Electronic Supplementary Material (ESI) for RSC Advances.

Triphenylbismuth carbonate-mediated oxidation of hydroxylamines to nitrones and *in situ* 1,3-dipolar cycloaddition

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1. General

Reactions were carried out using dry solvents. TLC was performed on silica gel plates visualized either with a UV lamp (254 nm/365 nm), or using solutions of phosphomolybdic acid in EtOH or $KMnO_4$–K_2CO_3 in water followed by heating. Flash chromatography was performed on Kieselgel 60 (230–240 mesh, Merck). NMR spectra were recorded on a Bruker AVANCE DPX 400 spectrometer. ^1H NMR spectra were recorded at 400 MHz and data are reported as follows: chemical shift in ppm from tetramethylsilane as internal standard, multiplicity: s = singlet, d = doublet, t = triplet, q = quartet, quint = quintuplet, m = multiplet), coupling constant (*J*) in Hz, integration. ^{13}C NMR spectra were recorded at 100 MHz and data are reported as follows: chemical shift in ppm from tetramethylsilane with the deuterated solvent signal(s) used for calibration, multiplicity with respect to proton (deduced from DEPT experiments, s = quaternary carbon (C), d = CH, t = CH_2, q = CH_3). Mass spectra were recorded using a Waters Micromass ZQ 2000 ESI spectrometer. IR-spectra were recorded using a Perkin-Elmer 2000 FT-IR. Wavenumbers are given in cm^{-1} at their maximum intensity.

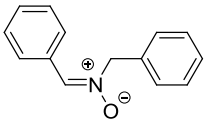
2. Gram-scale synthesis of compound 2a.

At room temperature, to a solution of *N,N*-dibenzyl hydroxylamine **1a** (1.02 g, 4.78 mmol, 1 equiv.) in CH_2Cl_2 (50 mL) was added portionwise Ph_3BiCO_3 (2.63 g, 1.1 equiv.). The white slurry was stirred at room temperature for 2 h. The mixture was then filtered over a pad of celite, rinsed with CH_2Cl_2, and the solvent was evaporated under vacuum. The crude product was purified by flash chromatography over silica (Pentane/EtOAc, 1:0 to 1:1) to afford 0.98 g of compound **2a** (97%, white solid).

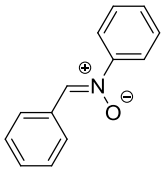
3. Gram-scale synthesis of compound 3a.

At room temperature, to a solution of *N,N*-dibenzyl hydroxylamine **1a** (1 g, 4.68 mmol, 1 equiv.) and benzannulated cyclooctyne **4** (0.96 g, 1 equiv.) in CH_2Cl_2 (70 mL) was added portionwise Ph_3BiCO_3 (2.57 g, 1.1 equiv.). The white slurry was stirred at room temperature for 2 h. The mixture was then filtered over a pad of celite, rinsed with CH_2Cl_2, and the solvent was evaporated under vacuum. The crude product was purified by flash chromatography over silica (Pentane/EtOAc, 1:0 to 9:1) to afford 1.47 g of compound **3a** (76%, white solid).

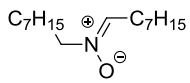
4. Analytical data



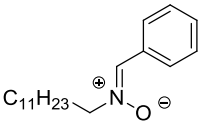
Compound 2a.[1] Flash chromatography (Pentane/EtOAc, from 1:0 to 1:1). ^{1}H NMR ($CDCl_3$) δ 8.23-8.18 (m, 2H), 7.52-7.26 (m, 9H), 5.06 (s, 2H) ppm. ^{13}C NMR ($CDCl_3$) δ 134.2 (d), 133.1 (s), 130.4 (d), 130.3 (d), 129.2 (d), 128.9 (d), 128.5 (d), 128.4 (d), 71.1 (t) ppm. IR (neat) 1580, 1457, 1147, 903, 724 cm^{-1}. MS (ES$^+$): 212 $[M+H]^+$.



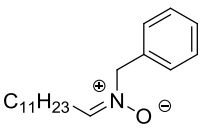
Compound 2b.[2] Flash chromatography (Pentane/EtOAc, from 1:0 to 8:2). ^{1}H NMR ($CDCl_3$) δ 8.41-8.39 (m, 2H), 7.92 (s, 1H), 7.79-7.77 (m, 2H), 7.51-7.46 (m, 6H) ppm. ^{13}C NMR ($CDCl_3$) δ 149.0 (s), 134.5 (d), 130.9 (s), 129.9 (d), 129.1 (d), 128.9 (d), 128.6 (d), 121.7 (d) ppm. IR (neat) 1549, 1484, 1444, 1065, 919, 764 cm^{-1}. MS (ES$^+$): 198 $[M+H]^+$.



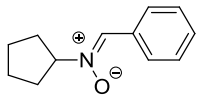
Compound 2c.[3] Flash chromatography (CH_2Cl_2/MeOH from 1:0 to 98:2). ^{1}H NMR ($CDCl_3$) δ 6.64 (t, *J* = 5.8 Hz, 1H), 3.73 (t, *J* = 7.0 Hz, 2H), 2.48 (q, *J* = 6.8 Hz, 2H), 1.89 (qt, *J* = 7.2 Hz, 2H), 1.51 (qt, *J* = 7.2 Hz, 2H), 1.35-1.24 (m, 18H), 0.87 (t, *J* = 6.7 Hz, 1H) ppm. ^{13}C NMR ($CDCl_3$) δ 139.2 (d), 65.3 (t), 31.6 (t, x2), 29.3 (t), 29.0 (t, x2), 28.9 (t), 27.3 (t), 26.5 (t), 26.4 (t), 25.5 (t), 22.5 (t), 14.0 (q) ppm. IR (neat) 1600, 1463, 904, 724 cm^{-1}. MS (ES$^+$): 256 $[M+H]^+$.



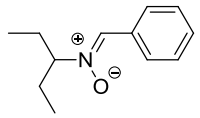
Compound 2d.[1] Compound **2d** was isolated from compound **2d'** by flash chromatography (Pentane/EtOAc, from 1:0 to 7:3 to 0:1, then CH_2Cl_2/MeOH 95:5). ^{1}H NMR ($CDCl_3$) δ 8.25-8.23 (m, 2H), 7.45-7.41 (m, 3H), 7.37 (s, 1H), 3.92 (t, *J* = 7.14 Hz, 2H), 2.00 (quint, *J* = 7.5 Hz, 2H), 1.40-1.24 (m, 18H), 0.87 (t, *J* = 6.39 Hz, 3H) ppm. ^{13}C NMR ($CDCl_3$) δ 134.1 (s), 130.4 (s), 130.2 (d), 128.4 (d), 121.7 (d), 67.3 (t), 31.8 (t), 29.5 (t), 29.4 (t), 29.3 (t), 29.2 (t), 29.1 (t), 27.7 (t), 26.4 (t), 22.6 (t), 14.0 (q) ppm. IR (neat) 1457, 1153, 903, 724 cm^{-1}. MS (ES$^+$): 290 $[M+H]^+$.



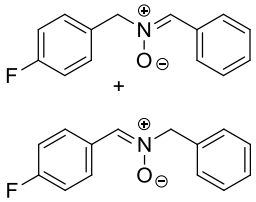
Compound 2d'.[1] ^{1}H NMR ($CDCl_3$) δ 7.40-7.33 (m, 5H), 6.62 (t, *J* = 5.6 Hz, 1H), 4.84 (s, 2H), 2.45 (dd, *J* = 13.8, 6.94 Hz, 2H), 1.48-1.40 (m, 2H), 1.31-1.19 (m, 16H), 0.87 (t, *J* = 6.44 Hz, 3H). ^{13}C NMR ($CDCl_3$) δ 137.5 (s), 130.4 (d), 129.3 (d), 128.9 (d), 128.2 (d), 85.8 (t), 31.8 (t), 29.6 (t), 29.5 (t), 29.4 (t), 29.2 (t), 22.6 (t), 14.0 (q) ppm. IR (neat) 1777, 1456, 1214, 908, 750 cm^{-1}. MS (ES$^+$): 290 $[M+H]^+$.



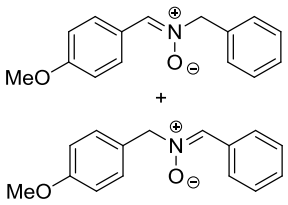
Compound 2e. Flash chromatography (Pentane/EtOAc, from 1:0 to 8:2). ^{1}H NMR ($CDCl_3$) δ 8.24-8.22 (m, 2H), 7.49 (s, 1H), 7.43-7.37 (m, 3H), 4.48-4.41 (m, 1H), 2.33-2.24 (m, 2H), 2.05-1.90 (m, 4H), 1.70-1.60 (m, 2H) ppm. ^{13}C NMR ($CDCl_3$) δ 132.8 (d), 130.6 (s), 130.0 (d), 128.4 (d, x2), 76.3 (d), 31.4 (t), 25.5 (t) ppm. IR (neat) 1572, 1451, 1328, 140, 903, 726, 695 cm^{-1}. MS (ES^+): 190 $[M+H]^+$. HRMS calcd for $C_{12}H_{16}NO$ $[M+H]^+$ 190.1232, found 190.1244.



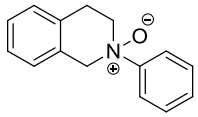
Compound 2f. Flash chromatography (Pentane/EtOAc, from 1:0 to 7:3). ^{1}H NMR ($CDCl_3$) δ 8.29-8.26 (m, 2H), 7.49-7.37 (m, 4H), 3.61-3.55 (m, 1H), 2.12-2.00 (m, 2H), 1.67-1.57 (m, 2H), 0.93 (td, J = 7.5, 1.4, Hz, 6H) ppm. ^{13}C NMR ($CDCl_3$) δ 134.0 (d), 130.3 (s), 130.0 (d), 128.5 (d), 128.4 (d), 80.7 (d), 25.9 (t), 10.7 (q) ppm. IR (neat) 1572, 1455, 1337, 1150 cm^{-1}. MS (ES^+): 192 $[M+H]^+$. HRMS calcd for $C_{12}H_{18}NO$ $[M+H]^+$ 192,1388, found 192.1401.



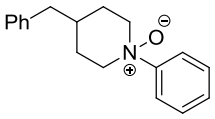
Compounds 2g and **2g'**. Flash chromatography (Pentane/EtOAc, from 1:0 to 6:4), 1:1 ratio of **2g** and **2g'**. ^{1}H NMR ($CDCl_3$) δ 8.27-8.18 (m, 4H), 7.47-7.32 (m, 11H), 7.12-7.04 (m, 3H), 5.02 (s, 2H), 4.99 (s, 1.4 H) ppm.^{13}C NMR ($CDCl_3$) δ 163.3 (s, $^{1}J_{C\text{-}F}$ = 253 Hz), 163.0 (s, $^{1}J_{C\text{-}F}$ = 253 Hz), 137.4 (d), 137.0 (d), 134.4 (d), 133.3 (d), 132.8 (s), 131.6 (d), 131.1 (d, $^{3}J_{C\text{-}F}$ = 9 Hz), 130.9 (d, $^{3}J_{C\text{-}F}$ = 9 Hz), 130.6 (d), 130.4 (d), 130.1 (s), 126.6 (s, $^{4}J_{C\text{-}F}$ = 3 Hz), 115.8 (d, $^{2}J_{C\text{-}F}$ = 21 Hz), 115.5 (d, $^{2}J_{C\text{-}F}$ = 21 Hz), 71.0 (t), 70.2 (t) ppm. IR (neat) 1597, 1503, 1231, 1147, 846, 703 cm^{-1}. MS (ES^+): 230 $[M+H]^+$. HRMS calcd for $C_{14}H_{13}FNO$ $[M+H]^+$ 230.0981, found 230.0965.



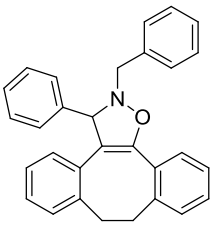
Compounds 2h and **2h'.** Flash chromatography (Pentane/EtOAc, 1:0 to 6:4), 1:1 ratio of **2h** and **2h'**. ^{1}H NMR ($CDCl_3$) δ 8.22-8.18 (m, 4H), 7.47 (dd, J = 7.9, 2.0 Hz, 2H), 7.43-7.37 (m, 7H), 7.32 (d, J = 8.7 Hz, 2H), 6.95-6.89 (m, 4H), 5.04 (d, J = 14.0 Hz, 2H), 4.97 (d, J = 14.0 Hz, 2H), 3.83 (s, 3H), 3.82 (s, 3H) ppm. ^{13}C NMR ($CDCl_3$) δ 161.0 (s), 160.0 (s), 133.9 (d), 133.97(d), 133.3 (s), 130.8 (d), 130.5 (d), 130.3 (d), 129.1 (d), 128.9 (d), 128.8 (d), 128.5 (d), 128.3 (d), 125.1 (s), 123.3 (s), 114.3 (d), 113.7 (d), 70.6 (t, x2), 55.2 (q) ppm. IR (neat) 1611, 1513, 1303, 1250, 1176, 1030, 903, 724 cm^{-1}. MS (ES^+): 242 $[M+H]^+$. HRMS calcd for $C_{15}H_{16}NO_2$ $[M+H]^+$ 242.1181, found 242.1171.



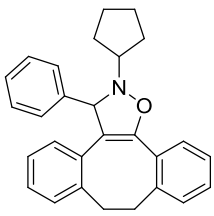
Compound 2i.[4] Flash chromatography (CH_2Cl_2/MeOH, from 1:0 96:4, silica deactivated with aqueous NH_4OH). ^{1}H NMR ($CDCl_3$) δ 7.97 (dd, J = 7.3, 1.6 Hz, 2H), 7.49-7.38 (m, 3H), 7.30-7.22 (m, 3H), 7.06 (dd, J = 7.9, 1.6 Hz, 1H), 5.06 (d, J = 15.5 Hz, 1H), 4.60 (d, J = 15.5 Hz, 1H), 4.17-4.11 (m, 1H), 3.79-3.66 (m, 2H), 2.84 (dt, J = 16.4, 5.0 Hz, 1H) ppm. ^{13}C NMR ($CDCl_3$) δ 131.1 (s), 129.9 (s), 129.1 (d), 129.0 (d), 128.5 (d), 127.5 (d), 126.7 (d), 126.2 (d), 120.5 (d), 71.3 (t), 65.5 (t), 26.2 (t) ppm. IR (neat) 1489, 903, 726 cm^{-1}. MS (ES$^+$): 226 $[M+H]^+$.



