Passerini/Tsuji-Trost strategies towards lactams and cyclopentane derivatives.

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Experimental part:

NMR spectra were recorded on a 400 MHz spectrometer, using deuterated solvent as reference and/or internal deuterium lock. Two-dimensional NMR spectroscopy [^{1}H -^{1}H COSY spectra, ^{1}H -^{13}C COSY spectra (HSQC) and long-range ^{1}H -^{13}C COSY spectra (HMBC)], were carried out to determine the correlation between ^{1}H and ^{13}C. The chemical shifts for all NMR spectra are expressed in parts per million to high frequency of TMS reference. Coupling constants (*J*) are quoted in Hz and are recorded to the nearest 0.1 Hz.

The IR spectra were obtained using ATR accessories. High-resolution (HR) mass spectra were performed on a GC/MS system spectrometer. TLC was carried out using precoated plates of silica gel $60F_{254}$.

General procedure A for Passerini reactions :
A mixture of aldehyde (1.0 equiv), acetic acid (1.0 equiv) and isocyanide (1.0 equiv) was stirred at room temperature for 2 days. The crude was purified by flash chromatography on silica gel.

General procedure Abis for Passerini reactions :
A mixture of aldehyde (1.0 equiv), acetic acid (1.0 equiv) and isocyanide (1.0 equiv) was stirred at 40°C for 2 days. The crude was purified by flash chromatography on silica gel.

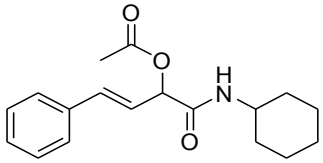
General procedure B for Tsuji-Trost reactions:
To a 0.25 M solution of Passerini adduct **1** (1.0 equiv.) in toluene were added dimethyl malonate (1.0 equiv), Cs_2CO_3 (1.0 equiv) and $Pd(PPh_3)_4$ (5 mol %). The resulting mixture was stirred at 50°C during 30 minutes. The solvent was removed afterwards under reduced pressure and the crude was purified by flash chromatography on silica gel.

General procedure C for cyclisation reactions:
To a 0.2 M solution of Tsuji-Trost adduct **2** (1.0 eq) in methanol was added Cs_2CO_3 (0.5eq). The resulting mixture was heated under microwave at 100°C during 30 minutes. The solvent was removed afterwards under reduced pressure. The crude was diluted with dichloromethane and the organic phase was washed with water acidified by citric acid. After drying the organic phase with $MgSO_4$, the solvent was removed under reduced pressure. The crude was purified by flash chromatography on silica gel.

General procedure D for Tsuji-Trost reaction followed by cyclisation for bis-nucleophiles :
To a 0.25 M solution of Passerini adduct **1** (1.0 eq) in toluene were added the bis-nucleophile **4** (1.0 eq), Cs_2CO_3 (1.2 eq) and $Pd(PPh_3)_4$ (5 mol%). The resulting mixture was stirred at 50°C during 30 minutes. The solvent was removed afterwards under reduced pressure and the crude was purified by flash chromatography on silica gel.

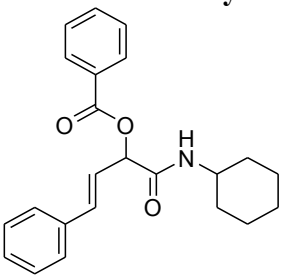
Passerini produtcs :

Acetic acid 1-cyclohexylcarbamoyl-3-phenyl-allyl ester (1a)



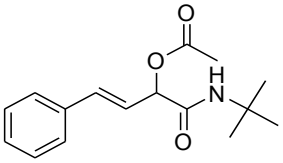
Compound **1a** was prepared according to general procedure A. Purification by flash chromatohraphy with a gradient Et_2O/EP (30/70 to 100/0) gave the desired product (567 mg, 94%) as a white solid. **CCM** Rf (80/20 Et_2O/EP) = 0.36 ; **Mp** 129 – 130°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.38 (d, J=7.6Hz, 2H), 7.31 (m, 3H), 6.72 (d, J=16.0 Hz, 1H), 6.26 (dd, J=16.0, 6.8Hz, 1H), 5.92 (d, J=7.6Hz, 1H), 5.70 (d, J=6.8Hz, 1H), 3.80 (m, 1H), 2.20 (s, 3H), 1.91 (m, 2H), 1.70 (m, 2H), 1.61 (m, 2H), 1.35 (m, 2H), 1.18 (m, 2H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 169.2 (Cq), 167.1 (Cq), 135.7 (CH), 134.7 (Cq), 128.6 (2CH), 128.4 (CH), 126.9 (2CH), 122.7 (CH), 74.5 (CH), 48.3 (CH), 33.0 ($2CH_2$), 25.5 (CH_2), 24.81($2CH_2$), 21.12 (Me) ; **IR** (υ, cm^{-1}) 3285, 2930, 2854, 1742, 1658, 1541, 1449, 1370, 1230, 1031 ; **HRMS** (EI) Calcd. for $C_{18}H_{23}NO_3$: 301.1678 found : 301.1681

Benzoic acid 1-cyclohexylcarbamoyl-3-phenyl-allyl ester (1b)



Compound **1b** was prepared according to general procedure Abis. Purification by flash chromatography with a gradient Et_2O/EP (30/70 to 100/0) gave the desired product (880 mg, 81%) as a white solid. **CCM** Rf (80/20 Et_2O/EP) = 0.75 ; **Mp** 180°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 8.12 (d, J= 7.6 Hz, 2H), 7.63 (t, J=7.2Hz, 1H), 7.50 (t, J=6.4Hz, 2H), 7.41 (d, J=8.0Hz, 2H), 7.32 (t, J=7.2Hz, 2H), 7.27 (d, J=7.6Hz, 1H), 6.81 (d, J=15.6Hz, 1H), 6.42 (dd, J=15.6, 6.8Hz, 1H), 6.02 (m, 1H), 5.99 (d, J=6.8Hz, 1H), 3.83 (m, 1H), 1.94 (m, 2H), 1.69 (m, 2H), 1.59 (m, 2H), 1.36 (m, 2H), 1.18 (m, 2H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 167.2 (Cq), 165.0 (Cq), 135.7 (Cq), 134.6 (CH), 133.7 (CH), 129.8 (2CH), 129.3 (Cq), 128.7 (2CH), 128.6 (2CH), 128.4 (CH), 126.9 (2CH), 122.7 (CH), 74.9 (CH), 48.3 (CH), 33.0 ($2CH_2$), 25.5 (CH_2), 24.8 (CH_2), 24.7 (CH_2) ; **IR** (υ, cm^{-1}) 3299, 2930, 2854, 1722, 1660, 1541, 1450, 1264, 1109, 1069, 1026 **HRMS** (EI) Calcd. for $C_{23}H_{25}NO_3$: 363.1834 found : 363.1834

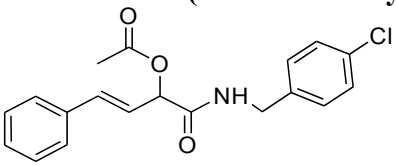
Acetic acid 1-tert-butylcarbamoyl-3-phenyl-allyl ester (1c)



Compound **1c** was prepared according to general procedure A. Purification by flash chromatography with a gradient Et_2O/EP (10/90 to 30/70) gave the desired product (606 mg, 73%) as a white solid. **CCM** Rf (80/20 Et_2O/EP) = 0.55 ; **Mp** 116 – 118°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.44 (d, J=7.6Hz, 2H), 7.37 (m, 3H), 6.76 (d, J=16.0Hz, 1H), 6.31 (dd, J=16.0, 6.8Hz, 1H), 5.90 (br s, 1H), 5.67 (d, J=6.8Hz, 1H), 2.24 (s, 3H), 1.42 (s, 9H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$,

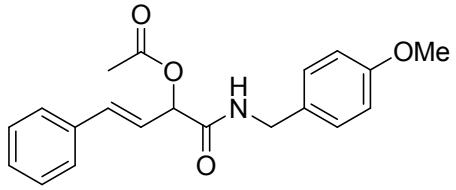
100.6 MHz) 169.2 (Cq), 167.1 (Cq), 135.7 (Cq), 134.7 (CH), 128.6 (2CH), 128.4 (CH), 126.8 (2CH), 122.8 (CH), 74.7 (CH), 51.5 (Cq), 28.7 ($3CH_3$), 21.1 (CH_3) ; **IR** (υ, cm^{-1}) 3306, 2968, 1741, 1665, 1541, 1451, 1366, 1227, 1033 ; **HRMS** (EI) Calcd. for $C_{16}H_{21}NO_3$: 275.1521 found : 275.1517

Acetic acid 1-(4-chloro-benzylcarbamoyl)-3-phenyl-allyl ester (1d)



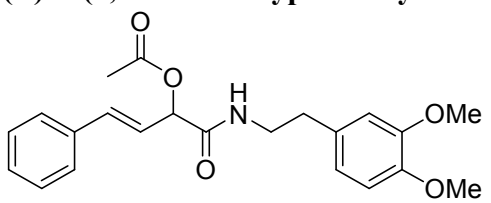
Compound **1d** was prepared according to general procedure A. Purification by flash chromatography with a gradient Et2O/EP (30/70 to 100/0) gave the desired product (621 mg, 90%) as a cream solid. **CCM** Rf (80/20 Et2O/EP) = 0.27 ; **Mp** 103-104°C ; **1H-NMR (δ, ppm)** (CDCl3, 400 MHz) 7.37 (d, J=7.6Hz, 2H), 7.30 (m, 5H), 7.21 (d, J=8.0Hz, 2H), 6.73 (d, J=15.6Hz, 1H), 6.42 (br s, 1H), 6.27 (dd, J=15.6, 6.8Hz, 1H), 5.75 (d, J=7.2Hz, 1H), 4.43 (m, 2H), 2.19 (s, 3H) ; **13C-NMR (δ, ppm)** (CDCl3, 100.6 MHz) 169.3 (Cq), 168.2 (Cq), 136.2 (Cq), 135.4 (Cq), 135.1 (CH), 133.5 (Cq), 129.0 (2CH), 128.9 (2CH), 128.6 (CH), 128.5 (2CH), 126.8 (2CH), 122.1 (CH), 74.5 (CH), 42.6 (CH2), 21.0 (CH3) ; **IR** (υ, cm-1) 3288, 3062, 2927, 1740, 1661, 1538, 1491, 1370, 1228, 1090, 1015 ; **HRMS** (EI) Calcd. for C19H18ClNO3 : 343.0975 found : 343.0979

(*E*)-1-((4-methoxybenzyl)amino)-1-oxo-4-phenylbut-3-en-2-yl acetate (1e)



Compound **1e** was prepared according to the general procedure A. Purification by flash chromatography with a gradient PE/AcOEt (from 70/30 to 50/50) as eluant gave the desired product (881 mg, 87%) as an yellow solid. **Mp** 115 – 116°C; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.39-7.37 (m, 2H), 7.34-7.27 (m, 3H), 7.21 (d, *J* = 8.6 Hz, 2H), 6.87 (d, *J* = 8.6 Hz, 2H), 6.73 (d, *J* = 15.9 Hz, 1H), 6.36 (br s, 1H), 6.29 (dd, *J* = 15.9, 6.8 Hz, 1H), 5.77 (dd, *J* = 6.8, 1.3 Hz, 1H), 4.45 (dd, *J* = 14.6, 5.6 Hz, 1H), 4.41 (dd, *J* = 14.6, 5.6 Hz, 1H), 3.79 (s, 3H), 2.17 (s, 3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 169.2, 167.9, 159.1, 135.5, 134.9, 129.6, 129.1, 128.6, 128.4, 126.8, 122.3, 114.1, 74.5, 55.3, 42.9, 21.0 ; **IR** (υ, cm^{-1}) 3296, 2934, 1742, 1662, 1613, 1513, 1450, 1371, 1301, 1231, 1177, 1111, 1070, 1033 ; **HRMS** (EI) Calcd. for $C_{10}H_{21}NO_4$: 339.1471 found : 339.1479

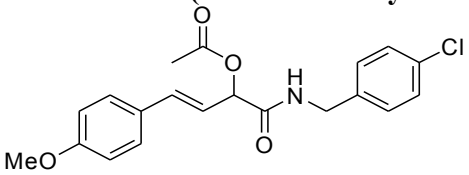
(*E*)-1-(3,4-dimethoxyphenethylamino)-1-oxo-4-phenylbut-3-en-2-yl acetate (1f)



Compound **1f** was prepared according to the general procedure A. Purification by flash chromatography with a gradient PE/Et_2O (from 30/70 to 0/100) as eluant gave the desired product (580 mg, 76%) as an yellow solid. **Mp** 115 – 116°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.38-7.28 (m, 5H), 6.76-6.67 (m, 4H), 6.23 (dd, *J* = 15.9, 6.8 Hz, 1H), 6.05 (br s, 1H), 5.70 (dd, *J* = 6.8, 1.0 Hz, 1H), 3.84 (s, 3H), 3.83 (s, 3H), 3.61-3.47 (m, 2H), 2.79 (dt, *J* = 6.6, 2.8 Hz, 2H), 2.13 (s, 3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 169.1, 168.0, 149.0, 147.7, 135.5, 134.7, 131.0,

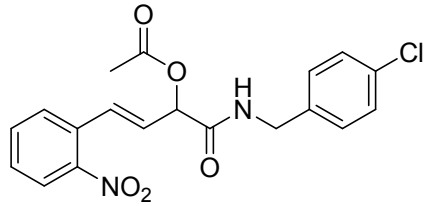
128.6, 128.4, 126.8, 122.4, 120.7, 111.9, 111.2, 74.4, 55.9, 55.8, 40.5, 35.0, 20.9 ; **IR** (υ, cm^{-1}) 3304, 2937, 1745, 1666, 1515, 1453, 1371, 1261, 1231, 1158, 1141, 1025 ; **HRMS** (EI) Calcd. for $C_{22}H_{25}NO_5$: 383.1733 found : 383.1731

Acetic acid 1-(4-chloro-benzylcarbamoyl)-3-(4-methoxy-phenyl)-allyl ester (1g)



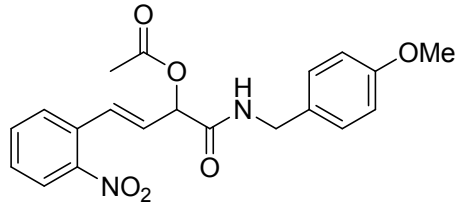
Compound **1g** was prepared according to general procedure A. Purification by flash chromatohraphy with a gradient Et_2O/EP (30/70 to 100/0) gave the desired product (734 mg, 65%) as a cream solid. **CCM** Rf (80/20 Et_2O/EP) = 0.31 ; **Mp** 92 – 94°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.30 (m, 5H), 7.21 (m, 3H), 6.85 (d, J=8.8Hz, 2H), 6.69 (d, J=15.6Hz, 1H), 6.41 (br s, 1H), 6.12 (dd, J=16.0, 7.2Hz, 1H), 5.72 (d, J=7.2Hz, 1H), 4.44 (m, 2H), 3.81 (s, 3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 169.9 (Cq), 169.0 (Cq), 161.53 (Cq), 160.4 (Cq), 136.7 (Cq), 135.5 (CH), 133.9 (Cq), 129.6 (2CH), 129.5 (2CH), 129.3 (2CH), 128.6 (2CH), 120.2 (CH), 114.5 (2CH), 75.2 (CH), 55.7 (OMe), 43.1 (CH_2), 21.4 (Me) ; **IR** (υ, cm^{-1}) 3275, 3048, 2931, 2836, 1739, 1656, 1605, 1509, 1490, 1370, 1221, 1173, 1088, 1013 ; **HRMS** (EI) Calcd. for $C_{20}H_{20}ClNO_4$: 373.1081 found : 373.1085

(*E*)-1-((4-chlorobenzyl)amino)-4-(2-nitrophenyl)-1-oxobut-3-en-2-yl acetate (1h)



Compound **1h** was prepared according to the general procedure A. Purification by flash chromatography with a gradient PE/AcOEt (from 70/30 to 50/50) as eluant gave the desired product (763 mg, 65%) as an yellow solid. **Mp** 106 – 107°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.99 (d, *J* = 8.1 Hz, 1H), 7.60-7.59 (m, 2H), 7.47-7.43 (m, 1H), 7.31 (d, *J* = 8.1 Hz, 2H), 7.23-7.17 (m, 3H), 6.50 (t, *J* = 5.8 Hz, 1H), 6.36 (dd, *J* = 15.9, 5.8 Hz, 1H), 5.85 (dd, *J* = 5.8, 1.3 Hz, 1H), 4.50 (dd, *J* = 14.9, 5.8 Hz, 1H), 4.45 (dd, *J* = 14.9, 5.8 Hz, 1H), 2.23 (s, 3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 169.1, 167.5, 147.7, 136.0, 133.5, 133.4, 131.6, 129.0, 129.0, 128.9, 128.9, 128.9, 127.9, 124.7, 73.6, 42.7, 20.9 ; **IR** (υ, cm^{-1}) 3294, 2934, 1745, 1665, 1523, 1492, 1345, 1227, 1091, 1016 ; **HRMS** (EI) Calcd. for $C_{19}H_{17}ClN_2O_5$: 388.0826 found : 388.0824

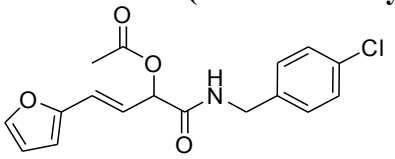
(*E*)-1-((4-methoxybenzyl)amino)-4-(2-nitrophenyl)-1-oxobut-3-en-2-yl acetate (1i)



Compound **1i** was prepared according to the general procedure A. Purification by flash chromatography with a gradient PE/AcOEt (from 50/50 to 40/60) as eluant gave the desired product (660 mg, 57%) as an yellow solid. **Mp** 119 – 120°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.97 (d, *J* = 8.1 Hz, 1H), 7.60-7.58 (m, 2H), 7.46-7.41 (m, 1H), 7.22 (d, *J* = 8.8 Hz, 2H), 7.18 (dd, *J* = 15.9, 1.5 Hz, 1H), 6.87 (d, *J* = 8.8 Hz, 2H), 6.43 (t, *J* = 5.5 Hz, 1H), 6.38 (dd, *J* = 15.9, 5.8 Hz, 1H),

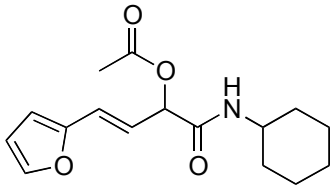
5.85 (dd, *J* = 5.8, 1.5 Hz, 1H), 4.46 (dd, *J* = 14.9, 5.5 Hz, 1H), 4.41 (dd, *J* = 14.9, 5.5 Hz, 1H), 3.79 (s, 3H), 2.21 (s, 3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 169.0, 167.3, 159.1, 147.7, 133.4, 131.7, 129.5, 129.1, 128.9, 128.8, 128.6, 128.1, 124.7, 114.2, 73.5, 55.3, 42.9, 20.9 ; **IR** (υ, cm^{-1}) 3299, 2936, 1744, 1667, 1517, 1346, 1234, 1175, 1036 ; **HRMS** (EI) Calcd. for $C_{20}H_{20}N_2O_6$: 384.1321 found : 384.1327

Acetic acid 1-(4-chloro-benzylcarbamoyl)-3-furan-2-yl-allyl ester (1j)



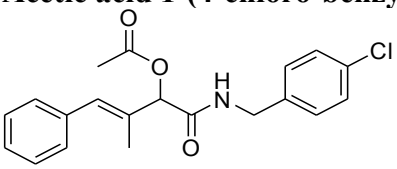
Compound **1j** was prepared according to general procedure Abis. Purification by flash chromatohraphy with a gradient Et_2O/EP (25/75 to 75/25) gave the desired product (451 mg, 62%) as a brown solid. **CCM** Rf (80/20 Et_2O/EP) = 0.44 ; **Mp** 86 – 88°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.36 (br s, 1H), 7.30 (d, J=8.4Hz, 2H), 7.20 (d, J=8.4Hz, 2H), 6.55 (d, J=16Hz, 2H), 6.40 (br s, 1H), 6.37 (dd, J=3.6, 2.0Hz, 1H), 6.32 (d, J=3.2Hz, 1H), 6.18 (dd, J=15.6, 6.8Hz, 1H), 5.76 (d, J=7.2Hz, 1H), 4.45 (m, 2H), 2.17 (s, 3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 169.3 (Cq), 168.1 (Cq), 151.2 (Cq), 142.8 (CH), 136.2 (Cq), 133.5 (Cq), 129.1 (2CH), 129.0 (2CH), 123.2 (CH), 120.4 (CH), 111.5 (CH), 110.2 (CH), 74.2 (CH), 42.7 (CH_2), 21.0 (CH_3) ; **IR** (υ, cm^{-1}) 3725, 3627, 3292, 3082, 2933, 1742, 1662, 1538, 1491, 1371, 1226, 1091, 1015 ; **HRMS** (EI) Calcd. for $C_{17}H_{16}ClNO_4$: 333.0768 found : 333.0771

(*E*)-1-(cyclohexylamino)-4-(furan-2-yl)-1-oxobut-3-en-2-yl acetate (1k)



Compound **1k** was prepared according to the general procedure A. Purification by flash chromatography with a gradient PE/Et_2O (from 30/70 to 0/100) as eluant gave the desired product (267 mg, 46%) as an yellow solid. **Mp** 133 – 134°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) ; 7.35 (d, *J* = 1.8 Hz, 1H), 6.54 (d, *J* = 15.9 Hz, 1H), 6.37 (dd, *J* = 3.3, 1.8 Hz, 1H), 6.31 (d, *J* = 3.3 Hz, 1H), 6.16 (dd, *J* = 15.9, 7.1 Hz, 1H), 5.89 (d, *J* = 7.8 Hz, 1H), 5.67 (d, *J* = 7.1 Hz, 1H), 3.84-3.74 (m, 1H), 2.19 (s, 3H), 1.94-1.91 (m, 2H), 1.73-1.59 (m, 3H), 1.42-1.32 (m, 2H), 1.21-1.12 (m, 3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 169.1, 166.8, 151.4, 142.6, 122.8, 121.0, 111.4, 109.8, 74.2, 48.2, 33.0, 33.0, 25.4, 24.8, 21.1 ; **IR** (υ, cm^{-1}) 3287, 2930, 2858, 1744, 1655, 1546, 1453, 1371, 1230, 1155, 1107, 1021 ; **HRMS** (EI) Calcd. for $C_{16}H_{21}NO_4$: 291.1471 found : 291.1461

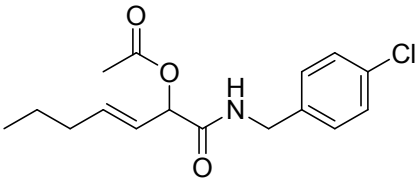
Acetic acid 1-(4-chloro-benzylcarbamoyl)-2-methyl-3-phenyl-allyl ester (1l)



Compound **1l** was prepared according to the general procedure Abis. Purification by flash chromatography with a gradient Et_2O/EP (from 20/80 to 100/0) as eluant gave the desired product (804 mg, 75%) as a brown solid. **CCM** Rf (80/20 Et_2O/EP) = 0.25 ; **Mp** 92 – 93°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.36 (m, 3H), 7.31 (m, 4H), 7.25 (d, J=8.4Hz, 2H), 6.76 (s, 1H), 6.51 (br

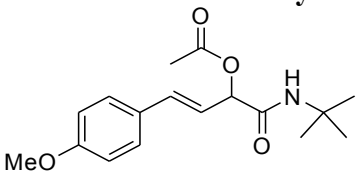
s, 1H), 5.71 (s, 1H), 4.48 (m, 2H), 2.22 (s, 3H), 1.95 (s, 3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 169.3 (Cq), 168.1 (Cq), 136.5 (Cq), 136.2 (Cq), 133.5 (Cq), 132.0 (Cq), 131.7 (CH), 129.2 (CH), 129.1 (4CH), 129.0 (2CH), 128.3 (2CH), 127.3 (CH), 79.2 (CH), 42.7 (CH_2), 21.1 (CH_3), 14.2 (CH_3) ; **IR** (υ, cm^{-1}) 3281, 3055, 2922, 1737, 1655, 1523, 1490, 1368, 1221, 1088, 1014 ; **HRMS** (EI) Calcd. for $C_{20}H_{20}ClNO_3$: 357.1132 found : 357.1136

(*E*)-1-((4-chlorobenzyl)amino)-1-oxohept-3-en-2-yl acetate (1m)



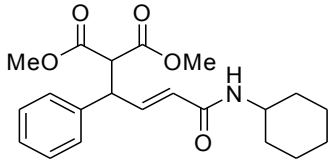
Compound **1m** was prepared according to the general procedure A. Purification by flash chromatography with a gradient PE/AcOEt (from 80/20 to 70/30) as eluant gave the desired product (647 mg, 71%) as an yellow oil. **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.30 (d, *J* = 8.3 Hz, 2H), 7.19 (d, *J* = 8.3 Hz, 2H), 6.29 (br s, 1H), 5.93-5.84 (m, 1H), 5.59-5.53 (m, 2H), 4.46 (dd, *J* = 14.9, 5.8 Hz, 1H), 4.41 (dd, *J* = 14.9, 5.8 Hz, 1H), 2.14 (s, 3H), .2.06 (q, *J* = 7.3 Hz, 2H), 1.41 (sext, *J* = 7.3 Hz, 2H), 0.89 (t, *J* = 7.3 Hz, 3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 169.3, 168.6, 137.7, 136.3, 133.4, 129.0, 128.9, 123.3, 74.7, 42.5, 34.2, 21.8, 21.0, 13.6 ; **IR** (υ, cm^{-1}) 3299, 2961, 2929, 1744, 1663, 1534, 1496, 1374, 1231, 1095, 1018 ; **HRMS** (EI) Calcd. for $C_{16}H_{20}ClNO_3$: 309.1132 found : 309.1129

Acetic acid 1-tert-butylcarbamoyl-3-(4-methoxy-phenyl)-allyl ester (1n)



Compound **1n** was prepared according to general procedure Abis. Purification by flash chromatohraphy with a gradient Et_2O/EP (20/80 to 60/40) gave the desired product (358 mg, 39%) as a yellow solid. **CCM** Rf (80/20 Et_2O/EP) = 0.18 ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.32 (d, J=7.6Hz, 2H), 6.84 (d, J=8.0Hz, 2H), 6.66 (d, J=16.0Hz, 1H), 6.10 (dd, J=16.0, 7.2Hz, 1H), 5.84 (brs, 1H), 5.58 (d, J=7.2Hz, 1H), 3.79 (s, 3H), 2.18 (s, 3H), 1.36 (s, 9H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 170.0 (Cq), 168.0 (Cq), 160.4 (Cq), 135.2 (CH), 128.7 (2CH), 121.0 (CH), 114.6 (2CH), 75.6 (CH), 55.9 (CH, OMe), 52.1 (Cq, tBu), 29.3 ($3CH_3$, tBu), 21.7 (Me) ; **IR** (υ, cm^{-1}) 3306, 2965, 1738, 1665, 1606, 1509, 1454, 1364, 1218, 1173, 1028. ; **HRMS** (EI) Calcd. for $C_{17}H_{23}NO_4$: 305.1627 found : 305.1621

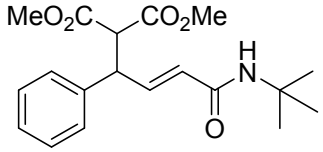
2-(3-Cyclohexylcarbamoyl-1-phenyl-allyl)-malonic acid dimethyl ester (2a)



Compound **2a** was prepared according to the general procedure B. Purification by flash chromatography with a gradient Et_2O/PE (from 20/80 to 80/20) as eluant gave the desired product (190 mg, 77%) as an yellow solid. **CCM** Rf (80/20 Et_2O/EP) = 0.20 ; **Mp** 151 – 152°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.33 (m, 2H), 7.29 (d, J=5.2Hz, 1H), 7.25 (m, 2H), 6.93 (1H, dd, J=14.8, 8.0Hz, 1H), 5.78 (d, J=15.2Hz, 1H), 5.33 (d, J=8.0Hz, 1H), 4.25 (t, J=11.2Hz, 1H), 3.92 (d, J=11.2Hz, 1H), 3.82 (m, 1H), 3.77 (s, 3H), 3.50 (s, 3H), 1.92 (m, 2H), 1.72 (m, 2H), 1.63 (m,

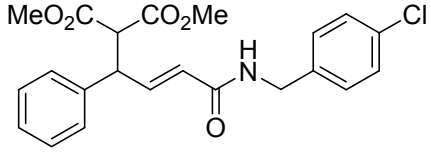
2H), 1.35 (m, 2H), 1.15 (m, 2H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 168.0 (Cq), 167.5 (Cq), 164.1 (Cq), 142.1 (CH), 138.5 (Cq), 128.9 (2CH), 128.2 (2CH), 127.6 (CH), 125.9 (CH), 56.9 (CH), 52.8 (CH_3), 52.6 (CH_3), 48.3 (CH), 48.0 (CH), 33.1 ($2CH_2$), 25.5 (CH_2), 24.8 ($2CH_2$) ; **IR** (υ, cm^{-1}) 3274, 2930, 2853, 1737, 1667, 1626, 1541, 1452, 1434, 1255, 1152 ; **HRMS** (EI) Calcd. for $C_{21}H_{27}NO_5$: 373.1889 found : 373.1887

(*E*)-dimethyl 2-(1-(*tert*-butylamino)-1-oxo-4-phenylbut-3-en-2-yl)malonate (2c)



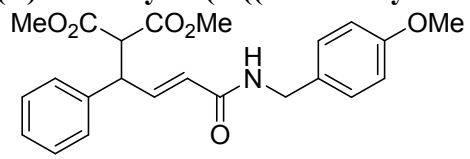
Compound **2c** was prepared according to the general procedure B. by flash chromatography with a gradient PE/Et_2O (from 60/40 to 50/50) as eluant gave the desired product (110 mg, 87%) as an yellow oil. **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.32-7.28 (m, 2H), 7.25-7.20 (m, 3H), 6.87 (dd, *J* = 15.2, 8.3 Hz, 1H), 5.69 (d, *J* = 15.2 Hz, 1H), 5.23 (br s, 1H), 4.22 (dd, *J* = 10.9, 8.3 Hz, 1H), 3.88 (d, *J* = 10.9 Hz, 1H), 3.75 (s, 3H), 3.46 (s, 3H), 1.34 (s, 9H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 167.9, 167.4, 164.3, 141.7, 138.5, 128.8, 128.2, 127.5, 126.6, 56.8, 52.8, 52.5, 51.4, 47.8, 28.7 ; **IR** (υ, cm^{-1}) 3291, 2962, 2931, 1738, 1668, 1632, 1541, 1454, 1436, 1362, 1261, 1224, 1154 ; **HRMS** (EI) Calcd. for $C_{19}H_{25}NO_5$: 347.1733 found : 347.1733

(*E*)-dimethyl 2-(1-(4-chlorobenzylamino)-1-oxo-4-phenylbut-3-en-2-yl)malonate (2d)



Compound **2d** was prepared according to the general procedure B. Purification by flash chromatography with a gradient PE/Et_2O (from 40/60 to 20/80) as eluant gave the desired product (83 mg, 69%) as an yellow solid. **Mp** 86 – 87°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.31-7.25 (m, 5H), 7.23-7.16 (m, 4H), 6.98 (dd, *J* = 15.2, 8.3 Hz, 1H), 5.89 (t, *J* = 5.8 Hz, 1H), 5.81 (d, *J* = 15.2 Hz, 1H), 4.44 (dd, *J* = 14.9, 5.8 Hz, 1H), 4.38 (dd, *J* = 14.9, 5.8 Hz, 1H), 4.23 (dd, *J* = 10.8, 8.3 Hz, 1H), 3.89 (d, *J* = 10.8 Hz, 1H), 3.73 (s, 3H), 3.46 (s, 3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 167.8, 167.4, 165.0, 143.2, 138.3, 136.6, 133.3, 129.2, 128.8, 128.8, 128.1, 127.6, 125.0, 56.7, 52.8, 52.5, 47.9, 42.9 ; **IR** (υ, cm^{-1}) 2956, 1736, 1668, 1633, 1538, 1491, 1434, 1259, 1149, 1091, 1015 ; **HRMS** (EI) Calcd. for $C_{22}H_{22}ClNO_5$: 415.1187 found : 415.1187

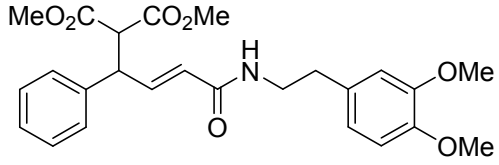
(*E*)-dimethyl 2-(4-((4-methoxybenzyl)amino)-4-oxo-1-phenylbut-2-en-1-yl)malonate (2e)



Compound **2e** was prepared according to the general procedure B. Purification by flash chromatography with a gradient PE/Et_2O (from 20/80 to 0/100) as eluant gave the desired product (95 mg, 78%) as an yellow oil. **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.31-7.27 (m, 2H), 7.24-7.19 (m, 3H), 7.17 (d, *J* = 8.6 Hz, 2H), 6.96 (dd, *J* = 15.2, 8.6 Hz, 1H), 6.83 (d, *J* = 8.6 Hz, 2H), 5.79-5.76 (m, 2H), 4.40 (dd, *J* = 14.9, 5.8 Hz, 1H), 4.35 (dd, *J* = 14.9, 5.8 Hz, 1H), 4.22 (dd, *J* = 10.9, 8.6 Hz, 1H), 3.89 (d, *J* = 10.9 Hz, 1H), 3.77 (s, 3H), 3.73 (s, 3H), 3.45 (s, 3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 167.8, 167.4, 164.8, 159.0, 142.8, 138.3, 130.0, 129.3, 128.8, 128.1, 127.5, 125.3, 114.0, 56.7, 55.2, 52.8, 52.5, 47.9, 43.1 ; **IR** (υ, cm^{-1}) 3282, 2954, 1737, 1670, 1632, 1552,

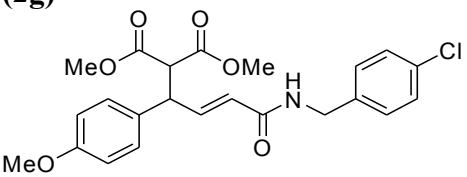
1514, 1437, 1301, 1248, 1175, 1029 ; **HRMS** (EI) Calcd. for $C_{23}H_{25}NO_6$: 411.1682 found : 411.1691

(*E*)-dimethyl 2-(4-(3.4-dimethoxyphenethylamino)-4-oxo-1-phenylbut-2-enyl)malonate (2f)



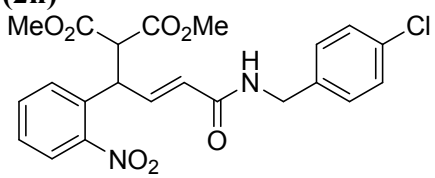
Compound **2f** was prepared according to the general procedure B. Purification by flash chromatography with a gradient PE/AcOEt (from 50/50 to 30/70) as eluant gave the desired product (171 mg, 72%) as an yellow solid. **Mp** 134 – 135°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.31-7.28 (m, 2H), 7.25-7.19 (m, 3H), 6.92 (dd, *J* = 15.2, 8.6 Hz, 1H), 6.79 (d, *J* = 8.1 Hz, 1H), 6.71-6.67 (m, 2H), 5.72 (d, *J* = 15.2 Hz, 1H), 5.46 (t, *J* = 5.6 Hz, 1H), 4.21 (dd, *J* = 10.9, 8.6 Hz, 1H), 3.88 (d, *J* = 10.9, 1H), 3.85 (s, 3H), 3.82 (s, 3H), 3.72 (s, 3H), 3.57-3.48 (m, 2H), 3.47 (s, 3H), 2.75 (t, *J* = 7.1 Hz, 2H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 167.8, 167.4, 165.0, 149.0, 147.7, 142.6, 138.4, 131.2, 128.8, 128.1, 127.6, 125.4, 120.5, 111.8, 111.3, 56.8, 55.9, 55.8, 52.8, 52.5, 47.9, 40.8, 35.1 ; **IR** (υ, cm^{-1}) 3285, 2957, 1737, 1670, 1628, 1544, 1517, 1454, 1433, 1321, 1259, 1234, 1196, 1147, 1029 ; **HRMS** (EI) Calcd. for $C_{25}H_{29}NO_7$: 455.1944 found : 455.1953

2-[3-(4-Chloro-benzylcarbamoyl)-1-(4-methoxy-phenyl)-allyl]-malonic acid dimethyl ester (2g)



Compound **2g** was prepared according to the general procedure B. Purification by flash chromatography with a gradient Et_2O/PE (from 20/80 to 100/0) as eluant gave the desired product (90 mg, 75%) as an yellow solid. **CCM** Rf (80/20 Et_2O/EP) = 0.22 ; **Mp** 116 – 118°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.27 (d, J=5.6Hz, 2H), 7.18 (d, J=6.8Hz, 2H), 7.12 (d, J=7.2Hz, 2H), 6.96 (dd, J=15.2, 8.4Hz, 1H), 6.83 (d, J=7.2Hz, 2H), 5.96 (br s, 1H), 5.79 (d, J=15.2Hz, 1H), 4.44 (dd, J=11.2, 6.0 Hz, AB,1H), 4,37 (dd, J=14.2, 6.0 Hz, AB, 1H), (dd, 4.19 (t, J=9.2Hz, 1H), 3.84 (d, J=10.8Hz, 1H), 3.77 (s, 3H), 3.73 (s, 3H), 3.48 (s, 3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 168.0 (Cq), 167.5 (Cq), 165.1 (Cq), 159.0 (Cq), 143.5 (CH), 136.6 (Cq), 133.5 (Cq), 130.22 (Cq), 129.3 (4CH), 128.8 (2CH), 124.8 (CH), 114.3 (2CH), 57.0 (CH), 55.2 (CH_3, OMe), 52.8 (CH_3, OMe), 52.6 (CH_3, OMe), 47.2 (CH), 43.0 (CH_2) ; **IR** (υ, cm^{-1}) 3275, 3066, 2952, 2838, 1735, 1666, 1631, 1511, 1434, 1248, 1178, 1091, 1031 ; **HRMS** (EI) Calcd. for $C_{23}H_{24}ClNO_6$: 445.1292 found : 445.1289

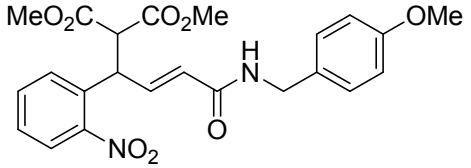
(*E*)-dimethyl 2-(4-((4-chlorobenzyl)amino)-1-(2-nitrophenyl)-4-oxobut-2-en-1-yl)malonate (2h)



Compound **2h** was prepared according to the general procedure B. Purification by flash chromatography with a gradient PE/Et_2O (from 50/50 to 0/100) as eluant gave the desired product (80 mg, 67%) as a beige solid. **Mp** 57 – 58°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.78 (d, *J* =

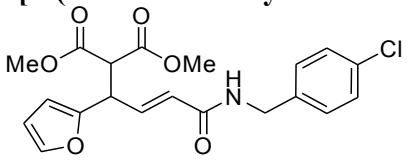
7.6 Hz, 1H), 7.57 (t, *J* = 7.6 Hz, 1H), 7.41 (d, *J* = 7.6 Hz, 1H), 7.39 (t, *J* = 7.6 Hz, 1H), 7.26 (d, *J* = 8.1 Hz, 2H), 7.18 (d, *J* = 8.1 Hz, 2H), 6.94 (dd, *J* = 14.9, 8.6 Hz, 1H), 6.07-6.01 (m, 2H), 4.90 (dd, *J* = 10.9, 8.6 Hz, 1H), 4.45 (dd, *J* = 14.9, 5.8 Hz, AB, 1H), 4.38 (dd, *J* = 14.9, 5.8 Hz, AB, 1H), 3.98 (d, *J* = 10.9 Hz, 1H), 3.72 (s, 3H), 3.49 (s, 3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 167.2, 166.8, 164.7, 150.0, 140.8, 136.4, 133.3, 133.3, 133.0, 129.2, 129.2, 128.8, 128.3, 126.6, 124.7, 56.5, 53.0, 52.9, 43.0, 41.4 ; **IR** (υ, cm^{-1}) 3282, 2953, 1740, 1670, 1636, 1524, 1493, 1437, 1356, 1294, 1252, 1165, 1095, 1014 ; **HRMS** (EI) Calcd. for $C_{22}H_{21}ClN_2O_7$: 460.1037 found : 460.1035

(*E*)-dimethyl 2-(4-((4-chlorobenzyl)amino)-1-(2-nitrophenyl)-4-oxobut-2-en-1-yl)malonate (2i)



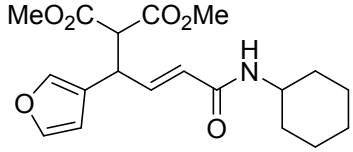
Compound **2i** was prepared according to the general procedure B. Purification by flash chromatography with a gradient PE/AcOEt (from 50/50 to 30/70) as eluant gave the desired product (88 mg, 74%) as an yellow oil. **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.79 (d, *J* = 7.8 Hz, 1H), 7.57 (t, *J* = 7.8 Hz, 1H), 7.42 (d, *J* = 7.8 Hz, 1H), 7.38 (t, *J* = 7.8 Hz, 1H), 7.19 (d, *J* = 8.6 Hz, 2H), 6.93 (dd, *J* = 15.2, 8.6 Hz, 1H), 6.84 (d, *J* = 8.6 Hz, 2H), 5.98 (d, *J* = 15.2 Hz, 1H), 5.80 (t, *J* = 5.6 Hz, 1H), 4.89 (dd, *J* = 10.6, 8.6 Hz, 1H), 4.42 (dd, *J* = 14.7, 5.6 Hz, AB, 1H), 4.37 (dd, *J* = 14.7, 5.6 Hz, AB, 1H), 3.97 (d, *J* = 10.6 Hz, 1H), 3.78 (s, 3H), 3.73 (s, 3H), 3.49 (s, 3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 167.2, 166.8, 164.5, 159.1, 150.1, 140.5, 133.4, 132.9, 129.9, 129.3, 129.2, 128.2, 126.8, 124.7, 114.1, 56.6, 55.3, 53.0, 52.8, 43.3, 41.3 ; **IR** (υ, cm^{-1}) 3276, 2955, 1735, 1669, 1632, 1525, 1512, 1435, 1353, 1295, 1246, 1174, 1111, 1030 ; **HRMS** (EI) Calcd. for $C_{23}H_{24}N_2O_8$: 456.1533 found : 456.1545

2-[3-(4-Chloro-benzylcarbamoyl)-1-furan-2-yl-allyl]-malonic acid dimethyl ester (2j)



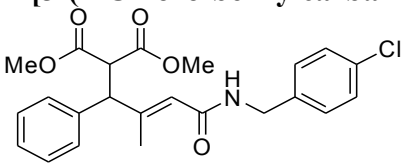
Compound **2j** was prepared according to the general procedure B. Purification by flash chromatography with a gradient Et_2O/PE (from 20/80 to 100/0) as eluant gave the desired product (90 mg, 37%) as a yellow solid. **CCM** Rf (80/20 Et_2O/EP) = 0.55 ; **Mp** 123 – 124°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.32 (s, 1H), 7.27 (d, J=8.4Hz, 2H), 7.18 (d, J=8.4Hz, 2H), 6.90 (dd, J=15.2, 8.4Hz, 1H), 6.27 (s, 1H), 6.13 (s, 1H), 5.92 (br s, 1H), 5.87 (d, J=14.8Hz, 1H), 4.42 (t, J=6.0Hz, 2H), 4.36 (t, J=9.2Hz, 1H), 3.89 (d, J=10.4Hz, 1H), 3.71 (s, 3H), 3.61 (s, 3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 167.5 (Cq), 167.4 (Cq), 164.9 (Cq), 151.2 (Cq), 142.4 (CH), 140.1 (CH), 136.6 (Cq), 133.41 (Cq), 129.3 (2CH), 128.9 (2CH), 126.2 (CH), 110.5 (CH), 107.42 (CH), 55.0 (CH), 52.9 ($2CH_3$), 43.0 (CH_2), 41.4 (CH) ; **IR** (υ, cm^{-1}) 3275, 2953, 1737, 1670, 1633, 1541, 1491, 1434, 1252, 1163, 1092, 1014 ; **HRMS** (EI) Calcd. for $C_{20}H_{20}ClNO_6$: 405.0979 found : 405.0987

(*E*)-dimethyl 2-(1-(cyclohexylamino)-4-(furan-3-yl)-1-oxobut-3-en-2-yl)malonate (2k)



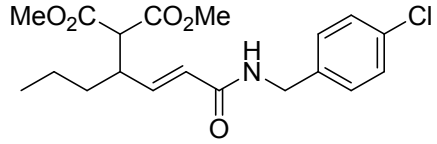
Compound **2k** was prepared according to the general procedure B. Purification by flash chromatography with a gradient PE/Et_2O (from 70/30 to 20/80) as eluant gave the desired product (92 mg, 74%) as an yellow solid. **Mp** 141 – 142°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.33 (d, *J* = 1.8 Hz, 1H), 6.83 (dd, *J* = 15.2, 8.3 Hz, 1H), 6.28 (dd, *J* = 3.3, 1.8 Hz, 1H), 6.14 (d, *J* = 3.3 Hz, 1H), 5.81 (d, *J* = 15.2 Hz, 1H), 5.34 (br s, 1H), 4.36 (dd, *J* = 10.3, 8.3 Hz, 1H), 3.90 (d, *J* = 10.3 Hz, 1H), 3.85-3.75 (m, 1H), 3.73 (s, 3H), 3.62 (s, 3H), 1.93-1.89 (m, 2H), 1.71-1.59 (m, 3H), 1.40-1.31 (m, 2H), 1.19-1.07 (m, 3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 167.5, 167.4, 163.9, 151.4, 142.3, 138.9, 126.9, 110.4, 107.2, 55.0, 52.8, 52.8, 48.3, 41.4, 33.1, 25.5, 24.8 ; **IR** (υ, cm^{-1}) 3280, 2933, 2859, 1741, 1669, 1628, 1546, 1453, 1436, 1347, 1261, 1152, 1011 ; **HRMS** (EI) Calcd. for $C_{19}H_{25}NO_6$: 363.1682 found : 363.1687

2-[3-(4-Chloro-benzylcarbamoyl)-2-methyl-1-phenyl-allyl]-malonic acid dimethyl ester (2l)



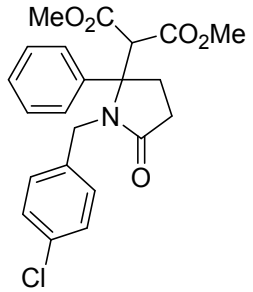
Compound **2l** was prepared according to the general procedure B. Purification by flash chromatography with a gradient Et_2O/PE (from 20/80 to 100/0) as eluant gave the desired product (103 mg, 57%) as an yellow solid. **CCM** Rf (50/50 Et_2O/EP) = 0.11 ; **Mp** 68°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.29 (m, 4H), 7.20 (m, 5H), 5.79 (s, 1H), 5.75 (br s, 1H), 4.42 (d, J=5.6Hz, 2H), 4.14 (d, J=12.0Hz, AB system, 1H), 4.11 (d, J=12.0Hz, AB system, 1H), 3.73 (s, 3H), 3.48 (s, 3H), 2.06 (s,3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 168.1 (Cq), 167.6 (Cq), 166.1 (Cq), 153.0 (Cq), 137.8 (Cq), 136.9 (Cq), 133.3 (Cq), 129.3 (2CH), 128.9 (2CH), 128.7 (2CH), 128.1 (2CH), 127.7 (CH), 118.7 (CH), 55.1 (CH), 54.4 (CH), 53.0 (CH_3), 52.7 (CH_3), 42.8 (CH_2), 17.0 (CH_3) ; **IR** (υ, cm^{-1}) 3293, 2951, 1736, 1635, 1524, 1433, 1091 ; **HRMS** (EI) Calcd. for $C_{23}H_{24}ClNO_5$: 429.1343 found : 429.1339

(*E*)-dimethyl 2-(1-((4-chlorobenzyl)amino)-1-oxohept-2-en-4-yl)malonate (2m)



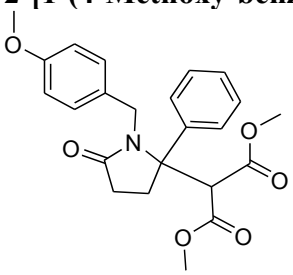
Compound **2m** was prepared according to the general procedure B. Purification by flash chromatography with a gradient PE/AcOEt (from 60/40 to 50/50) as eluant gave the desired product (53 mg, 43%) as a white solid. **Mp** 146 – 147°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.30 (d, *J* = 8.3 Hz, 2H), 7.22 (d, *J* = 8.3 Hz, 2H), 6.64 (dd, *J* = 15.2, 9.9 Hz, 1H), 5.85 (d, *J* = 15.2 Hz, 1H), 5.79 (br s, 1H), 4.48 (dd, *J* = 15.2, 5.8 Hz, AB, 1H), 4.43 (dd, *J* = 15.2, 5.8 Hz, AB,1H), 3.74 (s, 3H), 3.67 (s, 3H), 3.44 (d, *J* = 9.1 Hz, 1H), 2.97-2.89 (m, 1H),1.45-1.30 (m, 3H), 1.26-1.16 (m, 1H), 0.87 (t, *J* = 7.1 Hz, 3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 168.3, 168.2, 165.0, 143.8, 136.6, 133.3, 129.2, 128.8, 125.8, 56.0, 52.6, 52.4, 42.9, 42.1, 34.1, 20.2, 13.7 ; **IR** (υ, cm^{-1}) 3286, 2956, 2932, 1737, 1668, 1623, 1531, 1492, 1434, 1301, 1255, 1173, 1150, 1092, 1016 ; **HRMS** (EI) Calcd. for $C_{19}H_{24}ClNO_5$: 381.1343 found : 381.1349

dimethyl 2-(1-(4-chlorobenzyl)-5-oxo-2-phenylpyrrolidin-2-yl)malonate (3d)



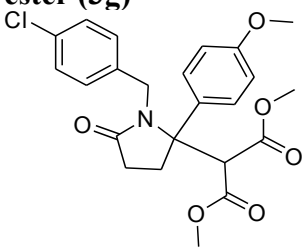
Compound **3d** was prepared according to the general procedure C. Purification by flash chromatography with a gradient PE/Et_2O (from 20/80 to 30/70) as eluant gave the desired product (39 mg, 33%) as an yellow oil.**^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.25-7.15 (m, 5H), 7.07 (d, *J* = 8.3 Hz, 2H), 6.91 (d, *J* = 8.3 Hz, 2H), 4.39 (s, 1H), 4.25 (d, *J* = 15.2 Hz, 1H), 4.01 (d, *J* = 15.2 Hz, 1H), 3.66 (s, 3H), 3.57 (s, 3H), 3.36-3.28 (m, 1H), 2.70-2.66 (m, 2H), 2.60-2.52 (m, 1H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 175.9, 167.1, 167.0, 142.6, 136.1, 132.8, 130.1, 128.7, 128.1, 128.1, 125.9, 69.1, 55.1, 52.9, 52.8, 44.0, 30.4, 29.9 ; **IR** (υ, cm^{-1}) 2953, 1754, 1733, 1678, 1492, 1434, 1395, 1328, 1265, 1203, 1142, 1092, 1016 ; **HRMS** (EI) Calcd. for $C_{22}H_{22}ClNO_5$: 415.1187 found : 415.1184

2-[1-(4-Methoxy-benzyl)-5-oxo-2-phenyl-pyrrolidin-2-yl]-malonic acid dimethyl ester (3e)



