Electronic Supplementary Information for

Ring expansion of spirocyclopropanes with stabilized sulfonium ylides: highly diastereoselective synthesis of cyclobutanes

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Contents:

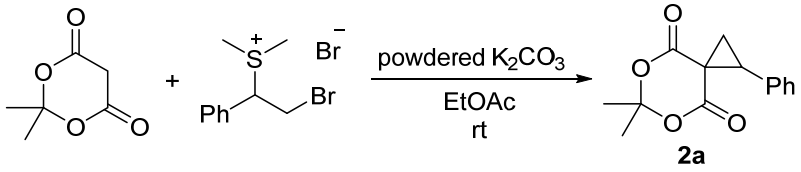
Experimental section

General. Melting points are uncorrected. IR spectra were recorded on a JASCO FT/IR-460 Plus spectrophotometer and absorbance bands are reported in wavenumber (cm^{-1}). All NMR spectra were recorded using Bruker Ascend 500 (500 MHz), JEOL JNM-ECX400P (400 MHz) and Bruker UltrashieldTM 300 (300 MHz) spectrometers. ^{1}H NMR spectra were recorded at 500 or 400 MHz. Chemical shifts are reported relative to internal standard (tetramethylsilane at δ_H 0.00 or $CDCl_3$ at δ_H 7.26). Data are presented as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant and integration. ^{13}C NMR spectra were recorded at 100 or 75 MHz. The following internal reference was used ($CDCl_3$ at δ 77.0). All ^{13}C NMR spectra were determined with complete proton decoupling. High-resolution mass spectra (HRMS) were determined with Thermo Scientific LTQ Orbitrap XL ETD [electrospray ionization (ESI)]. Column chromatography was performed on Silica Gel 60 PF_{254} (Nacalai Tesque) and Kanto silica gel 60 N (63–210 mesh) under pressure. Analytical thin layer chromatography (TLC) was carried out on Merck Kieselgel 60 F_{254} plates. Visualization was accomplished with UV light and phosphomolybdic acid stain solution or basic potassium permanganate stain solution followed by heating.

All reagents, such as Meldrum's acid, 1,3-dimethylbarbituric acid, powdered K_2CO_3, $Rh_2(esp)_2$, 4-acetoxystyrene, 3-methoxystyrene, 2-vinylnaphthalene, 1,4-dibromobut-2-ene, conc. H_2SO_4, C_6H_5Cl, dehydrated EtOAc, CH_2Cl_2, DMF, Et_2O and MeOH are commercially available and were purchased from suppliers such as Sigma-Aldrich Co.; Wako Pure Chemical Industries, Ltd.; Tokyo Chemical Industry Co., Ltd.; Nacalai Tesque, INC. Purchased C_6H_5Cl was distilled and used as an anhydrous solvent. (2-Bromo-1-phenylethyl)dimethylsulfonium bromide,[1] [2-bromo-1-(4-methylphenyl)ethyl]dimethylsulfonium bromide,[1] [2-bromo-1-(4-bromophenyl)ethyl]-dimethylsulfonium bromide,[1] (2-bromoethyl)diphenylsulfonium trifluoromethanesulfonate,[2] dimethylsulfonium benzoylmethylide (**1a**),[3] dimethylsulfonium 4-methoxybenzoylmethylide (**1b**),[3] dimethylsulfonium 3-methoxybenzoylmethylide (**1c**),[4] dimethylsulfonium 2-methoxybenzoylmethylide (**1d**),[5] dimethylsulfonium 4-nitrobenzoyl-methylide (**1e**),[3] dimethylsulfonium 4-chlorobenzoylmethylide (**1f**),[3] and tetrahydrothiophenium acetylmethylide (**1g**),[3] dimethylsulfonium ethoxycarbonylmethylide (**1h**)[3] were prepared according to literature procedures.

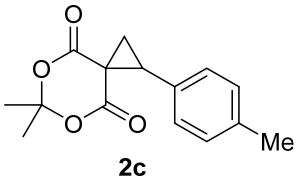
I. Preparation of spirocyclopropanes 2a–h and 6

6,6-Dimethyl-1-phenyl-5,7-dioxaspiro[2.5]octane-4,8-dione (2a).[6]



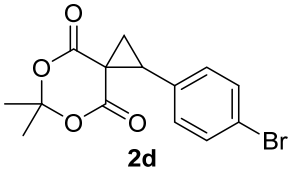
Meldrum's acid (288 mg, 2.0 mmol) and powdered K_2CO_3 (830 mg, 6.0 mmol) were added to a suspension of (2-bromo-1-phenylethyl)dimethylsulfonium bromide (782 mg, 2.4 mmol) in EtOAc (20 mL). After stirring at room temperature for 3 h, the reaction was quenched with water (20 mL), and the resulting mixture was extracted with EtOAc (3 × 20 mL). The combined organic layers were washed with brine (20 mL) and dried over anhydrous $MgSO_4$. The filtrate was concentrated in vacuo, and the residue was purified by column chromatography (silica gel, 25% EtOAc in hexane) to provide **2a** (422 mg, 86%) as a white solid: mp 134.2–135.8 °C [lit.,[6] mp 130–131 °C]; IR (film, cm^{-1}) ν 3002, 1766, 1741, 1328, 1292, 1203, 1176; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37–7.30 (m, 5H), 3.45 (t, *J* = 9.4 Hz, 1H), 2.69 (dd, *J* = 9.4, 4.8 Hz, 1H), 2.55 (dd, *J* = 9.4, 4.8 Hz, 1H), 1.73 (s, 3H), 1.72 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.7, 163.4, 131.0, 129.4, 128.7, 128.4, 104.9, 44.6, 33.1, 27.9, 27.6, 22.9.

6,6-Dimethyl-1-*p*-tolyl-5,7-dioxaspiro[2.5]octane-4,8-dione (2c).[6]



According to the procedure for the synthesis of **2a**, **2c** was prepared from Meldrum's acid (288 mg, 2.0 mmol) with [2-bromo-1-(4-methylphenyl)ethyl]dimethylsulfonium bromide (819 mg, 2.4 mmol) for 3 h. The crude product was purified by column chromatography (silica gel, 20% EtOAc in hexane) to provide **2c** (362 mg, 65%) as a white solid: mp 151.0–153.0 °C [lit.,[6] mp 137–138 °C]; IR (KBr, cm^{-1}) ν 3001, 1760, 1736, 1357, 1200, 1186; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (d, *J* = 8.0 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 3.42 (t, *J* = 9.5 Hz, 1H), 2.68 (dd, *J* = 9.2, 4.6 Hz, 1H), 2.54 (dd, *J* = 9.5, 4.6 Hz, 1H), 2.33 (s, 3H), 1.73 (s, 3H), 1.72 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.8, 163.5, 138,7, 129.3, 129.1, 128.0, 104.9, 44.8, 33.2, 27.9, 27.7, 22.9, 21.2.

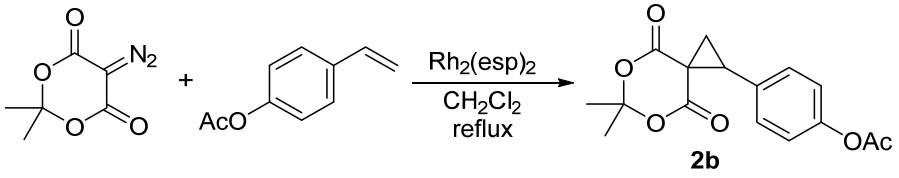
1-(4-Bromophenyl)-6,6-dimethyl-5,7-dioxaspiro[2.5]octane-4,8-dione (2d).[7]



2d

According to the procedure for the synthesis of **2a**, **2d** was prepared from Meldrum's acid (288 mg, 2.0 mmol) with [2-bromo-1-(4-bromophenyl)ethyl]dimethylsulfonium bromide (974 mg, 2.4 mmol) for 1 h. The crude product was purified by column chromatography (silica gel, 20% EtOAc in hexane) to provide **2d** (527 mg, 87%) as a white solid: mp 156.0–156.5 °C; IR (KBr, cm^{-1}) ν 3003, 1766, 1740, 1329, 1295, 1203; ^1H NMR (400 MHz, $CDCl_3$) δ 7.48 (d, J = 8.2 Hz, 1H), 7.21 (d, J = 8.2 Hz, 1H), 3.39 (t, J = 9.4 Hz, 1H), 2.63 (dd, J = 9.2, 5.0 Hz, 1H), 2.55 (dd, J = 9.4, 4.8 Hz, 1H), 1.74 (s, 3H), 1.71 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.5, 163.5, 131.6, 131.0, 130.2, 123.0, 105.0, 43.5, 32.9, 28.0, 27.6, 23.1.

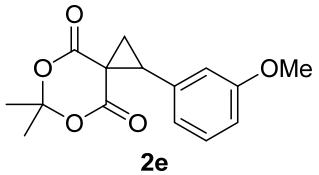
1-(4-Acetoxyphenyl)-6,6-dimethyl-5,7-dioxaspiro[2.5]octane-4,8-dione (2b).[6]



2b

$Rh_2(esp)_2$ (7.6 mg, 0.01 mmol, 1 mol%) was added to a solution of 5-diazo-2,2-dimethyl-1,3-dioxane-4,6-dione (170 mg, 1.0 mmol), 4-acetoxystyrene (324 mg, 2.0 mmol) in CH_2Cl_2 (2.0 mL) at room temperature. After stirring at reflux for 21 h, the reaction was cooled to room temperature. Evaporation in vacuo furnished the crude product, which was purified by column chromatography (silica gel, 10% EtOAc in hexane) to provide **2b** (226 mg, 70%) as a white solid: mp 181.2–182.0 °C [lit.,[6] mp 174–175 °C]; IR (film, cm^{-1}) ν 2997, 1761, 1739, 1335, 1296, 1197, 1169; ^1H NMR (400 MHz, $CDCl_3$) δ 7.35 (d, J = 8.7 Hz, 2H), 7.09 (d, J = 8.7 Hz, 2H), 3.42 (t, J = 9.4 Hz, 1H), 2.65 (dd, J = 9.4, 4.8 Hz, 1H), 2.55 (dd, J = 9.6, 4.6 Hz, 1H), 2.29 (s, 3H), 1.74 (s, 3H), 1.72 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.1, 167.6, 163.5, 150.9, 130.5, 128.6, 121.6, 105.0, 43.8, 33.1, 28.0, 27.6, 23.1, 21.1.

1-(3-Methoxyphenyl)-6,6-dimethyl-5,7-dioxaspiro[2.5]octane-4,8-dione (2e).[6]



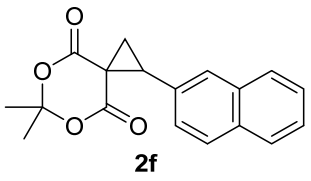
2e

According to the procedure for the synthesis of **2b**, **2e** was prepared from 5-diazo-2,2-dimethyl-1,3-dioxane-4,6-dione (170 mg, 1.0 mmol) with 3-methoxystyrene (268 mg, 2.0 mmol) for 9.5 h. The crude product was purified by column chromatography (silica gel, 10% EtOAc in hexane) to provide **2e** (237 mg, 81%) as a white solid: mp 92.0–93.0 °C [lit.,[6] mp

84–85 °C]; IR (KBr, cm^{-1}) ν 3003, 1766, 1741, 1329, 1296, 1205; ^1H NMR (400 MHz, $CDCl_3$) δ 7.25 (m, 1H), 6.92 (d, *J* = 7.8 Hz, 1H), 6.87–6.85 (m, 2H), 3.80 (s, 3H), 3.41 (t, *J* = 9.4 Hz, 1H), 2.66 (dd, *J* = 9.4, 4.8 Hz, 1H), 2.53 (dd, *J* = 9.4, 4.8 Hz, 1H), 1.74 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.8, 163.5, 159.5, 132.7, 129.4, 121.9, 115.1, 114.1, 104.9, 55.2, 44.5, 33.0, 27.9, 27.7, 23.0.

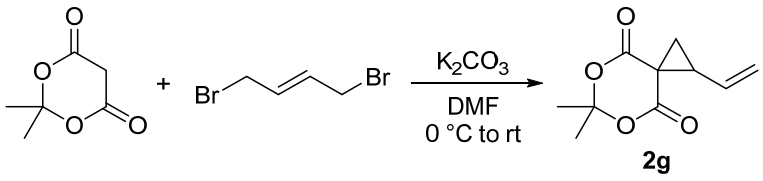
6,6-Dimethyl-1-naphthalen-2-yl-5,7-dioxaspiro[2.5]octane-4,8-dione (2f).[6]

According to the procedure for the synthesis of **2b**, **2f** was prepared from 5-diazo-2,2-dimethyl-1,3-dioxane-4,6-dione (170 mg, 1.0 mmol) with 2-vinylnaphthalene (308 mg, 2.0 mmol) for 24 h. The crude product was purified by column chromatography (silica gel, 10% EtOAc in hexane) to provide **2f** (234 mg, 75%) as a white solid: mp 142.3–143.8 °C [lit.,[6] mp 141–142 °C]; IR (KBr, cm^{-1}) ν 2998, 1766, 1740, 1332, 1296, 1200; ^1H NMR (400 MHz, $CDCl_3$) δ 7.85–7.80 (m, 4H), 7.51–7.48 (m, 2H), 7.42 (dd, *J* = 8.3, 2.0 Hz, 1H), 3.61 (t, *J* = 9.5 Hz, 1H), 2.83 (dd, *J* = 9.2, 4.6 Hz, 1H), 2.63 (dd, *J* = 9.5, 4.9 Hz, 1H), 1.74 (s, 3H), 1.73 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.7, 163.5, 133.2, 132.9, 129.1, 128.5, 128.1, 128.0, 127.6, 126.7, 126.6, 126.5, 104.9, 44.9, 33.3, 27.9, 27.7, 23.2.



2f

6,6-Dimethyl-1-vinyl-5,7-dioxaspiro[2.5]octane-4,8-dione (2g).[8]

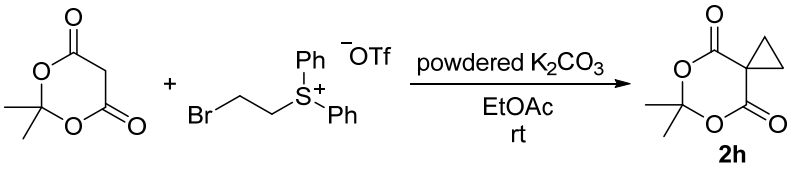


2g

K_2CO_3 (173 mg, 1.25 mmol) was added to a suspension of Meldrum's acid (144 mg, 1.0 mmol) in DMF (20 mL) at 0 °C. After stirring at 0 °C for 10 min, 1,4-dibromobut-2-ene (257 mg, 1.2 mmol) was added in a single portion and the reaction was additionally stirred at 0 °C for 1 h and at room temperature for 2 h. A further portion of K_2CO_3 (173 mg, 1.25 mmol) was added, and then the reaction was stirred at room temperature for 16 h. The reaction was quenched with sat. aq. NH_4Cl (10 mL), and the resulting mixture was extracted with Et_2O (3 × 20 mL). The combined organic layers were washed with brine (20 mL) and dried over anhydrous $MgSO_4$. The filtrate was concentrated in vacuo, and the residue was purified by column chromatography (silica gel, 1:1 Et_2O/hexane) to provide **2g** (112 mg, 57%) as a white solid: mp 48.0–48.5 °C; IR (film, cm^{-1}) ν 3002, 1768, 1742, 1353, 1326, 1284, 1198; ^1H NMR (400 MHz, $CDCl_3$) δ 5.76 (dt, *J* =

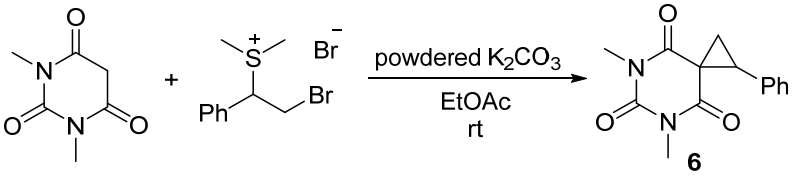
16.9, 10.1 Hz, 1H), 5.47 (dd, *J* = 16.9, 0.9 Hz, 1H), 5.35 (dd, *J* = 10.1, 0.9 Hz, 1H), 2.78 (q, *J* = 9.2 Hz, 1H), 2.37 (dd, *J* = 9.2, 4.6 Hz, 1H), 2.23 (dd, *J* = 8.7, 4.6 Hz, 1H), 1.78 (s, 3H), 1.73 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.4, 165.1, 131.2, 121.8, 105.0, 42.9, 31.4, 27.6, 27.5, 24.5.

6,6-Dimethyl-5,7-dioxaspiro[2.5]octane-4,8-dione (2h).[2]



Meldrum's acid (288 mg, 2.0 mmol) and powdered K_2CO_3 (829 mg, 6.0 mmol) were added to a suspension of (2-bromoethyl)diphenylsulfonium trifluoromethanesulfonate (1.33 g, 3.0 mmol) in EtOAc (20 mL). After stirring at room temperature for 1 h, the reaction was quenched with water (10 mL), and the resulting mixture was extracted with EtOAc (3 × 10 mL). The combined organic layers were washed with brine (10 mL) and dried over anhydrous $MgSO_4$. The filtrate was concentrated in vacuo, and the residue was purified by column chromatography (silica gel, 20% EtOAc in hexane) to provide **2h** (297 mg, 87%) as a white solid: mp 65.5–66.3 °C [lit.,[2] mp 60.5–61.5 °C]; IR (film, cm^{-1}) ν 3021, 1775, 1752, 1399, 1337, 1205; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.99 (s, 4H), 1.82 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.1, 105.1, 27.6, 24.1 23.9.

5,7-dimethyl-1-phenyl-5,7-diazaspiro[2.5]octane-4,6,8-trione (6).[9]



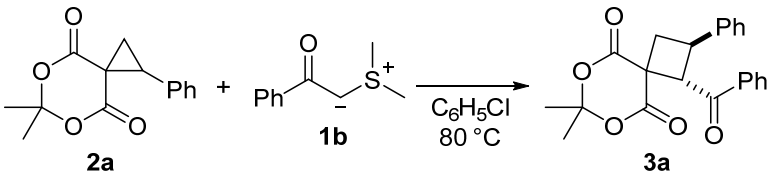
1,3-Dimethylbarbituric acid (156 mg, 1.0 mmol) and powdered K_2CO_3 (415 mg, 3.0 mmol) were added to a suspension of (2-bromo-1-phenylethyl)dimethylsulfonium bromide (391 mg, 1.2 mmol) in EtOAc (20 mL). After stirring at room temperature for 9 h, the reaction was quenched with water (20 mL), and the resulting mixture was extracted with EtOAc (3 × 20 mL). The combined organic layers were washed with brine (20 mL) and dried over anhydrous $MgSO_4$. The filtrate was concentrated in vacuo, and the residue was purified by column chromatography (silica gel, 40% EtOAc in hexane) to provide **6** (175 mg, 68%) as a white solid: mp 93.0–93.5 °C [lit.,[9a] mp 78–80 °C, lit.,[9b] mp 87–88 °C]; IR (film, cm^{-1}) ν 3019, 1737, 1690, 1673, 1420, 1387, 1214; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29–7.26 (m, 5H), 3.52 (t, *J* = 9.2 Hz, 1H), 3.37 (s, 3H), 3.11 (s,

3H), 2.59 (dd, J = 9.2, 3.2 Hz, 1H), 2.45 (dd, J = 9.2, 3.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.2, 164.8, 151.8, 132.4, 129.6, 128.1, 127.9, 46.2, 35.8, 28.7, 28.4, 24.5.

II. Ring expansion of spirocyclopropanes with sulfonium ylides

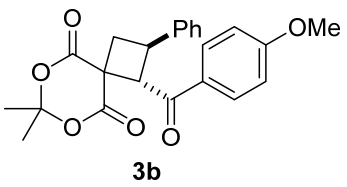
Typical procedure for the ring expansion of spirocyclopropane 2a with sulfonium ylide 1a (Table 1, entry 6):

***rac*-(1*R*,2*S*)-1-Benzoyl-7,7-dimethyl-2-phenyl-6,8-dioxaspiro[3.5]nonane-5,9-dione (3a).**



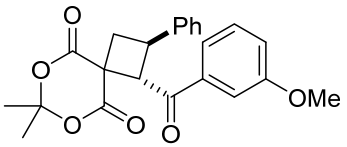
Dimethylsulfonium benzoylmethylide (**1a**) (81 mg, 0.45 mmol) was added to a solution of 6,6-dimethyl-1-phenyl-5,7-dioxaspiro[2.5]octane-4,8-dione (**2a**) (74 mg, 0.30 mmol) in C_6H_5Cl (1.5 mL) at room temperature. After stirring at 80 °C for 6 h, the reaction was cooled to room temperature. Evaporation in vacuo furnished the crude product, which was purified by column chromatography (silica gel, 10% EtOAc in hexane) to provide **3a** (94 mg, 86%) as a white solid: mp 134.0–135.0 °C; IR (film, cm^{-1}) ν 3003, 1771, 1739, 1685, 1301, 1203; ^1H NMR (400 MHz, $CDCl_3$) δ 7.48–7.23 (m, 10H), 4.86 (d, *J* = 10.1 Hz, 1H), 4.32 (dt, *J* = 10.1, 9.6 Hz, 1H), 2.92 (d, *J* = 9.6 Hz, 2H), 1.76 (s, 3H), 1.74 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.8, 170.0 168.4, 141.7, 134.6, 133.4, 129.0, 128.5, 128.3, 127.8, 127.7, 105.8, 57.4, 46.3, 39.1, 38.1, 29.1, 28.0; HRMS (ESI) *m/z* calcd for $C_{22}H_{21}O_5$ $(M+H)^+$ 365.1384, found 365.1383.

***rac*-(1*R*,2*S*)-1-(4-Methoxybenzoyl)-7,7-dimethyl-2-phenyl-6,8-dioxaspiro[3.5]nonane-5,9-dione (3b) (Table 2, entry 2).**



According to the typical procedure for the ring expansion of **2a** with **1a**, **3b** was prepared from **2a** (74 mg, 0.30 mmol) with **1b** (95 mg, 0.45 mmol) for 6 h. The crude product was purified by column chromatography (silica gel, 10% EtOAc in hexane) to provide **3b** (87 mg, 74%) as a white solid: mp 116.8–117.6 °C; IR (KBr, cm^{-1}) ν 3007, 1769, 1739, 1601, 1302, 1262, 1204, 1171; ^1H NMR (400 MHz, $CDCl_3$) δ 7.50–7.26 (m, 9H), 4.82 (d, *J* = 10.1 Hz, 1H), 4.31 (dt, *J* = 10.1, 9.6 Hz, 1H), 3.79 (s, 3H), 2.91 (d, *J* = 9.6 Hz, 2H), 1.79 (s, 3H), 1.74 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.9, 170.2, 168.6, 163.8, 141.8, 130.9, 129.0, 127.8, 127.3, 113.7, 105.7, 57.9, 55.4, 46.1, 39.6, 38.0, 29.1, 28.1; HRMS (ESI) *m/z* calcd for $C_{23}H_{23}O_6$ $(M+H)^+$ 395.1489, found 395.1490.

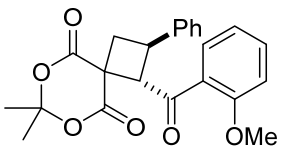
***rac*-(1*R*,2*S*)-1-(3-Methoxybenzoyl)-7,7-dimethyl-2-phenyl-6,8-dioxaspiro[3.5]nonane-5,9-dione (3c) (Table 2, entry 3).**



3c

According to the typical procedure for the ring expansion of **2a** with **1a**, **3c** was prepared from **2a** (74 mg, 0.30 mmol) with **1c** (95 mg, 0.45 mmol) for 6 h. The crude product was purified by column chromatography (silica gel, 10% to 30% EtOAc in hexane) to provide **3c** (102 mg, 86%) as a colorless amorphous: IR (ATR, cm^{-1}) ν 1737, 1667, 1597, 1283, 1246, 1199; ^1H NMR (500 MHz, $CDCl_3$) δ 7.57–6.65 (m, 9H), 4.83 (d, 1H, J = 10.0 Hz), 4.15 (dt, 1H, J = 10.0, 9.5 Hz), 3.27 (s, 3H), 2.88 (dd, 1H, J = 11.0, 10.0 Hz), 2.80 (dd, 1H, J = 10.5, 10.0 Hz), 1.75 (s, 3H), 1.70 (s, 3H); ^{13}C NMR (75 Hz, $CDCl_3$) δ 198.8, 170.0, 168.8, 157.0, 142.5, 133.5, 130.6, 128.4, 127.3, 127.0, 126.1, 120.8, 110.2, 105.3, 60.2, 54.1, 46.4, 38.8, 36.9, 29.2, 27.7; HRMS (FAB) *m/z* calcd for $C_{23}H_{23}O_6$ $(M+H)^+$ 395.1489, found 395.1488.

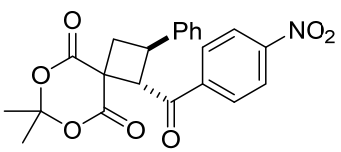
***rac*-(1*R*,2*S*)-1-(2-Methoxybenzoyl)-7,7-dimethyl-2-phenyl-6,8-dioxaspiro[3.5]nonane-5,9-dione (3d) (Table 2, entry 4).**



3d

According to the typical procedure for the ring expansion of **2a** with **1a**, **3d** was prepared from **2a** (74 mg, 0.30 mmol) with **1d** (95 mg, 0.45 mmol) for 6 h. The crude product was purified by column chromatography (silica gel, 9% to 15% EtOAc in hexane) to provide **3d** (103 mg, 87%) as a colorless amorphous: IR (ATR, cm^{-1}) ν 3001, 1735, 1681, 1283, 1256, 1199; ^1H NMR (500 MHz, $CDCl_3$) δ 7.50–7.00 (m, 9H), 4.87 (d, 2H, J = 10.5 Hz), 3.56 (s, 3H), 2.91 (m, 2H), 1.77 (s, 3H), 1.74 (s, 3H); ^{13}C NMR (75 Hz, $CDCl_3$) δ 196.5, 169.9, 168.2, 159.6, 141.7, 135.7, 129.4, 128.9, 127.6, 127.6, 121.0, 120.7, 111.5, 105.6, 57.1, 54.9, 46.3, 39.0, 38.1, 29.0, 27.9; HRMS (FAB) *m/z* calcd for $C_{23}H_{23}O_6$ $(M+H)^+$ 395.1489, found 395.1483.

***rac*-(1*R*,2*S*)-7,7-Dimethyl-1-(4-nitrobenzoyl)-2-phenyl-6,8-dioxaspiro[3.5]nonane-5,9-dione (3e) (Table 2, entry 5).**

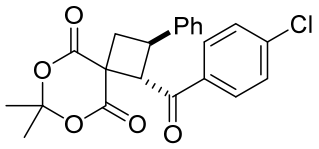


3e

According to the typical procedure for the ring expansion of **2a** with **1a**, **3e** was prepared from **2a** (74 mg, 0.30 mmol) with **1e** (102 mg, 0.45 mmol) for 24 h. The crude product was purified by column chromatography (silica gel, 10% EtOAc in hexane) to provide **3e** (75 mg, 61%) as a white solid: mp 108.0–110.0 °C; IR (KBr, cm^{-1}) ν 3003, 1771, 1738, 1696, 1526, 1302, 1202; ^1H NMR (400 MHz, $CDCl_3$) δ 7.26–8.11 (m, 9H), 4.88 (d, J = 10.1 Hz,

1H), 4.29 (dt, J = 10.1, 9.2 Hz, 1H), 2.94 (d, J = 9.2 Hz, 2H), 1.80 (s, 3H), 1.76 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.9, 169.5, 168.1, 150.4, 141.0, 139.4, 129.4, 128.2, 127.6, 123.7, 106.0, 56.8, 46.5, 39.0, 38.5, 29.0, 28.1; HRMS (ESI) *m/z* calcd for $C_{22}H_{19}NNaO_7$ $(M+Na)^+$ 432.1054, found 432.1059.

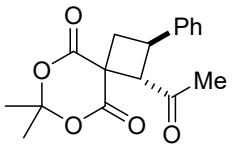
***rac*-(1*R*,2*S*)-1-(4-Chlorobenzoyl)-7,7-dimethyl-2-phenyl-6,8-dioxaspiro[3.5]nonane-5,9-dione (3f) (Table 2, entry 6).**



3f

According to the typical procedure for the ring expansion of **2a** with **1a**, **3f** was prepared from **2a** (74 mg, 0.30 mmol) with **1f** (97 mg, 0.45 mmol) for 5 h. The crude product was purified by column chromatography (silica gel, 10% EtOAc in hexane) to provide **3f** (100 mg, 83%) as a white solid: mp 166.5–168.0 °C; IR (KBr, cm^{-1}) ν 3007, 1771, 1739, 1684, 1302, 1202; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58–7.21 (m, 9H), 4.82 (d, J = 10.1 Hz, 1H), 4.30 (dt, J = 9.6 Hz, 10.1 Hz, 1H), 2.91 (d, J = 9.6 Hz, 2H), 1.80 (s, 3H), 1.75 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.7, 169.9, 168.4, 141.4, 140.0, 132.8, 129.8, 129.0, 128.0, 127.7, 105.9, 57.3, 46.2, 39.3, 38.2, 29.1, 28.1; HRMS (ESI) *m/z* calcd for $C_{22}H_{19}ClNaO_5$ $(M+Na)^+$ 421.0813, found 421.0817.

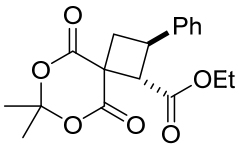
***rac*-(1*R*,2*S*)-1-Acetyl-7,7-dimethyl-2-phenyl-6,8-dioxaspiro[3.5]nonane-5,9-dione (3g) (Table 2, entry 7).**



3g

According to the typical procedure for the ring expansion of **2a** with **1a**, **3g** was prepared from **2a** (74 mg, 0.30 mmol) with **1g** (65 mg, 0.45 mmol) for 24 h. The crude product was purified by column chromatography (silica gel, 10% EtOAc in hexane) to provide **3g** (33 mg, 36%) as a white solid: mp 96.0–97.0 °C; IR (KBr, cm^{-1}) ν 3003, 1771, 1739, 1711, 1301, 1202; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51–7.26 (m, 5H), 4.19 (m, 1H), 2.84 (m, 1H), 2.76 (m, 2H), 1.99 (s, 3H), 1.87 (s, 3H), 1.75 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.6, 169.5, 168.8, 141.1, 129.1, 127.0, 105.9, 59.2, 45.7, 39.3, 38.5, 28.9, 28.1, 26.8; HRMS (ESI) *m/z* calcd for $C_{17}H_{18}NaO_5$ $(M+Na)^+$ 325.1046, found 325.1047.

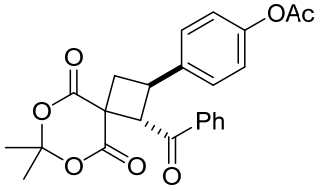
***rac*-(1*R*,2*S*)-Ethyl 7,7-dimethyl-5,9-dioxo-2-phenyl-6,8-dioxaspiro[3.5]nonane-1-carboxylate (3h) (Table 2, entry 8).**



3h

According to the typical procedure for the ring expansion of **2a** with **1a**, **3h** was prepared from **2a** (74 mg, 0.30 mmol) with **1h** (67 mg, 0.45 mmol) for 23 h. The crude product was purified by column chromatography (silica gel, 10% EtOAc in hexane) to provide **3h** (53 mg, 53%) as a white solid: mp 132.5–133.7 °C; IR (KBr, cm^{-1}) ν 2997, 1773, 1743, 1720, 1698, 1303, 1202; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43–7.26 (m, 5H), 4.32 (dt, *J* = 10.1, 9.6 Hz, 1H), 4.15 (m, 3H), 2.87 (dd, *J* = 10.5, 9.6 Hz, 1H), 2.78 (dd. *J* = 10.5, 10.1 Hz, 1H), 1.83 (s, 3H), 1.74 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.0, 169.1, 168.4, 141.4, 128.7, 127.3, 126.9, 105.6, 61.3, 50.3, 46.5, 38.0, 37.6, 29.0, 28.2, 14.0; HRMS (ESI) *m/z* calcd for $C_{18}H_{20}NaO_6$ $(M+Na)^+$ 355.1152, found 355.1155.

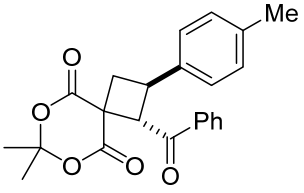
***rac*-(1*R*,2*S*)-4-(1-Benzoyl-7,7-dimethyl-5,9-dioxo-6,8-dioxaspiro[3.5]nonan-2-yl)phenyl acetate (3i) (Table 3, entry 1).**



3i

According to the typical procedure for the ring expansion of **2a** with **1a**, **3i** was prepared from **2b** (97 mg, 0.30 mmol) with **1a** (81 mg, 0.45 mmol) for 3 h. The crude product was purified by column chromatography (silica gel, 10% EtOAc in hexane) to provide **3i** (81 mg, 64%) as a white solid: mp 148.5–149.7 °C; IR (KBr, cm^{-1}) ν 3003, 1766, 1739, 1684, 1304, 1200; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49–7.11 (m, 9H), 4.82 (d, *J* = 10.1 Hz, 1H), 4.33 (dt, *J* = 10.1, 9.2 Hz 1H), 2.91 (d, *J* = 9.2 Hz, 2H), 2.32 (s, 3H), 1.73 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.7, 170.0, 169.5, 168.2, 150.1, 139.3, 134.6, 133.5, 128.8, 128.6, 128.3, 122.1, 105.8, 57.4, 46.3, 38.4, 38.0, 29.2, 28.0, 21.1; HRMS (ESI) *m/z* calcd for $C_{24}H_{23}O_7$ $(M+H)^+$ 423.1438, found 423.1437.

***rac*-(1*R*,2*S*)-1-Benzoyl-7,7-dimethyl-2-(4-methylphenyl)-6,8-dioxaspiro[3.5]nonane-5,9-dione (3j) (Table 3, entry 2).**

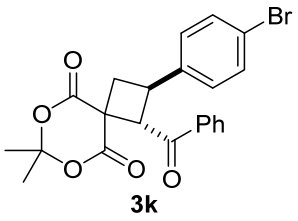


3j

According to the typical procedure for the ring expansion of **2a** with **1a**, **3j** was prepared from **2c** (83 mg, 0.30 mmol) with **1a** (81 mg, 0.45 mmol) for 6 h. The crude product was purified by column chromatography (silica gel, 10% EtOAc in hexane) to provide **3j** (84 mg, 74%) as a white solid: mp 181.5–183.0 °C; IR (KBr, cm^{-1}) ν 3003, 1771, 1739, 1685, 1301, 1203; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48–7.20 (m, 9H), 4.83 (d, *J* = 9.6 Hz, 1H), 4.27 (t, *J* = 9.6

Hz, 1H), 2.88 (d, J = 9.6 Hz, 2H), 1.76 (s, 3H), 1.73 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.0, 170.1, 168.5, 138.7, 137.5, 134.6, 133.4, 129.6, 128.5, 128.4, 127.6, 105.7, 57.7, 46.3, 38.9, 38.2, 29.1, 28.0, 21.1; HRMS (ESI) *m/z* calcd for $C_{23}H_{23}O_5$ $(M+H)^+$ 379.1540, found 379.1540.

