Supplemental Information

Synthesis of spirobarbiturate-pyrrolidinones via a domino aza-Michael/S_N2 cyclization of barbiturate-derived alkenes with *N*-alkoxy *α*-haloamides

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1. General Information

All reactions were performed in oven-dried glassware with magnetic stirring under air atmosphere. Unless otherwise stated, all reagents were purchased from commercial suppliers and used without further purification. All solvents were purified and dried according to standard methods prior to use. Organic solutions were concentrated under reduced pressure on a rotary evaporator or an oil pump. Reactions were monitored through thin layer chromatography (TLC) on silica gel-precoated glass plates. Subsequent to elution, plates were visualized using UV radiation at 254 nm and by staining with aqueous potassium permanganate or ethanolic phosphomolybdic acid solution. Flash column chromatography was performed using silica gel (300-400 mesh). Nuclear Magnetic Resonance (NMR) spectras were acquired on a Varian Mercury 400 operating at 400, 100 and 376 MHz for ^{1}H, ^{13}C and ^{19}F, respectively. Chemical shifts are reported in δ ppm referenced to an internal $SiMe_4$ standard for ^{1}H NMR, chloroform-d (δ 77.16) for ^{13}C NMR. Datas for ^{1}H NMR are recorded as follows: chemical shift (δ ppm), multiplicity (s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br s, broad), coupling constant (Hz) and integration. Datas for ^{13}C NMR and ^{19}F NMR are reported in terms of chemical shift (δ, ppm). High-resolution mass spectra (HRMS) were recorded on a Thermo Q-Exactive Spectrometer (ESI).

2. General Procedure for Synthesis of Barbiturate-Derived Alkenes 1

Method A: To a stirred solution of 1,3-dimethylbarbiturate (2 mmol, 1 equiv) in water (16 mL), the aldehydes was added (2 mmol, 1 equiv) rapidly and all at once. After stirring for about two hours, the produced solid was isolated by simple filtration. Dry the solid. The product **1** was identified by ^{1}H NMR analysis and could be used without further purification. [1]

Method B: 1,3-dimethylbarbiturate (10 mmol), the aldehydes (30 mmol) and BF_3•Et_2O (5 mmol) were added sequentially to 20 mL of CH_2Cl_2 and the resulting mixture was stirred at room temperature for about 20 h. After the reaction was completed, the solution was evaporated in vacuo. The residue was purified through flash column chromatography to afford the corresponding product **1**.[2]

*5-Benzylidene-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione (**1a**).*[3] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (s, 1H), 8.07-8.04 (m, 2H), 7.55-7.45 (m, 3H), 3.43 (s, 3H), 3.38 (s, 3H) ppm.

*5-(4-Methoxybenzylidene)-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione (**1b**).*[3] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (s, 1H), 8.35-8.31 (m, 2H), 7.00-6.96 (m, 2H), 3.91 (s, 3H), 3.42 (s, 3H), 3.40 (s, 3H) ppm.

*1,3-Dimethyl-5-(4-methylbenzylidene)pyrimidine-2,4,6(1H,3H,5H)-trione (**1c**).*[4] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.55 (s, 1H), 8.08-8.05 (m, 2H), 7.30-7.28 (m, 2H), 3.42 (s, 3H), 3.38 (s, 3H), 2.44 (s, 3H) ppm.

*5-(4-Fluorobenzylidene)-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione (**1d**).*[5] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (s, 1H), 8.22-8.17 (m, 2H), 7.19-7.13 (m, 2H), 3.43 (s, 3H), 3.39 (s, 3H) ppm.

*5-(4-Chlorobenzylidene)-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione (**1e**).*[6] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (s, 1H), 8.05-8.02 (m, 2H), 7.46-7.43 (m, 2H), 3.43 (s, 3H), 3.38 (s, 3H) ppm.

*5-(4-Bromobenzylidene)-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione (**1f**).*[3] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (s, 1H), 7.95-7.93 (m, 2H), 7.61-7.59 (m, 2H), 3.42 (s, 3H), 3.37 (s, 3H) ppm.

*4-((1,3-Dimethyl-2,4,6-trioxotetrahydropyrimidin-5(2H)-ylidene)methyl)benzonitrile (**1g**).*[7] Synthesized

according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (s, 1H), 7.95-7.93 (m, 2H), 7.74-7.72 (m, 2H), 3.44 (s, 3H), 3.36 (s, 3H) ppm.

1,3-Dimethyl-5-(4-(trifluoromethyl)benzylidene)pyrimidine-2,4,6(1H,3H,5H)-trione ***(1h)***.[8] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.57 (s, 1H), 7.80-7.98 (m, 2H), 7.72-7.70 (m, 2H), 3.44 (s, 3H), 3.37 (s, 3H) ppm.

1,3-Dimethyl-5-(4-nitrobenzylidene)pyrimidine-2,4,6(1H,3H,5H)-trione ***(1i)***.[3] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.84-8.83 (m, 1H), 8.57 (s, 1H), 8.36-8.34 (m, 1H), 8.19-8.16 (m, 1H), 7.68-7.64 (m,1H), 3.45 (s, 3H), 3.38 (s, 3H) ppm.

5-(3-Methoxybenzylidene)-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione ***(1j)***.[3] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.55 (s, 1H), 7.79-7.78 (m, 1H), 7.58-7.56 (m, 1H), 7.40-7.36 (m, 1H), 7.10-7.08 (m, 1H), 3.87 (s, 3H), 3.43 (s, 3H), 3.38 (s, 3H) ppm.

5-(3-Fluorobenzylidene)-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione ***(1k)***.[4] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (s, 1H), 7.93-7.90 (m, 1H), 7.69-7.67 (m, 1H), 7.47-7.41 (m, 1H), 7.25-7.21 (m, 1H), 3.43 (s, 3H), 3.38 (s, 3H) ppm.

5-(3-Chlorobenzylidene)-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione ***(1l)***.[6] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (s, 1H), 8.06-8.05 (m, 1H), 7.85-7.82 (m, 1H), 7.50-7.48 (m, 1H), 7.42-7.39 (m, 1H), 3.43 (s, 3H), 3.38 (s, 3H) ppm.

1,3-Dimethyl-5-(3-nitrobenzylidene)pyrimidine-2,4,6(1H,3H,5H)-trione ***(1m)***.[3] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.57 (s, 1H), 8.31-8.27 (m, 2H), 7.97-7.94 (m, 2H), 3.45 (s, 3H), 3.36 (s, 3H) ppm.

5-(2-Methoxybenzylidene)-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione ***(1n)***.[9] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.89 (s, 1H), 8.04 (dd, J = 7.8 Hz, J = 1.4 Hz, 1H), 7.52-7.47 (m, 1H), 7.03-7.00 (m, 1H), 6.94 (d, J = 8.4 Hz, 1H), 3.90 (s, 3H), 3.42 (s, 3H), 3.35 (s, 3H) ppm.

1,3-Dimethyl-5-(2-methylbenzylidene)pyrimidine-2,4,6(1H,3H,5H)-trione ***(1o)***.[10] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.77 (s, 1H), 7.63 (d, J = 7.6 Hz, 1H), 7.39-7.35 (m, 1H), 7.26-7.23 (m, 2H), 3.43 (s, 3H), 3.32 (s, 3H), 2.36 (s, 3H) ppm.

5-(2-Bromobenzylidene)-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione ***(1p)***.[4] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (s, 1H), 7.69-7.64 (m, 2H), 7.40-7.36 (m, 1H), 7.34-7.30 (m, 1H), 3.44 (s, 3H), 3.32 (s, 3H) ppm.

1,3-Dimethyl-5-(2-(trifluoromethyl)benzylidene)pyrimidine-2,4,6(1H,3H,5H)-trione ***(1q)***.[4] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.81-8.79 (m, 1H), 7.75 (d, J = 7.6 Hz, 1H), 7.62-7.53 (m, 2H), 7.46-7.45 (m, 1H), 3.44 (s, 3H), 3.28 (s, 3H) ppm.

1,3-Dimethyl-5-(2-nitrobenzylidene)pyrimidine-2,4,6(1H,3H,5H)-trione ***(1r)***.[6] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.89 (s, 1H), 8.30 (dd, J = 8.0 Hz, J = 1.0 Hz, 1H), 7.75-7.71 (m, 1H), 7.65-7.61 (m, 1H), 7.40-7.38 (m, 1H), 3.45 (s, 3H), 3.25 (s, 3H) ppm.

5-(2,4-Dimethoxybenzylidene)-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione ***(1s)***.[11] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.98 (s, 1H), 8.56 (d, J = 8.8 Hz, 1H), 6.59-6.56 (m, 1H), 6.42 (d, J = 2.4 Hz, 1H), 3.91-3.90 (m, 6H), 3.41 (s, 3H), 3.37 (s, 3H) ppm.

1,3-Dimethyl-5-(naphthalen-1-ylmethylene)pyrimidine-2,4,6(1H,3H,5H)-trione ***(1t)***.[6] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.24 (s, 1H), 8.00-7.95 (m, 2H), 7.93-7.86 (m, 2H), 7.59-7.53 (m, 3H), 3.48 (s, 3H), 3.32 (s, 3H) ppm.

1,3-Dimethyl-5-(naphthalen-2-ylmethylene)pyrimidine-2,4,6(1H,3H,5H)-trione ***(1u)***.[12] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.74 (s, 1H), 8.60 (s, 1H), 8.16 (dd, J = 8.8 Hz,

J = 1.6 Hz, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.89-7.86 (m, 2H), 7.63-7.59 (m, 1H), 7.57-7.53 (m, 1H), 3.45 (s, 3H), 3.41 (s, 3H) ppm.

1,3-Dimethyl-5-(thiophen-3-ylmethylene)pyrimidine-2,4,6(1H,3H,5H)-trione ***(1v)***.[13] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.01-9.00 (m, 1H), 8.54 (s, 1H), 7.90 (dd, *J* = 5.0 Hz, *J* = 1.0 Hz, 1H), 7.40-7.38 (m, 1H), 3.42 (s, 3H), 3.41 (s, 3H) ppm.

5-(Benzo[d][1,3]dioxol-5-ylmethylene)-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione ***(1w)***.[14] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.45 (s, 1H), 8.22 (d, *J* = 1.6 Hz, 1H), 7.62 (dd, *J* = 8.4 Hz, *J* = 1.6 Hz, 1H), 6.91 (d, *J* = 8.4 Hz, 1H), 6.10 (s, 2H), 3.41 (s, 3H), 3.39 (s, 3H) ppm.

5-(Cyclohexylmethylene)-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione ***(1x)***.[4] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 10.0 Hz, 1H), 3.76-3.66 (m, 1H), 3.36 (s, 3H), 3.35 (s, 3H), 1.82-1.72 (m, 5H), 1.46-1.36 (m, 2H), 1.30-1.20 (m, 3H) ppm.

1,3-Dimethyl-5-(2-methylpropylidene)pyrimidine-2,4,6(1H,3H,5H)-trione ***(1y)***.[15] Synthesized according to method B. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* =10.4 Hz, 1H), 4.03-3.94 (m, 1H), 3.36 (s, 3H), 3.34 (s, 3H), 1.16 (s, 3H), 1.15 (s, 3H) ppm.

3. General Procedure for Synthesis of *N*-Alkoxy *α*-Haloamides 2[16]

Method A: To a solution of O-substituted hydroxylamine hydrochloride (15 mmol, 1.0 equiv) in ethyl acetate/water (75/75 mL), Na_2CO_3 (30 mmol, 2.0 equiv) was added. The reaction mixture was then cooled to 0 ^{o}C in an ice water bath. Next, 2-halo acyl bromide (18 mmol, 1.2 equiv) was added dropwise to the reaction mixture. The reaction was stirred at 0 ℃ under air atmosphere. After 4 h, the mixture was allowed to warm to room temperature. The resulting mixture was diluted with ethyl acetate and washed with 3x water, followed by brine. The organic layers were dried over anhydrous sodium sulfate, filtered and concentrated under vacuum. When the solvent is concentrated to 3-5 mL, add 40-50 mL petroleum ether and place it in the refrigerator at -20 ^{o}C for 30 min. Filter to get white powdery *N*-alkoxy *α*-haloamides **2** and the products can be used without further purification.

Method B: To a solution of O-substituted hydroxylamine hydrochloride (12.5 mmol, 1.0 equiv) in dichloromethane (50 mL), triethylamine (1.75 mL, 12.5 mmol, 1.0 equiv) was added. The reaction mixture was then cooled to 0 ^{o}C in an ice water bath. Next, 2-halo acyl bromide (12.5 mmol, 1.0 equiv) was added dropwise to the reaction mixture. The reaction was stirred at 0 ℃ under air atmosphere. After 4 h, the mixture was allowed to warm to room temperature. The reaction was then quenched with water. The resulting mixture was diluted with CH_2Cl_2 and washed with 3x water, followed by brine. The organic layers were dried over anhydrous sodium sulfate, filtered, and concentrated under vacuum. The crude reaction mixture was purified by silica gel chromatography (PE/EtOAc) to give *N*-alkoxy *α*-haloamides **2**.

N-(benzyloxy)-2-bromoacetamide ***(2a)***.[16] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.69 (s, 1H), 7.42-7.38 (m, 5H), 4.95 (s, 2H), 3.80 (s, 2H) ppm.

N-(benzyloxy)-2-chloroacetamide ***(2b)***.[16] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.97 (s, 1H), 7.42-7.38 (m, 5H), 4.94 (s, 2H), 4.01 (s, 2H) ppm.

2-Bromo-N-methoxyacetamide ***(2c)***.[16] Synthesized according to method B. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.83 (s, 1H), 3.83-3.81 (m, 5H) ppm.

2-Bromo-N-(naphthalen-2-ylmethoxy)acetamide ***(2d)***.[16] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.75 (s, 1H), 7.90-7.85 (m, 4H), 7.55-7.51 (m, 3H), 5.10 (s, 2H), 3.77 (s, 2H) ppm.

N-benzyl-2-bromoacetamide ***(2e)***.[16] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ

7.39-7.29 (m, 5H), 6.75 (s, 1H), 4.49 (d, *J* = 6.0 Hz, 2H), 3.94 (s, 2H) ppm.

*N-(benzyloxy)-2-bromopropanamide (**2f**).*[16] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.84 (s, 1H), 7.42-7.36 (m, 5H), 4.96-4.90 (m, 2H), 4.28-4.27 (m, 1H), 1.86-1.83 (m, 3H) ppm.

*N-(benzyloxy)-2-bromobutanamide (**2g**).*[16] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.93-8.90 (m, 1H), 7.40-7.38 (m, 5H), 4.96-4.90 (m, 2H), 4.14-4.10 (m, 1H), 2.15-1.98 (m, 2H), 1.00 (t, *J* = 7.0 Hz, 3H) ppm.

*N-(benzyloxy)-2-bromopentanamide (**2h**).*[16] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.81 (s, 1H), 7.40-7.38 (m, 5H), 4.96-4.90 (m, 2H), 4.17-4.14 (m, 1H), 2.07-1.91 (m, 2H), 1.49-1.40 (m, 2H), 0.93(t, *J* = 7.4 Hz, 3H) ppm.

*N-(benzyloxy)-2-bromohexanamide (**2i**).*[16] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.95 (s, 1H), 7.40-7.38 (m, 5H), 4.96-4.90 (m, 2H), 4.16-4.12 (m, 1H), 1.98-1.86 (m, 2H), 1.41-1.32 (m, 4H), 0.92-0.88 (m, 3H) ppm.

*N-(benzyloxy)-2-bromo-2-phenylacetamide (**2j**).*[17] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.05 (br s, 1H), 7.40-7.32 (m, 10H), 5.19 (s, 1H), 4.92 (s, 2H) ppm.

*N-(benzyloxy)-2-bromo-2-methylpropanamide (**2k**).*[16] Synthesized according to method A. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.00 (s, 1H), 7.35-7.32 (m, 5H), 4.87-4.86 (m, 2H), 1.87-1.84 (m, 6H) ppm.

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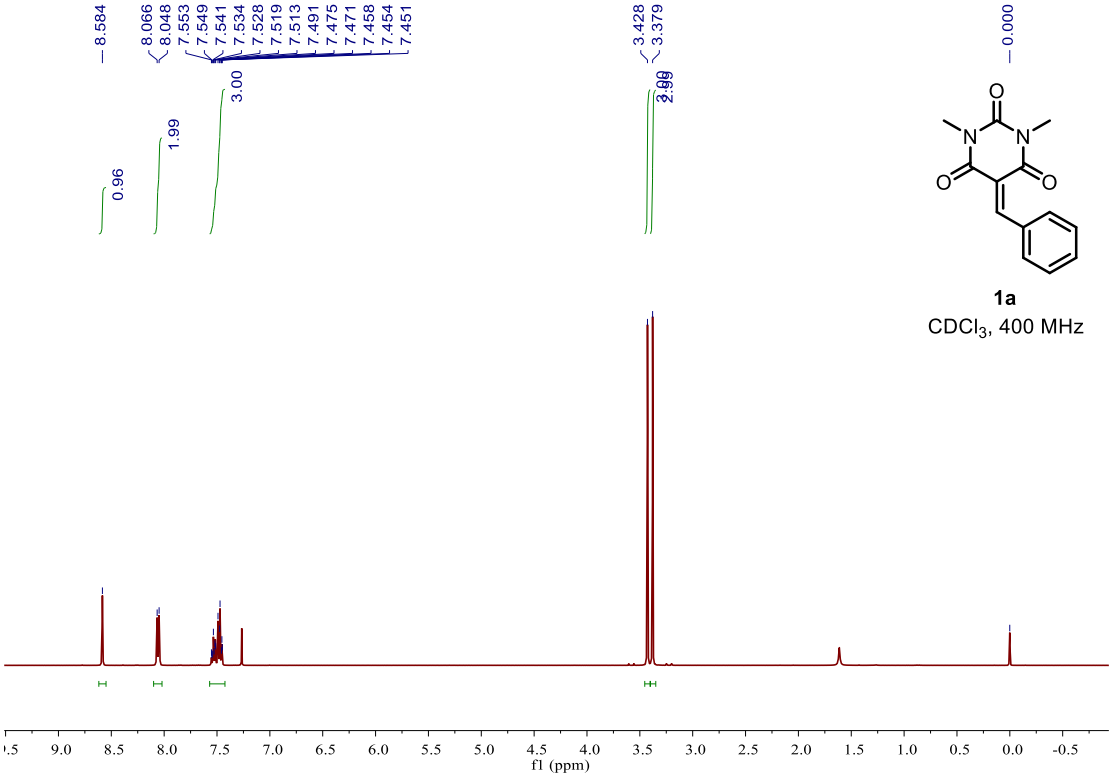
5. Copies of NMR Spectras of Barbiturate-Derived Alkenes 1



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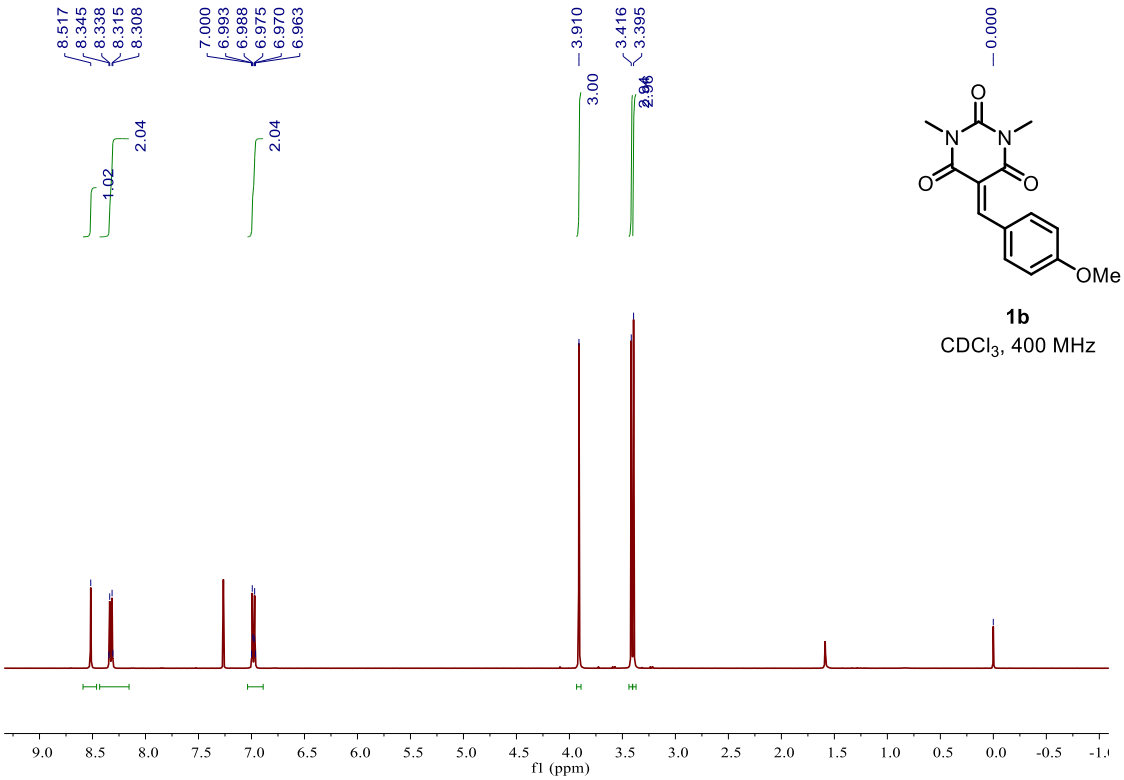

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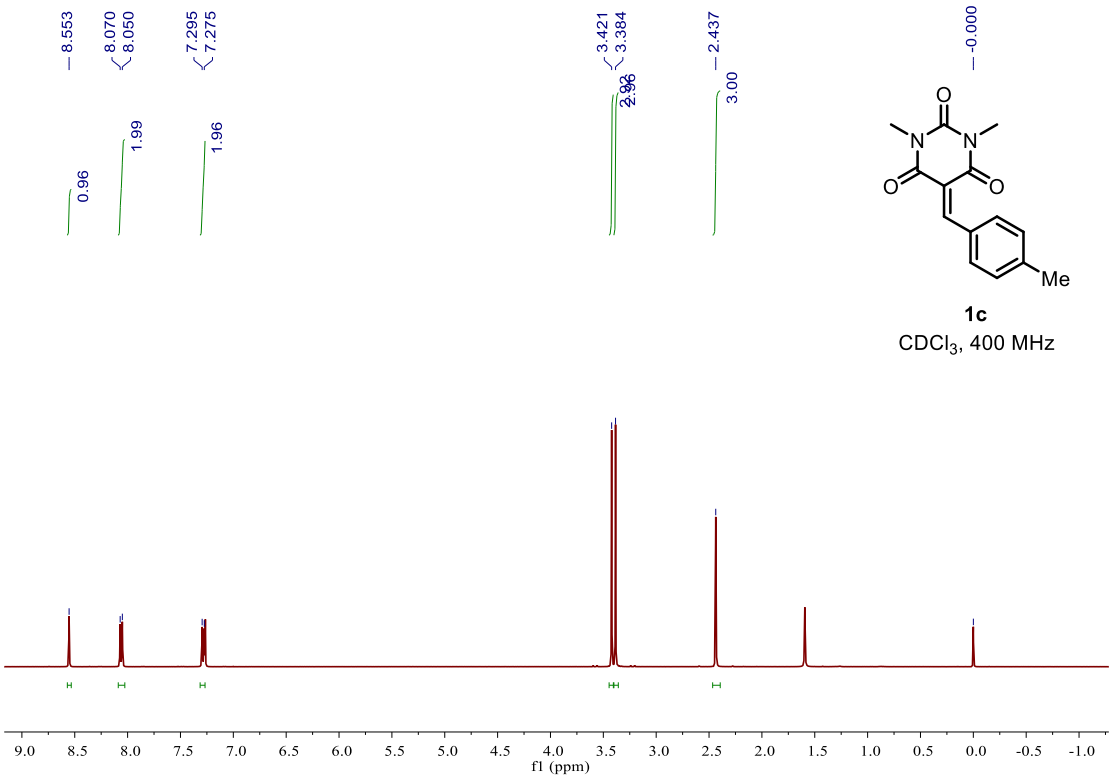

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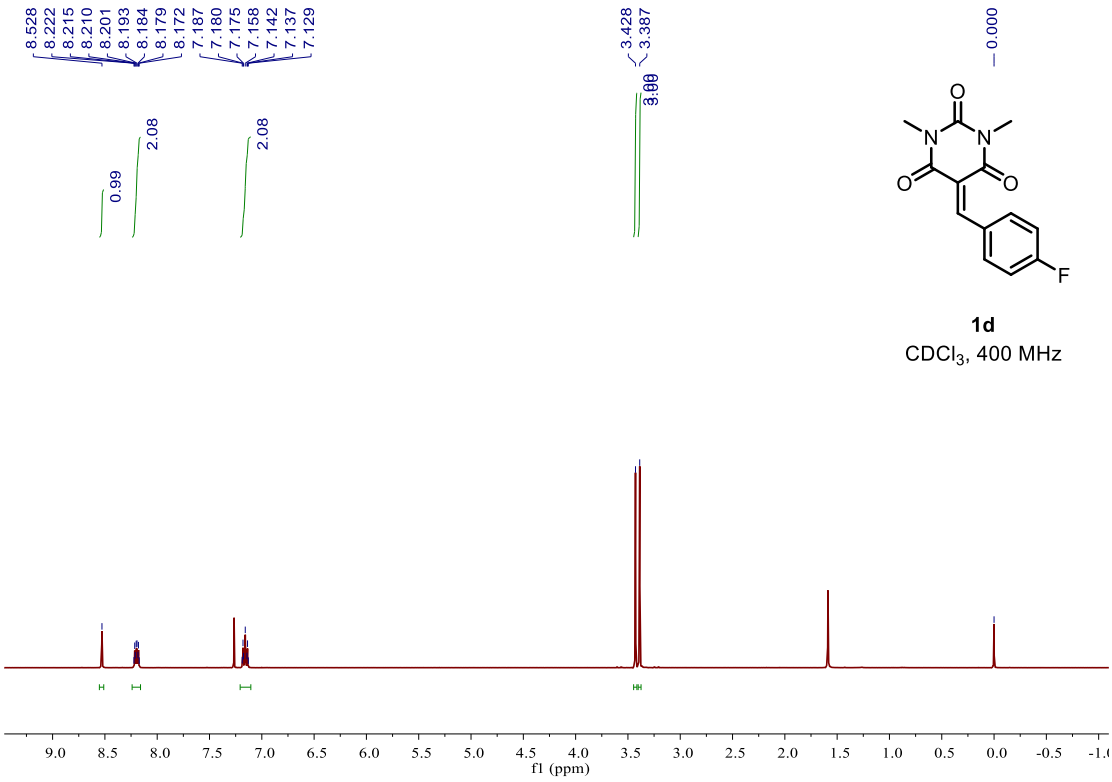

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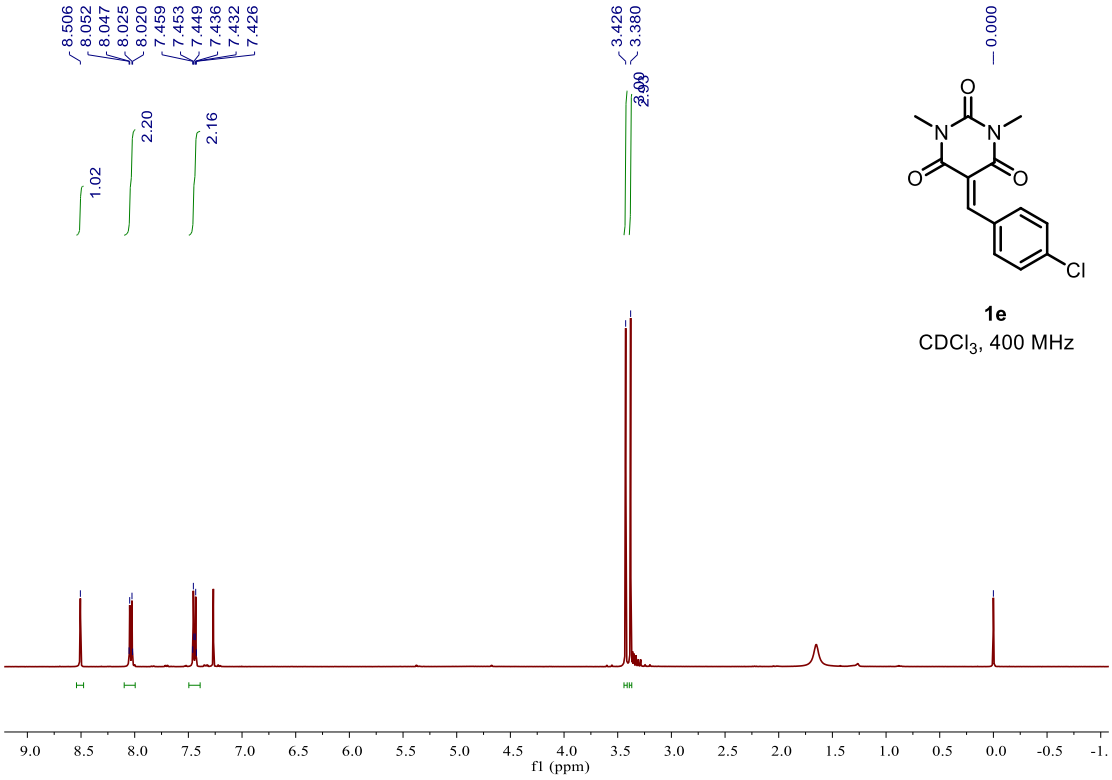

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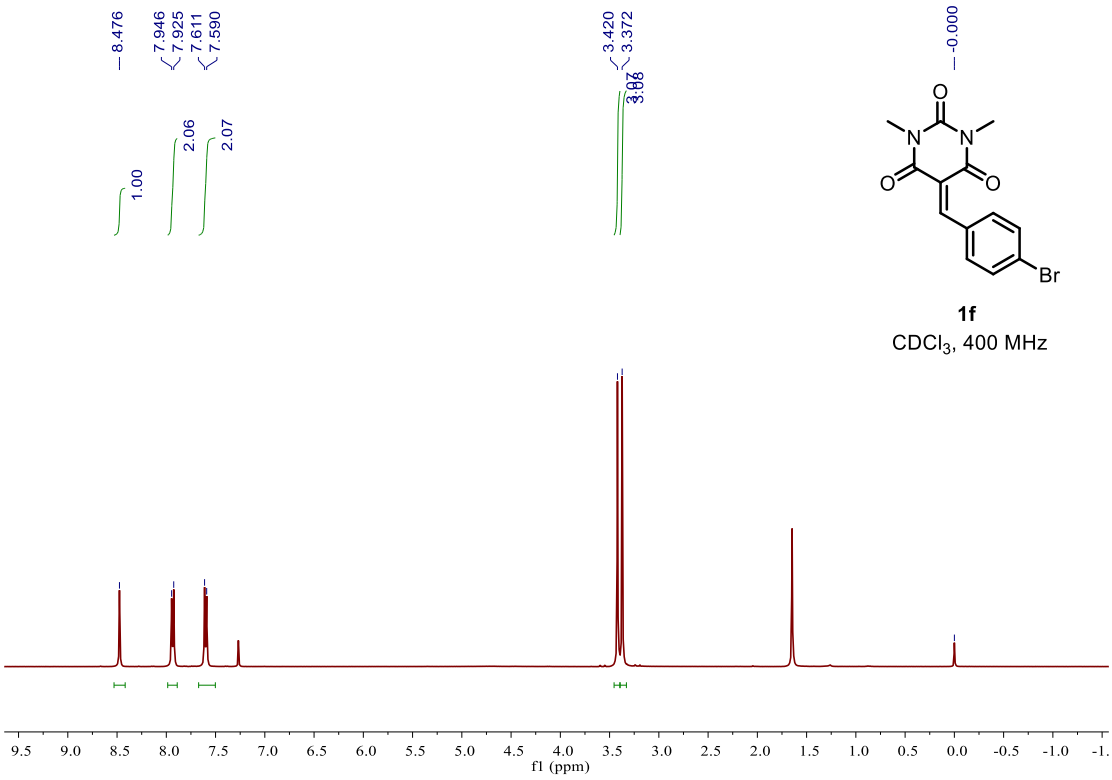

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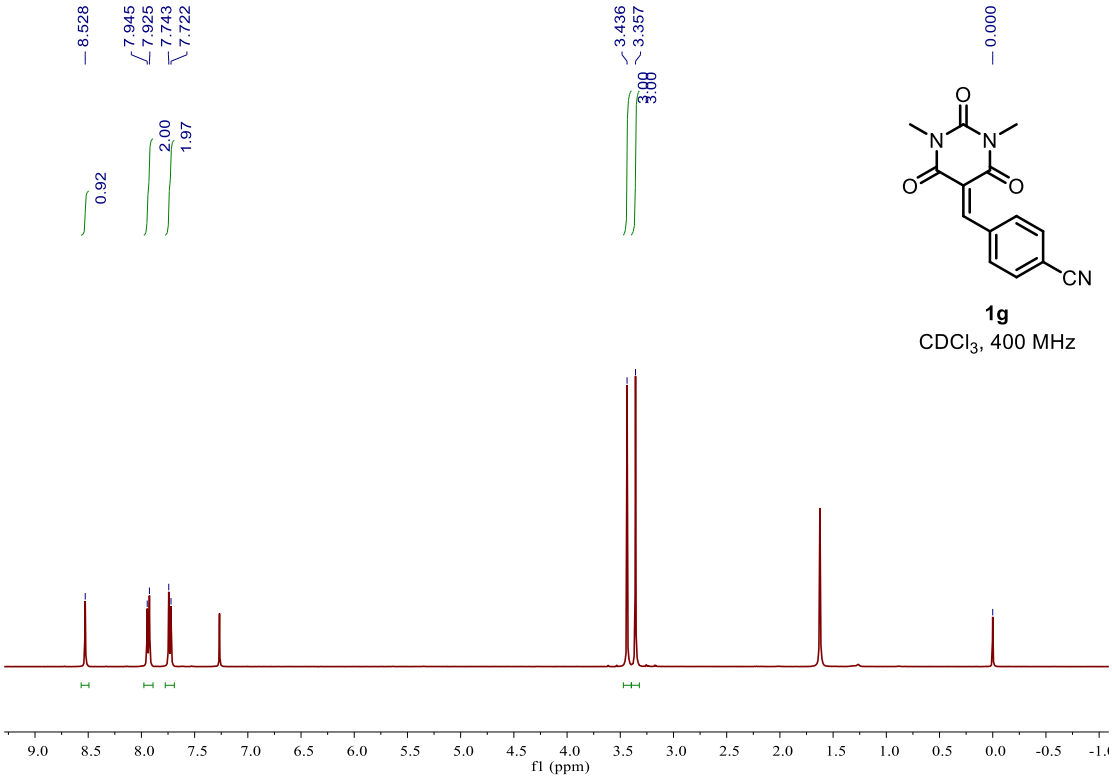
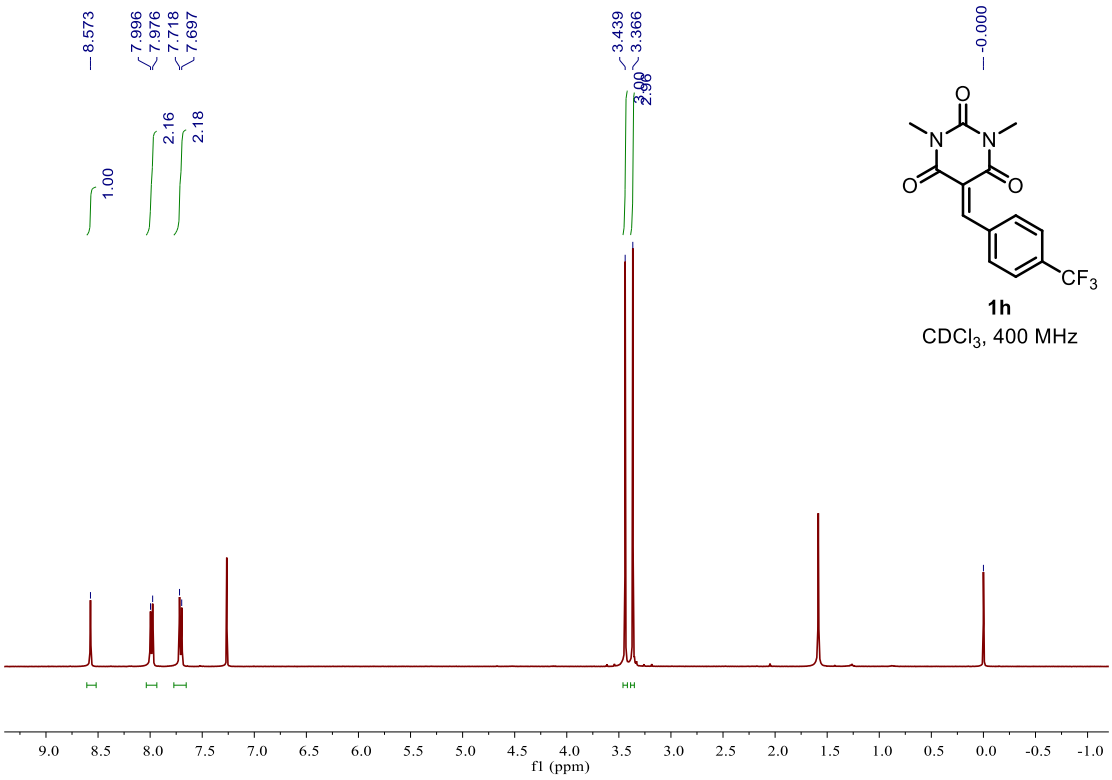


