Electronic Supporting Information

Rhodium(III) catalysed aerobic synthesis of highly functionalized indoles from *N*-arylurea under mild conditions through C-H activation

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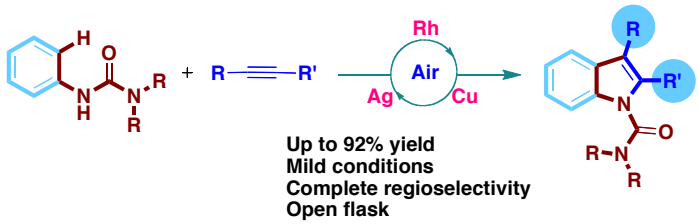
Abstract



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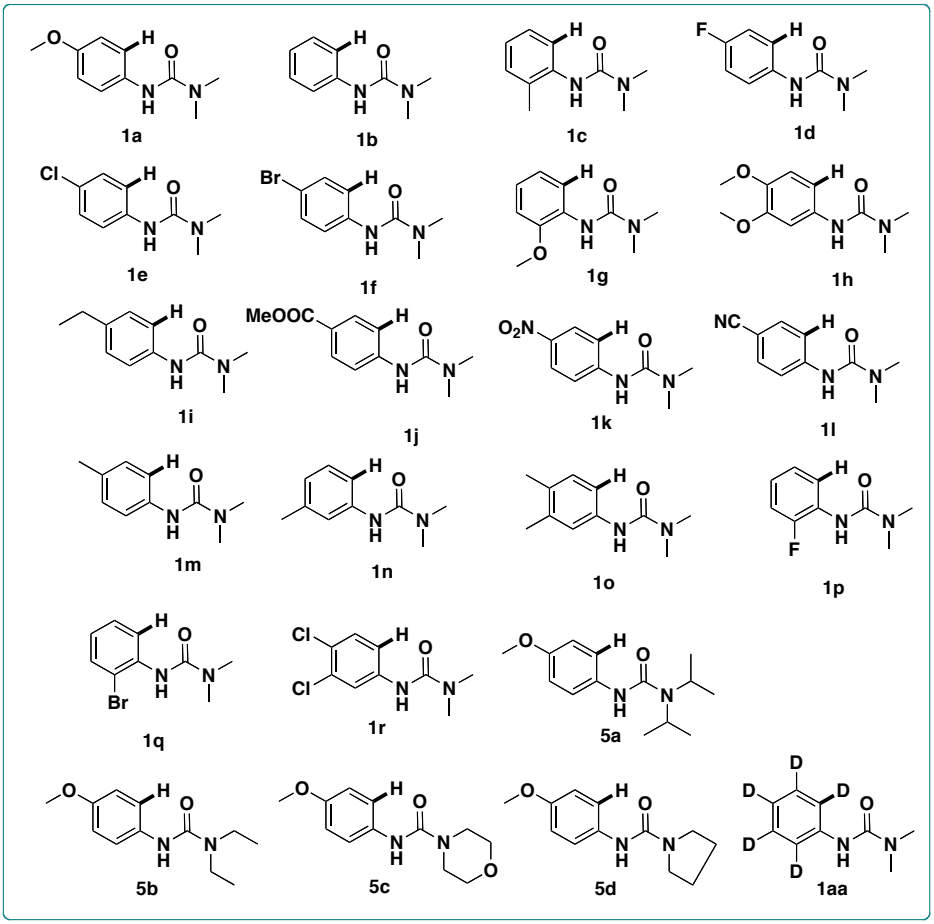
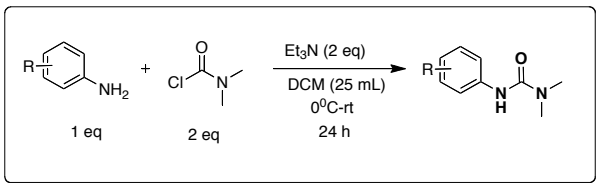
I. General Methods. Catalytic reactions were carried out under ambient air. Unless otherwise noted, all reagents were obtained from commercial suppliers and used without further purification. All glassware was dried overnight at 120 °C or flame-dried prior to use. Chromatography was performed on Sigma-Aldrich 160-220 mesh silica gel. Thin-layer chromatography (TLC) was conducted with precoated glass-backed plates and visualized by exposure to UV light (254 nm). Flash chromatography was performed with silica gel (100-120 μm); the eluent used is *n*-heptane and ethyl acetate. Solvents were used without further purification. H^1 NMR spectra were recorded on a 500 MHz Varian FT-NMR spectrometer. ^{13}C NMR spectra were recorded at 125 MHz Varian FT-NMR. Chemical shifts are reported in ppm relative to solvent signal. Multiplicity is indicated as follows: s (singlet); d (doublet); t (triplet); q (quartet); m (multiplet); dd (doublet of doublets). Mass spectra (HRMS) were performed by the Lund University Kemi Centrum Mass Spectrometry Facility. Melting points were determined on a micro melting point apparatus (Stuart Scientific).

II. Experimental Section.

General Procedure for Synthesis of *N*-arylurea

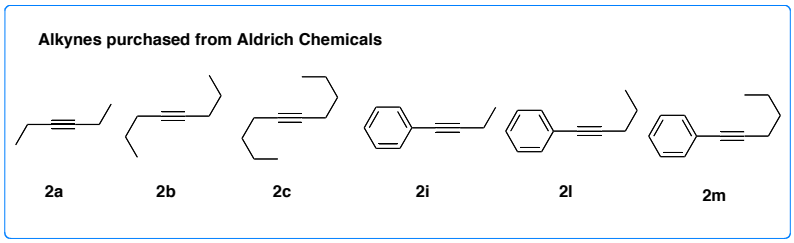


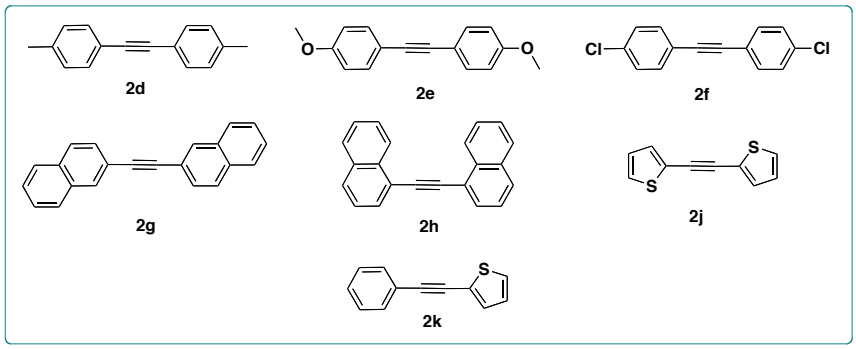


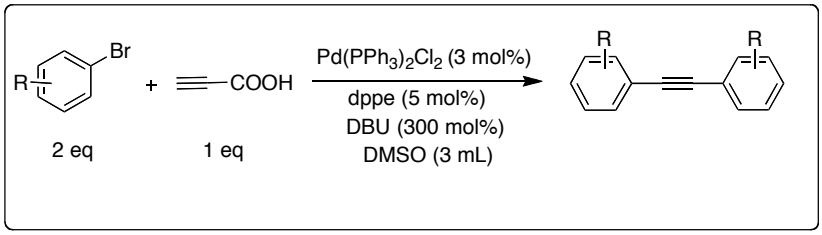
Aniline derivatives (1.00 g, 10 mmol) and triethylamine (2.9 mL, 20 mmol) were dissolved in anhydrous CH_2Cl_2 (10 mL) in a 50 mL three neck round bottom flask followed by drop wise addition of chloroformic acid dimethyl amide (1.8 mL, 20 mmol) using a syringe. The reaction mixture was stirred overnight. After completion, the reaction was diluted with CH_2Cl_2 (30 mL), washed by sat. $NaHCO_3$ (30 mL), 2N HCl (30 mL), brine (20 mL) and dried over $MgSO_4$. The organic solvent was removed by evaporation. Purification by recrystallization in diethylehter afforded 0.92 g of *N*-aryl urea as an off white solid. The spectroscopic values are identical that of with literature.[1]

SI 1. Houlden, C. E.; Bailey, C. D.; Ford, J. G.; Gagne, M. R.; Llyod-Jones, G. C.; Booker-Milburn, K. I. *J. Am. Chem. Soc*., **2008**, *130*, 10066.

General Procedure for Synthesis of Symmetrical Alkynes







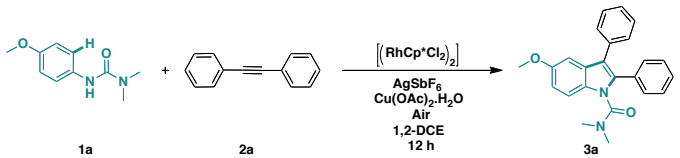
Propiolic acid (1.0 mmol), aryl bromides (2.0 mmol), $Pd(PPh_3)_2Cl_2$ (0.03 mmol), dppe (0.05 mmol) were combined with DBU (3 mmol) in a screw cap vial. DMSO was added and the vial was sealed and heated in a preheated oil bath at 80^0C for 3-4 h. The reaction mixture was poured into 25 mL of saturated aquous ammonium chloride and extracted with diethyl ether. The combined ether layer was washed with brine, dried over $MgSO_4$, and filtered. The solvent was removed under reduced pressure, and the resulting crude mixture was purified by column chromatography on silica gel. The product was eluted with 5% ethyl acetate in heptane. The spectroscopic values are identical that of with literature.[2]

SI 2. Park, K.; Bae, G.; Moon, J.; Choe, J.; Song, K. H.; Lee, S. *J. Org. Chem*. **2010**, *75*, 6244.

Optimization Studies

SI Table 1.

Screening of Temperature

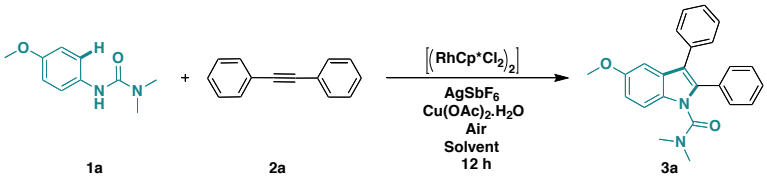


1a 2a 3a

Entry	Temperature	Yield (%)
1	r.t	--
2	50	12
3	80	60
4	100	92
5	120	92

SI Table 2

Screening of Solvents

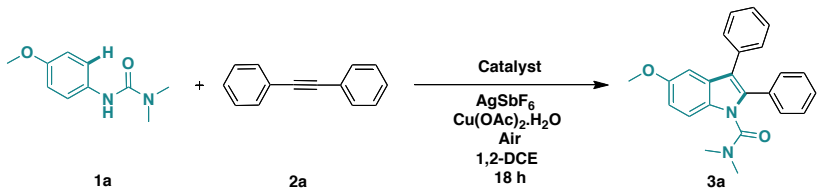


1a 2a 3a

Entry	Solvent	Yield (%)	Entry	Solvent	Yield (%)
1	2-Methyl-2-Butanol	48	9	Toluene	22
2	2-Methyl-1-Butanol	30	10	$ClCH_2CH_2Cl$	92
3	*t*-BuOH	20	11	1,4-Dioxane	5
4	THF	35	12	Toluene	trace
5	DMF	12	13	Methanol	13
6	DMSO	NR	14	Ethanol	10
7	DCM	10	15	H_2O	NR
8	AcOH	NR	16	CF_3COOH	NR

SI Table 3

Screening of Catalysts

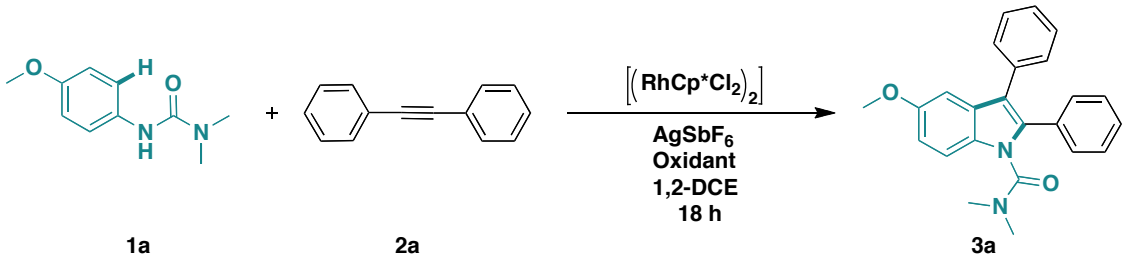


1a 2a 3a

Entry	Catalyst	Yield (%)
1	$[(RuCp^*Cl_2)_2]$	55
2	$RuCl_3.H_2O$	- -
3	$[(RhCp^*Cl_2)_2]$	92
4	$[(IrCp^*Cl_2)_2]$	- -
5	Ag_2O	- -
6	Ag	- -
7	$Cu(OAc)_2$	- -

SI Table 4

Screening of Oxidants



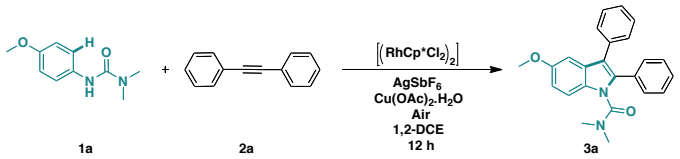
1a 2a 3a

Entry	Oxidants	Yield (%)
1	$Cu(OAc)_2$	90
2	$Cu(OAc)_2.H_2O$	92
3	Ag_2CO_3	40
4	Ag_2O	15
5	MnO_2	10
6	H_2O_2	5
7	m-CPBA	- -
8[a]	$Cu(OAc)_2.H_2O$	90
9[b]	$Cu(OAc)_2.H_2O$	92

[a]under nitrogen, [b]under oxygen.

SI Table 5

Screening of Equivalent of $Cu(OAc)_2.H_2O$

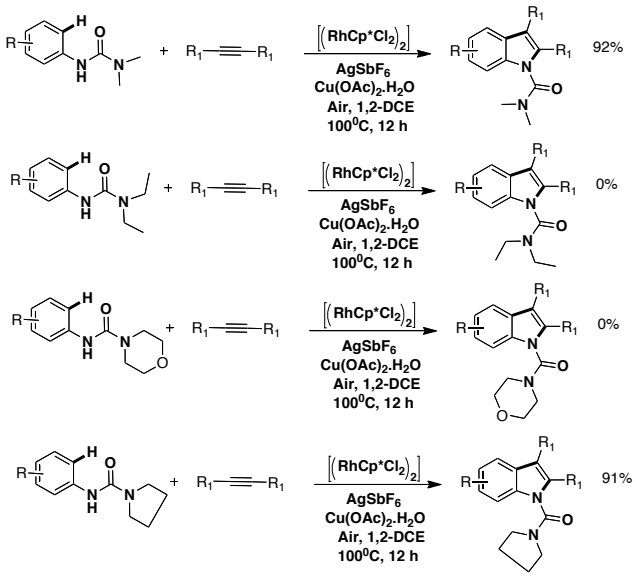


1a 2a 3a

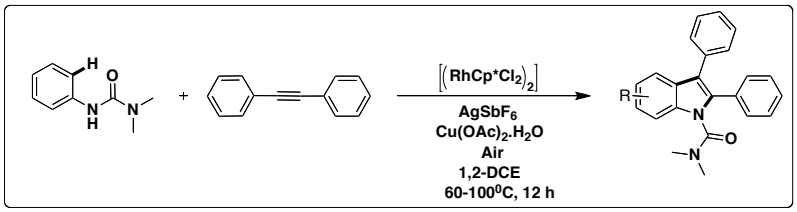
Entry	Copper Equivalent	Yield (%)
1	0.1 equiv.	trace
2	0.2 equiv.	trace
3	0.5 equiv.	25
4	1.0 equiv.	92
5	2.2 equiv.	90

SI Table 6

Screening of different N-carbamyl units of arylurea



General Procedure for C–H Activation Experiments:



$[RhCp^*Cl_2]_2$ (1 mol%), $AgSbF_6$ (20 mol%), and $Cu(OAc)_2 \cdot H_2O$ (1.0 equiv.), N-arylurea 1 (1.5 mmol), alkyne 2 (1.0 mmol) were added to a 5 mL Schlenk tube, which was equipped

with a magnetic stirrer and septum. (*Note:* Care must be taken to avoid reaction of $AgSbF_6$ with air.) To the tube were added by syringe 1,2-dichloroethane (2 mL) as the solvent, and the reaction mixture was allowed to stir at room temperature for 10 min. During this time, the tube was covered with a septum. Then, the septum was removed and the reaction mixture was allowed to stir under open air for and additional 10 min. The tube was now sealed with a screw cap, and the reaction mixture was stirred at 100^0C for 12h. After cooling to room temperature, the reaction mixture was diluted with dichloromethane (DCM) and then filtered through a pad of Celite and silica gel. The filtrate was concentrated, and the resulting crude mixture was purified through a silica gel column chromatography to give 3a using heptane and ethylacetate (4:1) as eluent.

