Electronic Supplementary Information

for

The syntheses of α-ketoamides via $^{n}Bu_4NI$-catalyzed multiple sp^3 C–H bonds oxidation of ethylarenes and sequential coupling with dialkylformamides

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1. General considerations

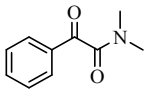
All reactions were run in a sealed tube with a Teflon lined cap under air atmosphere. All reagents were commercially available and were used without purification. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded on a Bruker Avance 400 spectrometers in $CDCl_3$ [using $(CH_3)_4Si$ (for ^{1}H, δ = 0.00; for ^{13}C, δ = 77.00) as internal standard]. The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. High-resolution mass spectra were obtained with a Waters Q-TOF mass spectrometer.

2. General experimental procedures

Ethylarene **1** (1.0 mmol), $^{n}Bu_4NI$ (0.2 mmol) and TBHP (3 mmol) were added in a 25 mL sealed tube with a Teflon lined cap. The mixture was stirred in an oil bath at 80 ^{o}C. Then dialkylformamide **2** (6.0 mmol) and TBHP (9 mmol) were added in batches. After 18 h, the reaction mixture was cooled to room temperature, and diluted with water, then extracted with ethyl acetate (15 mL × 3). The combined organic layer was washed with water and dried with anhydrous Na_2SO_4, the solvent was then removed under vacuum. The residue was purified by a flash column chromatography on silica gel using hexane/ethyl acetate as eluent to give the corresponding product.

3. Characterization data for all products

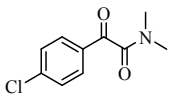
***N,N*-dimethyl-2-oxo-2-phenylacetamide**



3aa:[1] Yellow oil

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97-7.95 (m, 2H), 7.67-7.63 (m, 1H), 7.54-7.50 (m, 2H), 3.13 (s, 3H), 2.98 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.78, 167.05, 134.71, 133.08, 129.65, 129.01, 37.04, 34.00.

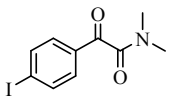
2-(4-chlorophenyl)-*N,N*-dimethyl-2-oxoacetamide



3ba:[1] Yellow oil

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91-7.89 (m, 2H), 7.50-7.48 (m, 2H), 3.12 (s, 3H), 2.97 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.31, 166.48, 141.32, 131.51, 131.03, 129.39, 37.05, 34.09.

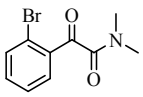
2-(4-iodophenyl)-*N,N*-dimethyl-2-oxoacetamide



3ca:[2] Yellow solid

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90-7.87 (m, 2H), 7.66-7.63 (m, 2H), 3.11 (s, 3H), 2.95 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.86, 166.42, 138.38, 132.40, 130.82, 103.31, 37.05, 34.09.

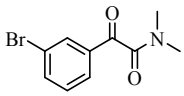
2-(2-bromophenyl)-*N,N*-dimethyl-2-oxoacetamide



3da:[1] Yellow oil

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85-7.83 (m, 1H), 7.67-7.64 (m, 1H), 7.48-7.40 (m, 2H), 3.11 (s, 3H), 3.09 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.88, 166.34, 135.46, 134.14, 134.09, 132.67, 127.77, 121.56, 37.27, 34.69.

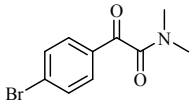
2-(3-bromophenyl)-*N,N*-dimethyl-2-oxoacetamide



3ea:[1] Yellow solid

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.08 (s, 1H), 7.87 (d, J = 7.6 Hz, 1H), 7.76 (d, J = 7.6 Hz, 1H), 7.49 (t, J = 7.6 Hz, 1H), 3.12 (s, 3H), 2.97 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.09, 166.20, 137.51, 134.87, 132.34, 130.58, 128.29, 123.24, 37.05, 34.12.

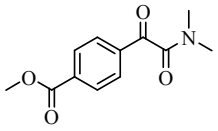
2-(4-bromophenyl)-*N,N*-dimethyl-2-oxoacetamide



3fa:[1] Yellow oil

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84-7.81 (m, 2H), 7.68-7.65 (m, 2H), 3.12 (s, 3H), 2.97 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.51, 166.44, 132.39, 131.90, 131.06, 130.20, 37.05, 34.10.

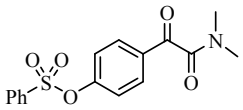
methyl 4-(aminoformyl-*N*,*N*-dimethylform)benzoate



3ga: Yellow solid

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.15-8.12 (m, 2H), 8.00-7.98 (m, 2H), 3.94 (s, 3H), 3.11 (s, 3H), 2.96 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.84, 166.38, 165.89, 136.19, 135.16, 130.06, 129.53, 52.56, 37.00, 34.08; HRMS (ESI): calcd. for $C_{12}H_{14}NO_4$(MH^+) 236.0923, found 236.0936.

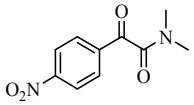
4-(2-(dimethylamino)-2-oxoacetyl)phenyl benzenesulfonate



3ha: Yellow oil

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94-7.89 (m, 2H), 7.88-7.84 (m, 2H), 7.73-7.66 (m, 1H), 7.59-7.52 (m, 2H), 7.17-7.13 (m, 2H), 3.11(s, 3H), 2.96 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.94, 166.36, 153.95, 134.67, 131.75, 131.55, 129.40, 128.46, 126.65, 122.90, 37.07, 34.12; HRMS (ESI): calcd. for $C_{16}H_{16}NO_5S$ (MH^+) 334.0749, found 334.0770.

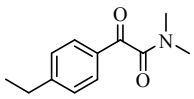
***N*,*N*-dimethyl-2-(4-nitrophenyl)-2-oxoacetamide**



3ia:[1] Yellow solid

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.37-8.34 (m, 2H), 8.17-8.14 (m, 2H), 3.16 (s, 3H), 3.02 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.25, 165.59, 151.08, 137.56, 130.79, 124.08, 37.08, 34.31.

2-(4-ethylphenyl)-*N*,*N*-dimethyl-2-oxoacetamide

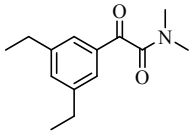


3ja: Yellow oil

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89-7.86 (m, 2H), 7.34 (d, *J* = 8.4 Hz, 2H), 3.13 (s, 3H), 2.91 (s,

3H), 2.74 (q, J = 7.6 Hz, 2H), 1.28 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.55, 167.28, 152.07, 130.89, 129.90, 128.56, 37.07, 33.97, 29.16, 15.08; HRMS (ESI): calcd. for $C_{12}H_{15}NO_2Na$ (MNa^+) 228.1000, found 228.1016.

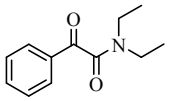
2-(3,5-diethylphenyl)-*N,N*-dimethyl-2-oxoacetamide



3ka: Yellow oil

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59 (s, 2H), 7.33 (s, 1H), 3.13 (s, 3H), 2.97 (s, 3H), 2.69 (q, J = 7.6 Hz, 4H), 1.26 (t, J = 7.6 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 192.31, 167.36, 145.24, 134.27, 133.27, 126.48, 37.09, 33.98, 28.62, 15.44; HRMS (ESI): calcd. for $C_{14}H_{19}NO_2Na$ (MNa^+) 256.1313, found 256.1326.

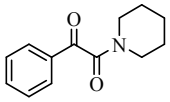
***N,N*-diethyl-2-oxo-2-phenylacetamide**



3ab:[1] Yellow oil

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97-7.95 (m, 2H), 7.67-7.63 (m, 1H), 7.54-7.50 (m, 2H), 3.59 (q, J = 7.2 Hz, 2H), 3.26 (q, J = 7.2 Hz, 2H), 1.31 (t, J = 7.2 Hz, 3H), 1.18 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.58, 166.74, 134.55, 133.30, 129.63, 128.95, 42.11, 38.80, 14.11, 12.84.

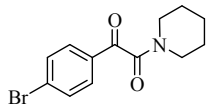
1-phenyl-2-(piperidin-1-yl)ethane-1,2-dione



3ac:[1] Yellow oil

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95 (dd, J = 8.0, 2.4 Hz, 2H), 7.66-7.61 (m, 1H), 7.53-7.49 (m, 2H), 3.70-3.68 (m, 2H), 3.29 (t, J = 5.6 Hz, 2H), 1.70-1.68 (m, 4H), 1.56-1.53 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.95, 165.44, 134.64, 133.27, 129.54, 128.99, 47.02, 42.13, 26.19, 25.44, 24.36.

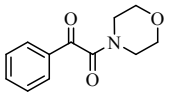
1-(4-bromophenyl)-2-(piperidin-1-yl)ethane-1,2-dione



3fc:[3] Yellow oil

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (dd, *J* = 6.8, 2.0 Hz, 2H), 7.67 (dd, *J* = 6.8, 2.0 Hz, 2H), 3.71 (d, 2H), 3.29 (t, *J* = 5.2 Hz, 2H), 1.71-1.70 (m, 4H), 1.57-1.55 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.74, 164.88, 132.40, 132.07, 130.97, 130.12, 47.06, 42.26, 26.26, 25.45, 24.35.

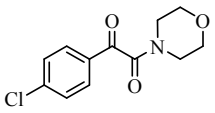
1-morpholino-2-phenylethane-1,2-dione



3ad:[1] Yellow oil

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 (dd, *J* = 8.0, 2.4 Hz, 2H), 7.70-7.65 (m, 1H), 7.56-7.52 (m, 2H), 3.81 (s, 4H), 3.68-3.66 (m, 2H), 3.41-3.39 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.16, 165.46, 134.94, 133.07, 129.68, 129.10, 66.74, 66.67, 46.27, 41.63.

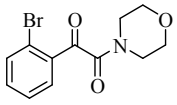
1-(4-chlorophenyl)-2-morpholinoethane-1,2-dione



3bd:[4] Yellow oil

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91 (dd, *J* = 8.0, 2.0 Hz, 2H), 7.51 (dd, *J* = 8.0, 2.0 Hz, 2H), 3.82-3.77 (m, 4H), 3.68-3.66 (m, 2H), 3.40-3.38 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.69, 164.90, 141.60, 131.48, 131.04, 129.50, 66.74, 66.64, 46.29, 41.71.

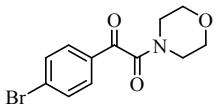
1-(2-bromophenyl)-2-morpholinoethane-1,2-dione



3dd:[5] Yellow solid

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (dd, *J* = 7.6, 2.0 Hz, 1H), 7.76 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.50-7.42 (m, 2H), 3.84-3.79 (m, 4H), 3.77-3.74 (m, 2H), 3.60-3.58 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.43, 164.84, 135.47, 134.29, 134.04, 132.68, 127.66,121.48,66.30, 66.25, 46.30, 42.05.

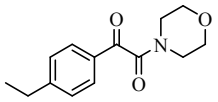
1-(4-bromophenyl)-2-morpholinoethane-1,2-dione



3fd:[4] Yellow oil

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (dd, *J* = 8.0, 2.0 Hz, 2H), 7.68 (dd, *J* = 8.0, 2.0 Hz, 2H), 3.82-3.78 (m, 4H), 3.68-3.66 (m, 2H), 3.40-3.38 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.89, 164.87, 132.50, 131.87, 131.06, 130.50, 66.73, 66.64, 46.29, 41.73.

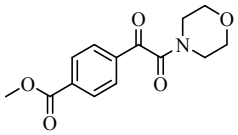
1-(4-ethylphenyl)-2-morpholinoethane-1,2-dione



3jd:[4] Yellow oil

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, *J* = 8.4 Hz, 2H), 7.36 (d, *J* = 8.4 Hz, 2H), 3.82-3.78 (m, 4H), 3.68-3.65 (m, 2H), 3.41-3.38 (m, 2H), 2.75 (q, *J* = 7.6 Hz, 2H), 1.28 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.91, 165.70, 152.38, 130.86, 129.92, 128.66, 66.77, 66.69, 46.28, 41.58, 29.18, 15.05.

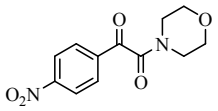
methyl 4-(2-morpholino-2-oxoacetyl)benzoate



3gd: Yellow oil

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.19 (d, *J* = 8.4 Hz, 2H), 8.05 (d, *J* = 8.4 Hz, 2H), 3.98 (s, 3H), 3.83 (s, 4H), 3.70-3.68 (m, 2H), 3.43-3.40 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.25, 165.85, 164.82, 136.17, 135.40, 130.17, 129.60, 66.73, 66.66, 52.64, 46.30, 41.77; HRMS (ESI): calcd. for $C_{14}H_{16}NO_5$ (MH^+) 278.1028, found 278.1049.

1-morpholino-2-(4-nitrophenyl)ethane-1,2-dione

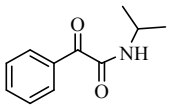


3id:[4] Yellow oil

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.37 (dd, *J* = 7.2, 2.0 Hz, 2H), 8.18 (dd, *J* = 7.2, 2.0 Hz, 2H), 3.84-3.82 (m, 4H), 3.73-3.70 (m, 2H), 3.46-3.43 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 188.67,

164.03, 151.20, 137.49, 130.84, 124.16, 66.74, 66.63, 46.35, 41.95.

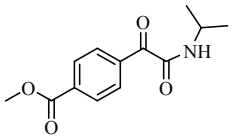
***N*-isopropyl-2-oxo-2-phenylacetamide**



3ae:[6] Yellow oil

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.36-8.33 (m, 2H), 7.65-7.61 (m, 1H), 7.50-7.46 (m, 2H), 6.96 (s, 1H), 4.20-4.15 (m, 1H), 1.27 (d, J = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 188.05, 160.95, 134.29, 133.43, 131.19, 128.44, 41.71, 22.42.

methyl 4-(aminoformyl-*N*-isopropylform)benzoate



3ge: Yellow solid

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.40 (d, J = 6.8 Hz, 2H), 8.13 (d, J = 6.8 Hz, 2H), 6.96 (s, 1H), 4.22-4.14 (m, 1H), 3.97 (s, 3H), 1.28 (d, J = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 187.49, 166.16, 160.29, 136.70, 134.66, 131.10, 129.47, 52.51, 41.85, 22.40; HRMS (ESI): calcd. for $C_{13}H_{16}NO_4$ (MH^+) 250.1079, found 250.1093.