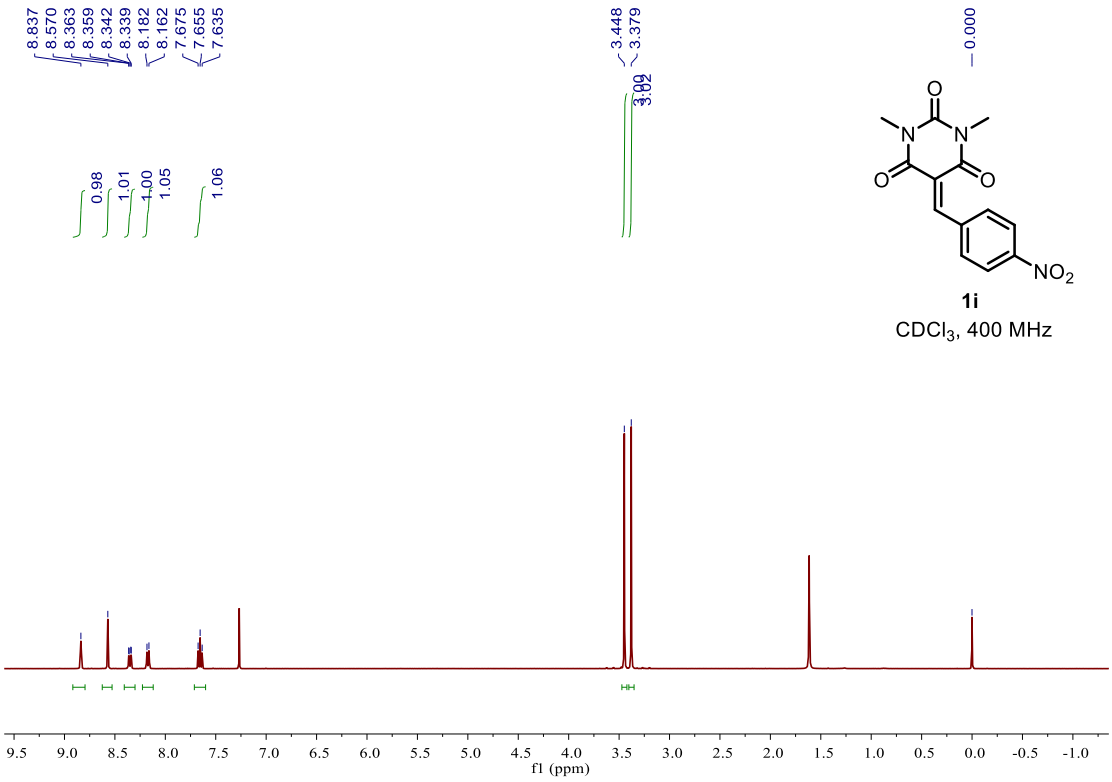
1g

$CDCl_3$, 400 MHz



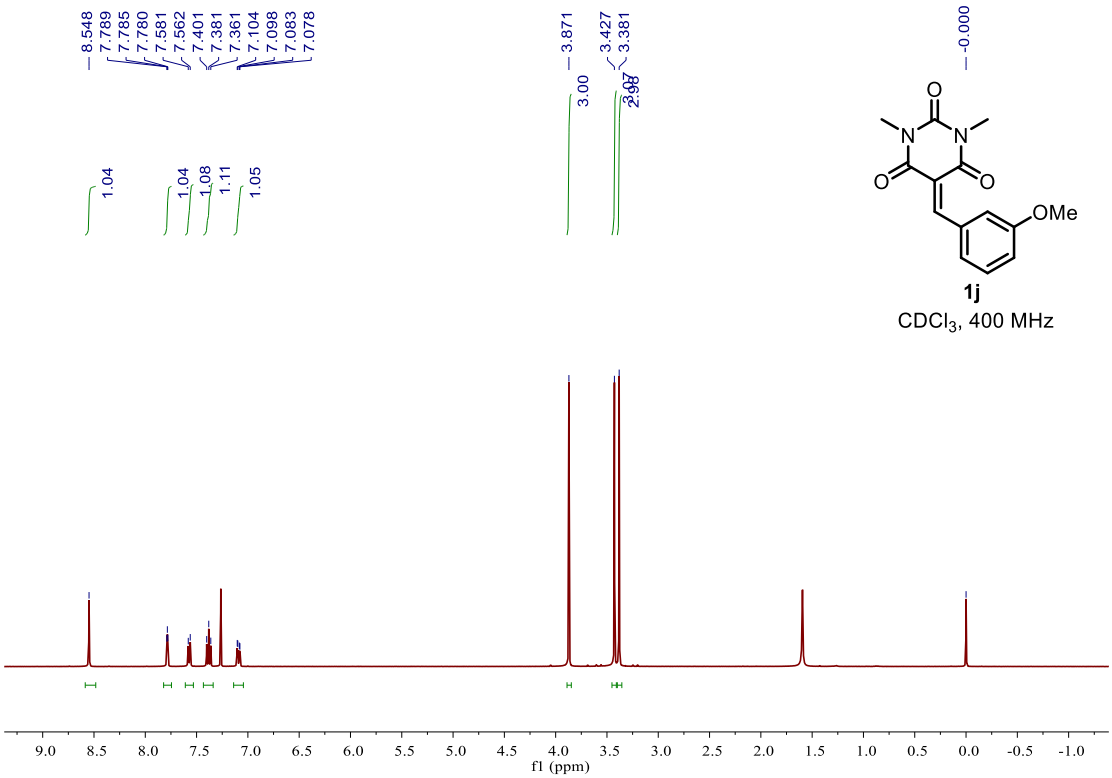
1h

$CDCl_3$, 400 MHz



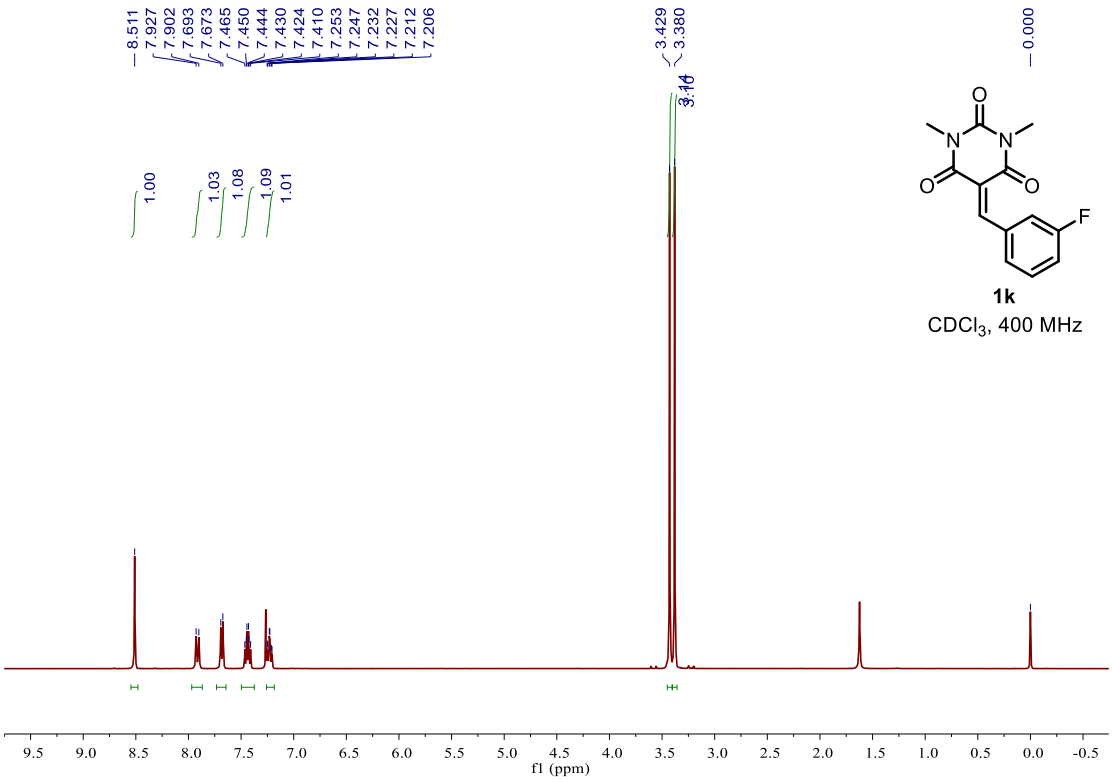
1i

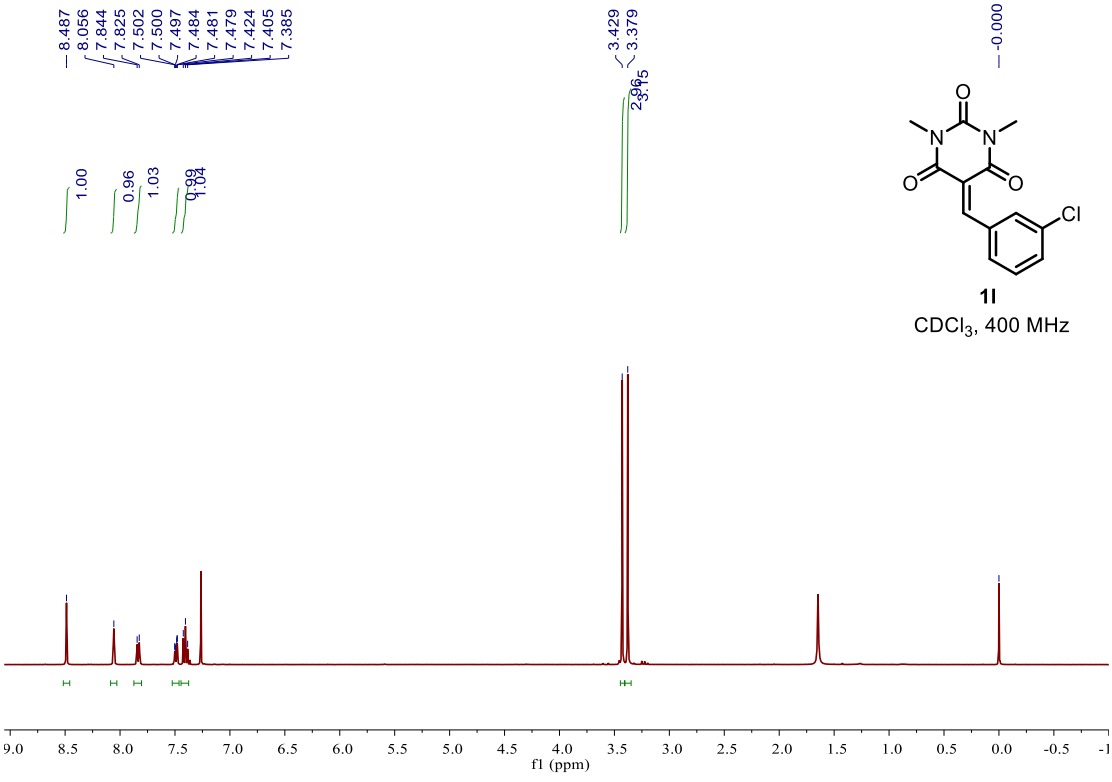
$CDCl_3$, 400 MHz

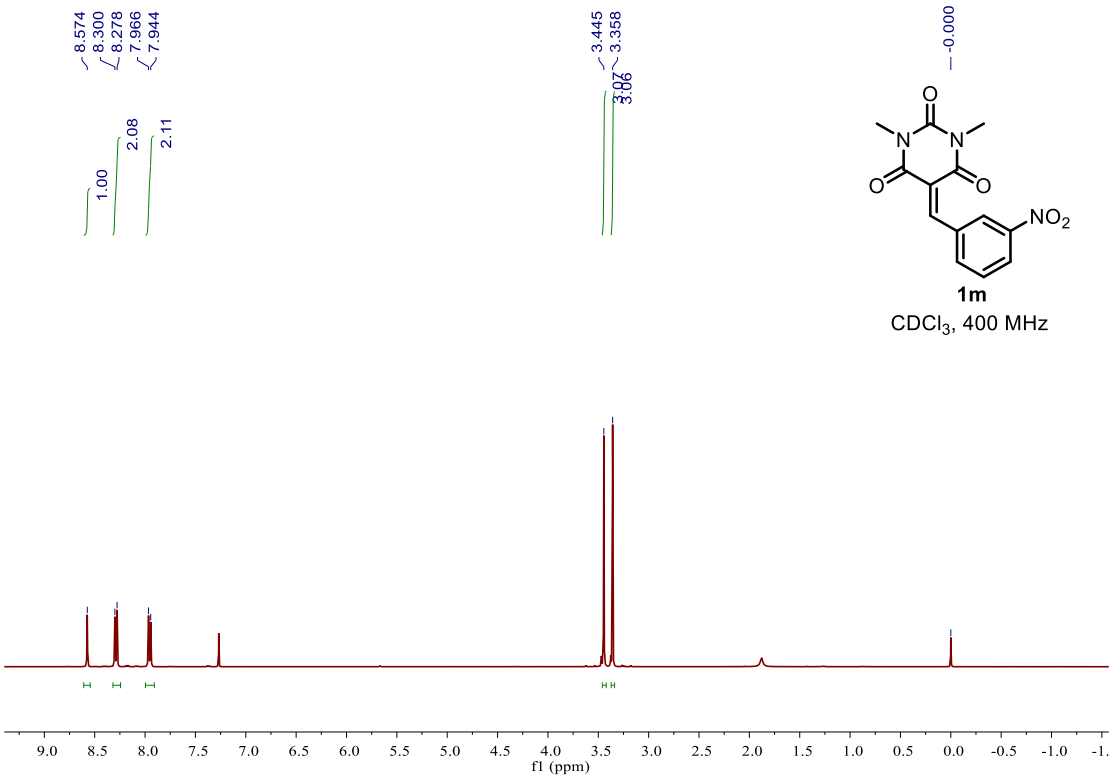


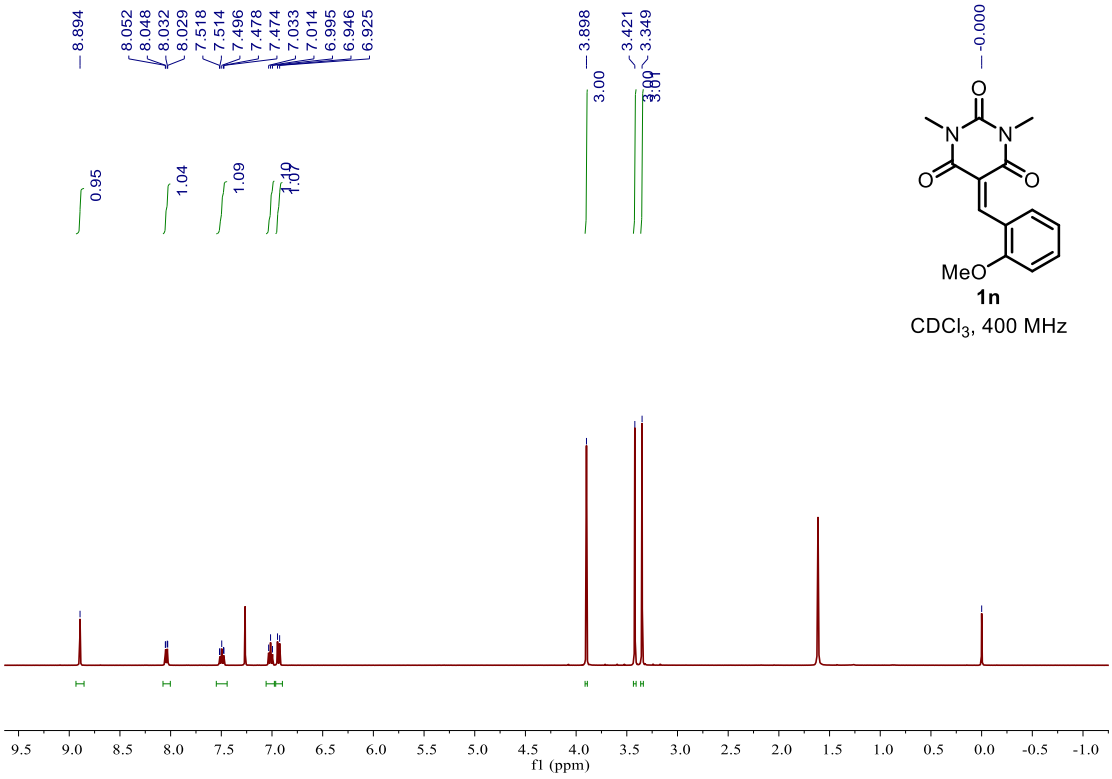
1j

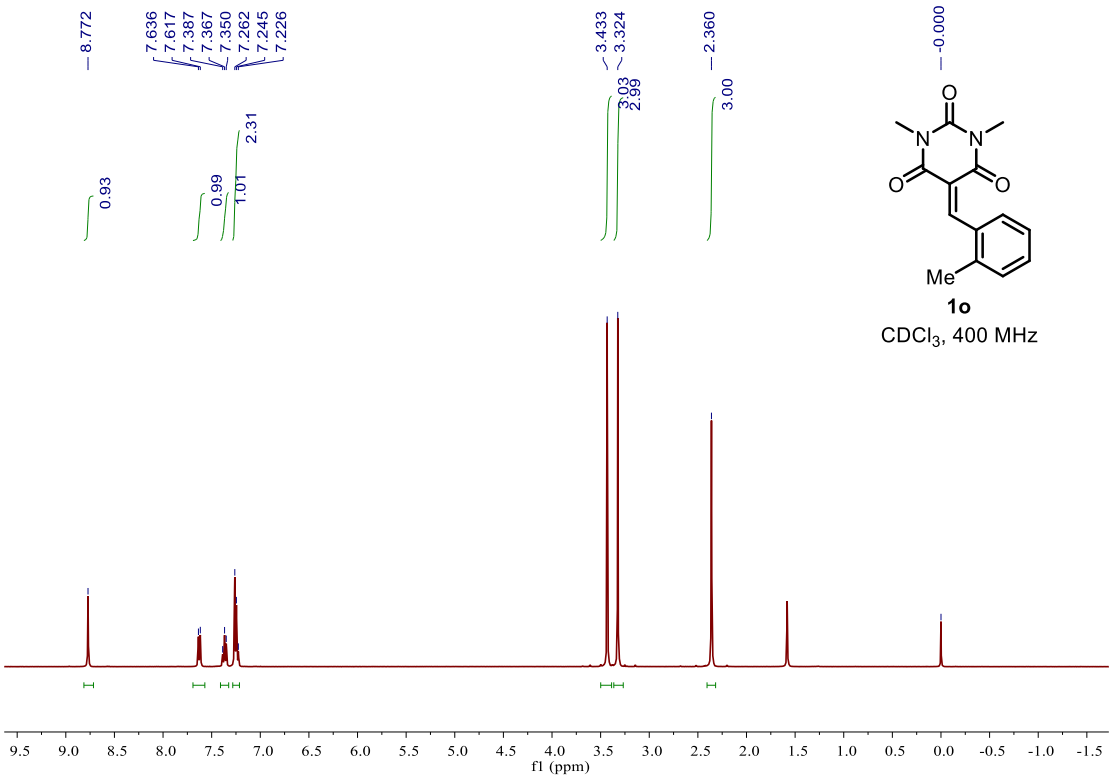
$CDCl_3$, 400 MHz

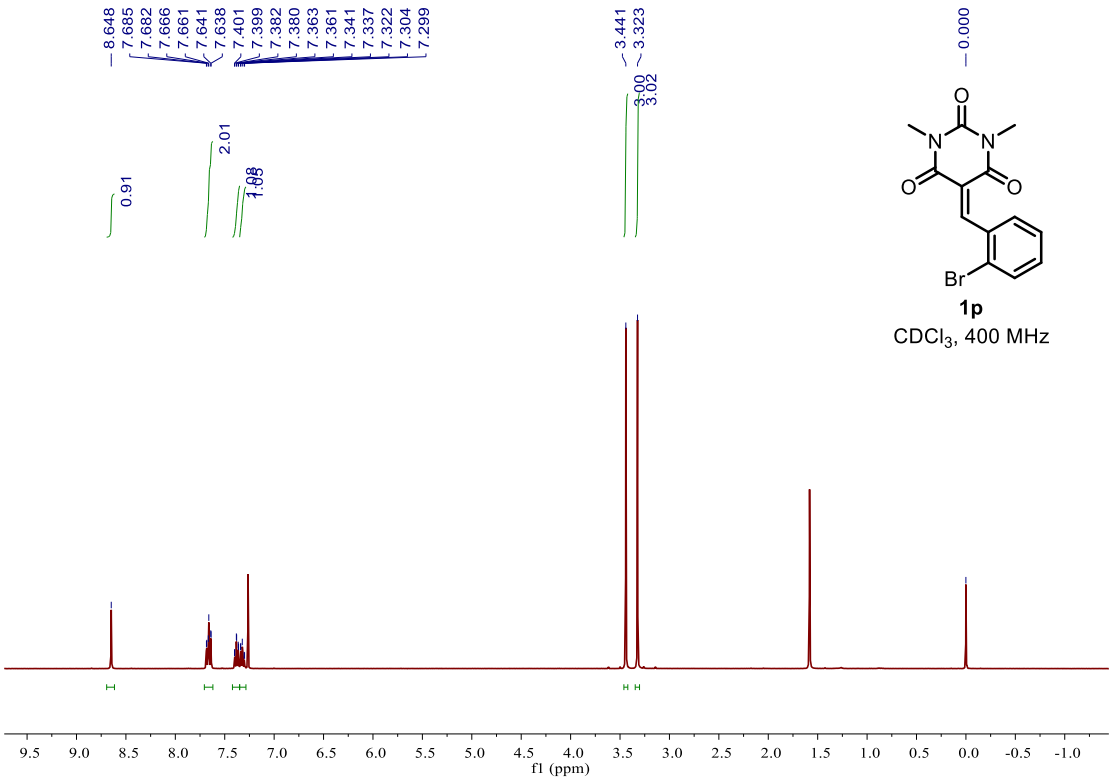

8.511
7.927
7.902
7.693
7.673
7.465
7.450
7.444
7.430
7.424
7.410
7.253
7.247
7.232
7.227
7.212
7.206
3.429
3.380
3.14
0.000
1.00
1.03
1.08
1.09
1.01
O
N
N
O
O
F
1k
CDCl3, 400 MHz
f1 (ppm)

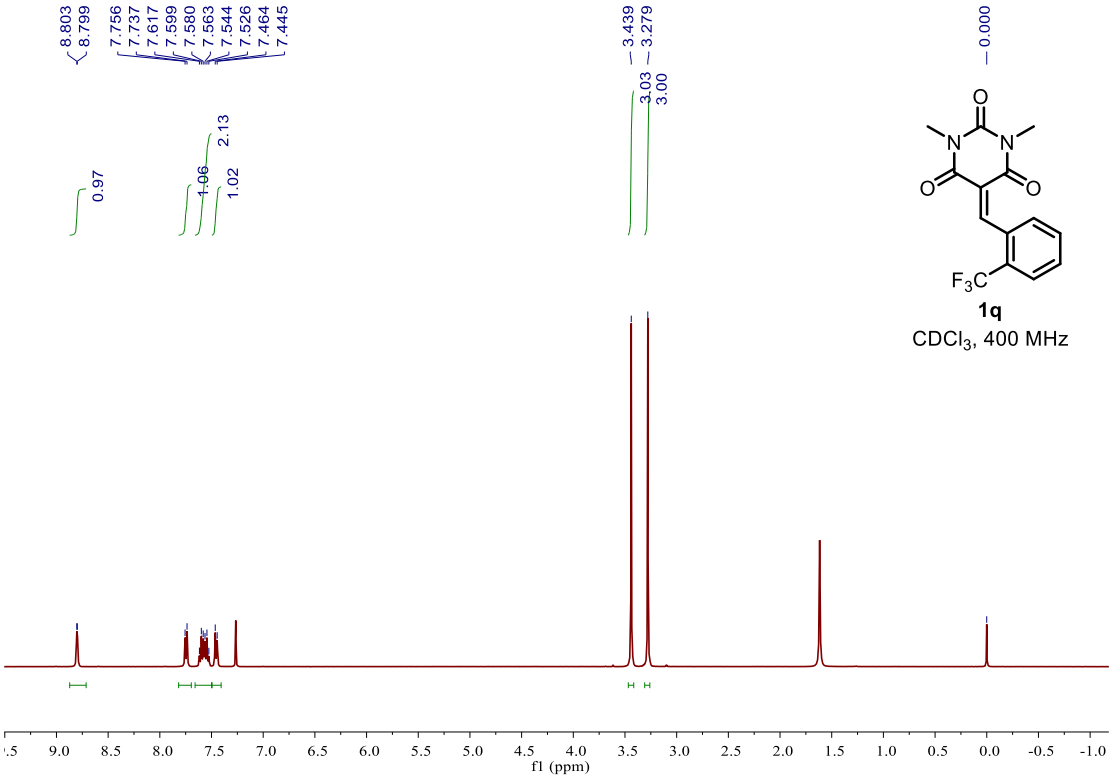

8.487
8.056
7.844
7.825
7.502
7.500
7.497
7.484
7.481
7.479
7.424
7.405
7.385
3.429
3.379
2.96
3.15
0.000
1.00
0.96
1.03
0.99
1.04
O
N
N
O
O
Cl
1l
CDCl3, 400 MHz
f1 (ppm)

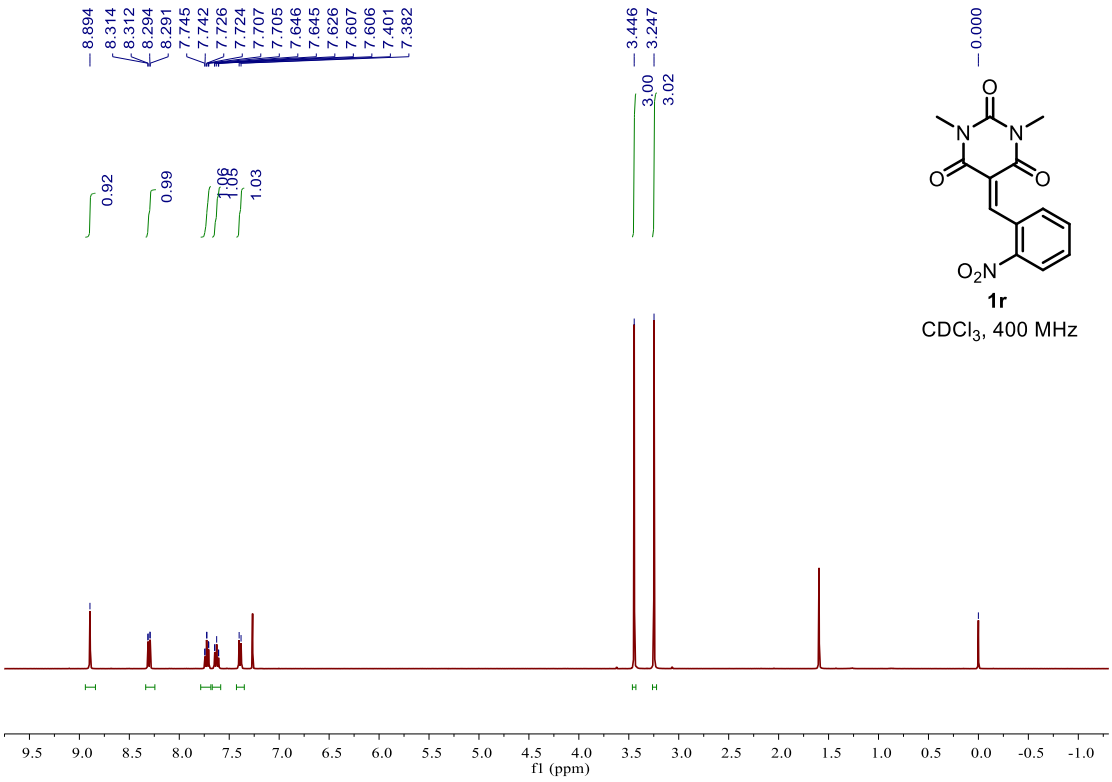

8.574
8.300
8.278
7.966
7.944
1.00
2.08
2.11
3.445
3.358
3.07
3.06
-0.000
NO2
1m
CDCl3, 400 MHz
f1 (ppm)

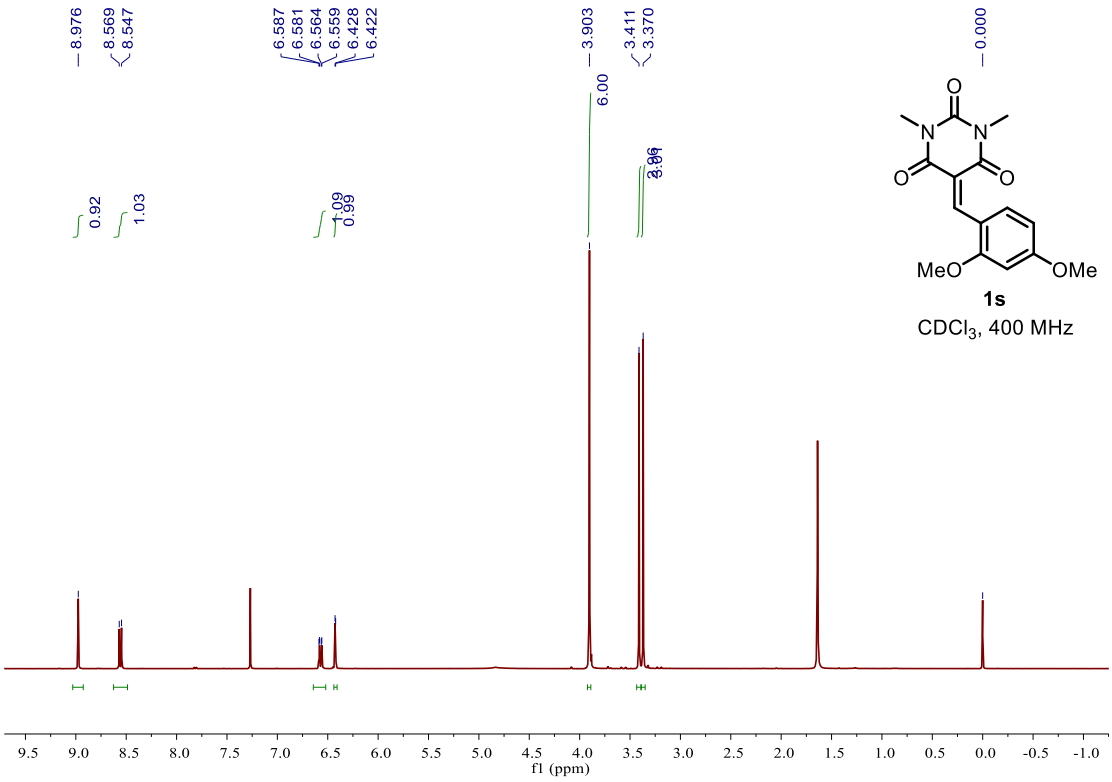

8.894
8.052
8.048
8.032
8.029
7.518
7.514
7.496
7.478
7.474
7.033
7.014
6.995
6.946
6.925
0.95
1.04
1.09
1.10
1.07
3.898
3.421
3.349
3.00
3.00
-0.000
MeO
1n
CDCl3, 400 MHz
f1 (ppm)


8.772
7.636
7.617
7.387
7.367
7.350
7.262
7.245
7.226
3.433
3.324
2.360
-0.000
0.93
0.99
1.01
2.31
3.03
2.99
3.00
O
N
N
O
O
Me
1o
CDCl3, 400 MHz
f1 (ppm)


8.648
7.685
7.682
7.666
7.661
7.641
7.638
7.401
7.399
7.382
7.380
7.363
7.361
7.341
7.337
7.322
7.304
7.299
3.441
3.323
0.000
0.91
2.01
3.00
3.02
O
N
N
O
O
Br
1p
CDCl3, 400 MHz
f1 (ppm)

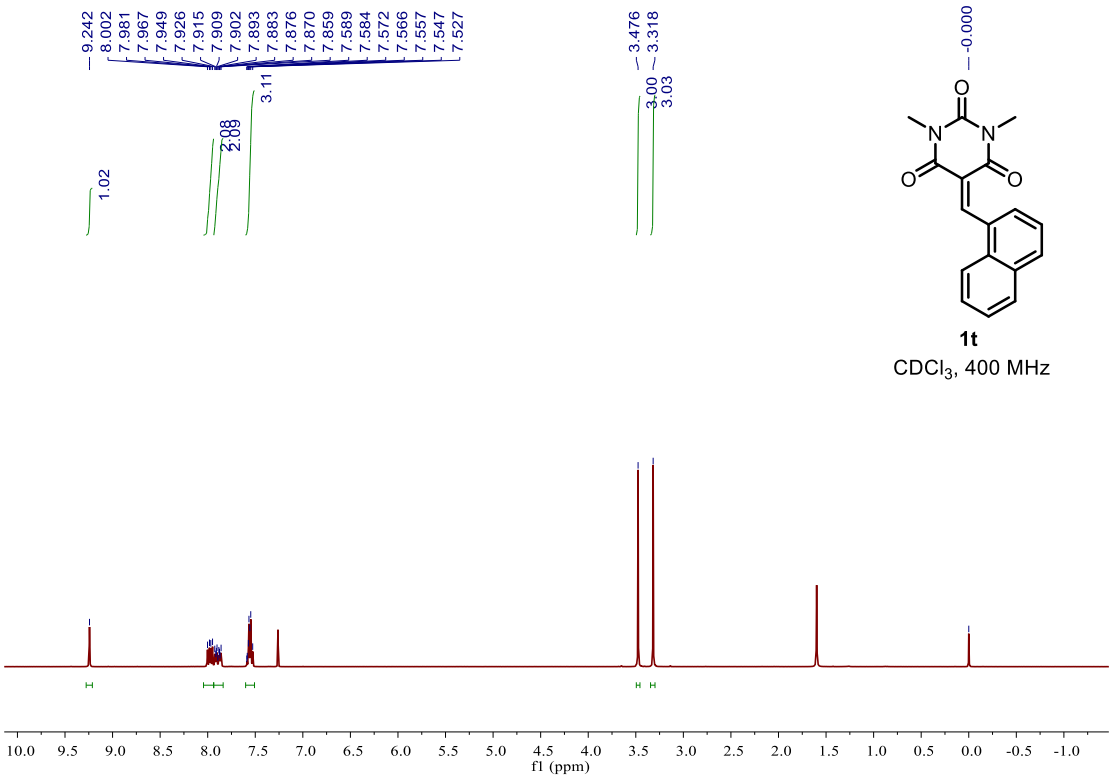

8.803
8.799
7.756
7.737
7.617
7.599
7.580
7.563
7.544
7.526
7.464
7.445
3.439
3.279
3.03
3.00
0.000
0.97
4.06
2.13
1.02
O
N
N
O
O
F3C
1q
CDCl3, 400 MHz
f1 (ppm)


8.894
8.314
8.312
8.294
8.291
7.745
7.742
7.726
7.724
7.707
7.705
7.646
7.645
7.626
7.607
7.606
7.401
7.382
3.446
3.247
3.00
3.02
0.000
0.92
0.99
1.06
1.05
1.03
O
N
N
O
O
O2N
1r
CDCl3, 400 MHz
f1 (ppm)



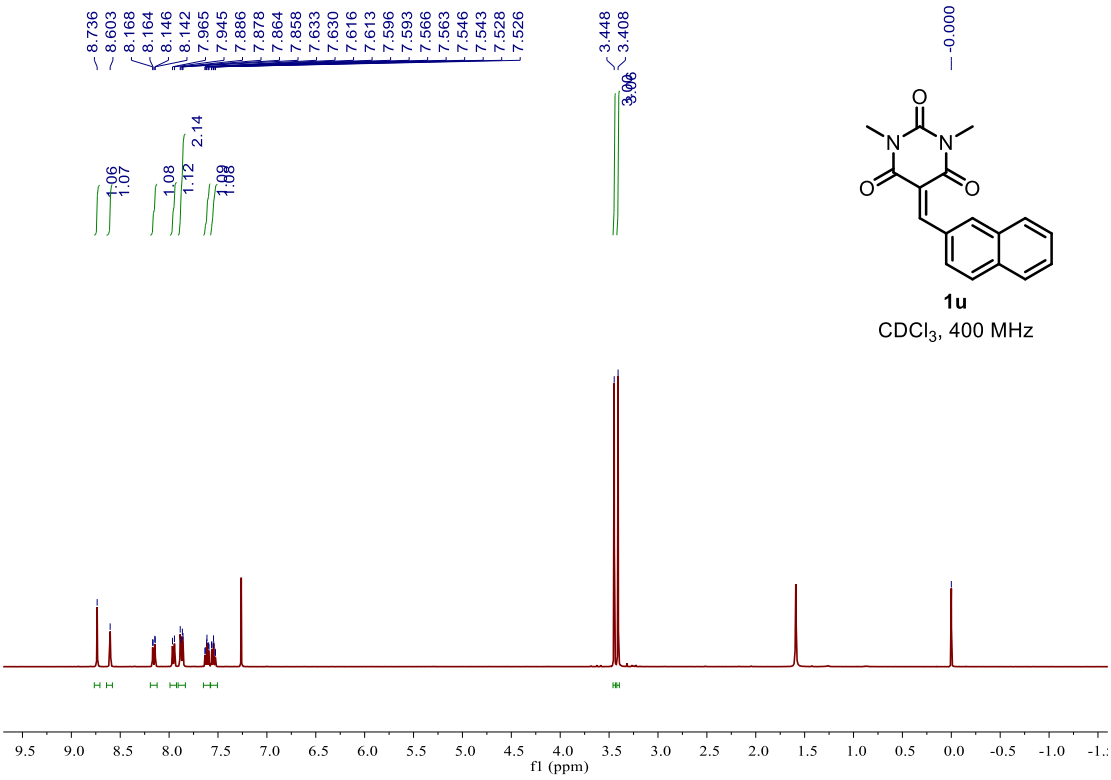
1s

$CDCl_3$, 400 MHz



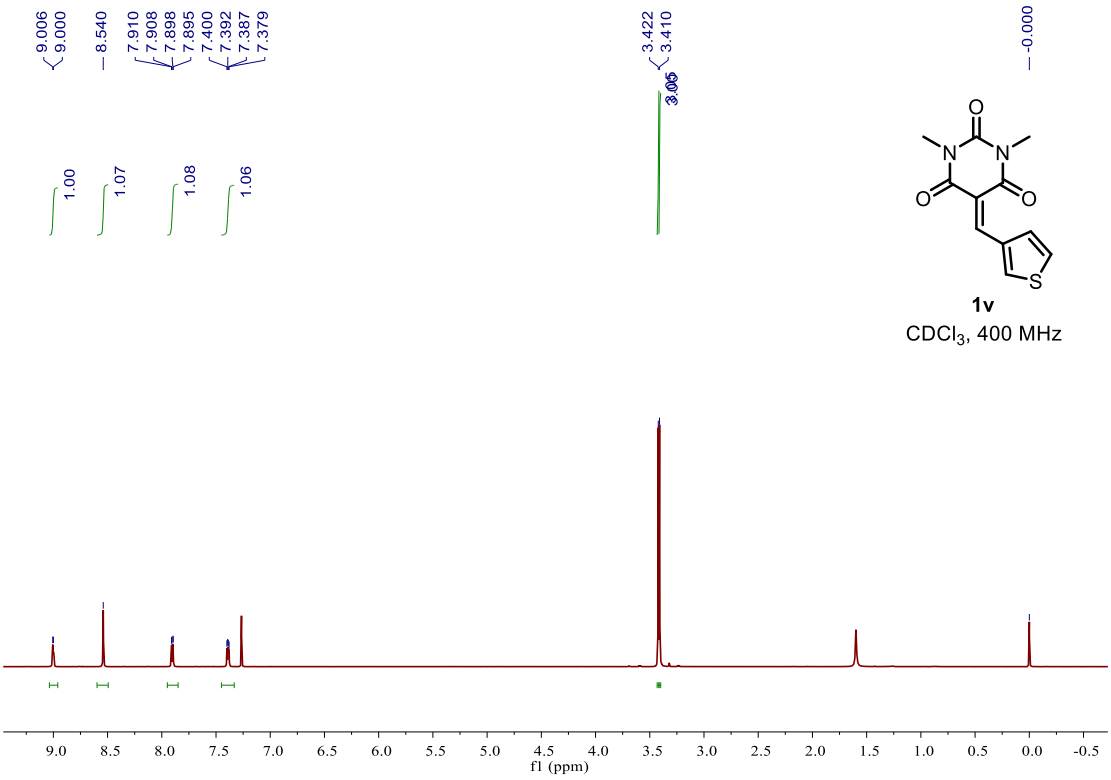
1t

$CDCl_3$, 400 MHz



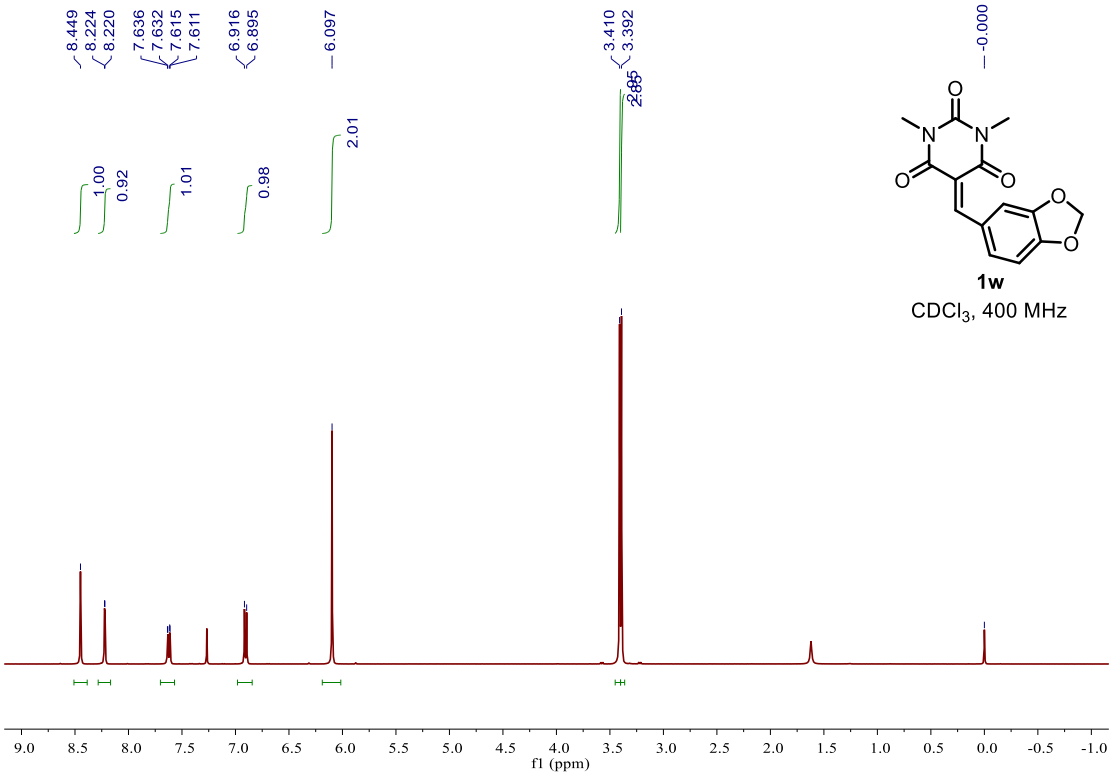
1u

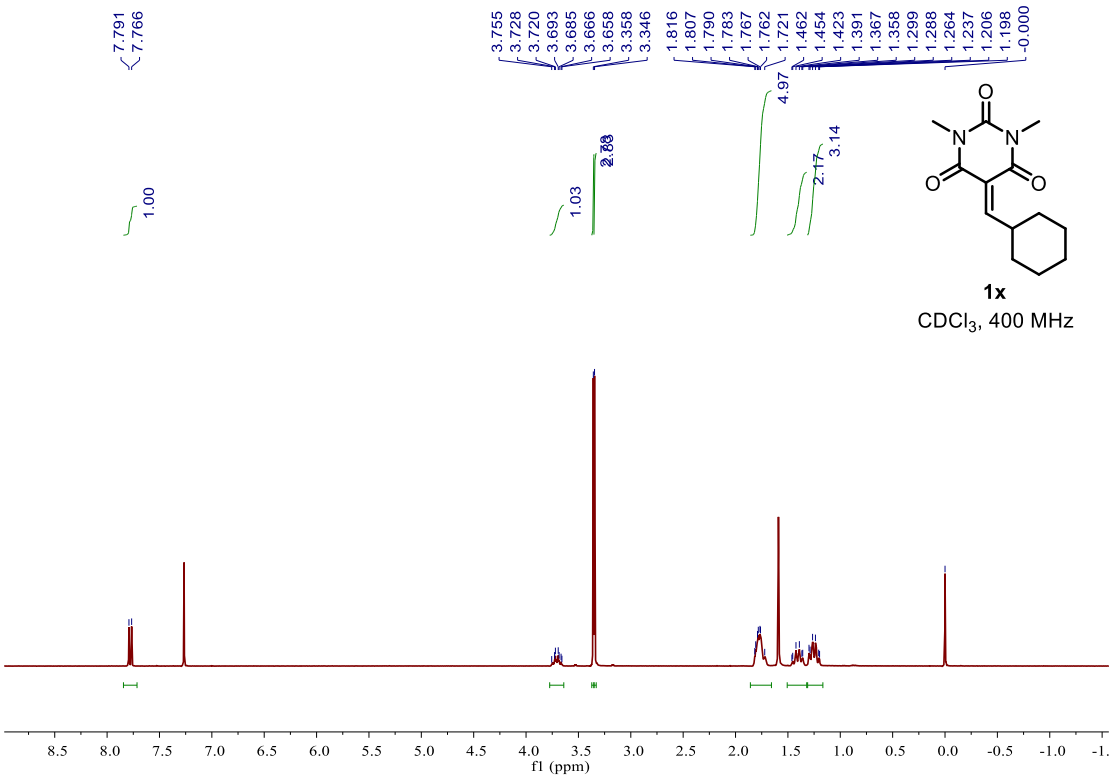
$CDCl_3$, 400 MHz

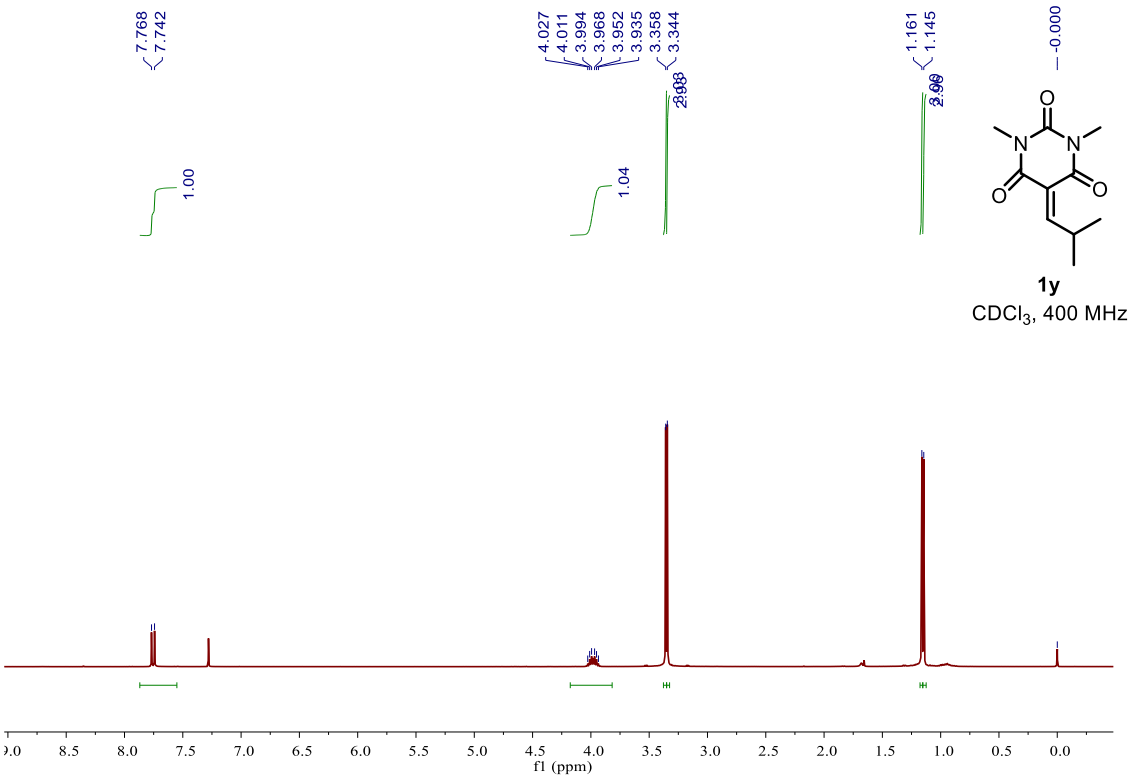


1v

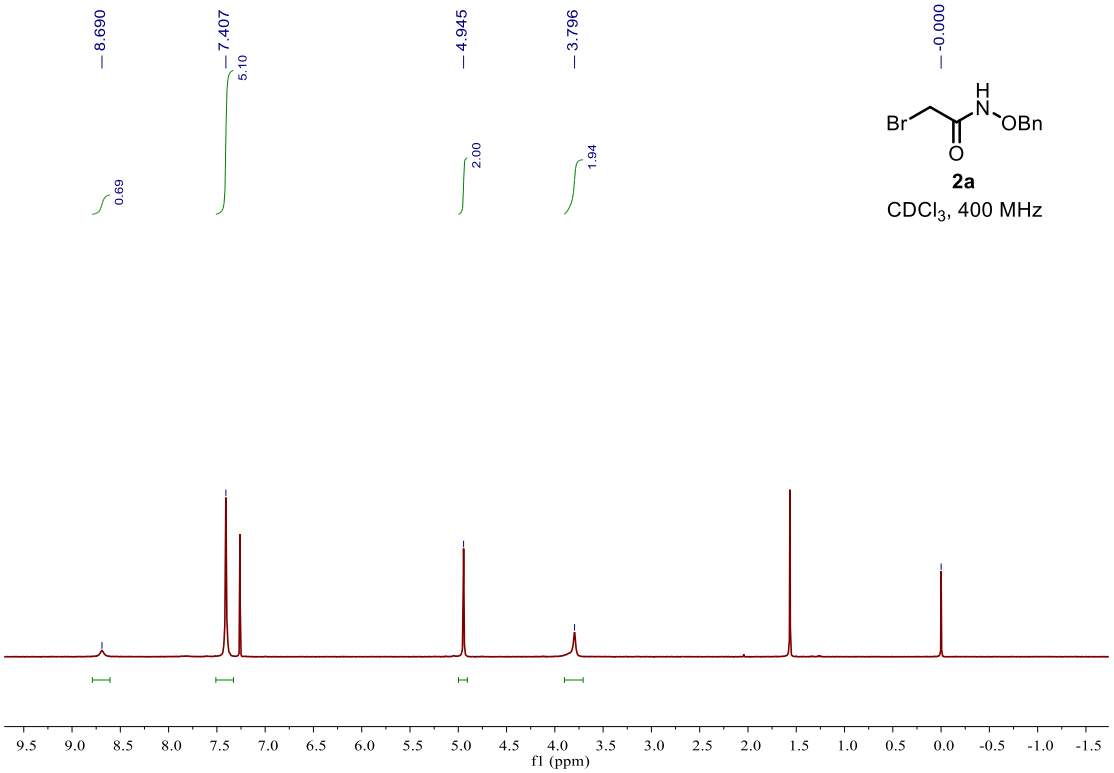
$CDCl_3$, 400 MHz

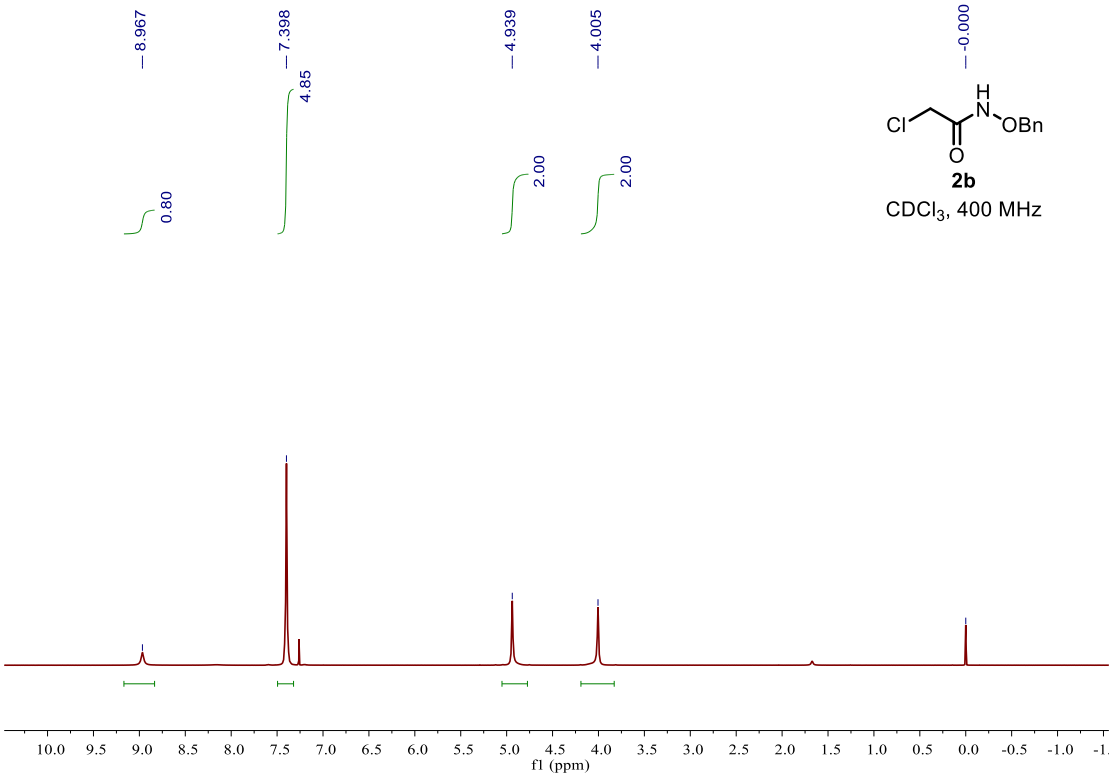

8.449
8.224
8.220
7.636
7.632
7.615
7.611
6.916
6.895
6.097
3.410
3.392
-0.000
1.00
0.92
1.01
0.98
2.01
1w
CDCl3, 400 MHz
f1 (ppm)

