Supporting Information for

Photocatalytic Direct Intermolecular Addition of 1,3-Dithianes

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1. General Information

All commercial reagents were purchased from Sigma-Aldrich, Fluka, Alfa Aesar, TCI, Acros, AKSci, and Energy Chemicals of the highest purity grade. They were used without further purification unless specified. Hexane and ethyl acetate (EtOAc) were distilled before use. Anhydrous THF were distilled from Na. HPLC grade solvents were obtained from suppliers and used as received. All reactions were conducted in 10 cm × 2 cm, 30 mL Pyrex Schlenk tube with PTFE-lined screw cap and a side arm. All experiments were monitored by analytical thin layer chromatography (TLC). TLC was performed on pre-coated plates, Merck 60 F_{254}. After elution, plate was visualized under UV illumination at 254 nm for UV active material. Further visualization was achieved by staining with iodine or $KMnO_4$ stain. Preparative TLC was performed on 0.5 mm silica gel (Merck). Solvents were removed *in vacuo* under ~30 mmHg and heated with a water bath at 40 °C using Eyela or Büchi rotary evaporator with Eyela A-3S aspirator. Columns for flash chromatography (FC) contained silica gel (32–63μ, Merck). Columns were packed as slurry of silica gel in hexane and equilibrated with the appropriate solvent/solvent mixture prior to use. The analyte was loaded neat or as a concentrated solution using the appropriate solvent system. The elution was assisted by applying pressure with constant flow of air.

Solid reagents were weighed using AND HR-200 analytical balance, with accuracy of 0.1 mg. Liquid reagents were measured using Hamilton microsyringes. Reactions requiring temperatures up to −40 °C were stirred in Greatwall DHJF-4002 cryobath with digital temperature controller. Isopropanol was used as the bath medium. ^{1}H and ^{13}C NMR spectra were recorded on Varian 400-MR (400 MHz and 100 MHz, respectively). The peaks were internally referenced to TMS (0.00 ppm) or residual solvent signal. The following abbreviations (or combinations thereof) were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and br = broad. High resolution mass spectra (ESI) were recorded on a Varian 901-MS with Varian HPLC ProStar 210 solvent delivery module, ProStar 410 auto-sampler, and ProStar 335 photodiode array detector. High resolution mass spectra (EI) were recorded on a Finnigan MAT-95XL. IR spectra were recorded on a Bruker ALPHA Ge-ATR FTIR spectrometer. Frequencies were given in reciprocal centimeters (cm^{-1}). Fluorescence spectrum were recorded on a Hitachi F–2500 fluorescence spectrophotometer.

2. Experimental Section

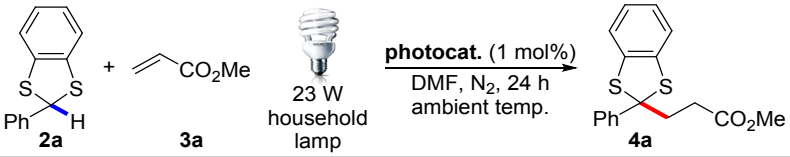
2.1. Standard reaction set-up

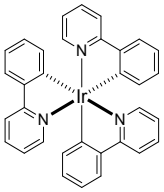
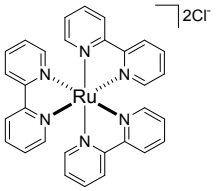
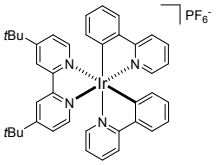
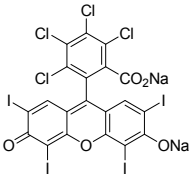
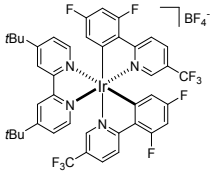
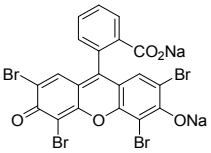
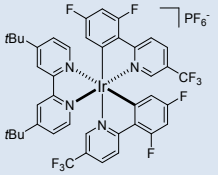
In a typical experiment, a 23 W compact fluorescent light (CFL) bulb (Philips Tornado 23 W cool daylight, model 317643, 6500K color temperature, 1403 Lm) was used. The lamp was purchased from IKEA (Tertial work lamp). The reaction tubes (10 cm × 2 cm, 30 mL Pyrex Schlenk tube with PTFE-lined screw cap and a side arm) were placed approximately 2 cm from the light source. The setup was then covered with aluminum foil to reduce the glares.

Figure S1 Typical experimental set-up

2.2. Optimization studies

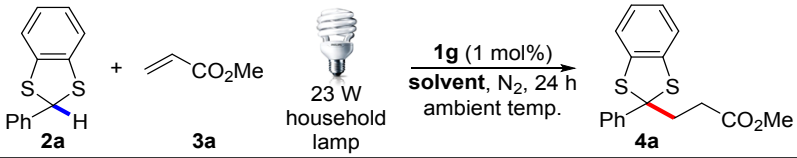
Table S1 Identification of optimal photocatalyst.[a]



entry	photocat.	yield (%)[b]	entry	photocat.	yield (%)[b]
1	—	<5	5	 **1d** $Ir(ppy)_3$ $E_{1/2}(M^*/M^-) = 0.31$ V vs SCE[1]	<5
2	 **1a** $Ru(bpy)_3Cl_2$ $E_{1/2}(M^*/M^-) = 0.77$ V vs SCE[1]	<5	6	 **1e** $Ir(ppy)_2(dtbbpy)PF_6$ $E_{1/2}(M^*/M^-) = 0.66$ V vs SCE[1]	<5
3	 **1b** rose bengal $E_{1/2}(M^*/M^-) = 0.99$ V vs SCE[2]	7	7	 **1f** $[Ir(dF(CF_3)ppy)_2(dtbbpy)]BF_4$ $E_{1/2}(M^*/M^-) = 1.21$ V vs SCE[1]	68
4	 **1c** eosin Y $E_{1/2}(M^*/M^-) = 0.79$ V vs SCE[3]	<5	**8**	 **1g** $[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$ $E_{1/2}(M^*/M^-) = 1.21$ V vs SCE[1]	**75**

[a] Unless otherwise noted, the reaction conditions were as followed: dithiane **2a** (0.10 mmol), methyl acrylate **3a** (0.5 mmol), **photocatalyst** (1 mol%), anhydrous DMF (1.0 mL), 24 h, ambient temperature, irradiated with 23 W 6500K compact fluorescent light under N_2 atmosphere. [b] Yield determined by ^1H NMR analysis of unpurified reaction mixture using $MeNO_2$ as internal standard. Abbreviations: bpy, 2,2'-bipyridyl; ppy, 2-phenylpyridinyl; dtbbpy, 4,4′-di-*tert*-butyl-2,2′-dipyridyl; $dF(CF_3)ppy$, 3,5-difluoro-5'-(trifluoromethyl)-2,2'-bipyridine.

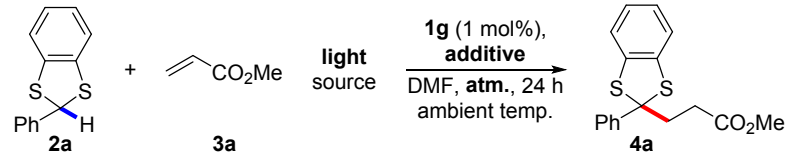
Table S2 Solvent screening.[a]



entry	solvent	yield (%)[b]	entry	solvent	yield (%)[b]
1	hexane	<5	6	THF	<5
2	toluene	<5	7	MeCN	<5
3	Et_2O	<5	8	MeOH	<5
4	EtOAc	<5	9	NMP	61
5	DCM	<5	**10**	**DMF**	**75**

[a] Unless otherwise noted, the reaction conditions were as followed: dithiane **2a** (0.10 mmol), methyl acrylate **3a** (0.5 mmol), **1g** (1 mol%), **solvent** (1.0 mL), 24 h, ambient temperature, irradiated with 23 W 6500K compact fluorescent light under N_2 atmosphere. [b] Yield determined by ^1H NMR analysis of unpurified reaction mixture using $MeNO_2$ as internal standard. Abbreviations: DCM, dichloromethane; THF, tetrahydrofuran; NMP, *N*-methyl-2-pyrrolidone; DMF, dimethylformamide.

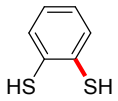
Table S3 Control experiments.[a]



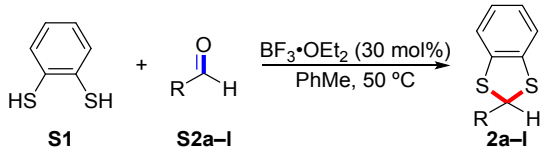
entry	atm.	additive	light	yield (%)[b]	entry	atm.	additive	light	yield (%)[b]
1	O_2	—	23 W CFL	<5	4	N_2	H_2O (1 equiv.)	23 W CFL	<5
2[c]	N_2	—	dark	<5	5	N_2	Et_3N (1 equiv.)	23 W CFL	<5
3[d]	N_2	—	13 W LEDs	71	6	N_2	K_2CO_3 (1 equiv.)	23 W CFL	59

[a] Unless otherwise noted, the reaction conditions were as followed: dithiane **2a** (0.10 mmol), methyl acrylate **3a** (0.5 mmol), **1g** (1 mol%), **additive**, anhydrous DMF (1.0 mL), 24 h, ambient temperature, irradiated with a **light** source under **designated atmosphere**. [b] Yield determined by ^1H NMR analysis of unpurified reaction mixture using $MeNO_2$ as internal standard. [c] Reaction conducted in dark at 40 °C. [d] Philips LED bulb 13W E27 (UV/IR free) was used.

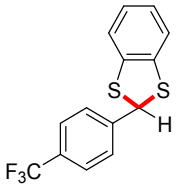
2.3. Synthesis for starting materials



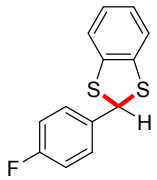
benzene-1,2-dithiol (S1)[4]: Synthesized according to literature procedure from thiophenol.



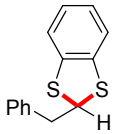
General procedure for the preparation of dithiane derivatives 2: To a solution of corresponding aldehyde **S2** (2.0 mmol, 1.0 equiv.) and dithiol **S1** (2.2 mmol, 1.1 equiv) in toluene (5.0 mL), $BF_3 \cdot OEt_2$ (30 mol%) was added at room temperature. The reaction was stirred at 50 °C and monitored by TLC. Saturated aqueous $NaHCO_3$ was added after completion of reaction (2 h). Organic layer was washed with saturated brine solution, dried over Na_2SO_4, concentrated and purified by column chromatography using hexane and EtOAc as eluents.



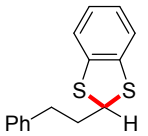
2-(4-(trifluoromethyl)phenyl)benzo[*d*][1,3]dithiole (2c): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 – 7.61 (m, 2H), 7.57 – 7.55 (m, 2H), 7.23 – 7.20 (m, 2H), 7.08 – 7.06 (m, 2H), 6.09 (s, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.5, 136.9, 130.7 (q, J = 32.3 Hz), 127.3, 126.0, 125.7 (q, J = 3.8 Hz), 123.8 (q, J = 270.6 Hz), 122.0, 55.3 ppm.



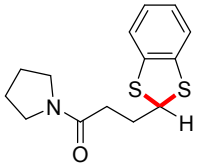
2-(4-fluorophenyl)benzo[*d*][1,3]dithiole (2d): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 – 7.49 (m, 2H), 7.20 – 7.18 (m, 2H), 7.06 – 6.96 (m, 4H), 6.15 (s, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.7 (d, J = 246.7 Hz), 137.3, 135.8 (d, J = 3.2 Hz), 128.9 (d, J = 8.2 Hz), 125.9, 121.9, 115.6 (d, J = 21.6 Hz), 55.8 ppm.



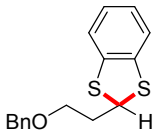
2-benzylbenzo[*d*][1,3]dithiole (2g): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.26 (m, 7H), 7.09 – 7.07 (m, 2H), 5.05 (t, J = 7.0 Hz, 1H), 3.26 (d, J = 7.4 Hz, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.6, 137.2, 129.5, 128.5, 128.5, 127.1, 125.6, 122.7, 55.7, 45.1 ppm.



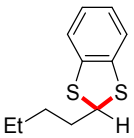
2-phenethylbenzo[*d*][1,3]dithiole (2h): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.20 (m, 7H), 7.08 – 7.06 (m, 2H), 4.79 (t, *J* = 7.2 Hz, 1H), 2.86 – 2.82 (m, 2H), 2.32 – 2.26 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.4, 137.3, 128.6, 126.6, 125.5, 122.6, 53.7, 40.8, 33.0 ppm.



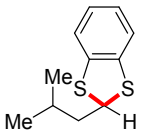
3-(benzo[*d*][1,3]dithiol-2-yl)-1-(pyrrolidin-1-yl)propan-1-one (2i): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 – 7.21 (m, 2H), 7.04 – 7.01 (m, 2H), 4.98 (t, *J* = 7.1 Hz, 1H), 3.44 (t, *J* = 10.1 Hz, 2H), 3.37 (t, *J* = 6.8 Hz, 2H), 2.47 (t, *J* = 7.0 Hz, 2H), 2.26 (q, *J* = 7.0 Hz, 2H), 1.96 – 1.93 (m, 2H), 1.88 – 1.82 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.9, 137.1, 125.4, 122.5, 53.7, 46.5, 45.7, 34.2, 31.0, 26.0, 24.4 ppm.



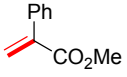
2-(2-(benzyloxy)ethyl)benzo[*d*][1,3]dithiole (2j): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.30 (m, 5H), 7.29 – 7.19 (m, 2H), 7.01 – 6.99 (m, 2H), 5.03 (t, *J* = 6.9 Hz, 1H), 4.49 (s, 2H), 3.60 (t, *J* = 5.6 Hz), 2.21 – 2.16 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.1, 128.4, 127.70, 127.65, 125.4, 122.6, 73.1, 66.7, 51.3, 39.3 ppm.



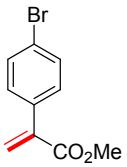
2-butylbenzo[*d*][1,3]dithiole (2k): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.19 (m, 2H), 7.02 – 6.99 (m, 2H), 4.85 (t, *J* = 7.2 Hz, 1H), 1.97 – 1.91 (m, 2H), 1.45, 1.45, 1.44 – 1.40 (m, 2H), 1.35 – 1.29 (m, 2H), 0.90 (t, *J* = 7.3 Hz, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.4, 125.3, 122.4, 54.7, 38.8, 29.3, 22.1, 13.9 ppm.



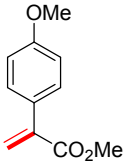
2-isobutylbenzo[*d*][1,3]dithiole (2l): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 – 7.19 (m, 2H), 7.02 – 6.99 (m, 2H), 4.96 (t, *J* = 7.5 Hz, 1H), 1.86 – 1.84 (m, 3H), 0.93 – 0.91 (m, 6H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.5, 125.4, 122.5, 53.1, 47.4, 26.3, 22.1 ppm.



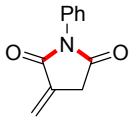
methyl 2-phenylacrylate (3d)[5]: Synthesized according to literature procedure from methyl 2-phenylacetate.



methyl 2-(4-bromophenyl)acrylate (3e)[5]: Synthesized according to literature procedure from methyl 2-(4-bromophenyl)acetate.



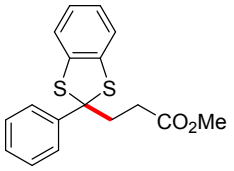
methyl 2-(4-methoxyphenyl)acrylate (3f)[5]: Synthesized according to literature procedure from methyl 2-(4-methoxyphenyl)acetate.



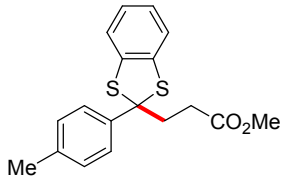
3-methylene-1-phenylpyrrolidine-2,5-dione (3i)[6]: Synthesized according to literature procedure from itaconic anhydride and aniline.

2.4. General procedure for photocatalytic direct intermolecular addition of various 1,3-dithiane derivatives with methyl acrylate

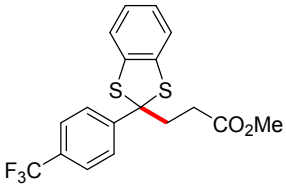
A 30 mL oven dried Schlenk tube (with PTFE-lined screw cap and a side arm) equipped with a magnetic stir bar was charged with dithiane **2** (0.10 mmol, 1.0 equiv.), methyl acrylate (0.2 mmol), and photocatalyst **1g** (1.2 mg, 0.0010 mmol, 1.0 mol%). Dry DMF (1.0 mL) was used to wash down the solids on the sides of wall under N_2 atmosphere. The tube was sealed, evacuated, and backfilled with nitrogen gas repeatedly for three times. It was then placed approximately 2 cm from the light source (23 W 6500K CFL). After stirring for 24 h, the resulting mixture was concentrated *in vacuo* at 50 °C, and the resulting residue was purified by column chromatography or preparative TLC using hexanes/EtOAc as the eluent.



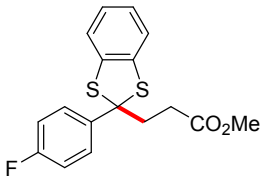
methyl 3-(2-phenylbenzo[*d*][1,3]dithiol-2-yl)propanoate (4a): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 – 7.60 (m, 2H), 7.37 – 7.26 (m, 3H), 7.21 – 7.19 (m, 2H), 7.03 – 7.01 (m, 2H), 3.57 (s, 3H), 2.80 – 2.76 (m, 2H), 2.42 – 2.38 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.9, 140.3, 137.6, 128.5, 128.1, 126.8, 125.8, 122.6, 51.7, 39.6, 30.9 ppm; IR (film): 2965, 1734, 1541, 1445 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{17}H_{16}O_2S_2Na^+$ [M+Na$^+$] 339.0489, found 339.0486.



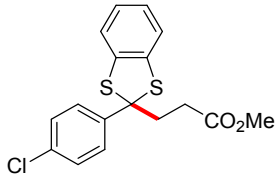
methyl 3-(2-(*p*-tolyl)benzo[*d*][1,3]dithiol-2-yl)propanoate (4b): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 – 7.48 (m, 2H), 7.20 – 7.13 (m, 2H), 7.03 – 7.00 (m, 2H), 3.57 (s, 3H), 2.78 – 2.74 (m, 2H), 2.42 – 2.38 (m, 2H), 2.32 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.0, 138.0, 137.7, 137.1, 129.2, 126.7, 125.7, 122.6, 74.2, 51.7, 39.5, 30.9, 21.0 ppm; IR (film): 2957, 1734, 1557 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{18}H_{18}O_2S_2Na^+$ [M+Na$^+$] 353.0646, found 353.0637.



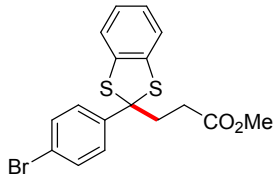
methyl 3-(2-(4-(trifluoromethyl)phenyl)benzo[*d*][1,3]dithiol-2-yl)propanoate (4c): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 – 7.78 (m, 2H), 7.62 – 7.60 (m, 2H), 7.22 – 7.20 (m, 2H), 7.06 – 7.04 (m, 2H), 3.60 (s, 3H), 2.82 – 2.78 (m, 2H), 2.46 – 2.42 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.6, 145.3, 137.1, 130.2 (q, J = 28.9 Hz), 127.3, 126.0, 125.5 (q, J = 3.8 Hz), 123.8 (q, J = 270.9 Hz), 122.6, 73.8, 51.8, 38.8, 31.1 ppm; IR (film): 2951, 1771, 1541, 1362, 1235 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{18}H_{15}O_2F_3S_2Na^+$ [M+Na$^+$] 407.0363, found 407.0365.



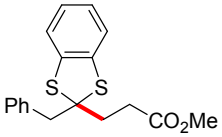
methyl 3-(2-(4-fluorophenyl)benzo[*d*][1,3]dithiol-2-yl)propanoate (4d): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 – 7.61 (m, 2H), 7.20 – 7.18 (m, 2H), 7.04 – 7.00 (m, 2H), 3.59 (s, 3H), 2.79 – 2.75 (m, 2H), 2.44 – 2.40 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.8, 162.2 (d, J = 247.1 Hz), 137.5, 136.5 (d, J = 3.2 Hz), 128.7 (d, J = 8.2 Hz), 125.9, 122.6, 115.3 (d, J = 21.6 Hz), 73.7, 51.7, 39.2, 31.0 ppm; IR (film): 2951, 1735, 1489, 1234, 1164 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{17}H_{15}O_2FS_2Na^+$ [M+Na$^+$] 357.0395, found 357.0386.



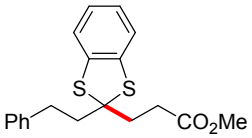
methyl 3-(2-(4-chlorophenyl)benzo[*d*][1,3]dithiol-2-yl)propanoate (4e): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 – 7.59 (m, 2H), 7.33 – 7.31 (m, 2H), 7.21 – 7.19 (m, 2H), 7.05 – 7.03 (m, 2H), 3.61 (s, 3H), 2.79 – 2.75 (m, 2H), 2.44 – 2.41 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.7, 139.5, 137.3, 134.0, 128.6, 128.3, 125.9, 122.6, 73.7, 51.8, 39.0, 31.0 ppm; IR (film): 1734, 1541, 1315 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{17}H_{15}O_2S_2ClNa^+$ [M+Na$^+$] 373.0100, found 373.0091.



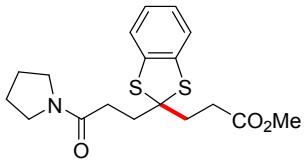
methyl 3-(2-(4-bromophenyl)benzo[*d*][1,3]dithiol-2-yl)propanoate (4f): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 – 7.52 (m, 2H), 7.47 – 7.45 (m, 2H), 7.20 – 7.17 (m, 2H), 7.04 – 7.01 (m, 2H), 3.59 (s, 3H), 2.77 – 2.73 (m, 2H), 2.43 – 2.39 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.7, 140.1, 137.3, 131.5, 128.6, 125.9, 122.6, 122.2, 73.8, 51.8, 38.9, 31.0 ppm; IR (film): 2952, 1716, 1541, 1465, 1258 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{17}H_{15}O_2S_2BrNa^+$ [M+Na$^+$] 416.9595, found 416.9588.



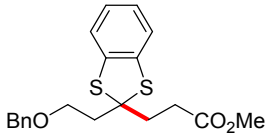
methyl 3-(2-benzylbenzo[*d*][1,3]dithiol-2-yl)propanoate (4g): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.24 (m, 5H), 7.14 – 7.12 (m, 2H), 6.99 – 6.97 (m, 2H), 3.63 (s, 3H), 3.41 (s, 2H), 2.71 – 2.67 (m, 2H), 2.28 – 2.24 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.3, 137.8, 135.8, 130.8, 128.0, 127.3, 125.4, 122.3, 72.8, 51.7, 48.6, 35.1, 31.01 ppm; IR (film): 2951, 1734, 1541, 1197 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{18}H_{16}O_2S_2Na^+$ [M+Na$^+$] 353.0646, found 353.0639.



methyl 3-(2-phenethylbenzo[*d*][1,3]dithiol-2-yl)propanoate (4h): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.25 (m, 2H), 7.20 – 7.14 (m, 5H), 7.02 – 6.99 (m, 2H), 3.67 (s, 3H), 2.90 – 2.86 (m, 2H), 2.70 – 2.66 (m, 2H), 2.42 – 2.33 (m, 4H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.3, 141.0, 137.7, 128.5, 128.4, 126.1, 125.4, 122.2, 72.2, 51.8, 44.4, 36.8, 32.5, 30.8 ppm; IR (film): 2950, 1736, 1445, 1258, 1194 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{19}H_{20}O_2S_2Na^+$ [M+Na$^+$] 367.0802, found 367.0799.

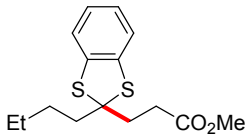


methyl 3-(2-(3-oxo-3-(pyrrolidin-1-yl)propyl)benzo[*d*][1,3]dithiol-2-yl)propanoate (4i): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 – 7.10 (m, 2H), 6.99 – 6.96 (m, 2H), 3.64 (s, 3H), 3.43 (t, J = 6.0 Hz, 2H) 3.35 (t, J = 6.0 Hz, 2H), 2.66 – 2.59 (m, 4H), 2.38 – 2.32 (m, 2H), 1.92 – 1.87 (m, 2H), 1.83 – 1.78 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.3, 170.1, 137.7, 125.4, 122.2, 72.4, 51.8, 46.5, 45.8, 38.0, 36.1, 31.6, 30.6, 26.0, 24.4 ppm; IR (film): 2951, 1734, 1636, 1436, 1225, 1194 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{18}H_{24}NO_3S_2Na^+$ [M+Na$^+$] 366.1198, found 366.1190.

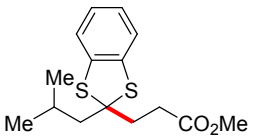


Alkene (5 equiv.) was used.

methyl 3-(2-(2-(benzyloxy)ethyl)benzo[*d*][1,3]dithiol-2-yl)propanoate (4j): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.28 (m, 5H), 7.13 – 7.11 (m, 2H), 6.99 – 6.97 (m, 2H), 4.49 (s, 2H), 3.73 (t, J = 6.6 Hz, 2H), 3.63 (s, 3H), 2.63 – 2.59 (m, 2H), 2.43 – 2.35 (m, 4H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.2, 137.7, 128.4, 127.6, 127.6, 125.4, 122.3, 73.2, 70.7, 67.3, 51.7, 40.8, 37.2, 30.7 ppm; IR (film): 1733, 1557, 1521, 1446, 1260 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{20}H_{22}O_3S_2Na^+$ [M+Na$^+$] 397.0908, found 397.0903.



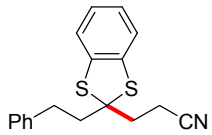
methyl 3-(2-butylbenzo[*d*][1,3]dithiol-2-yl)propanoate (4k): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 – 7.10 (m, 2H), 6.98 – 6.96 (m, 2H), 3.64 (s, 3H), 2.62 – 2.58 (m, 2H), 2.33 – 2.29 (m, 2H), 2.08 – 2.04 (m, 2H), 1.53 – 1.46 (m, 2H), 1.33 (m, 2H), 0.90 (t, J = 7.3 Hz, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.4, 137.8, 125.3, 122.2, 72.5, 51.7, 41.7, 36.8, 30.7, 28.4, 22.7, 13.9 ppm; IR (film): 2956, 1735, 1557, 1445, 1119 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{15}H_{21}O_2S_2^+$ [M+H$^+$] 297.0983, found 297.0976.



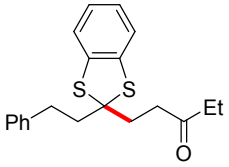
methyl 3-(2-isobutylbenzo[*d*][1,3]dithiol-2-yl)propanoate (4l): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 – 7.10 (m, 2H), 6.98 – 6.96 (m, 2H), 3.64 (s, 3H), 2.61 – 2.57 (m, 2H), 2.34 – 2.30 (m, 2H), 2.06 – 2.01 (m, 2H), 1.99 – 1.89 (m, 1H), 1.01 (d, J = 6.6 Hz, 6H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.4, 137.9, 125.3, 122.1, 71.7, 57.0, 49.2, 37.6, 34.5, 26.2, 24.0 ppm; IR (film): 1716, 1557, 1288 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{15}H_{21}O_2S_2^+$ [M+H$^+$] 297.0983, found 297.0977.

2.5. General procedure for photocatalytic direct intermolecular addition of 1,3-dithiane with various electron-deficient alkenes

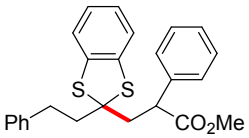
A 30 mL oven dried Schlenk tube (with PTFE-lined screw cap and a side arm) equipped with a magnetic stir bar was charged with dithiane **2h** (25.8 mg, 0.10 mmol, 1.0 equiv.), alkene **3** (0.2 mmol), and photocatalyst **1g** (1.2 mg, 0.0010 mmol, 1.0 mol%). Dry DMF (1.0 mL) was used to wash down the solids on the sides of wall under N_2 atmosphere. The tube was sealed, evacuated, and backfilled with nitrogen gas repeatedly for three times. It was then placed approximately 2 cm from the light source (23 W 6500K CFL). After stirring for 24 h, the resulting mixture was concentrated *in vacuo* at 50 °C, and the resulting residue was purified by column chromatography or preparative TLC using hexanes/EtOAc as the eluent.



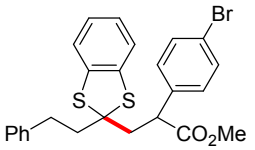
3-(2-phenethylbenzo[*d*][1,3]dithiol-2-yl)propanenitrile (4m): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.24 (m, 2H), 7.21 – 7.14 (m, 5H), 7.04 – 7.02 (m, 2H), 2.91 – 2.87 (m, 2H), 2.72 – 2.68 (m, 2H), 2.39 – 2.32 (m, 4H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.5, 137.0, 128.6, 128.4, 126.3, 125.8, 122.3, 119.2, 71.2, 43.9, 38.4, 32.7, 14.0 ppm; IR (film): 2946, 1734, 1535, 1258, 1008 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{18}H_{18}NS_2^+$ [M+H$^+$] 321.0881, found 321.0878.



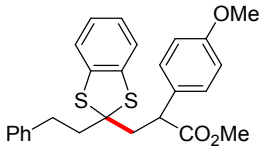
1-(2-phenethylbenzo[*d*][1,3]dithiol-2-yl)pentan-3-one (4n): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.24 (m, 2H), 7.19 – 7.13 (m, 2H), 7.00 – 6.98 (m, 2H), 2.89 – 2.85 (m, 2H), 2.80 – 2.76 (m, 2H), 2.43 (q, *J* = 7.4 Hz, 2H), 2.35 – 2.31 (m, 4H), 1.04 (t, *J* = 7.3 Hz, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.2, 141.1, 137.8, 128.5, 128.4, 126.1, 125.4, 122.2, 72.7, 45.2, 39.0, 36.1, 34.7, 32.3, 7.9 ppm; IR (film): 2951, 1733, 1557, 1456, 1287, 1091 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{20}H_{23}OS_2^+$ [M+H$^+$] 343.1190, found 343.1184.



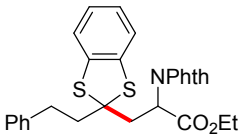
methyl 3-(2-phenethylbenzo[*d*][1,3]dithiol-2-yl)-2-phenylpropanoate (4o): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.21 (m, 7H), 7.16 – 7.09 (m, 5H), 7.01 – 6.98 (m, 2H), 4.23 (dd, *J* = 3.2, 8.9 Hz, 1H), 3.64 (s, 3H), 3.18 (dd, *J* = 9.2, 15.0 Hz, 1H), 2.85 – 2.80 (m, 2H), 2.36 – 2.29 (m, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.2, 141.1, 139.4, 138.0, 137.6, 128.9, 128.4, 127.8, 127.4, 126.0, 125.5, 122.5, 122.3, 72.8, 52.4, 49.1, 45.4, 43.3, 32.1 ppm; IR (film): 2950, 1733, 1445, 1197, 1161 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{25}H_{24}O_2S_2Na^+$ [M+Na$^+$] 443.1115, found 443.1111.



methyl 2-(4-bromophenyl)-3-(2-phenethylbenzo[*d*][1,3]dithiol-2-yl)propanoate (4p): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.42 (m, 2H), 7.25 – 7.23 (m, 2H), 7.19 – 7.10 (m, 7H), 7.02 – 7.00 (m, 2H), 4.20 (dd, *J* = 3.7, 8.7 Hz, 1H), 3.64 (s, 3H), 3.14 (dd, *J* = 8.6, 15.0 Hz, 1H), 2.86 – 2.81 (m, 2H), 2.33 – 2.27 (m, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.8, 140.9, 138.4, 137.8, 137.5, 131.9, 129.6, 128.4, 128.4, 126.0, 125.5, 122.5, 122.3, 121.5, 72.6, 52.5, 48.5, 45.3, 43.4, 32.1 ppm; IR (film): 2952, 1733, 1541, 1456, 1197 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{25}H_{23}O_2S_2BrNa^+$ [M+Na$^+$] 521.0221, found 521.0214.

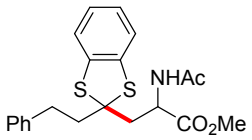


methyl 2-(4-methoxyphenyl)-3-(2-phenethylbenzo[*d*][1,3]dithiol-2-yl)propanoate (4q): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.21 (m, 4H), 7.16 – 7.09 (m, 5H), 7.00 – 6.98 (m, 2H), 6.85 – 6.82 (m, 2H), 4.16 (dd, *J* = 3.4, 8.6 Hz, 1H), 3.77 (s, 3H), 3.63 (s, 3H), 3.13 (dd, *J* = 8.6, 15.0 Hz, 1H), 2.84 – 2.79 (m, 2H), 2.35 – 2.28 (m, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.5, 158.9, 141.1, 138.0, 137.6, 131.4, 128.9, 128.4, 126.0, 125.5, 122.5, 122.3, 114.2, 72.8, 55.3, 52.4, 48.2, 45.3, 43.4, 32.0 ppm; IR (film): 1716, 1569, 1541, 1252 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{26}H_{26}O_3S_2Na^+$ [M+Na$^+$] 473.1221, found 473.1213.



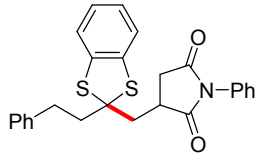
Alkene (1 equiv.) was used.

ethyl 2-(1,3-dioxoisoindolin-2-yl)-3-(2-phenethylbenzo[*d*][1,3]dithiol-2-yl)propanoate (4r): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 – 7.84 (m, 2H), 7.75 – 7.73 (m, 2H), 7.24 – 7.20 (m, 2H), 7.16 – 7.11 (m, 2H), 7.04 – 6.91 (m, 4H), 5.38 (dd, *J* = 2.8, 9.9 Hz, 1H), 4.22 – 4.12 (m, 2H), 3.23 (dd, *J* = 9.8, 15.7 Hz, 1H), 3.07 (dd, *J* = 2.8, 15.7 Hz, 1H), 2.88 – 2.83 (m, 2H), 2.43 – 2.39 (m, 2H), 1.18 (t, *J* = 7.1 Hz, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.8, 167.5, 140.7, 137.6, 137.1, 134.2, 132.0, 128.4, 128.4, 126.1, 125.5, 125.5, 123.6, 122.5, 122.2, 71.2, 62.4, 49.8, 44.0, 38.3, 32.1, 14.0 ppm; IR (film): 1733, 1541, 1386, 1258 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{28}H_{26}NO_4^+$ [M+H$^+$] 504.1303, found 504.1298.



Alkene (1 equiv.) was used.

methyl 2-acetamido-3-(2-phenethylbenzo[*d*][1,3]dithiol-2-yl)propanoate (4s): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.23 (m, 2H), 7.20 – 7.12 (m, 5H), 7.03 – 6.98 (m, 2H), 6.10 (d, *J* = 8.5 Hz, 1H), 4.91 (td, *J* = 4.3, 8.7 Hz, 1H), 3.73 (s, 3H), 2.89 – 2.83 (m, 2H), 2.71 (dd, *J* = 4.3, 15.1 Hz, 1H), 2.57 (dd, *J* = 11.5, 16.3 Hz, 1H), 2.45 – 2.39 (m, 2H), 2.00 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.2, 169.8, 140.6, 137.7, 136.8, 128.5, 128.4, 126.1, 125.7, 125.7, 122.4, 122.3, 70.6, 52.7, 50.2, 43.2, 42.5, 32.1, 23.3 ppm; IR (film): 3200, 2952, 1747, 1653, 1522, 1434, 1288, 1204 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{21}H_{24}NO_3S_2^+$ [M+H^+] 402.1998, found 402.1993.

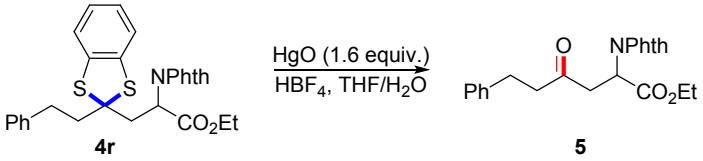


3-((2-phenethylbenzo[*d*][1,3]dithiol-2-yl)methyl)-1-phenylpyrrolidine-2,5-dione (4t): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.35 (m, 3H), 7.30 – 7.23 (m, 5H), 7.21 – 7.17 (m, 4H), 7.06 – 7.02 (m, 2H), 3.51 – 3.44 (m, 1H), 3.06 – 2.89 (m, 5H), 2.51 – 2.36 (m, 2H), 2.26 – 2.20 (m, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.4, 175.4, 140.7, 137.5, 131.8, 129.1, 128.6, 128.5, 128.4, 126.3, 126.2, 125.9, 125.8, 122.4, 122.3, 71.9, 45.0, 44.4, 38.6, 37.1, 32.7 ppm; IR (film): 1733, 1541, 1270, 1181 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{26}H_{23}NO_2S_2Na^+$ [M+Na^+] 468.1068, found 468.1063.

2.6. Scalable photocatalytic direct intermolecular addition of 1,3-dithiane

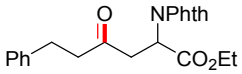
A 30 mL oven dried Schlenk tube (with PTFE-lined screw cap and a side arm) equipped with a magnetic stir bar was charged with dithiane **2h** (258 mg, 1.00 mmol, 1.0 equiv.), alkene **3r** (245 mg, 1.00 mmol, 1.0 equiv.), and photocatalyst **1g** (5.8 mg, 0.0050 mmol, 0.5 mol%). Dry DMF (4.0 mL) was used to wash down the solids on the sides of wall under N_2 atmosphere. The tube was sealed, evacuated, and backfilled with nitrogen gas repeatedly for three times. It was then placed approximately 2 cm from the light source (23 W 6500K CFL). After stirring for 24 h, the resulting mixture was concentrated *in vacuo* at 50 °C, and the resulting residue was purified by column chromatography using hexanes/EtOAc (5/1) as the eluent to give *α*-amino acid derivative **4r** as a yellow solid (466 mg, 92% yield).

2.7. Procedure for deprotection of 1,3-dithiane



4r **5**

Amino acid derivative **4r** (252 mg, 0.500 mmol, 1.0 equiv.) was dissolved in THF (1.0 mL). The resulting solution was added to a suspension of HgO (173 mg, 0.800 mmol, 1.6 equiv.) in H_2O (1.0 mL). This was followed by the addition of aqueous HBF_4 (0.20 mL, 0.2 M). After 15 h of stirring, solid $NaHCO_3$ was added until the solution turned basic on the pH scale. The reaction mixture was filtered through Celite and washed with diethyl ether. The filtrate was extracted with diethyl ether (2 × 3 mL). The combined organic phases were removed under reduced pressure to give the crude product. Purification by flash chromatography provided ketone **5** as the product (80% yield).



ethyl 2-(1,3-dioxoisoindolin-2-yl)-4-oxo-6-phenylhexanoate (5): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.83 (m, 2H), 7.73 – 7.71 (m, 2H), 7.23 – 7.19 (m, 2H), 7.15 – 7.12 (m, 3H), 5.47 (dd, *J* = 6.6, 7.6 Hz, 1H), 4.20 – 4.12 (m, 2H), 3.48 (dd, *J* = 6.4, 17.8 Hz, 1H), 3.13 (dd, *J* = 7.7, 17.8 Hz, 1H), 2.91 – 2.74 (m, 4H), 1.17 (t, *J* = 6.8 Hz, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.4, 168.7, 167.3, 140.6, 134.2, 131.8, 128.4, 128.2, 126.1, 123.6, 62.2, 47.5, 44.3, 41.7, 29.4, 14.0 ppm; IR (film): 2910, 1716, 1541, 1418, 1100 cm^{-1}; HRMS(*m/z*, ESI): Calcd for $C_{22}H_{21}NO_5Na^+$ [M+Na$^+$] 402.1317, found 402.1311.

2.8. Preliminary mechanistic studies

Fluorescent quenching experiment

In a typical experiment, to a 2.0 × 10^{-6} M solution of photocatalyst **1g** in DMF was added the appropriate amount of quencher (dithiane **2c**) in a quartz cuvette. Then the solution was excited at 380 nm and the fluorescence emission intensity was collected at 480 nm. The readings were taken as an average of two runs.

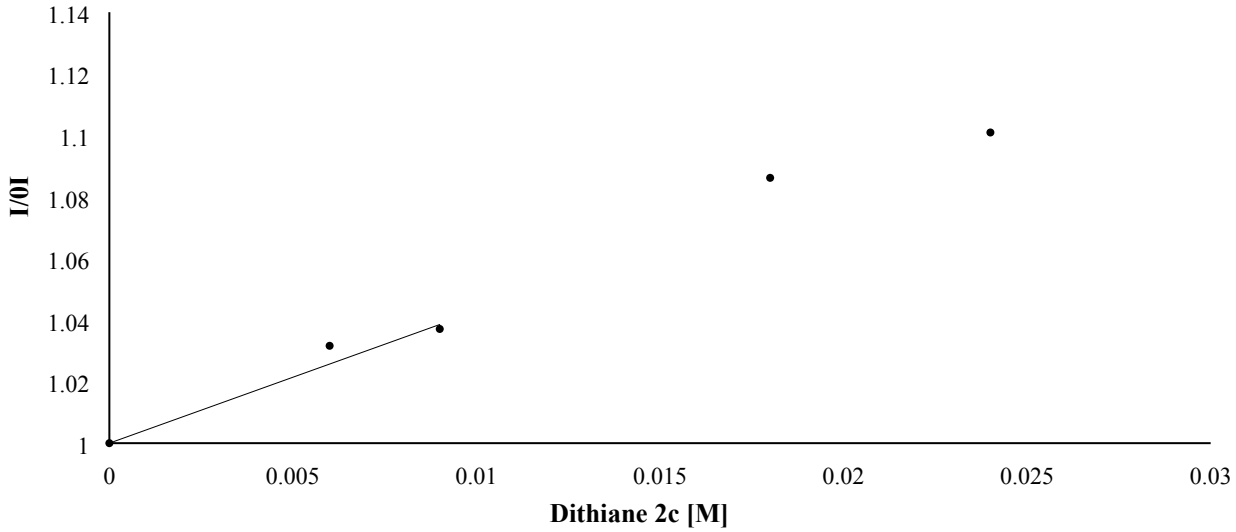


Figure S2 Plot of fluorescence intensity of photocatalyst **1g** versus concentration of dithiane **2c**.

Light/dark experiments

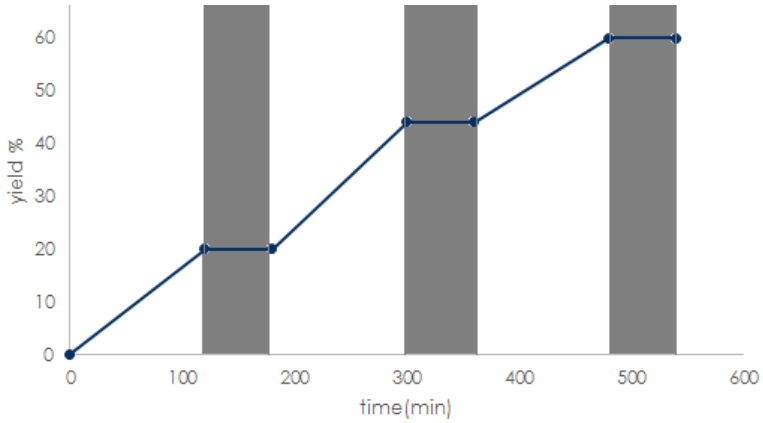
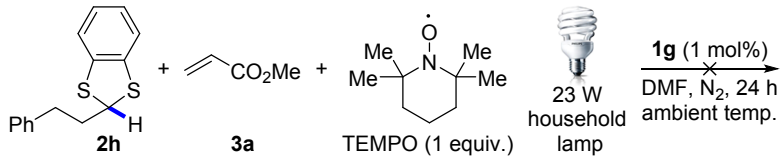


Figure S3 Light/dark experiments.

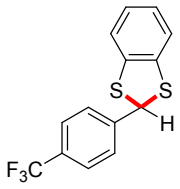
Inhibition of radical reaction by TEMPO



3. References

1 C. K. Prier, D. A. Rankic and D. W. C. MacMillan, *Chem. Rev.*, 2013, **113**, 5322.
2 D. Ravelli, M. Fagnoni and A. Albini, *Chem. Soc. Rev.*, 2013, **42**, 97.
3 D. DiRocco, http://www.princeton.edu/chemistry/macmillan/resources/Merck-Photocatalysis-Chart.pdf
4 D. M. Giolando and K. Kirschbaum, *Synthesis*, 1992, 451.
5 J. Pietruszka and M. Schölzel, *Adv. Synth. Catal.*, 2012, **354**, 751.
6 D. Leow, S. Lin, S. K. Chittimalla, X. Fu and C.-H. Tan, *Angew. Chem. Int. Ed.*, 2008, **47**, 5641.

4. NMR Spectra of New Compounds



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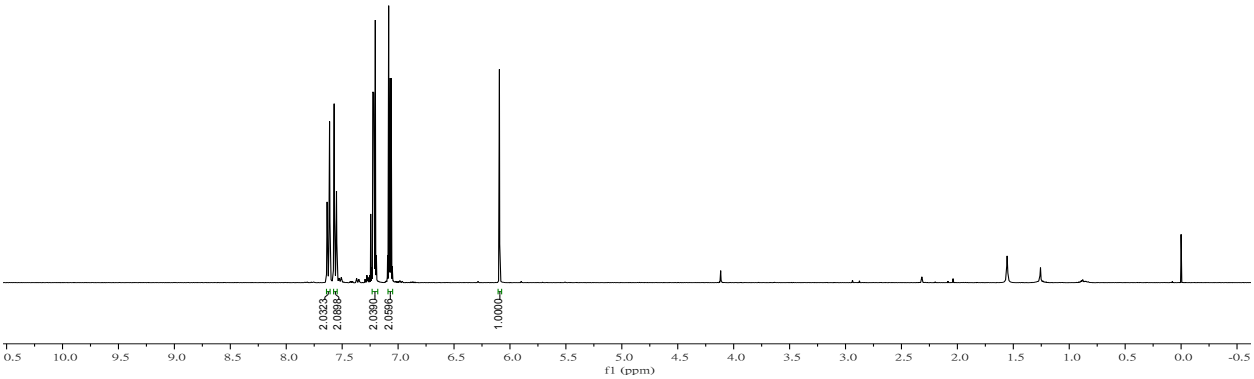
2c



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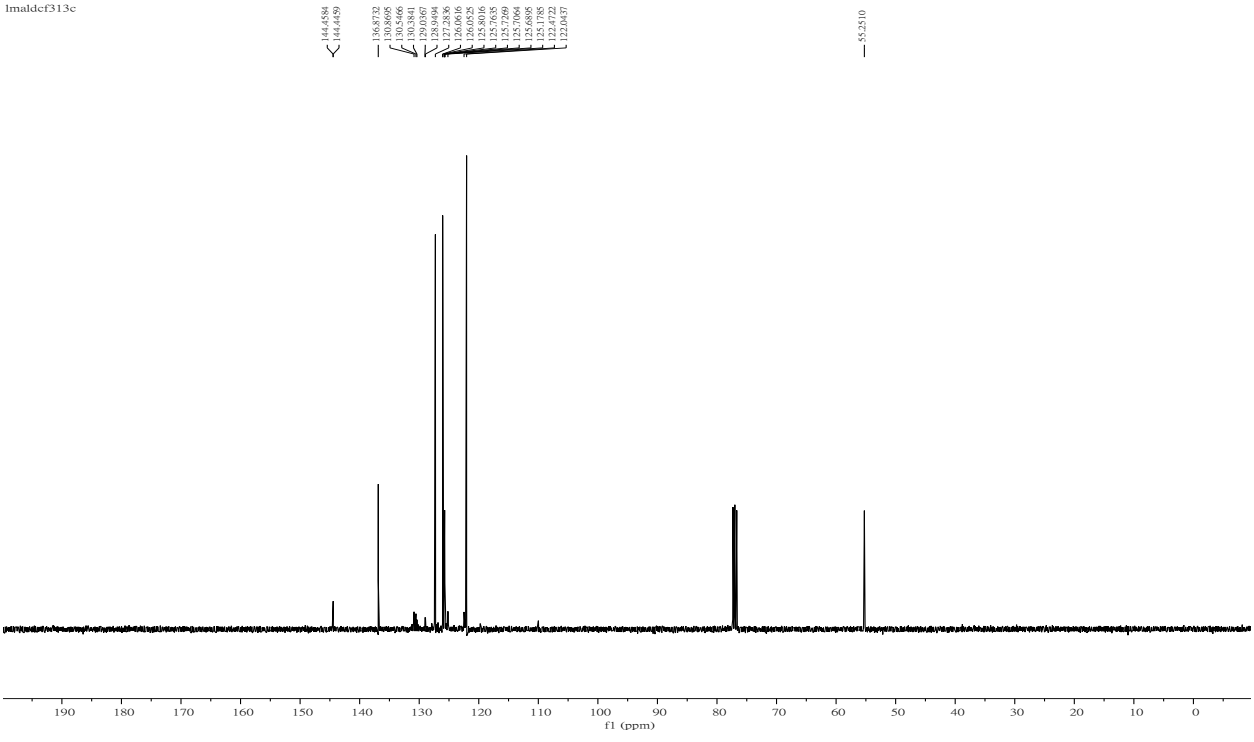
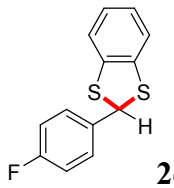
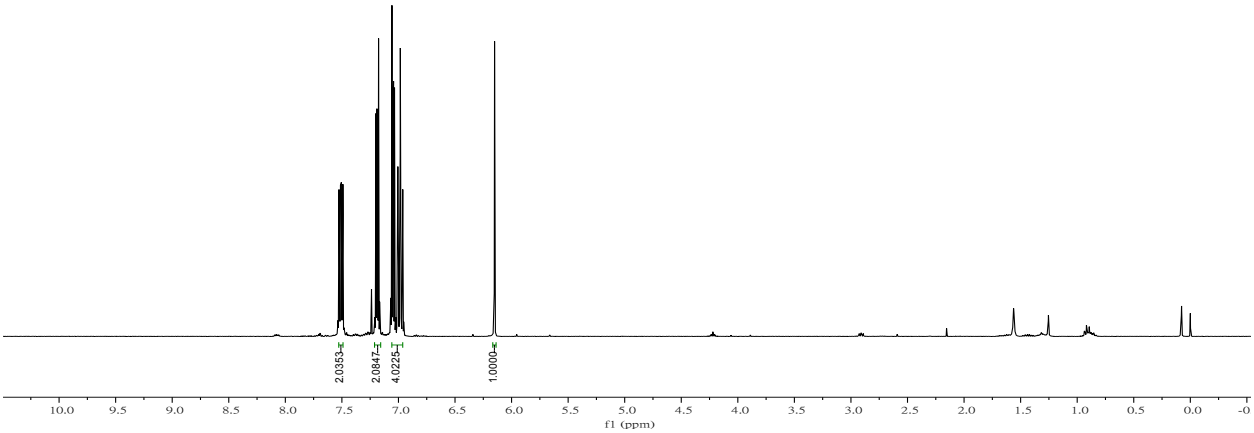




2d

lmald4f1h



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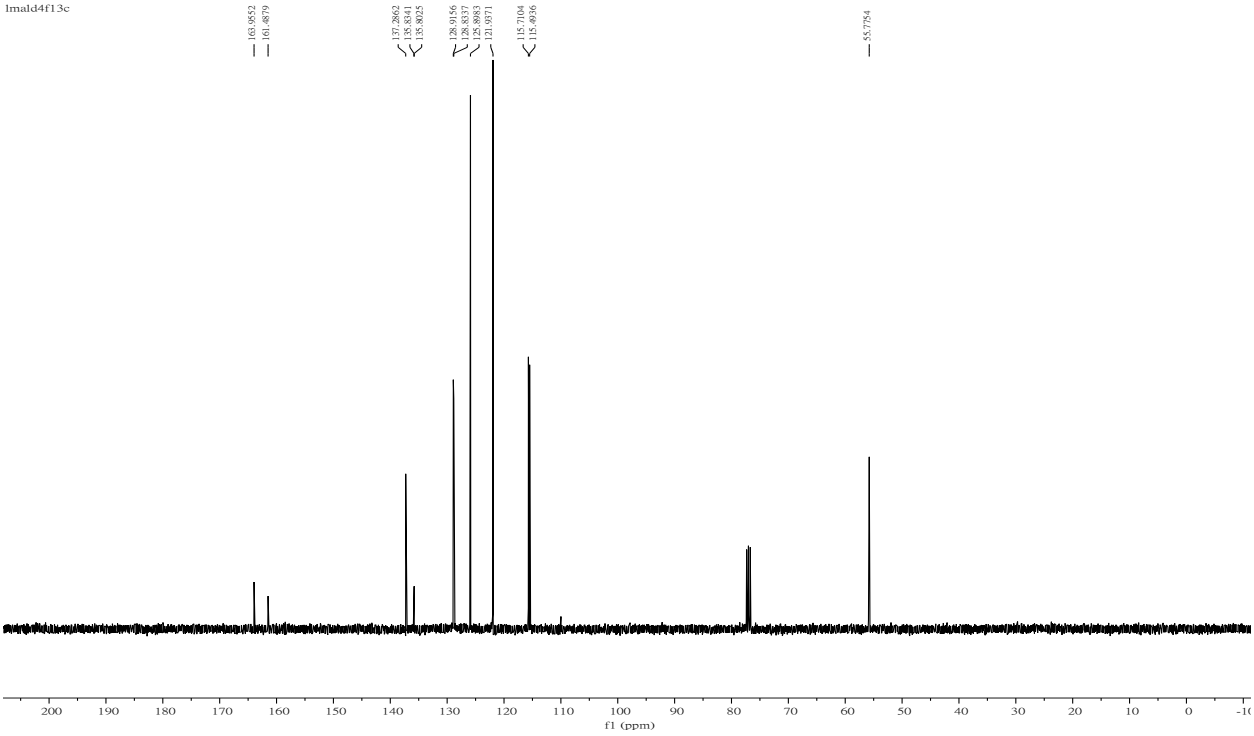
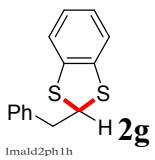
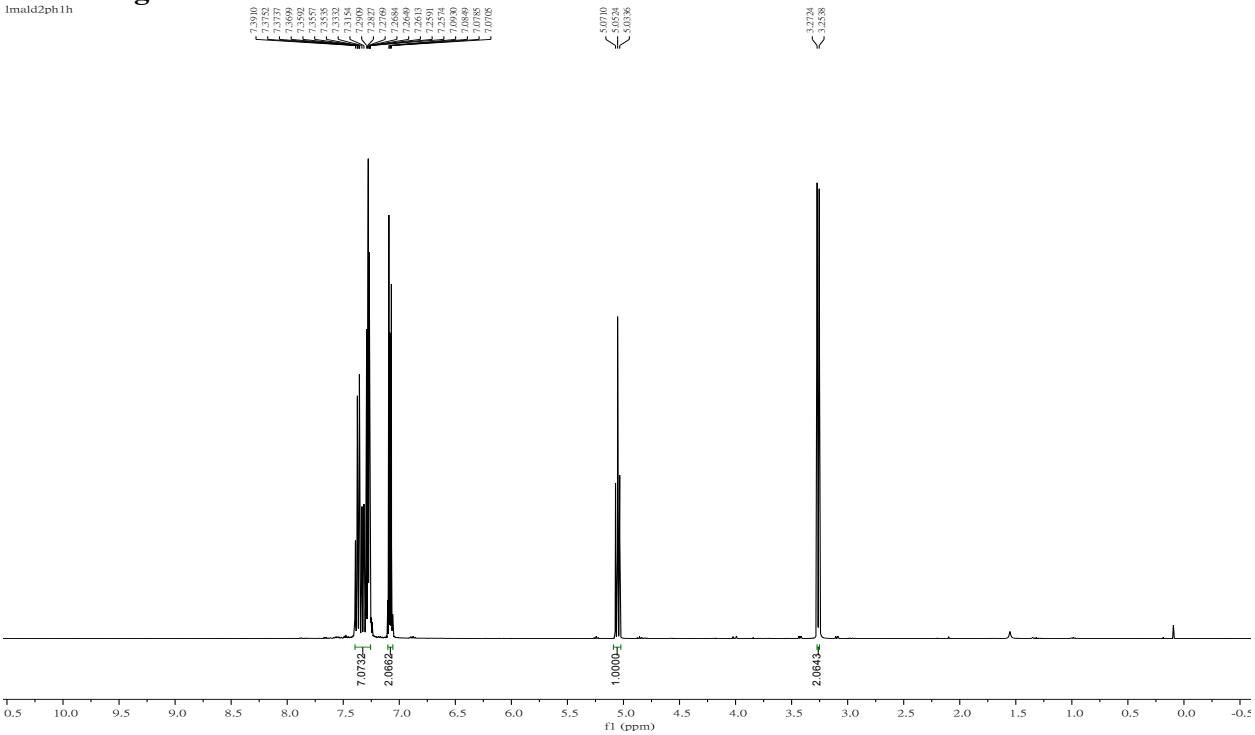




2g

lmald2ph1h



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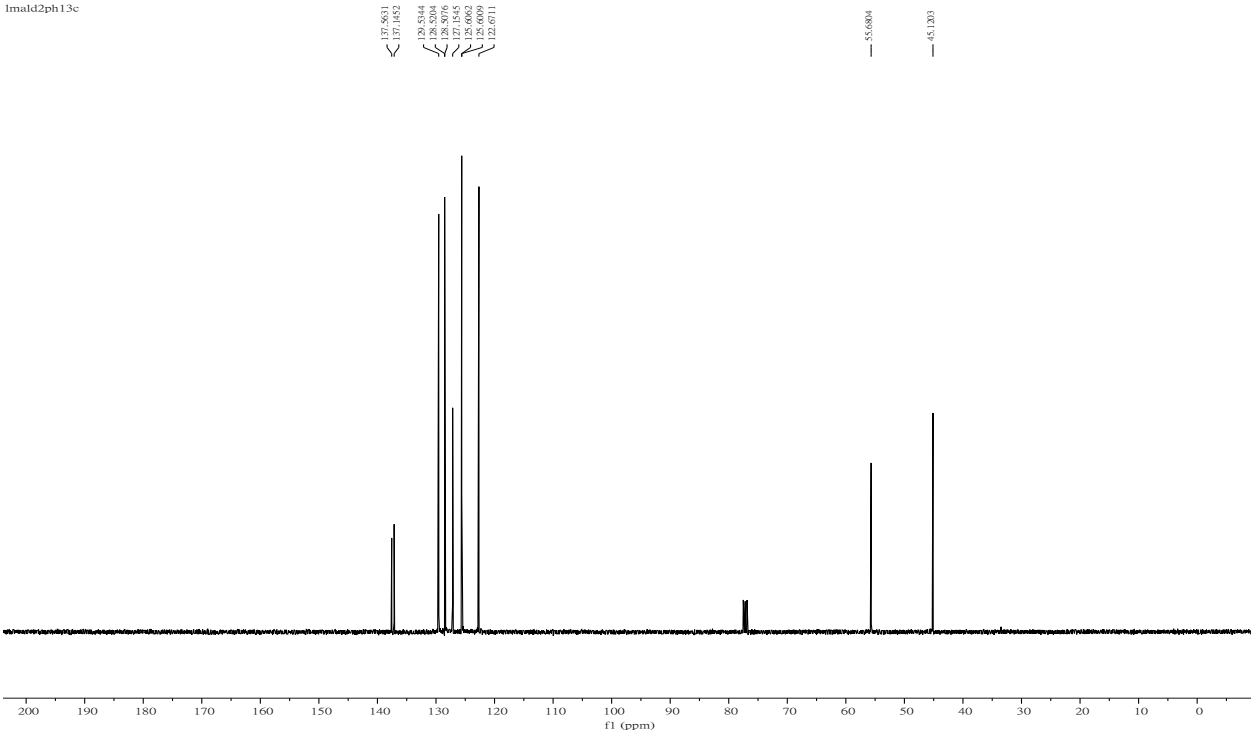

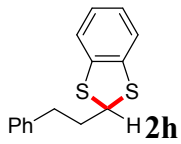

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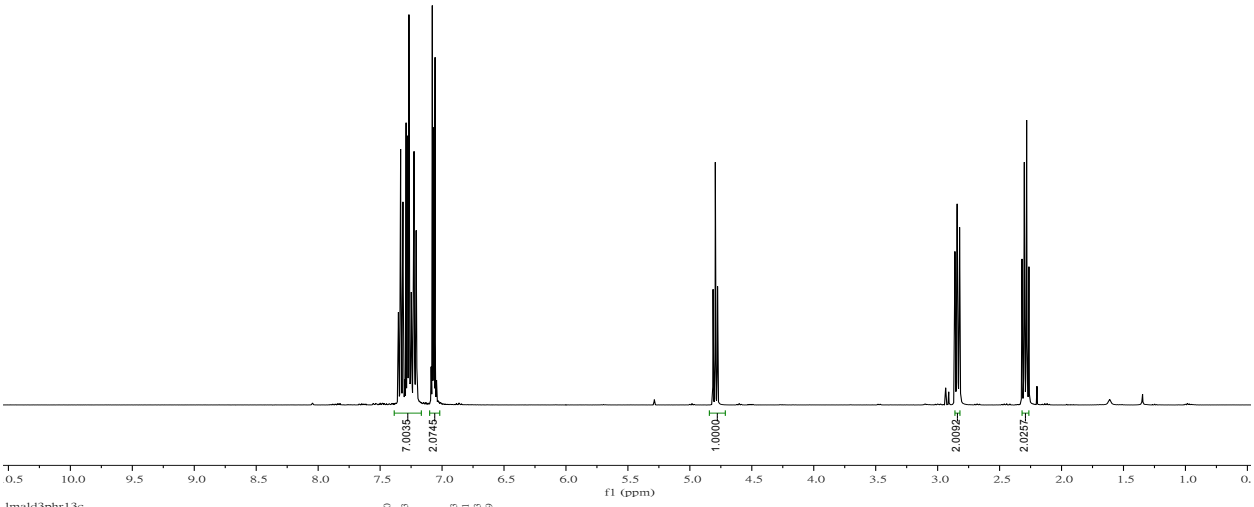

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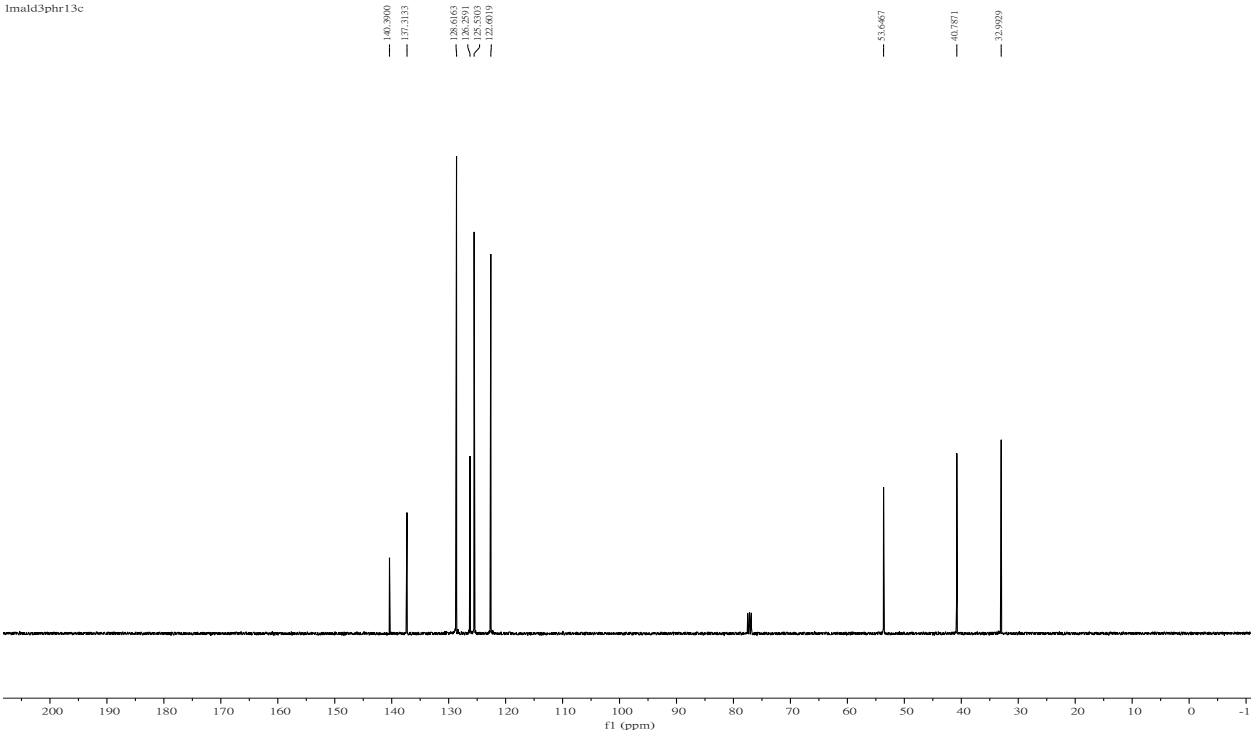

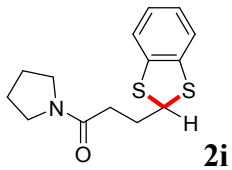

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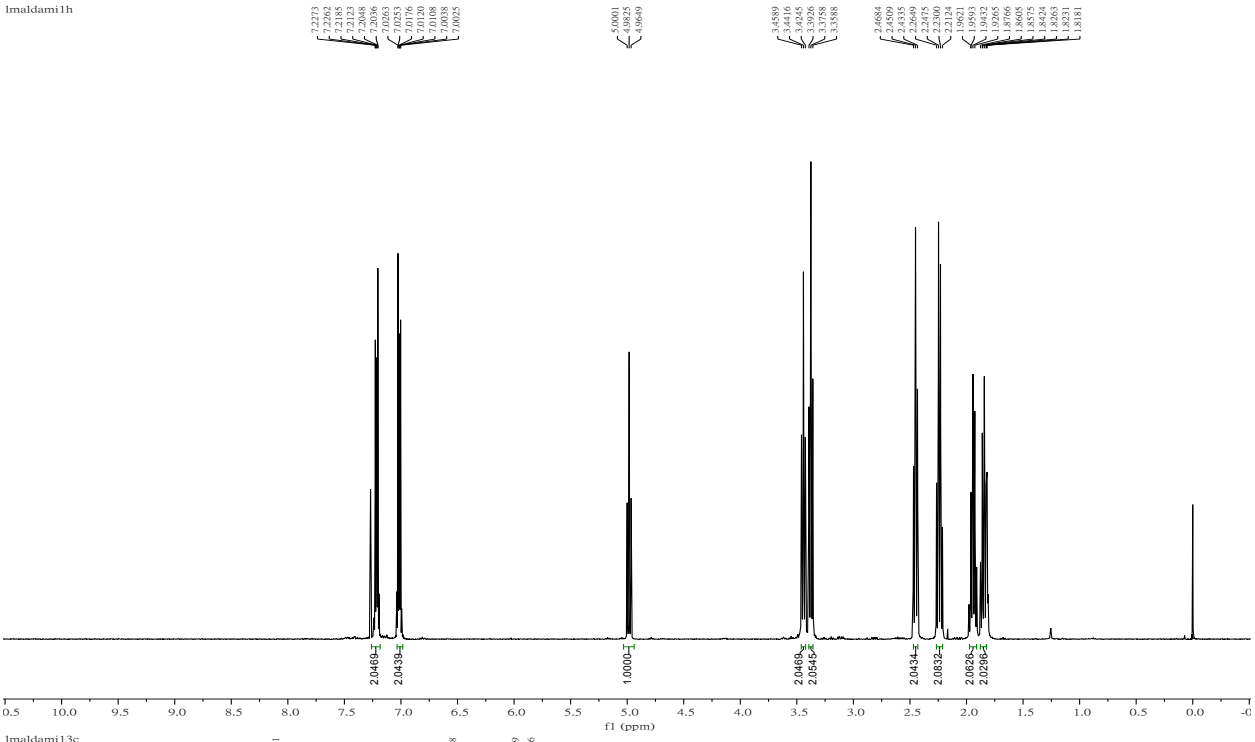

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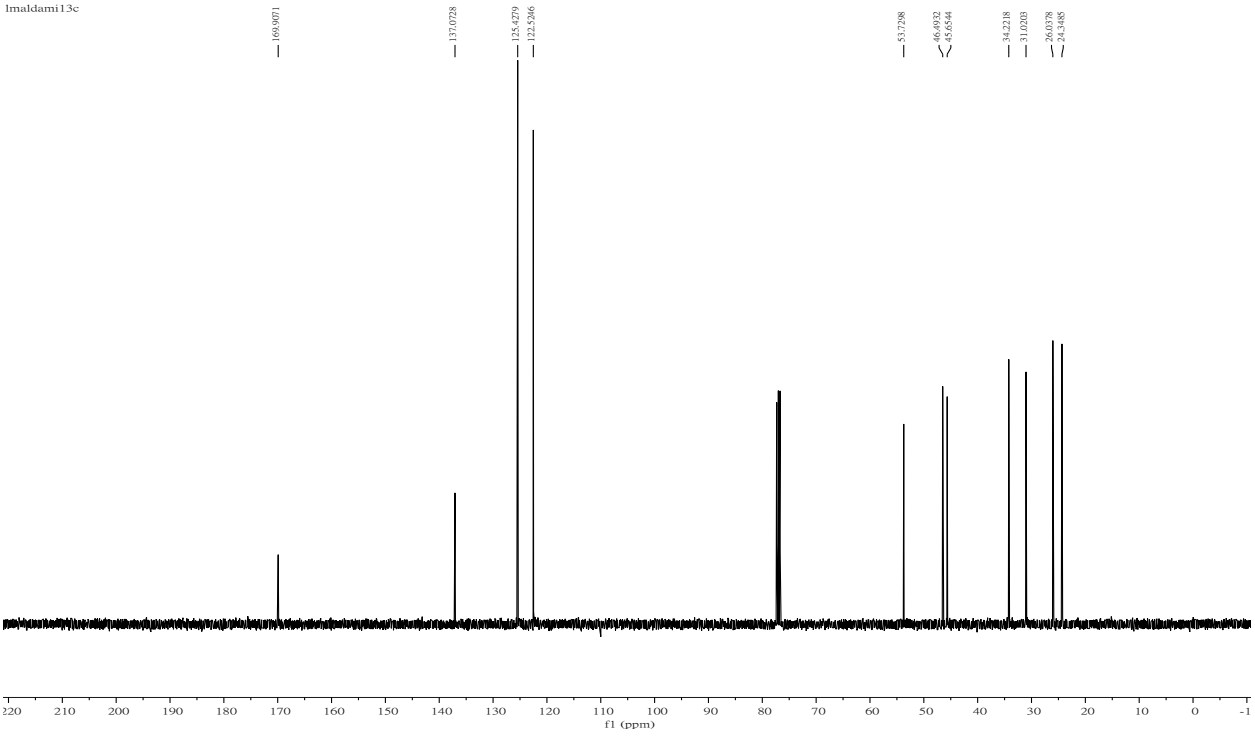

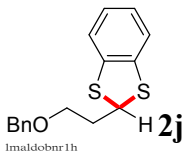

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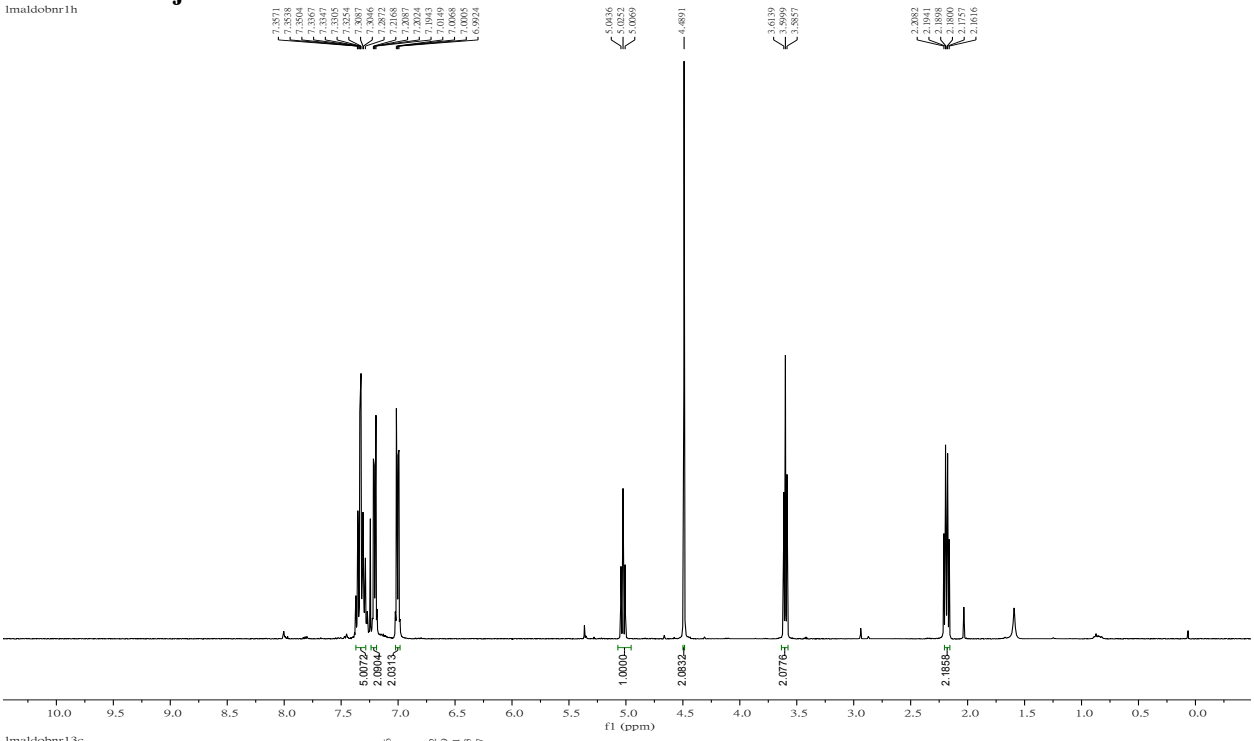

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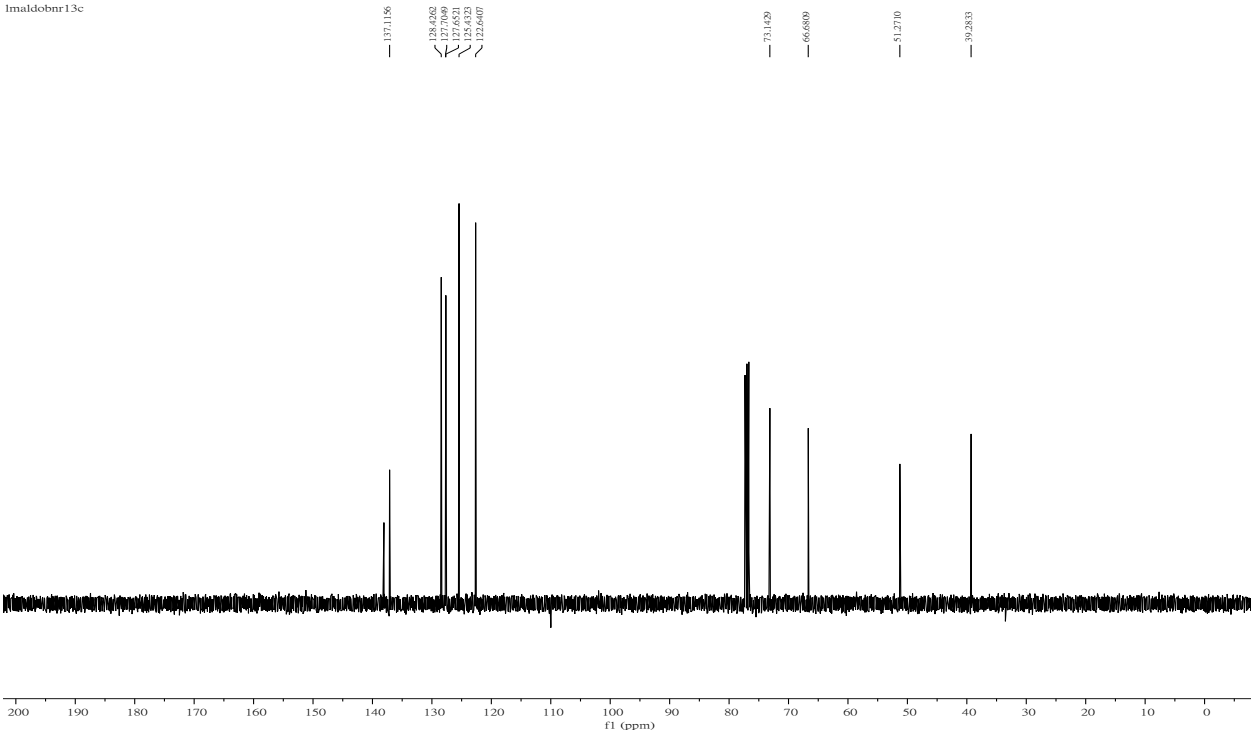

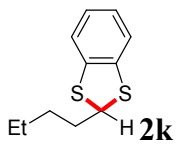

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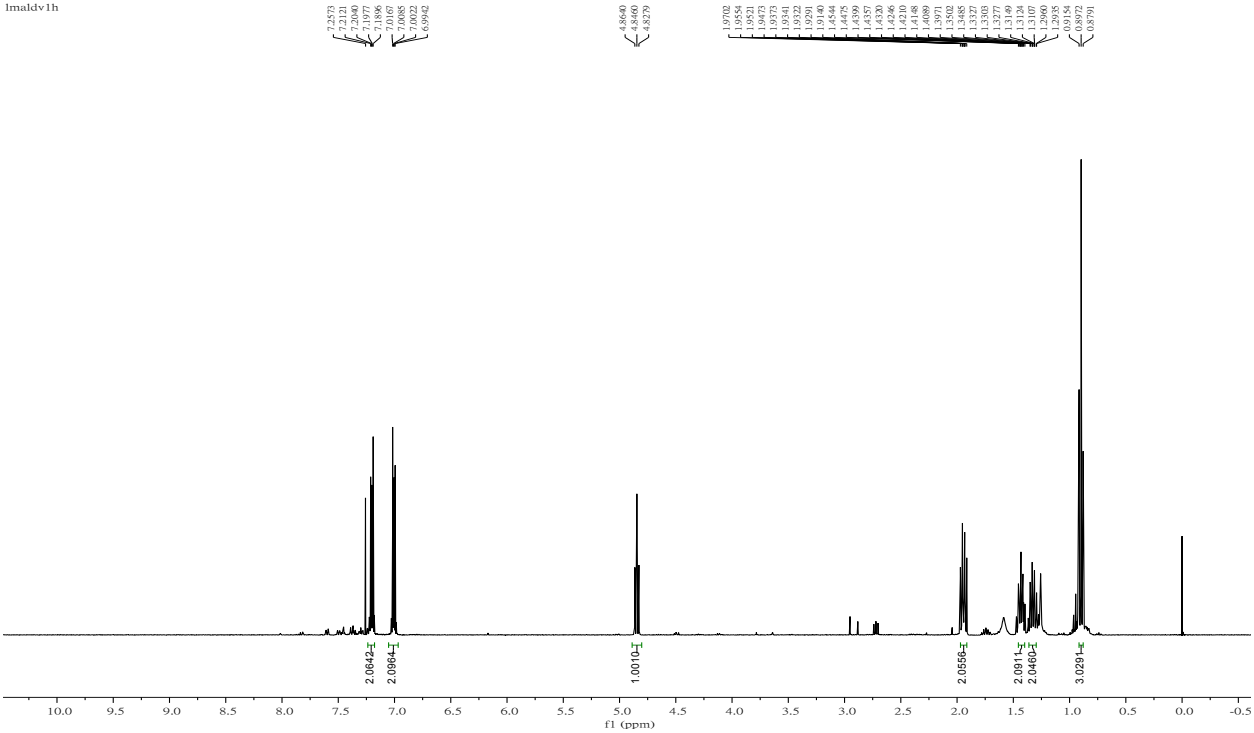

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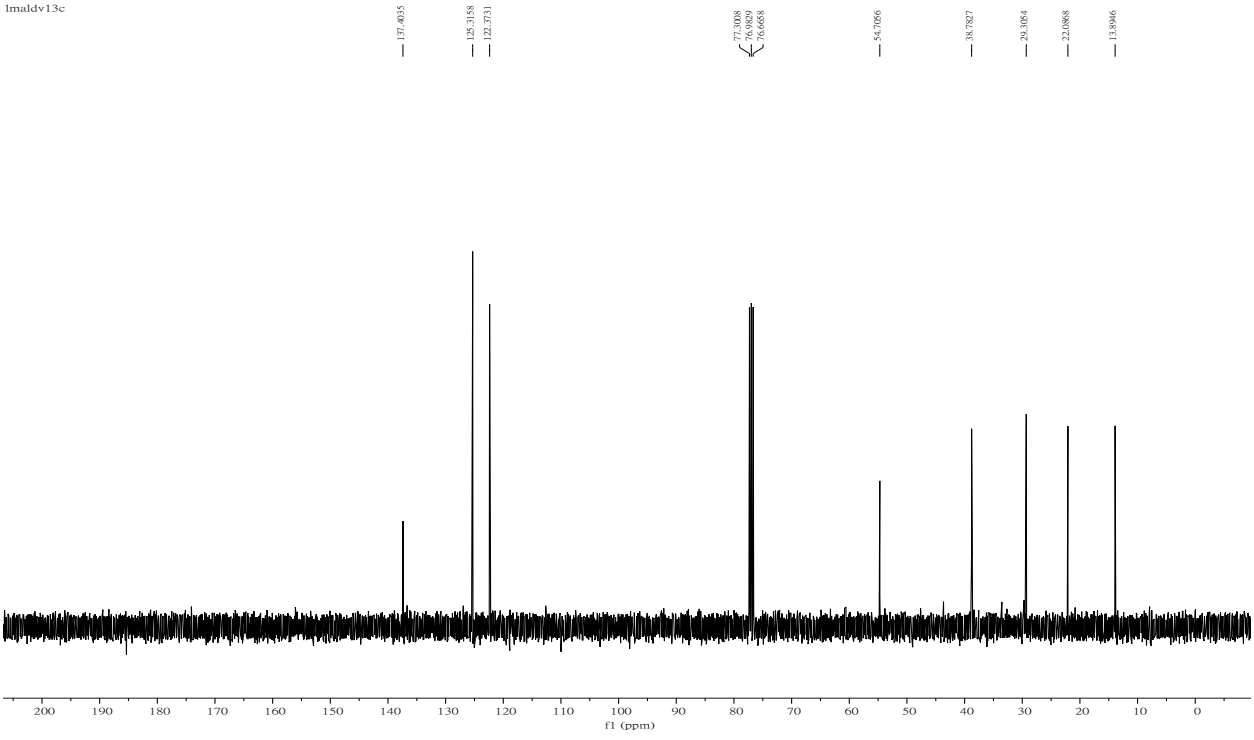

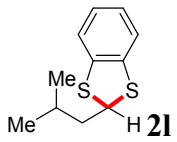

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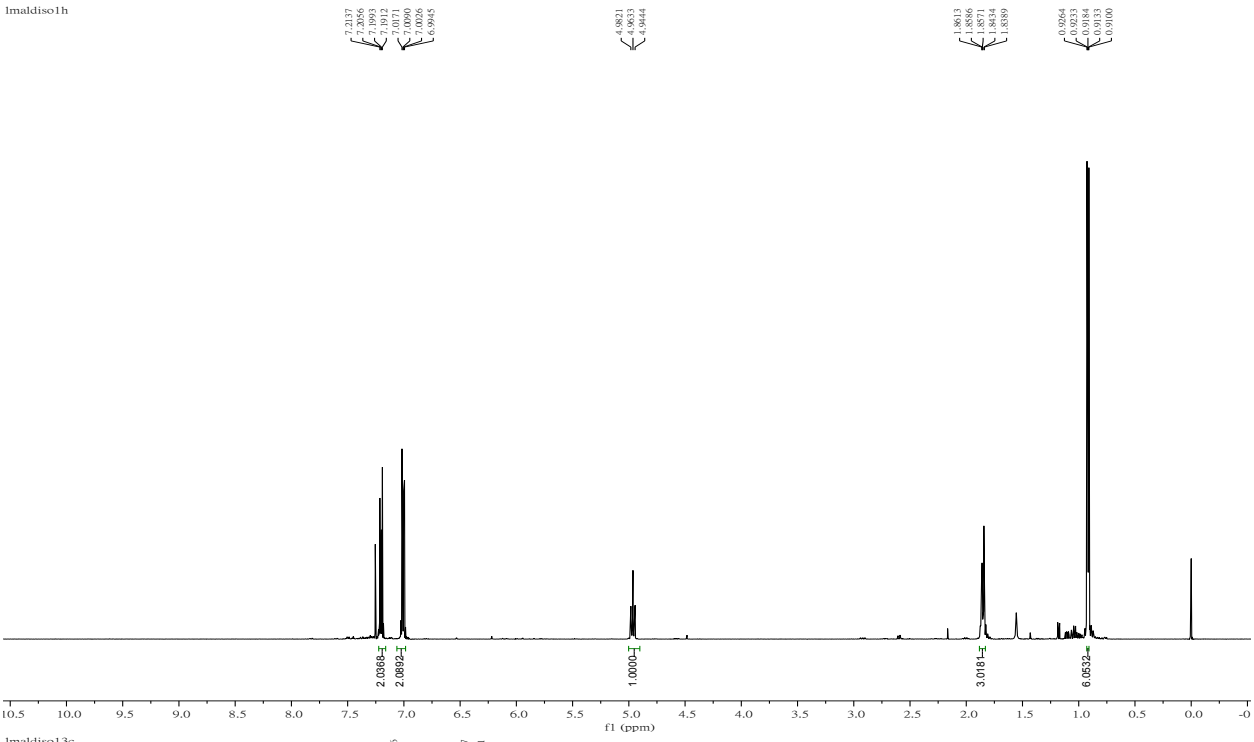

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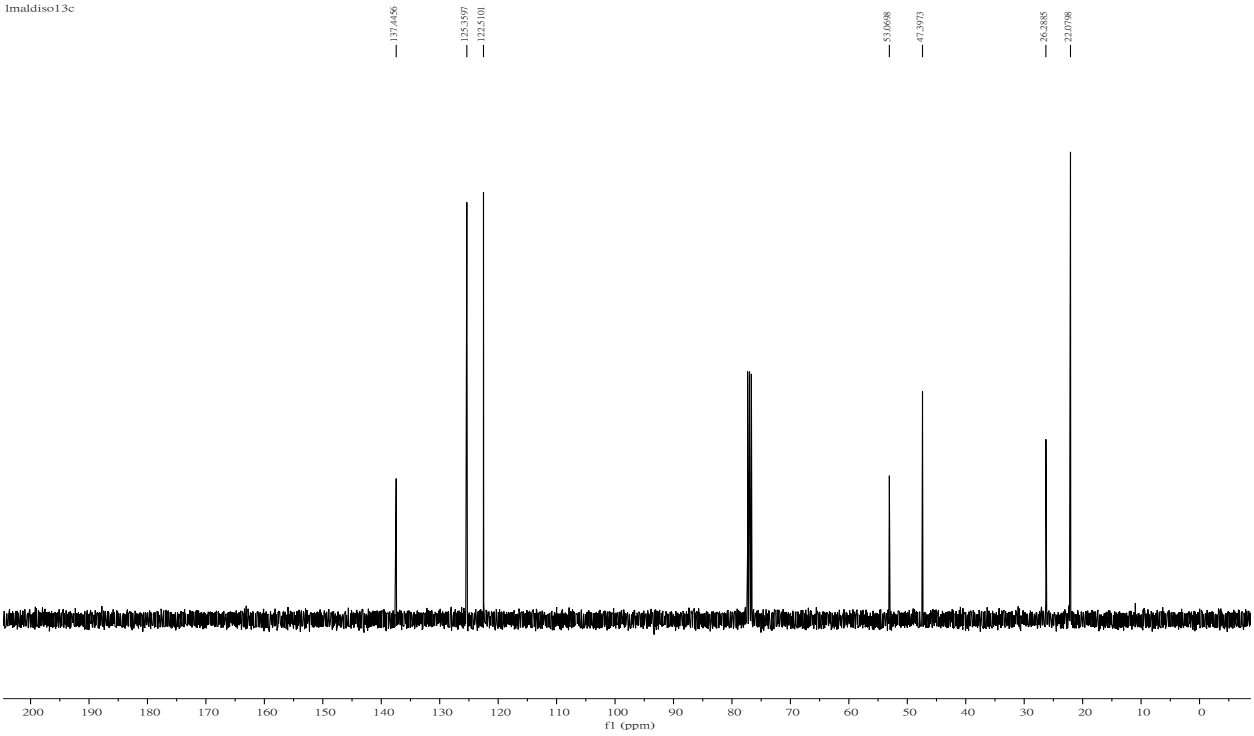

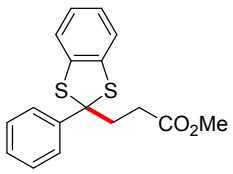
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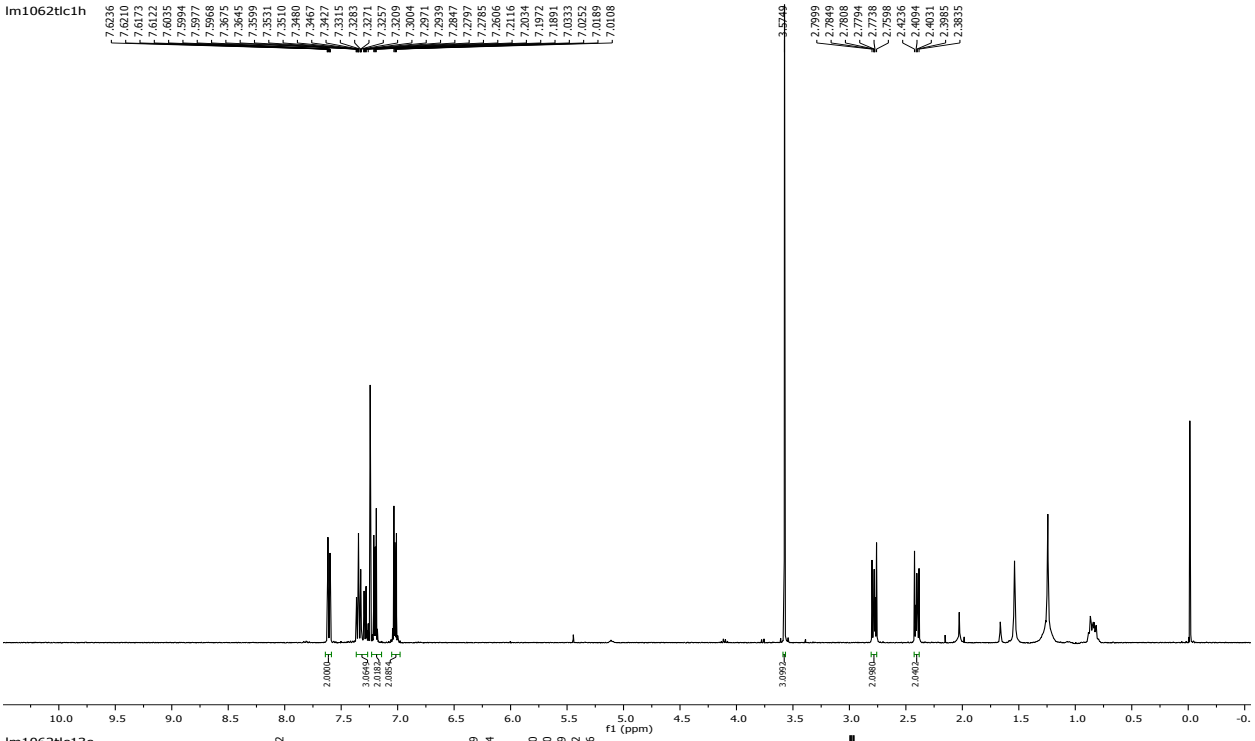
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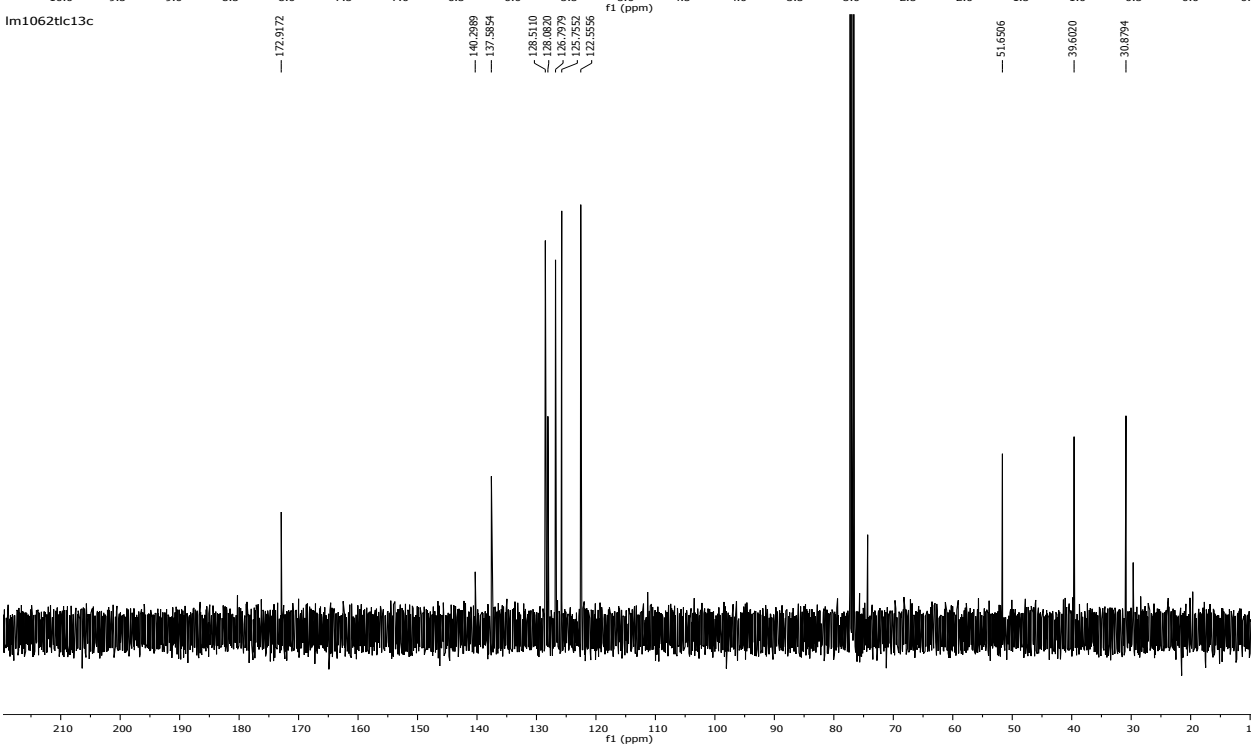
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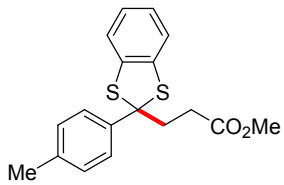

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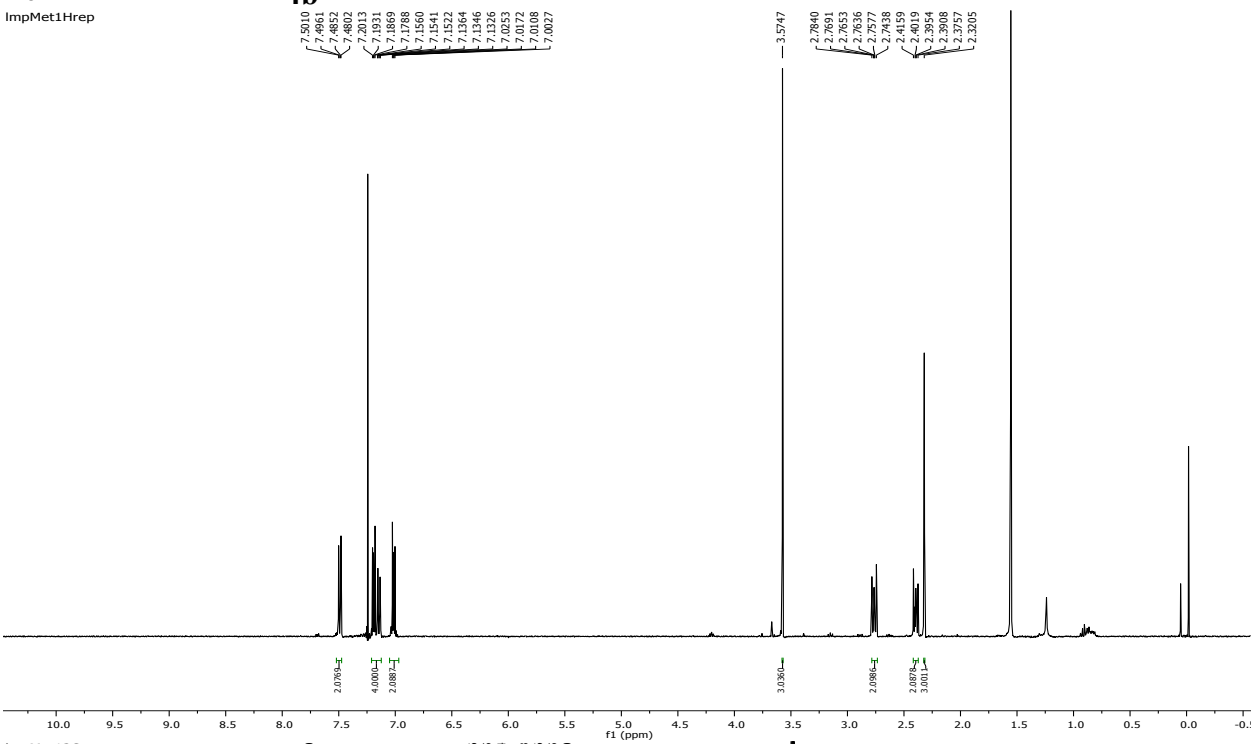

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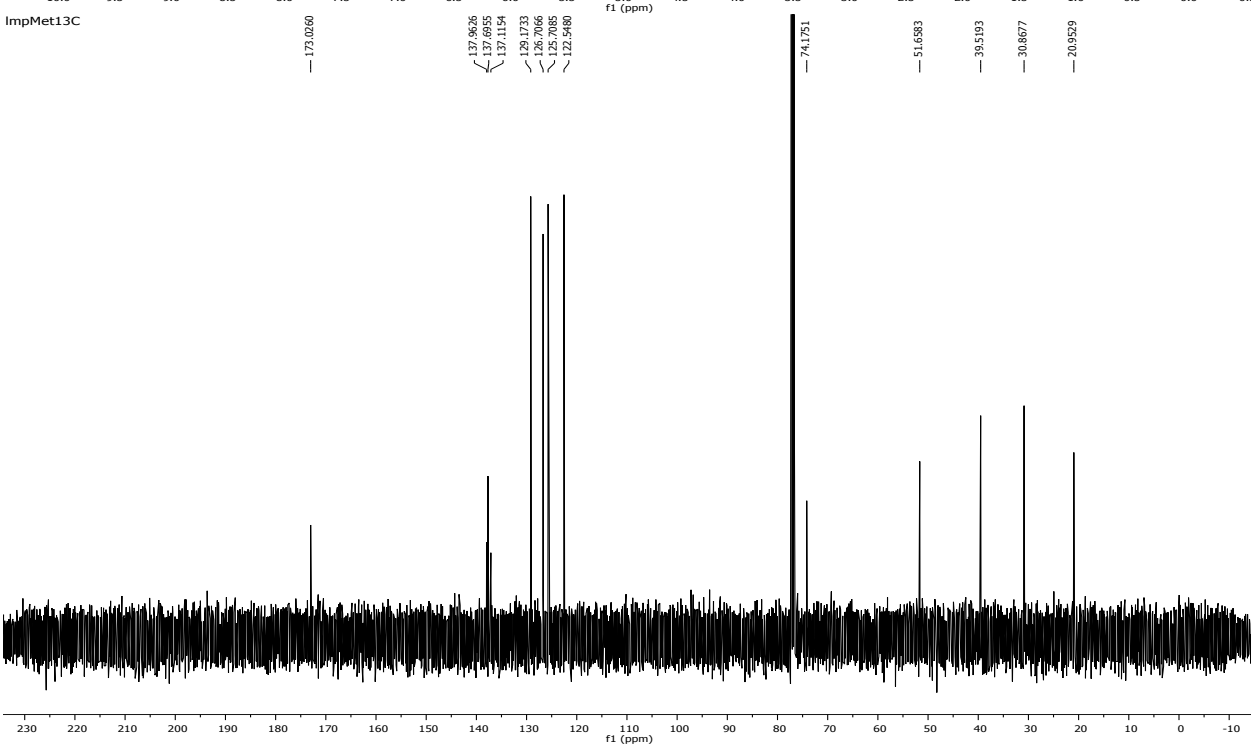

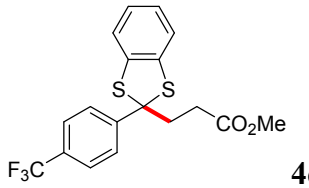
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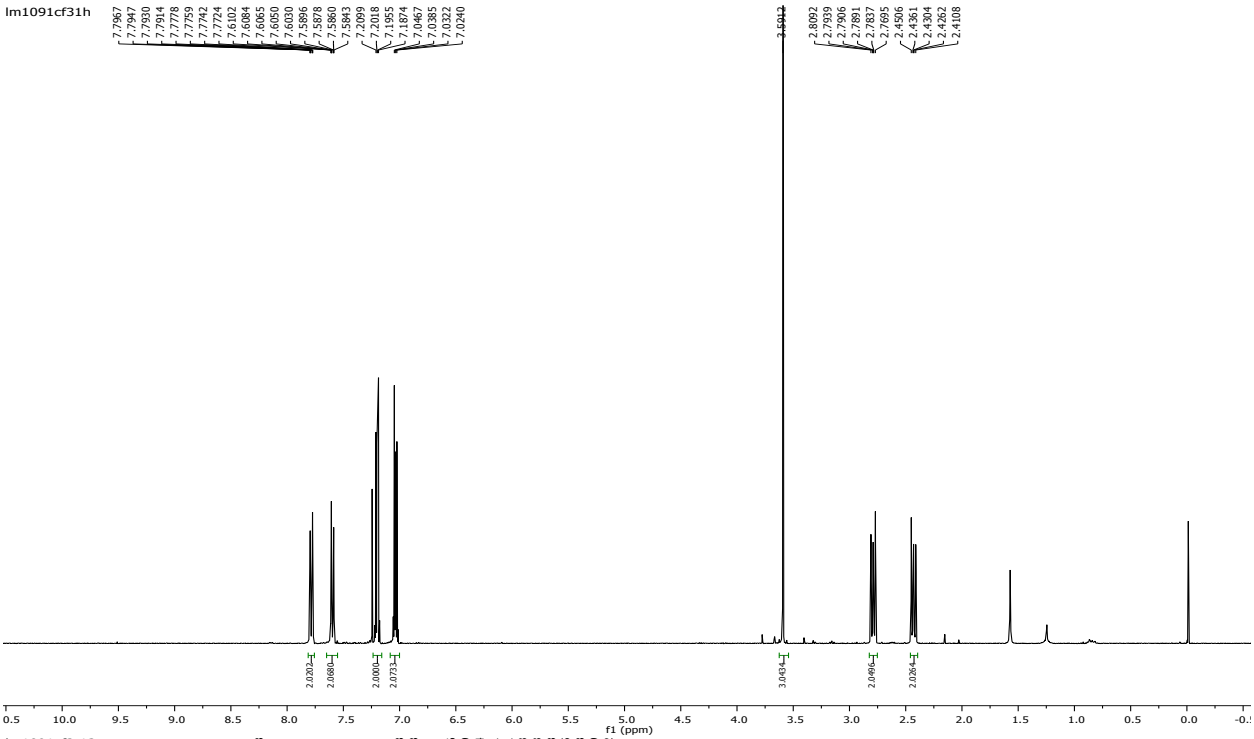
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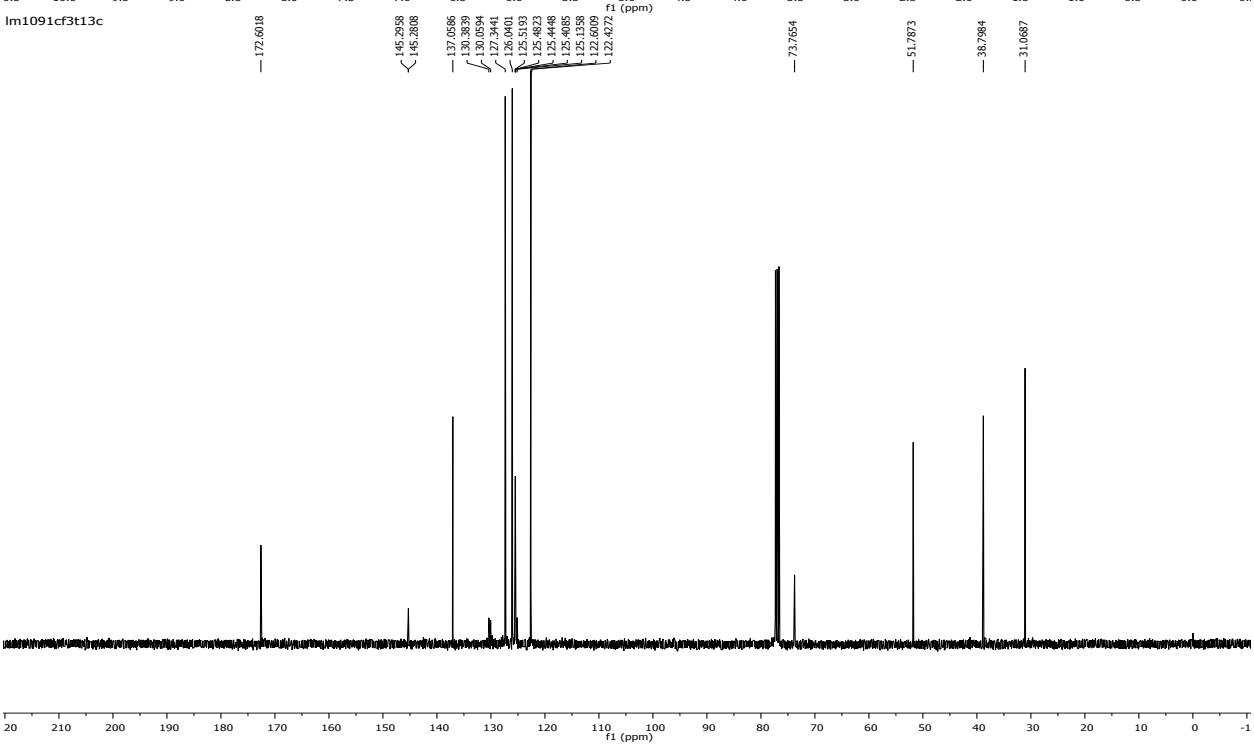
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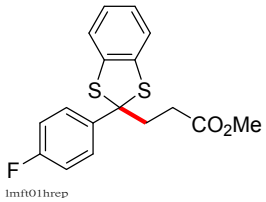
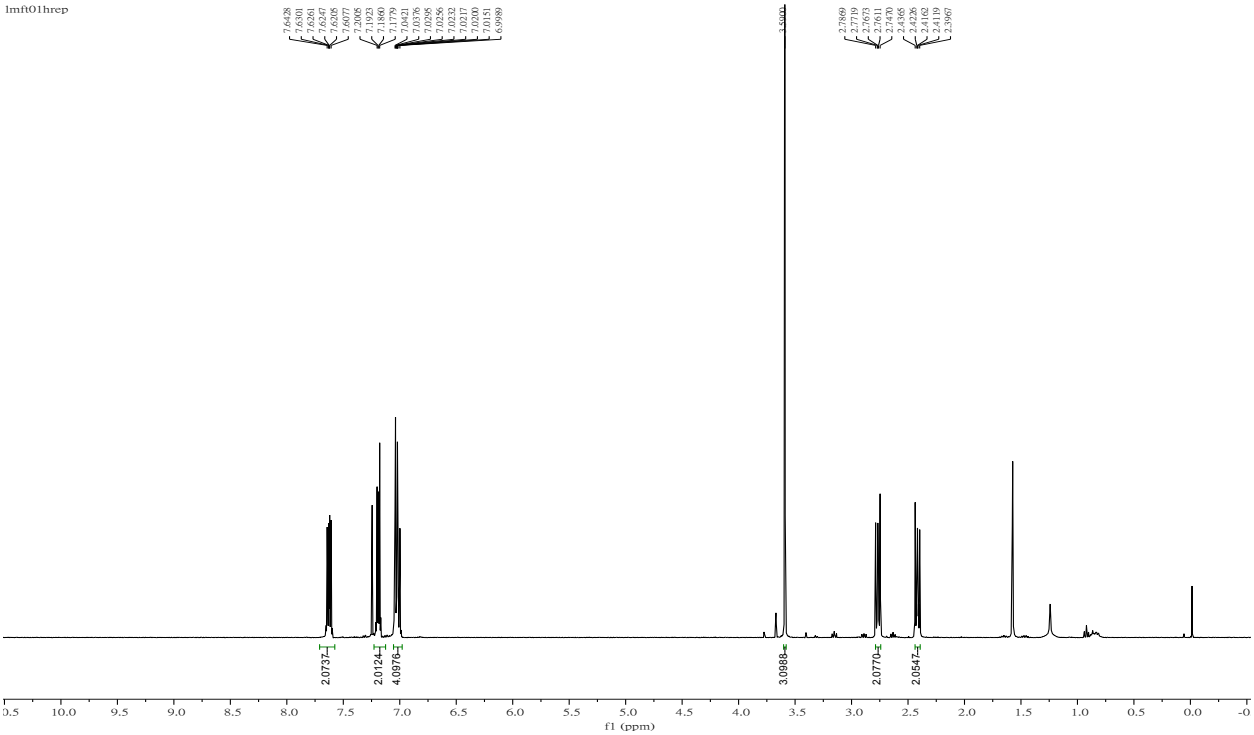
125.5193
125.4823
125.4448
125.4085
125.1358
122.6009
122.4272
73.7654
51.7873
38.7984
31.0687
f1 (ppm)

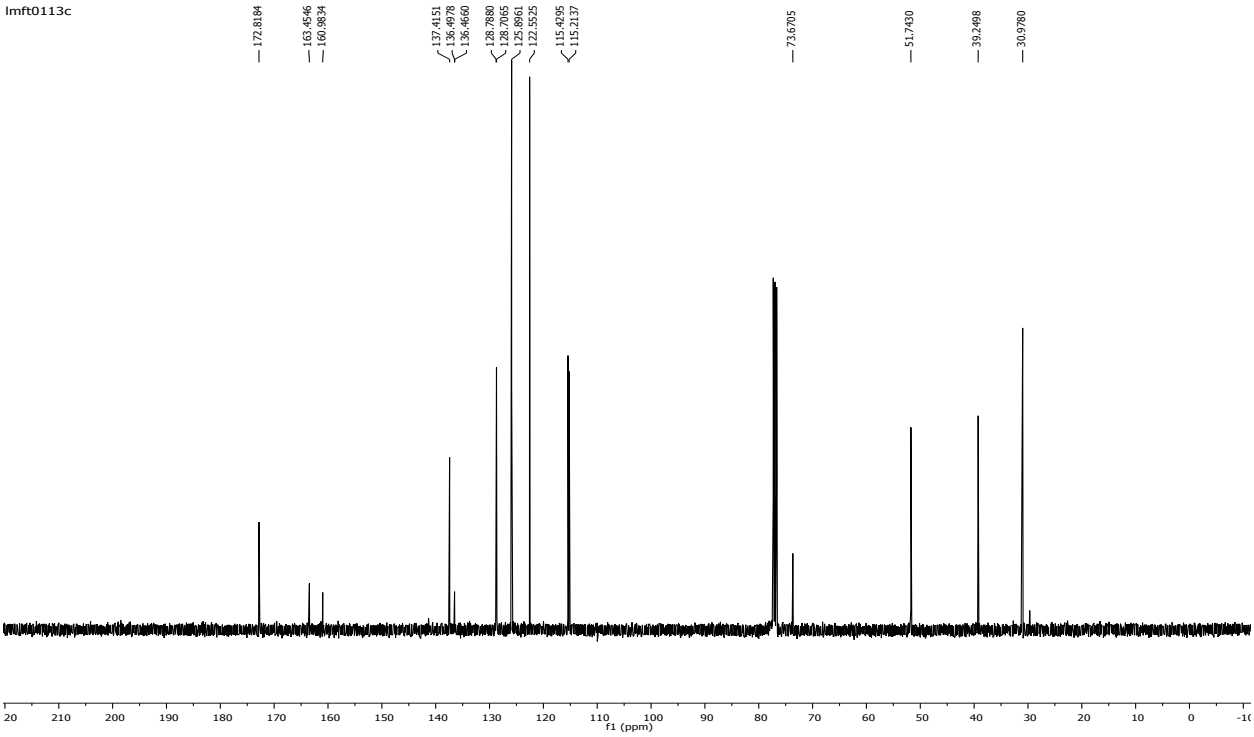


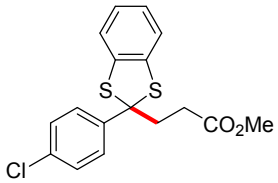
lmft01hrep

4d



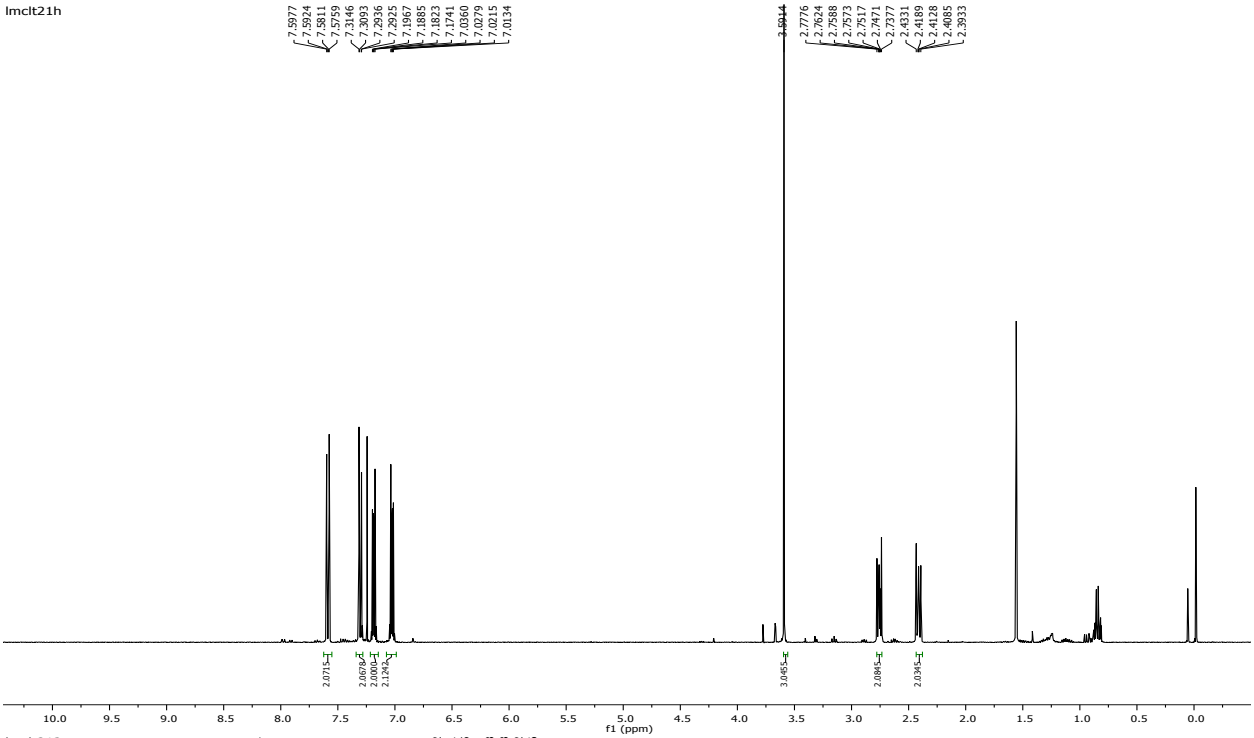
lmft0113c



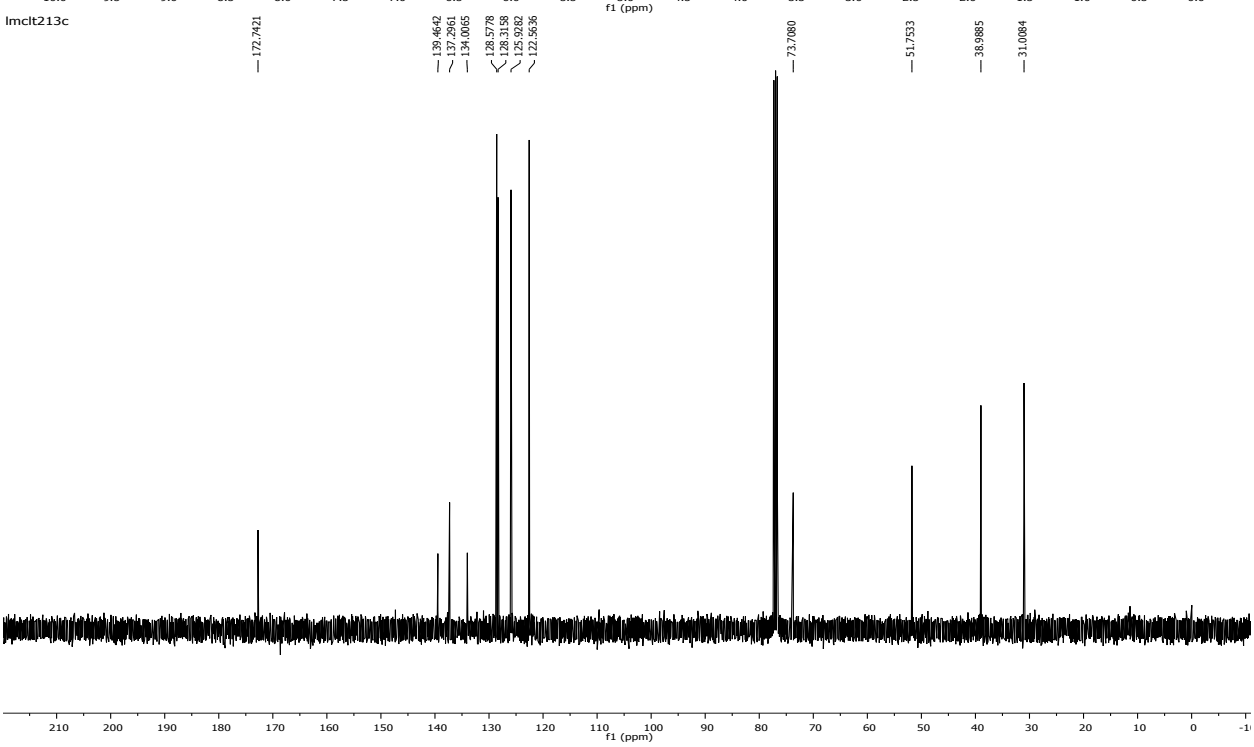


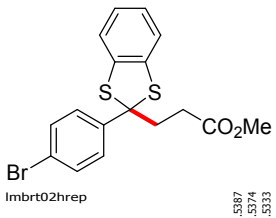
lmclt21h

4e



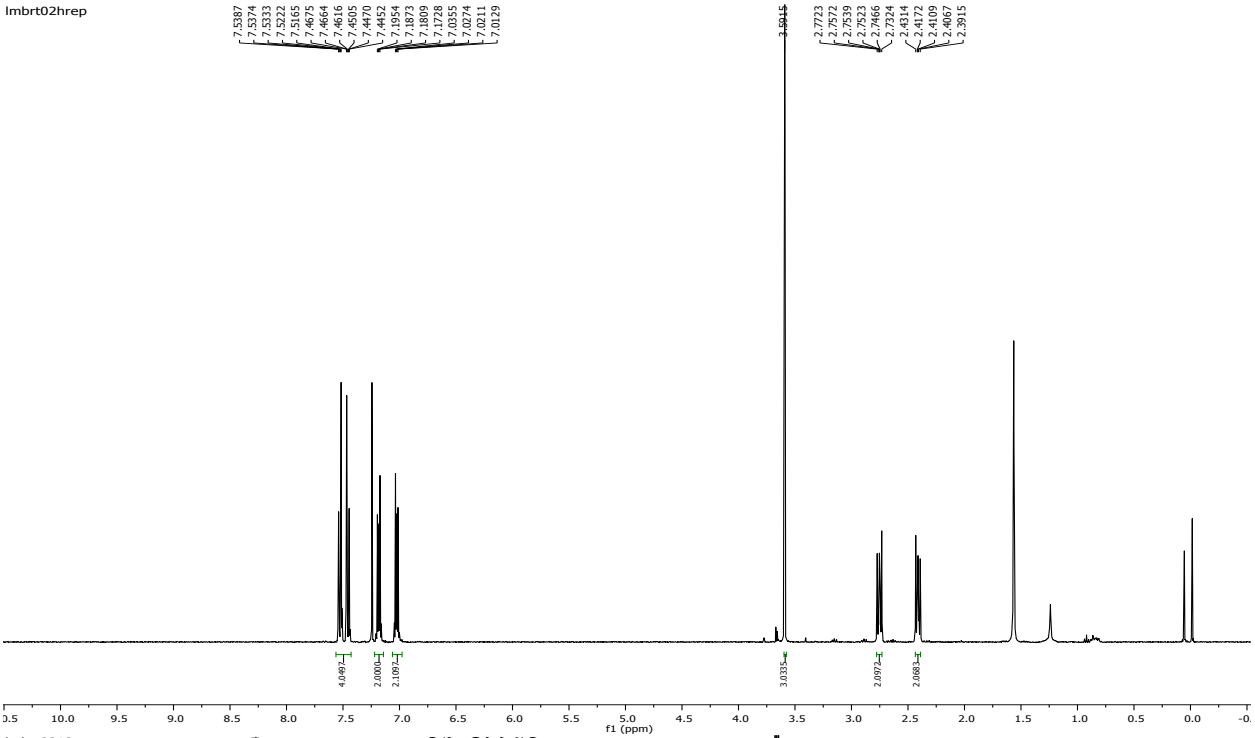
lmclt213c



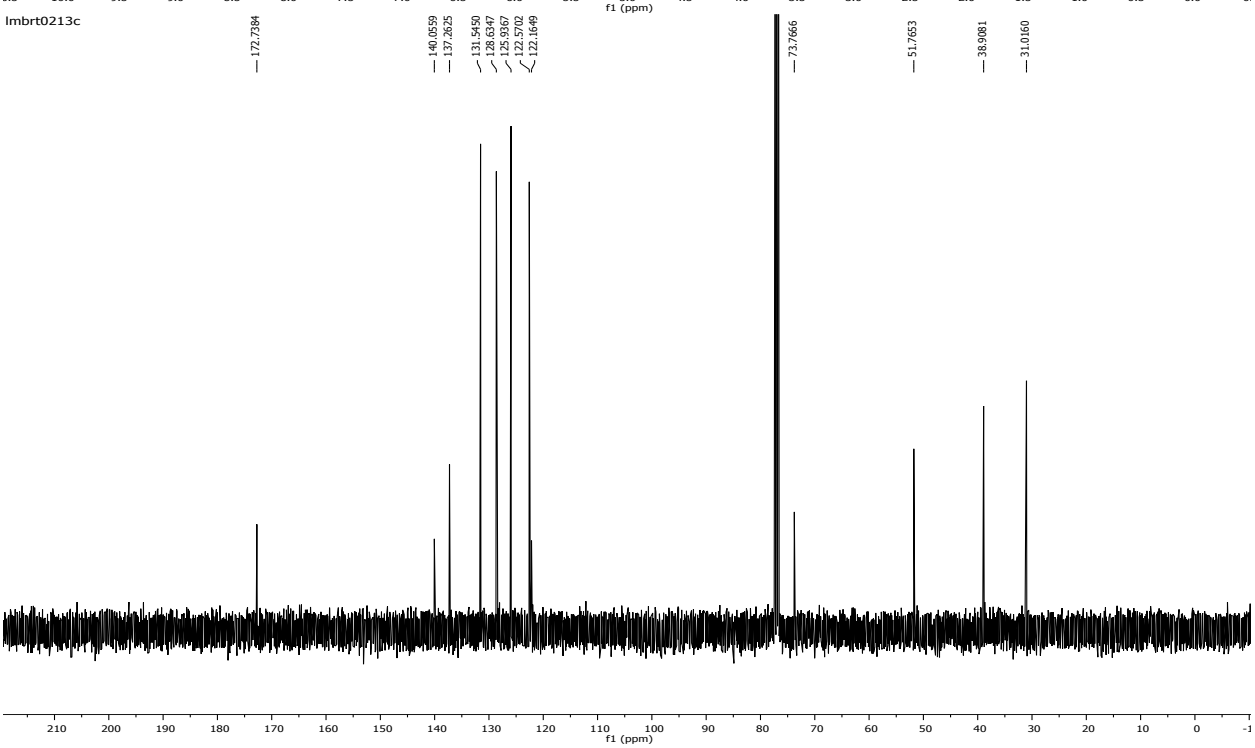


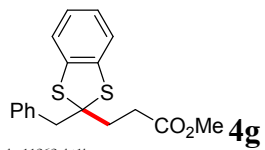
4f

lmbrt02hrep



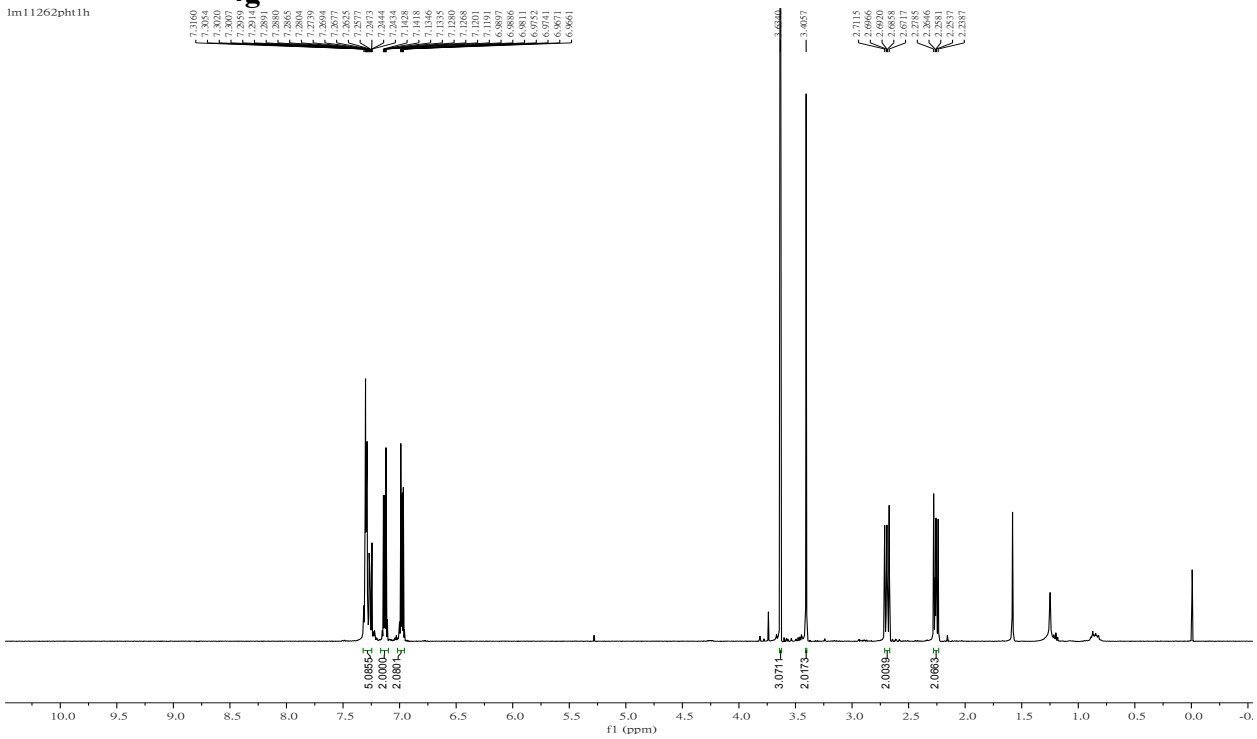
lmbrt0213c



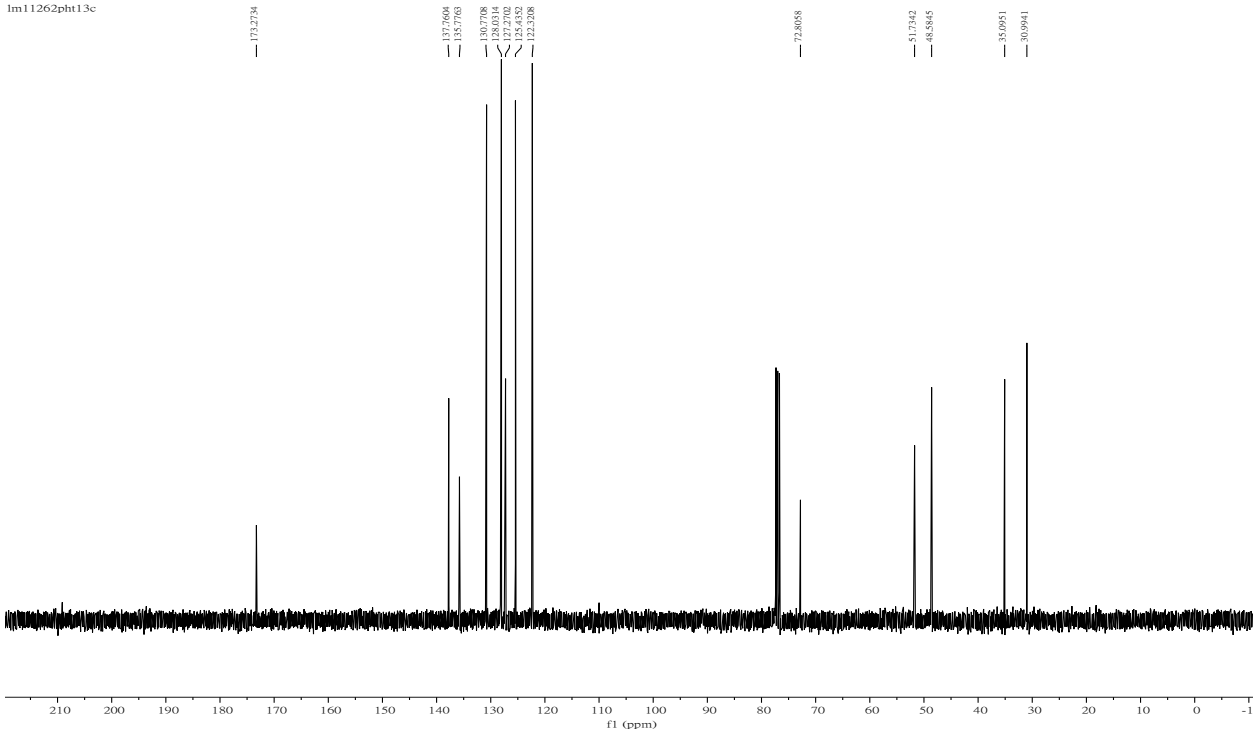


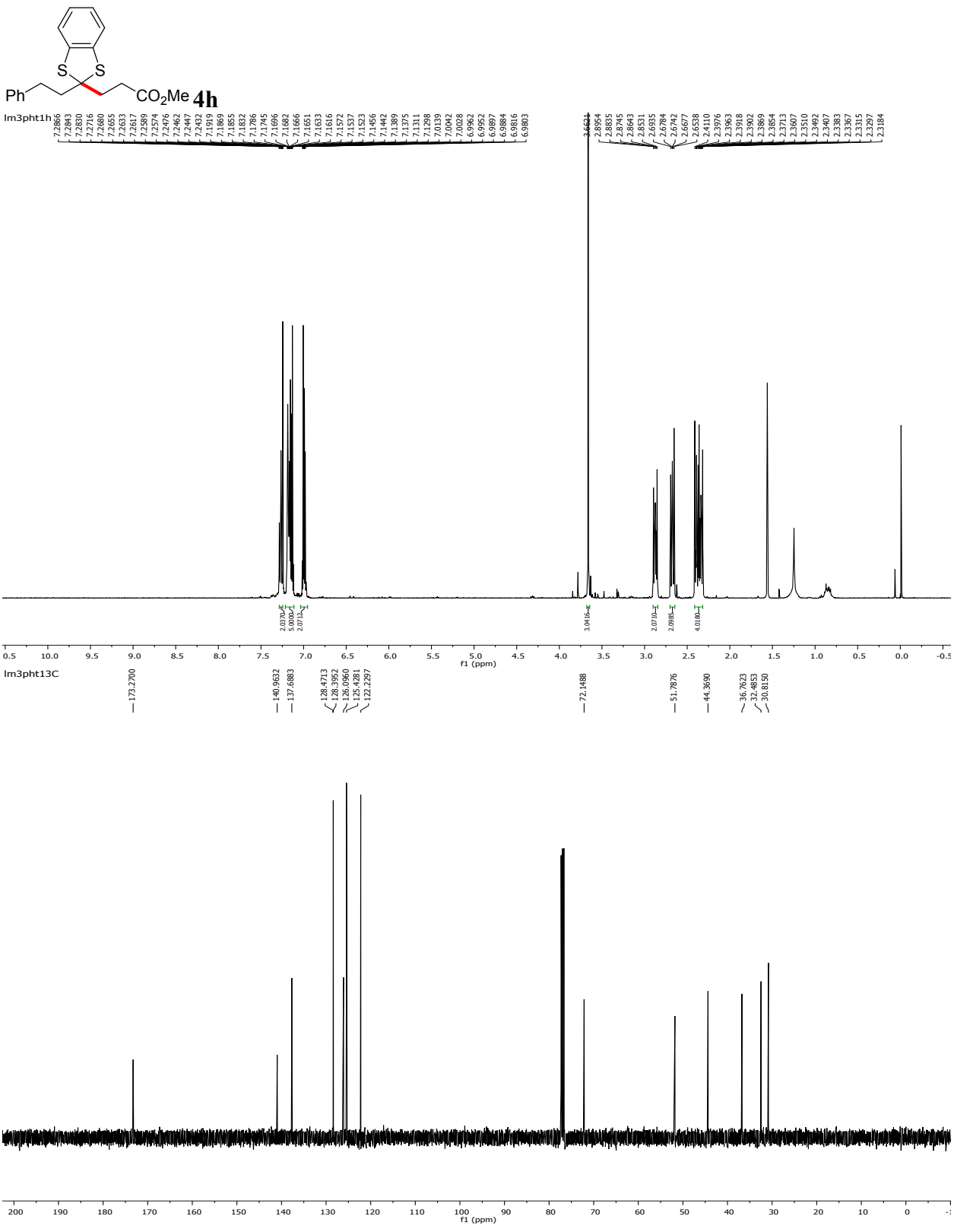
4g

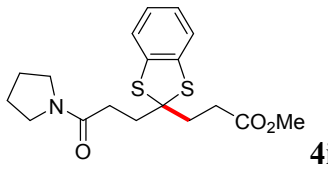
lm11262pht1h



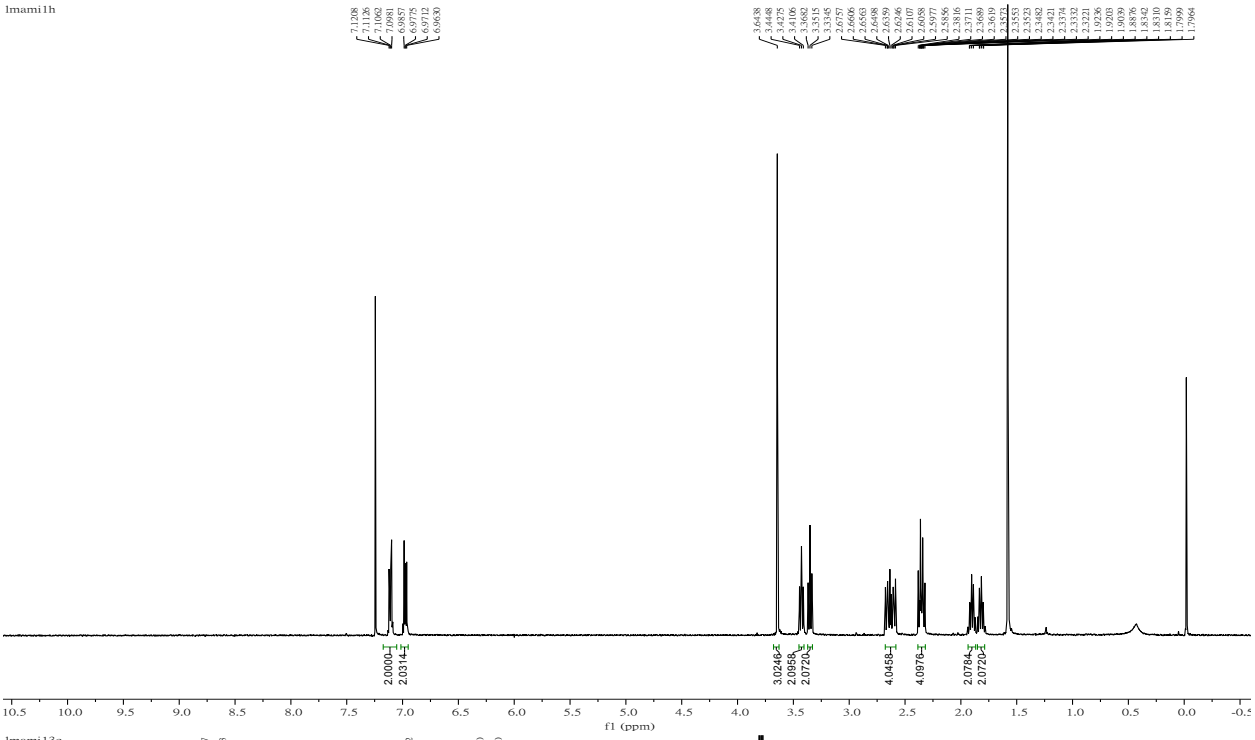
lm11262pht13c

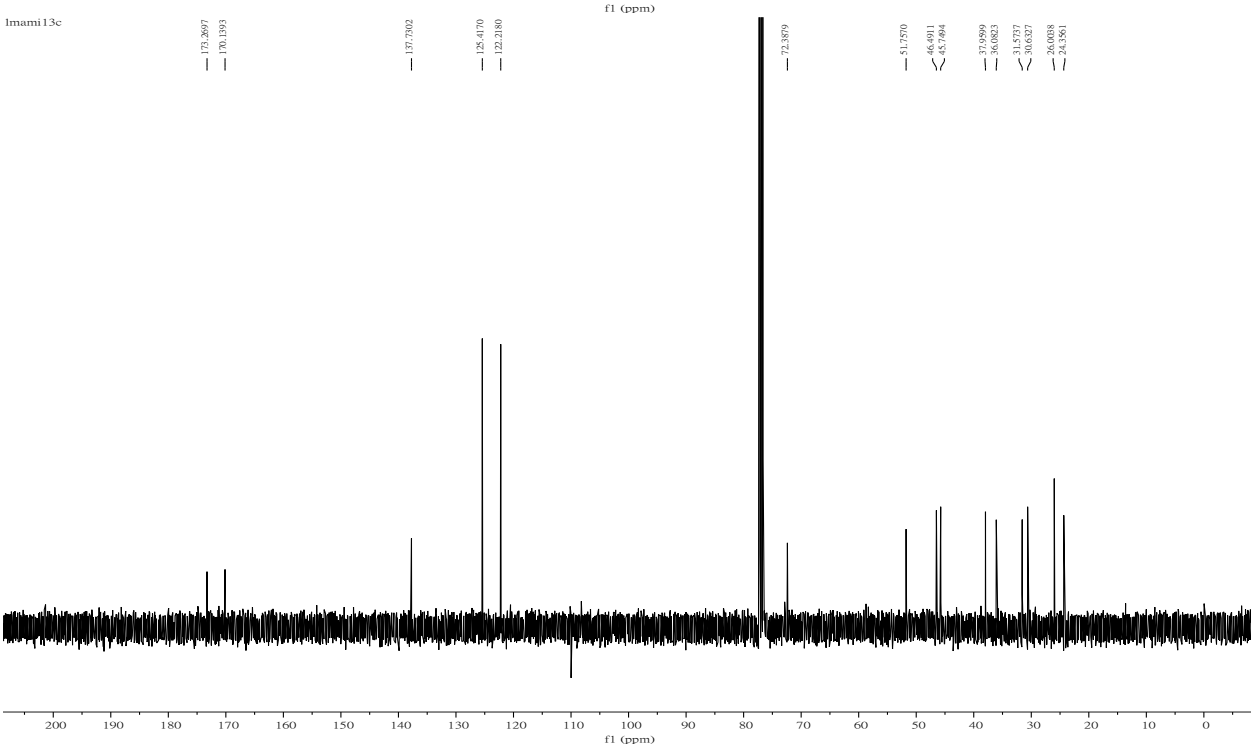


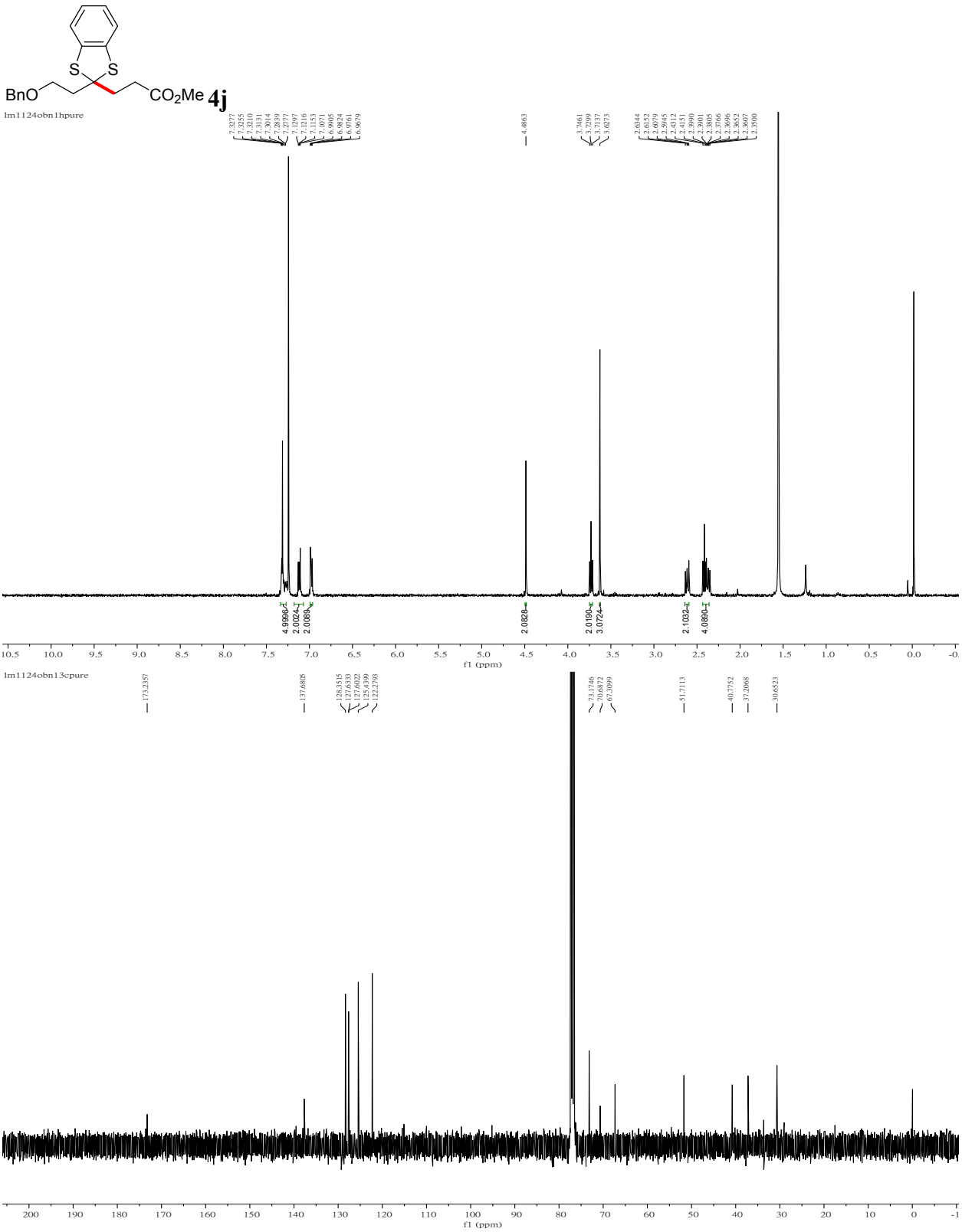

Ph
CO2Me
4h
lm3pht1h
lm3pht13C
f1 (ppm)

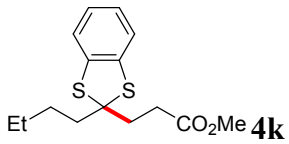


4i



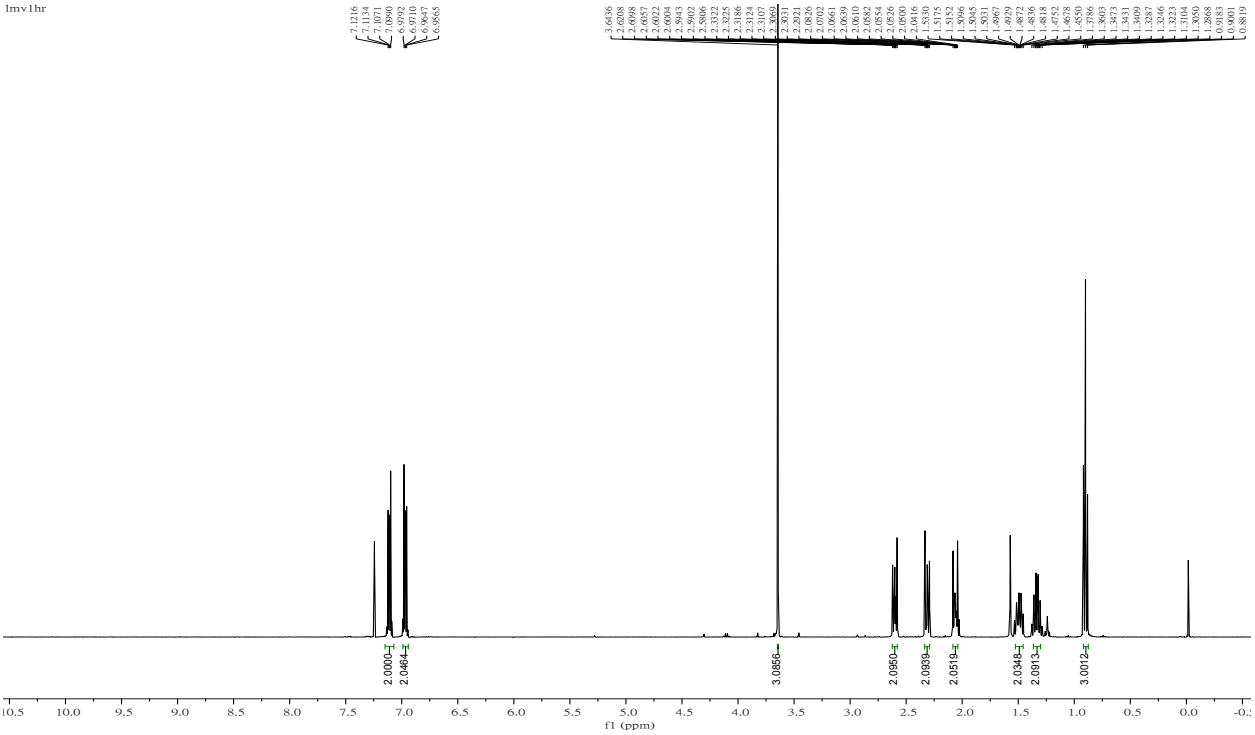



BnO
CO2Me
4j
lm1124obn1hpure
lm1124obn13cpure
f1 (ppm)
f1 (ppm)

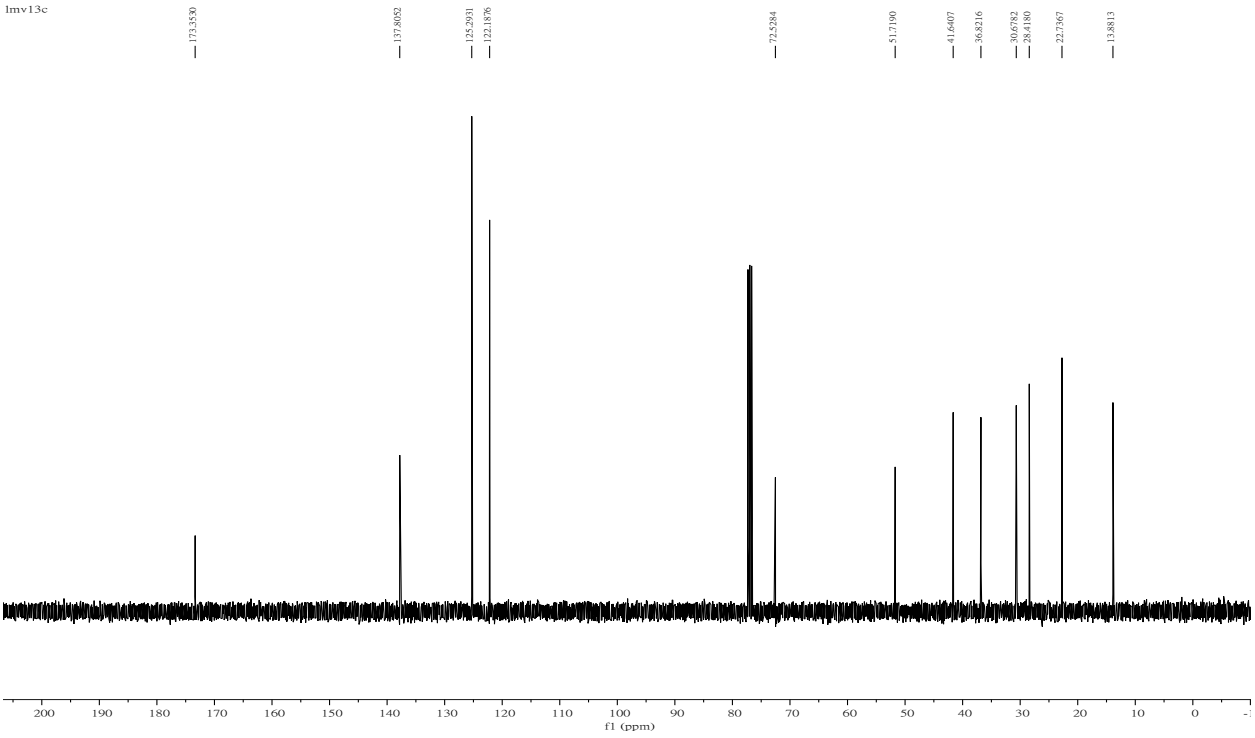


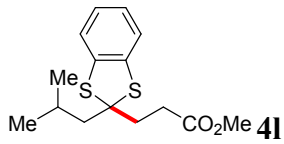
4k

lmv1hr



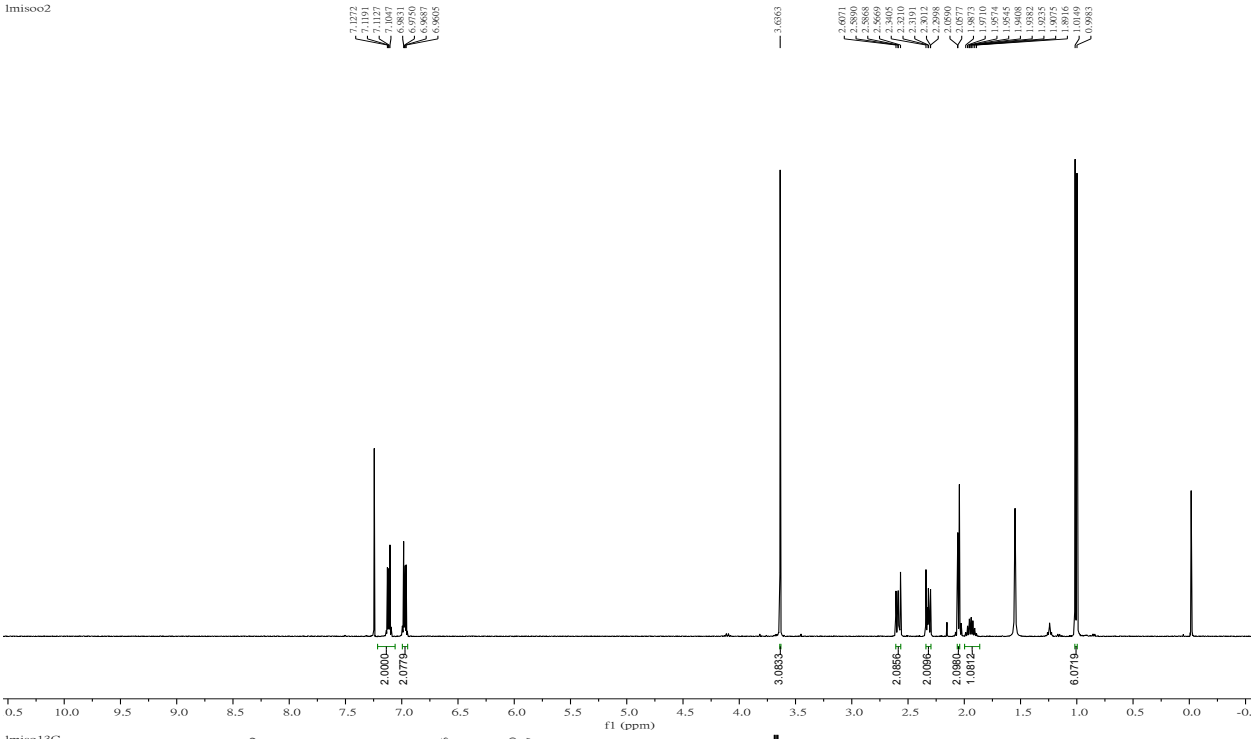
lmv13c



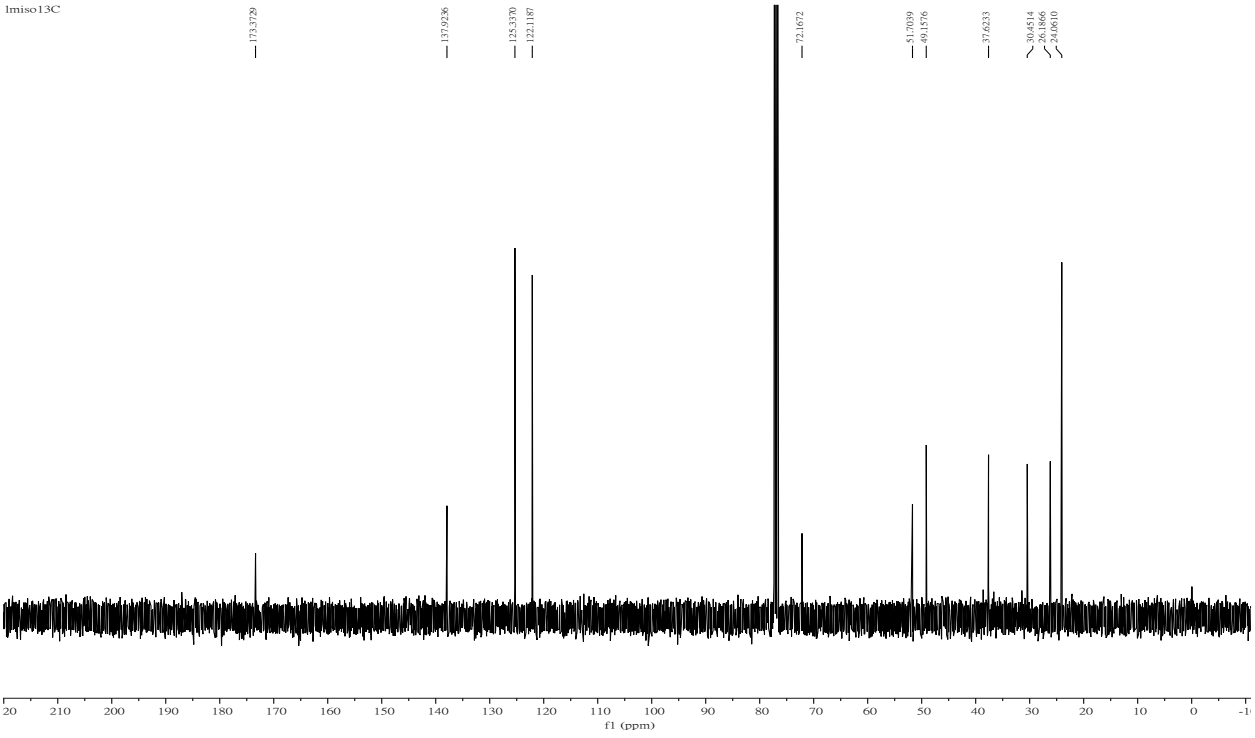


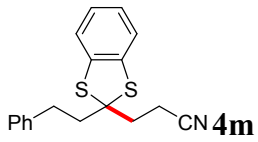
4l

lmisoo2



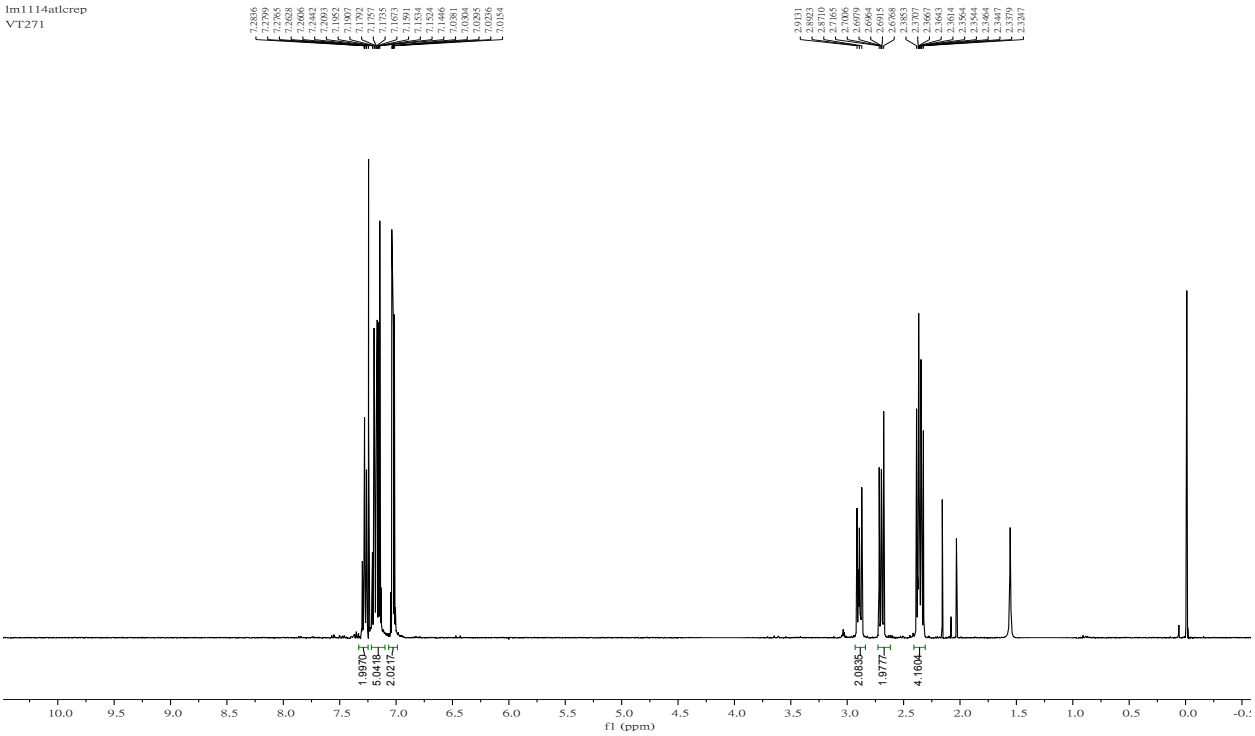
lmiso13C



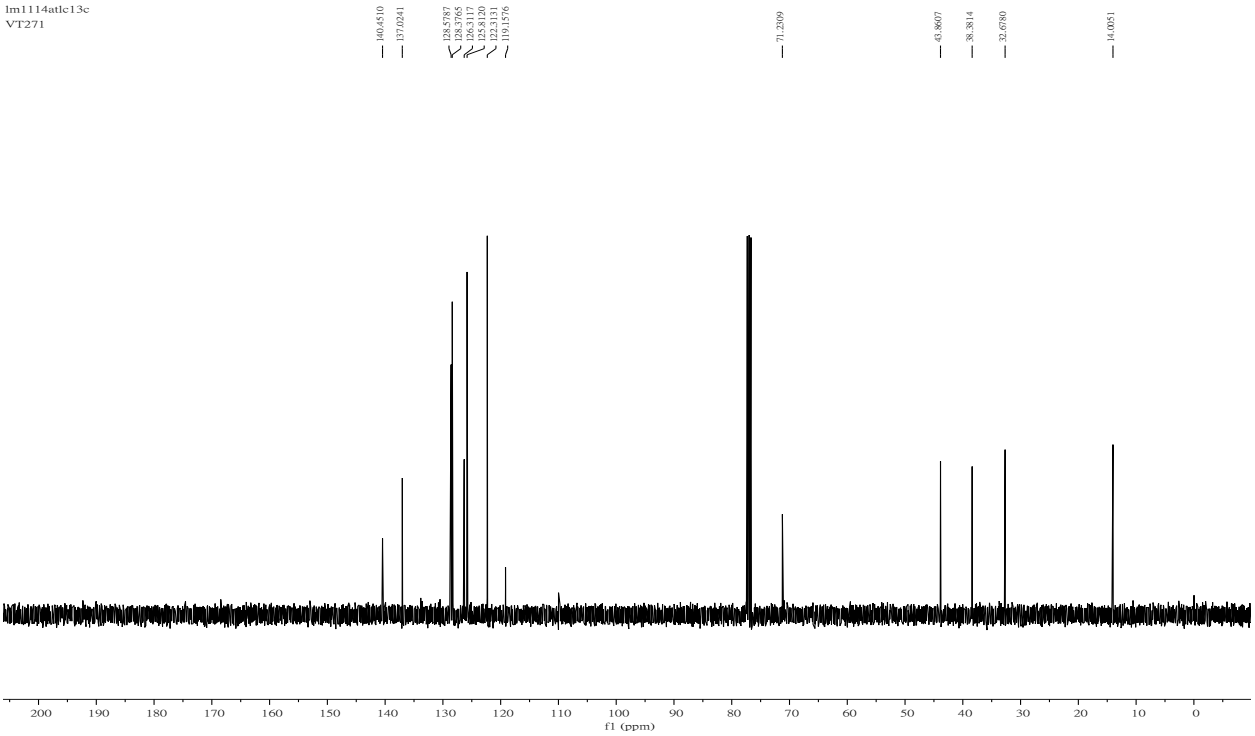


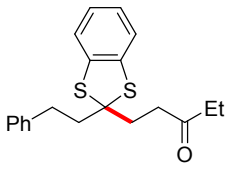
4m

lm1114atlcrep
VT271



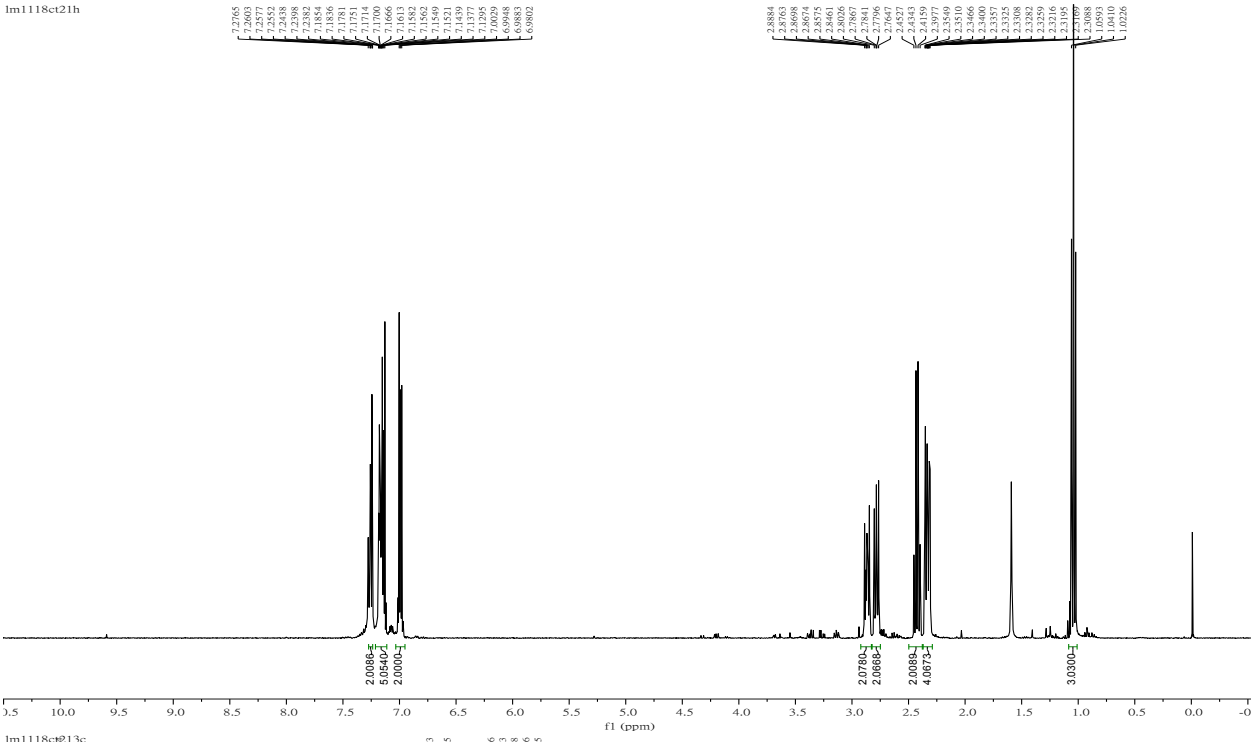
lm1114atlc13c
VT271



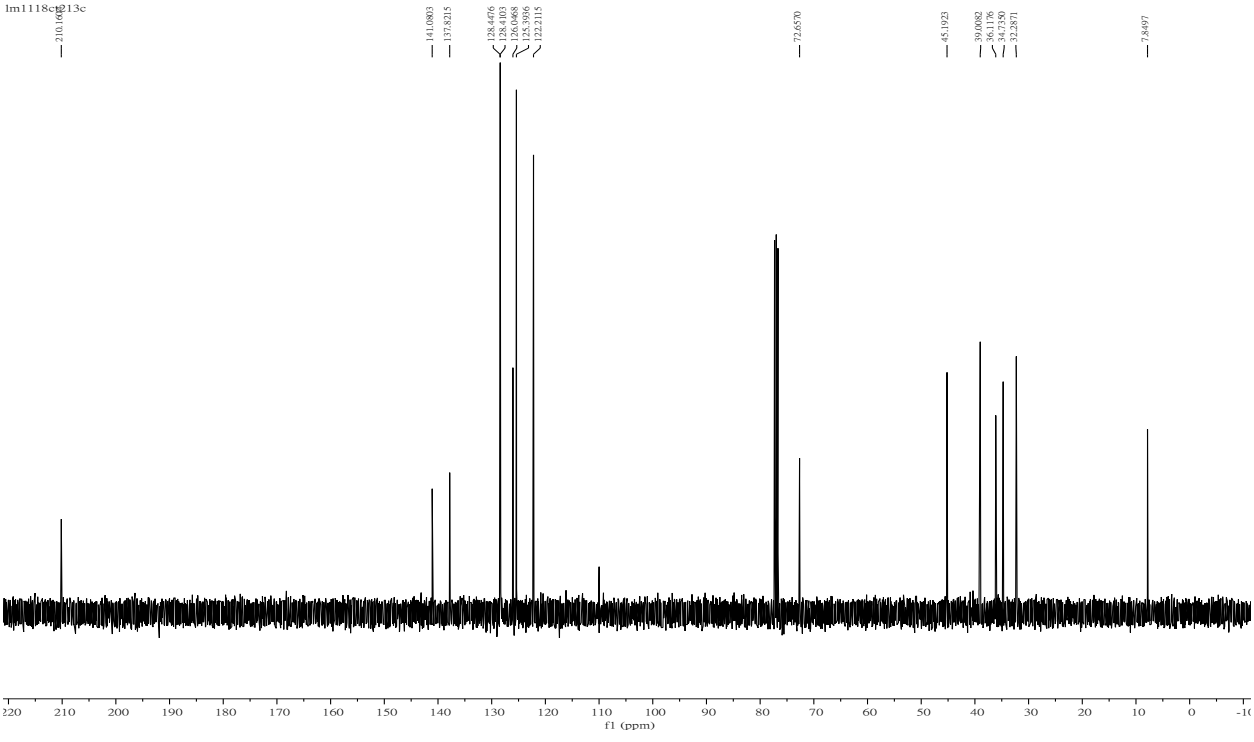


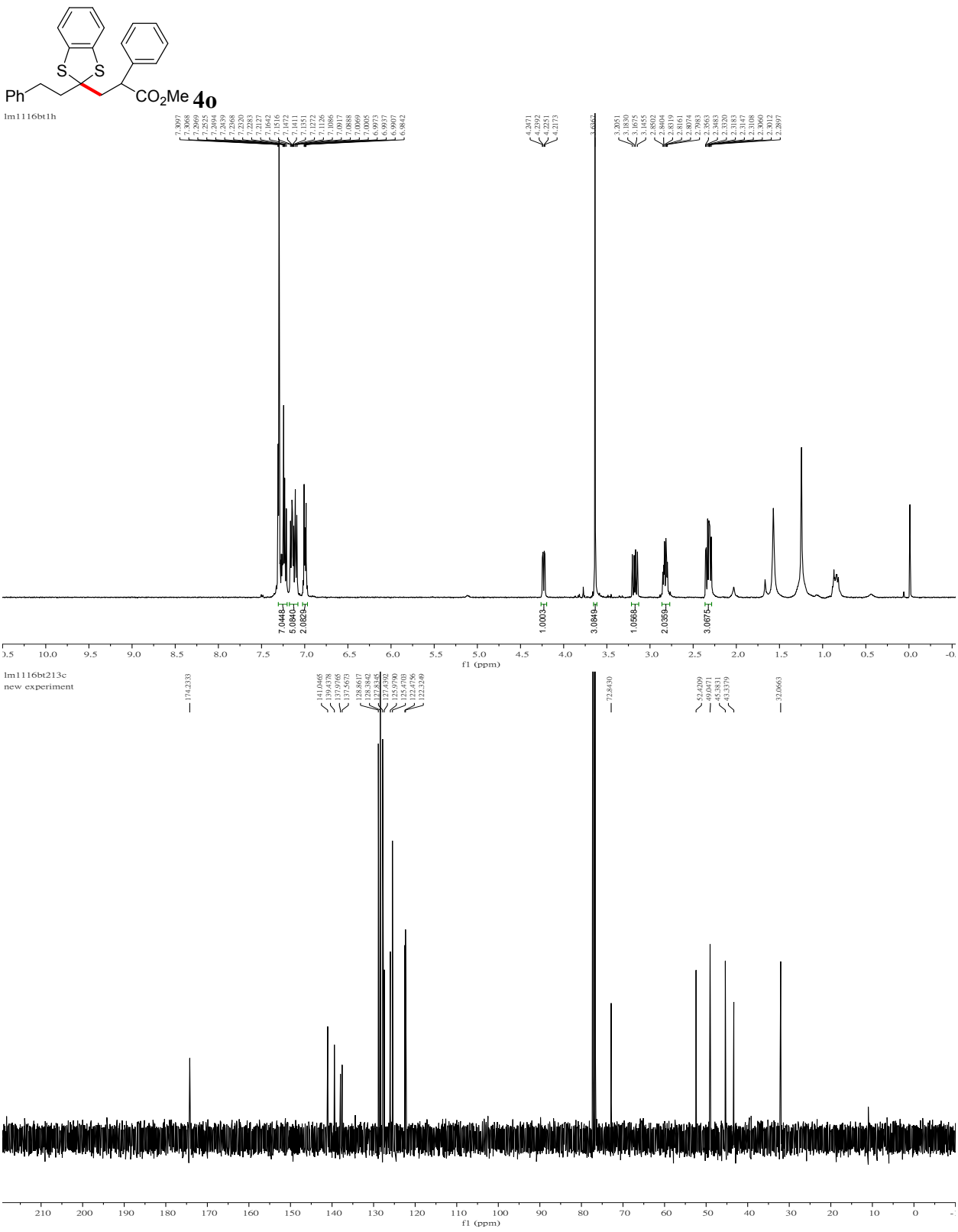
4n

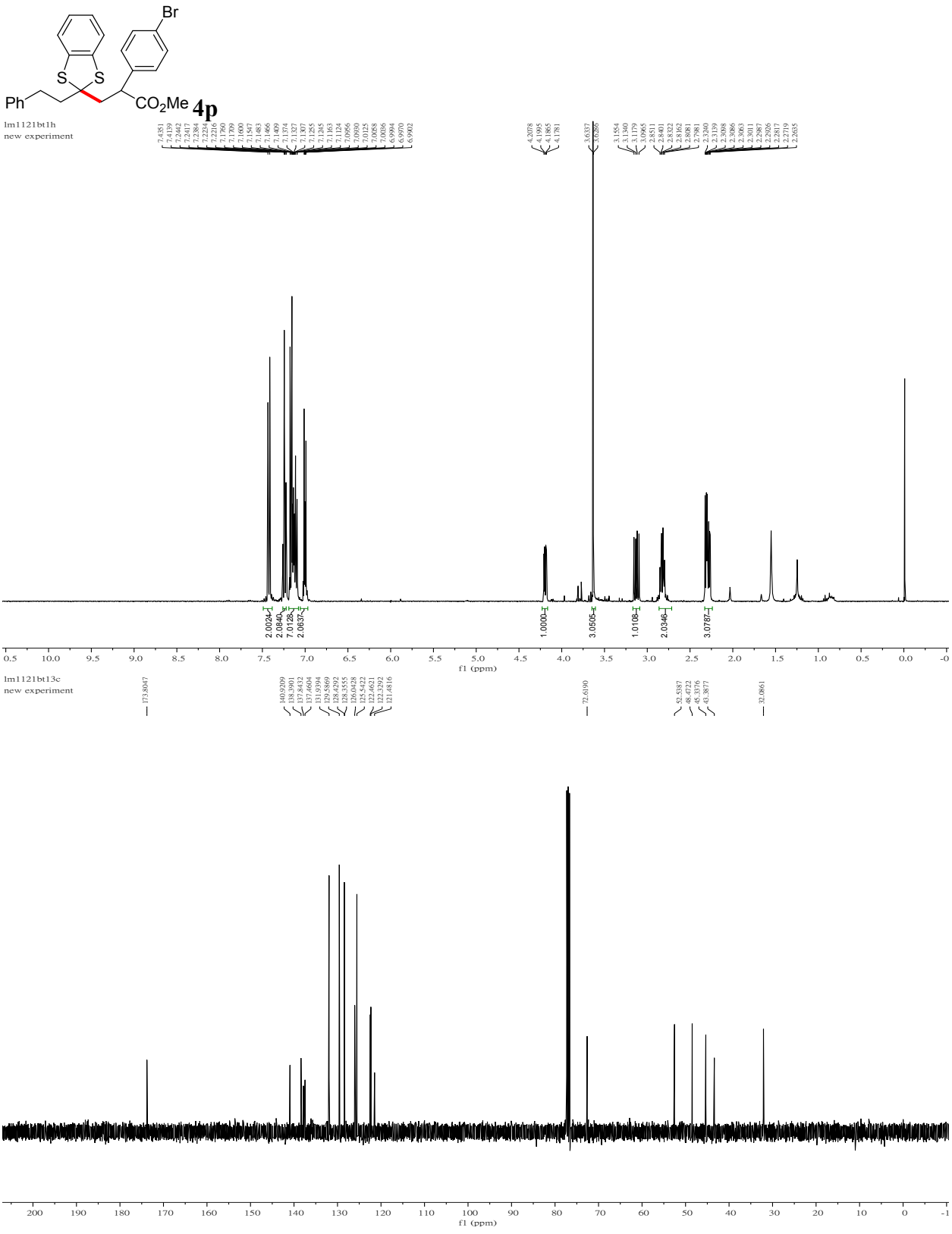
lm1118ct21h

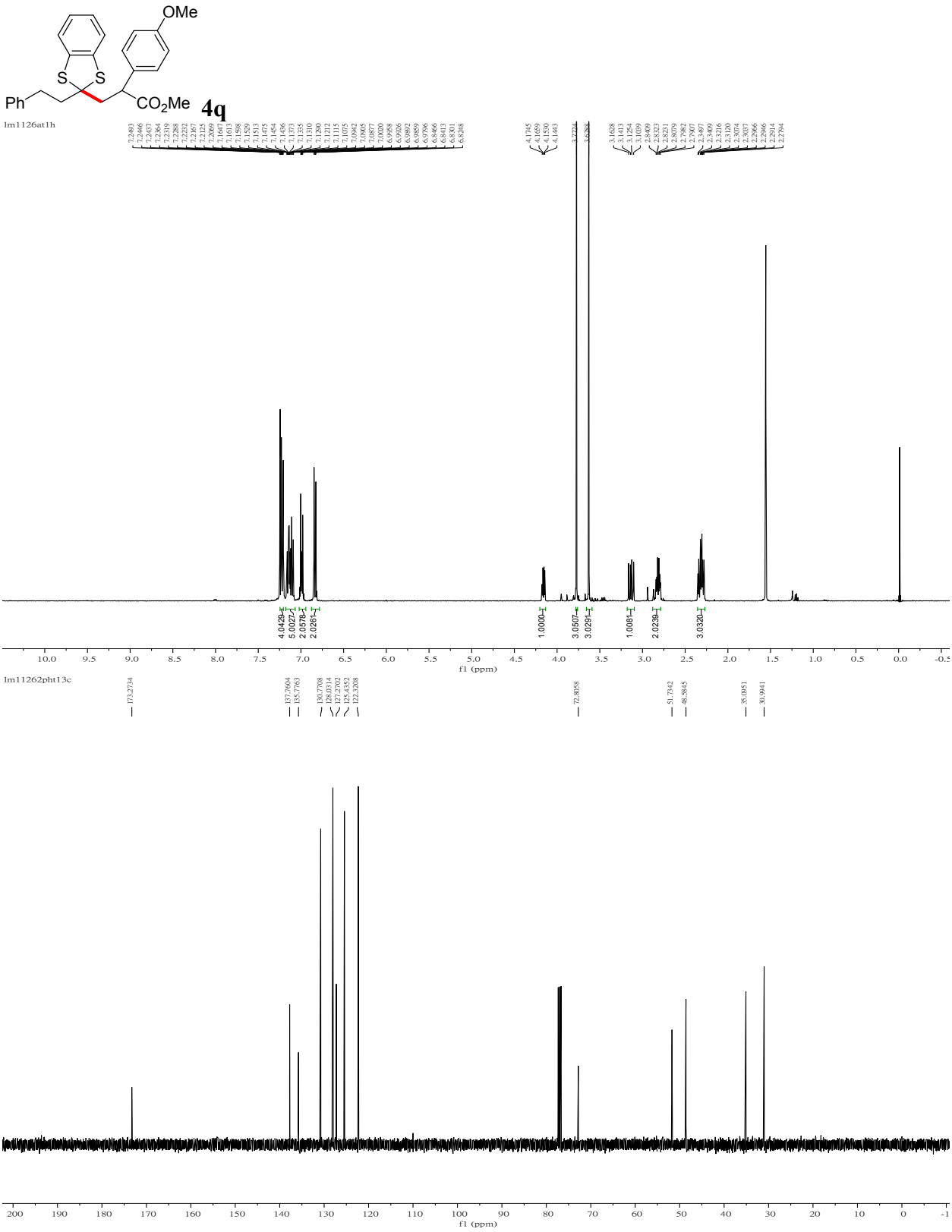


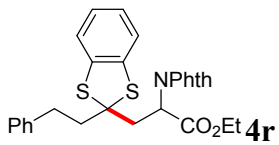
lm1118ct213c



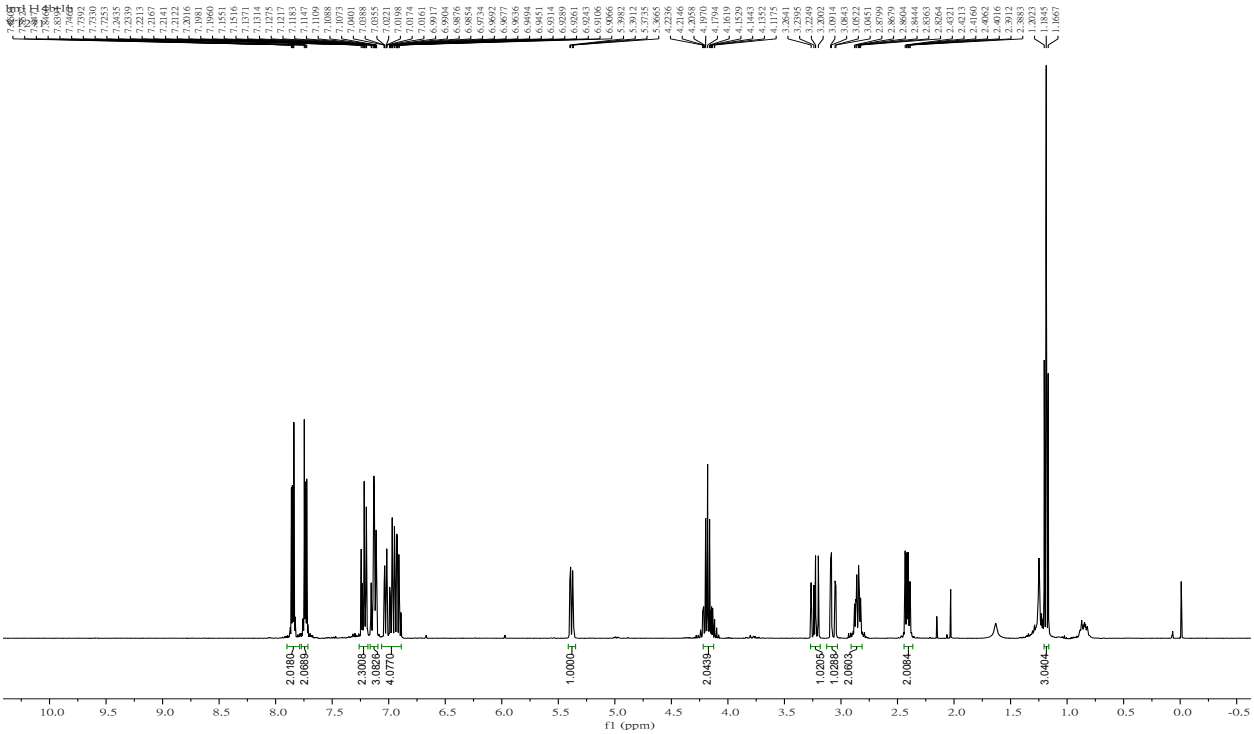

Ph
CO2Me
4o
lm1116bt1h
7.3097
7.2969
7.2969
7.2525
7.2494
7.2439
7.2368
7.2320
7.2265
7.2127
7.1642
7.1516
7.1472
7.1411
7.1351
7.1272
7.1126
7.1086
7.0917
7.0888
7.0769
7.0005
6.9973
6.9937
6.9907
6.8842
4.3471
4.2292
4.2251
4.2173
3.6362
3.3051
3.1830
3.1675
3.1455
2.8502
2.8404
2.8319
2.8191
2.8054
2.7983
2.3563
2.3483
2.3320
2.3193
2.3147
2.3108
2.3060
2.3012
2.2897
7.0448
5.0840
2.0829
1.0003
3.0849
1.0568
2.0359
3.0675
f1 (ppm)
lm1116bt213c
new experiment
174.2333
141.0468
139.4378
137.9746
129.2573
128.8617
128.3842
127.8345
127.4392
125.9790
125.4708
122.4196
122.2249
72.8430
52.4209
49.0471
45.3831
43.3779
32.0663
f1 (ppm)

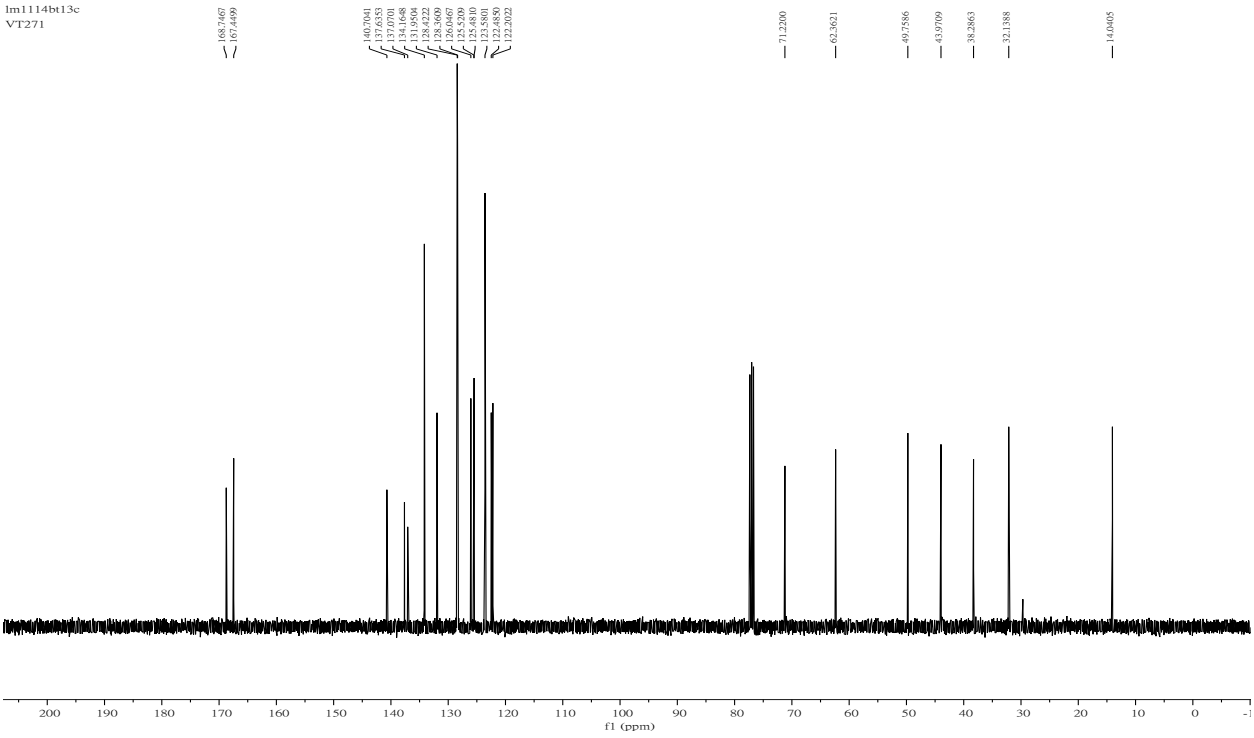

Br
S
S
Ph
CO2Me
4p
lm1121bt1h
new experiment
lm1121bt13c
new experiment
f1 (ppm)

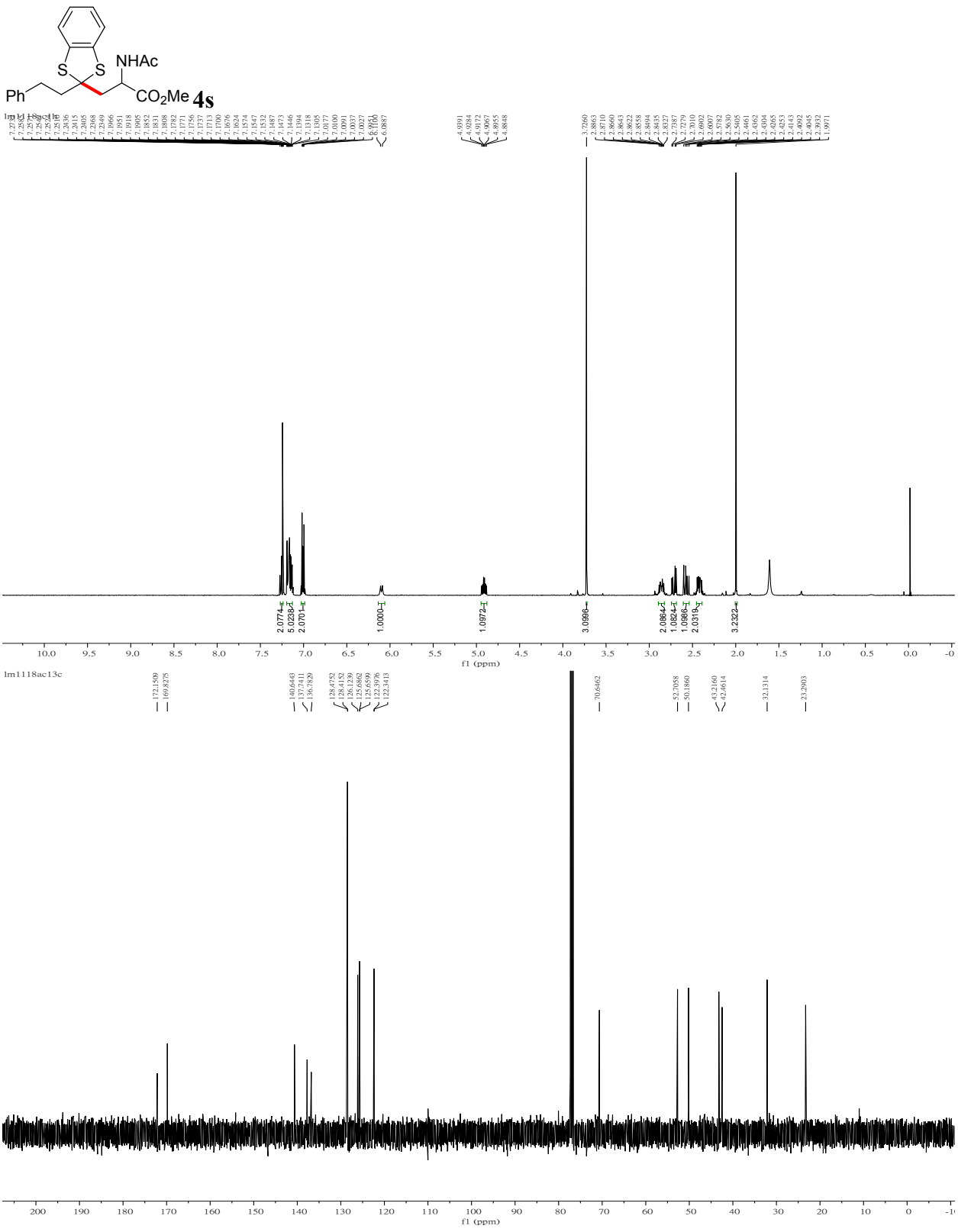

OMe
S
S
Ph
CO2Me
4q
lm1126at1h
4.0429
5.0027
2.0579
2.0261
1.0000
3.0507
3.0291
1.0081
2.0238
3.0320
f1 (ppm)
lm11262pht13c
173.2734
137.7604
135.7763
130.7708
128.0314
127.2702
125.4552
122.3208
72.8058
51.7342
48.5845
35.0951
30.9941
f1 (ppm)

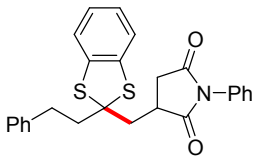

NPhth
S
S
Ph
CO2Et

4r

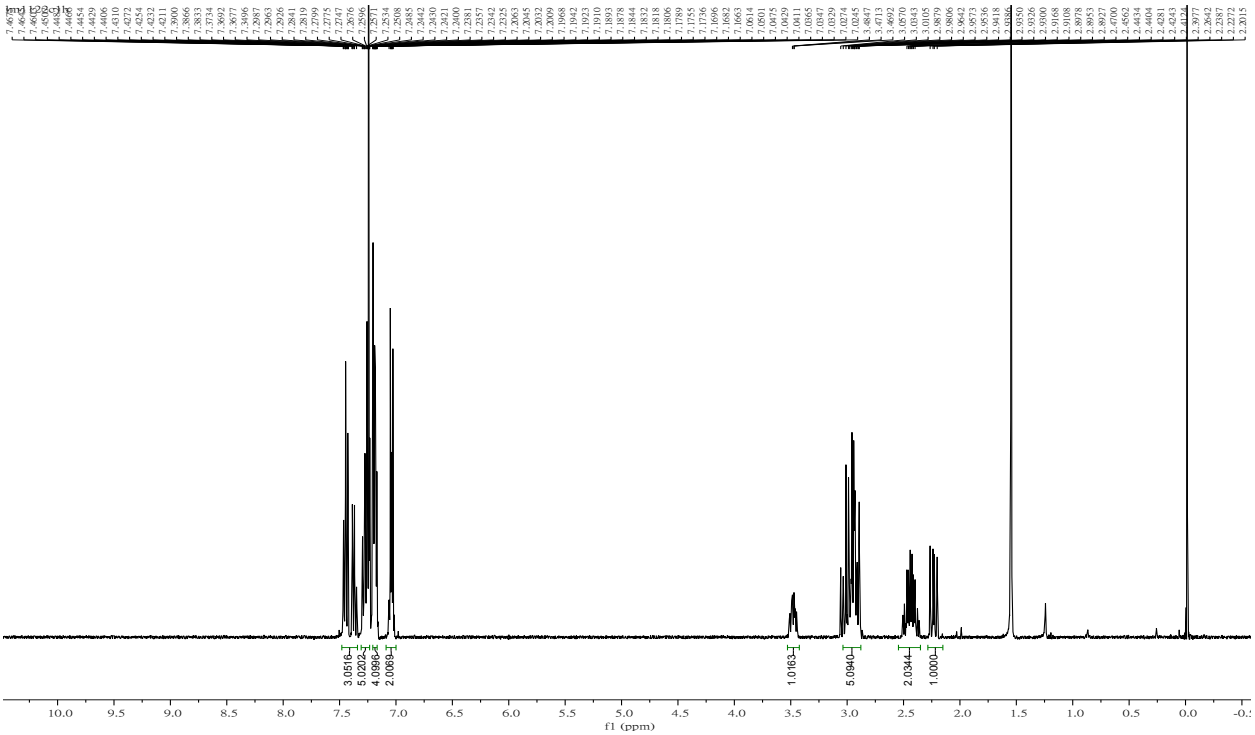

2.0180
2.0689
2.3008
3.0826
4.0770
1.0000
2.0439
1.0295
1.0286
2.0603
2.0084
3.0404
f1 (ppm)

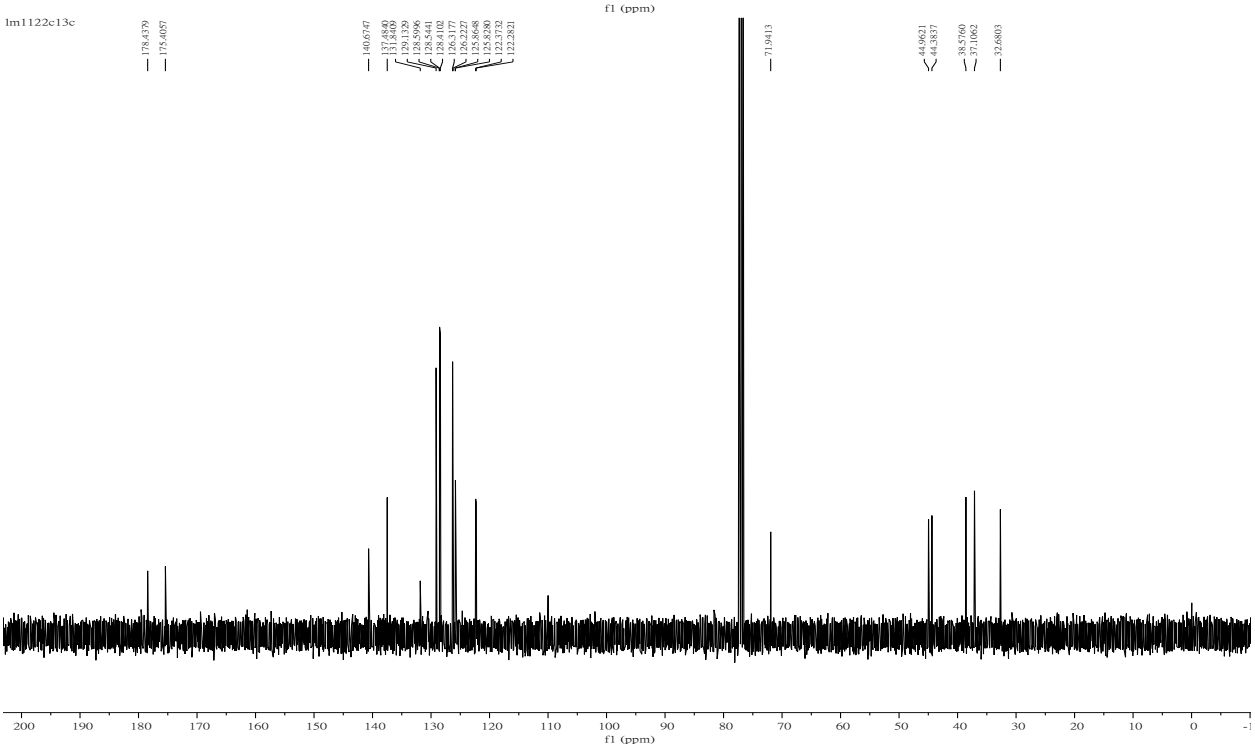

lm1114bt13c
VT271
168.7467
167.4499
140.7041
137.6353
137.0701
134.1648
131.9596
131.2523
128.3693
126.0467
125.5209
125.4810
123.5801
123.2485
122.2022
71.2200
62.3621
49.7586
43.9709
38.2863
32.1388
14.0405
f1 (ppm)

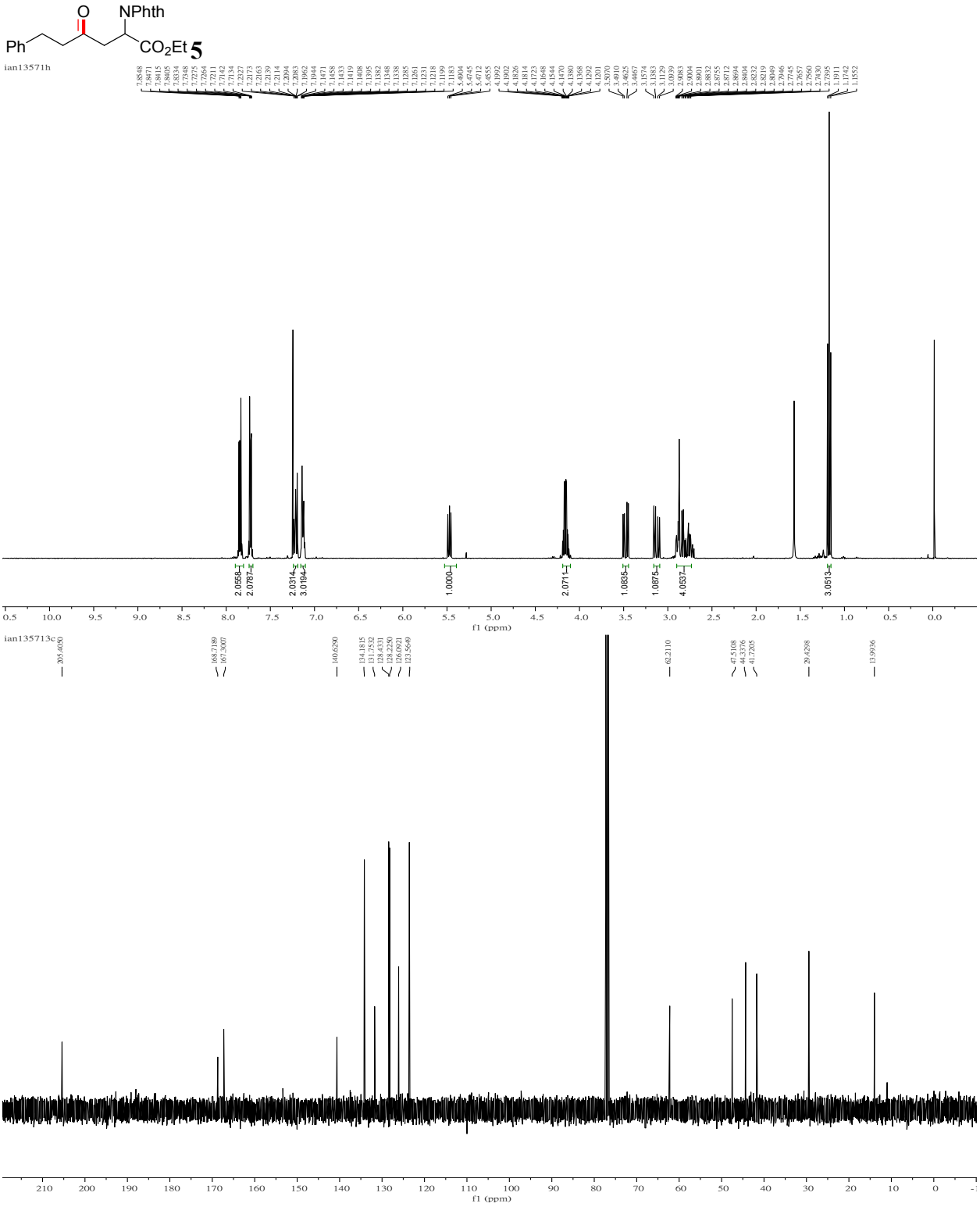

NHAc
S
S
Ph
CO2Me
4s
lm1118ac1h
lm1118ac13c
f1 (ppm)


O
S
S
N–Ph
Ph
O

4t


3.0516
5.0202
4.0996
2.0068
1.0163
5.0940
2.0344
1.0000
f1 (ppm)


lm1122c13c
178.4379
175.4057
140.6747
137.4840
131.8409
129.1329
128.5996
128.5441
128.4410
126.3177
126.2227
125.8648
125.8280
123.3732
122.2821
71.9413
44.9621
44.3837
38.5760
37.1062
32.6803
f1 (ppm)


O
NPhth
Ph
CO2Et
5
ian13571h
2.0558
2.0787
2.0314
3.0194
1.0000
2.0711
1.0835
1.0875
4.0537
3.0513
f1 (ppm)
ian135713c
205.4020
168.7189
167.3007
140.6290
134.1815
131.7532
128.4331
128.2250
126.6091
123.5649
62.2110
47.5108
44.3376
41.7205
29.4298
13.9936
f1 (ppm)