References

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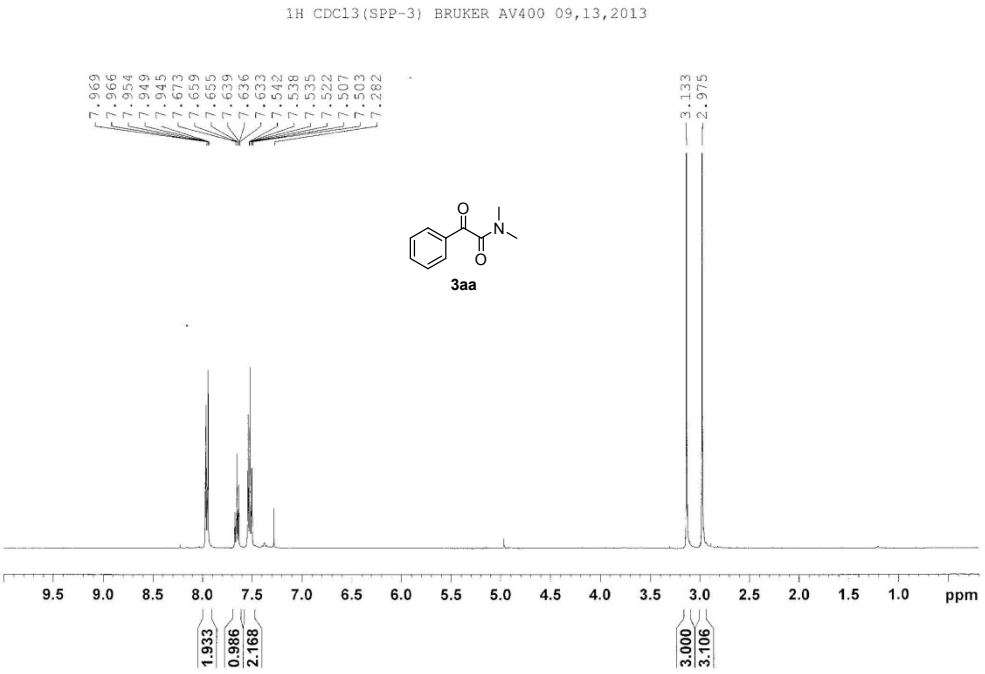
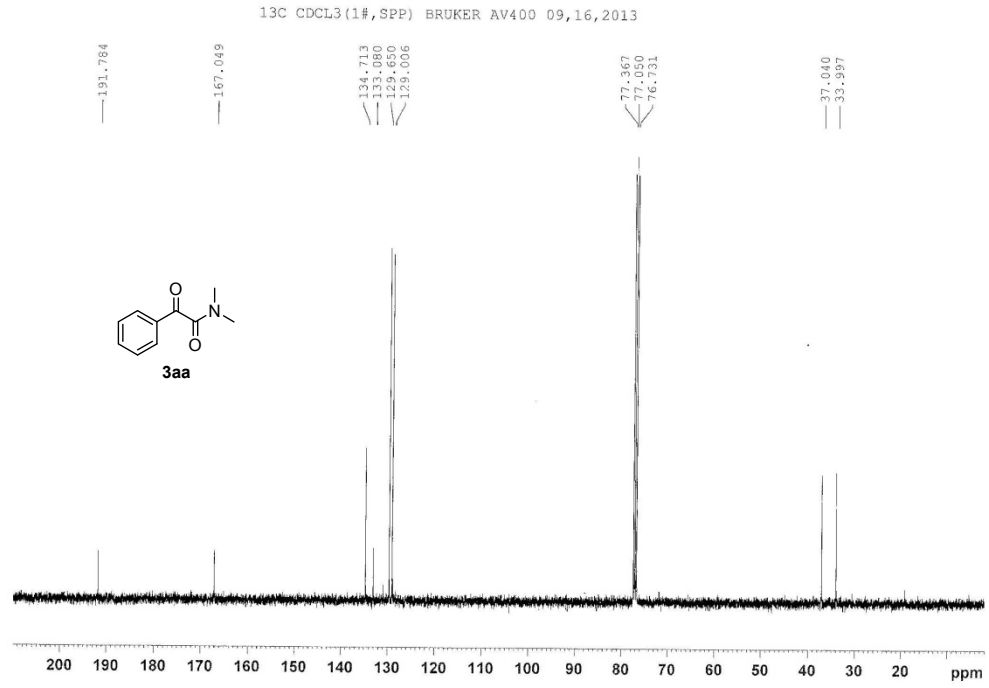
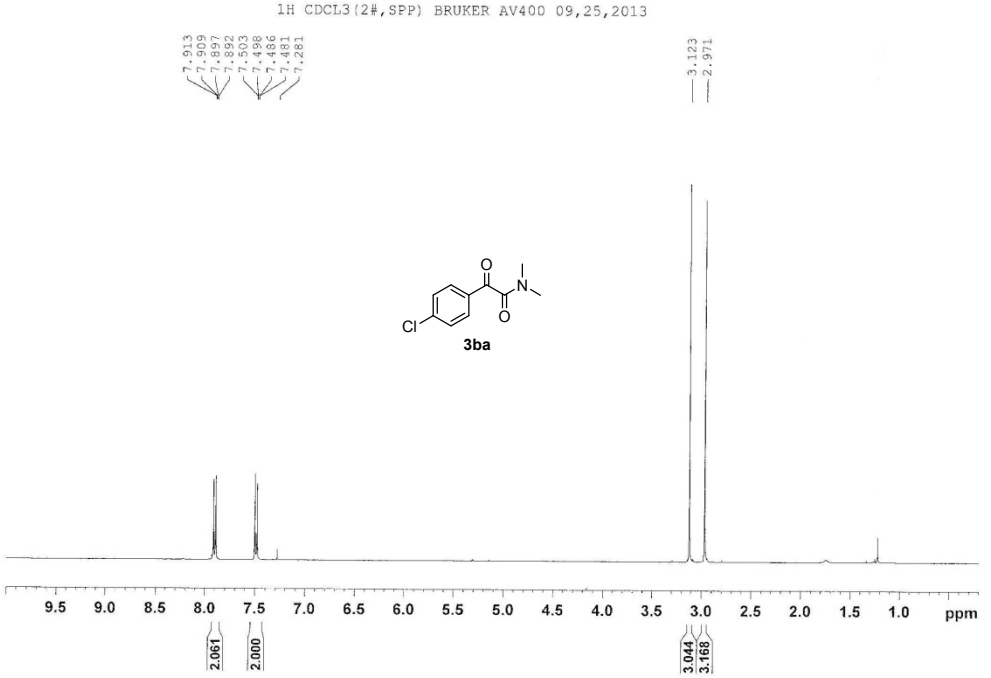
5 V. N. Lisitsyn and S. V. Komissarova, *Izv. Vyssh. Uchebn. Zaved., Khim. Khim. T.,* 1985, **28**, 37. (in Russian)

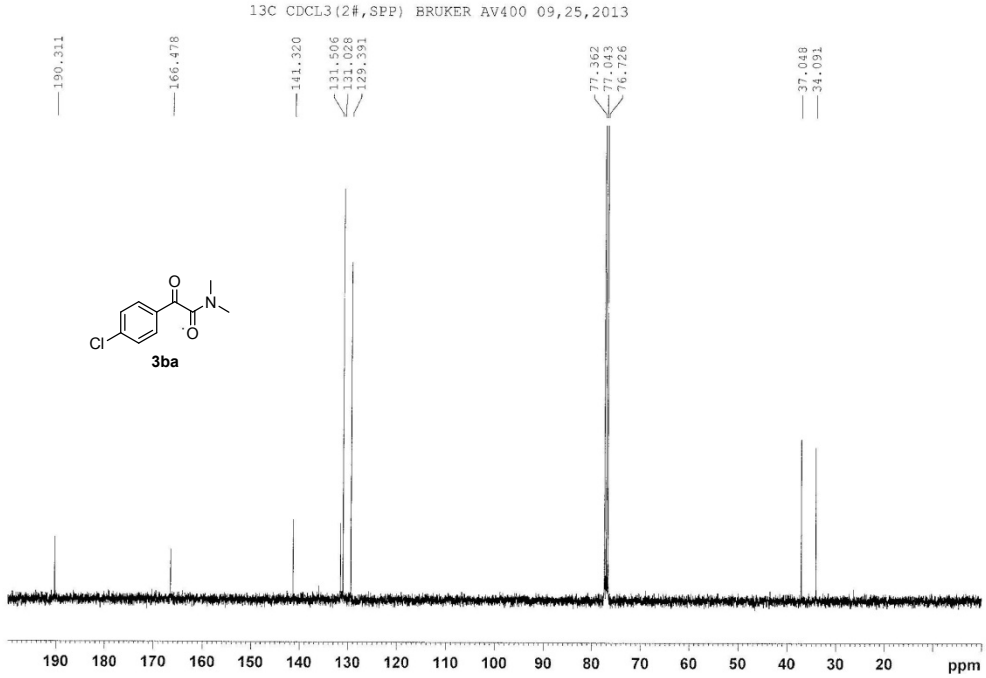
6 W. Wei, Y. Shao, H. Y. Hu, F. Zhang, C. Zhang, Y. Xu and X. B. Wan, *J. Org. Chem*., 2012, **77**, 7157.

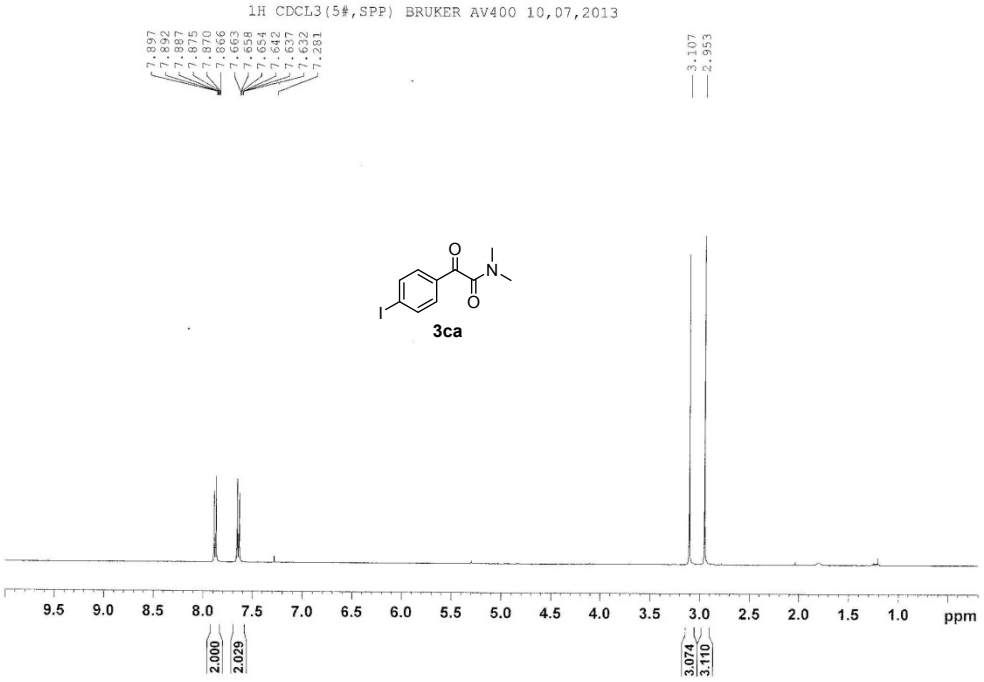
4. NMR spectra for all products

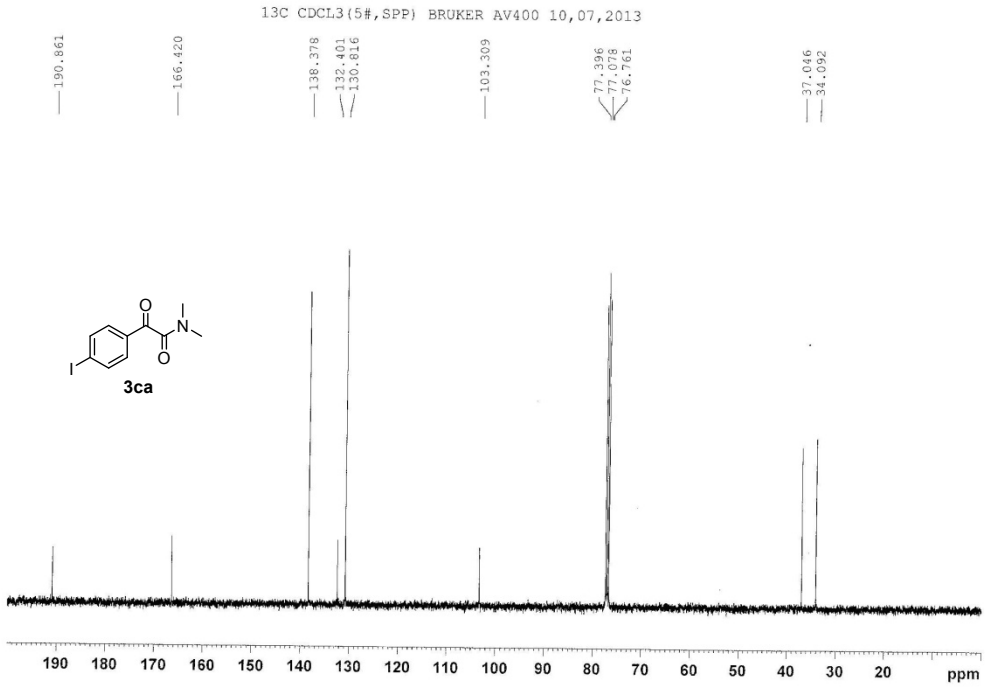

1H CDCl3(SPP-3) BRUKER AV400 09,13,2013
7.969
7.966
7.954
7.949
7.945
7.673
7.659
7.655
7.639
7.636
7.633
7.542
7.538
7.535
7.522
7.507
7.503
7.282
3.133
2.975
3aa
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.933
0.986
2.168
3.000
3.106

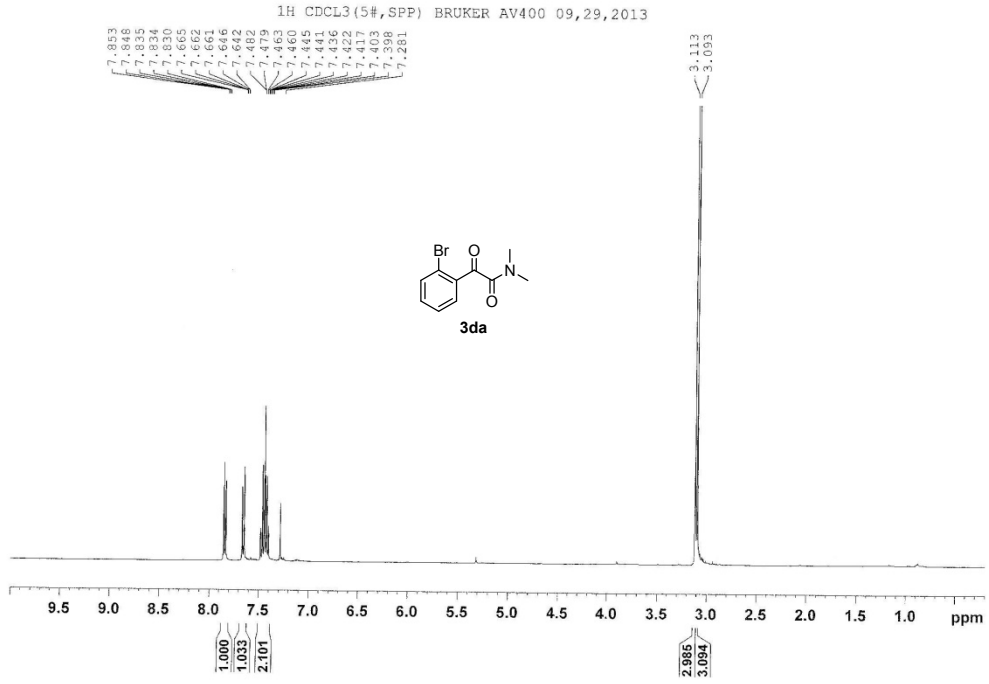

13C CDCL3(1#,SPP) BRUKER AV400 09,16,2013
191.784
167.049
134.713
133.080
129.650
129.006
77.367
77.050
76.731
37.040
33.997
3aa
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

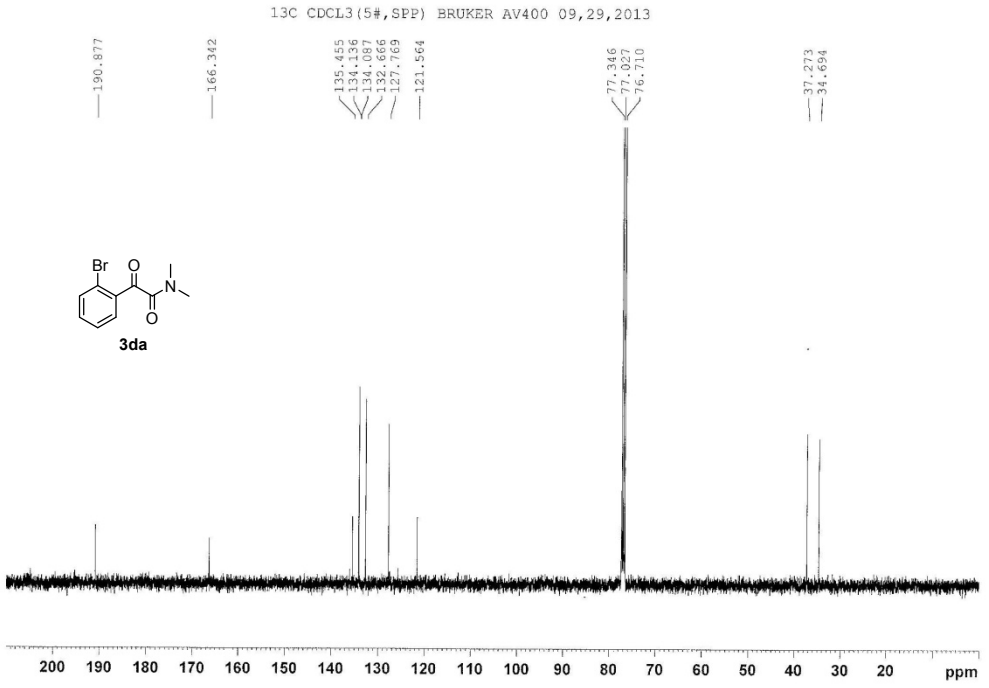

1H CDCL3(2#,SPP) BRUKER AV400 09,25,2013
7.913
7.909
7.897
7.892
7.503
7.498
7.486
7.481
7.281
3.123
2.971
3ba
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.061
2.000
3.044
3.168