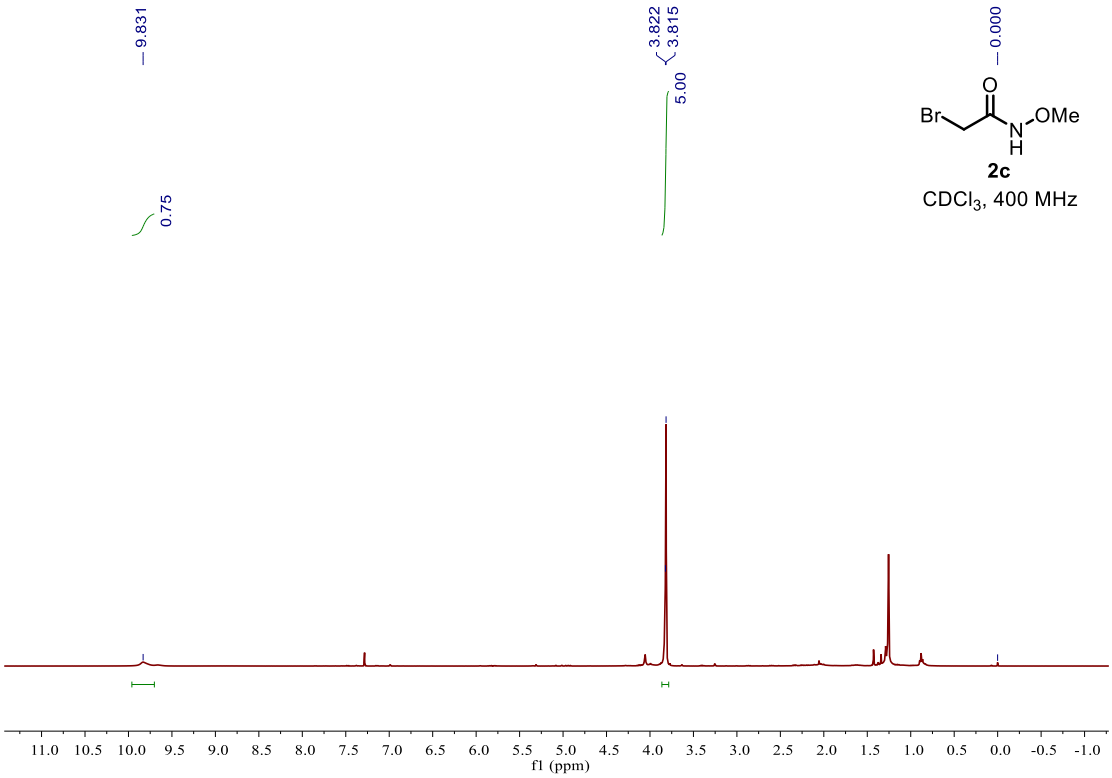

7.791
7.766
3.755
3.728
3.720
3.693
3.685
3.666
3.658
3.358
3.346
1.816
1.807
1.790
1.783
1.767
1.762
1.721
1.462
1.454
1.423
1.391
1.367
1.358
1.299
1.288
1.264
1.237
1.206
1.198
-0.000
1.00
1.03
4.97
2.17
3.14
1x
CDCl3, 400 MHz
f1 (ppm)

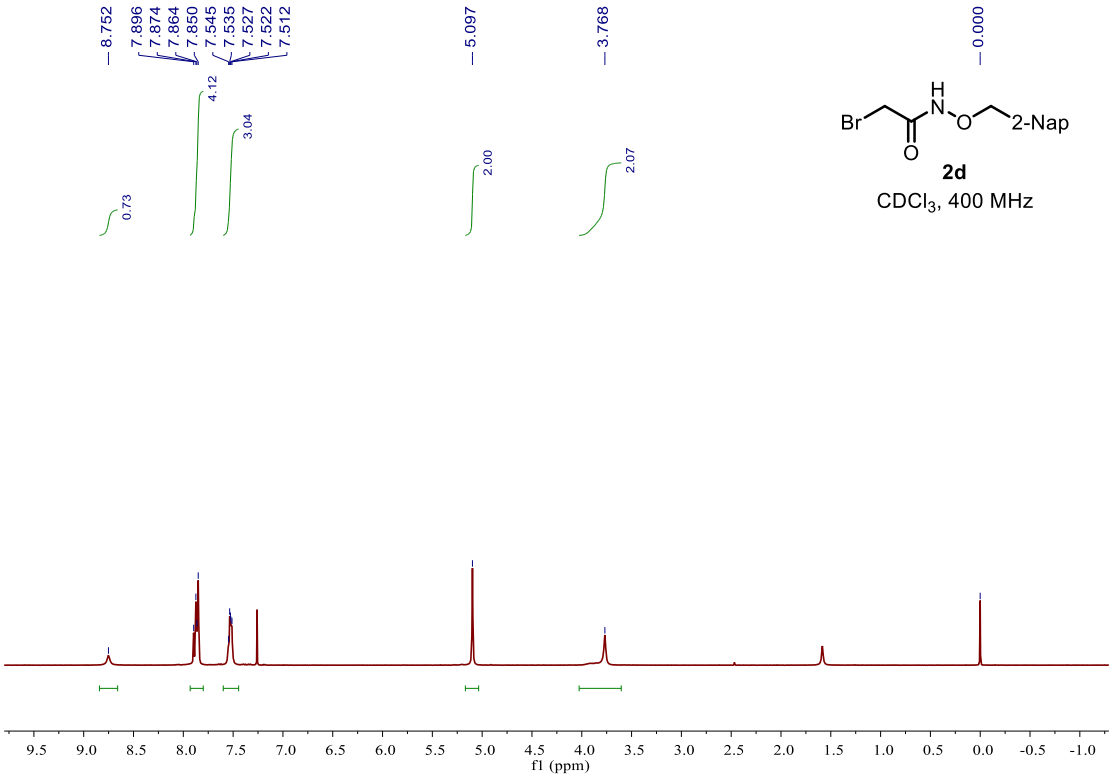

7.768
7.742
4.027
4.011
3.994
3.968
3.952
3.935
3.358
3.344
1.161
1.145
-0.000
1.00
1.04
1y
CDCl3, 400 MHz
f1 (ppm)

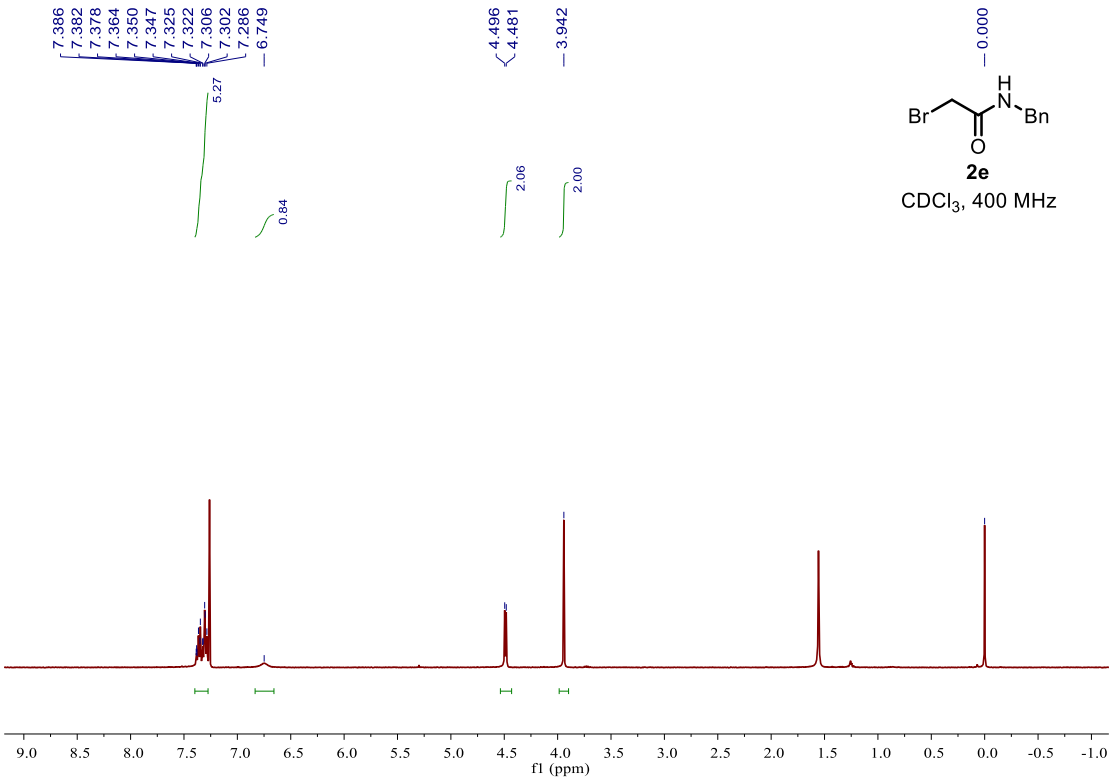
6. Copies of NMR Spectras of *N*-Alkoxy *α*-Haloamides 2

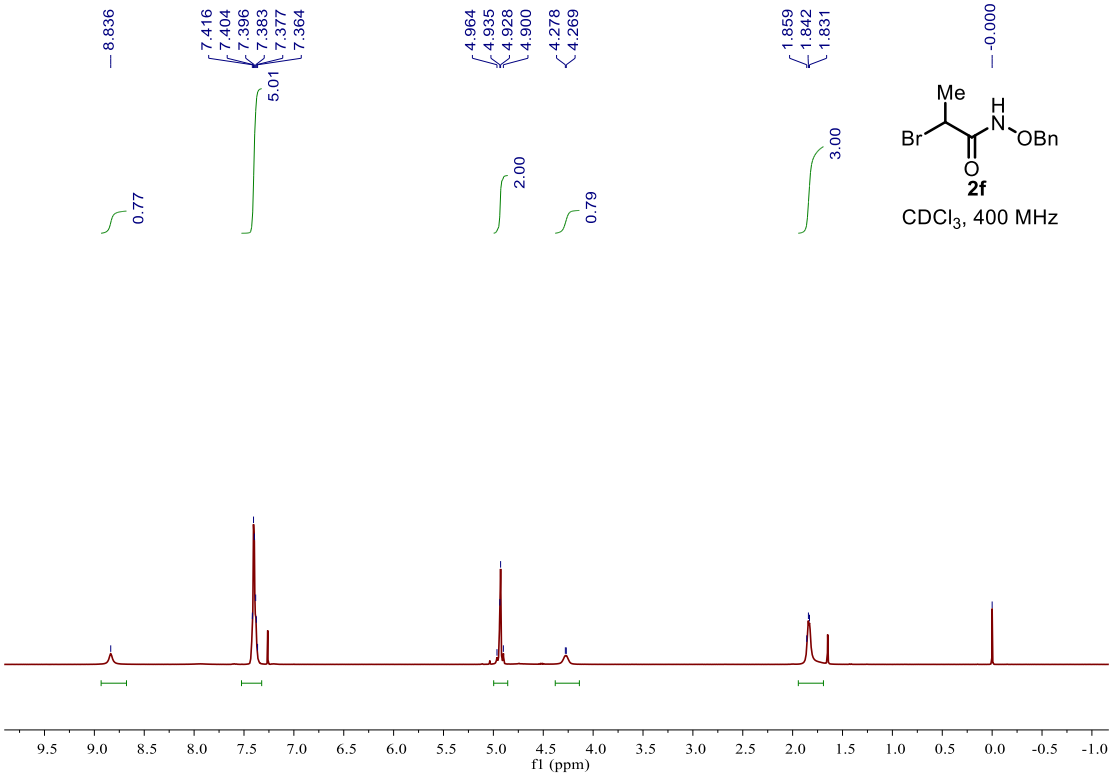


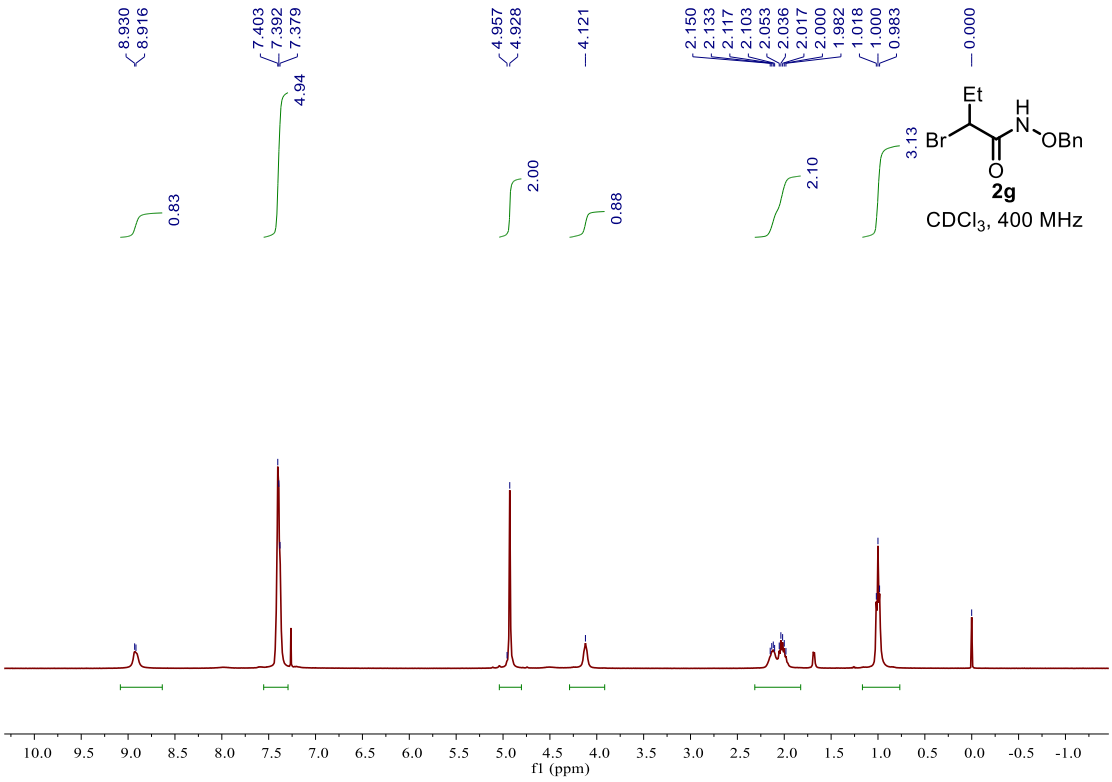


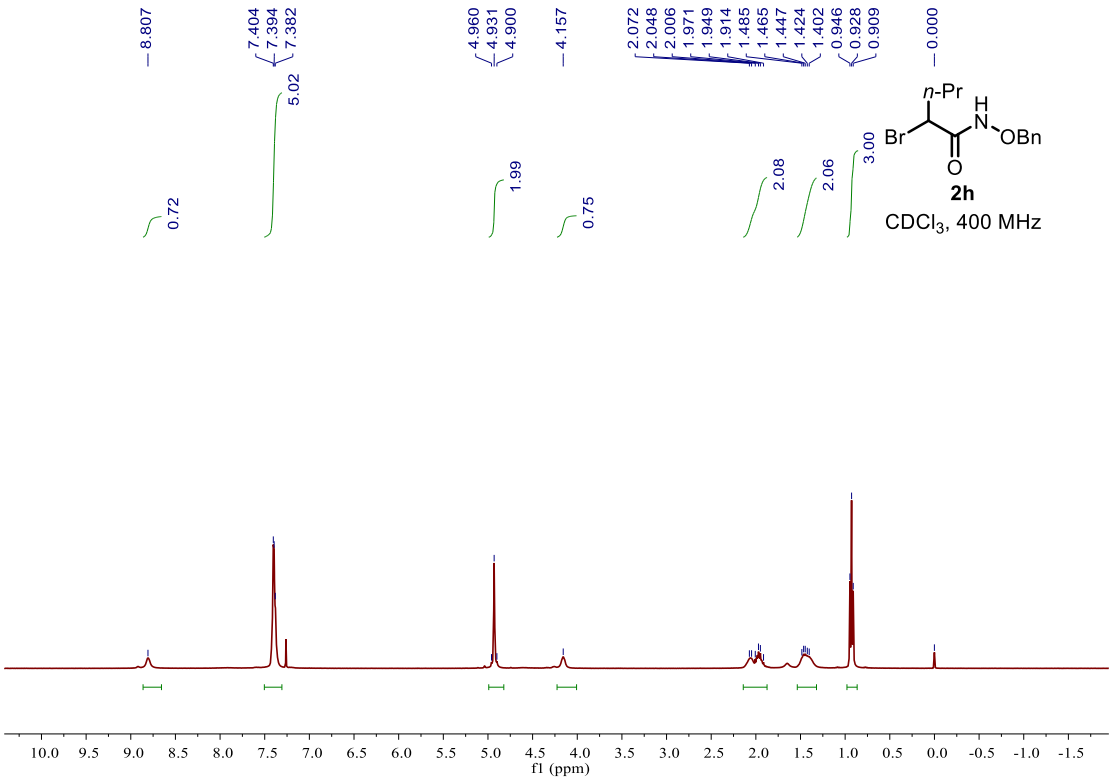

9.831
3.822
3.815
0.000
0.75
5.00
Br
O
N
H
OMe
2c
CDCl3, 400 MHz
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

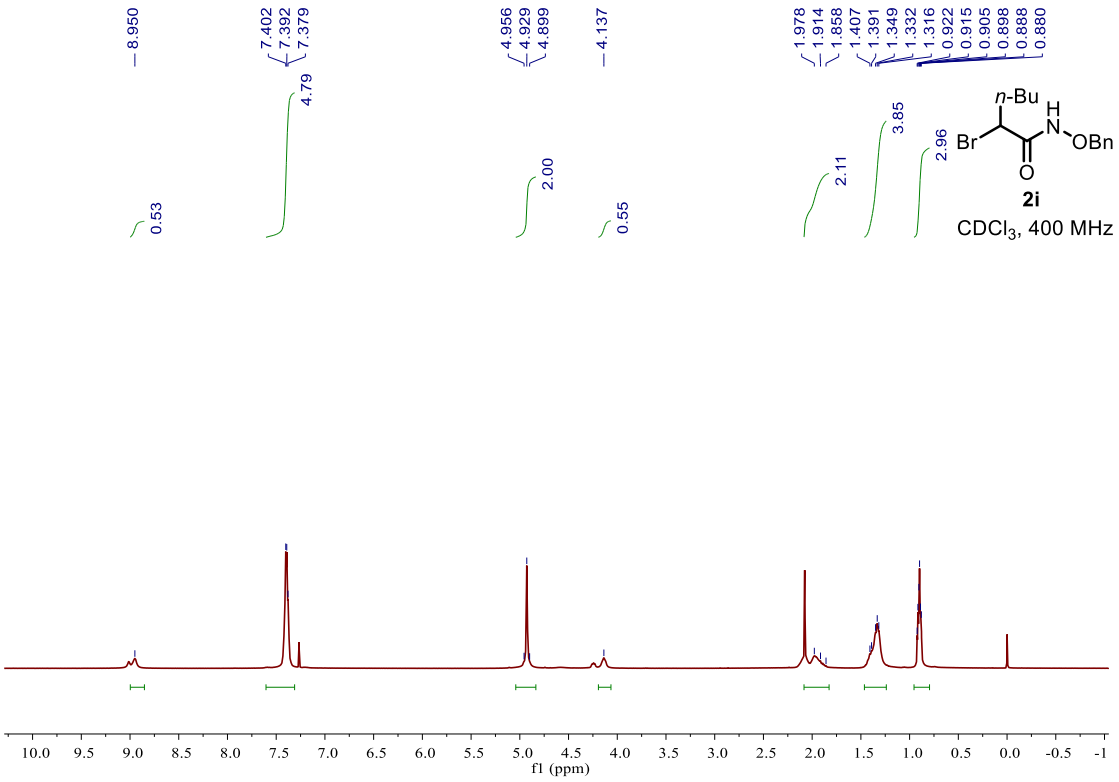

8.752
7.896
7.874
7.864
7.850
7.545
7.535
7.527
7.522
7.512
5.097
3.768
0.000
0.73
4.12
3.04
2.00
2.07
Br
H
N
O
O
2-Nap
2d
CDCl3, 400 MHz
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

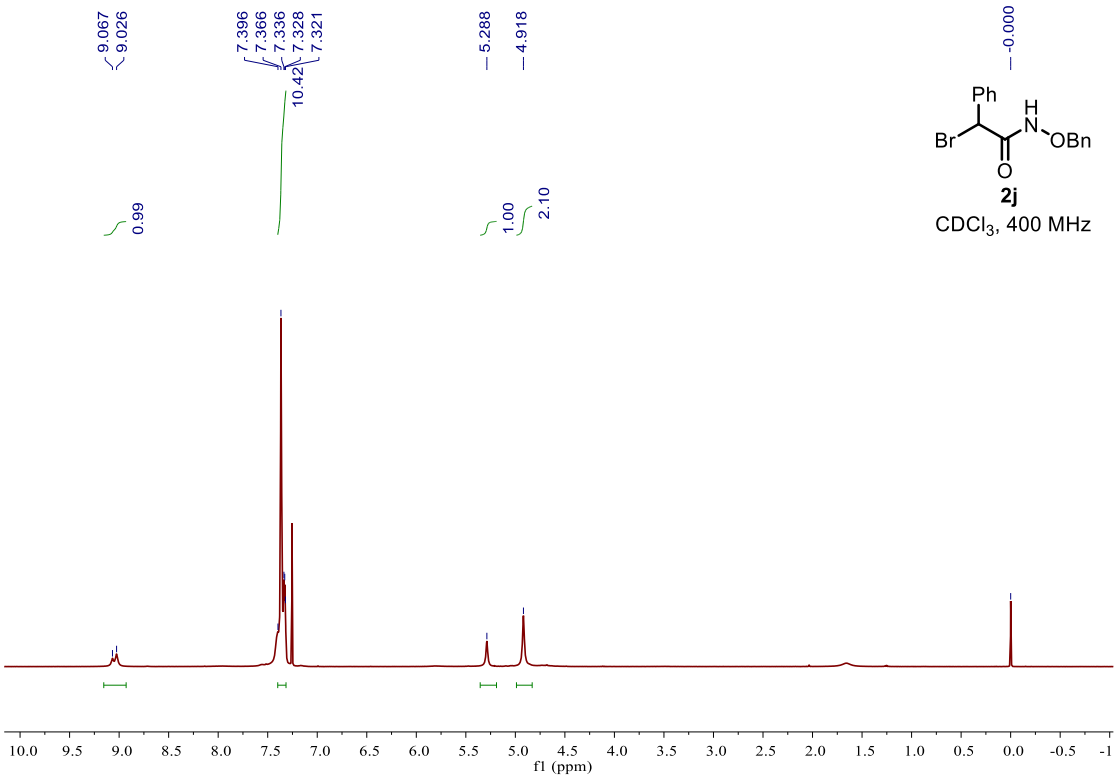

7.386
7.382
7.378
7.364
7.350
7.347
7.325
7.322
7.306
7.302
7.286
6.749
5.27
0.84
4.496
4.481
3.942
2.06
2.00
0.000
Br
N
H
Bn
O
2e
CDCl3, 400 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

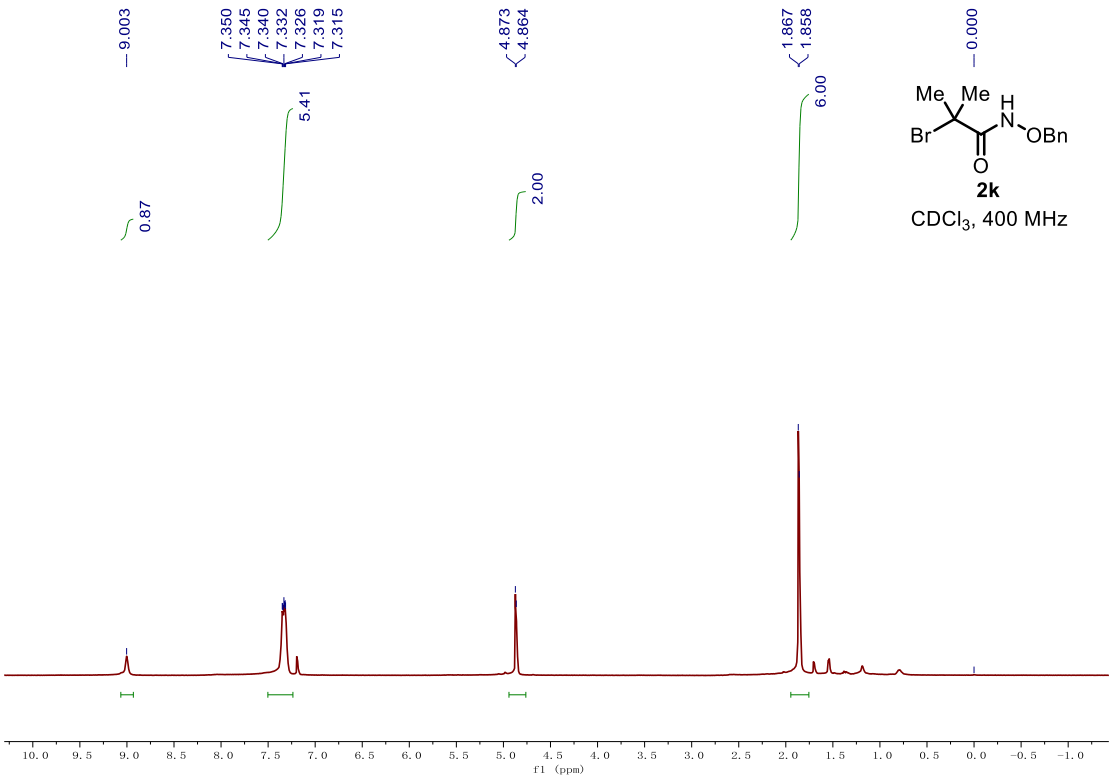

8.836
7.416
7.404
7.396
7.383
7.377
7.364
5.01
0.77
4.964
4.935
4.928
4.900
4.278
4.269
2.00
0.79
1.859
1.842
1.831
3.00
-0.000
Me
Br
N
H
OBn
O
2f
CDCl3, 400 MHz
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


8.930
8.916
7.403
7.392
7.379
4.957
4.928
4.121
2.150
2.133
2.117
2.103
2.053
2.036
2.017
2.000
1.982
1.018
1.000
0.983
0.000
0.83
4.94
2.00
0.88
2.10
3.13
Et
H
N
Br
OBn
O
2g
CDCl3, 400 MHz
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

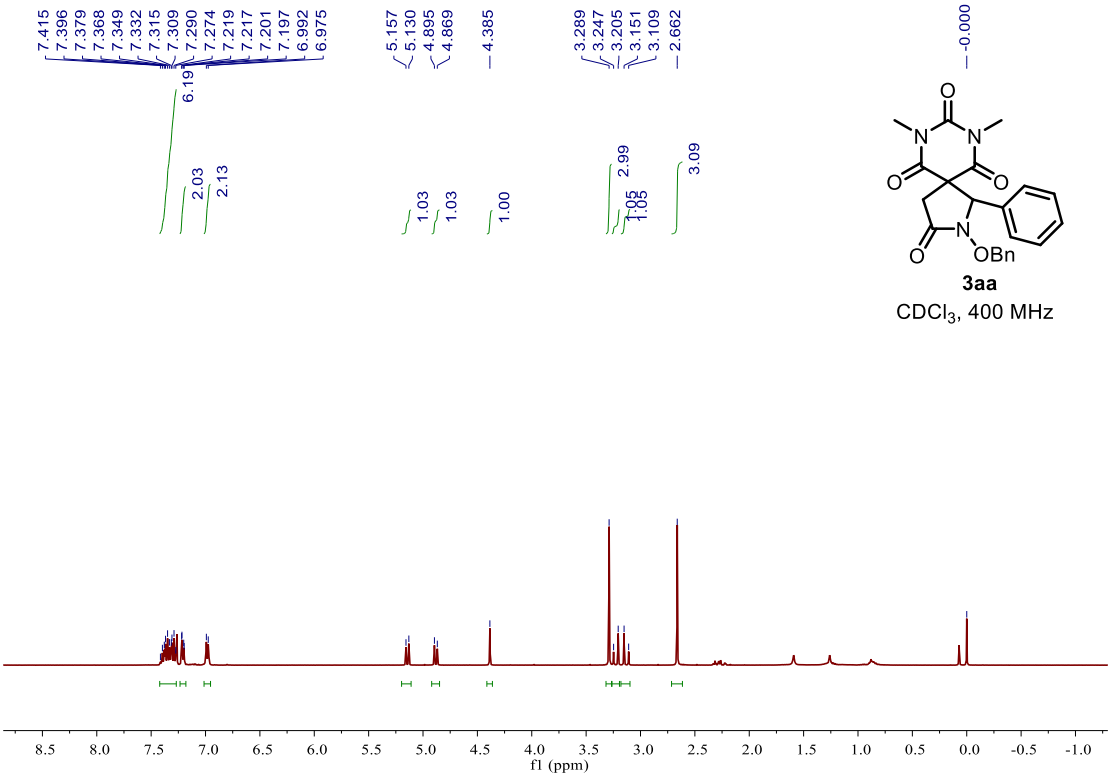

8.807
7.404
7.394
7.382
4.960
4.931
4.900
4.157
2.072
2.048
2.006
1.971
1.949
1.914
1.485
1.465
1.447
1.424
1.402
0.946
0.928
0.909
0.000
0.72
5.02
1.99
0.75
2.08
2.06
3.00
n-Pr
H
N
Br
OBn
O
2h
CDCl3, 400 MHz
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

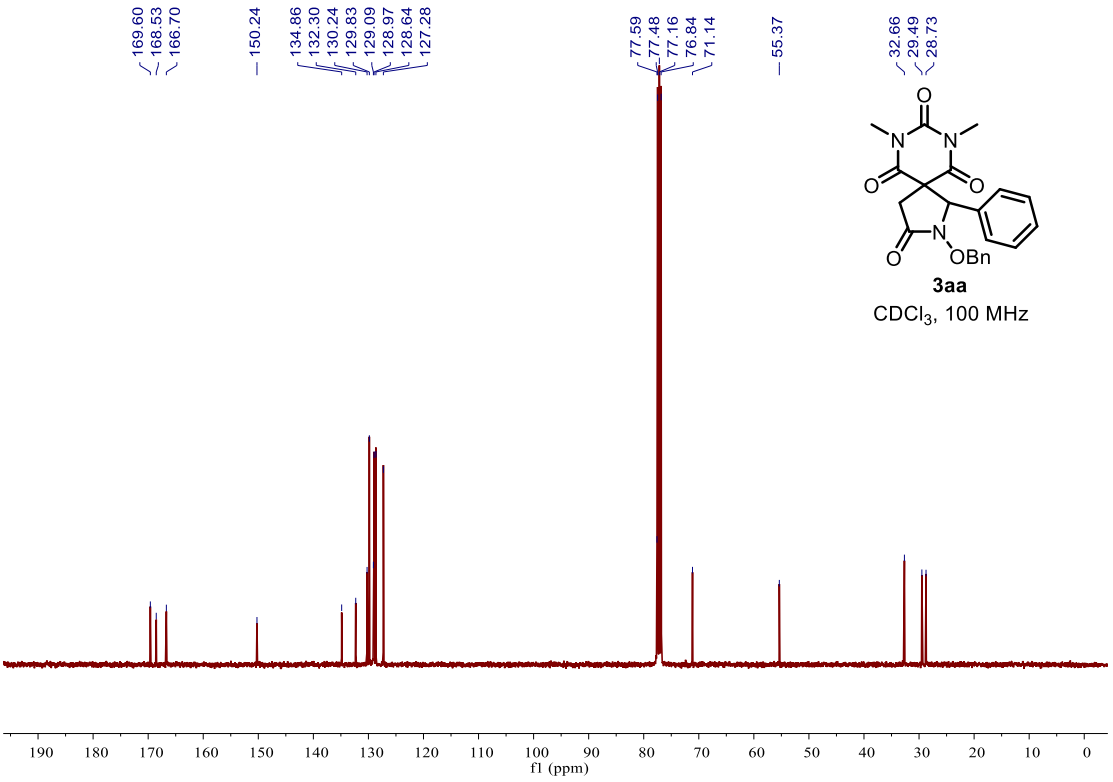

8.950
7.402
7.392
7.379
4.956
4.929
4.899
4.137
1.978
1.914
1.858
1.407
1.391
1.349
1.332
1.316
0.922
0.915
0.905
0.898
0.888
0.880
0.53
4.79
2.00
0.55
2.11
3.85
2.96
n-Bu
Br
H
N
OBn
O
2i
CDCl3, 400 MHz
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

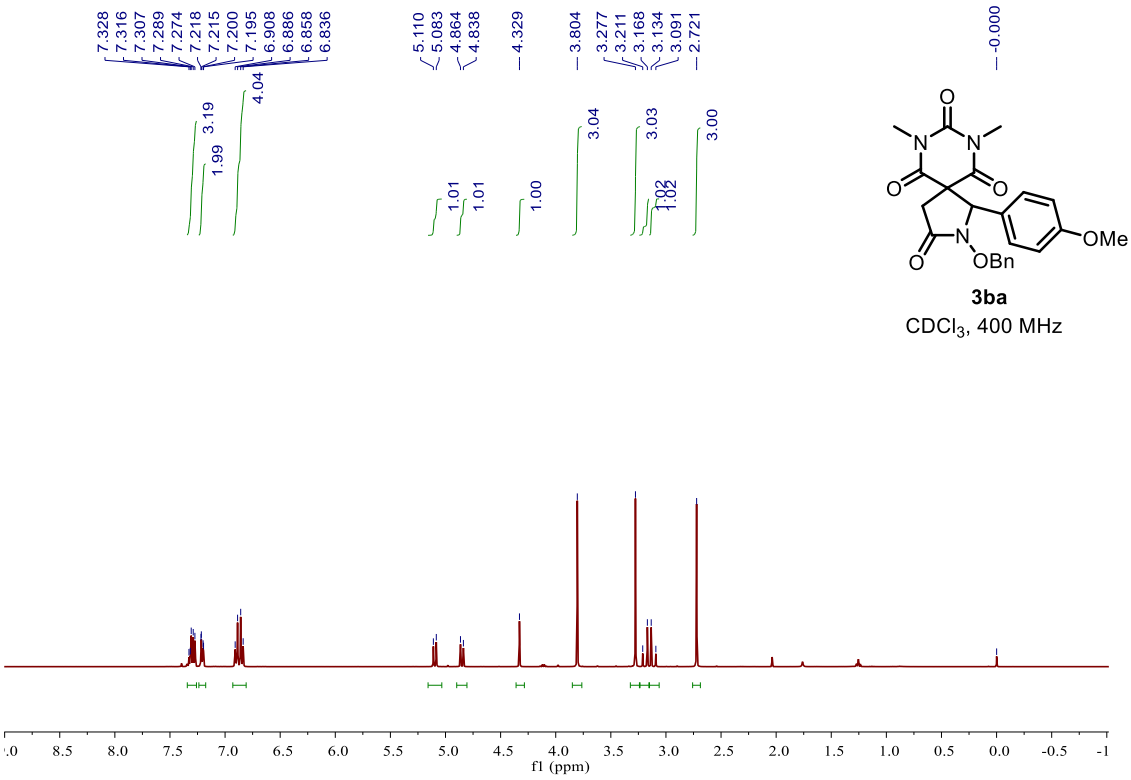

9.067
9.026
7.396
7.366
7.336
7.328
7.321
5.288
4.918
-0.000
0.99
10.42
1.00
2.10
Ph
Br
H
N
OBn
O
2j
CDCl3, 400 MHz
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)


9.003
7.350
7.345
7.340
7.332
7.326
7.319
7.315
4.873
4.864
1.867
1.858
0.000
0.87
5.41
2.00
6.00
Me
Me
H
N
Br
OBn
O
2k
CDCl3, 400 MHz
f1 (ppm)

7. Copies of NMR spectras of adducts 3, 4 and 5

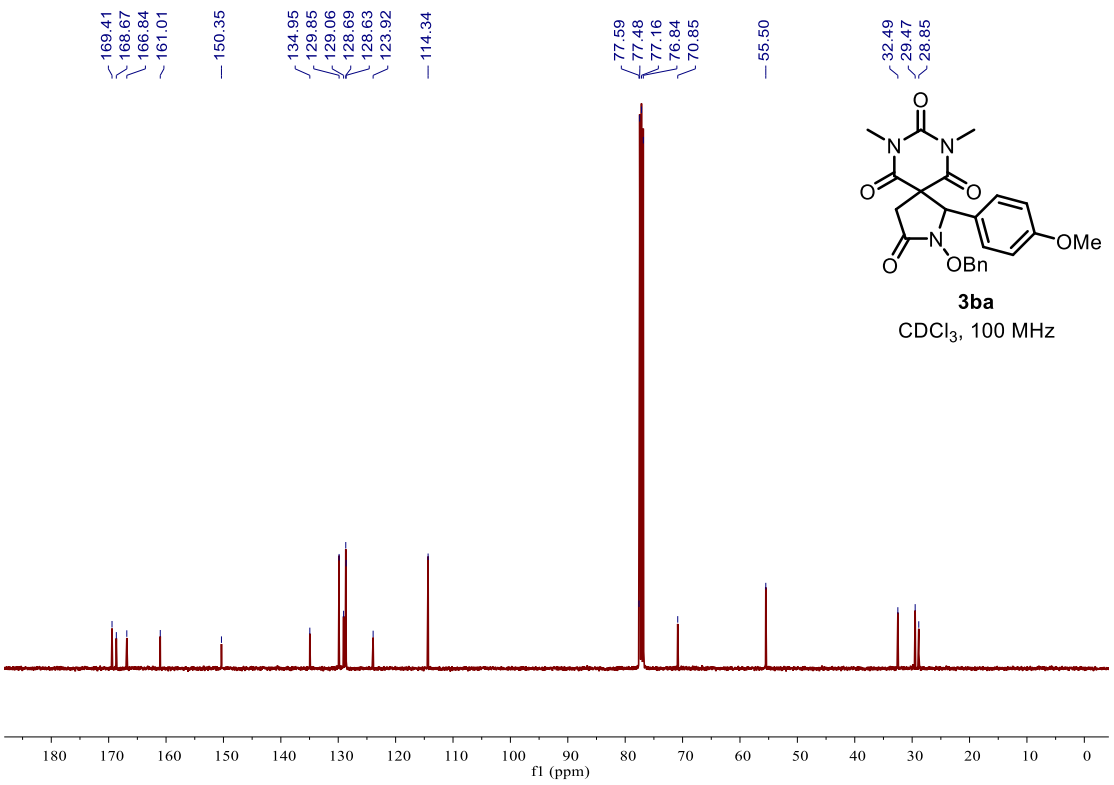






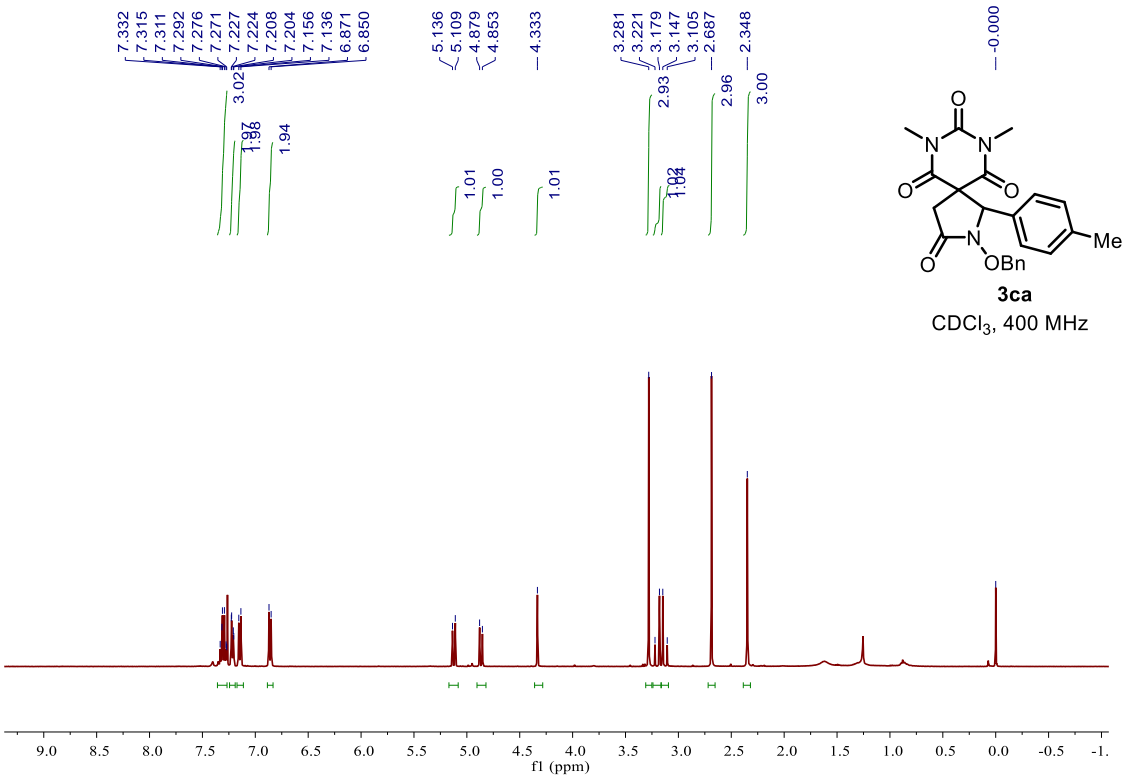
3ba

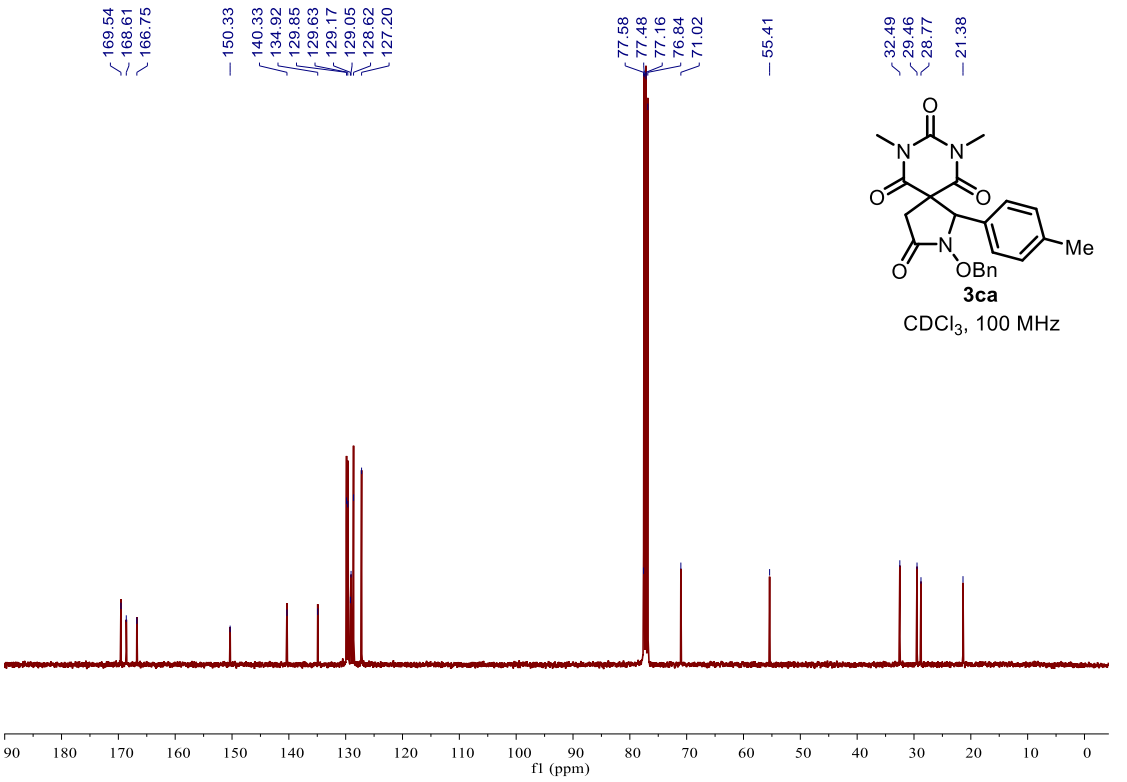
$CDCl_3$, 400 MHz

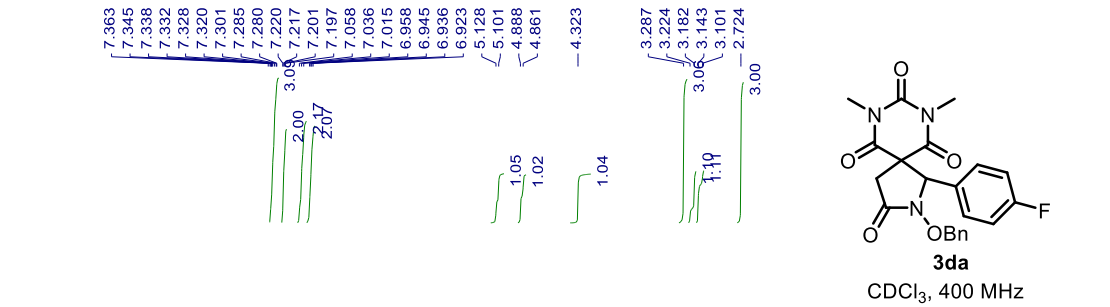


3ba

$CDCl_3$, 100 MHz

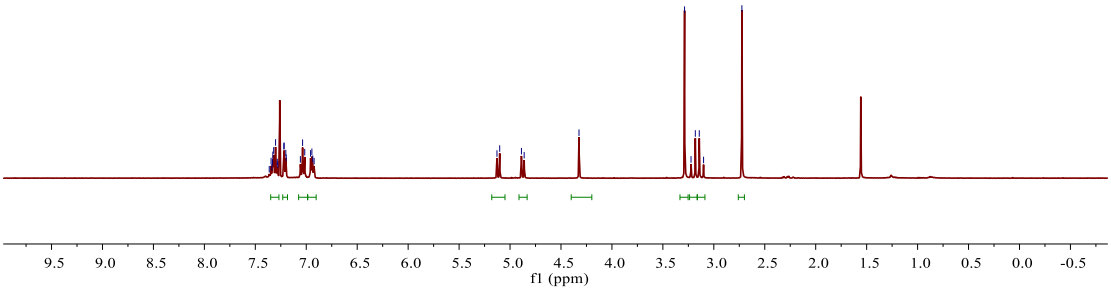

7.332
7.315
7.311
7.292
7.276
7.271
7.227
7.224
7.208
7.204
7.156
7.136
6.871
6.850
5.136
5.109
4.879
4.853
4.333
3.281
3.221
3.179
3.147
3.105
2.687
2.348
-0.000
3.02
1.97
1.98
1.94
1.01
1.00
1.01
2.93
1.02
1.04
2.96
3.00
Me
OBn
3ca
CDCl3, 400 MHz
f1 (ppm)

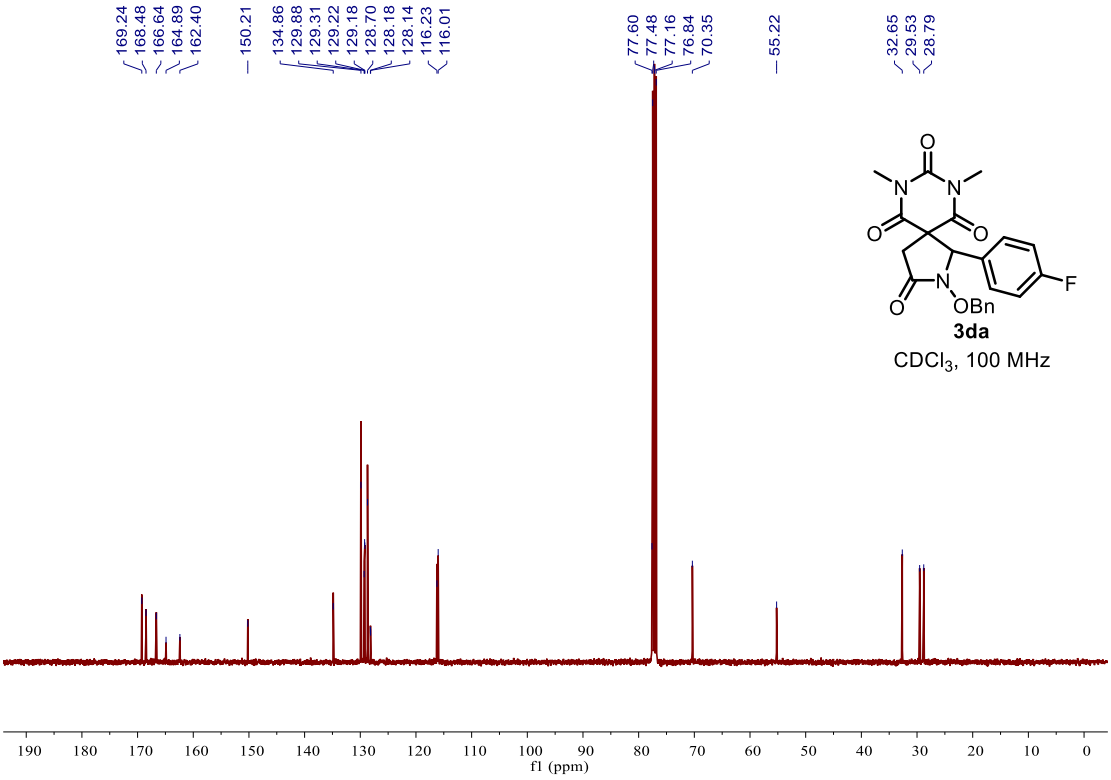

169.54
168.61
166.75
150.33
140.33
134.92
129.85
129.63
129.17
129.05
128.62
127.20
77.58
77.48
77.16
76.84
71.02
55.41
32.49
29.46
28.77
21.38
Me
OBn
3ca
CDCl3, 100 MHz
f1 (ppm)



3da

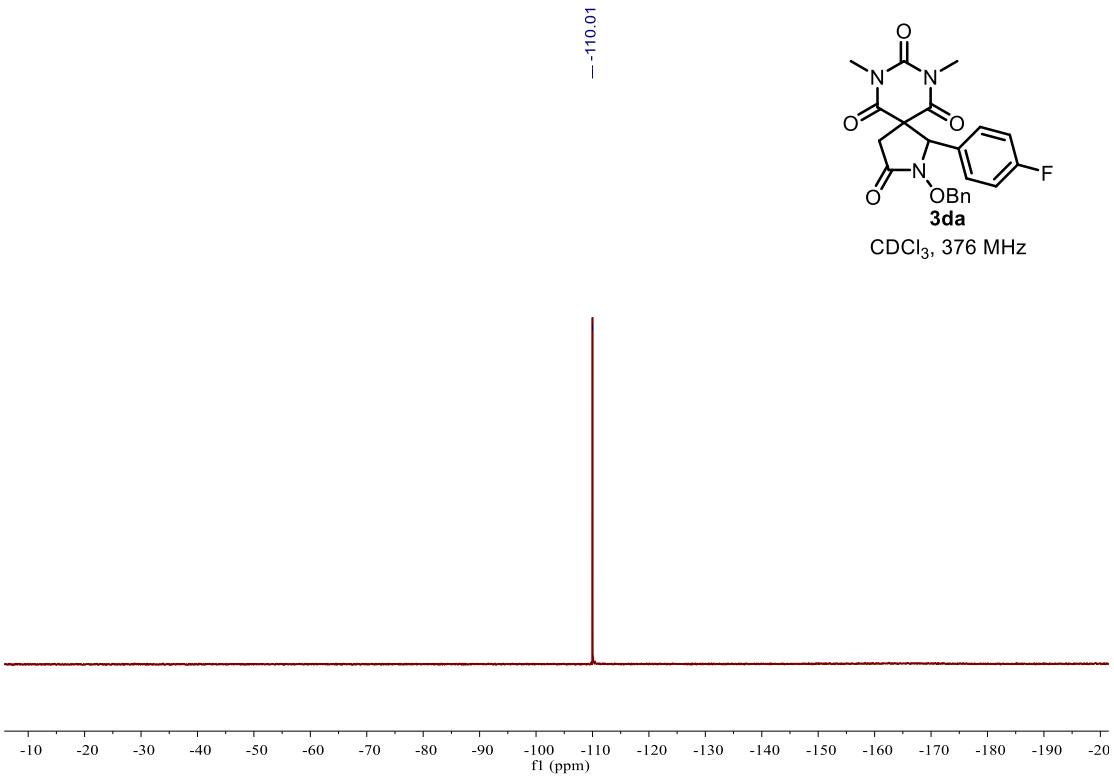
$CDCl_3$, 400 MHz

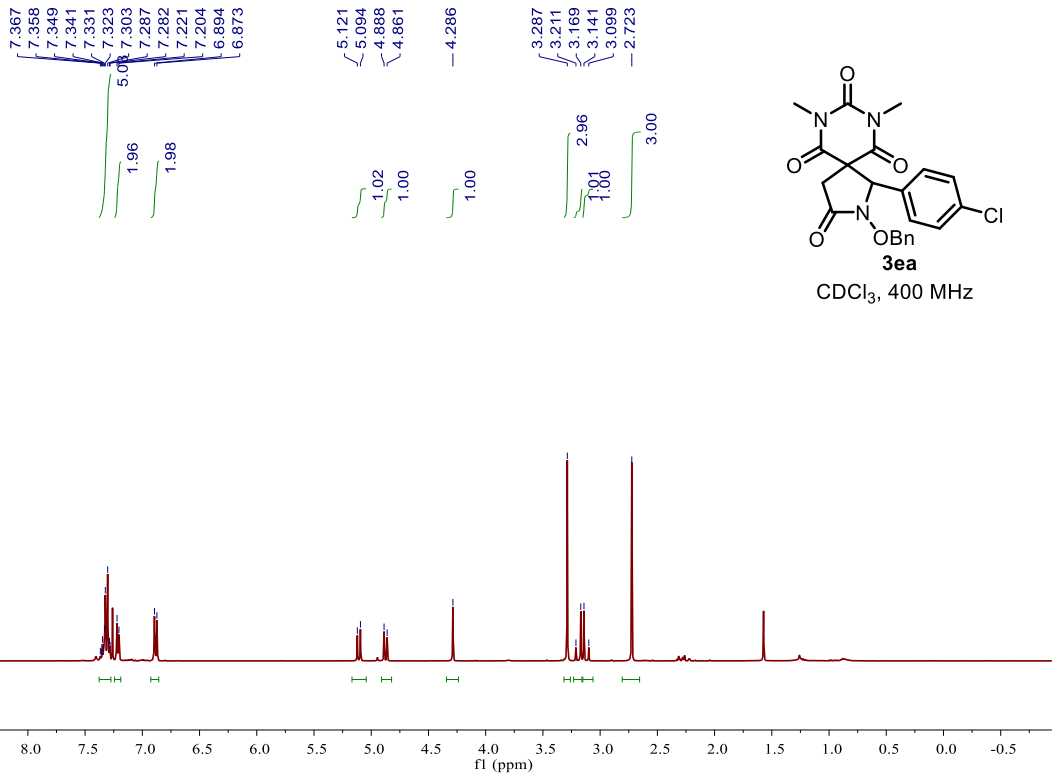


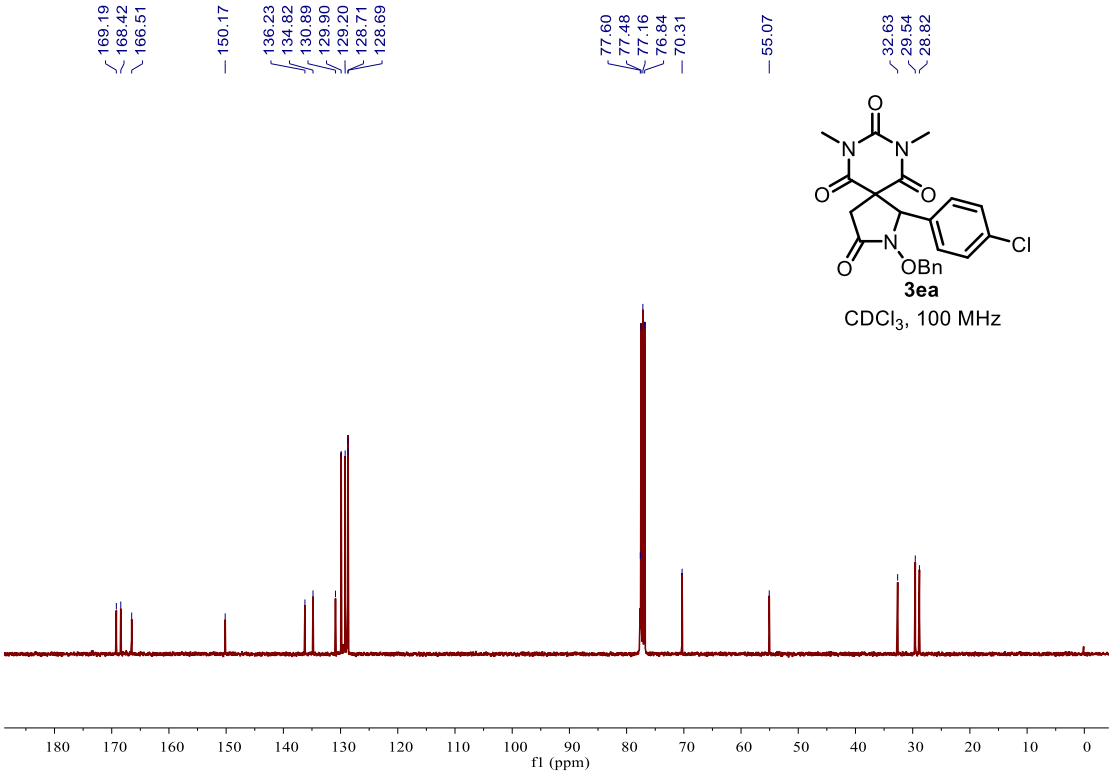


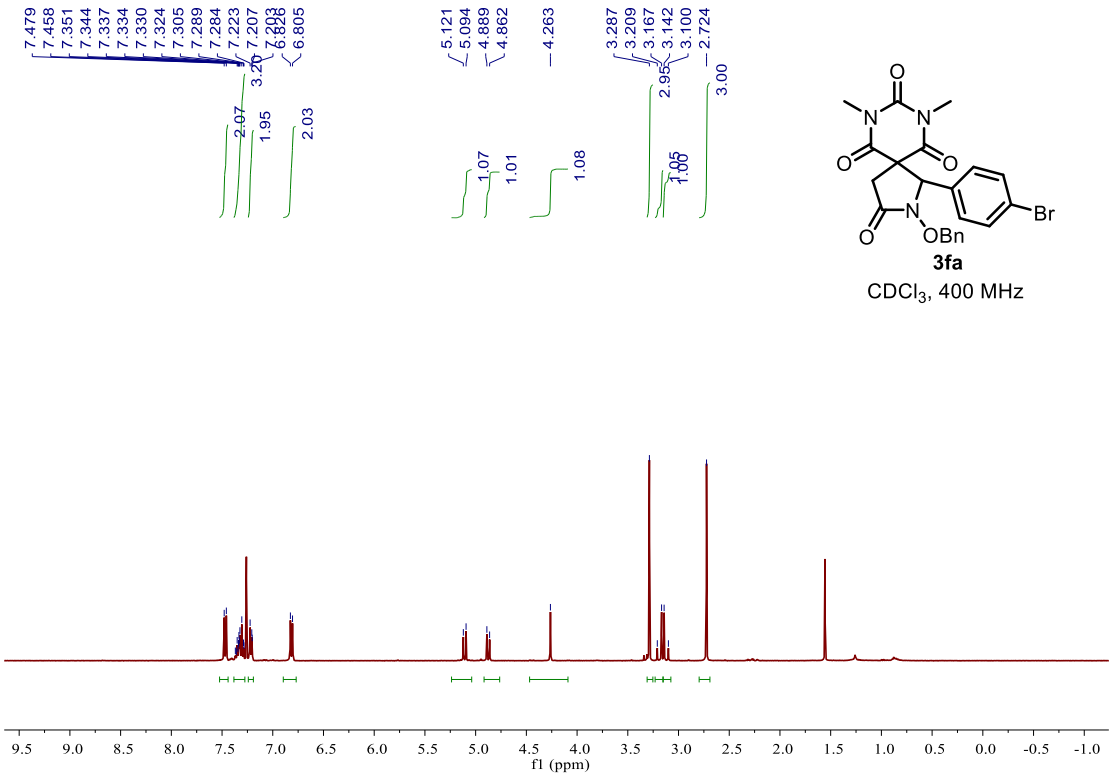
3da

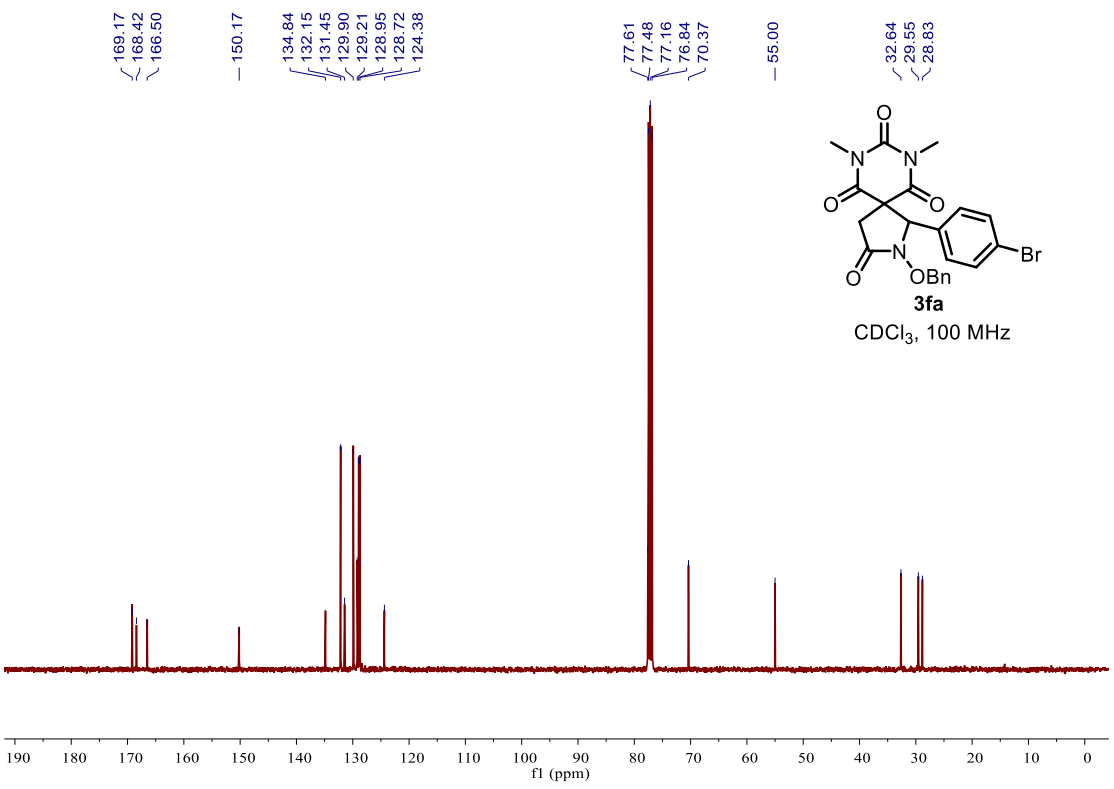
$CDCl_3$, 100 MHz

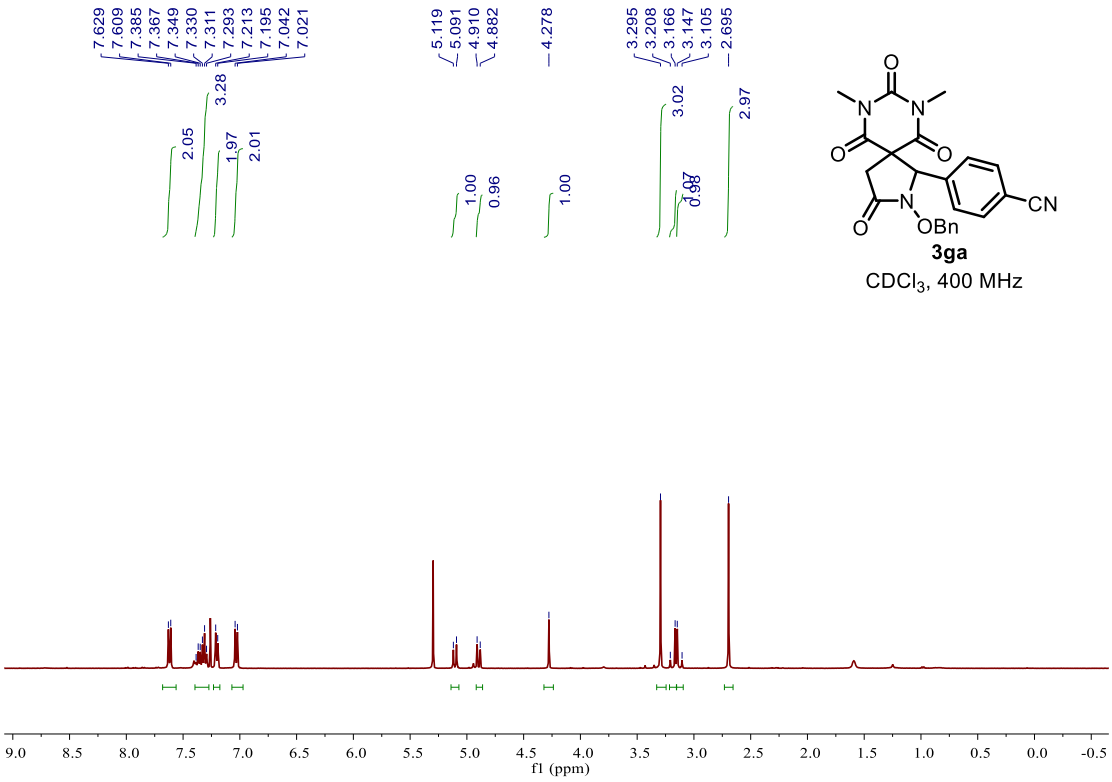

-110.01
O
N
N
O
O
O
N
OBn
F
3da
CDCl3, 376 MHz
-10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -20
f1 (ppm)


7.367
7.358
7.349
7.341
7.331
7.323
7.303
7.287
7.282
7.221
7.204
6.894
6.873
5.121
5.094
4.888
4.861
4.286
3.287
3.211
3.169
3.141
3.099
2.723
5.00
1.96
1.98
1.02
1.00
1.00
2.96
1.01
1.00
3.00
O
N
N
O
O
N
OBn
O
Cl
3ea
CDCl3, 400 MHz
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

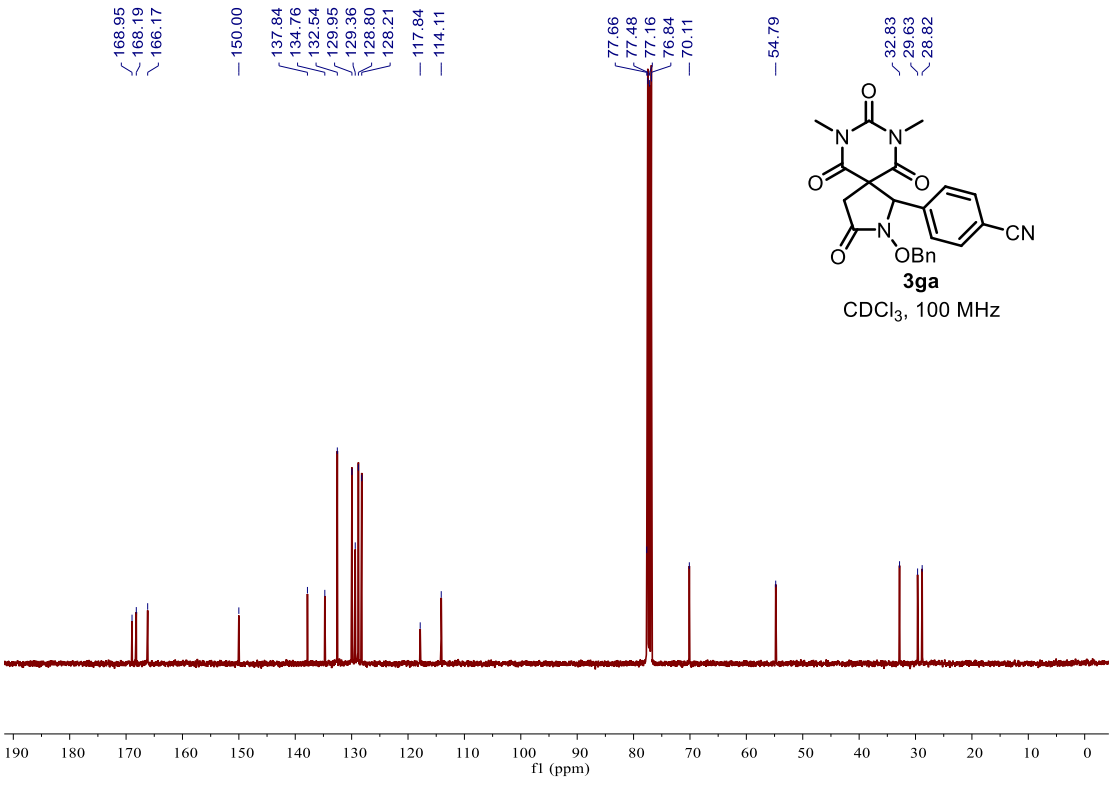

169.19
168.42
166.51
150.17
136.23
134.82
130.89
129.90
129.20
128.71
128.69
77.60
77.48
77.16
76.84
70.31
55.07
32.63
29.54
28.82
O
N
N
O
O
N
OBn
O
Cl
3ea
CDCl3, 100 MHz
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

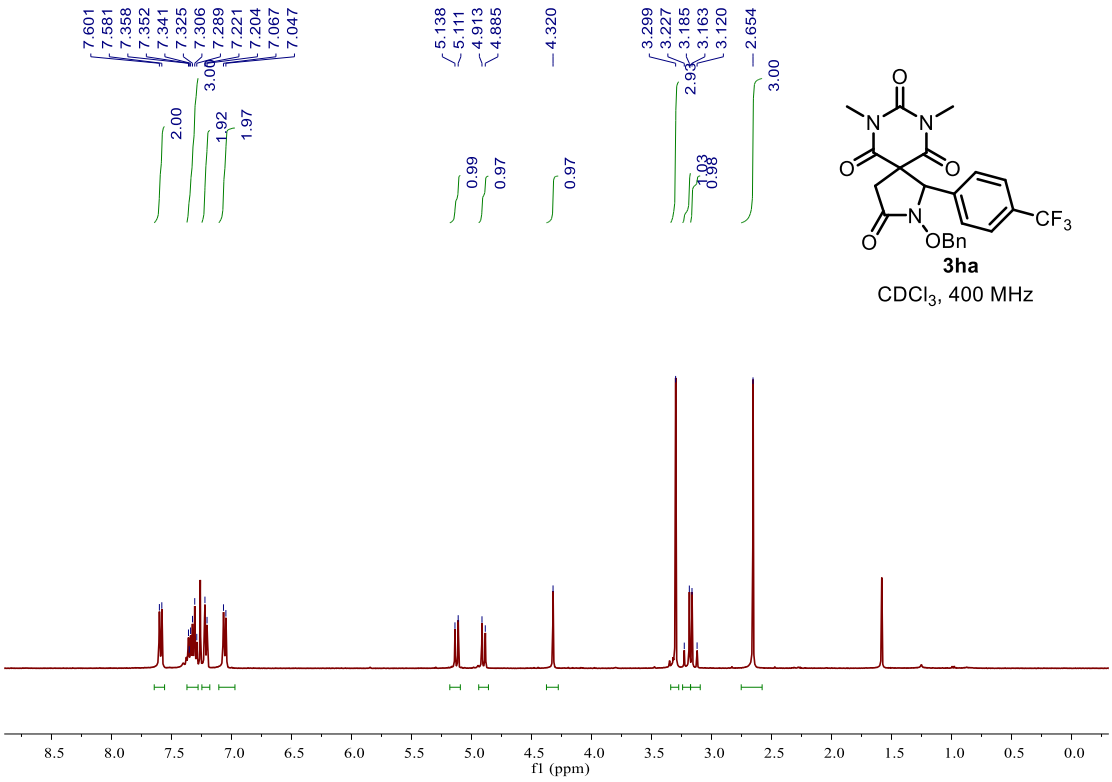

7.479
7.458
7.351
7.344
7.337
7.334
7.330
7.324
7.305
7.289
7.284
7.223
7.207
6.828
6.805
5.121
5.094
4.889
4.862
4.263
3.287
3.209
3.167
3.142
3.100
2.724
3.20
2.07
1.95
2.03
1.07
1.01
1.08
2.95
1.05
1.00
3.00
O
N
O
N
O
O
N
OBn
Br
3fa
CDCl3, 400 MHz
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

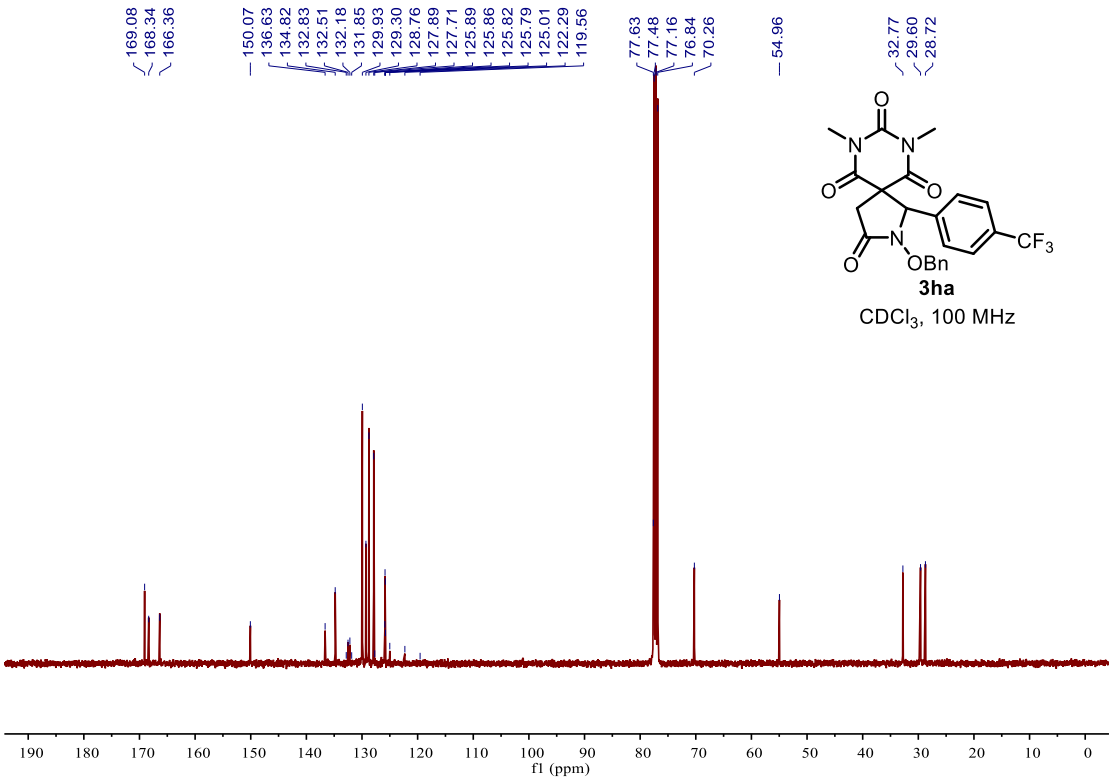

169.17
168.42
166.50
150.17
134.84
132.15
131.45
129.90
129.21
128.95
128.72
124.38
77.61
77.48
77.16
76.84
70.37
55.00
32.64
29.55
28.83
O
N
O
N
O
O
N
OBn
Br
3fa
CDCl3, 100 MHz
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

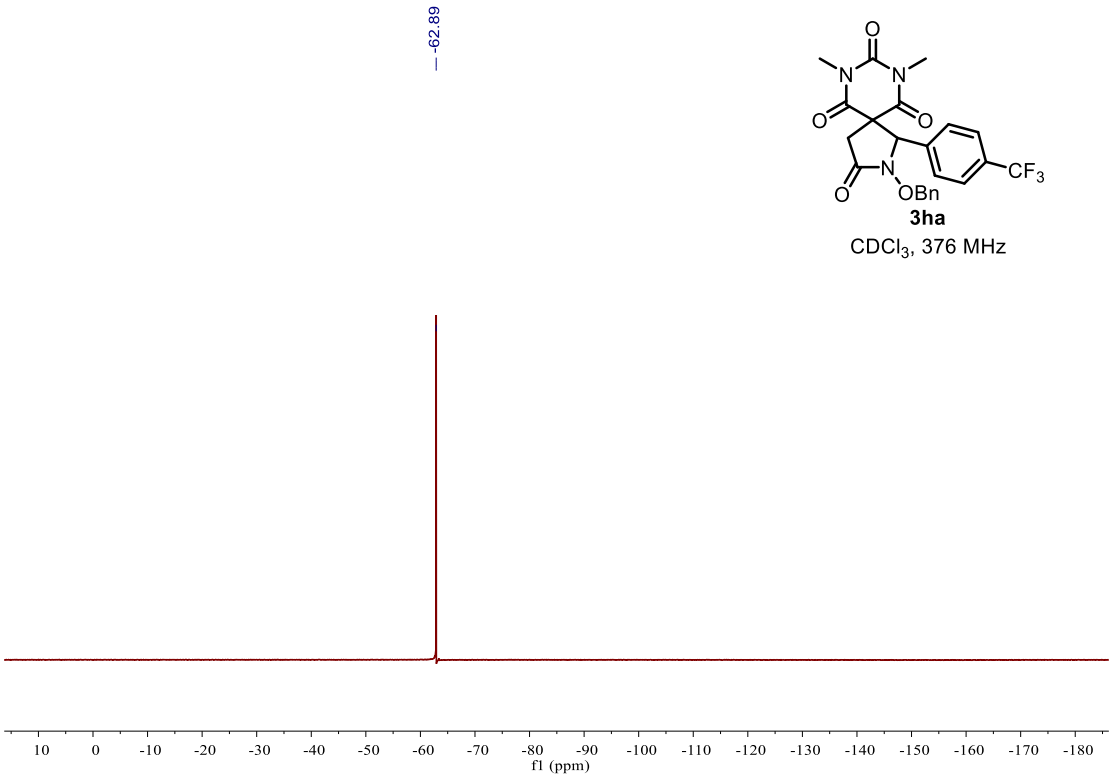

7.629
7.609
7.385
7.367
7.349
7.330
7.311
7.293
7.213
7.195
7.042
7.021
5.119
5.091
4.910
4.882
4.278
3.295
3.208
3.166
3.147
3.105
2.695
3.28
2.05
1.97
2.01
1.00
0.96
1.00
3.02
2.97
CN
OBn
3ga
CDCl3, 400 MHz

