Supporting Information

Tandem Elimination-Oxidation of Tertiary Benzylic Alcohols with an Oxoammonium Salt

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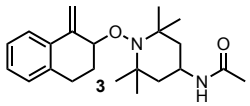
Contents

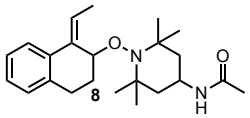
General Experimental Information

All anhydrous reactions were run under a positive pressure of argon. DCM (DCM) was dried by passage through an alumina column.[1] 1,2-Dichloroethane (DCE) was freshly distilled from calcium hydride before use. Silica gel column chromatography was performed using 60 Å silica gel (230−400 mesh). Melting points are uncorrected. The benzylic alcohols used in the study were prepared as reported in the literature.[2]

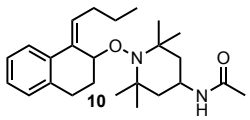
General procedure for allylic oxidation: To a solution of 0.33 mmol of alkene (1 equiv) in acetonitrile (0.33 M), 4 Å molecular sieves was added. The flask was put under an atmosphere of argon. Then 0.40 mmol of *N*-oxoammonium salt (1.2 equiv) was added in one portion. The flask was purged with argon. The reaction mixture was left to stir at room temperature for 6 hours. The reaction was quenched with saturated aqueous bicarbonate solution, extracted with ethyl acetate (3x), and washed with brine (3x). The organic layers were combined, dried over sodium sulfate, and concentrated. The residue was purified by silica chromatography to give the product.



***N*-{2,2,6,6-Tetramethyl-1-[(1-methylidene-3,4-dihydro-2H-naphthalen-2-yl)oxy]piperidin-4-yl} acetamide (3).** Followed general procedure. Purified by silica chromatography using a solvent gradient of 50-70%. EA/hexanes to give product as an off-white solid. When scaled up to 6 mmol obtained 1.32 g of product. Yield: 60%. IR (film): 3246, 3073, 2969, 1633, 1363, 777 cm^{-1}; TLC R_f = 0.30 (70% EA/30% hexanes); mp = 143-145 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.48-7.46 (m, 1H), 7.11-7.01 (m, 3H), 5.44 (s, 1H), 5.25 (bs, 2H), 4.43 (t, *J* = 4.63 Hz, 1H), 4.06- 4.02 (m, 1H), 3.08-3.00 (m, 1H), 2.69 (dt, *J* = 16.7, 5.4, 1H), 2.07-2.03 (m, 2H), 1.86 (s, 3H), 1.75-1.72 (m, 1H), 1.63 (dt, *J* =12.3, 3.6 Hz, 1H), 1.30-1.24 (m, 1H), 1.19-1.17 (m, 7H), 0.97 (s, 3H), 0.87 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 169.3, 144.5, 136.4, 134.3, 128.7, 127.5, 126.0, 125.0, 110.6, 82.0, 60.3, 59.8, 46.3, 46.1, 41.7, 34.4, 34.2, 29.4, 26.2, 23.6, 21.2, 21.0; Anal Calcd for $C_{22}H_{32}N_2O_2$: C, 74.12; H, 9.05; N, 7.86; Found: C, 73.56; H, 9.01; N, 7.75.

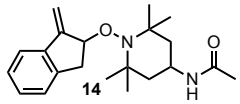


***N*-(1-{[(1*Z*)-1-Ethylidene-3,4-dihydro-2H-naphthalen-2-yl]oxy}-2,2,6,6-tetramethylpiperidin-4-yl) acetamide (8).** Followed general procedure. Purified by silica chromatography using a solvent gradient of 50-70%. EA/hexanes to give 0.070 g product as a white solid. Yield: 57%. IR (film) 3254, 3091, 2921, 1637, 1374, 952, 769 cm^{-1}; TLC R_f = 0.31 (70% EA/30% hexanes); mp = 141-143 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.36-7.34 (m, 1H), 7.06-7.00 (m, 3H), 5.96 (q, *J* = 6.7 Hz, 1H), 5.09 (d, *J* = 6.7 Hz, 1H), 4.91 (bs, 1H), 4.03-3.95 (m, 1H), 3.12-3.04 (m, 1H), 2.63 (dd, *J* = 16.5, 5.4 Hz, 1H), 2.28-2.25 (m, 1H), 1.87-1.86 (m, 3H), 1.85 (s, 3H), 1.75-1.59 (m, 5H), 1.26 (s, 3H), 1.15 (s, 3H), 0.80 (s, 3H), 0.78 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 169.2, 137.6, 136.1, 135.5, 128.5, 126.4, 125.9, 124.6, 121.7, 74.8, 60.6, 59.1, 46.4, 46.1, 41.2, 33.8, 33.6, 28.8, 24.8, 23.6, 20.9, 20.7, 14.5; Anal Calcd for $C_{23}H_{34}N_2O_2$: C, 74.55; H, 9.25; N, 7.56; Found: C, 74.34; H, 9.30; N, 7.25. Alkene geometry was determined to be Z via NOESY NMR.

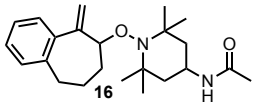


***N*-(1-{[(1Z)-1-Butylidene-3,4-dihydro-2H-naphthalen-2-yl]oxy}-2,2,6,6-tetramethylpiperidin-4-yl) acetamide (10).** Followed general procedure. Purified by silica chromatography using a solvent gradient of 50-70%. EA/hexanes to give 0.075 g product as an oil. Yield: 56%. IR (film) 3272, 2925, 2868, 1640, 1550, 1373, 1362, 777 cm^{-1}; TLC R_f = 0.32 (70% EA/30% hexanes); ^1H NMR (400 MHz, $CDCl_3$) δ 7.54-7.42 (m, 1H), 7.13-7.07 (m, 3H), 5.93 (t, *J* = 7.4 Hz, 1H), 5.33 (d, *J* = 7.2 Hz, 1H), 4.94 (s, 1H), 4.14-4.01 (m, 1H),

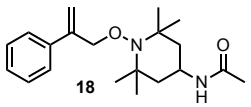
3.19-3.11 (m, 1H), 2.69 (dd, *J* = 5.6, 16.4 Hz, 1H), 2.46-2.39 (m, 1H), 2.27-2.25 (m, 2H), 1.91 (s, 3H), 1.81-1.72 (m, 3H), 1.67-1.64 (m, 1H), 1.58-1.42 (m, 3H), 1.31 (s, 3H), 1.22 (s, 3H), 0.96 (t, *J* = 7.2 Hz, 3H), 0.87 (s, 3H), 0.83 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 169.2, 136.8, 136.2, 135.7, 128.5, 127.8, 126.4, 125.9, 124.8, 75.1, 60.6, 59.1, 46.4, 46.1, 41.2, 33.9, 33.7, 30.8, 28.9, 24.8, 23.6, 23.0, 20.9, 20.7, 14.1; Anal Calcd for $C_{25}H_{38}N_2O_2$: C, 75.33; H, 9.61; N, 7.03; Found: C, 75.31; H, 9.50; N, 6.87.



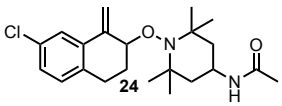
***N*-{2,2,6,6-Tetramethyl-1-[(1-methylidene-2,3-dihydroinden-2-yl)oxy]piperidin-4-yl}acetamide (14).** Followed general procedure. Purified by silica chromatography using a solvent gradient of 50-70%. EA/hexanes to give 0.061 g product as a solid. Yield: 53%. IR (film) 3267, 2924, 1637, 1362, 732 cm^{-1}; TLC R_f = 0.23 (70% EA/30% hexanes); mp = 150-153 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45-7.43 (m, 1H), 7.21 (s, 3H), 5.55 (d, *J* =1.7 Hz, 1H), 5.42 (s, 1H), 5.22 (d, *J* = 6.9 Hz, 1H), 5.11 (t, *J* = 6.9 Hz, 1H), 4.21-4.12 (m, 1H), 3.22 (dd, *J* = 7.5, 15.5 Hz, 1H), 3.06 (dd, *J* = 6.8, 15.5 Hz, 1H), 1.95 (s, 3H), 1.83-1.81 (m, 2H), 1.38-1.34 (m, 5H), 1.27-1.23 (m, 9H);$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 169.2, 149.7, 142.1, 139.1, 128.7, 126.9, 125.2, 120.8, 105.9, 87.1, 61.0, 59.5, 46.4, 46.3, 41.1, 38.7, 34.5, 33.0, 23.6, 21.2, 21.0; Anal Calcd for $C_{21}H_{30}N_2O_2$: C, 73.65; H, 8.83; N, 8.18; Found: C, 73.62; H, 8.55; N, 7.80.



***N*-[2,2,6,6-Tetramethyl-1-({5-methylidene-6,7,8,9-tetrahydrobenzo[7]annulen-6-yl}oxy)piperidin-4-yl] acetamide (16).** Followed general procedure. Purified by silica chromatography using a solvent gradient of 50-70%. EA/hexanes to give 0.058 g product as an oil. Yield: 47%. IR (film) 3267, 3081, 2971, 1636, 1550, 1362, 778 cm^{-1}; TLC R_f = 0.23 (70% EA/30% hexanes); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20-7.13 (m, 3H), 7.07-7.06 (m, 1H), 5.31 (s,1H), 5.18 (d, *J* = 3.8 Hz, 1H), 5.07 (s, 1H), 4.31 (bs, 1H), 4.14-4.07 (m, 2H), 2.79-2.69 (m, 2H), 2.21 (bs, 1H), 2.13-2.03 (m, 2H), 1.92 (s, 4H), 1.79-1.68 (m, 3H), 1.29-1.23 (m, 2H), 1.20 (s, 4H), 1.16 (s, 2H), 1.12 (s, 2H), 1.09 (s, 2H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 169.4, 153.6, 141.6, 140.1, 129.0, 128.6, 127.1, 126.0, 112.9, 86.2, 60.6, 59.4, 46.3, 41.0, 37.5, 35.9, 34.8, 34.6, 33.8, 23.9, 23.5, 21.1, 20.9; Anal Calcd for $C_{23}H_{34}N_2O_2$: C, 74.55; H, 9.25; N, 7.56; Found: C, 74.34; H, 9.30; N, 7.25.

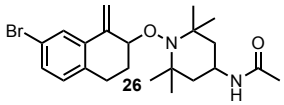


***N*-{2,2,6,6-Tetramethyl-1-[(2-phenylprop-2-en-1-yl)oxy]piperidin-4-yl}acetamide (18).** Followed general procedure. Purified by silica chromatography using a solvent gradient of 50-70% EA/hexanes to give product as colorless oil. Yield: 45%. IR (film) 3273, 3081, 2973, 1633, 1550, 705 cm^{-1}; TLC R_f = 0.26 (70% EA/30% hexanes); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.12 (m, 5H), 5.38 (s, 1H), 5.33 (s, 1H), 5.19 (bs, 1H), 4.57 (s, 1H), 4.09-4.04 (m, 1H), 1.88-1.71 (m, 5H), 1.29-1.23 (m, 3H), 1.16-1.15 (m, 12H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 169.3, 144.2, 139.4, 128.3, 127.6, 126.0, 112.7, 78.4, 60.13, 45.8, 41.1, 32.9, 23.6, 20.9; Anal Calcd for $C_{20}H_{30}N_2O_2$: C, 72.69; H, 9.15; N, 8.48; Found: C, 72.48; H, 8.79; N, 8.46.

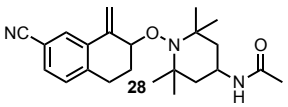


***N*-{1-[(7-Chloro-1-methylidene-3,4-dihydro-2H-naphthalen-2-yl)oxy]-2,2,6,6-tetramethylpiperidin-4-yl}acetamide (24).** Followed general procedure. Purified by silica chromatography using a solvent gradient of 50-70%. EA/hexanes to give 0.062 g product as a white solid. Yield: 48%. IR (film) 3255, 2970, 1634, 1362 cm^{-1}; TLC R_f = 0.25 (70% EA/30% hexanes); mp = 102-105 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, *J* = 2.1 Hz, 1H), 7.13 (dd, *J* = 2.1, 8.2 Hz, 1H), 7.04 (d, *J* = 8.2 Hz, 1H), 5.51 (s, 1H), 5.28 (s, 1H), 5.14 (d, *J* = 6.9 Hz, 1H), 4.49 (d, *J* = 3.5 Hz, 1H), 4.15-4.07 (m, 1H), 3.11-3.03 (m, 1H), 2.75-2.68 (m, 1H), 2.17- 2.14 (m,1H),

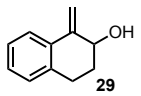
2.07-2.03 (m, 1H), 1.93 (s, 3H), 1.89- 1.69 (m, 4H), 1.24 (m, 6H), 1.03 (s, 3H), 0.91 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 169.3, 143.4, 135.9, 134.8, 131.7, 130.1, 127.5, 124.8, 112.1, 81.6, 60.5, 59.8, 46.2, 46.1, 41.1, 34.5, 34.2, 29.1, 25.5, 23.6, 21.2, 20.9; Anal calcd for $C_{22}H_{31}ClN_2O_2$: C, 67.59; H, 7.99; N, 7.17; Found: C, 67.26; H, 8.02; N, 7.33.



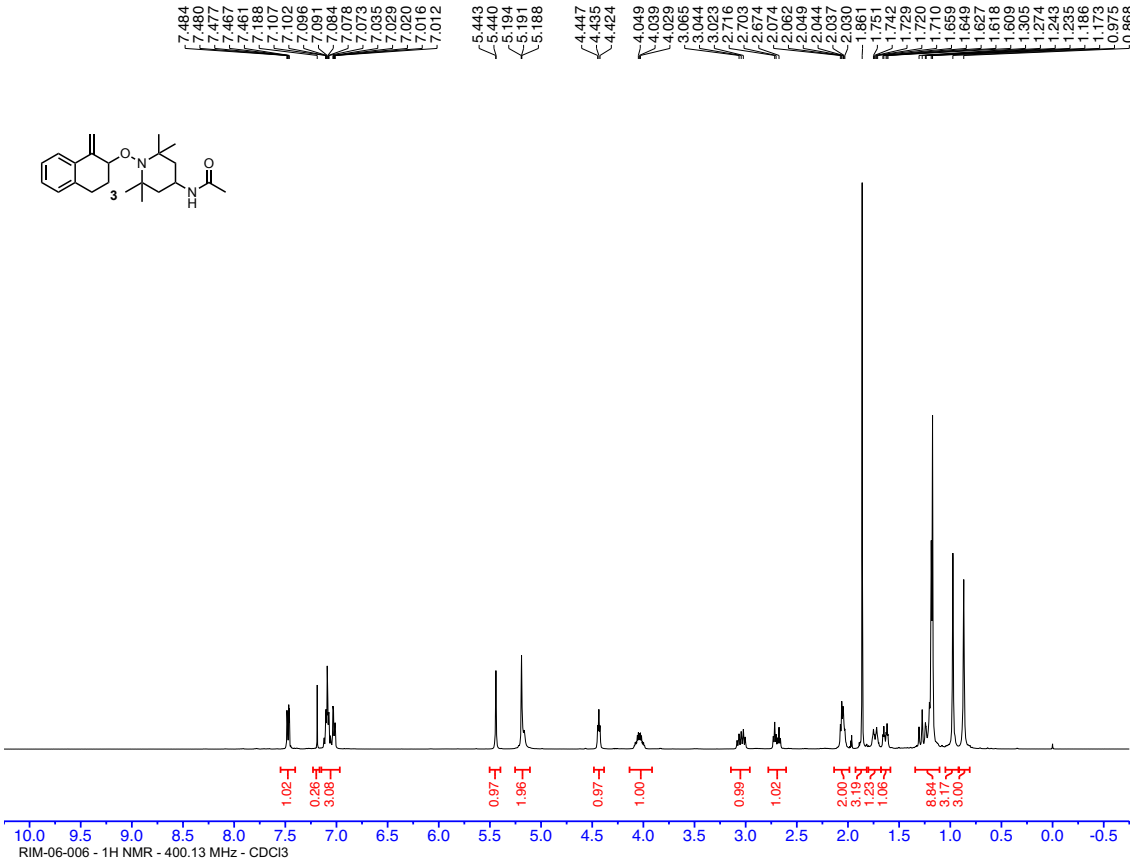
***N*-{1-[(7-Bromo-1-methylidene-3,4-dihydro-2H-naphthalen-2-yl)oxy]-2,2,6,6-tetramethylpiperidin-4-yl} acetamide (26).** Followed general procedure. Purified by silica chromatography using a solvent gradient of 50-70%. EA/hexanes to give 0.065 g product as a yellow solid. Yield: 45%. IR (film) 3270, 2928, 1640, 1555, 731 cm^{-1}; TLC R_f = 0.22 (70% EA/30% hexanes); mp = 140-144 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* =1.8 Hz, 1H), 7.27 (d, *J* = 10.0 Hz, 1H), 6.97 (d, *J* = 8.0 Hz, 1H), 5.50 (s, 1H), 5.28 (s, 1H), 5.17 (bs, 1H), 4.49 (bs,1H), 4.11 (bs, 1H), 3.09-3.01 (m, 1H), 2.72-2.68 (m, 1H), 2.15-2.13 (m, 1H), 2.07-2.03 (m, 1H). 1.93 (s, 3H), 1.81 (d, *J* = 11.6 Hz, 1H), 1.71 (d, *J* = 11.9 Hz, 1H), 1.25-1.23 (m, 8H), 1.03 (s, 3H), 0.91 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 169.4, 143.3, 136.3, 135.3, 130.4, 130.3, 127.8, 119.7, 112.2, 81.6, 60.5, 59.8, 46.2, 46.1, 41.1, 34.5, 34.1, 29.0, 25.6, 23.6, 21.2, 20.1; Anal calcd for $C_{22}H_{31}BrN_2O_2$: C, 60.69; H, 7.18; N, 6.43; Found: C, 60.53; H, 6.93; N, 6.14.



***N*-{1-[(7-Cyano-1-methylidene-3,4-dihydro-2H-naphthalen-2-yl)oxy]-2,2,6,6-tetramethylpiperidin-4-yl} acetamide (28).** Followed general procedure. Purified by silica chromatography using a solvent gradient of 50-70%. EA/hexanes to give 0.051 g product as a tan solid. Yield: 40%. IR (film) 3291, 2974, 2932, 1661, 1516, 902, 723 cm^{-1}; TLC R_f = 0.20 (70% EA/30% hexanes); mp = 153-157 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (s, 1H), 7.35 (d, *J* = 7.7 Hz, 1H), 7.13 (d, *J* = 7.7 Hz, 1H), 5.48 (s, 1H), 5.27 (s, 1H), 5.12 (d, *J* = 5.9 Hz, 1H), 4.45 (d, *J* = 3.0 Hz, 1H), 4.06-4.02 (m, 1H), 3.14-3.06 (m, 1H), 2.77-2.70 (m, 1H), 2.18-2.14 (m,1H), 1.97-1.95 (m, 1H), 1.87 (s, 3H), 1.76-1.61 (m, 3H), 1.31-1.23 (m, 1H), 1.19 (s, 3H), 1.16 (s, 3H), 0.91 (s, 3H), 0.79 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 169.3, 142.7, 141.9, 135.5, 130.3, 129.6, 129.2, 119.2, 113.4, 110.0, 81.4, 60.6, 59.7, 46.1, 46.0, 41.1, 34.6, 34.1, 28.6, 26.1, 23.6, 21.1, 20.1; Anal calcd for $C_{23}H_{31}N_3O_2$: C, 72.41; H, 8.19; N, 11.01; Found: C, 72.49; H, 8.16; N, 10.67.



1-Methylene-3,4-dihydro-2(2H)-naphthalene (29). In an oven dried round-bottom flask, 0.734 g (11.2 mmol, 10 equiv) of zinc powder was added and suspended in 18 mL ether (0.62 M). The zinc powder was activated with 0.099 g (0.896 mmol, 0.8 equiv) TMSCl. The reaction was refluxed for 30 minutes. After 30 minutes, the zinc suspension was cooled to room temperature and the ether was evaporated. A solution of 0.400 g (1.12 mmol) the ACT ether **3** was dissolved in 14.4 mL 1:1 AcOH:H_2O (0.08 M) and added drop wise to the activated zinc. The reaction was heated to 45 °C and left to stir for 2 h. After 2 h the reaction was cooled to room temperature, diluted with water and extracted with ether (3x). The organic layers were combined, dried over magnesium sulfate, and concentrated. The residue was purified by silica chromatography (30%. ether/pentanes) to give 0.088 g product as an oil. Yield: 49%. TLC R_f = 0.32 (30% ether/70% pentane); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58-7.55 (m, 1H), 7.18-7.04 (m, 3H), 5.54 (s, 1H), 5.23 (s, 1H), 4.45 (dd, *J* = 3.2, 7.6 Hz, 1H), 3.00 (dt, *J* = 6.4, 16.2 Hz, 1H), 2.80 (dt, *J* = 6.4, 16.9 Hz, 1H), 2.04-1.87 (m, 2H); $^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) δ 146.2, 136.2, 132.8, 128.9, 128.0, 126.2, 124.9, 108.5, 70.9, 31.4, 26.3. This compound has been previously reported.[3]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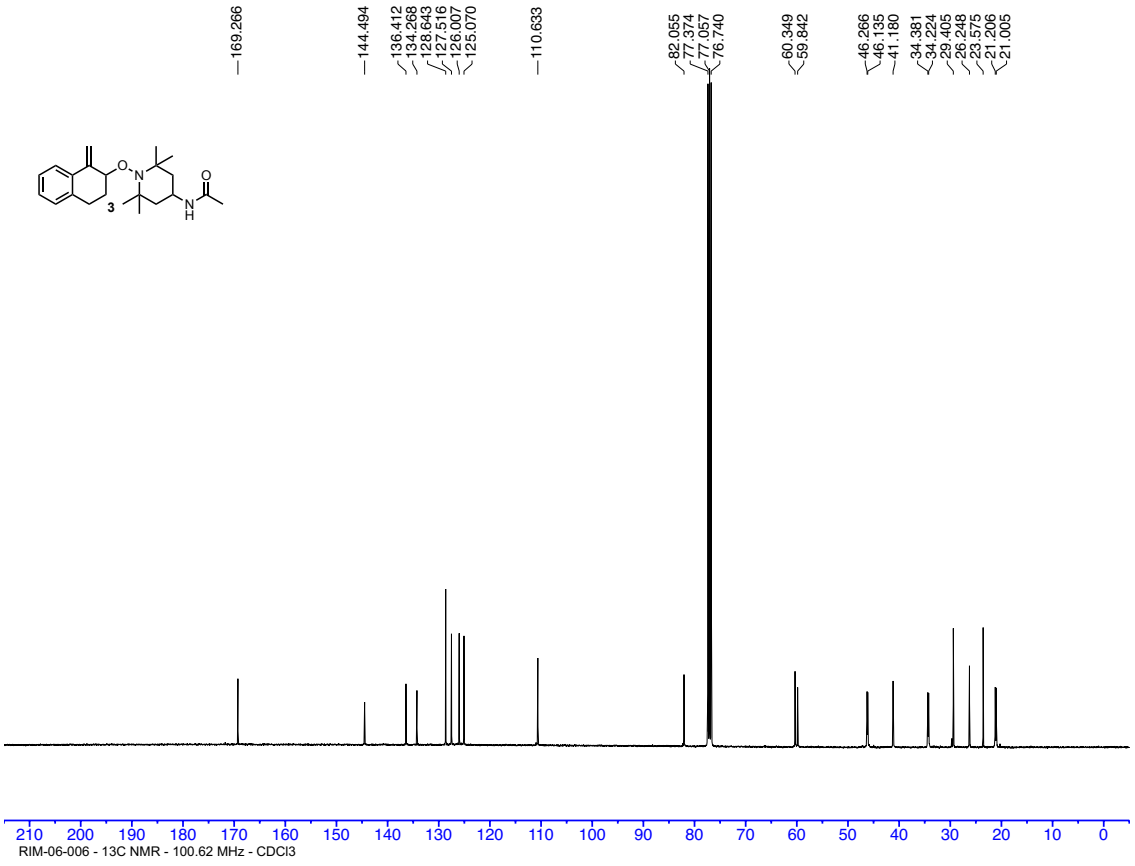
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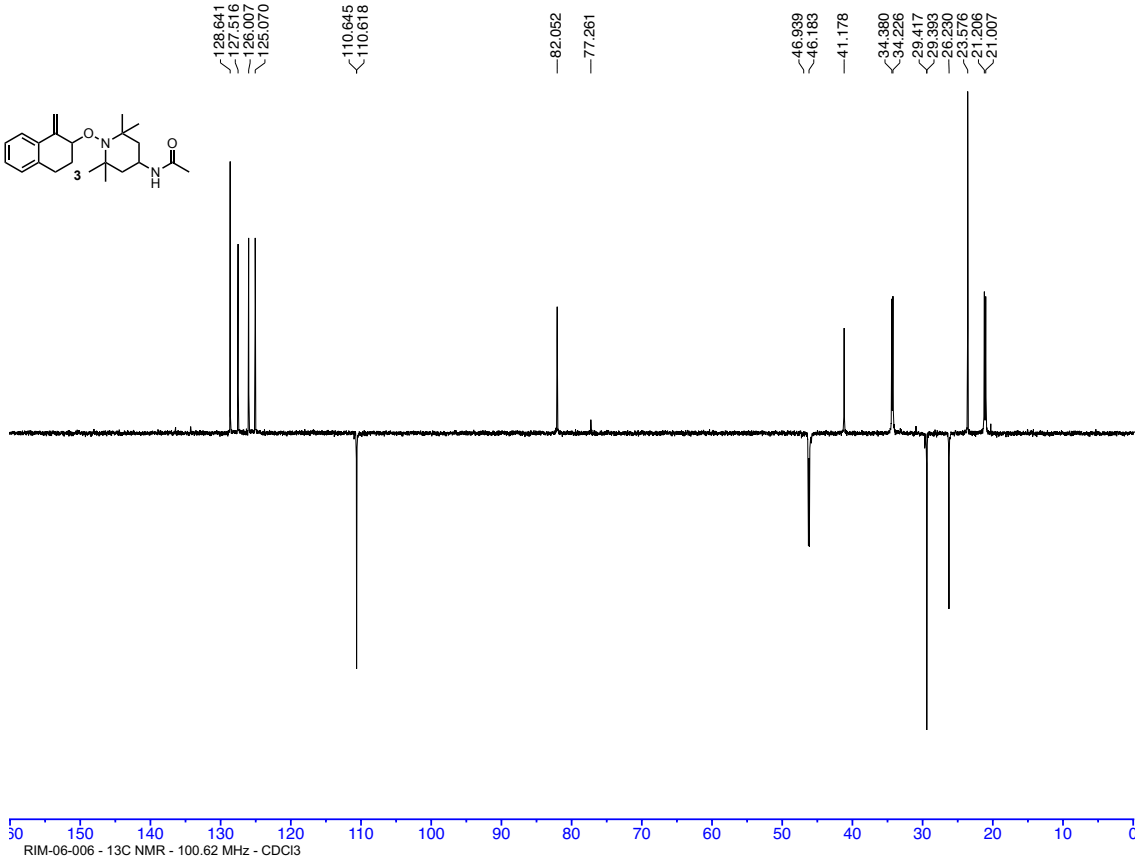
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100
90
80
70
60
50
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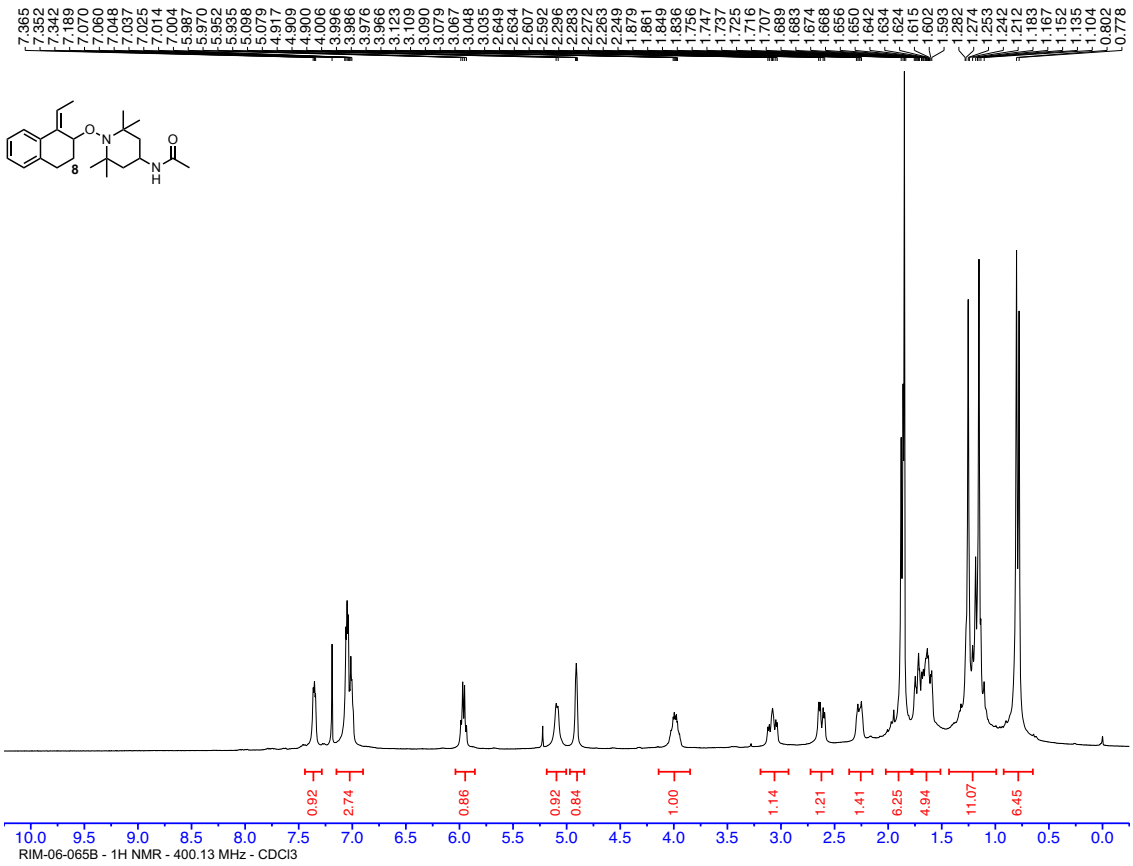
30
20
10
RIM-06-006 - 13C NMR - 100.62 MHz - CDCl3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7.037
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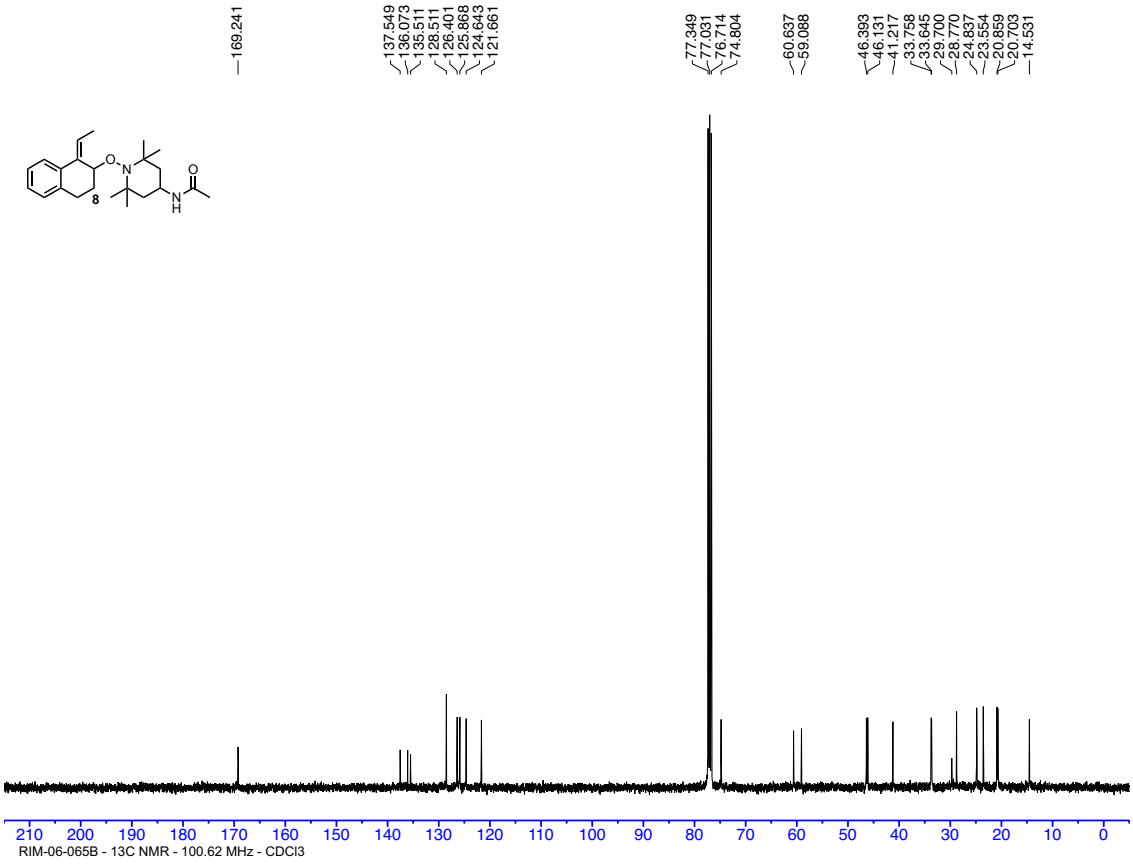
7.004
5.987
5.970
5.952
5.935
5.098
5.079
4.917
4.909
4.900
4.006
3.996
3.986
3.976
3.966
3.123
3.109
3.090
3.079
3.067
3.048
3.035
2.649
2.634
2.607
2.592
2.296
2.283
2.272
2.263
2.249
1.879
1.861
1.849
1.836
1.756
1.747
1.737
1.725
1.716
1.707
1.689
1.683
1.674
1.668
1.656
1.650
1.642
1.634
1.624
1.615
1.602
1.593
1.282
1.274
1.253
1.242
1.212
1.183
1.167
1.152
1.135
1.104
0.802
0.778
O
N
O
N
H
8
0.92
2.74
0.86
0.92
0.84
1.00
1.14
1.21
1.41
6.25
4.94
11.07
6.45
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
RIM-06-065B - 1H NMR - 400.13 MHz - CDCl3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135.511
128.511
126.401
125.868
124.643
121.661
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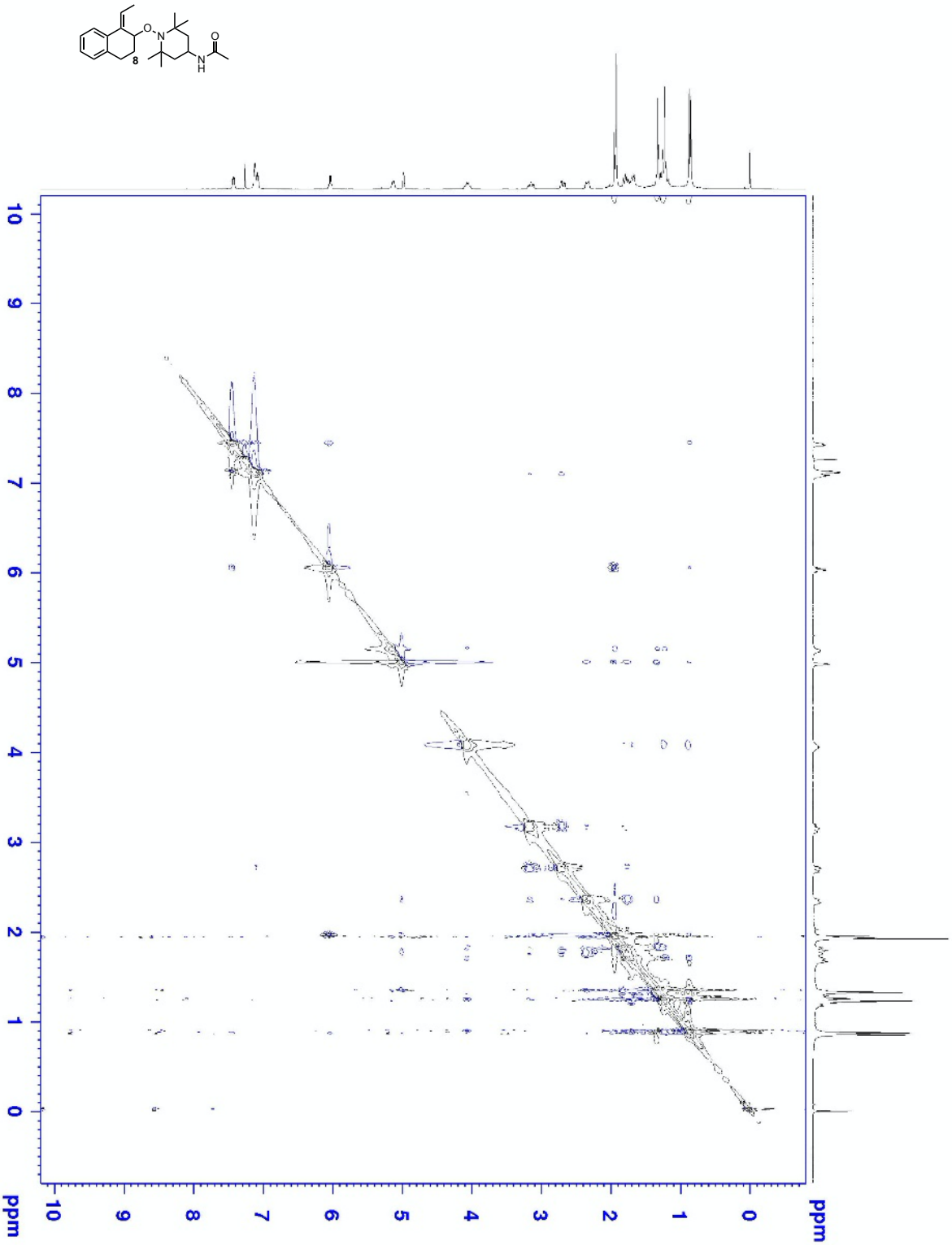
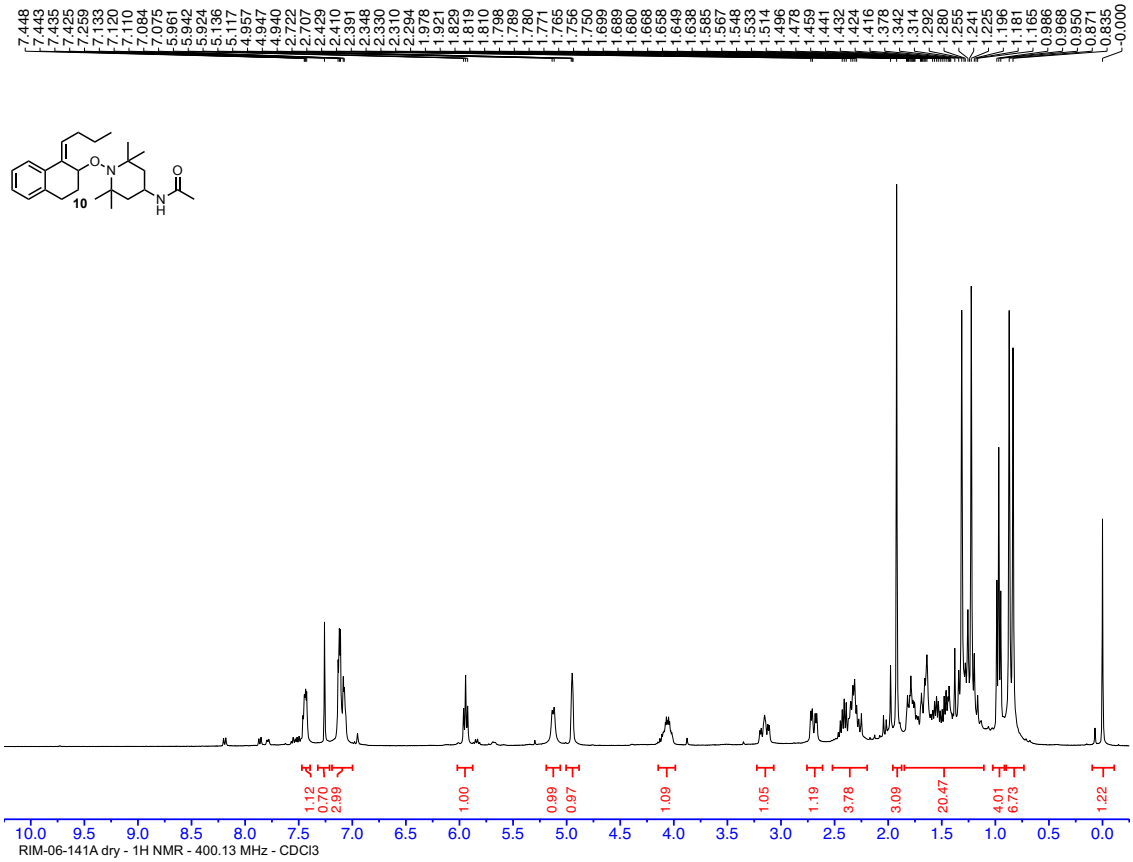
77.031
76.714
74.804
60.637
59.088
46.393
46.131
41.217
33.758
33.645
29.700
28.770
24.837
23.554
20.859
20.703
14.531
O
N
O
N
H
8
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
RIM-06-065B - 13C NMR - 100.62 MHz - CDCl3

NOESY spectra of Allylic Ether **8**

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7.435
7.425
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7.133
7.120
7.110
7.084
7.075
5.961
5.942
5.924
5.136
5.117
4.957
4.947
4.940
2.722
2.707
2.429
2.410
2.391
2.348
2.330
2.310
2.294
1.978
1.921
1.829
1.819
1.810
1.798
1.789
1.780
1.771
1.765
1.756
1.750
1.699
1.689
1.680
1.668
1.658
1.649
1.638
1.585
1.567
1.548
1.533
1.514
1.496
1.478
1.459
1.441
1.432
1.424
1.416
1.378
1.342
1.314
1.292
1.280
1.255
1.241
1.225
1.196
1.181
1.165
0.986
0.968
0.950
0.871
0.835
0.000
10
1.12
0.70
2.99
1.00
0.99
0.97
1.09
1.05
1.19
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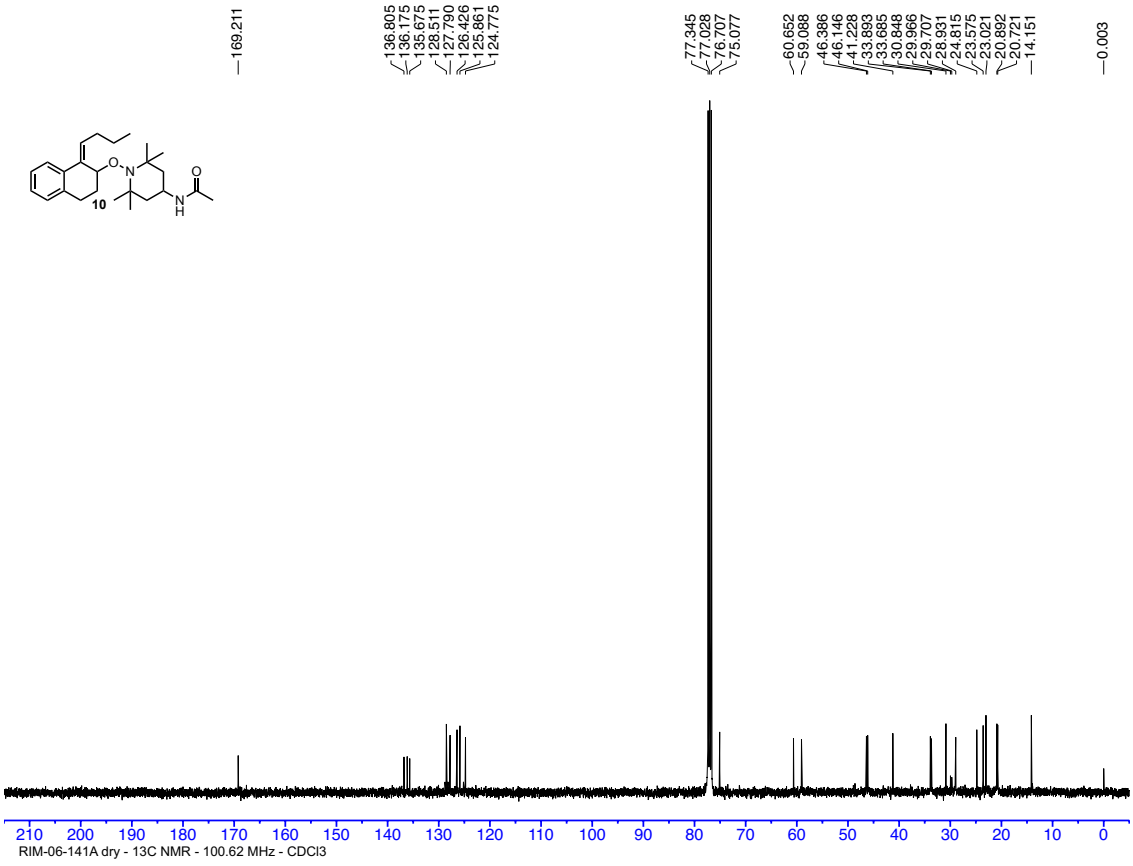
3.09
20.47
4.01
6.73
1.22
RIM-06-141A dry - 1H NMR - 400.13 MHz - CDCl3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127.790
126.426
125.861
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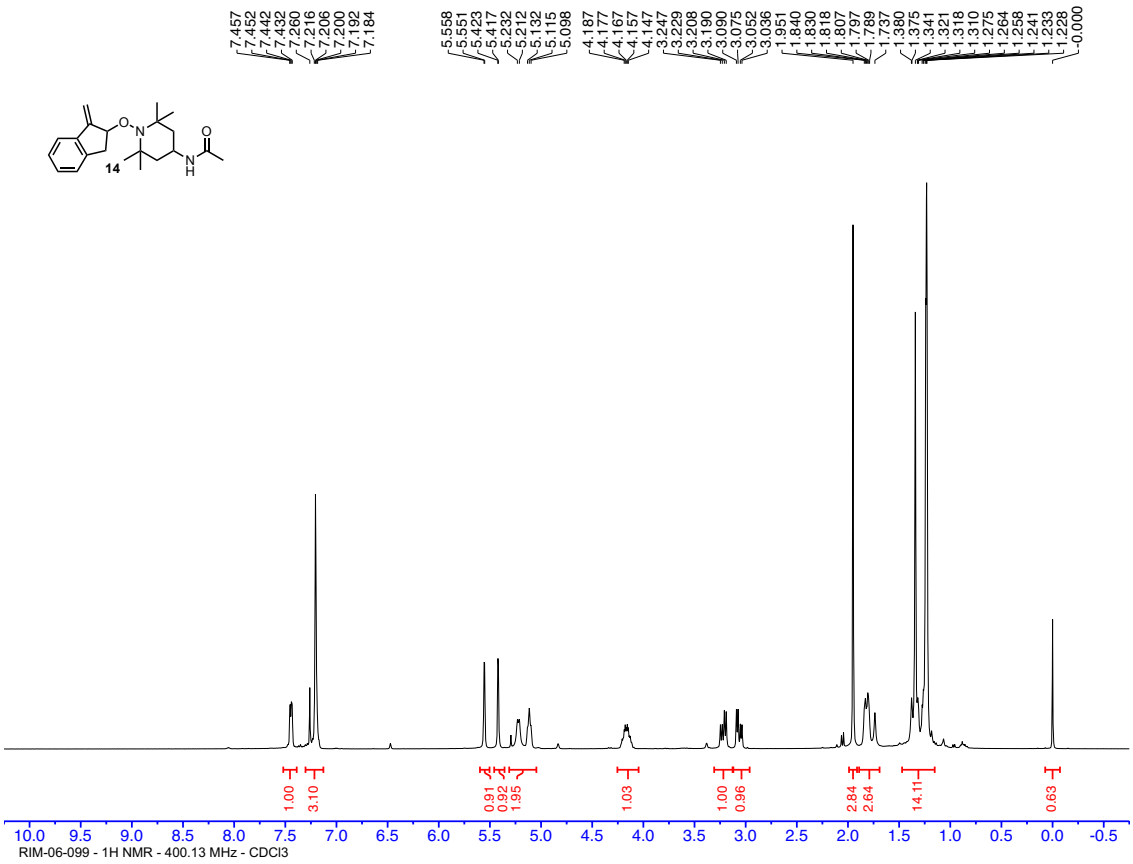
77.028
76.707
75.077
60.652
59.088
46.386
46.146
41.228
33.893
33.685
30.848
29.966
29.707
28.931
24.815
23.575
23.021
20.892
20.721
14.151
0.003
10
RIM-06-141A dry - 13C NMR - 100.62 MHz - CDCl3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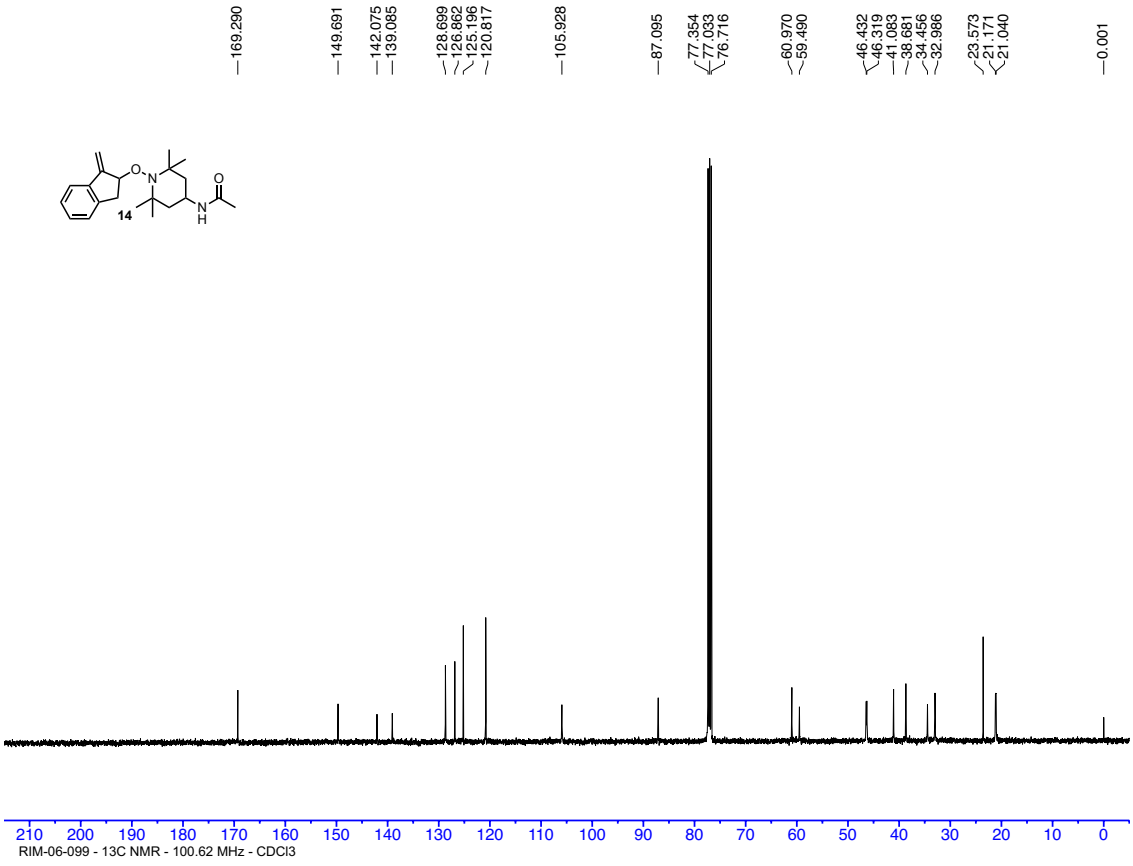
7.432
7.260
7.216
7.206
7.200
7.192
7.184
5.558
5.551
5.423
5.417
5.232
5.212
5.132
5.115
5.098
4.187
4.177
4.167
4.157
4.147
3.247
3.229
3.208
3.190
3.090
3.075
3.052
3.036
1.951
1.840
1.830
1.818
1.807
1.797
1.789
1.737
1.380
1.375
1.341
1.321
1.318
1.310
1.275
1.264
1.258
1.241
1.233
1.228
-0.000
14
1.00
3.10
0.91
0.92
1.95
1.03
1.00
0.96
2.84
2.64
14.11
0.63
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
RIM-06-099 - 1H NMR - 400.13 MHz - CDCl3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149.691
142.075
139.085
128.699
126.862
125.196
120.817
105.928
87.095
77.354
77.033
76.716
60.970
59.490
46.432
46.319
41.083
38.681
34.456
32.986
23.573
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21.040
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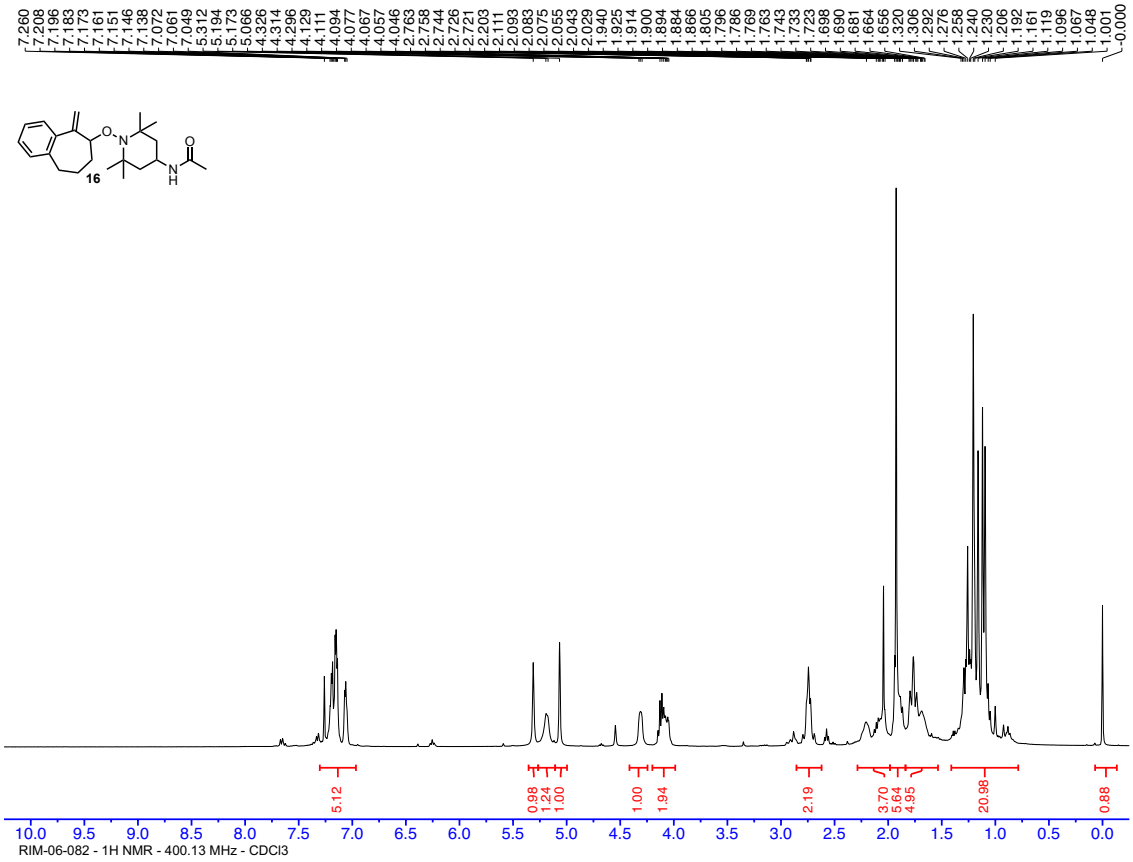
14
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
RIM-06-099 - 13C NMR - 100.62 MHz - CDCl3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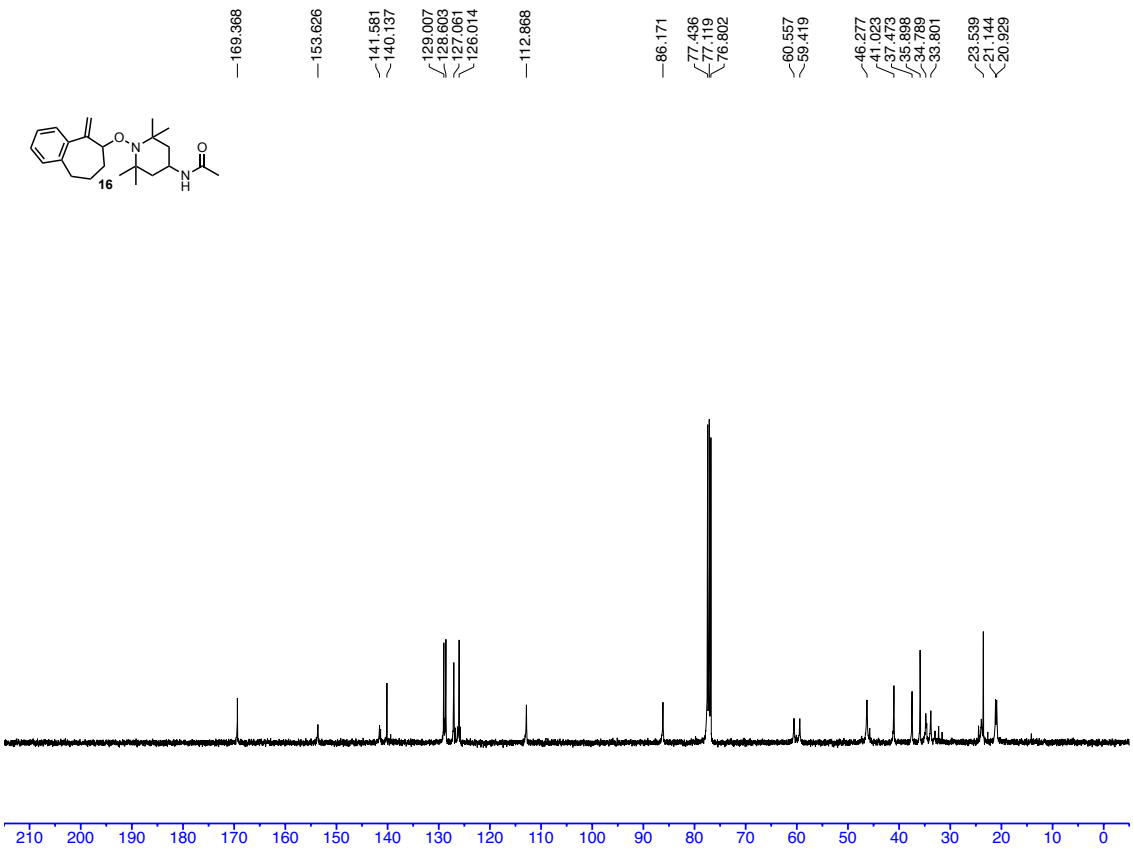
7.208
7.196
7.183
7.173
7.161
7.151
7.146
7.138
7.072
7.061
7.049
5.312
5.194
5.173
5.066
4.326
4.314
4.296
4.129
4.111
4.094
4.077
4.067
4.057
4.046
2.763
2.758
2.744
2.726
2.721
2.203
2.111
2.093
2.083
2.075
2.055
2.043
2.029
1.940
1.925
1.914
1.900
1.894
1.884
1.866
1.805
1.796
1.786
1.769
1.763
1.743
1.733
1.723
1.698
1.690
1.681
1.664
1.656
1.320
1.306
1.292
1.276
1.258
1.240
1.230
1.206
1.192
1.161
1.119
1.096
1.067
1.048
1.001
-0.000
16
5.12
0.98
1.24
1.00
1.00
1.94
2.19
3.70
5.64
4.95
20.98
0.88
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
RIM-06-082 - 1H NMR - 400.13 MHz - CDCl3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140.137
129.007
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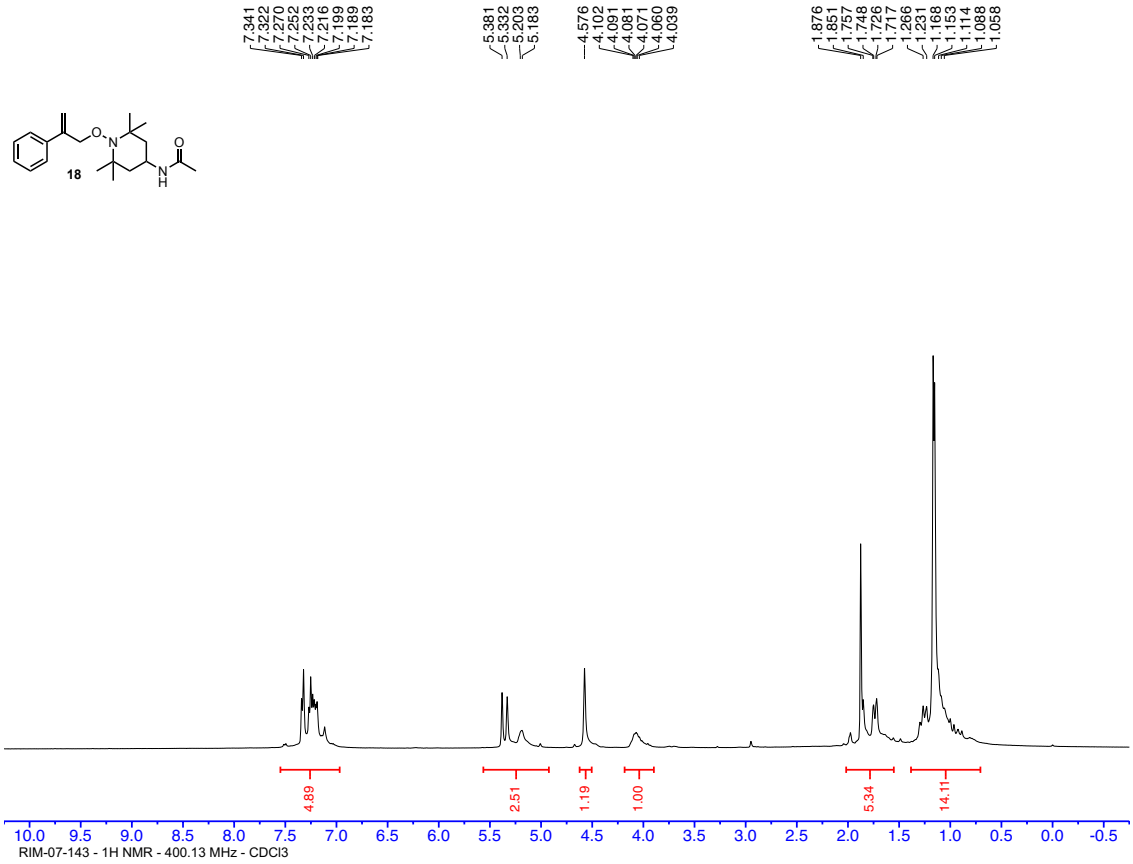
127.061
126.014
112.868
86.171
77.436
77.119
76.802
60.557
59.419
46.277
41.023
37.473
35.898
34.789
33.801
23.539
21.144
20.929
16
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
RA-2-115 recrystallized trial 2 - 13C NMR - 100.62 MHz - CDCl3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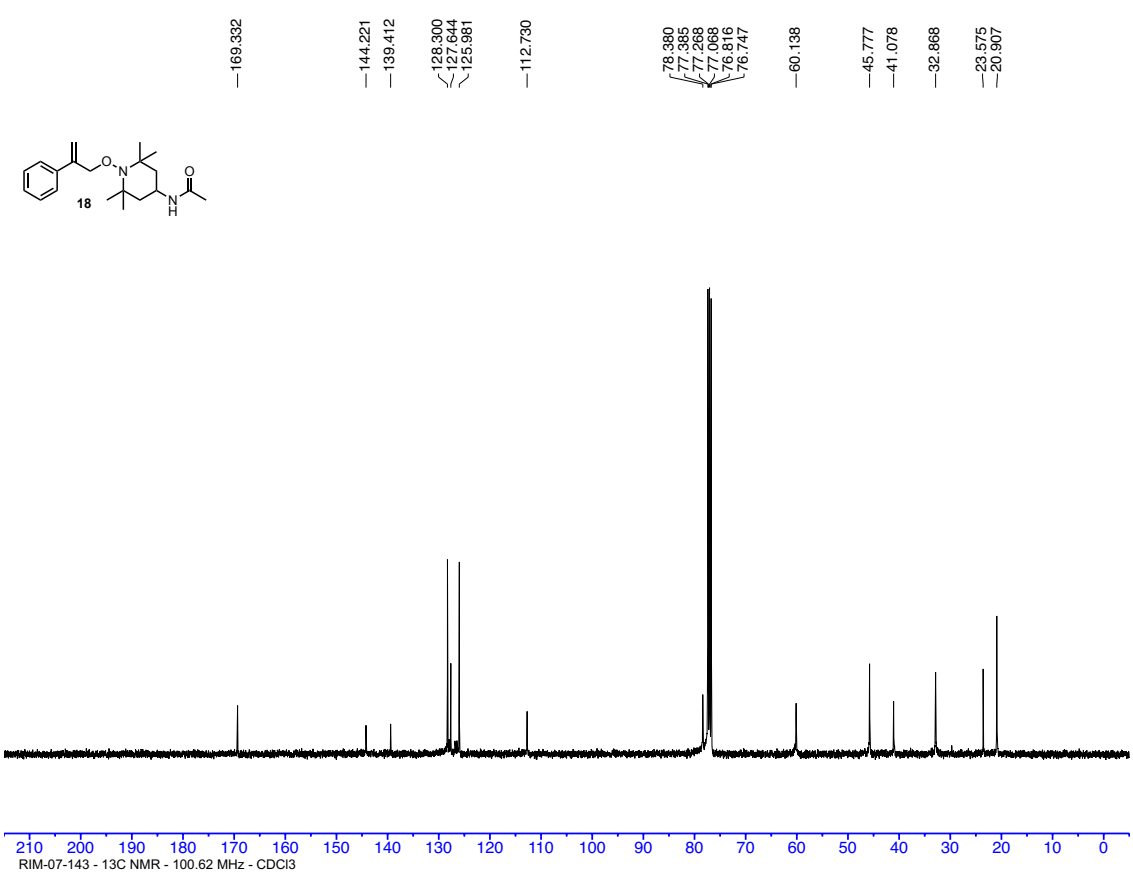
7.252
7.233
7.216
7.199
7.189
7.183
5.381
5.332
5.203
5.183
4.576
4.102
4.091
4.081
4.071
4.060
4.039
1.876
1.851
1.757
1.748
1.726
1.717
1.266
1.231
1.168
1.153
1.114
1.088
1.058
18
4.89
2.51
1.19
1.00
5.34
14.11
RIM-07-143 - 1H NMR - 400.13 MHz - CDCl3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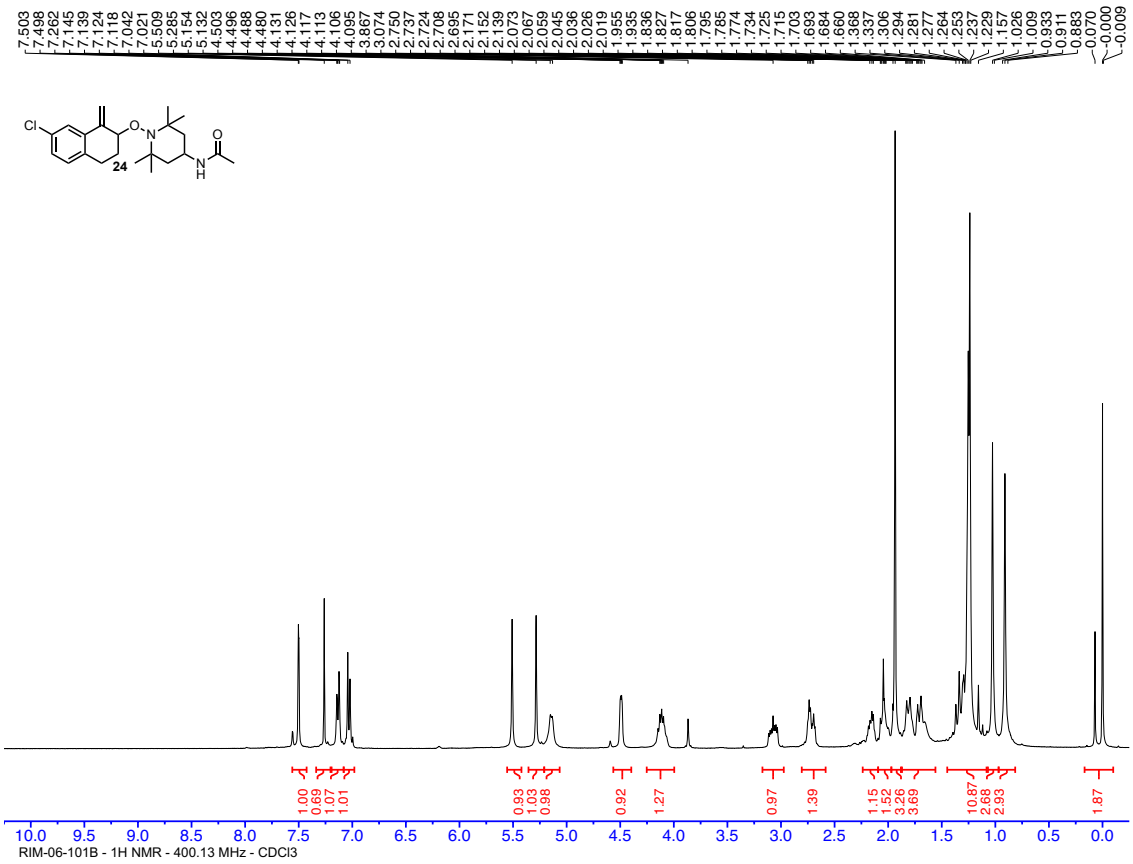
144.221
139.412
128.300
127.644
125.981
112.730
78.380
77.385
77.268
77.068
76.816
76.747
60.138
45.777
41.078
32.868
23.575
20.907
18
RIM-07-143 - 13C NMR - 100.62 MHz - CDCl3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7.021
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5.285
5.154
5.132
4.503
4.496
4.488
4.480
4.131
4.126
4.117
4.113
4.106
4.095
3.867
3.074
2.750
2.737
2.724
2.708
2.695
2.171
2.152
2.139
2.073
2.067
2.059
2.045
2.036
2.026
2.019
1.955
1.935
1.836
1.827
1.817
1.806
1.795
1.785
1.774
1.734
1.725
1.715
1.703
1.693
1.684
1.660
1.368
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1.281
1.277
1.264
1.253
1.237
1.229
1.157
1.026
1.009
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0.911
0.883
0.070
-0.000
-0.009
Cl
O
N
O
N
H
24
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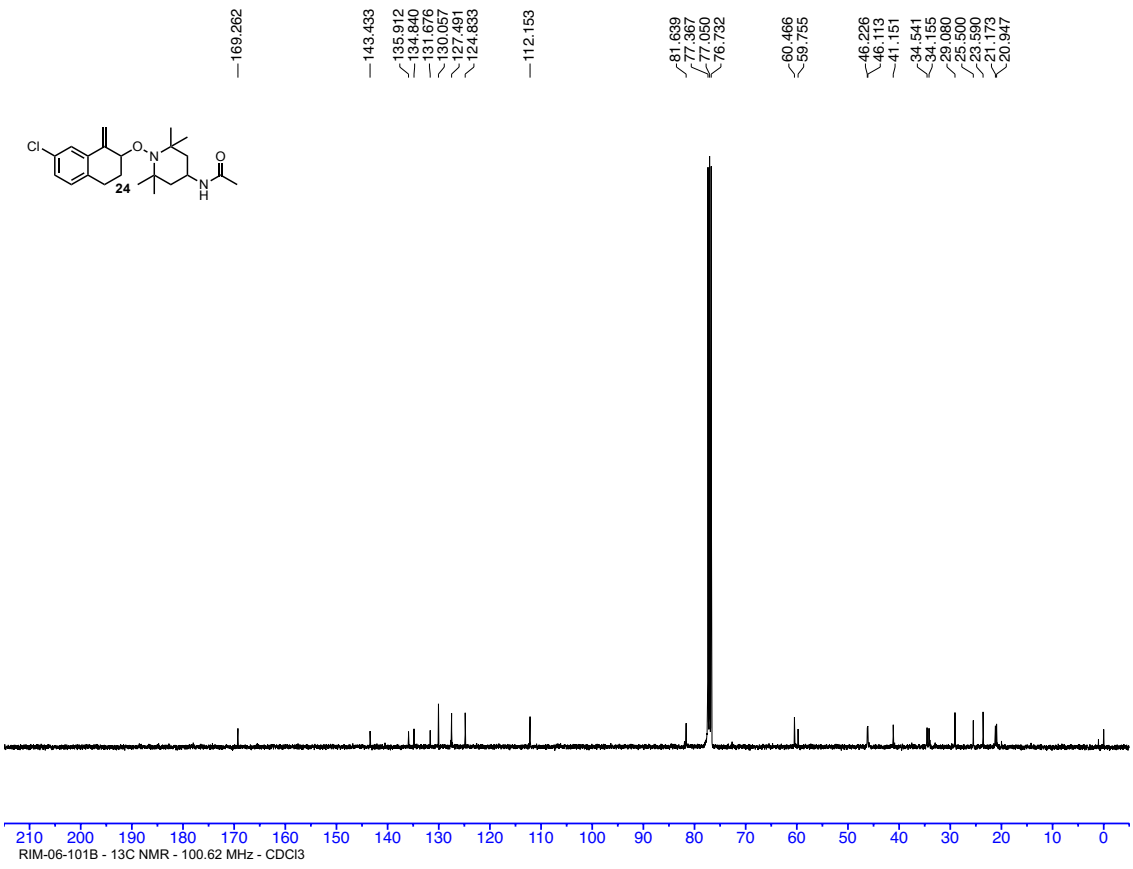
0.69
1.07
1.01
0.93
1.03
0.98
0.92
1.27
0.97
1.39
1.15
1.52
3.26
3.69
10.87
2.68
2.93
1.87
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
RIM-06-101B - 1H NMR - 400.13 MHz - CDCl3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135.912
134.840
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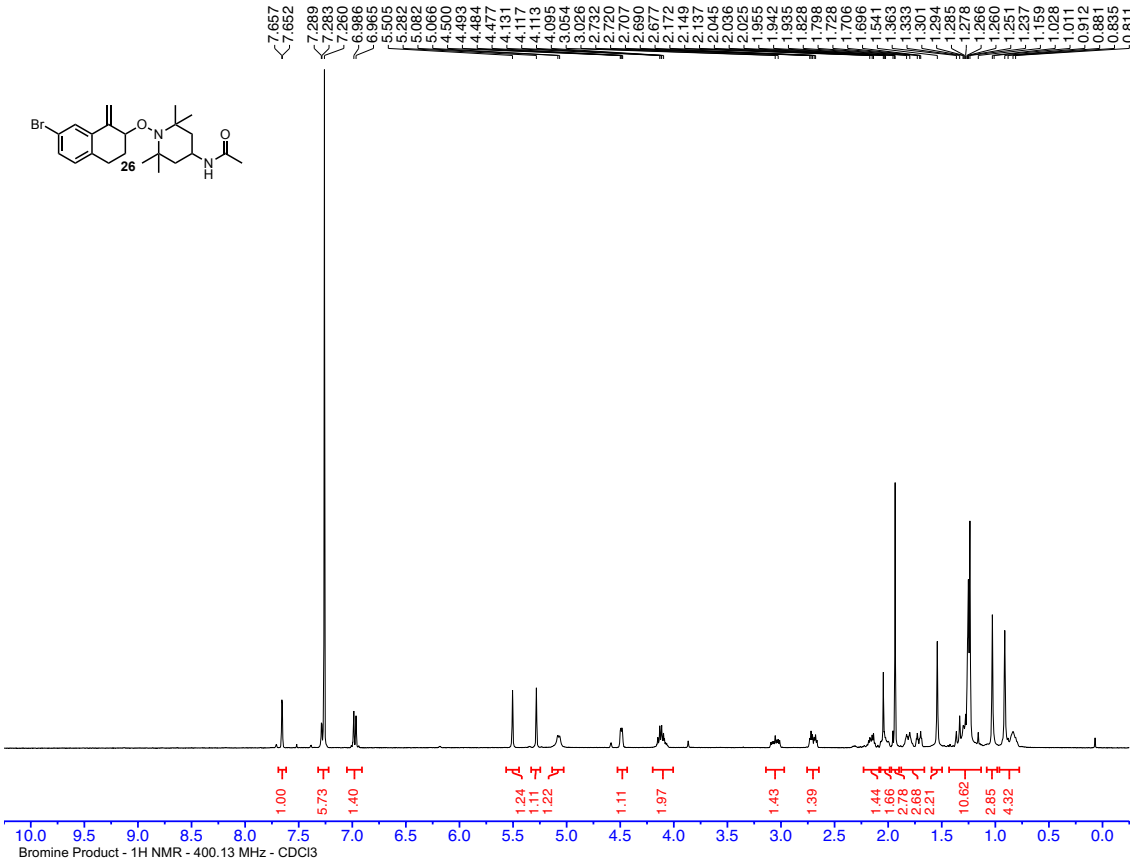
127.491
124.833
112.153
81.639
77.367
77.050
76.732
60.466
59.755
46.226
46.113
41.151
34.541
34.155
29.080
25.500
23.590
21.173
20.947
Cl
O
N
O
N
H
24
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
RIM-06-101B - 13C NMR - 100.62 MHz - CDCl3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6.986
6.965
5.505
5.282
5.082
5.066
4.500
4.493
4.484
4.477
4.131
4.117
4.113
4.095
3.054
3.026
2.732
2.720
2.707
2.690
2.677
2.172
2.149
2.137
2.045
2.036
2.025
1.955
1.942
1.935
1.828
1.798
1.728
1.706
1.696
1.541
1.363
1.333
1.301
1.294
1.285
1.278
1.266
1.260
1.251
1.237
1.159
1.028
1.011
0.912
0.881
0.835
Br
O
N
O
N
H
26
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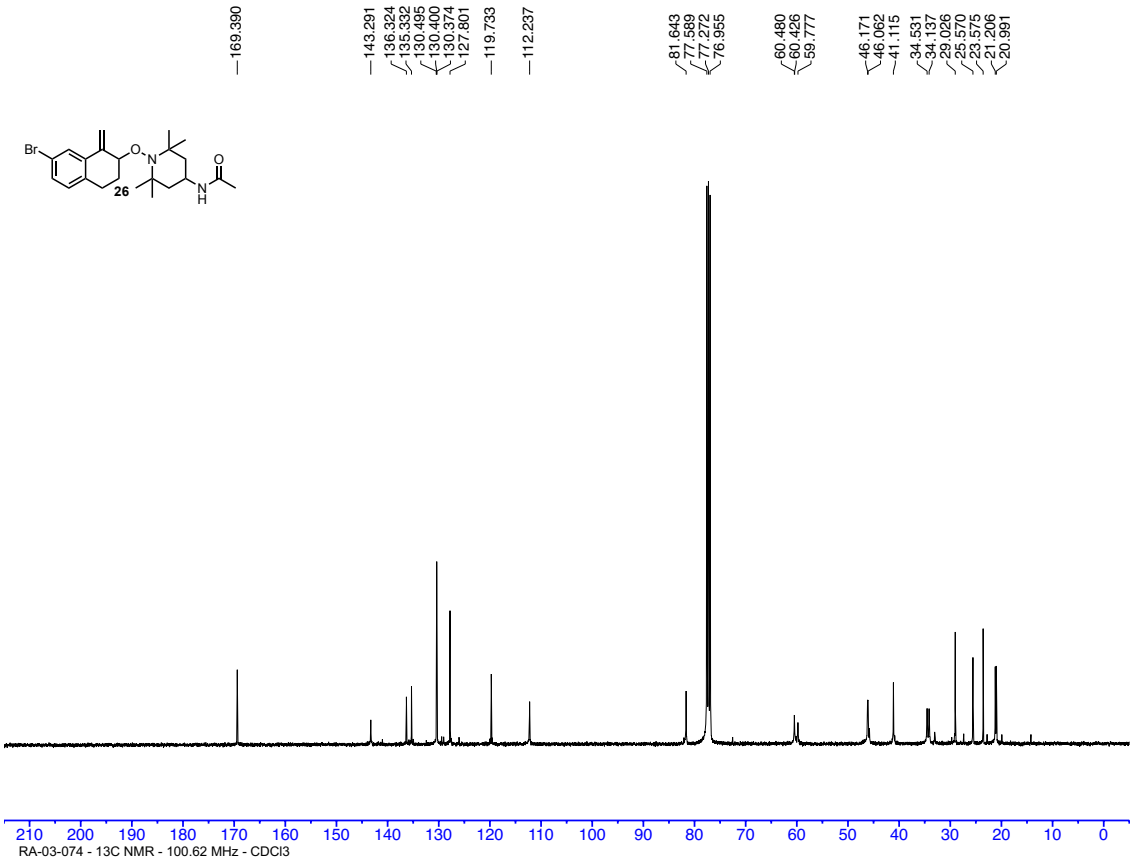
1.40
1.24
1.11
1.22
1.11
1.97
1.43
1.39
1.44
1.66
2.78
2.68
2.21
10.62
2.85
4.32
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
Bromine Product - 1H NMR - 400.13 MHz - CDCl3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130.400
130.374
127.801
119.733
112.237
81.643
77.589
77.272
76.955
60.480
60.426
59.777
46.171
46.062
41.115
34.531
34.137
29.026
25.570
23.575
21.206
20.991
Br
O
N
O
N
H
26
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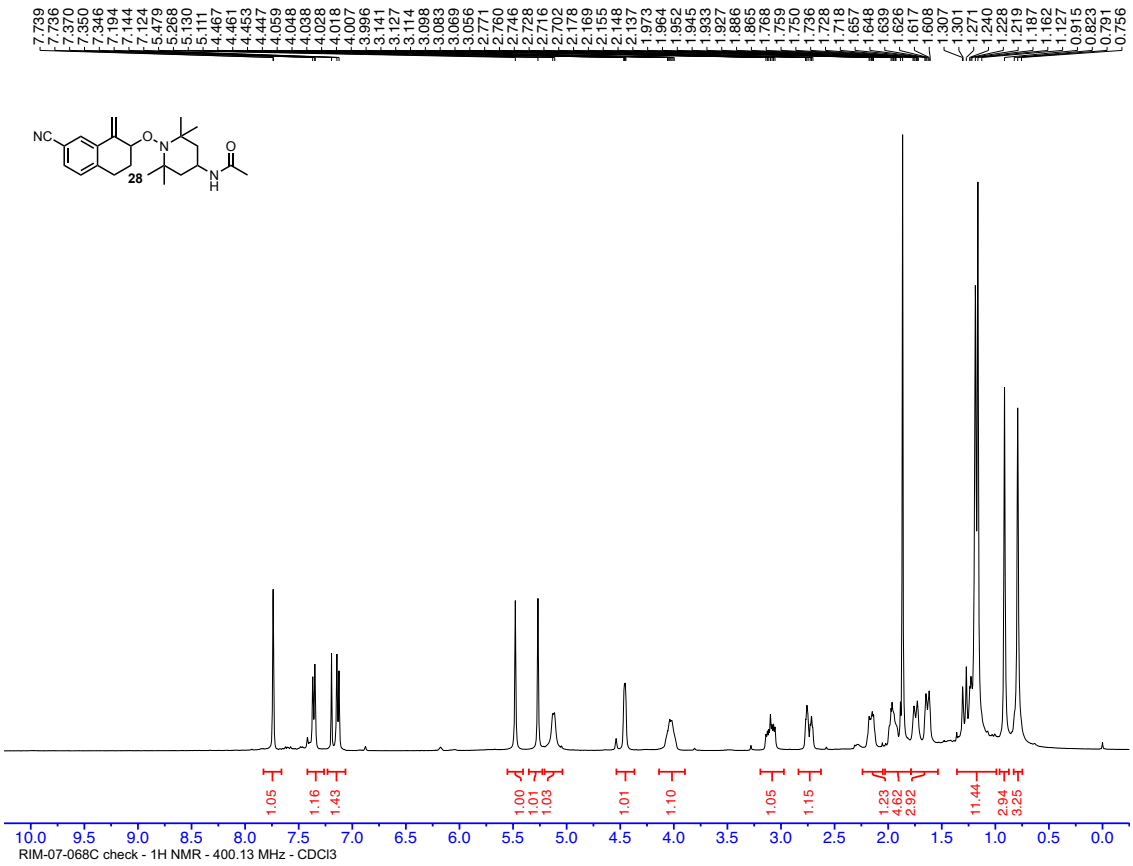
RA-03-074 - 13C NMR - 100.62 MHz - CDCl3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5.111
4.467
4.461
4.453
4.447
4.059
4.048
4.038
4.028
4.018
4.007
3.996
3.141
3.127
3.114
3.098
3.083
3.069
3.056
2.771
2.760
2.746
2.728
2.716
2.702
2.178
2.169
2.155
2.148
2.137
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1.964
1.952
1.945
1.933
1.927
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1.759
1.750
1.736
1.728
1.718
1.657
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1.626
1.617
1.608
1.307
1.301
1.271
1.240
1.228
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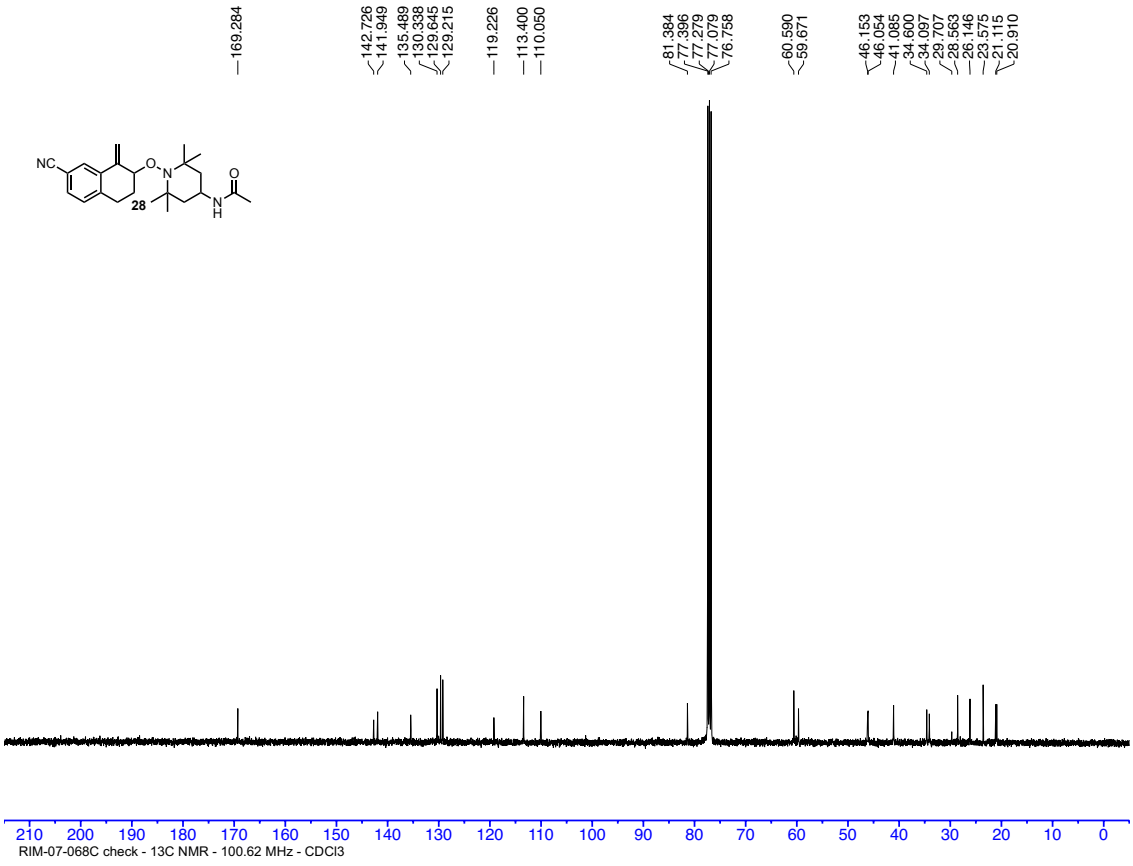
0.756
NC
O
N
O
N
H
28
1.05
1.16
1.43
1.00
1.01
1.03
1.01
1.10
1.05
1.15
1.23
4.62
2.92
11.44
2.94
3.25
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
RIM-07-068C check - 1H NMR - 400.13 MHz - CDCl3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129.215
119.226
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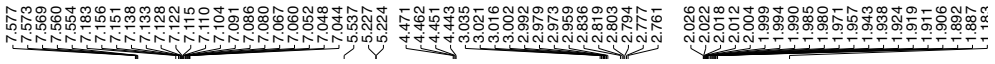
110.050
81.384
77.396
77.279
77.079
76.758
60.590
59.671
46.153
46.054
41.085
34.600
34.097
29.707
28.563
26.146
23.575
21.115
20.910
NC
O
N
O
N
H
28
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
RIM-07-068C check - 13C NMR - 100.62 MHz - CDCl3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7.560
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7.110
7.104
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7.052
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5.537
5.227
5.224
4.471
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4.443
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3.021
3.016
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1.924
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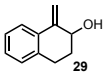
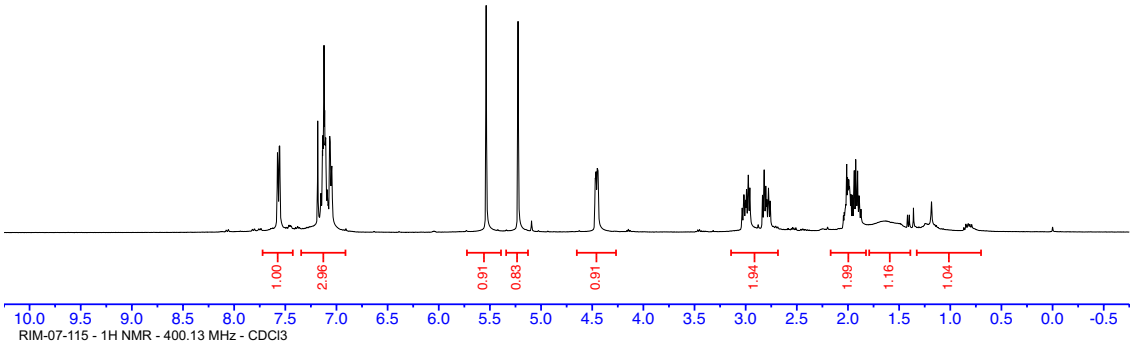
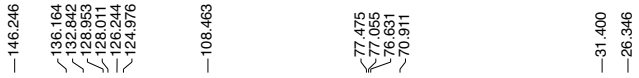
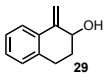
1.892
1.887


OH
29


1.00
2.96
0.91
0.83
0.91
1.94
1.99
1.16
1.04
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
RIM-07-115 - 1H NMR - 400.13 MHz - CDCl3


146.246
136.164
132.842
128.953
128.011
126.244
124.976
108.463
77.475
77.055
76.631
70.911
31.400
26.346


OH
29

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
RIM-07-115 - 13C NMR - 75.49 MHz - CDCl3

References

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