III. Spectral Data

***N, N*-Dimethyl-2,3-Diphenyl-4-Methoxyindole-1-Carboxamide (3a):** 92 mg, Yellow Solid, mp-98^0C; **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.43-7.41 (m, 1H), 7.32-7.28 (m, 10H), 7.10 (s, 1H), 6.97-6.95 (m, 1H), 3.80 (s, 3H), 2.95 (brs, 3H), 2.51 (brs, 3H).
^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 155.6, 154.0, 135.5, 134.1, 131.3, 130.8, 130.0, 129.5, 128.8, 128.5, 128.2, 128.0, 126.5, 117.6, 113.6, 112.3, 101.6, 55.9, 31.9, 37.9, 36.5.
HRMS (ESI+) Calcd for $C_{24}H_{22}N_2O_2$ $[M+Na]^+$ 393.1579; Found 393.1579.

***N, N*-Dimethyl-2,3-Diphenyl-Indole-1-Carboxamide (3b):** 80 mg, Colorless Solid, mp-112^0C; **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.70 (d, *J*=5Hz, 1H), 7.54 (d, *J*=5Hz, 1H), 7.35-7.28 (m, 10H), 7.25-7.20 (m, 2H), 3.01 (brs, 3H), 2.55 (brs, 3H).
^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 154.0, 135.7, 134.8, 134.0, 131.2, 130.1, 129.6, 128.4, 128.3, 128.2, 128.0, 126.5, 123.7, 121.7, 119.8, 117.8, 111.5, 38.3, 36.5.
HRMS (ESI+) Calcd for $C_{23}H_{20}N_2O$ $[M+Na]^+$ 363.1473; Found 363.1473.

***N, N*-Dimethyl-2,3-Diphenyl-2-Methylindole-1-Carboxamide (3c):** 68 mg, Yellow Solid, mp-127^0C; **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.59 (d, *J*=10 Hz, 1H), 7.41-7.40 (m, 2H), 7.32-7.31 (m, 7H), 7.26-7.23 (m, 1H), 7.14-7.07 (m, 2H), 2.92 (s, 3H), 2.52 (s, 3H), 2.44 (s, 3H).
^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 154.9, 134.7, 134.4, 131.2, 130.3, 128.4, 128.4, 128.3, 126.3, 125.7, 121.6, 121.5, 117.8, 37.9, 36.4, 18.2.
HRMS (ESI+) Calcd for $C_{24}H_{22}N_2O$ $[M+Na]^+$ 377.1630; Found 377.1630.

***N, N*-Dimethyl-2,3-Diphenyl-4-Fluoroindole-1-Carboxamide (3d):** 58 mg, Yellow Solid, mp-114^{0}C; **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.46 (m, 1H), 7.33-7.25 (m, 8H), 7.16-7.03 (m, 4H), 2.97 (s, 3H), 2.50 (s, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 160.1, 158.2, 153.8, 136.5, 133.7, 132.2, 131.1, 130.0, 129.6, 129.0, 128.6, 128.5, 128.4, 126.8, 117.8, 112.5-111.9 (q), 105.2, 105.0, 38.1, 36.5.

HRMS (ESI+) Calcd for $C_{23}H_{19}FN_2O_2$ $[M+Na]^+$ 381.1379; Found 381.1377.

***N, N*-Dimethyl-2,3-Diphenyl-4-Chloroindole-1-Carboxamide (3e):** 80 mg, Yellow Solid, mp-121^{0}C; **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.62 (s, 1H), 7.44 (d, *J*=5Hz, 1H), 7.35-7.25 (m, 10H), 7.14-7.06 (m, 1H), 2.97 (s, 3H), 2.49 (s, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 153.6, 136.1, 134.1, 133.4, 130.8, 130.1, 129.6, 129.4, 128.6, 128.5, 127.5, 126.9, 124.0, 119.4, 117.3, 112.6, 38.0, 36.5.

HRMS (ESI+) Calcd for $C_{23}H_{19}ClN_2O$ $[M+Na]^+$ 397.1084; Found 397.1080.

***N, N*-Dimethyl-2,3-Diphenyl-4-Bromoindole-1-Carboxamide (3f):** 65 mg, Yellow Solid, mp-143^{0}C; **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.70 (s, 1H), 7.31 (s, 2H), 7.26-7.19 (m, 10H), 2.88 (s, 3H), 2.39 (s, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 153.5, 136.0, 134.4, 133.4, 130.8, 130.1, 130.0, 129.6, 128.6, 128.5, 126.9, 126.6, 122.4, 117.2, 115.0, 113.0, 38.0, 36.5.

HRMS (ESI+) Calcd for $C_{23}H_{19}BrN_2O$ $[M+Na]^+$ 441.0578; Found 441.0580.

***N, N*-Dimethyl-2,3-Diphenyl-2-Methoxyindole-1-Carboxamide (3g):** 81 mg, Yellow Solid, mp-103^{0}C; **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.38 (brs, 2H), 7.34-7.27 (m, 8H), 7.22-7.19 (m, 1H), 7.13-7.09 (m, 1H), 6.76 (d, *J*=10Hz, 1H), 3.94 (s, 3H), 2.98 (s, 3H), 2.61 (s, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 154.4, 146.7, 135.1, 134.4, 131.1, 130.3, 130.1, 129.5, 128.3, 128.2, 126.2, 125.4, 121.8, 117.5, 112.6, 104.4, 56.4, 38.0, 36.5.

HRMS (ESI+) Calcd for $C_{24}H_{22}N_2O_2$ $[M+Na]^+$ 393.1579; Found 393.1579.

***N, N*-Dimethyl-2,3-Diphenyl-3,4-Dimethoxyindole-1-Carboxamide (3h):** 76 mg, Yellow Solid, mp-113^{0}C; **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.26-7.18 (m, 10 H), 7.01 (s, 1H), 6.97 (s, 1H), 3.87 (s, 3H), 3.78 (s, 3H), 2.83 (brs, 3H), 2.30 (s, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 154.3, 148.1, 146.3, 134.3, 133.2, 131.4, 130.1, 130.0, 129.3, 128.5, 128.2, 127.6, 126.4, 120.9, 117.5, 101.1, 94.8, 60.4, 56.3, 37.9, 36.5.

HRMS (ESI+) Calcd for $C_{25}H_{24}N_2O_3$ $[M+Na]^+$ 423.1685; Found 423.1684.

***N, N*-Dimethyl-2,3-Diphenyl-4-Eethylindole-1-Carboxamide (3i):** 92 mg, Yellow Solid, mp-109^{0}C; **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.53 (s, 1H) 7.51 (d, *J*=10 Hz, 1H), 7.38-7.32 (m, 8H), 7.24-7.23 (m, 1H), 7.16-7.10 (m, 1H), 7.03-7.01 (m, 1H), 3.02 (s, 3H), 2.80 (m, 2H), 2.60 (s, 3H), 1.32 (t, *J*=5Hz, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 154.2, 137.9, 135.0, 134.3, 134.2, 131.4, 130.2, 129.6, 128.5, 128.4, 128.3, 128.1, 128.0, 126.5, 124.3, 117.7, 118.3, 117.7, 111.3, 38.0, 36.5, 29.1, 16.5.

HRMS (ESI+) Calcd for $C_{25}H_{24}N_2O$ $[M+Na]^+$ 368.1889; Found 368.1888.

***N, N*-Dimethyl-2,3-Diphenyl-4-methylbenzoate-1-Carboxamide (3j) :** 84 mg, Yellow Solid, mp-56^{0}C, **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 8.32 s, (1H), 7.95 (d, *J*=5Hz, 1H), 7.45 (d, *J*=10 Hz, 1H), 7.27-7.25 (m, 5H), 7.23-7.21 (m, 5H), 3.82 (s, 3H), 2.91 (s, 3H), 2.43 (s, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 167.7, 153.3, 138.0, 136.1, 133.2, 130.6, 130.1, 129.5, 128.6, 128.4, 127.8, 126.8, 125.0, 123.7, 122.6, 118.4, 111.1, 52.0, 37.9, 36.5.

HRMS (ESI+) Calcd for $C_{25}H_{22}N_2O_3$ $[M+H]^+$ 399.1709; Found 399.1704.

***N, N*-Dimethyl-2,3-Diphenyl-4-Methylindole-1-Carboxamide (3m):** 65 mg, colorless Solid, mp-120^{0}C; **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.44 (s, 1H), 7.41 (d, *J*=5Hz, 1H), 7.33-7.28 (m, 10H), 7.15 (d, *J*=10 Hz, 1H), 2.97 (s, 3H), 2.53 (s, 3H), 2.44 (s, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 154.2, 135.1, 134.3, 134.1, 131.5, 131.2, 130.3, 129.6, 128.5, 128.3, 128.0, 126.5, 125.3, 119.5, 117.6, 111.2, 37.9, 36.5, 21.6.

HRMS (ESI+) Calcd for $C_{24}H_{22}N_2O$ $[M+Na]^+$ 377.1630; Found 377.1627.

***N, N*-Dimethyl-2,3-Diphenyl-3-Methylindole-1-Carboxamide (3n):** 72 mg, Brown Solid, mp-78^{0}C; **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.56 (d, *J*=10 Hz, 1H), 7.32-7.25 (m, 10H), 7.10 (s, 1H), 7.05 (d, *J*=10 Hz, 1H), 2.97 (s, 3H), 2.49 (s, 6H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 154.3, 136.1, 134.3, 134.0, 131.5, 130.2, 129.6, 128.5, 128.4, 128.0, 126.5, 126.2, 123.5, 119.6, 117.7, 111.5, 38.0, 36.4, 21.9.

HRMS (ESI+) Calcd for $C_{24}H_{22}N_2O$ $[M+Na]^+$ 377.1630; Found 377.1628.

***N, N*-Dimethyl-2,3-Diphenyl-3,4-Dimethylindole-1-Carboxamide (3o) :** 65 mg, Yellow Solid, mp-133^{0}C; **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.32 (s, 1H), 7.23 (s, 6H), 7.18 (s, 5H), 2.87 (s, 3H), 2.37 (s, 3H), 2.30 (s, 3H), 2.24 (s, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 154.3, 134.7, 134.4, 134.0, 133.1, 131.6, 130.5, 130.2, 129.6, 128.4, 128.3, 127.8, 126.6, 126.4, 119.9, 117.4, 111.1, 38.0, 36.5, 20.6, 20.2.

HRMS (ESI+) Calcd for $C_{25}H_{24}N_2O$ $[M+Na]^+$ 391.1786; Found 391.1766.

***N, N*-Dimethyl-2,3-Diethyl-4-Methoxyindole-1-Carboxamide (4a):** 93 mg, Colorless Liquid, **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.00-6.96 (m, 1H), 6.90 (s, 1H), 6.75-6.73 (m, 1H), 3.78 (s, 3H), 2.95 (s, 6H), 2.76 (brs, 2H), 2.62-2.58 (m, 2H), 1.17-1.14 (m, 3H), 1.11-1.08 (m, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 154.9, 154.7, 138.4, 129.9, 129.3, 122.2, 116.8, 114.0, 111.5, 111.1, 101.3, 55.9, 37.7, 36.4, 18.2, 17.3, 15.4, 14.9.

HRMS (ESI+) Calcd for $C_{16}H_{22}N_2O_2$ $[M+Na]^+$ 297.1579; Found 297.1579.

***N, N*-Dimethyl-2,3-Dipropyl-4-Methoxyindole-1-Carboxamide (4b):** 86 mg, Colorless Liquid, **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 6.98 (d, *J*=5 Hz, 1H), 6.88 (s, 1H), 6.76-6.72 (m, 1H), 3.78 (s, 3H), 2.95 (s, 4H), 2.73 (brs, 2H), 2.56-2.53 (m, 3H), 1.58-1.54 (m, 3H), 1.48-1.44 (m, 2H), 0.91-0.84 (m, 6H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 155.0, 154.7, 137.4, 130.0, 129.7, 116.0, 111.5, 111.1, 101.6, 56.0, 37.9, 34.0, 26.9, 26.3, 23.7, 23.4, 14.4, 14.1 26.9, 26.3, 23.7, 23.4, 14.4, 14.1.

HRMS (ESI+) Calcd for $C_{18}H_{26}N_2O_2$ $[M+Na]^+$ 325.1892; Found 325.1892.

***N, N*-Dimethyl-2,3-Dibutyl-4-Methoxyindole-1-Carboxamide (4c):** 89 mg, Colorless Liquid, **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.07 (d, *J*=10 Hz, 1H), 6.96 (s, 1H), 6.82-6.80 (m, 1H), 3.86 (s, 3H), 3.02 (s, 6H), 2.83 (brs, 2H), 2.64 (t, *J*=5Hz, 2H), 1.61-1.58 (m, 2H), 1.51-1.48 (m, 2H), 1.43-1.32 (m, 4H), 0.96-0.90 (m, 6H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 155.0, 154.7, 135.7, 130.0, 129.7, 116.0, 111.5, 110.9, 101.6, 55.9, 37.7, 32.8, 32.3, 24.0, 22.9, 22.5, 14.1, 14.0.

HRMS (ESI+) Calcd for $C_{20}H_{30}N_2O_2$ $[M+Na]^+$ 353.2205; Found 353.2205.

***N,N*-Dimethyl-2,3-(*p*-Methylphenyl)-4-Methoxyindole-1-Carboxamide (4d):** 88 mg, Colorless Solid, mp-94^{0}C; **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.33 (d, *J*=5Hz, 1H), 7.18 (s, 1H), 7.15-7.07 (m, 5H), 7.03-7.01 (m, 3H), 6.87 (dd, *J*=5Hz, 1H), 3.74 (s, 3H), 2.90 (brs, 3H), 2.46 (brs, 3H), 2.30 (s, 3H), 2.27 (s, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 155.5, 154.3, 137.8, 136.0, 130.8, 130.0, 129.4, 129.3, 129.1, 128.6, 117.3, 114.2, 113.4, 112.3, 55.9, 38.1, 36.6, 21.5, 21.4.

HRMS (ESI+) Calcd for $C_{26}H_{26}N_2O_2$ $[M+Na]^+$ 421.1892; Found 421.1891.

***N, N*-Dimethyl-2,3-(*p*-Methoxyphenyl)-4-Methoxyindole-1-Carboxamide (4e):** 89 mg, Yellow Solid, mp-112^{0}C; **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.41 (d, *J*=10 Hz, 1H), 7.26 (m, 4H), 7.08 (s, 1H), 6.95-6.90 (m, 3H), 6.84-6.83 (m, 2H), 3.82 (s, 9H), 2.98 (s, 3H), 2.53 (s, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 159.3, 158.2, 155.5, 154.4, 135.3, 131.2, 130.8, 130.7, 129.2, 126.7, 123.9, 114.1, 113.8, 113.2, 112.3, 101.6, 56.0, 55.3, 37.9, 36.6.

HRMS (ESI+) Calcd for $C_{26}H_{26}N_2O_2$ $[M+Na]^+$ 453.1790; Found 453.1789.

***N, N*-Dimethyl-2,3-(*p*-Chlorophenyl)-4-Methoxyindole-1-Carboxamide (4f):** 80 mg, Yellow Solid, mp-129^{0}C; **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.39 (d, *J*=10 Hz, 1H), 7.34-7.28 (m, 4H), 7.26-7.22 (m, 4H), 7.02 (s, 1H), 6.99 (m, 1H), 3.82 (s, 3H), 3.00 (brs, 3H), 2.60 (brs, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 155.9, 153.8, 134.5, 134.3, 132.7, 132.4, 131.3, 130.8, 129.6, 129.0, 128.9, 128.5, 117.0, 114.1, 112.5, 101.4, 56.0, 37.9, 36.6.

HRMS (ESI+) Calcd for $C_{24}H_{20}Cl_2N_2O_2$ $[M+Na]^+$ 461.0800; Found 461.0800.

***N, N*-Dimethyl-2,3-(2-Naphthalene)-4-Methoxyindole-1-Carboxamide (4g):** 75 mg,Yellow Solid, mp-146^{0}C; **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.95 (s, 1H), 7.88 (s, 1H), 7.83-7.79 (m, 3H), 7.74 (d, *J*=10Hz, 2H), 7.70 (d, *J*=10 Hz, 1H), 7.51-7.45 (m, 5H), 7.41 (m, 1H), 7.33 (m, 1H), 7.20 (m, 1H), 7.02 (m, 1H), 3.83 (s, 3H), 2.94 (brs, 3H), 2.52 (brs, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 155.8, 154.2, 133.7, 133.2, 132.8, 132.3, 131.9, 131.1, 129.1, 128.9, 128.7, 128.3, 128.1, 128.0, 127.9, 127.8, 127.3, 126.6, 126.4, 126.1, 125.8, 117.9, 113.8, 112.5, 101.7, 56.0, 37.9, 36.5.

HRMS (ESI+) Calcd for $C_{32}H_{26}N_2O_2$ $[M+Na]^+$ 493.5508; Found 493.5508.

***N, N*-Dimethyl-2,3-(1-Naphthalene)-4-Methoxyindole-1-Carboxamide (4h):** 63 mg, Yellow liquid, **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.92-7.90 (m, 1H), 7.84-7.82 (m, 1H), 7.78-7.76 (m, 1H), 7.73-7.60 (m, 3H), 7.67 (d, *J*=10 Hz, 1H), 7.55 (d, *J*=10Hz, 1H), 7.48-7.39 (m, 4H), 7.26-7.19 (m, 4H), 7.04-7.02 (m, 1H), 6.73-6.70 (m, 1H), 3.67 (s, 3H), 2.80 (s, 3H), 2.57 (s, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 155.5, 154.0, 130.9, 129.0, 128.9, 128.3, 127.6, 127.5, 126.5, 125.8, 125.7, 125.7, 125.6, 113.8, 113.8, 102.4, 55.9, 37.7.

HRMS (ESI+) Calcd for $C_{32}H_{26}N_2O_2$ $[M+Na]^+$ 493.5508; Found 493.5508.

***N, N*-Dimethyl-2-Phenyl-3-Ethyl-4-Methoxyindole-1-Carboxamide (4i):** White Liquid,

^{1}H NMR (500MHz, $CDCl_3$) δ (ppm): 7.43-7.42 (m, 3H), 7.39-7.35 (m, 3H), 7.06 (s, 1H), 6.93-6.91 (m, 1H), 3.88 (s, 3H), 2.78 (s, 6H), 1.30-1.27 (m, 5H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 155.0, 154.4, 135.4, 132.0, 131.0, 129.3, 128.9, 128.4, 127.9, 118.5, 112.8, 112.5, 101.6, 55.9, 37.2, 17.7, 15.4.

HRMS (ESI+) Calcd for $C_{20}H_{22}N_2O_2$ $[M+Na]^+$ 345.1579; Found 345.1780.

***N, N*-Dimethyl-2,3-Dithiophene-4-Methoxyindole-1-Carboxamide (4j):** 66 mg, Yellow Solid, **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.38-7.34 (m, 1H), 7.31-7.29 (m, 1H), 7.26 (s, 1H), 7.17-7.16 (m, 2H), 7.11-7.09 (m, 2H), 7.05-7.04 (m, 1H), 6.98 (m, 1H), 3.84 (s, 3H), 3.07 (brs, 3H), 2.73 (brs, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 155.7, 153.4, 134.7, 131.4, 130.4, 129.7, 128.8, 128.7, 127.5, 127.3, 127.2, 127.1, 125.5, 114.3, 111.9, 101.6, 55.9, 38.3, 36.8.

HRMS (ESI+) Calcd for $C_{20}H_{18}N_2O_2S_2$ $[M+Na]^+$ 405.0707; Found 405.0707.

***N, N*-Dimethyl-2-Phenyl-3-Thiophene-4-Methoxyindole-1-Carboxamide (4k):** 67 mg, Yellow Liquid, **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.42-7.39 (m, 3H), 7.37-7.32 (m, 2H), 7.29-7.28 (m, 1H), 7.26-7.24 (m, 1H), 7.04-7.03 (m, 1H), 7.01 (d, *J*=5Hz, 1H), 6.99-6.95 (m, 2H), 3.80 (s, 3H), 3.09 (s, 3H), 2.70 (s, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 154.9, 134.7, 134.4, 134.1, 131.2, 130.3, 128.4, 128.4, 128.3, 128.2, 126.3, 125.7, 121.6, 121.5, 117.8, 117.4, 37.9, 36.4, 18.1.

HRMS (ESI+) Calcd for $C_{22}H_{20}N_2O_2S$ $[M+Na]^+$ 399.1143; Found 399.1145.

***N, N*-Dimethyl-2-Phenyl-3-propyl-4-Methoxyindole-1-Carboxamide (4l):** 86 mg, Yellow Liquid, **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.34-7.33 (m, 4H), 7.30 (d, J=5 Hz, 1H), 7.27-7.25 (m, 1H), 6.95 (s, 1H), 6.84-6.81 (m, 1H), 3.78 (s, 3H), 2.64-2.62 (m, 6H), 1.95-1.94 (m, 2H), 1.62-1.59 (m, 2H), 0.85-0.82 (t, J=7.5 Hz, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 154.9, 154.5, 135.8, 132.0, 130.9, 129.5, 129.0, 128.3, 127.8, 117.0, 112.7, 112.4, 101.7, 55.9, 37.1, 26.4, 23.8, 14.3.

HRMS (ESI+) Calcd for $C_{21}H_{24}N_2O_2$ $[M+Na]^+$ 337.1916; Found 337.1915.

***N, N*-Dimethyl-2-Phenyl-3-butyl-4-Methoxyindole-1-Carboxamide (4m):** 89 mg, Yellow Liquid, **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.32-7.31 (m, 4H), 7.29 (d, J=10 Hz, 1H), 7.25-7.22 (m, 1H), 6.95 (s, 1H), 6.82-6.80 (m, 1H), 3.75 (s, 3H), 2.65-2.62 (m, 6H), 1.56-1.53 (m, 2H), 1.26-1.21 (m, 2H), 0.78-0.75 (t, J=7.5 Hz, 3H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 154.8, 154.4, 135.6, 131.9, 130.8, 129.4, 128.9, 128.2, 127.7, 117.0, 112.5, 112.3, 101.6, 55.7, 37.2, 32.7, 23.9, 22.6, 13.8.

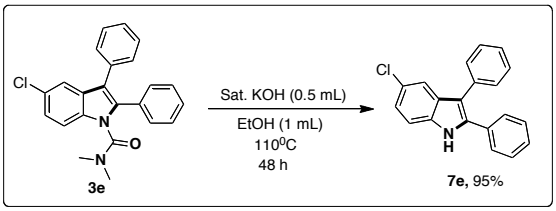
HRMS (ESI+) Calcd for $C_{22}H_{27}N_2O_2$ $[M+Na]^+$ 351.2073; Found 351.2071.

(2,3-Diphenyl-4-Methoxyndole-1-yl)(Pyrrolidin-1-yl)Methanone (6a): 90 mg, Yellow Liquid, **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 7.47 (d, *J*=5 Hz, 1H), 7.37-7.28 (m, 10H), 7.11 (s, 1H), 6.98 (m, 1H), 3.83 (s, 3H), 3.68 (s, 1H), 3.41 (s, 1H), 2.99 (s, 1H), 2.82 (s, 1H), 1.85-1.52 (m, 4H).

^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ (ppm): 155.5, 152.3, 135.0, 134.4, 131.5, 130.5, 130.2, 129.5, 128.8, 128.5, 128.3, 128.0, 126.5, 117.5, 113.6, 112.3, 101.5, 55.9, 31.9, 29.1 22.7, 14.2.

HRMS (ESI+) Calcd for $C_{26}H_{24}N_2O_2$ $[M+Na]^+$ 419.1735; Found 419.1735.

IV. **Deprotection of directing group**

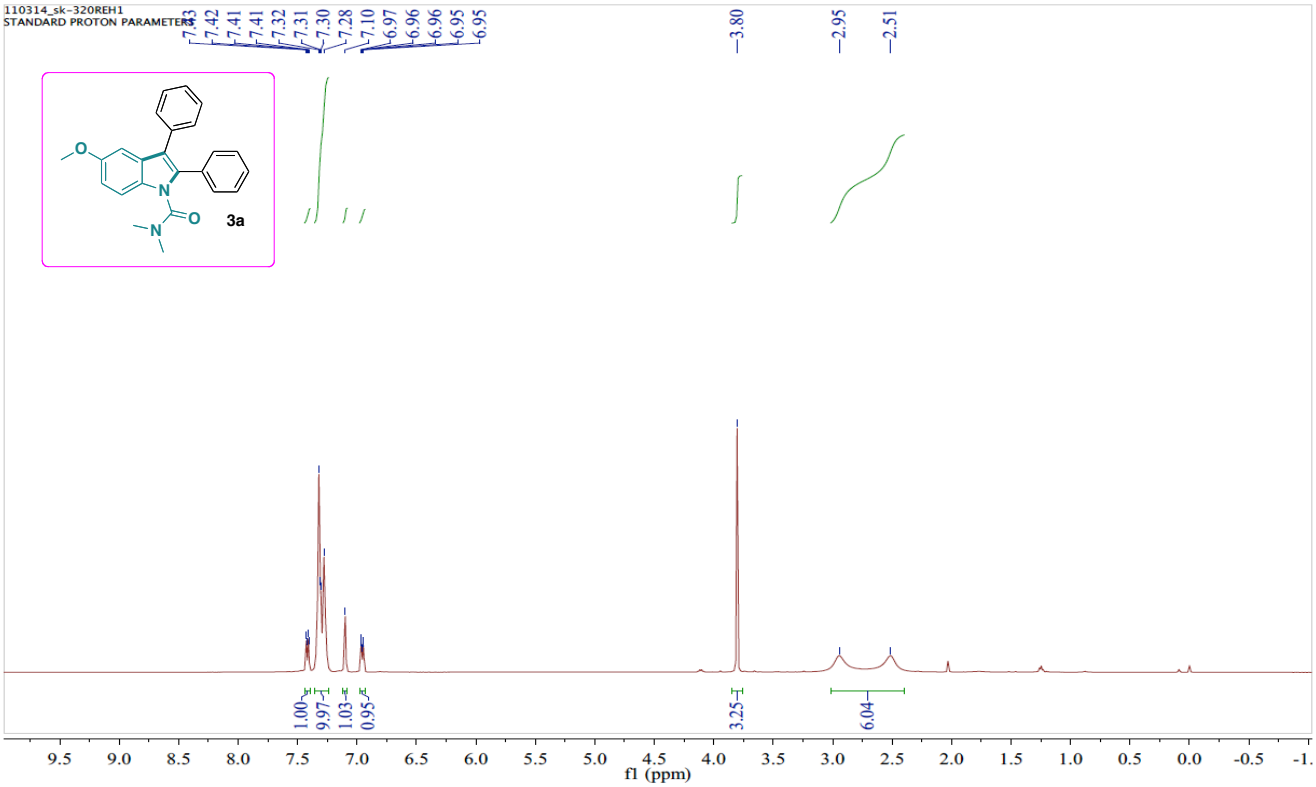


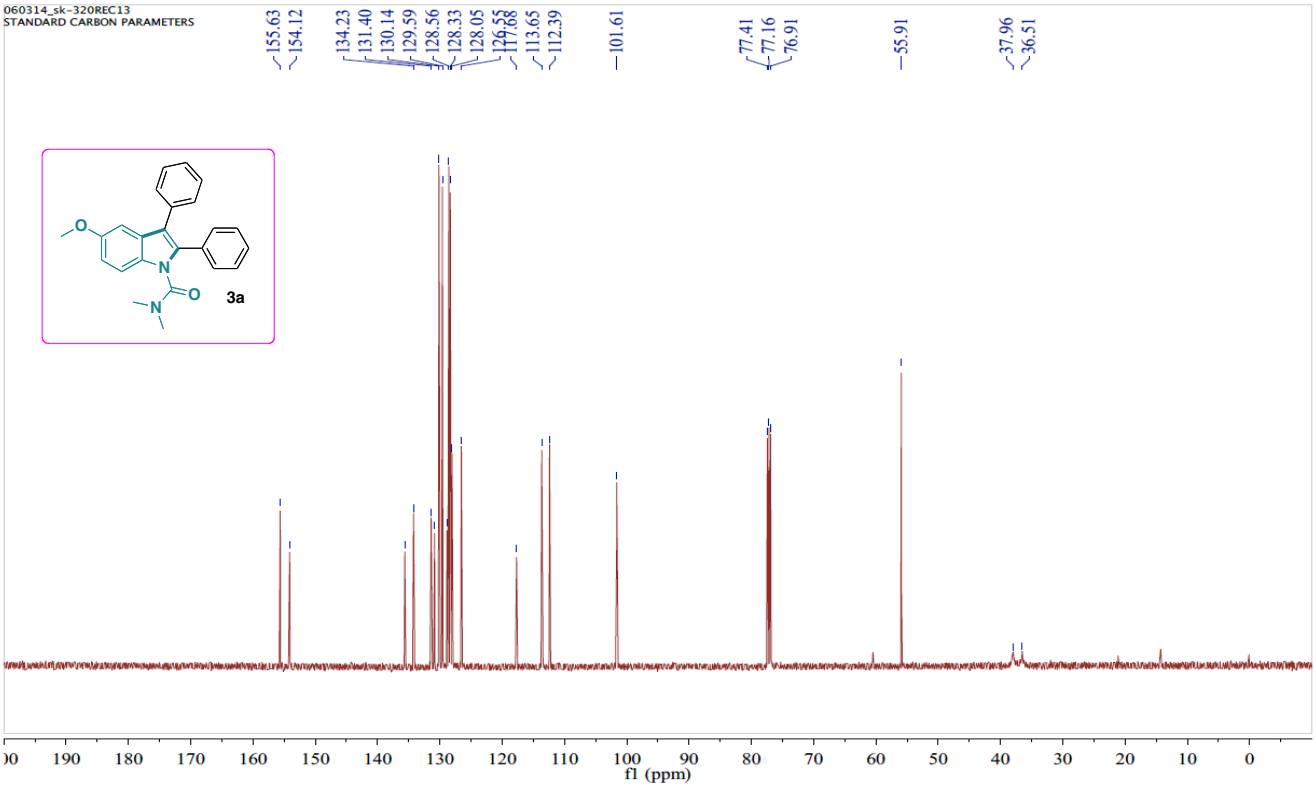
A solution of *N, N*-Dimethyl-2,3-Diphenyl-4-Chloroindole-1-Carboxamide (3e) (35.3 mg) in 3:1 EtOH/sat. aq. KOH (1.5 mL) was prepared in a 10 mL vial, closed tightly with a screw-cap, and stirred at 110ºC for 48 h. The mixture was diluted with sat. aq. NH_4Cl and extracted with CH_2Cl_2 and the aqueous layer was again extracted a second time with CH_2Cl_2. The combined organics were dried ($MgSO_4$) and concentrated in vacuum and gave the product in 95% yield without column chromatography.

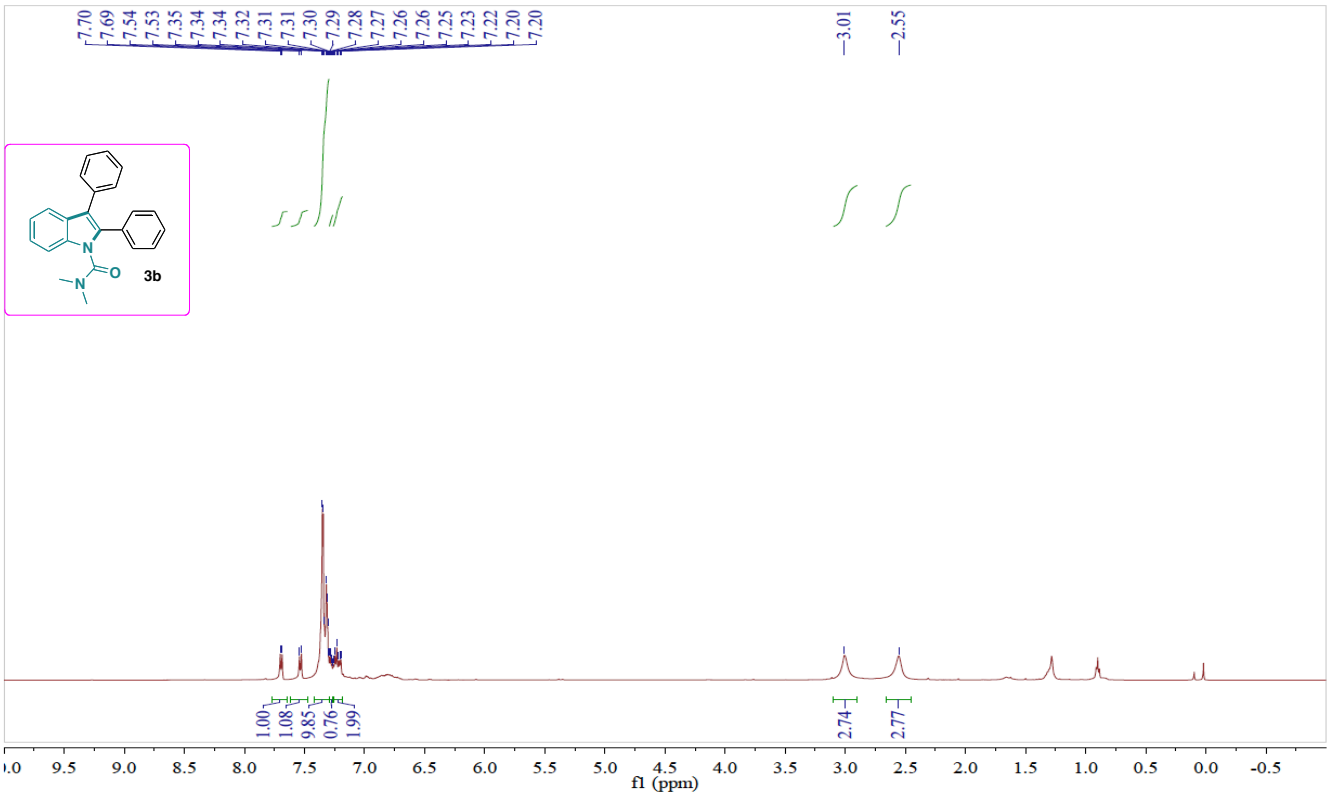
2,3-diphenyl-4-chloroindole (7e): 32 mg, Yellow Liquid, **^{1}H NMR** (500MHz, $CDCl_3$) δ (ppm): 8.32 (s, 1H), 7.64 (d, *J*=5Hz, 1H), 7.42-7.39 (5H), 7.33-7.31 (m, 5H), 7.26 (s, 1H), 7.20-7.18 (m, 1H). **^{13}C{^{1}H} NMR** (125 MHz, $CDCl_3$) δ (ppm): 135.5, 134.4, 134.3, 132.2, 130.1, 129.9, 129.2, 128.8, 128.8, 128.2, 128.1, 126.6, 126.2, 123.0, 119.2, 116.3, 114.8, 112.0.

HRMS (ESI+) Calcd for $C_{20}H_{15}ClN$ $[M+H]^+$ 304.0893; Found 304.0890.