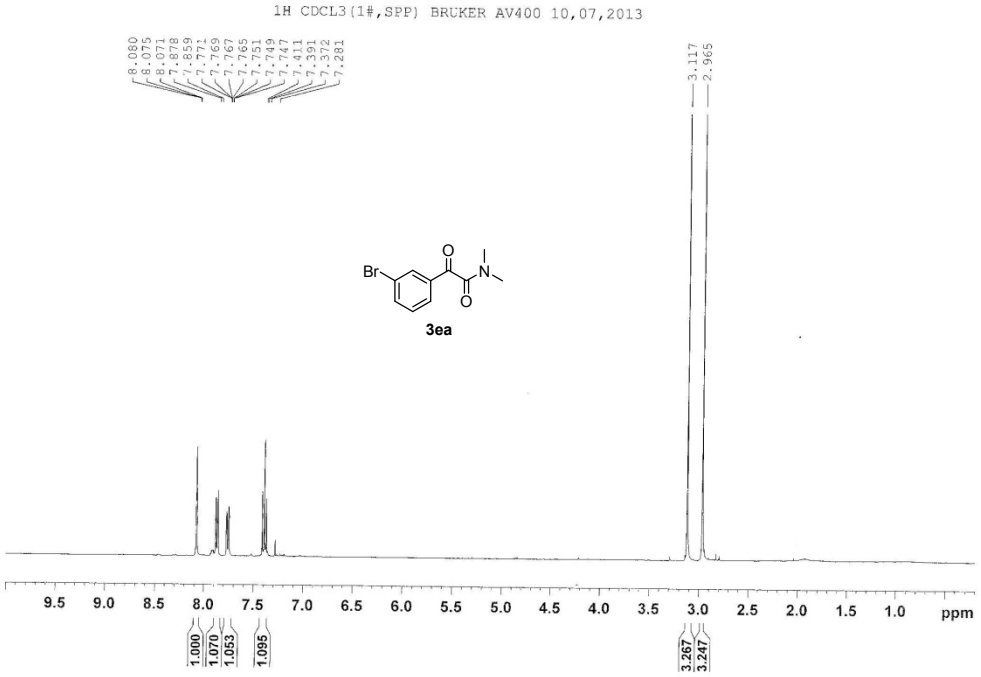

13C CDCL3(2#,SPP) BRUKER AV400 09,25,2013
190.311
166.478
141.320
131.506
131.028
129.391
77.362
77.043
76.726
37.048
34.091
3ba
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

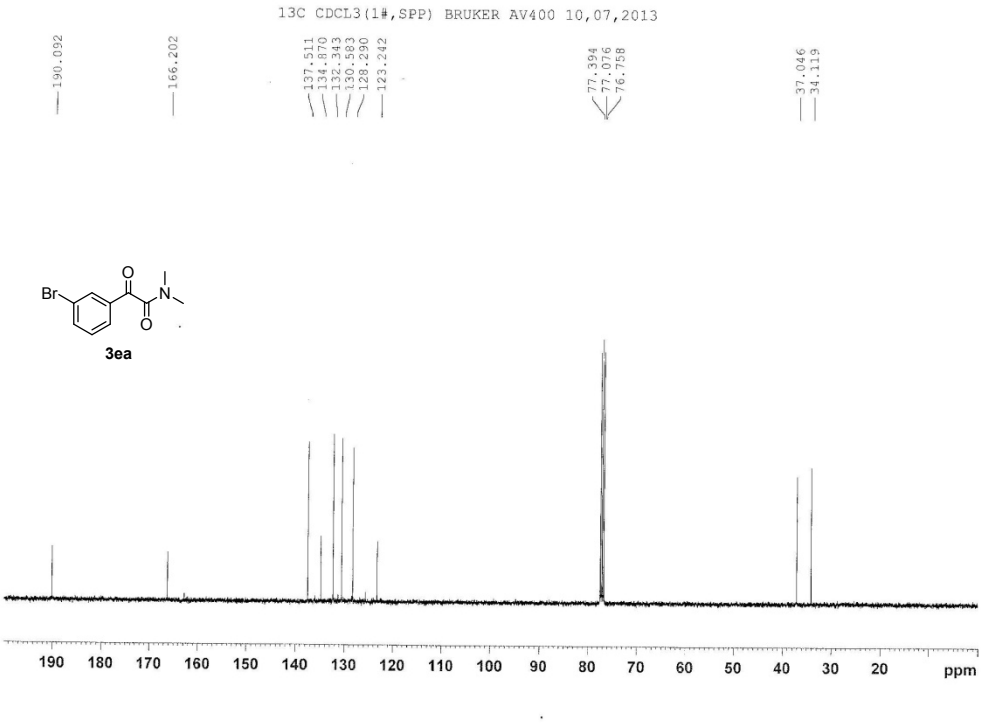

1H CDCL3(5#,SPP) BRUKER AV400 10,07,2013
7.897
7.892
7.887
7.875
7.870
7.666
7.663
7.658
7.654
7.642
7.637
7.632
7.281
3.107
2.953
3ca
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.000
2.029
3.074
3.110

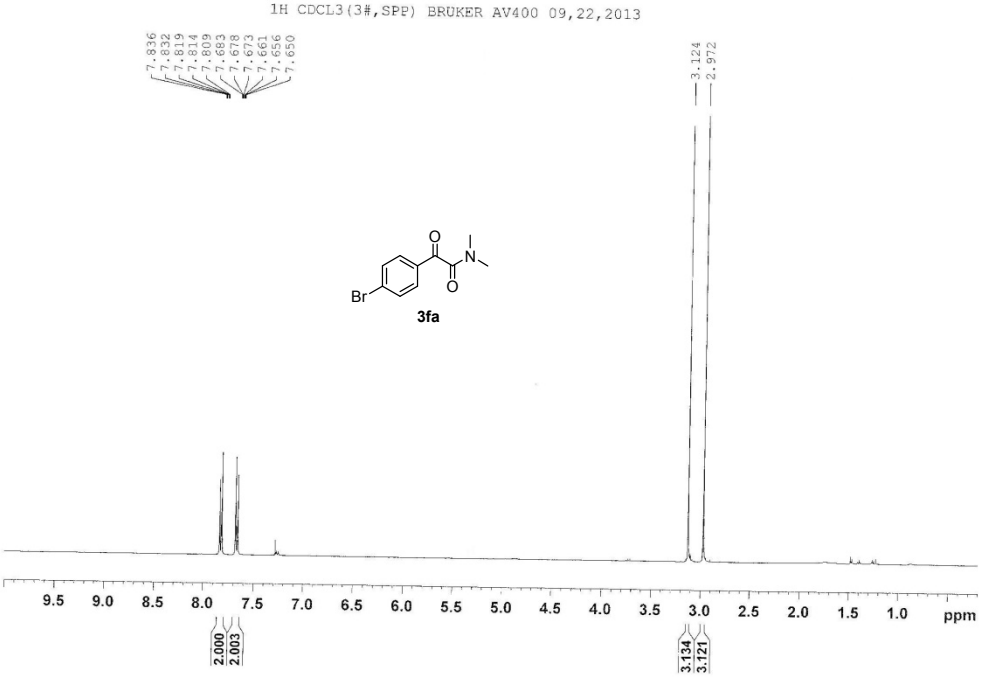

13C CDCL3(5#,SPP) BRUKER AV400 10,07,2013
190.861
166.420
138.378
132.401
130.816
103.309
77.396
77.078
76.761
37.046
34.092
3ca
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

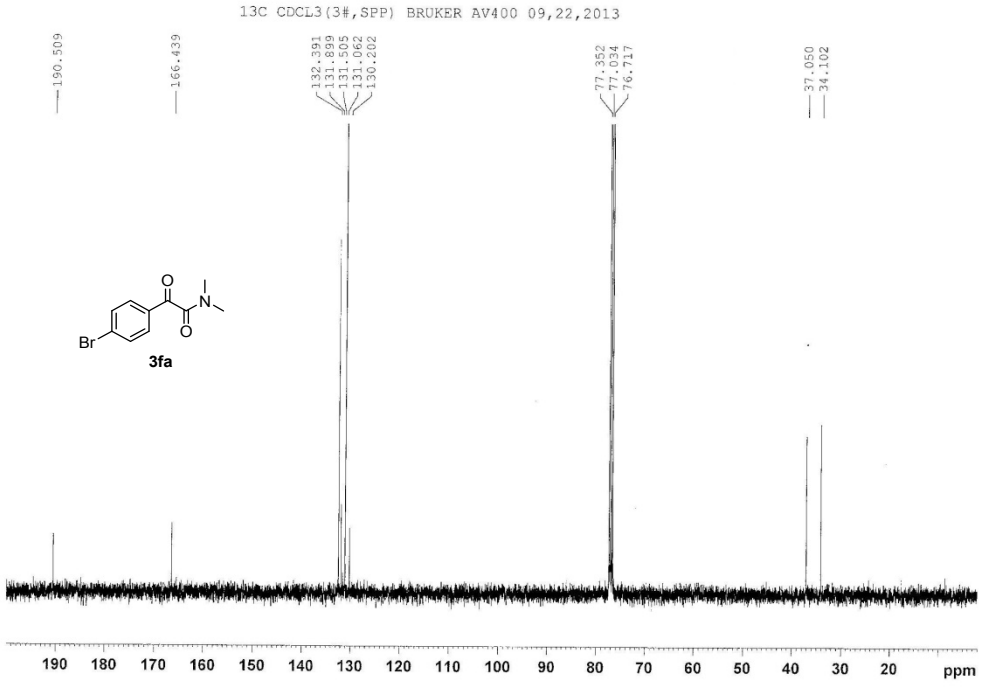

1H CDCL3(5#,SPP) BRUKER AV400 09,29,2013
7.853
7.848
7.835
7.834
7.830
7.665
7.662
7.661
7.646
7.642
7.482
7.479
7.463
7.460
7.445
7.441
7.436
7.422
7.417
7.403
7.398
7.281
3.113
3.093
Br
O
N
O
3da
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.000
1.033
2.101
2.985
3.094

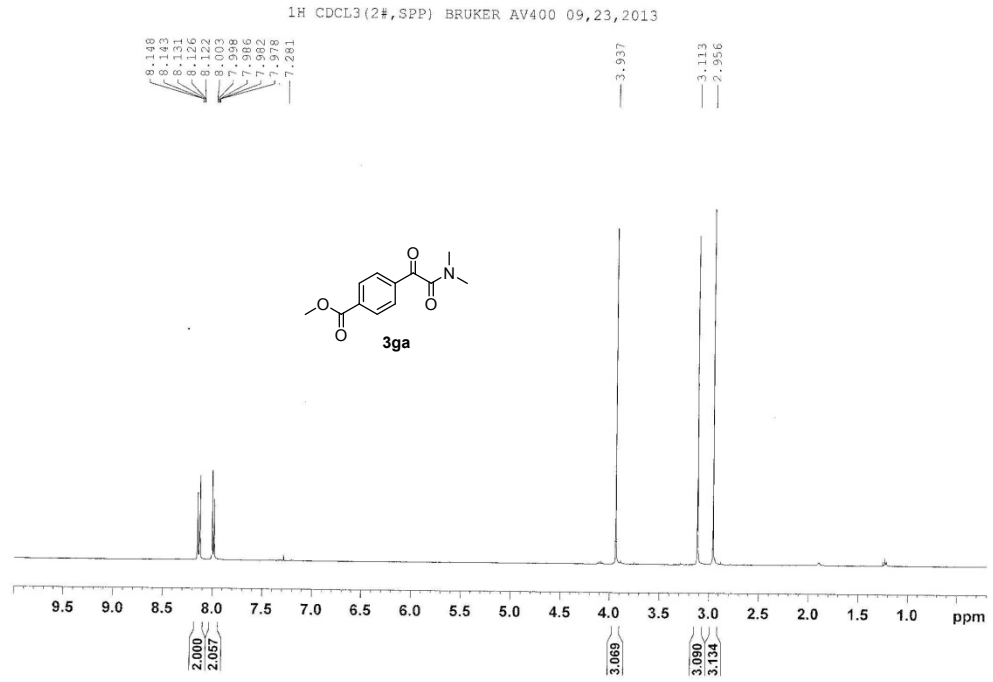

13C CDCL3(5#,SPP) BRUKER AV400 09,29,2013
190.877
166.342
135.455
134.136
134.087
132.666
127.769
121.564
77.346
77.027
76.710
37.273
34.694
Br
O
N
O
3da
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

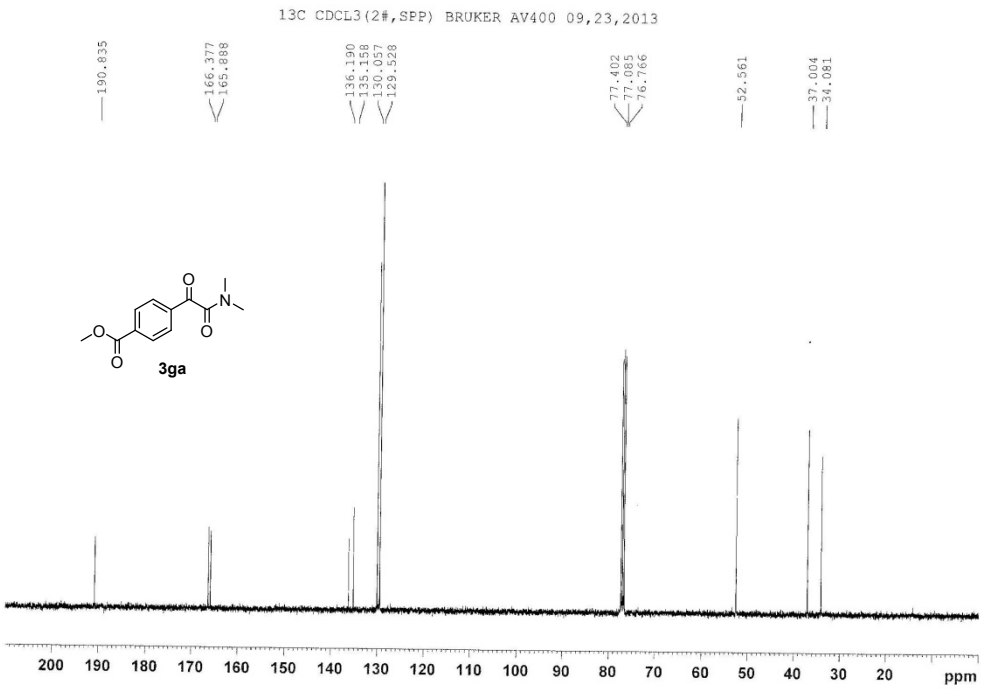

1H CDCL3(1#,SPP) BRUKER AV400 10,07,2013
8.080
8.075
8.071
7.878
7.859
7.772
7.769
7.767
7.765
7.751
7.749
7.747
7.411
7.391
7.372
7.281
3.117
2.965
Br
O
N
O
3ea
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.000
1.070
1.053
1.095
3.267
3.247

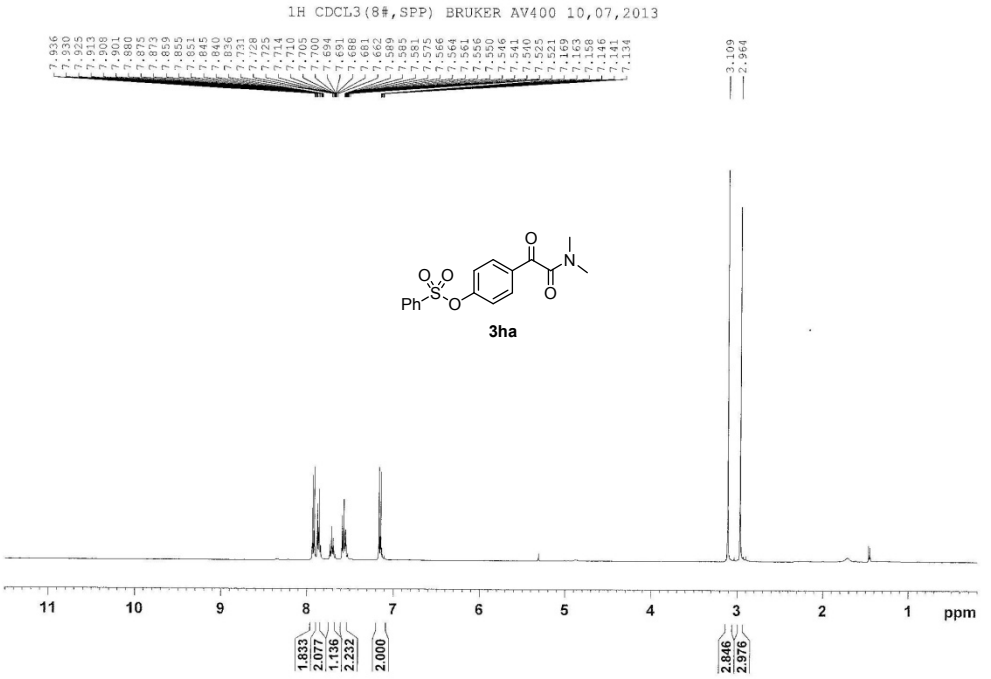

13C CDCL3(1#,SPP) BRUKER AV400 10,07,2013
190.092
166.202
137.511
134.970
134.343
130.583
128.290
123.242
77.394
77.076
76.758
37.046
34.119
Br
O
N
O
3ea
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