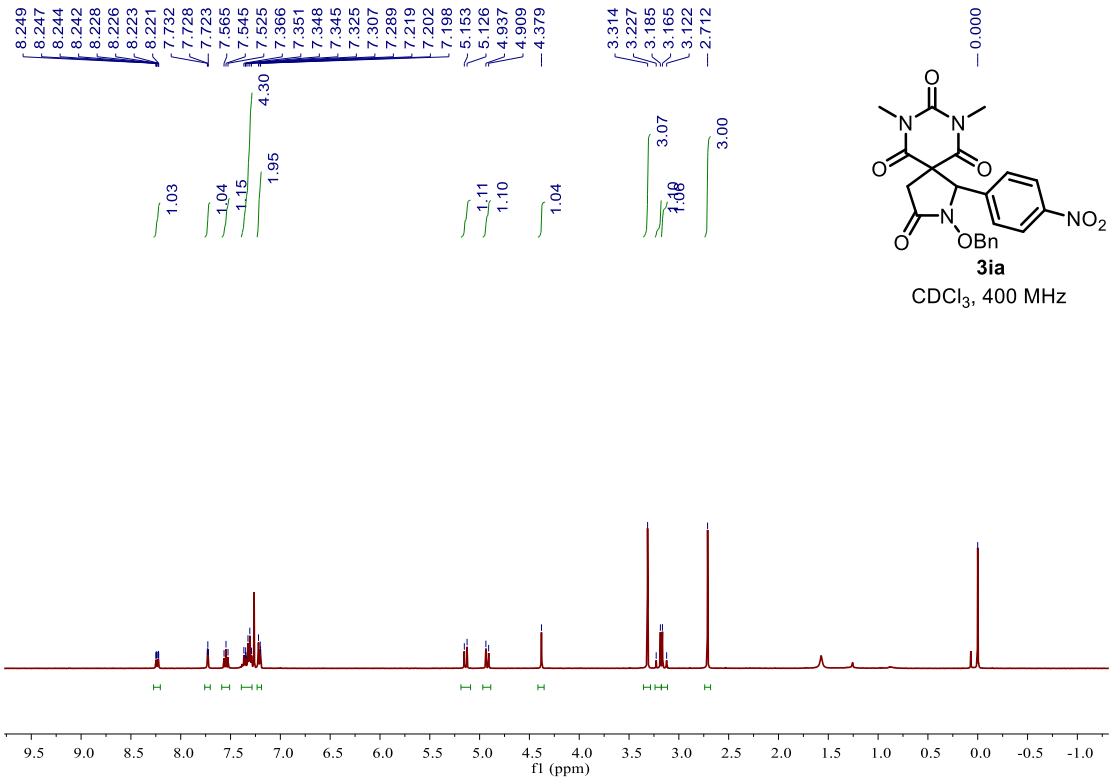
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


168.95
168.19
166.17
150.00
137.84
134.76
132.54
129.95
129.36
128.80
128.21
117.84
114.11
77.66
77.48
77.16
76.84
70.11
54.79
32.83
29.63
28.82
CN
OBn
3ga
CDCl3, 100 MHz
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

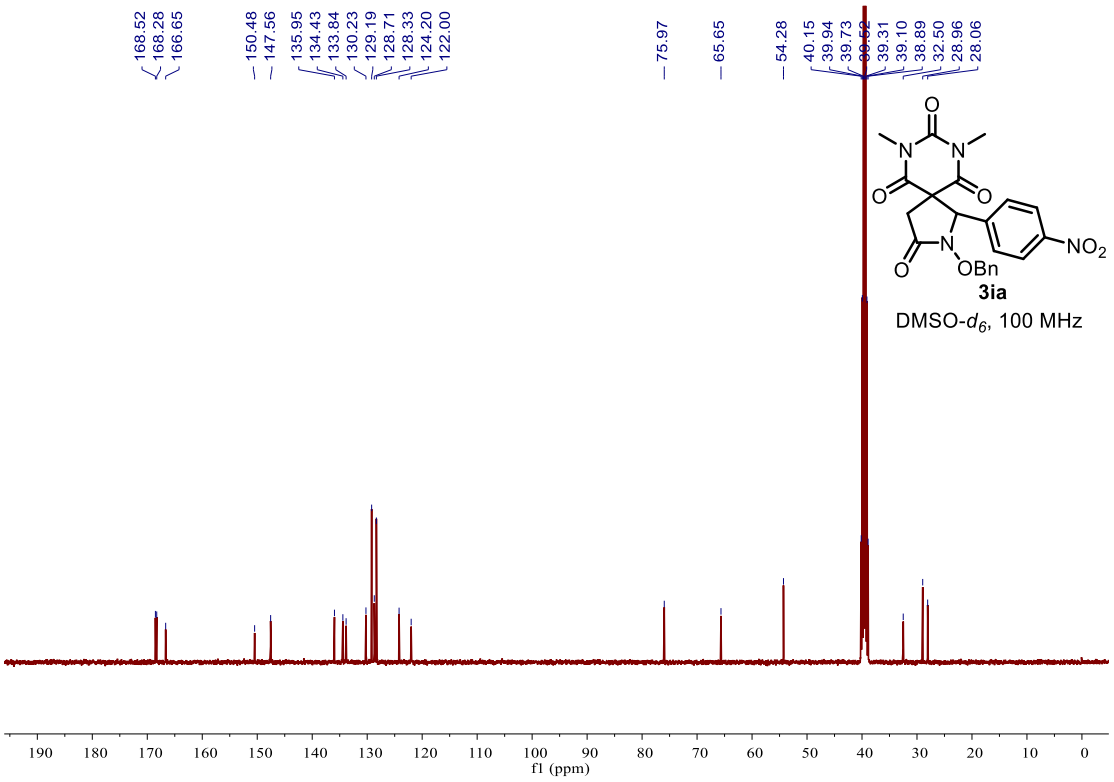

7.601
7.581
7.358
7.352
7.341
7.325
7.306
7.289
7.221
7.204
7.067
7.047
5.138
5.111
4.913
4.885
4.320
3.299
3.227
3.185
3.163
3.120
2.654
3.00
2.93
2.00
1.92
1.97
0.99
0.97
0.97
1.03
0.98
CF3
OBn
3ha
CDCl3, 400 MHz
f1 (ppm)

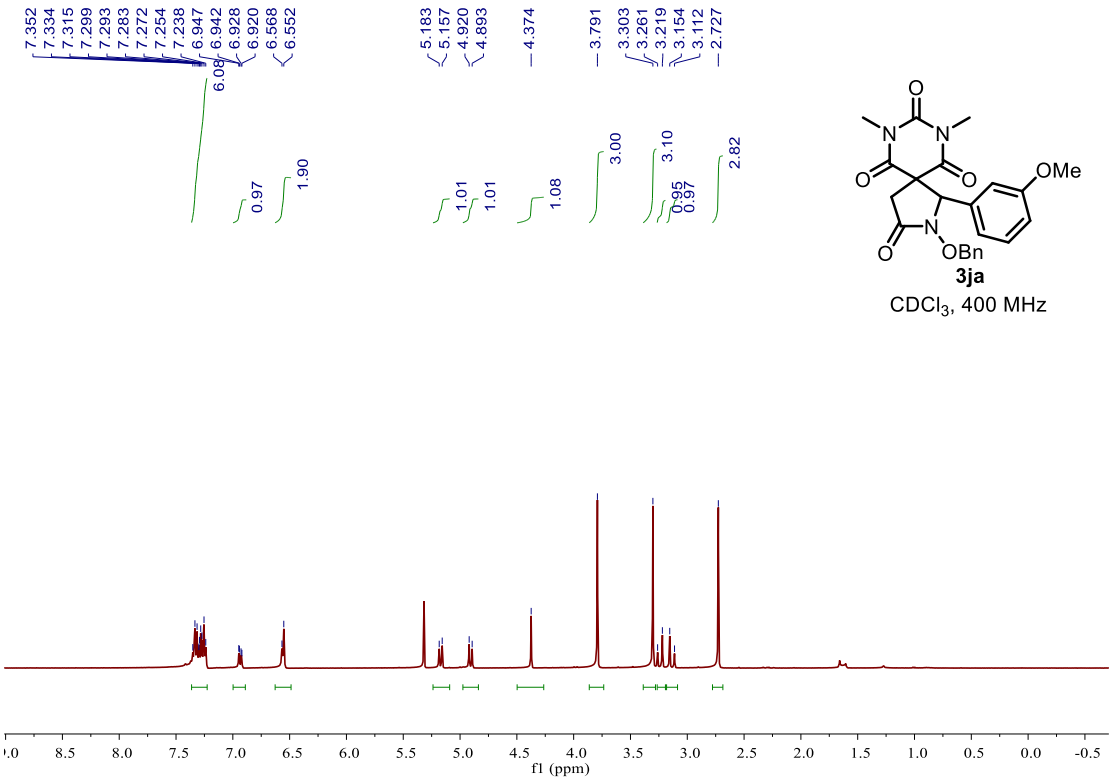

169.08
168.34
166.36
150.07
136.63
134.82
132.83
132.51
132.18
131.85
129.93
129.30
128.76
127.89
127.71
125.89
125.86
125.82
125.79
125.01
122.29
119.56
77.63
77.48
77.16
76.84
70.26
54.96
32.77
29.60
28.72
CF3
OBn
3ha
CDCl3, 100 MHz
f1 (ppm)

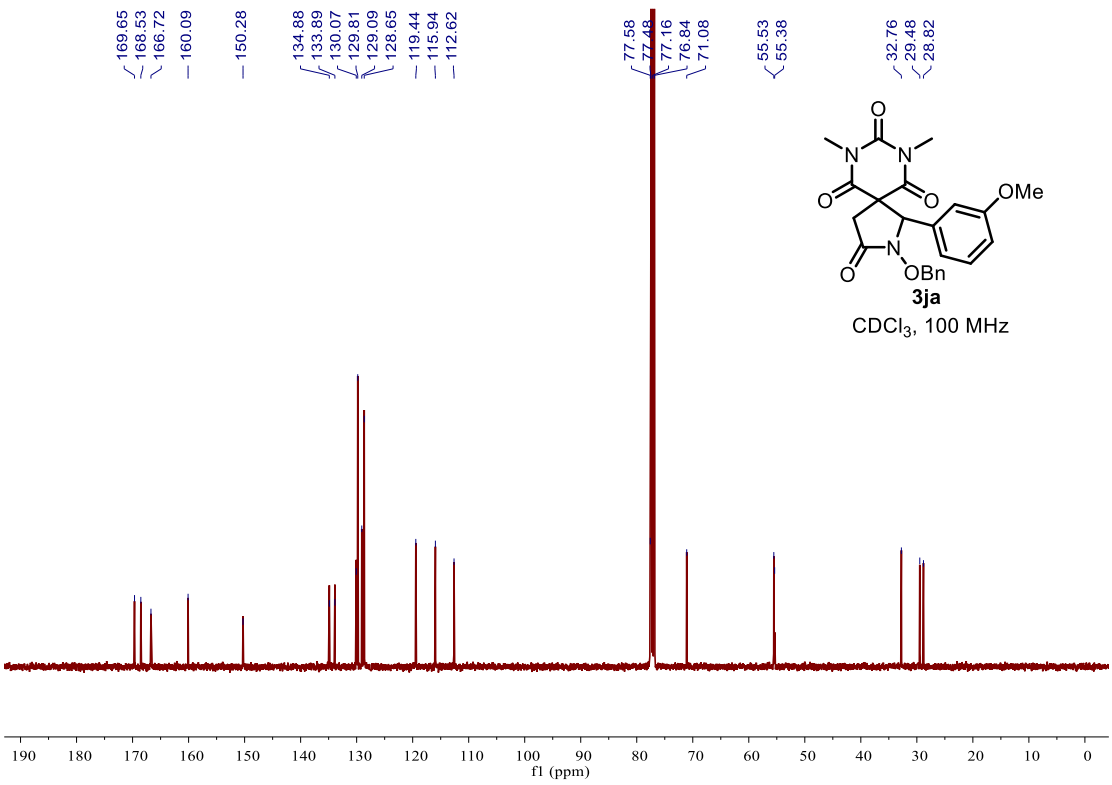

-62.89
O
N
N
O
O
O
N
OBn
CF3
3ha
CDCl3, 376 MHz
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
f1 (ppm)

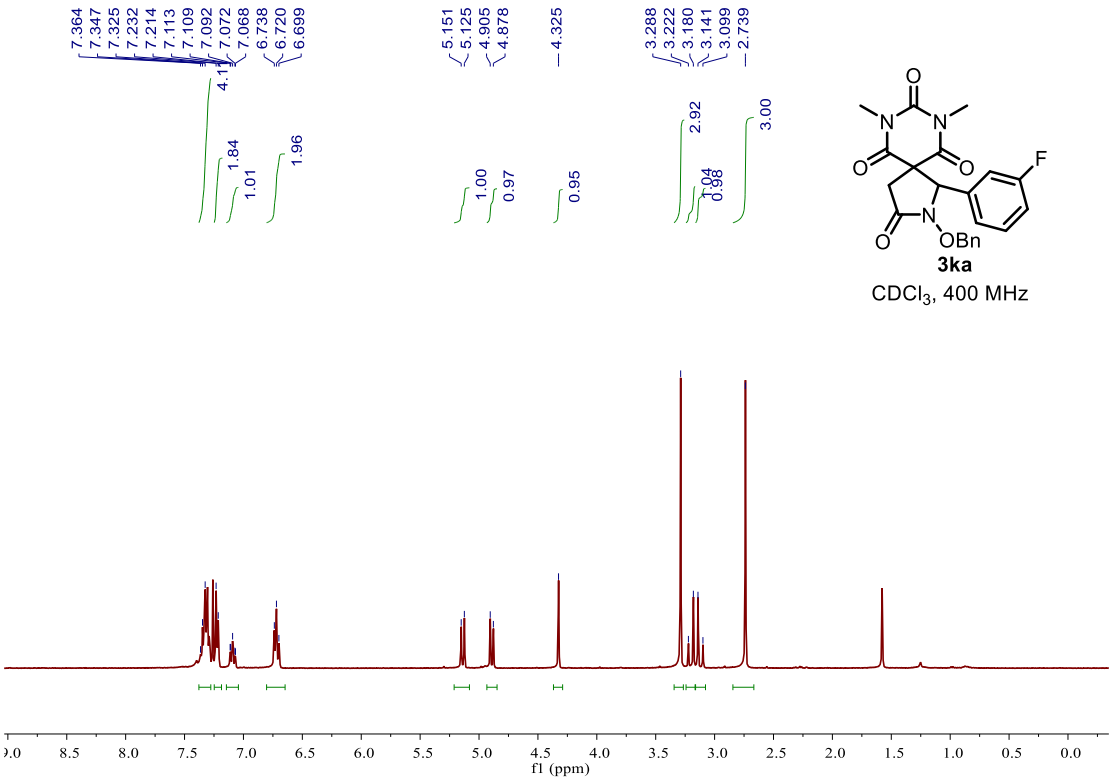

8.249
8.247
8.244
8.242
8.228
8.226
8.223
8.221
7.732
7.728
7.723
7.565
7.545
7.525
7.366
7.351
7.348
7.345
7.325
7.307
7.289
7.219
7.202
7.198
5.153
5.126
4.937
4.909
4.379
3.314
3.227
3.185
3.165
3.122
2.712
0.000
1.03
1.04
1.15
4.30
1.95
1.11
1.10
1.04
3.07
1.08
3.00
O
N
O
O
N
O
OBn
NO2
3ia
CDCl3, 400 MHz

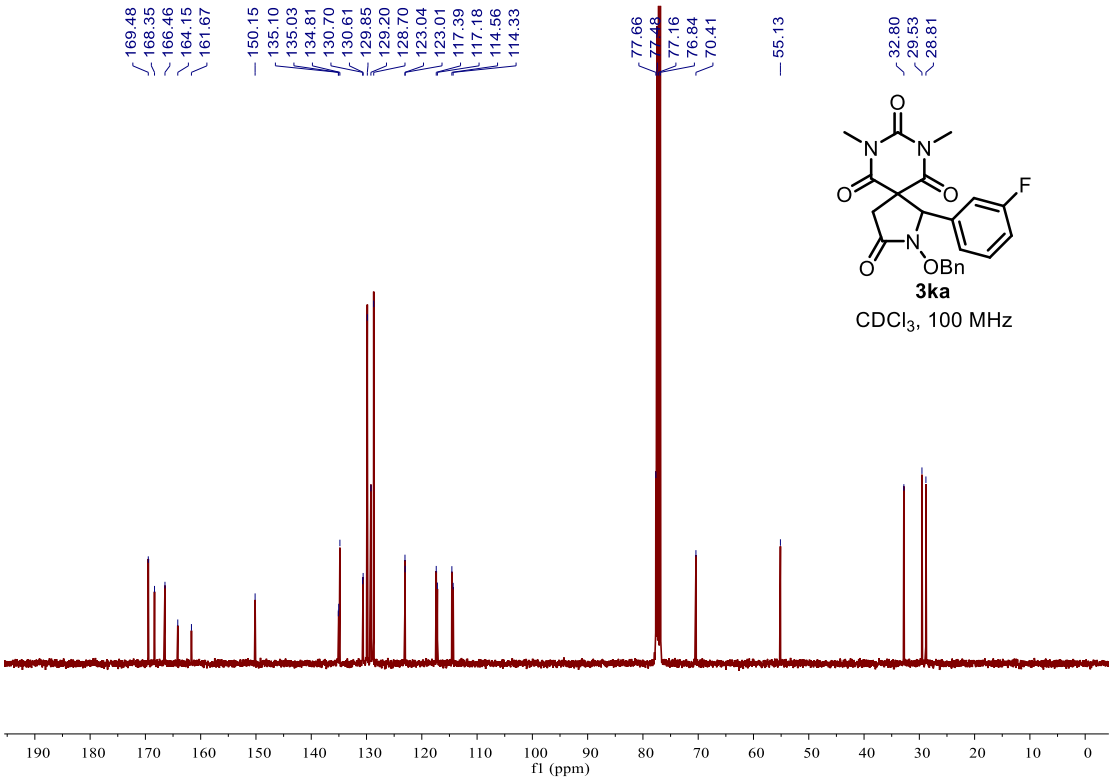
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

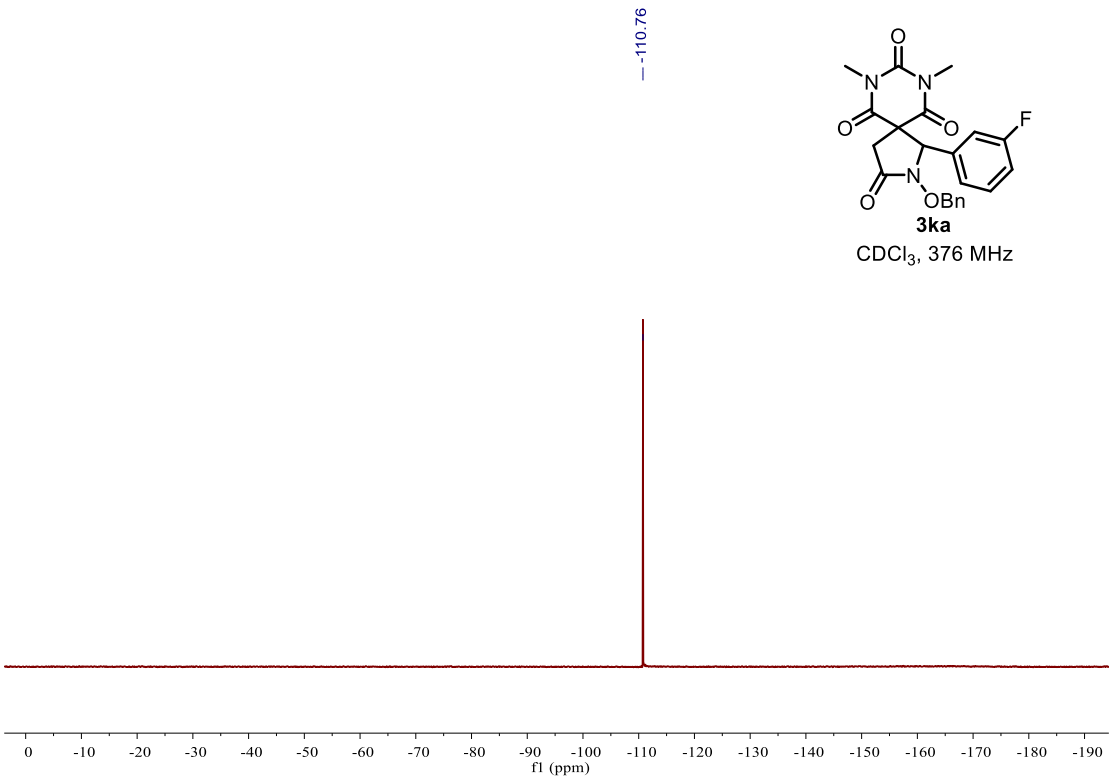

168.52
168.28
166.65
150.48
147.56
135.95
134.43
133.84
130.23
129.19
128.71
128.33
124.20
122.00
75.97
65.65
54.28
40.15
39.94
39.73
39.52
39.31
39.10
38.89
32.50
28.96
28.06
O
N
O
O
N
O
OBn
NO2
3ia
DMSO-d6, 100 MHz
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

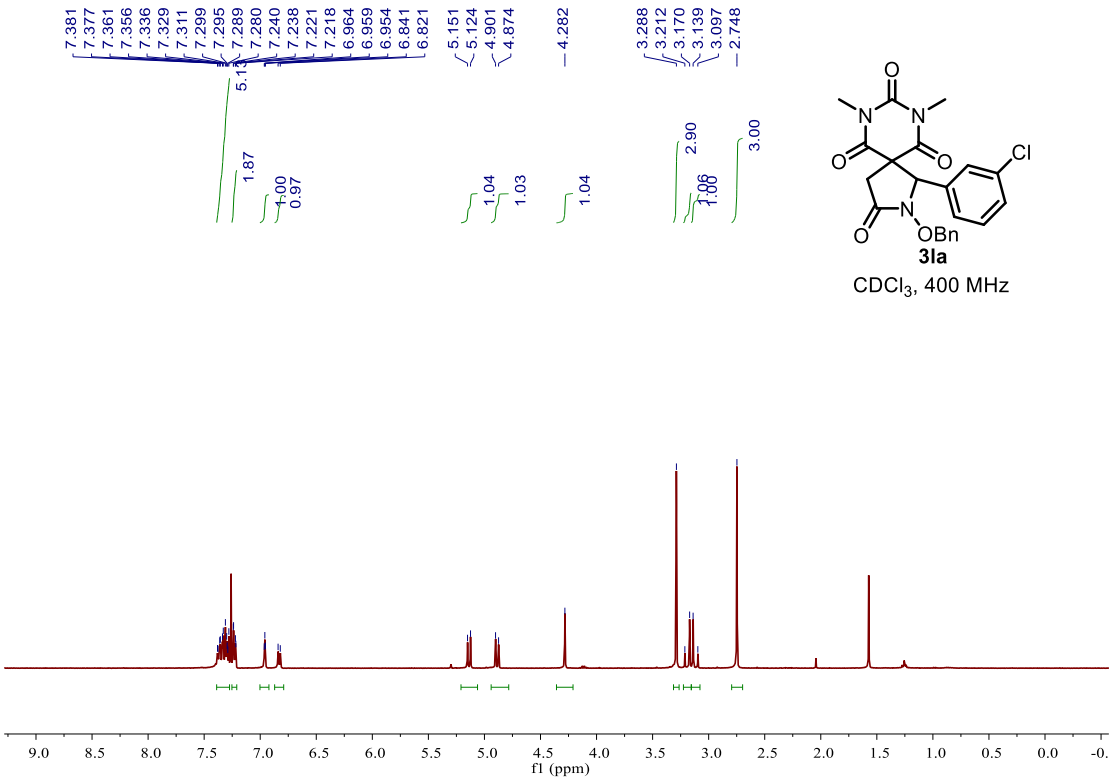

7.352
7.334
7.315
7.299
7.293
7.283
7.272
7.254
7.238
6.947
6.942
6.928
6.920
6.568
6.552
5.183
5.157
4.920
4.893
4.374
3.791
3.303
3.261
3.219
3.154
3.112
2.727
6.08
0.97
1.90
1.01
1.01
1.08
3.00
3.10
0.95
0.97
2.82
O
N
N
O
O
OMe
N
OBn
O
3ja
CDCl3, 400 MHz
f1 (ppm)

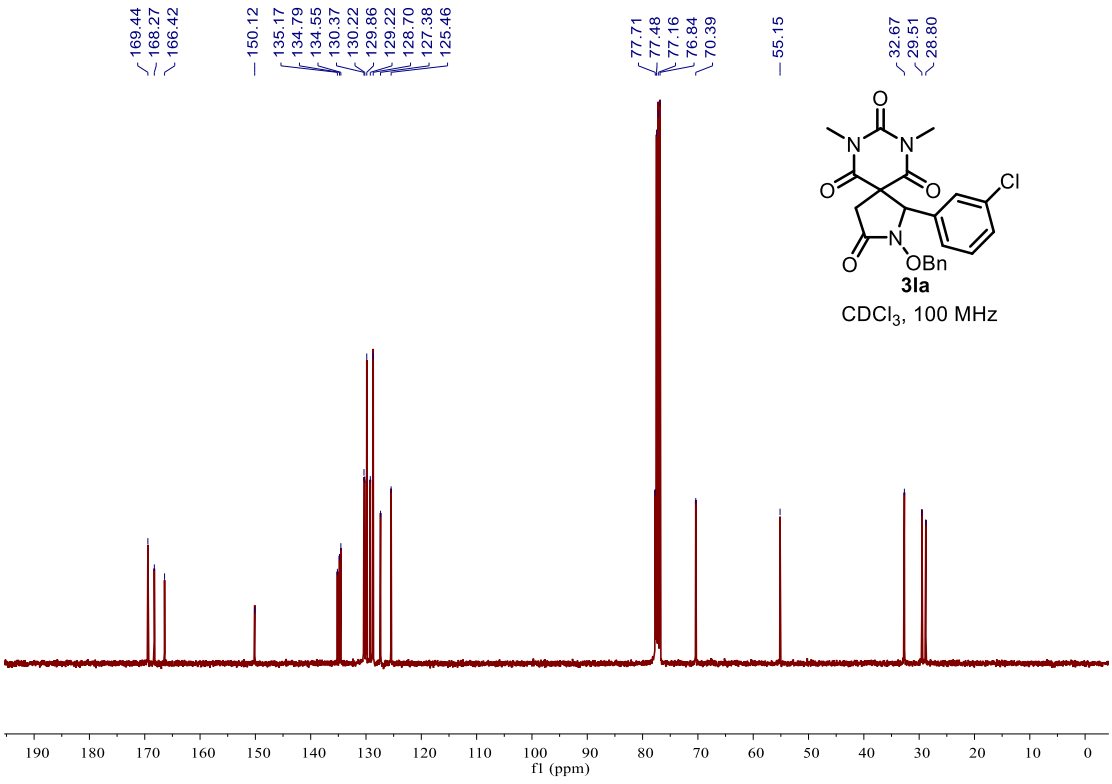

169.65
168.53
166.72
160.09
150.28
134.88
133.89
130.07
129.81
129.09
128.65
119.44
115.94
112.62
77.58
77.48
77.16
76.84
71.08
55.53
55.38
32.76
29.48
28.82
O
N
N
O
O
OMe
N
OBn
O
3ja
CDCl3, 100 MHz
f1 (ppm)

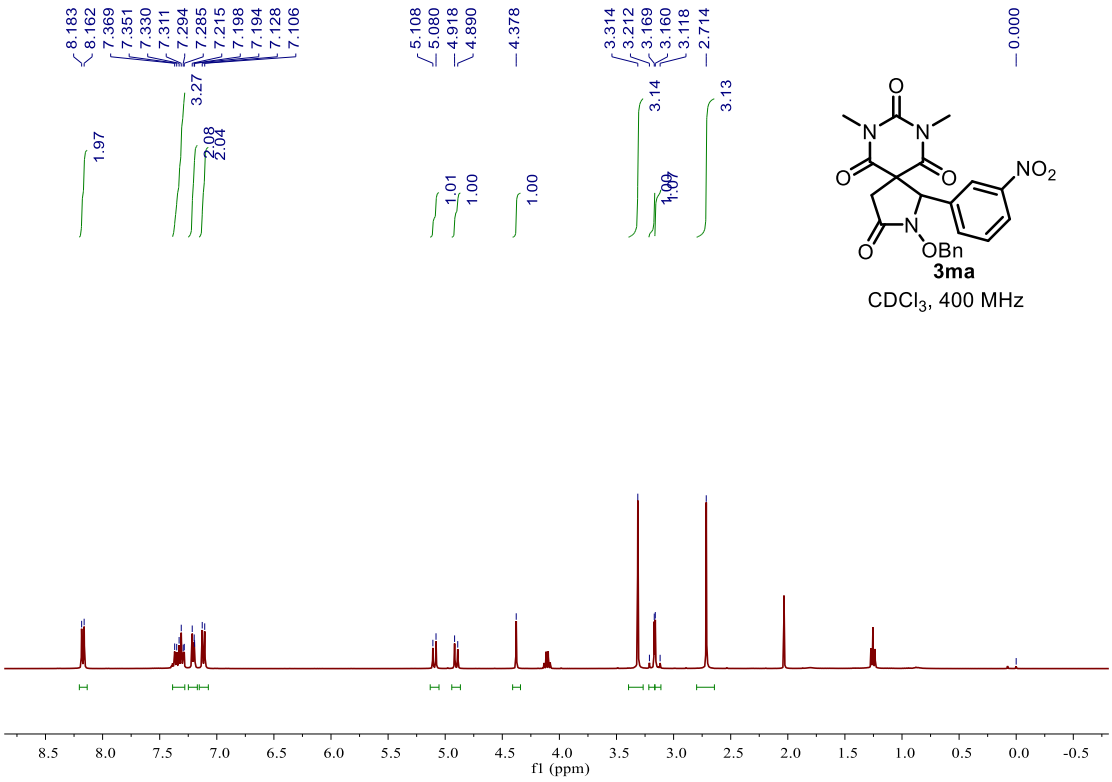

7.364
7.347
7.325
7.232
7.214
7.113
7.109
7.092
7.072
7.068
6.738
6.720
6.699
5.151
5.125
4.905
4.878
4.325
3.288
3.222
3.180
3.141
3.099
2.739
4.11
1.84
1.01
1.96
1.00
0.97
0.95
2.92
1.04
0.98
3.00
O
N
O
N
O
F
N
O
OBn
3ka
CDCl3, 400 MHz
f1 (ppm)

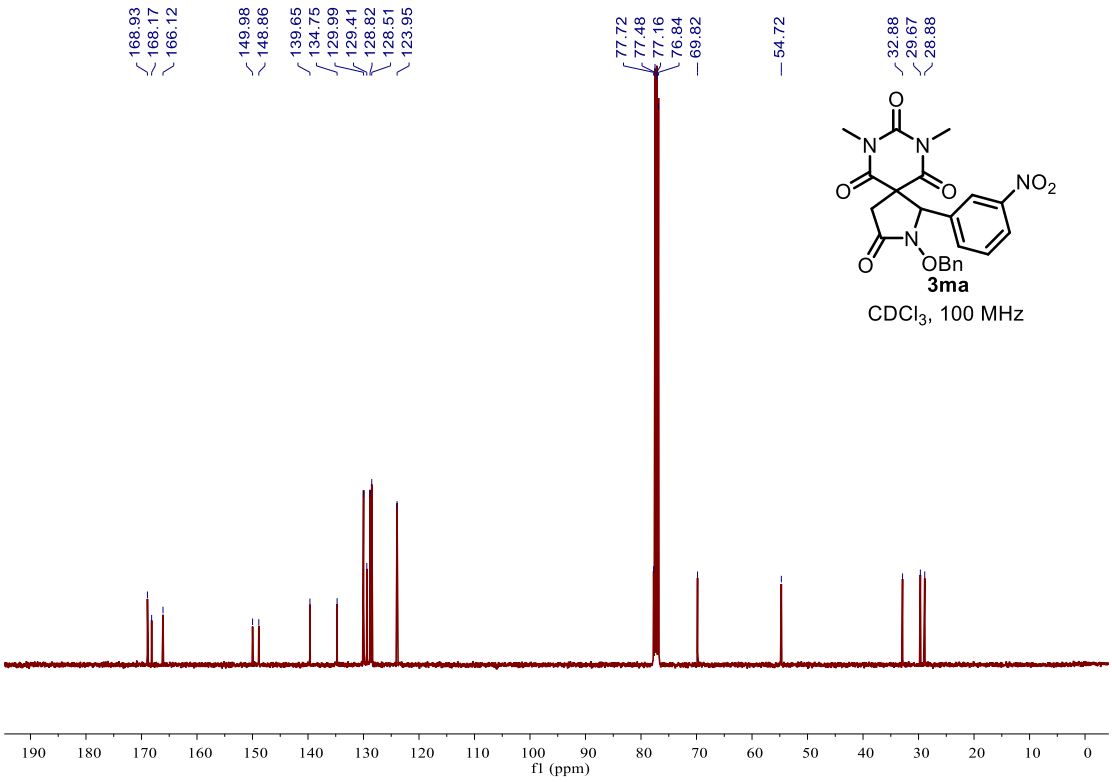

169.48
168.35
166.46
164.15
161.67
150.15
135.10
135.03
134.81
130.70
130.61
129.85
129.20
128.70
123.04
123.01
117.39
117.18
114.56
114.33
77.66
77.48
77.16
76.84
70.41
55.13
32.80
29.53
28.81
O
N
O
N
O
F
N
O
OBn
3ka
CDCl3, 100 MHz
f1 (ppm)

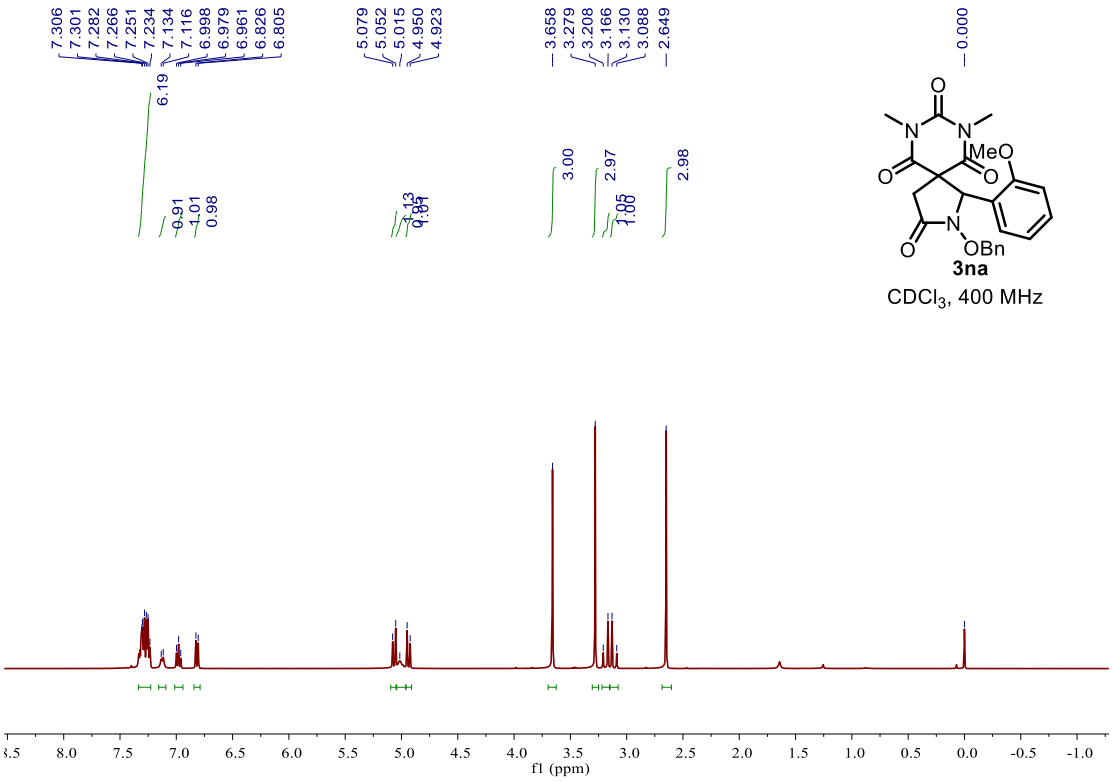

-110.76
O
N
N
O
O
F
N
O
OBn
3ka
CDCl3, 376 MHz
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

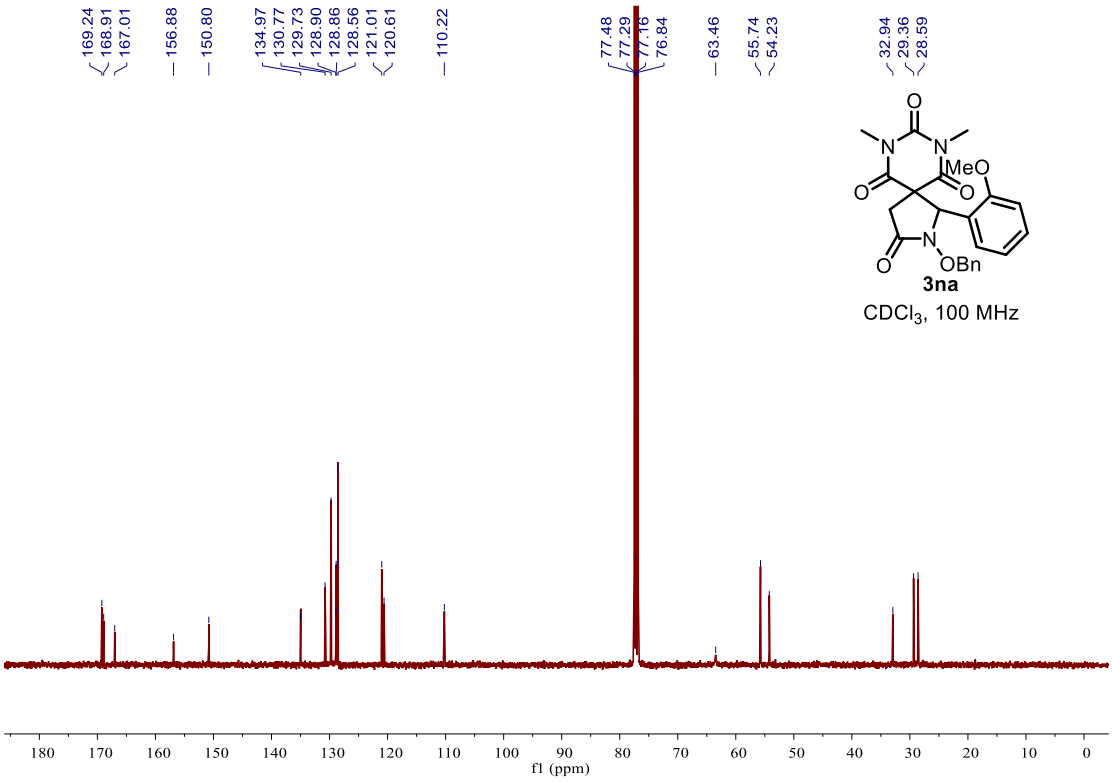

7.381
7.377
7.361
7.356
7.336
7.329
7.311
7.299
7.295
7.289
7.280
7.240
7.238
7.221
7.218
6.964
6.959
6.954
6.841
6.821
5.151
5.124
4.901
4.874
4.282
3.288
3.212
3.170
3.139
3.097
2.748
1.87
1.00
0.97
1.04
1.03
1.04
2.90
1.06
1.00
3.00
O
N
N
O
O
Cl
N
O
OBn
3la
CDCl3, 400 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

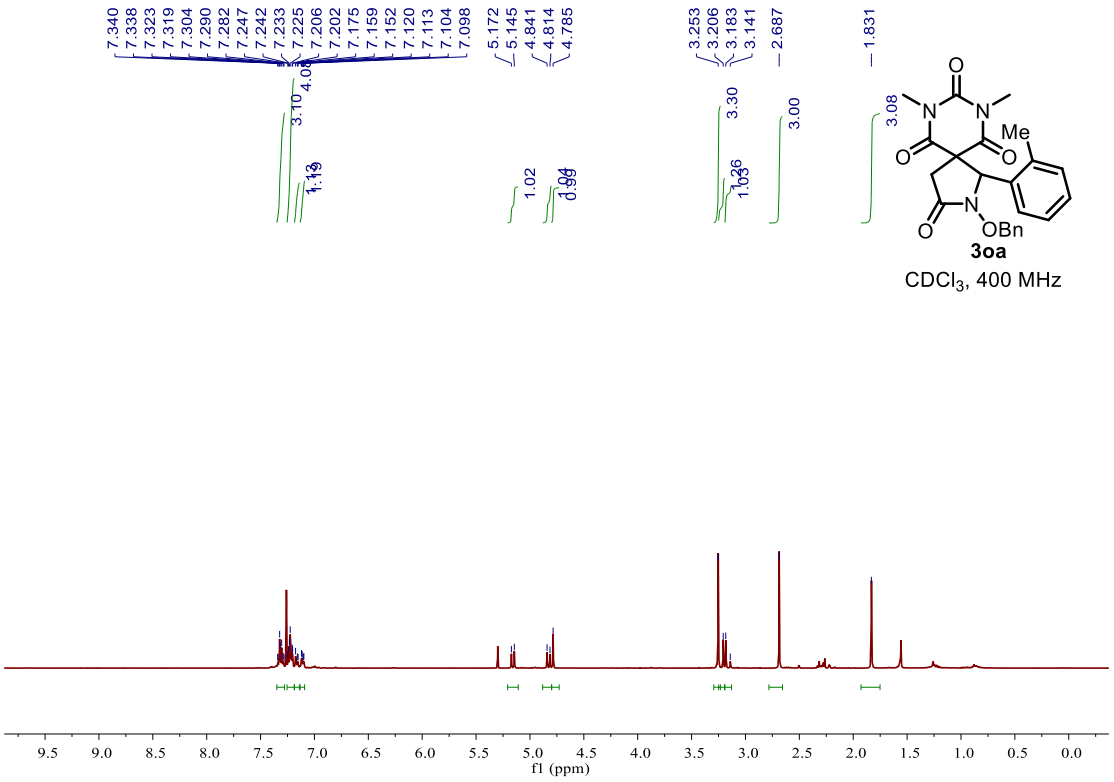

169.44
168.27
166.42
150.12
135.17
134.79
134.55
130.37
130.22
129.86
129.22
128.70
127.38
125.46
77.71
77.48
77.16
76.84
70.39
55.15
32.67
29.51
28.80
O
N
N
O
O
Cl
N
O
OBn
3la
CDCl3, 100 MHz
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

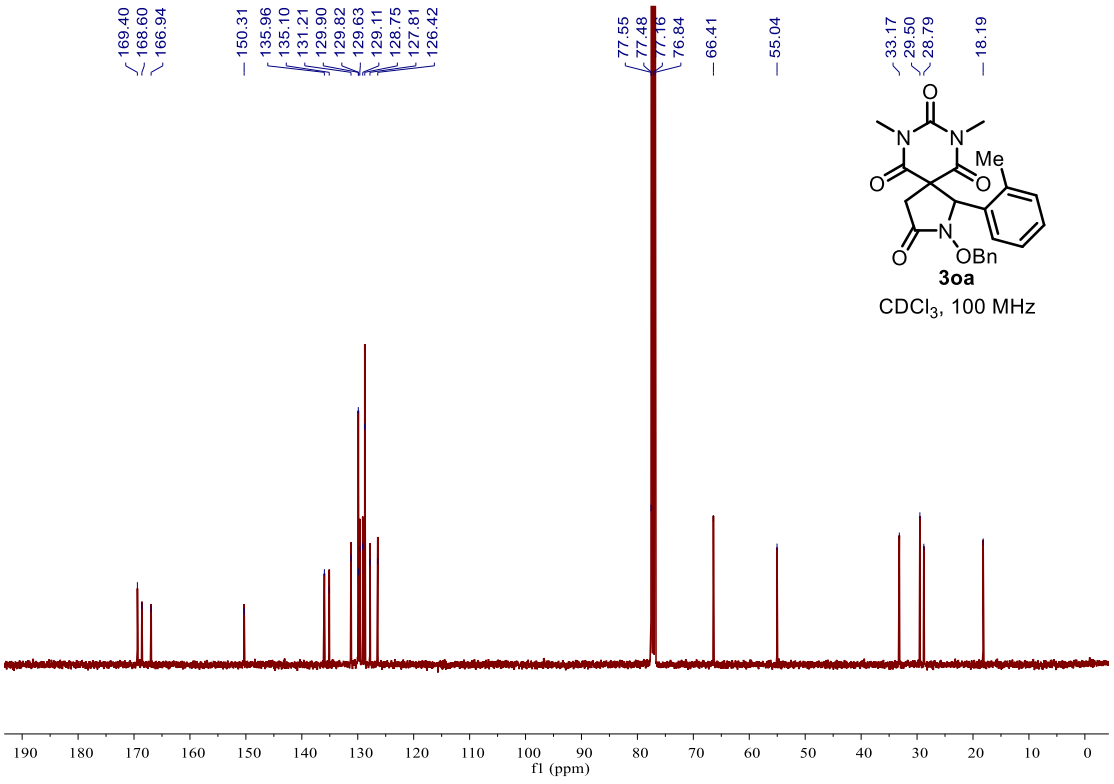

8.183
8.162
7.369
7.351
7.330
7.311
7.294
7.285
7.215
7.198
7.194
7.128
7.106
5.108
5.080
4.918
4.890
4.378
3.314
3.212
3.169
3.160
3.118
2.714
0.000
3.27
1.97
2.08
2.04
1.01
1.00
1.00
3.14
1.07
1.07
3.13
NO2
OBn
3ma
CDCl3, 400 MHz
f1 (ppm)

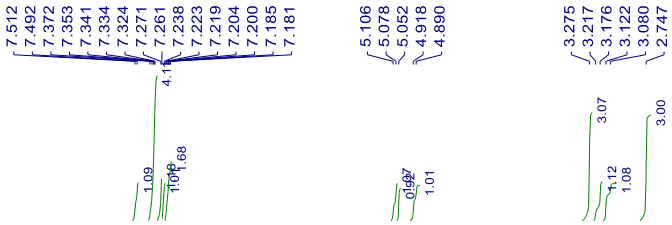

168.93
168.17
166.12
149.98
148.86
139.65
134.75
129.99
129.41
128.82
128.51
123.95
77.72
77.48
77.16
76.84
69.82
54.72
32.88
29.67
28.88
NO2
OBn
3ma
CDCl3, 100 MHz
f1 (ppm)


7.306
7.301
7.282
7.266
7.251
7.234
7.134
7.116
6.998
6.979
6.961
6.826
6.805
5.079
5.052
5.015
4.950
4.923
3.658
3.279
3.208
3.166
3.130
3.088
2.649
0.000
6.19
0.91
1.01
0.98
2.13
2.04
3.00
2.97
1.05
1.00
2.98
MeO
OBn
3na
CDCl3, 400 MHz
f1 (ppm)


169.24
168.91
167.01
156.88
150.80
134.97
130.77
129.73
128.90
128.86
128.56
121.01
120.61
110.22
77.48
77.29
77.16
76.84
63.46
55.74
54.23
32.94
29.36
28.59
MeO
OBn
3na
CDCl3, 100 MHz
f1 (ppm)

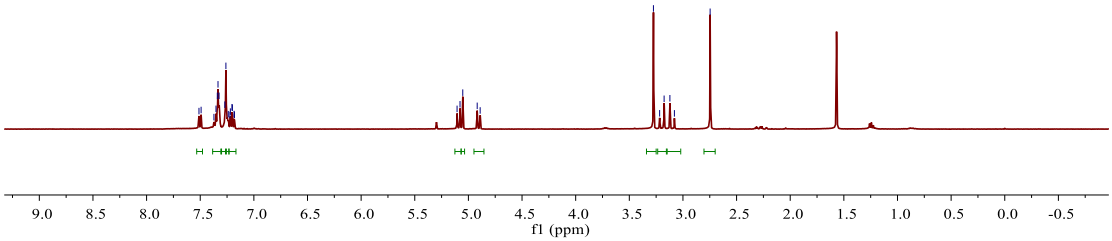

7.340
7.338
7.323
7.319
7.304
7.290
7.282
7.247
7.242
7.233
7.225
7.206
7.202
7.175
7.159
7.152
7.120
7.113
7.104
7.098
5.172
5.145
4.841
4.814
4.785
3.253
3.206
3.183
3.141
2.687
1.831
4.00
3.10
1.13
1.02
0.99
0.94
3.30
1.36
1.03
3.00
3.08
Me
OBn
3oa
CDCl3, 400 MHz
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

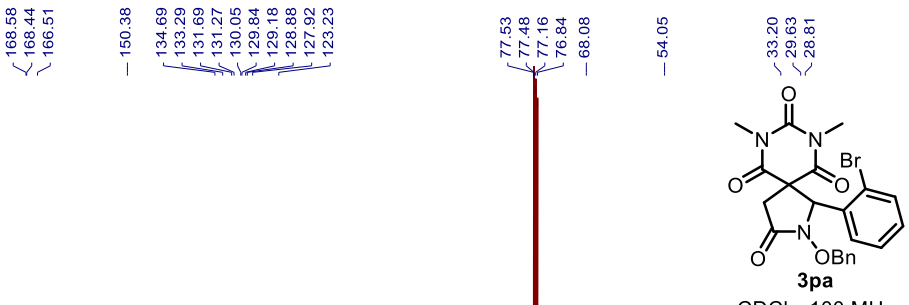

169.40
168.60
166.94
150.31
135.96
135.10
131.21
129.90
129.82
129.63
129.11
128.75
127.81
126.42
77.55
77.48
77.16
76.84
66.41
55.04
33.17
29.50
28.79
18.19
Me
OBn
3oa
CDCl3, 100 MHz
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



3pa

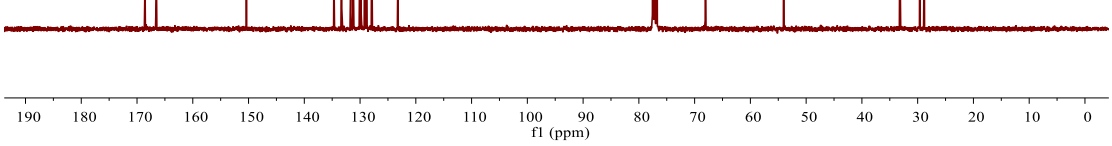
$CDCl_3$, 400 MHz

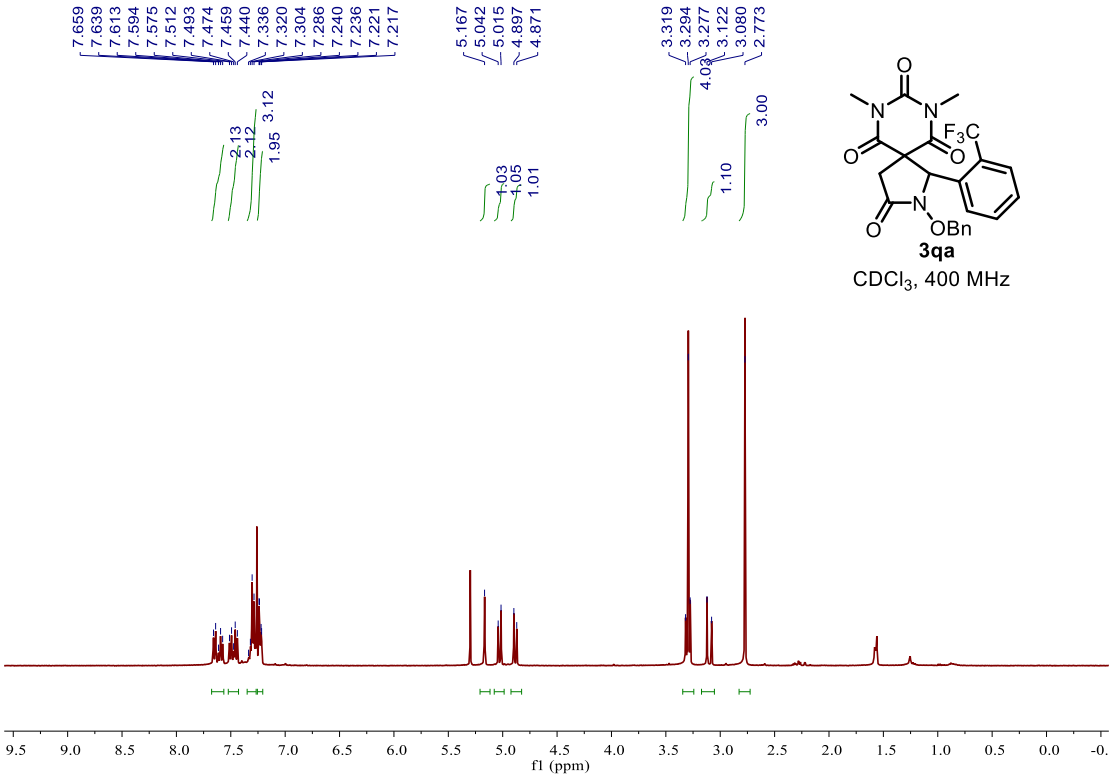


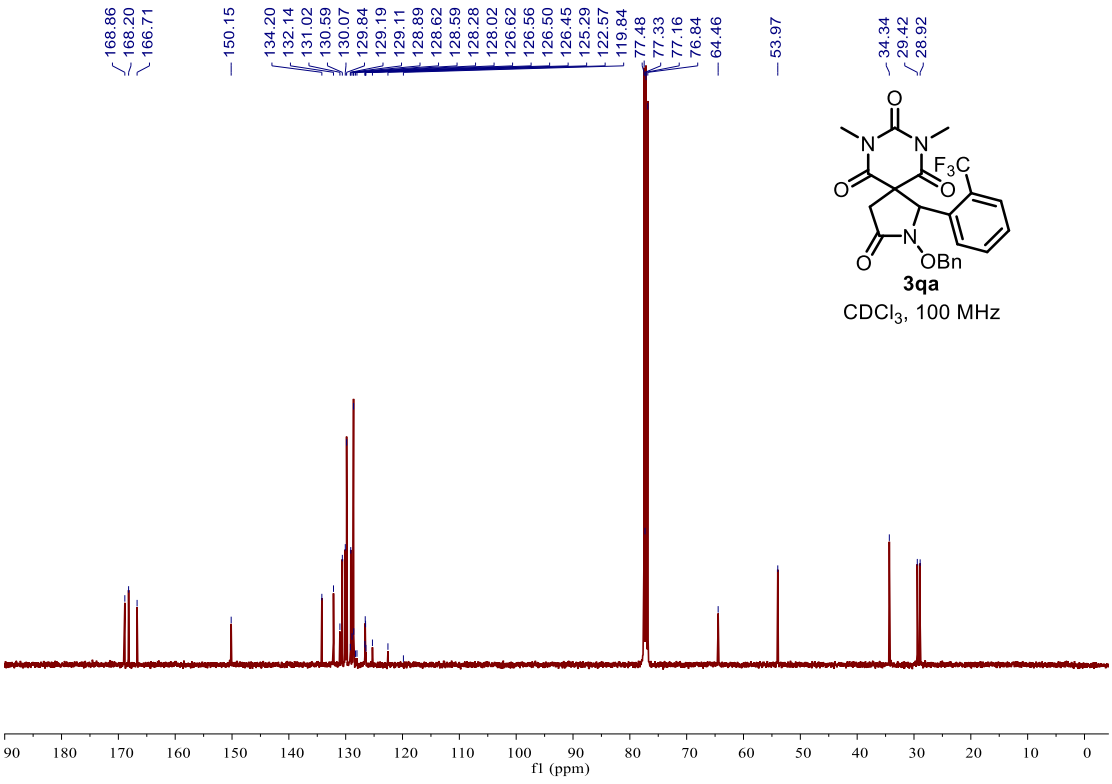


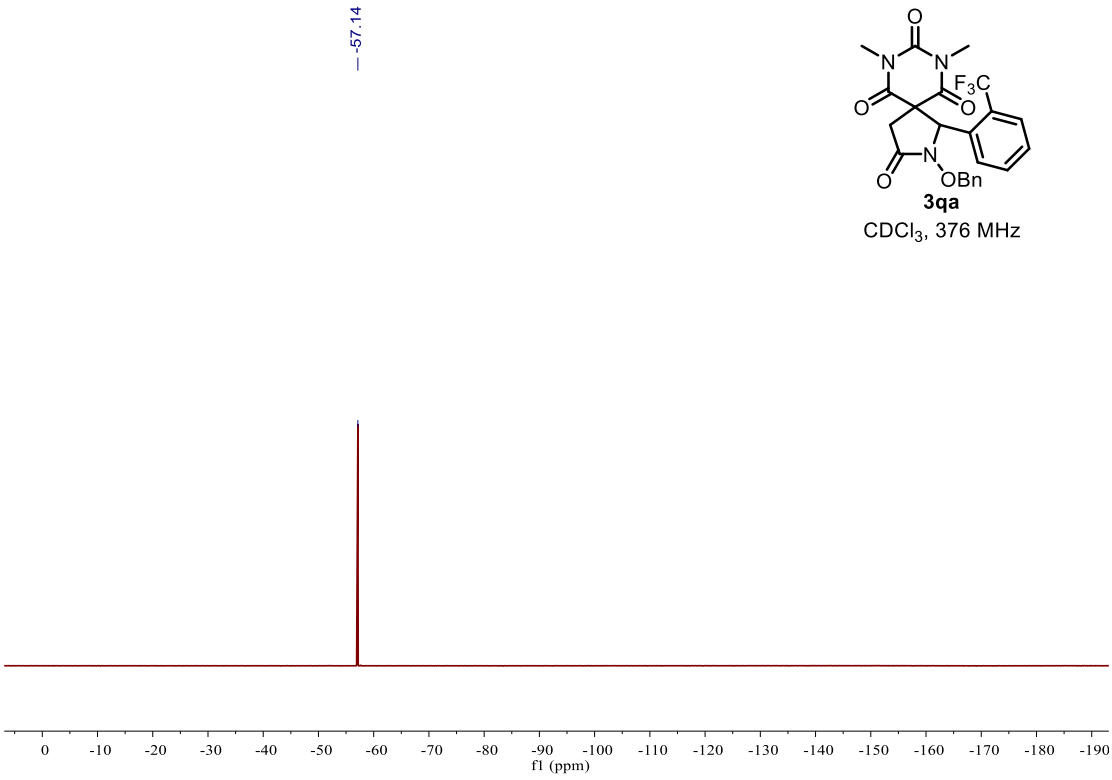
3pa

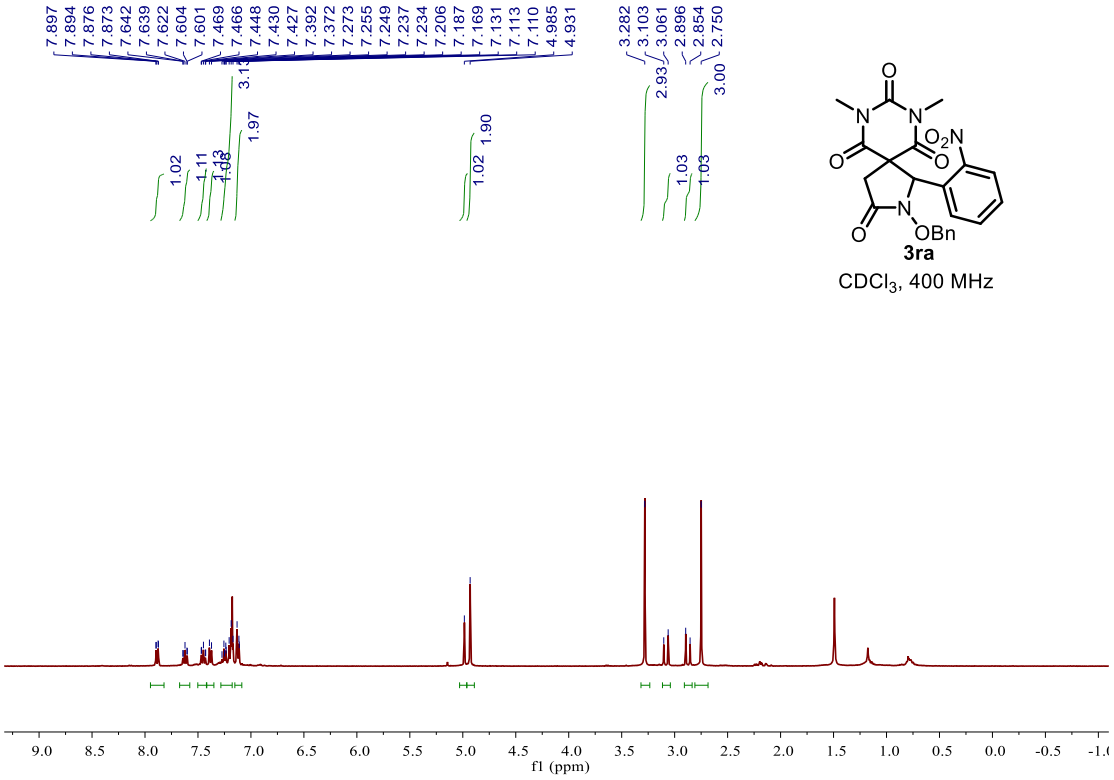
$CDCl_3$, 100 MHz

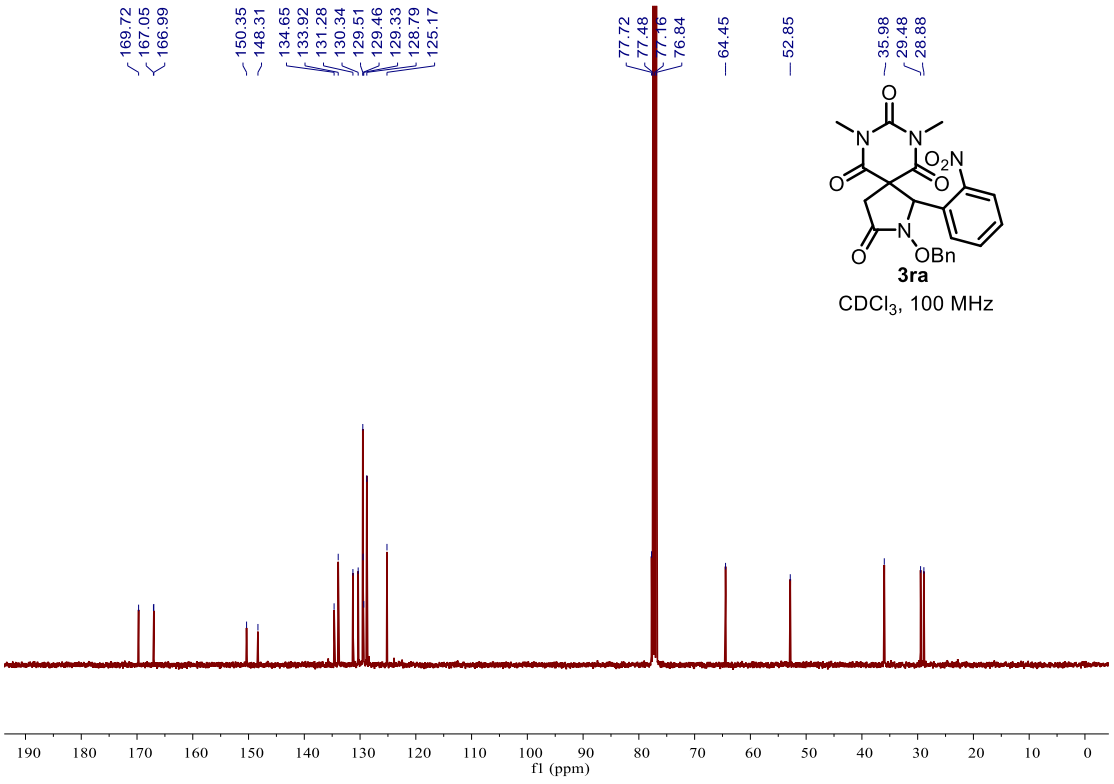


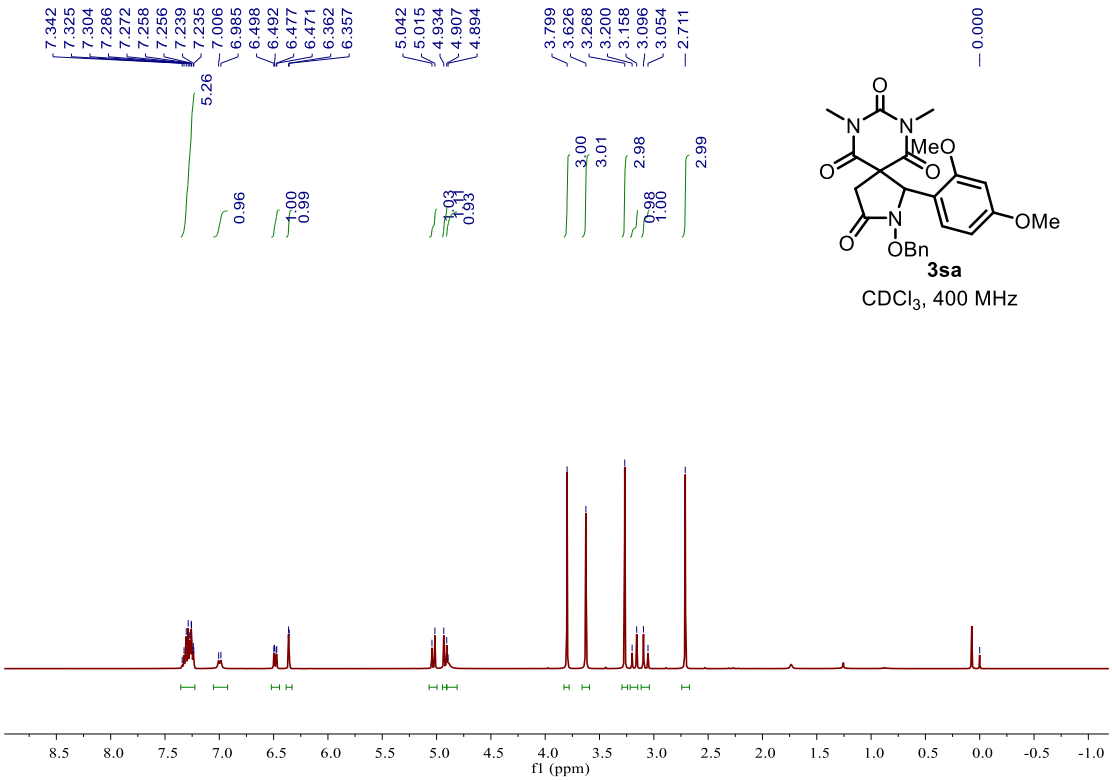

7.659
7.639
7.613
7.594
7.575
7.512
7.493
7.474
7.459
7.440
7.336
7.320
7.304
7.286
7.240
7.236
7.221
7.217
5.167
5.042
5.015
4.897
4.871
3.319
3.294
3.277
3.122
3.080
2.773
2.13
2.12
3.12
1.95
1.03
1.05
1.01
4.08
1.10
3.00
F3C
OBn
3qa
CDCl3, 400 MHz
f1 (ppm)

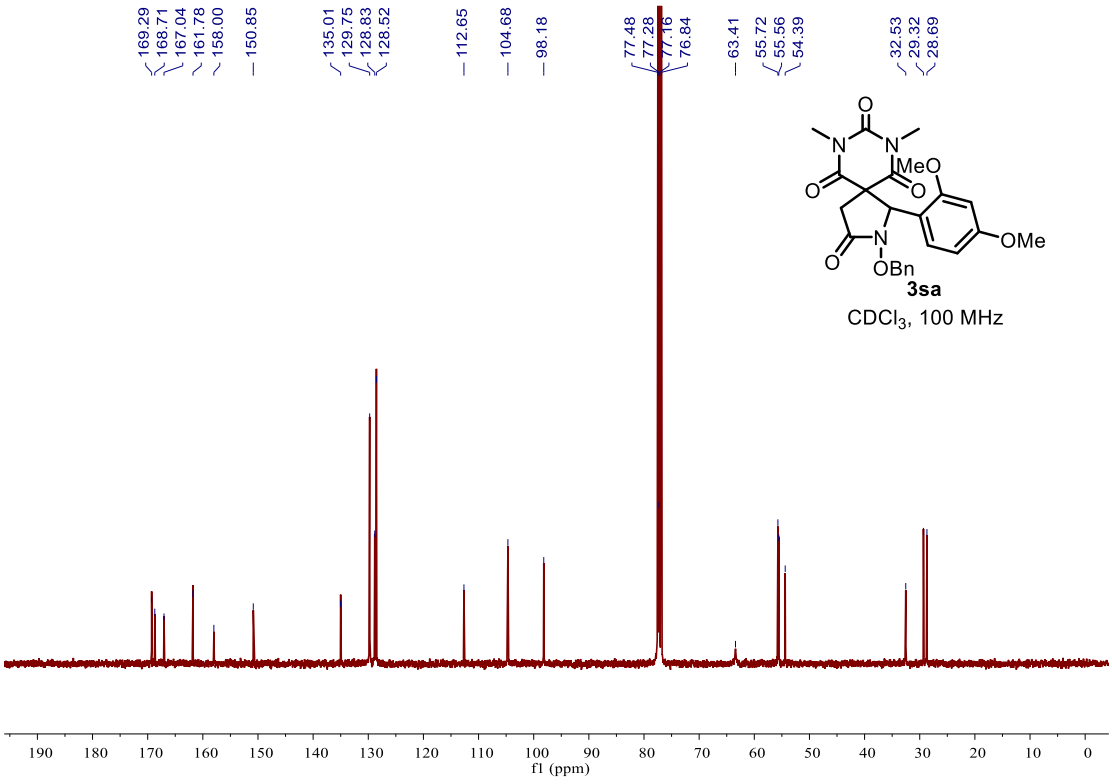

168.86
168.20
166.71
150.15
134.20
132.14
131.02
130.59
130.07
129.84
129.19
129.11
128.89
128.62
128.59
128.28
128.02
126.62
126.56
126.50
126.45
125.29
122.57
119.84
77.48
77.33
77.16
76.84
64.46
53.97
34.34
29.42
28.92
F3C
OBn
3qa
CDCl3, 100 MHz
f1 (ppm)

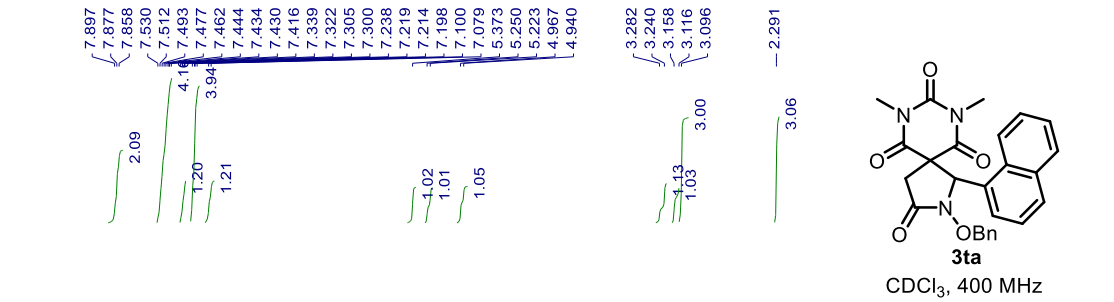

-57.14
O
N
N
F3C
O
O
N
O
OBn
3qa
CDCl3, 376 MHz
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)


7.897
7.894
7.876
7.873
7.642
7.639
7.622
7.604
7.601
7.469
7.466
7.448
7.430
7.427
7.392
7.372
7.273
7.255
7.249
7.237
7.234
7.206
7.187
7.169
7.131
7.113
7.110
4.985
4.931
3.282
3.103
3.061
2.896
2.854
2.750
1.02
1.11
1.13
1.18
1.97
1.02
1.90
2.93
1.03
1.03
3.00
O
N
N
O2N
O
O
N
O
OBn
3ra
CDCl3, 400 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


169.72
167.05
166.99
150.35
148.31
134.65
133.92
131.28
130.34
129.51
129.46
129.33
128.79
125.17
77.72
77.48
77.16
76.84
64.45
52.85
35.98
29.48
28.88
O
N
N
O2N
O
O
N
O
OBn
3ra
CDCl3, 100 MHz
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

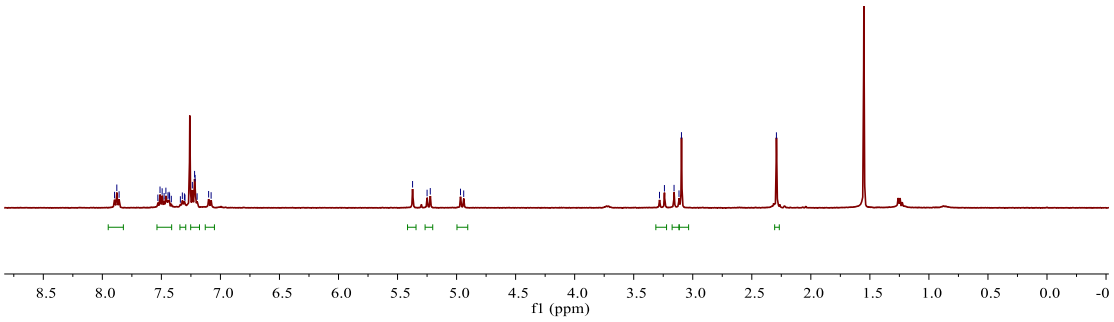

7.342
7.325
7.304
7.286
7.272
7.258
7.256
7.239
7.235
7.006
6.985
6.498
6.492
6.477
6.471
6.362
6.357
5.042
5.015
4.934
4.907
4.894
3.799
3.626
3.268
3.200
3.158
3.096
3.054
2.711
0.000
5.26
0.96
1.00
0.99
1.03
1.01
0.93
3.00
3.01
2.98
0.98
1.00
2.99
MeO
OMe
OBn
3sa
CDCl3, 400 MHz
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

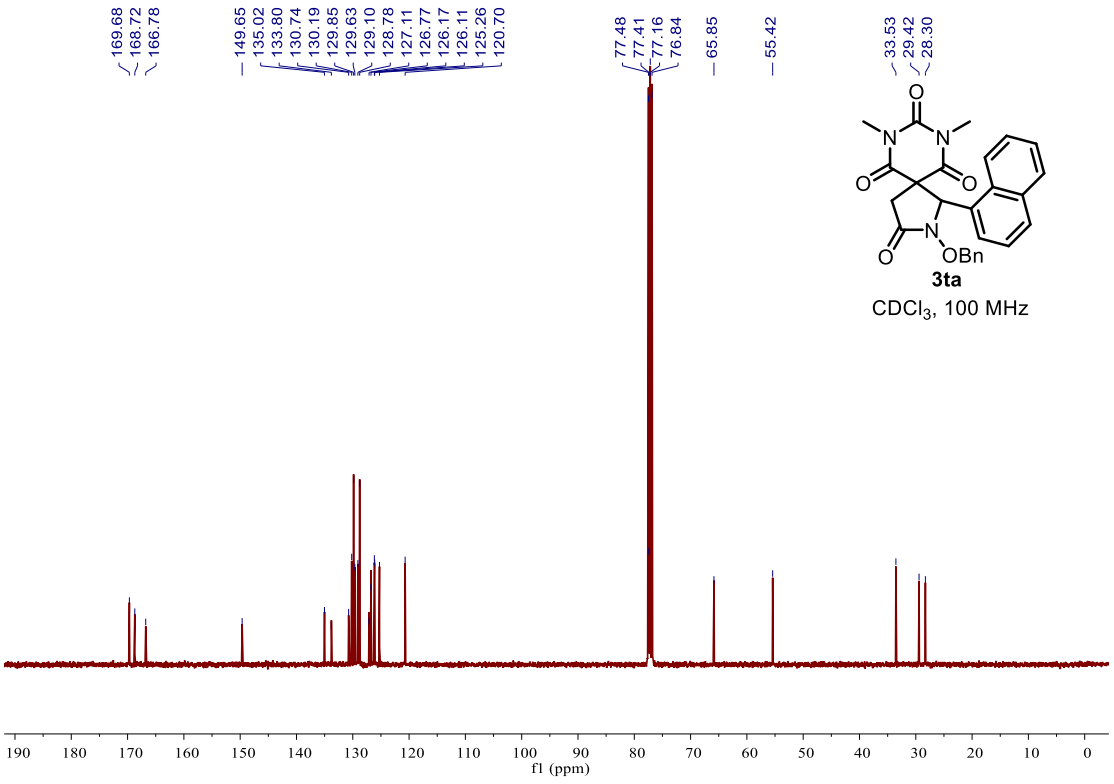

169.29
168.71
167.04
161.78
158.00
150.85
135.01
129.75
128.83
128.52
112.65
104.68
98.18
77.48
77.28
77.16
76.84
63.41
55.72
55.56
54.39
32.53
29.32
28.69
MeO
OMe
OBn
3sa
CDCl3, 100 MHz
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



3ta

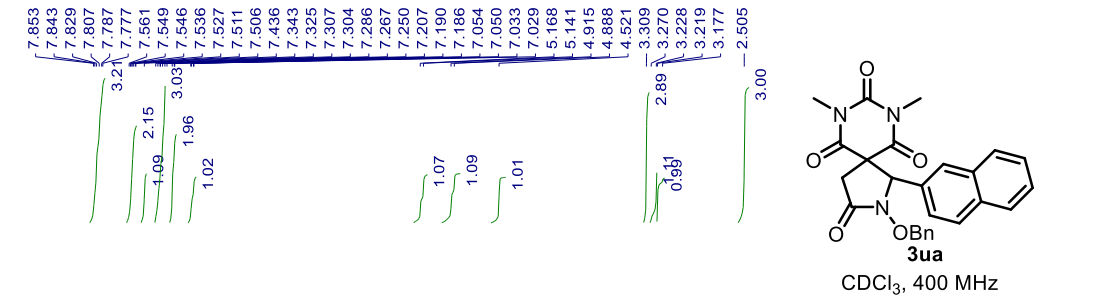
$CDCl_3$, 400 MHz





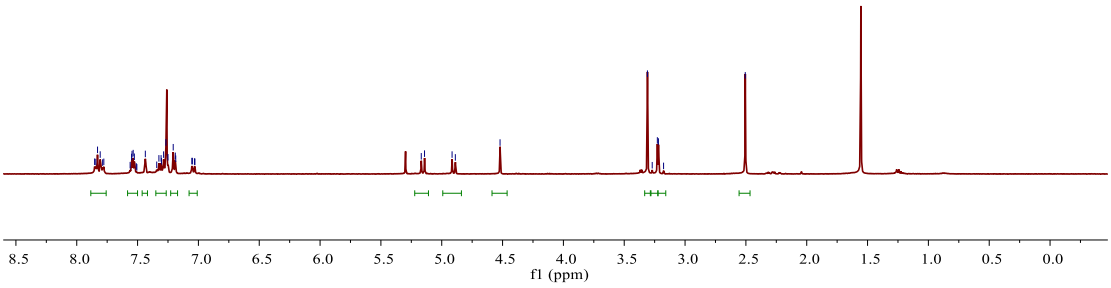
3ta

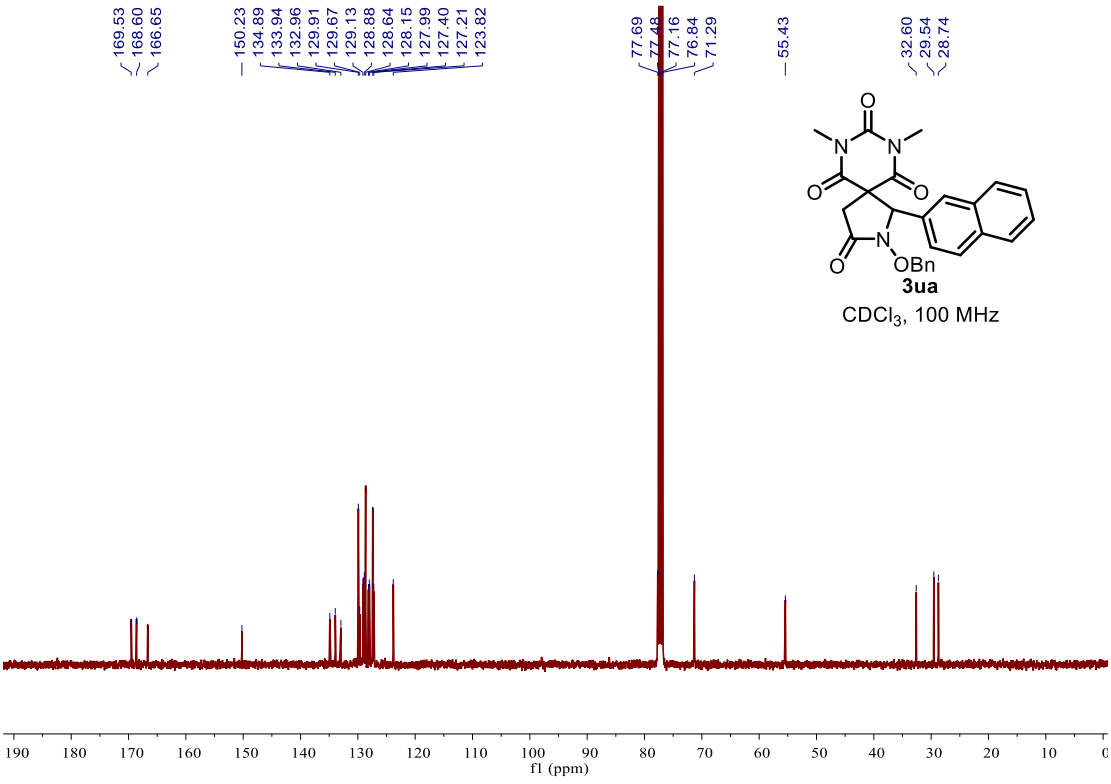
$CDCl_3$, 100 MHz



3ua

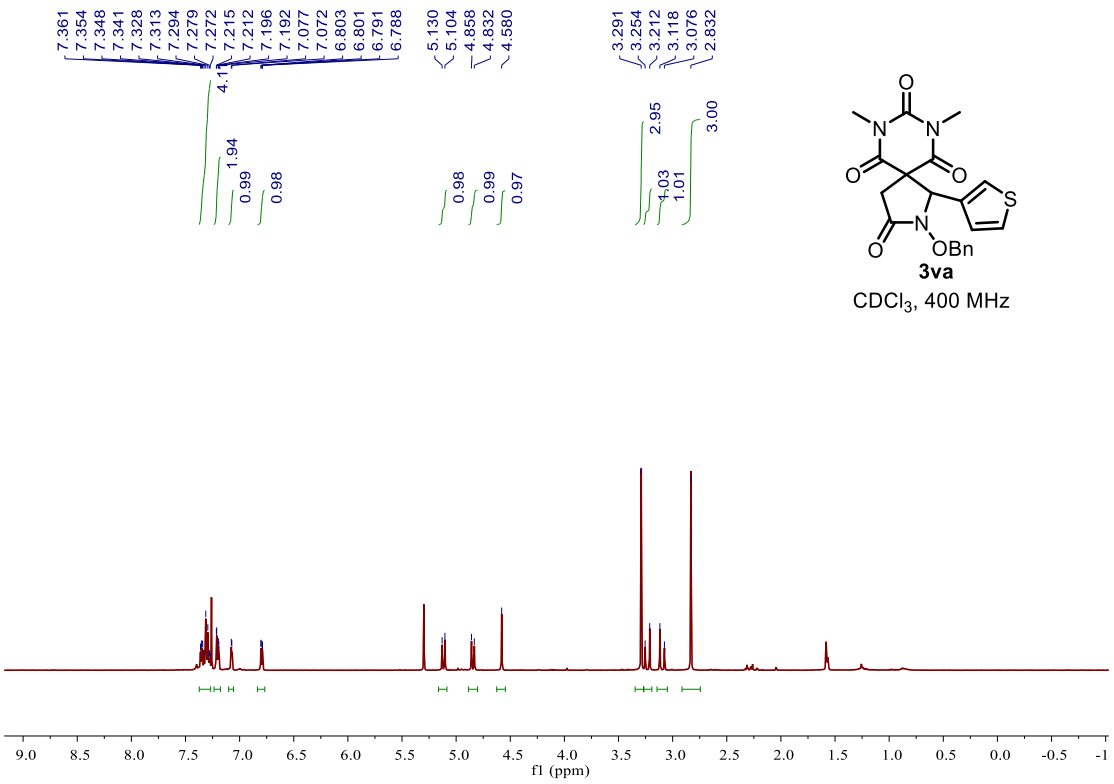
$CDCl_3$, 400 MHz

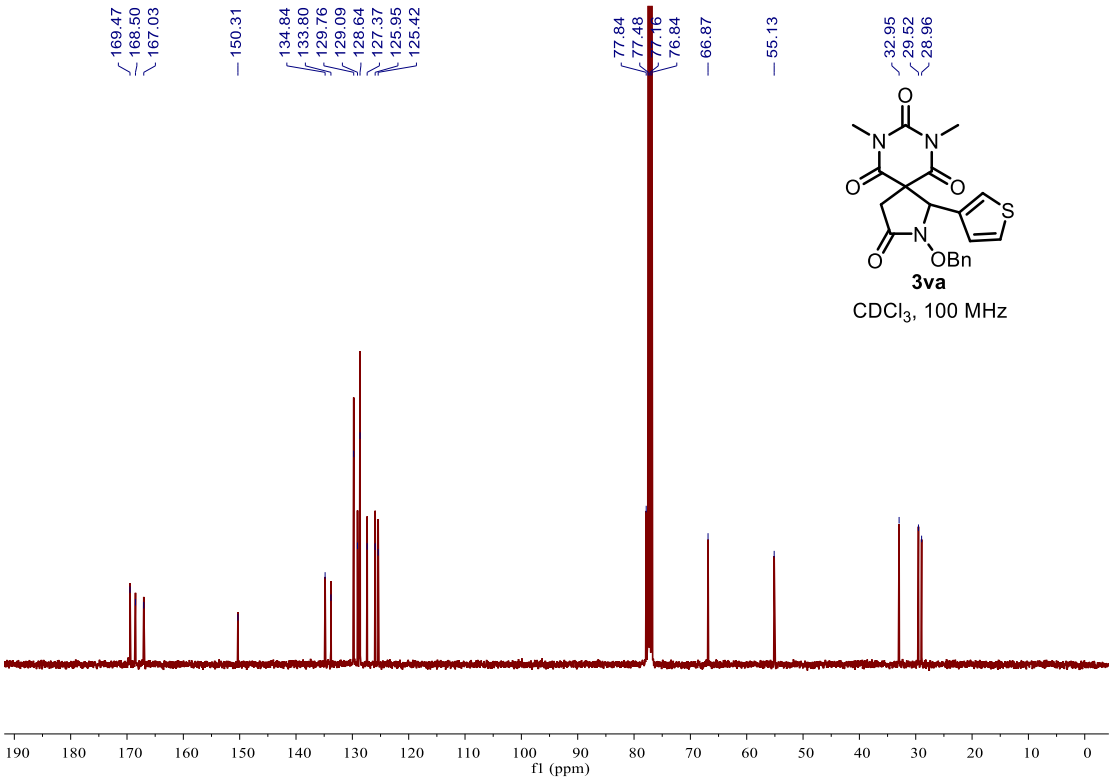


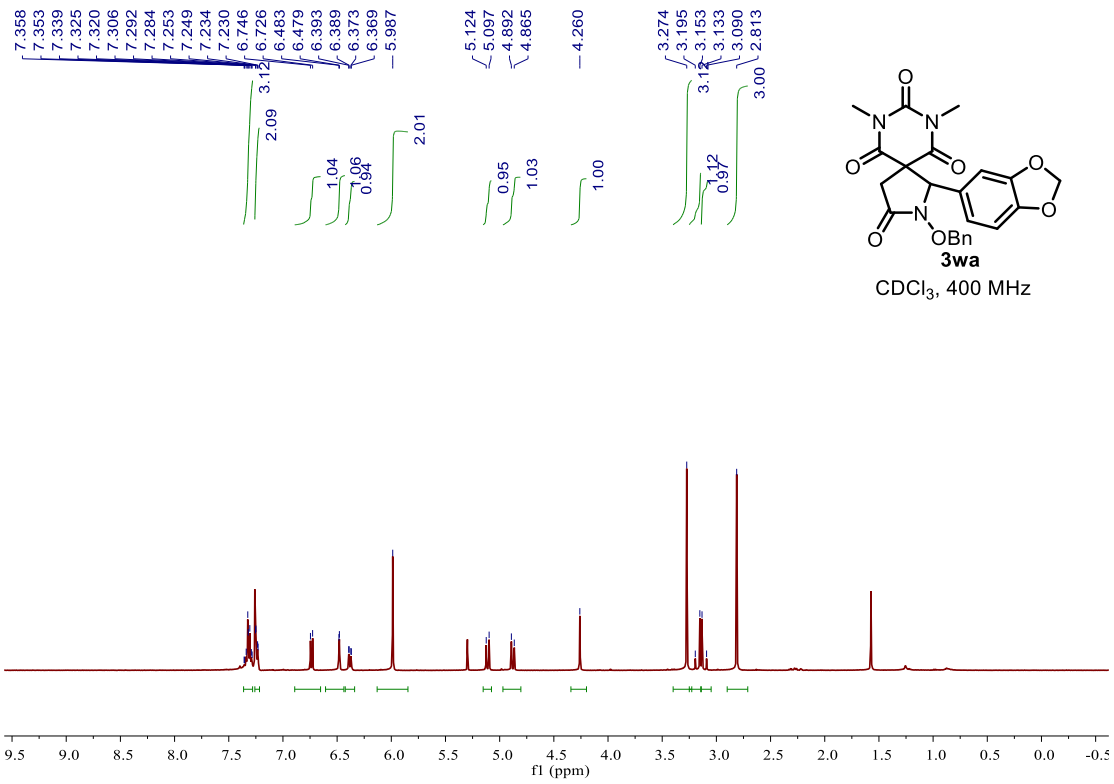


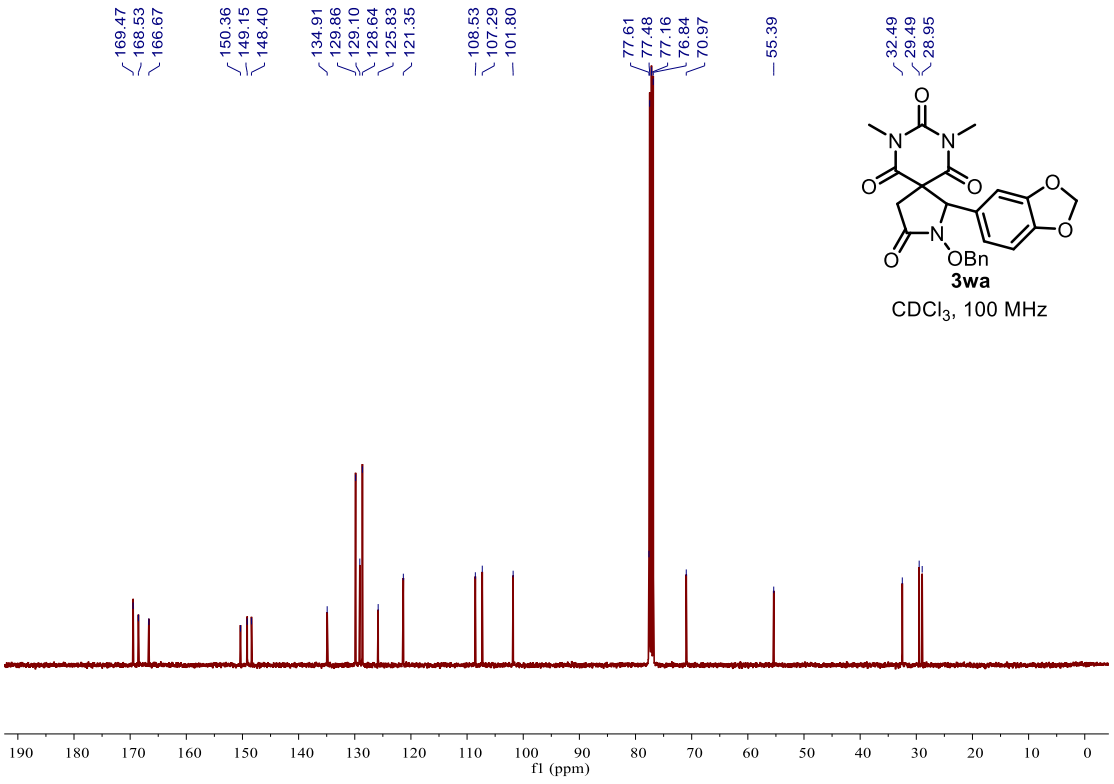
3ua

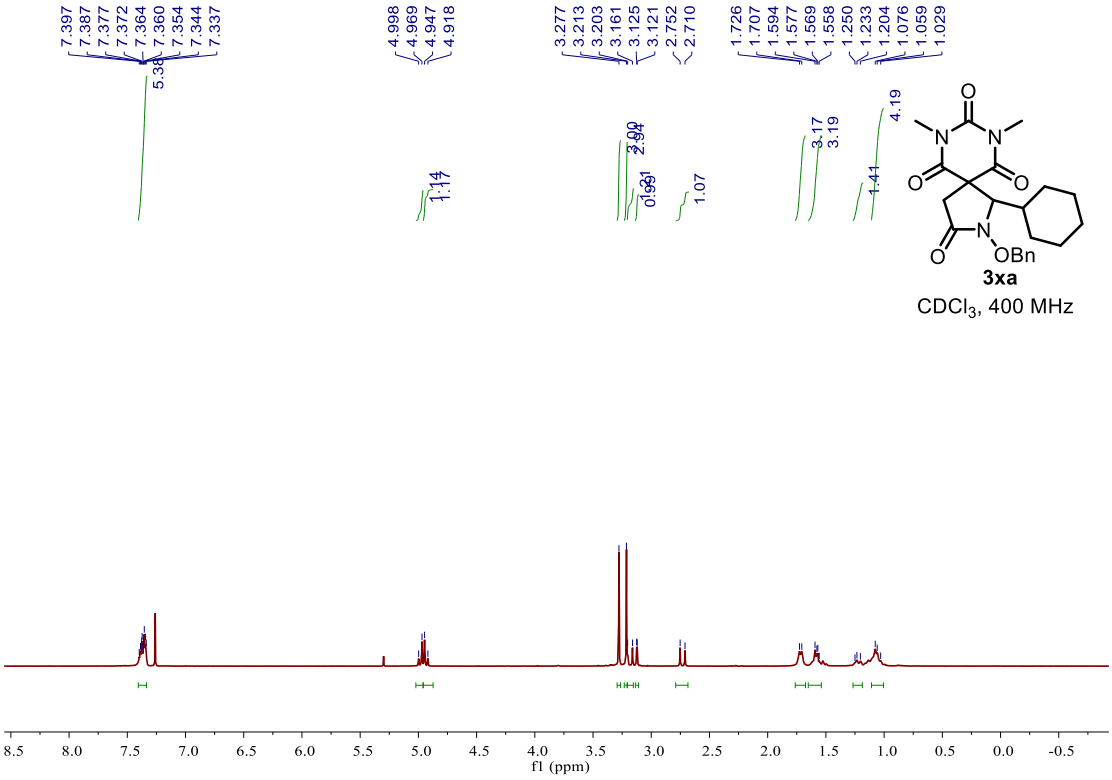
$CDCl_3$, 100 MHz

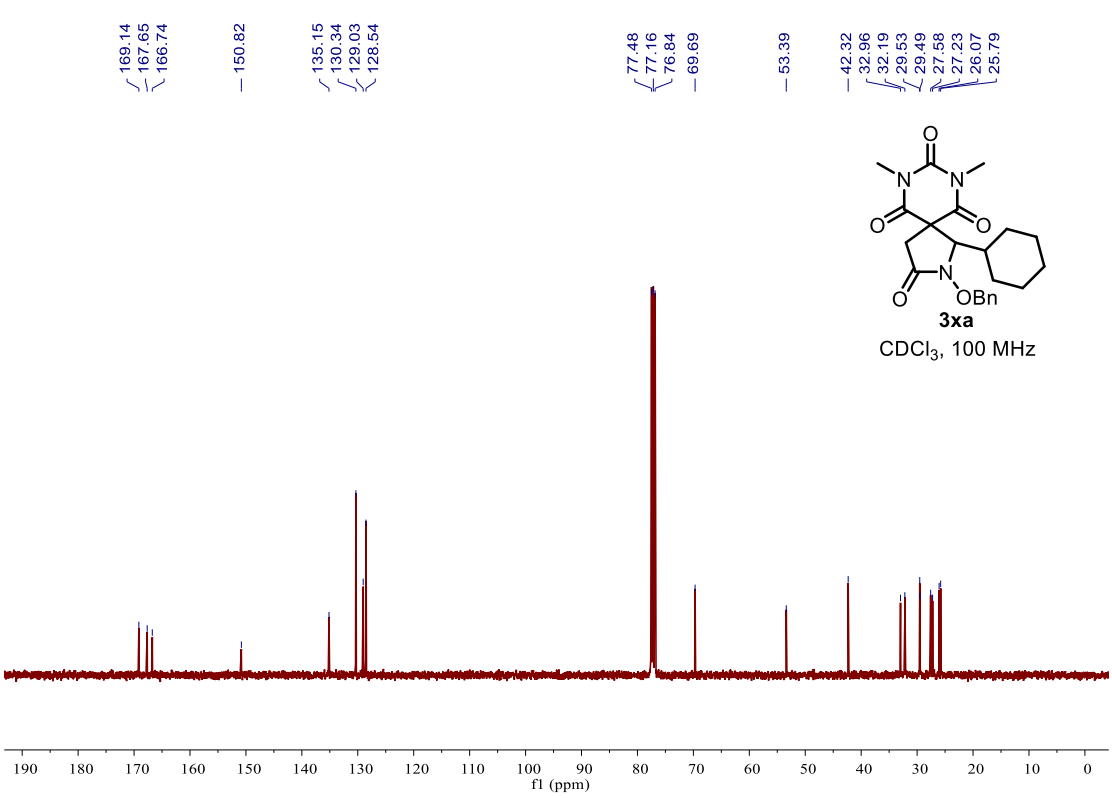

7.361
7.354
7.348
7.341
7.328
7.313
7.294
7.279
7.272
7.215
7.212
7.196
7.192
7.077
7.072
6.803
6.801
6.791
6.788
5.130
5.104
4.858
4.832
4.580
3.291
3.254
3.212
3.118
3.076
2.832
4.11
1.94
0.99
0.98
0.98
0.99
0.97
2.95
1.03
1.01
3.00
O
N
N
O
O
S
N
O
OBn
3va
CDCl3, 400 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1
f1 (ppm)

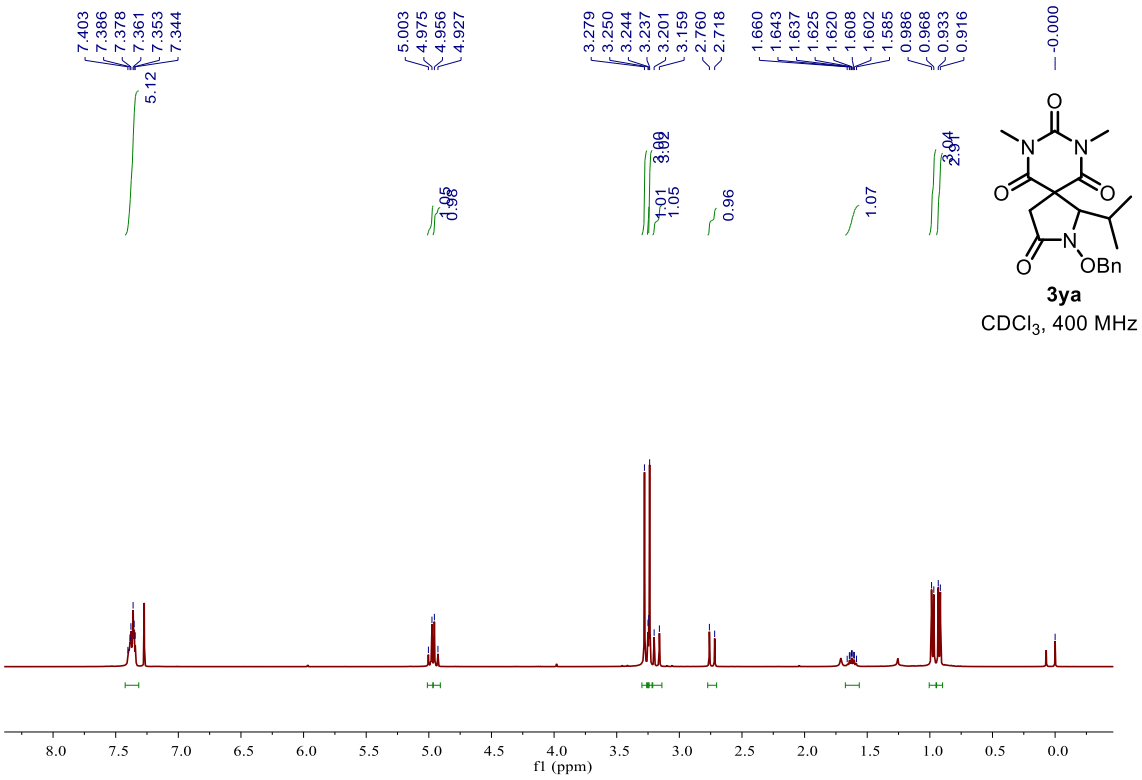

169.47
168.50
167.03
150.31
134.84
133.80
129.76
129.09
128.64
127.37
125.95
125.42
77.84
77.48
77.16
76.84
66.87
55.13
32.95
29.52
28.96
O
N
N
O
O
S
N
O
OBn
3va
CDCl3, 100 MHz
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

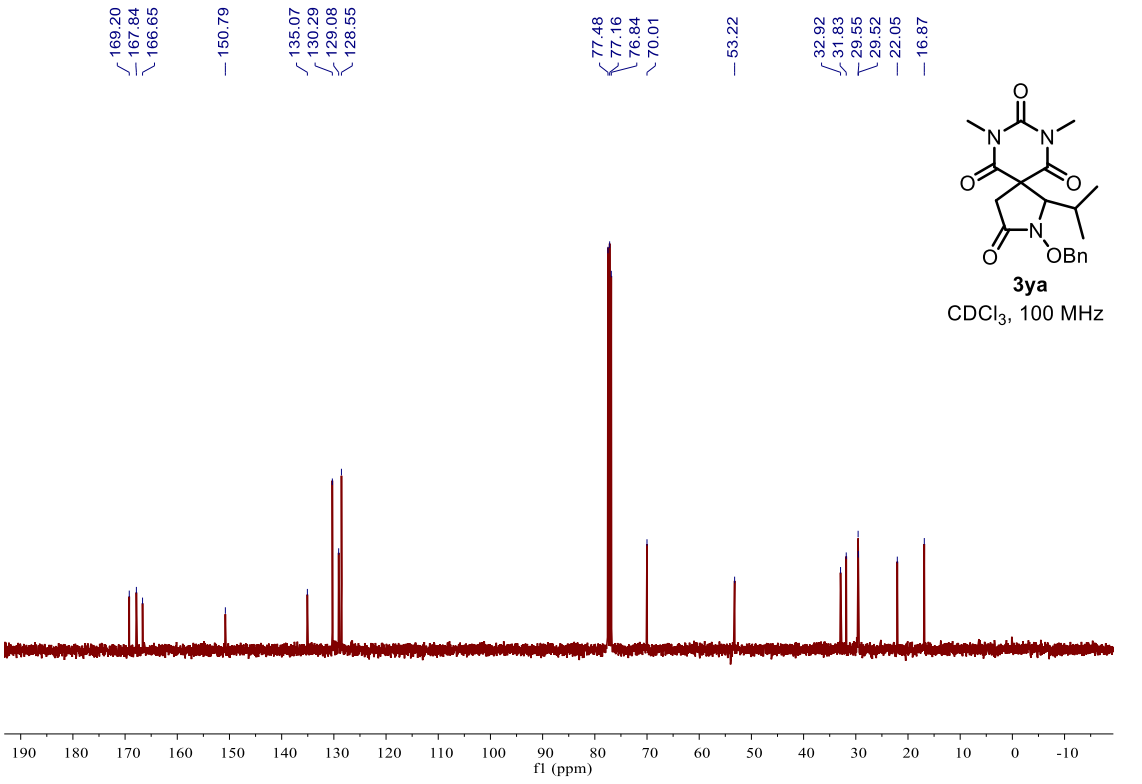

7.358
7.353
7.339
7.325
7.320
7.306
7.292
7.284
7.253
7.249
7.234
7.230
6.746
6.726
6.483
6.479
6.393
6.389
6.373
6.369
5.987
5.124
5.097
4.892
4.865
4.260
3.274
3.195
3.153
3.133
3.090
2.813
3.12
2.09
1.04
1.06
0.94
2.01
0.95
1.03
1.00
3.12
0.97
3.00
O
N
O
O
O
N
O
OBn
3wa
CDCl3, 400 MHz
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

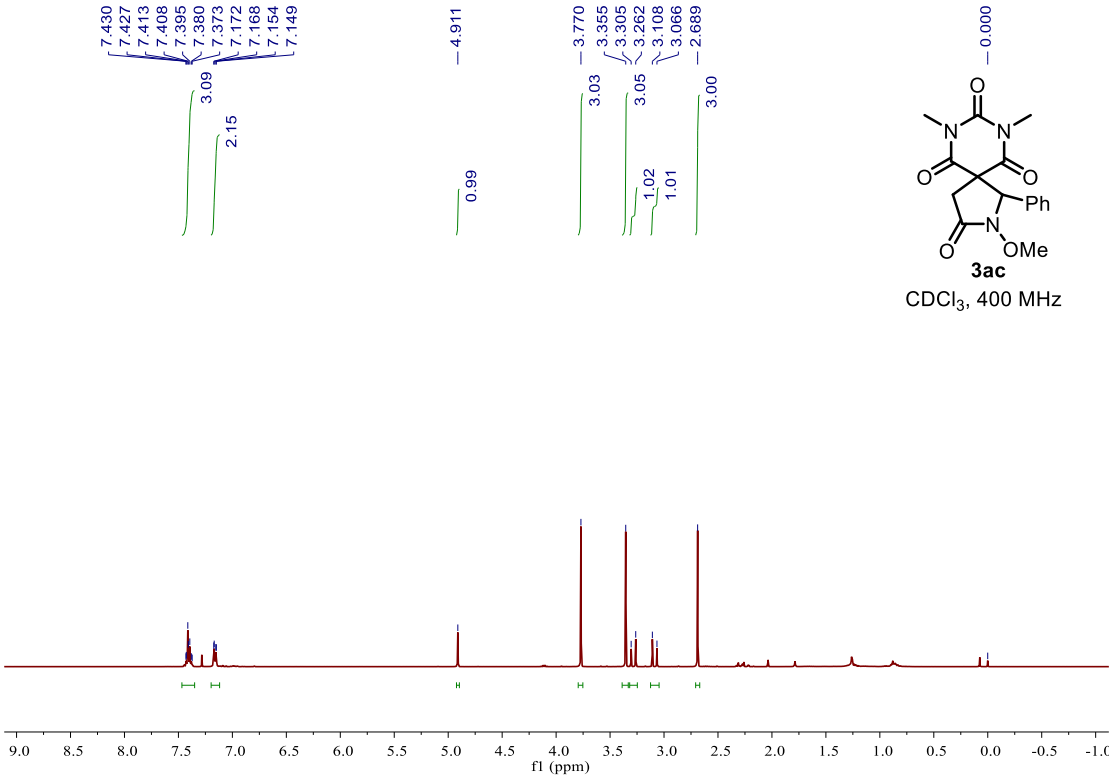

169.47
168.53
166.67
150.36
149.15
148.40
134.91
129.86
129.10
128.64
125.83
121.35
108.53
107.29
101.80
77.61
77.48
77.16
76.84
70.97
55.39
32.49
29.49
28.95
O
N
O
O
O
N
O
OBn
3wa
CDCl3, 100 MHz
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

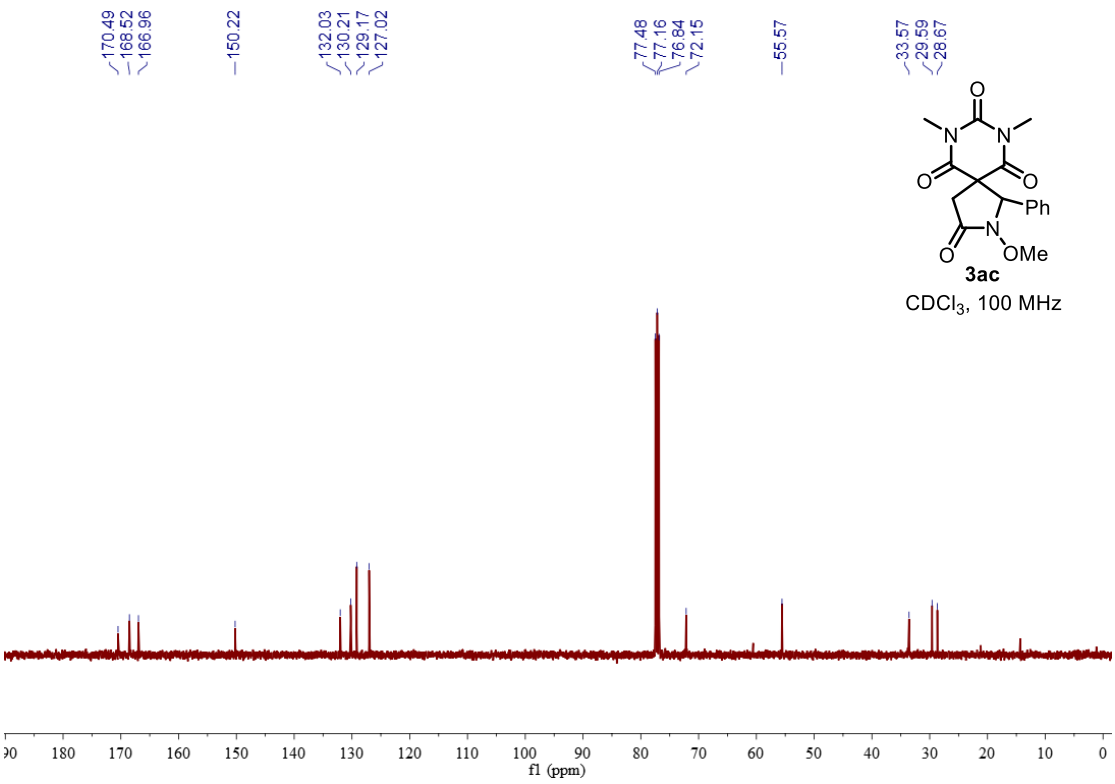

7.397
7.387
7.377
7.372
7.364
7.360
7.354
7.344
7.337
5.38
4.998
4.969
4.947
4.918
3.277
3.213
3.203
3.161
3.125
3.121
2.752
2.710
1.726
1.707
1.594
1.577
1.569
1.558
1.250
1.233
1.204
1.076
1.059
1.029
4.19
3.17
3.19
4.41
1.07
3xa
CDCl3, 400 MHz
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

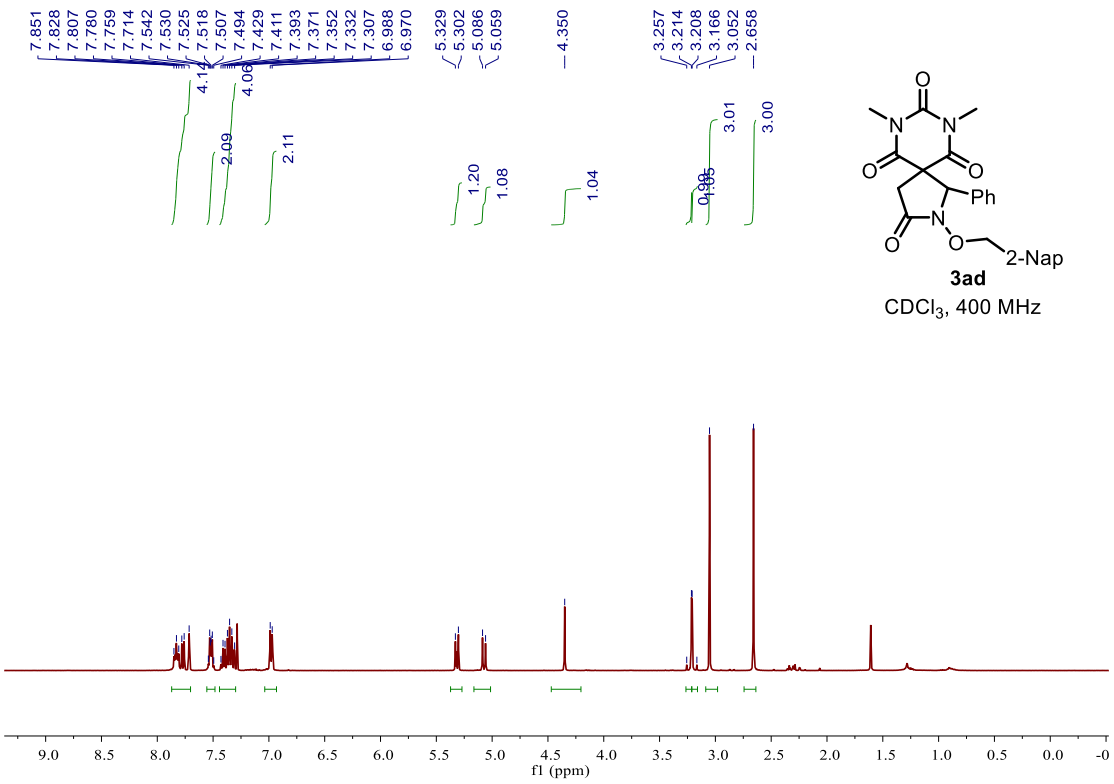

169.14
167.65
166.74
150.82
135.15
130.34
129.03
128.54
77.48
77.16
76.84
69.69
53.39
42.32
32.96
32.19
29.53
29.49
27.58
27.23
26.07
25.79
3xa
CDCl3, 100 MHz
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

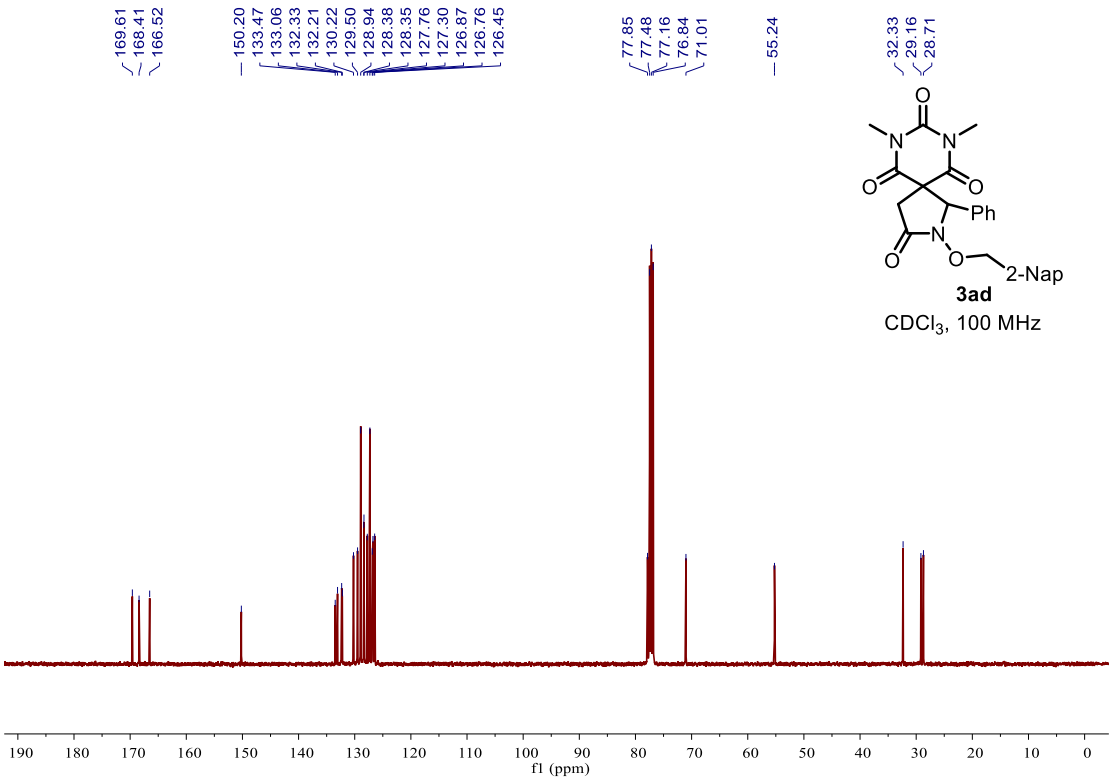

7.403
7.386
7.378
7.361
7.353
7.344
5.12
5.003
4.975
4.956
4.927
3.279
3.250
3.244
3.237
3.201
3.159
2.760
2.718
1.660
1.643
1.637
1.625
1.620
1.608
1.602
1.585
0.986
0.968
0.933
0.916
-0.000
1.05
0.98
3.02
3.00
1.01
1.05
0.96
1.07
3.04
2.91
3ya
CDCl3, 400 MHz
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

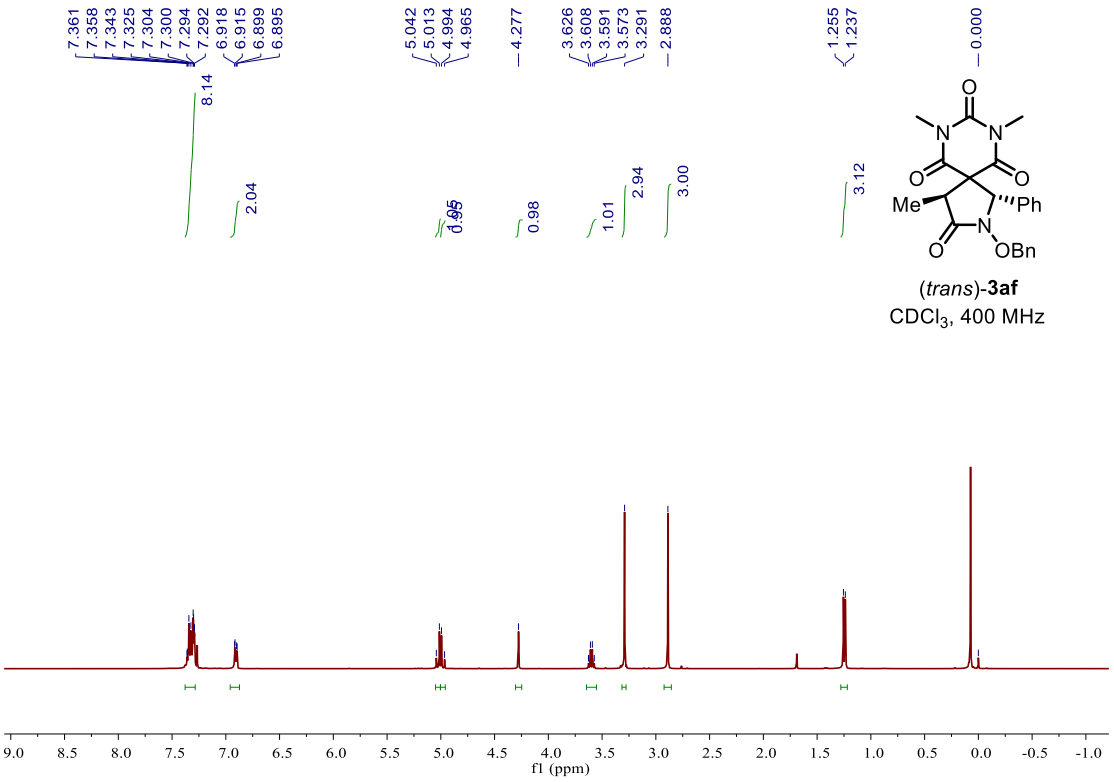

169.20
167.84
166.65
150.79
135.07
130.29
129.08
128.55
77.48
77.16
76.84
70.01
53.22
32.92
31.83
29.55
29.52
22.05
16.87
3ya
CDCl3, 100 MHz
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

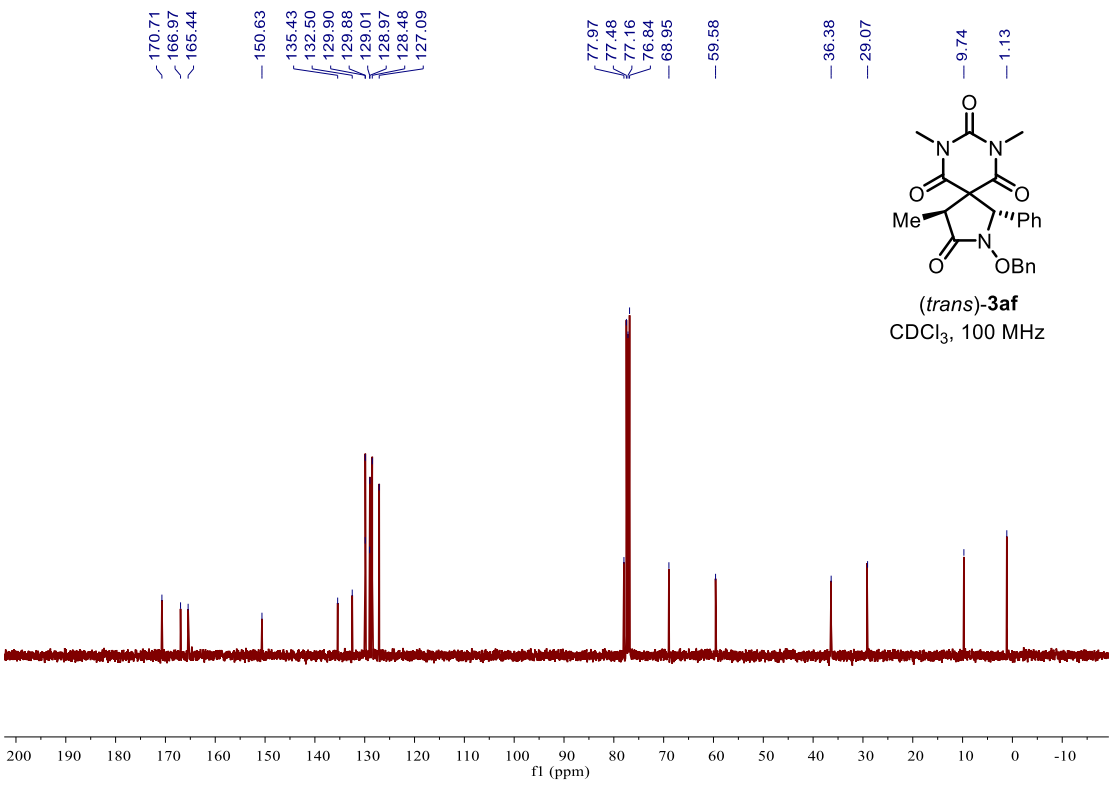

7.430
7.427
7.413
7.408
7.395
7.380
7.373
7.172
7.168
7.154
7.149
4.911
3.770
3.355
3.305
3.262
3.108
3.066
2.689
0.000
3.09
2.15
0.99
3.03
3.05
1.02
1.01
3.00
O
N
N
O
O
Ph
N
O
OMe
3ac
CDCl3, 400 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

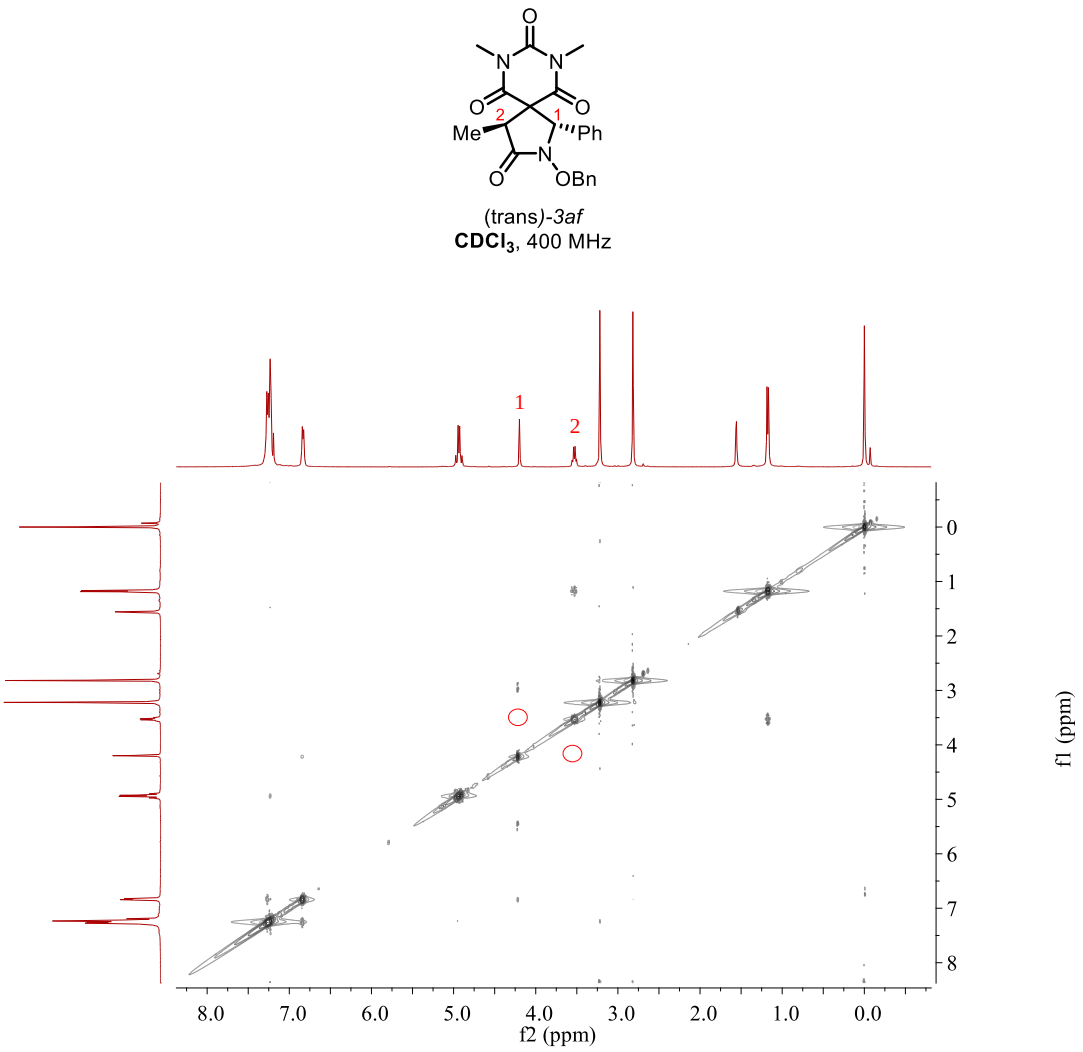

170.49
168.52
166.96
150.22
132.03
130.21
129.17
127.02
77.48
77.16
76.84
72.15
55.57
33.57
29.59
28.67
O
N
N
O
O
Ph
N
O
OMe
3ac
CDCl3, 100 MHz
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.851
7.828
7.807
7.780
7.759
7.714
7.542
7.530
7.525
7.518
7.507
7.494
7.429
7.411
7.393
7.371
7.352
7.332
7.307
6.988
6.970
5.329
5.302
5.086
5.059
4.350
3.257
3.214
3.208
3.166
3.052
2.658
4.12
4.06
2.09
2.11
1.20
1.08
1.04
0.99
0.95
3.01
3.00
O
N
O
Ph
N
O
O
2-Nap
3ad
CDCl3, 400 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

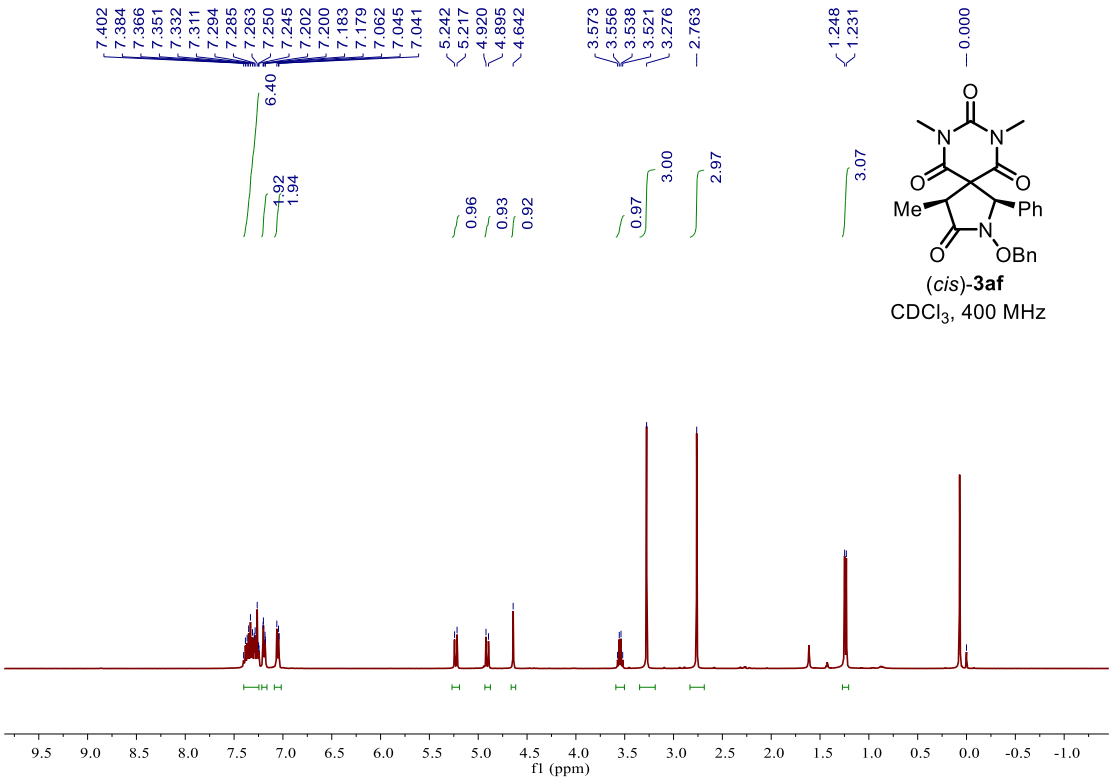

169.61
168.41
166.52
150.20
133.47
133.06
132.33
132.21
130.22
129.50
128.94
128.38
128.35
127.76
127.30
126.87
126.76
126.45
77.85
77.48
77.16
76.84
71.01
55.24
32.33
29.16
28.71
O
N
O
Ph
N
O
O
2-Nap
3ad
CDCl3, 100 MHz
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

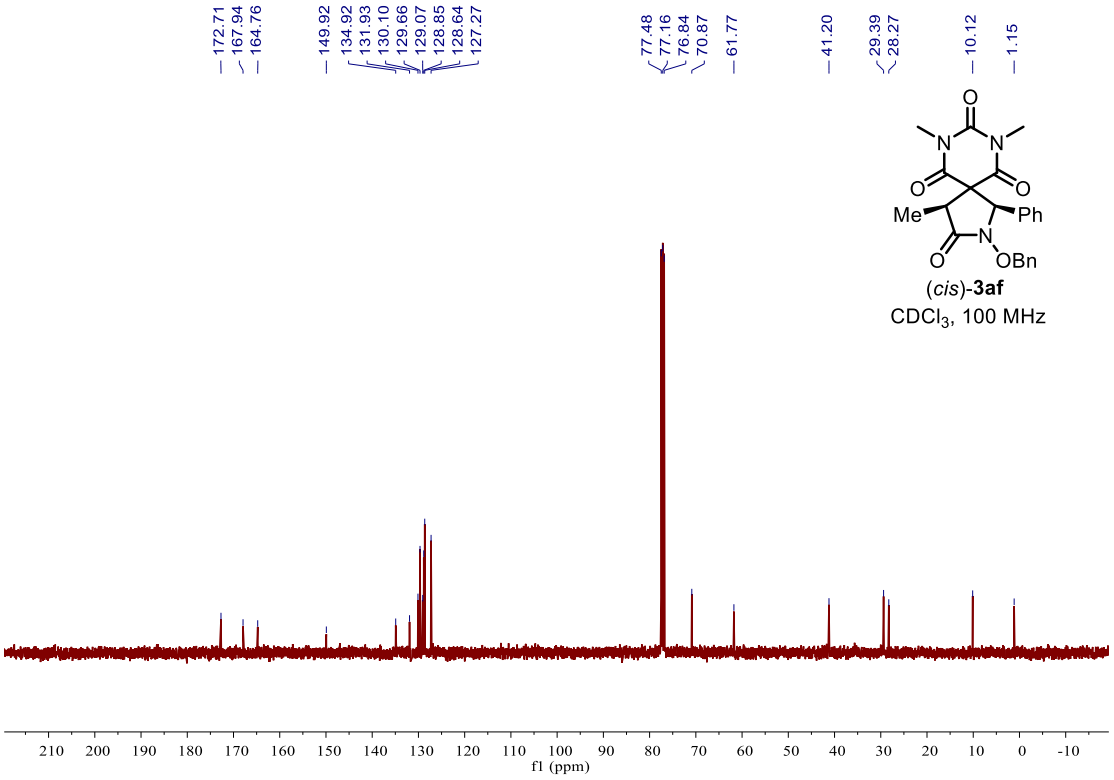

7.361
7.358
7.343
7.325
7.304
7.300
7.294
7.292
6.918
6.915
6.899
6.895
5.042
5.013
4.994
4.965
4.277
3.626
3.608
3.591
3.573
3.291
2.888
1.255
1.237
0.000
8.14
2.04
0.98
1.01
2.94
3.00
3.12
Me
Ph
OBn
(trans)-3af
CDCl3, 400 MHz
f1 (ppm)

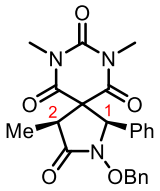

170.71
166.97
165.44
150.63
135.43
132.50
129.90
129.88
129.01
128.97
128.48
127.09
77.97
77.48
77.16
76.84
68.95
59.58
36.38
29.07
9.74
1.13
Me
Ph
OBn
(trans)-3af
CDCl3, 100 MHz
f1 (ppm)



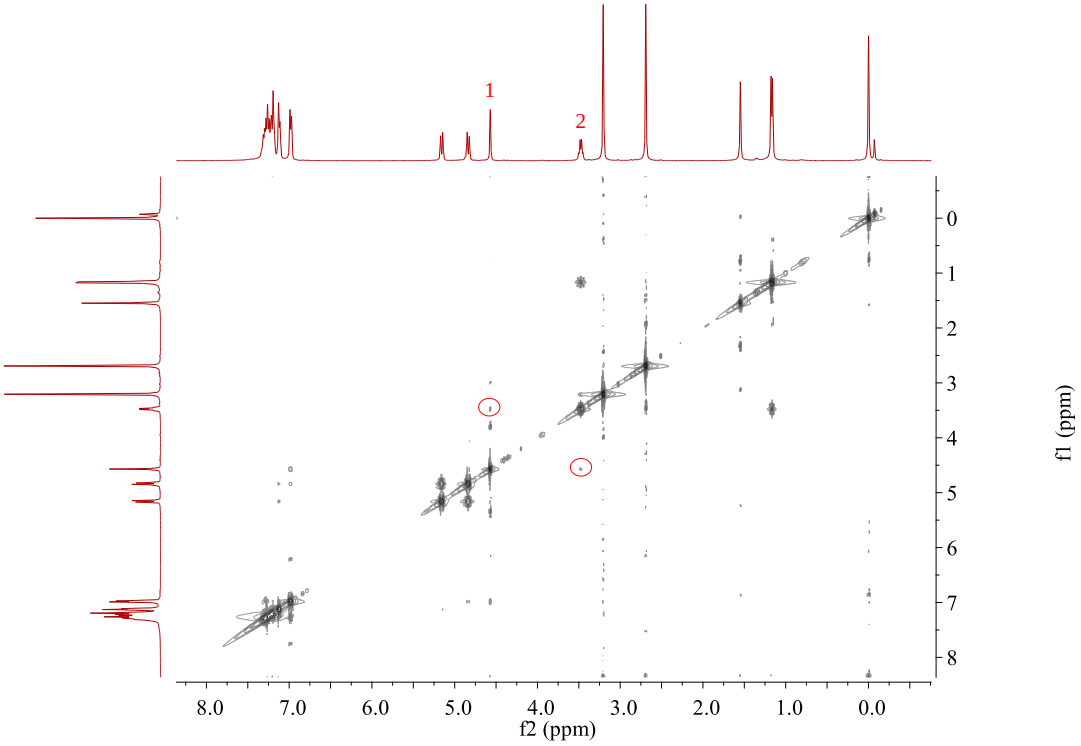
(trans)-*3af*
$CDCl_3$, 400 MHz

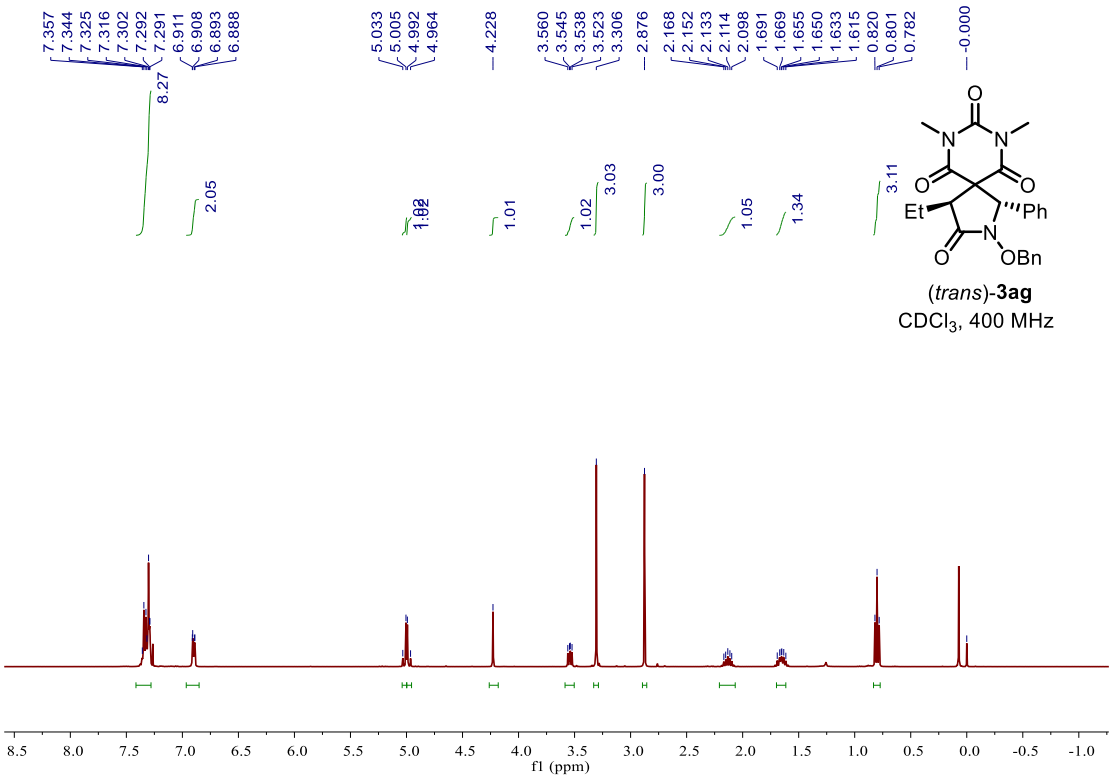

7.402
7.384
7.366
7.351
7.332
7.311
7.294
7.285
7.263
7.250
7.245
7.202
7.200
7.183
7.179
7.062
7.045
7.041
5.242
5.217
4.920
4.895
4.642
3.573
3.556
3.538
3.521
3.276
2.763
1.248
1.231
0.000
6.40
1.92
1.94
0.96
0.93
0.92
0.97
3.00
2.97
3.07
Me
Ph
OBn
(cis)-3af
CDCl3, 400 MHz
f1 (ppm)


172.71
167.94
164.76
149.92
134.92
131.93
130.10
129.66
129.07
128.85
128.64
127.27
77.48
77.16
76.84
70.87
61.77
41.20
29.39
28.27
10.12
1.15
Me
Ph
OBn
(cis)-3af
CDCl3, 100 MHz
f1 (ppm)

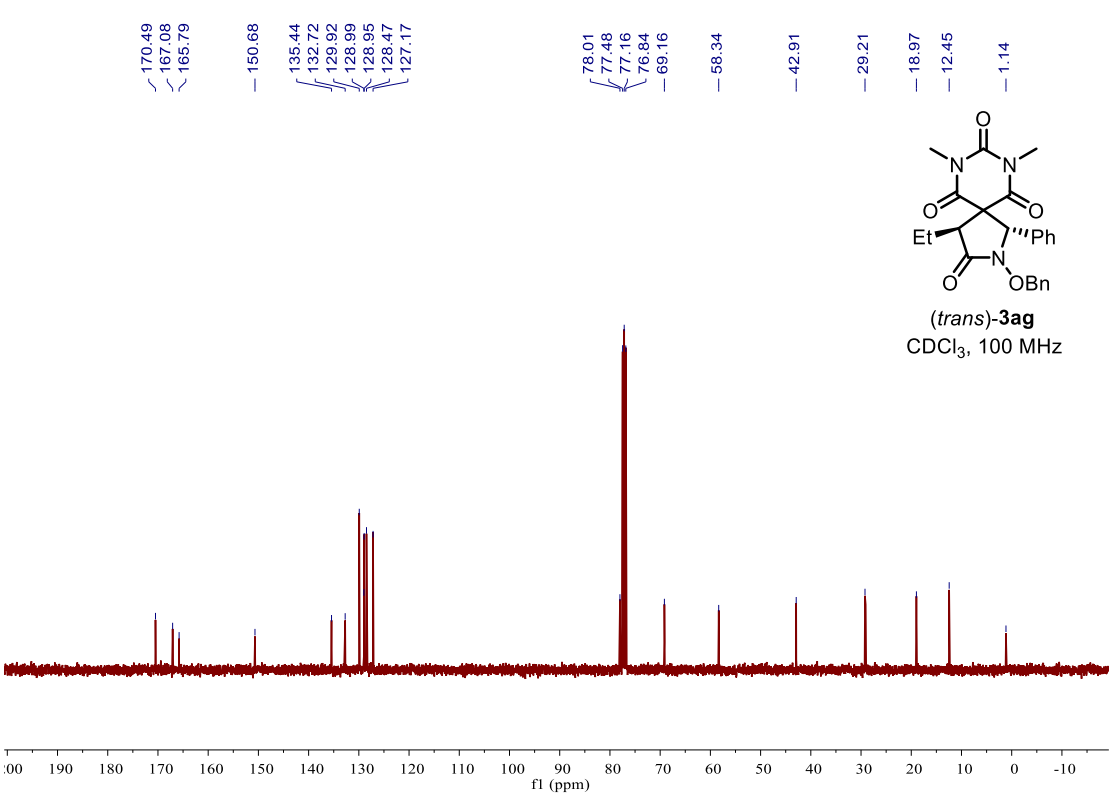


(*cis*)-**3af**
$CDCl_3$, 400 MHz

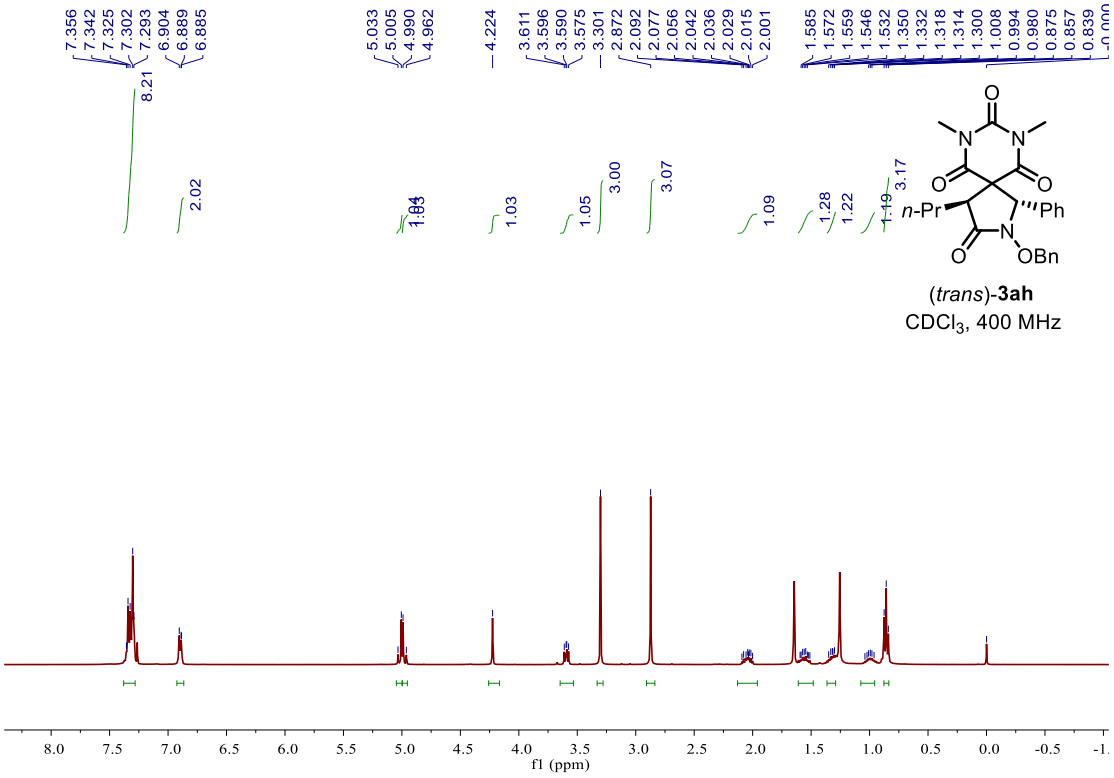




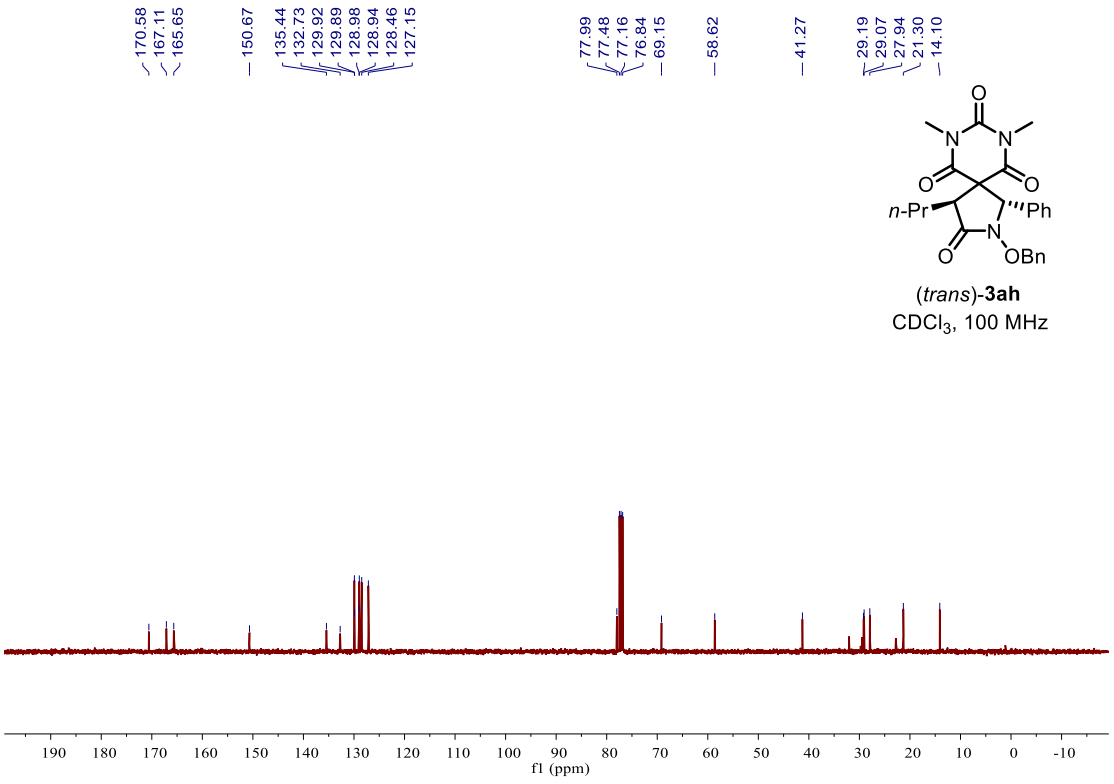
(*trans*)-**3ag**
$CDCl_3$, 400 MHz



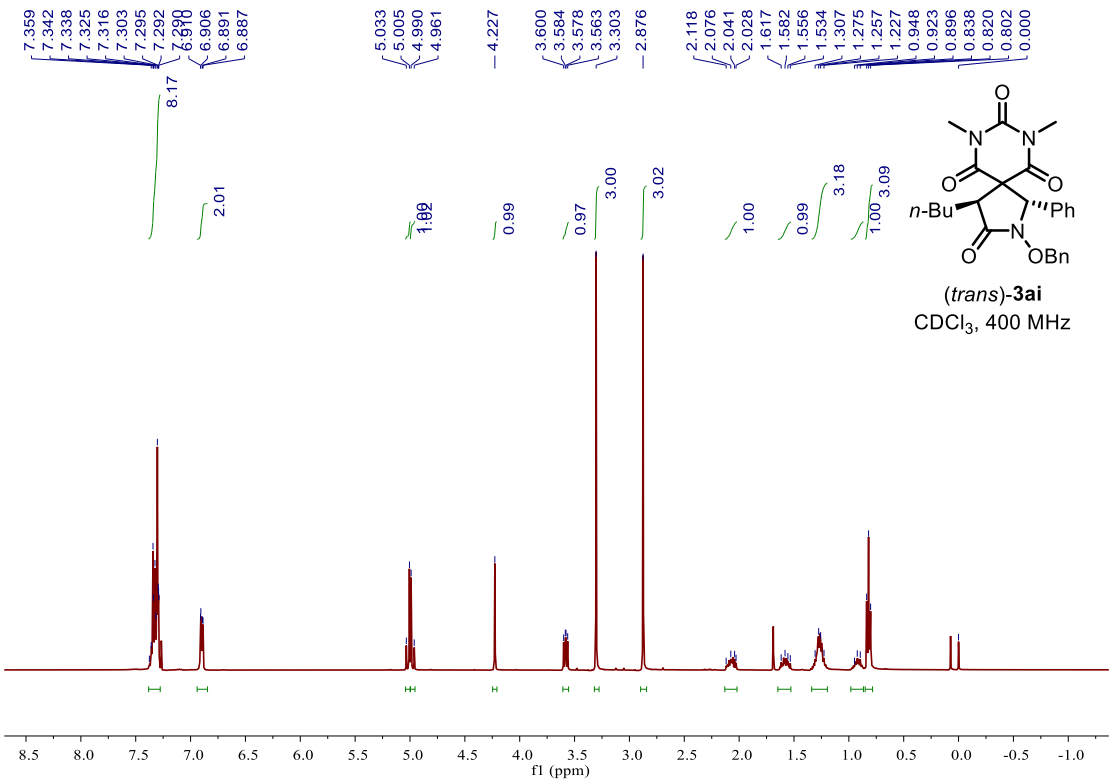
(*trans*)-**3ag**
$CDCl_3$, 100 MHz

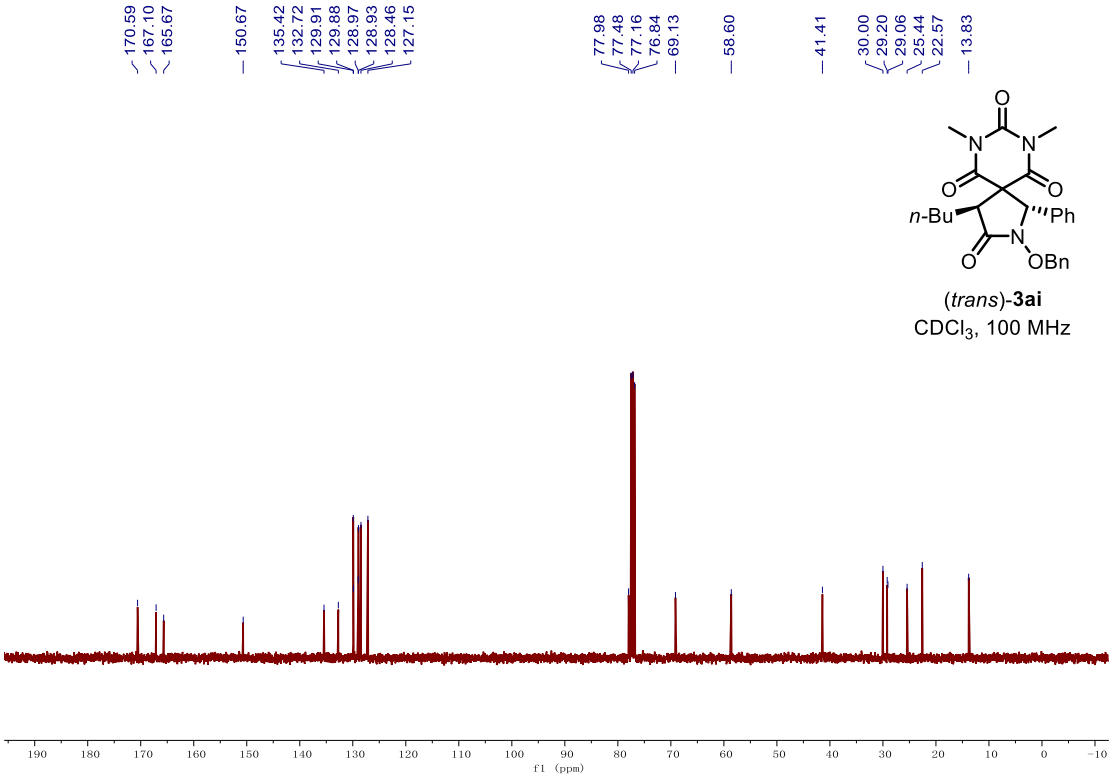


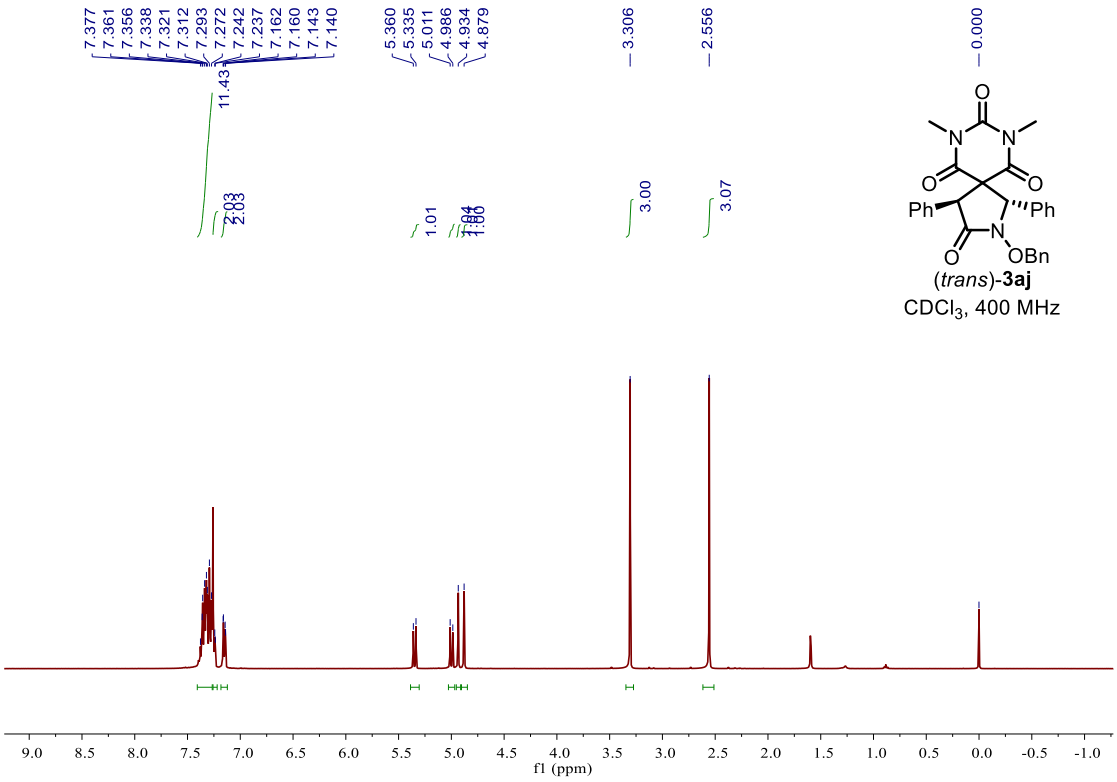
(*trans*)-**3ah**
$CDCl_3$, 400 MHz

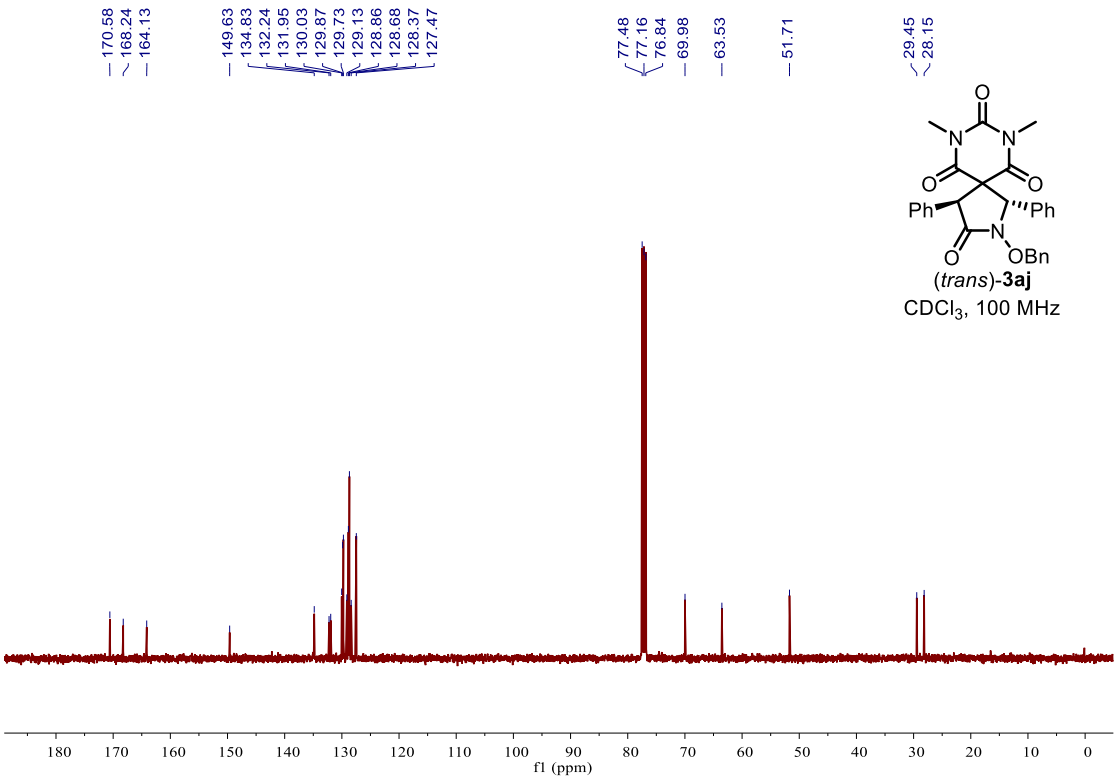


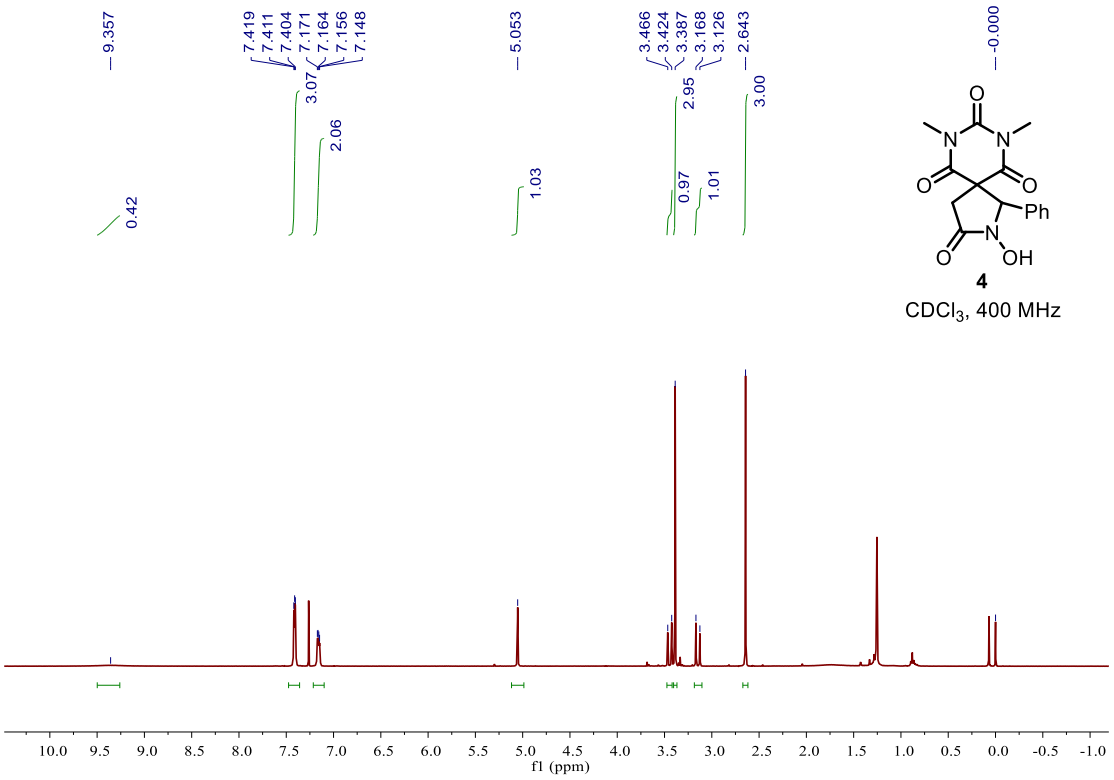
(*trans*)-**3ah**
$CDCl_3$, 100 MHz

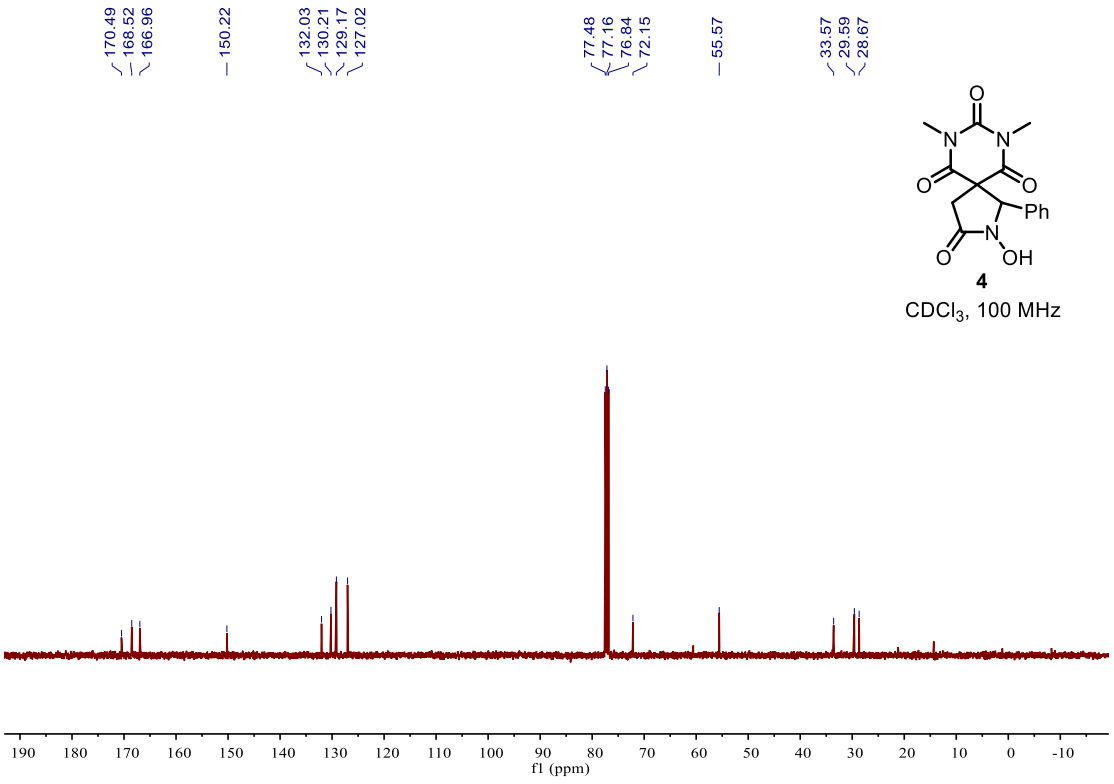

7.359
7.342
7.338
7.325
7.316
7.303
7.295
7.292
6.906
6.891
6.887
5.033
5.005
4.990
4.961
4.227
3.600
3.584
3.578
3.563
3.303
2.876
2.118
2.076
2.041
2.028
1.617
1.582
1.556
1.534
1.307
1.275
1.257
1.227
0.948
0.923
0.896
0.838
0.820
0.802
0.000
8.17
2.01
1.00
0.99
0.97
3.00
3.02
1.00
0.99
3.18
1.00
3.09
n-Bu
Ph
OBn
(trans)-3ai
CDCl3, 400 MHz
f1 (ppm)

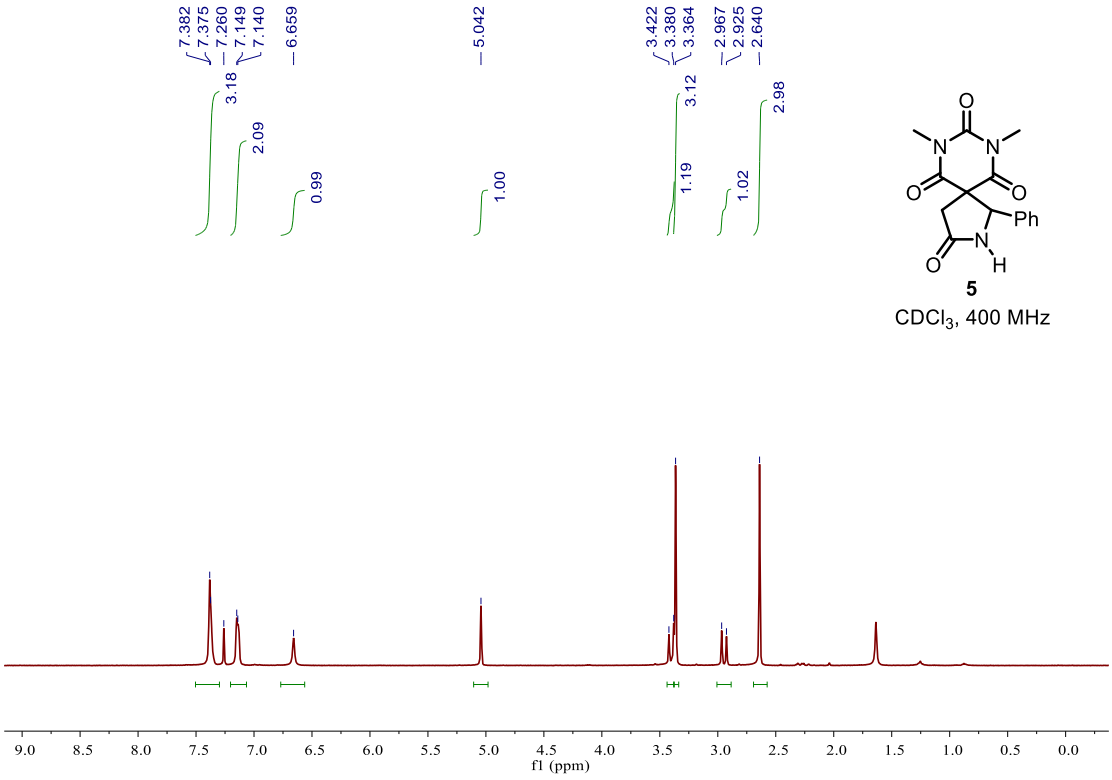

170.59
167.10
165.67
150.67
135.42
132.72
129.91
129.88
128.97
128.93
128.46
127.15
77.98
77.48
77.16
76.84
69.13
58.60
41.41
30.00
29.20
29.06
25.44
22.57
13.83
n-Bu
Ph
OBn
(trans)-3ai
CDCl3, 100 MHz
f1 (ppm)

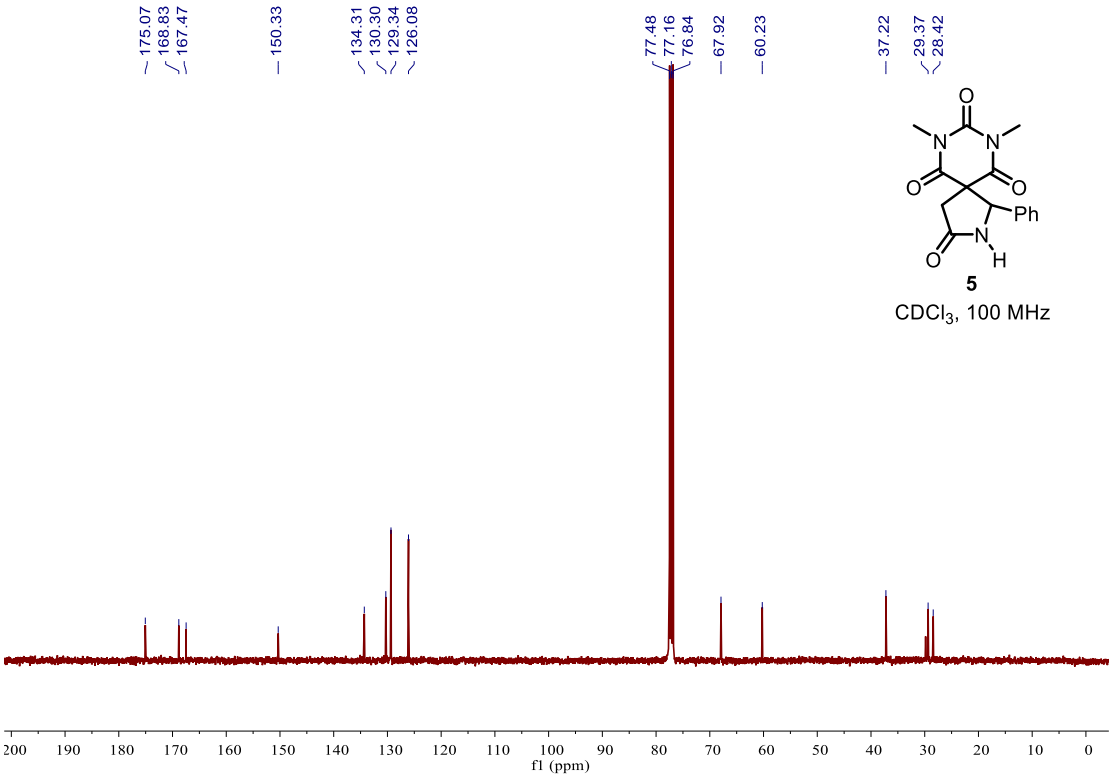

7.377
7.361
7.356
7.338
7.321
7.312
7.293
7.272
7.242
7.237
7.162
7.160
7.143
7.140
5.360
5.335
5.011
4.986
4.934
4.879
3.306
2.556
0.000
11.43
2.03
2.03
1.01
3.00
3.07
Ph
Ph
OBn
(trans)-3aj
CDCl3, 400 MHz
f1 (ppm)


170.58
168.24
164.13
149.63
134.83
132.24
131.95
130.03
129.87
129.73
129.13
128.86
128.68
128.37
127.47
77.48
77.16
76.84
69.98
63.53
51.71
29.45
28.15
Ph
Ph
OBn
(trans)-3aj
CDCl3, 100 MHz
f1 (ppm)


9.357
7.419
7.411
7.404
7.171
7.164
7.156
7.148
5.053
3.466
3.424
3.387
3.168
3.126
2.643
-0.000
0.42
3.07
2.06
1.03
2.95
0.97
1.01
3.00
O
N
N
O
O
Ph
N
OH
O
4
CDCl3, 400 MHz
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


170.49
168.52
166.96
150.22
132.03
130.21
129.17
127.02
77.48
77.16
76.84
72.15
55.57
33.57
29.59
28.67
O
N
N
O
O
Ph
N
OH
O
4
CDCl3, 100 MHz
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.382
7.375
7.260
7.149
7.140
6.659
5.042
3.422
3.380
3.364
2.967
2.925
2.640
3.18
2.09
0.99
1.00
3.12
1.19
1.02
2.98
O
N
N
O
O
Ph
N
H
O
5
CDCl3, 400 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


175.07
168.83
167.47
150.33
134.31
130.30
129.34
126.08
77.48
77.16
76.84
67.92
60.23
37.22
29.37
28.42
O
N
N
O
O
Ph
N
H
O
5
CDCl3, 100 MHz
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)