Supplementary Material (ESI) for Chemical Communications
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Supplementary information:

Diastereoselective HOTf-catalyzed three-component one-pot 1,3-dipolar cycloaddition of α-diazo ester, nitrosobenzene and electron-deficient alkene

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General information: Unless otherwise stated, all reagents were purchased from commercial suppliers and used without further purification. All solvents employed in the reactions were used directly without purification. Analytical thin layer chromatography (TLC) was performed using Merck 60 F254 precoated silica gel plate (0.2 mm thickness). Subsequent to elution, plates were visualized using UV radiation (254 nm) on Spectroline Model ENF-24061/F 254 nm. Further visualization was possible by staining with basic solution of potassium permanganate or acidic solution of ceric molybdate.

Flash chromatography was performed using Merck silica gel 60 with freshly distilled solvents. Columns were typically packed as slurry and equilibrated with the appropriate solvent system prior to use.

Proton nuclear magnetic resonance spectra (^{1}H NMR) were recorded on Bruker AMX 500 and Bruker Avance DPX300 spectrophotometer ($CDCl_3$ as solvent). Chemical shifts for ^{1}H NMR spectra are reported as δ in units of parts per million (ppm) relative to the signal of chloroform-d (δ 7.26, single) or solvent. Multiplicities were given as: s (singlet), d (doublet), t (triplet), dd (double of doublet) or m (multiplets). The number of protons (n) for a given resonance is indicated by nH. Coupling constants are reported as a *J* value in Hz. Carbon nuclear magnetic resonance spectra (^{13}C NMR) are reported as δ in units of parts per million (ppm) downfield relative to the signal of chloroform-d (δ 77.0, triplet).

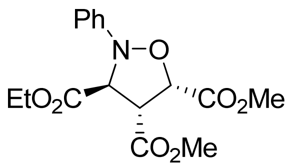
High resolution mass spectrometry (HRMS) was recorded on Finnigan MAT 95 × P spectrometer.

Experimental procedures and characterizations:

General procedure for the HOTf catalyzed three component 1,3-dipolar cycloaddition reaction of α-diazo ester, nitrosobenzene and electron deficient alkene

To a solution of nitrosobenzene[1] (0.25 mmol), electron deficient alkene (0.5 mmol) and HOTf (2 mol%) in CH_2Cl_2 (1.0 mL) was added α-diazo ester (0.3 mmol), the resulted solution was stirred at room temperature (23 °C) and monitored by TLC. Upon completion of consumption of nitrosobenze, the solution was concentrated and purified by flush column chromatography using an ethyl acetate/ hexane as eluant on silica gel to afford the desired cycloadduct.

Characterization of the product:

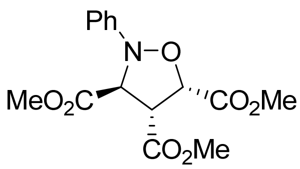


4a

^{1}H NMR (CDCl$_3$, 500 MHz): δ 1.31 (t, *J* = 7.2 Hz,3H), 3.52 (s, 3H), 3.68 (s, 3H), 4.26-4.34 (m, 3H), 4.79 (d, *J* = 7.0 Hz, 1H), 5.01 (d, *J* = 7.5 Hz, 1H), 6.99 (t, *J* = 7.5 Hz, 1H), 7.14 (d, *J* = 8.0 Hz, 2H), 7.26-7.27 (m, 2H).

^{13}C NMR (CDCl$_3$, 125 MHz): δ 14.1, 52.4, 52.7, 53.8, 62.4, 68.1, 77.6, 114.6, 122.7, 128.7, 150.4, 168.4, 169.7

HRMS (ESI): Anal. For $C_{16}H_{20}NO_7^{+1}$ (M^++1) Calcd.: 338.1240, Found: 338.1236

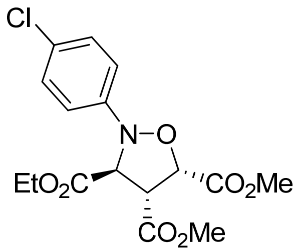


4b

^{1}H NMR (CDCl$_3$, 500 MHz): δ 3.51 (s, 3H), 3.68 (s, 3H), 3.84 (s, 3H), 4.33 (t, *J* = 7.0 Hz, 3H), 4.83 (d, *J* = 7.0 Hz, 1H), 5.01 (d, *J* = 7.0 Hz, 1H), 6.95 (t, *J* = 7.5 Hz, 1H), 7.13 (d, *J* = 8.0 Hz, 2H), 7.26-7.29 (m, 2H).

^{13}C NMR (CDCl$_3$, 125 MHz): δ 52.4, 52.7, 53.3, 53.8, 67.8, 77.7, 114.5, 122.7, 128.8, 150.3, 168.4, 170.2

HRMS (ESI): Anal. For $C_{15}H_{18}NO_7^{+1}$ (M^++1) Calcd.: 324.1083, Found: 324.1082

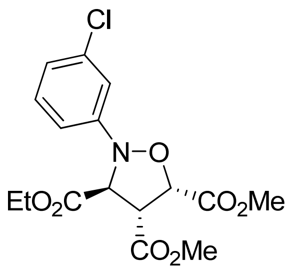


4c

^{1}H NMR (CDCl$_3$, 500 MHz): δ 1.30 (t, *J* = 7.2 Hz, 3H), 3.56 (s, 3H), 3.68 (s, 3H), 4.26-4.30 (m, 2H), 4.30-4.32 (m, 1H), 4.73 (d, *J* = 6.8 Hz, 1H), 4.99 (d, *J* = 7.4 Hz, 1H), 7.08 (d, *J* = 9.0 Hz, 2H), 7.22 (d, *J* = 9.0 Hz, 1H).

^{13}C NMR (CDCl$_3$, 125 MHz): δ 14.1, 52.6, 52.8, 53.8, 62.5, 68.0, 77.6, 115.9, 127.7, 128.7, 149.0, 168.3, 168.3, 169.3

HRMS (ESI): Anal. For $C_{16}H_{19}NO_7Cl^{+1}$ (M^++1) Calcd.: 372.0850, Found: 372.0846

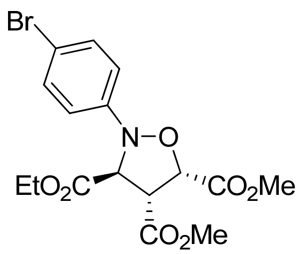


4d

^{1}H NMR (CDCl$_3$, 500 MHz): δ 1.32 (t, *J* = 7.2 Hz, 3H), 3.56 (s, 3H), 3.69 (s, 3H), 4.28-4.35 (m, 3H), 4.73 (d, *J* = 6.8 Hz, 1H), 4.99 (d, *J* = 7.4 Hz, 1H), 6.92-6.95 (m, 1H), 7.97-7.99 (m, 1H). 7.20-7.16 (m, 2H)

^{13}C NMR (CDCl$_3$, 125 MHz): δ 14.1, 52.6, 52.8, 53.6, 62.6, 67.8, 77.7, 112.2, 114.4, 122.3, 129.9, 134.6, 151.7, 168.2, 168.3, 169.4

HRMS (ESI): Anal. For $C_{16}H_{19}NO_7Cl^{+1}$ (M^++1) Calcd.: 372.0850, Found: 372.0853

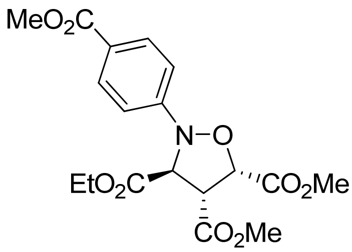


4e

^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.33 (t, *J* = 7.5 Hz, 3H), 3.58 (s, 3H), 3.70 (s, 3H), 4.29-4.32 (m, 2H), 4.33-4.35 (m, 1H), 4.75 (d, *J* = 6.8 Hz, 1H), 5.01 (d, *J* = 7.4 Hz, 1H), 7.04 (d, *J* = 9.0 Hz, 2H), 7.39 (d, *J* = 9.0 Hz, 1H).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 14.1, 52.6, 52.8, 53.7, 62.6, 67.9, 77.6, 115.2, 116.2, 131.6, 149.5, 168.2, 168.3, 169.3

HRMS (ESI): Anal. For $C_{16}H_{19}NO_7Br^{+1}$ (M^{+}+1) Calcd.: 416.0345, Found: 416.0359

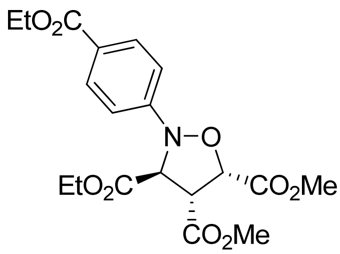


4f

^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.33 (t, *J* = 7.0 Hz, 3H), 3.51 (s, 3H), 3.69 (s, 3H), 3.88 (s, 3H), 4.29-4.34 (m, 2H), 4.37 (t, *J* = 7.0 Hz, 1H), 4.88 (d, *J* = 7.0 Hz, 1H), 5.03 (d, *J* = 7.0 Hz, 1H), 7.12 (d, *J* = 8.5 Hz, 2H), 7.60 (d, *J* = 8.5 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 14.1, 51.8, 52.6, 52.8, 53.6, 62.7, 67.0, 77.8, 112.8, 123.4, 130.7, 154.0, 166.8, 168.0, 168.1, 169.3

HRMS (ESI): Anal. For $C_{18}H_{22}NO_9^{+1}$ (M^{+}+1) Calcd.: 396.1295, Found: 396.1293

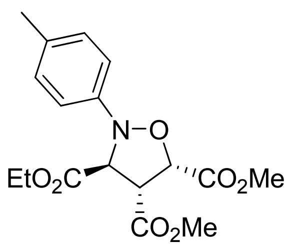


4g

^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.31 (t, *J* = 7.0 Hz, 3H), 1.35 (t, *J* = 7.0 Hz, 3H), 3.49 (s, 3H), 3.67 (s, 3H), 4.29-4.35 (m, 5H), 4.86 (d, *J* = 6.5 Hz, 1H), 5.00 (d, *J* = 7.5 Hz, 1H), 7.09 (d, *J* = 9.0 Hz, 2H), 7.94 (d, *J* = 9.0 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 14.1, 14.4, 52.6, 52.8, 53.7, 60.7, 62.7, 67.0, 77.8, 112.8, 123.8, 130.7, 154.0, 166.3, 168.1, 168.2, 169.4

HRMS (ESI): Anal. For $C_{19}H_{24}NO_9^{+1}$ (M^{+}+1) Calcd.: 410.1451, Found: 410.1451

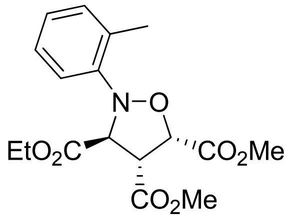


4h

^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.32 (t, *J* = 7.1 Hz, 3H), 2.30 (s, 3H), 3.59 (s, 3H), 3.70 (s, 3H), 4.26-4.32 (m, 3H), 4.74 (d, *J* = 7.0 Hz, 1H), 5.02 (d, *J* = 7.4 Hz, 1H), 7.07-7.11 (m, 4H).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 14.1, 20.7, 52.4, 52.7, 53.9, 62.3, 68.4, 77.5, 115.4, 129.3, 132.5, 147.8, 168.5, 168.6, 169.6

HRMS (ESI): Anal. For $C_{17}H_{22}NO_7^{+1}$ (M^{+}+1) Calcd.: 352.1396, Found: 352.1397

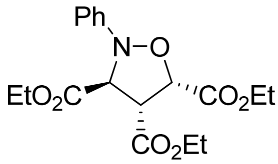


4i

^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.01 (t, *J* = 7.2 Hz, 3H), 2.30 (s, 3H), 3.73 (s, 3H), 3.76 (s, 3H), 3.94-4.01 (m, 2H), 4.21 (dd, *J* = 8.1, 5.6 Hz, 1H), 4.69 (d, *J* = 5.6 Hz, 1H), 5.04 (d, *J* = 8.1 Hz, 1H), 7.09-7.13 (m, 2H), 7.13-7.18 (m, 2H), 7.49 (d, *J* = 7.8 Hz, 1H).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 13.7, 17.9, 52.6, 52.7, 54.0, 61.7, 67.4, 76.7, 120.5, 126.4, 126.6, 130.4, 132.7, 145.0, 168.4, 168.9, 169.4

HRMS (ESI): Anal. For $C_{17}H_{22}NO_7^{+1}$ (M^{+}+1) Calcd.: 352.1396, Found: 352.1393

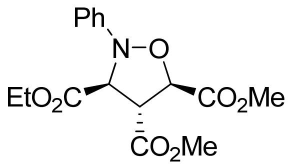


5a

^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.01 (t, *J* = 7.5 Hz, 3H), 1.17 (t, *J* = 7.1 Hz, 3H), 1.31 (t, *J* = 7.1 Hz, 3H), 3.82-4.00 (m, 2H), 4.09-4.13 (m, 2H), 4.21-4.30 (m, 2H), 4.31-4.34 (m, 1H), 4.77 (d, *J* = 7.1 Hz, 1H), 4.96 (d, *J* = 7.4 Hz, 1H), 6.95 (t, *J* = 7.4 Hz, 1H), 7.13 (d, *J* = 8.7 Hz, 2H), 7.24 (dt, *J* = 8.7, 7.4 Hz, 2H).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 13.5, 13.9, 14.1, 54.1, 61.7, 61.8, 62.4, 68.2, 77.9, 114.4, 122.5, 128.8, 150.9, 167.9, 167.9, 169.9

HRMS (ESI): Anal. For $C_{18}H_{24}NO_7^{+1}$ (M^{+}+1) Calcd.: 366.1553, Found: 366.1551

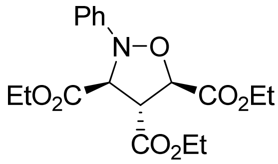


5b

^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.30 (t, *J* = 7.2 Hz, 3H), 3.59 (s, 3H), 3.84 (s, 3H), 4.25-4.29 (m, 2H), 4.33 (dd, *J* = 5.3, 3.6 Hz, 1H), 4.79 (d, *J* = 3.6 Hz, 1H), 5.11 (d, *J* = 5.3 Hz, 1H), 7.01 (t, *J* = 7.2 Hz, 1H), 7.11 (d, *J* = 7.2 Hz, 2H), 7.26-7.29 (m, 2H).

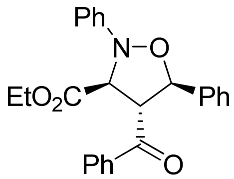
^{13}C NMR ($CDCl_3$, 125 MHz): δ 14.1, 52.9, 53.0, 54.2, 62.4, 70.4, 78.0, 115.4, 123.5, 129.0, 148.9, 168.8, 169.0, 169.9

HRMS (ESI): Anal. For $C_{16}H_{20}NO_7^{+1}$ (M^++1) Calcd.: 338.1240, Found: 338.1237



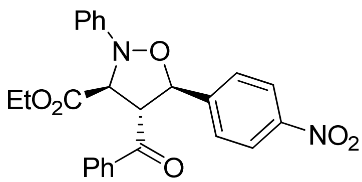
5c

^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.08 (t, *J* = 7.2 Hz, 3H), 1.30 (t, *J* = 7.2 Hz, 3H), 1.32 (t, *J* = 7.2 Hz, 3H), 4.02 (q, *J* = 7.2 Hz, 2H), 4.26-4.29 (m, 4H), 4.30-4.33 (m, 1H), 4.82 (d, *J* = 3.4 Hz, 1H), 5.10 (d, *J* = 5.4 Hz, 1H), 7.00 (t, *J* = 7.4 Hz, 1H), 7.09 (d, *J* = 7.8 Hz, 2H), 7.26 (m, 2H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 13.9, 14.1, 14.1, 54.5, 62.1, 62.4, 70.6, 78.2, 115.3, 123.4, 129.0, 149.1, 168.3, 169.1, 169.4
HRMS (ESI): Anal. For $C_{18}H_{24}NO_7^{+1}$ (M^++1) Calcd.: 366.1553, Found: 366.1549



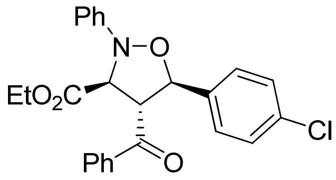
5d

^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.31 (t, *J* = 6.9 Hz, 3H), 4.29-4.38 (m, 2H), 4.98 (d, *J* = 5.1 Hz, 1H), 5.03 (dd, *J* = 8.7, 5.1 Hz, 1H), 5.30 (d, *J* = 8.7 Hz, 1H), 7.02 (t, *J* = 7.2 Hz, 1H), 7.17 (d, *J* = 7.8 Hz, 2H), 7.27-7.36 (m, 7H), 7.44-7.52 (m, 3H), 7.64 (d, *J* = 7.2 Hz, 2H).
^{13}C NMR ($CDCl_3$, 75 MHz): δ 14.1, 62.3, 62.4, 71.9, 84.4, 114.3, 122.4, 127.4, 128.6, 128.8, 128.9, 129.1, 129.2, 133.8, 135.9, 136.0, 150.4, 170.8, 195.9
HRMS (ESI): Anal. For $C_{25}H_{24}NO_4^{+1}$ (M^++1) Calcd.: 402.1705, Found: 402.1709



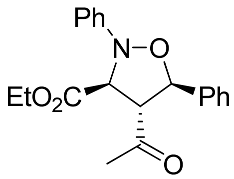
5e

^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.33 (t, *J* = 7.2 Hz, 3H), 4.31-4.39 (m, 2H), 4.87 (d, *J* = 4.8 Hz, 1H), 4.96 (dd, *J* = 8.1, 4.8 Hz, 1H), 5.58 (d, *J* = 8.1 Hz, 1H), 7.05 (t, *J* = 7.2 Hz, 1H), 7.13 (d, *J* = 8.7 Hz, 2H), 7.31-7.41 (m, 4H), 7.54-7.57 (m, 1H), 7.64-7.71 (m, 4H), 8.22 (d, *J* = 8.7 Hz, 2H).
^{13}C NMR ($CDCl_3$, 75 MHz): δ 14.1, 62.1, 62.7, 71.8, 82.2, 114.4, 123.0, 124.0, 128.1, 128.7, 128.9, 129.3, 134.2, 135.7, 143.9, 148.2, 149.7, 170.3, 195.4
HRMS (ESI): Anal. For $C_{25}H_{23}N_2O_6^{+1}$ (M^++1) Calcd.: 447.1556, Found: 447.1551



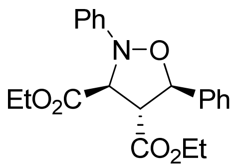
5f

^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.31 (t, *J* = 7.2 Hz, 3H), 4.28-4.39 (m, 2H), 4.93 (d, *J* = 6.0 Hz, 1H), 4.97 (dd, *J* = 8.4, 6.0 Hz, 1H), 5.33 (d, *J* = 8.4 Hz, 1H), 7.03 (t, *J* = 7.5 Hz, 1H), 7.15 (d, *J* = 7.8 Hz, 2H), 7.30-7.42 (m, 8H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.67 (d, *J* = 7.2 Hz, 2H).
^{13}C NMR ($CDCl_3$, 75 MHz): δ 14.1, 62.2, 62.5, 71.8, 83.4, 114.3, 122.6, 128.7, 128.8, 129.0, 129.2, 132.5, 134.0, 134.7, 134.9, 135.8, 150.1, 170.7, 195.8
HRMS (ESI): Anal. For $C_{25}H_{23}NO_4Cl^{+1}$ (M^++1) Calcd.: 436.1316, Found: 436.1317



5g

^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.36 (t, *J* = 7.2 Hz, 3H), 2.00 (s, 3H), 4.22 (dd, *J* = 9.3, 5.7 Hz, 1H), 4.31-4.36 (m, 2H), 4.94 (d, *J* = 5.7 Hz, 1H), 5.03 (d, *J* = 9.3 Hz, 1H), 7.00 (t, *J* = 7.2 Hz, 1H), 7.15 (d, *J* = 7.8 Hz, 2H), 7.26-7.34 (m, 2H), 7.40-7.46 (m, 3H), 7.53-7.56 (m, 2H).
^{13}C NMR ($CDCl_3$, 75 MHz): δ 14.2, 30.3, 62.3, 67.7, 70.2, 83.3, 114.2, 122.4, 127.7, 129.0, 129.2, 129.5, 135.7, 150.4, 170.8, 203.0
HRMS (ESI): Anal. For $C_{20}H_{22}NO_4^{+1}$ (M^++1) Calcd.: 340.1549, Found: 340.1553



5h

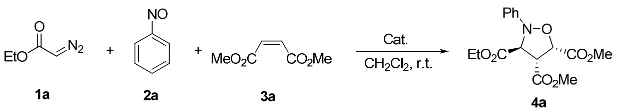
^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.12 (t, *J* = 7.2 Hz, 3H), 1.34 (t, *J* = 7.2 Hz, 3H), 4.04-4.11 (m, 3H), 4.29-4.36 (m, 2H), 4.96 (d, *J* = 4.8 Hz, 1H), 5.48 (d, *J* = 8.4 Hz, 1H), 7.01 (t, *J* = 7.2 Hz, 1H), 7.17 (d, *J* = 7.8 Hz, 2H), 7.29-7.34 (m, 2H), 7.36-7.44 (m, 3H), 7.51-7.55 (m, 2H).
^{13}C NMR ($CDCl_3$, 75 MHz): δ 14.0, 14.2, 59.3, 61.7, 62.3, 71.1, 83.1, 114.4, 122.5, 127.4, 128.7, 129.0, 129.2, 136.2, 150.1, 170.0, 170.3
HRMS (ESI): Anal. For $C_{21}H_{24}NO_5^{+1}$ (M^++1) Calcd.: 370.1654, Found: 370.1663

All the above cycloaddition products are liquid except products **4a** and **5e,** which X-ray crystal structures were determined as CCDC 755558 (**4a**: zgf43.cif) and CCDC 755559 (**5e**: zgf51.cif).

References:

1. D. Zhu, M. Lu, P. J. Chua, B. Tan, F. Wang, X. Yang and G. Zhong, *Org. Lett.*, 2008, **10**, 4585-4588.
2. A. Defoin, *Synthesis*, 2004, 706-710.

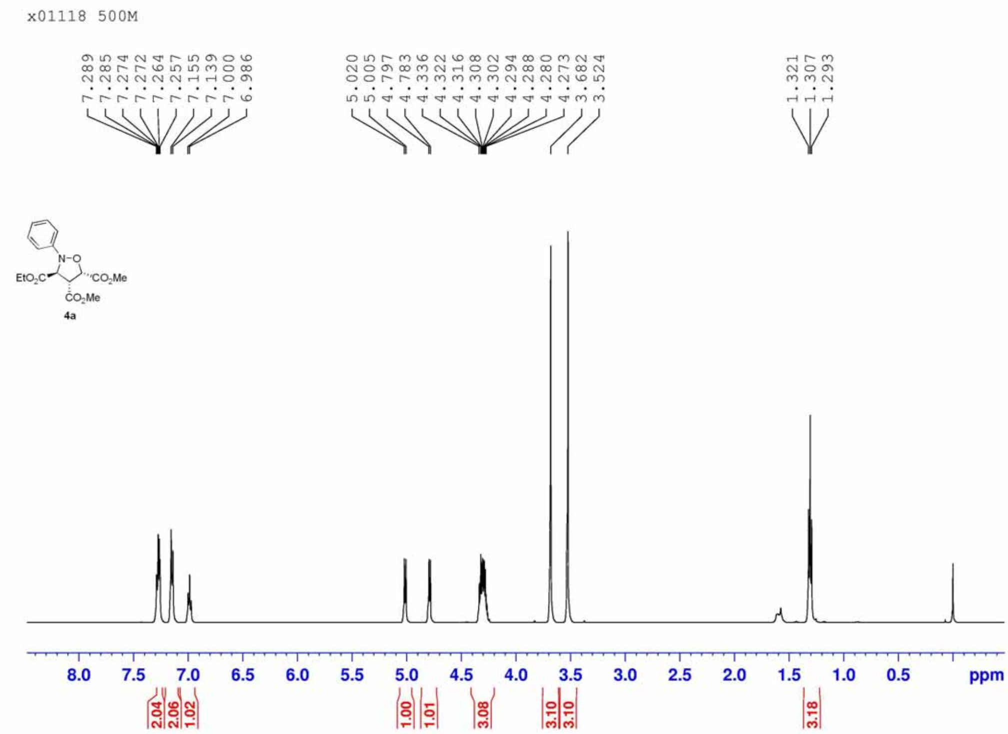
Table Effect of solvents[a]



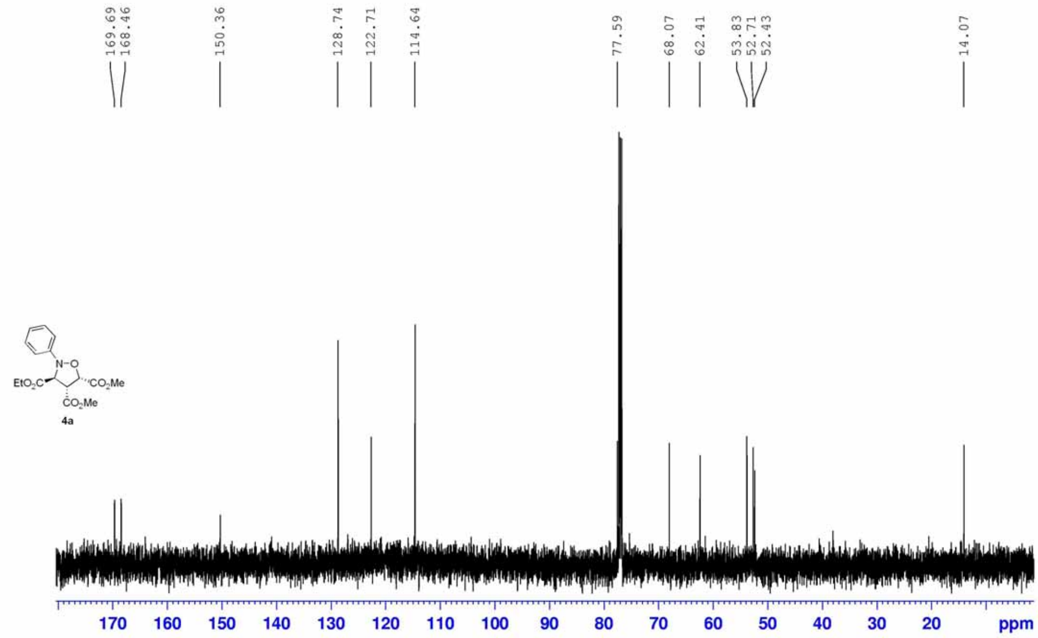
Entry	Solvent	Yield (%)[b]
1	CH_2Cl_2	98
2	$CHCl_3$	75
3	toluene	81
4	benzene	80
5	CH_3CN	85
6	THF	53
7	Et_2O	68

[a] Reaction conditions: HOTf/**1a**/**2a**/**3a** =0.02:1.2:1:2 at room temperature (23 ^{o}C). [b] Isolated yield.

NMR spectra:

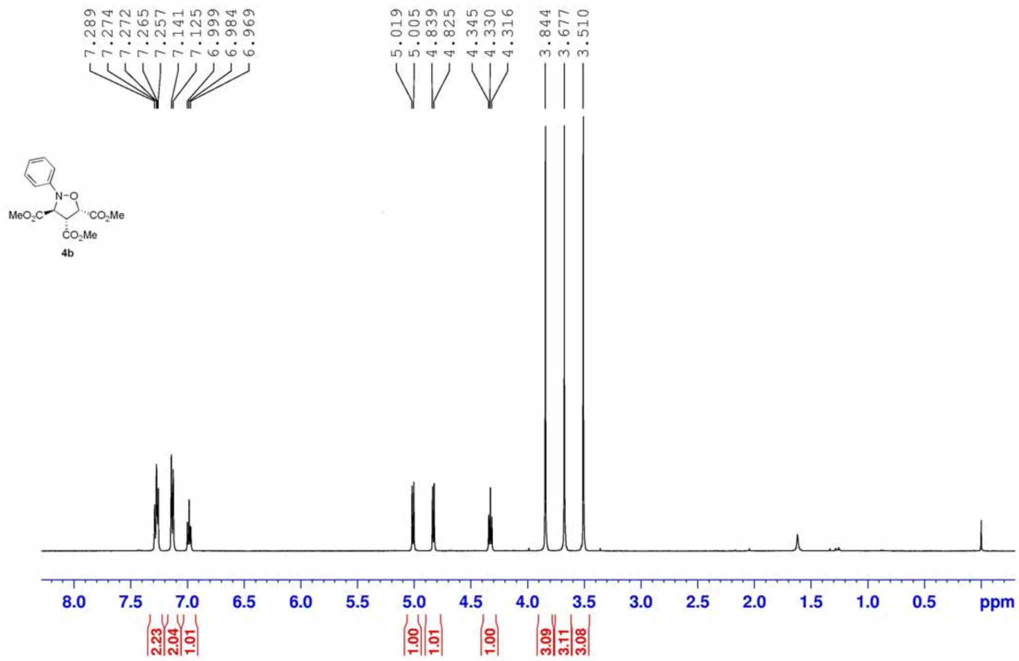

x01118 500M
7.289
7.285
7.274
7.272
7.264
7.257
7.155
7.139
7.000
6.986
5.020
5.005
4.797
4.783
4.336
4.322
4.316
4.308
4.302
4.294
4.288
4.280
4.273
3.682
3.524
1.321
1.307
1.293
N–O
EtO2C
CO2Me
CO2Me
4a
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.04
2.06
1.02
1.00
1.01
3.08
3.10
3.10
3.18

x01118 500M 13C

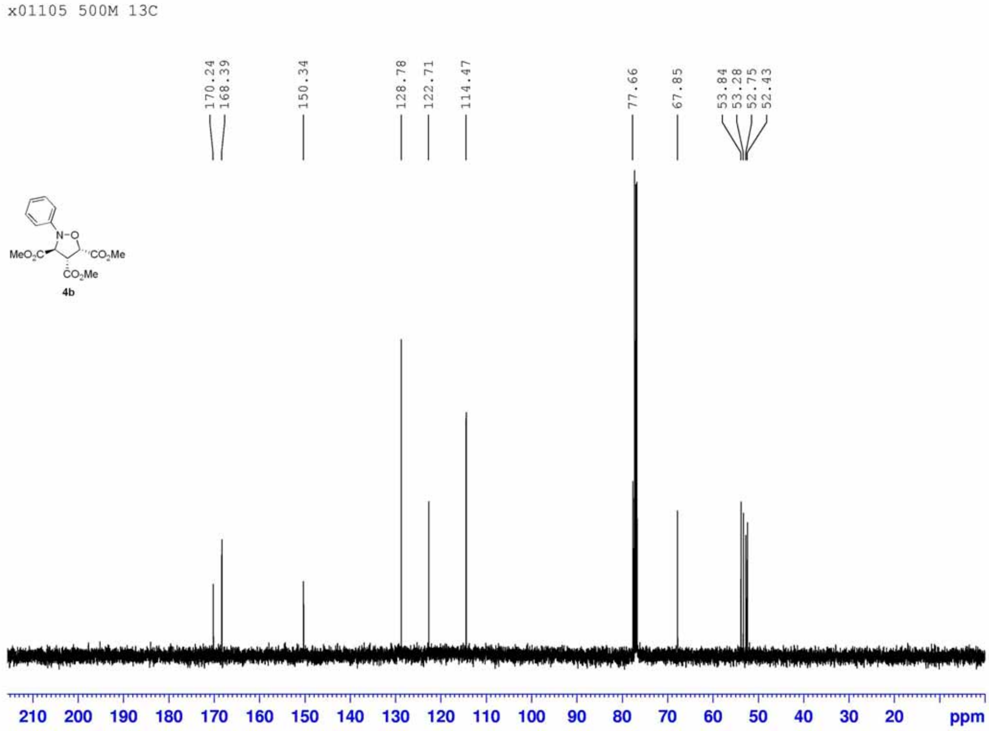


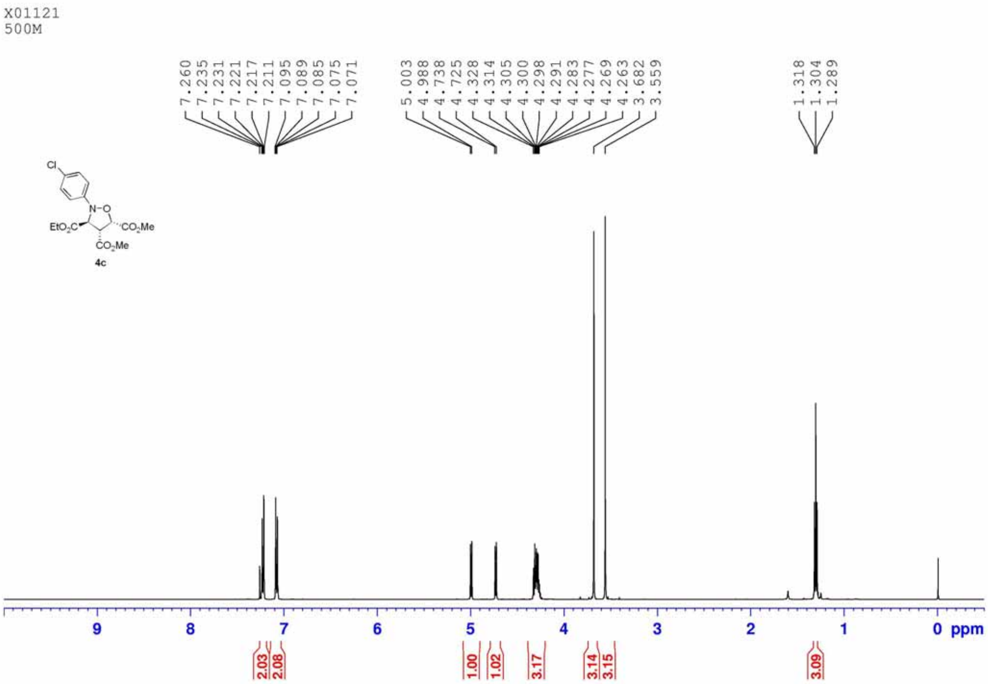
4a

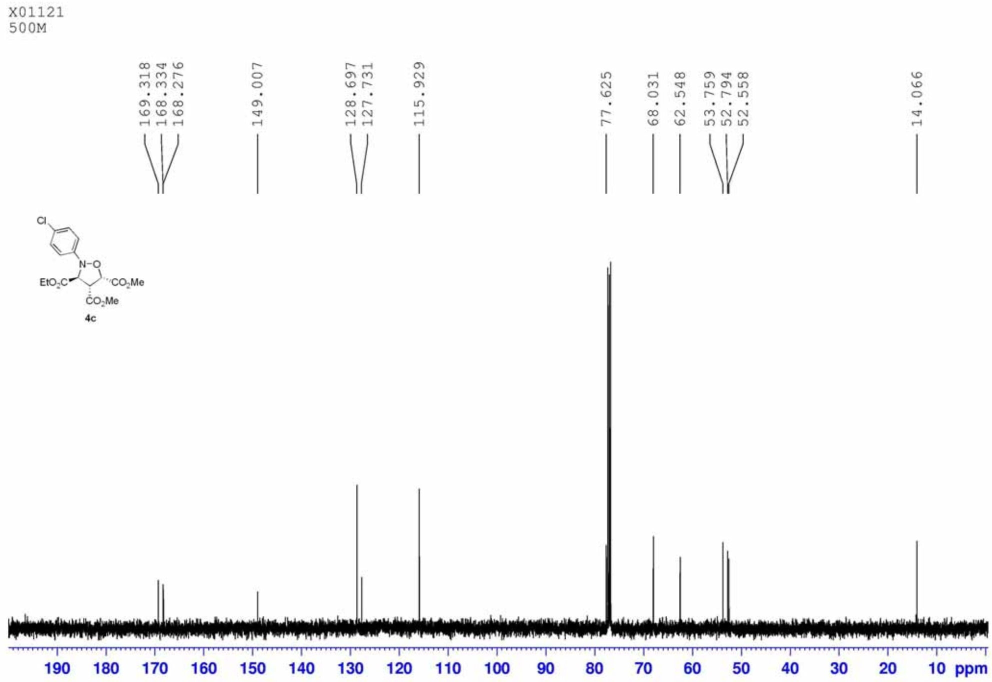
x01105 500M

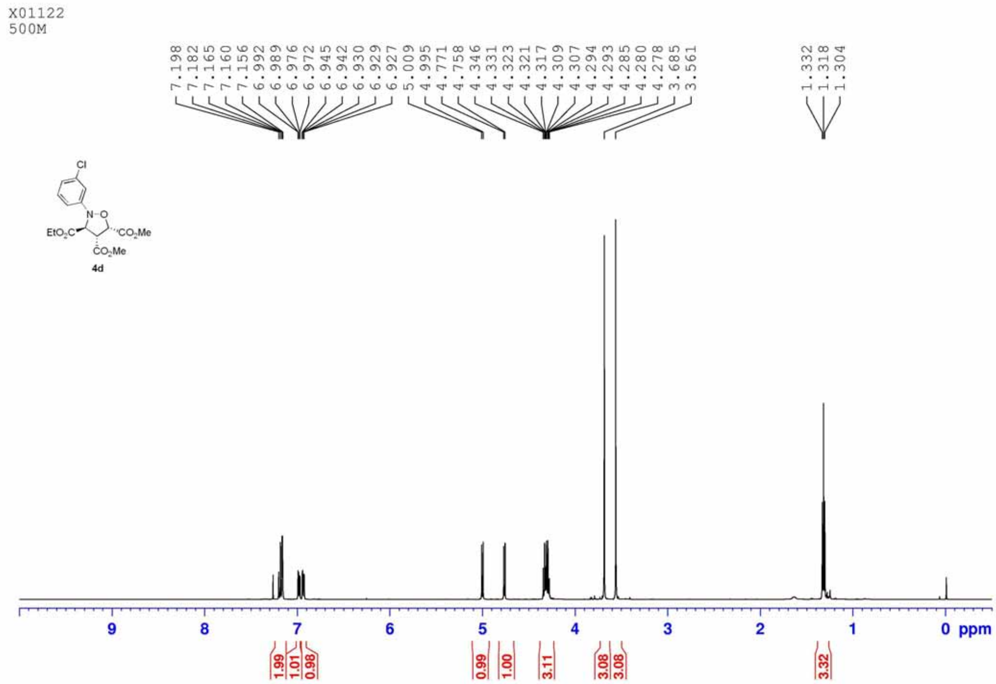


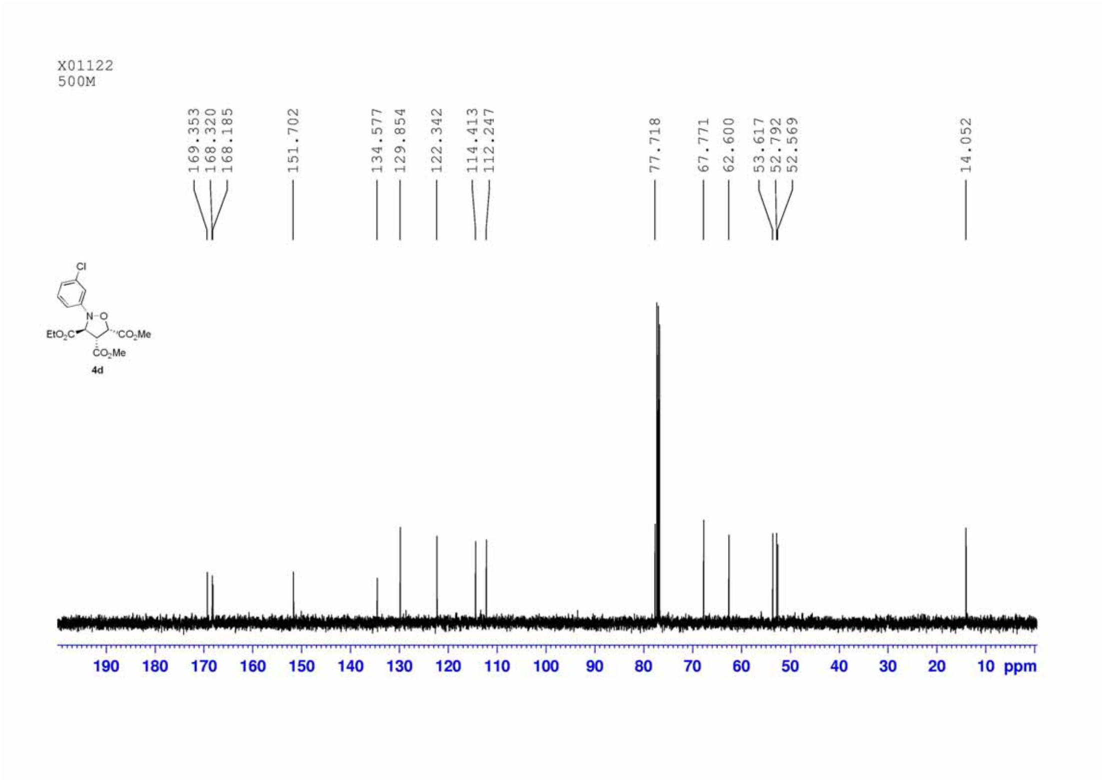
4b

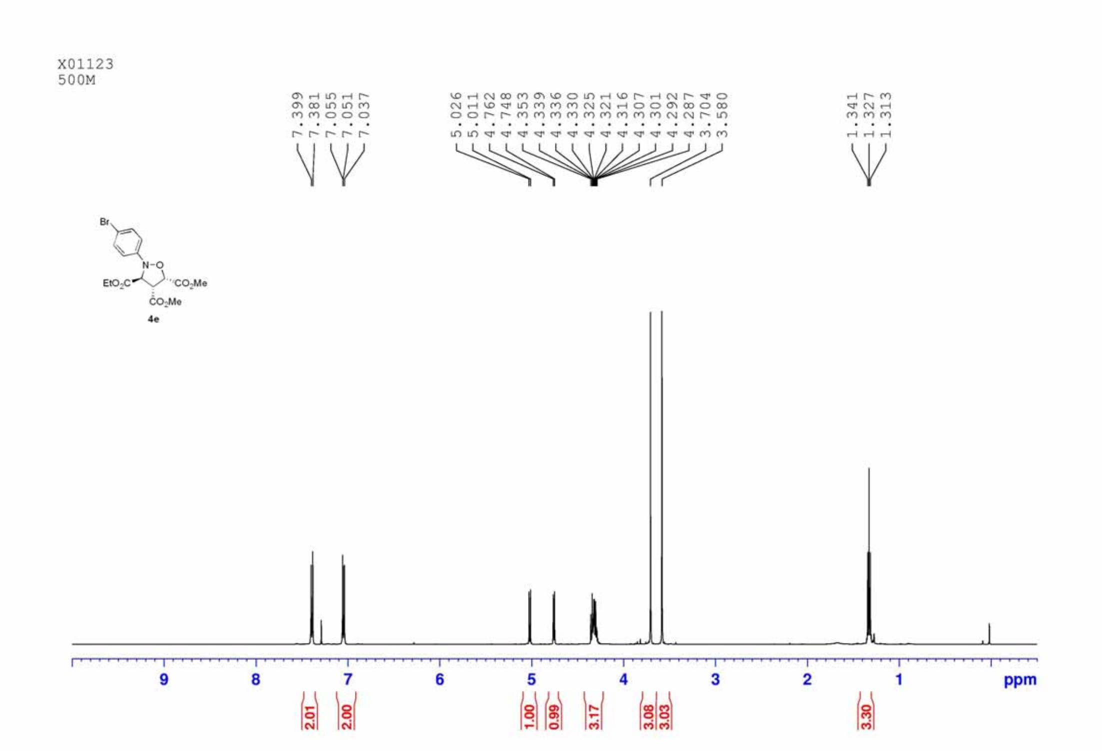

x01105 500M 13C
170.24
168.39
150.34
128.78
122.71
114.47
77.66
67.85
53.84
53.28
52.75
52.43
4b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