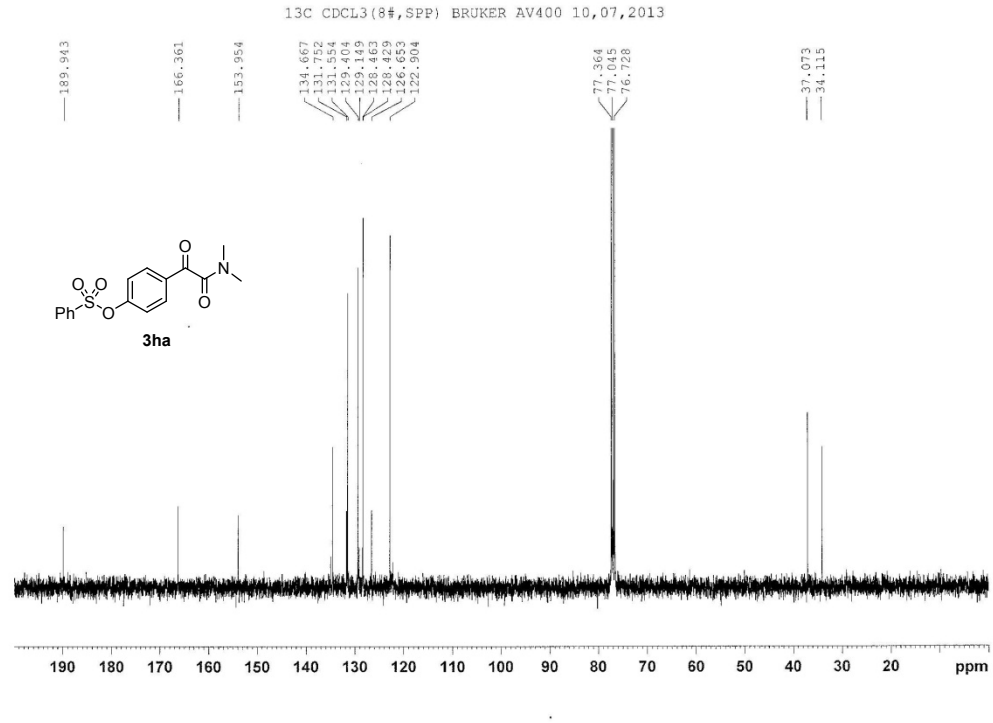

1H CDCL3(3#,SPP) BRUKER AV400 09,22,2013
7.836
7.832
7.819
7.814
7.809
7.683
7.678
7.673
7.661
7.656
7.650
3.124
2.972
Br
3fa
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.000
2.003
3.134
3.121

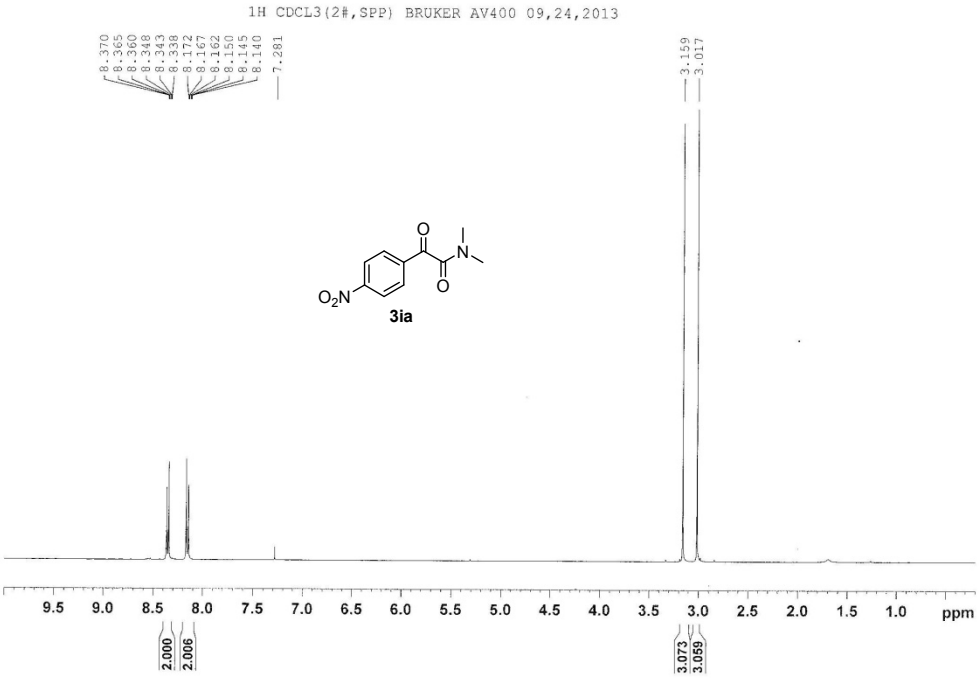

13C CDCL3(3#,SPP) BRUKER AV400 09,22,2013
190.509
166.439
132.391
131.899
131.655
131.062
130.202
77.352
77.034
76.717
37.050
34.102
Br
3fa
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

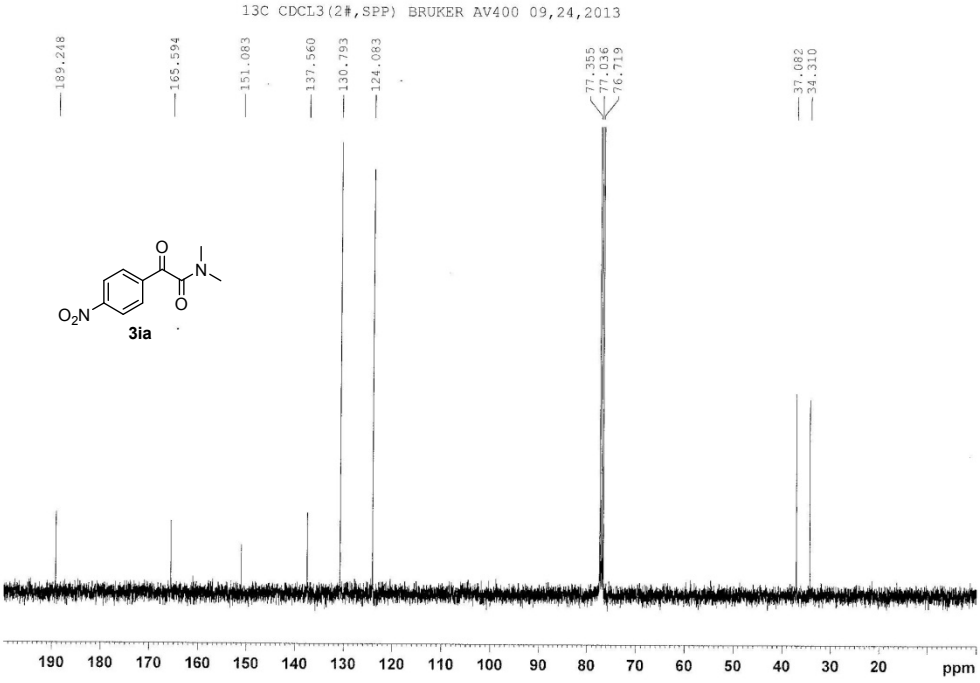

1H CDCL3(2#,SPP) BRUKER AV400 09,23,2013
8.148
8.143
8.131
8.126
8.122
8.003
7.998
7.986
7.982
7.978
7.281
3.937
3.113
2.956
3ga
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.000
2.057
3.069
3.090
3.134

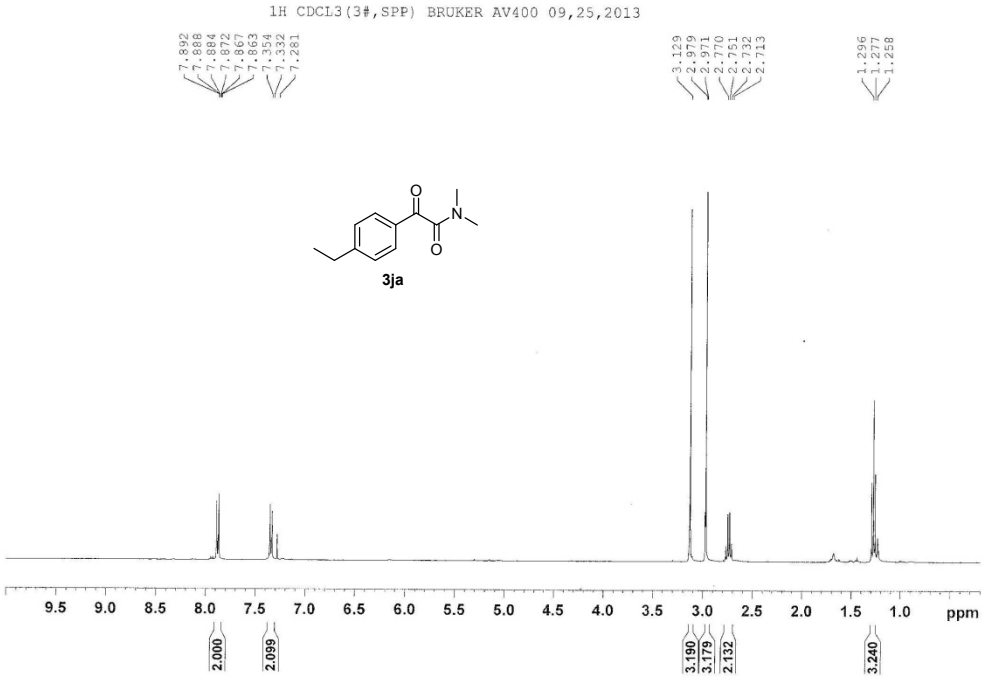

13C CDCL3(2#,SPP) BRUKER AV400 09,23,2013
190.835
166.377
165.888
136.190
135.158
130.057
129.528
77.402
77.085
76.766
52.561
37.004
34.081
3ga
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

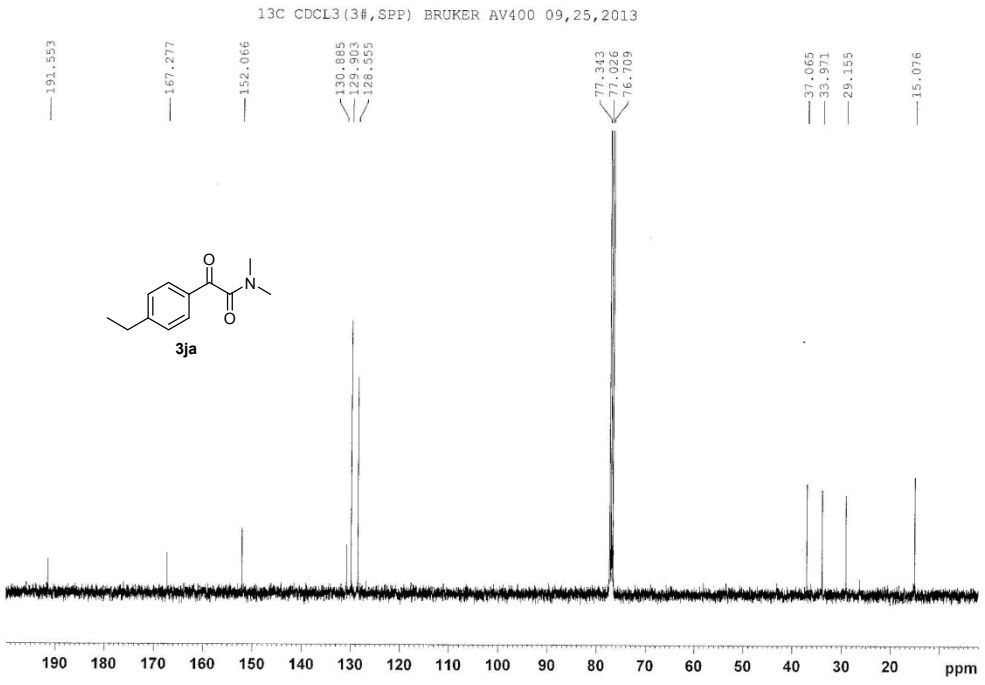

1H CDCL3(8#,SPP) BRUKER AV400 10,07,2013
3ha
11 10 9 8 7 6 5 4 3 2 1 ppm
1.833
2.077
1.136
2.232
2.000
2.846
2.976

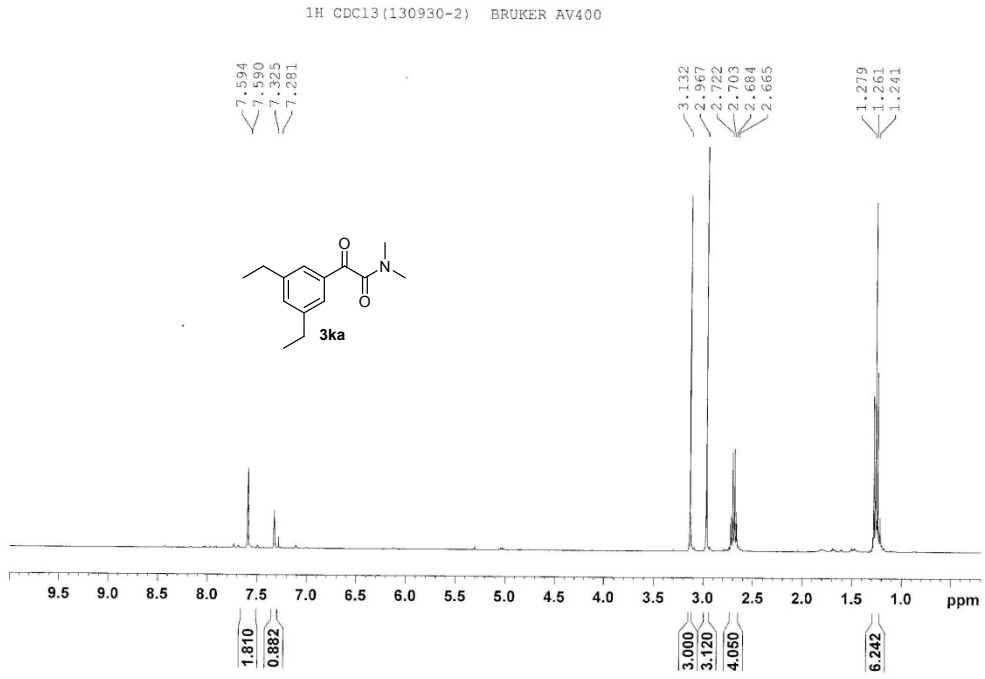

13C CDCL3(8#,SPP) BRUKER AV400 10,07,2013
189.943
166.361
153.954
134.667
131.752
131.454
129.414
128.463
128.423
126.653
122.904
77.364
77.045
76.728
37.073
34.115
3ha
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