Compound 2j. Could not be purified by chromatography, NMR provided for the crude. ^{1}H NMR ($CDCl_3$) δ 7.86 (d, J = 8.0 Hz, 2H), 7.48 (t, J = 7.8 Hz, 2H), 7.31-7.21 (m, 4H), 7.14 (d, J = 8.0 Hz, 2H), 4.07 (t, J = 11.0 Hz, 2H), 3.35-3.30 (m, 2H), 2.67 (d, J = 8 .4 Hz, 2H), 2.56-2.47 (m, 2H), 2.15 (m, 1H), 1.50-1.43 (m, 2H) ppm. MS (ES$^+$): 268 $[M+H]^+$. HRMS calcd for $C_{18}H_{22}NO$ $[M+H]^+$ 268.1701, found 268.1699.



Compound 3a.[5] Flash chromatography (Pentane/EtOAc from 1:0 to 9:1). ^{1}H NMR ($CDCl_3$) δ 7.53-7.50 (m, 3H), 7.39-7.35 (m, 2H), 7.33-7.23 (m, 7H), 7.18-7.11 (m, 4H), 7.09 (td, J = 7.5, 1.5 Hz, 1H), 7.03 (td, J = 7.5, 1.5 Hz, 1H), 6.97 (dd, J = 7.5, 1.5 Hz, 1H), 5.24 (s, 1H), 4.65 (d, J = 13.0 Hz, 1H), 4.31 (d, J = 13.0 Hz, 1H), 3.43-3.35 (m, 1H), 3.22-3.15 (m, 1H), 3.12-3.05 (m, 1H), 2.97-2.90 (m, 1H) ppm. ^{13}C NMR ($CDCl_3$) δ 147.8 (s), 141.3 (s), 140.9 (s), 138.9 (s), 136.1 (s), 132.4 (s), 130.9 (s), 129.9 (d), 129.6 (d), 129.3 (d), 128.5 (d), 128.4 (d, x2), 127.8 (d), 127.6 (d), 127.4 (d), 126.9 (d), 126.8 (d), 125.6 (d), 125.4 (d), 110.3 (s), 63.0 (s), 36.8 (t), 32.9 (t) ppm. IR (neat) 1502, 1382, 872, 735 cm^{-1}. MS (ES$^+$): 416 $[M+H]^+$.

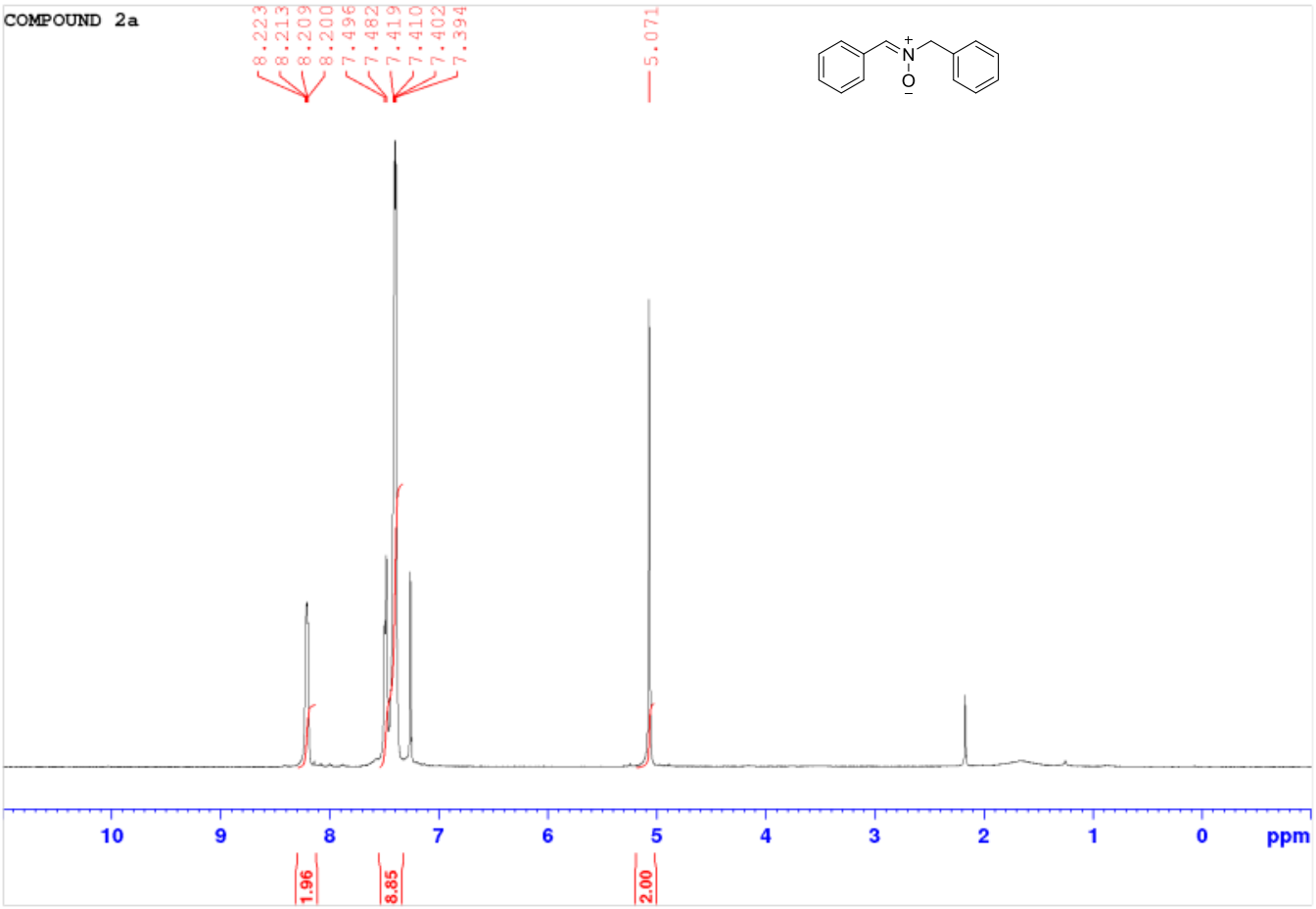


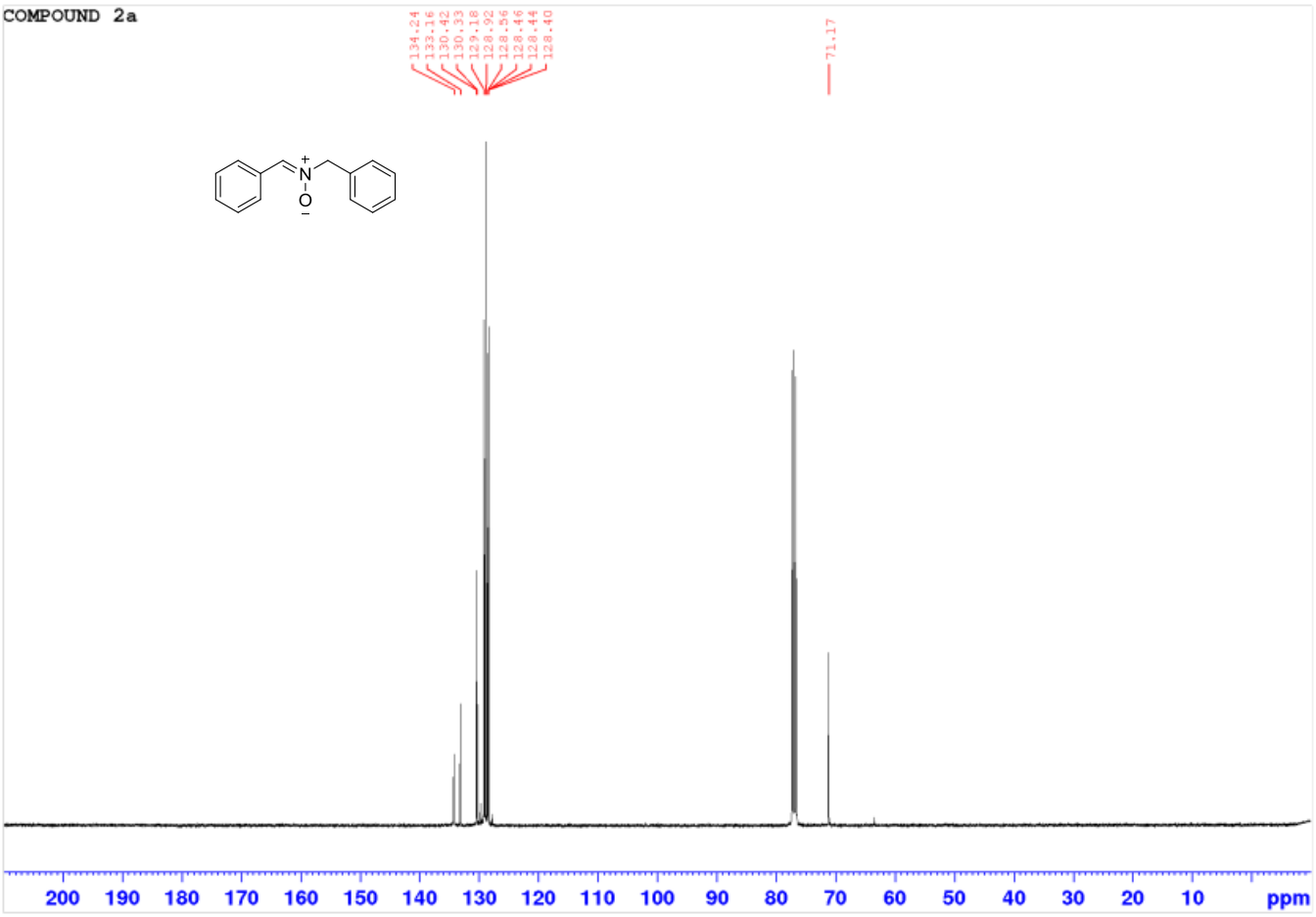
Compound 3e. Flash chromatography (eluent Pentane/EtOAc from 1:0 to 94:6). ^{1}H NMR ($CDCl_3$) δ 7.42 (dd, J = 6.9, 1.8 Hz, 2H), 7.30 (t, J = 7.7 Hz, 2H), 7.27-7.22 (m, 1H), 7.17-7.13 (m, 4H), 7.08 (dd, J = 7.3, 1.6 Hz, 1H), 7.05 (dd, J = 3.3, 1.7 Hz, 1H), 7.01 (dd, J = 7.3, 1.7 Hz, 1H), 6.97 (dd, J = 7.6, 1.9 Hz, 1H), 5.26 (s, 1H), 3.83 (quint, J = 6.6 Hz, 1H), 3.45-3.38 (m, 1H), 3.23-3.09 (m, 2H), 3.02-2.95 (m, 1H), 2.16-2.03 (m, 2H), 1.97-1.74 (m, 4H), 1.70-1.61 (m, 2H) ppm. ^{13}C NMR ($CDCl_3$) δ 147.6 (s), 142.1 (s), 140.9 (s), 138.8 (s), 132.6 (s), 130.9 (d), 129.9 (d), 129.5 (d), 128.5 (d), 128.4 (d), 127.7 (d), 127.3 (d), 126.9 (d), 126.7 (d), 125.6 (d), 125.4 (d), 110.2 (s), 77.7 (d), 68.0 (d), 36.9 (t), 33.0 (t), 31.2 (t), 30.3 (t), 24.8 (t), 24.7 (t) ppm. IR (neat) 1674, 1493, 1451, 1029, 905, 729 cm^{-1}. MS (ES$^+$): 394 $[M+H]^+$. HRMS calcd for $C_{28}H_{28}NO$ $[M+H]^+$ 394.2171, found 394.2183.

5. References

1) C. Matassini, C. Parmeggiani, F. Cardona, A. Goti, *Org. Lett.* 2015, **17**, 4082.
2) D. A. Evans, H. –J. Song, K. R. Fandrick, *Org. Lett.* 2006, **8**, 3351.
3) C. Gella, E. Ferrer, R. Alibés, F. Busqué, P. March, M. Figueredo, J. Font, *J. Org. Chem.* 2009, **74**, 6365.
4) H. Ueda, K. Yoshida, H. Tokuyama, *Org. Lett.* 2014, **16**, 4194.
5) C. S. McKay, J. Moran, J. P. Pezacki, *Chem. Commun.* 2010, **46**, 931.

6. Copies of ^{1}H and ^{13}C NMR Spectra

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130.42
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128.56
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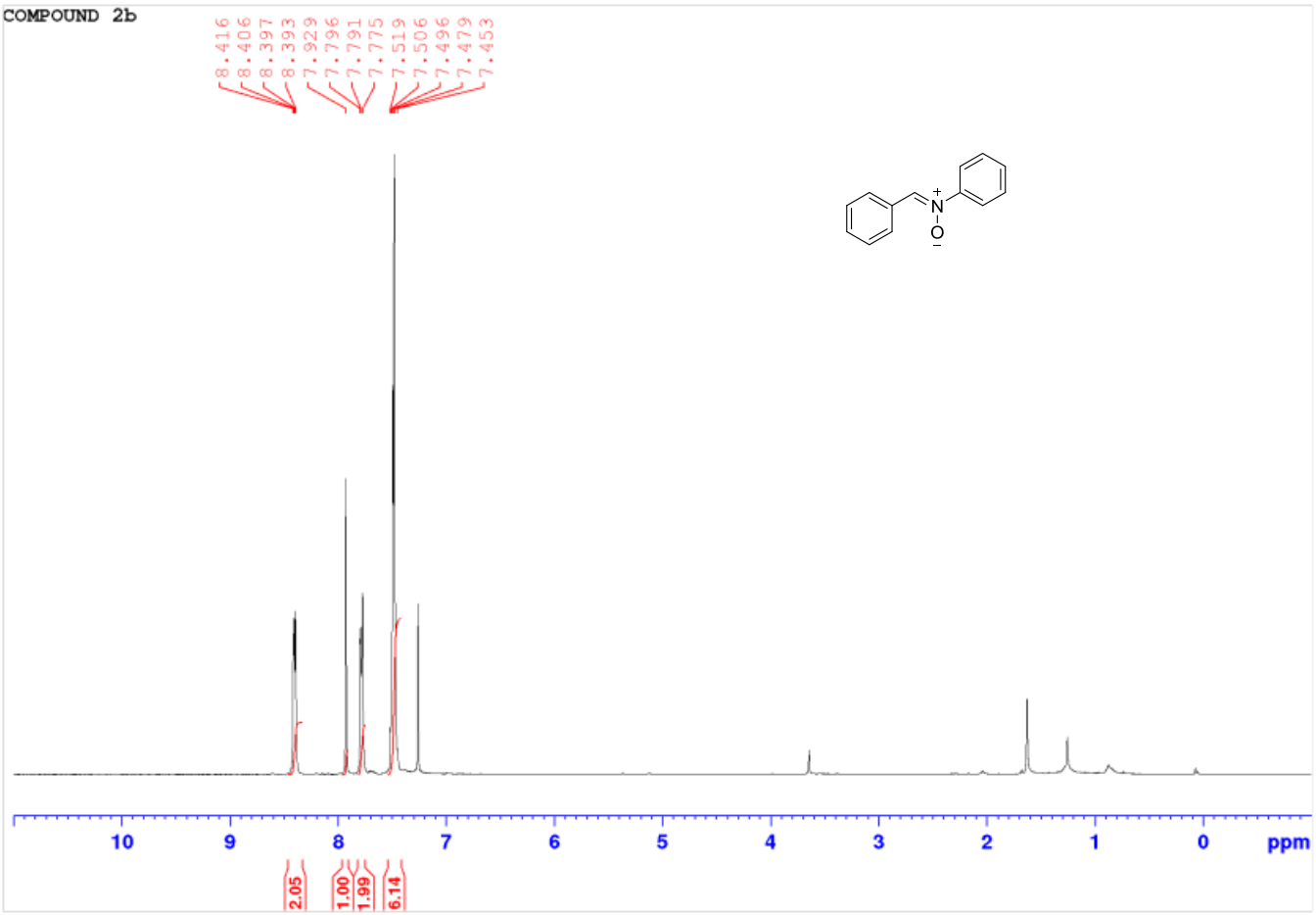
128.40
71.17
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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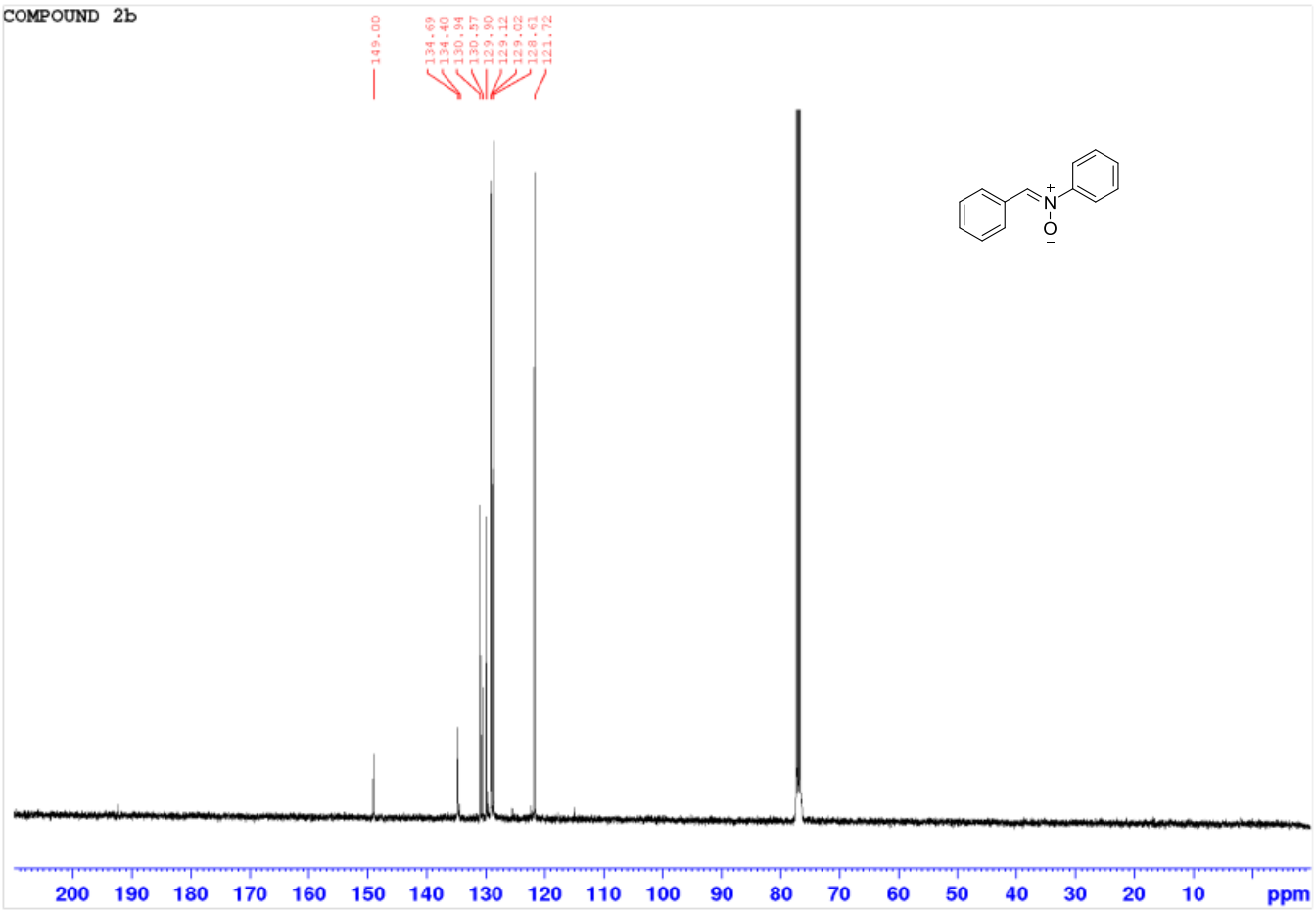
7.519
7.506
7.496
7.479
7.453
10
9
8
7
6
5
4
3
2
1
0
ppm
2.05
1.00
1.99
6.14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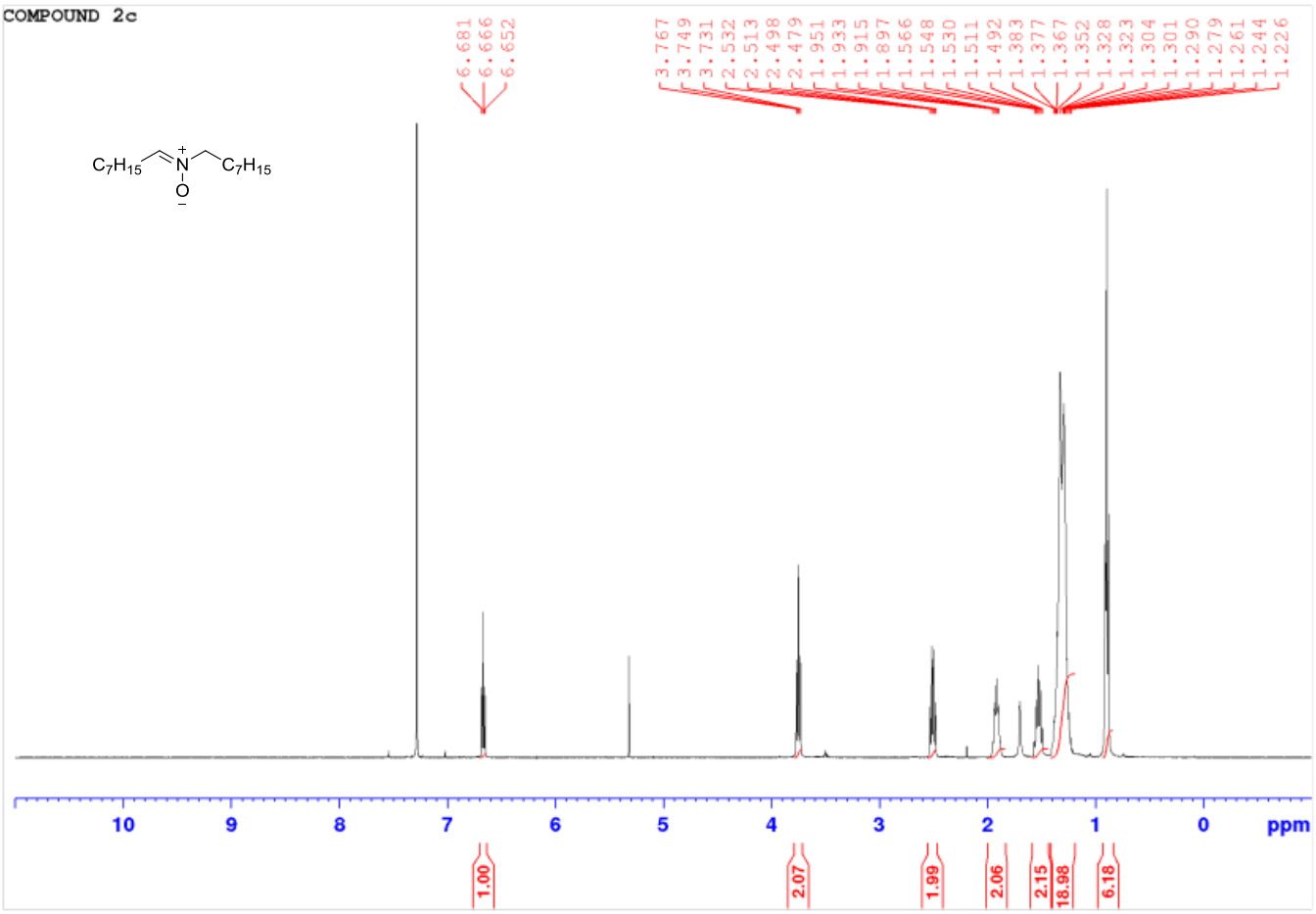
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1.933
1.915
1.897
1.566
1.548
1.530
1.511
1.492
1.383
1.377
1.367
1.352
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1.244
1.226
C7H15
N
O
C7H15
10
9
8
7
6
5
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ppm
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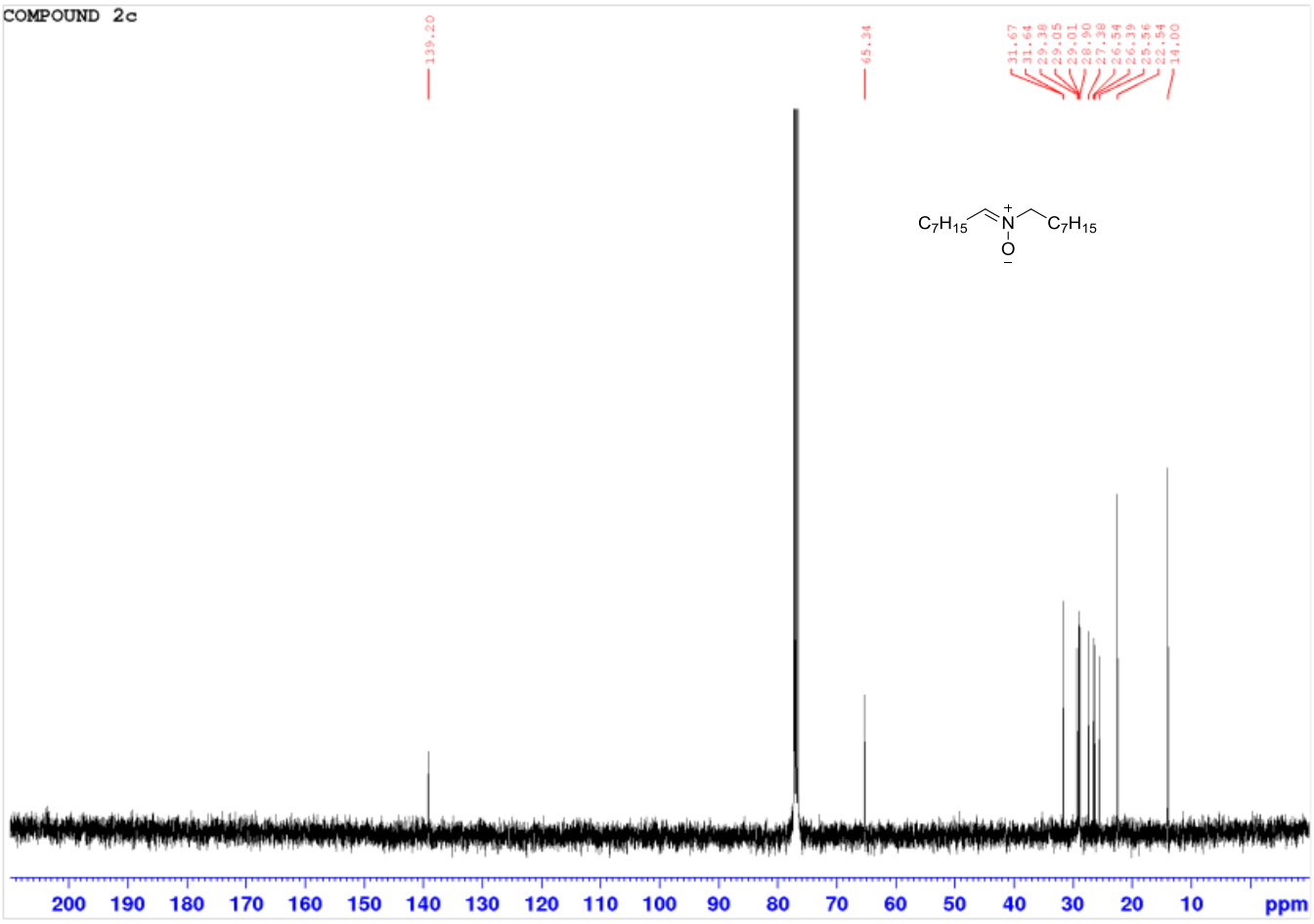
6.18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31.64
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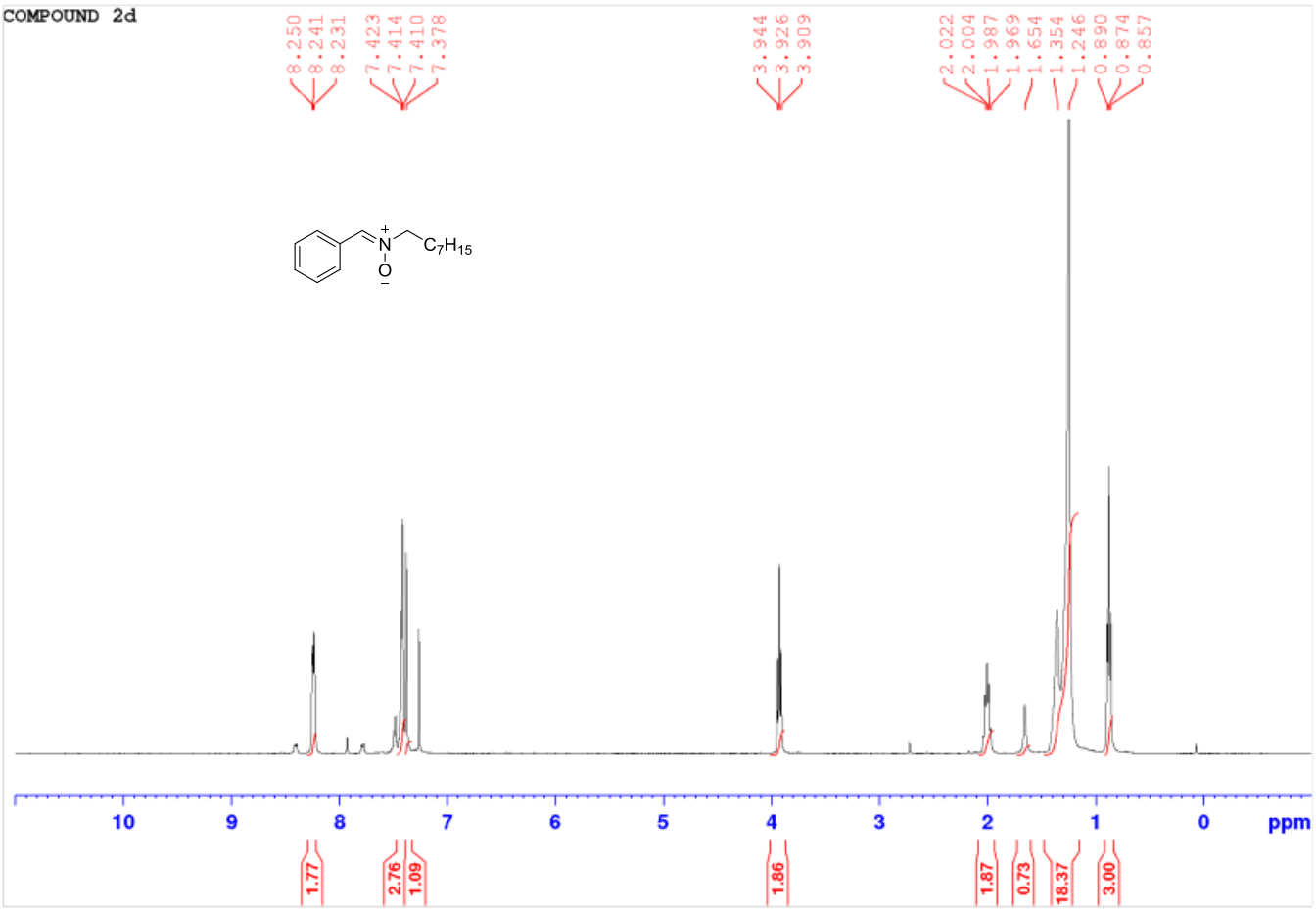
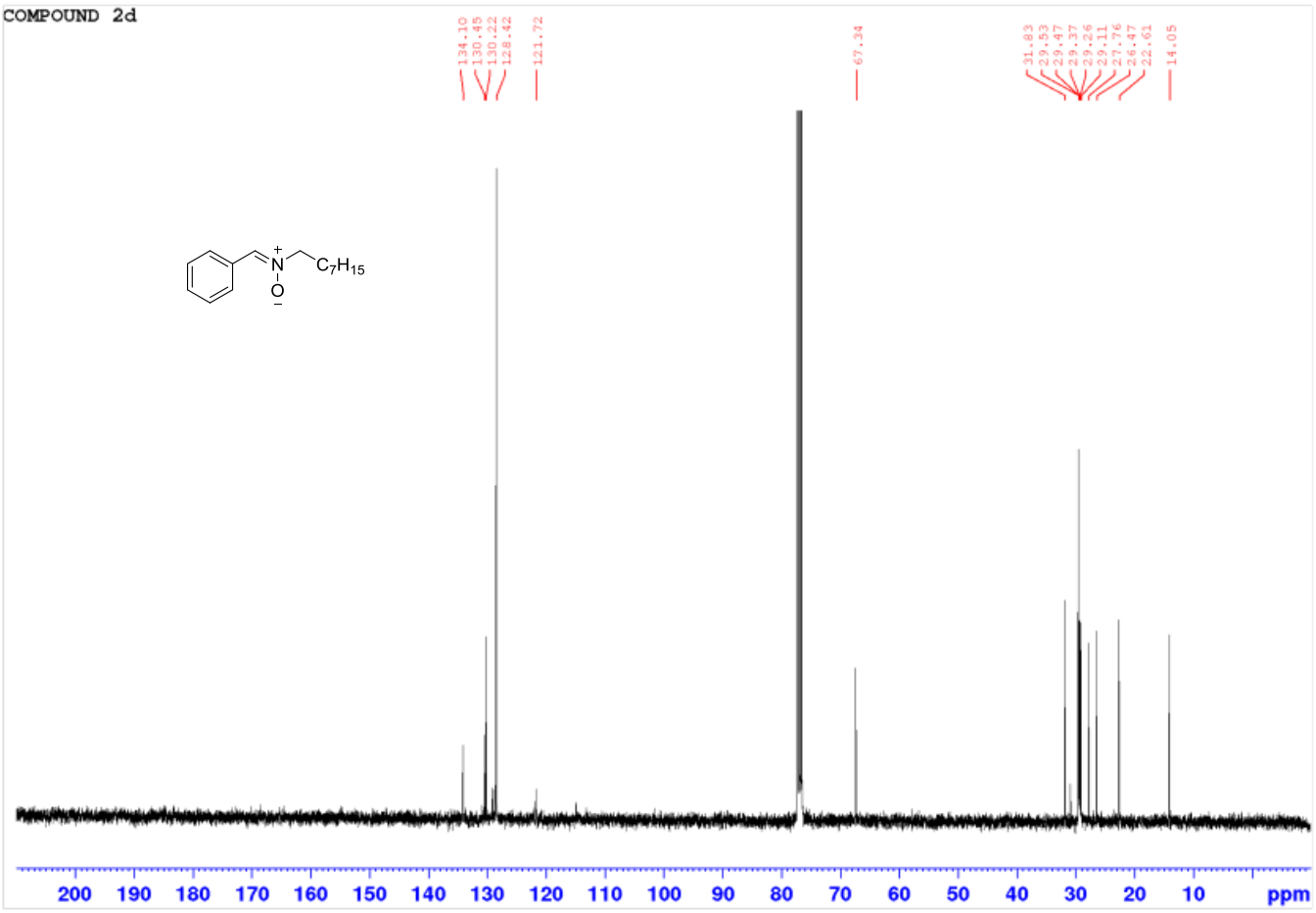
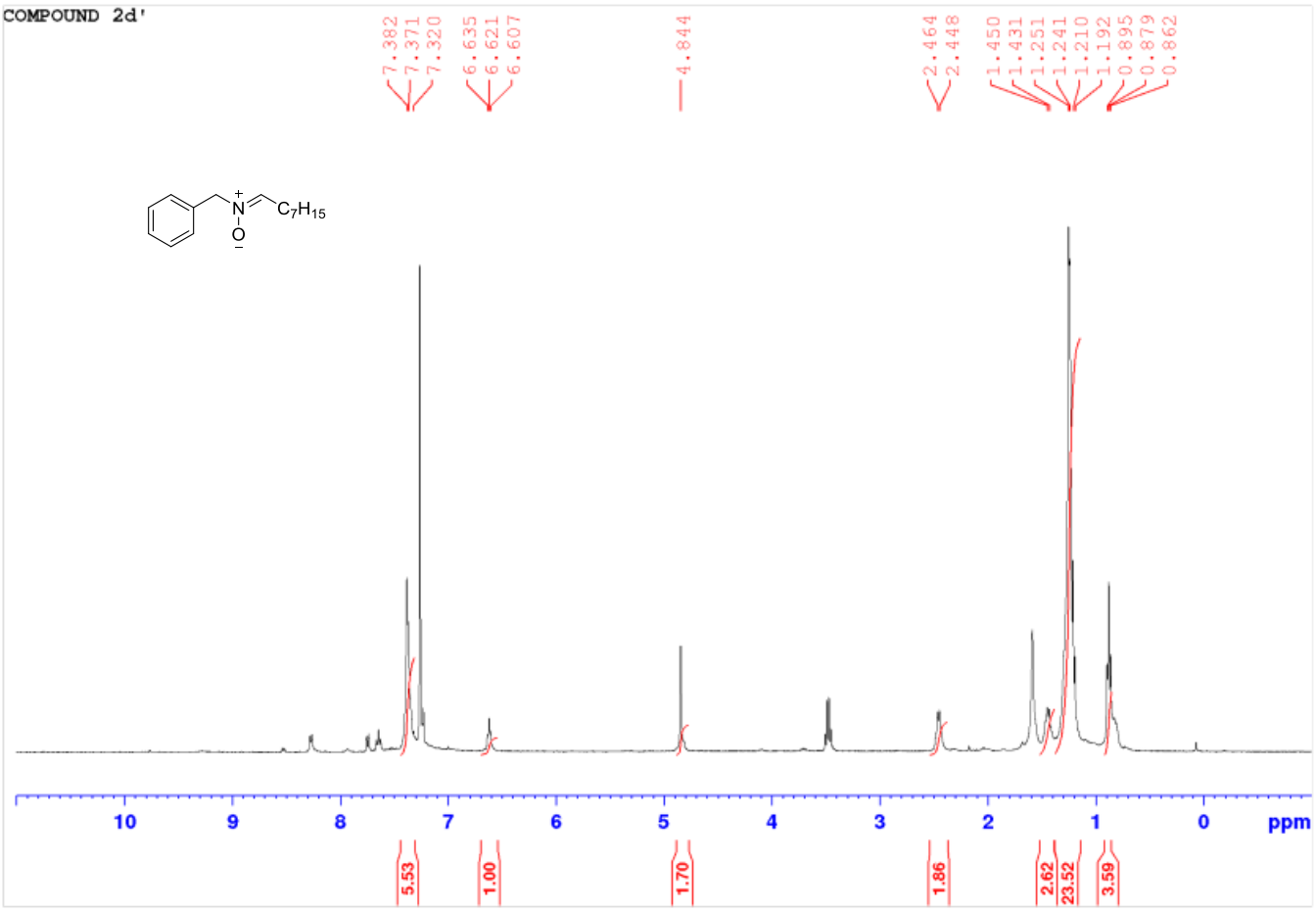
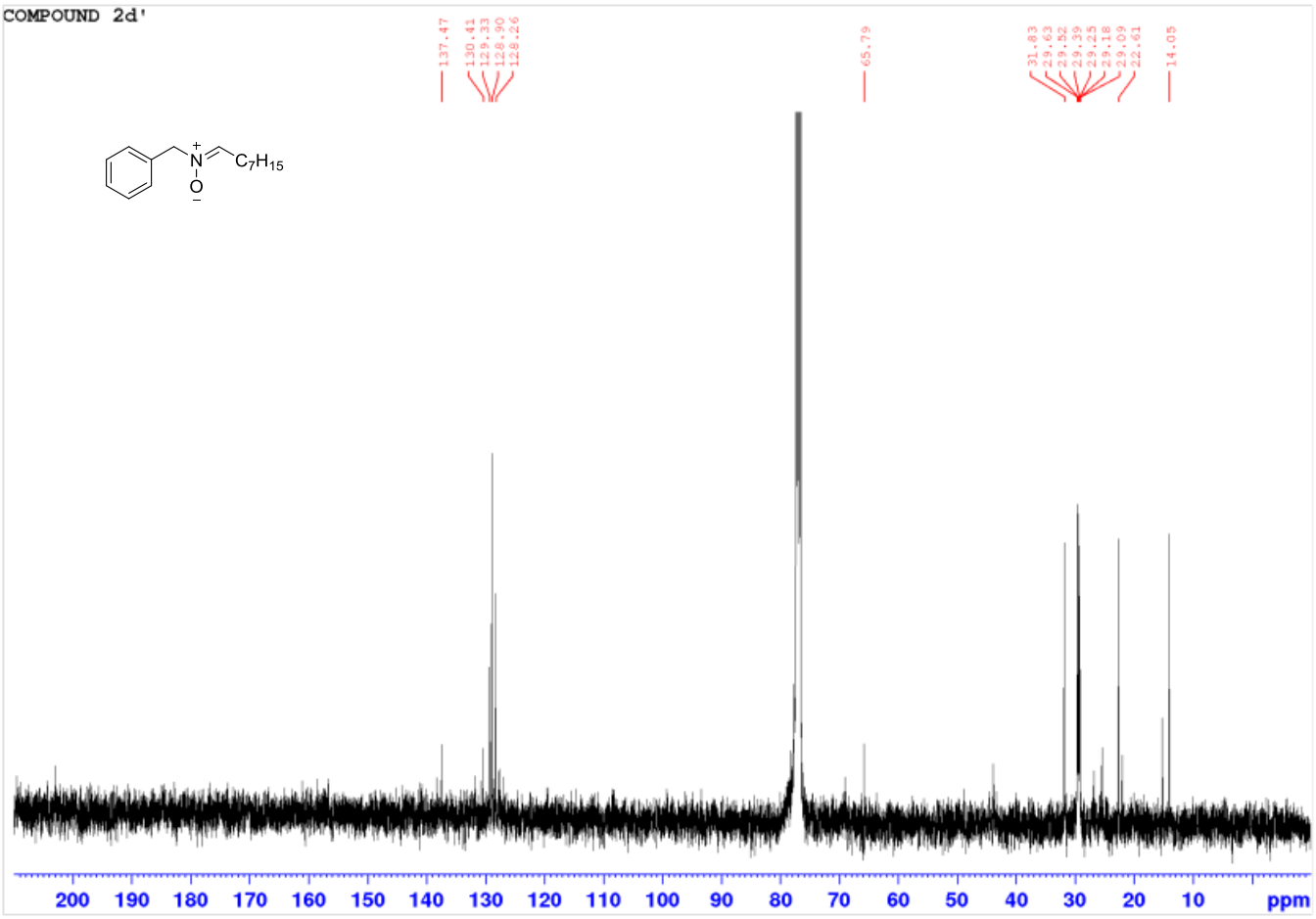
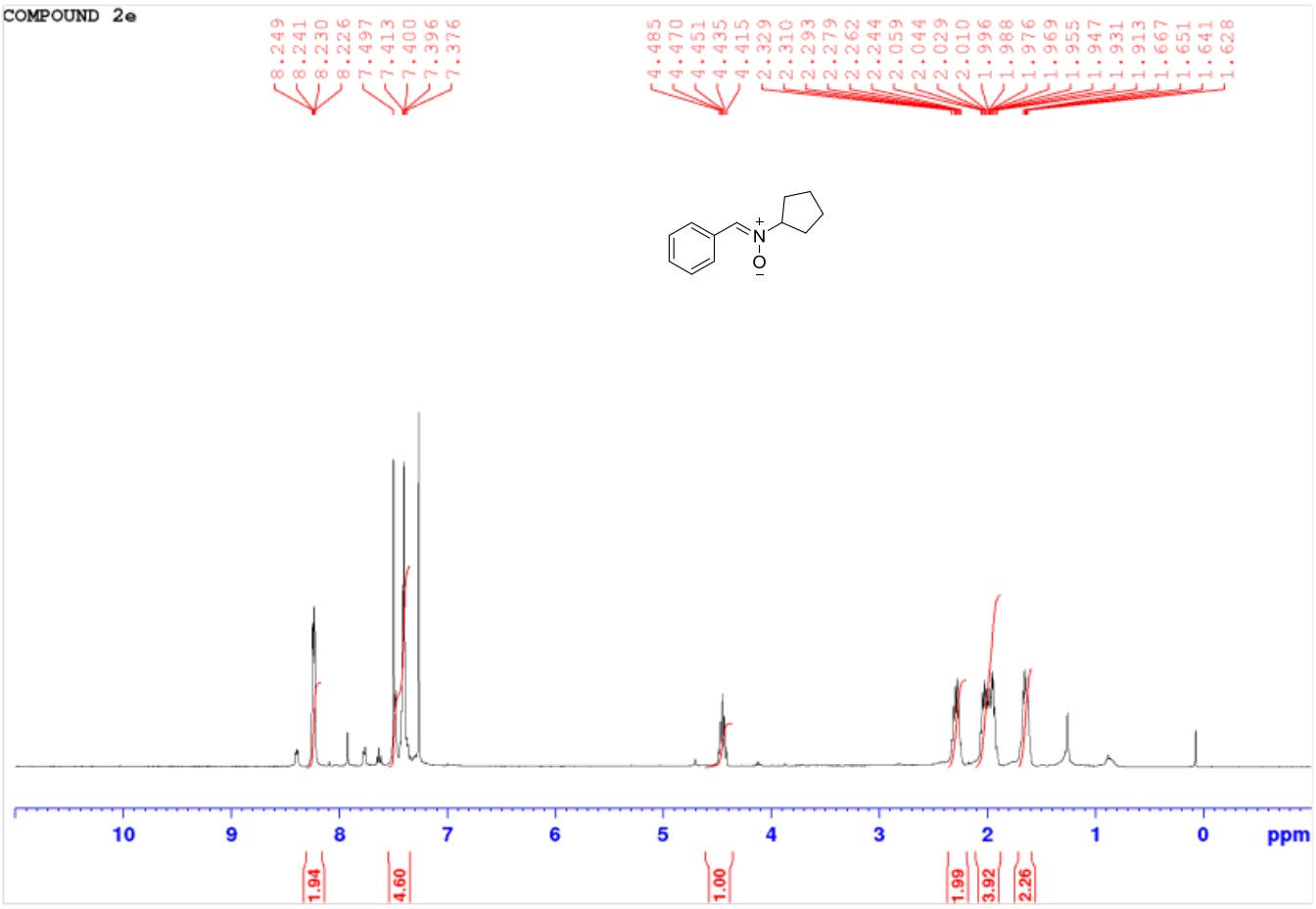
28.90
27.38
26.54
26.39
25.56
22.54
14.00
C_7H_{15}
N
O
C_7H_{15}
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

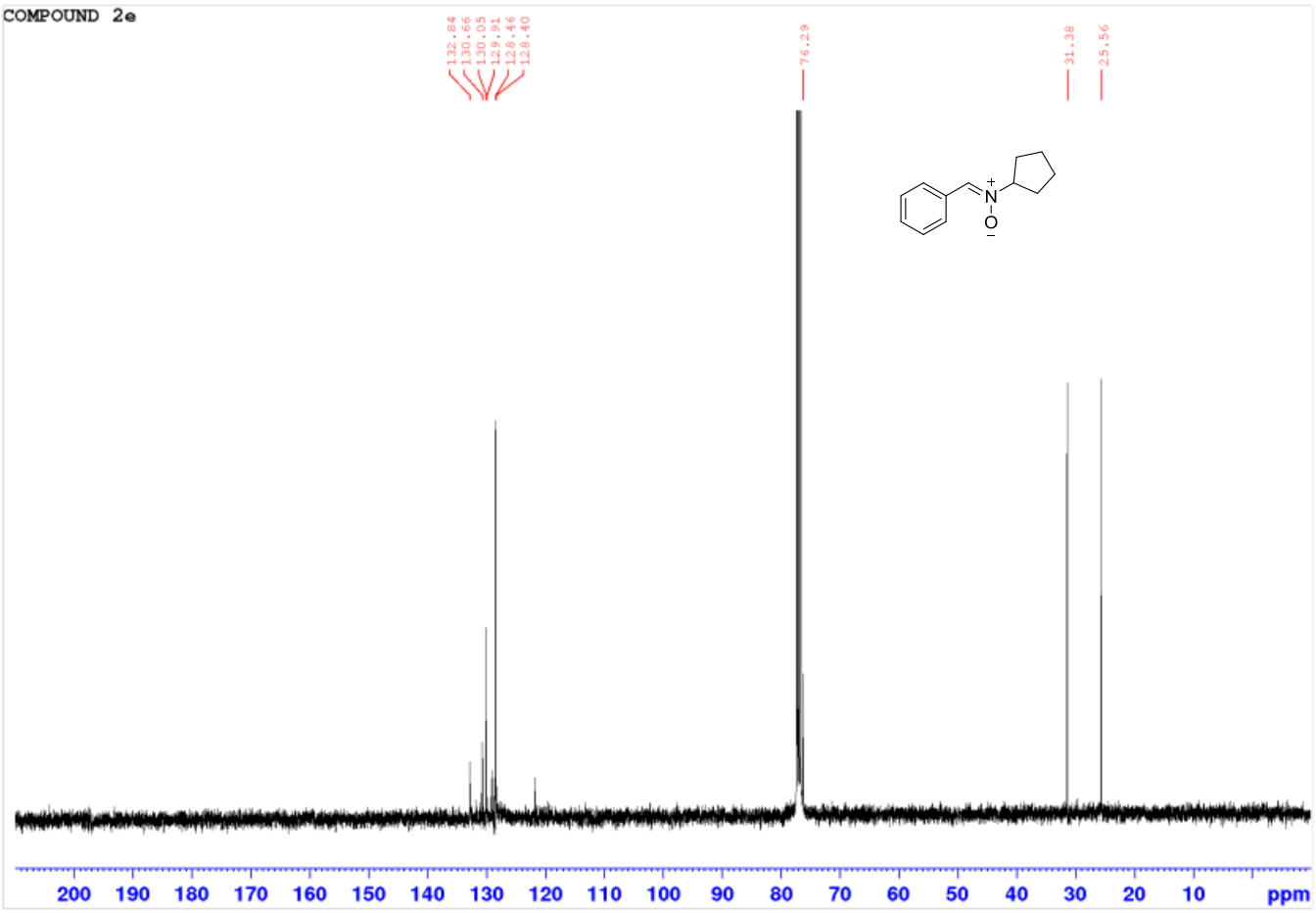

COMPOUND 2d
8.250
8.241
8.231
7.423
7.414
7.410
7.378
3.944
3.926
3.909
2.022
2.004
1.987
1.969
1.654
1.354
1.246
0.890
0.874
0.857
C7H15
10
9
8
7
6
5
4
3
2
1
0
ppm
1.77
2.76
1.09
1.86
1.87
0.73
18.37
3.00

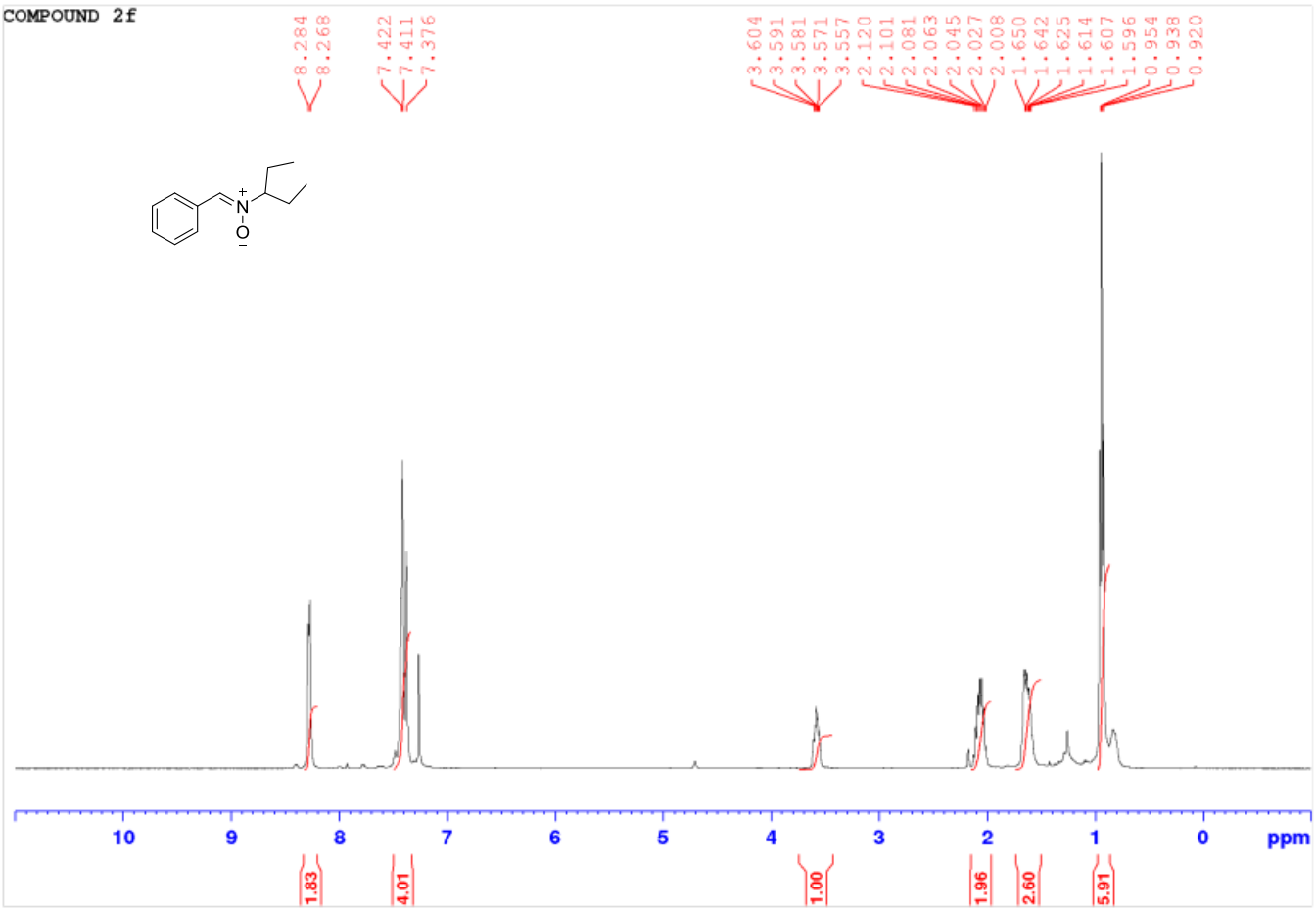

COMPOUND 2d
134.10
130.45
130.22
128.42
121.72
67.34
31.83
29.53
29.47
29.37
29.26
29.11
27.76
26.47
22.61
14.05
C_7H_{15}
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

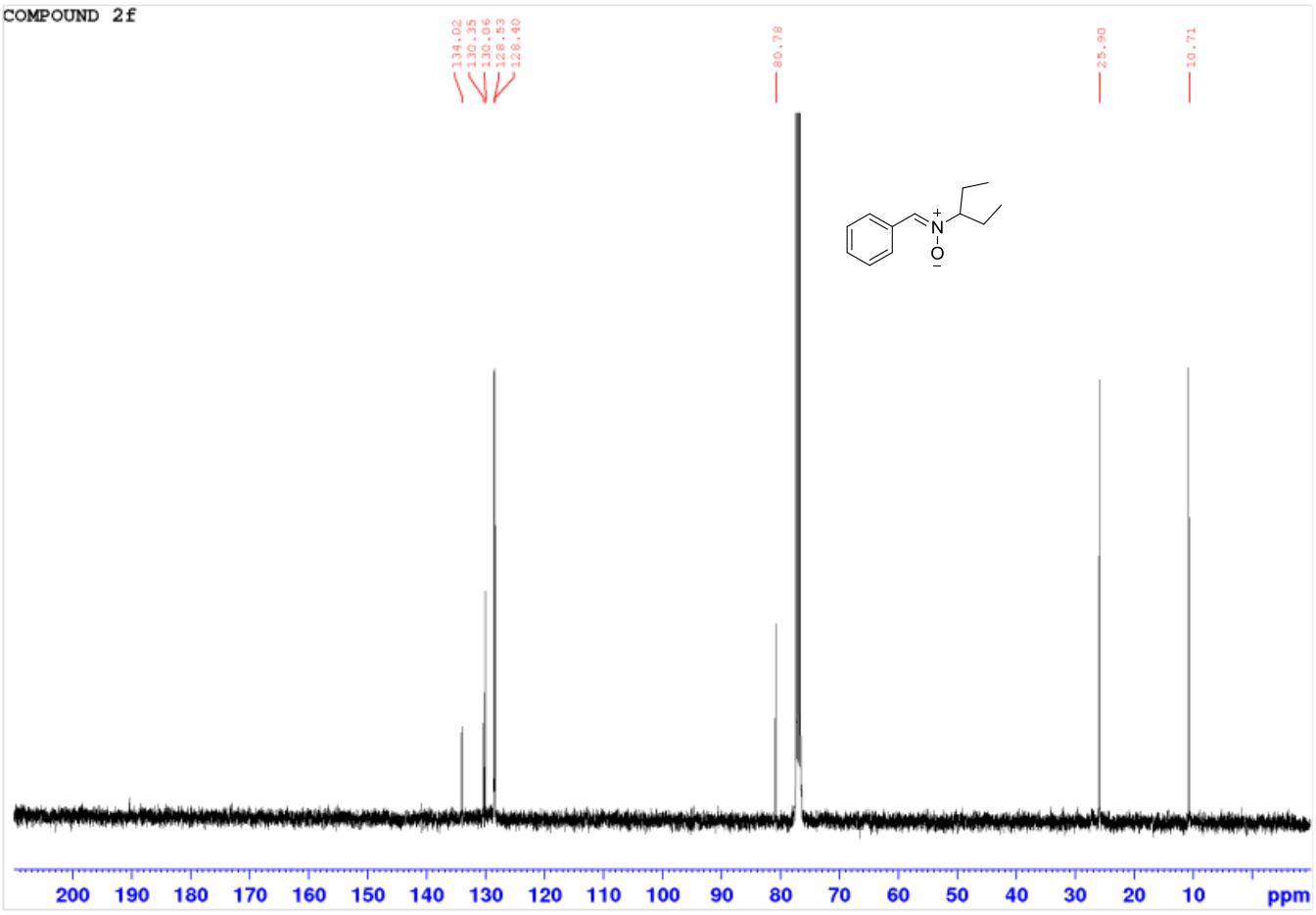

COMPOUND 2d'
7.382
7.371
7.320
6.635
6.621
6.607
4.844
2.464
2.448
1.450
1.431
1.251
1.241
1.210
1.192
0.895
0.879
0.862
C7H15
10
9
8
7
6
5
4
3
2
1
0
ppm
5.53
1.00
1.70
1.86
2.62
23.52
3.59

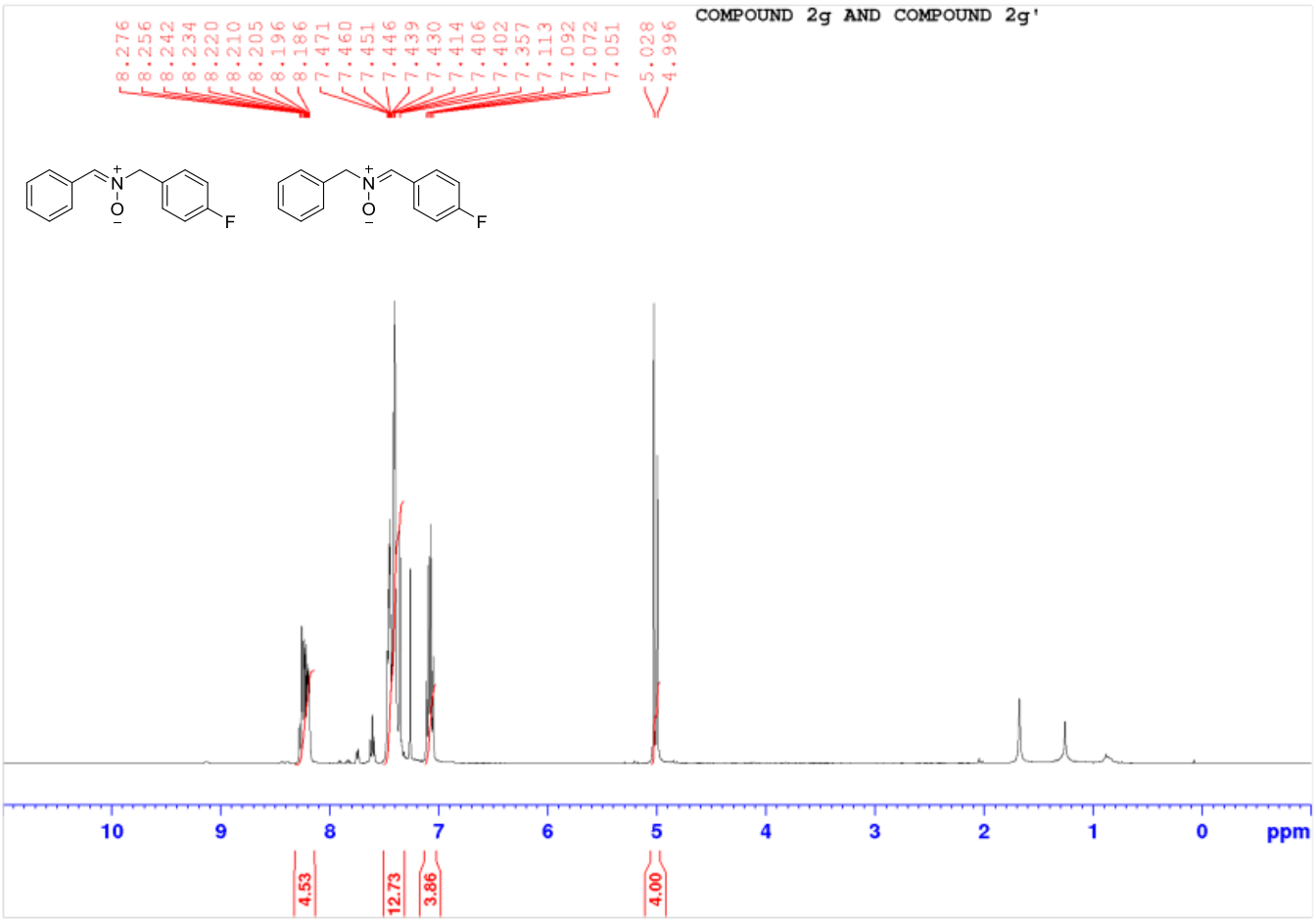

COMPOUND 2d'
137.47
130.41
129.33
128.90
128.26
65.79
31.83
29.63
29.52
29.39
29.25
29.18
29.09
22.61
14.05
C_7H_{15}
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

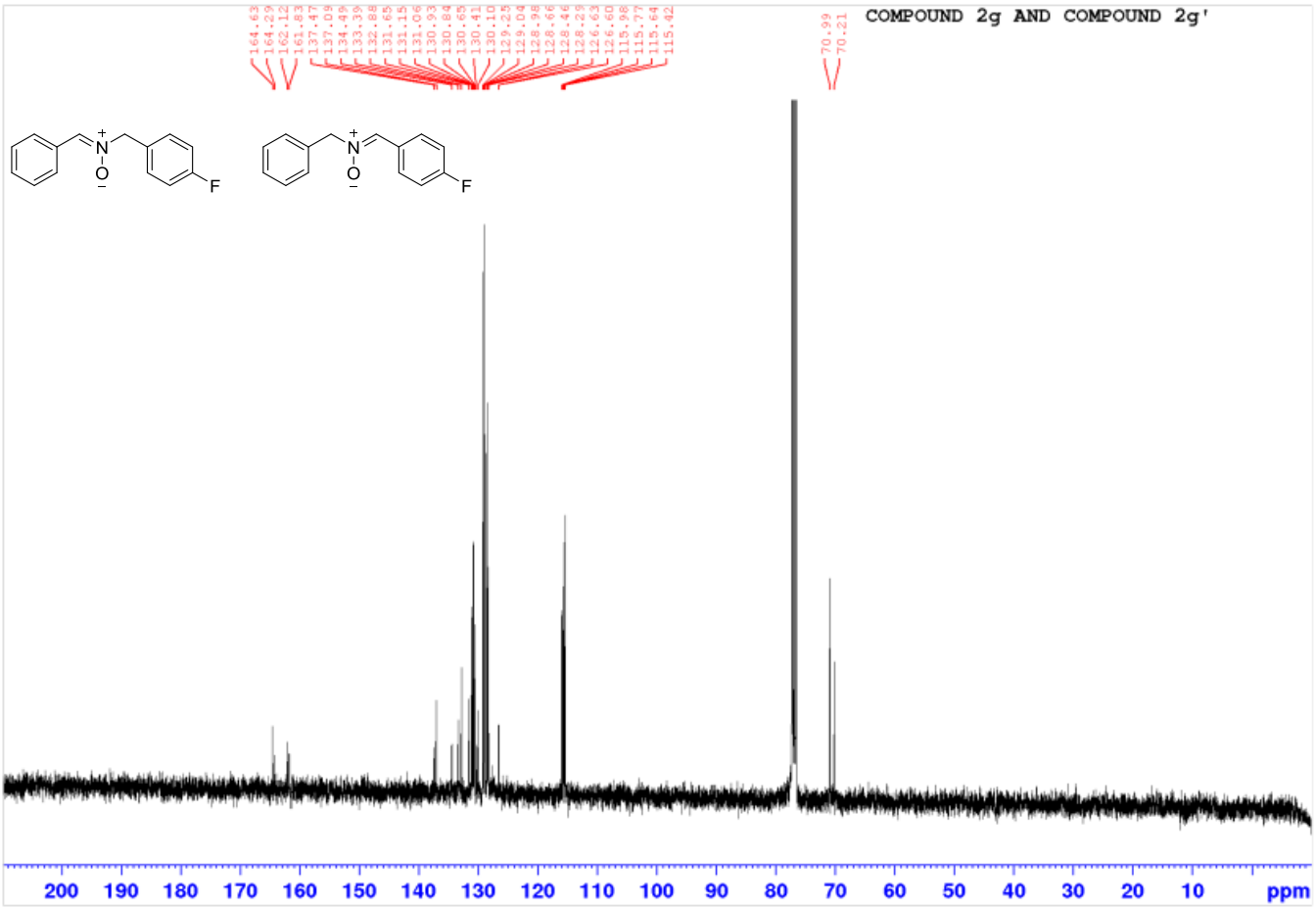

COMPOUND 2e
8.249
8.241
8.230
8.226
7.497
7.413
7.400
7.396
7.376
4.485
4.470
4.451
4.435
4.415
2.329
2.310
2.293
2.279
2.262
2.244
2.059
2.044
2.029
2.010
1.996
1.988
1.976
1.969
1.955
1.947
1.931
1.913
1.667
1.651
1.641
1.628
10
9
8
7
6
5
4
3
2
1
0
ppm
1.94
4.60
1.00
1.99
3.92
2.26

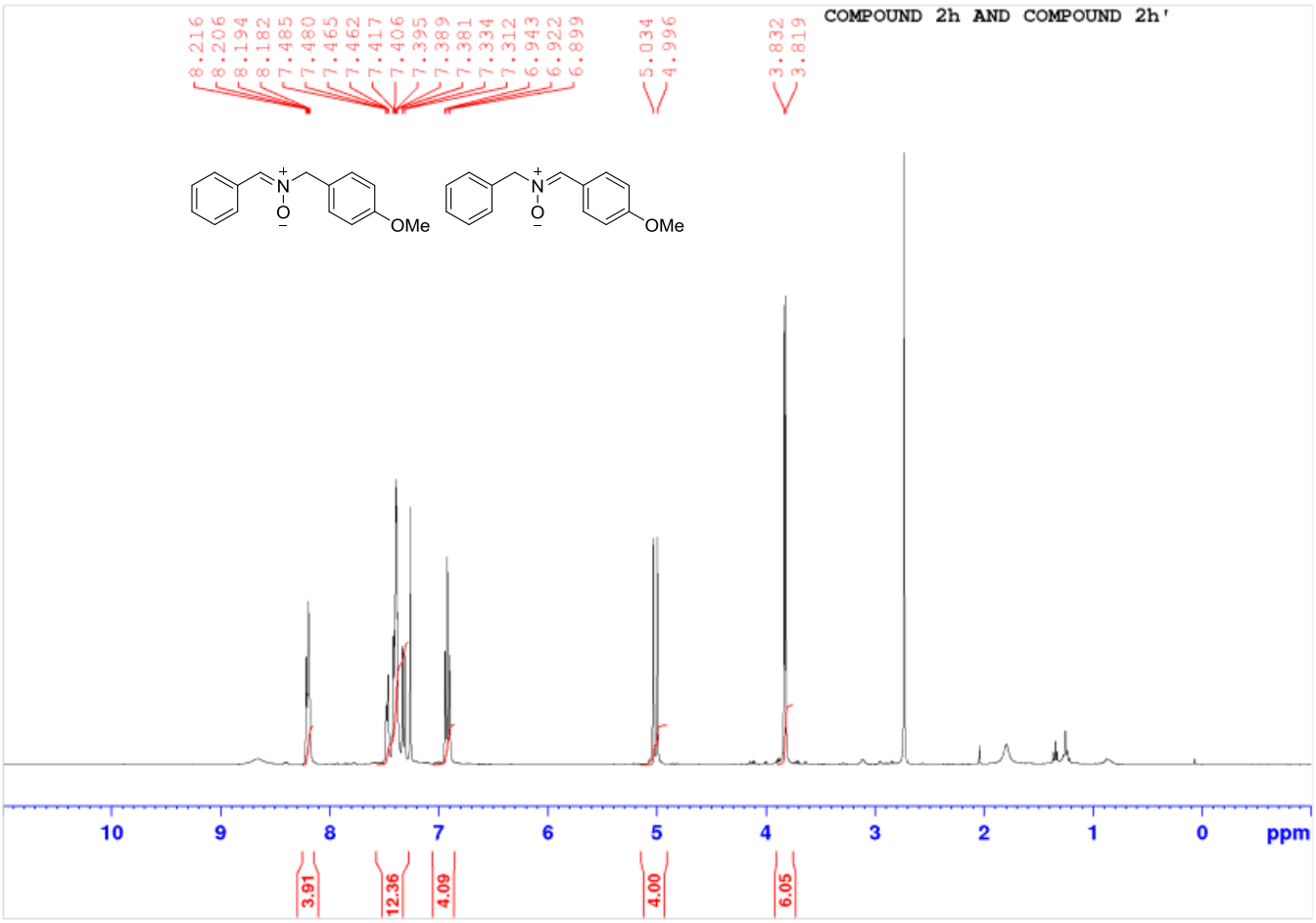

COMPOUND 2e
132.84
130.66
130.05
129.91
128.46
128.40
76.29
31.38
25.56
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

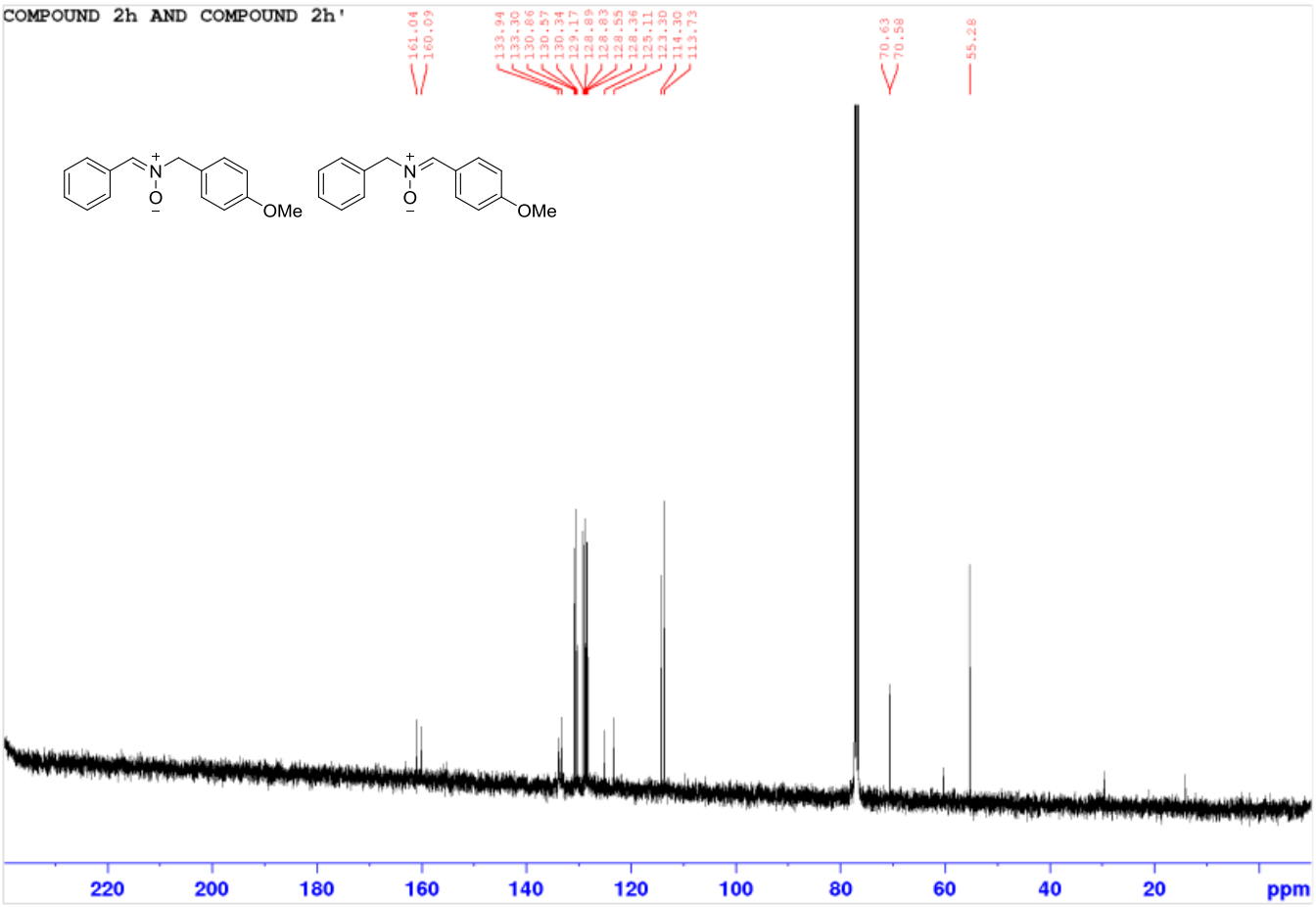

COMPOUND 2f
8.284
8.268
7.422
7.411
7.376
3.604
3.591
3.581
3.571
3.557
2.120
2.101
2.081
2.063
2.045
2.027
2.008
1.650
1.642
1.625
1.614
1.607
1.596
0.954
0.938
0.920
10
9
8
7
6
5
4
3
2
1
0
ppm
1.83
4.01
1.00
1.96
2.60
5.91

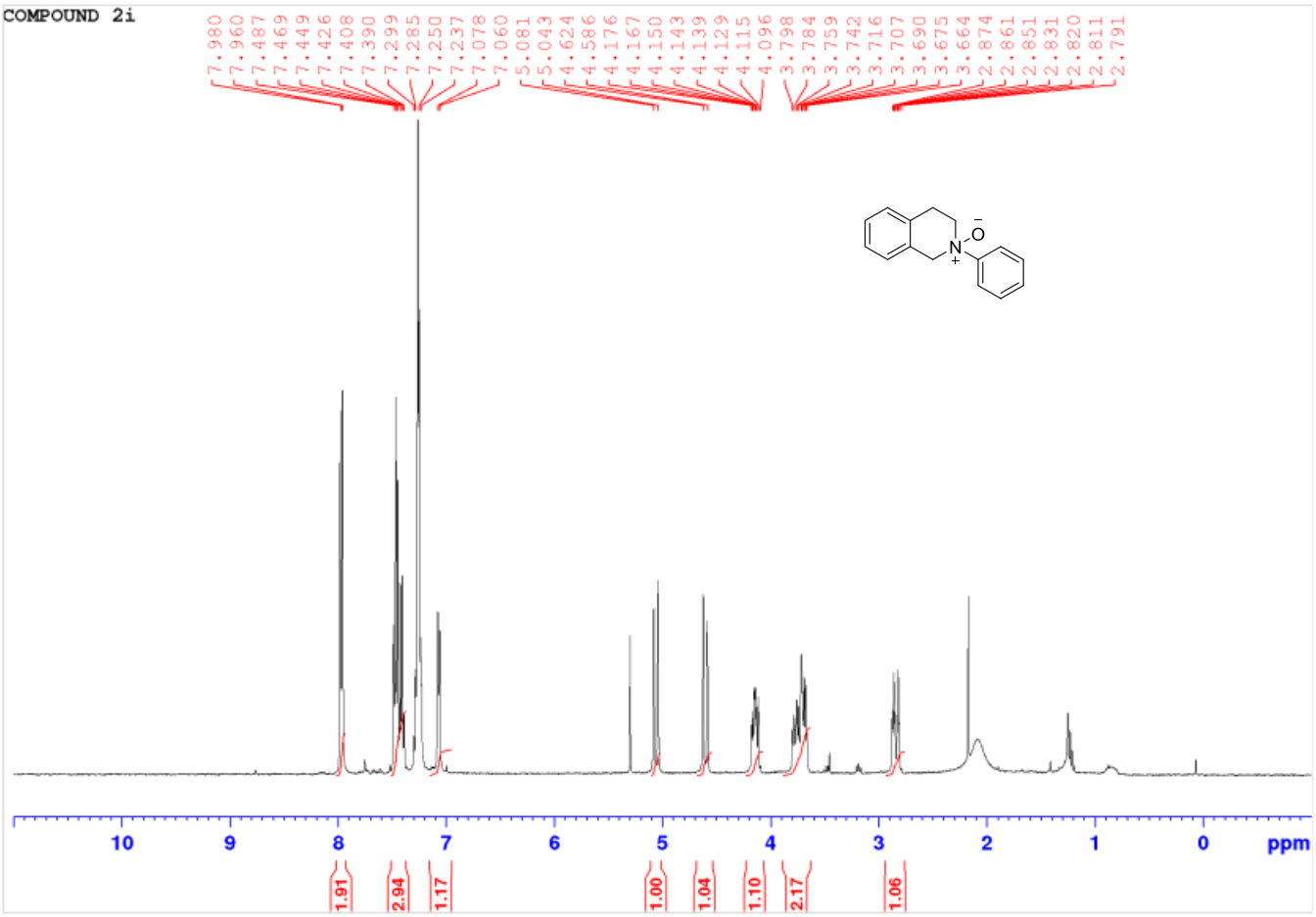

COMPOUND 2f
134.02
130.35
130.06
128.53
126.40
80.78
25.90
10.71
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

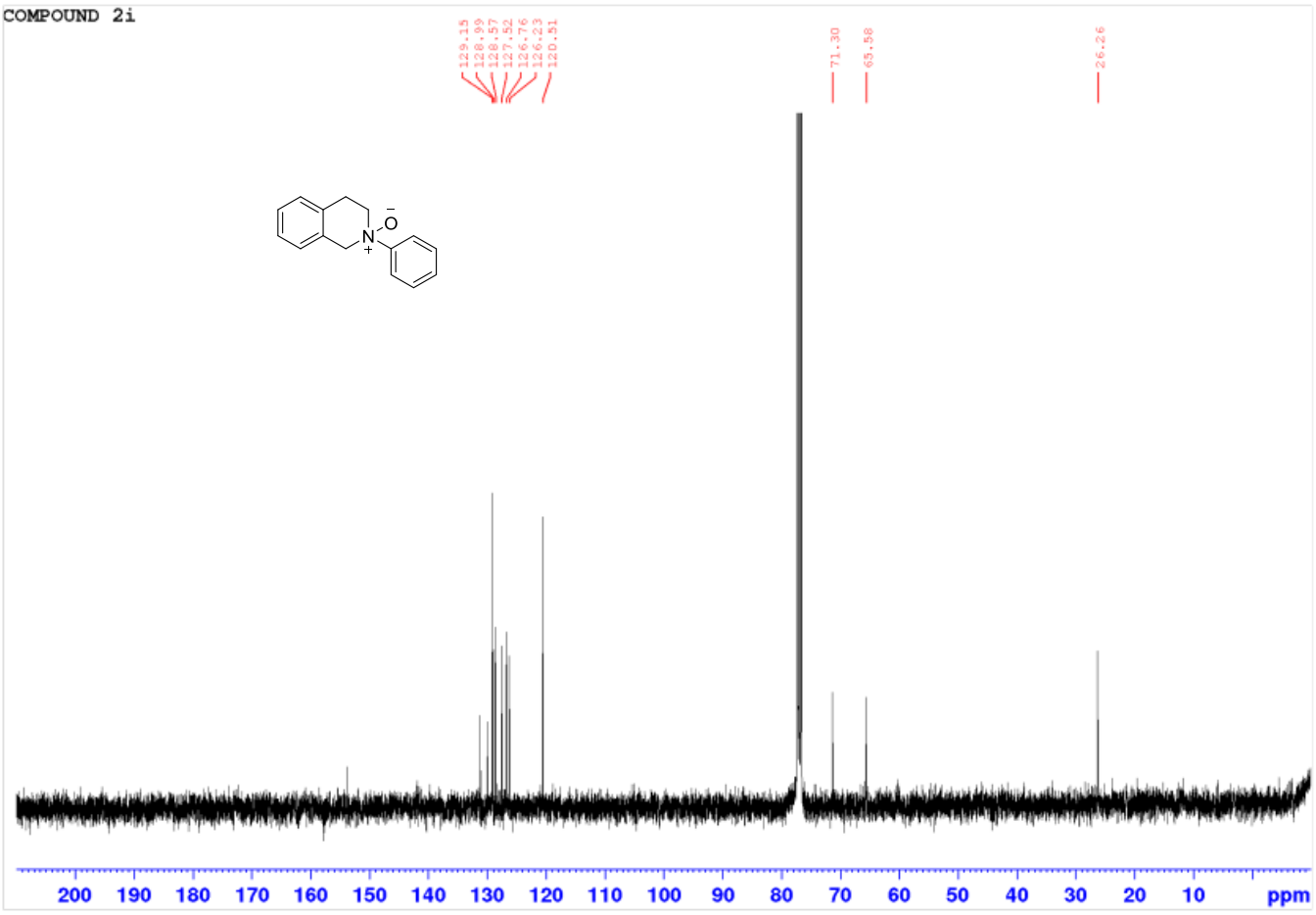

COMPOUND 2g AND COMPOUND 2g'
8.276
8.256
8.242
8.234
8.220
8.210
8.205
8.196
8.186
7.471
7.460
7.451
7.446
7.439
7.430
7.414
7.406
7.402
7.357
7.113
7.092
7.072
7.051
5.028
4.996
10
9
8
7
6
5
4
3
2
1
0
ppm
4.53
12.73
3.86
4.00

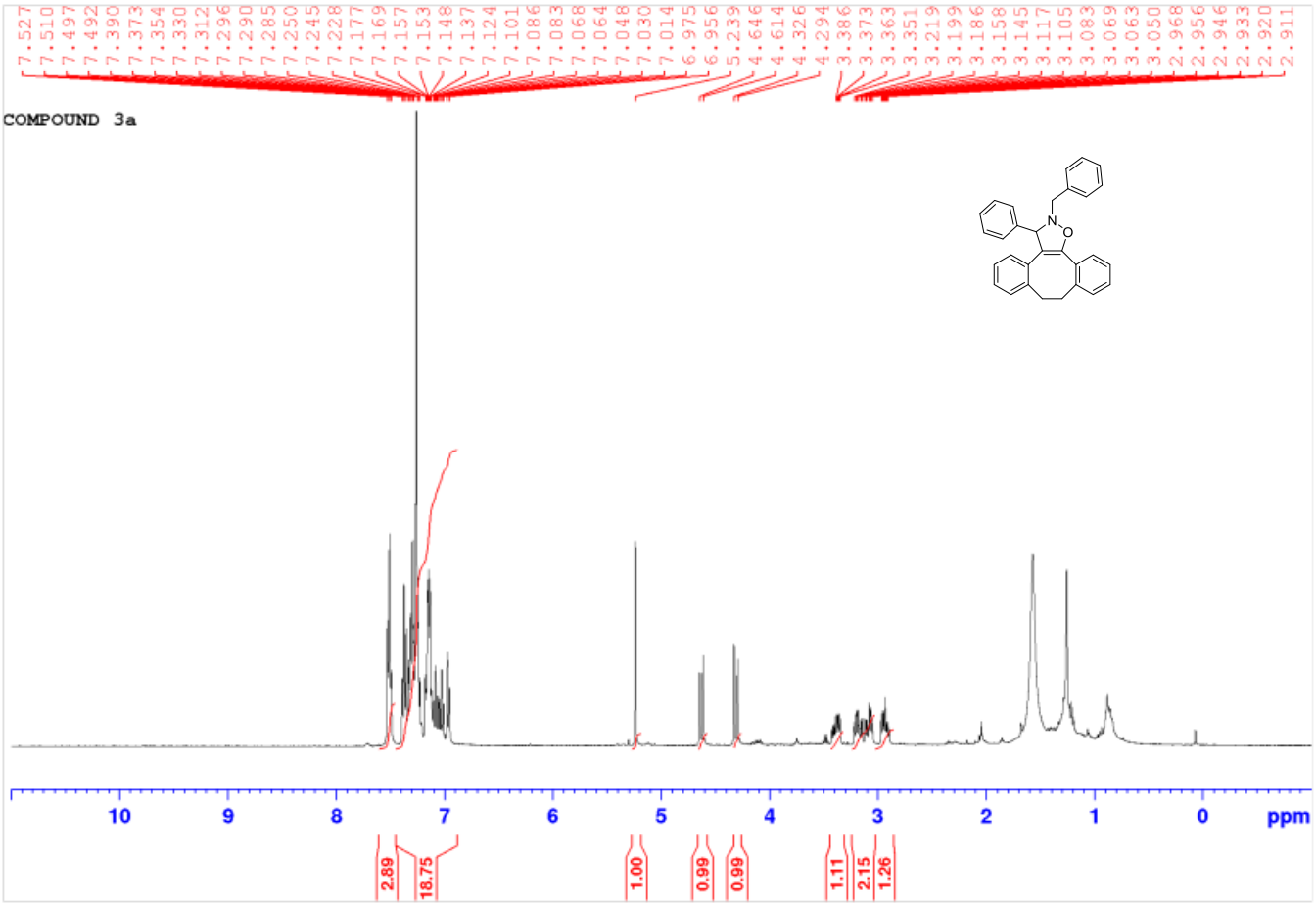

COMPOUND 2g AND COMPOUND 2g'
164.63
164.29
162.12
161.83
137.47
137.09
134.49
133.39
132.88
131.65
131.15
131.06
130.93
130.84
130.65
130.41
130.10
129.25
129.04
128.98
128.66
128.46
128.29
126.63
126.60
115.98
115.77
115.64
115.42
70.99
70.21
F
F
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

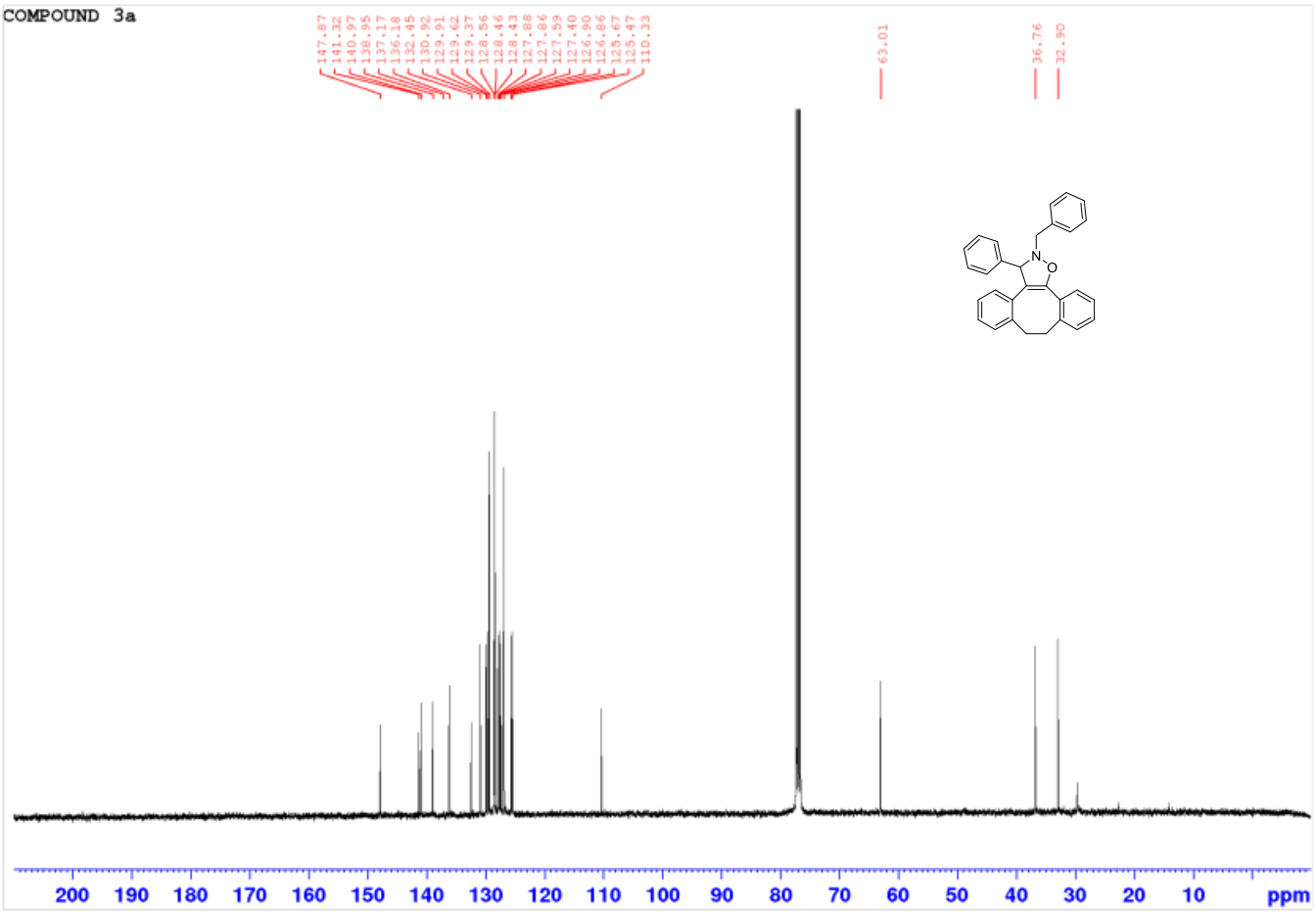

COMPOUND 2h AND COMPOUND 2h'
8.216
8.206
8.194
8.182
7.485
7.480
7.465
7.462
7.417
7.406
7.395
7.389
7.381
7.334
7.312
6.943
6.922
6.899
5.034
4.996
3.832
3.819
OMe
OMe
10
9
8
7
6
5
4
3
2
1
0
ppm
3.91
12.36
4.09
4.00
6.05

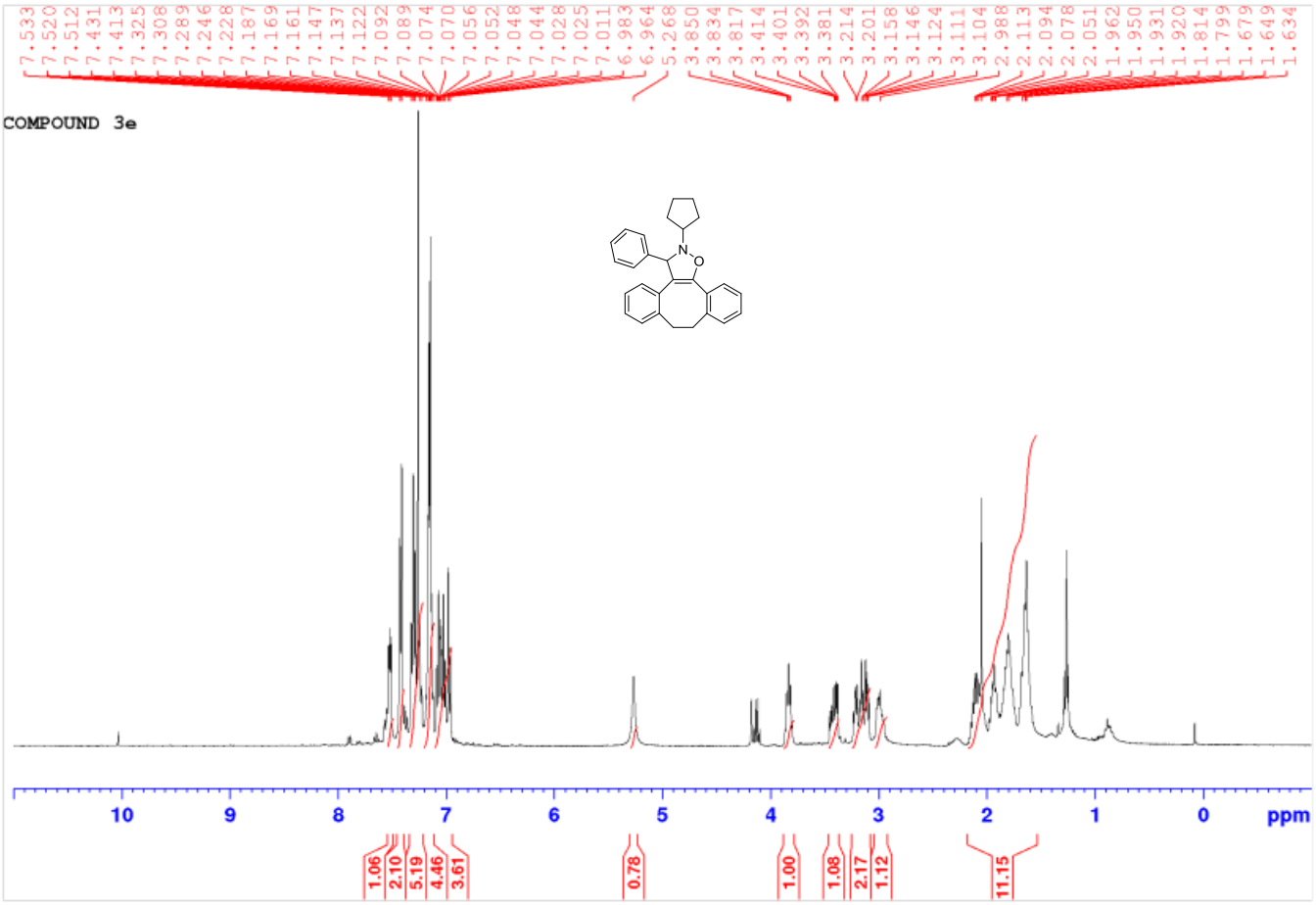

COMPOUND 2h AND COMPOUND 2h'
161.04
160.09
133.94
133.30
130.66
130.57
130.34
129.17
128.89
128.83
128.55
128.36
125.11
123.30
114.30
113.73
70.63
70.58
55.28
OMe
OMe
220
200
180
160
140
120
100
80
60
40
20
ppm

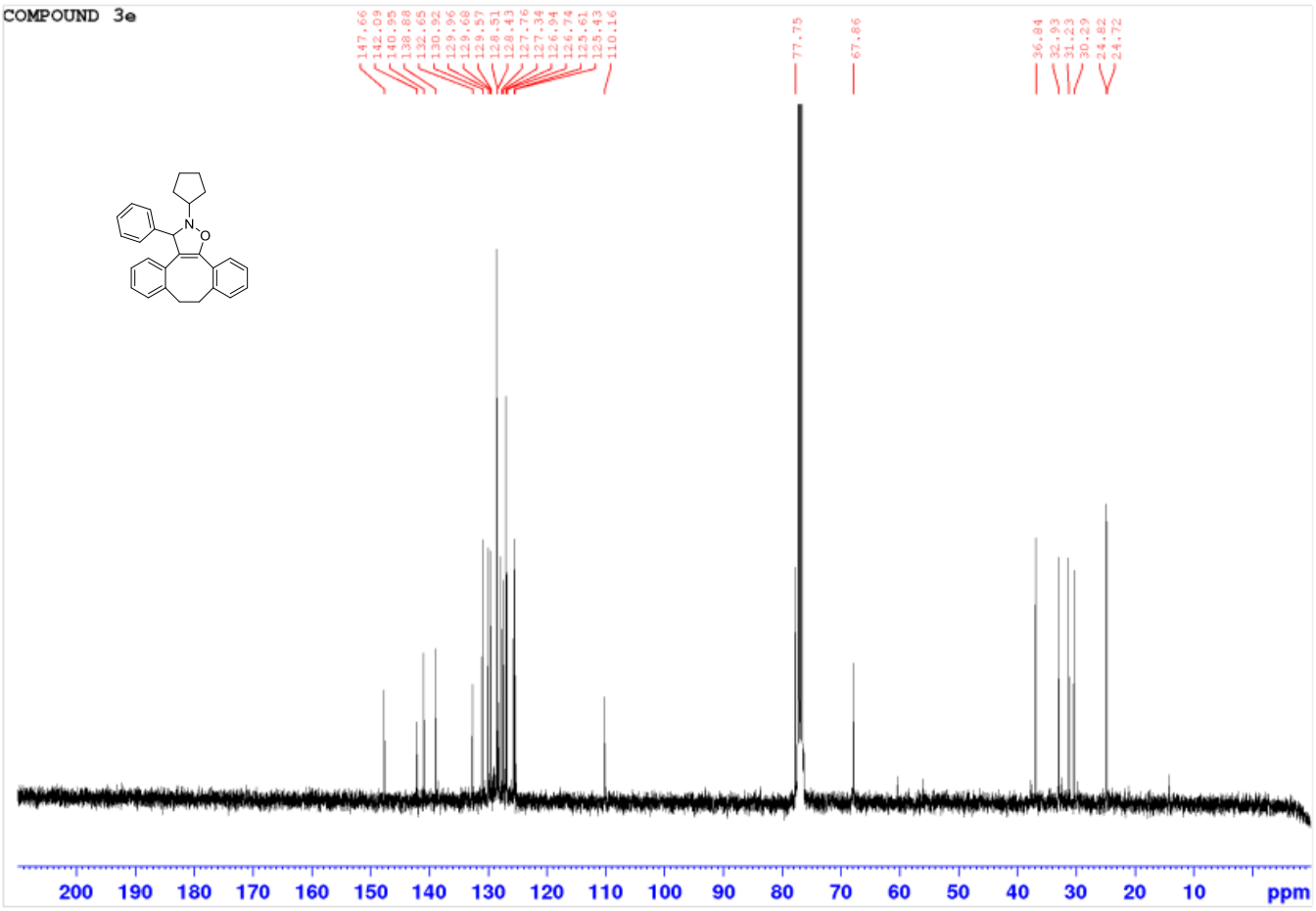

COMPOUND 2i
7.980
7.960
7.487
7.469
7.449
7.426
7.408
7.390
7.299
7.285
7.250
7.237
7.078
7.060
5.081
5.043
4.624
4.586
4.176
4.167
4.150
4.143
4.139
4.129
4.115
4.096
3.798
3.784
3.759
3.742
3.716
3.707
3.690
3.675
3.664
2.874
2.861
2.851
2.831
2.820
2.811
2.791
10
9
8
7
6
5
4
3
2
1
0
ppm
1.91
2.94
1.17
1.00
1.04
1.10
2.17
1.06


COMPOUND 2i
129.15
128.99
128.57
127.52
126.76
126.23
120.51
71.30
65.58
26.26
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.527
7.510
7.497
7.492
7.390
7.373
7.354
7.330
7.312
7.296
7.290
7.285
7.250
7.245
7.228
7.177
7.169
7.157
7.153
7.148
7.137
7.124
7.101
7.086
7.083
7.068
7.064
7.048
7.030
7.014
6.975
6.956
5.239
4.646
4.614
4.326
4.294
3.386
3.373
3.363
3.351
3.219
3.199
3.186
3.158
3.145
3.117
3.105
3.083
3.069
3.063
3.050
2.968
2.956
2.946
2.933
2.920
2.911
COMPOUND 3a
N
O
10
9
8
7
6
5
4
3
2
1
0
ppm
2.89
18.75
1.00
0.99
0.99
1.11
2.15
1.26


COMPOUND 3a
147.87
141.32
140.97
138.95
137.17
136.18
132.45
130.92
129.91
129.62
129.37
128.56
128.46
128.43
127.88
127.86
127.59
127.40
126.90
126.86
125.67
125.47
110.33
63.01
36.76
32.90
N
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.533
7.520
7.512
7.431
7.413
7.325
7.308
7.289
7.246
7.228
7.187
7.169
7.161
7.147
7.137
7.122
7.092
7.089
7.074
7.070
7.056
7.052
7.048
7.044
7.028
7.025
7.011
6.983
6.964
5.268
3.850
3.834
3.817
3.414
3.401
3.392
3.381
3.214
3.201
3.158
3.146
3.124
3.111
3.104
2.988
2.113
2.094
2.078
2.051
1.962
1.950
1.931
1.920
1.814
1.799
1.679
1.649
1.634
COMPOUND 3e
10
9
8
7
6
5
4
3
2
1
0
ppm
1.06
2.10
5.19
4.46
3.61
0.78
1.00
1.08
2.17
1.12
11.15


COMPOUND 3e
147.66
142.09
140.95
138.88
132.65
130.92
129.96
129.68
129.57
128.51
128.43
127.76
127.34
126.94
126.74
125.61
125.43
110.16
77.75
67.86
36.84
32.93
31.23
30.29
24.82
24.72
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm