Supporting Information

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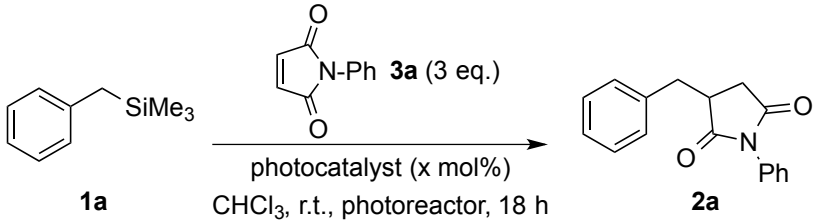
General Information

^{1}H, **^{13}C** and **^{19}F-NMR** spectra were recorded in $CDCl_3$ (reference signals: ^{1}H = 7.26 ppm, ^{13}C = 77.2 ppm) on a Bruker ARX-300, Bruker Avance II 300 or Bruker Avance II 400. ^{1}H-NMR chemical shifts are given relative to TMS and are referenced to the solvent signal. Spectra of ^{13}C and ^{19}F nucleotides are referenced according to the proton resonance of TMS as the primary reference for the unified chemical shift scale Chemical shifts (δ) are given in ppm and spin-spin coupling constants (J) are given in Hz. Analytical thin layer chromatography was performed using silica gel 60 F254 and a solution of $KMnO_4$ served as staining agent. Column chromatography was performed on silica gel 60 (0.040-0.063 mm). ESI accurate masses were measured on a MicroTof (Bruker Daltronics, Bremen) with loop injection or on an LTQ Orbitap LTQ XL (Thermo-Fisher Scientific, Bremen) with nano spray (alternatively HPLC, loop injection, syringe pump). Mass calibration on the MicroTof device was performed by using sodium formate cluster ions, immediately followed by the sample in a quasi-internal calibration. APCI mass spectra were recored on an LTQ Orbitap LTQ XL (Thermo-Fisher Scientific, Bremen) with loop injection. Steady-state fluorescence measurements were performed with FP-8500-Spectrofluorometer JASCO with the excitation of the chromophore at 420 nm. Photoreactions were carried out in 7 mL clear vials with screw caps from SUPELCO. The vials were irradiated in the photoreactor, which was wrapped with a 3.5m stripe of blue LEDs (450 nm, 250 lm/m, 4.8W/m). The benzyltrimethylsilane derivatives **1**[1] (except for commercially available **1a**) and the acceptor methyl 2-((phenylsulfonyl)methyl)acrylate (**3f**)[2] were prepared by modified literature procedures. Catalyst **III** was prepared according to a recently published protocol by our group.[3] Other solvents and commercially available reagents were used without further purification.

Optimization Screening

Table S1: Optimization of the catalyst and atmosphere.

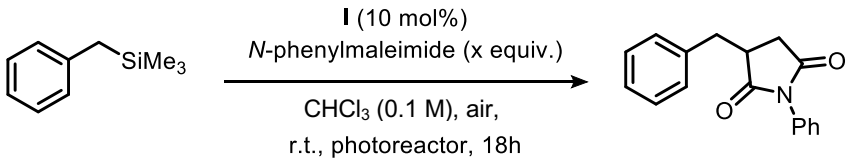


Entry	Catalyst (x mol%)	Atmosphere	Yield (%)[a]
1	**I** (10)	N_2	30
2	**II** (10)	N_2	36
3	**III** (10)	N_2	16
4	**Na_2-Eosin Y** (10)	N_2	--[b]
5	**$Ru(bpy)_3(PF_6)_2$** (10)	N_2	--[b]
6	**I** (10)	O_2	17
7	**I** (10)	air	49
8	**I** (7.5)	air	46
9	**I** (5)	air	46
10	**I** (2.5)	air	35

[a] Yields after flash column chromatography. [b] Product no detected by GC-MS.

Note: No conversion of **1a** was observed in the presence of H_2O_2, which could be produced in the catalytic cycle under aerobic conditions.

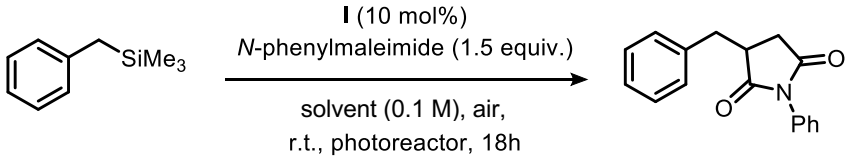
Table S2: Optimization of the amount of the Michael acceptor.



Entry	*N*-Phenylmaleimide (equiv.)	Yield (%)[a]
1	1	65
2	1.2	56
3	1.5	78
4	2	66
5	3	49

[a] Yields after flash column chromatography.

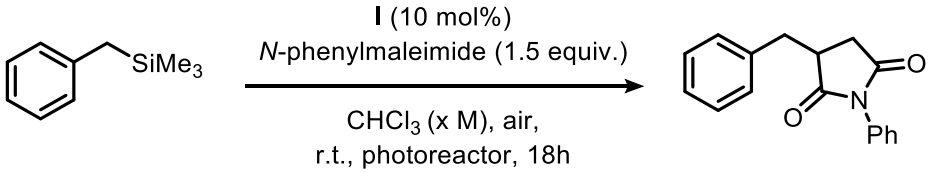
Table S3: Solvent screening.



Entry	Solvent	Yield (%)[a]
1	$CHCl_3$	78
2	DCM	15
3	MeOH	7
4	DMF	12
5	DMSO	6
6	acetone	5
7	MeCN	8
8	EtOH	13

[a] Yields after flash column chromatography.

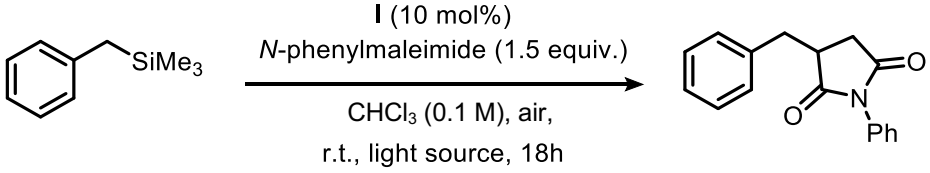
Table S4: Optimization of the concentration.



Entry	Solvent (mL)	Yield (%)[a]
1	$CHCl_3$ (1)	57
2	$CHCl_3$ (1.5)	54
3	$CHCl_3$ (2)	78
4	$CHCl_3$ (2.5)	62
5	$CHCl_3$ (3)	66
6	dry[b] $CHCl_3$ (2)	8

[a] Yields after flash column chromatography. [b] Chloroform was dried by distillation over CaH_2.

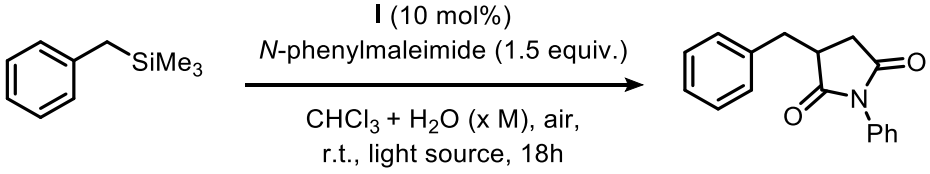
Table S5: Optimization of the light source.



Entry	Light source	Yield (%)[a]
1	cold CFL	60
2	warm CFL	28
3	LED block	51
4	LED photoreactor	78

[a] Yields after flash column chromatography.

Table S6: Optimization of the water content.



Entry	Solvent (mL)	Yield (%)[a]
1	$CHCl_3$ (2) + H_2O (0%)	78
2	$CHCl_3$ (2) + H_2O (2%) + HCl (1 %)	55
3	dry $CHCl_3$ (2) + dioxane (7%) + HCl (1 %)	23
4	dry $CHCl_3$ (2) + H_2O (1%)	14
5	dry $CHCl_3$ (2) + H_2O (5%)	17
6	dry $CHCl_3$ (2) + H_2O (10%)	18
7	dry $CHCl_3$ (2) + H_2O (20%)	17

[a] Yields after flash column chromatography.

Note: Freshly distilled dry solvents or the presence of H_2O led to notable lower yields (14-23% vs. 78%), whereas the addition of aq. HCl did not improve the outcome, suggesting that as much as ~1-2% vol. of HCl and/or H_2O hampers the reaction.

General Procedures

Synthesis of benzyltrimethylsilanes[1]

General procedure A: A solution of the appropriate benzylhalide (1 equiv.) and trimethylsilyl chloride (15 equiv.) in dry THF (0.12 M) was cooled down to -78 °C with an acetone-liquid nitrogen mixture under nitrogen atmosphere. *n*-Butyllithium (3.6 equiv., 1.6 M or 2.5 M in hexane) was added dropwise *via* a syringe pump (30 µL/min) at the give temperature. After the addition, the mixture was stirred for further 10 minutes at -78 °C, warmed up to room temperature and stirred for further 18 h at ambient temperature. A saturated solution of NH_4Cl was at 0 °C and the reaction mixture was extracted with ethyl acetate three times. The combined organic layers were washed with water (1x) and brine (1x) and dried over $MgSO_4$. The solvent was removed under reduced pressure and the desired benzyltrimethylsilane derivatives were obtained after flash column chromatography with petroleum ether or pentane.

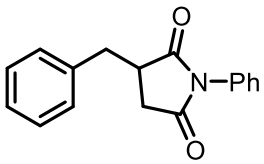
Photocatalyzed desilylative C-C bond formation reactions

General procedure B: The corresponding benzyltrimethylsilane **1** (0.2 mmol, 1.0 equiv.), Michael acceptor **3** (0.3 mmol, 1.5 equiv.) and 9-mesityl-10-methylacridinium perchlorate **I** (8.4 mg, 0.02 mmol, 10 mol%) were weight in a 7 mL clear vial with a screw cap from Supelco. The substrates were dissolved in chloroform (2 mL) and the reaction mixture was irradiated in a photoreactor wrapped by a stripe of blue LEDs for 18 h (Figure S1). The solvent was evaporated and the crude mixture was purified by flash column chromatography with a mixture of petroleum ether / ethyl acetate or pentane / ethyl acetate to give the corresponding coupling product.

Figure S1: Setup for the photoreactions.

Analytical Data of the Photoreaction Products

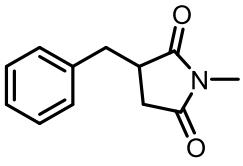
3-Benzyl-1-phenylpyrrolidine-2,5-dione (2a)[4]



According to general procedure B, the reaction of benzyltrimethylsilane (**1a**, 37.8 µL, 0.20 mmol, 1.0 equiv.) with **3a** (51.9 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (petroleum ether/ethyl acetate 9:1 → 8:2) as a white solid (41.4 mg, 0.156 mmol, 78%). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 7.50 – 7.42 (m, 2H), 7.42 – 7.26 (m, 4H), 7.24 – 7.14 (m, 4H), 3.37 – 3.28 (m, 1H), 3.25 (dd, *J* = 13.7, 4.5

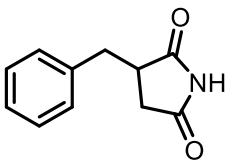
Hz, 1H), 3.13 – 3.04 (m, 1H), 2.88 (dd, *J* = 18.5, 9.1 Hz, 1H), 2.65 (dd, *J* = 18.5, 4.7 Hz, 1H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ 178.3, 175.3, 136.7, 131.8, 129.2, 129.2, 128.9, 128.7, 127.3, 126.5, 41.3, 36.6, 33.4. The spectra were in accordance with the ones in the literature.[4]

3-Benzyl-1-methylpyrrolidine-2,5-dione (2b)[4]



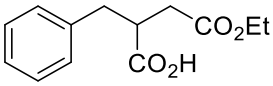
According to general procedure B, the reaction of **1a** (37.8 mg, 0.20 mmol, 1.0 equiv.) with **3b** (33.3 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a colorless oil (31.4 mg, 0.15 mmol, 77%). **^{1}H-NMR** (400 MHz, $CDCl_3$) δ 7.38 – 7.23 (m, 3H), 7.22 – 7.13 (m, 2H), 3.26 (dd, *J* = 13.8, 4.4 Hz, 1H), 3.15 (tt, *J* = 9.0, 4.6 Hz, 1H), 2.97 (s, 3H), 2.87 (dd, *J* = 13.8, 8.9 Hz, 1H), 2.70 (dd, *J* = 18.3, 8.9 Hz, 1H), 2.46 (dd, *J* = 18.4, 4.6 Hz, 1H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 179.4, 176.4, 137.2, 129.0, 128.8, 127.1, 41.4, 36.6, 33.3, 24.8. **HRMS** (ESI-MS): Mass calc. for $C_{12}H_{13}NO_2Na$ [M+Na$^+$]: *m/z* = 226.0844, found: *m/z* = 226.0829. The spectra were in accordance with the ones in the literature.[4]

3-Benzylpyrrolidine-2,5-dione (2c)[4]



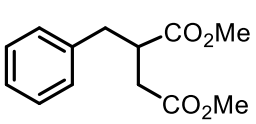
According to general procedure B, the reaction of **1a** (37.8 mg, 0.20 mmol, 1.0 equiv.) with **3c** (29.1 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a colorless oil (27.0 mg, 0.14 mmol, 71%). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 8.77 (s, 1H), 7.40 – 7.24 (m, 3H), 7.20 (d, *J* = 7.1 Hz, 2H), 3.22 (tt, *J* = 13.4, 4.7 Hz, 2H), 2.98 – 2.84 (m, 1H), 2.80 – 2.68 (m, 1H), 2.51 (dd, *J* = 18.5, 4.6 Hz, 1H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ 179.8, 176.7, 137.1, 129.0, 128.9, 127.2, 42.8, 36.3, 34.4. The spectra were in accordance with the ones in the literature.[4]

2-benzyl-4-ethoxy-4-oxobutanoic acid (2d)[5]



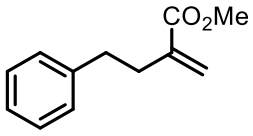
According to general procedure B, the reaction of **1a** (37.8 mg, 0.20 mmol, 1.0 equiv.) with **3d** (29.4 mg, 0.30 mmol, 1.5 equiv.) provided the title compound as a colorless oil (30.2 mg, 0.13 mmol, 64%). **^{1}H-NMR** (300 MHz, $CDCl_3$): δ 7.28 (m, 3H), 7.21 – 7.14 (m, 2H), 4.10 (q, *J* = 7.2 Hz, 2H), 3.23 – 3.05 (m, 2H), 2.85 – 2.71 (m, 1H), 2.71 – 2.56 (m, 1H), 2.40 (dd, *J* = 17.0, 4.7 Hz, 1H), 1.22 (t, *J* = 7.2 Hz, 3H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ 180.2, 171.9, 138.0, 129.2, 129.2, 128.8, 127.0, 60.9, 43.0, 37.5, 34.8, 14.2. The spectra were in accordance with the ones in the literature.[5]

Dimethyl 2-benzylsuccinate (2e)[4]



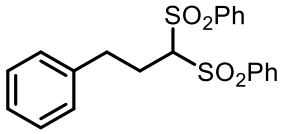
According to general procedure B, the reaction of **1a** (37.8 mg, 0.20 mmol, 1.0 equiv.) with **3e** (43.2 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a colorless oil (21.1 mg, 0.09 mmol, 45%). **^{1}H-NMR** (400 MHz, $CDCl_3$) δ 7.38 – 7.27 (m, 2H), 7.27 – 7.21 (m, 1H), 7.21 – 7.14 (m, 2H), 3.72 – 3.68 (m, 3H), 3.66 (s, 3H), 3.21 – 3.11 (m, 1H), 3.08 (dd, *J* = 13.5, 6.3 Hz, 1H), 2.83 – 2.66 (m, 2H), 2.43 (dd, *J* = 16.8, 4.9 Hz, 1H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 174.8, 172.4, 138.3, 129.1, 128.7, 126.9, 52.1, 51.9, 43.2, 37.9, 35.0. The spectra were in accordance with the ones in the literature.[4]

Methyl 2-methylene-4-phenylbutanoate (2f)[2]



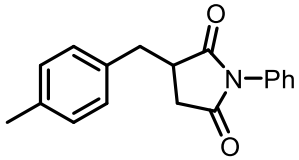
According to general procedure B, the reaction of **1a** (37.8 mg, 0.20 mmol, 1.0 equiv.) with **3f** (72.0 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a colorless oil (26.4 mg, 0.14 mmol, 71%). **^{1}H-NMR** (300 MHz, $CDCl_3$): δ 7.50 – 7.44 (m, 1H), 7.21 – 7.15 (m, 2H), 7.14 – 7.10 (m, 2H), 6.10 – 6.04 (m, 1H), 5.45 – 5.38 (m, 1H), 3.67 (s, 3H), 2.76 – 2.65 (m, 2H), 2.58 – 2.49 (m, 2H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 167.6, 141.4, 139.8, 136.6, 133.6, 128.5, 128.3, 126.0, 125.4, 77.4, 77.0, 76.7, 51.9, 34.9, 33.9. The spectra were in accordance with the ones in the literature.[2]

(3-Phenylpropane-1,1-diyldisulfonyl)dibenzene (2g)[6]



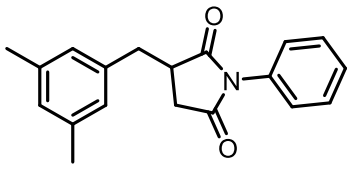
According to general procedure B, the reaction of **1a** (37.8 mg, 0.20 mmol, 1.0 equiv.) with **3g** (92.5 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a white solid (66.1 mg, 0.17 mmol, 83%). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 7.82 (d, *J* = 8.3 Hz, 4H), 7.68 – 7.59 (m, 2H), 7.49 (t, *J* = 7.4 Hz, 4H), 7.23 – 7.11 (m, 3H), 6.98 (d, *J* = 6.9 Hz, 2H), 4.31 (td, *J* = 5.7, 1.5 Hz, 1H), 2.88 (t, *J* = 7.4 Hz, 2H), 2.43 (q, *J* = 6.6 Hz, 2H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 138.7, 137.9, 134.5, 129.5, 129.2, 128.8, 128.68, 126.7, 81.6, 33.3, 27.0. **HRMS** (ESI-MS): Mass calc. for $C_{21}H_{20}O_4SNa$ [M+Na$^+$]: *m/z* = 423.0701, found: *m/z* = 423.0704.

3-(4-Methylbenzyl)-1-phenylpyrrolidine-2,5-dione (2h)



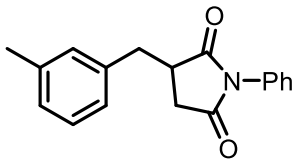
According to general procedure B, the reaction of 4-methyl-benzyltrimethylsilane (35.6 mg, 0.20 mmol, 1.0 equiv.) with **3a** (51.9 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a white solid (30.1 mg, 0.11 mmol, 54%). **^{1}H-NMR** (300 MHz, $CDCl_3$): δ 7.52 – 7.35 (m, 3H), 7.23 – 7.06 (m, 6H), 3.38 – 3.24 (m, 1H), 3.20 (dd, *J* = 13.8, 4.5 Hz, 1H), 3.06 (dd, *J* = 13.8, 7.9 Hz, 1H), 2.88 (dd, *J* = 18.5, 9.0 Hz, 1H), 2.64 (dd, *J* = 18.5, 4.7 Hz, 1H), 2.34 (s, 3H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 178.4, 175.4, 136.9, 133.5, 131.9, 129.6, 129.2, 129.1, 128.7, 126.5, 41.3, 36.2, 33.3, 21.1. **HRMS** (ESI-MS): Mass calc. for $C_{18}H_{17}NO_2Na$ [M+Na$^+$]: *m/z* = 302.1151, found: *m/z* = 302.1153.

3-(3,5-Dimethylbenzyl)-1-phenylpyrrolidine-2,5-dione (2i)



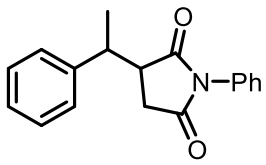
According to general procedure B, the reaction of (3,5-dimethylbenzyl)trimethylsilane (38.5 mg, 0.20 mmol, 1.0 equiv.) with **3a** (51.9 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a white solid (23.5 mg, 0.08 mmol, 40%). **^{1}H-NMR** (400 MHz, $CHCl_3$): δ 7.46 (dd, *J* = 7.4, 6.9 Hz, 2H), 7.39 (t, *J* = 7.3 Hz, 1H), 7.18 (d, *J* = 7.9 Hz, 2H), 6.91 (s, 1H), 6.83 (s, 2H), 3.33 – 3.24 (m, 1H), 3.16 (dd, *J* = 13.8, 4.5 Hz, 1H), 3.01 (dd, *J* = 13.5, 7.9 Hz, 1H), 2.86 (dd, *J* = 18.5, 9.1 Hz, 1H), 2.65 (dd, *J* = 18.5, 3.4 Hz, 1H). **^{13}C-NMR** (100 MHz, $CHCl_3$): δ 178.6, 175.6, 138.5, 136.6, 132.0, 129.3, 129.0, 128.7, 127.2, 126.6, 41.4, 36.6, 33.4, 21.4. **HRMS** (ESI-MS): Mass calc. for $C_{19}H_{19}NO_2Na$ [M+Na$^+$]: *m/z* = 316.1313, found: *m/z* = 316.1315.

3-(3-Methylbenzyl)-1-phenylpyrrolidine-2,5-dione (2j)



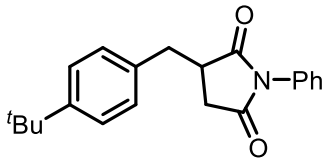
According to general procedure B, the reaction of 3-methyl-benzyltrimethylsilane (35.6 mg, 0.20 mmol, 1.0 equiv.) with **3a** (51.9 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a white solid (42.4 mg, 0.15 mmol, 76%). **^{1}H-NMR** (300 MHz, $CDCl_3$): δ 7.52 – 7.35 (m, 3H), 7.25 – 6.98 (m, 6H), 3.38 – 3.26 (m, 1H), 3.25 – 3.14 (m, 1H), 3.05 (dd, *J* = 13.7, 8.0 Hz, 1H), 2.88 (dd, *J* = 18.5, 9.0 Hz, 1H), 2.65 (dd, *J* = 18.5, 4.6 Hz, 1H), 2.34 (s, 3H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 178.4, 175.4, 138.6, 136.6, 131.9, 130.0, 129.2, 128.8, 128.7, 128.0, 126.5, 126.3, 41.3, 36.6, 33.3, 21.4. **HRMS** (ESI-MS): Mass calc. for $C_{18}H_{17}NO_2Na$ [M+Na$^+$]: *m/z* = 302.1151, found: *m/z* = 302.1155.

1-Phenyl-3-(1-phenylethyl)pyrrolidine-2,5-dione (2k)



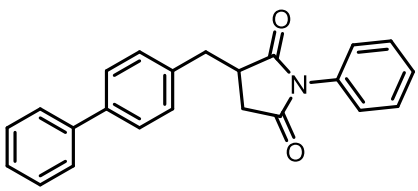
According to general procedure B, the reaction of trimethyl(1-phenylethyl)silane (35.6 mg, 0.20 mmol, 1.0 equiv.) with **3a** (51.9 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a mixture of diastereoisomers (79:21 d.r.) and a white solid (27.9 mg, 0.1 mmol, 50%). **^{1}H-NMR** (300 MHz, $CDCl_3$) δ 7.48 – 7.42 (m, 1H), 7.42 – 7.38 (m, 2H), 7.38 – 7.34 (m, 1H), 7.34 – 7.30 (m, 2H), 7.23 (dd, *J* = 8.1, 1.7 Hz, 2H), 7.03 – 6.93 (m, 2H), 3.73 – 3.61 (m, 1H), 3.22 (ddd, *J* = 9.1, 5.0, 4.3 Hz, 1H), 2.82 (dd, *J* = 18.5, 9.1 Hz, 1H), 2.64 (dd, *J* = 18.5, 4.3 Hz, 1H), 1.50 (d, *J* = 7.2 Hz, 3H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 178.1, 175.3, 140.3, 131.7, 129.2, 129.1, 128.9, 128.8, 128.6, 127.9, 127.6, 127.4, 126.5, 126.5, 46.6, 39.8, 30.9, 18.9. **HRMS** (ESI-MS): Mass calc. for $C_{18}H_{17}NO_2Na$ [M+Na$^+$]: *m/z* = 302.1151, found: *m/z* = 302.1158.

3-(4-(*tert*-Butyl)benzyl)-1-phenylpyrrolidine-2,5-dione (2l)



According to general procedure B, the reaction of 4-*tert*-butyl-benzyltrimethylsilane (44.0 mg, 0.20 mmol, 1.0 equiv.) with **3a** (51.9 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a colorless oil (39.8 mg, 0.124 mmol, 62%). **^{1}H-NMR** (300 MHz, $CDCl_3$): δ 7.52 – 7.30 (m, 5H), 7.18 – 7.10 (m, 4H), 3.39 – 3.24 (m, 1H), 3.22 (dd, *J* = 13.8, 4.3 Hz, 1H), 3.05 (dd, *J* = 13.8, 8.1 Hz, 1H), 2.90 (dd, *J* = 18.5, 9.0 Hz, 1H), 2.67 (dd, *J* = 18.5, 4.6 Hz, 1H), 1.33 (s, 9H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ 178.4, 175.5, 150.2, 133.6, 131.9, 129.2, 128.9, 128.7, 126.5, 125.8, 41.4, 36.2, 34.5, 33.5, 31.4. **HRMS** (ESI-MS): Mass calc. for $C_{21}H_{23}NO_2Na$ [M+Na$^+$]: *m/z* = 344.1626, found: *m/z* = 344.1621.

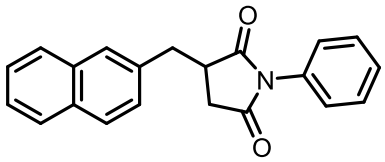
3-([1,1'-Biphenyl]-4-ylmethyl)-1-phenylpyrrolidine-2,5-dione (2m)



According to general procedure B, the reaction of ([1,1'-biphenyl]-4-ylmethyl)trimethylsilane (48.1 mg, 0.20 mmol, 1.0 equiv.) with **3a** (51.9 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a white solid (39.6 mg, 0.12 mmol, 58%). **^{1}H-NMR** (500 MHz, $CHCl_3$): δ 7.62 – 7.54 (m, 4H), 7.49 – 7.43 (m, 4H), 7.41 – 7.34 (m, 2H), 7.32 – 7.28 (m, 2H), 7.21 – 7.17 (m, 2H), 3.39 –

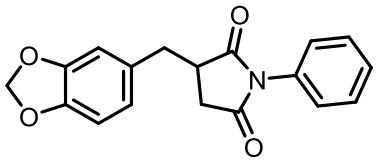
3.33 (m, 1H), 3.30 (dd, *J* = 13.8, 4.5 Hz, 1H), 3.13 (dd, *J* = 13.8, 8.0 Hz, 1H), 2.93 (dd, *J* = 18.4, 9.2 Hz, 1H), 2.70 (dd, *J* = 18.4, 4.7 Hz, 1H). **^{13}C-NMR** (126 MHz, $CHCl_3$): δ 178.4, 175.4, 140.7, 140.4, 135.9, 132.0, 129.8, 129.3, 129.0, 128.8, 127.7, 127.5, 127.2, 126.6, 41.4, 36.4, 33.6. **HRMS** (ESI-MS): Mass calc. for $C_{23}H_{19}NO_2Na$ [M+Na^+]: *m/z* = 364.1308, found: *m/z* = 364.1304.

3-(Naphthalen-2-ylmethyl)-1-phenylpyrrolidine-2,5-dione (2n)



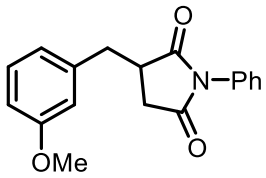
According to general procedure B, the reaction of trimethyl(naphthalen-2-ylmethyl)silane (42.9 mg, 0.20 mmol, 1.0 equiv.) with **3a** (51.9 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a white solid (23.3 mg, 0.07 mmol, 37%). **^{1}H-NMR** (400 MHz, $CHCl_3$): δ 7.88 – 7.78 (m, 3H), 7.68 (s, 1H), 7.52 – 7.33 (m, 6H), 7.18 (d, *J* = 7.7 Hz, 2H), 3.48 – 3.36 (m, 2H), 3.27 – 3.19 (m, 1H), 2.88 (dd, *J* = 18.5, 9.0 Hz, 1H), 2.69 (dd, *J* = 18.5, 4.3 Hz, 1H). **^{13}C NMR** (100 MHz, $CHCl_3$): δ 178.4, 175.3, 134.4, 133.6, 132.6, 132.0, 129.3, 128.8, 128.8, 128.0, 127.8, 127.7, 127.2, 126.6, 126.1, 41.4, 36.9, 33.5. **HRMS** (ESI-MS): Mass calc. for $C_{21}H_{17}NO_2Na$ [M+Na^+]: *m/z* = 338.1157, found: *m/z* = 338.1160.

3-(Benzo[d][1,3]dioxol-5-ylmethyl)-1-phenylpyrrolidine-2,5-dione (2o)



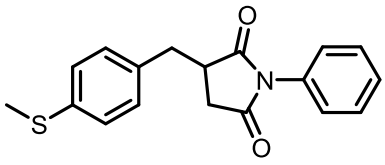
According to general procedure B, the reaction of benzo[d][1,3]dioxol-5-ylmethyl)trimethylsilane (41.7 mg, 0.20 mmol, 1.0 equiv.) with **3a** (51.9 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a white solid (24.6 mg, 0.08 mmol, 40%). **^{1}H-NMR** (400 MHz, $CHCl_3$): δ 7.49 – 7.43 (m, 2H), 7.41 – 7.36 (m, 1H), 7.20 (d, J = 7.9 Hz, 2H), 6.76 (d, *J* = 7.9 Hz, 1H), 6.70 (s, 1H), 6.66 (d, *J* = 7.9 Hz, 1H), 5.96 (s, 2H), 3.30 – 3.22 (m, 1H), 3.14 (dd, *J* = 14.0, 4.6 Hz, 1H), 3.03 (dd, *J* = 14.0, 7.8 Hz, 1H), 2.88 (dd, *J* = 18.5, 9.2 Hz, 1H), 2.64 (dd, *J* = 18.5, 4.7 Hz, 1H). **^{13}C-NMR** (100 MHz, $CHCl_3$): δ 178.4, 175.5, 148.2, 146.9, 132.0, 130.4, 129.3, 128.8, 126.6, 122.5, 109.6, 108.7, 101.3, 41.5, 36.4, 33.3. **HRMS** (ESI-MS): Mass calc. for $C_{18}H_{15}NO_4Na$ [M+Na^+]: *m/z* = 332.0893, found: *m/z* = 332.0892.

3-(3-Methoxybenzyl)-1-phenylpyrrolidine-2,5-dione (2p)



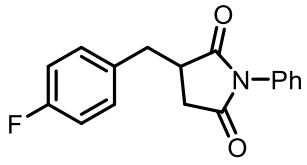
According to general procedure B, the reaction of 3-methoxylbenzyltrimethylsilane (38.8 mg, 0.20 mmol, 1.0 equiv.) with **3a** (51.9 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a colorless oil (44.8 mg, 0.152 mmol, 76%). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 7.53 – 7.38 (m, 3H), 7.32 – 7.18 (m, 3H), 6.88 – 6.77 (m, 3H), 3.81 (s, 3H), 3.40 – 3.29 (m, 1H), 3.26 (dd, *J* = 13.8, 4.5 Hz, 1H), 3.15 – 3.03 (m, 1H), 2.91 (dd, *J* = 18.5, 9.1 Hz, 1H), 2.68 (dd, *J* = 18.4, 4.6 Hz, 1H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 178.3, 175.3, 160.0, 138.3, 131.9, 129.9, 129.2, 128.7, 126.5, 121.5, 115.0, 112.5, 55.2, 41.2, 36.6, 33.4. **HRMS** (ESI-MS): Mass calc. for $C_{18}H_{17}NO_3Na$ [M+Na^+]: *m/z* = 318.1106, found: *m/z* = 318.1105.

3-(4-(Methylthio)benzyl)-1-phenylpyrrolidine-2,5-dione (2q)



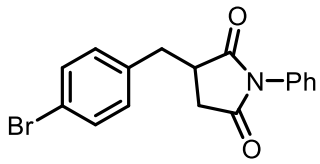
According to general procedure B, the reaction of 4-methylsulfide-benzyltrimethylsilane (42.0 mg, 0.20 mmol, 1.0 equiv.) with **3a** (51.9 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a white solid (44.8 mg, 0.14 mmol, 72%). **^{1}H-NMR** (300 MHz, $CDCl_3$): δ 7.46 – 7.30 (m, 3H), 7.22 – 7.04 (m, 6H), 3.30 – 3.19 (m, 1H), 3.15 (dd, *J* = 13.9, 4.5 Hz, 1H), 2.99 (dd, *J* = 13.9, 8.0 Hz, 1H), 2.83 (dd, *J* = 18.5, 9.1 Hz, 1H), 2.56 (dd, *J* = 18.4, 4.8 Hz, 1H), 2.43 (s, 3H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ 178.2, 175.2, 137.5, 133.5, 131.8, 129.7, 129.2, 128.7, 127.1, 126.46, 41.3, 36.0, 33.3, 15.9. **HRMS** (ESI-MS): Mass calc. for $C_{18}H_{17}NO_2SNa$ [M+Na^+]: *m/z* = 334.0872, found: *m/z* = 334.0877.

3-(4-Fluorobenzyl)-1-phenylpyrrolidine-2,5-dione (2r)



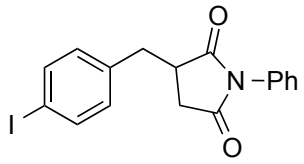
According to general procedure B the reaction of 4-fluoro-benzyltrimethylsilane (36.4 mg, 0.20 mmol, 1.0 equiv.) with **3a** (51.9 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a white solid (50.4 mg, 0.18 mmol, 89%). **^{1}H-NMR** (300 MHz, $CDCl_3$): δ 7.52 – 7.33 (m, 3H), 7.24 – 7.12 (m, 4H), 7.09 – 6.97 (m, 2H), 3.30 (ddt, *J* = 9.1, 7.8, 4.7 Hz, 1H), 3.21 (dd, *J* = 13.9, 4.5 Hz, 1H), 3.14 – 3.02 (m, 1H), 2.90 (dd, *J* = 18.4, 9.1 Hz, 1H), 2.61 (dd, *J* = 18.4, 4.7 Hz, 1H). **^{13}C-NMR** (500 MHz, $CDCl_3$): δ 178.1, 175.1, 162.2 (d, J_{CF} = 245 Hz), 132.4 (d, J_{CF} = 3.7 Hz), 131.7, 130.8 (d, J_{CF} = 8.0 Hz), 129.2, 128.7, 126.4, 115.8 (d, J_{CF} = 21.3 Hz), 41.3, 35.7, 33.2, 29.7. **^{19}F-NMR** (282 MHz, $CDCl_3$): δ -115.21. **HRMS** (ESI-MS): Mass calc. for $C_{17}H_{14}FNNaO_2$ [M+Na^+]: *m/z* = 306.0906, found: *m/z* = 306.0904.

3-(4-Bromobenzyl)-1-phenylpyrrolidine-2,5-dione (2s)



According to general procedure B, the reaction of 4-bromo-benzyltrimethylsilane (48.4 mg, 0.20 mmol, 1.0 equiv.) with **3a** (51.9 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a white solid (57.5 mg, 0.17 mmol, 84%). **^{1}H-NMR** (300 MHz, $CDCl_3$): δ 7.55 – 7.34 (m, 5H), 7.22 – 7.14 (m, 2H), 7.14 – 7.05 (m, 2H), 3.36 – 3.25 (m, 1H), 3.20 (dd, *J* = 13.9, 4.6 Hz, 1H), 3.04 (dd, *J* = 13.9, 8.0 Hz, 1H), 2.90 (dd, *J* = 18.4, 9.1 Hz, 1H), 2.59 (dd, *J* = 18.5, 4.9 Hz, 1H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ 178.0, 175.0, 135.7, 132.1, 131.7, 130.9, 129.3, 128.8, 126.4, 121.3, 41.1, 36.0, 33.3. **HRMS** (ESI-MS): Mass calc. for $C_{17}H_{14}BrNO_2Na$ [M+Na^+]: *m/z* = 366.0106, found: *m/z* = 366.0100.

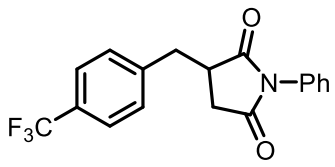
1-Phenyl-3-(4-(iodo)benzyl)pyrrolidine-2,5-dione (2t)



According to general procedure B, the reaction of 4-iodo-benzyltrimethylsilane (58.0 mg, 0.20 mmol, 1.0 equiv.) with **3a** (51.9 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a white solid (39.2 mg, 0.10 mmol, 50%). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 7.66 (d, J = 8.3 Hz, 2H), 7.50 – 7.43 (m, 2H), 7.42 – 7.34 (m, 1H), 7.20 – 7.11 (m, 2H), 6.98 (d, J = 8.3 Hz, 2H), 3.29 (ddt, J = 9.3, 8.0, 4.7 Hz, 1H), 3.19 (dd, J = 13.9, 4.6 Hz, 1H), 3.02 (dd, J = 13.9, 8.1

Hz, 1H), 2.89 (dd, J = 18.4, 9.2 Hz, 1H), 2.59 (dd, J = 18.4, 4.9 Hz, 1H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 178.0, 175.1, 138.1, 136.6, 131.8, 131.3, 129.4, 128.9, 126.5, 92.8, 41.2, 36.2, 33.4. **HRMS** (ESI-MS): Mass calc. for $C_{17}H_{14}INO_2Na$ [M+Na$^+$]: *m/z* = 413.9967, found: *m/z* = 413.9964.

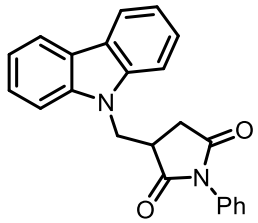
1-Phenyl-3-(4-(trifluoromethyl)benzyl)pyrrolidine-2,5-dione (2u)



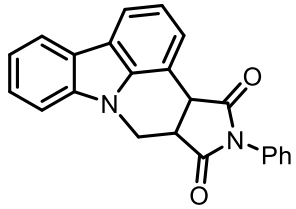
According to general procedure B, the reaction of 4-trifluoromethyl-benzyltrimethylsilane (46.4 mg, 0.20 mmol, 1.0 equiv.) with **3a** (51.9 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a white solid (23.3 mg, 0.07 mmol, 35%). **^{1}H-NMR** (300 MHz, $CDCl_3$): δ 7.61 (d, *J* = 7.9 Hz, 2H), 7.52 – 7.31 (m, 5H), 7.22 – 7.14 (m, 2H), 3.41 – 3.28 (m, 2H), 3.11 (dd, *J* = 15.0, 9.5 Hz, 1H), 2.92 (dd, *J* = 18.4, 8.8 Hz, 1H), 2.68 – 2.53 (m, 1H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ 177.8, 174.8, 141.0, 131.7, 129.7, 129.4 (q, J_{CF} = 32.7 Hz), 128.8, 126.4, 125.9 (q, J_{CF} = 3.7 Hz), 124.2 (q, J_{CF} = 273.2 Hz), 41.1, 36.4, 33.4. **^{19}F-NMR** (282 MHz, $CDCl_3$): δ -62.5. **HRMS** (ESI-MS): Mass calc. for $C_{18}H_{14}F_3NO_2Na$ [M+Na$^+$]: *m/z* = 356.0874, found: *m/z* = 356.0885.

3-((9H-Carbazol-9-yl)methyl)-1-phenylpyrrolidine-2,5-dione (4a) and 2-phenyl-3a,12a-dihydropyrroloz[3',4':5,6]pyrido[3,2,1-jk]carbazole-1,3(2H,4H)-dione (4a')

According to general procedure B, the reaction of 9-((trimethylsilyl)methyl)-9H-carbazole (50.6 mg, 0.2 mmol, 1 equiv.) with *N*-phenylmaleimide (51.9 mg, 0.3 mmol, 1.5 equiv.) provided after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) an inseparable mixture of the title compounds as a white solid (36 mg, 60%).

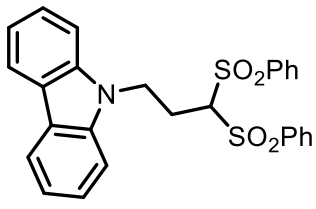


4a: ^{1}H-NMR of (400 MHz, $CHCl_3$): 7.47 – 7.02 (m, 13 H), 4.79 (d, *J* = 5.9 Hz, 2H), 3.62 –3.50 (m, 1H), 2.75 (dd, *J* = 18.5, 9.3 Hz, 1H), 2.61 (dd, *J* = 18.5, 5.4 Hz, 1H). **^{13}C-NMR** (100 MHz, $CHCl_3$): 175.1, 174.6, 140.3, 131.6, 128.9, 128.7, 126.3, 123.6, 122.7, 121.0, 120.7, 120.4, 120.1, 120.0, 119.9, 108.7, 42.9, 41.0, 32.4. **HRMS** (ESI-MS): Mass calc. for $C_{23}H_{18}N_2O_2Na$ [M+Na$^+$]: *m/z* = 377.1260, found: *m/z* = 377.1284.



4a': ^{1}H-NMR (400 MHz, $CHCl_3$): δ 8.09 (t, *J* = 8.3 Hz, 2H), 7.99 (d, *J* = 7.8 Hz, 1H), 7.75 (d, *J* = 7.5 Hz, 1H), 7.54 - 7.09 (m, 8H), 5.07 (dd, *J* = 12.1, 2.3 Hz, 1H), 4.58 (d, *J* = 8.4 Hz, 1H), 4.09 (dd, *J* = 12.1, 5.5 Hz, 1H), 4.00 – 3.90 (m, 1H). **^{13}C-NMR** (100 MHz, $CHCl_3$): 177.2, 175.8, 141.0, 137.3, 129.2, 129.1, 126.4, 128.2, 124.1, 123.3, 121.0, 120.7, 120.4, 120.1, 120.0, 119.9, 109.5, 112.7, 41.4, 40.0, 37.4. **HRMS** (ESI-MS): Mass calc. for $C_{23}H_{16}N_2O_2Na$ [M+Na$^+$]: *m/z* = 375.1104, found: *m/z* = 375.1128.

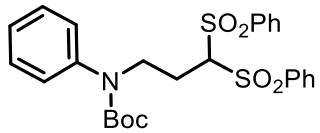
9-(3,3-Bis(phenylsulfonyl)propyl)-9H-carbazole (4b)



According to general procedure B, the reaction of 9-((trimethylsilyl)methyl)-9H-carbazole (50.6 mg, 0.2 mmol, 1 equiv.) with (ethene-1,1-diyldisulfonyl) dibenzene (92.4 mg, 0.3 mmol) provided after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) the title compound as a white solid (70.2 mg, 0.14 mmol, 72%). **^{1}H-NMR** (300 MHz, $CHCl_3$): δ 7.93 (d, *J* = 7.8 Hz, 2H), 7.63

– 7.54 (m, 4H), 7.41 – 7.29 (m, 4H), 7.29 – 7.09 (m, 8H), 4.58 (t, *J* = 6.3 Hz, 2H), 4.39 (q, *J* = 5.6, 5.2 Hz, 1H), 2.63 (q, *J* = 6.0 Hz, 2H). **^{13}C-NMR** (75 MHz, $CDCl_3$) δ 139.9, 137.2, 134.5, 129.2, 129.0, 128.9, 126.1, 123.0, 120.5, 119.5, 108.2, 79.6, 41.0, 25.9. **HRMS**: (ESI-MS): Mass calc. for $C_{27}H_{23}NO4S_2Na$ [M+Na$^+$]: *m/z* = 512.0966, found: *m/z* = 512.0953.

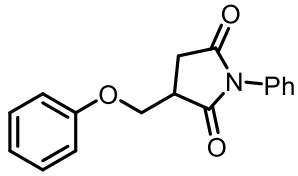
***tert*-Butyl (3,3-bis(phenylsulfonyl)propyl)(phenyl)carbamate (4c)** [7]



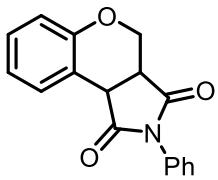
According to general procedure B, the reaction of *tert*-butyl phenyl((trimethylsilyl)methyl)carbamate (55.8 mg, 0.2 mmol, 1 equiv.) with (ethene-1,1-diyldisulfonyl)dibenzene (92.4 mg, 0.3 mmol) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a white solid (51 mg, 0.1 mmol, 50%). **^{1}H-NMR** (300 MHz, $CHCl_3$) δ 7.76 (d, *J* = 7.4 Hz, 4H), 7.62 – 7.53 (m, 2H), 7.42 (t, *J* = 7.8 Hz, 4H), 7.32 – 7.23 (m, 2H), 7.22 – 7.14 (m, 1H), 7.11 – 7.04 (m, 2H), 3.94 (t, *J* = 5.9 Hz, 2H), 2.35 (q, *J* = 5.7 Hz, 2H), 1.34 (s, 1H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ 155.2, 142.5, 137.8, 134.5, 129.7, 129.2, 129.1, 127.4, 126.5, 81.0, 80.3, 48.5, 28.3, 25.5. **HRMS**: (ESI-MS): Mass calc. for $C_{26}H_{29}NO_6S_2Na$ [M+Na$^+$]: *m/z* = 538.1334, found: *m/z* = 538.1324. The spectra are in accordance with already published data.[7]

3-(Phenoxymethyl)-1-phenylpyrrolidine-2,5-dione (4d) and 2-phenyl-3a,9b-dihydrochromeno-[3,4-c]pyrrole-1,3(2H,4H)-dione (4d') [8]

According to general procedure B, the reaction of trimethyl(phenoxymethyl)silane (36.0 mg, 0.2 mmol, 1 equiv.) with *N*-phenylmaleimide (51.9 mg, 0.3 mmol) provided after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) the title compounds **4e** as a white solid (23.7 mg, 0.084 mmol, 42%) and **4e'** as a colorless oil (28.9 mg, 0.104 mmol, 52%).



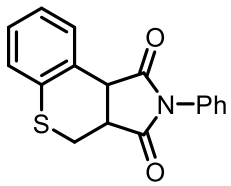
4e: ^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.48 – 7.27 (m, 3H), 7.27 – 7.19 (m, 4H), 6.97 – 6.88 (m, 1H), 6.88 – 6.78 (m, 2H), 4.45 (dd, *J* = 9.1, 3.9 Hz, 1H), 4.16 (dd, *J* = 9.1, 3.2 Hz, 1H), 3.35 – 3.22 (m, 1H), 3.02 – 2.94 (m, 2H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 176.8, 175.6, 158.2, 132.1, 129.7, 129.3, 128.8, 126.7, 121.8, 114.8, 114.8, 77.5, 77.2, 76.8, 66.55, 40.8, 32.1.



4e': ^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.58 – 7.52 (m, 1H), 7.42 – 7.26 (m, 3H), 7.22 – 7.13 (m, 3H), 7.00 (td, *J* = 7.5, 1.3 Hz, 1H), 6.87 (dd, *J* = 8.2, 1.3 Hz, 1H), 4.62 (dd, *J* = 11.3, 3.2 Hz, 1H), 4.15 (d, *J* = 9.3 Hz, 1H), 4.03 (dd, *J* = 11.4, 4.2 Hz, 1H), 3.52 – 3.43 (m, 1H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 175.8, 175.2, 155.4, 131.8, 130.4, 129.3, 129.2, 128.9, 126.5, 123.0, 118.1, 117.4, 77.5, 77.2, 76.8, 64.1, 42.4, 40.0.

The spectra are in accordance with already published data.[8]

2-Phenyl-3a,9b-dihydrothiochromeno[3,4-c]pyrrole-1,3(2H,4H)-dione (4e')



According to general procedure B, the reaction of trimethyl((phenylthio)methyl)silane (51.0 mg, 0.2 mmol, 1 equiv.) with *N*-phenylmaleimide (51.9 mg, 0.3 mmol) provided the title compound after flash column chromatography (pentane/ethyl acetate 9:1 → 8:2) as a white solid (29.8 mg, 0.1 mmol, 50%). **^{1}H-NMR** (300 MHz, $CDCl_3$) δ 7.40 – 7.23 (m, 4H), 7.23 – 7.10 (m, 5H), 4.14 (d, *J* = 9.4 Hz, 1H), 3.76 – 3.67 (m, 1H), 3.31 (dd, *J* = 13.3, 2.4 Hz, 1H), 2.82 (dd, *J* = 13.3, 4.8 Hz, 1H). **^{13}C-NMR** (75 MHz, $CDCl_3$) δ 177.2, 175.0, 134.8, 132.0, 131.9, 130.5, 129.8,

129.3, 129.2, 128.8, 128.1, 127.1, 126.5, 126.5, 45.7, 44.7, 30.5. **HRMS**: (ESI-MS): Mass calc. for $C_{17}H_{13}NO_2SNa$ [M+Na$^+$]: *m/z* = 318.0565, found: *m/z* = 318.0549.

Quenching Experiment and Quantum Yield Determination

Stern-Volmer measurements

Emission intensities were recorded on a Jasco FP-8500 spectrofluorometer. Dry MeCN was degassed by argon bubbling for 30 minutes before using. The catalyst **I** was excited at 420 nm and the emission spectra were recorded between 430 and 800 nm. In a typical experiment, 0.1 mM solutions of **I** in MeCN were prepared with the appropriate concentration of quencher in a 1.0 cm quartz cuvette and covered.

(Note: It is reported that the excited-state of **I** is only able to oxidize electron-rich olefins, not being quenched by acceptors like **3**: K. A. Margrey and D. A. Nicewicz, *Acc. Chem. Res.,* 2016, **49**, 1997-2006.)

Figure S2a: *Fluorescence quenching of catalyst Mes-Acr$^+$* ***I*** *by different concentrations of* ***1a*** *(Q).*

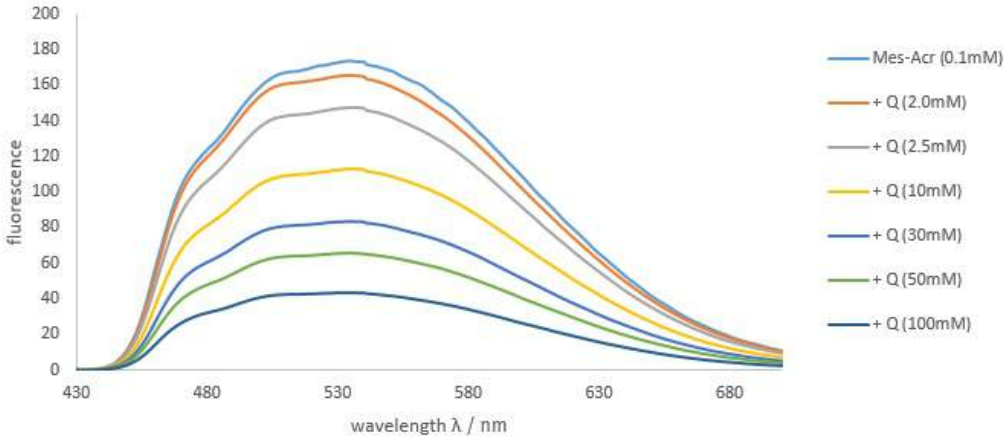
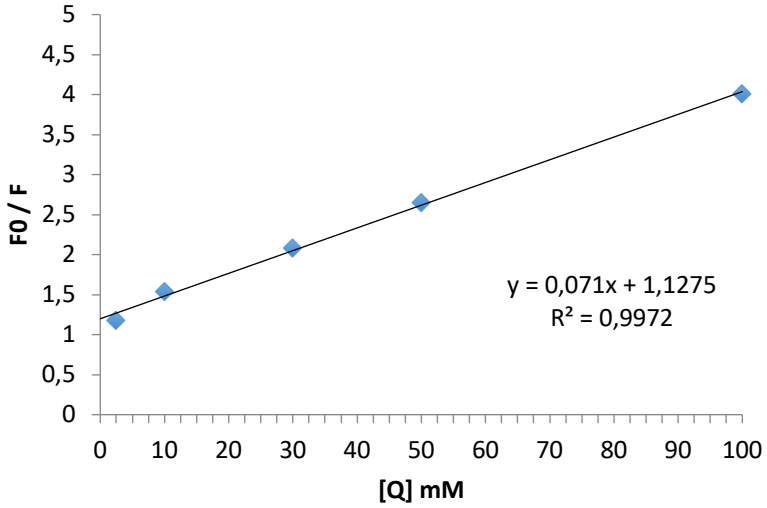


Figure S2b: *Stern-Volmer plot of Mes-Acr* ***I*** *with* ***1a****.*



Entry	**1a**, Q (mM)	Fluorescence Intensity **F** (u.a. @ max. 534 nm)	**F^0 / F**
1	--	173,6	
2	2,0	165,3	1,050
3	2,5	147,3	1,179
4	10	112,9	1,538
5	30	83,3	2,084
6	50	65,6	2,646
7	100	43,3	4,009

Determination of the Reaction Quantum Yield (Φ) for the Desilylative C-C-bond Formation Reaction

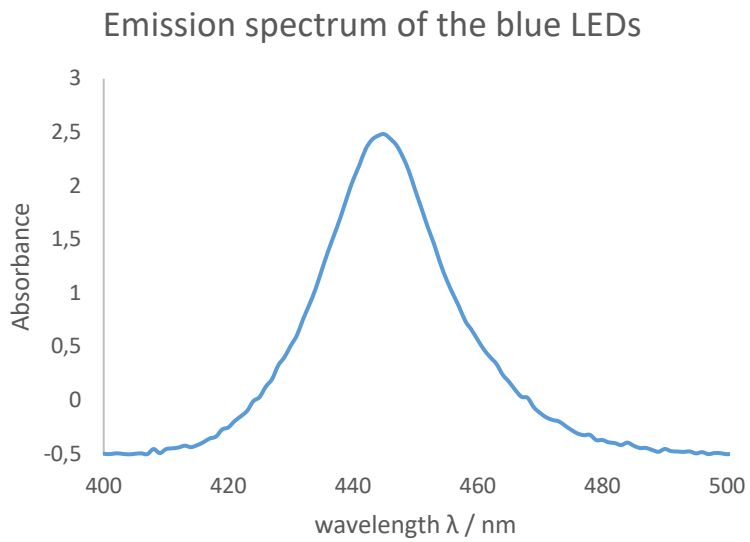


Figure S3: Emission spectrum of the used LED stripes.

Determination of the photon flux at 450 nm *via* ferrioxalate actinometry

According to the procedure of Yoon,[10] the photon flux of the LEDs (λ_{max} = 450 nm) was determined by standard ferrioxalate actinometry. A 0.15 M solution of ferrioxalate was prepared by dissolving potassium ferrioxalate trihydrate (0.737 g) in H_2SO_4 (10 mL of a 0.05 M solution). A buffered solution of 1,10-phenanthroline was prepared by dissolving 1,10-phenanthroline (25 mg) and sodium acetate (5.63 g) in H_2SO_4 (25 mL of a 0.5 M solution). Both solutions were stored in the dark. To determine the photon flux of the LED, the ferrioxalate solution (1.0 mL) was placed in a cuvette and irradiated for 93 seconds at λ_{max} = 450 nm. After irradiation, the phenanthroline solution (0.175 mL) was added to the cuvette and the mixture was allowed to stir in the dark for 1 h to allow the ferrous ions to completely coordinate to the phenanthroline. The absorbance of the solution was measured at 510 nm. A non-irradiated sample was also prepared and the absorbance at 510 nm was measured. Conversion was calculated according to the following formula:

$$mol\ Fe^{2+} = \frac{\mathrm{V} \bullet \Delta\mathrm{A}(510\ \mathrm{nm})}{l \bullet \varepsilon}$$

where V is the total volume (0.001175 L) of the solution after addition of phenanthroline, ΔA is the difference in absorbance at 510 nm between the irradiated and non-irradiated solutions, l is the path length (1.00 cm), and ε is the molar absorptivity of the ferrioxalate actinometer at 510 nm (11,100 $Lmol^{-1}cm^{-1}$). The photon flux can be calculated with the following equation:

$$\text{Photon flux} = \frac{\text{mol } Fe^{2+}}{\Phi \bullet \varepsilon \bullet \mathrm{f}}$$

where Φ is the quantum yield for the ferrioxalate actinometer (0.9 at λ_{ex} = 450 nm),[11] t is the irradiation time (93 s), and f is the fraction of light absorbed at λ_{ex} = 450 nm by the ferrioxalate actinometer. This value is calculated using the following equation, where A(450 nm) is the absorbance of the ferrioxalate solution at 450

nm. An absorption spectrum gave an A(450 nm) value of about 1.8, indicating that the fraction of absorbed light (f) is around 0.984.

$$f = 1 - 10^{-A(450nm)}$$

The photon flux was thus calculated (average of two experiments) to be 5.64×10^{-9} einsteins s^{-1}.

Determination of the reaction quantum yield:

Benzyltrimethylsilane (0.1 mmol, 18.9 µL), *N*-phenylmaleimide (0.15 mmol, 1.5 equiv., 25.9 mg) and 9-mesityl-10-methylacridinium perchlorate **I** (4.2 mg, 0.01 mmol, 10 mol%) were weight in a cuvette. The substrates were dissolved in chloroform and the reaction mixture was irradiated in the photoreactor wrapped by a stripe of blue LEDs for 1 h. Dibromomethane (0.1 mmol, 6.98 µL) was added as an internal standard and the yield was determined by analysis of the crude mixture by ^{1}H-NMR, which gave the desired product in 9% of yield (1.87 x 10^{-5} mol). The reaction quantum yield could be calculated with the following formula:

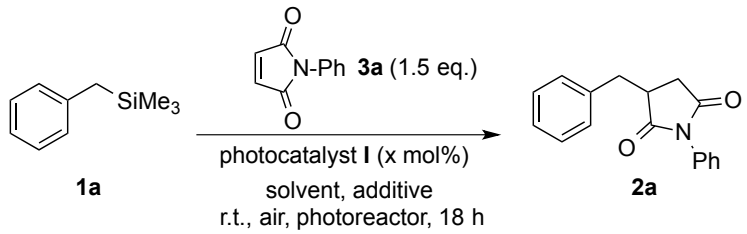
$$\Phi = \frac{\text{mol of formed product}}{\text{photon flux} \bullet t \bullet f}$$

with a photon flux of 5.64×10^{-9} einsteins s^{-1}, t = 3600 s and f > 0.999.

The reaction quantum yield (Φ) was determined as 0.92.

Mechanistic Experiments

Table S7. Control experiments[a]

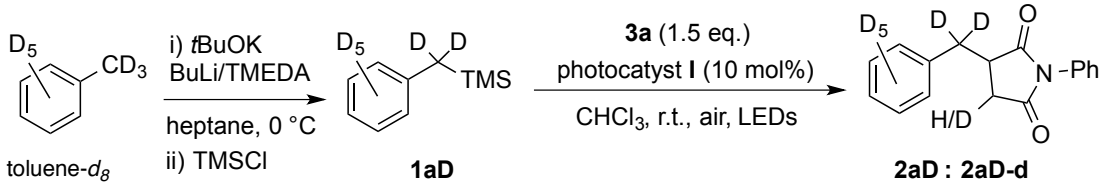


Entry	Cat. **I** (x mol%)	Solvent	Additive	Yield (%)[b]
1	--	$BrCCl_3$	--	--
2	10	$BrCCl_3$	--	traces
3[c]	--	$CHCl_3$	HCl conc.	--
4	10	MeCN	--	8
5[c]	10	MeCN	HCl conc.	52
6[c]	10	MeCN	$HClO_4$ conc.	24
7[d]	--	$CHCl_3$	H_2O_2	--
8[e]	10	$CHCl_3$	--	--

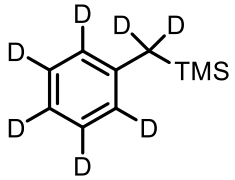
[a] All reactions were performed in 0.2 mmol scale. [b] Yields after flash column chromatography. [c] 65 µL of aq. concentrated acid was added. [d] 1 mL of hydrogenperoxide (50 wt. % in H_2O) was added. [e] This reaction was performed in the dark.

Deuterium experiments:

A) Reaction with the deuterated substrate 1aD

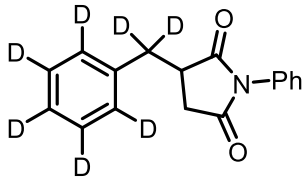


((Phenyl-d5)methyl-d2)trimethylsilane (1aD)[9]



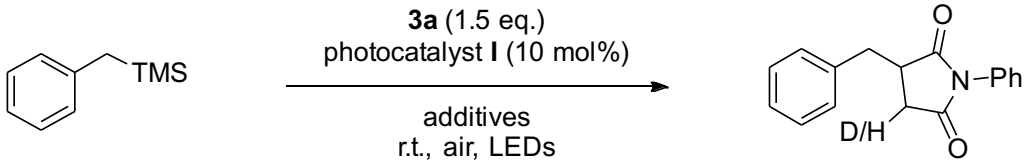
The deuterated starting material was synthesized according to a modified literature procedure.[6] *t*BuOK (336 mg, 3 mmol, 1.0 equiv.) was added to dry heptane (3 mL) under Ar-atmosphere and cooled down to 0 °C. *n*-BuLi (2.5 M in hexane, 1.3 mL, 3 mmol, 1.0 equiv.)) and 2,2,6,6-tetramethylpiperidine (0.5 mL, 3 mmol, 1.0 equiv.) were added dropwise at 0 °C. A solution of toluene-d_8 (160 µL, 1.5 mmol) in heptane (1 mL) was added to the mixture and stirred for 15 minutes at the given temperature. Trimethylsilyl chloride (476 µL, 3.75 mmol, 1.25 equiv.) was given into the reaction and stirred for an hour at 0 °C. The reaction was quenched with methanol-d_4 and extracted with diethyl ether (3x). The combined organic phases were washed with water (1x) and brine (1x) and dried over $MgSO_4$. The crude mixture was purified by flash column chromatography with pentane as eluent to give to desired product as colorless oil (118 mg, 0.7 mmol, 67%). **^{1}H-NMR** (300 MHz, $CDCl_3$): δ 0.01 (s, 9H). **HRMS** (FT-MS): Mass calc. for $C_{10}H_9D_7SiH$ [M+H$^+$]: *m/z* = 172.15334, found: *m/z* = 172.15291. The spectra were in accordance with the ones in the literature.[9]

1-Phenyl-3-((phenyl-d5)methyl-d2)pyrrolidine-2,5-dione (2aD)



According to general procedure, the reaction of **1aD** (34.2 mg, 0.20 mmol, 1.0 equiv.) with **3a** (51.9 mg, 0.30 mmol, 1.5 equiv.) provided the title compound after flash column chromatography (petroleum ether/ethyl acetate 9:1 → 8:2) as a white solid (18.9 mg, 0.07 mmol, 35%). **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 7.52 – 7.35 (m, 3H), 7.21 – 7.13 (m, 2H), 3.37 – 3.28 (m, 1H), 2.88 (dd, *J* = 18.5, 9.2 Hz, 1H), 2.65 (dd, *J* = 18.5, 4.7 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 178.3, 175.3, 136.4, 131.8, 129.2, 128.9 (d, *J* = 22.0 Hz), 128.7, 128.3 (d, *J* = 24.6 Hz), 126.9 (d, *J* = 24.3 Hz), 126.5, 41.1, 35.8 (t, *J* = 16.8 Hz), 33.3. **HRMS** (ESI-MS): Mass calc. for $C_{17}H_8D_7NO_2Na$ [M+Na$^+$]: *m/z* = 295.1434 found: *m/z* = 295.1436.

B) Reaction in $CDCl_3$, D_2O and DCl

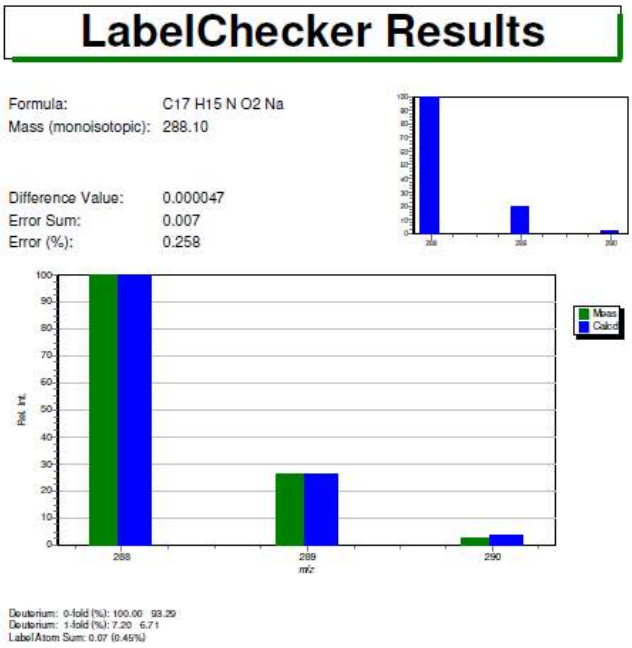


1a **2a : 2a-d**

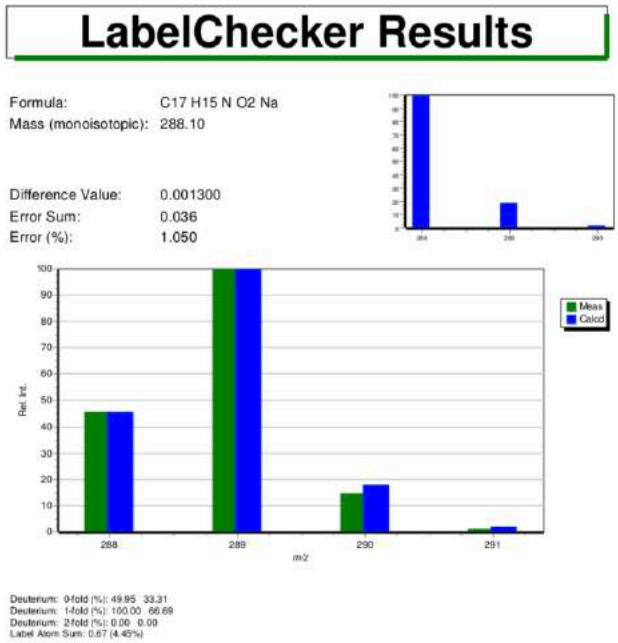
A in $CDCl_3$	94 : 6
B in $CHCl_3$, DCl (D_2O)	33 : 67
C in dry MeCN, DCl (D_2O)	9 : 91
D in dry $CHCl_3$, DCl (D_2O)	22 : 78
E in dry $CDCl_3$	99.9 : 0.1

HRMS D-Content Analysis for experiments A - E:

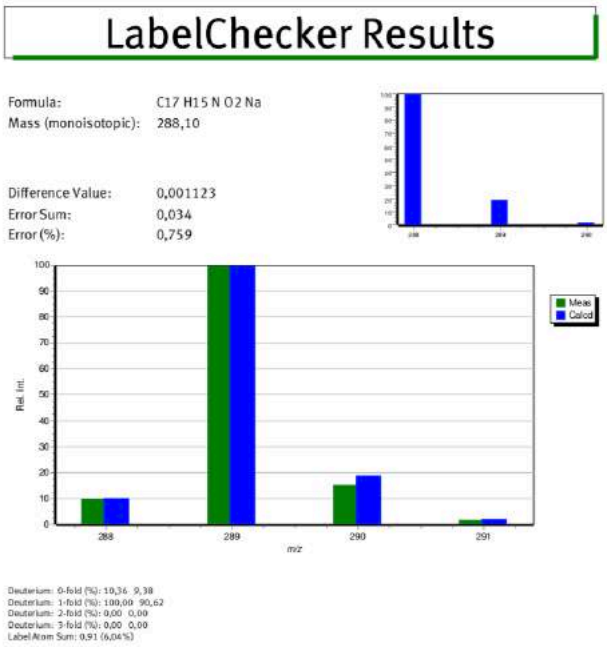
A



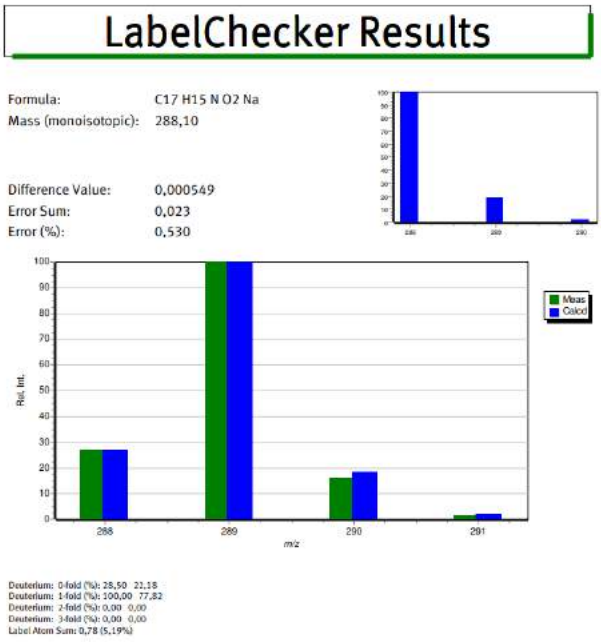
B



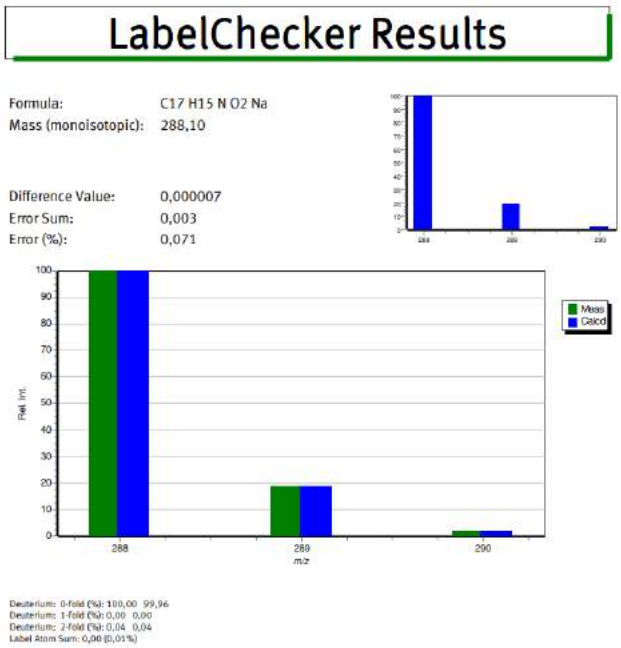
C



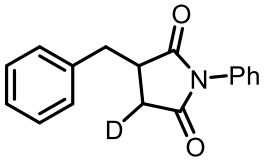
D



E



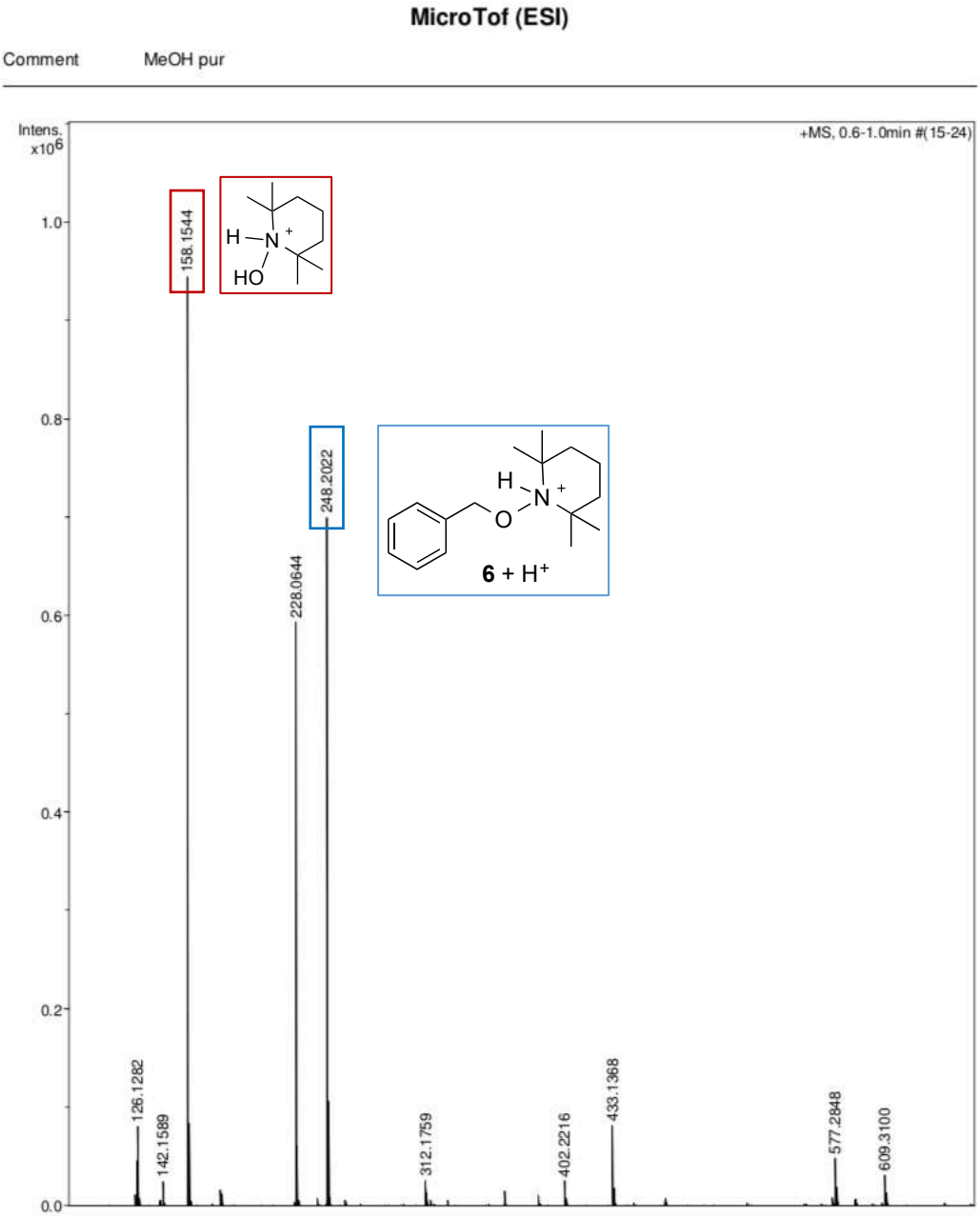
3-Benzyl-1-phenylpyrrolidine-2,5-dione-4-d (2a-d)



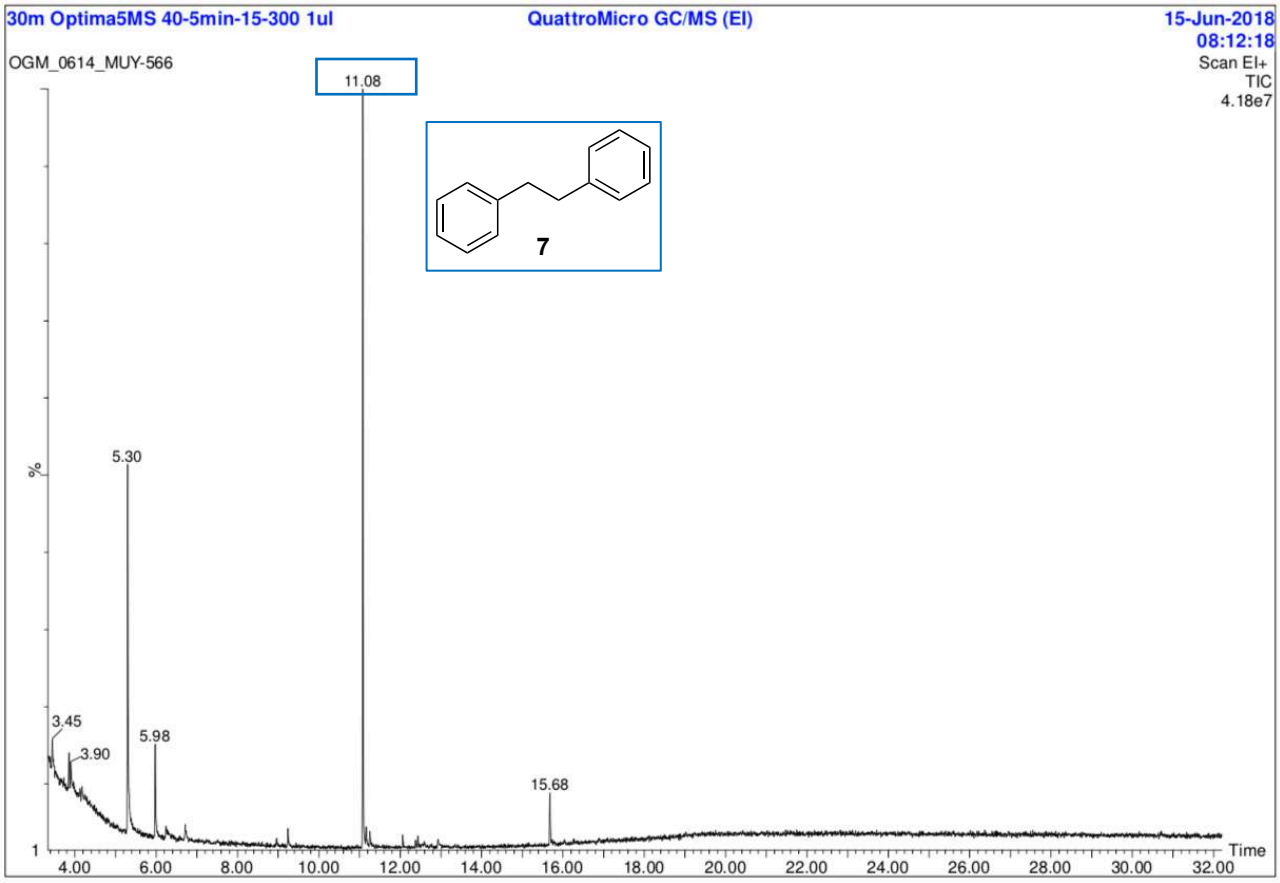
1a (37.8 µL, 0.20 mmol, 1.0 equiv.), **3a** (51.9 mg, 0.30 mmol, 1.5 equiv.) and catalyst **I** (8.4 mg, 0.02 mmol, 10 mol%) were dissolved in MeCN (2 mL) under air. A solution of DCl in D_2O (20 wt%, 50 µL) was added and the mixture was irradiated in the photoreactor for 18 h. The solvent was evaporated and the crude mixture was purified by flash column chromatography with pentane/ethyl acetate 9:1 → 8:2 to give a mixture of **2a** and deuterated product **2a-d** as a white solid (8.0 mg, 0.03 mmol, 15%) in a 9:91 ratio (determined by HRMS analysis), which suffered a fast D/H exchange in $CDCl_3$ in the NMR probe to lead to the same 33:67 ratio obtained in the reaction in $CHCl_3$/DCl. **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 7.49 – 7.42 (m, 2H), 7.41 – 7.27 (m, 4H), 7.24 – 7.14 (m, 4H), 3.37 – 3.29 (m, 1H), 3.25 (dd, *J* = 14.0, 4.5 Hz, 1H), 3.09 (dd, *J* = 13.7, 8.0 Hz, 1H), 2.63 (br.s., 1H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ 177.3, 174.3, 135.7, 130.8, 128.2, 128.2, 127.9, 127.6, 126.3, 125.4, 40.2, 35.6, 32.5 – 31.5 (m). **HRMS** (ESI-MS): Mass calc. for $C_{17}H_{14}DNO_2Na$ [M+Na$^+$]: *m/z* = 289.1058, found: *m/z* = 289.1064.

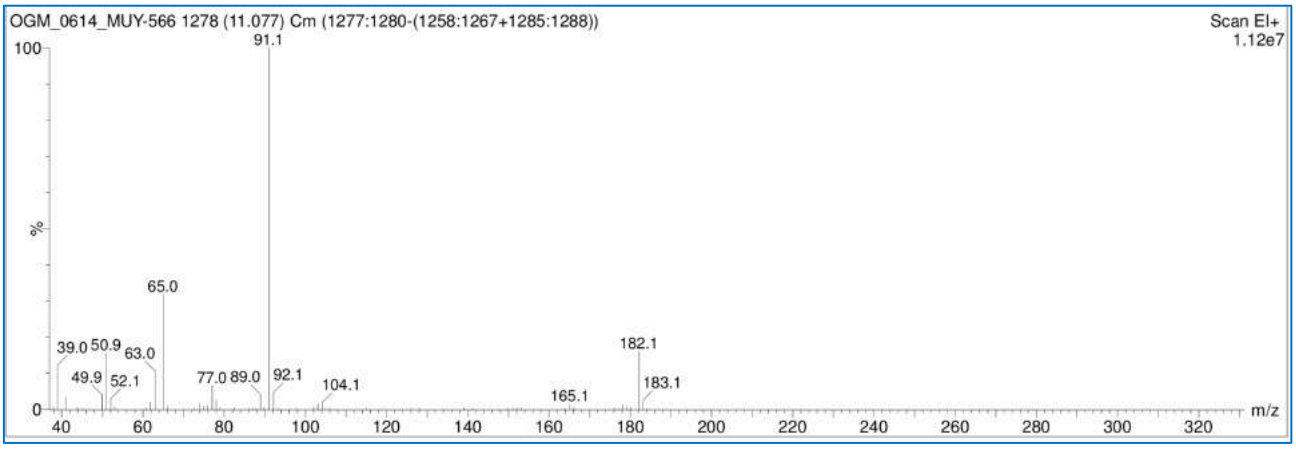
GC-MS Analysis: Observation of radical coupling products

TEMPO-trapping Product 6:



Homocoupling Product 7:





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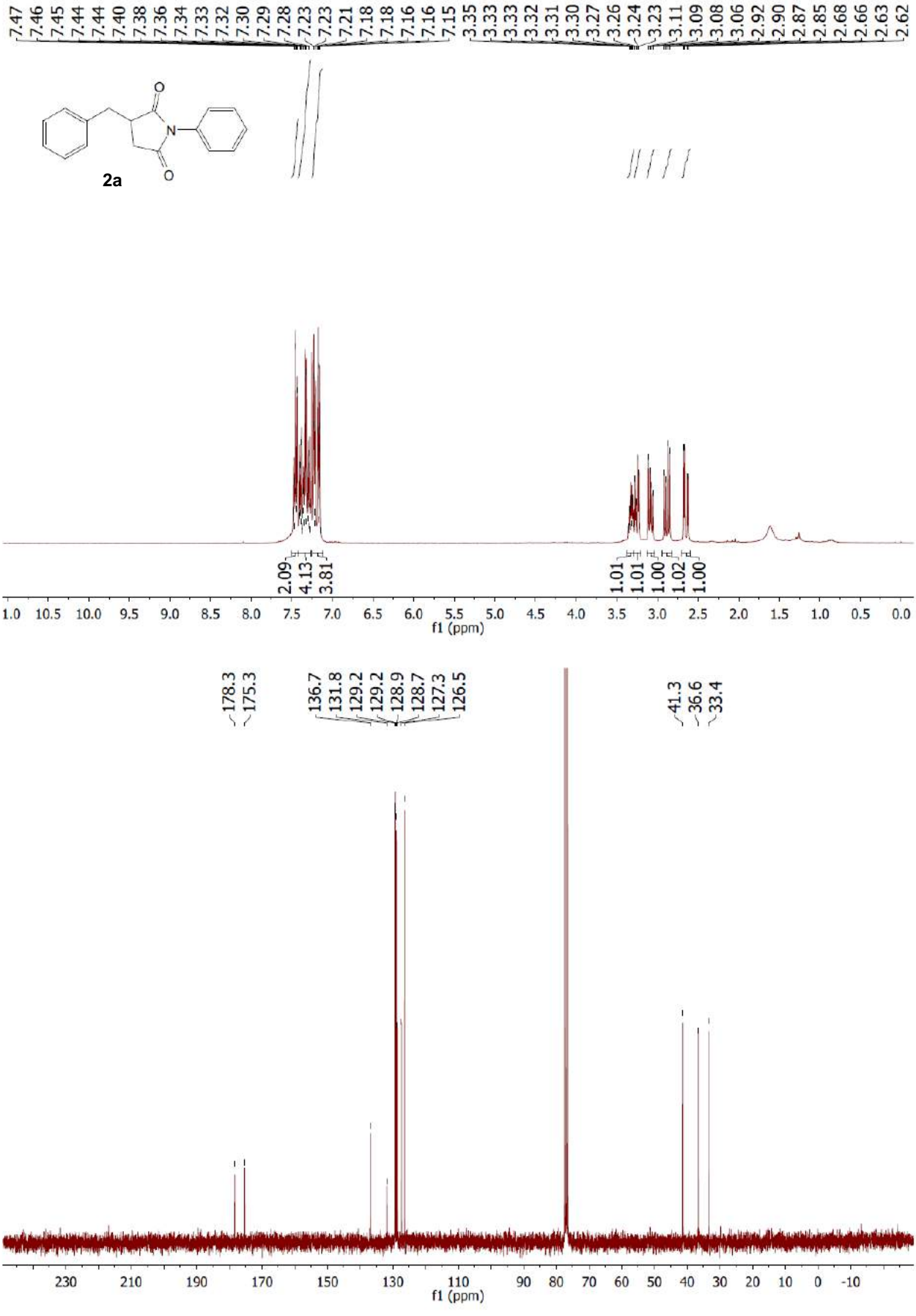
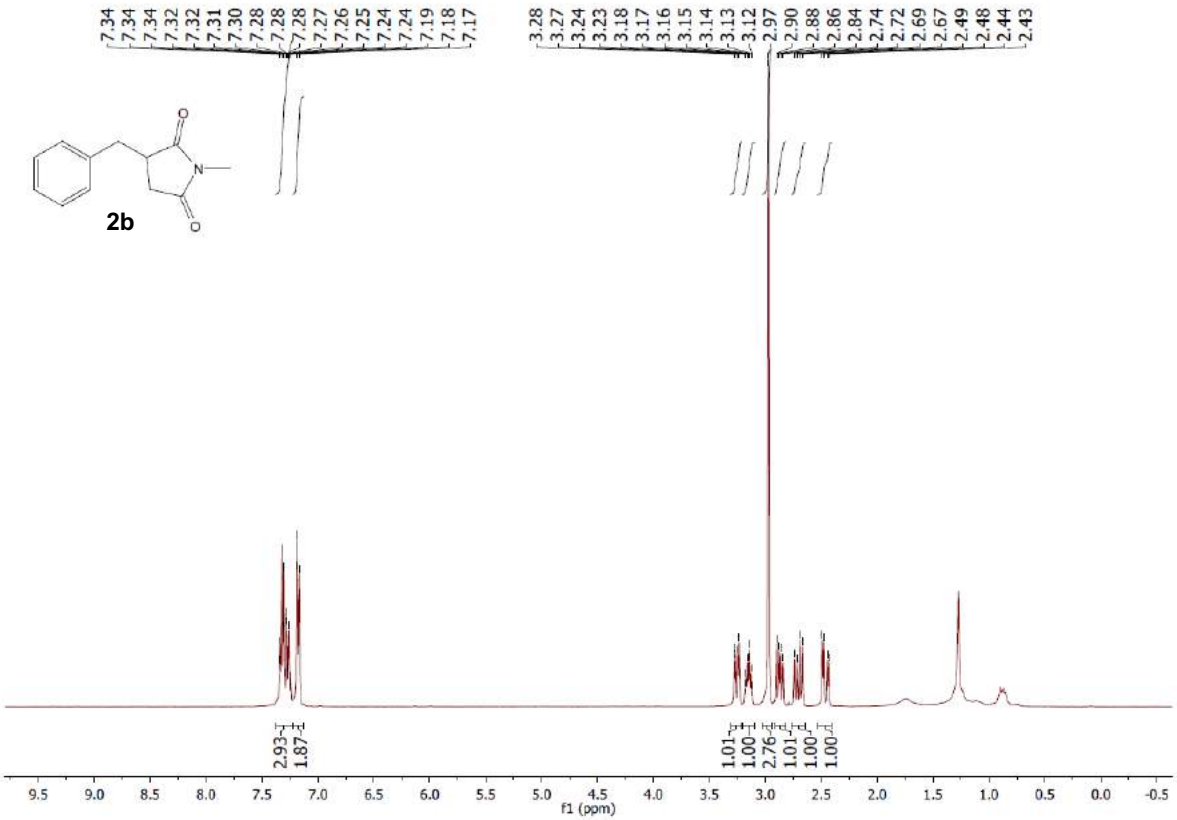
NMR Spectra Collection

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7.28
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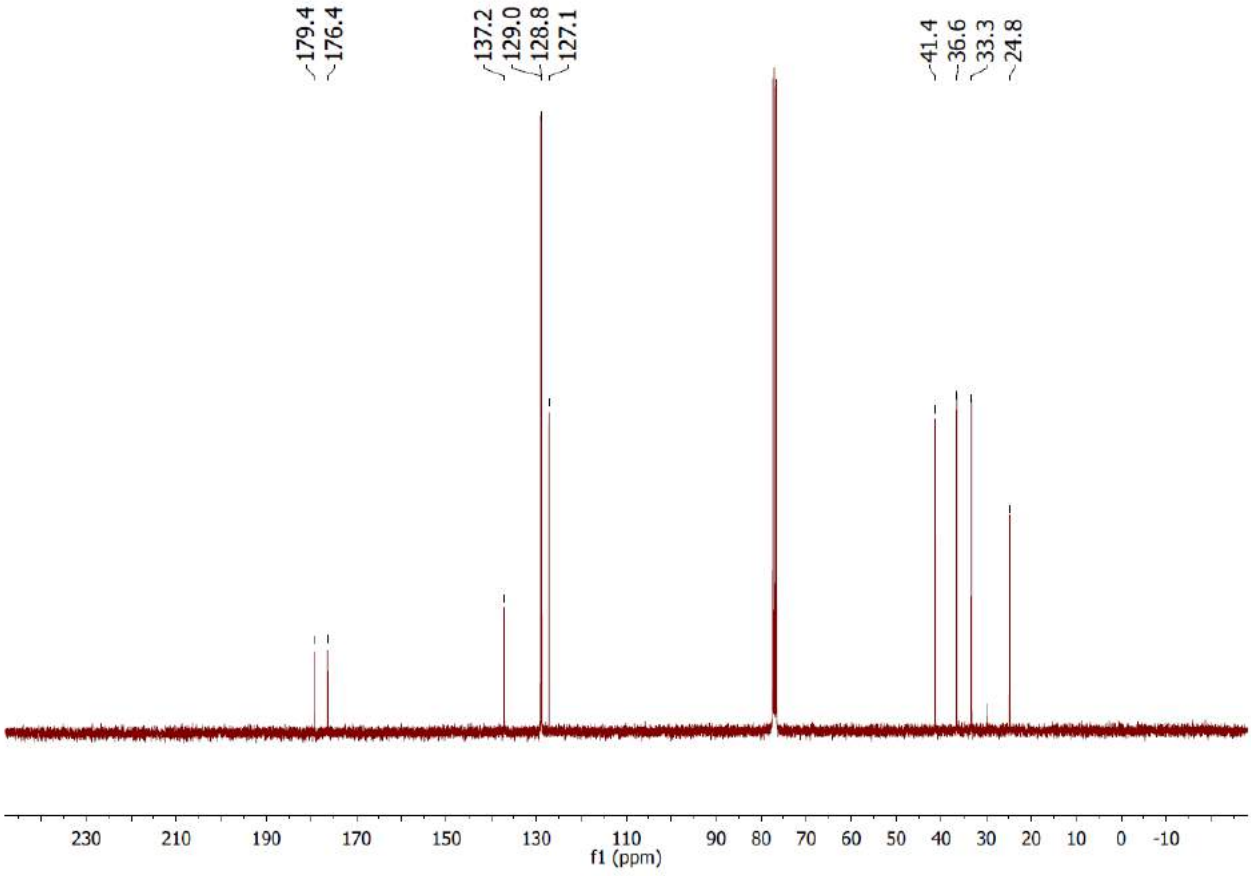
7.28
7.27
7.26
7.25
7.24
7.24
7.19
7.18
7.17
3.28
3.27
3.24
3.23
3.18
3.17
3.16
3.15
3.14
3.13
3.12
2.97
2.90
2.88
2.86
2.84
2.74
2.72
2.69
2.67
2.49
2.48
2.44
2.43
O
N
O
2b
2.93
1.87
1.01
1.00
2.76
1.01
1.00
1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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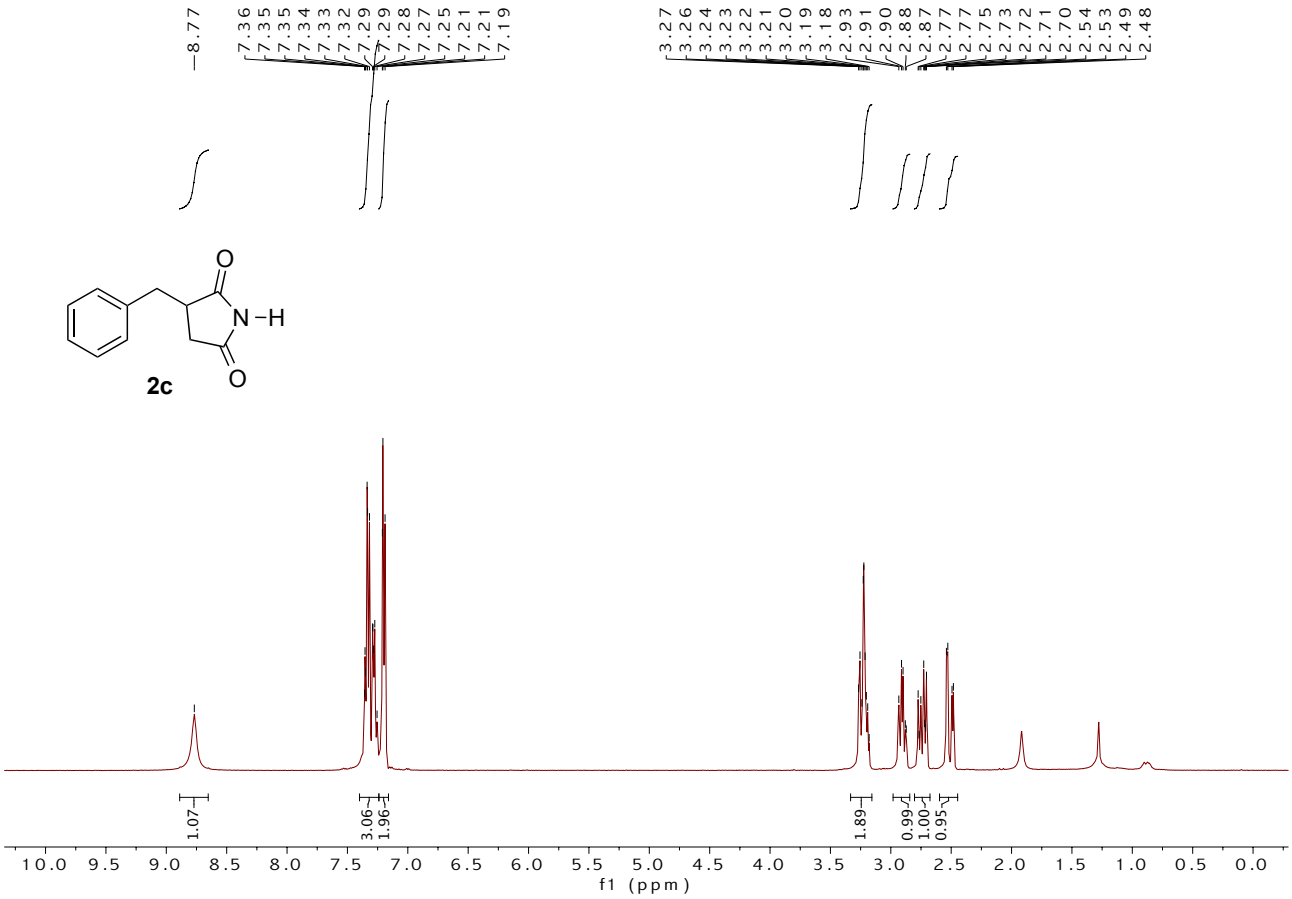
176.4
137.2
129.0
128.8
127.1
41.4
36.6
33.3
24.8
230 210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.35
7.35
7.34
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7.32
7.29
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7.21
7.21
7.19
3.27
3.26
3.24
3.23
3.22
3.21
3.20
3.19
3.18
2.93
2.91
2.90
2.88
2.87
2.77
2.77
2.75
2.73
2.72
2.71
2.70
2.54
2.53
2.49
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N–H
O
2c
1.07
3.06
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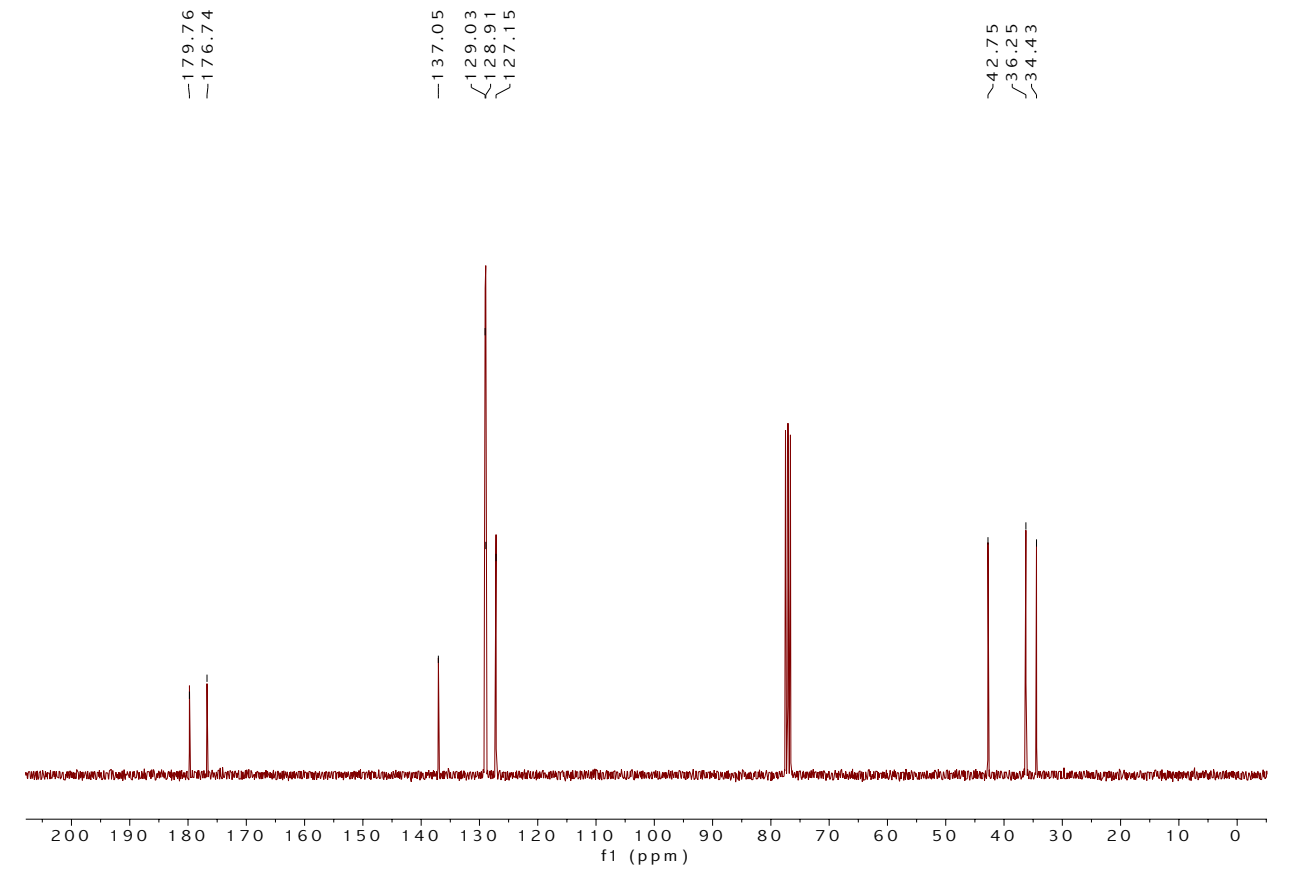
1.89
0.99
1.00
0.95
f1 (ppm)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34.43
f1 (ppm)


7.33
7.31
7.30
7.30
7.28
7.28
7.28
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7.19
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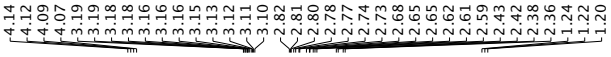
7.18
7.17
7.17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3.13
3.12
3.11
3.10
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2.81
2.80
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2.77
2.74
2.73
2.68
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2.38
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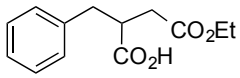
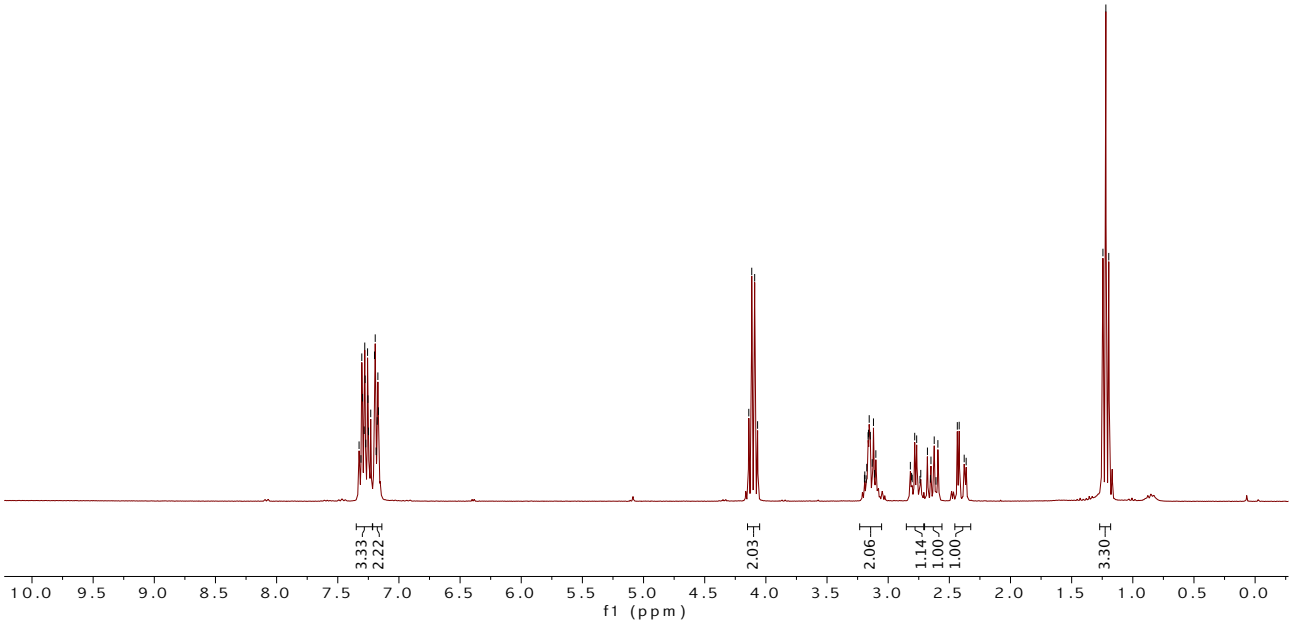
1.24
1.22
1.20


CO2Et
CO2H

2d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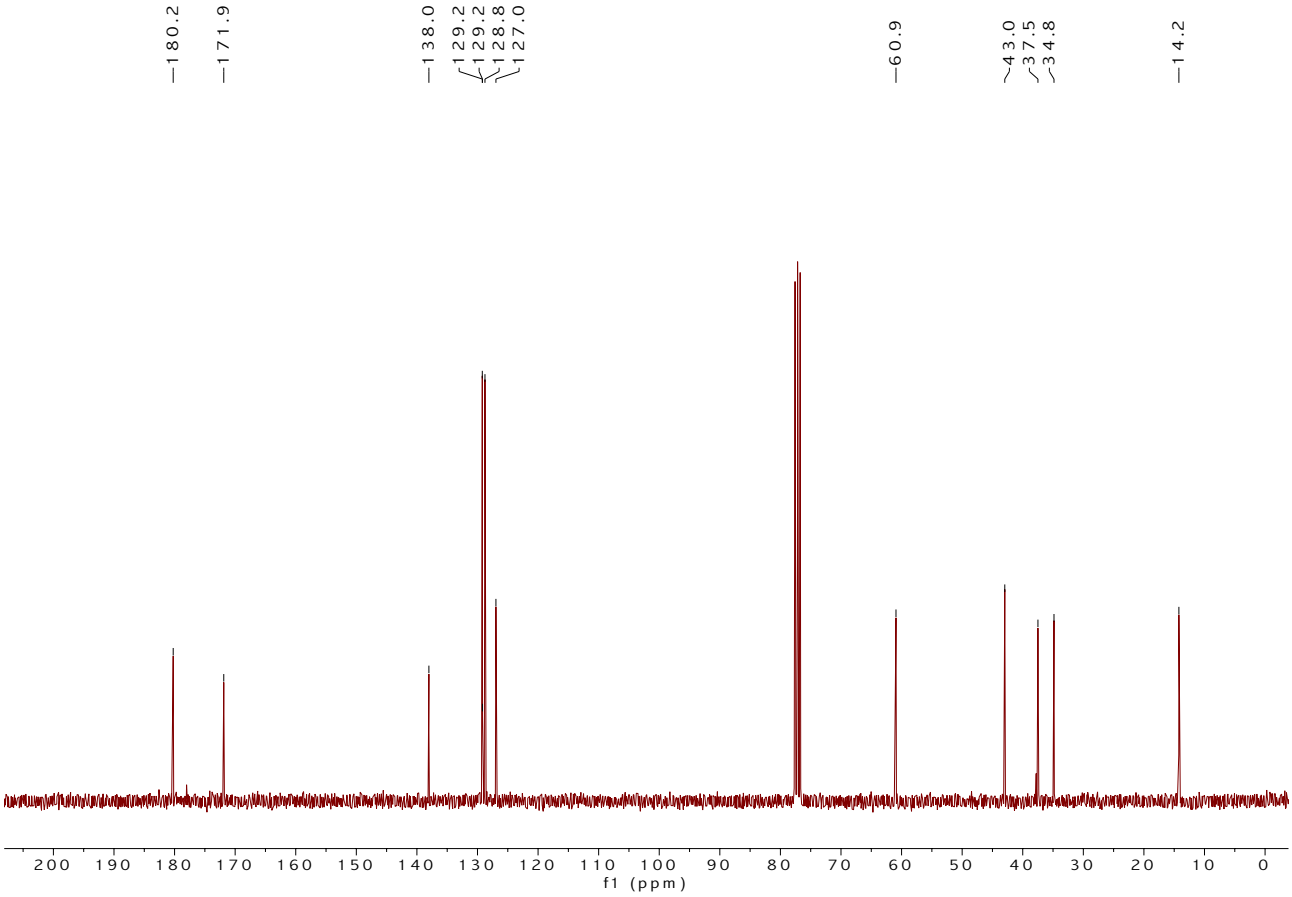
2.03
2.06
1.14
1.00
1.00
3.30
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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171.9
138.0
129.2
129.2
128.8
127.0
60.9
43.0
37.5
34.8
14.2
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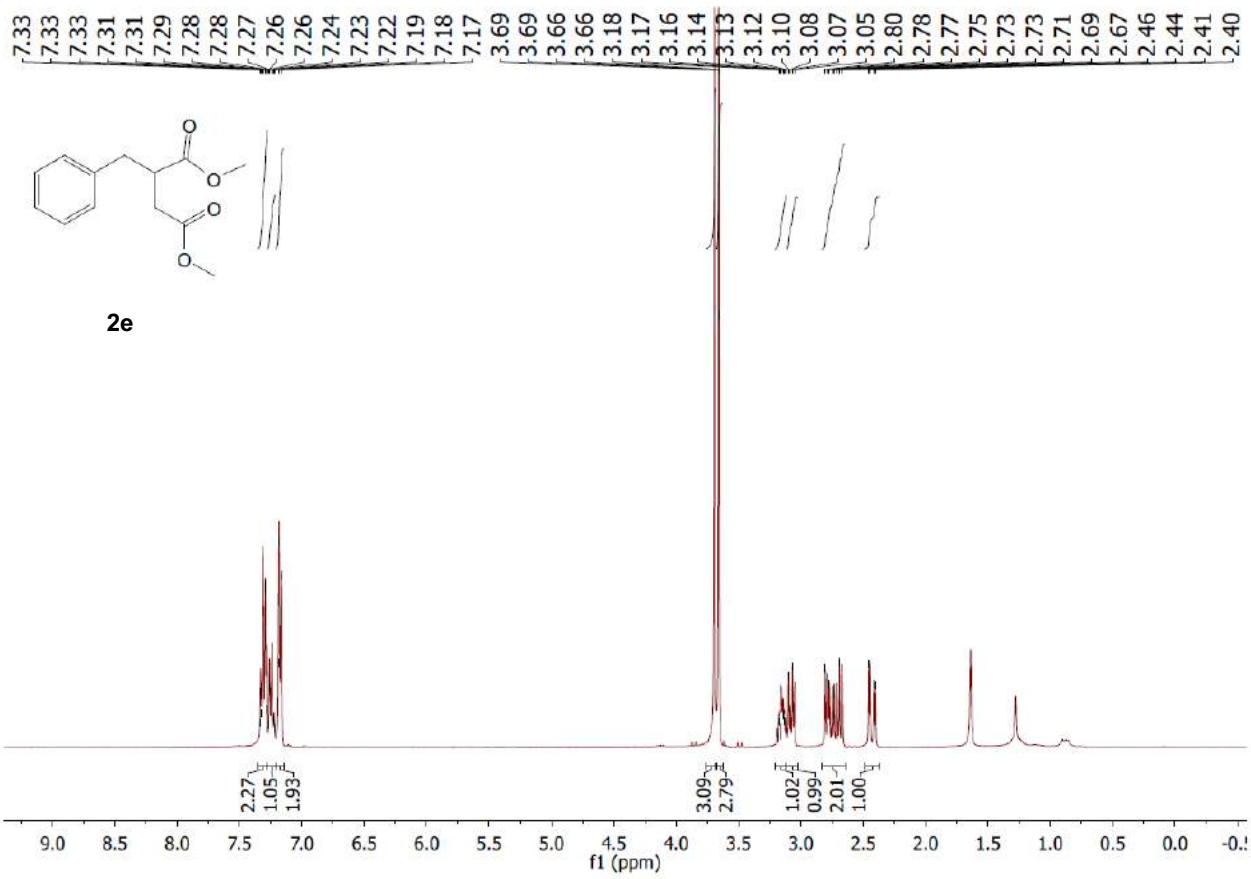
f1 (ppm)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7.23
7.22
7.19
7.18
7.17
3.69
3.69
3.66
3.66
3.18
3.17
3.16
3.14
3.13
3.12
3.10
3.08
3.07
3.05
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2.73
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2.69
2.67
2.46
2.44
2.41
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3.09
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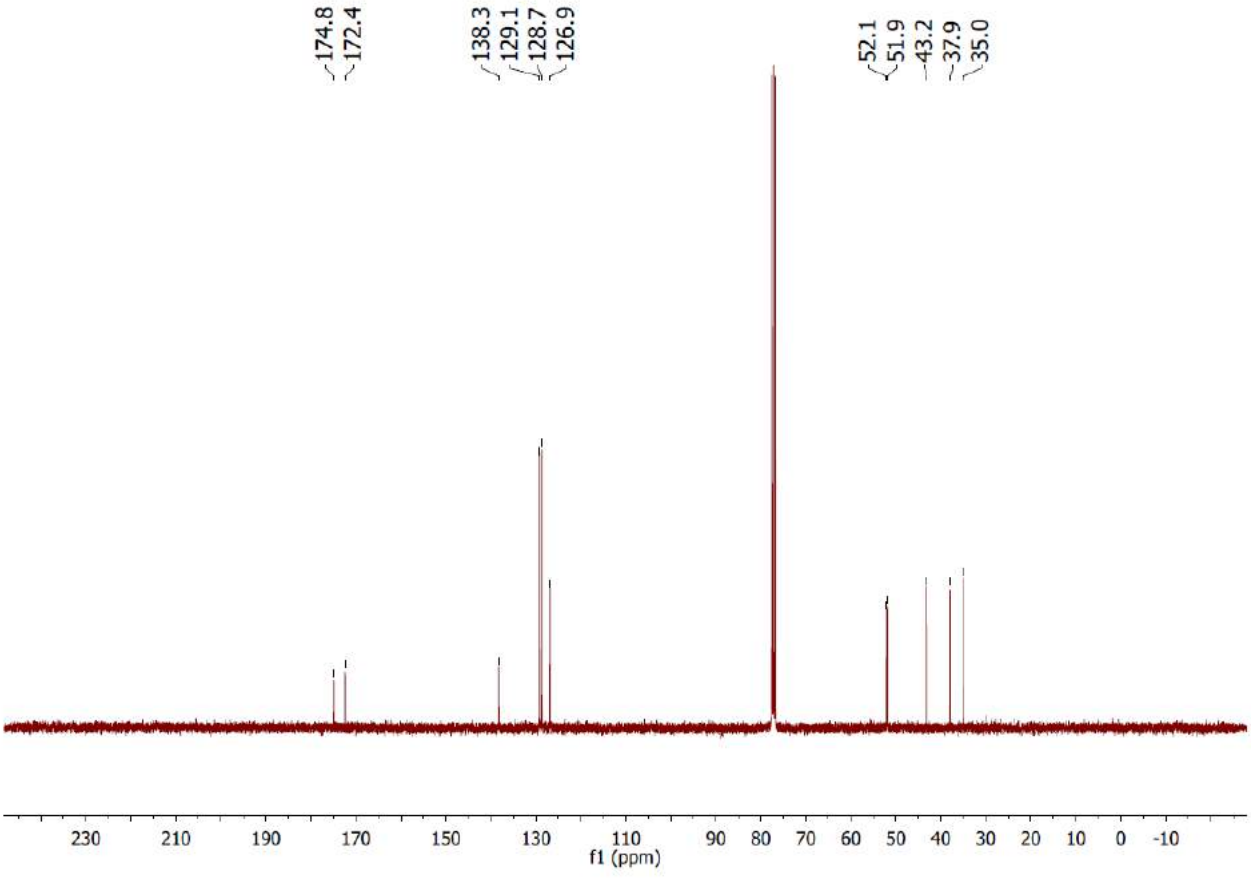
2.01
1.00
f1 (ppm)

2e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52.1
51.9
43.2
37.9
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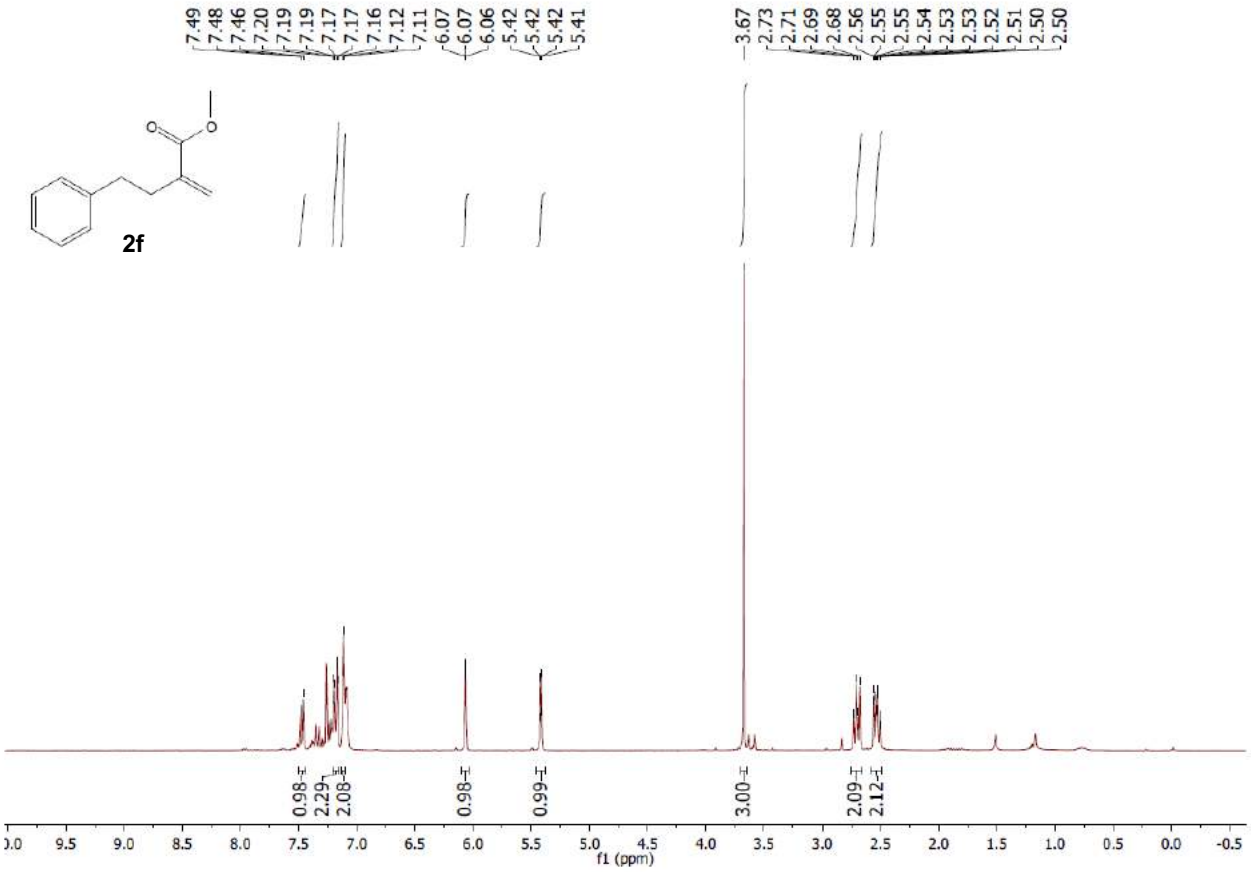
f1 (ppm)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7.17
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7.12
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6.06
5.42
5.42
5.41
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2.55
2.55
2.54
2.53
2.53
2.52
2.51
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2.50
2f
0.98
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0.98
0.99
3.00
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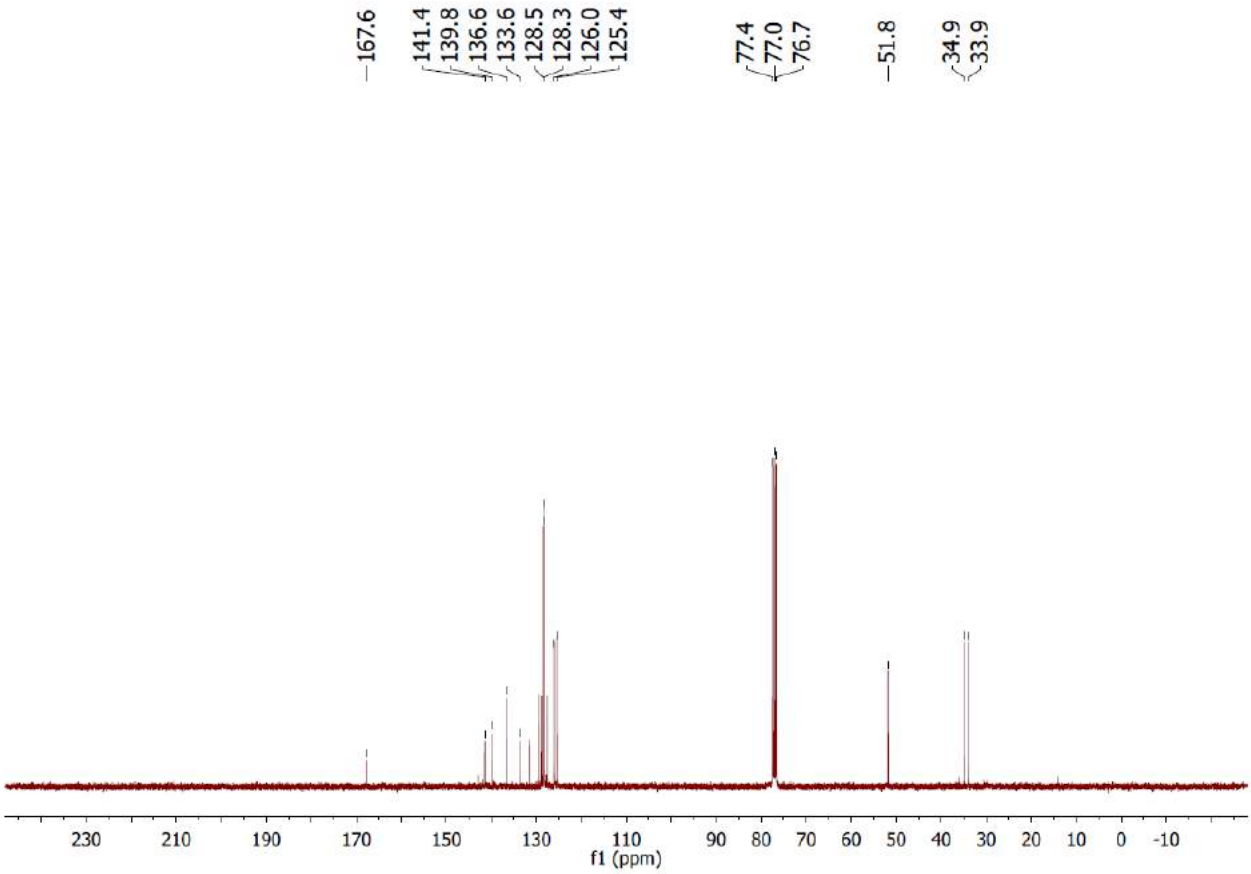
2.12
f1 (ppm)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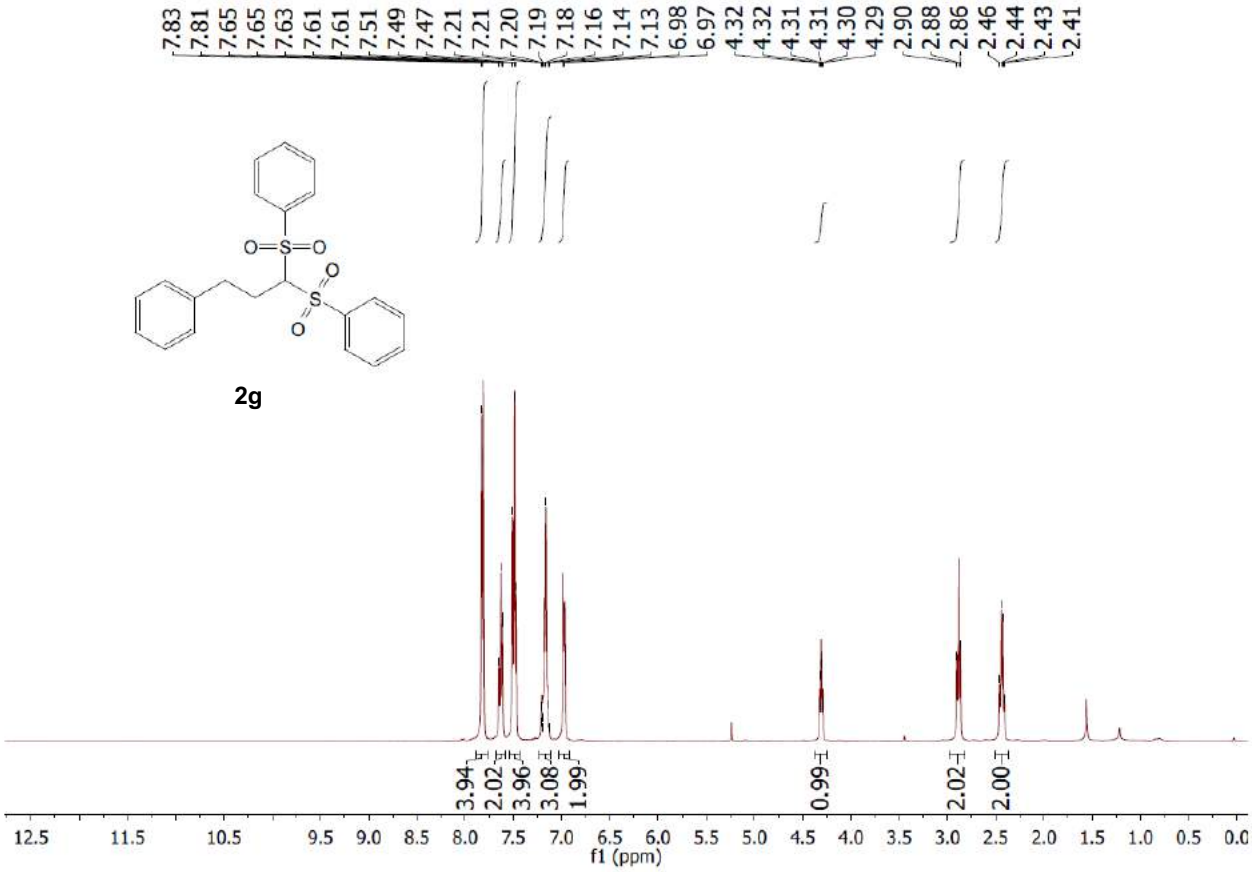
51.8
34.9
33.9
f1 (ppm)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7.20
7.19
7.18
7.16
7.14
7.13
6.98
6.97
4.32
4.32
4.31
4.31
4.30
4.29
2.90
2.88
2.86
2.46
2.44
2.43
2.41
O=S=O
O
S
O
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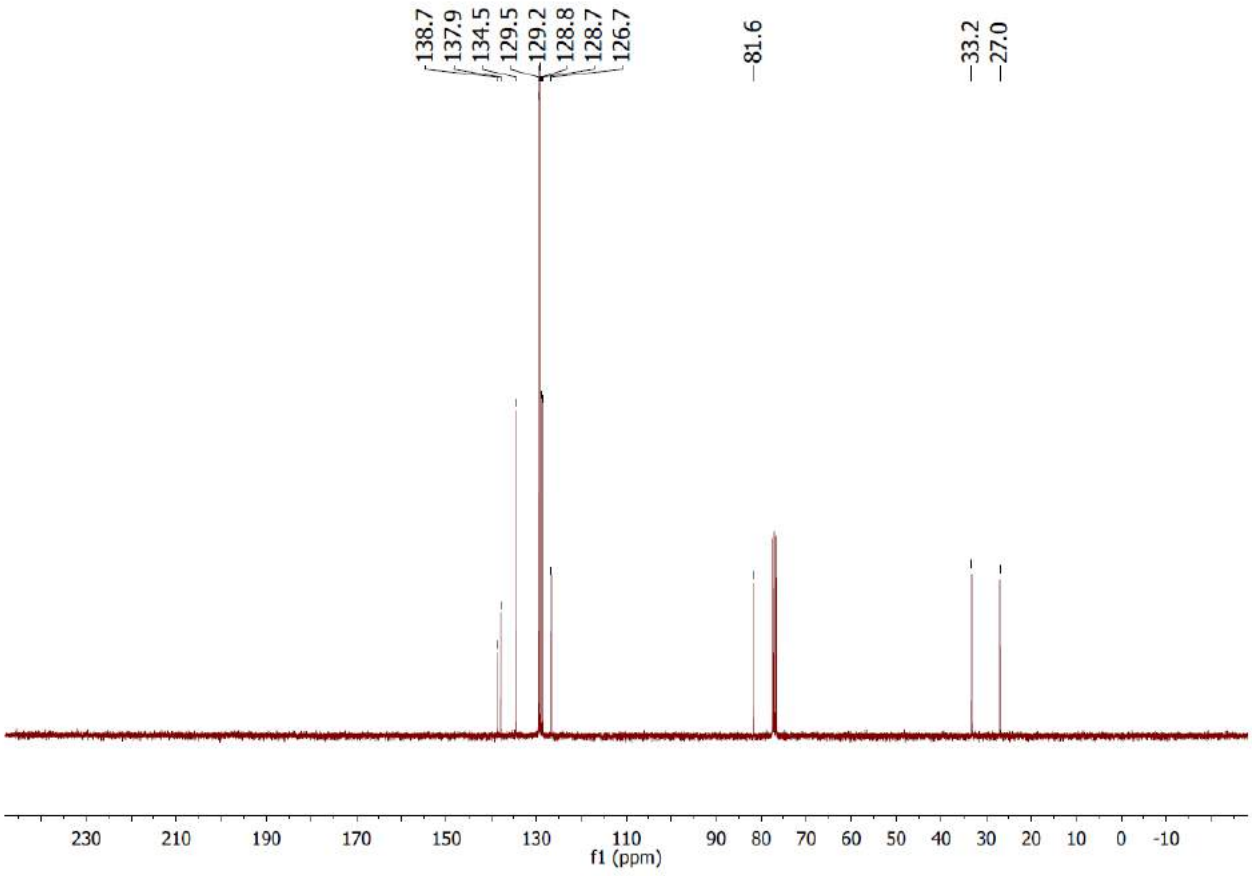
2.02
3.96
3.08
1.99
0.99
2.02
2.00
f1 (ppm)

2g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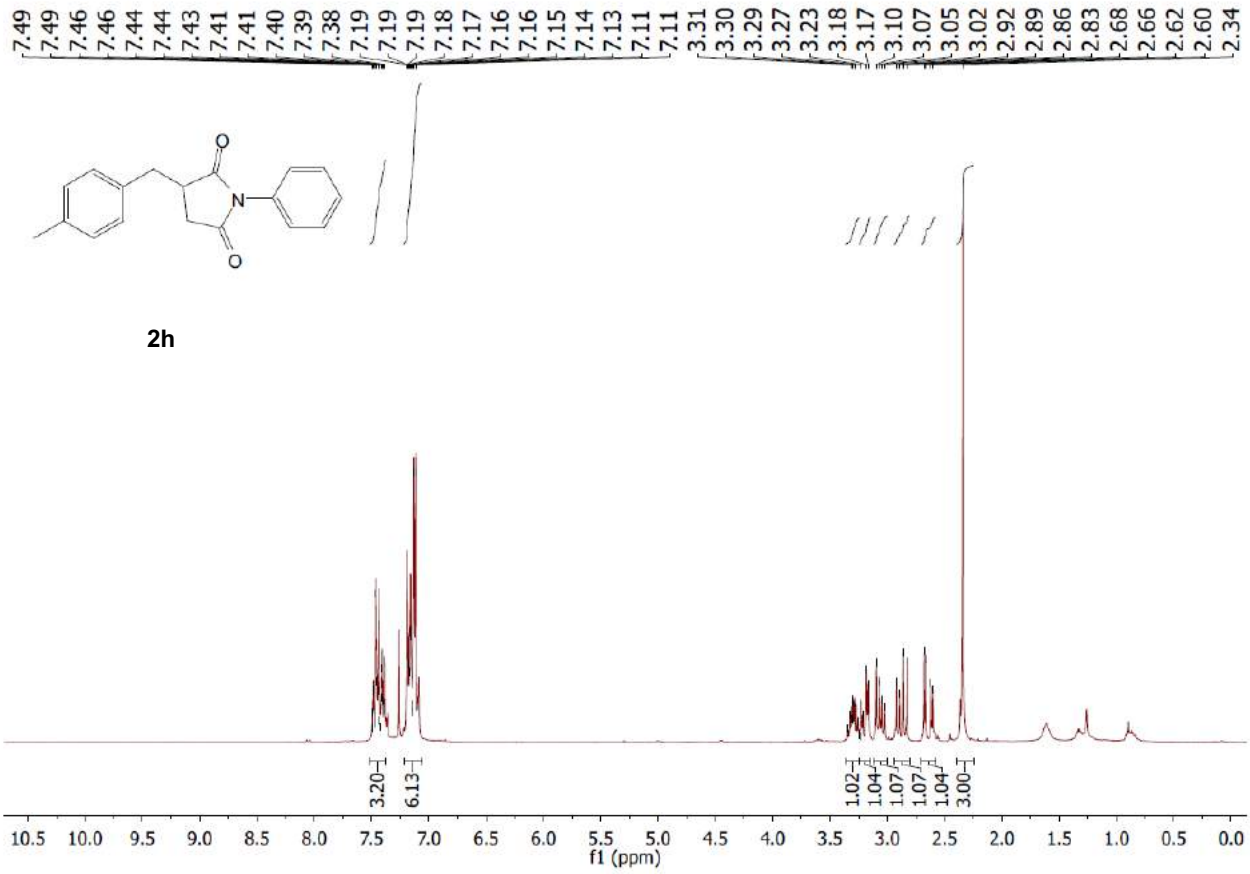
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7.17
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7.16
7.15
7.14
7.13
7.11
7.11
3.31
3.30
3.29
3.27
3.23
3.18
3.17
3.10
3.07
3.05
3.02
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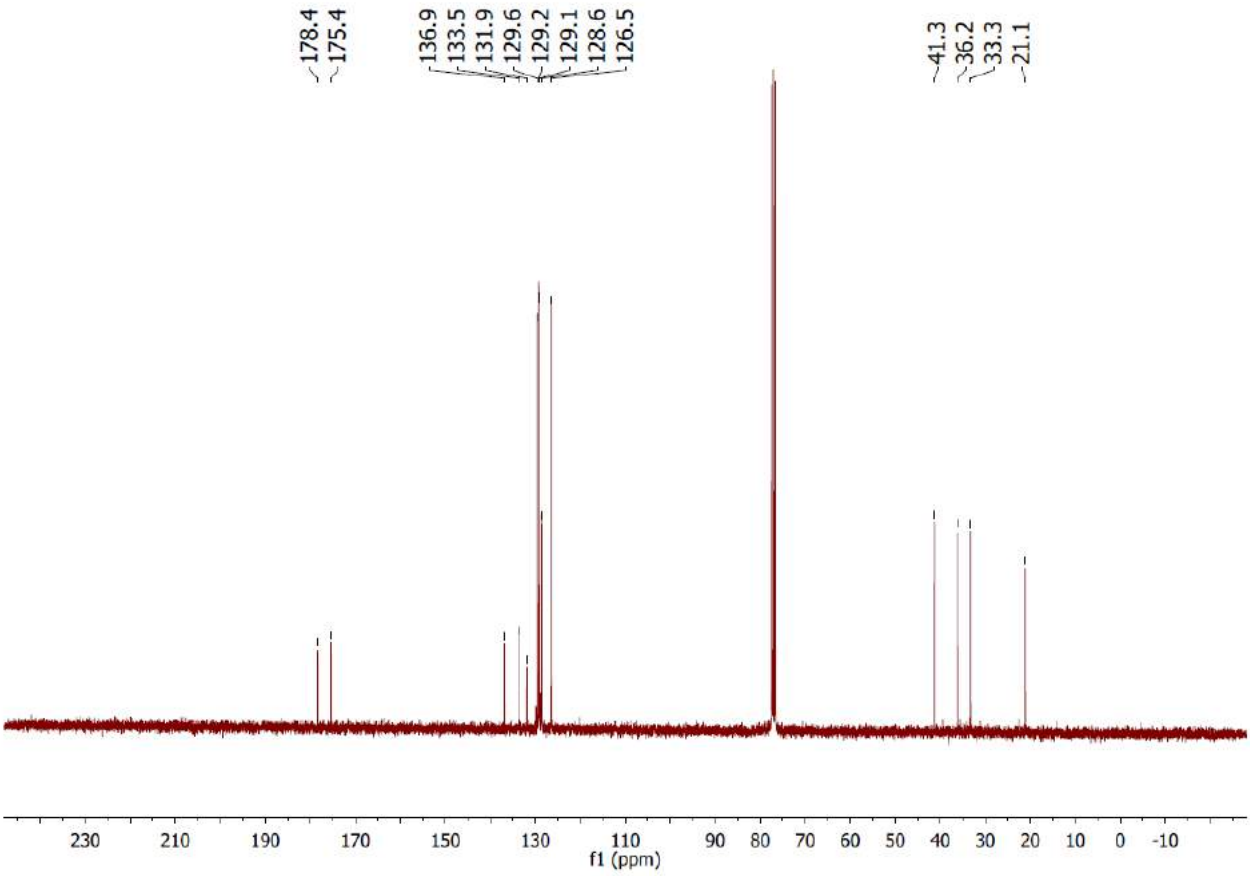
2.89
2.86
2.83
2.68
2.66
2.62
2.60
2.34
O
N
O
3.20
6.13
1.02
1.04
1.07
1.07
1.04
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

2h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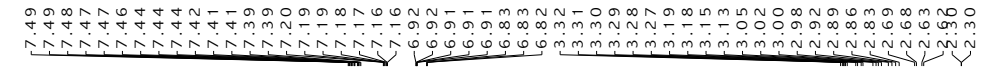
133.5
131.9
129.6
129.2
129.1
128.6
126.5
41.3
36.2
33.3
21.1
230 210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.39
7.20
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7.19
7.18
7.17
7.16
7.16
6.92
6.92
6.91
6.91
6.91
6.83
6.83
6.82
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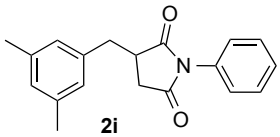
2.30


O
N
O
O

2i

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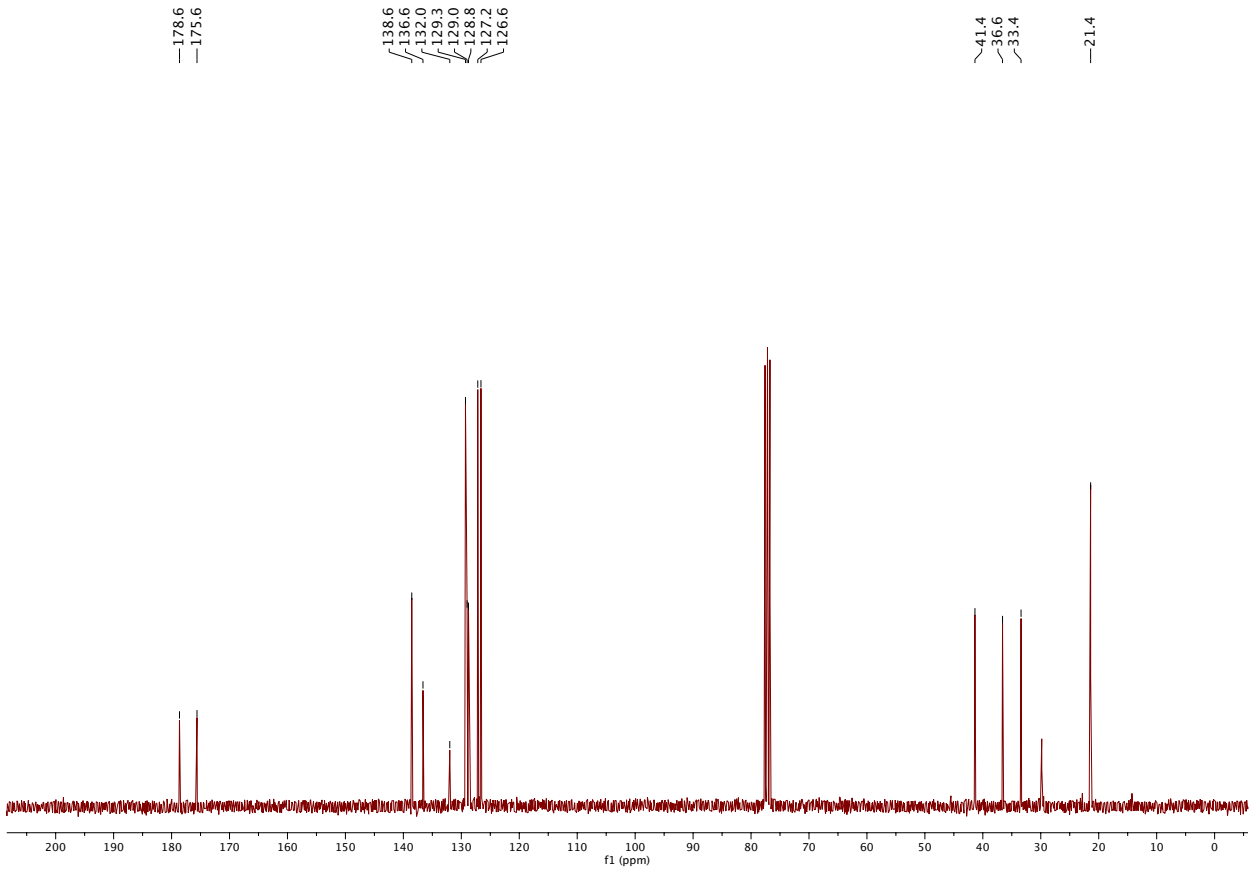
1.00
1.96
1.02
1.00
1.03
1.05
1.03
6.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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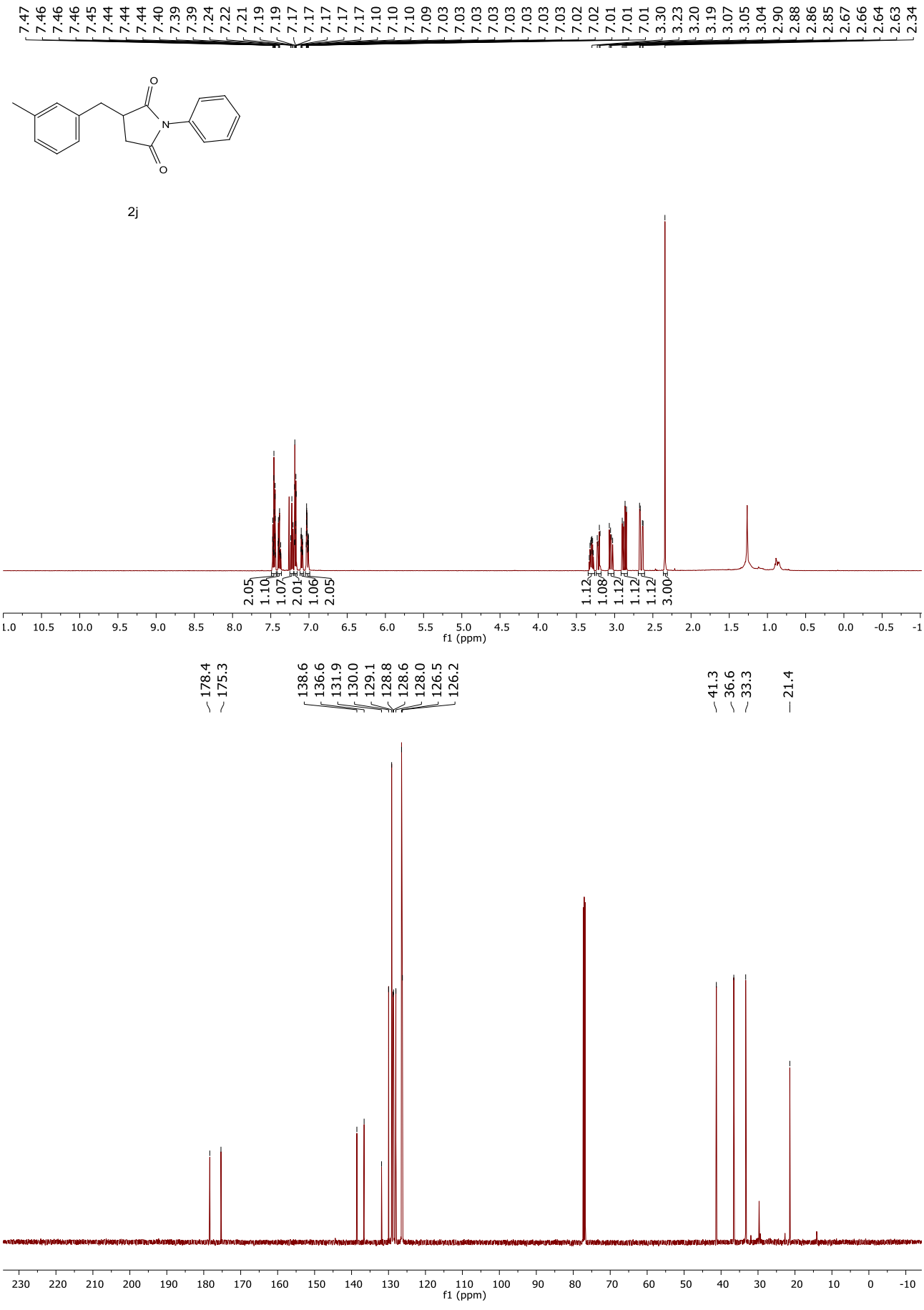
138.6
136.6
132.0
129.3
129.0
128.8
127.2
126.6
41.4
36.6
33.4
21.4
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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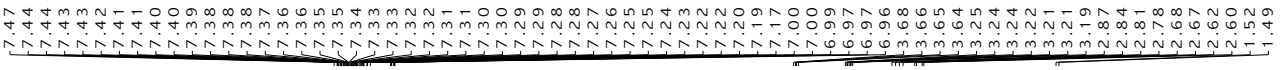
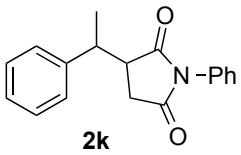
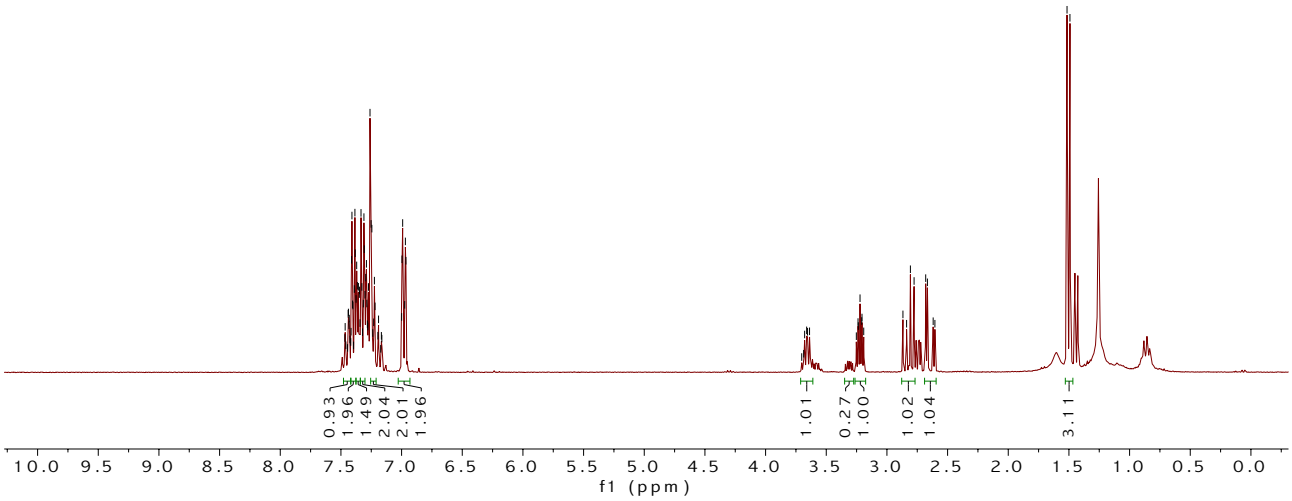
O
N
O
2j
2.05 1.10 1.07 2.01 1.06 2.05
1.12 1.08 1.12 1.12 3.00
f1 (ppm)
178.4 175.3
138.6 136.6 131.9 130.0 129.1 128.8 128.6 128.0 126.5 126.2
41.3 36.6 33.3
21.4
f1 (ppm)




O
N–Ph
O

2k

(79:21 d.r.)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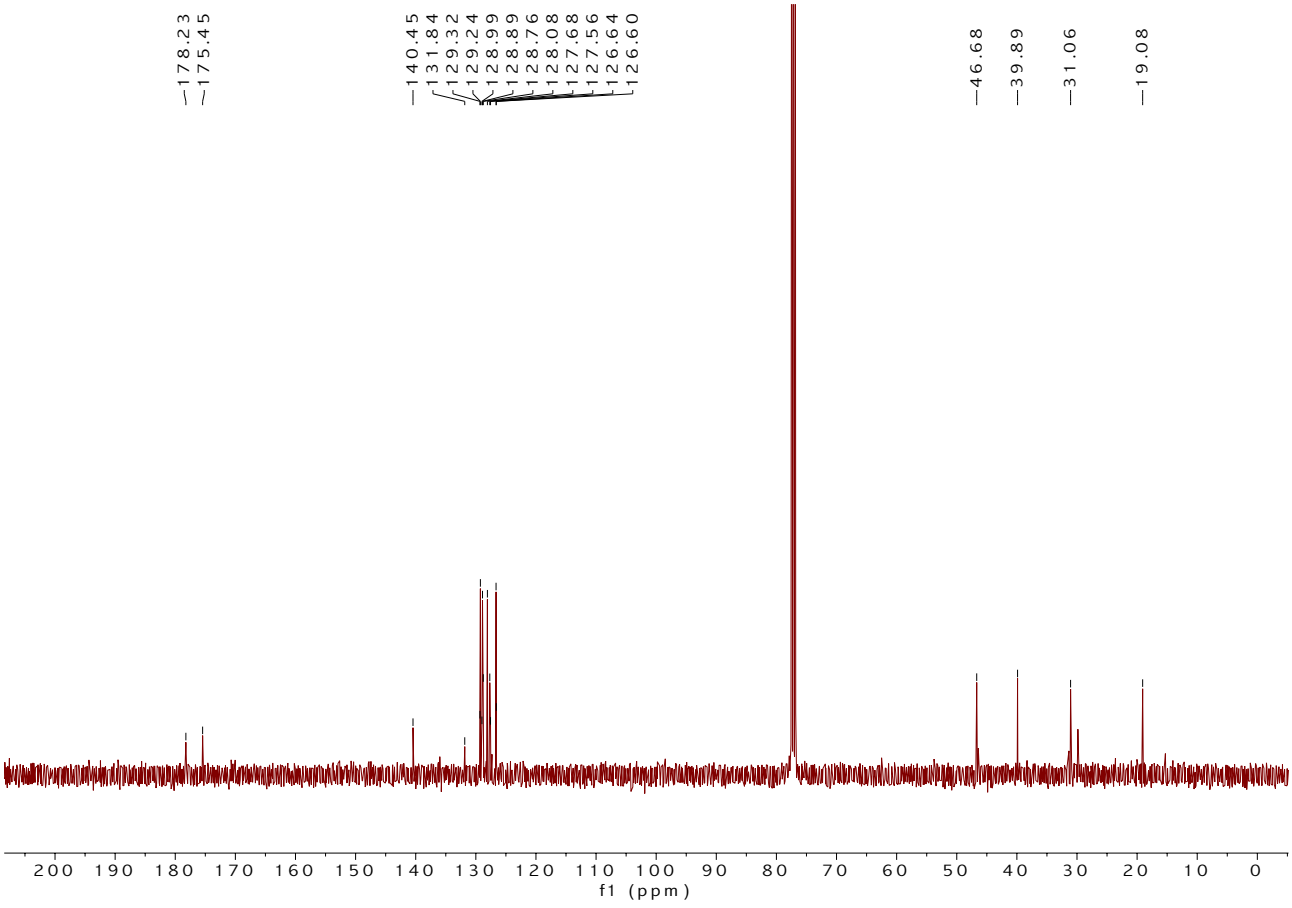
1.96
1.01
0.27
1.00
1.02
1.04
3.11
f1 (ppm)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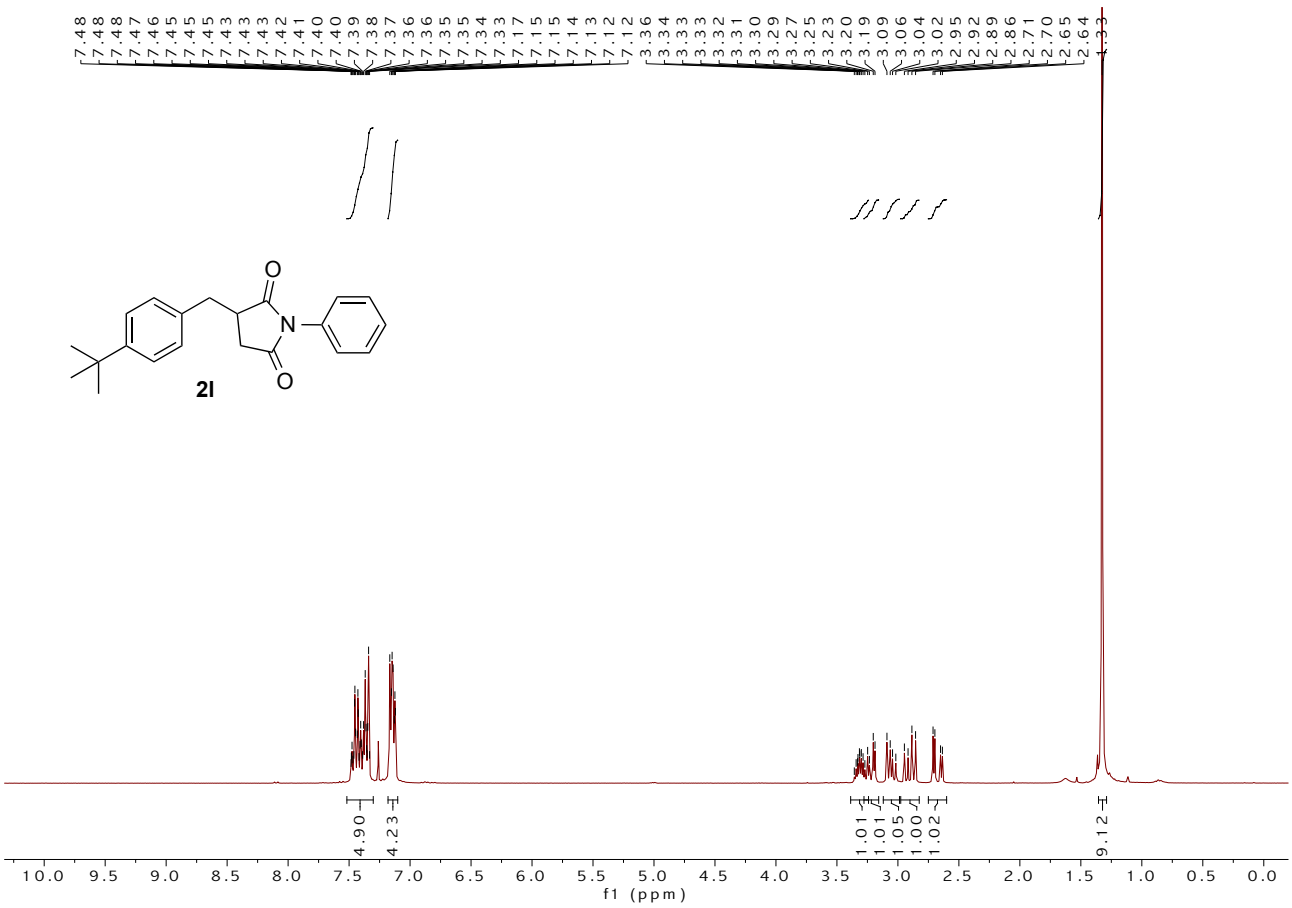
126.60
46.68
39.89
31.06
19.08
f1 (ppm)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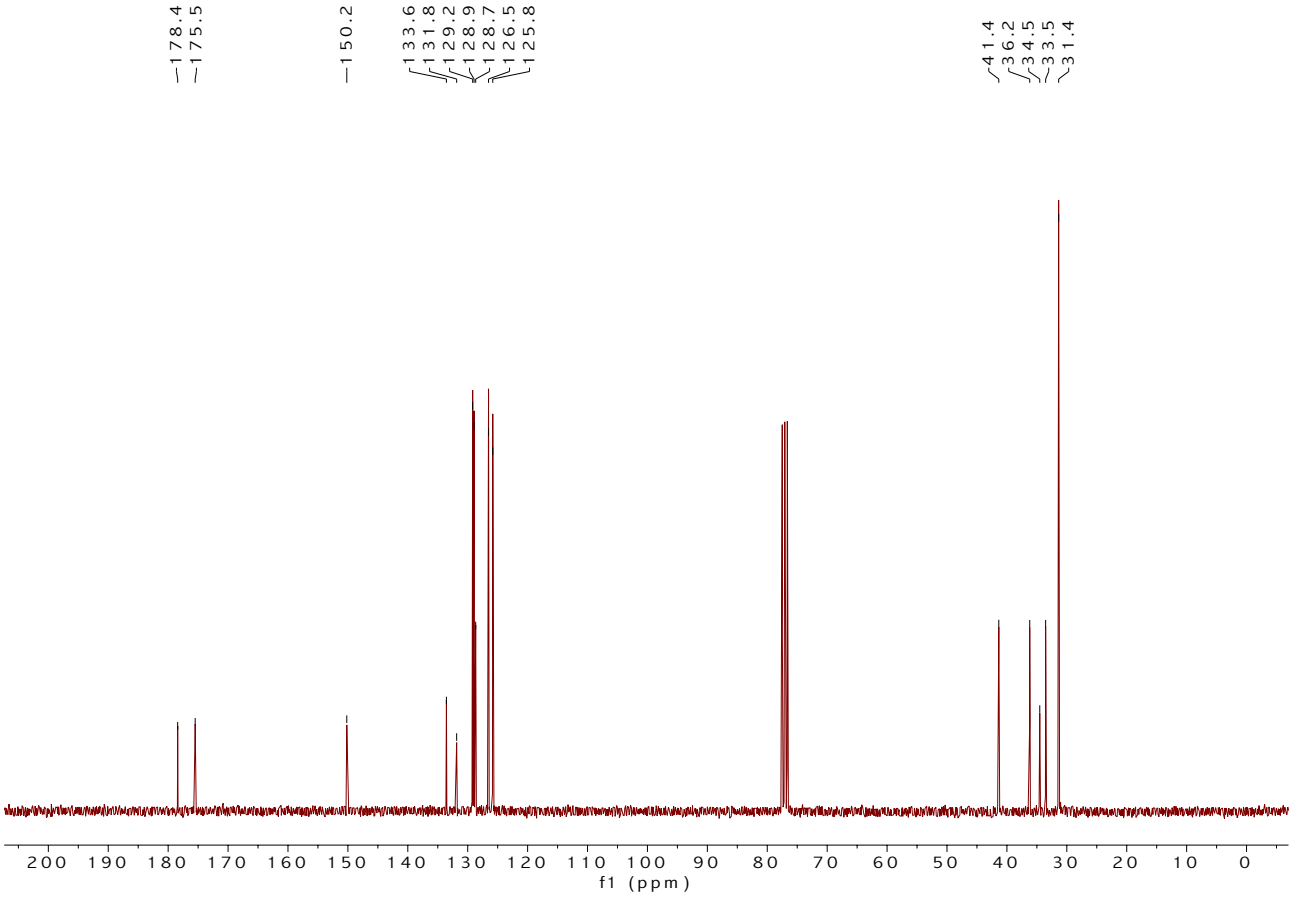
3.36 3.34 3.33 3.33 3.32 3.31 3.30 3.29 3.27 3.25 3.23 3.20 3.19 3.09 3.06 3.04 3.02 2.95 2.92 2.89 2.86 2.71 2.70 2.65 2.64 1.33
O
N
O
2l
4.90
4.23
1.01
1.01
1.05
1.00
1.02
9.12
f1 (ppm)


178.4
175.5
150.2
133.6
131.8
129.2
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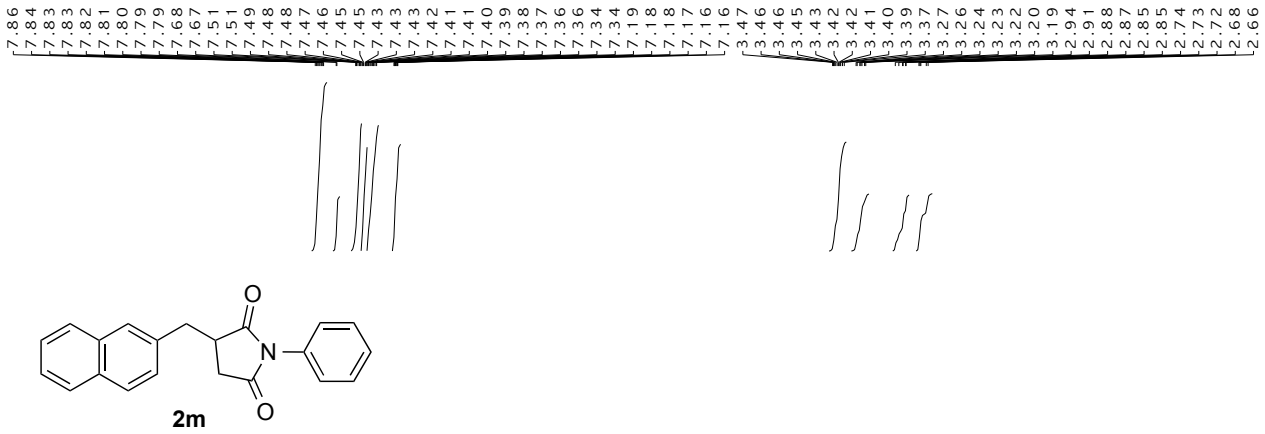
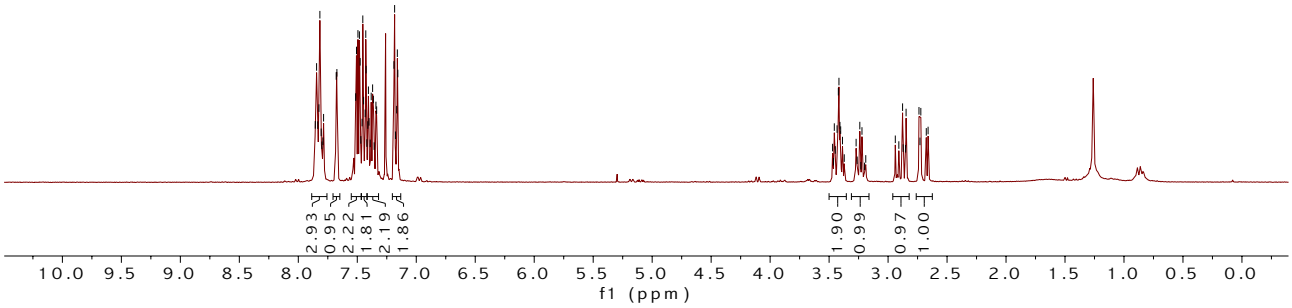
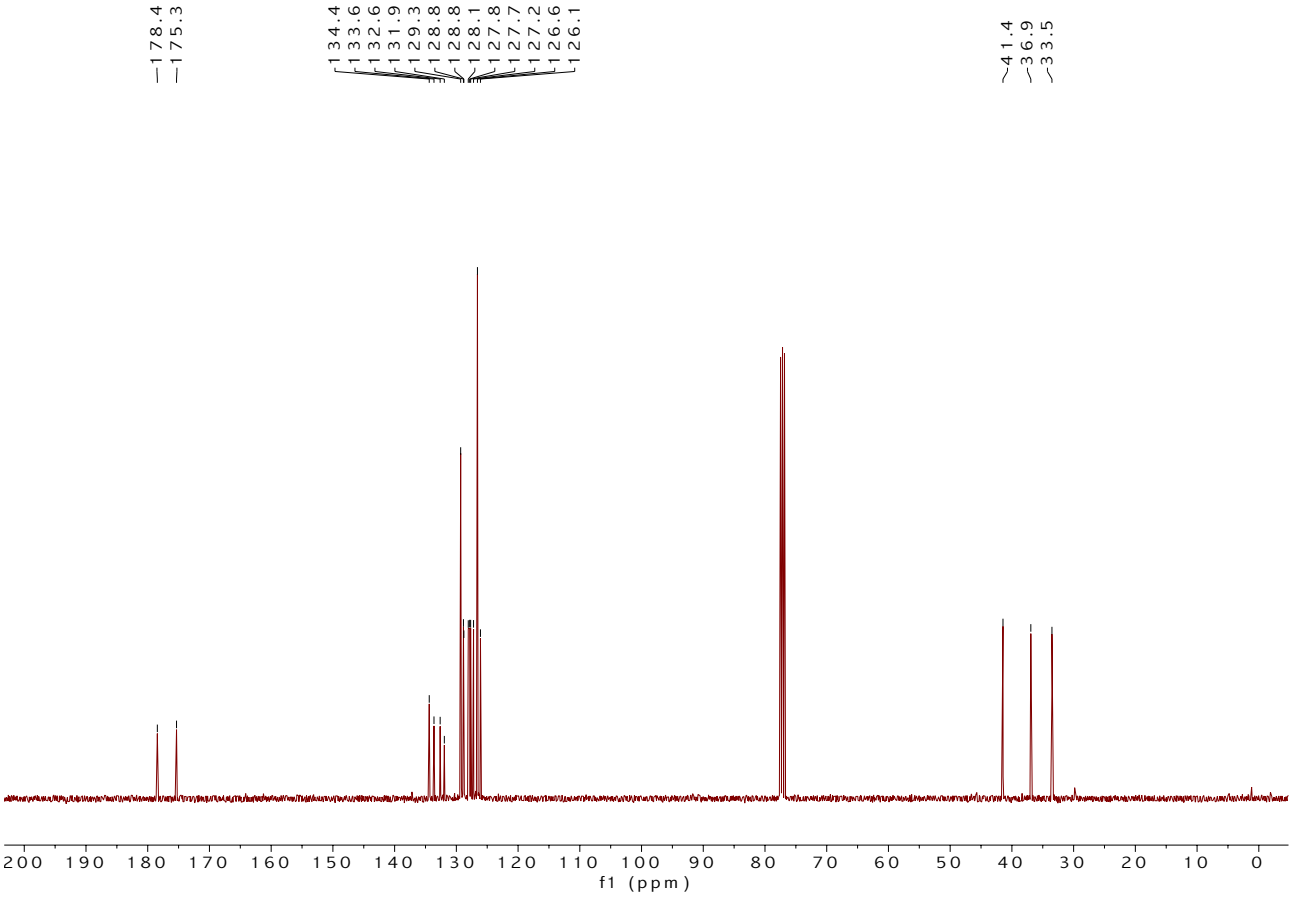
128.7
126.5
125.8
41.4
36.2
34.5
33.5
31.4
f1 (ppm)



O
N
O

2m


2.93
0.95
2.22
1.81
2.19
1.86
1.90
0.99
0.97
1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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175.3
134.4
133.6
132.6
131.9
129.3
128.8
128.8
128.1
127.8
127.7
127.2
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126.1
41.4
36.9
33.5
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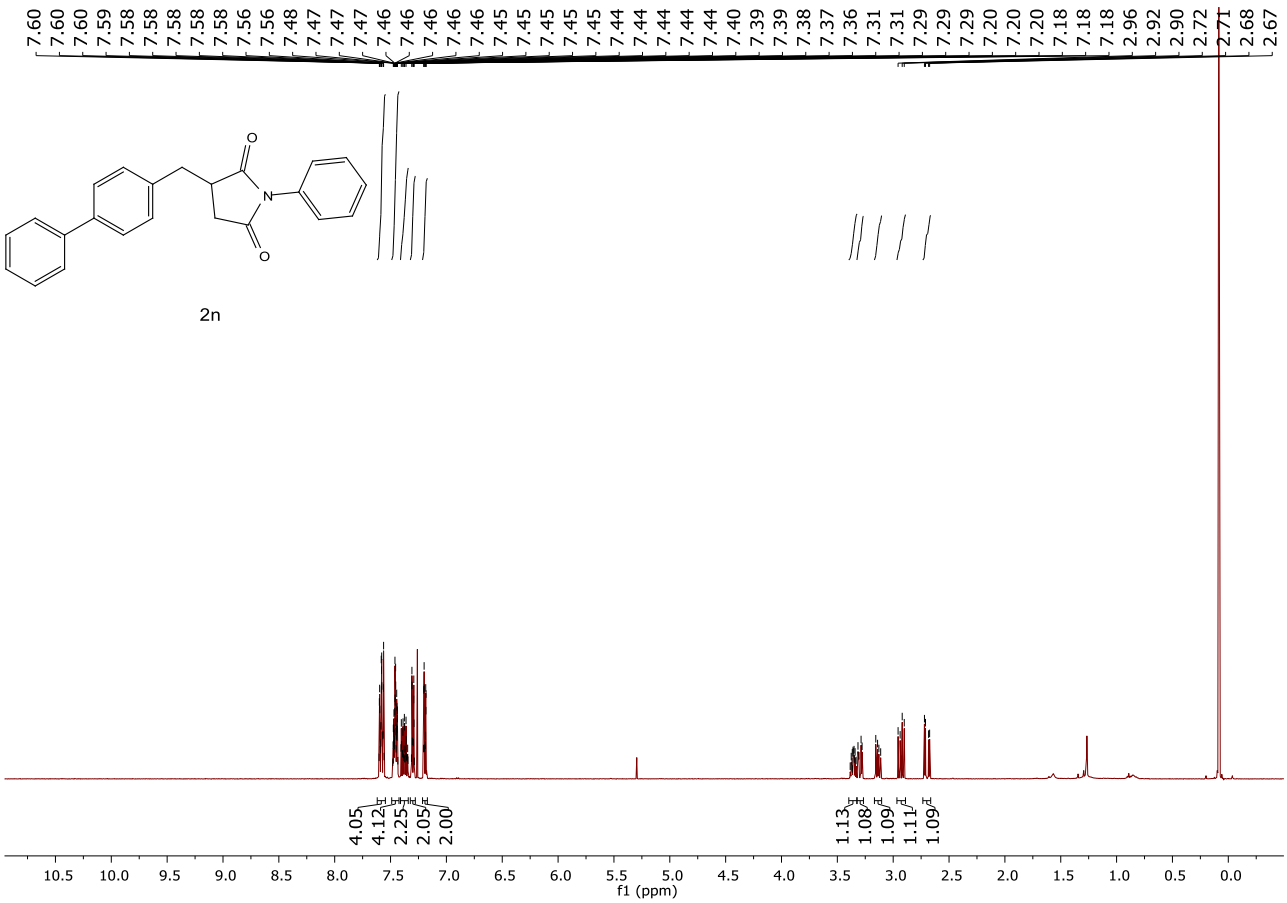
f1 (ppm)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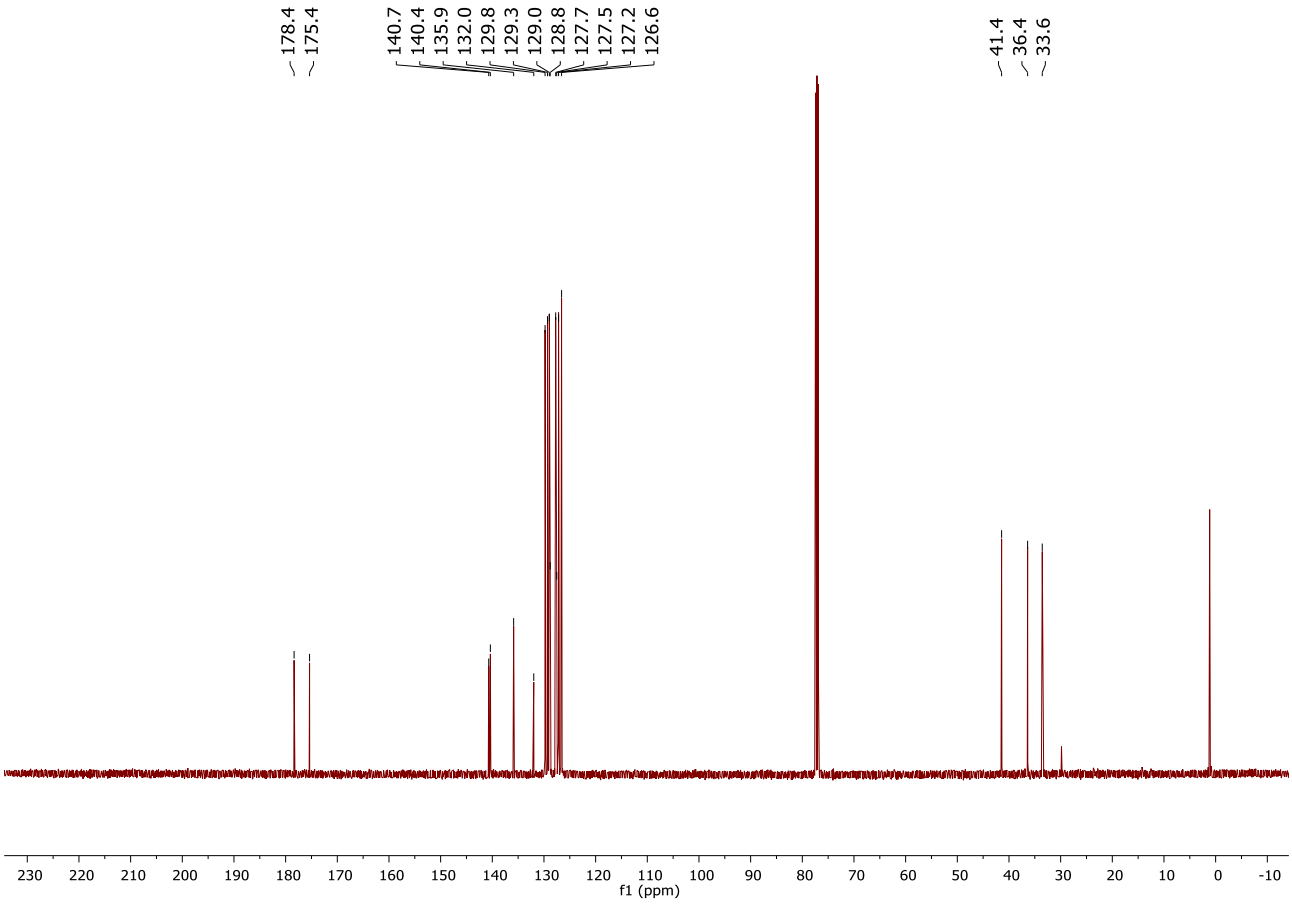
O
N
O
4.05
4.12
2.25
2.05
2.00
1.13
1.08
1.09
1.11
1.09
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

2n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129.8
129.3
129.0
128.8
127.7
127.5
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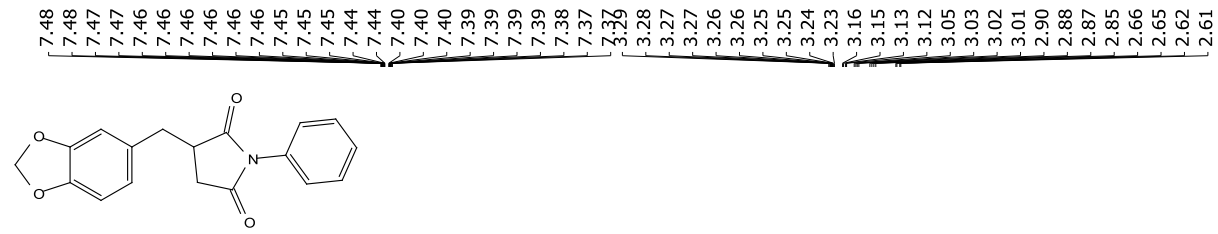
41.4
36.4
33.6
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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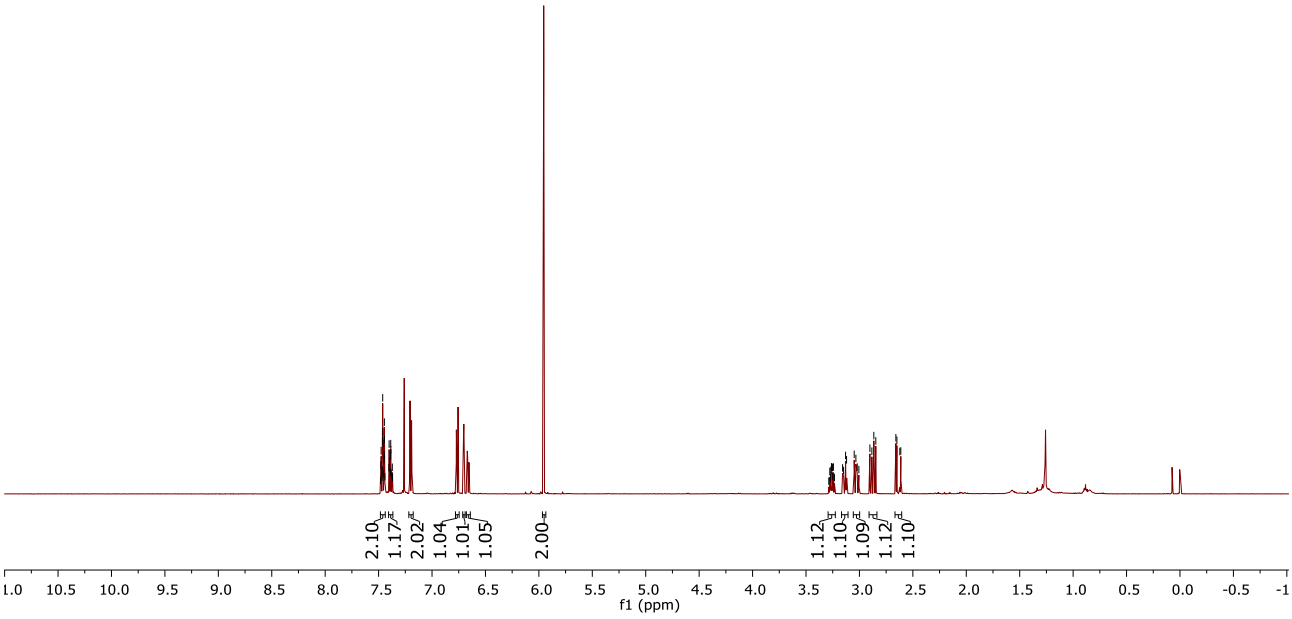
O
N
O

2o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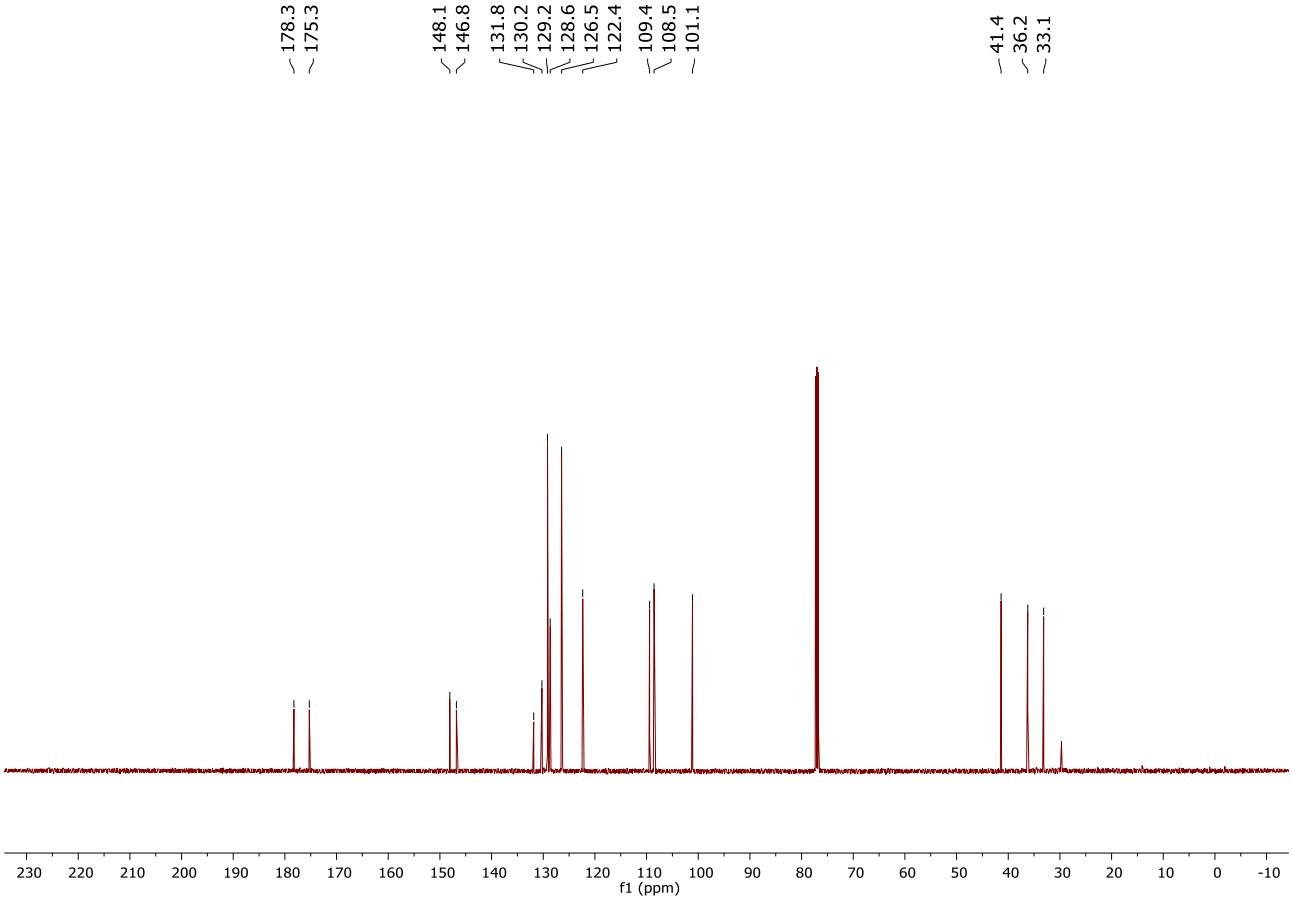
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)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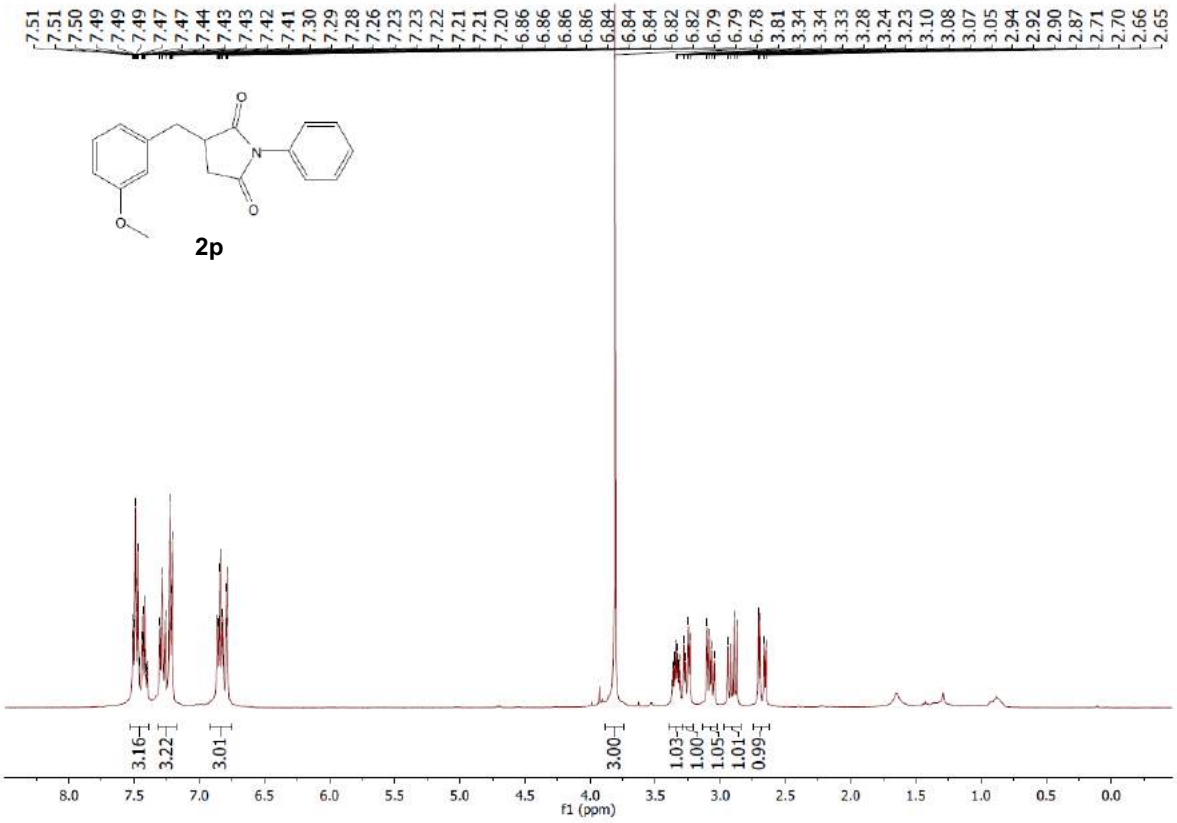
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.20
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6.86
6.86
6.84
6.84
6.84
6.82
6.82
6.79
6.79
6.78
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3.34
3.34
3.33
3.28
3.24
3.23
3.10
3.08
3.07
3.05
2.94
2.92
2.90
2.87
2.71
2.70
2.66
2.65
O
N
O
O
2p
3.16
3.22
3.01
3.00
1.03
1.00
1.05
1.01
0.99
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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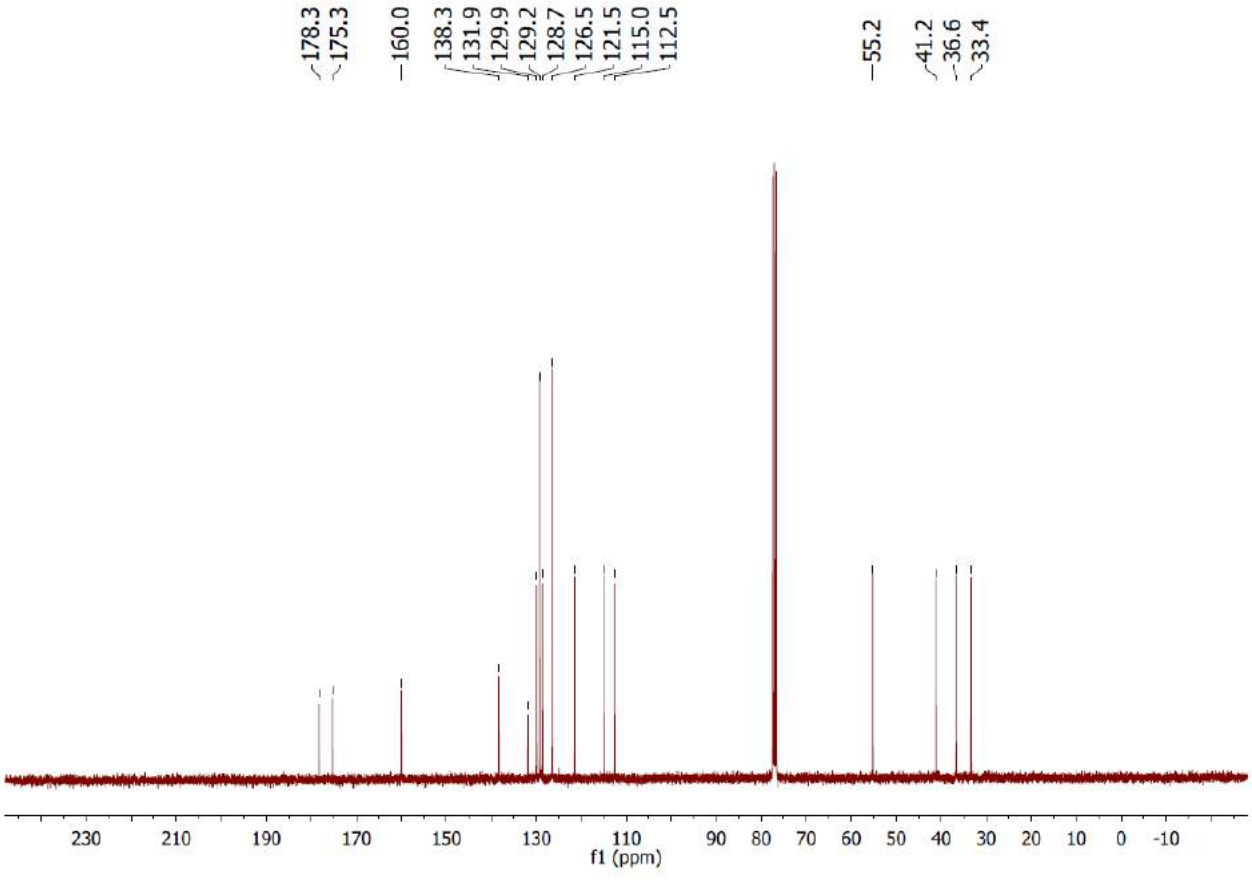
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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129.9
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112.5
55.2
41.2
36.6
33.4
230
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190
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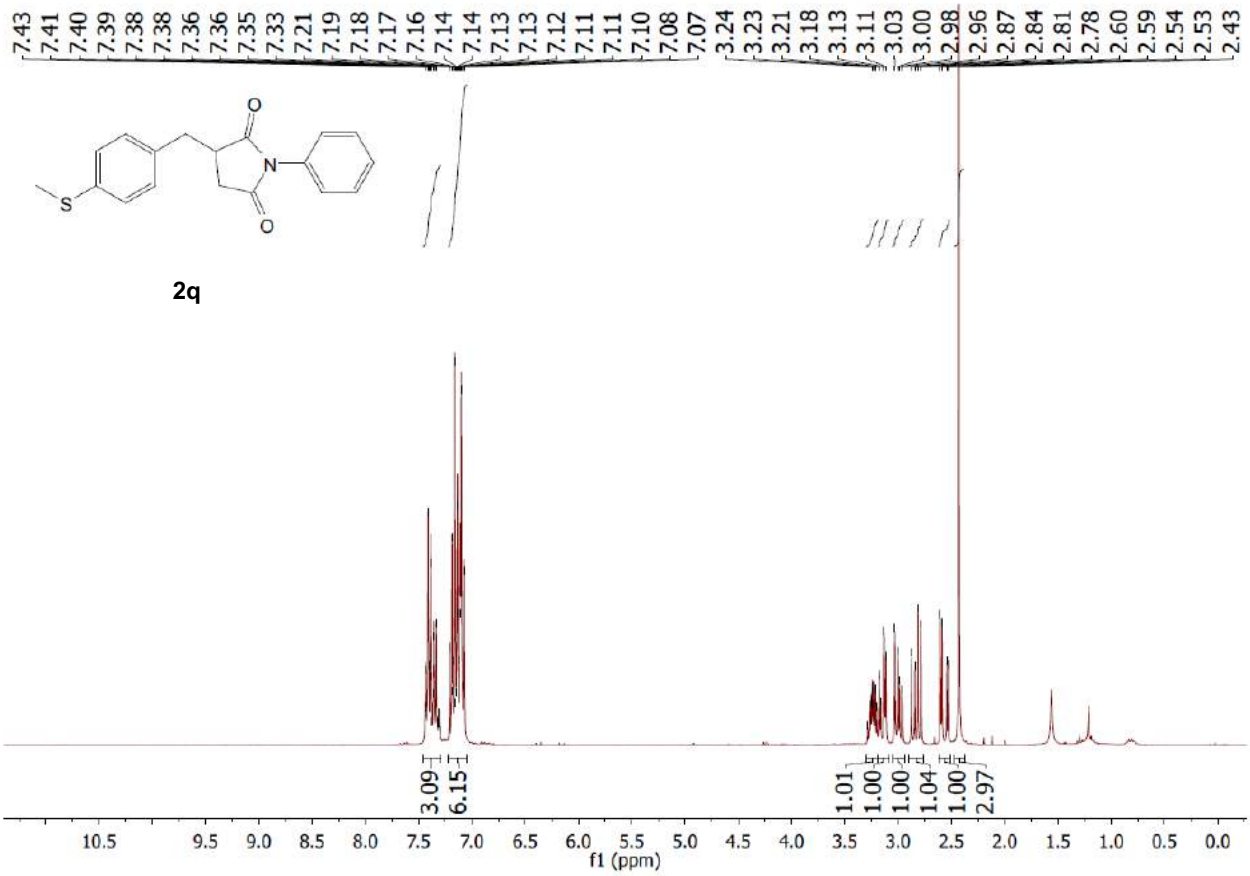
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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7.16
7.14
7.14
7.13
7.13
7.12
7.11
7.11
7.10
7.08
7.07
3.24
3.23
3.21
3.18
3.13
3.11
3.03
3.00
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2.96
2.87
2.84
2.81
2.78
2.60
2.59
2.54
2.53
2.43
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O
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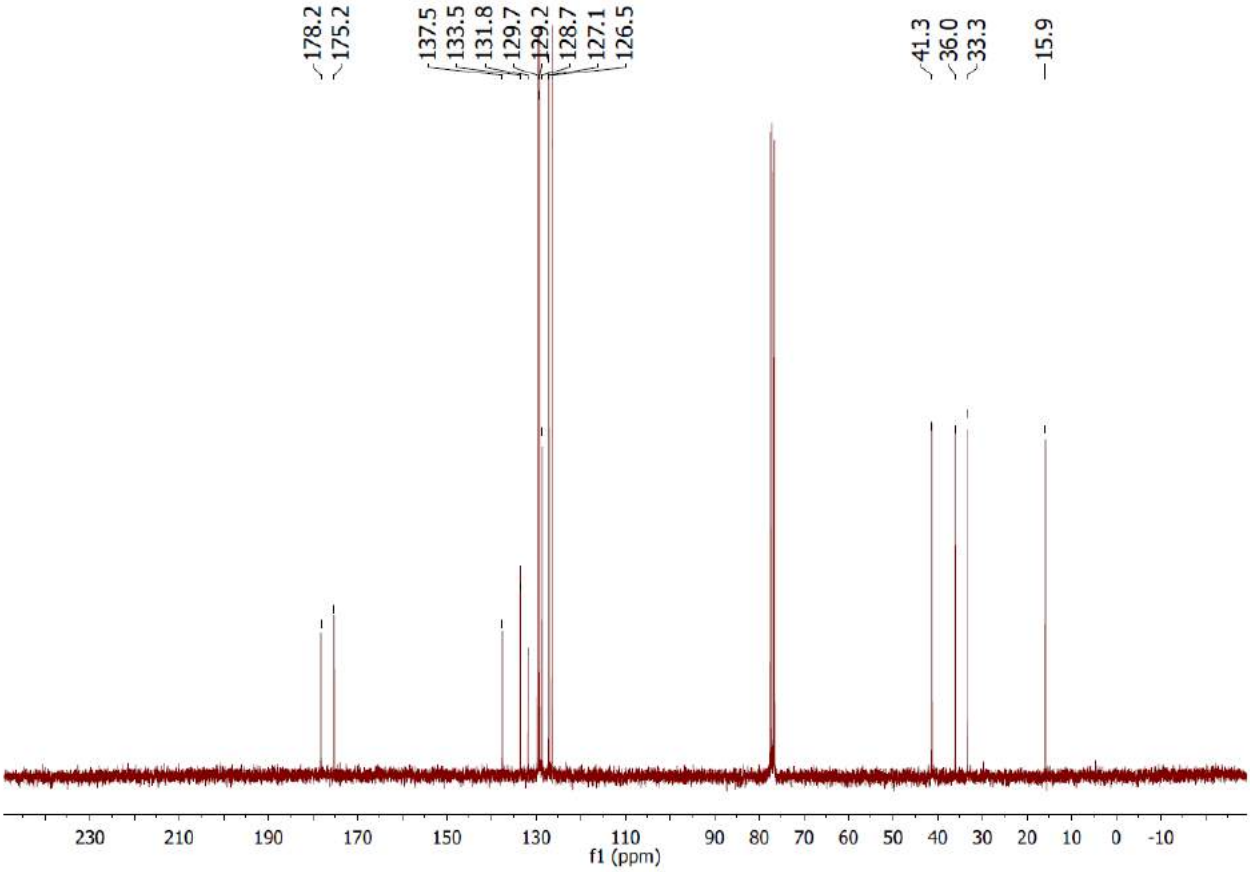
1.00
1.00
1.04
1.00
2.97
f1 (ppm)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129.2
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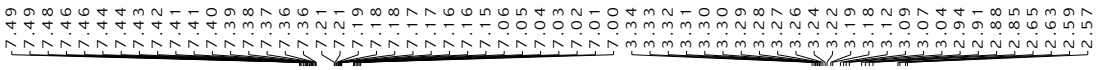
127.1
126.5
41.3
36.0
33.3
15.9
f1 (ppm)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7.18
7.17
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3.19
3.18
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2.85
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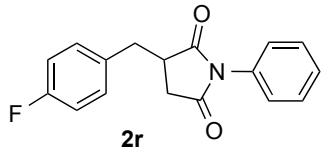
2.57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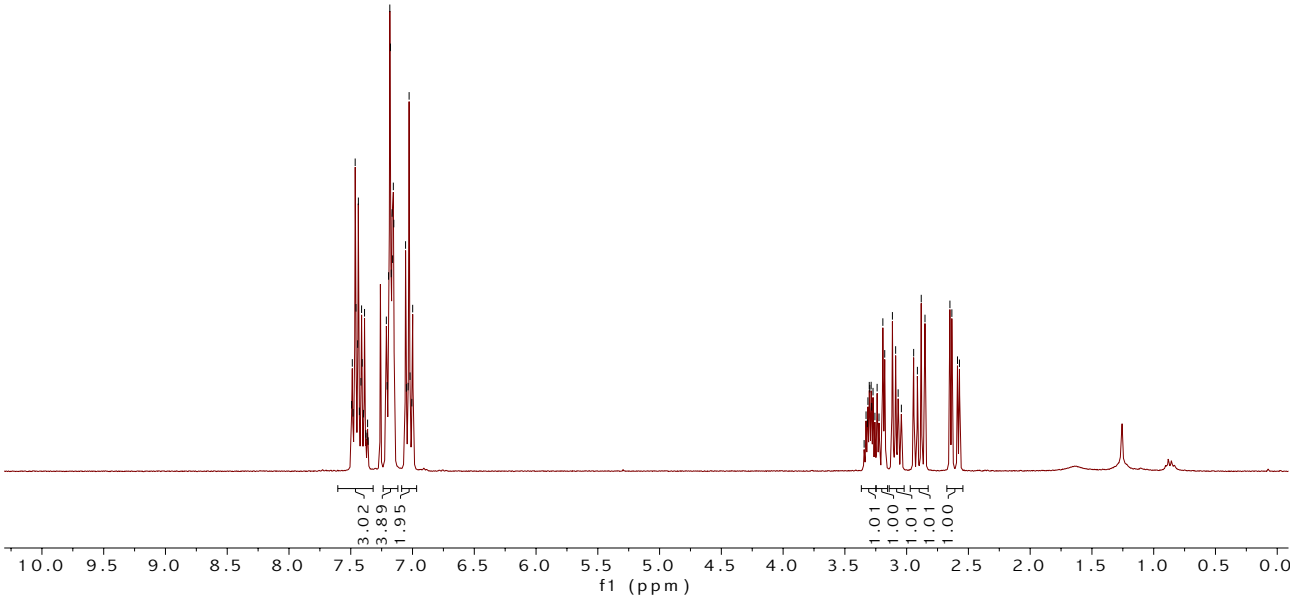
O
F

2r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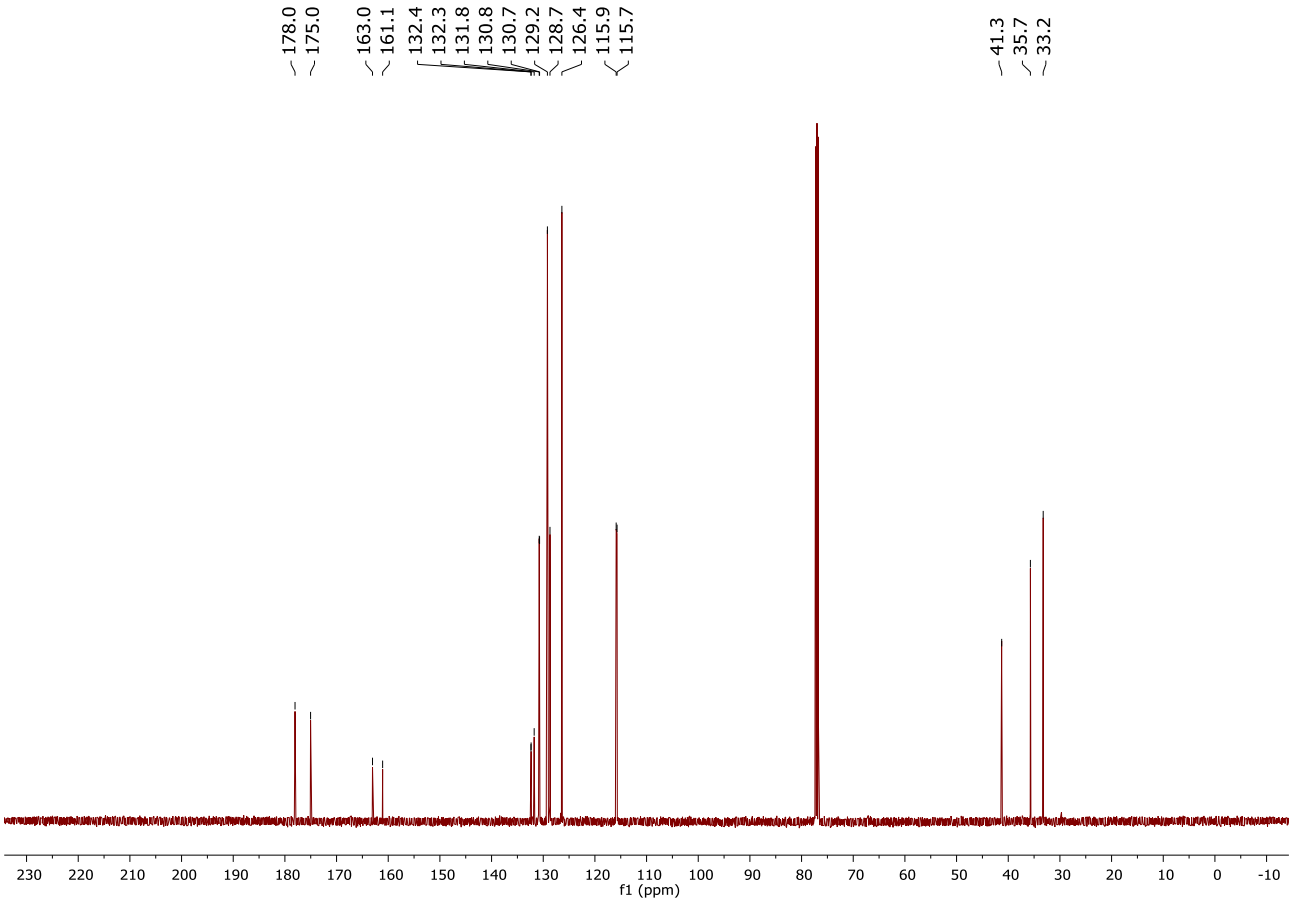
3.89
1.95
1.01
1.00
1.01
1.01
1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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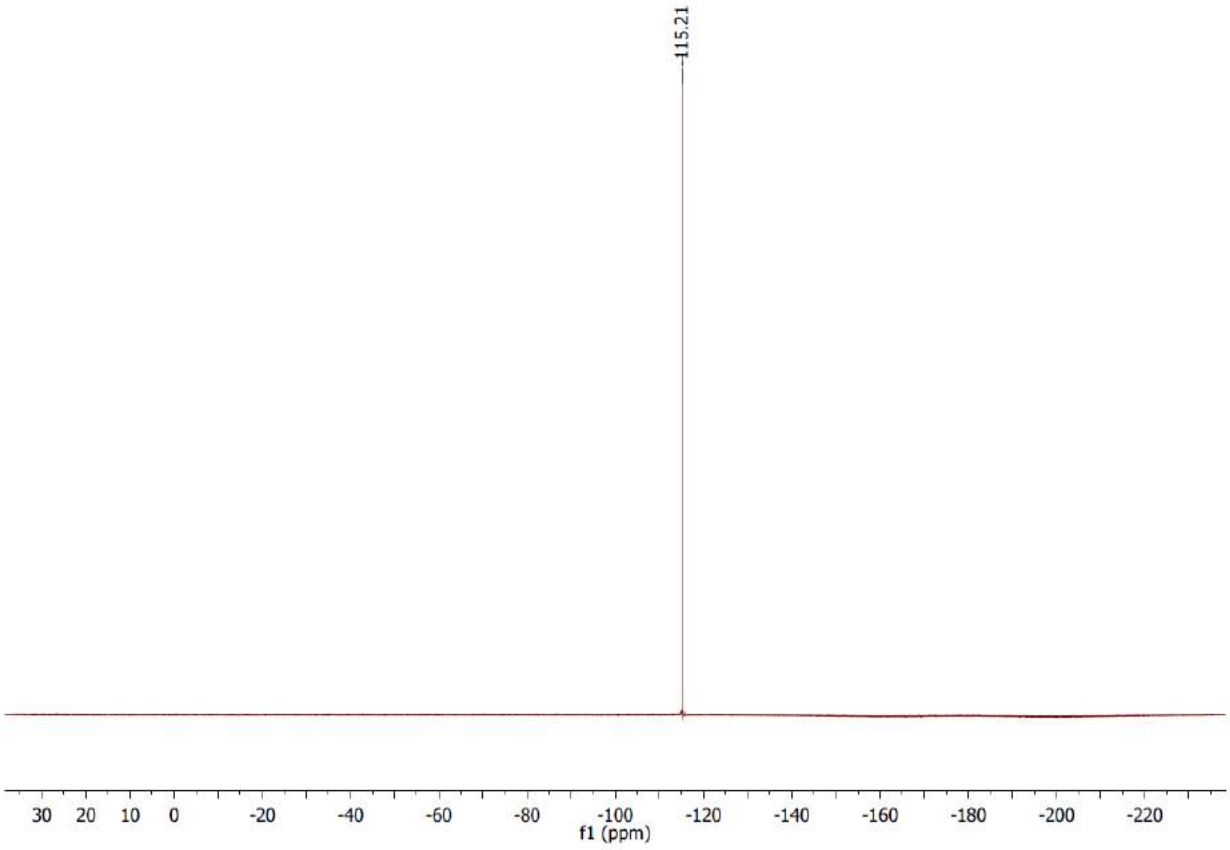
132.3
131.8
130.8
130.7
129.2
128.7
126.4
115.9
115.7
41.3
35.7
33.2
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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10
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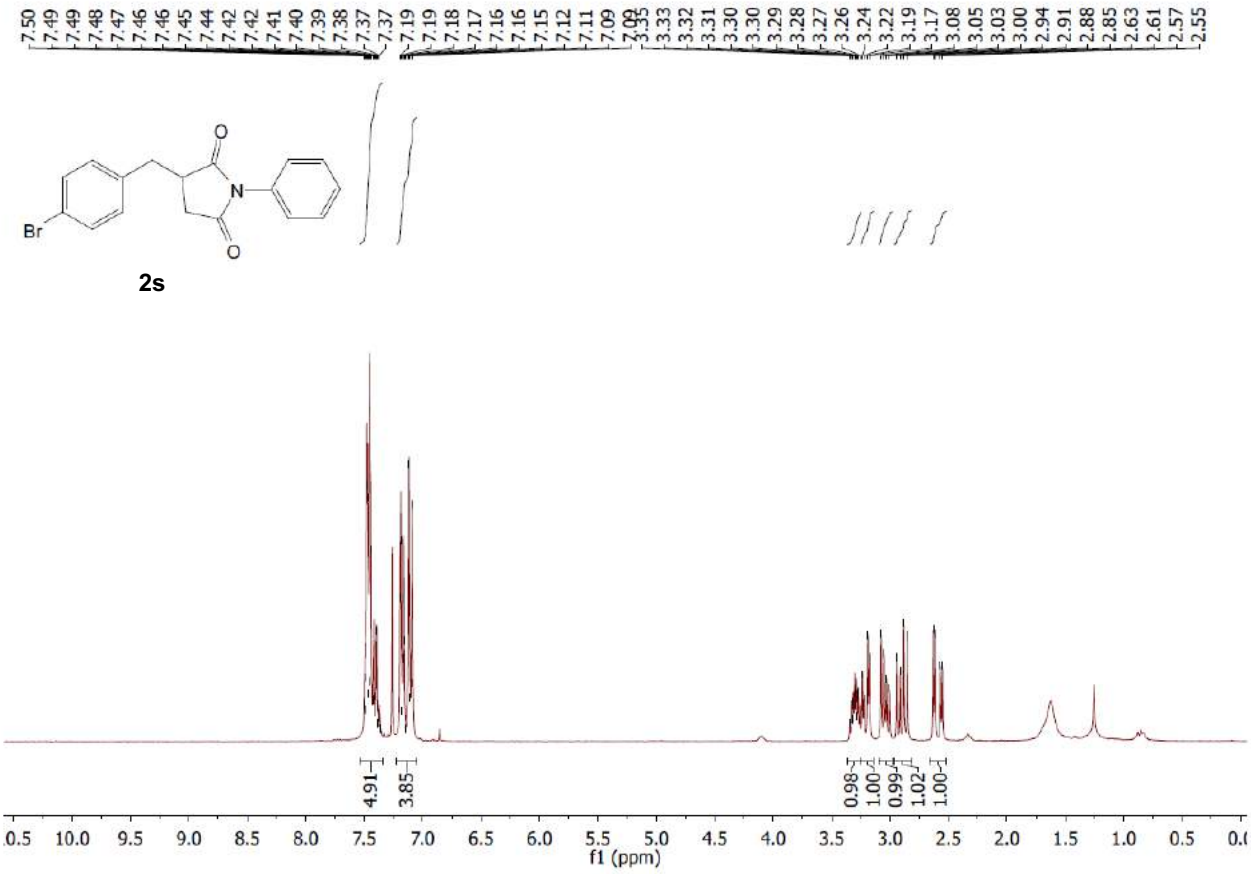
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
f1 (ppm)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Br
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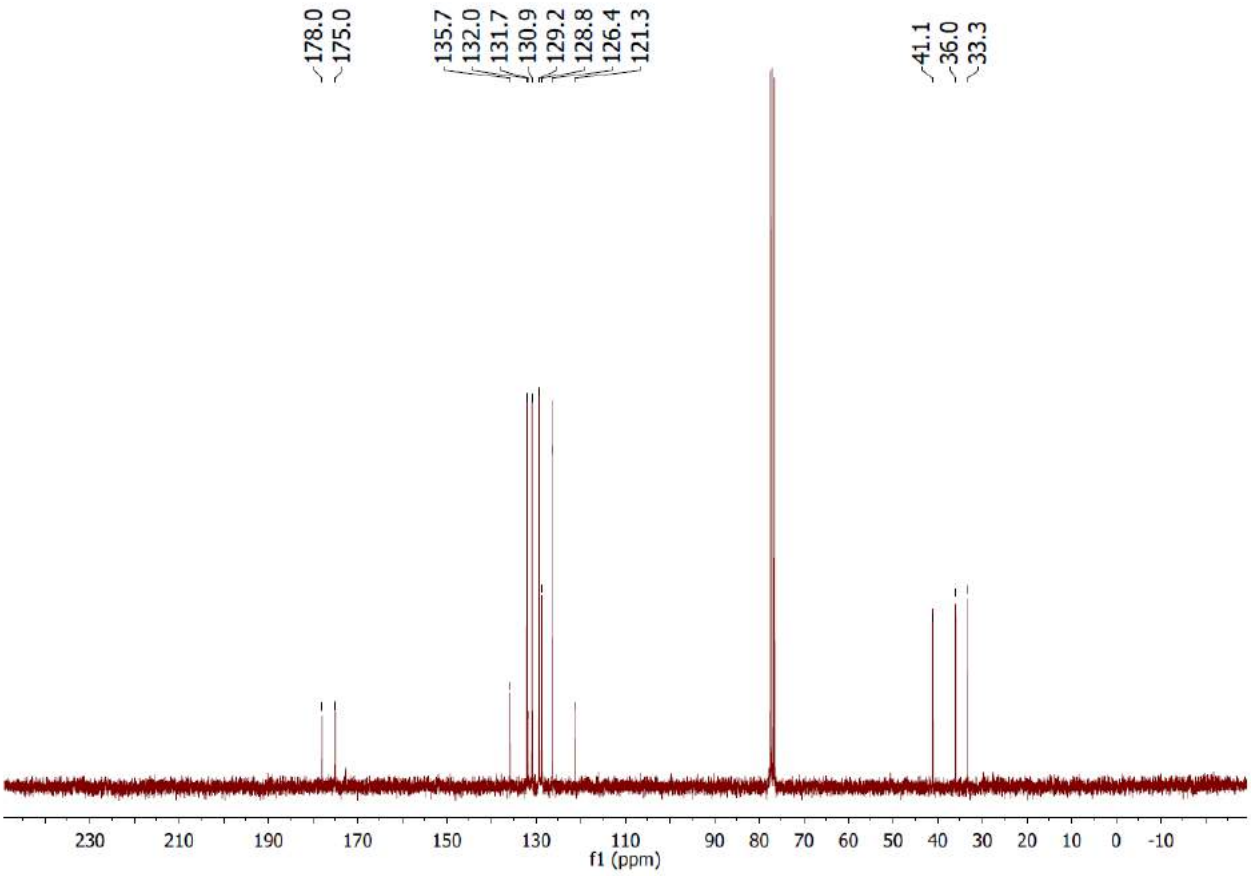
3.85
0.98
1.00
0.99
1.02
1.00
f1 (ppm)

2s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175.0
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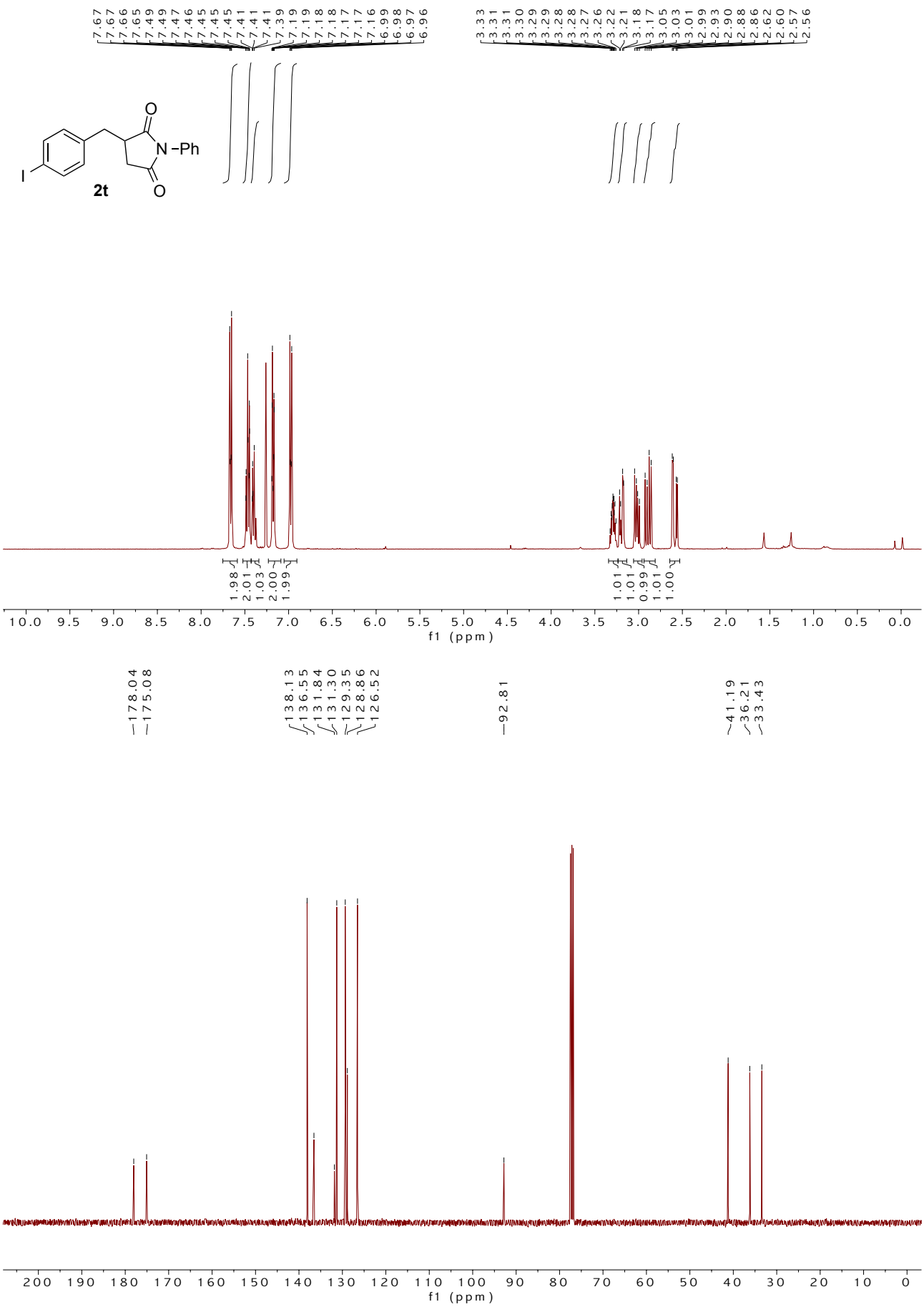
131.7
130.9
129.2
128.8
126.4
121.3
41.1
36.0
33.3
f1 (ppm)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3.05
3.03
3.01
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2.86
2.62
2.60
2.57
2.56
O
N –Ph
O
I
2t
1.98
2.01
1.03
2.00
1.99
1.01
1.01
0.99
1.01
1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
178.04
175.08
138.13
136.55
131.84
131.30
129.35
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92.81
41.19
36.21
33.43
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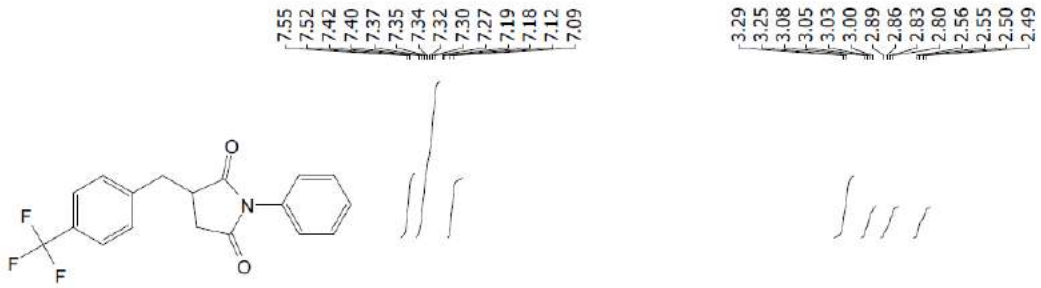
f1 (ppm)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3.03
3.00
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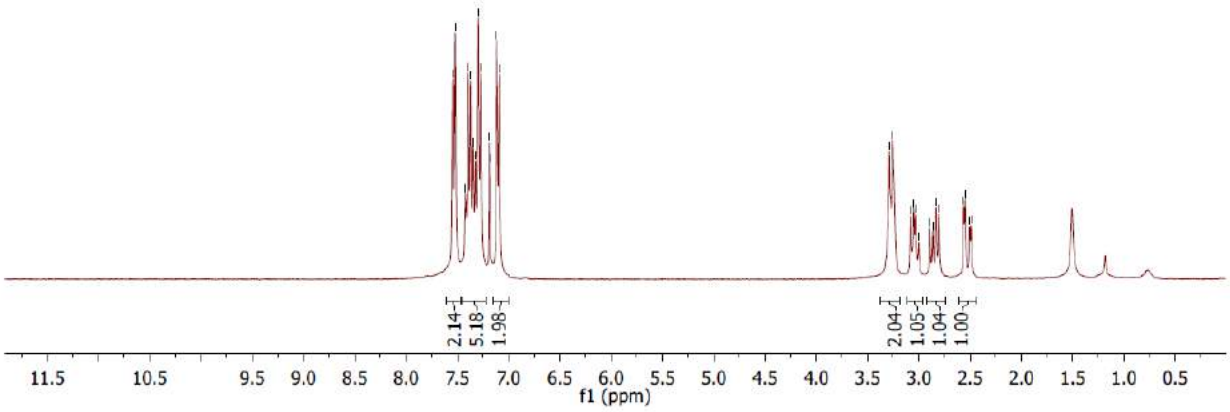
O
F
F
F

2u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8.5
8.0
7.5
7.0
6.5
6.0
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4.0
3.5
3.0
2.5
2.0
1.5
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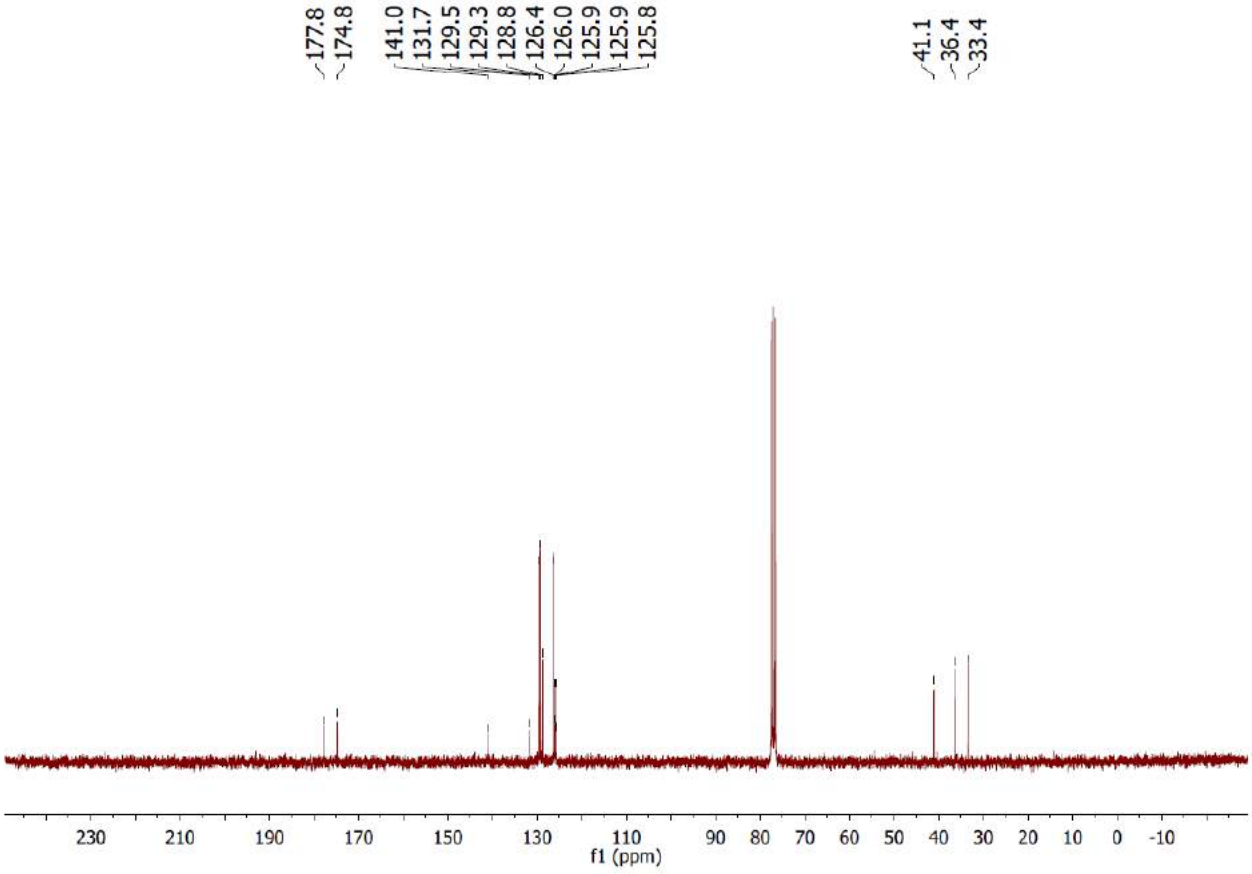
f1 (ppm)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125.9
125.8
41.1
36.4
33.4
230
210
190
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150
130
110
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60
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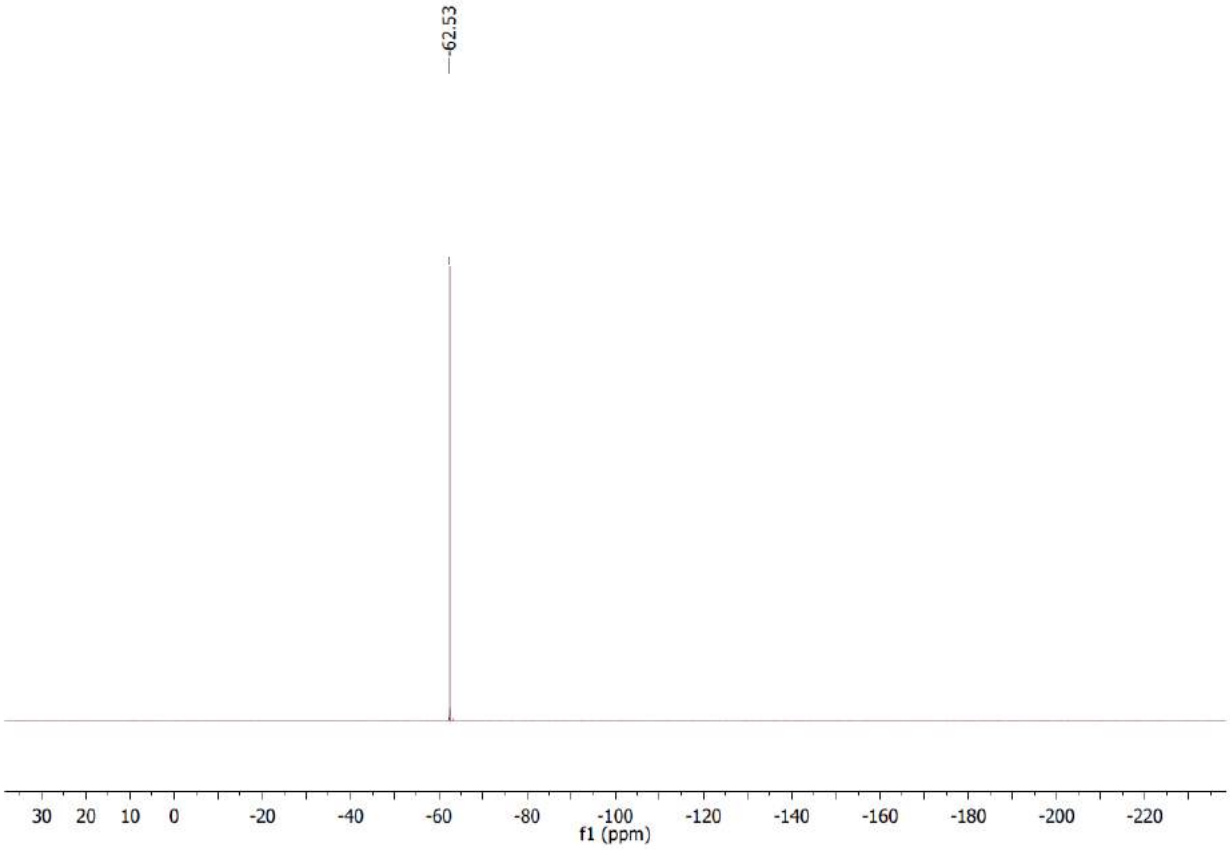
40
30
20
10
0
-10
f1 (ppm)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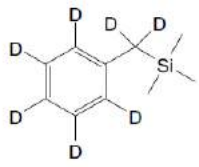
f1 (ppm)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D
D
D
D
D
D
Si

1aD

0.01
9.00
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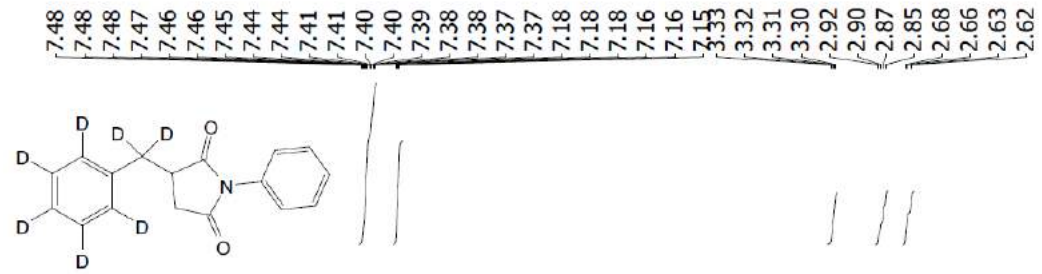
f1 (ppm)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2.90
2.87
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2.63
2.62
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D
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D
D
D
D
D
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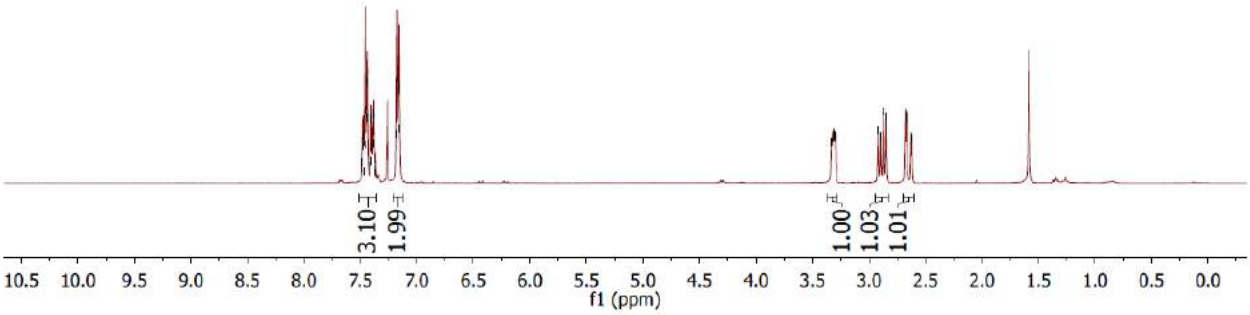
N
O

2aD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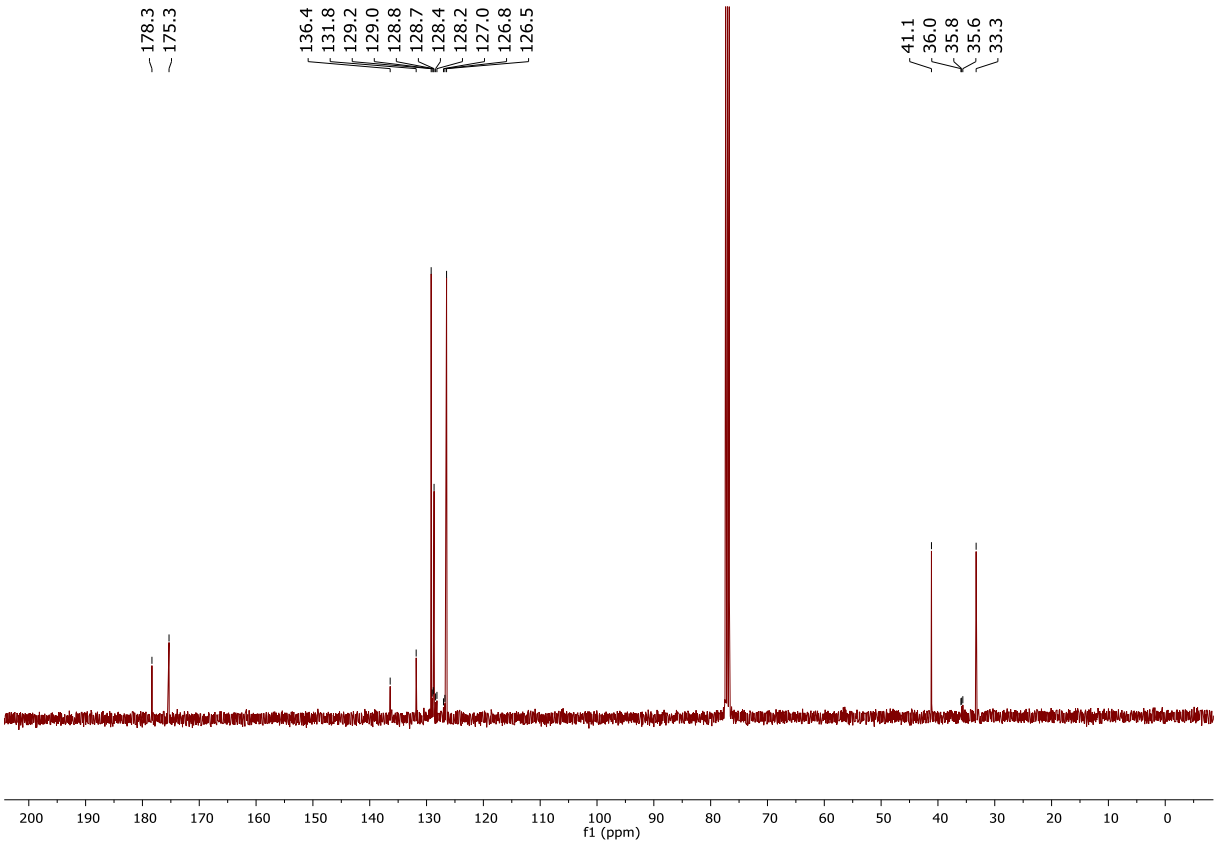
1.00
1.03
1.01
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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131.8
129.2
129.0
128.8
128.7
128.4
128.2
127.0
126.8
126.5
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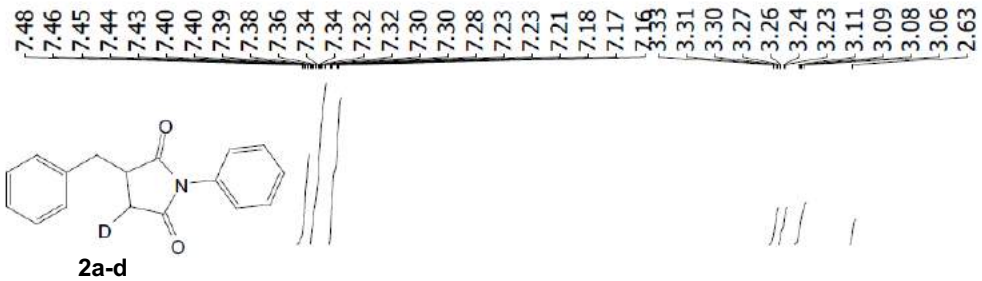
36.0
35.8
35.6
33.3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.23
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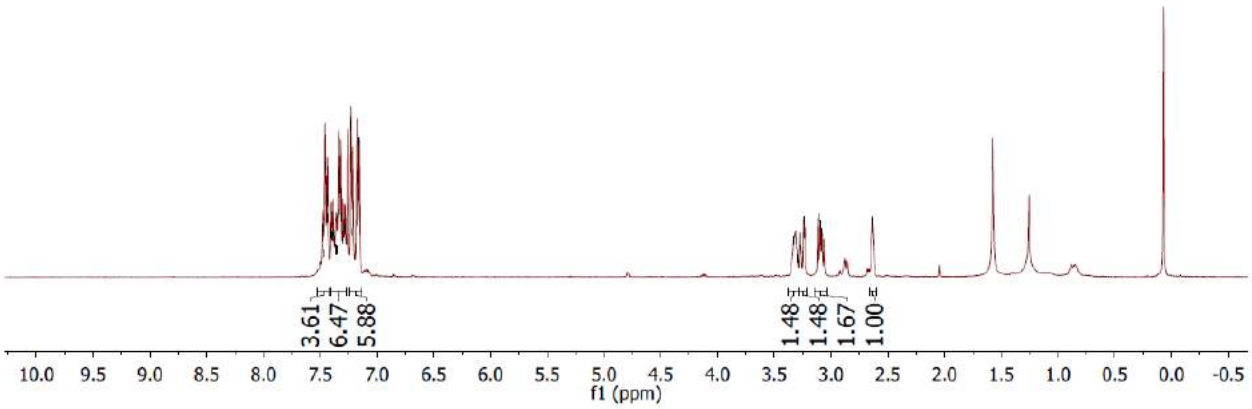
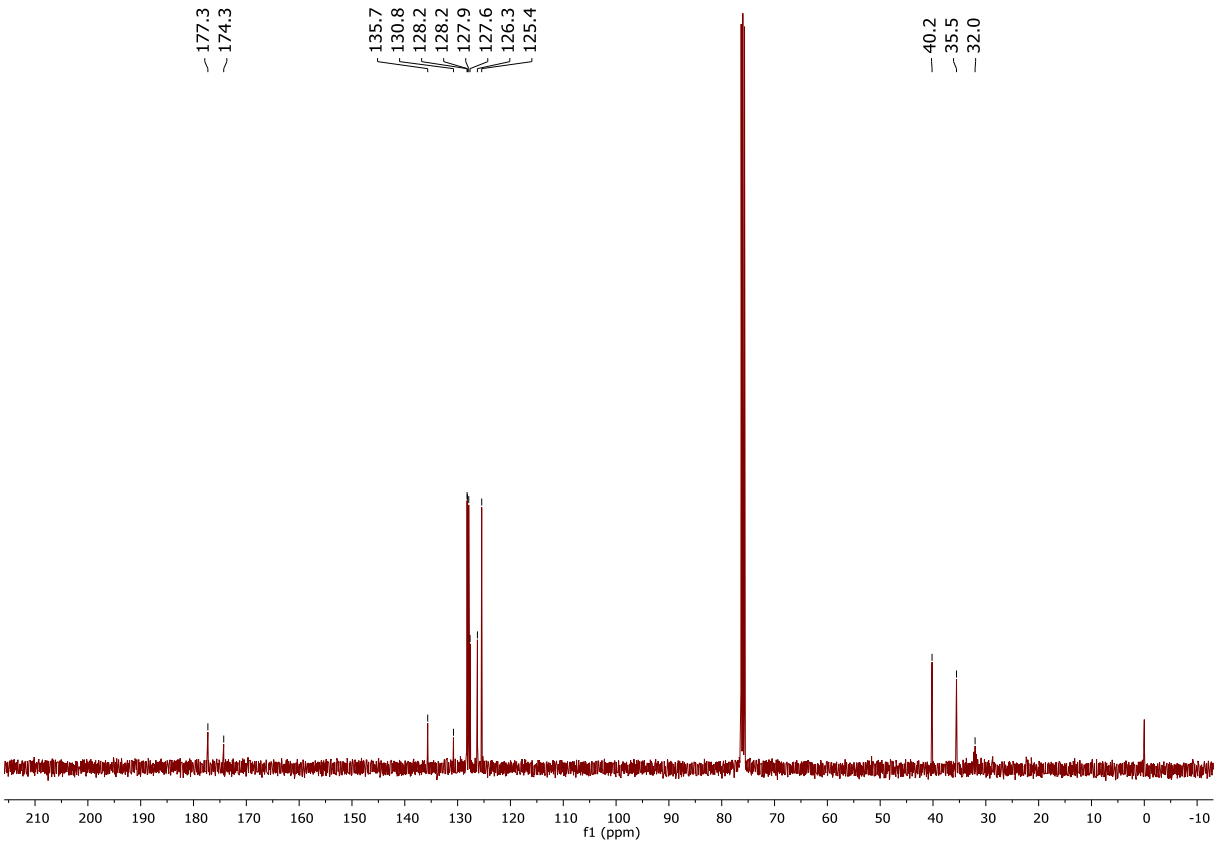
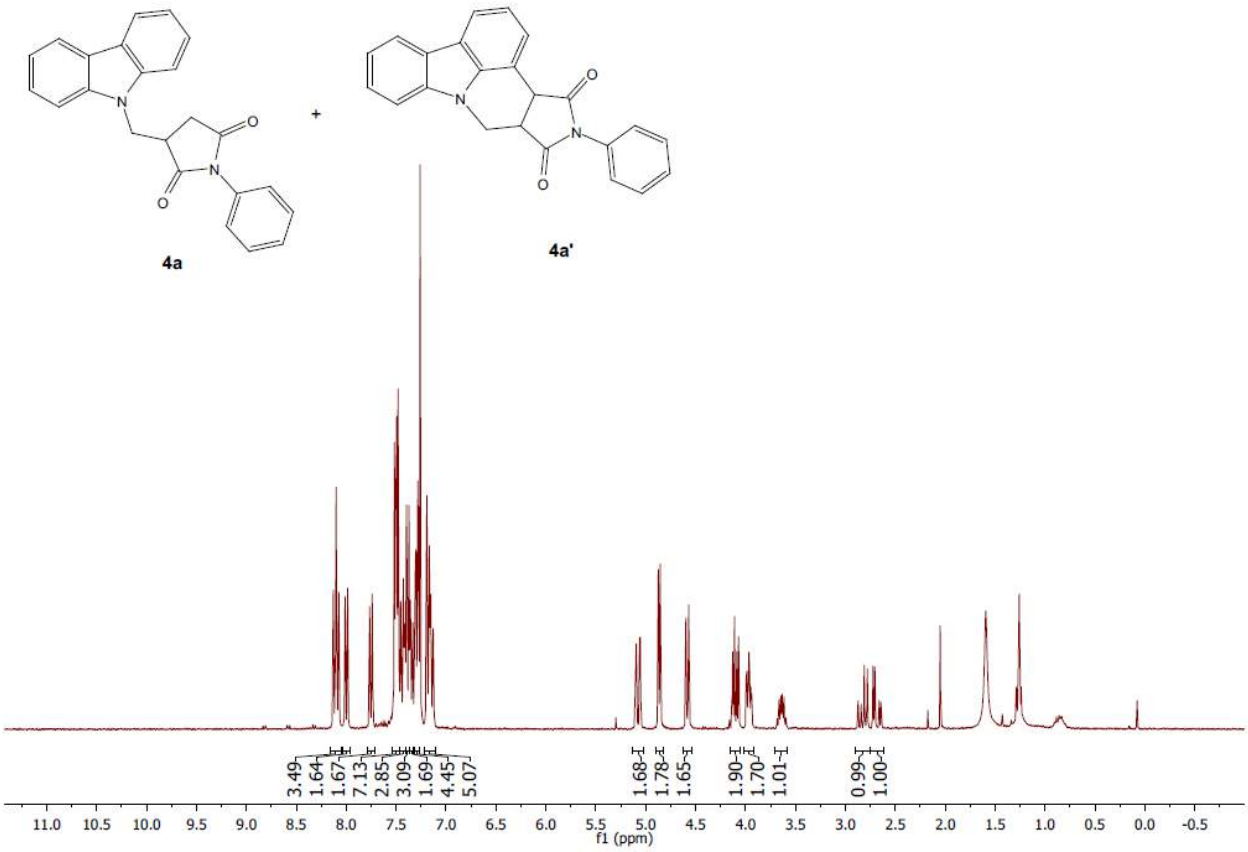
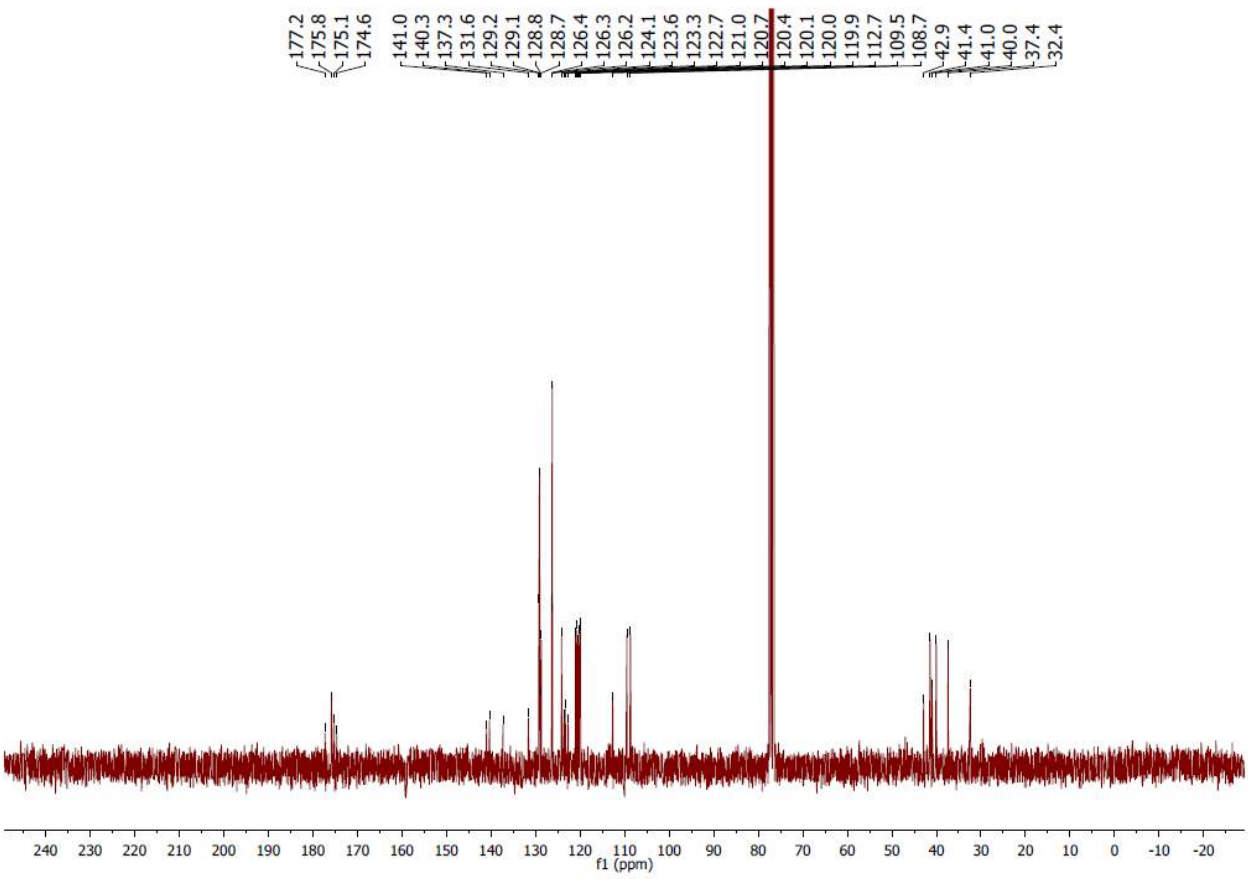
3.33
3.31
3.30
3.27
3.26
3.24
3.23
3.11
3.09
3.08
3.06
2.63
O
N
D
O

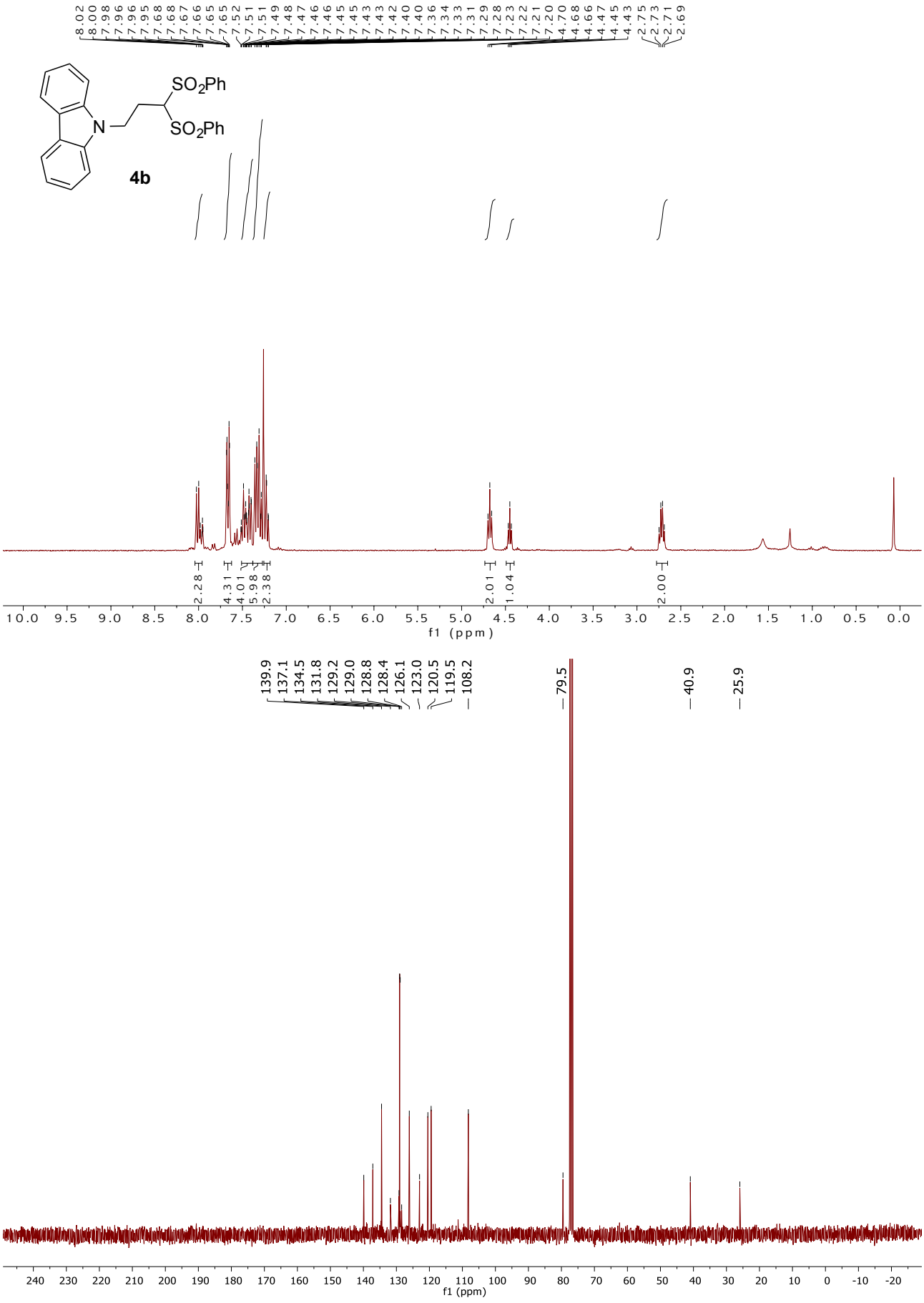
2a-d

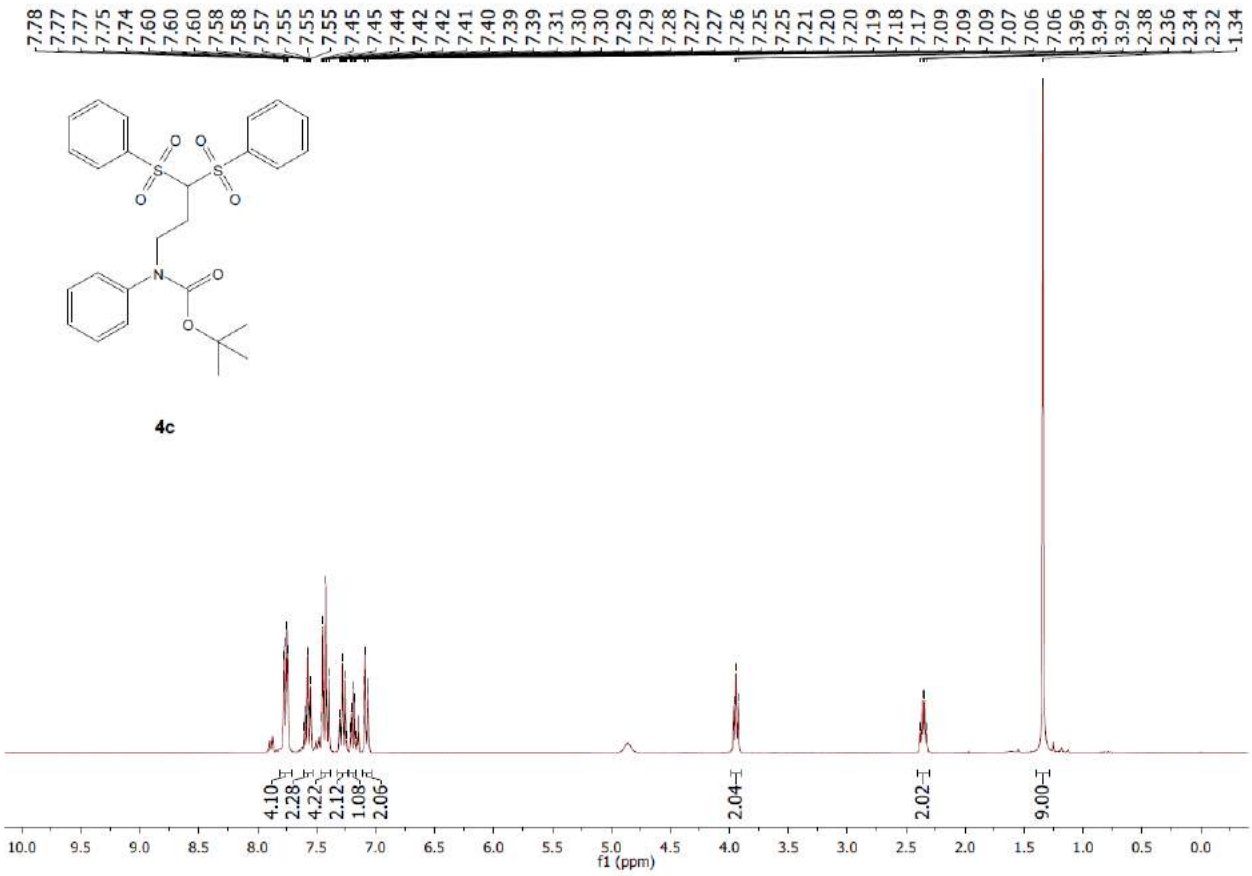

3.61
6.47
5.88
1.48
1.48
1.67
1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


177.3
174.3
135.7
130.8
128.2
128.2
127.9
127.6
126.3
125.4
40.2
35.5
32.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

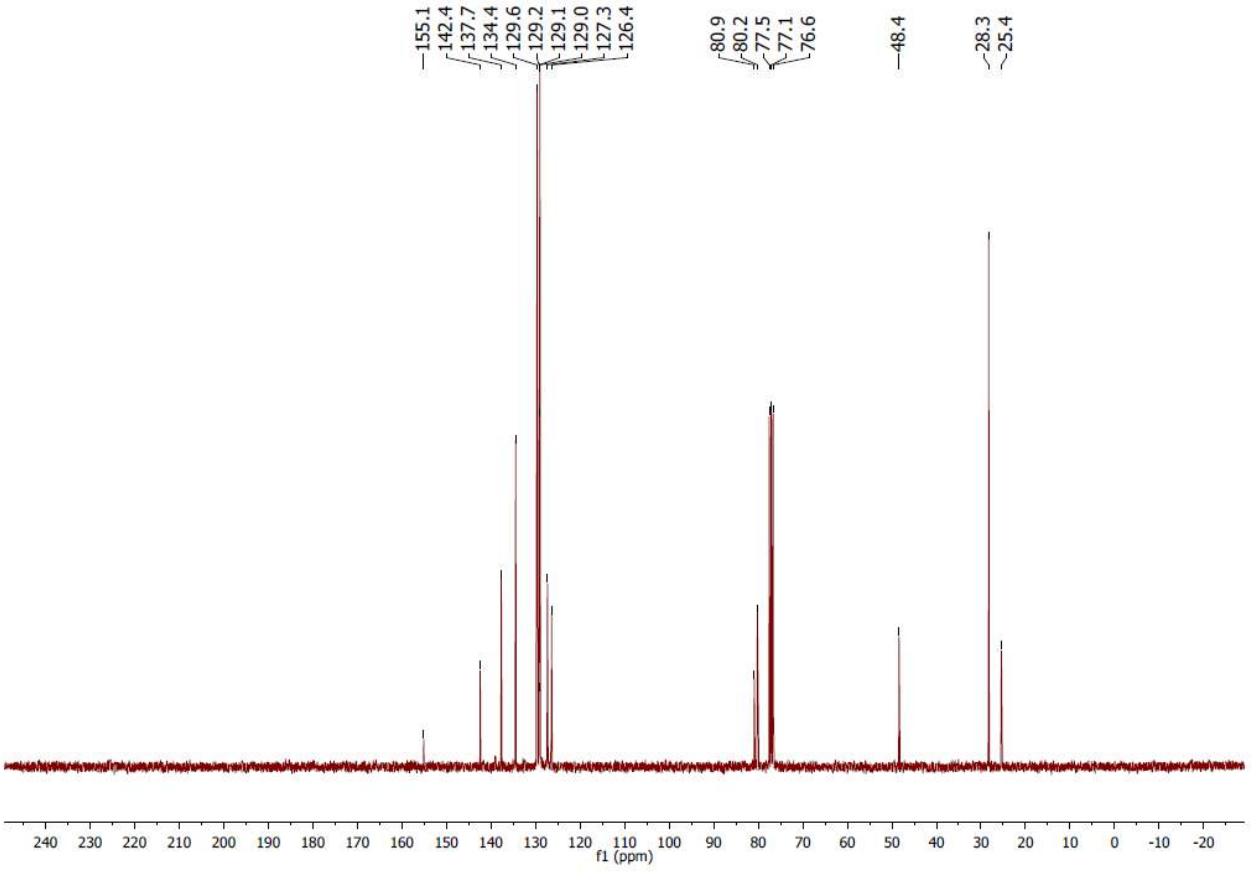

4a
4a'
3.49
1.64
1.67
7.13
2.85
3.09
1.69
4.45
5.07
1.68
1.78
1.65
1.90
1.70
1.01
0.99
1.00
f1 (ppm)

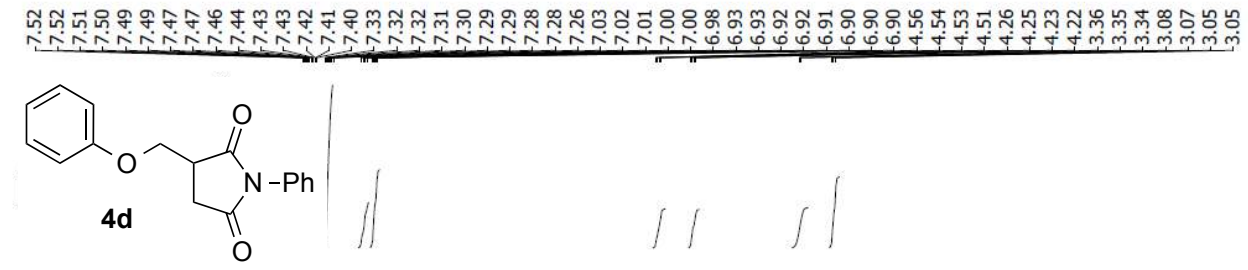

177.2
175.8
175.1
174.6
141.0
140.3
137.3
131.6
129.2
129.1
128.8
128.7
126.4
126.3
126.2
124.1
123.6
123.3
122.7
121.0
120.7
120.4
120.1
120.0
119.9
112.7
109.5
108.7
42.9
41.4
41.0
40.0
37.4
32.4
f1 (ppm)


8.02
8.00
7.98
7.96
7.96
7.95
7.68
7.68
7.67
7.66
7.65
7.65
7.52
7.51
7.51
7.49
7.48
7.47
7.46
7.46
7.45
7.45
7.43
7.43
7.42
7.40
7.40
7.36
7.34
7.33
7.31
7.29
7.28
7.23
7.22
7.21
7.20
4.70
4.68
4.66
4.47
4.45
4.43
2.75
2.73
2.71
2.69
SO2Ph
SO2Ph
N
4b
2.28
4.31
4.01
5.98
2.38
2.01
1.04
2.00
f1 (ppm)
139.9
137.1
134.5
131.8
129.2
129.0
128.8
128.4
126.1
123.0
120.5
119.5
108.2
79.5
40.9
25.9
f1 (ppm)


7.78 7.77 7.77 7.75 7.74 7.60 7.60 7.58 7.58 7.57 7.55 7.55 7.45 7.45 7.44 7.42 7.42 7.41 7.40 7.39 7.39 7.31 7.30 7.30 7.29 7.29 7.28 7.27 7.27 7.26 7.25 7.25 7.21 7.20 7.20 7.19 7.18 7.17 7.09 7.09 7.07 7.06 7.06 3.96 3.94 3.92 2.38 2.36 2.34 2.32 1.34
4.10 2.28 4.22 2.12 1.08 2.06 2.04 2.02 9.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

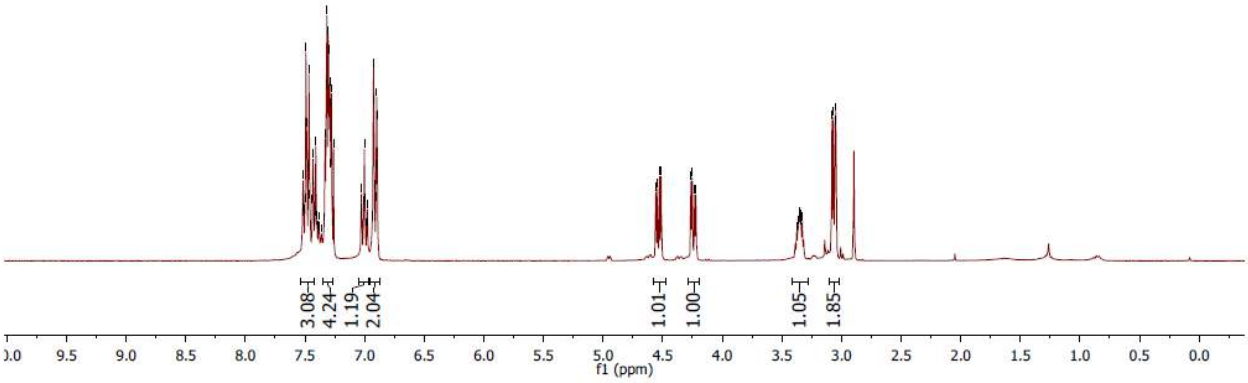
4c

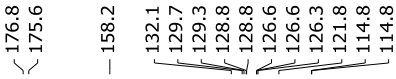

155.1 142.4 137.7 134.4 129.6 129.2 129.1 129.0 127.3 126.4 80.9 80.2 77.5 77.1 76.6 48.4 28.3 25.4
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

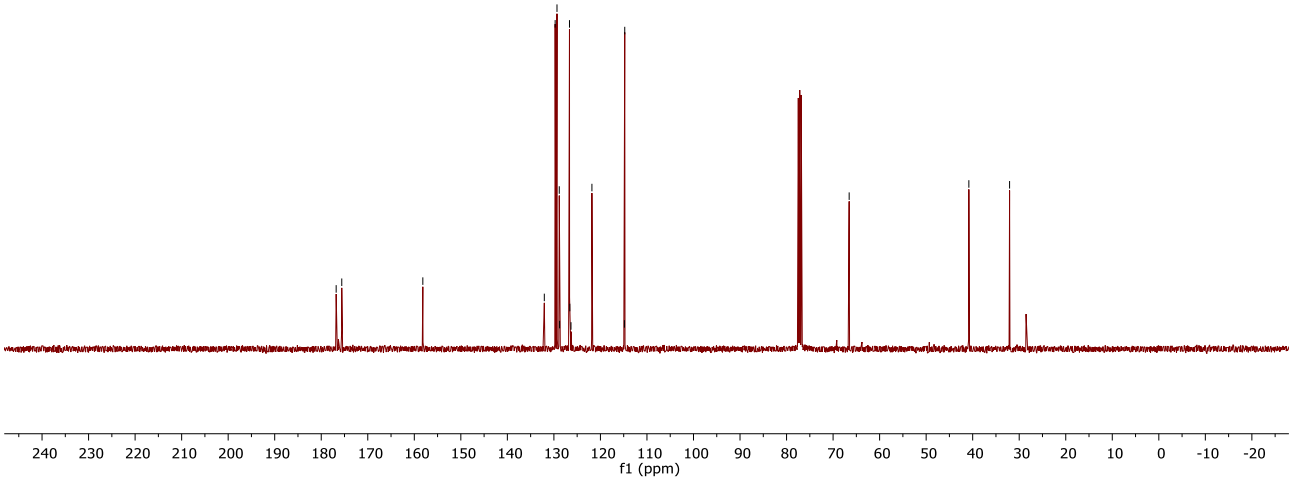

7.52 7.52 7.51 7.50 7.49 7.49 7.47 7.47 7.46 7.44 7.43 7.43 7.42 7.41 7.40 7.33 7.32 7.32 7.31 7.30 7.29 7.29 7.28 7.28 7.26 7.03 7.02 7.01 7.00 7.00 6.98 6.93 6.92 6.92 6.91 6.90 6.90 6.90 4.56 4.54 4.53 4.51 4.26 4.25 4.23 4.22 3.36 3.35 3.34 3.08 3.07 3.05 3.05

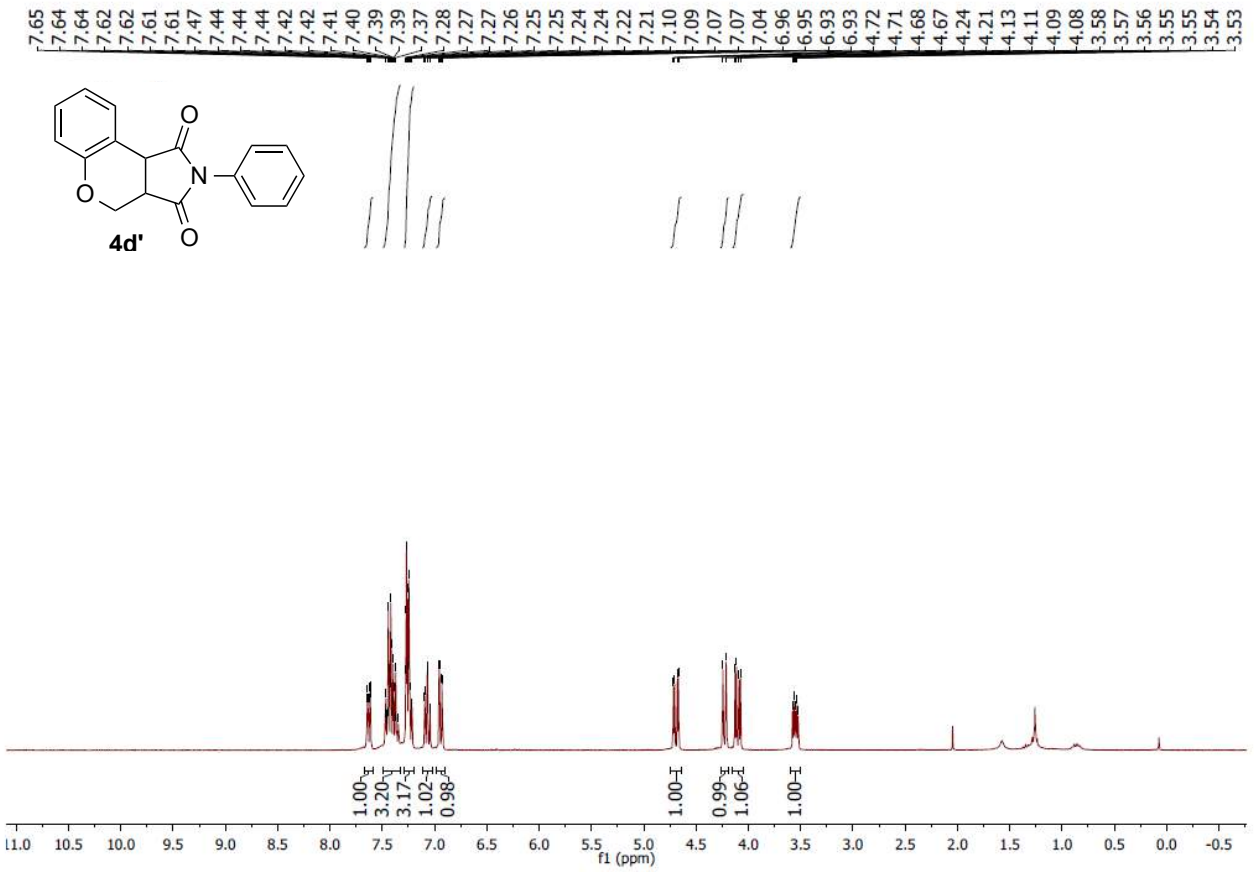
O
O
N–Ph
O

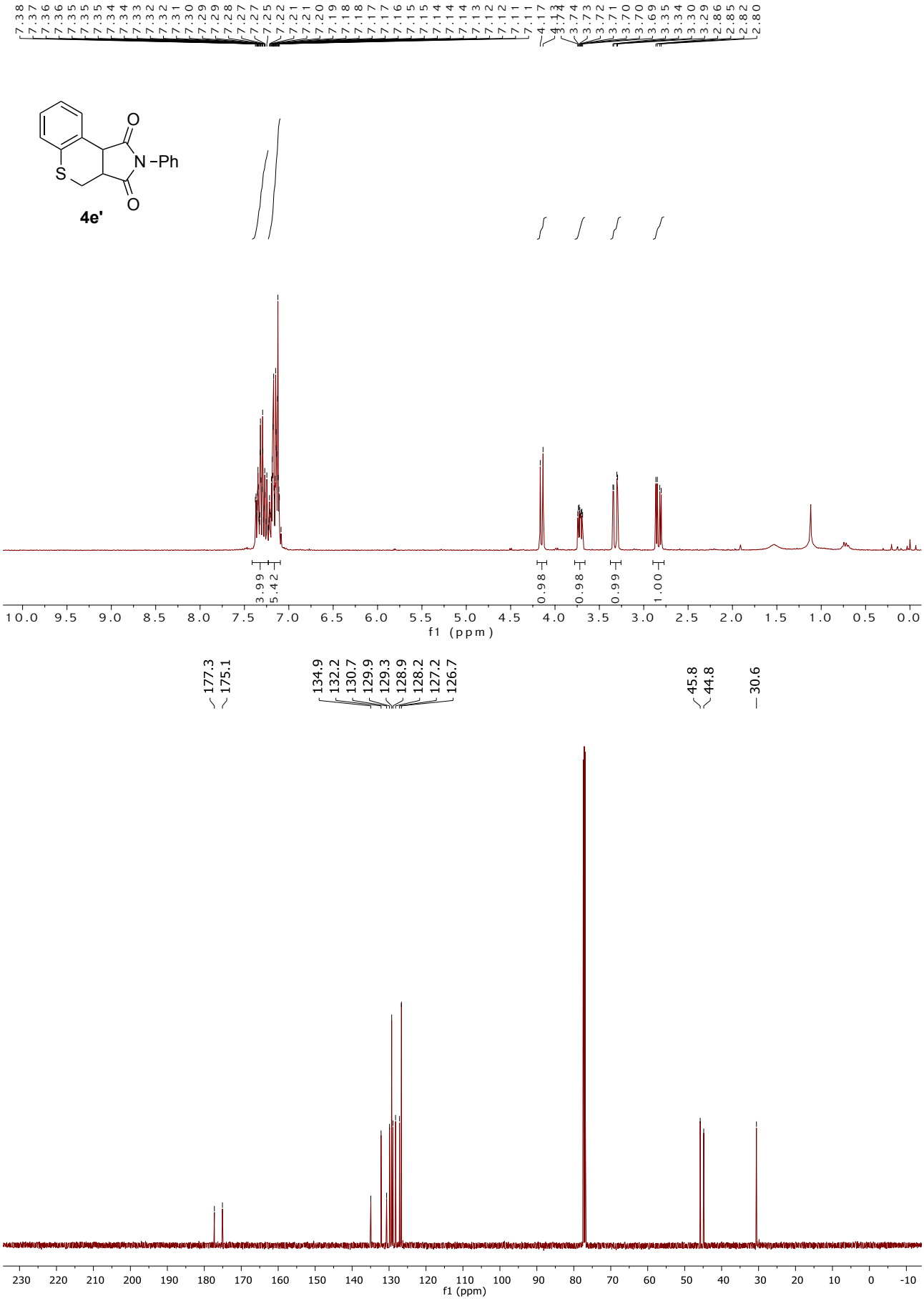
4d


3.08
4.24
1.19
2.04
1.01
1.00
1.05
1.85
f1 (ppm)


176.8
175.6
158.2
132.1
129.7
129.3
128.8
128.8
126.6
126.6
126.3
121.8
114.8
114.8
66.5
40.8
32.1


f1 (ppm)


7.65 7.64 7.64 7.62 7.62 7.61 7.61 7.47 7.44 7.44 7.44 7.42 7.42 7.41 7.40 7.39 7.39 7.37 7.28 7.27 7.27 7.26 7.25 7.25 7.24 7.24 7.22 7.21 7.10 7.09 7.07 7.07 7.04 6.96 6.95 6.93 6.93 4.72 4.71 4.68 4.67 4.24 4.21 4.13 4.11 4.09 4.08 3.58 3.57 3.56 3.55 3.55 3.54 3.53
O
N
O
O
4d'
1.00 3.20 3.17 1.02 0.98 1.00 0.99 1.06 1.00
f1 (ppm)
175.8 175.2 155.4 131.8 130.4 129.3 129.2 128.9 126.4 123.0 118.0 117.4 64.1 42.3 40.0
f1 (ppm)


7.38 7.37 7.36 7.36 7.35 7.35 7.34 7.34 7.33 7.32 7.32 7.31 7.30 7.29 7.29 7.28 7.27 7.27 7.25 7.22 7.21 7.21 7.20 7.19 7.18 7.18 7.17 7.17 7.16 7.15 7.15 7.14 7.14 7.14 7.13 7.12 7.12 7.11 7.11 4.17 4.13 3.74 3.74 3.73 3.72 3.71 3.70 3.70 3.69 3.35 3.34 3.30 3.29 2.86 2.85 2.82 2.80
O
N-Ph
S
O
4e'
3.99
5.42
0.98
0.98
0.99
1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
177.3
175.1
134.9
132.2
130.7
129.9
129.3
128.9
128.2
127.2
126.7
45.8
44.8
30.6
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)