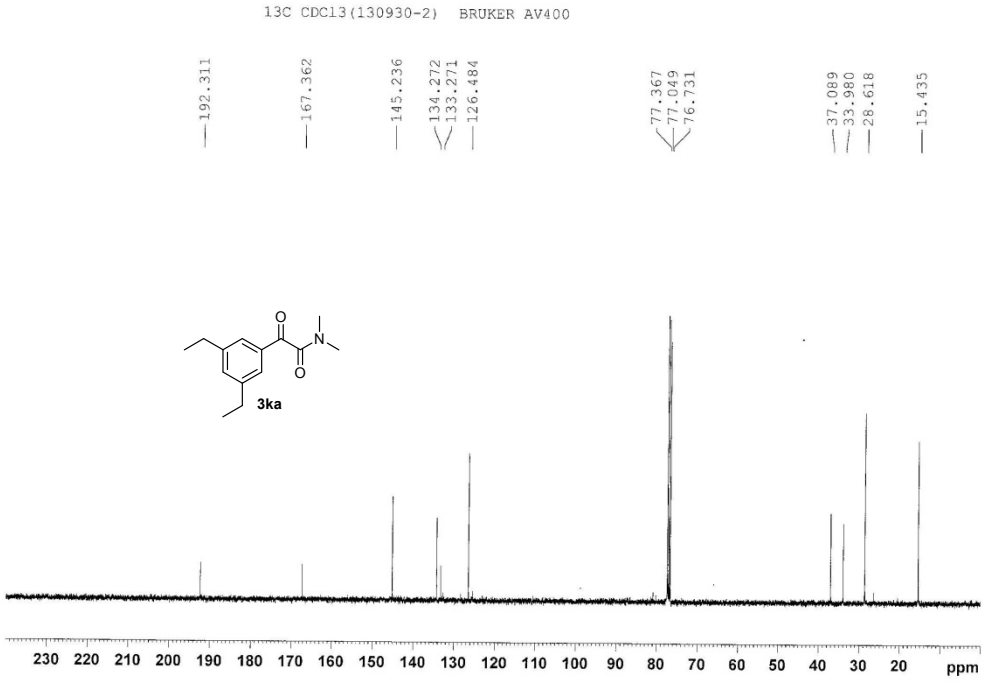

1H CDCL3(2#,SPP) BRUKER AV400 09,24,2013
8.370
8.365
8.360
8.348
8.343
8.338
8.172
8.167
8.162
8.150
8.145
8.140
7.281
3.159
3.017
3ia
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.000
2.006
3.073
3.059

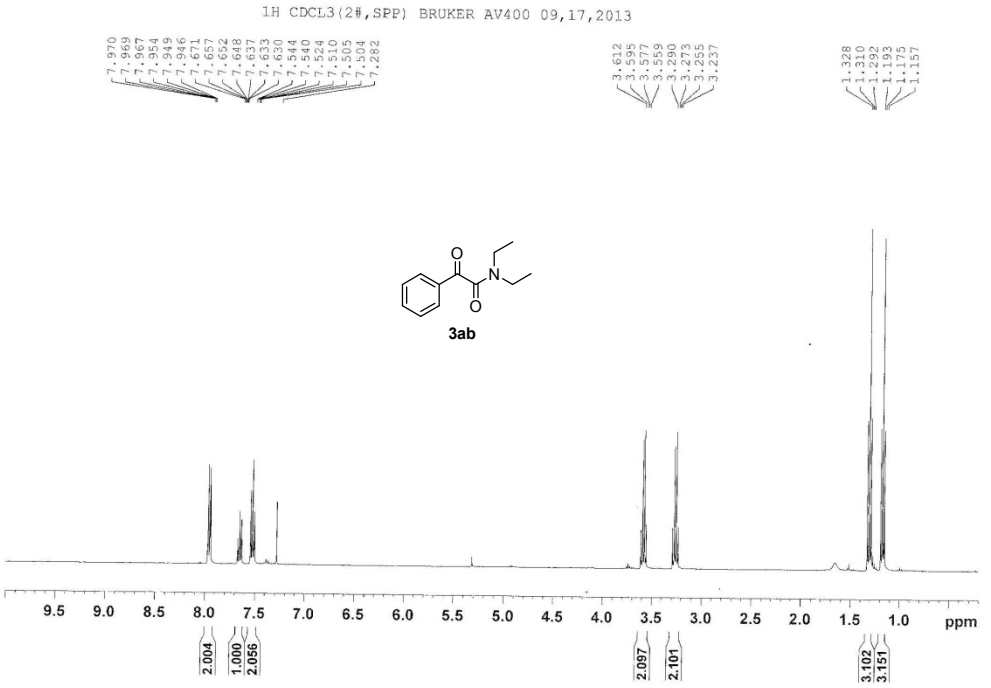

13C CDCL3(2#,SPP) BRUKER AV400 09,24,2013
189.248
165.594
151.083
137.560
130.793
124.083
77.355
77.036
76.719
37.082
34.310
3ia
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

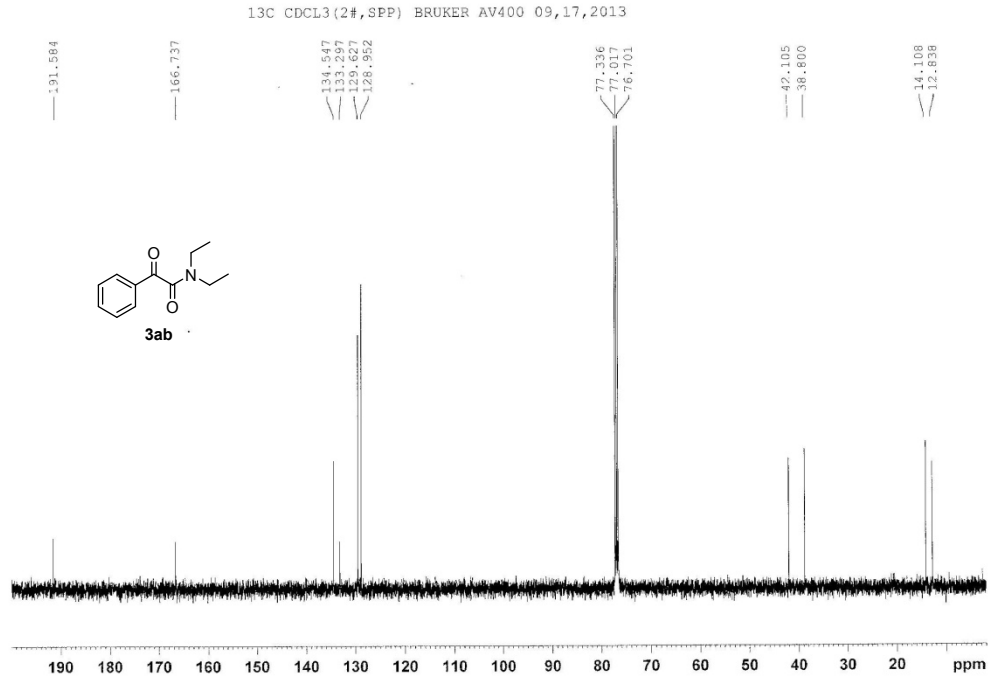

1H CDCL3(3#,SPP) BRUKER AV400 09,25,2013
7.892
7.888
7.884
7.872
7.867
7.863
7.354
7.332
7.281
3.129
2.979
2.971
2.770
2.751
2.732
2.713
1.296
1.277
1.258
3ja
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.000
2.099
3.190
3.179
2.132
3.240

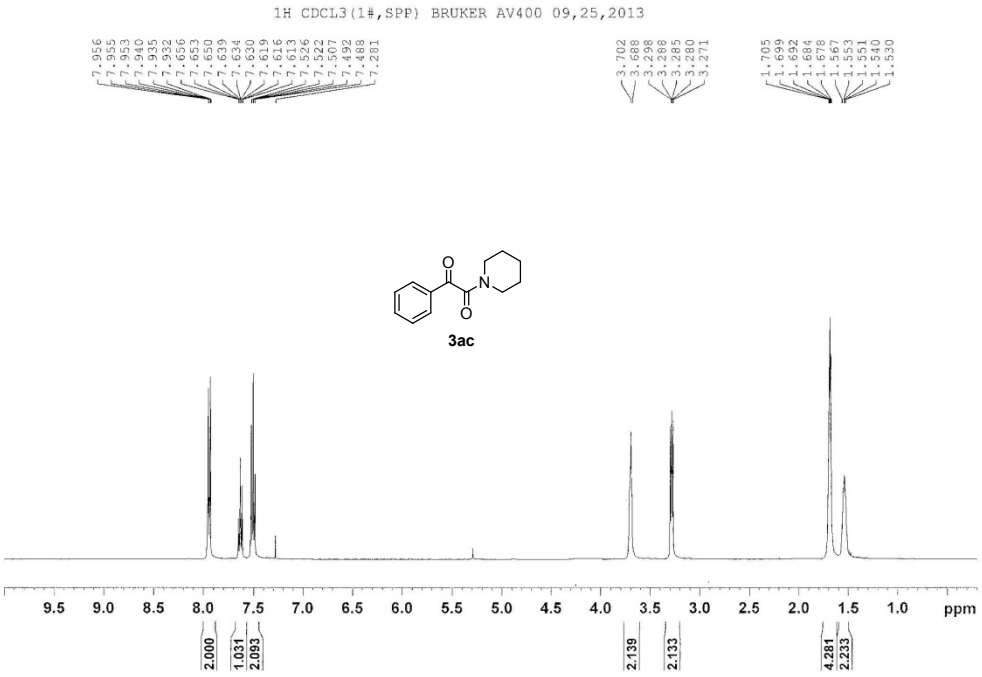

13C CDCL3(3#,SPP) BRUKER AV400 09,25,2013
192.553
167.277
152.066
130.885
129.903
128.555
77.343
77.026
76.709
37.065
33.971
29.155
15.076
3ja
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

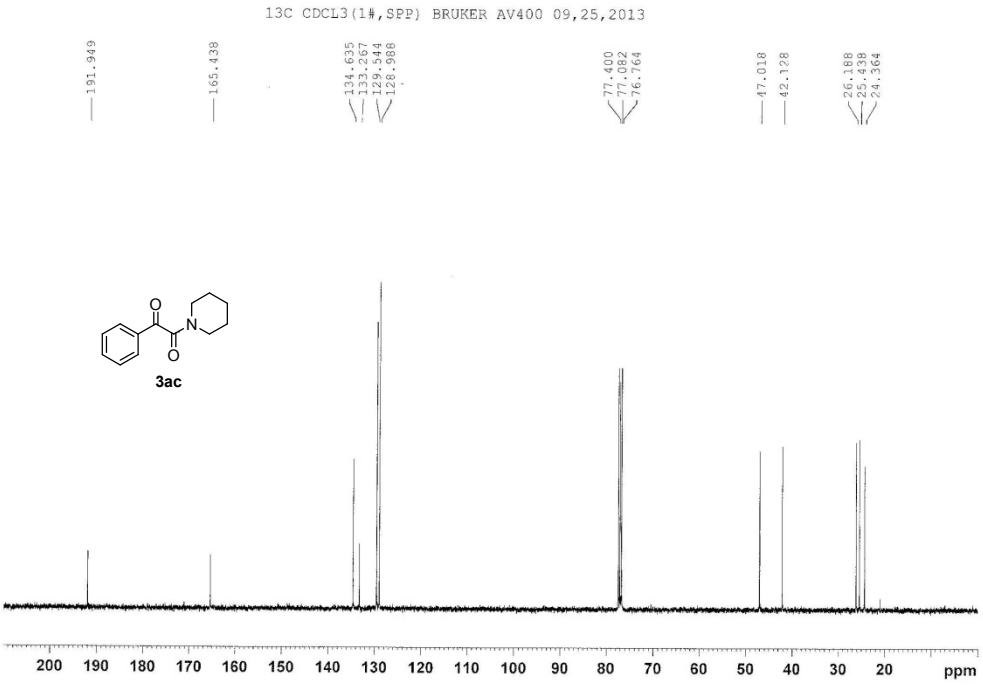

1H CDCl3(130930-2) BRUKER AV400
7.594
7.590
7.325
7.281
3.132
2.967
2.722
2.703
2.684
2.665
1.279
1.261
1.241
3ka
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.810
0.882
3.000
3.120
4.050
6.242

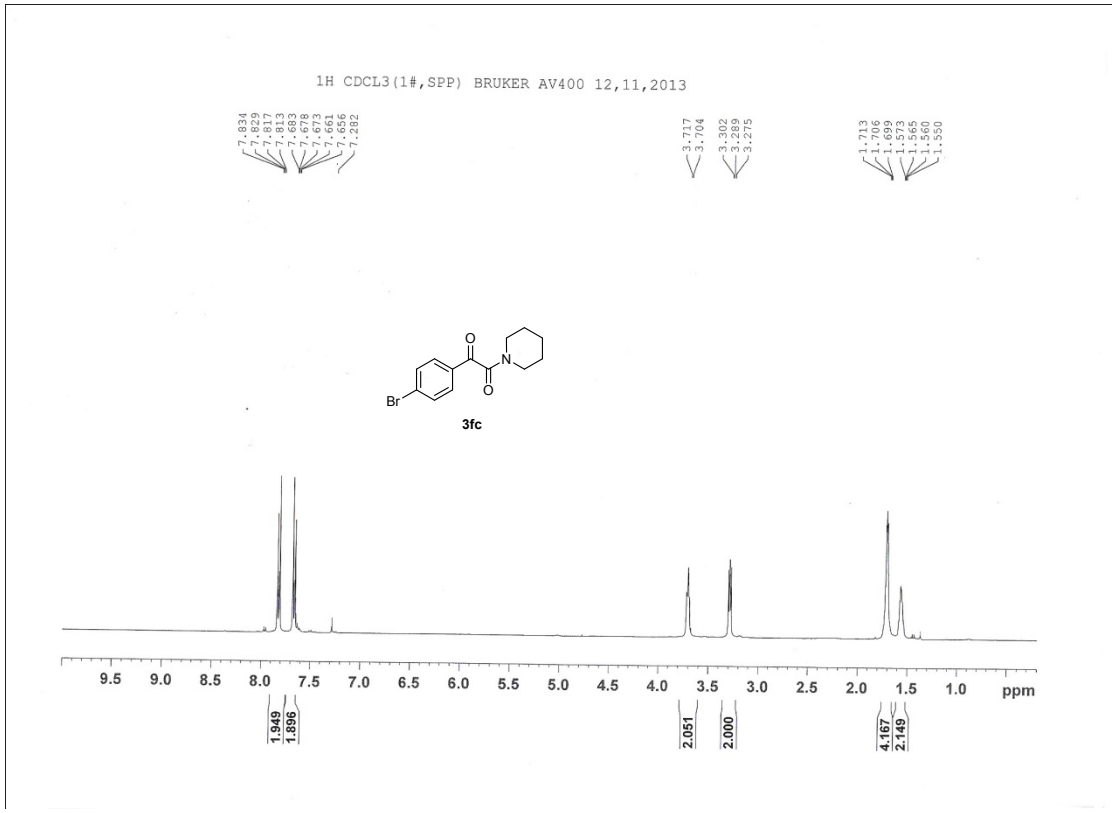

13C CDCl3(130930-2) BRUKER AV400
192.311
167.362
145.236
134.272
133.271
126.484
77.367
77.049
76.731
37.089
33.980
28.618
15.435
3ka
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

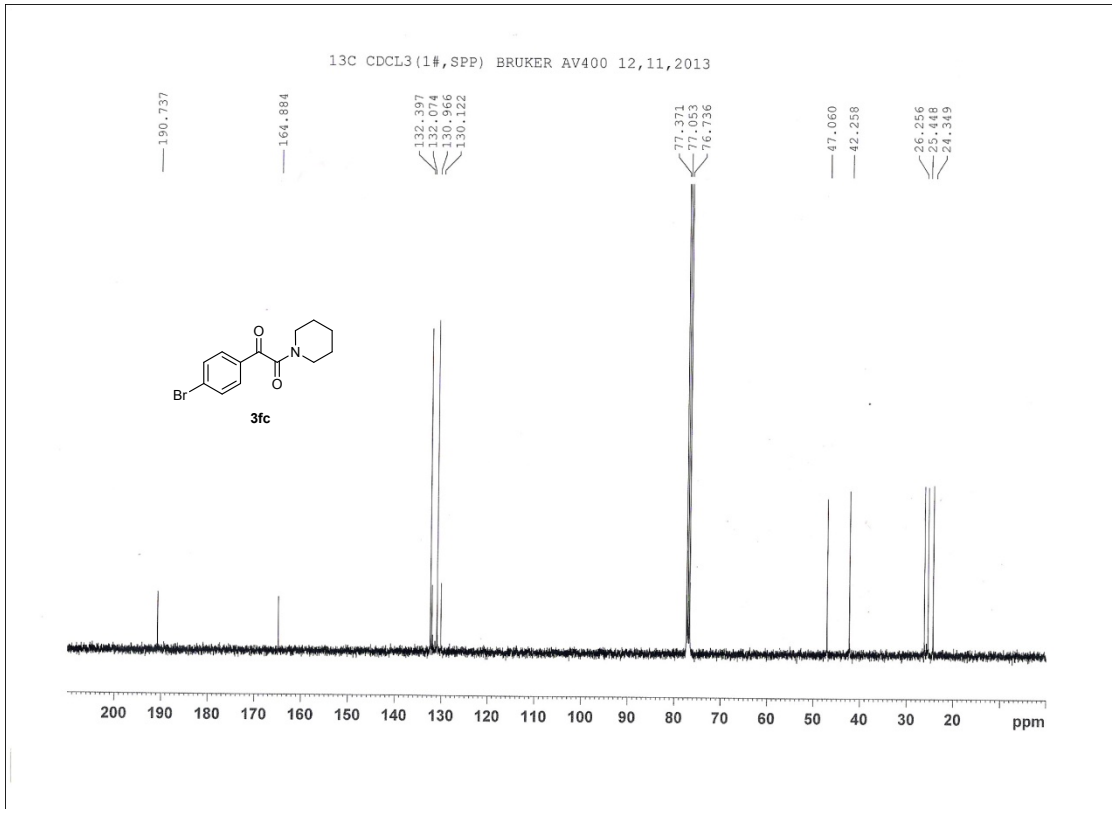

1H CDCL3(2#,SPP) BRUKER AV400 09,17,2013
7.970
7.969
7.967
7.954
7.949
7.946
7.671
7.657
7.652
7.648
7.637
7.633
7.630
7.544
7.540
7.524
7.510
7.505
7.504
7.282
3.612
3.595
3.577
3.559
3.290
3.273
3.255
3.237
1.328
1.310
1.292
1.193
1.175
1.157
3ab
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.004
1.000
2.056
2.097
2.101
3.102
3.151

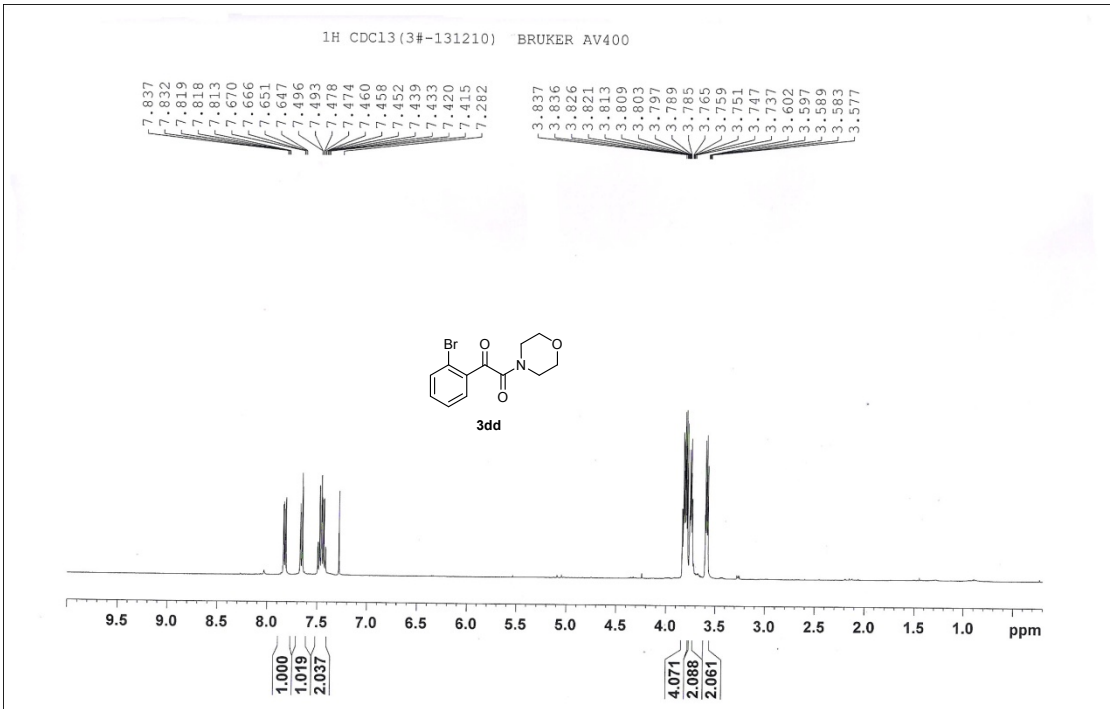

13C CDCL3(2#,SPP) BRUKER AV400 09,17,2013
191.584
166.737
134.547
133.297
129.627
128.952
77.336
77.017
76.701
42.105
38.800
14.108
12.838
3ab
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

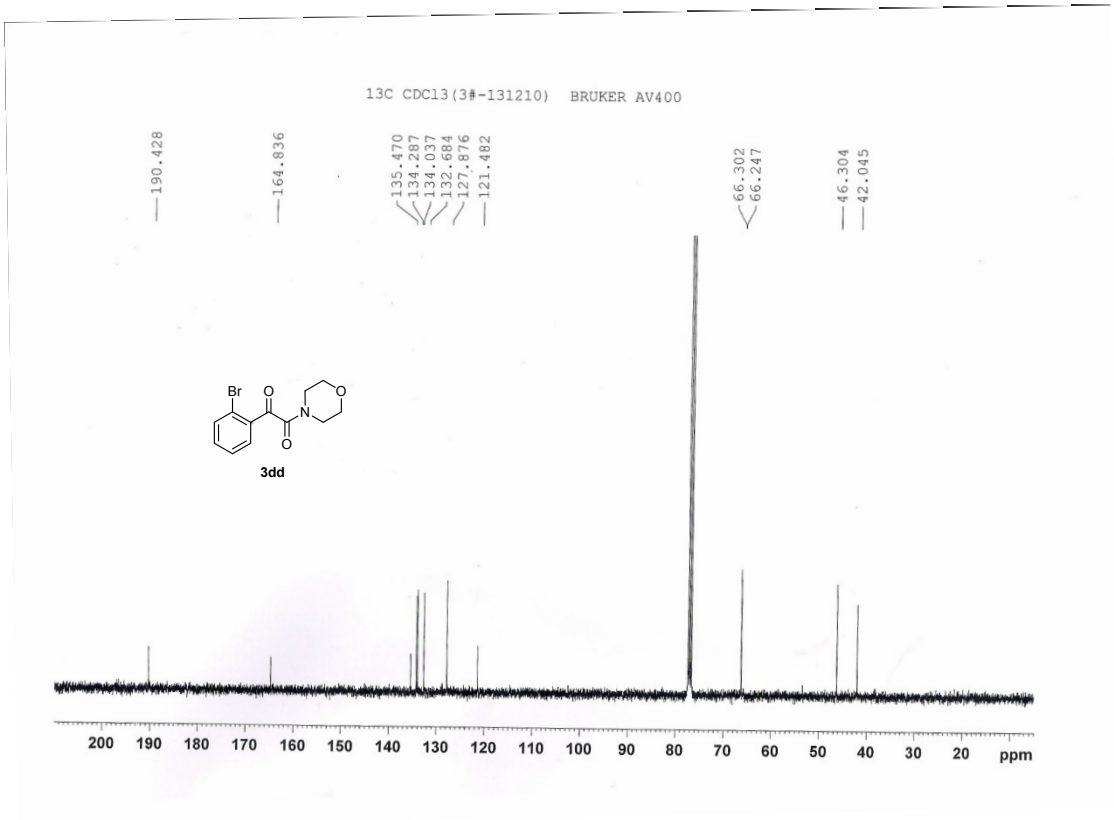

1H CDCL3(1#,SPP) BRUKER AV400 09,25,2013
7.956
7.955
7.953
7.940
7.935
7.932
7.656
7.653
7.650
7.639
7.634
7.630
7.619
7.616
7.613
7.526
7.522
7.507
7.492
7.488
7.281
3.702
3.688
3.298
3.288
3.285
3.280
3.271
1.705
1.699
1.692
1.684
1.678
1.567
1.553
1.551
1.540
1.530
3ac
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.000
1.031
2.093
2.139
2.133
4.281
2.233

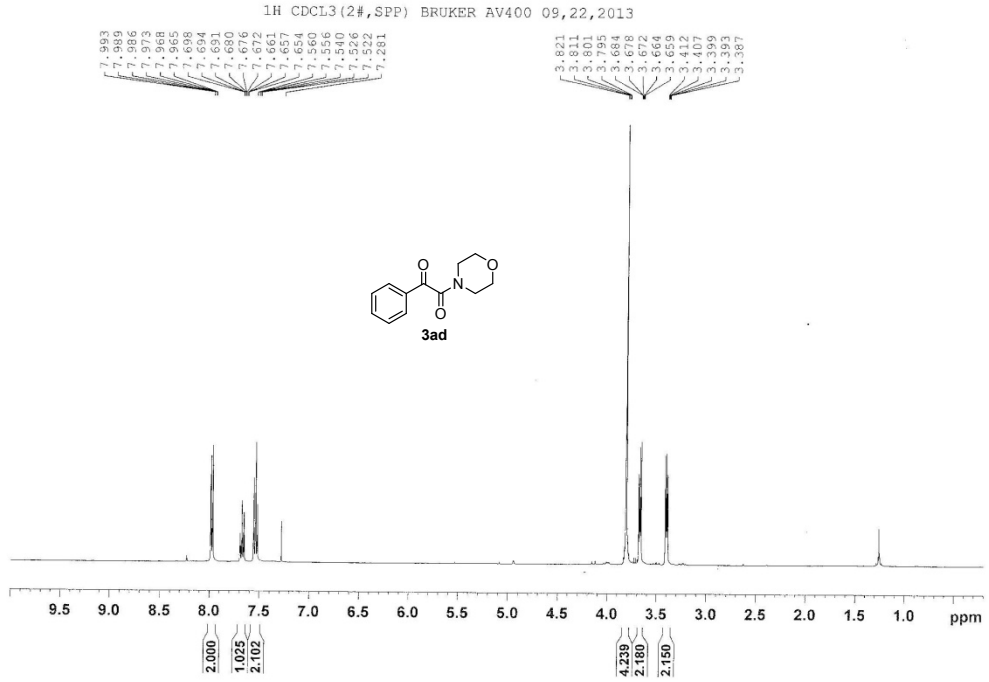

13C CDCL3(1#,SPP) BRUKER AV400 09,25,2013
191.949
165.438
134.635
133.267
129.544
128.988
77.400
77.082
76.764
47.018
42.128
26.188
25.438
24.364
3ac
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

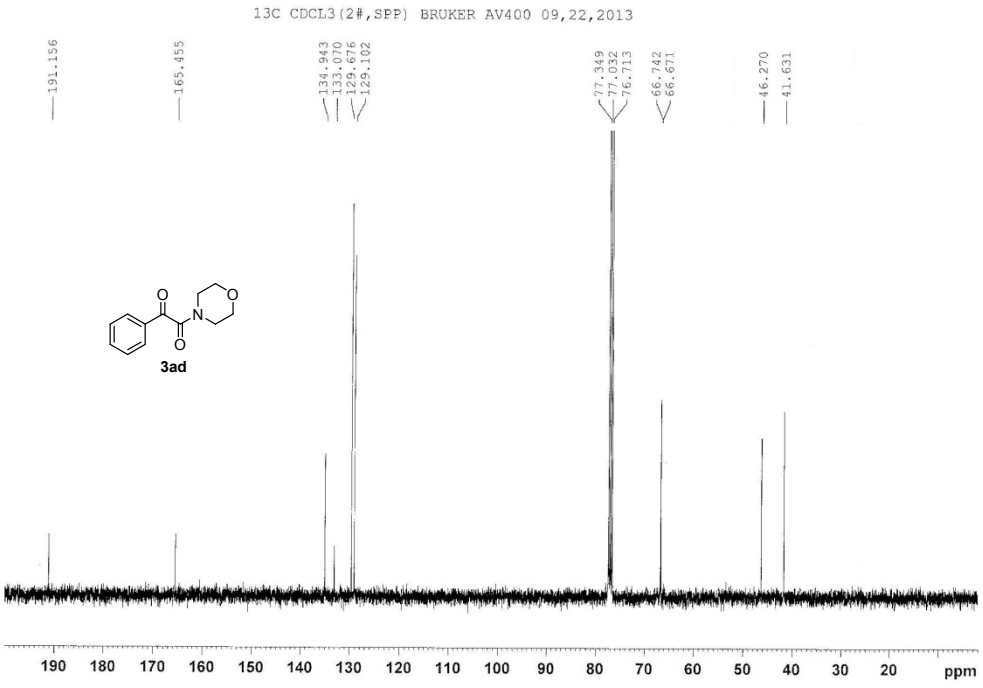

1H CDCL3(1#,SPP) BRUKER AV400 12,11,2013
7.834
7.829
7.817
7.813
7.683
7.678
7.673
7.661
7.656
7.282
3.717
3.704
3.302
3.289
3.275
1.713
1.706
1.699
1.573
1.565
1.560
1.550
Br
O
N
O
3fc
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.949
1.896
2.051
2.000
4.167
2.149

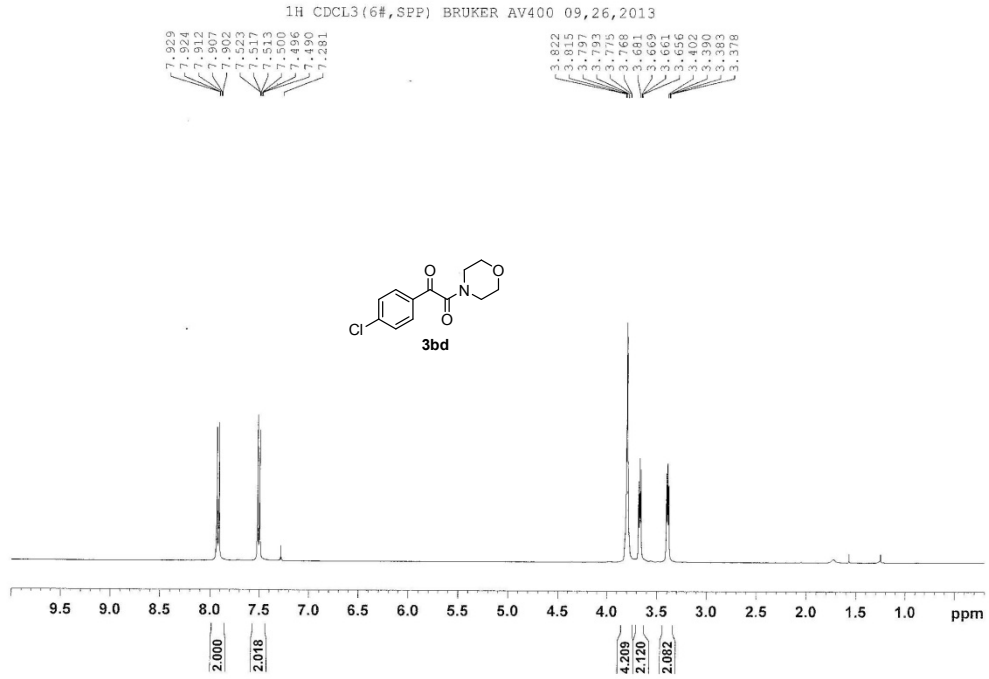

13C CDCL3(1#,SPP) BRUKER AV400 12,11,2013
190.737
164.884
132.397
132.074
130.966
130.122
77.371
77.053
76.736
47.060
42.258
26.256
25.448
24.349
Br
O
N
O
3fc
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

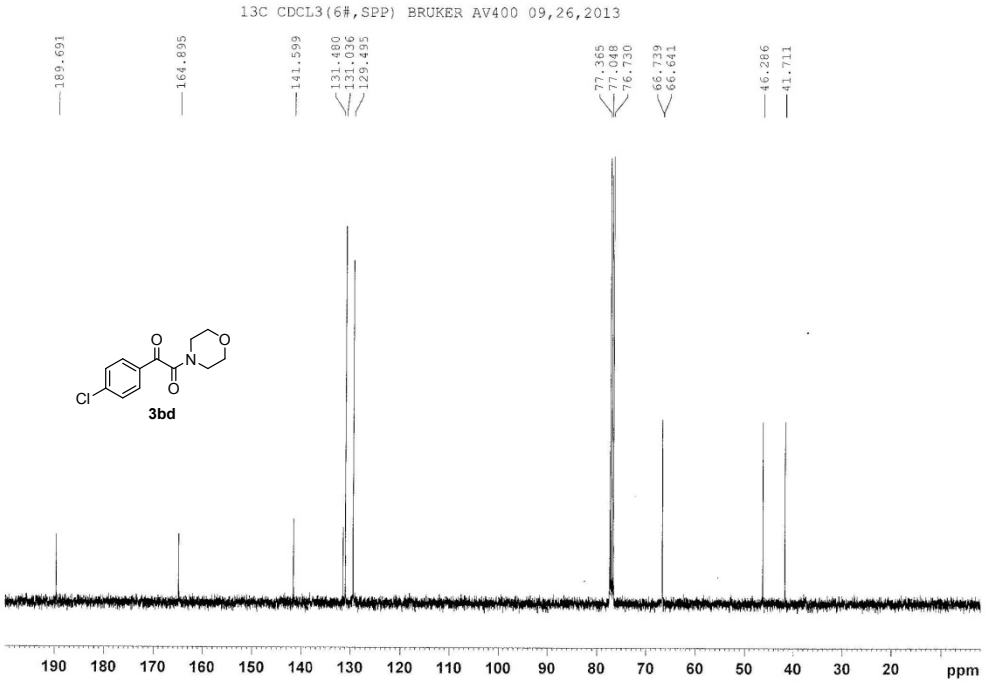

1H CDCl3(3#-131210) BRUKER AV400
7.837
7.832
7.819
7.818
7.813
7.670
7.666
7.651
7.647
7.496
7.493
7.478
7.474
7.460
7.458
7.452
7.439
7.433
7.420
7.415
7.282
3.837
3.836
3.826
3.821
3.813
3.809
3.803
3.797
3.789
3.785
3.765
3.759
3.751
3.747
3.737
3.602
3.597
3.589
3.583
3.577
Br
O
N
O
O
3dd
9.5 9.0 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 ppm
1.000
1.019
2.037
4.071
2.088
2.061

