Cl
O
O
Ph
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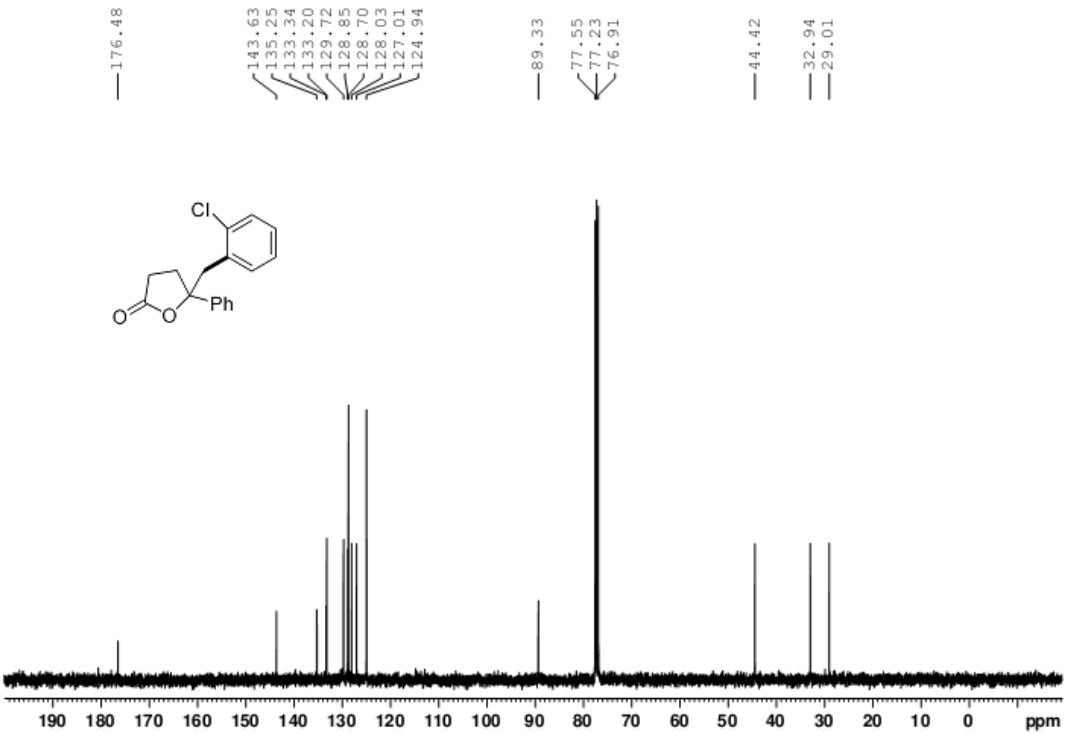
6.00
3.01
1.01
1.01
1.02
1.03
1.05
1.02

3m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128.70
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127.01
124.94
89.33
77.55
77.23
76.91
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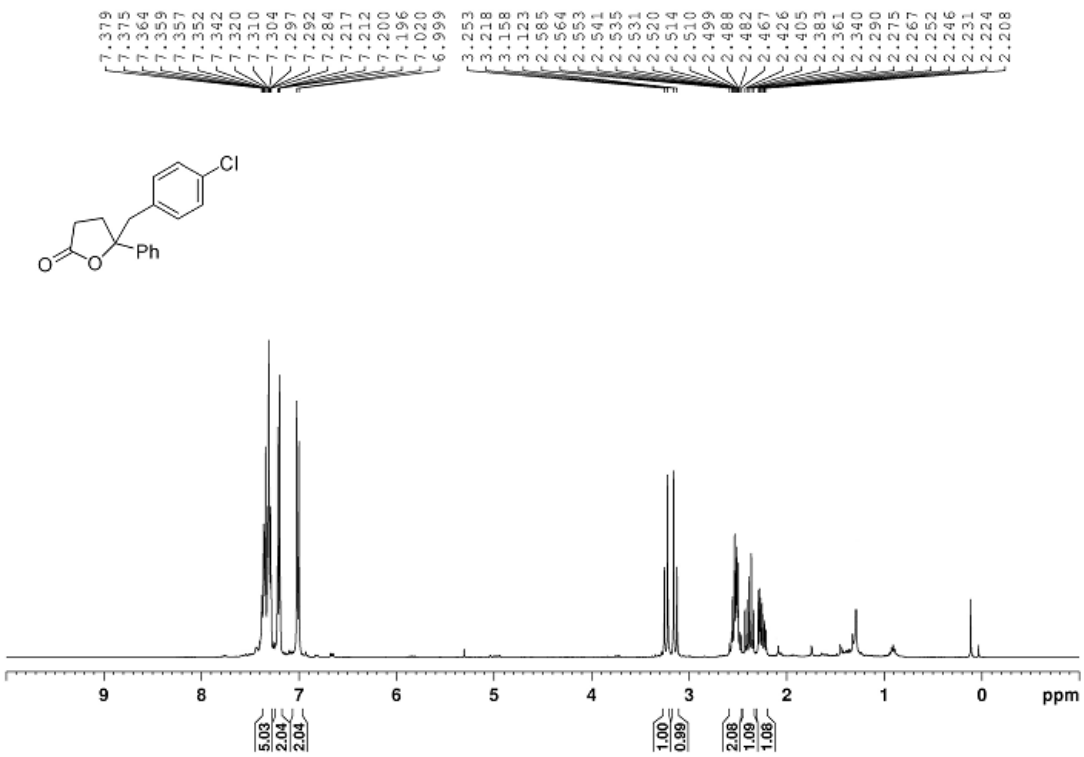
32.94
29.01
Cl
O
O
Ph
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

3n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7.200
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7.020
6.999
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3.218
3.158
3.123
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2.531
2.520
2.514
2.510
2.499
2.488
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2.426
2.405
2.383
2.361
2.340
2.290
2.275
2.267
2.252
2.246
2.231
2.224
2.208
Cl
Ph
O
O
9
8
7
6
5
4
3
2
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ppm
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0.99
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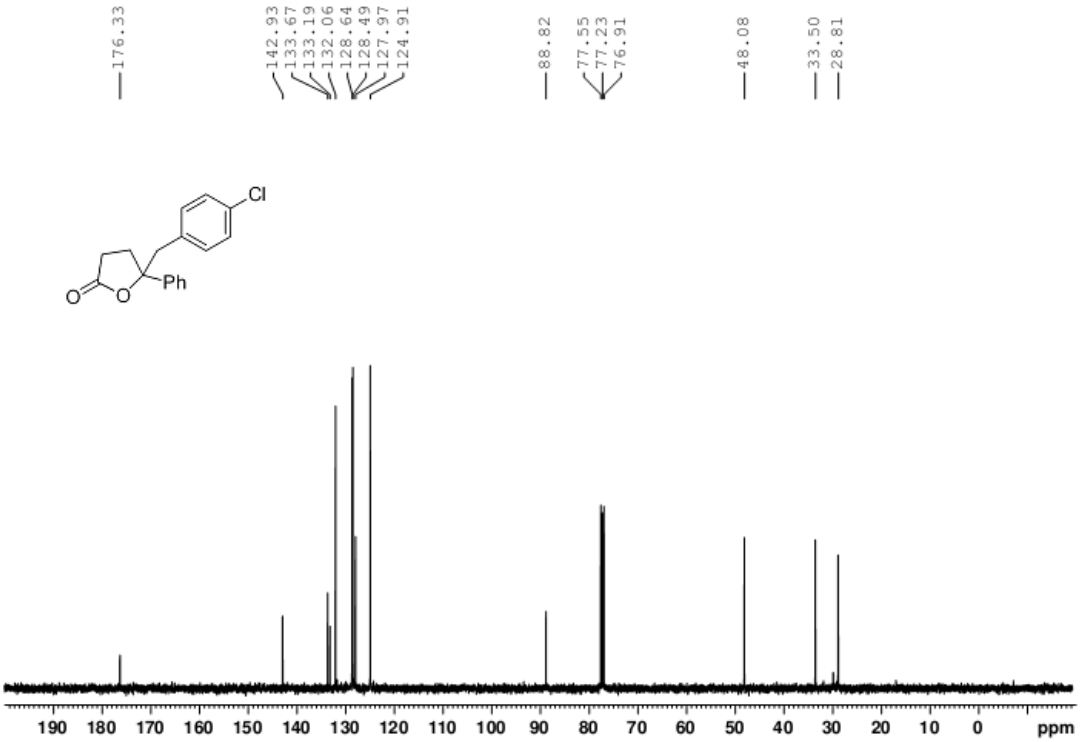
1.09
1.08

3n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88.82
77.55
77.23
76.91
48.08
33.50
28.81
Cl
Ph
O
O
190
180
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130
120
110
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90
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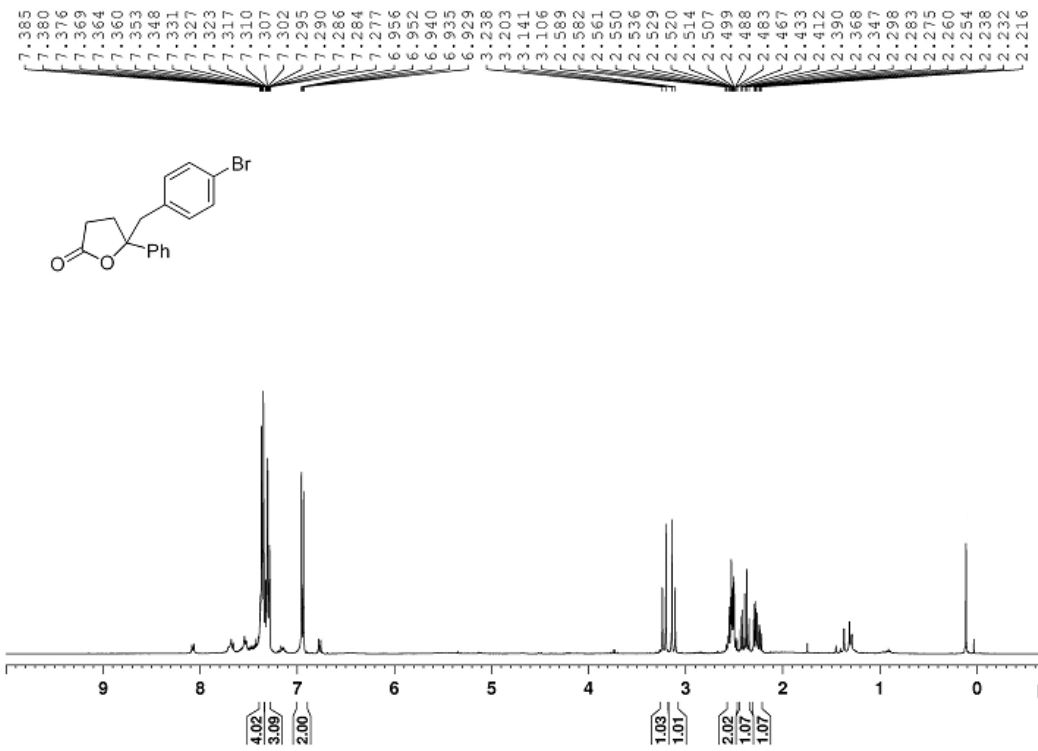
50
40
30
20
10
0
ppm

3o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6.940
6.935
6.929
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3.141
3.106
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2.582
2.561
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2.529
2.520
2.514
2.507
2.499
2.488
2.483
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2.433
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2.390
2.368
2.347
2.298
2.283
2.275
2.260
2.254
2.238
2.232
2.216
Br
Ph
O
O
9
8
7
6
5
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ppm
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1.01
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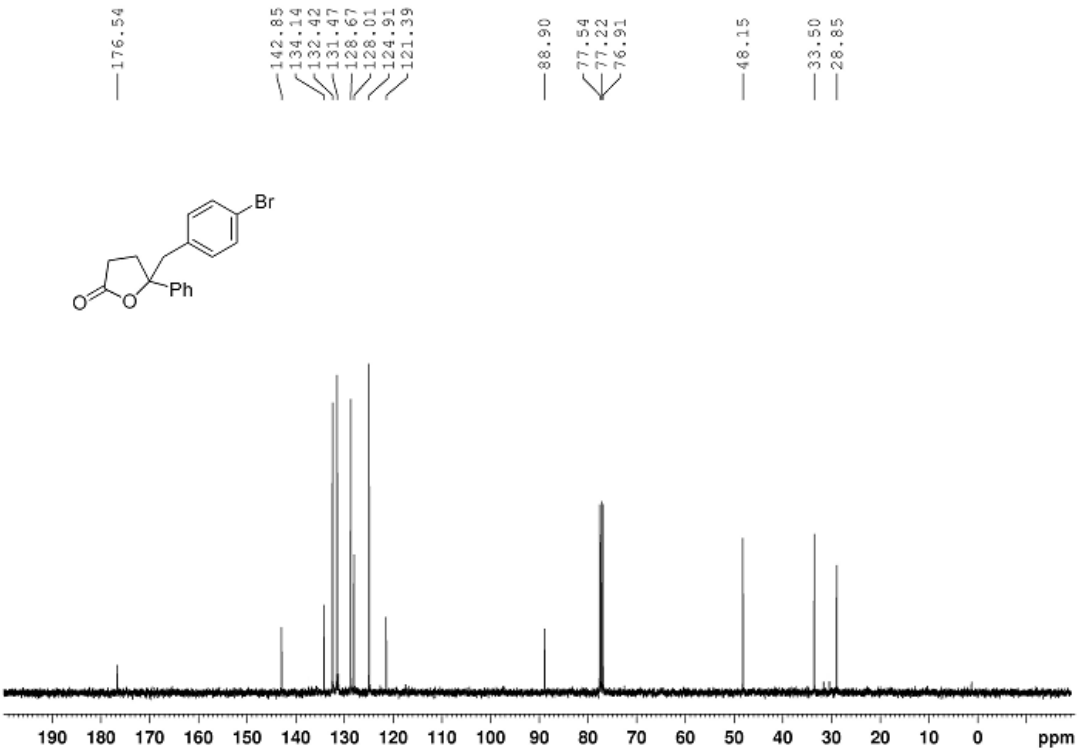
1.07
1.07

3o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77.54
77.22
76.91
48.15
33.50
28.85
Br
Ph
O
O
190
180
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150
140
130
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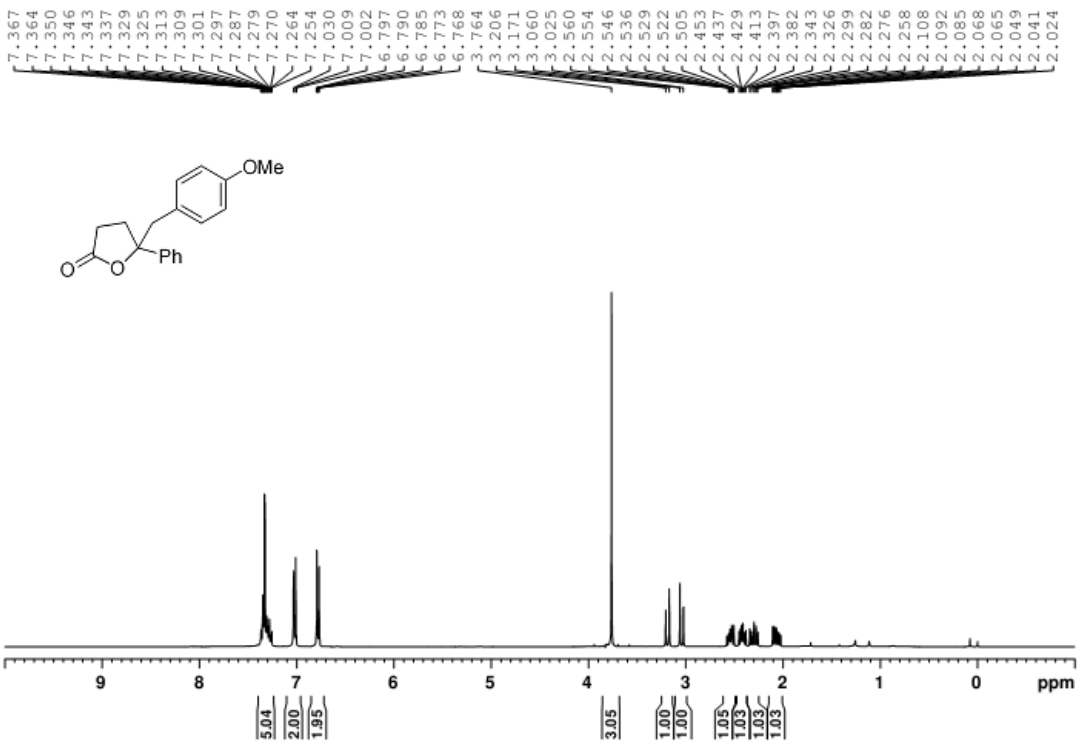
ppm

3p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7.343
7.337
7.329
7.325
7.313
7.309
7.301
7.297
7.287
7.279
7.270
7.264
7.254
7.030
7.009
7.002
6.797
6.790
6.785
6.773
6.768
3.764
3.206
3.171
3.060
3.025
2.560
2.554
2.546
2.536
2.529
2.522
2.505
2.453
2.437
2.429
2.413
2.397
2.382
2.343
2.326
2.299
2.282
2.276
2.258
2.108
2.092
2.085
2.068
2.065
2.049
2.041
2.024
OMe
Ph
O
O
9
8
7
6
5
4
3
2
1
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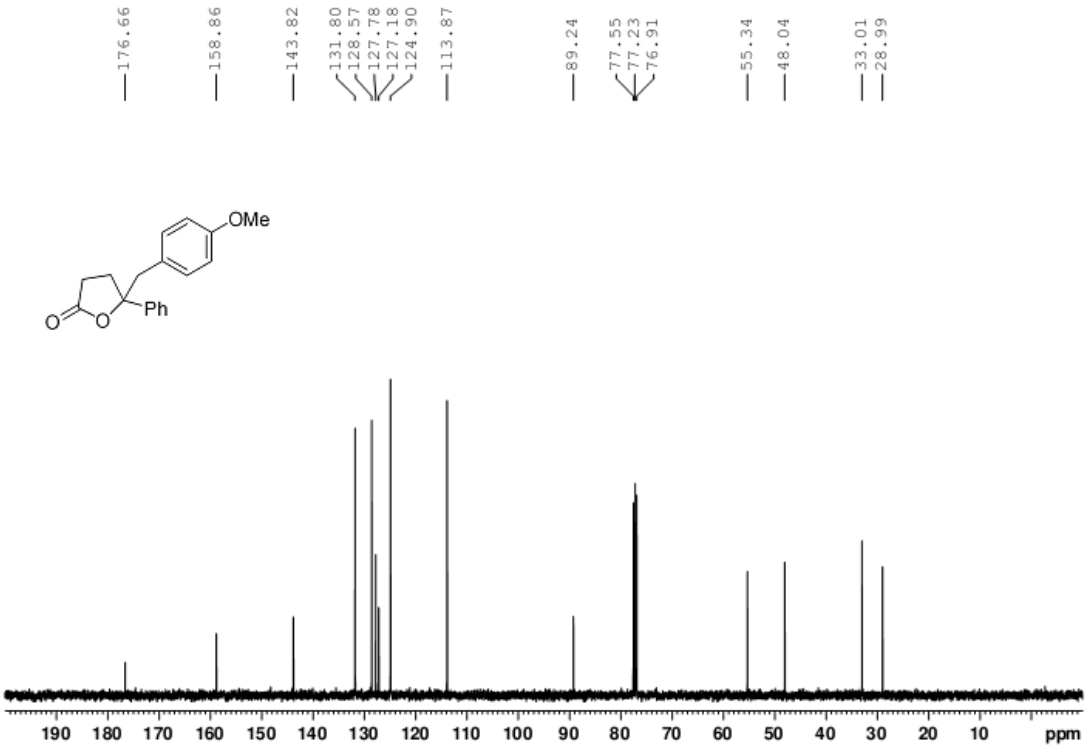
ppm
5.04
2.00
1.95
3.05
1.00
1.00
1.05
1.03
1.03
1.03

3p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158.86
143.82
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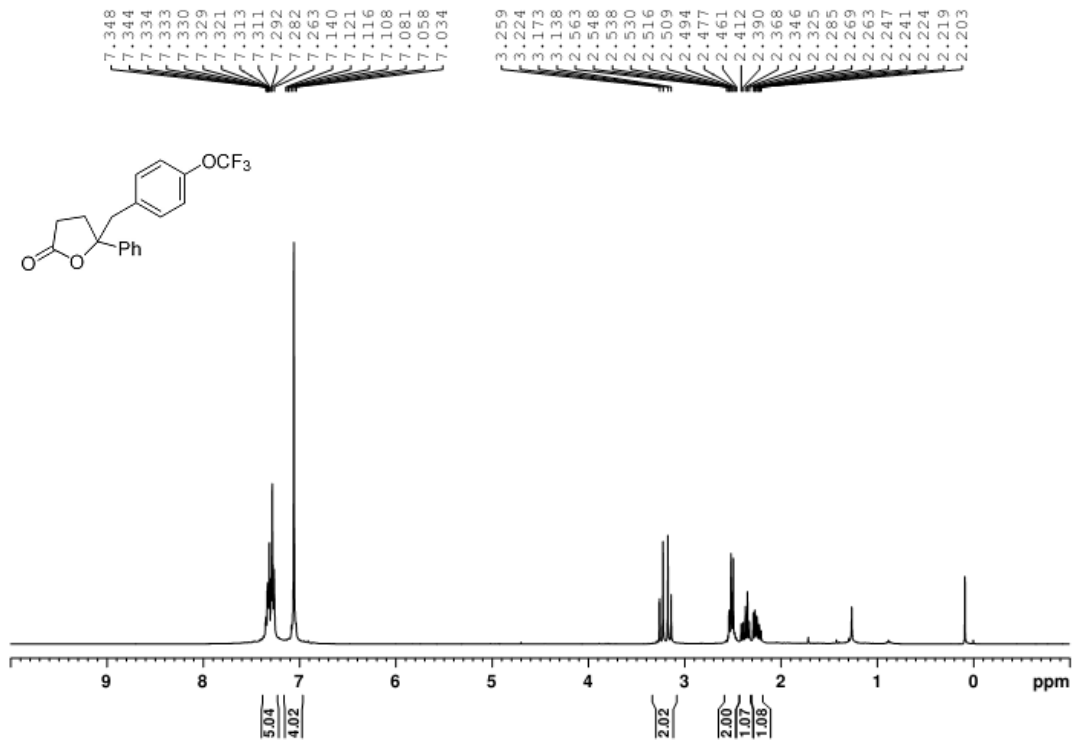
127.78
127.18
124.90
113.87
89.24
77.55
77.23
76.91
55.34
48.04
33.01
28.99
OMe
Ph
O
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

3q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7.313
7.311
7.292
7.282
7.263
7.140
7.121
7.116
7.108
7.081
7.058
7.034
3.259
3.224
3.173
3.138
2.563
2.548
2.538
2.530
2.516
2.509
2.494
2.477
2.461
2.412
2.390
2.368
2.346
2.325
2.285
2.269
2.263
2.247
2.241
2.224
2.219
2.203
OCF3
Ph
O
O
9
8
7
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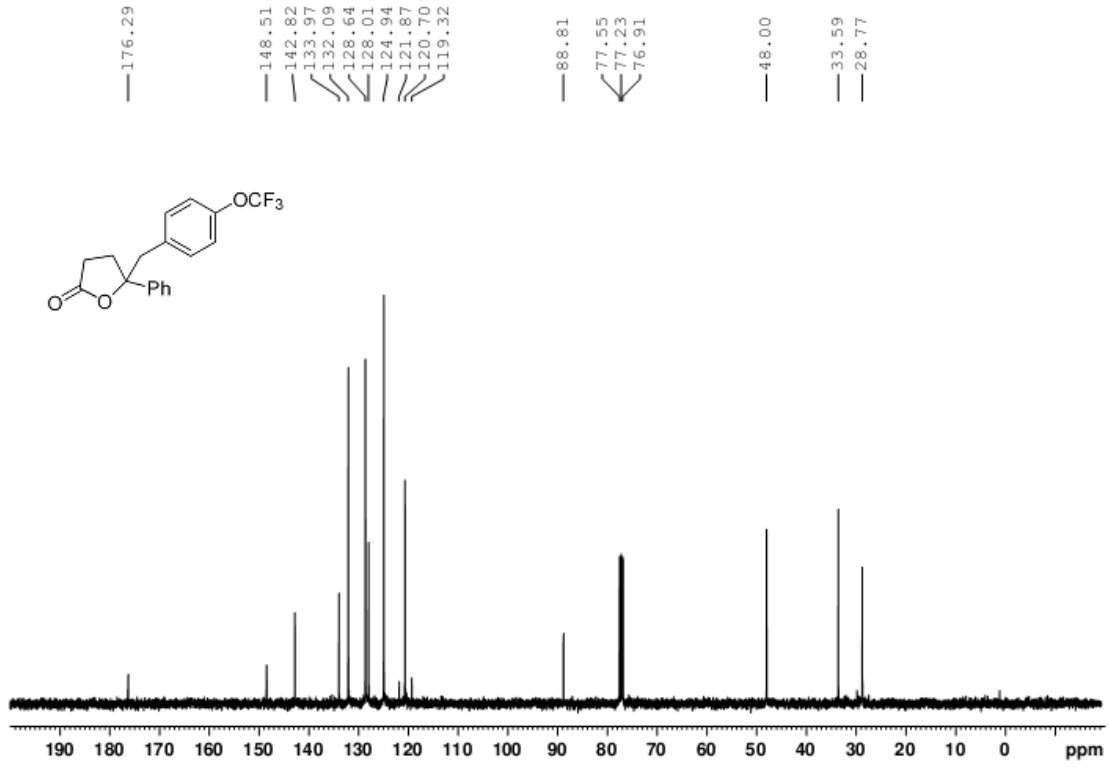
5
4
3
2
1
0
ppm
5.04
4.02
2.02
2.00
1.07
1.08

3q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132.09
128.64
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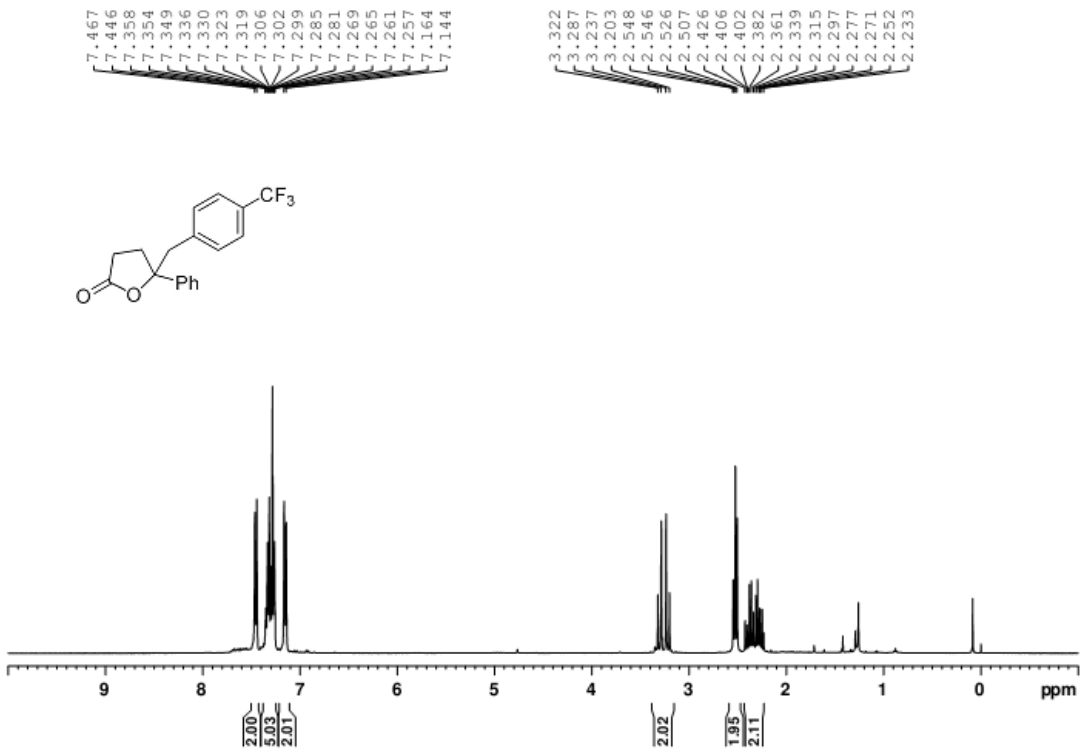
124.94
121.87
120.70
119.32
88.81
77.55
77.23
76.91
48.00
33.59
28.77
OCF3
Ph
O
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3r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7.323
7.319
7.306
7.302
7.299
7.285
7.281
7.269
7.265
7.261
7.257
7.164
7.144
3.322
3.287
3.237
3.203
2.548
2.546
2.526
2.507
2.426
2.406
2.402
2.382
2.361
2.339
2.315
2.297
2.277
2.271
2.252
2.233
CF3
Ph
O
O
9
8
7
6
5
4
3
2
1
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ppm
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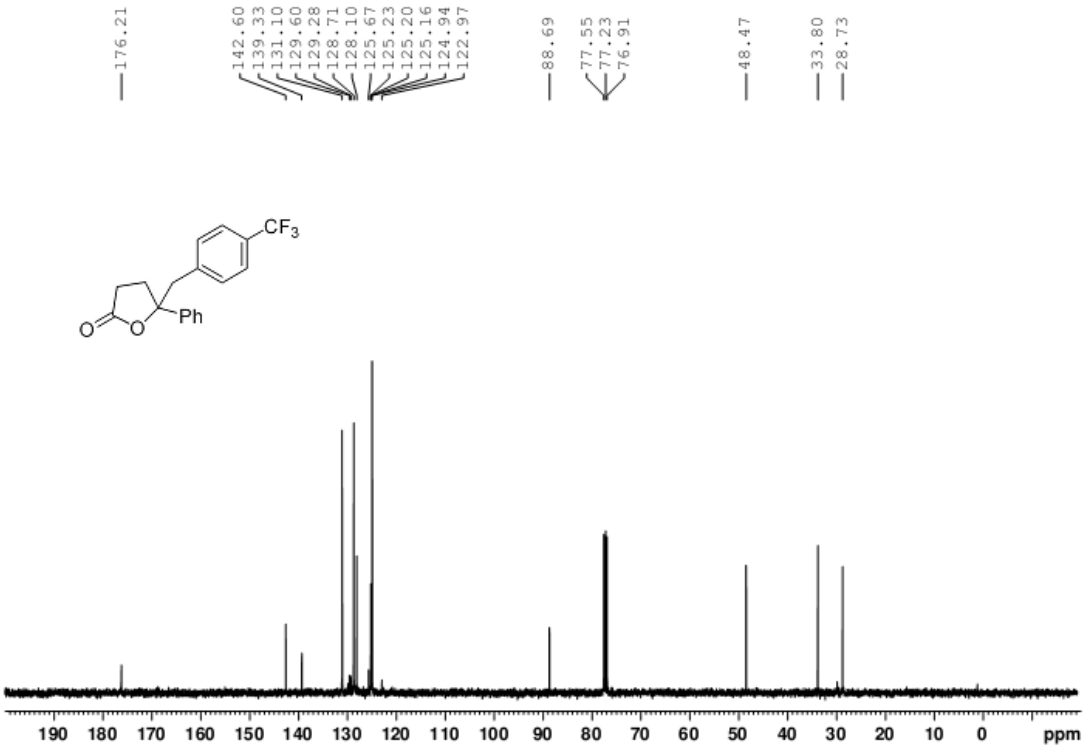
5.03
2.01
2.02
1.95
2.11

3r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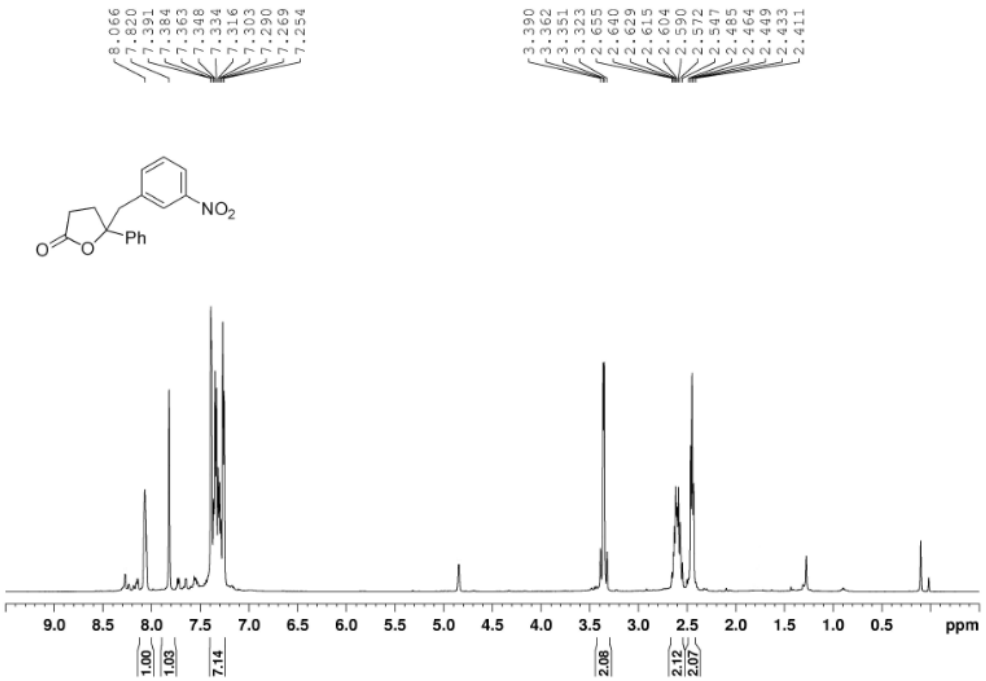
142.60
139.33
131.10
129.60
129.28
128.71
128.10
125.67
125.23
125.20
125.16
124.94
122.97
88.69
77.55
77.23
76.91
48.47
33.80
28.73
CF3
Ph
O
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3s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7.820
7.391
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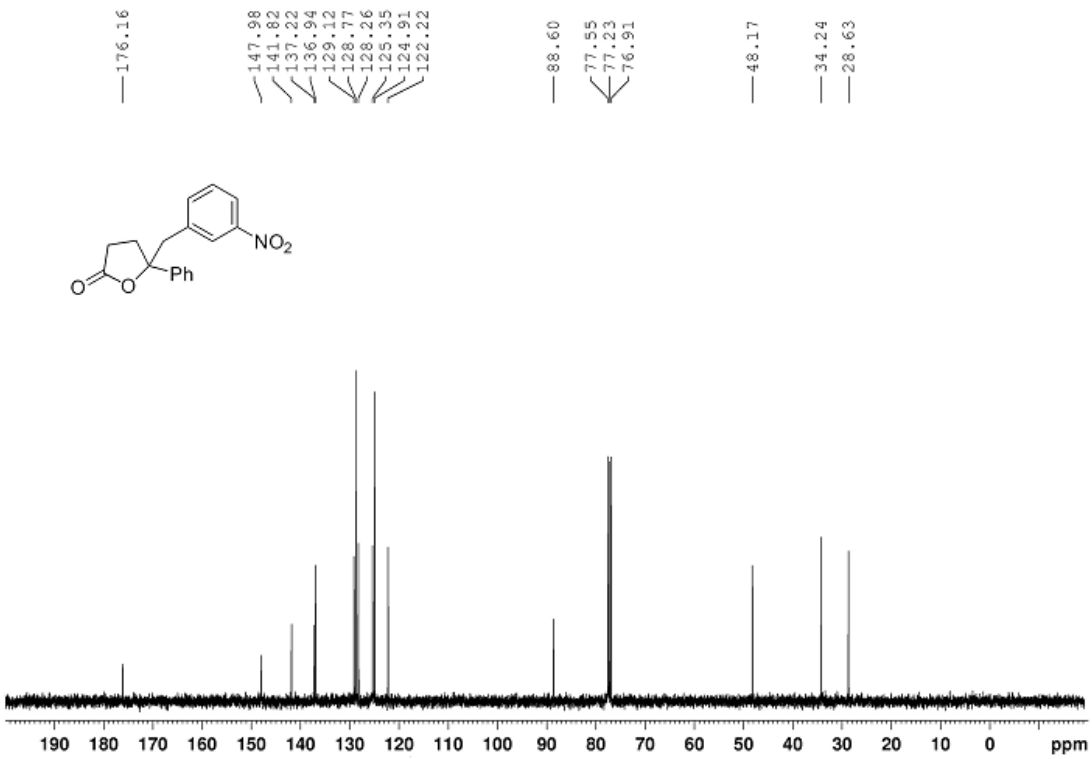
7.363
7.348
7.334
7.316
7.303
7.290
7.269
7.254
3.390
3.362
3.351
3.323
2.655
2.640
2.629
2.615
2.604
2.590
2.572
2.547
2.485
2.464
2.449
2.433
2.411
NO2
Ph
O
O
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.03
7.14
2.08
2.12
2.07

3s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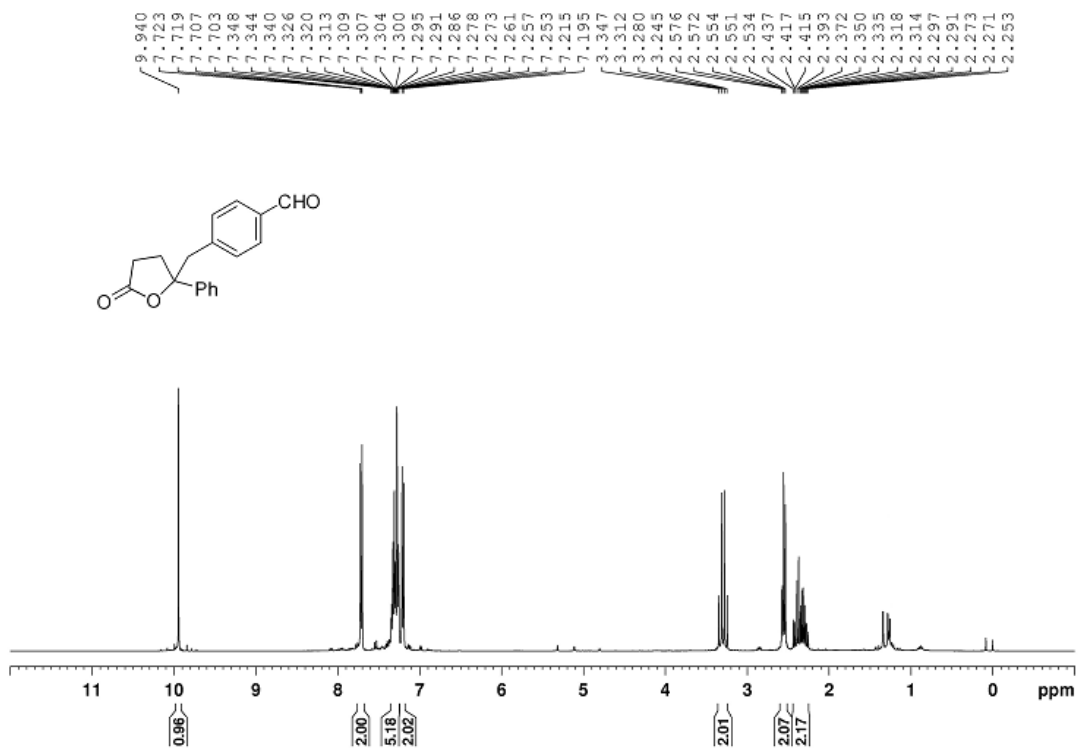
147.98
141.82
137.22
136.94
129.12
128.77
128.26
125.35
124.91
122.22
88.60
77.55
77.23
76.91
48.17
34.24
28.63
NO2
Ph
O
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

3t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7.723
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7.703
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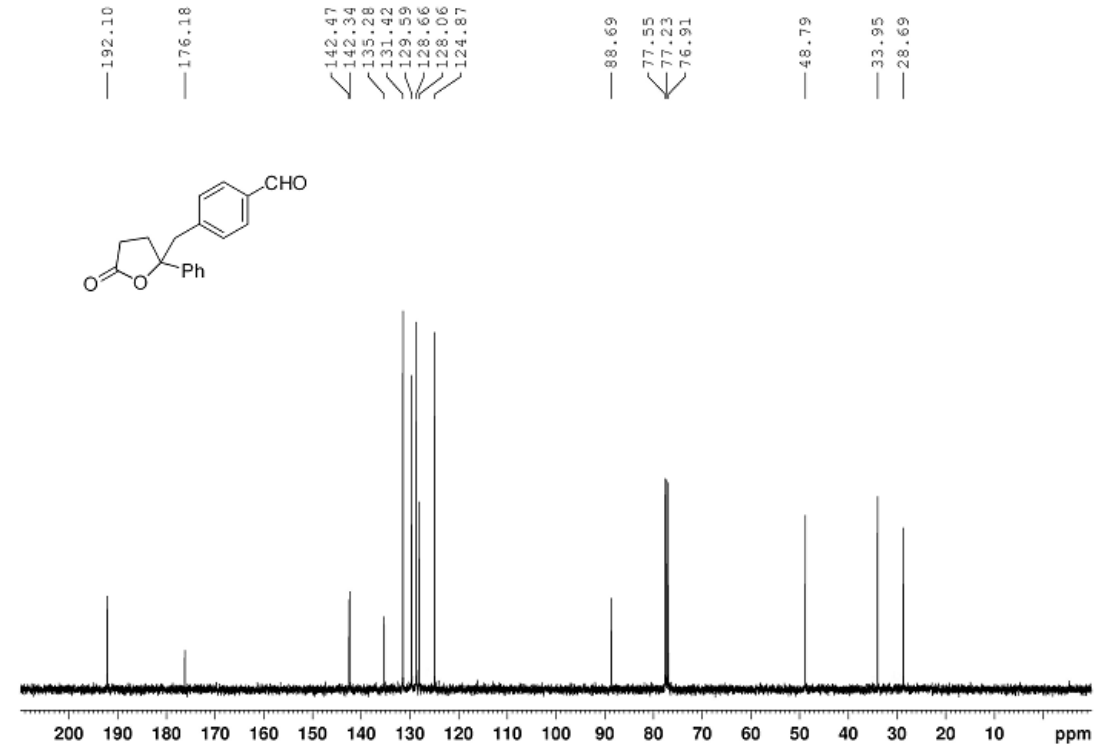
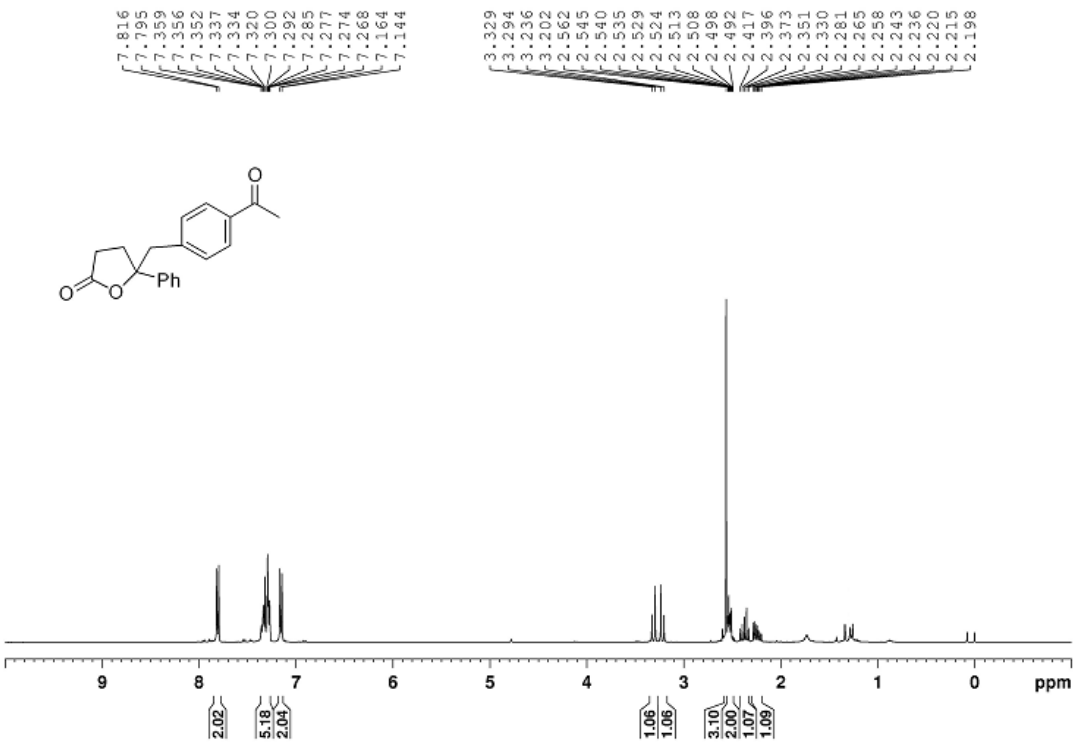
7.344
7.340
7.326
7.320
7.313
7.309
7.307
7.304
7.300
7.295
7.291
7.286
7.278
7.273
7.261
7.257
7.253
7.215
7.195
3.347
3.312
3.280
3.245
2.576
2.572
2.554
2.551
2.534
2.437
2.417
2.415
2.393
2.372
2.350
2.335
2.318
2.314
2.297
2.291
2.273
2.271
2.253
CHO
Ph
O
O
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.96
2.00
5.18
2.02
2.01
2.07
2.17

3t


192.10
176.18
142.47
142.34
135.28
131.42
129.59
128.66
128.06
124.87
88.69
77.55
77.23
76.91
48.79
33.95
28.69
CHO
Ph
O
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

3u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7.795
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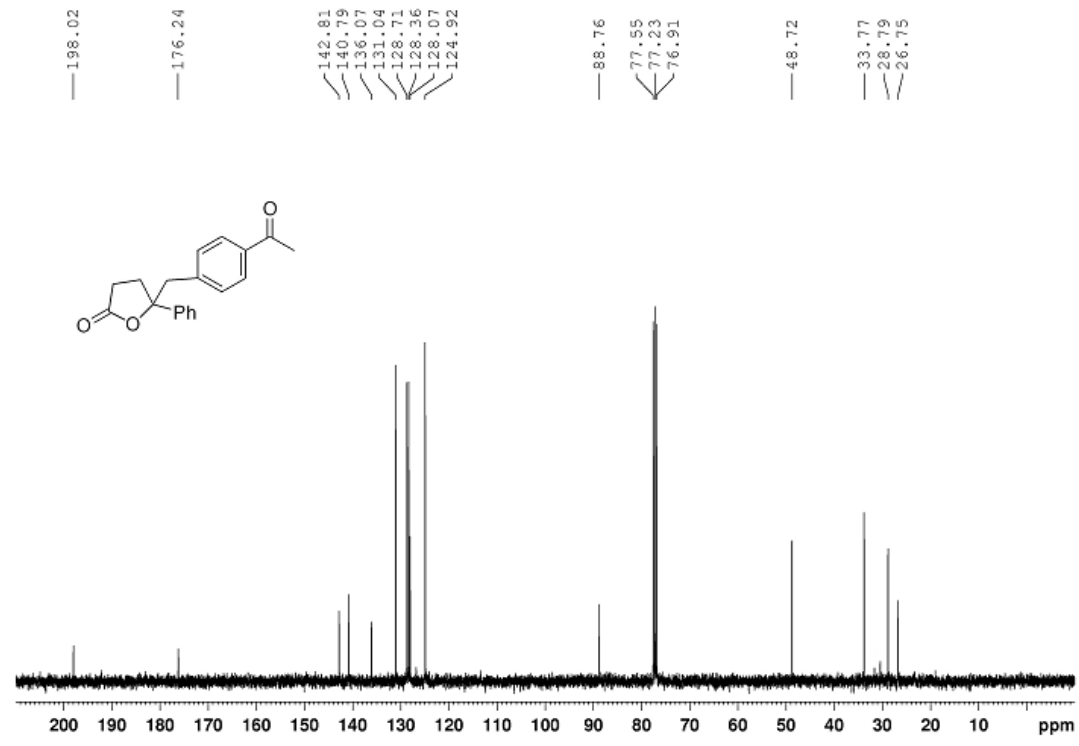
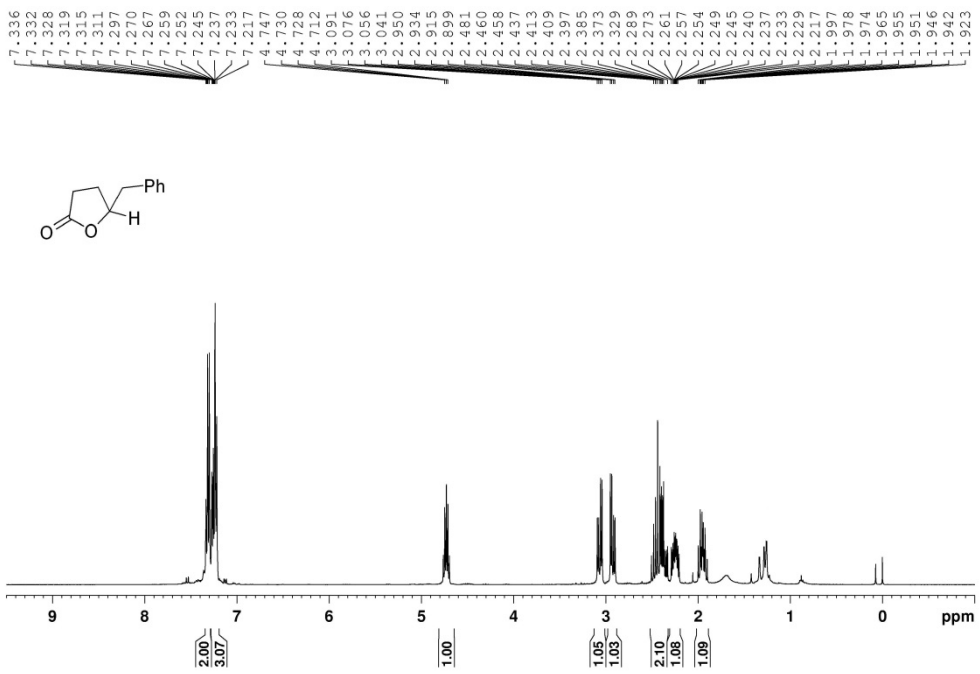
7.337
7.334
7.320
7.300
7.292
7.285
7.277
7.274
7.268
7.164
7.144
3.329
3.294
3.236
3.202
2.562
2.545
2.540
2.535
2.529
2.524
2.513
2.508
2.498
2.492
2.417
2.396
2.373
2.351
2.330
2.281
2.265
2.258
2.243
2.236
2.220
2.215
2.198
O
O
O
Ph
9
8
7
6
5
4
3
2
1
0
ppm
2.02
5.18
2.04
1.06
1.06
3.10
2.00
1.07
1.09

3u


198.02
176.24
142.81
140.79
136.07
131.04
128.71
128.36
128.07
124.92
88.76
77.55
77.23
76.91
48.72
33.77
28.79
26.75
O
O
O
Ph
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

3v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7.332
7.328
7.319
7.315
7.311
7.297
7.270
7.267
7.259
7.252
7.245
7.237
7.233
7.217
4.747
4.730
4.728
4.712
3.091
3.076
3.056
3.041
2.950
2.934
2.915
2.899
2.481
2.460
2.458
2.437
2.413
2.409
2.397
2.385
2.373
2.329
2.289
2.273
2.261
2.257
2.254
2.249
2.245
2.240
2.237
2.233
2.229
2.217
1.997
1.978
1.974
1.965
1.955
1.951
1.946
1.942
1.923
Ph
H
O
O
9
8
7
6
5
4
3
2
1
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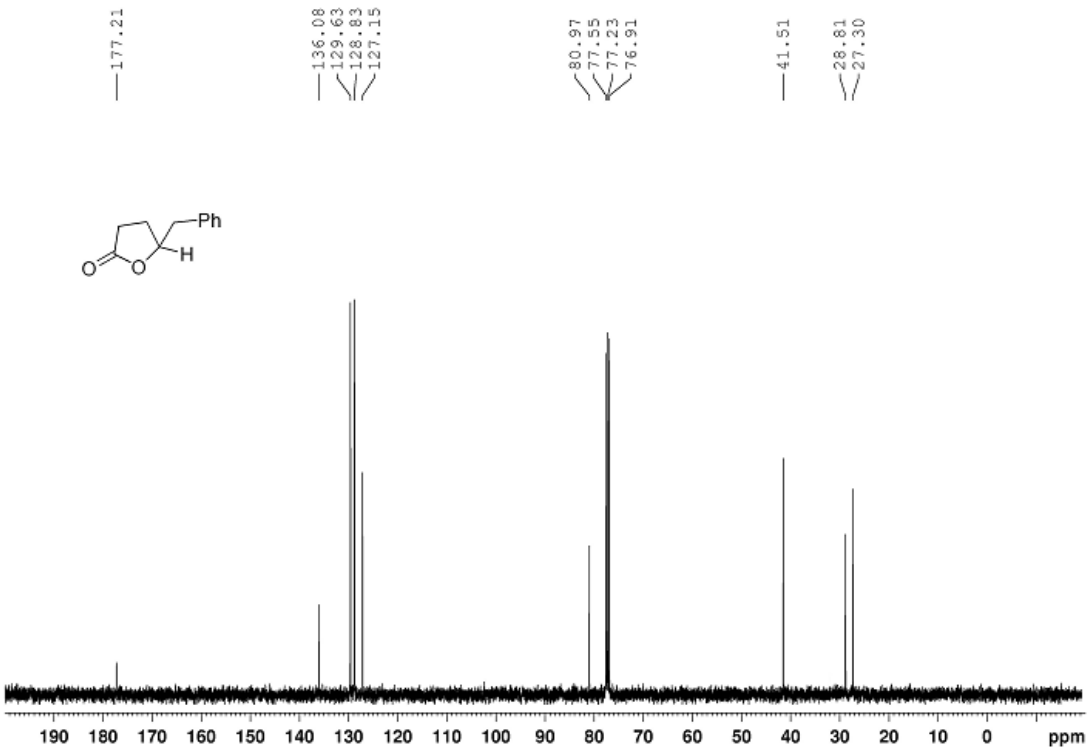
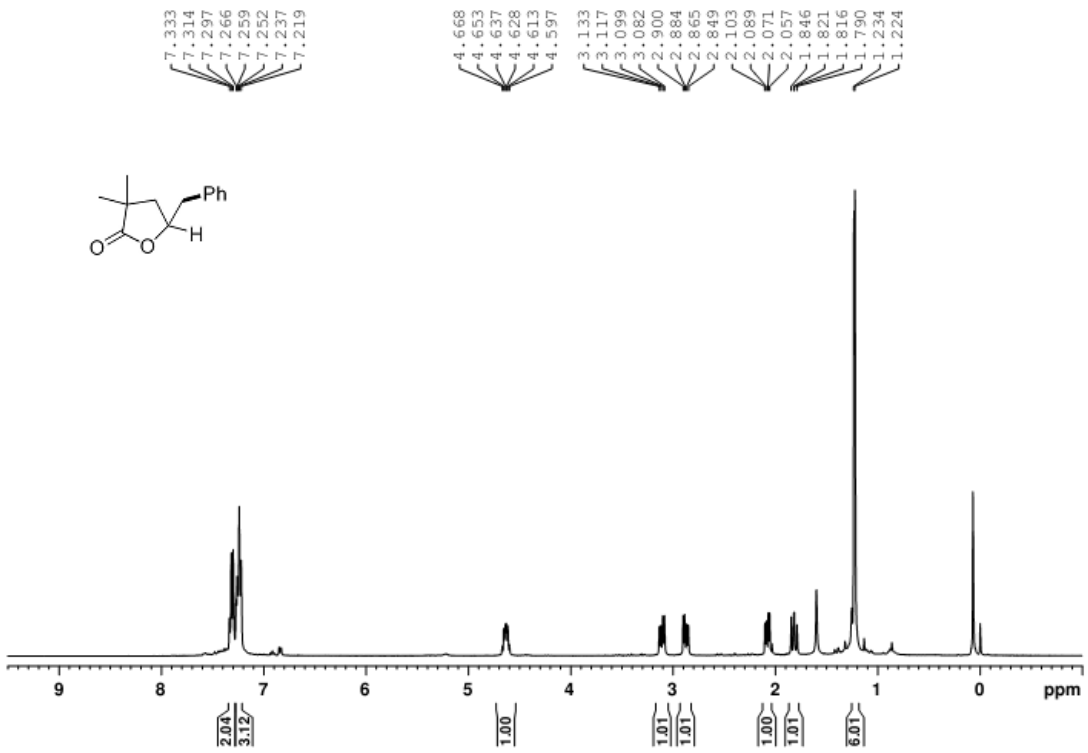
ppm
2.00
3.07
1.00
1.05
1.03
2.10
1.08
1.09

3v


177.21
136.08
129.63
128.83
127.15
80.97
77.55
77.23
76.91
41.51
28.81
27.30
Ph
H
O
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3w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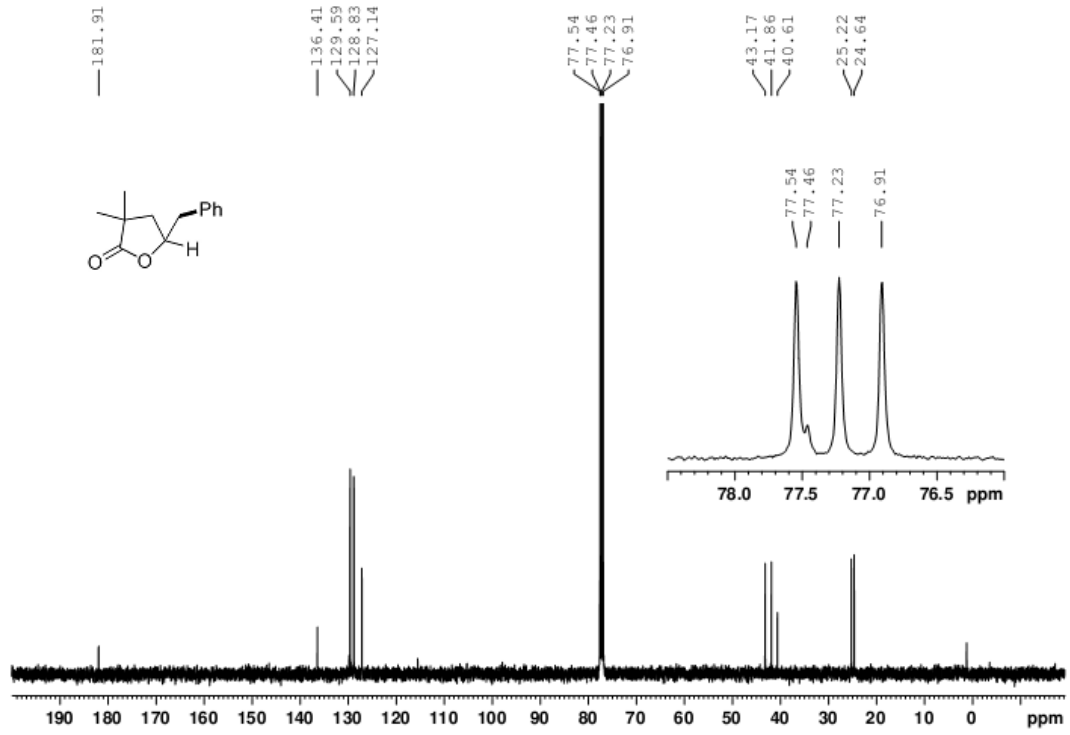
7.314
7.297
7.266
7.259
7.252
7.237
7.219
4.668
4.653
4.637
4.628
4.613
4.597
3.133
3.117
3.099
3.082
2.900
2.884
2.865
2.849
2.103
2.089
2.071
2.057
1.846
1.821
1.816
1.790
1.234
1.224
Ph
H
O
O
9
8
7
6
5
4
3
2
1
0
ppm
2.04
3.12
1.00
1.01
1.01
1.00
1.01
6.01

3w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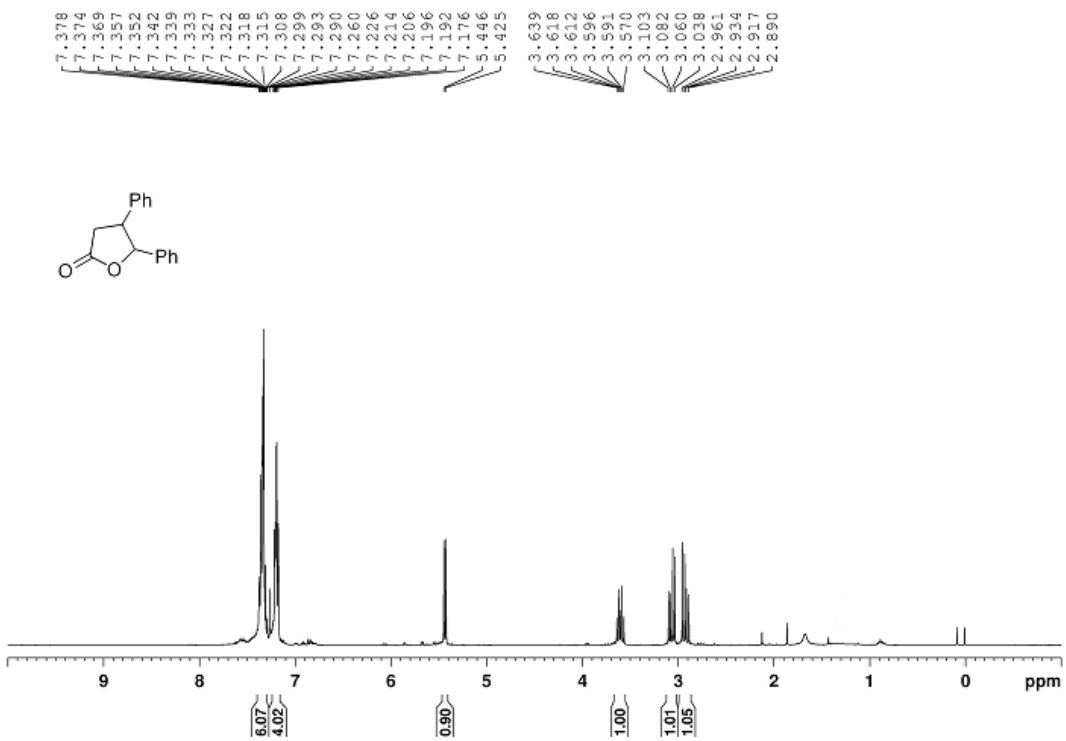
136.41
129.59
128.83
127.14
77.54
77.46
77.23
76.91
43.17
41.86
40.61
25.22
24.64
Ph
H
O
O
78.0
77.5
77.0
76.5
ppm
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3x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7.374
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7.352
7.342
7.339
7.333
7.327
7.322
7.318
7.315
7.308
7.299
7.293
7.290
7.260
7.226
7.214
7.206
7.196
7.192
7.176
5.446
5.425
3.639
3.618
3.612
3.596
3.591
3.570
3.103
3.082
3.060
3.038
2.961
2.934
2.917
2.890
Ph
Ph
O
O
9
8
7
6
5
4
3
2
1
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ppm
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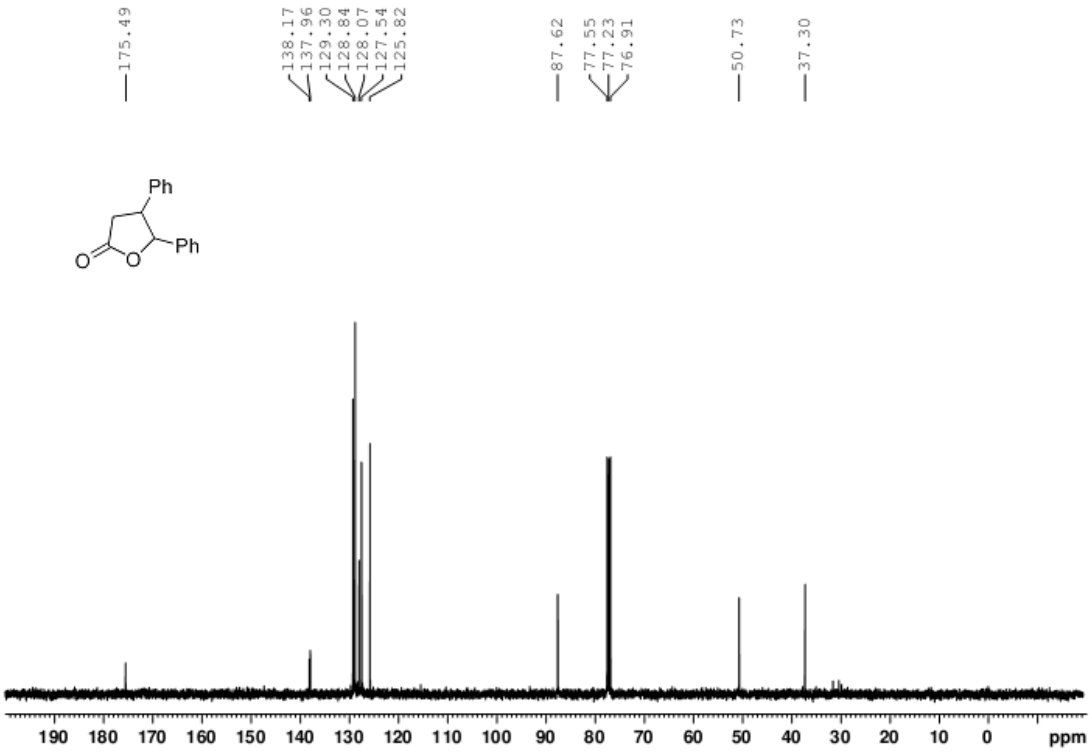
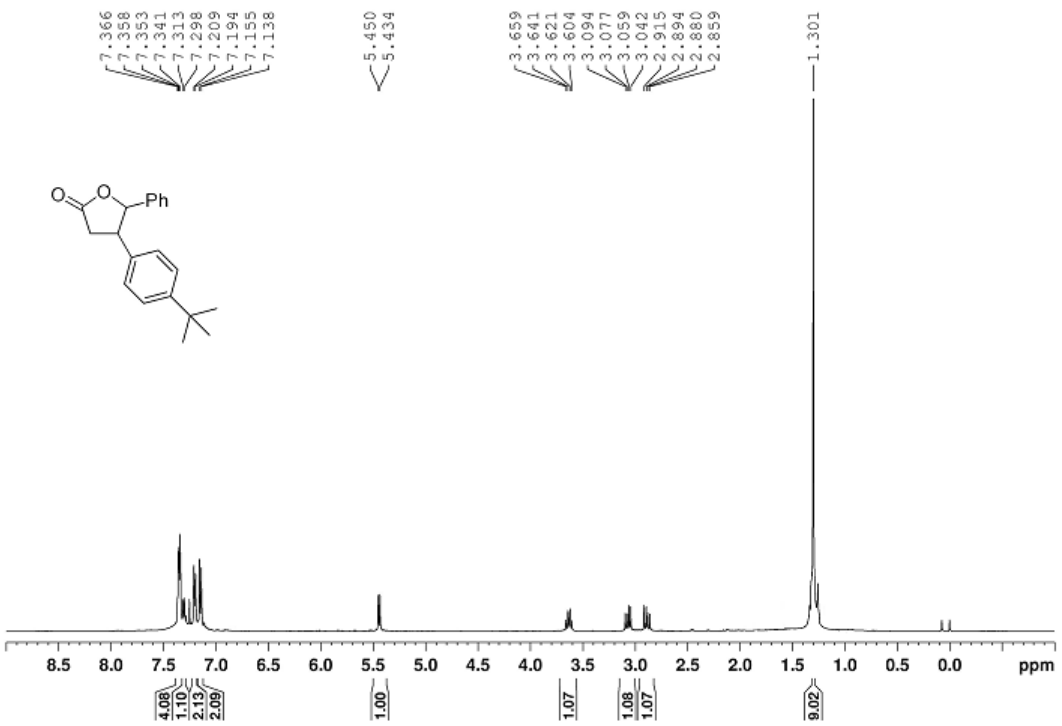
4.02
0.90
1.00
1.01
1.05

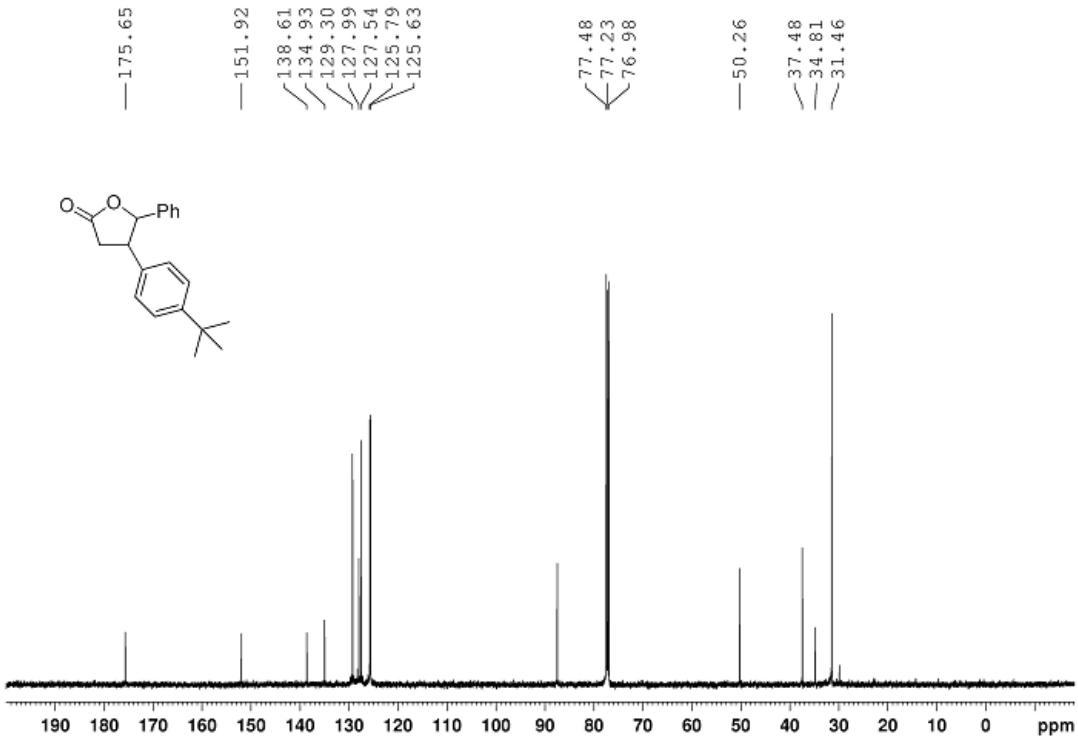
3x


175.49
138.17
137.96
129.30
128.84
128.07
127.54
125.82
87.62
77.55
77.23
76.91
50.73
37.30
Ph
Ph
O
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3y


7.366
7.358
7.353
7.341
7.313
7.298
7.209
7.194
7.155
7.138
5.450
5.434
3.659
3.641
3.621
3.604
3.094
3.077
3.059
3.042
2.915
2.894
2.880
2.859
1.301
O
O
Ph
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 ppm
4.08
1.10
2.13
2.09
1.00
1.07
1.08
1.07
9.02

3y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138.61
134.93
129.30
127.99
127.54
125.79
125.63
77.48
77.23
76.98
50.26
37.48
34.81
31.46
O
O
Ph
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm