Electronic Supplementary Material (ESI) for ChemComm.

Nickel-Catalyzed Reductive Dicarbofunctionalization of Alkenes via Radical Cyclization

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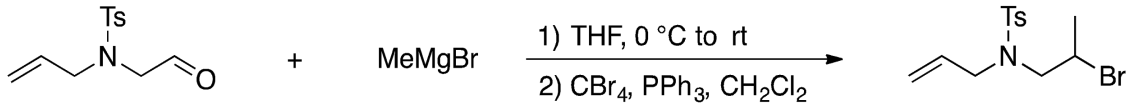
1. General Considerations

Metal, ligands, solvents, and most substrates were obtained from commercial sources. All air- and moisture-sensitive manipulations were carried out in a glove box or using standard Schlenk techniques. ^{1}H NMR spectra were recorded on Agilent 400, Villiane 400, and Bruker 400, 500, or 600 Avance spectrometer (400 MHz, 500 MHz, or 600 MHz). Chemical shifts are reported in ppm relative to tetramethylsilane, with the residual solvent resonance ($CDCl_3$, δ = 7.26) or TMS (δ = 0.00) as the internal reference. Spectra are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant (Hz), integration and assignment. ^{13}C NMR spectra were recorded on Agilent 400, Varian 400, Bruker 400 and Bruker 500 Avance spectrometers (101 or 151 MHz). Chemical shifts were reported in ppm relative to tetramethylsilane with the solvent resonance used as the internal reference ($CDCl_3$, δ = 77.2). HRMS was recorded on an Agilent 6224 TOF LC/MS (EI, ESI, or APCI source).

2. Synthesis of substrates

1) Synthesis of secondary bromide substrate

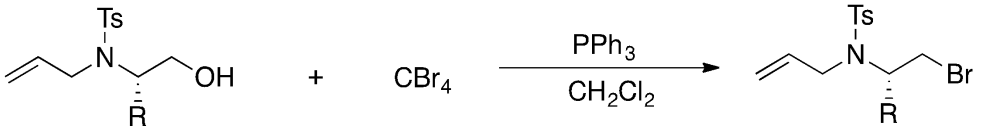


To a MeMgBr solution (1 N in THF, 10 mL) was added a THF solution of aldehyde (506 mg, 2 mmol), prepared according to a reported procedure[1], at 0 °C under nitrogen. Then the mixture was warmed up to room temperature and stirred for 1 hour, followed by quenching with saturated aqueous NH_4Cl solution. The reaction mixture was extracted with EtOAc (3 x 10 mL), dried over $MgSO_4$, filtered, and concentrated. The crude material was diluted with 10 mL CH_2Cl_2 and to this solution was added PPh_3 (2.4 equiv., 4.8 mmol) and CBr_4 (1.2 equiv., 2.4 mmol). After stirring at room temperature for 1 hour, the mixture was concentrated and purified by column chromatography on silica gel (hexanes:EtOAc = 8:1), which gave 265 mg (40% total yield) colorless oil. ^{1}H NMR (400

1. O. Tamura, T. Mitsuya, X. Huang, Y. Tsutsumi, S. Hattori and H. Ishibashi, *J. Org. Chem.* 2005, **70**, 10720.

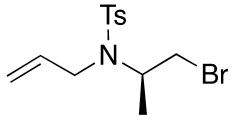
MHz, Chloroform-*d*) δ 7.71 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 5.68 – 5.47 (m, 1H), 5.20 – 5.17 (m, 1H), 5.17 – 5.14 (m, 1H), 4.29 (dt, *J* = 8.2, 6.6 Hz, 1H), 3.85 (d, *J* = 6.8 Hz, 1H), 3.79 (d, *J* = 6.8 Hz, 1H), 3.44 (dd, *J* = 14.6, 8.2 Hz, 1H), 3.33 (dd, *J* = 14.6, 6.4 Hz, 1H), 2.44 (s, 3H), 1.73 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 143.7, 136.2, 132.7, 129.9, 127.3, 119.9, 55.2, 52.5, 46.8, 23.3, 21.6. HRMS (APCI-TOF) calcd for $C_{13}H_{19}{}^{79}BrNO_2S^+$ (M + H$^+$) = 332.0314, found: 332.0314; HRMS (APCI-TOF) calcd for $C_{13}H_{19}{}^{81}BrNO_2S^+$ (M + H$^+$) = 334.0294, found: 334.0292.

2) Synthesis of chiral α-substituted bromide substrate



R = Me and Bn

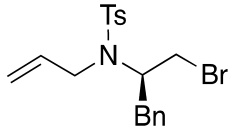
Alcohol was prepared according to a reported procedure[2] from methyl *L*-alaninate•HCl or methyl *L*-phenylalaninate•HCl. The corresponding alcohol (2 mmol) was diluted with 10 mL CH_2Cl_2 and to this solution was added PPh_3 (2.4 equiv., 4.8 mmol) and CBr_4 (1.2 equiv., 2.4 mmol). After stirring at room temperature for 1 hour, the reaction mixture was concentrated and purified by column chromatography on silica gel (hexanes:EtOAc = 8:1).



40% yield, colorless oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.72 (d, *J* = 8.3 Hz, 2H), 7.30 (d, *J* = 8.3 Hz, 2H), 5.84 (dddd, *J* = 17.1, 10.1, 6.9, 5.7 Hz, 1H), 5.21 (ddd, *J* = 17.2, 2.7, 1.3 Hz, 1H), 5.14 (ddd, *J* = 10.2, 2.5, 1.2 Hz, 1H), 4.18 – 4.07 (m, 1H), 3.94 (ddt, *J* = 16.3, 5.7, 1.3 Hz, 1H), 3.72 (ddt, *J* = 16.3, 6.9, 1.2 Hz, 1H), 3.48 (dd, *J* = 10.2, 6.1 Hz, 1H), 3.33 (dd, *J* = 10.1, 8.4 Hz, 1H), 2.43 (s, 3H), 1.24 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 143.6, 137.8, 135.9, 129.9, 127.4, 118.1, 55.5, 47.2, 35.4, 21.7, 17.3. HRMS (APCI-TOF) calcd for $C_{13}H_{19}BrNO_2S^+$ (M + H$^+$) = 332.0314, found: 332.0316

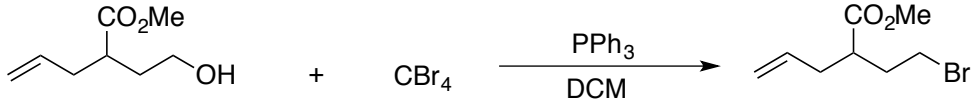
60% yield, white solid, M.P. 67.2 ~ 67.4 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.59 (d, *J* = 8.4, 2H), 7.36 – 7.16 (m, 5H), 7.12 (d, *J* = 8.0, 2H), 5.92 – 5.76 (m, 1H), 5.23 (d, *J* = 17.2, 1H), 5.15 (d, *J* = 10.1, 1H), 4.31 – 4.10 (m, 1H), 3.85 (d, *J* = 6.4 Hz, 2H), 3.55

2. S. Bera and G. Panda, *ACS Comb. Sci.*, 2012, **14** (1), 1–4.



(ddd, *J* = 10.8, 7.7, 1.4 Hz, 1H), 3.43 (ddd, *J* = 10.8, 6.1, 1.4 Hz, 1H), 3.16 – 3.02 (m, 1H), 3.01 – 2.91 (m, 1H), 2.38 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 143.3, 137.4, 137.4, 135.7, 129.5, 129.0, 128.7, 127.4, 126.8, 118.2, 61.8, 48.0, 38.8, 33.5, 21.5. HRMS (ESI-TOF) calcd for $C_{19}H_{23}{}^{79}BrNO_2S^+$ (M + H$^+$) = 408.0627, found 408.0623; HRMS (ESI-TOF) calcd for $C_{19}H_{23}{}^{81}BrNO_2S^+$ (M + H$^+$) = 410.0612, found 410.0604.

3) Synthesis of mono-substituted carbo-substrate



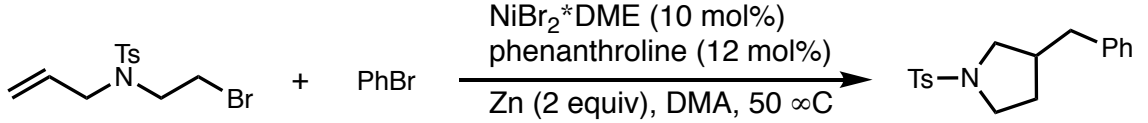
Alcohol was prepared according to the reported procedure[3] from γ-lactone. And then, the corresponding alcohol (2 mmol) was diluted with 10 mL DCM and added with PPh_3 (2.4 eq, 4.8 mmol) and CBr_4 (1.2 eq, 2.4 mmol) at 0 °C. After stirring for 1 hour, it was concentrated and purified by column chromatography on silica gel (PE:EtOAc = 20:1) to give 250 mg (57% yield) colorless oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 5.72 (ddt, *J* = 17.1, 10.1, 7.0 Hz, 1H), 5.14 – 5.03 (m, 2H), 3.70 (s, 3H), 3.44 (ddd, *J* = 10.1, 7.0, 5.9 Hz, 1H), 3.36 (ddd, *J* = 10.1, 7.9, 6.6 Hz, 1H), 2.73 (dtd, *J* = 9.1, 6.9, 4.9 Hz, 1H), 2.44 – 2.35 (m, 1H), 2.33 – 2.26 (m, 1H), 2.26 – 2.17 (m, 1H), 2.05 – 1.95 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.9, 134.5, 117.5, 51.8, 43.4, 36.0, 34.1, 31.0.

3. General procedure

To a 10 mL Schlenk tube, 1,10-phenanthroline (12 mol%). $NiBr_2$•DME (10 mol%) and ground, activated zinc (2.0 equiv., 0.2 mmol) were added under N_2 flow. Subsequently, 6-bromo-1-ene (0.1 mmol, 0.1 equiv.) and the electrophile (0.2 mmol, 2.0 equiv.) were injected via syringe. The reaction was allowed to stir at 50 °C for 16 hours under N_2, the product was purified by column chromatography.

3. B. M. Trost and K. D. Moeller, *Heterocycles*, 1989, **20**, 321.

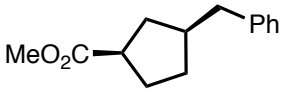
Gram-scale reaction



1.26 g, 80% yield

A reaction on the 5 mmol scale reaction was carried out by using 50 mL DMA in 100 mL bomb flask following the general procedure. After 16 hours, the solvent was removed at 80 °C by rotor-vapor. Finally, 1.26 g (80% yield) colorless oil was collected by column chromatography on silica gel (PE:EtOAc = 8:1).

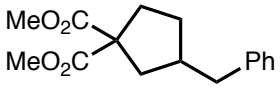
methyl 3-benzylcyclopentane-1-carboxylate



This compound was purified by column chromatography on silica gel (PE:Et_2O = 20 :1) to give colorless oil following the general procedure (as a 2:1 mixture of *cis*:*trans* diastereomers). Due to its overlap with the direct coupling byproduct on column separation, the accurate yield and diastereoselectivity (64% NMR yield, 3:1 *cis*:*trans*) were detected by using NMR spectra after a short pad of silica gel with 10:1 PE:EtOAc as eluent. The relative configuration was confirmed by using the 2D COSY and NOESY spectra, ^{13}C NMR was assigned according to 2D HSQC spectra. Major diastereomer (*cis*): ^1H NMR (400 MHz, Chloroform-*d*) δ 7.30 – 7.24 (m, 2H, *m*-Ar), 7.21 – 7.12 (m, 3H, *o*-Ar and *p*-Ar), 3.67 (s, 3H, CO_2Me), 2.81 – 2.71 (m, 1H, $CHCO_2Me$), 2.66 (dd, *J* = 7.4, 4.4, 2H, CH_2Ar), 2.22 – 2.02 (m, 1H, $CHCH_2Ar$), 2.08 – 1.95 (m, 1H, $CO_2MeCHCH_2CH_2$), 1.95 -1.89 (m, 1H, $CHCH_2CHBn$), 1.89 – 1.82 (m, 1H, $CHCH_2CHBn$), 1.82 – 1.71 (m, 1H, $CO_2MeCHCH_2CH_2$), 1.54 – 1.46 (m, 1H, $CO_2MeCHCH_2CH_2$), 1.45 – 1.37 (m, 1H, $CO_2MeCHCH_2CH_2$). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.1 (CO_2Me), 141.7 (*i*-Ar), 128.7 (*o*-Ar), 128.2 (*m*-Ar), 125.7 (*p*-Ar), 51.6 (CO_2Me), 43.3 (CO_2MeCH), 42.5 ($CHCH_2Ar$), 41.6 (CH_2Ar), 36.7 ($CO_2MeCHCH_2CH_2$), 31.8 (CH_2CH_2CHBn), 28.7 ($CHCH_2CH$). Minor diastereomer (*trans*): ^1H NMR (400 MHz, Chloroform-*d*) δ 7.30 – 7.24 (m, 2H, *m*-Ar), 7.21 – 7.12 (m, 3H, *o*-Ar and *p*-Ar), 3.65 (s, 3H, CO_2Me), 2.92 – 2.81 (m, 1H, $CHCO_2Me$), 2.61 (dd, *J* = 7.4, 4.4, 2H, CH_2Ar), 2.36 – 2.24 (m, 1H, $CHCH_2Ar$), 1.82 – 1.76 (m, 1H, $CO_2MeCHCH_2CH_2$), 1.76 – 1.66 (m, 1H, $CO_2MeCHCH_2CH_2$), 1.61 – 1.54 (m, 2H, $CHCH_2CH$), 1.32 – 1.19 (m, 2H, $CO_2MeCHCH_2CH_2$ and $CO_2MeCHCH_2CH_2$). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.2 (CO_2Me), 141.4 (*i*-Ar), 128.8 (*m*-Ar), 125.8 (*p*-Ar), 42.6 ($CHCO_2Me$), 41.6 (CH_2Ar), 41.2 ($CHCH_2Ar$), 35.6 ($CHCH_2CH$), 32.6 ($CO_2MeCHCH_2CH_2$), 29.5 ($CO_2MeCHCH_2CH_2$).

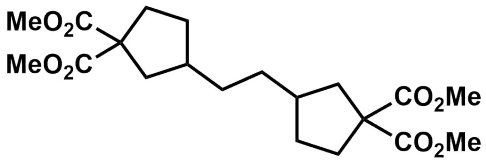
4. Characterization of products

dimethyl 3-benzylcyclopentane-1,1-dicarboxylate **2**:



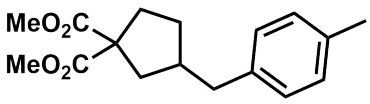
This compound was purified by column chromatography on silica gel (PE:EtOAc = 15:1) to give 25.9 mg (94% yield) colorless oil following the general procedure with PhBr as the cross coupling partner, and 21.4 mg (77% yield) when PhI was used as the cross coupling partner. NMR of the resulting product matched with the reported one[4]. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.27 (t, *J* = 7.6 Hz, 2H), 7.21 – 7.13 (m, 3H), 3.72 (s, 3H), 3.69 (s, 3H), 2.71 – 2.58 (m, 2H), 2.42 (dd, *J* = 13.3, 7.3 Hz, 1H), 2.38 – 2.30 (m, 1H), 2.30 – 2.21 (m, 1H), 2.20 – 2.10 (m, 1H), 1.88 – 1.77 (m, 2H), 1.38 (dq, *J* = 12.5, 9.2 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.3, 173.2, 141.3, 128.9, 128.4, 126.0, 60.0, 52.8, 52.8, 41.7, 41.4, 40.7, 34.0, 32.1. HRMS (ESI-TOF) calcd for $C_{16}H_{21}O_4^+$ (M + H^+) = 277.1434, found 277.1434.

tetramethyl 3,3'-(ethane-1,2-diyl)bis(cyclopentane-1,1-dicarboxylate) **4**:



This compound was purified by column chromatography on silica gel (PE:EtOAc = 2:1) to give 20.1 mg (99% yield) colorless oil following the general procedure, except using 4,4'-di-*tert*-butyl-2,2'-bipyridine instead of 1,10-phenanthroline and without adding a cross coupling partner. NMR of the resulting product matched with the reported one[5]. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 3.68 (s, 12H), 2.41 (dd, *J* = 13.2, 8.0 Hz, 2H), 2.32 – 2.21 (m, 2H), 2.16 – 2.05 (m, 2H), 1.91 – 1.77 (m, 4H), 1.68 – 1.61 (m, 2H), 1.33 – 1.25 (m, 4H), 1.25 – 1.17 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.2, 59.8, 52.6, 40.8, 39.9, 39.9, 34.1, 33.9, 32.0. HRMS (ESI-TOF) calcd for $C_{20}H_{34}NO_8^+$ (M + NH_4^+) = 416.2279, found 416.2271.

dimethyl 3-(4-methylbenzyl)cyclopentane-1,1-dicarboxylate:

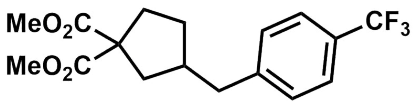


This compound was purified by column chromatography on silica gel (PE:EtOAc = 15:1) to give 29.1 mg (99%

4. A. Millán, L. Álvarez de Cienfuegos, D. Miguel, A. G. Campaña and J. M. Cuerva, *Org. Lett.* 2012, **14**, 5984.
5. H. Tran, T. McCallum, M. Morin and L. Barriault, *Org. Lett.* 2016, **18**, 4308.

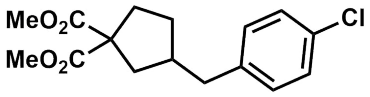
yield) colorless oil following the general procedure. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.08 (d, *J* = 8.0 Hz, 2H), 7.05 (d, *J* = 8.1 Hz, 2H), 3.72 (s, 3H), 3.70 (s, 3H), 2.61 (qd, *J* = 13.6, 7.4 Hz, 2H), 2.42 (dd, *J* = 13.3, 7.3 Hz, 1H), 2.37 – 2.30 (m, 4fH), 2.29 – 2.21 (m, 1H), 2.15 (ddd, *J* = 13.6, 9.3, 7.6 Hz, 1H), 1.86 – 1.81 (m, 2H), 1.37 (dq, *J* = 12.6, 9.3 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.34, 173.25, 138.2, 135.4, 129.1, 128.7, 60.0, 52.8, 52.8, 41.8, 41.0, 40.7, 34.0, 32.1, 21.1. HRMS (ESI-TOF) calcd for $C_{17}H_{22}NaO_4^+$ ($M + Na^+$) = 313.1410, found 313.1414.

dimethyl 3-(4-(trifluoromethyl)benzyl)cyclopentane-1,1-dicarboxylate :



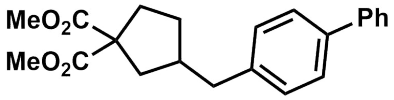
This compound was purified by column chromatography on silica gel (PE:EtOAc = 15:1) to give 32.7 mg (95% yield) colorless oil following the general procedure in a 1.8 mL GC vial in a thermoshaker. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.53 (d, *J* = 8.1 Hz, 2H), 7.27 (d, *J* = 7.4 Hz, 2H), 3.73 (s, 3H), 3.70 (s, 3H), 2.77 – 2.66 (m, 2H), 2.40 (dd, *J* = 13.4, 7.3 Hz, 1H), 2.37 – 2.32 (m, 1H), 2.32 – 2.23 (m, 1H), 2.16 (ddd, *J* = 13.7, 9.3, 7.8 Hz, 1H), 1.87 – 1.77 (m, 2H), 1.38 (dq, *J* = 12.5, 9.3 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.2, 173.1, 145.4, 129.2, 128.6, 125.4 (q, *J* = 3.6 Hz), 123.4, 59.9, 52.9, 52.9, 41.5, 41.2, 40.6, 34.0, 32.1. HRMS (APCI-TOF) calcd for $C_{17}H_{20}F_3O_4^+$ ($M + H^+$) = 345.1308, found: 345.1309.

dimethyl 3-(4-chlorobenzyl)cyclopentane-1,1-dicarboxylate:



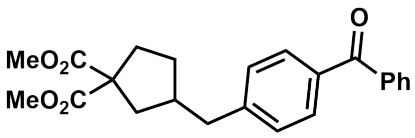
This compound was purified by column chromatography on silica gel (PE:EtOAc = 15:1) to give 24.8 mg (80% yield) colorless oil following the general procedure. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.23 (d, *J* = 8.0 Hz, 2H), 7.08 (d, *J* = 8.0 Hz, 2H), 3.72 (s, 3H), 3.70 (s, 3H), 2.67 – 2.55 (m, 2H), 2.38 (dd, *J* = 13.3, 7.2 Hz, 1H), 2.35 – 2.29 (m, 1H), 2.27 – 2.18 (m, 1H), 2.18 – 2.09 (m, 1H), 1.85 – 1.77 (m, 2H), 1.35 (dq, *J* = 18.7, 9.4 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.1, 173.0, 139.5, 131.6, 130.0, 128.4, 59.7, 52.7, 41.4, 40.5, 40.4, 33.8, 31.9. HRMS (ESI-TOF) calcd for $C_{16}H_{23}NClO_4^+$ ($M + NH_4^+$) = 328.1310, found 328.1310.

dimethyl 3-([1,1'-biphenyl]-4-ylmethyl)cyclopentane-1,1-dicarboxylate:



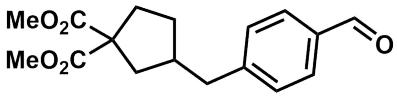
This compound was purified by column chromatography on silica gel (PE:EtOAc = 15:1) to give 35.8 mg (99% yield) white solid following the general procedure, M.P. 73.6 ~ 73.8 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.59 (d, *J* = 7.8 Hz, 2H), 7.52 (d, *J* = 7.9 Hz, 2H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.34 (t, *J* = 7.2 Hz, 1H), 7.24 (d, *J* = 7.9 Hz, 2H), 3.74 (s, 3H), 3.71 (s, 3H), 2.77 – 2.65 (m, 2H), 2.47 (dd, *J* = 13.4, 7.2 Hz, 1H), 2.42 – 2.35 (m, 1H), 2.34 – 2.26 (m, 1H), 2.23 – 2.13 (m, 1H), 1.93 – 1.85 (m, 1H), 1.42 (dq, *J* = 18.6, 9.2 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.3, 173.3, 141.2, 140.4, 139.0, 129.3, 128.9, 127.2, 127.1, 60.0, 52.9, 52.8, 41.7, 41.0, 40.7, 34.0, 32.2. HRMS (ESI-TOF) calcd for $C_{22}H_{28}NO_4^+$ (M + NH_4^+) = 370.2013 found 370.2009.

dimethyl 3-(4-benzoylbenzyl)cyclopentane-1,1-dicarboxylate:



This compound was purified by column chromatography on silica gel (PE:EtOAc = 15:1) to give 28.1 mg (74% yield) white solid following the general procedure, 57.6 ~ 57.8 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.79 (d, *J* = 8.2 Hz, 2H), 7.73 (d, *J* = 7.7 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.27 (d, *J* = 7.4 Hz, 2H), 3.73 (s, 3H), 3.70 (s, 3H), 2.80 – 2.67 (m, 2H), 2.43 (dd, *J* = 13.3, 7.2 Hz, 1H), 2.39 – 2.34 (m, 1H), 2.32 – 2.25 (m, 1H), 2.22 – 2.12 (m, 1H), 1.90 – 1.82 (m, 2H), 1.40 (dq, *J* = 18.4, 9.1 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 196.6, 173.2, 173.1, 146.4, 138.0, 135.5, 132.4, 130.5, 130.1, 128.8, 128.4, 59.9, 52.9, 41.4, 41.4, 40.6, 34.0, 32.1. HRMS (ESI-TOF) calcd for $C_{23}H_{28}NO_5^+$ (M + NH_4^+) = 398.1962, found 398.1956.

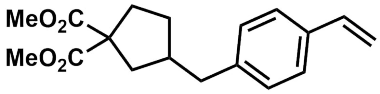
dimethyl 3-(4-formylbenzyl)cyclopentane-1,1-dicarboxylate:



This compound was purified by column chromatography on neutral Al_2O_3 (PE:EtOAc = 5:1) to give 26.0 mg (85% yield) colorless oil following the general procedure. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.97 (s, 1H), 7.80 (d, *J* = 8.2 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 3.73 (s, 3H), 3.70 (s, 3H), 2.80 – 2.68 (m, 2H), 2.44 – 2.38 (m, 1H), 2.37 – 2.32 (m, 1H), 2.32 –

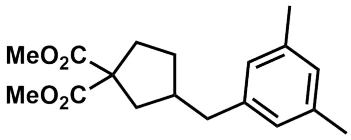
2.24 (m, 1H), 2.16 (ddd, *J* = 13.7, 9.4, 7.6 Hz, 1H), 1.88 – 1.77 (m, 2H), 1.44 – 1.32 (m, 1H). z^{13}C NMR (101 MHz, Chloroform-*d*) δ 192.0, 173.0, 173.0, 148.6, 134.6, 130.0, 129.4, 59.7, 52.8, 52.8, 41.4, 41.2, 40.4, 33.8, 32.0. HRMS (ESI-TOF) calcd for $C_{17}H_{21}O_5^+$ (M + H^+) = 305.1384, found: 305.1376.

dimethyl 3-(4-vinylbenzyl)cyclopentane-1,1-dicarboxylate:



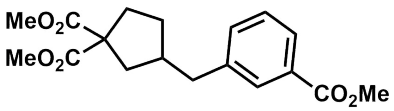
This compound was purified by column chromatography (PE:EtOAc = 15:1) to give 26.0 mg (86% yield) colorless oil following the general procedure. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.32 (d, *J* = 7.7 Hz, 2H), 7.12 (d, *J* = 7.8 Hz, 2H), 6.69 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.70 (d, *J* = 17.6 Hz, 1H), 5.19 (d, *J* = 10.9 Hz, 1H), 3.72 (s, 3H), 3.69 (s, 3H), 2.71 – 2.57 (m, 2H), 2.41 (dd, *J* = 13.4, 7.3 Hz, 1H), 2.37 – 2.29 (m, 1H), 2.25 (dd, *J* = 16.1, 8.6 Hz, 1H), 2.20 – 2.09 (m, 1H), 1.88 – 1.76 (m, 2H), 1.43 – 1.30 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.1, 173.1, 140.9, 136.6, 135.3, 128.8, 126.1, 113.0, 59.8, 52.7, 52.6, 41.5, 40.9, 40.5, 33.8, 31.9. HRMS (ESI-TOF) calcd for $C_{18}H_{26}NO_4^+$ (M + NH_4^+) = 320.1856, found 320.1854.

dimethyl 3-(3,5-dimethylbenzyl)cyclopentane-1,1-dicarboxylate:



This compound was obtained from column chromatography on silica gel (PE:EtOAc = 15:1) to give 26.3 mg (86% yield) colorless oil under the general procedure. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.83 (s, 1H), 6.78 (s, 2H), 3.73 (s, 3H), 3.70 (s, 3H), 2.57 (qd, *J* = 13.5, 7.4 Hz, 2H), 2.42 (dd, *J* = 13.3, 7.2 Hz, 1H), 2.34 (ddd, *J* = 13.3, 8.3, 4.0 Hz, 1H), 2.28 (s, 6H), 2.27 – 2.20 (m, 1H), 2.20 – 2.10 (m, 1H), 1.88 – 1.78 (m, 2H), 1.48 – 1.25 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.4, 173.3, 141.2, 137.8, 127.7, 126.7, 60.0, 52.8, 52.8, 41.7, 41.3, 40.7, 34.0, 32.2, 21.4. HRMS (ESI-TOF) calcd for $C_{18}H_{28}NO_4^+$ (M + NH_4^+) = 322.2013, found 322.2009.

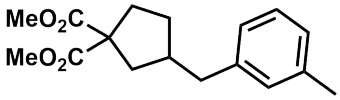
dimethyl 3-(3-(methoxycarbonyl)benzyl)cyclopentane-1,1-dicarboxylate:



This compound was obtained from column chromatography on silica gel (PE:EtOAc = 10:1) to give

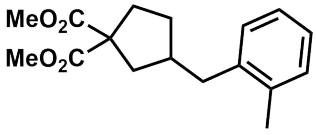
23.4 mg (70% yield) colorless oil following the general procedure in a 1.8 mL GC vial in a thermoshaker. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.88 – 7.80 (m, 2H), 7.37 – 7.30 (m, 2H), 3.90 (s, 3H), 3.72 (s, 3H), 3.69 (s, 3H), 2.69 (qd, *J* = 13.6, 7.5 Hz, 2H), 2.39 (dd, *J* = 13.3, 7.3 Hz, 1H), 2.36 – 2.30 (m, 1H), 2.30 – 2.23 (m, 1H), 2.14 (ddd, *J* = 13.7, 9.2, 7.8 Hz, 1H), 1.86 – 1.76 (m, 2H), 1.37 (dq, *J* = 12.5, 9.3 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.2, 173.1, 167.4, 141.6, 133.5, 130.3, 129.9, 128.5, 127.4, 60.0, 52.9, 52.8, 52.2, 41.6, 41.1, 40.6, 34.0, 32.0. HRMS (APCI-TOF) calcd for $C_{18}H_{23}O_6^+$ (M + H^+) = 335.1489, found: 335.1490.

dimethyl 3-(3-methylbenzyl)cyclopentane-1,1-dicarboxylate:



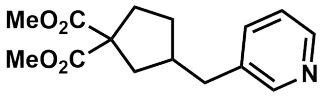
This compound was purified by column chromatography on silica gel (PE:EtOAc = 15:1) to give 24.1 mg (83% yield) colorless oil under the general procedure. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.16 (t, *J* = 7.4 Hz, 1H), 7.02 – 6.93 (m, 3H), 3.73 (s, 3H), 3.70 (s, 3H), 2.61 (qd, *J* = 13.5, 7.4 Hz, 2H), 2.42 (dd, *J* = 13.3, 7.3 Hz, 1H), 2.37 – 2.30 (m, 4H), 2.30 – 2.21 (m, 1H), 2.14 (ddd, *J* = 13.6, 9.3, 7.6 Hz, 1H), 1.87 – 1.77 (m, 2H), 1.37 (ddd, *J* = 18.1, 12.4, 9.2 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.4, 173.3, 141.3, 138.0, 129.7, 128.3, 126.8, 125.9, 60.0, 52.8, 41.7, 41.4, 40.7, 34.0, 32.2, 21.6. HRMS (ESI-TOF) calcd for $C_{17}H_{26}NO_4^+$ (M + NH_4^+) = 308.1856, found 308.1854.

dimethyl 3-(2-methylbenzyl)cyclopentane-1,1-dicarboxylate:



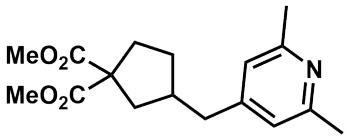
This compound was purified by column chromatography on silica gel (PE:EtOAc = 15:1) to give 23.4 mg (81% yield) colorless oil following the general procedure. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.15 – 7.06 (m, 4H), 3.73 (s, 3H), 3.70 (s, 3H), 2.72 – 2.60 (m, 2H), 2.44 (dd, *J* = 13.4, 7.2 Hz, 1H), 2.35 (ddd, *J* = 13.5, 8.4, 3.8 Hz, 1H), 2.30 (s, 3H), 2.29 – 2.23 (m, 1H), 2.14 (ddd, *J* = 13.5, 9.5, 7.5 Hz, 1H), 1.91 – 1.78 (m, 2H), 1.46 – 1.33 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.4, 173.3, 139.5, 136.1, 130.4, 129.5, 126.2, 126.0, 60.0, 52.9, 40.8, 40.3, 38.5, 34.1, 32.4, 19.7. HRMS (ESI-TOF) calcd for $C_{17}H_{26}NO_4^+$ (M + NH_4^+) = 308.1856, found 308.1855.

dimethyl 3-(pyridin-3-ylmethyl)cyclopentane-1,1-dicarboxylate:



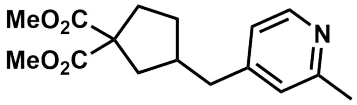
This compound was purified by column chromatography on silica gel which was dealt with 1 mL NEt_3 before loading the sample (PE:EtOAc = 1:1) to give 25.5 mg (92% yield) colorless oil following the general procedure. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.45 – 8.38 (m, 2H), 7.46 (d, *J* = 7.8 Hz, 1H), 7.19 (dd, *J* = 7.6, 4.9 Hz, 1H), 3.71 (s, 3H), 3.68 (s, 3H), 2.70 – 2.57 (m, 2H), 2.39 (dd, *J* = 13.5, 7.4 Hz, 1H), 2.36 – 2.29 (m, 1H), 2.29 – 2.18 (m, 1H), 2.19 – 2.12 (m, 1H), 1.86 – 1.75 (m, 2H), 1.35 (dq, *J* = 18.5, 9.1 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.0, 172.9, 150.0, 147.5, 136.3, 136.1, 123.3, 59.7, 52.7, 41.2, 40.3, 38.2, 33.7, 31.8, 29.7. HRMS (ESI-TOF) calcd for $C_{15}H_{20}NO_4^+$ (M + H^+) = 278.1387, found 278.1389.

dimethyl 3-((2,6-dimethylpyridin-4-yl)methyl)cyclopentane-1,1-dicarboxylate:



This compound was purified by column chromatography on silica gel which was treated with 1 mL Et_3N before loading the sample (PE:EtOAc = 2:1) to give 30.8 mg (99% yield) colorless oil following the general procedure. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.75 (s, 2H), 3.72 (s, 3H), 3.70 (s, 3H), 2.61 – 2.50 (m, 2H), 2.47 (s, 6H), 2.39 (dd, *J* = 13.6, 7.3 Hz, 1H), 2.36 – 2.29 (m, 1H), 2.28 – 2.19 (m, 1H), 2.19 – 2.10 (m, 1H), 1.84 – 1.76 (m, 2H), 1.34 (dq, *J* = 18.4, 9.3 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.2, 173.0, 157.7, 150.6, 120.8, 59.9, 52.9, 40.8, 40.6, 33.9, 32.2, 24.6. HRMS (ESI-TOF) calcd for $C_{17}H_{24}NO_4^+$ (M + H^+) = 306.1700, found 306.1695.

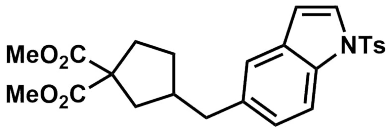
dimethyl 3-((2-methylpyridin-4-yl)methyl)cyclopentane-1,1-dicarboxylate:



This material was purified by column chromatography on silica gel which was treated with 1 mL Et_3N before loading sample (PE:EtOAc = 2:1) to give 28.6 mg (98% yield) colorless oil following the general procedure. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.35 (d, *J* = 5.0 Hz, 1H), 6.95 (s, 1H), 6.89 (d, *J* = 5.2 Hz, 1H), 3.72 (s, 3H), 3.70 (s, 3H), 2.65 – 2.53 (m, 2H), 2.51 (s, 3H), 2.40 (dd, *J* = 13.6, 7.5 Hz, 1H), 2.36 – 2.29 (m, 1H), 2.29 – 2.20 (m, 1H), 2.21 – 2.10 (m, 1H), 1.85 – 1.76 (m, 2H), 1.42 – 1.28 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ

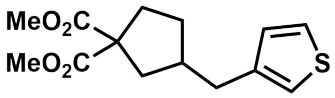
173.2, 173.1, 158.5, 150.4, 149.2, 123.8, 121.4, 59.9, 52.9, 52.9, 40.8, 40.7, 40.6, 33.9, 32.1, 24.5. HRMS (ESI-TOF) calcd for $C_{16}H_{22}NO_4^+$ (M + H^+) = 292.1543, found 292.1540.

dimethyl 3-((1-tosyl-1*H*-indol-5-yl)methyl)cyclopentane-1,1-dicarboxylate:



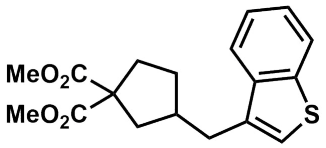
This compound was purified by column chromatography on silica gel (PE:Et_2O = 1:1) to give 31.0 mg (66% yield) colorless oil by using 1.0 equiv. cross coupling partner following the general procedure; 47.9 mg (99% yield) colorless oil by using 2.0 equiv. cross coupling partner following the general procedure. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.89 (d, *J* = 8.4 Hz, 1H), 7.78 (d, *J* = 8.4 Hz, 2H), 7.54 (d, *J* = 3.6 Hz, 1H), 7.35 – 7.30 (m, 1H), 7.24 (d, *J* = 8.4 Hz, 2H), 7.12 (d, *J* = 8.4 Hz, 1H), 6.60 (d, *J* = 3.7 Hz, 1H), 3.74 (s, 3H), 3.70 (s, 3H), 2.78 – 2.65 (m, 2H), 2.40 (dd, *J* = 12.8, 6.7 Hz, 1H), 2.36 (s, 3H), 2.35 – 2.22 (m, 2H), 2.15 (ddd, *J* = 13.6, 9.2, 7.6 Hz, 1H), 1.90 – 1.76 (m, 2H), 1.51 – 1.36 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.2, 173.1, 144.8, 136.3, 135.4, 133.3, 130.9, 129.9, 126.8, 126.4, 125.6, 121.0, 113.2, 108.9, 59.8, 52.7, 52.7, 41.8, 41.1, 40.5, 33.8, 32.0, 21.6. HRMS (ESI-TOF) calcd for $C_{25}H_{31}N_2O_6S^+$ (M + NH_4^+) = 487.1897, found 487.1895.

dimethyl 3-(thiophen-3-ylmethyl)cyclopentane-1,1-dicarboxylate:



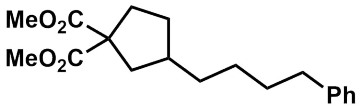
This compound was purified by column chromatography on silica gel (PE:EtOAc = 8:1) to give 24.0 mg (85% yield) colorless oil following the general procedure. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.25 – 7.21 (m, 1H), 6.95 – 6.90 (m, 2H), 3.72 (s, 3H), 3.71 (s, 3H), 2.74 – 2.61 (m, 2H), 2.44 (dd, *J* = 13.3, 7.3 Hz, 1H), 2.38 – 2.22 (m, 2H), 2.21 – 2.10 (m, 1H), 1.91 – 1.77 (m, 2H), 1.37 (dq, *J* = 18.1, 9.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.1, 173.1, 141.4, 128.3, 125.2, 120.6, 59.9, 52.7, 40.8, 40.5, 35.6, 33.8, 32.0. HRMS (ESI-TOF) calcd for $C_{14}H_{22}NO_4S^+$ (M + NH_4^+) = 300.1264, found 300.1253.

dimethyl 3-(benzo[*b*]thiophen-3-ylmethyl)cyclopentane-1,1-dicarboxylate:



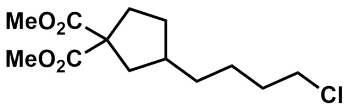
This compound was purified by column chromatography on silica gel (PE:EtOAc = 8:1) to give 23.6 mg (71% yield) colorless oil following the general procedure. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.85 (d, *J* = 7.4 Hz, 1H), 7.74 (d, *J* = 7.7 Hz, 1H), 7.42 – 7.31 (m, 2H), 7.11 (s, 1H), 3.73 (s, 3H), 3.70 (s, 3H), 2.95 – 2.82 (m, 2H), 2.55 – 2.43 (m, 2H), 2.41 – 2.31 (m, 1H), 2.23 – 2.12 (m, 1H), 1.97 – 1.84 (m, 2H), 1.52 – 1.39 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.3, 173.2, 140.6, 139.2, 135.7, 124.3, 124.0, 123.0, 122.0, 121.8, 60.1, 52.9, 40.9, 39.6, 34.1, 34.1, 32.5. HRMS (ESI-TOF) calcd for $C_{18}H_{24}NO_4S^+$ (M + NH_4^+) = 350.1421, found 350.1418.

dimethyl 3-(4-phenylbutyl)cyclopentane-1,1-dicarboxylate:



This compound was purified by column chromatography on silica gel (PE:Et_2O = 20:1) to give 34.7 mg (54% yield) colorless oil following the general procedure but using 5 mol% $NiBr_2$•DME, 6 mol% 1,10-phenanthroline, 0.2 mmol dimethyl 2-allyl-2-(2-bromoethyl)malonate, and 0.8 mmol (3-bromopropyl)benzene in 1.0 mL DMA. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.27 (t, *J* = 7.5 Hz, 2H), 7.20 – 7.14 (m, 3H), 3.72 (s, 3H), 3.71 (s, 3H), 2.60 (t, *J* = 7.7 Hz, 2H), 2.46 (dd, *J* = 13.1, 7.1 Hz, 1H), 2.34 – 2.27 (m, 1H), 2.19 – 2.09 (m, 1H), 1.99 – 1.81 (m, 2H), 1.69 (dd, *J* = 13.3, 10.0 Hz, 1H), 1.66 – 1.59 (m, 2H), 1.42 – 1.30 (m, 4H), 1.29 – 1.21 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.4, 142.9, 128.5, 128.4, 125.8, 60.0, 52.8, 41.1, 39.9, 36.1, 35.3, 34.1, 32.3, 31.8, 28.3. HRMS (ESI-TOF) calcd for $C_{19}H_{30}NO_4^+$ (M + NH_4^+) = 336.2169, found 336.2165.

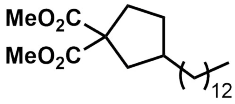
dimethyl 3-(4-chlorobutyl)cyclopentane-1,1-dicarboxylate:



This compound was purified by column chromatography on silica gel (PE:Et_2O = 20:1) to give 33.6 mg (61% yield) colorless oil following the general procedure but using 5 mol% $NiBr_2$•DME, 6 mol% 1,10-phenanthroline, 0.2 mmol dimethyl 2-allyl-2-(2-bromoethyl)malonate, and 0.8 mmol 1-bromo-3-chloropropane in 1.0 mL DMA. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 3.71 (s, 3H), 3.71 (s, 3H), 3.52 (t, *J* = 6.7 Hz, 2H), 2.46 (dd, *J* = 13.4, 7.5 Hz, 1H), 2.35 – 2.26 (m, 1H), 2.14 (ddd, *J* = 13.6, 9.3, 7.5 Hz, 1H), 1.98 – 1.83 (m, 2H), 1.80 – 1.65 (m,

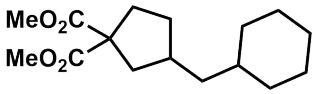
2H), 1.48 – 1.25 (m, 5H).. ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.3, 60.0, 52.8, 45.2, 41.0, 39.8, 34.6, 34.1, 32.9, 32.2, 26.0. HRMS (ESI-TOF) calcd for $C_{13}H_{25}ClNO_4^+$ (M + NH_4^+) = 294.1467, found 294.1466.

dimethyl 3-tetradecylcyclopentane-1,1-dicarboxylate:



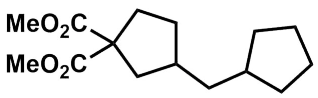
This compound was purified by column chromatography on silica gel (PE:Et_2O = 20:1) to give 25.1 mg (34% yield) colorless oil following the general procedure but using 5 mol% $NiBr_2$•DME, 6 mol% 1,10-phenanthroline, 0.2 mmol dimethyl 2-allyl-2-(2-bromoethyl)malonate, and 0.8 mmol 1-bromododecane in 1.0 mL DMA. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 3.71 (s, 6H), 2.45 (dd, *J* = 13.1, 6.9 Hz, 1H), 2.34 – 2.25 (m, 1H), 2.19 – 2.08 (m, 1H), 1.97 – 1.80 (m, 2H), 1.72 – 1.63 (m, 1H), 1.35 – 1.18 (m, 24H), 0.91 – 0.84 (m, 4H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.6, 173.2, 59.9, 40.9, 39.8, 35.3, 31.9, 29.8, 29.6, 29.6, 29.6, 29.3, 28.5, 14.1. HRMS (ESI-TOF) calcd for $C_{22}H_{41}O_4^+$ (M + H^+) = 369.2999, found 369.2996.

dimethyl 3-(cyclohexylmethyl)cyclopentane-1,1-dicarboxylate:



This compound was purified by column chromatography on silica gel (PE:Et_2O = 20:1) to give 25.4 mg (45% yield) colorless oil following the general procedure but using 5 mol% $NiBr_2$•DME, 6 mol% 1,10-phenanthroline, 0.2 mmol dimethyl 2-allyl-2-(2-bromoethyl)malonate, and 0.8 mmol bromocyclohexane in 1.0 mL DMA. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 3.71 (s, 3H), 3.71 (s, 3H), 2.45 (dd, *J* = 13.2, 7.2 Hz, 1H), 2.31 (ddd, *J* = 12.6 8.6, 3.6 Hz, 1H), 2.17 – 2.08 (m, 1H), 2.08 – 1.97 (m, 1H), 1.90 – 1.79 (m, 1H), 1.73 – 1.59 (m, 6H), 1.33 – 1.07 (m, 7H), 0.93 – 0.73(m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.3, 173.3, 59.8, 52.6, 43.2, 41.1, 36.9, 36.5, 33.9, 33.6, 33.5, 32.4, 26.6, 26.3. HRMS (ESI-TOF) calcd for $C_{16}H_{27}O_4^+$ (M + H^+) = 283.1904, found 283.1905.

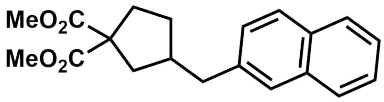
dimethyl 3-(cyclopentylmethyl)cyclopentane-1,1-dicarboxylate:



This compound was purified by column chromatography on silica gel (PE:Et_2O = 20:1) to give 26.0 mg (48% yield) colorless oil following the general procedure but using 5 mol% $NiBr_2$•DME, 6 mol%

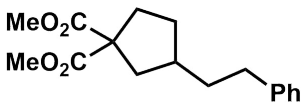
1,10-phenanthroline, 0.2 mmol dimethyl 2-allyl-2-(2-bromoethyl)malonate, and 0.8 mmol bromocyclopentane in 1.0 mL DMA. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 3.71 (s, 3H), 3.71 (s, 3H), 2.47 (dd, *J* = 13.4, 7.0 Hz, 1H), 2.35 – 2.24 (m, 1H), 2.19 – 2.07 (m, 1H), 2.04 – 1.91 (m, 1H), 1.91 – 1.81 (m, 1H), 1.79 – 1.64 (m, 4H), 1.61 – 1.54 (m, 2H), 1.52 – 1.43 (m, 2H), 1.38 – 1.32 (m, 1H), 1.31 – 1.17 (m, 2H), 1.10 – 1.01 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.3, 59.9, 52.6, 41.7, 41.1, 39.2, 39.1, 33.9, 32.9, 32.9, 32.4, 25.1. HRMS (ESI-TOF) calcd for $C_{15}H_{25}O_4^+$ (M + H^+) = 269.1747, found 269.1748.

dimethyl 3-(naphthalen-2-ylmethyl)cyclopentane-1,1-dicarboxylate:



This compound was purified by column chromatography on silica gel (PE:EtOAc = 15:1) to give 12.1 mg (37% yield) colorless oil following the general procedure by using naphthalen-2-yl methanesulfonate as the cross coupling partner. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.87 – 7.73 (m, 3H), 7.60 (s, 1H), 7.49 – 7.38 (m, 2H), 7.31 (dd, *J* = 8.4, 1.6 Hz, 1H), 3.73 (s, 3H), 3.69 (s, 3H), 2.89 – 2.73 (m, 2H), 2.48 – 2.31 (m, 3H), 2.17 (ddd, *J* = 13.6, 9.2, 7.6 Hz, 1H), 1.94 – 1.77 (m, 2H), 1.53 – 1.37 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.4, 173.3, 138.9, 133.7, 132.2, 128.0, 127.8, 127.6, 127.6, 127.0, 126.1, 125.3, 60.0, 52.9, 52.9, 41.7, 41.6, 40.7, 34.0, 32.2. HRMS (ESI-TOF) calcd for $C_{20}H_{26}NO_4^+$ (M + NH_4^+) = 344.1856, found 344.1854.

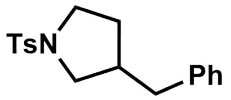
dimethyl 3-phenethylcyclopentane-1,1-dicarboxylate:



This compound was purified by column chromatography on silica gel (PE:Et_2O = 10:1) to give 8.9 mg (31% yield) colorless oil following the general procedure by using benzyl chloride as the cross coupling partner. The corresponding NMR spectra match with the reported ones[3]. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.30 – 7.24 (m, 2H), 7.20 – 7.14 (m, 3H), 3.72 (s, 3H), 3.72 (s, 3H), 2.61 (t, *J* = 8.0 Hz, 2H), 2.51 (dd, *J* = 13.2, 7.2 Hz, 1H), 2.32 (ddd, *J* = 13.3, 8.5, 3.7 Hz, 1H), 2.15 (ddd, *J* = 13.6, 9.2, 7.5 Hz, 1H), 2.03 – 1.86 (m, 2H), 1.77 (dd, *J* = 13.3, 9.8 Hz, 1H), 1.72 – 1.62 (m, 2H), 1.32 (dq, *J* = 12.3, 9.2 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.3, 173.3, 142.5, 128.5, 128.5, 125.9, 60.1, 52.8, 40.9, 39.5, 37.3,

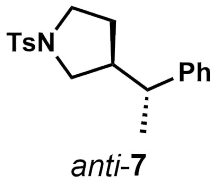
35.0, 34.1, 32.1. HRMS (ESI-TOF) calcd for $C_{17}H_{26}NO_4^+$ (M + NH_4^+) = 308.1856, found 308.1855.

3-benzyl-1-tosylpyrrolidine **5**:



This compound was purified by column chromatography on silica gel (PE:EtOAc = 8:1) to give 25.8 mg (82% yield) colorless oil following the general procedure. The corresponding spectra match with the reported ones[6]. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.70 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.26 (t, *J* = 7.3 Hz, 2H), 7.20 (t, *J* = 7.3 Hz, 1H), 7.06 (d, *J* = 7.0 Hz, 2H), 3.39 (ddd, *J* = 9.8, 8.3, 4.2 Hz, 1H), 3.35 (dd, *J* = 9.8, 7.2 Hz, 1H), 3.20 (dt, *J* = 9.8, 7.8 Hz, 1H), 2.92 (dd, *J* = 9.8, 7.5 Hz, 1H), 2.60 – 2.50 (m, 2H), 2.44 (s, 3H), 2.38 – 2.28 (m, 1H), 1.88 (ddd, *J* = 18.9, 7.0, 4.3 Hz, 1H), 1.50 (dq, *J* = 12.5, 8.2 Hz, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 143.5, 139.9, 134.1, 129.8, 128.7, 128.6, 127.6, 126.4, 53.0, 47.5, 40.6, 39.2, 31.2, 21.7. HRMS (ESI-TOF) calcd for $C_{18}H_{22}NO_2S^+$ (M + H^+) = 316.1366, found 316.1361.

3-(1-phenylethyl)-1-tosylpyrrolidine **7**:

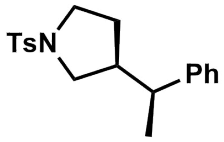


anti-**7**

This compound was purified by column chromatography on silica gel (PE:EtOAc = 10:1) to give 15.0 mg (46% yield) colorless oil as the first fraction following the general procedure. The relative configuration was confirmed by using the 2D COSY and NOESY spectra, which showed the interaction of TsNCH_2 and CHCH_3Ph. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.77 – 7.74 (m, 2H, *m*-Ts), 7.36 (d, *J* = 7.9 Hz, 2H, *o*-Ts), 7.31 – 7.27 (m, 2H, *m*-Ar), 7.23 – 7.17 (m, 1H, *p*-Ar), 7.12 – 7.06 (m, 2H, *o*-Ar), 3.61 (dd, *J* = 9.5, 7.5 Hz, 1H, TsNCH_2CH), 3.35 – 3.29 (m, 1H, TsNCH_2CH$_2$), 3.12 (td, *J* = 9.5, 7.0 Hz, 1H, TsNCH_2CH$_2$), 3.03 – 2.94 (m, 1H, TsNCH_2CH), 2.47 (s, 3H, CH_3 of Ts), 2.44 – 2.39 (m, 1H, C*H*CH$_3$Ph), 2.26 – 2.16 (m, 1H, TsNCH$_2$C*H*), 1.59 – 1.51 (m, 1H, TsNCH$_2$CH_2), 1.32 – 1.26 (m, 1H, TsNCH$_2$CH_2), 1.23 (d, *J* = 7.0 Hz, 3H, CHCH_3Ph). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 145.4, 143.3, 134.0, 129.7, 128.5, 127.5, 127.0, 126.4, 52.3, 47.7,

6. J. Y. Hwang, J. H. Baek, T. I. Shin, J. H. Shin, J. W. Oh, K. P. Kim, Y. You and E. J. Kang, Org. Lett. 2016, **18**, 4900.

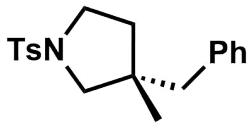
46.0, 43.8, 30.7, 21.6, 21.1. HRMS (ESI-TOF) calcd for $C_{19}H_{24}NO_2S^+$ (M + H^+) = 330.1522, found 330.1521.



syn-**7**

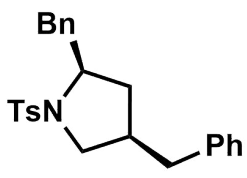
This compound was purified by column chromatography on silica gel (PE:EtOAc = 10:1) to give 16.9 mg (51% yield) white solid as the second fraction following the general procedure, 80.4 ~ 80.6 °C. ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.65 (d, *J* = 8.0 Hz, 2H, *m*-Ts), 7.30 (d, *J* = 8.0 Hz, 2H, *o*-Ts), 7.28 – 7.23 (m, 2H, *m*-Ar), 7.23 – 7.16 (m, 1H, *p*-Ar), 7.09 (d, *J* = 8.0 Hz, 2H, *o*-Ar), 3.43 (td, *J* = 8.8, 2.8 Hz, 1H, TsNCH_2CH$_2$), 3.24 (td, *J* = 10.0, 7.2 Hz, 1H, TsNCH_2CH$_2$), 3.09 (dd, *J* = 10.0, 7.6 Hz, 1H, TsNCH_2CH), 2.70 (t, *J* = 9.6 Hz, 1H, TsNCH_2CH), 2.46 (s, 3H, CH_3 of Ts), 2.46 – 2.37 (m, 1H, C*H*CH$_3$Ph), 2.27 – 2.16 (m, 1H, TsNCH$_2$C*H*), 2.13 – 2.05 (m, 1H, TsNCH$_2$CH_2), 1.52 (tt, *J* = 12.4, 9.2 Hz, 1H, TsNCH$_2$CH_2), 1.22 (d, *J* = 6.8 Hz, 3H, CHCH_3Ph). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 145.4, 143.3, 134.0, 129.7, 128.5, 127.5, 127.0, 126.4, 52.3, 47.7, 46.0, 43.8, 30.7, 21.6, 21.1. HRMS (ESI-TOF) calcd for $C_{19}H_{24}NO_2S^+$ (M + H^+) = 330.1522, found 330.1520.

3-benzyl-3-methyl-1-tosylpyrrolidine **8**:



This compound was purified by column chromatography on silica gel (PE:EtOAc = 8:1) to give 20.0 mg (61% yield) colorless oil following the general procedure. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.71 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.29 – 7.20 (m, 3H), 7.04 (d, *J* = 6.8. Hz, 2H), 3.43 (ddd, *J* = 9.7, 8.4, 5.1 Hz, 1H), 3.30 (dt, *J* = 9.8, 7.6 Hz, 1H), 3.19 (d, *J* = 9.6 Hz, 1H), 2.90 (d, *J* = 9.5 Hz, 1H), 2.60 (d, *J* = 13.3 Hz, 1H), 2.52 (d, *J* = 13.3 Hz, 1H), 2.43 (s, 3H), 1.73 (dt, *J* = 12.5, 7.9 Hz, 1H), 1.48 (ddd, *J* = 12.7, 7.8, 5.1 Hz, 1H), 0.82 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 143.4, 138.1, 134.3, 130.3, 129.8, 128.3, 127.6, 126.6, 58.6, 46.7, 45.1, 43.0, 37.4, 23.5, 21.7. HRMS (ESI-TOF) calcd for $C_{19}H_{23}NNaO_2S^+$ (M + Na^+) = 352.1342, found 352.1346.

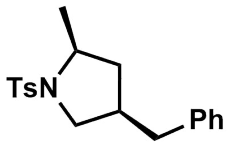
(2*R*)-2,4-dibenzyl-1-tosylpyrrolidine **9**:



This compound was purified by column chromatography on silica gel (PE:EtOAc = 8:1) to give 39.8 mg (98% yield) colorless oil

(3.6:1 mixture of *cis*:*trans* diastereomers) following the general procedure. The ^{1}H NMR match with the reported one[5]. The relative configuration was confirmed by using the 2D COSY and NOESY spectra. ^{13}C NMR was assigned according to 2D HSQC spectra, and for the minor diastereomer, not all ^{13}C NMR peaks could be identified due to overlap with the major diastereomer peaks and lack of signal in the HSQC. Major diastereomer: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.68 (d, *J* = 8.4 Hz, 2H, *m*-Ts), 7.29 – 7.24 (m, 2H, *o*-Ts), 7.25 – 7.13 (m, 8H, Ar of Bn and *m*-Ph, *p*-Ph), 6.93 (d, *J* = 7.7 Hz, 2H, c*o*-Ph), 3.76 – 3.67 (m, 1H, TsNC*H*Bn), 3.54 – 3.45 (m, 1H, TsNCH_2), 3.41 (dd, *J* = 13.2, 3.6 Hz, 1H, TsCHC$H_2C_6H_5$), 2.93 – 2.84 (m, 1H, TsNCH_2), 2.84 – 2.78 (m, 1H, TsCHC$H_2C_6H_5$), 2.49 (dd, *J* = 13.8, 6.8 Hz, 1H, CHCH_2Ph), 2.42 (s, 3H, CH_3 of Ts), 2.41 – 2.35 (m, 1H, CHCH_2Ph), 1.79 (dt, *J* = 12.8, 6.6 Hz, 1H, TsNCHBnCH_2), 1.64 – 1.52 (m, 1H, TsCH_2C*H*Bn), 1.38 – 1.27 (m, 1H, TsNCHBnCH_2). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 143.6 (*i*-Ts), 139.8 (*i*-Ar), 138.3 (*i*-Ar), 135.1, 129.9, 129.8 (*o*-Ts), 128.6 (*o*-Ar), 128.54 (*m*-Ar), 128.45 (*p*-Ar), 127.60 (*m*-Ts), 126.56, 126.4, 62.3 (TsN*C*HBn), 54.9 (TsN*C*H_2), 43.1 (TsCH*C*$H_2C_6H_5$), 39.8 (CH*C*H_2Ph), 38.5 (TsNCHBn*C*H_2), 38.1 (TsCH_2*C*HBn), 21.7 (*C*H_3 of Ts). Minor diastereomer: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.74 (d, *J* = 8.0 Hz, 2H, *m*-Ts), 7.34 – 7.29 (m, 2H, *o*-Ts), 7.26 – 7.15 (m, 8H, Ar of Bn and *m*-Ph, *p*-Ph), 6.96 (d, *J* = 7.6 Hz, 2H, *o*-Ph), 3.92 – 3.83 (m, 1H, TsNC*H*Bn), 3.51 – 3.38 (m, 1H, TsNH_2), 3.23 – 3.13 (m, 1H, TsCHC$H_2C_6H_5$), 2.74 – 2.65 (m, 2H, TsCHC$H_2C_6H_5$), 2.42 (s, 3H, CH_3 of Ts), 2.45 – 2.32 (m, 2H), 1.72 – 1.64 (m, 1H), 1.64 – 1.52 (m, 1H), 1.17 – 1.06 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 139.5, 138.4, 134.3, 129.7 (*o*-Ts), 127.5 (*m*-Ts), 126.4, 61.4 (TsN*C*HBn), 54.1 (TsN*C*H_2), 38.6 (CH*C*H_2Ph), 38.2 (TsNCHBn*C*H_2), 35.6 (*C*H_3 of Ts). HRMS (ESI-TOF) calcd for $C_{25}H_{28}NO_2S^+$ (M + H^+) = 406.1835, found 406.1830.

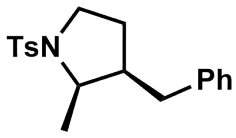
(2*S*)-4-benzyl-2-methyl-1-tosylpyrrolidine **10**:



This compound was purified by column chromatography on silica gel (PE:EtOAc = 8:1) to give 28.6 mg (87% yield) colorless oil (4:1 mixture of *cis*:*trans* diastereomers) following the general procedure in a 1.8 mL vial in a thermoshaker. The relative configuration was confirmed by 2D COSY and NOESY spectra, ^{13}C NMR was assigned according to 2D HSQC spectra. Major

diastereomer: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.66 (d, *J* = 8.2 Hz, 2H, *m*-Ts), 7.38 (d, *J* = 8.0 Hz, 2H, *o*-Ts), 7.37 – 7.32 (m, 2H, *m*-C_6H_5), 7.31 – 7.25 (m, 1H, *p*-C_6H_5), 7.06 – 6.99 (m, 2H, *o*-C_6H_5), 3.55 (dt, *J* = 8.8, 6.3 Hz, 1H, TsNC*H*CH_3), 3.48 (m, 1H, TsNC*H*$_2$), 3.05 (dd, *J* = 11.3, 10.0 Hz, 1H, TsNC*H*$_2$), 2.61 – 2.50 (m, 2H, C*H*$_2C_6H_5$), 2.43 (s, 3H, C*H*$_3$ of Ts), 2.07 – 1.98 (m, 1H, TsNCH(CH_3)C*H*$_2$), 1.84 – 1.70 (m, 1H, TsNCH$_2$C*H*Bn), 1.40 (d, *J* = 6.2 Hz, 3H, TsNCHC*H*$_3$), 1.25 – 1.20 (m, 1H, TsNCH(CH_3)C*H*$_2$). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 143.36 (*i*-Ts), 140.0 (*i*-C_6H_5), 135.3 (*p*-C_6H_5), 129.8 (*o*-Ts), 128.62 (*m*-C_6H_5), 128.59 (*o*-C_6H_5), 127.58 (*m*-Ts), 126.44 (*p*-Ts), 57.0 (TsN*C*HCH_3), 54.6 (TsNC*H*$_2$), 41.3 (TsNCH(CH_3)*C*H$_2$), 39.8 (TsNCH$_2$*C*HBn), 38.9 (*C*H$_2C_6H_5$), 23.0 (CH*C*H$_3$) , 21.7 (*C*H$_3$ of Ts). Minor diastereomer: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.72 (d, *J* = 8.0 Hz, 2H, *m*-Ts), 7.32 (d, *J* = 8.0 Hz, 2H, *o*-Ts), 7.27 – 7.24 (m, 2H, *m*-C_6H_5), 7.21 – 7.17 (m, 1H, *p*-C_6H_5), 7.04 – 7.00 (m, 2H, *o*-C_6H_5), 3.86 – 3.77 (m, 1H, TsNC*H*CH_3), 3.53 – 3.50 (m, 1H, TsNC*H*$_2$), 2.75 (t, *J* = 8.8 Hz, 1H, TsNC*H*$_2$), 2.55 – 2.46 (m, 2H, C*H*$_2C_6H_5$), 2.44 (s, 3H, C*H*$_3$ of Ts), 1.63 – 1.52 (m, H, C*H*$_2C_6H_5$, 1.46 – 1.38 (m, 1H, TsNCH(CH_3)C*H*$_2$), 1.27 (d, *J* = 6.0 Hz, 3H, CHC*H*$_3$), 1.25 – 1.20 (m, 1H, TsNCH(CH_3)C*H*$_2$). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 143.37 (*i*-Ts), 139.9 (*i*-C_6H_5), 134.9 (*p*-C_6H_5), 129.8 (*o*-Ts), 128.67 (*m*-C_6H_5), 128.64 (*o*-C_6H_5), 127.66 (*m*-Ts), 126.44 (*p*-Ts), 55.9 (TsN*C*HCH_3), 54.2 (TsNC*H*$_2$), 39.4 (TsNCH(CH_3)*C*H$_2$), 39.0 (TsNCH$_2$*C*HBn), 38.8 (*C*H$_2C_6H_5$), 23.4 (CH*C*H$_3$), 21.7 (*C*H$_3$ of Ts). HRMS (APCI-TOF) calcd for $C_{19}H_{24}NO_2S^+$ (M+H$^+$) = 330.1522, found 330.1526.

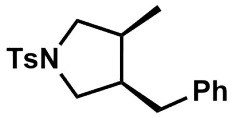
3-benzyl-2-methyl-1-tosylpyrrolidine **11**:



This compound was purified by column chromatography on silica gel (PE:EtOAc = 8:1) to give 24.0 mg (73% yield) colorless oil (1.7:1 mixture of *cis*:*trans* diastereomers) following the general procedure. The relative configuration was confirmed by 2D COSY and NOESY spectra, ^{13}C NMR was assigned according to 2D HSQC spectra. Major diastereomer: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.73 (d, *J* = 8.0 Hz, 2H, *m*-Ts), 7.33 (d, *J* = 8.0 Hz, 2H, *o*-Ts), 7.30 – 7.23 (m, 2H, *m*-C_6H_5), 7.23 – 7.17 (m, 1H, *p*-C_6H_5), 7.07 (d, *J* = 6.0 Hz, 2H, *o*-C_6H_5), 3.56 – 3.49 (m, 1H, TsNC*H*CH_3), 3.39 – 3.33 (m, 1H, TsNC*H*$_2$), 3.05 – 2.96 (m, 1H, TsNC*H*$_2$), 2.67 (dd, *J* = 14.0, 6.0 Hz, 1H, C*H*$_2$Ar), 2.51 (dd, *J* = 14.0, 9.0 Hz, 1H,

CH_2Ar), 2.46 (s, 3H, CH_3 of Ts), 2.15 – 2.05 (m, 1H, C*H*Bn), 2.00 – 1.90 (m, 1H, TsNCH$_2$CH_2), 1.76 – 1.69 (m, 1H, TsNCH$_2$CH_2), 1.25 (d, *J* = 6.5 Hz, 3H, CHCH_3). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 143.3 (*i*-Ts), 140.1 (*i*-C_6H_5), 135.2 (*m*-C_6H_5), 129.8 (*o*-Ts), 128.63 (*o*-C_6H_5), 128.62 (*p*-C_6H_5), 127.5 (*m*-Ts), 126.38 (*p*-Ts), 58.2 (TsN*C*HCH$_3$), 47.4 (TsN*C*H$_2$), 44.1 (*C*HBn), 35.6 (*C*H$_2$Ar), 29.0 (TsNCH$_2$*C*H$_2$), 21.7 (*C*H$_3$ of Ts), 17.6 (CH*C*H$_3$). Minor diastereomer: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.76 (d, *J* = 8.0 Hz, 2H, *m*-Ts), 7.35 (d, *J* = 8.0 Hz, 2H, *o*-Ts), 7.30 - 7.24 (m, 2H, *m*-C_6H_5), 7.23 – 7.18 (m, 1H, *p*-C_6H_5), 6.94 (d, *J* = 6.0 Hz, 2H, *o*-C_6H_5), 3.86 (p, *J* = 6.8 Hz, 1H, TsNC*H*CH$_3$), 3.46 – 3.41 (m, 2H, TsNCH_2), 2.48 (s, 3H, CH_3 of Ts), 2.38 (dd, *J* = 13.5, 6.5 Hz, 1H, CH_2Ar), 2.11 (dd, *J* = 13.5, 8.5 Hz, 1H, CH_2Ar), 2.08 – 2.03 (m, 1H, C*H*Bn), 1.72 – 1.68 (m, 1H, TsNCH$_2$CH_2), 1.35 (d, *J* = 6.5 Hz, 3H, CHCH_3), 1.24 – 1.22 (m, 1H, TsNCH$_2$CH_2). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 143.4 (*i*-Ts), 139.6 (*i*-C_6H_5), 135.3 (*m*-C_6H_5), 129.8 (*o*-Ts), 128.9 (*o*-C_6H_5), 128.6 (*p*-C_6H_5), 127.6 (*m*-Ts), 126.42 (*p*-Ts), 61.1 (TsN*C*HCH$_3$), 48.3 (TsN*C*H$_2$), 47.7 (*C*HBn), 38.7 (*C*H$_2$Ar), 29.3 (TsNCH$_2$*C*H$_2$), 22.5 (CH*C*H$_3$), 21.7 (*C*H$_3$ of Ts). HRMS (ESI-TOF) calcd for $C_{19}H_{23}NNaO_2S^+$ (M + Na^+) = 352.1342, found 352.1341.

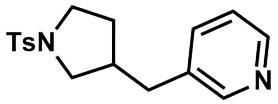
3-benzyl-4-methyl-1-tosylpyrrolidine **12**:



This compound was purified by column chromatography on silica gel (PE:EtOAc = 8:1) to give 23.7 mg (72% yield) colorless oil (2.6:1 mixture of *cis*:*trans* diastereomers) following the general procedure. The relative configuration was confirmed by 2D COSY and NOESY spectra, ^{13}C NMR was assigned according to 2D HSQC spectra. Major diastereomer: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.69 (d, *J* = 8.2 Hz, 2H), 7.31 (t, *J* = 7.7 Hz, 2H), 7.28 – 7.23 (m, 2H), 7.22 – 7.17 (m, 1H), 7.04 (t, *J* = 7.7 Hz, 2H), 3.39 (dd, *J* = 9.8, 6.5 Hz, 1H), 3.24 (dd, *J* = 9.8, 6.5 Hz, 1H), 3.08 (dd, *J* = 9.8, 4.4 Hz, 1H), 3.02 (dd, *J* = 10.0, 7.0 Hz, 1H), 2.64 (q, *J* = 10.3 Hz, 1H), 2.44 (s, 3H), 2.39 – 2.28 (m, 2H), 2.25 – 2.16 (m, 1H), 0.85 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 143.4 (*i*-Ts), 140.0 (*i*-C_6H_5), 134.5 (*p*-Ts), 129.8 (*o*-Ts), 128.7 (*m*-C_6H_5), 128.7 (*o*-C_6H_5), 127.6 (*m*-Ts), 126.4 (*p*-C_6H_5), 54.8 (TsN*C*H$_2$CHCH$_3$), 51.0 (TsN*C*H$_2$CHBn), 43.8 (*C*HBn), 35.6 (*C*HCH$_3$), 34.0 (*C*H$_2$C$_6$H$_5$), 21.7 (*C*H$_3$ of Ts), 13.4 (CH*C*H$_3$). Minor diastereomer: ^{1}H NMR (500 MHz, Chloroform-

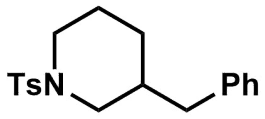
d) δ 7.66 (d, *J* = 8.2 Hz, 2H), 7.31 (t, *J* = 7.7 Hz, 2H), 7.28 – 7.23 (m, 2H), 7.22 – 7.16 (m, 1H), 7.04 (t, *J* = 7.7 Hz, 2H), 3.53 (dd, *J* = 9.8, 7.3 Hz, 1H), 3.31 (dd, *J* = 10.0, 7.2 Hz, 1H), 2.93 (d, *J* = 10.0, 8.2 Hz, 1H), 2.80 (dd, *J* = 9.7, 8.4 Hz, 1H), 2.76 (dd, *J* = 13.8, 4.9 Hz, 1H), 2.43 (s, 3H), 2.39 – 2.28 (m, 1H), 1.91 – 1.75 (m, 2H), 0.92 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 143.4 (*i*-Ts), 139.7 (*i*-C_6H_5), 134.1 (*p*-Ts), 129.8 (*o*-Ts), 128.7 (*m*-C_6H_5), 128.6 (*o*-C_6H_5), 127.6 (*m*-Ts), 126.5 (*p*-C_6H_5), 54.9 (TsN*C*H_2CHCH$_3$), 53.1 (TsN*C*H_2CHBn), 48.0 (*C*HBn), 47.5 (*C*HCH$_3$), 38.7 (*C*$H_2C_6H_5$), 38.2 (*C*H$_3$ of Ts), 16.6 (CH*C*H$_3$). HRMS (APCI-TOF) calcd for $C_{19}H_{24}NO_2S^+$ (M + H^+) = 330.1522, found 330.1522.

3-((1-tosylpyrrolidin-3-yl)methyl)pyridine **13**:



This compound was purified by column chromatography on silica gel which was dealt with 1 mL NEt_3 before loading the sample (PE:EtOAc = 1:2) to give 25.9 mg (82% yield) colorless oil following the general procedure. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.45 (dd, *J* = 4.8, 1.5 Hz, 1H), 8.32 (d, *J* = 1.9 Hz, 1H), 7.69 (d, *J* = 8.2 Hz, 2H), 7.39 (d, *J* = 7.8 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.20 (dd, *J* = 7.8, 4.8 Hz, 1H), 3.39 (ddd, *J* = 9.9, 8.3, 4.2 Hz, 1H), 3.33 (dd, *J* = 9.8, 7.1 Hz, 1H), 3.19 (dt, *J* = 9.8, 7.8 Hz, 1H), 2.90 (dd, *J* = 9.8, 7.5 Hz, 1H), 2.61 – 2.50 (m, 2H), 2.43 (s, 3H), 2.38 – 2.26 (m, 1H), 1.88 (ddd, *J* = 19.0, 7.0, 4.2 Hz, 1H), 1.49 (dq, *J* = 12.6, 8.3 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 149.9, 147.9, 143.5, 136.0, 135.0, 133.7, 129.7, 127.5, 123.4, 52.6, 47.3, 40.0, 36.2, 31.0, 21.6. HRMS (ESI-TOF) calcd for $C_{17}H_{21}N_2O_2S^+$ (M + H^+) = 317.1318, found 317.1314.

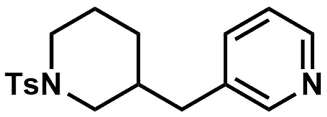
3-benzyl-1-tosylpiperidine **15**:



This compound was purified by column chromatography on silica gel (PE:EtOAc = 15:1) to give 13.5 mg (41% yield) colorless oil following the general procedure. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.62 (d, *J* = 7.7 Hz, 2H), 7.32 (d, *J* = 8.1 Hz, 2H), 7.29 – 7.25 (m, 2H), 7.20 (t, *J* = 6.8 Hz, 1H), 7.11 (d, *J* = 7.7 Hz, 2H), 3.55 (d, *J* = 11.3 Hz, 2H), 2.61 (dd, *J* = 13.5, 6.4 Hz, 1H), 2.44 (s, 3H), 2.42 – 2.32 (m, 2H), 2.12 (t, *J* = 10.7 Hz, 1H), 1.96 – 1.85 (m, 1H), 1.75 – 1.61 (m, 2H), 1.57 – 1.48 (m, 1H), 0.97 – 0.84 (m, 1H). ^{13}C NMR (101 MHz,

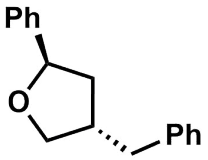
Chloroform-*d*) δ 143.5, 139.5, 133.4, 129.7, 129.2, 128.6, 127.9, 126.4, 51.7, 47.0, 40.2, 37.5, 29.7, 24.4, 21.7. HRMS (ESI-TOF) calcd for $C_{19}H_{24}NO_2S^+$ (M + H^+) = 330.1522, found 330.1520.

3-((1-tosylpiperidin-3-yl)methyl)pyridine **16**:



This compound was purified by column chromatography on alkaline Al_2O_3 (PE:Et_2O = 1:2), and then on silica gel (PE:acetone = 1:1) to give 38.6 mg (29% yield) colorless oil following the general procedure in 0.4 mmol scale. To achieve relative pure staff, some staff was abandoned on the first column chromatography. So, it was double checked by using NMR yield with 20 uL CH_3NO_2 solution (1N in Chloroform-*d*) as internal standard after removing the DMA by column chromatography on silica gel (PE:acetone = 1:2), which showed 40% NMR yield. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.47 (dd, *J* = 4.7, 1.2 Hz, 1H), 8.37 (d, *J* = 1.6 Hz, 1H), 7.61 (d, *J* = 8.2 Hz, 2H), 7.48 (d, *J* = 7.8 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.23 (dd, *J* = 7.7, 4.9 Hz, 1H), 3.57 – 3.42 (m, 2H), 2.62 (dd, *J* = 13.8, 6.8 Hz, 1H), 2.48 – 2.37 (m, 5H), 2.17 (t, *J* = 10.5 Hz, 1H), 1.99 – 1.83 (m, 1H), 1.80 – 1.73 (m, 1H), 1.67 – 1.50 (m, 2H), 1.01 – 0.90 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 150.5, 148.0, 143.6, 136.6, 134.9, 133.3, 129.8, 127.8, 123.6, 51.4, 46.9, 37.2, 37.1, 29.6, 24.2, 21.7. HRMS (ESI-TOF) calcd for $C_{18}H_{23}N_2O_2S^+$ (M + H^+) = 331.1475, found: 331.1470.

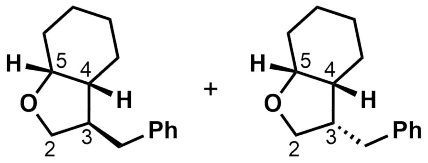
4-benzyl-2-phenyltetrahydrofuran **17**:



This compound was purified by column chromatography on silica gel (PE:EtOAc = 15:1) to give 15.2 mg (64% yield) colorless oil following the general procedure (as a 3.7:1 mixture of *trans*:*cis* diastereomers). The relative configuration was confirmed by using the 2D COSY and NOESY spectra, ^{13}C NMR was assigned according to 2D HSQC spectra. Major diastereomer (*trans*): ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.35 – 7.27 (m, 6H, Ar), 7.26 – 7.15 (m, 4H, Ar), 5.09 (dd, *J* = 7.6, 6.4 Hz, 1H, OC*H*Ph), 4.18 (dd, *J* = 8.4, 6.8 Hz, 1H, OCH_2CHBn), 3.67 (dd, *J* = 8.4, 6.8 Hz, 1H, OCH_2CHBn), 2.77 (d, *J* = 7.6 Hz, 2H, CH_2Ar), 2.70 – 2.60 (m, 1H, OCH_2C*H*Bn), 2.17 – 2.09 (m, 1H, OCHPhCH_2), 2.03 – 1.94 (m, 1H, OCHPhCH_2).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 143.9, 140.7, 128.9, 128.6, 128.5, 127.2, 126.3, 125.6, 80.2 (O*C*HPh), 74.0 (O*C*H_2CHBn), 40.8 (*C*H_2Ar), 40.7 (OCHPh*C*H_2), 39.3 (OCH$_2$*C*HBn). Minor diastereomer, known peaks from the spectra (*cis*): ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.38 – 7.13 (m, 10H, Ar), 4.91 (dd, *J* = 9.8, 6.0 Hz, 1H, OC*H*Ph), 4.07 (dd, *J* = 8.0, 7.1 Hz,1H, OC*H*$_2$CHBn), 3.79 (dd, *J* = 8.1, 7.4 Hz, 1H, OC*H*$_2$CHBn), 2.77 (s, *J* = 7.6 Hz, 1H, OCH2C*H*Bn), 2.73 – 2.60 (m, 2H), 2.47 – 2.37 (m, 1H, OCHPhC*H*$_2$). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 128.8, 128.5, 125.8, 81.4 (O*C*HPh), 73.6 (O*C*H_2CHBn), 42.4 (*C*H_2Ar), 42.1 (OCHPh*C*H_2), 39.6 (OCH$_2$*C*HBn). HRMS (EI) calcd for $C_{17}H_{18}O^+$ (M^+) = 238.1358, found 238.1362.

4,5-*cis*-3-benzyloctahydrobenzofuran **18**:



trans-**18** *cis*-**18**

This compound was purified by column chromatography on silica gel (PE:EtOAc = 15:1) to give 11.2 mg (52% yield) colorless oil following the general procedure from *trans*-1-(allyloxy)-2-bromocyclohexane. The structure and conformation were determined by comparison to the reported spectra[7] to show a *trans*:*cis* diastereomeric ratio of 1.6:1. When the general procedure was followed with the *cis*- diastereomer of the substrate, 5.7 mg (27% yield) colorless oil with identical NMR spectra and *trans*:*cis* ratio of 1.5:1 was obtained. Major diastereomer, *trans*: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.30 – 7.24 (m, 2H, Ar), 7.23 – 7.14 (m, 3H, Ar), 4.08 (t, *J* = 8.1, 1H), 4.02 (q, *J* = 4.7 Hz, 1H), 3.52 (dd, *J* = 8.6, 4.7 Hz, 1H), 2.77 (dd, *J* = 14.2, 7.5 Hz, 1H), 2.60 (dd, *J* = 13.7, 8.5 Hz, 1H), 2.35 – 2.24 (m, 1H), 1.89 – 1.70 (m, 2H), 1.62 – 1.43 (m, 4H), 1.42 – 1.10 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 140.8, 128.9, 128.57, 126.1, 76.4, 72.1, 45.8, 43.1, 40.0, 28.5, 27.6, 23.8, 21.3. Minor diastereomer, selected peak of *cis*: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.30 – 7.24 (m, 2H, Ar), 7.23 – 7.14 (m, 3H, Ar), 3.98 – 3.94 (m, 1H), 3.90 (t, *J* = 7.9 Hz, 1H), 3.69 – 3.62 (m,1H), 2.76 – 2.66 (m, 1H), 2.65 – 2.57 (m, 1H), 1.97 (d, *J* = 12.5 Hz, 1H), 1.89 – 1.70 (m, 2H), 1.62 – 1.43 (m, 4H), 1.42 – 1.10 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 141.1, 128.9, 128.60, 126.2, 78.5, 71.1, 45.8, 40.1, 33.7, 28.8, 24.7, 22.3. HRMS (EI) calcd for $C_{15}H_{20}O^+$ (M^+) = 216.1514, found 216.1511.

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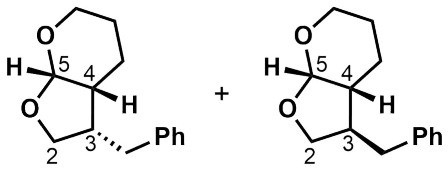
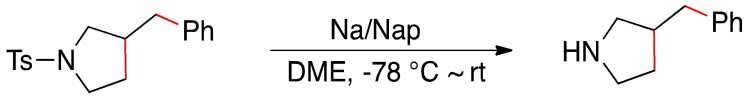


cis-**19** *trans*-**19**

4,5-*cis*-3-benzylhexahydro-4*H*-furo[2,3-*b*]pyran **19**: This compound was purified by column chromatography on silica gel (PE:EtOAc = 5:1) to give 10.9 mg (50% yield) colorless oil following the general procedure. The conformation was determined by comparison to the reported spectra[5] to show 11.5:1 (*cis*: *trans*). For the minor diastereomer, not all ^{13}C NMR peaks could be identified due to overlap with the major diastereomer peaks and lack of signal in 2D HSQC. Major diastereomer, *cis*: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.29 (t, *J* = 7.4 Hz, 2H), 7.20 (t, *J* = 7.5 Hz, 2H), 7.17 (d, *J* = 7.2 Hz, 1H), 5.28 (d, *J* = 3.7 Hz, 1H), 3.88 (t, *J* = 7.7 Hz, 1H), 3.78 (t, *J* = 8.7 Hz, 2H), 3.65 (d, *J* = 11.3 Hz, 1H), 2.78 – 2.59 (m, 3H), 2.00 – 1.91 (m, 1H), 1.81 – 1.72 (m, 1H), 1.66 – 1.51 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 140.3, 128.70, 128.5, 126.36, 102.1, 70.1, 61.2, 42.7, 36.7, 33.5, 23.3, 19.7. Minor diastereomer, *trans*: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.32 – 7.26 (m, 2H), 7.24 – 7.18 (m, 2H), 7.18 – 7.14 (m, 1H), 5.03 (d, *J* = 3.5 Hz, 1H), 4.17 (t, *J* = 8.3 Hz, 1H), 3.69 – 3.61 (m, 2H), 3.42 (td, *J* = 11.5, 2.2 Hz, 1H), 2.86 (dd, *J* = 12.9, 5.1 Hz, 1H), 2.78 – 2.54 (m, 2H), 1.82 – 1.70 (m, 1H), 1.67 – 1.50 (m, 4H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 128.67, 126.40, 102.3, 73.9, 64.6, 44.0, 39.6, 38.9, 22.6, 20.9. HRMS (ESI-TOF) calcd for $C_{14}H_{19}O_2^+$ (M + H^+) = 219.1380, found 219.1382.

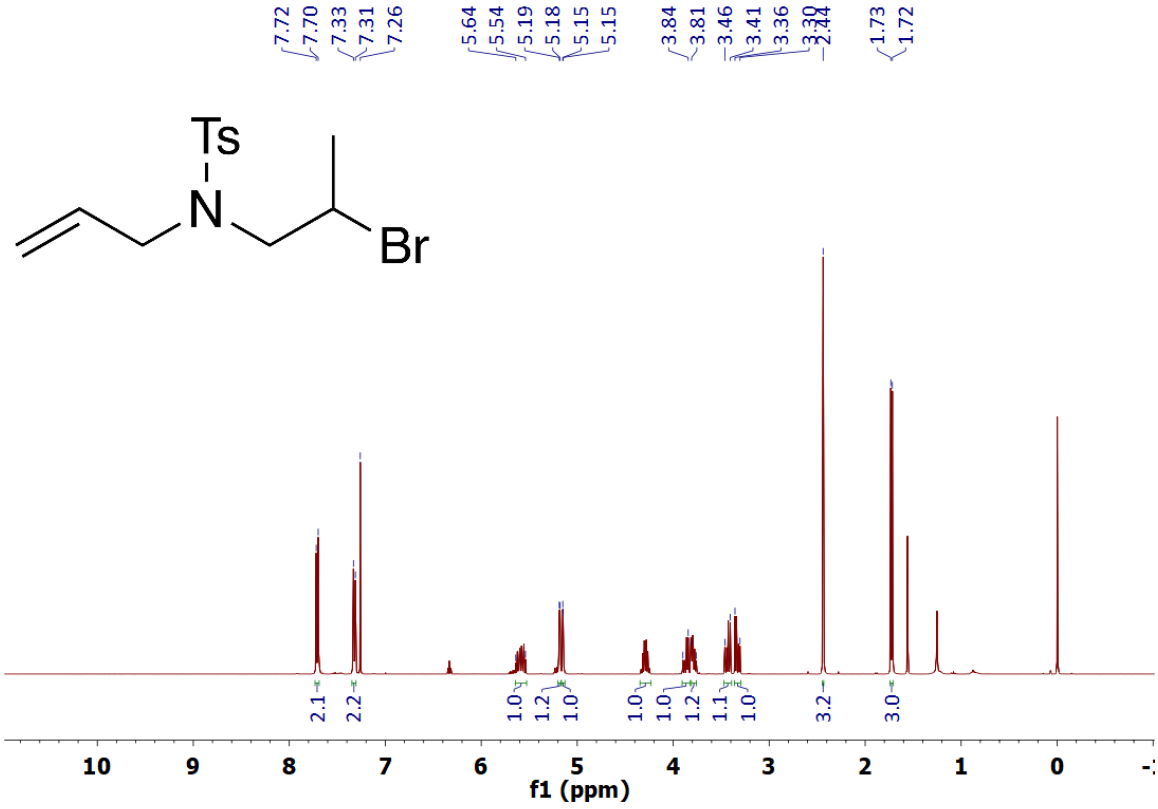
5. Synthesis of the *N*-free pyrrolidine derivative 6

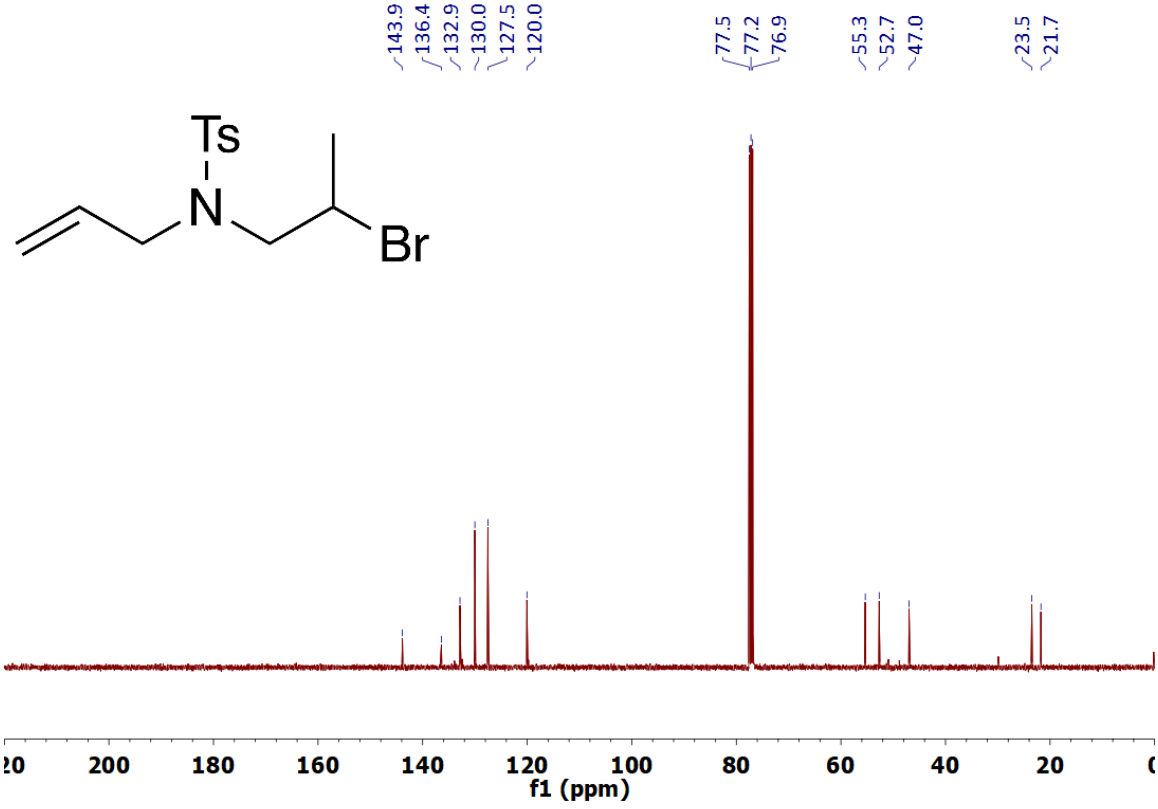


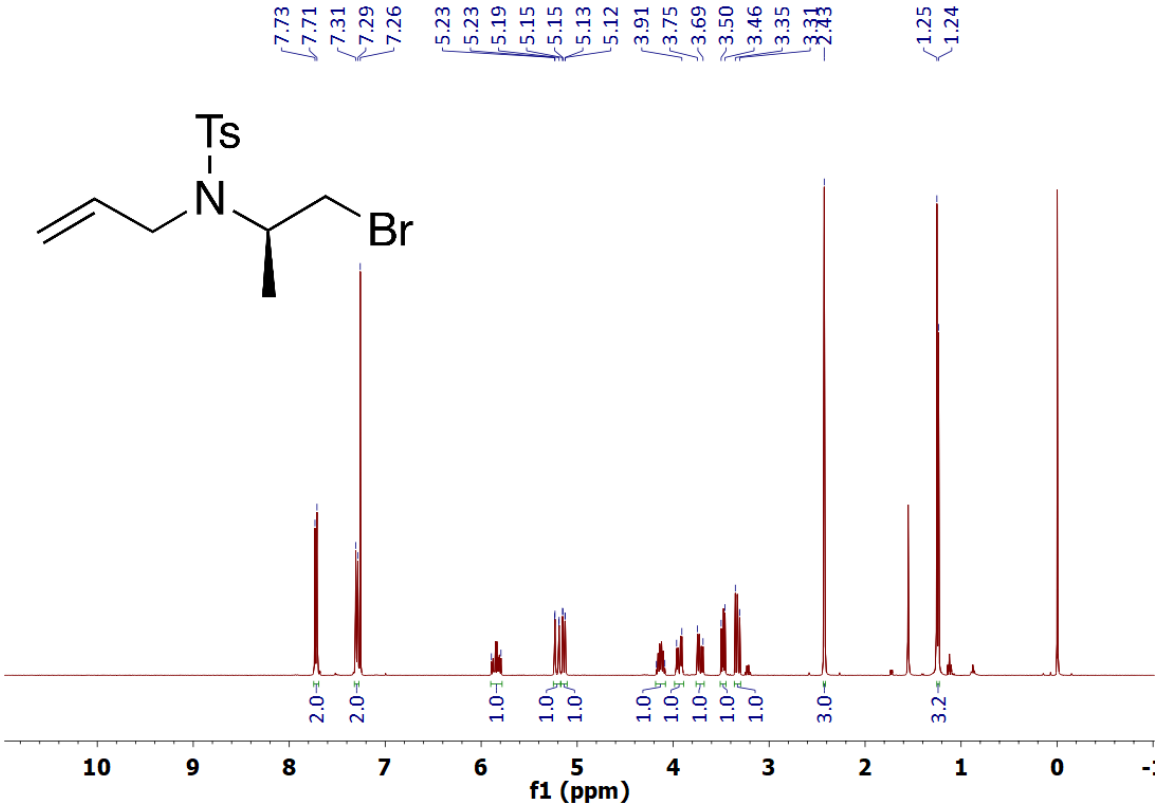
A solution of sodium naphthalenide in DME was prepared by adding DME to a mixture of sodium (100 mg, 4.34 mmol) and naphthalene (700 mg, 5.48 mmol) under nitrogen and stirring the resulting mixture for 2 hours to give a blue solution. Then this solution was added to a solution of starting material (53.7 mg, 0.17 mmol) in 1.0 mL DME dropwise at -78 °C under nitrogen until a green color persisted. Then the mixture was left stirring with the reaction allowed to warm to rt for 2 hours. After another 2 hours, 2 drops saturated aqueous $NaHCO_3$ and 500 mg K_2CO_3 were added to the mixture. After stirring

for 1 hour, the mixture was filtered, washed with Et_2O, and concentrated. Finally, the product was purified by column chromatography on silica gel (MeOH:CH_2Cl_2 = 4:1 to pure MeOH) to give a colorless oil (26.9 mg, 98% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.29 – 7.24 (m, 2H), 7.24 – 7.18 (m, 1H), 7.18 – 7.13 (m, 2H), 3.46 – 3.24 (m, 3H), 3.13 (td, *J* = 7.2, 2.8 Hz, 1H), 3.05 – 2.90 (m, 1H), 2.70 – 2.65 (m, 2H), 2.48 – 2.32 (m, 1H), 2.00 – 1.85 (m, 1H), 1.60 – 1.47 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 140.6, 128.8, 128.6, 126.3, 51.2, 45.5, 41.0, 39.7, 31.6. HRMS (EI) calcd for $C_{11}H_{15}N$ (M^+) = 161.1204, found 161.1202.

6. NMR spectra



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3.46
3.35
3.31
2.43
1.25
1.24
Ts
N
Br
2.0
2.0
1.0
1.0
1.0
1.0
1.0
1.0
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3.0
3.2
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9
8
7
6
5
4
3
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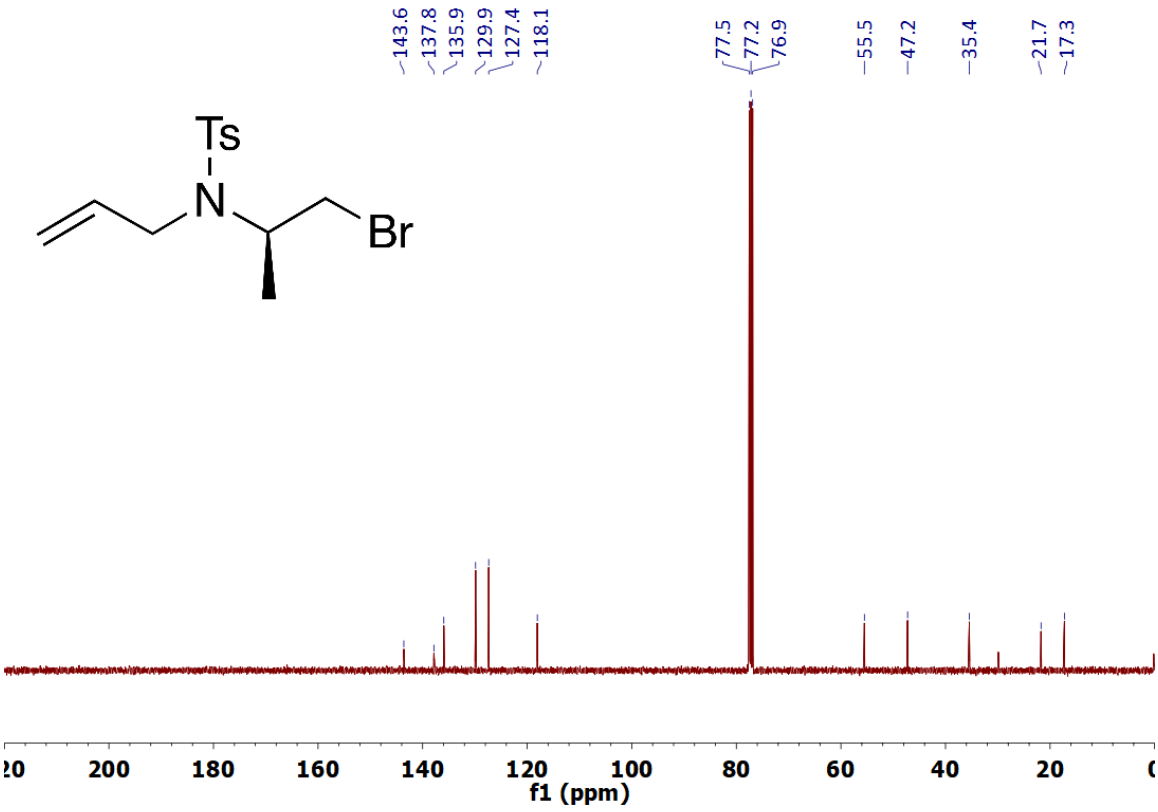
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77.2
76.9
55.5
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Ts
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Br
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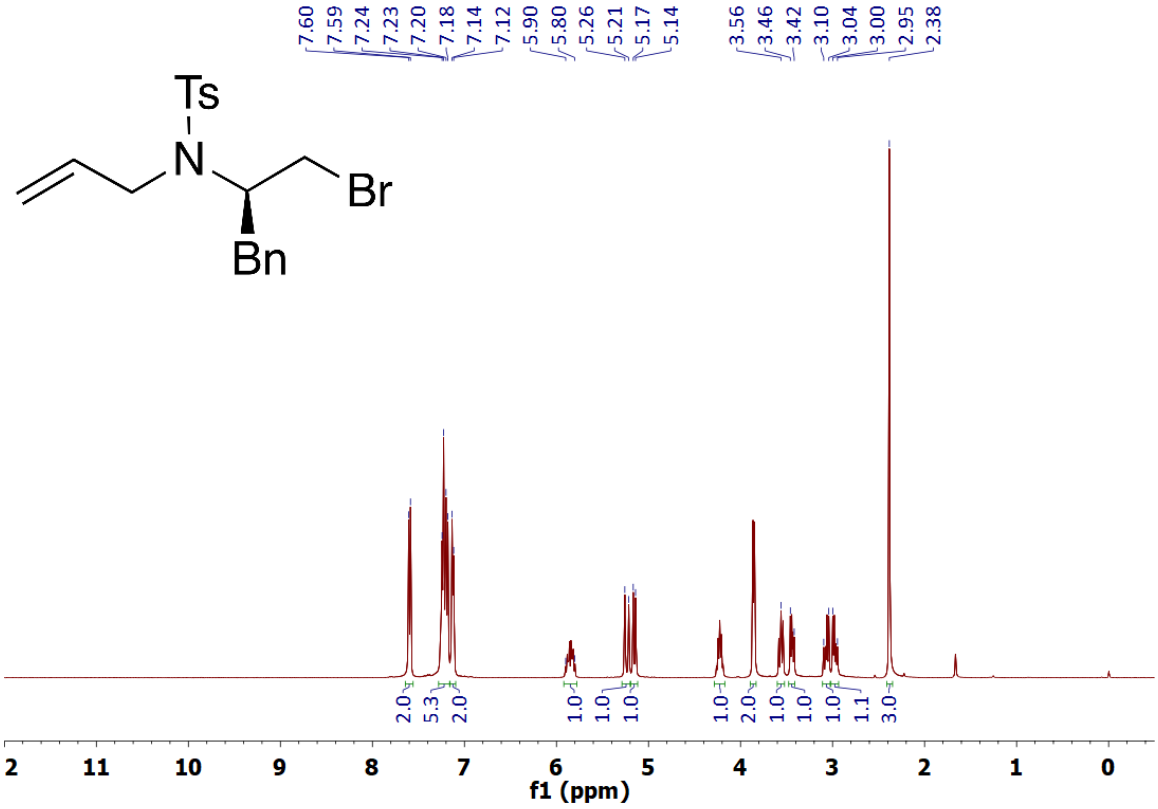
120
100
80
60
40
20
f1 (ppm)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5.14
3.56
3.46
3.42
3.10
3.04
3.00
2.95
2.38
Ts
N
Br
Bn
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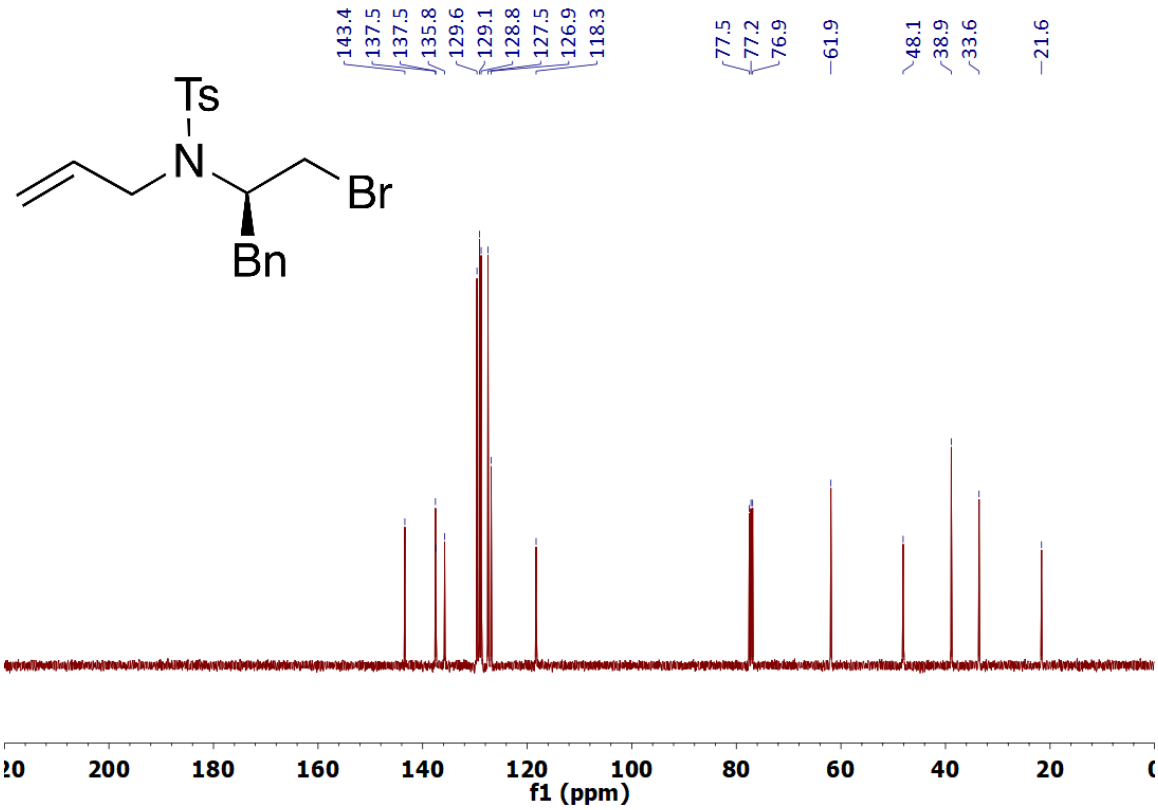
3.0
f1 (ppm)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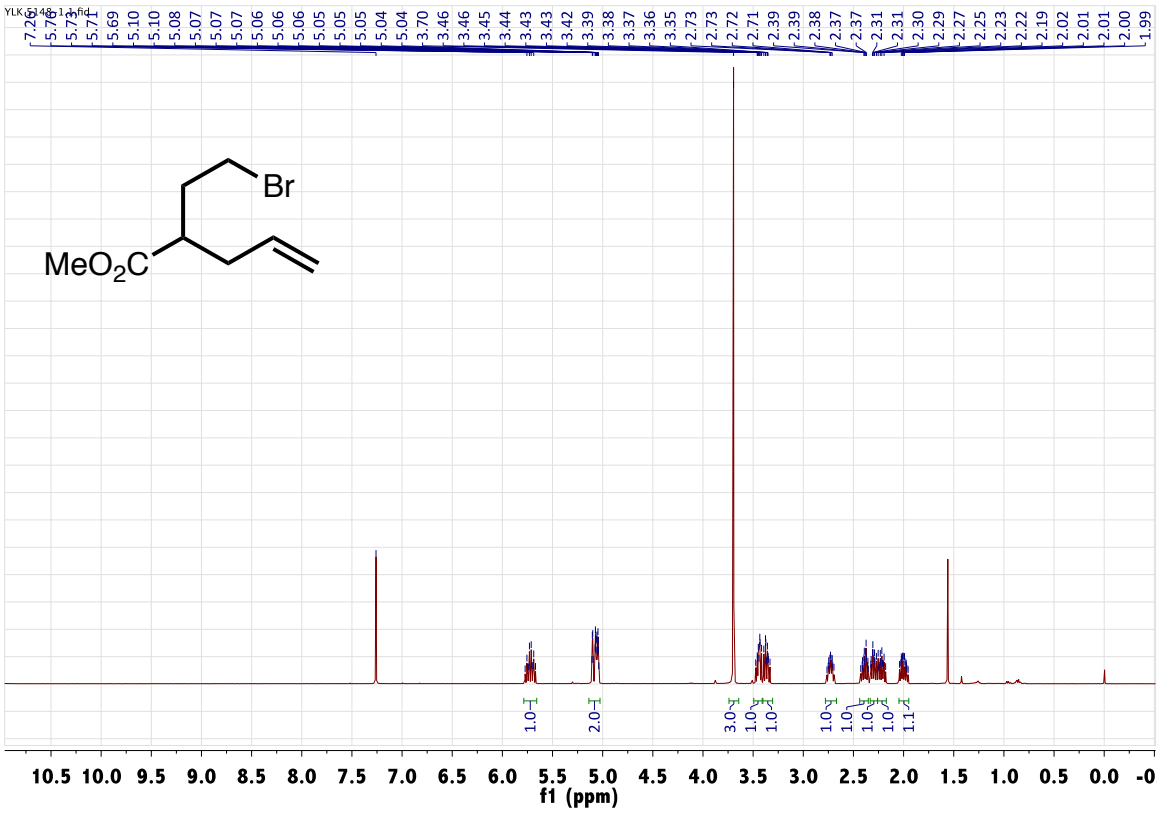
21.6
Ts
N
Br
Bn
f1 (ppm)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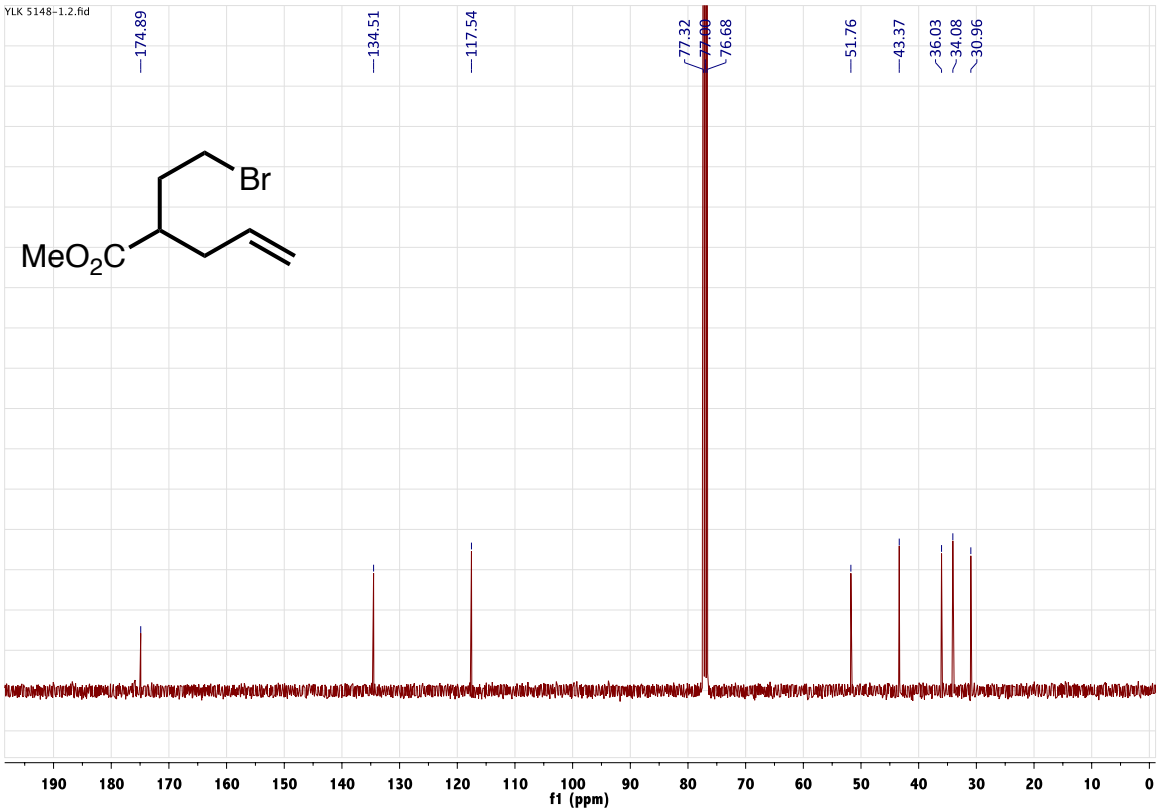
f1 (ppm)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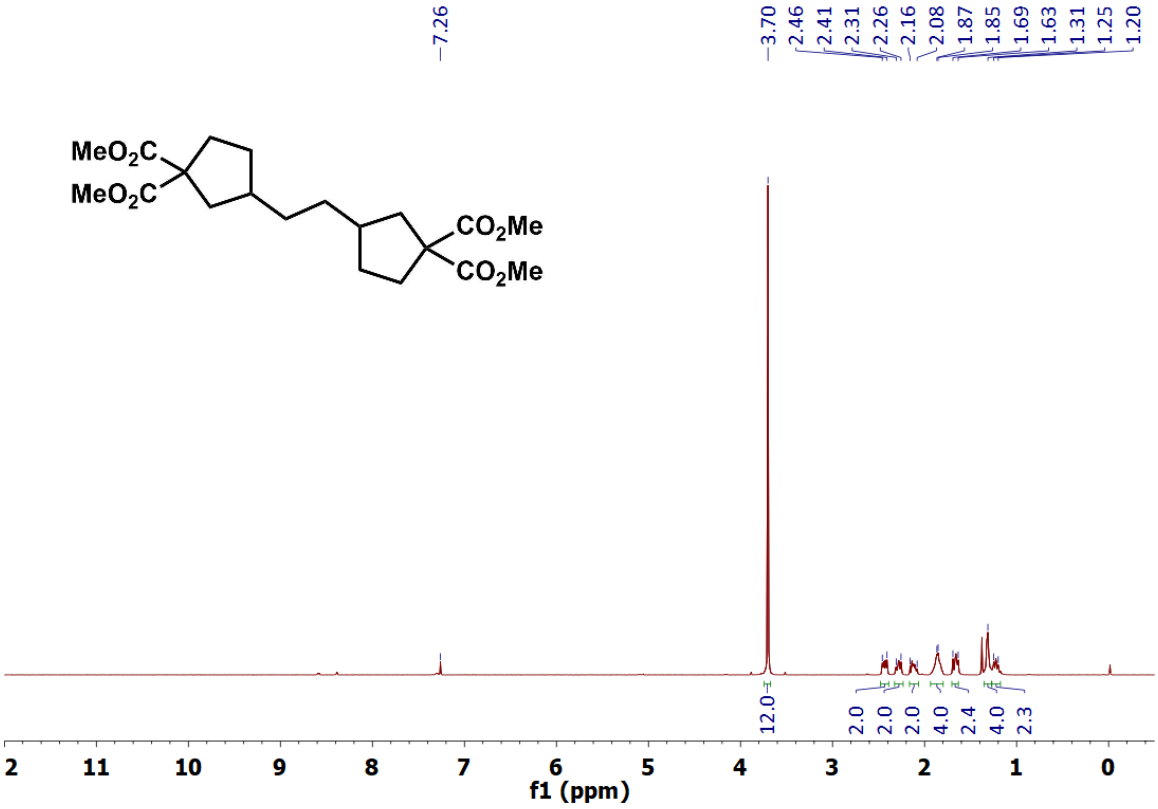
MeO2C
f1 (ppm)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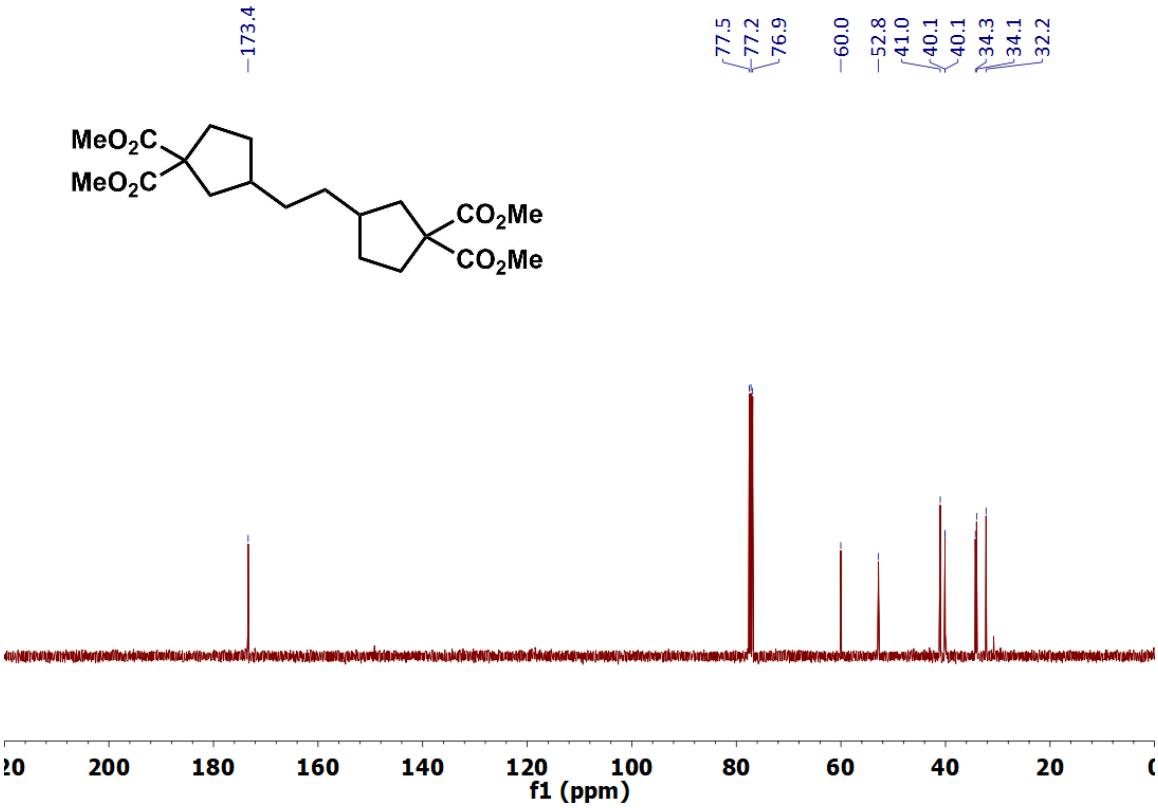
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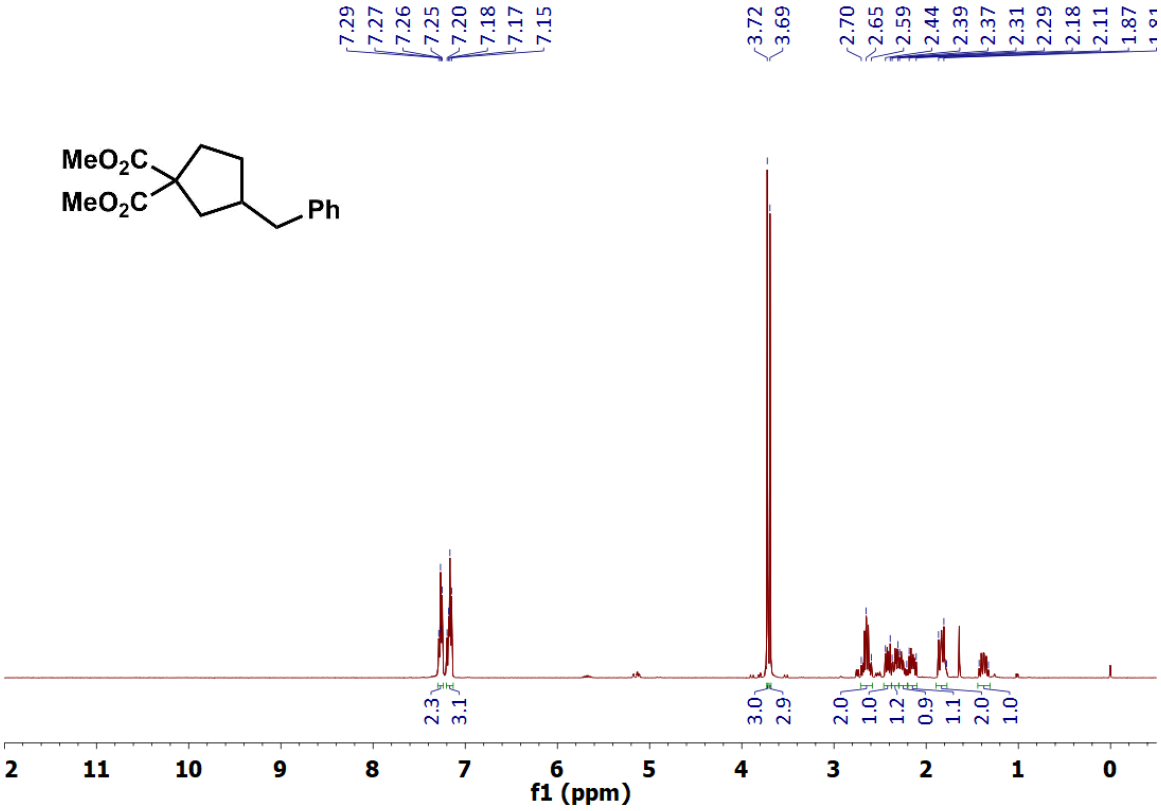
MeO2C
CO2Me
CO2Me
f1 (ppm)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2.18
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MeO2C
Ph
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1.0
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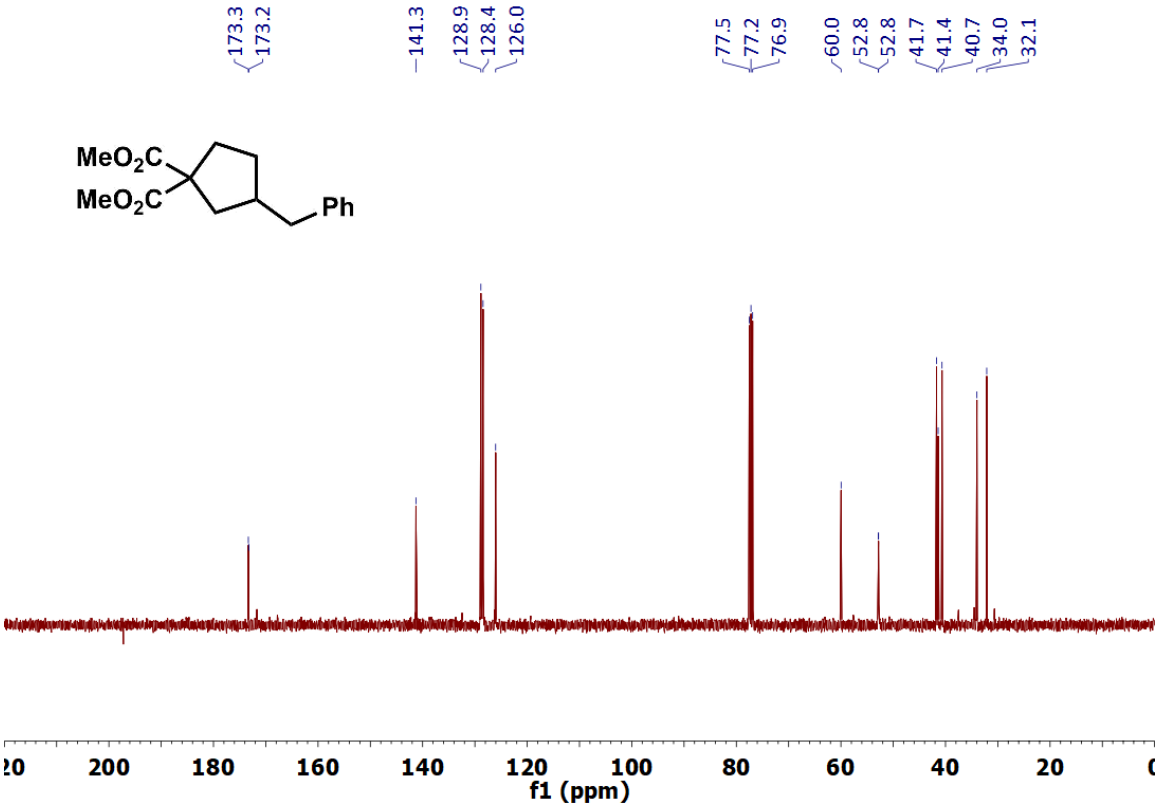
1.0
f1 (ppm)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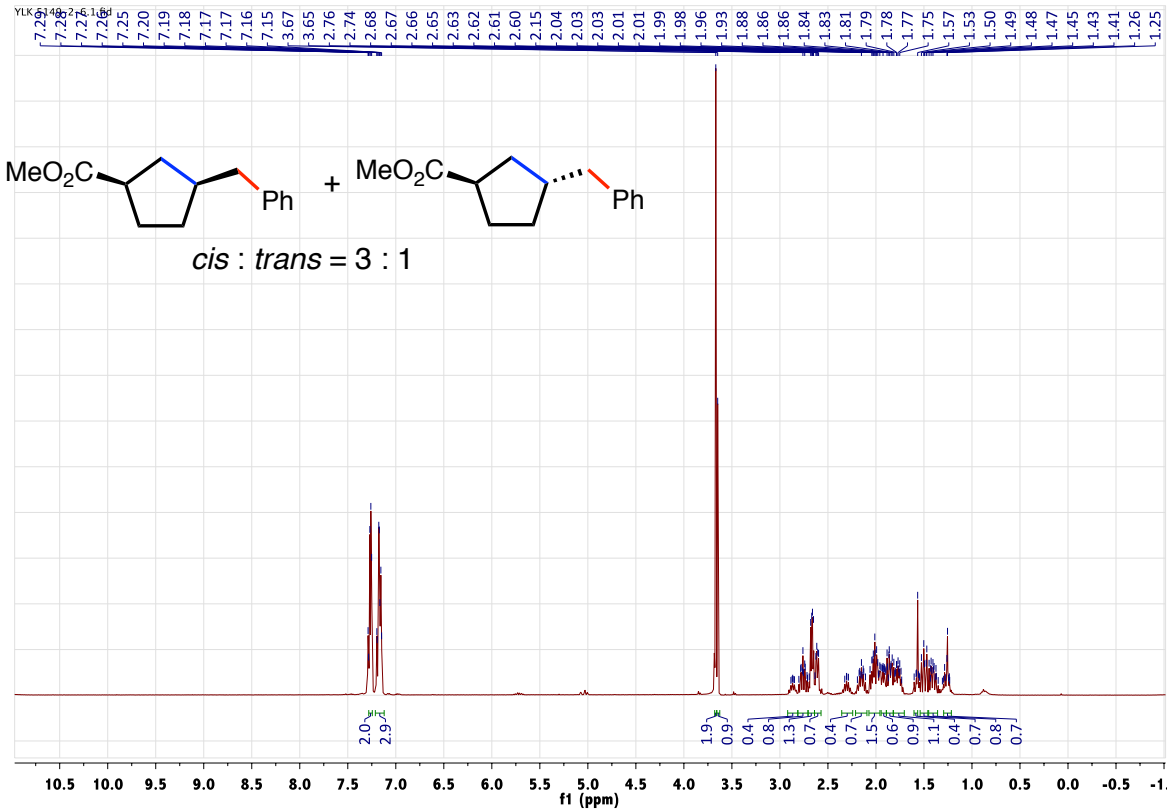
MeO2C
Ph
f1 (ppm)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Ph
+
MeO2C
Ph
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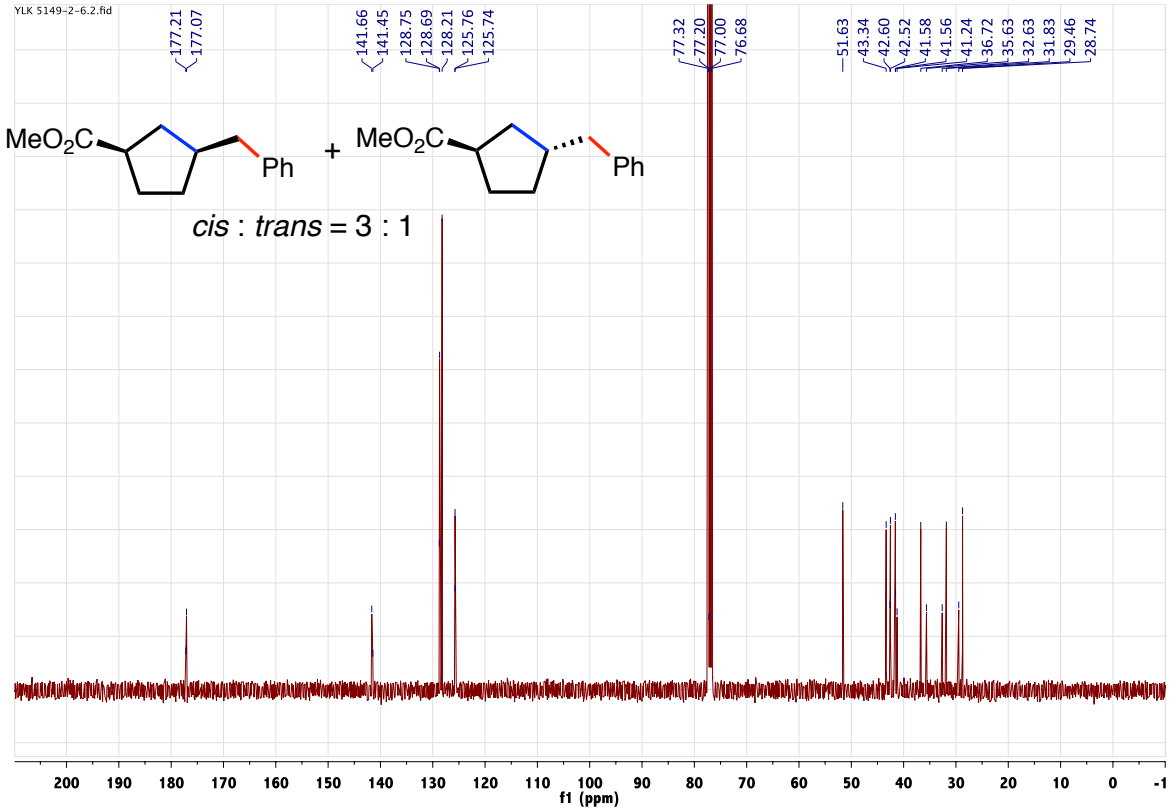
f1 (ppm)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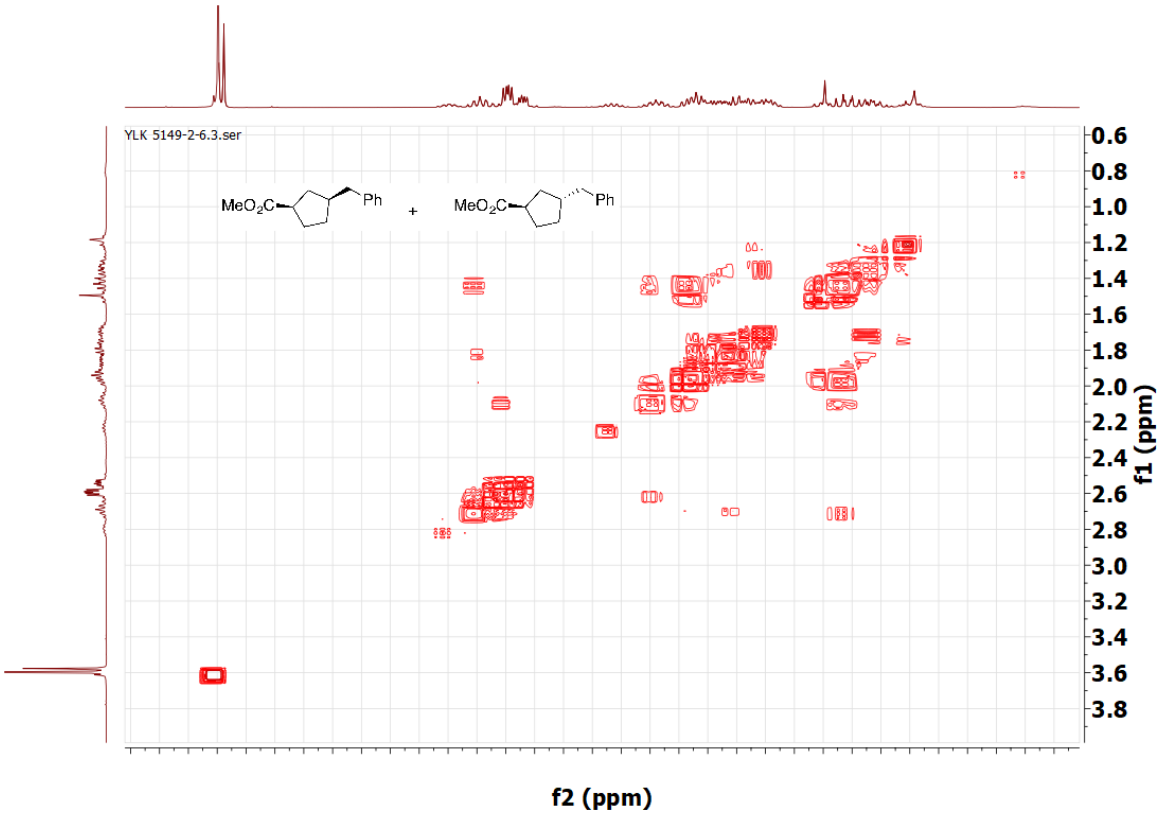
Ph
+
MeO2C
Ph
cis : trans = 3 : 1
f1 (ppm)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Ph
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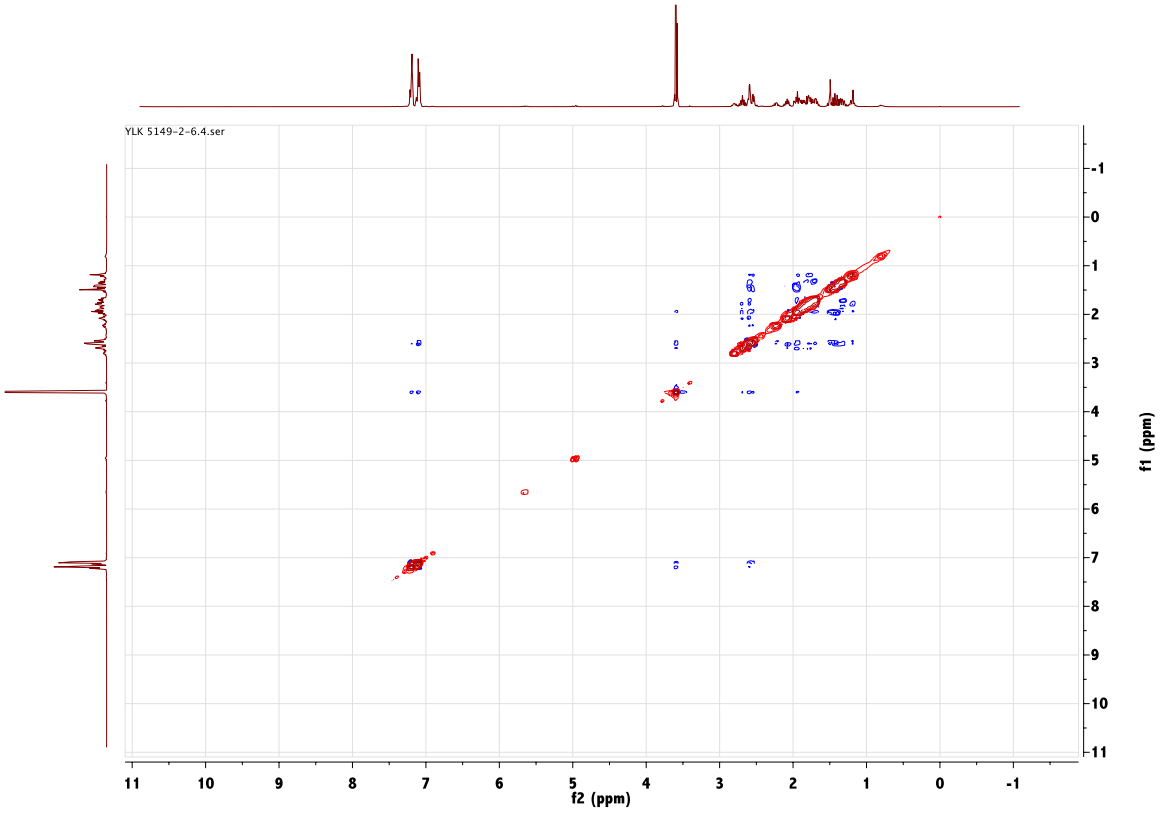
f2 (ppm)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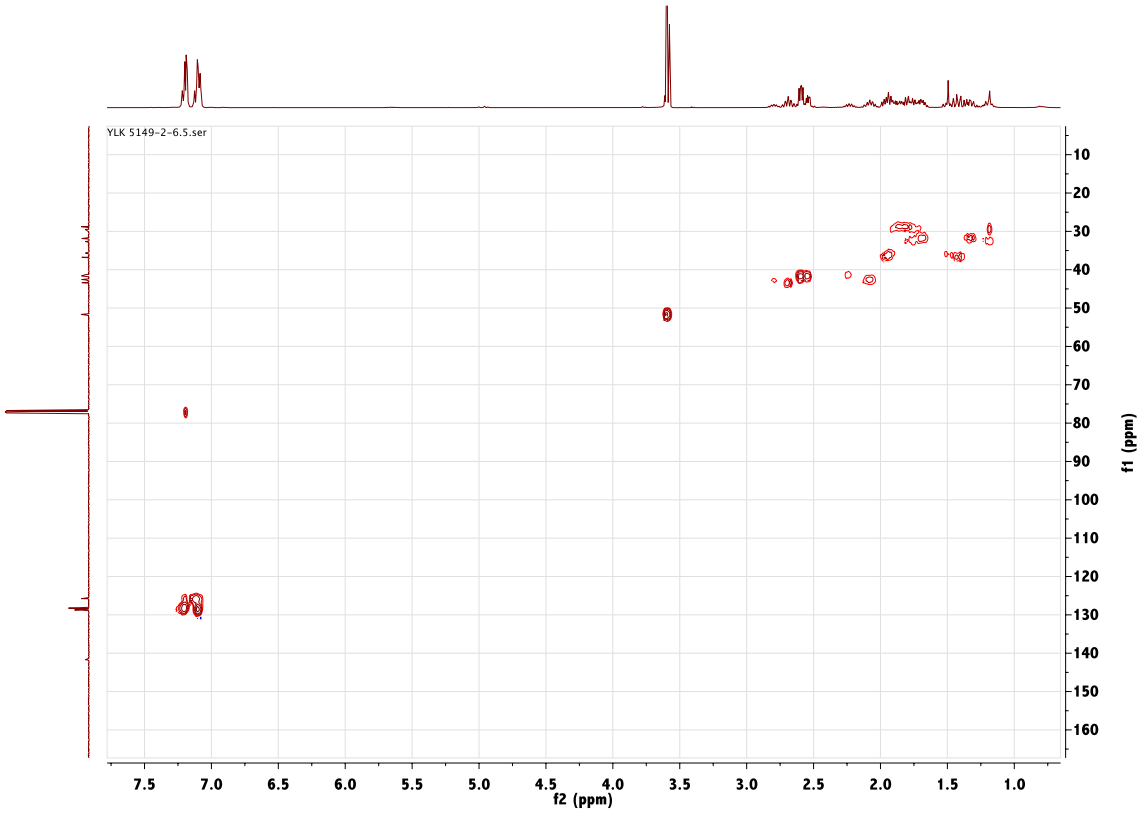
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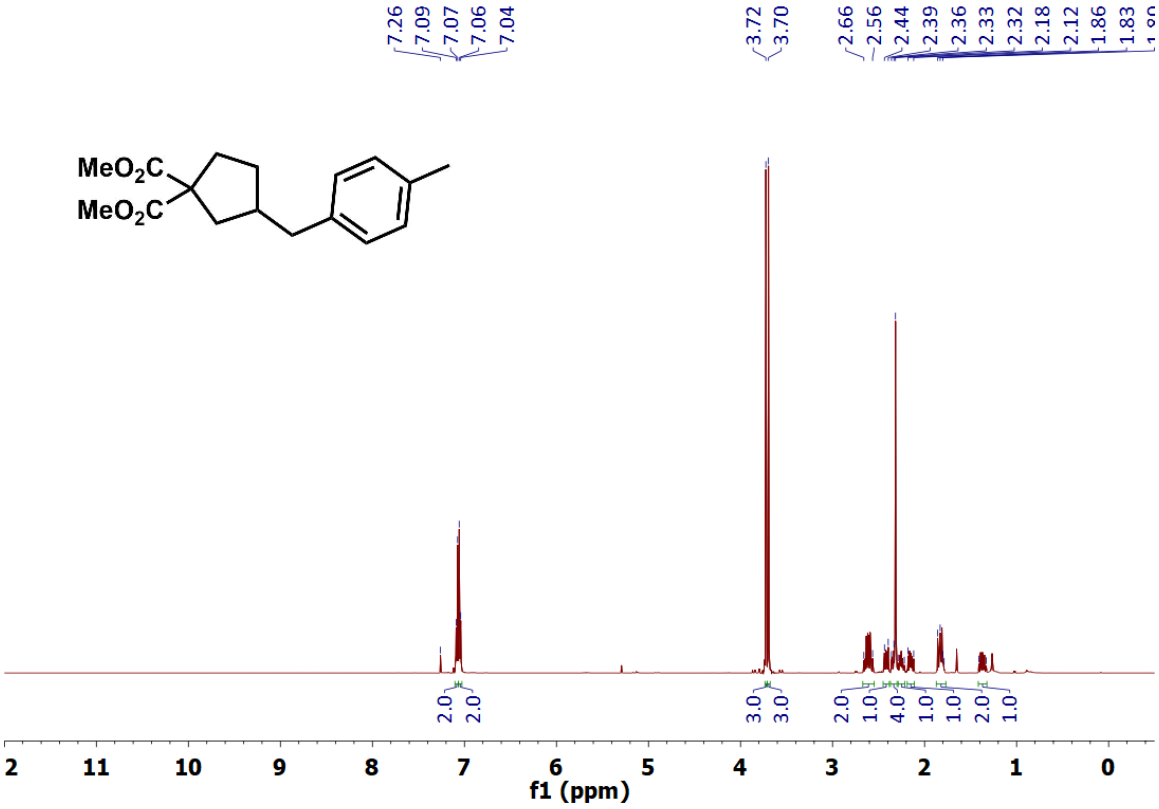
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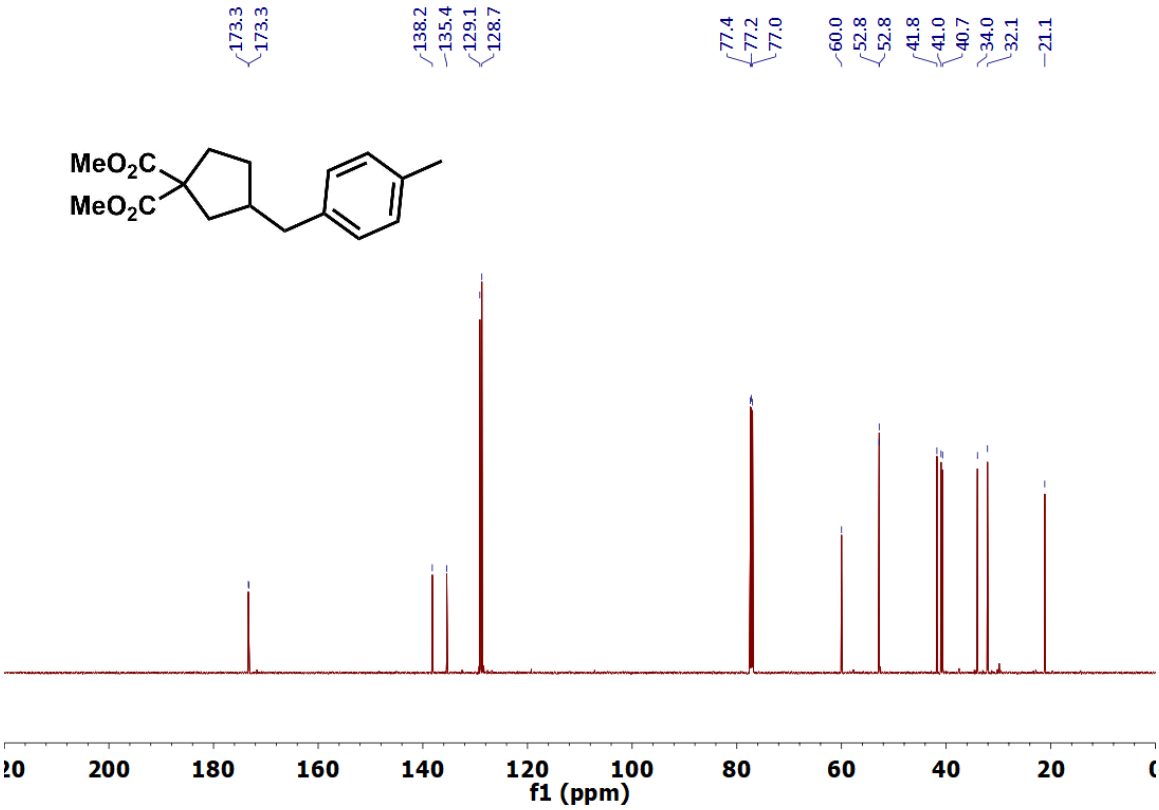
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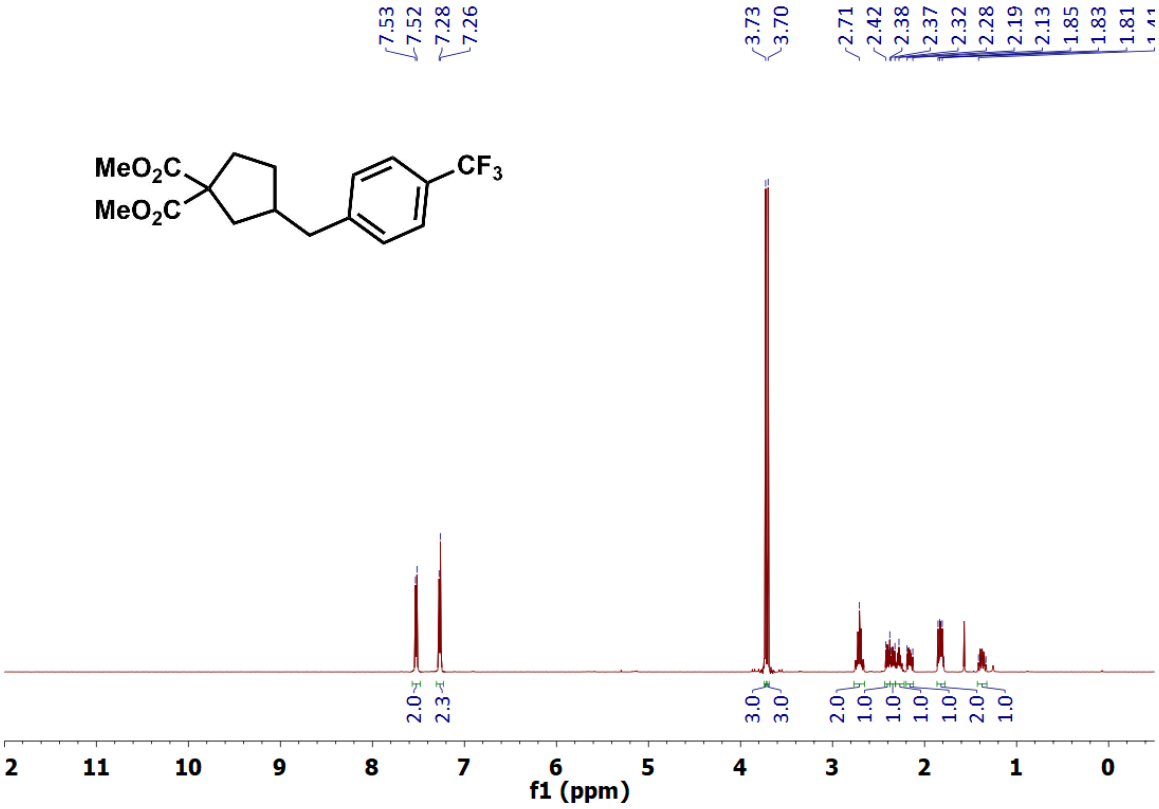
MeO2C
f1 (ppm)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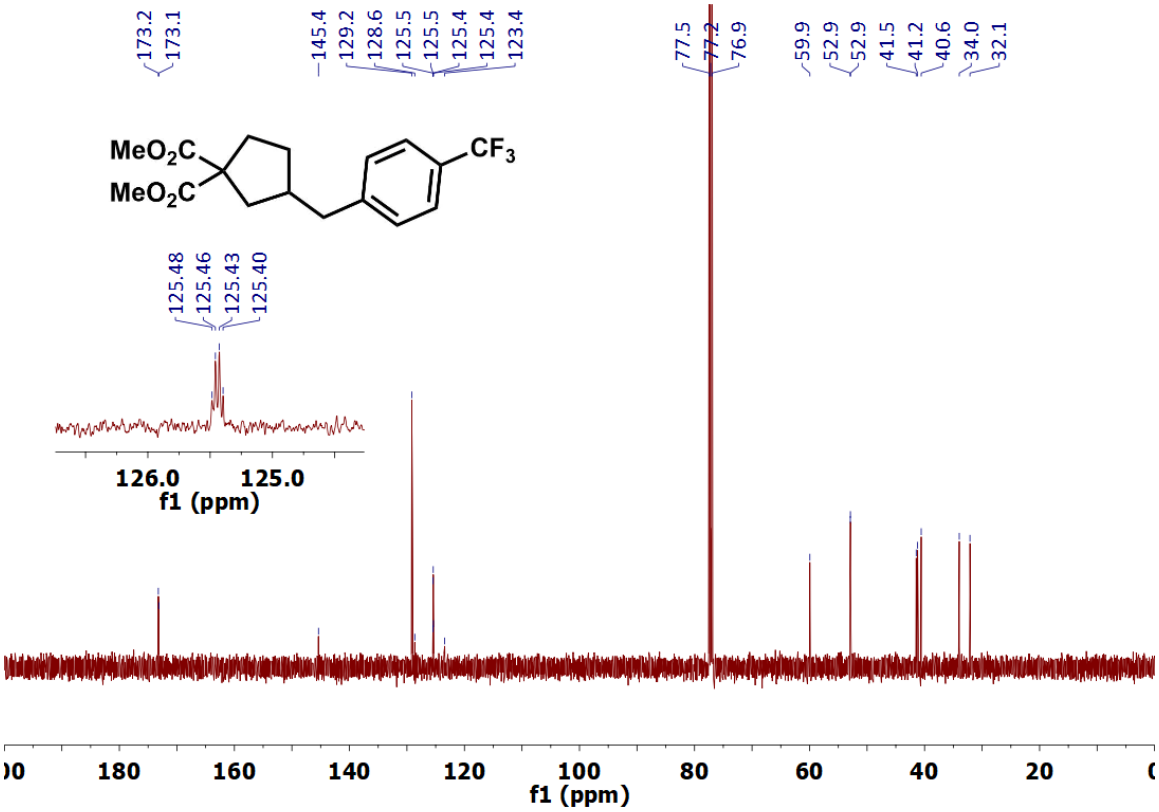
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52.9
41.5
41.2
40.6
34.0
32.1
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MeO2C
CF3
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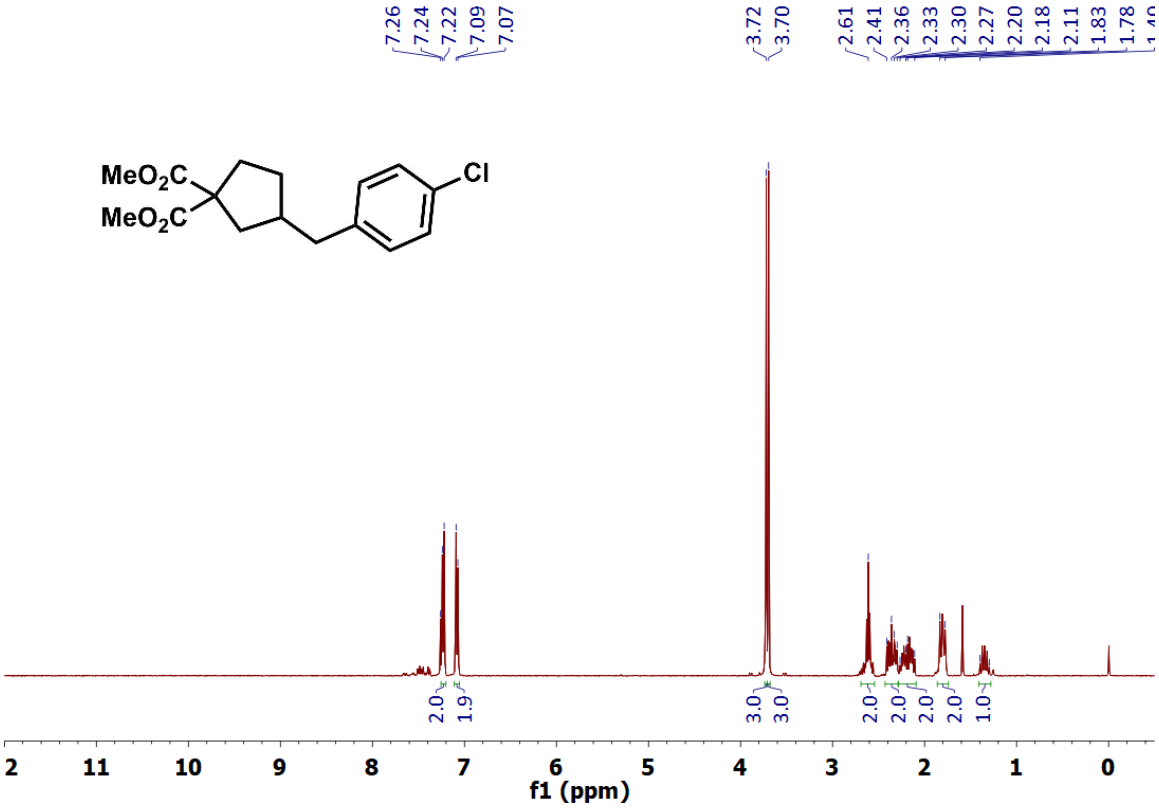
f1 (ppm)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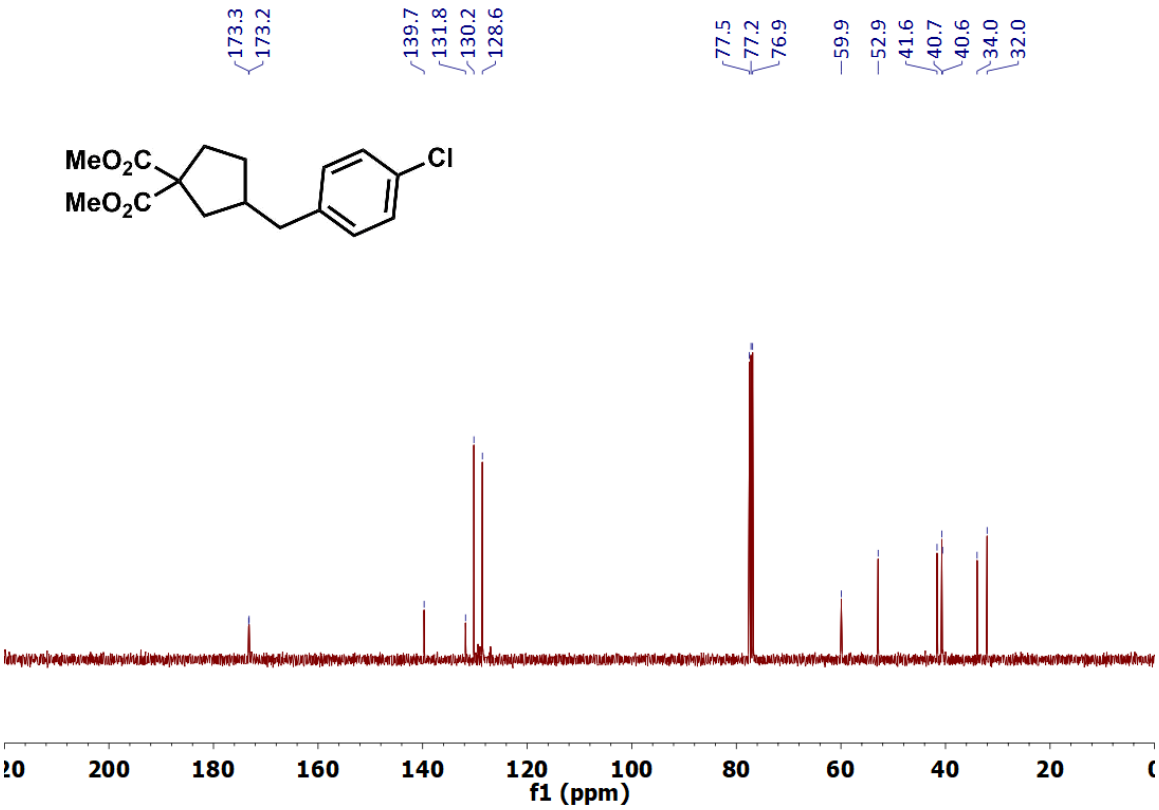
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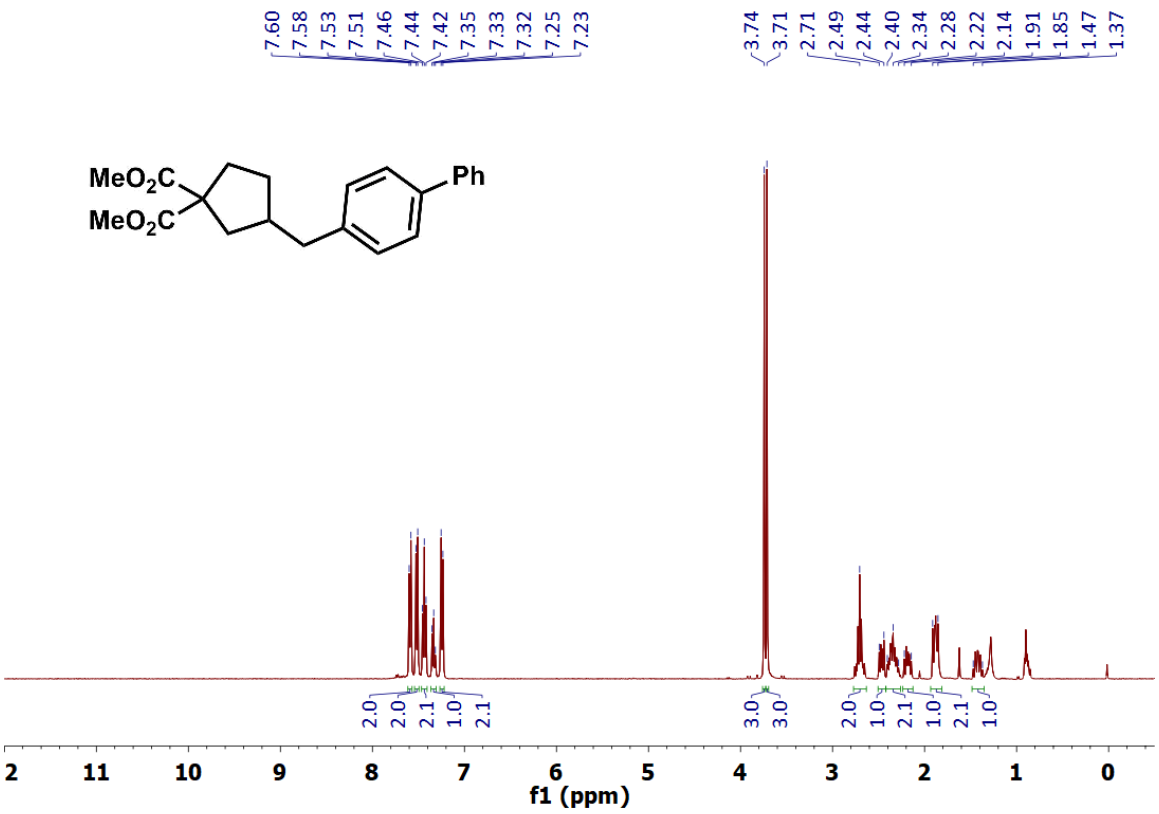
Cl
f1 (ppm)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MeO2C
Ph
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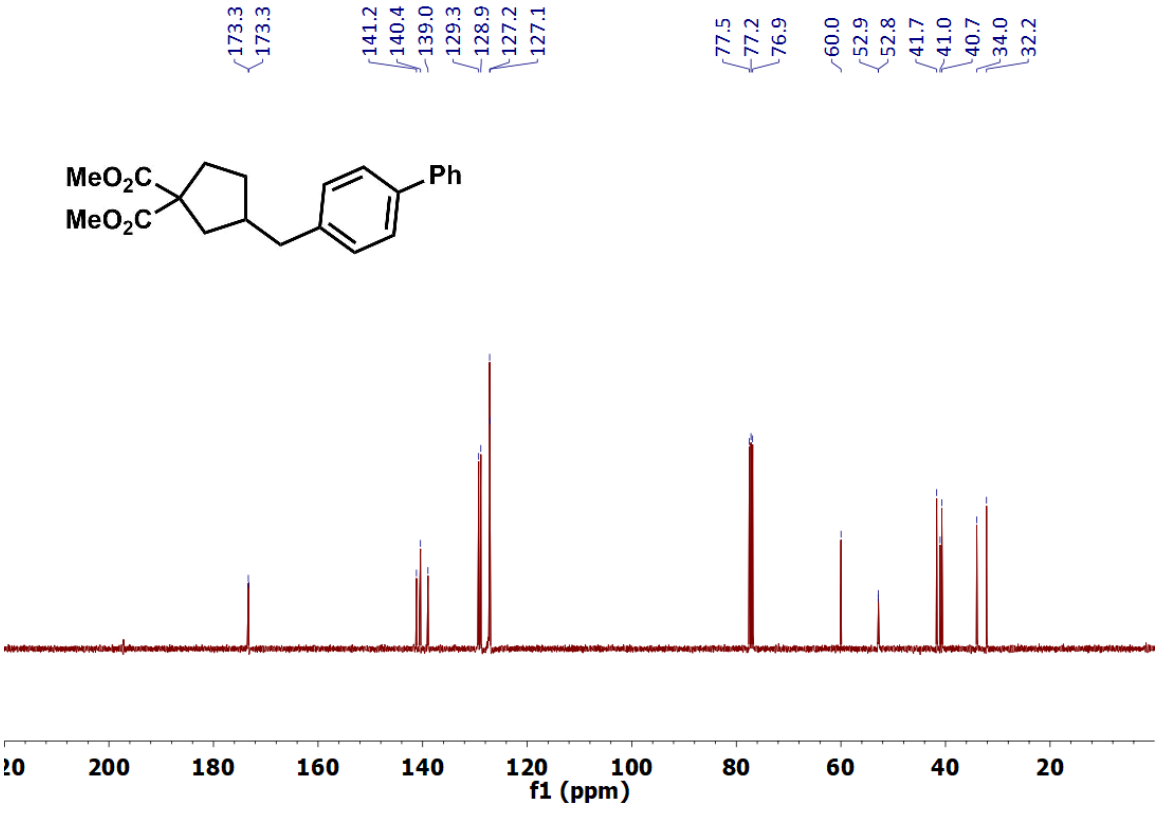
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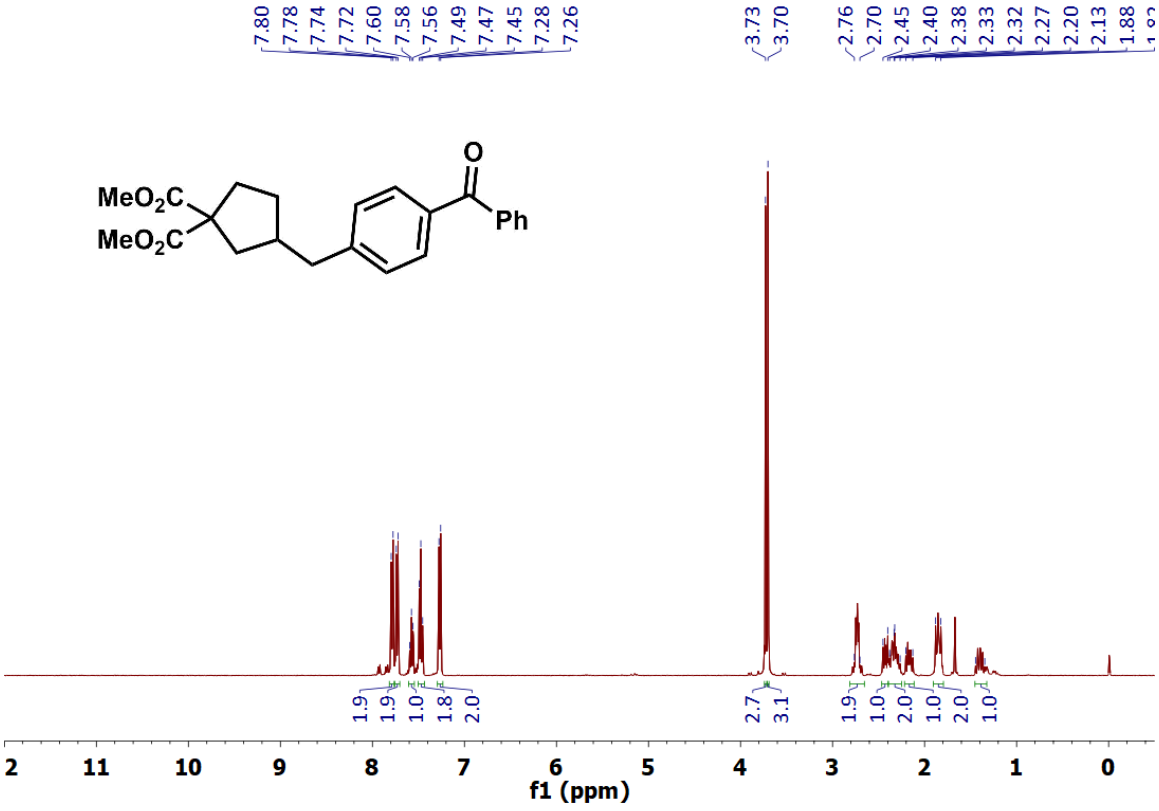
40.7
34.0
32.2
MeO2C
MeO2C
Ph
f1 (ppm)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MeO2C
MeO2C
O
Ph
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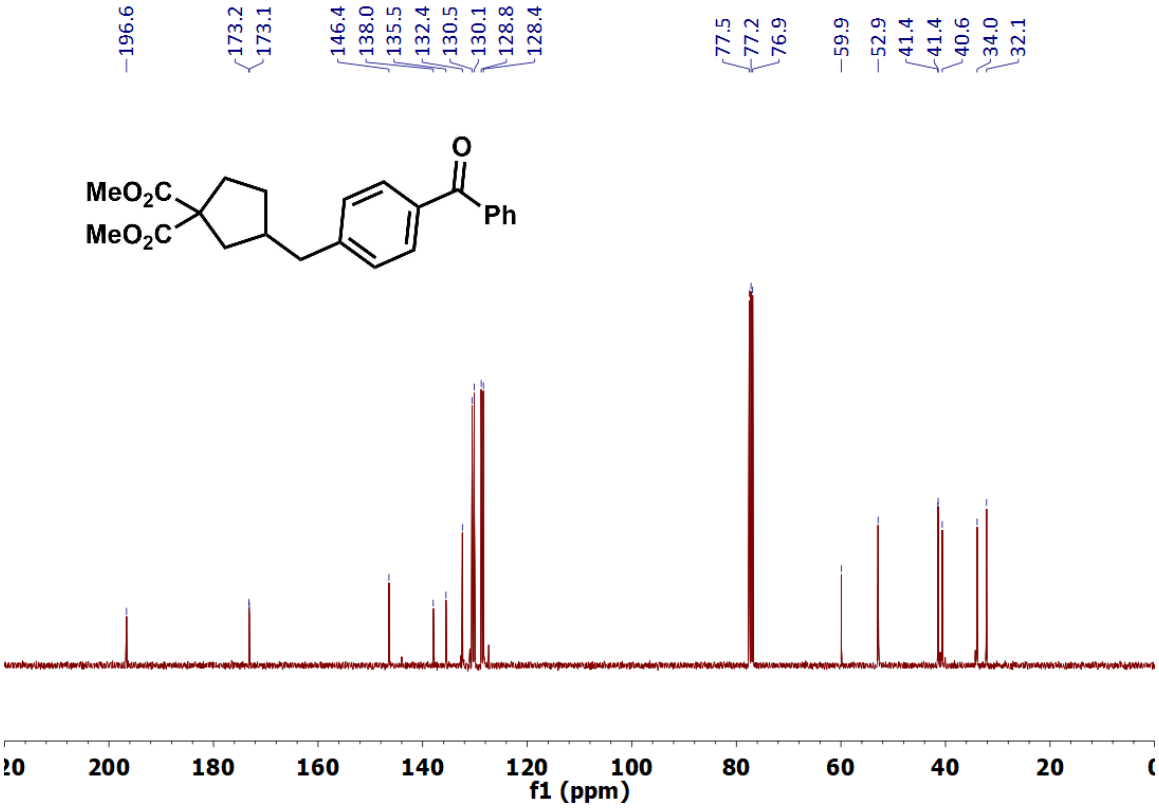
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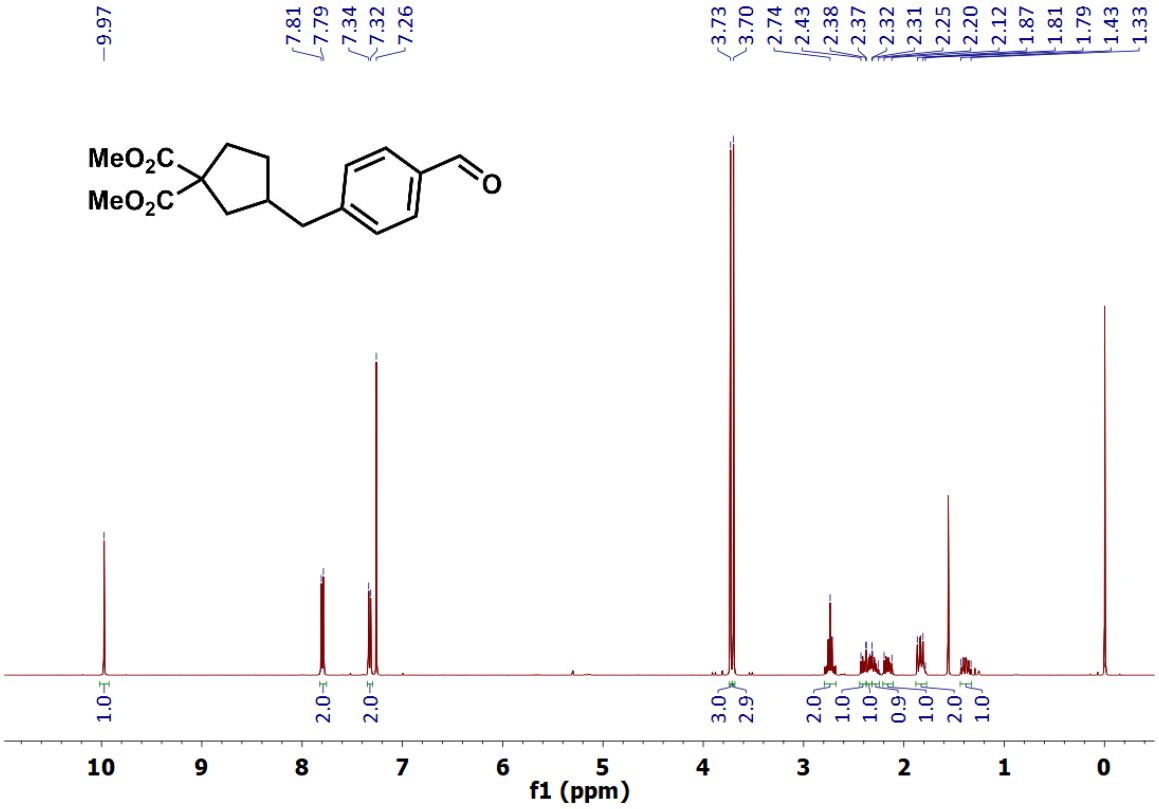
59.9
52.9
41.4
41.4
40.6
34.0
32.1
MeO2C
MeO2C
O
Ph
f1 (ppm)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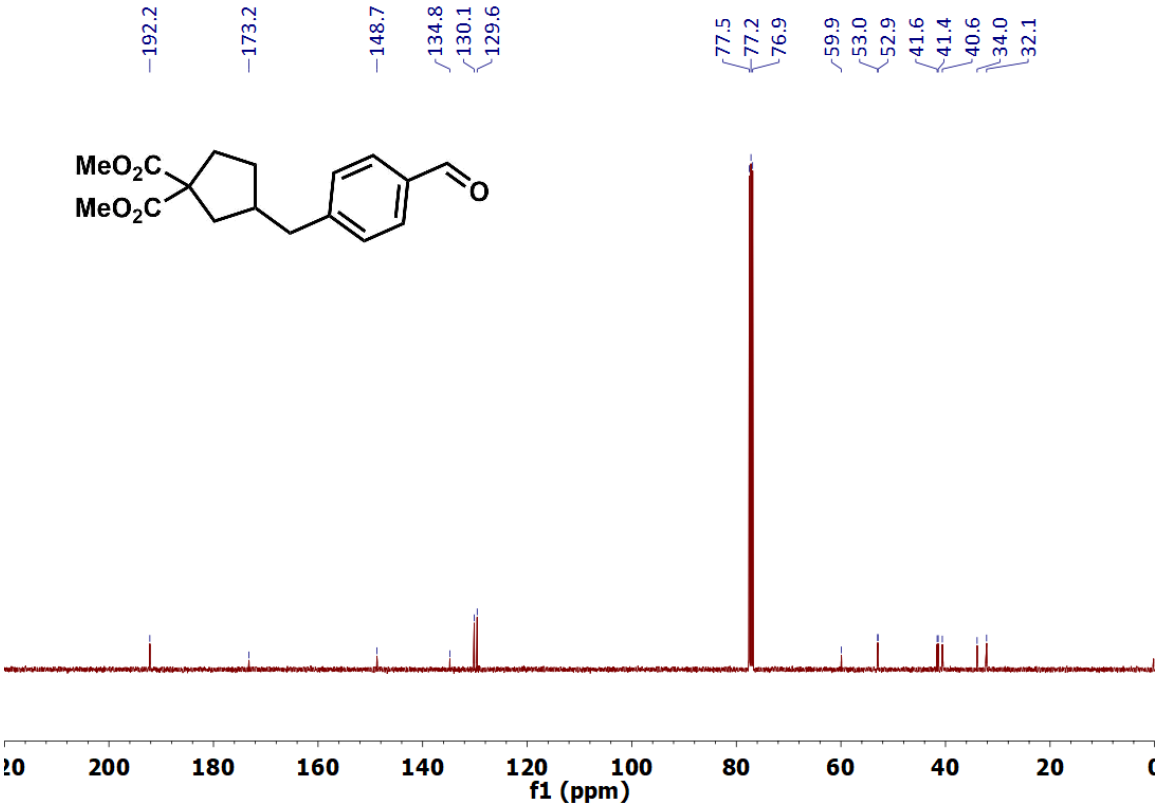
7.79
7.34
7.32
7.26
3.73
3.70
2.74
2.43
2.38
2.37
2.32
2.31
2.25
2.20
2.12
1.87
1.81
1.79
1.43
1.33
MeO2C
MeO2C
O
1.0
2.0
2.0
3.0
2.9
2.0
1.0
1.0
0.9
1.0
2.0
1.0
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)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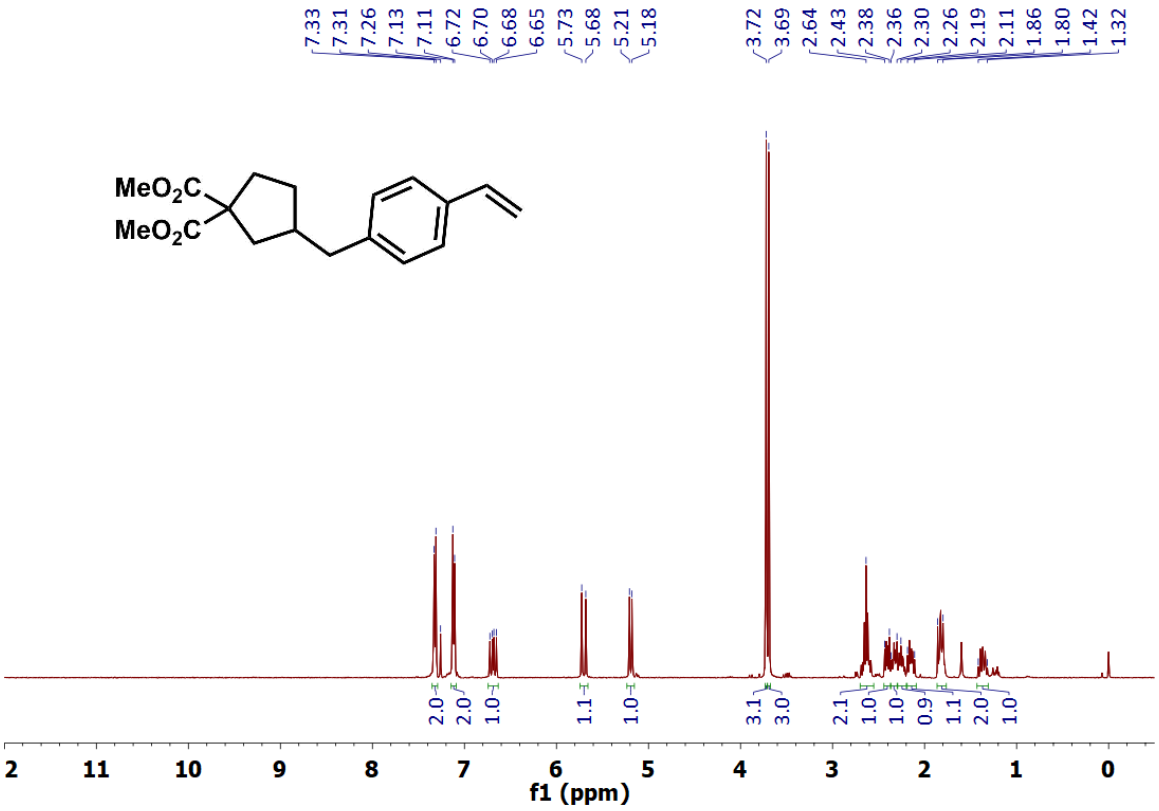
129.6
77.5
77.2
76.9
59.9
53.0
52.9
41.6
41.4
40.6
34.0
32.1
MeO2C
MeO2C
O
200
180
160
140
120
100
80
60
40
20
f1 (ppm)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6.68
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5.73
5.68
5.21
5.18
3.72
3.69
2.64
2.43
2.38
2.36
2.30
2.26
2.19
2.11
1.86
1.80
1.42
1.32
MeO2C
MeO2C
2.0
2.0
1.0
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2.1
1.0
1.0
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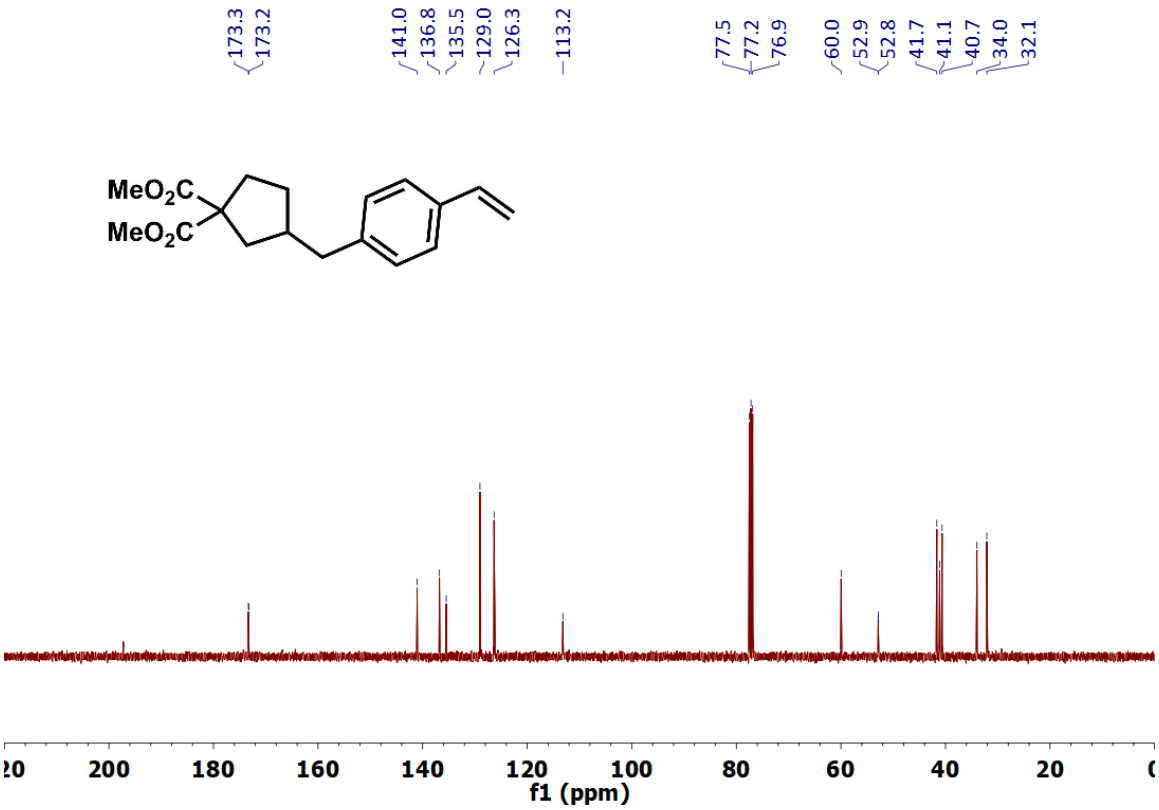
1.1
2.0
1.0
f1 (ppm)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173.2
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135.5
129.0
126.3
113.2
77.5
77.2
76.9
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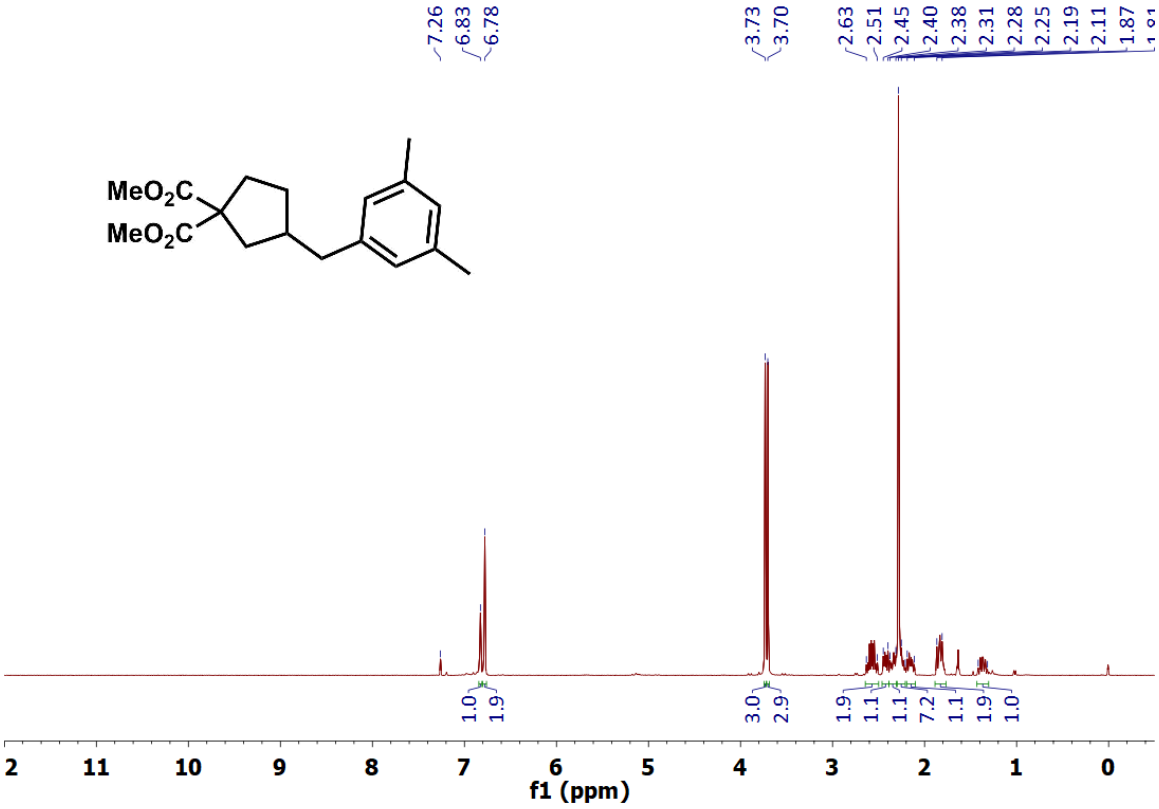
52.9
52.8
41.7
41.1
40.7
34.0
32.1
MeO2C
MeO2C
f1 (ppm)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6.78
3.73
3.70
2.63
2.51
2.45
2.40
2.38
2.31
2.28
2.25
2.19
2.11
1.87
MeO2C
MeO2C
1.0
1.9
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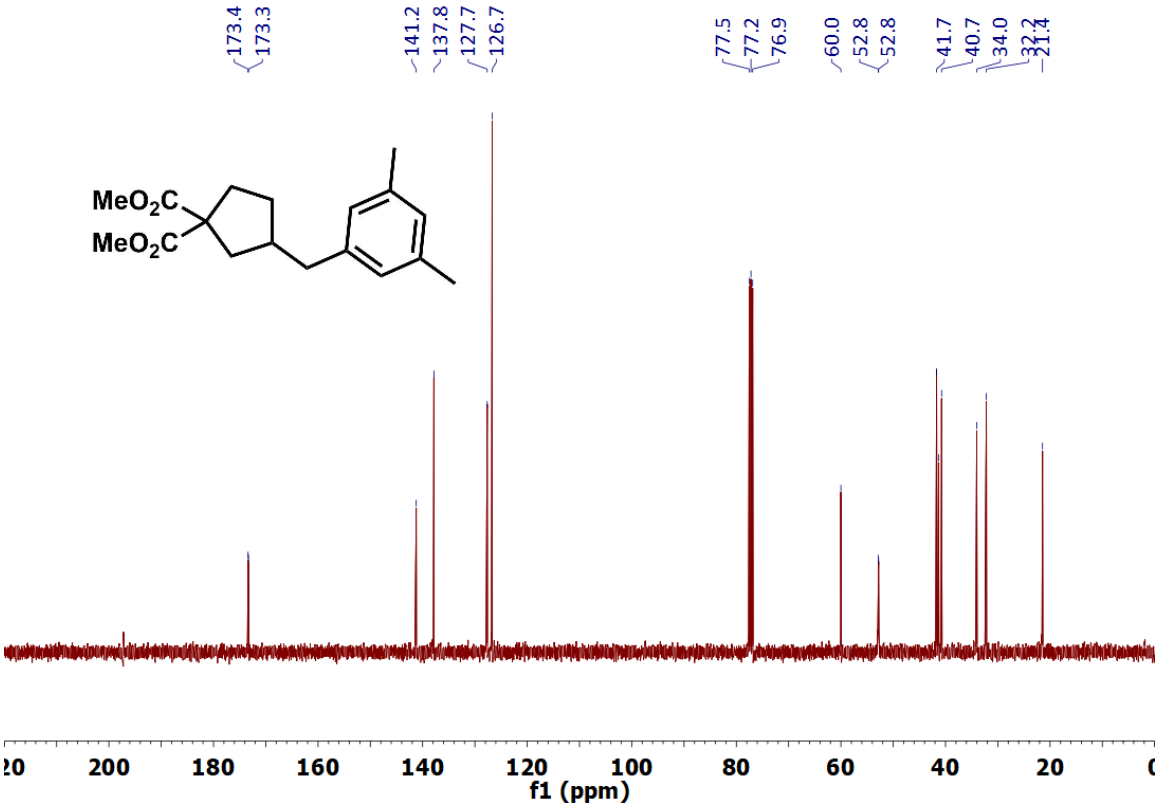
2.9
1.9
1.1
1.1
7.2
1.1
1.9
1.0
f1 (ppm)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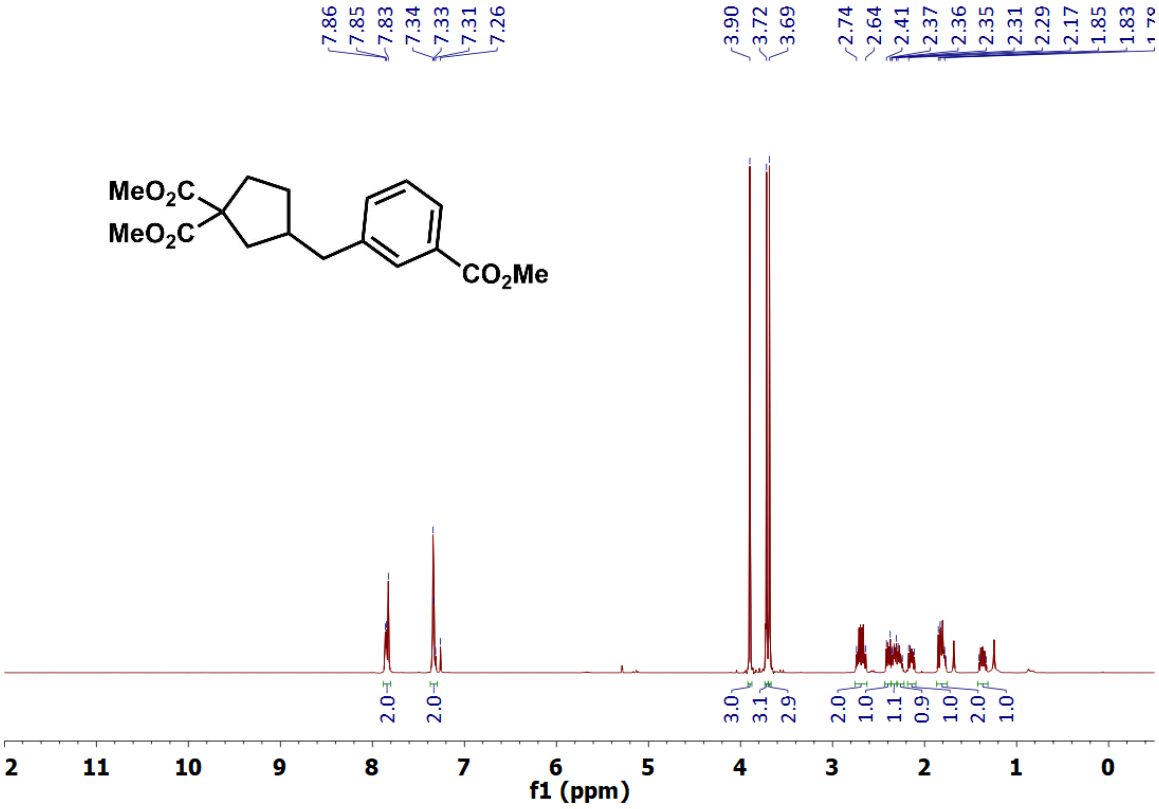
141.2
137.8
127.7
126.7
77.5
77.2
76.9
60.0
52.8
52.8
41.7
40.7
34.0
MeO2C
MeO2C
f1 (ppm)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7.83
7.34
7.33
7.31
7.26
3.90
3.72
3.69
2.74
2.64
2.41
2.37
2.36
2.35
2.31
2.29
2.17
1.85
1.83
MeO2C
MeO2C
CO2Me
2.0
2.0
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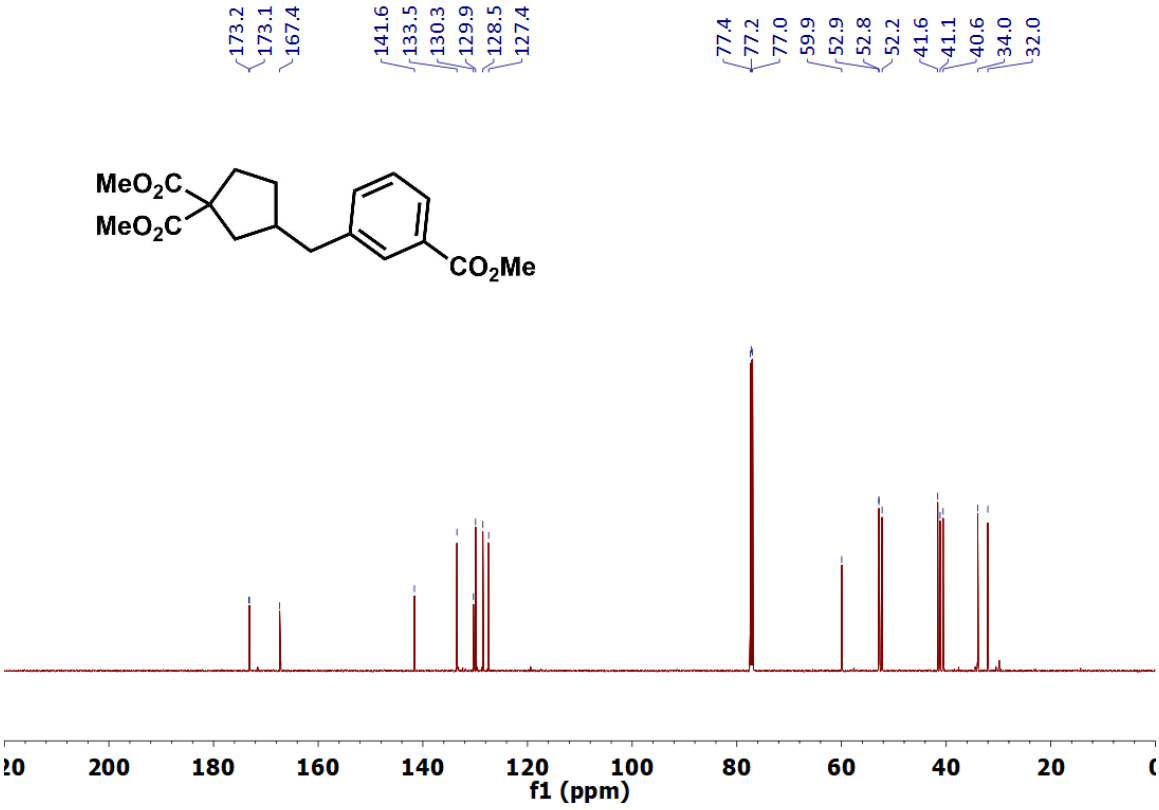
2.9
2.0
1.0
1.1
0.9
1.0
2.0
1.0
f1 (ppm)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173.1
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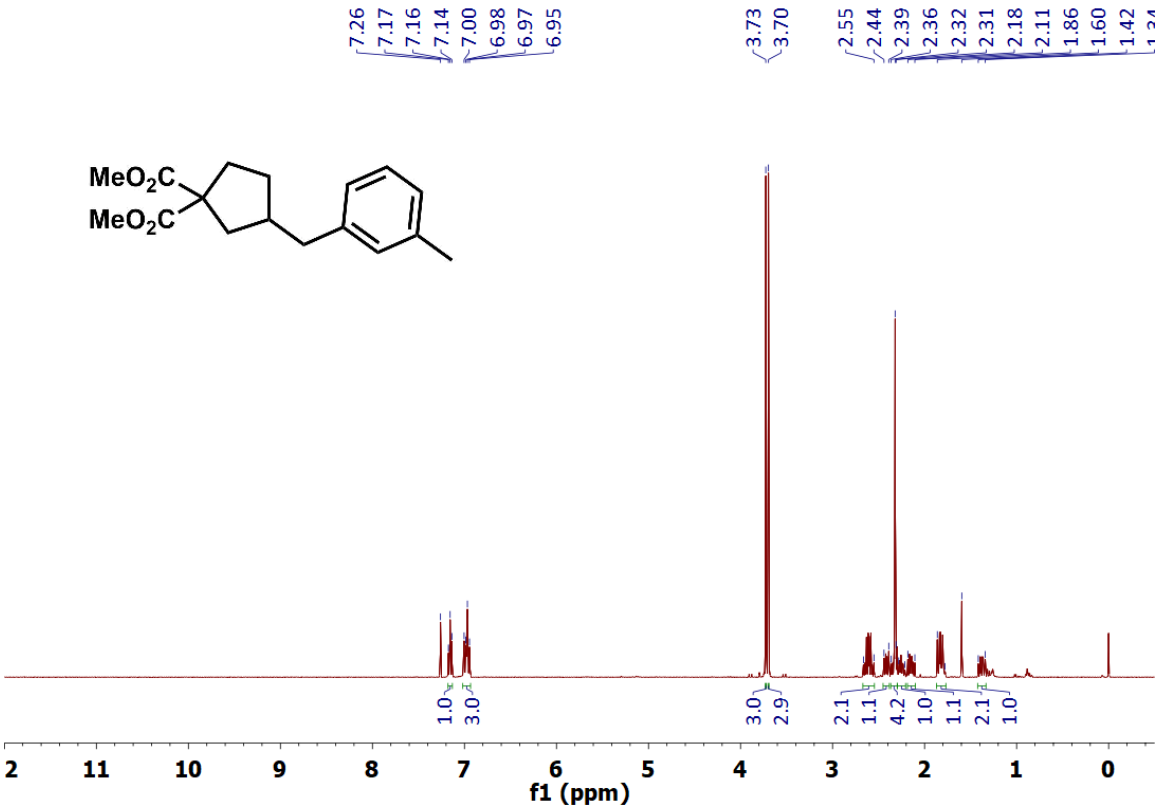
133.5
130.3
129.9
128.5
127.4
77.4
77.2
77.0
59.9
52.9
52.8
52.2
41.6
41.1
40.6
34.0
32.0
MeO2C
MeO2C
CO2Me
f1 (ppm)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6.95
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3.70
2.55
2.44
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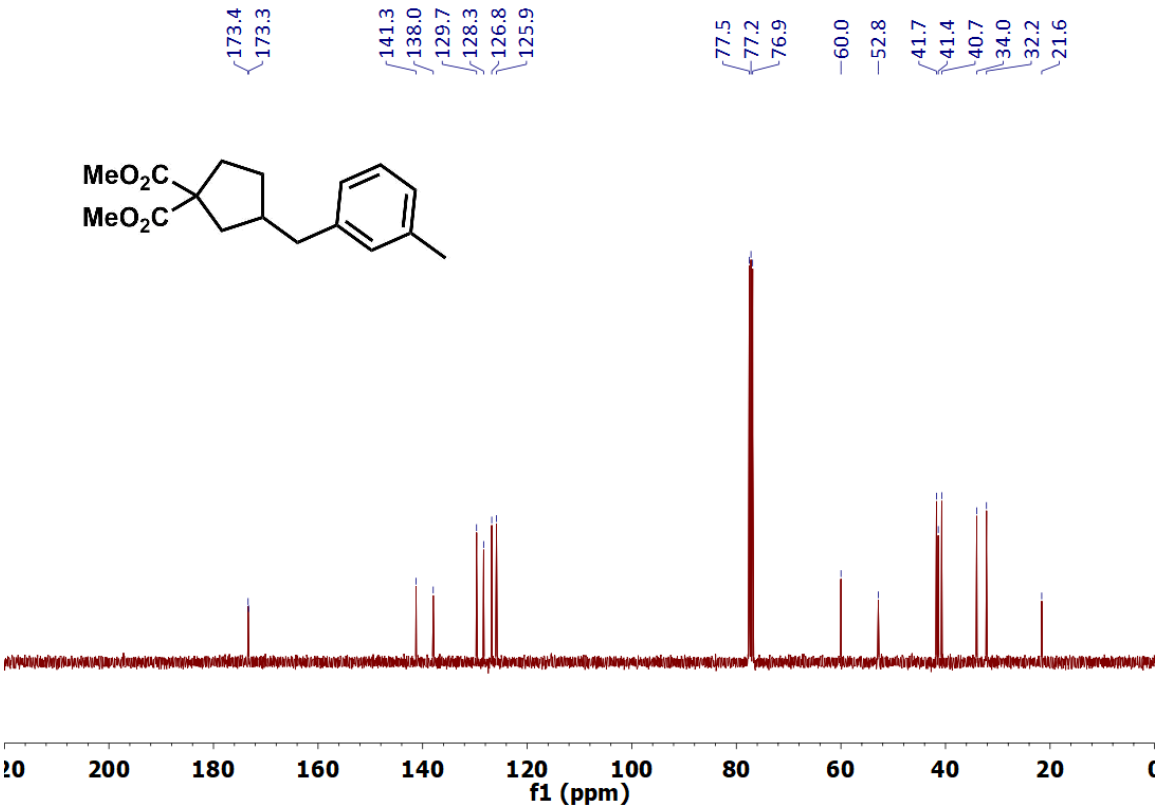
2.36
2.32
2.31
2.18
2.11
1.86
1.60
1.42
MeO2C
MeO2C
1.0
3.0
3.0
2.9
2.1
1.1
4.2
1.0
1.1
2.1
1.0
f1 (ppm)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173.3
141.3
138.0
129.7
128.3
126.8
125.9
77.5
77.2
76.9
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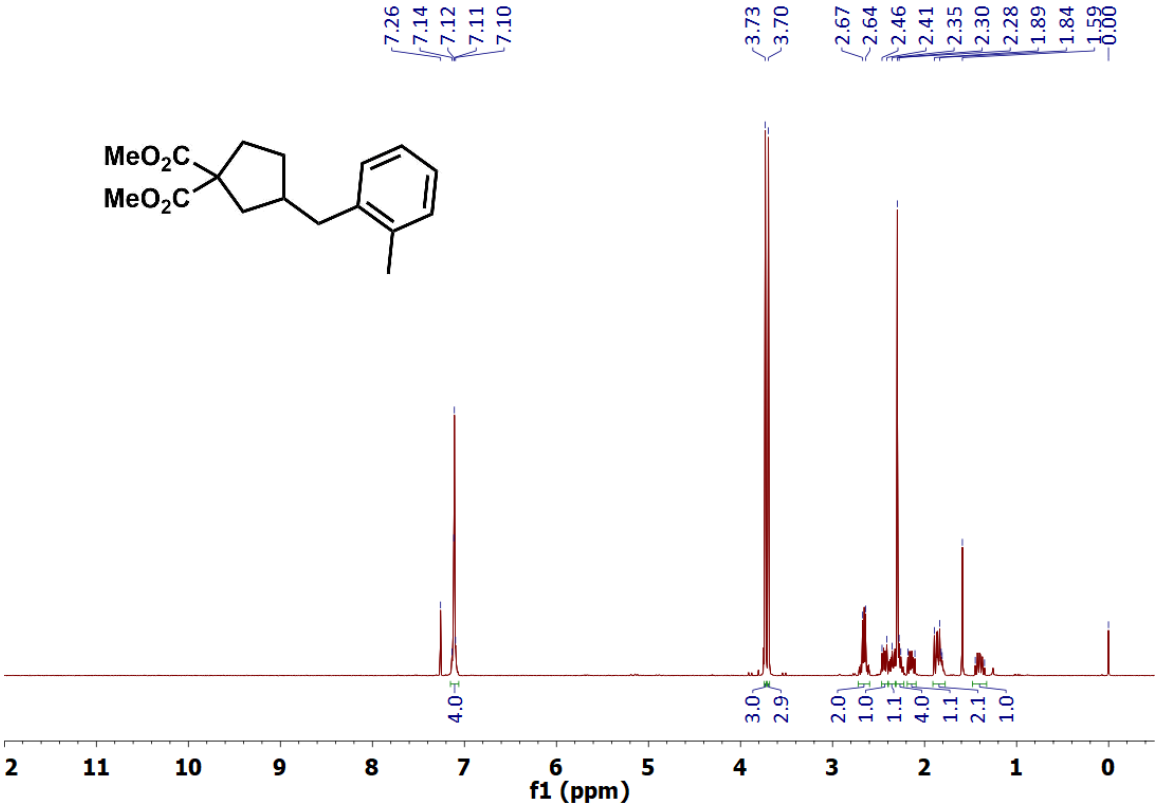
52.8
41.7
41.4
40.7
34.0
32.2
21.6
MeO2C
MeO2C
f1 (ppm)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7.14
7.12
7.11
7.10
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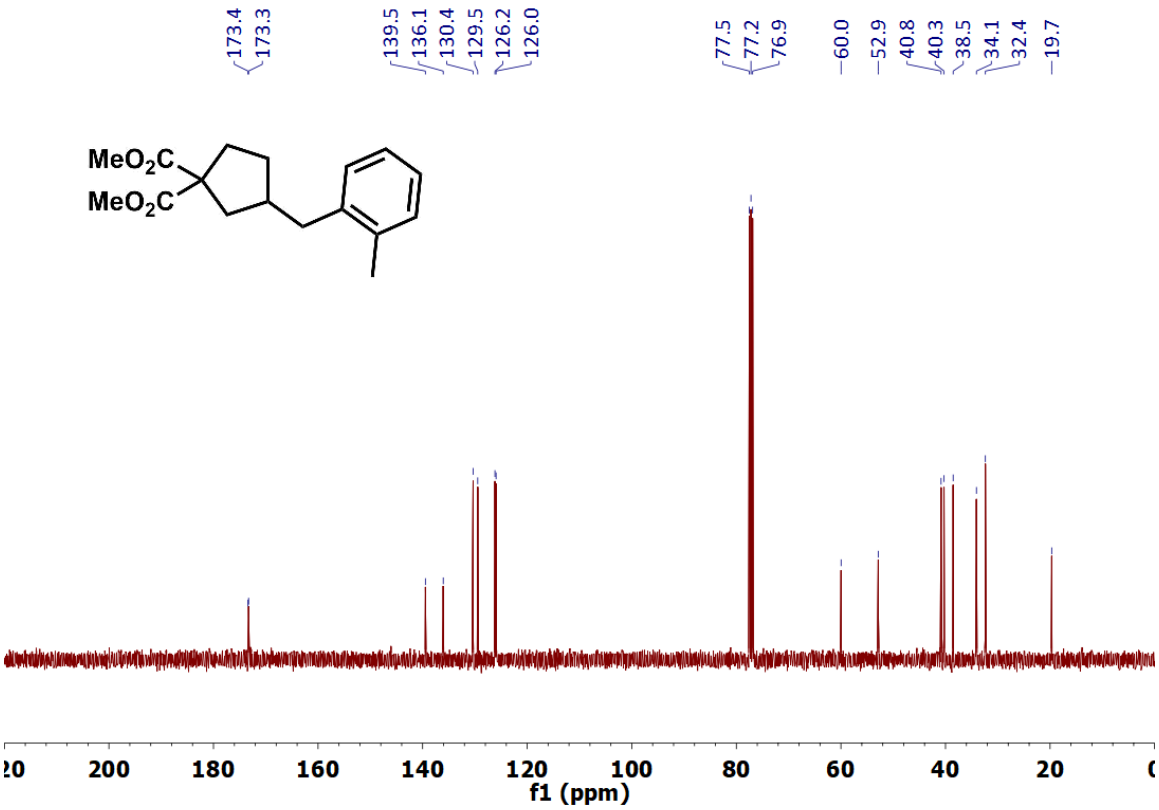
2.67
2.64
2.46
2.41
2.35
2.30
2.28
1.89
1.84
1.59
0.00
MeO2C
MeO2C
4.0
3.0
2.9
2.0
1.0
1.1
4.0
1.1
2.1
1.0
f1 (ppm)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173.3
139.5
136.1
130.4
129.5
126.2
126.0
77.5
77.2
76.9
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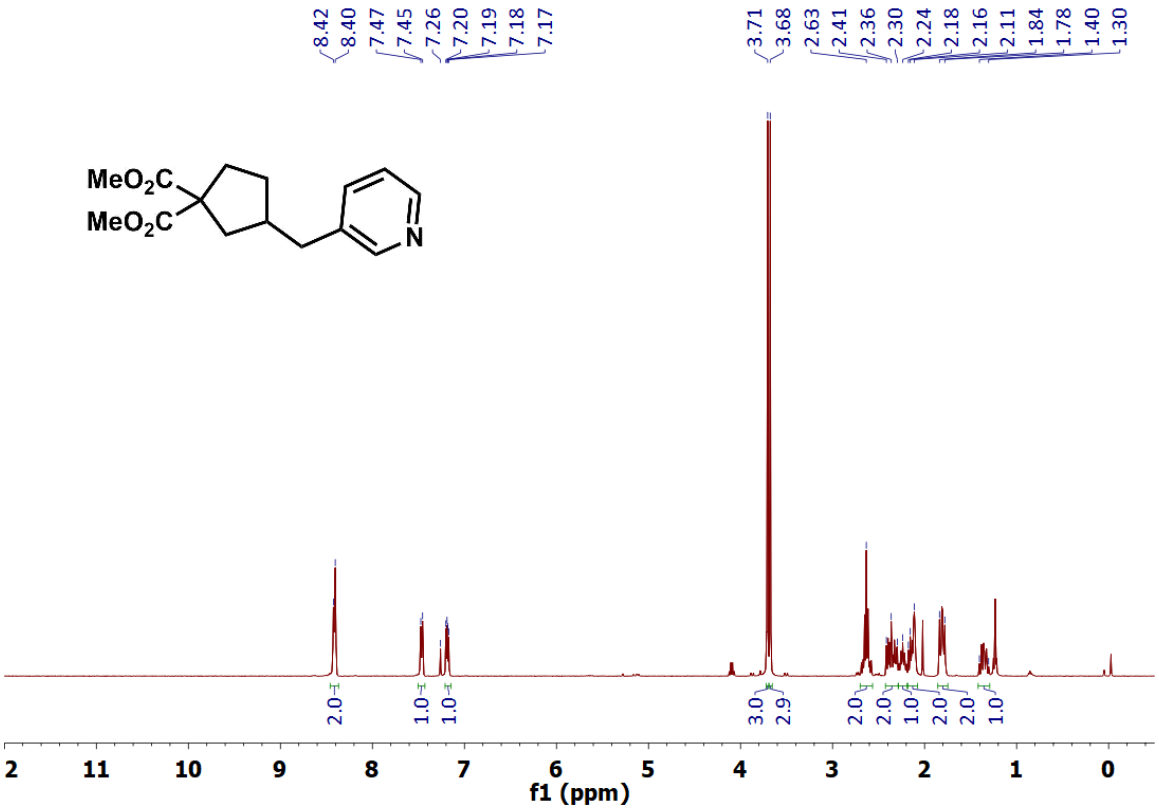
52.9
40.8
40.3
38.5
34.1
32.4
19.7
MeO2C
MeO2C
f1 (ppm)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7.18
7.17
3.71
3.68
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2.41
2.36
2.30
2.24
2.18
2.16
2.11
1.84
1.78
1.40
1.30
MeO2C
MeO2C
N
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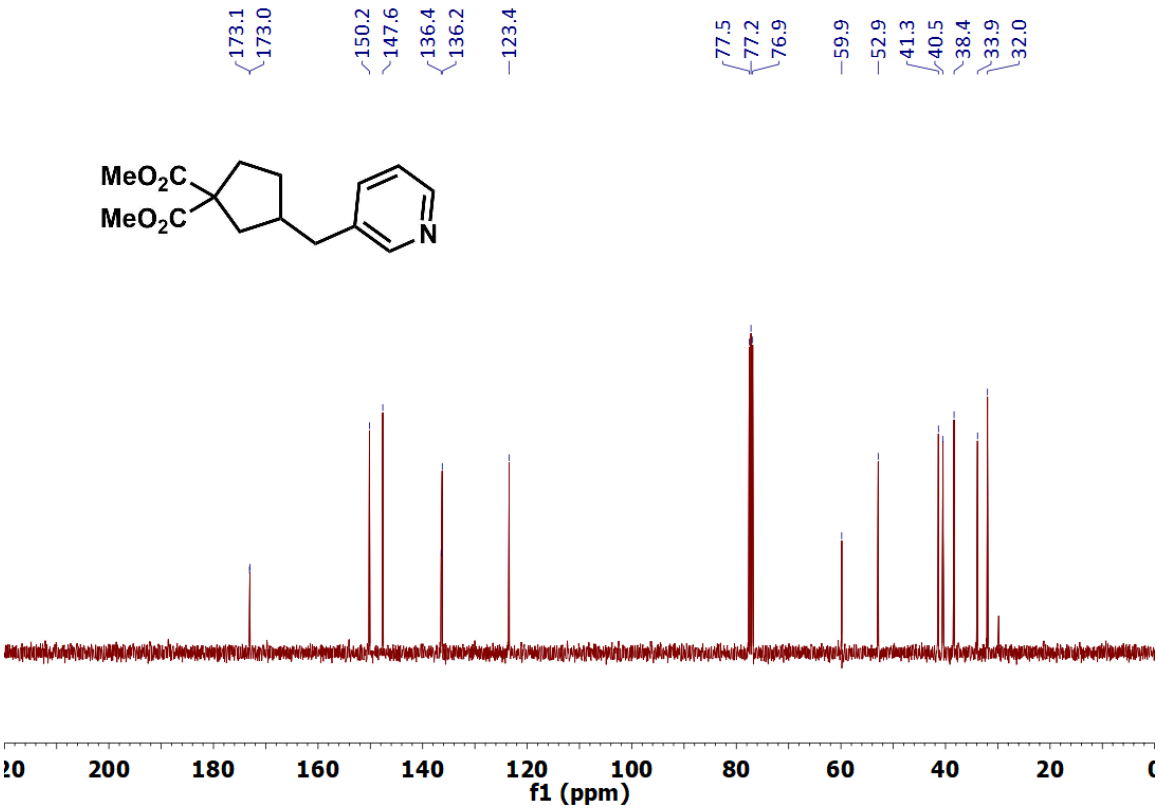
1.0
1.0
3.0
2.9
2.0
2.0
1.0
2.0
2.0
1.0
f1 (ppm)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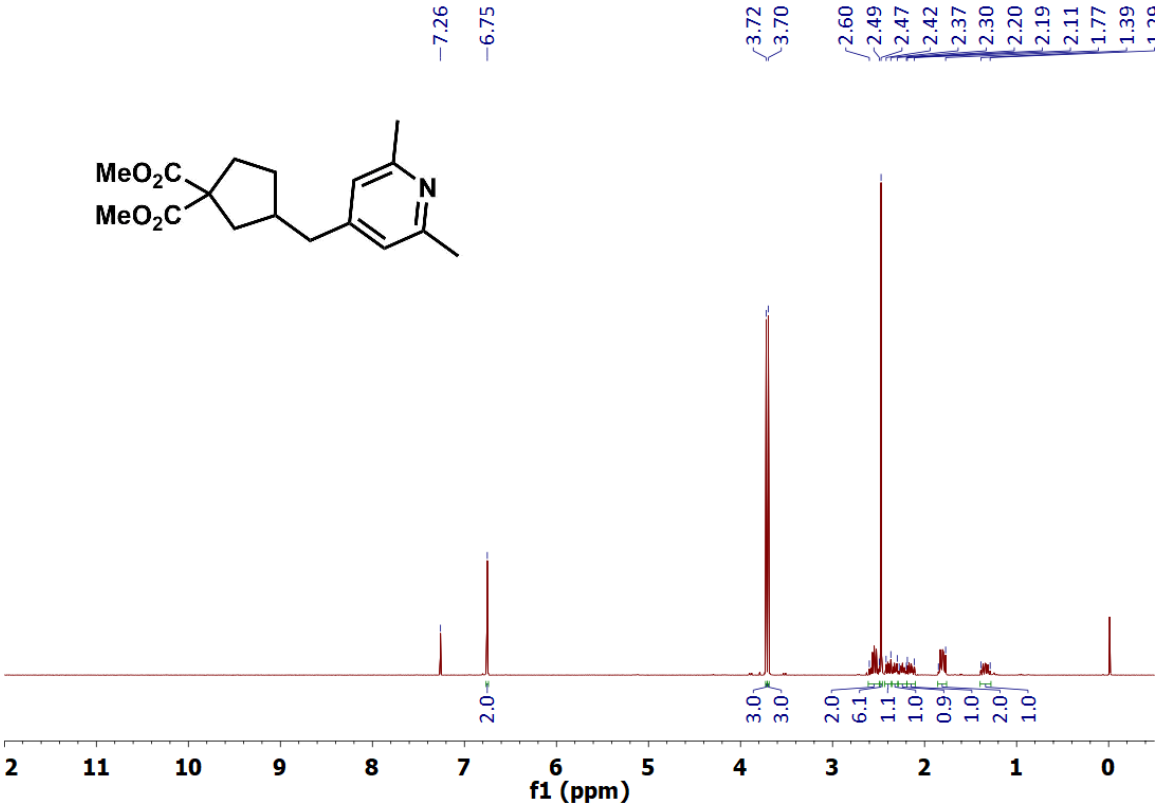
173.0
150.2
147.6
136.4
136.2
123.4
77.5
77.2
76.9
59.9
52.9
41.3
40.5
38.4
33.9
32.0
MeO2C
MeO2C
N
f1 (ppm)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2.42
2.37
2.30
2.20
2.19
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1.77
1.39
MeO2C
MeO2C
N
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2.0
6.1
1.1
1.0
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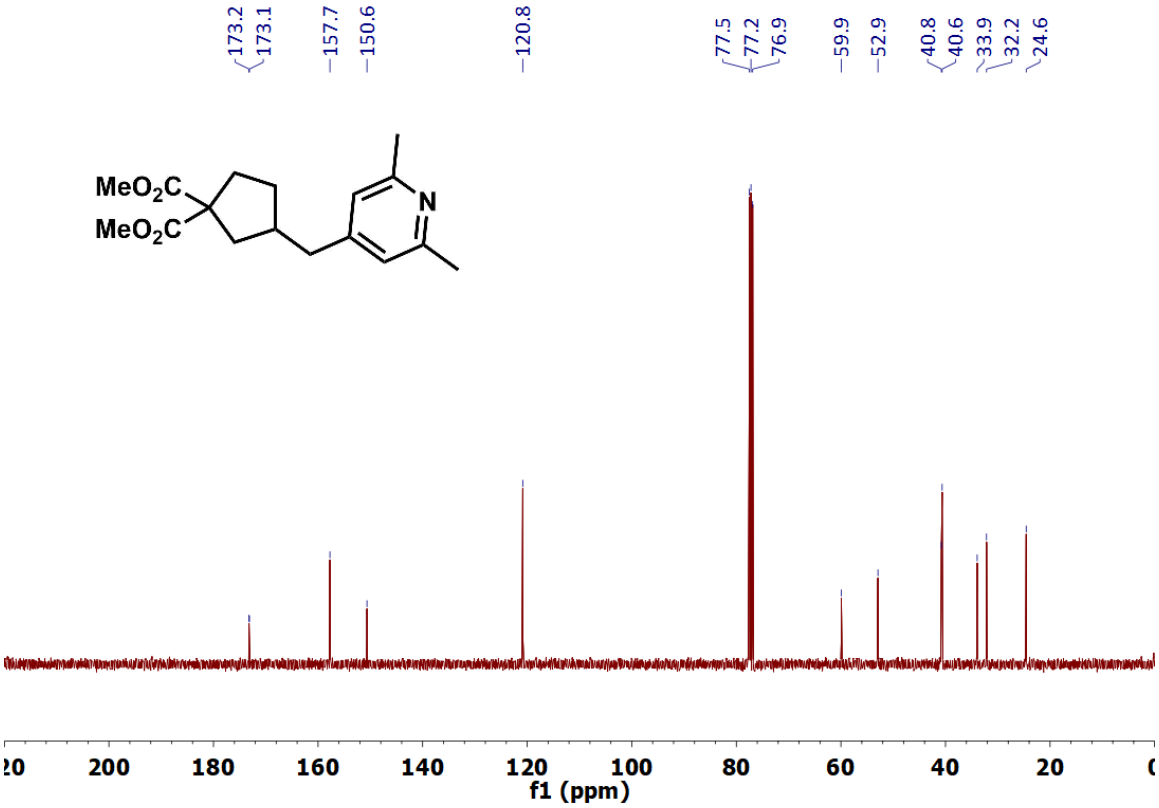
1.0
2.0
1.0
f1 (ppm)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173.1
157.7
150.6
120.8
77.5
77.2
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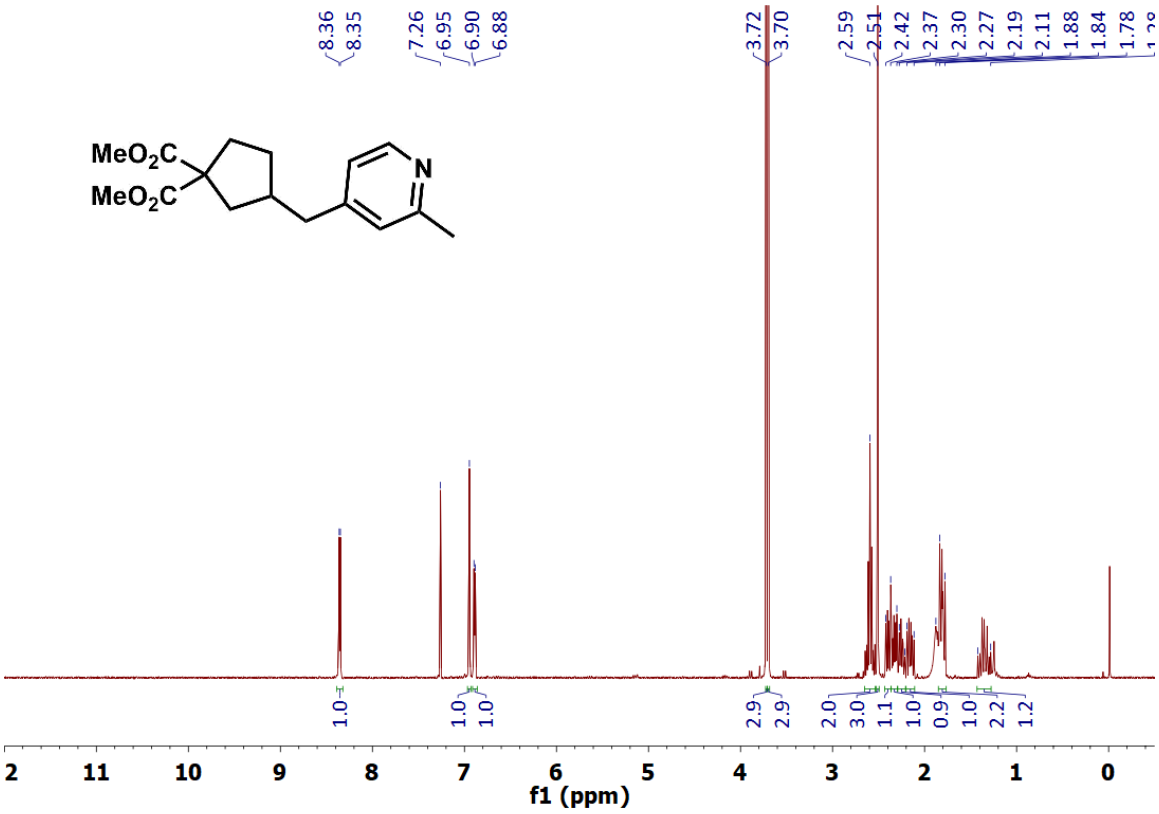
40.8
40.6
33.9
32.2
24.6
MeO2C
MeO2C
N
f1 (ppm)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3.70
2.59
2.51
2.42
2.37
2.30
2.27
2.19
2.11
1.88
1.84
1.78
MeO2C
MeO2C
N
1.0
1.0
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2.9
2.0
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1.1
1.0
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1.0
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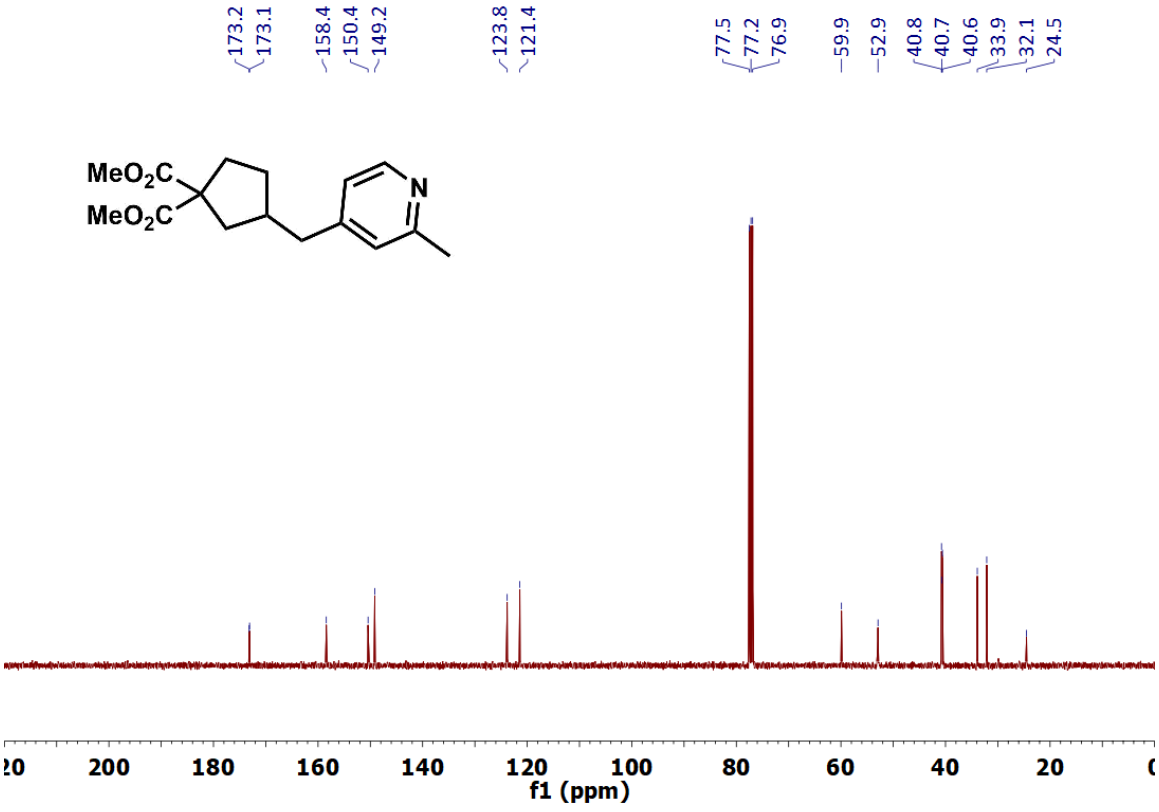
1.2
f1 (ppm)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173.1
158.4
150.4
149.2
123.8
121.4
77.5
77.2
76.9
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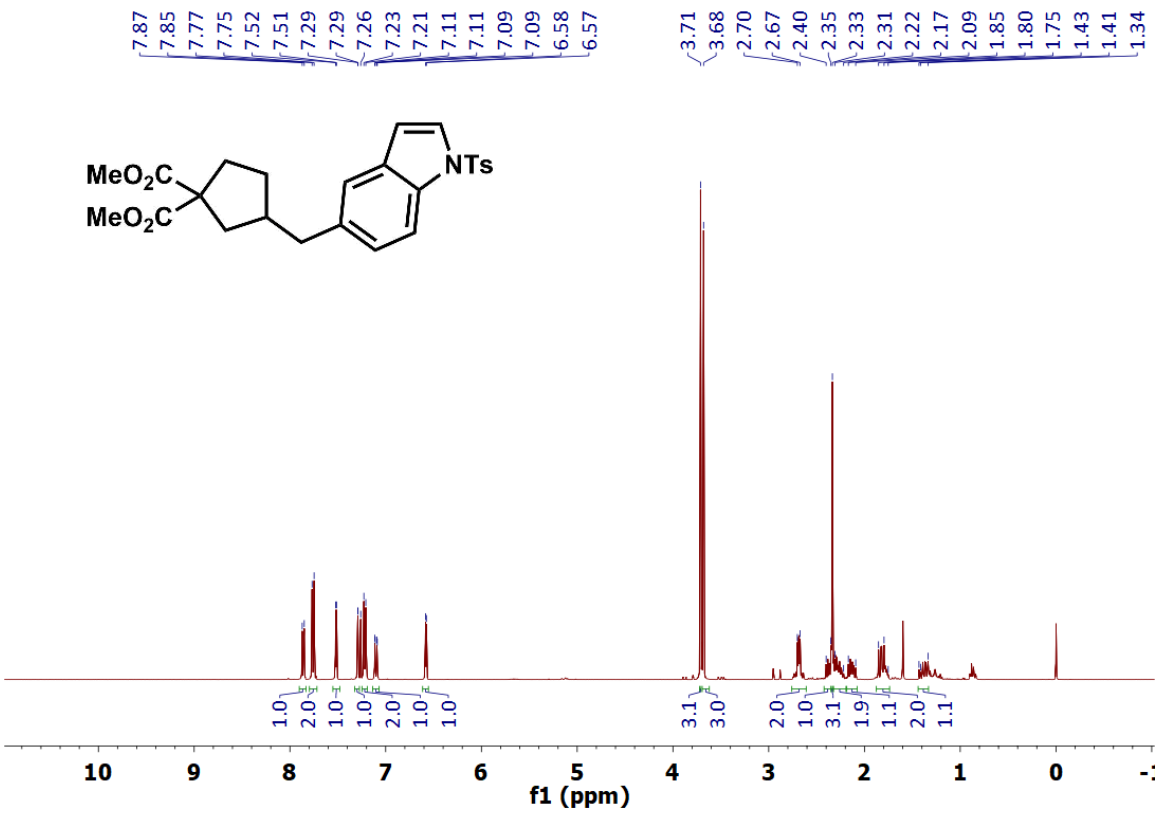
52.9
40.8
40.7
40.6
33.9
32.1
24.5
MeO2C
MeO2C
N
f1 (ppm)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7.21
7.11
7.11
7.09
7.09
6.58
6.57
3.71
3.68
2.70
2.67
2.40
2.35
2.33
2.31
2.22
2.17
2.09
1.85
1.80
1.75
1.43
1.41
1.34
MeO2C
MeO2C
NTs
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2.0
1.0
1.0
2.0
1.0
1.0
3.1
3.0
2.0
1.0
3.1
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5
4
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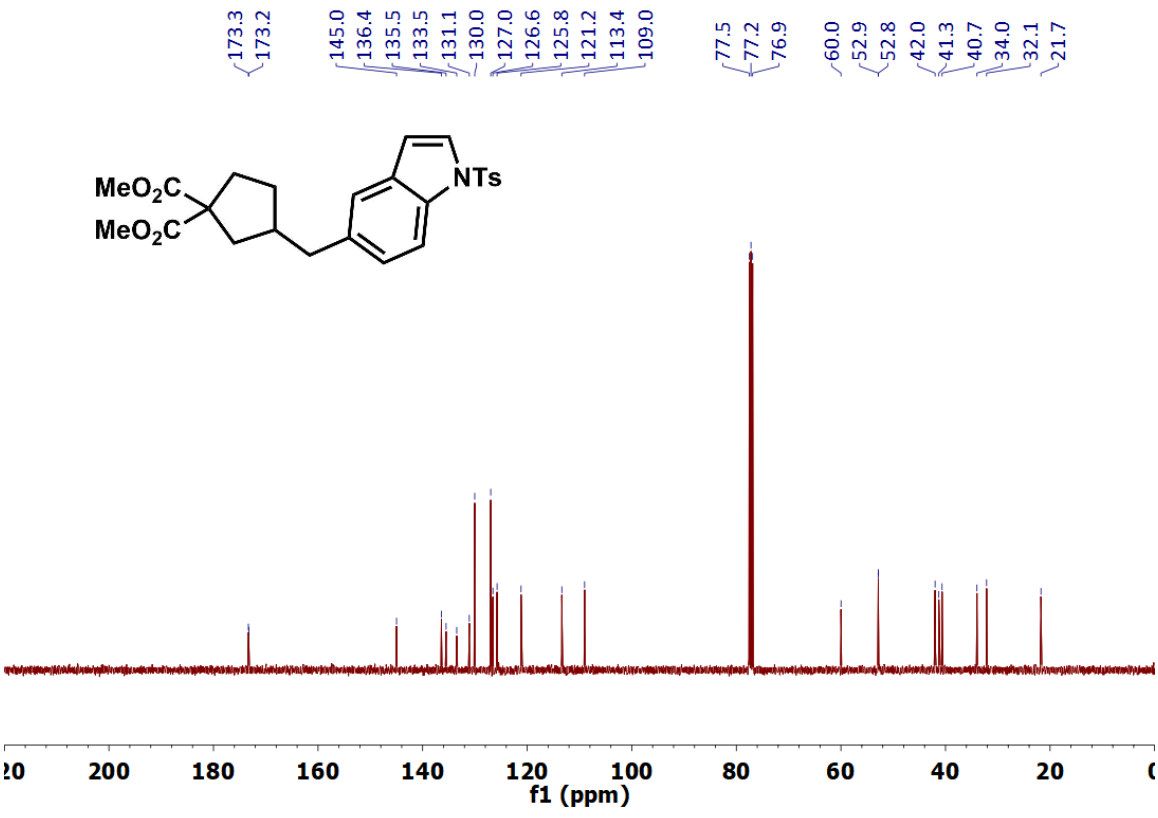
1
0
f1 (ppm)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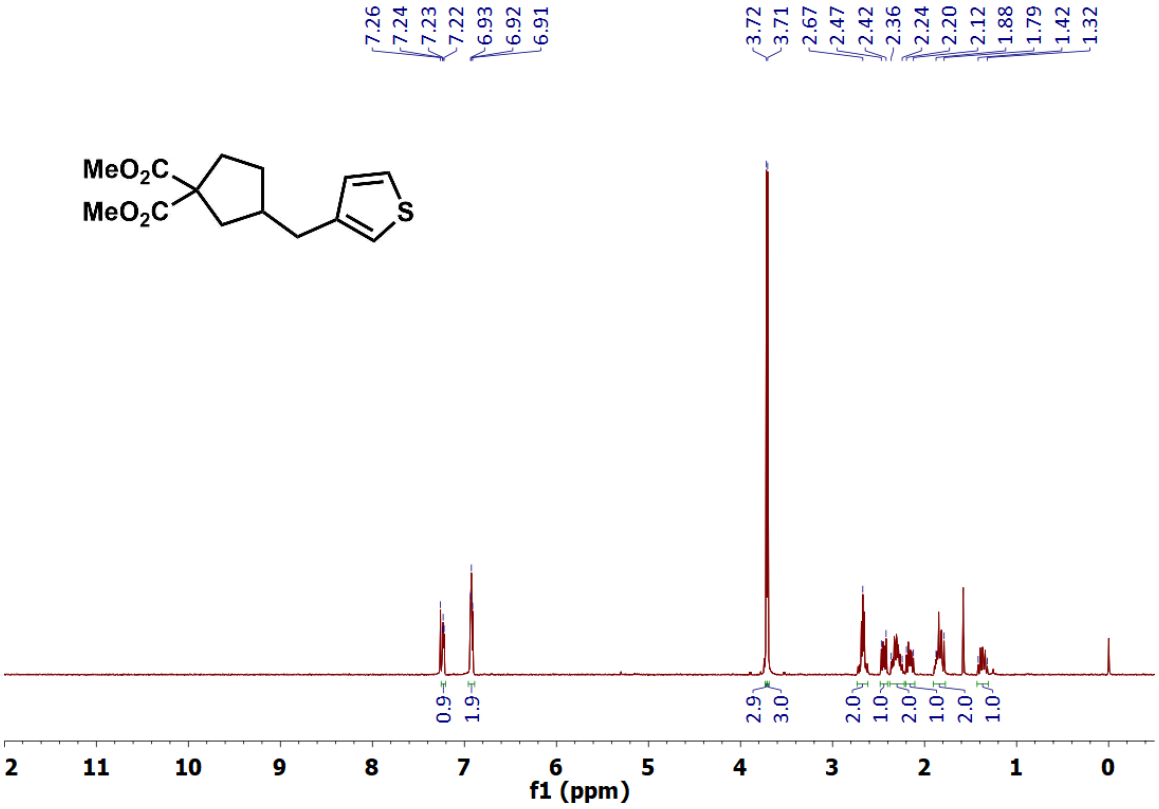
130.0
127.0
126.6
125.8
121.2
113.4
109.0
77.5
77.2
76.9
60.0
52.9
52.8
42.0
41.3
40.7
34.0
32.1
21.7
MeO2C
MeO2C
NTs
200
180
160
140
120
100
80
60
40
20
f1 (ppm)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2.36
2.24
2.20
2.12
1.88
1.79
1.42
1.32
MeO2C
MeO2C
S
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2.9
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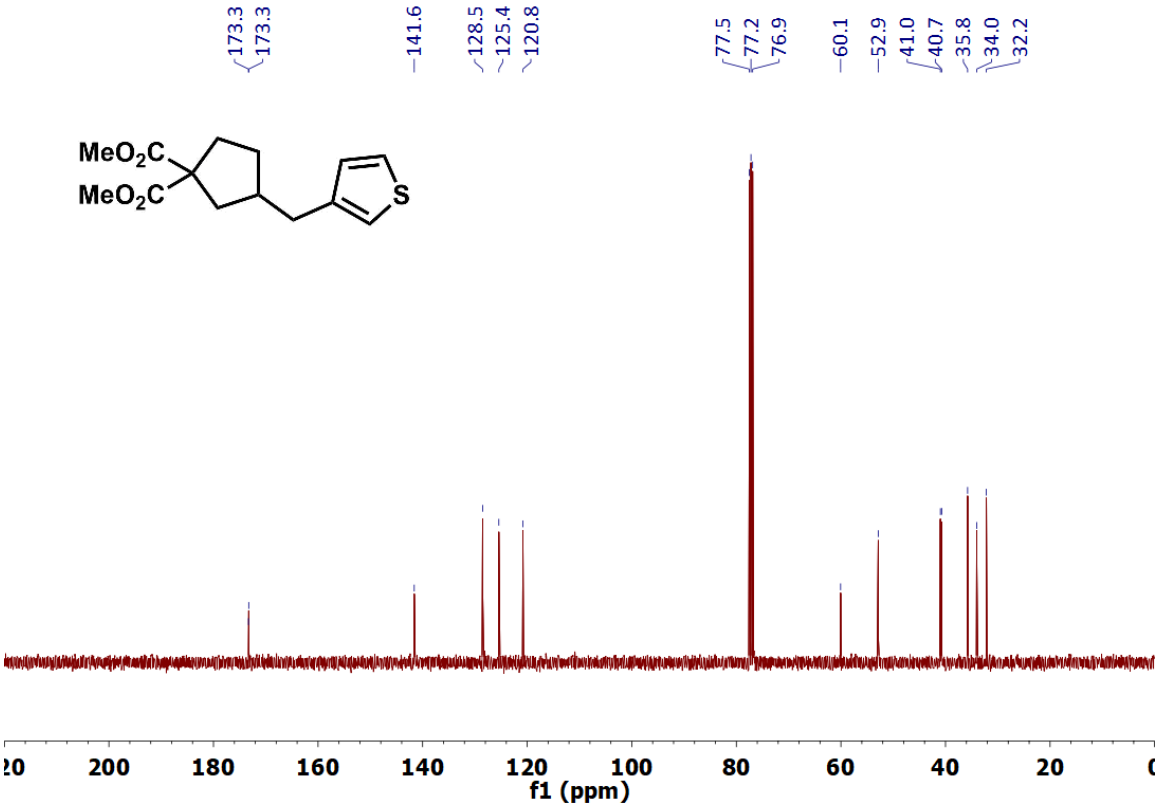
2.0
1.0
2.0
1.0
2.0
1.0
f1 (ppm)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173.3
141.6
128.5
125.4
120.8
77.5
77.2
76.9
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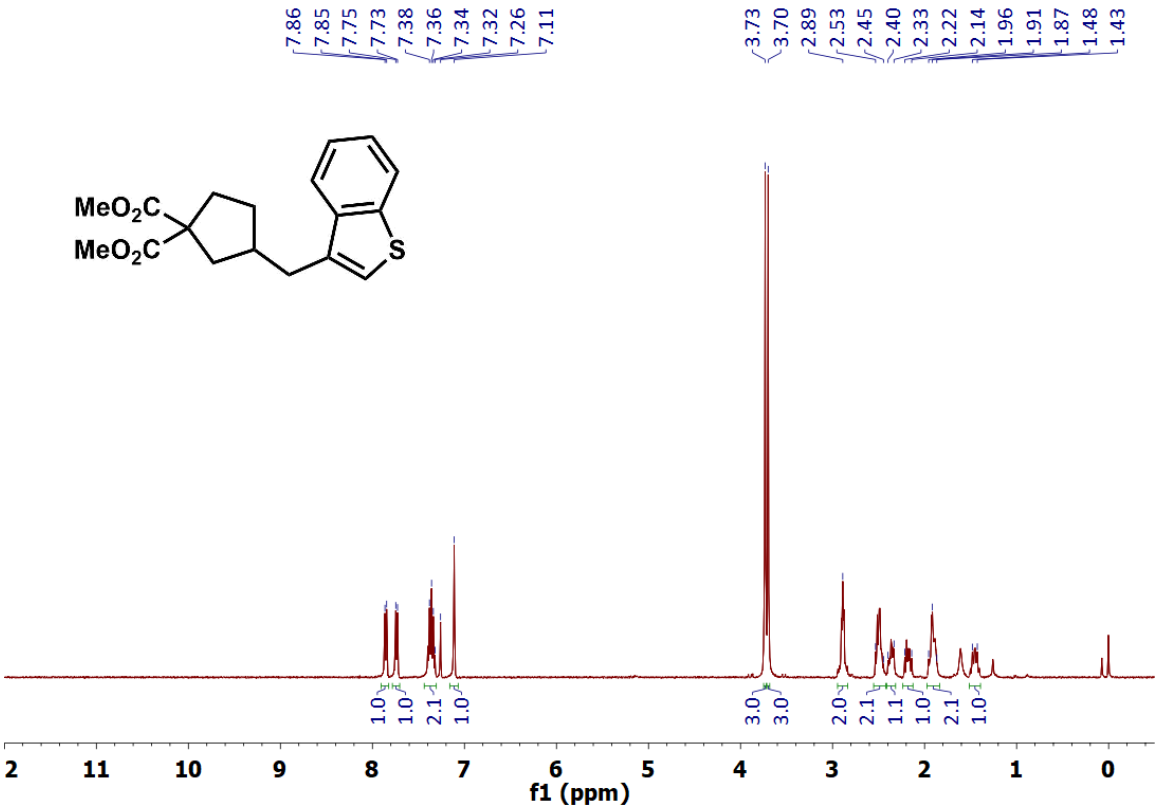
52.9
41.0
40.7
35.8
34.0
32.2
MeO2C
MeO2C
S
f1 (ppm)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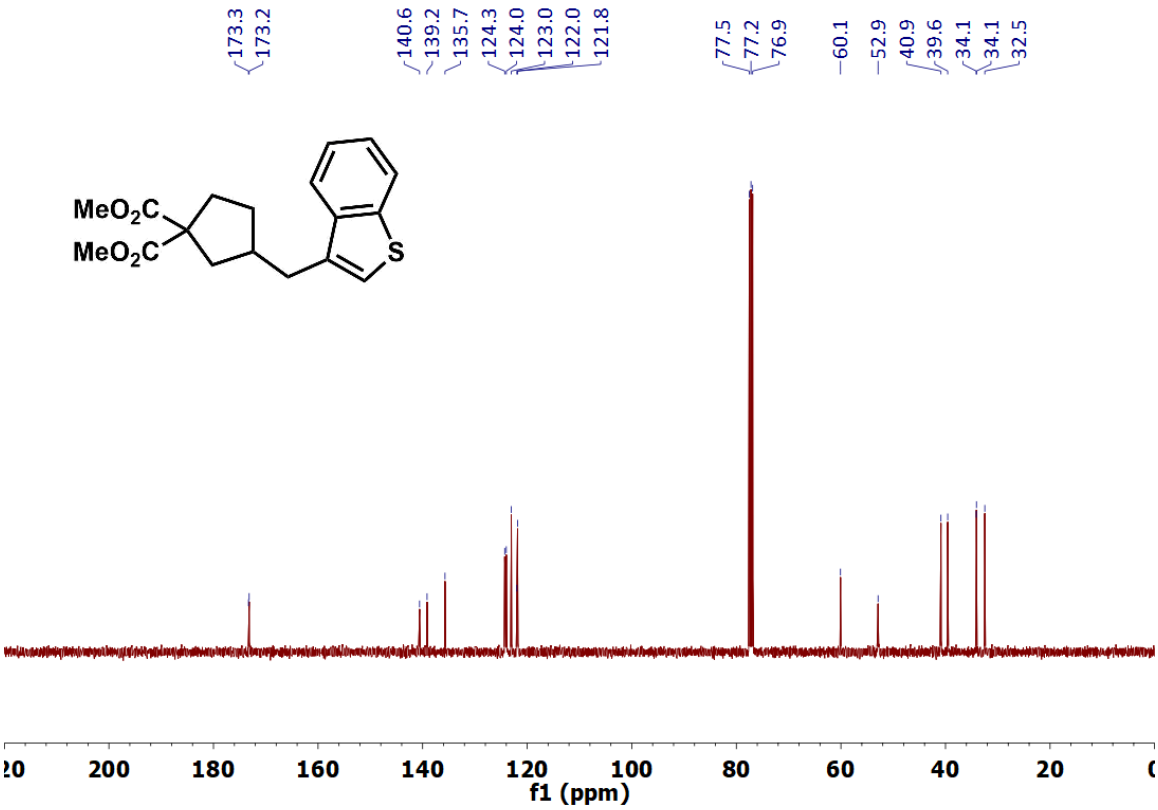
2.53
2.45
2.40
2.33
2.22
2.14
1.96
1.91
1.87
1.48
1.43
MeO2C
MeO2C
S
1.0
1.0
2.1
1.0
3.0
3.0
2.0
2.1
1.1
1.0
2.1
1.0
f1 (ppm)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139.2
135.7
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124.0
123.0
122.0
121.8
77.5
77.2
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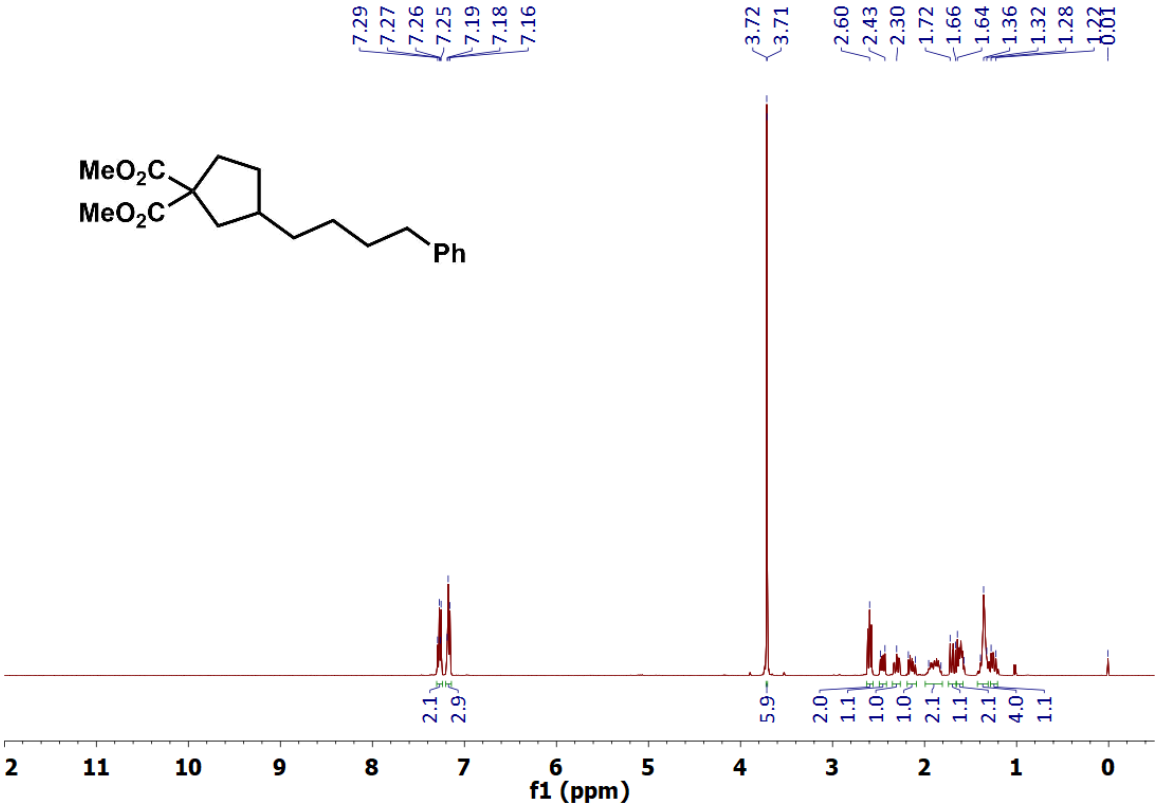
40.9
39.6
34.1
34.1
32.5
MeO2C
MeO2C
S
f1 (ppm)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2.30
1.72
1.66
1.64
1.36
1.32
1.28
1.22
0.01
MeO2C
MeO2C
Ph
2.1
2.9
5.9
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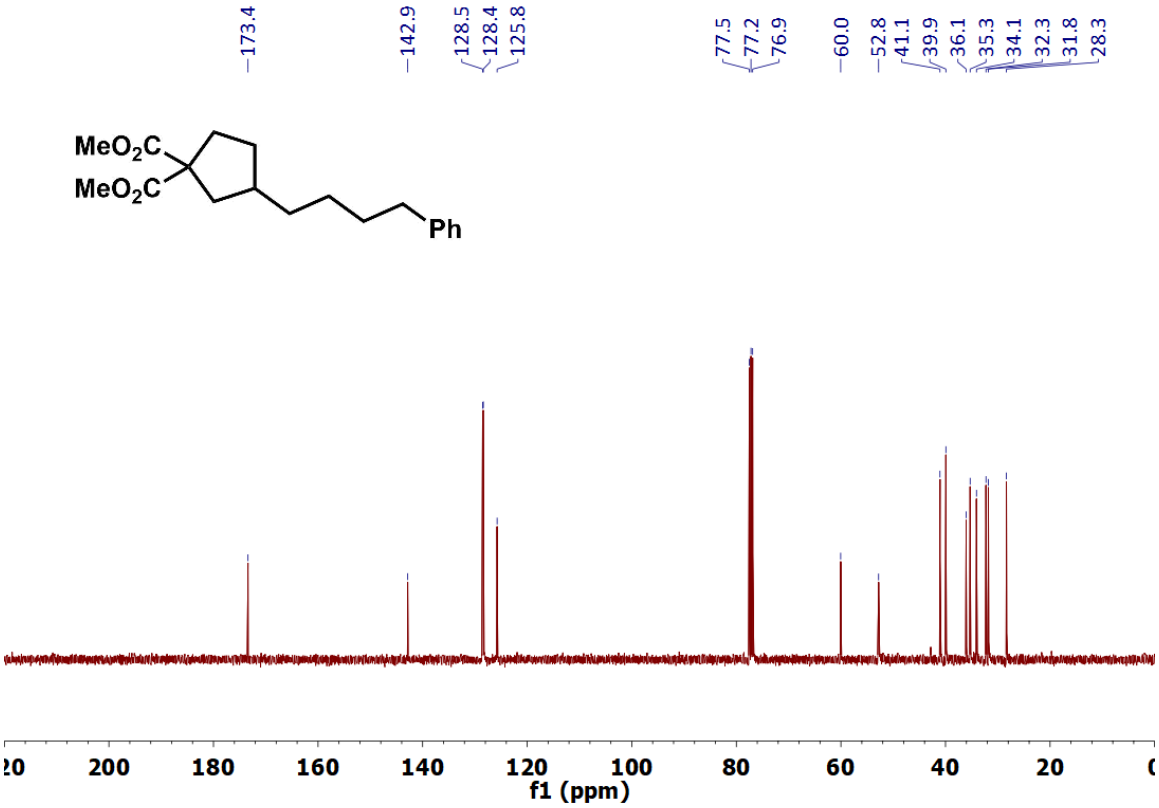
1.1
1.0
1.0
2.1
1.1
2.1
4.0
1.1
f1 (ppm)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128.5
128.4
125.8
77.5
77.2
76.9
60.0
52.8
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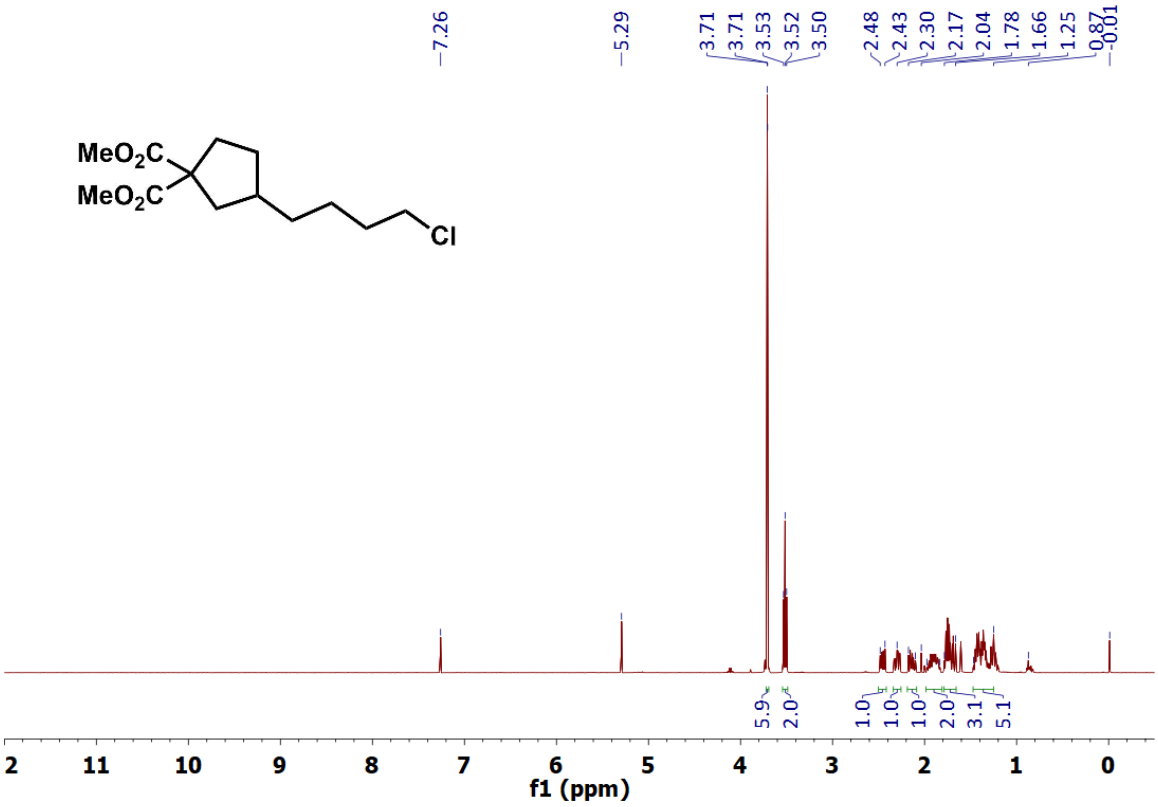
34.1
32.3
31.8
28.3
MeO2C
MeO2C
Ph
f1 (ppm)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3.71
3.53
3.52
3.50
2.48
2.43
2.30
2.17
2.04
1.78
1.66
1.25
0.87
0.01
MeO2C
MeO2C
Cl
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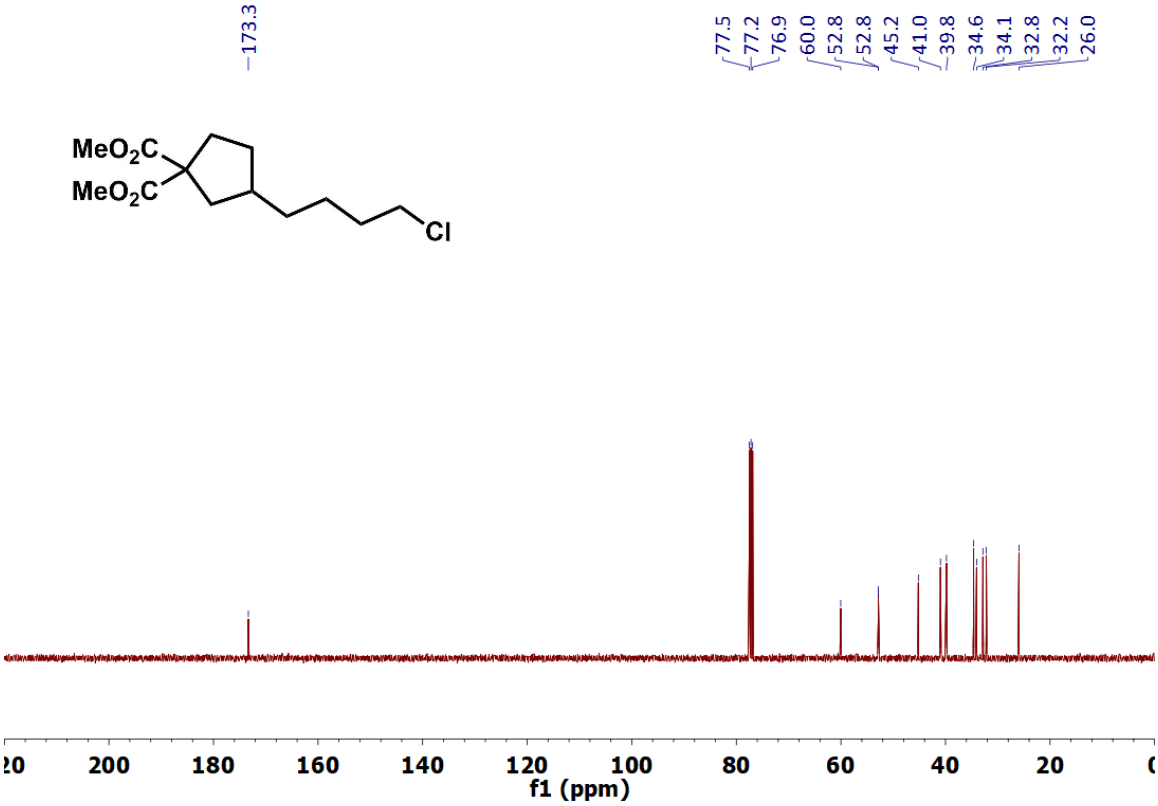
1.0
1.0
1.0
2.0
3.1
5.1
f1 (ppm)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76.9
60.0
52.8
52.8
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41.0
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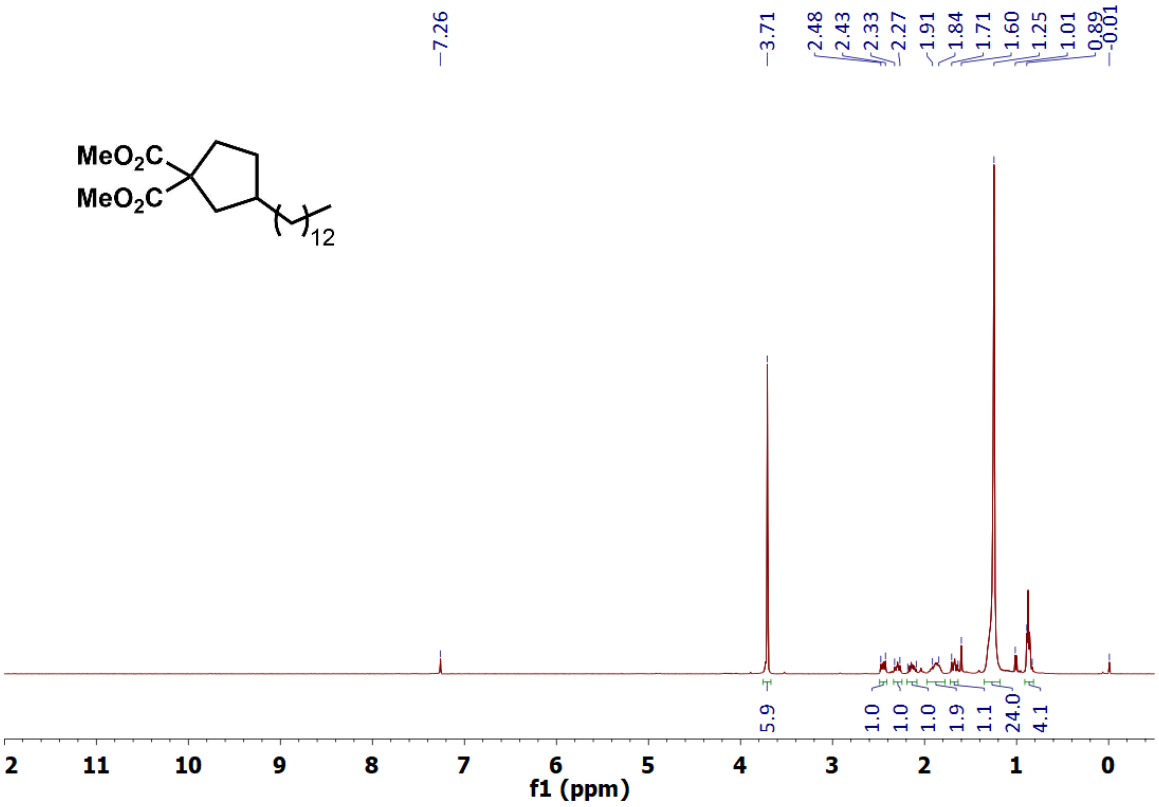
34.6
34.1
32.8
32.2
26.0
MeO2C
MeO2C
Cl
f1 (ppm)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2.27
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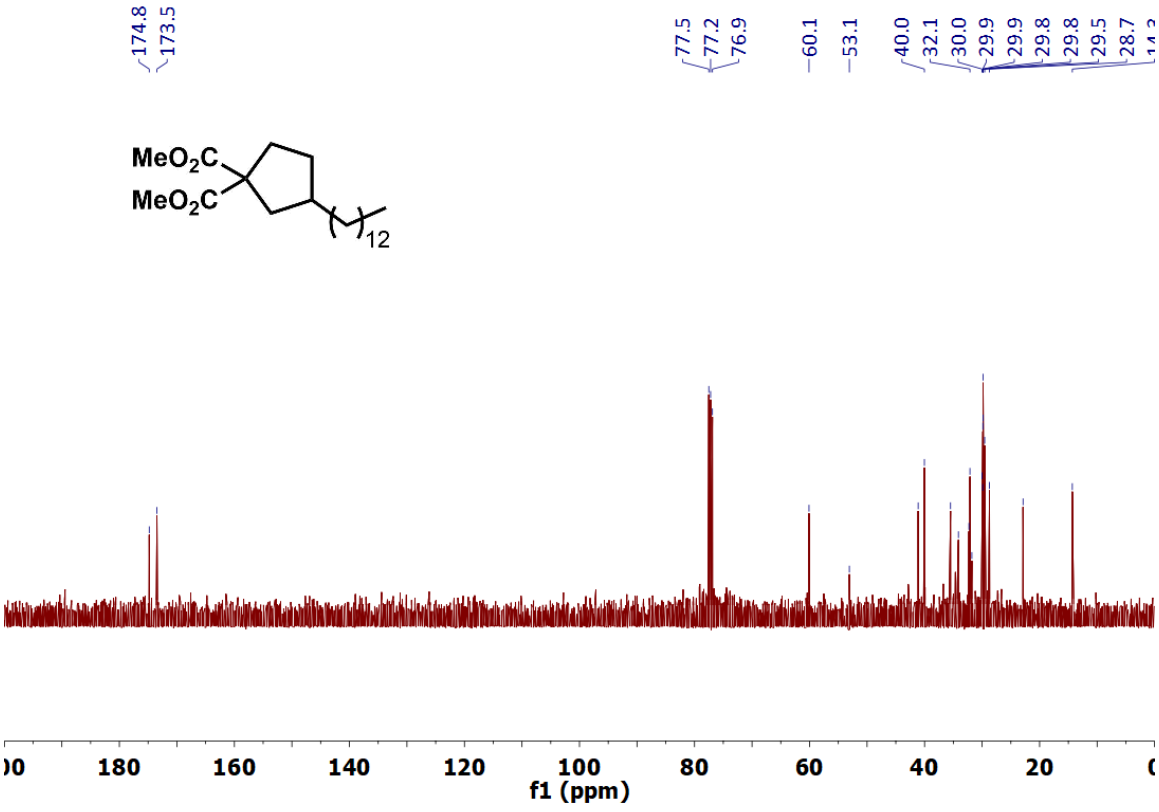
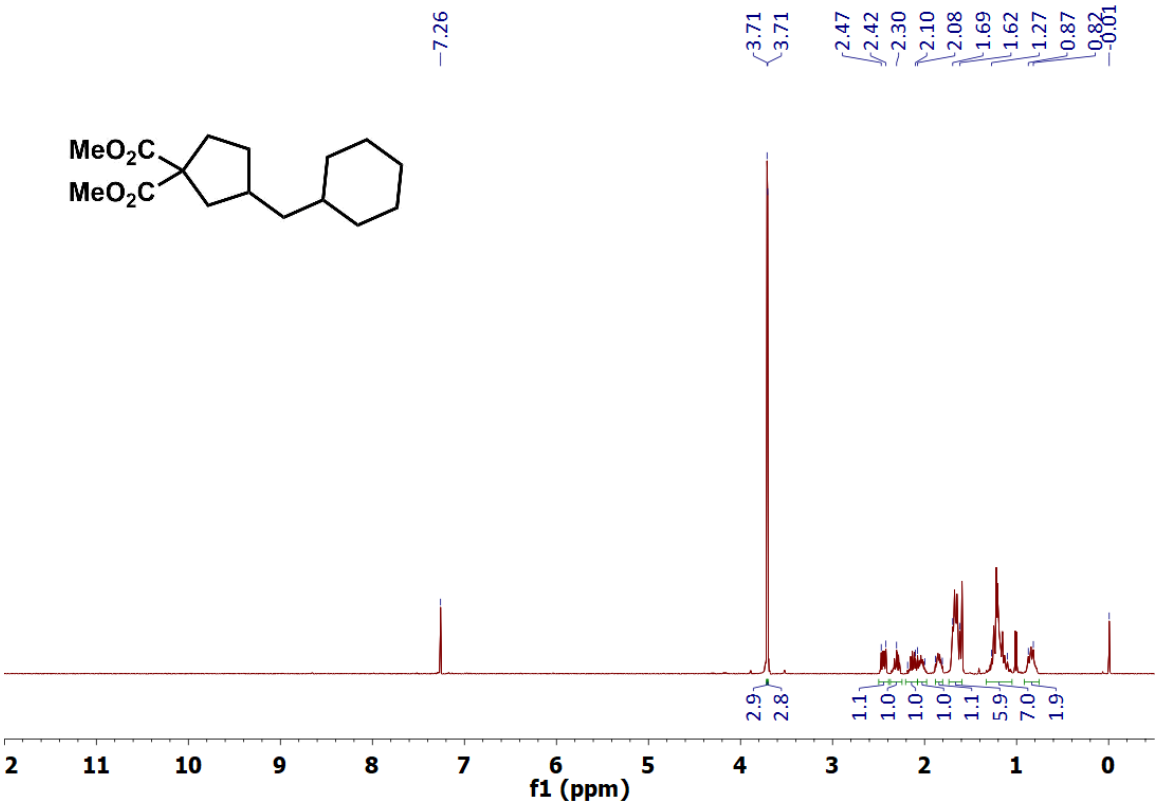
1.60
1.25
1.01
0.89
0.01
MeO2C
MeO2C
12
5.9
1.0
1.0
1.0
1.9
1.1
24.0
4.1
f1 (ppm)


174.8
173.5
77.5
77.2
76.9
60.1
53.1
40.0
32.1
30.0
29.9
29.9
29.8
29.8
29.5
28.7
MeO2C
MeO2C
12
f1 (ppm)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3.71
3.71
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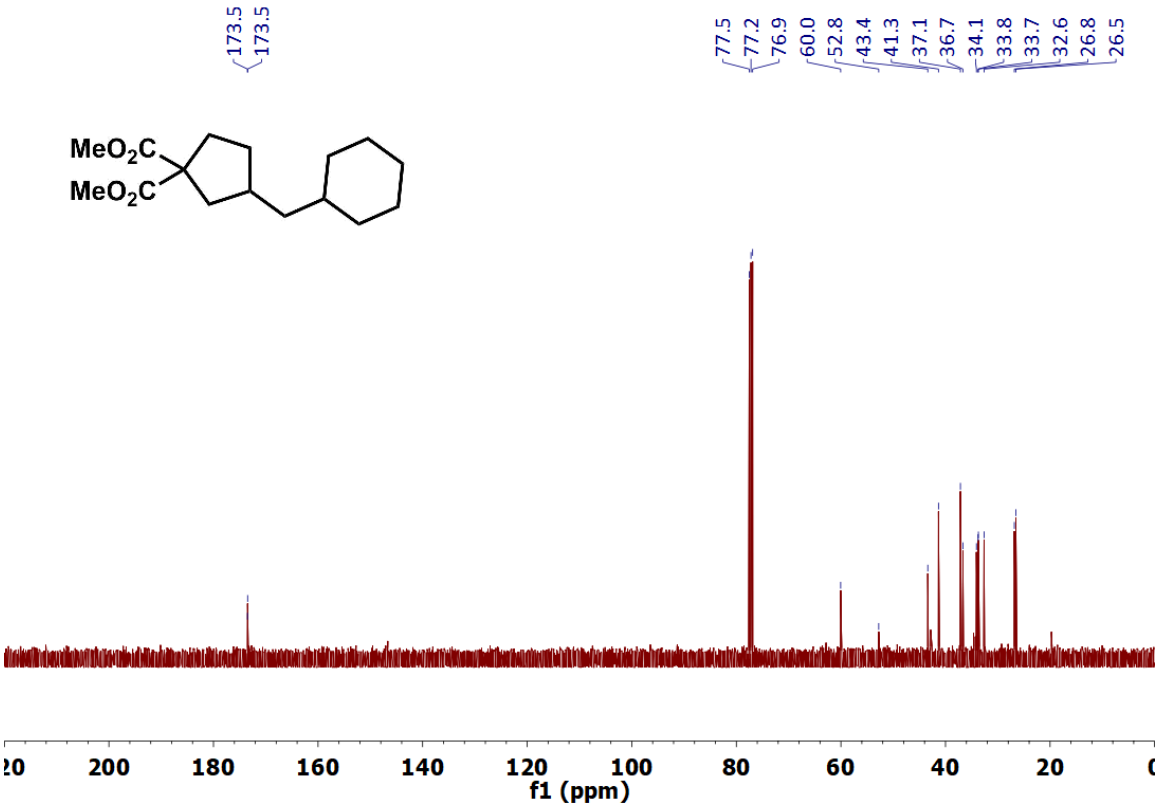
2.42
2.30
2.10
2.08
1.69
1.62
1.27
0.87
0.82
0.01
MeO2C
MeO2C
2.9
2.8
1.1
1.0
1.0
1.0
1.1
5.9
7.0
1.9
f1 (ppm)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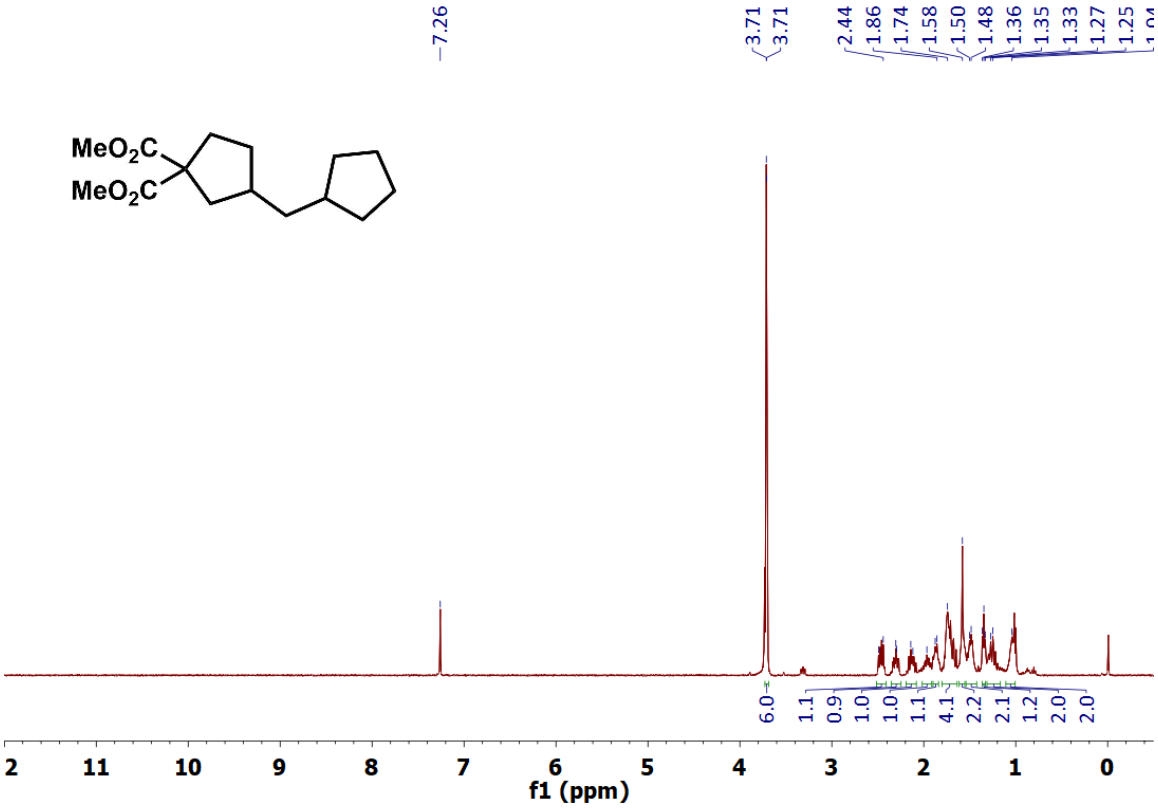
173.5
77.5
77.2
76.9
60.0
52.8
43.4
41.3
37.1
36.7
34.1
33.8
33.7
32.6
26.8
26.5
MeO2C
MeO2C
f1 (ppm)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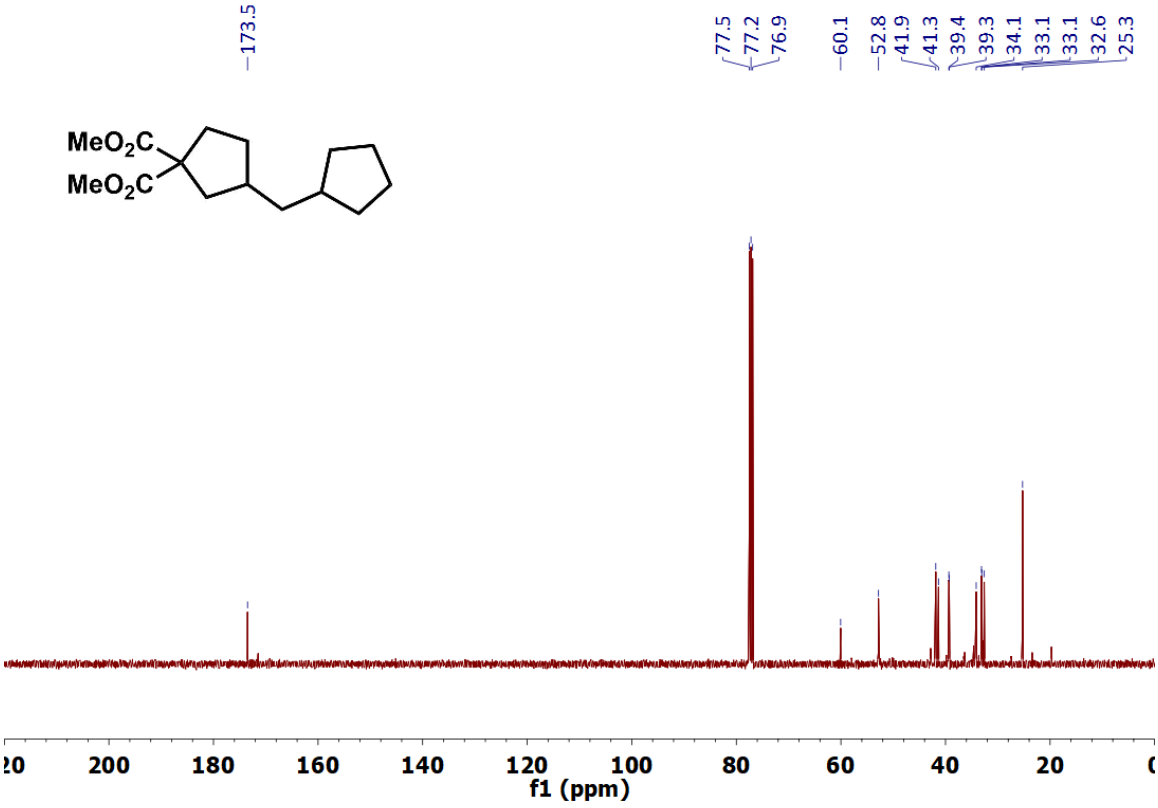
3.71
2.44
1.86
1.74
1.58
1.50
1.48
1.36
1.35
1.33
1.27
1.25
MeO2C
MeO2C
6.0
1.1
0.9
1.0
1.0
1.1
4.1
2.2
2.1
1.2
2.0
2.0
f1 (ppm)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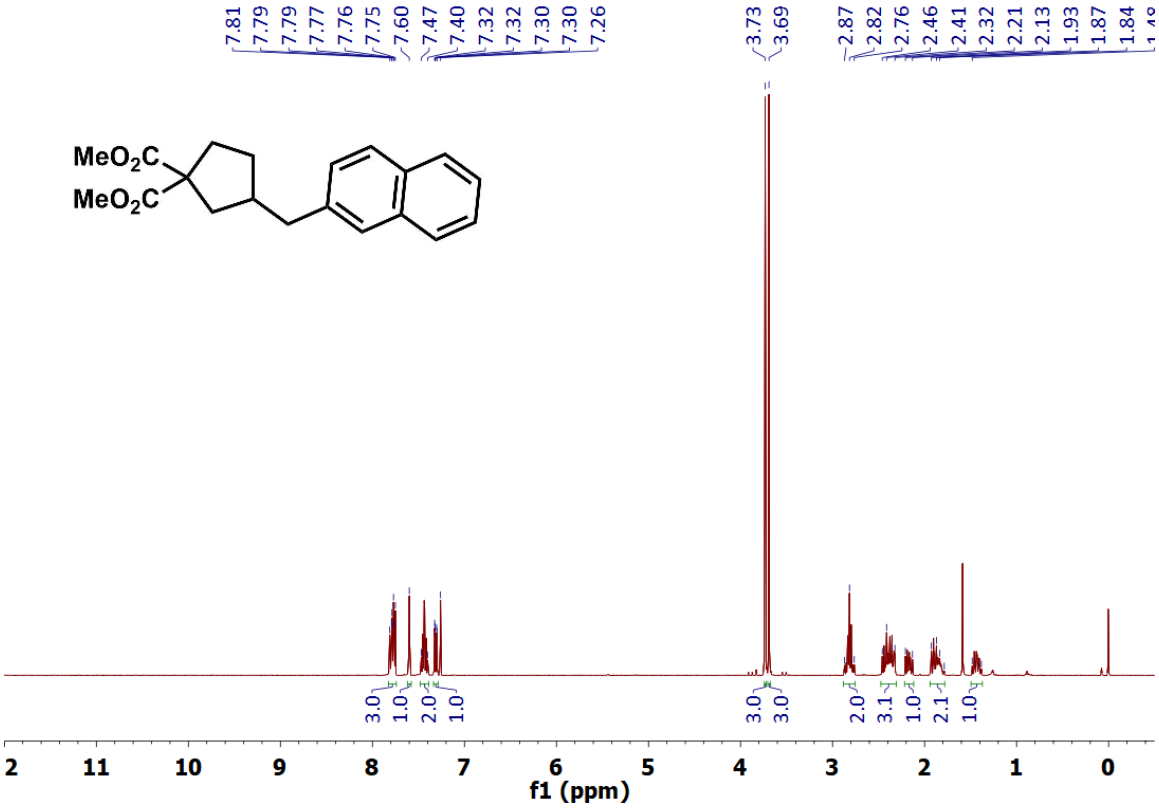
77.2
76.9
60.1
52.8
41.9
41.3
39.4
39.3
34.1
33.1
33.1
32.6
25.3
MeO2C
MeO2C
f1 (ppm)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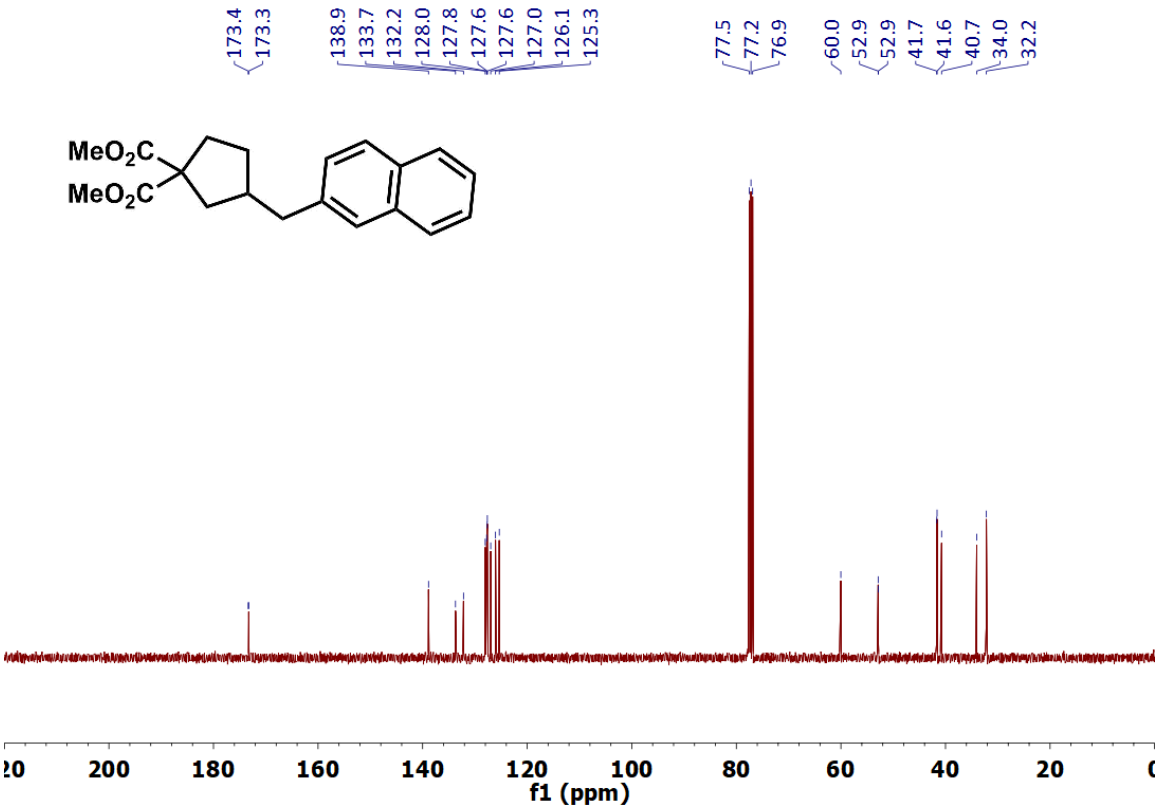
7.79
7.79
7.77
7.76
7.75
7.60
7.47
7.40
7.32
7.32
7.30
7.30
7.26
3.73
3.69
2.87
2.82
2.76
2.46
2.41
2.32
2.21
2.13
1.93
1.87
1.84
MeO2C
MeO2C
3.0
1.0
2.0
1.0
3.0
3.0
2.0
3.1
1.0
2.1
1.0
f1 (ppm)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173.3
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132.2
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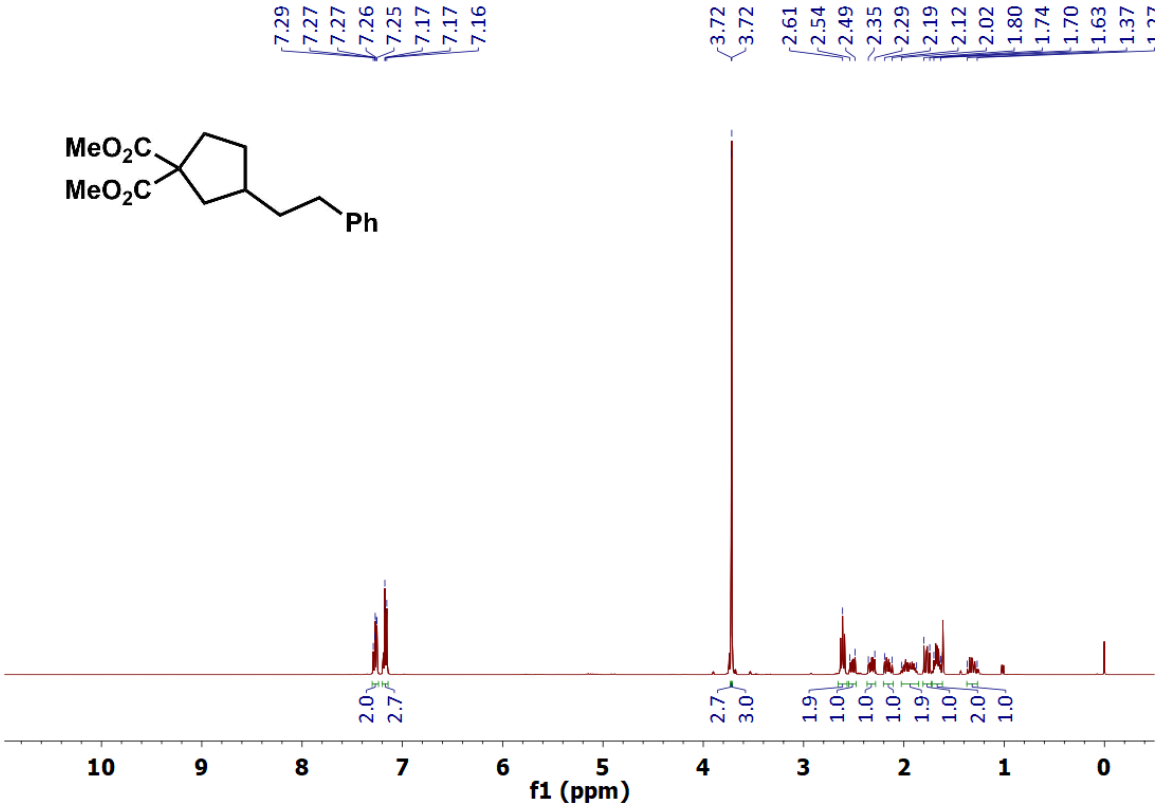
127.8
127.6
127.6
127.0
126.1
125.3
77.5
77.2
76.9
60.0
52.9
52.9
41.7
41.6
40.7
34.0
32.2
MeO2C
MeO2C
f1 (ppm)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7.27
7.27
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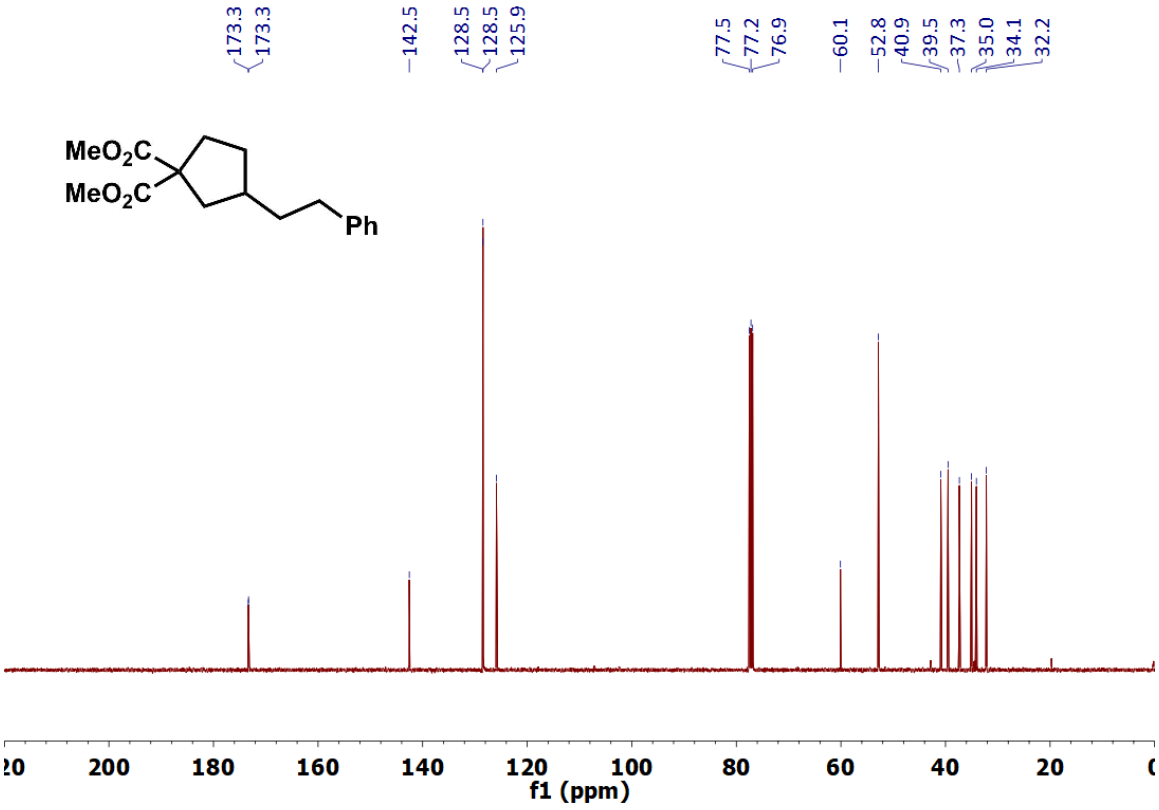
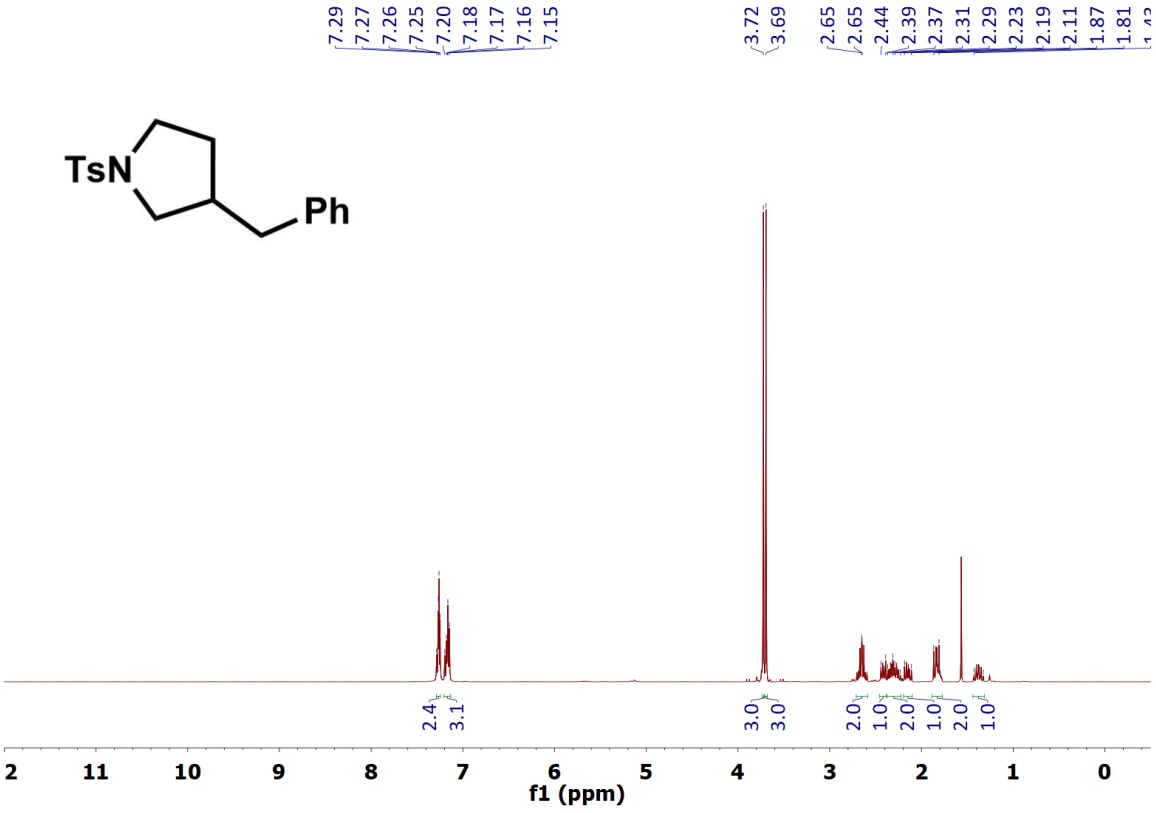
7.25
7.17
7.17
7.16
3.72
3.72
2.61
2.54
2.49
2.35
2.29
2.19
2.12
2.02
1.80
1.74
1.70
1.63
1.37
MeO2C
MeO2C
Ph
2.0
2.7
2.7
3.0
1.9
1.0
1.0
1.0
1.9
1.0
2.0
1.0
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)


173.3
173.3
142.5
128.5
128.5
125.9
77.5
77.2
76.9
60.1
52.8
40.9
39.5
37.3
35.0
34.1
32.2
MeO2C
MeO2C
Ph
200
180
160
140
120
100
80
60
40
20
f1 (ppm)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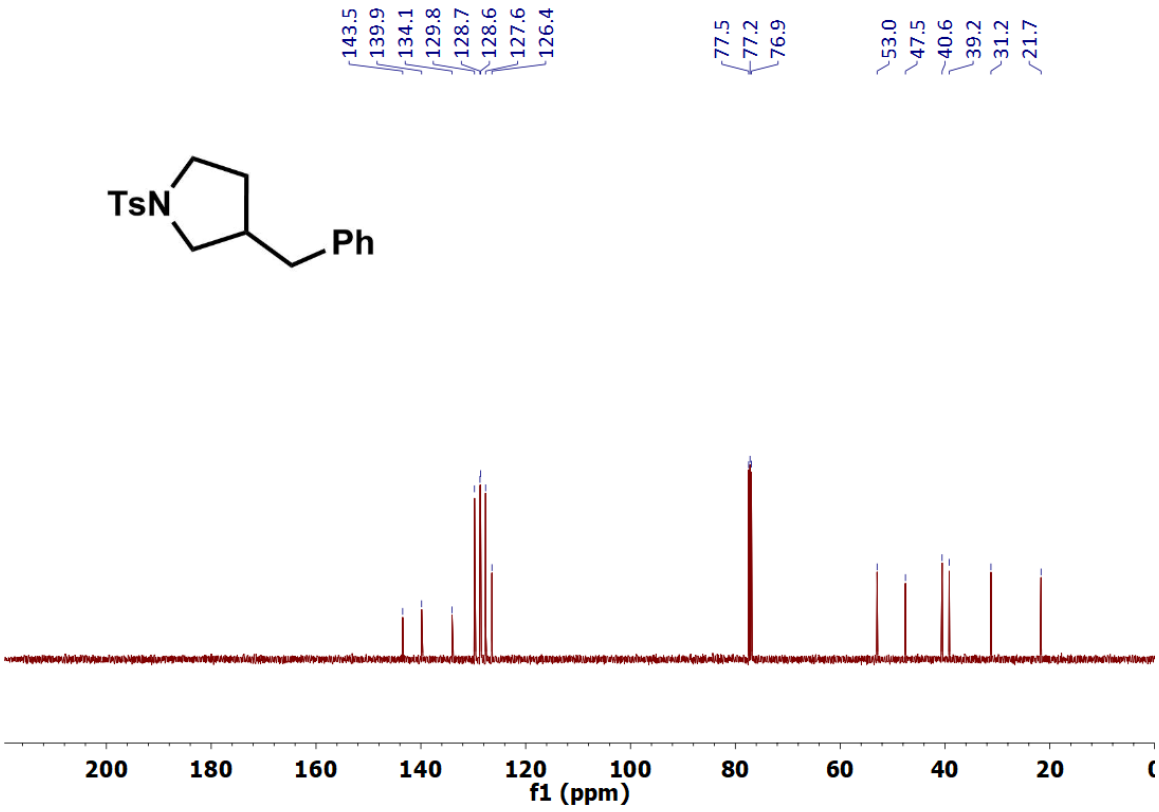
7.27
7.26
7.25
7.20
7.18
7.17
7.16
7.15
3.72
3.69
2.65
2.65
2.44
2.39
2.37
2.31
2.29
2.23
2.19
2.11
1.87
1.81
TsN
Ph
2.4
3.1
3.0
3.0
2.0
1.0
2.0
1.0
2.0
1.0
2
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)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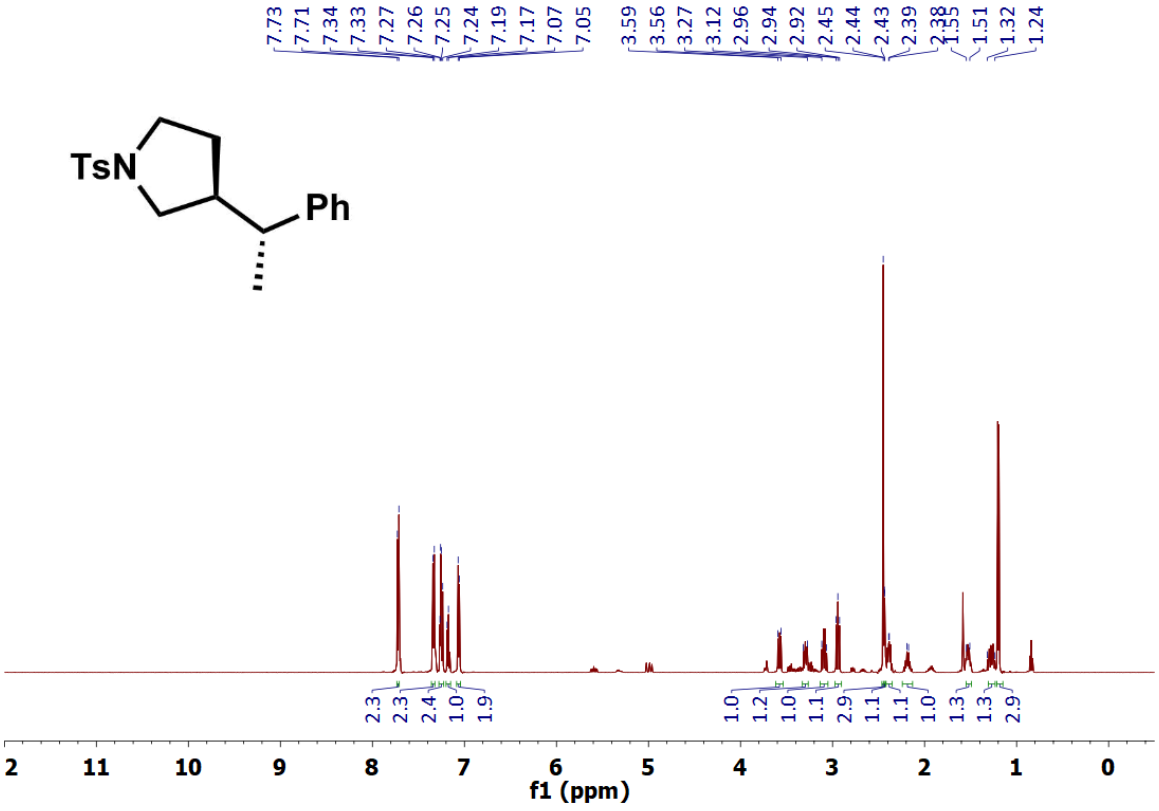
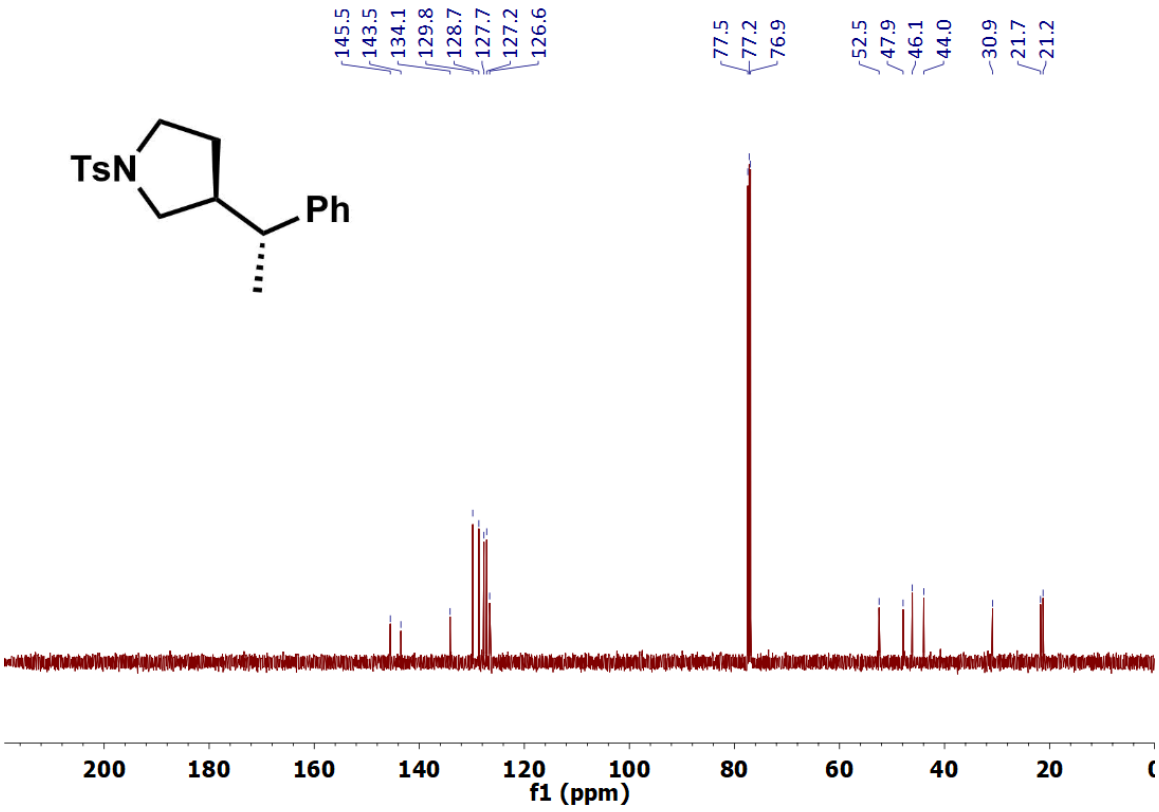
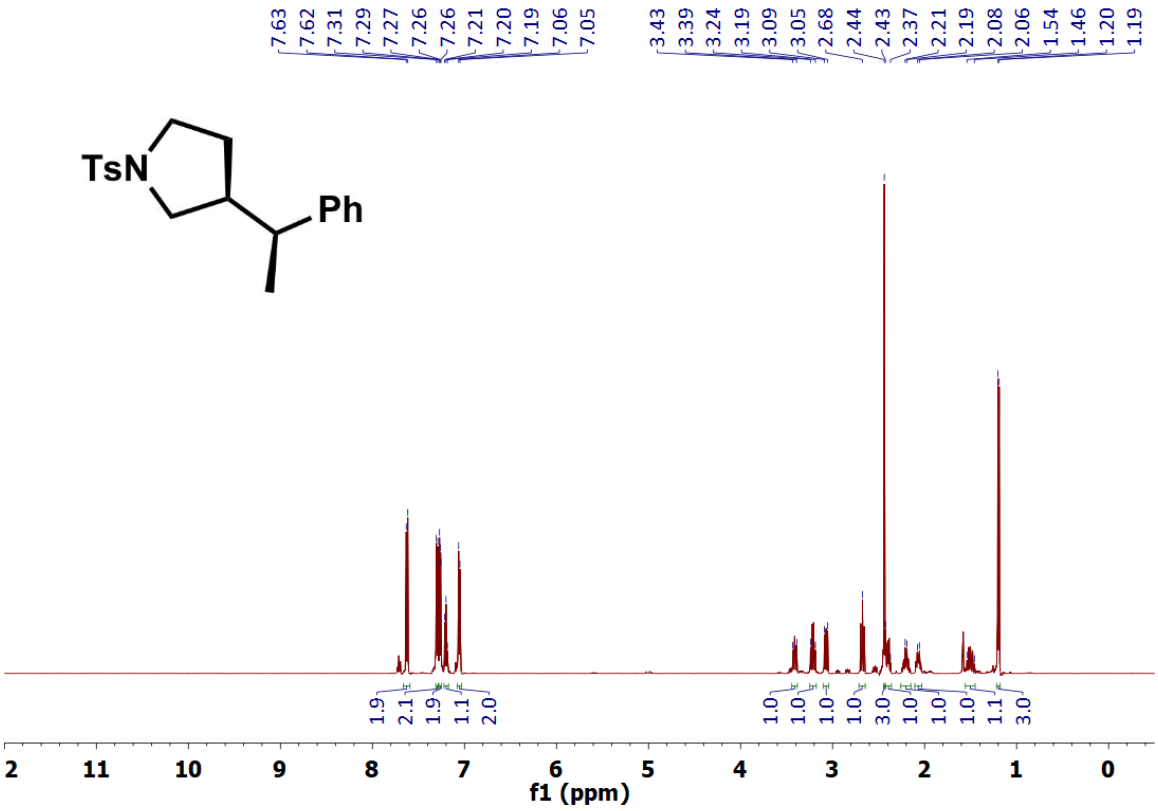
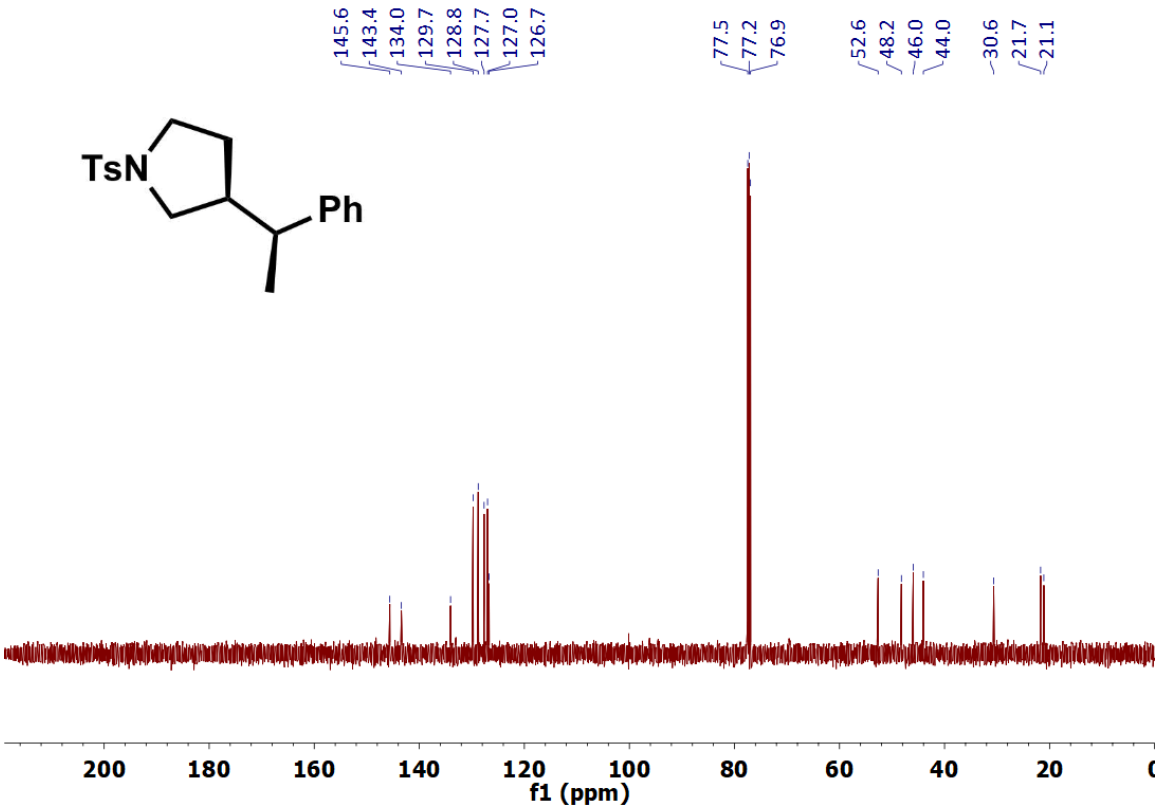
139.9
134.1
129.8
128.7
128.6
127.6
126.4
77.5
77.2
76.9
53.0
47.5
40.6
39.2
31.2
21.7
TsN
Ph
200
180
160
140
120
100
80
60
40
20
f1 (ppm)


7.73
7.71
7.34
7.33
7.27
7.26
7.25
7.24
7.19
7.17
7.07
7.05
3.59
3.56
3.27
3.12
2.96
2.94
2.92
2.45
2.44
2.43
2.39
1.55
1.51
1.32
1.24
TsN
Ph
2.3
2.3
2.4
1.0
1.9
1.0
1.2
1.0
1.1
2.9
1.1
1.1
1.0
1.3
1.3
2.9
f1 (ppm)


145.5
143.5
134.1
129.8
128.7
127.7
127.2
126.6
77.5
77.2
76.9
52.5
47.9
46.1
44.0
30.9
21.7
21.2
TsN
Ph
f1 (ppm)


7.63
7.62
7.31
7.29
7.27
7.26
7.26
7.21
7.20
7.19
7.06
7.05
3.43
3.39
3.24
3.19
3.09
3.05
2.68
2.44
2.43
2.37
2.21
2.19
2.08
2.06
1.54
1.46
1.20
1.19
TsN
Ph
1.9
2.1
1.9
1.1
2.0
1.0
1.0
1.0
1.0
3.0
1.0
1.0
1.0
1.1
3.0
f1 (ppm)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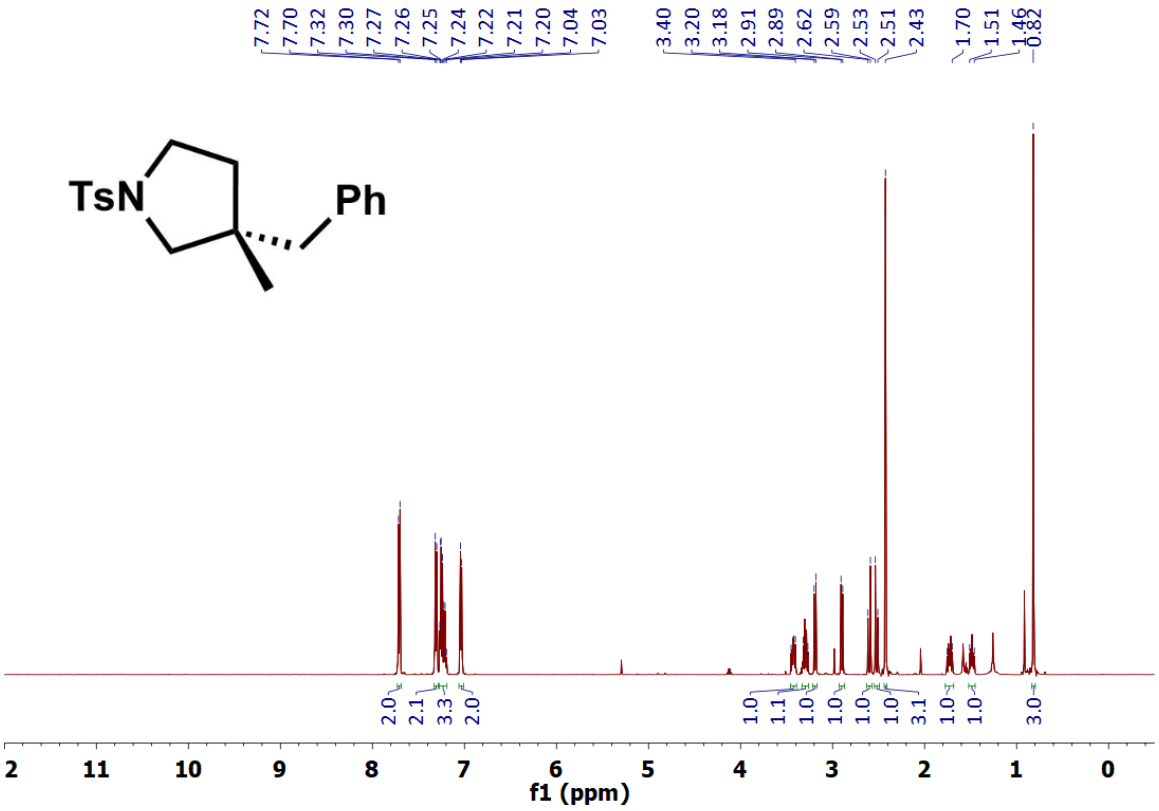
143.4
134.0
129.7
128.8
127.7
127.0
126.7
77.5
77.2
76.9
52.6
48.2
46.0
44.0
30.6
21.7
21.1
TsN
Ph
f1 (ppm)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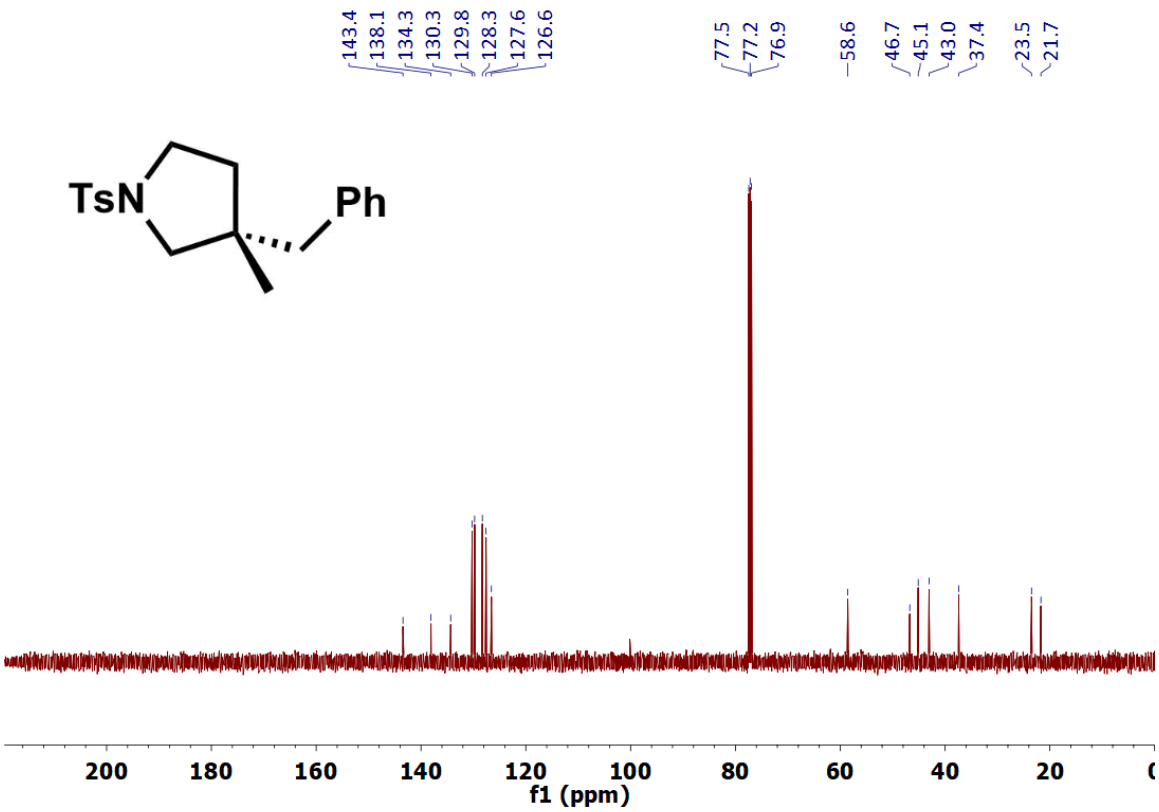
7.70
7.32
7.30
7.27
7.26
7.25
7.24
7.22
7.21
7.20
7.04
7.03
3.40
3.20
3.18
2.91
2.89
2.62
2.59
2.53
2.51
2.43
1.70
1.51
1.46
0.82
TsN
Ph
2.0
2.1
3.3
2.0
1.0
1.1
1.0
1.0
1.0
1.0
3.1
1.0
1.0
3.0
f1 (ppm)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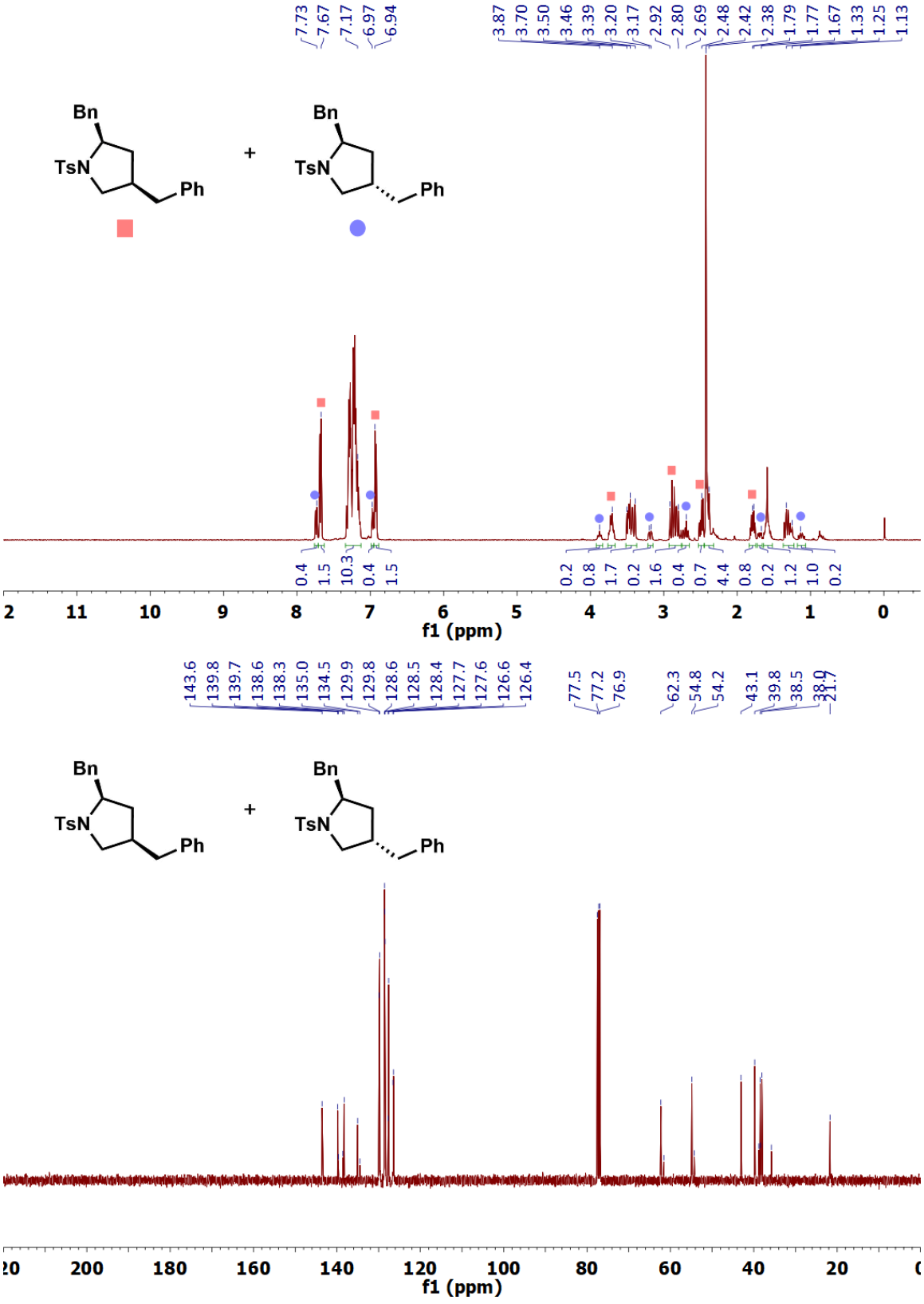
138.1
134.3
130.3
129.8
128.3
127.6
126.6
77.5
77.2
76.9
58.6
46.7
45.1
43.0
37.4
23.5
21.7
TsN
Ph
f1 (ppm)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7.67
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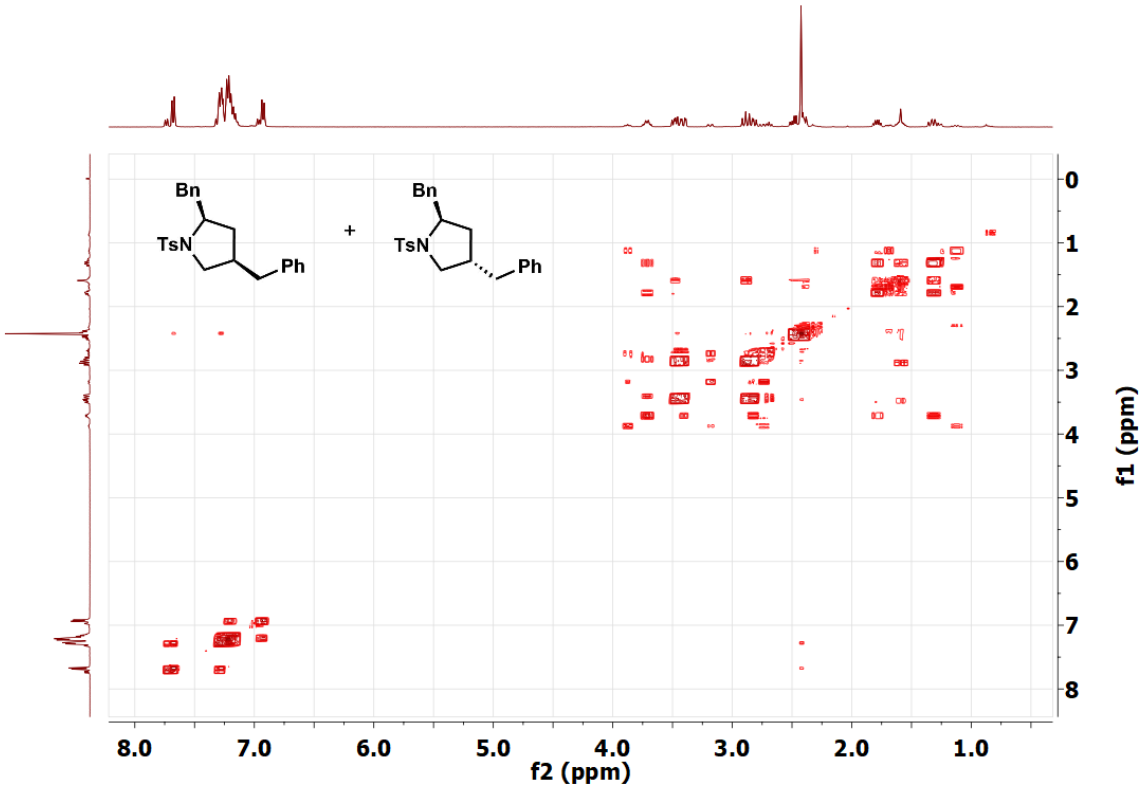
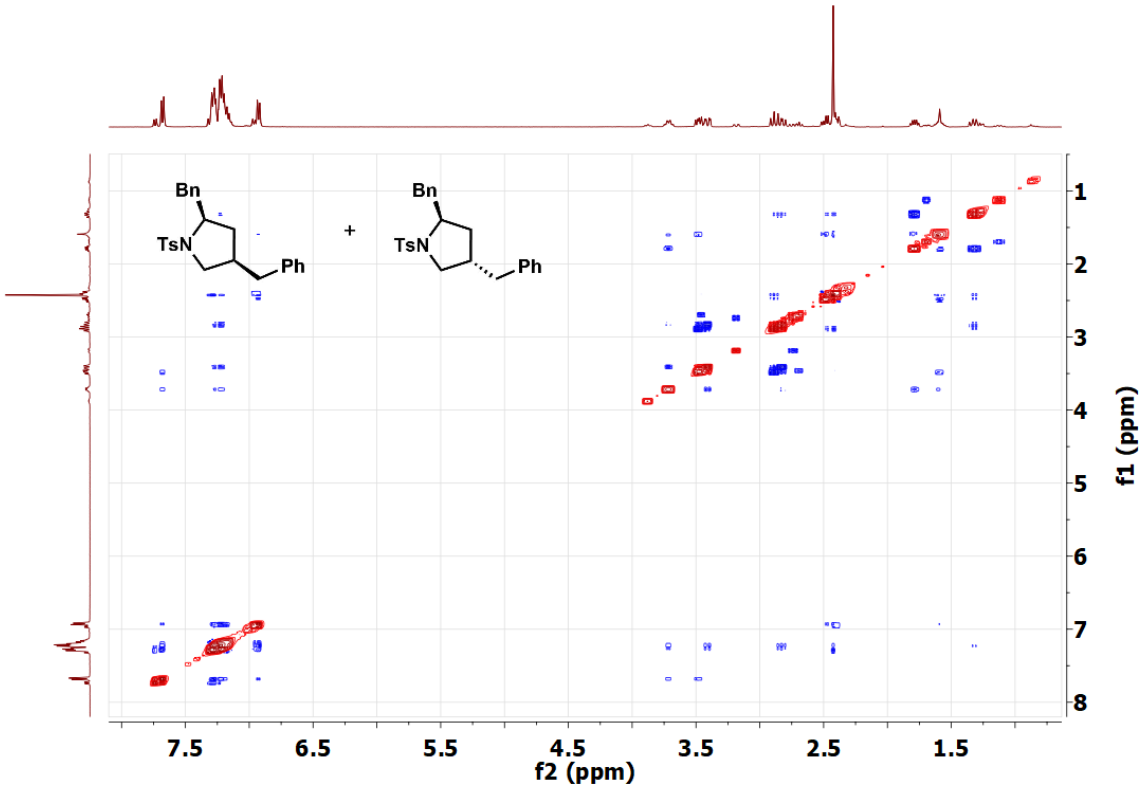
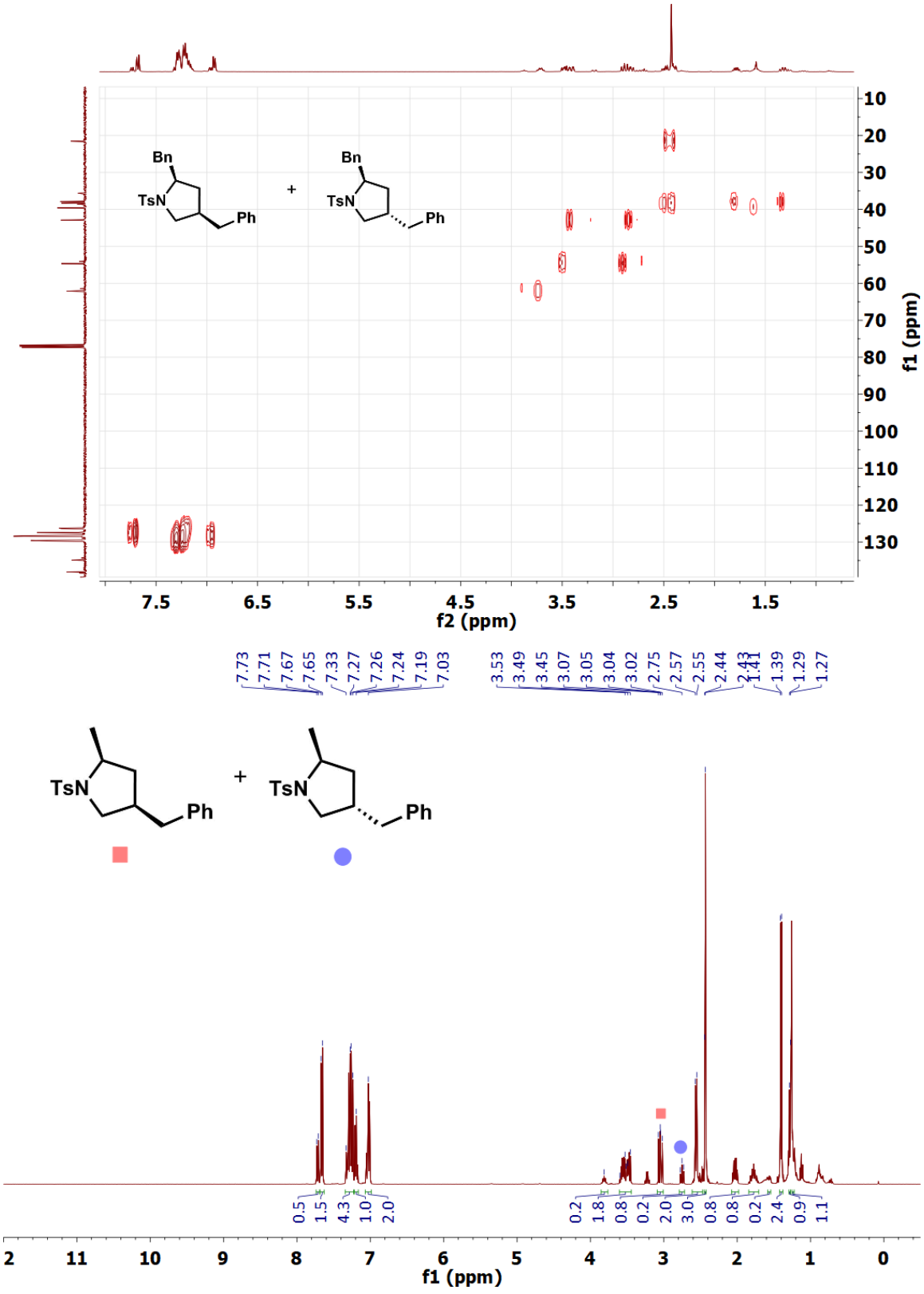
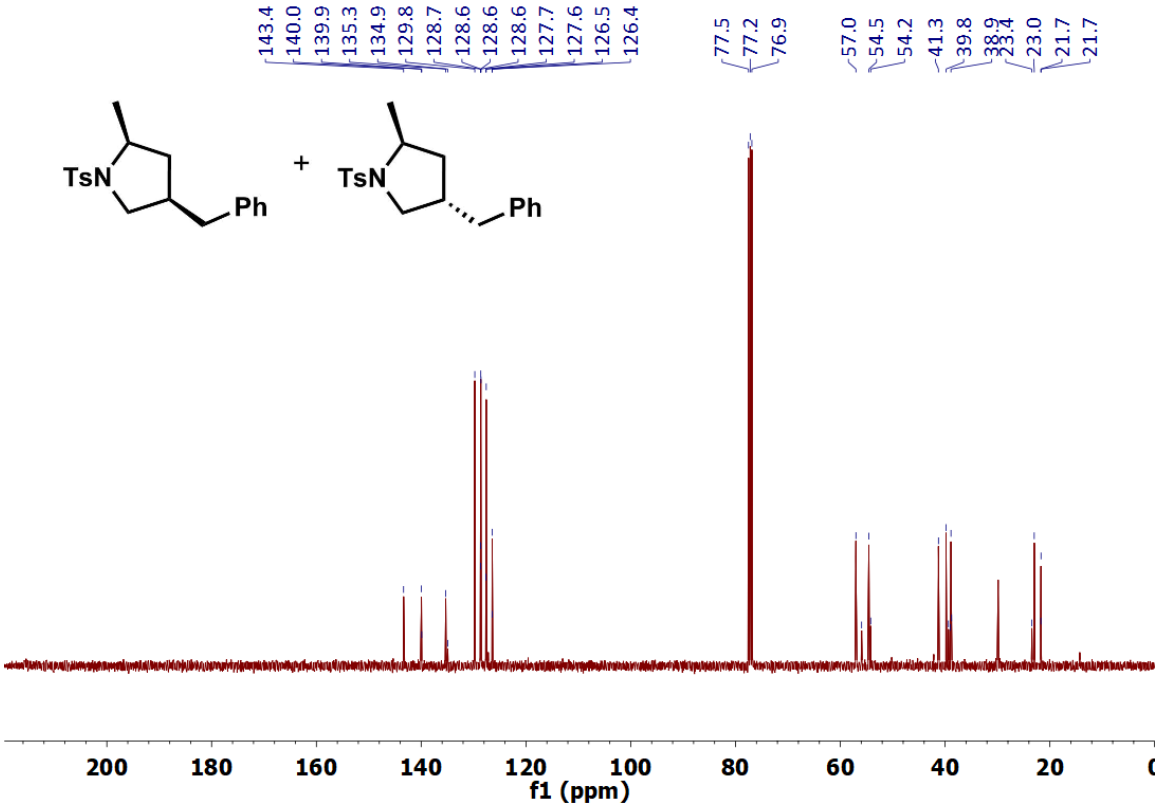
6.94
3.87
3.70
3.50
3.46
3.39
3.20
3.17
2.92
2.80
2.69
2.48
2.42
2.38
1.79
1.77
1.67
1.33
1.25
1.13
Bn
TsN
Ph
+
Bn
TsN
Ph
0.4
1.5
10.3
0.4
1.5
0.2
0.8
1.7
0.2
1.6
0.4
0.7
4.4
0.8
0.2
1.2
1.0
0.2
2
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)
143.6
139.8
139.7
138.6
138.3
135.0
134.5
129.9
129.8
128.6
128.5
128.4
127.7
127.6
126.6
126.4
77.5
77.2
76.9
62.3
54.8
54.2
43.1
39.8
38.5
38.0
21.7
Bn
TsN
Ph
+
Bn
TsN
Ph
200
180
160
140
120
100
80
60
40
20
f1 (ppm)


Bn
TsN
Ph
+
Bn
TsN
Ph
f1 (ppm)
f2 (ppm)


Bn
TsN
Ph
+
Bn
TsN
Ph
f1 (ppm)
f2 (ppm)


Bn
TsN
Ph
+
Bn
TsN
Ph
f1 (ppm)
f2 (ppm)
7.73 7.71 7.67 7.65 7.33 7.27 7.26 7.24 7.19 7.03
3.53 3.49 3.45 3.07 3.05 3.04 3.02 2.75 2.57 2.55 2.44 2.43 1.41 1.39 1.29 1.27
TsN
Ph
+
TsN
Ph
0.5 1.5 4.3 1.0 2.0
0.2 1.8 0.8 0.2 2.0 3.0 0.8 0.8 0.2 2.4 0.9 1.1
f1 (ppm)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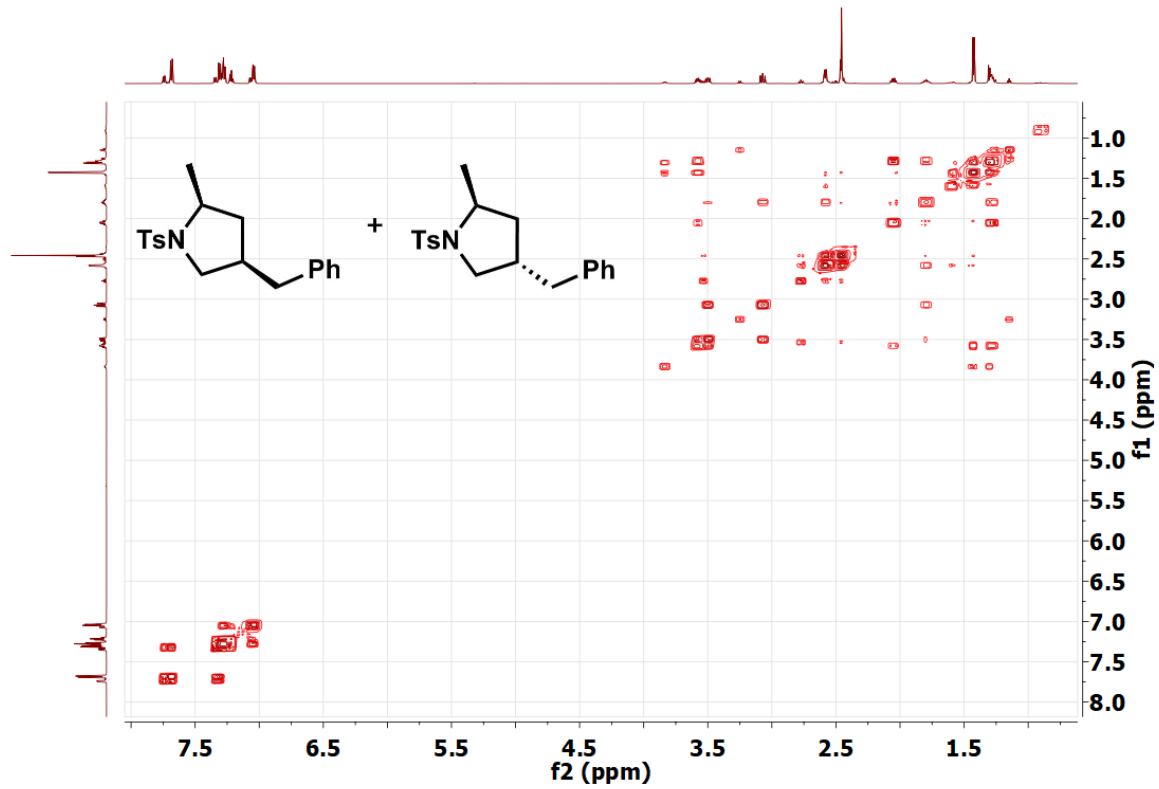
139.9
135.3
134.9
129.8
128.7
128.6
128.6
128.6
127.7
127.6
126.5
126.4
77.5
77.2
76.9
57.0
54.5
54.2
41.3
39.8
38.9
23.4
23.0
21.7
21.7
TsN
Ph
+
TsN
Ph
200
180
160
140
120
100
80
60
40
20
f1 (ppm)


TsN
Ph
+
TsN
Ph
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
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7.5
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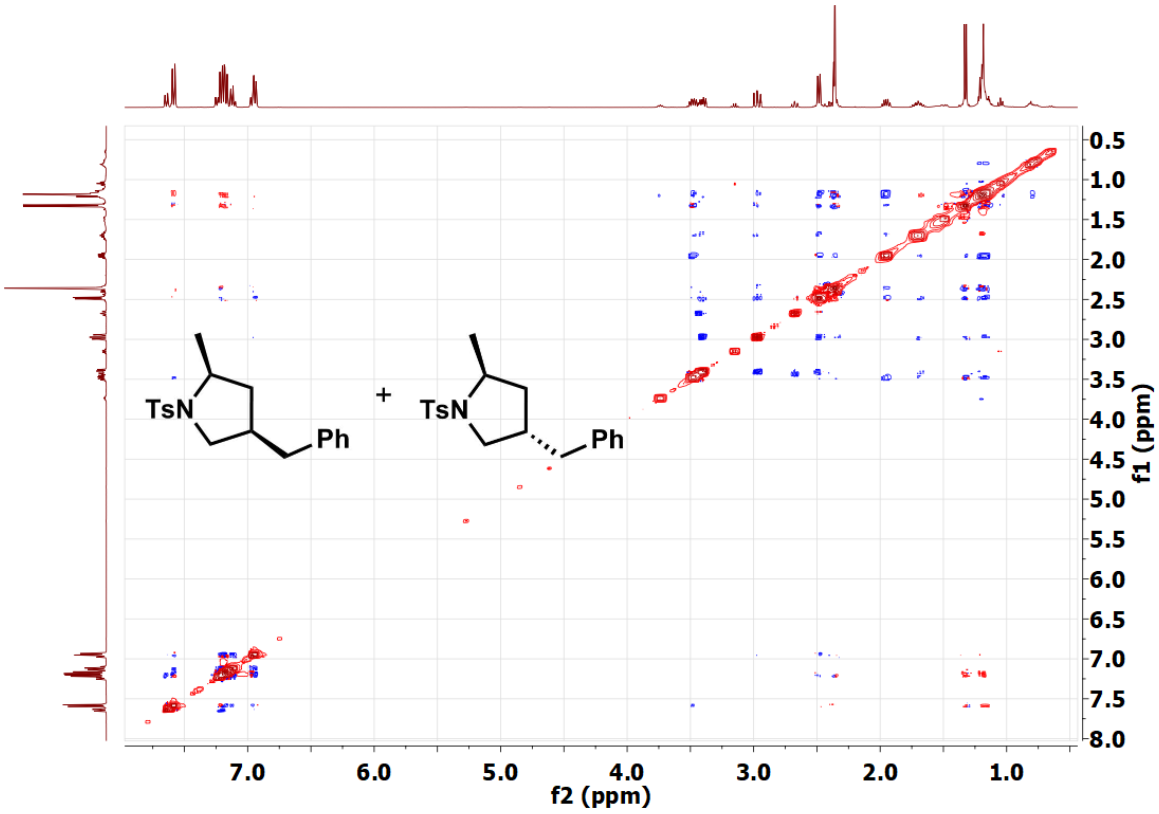
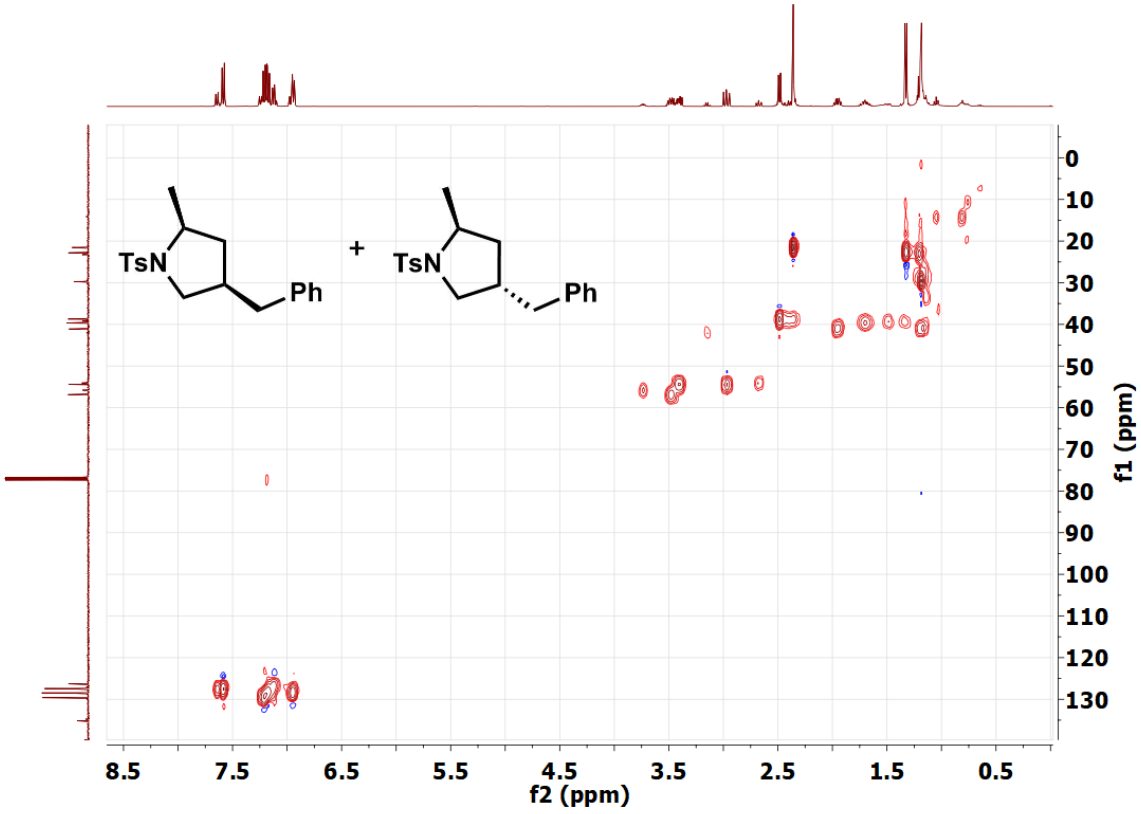
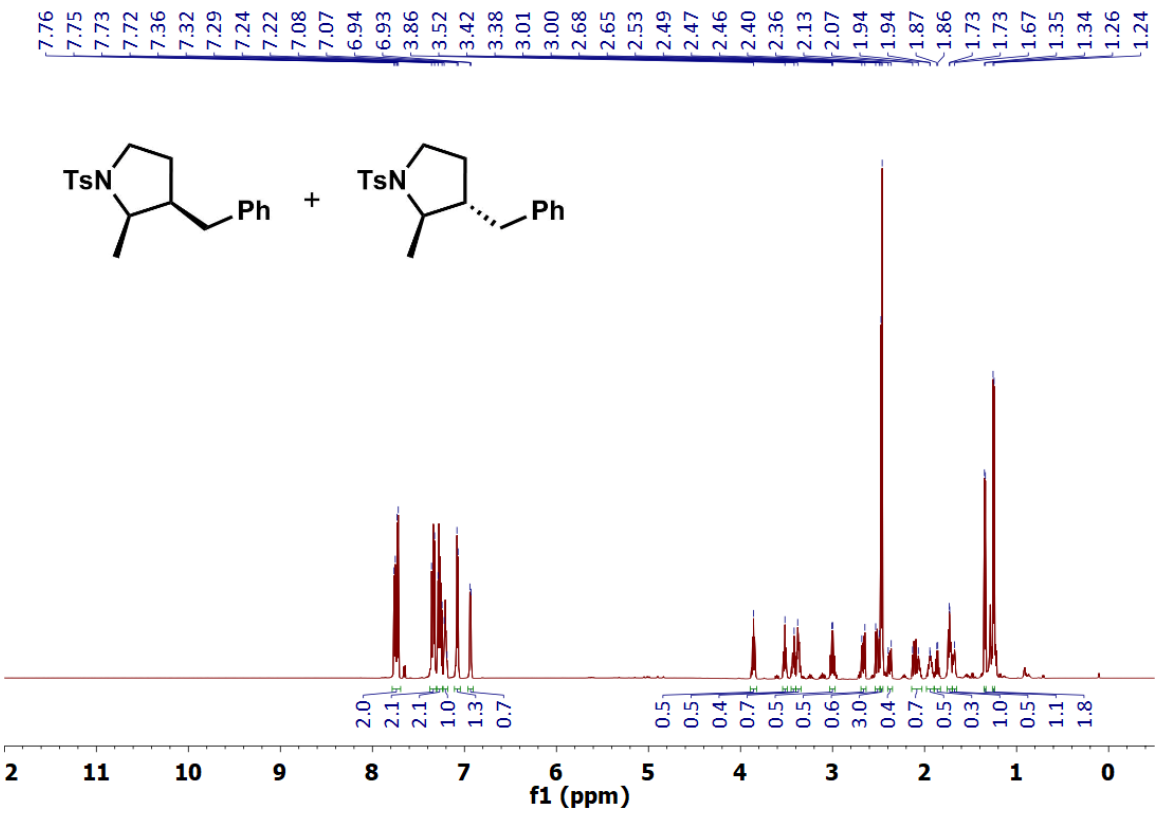
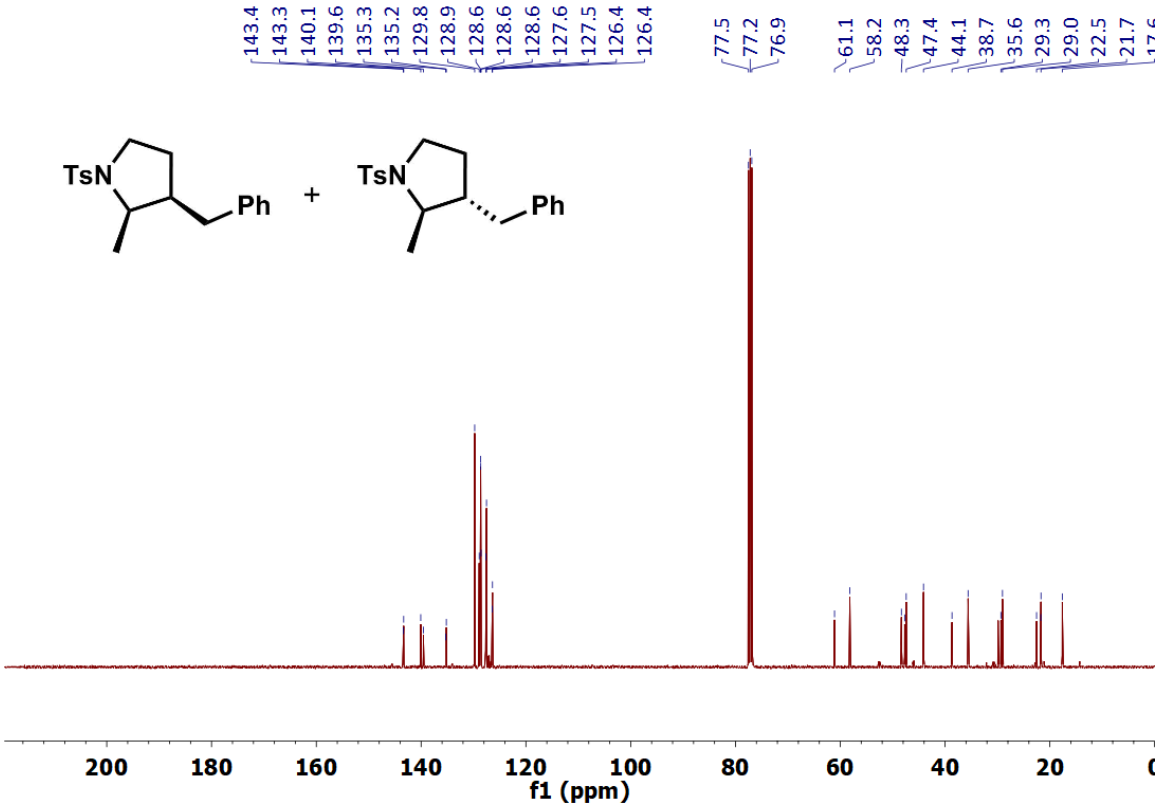
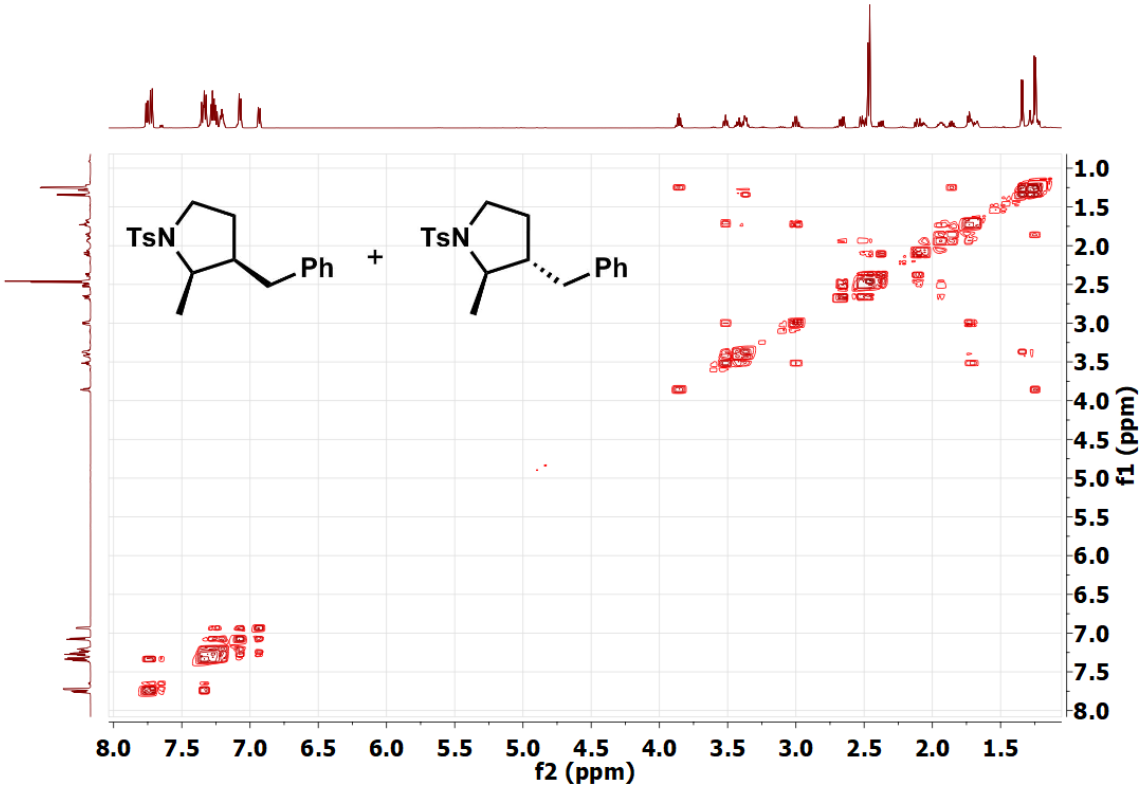
4.5
3.5
2.5
1.5
f2 (ppm)

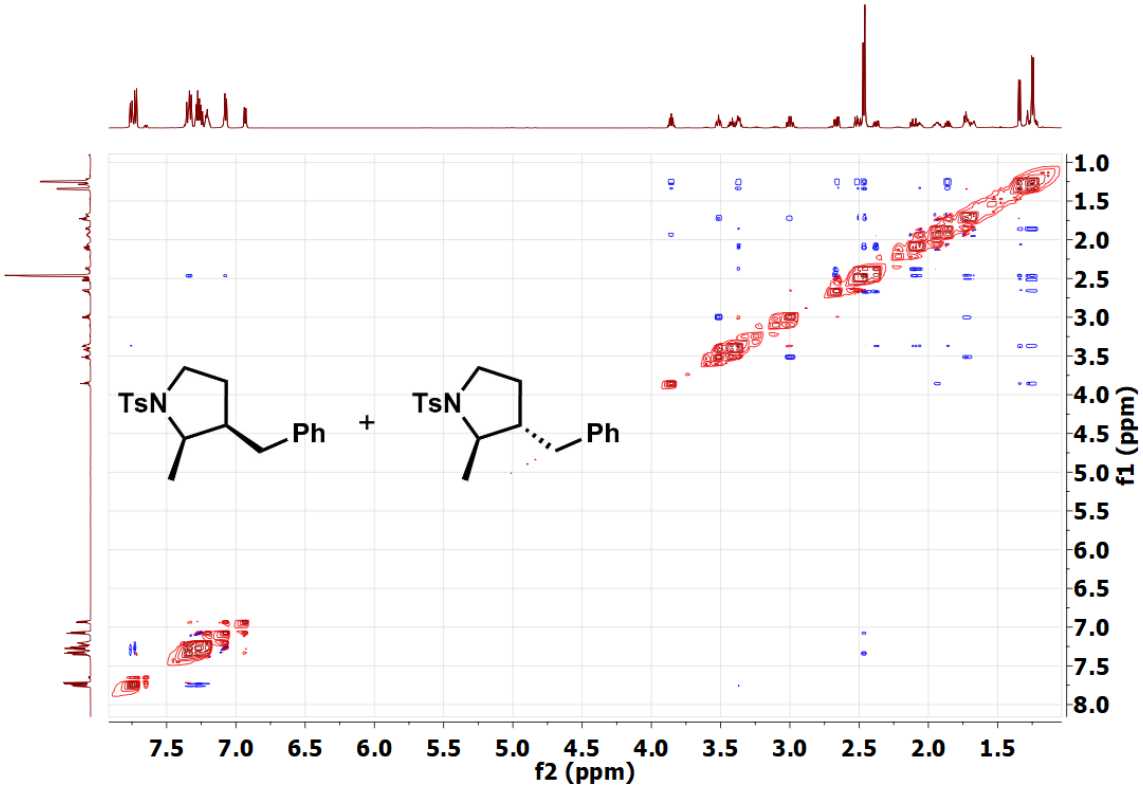

TsN
Ph
+
TsN
Ph
f1 (ppm)
f2 (ppm)

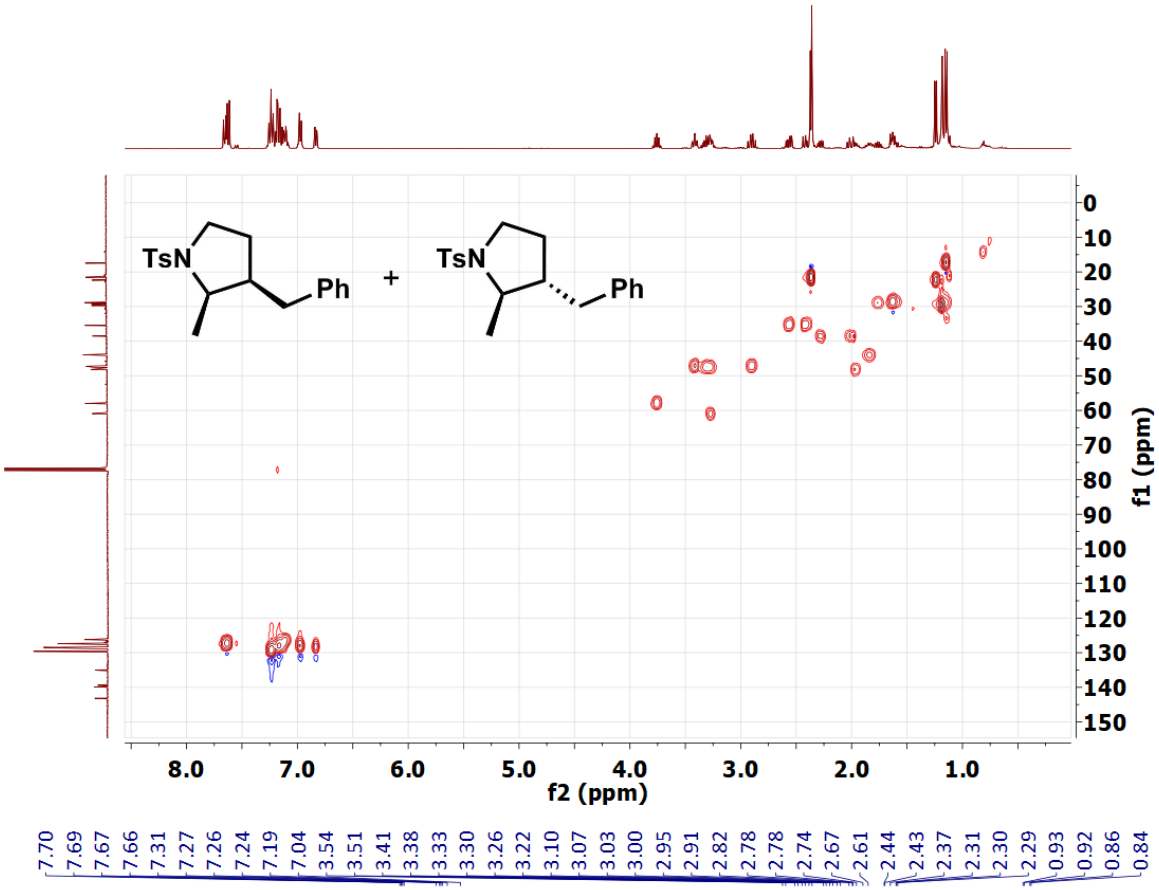

TsN
Ph
+
TsN
Ph
f1 (ppm)
f2 (ppm)

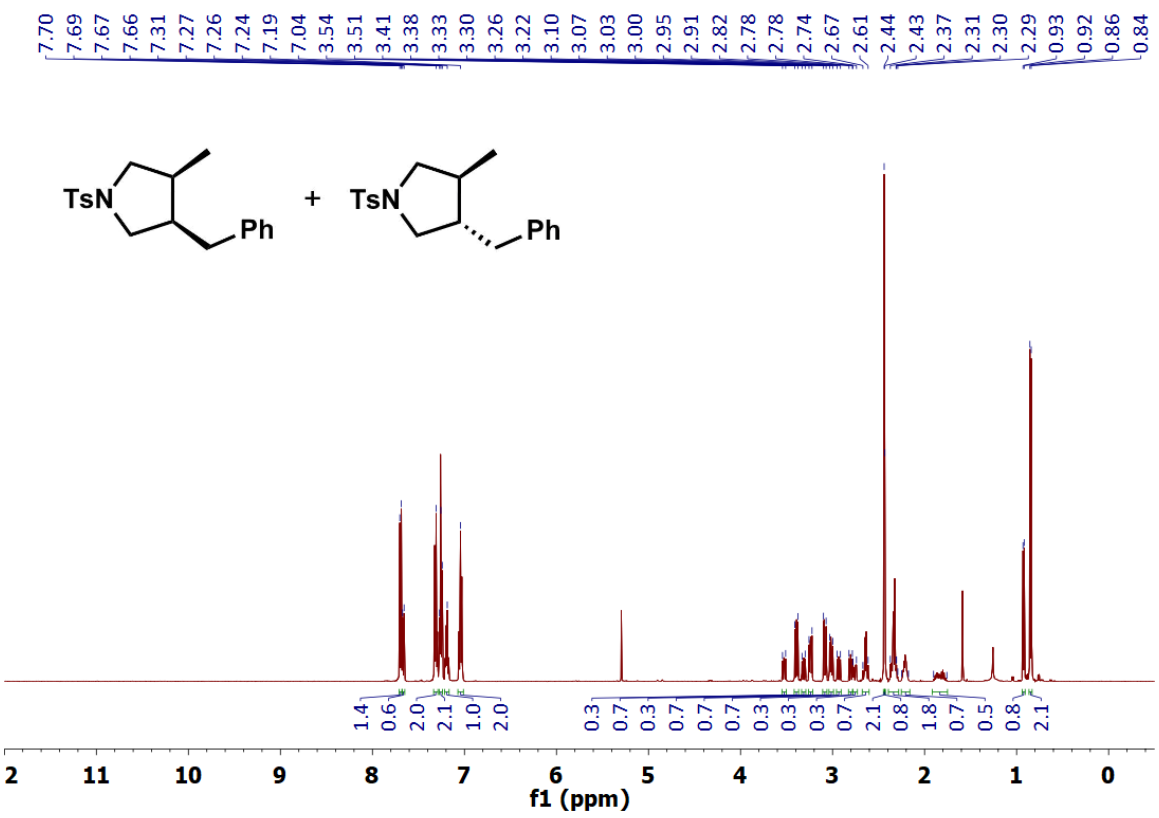

7.76 7.75 7.73 7.72 7.36 7.32 7.29 7.24 7.22 7.08 7.07 6.94 6.93 3.86 3.52 3.42 3.38 3.01 3.00 2.68 2.65 2.53 2.49 2.47 2.46 2.40 2.36 2.13 2.07 1.94 1.94 1.87 1.86 1.73 1.73 1.67 1.35 1.34 1.26 1.24
TsN
Ph
+
TsN
Ph
2.0 2.1 2.1 1.0 1.3 0.7 0.5 0.5 0.4 0.7 0.5 0.5 0.6 3.0 0.4 0.7 0.5 0.3 1.0 0.5 1.1 1.8
2 11 10 9 8 7 6 5 4 3 2 1 0
f1 (ppm)

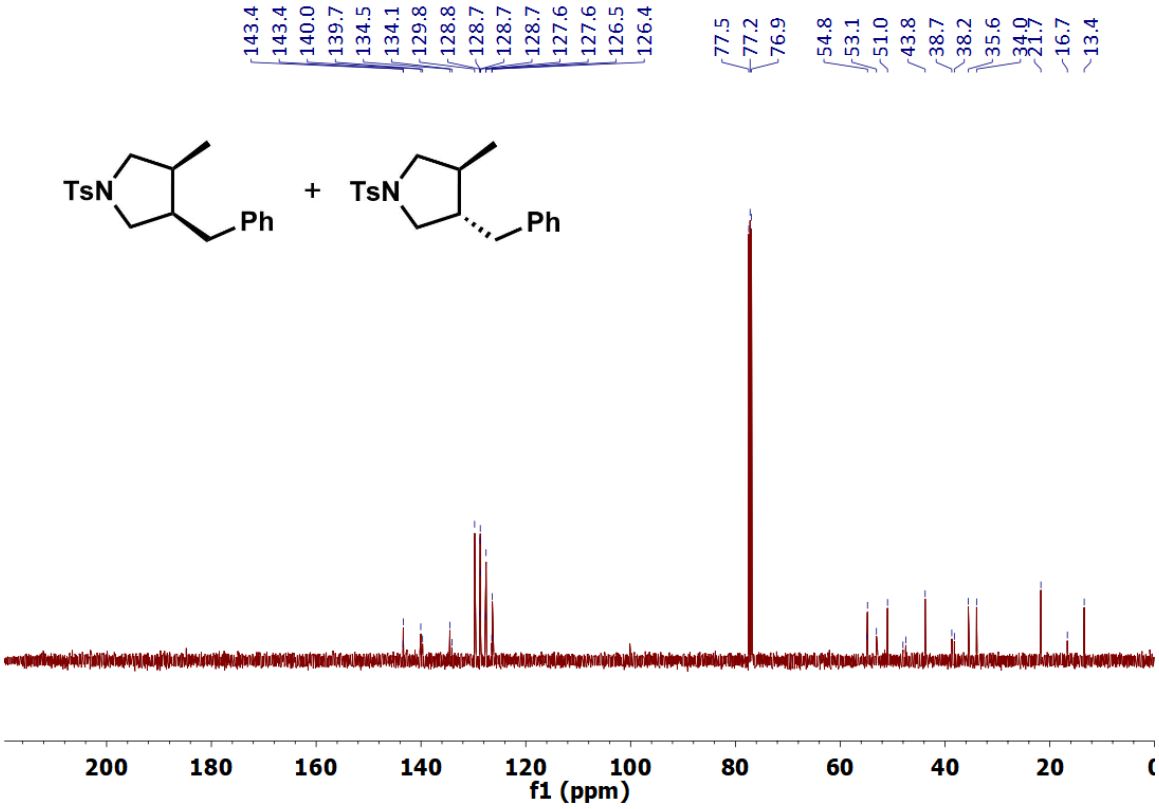

143.4 143.3 140.1 139.6 135.3 135.2 129.8 128.9 128.6 128.6 128.6 127.6 127.5 126.4 126.4 77.5 77.2 76.9 61.1 58.2 48.3 47.4 44.1 38.7 35.6 29.3 29.0 22.5 21.7
TsN
Ph
+
TsN
Ph
200 180 160 140 120 100 80 60 40 20
f1 (ppm)


TsN
Ph
+
TsN
Ph
f1 (ppm)
f2 (ppm)


TsN
Ph
+
TsN
Ph
f1 (ppm)
f2 (ppm)


TsN
Ph
+
TsN
Ph
f1 (ppm)
f2 (ppm)


TsN
Ph
+
TsN
Ph
f1 (ppm)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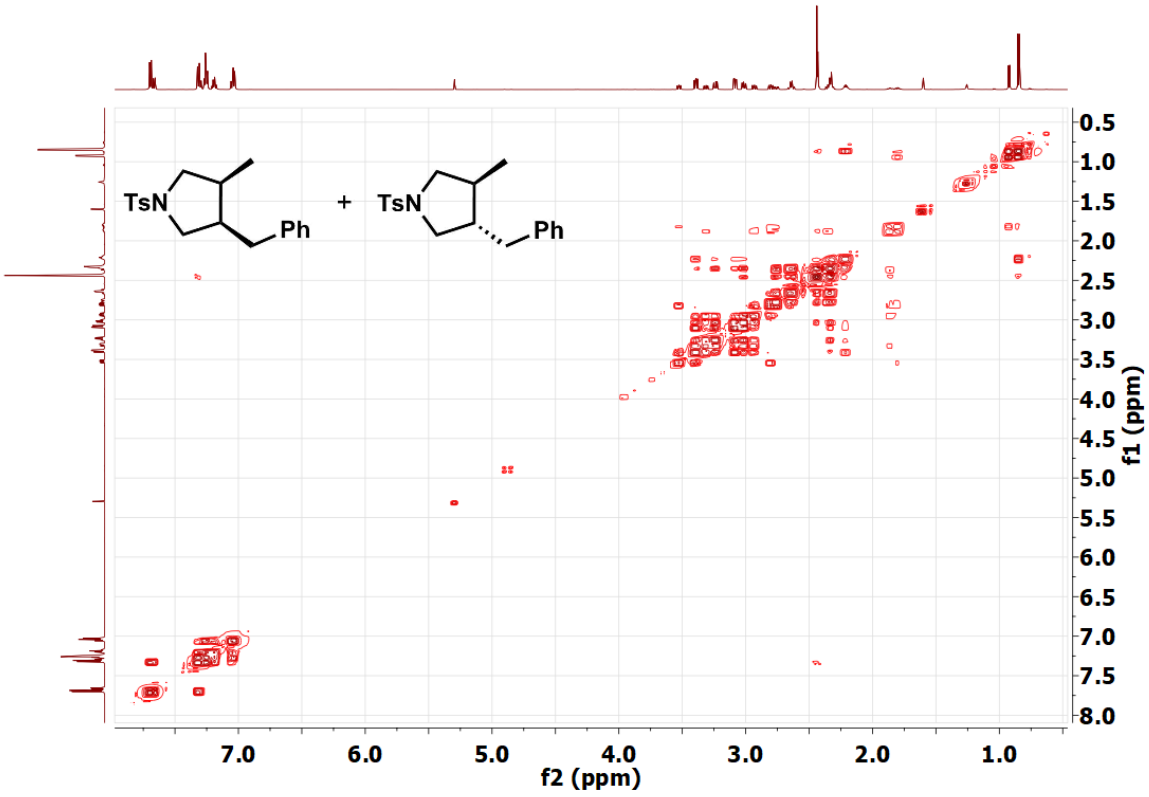
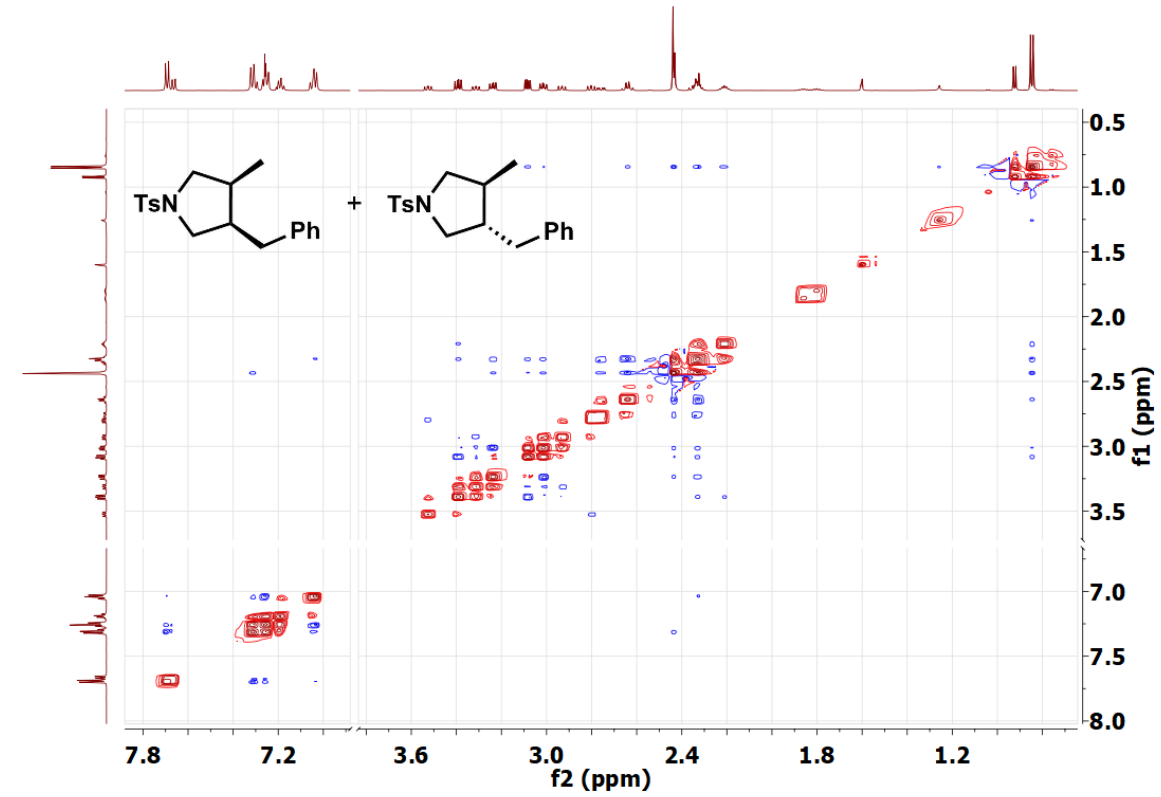
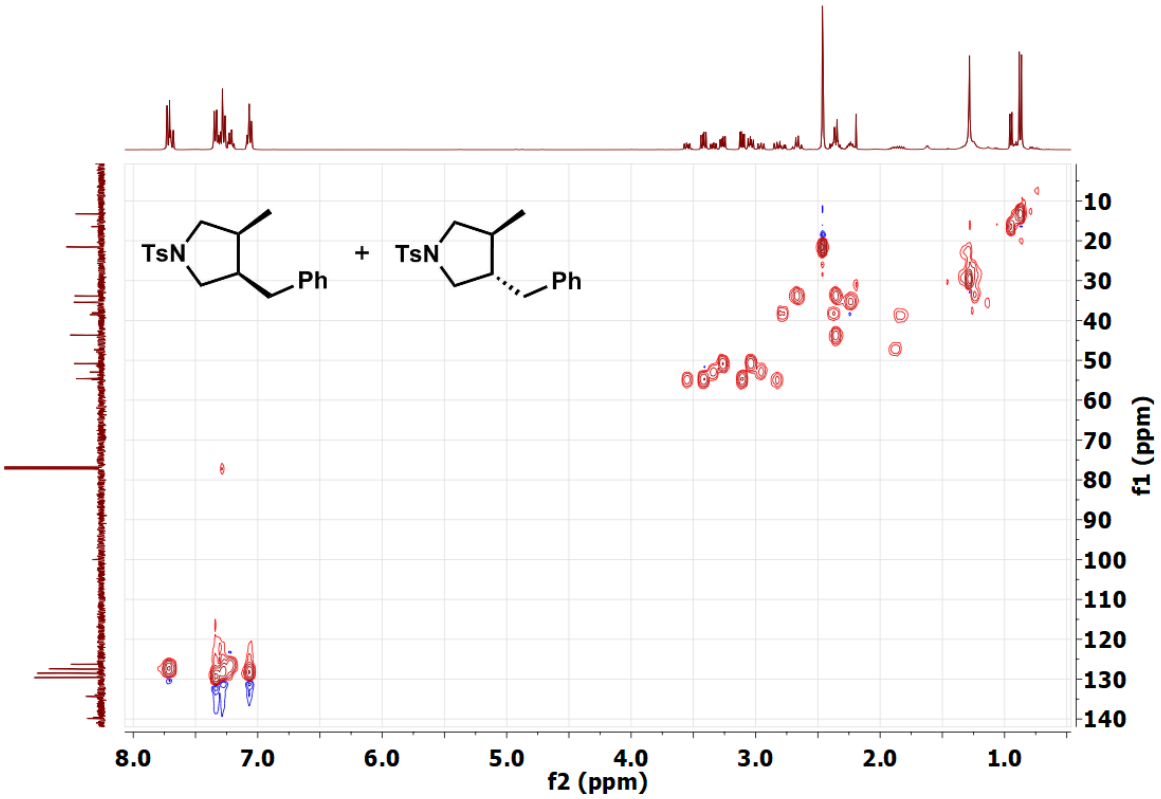
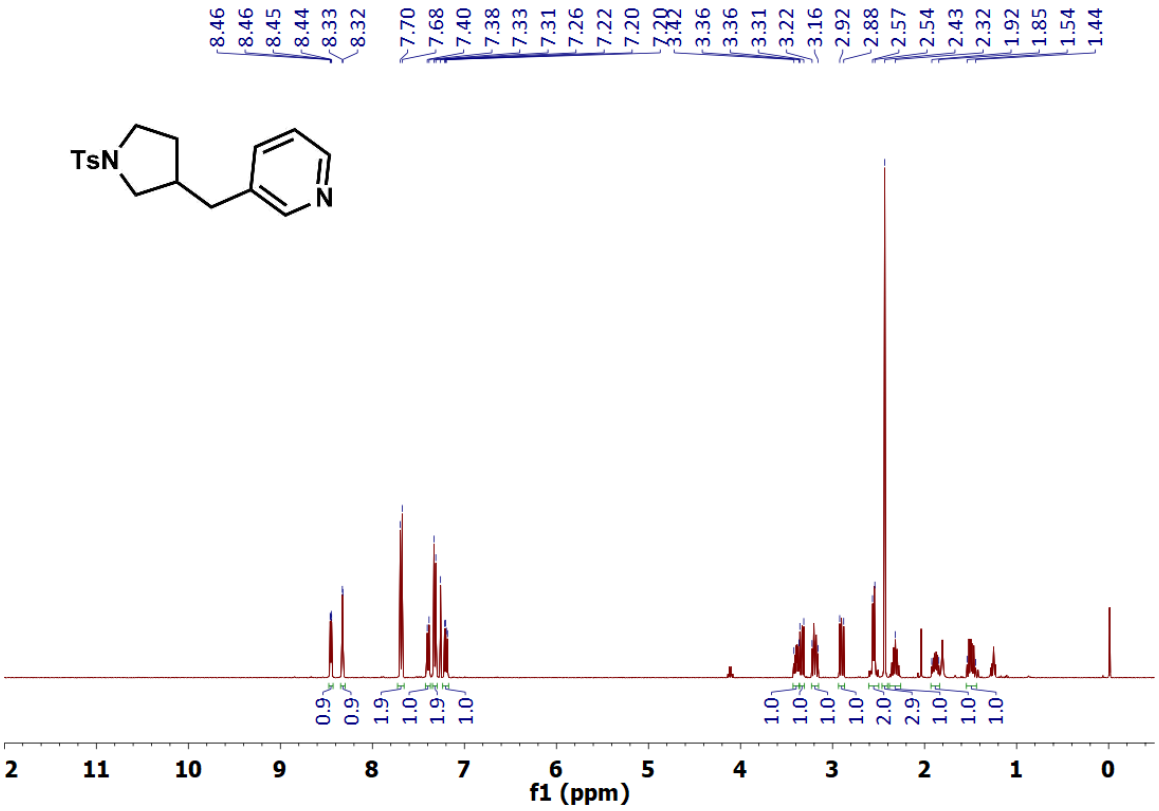
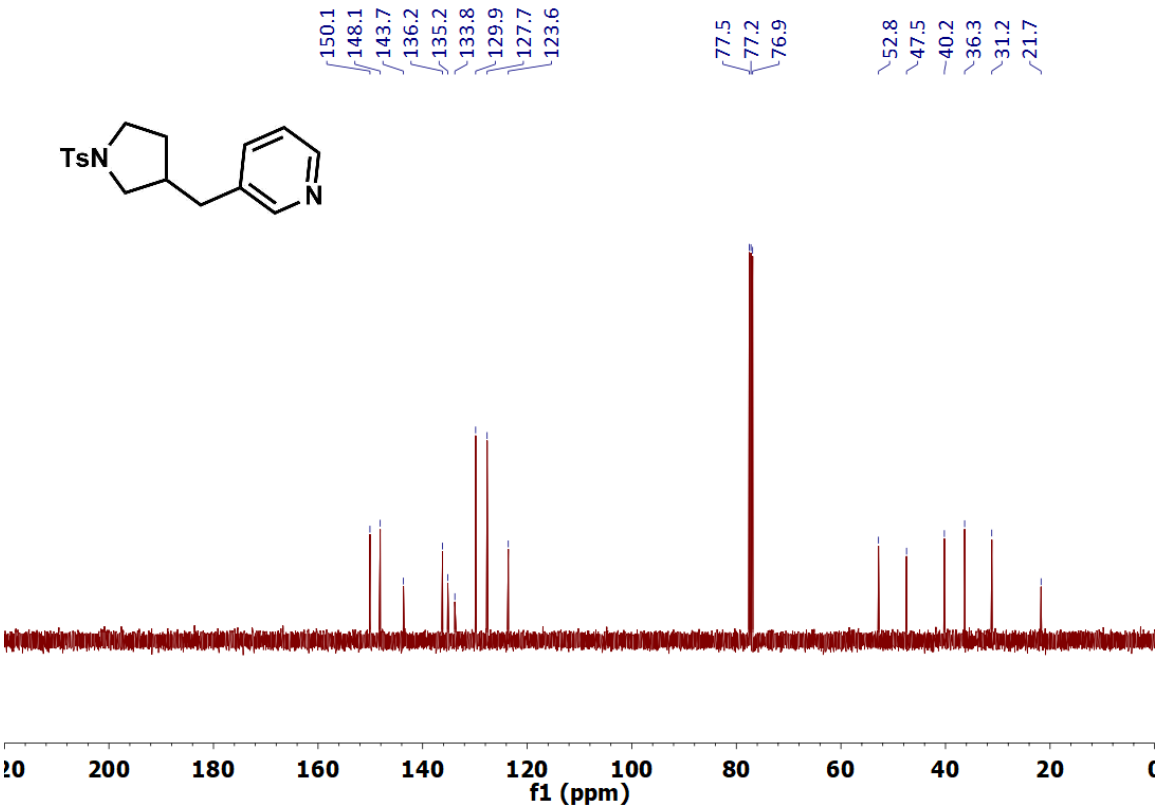
143.4
140.0
139.7
134.5
134.1
129.8
128.8
128.7
128.7
128.7
127.6
127.6
126.5
126.4
77.5
77.2
76.9
54.8
53.1
51.0
43.8
38.7
38.2
35.6
34.0
21.7
16.7
13.4
TsN
Ph
+
TsN
Ph
200
180
160
140
120
100
80
60
40
20
f1 (ppm)

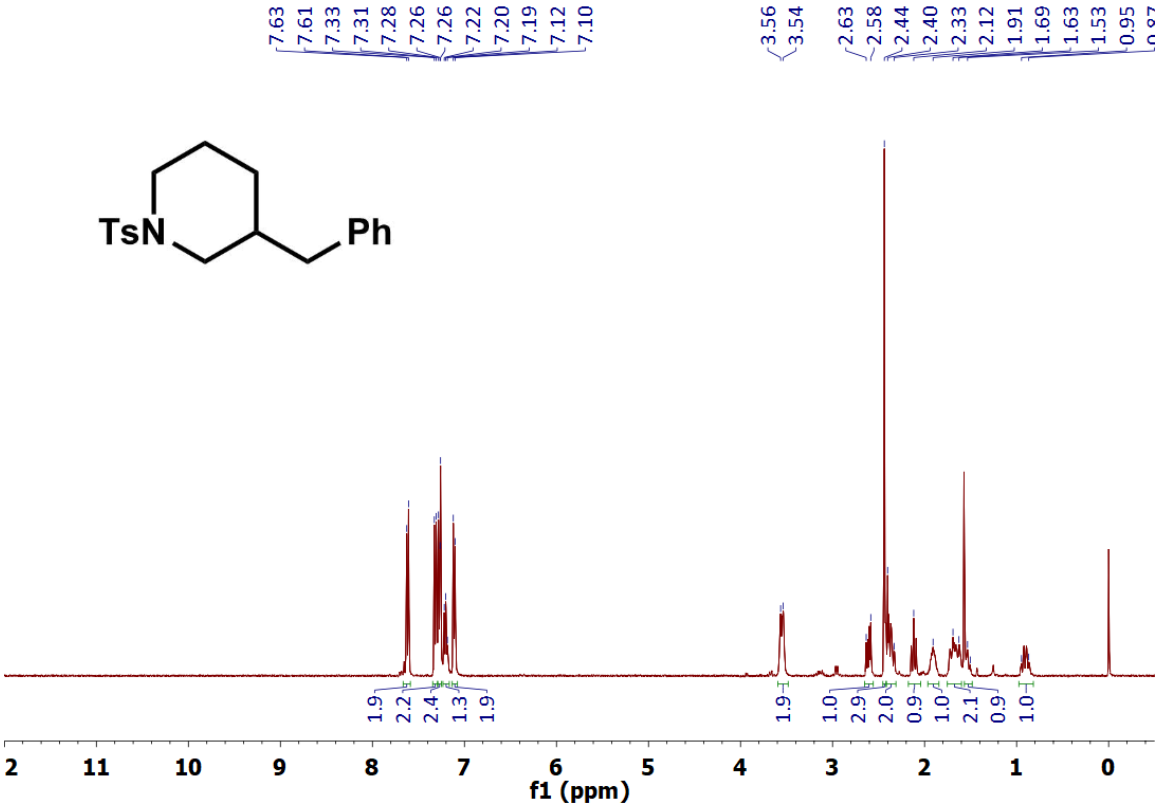

TsN
Ph
+
TsN
Ph
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
f1 (ppm)
7.0
6.0
5.0
4.0
3.0
2.0
1.0
f2 (ppm)

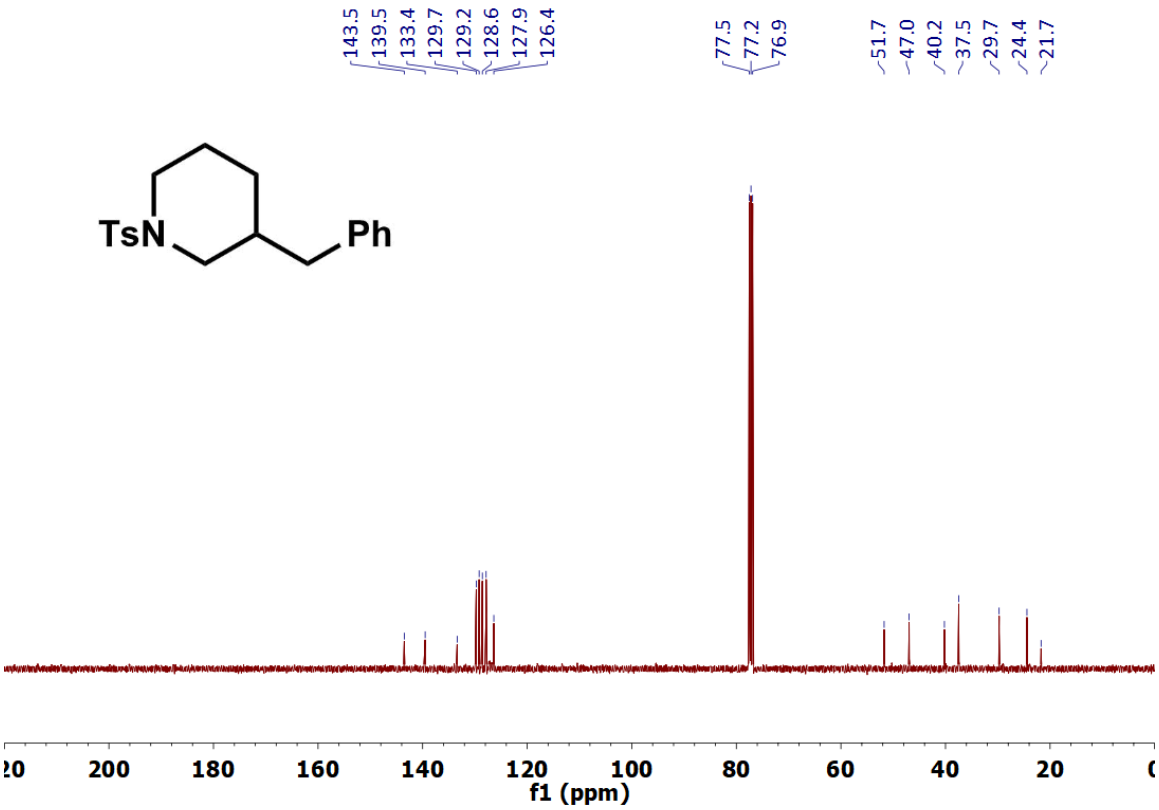

TsN
Ph
+
TsN
Ph
0.5
1.0
1.5
2.0
2.5
3.0
3.5
7.0
7.5
8.0
f1 (ppm)
7.8
7.2
3.6
3.0
2.4
1.8
1.2
f2 (ppm)

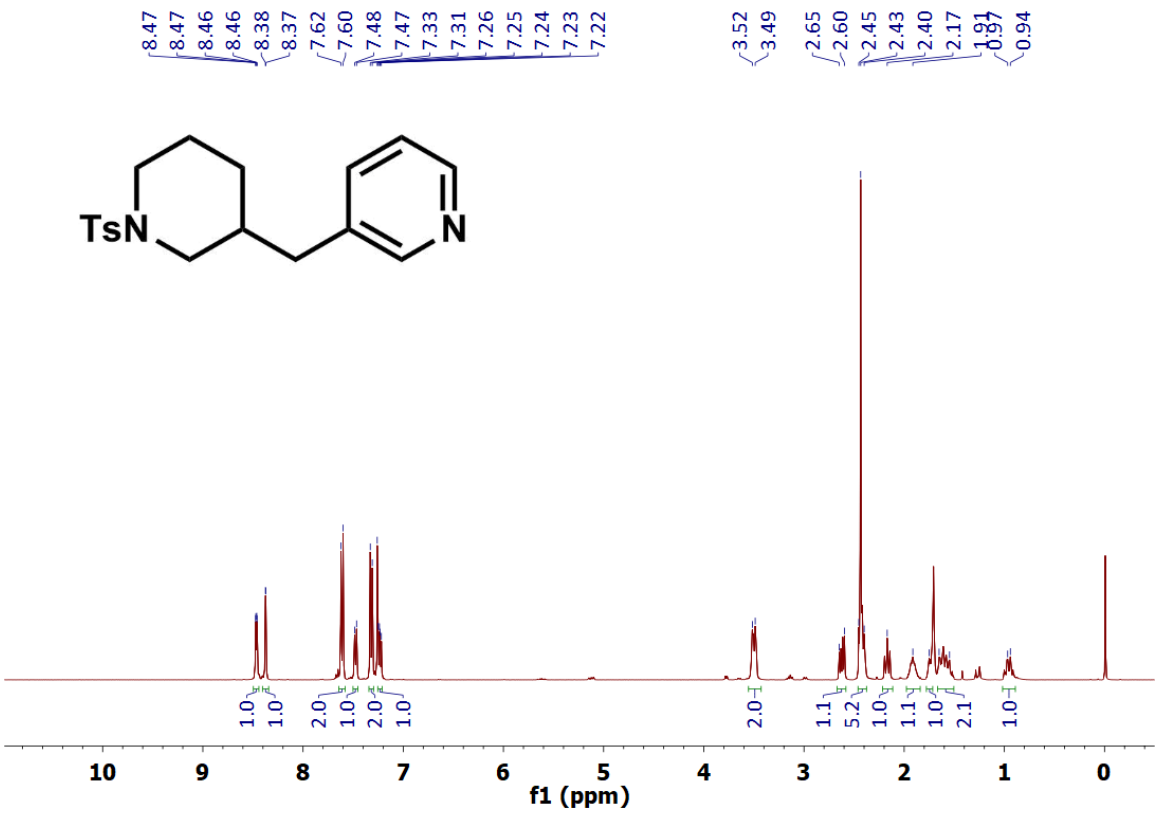

TsN
Ph
+
TsN
Ph
10
20
30
40
50
60
70
80
90
100
110
120
130
140
f1 (ppm)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
f2 (ppm)

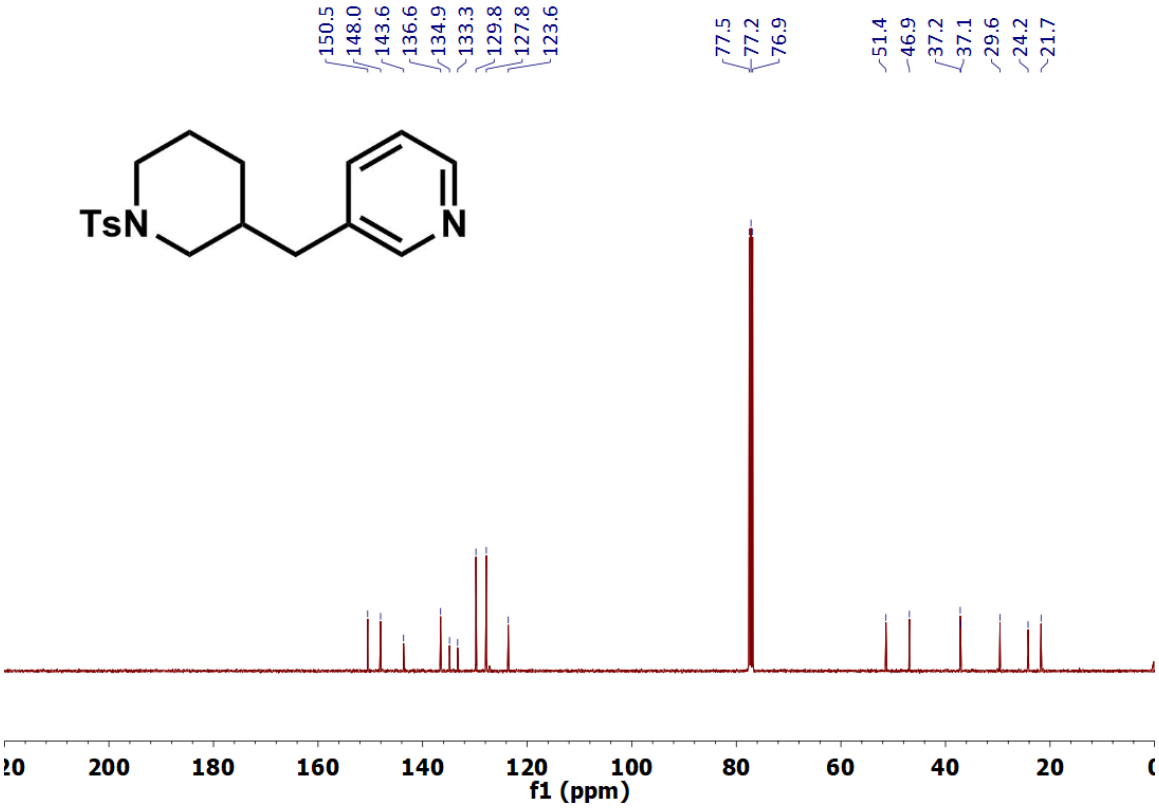

8.46
8.46
8.45
8.44
8.33
8.32
7.70
7.68
7.40
7.38
7.33
7.31
7.26
7.22
7.20
3.42
3.36
3.36
3.31
3.22
3.16
2.92
2.88
2.57
2.54
2.43
2.32
1.92
1.85
1.54
1.44
TsN
N
0.9
0.9
1.9
1.0
1.9
1.0
1.0
1.0
1.0
1.0
2.0
2.9
1.0
1.0
1.0
2
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

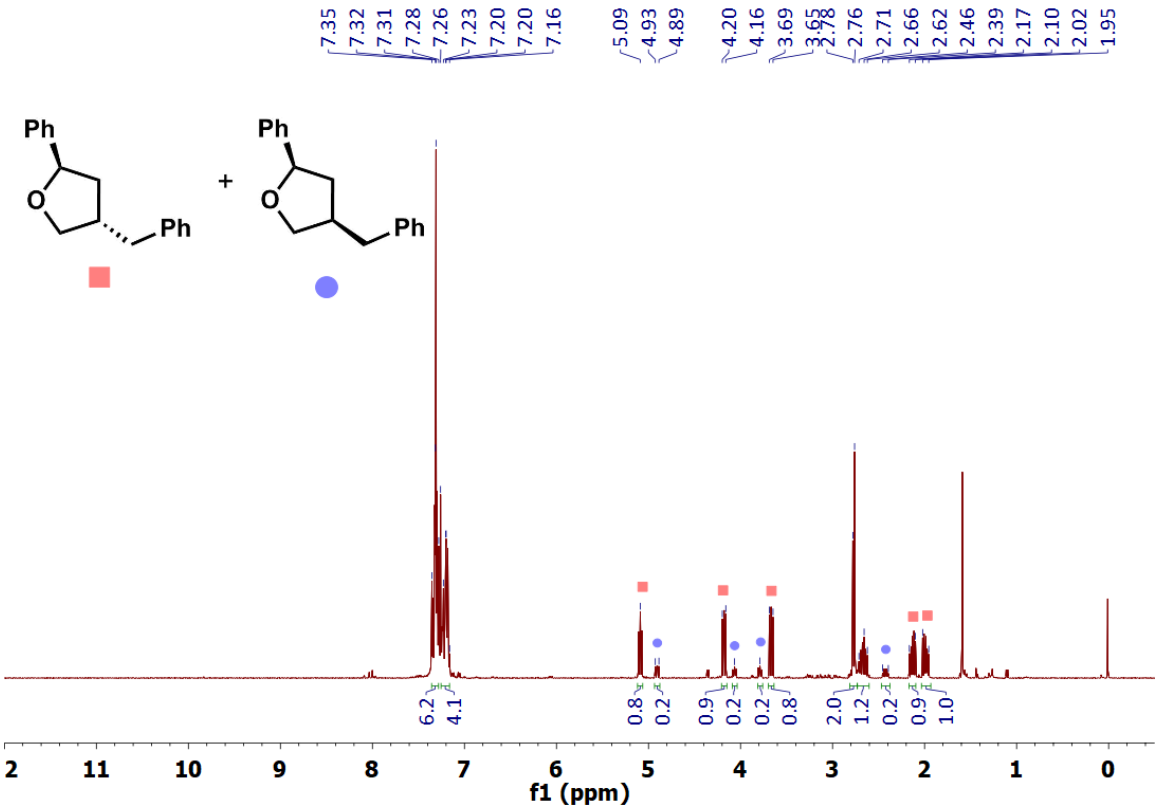

150.1
148.1
143.7
136.2
135.2
133.8
129.9
127.7
123.6
77.5
77.2
76.9
52.8
47.5
40.2
36.3
31.2
21.7
TsN
N
200
180
160
140
120
100
80
60
40
20
f1 (ppm)

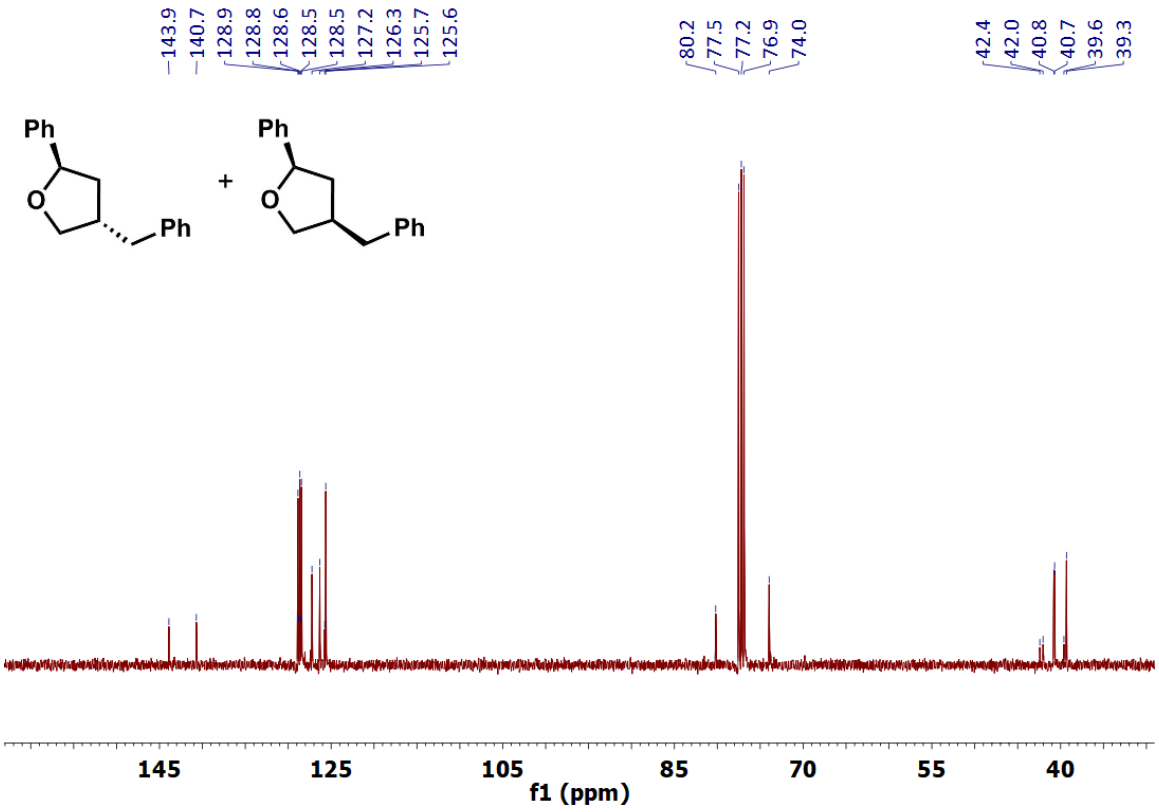

7.63
7.61
7.33
7.31
7.28
7.26
7.26
7.22
7.20
7.19
7.12
7.10
3.56
3.54
2.63
2.58
2.44
2.40
2.33
2.12
1.91
1.69
1.63
1.53
0.95
TsN
Ph
1.9
2.2
2.4
1.3
1.9
1.9
1.0
2.9
2.0
0.9
1.0
2.1
0.9
1.0
f1 (ppm)

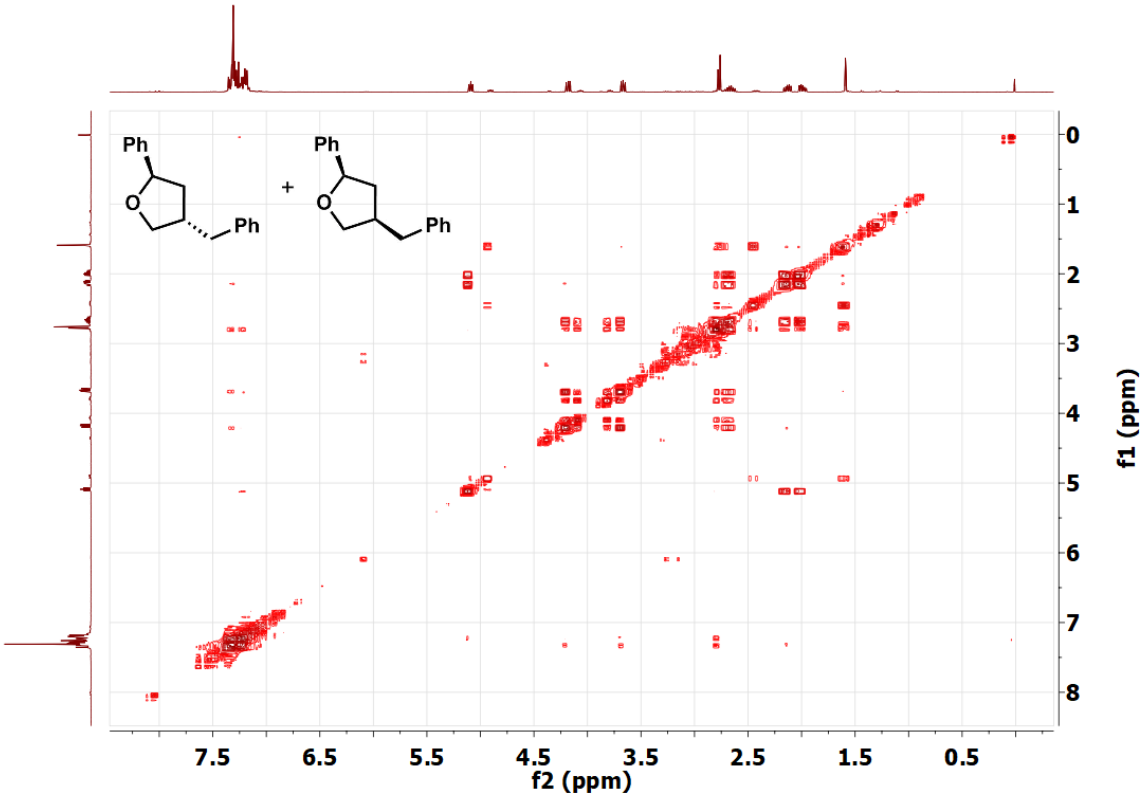

143.5
139.5
133.4
129.7
129.2
128.6
127.9
126.4
77.5
77.2
76.9
51.7
47.0
40.2
37.5
29.7
24.4
21.7
TsN
Ph
f1 (ppm)

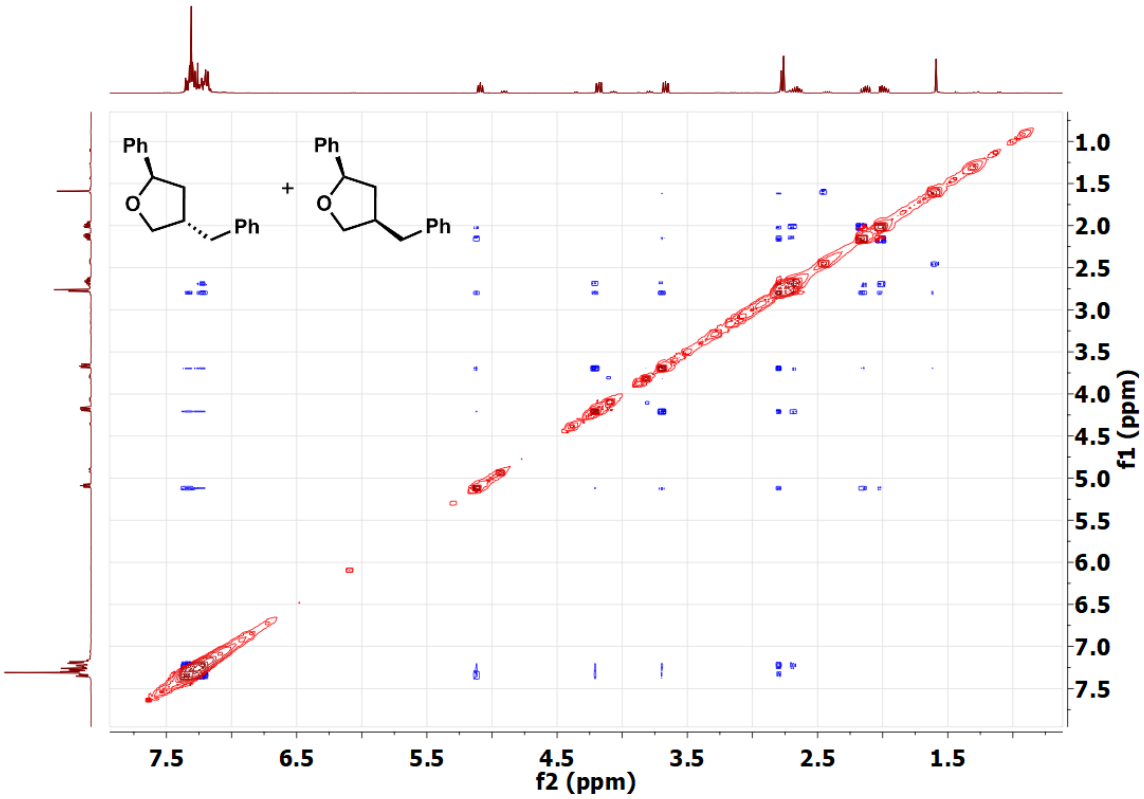

8.47
8.47
8.46
8.46
8.38
8.37
7.62
7.60
7.48
7.47
7.33
7.31
7.26
7.25
7.24
7.23
7.22
3.52
3.49
2.65
2.60
2.45
2.43
2.40
2.17
1.91
0.97
0.94
TsN
N
1.0
1.0
2.0
1.0
2.0
1.0
2.0
1.1
5.2
1.0
1.1
1.0
2.1
1.0
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

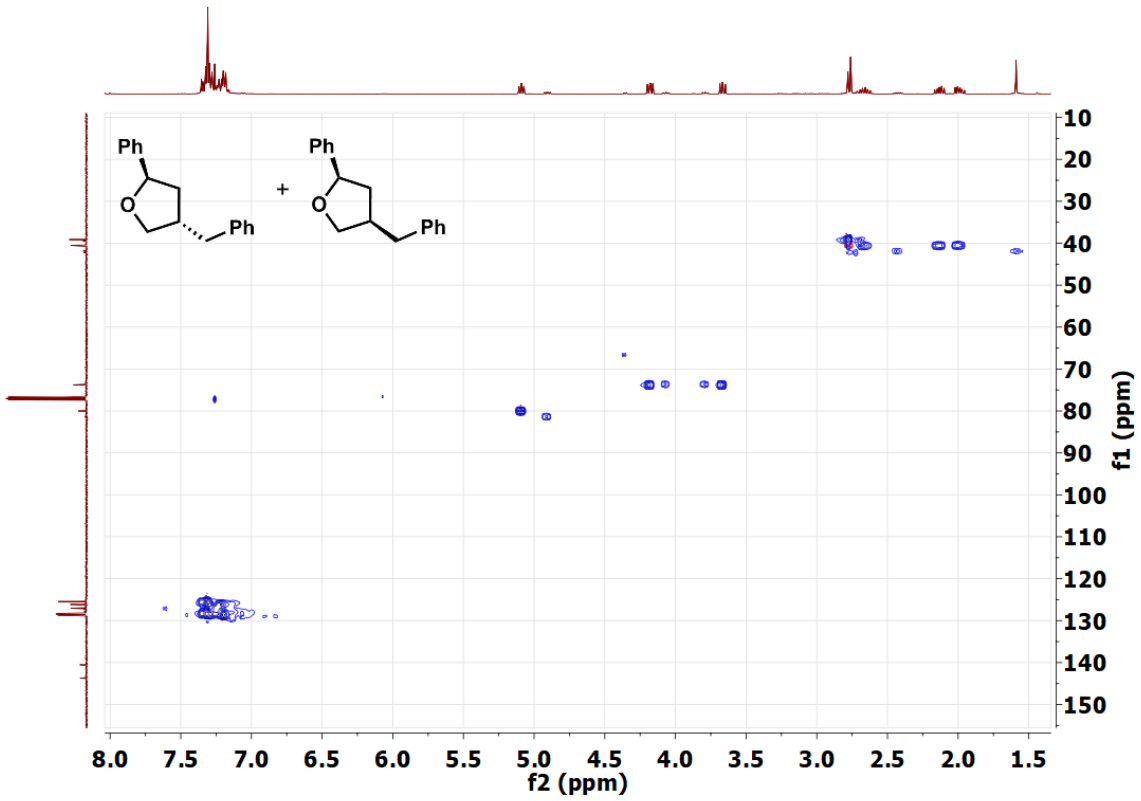

150.5
148.0
143.6
136.6
134.9
133.3
129.8
127.8
123.6
77.5
77.2
76.9
51.4
46.9
37.2
37.1
29.6
24.2
21.7
TsN
N
200
180
160
140
120
100
80
60
40
20
f1 (ppm)

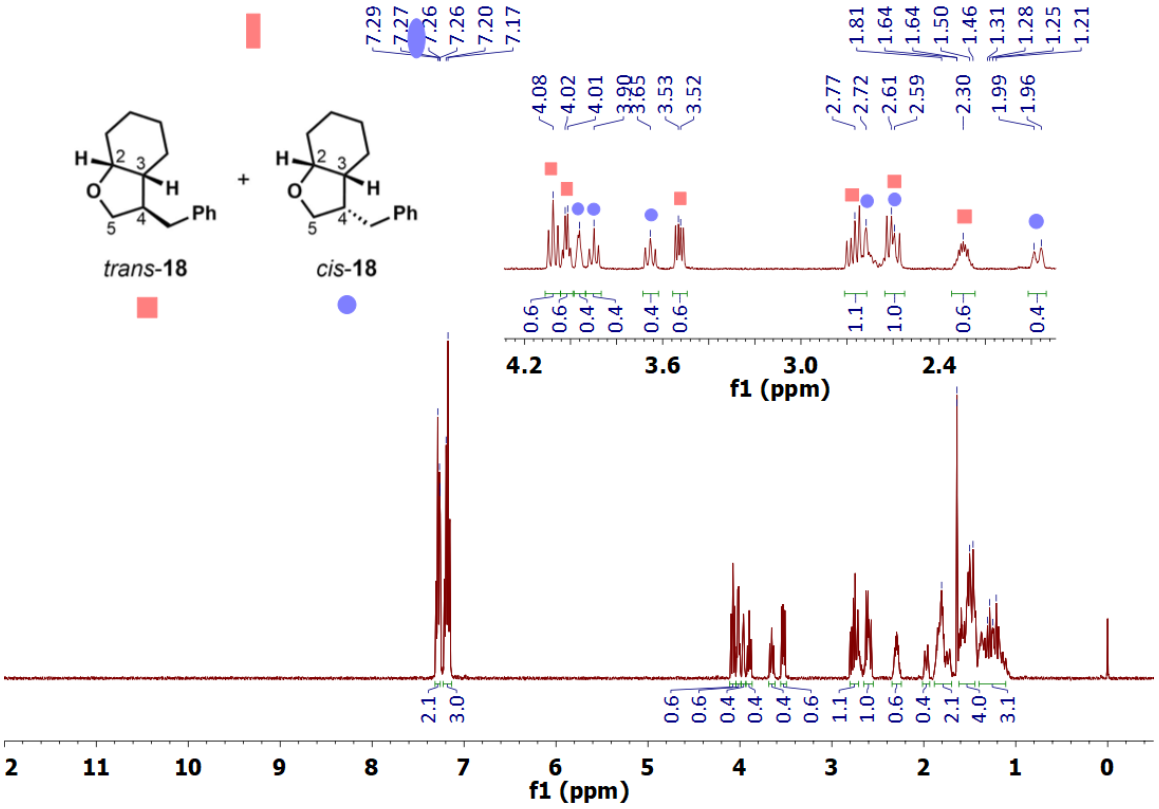

7.35
7.32
7.31
7.28
7.26
7.23
7.20
7.20
7.16
5.09
4.93
4.89
4.20
4.16
3.69
3.65
2.78
2.76
2.71
2.66
2.62
2.46
2.39
2.17
2.10
2.02
1.95
Ph
O
Ph
+
Ph
O
Ph
6.2
4.1
0.8
0.2
0.9
0.2
0.2
0.8
2.0
1.2
0.2
0.9
1.0
2
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

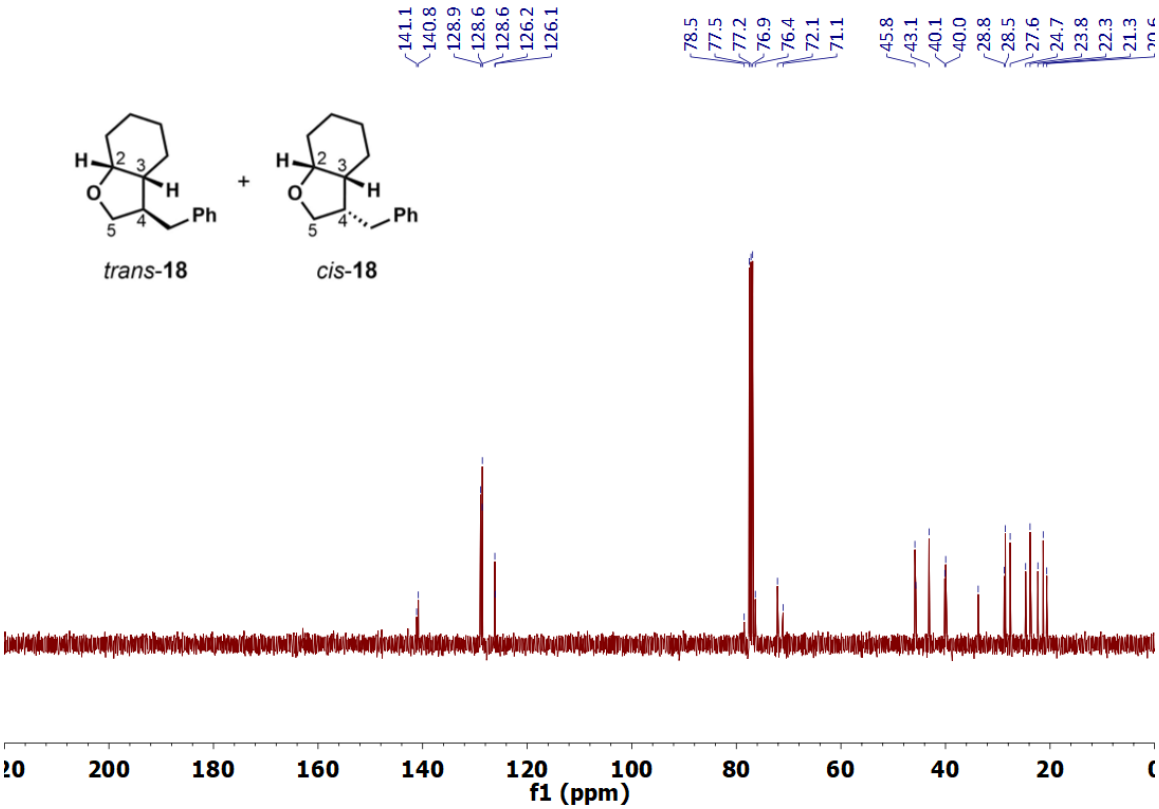

143.9
140.7
128.9
128.8
128.6
128.5
128.5
127.2
126.3
125.7
125.6
80.2
77.5
77.2
76.9
74.0
42.4
42.0
40.8
40.7
39.6
39.3
Ph
O
Ph
+
Ph
O
Ph
145
125
105
85
70
55
40
f1 (ppm)

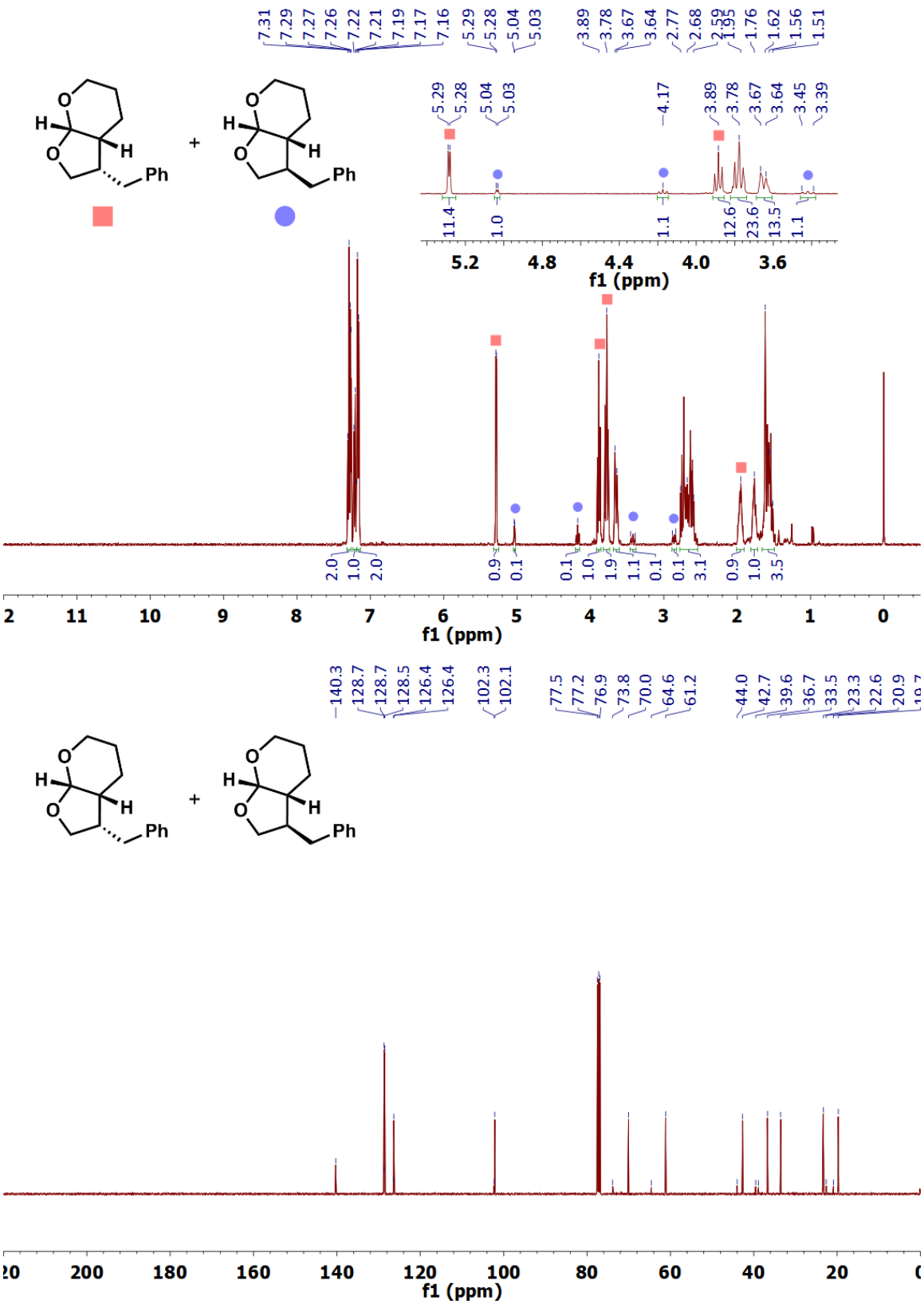

Ph
O
Ph
+
Ph
O
Ph
f1 (ppm)
f2 (ppm)

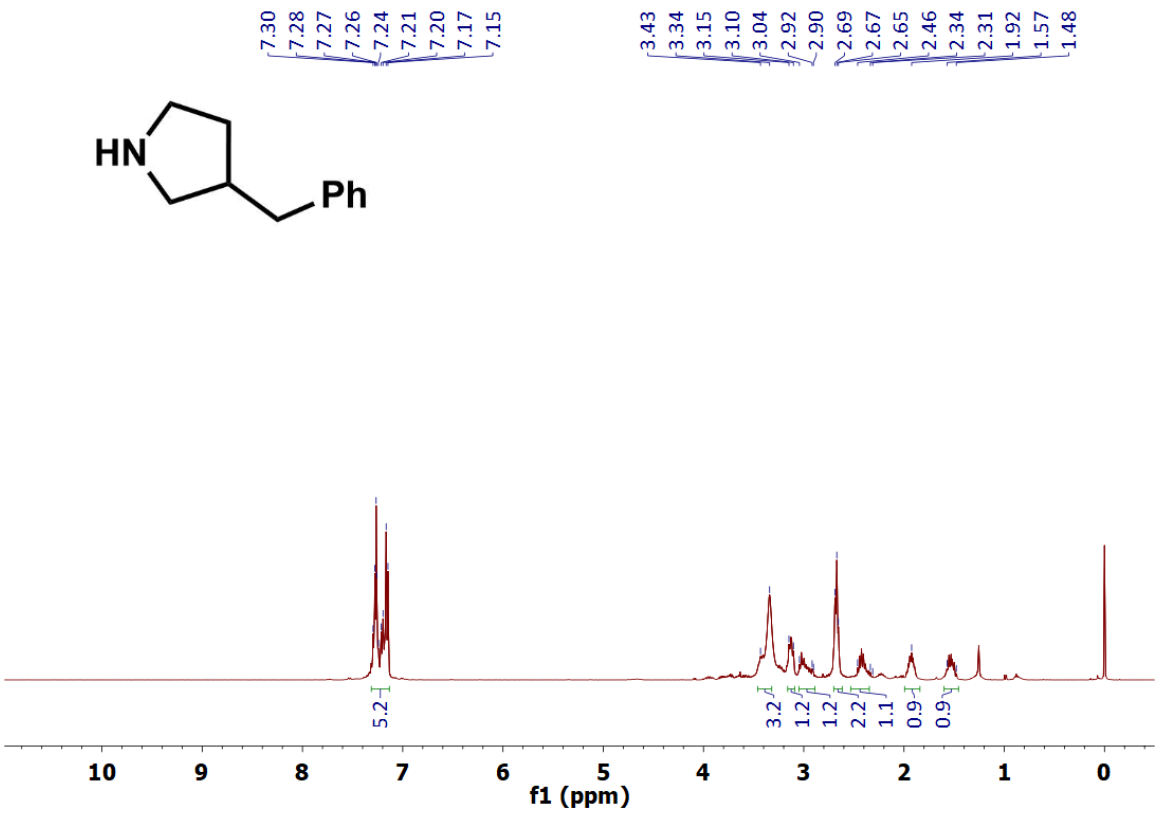

Ph
O
Ph
+
Ph
O
Ph
f1 (ppm)
f2 (ppm)

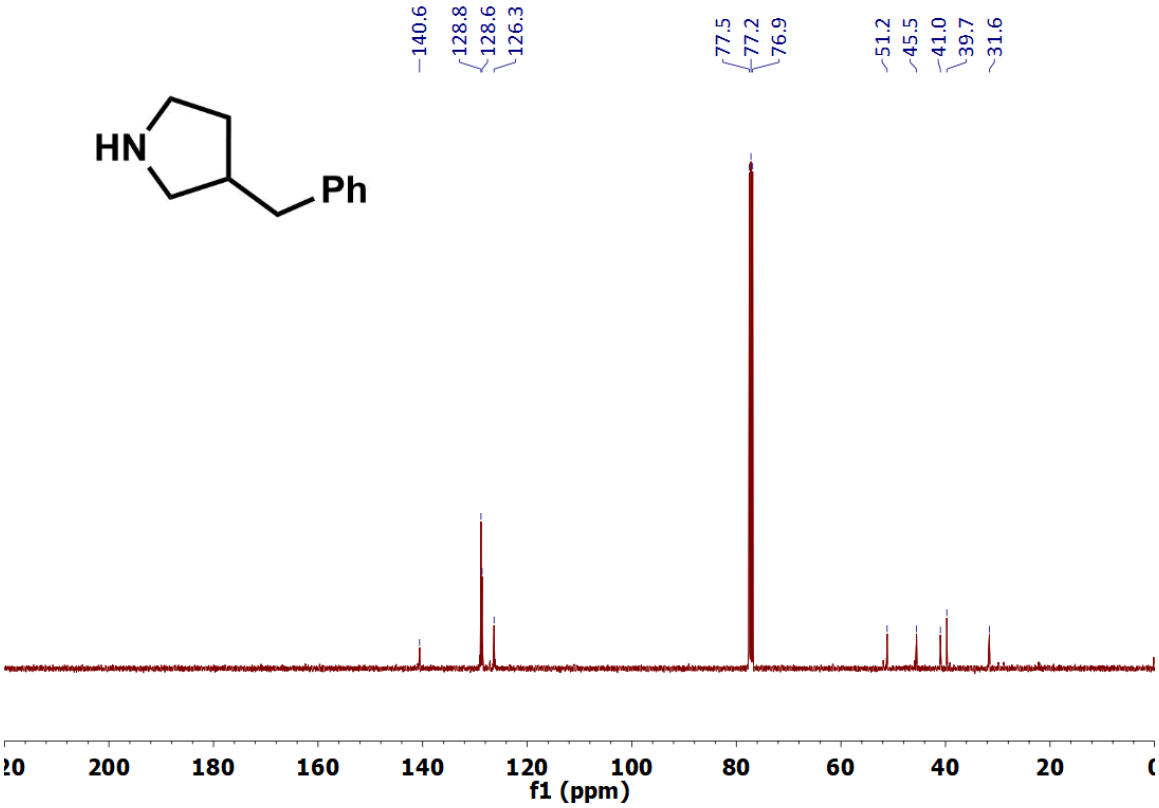

Ph
O
Ph
+
Ph
O
Ph
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
f2 (ppm)
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
f1 (ppm)


trans-18
cis-18
f1 (ppm)


trans-18
cis-18
f1 (ppm)


7.31
7.29
7.27
7.26
7.22
7.21
7.19
7.17
7.16
5.29
5.28
5.04
5.03
3.89
3.78
3.67
3.64
2.77
2.68
2.59
1.95
1.76
1.62
1.56
1.51
4.17
3.45
3.39
11.4
1.0
1.1
12.6
23.6
13.5
5.2
4.8
4.4
4.0
3.6
f1 (ppm)
2.0
1.0
2.0
0.9
0.1
0.1
1.0
1.9
1.1
0.1
0.1
3.1
0.9
1.0
3.5
2
11
10
9
8
7
6
5
4
3
2
1
0
140.3
128.7
128.7
128.5
126.4
126.4
102.3
102.1
77.5
77.2
76.9
73.8
70.0
64.6
61.2
44.0
42.7
39.6
36.7
33.5
23.3
22.6
20.9
200
180
160
140
120
100
80
60
40
20
Ph
O
H


7.30
7.28
7.27
7.26
7.24
7.21
7.20
7.17
7.15
3.43
3.34
3.15
3.10
3.04
2.92
2.90
2.69
2.67
2.65
2.46
2.34
2.31
1.92
1.57
1.48
HN
Ph
5.2
3.2
1.2
1.2
2.2
1.1
0.9
0.9
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)


140.6
128.8
128.6
126.3
77.5
77.2
76.9
51.2
45.5
41.0
39.7
31.6
HN
Ph
200
180
160
140
120
100
80
60
40
20
f1 (ppm)