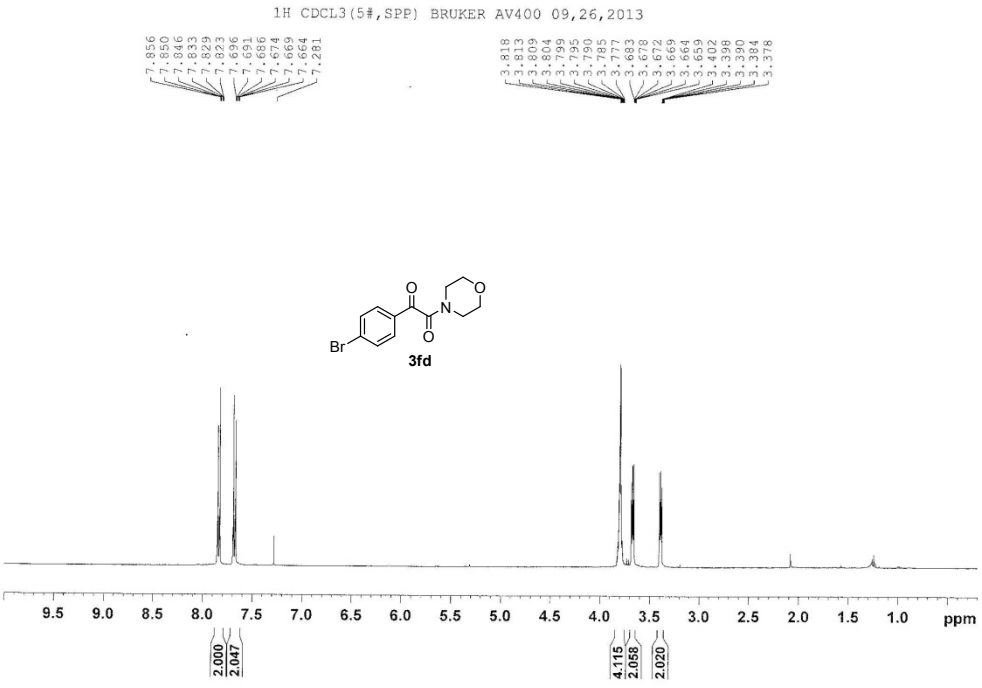

13C CDCl3(3#-131210) BRUKER AV400
190.428
164.836
135.470
134.287
134.037
132.684
127.876
121.482
66.302
66.247
46.304
42.045
Br
O
N
O
O
3dd
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

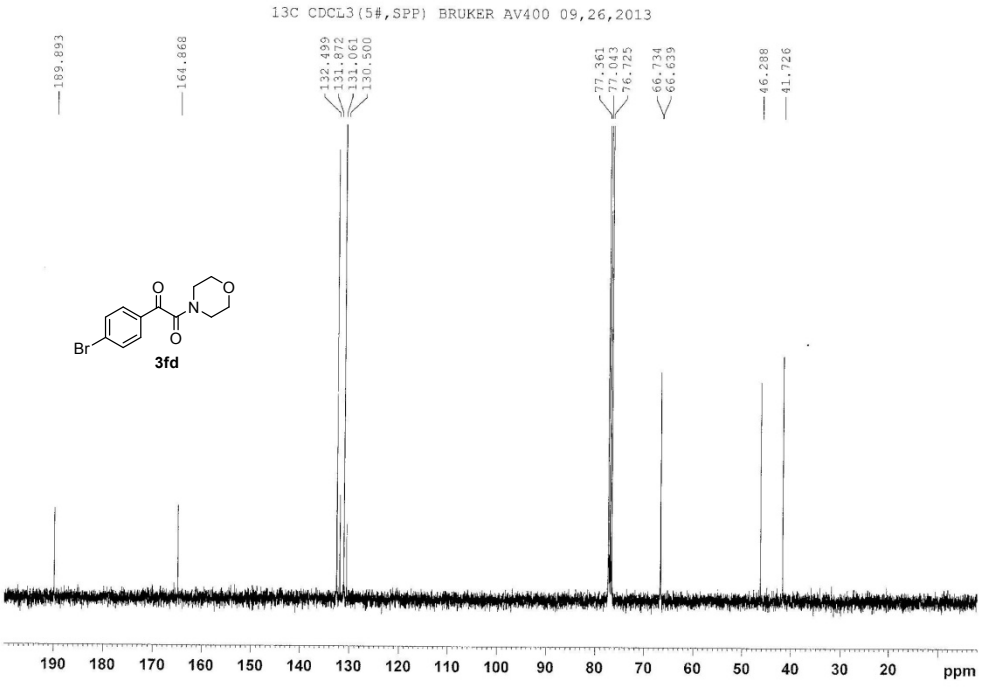

1H CDCL3(2#,SPP) BRUKER AV400 09,22,2013
7.993
7.989
7.986
7.973
7.968
7.965
7.698
7.694
7.691
7.680
7.676
7.672
7.661
7.657
7.654
7.560
7.556
7.540
7.526
7.522
7.281
3.821
3.811
3.801
3.795
3.684
3.678
3.672
3.664
3.659
3.412
3.407
3.399
3.393
3.387
3ad
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.000
1.025
2.102
4.239
2.180
2.150

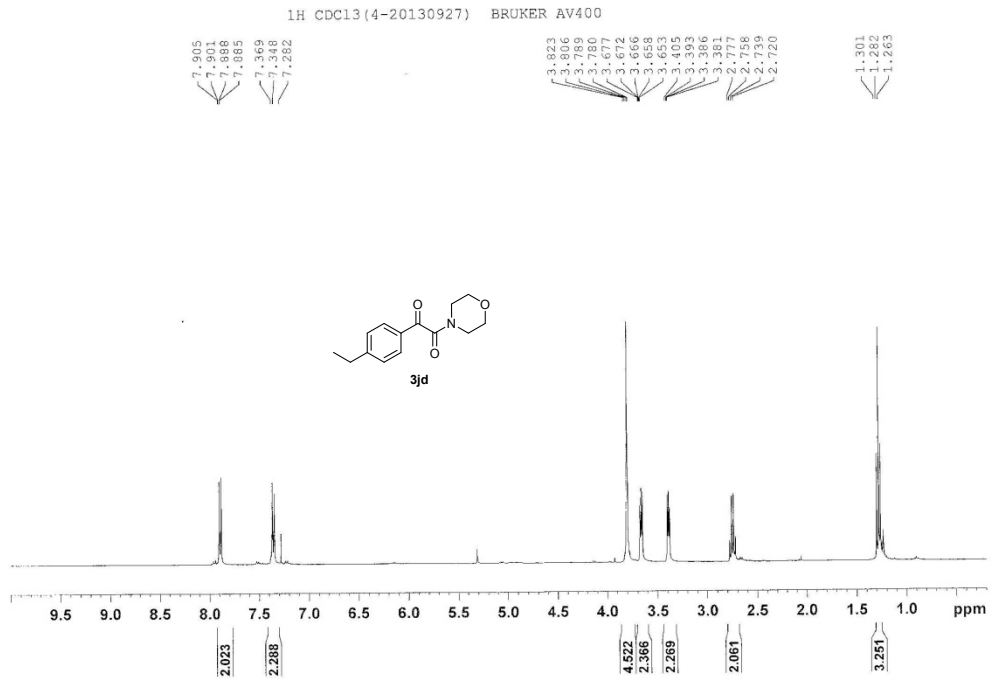

13C CDCL3(2#,SPP) BRUKER AV400 09,22,2013
191.156
165.455
134.943
133.070
129.676
129.102
77.349
77.032
76.713
66.742
66.671
46.270
41.631
3ad
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

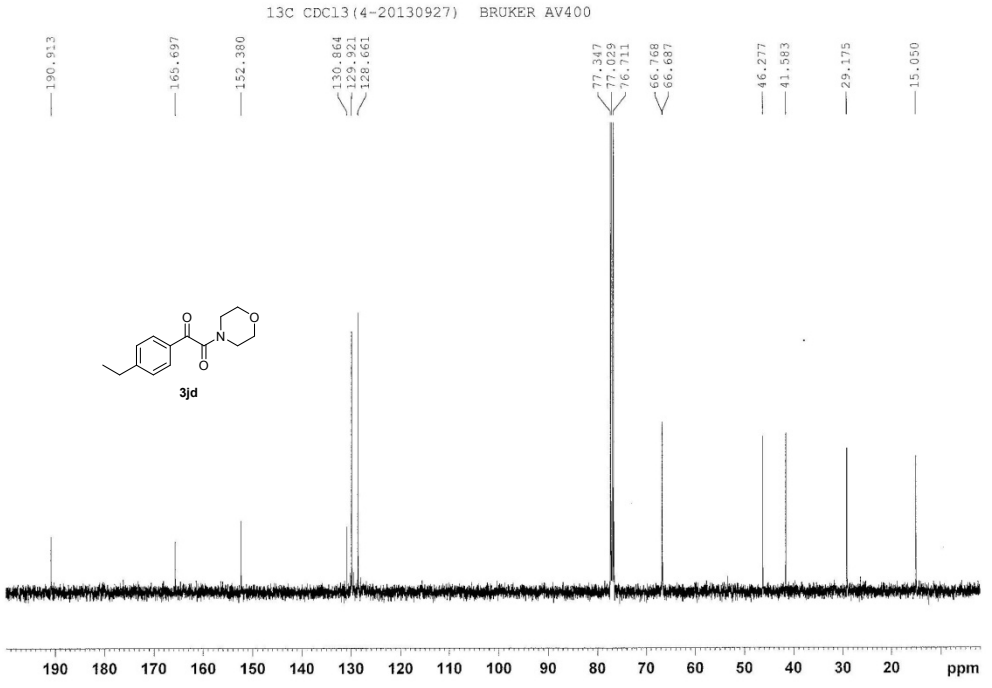

1H CDCL3(6#,SPP) BRUKER AV400 09,26,2013
7.929
7.924
7.912
7.907
7.902
7.523
7.517
7.513
7.500
7.496
7.490
7.281
3.822
3.815
3.797
3.793
3.775
3.768
3.681
3.669
3.661
3.656
3.402
3.390
3.383
3.378
3bd
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.000
2.018
4.209
2.120
2.082

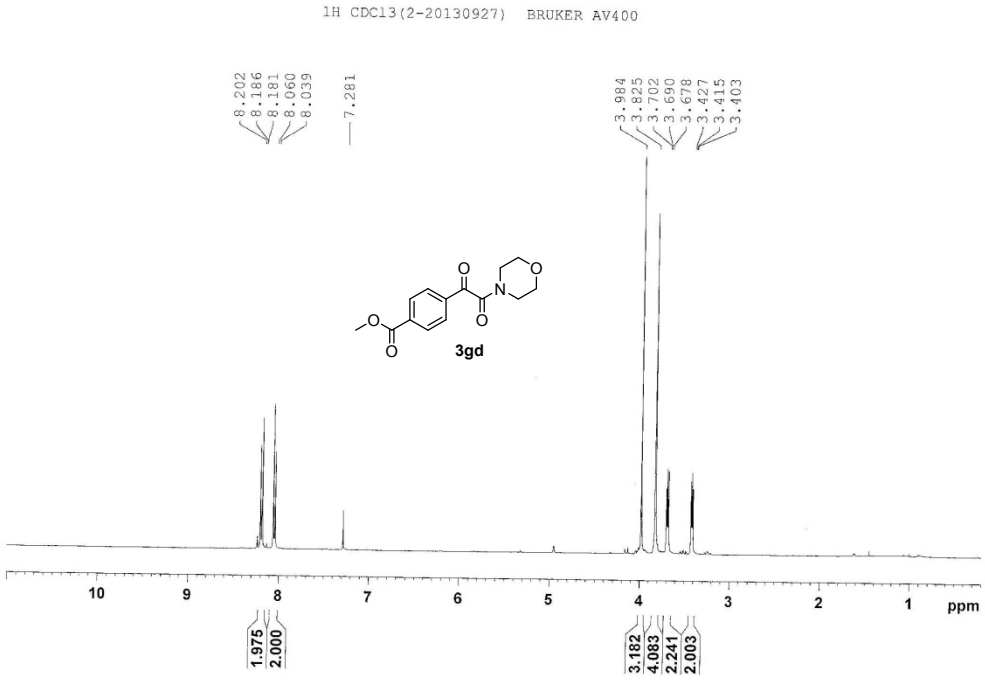

13C CDCL3(6#,SPP) BRUKER AV400 09,26,2013
189.691
164.895
141.599
131.480
131.036
129.495
77.365
77.048
76.730
66.739
66.641
46.286
41.711
3bd
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

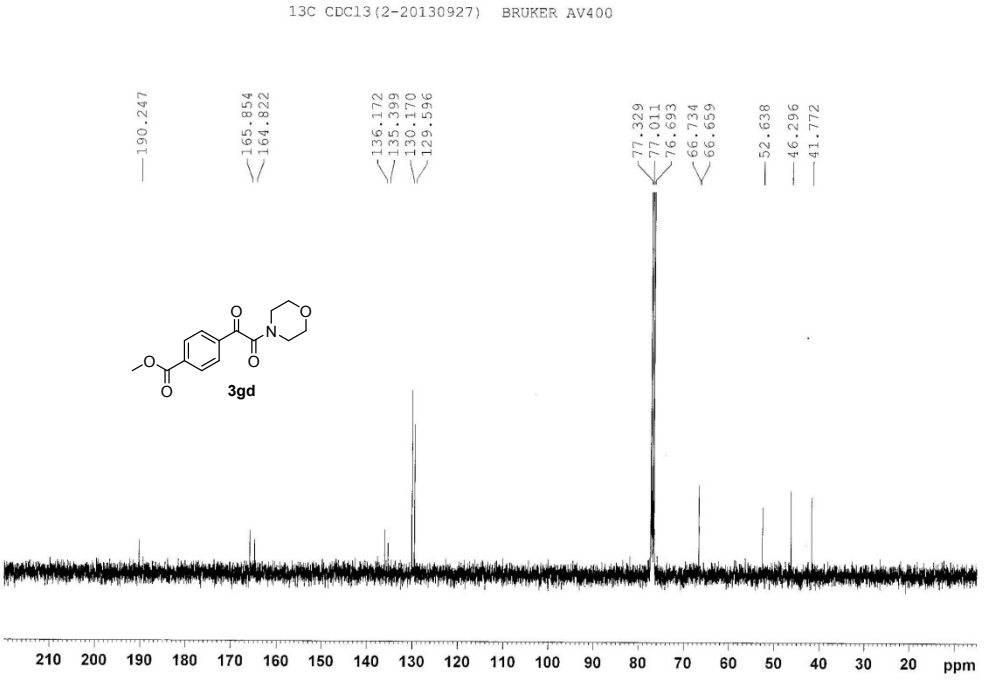

1H CDCL3(5#,SPP) BRUKER AV400 09,26,2013
7.856
7.850
7.846
7.833
7.829
7.823
7.696
7.691
7.686
7.674
7.669
7.664
7.281
3.818
3.813
3.809
3.804
3.799
3.795
3.790
3.785
3.777
3.683
3.678
3.672
3.669
3.664
3.659
3.402
3.398
3.390
3.384
3.378
Br
O
N
O
O
3fd
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.000
2.047
4.115
2.058
2.020

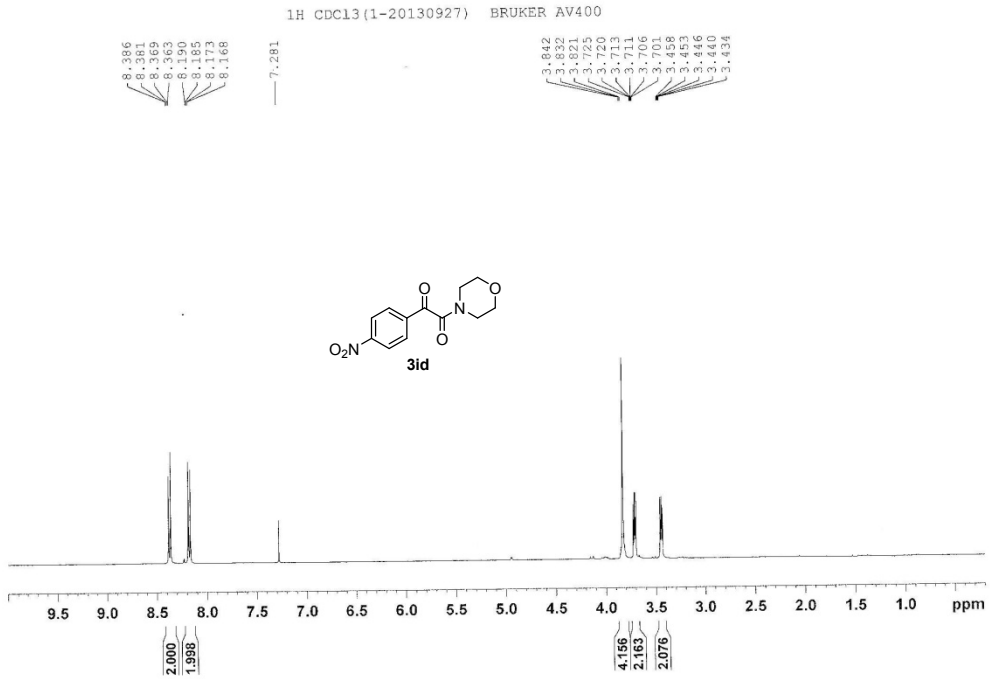

13C CDCL3(5#,SPP) BRUKER AV400 09,26,2013
189.893
164.868
132.499
131.872
131.061
130.500
77.361
77.043
76.725
66.734
66.639
46.288
41.726
Br
O
N
O
O
3fd
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

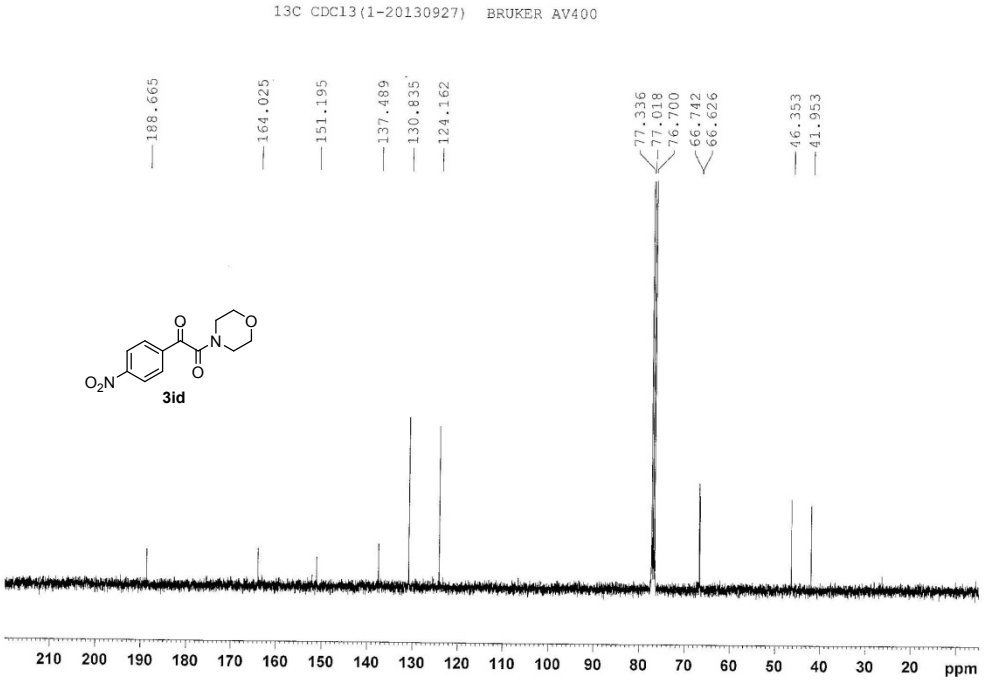

1H CDCl3(4-20130927) BRUKER AV400
7.905
7.901
7.888
7.885
7.369
7.348
7.282
3.823
3.806
3.789
3.780
3.677
3.672
3.666
3.658
3.653
3.405
3.393
3.386
3.381
2.777
2.758
2.739
2.720
1.301
1.282
1.263
3jd
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.023
2.288
4.522
2.366
2.269
2.061
3.251

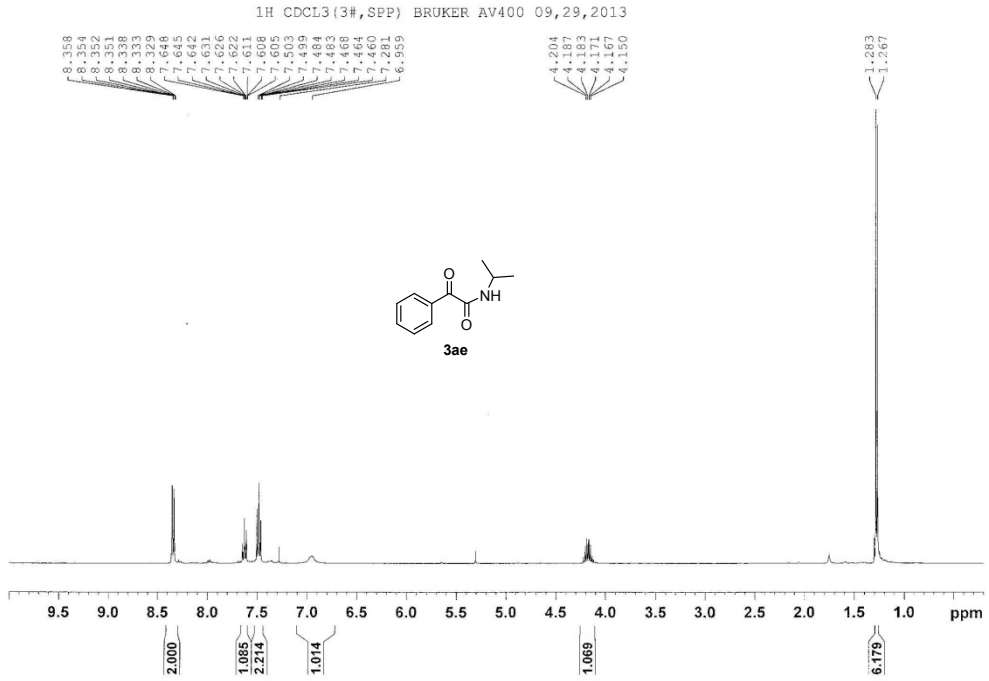

13C CDCl3(4-20130927) BRUKER AV400
190.913
165.697
152.380
130.864
129.921
128.661
77.347
77.029
76.711
66.768
66.687
46.277
41.583
29.175
15.050
3jd
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

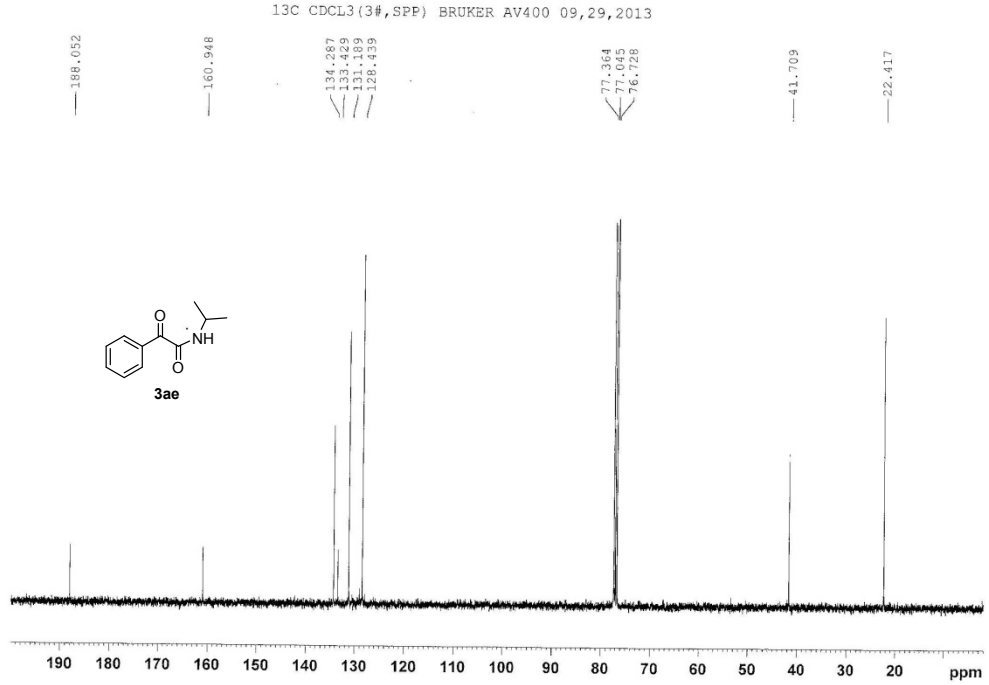

1H CDCl3(2-20130927) BRUKER AV400
8.202
8.186
8.181
8.060
8.039
7.281
3.984
3.825
3.702
3.690
3.678
3.427
3.415
3.403
3gd
10
9
8
7
6
5
4
3
2
1
ppm
1.975
2.000
3.182
4.083
2.241
2.003

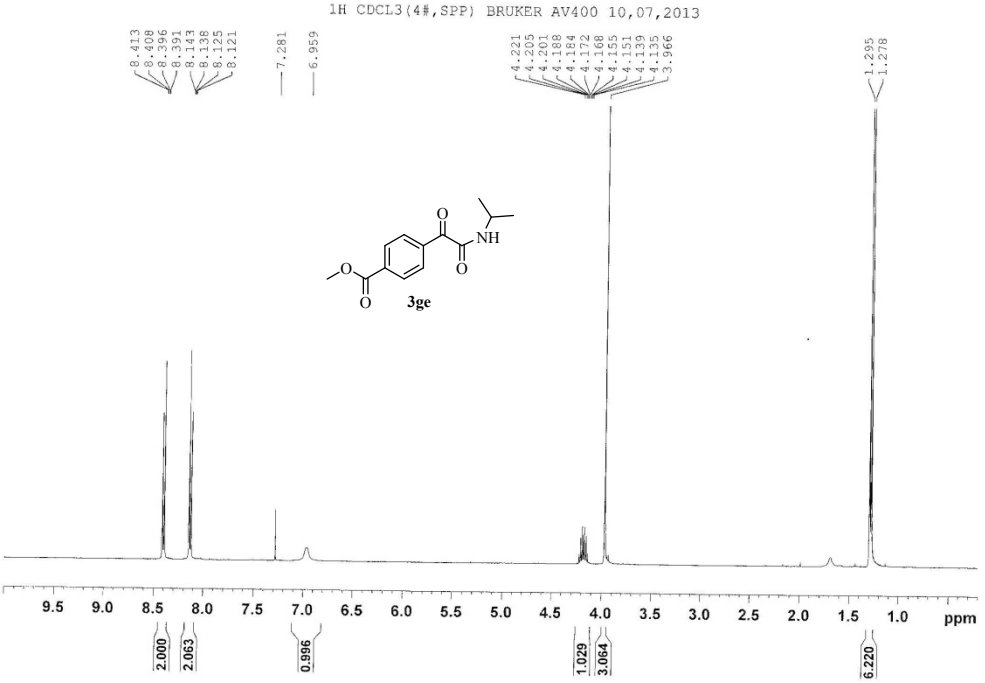

13C CDCl3(2-20130927) BRUKER AV400
190.247
165.854
164.822
136.172
135.399
130.170
129.596
77.329
77.011
76.693
66.734
66.659
52.638
46.296
41.772
3gd
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

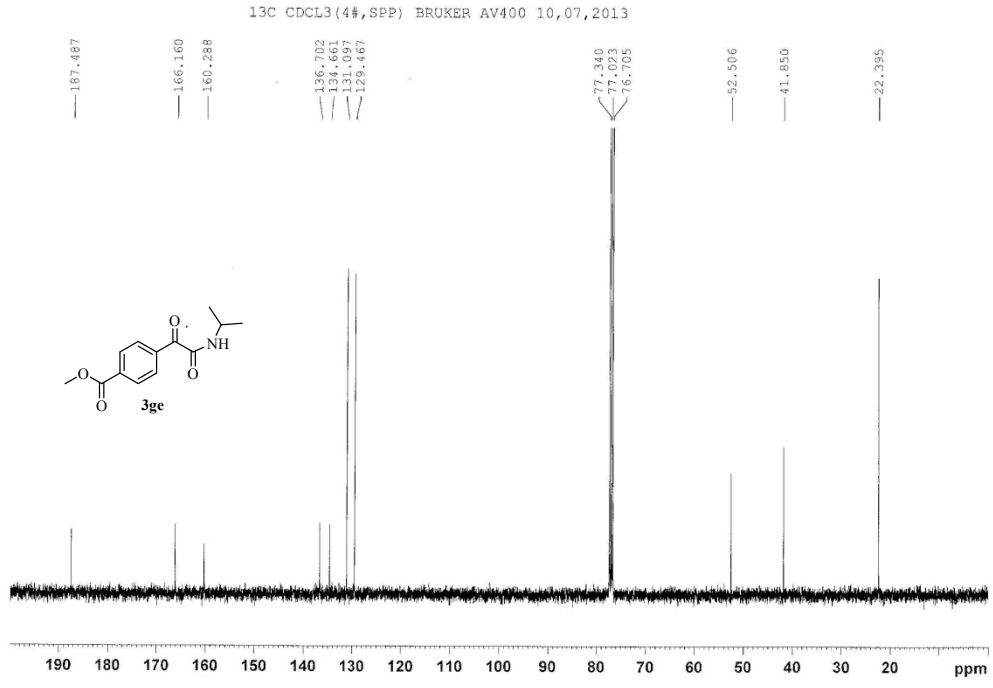

1H CDCl3(1-20130927) BRUKER AV400
8.386
8.381
8.369
8.363
8.190
8.185
8.173
8.168
7.281
3.842
3.832
3.821
3.725
3.720
3.713
3.711
3.706
3.701
3.458
3.453
3.446
3.440
3.434
3id
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.000
1.998
4.156
2.163
2.076


13C CDCl3(1-20130927) BRUKER AV400
188.665
164.025
151.195
137.489
130.835
124.162
77.336
77.018
76.700
66.742
66.626
46.353
41.953
3id
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


1H CDCL3(3#,SPP) BRUKER AV400 09,29,2013
8.358
8.354
8.352
8.351
8.338
8.333
8.329
7.648
7.645
7.642
7.631
7.626
7.622
7.611
7.608
7.605
7.503
7.499
7.484
7.468
7.463
7.464
7.460
7.281
6.959
4.204
4.187
4.183
4.171
4.167
4.150
1.283
1.267
O
NH
O
3ae
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 ppm
2.000
1.085
2.214
1.014
1.069
6.179


13C CDCL3(3#,SPP) BRUKER AV400 09,29,2013
188.052
160.948
134.287
133.429
131.139
128.439
77.364
77.045
76.728
41.709
22.417
O
NH
O
3ae
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


1H CDCL3(4#,SPP) BRUKER AV400 10,07,2013
8.413
8.408
8.396
8.391
8.143
8.138
8.125
8.121
7.281
6.959
4.221
4.205
4.201
4.188
4.184
4.172
4.168
4.155
4.151
4.139
4.135
3.966
1.295
1.278
3ge
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 ppm
2.000
2.063
0.996
1.029
3.064
6.220


13C CDCL3(4#,SPP) BRUKER AV400 10,07,2013
187.487
166.160
160.288
136.702
134.661
131.097
129.467
77.340
77.023
76.705
52.506
41.850
22.395
3ge
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm