Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Benzoylureas as Removable Cis Amide Inducers: Synthesis of Cyclic Amides via Ring Closing Metathesis (RCM).

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Experimental procedures

General Procedure

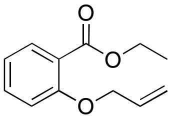
All non-aqueous reactions were carried out in oven-dried glassware under an atmosphere of nitrogen, unless otherwise specified. DCM, THF, DMF and diethyl ether were dried on an M-Braun Solvent Purification system. Anhydrous acetonitrile (99.8%) was purchased from Sigma-Aldrich and used as supplied. Triethylamine was distilled over calcium hydride and stored over molecular sieves. Petroleum ether describes a mixture of hexanes in the bp range 40-60°C.

Analytical thin-layer chromatography was performed on Merck silica gel $60F_{254}$ aluminium-backed plates and were visualised by fluorescence quenching under UV light and / or a permanganate stain consisting of $KMNO_4$ (3.0 g), K_2CO_3 (20 g) and 5% w/v aqueous NaOH (5 mL) in H_2O (300 mL). Flash column chromatography was performed with silica gel 60 (particle size 0.040-0.063 mm).

All NMR spectra were recorded were on a Bruker Advance DRX 300 with solvents indicated (^1H NMR at 300 MHz; ^{13}C at 75 MHz). Chemical shifts are reported in ppm on the δ scale, referenced to the appropriate solvent peak. Infrared spectra were recorded on a Bruker Tensor27 FTIR spectrometer and absorptions are given in wavenumbers (cm^{-1}). Oils were analysed using sodium chloride plates. Solids were analysed using a diffuse resistance accessory. Mass spectrometry was performed on a Finigan LCQ Advantage MAX. High resolution mass spectra were measured at the Australian National University on a Bruker Apex 3 HR FTICR ESI mass spectrometer. Elemental analyses were carried out for carbon, nitrogen, hydrogen and sulfur and hydrogen at the university of Otago microanalytical laboratory.

General procedure A: Phenol alkylation,

Ethyl-2-(allyloxy) benzoate (6): Ethyl salicylate **5** (2.0 g, 12 mmol) was dissolved in anhydrous DMF (20 mL) and treated with Cs_2CO_3 (19.6 g, 60 mmol). Allyl bromide (1.53 mL, 18 mmol) was added and the mixture was stirred at 60°C for 4 h. The reaction was quenched with 1M HCl and extracted with EtOAc (3 x 20 mL). The combined organics were washed with H_2O (15 mL), brine (15 mL), dried over $MgSO_4$, filtered and concentrated. Flash column chromatography, eluting with EtOAc/pet. ether (0-10 %) afforded ethyl-2-(allyloxy) benzoate **6** as a clear oil (1.56 g, 88 %)



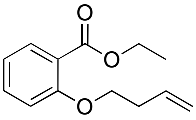
^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.77 (dd, J = 1.97, 7.51 Hz, 1H), 7.39 (ddd, J = 9.27, 7.41, 1.86 Hz, 1H), 6.98-6.91 (m, 2H), 6.04 (dddd, J = 20.3, 9.75, 4.88, 4.86 Hz, 1H), 5.49 (dddd, J = 18.99, 1.77, 1.77, 1.71 Hz, 1H, CH), 5.26 (dddd, J = 12.06, 1.56, 1.56, 1.56 Hz, 1H, CH), 4.58 (ddd, J = 4.86, 1.68, 1.65 Hz, 2H, CH_2), (q, J = 7.28 Hz, 2H, CH_2), 1.35 (t, J = 7.13 Hz, 3H, CH_3).

^{13}C NMR ($CDCl_3$, 75 MHz) δ 166.3, 157.9, 133.1, 132.8, 131.4, 121.1, 120.4, 117.2, 113.7, 69.4, 60.7, 14.3.

IR (ATR, cm^{-1}) 3706, 2972, 1723, 1600, 1244.

HRMS (ES^+) Calculated for $C_{12}H_{14}O_3Na$ (M + Na): 229.8841; found 229.8841

Ethyl-2-(but-3-enyloxy)benzoate (7)



Ethyl salicylate **5** (1.0 g, 6 mmol) and 4-bromobutene (0.76 mL, 6.2 mmol) were treated as described in general procedure A. Purification via flash column chromatography, eluting with EtOAc/pet. ether, afforded **7** as a clear oil (0.403 g, 30 %).

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.79 (d, J = 1.68 Hz, 1H), 7.76-7.40 (m, 1H), 7,28-6.90 (m, 2H), 6.04-5.90 (m, 1H), 5.23-5.10 (m, 2H), 4.37 Hz, (q, J = 7.11 Hz, 2H, CH_2), 4.09 (t, J = 6.72 Hz, 2H, CH_2), 2.64-2.57, (m, 2H), 1.39 (t, J = 7.14 Hz, 3H, CH_3).

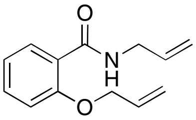
^{13}C NMR ($CDCl_3$, 75 MHz**)** δ 166.6, 158.2, 134.3, 133.1, 131.5, 121.2, 120.3, 117.0, 113.4, 68.4, 60.7, 33.7, 14.3.

IR (ATR, cm^{-1}) 3706, 2936, 1724, 1600, 1300, 1243.

HRMS (ES^+) Calculated for $C_{13}H_{17}O_3$ (M+H): 221.1178; found 221.1177

General procedure B: Synthesis of amides

***N*-allyl-2- (prop-2-enyloxy) benzamide (8)**: The ester **6** (3.0 g, 15 mmol) was dissolved in MeOH (10 mL). An aqueous solution of LiOH (11 mL, 8M) was added and the mixture allowed to stir for 12 h. The reaction was quenched with 10% citric acid (15 mL) and extracted with EtOAc (3 x 10 mL). The combined organic layers were washed with brine (10 mL), H_2O (10 mL), dried over $MgSO_4$ and concentrated. The crude product (2.64 g, 15 mmol), along with EDCI (30 mmol) and HOBT (3.21 g, 30 mmol) was dissolved in anhydrous DCM (40 mL). The solution was cooled to 0°C and treated with allyl amine (1.40 mL, 23 mmol). After 2 h, the reaction was quenched with 1M HCl (15 mL), washed with brine (15 mL), dried over $MgSO_4$ and concentrated. The crude oil was purified *via* Flash column chromatography, eluting with EtOAc/pet. ether (0–10 %), yielding **8** as a slightly yellow oil (2.15 g, 66 %).



^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.23-8.20 (m,1H), 8.02 (br.s, 1H, NH), 7.44–7.38 (m, 1H), 7.10–7.34 (m, 1H), 6.97–6.94 (m, 1H), 6.10–5.90 (m, 2H), 5.23–4.96 (m, 4H), 4.45 (ddd, J = 5.43, 1.41, 1.41 Hz, 2H, CH_2), 3.92 (dddd, J = 5.52, 5.52, 1.65, 1.65 Hz, 2H, CH_2).

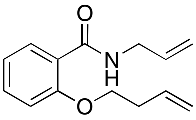
^{13}C NMR ($CDCl_3$, 75 MHz**)** δ 165.1, 165.6, 134.4, 132.7, 132.4, 132.0, 121.8, 121.6, 119.2, 115.9, 112.8, 69.9, 42.2.

IR (ATR, cm^{-1}) 3706, 2970, 2865, 1639, 1599, 1526, 1226.

MS (ES$^+$) *m/z* 218 (M + H)

HRMS (ES$^+$) Calculated for $C_{13}H_{16}NO_2$ (M+H): 218.1181; found 218.1173.

***N*-allyl-2-(but-enyloxy)benzamide (9)**



The ester **7** (0.183 g, 0.82 mmol) was converted to the amide **9** as described in general procedure B. The crude oil was purified via flash column chromatography, eluting with EtOAc/pet. ether (0-10 %), yielding a slightly yellow solid (0.134 g, 73 %).

^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.2 (dd, *J* = 8, 2 Hz, 1H), 8.1 (br.s, 1H), 7.4 (ddd, *J* = 9, 7, 2 Hz, 1H), 7.0 (ddd, *J* = 8, 7, 1 Hz, 1H), 6.9 (dd, *J* = 8, 1 Hz, 1H), 5.92-5.83 (m, 2H), 5.24-5.10 (m, 4H), 4.14 (t, *J* = 6.2 Hz, 2H), 4.03 (m, 2H), 2.61-2.54 (m, 2H).

^{13}C NMR ($CDCl_3$, 75 MHz) δ 164.9, 156.8, 134.6, 134.4, 132.6, 132.2, 121.3, 121.1, 117.8, 115.7, 112.0, 67.7, 41.9, 33.6.

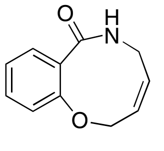
IR (ATR, cm^{-1}) 3396, 2942, 1653, 1530, 1297, 1233, 732.

MS (ES^+) *m/z* 232 (M + H)

HRMS (ES^+) Calculated for $C_{14}H_{19}NO_2$ (M+H): 232.1338; found 232.1338.

General procedure C: Amide deprotection,

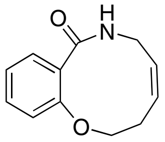
(Z)-5,6-dihydrobenzo[b][1,5]oxazecin-8-one (10): Benzoylurea **29** (0.314 g, 0.72 mmol) was dissolved in dry Et_2O (5 mL). In a separate flask, HCl gas was generated from $NaCl/H_2SO_4$ and bubbled through the ether solution. After 20 mins the free amine precipitated from the solution as the hydrochloride salt. The salt was dissolved in dry DMF (240 μL), neutralised with DIPEA (342 μL, 1.2 mmol) and stirred at 60°C for 12 h. The mixture was quenched with 1M HCl (5 mL) and extracted with EtOAc (3 x 5 mL). The combined organics were washed with brine, H_2O (5 mL), dried over $MgSO_4$, filtered and concentrated. The crude product was purified via flash column chromatography, eluting with MeOH/DCM (5 %), affording the lactam **10** as a white solid (0.078 g, 58 %).



^{1}H NMR (MeOD, 300 MHz) δ 7.50 (ddd, *J* = 8.52, 7.50, 1.5 Hz, 1H), 7.40 (dd, *J* = 7.62, 1.62 Hz), 7.19-7.09 (m, 1H), 6.12-6.05 (m, 1H), 5.99-5.90 (m, 1H), 4.78 (d, *J* = 5.88 Hz, 2H), 3.48 (d, *J* = 7.83 Hz, 2H).

^{13}C NMR (MeOD, 75 MHz) δ 170.4, 156.5, 137.5, 131.3, 129.9, 127.8, 125.7, 122.4, 118.1, 69.3, 40.95.
MS (ES$^+$): *m/z* 190.2 (M+H)
HRMS (ES$^+$) Calculated for $C_{11}H_{12}NO_2$ (M + H): 190.0868; found 190.0873.

(Z)-2,3,6,7-tetrahydrobenzo[b][1,5]oxacin-8-one (11)

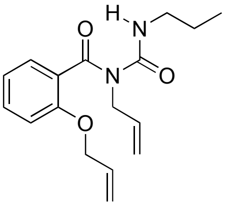


The benzoylurea **30** (0.104 g, 0.23 mmol) was treated according to general procedure C. Flash column chromatography, eluting with MeOH/DCM (5 %), afforded **11** as a white solid (0.029 g, 64 %).
^{1}NMR ($CDCl_3$, 300 MHz): δ mixture of 1:2 of 2 conformers 7.92 and 7.48 (d, *J* = 6 Hz, 1H), 7.24-7.29 (m, 1H), 7.24 (t, *J* = 1 Hz, 1H), 7.04-7.14 (m, 1H), 5.96-6.08 (m, 2H), 5.67-5.81 (m, 1H), 4.28 and 4.16 (m, 2H), 4.10 and 3.56 (m, 2H), 2.26 and 2.15 (m, 2H).
^{13}C NMR ($CDCl_3$, 75 MHz): δ 170.74, 167.75, 158.11, 153.42, 133.25, 131.23, 131.04, 130.79, 130.15, 129.84, 129.25, 128.89, 125.39, 124.16, 122.38, 113.83, 76.94, 66.03, 40.65, 38.10, 29.03, 26.17.
MS (ES$^+$): *m/z* (M+H) 204.0
HRMS (ES$^+$) *m/z* Calculated for $C_{12}H_{14}NO_2$ (M+1) 204.1025, found 204.1020.

General procedure D: Synthesis of Benzoylureas

***N*-allyl-2-(allyloxy)-*N*-(propylcarbamoyl)benzamide (12):** In an oven dried schlenk tube, the amide **8** (0.257 g, 1.2 mmol) in dry Et_2O (4.8 mL), was treated with Et_3N (362 µL, 2.6 mmol), followed by TMSOTf (476 µL, 1.9 mmol). After 45 mins, the solution was cooled to 0°C and phosgene (20 % in toluene) (10.2 mmol, 3.8 mL) was added and the mixture warmed to room temperature over 1 h. The solution was concentrated under vacuo and the thick residue resuspended in CH_3CN (mL/mmol). At 0°C, the amine, propylamine (129 µL, 1.6 mmol), along with DIPEA (1 mL, 5.7 mmol) was added and the mixture warmed to room temperature. After 15 mins, the reaction was quenched with

10 % citric acid and extracted with EtOAc. The combined extracts were washed with brine, H_2O, dried, filtered and concentrated. The crude residue was purified via flash column chromatography, eluting with EtOAc/Pet. Ether (0 – 30%), yielding **12** as a clear oil (0.423 g, 61 %).



^{1}H NMR ($CDCl_3$, 300 MHz) δ 9.21 (br. s, 1H, NH), 7.28 (ddd, J = 9.21, 7.53, 1.74 Hz, 1H, CH), 7.13 (dd, J = 7.5, 1.71 Hz, 1H, CH), 6.93-6.84 (m, 2H), 5.97-5.85 (m, 1H), 5.73-5.60 (m, 1H), 5.42 (dddd, J = 11.71, 1.65, 1.65, 1.56 Hz, 1H, CH), 5.17 (dddd, J = 12.0, 1.47, 1.41, 1.47 Hz, 1H, CH), 4.56 (dddd, J = 11.70, 1.32, 1.35, 1.29 Hz, 1H, CH), 4.77 (dddd, J =17.21, 1.50, 1.44, 1.44 Hz, 1H, CH), 4.51-4.49 (m, 2H, CH_2), 4.46 (br.s, 1H, CH), 3.91 (br.s, 1H, CH), 3.27 (q, J = 6.9 Hz, 2H, CH_2), 1.56 (pent, J = 7.26 Hz, 2H, CH_2), 0.91 (t, J = 7.41 Hz, 3H, CH_3).

^{13}C NMR ($CDCl_3$, 75 MHz) δ 172.1, 153.4, 152.5, 132.9, 131.5, 130.0, 126.2, 125.5, 119.8, 116.5, 115.2, 111.3, 67.9, 47.2, 41.2, 21.7, 10.4.

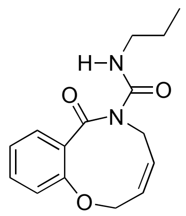
MS (ES^+) m/z 303 (M+H)

IR (ATR cm^{-1}) 3294, 2963, 1700, 1653, 1526, 1215.

HRMS (ES^+) Calculated for $C_{17}H_{22}N_2O_3Na$ (M + Na): 325.1528; found 325.1528

General procedure E: Synthesis of cyclic amides via RCM.

(*Z*)-7-Oxo-*N*-propyl-5,7-dihydrobenzo[*b*][1,5]oxazonine-6(2*H*)-carboxamide (13): An oven dried 2-neck flask was purged with N_2, evacuated and refilled with N_2. Anhydrous DCM (53 mL) was added and the solution degassed. Under an atmosphere of N_2, Grubbs 2nd generation catalyst (0.014 g, 0.016 mmol) was added, followed by **12** (0.050 g, 0.16 mmol). The mixture was heated to reflux (35°C) for 2 h, after which time, the solution was concentrated. The brown residue was purified via flash column chromatography, eluting with EtOAc/pet.ether (0-30%), affording **13** as a light brown solid (0.028 g, 64 %).



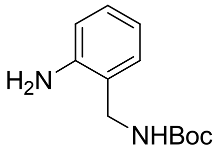
^{1}H NMR ($CDCl_3$, 300 MHz) δ 9.26 (br. s, 1H, NH), 7.51-7.26 (m, 2H), 7.15 (ddd, *J* = 8.46, 7.53, 1.02 Hz, 1H, CH), 7.02 (dd, *J* = 8.25, 0.6 Hz, 1H, CH), 6.28-6.18 (m, 1H), 6.18-5.30 (m, 1H), 4.82 (br. s, 2H, CH_2), 3.98 (br. s, 2H, CH_2), 3.31 (q, *J* = 7.0 Hz, 2H, CH_2), 1.62 (pent, *J* = 7.29 Hz, 2H, CH_2), 0.97 (t, *J* = 7.14 Hz, 3H, CH_3).

^{13}C NMR ($CDCl_3$, 75 MHz) δ 171.4, 155.6, 153.7, 131.6, 131.2, 129.3, 127.8, 127.6, 122.4, 117.1, 70.7, 45.3, 41.2, 21.7, 10.5.

MS (ES^+) m/z 275 (M+H).

HRMS (ES^+) Calculated for $C_{15}H_{19}N_2O_3$ (M + H): 275.1396; found 275.1396

tert-Butyl 2-aminobenzylcarbamate (19)



2-Aminobenzyl amine (3.0 g, 0.025 mol), along with DIPEA (13 mL, 0.026 mol) was dissolved in dry THF (50 mL). $(BOC)_2O$ (5.0 g, 0.026 mol) was added and the mixture was stirred at room temperature under a N_2 atmosphere. After 2 h, the reaction was poured onto 10% citric acid (25 mL) and extracted with EtOAc (3 x 15 mL). The combined organics were washed with brine (15 mL), dried over $MgSO_4$, filtered and concentrated, affording an off white solid (4.98 g, 90%).

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.10 (ddd, *J* = 15, 8, 2 Hz, 1H), 7.02 (dd, *J* = 7 Hz, 1H), 6.70-6.64 (m, 2H), 4.94 (br.s, 1H), 4.24-4.22 (m, 4H), 1.46 (s, 9H).

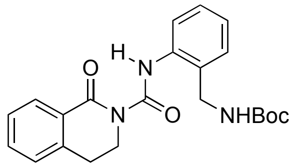
^{13}C NMR ($CDCl_3$, 75 MHz) δ 156.4, 145.5, 130.2, 129.0, 122.5, 117.8, 115.7, 79.7, 42.1, 28.4.

IR (ATR, cm^{-1}) 3357, 2975, 1668, 1579, 1163.

MS (ES^+) *m/z* 224 (M + H)

HRMS (ES^+) Calculated for $C_{12}H_{19}N_2O_2$ (M+H): 223.1447; found 223.1447.

tert-Butyl 2-(1-oxo-1,2,3,4-tetrahydroisoquinoline-2-carboxamido)benzylcarbamate (20)



The amide **18** (0.120 g, 0.8 mmol) and the aniline **19** (0.231 g, 1.0 mmol) were treated according to general procedure D. Flash column chromatography, eluting with EtOAc/pet. ether (20 %), afforded the benzoylurea **20** as a white solid (0.126 g, 40%).

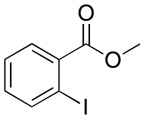
^{1}H NMR ($CDCl_3$, 300 MHz) δ 11.6 (s, 1H, NH), 8.15 (dd, *J* = 8.01 Hz, 1H, CH), 7.54 (ddd, *J* = 7.47, 7.47, 1.44 Hz, 1H, CH), 7.84 (d, *J* = 7.74 Hz, 1H, CH), 7.44-7.26 (m, 3H), 7.16 (m, 1H), 5.13 (br.s, 1H, NH), 4.40 (d, *J* = 5.55 Hz, 2H, CH_2), 4.27-4.23 (m, 2H, CH_2), 3.06 (t, *J* = 6.42 Hz, 2H, CH_2), 1.45 (s, 9H, 3 x CH_3).

^{13}C NMR ($CDCl_3$, 75 MHz) δ 171.2, 167.3, 155.7, 152.2, 140.4, 134.9, 133.6, 131.2, 128.6, 128.5, 127.5, 127.2, 124.6, 122.9, 77.9, 41.5, 40.2, 28.2, 26.9.

MS (ES$^+$) *m/z* 418 (M+H).

HRMS (ES$^+$) Calculated for $C_{22}H_{26}N_3O_4$a(M + H): 396.1923; found 396.1923

Methyl 2-iodobenzoate (22)

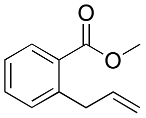


2-Iodobenzoic acid **21**(1.0 g, 4.0 mmol) was dissolved in dry DMF (4 mL) and treated with Cs_2CO_3 (5.23 g, 16 mmol). Methyl iodide (2.3 mL, 16 mmol) was added and the suspension was warmed to 50°C. After 1 h, the reaction was poured onto 1M HCl (10 mL) and extracted with EtOAc (3 x 10 mL). The combined organic layers were washed with $NaHCO_3$ (10 mL), brine (10 mL), H_2O (10 mL), dried ($MgSO_4$), filtered and concentrated, yielding a slightly yellow oil (1.0 g, 95%) as reported in the literature.[1]

^{1}H NMR ($CDCl_3$, 300 MHz) δ7.86 (ddd, *J* = 6.10, 4.89, 4.89 Hz, 1H, CH), 7.67 (dd, *J* = 7.77, 1.74 Hz, 1H, CH), 7.30-7.25 (m, 1H), 7.05-6.99 (m, 1H), 3.81 (s, 3H, CH_3).

^{13}C NMR ($CDCl_3$, 75 MHz) δ 166.8, 141.2, 135.1, 132.6, 130.8, 127.8, 94.0, 52.4
MS (ES^+) *m/z* 263 (M+H).
JOC, 73 (17), 6642-6646, 2008

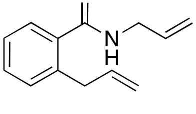
Methyl 2-allylbenzoate (23)



The aryl iodide **22** (0.58 g, 2.2 mmol), $PdPPh_3$ (0.12 g, 0.11 mmol) and allyltributyltin (0.82 mL, 2.6 mmol) were dissolved in dry DMF (2 mL) and microwave irradiated at 120°C for 10 mins. The black suspension was filtered through celite and the cake washed with EtOAc (10 mL). The filtrate washed with 1M HCl (2 x 10 mL), H_2O (10 mL), dried, filtered and concentrated. The crude product was purified via flash column chromatography, eluting with EtOAc/pet. ether (0-5%), affording a clear oil (0.274 g, 74%) as reported in the literature.[2]

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.89 (ddd, J = 7.32, 3.03, 3.03 Hz, 1H, CH), 7.43 (ddd, J = 7.02, 2.60, 2.60 Hz, 1H, CH), 7.24 (m, 2H), 6.08-5.95 (m, 1H), 5.06-4.99 (m, 2H), 3.87 (s, 3H, CH_3), 3.77 (d, J = 6.40 Hz, 2H, CH_2).
^{13}C NMR ($CDCl_3$, 75 MHz) δ 168.3, 141.7, 137.6, 132.2, 131.1, 130.8, 129.9, 126.4, 115.8, 52.1, 38.6
MS (ES^+) *m/z* 177 (M+H).
JOC, 68 (6), 2195-2199, 2003

N-2-diallybenzamide (24)



Allylamine (193 µL, 3.1 mmol) in dry THF (5 mL) was treated with DABAL (0.531 g, 2.0 mmol). The mixture was stirred at room temperature for 15 mins. After this time, the ester **23** (0.450 g, 2.5 mmol) was added and the mixture was microwave irradiated for 2h at 100°C. The reaction was quenched with 1M HCl (10 mL) and extracted with Et_2O (3 x 10 mL). The combined extracts were washed with H_2O (10 mL), brine (10 mL), dried over $MgSO_4$ and concentrated. Flash column chromatography, eluting with 20 % EtOAc/pet.ether afforded **24** as white solid (0.312 g, 60%).

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.33-7.26 (m, 2H), 7.20-7.10 (m, 2H), 6.27 (br. s, 1H), 6.01-5.78 (m, 2H), 5.21-4.93 (m, 4H), 3.96-3.92 (m, 2H) 3.50 (d, *J* = 6.24 Hz, 2H).

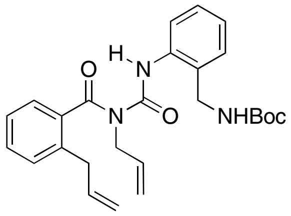
^{13}C NMR ($CDCl_3$, 75 MHz) δ 169.72, 137.65, 137.56, 136.37, 134.07, 130.34, 129.96, 127.23, 126.23, 116.47, 115.97, 42.16, 37.41.

IR (ATR cm^{-1}) 3289, 3076, 1638, 914.

MS (ES$^+$) *m/z* 202 (M+H).

HRMS (ES$^+$) Calculated for $C_{13}H_{16}NO$ (M+H): 202.1232; found 202.1233.

***tert*-Butyl 2-(3-allyl-3-(2-allylbenzoyl)ureido)benzylcarbamate (25)**:



The amide **24** (0.378 g, 1.9 mmol) and the amine **19** (0.464 g, 2.1 mmol) were treated according to general procedure D. Flash column chromatography, eluting with EtOAc/pet. ether (20 %), afforded the benzoylurea **25** as a clear solid (0.725 g, 85%).

^{1}H NMR ($CDCl_3$, 300 MHz) δ 11.25 (s, 1H), 7.85-7.19 (m, 8H), 5.91-5.79 (m, 2H), 5.16-4.94 (m, 4H), 4.99-4.94 (m, 1H), 4.36-4.34 (m, 4H), 3.45 (d, *J* = 6.5 Hz, 2H), 1.45 (s, 9H).

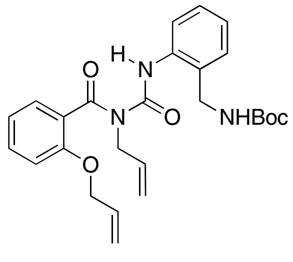
^{13}C NMR ($CDCl_3$, 75 MHz) δ 175.4, 155.9, 152.4, 135.9, 135.7, 135.5, 135.3, 133.1, 130.9, 130.1, 130.0, 129.7, 128.3, 126.2, 125.5, 123.7, 117.4, 117.2, 79.5, 49.1, 41.6, 37.6, 28.4.

IR (ATR cm^{-1}) 3763, 3367, 3221, 1706, 1074, 750

MS (ES$^+$) *m/z* 450 (M+H)

HRMS (ES+) Calculated for $C_{26}H_{31}N_3NaO_4$ (M + Na): 472.2212; found 472.2215

tert-Butyl 2-(3-allyl-3-(2-(allyloxy)benzoyl)ureido)benzylcarbamate (26)



The amide **8** (0.502 g, 2.3 mmol) and the aniline **19** (0.562 g, 2.5 mmol) were treated as described in general procedure D. Purification via flash column chromatography, eluting with EtOAc/pet.ether (25 %) afforded the benzoylurea **26** as a clear oil (0.810 g, 74 %).

^{1}H NMR (CDCl$_3$, 300 MHz) δ 11.23 (s, 1H), 7.86-6.63 (m, 8H), 6.03-5.93 (m, 1H), 5.83-5.74 (m, 1H), 5.41-4.88 (m, 5H), 4.60-4.58 (m, 2H), 4.31-4.29 (m, 2H), 4.21 (d, *J* = 6.2 Hz, 2H), 1.43 (s, 9H).

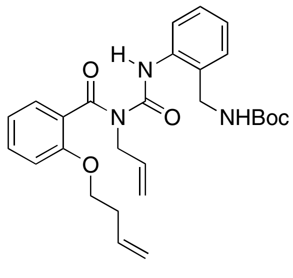
^{13}C NMR (CDCl$_3$, 75 MHz) δ 173.7, 155.9, 153.6, 152.4, 145.7, 135.4, 133.4, 132.4, 131.4, 129.6, 128.2, 127.5, 125.9, 125.3, 123.6, 120.9, 117.8, 116.8, 112.4, 79.7, 69.1, 48.5, 41.4, 28.4.

IR (ATR cm^{-1}) 3368, 3221, 2979, 1703, 1103, 746.

MS (ES$^+$) *m/z* 466 (M + H)

HRMS (ES$^+$) Calculated for $C_{26}H_{32}N_3O_5$ (M+H): 466.2342; found 466.2337.

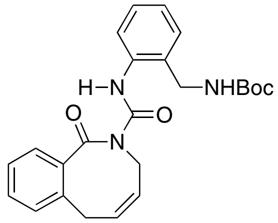
tert-Butyl 2-(3-allyl-3-(2-(but-3-enyloxy)benzoyl)ureido)benzylcarbamate (27)



The amide **9** (0.134 g, 0.58 mmol) and the aniline **19** (0.135 g, 0.61 mmol) were treated as described in general procedure D. Purification *via* flash column chromatography, eluting with EtOAc/pet. ether (40 %) afforded the benzoylurea **27** as a yellow oil (0.194 g, 70 %).

^{1}H NMR ($CDCl_3$, 300 MHz) δ 11.22 (s, 1H), 7.86-6.91 (m, 8H), 5.84-5.73 (m, 2H), 5.16-4.87 (m, 5H), 4.71-4.33 (br.s, 2H), 4.31 (d, J = 6 Hz, 2H), 4.06 (t, J = 6.5 Hz, 2H), 2.54 - 2.52 (m, 2H), 1.48 (s, 9H).
^{13}C NMR ($CDCl_3$, 75 MHz) δ 171.3, 153.5, 151.4, 149.9, 132.9, 131.4, 130.9, 128.9, 128.6, 127.1, 125.8, 125.1, 123.4, 122.8, 121.1, 118.3, 115.1, 114.4, 109.5, 76.9, 69.1, 45.9, 38.9, 31.1, 25.9.
IR (ATR, cm^{-1}) 3353, 3077, 2977, 1707, 1649, 1589, 1509, 1451, 1366, 1162, 859
MS (ES^{+}) *m/z* 502 (M + Na),
HRMS (ES^{+}) Calculated for $C_{27}H_{34}N_3O_5$ (M + H): 480.2498; found 480.2496

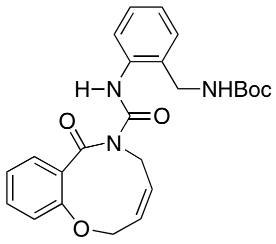
(*Z*)-*tert*-Butyl-2-(1-oxo-1,2,3,6-tetrahydrobenzo[*c*]azocine-2-carboxamido)benzylcarbamate (28)



The benzoylurea **25** (0.620 g, 1.38 mmol) and Grubbs-II (0.059 g, 0.77 mmol) were treated according to general procedure E. Purification via flash column chromatography, eluting with EtOAc/pet. ether (20 %), afforded **28** as a light grey residue (0.414 g, 74 %).

^{1}H NMR ($CDCl_3$, 300 MHz) δ 11.28 (s, 1H), 7.86 (1H, s, CH), 7.48 (2H, m), 7.36-7.25 (3H, m), 7.17-7.12 (2H, m), 5.60-5.89 (2H, m), 5.18 (1H, br.s, CH), 4.49 (1H, br.s, CH), 4.33 (2H, d, J = 5.73 Hz, CH_2), 3.83-3.51 (2H, m), 3.31 (1H, br.s, CH), 1.44 (9H,s, CH_3).
^{13}C NMR ($CDCl_3$, 75 MHz) δ 175.8, 158.8, 151.9, 136.6, 135.4, 133.1, 131.9, 130.5, 129.8, 129.5, 129.4, 128.3, 127.9, 127.2, 125.8, 125.2, 123.4, 79.5, 43.3, 41.7, 32.9, 28.4.
IR (ATR cm^{-1}) 3361, 3222, 2977, 1702, 1140, 752.
MS (ES^{+}) *m/* 444 (M+H)
HRMS (ES^{+}) Calculated for $C_{24}H_{27}N_3NaO_4$ (M + Na): 444.1899; found 444.1901

(*Z*)-*tert*-butyl(4-(7-oxo-2,5,6,7-tetrahydrobenzo[*b*][1,5]oxazonine-6-carboxamido)-1,3-phenylene)bis(methylene)dicarbamate (29)



The benzoylurea **26** (0.500 g, 1.1 mmol) and Grubbs-II (0.091g, 0.11 mmol) were treated according to the general procedure E. Purification via flash column chromatography, eluting with EtOAc/pet. ether (20 %), afforded **29** as a light brown residue (0.325 g, 70 %).

^{1}H NMR ($CDCl_3$, 300 MHz) δ 11.3 (s, 1H), 7.85-7.05 (m, 8H), 6.30-6.26 (m, 1H), 6.03-5.98 (m, 1H), 5.21 (br.s, 1H), 4.86 (br.s, 1H), 4.35 (d, *J* = 6.6 Hz, 2H, CH_2), 4.09 (br.s, 3H), 1.44 (s, 9H).

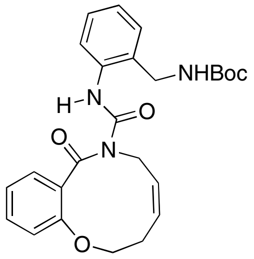
^{13}C NMR ($CDCl_3$, 75 MHz) δ 173.1, 155.9, 152.7, 135.4, 133.0, 132.8, 130.9, 129.7, 129.5, 129.0, 128.2, 128.0, 125.3, 123.6, 123.5, 123.4, 118.1, 79.4, 71.7, 46.5, 41.5, 28.4.

IR (ATR, cm^{-1}) 2961, 2340, 1710, 1589, 1367, 1166, 756

MS (ES$^+$) *m/z* 460 (M + Na)

HRMS (ES$^+$) Calculated for $C_{24}H_{287}N_3O_5$ (M + H): 438.2029; found 438.2032.

(*Z*)-*tert*-butyl-2-(8-oxo-3,6,7,8-tetrahydro-2*H*-benzo[*b*][1,5]oxazecine-7-carboxamido)benzylcarbamate (30)



The benzoylurea **27** (0.175 g, 0.36 mmol) and Grubbs-II (0.031 g, 0.038 mmol) were treated according to general procedure E. Purification via flash column chromatography, eluting with EtOAc/pet. ether (20 %), afforded **30** as a light brown residue (0.113 g, 71 %).

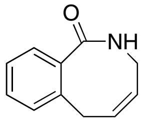
^{1}H NMR ($CDCl_3$, 300 MHz) δ 11.28 (s, 1H), 7.87 (s, 1H, CH), 7.52-7.48 (m, 2H), 7.37-7.28 (m, 2H), 7.22-7.10 (m, 3H), 6.25-6.14 (m, 1H), 5.86-5.74 (m, 1H), 5.23 (br.s, 1H, NH), 4.70 (br.s, 1H, CH), 4.35 (s, 2H, CH_2), 4.31-3.98 (m, 3H), 2.44 (br.s, 2H, CH_2), 1.42 (s, 9H, CH_3).

^{13}C NMR ($CDCl_3$, 75 MHz) δ 173.8, 155.9, 152.7, 152.5, 135.4, 131.3, 129.6, 129.4, 128.7, 128.3, 128.2, 125.5, 123.8, 122.5, 114.6, 80.0, 66.5, 44.5, 40.1, 28.4, 25.9.

IR (ATR, cm^{-1}) 2960, 2340, 1710, 1522, 1166,756.

HRMS (ES^+) Calculated for $C_{25}H_{29}N_3NaO_5$ (M + Na): 474.2005; found 474.2015.

(Z)-2,3-dihydrobenzo[c]azocin-1(6H)-one (31)



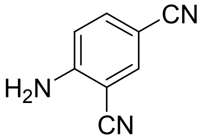
The benzoylurea **28** (0.320 g, 0.76 mmol) was treated according to general procedure C. Flash column chromatography, eluting with MeOH/DCM (5 %), afforded **31** as a white solid (0.082 g, 62 %).

^{1}H NMR (MeOD, 300 MHz) δ 7.46-7.30 (m, 3H), 7.19 (dd, J = 7.56, 0.48 Hz, 1H, CH), 6.05-5.96 (m, 1H), 5.66-5.48 (m, 1H), 3.51-3.45 (m, 4H).

^{13}C NMR (MeOD, 75 MHz) δ 179.7, 139.4, 138.7, 134.6, 133.4, 132.9, 131.1, 130.5, 129.5, 43.7, 35.8.

HRMS (ES^+) Calculated for $C_{11}H_{12}NO$ (M + H): 174.0917; found 174.0919.

4-Aminoisophthalonitrile (33)



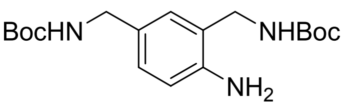
A dry round bottom flask was charged with 2-amino-5-bromo benzonitrile, **33** (1.0 g, 5.1 mmol) and zinc cyanide (0.6 g, 5.1 mmol), and DMF (5 mL) was added. Nitrogen was bubbled through the solution for 5 minutes, and then Pd[$(Ph)_3]_4$ (0.47 g, 0.41 mmol) was added in one portion. The reaction mixture was stirred at 90°C overnight. After cooling to room temperature, saturated $NaHC0_3$ (10 mL) was added, and the mixture was extracted with EtOAc (3 x 10 mL). The organic extracts were collected, washed with brine (15 mL) and dried over $NaSO_4$, filtered and concentrated. The crude product was purified via flash chromatography, eluting with MeOH/DCM (2 %), affording **33** as a white solid (0.45 g, 62 %) as described in the literature[3].

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.70 (d, J = 1.95 Hz, 1H), 7.53 (d, J = 6.48 Hz, 1H), 6.77 (d, 7.86, 1H), 4.94 (br s, 2H)

^{13}C NMR ($CDCl_3$, 75 MHz) δ 152.2, 137.1, 136.9, 117.7, 115.4, 101.2, 96.5

MP 212 - 215°C

***tert*-Butyl (4-amino-1,3-phenylene)bis(methylene)dicarbamate (34)**



The 4-aminoisophthalonitrile, **33** (0.352 g, 2 mmol), along with $NiCl_2.6H_2O$ (32 mg, 0.244 mmol) was dissolved in dry MeOH (5 mL). The reaction was cooled to 0°C and $NaBH_4$ (0.65 g, 0.017 mol) was added in portions over 30 min. $(BOC)_2O$ (1.07 g, 5 mmol) was dissolved in dry MeOH (2 mL) and was added dropwise into the mixture, the reaction warmed up to room temperature and left stirring under a N_2 atmosphere for 5 h. Diethylaminetriamine (264 µl, 2 mmol) was added and left stirring for 30 min. After the completion of the reaction the MeOH was concentrated *in vacuo*, redissolved in EtOAc (10 mL) and extracted with saturated $NaHCO_3$ (3 x 10 mL), water (2 x 15 mL) and brine (15 mL), dried over $MgSO_4$, filtered and concentrated, affording a clear oil (0.56 g, 65%).

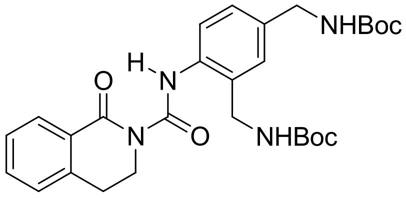
^{1}H NMR (CDCl3, 300 MHz): δ 6.98 (dd J = 10.0, 6.0 Hz, 1H), 6.93 (d, J = 1.7 Hz, 1H), 6.59 (d, J = 8.0 Hz, 1H), 4.82 (br s, 1H), 4.73 (br s, 1H), 4.21 (br s, 2H), 4.19 (d, J = 6.0 Hz, 2H), 4.14 (d, J = 6.0 Hz, 2H), 1.45 (s, 18H).

^{13}C NMR (CDCl3, 75 MHz): δ 156.60, 155.97, 144.91, 129.89, 128.62, 128.21, 122.85, 115.97, 79.96, 79.46, 44.39, 42.17, 28.57, 28.50.

MS (ES$^+$) *m/z* 702.9 (2M+H);

HRMS (ES$^+$) Calculated for $C_{18}H_{29}N_3NaO_4$ (M+Na): 374.2056; found 374.2051.

tert-Butyl-(4-(1-oxo-1,2,3,4-tetrahydroisoquinoline-2-carboxamido)-1,3-phenylene)bis(methylene)dicarbamate (35)



The amide **18** (0.050 g, 0.3 mmol) and the aniline **34** (0.119 g 0.3 mmol) were treated according to general procedure D. Flash column chromatography, eluting with EtOAc/pet. ether (25 %), afforded the benzoylurea **35** as a white solid (0.111 g, 62%).

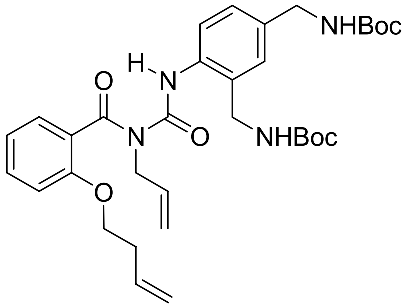
^{1}H NMR (CDCl$_3$, 300 MHz): δ 11.57 (br s, 1H), 8.12 (d, J = 7.8 Hz, 1H), 7.78 (d, J = 6.9 Hz, 1H), 7.51 (t, J = 7.5 Hz, 1H), 7.38 (t, J = 7.0 Hz, 1H), 7.22 (m, 3H), 5.10 (br s, 1H), 4.81 (br s, 1H), 4.36-4.23 (m, 6H), 3.03 (t, J = 6.4 Hz, 2H), 1.46 (s, 9H), 1.45 (s, 9H).

^{13}C NMR (CDCl$_3$, 75 MHz): δ 156.01, 153.08, 140.09, 136.03, 134.88, 133.70, 131.24, 129.57, 129.04, 128.56, 127.55, 127.30, 124.05, 115.96, 79.63, 77.36, 44.39, 41.91, 41.73, 28.56, 28.01.

MS (ES$^+$) *m/z* 547.2 (M+Na);

HRMS (ES$^+$) Calculated for $C_{28}H_{36}N_4NaO_6$ (M+Na): 547.2533, found 547.2529

tert-Butyl-(4-(3-allyl-3-(2-(but-3-enyloxy)benzoyl)ureido)-1,3-phenylene)bis(methylene)dicarbamate (36)



The amide **9** (0.050 g, 0.2 mmol) and the aniline **34** (0.076 g, 0.2 mmol) were treated as described in general procedure D. Column chromatography, eluting with EtOAc/pet. ether (25 %), afforded the benzoylurea **36** as a clear oil (0.102 g, 65%).

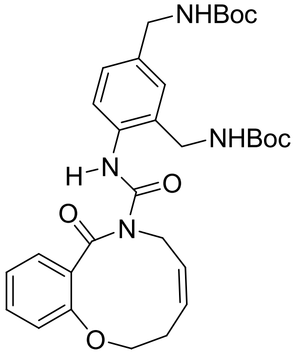
^{1}**H NMR** (CDCl$_3$, 300 MHz): δ 11.19 (br s, 1H), 7.78 (s, 1H), 7.37 (t, *J* = 8.3 Hz, 1H), 7.21- 7.28 (m, 3H), 6.98 (t, *J* = 7.5 Hz, 1H), 6.92 (d, *J* = 8.4 Hz, 1H), 5.72- 5.84 (m, 2H), 4.85-5.15 (m, 6H), 4.28 (d, *J* = 5.7 Hz, 4H), 4.04 (t, *J* = 6.5 Hz, 4H), 2.52 (q, J = 9.6 Hz, 2H), 1.46 (s, 9H), 1.43 (s, 9H).

^{13}**C NMR** (CDCl$_3$, 75 MHz): δ 173.90, 156.08, 155.96, 154.00, 152.58, 136.09, 134.74, 133.98, 133.54, 131.55, 131.41, 128.60, 127.66, 127.47, 126.01, 123.99, 120.95, 117.70, 116.98, 112.05, 79.67, 77.36, 67.73, 48.59, 44.44, 41.55, 33.65, 28.55.

MS (ES$^+$) *m/z* (M+Na) 631.1

HRMS (ES$^+$) *m/z* Calculated for $C_{33}H_{44}N_4NaO_7$ (M+Na) 631.3108, found 631.3106

(Z)-*tert*-butyl-(4-(8-oxo-3,6,7,8-tetrahydro-2H-benzo[b][1,5]oxazecine-7-carboxamido)-1,3-phenylene)bis(methylene)dicarbamate (37)



The benzoylurea **36** (0.102 g, 0.21 mmol) and Grubbs-II (0.014 g, 0.02 mmol) were treated according to the general procedure E. Purification via flash column chromatography, eluting with EtOAc/pet. spirits (25 %), afforded **37** as a light brown residue (0.068 g, 70 %).

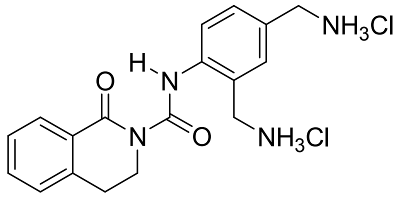
^{1}**H NMR** (CDCl$_3$, 300 MHz): δ 11.30 (br s, 1H), 7.76 (s, 1H), 7.43 (td, *J* = 8.4, *J* = 1.1 Hz, 1H), 7.09-7.34 (m, 5H), 6.12 (q, *J* = 8 Hz, 1H), 5.74 (q, *J* = 9 Hz, 1H), 5.18 (br s, 1H), 4.82 (br s, 1H), 4.28-4.32 (m, 6H), 2.15 (t, *J* = 1.77 Hz, 2H), 1.46 (s, 9H), 1.42 (s, 9H).

^{13}**C NMR** (CDCl$_3$, 75 MHz): δ 173.95, 156.07, 155.97, 152.86, 152.65, 134.65, 131.54, 129.53, 128.86, 128.63, 128.45, 128.41, 127.48, 122.72, 114.77, 79.66, 77.37, 66.64, 53.99, 44.65, 44.45, 41.63, 31.86, 29.42, 28.57, 26.06.

MS (ES$^+$): *m/z* (M+Na) 603.1

HRMS (ES$^+$) *m/z* Calculated for $C_{31}H_{40}N_4NaO_7$ (M+Na) 603.2795, found 603.2789

N-(2,4-bis(aminomethyl)phenyl)-1-oxo-3,4-dihydroisoquinoline-2(1H)-carboxamide hydrochloride (38)



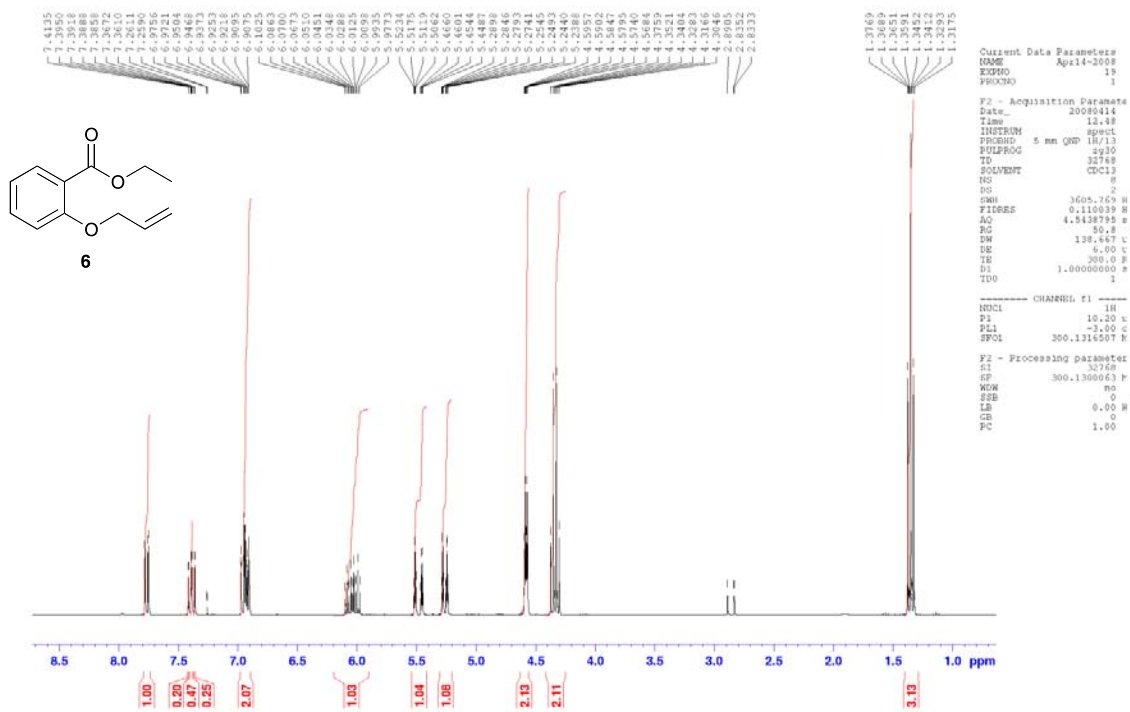
Benzoylurea **35** (0.072 g 0.2 mmol) was dissolved in dry 1,4-dioxane (1 mL). In a separate flask, HCl gas was generated from NaCl/H_2SO_4 and bubbled through the solution. After 20 mins the free amine precipitated from the solution as the hydrochloride salt, filtered and washed with diethyl ether (5 mL)

^{1}H NMR (D_2O, 300 MHz): δ 8.09 (d, J = 8.0 Hz, 1H), 7.65 (m, 4H), 7.52 (d, J = 7.7 Hz, 1H), 7.44 (t, J = 7.6 Hz, 1H), 4.30 (d, J = 5.6 Hz, 4H), 4.15 (t, J = 6.5 Hz, 2H), 3.11 (t, J = 6.0 Hz, 2H).

^{13}C NMR ($CDCl_3$, 75 MHz): δ 169.18, 155.57, 140.80, 135.88, 134.38, 132.11, 130.86, 130.76, 128.93, 128.76, 127.91, 127.75, 127.58, 127.43, 42.65, 42.42, 39.15, 26.89.

MS (ES$^+$) *m/z* (M+H) 395.28

HRMS (ES$^+$) *m/z* Calculated for $C_{18}H_{21}N_4O_2$ (M+1); 325.1665, found 325.1660


O
O
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7.3918
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6.9721
6.9504
6.9468
6.9373
6.9253
6.9218
6.9095
6.9075
6.1025
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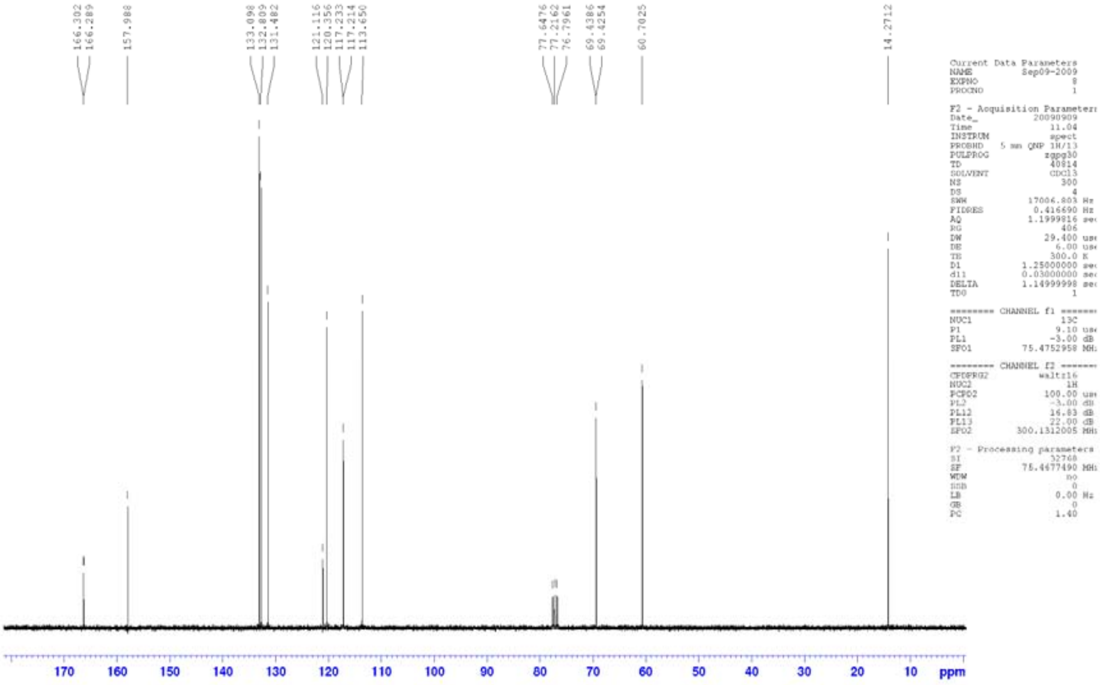
6.0700
6.0673
6.0510
6.0451
6.0348
6.0288
6.0125
5.9938
5.9935
5.9773
5.5234
5.5175
5.5119
5.5062
5.4660
5.4601
5.4544
5.4487
5.2898
5.2846
5.2793
5.2741
5.2545
5.2493
5.2440
5.2388
4.5957
4.5902
4.5847
4.5795
4.5740
4.5684
4.3759
4.3521
4.3404
4.3283
4.3166
4.3046
2.8905
2.8352
2.8333
1.3769
1.3689
1.3651
1.3591
1.3452
1.3412
1.3293
1.3175
Current Data Parameters
NAME Apr14-2008
EXPNO 19
PROCNO 1
F2 - Acquisition Paramete
Date_ 20080414
Time 12.48
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 3605.769 H
FIDRES 0.110039 H
AQ 4.5438795 s
RG 50.8
DW 138.667 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 10.20 u
PL1 -3.00 d
SFO1 300.1316507 M
F2 - Processing parameter
SI 32768
SF 300.1300063 M
WDW no
SSB 0
LB 0.00 H
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0 ppm
1.00
0.20
0.47
0.25
2.07
1.03
1.04
1.08
2.13
2.11
3.13

6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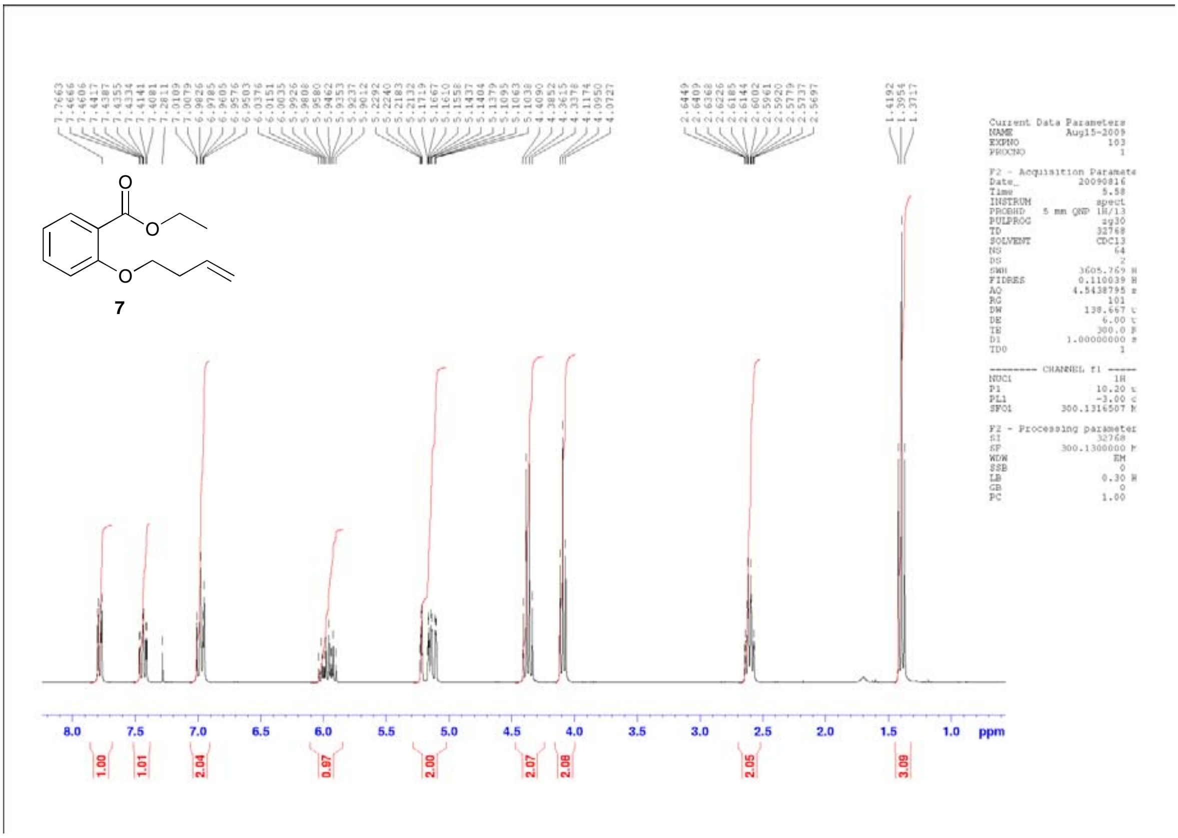
166.289
157.988
133.098
132.809
131.482
121.116
120.356
117.233
117.214
113.650
77.6476
77.2162
76.7961
69.4386
69.4254
60.7025
14.2712
Current Data Parameters
NAME Sep09-2009
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090909
Time 11.04
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 40814
SOLVENT CDCl3
NS 300
DS 4
SWH 17006.803 Hz
FIDRES 0.416690 Hz
AQ 1.1999816 sec
RG 406
DW 29.400 use
DE 6.00 use
TE 300.0 K
D1 1.25000000 sec
d11 0.03000000 sec
DELTA 1.14999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.10 use
PL1 -3.00 dB
SFO1 75.4752958 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 use
PL2 -3.00 dB
PL12 16.83 dB
PL13 22.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4477490 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


7
7.7663
7.4666
7.4606
7.4417
7.4387
7.4355
7.4334
7.4141
7.4081
7.2811
7.0109
7.0079
6.9826
6.9785
6.9605
6.9576
6.9503
6.0376
6.0151
6.0035
5.9926
5.9808
5.9580
5.9462
5.9353
5.9237
5.9012
5.2292
5.2240
5.2183
5.2132
5.1719
5.1667
5.1610
5.1558
5.1437
5.1404
5.1379
5.1095
5.1063
5.1038
4.4090
4.3852
4.3615
4.3378
4.1174
4.0950
4.0727
2.6449
2.6409
2.6368
2.6225
2.6185
2.6144
2.6002
2.5961
2.5920
2.5779
2.5737
2.5697
1.4192
1.3954
1.3717
Current Data Parameters
NAME Aug15-2009
EXPNO 103
PROCNO 1
F2 - Acquisition Paramete
Date_ 20090816
Time 5.58
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 2
SWH 3605.769 H
FIDRES 0.110039 H
AQ 4.5438795 s
RG 101
DW 138.667 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
TD0 1
-------- CHANNEL f1 -----
NUC1 1H
P1 10.20 u
PL1 -3.00 d
SFO1 300.1316507 M
F2 - Processing parameter
SI 32768
SF 300.1300000 M
WDW EM
SSB 0
LB 0.30 H
GB 0
PC 1.00
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
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ppm
1.00
1.01
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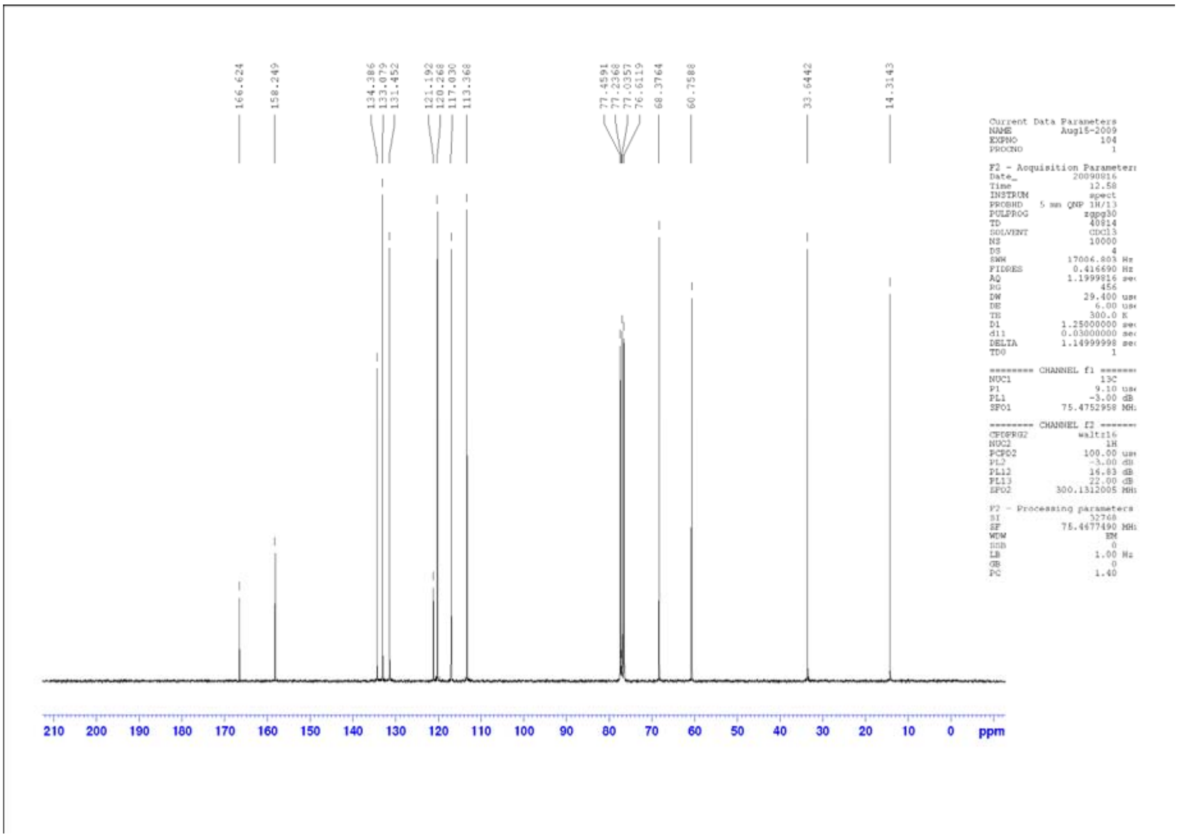
0.97
2.00
2.07
2.08
2.05
3.09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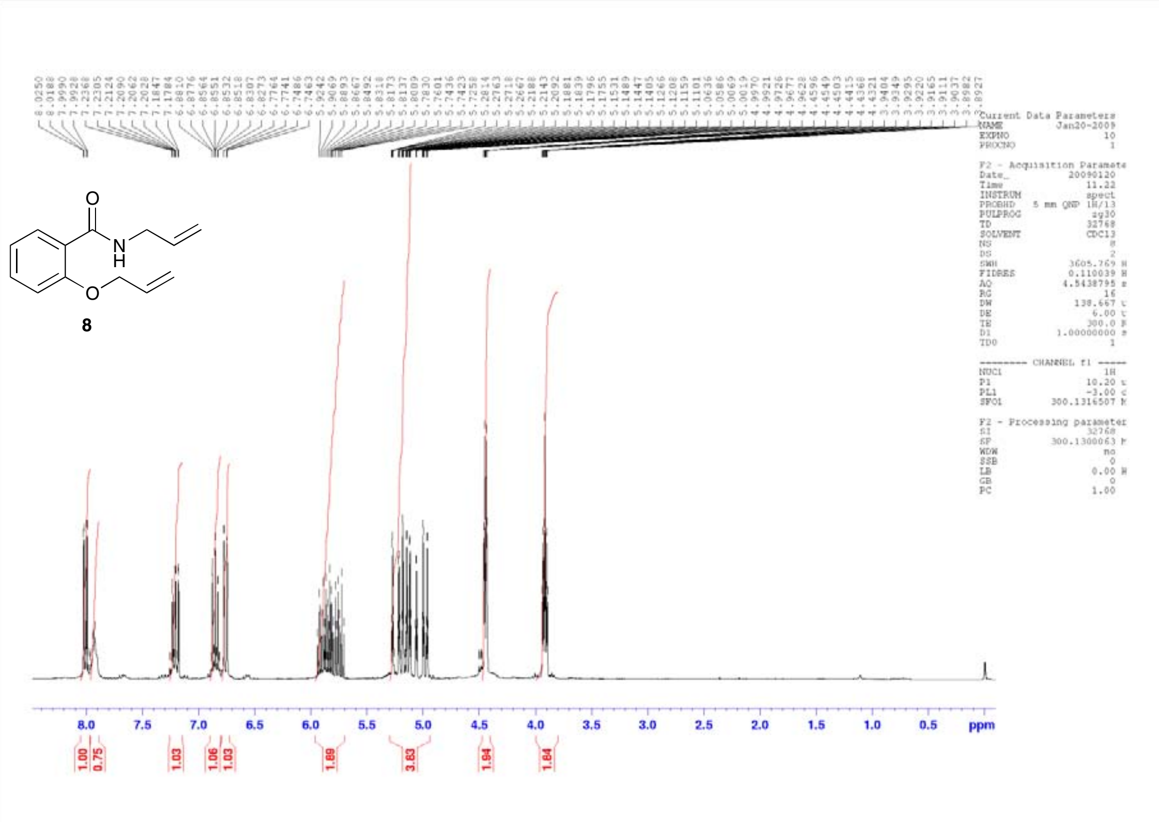
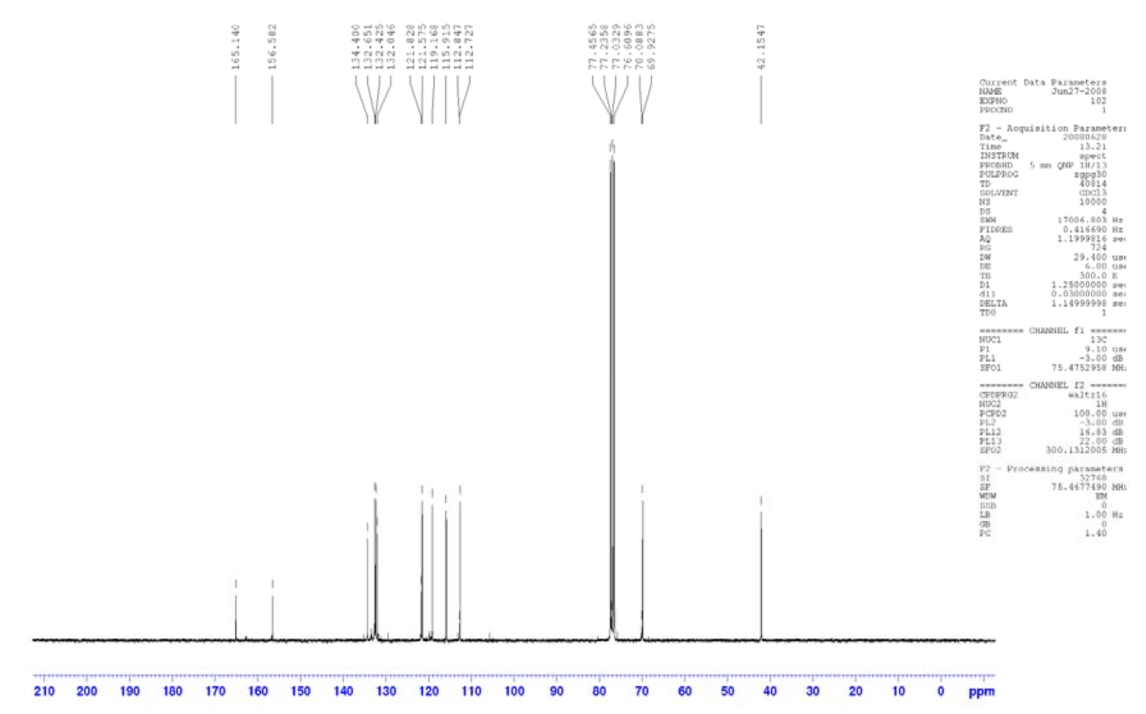
134.386
133.079
131.452
121.192
120.268
117.030
113.368
77.4591
77.2368
77.0357
76.6119
68.3764
60.7588
33.6442
14.3143
Current Data Parameters
NAME Aug15-2009
EXPNO 104
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090816
Time 12.58
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 40814
SOLVENT CDCl3
NS 10000
DS 4
SWH 17006.803 Hz
FIDRES 0.416690 Hz
AQ 1.1999816 sec
RG 456
DW 29.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.25000000 sec
d11 0.03000000 sec
DELTA 1.14999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.10 usec
PL1 -3.00 dB
SFO1 75.4752958 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 16.83 dB
PL13 22.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

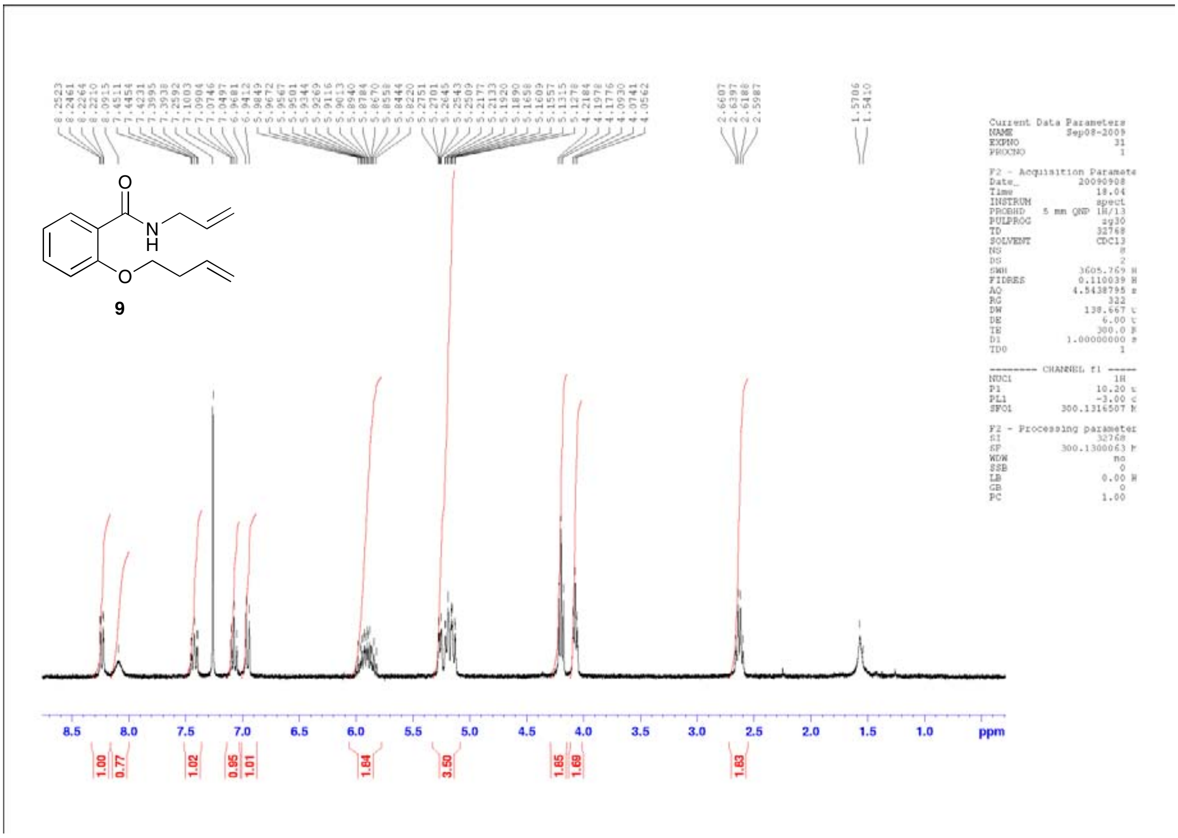


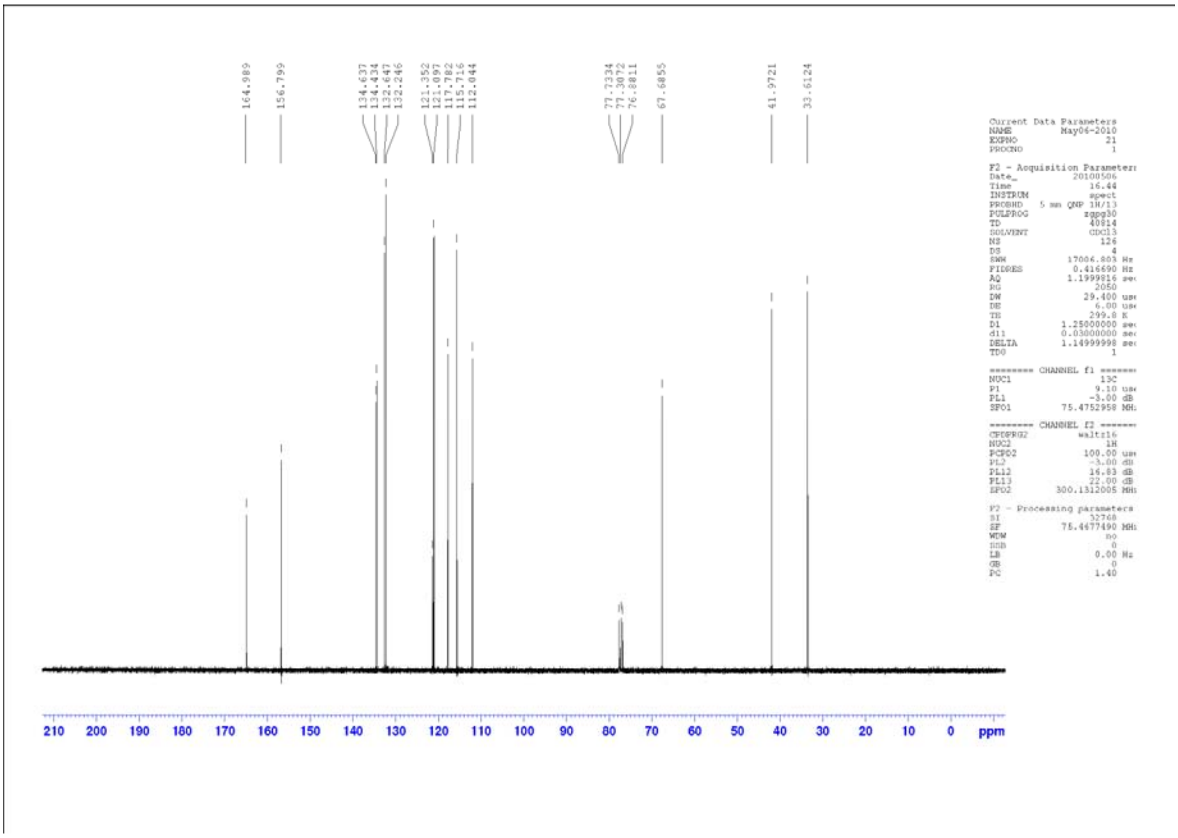
8

8.0250
8.0188
7.9990
7.9928
7.2368
7.2305
7.2124
7.2090
7.2062
7.2028
7.1847
7.1784
6.8810
6.8776
6.8564
6.8551
6.8532
6.8518
6.8307
6.8273
6.7764
6.7741
6.7486
6.7463
5.9242
5.9069
5.8893
5.8667
5.8492
5.8318
5.8173
5.8137
5.8009
5.7830
5.7601
5.7436
5.7423
5.7258
5.2814
5.2763
5.2718
5.2667
5.2188
5.2143
5.2092
5.1881
5.1839
5.1796
5.1755
5.1531
5.1489
5.1447
5.1405
5.1266
5.1208
5.1159
5.1101
5.0636
5.0586
5.0069
5.0019
4.9970
4.9921
4.9726
4.9677
4.9628
4.4596
4.4549
4.4503
4.4415
4.4368
4.4321
3.9404
3.9349
3.9295
3.9220
3.9165
3.9111
3.9037
3.8982
3.8927
O
N
H
O
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
1.00
0.75
1.03
1.06
1.03
1.89
3.83
1.84
1.84
Current Data Parameters
NAME Jan20-2009
EXPNO 10
PROCNO 1
F2 - Acquisition Paramete
Date_ 20090120
Time 11.22
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 3605.769 H
FIDRES 0.110039 H
AQ 4.5438795 s
RG 16
DW 138.667 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 10.20 u
PL1 -3.00 d
SFO1 300.1314507 M
F2 - Processing parameter
SI 32768
SF 300.1300063 M
WDW no
SSB 0
LB 0.00 H
GB 0
PC 1.00



165.140
156.582
134.400
132.651
132.425
132.046
121.828
121.575
119.168
115.915
112.847
112.727
77.4565
77.2358
77.0329
76.6096
70.0083
69.9275
42.1547
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm
Current Data Parameters
NAME Jun27-2008
EXPNO 102
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080628
Time 13.21
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 40814
SOLVENT CDCl3
NS 10000
DS 4
SWH 17006.803 Hz
FIDRES 0.416690 Hz
AQ 1.1999916 sec
RG 724
DW 29.400 use
DE 6.00 use
TE 300.0 K
D1 1.25000000 sec
d11 0.03000000 sec
DELTA 1.14999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.10 use
PL1 -3.00 dB
SFO1 75.4752958 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 use
PL2 -3.00 dB
PL12 16.83 dB
PL13 22.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4477490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


8.2523
8.2461
8.2264
8.2210
8.0915
7.4511
7.4454
7.4231
7.3995
7.3938
7.2592
7.1003
7.0904
7.0746
7.0497
6.9681
6.9412
5.9849
5.9672
5.9567
5.9501
5.9344
5.9269
5.9116
5.9013
5.8940
5.8784
5.8670
5.8558
5.8444
5.8220
5.2751
5.2701
5.2645
5.2543
5.2509
5.2177
5.2133
5.1920
5.1890
5.1658
5.1609
5.1557
5.1315
5.1278
4.2184
4.1978
4.1776
4.0930
4.0741
4.0562
2.6607
2.6397
2.6188
2.5987
1.5786
1.5410
O
N
H
O
9
Current Data Parameters
NAME Sep08-2009
EXPNO 31
PROCNO 1
F2 - Acquisition Paramete
Date_ 20090908
Time 18.04
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 3605.769 H
FIDRES 0.110039 H
AQ 4.5438795 s
RG 322
DW 138.667 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 10.20 u
PL1 -3.00 d
SFO1 300.1316507 M
F2 - Processing parameter
SI 32768
SF 300.1300063 M
WDW no
SSB 0
LB 0.00 H
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.00
0.77
1.02
0.95
1.01
1.84
3.50
1.85
1.69
1.83


164.989
156.799
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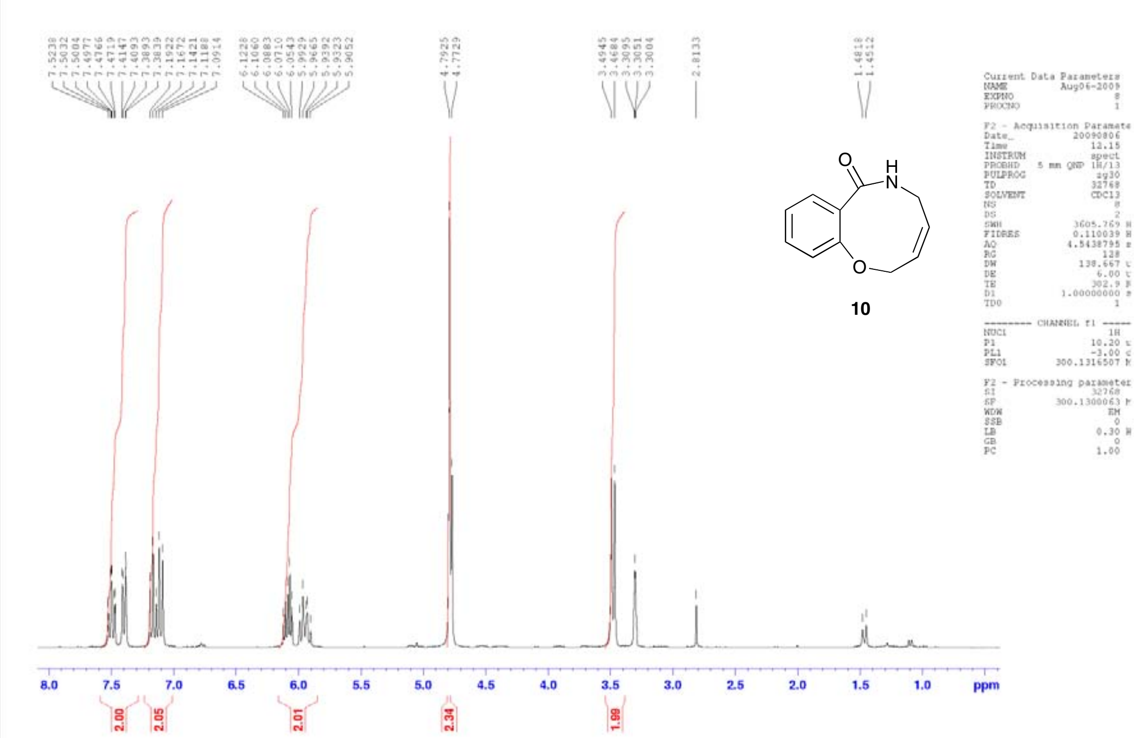
134.434
132.447
132.246
121.352
121.097
117.782
115.716
112.044
77.7334
77.3072
76.8811
67.6855
41.9721
33.6124
Current Data Parameters
NAME May06-2010
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100506
Time 16.44
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 40814
SOLVENT CDCl3
NS 126
DS 4
SWH 17006.803 Hz
FIDRES 0.416690 Hz
AQ 1.1999816 sec
RG 2050
DW 29.400 usec
DE 6.00 usec
TE 299.8 K
D1 1.25000000 sec
d11 0.03000000 sec
DELTA 1.14999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.10 usec
PL1 -3.00 dB
SFO1 75.4752958 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 16.83 dB
PL13 22.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

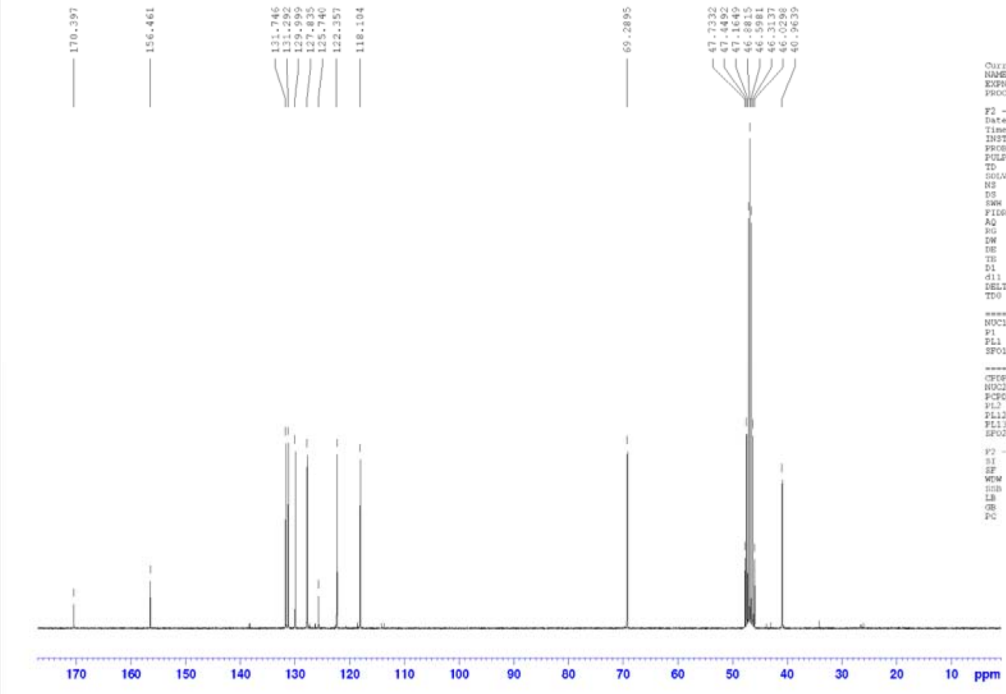


```
Current Data Parameters
NAME          Aug06-2009
EXPNO                  8
PROCNO                 1

F2 - Acquisition Paramete
Date_           20090806
Time               12.15
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             3605.769 H
FIDRES          0.110039 H
AQ             4.5438795 s
RG                   128
DW               138.667 u
DE                  6.00 u
TE                 302.9 K
D1            1.00000000 s
TD0                    1

======== CHANNEL f1 =====
NUC1                  1H
P1                 10.20 u
PL1                -3.00 d
SFO1         300.1316507 M

F2 - Processing parameter
SI                 32768
SF           300.1300063 M
WDW                   EM
SSB                    0
LB                  0.30 H
GB                     0
PC                  1.00
```



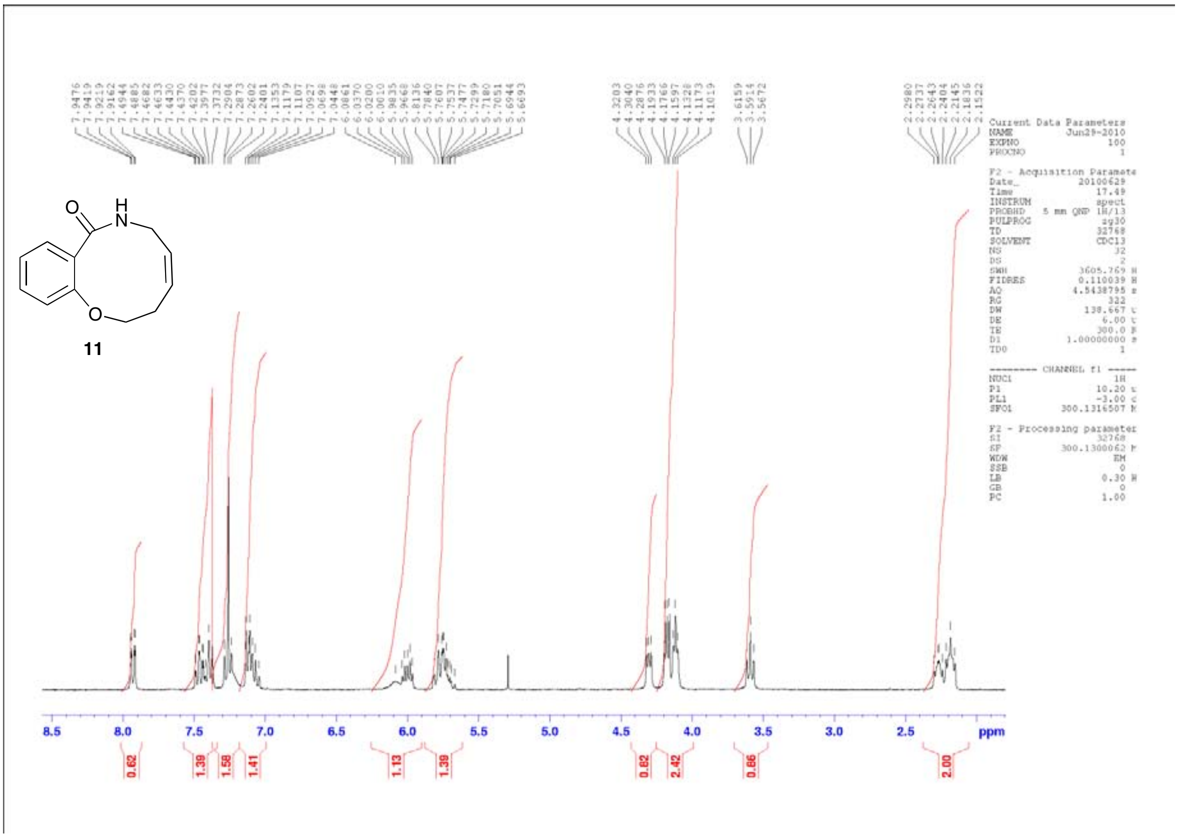
```
Current Data Parameters
NAME          Aug06-2009
EXPNO                100
PROCNO                 1

F2 - Acquisition Parameter
Date_           20090807
Time                4.05
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG           zgpg30
TD                 40814
SOLVENT             MeOD
NS                 15000
DS                     4
SWH            17006.803 Hz
FIDRES          0.416690 Hz
AQ             1.1999816 sec
RG                   362
DW                29.400 use
DE                  6.00 use
TE                 300.0 K
D1            1.25000000 sec
d11           0.03000000 sec
DELTA         1.14999998 sec
TD0                    1

======== CHANNEL f1 ======
NUC1                 13C
P1                  9.10 use
PL1                -3.00 dB
SFO1          75.4752958 MH

======== CHANNEL f2 ======
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 use
PL2                -3.00 dB
PL12               16.83 dB
PL13               22.00 dB
SFO2         300.1312005 MH

F2 - Processing parameters
SI                 32768
SF            75.4476944 MH
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```


11
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7.9419
7.9219
7.9162
7.4944
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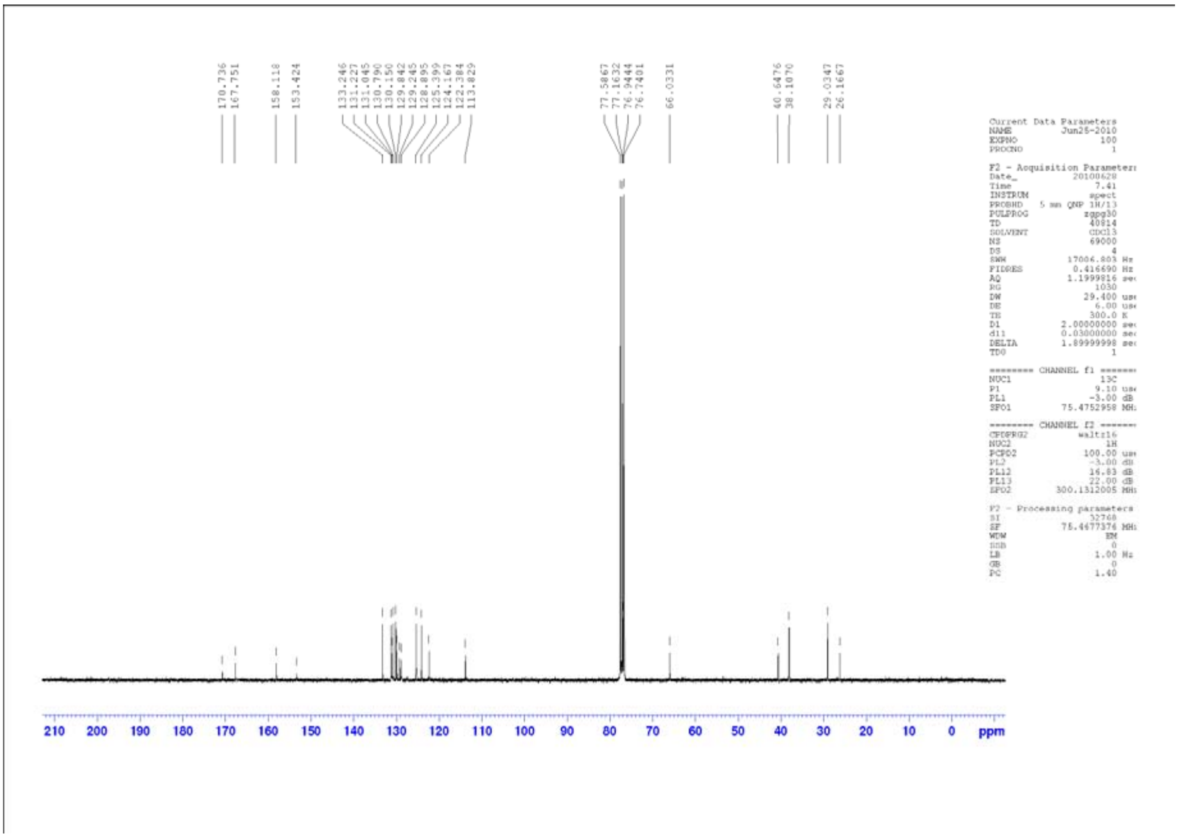
7.4682
7.4633
7.4430
7.4370
7.4202
7.3977
7.3732
7.2904
7.2873
7.2602
7.2401
7.1353
7.1179
7.1107
7.0927
7.0698
7.0448
6.0861
6.0370
6.0200
6.0010
5.9835
5.9668
5.8136
5.7840
5.7607
5.7537
5.7477
5.7299
5.7180
5.7051
5.6944
5.6893
4.3203
4.3040
4.2876
4.1933
4.1766
4.1597
4.1328
4.1173
4.1019
3.6159
3.5914
3.5672
2.2980
2.2737
2.2643
2.2404
2.2145
2.1836
2.1522
O
H
N
O
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
ppm
0.62
1.39
1.58
1.41
1.13
1.39
0.82
2.42
0.86
2.00
Current Data Parameters
NAME Jun29-2010
EXPNO 100
PROCNO 1
F2 - Acquisition Paramete
Date_ 20100629
Time 17.49
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 3605.769 H
FIDRES 0.110039 H
AQ 4.5438795 s
RG 322
DW 138.667 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.20 u
PL1 -3.00 d
SFO1 300.1314507 M
F2 - Processing parameter
SI 32768
SF 300.1300062 M
WDW EM
SSB 0
LB 0.30 H
GB 0
PC 1.00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158.118
153.424
133.246
131.227
131.045
130.790
130.150
129.842
129.445
128.895
125.399
124.167
122.384
113.829
77.5867
77.1632
76.9444
76.7401
66.0331
40.6476
38.1070
29.0347
26.1667
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm
Current Data Parameters
NAME Jun25-2010
EXPNO 100
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100628
Time 7.41
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 40814
SOLVENT CDCl3
NS 69000
DS 4
SWH 17006.803 Hz
FIDRES 0.416690 Hz
AQ 1.1999816 sec
RG 1030
DW 29.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.10 usec
PL1 -3.00 dB
SFO1 75.4752958 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 16.83 dB
PL13 22.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677376 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

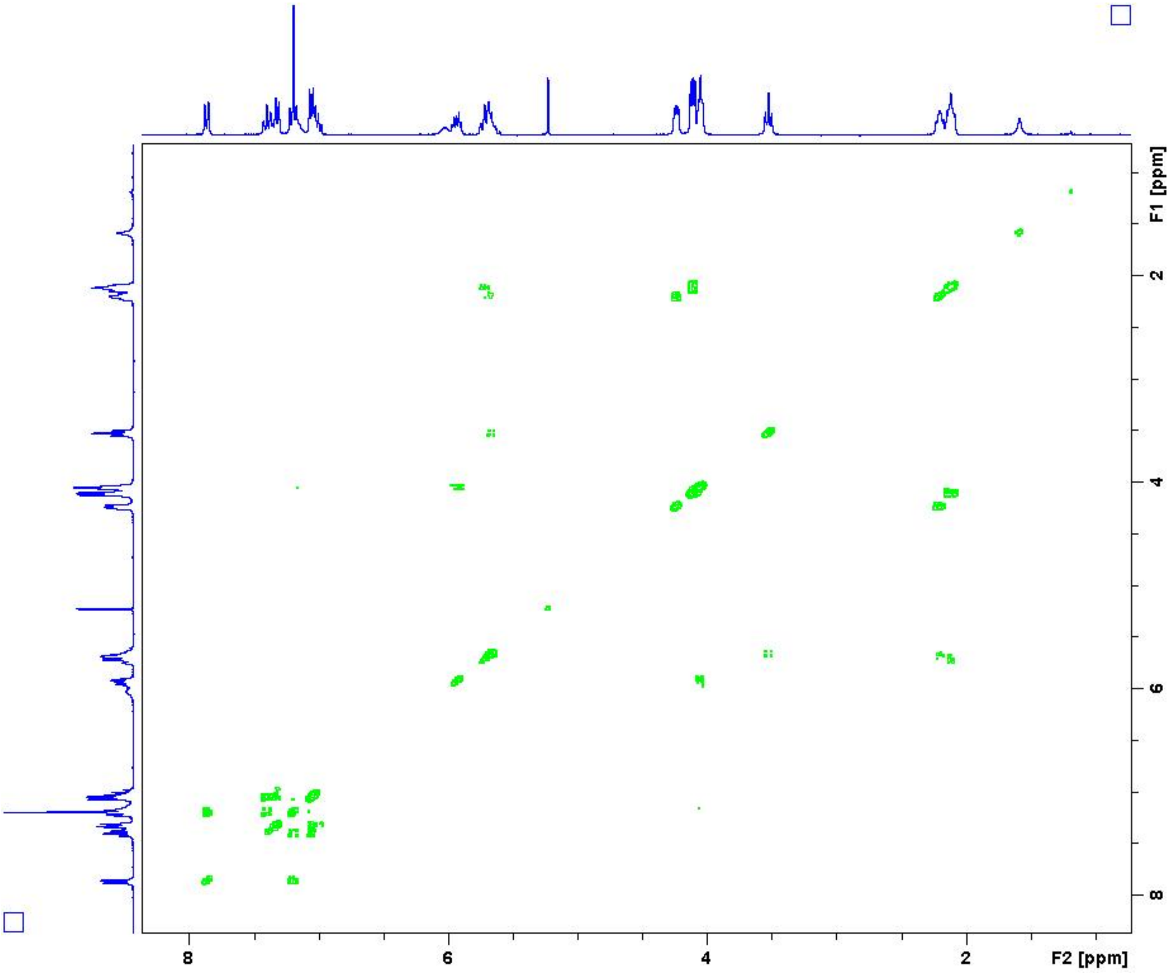


Figure 1: COSY

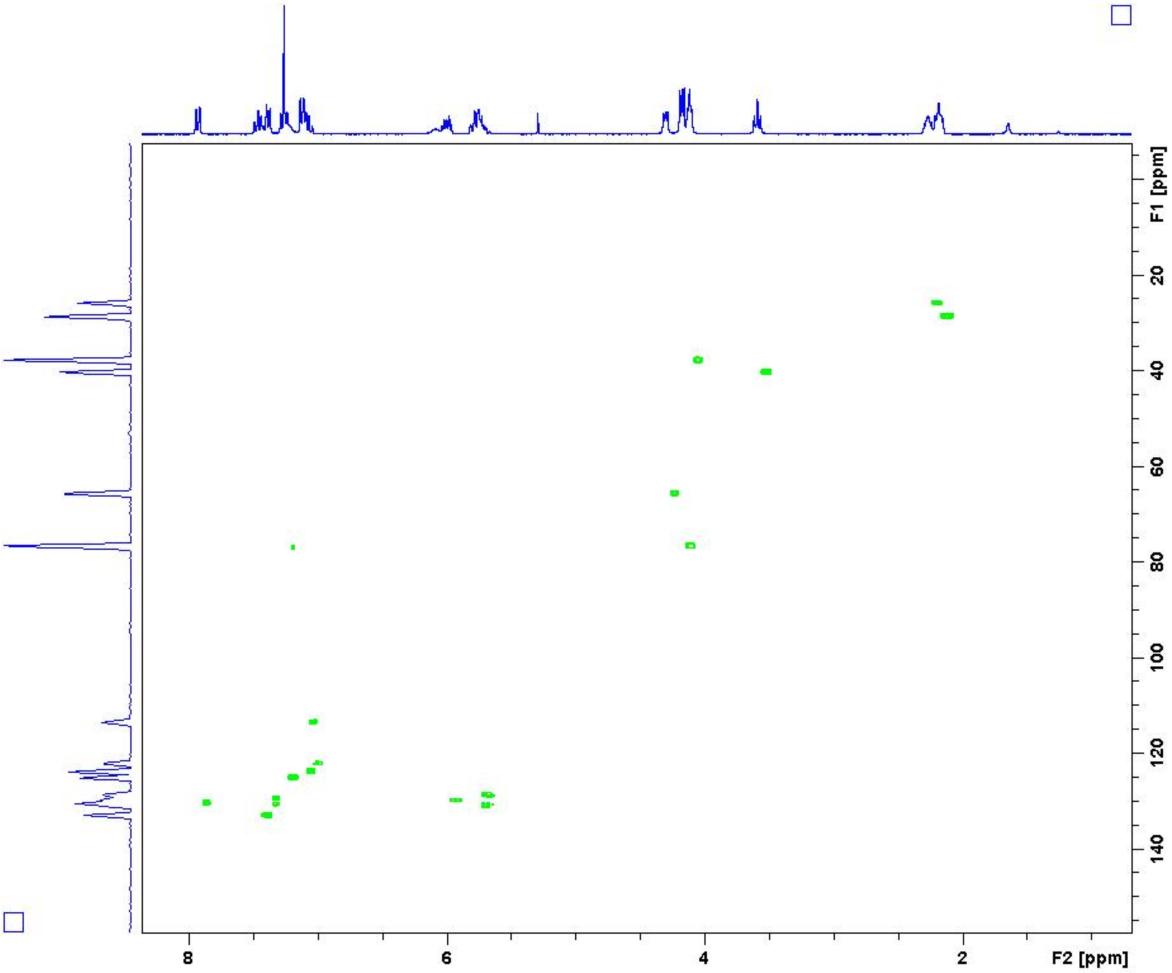
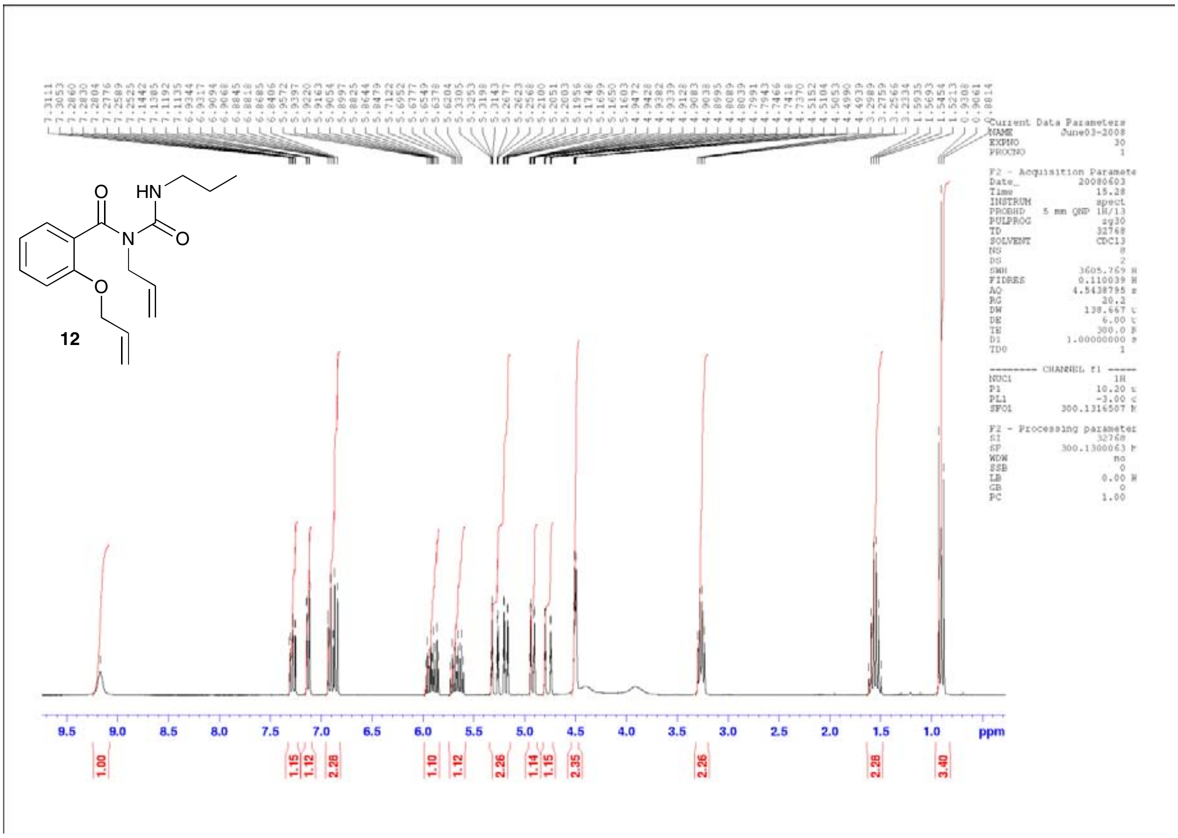
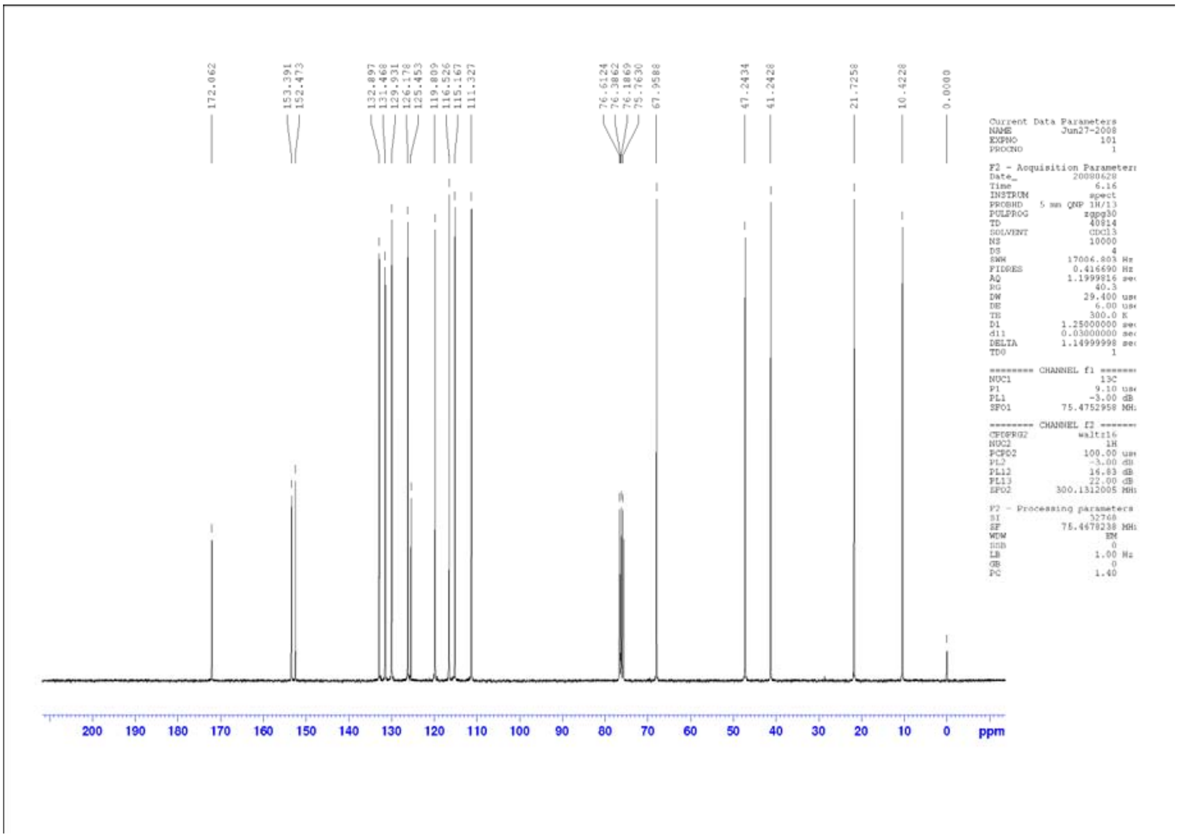
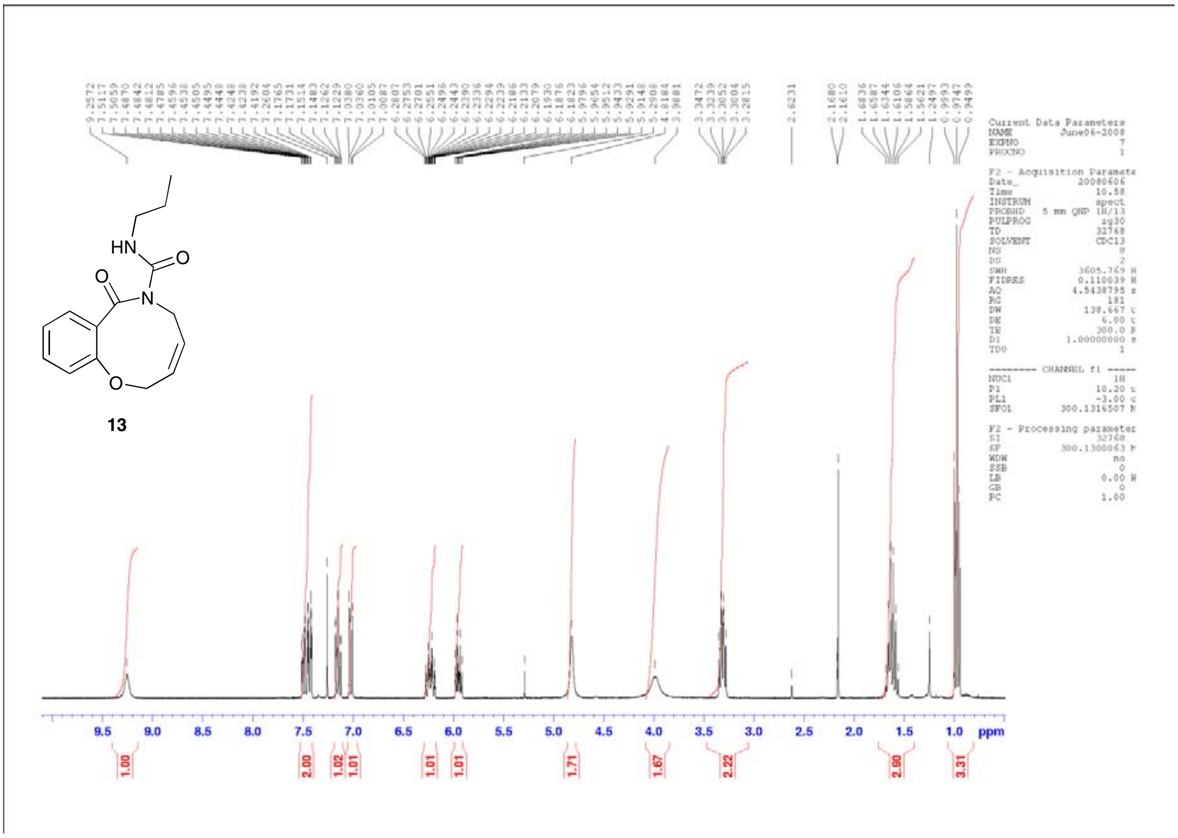
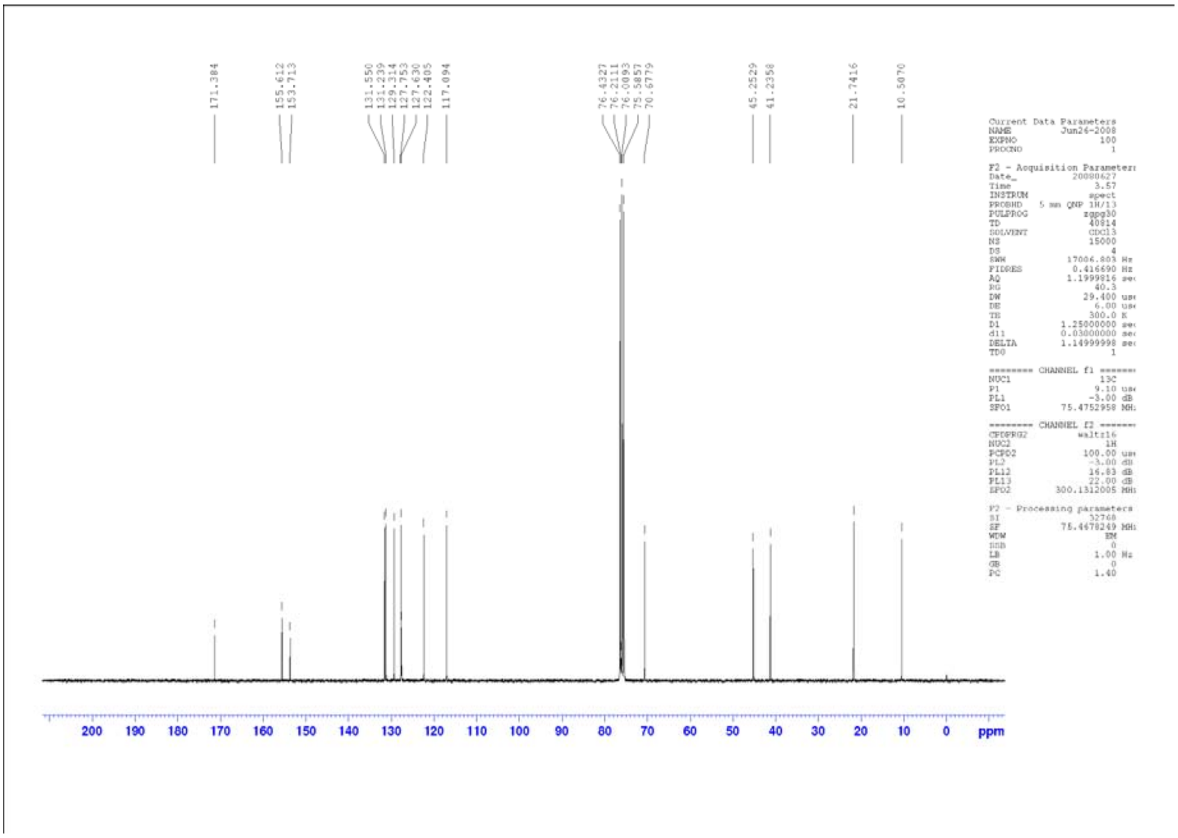


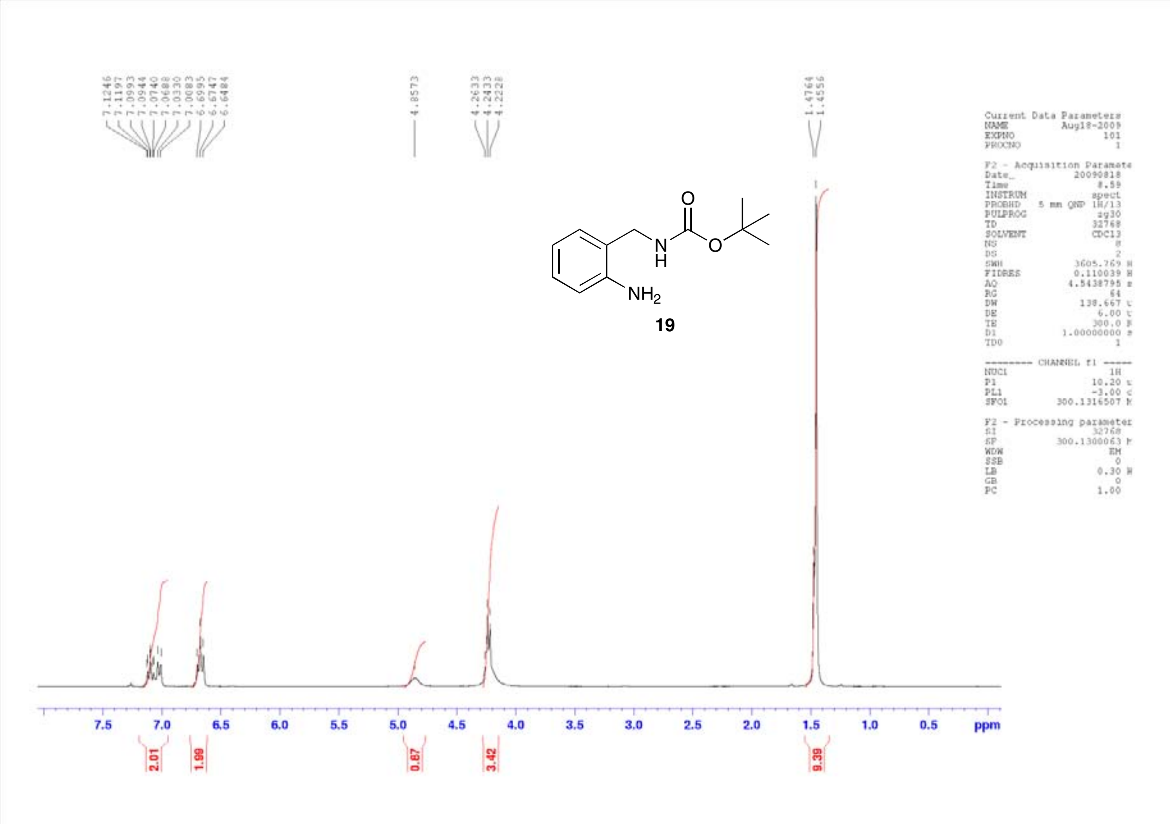
Figure 2: HMQC


12
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.00
1.15
1.12
2.28
1.10
1.12
2.26
1.14
1.15
2.35
2.26
2.28
3.40


172.062
153.391
152.473
132.897
131.468
129.931
126.178
125.453
119.809
116.526
115.167
111.327
76.6124
76.3862
76.1869
75.7630
67.9588
47.2434
41.2428
21.7258
10.4228
0.0000
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


9.2572
7.5117
7.5059
7.4870
7.4842
7.4812
7.4785
7.4596
7.4538
7.4505
7.4448
7.4248
7.4238
7.4192
7.2604
7.1765
7.1731
7.1514
7.1483
7.1262
7.1229
7.0380
7.0360
7.0105
7.0087
6.2807
6.2753
6.2701
6.2551
6.2496
6.2443
6.2390
6.2336
6.2294
6.2239
6.2186
6.2133
6.2079
6.1930
6.1876
6.1823
5.9796
5.9654
5.9512
5.9433
5.9291
5.9148
5.2988
4.8184
3.9881
3.3472
3.3239
3.3052
3.3004
3.2815
2.6231
2.1680
2.1610
1.6836
1.6587
1.6344
1.6106
1.5864
1.5621
1.2497
0.9993
0.9747
0.9499
HN
O
O
N
O
13
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.00
2.00
1.02
1.01
1.01
1.01
1.71
1.67
2.22
2.90
3.31
Current Data Parameters
NAME June06-2008
EXPNO 7
PROCNO 1
F2 - Acquisition Paramete
Date_ 20080606
Time 10.58
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 3605.769 H
FIDRES 0.110039 H
AQ 4.5438795 s
RG 181
DW 138.667 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.20 u
PL1 -3.00 d
SFO1 300.1316507 M
F2 - Processing parameter
SI 32768
SF 300.1300063 M
WDW no
SSB 0
LB 0.00 H
GB 0
PC 1.00


171.384
155.612
153.713
131.550
131.239
129.314
127.753
127.630
122.405
117.094
76.4327
76.2111
76.0093
75.5857
70.6779
45.2529
41.2358
21.7416
10.5070
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm
Current Data Parameters
NAME Jun26-2008
EXPNO 100
PROCNO 1
F2 - Acquisition Parameter
Date_ 20080627
Time 3.57
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 40814
SOLVENT CDCl3
NS 15000
DS 4
SWH 17006.803 Hz
FIDRES 0.416690 Hz
AQ 1.1999816 sec
RG 40.3
DW 29.400 use
DE 6.00 use
TE 300.0 K
D1 1.25000000 sec
d11 0.03000000 sec
DELTA 1.14999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.10 use
PL1 -3.00 dB
SFO1 75.4752958 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 use
PL2 -3.00 dB
PL12 14.83 dB
PL13 22.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4678249 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



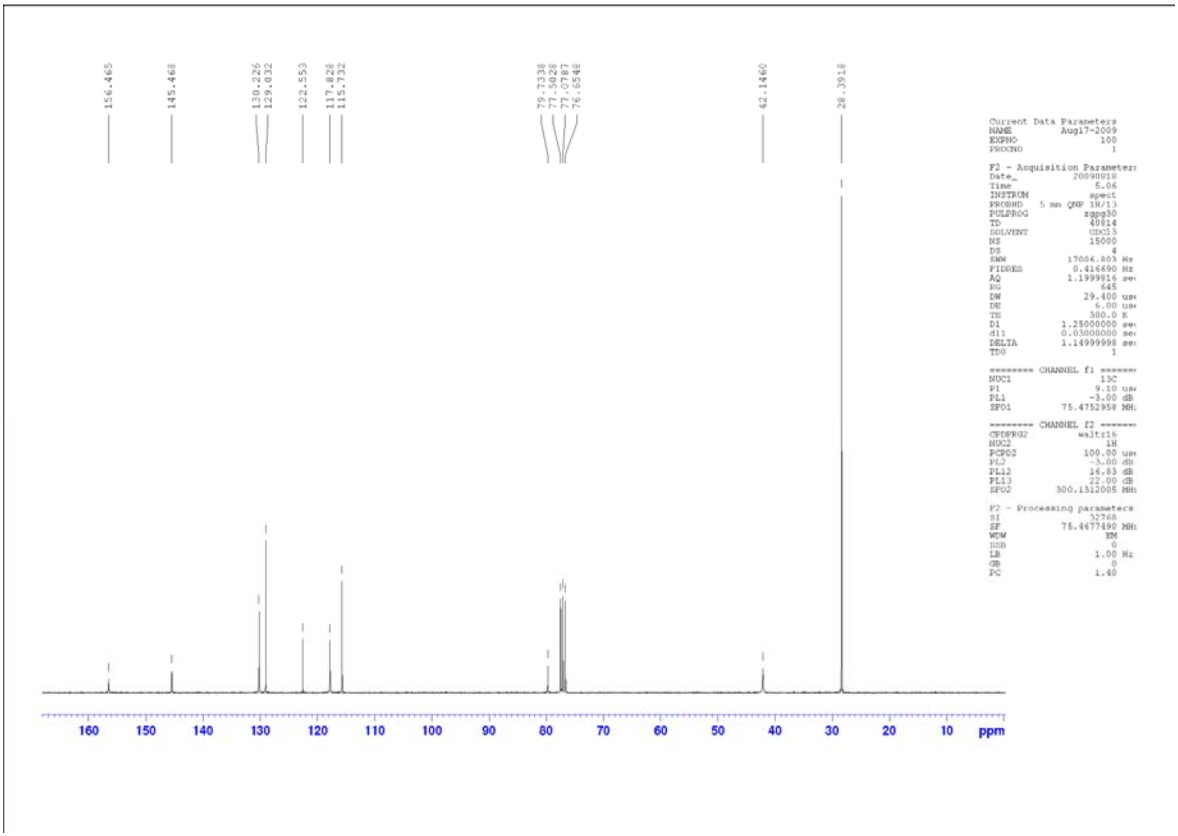
19

```
Current Data Parameters
NAME          Aug18-2009
EXPNO                101
PROCNO                 1

F2 - Acquisition Paramete
Date_           20090818
Time                8.59
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             3605.769 H
FIDRES          0.110039 H
AQ             4.5438795 s
RG                    64
DW               138.667 u
DE                  6.00 u
TE                 300.0 K
D1            1.00000000 s
TD0                    1

======== CHANNEL f1 =====
NUC1                  1H
P1                 10.20 u
PL1                -3.00 d
SFO1         300.1316507 M

F2 - Processing parameter
SI                 32768
SF           300.1300063 M
WDW                   EM
SSB                    0
LB                  0.30 H
GB                     0
PC                  1.00
```



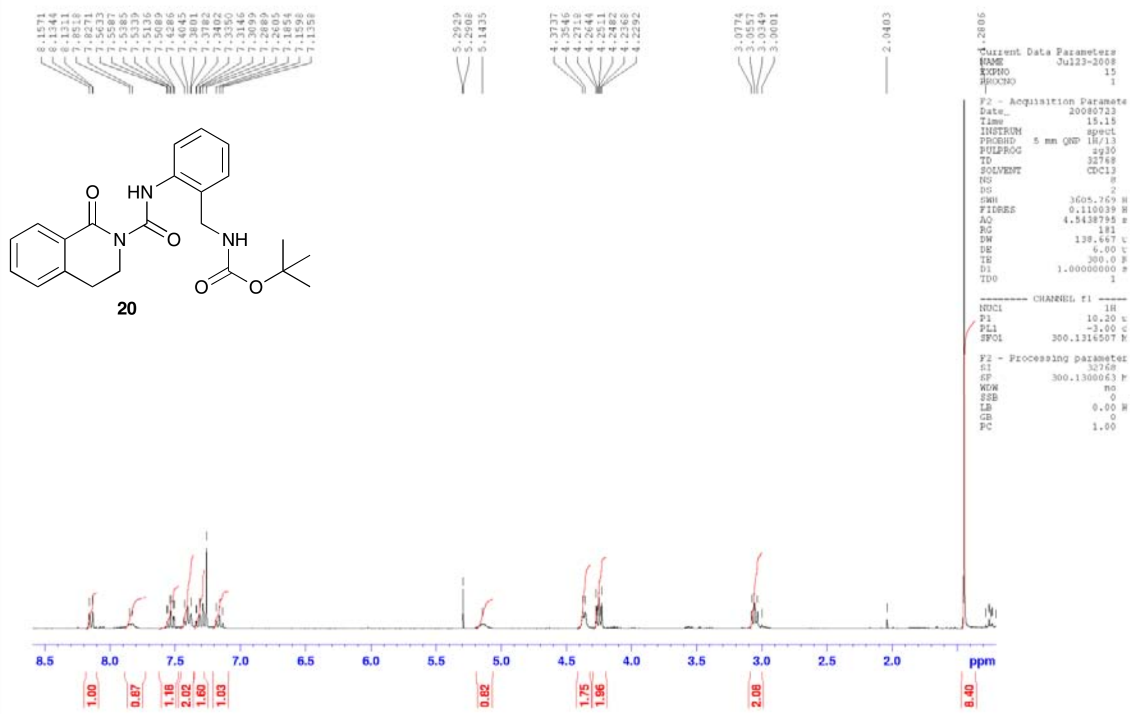
```
Current Data Parameters
NAME          Aug17-2009
EXPNO                100
PROCNO                 1

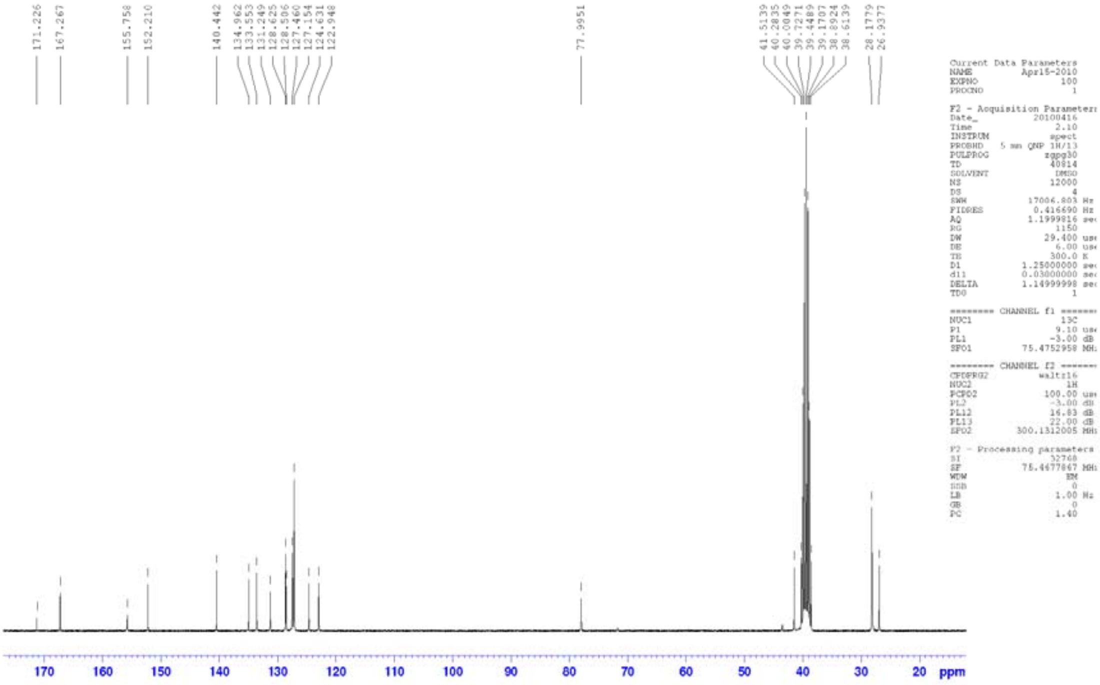
F2 - Acquisition Parameter
Date_           20090818
Time                5.06
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                 40814
SOLVENT            CDCl3
NS                 15000
DS                     4
SWH            17006.803 Hz
FIDRES          0.416690 Hz
AQ             1.1999816 se
RG                   645
DW                29.400 us
DE                  6.00 us
TE                 300.0 K
D1            1.25000000 se
d11           0.03000000 se
DELTA         1.14999998 se
TD0                    1

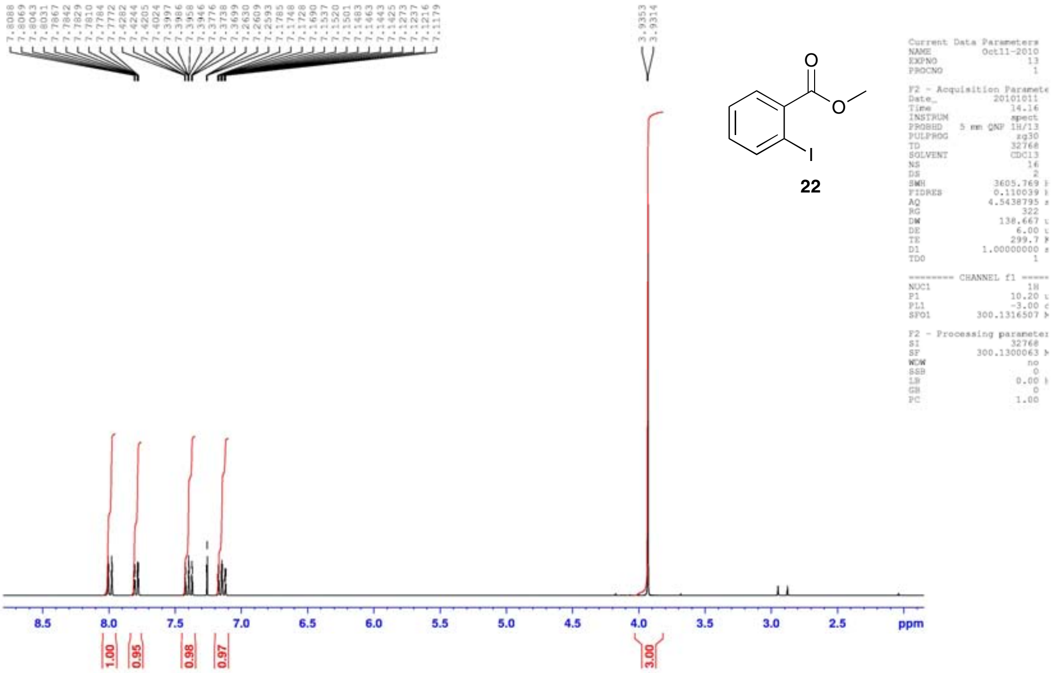
======== CHANNEL f1 ======
NUC1                 13C
P1                  9.10 us
PL1                -3.00 dB
SFO1          75.4752958 MH

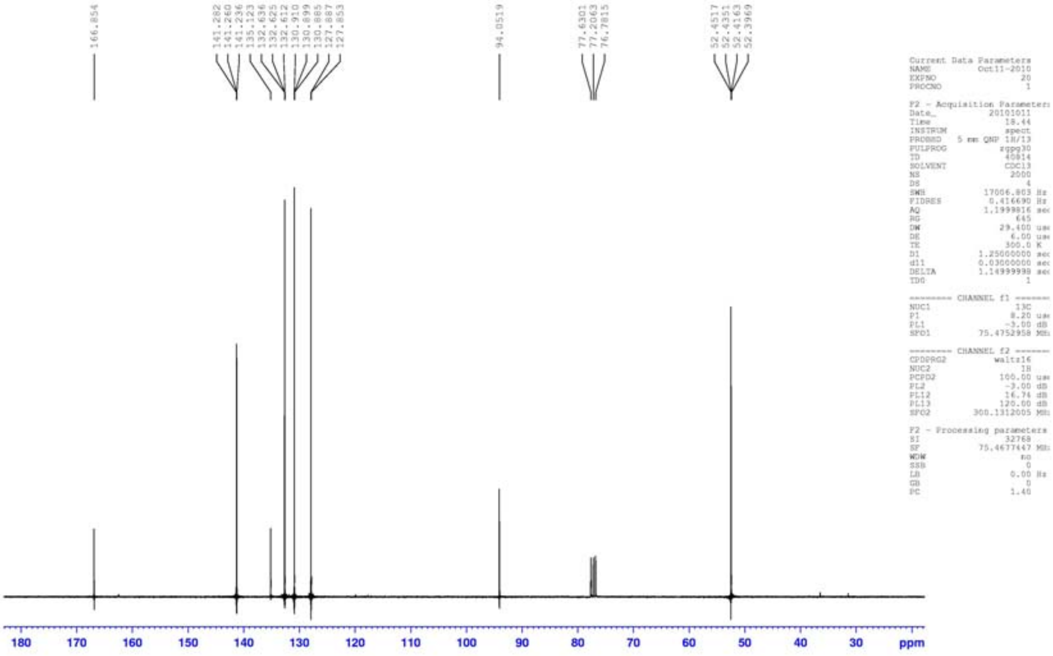
======== CHANNEL f2 ======
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 us
PL2                -3.00 dB
PL12               16.83 dB
PL13               22.00 dB
SFO2         300.1312005 MH

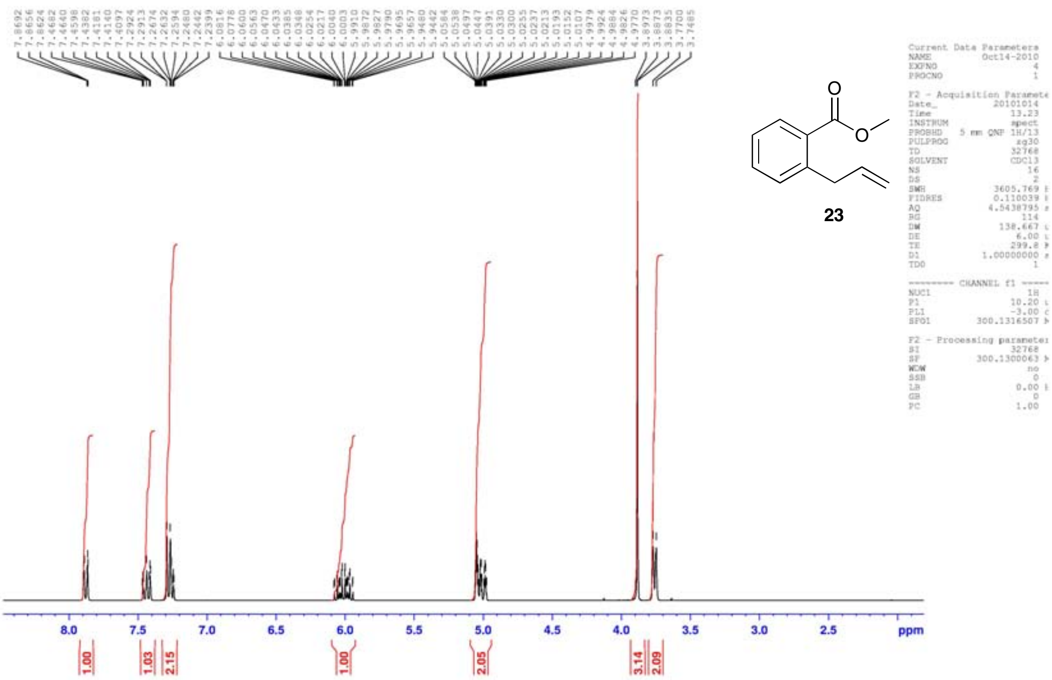
F2 - Processing parameters
SI                 32768
SF            75.4677490 MH
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```


8.1571
8.1344
8.1311
7.8518
7.8271
7.5633
7.5587
7.5385
7.5339
7.5136
7.5089
7.4286
7.4045
7.3801
7.3782
7.3402
7.3350
7.3146
7.3099
7.2889
7.2605
7.1854
7.1598
7.1358
5.2929
5.2908
5.1435
4.3737
4.3546
4.2719
4.2644
4.2511
4.2482
4.2368
4.2292
3.0774
3.0557
3.0349
3.0001
2.0403
1.2806
O
HN
N
O
NH
O
O
20
Current Data Parameters
NAME Jul23-2008
EXPNO 15
PROCNO 1
F2 - Acquisition Paramete
Date_ 20080723
Time 15.15
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 3605.769 H
FIDRES 0.110039 H
AQ 4.5438795 s
RG 181
DW 138.667 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 10.20 u
PL1 -3.00 d
SFO1 300.1316507 M
F2 - Processing parameter
SI 32768
SF 300.1300063 M
WDW no
SSB 0
LB 0.00 H
GB 0
PC 1.00
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
ppm
1.00
0.87
1.18
2.02
1.60
1.03
0.82
1.75
1.96
2.08
8.40

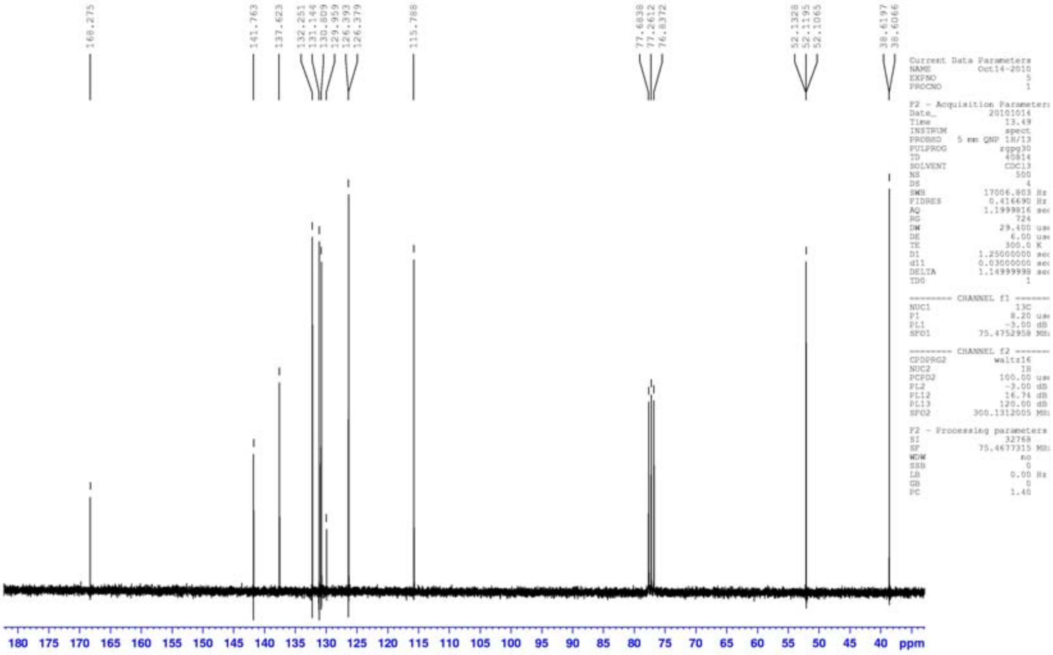

171.226
167.267
155.758
152.210
140.442
134.962
133.553
131.249
128.625
128.506
127.460
127.154
124.631
122.948
77.9951
41.5139
40.2835
40.0049
39.7271
39.4489
39.1707
38.8924
38.6139
28.1779
26.9377
Current Data Parameters
NAME Apr15-2010
EXPNO 100
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100416
Time 2.10
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 40814
SOLVENT DMSO
NS 12000
DS 4
SWH 17006.803 Hz
FIDRES 0.416690 Hz
AQ 1.1999816 sec
RG 1150
DW 29.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.25000000 sec
d11 0.03000000 sec
DELTA 1.14999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.10 usec
PL1 -3.00 dB
SFO1 75.4752958 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 16.83 dB
PL13 22.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4477867 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

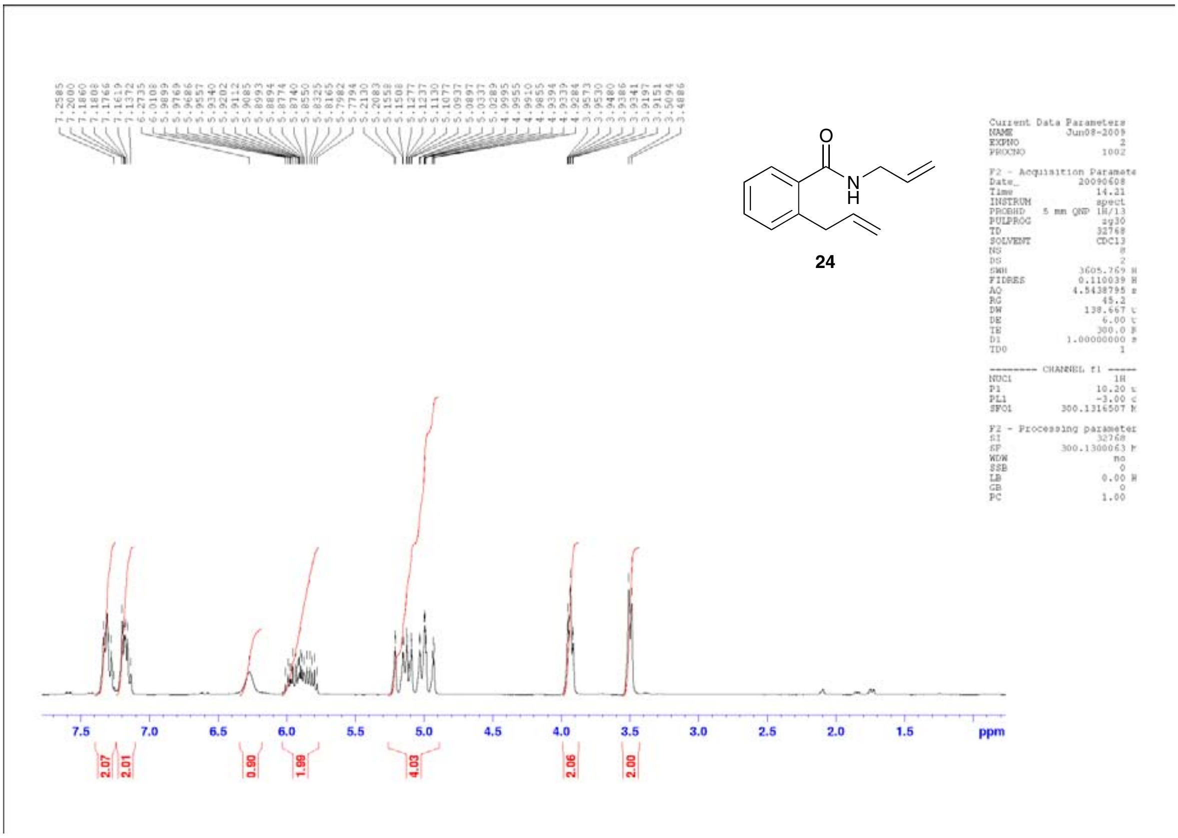

7.8088
7.8069
7.8043
7.8031
7.7867
7.7842
7.7829
7.7810
7.7784
7.7772
7.4282
7.4244
7.4205
7.4024
7.3997
7.3986
7.3958
7.3946
7.3776
7.3738
7.3699
7.2630
7.2609
7.2593
7.1785
7.1748
7.1728
7.1690
7.1537
7.1520
7.1501
7.1483
7.1463
7.1443
7.1425
7.1273
7.1237
7.1216
7.1179
3.9353
3.9314
O
O
I
22
Current Data Parameters
NAME Oct11-2010
EXPNO 13
PROCNO 1
F2 - Acquisition Paramete
Date_ 20101011
Time 14.16
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 3605.769
FIDRES 0.110039
AQ 4.5438795
RG 322
DW 138.667
DE 6.00
TE 299.7
D1 1.00000000
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 10.20
PL1 -3.00
SFO1 300.1316507
F2 - Processing paramete
SI 32768
SF 300.1300063
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00
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
ppm
1.00
0.95
0.98
0.97
3.00

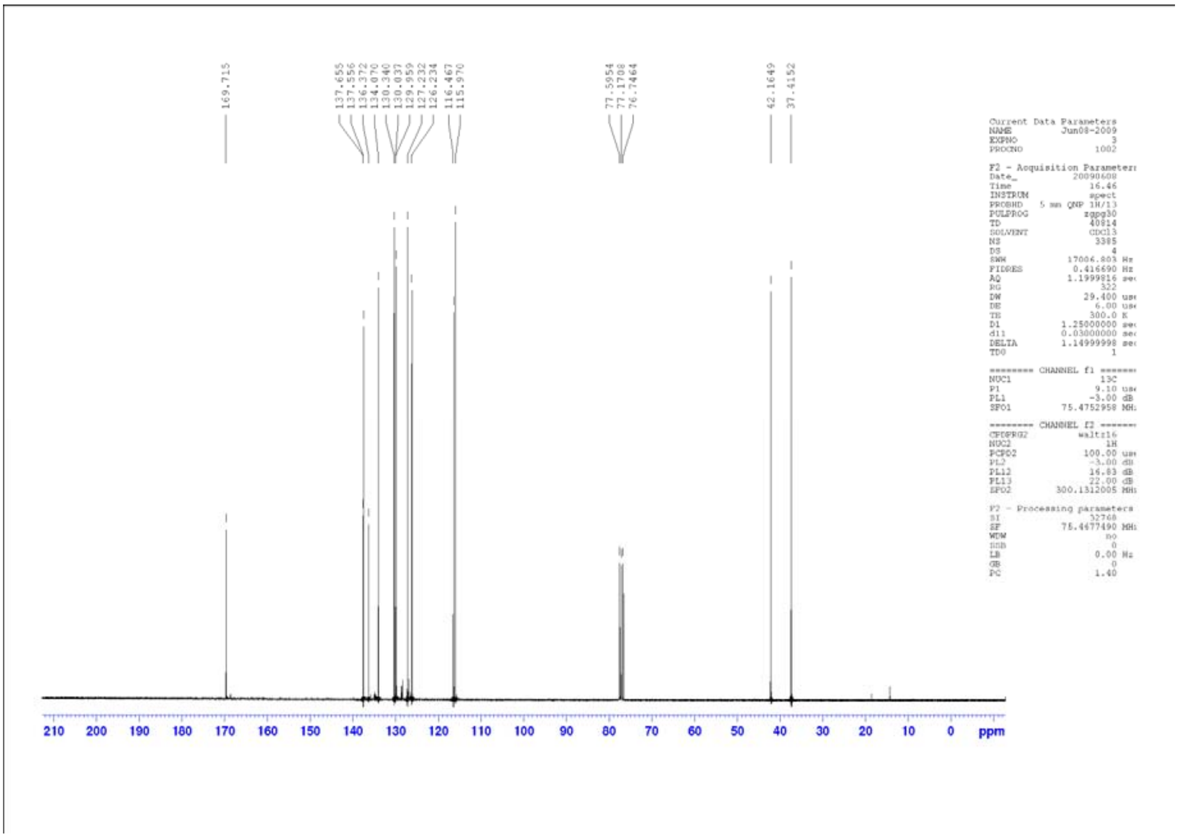

166.854
141.282
141.260
141.236
135.123
132.636
132.625
132.612
130.910
130.899
130.885
127.887
127.853
94.0519
77.6301
77.2063
76.7815
52.4517
52.4351
52.4163
52.3969
Current Data Parameters
NAME Oct11-2010
EXPNO 20
PROCNO 1
F2 - Acquisition Parameter
Date_ 20101011
Time 18.44
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 40814
SOLVENT CDCl3
NS 2000
DS 4
SWH 17006.803 Hz
FIDRES 0.416690 Hz
AQ 1.1999816 sec
RG 645
DW 29.400 use
DE 6.00 use
TE 300.0 K
D1 1.25000000 sec
d11 0.03000000 sec
DELTA 1.14999998 sec
TD0 1
======== CHANNEL f1 ======
NUC1 13C
P1 8.20 use
PL1 -3.00 dB
SFO1 75.4752958 MHz
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 use
PL2 -3.00 dB
PL12 16.74 dB
PL13 120.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677447 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

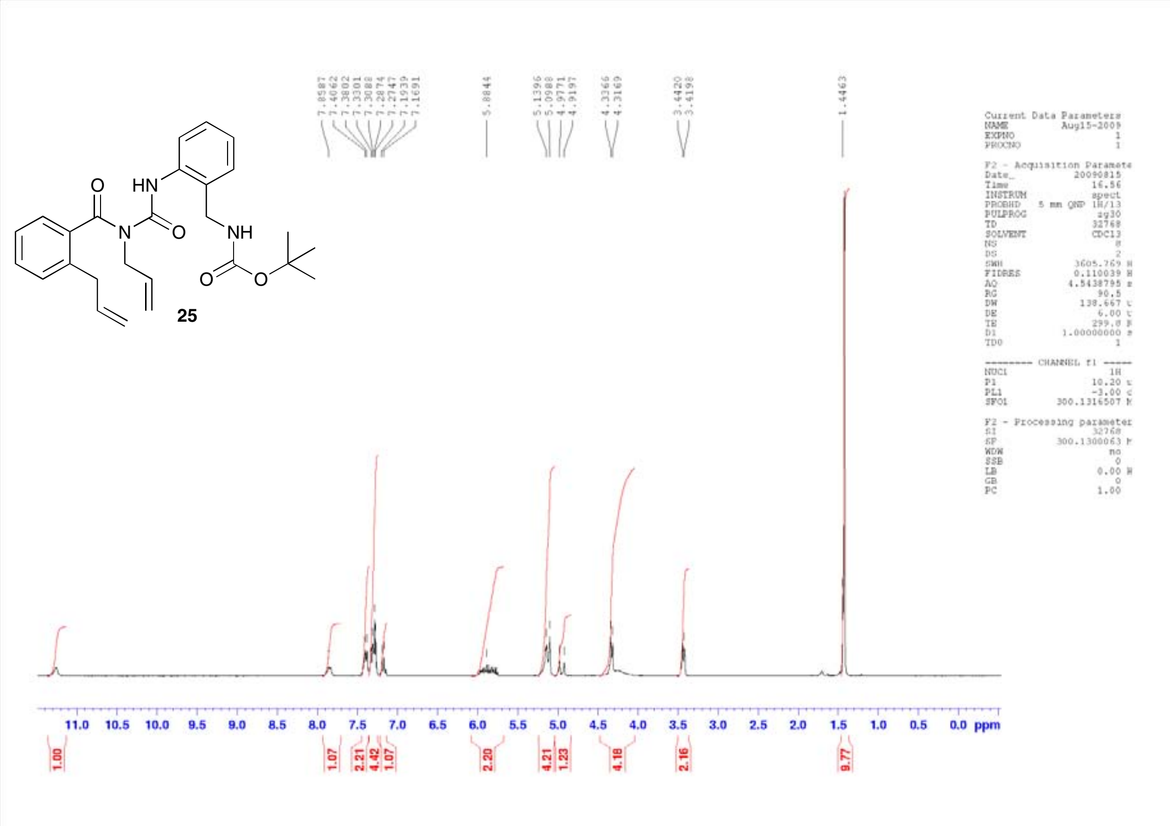

7.8692
7.8656
7.8624
7.4682
7.4640
7.4598
7.4382
7.4181
7.4140
7.4097
7.2924
7.2913
7.2674
7.2632
7.2594
7.2480
7.2442
7.2399
6.0816
6.0778
6.0600
6.0563
6.0470
6.0433
6.0385
6.0348
6.0254
6.0217
6.0040
6.0003
5.9910
5.9872
5.9827
5.9790
5.9695
5.9657
5.9480
5.9442
5.0584
5.0538
5.0497
5.0447
5.0391
5.0330
5.0300
5.0255
5.0237
5.0213
5.0193
5.0152
5.0107
4.9979
4.9924
4.9884
4.9826
4.9770
3.8973
3.8873
3.8835
3.7700
3.7485
O
O
Current Data Parameters
NAME Oct14-2010
EXPNO 4
PROCNO 1
F2 - Acquisition Paramete
Date_ 20101014
Time 13.23
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 3605.769
FIDRES 0.110039
AQ 4.5438795
RG 114
DW 138.667
DE 6.00
TE 299.8
D1 1.00000000
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 10.20
PL1 -3.00
SFO1 300.1316507
F2 - Processing paramete
SI 32768
SF 300.1300063
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
1.00
1.03
2.15
1.00
2.05
3.14
2.09

23


168.275
141.763
137.623
132.251
131.144
130.809
129.959
126.393
126.379
115.788
77.6838
77.2612
76.8372
52.1328
52.1195
52.1065
38.6197
38.6066
Current Data Parameters
NAME Oct14-2010
EXPNO 5
PROCNO 1
F2 - Acquisition Parameter
Date_ 20101014
Time 13.49
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 40814
SOLVENT CDCl3
NS 500
DS 4
SWH 17006.803 Hz
FIDRES 0.416690 Hz
AQ 1.1999816 sec
RG 724
DW 29.400 use
DE 6.00 use
TE 300.0 K
D1 1.25000000 sec
d11 0.03000000 sec
DELTA 1.14999998 sec
TD0 1
======== CHANNEL f1 ======
NUC1 13C
P1 8.20 use
PL1 -3.00 dB
SFO1 75.4752958 MHz
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 use
PL2 -3.00 dB
PL12 16.74 dB
PL13 120.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677315 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
180
175
170
165
160
155
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
40
ppm


24
7.2585
7.2000
7.1860
7.1808
7.1766
7.1619
7.1372
6.2735
6.0108
5.9899
5.9769
5.9686
5.9557
5.9340
5.9202
5.9112
5.9085
5.8993
5.8894
5.8774
5.8740
5.8550
5.8325
5.8165
5.7982
5.7794
5.2130
5.2083
5.1558
5.1508
5.1277
5.1237
5.1130
5.1077
5.0937
5.0897
5.0337
5.0289
4.9995
4.9955
4.9910
4.9855
4.9394
4.9339
4.9284
3.9573
3.9530
3.9480
3.9386
3.9341
3.9197
3.9151
3.5094
3.4886
O
N
H
Current Data Parameters
NAME Jun08-2009
EXPNO 2
PROCNO 1002
F2 - Acquisition Paramete
Date_ 20090608
Time 14.21
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 3605.769 H
FIDRES 0.110039 H
AQ 4.5438795 s
RG 45.2
DW 138.667 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 10.20 u
PL1 -3.00 d
SFO1 300.1316507 M
F2 - Processing parameter
SI 32768
SF 300.1300063 M
WDW no
SSB 0
LB 0.00 H
GB 0
PC 1.00
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
ppm
2.07
2.01
0.90
1.99
4.03
2.06
2.00


169.715
137.655
137.556
136.372
134.070
130.340
130.037
129.959
127.232
126.234
116.467
115.970
77.5954
77.1708
76.7464
42.1649
37.4152
Current Data Parameters
NAME Jun08-2009
EXPNO 3
PROCNO 1002
F2 - Acquisition Parameters
Date_ 20090608
Time 16.46
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 40814
SOLVENT CDCl3
NS 3385
DS 4
SWH 17006.803 Hz
FIDRES 0.416690 Hz
AQ 1.1999816 sec
RG 322
DW 29.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.25000000 sec
d11 0.03000000 sec
DELTA 1.14999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.10 usec
PL1 -3.00 dB
SFO1 75.4752958 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 16.83 dB
PL13 22.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

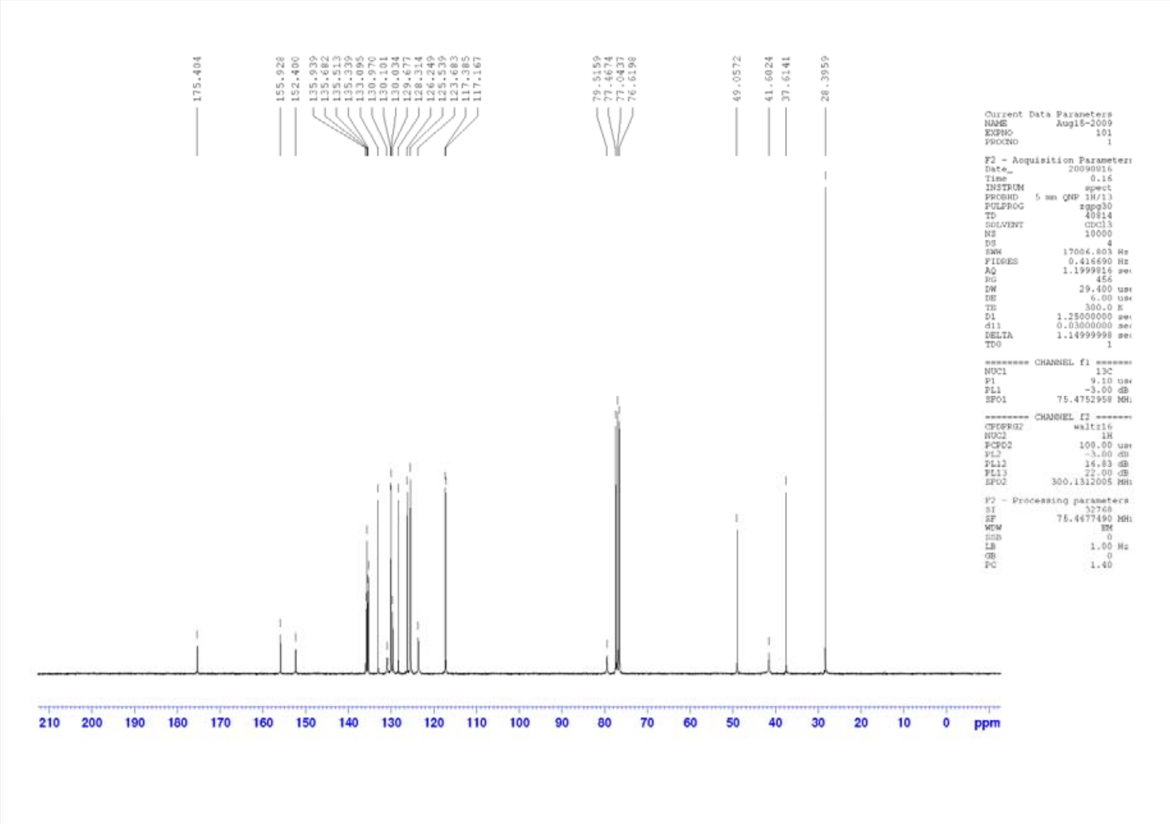


```
Current Data Parameters
NAME          Aug15-2009
EXPNO                  1
PROCNO                 1

F2 - Acquisition Paramete
Date_           20090815
Time               16.56
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             3605.769 H
FIDRES          0.110039 H
AQ             4.5438795 s
RG                  90.5
DW               138.667 u
DE                  6.00 u
TE                 299.8 K
D1            1.00000000 s
TD0                    1

======== CHANNEL f1 =====
NUC1                  1H
P1                 10.20 u
PL1                -3.00 d
SFO1         300.1316507 M

F2 - Processing parameter
SI                 32768
SF           300.1300063 M
WDW                   no
SSB                    0
LB                  0.00 H
GB                     0
PC                  1.00
```



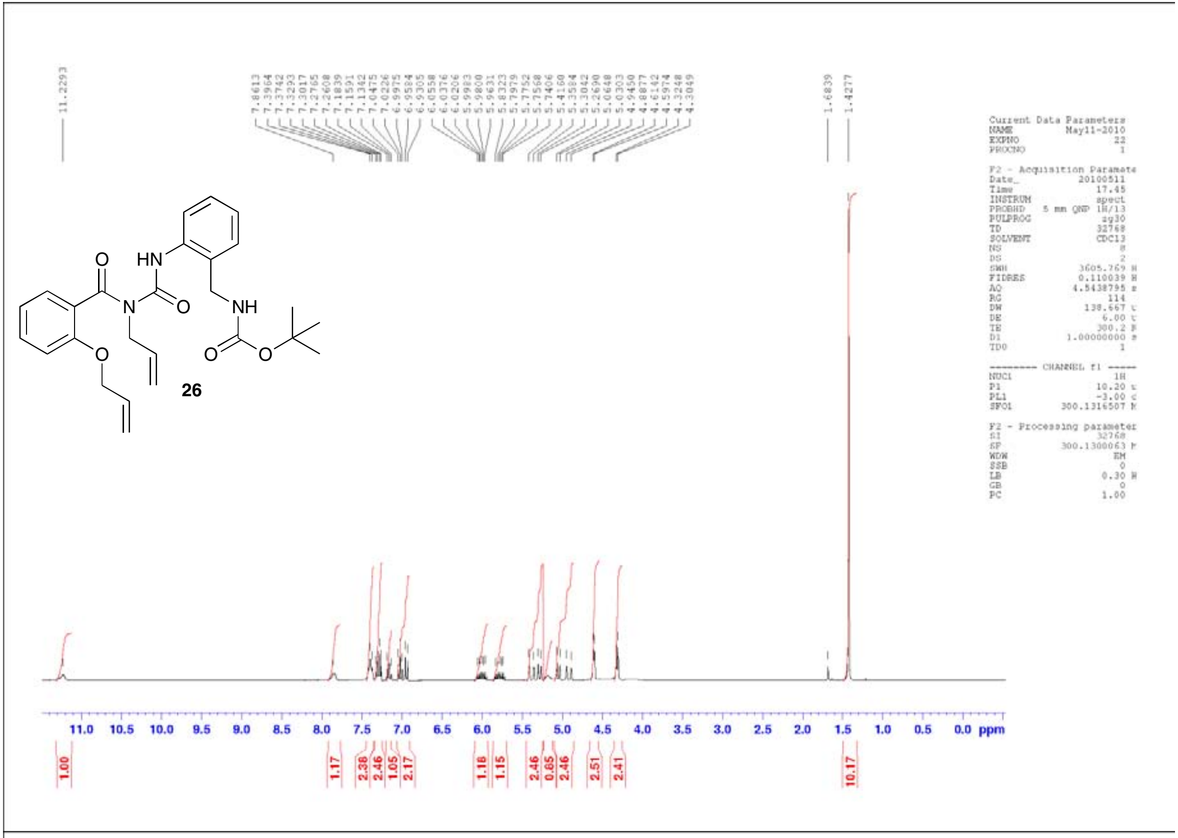
```
Current Data Parameters
NAME          Aug15-2009
EXPNO                101
PROCNO                 1

F2 - Acquisition Parameter
Date_           20090816
Time                0.16
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG           zgpg30
TD                 40814
SOLVENT            CDCl3
NS                 10000
DS                     4
SWH            17006.803 Hz
FIDRES          0.416690 Hz
AQ             1.1999816 sec
RG                   456
DW                29.400 use
DE                  6.00 use
TE                 300.0 K
D1            1.25000000 sec
d11           0.03000000 sec
DELTA         1.14999998 sec
TD0                    1

======== CHANNEL f1 =======
NUC1                 13C
P1                  9.10 use
PL1                -3.00 dB
SFO1          75.4752958 MH

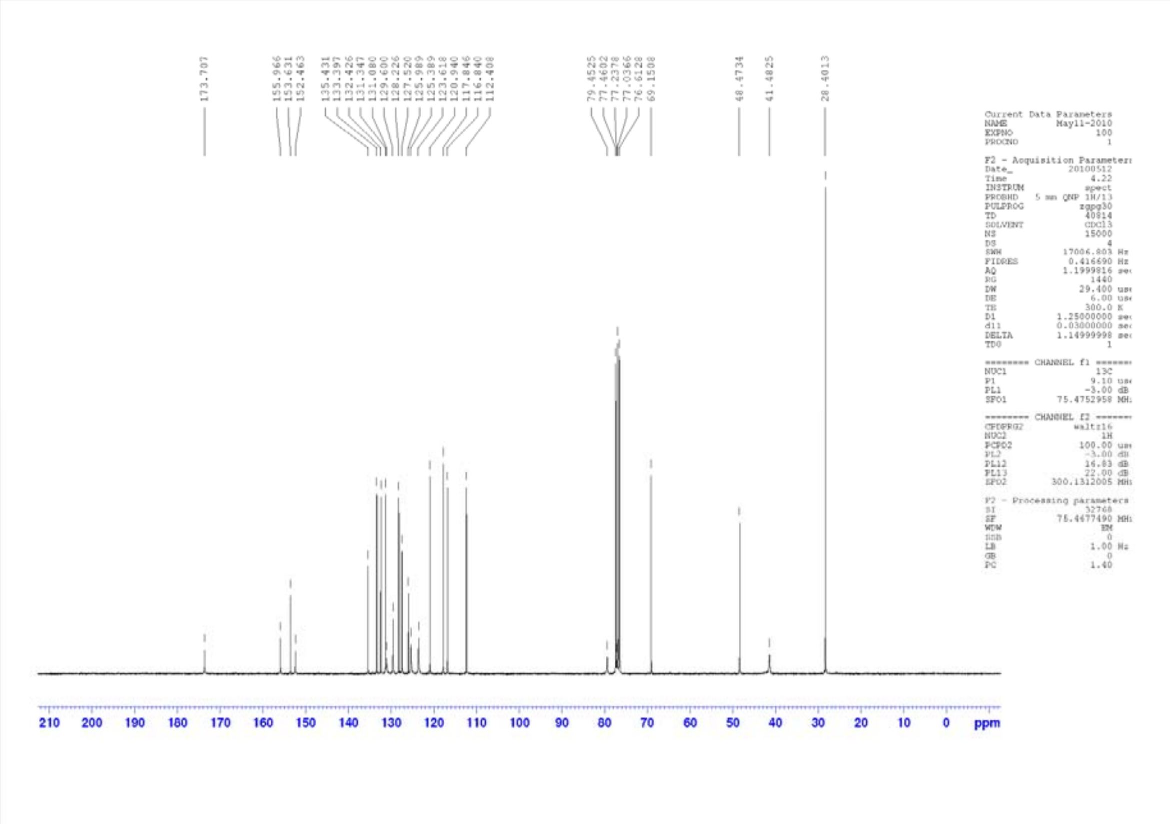
======== CHANNEL f2 =======
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 use
PL2                -3.00 dB
PL12               16.83 dB
PL13               22.00 dB
SFO2         300.1312005 MH

F2 - Processing parameters
SI                 32768
SF            75.4477490 MH
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

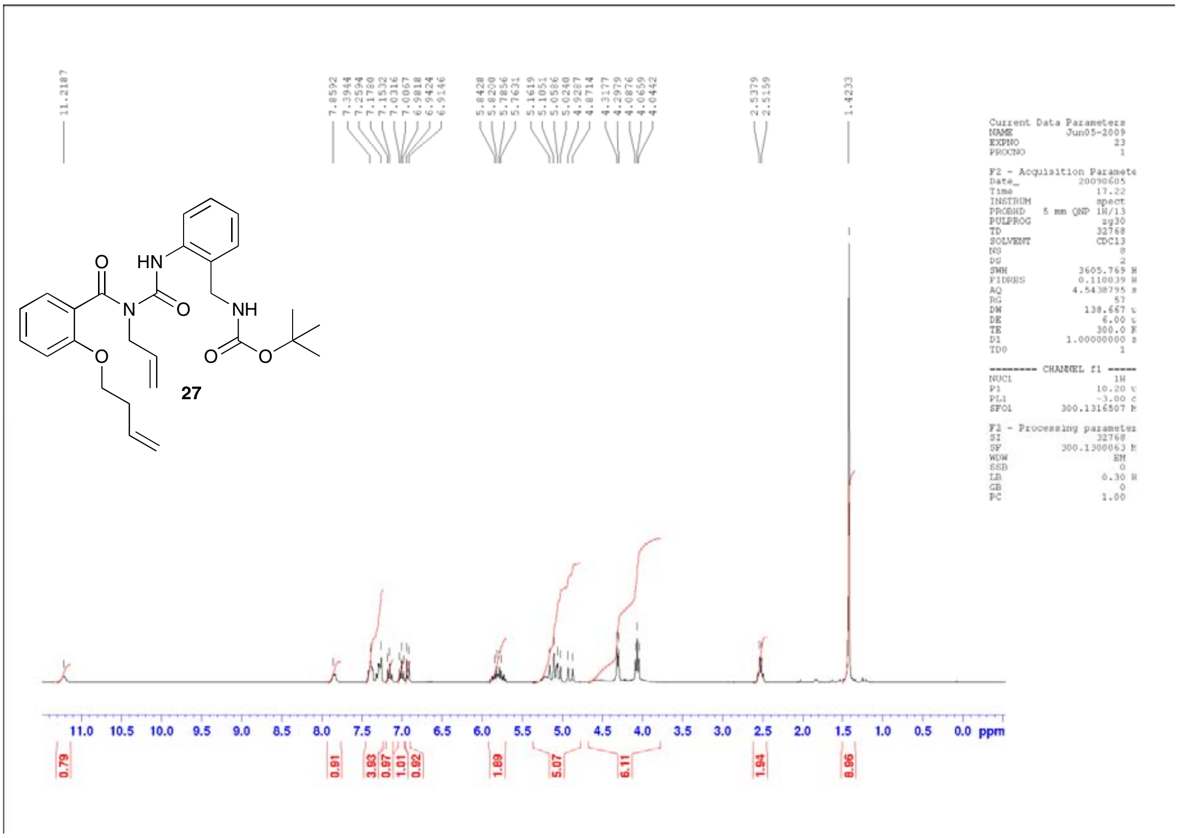


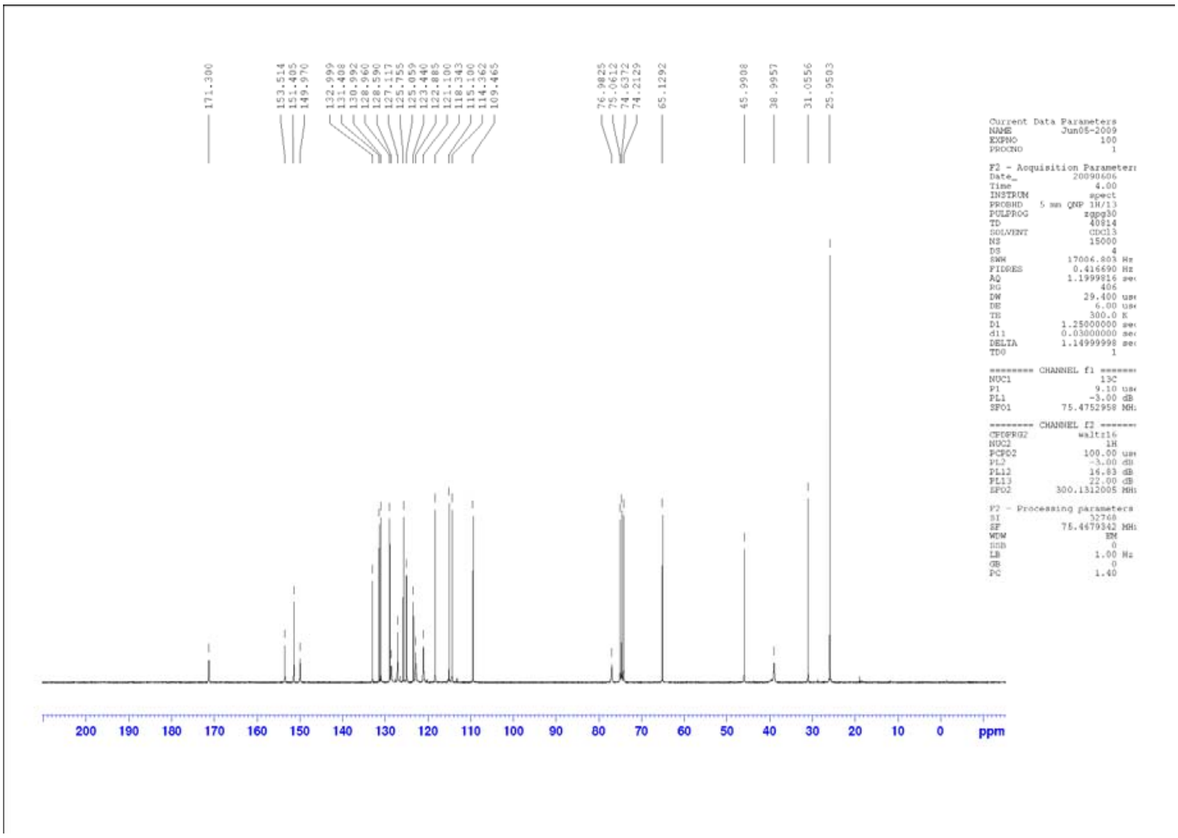
26

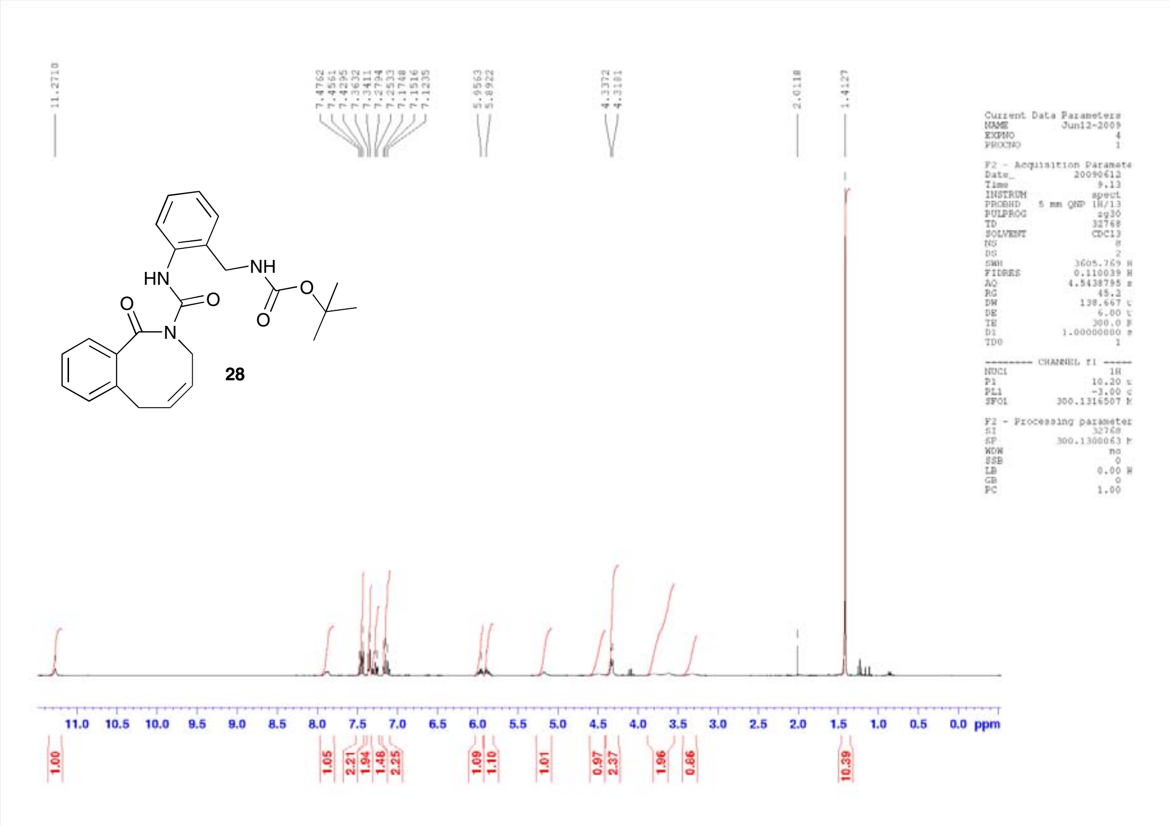
11.2293
7.8613
7.3964
7.3742
7.3523
7.3017
7.2765
7.2608
7.1839
7.1591
7.1342
7.0475
7.0226
6.9975
6.9584
6.9305
6.0558
6.0376
6.0206
5.9983
5.9800
5.9631
5.8323
5.7979
5.7758
5.7568
5.7486
5.4160
5.3584
5.3042
5.2690
5.0648
5.0353
4.9459
4.8877
4.6142
4.5974
4.3248
4.3049
1.6839
1.4277
O
HN
N
O
NH
O
O
O
Current Data Parameters
NAME May11-2010
EXPNO 22
PROCNO 1
F2 - Acquisition Paramete
Date_ 20100511
Time 17.45
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 3605.769 H
FIDRES 0.110039 H
AQ 4.5438795 s
RG 114
DW 138.667 u
DE 6.00 u
TE 300.2 K
D1 1.00000000 s
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 10.20 u
PL1 -3.00 d
SFO1 300.1314507 M
F2 - Processing parameter
SI 32768
SF 300.1300063 M
WDW EM
SSB 0
LB 0.30 H
GB 0
PC 1.00
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0 ppm
1.00
1.17
2.38
2.46
1.05
2.17
1.18
1.15
2.46
0.85
2.46
2.51
2.41
10.17



173.707
155.966
153.631
152.463
135.431
133.397
132.426
131.347
131.080
129.600
128.226
127.520
125.989
125.389
123.618
120.940
117.846
116.840
112.408
79.4525
77.4602
77.0378
76.6128
69.1508
48.4734
41.4825
28.4013
Current Data Parameters
NAME May11-2010
EXPNO 100
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100512
Time 4.22
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 40814
SOLVENT CDCl3
NS 15000
DS 4
SWH 17006.803 Hz
FIDRES 0.416690 Hz
AQ 1.1999916 sec
RG 1440
DW 29.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.25000000 sec
d11 0.03000000 sec
DELTA 1.14999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.10 usec
PL1 -3.00 dB
SFO1 75.4752958 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 16.83 dB
PL13 22.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


27
O
HN
N
O
NH
O
O
O
11.2187
7.8592
7.3944
7.2594
7.1780
7.1532
7.0316
7.0067
6.9818
6.9424
6.9146
5.8428
5.8200
5.7856
5.7631
5.1619
5.1051
5.0586
5.0240
4.9287
4.8714
4.3177
4.2979
4.0876
4.0659
4.0442
2.5379
2.5159
1.4233
Current Data Parameters
NAME Jun05-2009
EXPNO 23
PROCNO 1
F2 - Acquisition Paramete
Date_ 20090605
Time 17.22
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 3605.769 H
FIDRES 0.110039 H
AQ 4.5438795 s
RG 57
DW 138.667 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 10.20 u
PL1 -3.00 d
SFO1 300.1316507 M
F2 - Processing parameter
SI 32768
SF 300.1300063 M
WDW EM
SSB 0
LB 0.30 H
GB 0
PC 1.00
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.79
0.91
3.83
0.97
1.01
0.92
1.89
5.07
6.11
1.94
8.96


171.300
153.514
151.405
149.970
132.999
131.408
130.992
128.960
128.590
127.117
125.755
125.059
123.440
122.885
121.100
118.343
115.100
114.362
109.465
76.9825
75.0612
74.6372
74.2129
65.1292
45.9908
38.9957
31.0556
25.9503
Current Data Parameters
NAME Jun05-2009
EXPNO 100
PROCNO 1
F2 - Acquisition Parameter
Date_ 20090606
Time 4.00
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 40814
SOLVENT CDCl3
NS 15000
DS 4
SWH 17006.803 Hz
FIDRES 0.416690 Hz
AQ 1.1999816 se
RG 406
DW 29.400 us
DE 6.00 us
TE 300.0 K
D1 1.25000000 se
d11 0.03000000 se
DELTA 1.14999998 se
TD0 1
======== CHANNEL f1 =======
NUC1 13C
P1 9.10 us
PL1 -3.00 dB
SFO1 75.4752958 MH
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 us
PL2 -3.00 dB
PL12 16.83 dB
PL13 22.00 dB
SFO2 300.1312005 MH
F2 - Processing parameters
SI 32768
SF 75.4679342 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

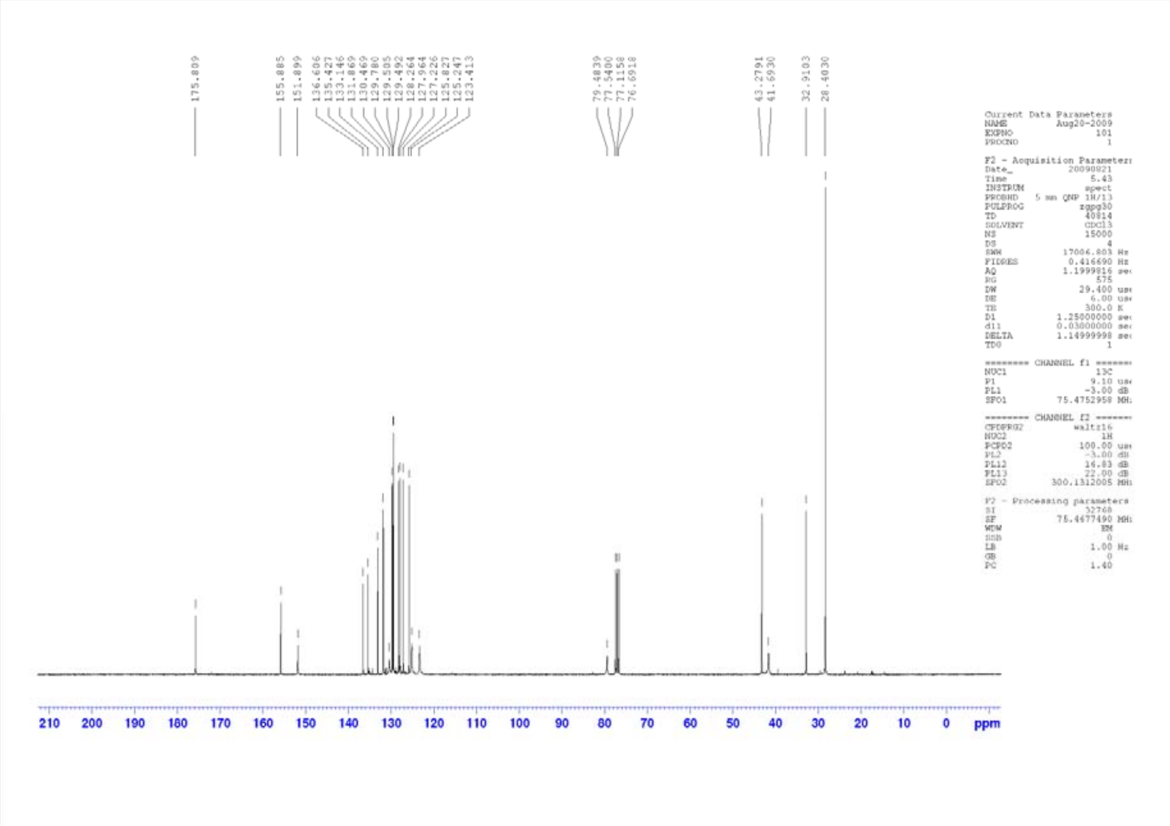


```
Current Data Parameters
NAME          Jun12-2009
EXPNO                  4
PROCNO                 1

F2 - Acquisition Paramete
Date_           20090612
Time                9.13
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             3605.769 H
FIDRES          0.110039 H
AQ             4.5438795 s
RG                  45.2
DW               138.667 u
DE                  6.00 u
TE                 300.0 K
D1            1.00000000 s
TD0                    1

======== CHANNEL f1 =====
NUC1                  1H
P1                 10.20 u
PL1                -3.00 d
SFO1         300.1316507 M

F2 - Processing parameter
SI                 32768
SF           300.1300063 M
WDW                   no
SSB                    0
LB                  0.00 H
GB                     0
PC                  1.00
```



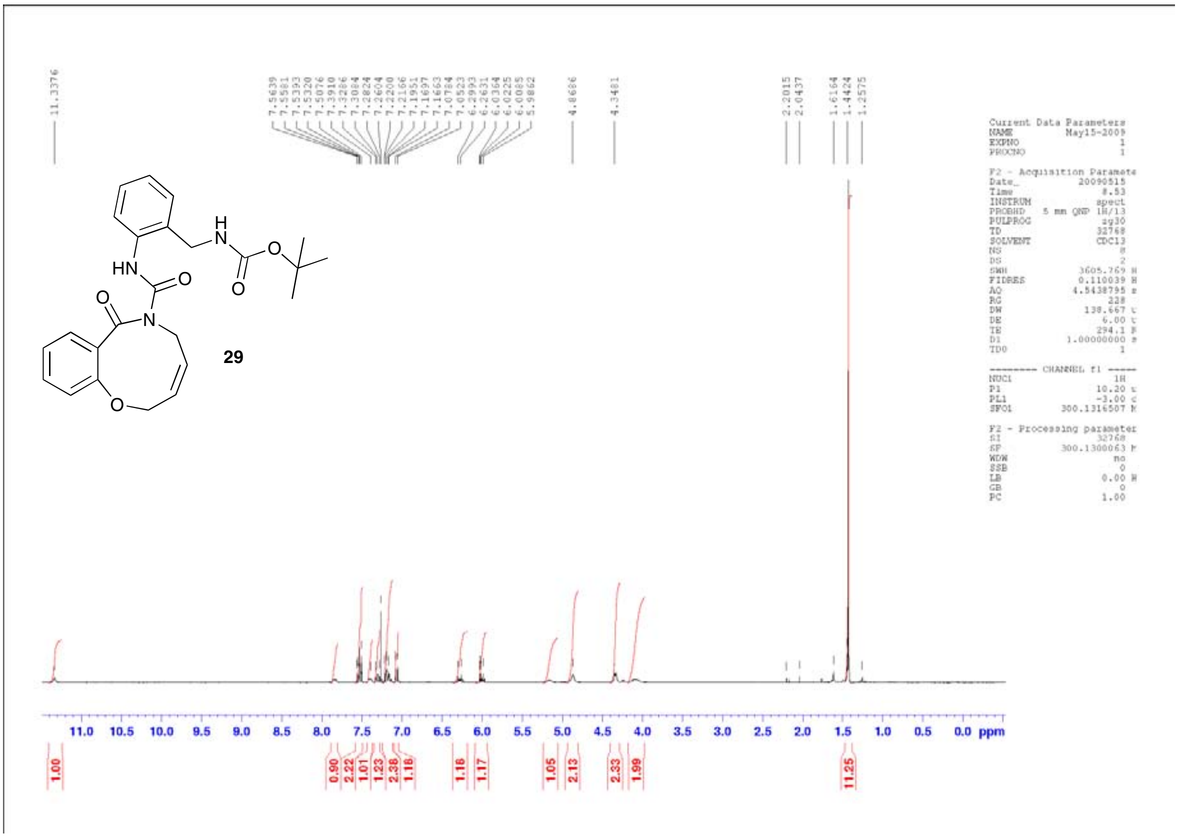
```
Current Data Parameters
NAME          Aug20-2009
EXPNO                101
PROCNO                 1

F2 - Acquisition Parameter
Date_           20090821
Time                5.43
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                 40814
SOLVENT            CDCl3
NS                 15000
DS                     4
SWH            17006.803 Hz
FIDRES          0.416690 Hz
AQ             1.1999816 sec
RG                   575
DW                29.400 use
DE                  6.00 use
TE                 300.0 K
D1            1.25000000 sec
d11           0.03000000 sec
DELTA         1.14999998 sec
TD0                    1

======== CHANNEL f1 =======
NUC1                 13C
P1                  9.10 use
PL1                -3.00 dB
SFO1          75.4752958 MH

======== CHANNEL f2 =======
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 use
PL2                -3.00 dB
PL12               16.83 dB
PL13               22.00 dB
SFO2         300.1312005 MH

F2 - Processing parameters
SI                 32768
SF            75.4677490 MH
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

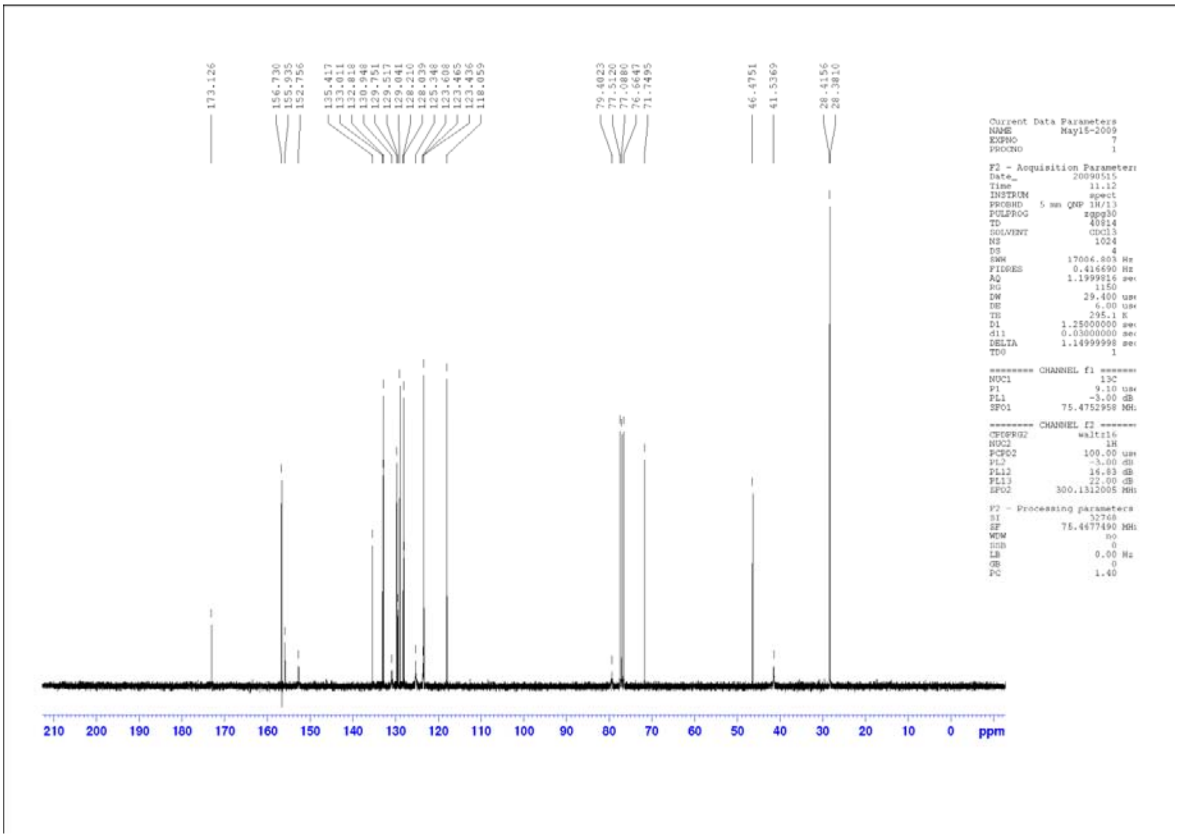


```
Current Data Parameters
NAME         May15-2009
EXPNO                 1
PROCNO                1

F2 - Acquisition Paramete
Date_          20090515
Time               8.53
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            3605.769 H
FIDRES         0.110039 H
AQ            4.5438795 s
RG                  228
DW              138.667 u
DE                 6.00 u
TE                294.1 K
D1           1.00000000 s
TD0                   1

======== CHANNEL f1 =====
NUC1                 1H
P1                10.20 u
PL1               -3.00 d
SFO1        300.1316507 M

F2 - Processing parameter
SI                32768
SF          300.1300063 M
WDW                  no
SSB                   0
LB                 0.00 H
GB                    0
PC                 1.00
```



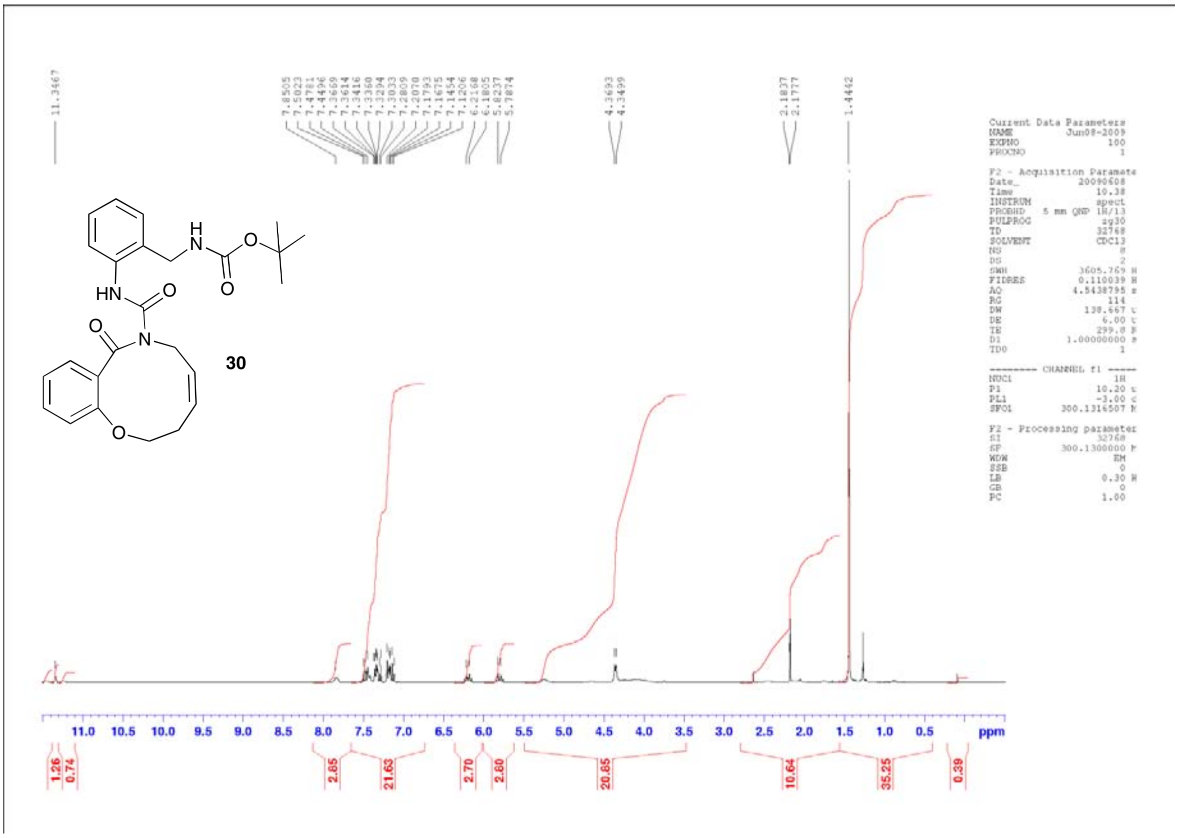
```
Current Data Parameters
NAME         May15-2009
EXPNO                 7
PROCNO                1

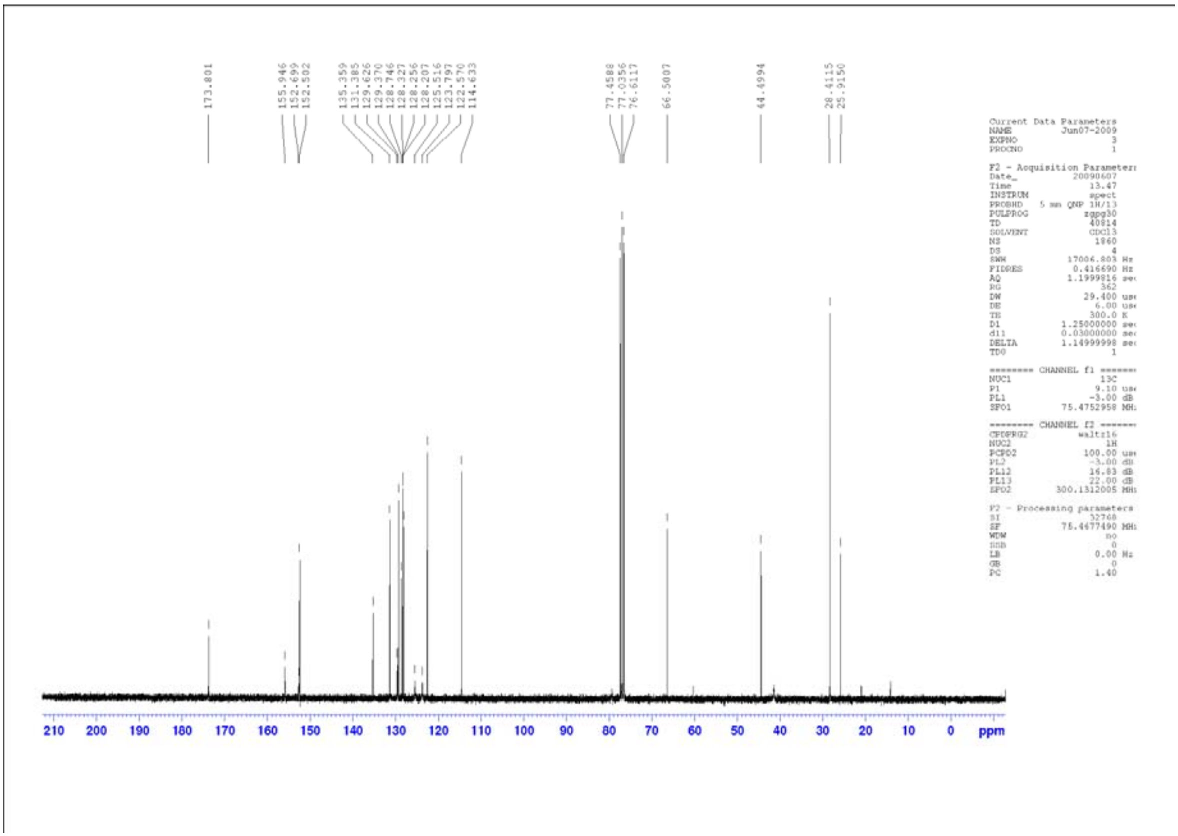
F2 - Acquisition Parameter
Date_          20090515
Time              11.12
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                40814
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           17006.803 Hz
FIDRES         0.416690 Hz
AQ            1.1999816 sec
RG                 1150
DW               29.400 use
DE                 6.00 use
TE                295.1 K
D1           1.25000000 sec
d11          0.03000000 sec
DELTA        1.14999998 sec
TD0                   1

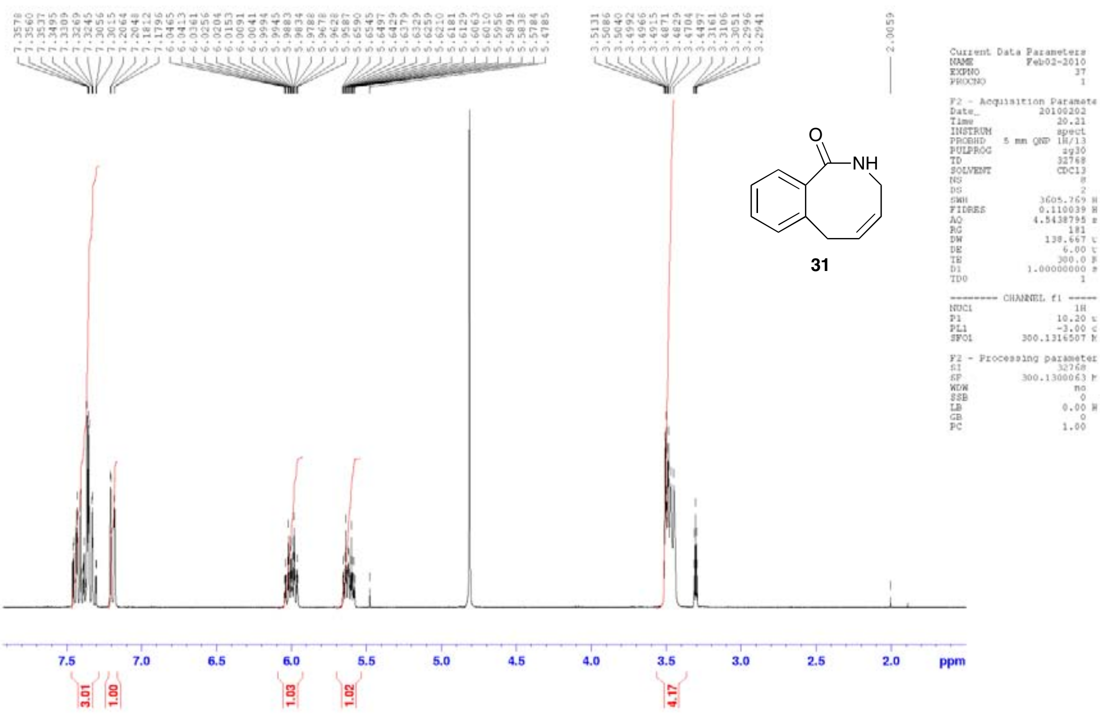
======== CHANNEL f1 ======
NUC1                13C
P1                 9.10 use
PL1               -3.00 dB
SFO1         75.4752958 MH

======== CHANNEL f2 ======
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 use
PL2               -3.00 dB
PL12              16.83 dB
PL13              22.00 dB
SFO2        300.1312005 MH

F2 - Processing parameters
SI                32768
SF           75.4477490 MH
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.40
```


30
11.3467
7.8505
7.5023
7.4781
7.4496
7.3669
7.3614
7.3416
7.3360
7.3294
7.3033
7.2809
7.2078
7.1793
7.1675
7.1454
7.1206
6.2168
6.1805
5.8237
5.7874
4.3693
4.3499
2.1837
2.1777
1.4442
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.26
0.74
2.85
21.63
2.70
2.60
20.85
10.64
35.25
0.39
Current Data Parameters
NAME Jun08-2009
EXPNO 100
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090608
Time 10.38
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 3605.769 Hz
FIDRES 0.110039 Hz
AQ 4.5438795 sec
RG 114
DW 138.667 usec
DE 6.00 usec
TE 299.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 10.20 usec
PL1 -3.00 dB
SFO1 300.1314507 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


173.801
155.946
152.699
152.502
135.359
131.385
129.626
129.370
128.746
128.327
128.256
128.207
125.516
123.797
122.570
114.633
77.4588
77.0356
76.6117
66.5007
44.4994
28.4115
25.9150
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
Current Data Parameters
NAME Jun07-2009
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090607
Time 13.47
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 40814
SOLVENT CDCl3
NS 1860
DS 4
SWH 17006.803 Hz
FIDRES 0.416690 Hz
AQ 1.1999816 sec
RG 362
DW 29.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.25000000 sec
d11 0.03000000 sec
DELTA 1.14999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.10 usec
PL1 -3.00 dB
SFO1 75.4752958 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 16.83 dB
PL13 22.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

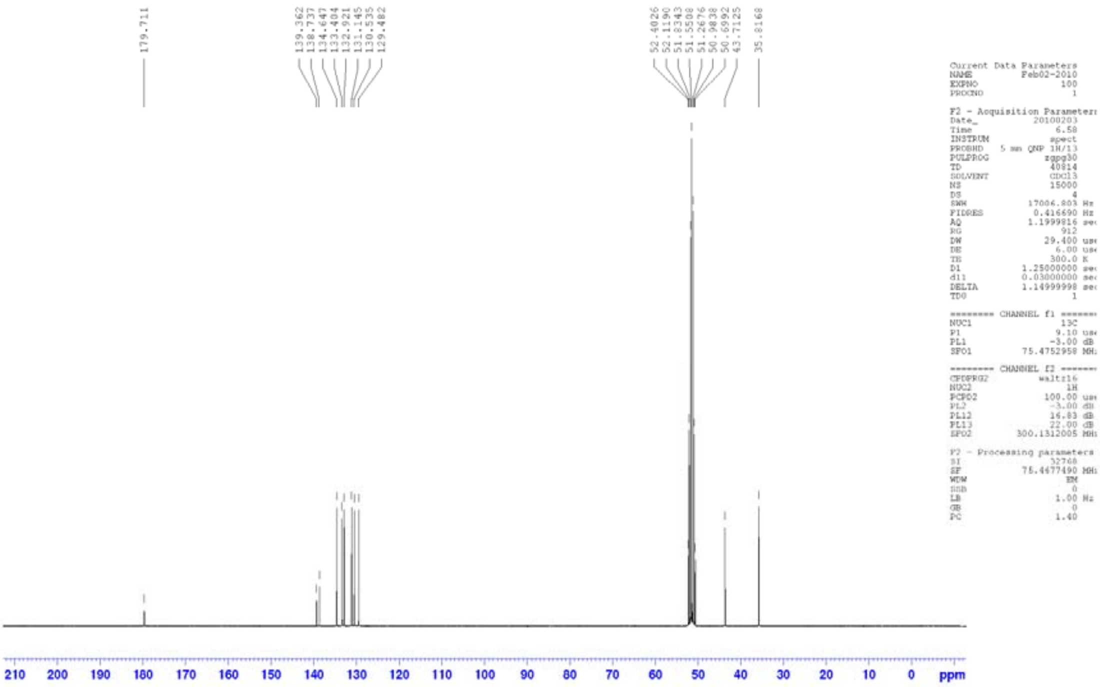


```
Current Data Parameters
NAME          Feb02-2010
EXPNO                 37
PROCNO                 1

F2 - Acquisition Paramete
Date_           20100202
Time               20.21
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             3605.769 H
FIDRES          0.110039 H
AQ             4.5438795 s
RG                   181
DW               138.667 u
DE                  6.00 u
TE                 300.0 K
D1            1.00000000 s
TD0                    1

======== CHANNEL f1 =====
NUC1                  1H
P1                 10.20 u
PL1                -3.00 d
SFO1         300.1316507 M

F2 - Processing parameter
SI                 32768
SF           300.1300063 M
WDW                   no
SSB                    0
LB                  0.00 H
GB                     0
PC                  1.00
```



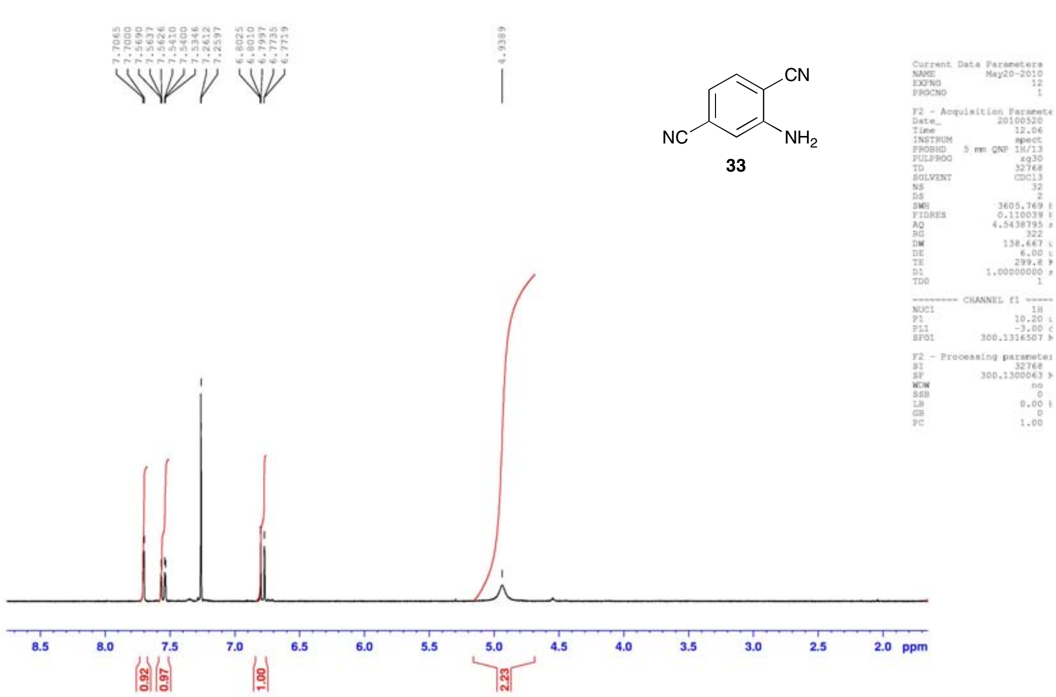
```
Current Data Parameters
NAME          Feb02-2010
EXPNO                100
PROCNO                 1

F2 - Acquisition Parameter
Date_           20100203
Time                6.58
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                 40814
SOLVENT            CDCl3
NS                 15000
DS                     4
SWH            17006.803 Hz
FIDRES          0.416690 Hz
AQ             1.1999816 sec
RG                   912
DW                29.400 use
DE                  6.00 use
TE                 300.0 K
D1            1.25000000 sec
d11           0.03000000 sec
DELTA         1.14999998 sec
TD0                    1

======== CHANNEL f1 ======
NUC1                 13C
P1                  9.10 use
PL1                -3.00 dB
SFO1          75.4752958 MH

======== CHANNEL f2 ======
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 use
PL2                -3.00 dB
PL12               16.83 dB
PL13               22.00 dB
SFO2         300.1312005 MH

F2 - Processing parameters
SI                 32768
SF            75.4677490 MH
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```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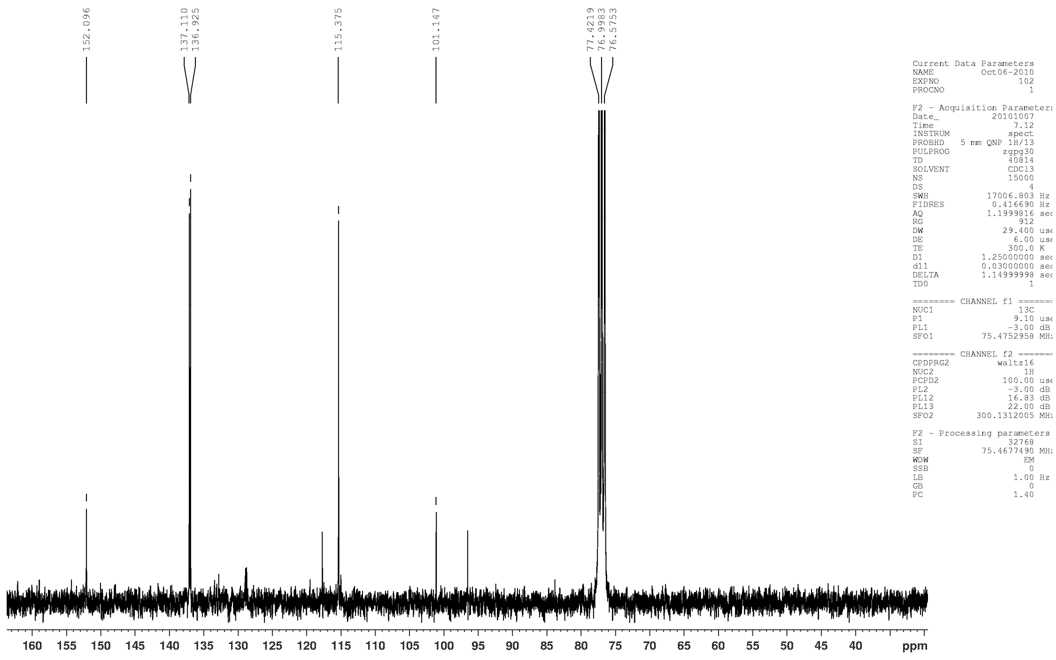
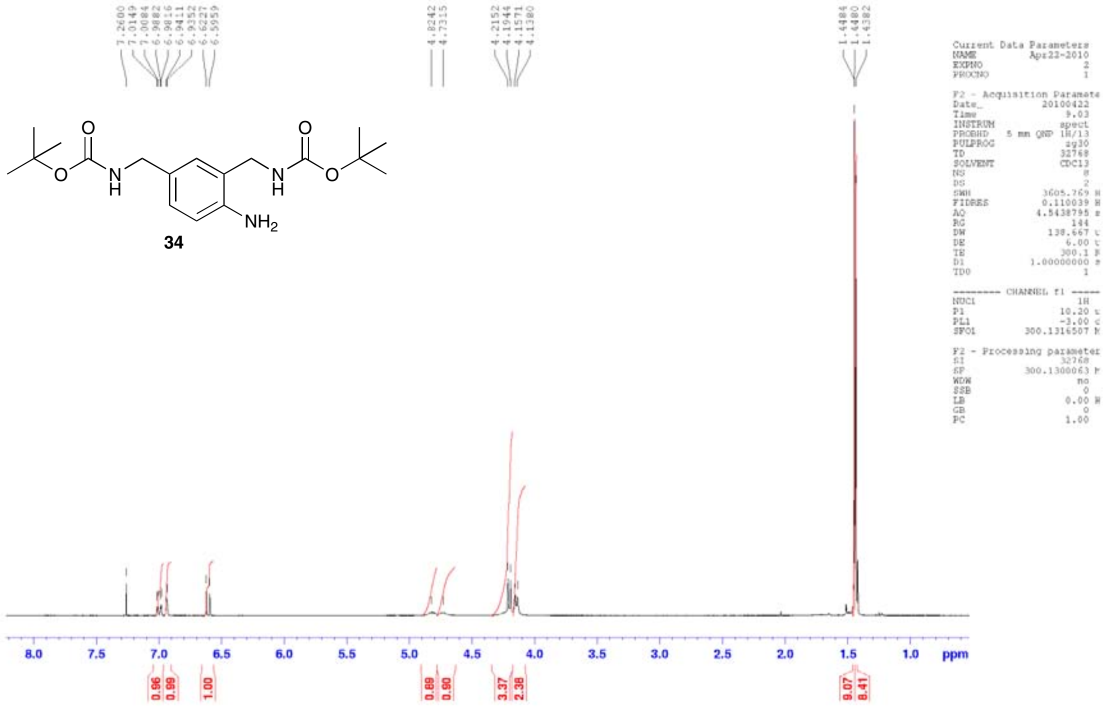
7.7000
7.5690
7.5637
7.5626
7.5410
7.5400
7.5346
7.2612
7.2597
6.8025
6.8010
6.7997
6.7735
6.7719
4.9389
CN
NC
NH2
Current Data Parameters
NAME May20-2010
EXPNO 12
PROCNO 1
F2 - Acquisition Paramete
Date_ 20100520
Time 12.06
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 3605.769
FIDRES 0.110039
AQ 4.5438795
RG 322
DW 138.667
DE 6.00
TE 299.8
D1 1.00000000
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.20
PL1 -3.00
SFO1 300.1316507
F2 - Processing paramete
SI 32768
SF 300.1300063
WDW no
SSB 0
LB 0.00
GB 0
PC 1.00
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
ppm
0.92
0.97
1.00
2.23

33


152.096
137.110
136.925
115.375
101.147
77.4219
76.9983
76.5753
Current Data Parameters
NAME Oct06-2010
EXPNO 102
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101007
Time 7.12
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 40814
SOLVENT CDCl3
NS 15000
DS 4
SWH 17006.803 Hz
FIDRES 0.416690 Hz
AQ 1.1999816 sec
RG 912
DW 29.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.25000000 sec
d11 0.03000000 sec
DELTA 1.14999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.10 usec
PL1 -3.00 dB
SFO1 75.4752958 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 16.83 dB
PL13 22.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
155
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
40
ppm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7.0149
7.0084
6.9882
6.9816
6.9411
6.9352
6.6227
6.5959
4.8242
4.7315
4.2152
4.1944
4.1571
4.1380
1.4484
1.4480
1.4382
O
O
N
H
O
N
H
O
NH2
34
Current Data Parameters
NAME Apr22-2010
EXPNO 2
PROCNO 1
F2 - Acquisition Paramete
Date_ 20100422
Time 9.03
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 3605.769 H
FIDRES 0.110039 H
AQ 4.5438795 s
RG 144
DW 138.667 u
DE 6.00 u
TE 300.1 K
D1 1.00000000 s
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 10.20 u
PL1 -3.00 d
SFO1 300.1316507 M
F2 - Processing parameter
SI 32768
SF 300.1300063 M
WDW no
SSB 0
LB 0.00 H
GB 0
PC 1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
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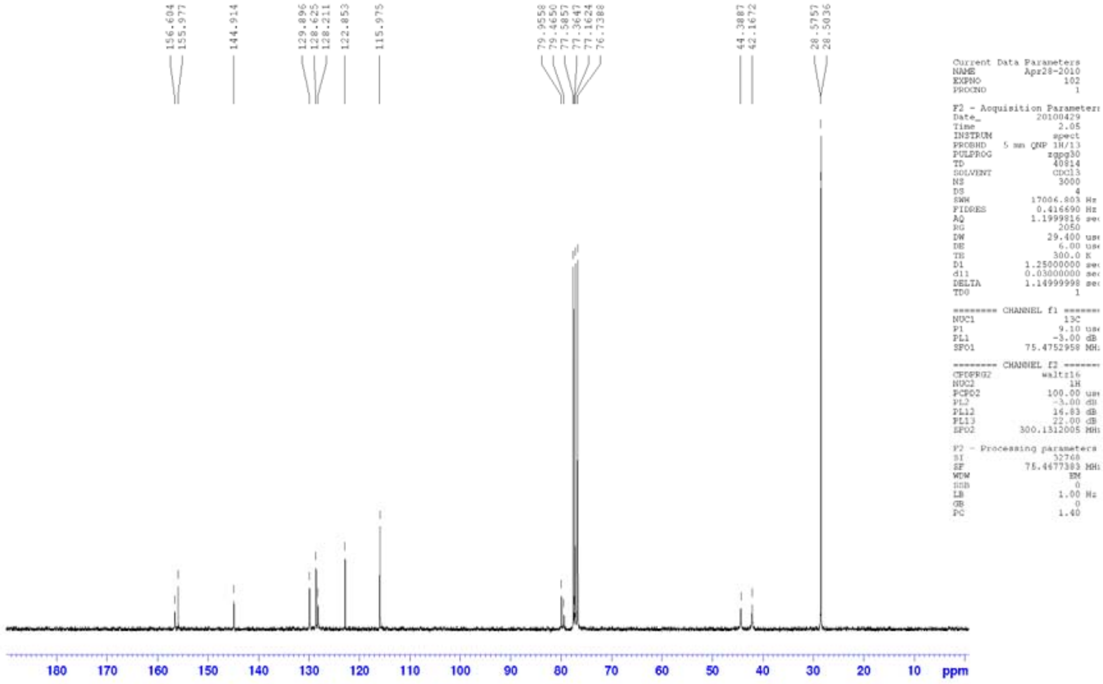
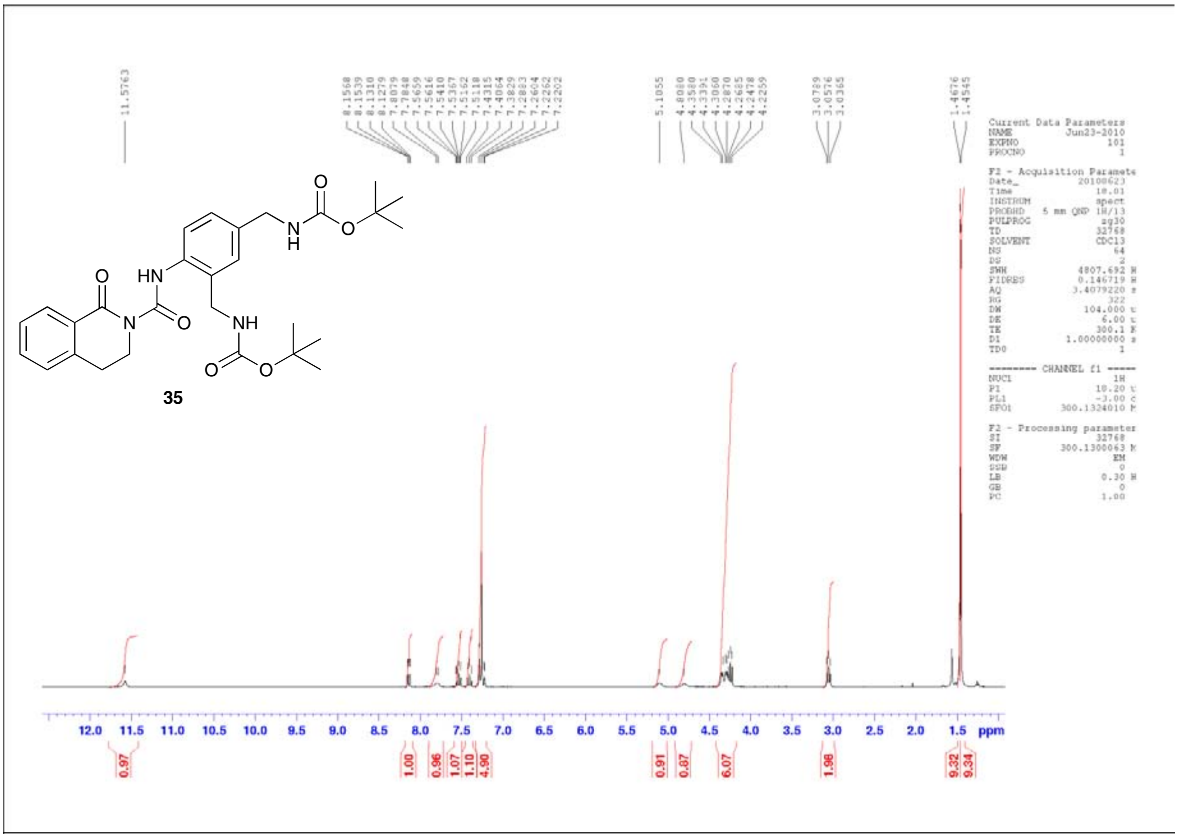
0.99
1.00
0.89
0.90
3.37
2.38
9.07
8.41


156.604
155.977
144.914
129.896
128.625
128.211
122.853
115.975
79.9558
79.4650
77.5857
77.3647
77.1624
76.7388
44.3887
42.1672
28.5757
28.5036
Current Data Parameters
NAME Apr28-2010
EXPNO 102
PROCNO 1
F2 - Acquisition Parameter
Date_ 20100429
Time 2.05
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 40814
SOLVENT CDCl3
NS 3000
DS 4
SWH 17006.803 Hz
FIDRES 0.416690 Hz
AQ 1.1999816 sec
RG 2050
DW 29.400 use
DE 6.00 use
TE 300.0 K
D1 1.25000000 sec
d11 0.03000000 sec
DELTA 1.14999998 sec
TD0 1
======== CHANNEL f1 =======
NUC1 13C
P1 9.10 use
PL1 -3.00 dB
SFO1 75.4752958 MH
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 use
PL2 -3.00 dB
PL12 16.83 dB
PL13 22.00 dB
SFO2 300.1312005 MH
F2 - Processing parameters
SI 32768
SF 75.4477383 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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8.1539
8.1310
8.1279
7.8079
7.7848
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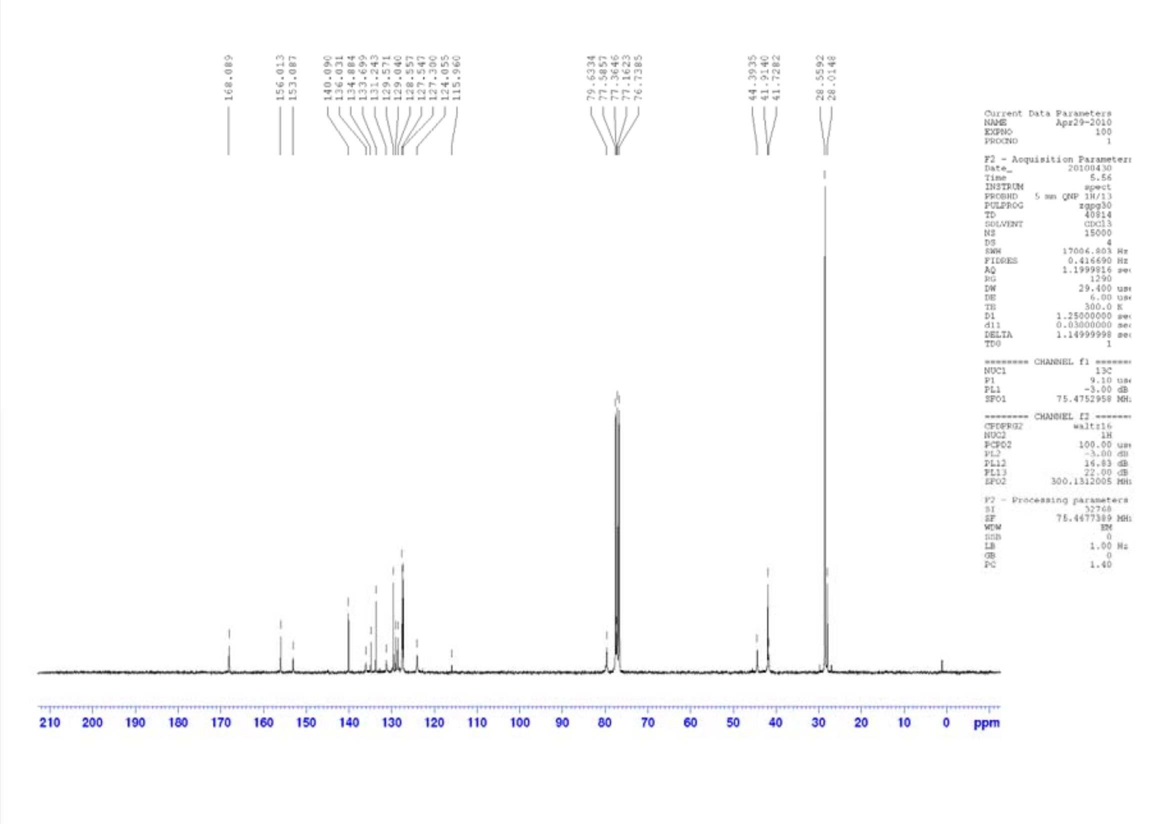
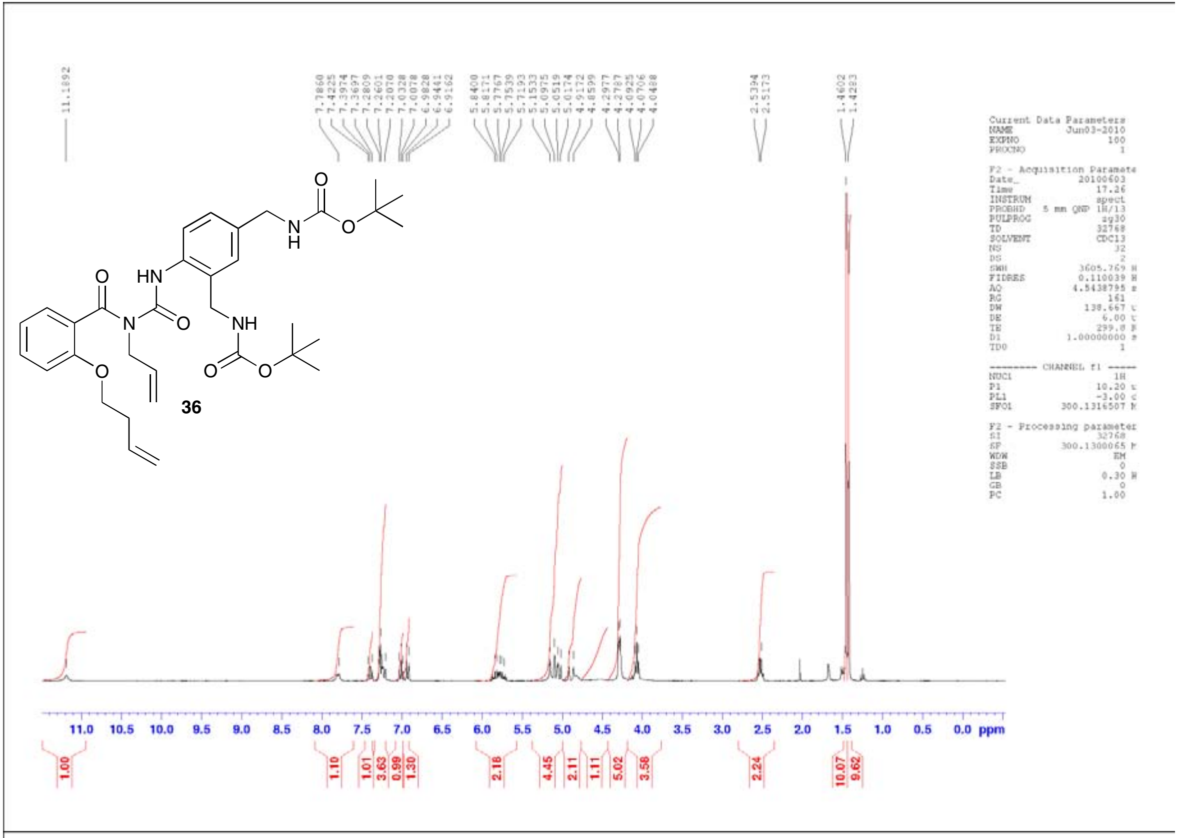
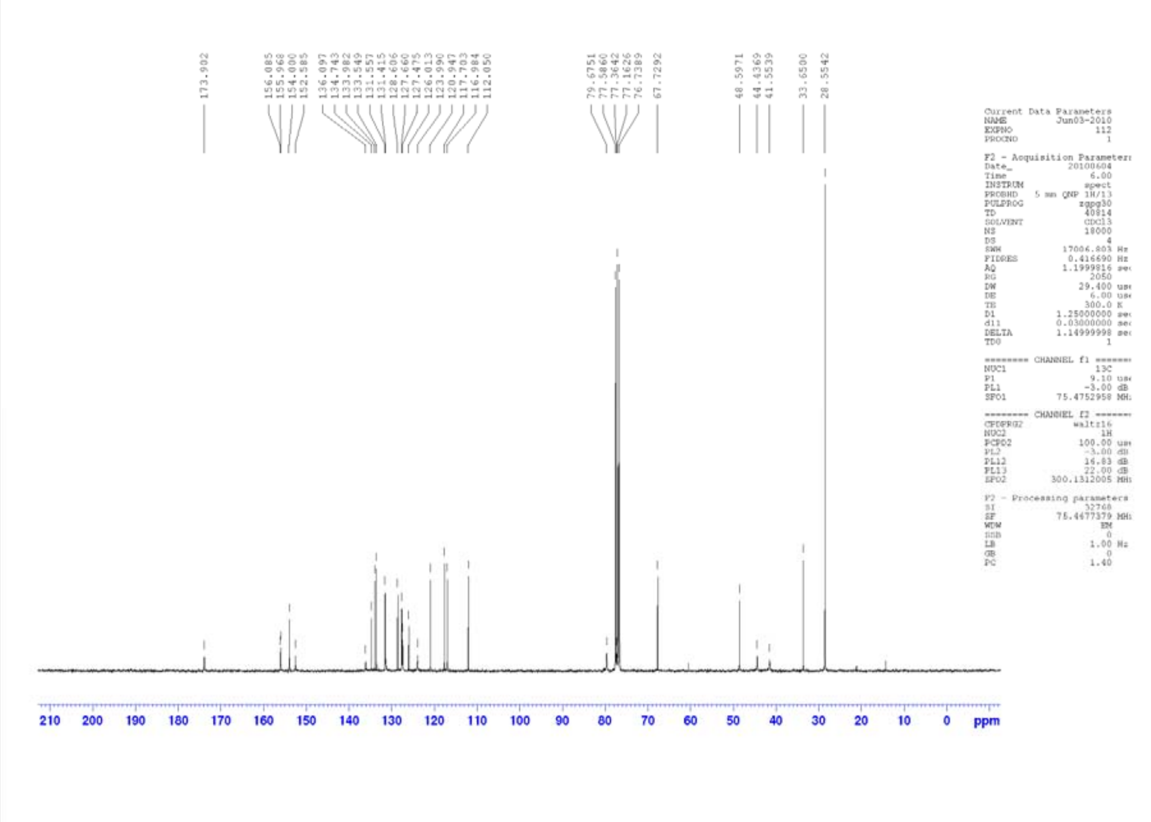
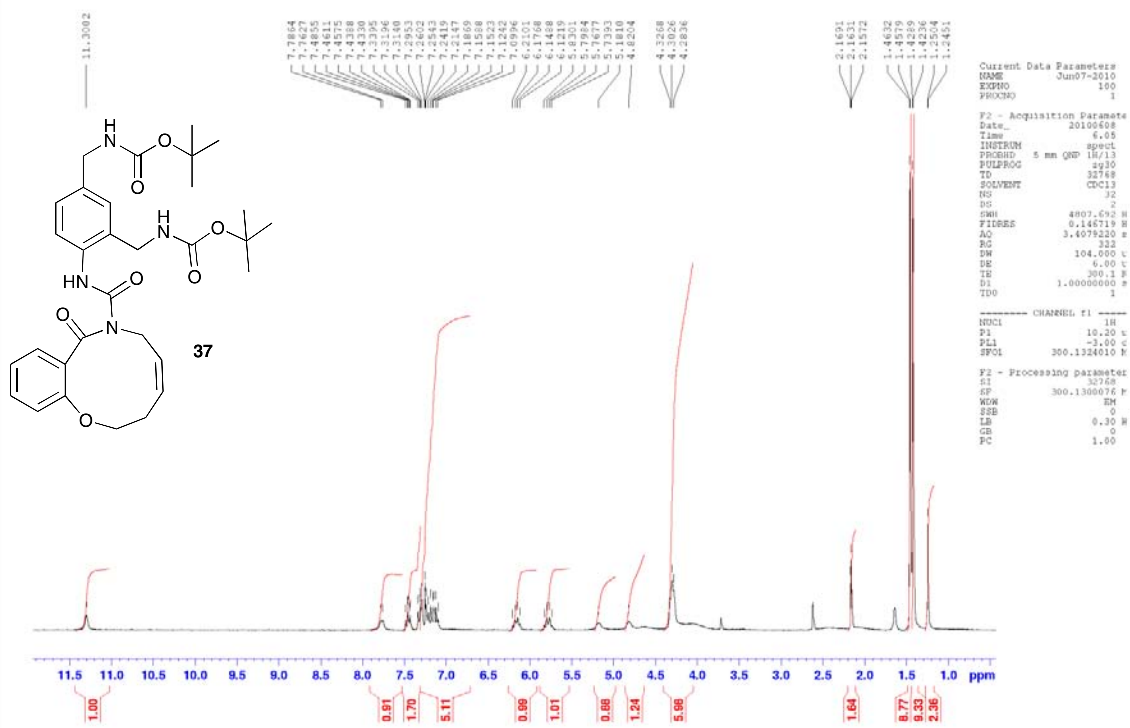
7.5616
7.5410
7.5367
7.5162
7.5118
7.4315
7.4064
7.3829
7.3583
7.2604
7.2262
7.2202
5.1055
4.8080
4.3580
4.3391
4.3060
4.2870
4.2685
4.2478
4.2259
3.0789
3.0576
3.0365
1.4676
1.4545
O
HN
N
O
N
H
O
O
NH
O
O
35
Current Data Parameters
NAME Jun23-2010
EXPNO 101
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100623
Time 18.01
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 2
SWH 4807.692 Hz
FIDRES 0.146719 Hz
AQ 3.4079220 sec
RG 322
DW 104.000 usec
DE 6.00 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.20 usec
PL1 -3.00 dB
SFO1 300.1324010 MHz
F2 - Processing parameters
SI 32768
SF 300.1300063 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
0.97
1.00
0.96
1.07
1.10
4.90
0.91
0.87
6.07
1.98
9.32
9.34


168.089
156.013
153.087
140.090
136.031
134.884
133.699
131.243
129.571
129.040
128.457
127.547
127.380
124.055
115.960
79.6334
79.5857
77.3640
77.1623
76.7385
44.3935
41.9140
41.7282
28.5592
28.0148
Current Data Parameters
NAME Apr29-2010
EXPNO 100
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100430
Time 5.56
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 40814
SOLVENT CDCl3
NS 15000
DS 4
SWH 17006.803 Hz
FIDRES 0.416690 Hz
AQ 1.1999916 sec
RG 1290
DW 29.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.25000000 sec
d11 0.03000000 sec
DELTA 1.14999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.10 usec
PL1 -3.00 dB
SFO1 75.4752958 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 16.83 dB
PL13 22.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4477389 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


11.1892
7.7860
7.4325
7.3974
7.3697
7.2809
7.2601
7.2070
7.0328
7.0078
6.9828
6.9441
6.9162
5.8400
5.8271
5.7767
5.7559
5.7193
5.1533
5.0975
5.0519
5.0174
4.9172
4.8529
4.2977
4.2787
4.0925
4.0706
4.0488
2.5394
2.5173
1.4602
1.4283
O
HN
N
O
O
N
H
O
O
NH
O
O
36
Current Data Parameters
NAME Jun03-2010
EXPNO 100
PROCNO 1
F2 - Acquisition Paramete
Date_ 20100603
Time 17.26
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 3605.769 H
FIDRES 0.110039 H
AQ 4.5438795 s
RG 161
DW 138.667 u
DE 6.00 u
TE 299.8 K
D1 1.00000000 s
TD0 1
-------- CHANNEL f1 -----
NUC1 1H
P1 10.20 u
PL1 -3.00 d
SFO1 300.1316507 M
F2 - Processing parameter
SI 32768
SF 300.1300065 M
WDW EM
SSB 0
LB 0.30 H
GB 0
PC 1.00
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0 ppm
1.00
1.10
1.01
3.63
0.99
1.30
2.18
4.45
2.11
1.11
5.02
3.58
2.24
10.07
9.62


173.902
156.085
155.968
154.090
152.585
136.097
134.743
133.982
133.549
131.557
131.415
128.606
127.660
127.475
126.013
123.990
120.947
117.733
116.984
112.050
79.6751
77.5860
77.3642
77.1626
76.7389
67.7292
48.5971
44.4369
41.5539
33.6500
28.5542
Current Data Parameters
NAME Jun03-2010
EXPNO 112
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100604
Time 6.00
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 40814
SOLVENT CDCl3
NS 18000
DS 4
SWH 17006.803 Hz
FIDRES 0.416690 Hz
AQ 1.1999816 sec
RG 2050
DW 29.400 use
DE 6.00 use
TE 300.0 K
D1 1.25000000 sec
d11 0.03000000 sec
DELTA 1.14999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.10 use
PL1 -3.00 dB
SFO1 75.4752958 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 use
PL2 -3.00 dB
PL12 16.83 dB
PL13 22.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4477379 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


37
11.3002
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7.4855
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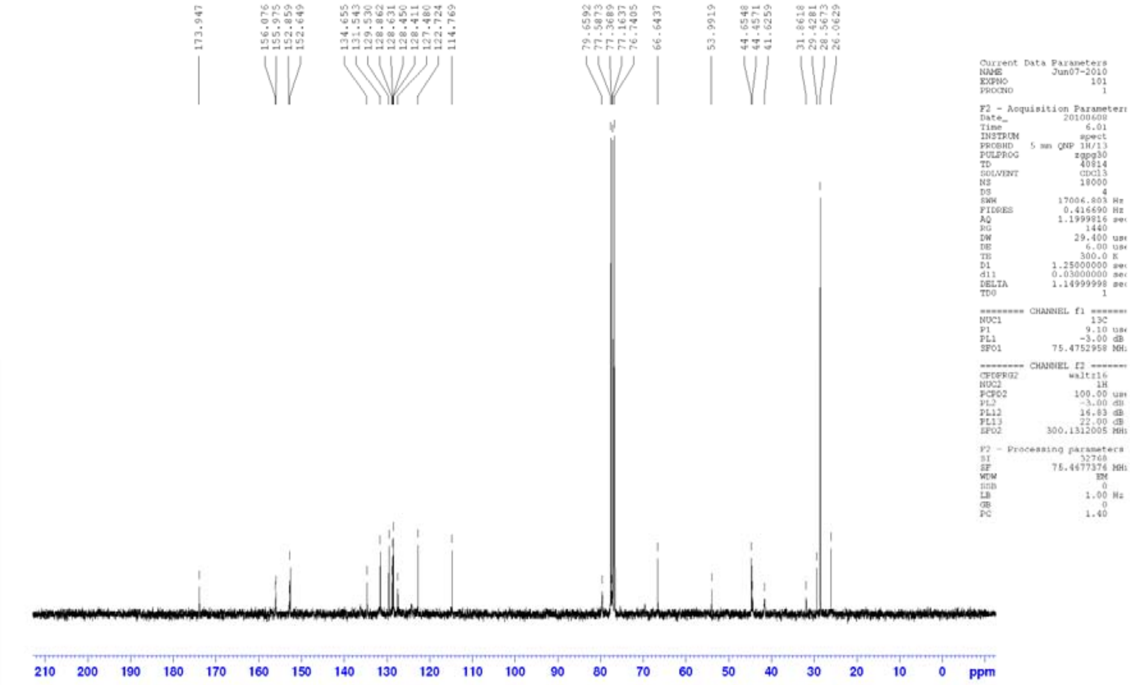
7.4388
7.4330
7.3395
7.3196
7.3140
7.2953
7.2602
7.2543
7.2419
7.2147
7.1869
7.1588
7.1523
7.1242
7.0996
6.2101
6.1768
6.1488
6.1219
5.8301
5.7984
5.7677
5.7393
5.1810
4.8204
4.3268
4.3026
4.2836
2.1691
2.1631
2.1572
1.4632
1.4579
1.4289
1.4236
1.2504
1.2451
Current Data Parameters
NAME Jun07-2010
EXPNO 100
PROCNO 1
F2 - Acquisition Paramete
Date_ 20100608
Time 6.05
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 4807.692 H
FIDRES 0.146719 H
AQ 3.4079220 s
RG 322
DW 104.000 u
DE 6.00 u
TE 300.1 K
D1 1.00000000 s
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 10.20 u
PL1 -3.00 d
SFO1 300.1324010 M
F2 - Processing parameter
SI 32768
SF 300.1300076 M
WDW EM
SSB 0
LB 0.30 H
GB 0
PC 1.00
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.00
0.91
1.70
5.11
0.99
1.01
0.88
1.24
5.98
1.64
8.77
9.33
2.36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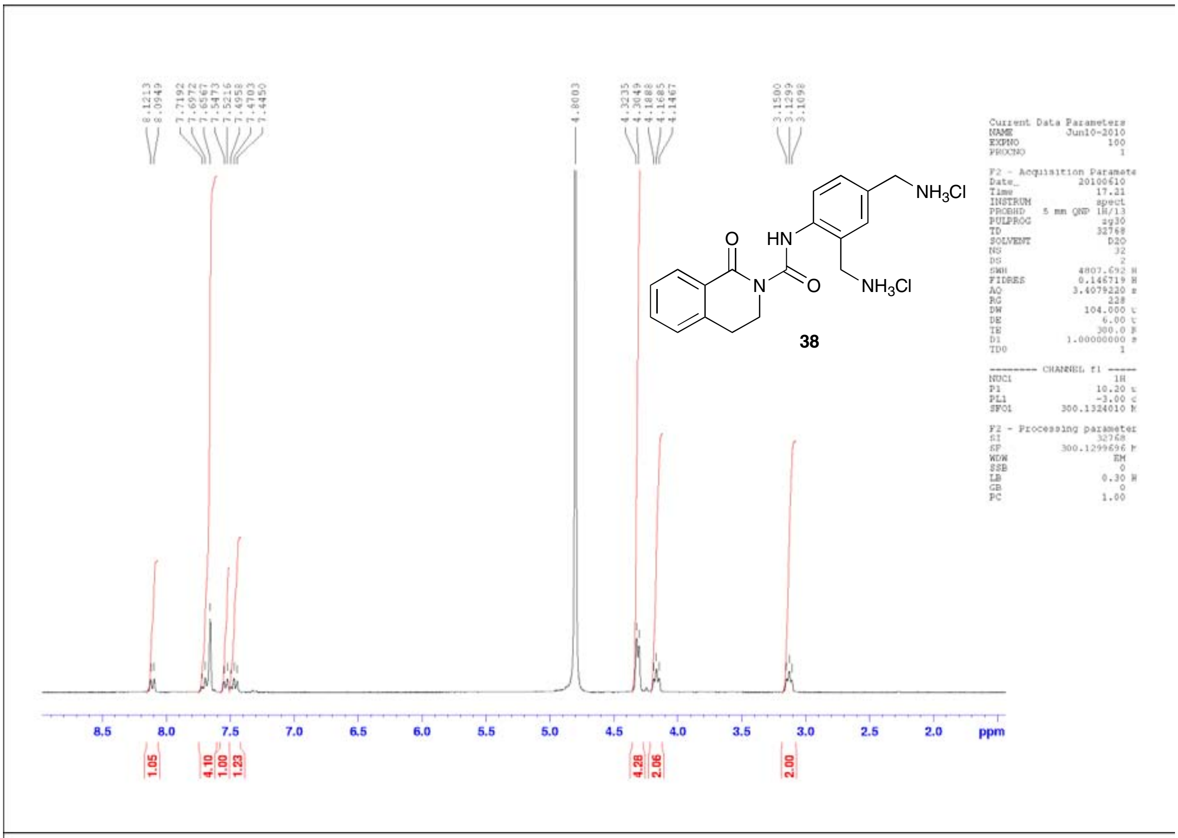
155.975
152.859
152.649
134.655
131.543
129.530
128.862
128.631
128.450
128.411
127.480
122.724
114.769
79.6592
77.5873
77.3689
77.1637
76.7405
66.6437
53.9919
44.6548
44.4571
41.6259
31.8618
29.4281
28.5673
26.0629
Current Data Parameters
NAME Jun07-2010
EXPNO 101
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100608
Time 6.01
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 40814
SOLVENT CDCl3
NS 18000
DS 4
SWH 17006.803 Hz
FIDRES 0.416690 Hz
AQ 1.1999816 sec
RG 1440
DW 29.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.25000000 sec
d11 0.03000000 sec
DELTA 1.14999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.10 usec
PL1 -3.00 dB
SFO1 75.4752958 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 16.83 dB
PL13 22.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4477376 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



```
Current Data Parameters
NAME          Jun10-2010
EXPNO                100
PROCNO                 1

F2 - Acquisition Paramete
Date_           20100610
Time               17.21
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT              D2O
NS                    32
DS                     2
SWH             4807.692 H
FIDRES          0.146719 H
AQ             3.4079220 s
RG                   228
DW               104.000 u
DE                  6.00 u
TE                 300.0 K
D1            1.00000000 s
TD0                    1

======== CHANNEL f1 =====
NUC1                  1H
P1                 10.20 u
PL1                -3.00 c
SFO1         300.1324010 M

F2 - Processing parameter
SI                 32768
SF           300.1299696 M
WDW                   EM
SSB                    0
LB                  0.30 H
GB                     0
PC                  1.00
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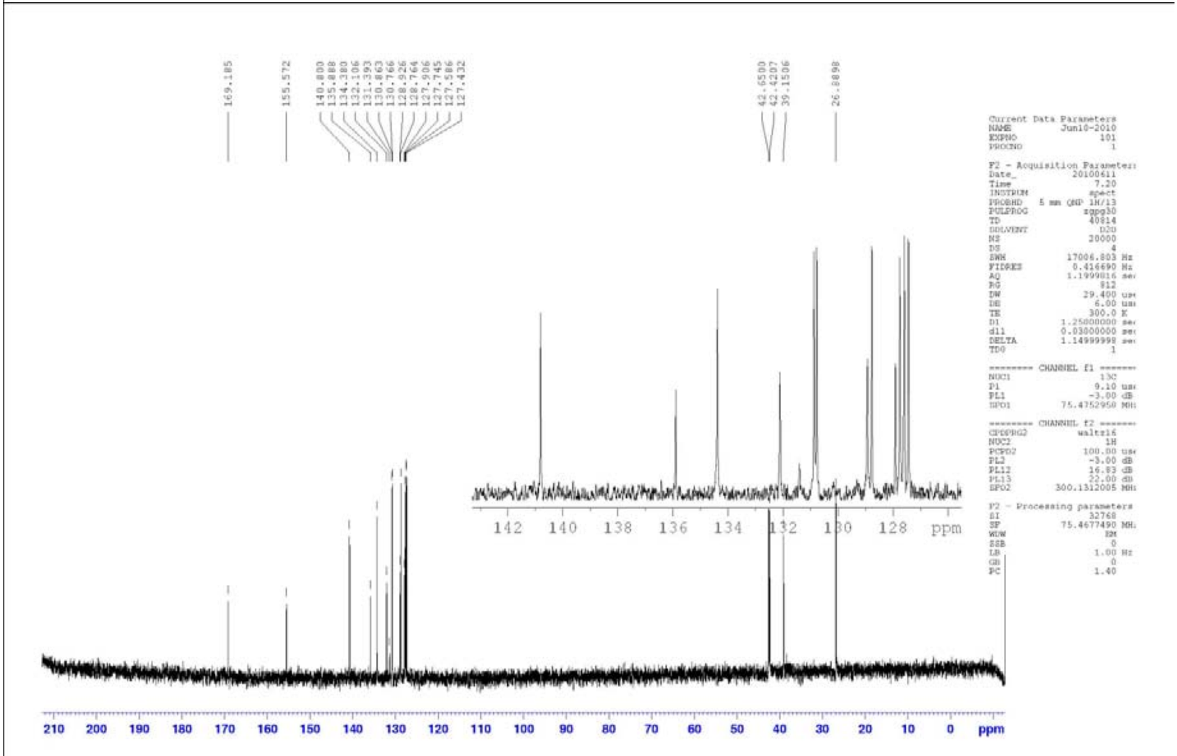

```
Current Data Parameters
NAME          Jun10-2010
EXPNO                101
PROCNO                 1

F2 - Acquisition Parameter
Date_           20100611
Time                7.20
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG           zgpg30
TD                 40814
SOLVENT              D2O
NS                 20000
DS                     4
SWH            17006.803 Hz
FIDRES          0.416690 Hz
AQ             1.1999816 sec
RG                   812
DW                29.400 use
DE                  6.00 use
TE                 300.0 K
D1            1.25000000 sec
d11           0.03000000 sec
DELTA         1.14999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.10 use
PL1                -3.00 dB
SFO1          75.4752950 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 use
PL2                -3.00 dB
PL12               16.83 dB
PL13               22.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF            75.4677490 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

References:

1) Ikegami, R.; Koresawa, A.; Shibata, T.; Takagi, K., *Journal of Organic Chemistry* **2003,** 68, (6), 2195-2199.

2) Harvey, D. F.; Sigano, D. M., *Journal of Organic Chemistry* **1996,** 61, (7), 2268-2272.
3) Renhowe, P.; Pecchi, S; Shafer, C.; Machajewski, T.; Jazan, E.; Taylor, C.; Antonios-McCrea, W.; McBride, C.; Frazier, K.; Wiesmann, M.; Lapointe, G.; Feucht, P.; Warne, R.; Heise, C..; Menezes, D.; Aardalen, K.; Ye, H.; He, M.; Le, V.; Vora, J.; Jansen, J.; Wernette-Hammond, M.; Harris, A., *Journal of Medicinal Chemistry*, **2009** , 52, (2), p. 278 - 292