IV. Copies of NMR Spectra



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7.26
7.25
7.23
7.22
7.20
7.20
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3b
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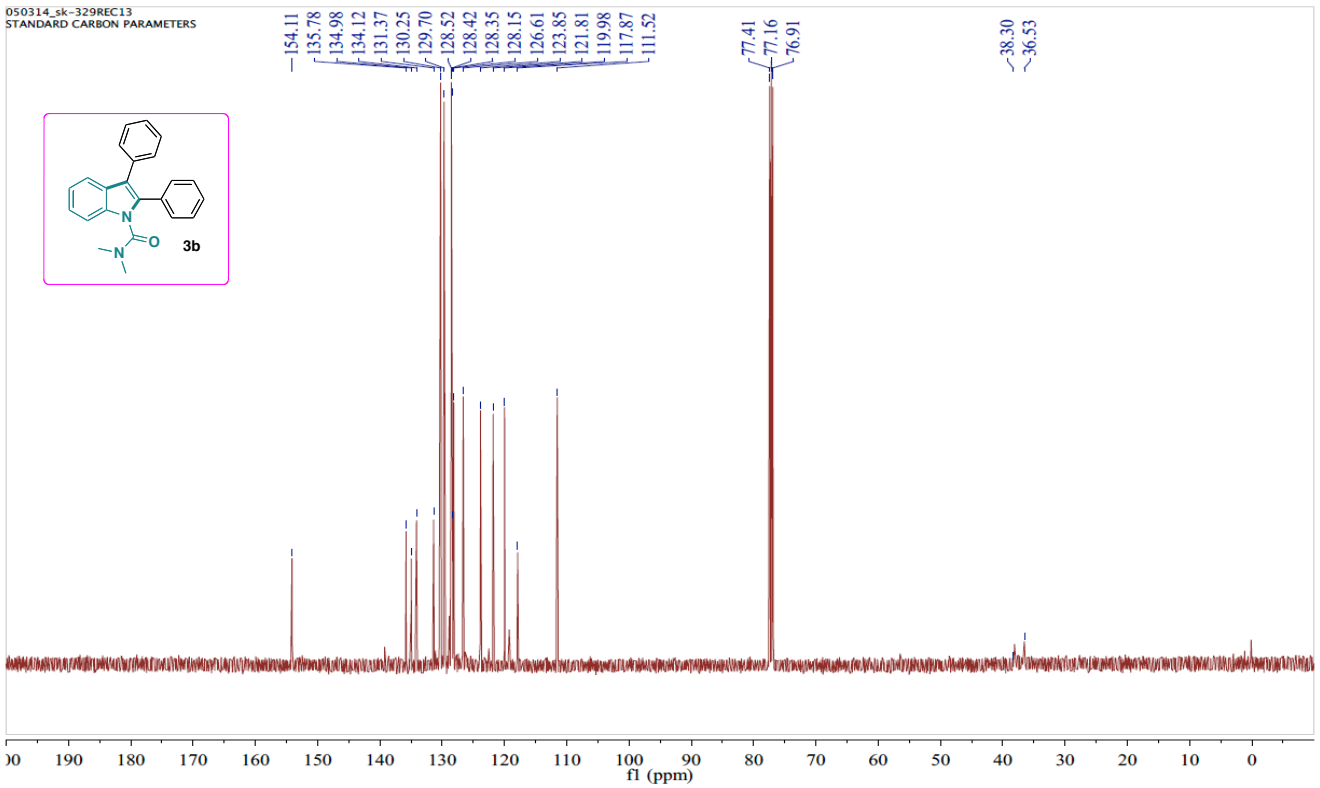
2.77
f1 (ppm)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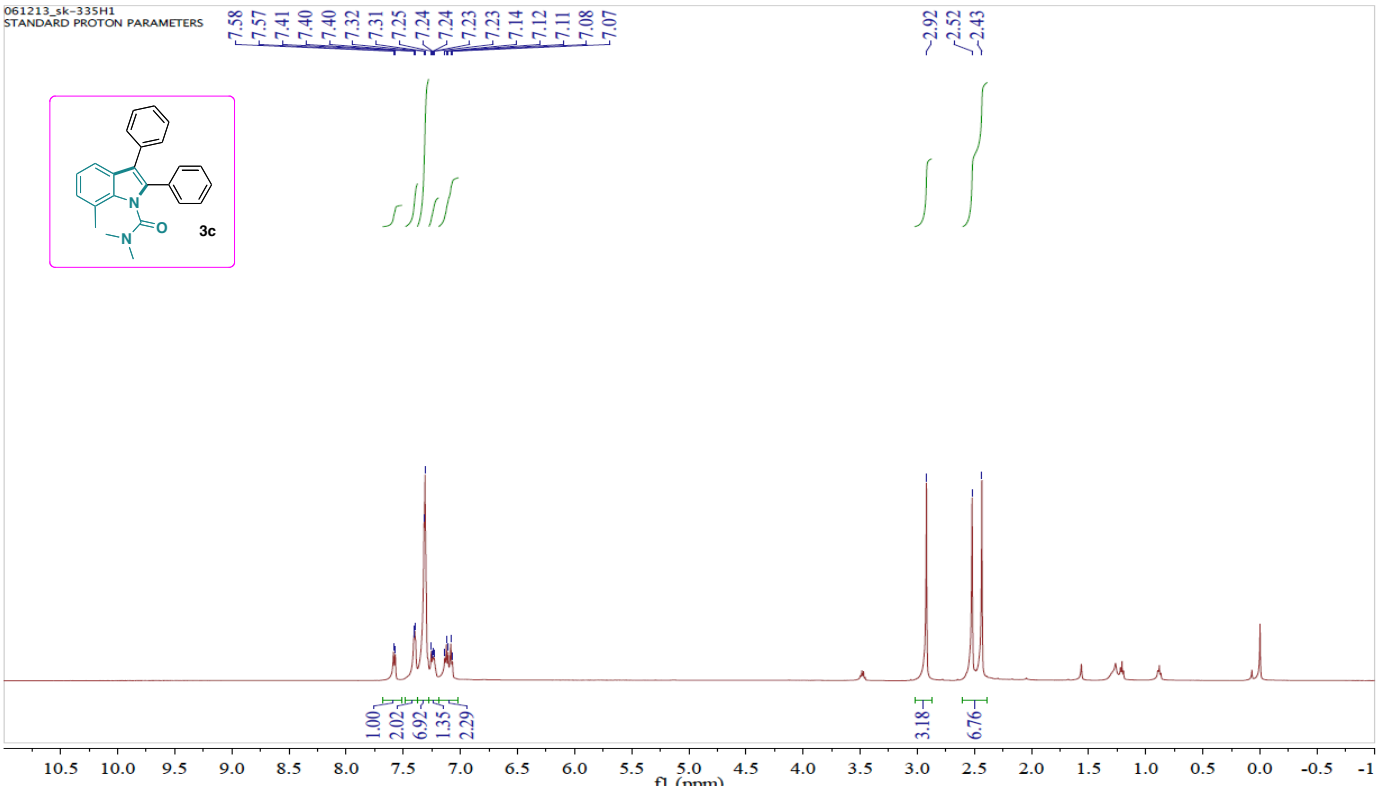
3b
f1 (ppm)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7.23
7.14
7.12
7.11
7.08
7.07
2.92
2.52
2.43
3c
1.00
2.02
6.92
1.35
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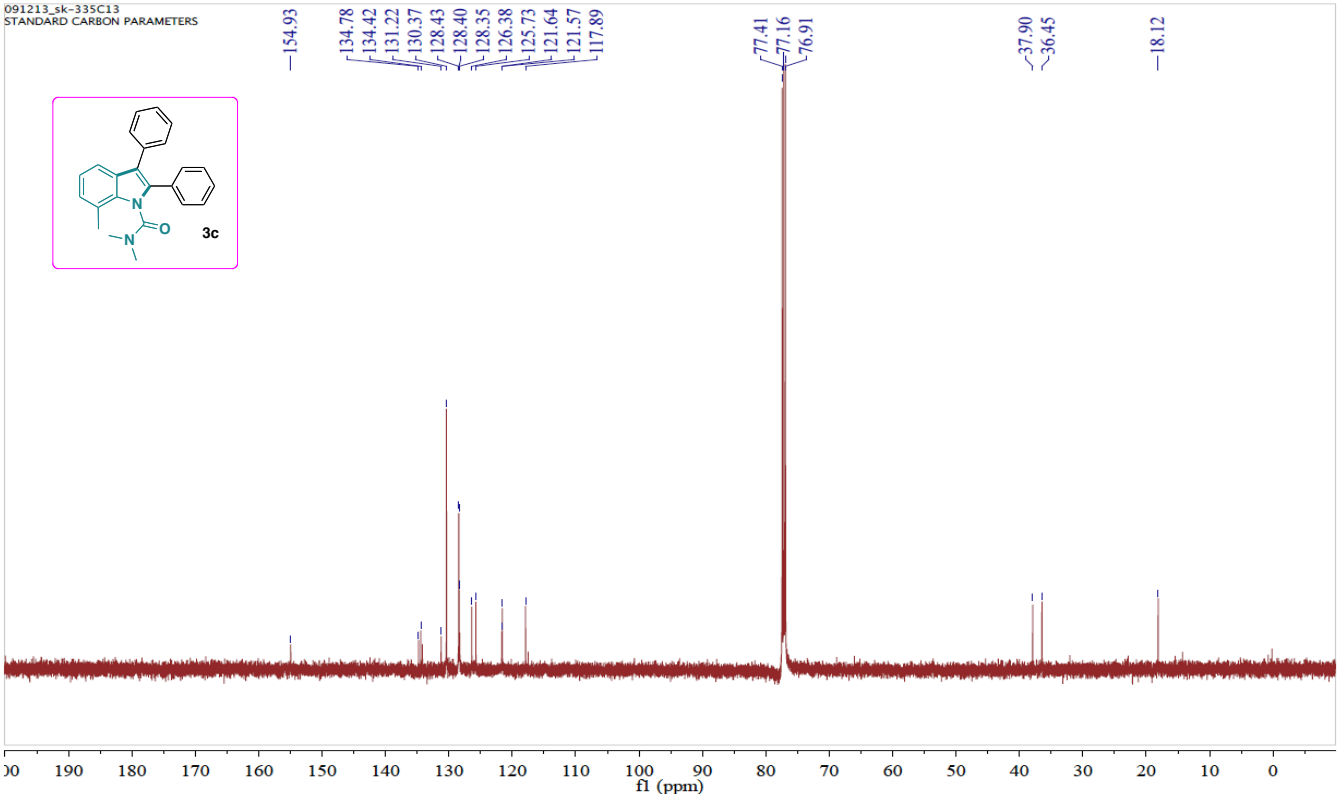
f1 (ppm)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121.57
117.89
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36.45
18.12
3c
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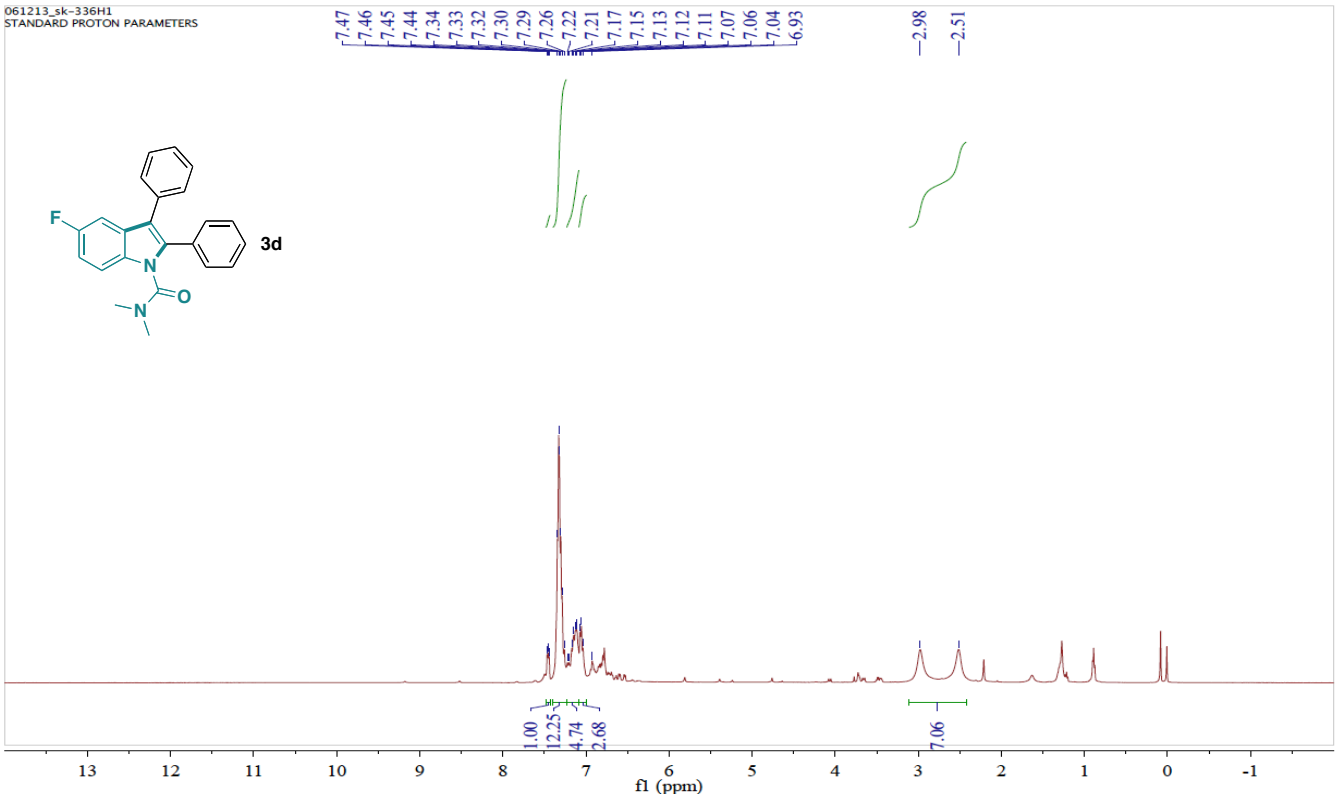
f1 (ppm)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7.21
7.17
7.15
7.13
7.12
7.11
7.07
7.06
7.04
6.93
2.98
2.51
F
N
O
N
3d
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12.25
4.74
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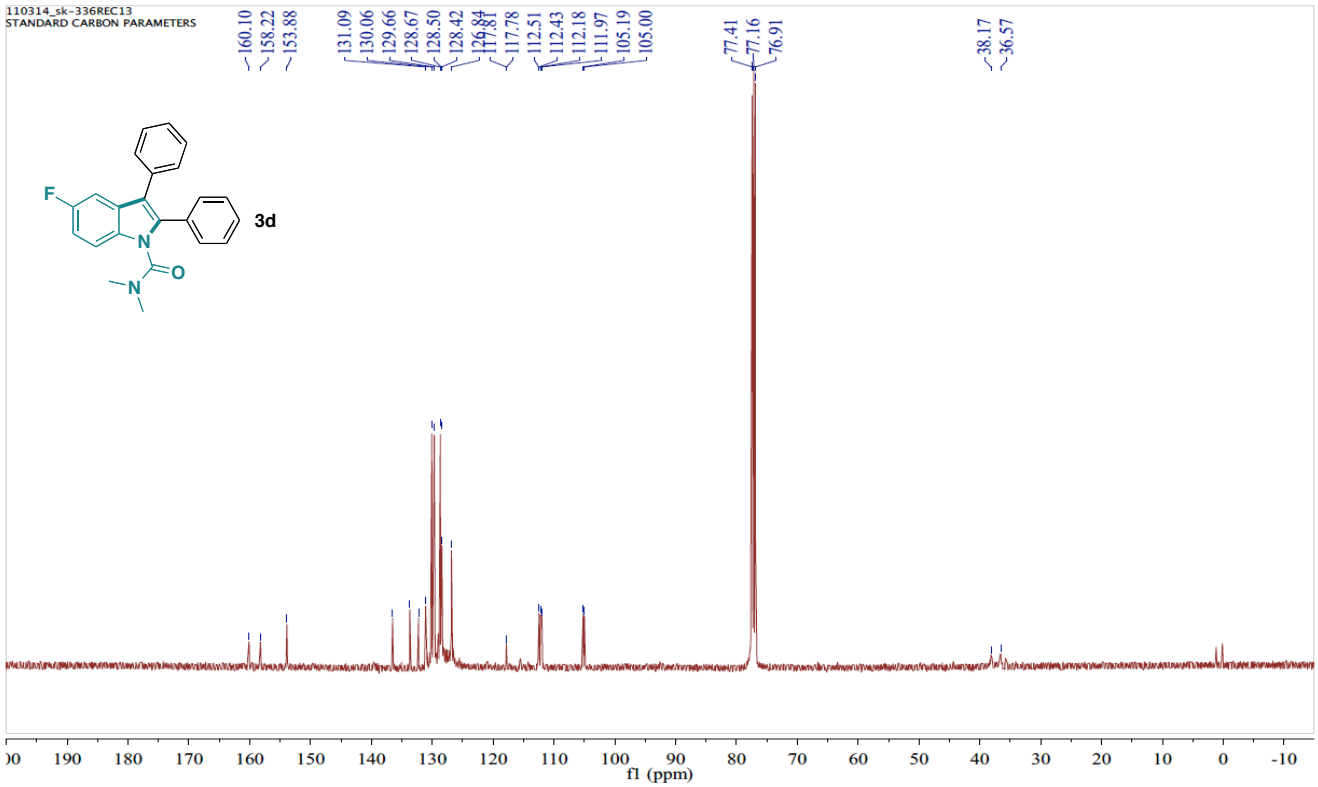
f1 (ppm)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112.18
111.97
105.19
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77.16
76.91
38.17
36.57
F
N
O
N
3d
190
180
170
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140
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120
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100
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80
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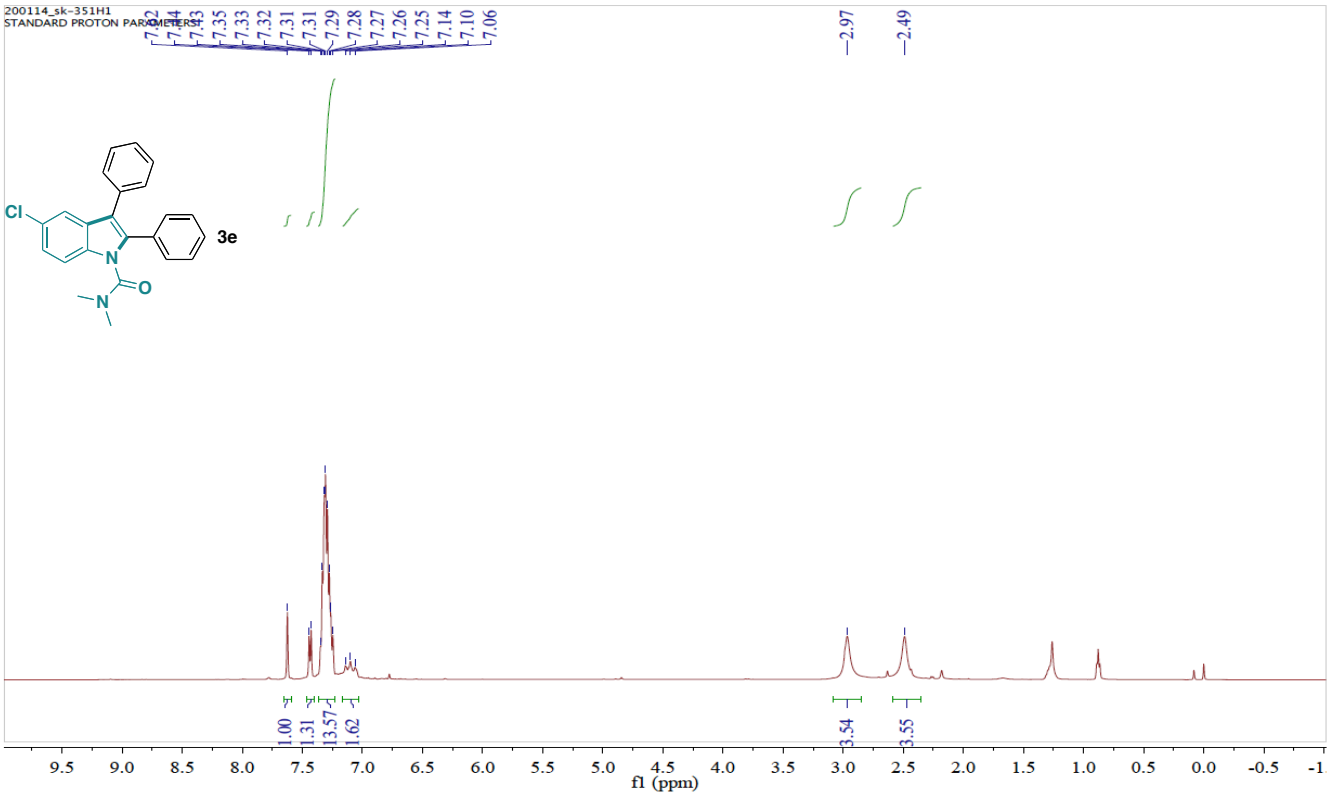
f1 (ppm)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7.27
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7.25
7.14
7.10
7.06
2.97
2.49
Cl
N
O
N
3e
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8.0
7.5
7.0
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6.0
5.5
5.0
4.5
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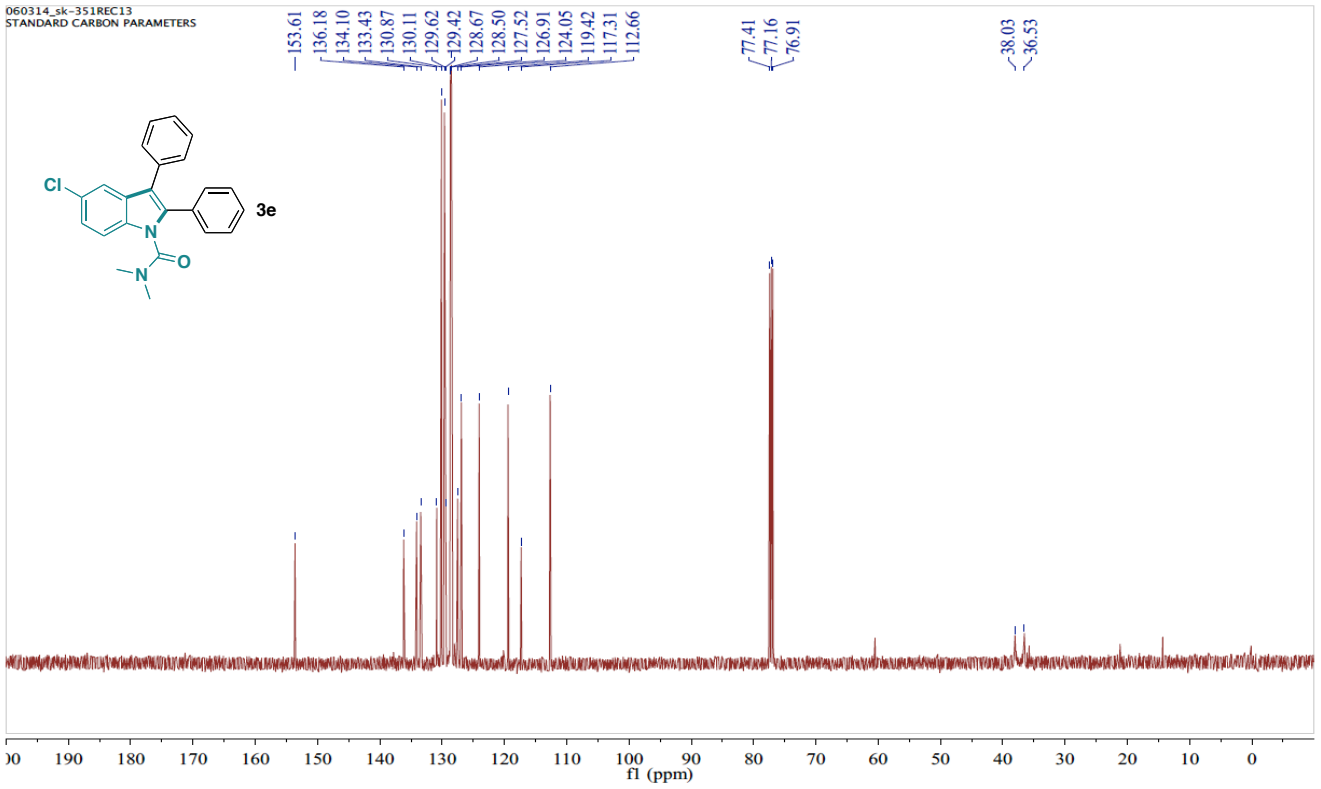
f1 (ppm)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128.50
127.52
126.91
124.05
119.42
117.31
112.66
77.41
77.16
76.91
38.03
36.53
Cl
N
O
N
3e
190
180
170
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90
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70
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50
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30
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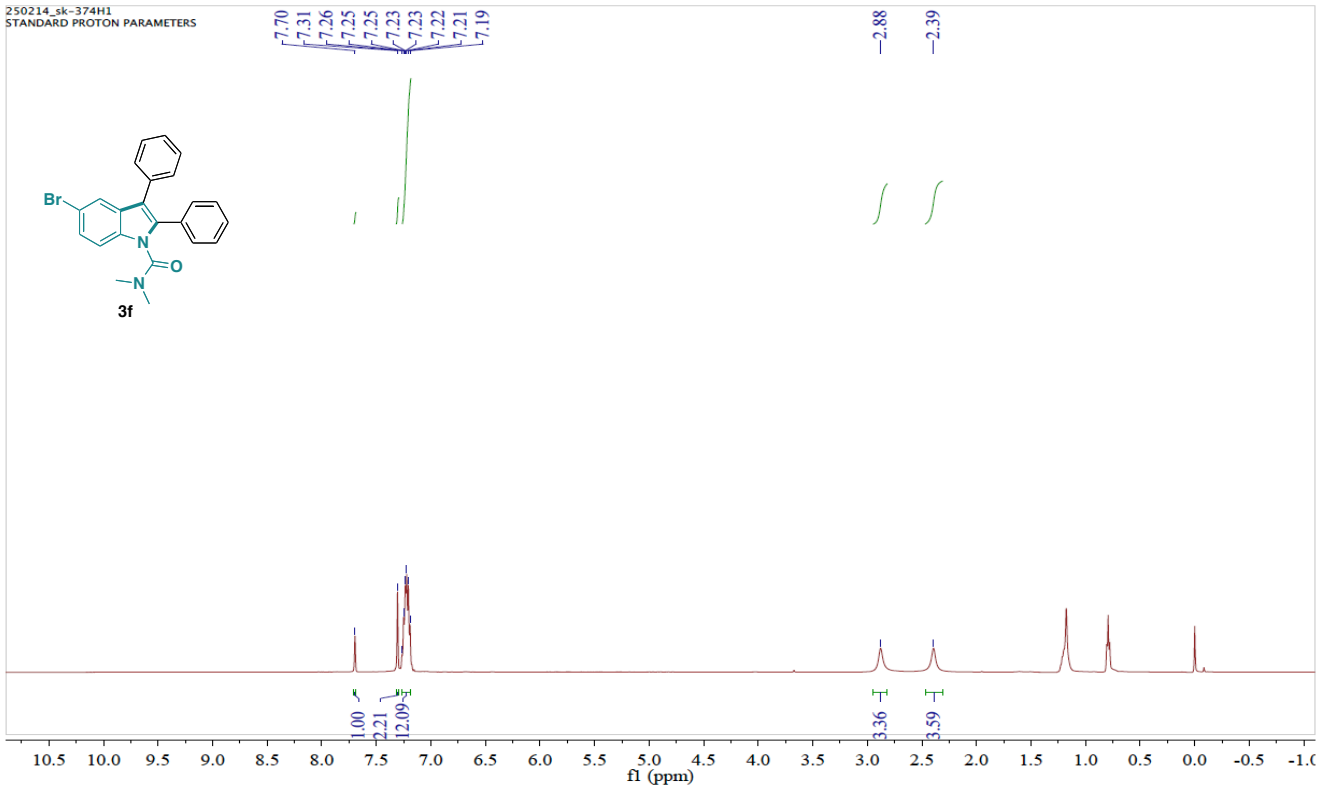
10
0
f1 (ppm)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O
N
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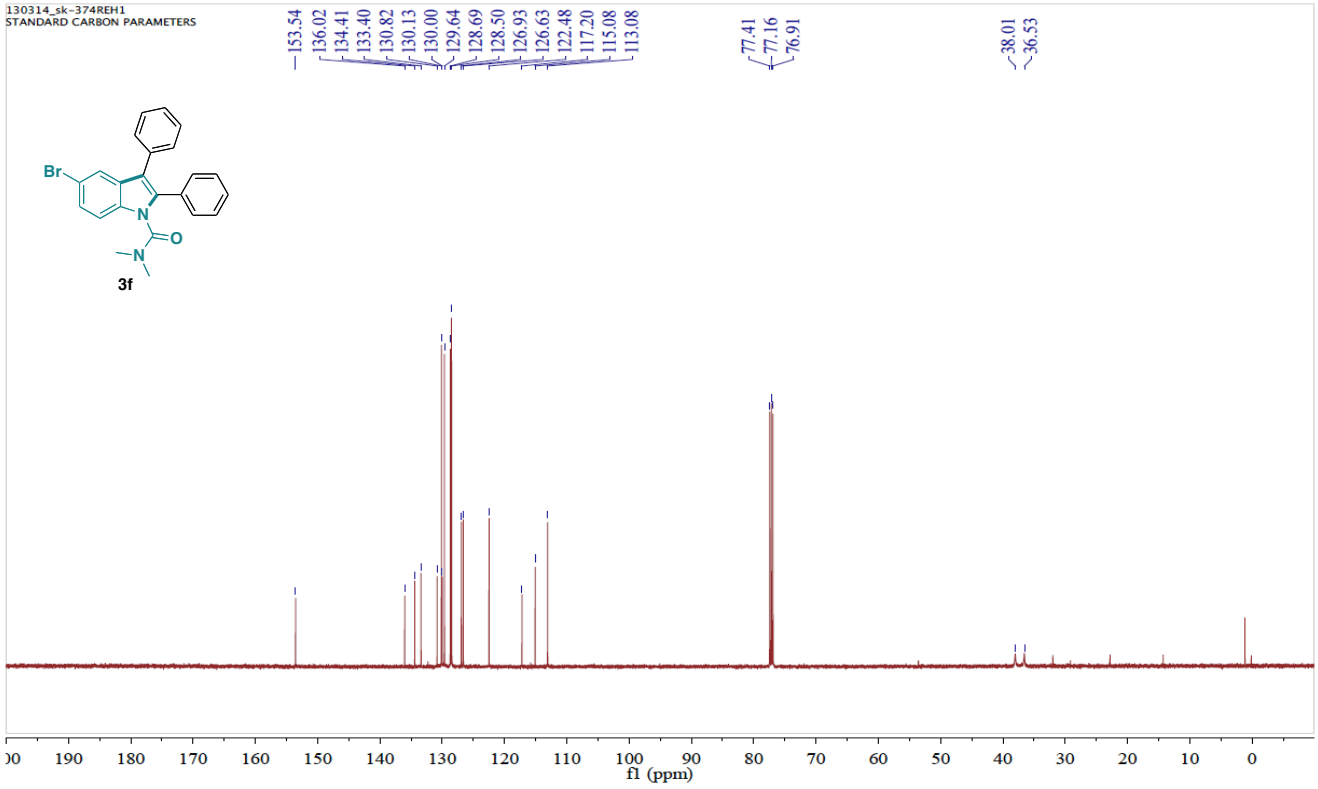
1.00
2.21
12.09
3.36
3.59
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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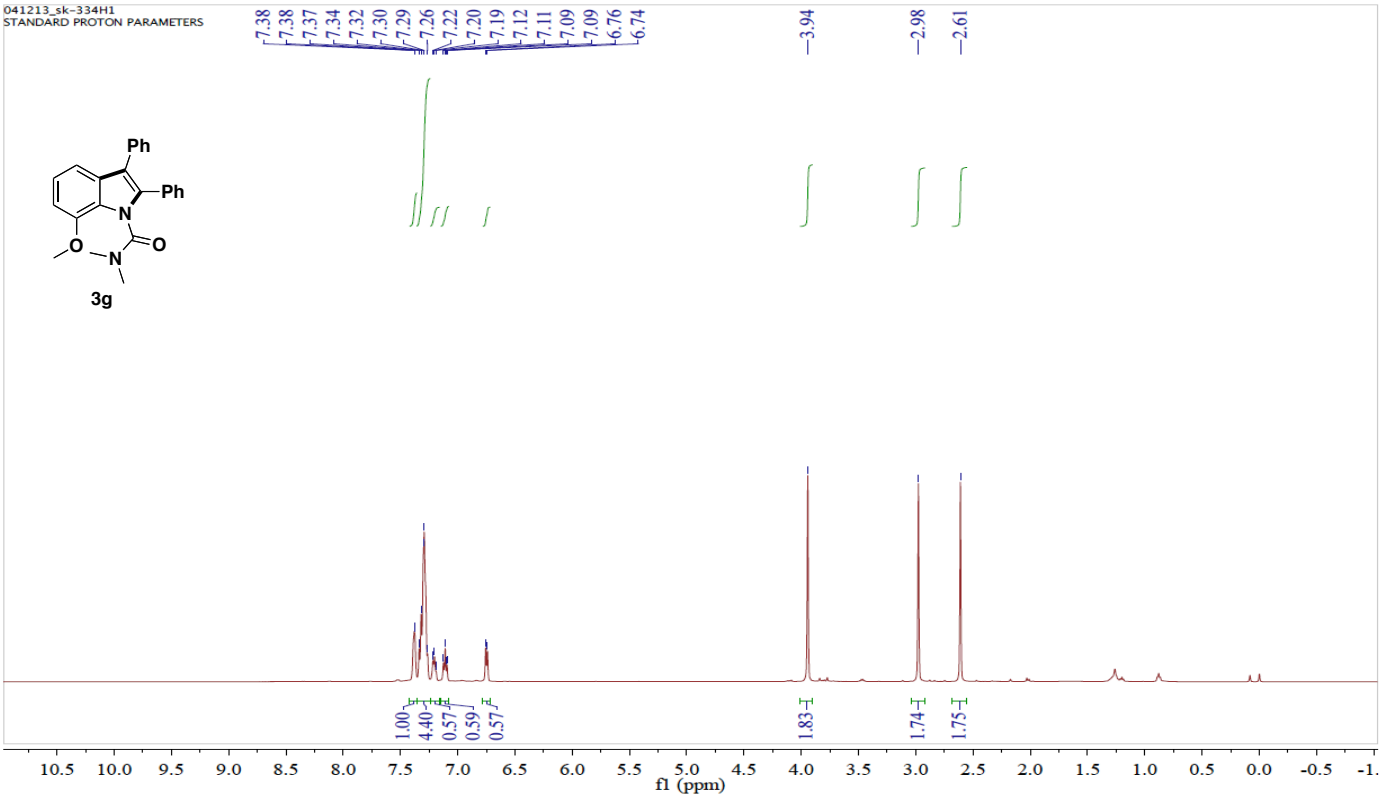
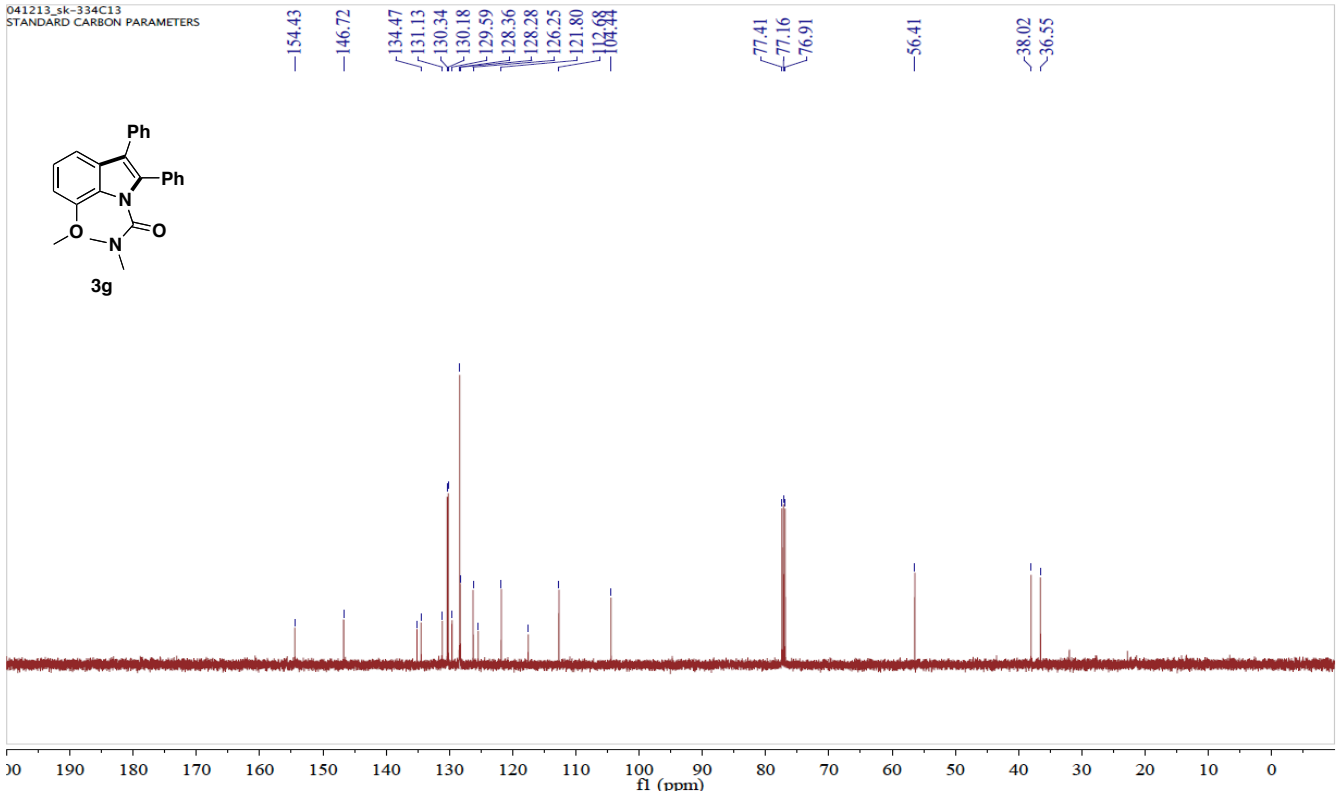
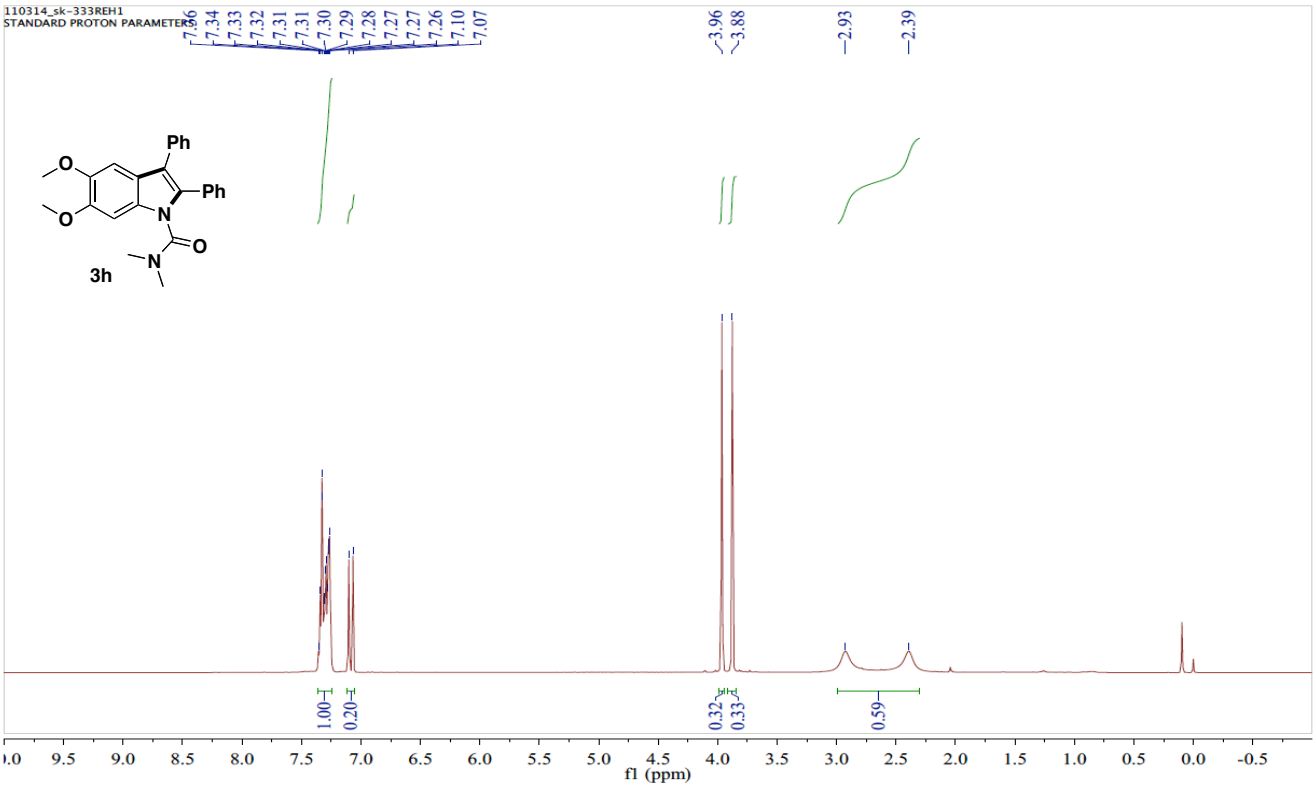
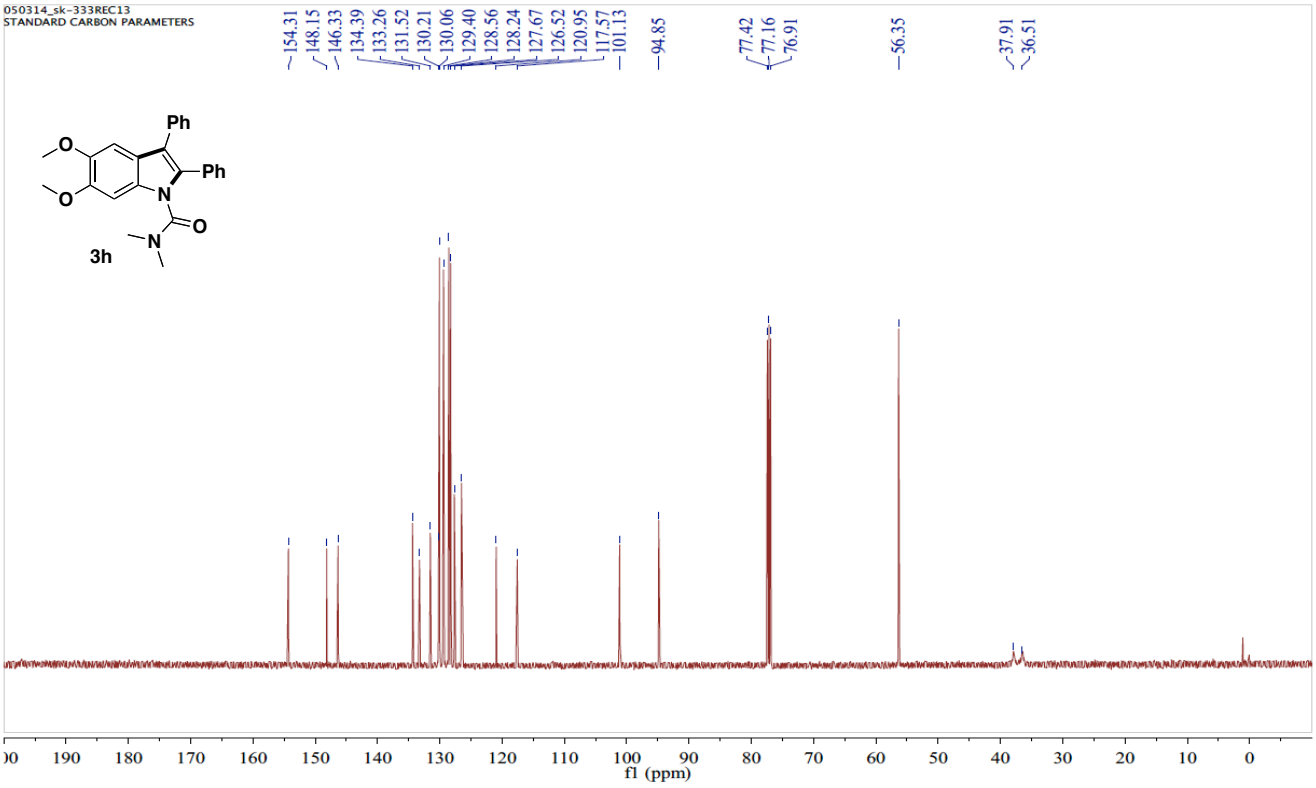
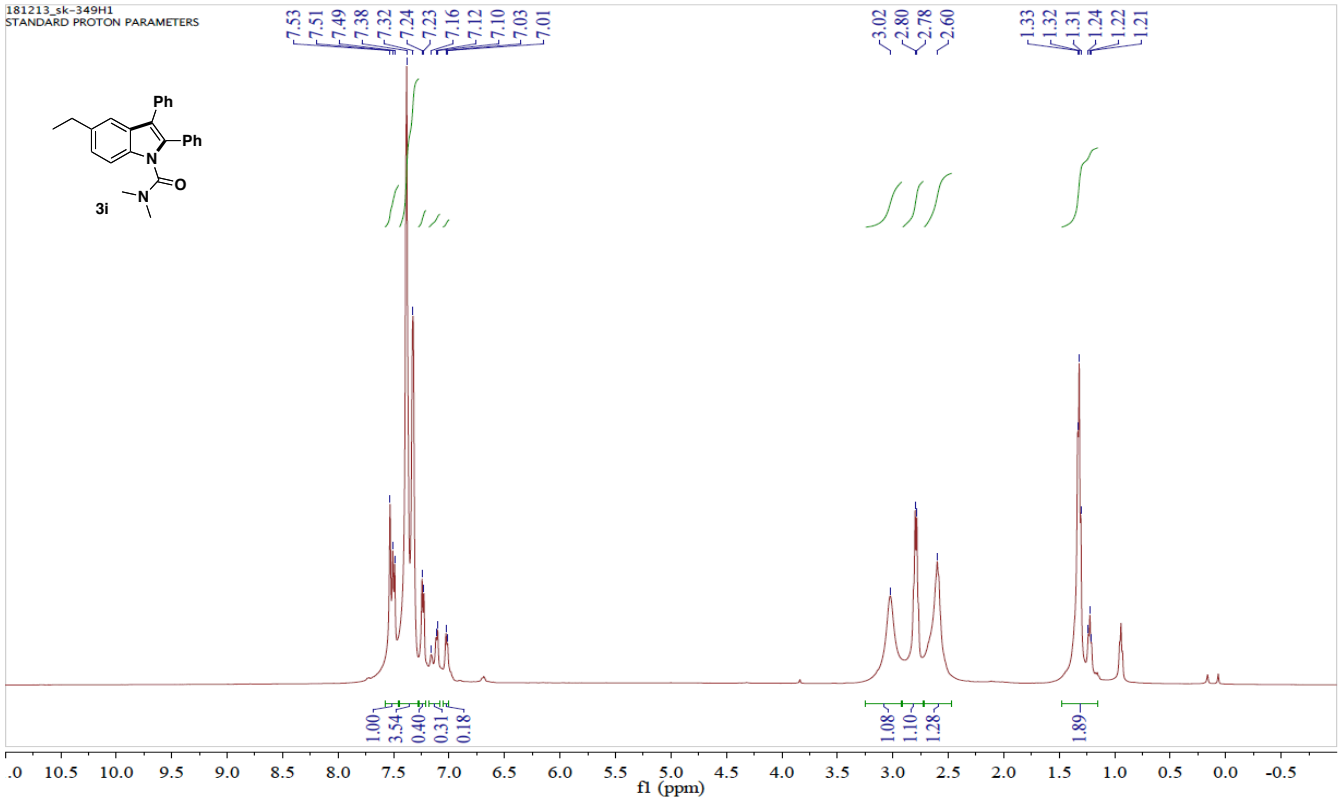
128.69
128.50
126.93
126.63
122.48
117.20
115.08
113.08
77.41
77.16
76.91
38.01
36.53
Br
N
O
N
3f
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

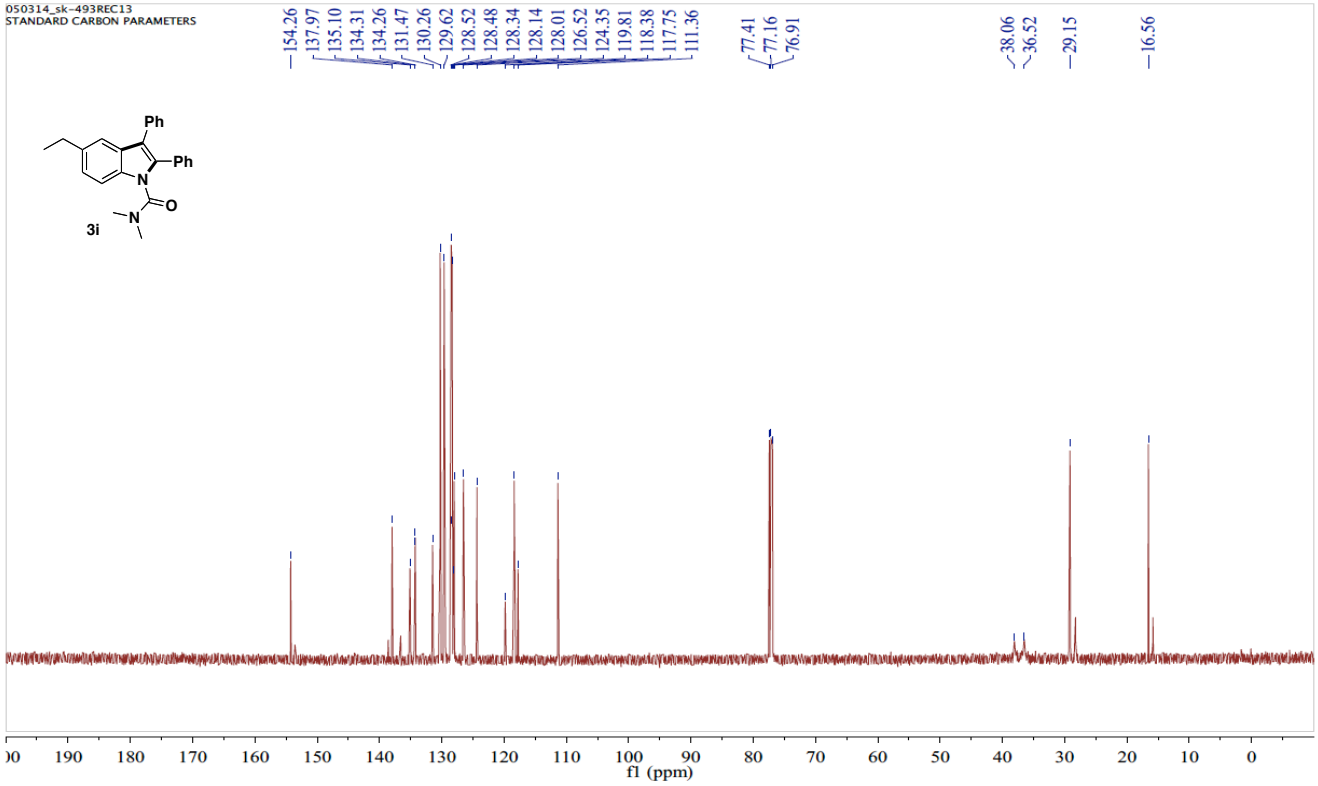

041213_sk-334H1
STANDARD PROTON PARAMETERS
7.38
7.38
7.37
7.34
7.32
7.30
7.29
7.26
7.22
7.20
7.19
7.12
7.11
7.09
7.09
6.76
6.74
3.94
2.98
2.61
Ph
Ph
N
O
O
N
3g
1.00
4.40
0.57
0.59
0.57
1.83
1.74
1.75
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.
f1 (ppm)

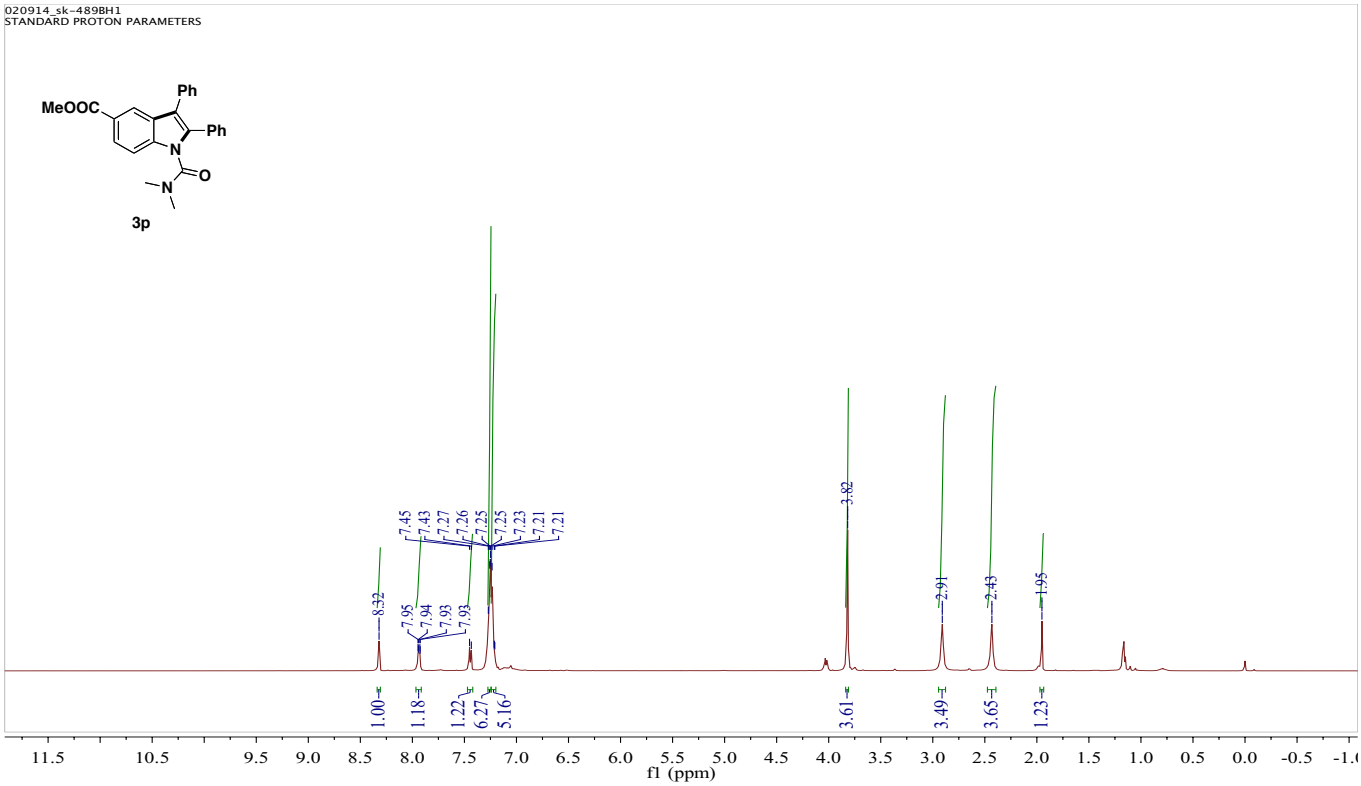

041213_sk-334C13
STANDARD CARBON PARAMETERS
154.43
146.72
134.47
131.13
130.34
130.18
129.59
128.36
128.28
126.25
121.80
112.68
104.44
77.41
77.16
76.91
56.41
38.02
36.55
Ph
Ph
N
O
O
N
3g
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

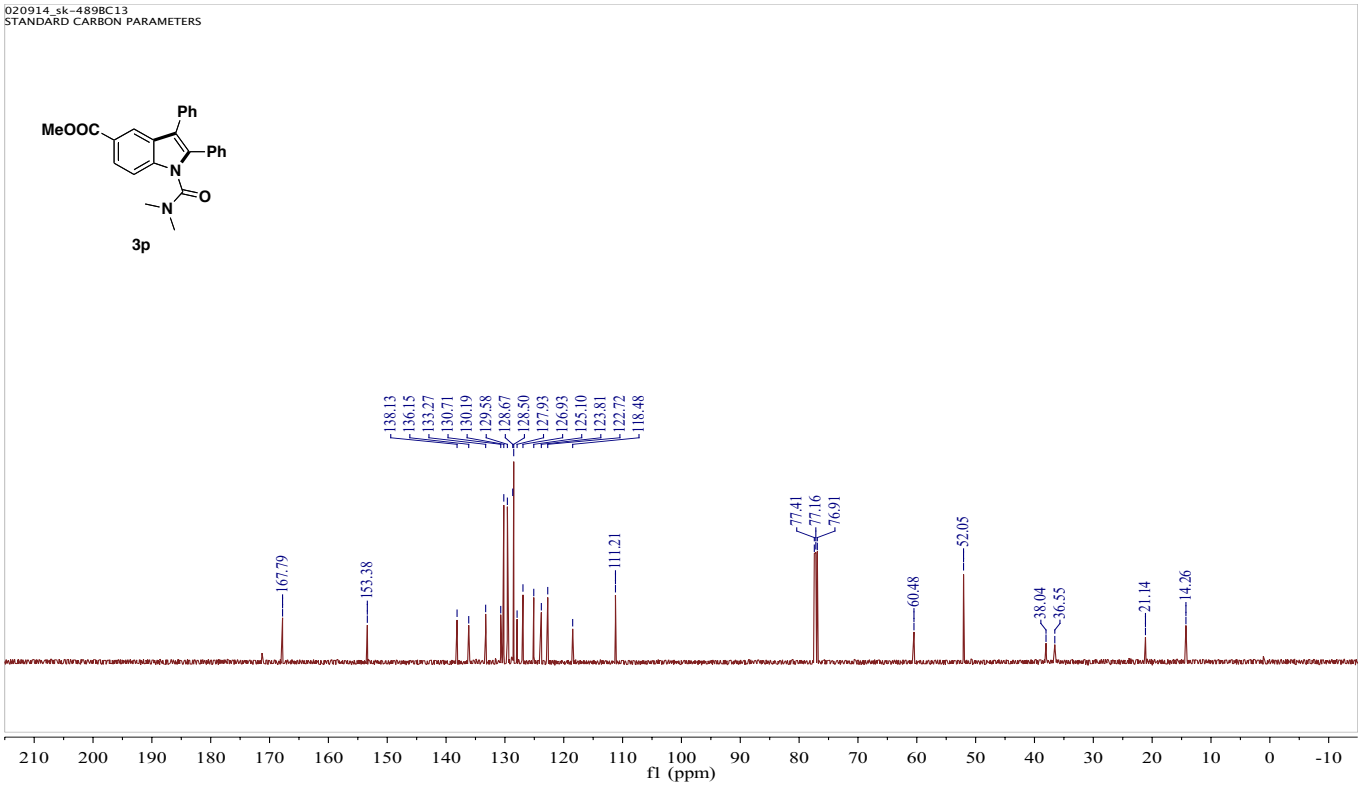

110314_sk-333REH1
STANDARD PROTON PARAMETERS
7.36
7.34
7.33
7.32
7.31
7.31
7.30
7.29
7.28
7.27
7.27
7.26
7.10
7.07
3.96
3.88
2.93
2.39
Ph
Ph
O
O
N
O
N
3h
1.00
0.20
0.32
0.33
0.59
fl (ppm)