Compound **3e** was prepared according to the general procedure C. Purification by flash chromatography with a gradient Et_2O/PE (from 20/80 to 100/0) as eluant gave the desired product (63 mg, 38%) as an yellow oil. **CCM** Rf (80/20 Et_2O/EP) = 0.42 ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.20 (m, 3H), 6.91 (d, J=8.8Hz, 2H), 6.85 (t, J=8.8Hz, 2H), 6.65 (d, J=8.8 Hz, 2H), 4.39 (s, 1H), 4.31 (d, J=15.2Hz, 2H), 6.73 (s, 3H), 6.66 (s, 3H), 6.53 (s, 3H), 3.32 (m, AB system, 1H), 2.68 (m, 2H), 2.54 (m, AB system, 1H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 175.9 (Cq), 167.3 (Cq), 167.2 (Cq), 158.6 (Cq), 142.9 (Cq), 130.1 (2CH), 128.7 (2CH), 128.1 (CH), 126.1 (2CH), 113.5 (2CH), 69.2 (Cq), 55.3 (CH_3), 55.1 (CH), 52.9 (CH_3), 52.8 (CH_3), 44.0 (CH_2), 30.5 (CH_2), 30.1 (CH_2) ; **IR** (υ, cm^{-1}) 3293, 2951, 2836, 1731, 1662, 1509, 1398, 1240, 1113, 1028 ; **HRMS** (EI) Calcd. for $C_{23}H_{25}NO_6$: 411.1682 found : 411.1684

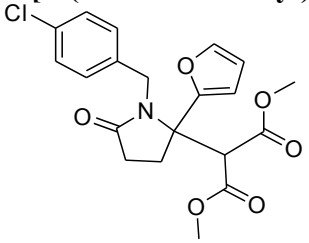
2-[1-(4-Chloro-benzyl)-2-(4-methoxy-phenyl)-5-oxo-pyrrolidin-2-yl]-malonic acid dimethyl ester (3g)



Compound **3g** was prepared according to the general procedure C. Purification by flash chromatography with a gradient Et_2O/PE (from 20/80 to 100/0) as eluant gave the desired product (64 mg, 64%) as an yellow oil. **CCM** Rf (80/20 Et_2O/EP) = 0.17 ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.08 (m, 4H), 6.90 (d, J=8.4Hz, 2H), 6.73 (d, J=8.4Hz, 2H), 4.35 (s, 1H), 4.17 (d, J=15.2Hz, AB system, 1H), 4.03 (d, J=15.2Hz, AB system, 1H), 3.77 (s, 3H), 3.66 (s, 3H), 3.56 (s, 3H), 3.30

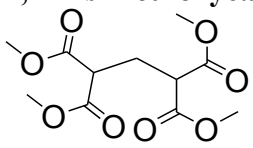
(m, AB system, 1H), 2.65 (t, J=8.8Hz, 2H), 2.52 (m, AB system, 1H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 175.8 (Cq), 167.2 (Cq), 167.1 (Cq), 159.2 (Cq), 136.4 (Cq), 134.4 (Cq), 132.7 (Cq), 130.1 (2CH), 128.1 (2CH), 127.3 (2CH), 113.9 (2CH), 68.8 (Cq), 55.4 (CH_3), 55.1 (CH), 52.9 (CH_3), 52.8 (CH_3), 43.9 (CH_2), 30.2 (CH_2), 30.0 (CH_2) ; **IR** (υ, cm^{-1}) 2952, 2838, 1754, 1733, 1671, 1513, 1450, 1433, 1249, 1188, 1088, 1015 ; **HRMS** (EI) Calcd. for $C_{23}H_{24}ClNO_6$: 445.1292 found : 445.1292

2-[1-(4-Chloro-benzyl)-2-furan-2-yl-5-oxo-pyrrolidin-2-yl]-malonic acid dimethyl ester (3j)



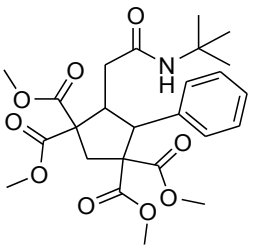
Compound **3j** was prepared according to the general procedure C. Purification by flash chromatography with a gradient Et_2O/PE (from 20/80 to 100/0) as eluant gave the desired product (48 mg, 53%) as an yellow oil. **CCM** Rf (80/20 Et_2O/EP) = 0.32 ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.22 (s, 1H), 7.12 (d, J=8.4Hz, 2H), 6.98 (d, J=8.0 Hz, 2H), 6.23 (s, 1H), 6.21 (s, 1H), 4.32 (d, J=15.6Hz, AB system, 1H), 4.30 (s, 1H), 4.24 (d, J=15.6Hz, AB system, 1H), 3.64 (s, 3H), 3.56 (s, 3H), 3.21 .(m, 2H), 2.61 (m, 2H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 175.6 (Cq), 166.5 (Cq), 166.4 (Cq), 153.2 (Cq), 142.5 (CH), 135.9 (Cq), 132.8 (Cq), 129.5 (2CH), 128.3 (2CH), 110.6 (CH), 108.0 (CH), 65.1 (Cq), 54.1 (CH), 53.0 (CH_3), 52.9 (CH_3), 43.5 (CH_2), 29.5 (CH_2), 26.1 (CH_2) ; **IR** (υ, cm^{-1}) 3725, 3627, 3307, 2952, 1734, 1669, 1434, 1399, 1328, 1149, 1089, 1015 ; **HRMS** (EI) Calcd. for $C_{20}H_{20}ClNO_6$: 405.0979 found : 405.0982

2,4-Bis-methoxycarbonyl-pentanedioic acid dimethyl ester (4)



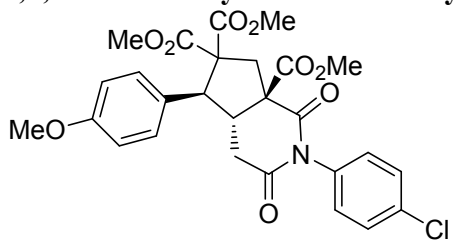
A mixture of dimethylmalonate (5.5 mL, 2.4eq), diiodomethane (1eq) and potassium carbonate (2.4 equiv) in 50 mL of DMF was stirred at room temperature during 24h, then at 100°C during 4h. 150 mL of diethyl ether was added. The crude was filtrated, washed with diethylether. The filtrate was washed with 2*50 mL of water and 50mL of brine, and dried with MgSO4. The crude was purified by silica gel column chromatography with a gradient of AcOEt in EP (10:90 to 30:70), to afford 2.66g of uncolored oil (48% yield). **CCM** Rf (30/70 AcOEt/EP) = 0.39 ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 3.74 (s, 12H), 3.50 (t, J=7.6 Hz, 2H), 2.48 (t, J=7.6 Hz, 2H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 139.0 (4Cq), 52.9 ($4CH_3$), 49.0 (2CH), 27.4 (CH_2) ; **IR** (υ, cm^{-1}) 2956, 1728, 1434, 1198, 1149, 1033 ; **HRMS** (EI) Calcd. for $C_{11}H_{16}O_8$: 276.0845 found : 276.0847

4-(tert-Butylcarbamoyl-methyl)-5-phenyl-cyclopentane-1,1,3,3-tetracarboxylic acid tetramethyl ester (5c)



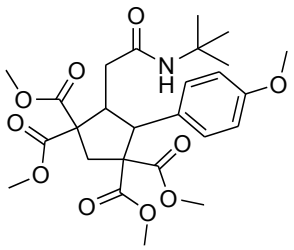
Compound **5c** was prepared according to the general procedure D. Purification by flash chromatography with a gradient Et_2O/PE (from 50/50 to 70/30) as eluant gave the desired product (140 mg, 78%) as a yellow oil. **CCM** Rf (70/2=30 Et_2O/EP) = 0.36 ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.21 (m, 5H), 5.67 (br s, 1H), 3.89 (d, J=13.2 Hz, 1H), 3.78 (s, 3H), 3.70 (br s, 6H), 3.51 (m, 1H), 3.15 (d, J=16.0Hz, AB system, 1H), 3.08 (s, 3H), 2.95 (d, J=16.0Hz, AB system, 1H), 2.41 (dd, J^1=14.8, 6.4 Hz, AB, 1H), 1.94 (dd, J^1=16.0, 4.8 Hz, AB, 1H), 1.13 (s, 9H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 171.8 (Cq), 171.2 (Cq), 170.9 (Cq), 170.5 (Cq), 169.6 (Cq), 136.4 (Cq), 128.2 (4CH), 127.7 (CH), 63.4 (Cq), 61.2 (Cq), 55.1 (CH), 53.1 (CH_3), 52.9 (CH_3), 52.8 (CH_3), 52.4 (CH_3), 50.8 (Cq), 46.1 (CH), 41.6 (CH_2), 38.2 (CH_2), 28.5 ($3CH_3$) ; **IR** (υ, cm^{-1}) 3395, 2954, 1727, 1666, 1530, 1433, 1208, 1077 ; **HRMS** (EI) Calcd. for $C_{25}H_{33}NO_9$: 491.2155 found : 491.2161

3-(4-Chloro-benzyl)-6-(4-methoxy-phenyl)-1,4-dioxo-tetrahydro-cyclopenta[e][1,2]oxazepine-7,7,8a-tricarboxylic acid trimethyl ester (5g)



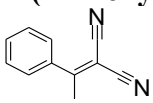
Compound **5g** was prepared according to the general procedure D. Purification by flash chromatography with a gradient AcOEt/PE (from 10/90 to 50/50) as eluant gave the desired product (159 mg, 67%) as white crystals. **CCM** Rf (50/50 AcOEt/EP) = 0.37 ; **Mp** 134 – 136°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.27 (s, 4H), 7.10 (d, J=8.8Hz, 2H), 7.07 (d, J=8.8 Hz, 2H), 5.01 (d, J=14.4Hz, AB system, 1H), 4.92 (d, J=14.4Hz, AB system, 1H), 3.82 (d, J=12.8 Hz, 1H), 3.77 (s, 3H), 3.73 (s, 3H), 3.70 (s, 3H), 3.51 (d, J=14.8 Hz, AB system, 1H), 3.17 (d, J=14.8 Hz, AB system, 1H), 3.14 (s, 3H), 3.09 (dd, J=12.0, 5.6 Hz, 1H), 2.86 (dd , J=18.4, 6.4 Hz, AB system, 1H), 2.54 (dd , J=18.4, 6.4 Hz, AB system, 1H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 171.6 (Cq), 170.0 (2Cq), 169.9 (Cq), 169.4 (Cq), 159.3 (Cq), 135.1 (Cq), 133.3 (Cq), 130.0 (2CH), 129.9 (2CH), 128.5 (2CH), 127.4 (Cq), 114.0 (2CH), 63.0 (Cq), 58.5 (Cq), 55.3 (CH_3), 53.6 (CH_3), 53.5 (CH_3), 53.3 (CH), 52.6 (CH_3), 43.2 (CH_2), 42.3 (CH), 40.8 (CH_2), 31.1 (CH_2) ; **IR** (υ, cm^{-1}) 3002, 2953, 2840, 1726, 1677, 1610, 1514, 1433, 1089 ; **HRMS** (EI) Calcd. for $C_{29}H_{32}ClNO_{10}$: 589.1715 found : 589.1721

4-(tert-Butylcarbamoyl-methyl)-5-(4-methoxy-phenyl)-cyclopentane-1,1,3,3-tetracarboxylic acid tetramethyl ester (5n)



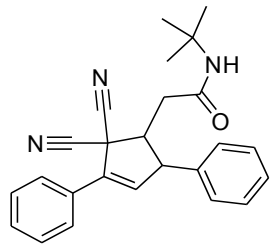
Compound **5n** was prepared according to the general procedure D. Purification by flash chromatography with with a gradient AcOEt/PE (from 10/90 to 30/70) as eluant gave the desired product (99 mg, 58%) as an uncolored oil. **CCM** Rf (50/50 AcOEt/EP) = 0.33 ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.21 (d, J=8.8Hz, 2H), 6.78 (d, J=8.8Hz, 2H), 5.71 (br s, 1H), 3.85 (d, J=13.2 Hz, 1H), 3.80 (s, 3H), 3.74 (s, 6H), 3.73 (s, 3H), 3.47 (m, 1H), 3.18 (s, 3H), 3.15 (d, J=14.8 Hz, AB system, 1H), 2.96 (d, J=14.8 Hz, AB system, 1H), 2.41 (dd, J=14.8, 6.8Hz, 2H), 1.95 (dd, J=15.2, 6.8Hz, 2H), 1.17 (s, 9H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 171.9 (Cq), 171.3 (Cq), 171.0 (Cq), 170.7 (Cq), 169.7 (Cq), 159.0 (Cq), 130.34 (2CH), 128.2 (Cq), 113.6 (2CH), 63.3 (Cq), 61.2 (Cq), 55.2 (CH_3), 54.3 (CH), 53.1 (CH_3), 52.9 (CH_3), 52.8 (CH_3), 52.6 (CH_3), 50.9 (Cq), 46.1 (CH), 41.5 (CH_2), 38.2 (CH_2), 28.6 ($3CH_3$) ; **IR** (υ, cm^{-1}) 3637, 3396, 2954, 2839, 1727, 1661, 1611, 1513, 1433, 1208, 1178, 1078 ; **HRMS** (EI) Calcd. for $C_{26}H_{35}NO_{10}$: 521.2261 found : 521.2265

2-(1-Phenyl-ethylidene)-malononitrile (6)



A mixture of acetophenone (5.6 mL, 1 eq), malononitrile (1 eq) and ammonium acetate (1.88 eq) in toluene (1 M) was stirred during 6h at reflux. The crude was diluted in Et_2O and extracted with water and dryied with MgSO4. The crude was purified by silica gel column chromatography with Et_2O in EP (20:80), to afford 2.19 g of pale yellow solid (27% yield). **CCM** Rf (20/80 Et_2O/EP) = 0.26 ; **Mp** 98 – 99°C ; **^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.54 (m, 5H), 2.64 (s, 3H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 175.6 (Cq), 135.9 (Cq), 132.3 (CH), 129.2 (2CH), 127.4 (2CH), 112.9 (Cq), 112.8 (Cq), 84.7 (Cq), 24.3 (CH_3) ; **IR** (υ, cm^{-1}) 3067, 2226, 1583, 1564, 1491, 1440, 1376, 1306, 1190, 1051 ; **HRMS** (EI) Calcd. for $C_{11}H_8N_2$: 168.0687 found : 168.0695

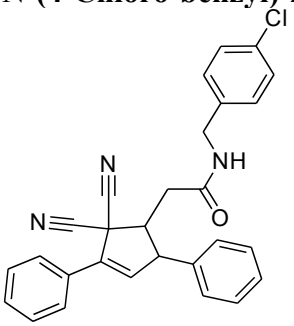
N-tert-Butyl-2-(2,2-dicyano-3,5-diphenyl-cyclopent-3-enyl)-acetamide (7c)



A mixture of Passerini adduct **1c** (200 mg, 1 eq), malonitrile derivative **6** (1 eq), cesium carbonate (1.2 eq) and tetrakis(triphenylphosphine)palladium (0.05 eq) in 1.3 mL of toluene was stirred at 50°C during 30min. Then the crude was heated under micro-wave at 120°C during 30min.The crude was purified by silica gel column chromatography with a gradient of Et_2O in EP (20:80 to 30:70), to afford 84mg of yellow solid (30% yield). **CCM :** Rf (50/50 Et_2O/EP) = 0.35 ; **Mp** 77 – 78°C ; **^{1}H-NMR (δ, ppm) :** ($CDCl_3$, 400 MHz) 7.57 (d, J=7.2 Hz, 2H), 7.36 (m, 6H), 7.16 (d, J=7.6Hz, 2H), 6.34 (s, 1H), 5.48 (br s, 1H), 3.77 (d, J=9.2 Hz, 1H), 3.41 (qd, J=7.2Hz, 1H), 2.61 (d, J=7.2Hz, 2H), 1.26 (s, 9H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) : 166.4 (Cq), 138.0 (Cq), 136.7 (Cq), 135.5 (CH), 129.8 (Cq), 128.5 (CH), 128.2 (2CH), 128.0 (2CH), 127.1 (CH), 126.9 (2CH), 125.3 (2CH), 113.4 (Cq), 112.4 (Cq), 54.8 (CH), 54.2 (CH), 50.8 (Cq), 44.0 (Cq), 36.7 (CH_2), 27.6 ($3CH_3$) ;**IR** (υ, cm^{-1}) 3374, 2965, 1732, 1650, 1532, 1494, 1454, 1363, 1263, 1219,

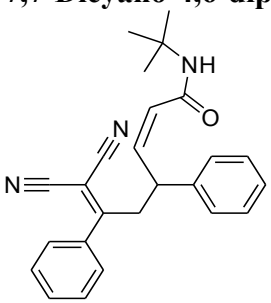
1028 ; **HRMS** (EI) Calcd. for $C_{25}H_{25}N_3O$: 383.1998 found : 383.1999

N-(4-Chloro-benzyl)-2-(2,2-dicyano-3,5-diphenyl-cyclopent-3-enyl)-acetamide (7d)

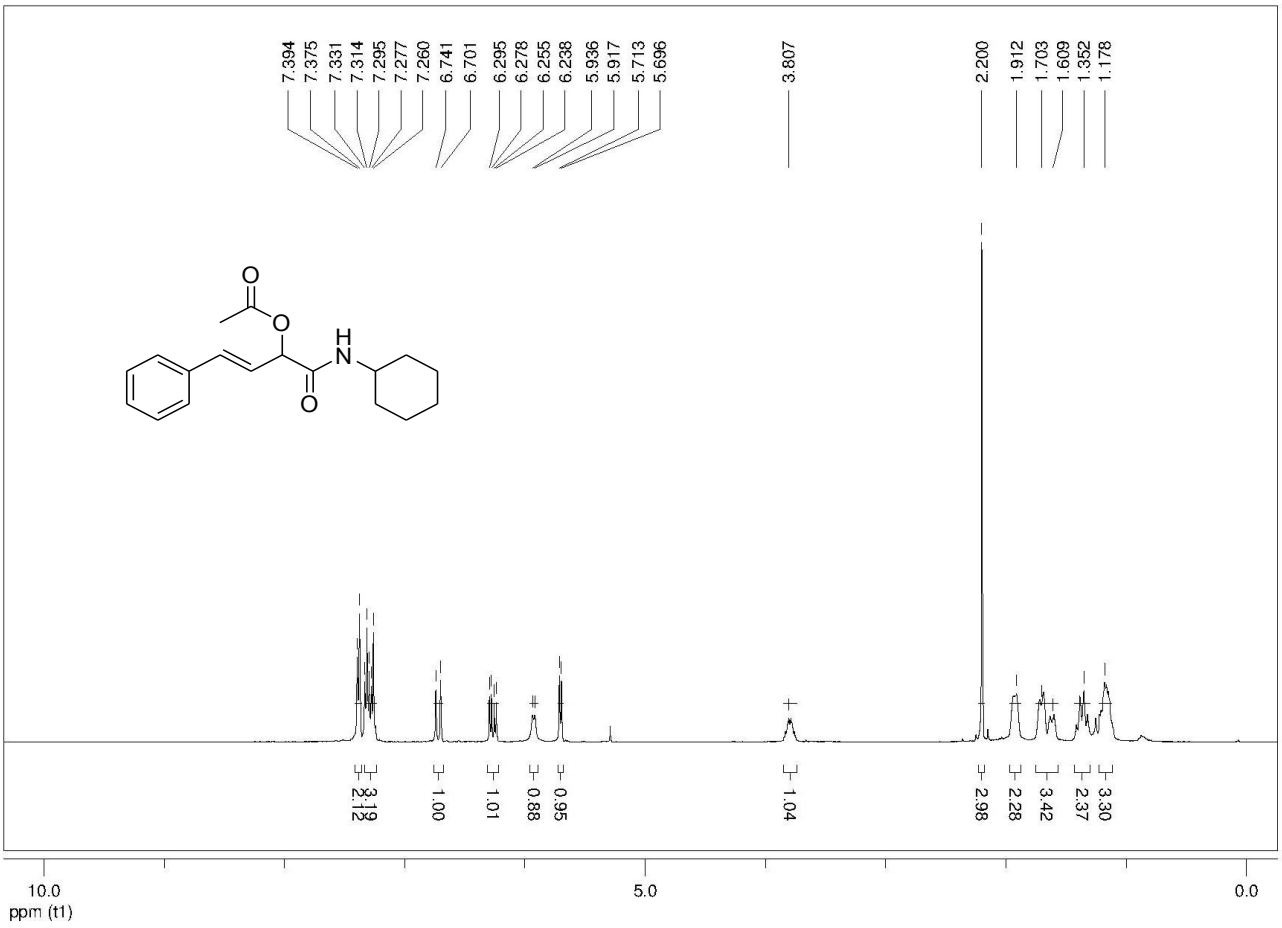


A mixture of Passerini adduct **1d** (100 mg, 1 eq), malonitrile derivative **6** (1 eq), cesium carbonate (1.2 eq) and tetrakis(triphenylphosphine)palladium (0.05 eq) in 1.3 mL of toluene was stirred at 50°C during 30min. The crude was purified by silica gel column chromatography with a gradient of AcOEt in EP (20:80 to 40:60), to afford 69mg of green solid (62% yield). **CCM** Rf (40/60 AcOEt/EP) = 0.59 ; **Mp** 153 – 155°C **; ^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.65 (d, J=7.6 Hz, 2H), 7.45 (m, 4H), 7.39 (m, 4H), 7.29 (d, J=8.4Hz, 2H), 7.20 (d, J=8.4 Hz, 2H), 6.42 (s, 1H), 5.90 (br s, 1H), 4.40 (d, J=5.6 Hz, 2H), 3.89 (dd, J=9.2, 2.0 Hz, 1H), 3.49 (dt, J=9.2, 5.2 Hz, 2H), 2.80 (dq, J=15.2, 9.6Hz, 2H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 168.5 (Cq), 139.0 (Cq), 137.8 (Cq), 136.5 (CH), 136.3 (Cq), 133.4 (Cq), 130.8 (Cq), 129.7 (CH), 129.4 (2CH), 129.3 (2CH), 129.1 (2CH), 128.9 (2CH), 128.3 (CH), 128.0 (2CH), 126.4 (2CH), 114.6 (Cq), 113.3 (Cq), 55.7 (CH), 55.2 (CH), 45.3 (Cq), 43.1 (CH_2), 36.5 (CH_2) ; **IR** (υ, cm^{-1}) 3292, 3058, 2925, 1741, 1650, 1539, 1491, 1445, 1263, 1226, 1090, 1014 ; **HRMS** (EI) Calcd. for $C_{28}H_{22}ClN_3O$: 451.1451 found : 451.1459

7,7-Dicyano-4,6-diphenyl-hepta-2,6-dienoic acid tert-butylamide (8c)



A mixture of Passerini adduct **1c** (100 mg, 1 eq), malonitrile derivative **6** (1 eq), cesium carbonate (1.2 eq) and tetrakis(triphenylphosphine)palladium (0.05 eq) in 1.3 mL of toluene was stirred at 50°C during 30min. The crude was purified by silica gel column chromatography with a gradient of Et_2O in EP (20:80 to 50:50), to afford 82 mg of yellow solid (59% yield). **CCM** Rf (70/30 Et_2O/EP) = 0.32 **Mp** 75 – 77°C **; ^{1}H-NMR (δ, ppm)** ($CDCl_3$, 400 MHz) 7.54 (d, J=7.2 Hz, 1H), 7.48 (t, J=6.8 Hz, 2H), 7.29 (m, 5H), 6.99 (d, J=7.6 Hz, 2H), 6.84 (dd, J=15.2, 6.0 Hz, 1H), 5.54 (d, J=15.2 Hz, 1H), 5.23 (br s, 1H), 3.48 (dd, J=14.8, 11.2Hz, 1H), 3.40 (m, 2H), 1.35 (s, 9H) ; **^{13}C-NMR (δ, ppm)** ($CDCl_3$, 100.6 MHz) 177.0 (Cq), 164.2 (Cq), 143.06 (CH), 138.6 (Cq), 134.3 (Cq), 132.2 (CH), 129.3 (2CH), 129.0 (2CH), 127.9 (2CH), 127.6 (2CH), 125.9 (CH), 112.5 (Cq), 112.4 (Cq), 86.5 (Cq), 51.5 (Cq), 46.8 (CH), 42.6 (CH_2), 28.7 ($3CH_3$) ; **IR** (υ, cm^{-1}) 3290, 2965, 2925, 2228, 1667, 1629, 1540, 1453, 1391, 1362, 1265, 1222, 1077 ; **HRMS** (EI) Calcd. for $C_{25}H_{25}N_3O$: 383.1998 found : 383.1992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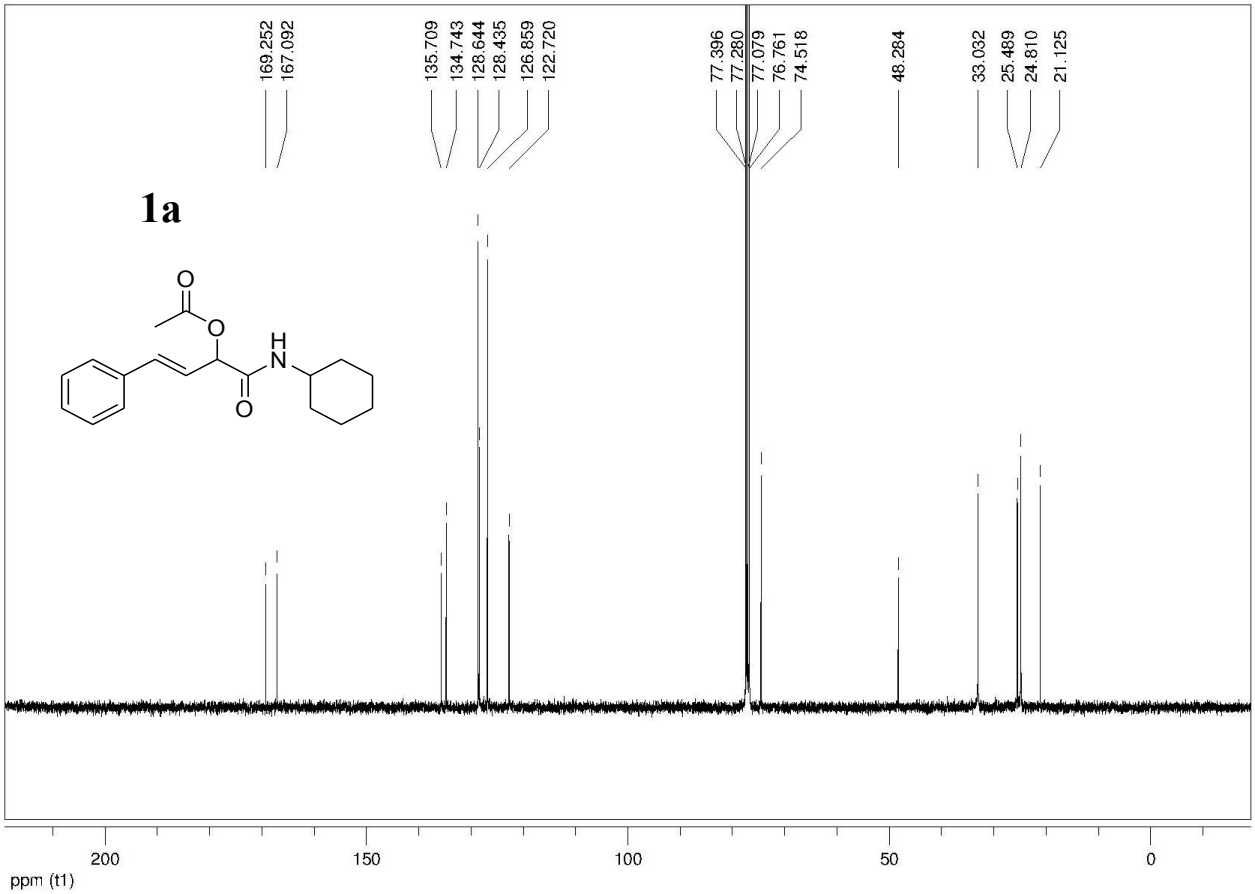
7.331
7.314
7.295
7.277
7.260
6.741
6.701
6.295
6.278
6.255
6.238
5.936
5.917
5.713
5.696
3.807
2.200
1.912
1.703
1.609
1.352
1.178
2.19
1.00
1.01
0.88
0.95
1.04
2.98
2.28
3.42
2.37
3.30
10.0
5.0
0.0
ppm (t1)

1a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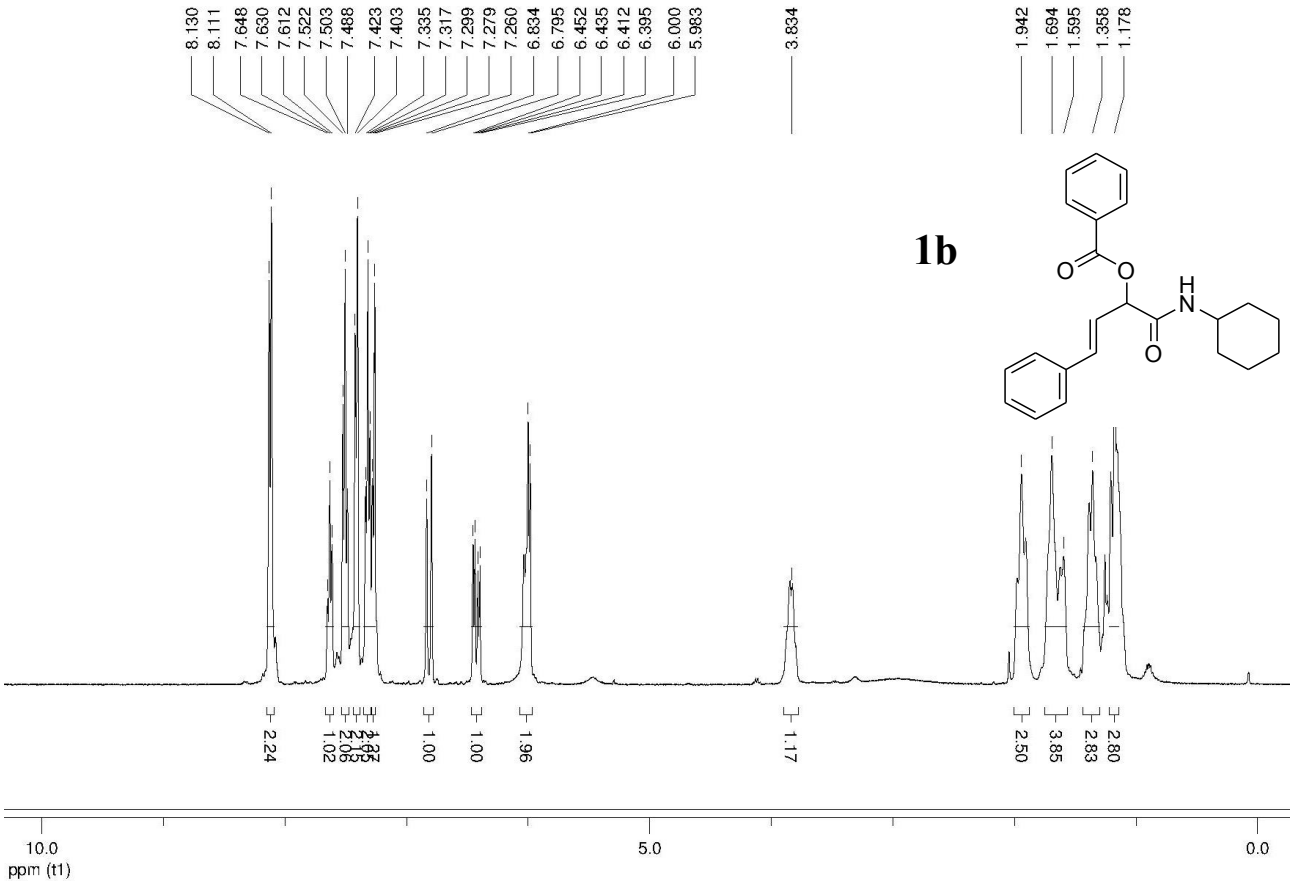
167.092
135.709
134.743
128.644
128.435
126.859
122.720
77.396
77.280
77.079
76.761
74.518
48.284
33.032
25.489
24.810
21.125
200
150
100
50
0
ppm (t1)


8.130
8.111
7.648
7.630
7.612
7.522
7.503
7.488
7.423
7.403
7.335
7.317
7.299
7.279
7.260
6.834
6.795
6.452
6.435
6.412
6.395
6.000
5.983
3.834
1.942
1.694
1.595
1.358
1.178
1b
2.24
1.02
1.00
1.00
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1.17
2.50
3.85
2.83
2.80
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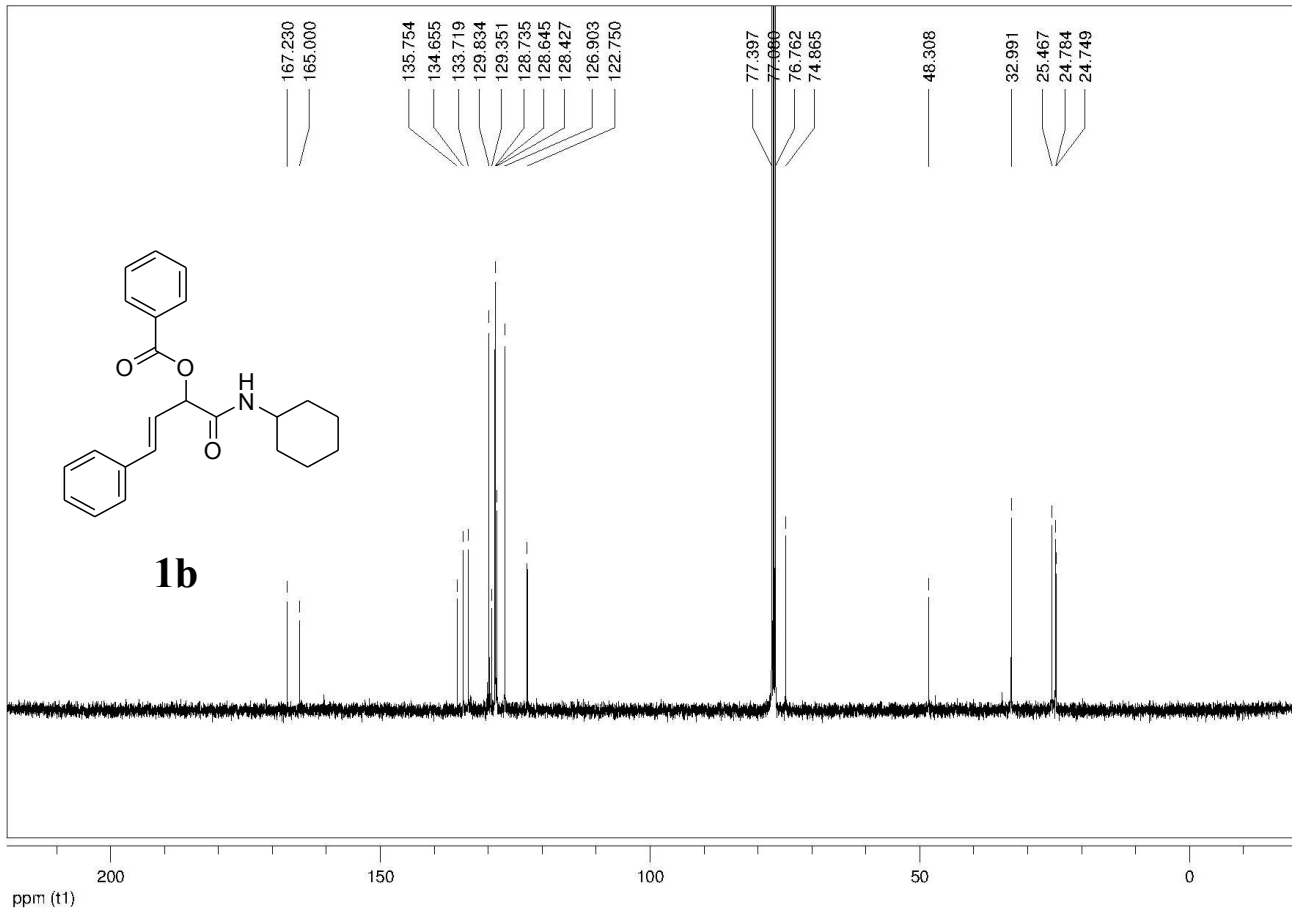
5.0
0.0
ppm (t1)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134.655
133.719
129.834
129.351
128.735
128.645
128.427
126.903
122.750
77.397
77.000
76.762
74.865
48.308
32.991
25.467
24.784
24.749
1b
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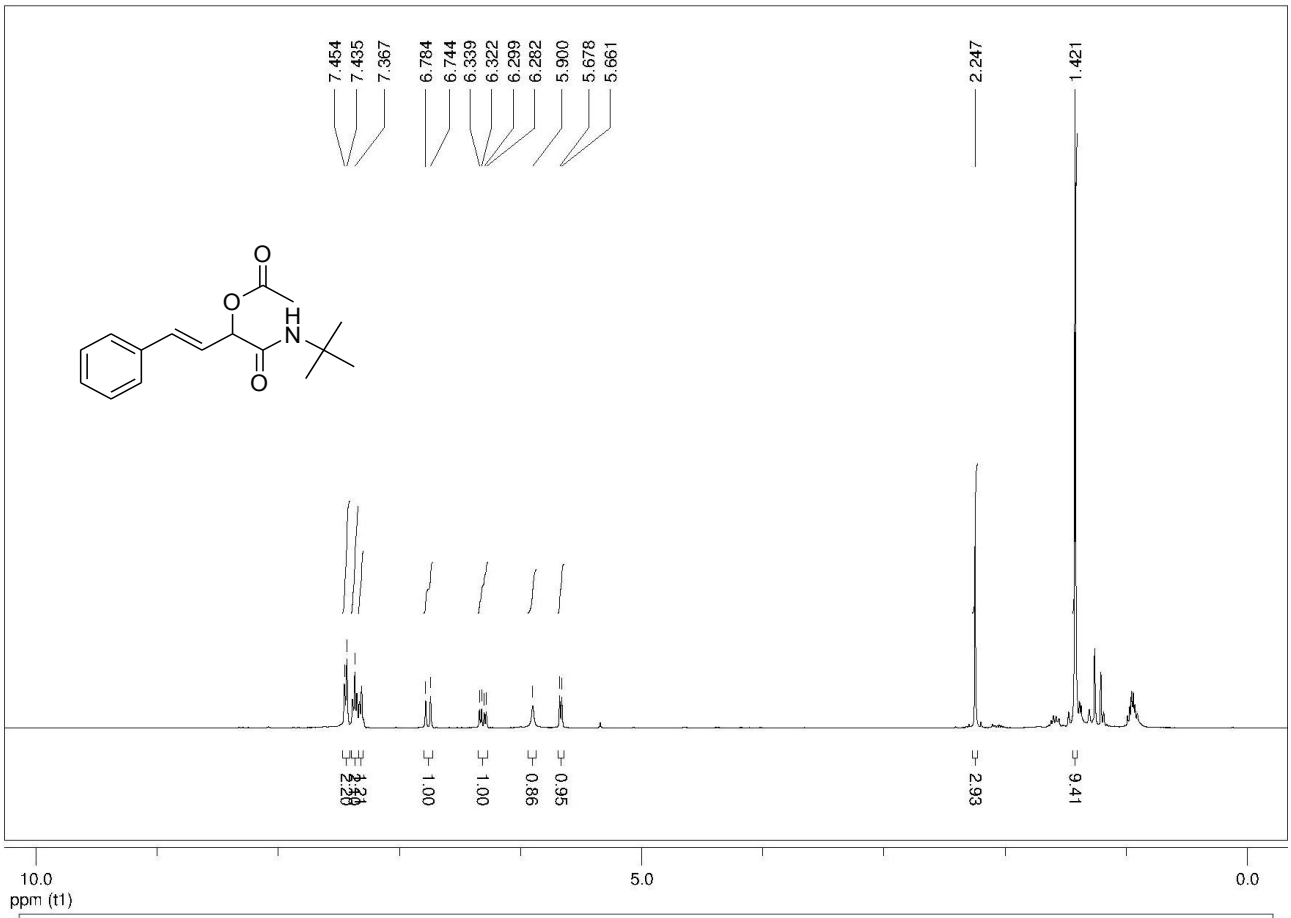
150
100
50
0
ppm (t1)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6.744
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6.322
6.299
6.282
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5.678
5.661
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1.421
1.00
1.00
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0.96
2.93
9.41
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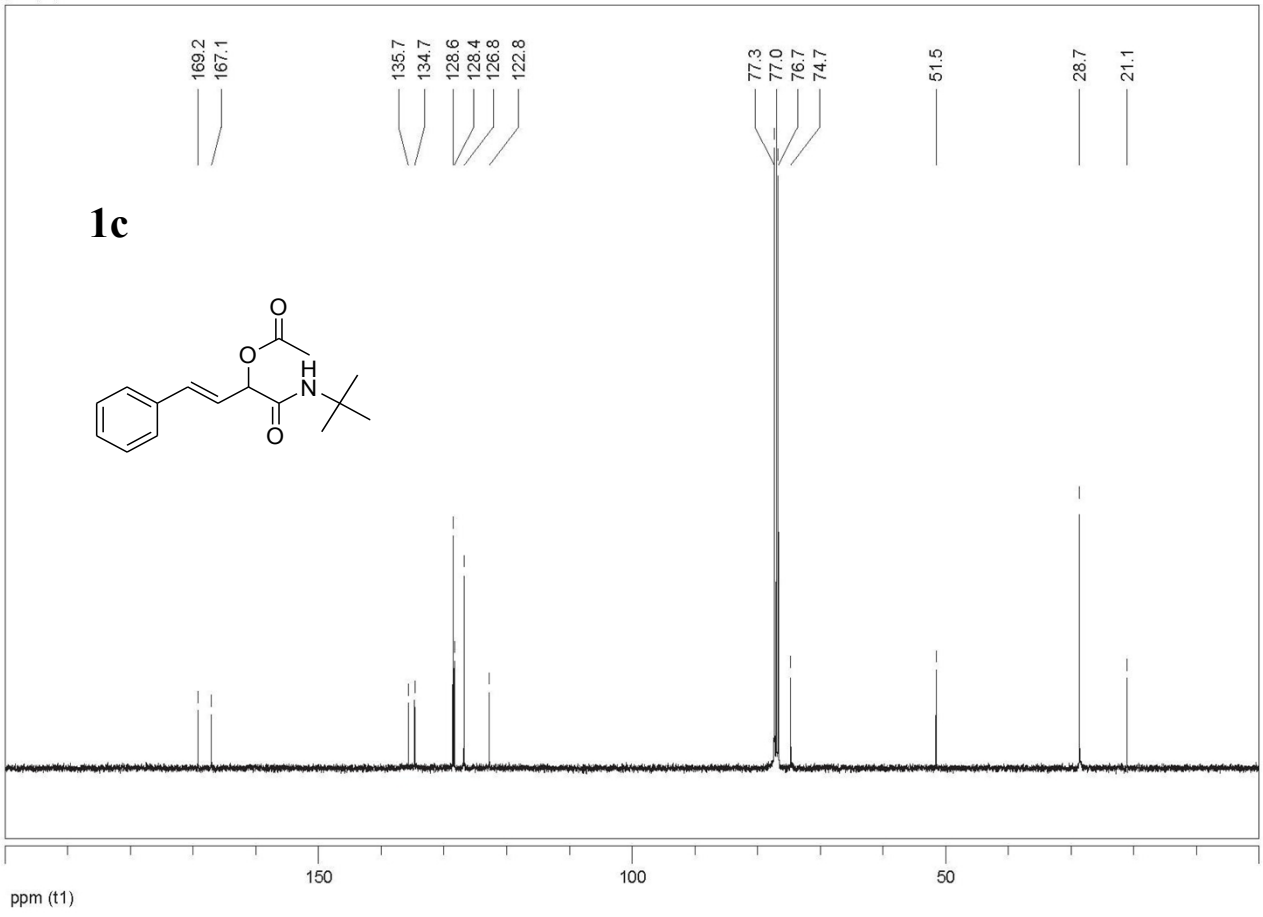
ppm (t1)
5.0
0.0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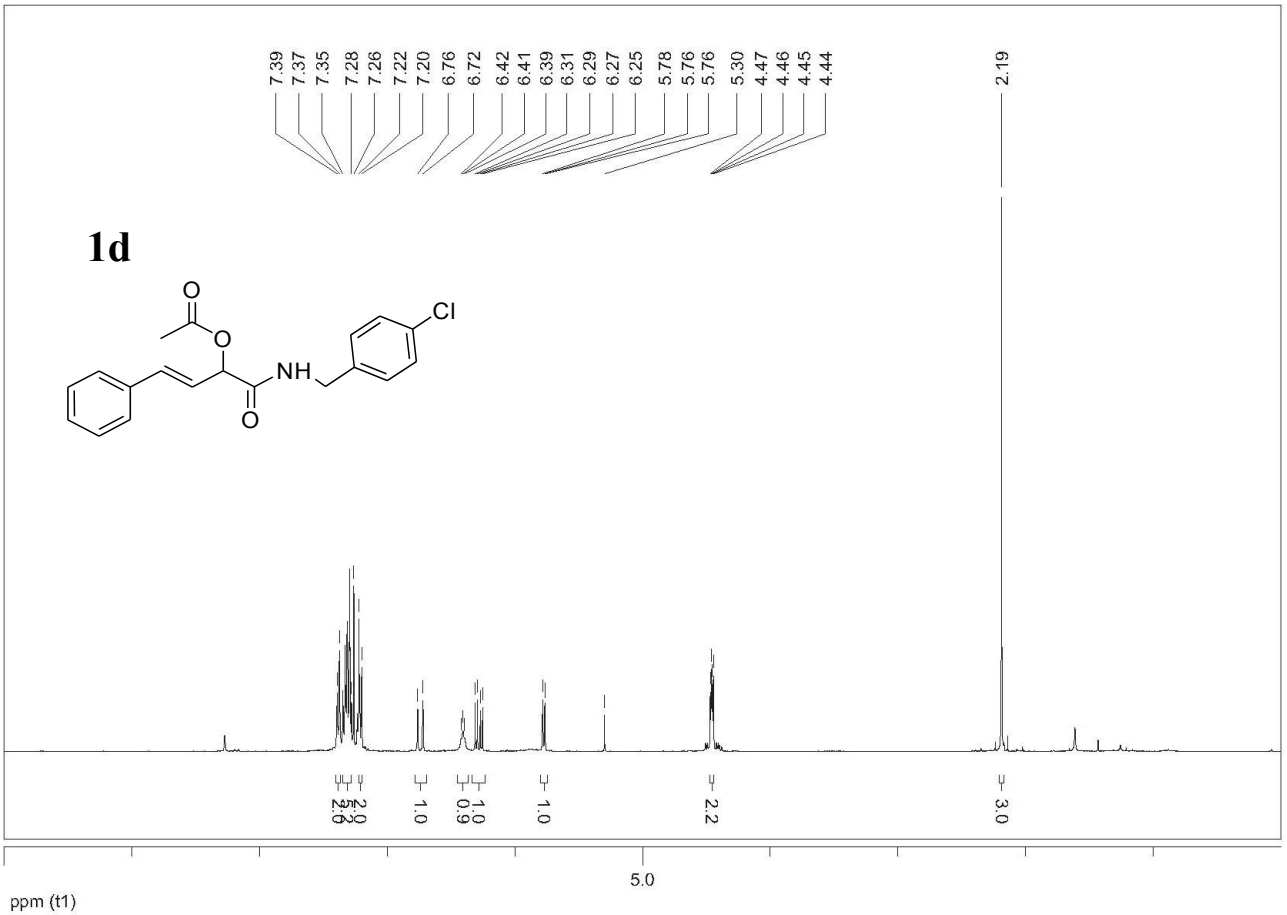
135.7
134.7
128.6
128.4
126.8
122.8
77.3
77.0
76.7
74.7
51.5
28.7
21.1
150
100
50
ppm (t1)

1c

1c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7.26
7.22
7.20
6.76
6.72
6.42
6.41
6.39
6.31
6.29
6.27
6.25
5.78
5.76
5.76
5.30
4.47
4.46
4.45
4.44
2.19
1d
O
O
NH
O
Cl
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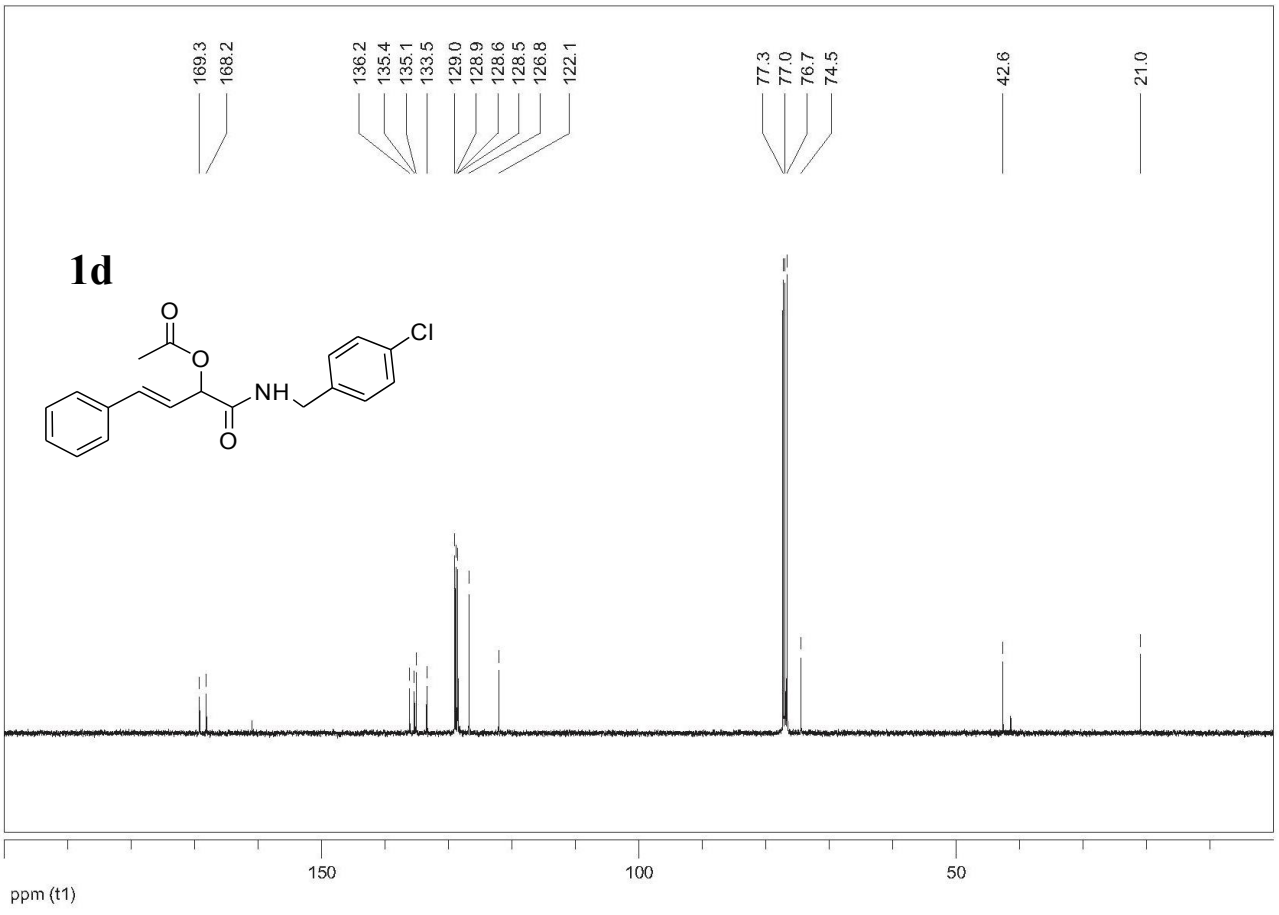
1.0
0.9
1.0
1.0
2.2
3.0
5.0
ppm (t1)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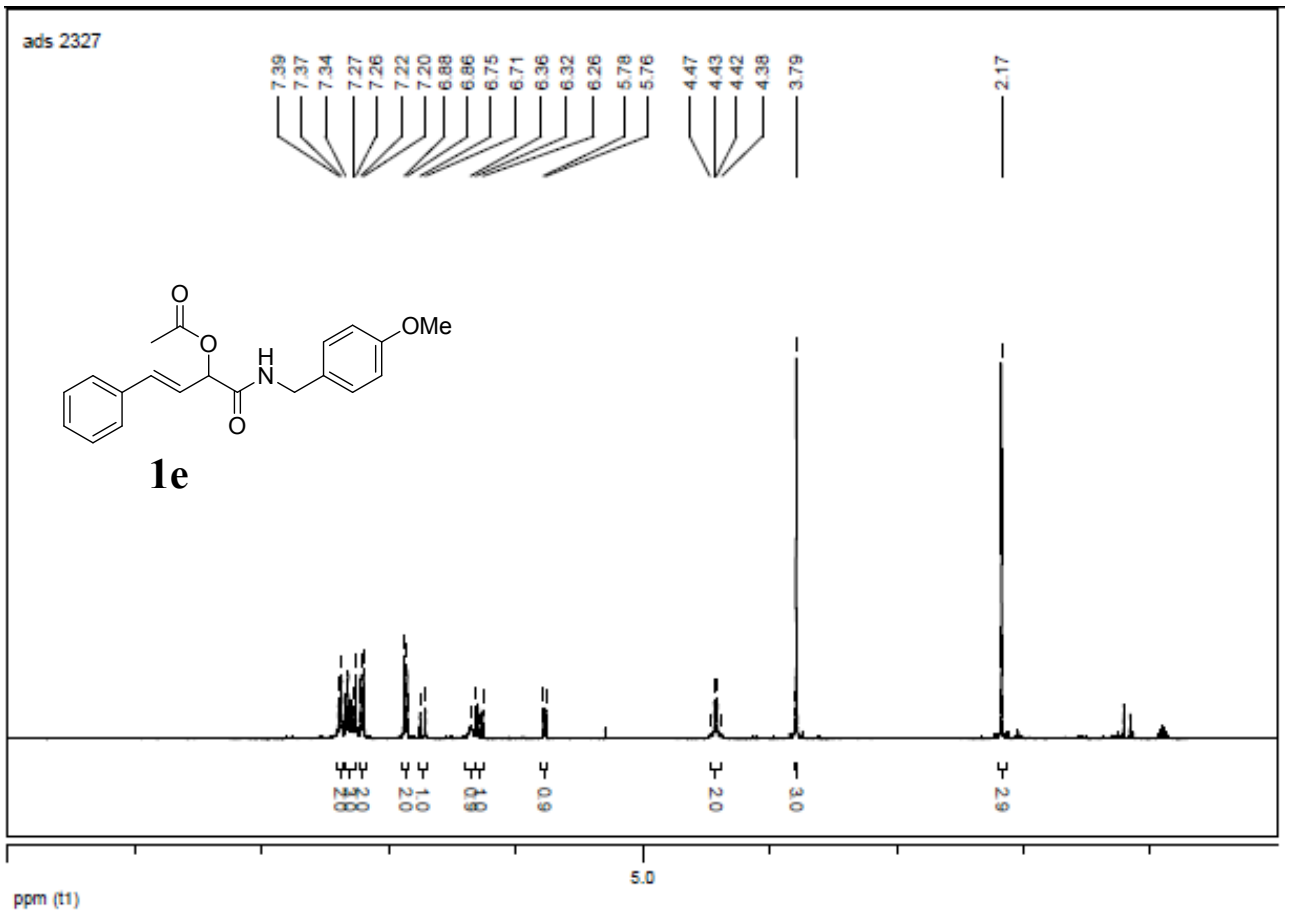
168.2
136.2
135.4
135.1
133.5
129.0
128.9
128.6
128.5
126.8
122.1
77.3
77.0
76.7
74.5
42.6
21.0
1d
O
O
NH
O
Cl
150
100
50
ppm (t1)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7.26
7.22
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6.86
6.75
6.71
6.36
6.32
6.26
5.78
5.76
4.47
4.43
4.42
4.38
3.79
2.17
O
O
H
N
O
OMe
1e
2.29
3.30
2.20
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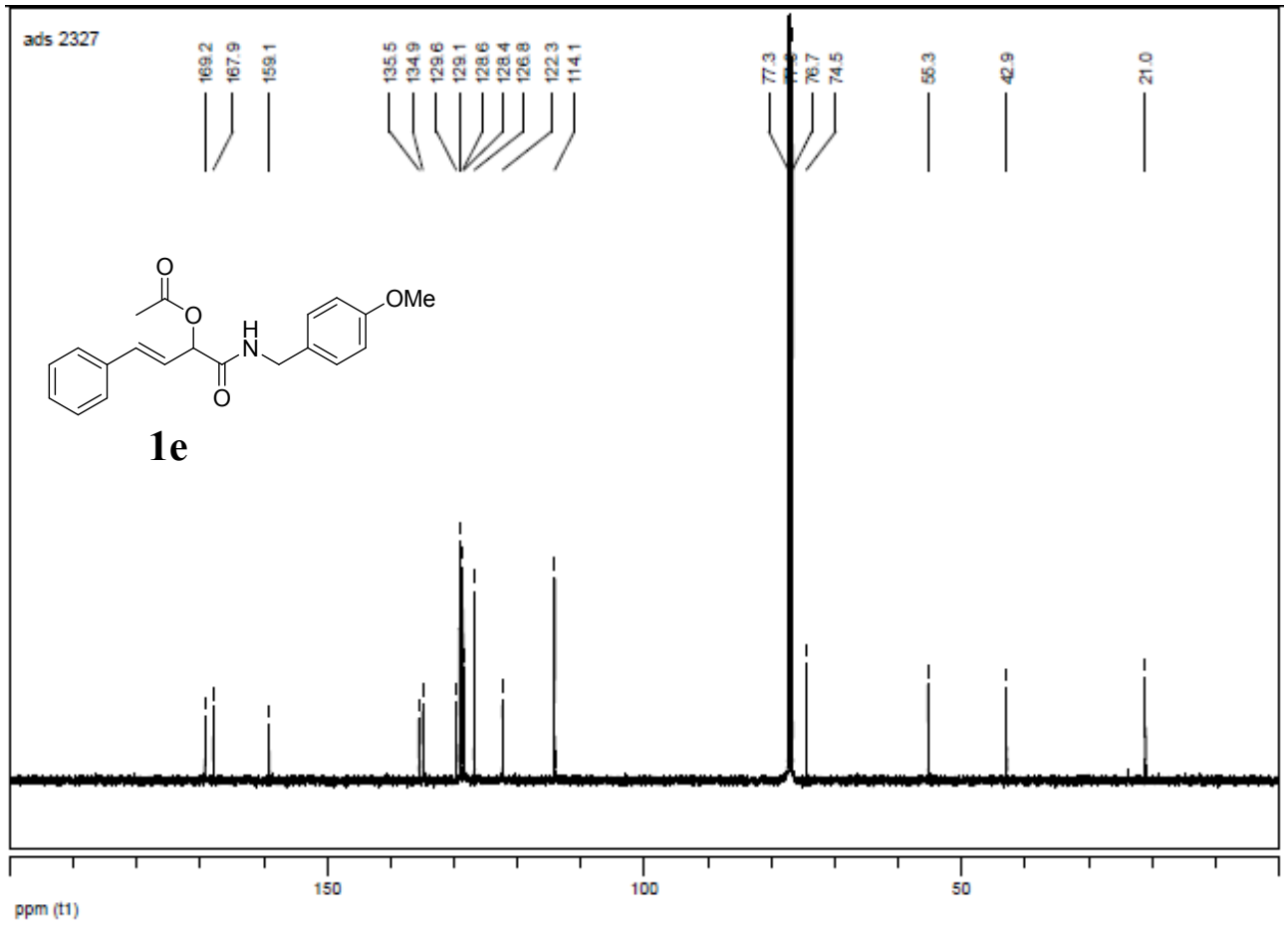
1.20
2.20
5.0
ppm (t1)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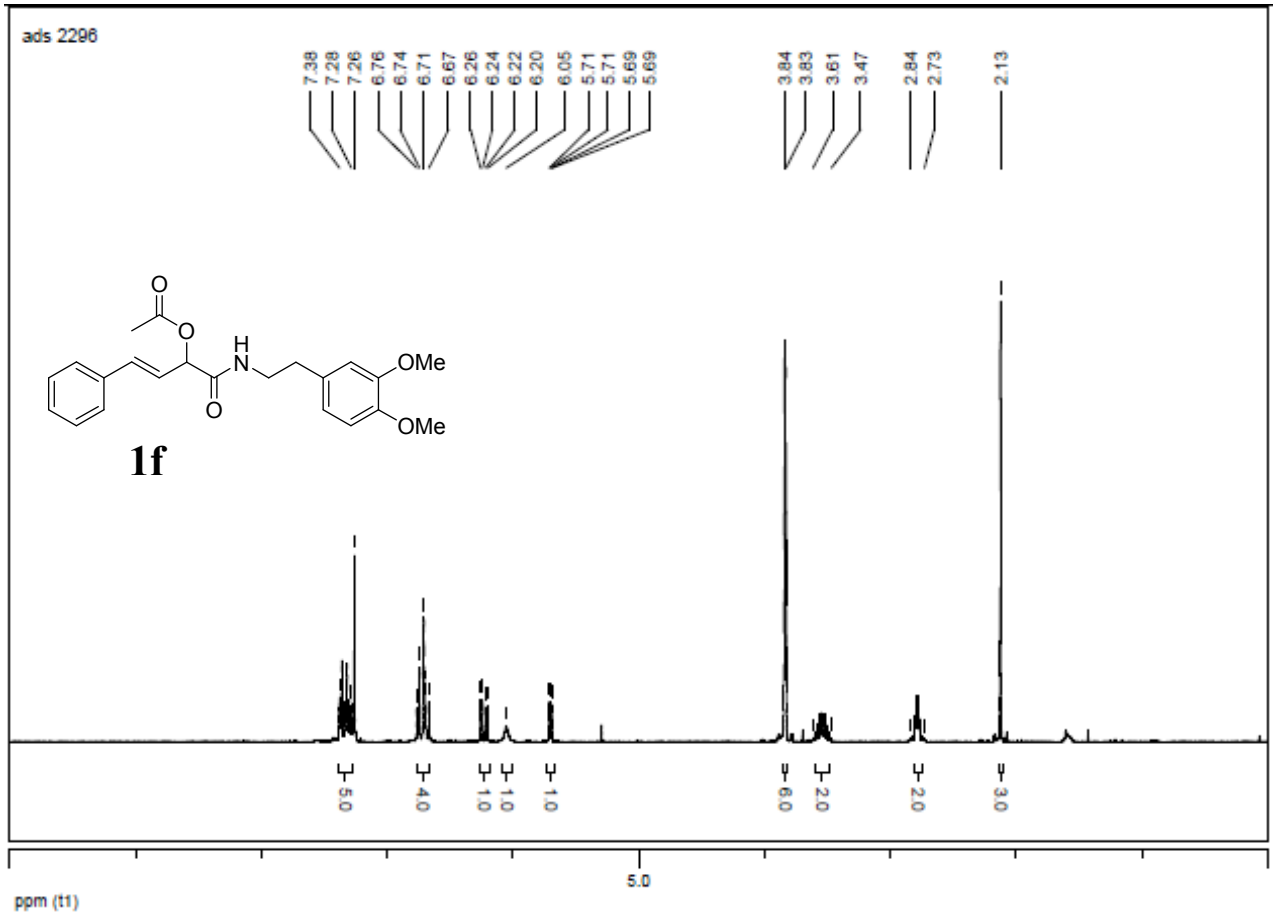
167.9
159.1
135.5
134.9
129.6
129.1
128.6
128.4
126.8
122.3
114.1
77.3
76.7
74.5
55.3
42.9
21.0
O
O
H
N
O
OMe
1e
150
100
50
ppm (t1)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7.28
7.26
6.76
6.74
6.71
6.67
6.26
6.24
6.22
6.20
6.05
5.71
5.71
5.69
5.69
3.84
3.83
3.61
3.47
2.84
2.73
2.13
O
O
H
N
O
OMe
OMe
1f
6.0
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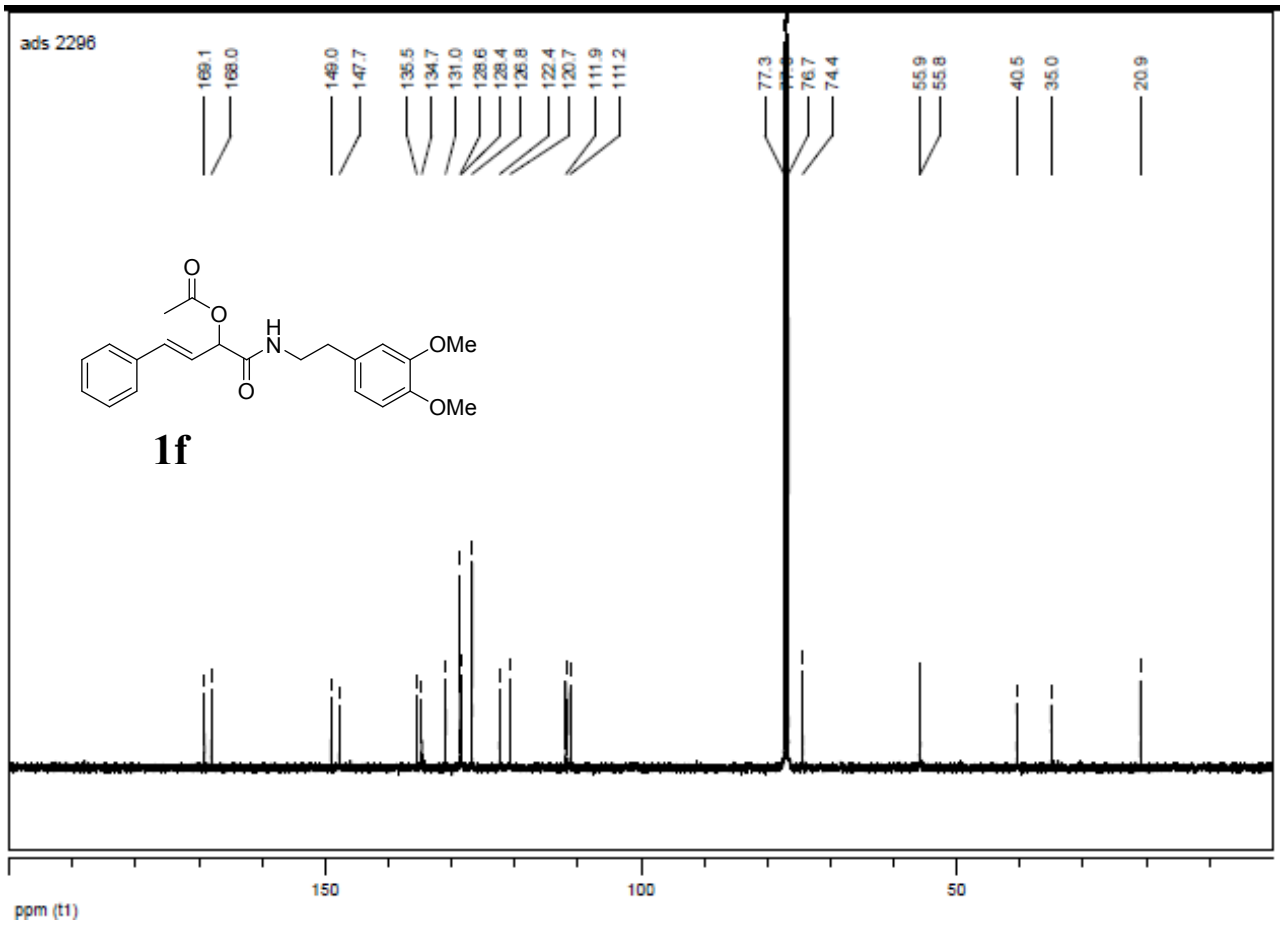
1.0
1.0
1.0
6.0
2.0
2.0
3.0
5.0
ppm (t1)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135.5
134.7
131.0
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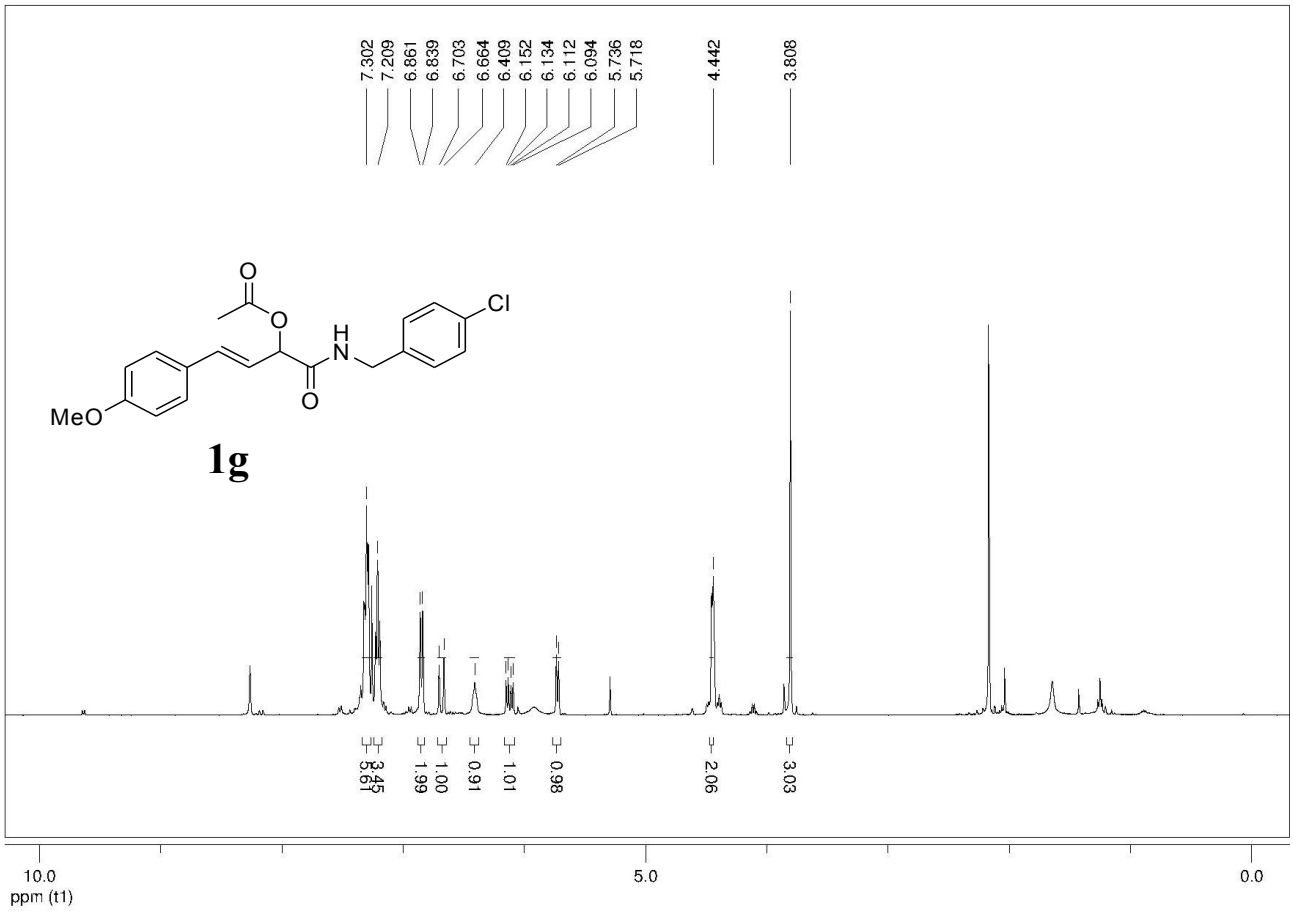
128.4
126.8
122.4
120.7
111.9
111.2
77.3
76.7
74.4
55.9
55.8
40.5
35.0
20.9
O
O
H
N
O
OMe
OMe
1f
150
100
50
ppm (t1)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6.839
6.703
6.664
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6.152
6.134
6.112
6.094
5.736
5.718
4.442
3.808
O
O
H
N
Cl
O
MeO
1g
5.61
3.45
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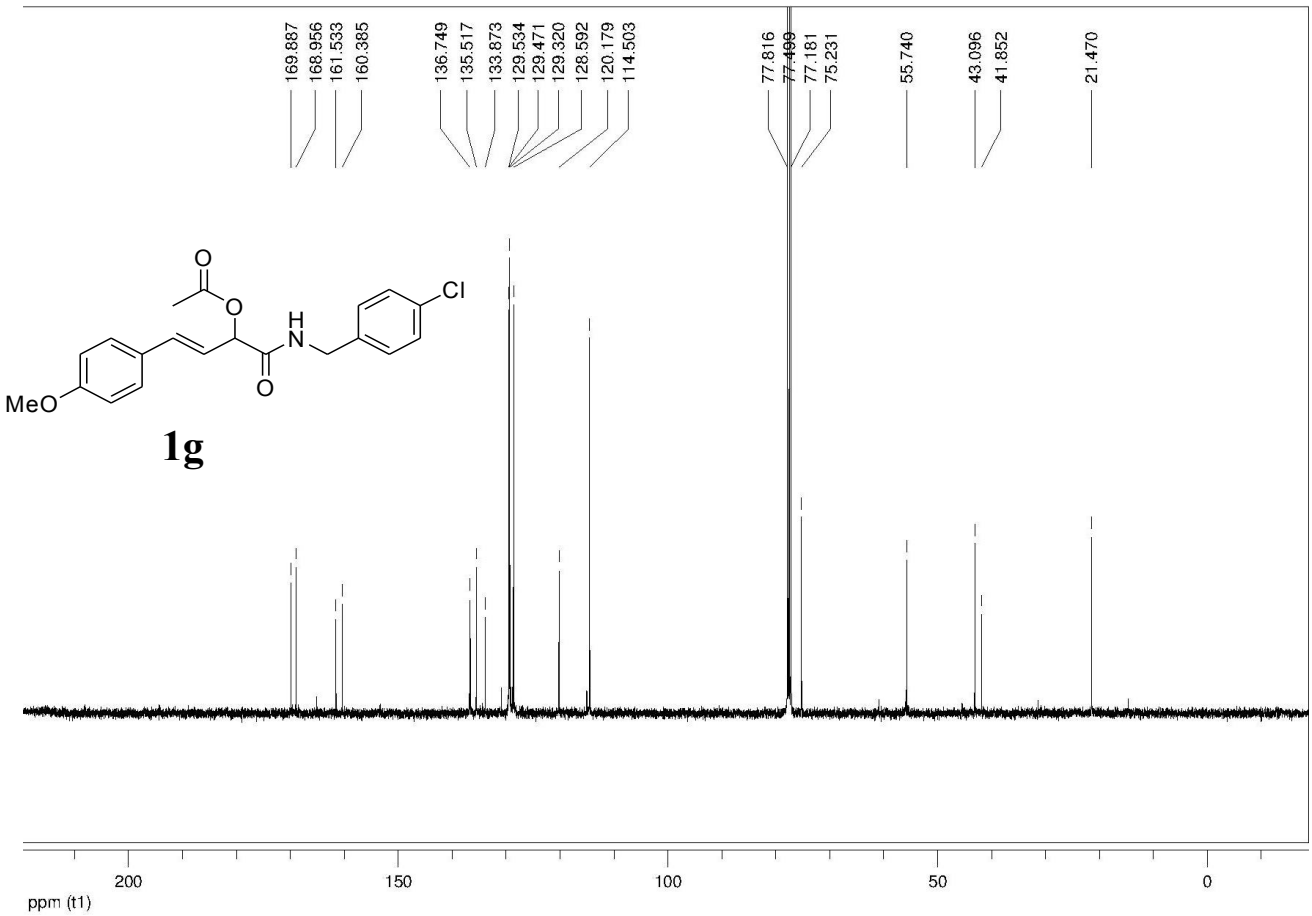
0.91
1.01
0.98
2.06
3.03
10.0
5.0
0.0
ppm (t1)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135.517
133.873
129.534
129.471
129.320
128.592
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114.503
77.816
77.499
77.181
75.231
55.740
43.096
41.852
21.470
O
O
H
N
Cl
O
MeO
1g
200
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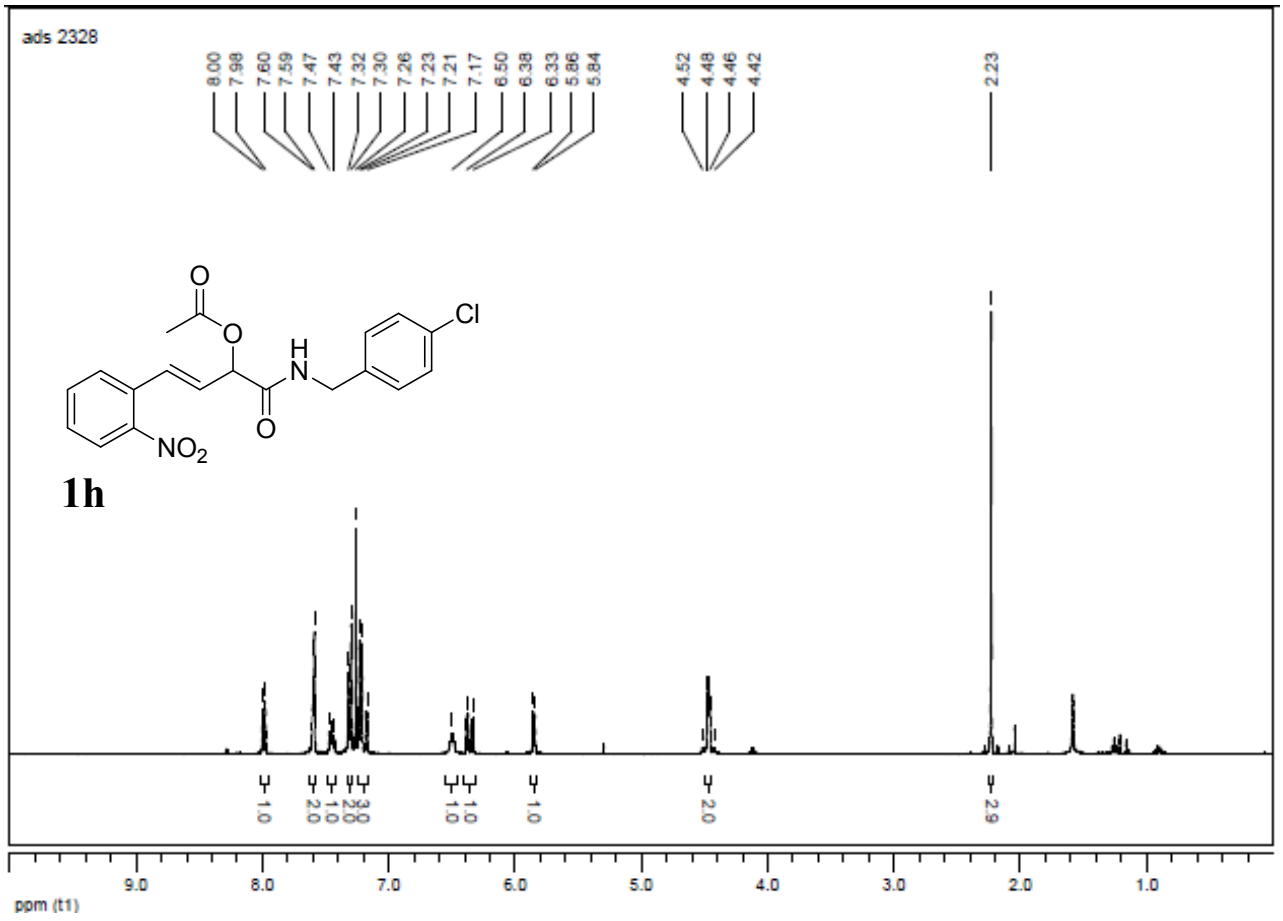
100
50
0
ppm (t1)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7.30
7.26
7.23
7.21
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6.38
6.33
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4.52
4.48
4.46
4.42
2.23
O
O
H
N
O
Cl
NO2
1h
1.0
2.0
2.0
3.0
1.0
1.0
1.0
2.0
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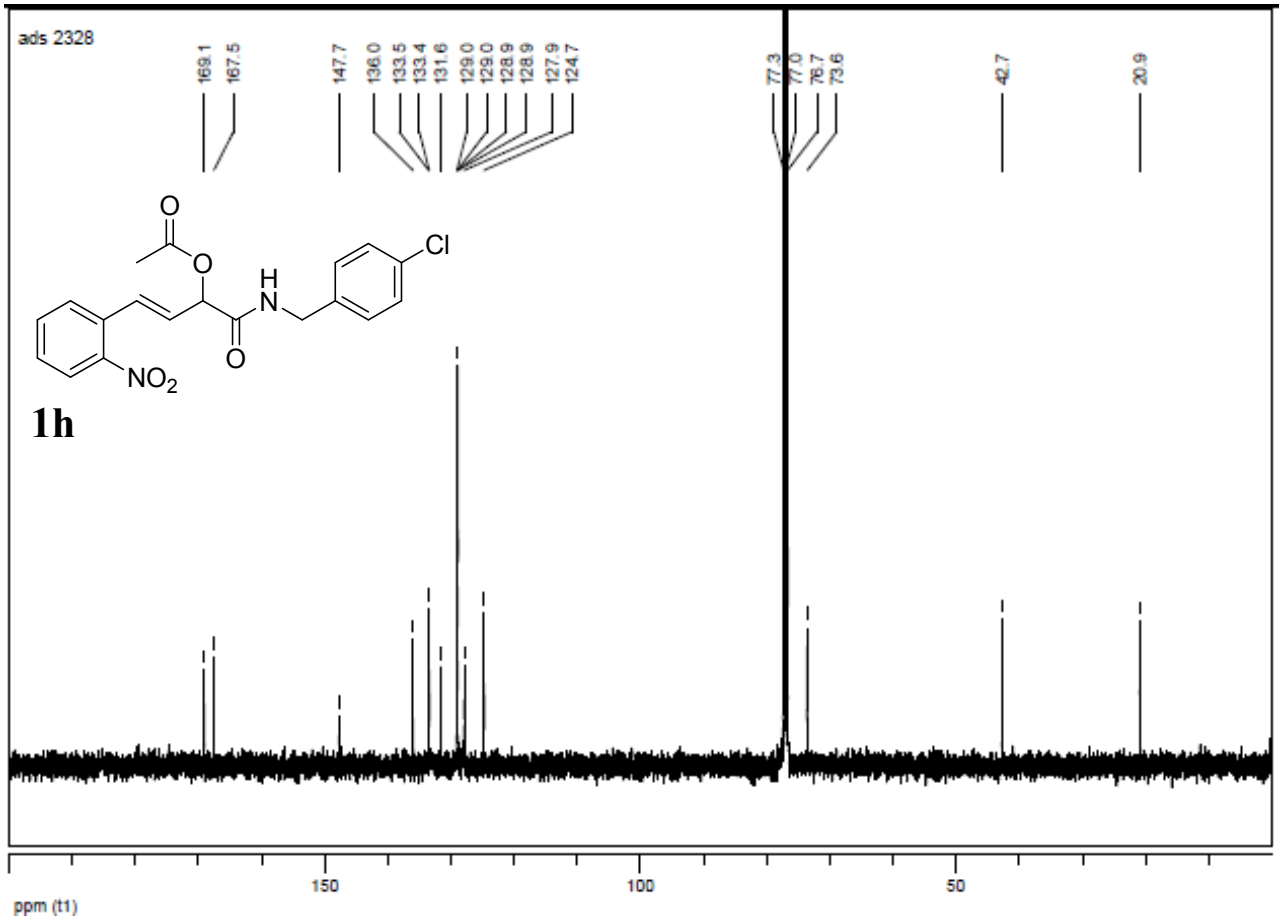
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)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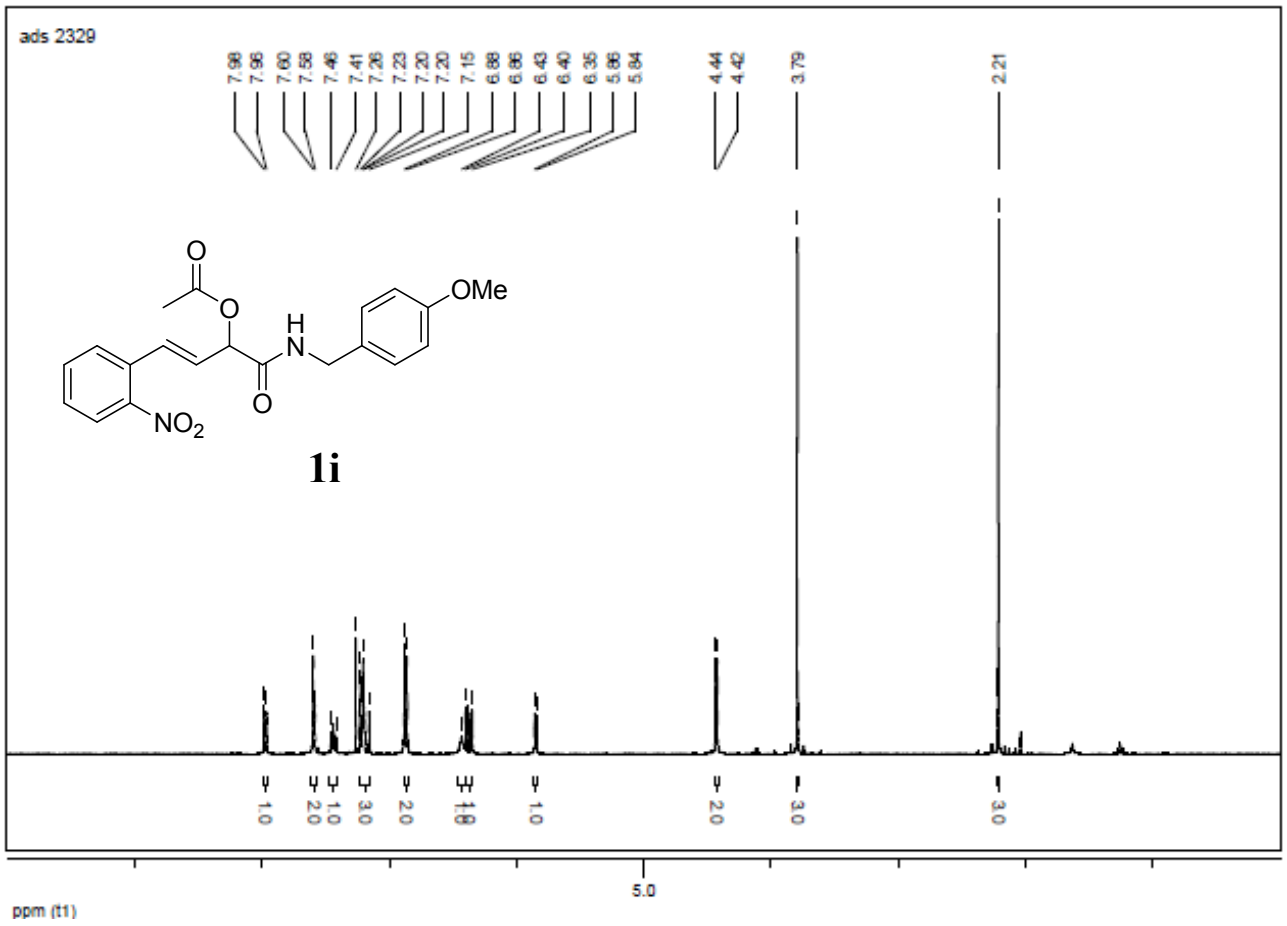
147.7
136.0
133.5
133.4
131.6
129.0
129.0
128.9
128.9
127.9
124.7
77.3
77.0
76.7
73.6
42.7
20.9
O
O
H
N
O
Cl
NO2
1h
150
100
50
ppm (t1)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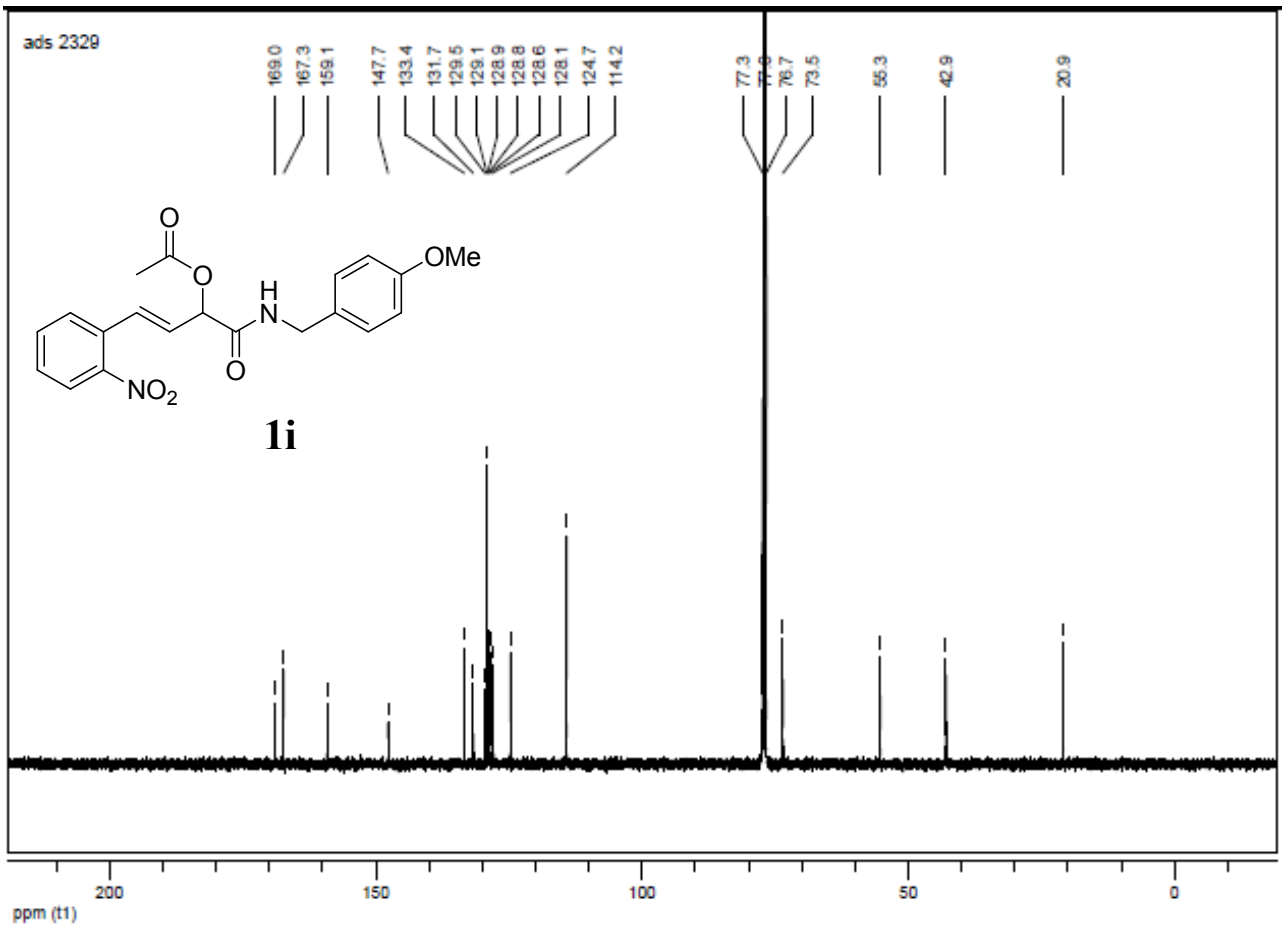
7.46
7.41
7.26
7.23
7.20
7.20
7.15
6.88
6.86
6.43
6.40
6.35
5.86
5.84
4.44
4.42
3.79
2.21
O
O
H
N
OMe
O
NO2
1i
10
20
10
30
20
18
10
20
30
30
5.0
ppm (t1)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129.5
129.1
128.9
128.8
128.6
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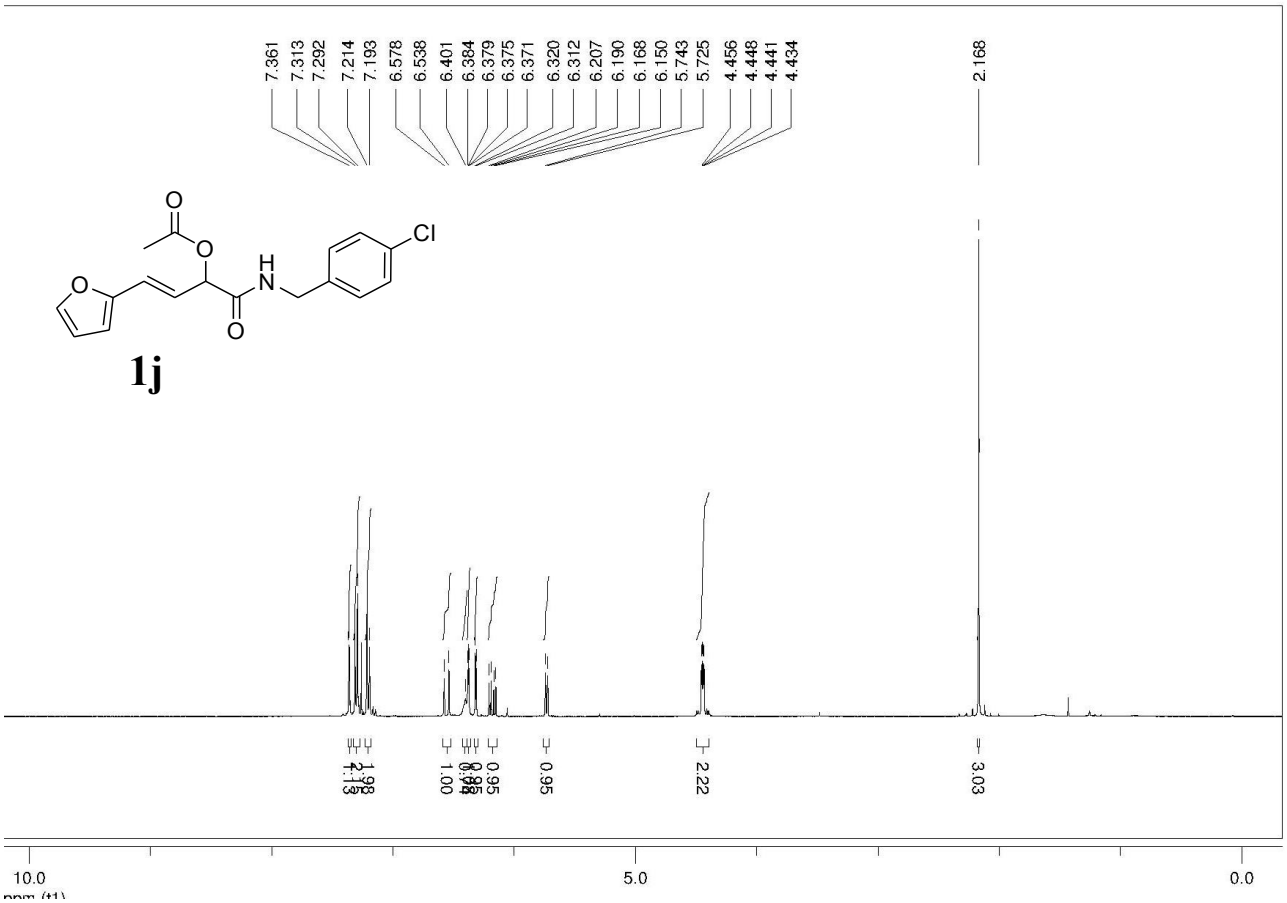
124.7
114.2
77.3
76.7
73.5
55.3
42.9
20.9
O
O
H
N
OMe
O
NO2
1i
200
150
100
50
0
ppm (t1)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4.448
4.441
4.434
2.168
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O
H
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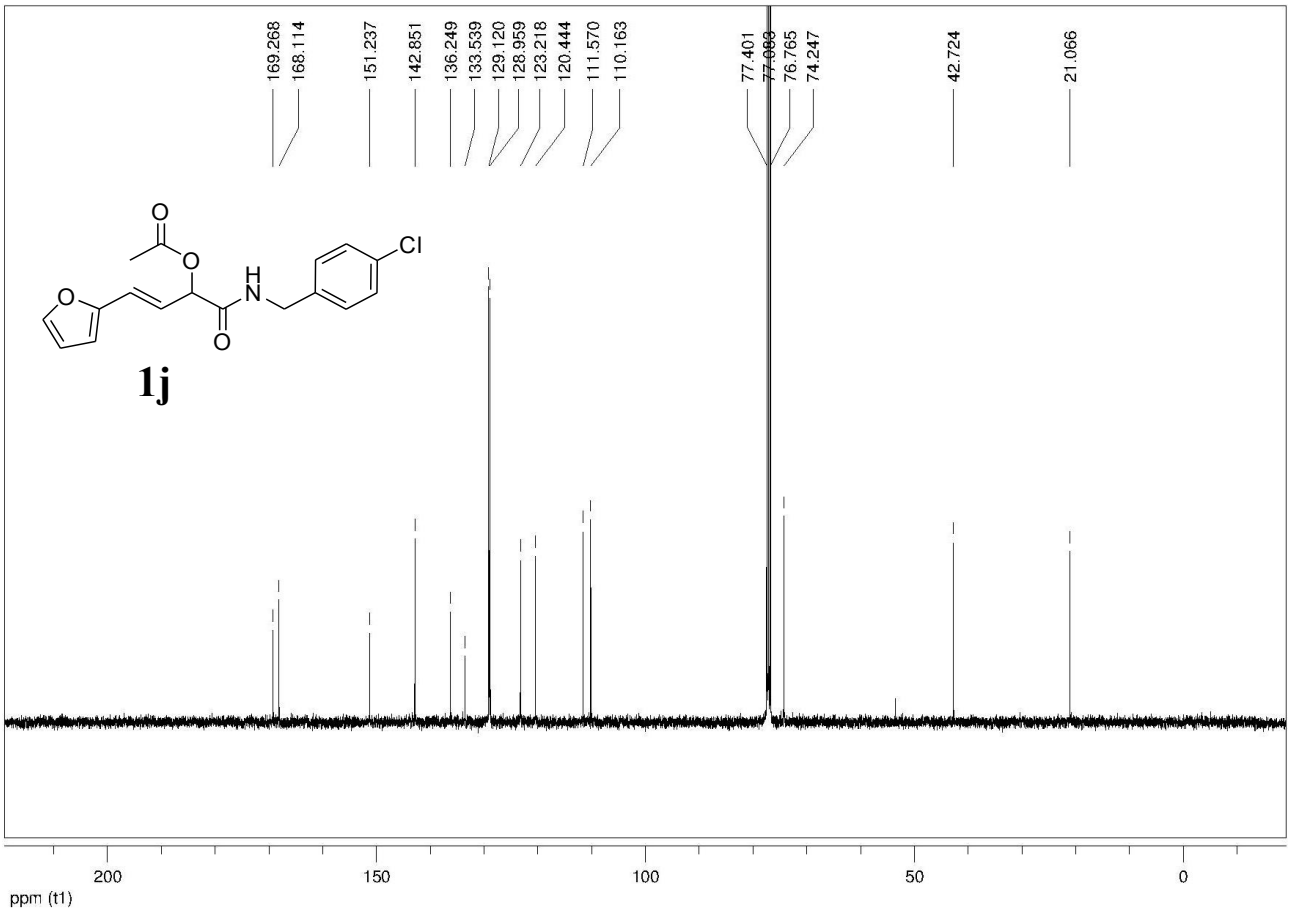
2.22
3.03
10.0
5.0
0.0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77.000
76.765
74.247
42.724
21.066
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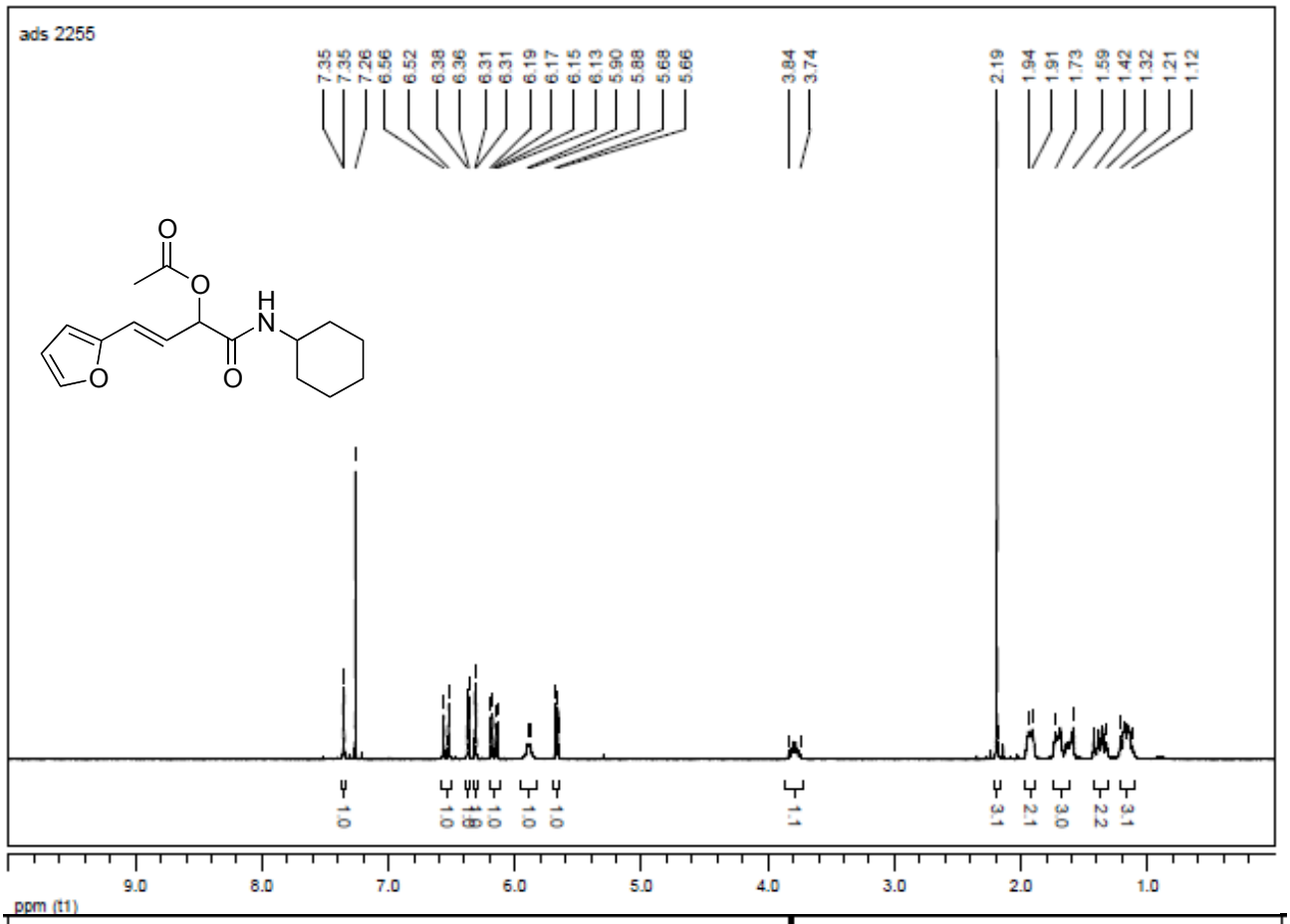
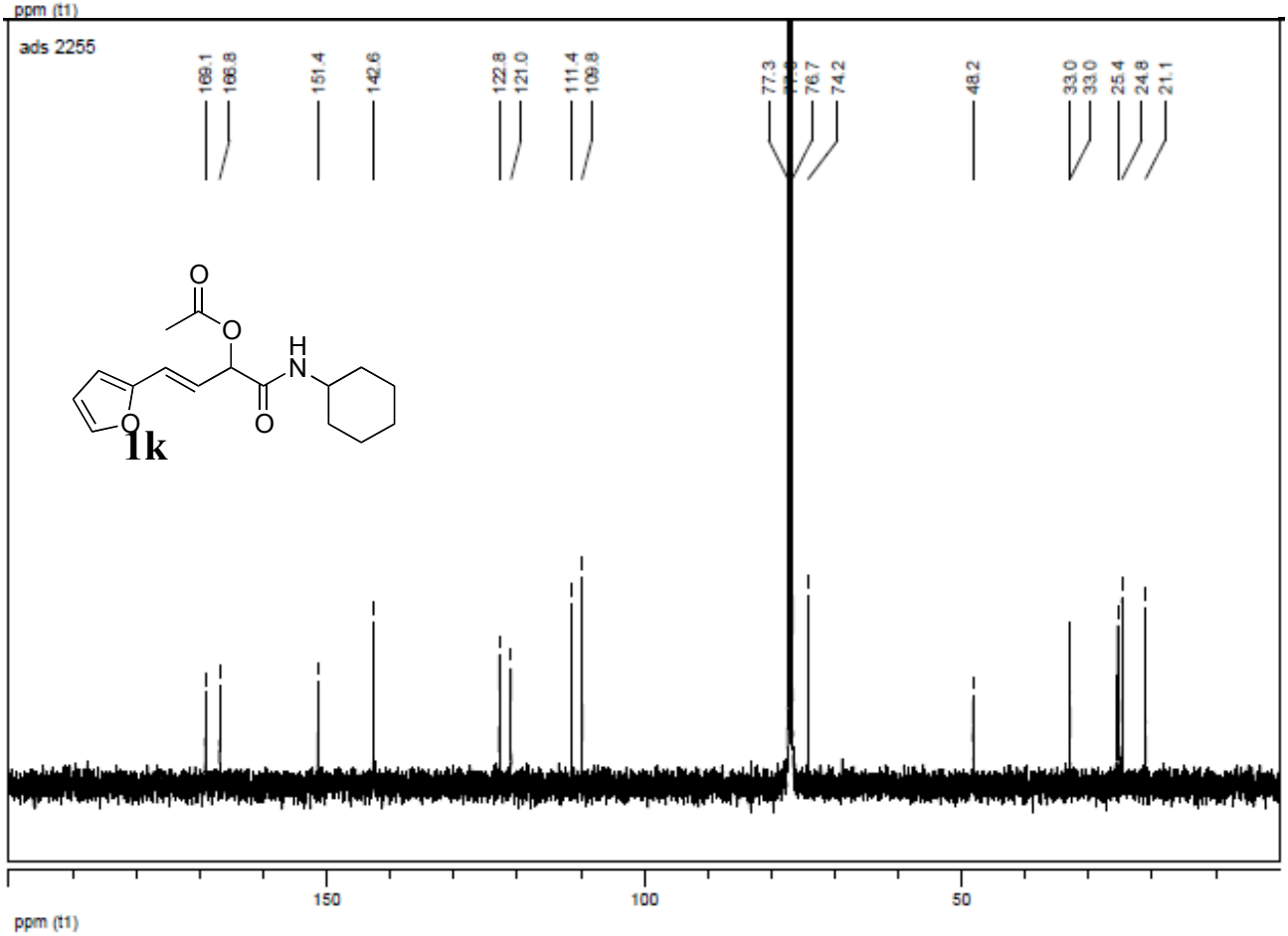
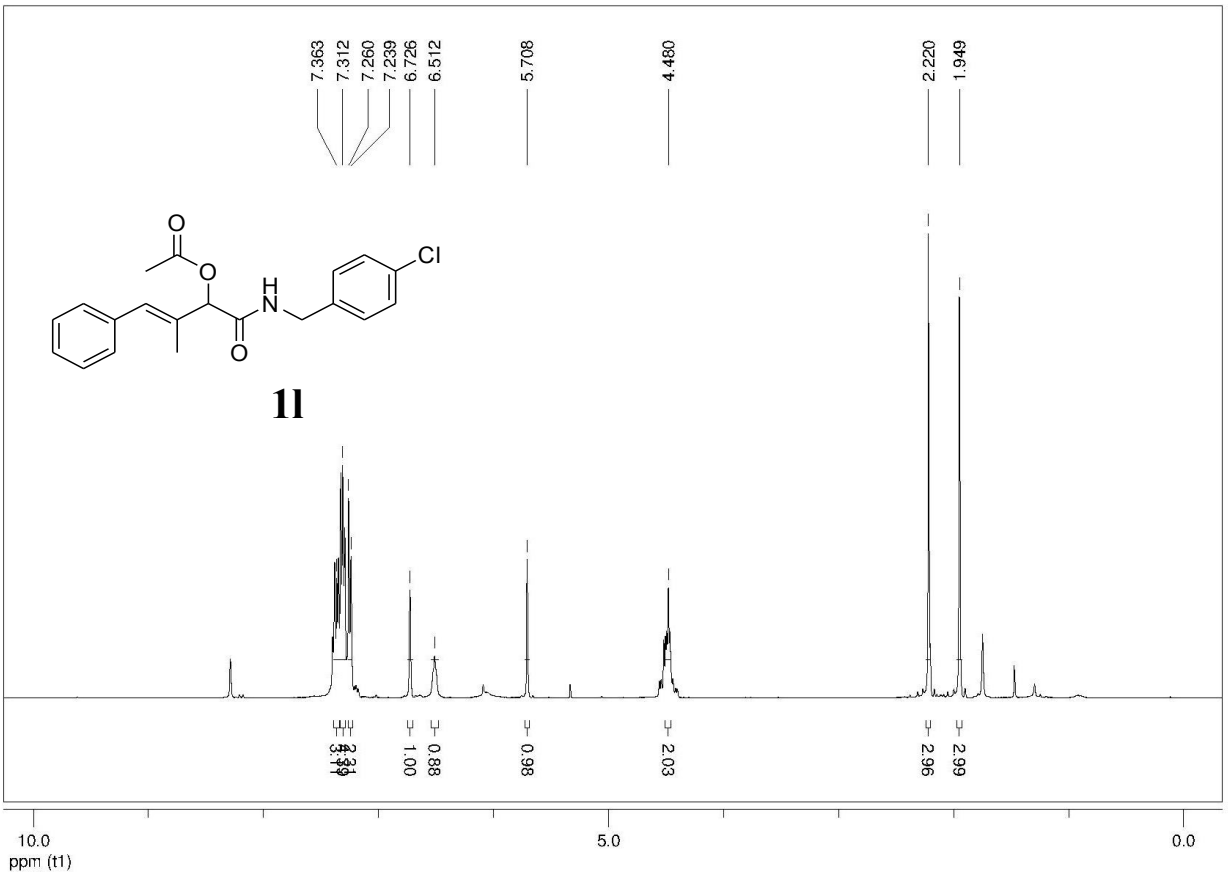
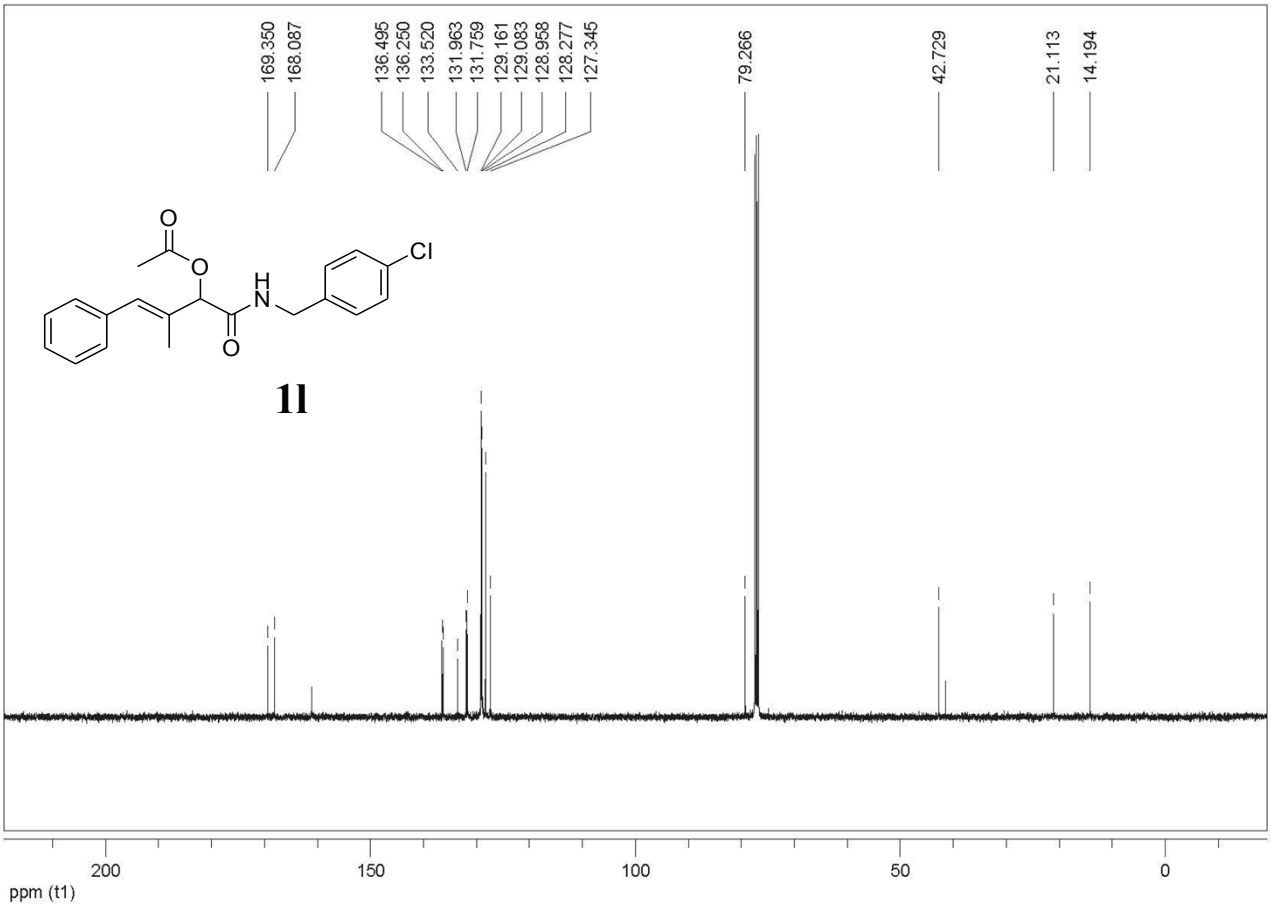
O
H
N
O
O
Cl
1j
200
150
100
50
0
ppm (t1)

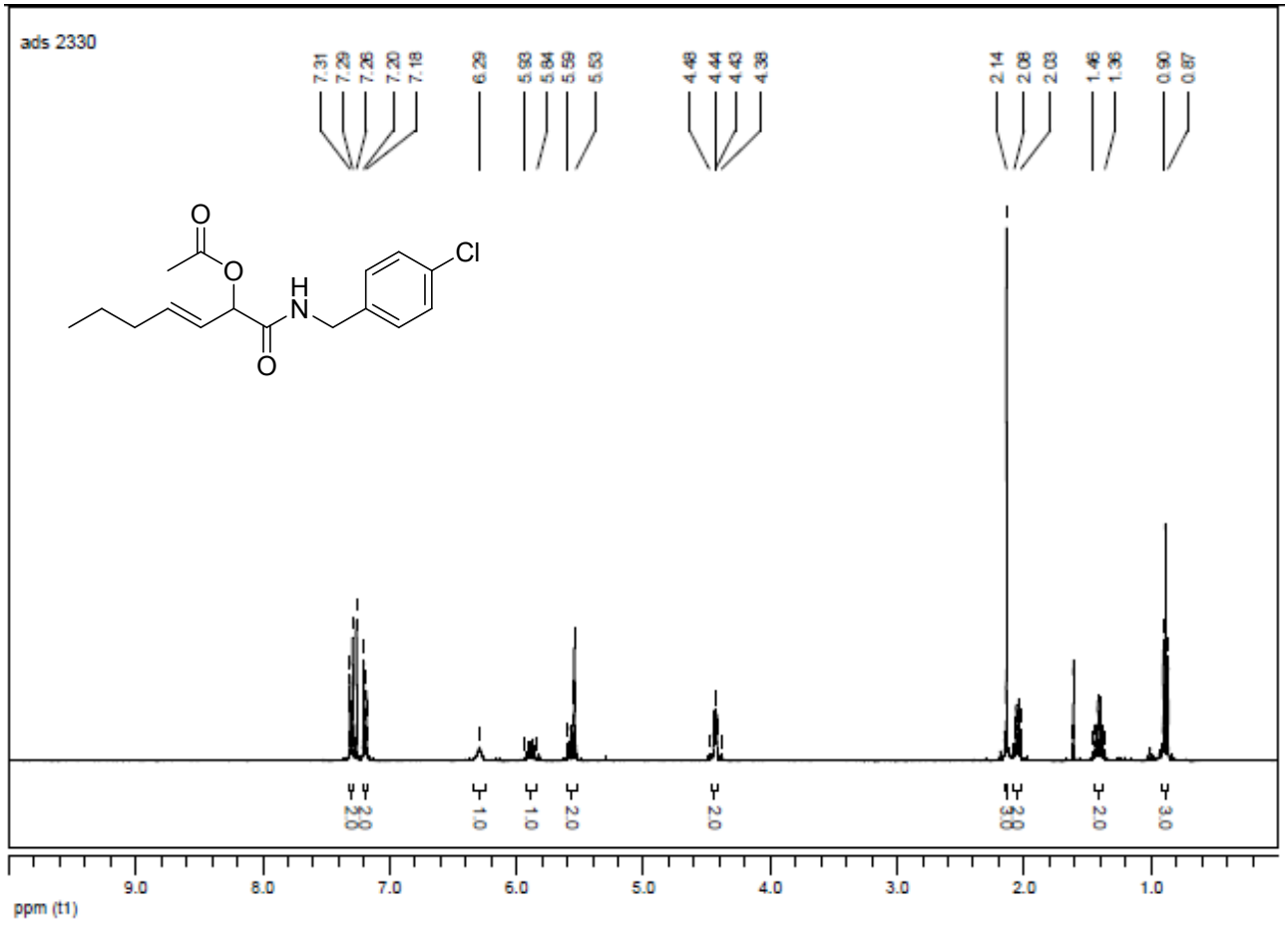

ads 2255
7.35
7.35
7.26
6.56
6.52
6.38
6.36
6.31
6.31
6.19
6.17
6.15
6.13
5.90
5.88
5.68
5.66
3.84
3.74
2.19
1.94
1.91
1.73
1.59
1.42
1.32
1.21
1.12
O
O
H
N
O
O
1.0
1.0
1.0
1.0
1.0
1.1
3.1
2.1
3.0
2.2
3.1
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

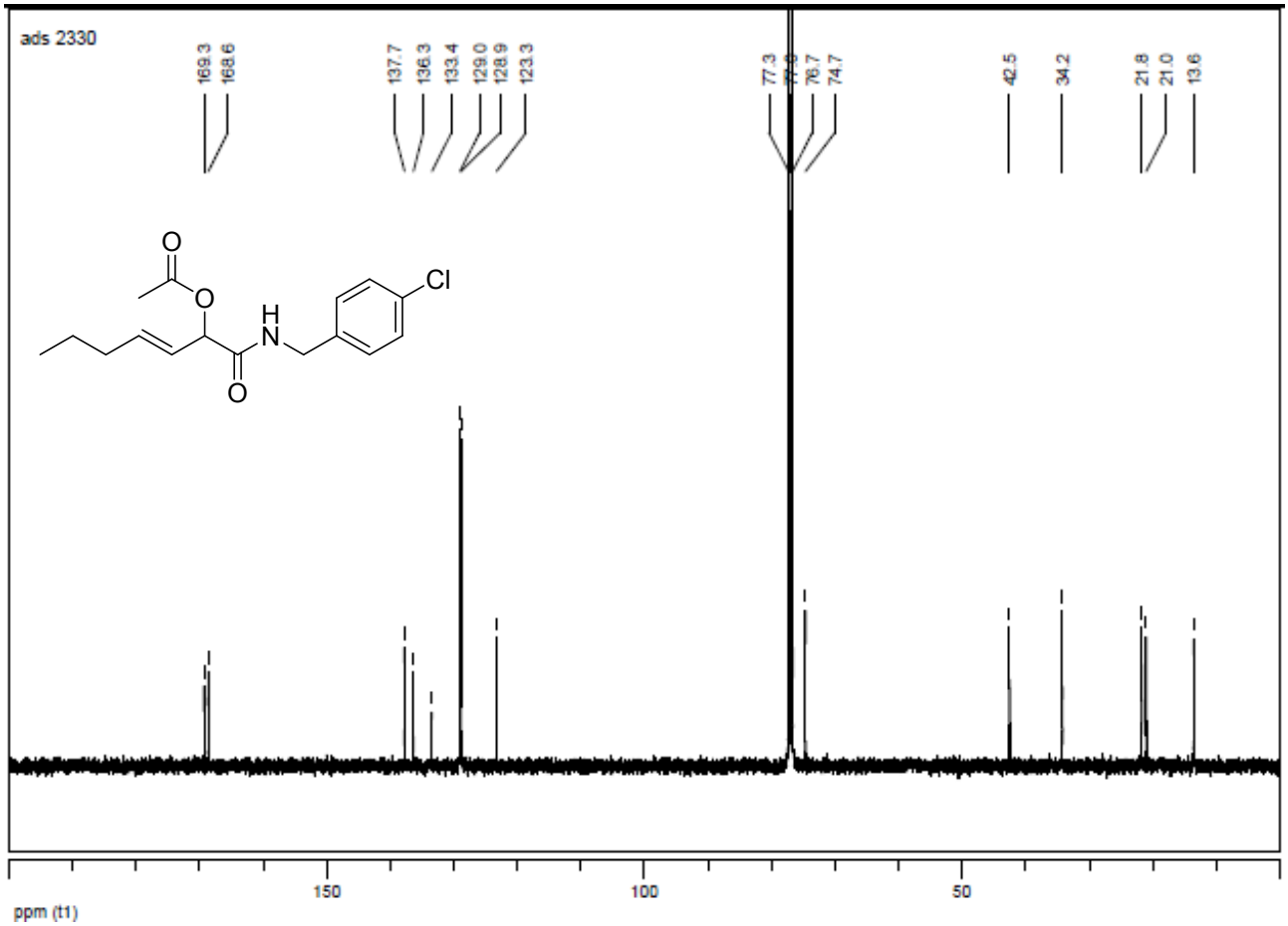

ads 2255
169.1
166.8
151.4
142.6
122.8
121.0
111.4
109.8
77.3
76.7
74.2
48.2
33.0
33.0
25.4
24.8
21.1
O
O
H
N
O
O
150
100
50
ppm (t1)

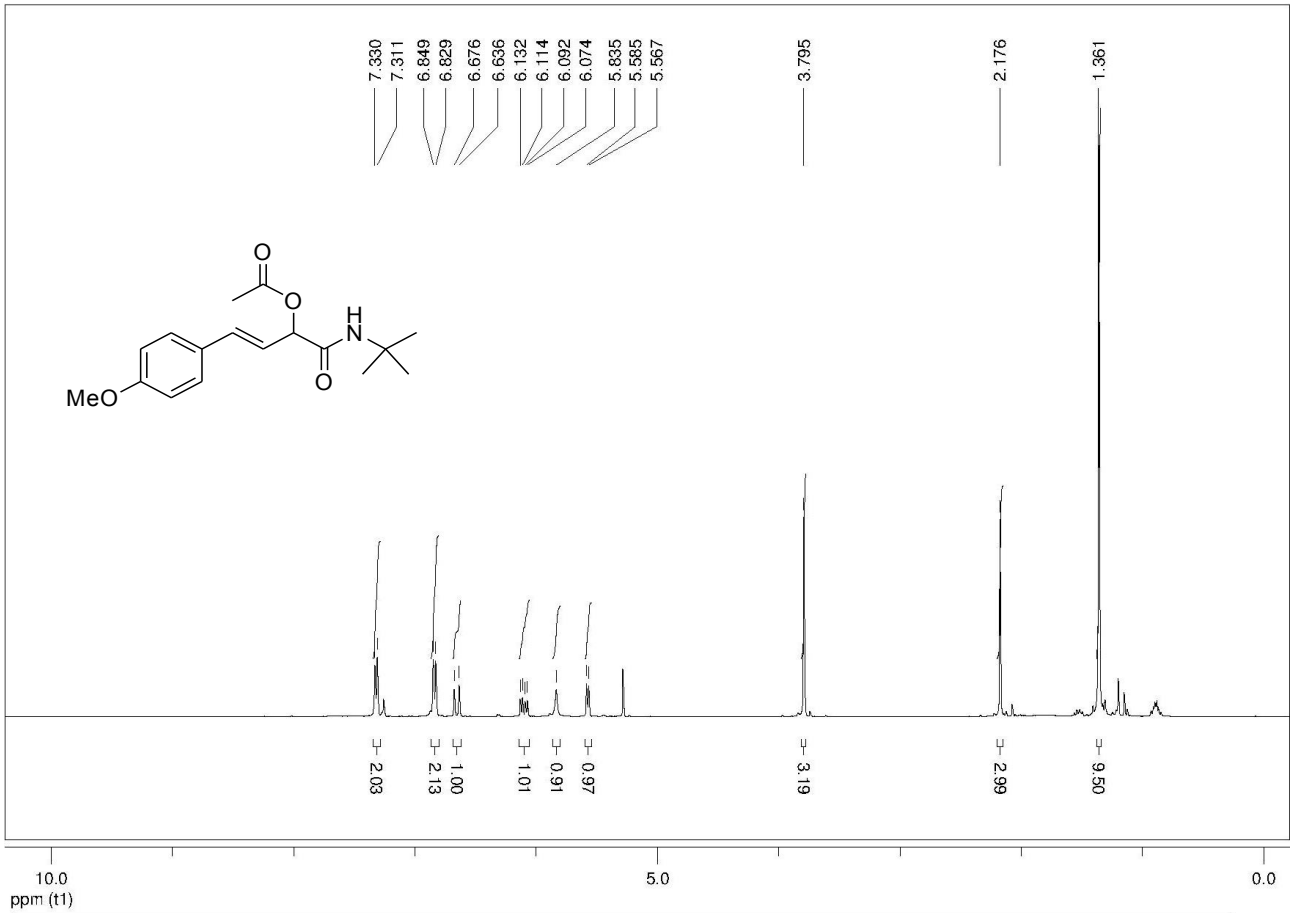
1k

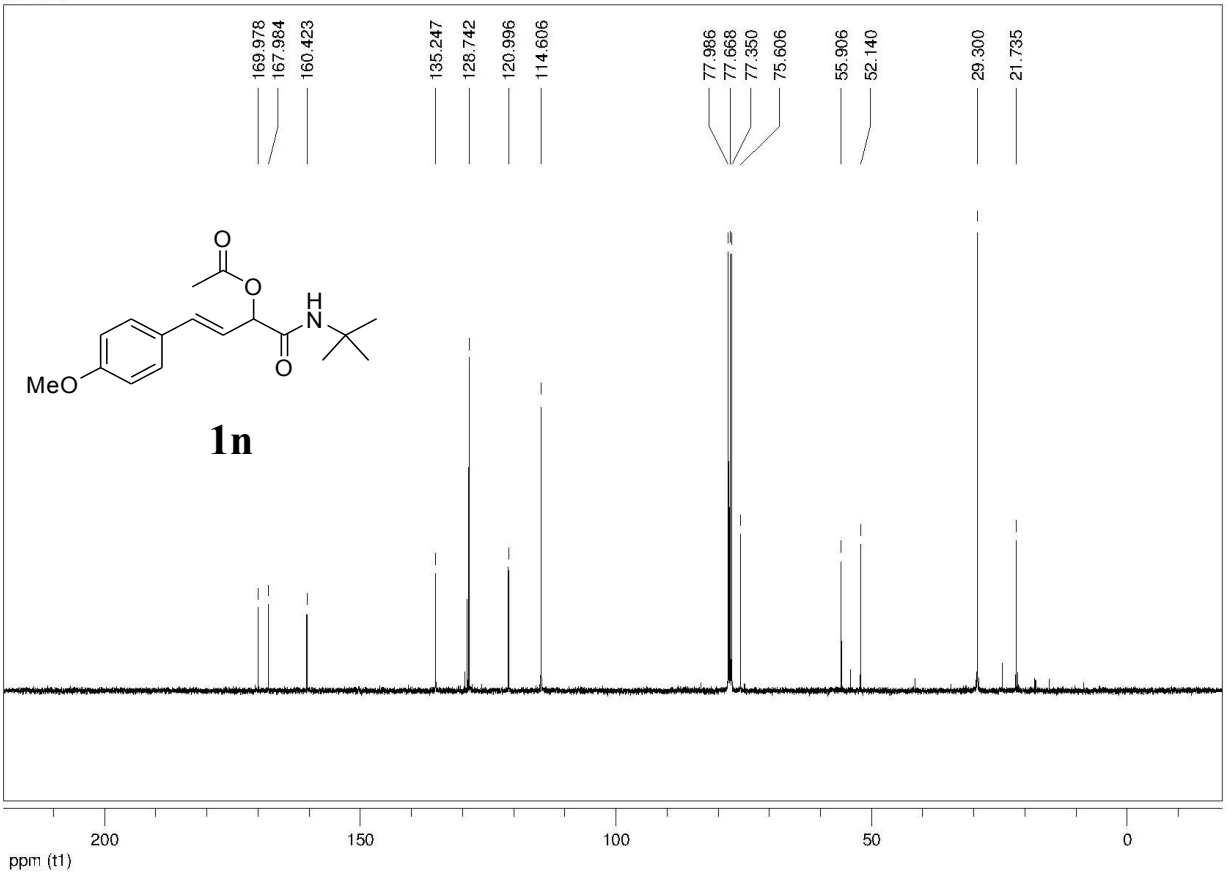

7.363
7.312
7.260
7.239
6.726
6.512
5.708
4.480
2.220
1.949
Cl
1l
1.00
0.88
0.98
2.03
2.96
2.99
10.0
5.0
0.0
ppm (t1)


169.350
168.087
136.495
136.250
133.520
131.963
131.759
129.161
129.083
128.958
128.277
127.345
79.266
42.729
21.113
14.194
Cl
1l
200
150
100
50
0
ppm (t1)

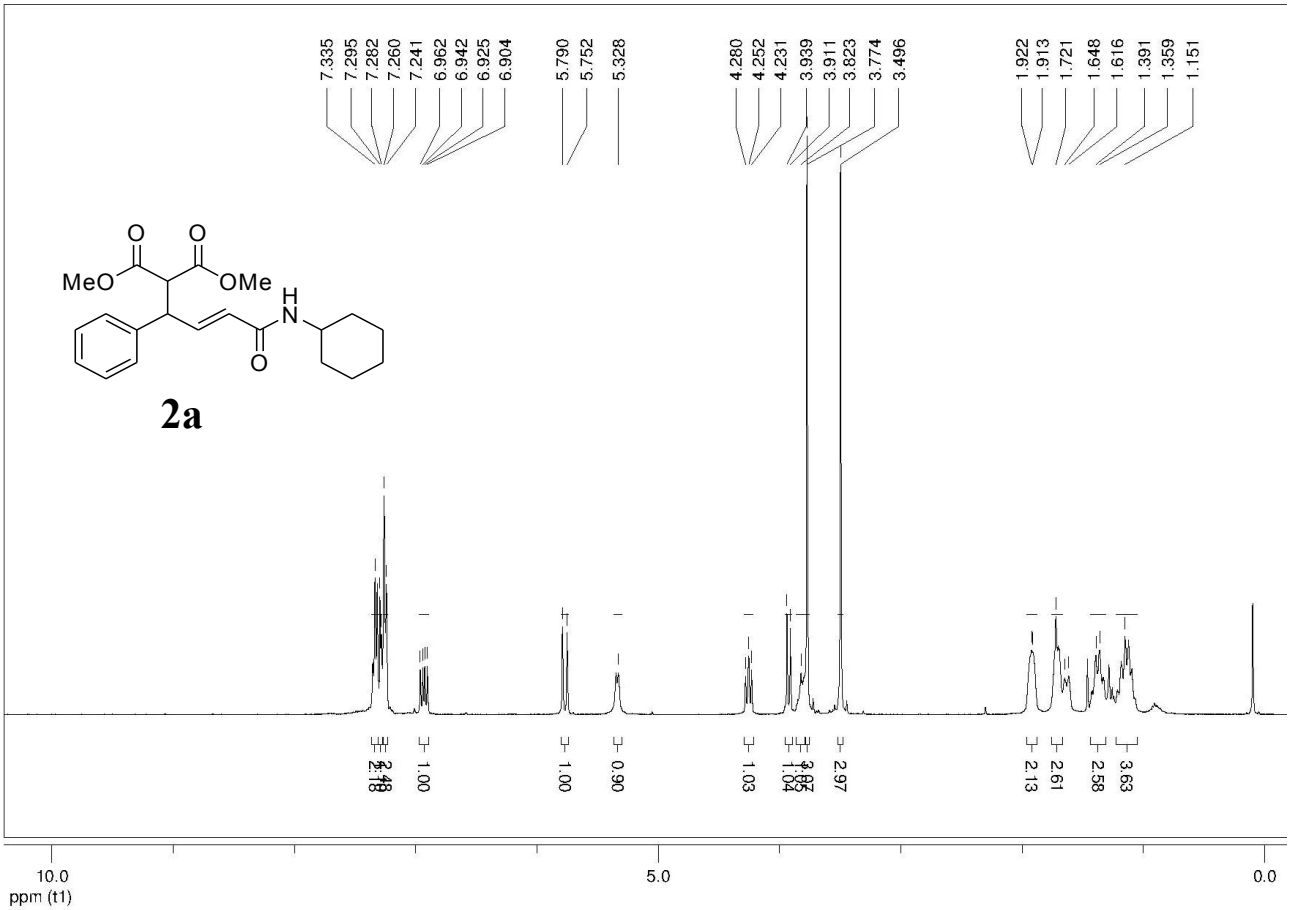

ads 2330
7.31
7.29
7.26
7.20
7.18
6.29
5.93
5.84
5.59
5.53
4.48
4.44
4.43
4.38
2.14
2.08
2.03
1.46
1.36
0.90
0.87
O
O
H
N
O
Cl
2.28
1.10
1.10
2.00
2.00
3.18
2.00
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)


ads 2330
169.3
168.6
137.7
136.3
133.4
129.0
128.9
123.3
77.3
77.0
76.7
74.7
42.5
34.2
21.8
21.0
13.6
O
O
H
N
O
Cl
150
100
50
ppm (t1)

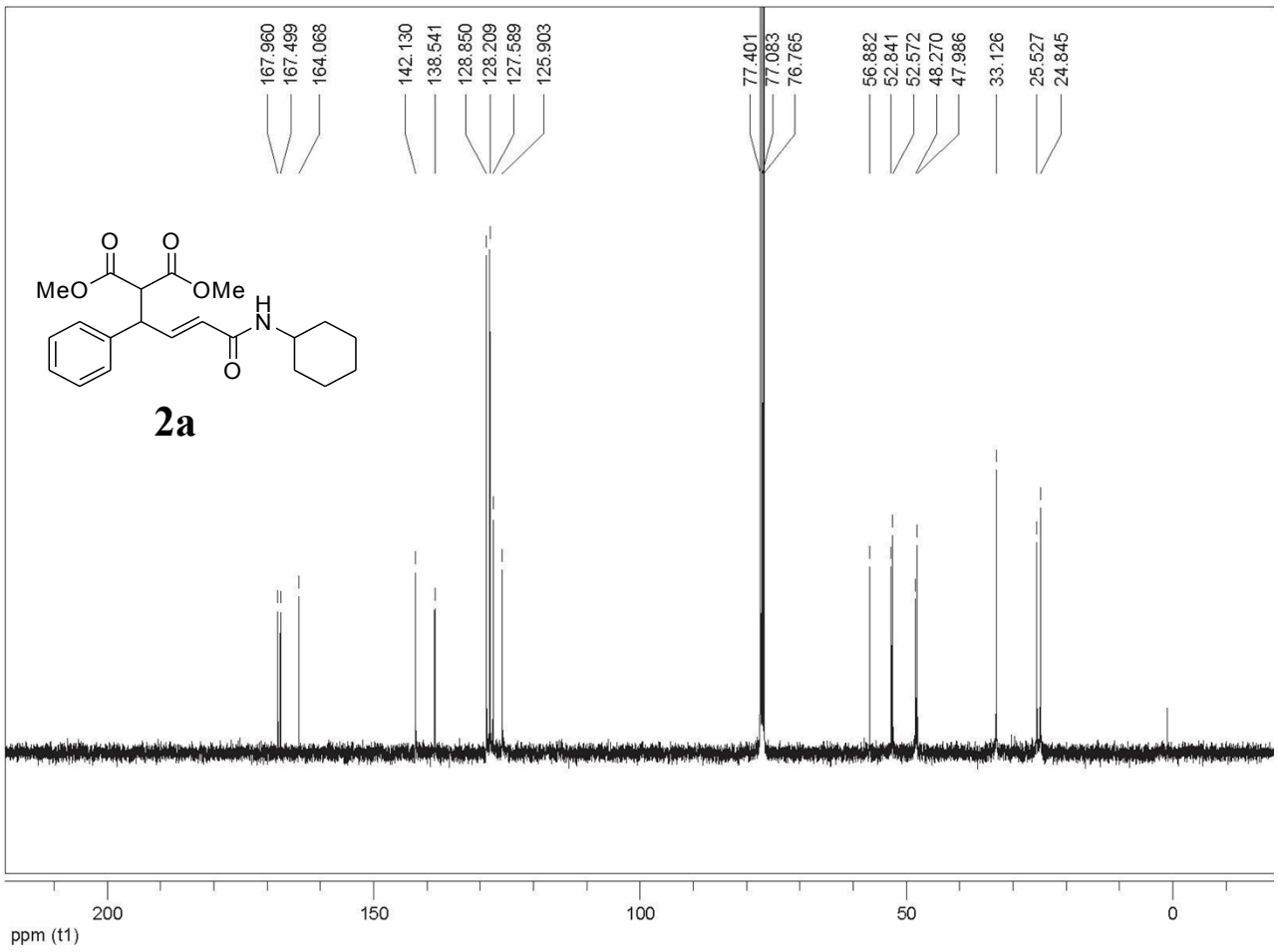

7.330
7.311
6.849
6.829
6.676
6.636
6.132
6.114
6.092
6.074
5.835
5.585
5.567
3.795
2.176
1.361
MeO
2.03
2.13
1.00
1.01
0.91
0.97
3.19
2.99
9.50
10.0
5.0
0.0
ppm (t1)


169.978
167.984
160.423
135.247
128.742
120.996
114.606
77.986
77.668
77.350
75.606
55.906
52.140
29.300
21.735
MeO
200
150
100
50
0
ppm (t1)

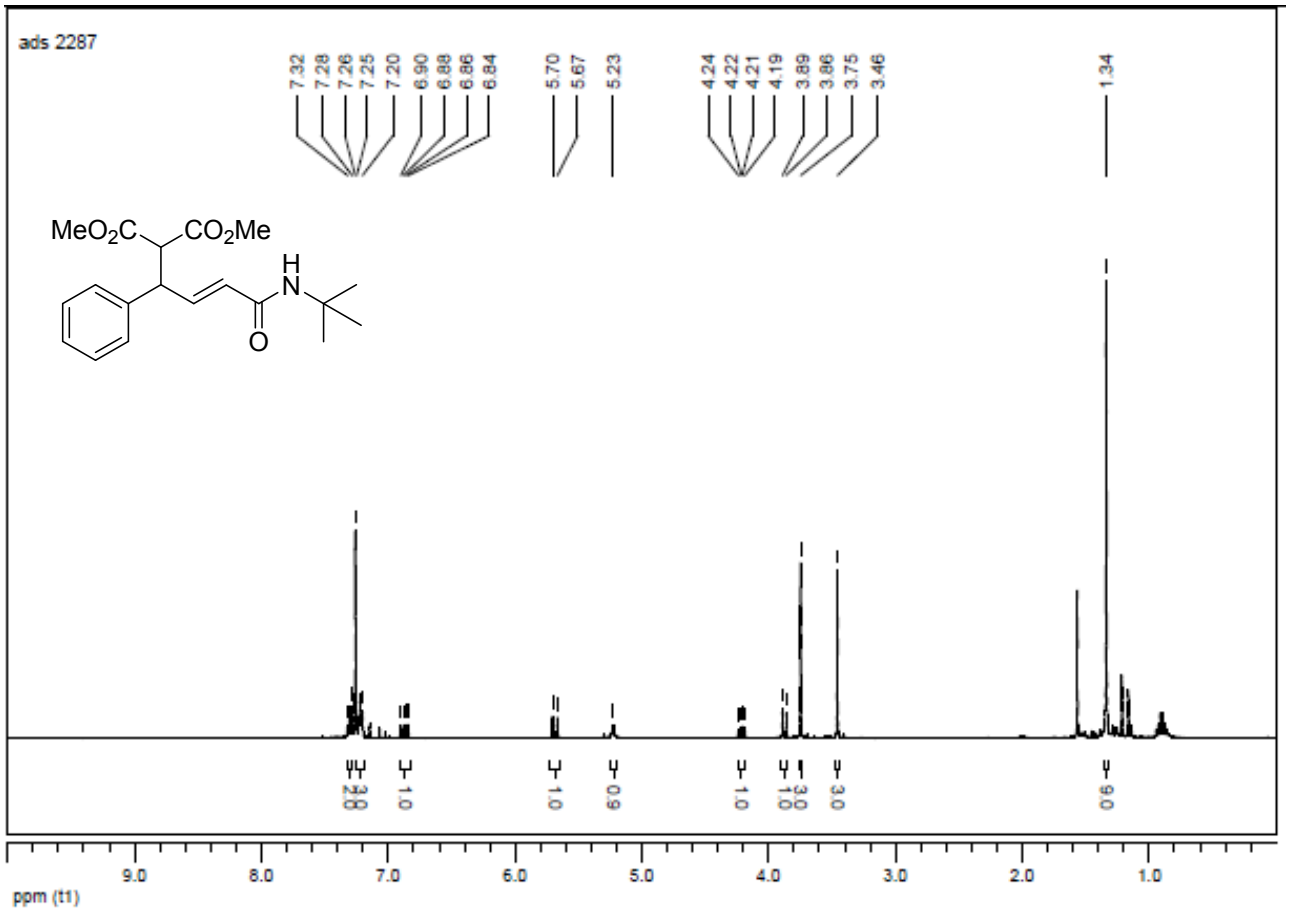
1n

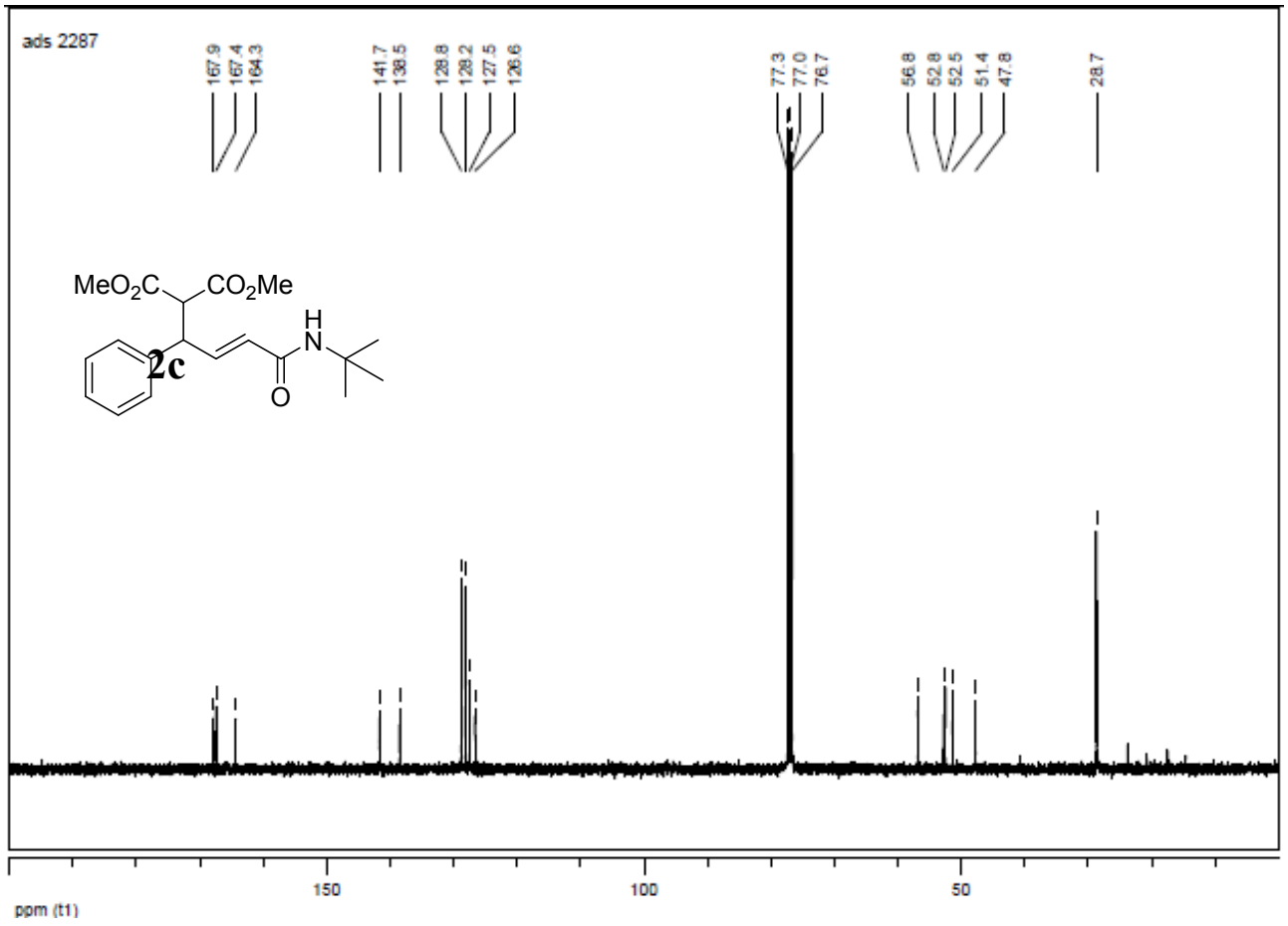

7.335
7.295
7.282
7.260
7.241
6.962
6.942
6.925
6.904
5.790
5.752
5.328
4.280
4.252
4.231
3.939
3.911
3.823
3.774
3.496
1.922
1.913
1.721
1.648
1.616
1.391
1.359
1.151
MeO
OMe
H
N
O
O
O
O
2.48
2.48
1.00
1.00
0.90
1.03
1.07
1.14
2.97
2.13
2.01
2.58
3.63
10.0
5.0
0.0
ppm (t1)

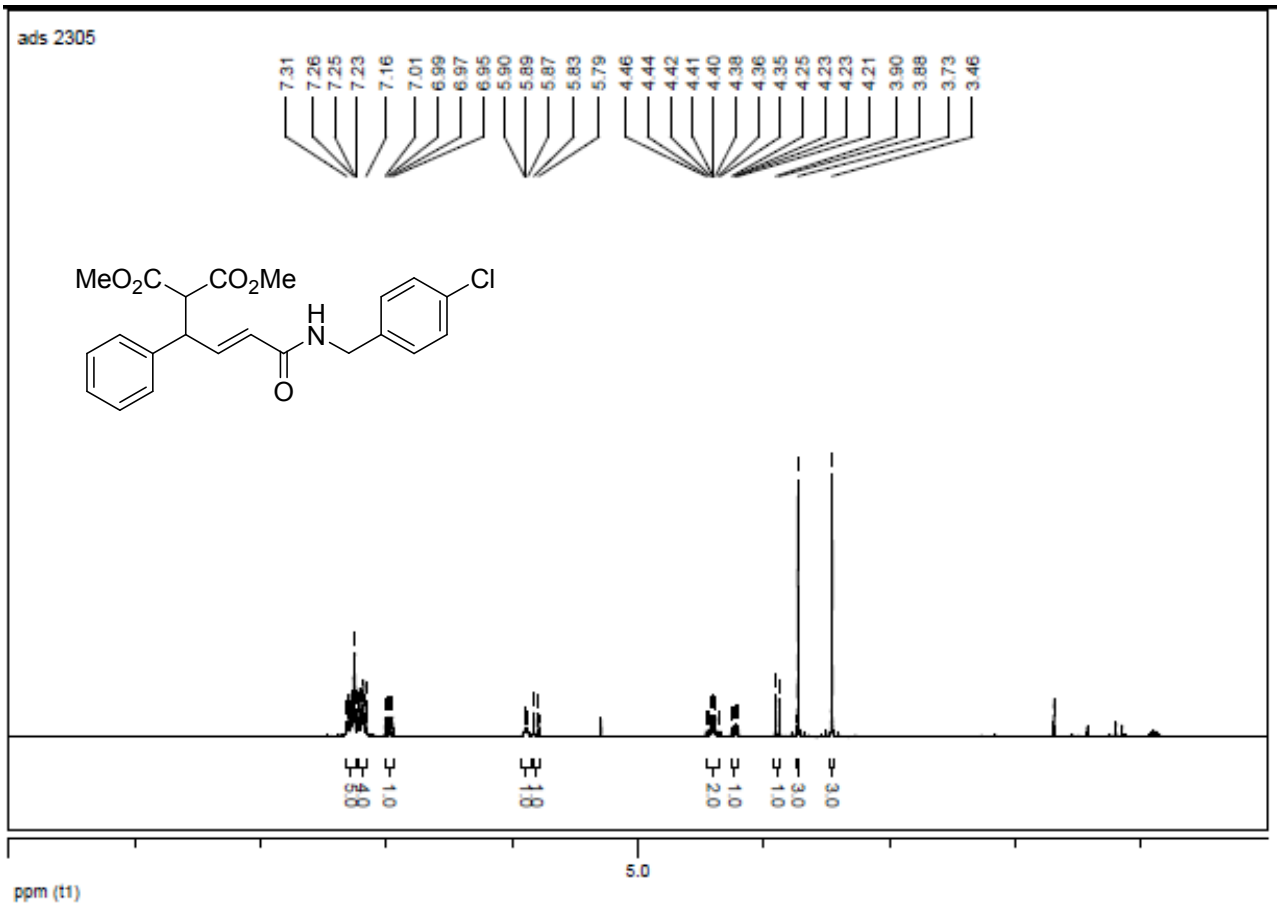
2a

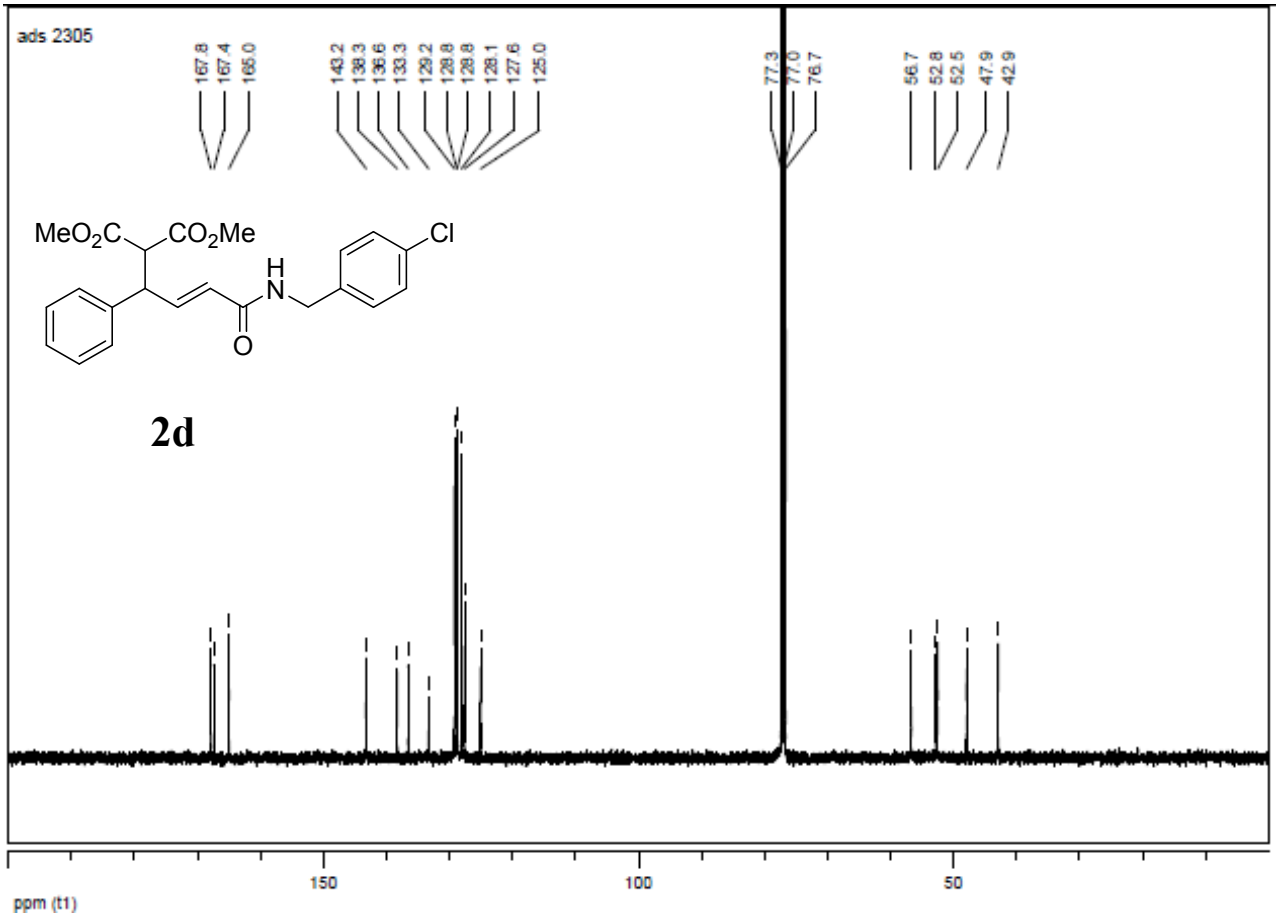

167.960
167.499
164.068
142.130
138.541
128.850
128.209
127.589
125.903
77.401
77.083
76.765
56.882
52.841
52.572
48.270
47.986
33.126
25.527
24.845
MeO
OMe
H
N
O
O
O
O
200
150
100
50
0
ppm (t1)

2a

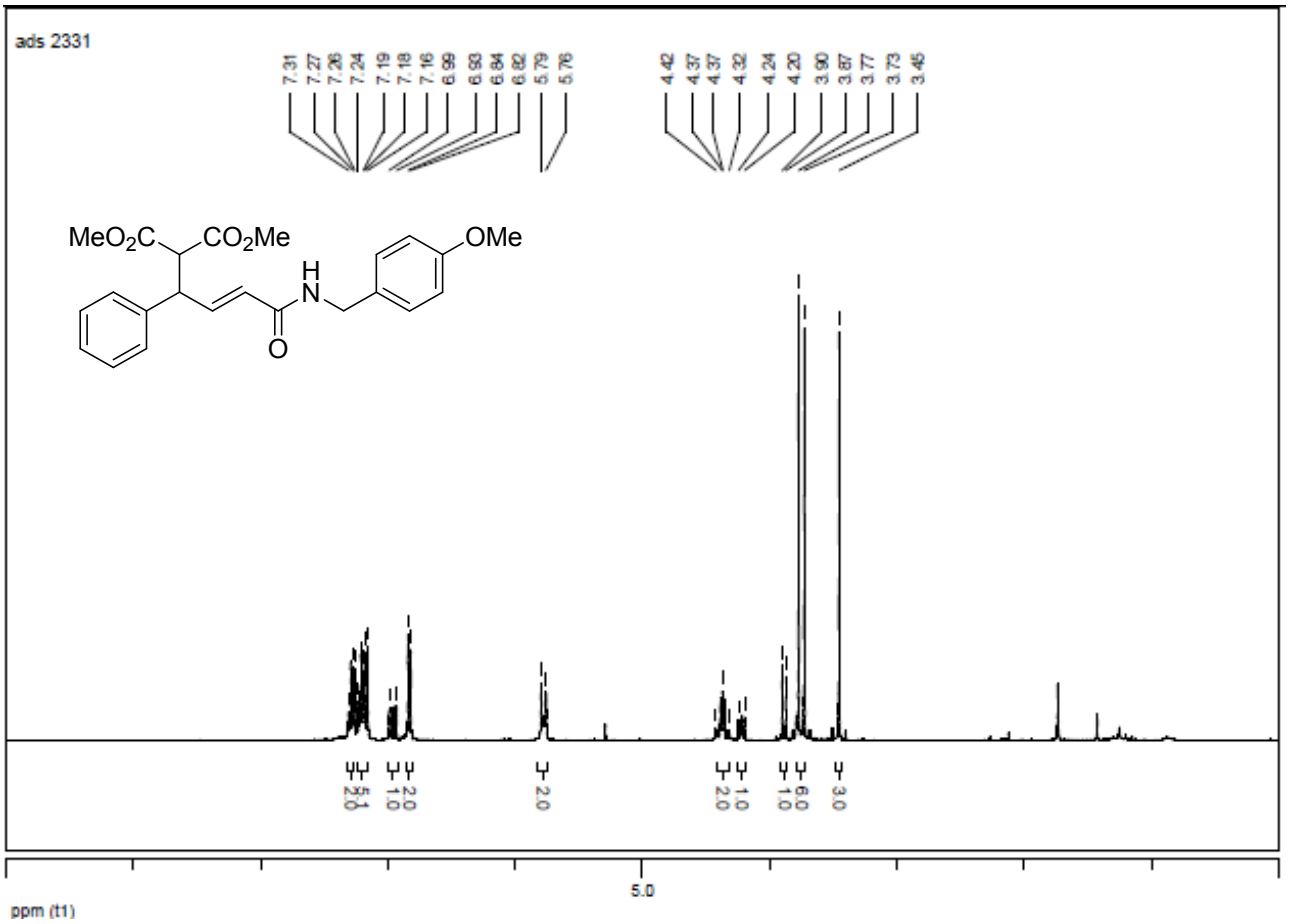

ads 2287
7.32
7.28
7.26
7.25
7.20
6.90
6.88
6.86
6.84
5.70
5.67
5.23
4.24
4.22
4.21
4.19
3.89
3.86
3.75
3.46
1.34
MeO2C
CO2Me
H
N
O
2.0
1.0
1.0
0.9
1.0
1.0
3.0
9.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

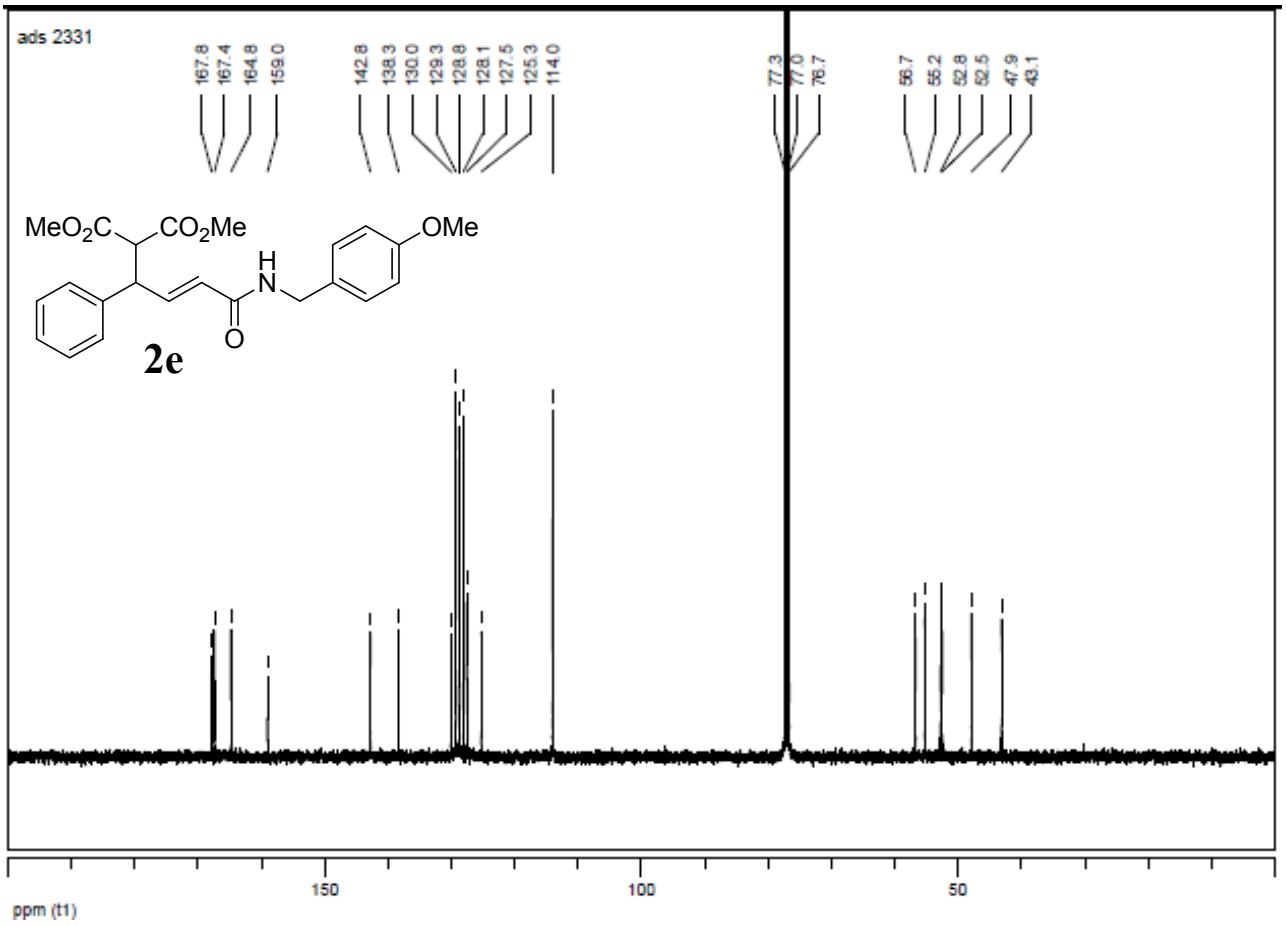

ads 2287
167.9
167.4
164.3
141.7
138.5
128.8
128.2
127.5
126.6
77.3
77.0
76.7
56.8
52.8
52.5
51.4
47.8
28.7
MeO2C
CO2Me
H
N
O
2c
150
100
50
ppm (t1)

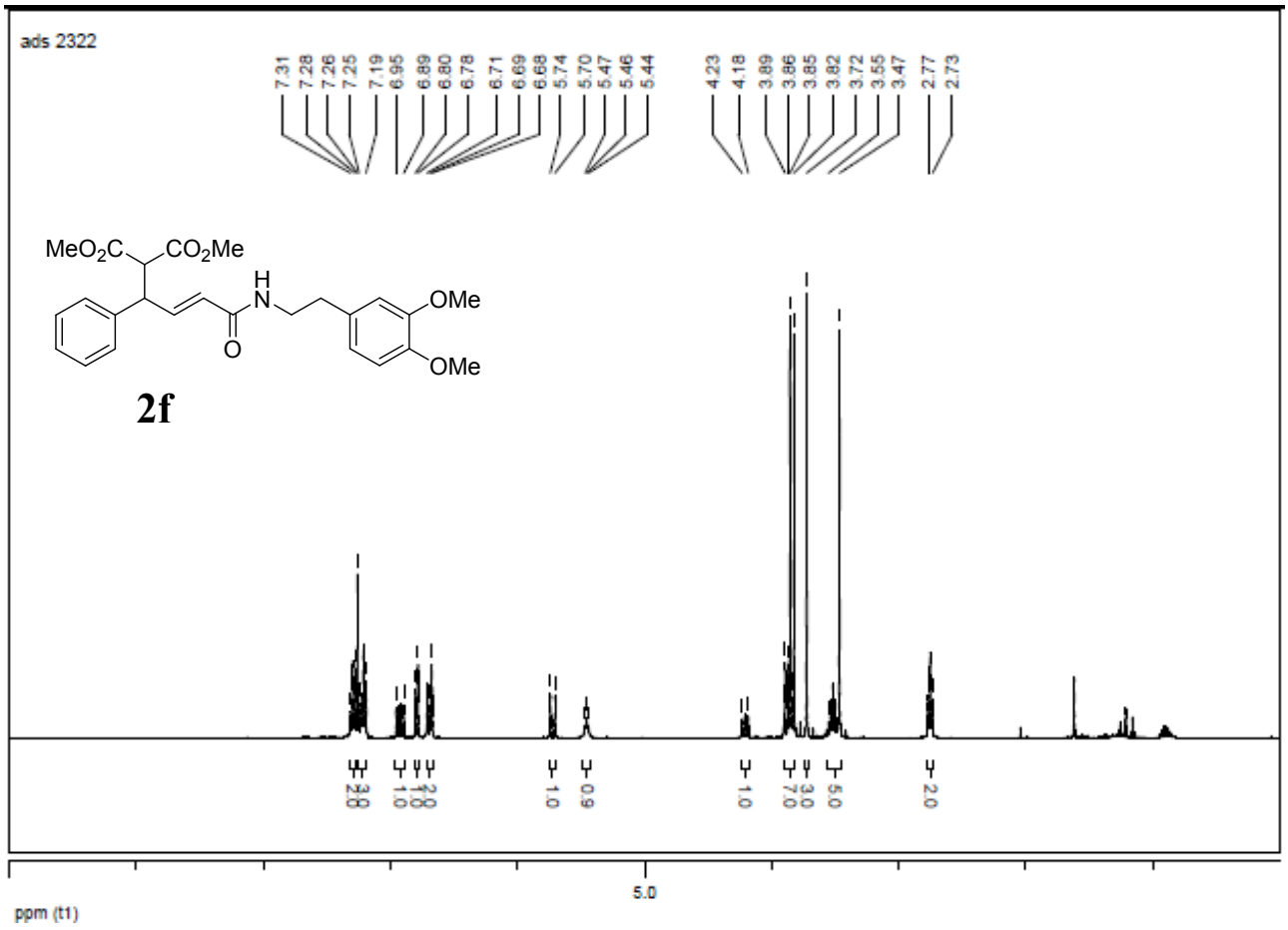

ads 2305
7.31
7.26
7.25
7.23
7.16
7.01
6.99
6.97
6.95
5.90
5.89
5.87
5.83
5.79
4.46
4.44
4.42
4.41
4.40
4.38
4.36
4.35
4.25
4.23
4.23
4.21
3.90
3.88
3.73
3.46
MeO2C
CO2Me
Cl
H
N
O
5.0
ppm (t1)

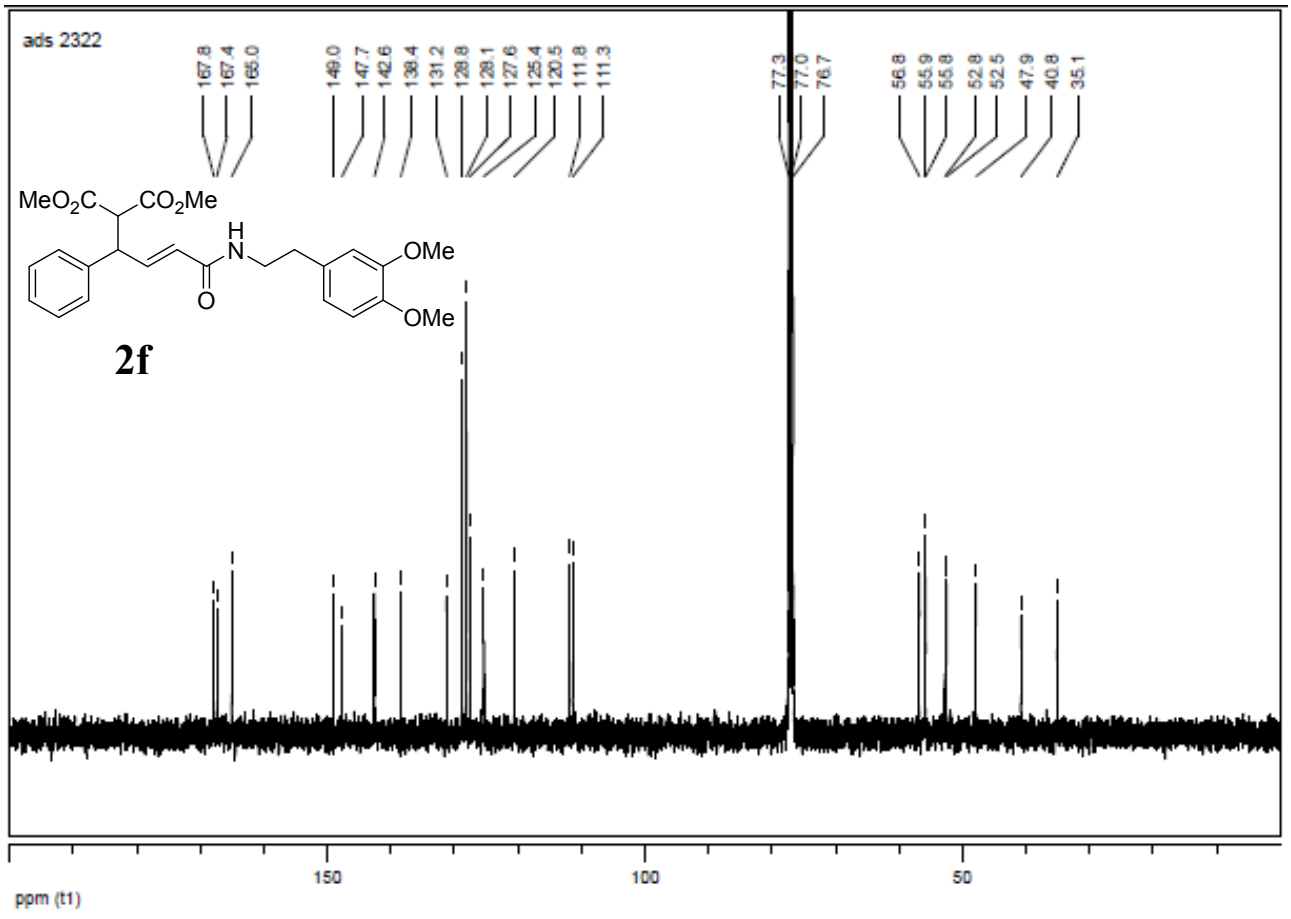

ads 2305
167.8
167.4
165.0
143.2
138.3
136.6
133.3
129.2
128.8
128.8
128.1
127.6
125.0
77.3
77.0
76.7
56.7
52.8
52.5
47.9
42.9
MeO2C
CO2Me
Cl
H
N
O
150
100
50
ppm (t1)

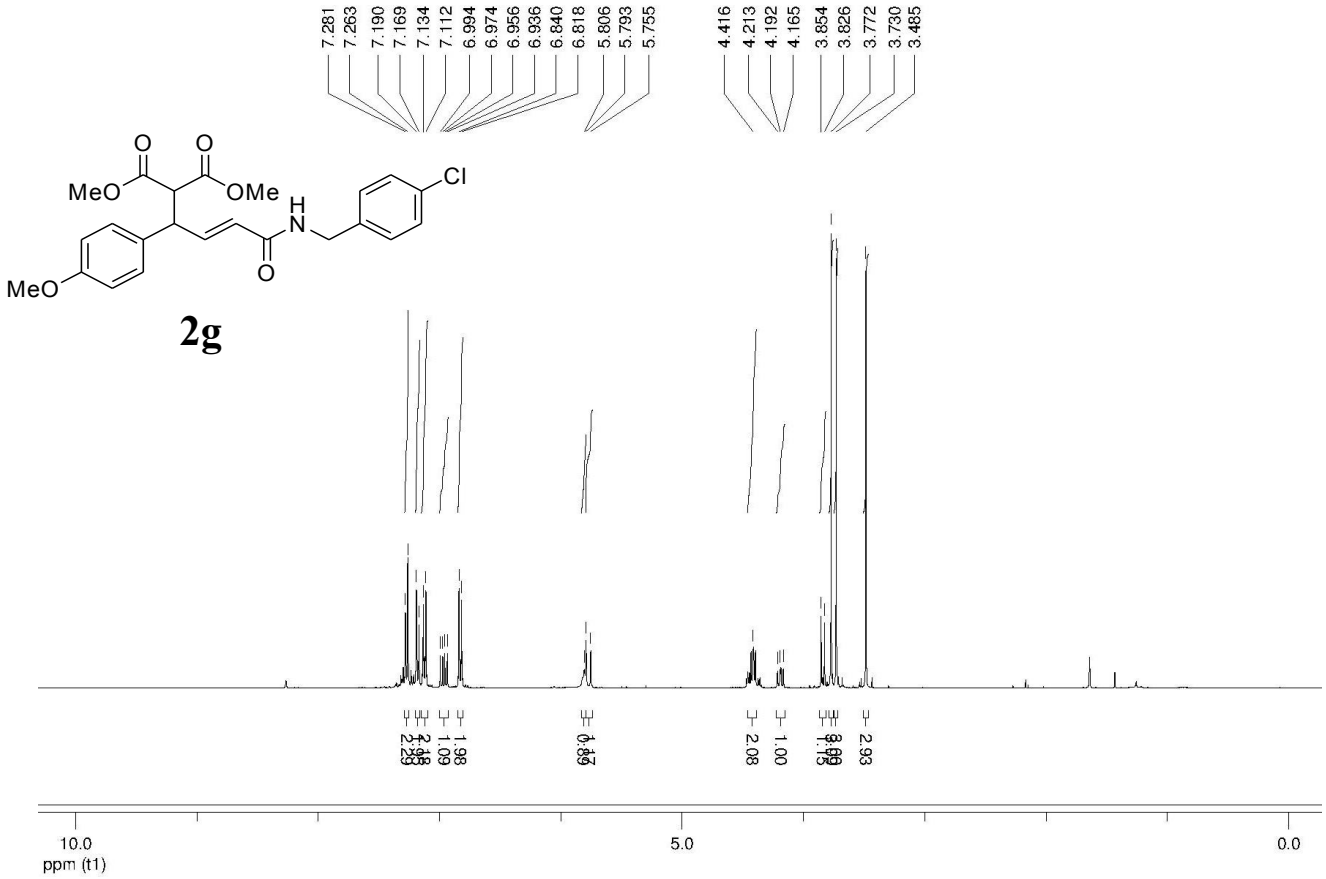
2d

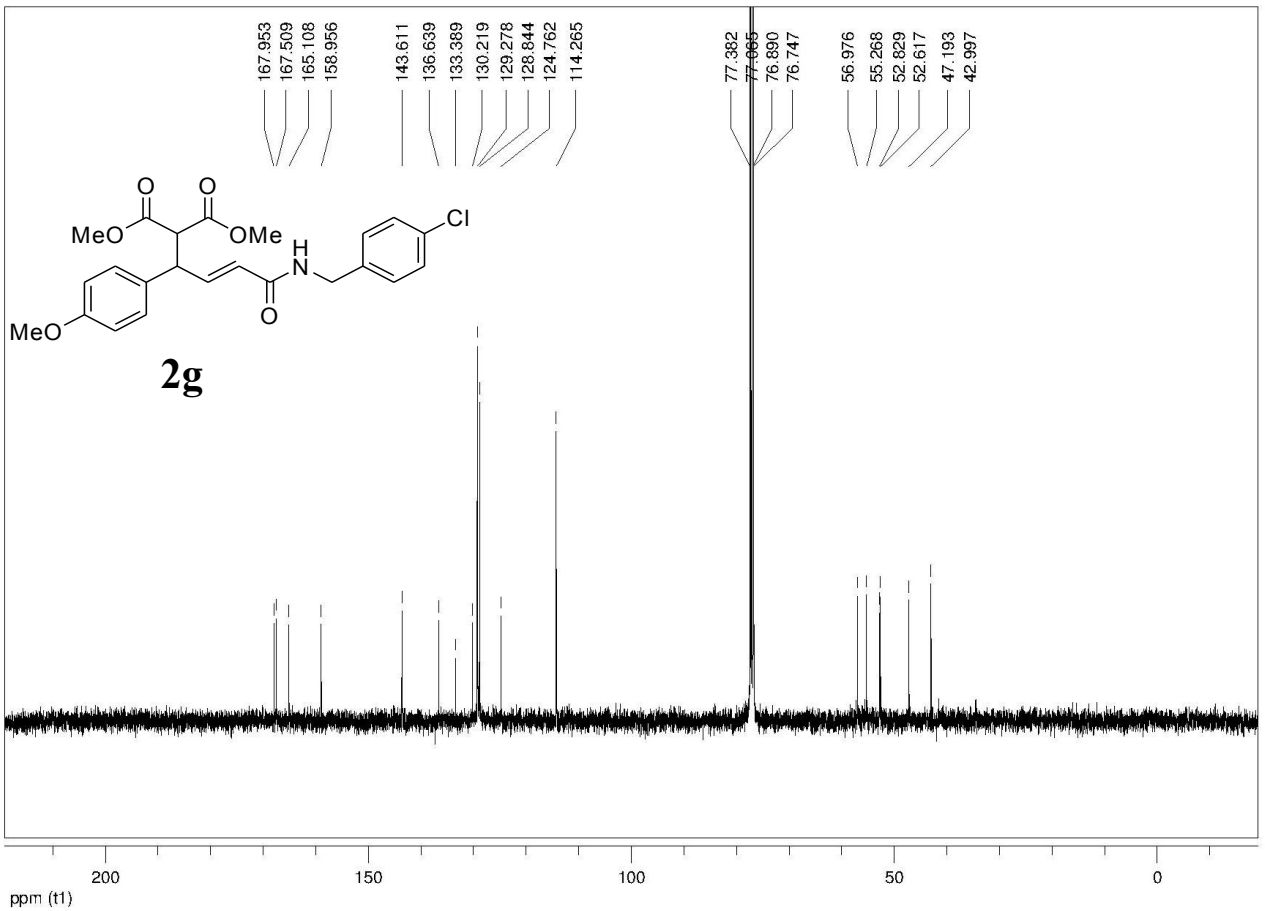

ads 2331
7.31
7.27
7.26
7.24
7.19
7.18
7.16
6.99
6.93
6.84
6.82
5.79
5.76
4.42
4.37
4.37
4.32
4.24
4.20
3.90
3.87
3.77
3.73
3.45
MeO2C
CO2Me
OMe
H
N
O
2.10
5.14
2.10
2.10
2.10
2.10
1.10
6.10
3.10
5.0
ppm (t1)

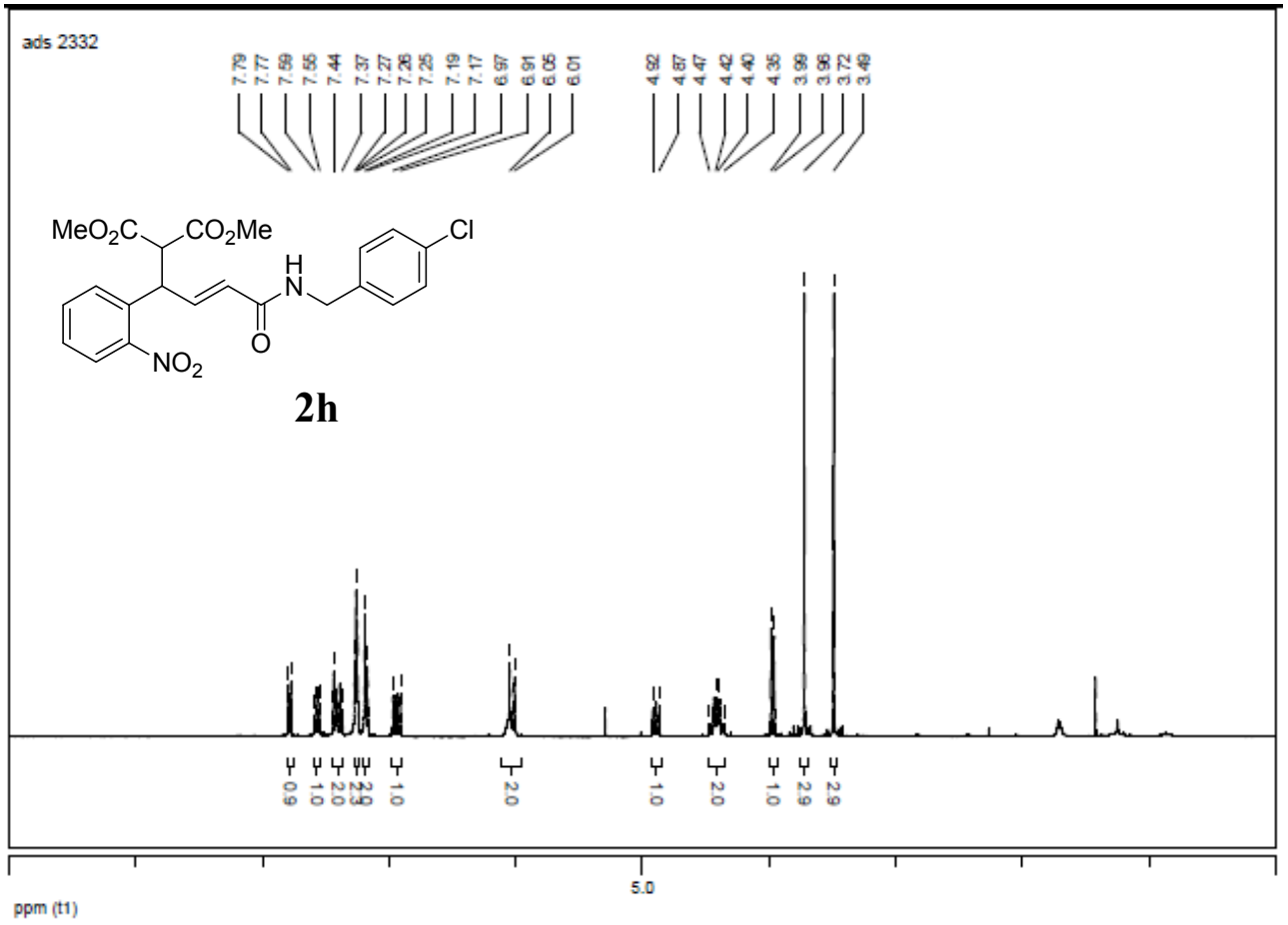

ads 2331
167.8
167.4
164.8
159.0
142.8
138.3
130.0
129.3
128.8
128.1
127.5
125.3
114.0
77.3
77.0
76.7
56.7
55.2
52.8
52.5
47.9
43.1
MeO2C
CO2Me
OMe
H
N
O
2e
150
100
50
ppm (t1)

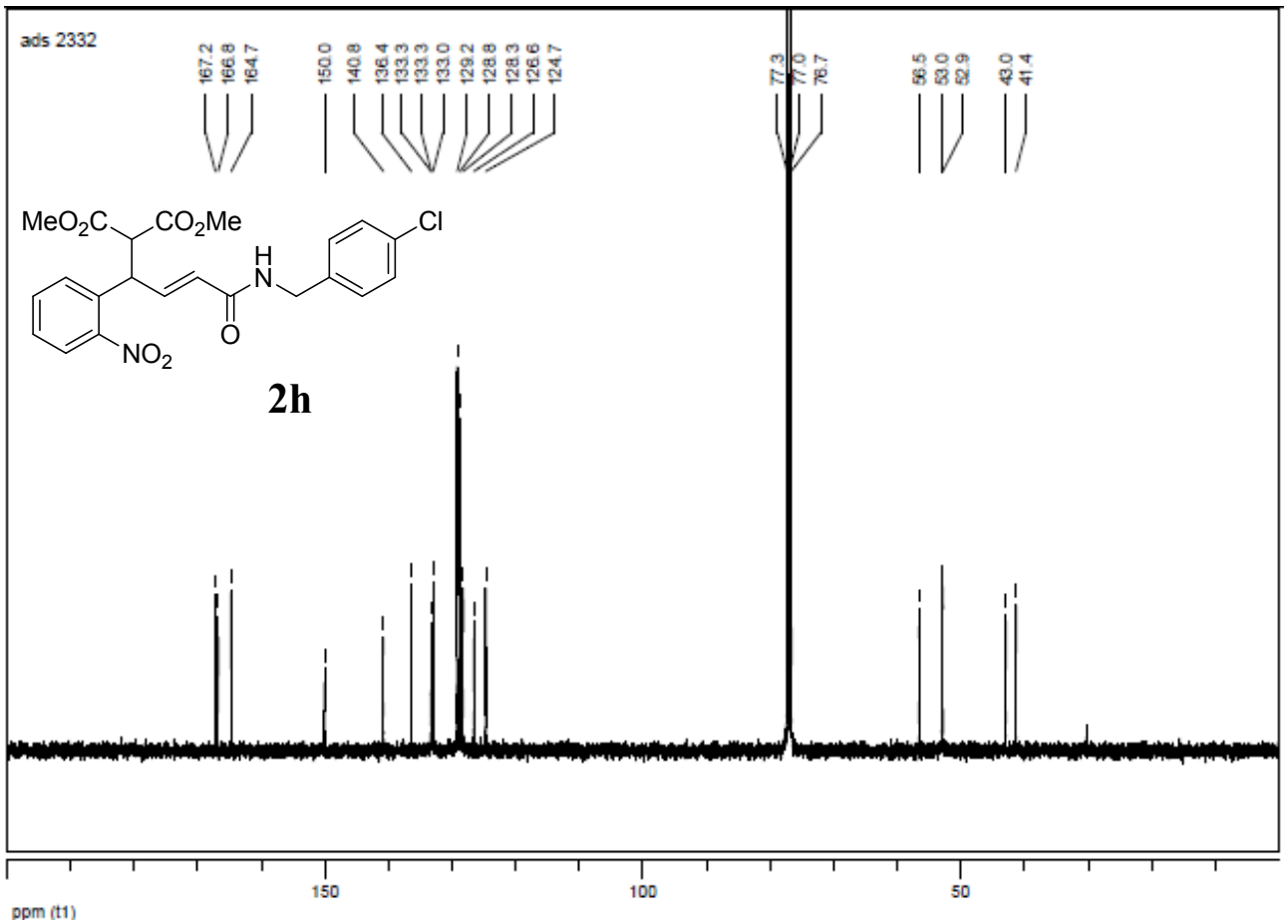

ads 2322
7.31
7.28
7.26
7.25
7.19
6.95
6.89
6.80
6.78
6.71
6.69
6.68
5.74
5.70
5.47
5.46
5.44
4.23
4.18
3.89
3.86
3.85
3.82
3.72
3.55
3.47
2.77
2.73
MeO2C
CO2Me
H
N
O
OMe
OMe
2f
3.0
1.0
2.0
1.0
1.0
0.9
1.0
7.0
3.0
5.0
2.0
5.0
ppm (t1)

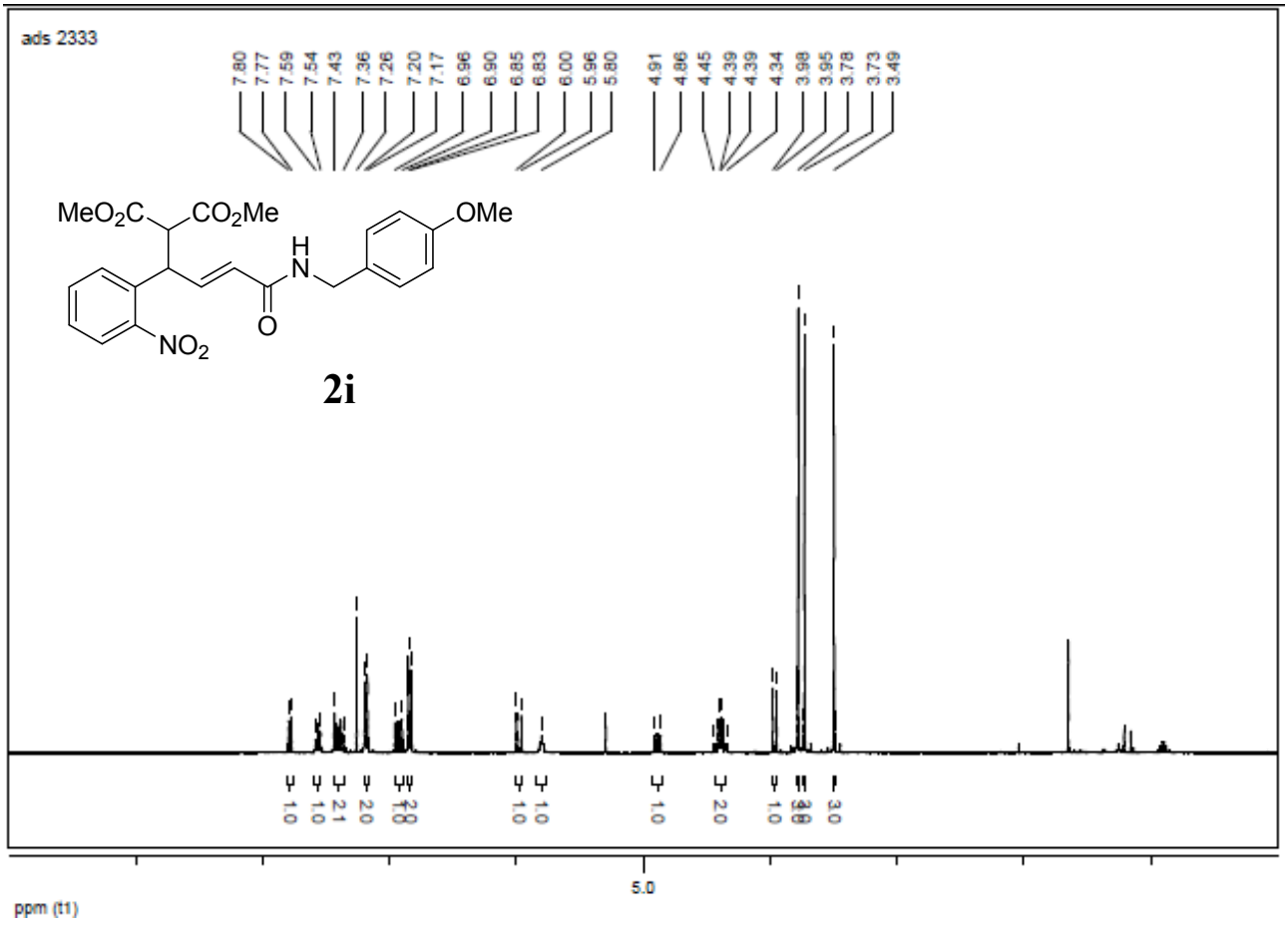

ads 2322
167.8
167.4
166.0
149.0
147.7
142.6
138.4
131.2
128.8
128.1
127.6
125.4
120.5
111.8
111.3
77.3
77.0
76.7
56.8
55.9
55.8
52.8
52.5
47.9
40.8
35.1
MeO2C
CO2Me
H
N
O
OMe
OMe
2f
150
100
50
ppm (t1)

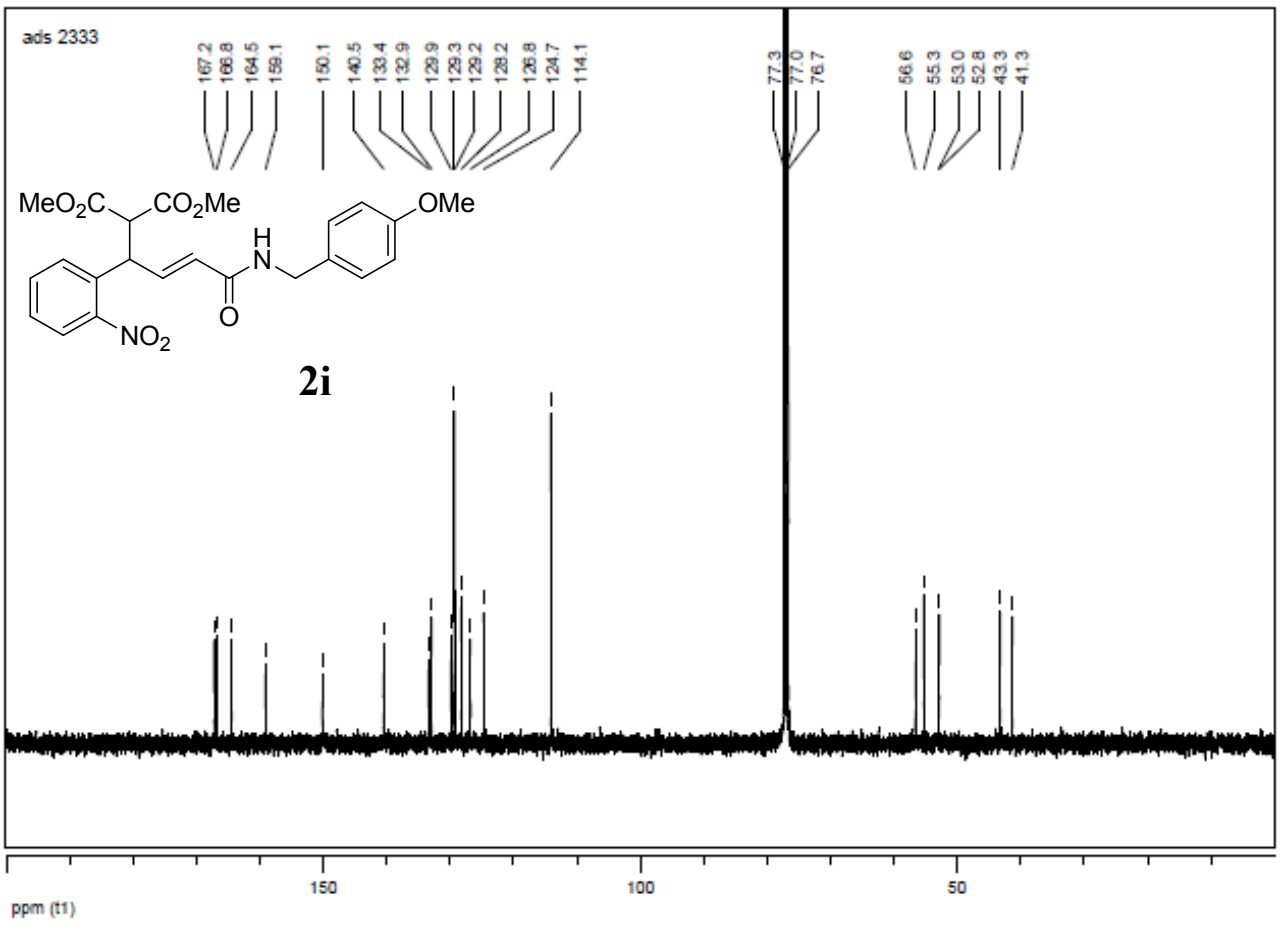

7.281
7.263
7.190
7.169
7.134
7.112
6.994
6.974
6.956
6.936
6.840
6.818
5.806
5.793
5.755
4.416
4.213
4.192
4.165
3.854
3.826
3.772
3.730
3.485
MeO
OMe
Cl
H
N
O
MeO
2g
2.28
2.28
2.28
1.09
1.96
0.80
2.08
1.00
8.00
2.93
10.0
ppm (t1)
5.0
0.0

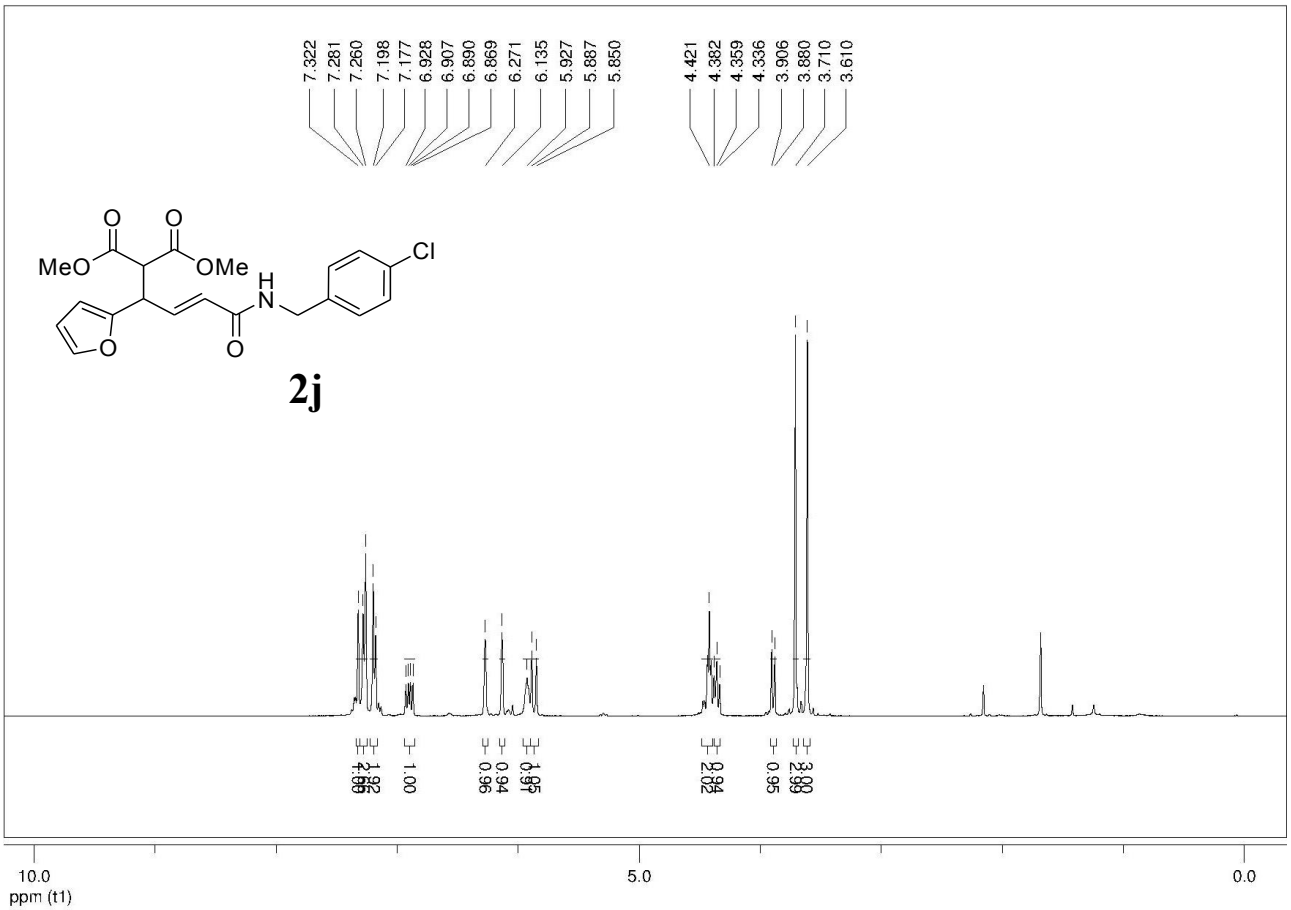

167.953
167.509
165.108
158.956
143.611
136.639
133.389
130.219
129.278
128.844
124.762
114.265
77.382
77.065
76.890
76.747
56.976
55.268
52.829
52.617
47.193
42.997
MeO
OMe
Cl
H
N
O
MeO
2g
200
150
100
50
0
ppm (t1)


ads 2332
7.79
7.77
7.59
7.55
7.44
7.37
7.27
7.26
7.25
7.19
7.17
6.97
6.91
6.05
6.01
4.92
4.87
4.47
4.42
4.40
4.35
3.99
3.96
3.72
3.49
MeO2C
CO2Me
Cl
H
N
O
NO2
2h
0.9
1.0
2.0
2.2
2.0
1.0
2.0
1.0
2.9
2.9
5.0
ppm (t1)

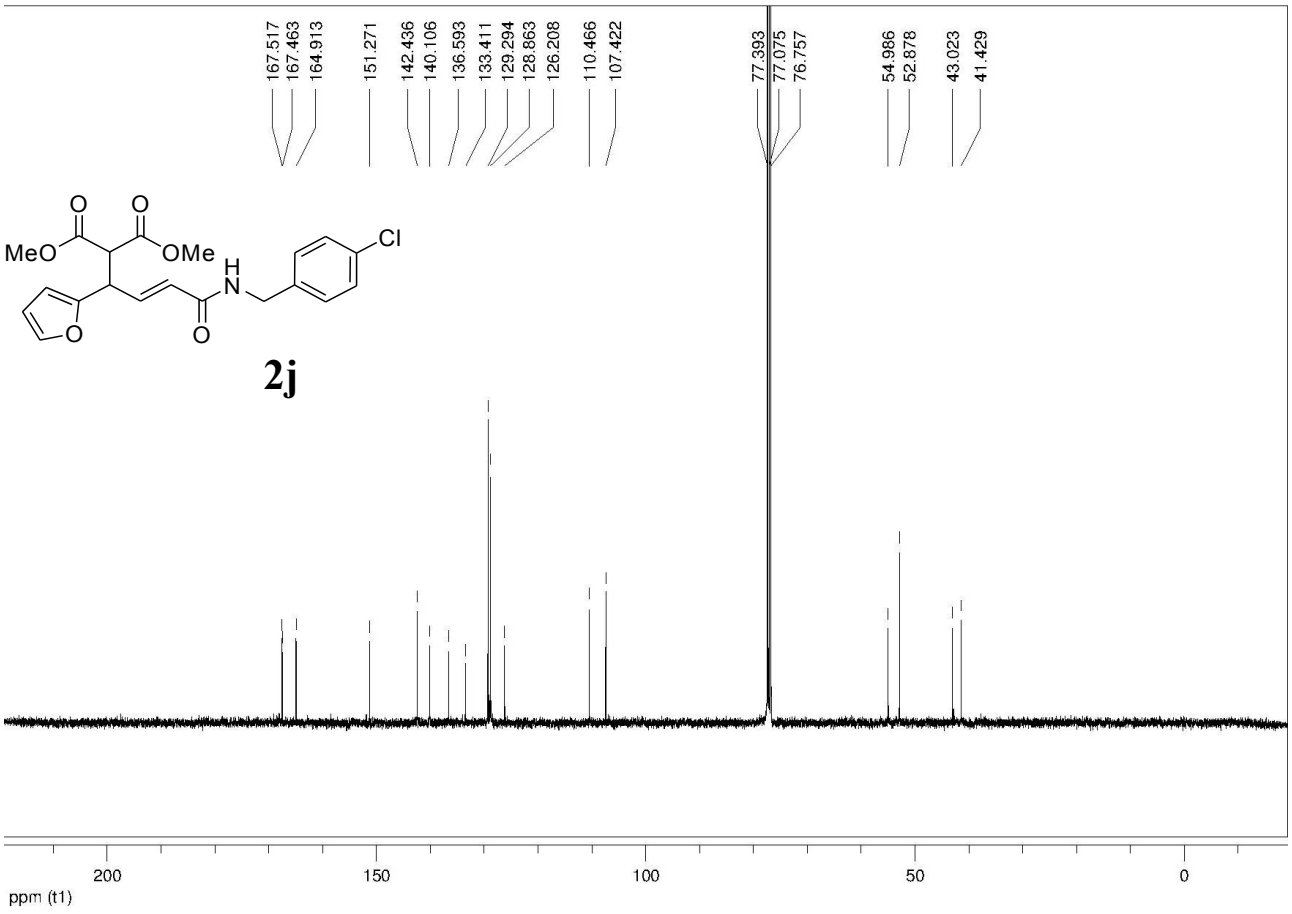

ads 2332
167.2
166.8
164.7
150.0
140.8
136.4
133.3
133.3
133.0
129.2
128.8
128.3
126.6
124.7
77.3
77.0
76.7
56.5
53.0
52.9
43.0
41.4
MeO2C
CO2Me
Cl
H
N
O
NO2
2h
150
100
50
ppm (t1)


ads 2333
7.80
7.77
7.59
7.54
7.43
7.36
7.26
7.20
7.17
6.96
6.90
6.85
6.83
6.00
5.96
5.80
4.91
4.86
4.45
4.39
4.39
4.34
3.98
3.95
3.78
3.73
3.49
MeO2C
CO2Me
OMe
H
N
O
NO2
2i
5.0
ppm (t1)

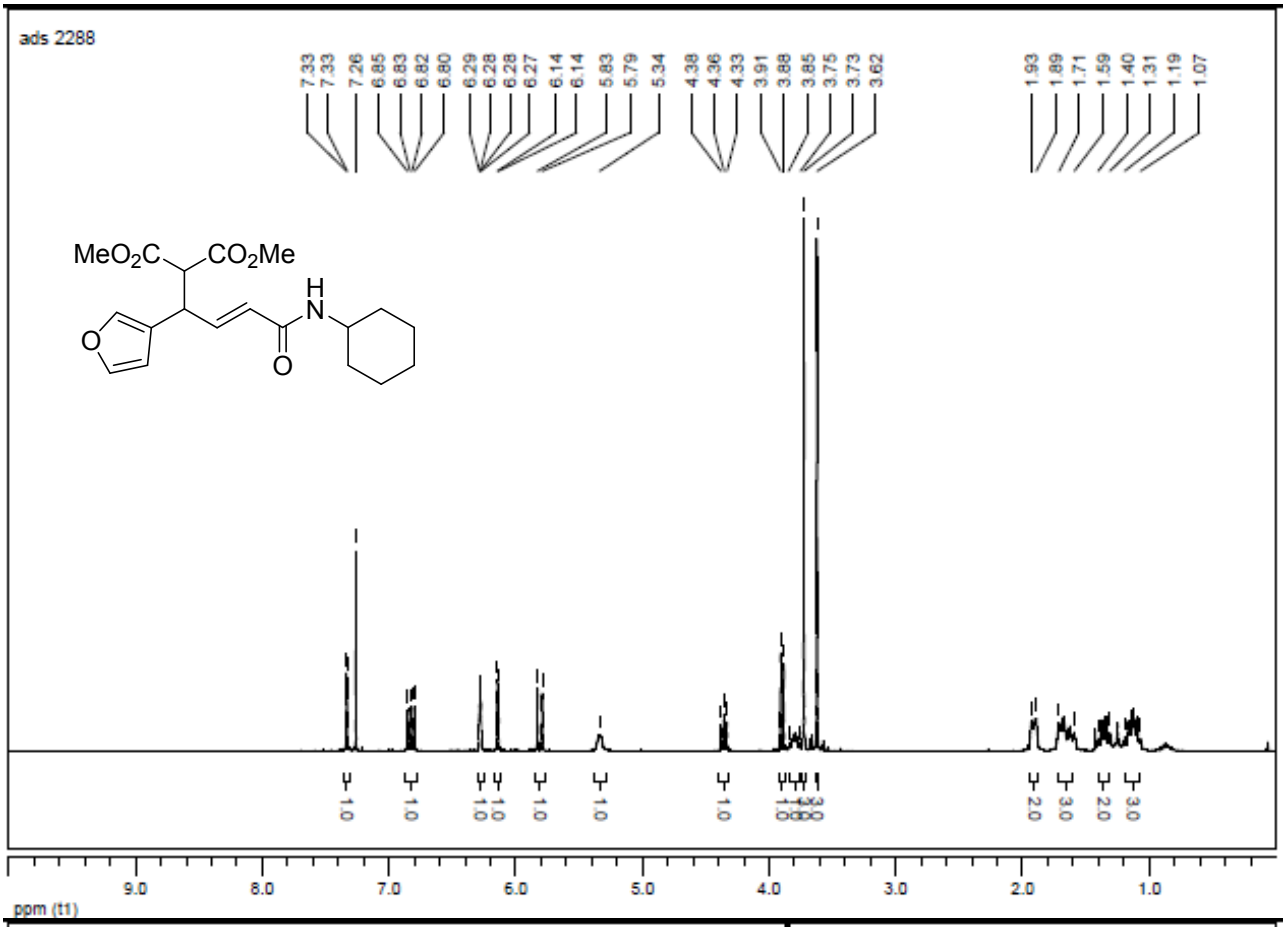

ads 2333
167.2
166.8
164.5
159.1
150.1
140.5
133.4
132.9
129.9
129.3
129.2
128.2
126.8
124.7
114.1
77.3
77.0
76.7
56.6
55.3
53.0
52.8
43.3
41.3
MeO2C
CO2Me
OMe
H
N
O
NO2
2i
150
100
50
ppm (t1)

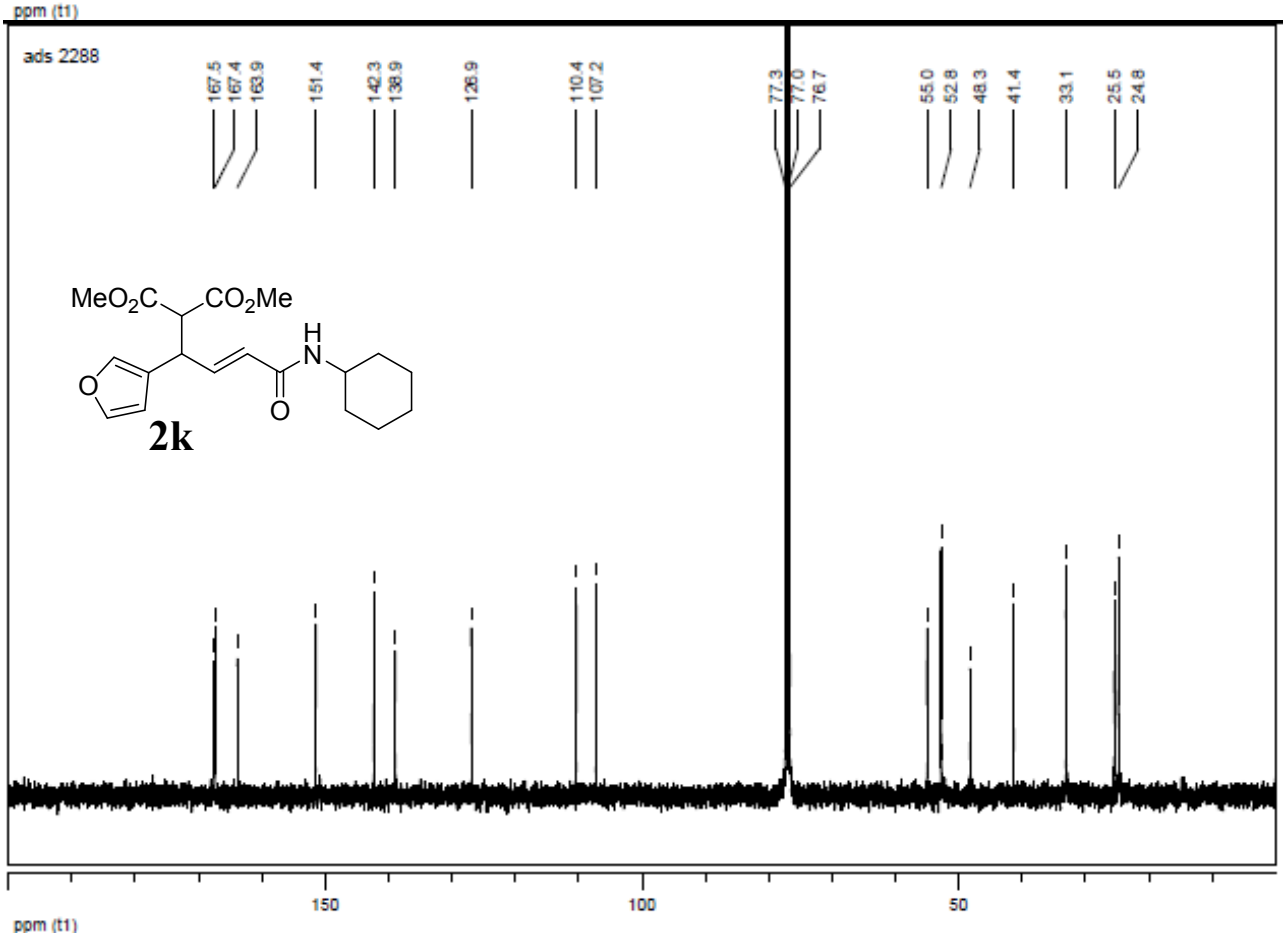

7.322
7.281
7.260
7.198
7.177
6.928
6.907
6.890
6.869
6.271
6.135
5.927
5.887
5.850
4.421
4.382
4.359
4.336
3.906
3.880
3.710
3.610
MeO
OMe
O
O
H
N
Cl
O
O
1.00
0.96
0.94
0.95
2.02
0.95
3.00
10.0
ppm (t1)
5.0
0.0

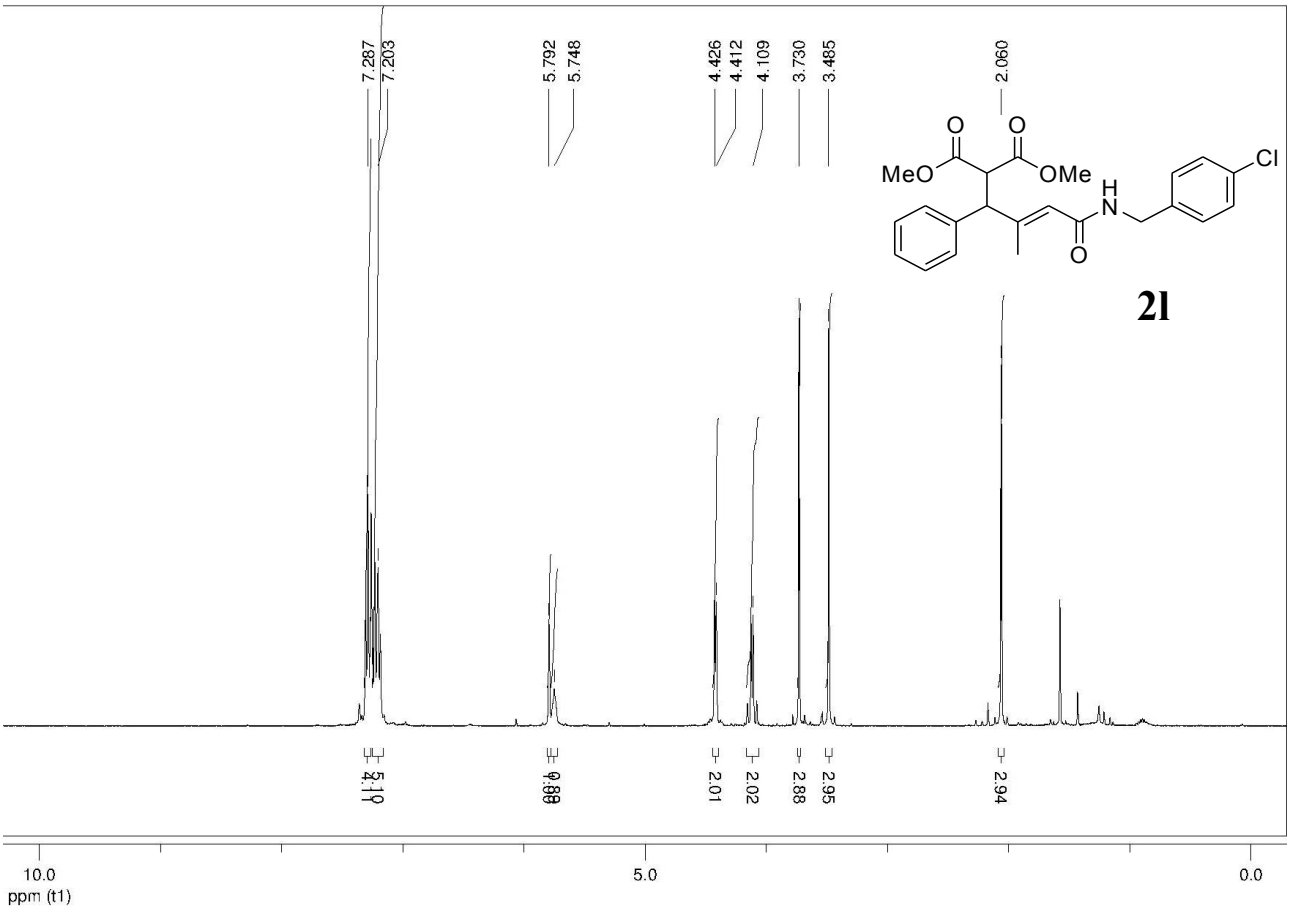
2j

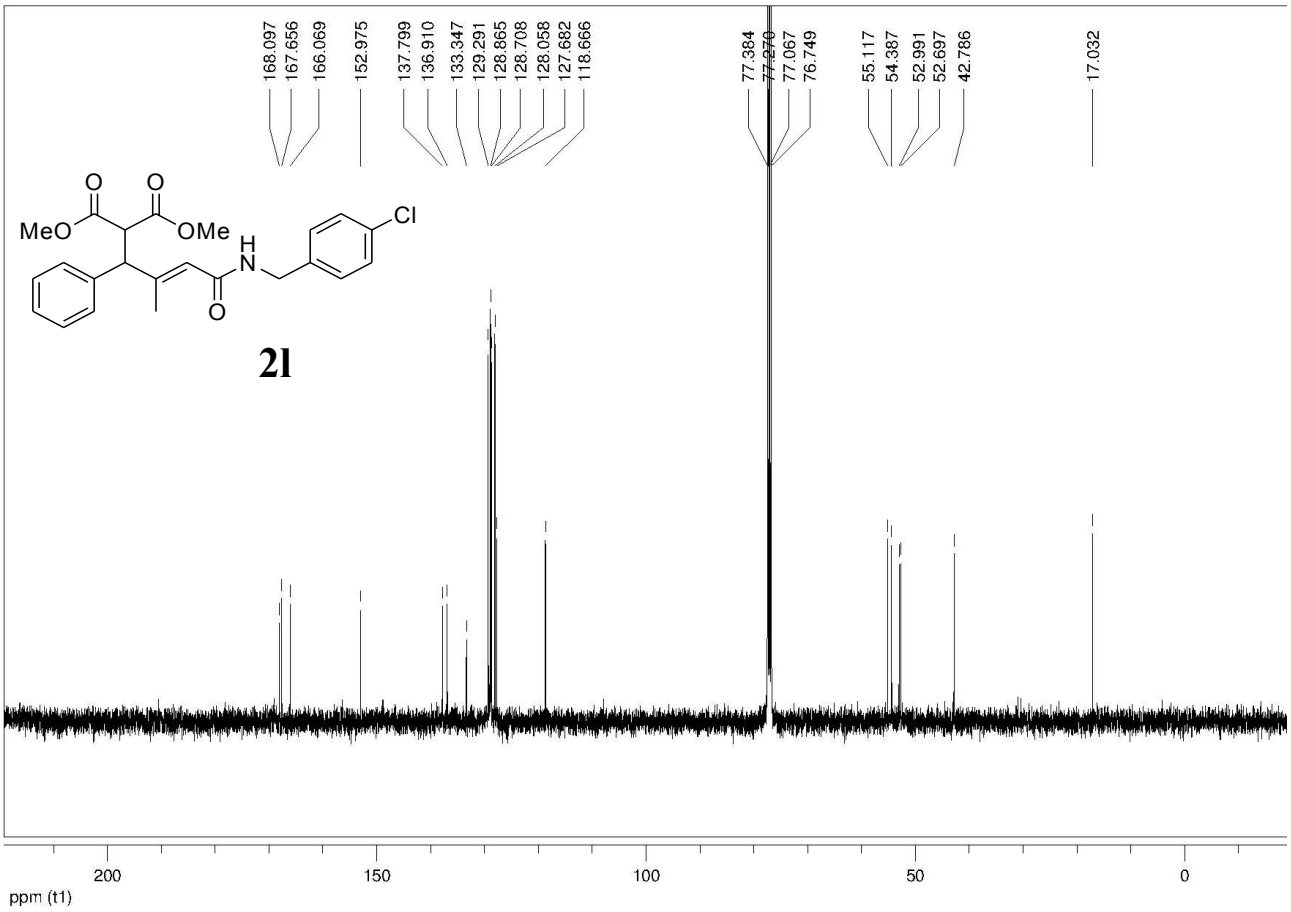

167.517
167.463
164.913
151.271
142.436
140.106
136.593
133.411
129.294
128.863
126.208
110.466
107.422
77.393
77.075
76.757
54.986
52.878
43.023
41.429
MeO
OMe
O
O
H
N
Cl
O
O
200
150
100
50
0
ppm (t1)

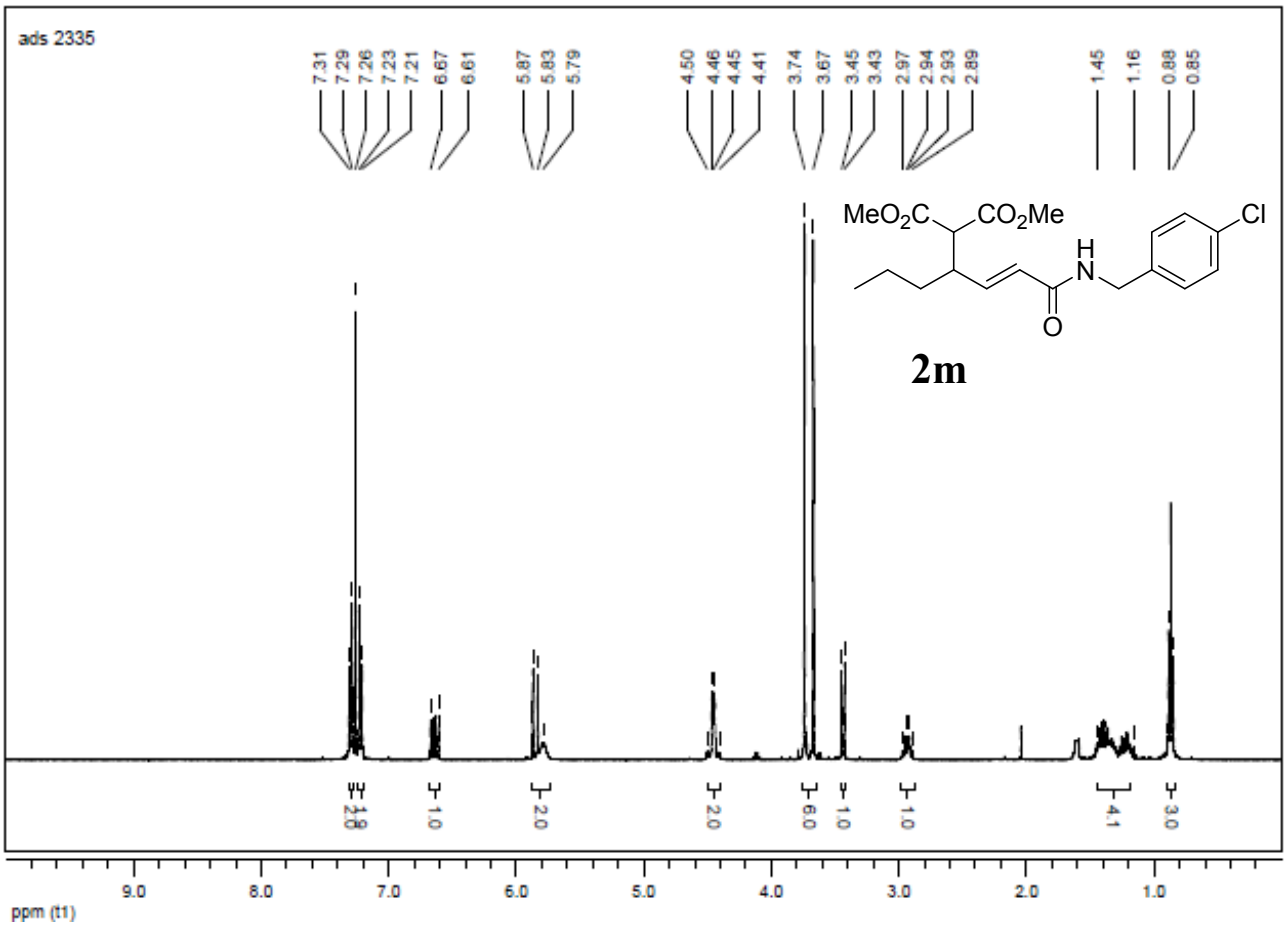
2j

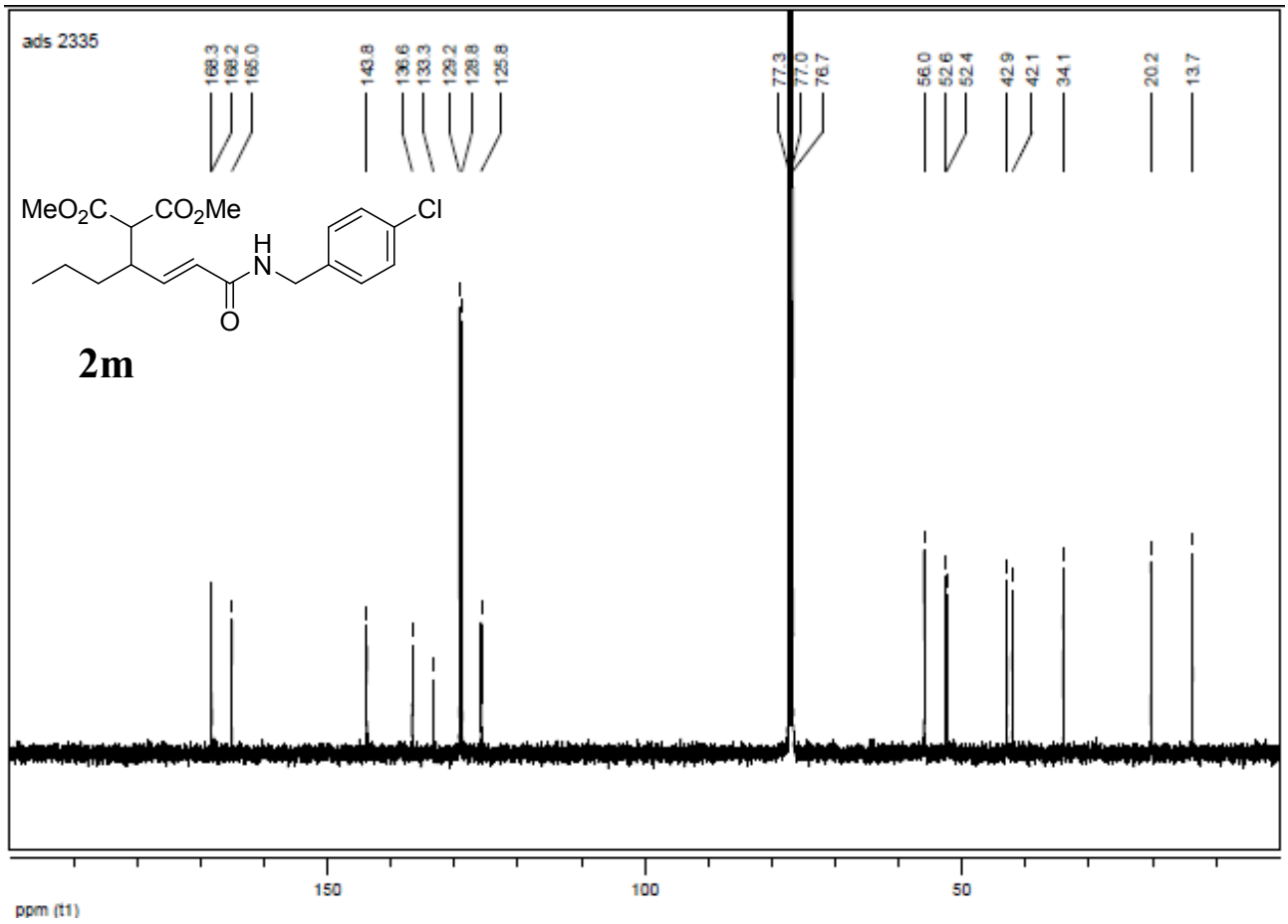

ads 2288
7.33
7.33
7.26
6.85
6.83
6.82
6.80
6.29
6.28
6.28
6.27
6.14
6.14
5.83
5.79
5.34
4.38
4.36
4.33
3.91
3.88
3.85
3.75
3.73
3.62
1.93
1.89
1.71
1.59
1.40
1.31
1.19
1.07
MeO2C
CO2Me
H
N
O
O
1.0
1.0
1.0
1.0
1.0
1.0
3.0
2.0
3.0
2.0
3.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

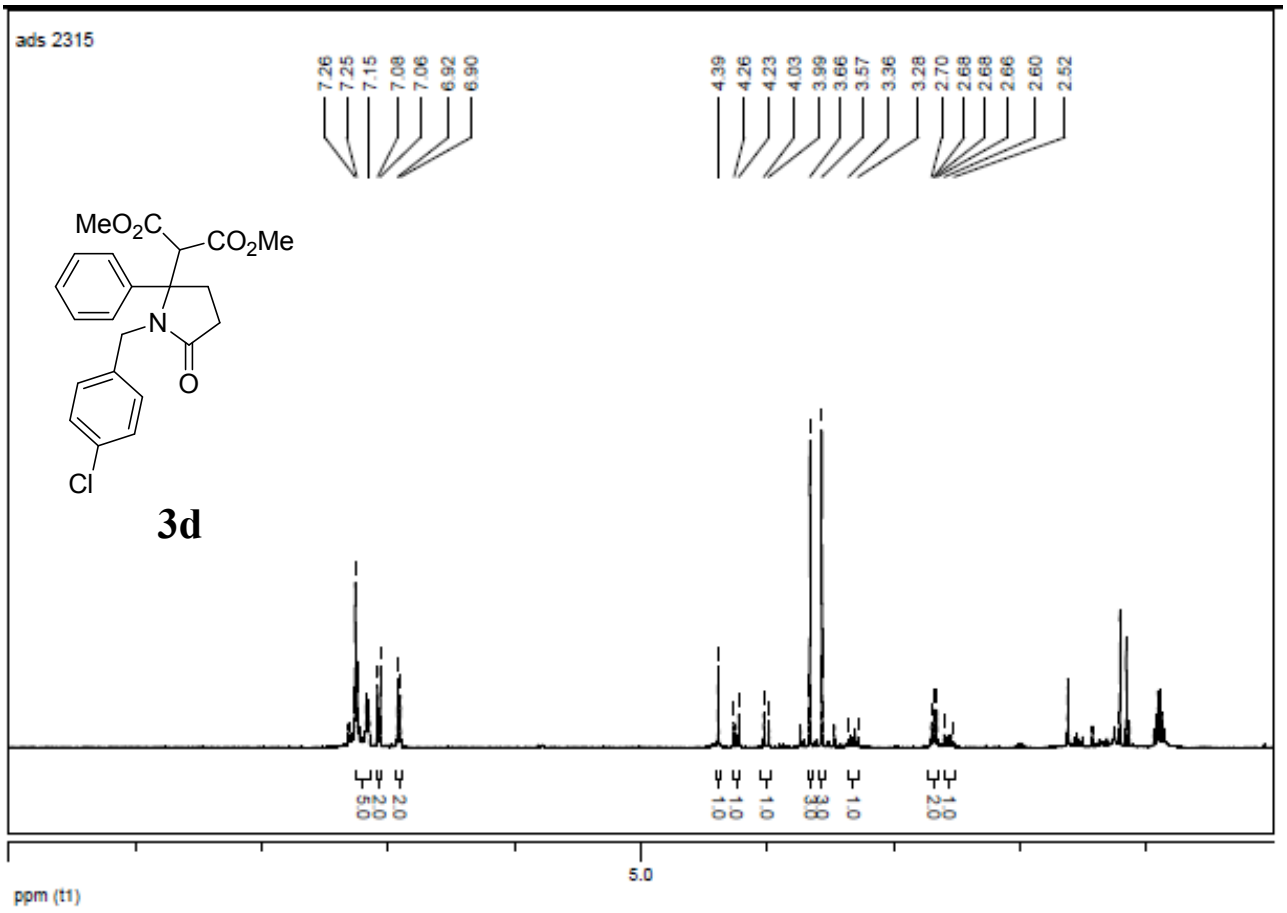

ads 2288
167.5
167.4
163.9
151.4
142.3
138.9
126.9
110.4
107.2
77.3
77.0
76.7
55.0
52.8
48.3
41.4
33.1
25.5
24.8
MeO2C
CO2Me
H
N
O
O
2k
150
100
50
ppm (t1)

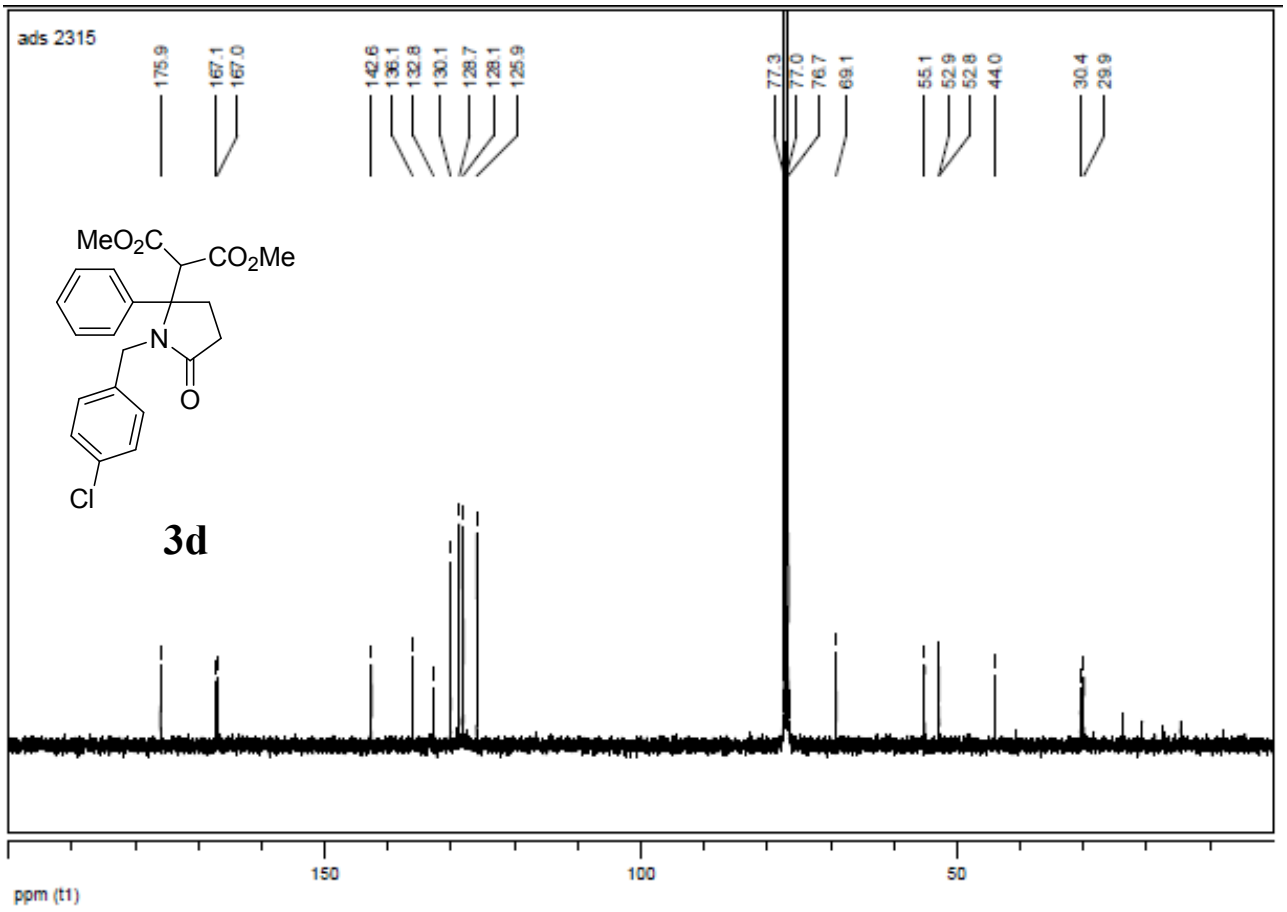

7.287
7.203
5.792
5.748
4.426
4.412
4.109
3.730
3.485
2.060
MeO
OMe
H
N
Cl
O
2l
3.10
2.00
2.01
2.02
2.88
2.95
2.94
10.0
5.0
0.0
ppm (t1)

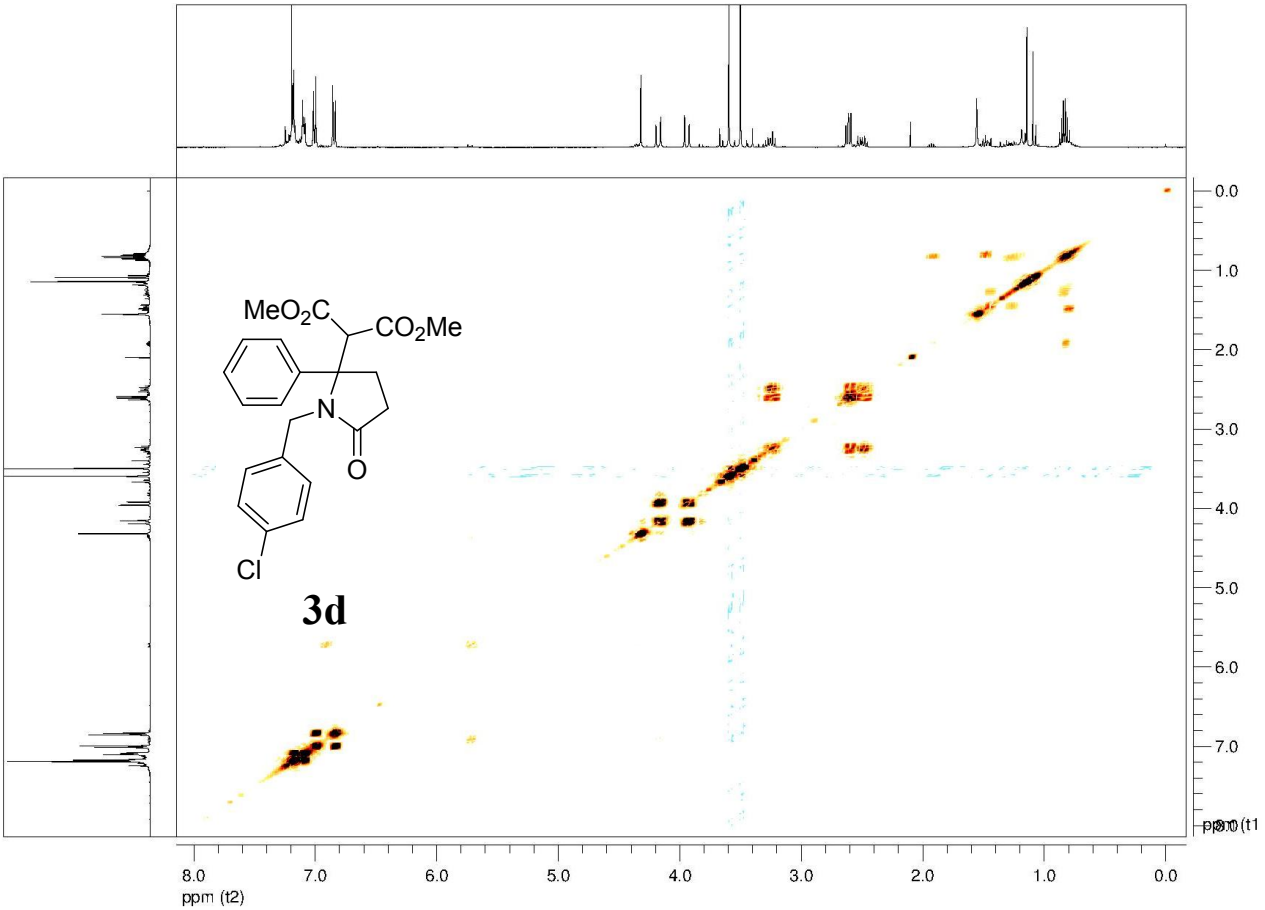

168.097
167.656
166.069
152.975
137.799
136.910
133.347
129.291
128.865
128.708
128.058
127.682
118.666
77.384
77.067
76.749
55.117
54.387
52.991
52.697
42.786
17.032
MeO
OMe
H
N
Cl
O
2l
200
150
100
50
0
ppm (t1)

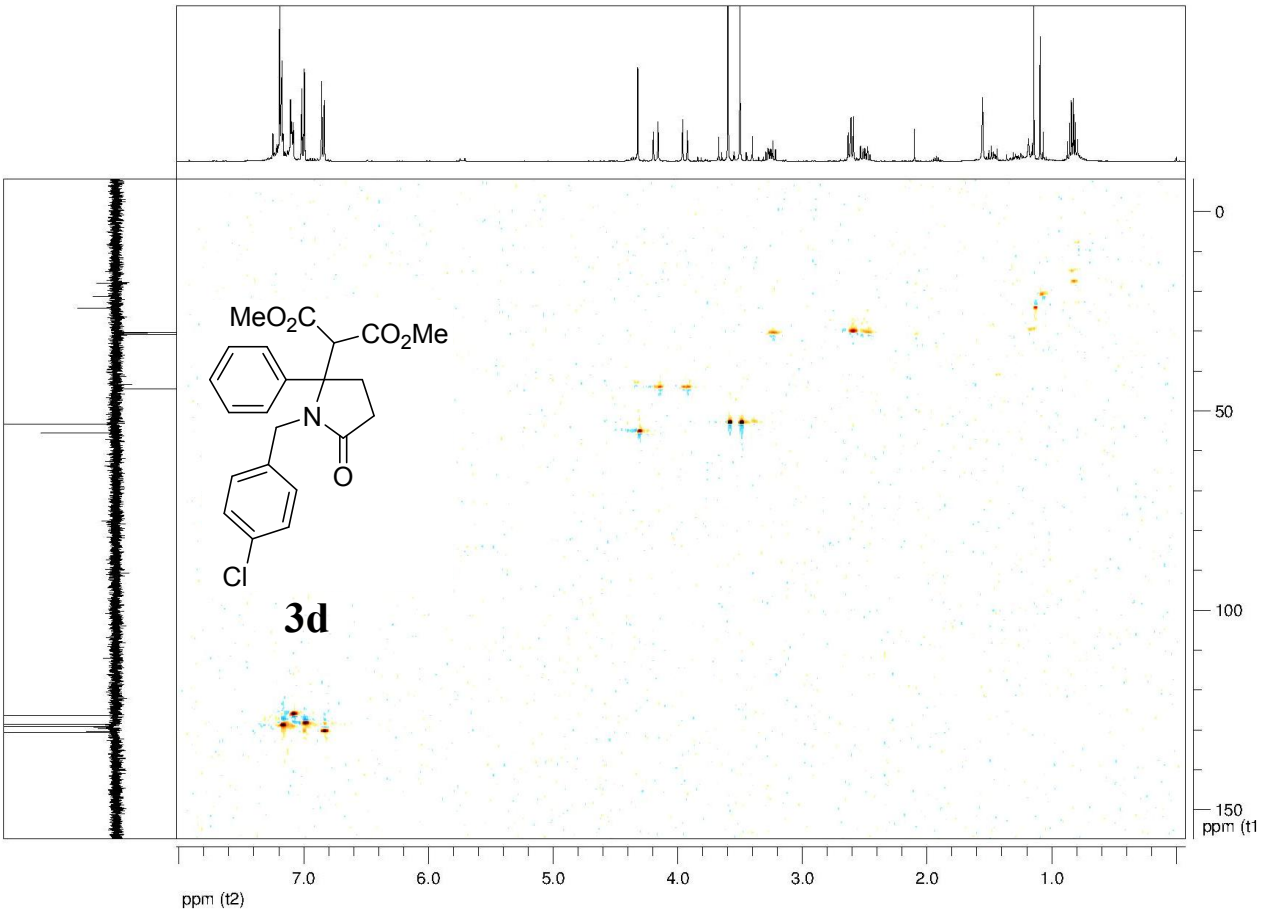

ads 2335
7.31
7.29
7.26
7.23
7.21
6.67
6.61
5.87
5.83
5.79
4.50
4.46
4.45
4.41
3.74
3.67
3.45
3.43
2.97
2.94
2.93
2.89
1.45
1.16
0.88
0.85
MeO2C
CO2Me
Cl
H
N
O
2m
2.18
1.10
2.20
2.00
6.00
1.00
1.10
4.11
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)

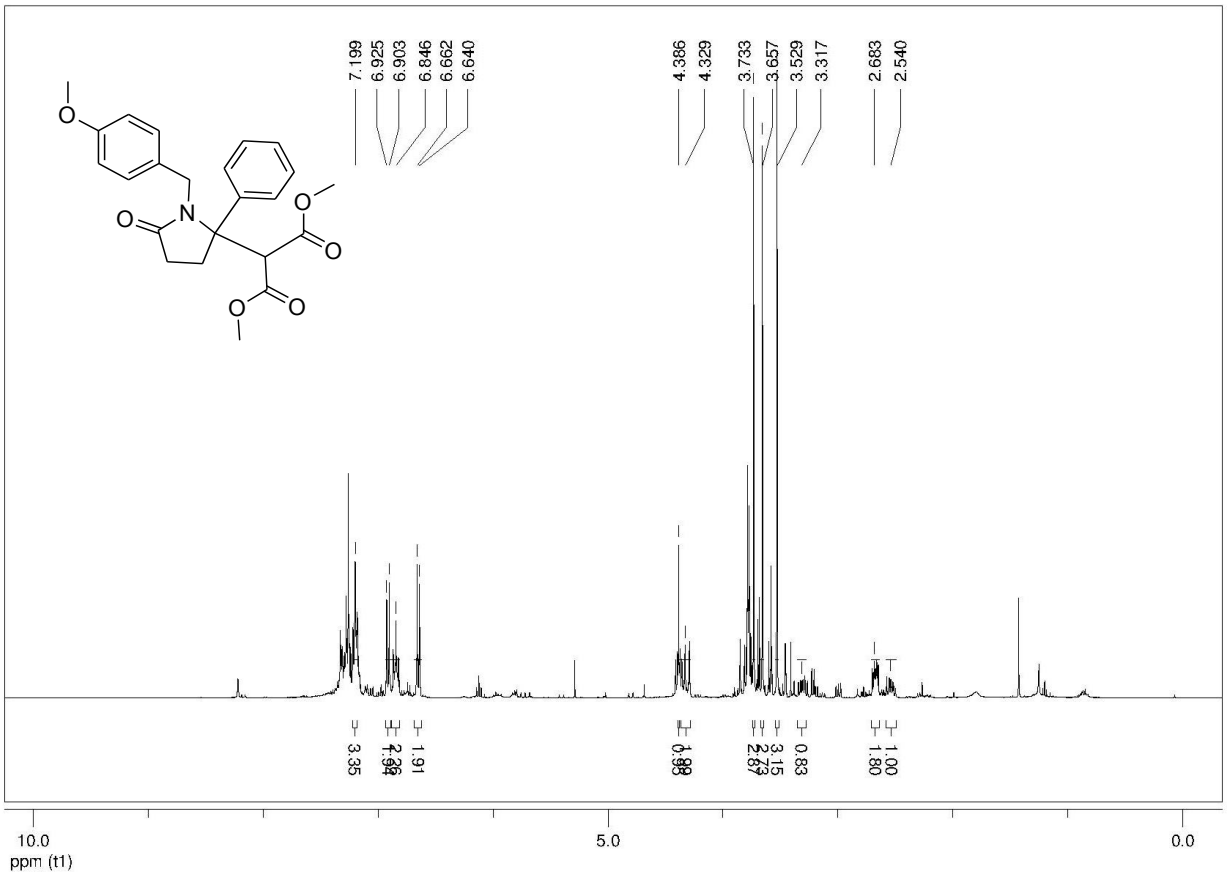

ads 2335
168.3
168.2
165.0
143.8
136.6
133.3
129.2
128.8
125.8
77.3
77.0
76.7
56.0
52.6
52.4
42.9
42.1
34.1
20.2
13.7
MeO2C
CO2Me
Cl
H
N
O
2m
150
100
50
ppm (t1)

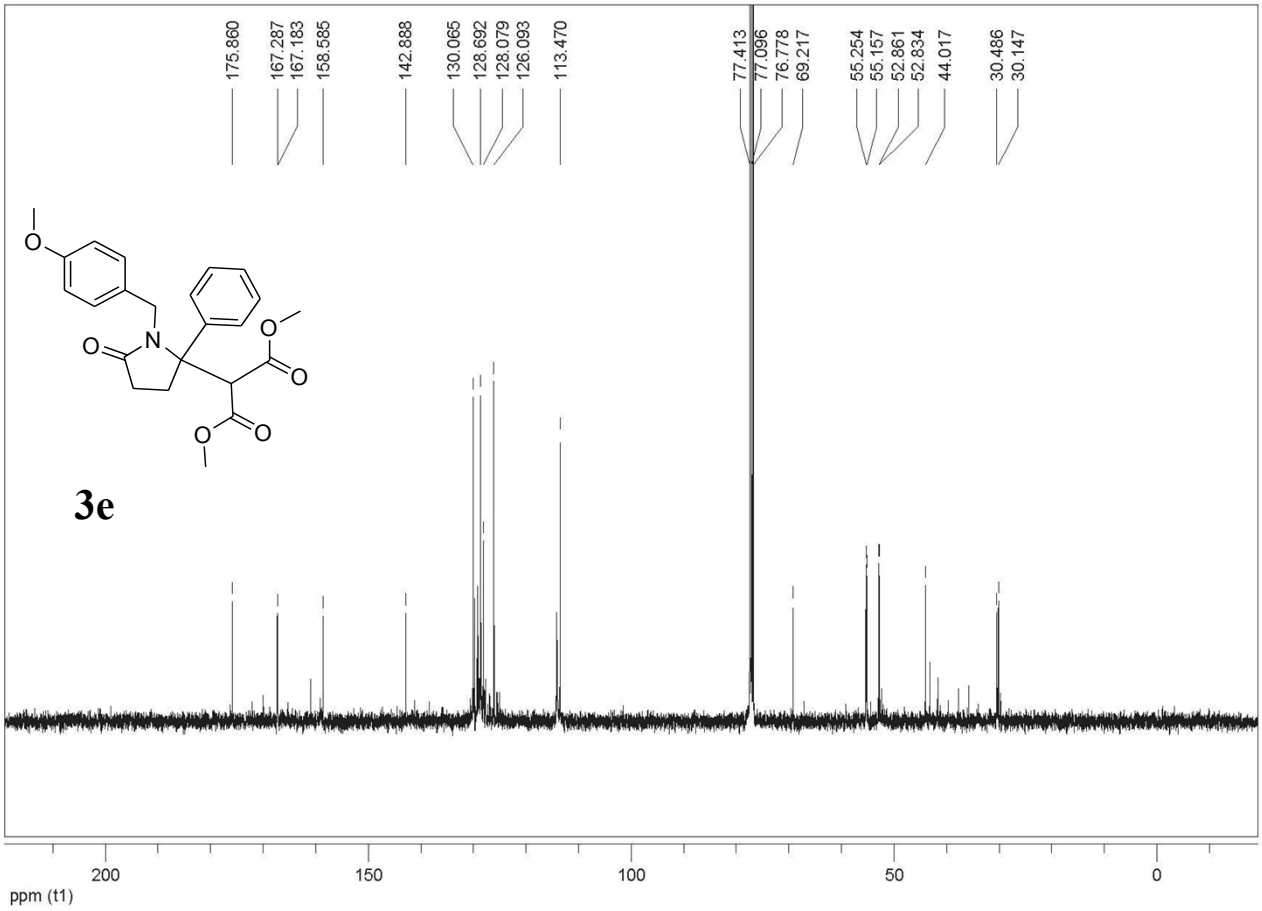

ads 2315
7.26
7.25
7.15
7.08
7.06
6.92
6.90
4.39
4.26
4.23
4.03
3.99
3.66
3.57
3.36
3.28
2.70
2.68
2.68
2.66
2.60
2.52
MeO2C
CO2Me
N
O
Cl
3d
5.0
ppm (t1)


ads 2315
175.9
167.1
167.0
142.6
136.1
132.8
130.1
128.7
128.1
125.9
77.3
77.0
76.7
69.1
55.1
52.9
52.8
44.0
30.4
29.9
MeO2C
CO2Me
N
O
Cl
3d
150
100
50
ppm (t1)

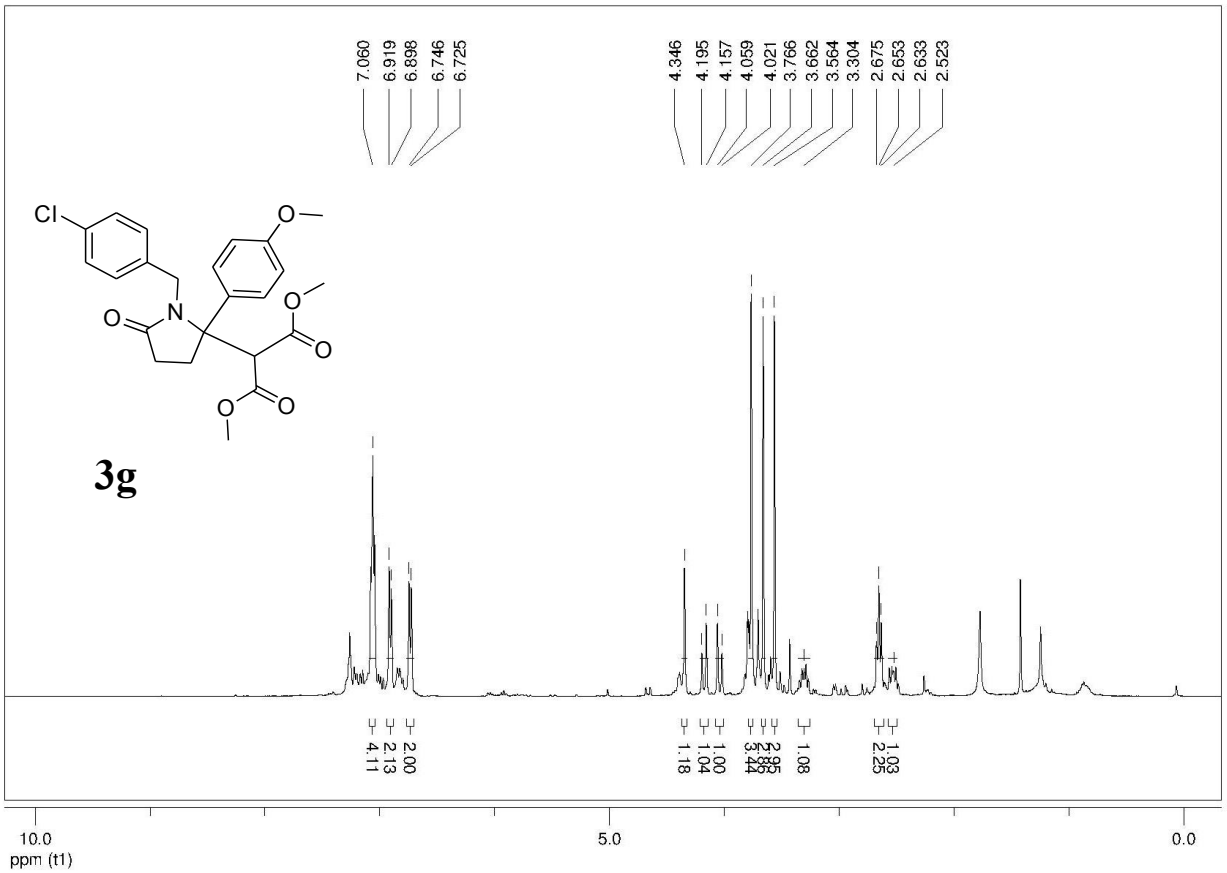

MeO2C
CO2Me
N
O
Cl
3d
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t2)

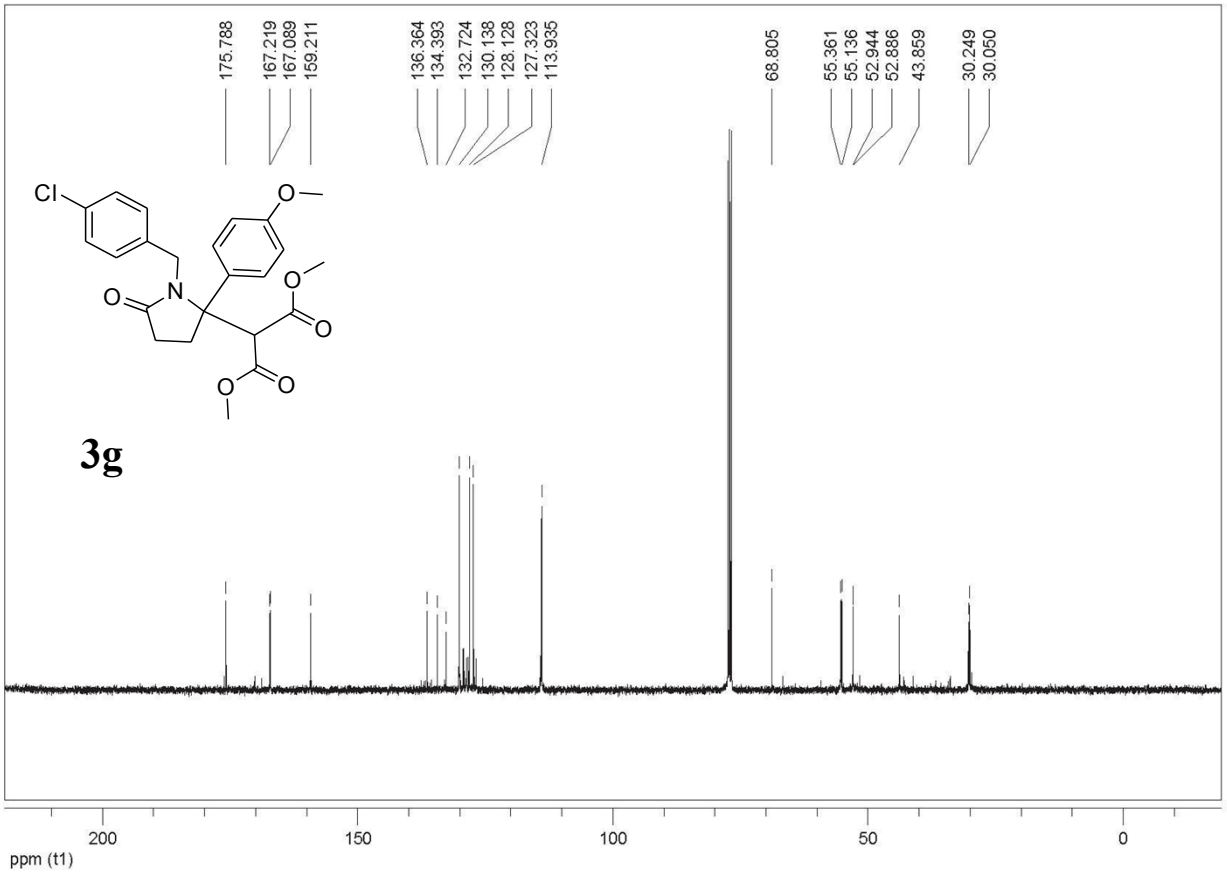

MeO2C
CO2Me
N
O
Cl
3d
0
50
100
150
ppm (t1
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t2)

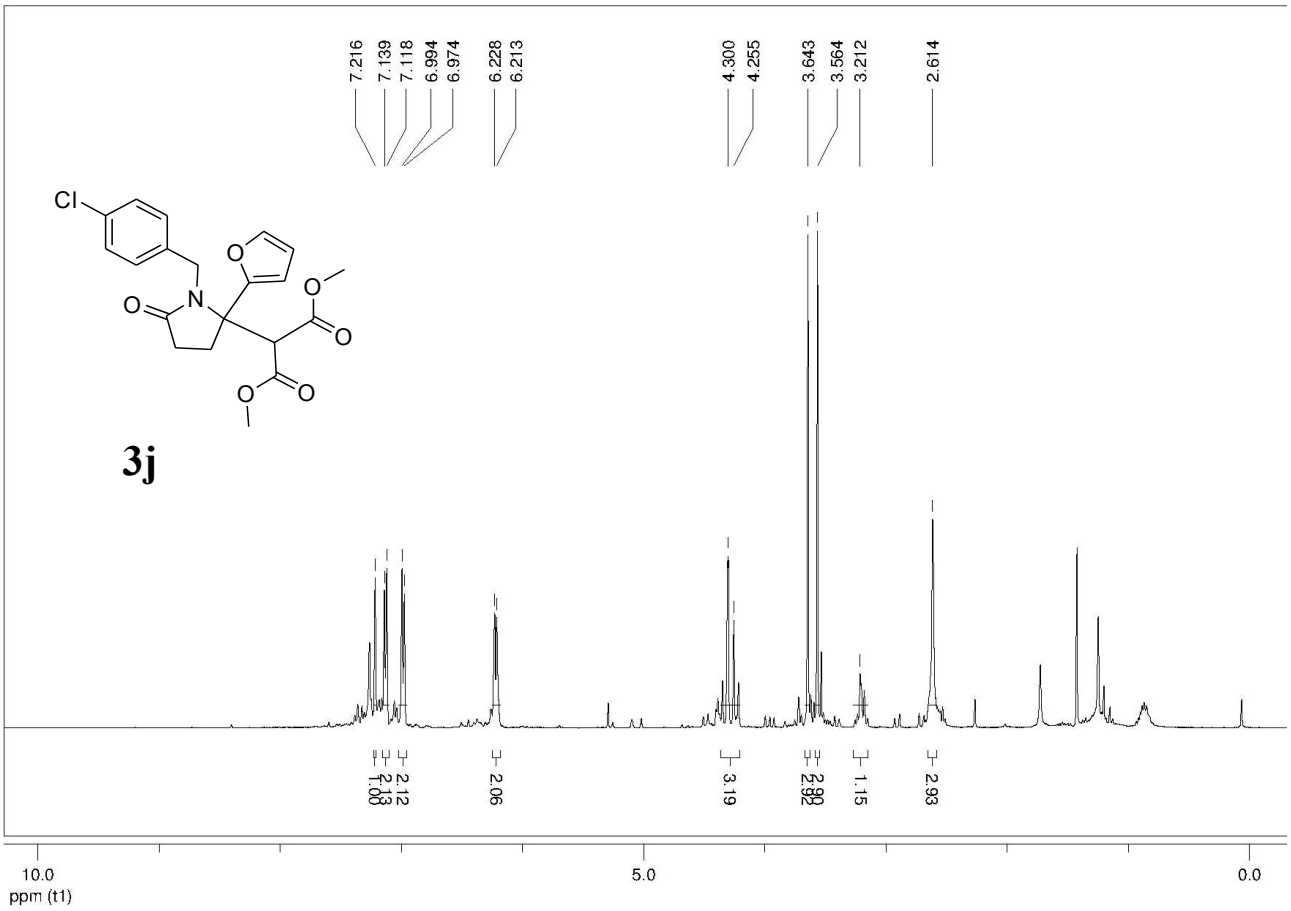

7.199
6.925
6.903
6.846
6.662
6.640
4.386
4.329
3.733
3.657
3.529
3.317
2.683
2.540
3.36
2.26
1.91
0.83
1.80
1.00
10.0
5.0
0.0
ppm (t1)

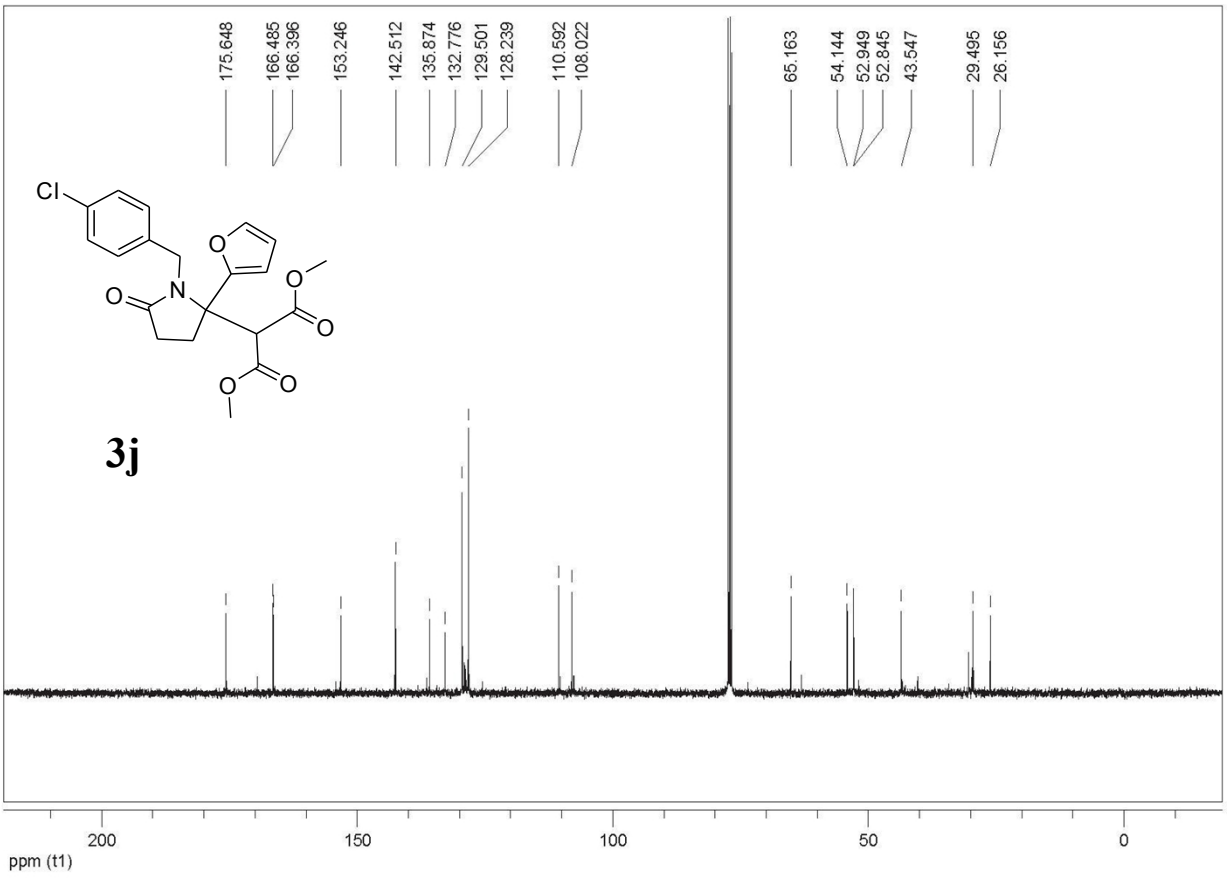

175.860
167.287
167.183
158.585
142.888
130.065
128.692
128.079
126.093
113.470
77.413
77.096
76.778
69.217
55.254
55.157
52.861
52.834
44.017
30.486
30.147
200
150
100
50
0
ppm (t1)

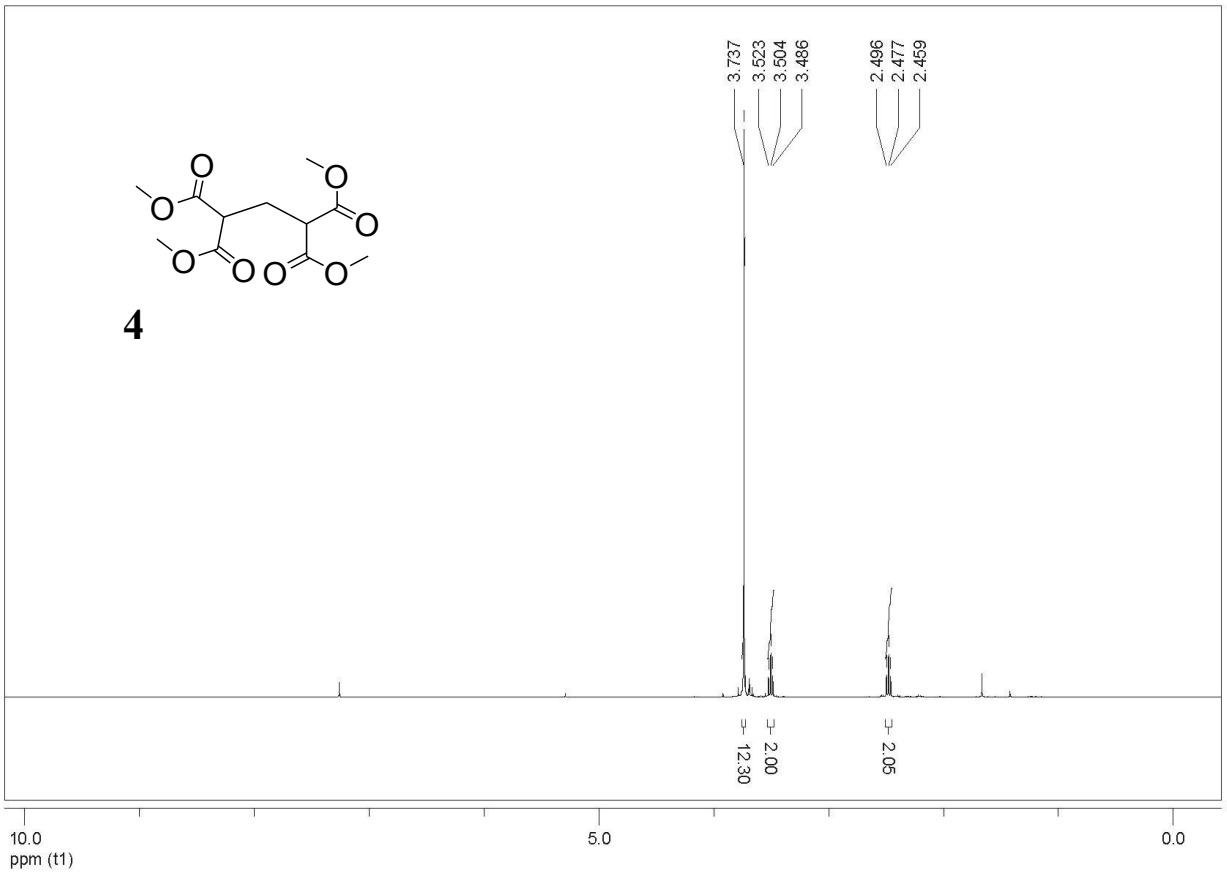
3e


7.060
6.919
6.898
6.746
6.725
4.346
4.195
4.157
4.059
4.021
3.766
3.662
3.564
3.304
2.675
2.653
2.633
2.523
Cl
O
N
O
O
O
O
O
3g
4.11
2.13
2.00
1.18
1.04
1.00
3.44
2.96
2.85
1.08
2.25
1.09
10.0
5.0
0.0
ppm (t1)

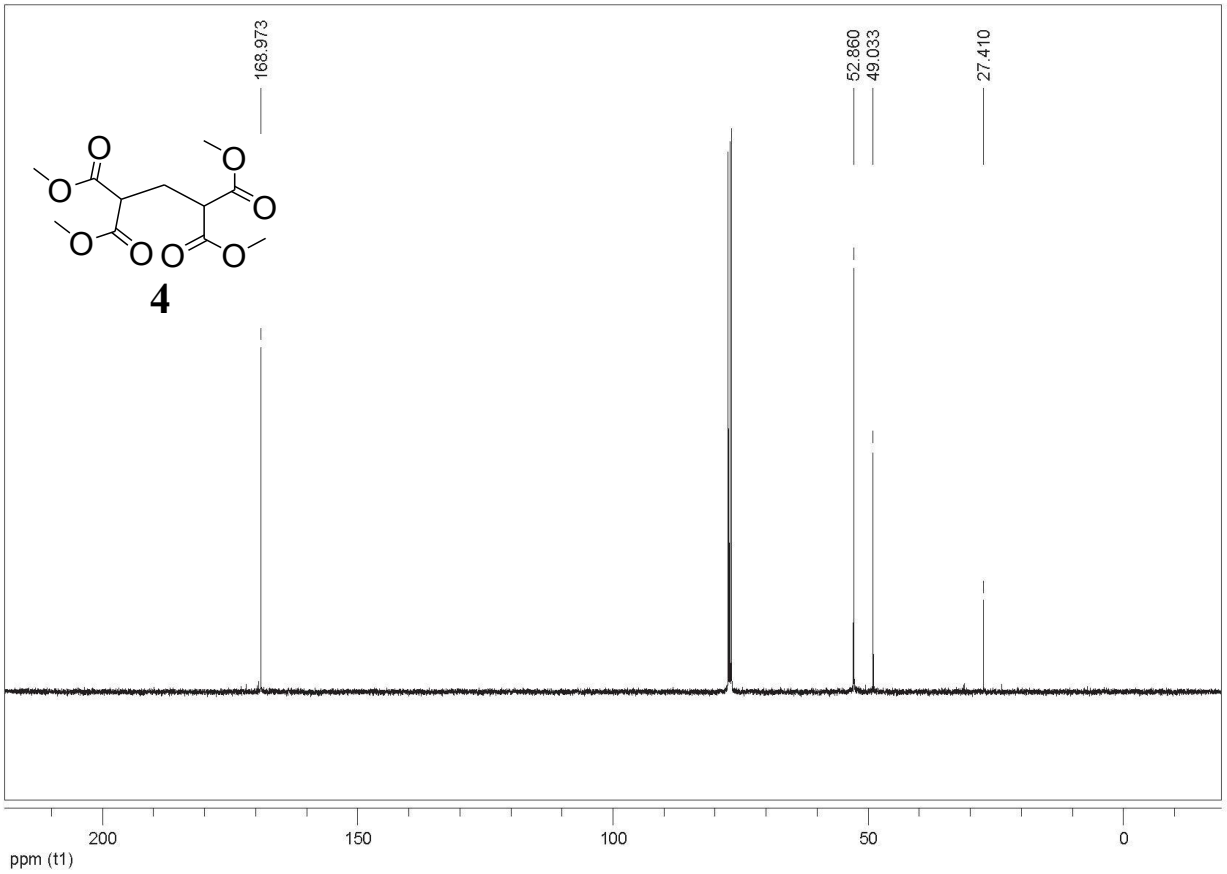

175.788
167.219
167.089
159.211
136.364
134.393
132.724
130.138
128.128
127.323
113.935
68.805
55.361
55.136
52.944
52.886
43.859
30.249
30.050
Cl
O
N
O
O
O
O
O
3g
200
150
100
50
0
ppm (t1)


7.216
7.139
7.118
6.994
6.974
6.228
6.213
4.300
4.255
3.643
3.564
3.212
2.614
Cl
O
N
O
O
O
O
O
O
3j
2.13
2.12
2.06
3.19
2.90
1.15
2.93
10.0
ppm (t1)
5.0
0.0

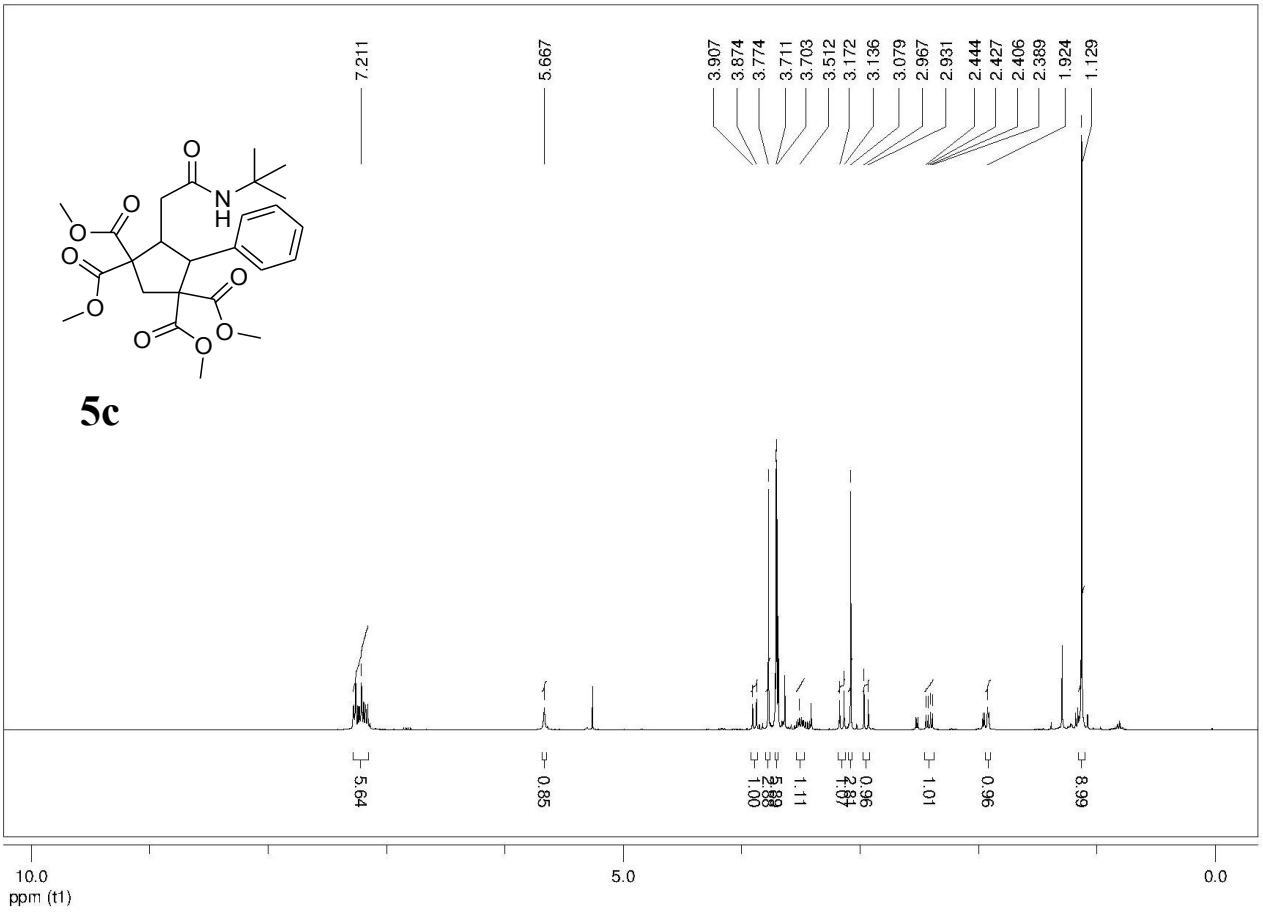

175.648
166.485
166.396
153.246
142.512
135.874
132.776
129.501
128.239
110.592
108.022
65.163
54.144
52.949
52.845
43.547
29.495
26.156
Cl
O
N
O
O
O
O
O
O
3j
200
150
100
50
0
ppm (t1)


3.737
3.523
3.504
3.486
2.496
2.477
2.459
12.30
2.00
2.05
10.0
5.0
0.0
ppm (t1)

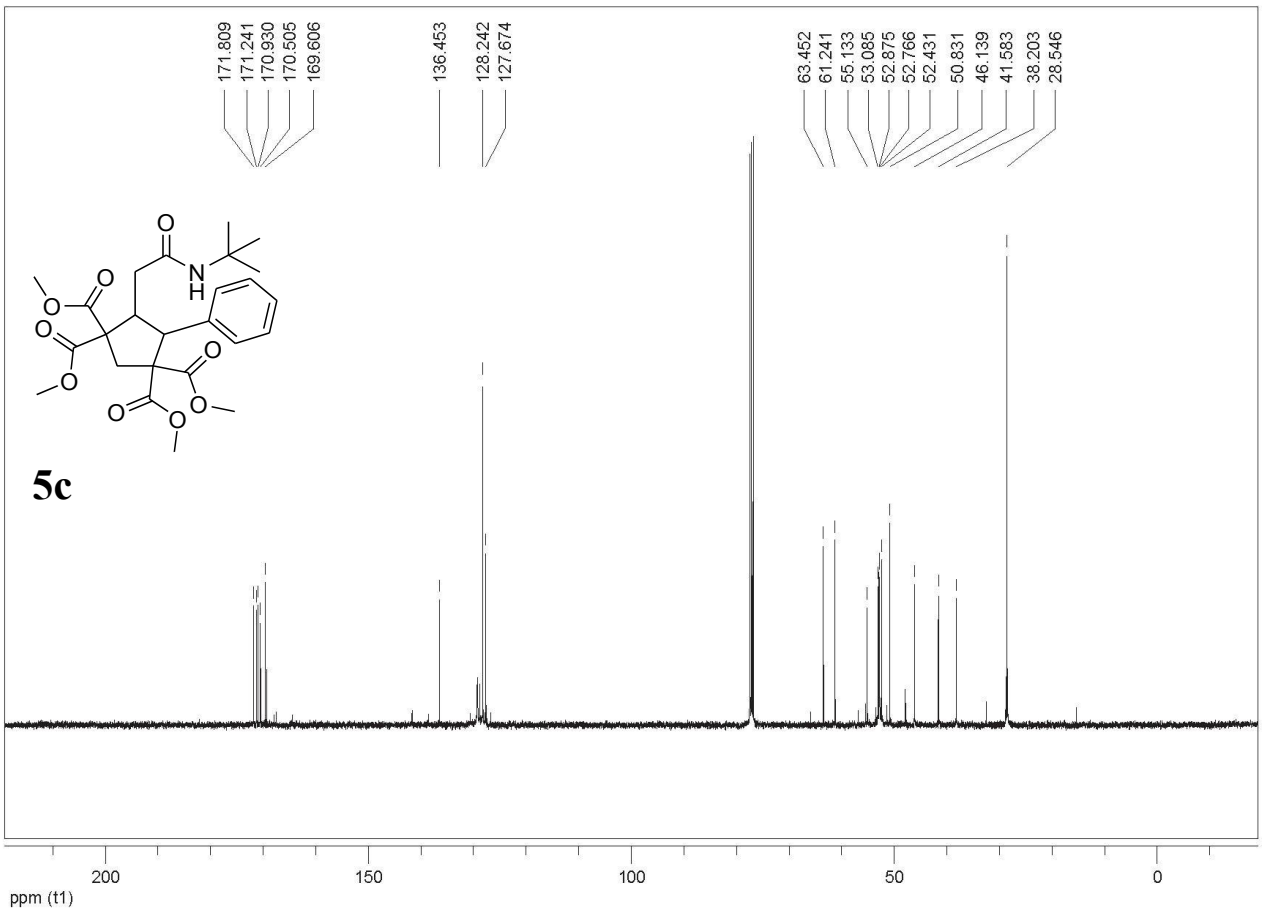
4


168.973
52.860
49.033
27.410
200
150
100
50
0
ppm (t1)

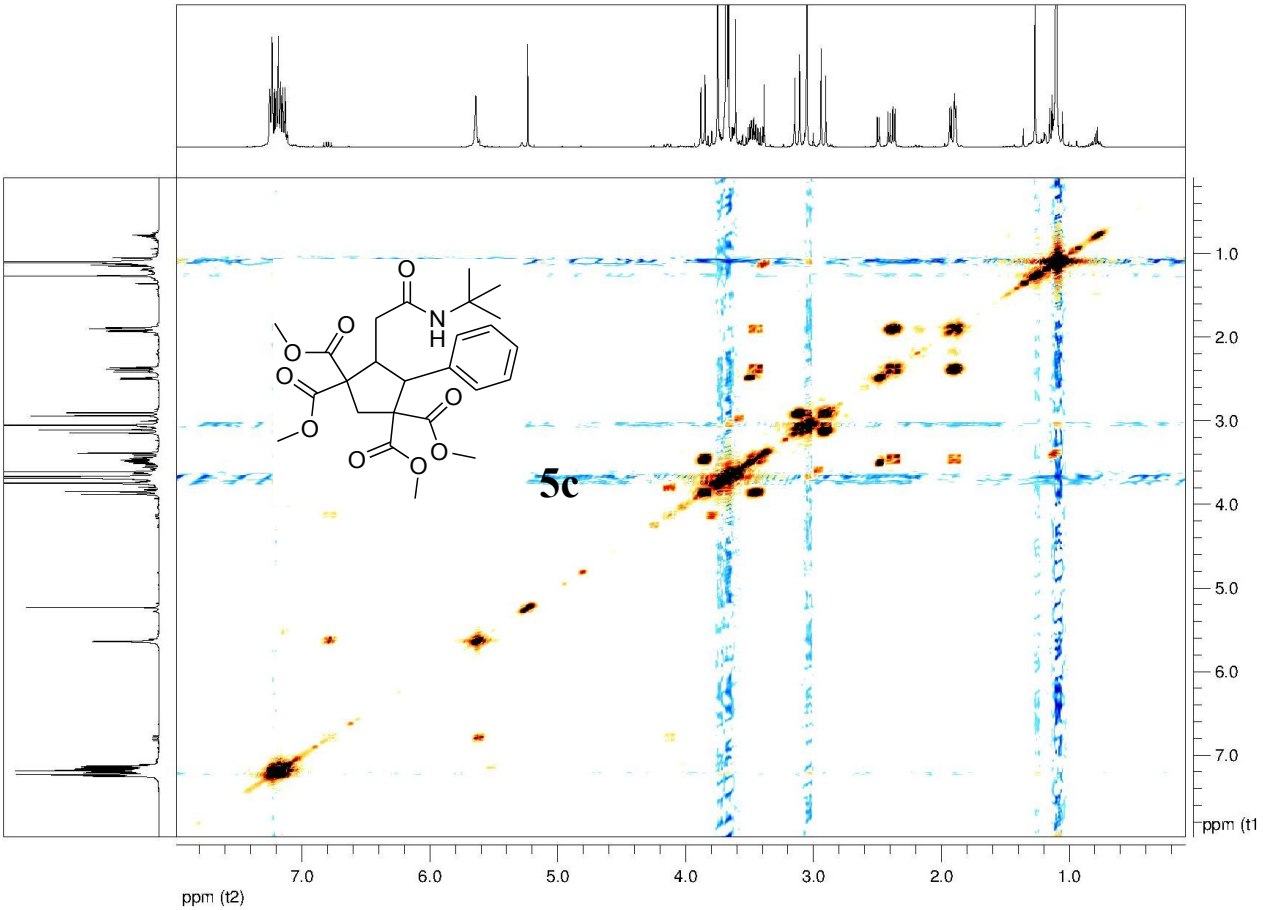
4

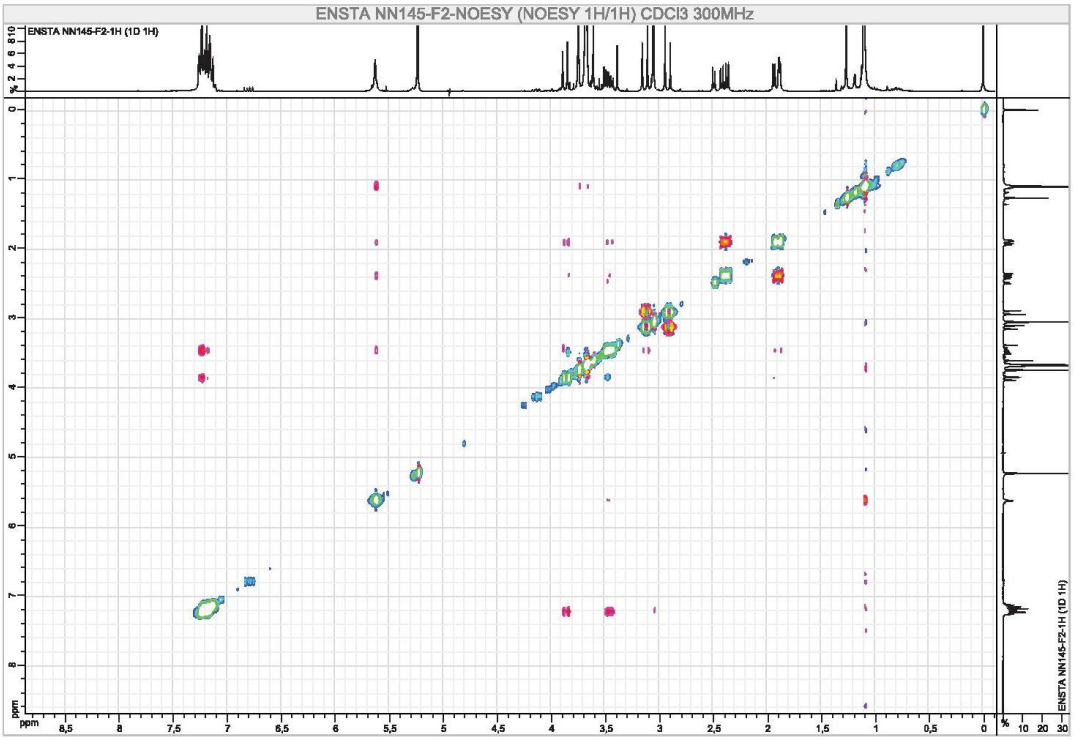

7.211
5.667
3.907
3.874
3.774
3.711
3.703
3.512
3.172
3.136
3.079
2.967
2.931
2.444
2.427
2.406
2.389
1.924
1.129
5.64
0.85
1.11
0.96
1.01
0.96
8.99
10.0
5.0
0.0
ppm (t1)

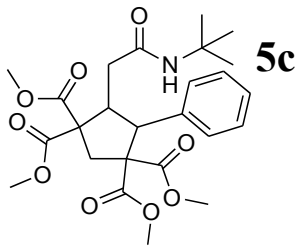
5c


171.809
171.241
170.930
170.505
169.606
136.453
128.242
127.674
63.452
61.241
55.133
53.085
52.875
52.766
52.431
50.831
46.139
41.583
38.203
28.546
200
150
100
50
0
ppm (t1)

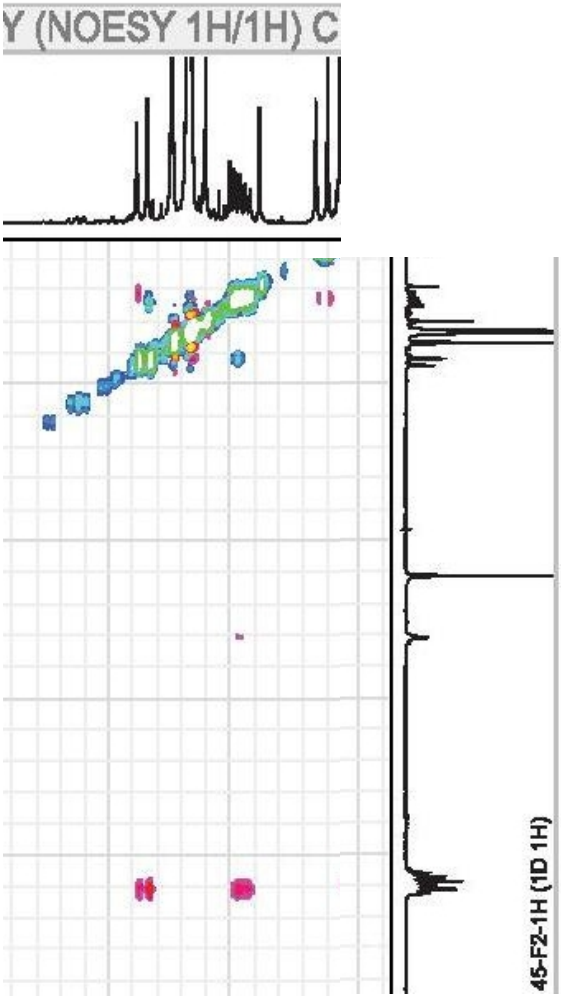
5c

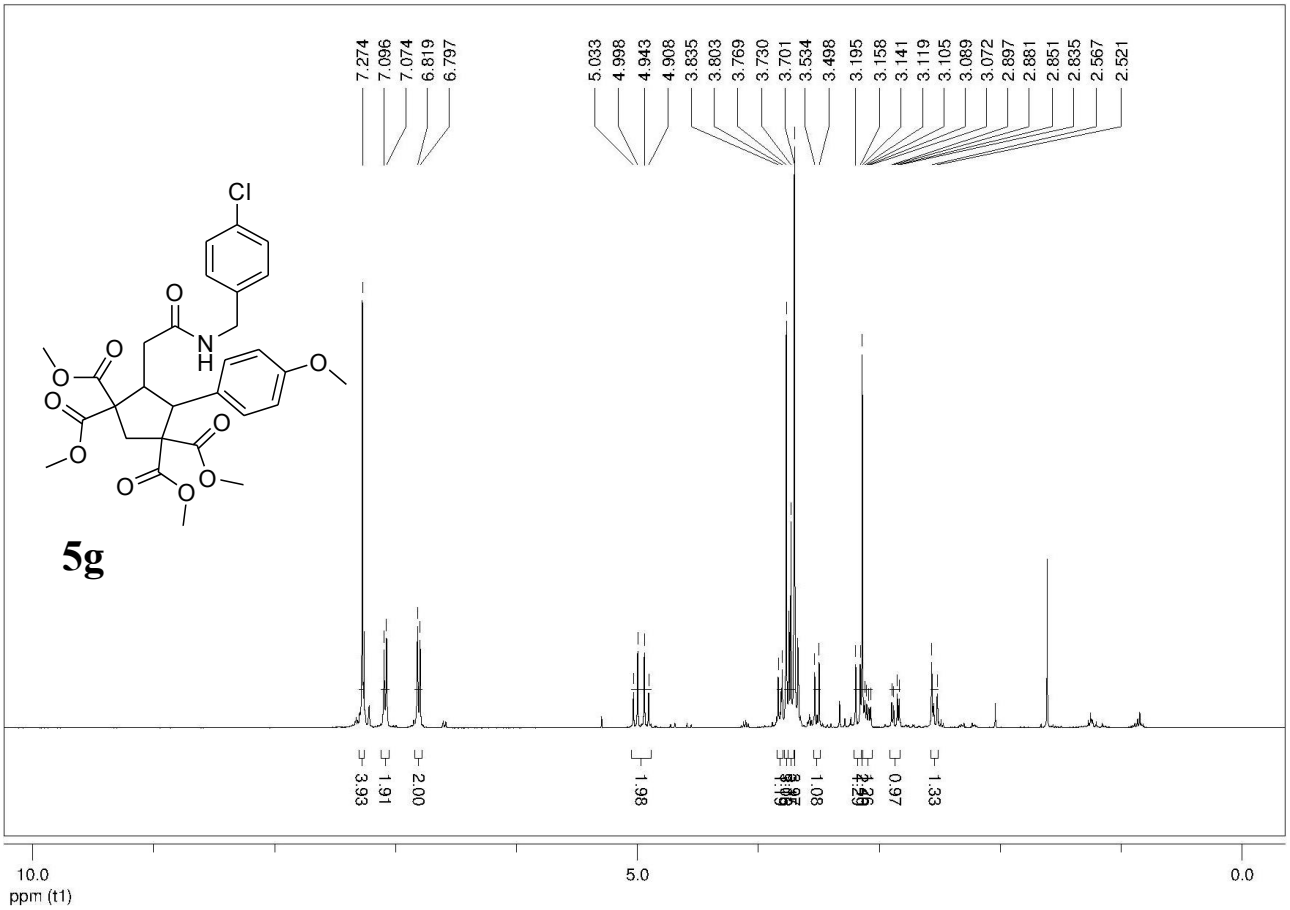

5c
ppm (t2)
ppm (t1
7.0
6.0
5.0
4.0
3.0
2.0
1.0

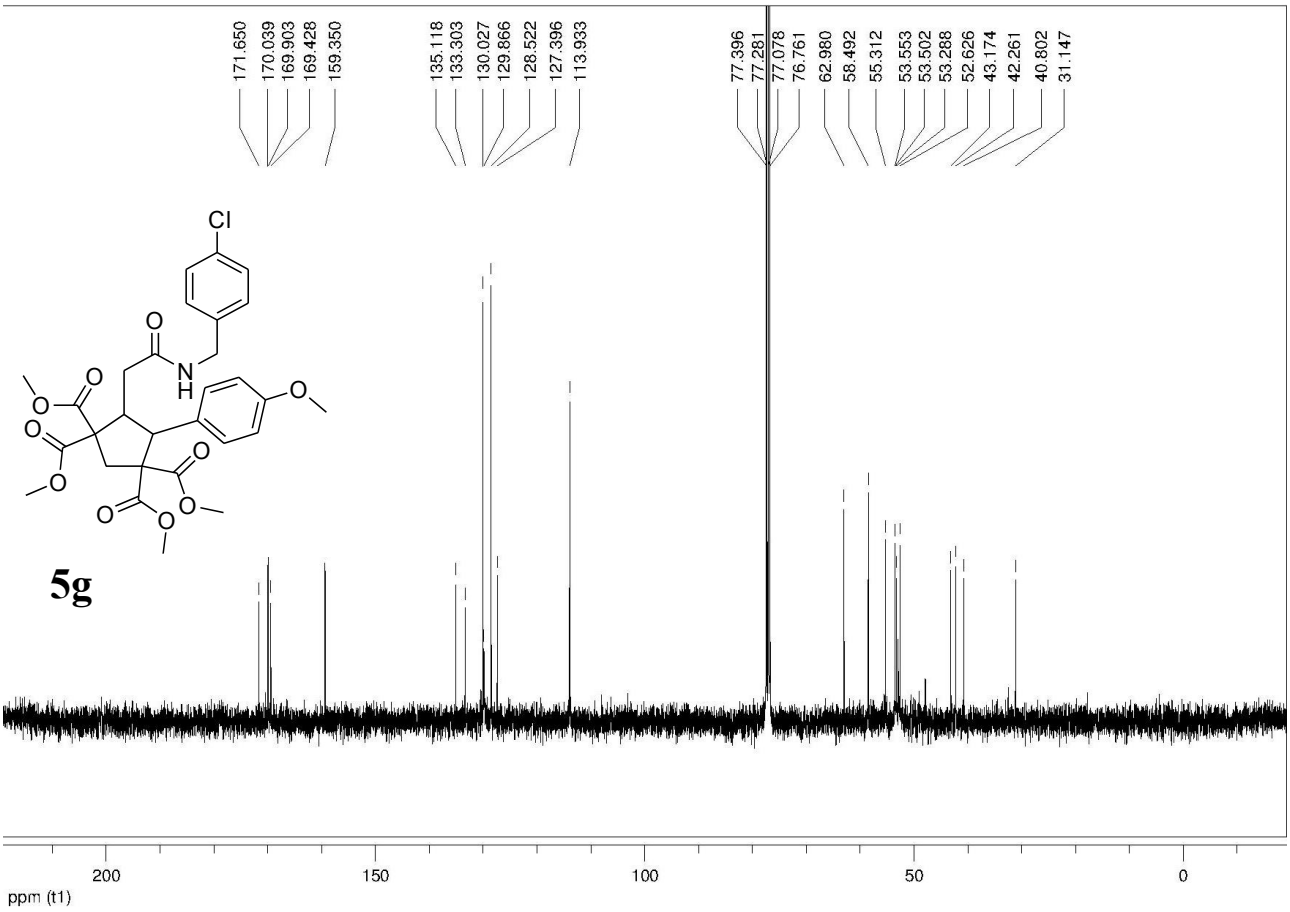

ENSTA NN145-F2-NOESY (NOESY 1H/1H) CDCl3 300MHz
ENSTA NN145-F2-1H (1D 1H)
ppm

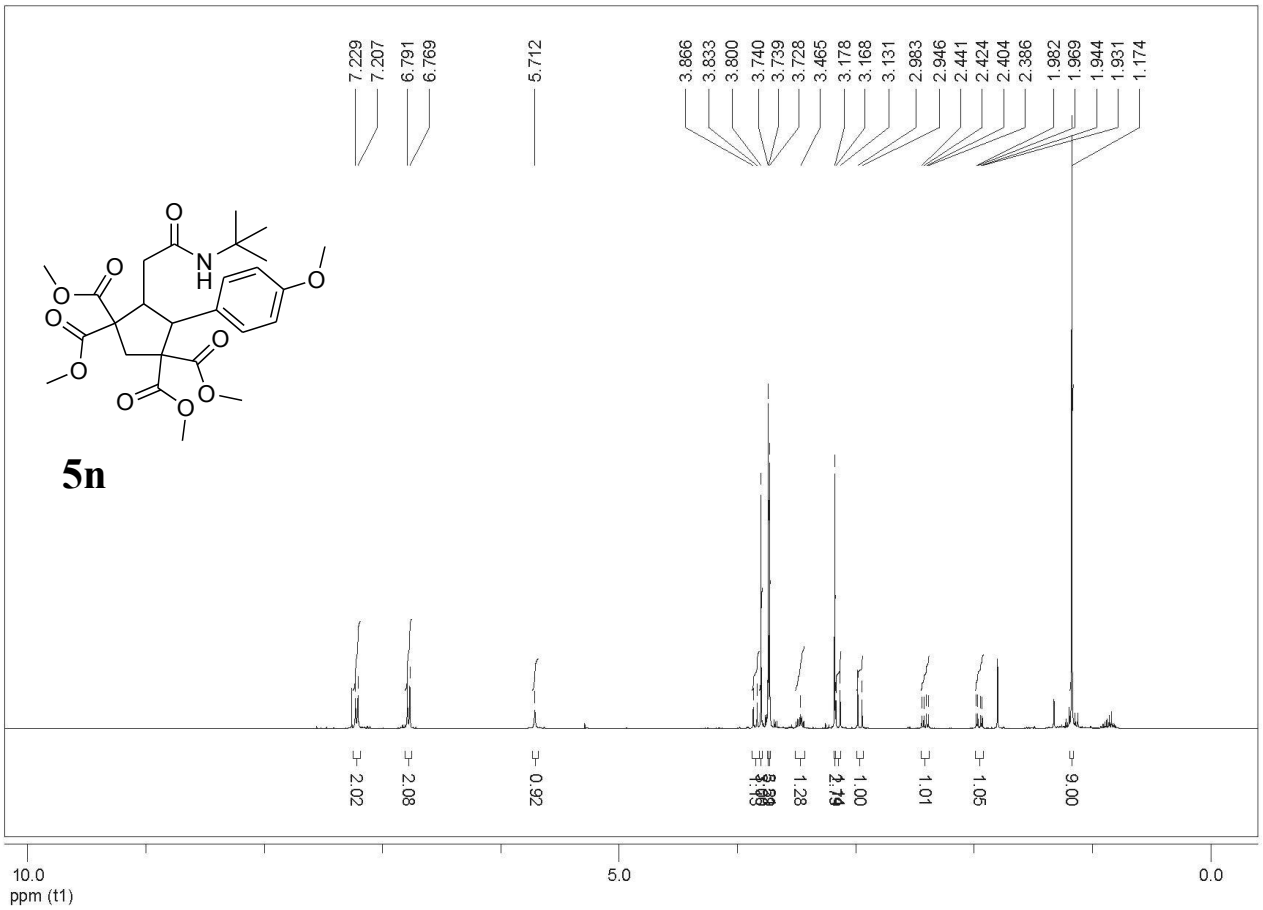


5c

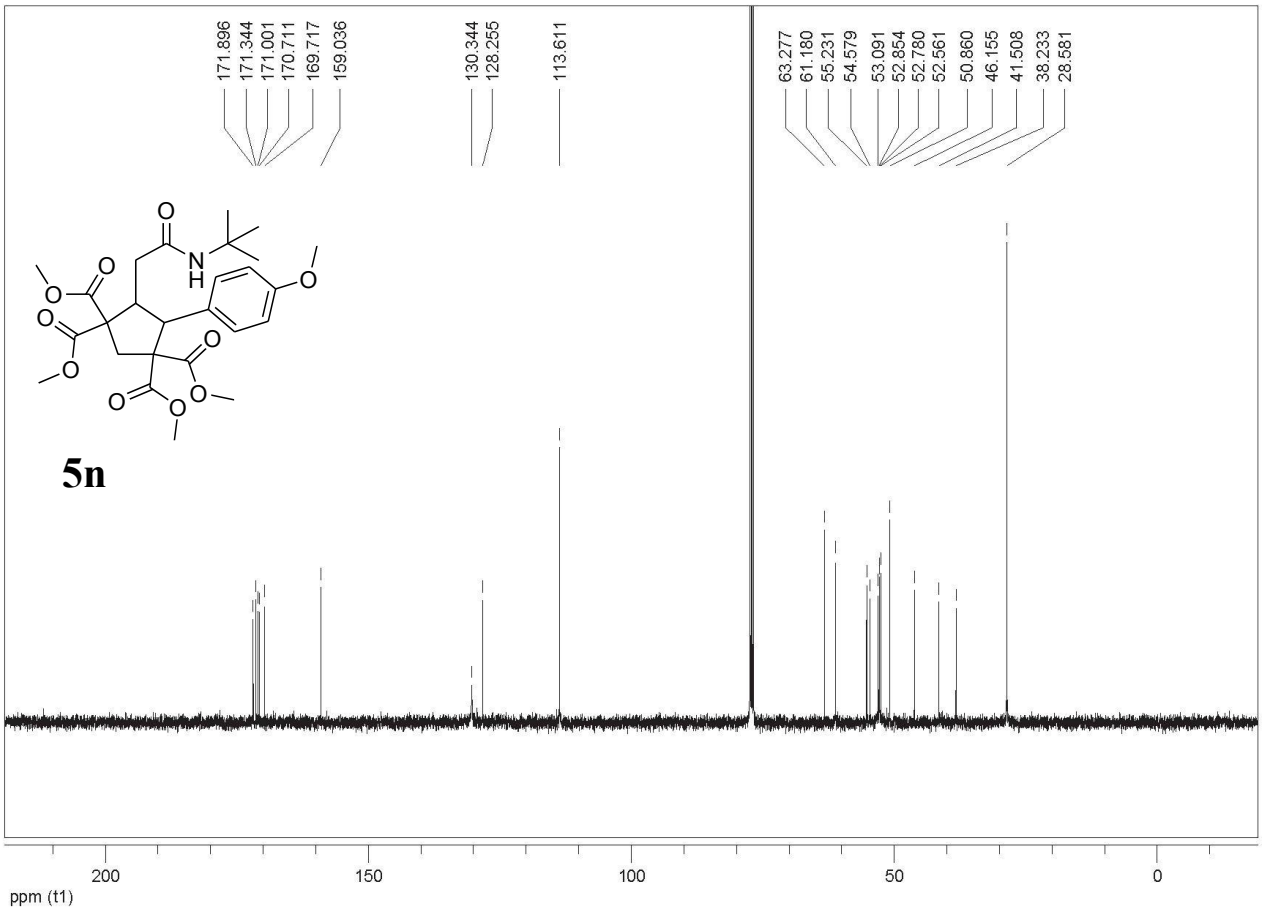



7.274
7.096
7.074
6.819
6.797
5.033
4.998
4.943
4.908
3.835
3.803
3.769
3.730
3.701
3.534
3.498
3.195
3.158
3.141
3.119
3.105
3.089
3.072
2.897
2.881
2.851
2.835
2.567
2.521
Cl
O
N
H
O
O
O
O
O
O
O
O
O
O
O
5g
3.93
1.91
2.00
1.98
1.08
0.97
1.33
10.0
5.0
0.0
ppm (t1)

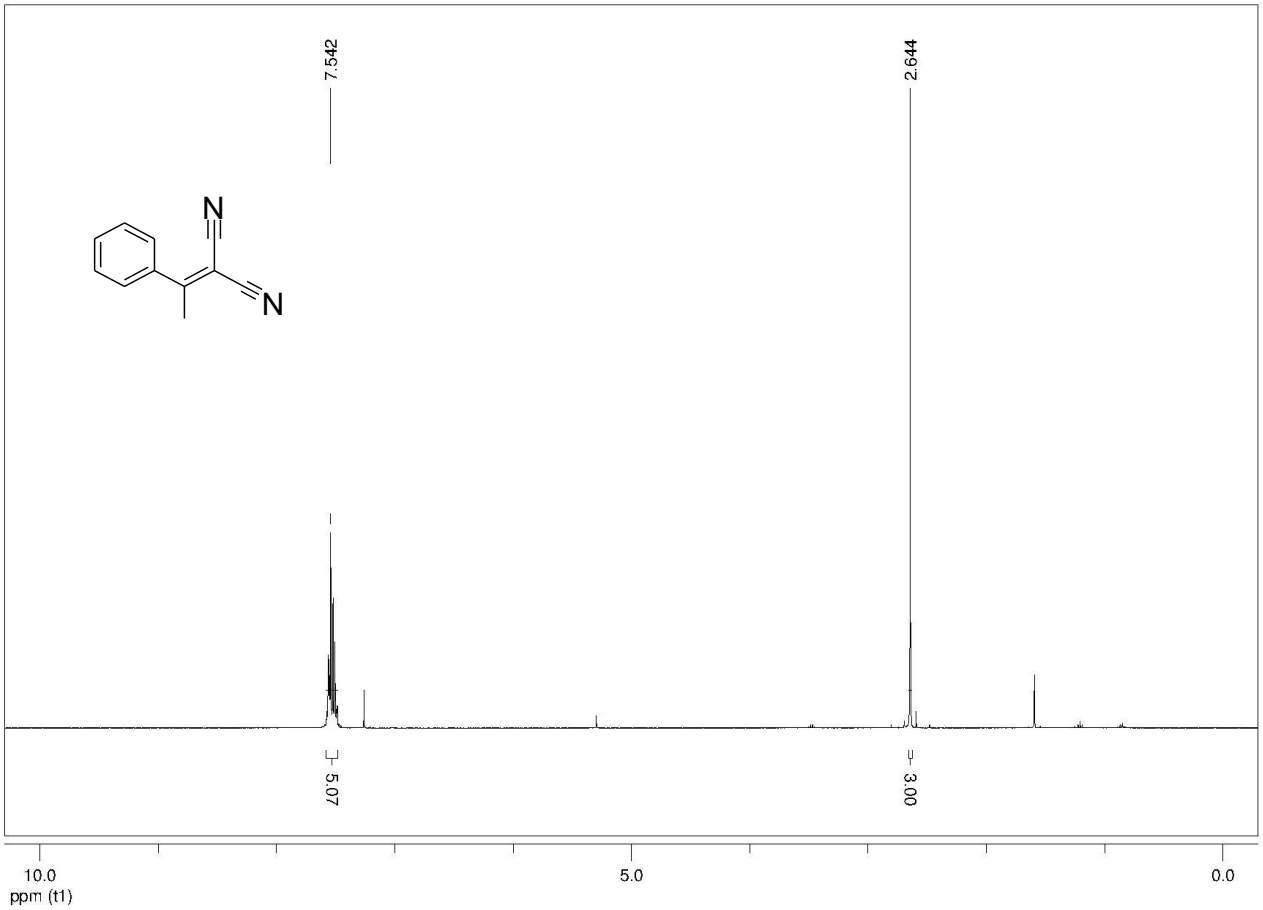

171.650
170.039
169.903
169.428
159.350
135.118
133.303
130.027
129.866
128.522
127.396
113.933
77.396
77.281
77.078
76.761
62.980
58.492
55.312
53.553
53.502
53.268
52.626
43.174
42.261
40.802
31.147
Cl
O
N
H
O
O
O
O
O
O
O
O
O
O
O
5g
200
150
100
50
0
ppm (t1)

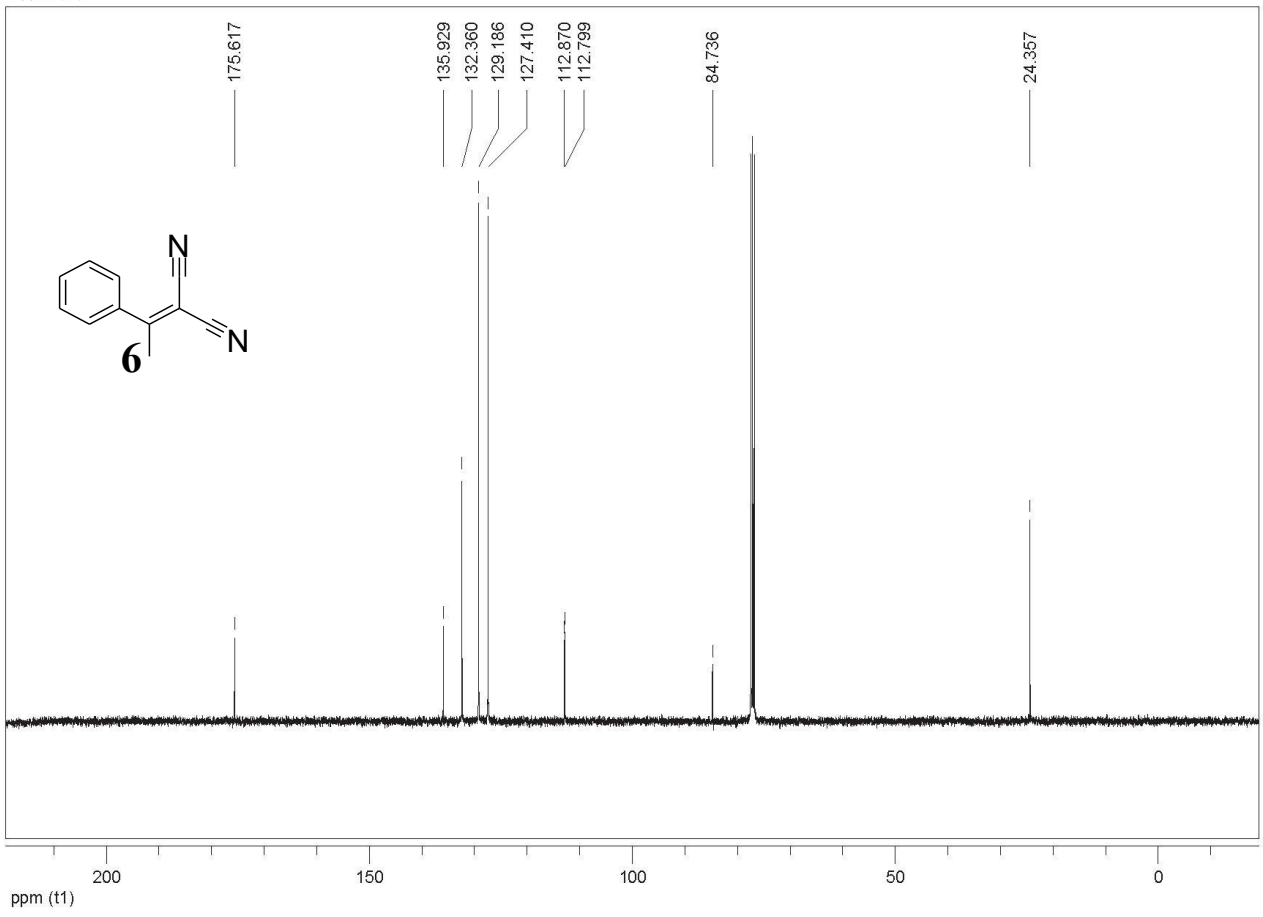

7.229
7.207
6.791
6.769
5.712
3.866
3.833
3.800
3.740
3.739
3.728
3.465
3.178
3.168
3.131
2.983
2.946
2.441
2.424
2.404
2.386
1.982
1.969
1.944
1.931
1.174
2.02
2.08
0.92
1.28
1.00
1.01
1.05
9.00
10.0
5.0
0.0
ppm (t1)

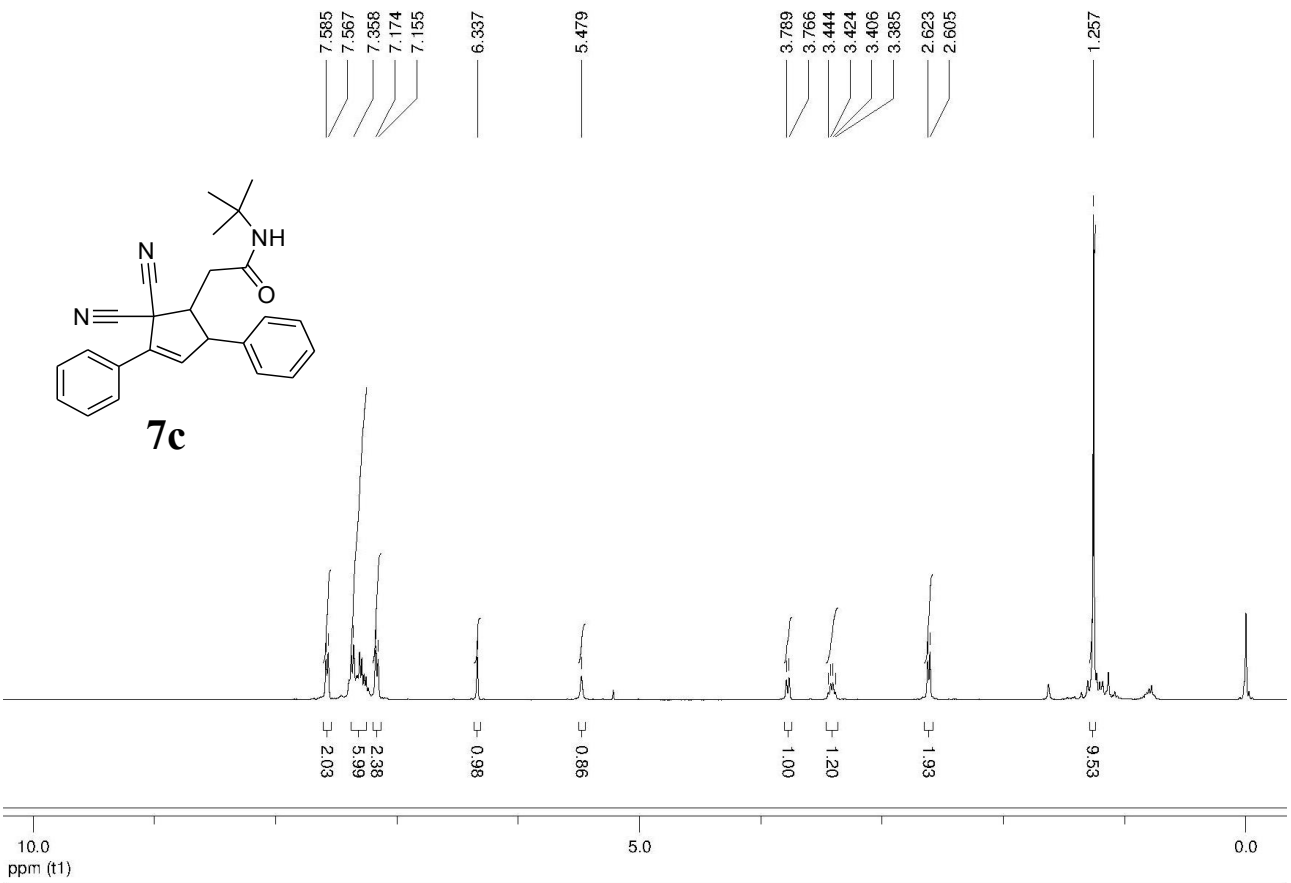
5n

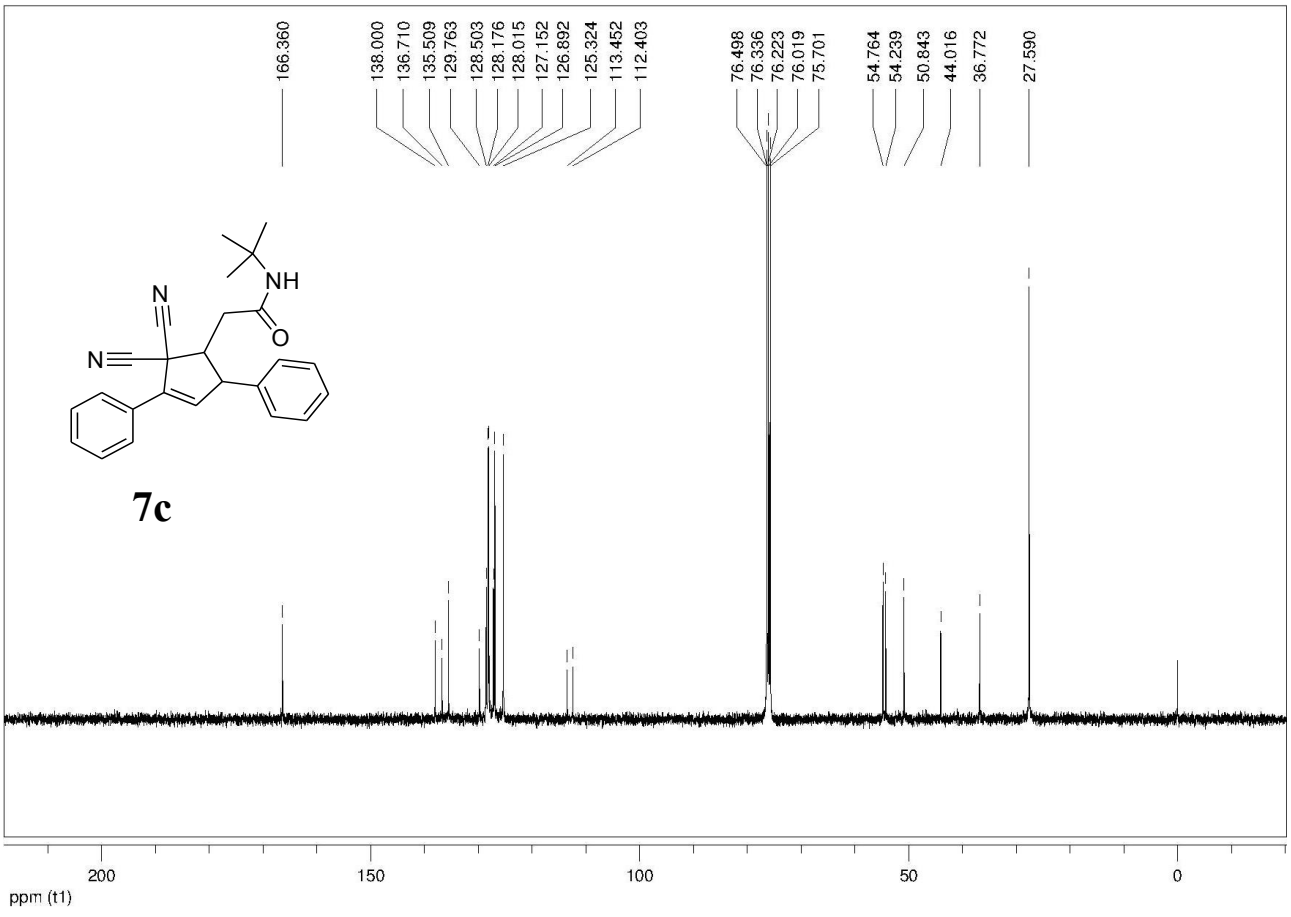

171.896
171.344
171.001
170.711
169.717
159.036
130.344
128.255
113.611
63.277
61.180
55.231
54.579
53.091
52.854
52.780
52.561
50.860
46.155
41.508
38.233
28.581
200
150
100
50
0
ppm (t1)

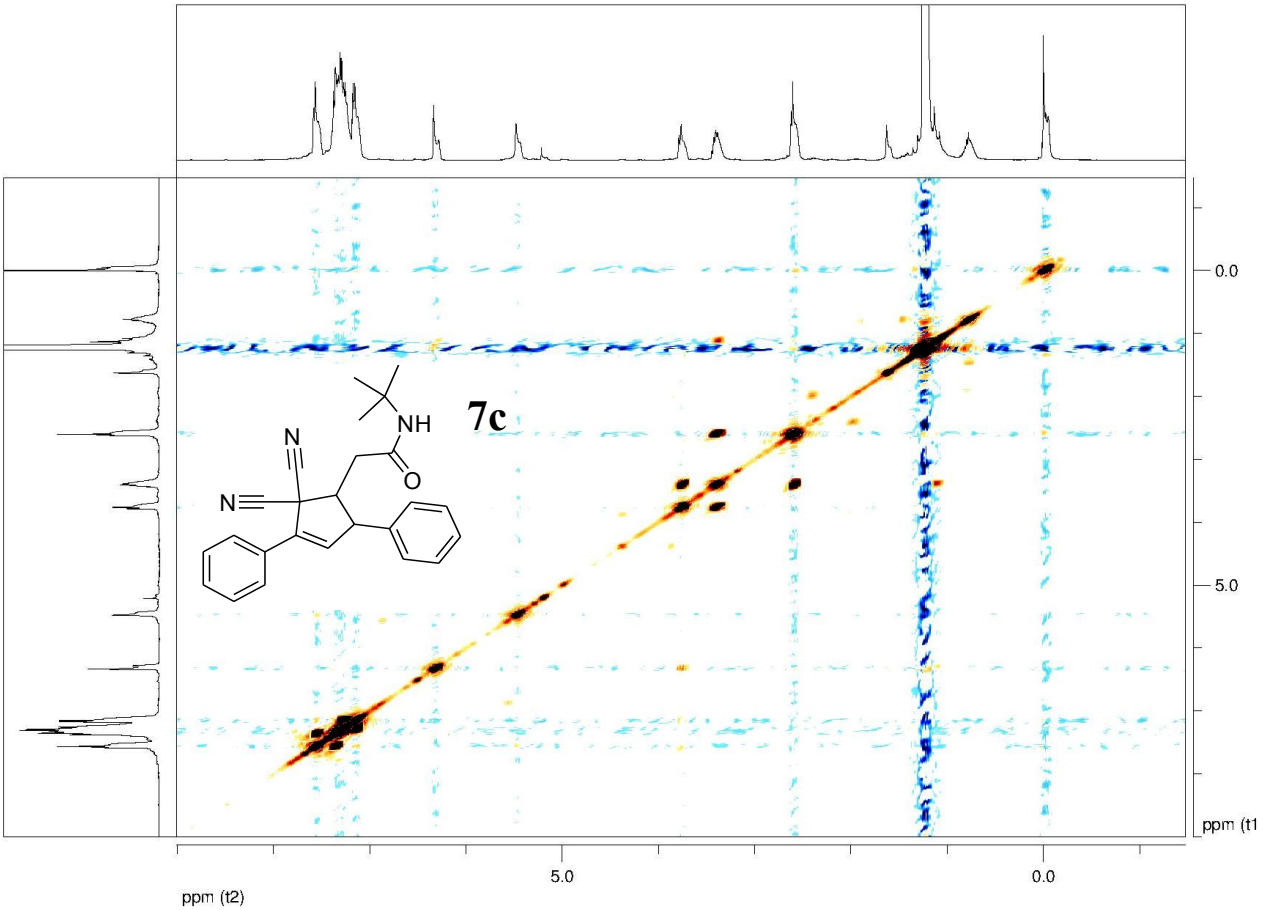
5n

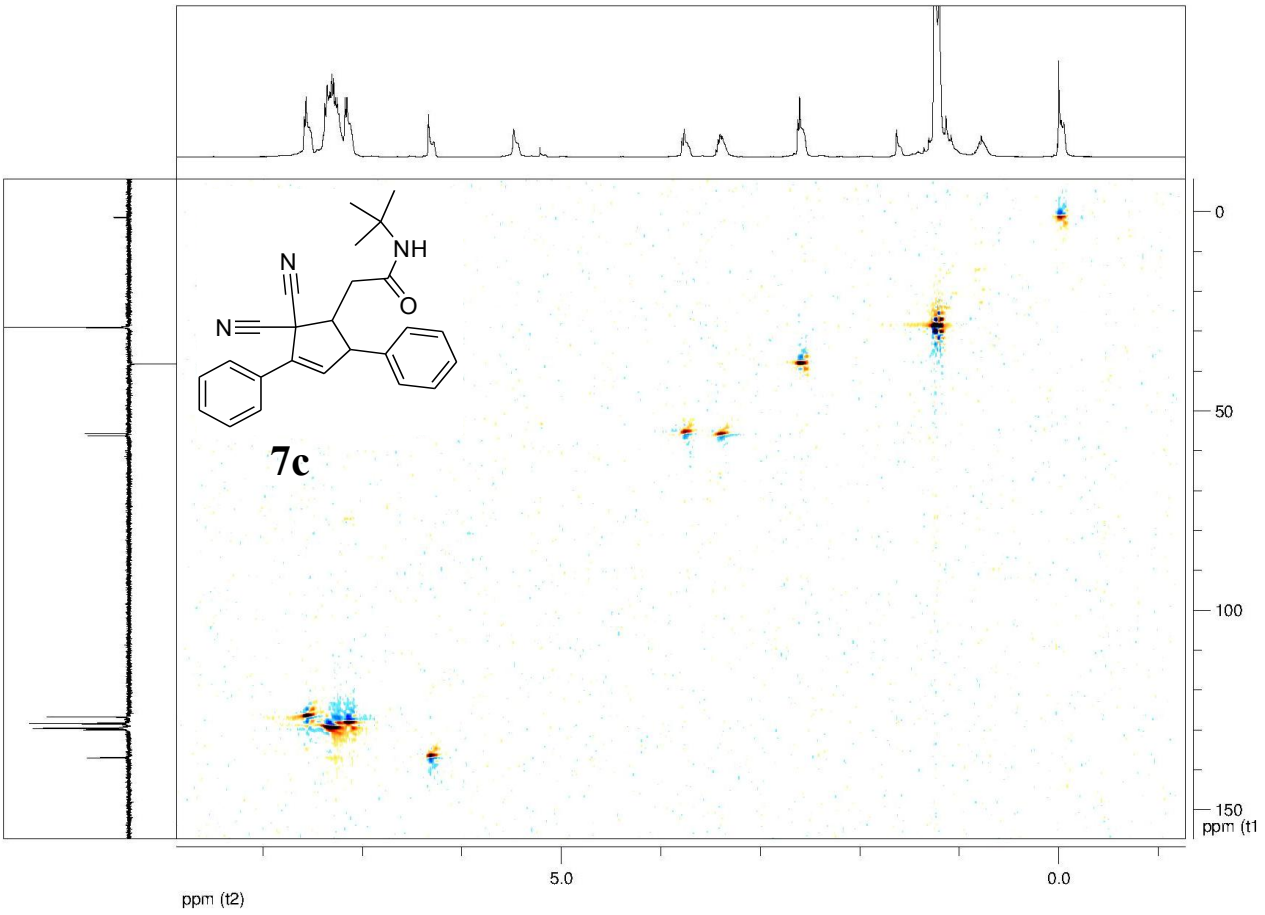

7.542
2.644
N
N
5.07
3.00
10.0
5.0
0.0
ppm (t1)

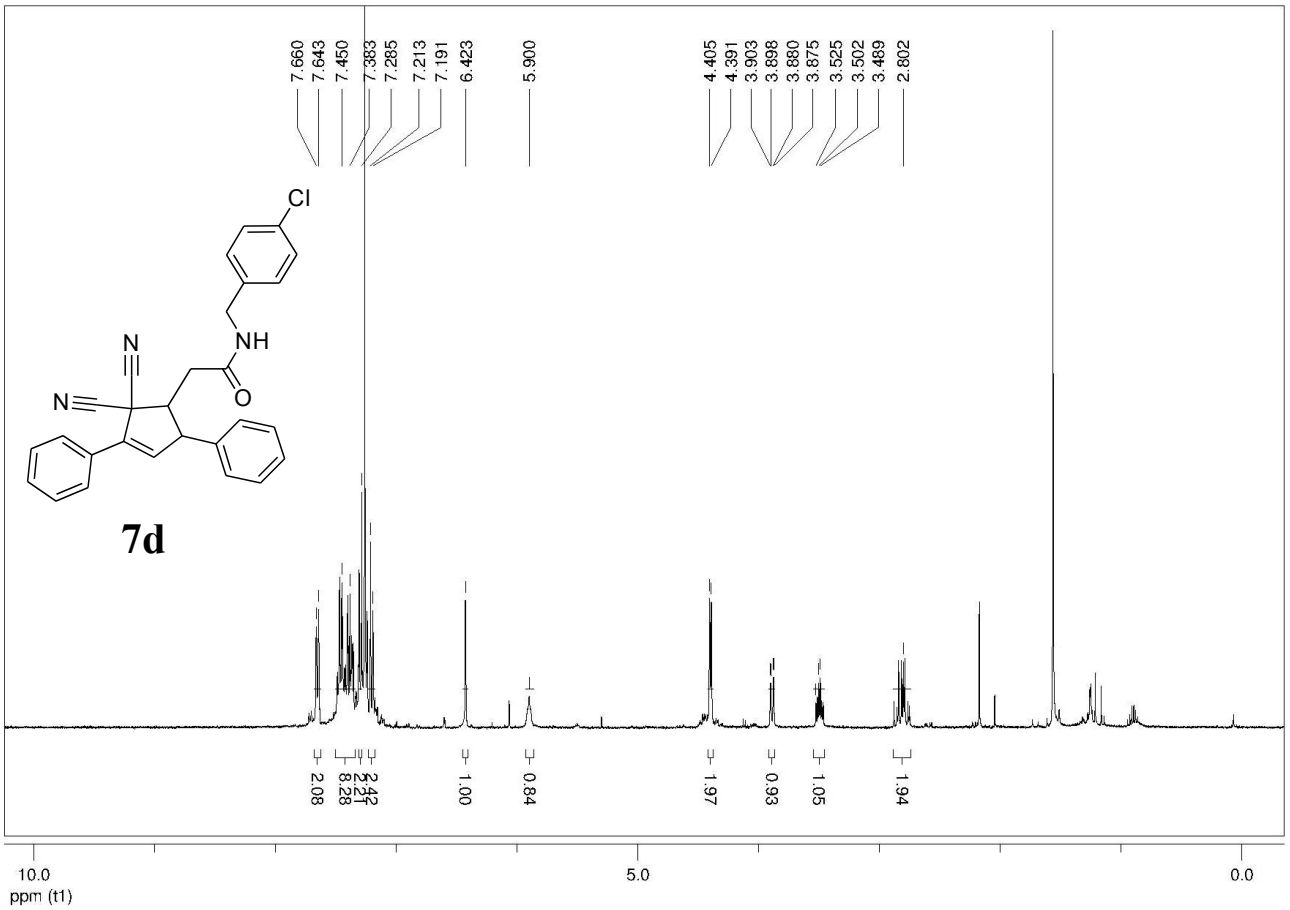

175.617
135.929
132.360
129.186
127.410
112.870
112.799
84.736
24.357
N
N
6
200
150
100
50
0
ppm (t1)


7.585
7.567
7.358
7.174
7.155
6.337
5.479
3.789
3.766
3.444
3.424
3.406
3.385
2.623
2.605
1.257
NH
O
N
N
7c
2.03
5.99
2.38
0.98
0.86
1.00
1.20
1.93
9.53
10.0
5.0
0.0
ppm (t1)

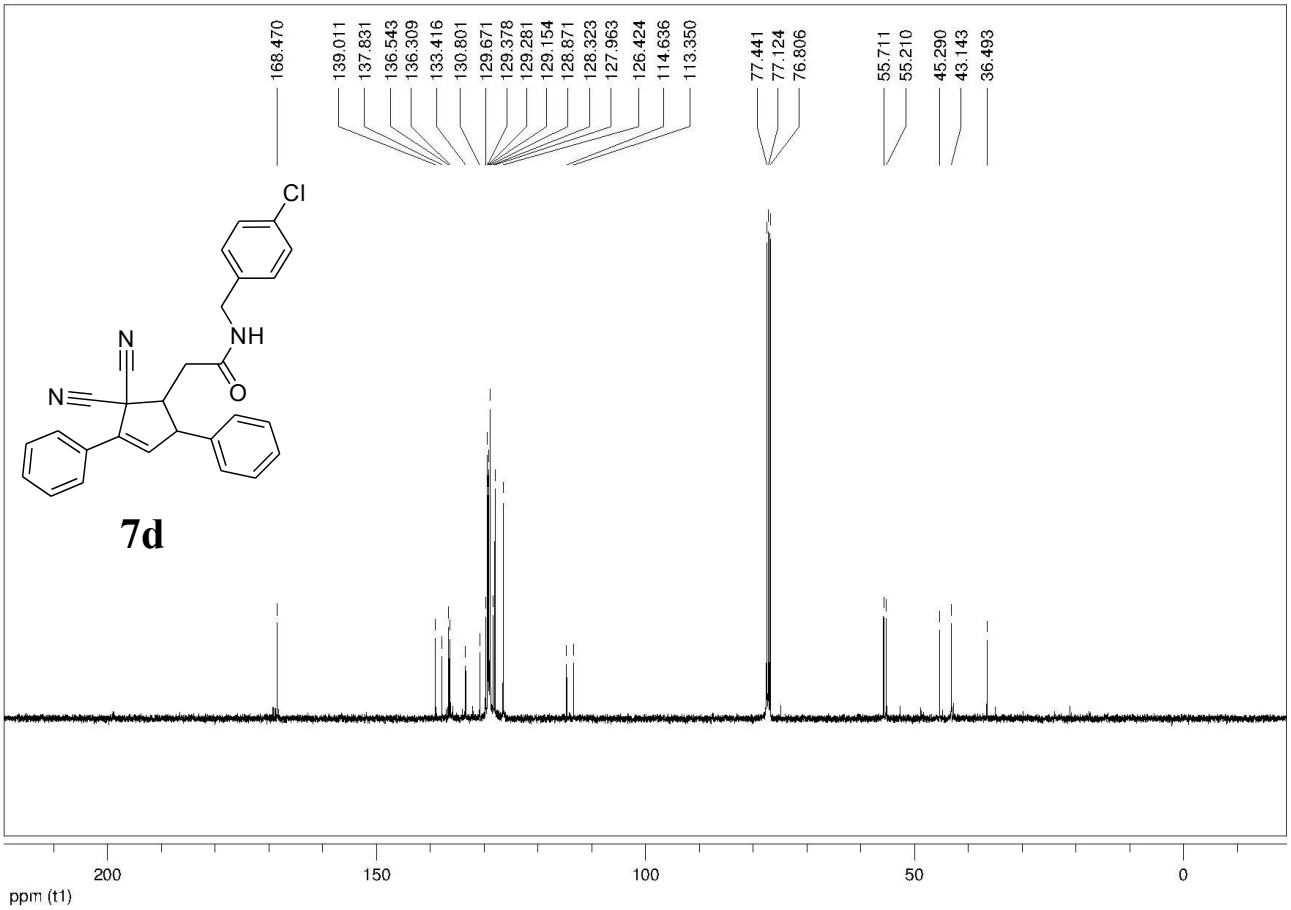

166.360
138.000
136.710
135.509
129.763
128.503
128.176
128.015
127.152
126.892
125.324
113.452
112.403
76.498
76.336
76.223
76.019
75.701
54.764
54.239
50.843
44.016
36.772
27.590
NH
O
N
N
7c
200
150
100
50
0
ppm (t1)


7c
NH
O
N
N
0.0
5.0
ppm (t1
5.0
0.0
ppm (t2)

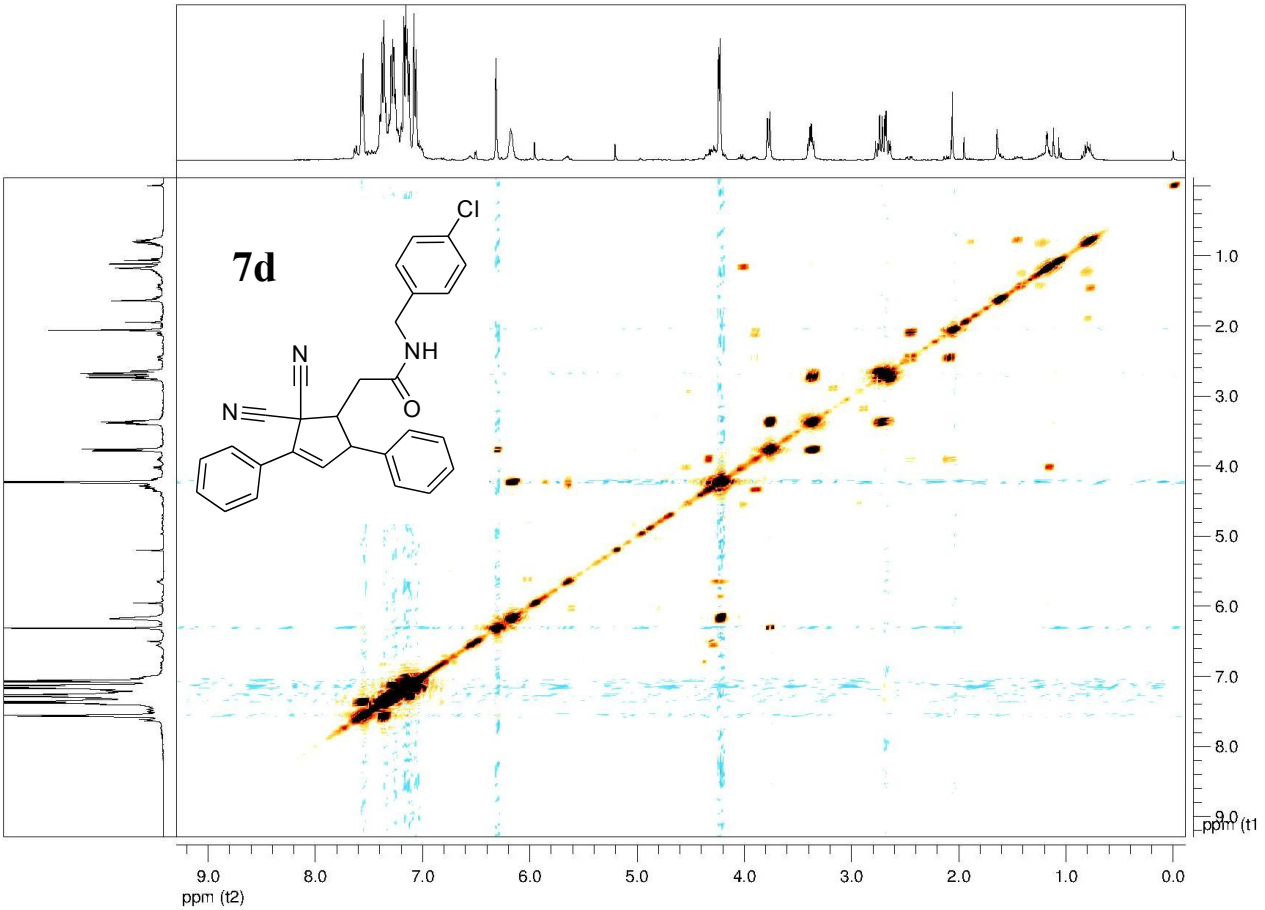

7c
NH
O
N
N
0
50
100
150
ppm (t1
5.0
0.0
ppm (t2)

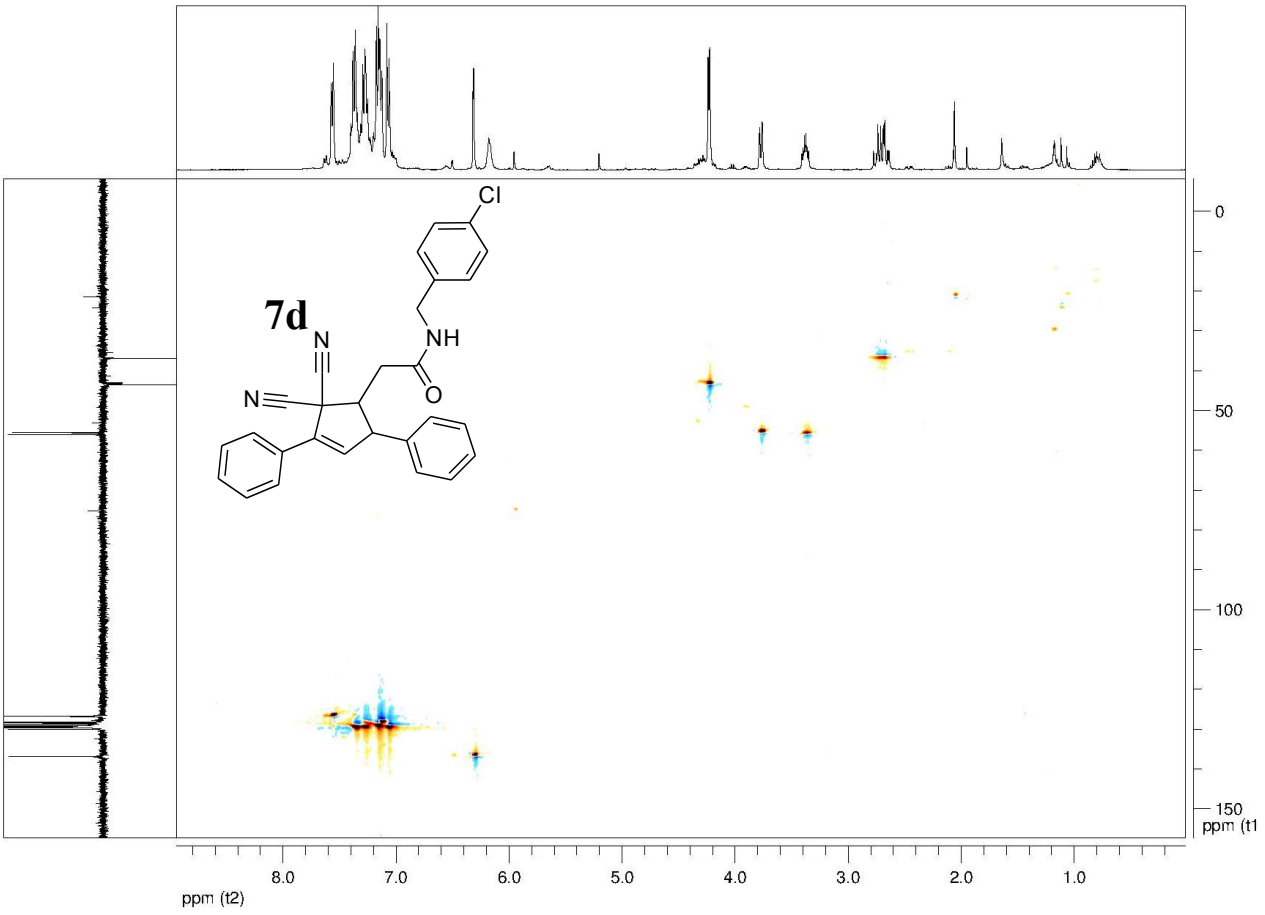

7.660
7.643
7.450
7.383
7.285
7.213
7.191
6.423
5.900
4.405
4.391
3.903
3.898
3.880
3.875
3.525
3.502
3.489
2.802
Cl
NH
N
N
O
2.08
2.28
2.28
2.21
1.00
0.84
1.97
0.93
1.05
1.94
10.0
5.0
0.0
ppm (t1)

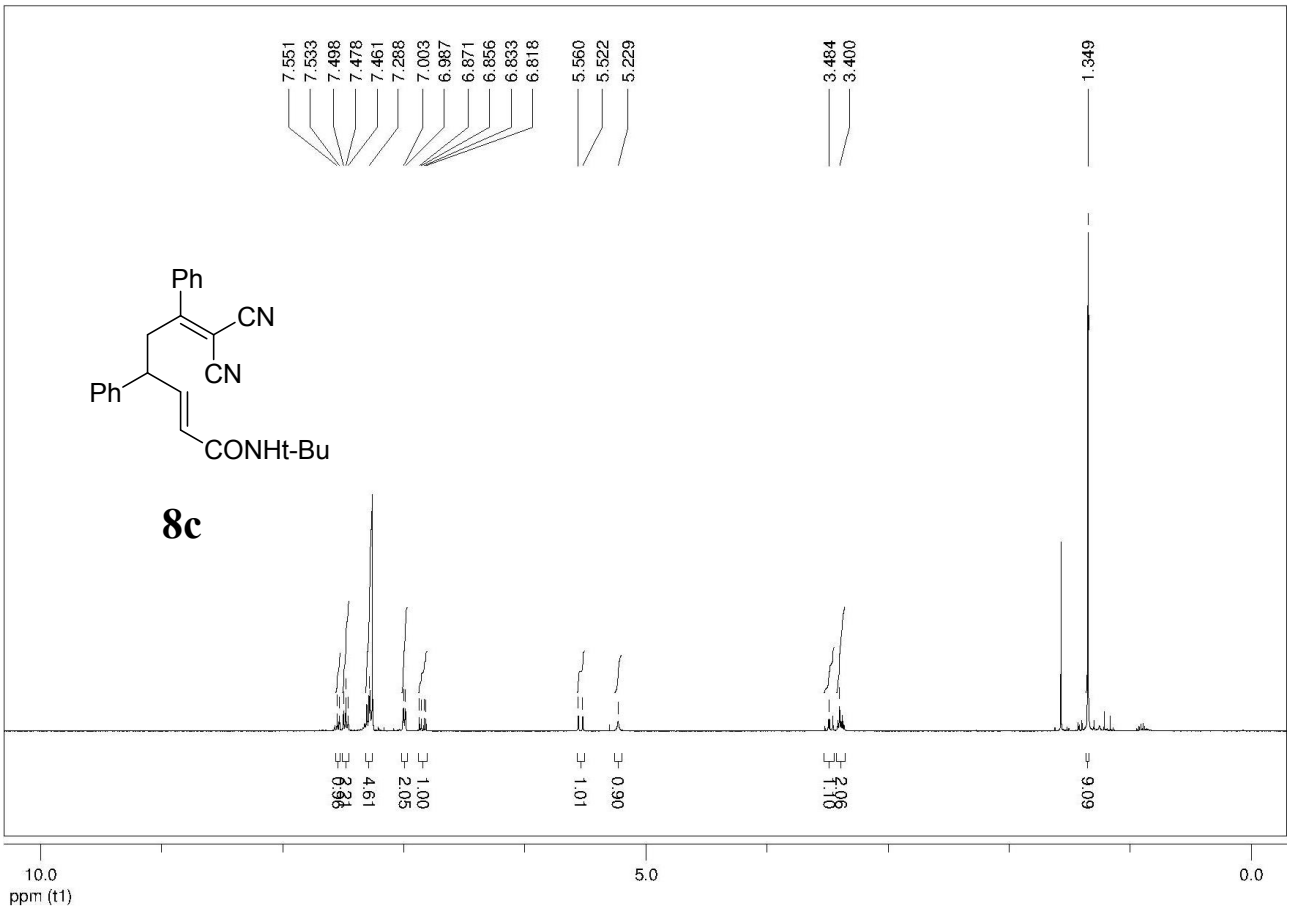
7d

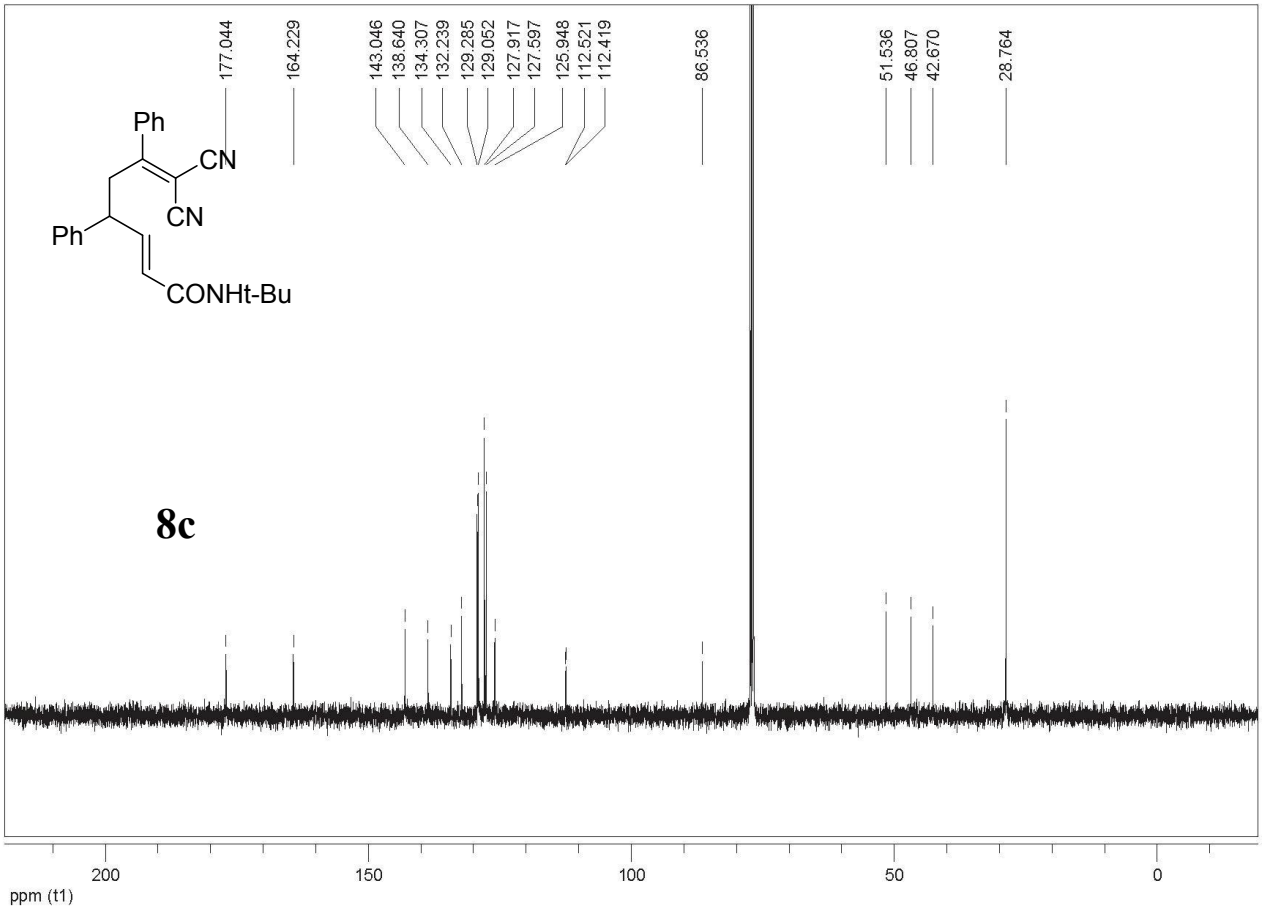

168.470
139.011
137.831
136.543
136.309
133.416
130.801
129.671
129.378
129.281
129.154
128.871
128.323
127.963
126.424
114.636
113.350
77.441
77.124
76.806
55.711
55.210
45.290
43.143
36.493
Cl
NH
N
N
O
200
150
100
50
0
ppm (t1)

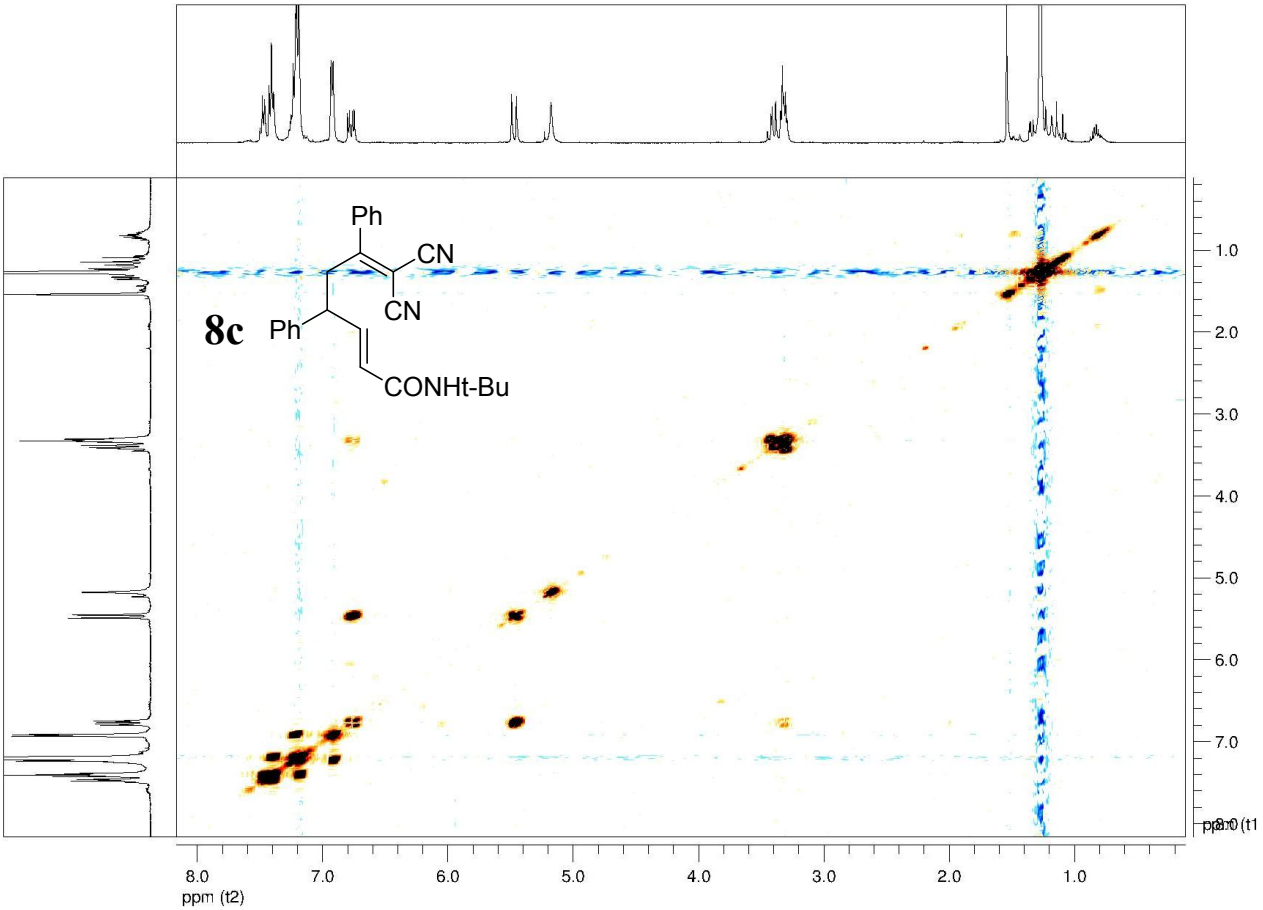
7d

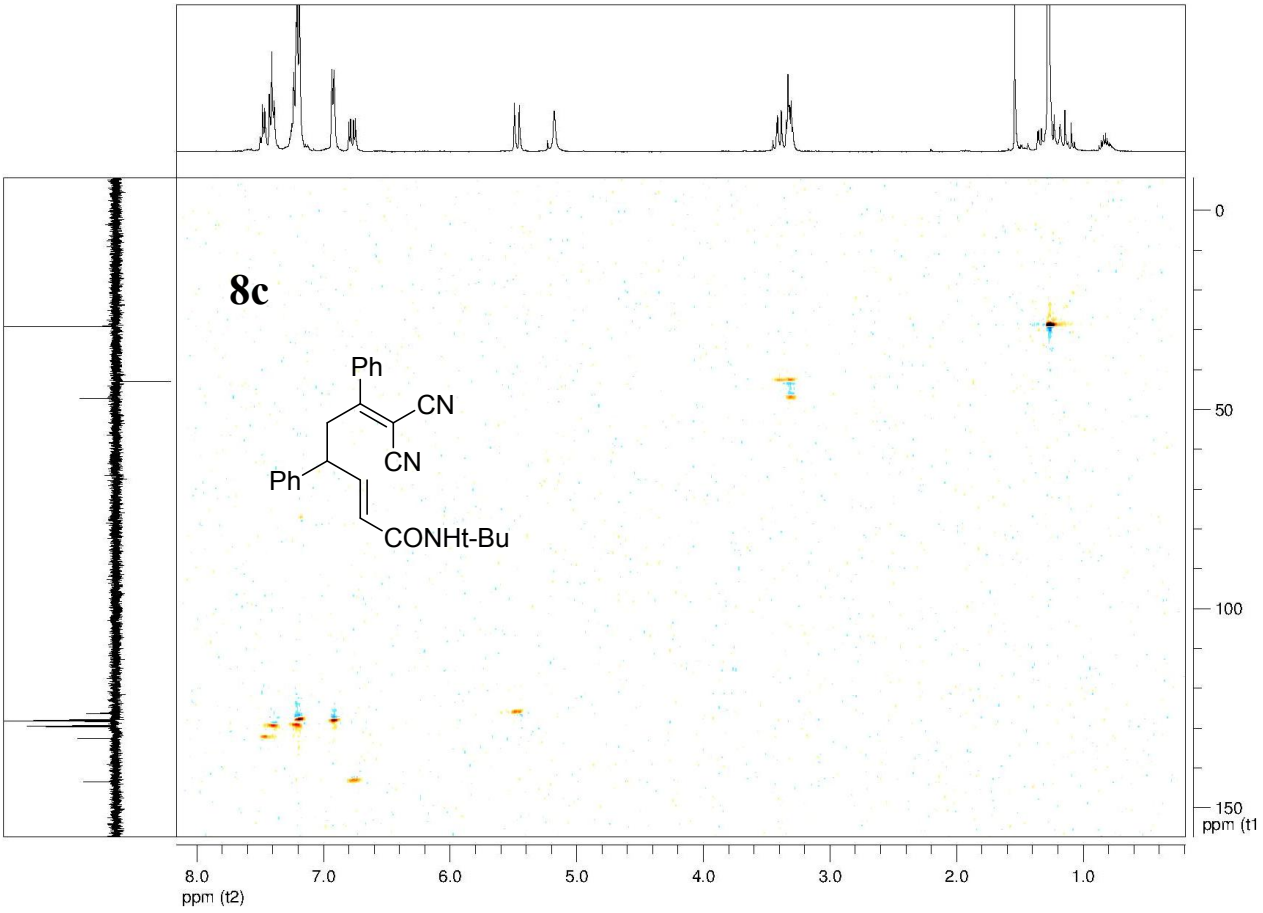

7d
Cl
NH
N
N
O
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
ppm (t1
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t2)


7d
Cl
NH
N
N
O
0
50
100
150
ppm (t1
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t2)


7.551
7.533
7.498
7.478
7.461
7.288
7.003
6.987
6.871
6.856
6.833
6.818
5.560
5.522
5.229
3.484
3.400
1.349
Ph
CN
CN
Ph
CONHt-Bu
8c
0.96
0.98
4.61
2.05
1.00
1.01
0.90
2.10
2.06
9.09
10.0
5.0
0.0
ppm (t1)


177.044
164.229
143.046
138.640
134.307
132.239
129.285
129.052
127.917
127.597
125.948
112.521
112.419
86.536
51.536
46.807
42.670
28.764
Ph
CN
CN
Ph
CONHt-Bu
8c
200
150
100
50
0
ppm (t1)


8c
Ph
CN
CN
Ph
CONHt-Bu
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
ppm (t1
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t2)


8c
Ph
CN
CN
Ph
CONHt-Bu
0
50
100
150
ppm (t1
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t2)