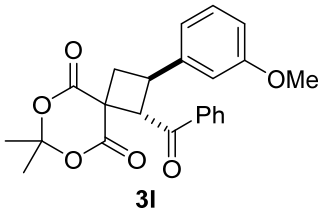
***rac*-(1*R*,2*S*)-1-Benzoyl-2-(4-bromophenyl)-7,7-dimethyl-6,8-dioxaspiro[3.5]nonane-5,9-dione (3k) (Table 3, entry 3).**



3k

According to the typical procedure for the ring expansion of **2a** with **1a**, **3k** was prepared from **2d** (90 mg, 0.30 mmol) with **1a** (81 mg, 0.45 mmol) for 6 h. The crude product was purified by column chromatography (silica gel, 10% EtOAc in hexane) to provide **3k** (91 mg, 80%) as a white solid: mp 163.0–164.0 °C; IR (KBr, cm^{-1}) ν 2942, 1772, 1738, 1685, 1307, 1298, 1203; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50–6.87 (m, 9H), 4.79 (d, J = 10.1 Hz, 1H), 4.30 (q, J = 9.6 Hz, 1H), 2.89 (m, 2H), 1.73 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.6, 169.9, 168.1, 140.7, 134.6, 133.6, 132.0, 129.4, 128.7, 128.2, 121.6, 105.9, 56.9, 46.4, 38.2, 37.8, 29.1, 28.0; HRMS (ESI) *m/z* calcd for $C_{22}H_{20}Br$ O_5 $(M+H)^+$ 443.0489, found 443.0489.

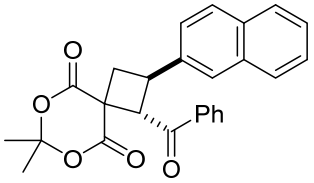
***rac*-(1*R*,2*S*)-1-Benzoyl-2-(3-methoxyphenyl)-7,7-dimethyl-6,8-dioxaspiro[3.5]nonane-5,9-dione (3l) (Table 3, entry 4).**



3l

According to the typical procedure for the ring expansion of **2a** with **1a**, **3l** was prepared from **2e** (88 mg, 0.30 mmol) with **1a** (81 mg, 0.45 mmol) for 12 h. The crude product was purified by column chromatography (silica gel, 10% EtOAc in hexane) to provide **3l** (81 mg, 69%) as a white solid: mp 116.1–118.0 °C; IR (KBr, cm^{-1}) ν 2924, 1771, 1738, 1684, 1303, 1203; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49–7.11 (m, 9H), 4.82 (d, J = 10.1 Hz, 1H), 4.33 (dt, J = 9.2 Hz, 10.1 Hz 1H), 2.91 (d, J = 9.2 Hz, 2H), 2.32 (s, 3H), 1.73 (s, 3H), 1.73 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.9, 169.9, 168.4, 160.0, 143.3, 134.6, 133.4, 130.0, 128.5, 128.4, 120.0, 113.3, 105.8, 57.2, 46.4, 39.2, 37.9, 29.1, 28.0; HRMS (ESI) *m/z* calcd for $C_{23}H_{23}O_6$ $(M+H)^+$ 395.1489, found 395.1489.

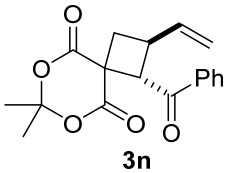
***rac*-(1*R*,2*S*)-1-Benzoyl-7,7-dimethyl-2-(naphthalen-2-yl)-6,8-dioxaspiro[3.5]nonane-5,9-dione (3m) (Table 3, entry 5).**



3m

According to the typical procedure for the ring expansion of **2a** with **1a**, **3m** was prepared from **2f** (94 mg, 0.30 mmol) with **1a** (81 mg, 0.45 mmol) for 48 h. The crude product was purified by column chromatography (silica gel, 10% EtOAc in hexane) to provide **3m** (99 mg, 80%) as a white solid: mp 212.5–214.0 °C; IR (KBr, cm^{-1}) ν 3007, 1772, 1736, 1684, 1299, 1201; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92–7.18 (m, 12H), 4.97 (d, *J* = 10.1 Hz, 1H), 4.50 (dt, *J* = 9.6 Hz, 10.1 Hz 1H), 3.03 (t, *J* = 10.1 Hz, 1H), 2.97 (t, *J* = 9.6 Hz, 1H) 1.77 (s, 3H), 1.75 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.9, 170.1, 168.4, 138.9, 134.6, 133.4, 133.3, 132.8, 129.0, 128.5, 128.4, 127.9, 127.7, 126.7, 126.4, 126.1, 125.3, 105.8, 57.2, 46.5, 39.3, 38.0, 29.2, 28.0; HRMS (ESI) *m/z* calcd for $C_{26}H_{23}O_5$ $(M+H)^+$ 415.1540, found 415.1539.

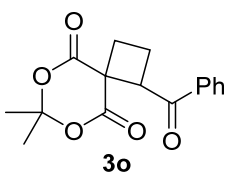
***rac*-(1*R*,2*R*)-1-Benzoyl-7,7-dimethyl-2-vinyl-6,8-dioxaspiro[3.5]nonane-5,9-dione (3n) (Table 3, entry 6).**



3n

According to the typical procedure for the ring expansion of **2a** with **1a**, **3n** was prepared from **2g** (59 mg, 0.30 mmol) with **1a** (81 mg, 0.45 mmol) for 24 h. The crude product was purified by column chromatography (silica gel, 10% EtOAc in hexane) to provide **3n** (61 mg, 68%) as a white solid: mp 105.0–107.0 °C; IR (KBr, cm^{-1}) ν 3001, 1772, 1740, 1684, 1295, 1204; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76–7.27 (m, 5H), 6.12 (m, 1H), 5.19–5.26 (m, 2H), 4.54 (d, *J* = 9.6 Hz, 1H), 3.82 (m, 1H), 2.69 (dd, *J* = 11.0, 10.1 Hz, 1H), 2.59 (dd, *J* = 10.5, 9.6 Hz, 1H), 1.72 (s, 3H), 1.70 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.9, 169.7 168.2, 139.2, 134.8, 133.5, 128.6, 128.5, 117.9, 105.6, 55.4, 46.8, 37.7, 36.0, 29.1, 28.0; HRMS (ESI) *m/z* calcd for $C_{18}H_{19}O_5$ $(M+H)^+$ 315.1227, found 315.1226.

1-Benzoyl-7,7-dimethyl-6,8-dioxaspiro[3.5]nonane-5,9-dione (3o) (Table 3, entry 7).

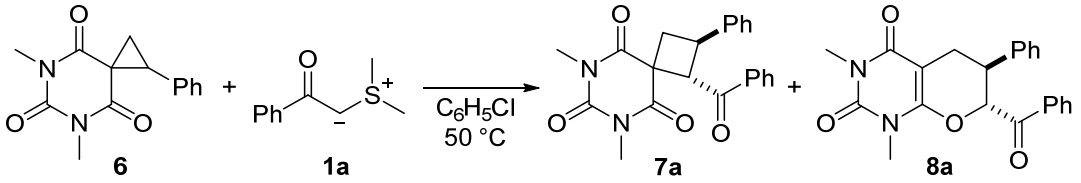


3o

According to the typical procedure for the ring expansion of **2a** with **1a**, **3o** was prepared from **2h** (51 mg, 0.30 mmol) with **1a** (81 mg, 0.45 mmol) for 24 h. The crude product was purified by column chromatography (silica gel, 10% EtOAc in hexane) to provide **3o** (22 mg, 26%) as a pale yellow oil; IR (KBr, cm^{-1}) ν 3003, 1771, 1739, 1683, 1305, 1284, 1203; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77–7.26 (m, 5H), 4.75 (dd, *J* = 10.5, 9.6 Hz, 1H), 3.02 (m, 1H), 2.78 (m, 1H), 2.66 (m, 1H), 2.48 (m,

1H), 1.78 (s, 3H), 1.71 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.0, 169.7, 168.4, 134.4, 133.6, 128.8, 128.2, 105.5, 58.1, 49.3, 30.3, 29.1, 28.0, 27.8, 23.0; HRMS (ESI) *m/z* calcd for $C_{16}H_{16}NaO_5$ $(M+Na)^+$ 311.0890, found 311.0890.

***rac*-(1*R*,2*S*)-1-Benzoyl-6,8-dimethyl-2-phenyl-6,8-diazaspiro[3.5]nonane-5,7,9-trione (7a) and *rac*-(6*R*,7*S*)-7-benzoyl-1,3-dimethyl-6-phenyl-6,7-dihydro-4*H*-pyrano[2,3-*d*]pyrimidine-2,4(3*H*,5*H*)-dione (8a) (Scheme 4).**

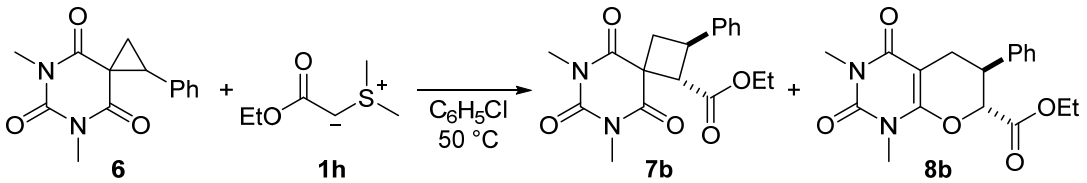


Sulfonium ylide **1a** (54 mg, 0.30 mmol) was added to a solution of 5,7-dimethyl-1-phenyl-5,7-diazaspiro[2.5]-octane-4,6,8-trione (**6**) (52 mg, 0.20 mmol) in C_6H_5Cl (1.0 mL) at room temperature. After stirring at 50 °C for 4 h, the reaction was cooled to room temperature. Evaporation in vacuo furnished the crude product, which was purified by column chromatography (silica gel, 10% EtOAc in hexane) to provide **7a** (48 mg, 64%) as a white solid and **8a** (6 mg, 8%) as a pale yellow solid.

7a: mp 148.0–148.5 °C; IR (film, cm^{-1}) ν 3020, 1744, 1674, 1457, 1421, 1383, 1215; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (d, *J* = 6.9 Hz, 2H), 7.48–7.39 (m, 5H), 7.32 (tt, *J* = 7.3, 1.6 Hz, 1H), 7.23 (t, *J* = 8.0 Hz, 2H), 4.70 (d, *J* = 9.6 Hz, 1H), 4.31 (q, *J* = 9.6 Hz, 1H), 3.36 (s, 3H), 3.24 (s, 3H), 2.96 (t, *J* = 10.5 Hz, 1H), 2.80 (dd, *J* = 10.8, 9.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.3, 171.0, 170.6, 150.9, 142.2, 134.8, 133.5, 128.9, 128.4, 128.2, 127.8, 127.6, 60.5, 49.4, 38.9, 36.0, 28.9, 28.6; HRMS (ESI) *m/z* calcd for $C_{22}H_{20}N_2NaO_4$ $(M+Na)^+$ 399.1315, found 399.1317.

8a: mp 180.5–181.0 °C; IR (film, cm^{-1}) ν 3026, 2925, 1744, 1674, 1651, 1453, 1382, 1223; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 6.9 Hz, 2H), 7.63 (t, *J* = 7.6 Hz, 1H), 7.50 (t, *J* = 7.8 Hz, 2H), 7.31–7.24 (m, 5H), 5.87 (d, *J* = 4.6 Hz, 1H), 3.65 (q, *J* = 5.6 Hz, 1H), 3.38 (s, 3H), 3.37 (s, 3H), 2.80 (dd, *J* = 16.7, 5.3 Hz, 1H), 2.67 (dd, *J* = 16.7, 6.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.5, 162.7, 155.1, 150.9, 139.5, 134.3, 133.8, 129.1, 129.0, 128.5, 127.6, 127.3, 85.8, 82.1, 38.3, 28.8, 28.0, 22.0; HRMS (ESI) *m/z* calcd for $C_{22}H_{21}N_2O_4$ $(M+H)^+$ 377.1496, found 377.1497.

***rac*-(1*R*,2*S*)-1-Benzoyl-7,7-dimethyl-2-phenyl-6,8-dioxaspiro[3.5]nonane-5,9-dione (7b) and *rac*-(2*R*,3*S*)-ethyl 6,8-dimethyl-5,7-dioxo-2-phenyl-2,3-dihydro-4*H*-pyrano[5,6-*d*]pyrimidine-2-carboxylate (8b) (Scheme 4).**



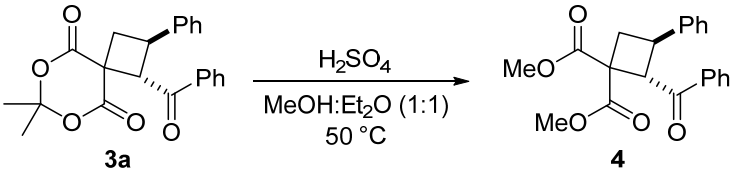
Sulfonium ylide **1h** (67 mg, 0.45 mmol) was added to a solution of **6** (77 mg, 0.30 mmol) in C_6H_5Cl (1.5 mL) at room temperature. After stirring at 50 °C for 2 h, the reaction was cooled to room temperature. Evaporation in vacuo furnished the crude product, which was purified by column chromatography (silica gel, 25% EtOAc in hexane) to provide **7b** (85 mg, 83%) as a white solid and **8b** (9 mg, 9%) as a white solid.

7b: mp 80.5–81.0 °C; IR (film, cm^{-1}) ν 3018, 1749, 1703, 1651, 1489, 1216, 1183; ^1H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 7.3 Hz, 2H), 7.37 (t, *J* = 7.5 Hz, 2H), 7.37 (m, 1H), 4.28 (q, *J* = 9.8 Hz, 1H), 4.11 (qd, *J* = 7.2, 2.2 Hz, 2H), 4.00 (d, *J* = 10.0 Hz, 1H), 3.37 (s, 3H), 3.36 (s, 3H), 2.84–2.73 (m, 2H), 1.19 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 170.3, 170.2, 151.1, 142.0, 128.6, 127.2, 127.1, 61.2, 52.9, 49.2, 37.9, 35.9, 29.1, 28.7, 14.1; HRMS (ESI) *m/z* calcd for $C_{18}H_{20}N_2NaO_5$ $(M+Na)^+$ 367.1264, found 367.1269.

8b: mp 146.0–147.0 °C; IR (film, cm^{-1}) ν 3028, 2960, 1740, 1677, 1456, 1422, 1383, 1219; ^1H NMR (400 MHz, $CDCl_3$) δ 7.36–7.28 (m, 3H), 7.22 (dd, *J* = 5.3, 3.0 Hz, 2H), 4.90 (d, *J* = 6.0 Hz, 1H), 4.17–4.09 (m, 2H), 3.50 (q, *J* = 6.3 Hz, 1H), 3.39 (s, 3H), 3.36 (s, 3H), 2.78 (dd, *J* = 6.4, 3.2 Hz, 2H), 1.11 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.8, 162.7, 154.8, 150.9, 138.8, 128.9, 127.7, 127.4, 86.4, 80.4, 62.1, 39.2, 28.8, 28.1, 22.7, 13.8; HRMS (ESI) *m/z* calcd for $C_{18}H_{21}N_2O_5$ $(M+H)^+$ 345.1445, found 345.1447.

III. Conversion of spirocyclopropane 3a into cyclobutane 4 (Scheme 3)

***rac*-(1*R*,2*S*)-Dimethyl 2-benzoyl-3-phenylcyclobutane-1,1-dicarboxylate (4).**



3a **4**

conc. H_2SO_4 (0.023 mL, 0.45 mmol) was added to a solution of **3a** (109 mg, 0.30 mmol) in Et_2O/MeOH (1:1, 1.5 mL) at room temperature. After stirring at 50 °C for 24 h, the reaction mixture was cooled to room temperature and diluted with Et_2O (10 mL). The resulting mixture was washed with saturated aqueous $NaHCO_3$ (5 mL) and brine (10 mL), and dried over anhydrous $MgSO_4$. Filtration and evaporation in vacuo furnished the crude product, which was purified by column chromatography (silica gel, 10% EtOAc in hexane) to provide **4** (87 mg, 82%) as a pale yellow oil; IR (film, cm^{-1}) ν 2952, 1734, 1677, 1273, 1165; ^1H NMR (400 MHz, $CDCl_3$) δ 7.99–7.20 (m, 10H), 4.84 (d, J = 10.1 Hz, 1H), 4.19 (dt, J = 9.1 Hz, 10.1 Hz, 1H), 3.80 (s, 3H), 3.59 (s, 3H), 3.19 (dd, J =11.5, 9.1 Hz, 1H), 2.49 (dd, J = 11.5, 10.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.9, 171.2, 169.2, 142.1, 136.2, 133.3, 128.6, 128.6, 128.5, 126.9, 126.8, 53.7, 53.0, 52.8, 52.6, 36.4, 33.4; HRMS (ESI) *m/z* calcd for $C_{21}H_{20}O_5$ $(M+H)^+$ 353.1384, found 353.1382.

References

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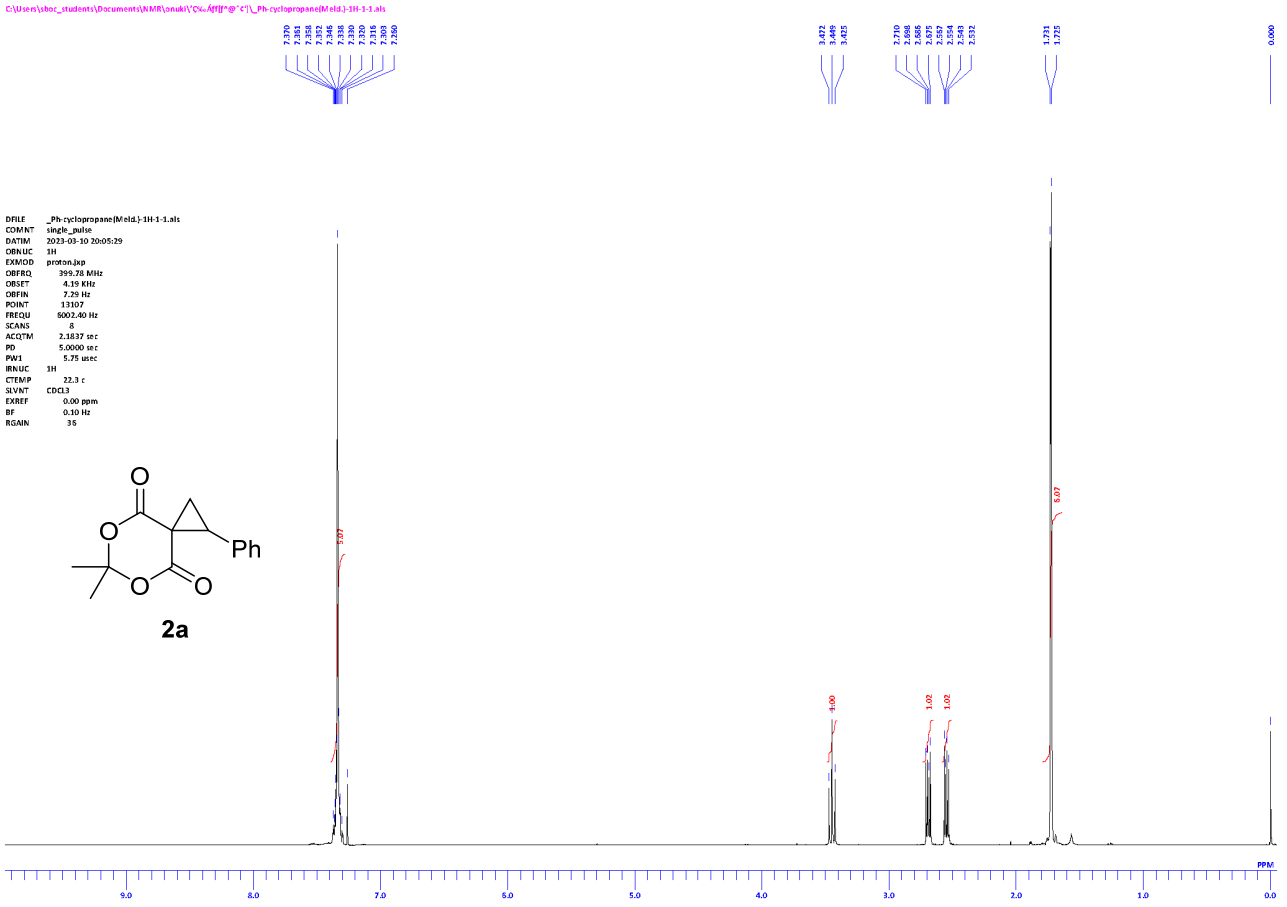
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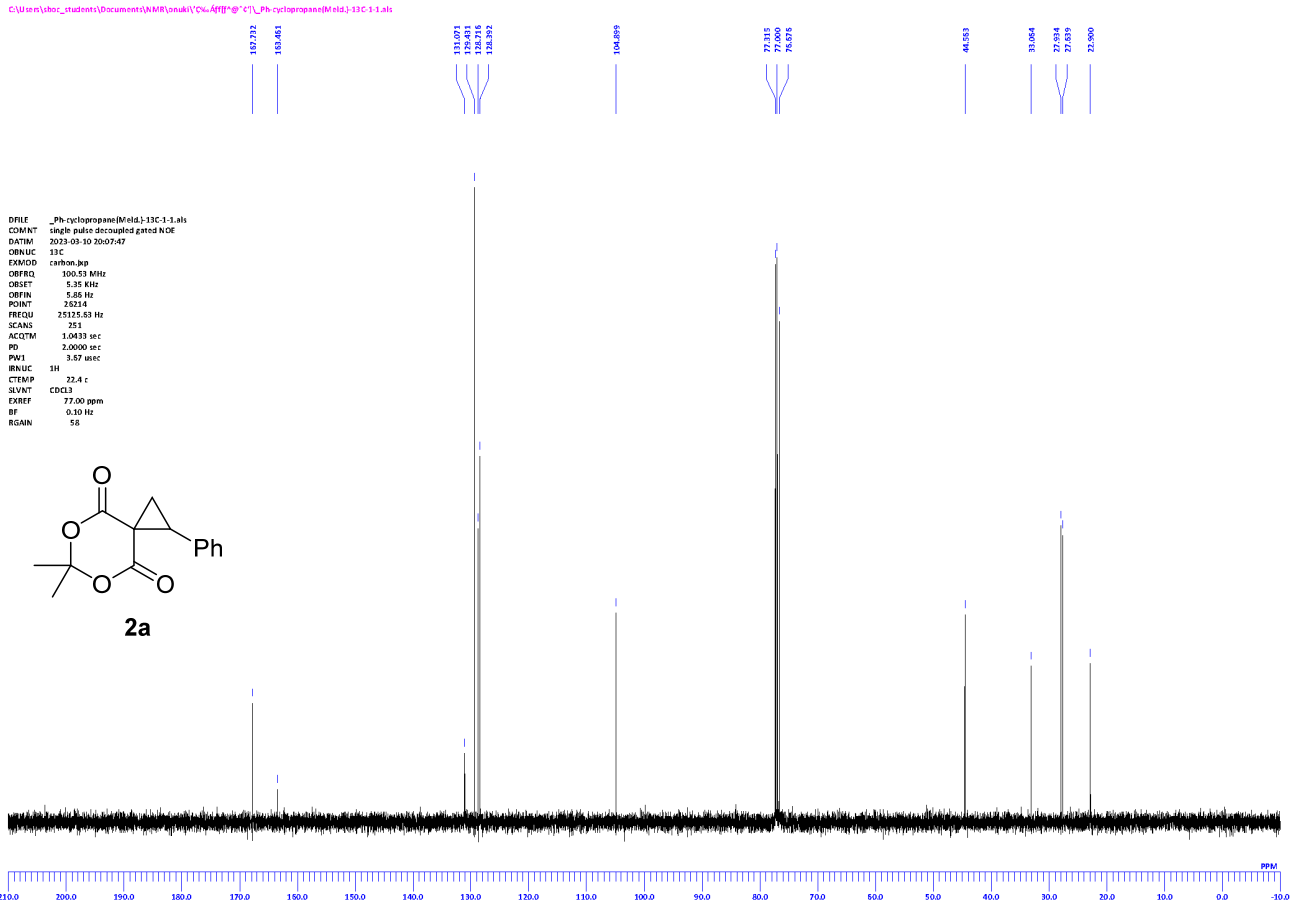
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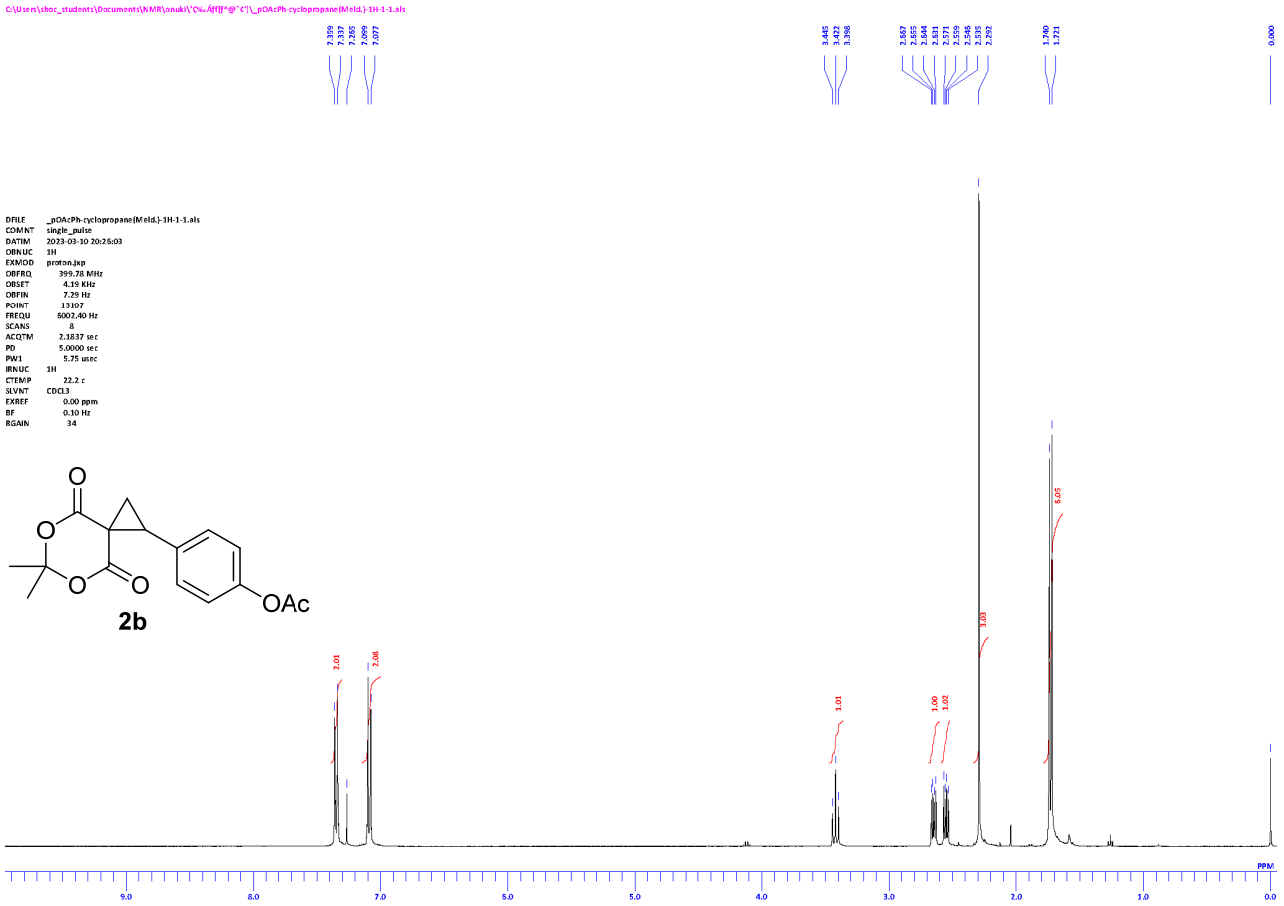
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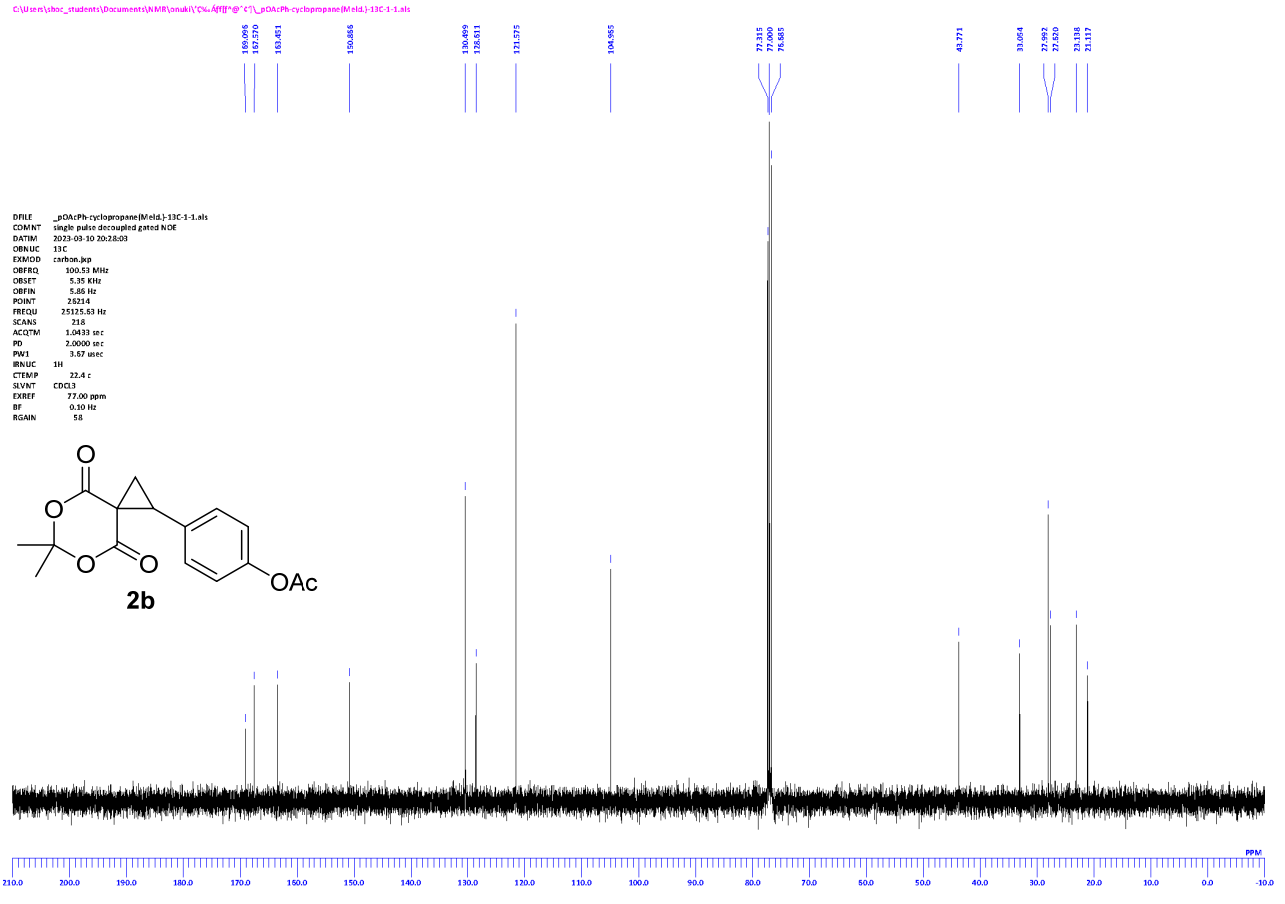
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O
O
O
OAc
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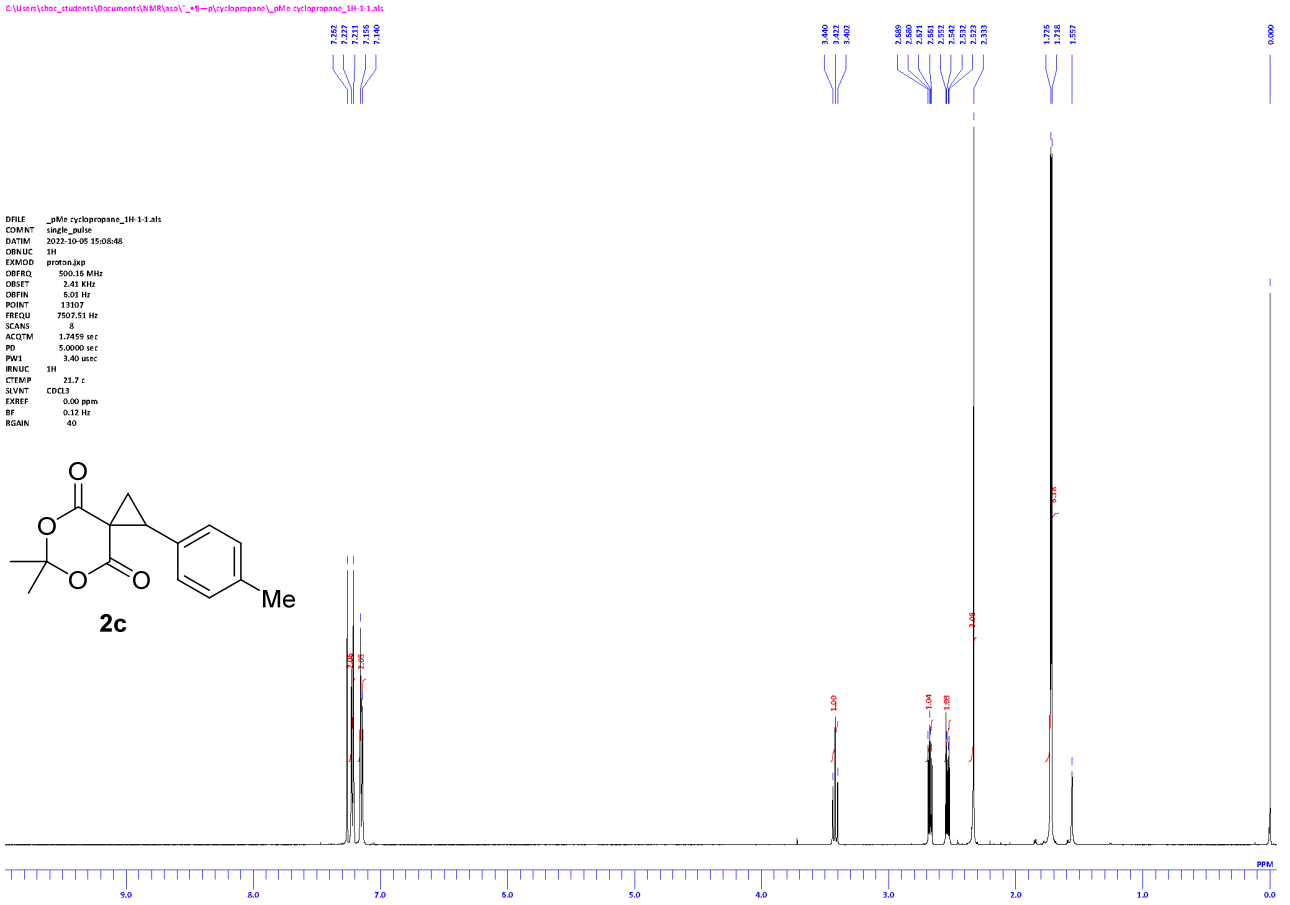
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2c
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3.08
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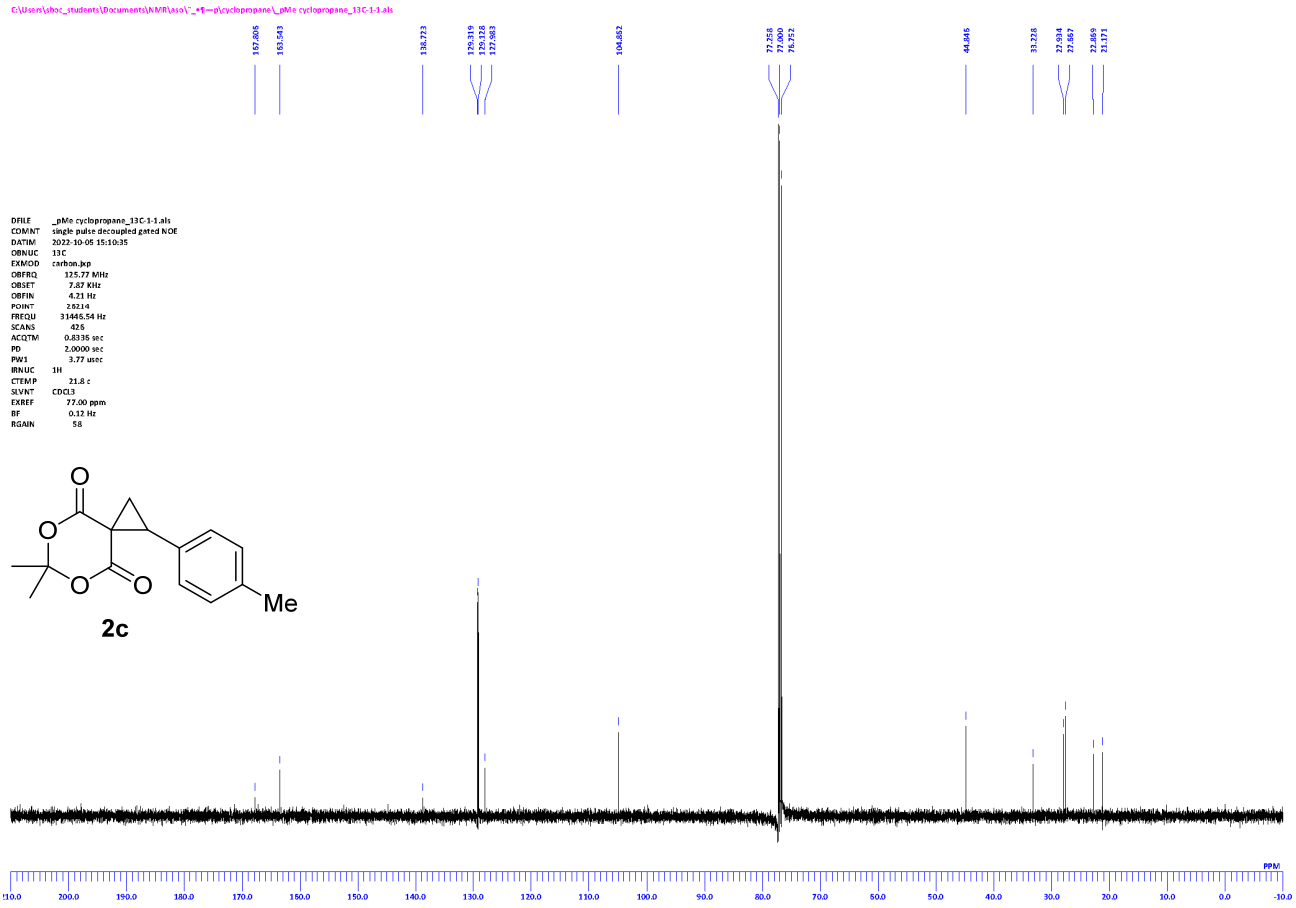
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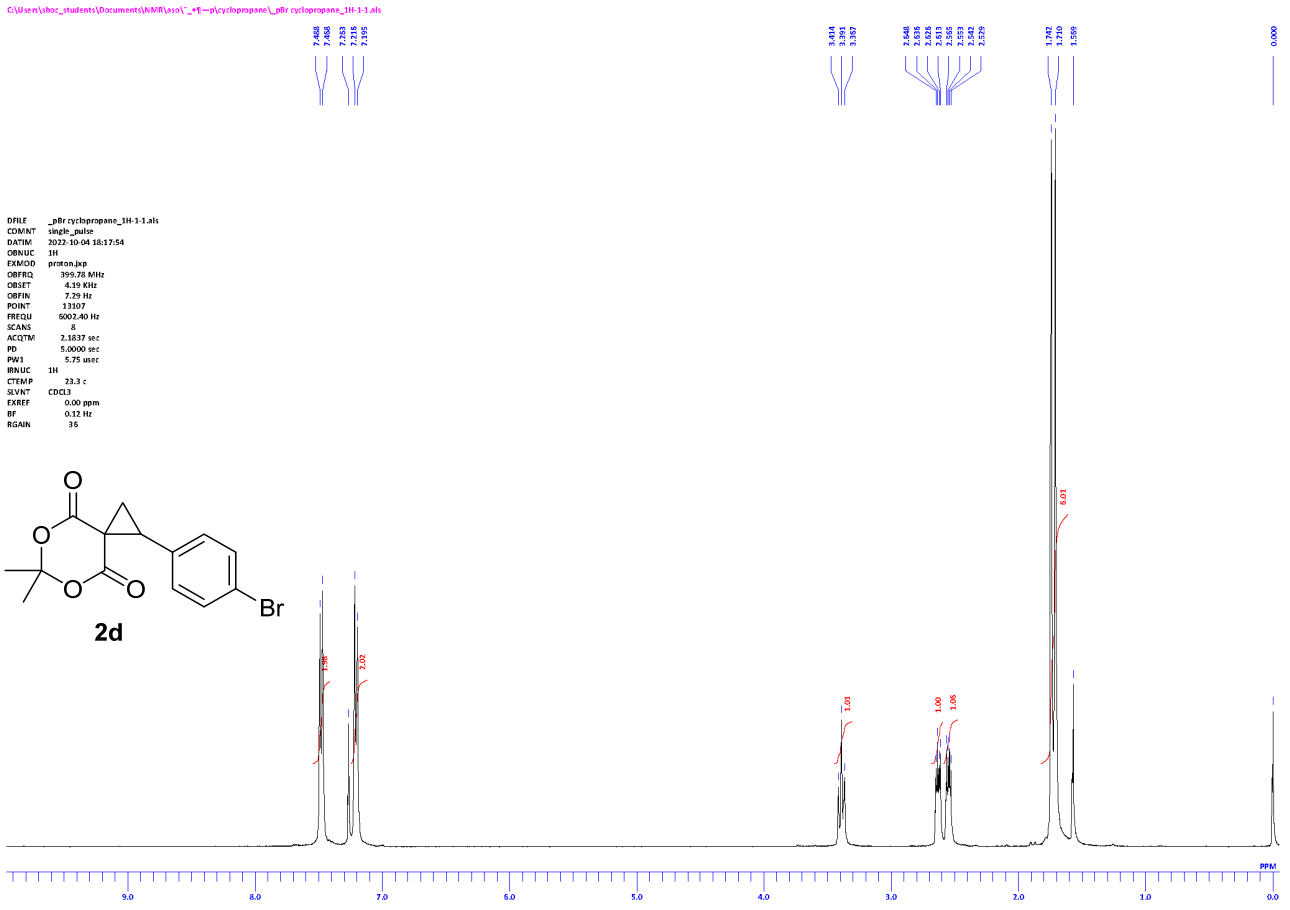
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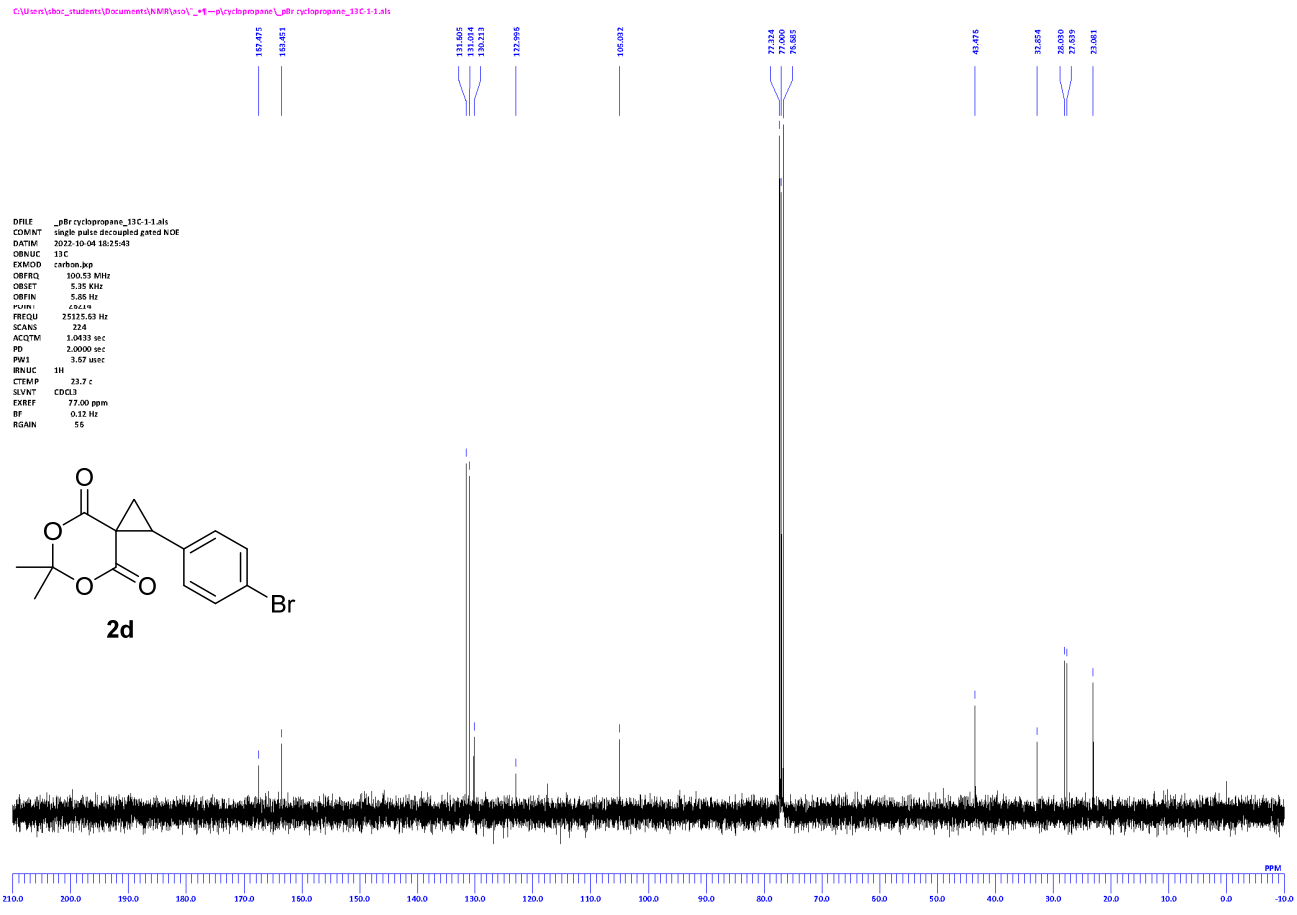
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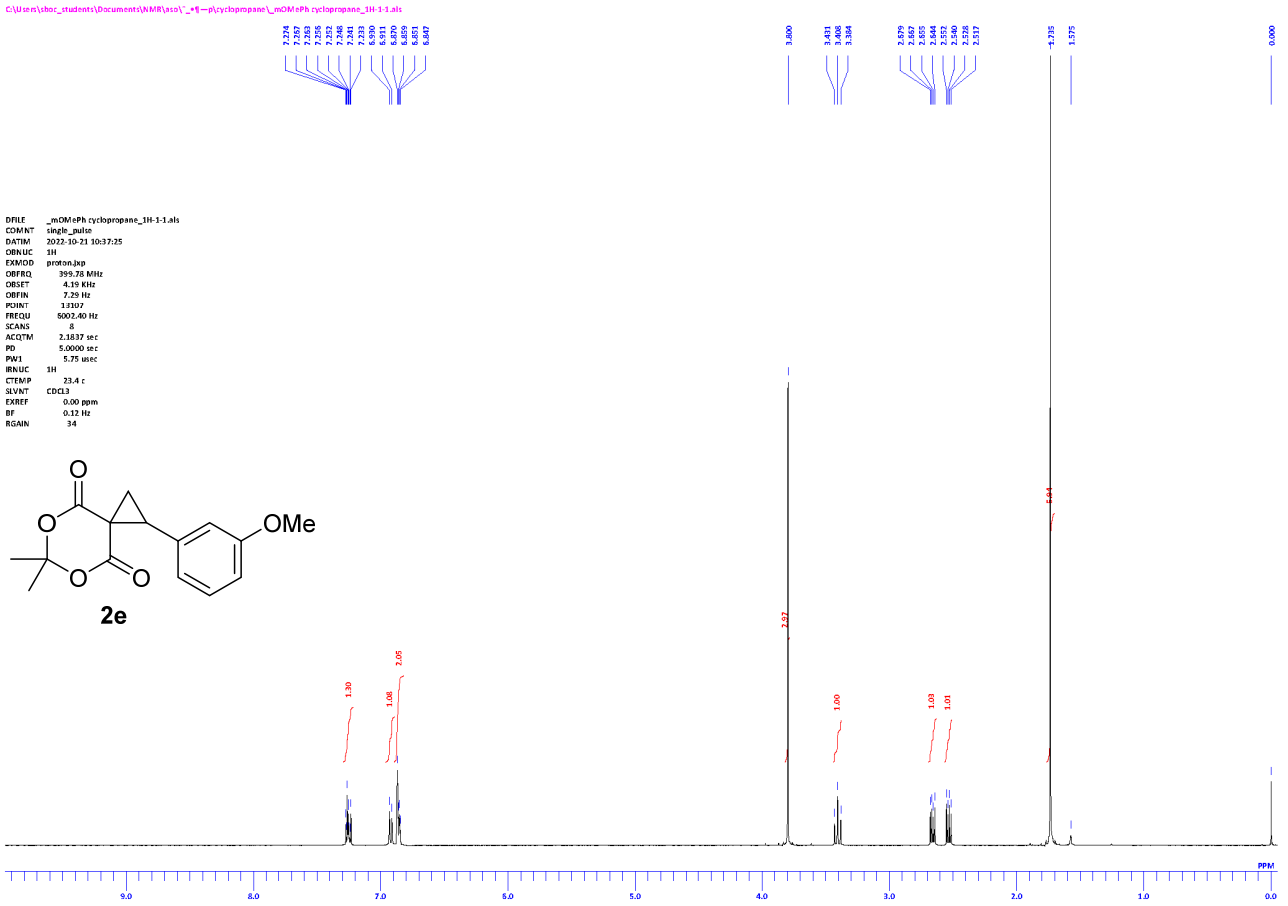
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PW1 5.75 usec
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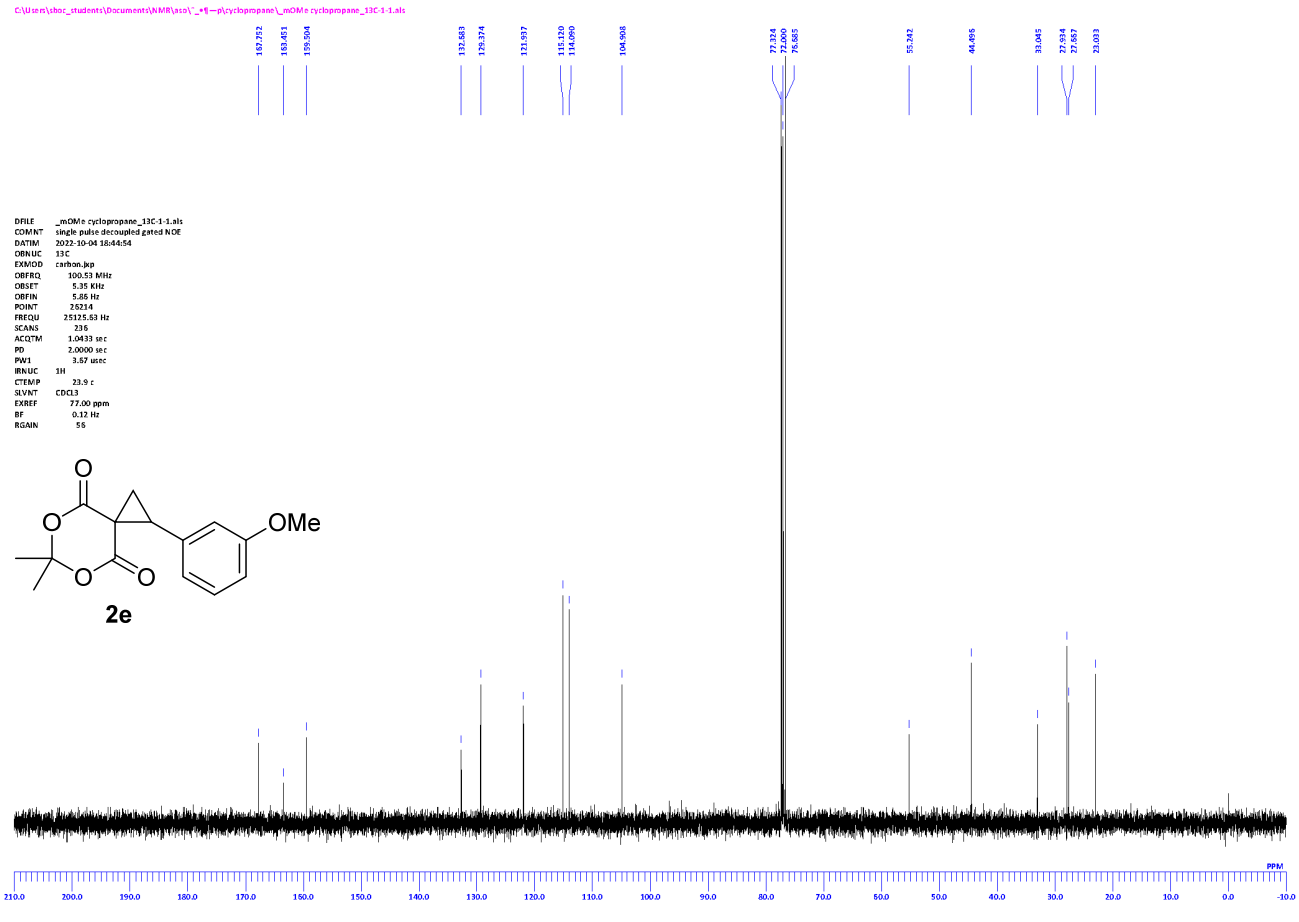
RGAIN 34
OMe
2e
PPM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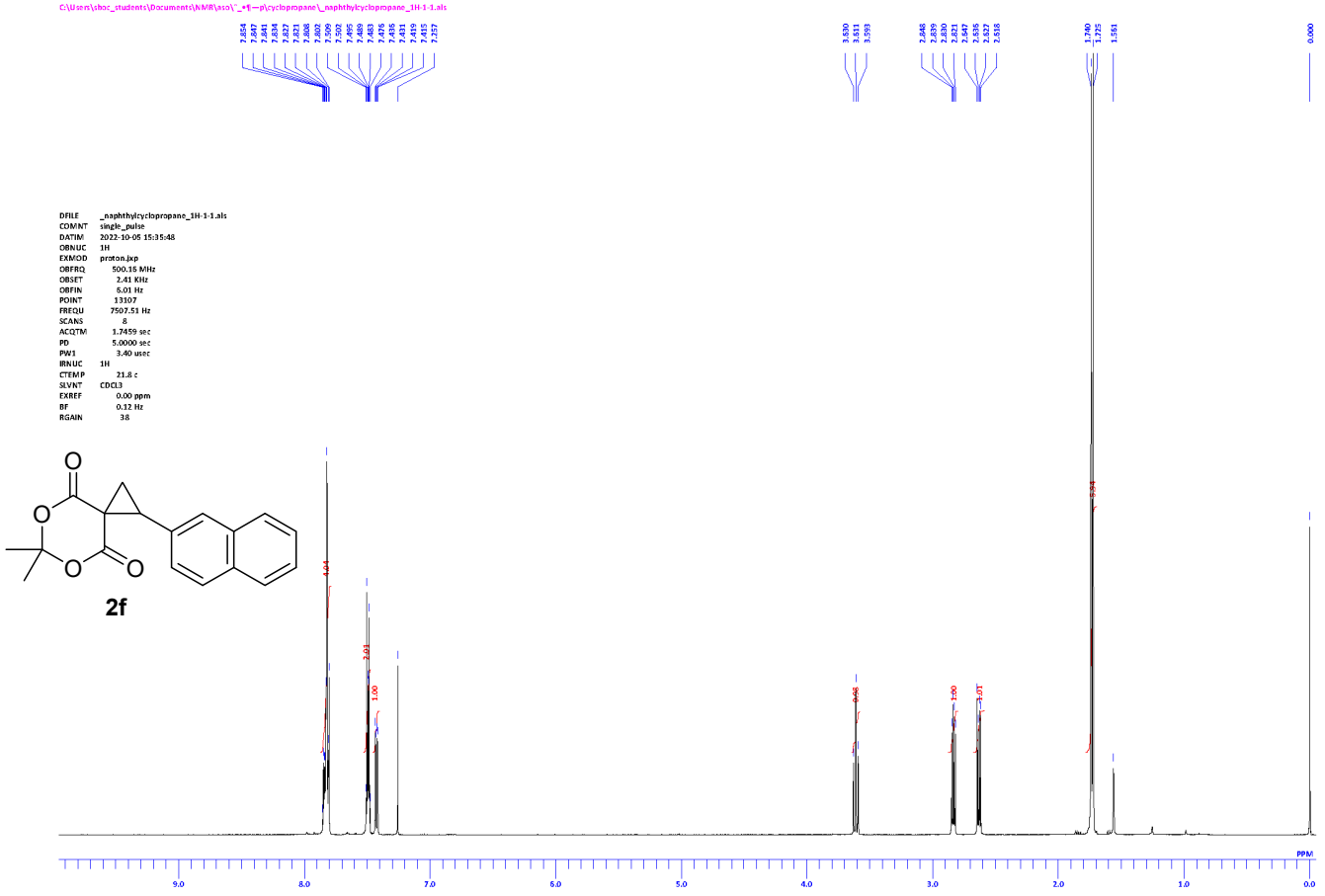
PPM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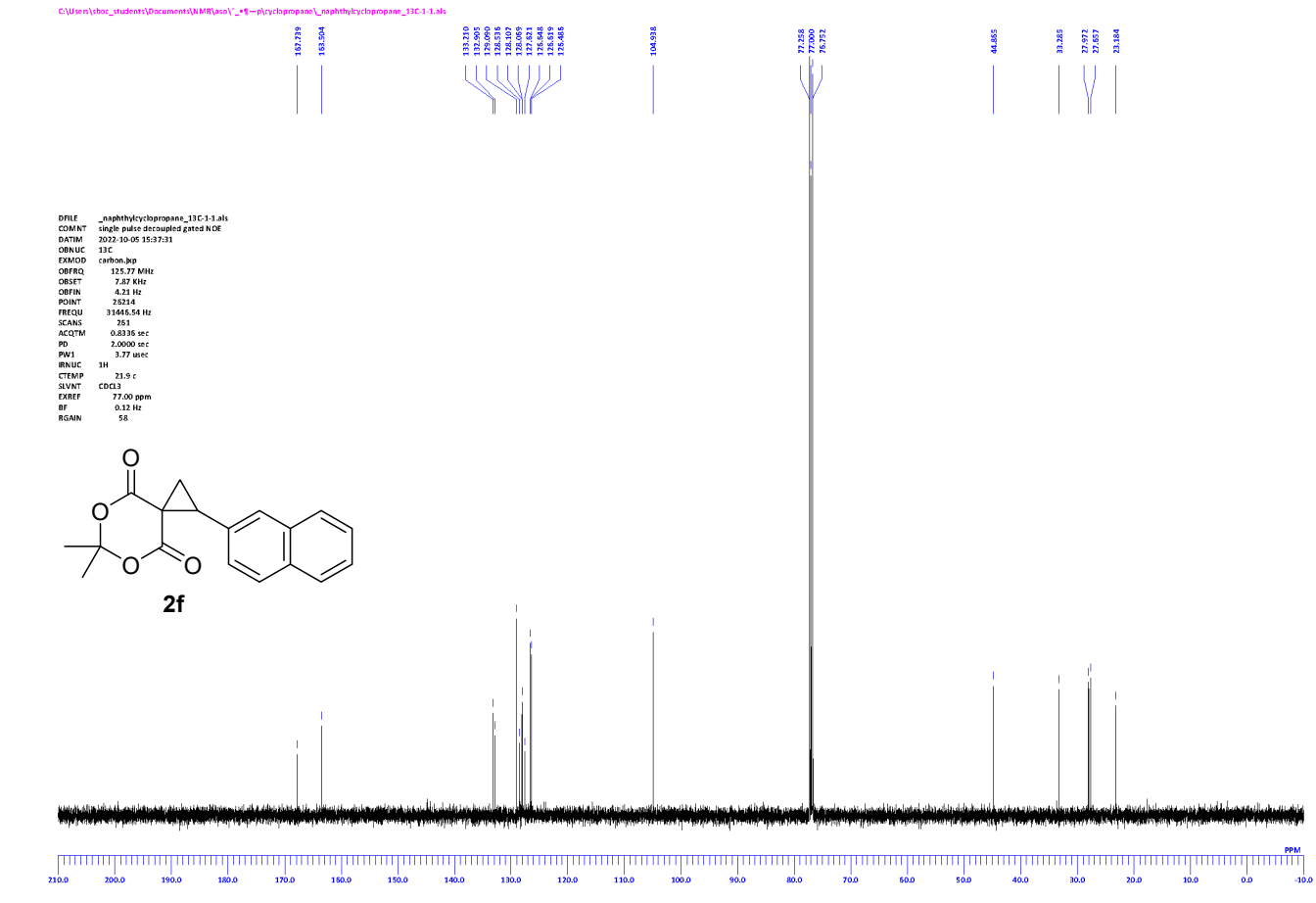
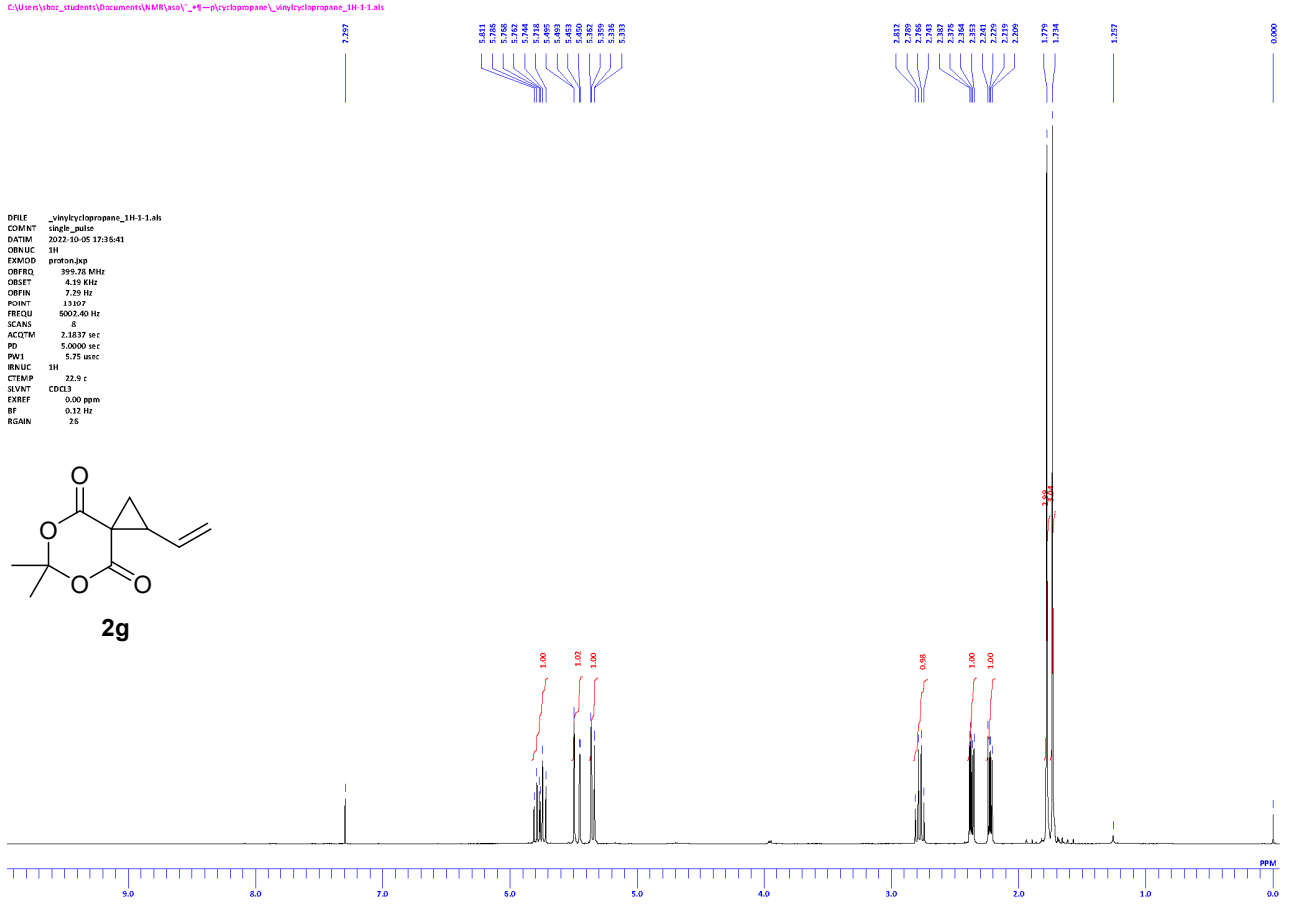
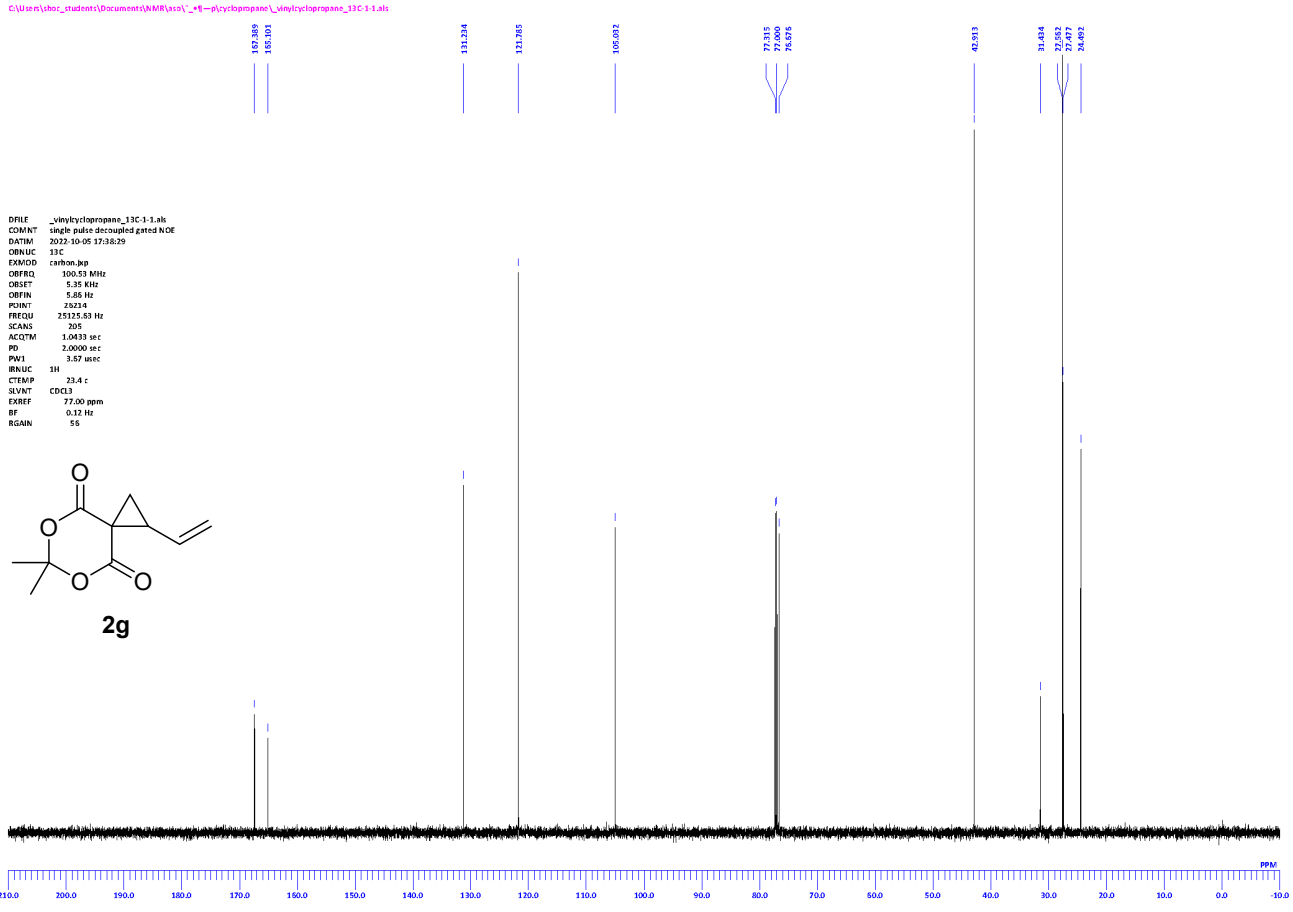
7.821
7.806
7.802
7.509
7.502
7.485
7.480
7.476
7.436
7.431
7.419
7.415
7.257
3.830
3.811
3.593
2.848
2.839
2.830
2.821
2.547
2.536
2.527
2.516
1.740
1.725
1.561
0.000
DFILE _naphthylcyclopropane_1H-1-1.als
COMNT single_pulse
DATIM 2022-10-05 15:35:48
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38
1.01
2.01
1.00
0.98
1.00
3.07
5.64
PPM
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

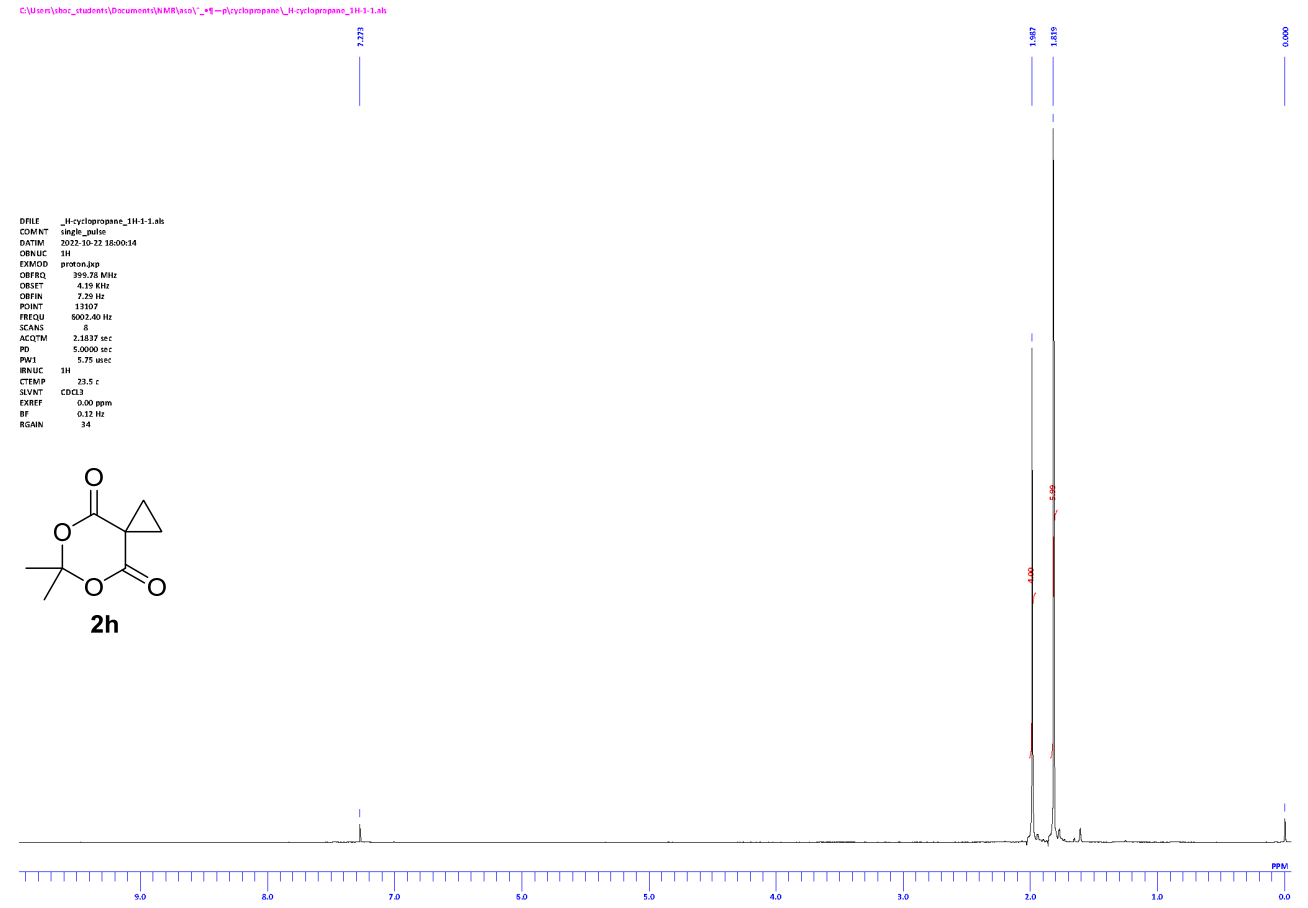
2f

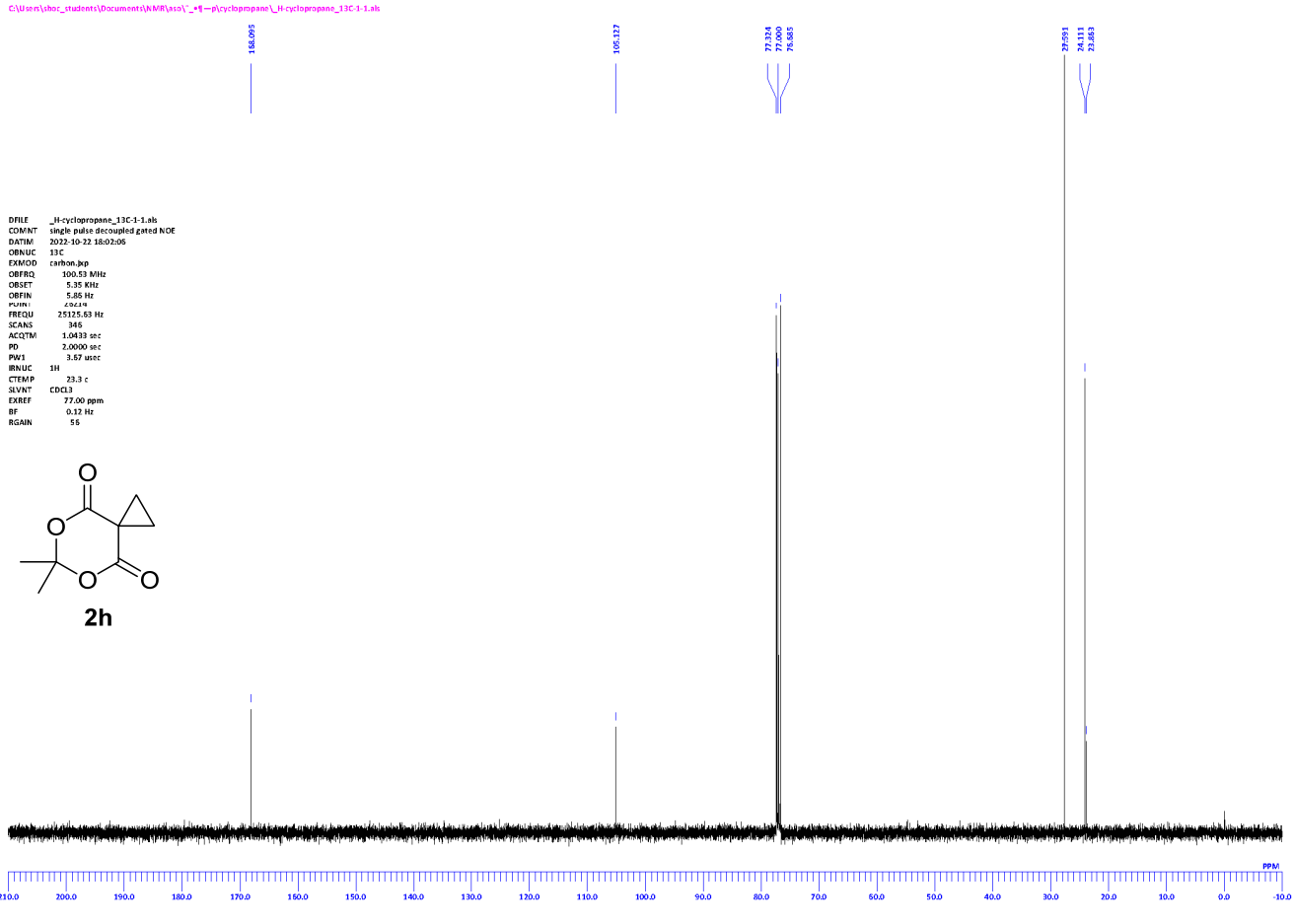

C:\Users\sbcc_students\Documents\NMR\sso\"_•¶—p\cyclopropane_naphthylcyclopropane_13C-1-1.als
167.739
163.504
133.210
132.905
129.090
128.376
128.103
128.069
127.621
126.648
126.469
125.485
104.938
77.258
77.000
76.742
44.885
33.285
27.972
27.657
23.184
DFILE _naphthylcyclopropane_13C-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2022-10-05 15:37:31
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 25214
FREQU 31446.54 Hz
SCANS 251
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.77 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 58
PPM
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
-10.0

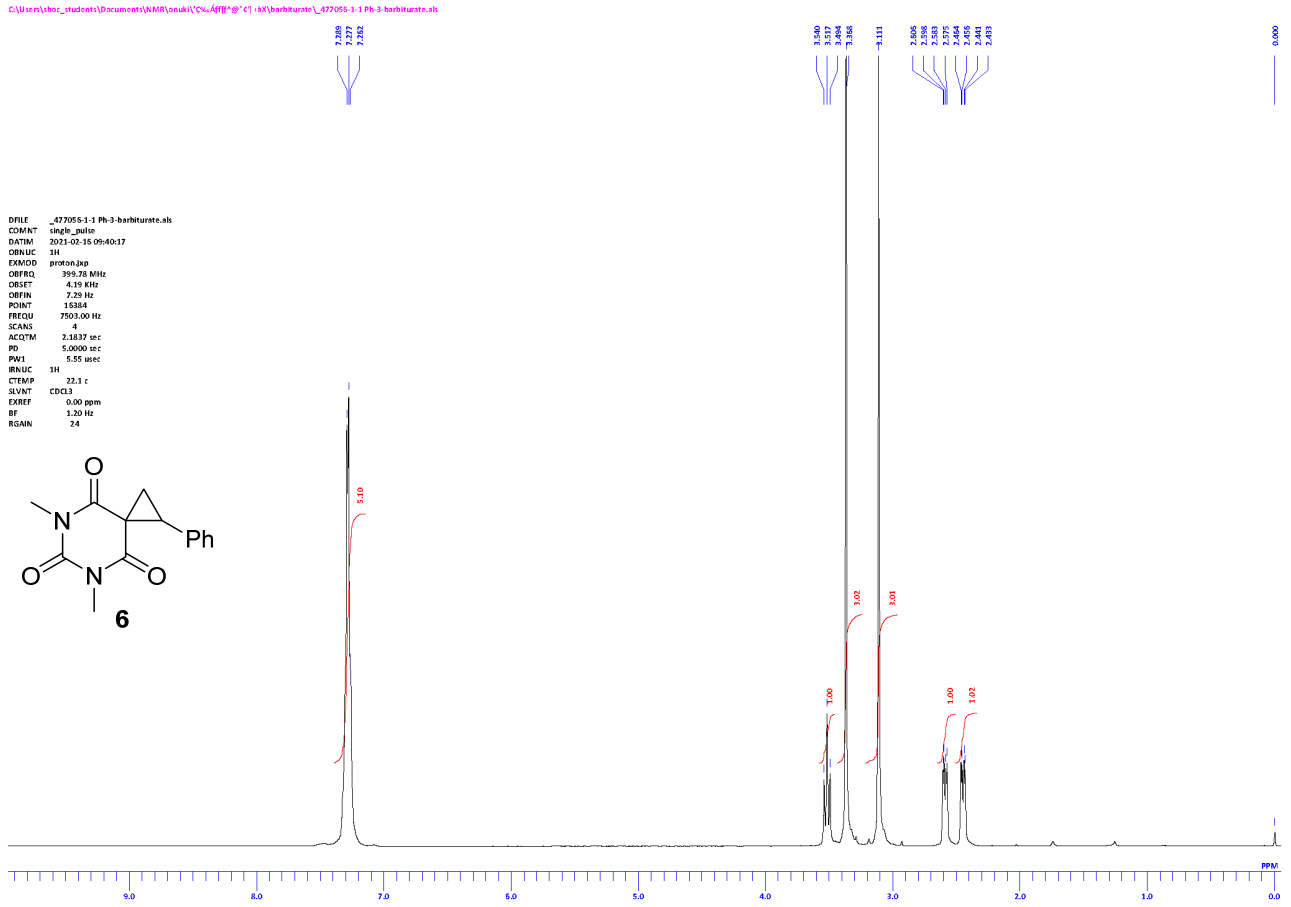
2f

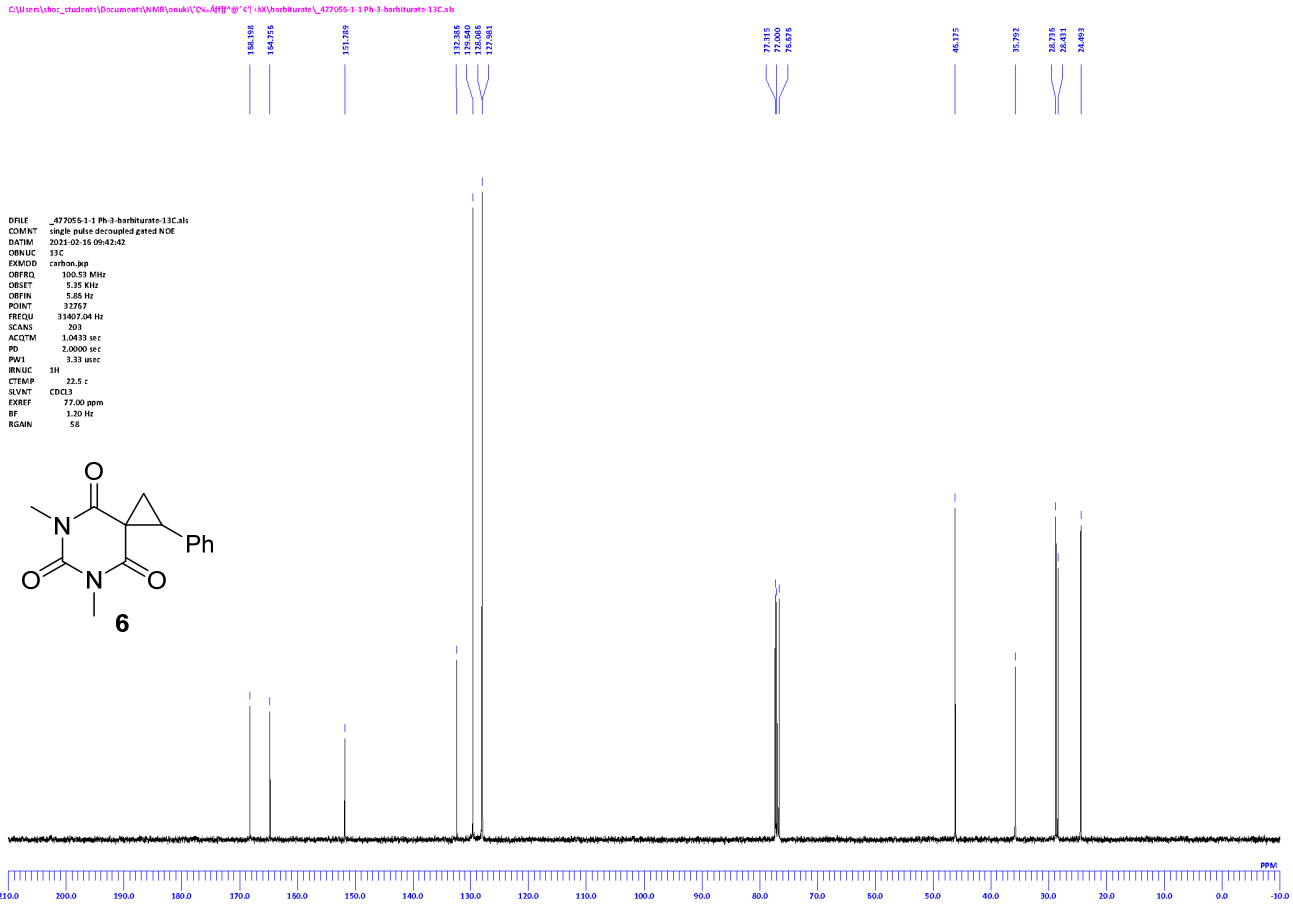

C:\Users\sbcc_students\Documents\NMR\sss\~_•¶—p\cyclopropane_vinylcyclopropane_1H-1-1.als
7.297
5.811
5.806
5.788
5.782
5.744
5.736
5.495
5.493
5.483
5.480
5.362
5.359
5.336
5.333
2.812
2.789
2.766
2.743
2.730
2.375
2.364
2.353
2.341
2.229
2.219
2.209
1.779
1.734
1.257
0.000
DFILE _vinylcyclopropane_1H-1-1.als
COMNT single_pulse
DATIM 2022-10-05 17:36:41
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 22.9 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 26
2g
1.00
1.02
1.00
0.98
1.00
1.00
PPM
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

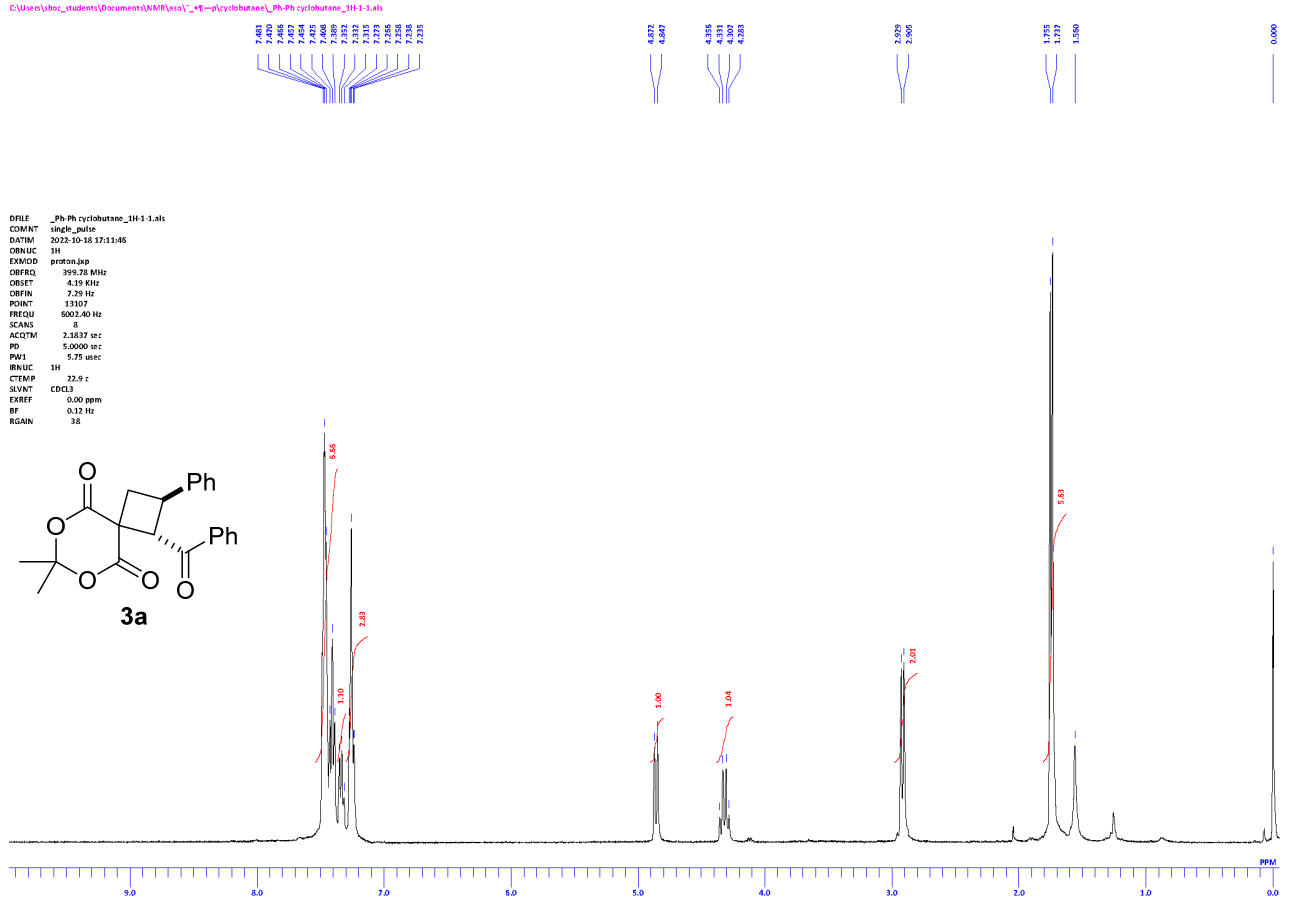

C:\Users\sbcc_students\Documents\NMR\sss\~_•¶—p\cyclopropane_vinylcyclopropane_13C-1-1.als
167.389
165.101
131.234
121.765
105.032
77.315
77.000
76.678
42.913
31.434
27.562
27.477
24.492
DFILE _vinylcyclopropane_13C-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2022-10-05 17:38:29
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 205
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 23.4 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 56
2g
PPM
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
-10.0

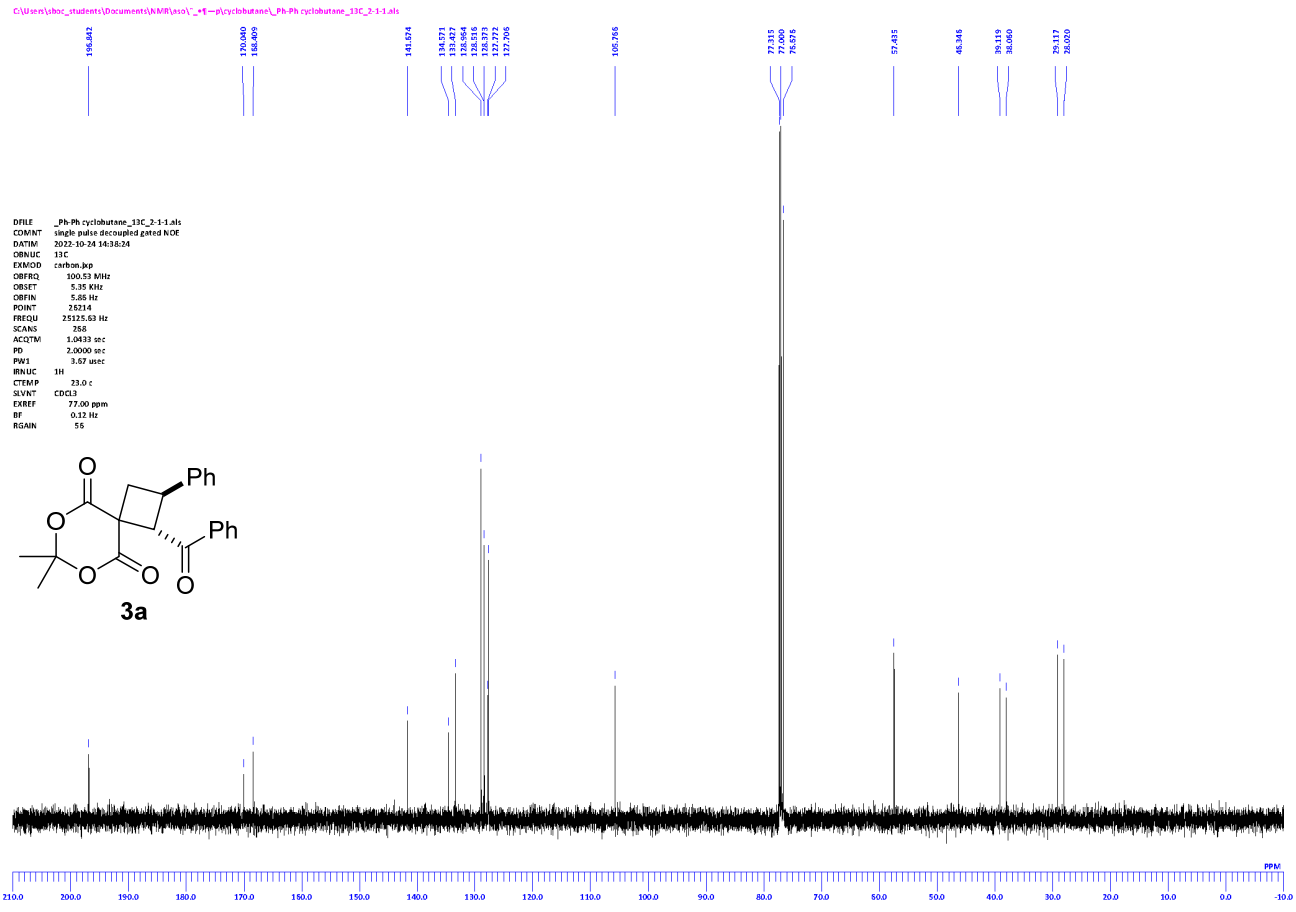

C:\Users\shoc_students\Documents\NMR\sos\"_•¶—p\cyclopropane_H-cyclopropane_1H-1-1.als
7.273
1.587
1.519
0.000
5.00
5.60
DFILE _H-cyclopropane_1H-1-1.als
COMNT single_pulse
DATIM 2022-10-22 18:00:14
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 23.5 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34
2h
PPM
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

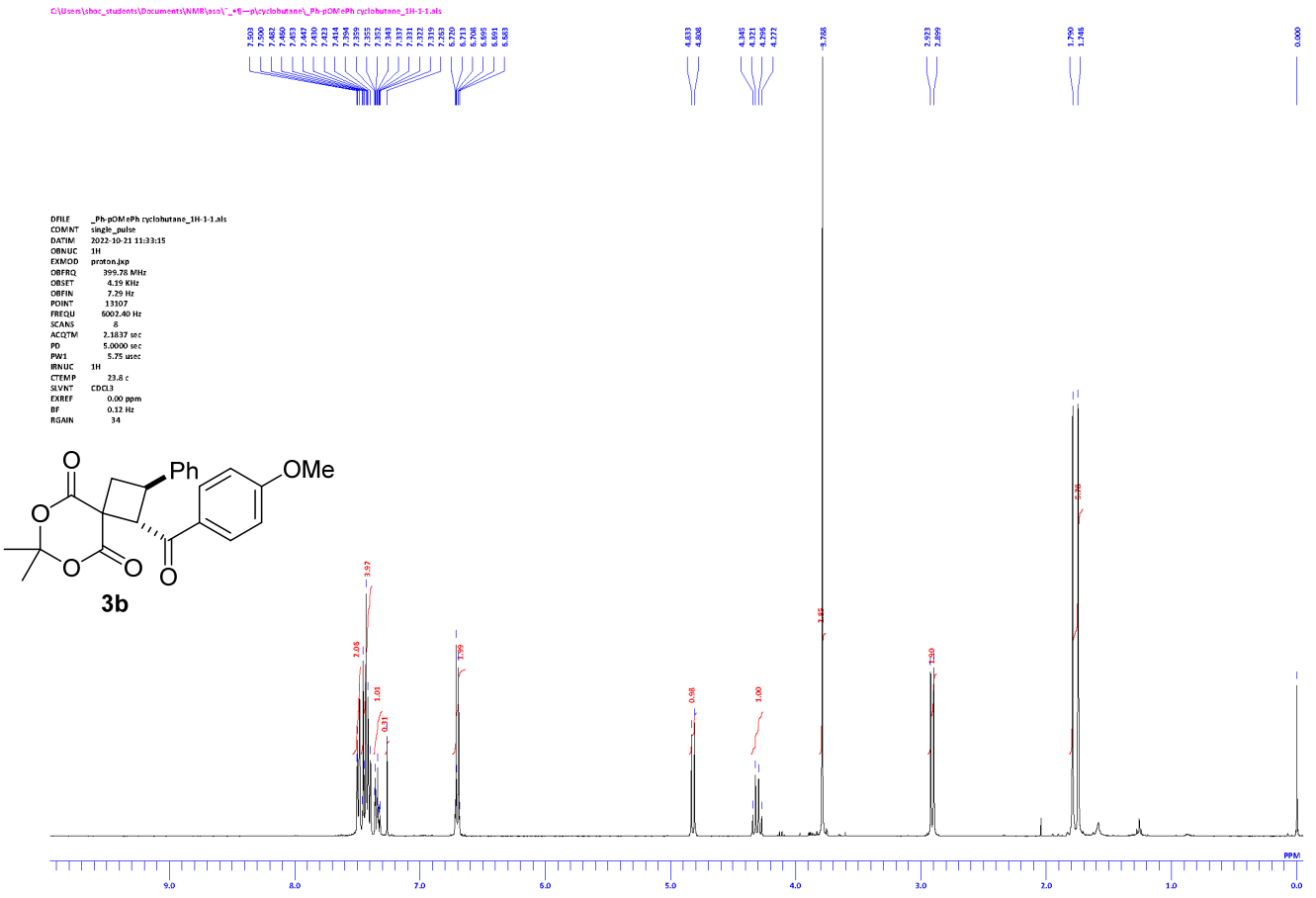

C:\Users\shoc_students\Documents\NMR\sos\"_•¶—p\cyclopropane_H-cyclopropane_13C-1-1.als
166.095
105.127
77.324
77.000
76.685
29.931
24.111
23.863
DFILE _H-cyclopropane_13C-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2022-10-22 18:02:06
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.85 Hz
FREQU 25125.63 Hz
SCANS 346
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 56
2h
PPM
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
-10.0

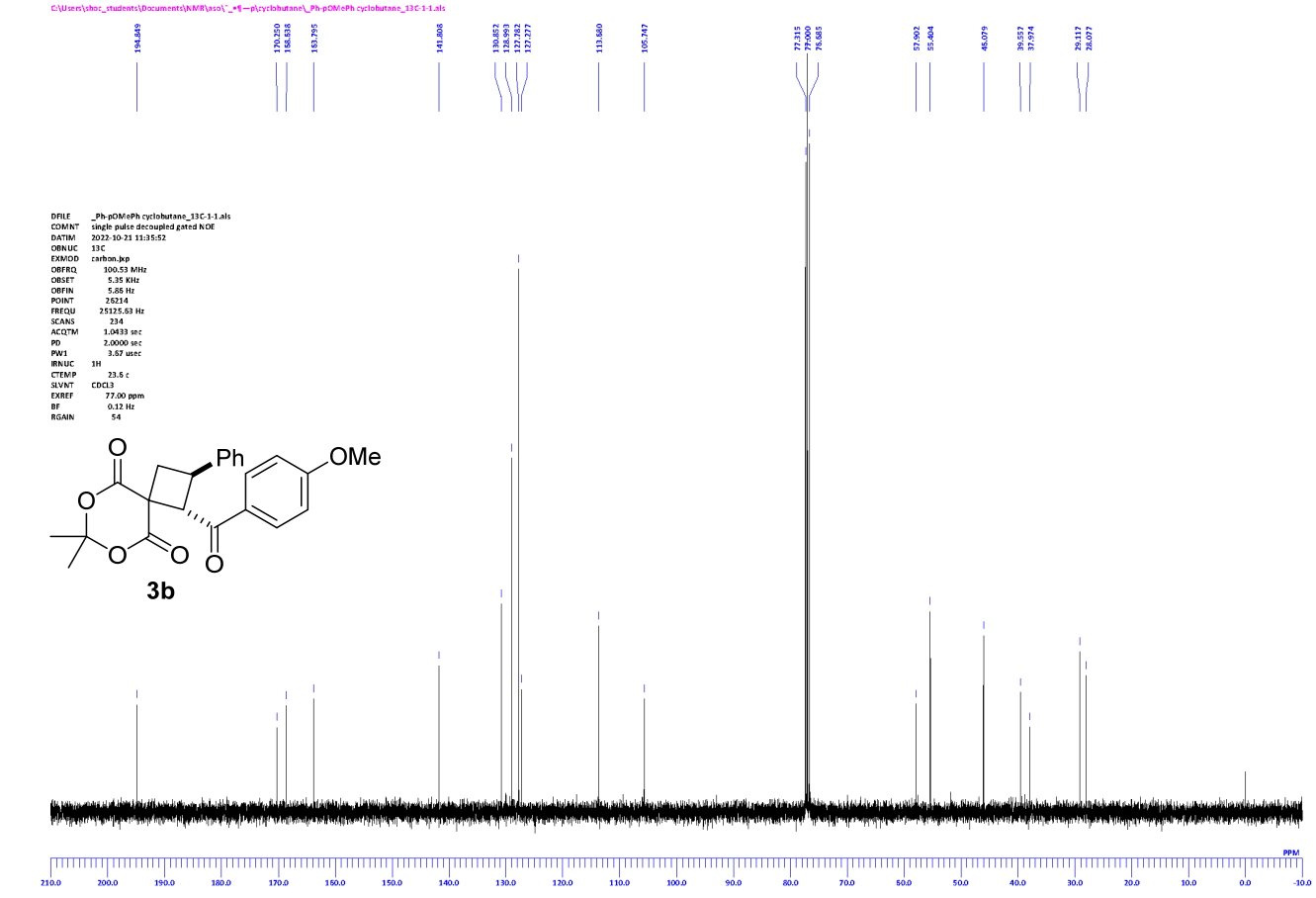

DFILE _477056-1-1 Ph-3-barbiturate.als
COMNT single_pulse
DATIM 2021-02-16 09:40:17
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 4
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 24
O
N
O
N
O
Ph
6
PPM

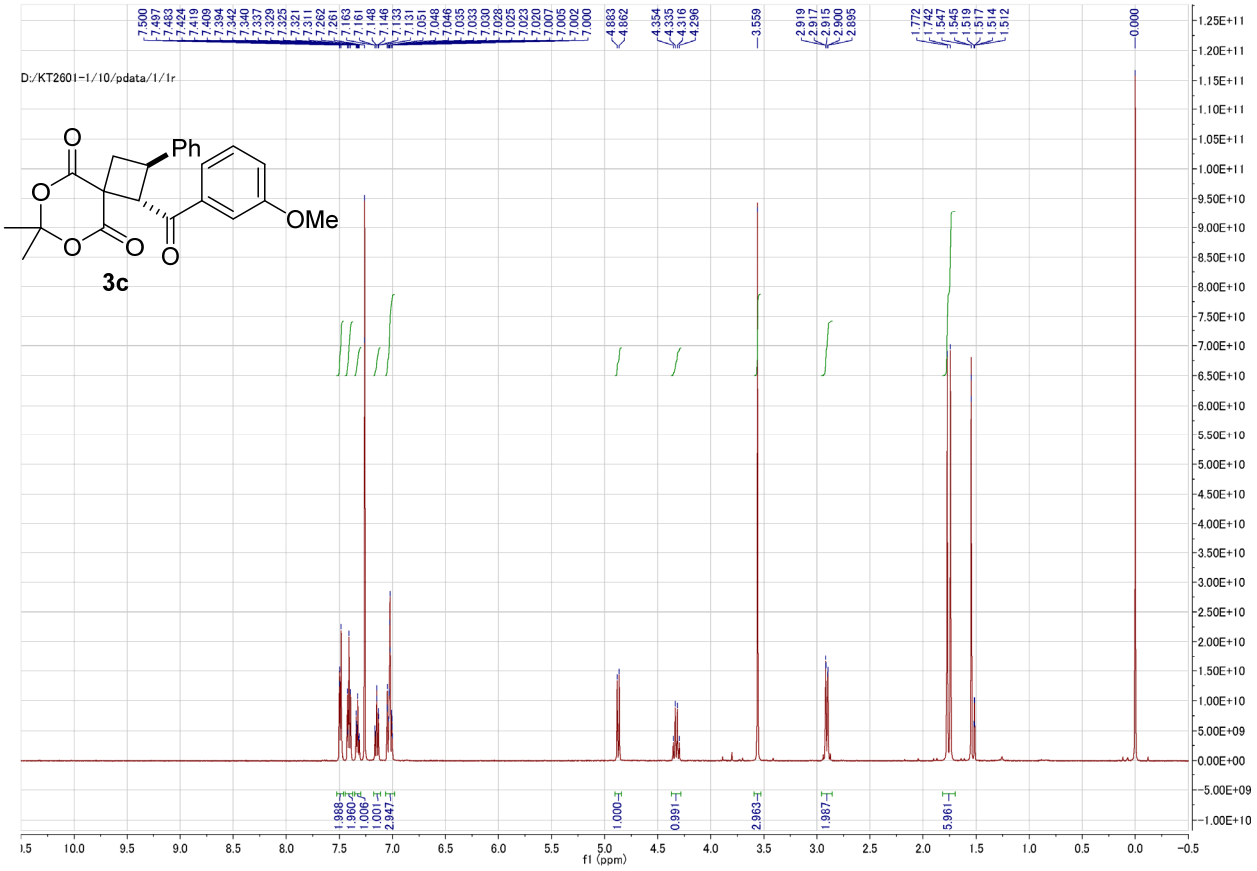

DFILE _477056-1-1 Ph-3-barbiturate-13C.als
COMNT single pulse decoupled gated NOE
DATIM 2021-02-16 09:42:42
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 32767
FREQU 33407.04 Hz
SCANS 203
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 22.5 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 58
O
N
O
N
O
Ph
6
PPM

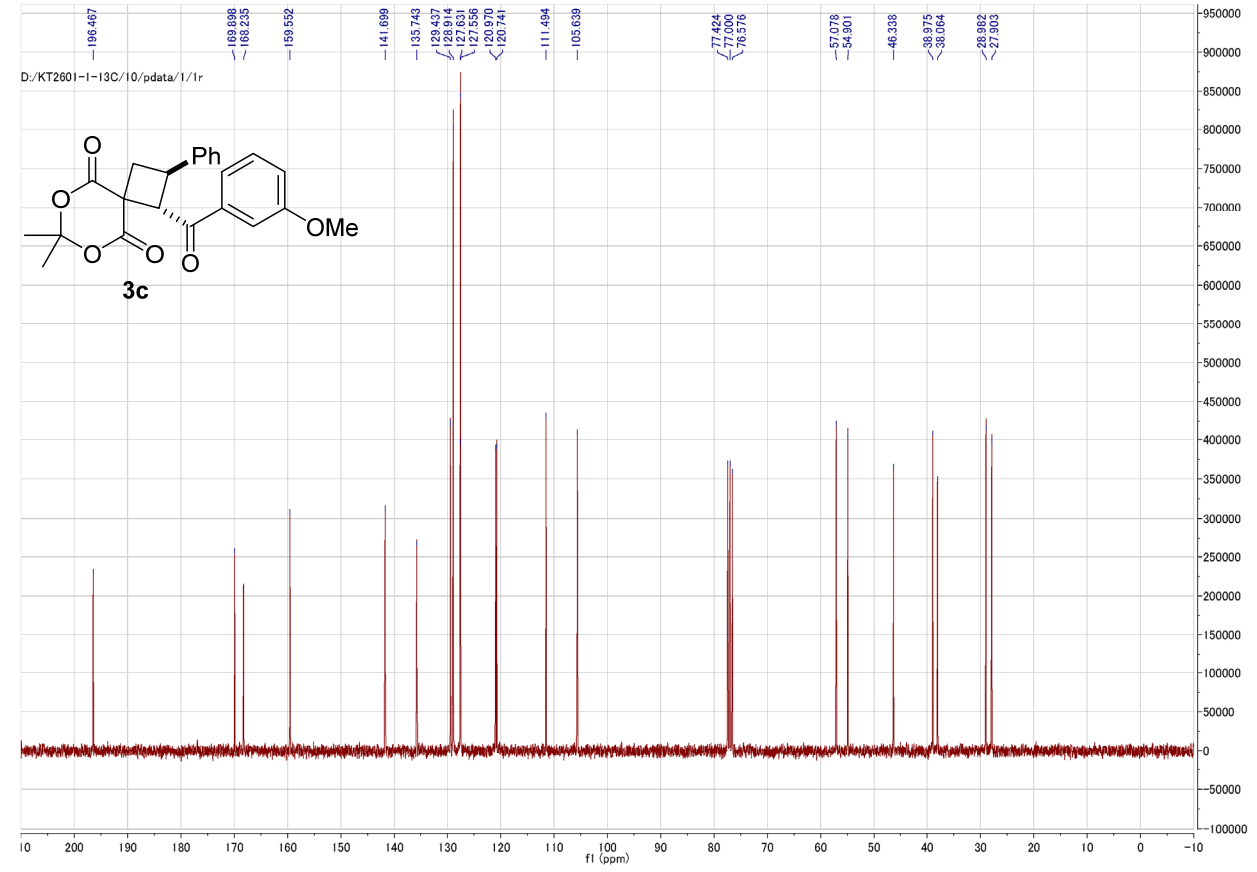

C:\Users\sbcc_students\Documents\NMR\sso\~_•¶—p\cyclobutane_Ph-Ph cyclobutane_1H-1-1.als
7.601 7.600 7.486 7.457 7.454 7.435 7.408 7.389 7.352 7.333 7.311 7.273 7.266 7.258 7.236 7.235
4.872 4.847
4.356 4.331 4.307 4.283
2.929 2.905
1.755 1.737 1.560
0.000
DFILE _Ph-Ph cyclobutane_1H-1-1.als
COMNT single_pulse
DATIM 2022-10-18 17:11:46
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 22.9 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38
O
O
Ph
Ph
O
O
O
3a
5.65
2.83
3.10
1.00
1.04
2.01
5.83
PPM
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

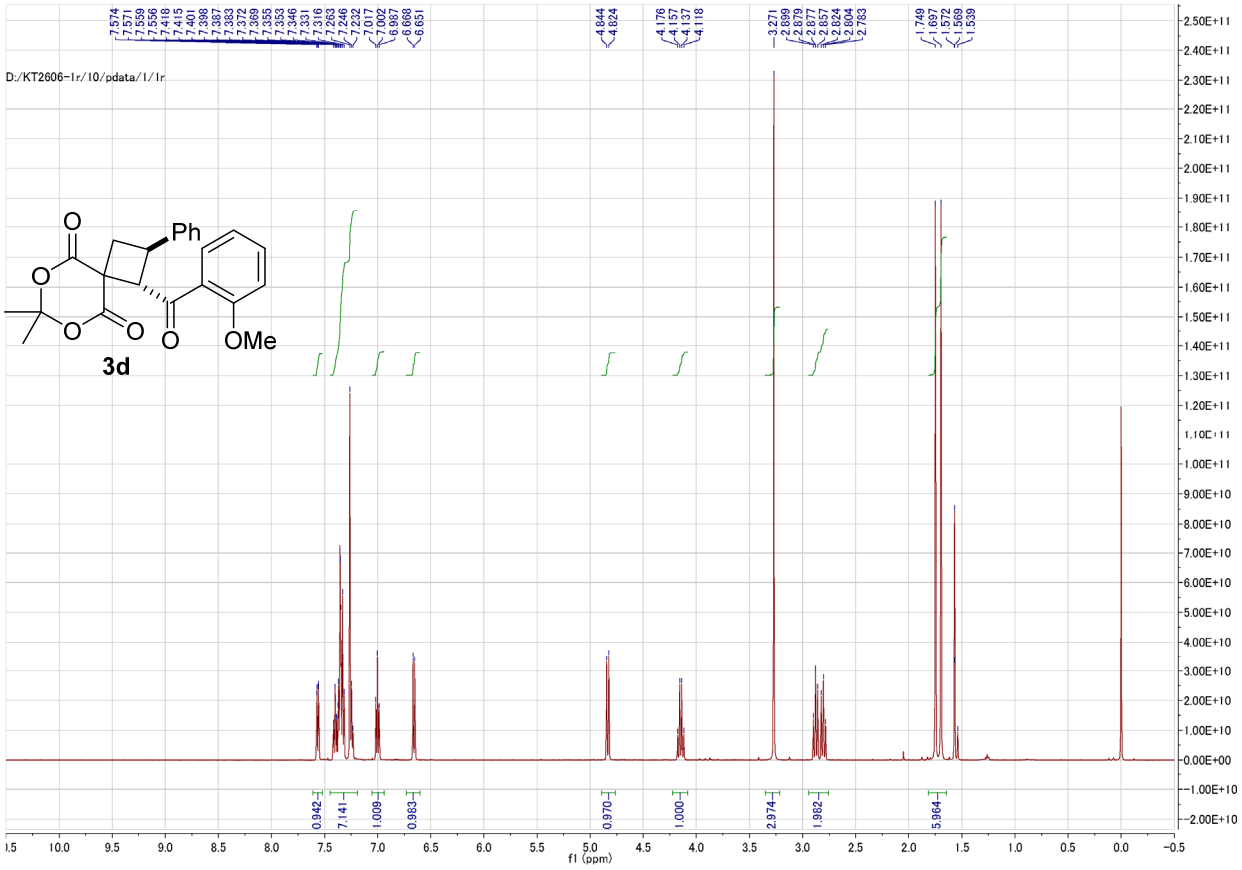

C:\Users\sbcc_students\Documents\NMR\sso\~_•¶—p\cyclobutane_Ph-Ph cyclobutane_13C_2-1-1.als
196.642
170.040
168.409
141.674
134.571
133.427
128.954
128.516
128.373
127.772
127.705
105.766
77.315
77.000
76.676
57.435
45.346
39.119
38.060
29.117
26.020
DFILE _Ph-Ph cyclobutane_13C_2-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2022-10-24 14:38:24
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 288
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 23.0 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 56
O
O
Ph
Ph
O
O
O
3a
PPM
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
-10.0

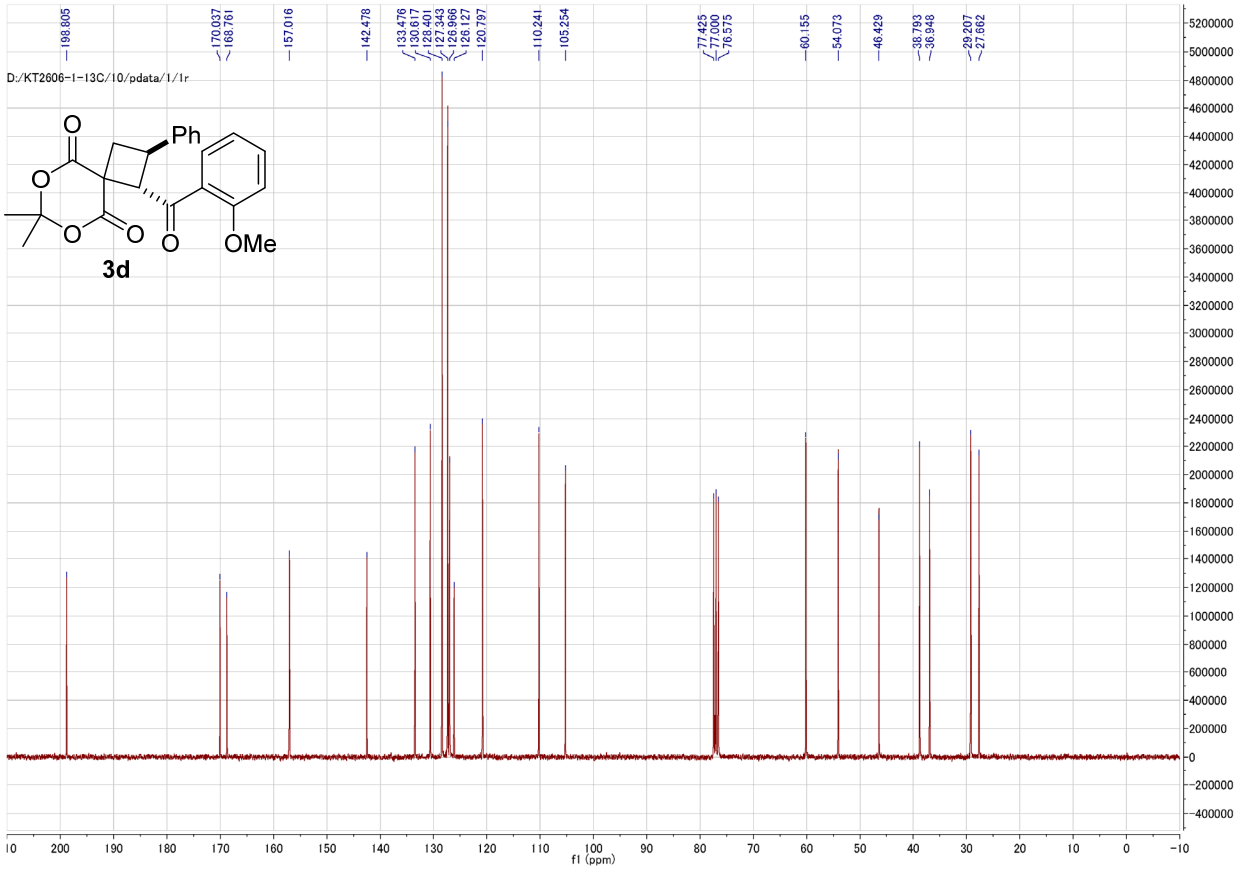

C:\Users\sboc_students\Documents\NMR\sso\~_*¶—p\cyclobutane_Ph-pOMePh cyclobutane_1H-1-1.als
DFILE _Ph-pOMePh cyclobutane_1H-1-1.als
COMNT single_pulse
DATIM 2022-10-21 11:33:15
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 23.8 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34
Ph
OMe
O
O
O
O
O
3b
PPM

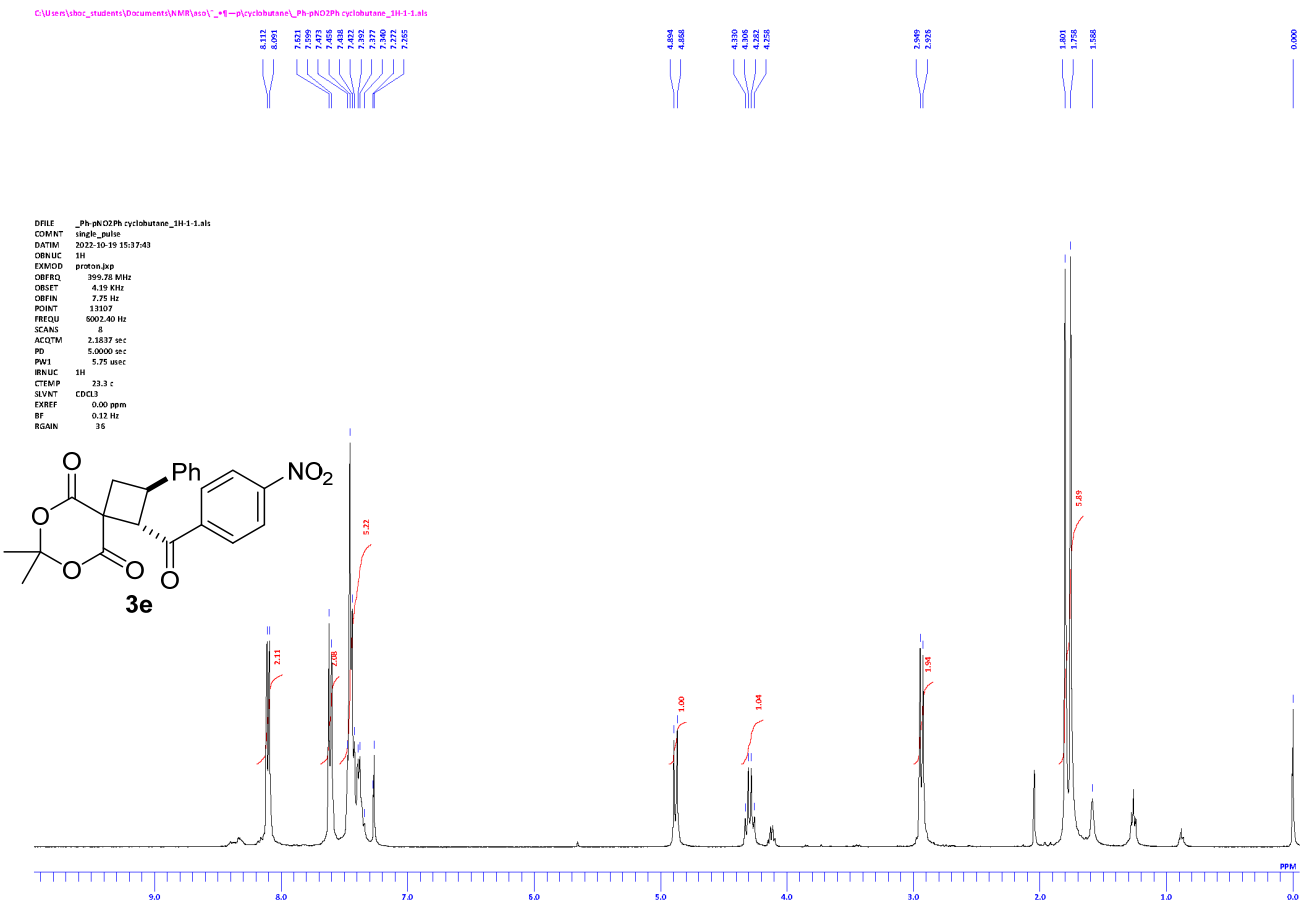

C:\Users\sboc_students\Documents\NMR\sso\~_*¶—p\cyclobutane_Ph-pOMePh cyclobutane_13C-1-1.als
DFILE _Ph-pOMePh cyclobutane_13C-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2022-10-21 13:35:52
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.85 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 234
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 23.6 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 54
Ph
OMe
O
O
O
O
O
3b
PPM

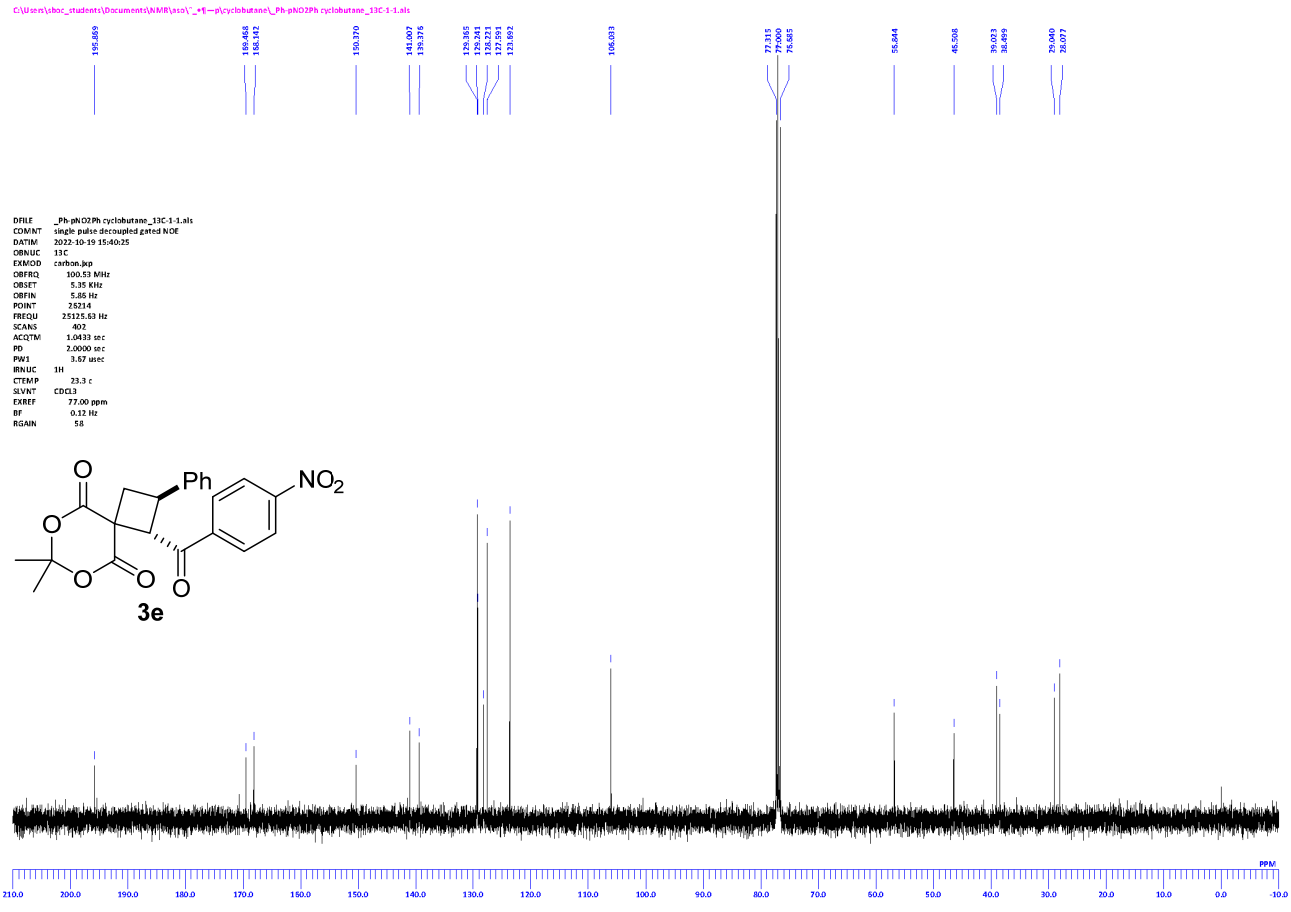

D:/KT2601-1/10/pdata/1/1r
Ph
OMe
O
O
O
O
O
3c
f1 (ppm)

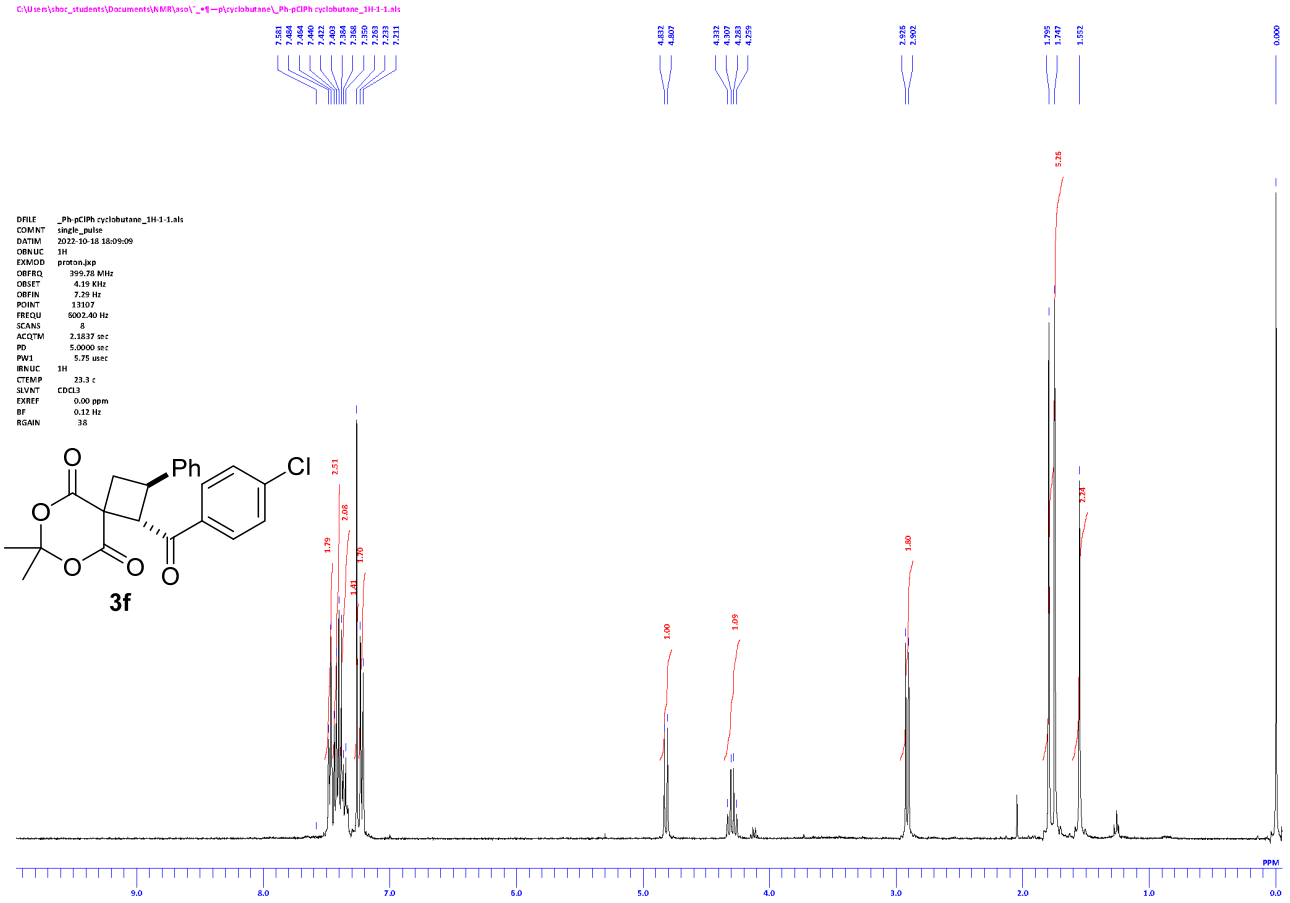

D:/KT2601-1-13C/10/pdata/1/1r
Ph
OMe
O
O
O
O
O
3c
f1 (ppm)

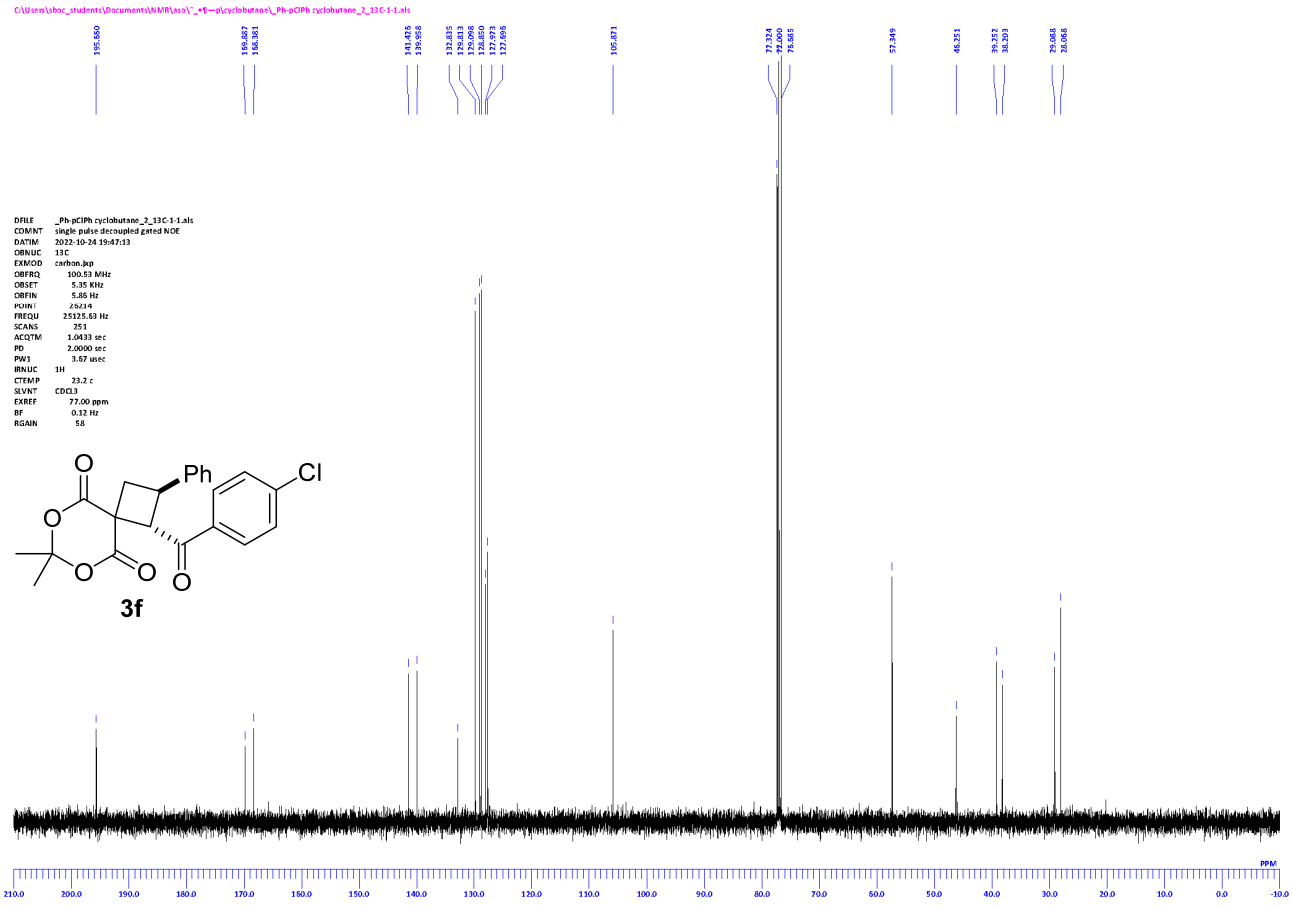

D:/KT2606-1r/10/pdata/1/1r
Ph
O
OMe
3d
f1 (ppm)

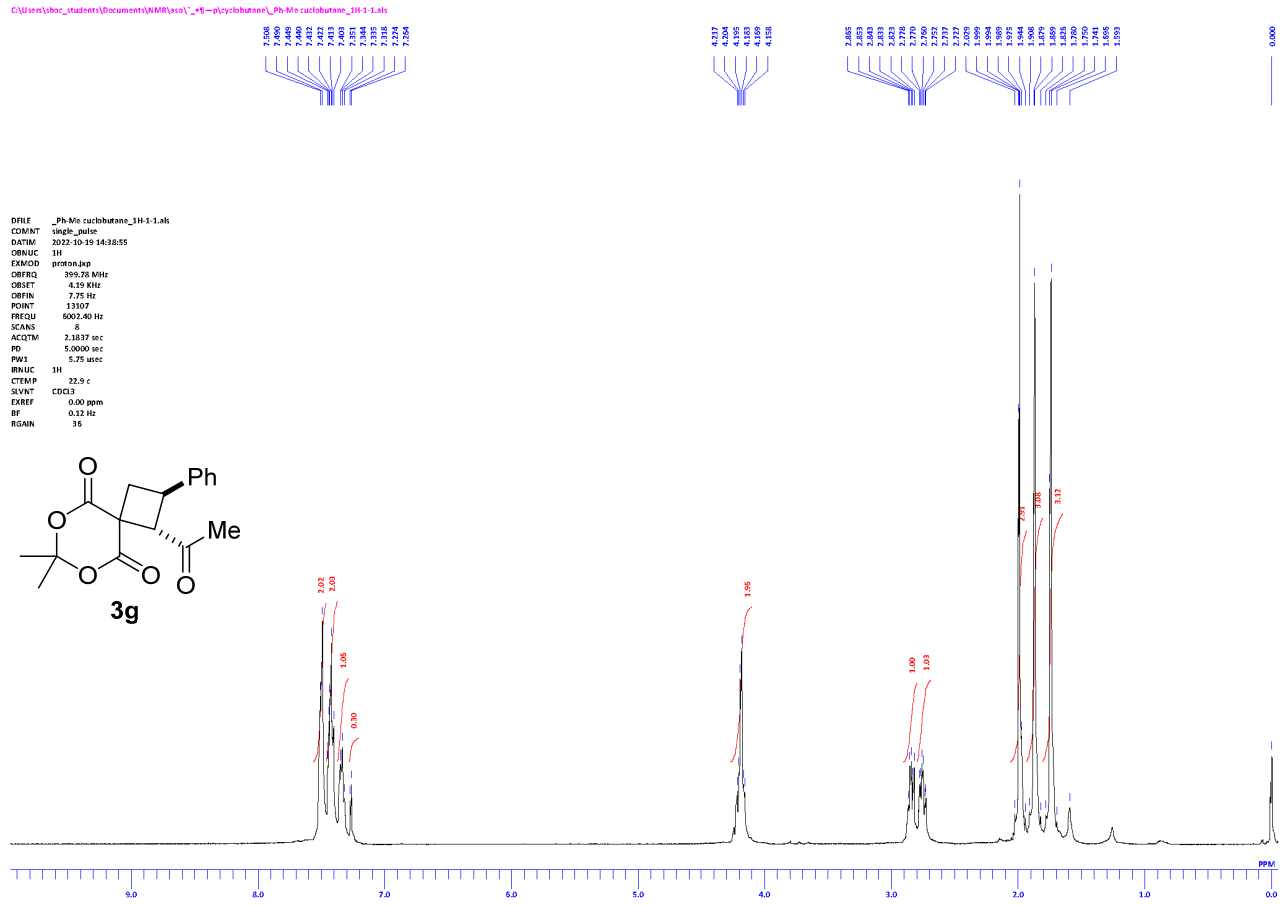

D:/KT2606-1-13C/10/pdata/1/1r
Ph
O
OMe
3d
f1 (ppm)

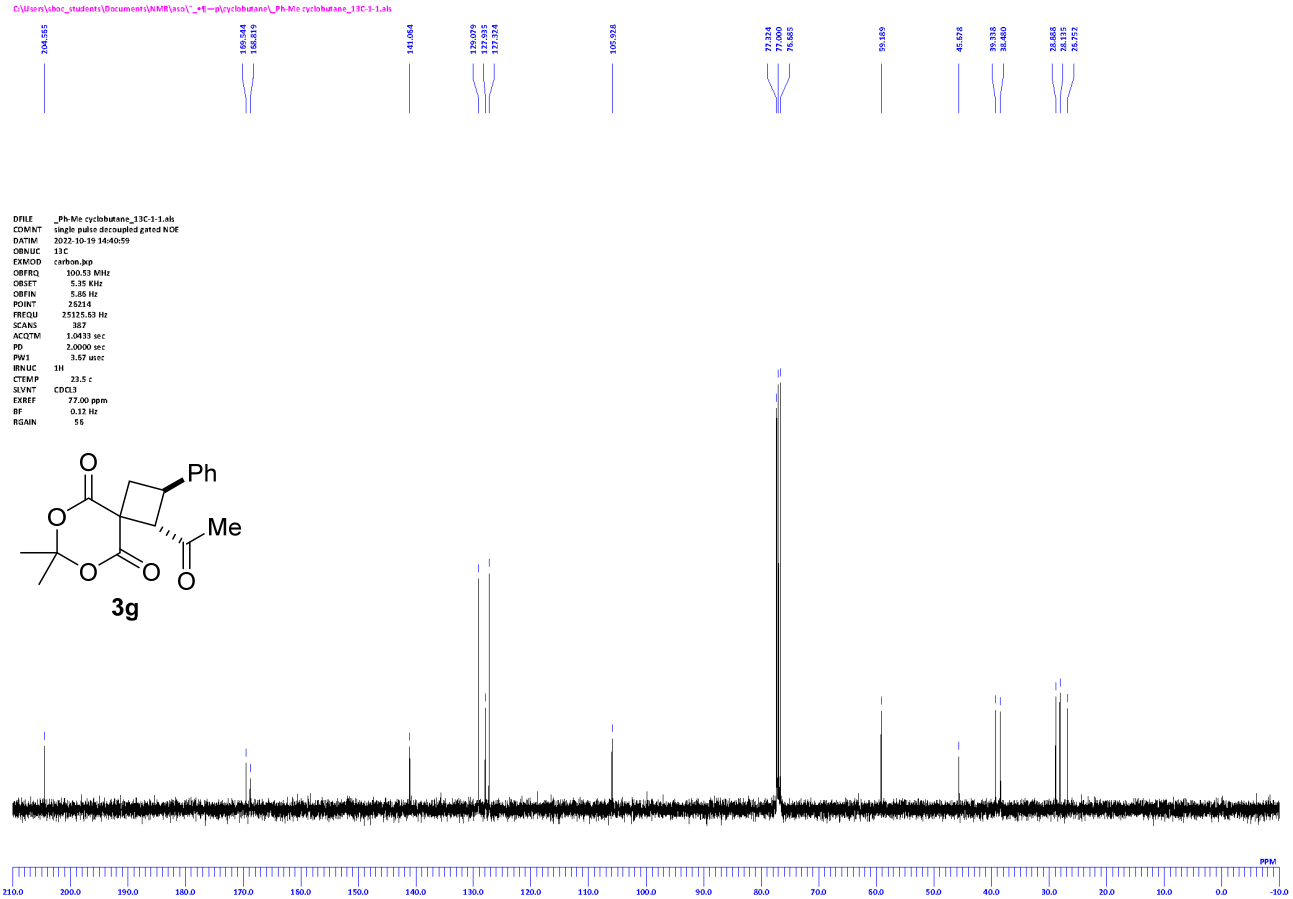

C:\Users\sbcc_students\Documents\NMR\asa\~_•¶—p\cyclobutane_Ph-pNO2Ph cyclobutane_1H-1-1.als
8.112
8.091
7.621
7.599
7.493
7.485
7.468
7.422
7.402
7.377
7.340
7.272
7.265
4.884
4.868
4.330
4.306
4.282
4.258
2.949
2.935
1.801
1.758
1.588
0.000
DFILE _Ph-pNO2Ph cyclobutane_1H-1-1.als
COMNT single_pulse
DATIM 2022-10-19 15:37:43
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.75 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 36
Ph
NO2
O
O
O
O
O
O
3e
2.11
2.08
5.22
1.00
1.04
1.94
5.89
PPM
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

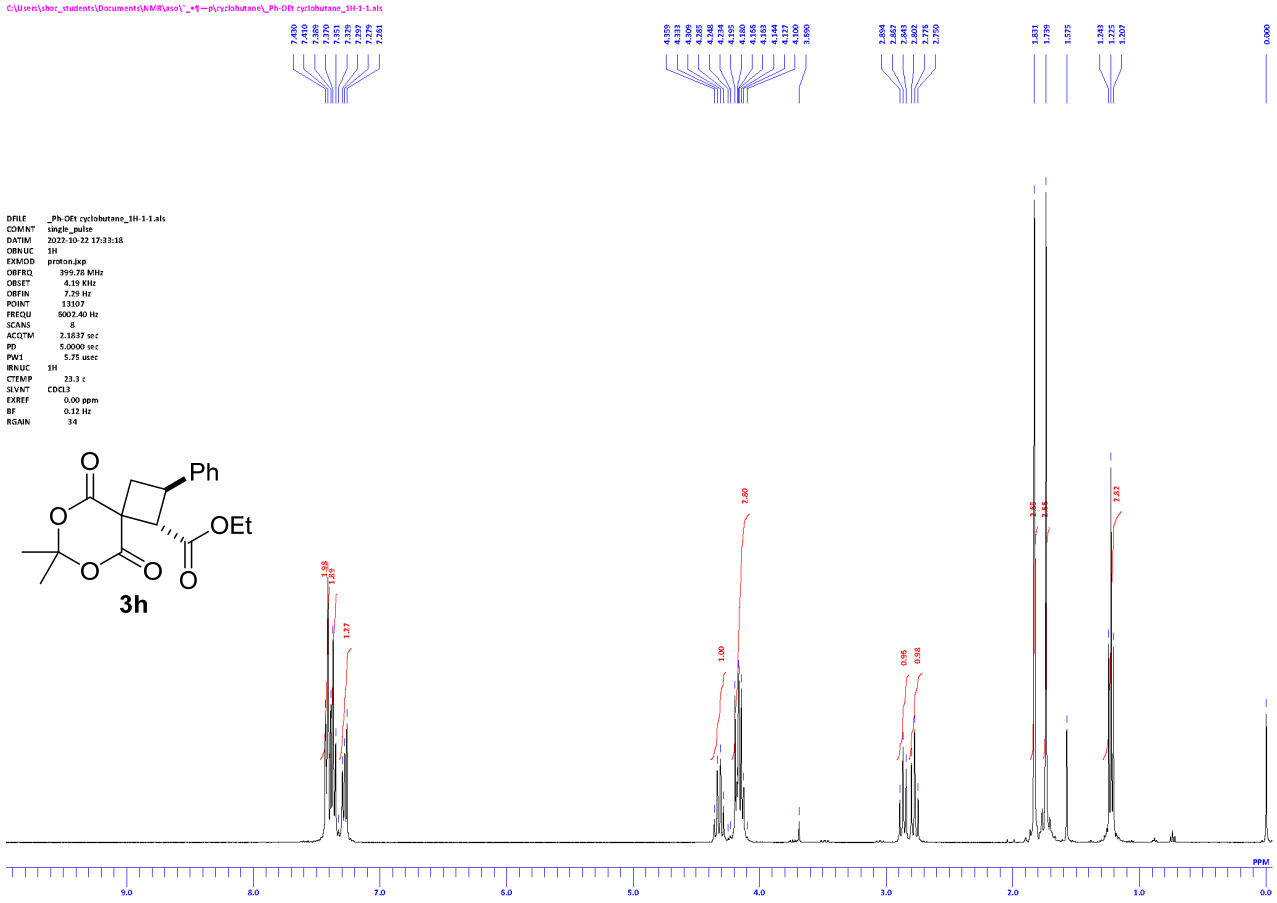

C:\Users\sbcc_students\Documents\NMR\asa\~_•¶—p\cyclobutane_Ph-pNO2Ph cyclobutane_13C-1-1.als
195.889
169.458
168.142
150.370
141.607
139.376
129.365
129.241
128.221
123.761
123.693
106.033
77.315
77.000
75.685
55.844
45.506
39.023
38.489
29.040
28.077
DFILE _Ph-pNO2Ph cyclobutane_13C-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2022-10-19 15:40:25
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 402
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 58
Ph
NO2
O
O
O
O
O
O
3e
PPM
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
-10.0

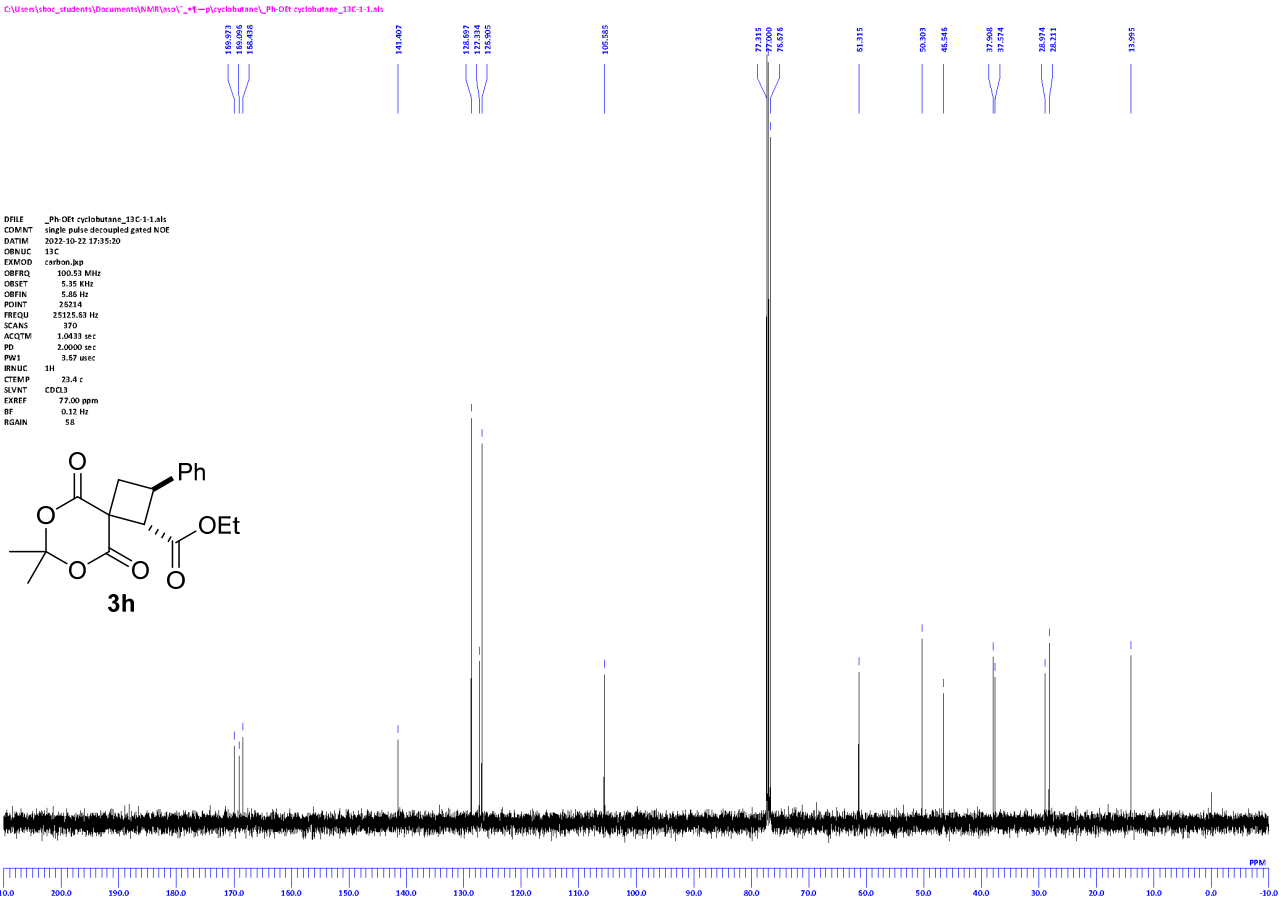

DFILE _Ph-pClPh cyclobutane_1H-1-1.als
COMNT single_pulse
DATIM 2022-10-18 18:09:09
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38
Ph
Cl
O
O
O
O
O
3f
PPM

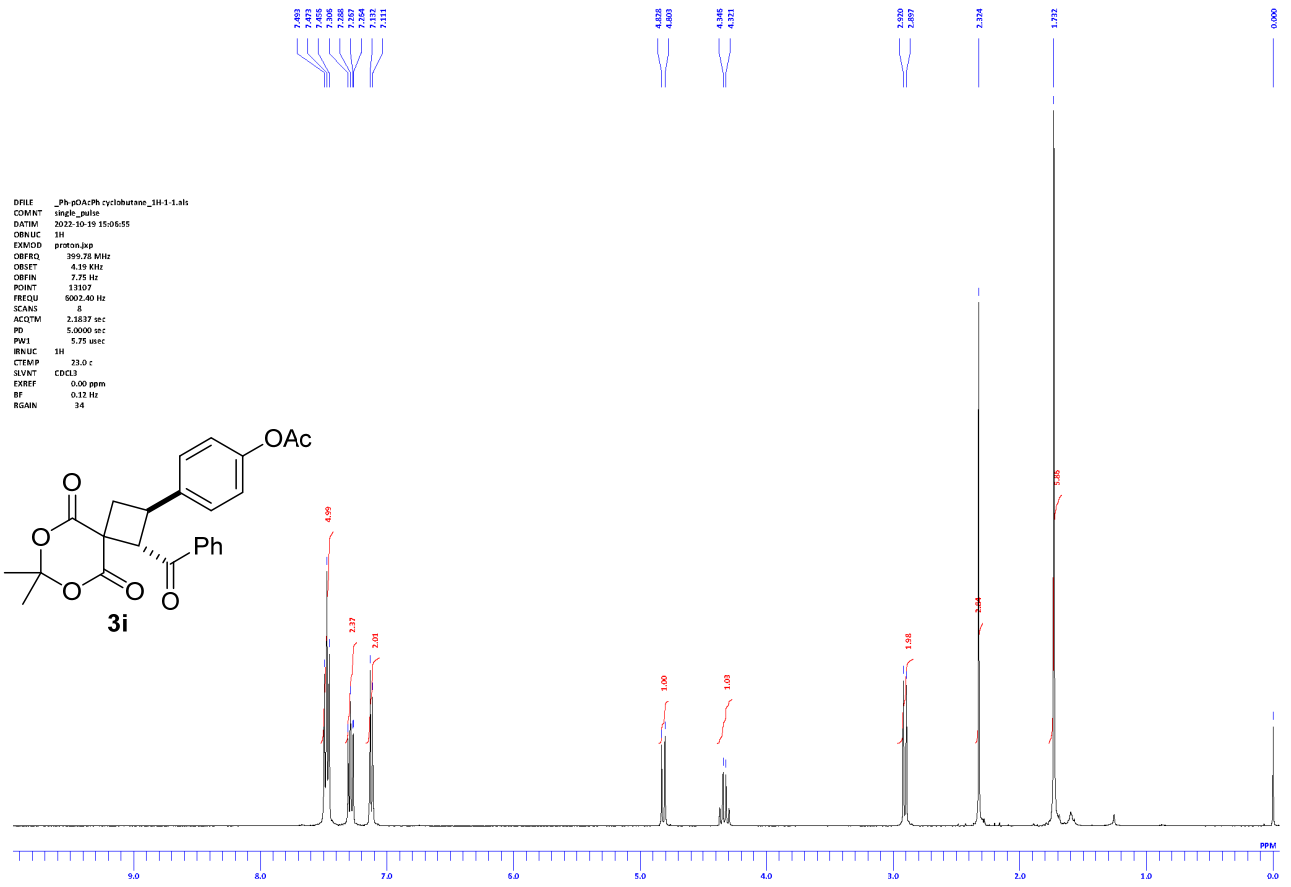

DFILE _Ph-pClPh cyclobutane_2_13C-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2022-10-24 19:47:13
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 251
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 23.2 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 58
Ph
Cl
O
O
O
O
O
3f
PPM

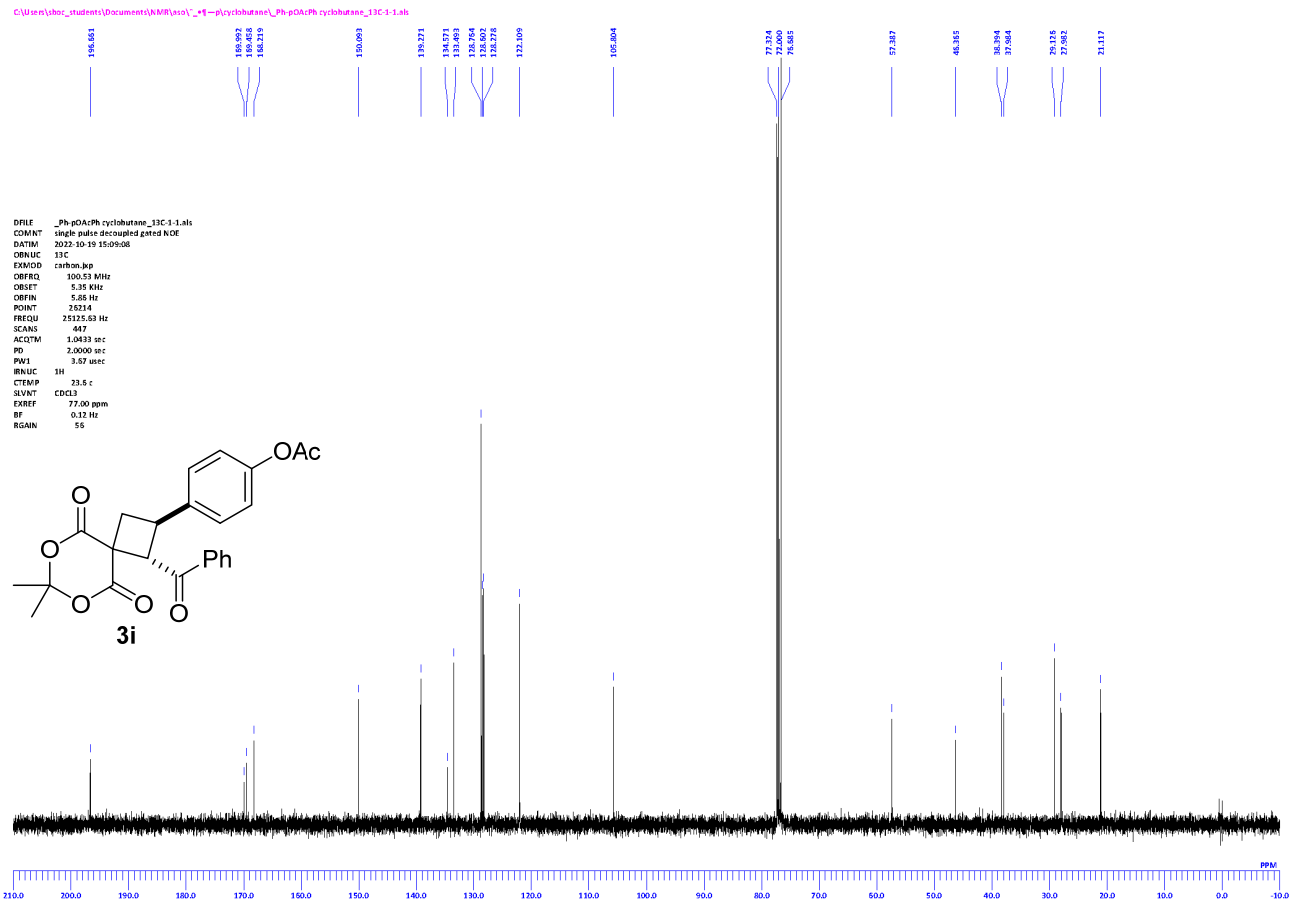

DFILE _Ph-Me cyclobutane_1H-1-1.als
COMNT single_pulse
DATIM 2022-10-19 14:38:55
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.75 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 22.9 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 36
Ph
Me
O
O
O
O
O
3g
PPM

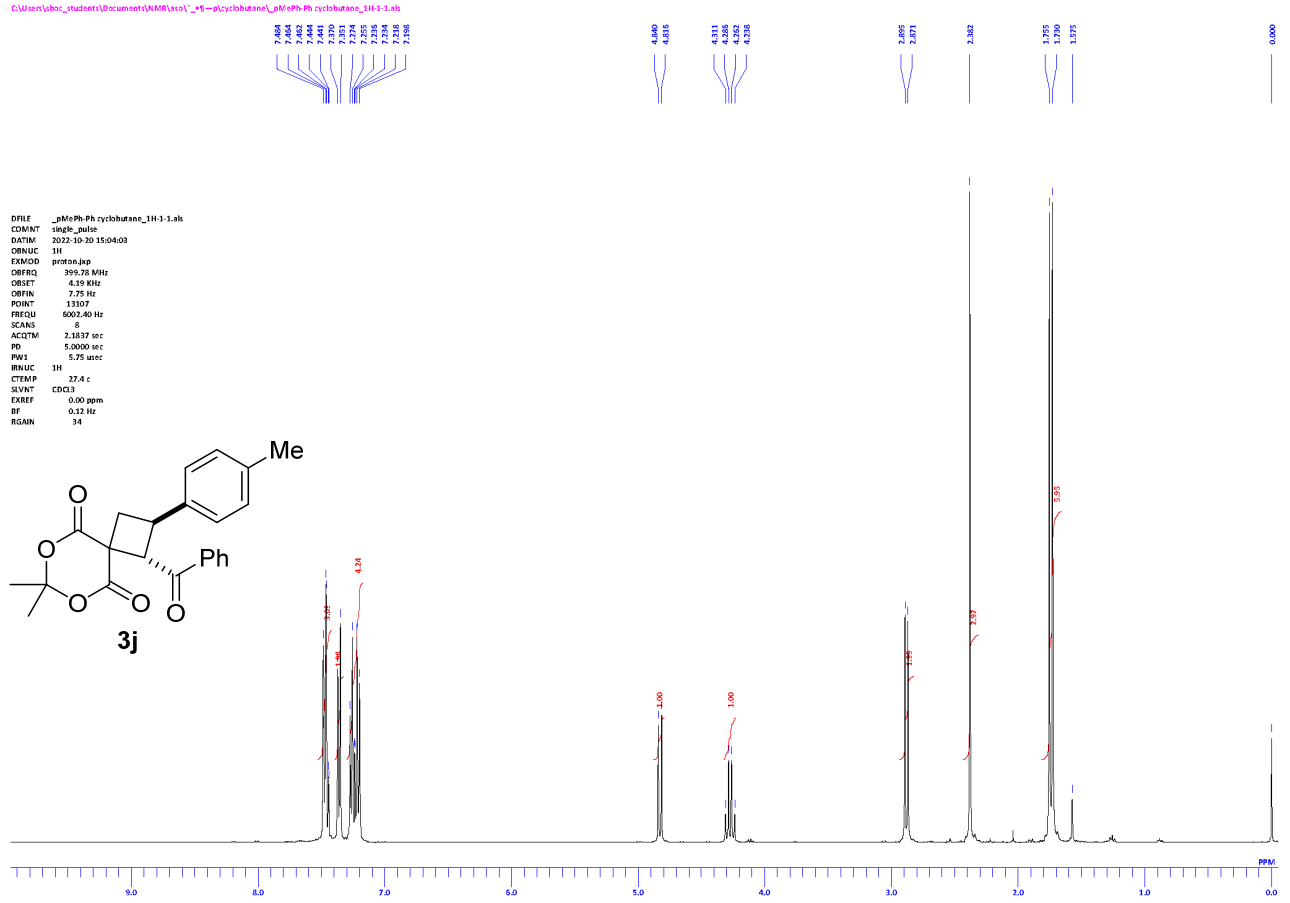

DFILE _Ph-Me cyclobutane_13C-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2022-10-19 14:40:59
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 367
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 23.5 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 56
Ph
Me
O
O
O
O
O
3g
PPM

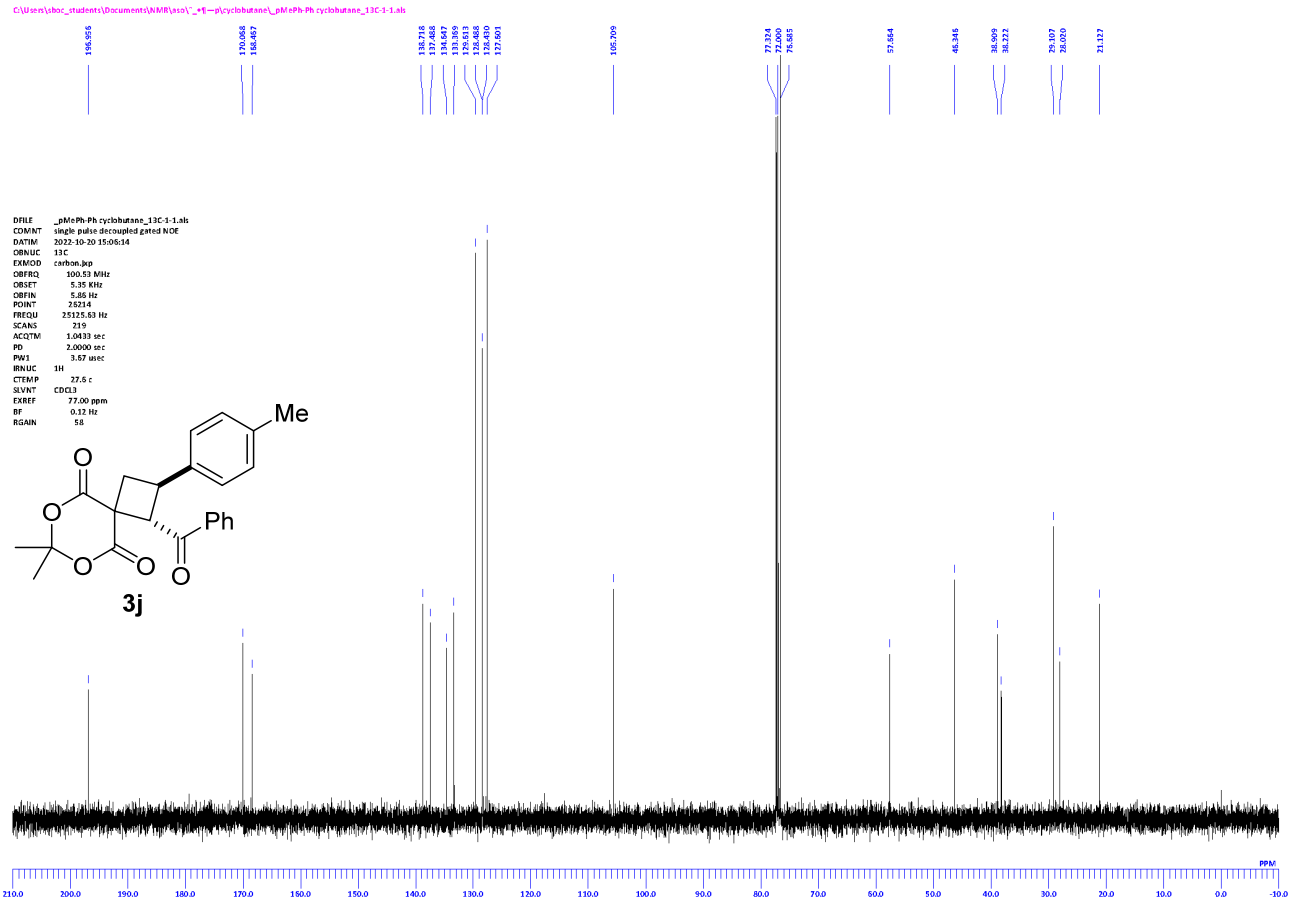

C:\Users\sboc_students\Documents\NMR\sso_•¶—p\cyclobutane_Ph-OEt cyclobutane_1H-1-1.als
DFILE _Ph-OEt cyclobutane_1H-1-1.als
COMNT single_pulse
DATIM 2022-10-22 17:33:18
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 5002.40 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34
Ph
OEt
3h
PPM

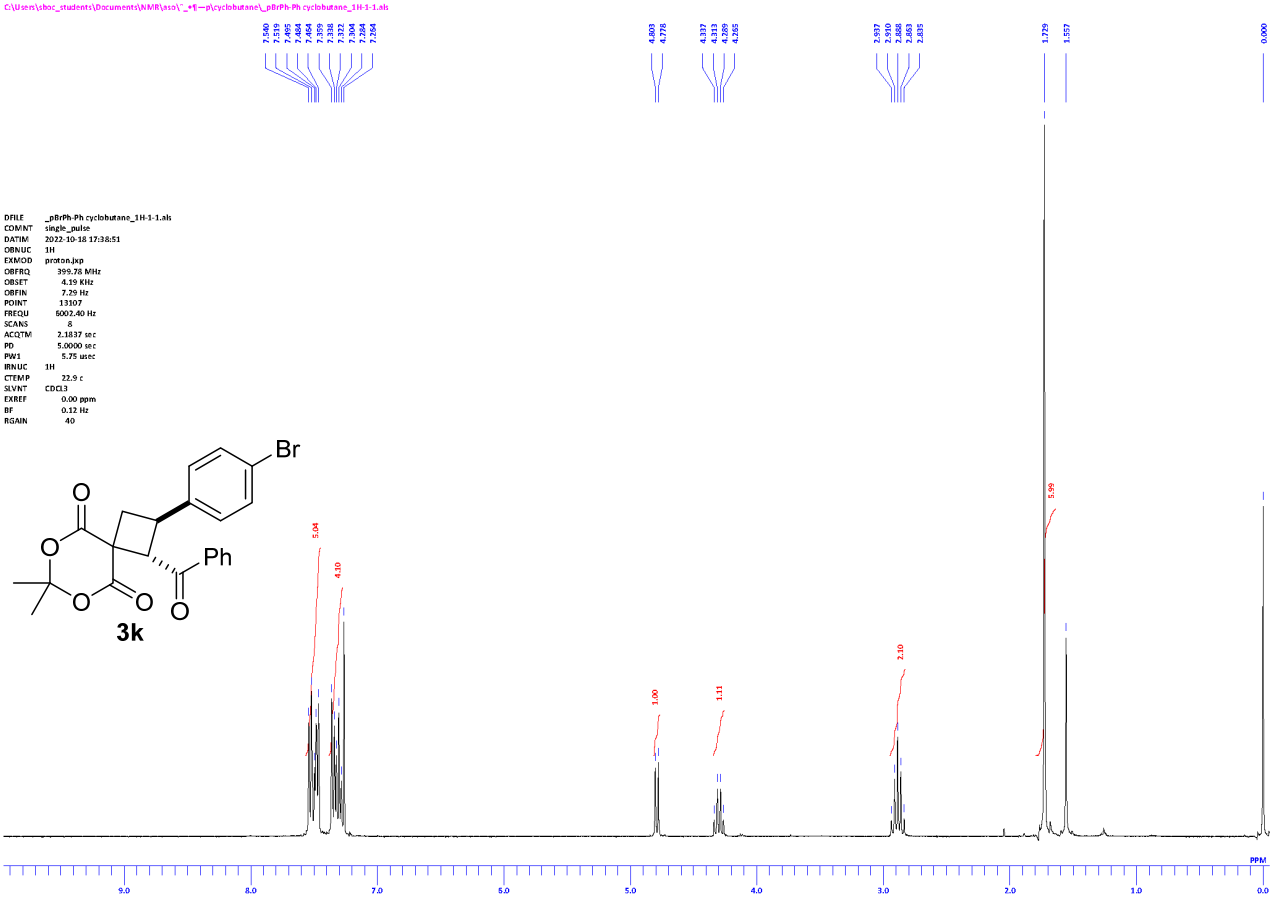

C:\Users\sboc_students\Documents\NMR\sso_•¶—p\cyclobutane_Ph-OEt cyclobutane_13C-1-1.als
DFILE _Ph-OEt cyclobutane_13C-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2022-10-22 17:35:20
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 25214
FREQU 25125.63 Hz
SCANS 370
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.57 usec
IRNUC 1H
CTEMP 23.4 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 58
Ph
OEt
3h
PPM

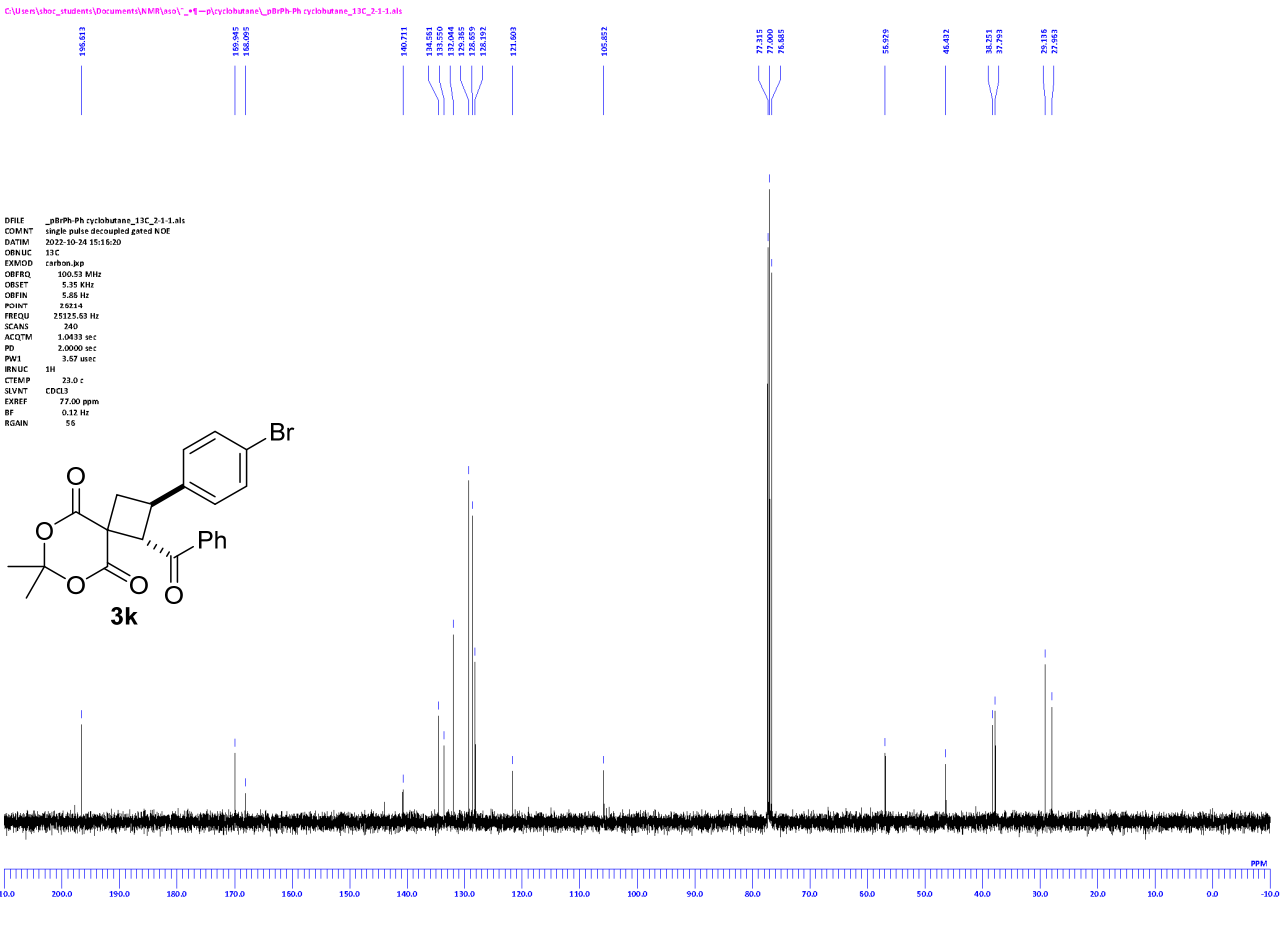

C:\Users\sbcr_students\Documents\NMR\aso\~_*¶—p\cyclobutane_Ph-pOAcPh cyclobutane_1H-1-1.als
7.493
7.473
7.456
7.306
7.289
7.283
7.264
7.132
7.111
4.828
4.803
4.365
4.321
2.920
2.897
2.324
1.732
0.000
DFILE _Ph-pOAcPh cyclobutane_1H-1-1.als
COMNT single_pulse
DATIM 2022-10-19 15:06:55
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.75 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 23.0 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34
OAc
Ph
3i
4.99
2.37
2.01
1.00
1.03
1.98
2.94
5.85
PPM
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

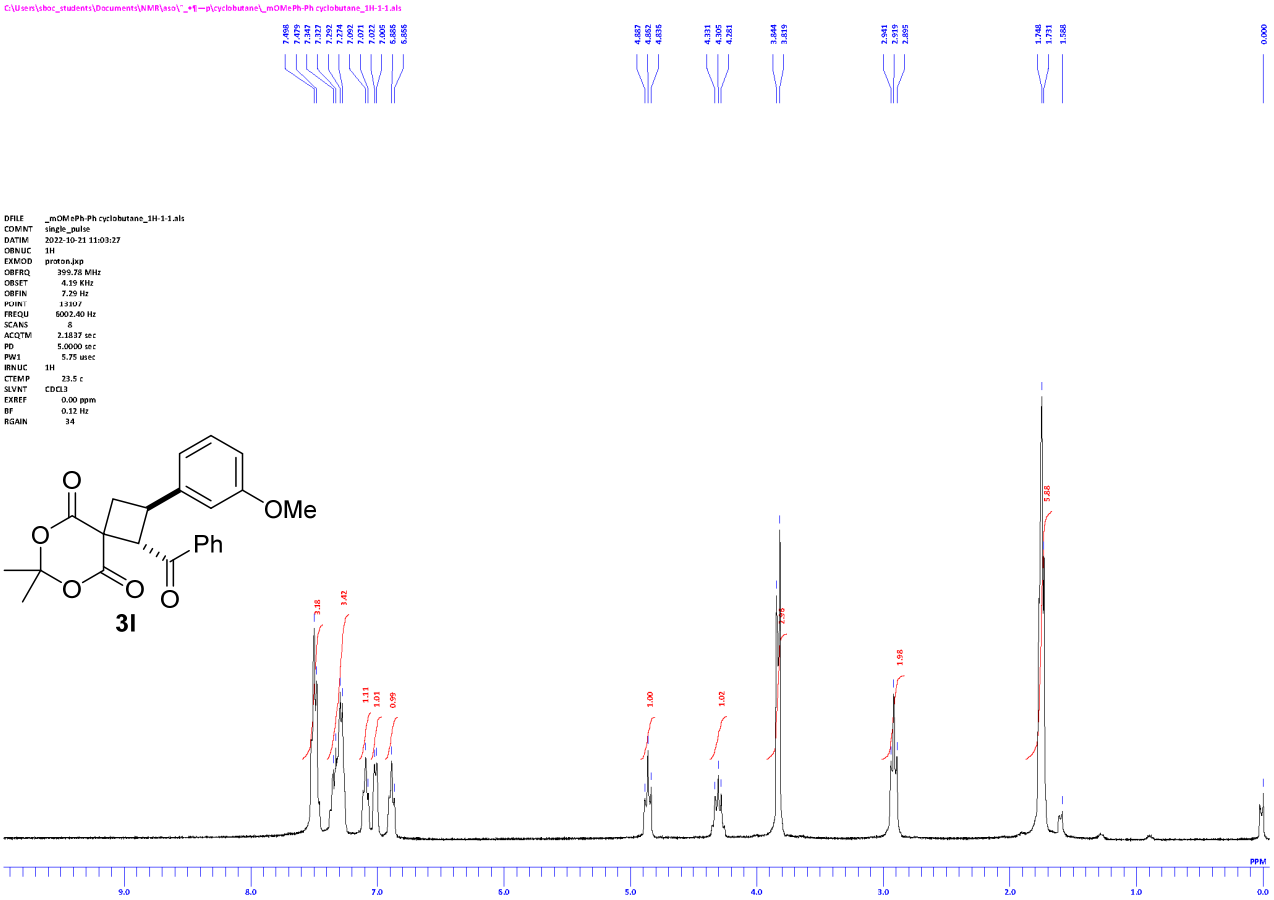

C:\Users\sbcr_students\Documents\NMR\aso\~_*¶—p\cyclobutane_Ph-pOAcPh cyclobutane_13C-1-1.als
156.651
169.992
169.458
168.219
150.093
139.271
134.571
132.464
128.602
128.276
122.109
105.804
77.324
77.000
76.683
57.387
46.355
38.394
37.984
29.125
27.982
21.117
DFILE _Ph-pOAcPh cyclobutane_13C-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2022-10-19 15:09:08
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 447
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 23.5 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 56
OAc
Ph
3i
PPM
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
-10.0

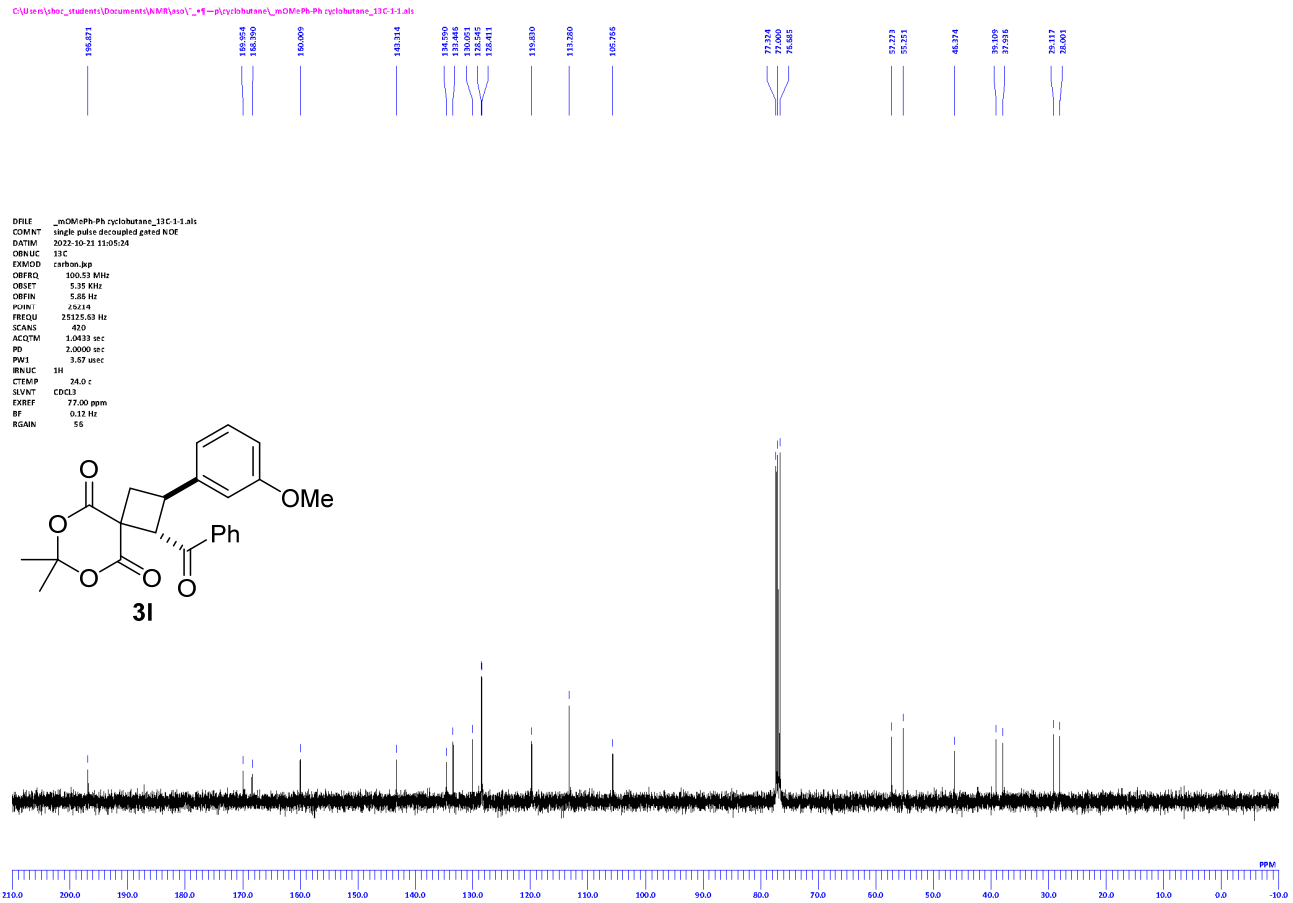

Me
Ph
O
O
O
O
O
3j
PPM

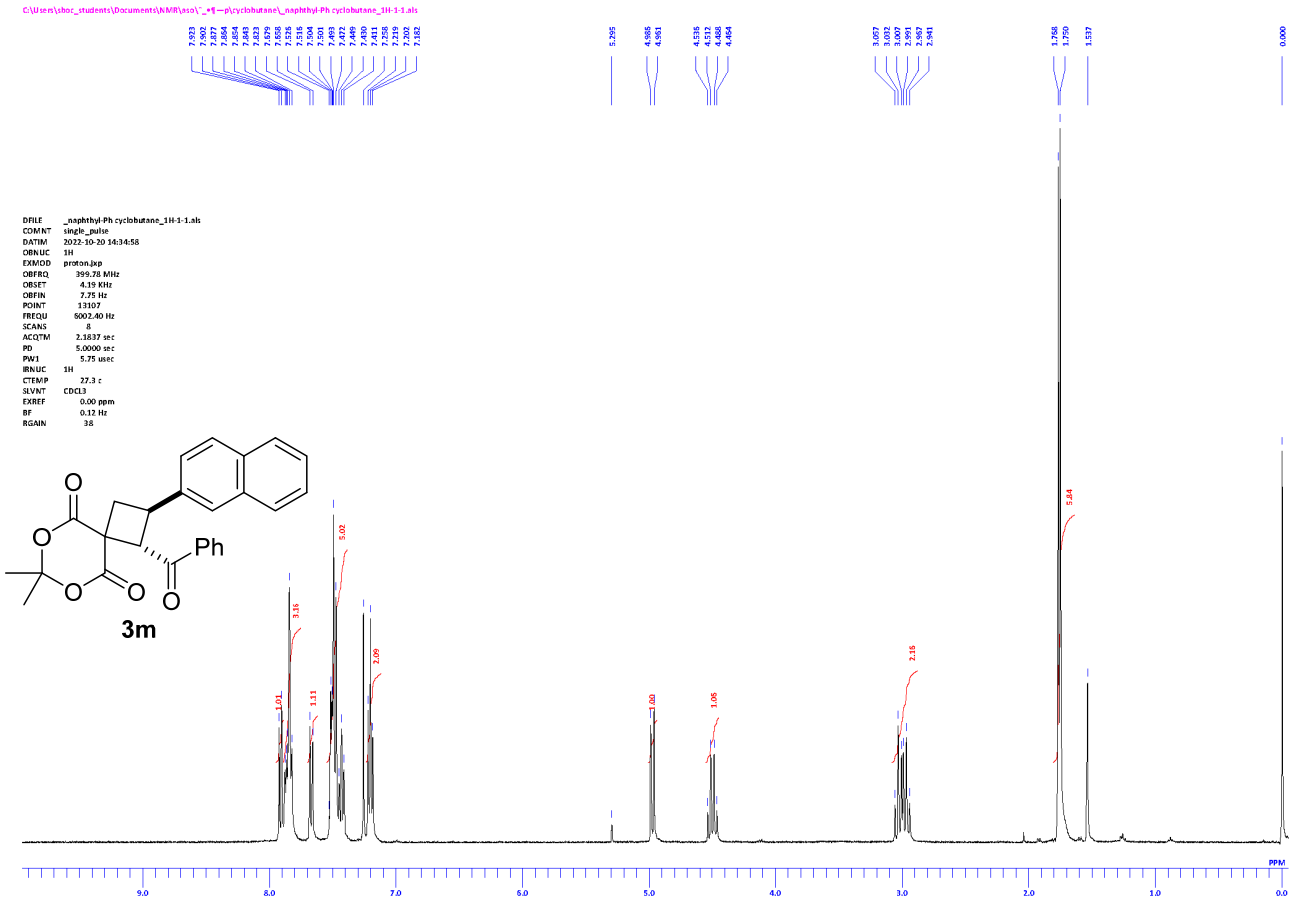

Me
Ph
O
O
O
O
O
3j
PPM

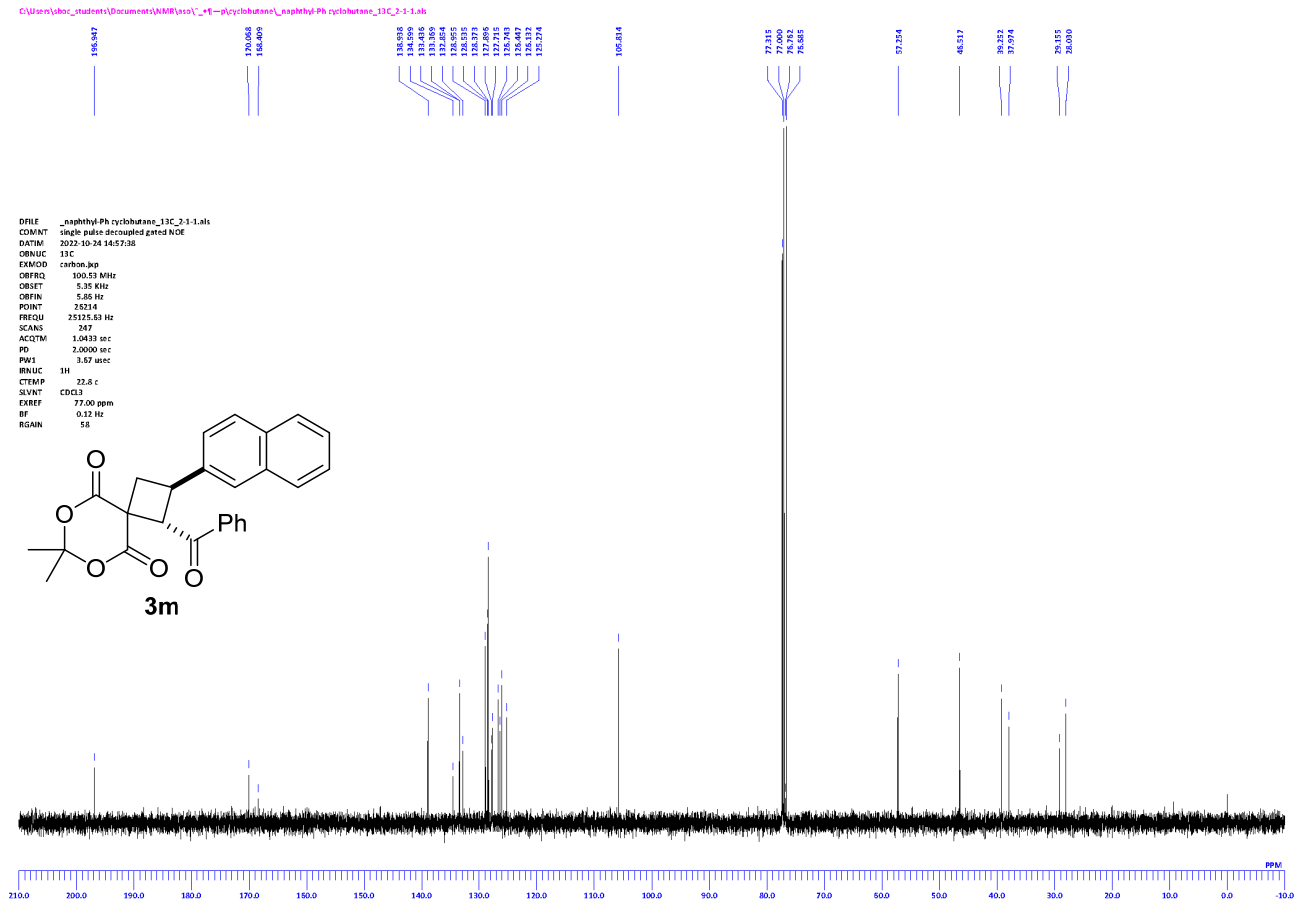

C:\Users\sbcc_students\Documents\NMR\aso\~_*¶—p\cyclobutane_pBrPh-Ph cyclobutane_1H-1-1.als
7.540
7.516
7.495
7.484
7.464
7.359
7.336
7.322
7.304
7.284
7.264
4.803
4.778
4.337
4.313
4.295
4.285
3.937
3.910
3.886
3.863
3.835
1.729
1.557
0.000
DFILE _pBrPh-Ph cyclobutane_1H-1-1.als
COMNT single_pulse
DATIM 2022-10-18 17:38:51
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 22.9 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40
Br
O
O
O
O
O
Ph
O
3k
5.04
4.10
1.00
1.11
2.10
5.99
PPM
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

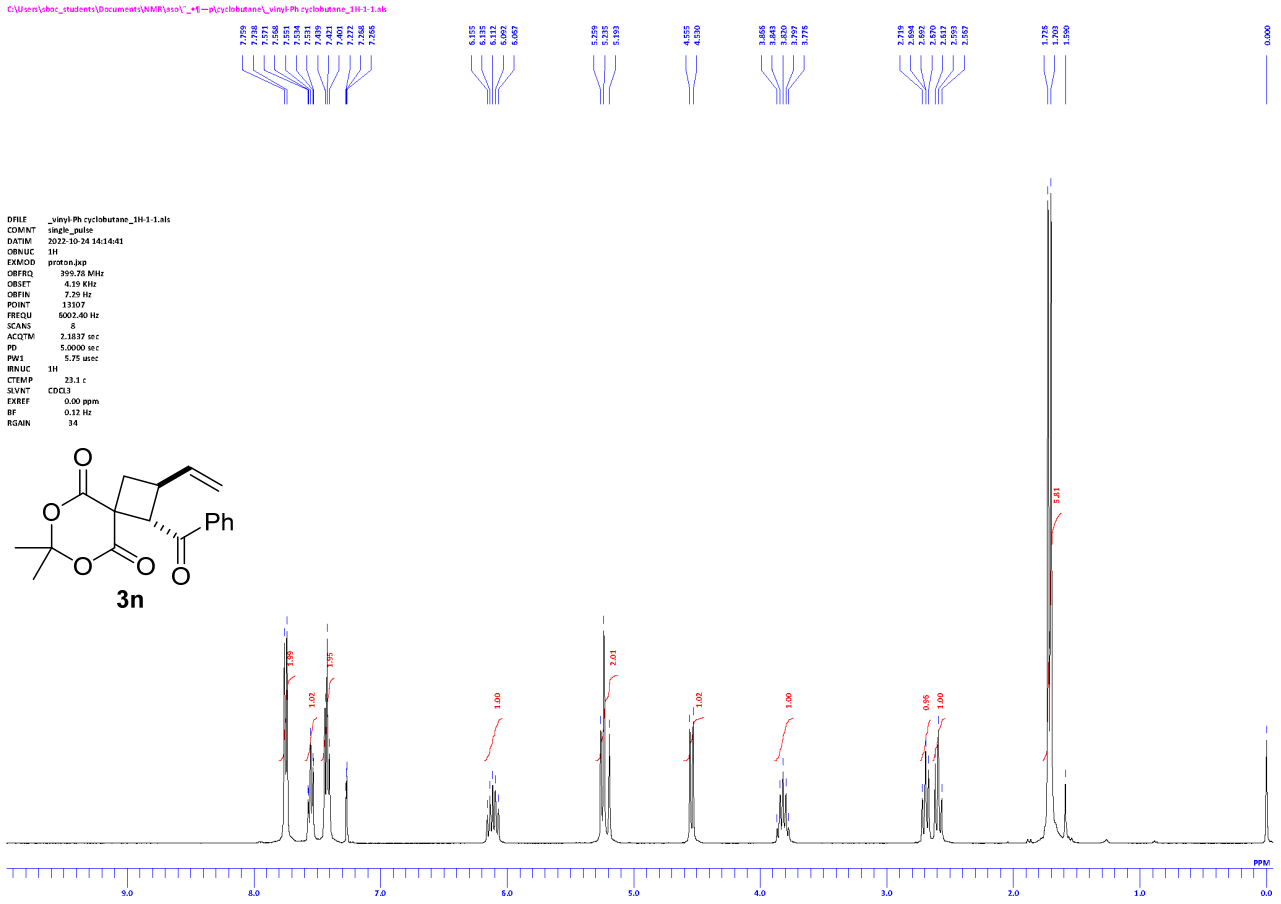

C:\Users\sbcc_students\Documents\NMR\aso\~_*¶—p\cyclobutane_pBrPh-Ph cyclobutane_13C_2-1-1.als
196.513
169.945
169.095
140.711
134.561
133.550
132.044
129.485
128.909
128.192
121.603
105.852
77.315
77.000
76.685
56.929
46.432
38.251
37.793
29.136
27.963
DFILE _pBrPh-Ph cyclobutane_13C_2-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2022-10-24 15:16:20
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 240
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 23.0 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 56
Br
O
O
O
O
O
Ph
O
3k
PPM
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
-10.0

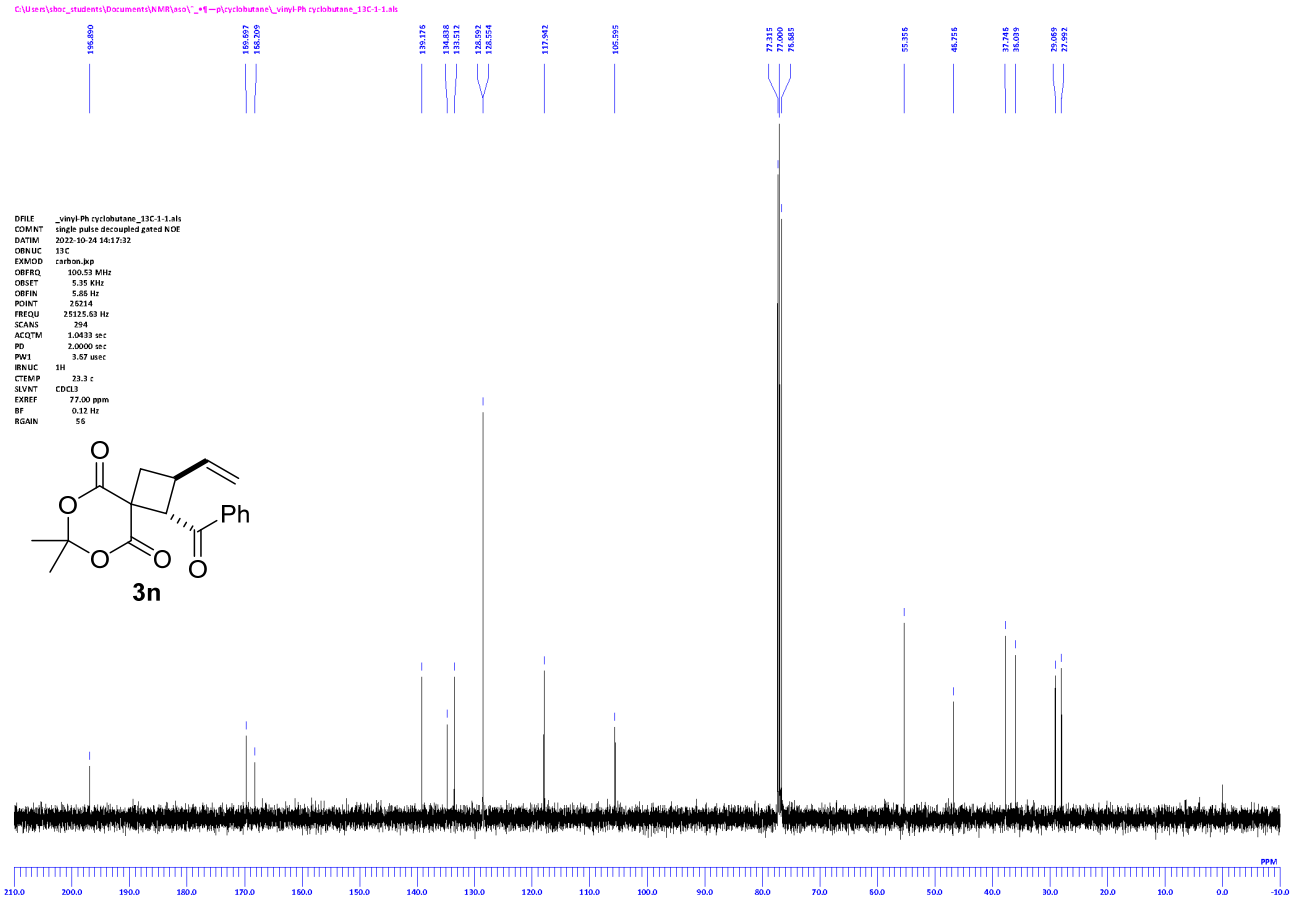

OMe
Ph
O
O
O
O
O
3l
PPM

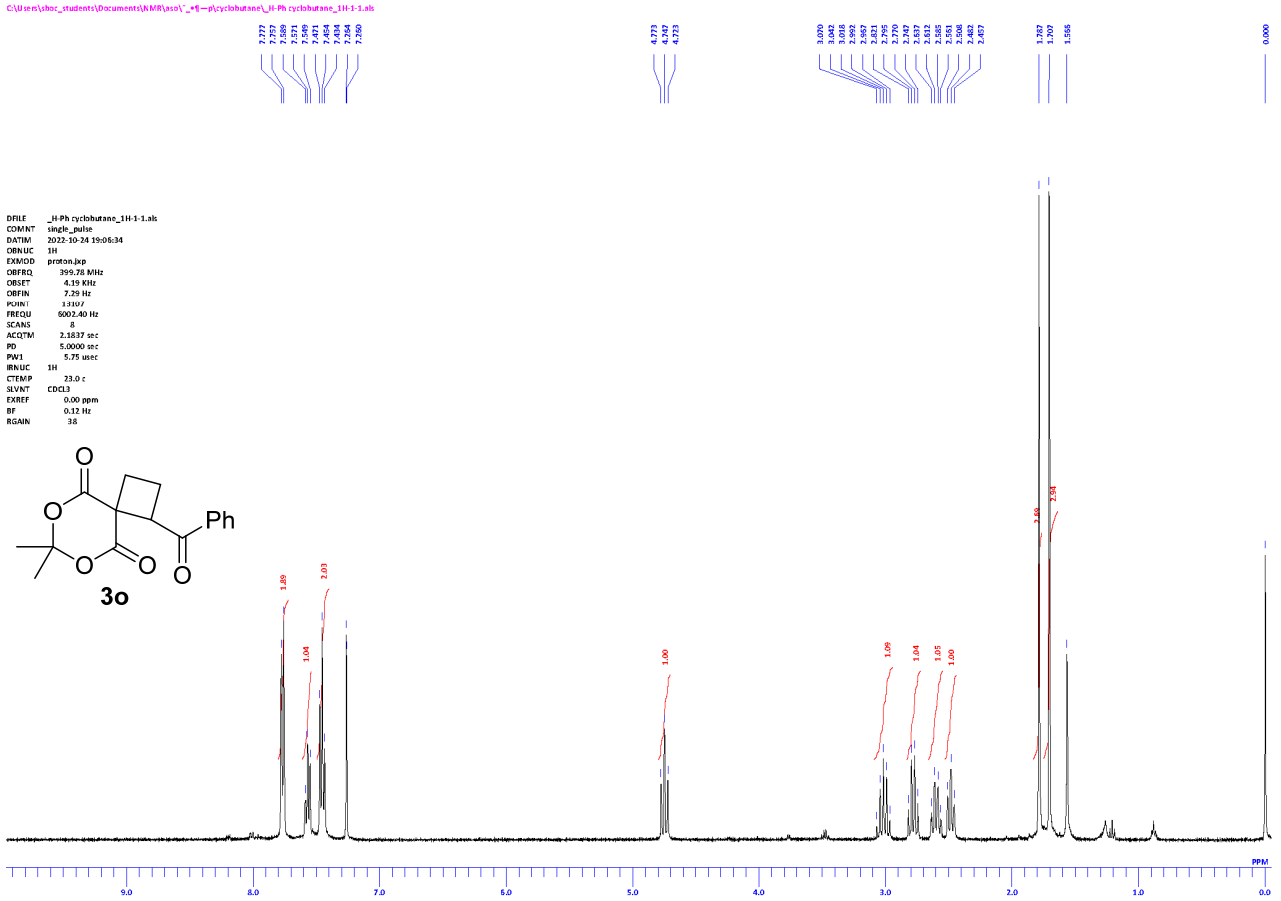

OMe
Ph
O
O
O
O
O
3l
PPM

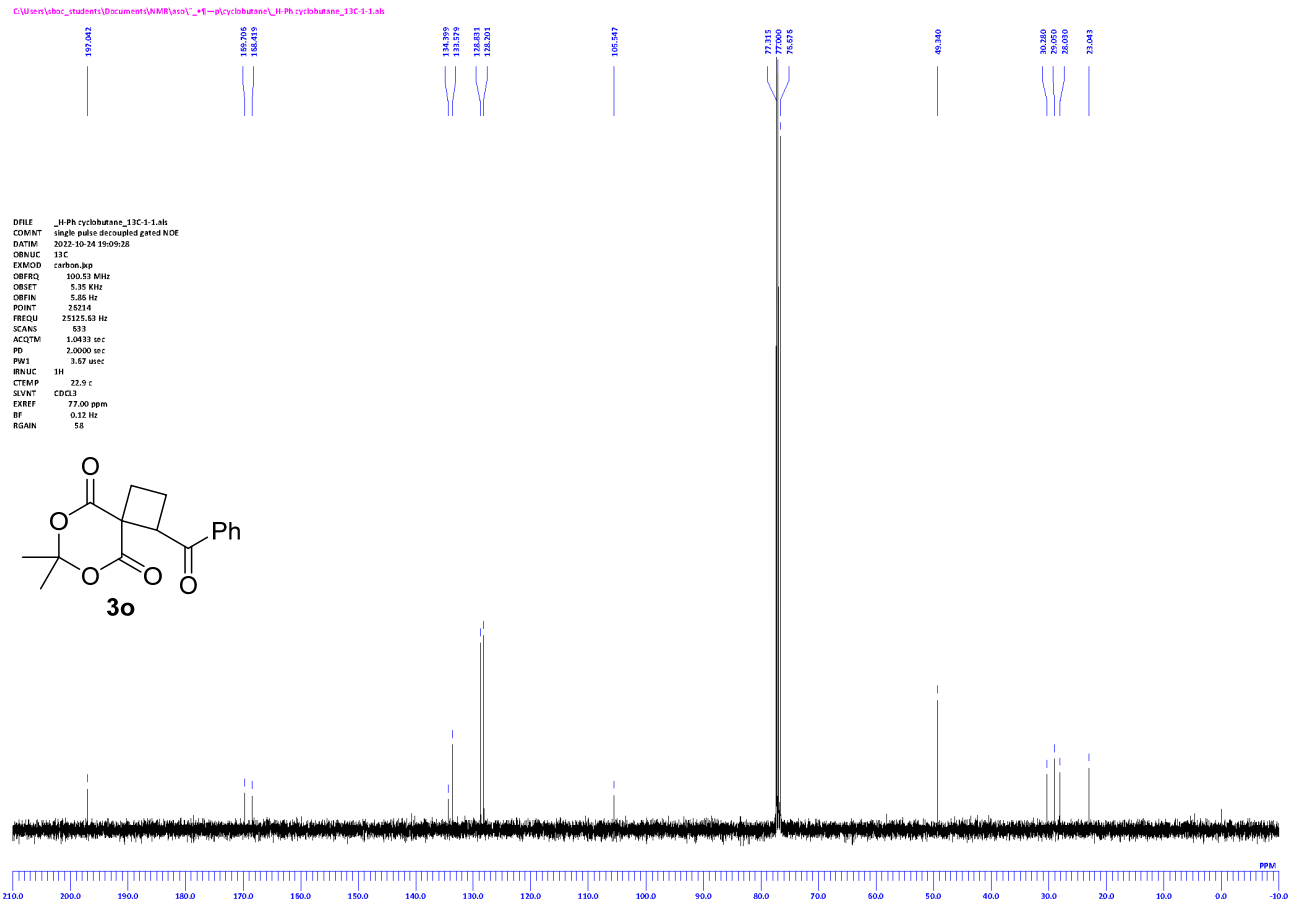

C:\Users\shoc_students\Documents\NMR\sso\'_*¶—p\cyclobutane_naphthyl-Ph cyclobutane_1H-1-1.als
DFILE _naphthyl-Ph cyclobutane_1H-1-1.als
COMNT single_pulse
DATIM 2022-10-20 14:34:58
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.75 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 27.3 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38
Ph
3m
PPM

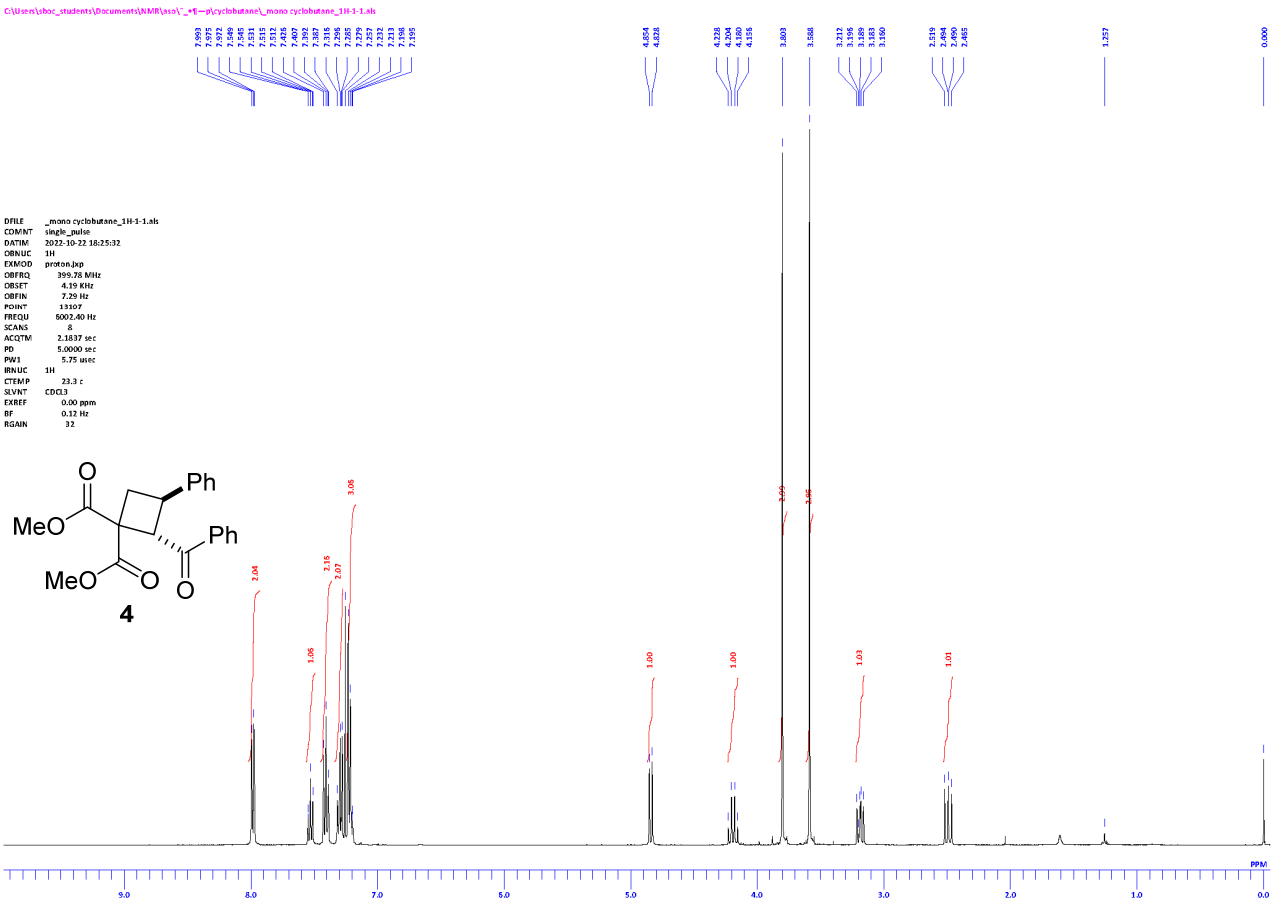

C:\Users\shoc_students\Documents\NMR\sso\'_*¶—p\cyclobutane_naphthyl-Ph cyclobutane_13C_2-1-1.als
DFILE _naphthyl-Ph cyclobutane_13C_2-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2022-10-24 14:57:38
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 247
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 22.8 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 58
Ph
3m
PPM

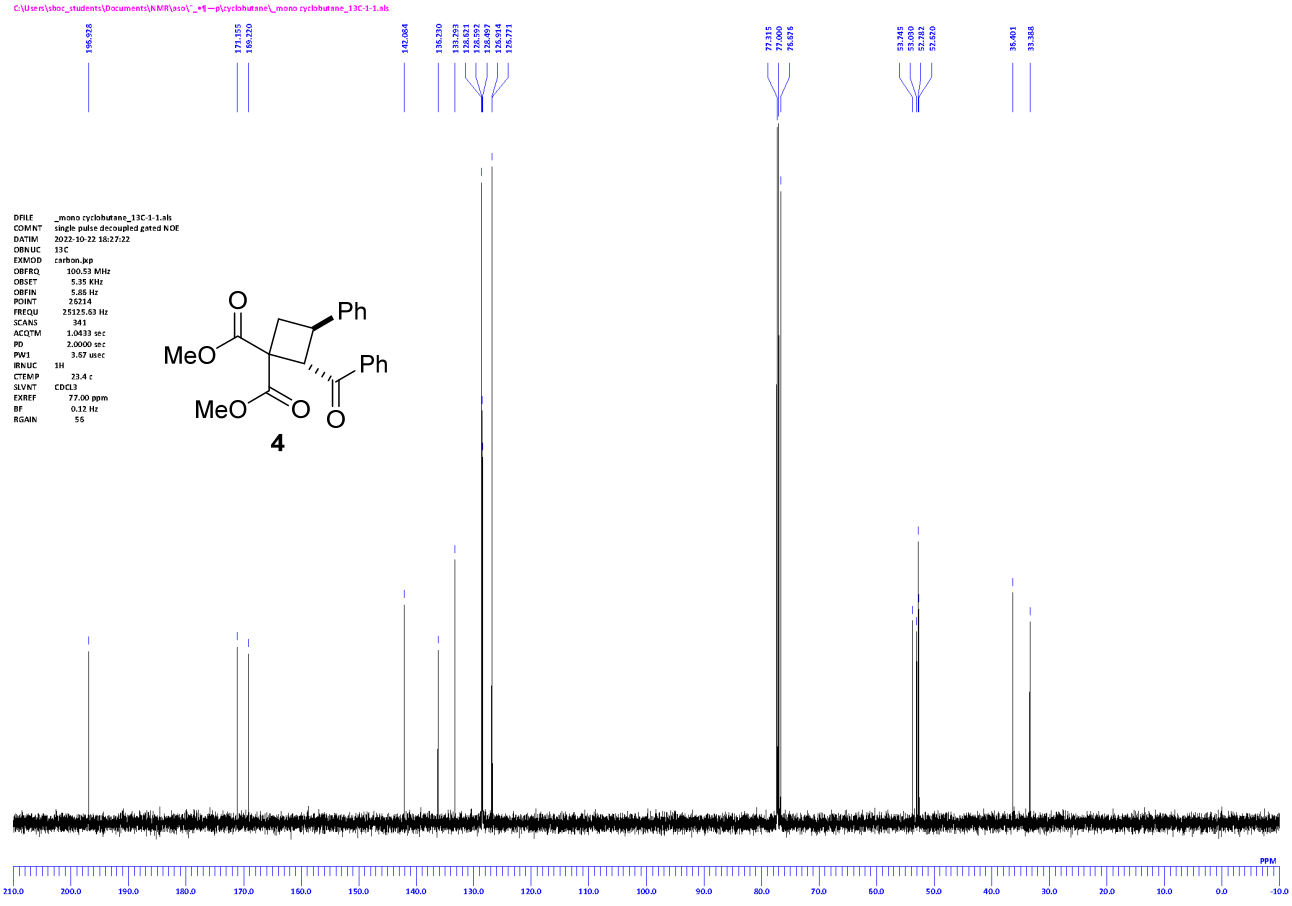

C:\Users\shoc_students\Documents\NMR\sso\'_*¶—p\cyclobutane_vinyl-Ph cyclobutane_1H-1-1.als
DFILE _vinyl-Ph cyclobutane_1H-1-1.als
COMNT single_pulse
DATIM 2022-10-24 14:14:41
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 23.1 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34
Ph
3n
PPM

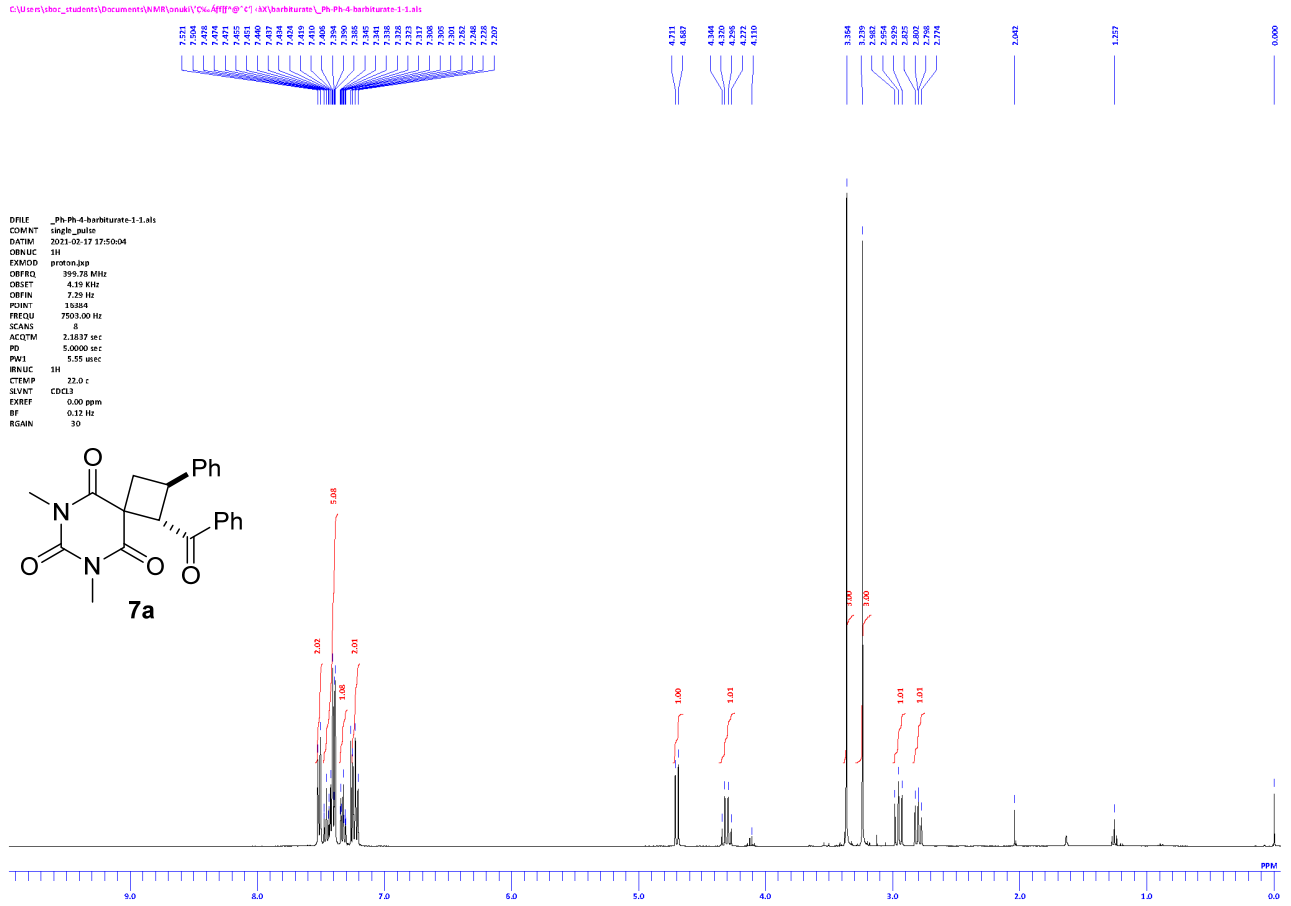

C:\Users\shoc_students\Documents\NMR\sso\'_*¶—p\cyclobutane_vinyl-Ph cyclobutane_13C-1-1.als
DFILE _vinyl-Ph cyclobutane_13C-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2022-10-24 14:17:32
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.85 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 294
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 56
Ph
3n
PPM

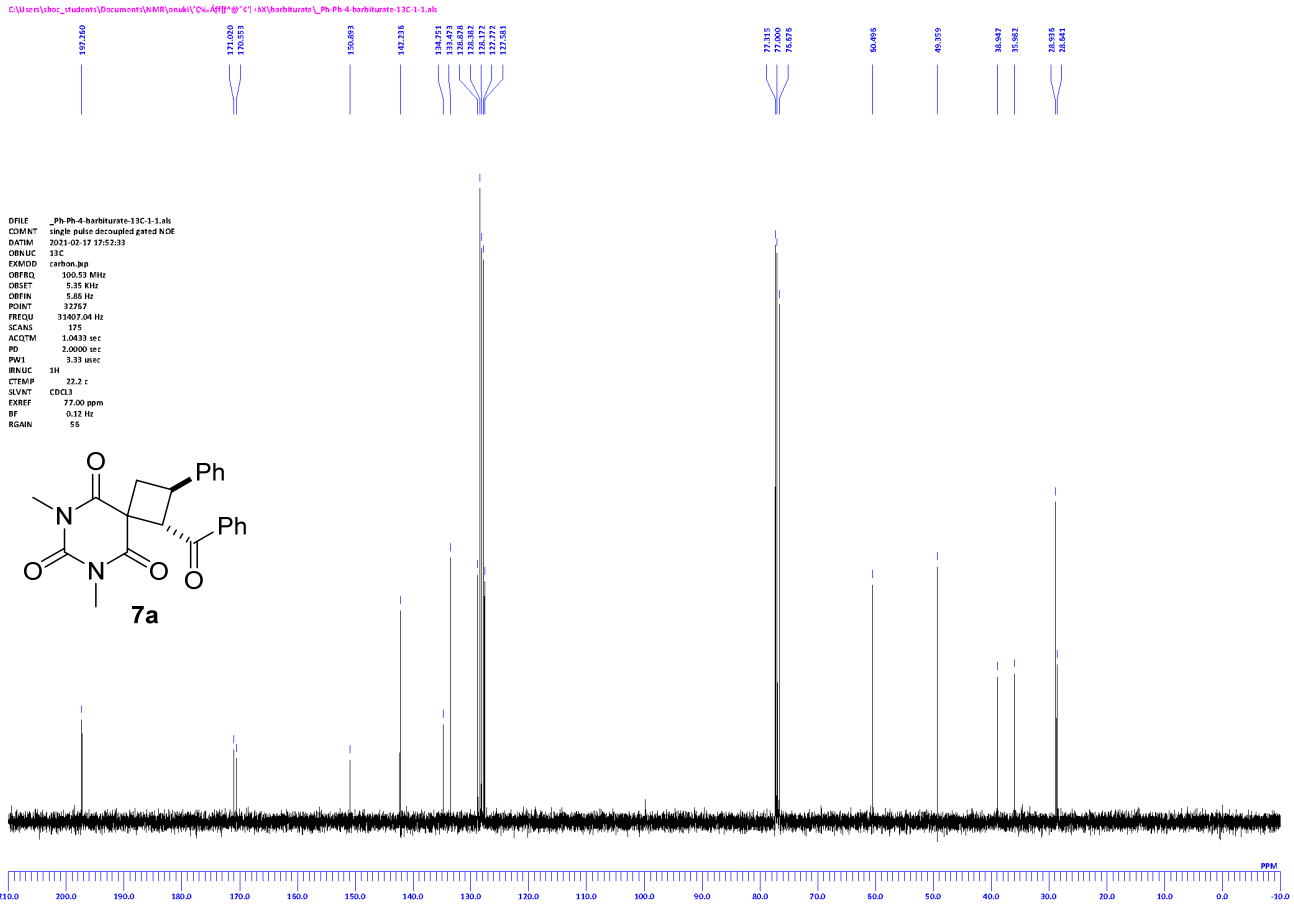

C:\Users\sbac_students\Documents\NMR\aso\~_•¶—p\cyclobutane_H-Ph cyclobutane_1H-1-1.als
DFILE _H-Ph cyclobutane_1H-1-1.als
COMNT single_pulse
DATIM 2022-10-24 19:08:34
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 23.0 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38
Ph
O
3o
PPM

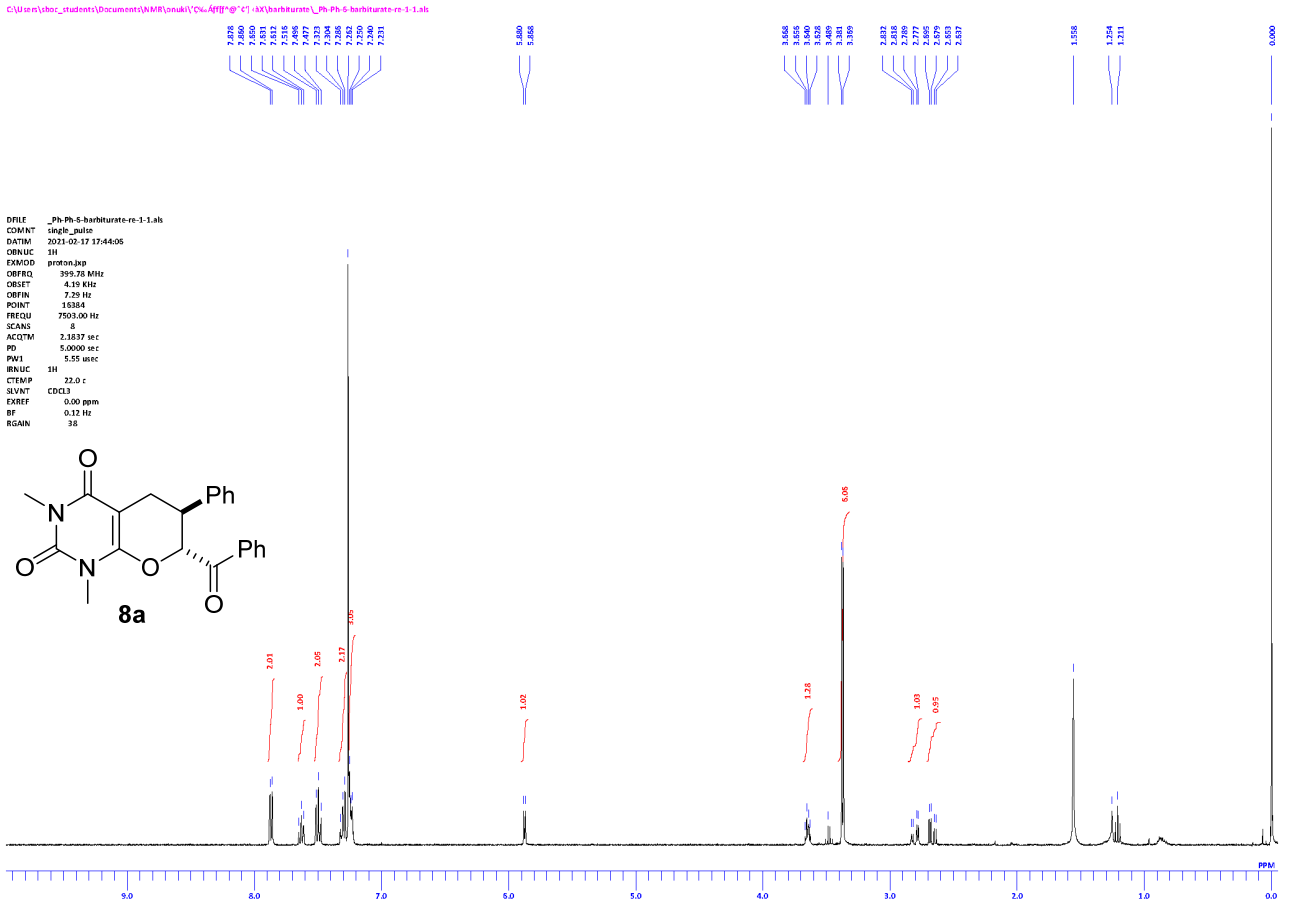

C:\Users\sbac_students\Documents\NMR\aso\~_•¶—p\cyclobutane_H-Ph cyclobutane_13C-1-1.als
DFILE _H-Ph cyclobutane_13C-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2022-10-24 19:09:28
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.85 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 533
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 22.9 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 58
Ph
O
3o
PPM

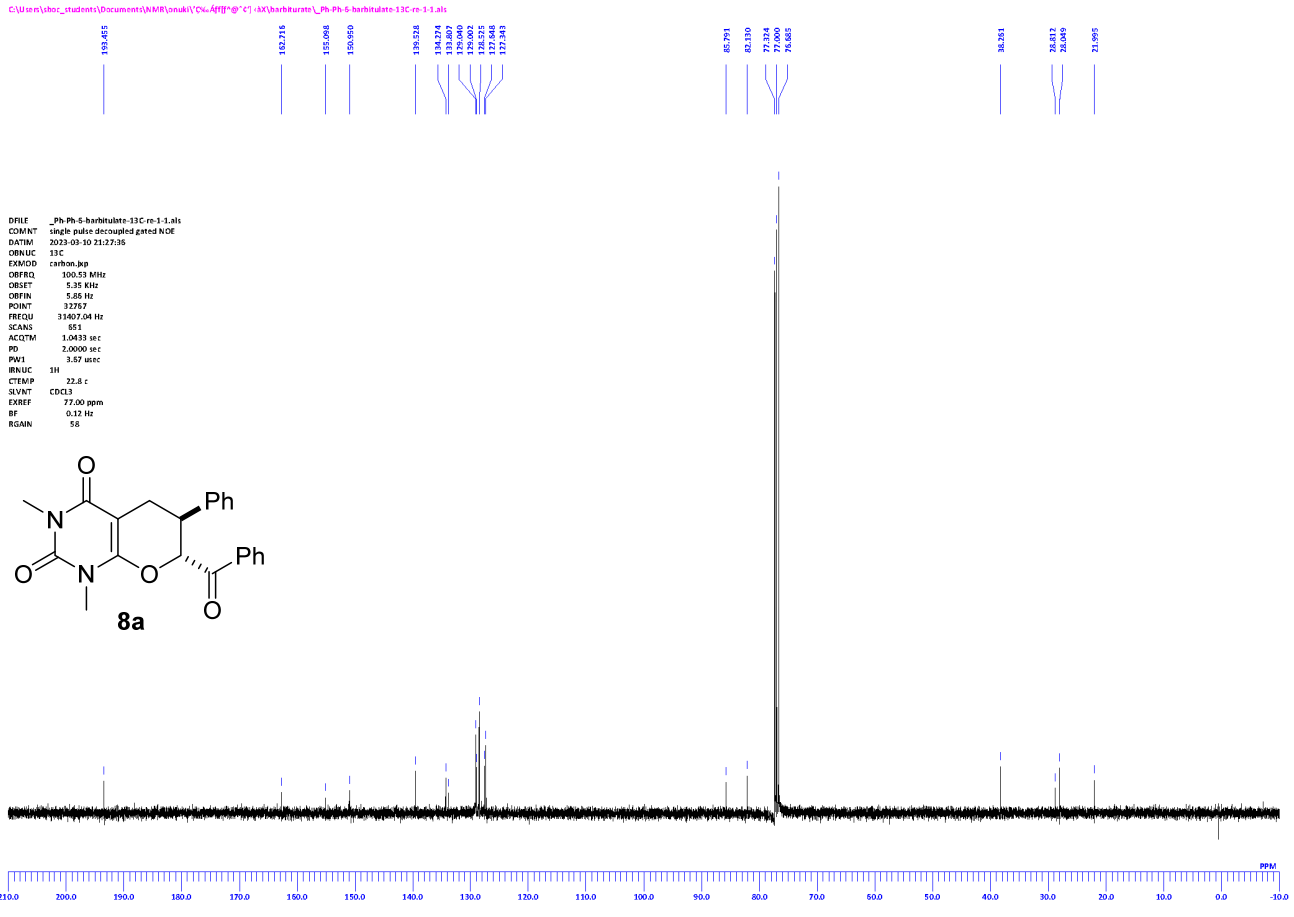

C:\Users\shoc_students\Documents\NMR\aso\~_+¶—p\cyclobutane_mono cyclobutane_1H-1-1.als
DFILE _mono cyclobutane_1H-1-1.als
COMNT single_pulse
DATIM 2022-10-22 18:25:32
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 5002.40 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 32
MeO
Ph
Ph
MeO
O
4
PPM

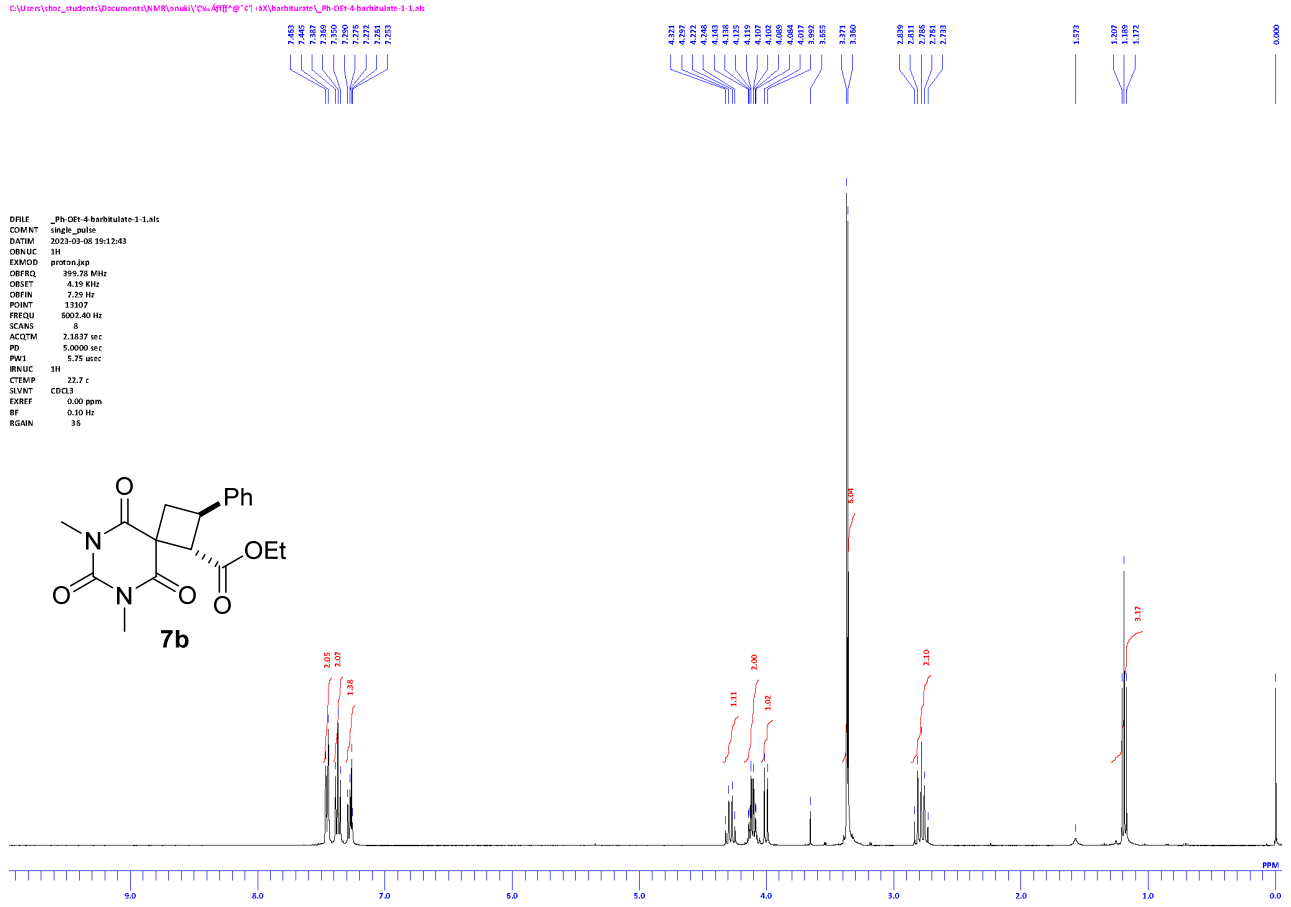

C:\Users\shoc_students\Documents\NMR\aso\~_+¶—p\cyclobutane_mono cyclobutane_13C-1-1.als
DFILE _mono cyclobutane_13C-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2022-10-22 18:27:22
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.85 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 341
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 23.4 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 56
MeO
Ph
Ph
MeO
O
4
PPM

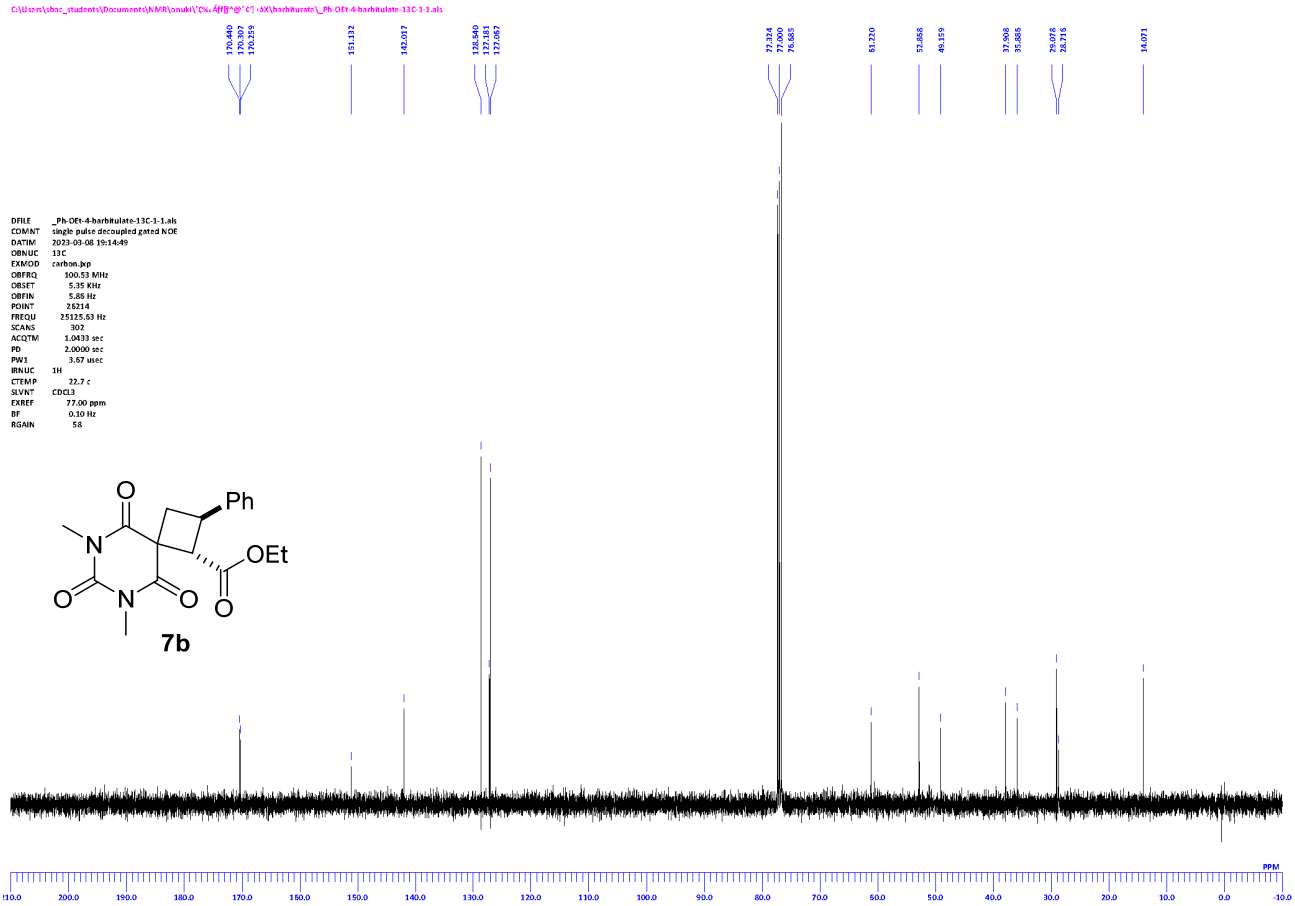

DFILE _Ph-Ph-4-barbiturate-1-1.als
COMNT single_pulse
DATIM 2021-02-17 17:50:04
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 30
7a
PPM

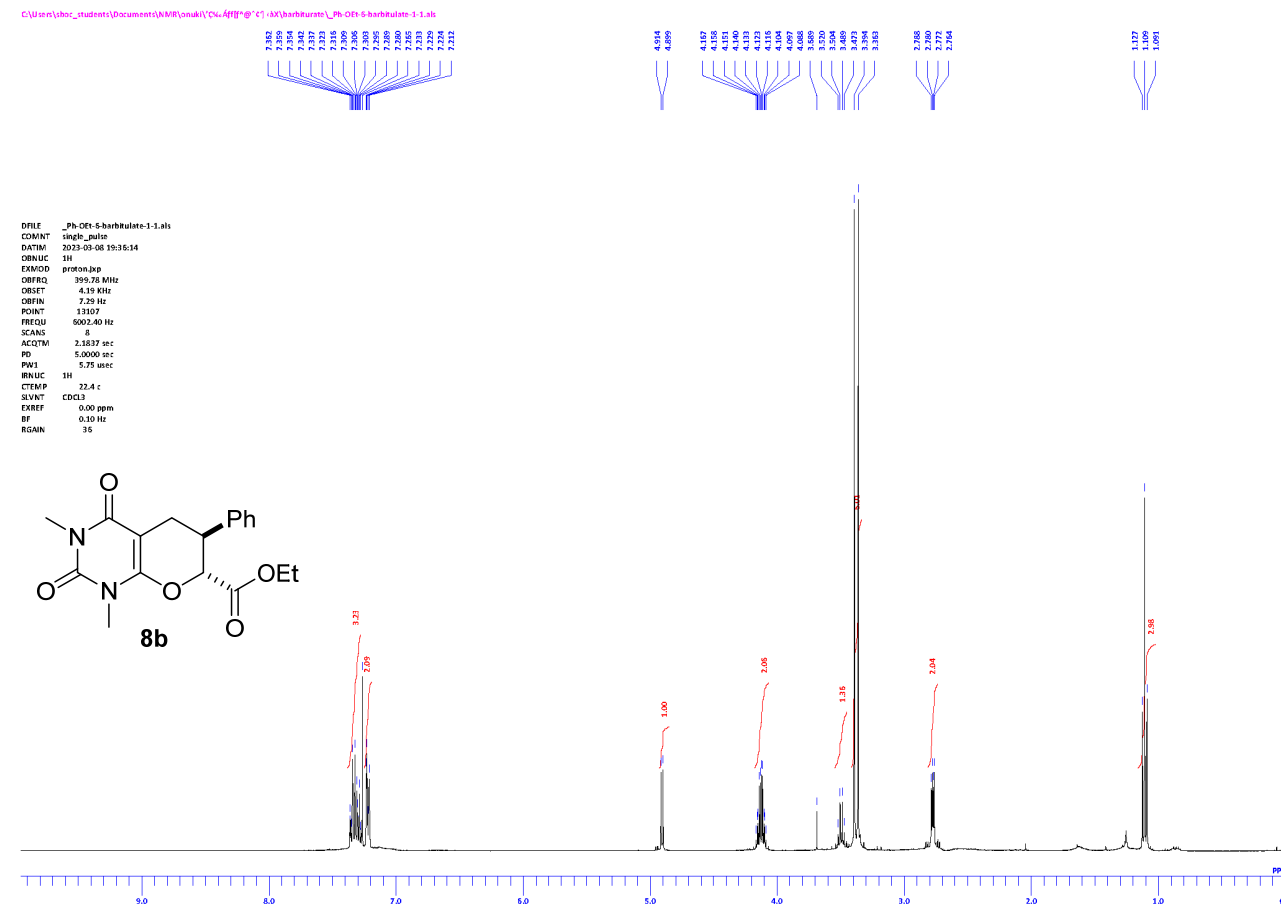

DFILE _Ph-Ph-4-barbiturate-13C-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2021-02-17 17:52:33
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 32767
FREQU 33407.04 Hz
SCANS 175
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 58
7a
PPM

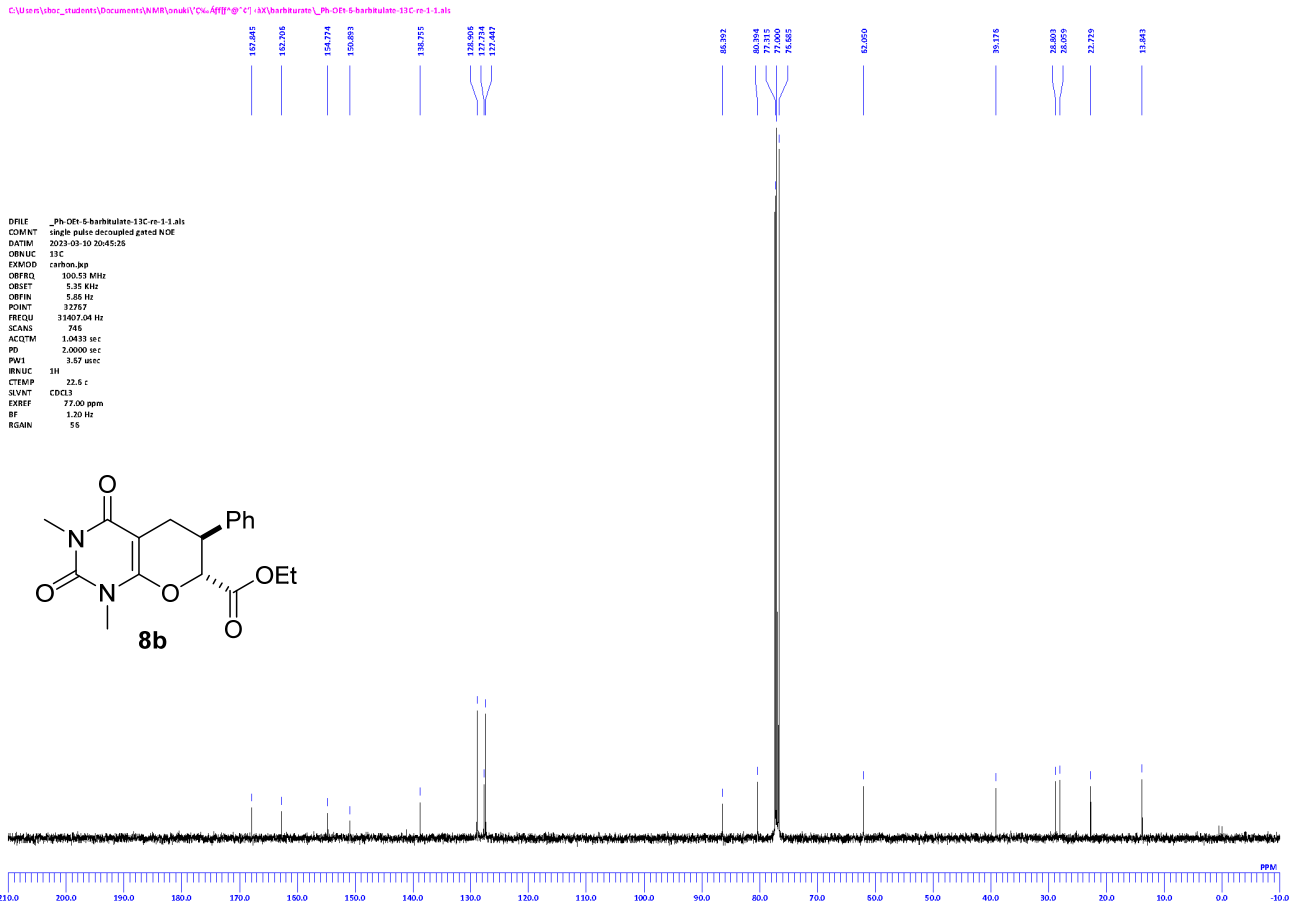

DFILE _Ph-Ph-6-barbiturate-re-1-1.als
COMNT single_pulse
DATIM 2021-02-17 17:44:05
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38
8a
PPM


DFILE _Ph-Ph-6-barbitulate-13C-re-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2023-03-10 21:27:35
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 32767
FREQU 31407.04 Hz
SCANS 851
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 22.8 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 58
8a
PPM


DFILE _Ph-OEt-4-barbitulate-1-1.als
COMNT single_pulse
DATIM 2023-03-08 19:12:43
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 5002.40 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 22.7 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 36
Ph
OEt
7b
PPM


DFILE _Ph-OEt-4-barbitulate-13C-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2023-03-08 19:14:49
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.88 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 302
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 22.7 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.10 Hz
RGAIN 58
Ph
OEt
7b
PPM


8b


8b