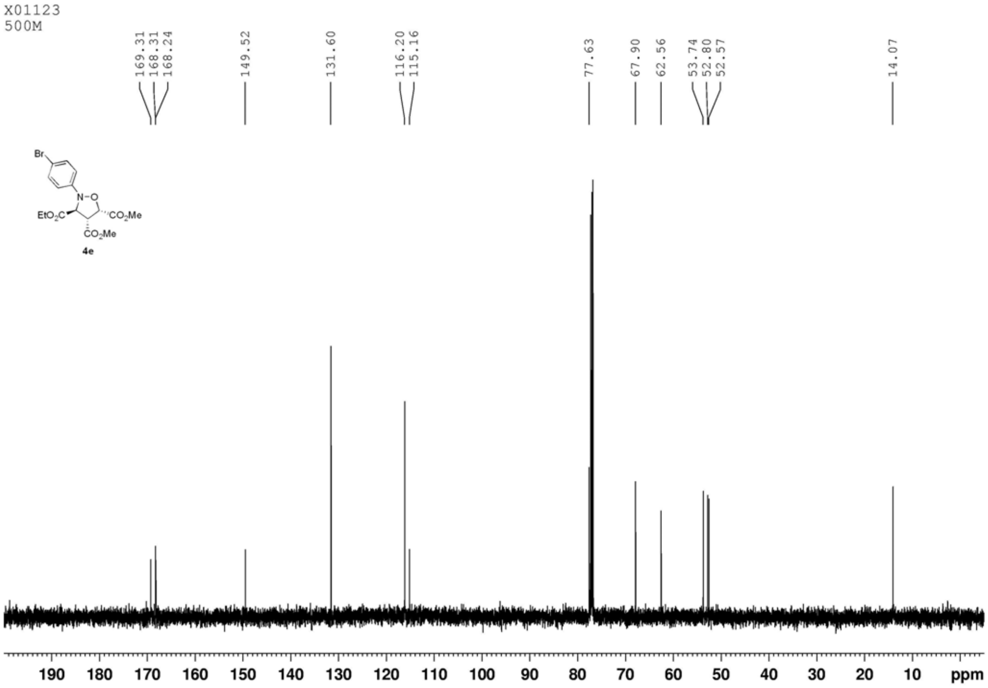

X01121
500M
7.260
7.235
7.231
7.221
7.217
7.211
7.095
7.089
7.085
7.075
7.071
5.003
4.988
4.738
4.725
4.328
4.314
4.305
4.300
4.298
4.291
4.283
4.277
4.269
4.263
3.682
3.559
1.318
1.304
1.289
4c
9 8 7 6 5 4 3 2 1 0 ppm
2.03
2.08
1.00
1.02
3.17
3.14
3.15
3.09

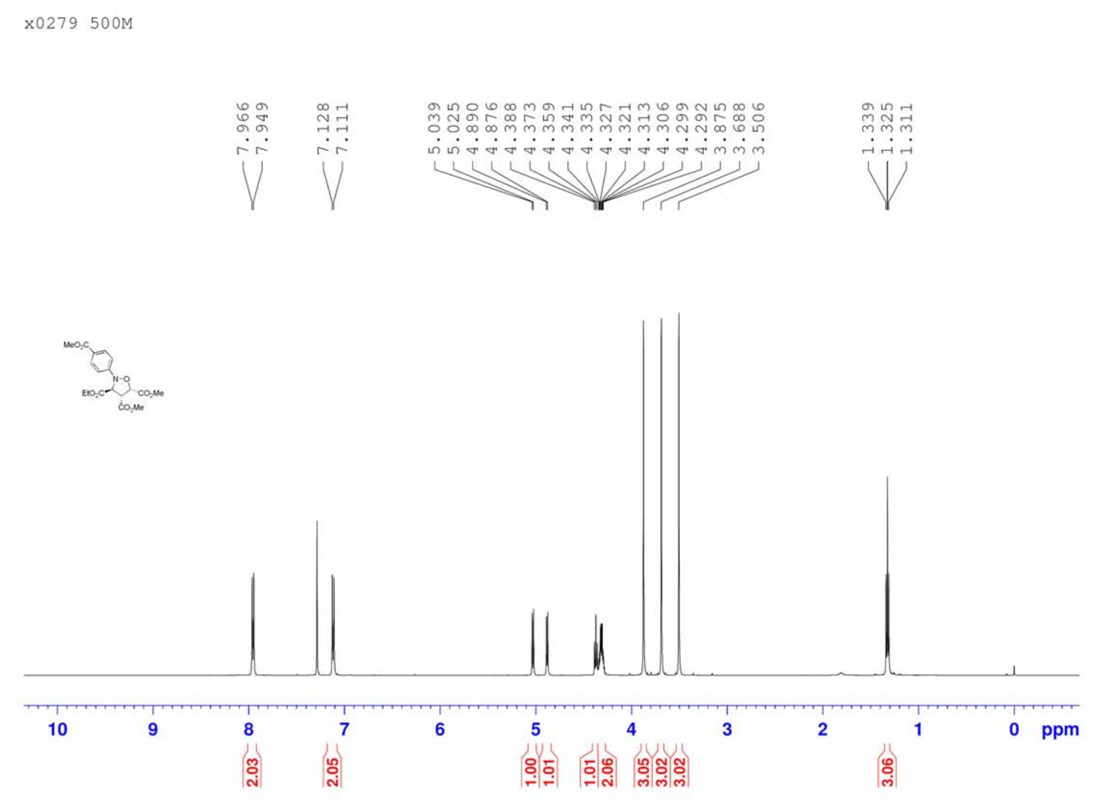

X01121
500M
169.318
168.334
168.276
149.007
128.697
127.731
115.929
77.625
68.031
62.548
53.759
52.794
52.558
14.066
Cl
N-O
EtO2C
CO2Me
CO2Me
4c
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

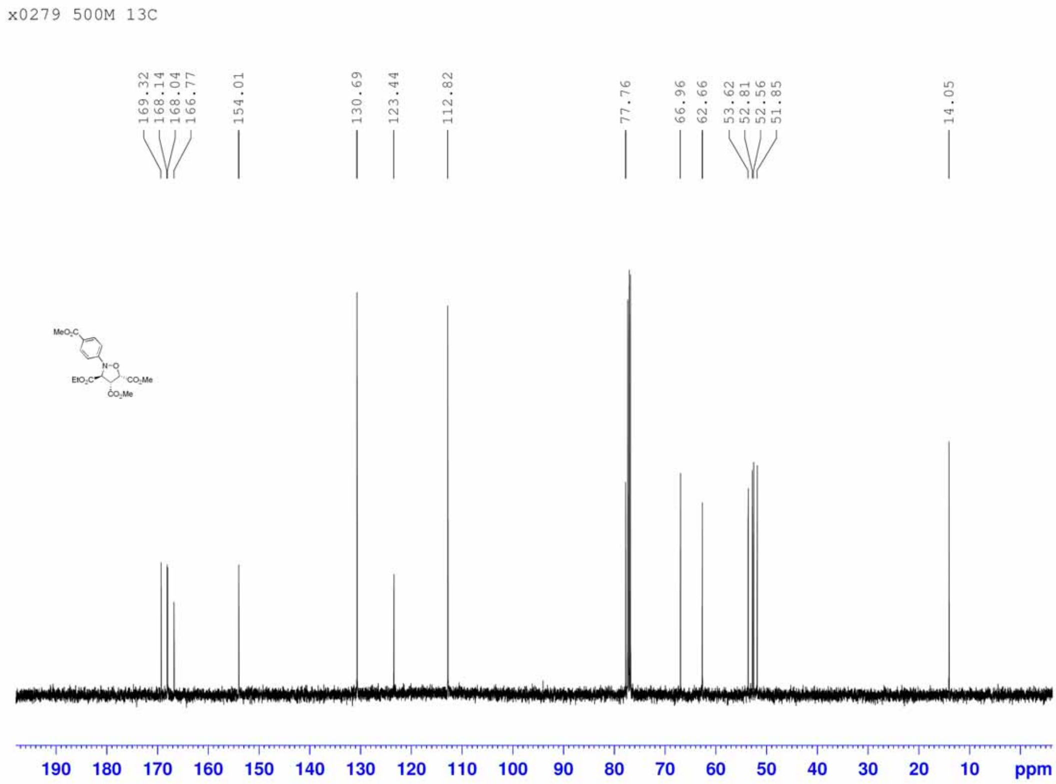

X01122
500M
7.198
7.182
7.165
7.160
7.156
6.992
6.989
6.976
6.972
6.945
6.942
6.930
6.929
6.927
5.009
4.995
4.771
4.758
4.346
4.331
4.323
4.321
4.317
4.309
4.307
4.294
4.293
4.285
4.280
4.278
3.685
3.561
1.332
1.318
1.304
Cl
N-O
EtO2C
CO2Me
CO2Me
4d
9 8 7 6 5 4 3 2 1 0 ppm
1.99
1.01
0.98
0.99
1.00
3.11
3.08
3.08
3.32

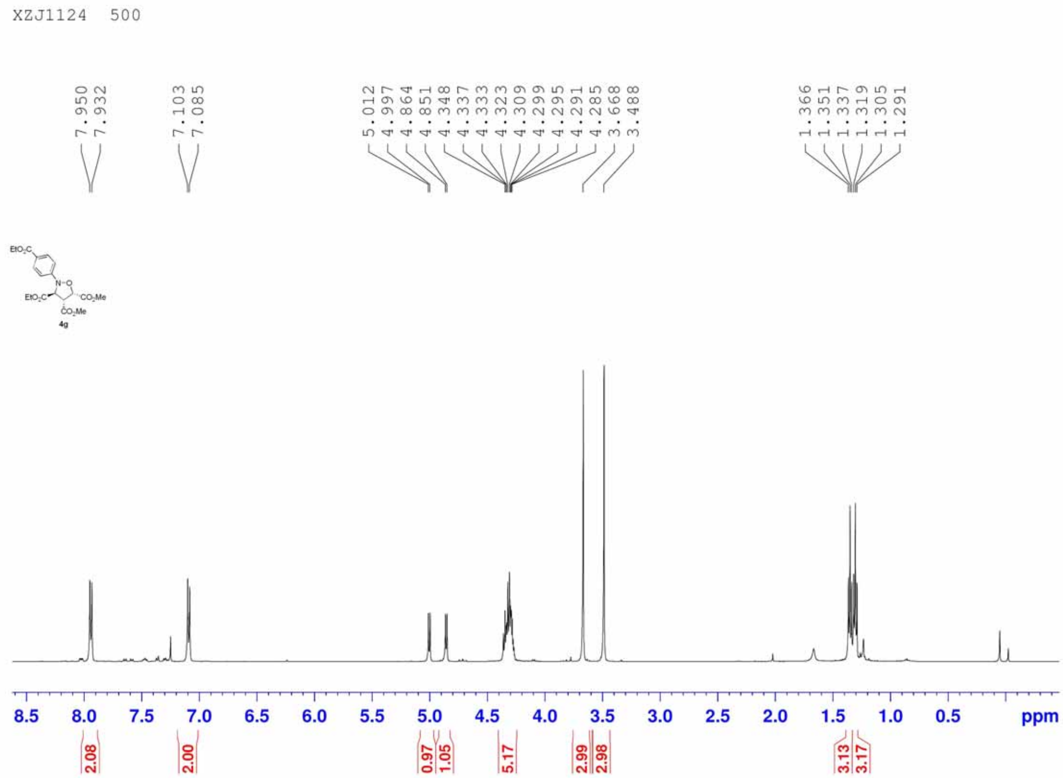

X01122
500M
169.353
168.320
168.185
151.702
134.577
129.854
122.342
114.413
112.247
77.718
67.771
62.600
53.617
52.792
52.569
14.052
4d
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

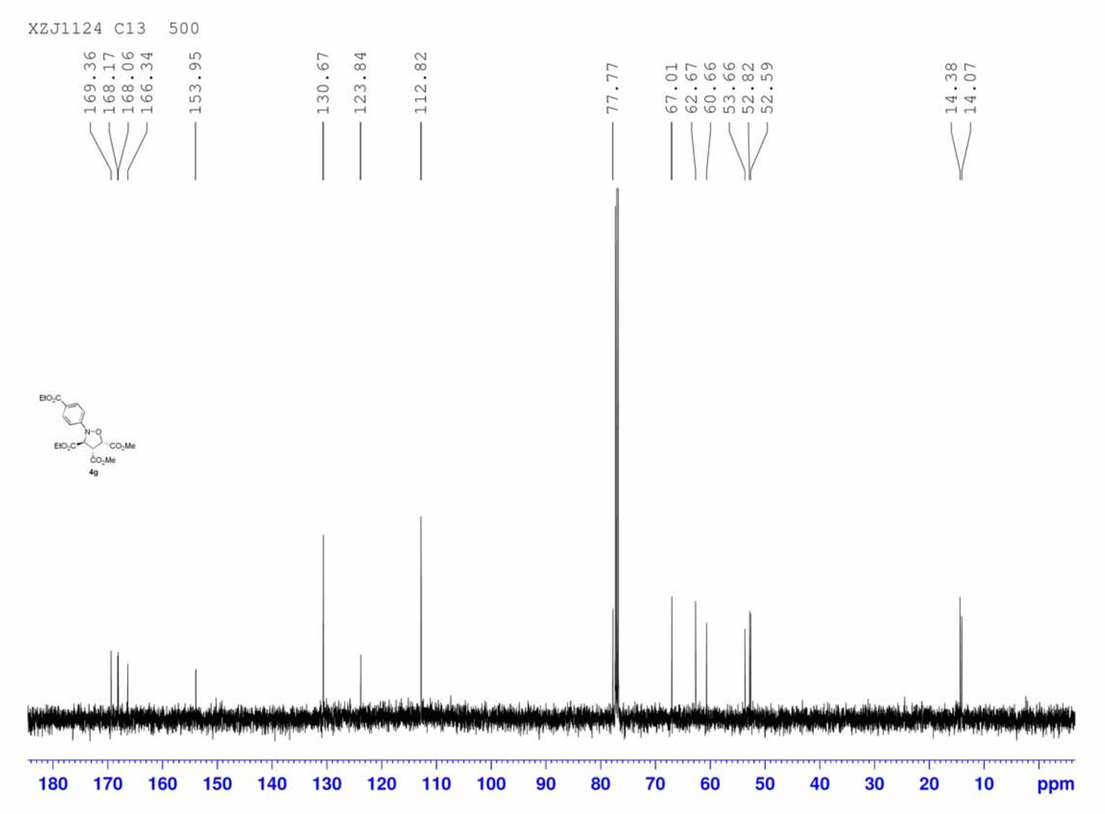

X01123
500M
7.399
7.381
7.055
7.051
7.037
5.026
5.011
4.762
4.748
4.353
4.339
4.336
4.330
4.325
4.321
4.316
4.307
4.301
4.292
4.287
3.704
3.580
1.341
1.327
1.313
4e
9 8 7 6 5 4 3 2 1 ppm
2.01
2.00
1.00
0.99
3.17
3.08
3.03
3.30

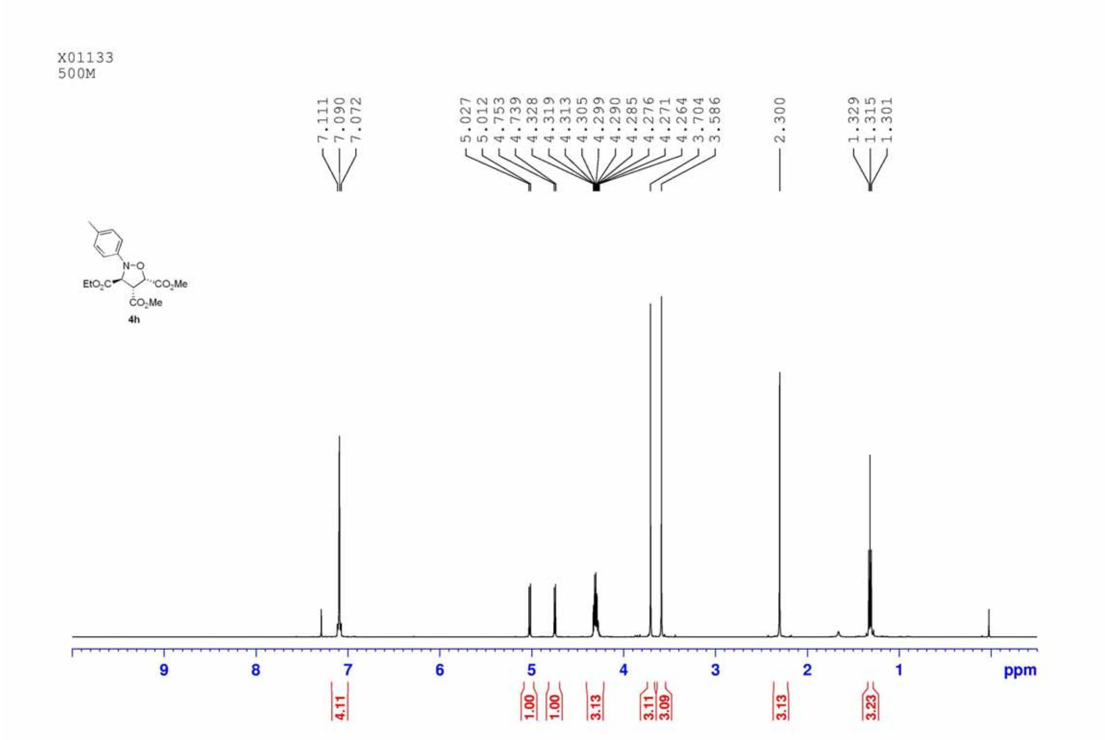

X01123
500M
169.31
168.31
168.24
149.52
131.60
116.20
115.16
77.63
67.90
62.56
53.74
52.80
52.57
14.07
4e
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

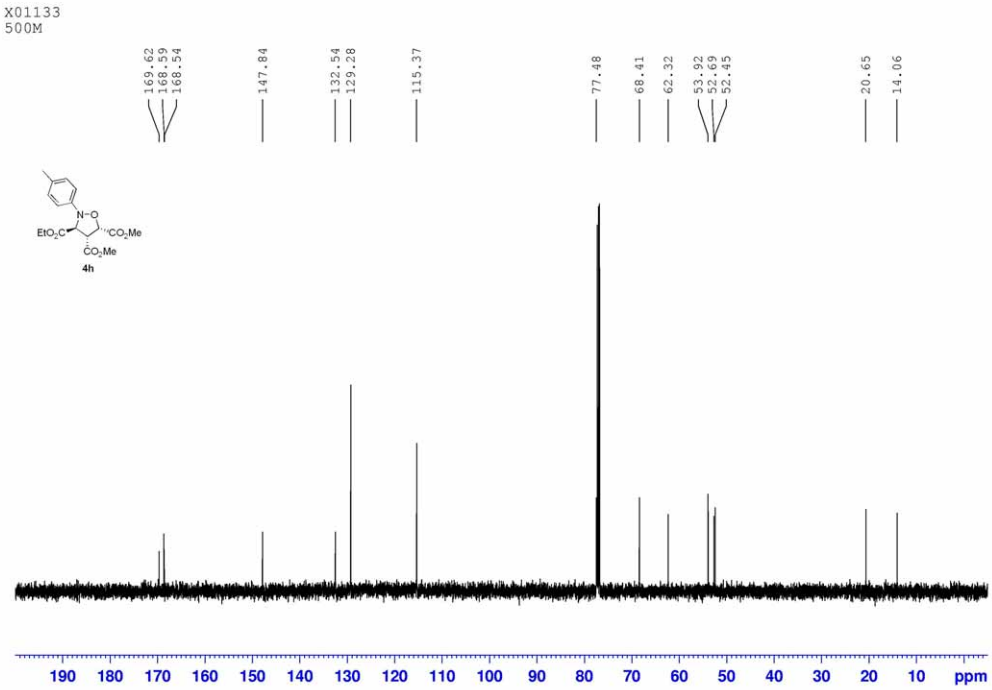

x0279 500M
7.966
7.949
7.128
7.111
5.039
5.025
4.890
4.876
4.388
4.373
4.359
4.341
4.335
4.327
4.321
4.313
4.306
4.299
4.292
3.875
3.688
3.506
1.339
1.325
1.311
10 9 8 7 6 5 4 3 2 1 0 ppm
2.03
2.05
1.00
1.01
1.01
2.06
3.05
3.02
3.02
3.06

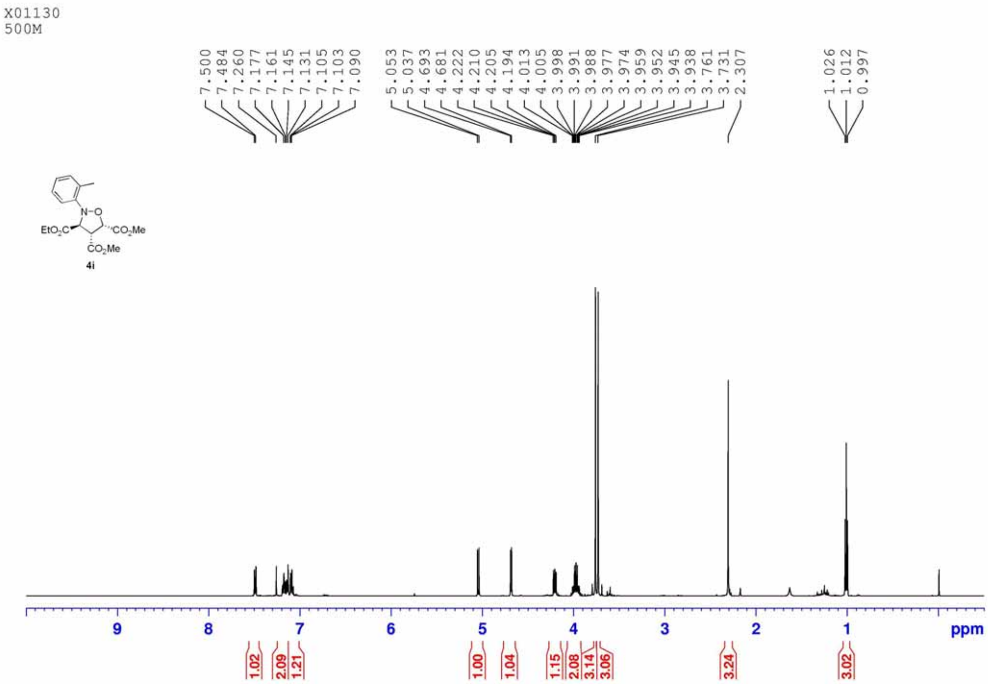

x0279 500M 13C
169.32
168.14
168.04
166.77
154.01
130.69
123.44
112.82
77.76
66.96
62.66
53.62
52.81
52.56
51.85
14.05
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

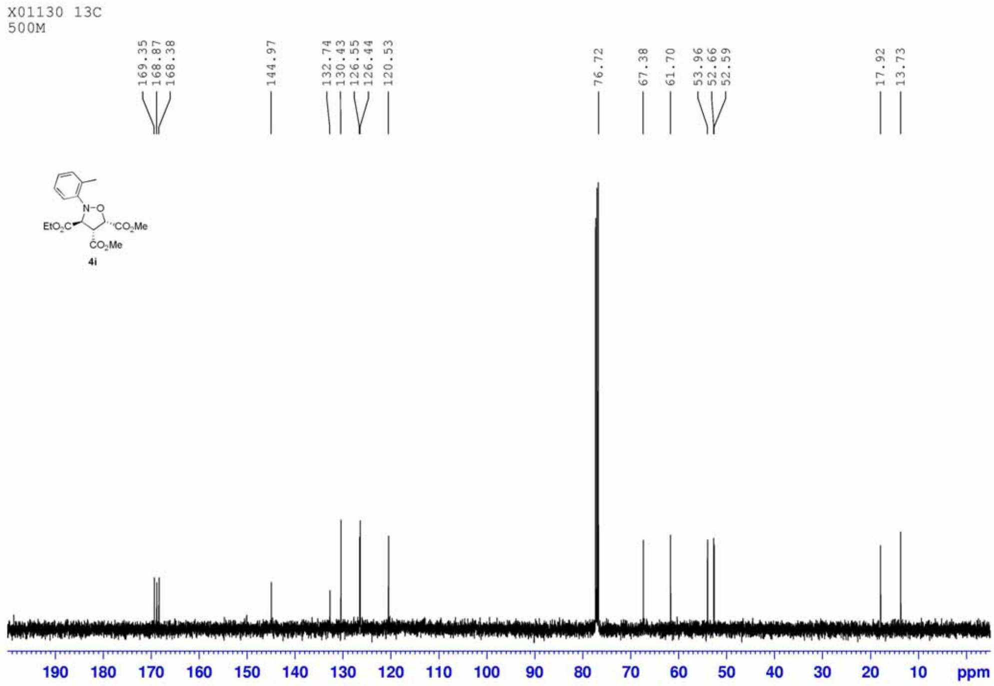

XZJ1124 500
7.950
7.932
7.103
7.085
5.012
4.997
4.864
4.851
4.348
4.337
4.333
4.323
4.309
4.299
4.295
4.291
4.285
3.668
3.488
1.366
1.351
1.337
1.319
1.305
1.291
4g
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.08
2.00
0.97
1.05
5.17
2.99
2.98
3.13
3.17

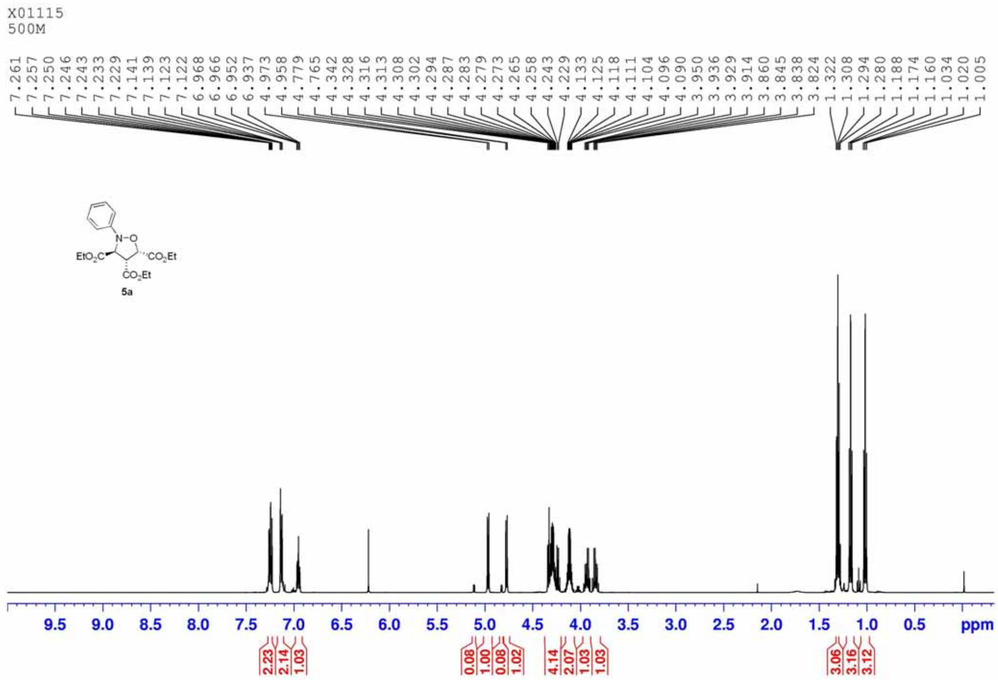

XZJ1124 C13 500
169.36
168.17
168.06
166.34
153.95
130.67
123.84
112.82
77.77
67.01
62.67
60.66
53.66
52.82
52.59
14.38
14.07
4g
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

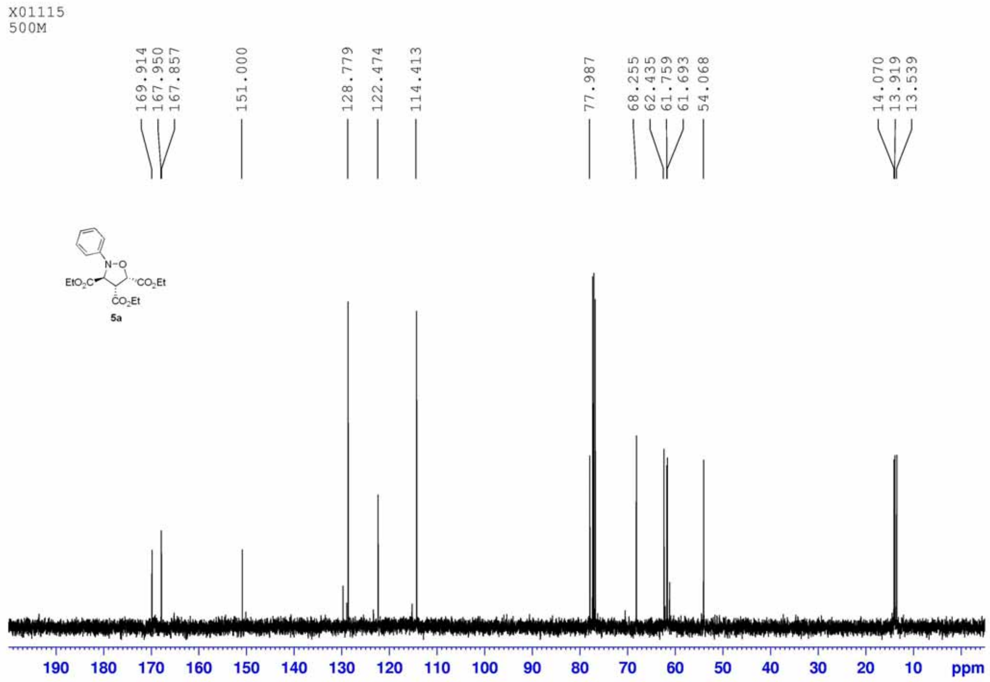

X01133
500M
7.111
7.090
7.072
5.027
5.012
4.753
4.739
4.328
4.319
4.313
4.305
4.299
4.290
4.285
4.276
4.271
4.264
3.704
3.586
2.300
1.329
1.315
1.301
4h
9 8 7 6 5 4 3 2 1 ppm
4.11
1.00
1.00
3.13
3.11
3.09
3.13
3.23

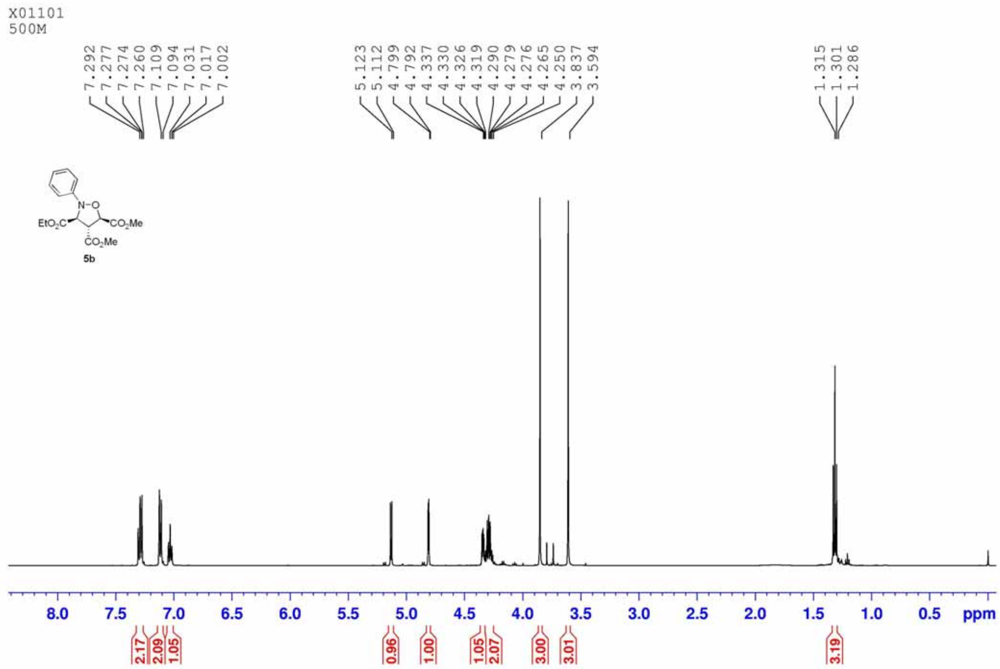

X01133
500M
169.62
168.59
168.54
147.84
132.54
129.28
115.37
77.48
68.41
62.32
53.92
52.69
52.45
20.65
14.06
EtO2C
CO2Me
CO2Me
4h
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

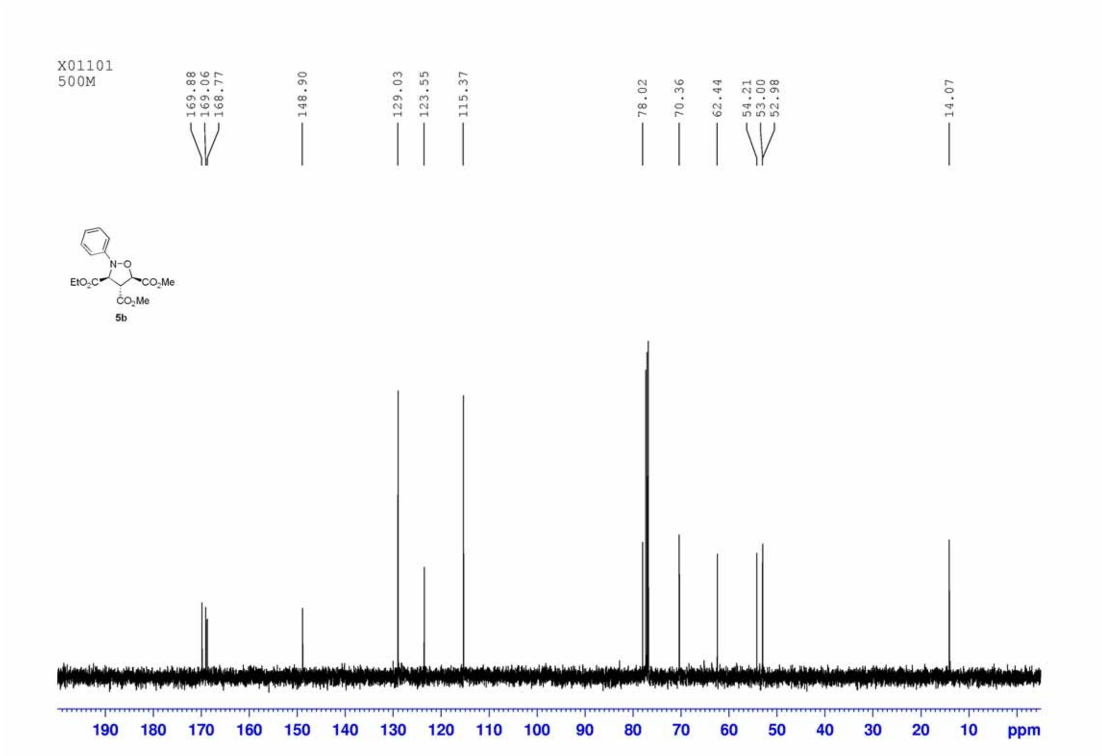

X01130
500M
7.500
7.484
7.260
7.177
7.161
7.145
7.131
7.105
7.103
7.090
5.053
5.037
4.693
4.681
4.222
4.210
4.205
4.194
4.013
4.005
3.998
3.991
3.988
3.977
3.974
3.959
3.952
3.945
3.938
3.761
3.731
2.307
1.026
1.012
0.997
EtO2C
CO2Me
CO2Me
4i
9 8 7 6 5 4 3 2 1 ppm
1.02
2.09
1.21
1.00
1.04
1.15
2.08
3.14
3.06
3.24
3.02

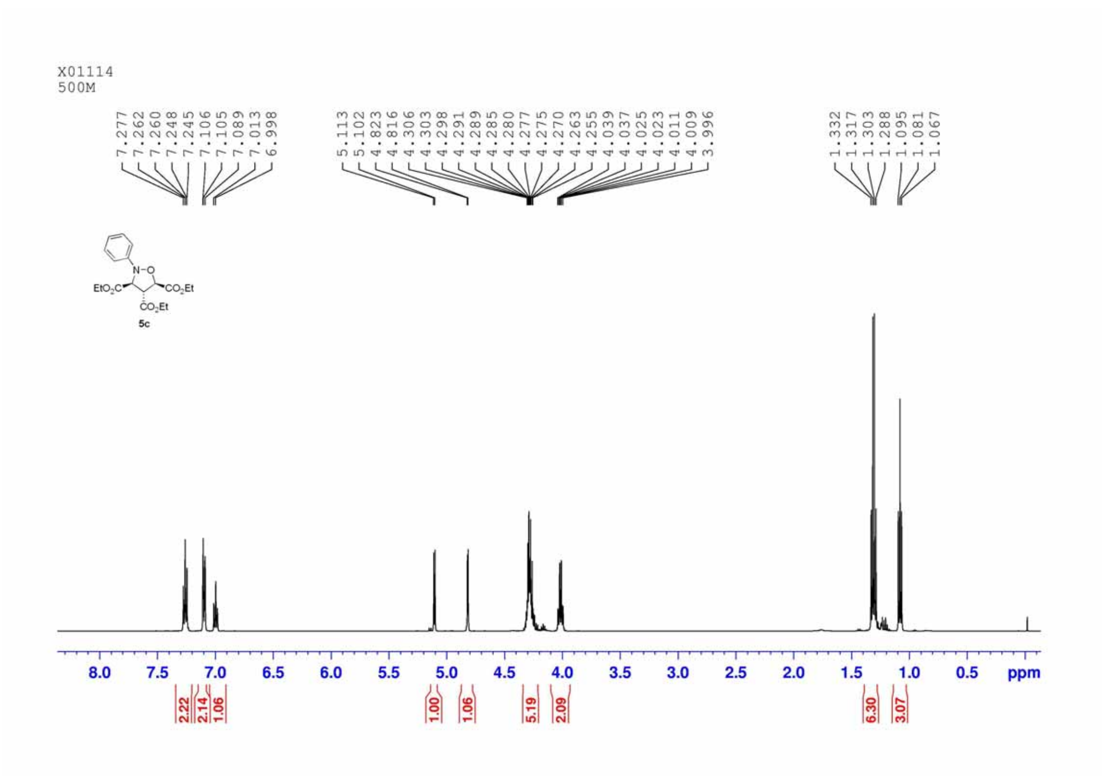

X01130 13C
500M
169.35
168.87
168.38
144.97
132.74
130.43
126.55
126.44
120.53
76.72
67.38
61.70
53.96
52.66
52.59
17.92
13.73
EtO₂C
CO₂Me
CO₂Me
4i
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

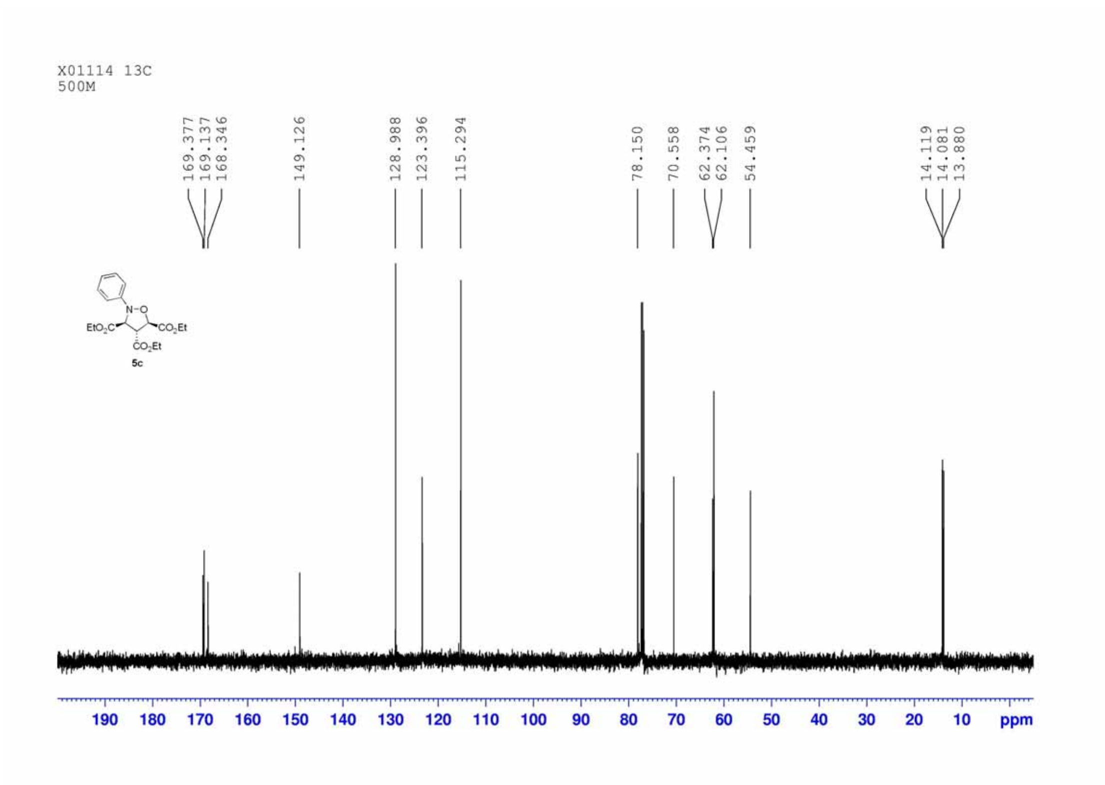

X01115
500M
7.261
7.257
7.250
7.246
7.243
7.233
7.229
7.141
7.139
7.123
7.122
6.968
6.966
6.952
6.937
4.973
4.958
4.779
4.765
4.342
4.328
4.316
4.313
4.308
4.302
4.294
4.287
4.283
4.279
4.273
4.265
4.258
4.243
4.229
4.133
4.125
4.118
4.111
4.104
4.096
4.090
3.950
3.936
3.929
3.914
3.860
3.845
3.838
3.824
1.322
1.308
1.294
1.280
1.188
1.174
1.160
1.034
1.020
1.005
EtO₂C
CO₂Et
CO₂Et
5a
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 ppm
2.23
2.14
1.03
0.08
1.00
0.08
1.02
4.14
2.07
1.03
1.03
3.06
3.16
3.12

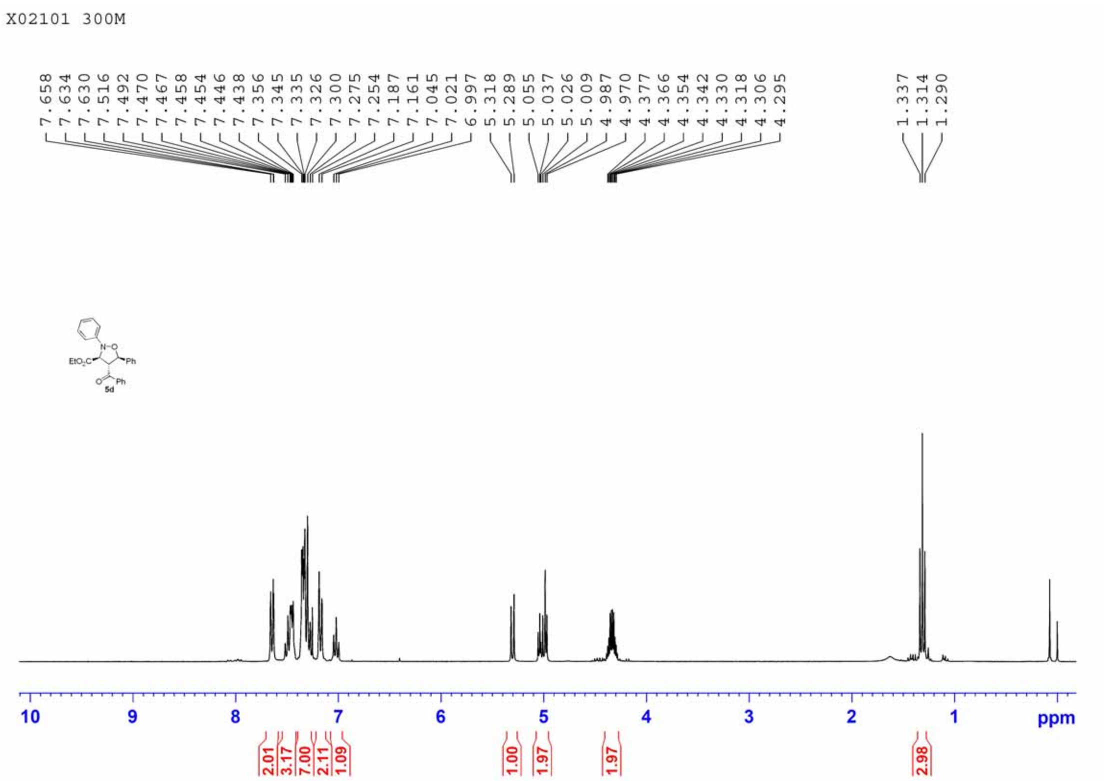

X01115
500M
169.914
167.950
167.857
151.000
128.779
122.474
114.413
77.987
68.255
62.435
61.759
61.693
54.068
14.070
13.919
13.539
5a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


X01101
500M
7.292
7.277
7.274
7.260
7.109
7.094
7.031
7.017
7.002
5.123
5.112
4.799
4.792
4.337
4.330
4.326
4.319
4.290
4.279
4.276
4.265
4.250
3.837
3.594
1.315
1.301
1.286
5b
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 ppm
2.17
2.09
1.05
0.96
1.00
1.05
2.07
3.00
3.01
3.19

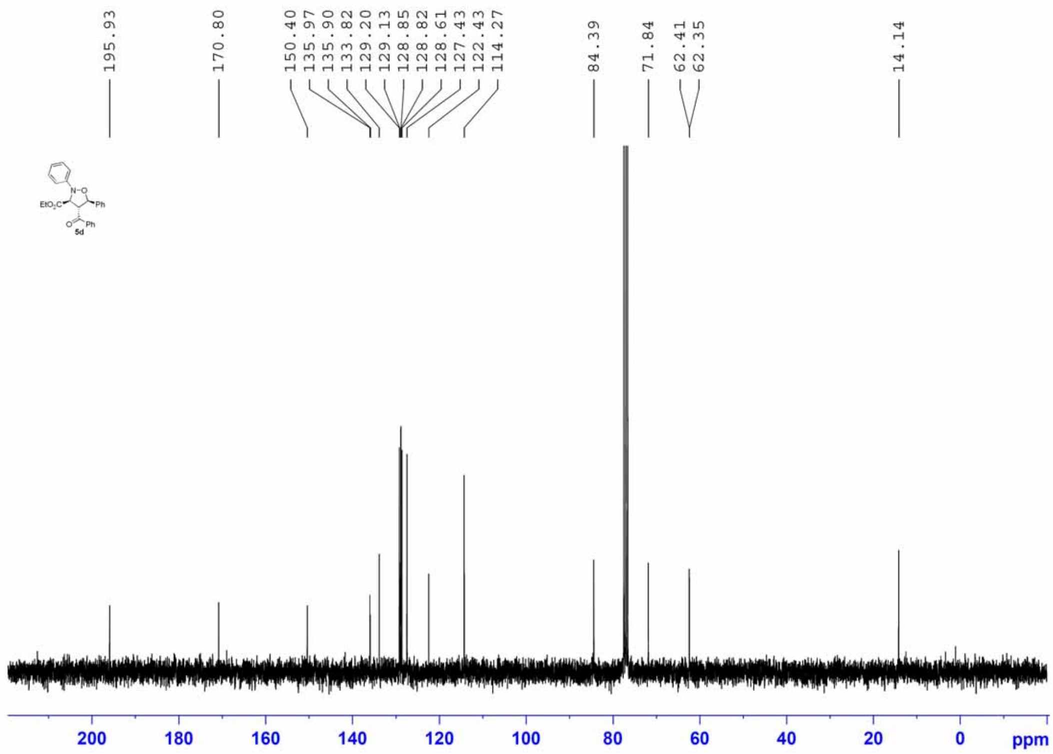

X01101
500M
169.88
169.06
168.77
148.90
129.03
123.55
115.37
78.02
70.36
62.44
54.21
53.00
52.98
14.07
EtO_2C
CO_2Me
CO_2Me
5b
ppm


X01114
500M
7.277
7.262
7.260
7.248
7.245
7.106
7.105
7.089
7.013
6.998
5.113
5.102
4.823
4.816
4.306
4.303
4.298
4.291
4.289
4.285
4.280
4.277
4.275
4.270
4.263
4.255
4.039
4.037
4.025
4.023
4.011
4.009
3.996
1.332
1.317
1.303
1.288
1.095
1.081
1.067
EtO_2C
CO_2Et
CO_2Et
5c
2.22
2.14
1.06
1.00
1.06
5.19
2.09
6.30
3.07
ppm

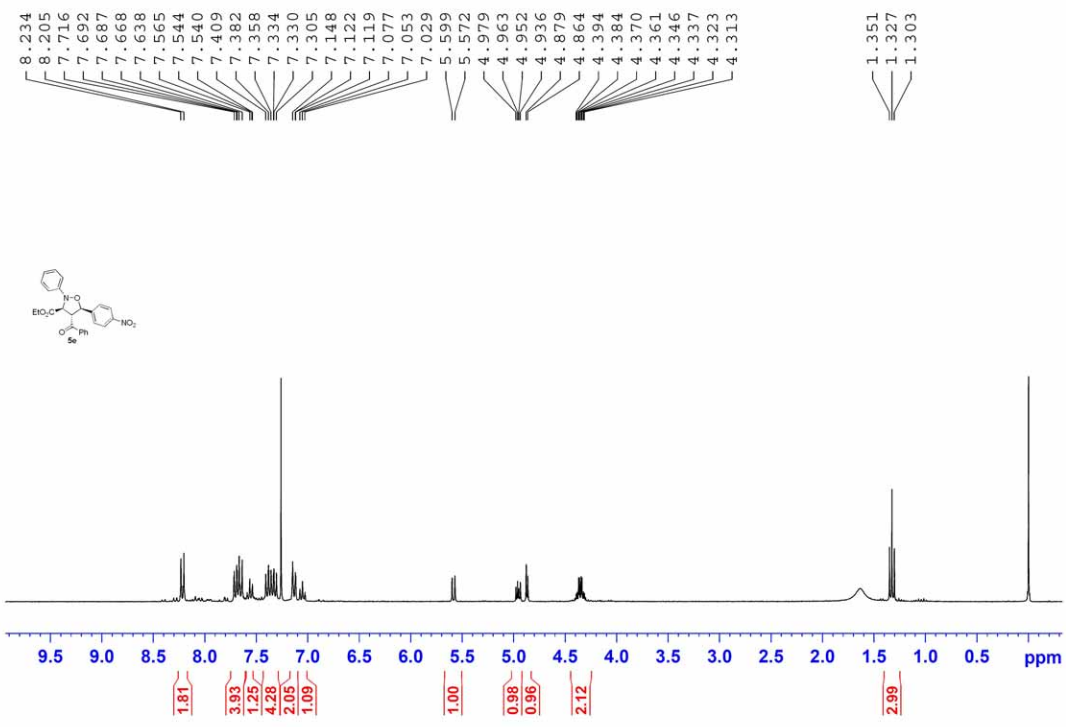

X01114 13C
500M
169.377
169.137
168.346
149.126
128.988
123.396
115.294
78.150
70.558
62.374
62.106
54.459
14.119
14.081
13.880
5c
ppm

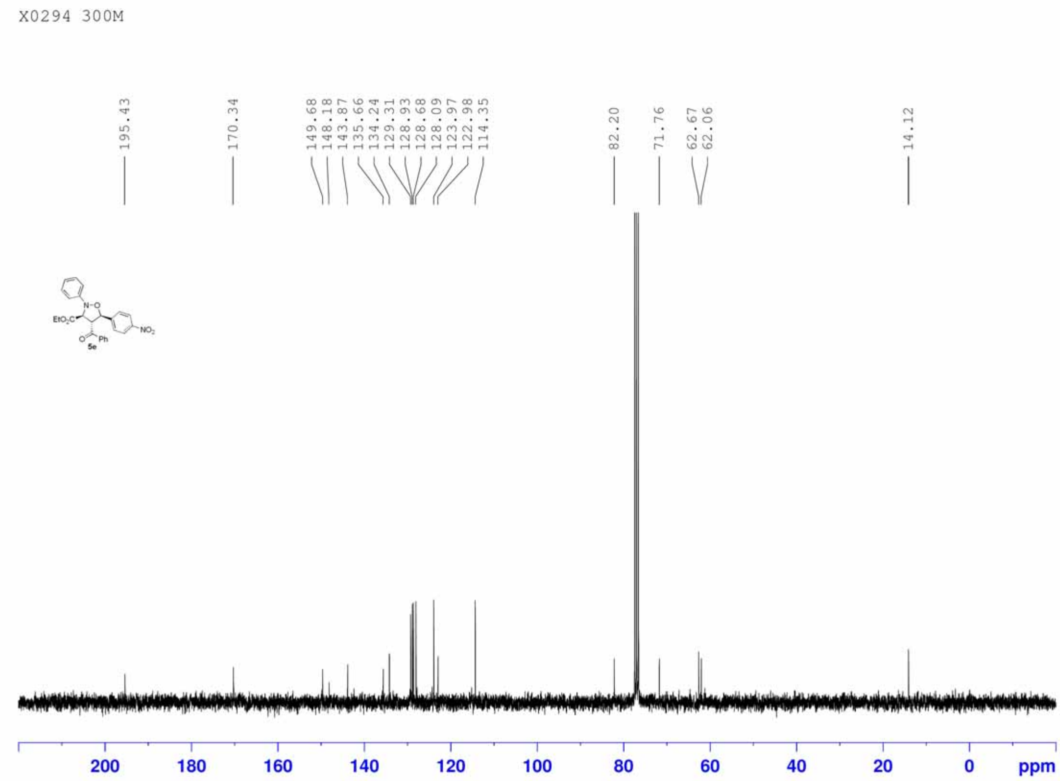

X02101 300M
7.658
7.634
7.630
7.516
7.492
7.470
7.467
7.458
7.454
7.446
7.438
7.356
7.345
7.335
7.326
7.300
7.275
7.254
7.187
7.161
7.045
7.021
6.997
5.318
5.289
5.055
5.037
5.026
5.009
4.987
4.970
4.377
4.366
4.354
4.342
4.330
4.318
4.306
4.295
1.337
1.314
1.290
5d
ppm
2.01
3.17
7.00
2.11
1.09
1.00
1.97
1.97
2.98

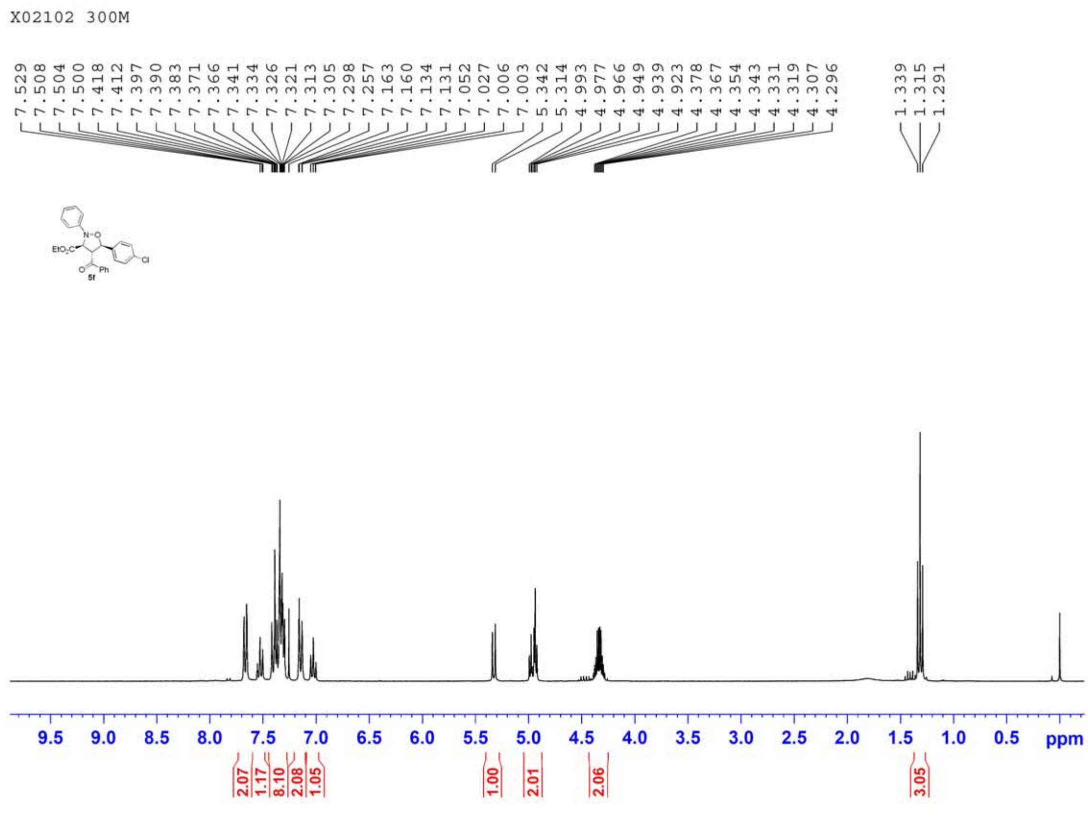
X02101 300M 13C

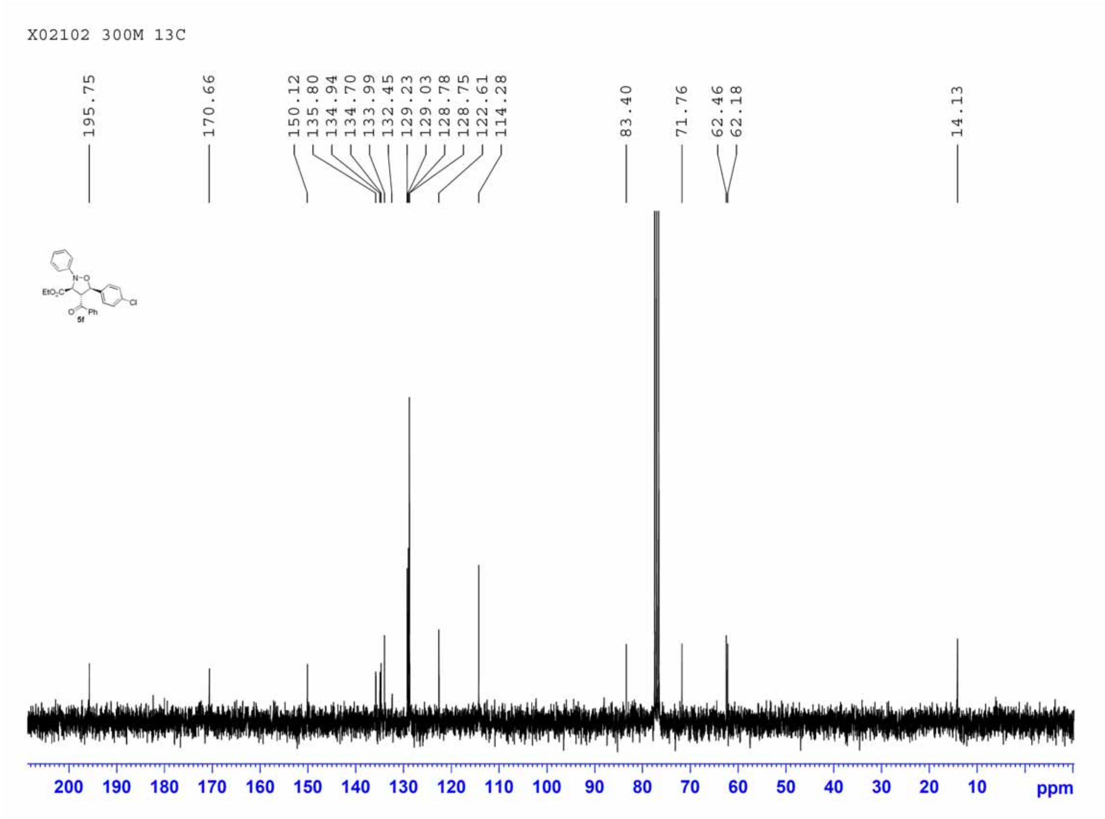


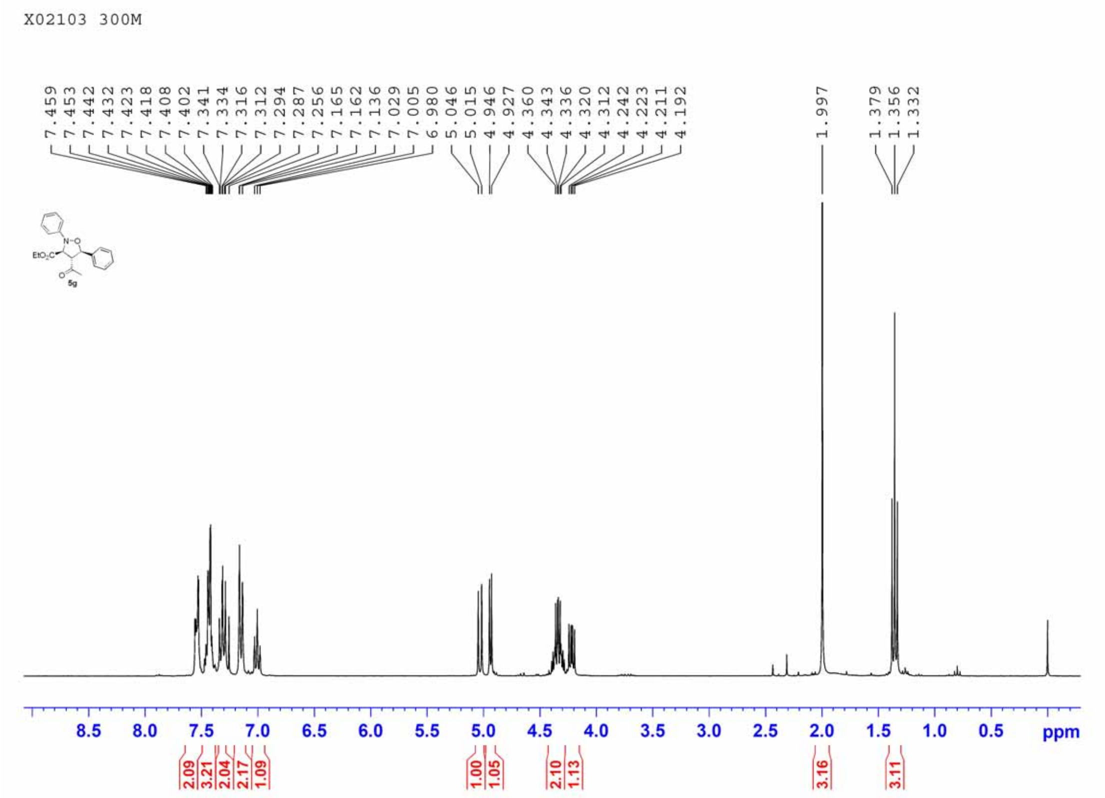
X0294 300M




X0294 300M
195.43
170.34
149.68
148.18
143.87
135.66
134.24
129.31
128.93
128.68
128.09
123.97
122.98
114.35
82.20
71.76
62.67
62.06
14.12
EtO2C
Ph
NO2
5e
200
180
160
140
120
100
80
60
40
20
0
ppm


X02102 300M
7.529
7.508
7.504
7.500
7.418
7.412
7.397
7.390
7.383
7.371
7.366
7.341
7.334
7.326
7.321
7.313
7.305
7.298
7.257
7.163
7.160
7.134
7.131
7.052
7.027
7.006
7.003
5.342
5.314
4.993
4.977
4.966
4.949
4.939
4.923
4.378
4.367
4.354
4.343
4.331
4.319
4.307
4.296
1.339
1.315
1.291
EtO2C
Ph
Cl
5f
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
ppm
2.07
1.17
8.10
2.08
1.05
1.00
2.01
2.06
3.05


X02102 300M 13C
195.75
170.66
150.12
135.80
134.94
134.70
133.99
132.45
129.23
129.03
128.78
128.75
122.61
114.28
83.40
71.76
62.46
62.18
14.13
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


X02103 300M
7.459
7.453
7.442
7.432
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7.341
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7.316
7.312
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7.287
7.256
7.165
7.162
7.136
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7.005
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5.046
5.015
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4.927
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4.343
4.336
4.320
4.312
4.242
4.223
4.211
4.192
1.997
1.379
1.356
1.332
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 ppm
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1.05
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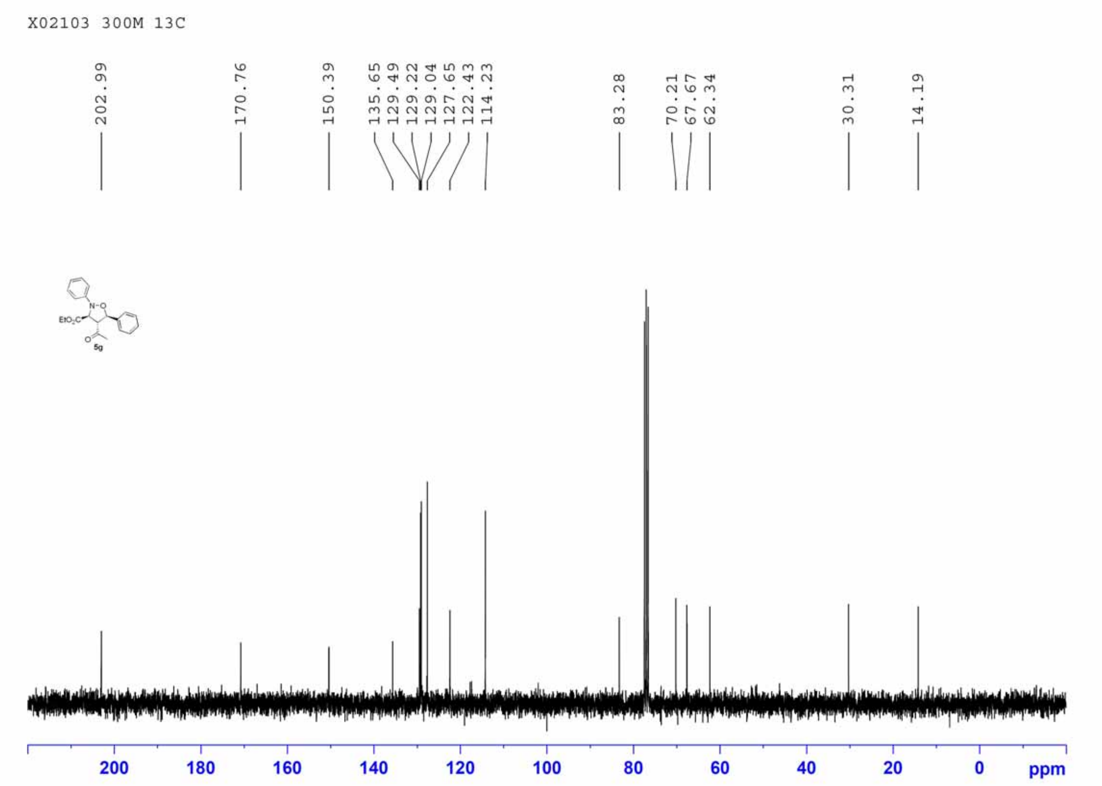
3.11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83.28
70.21
67.67
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14.19
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160
140
120
100
80
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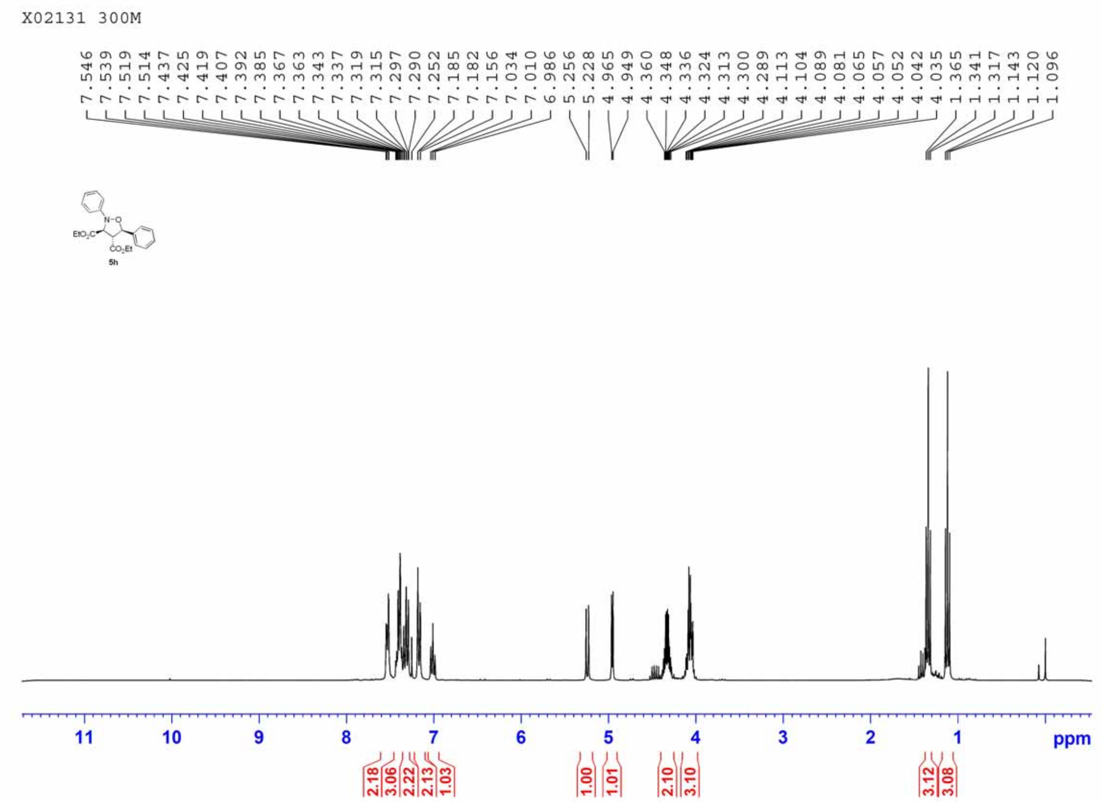
40
20
0
ppm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7.343
7.337
7.319
7.315
7.297
7.290
7.252
7.185
7.182
7.156
7.034
7.010
6.986
5.256
5.228
4.965
4.949
4.360
4.348
4.336
4.324
4.313
4.300
4.289
4.113
4.104
4.089
4.081
4.065
4.057
4.052
4.042
4.035
1.365
1.341
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1.143
1.120
1.096
5h
11
10
9
8
7
6
5
4
3
2
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2.13
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1.01
2.10
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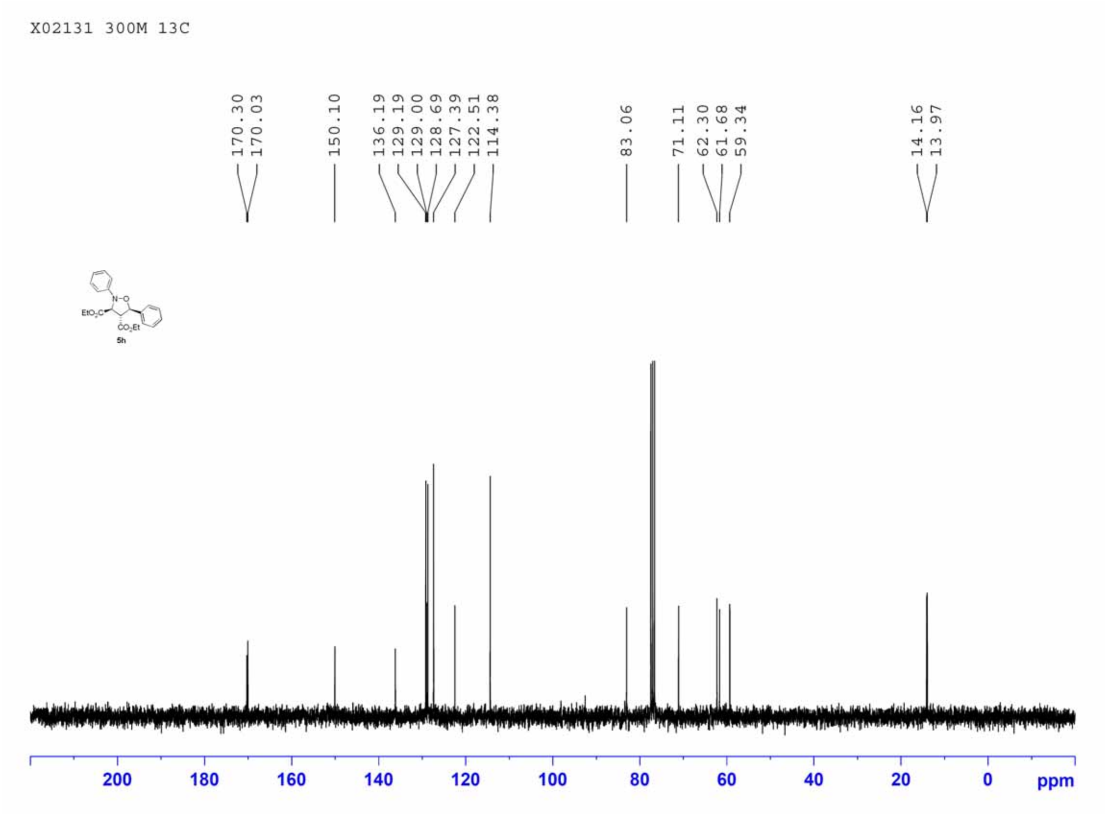
3.08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71.11
62.30
61.68
59.34
14.16
13.97
5h
200
180
160
140
120
100
80
60
40
20
0
ppm