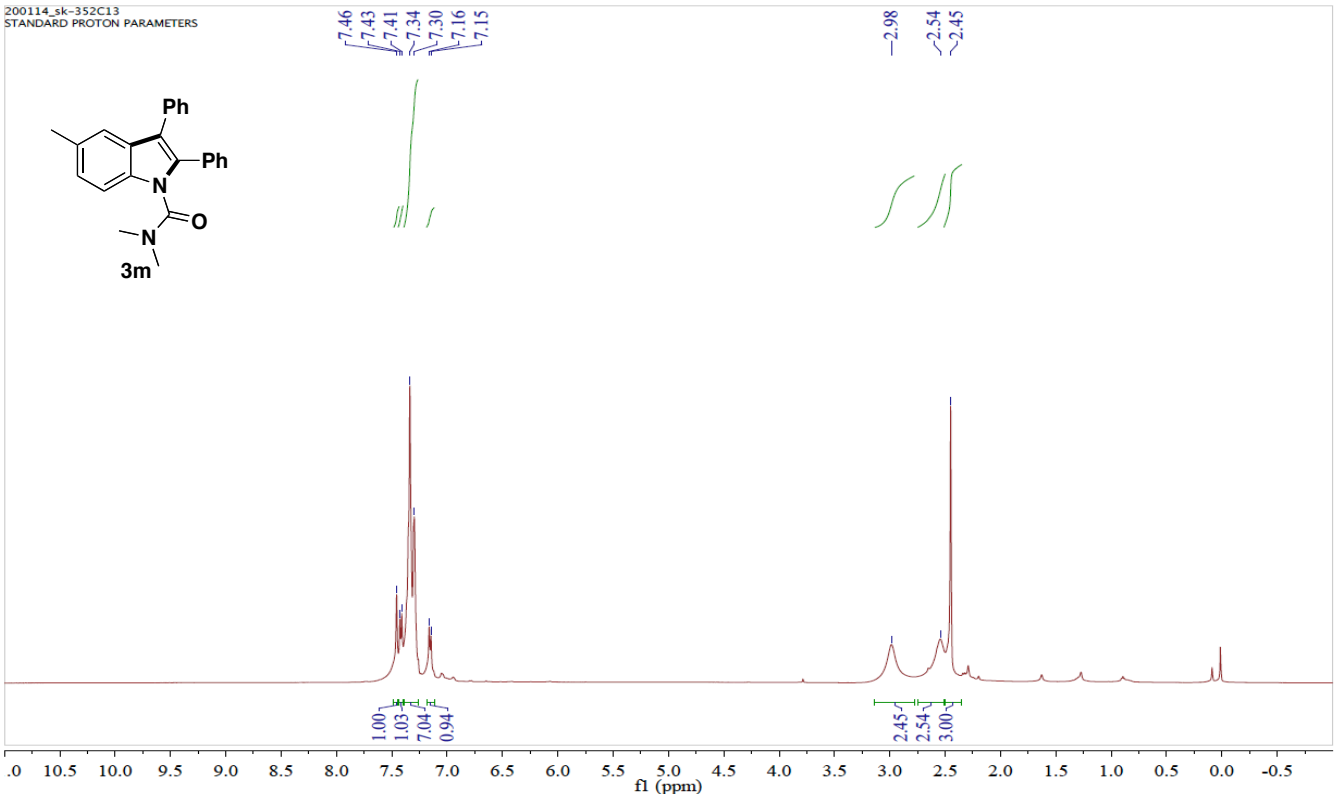

050314_sk-333REC13
STANDARD CARBON PARAMETERS
154.31
148.15
146.33
134.39
133.26
131.52
130.21
130.06
129.40
128.56
128.24
127.67
126.52
120.95
117.57
101.13
94.85
77.42
77.16
76.91
56.35
37.91
36.51
Ph
Ph
O
O
N
O
N
3h
fl (ppm)

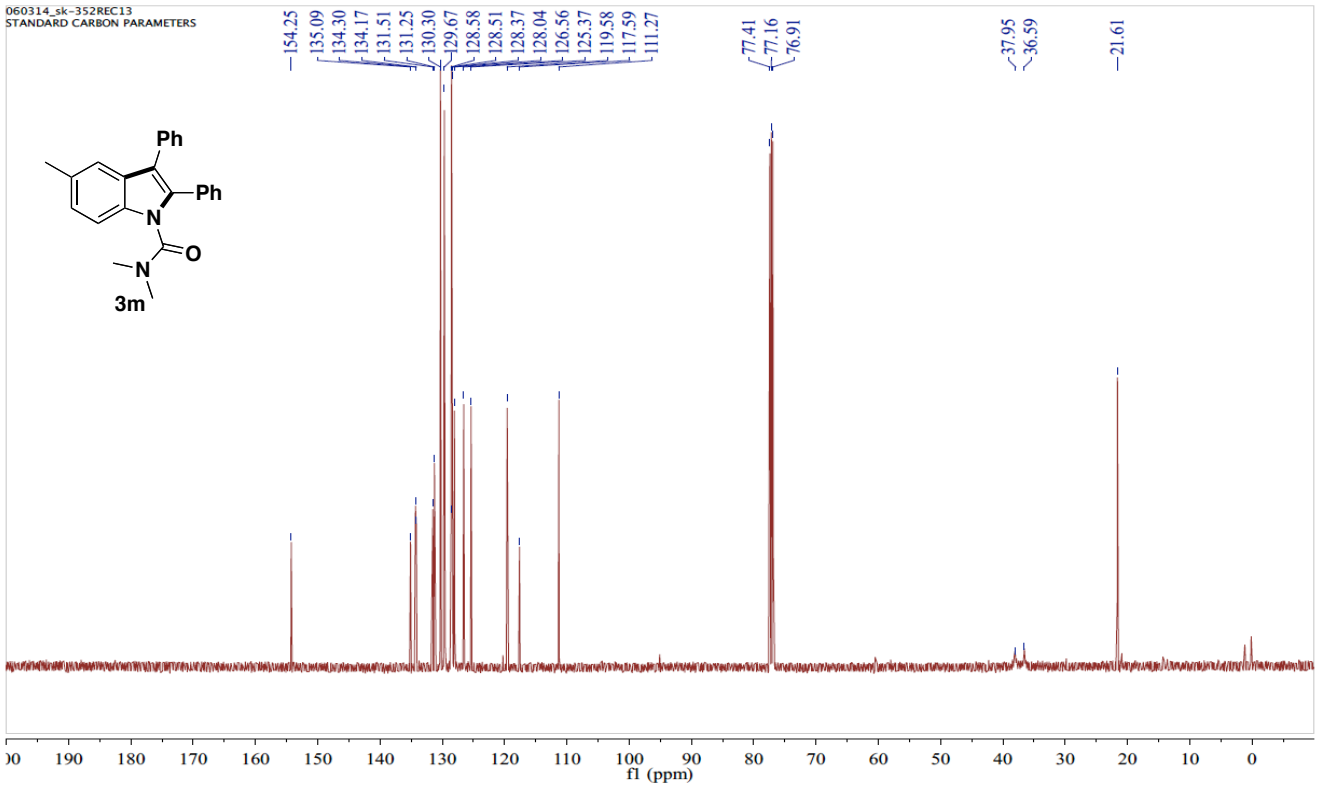

181213_sk-349H1
STANDARD PROTON PARAMETERS
Ph
Ph
N
O
N
3i
7.53
7.51
7.49
7.38
7.32
7.24
7.23
7.16
7.12
7.10
7.03
7.01
3.02
2.80
2.78
2.60
1.33
1.32
1.31
1.24
1.22
1.21
1.00
3.54
0.40
0.31
0.18
1.08
1.10
1.28
1.89
f1 (ppm)

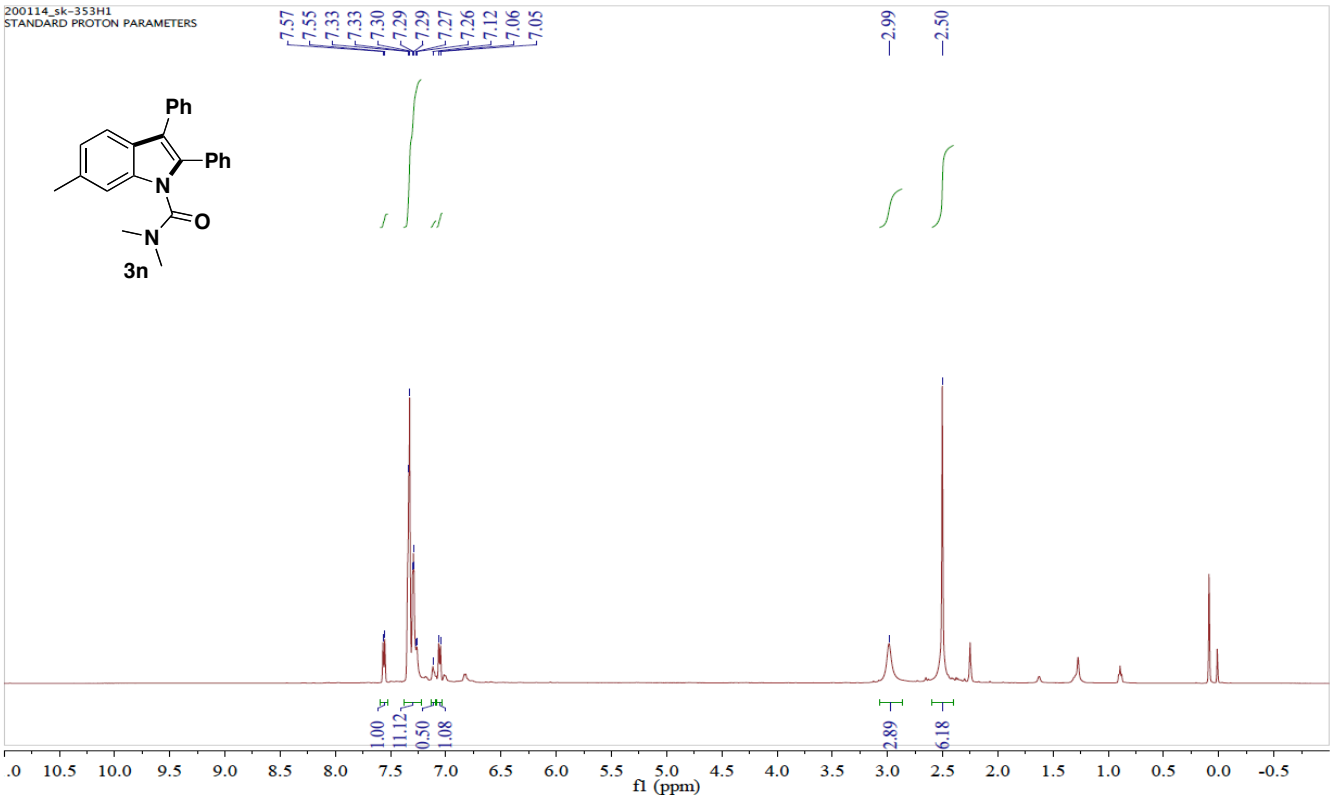

050314_sk-493REC13
STANDARD CARBON PARAMETERS
Ph
Ph
N
O
N
3i
154.26
137.97
135.10
134.31
134.26
131.47
130.26
129.62
128.52
128.48
128.34
128.14
128.01
126.52
124.35
119.81
118.38
117.75
111.36
77.41
77.16
76.91
38.06
36.52
29.15
16.56
f1 (ppm)

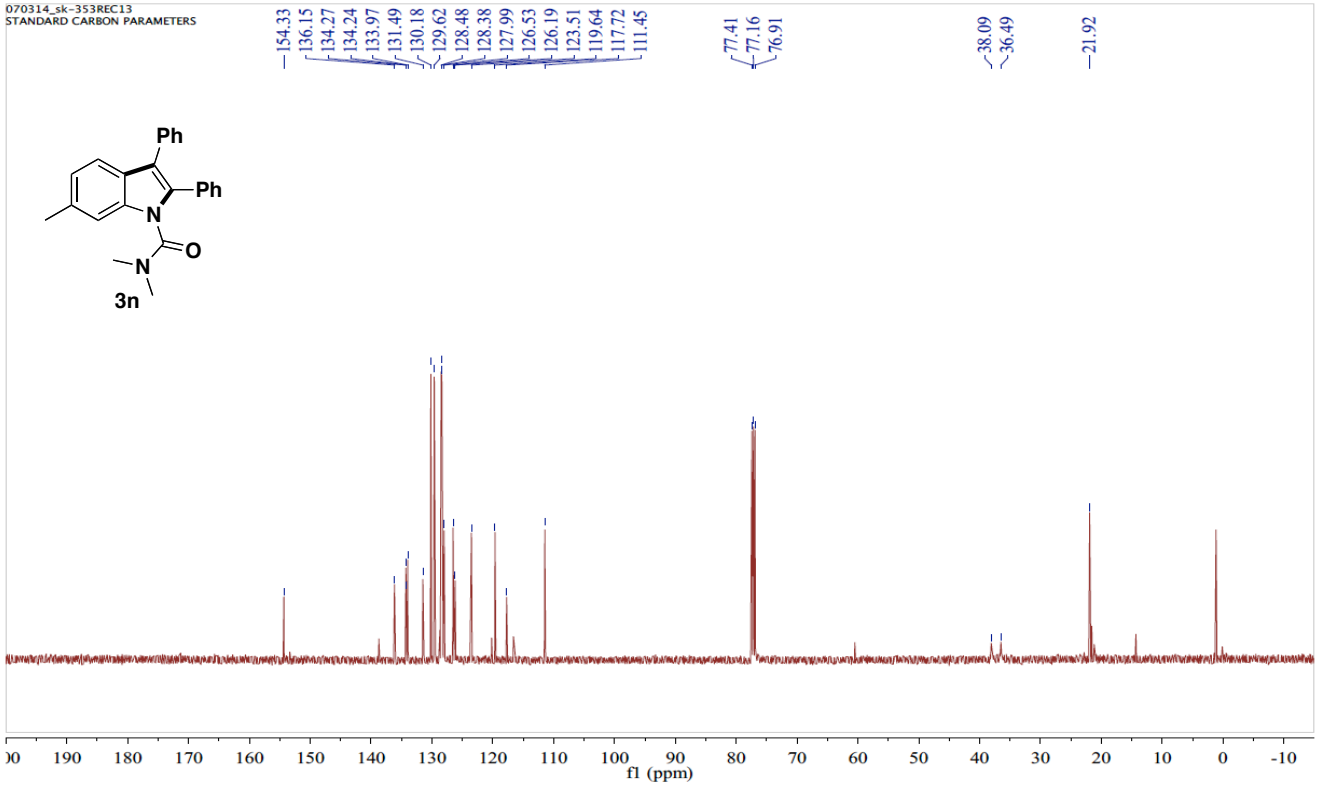

020914_sk-489BH1
STANDARD PROTON PARAMETERS
MeOOC
Ph
Ph
N
O
N
3p
8.32
7.95
7.94
7.93
7.93
7.45
7.43
7.27
7.26
7.25
7.25
7.23
7.21
7.21
3.82
2.91
2.43
1.95
1.00
1.18
1.22
6.27
5.16
3.61
3.49
3.65
1.23
fl (ppm)

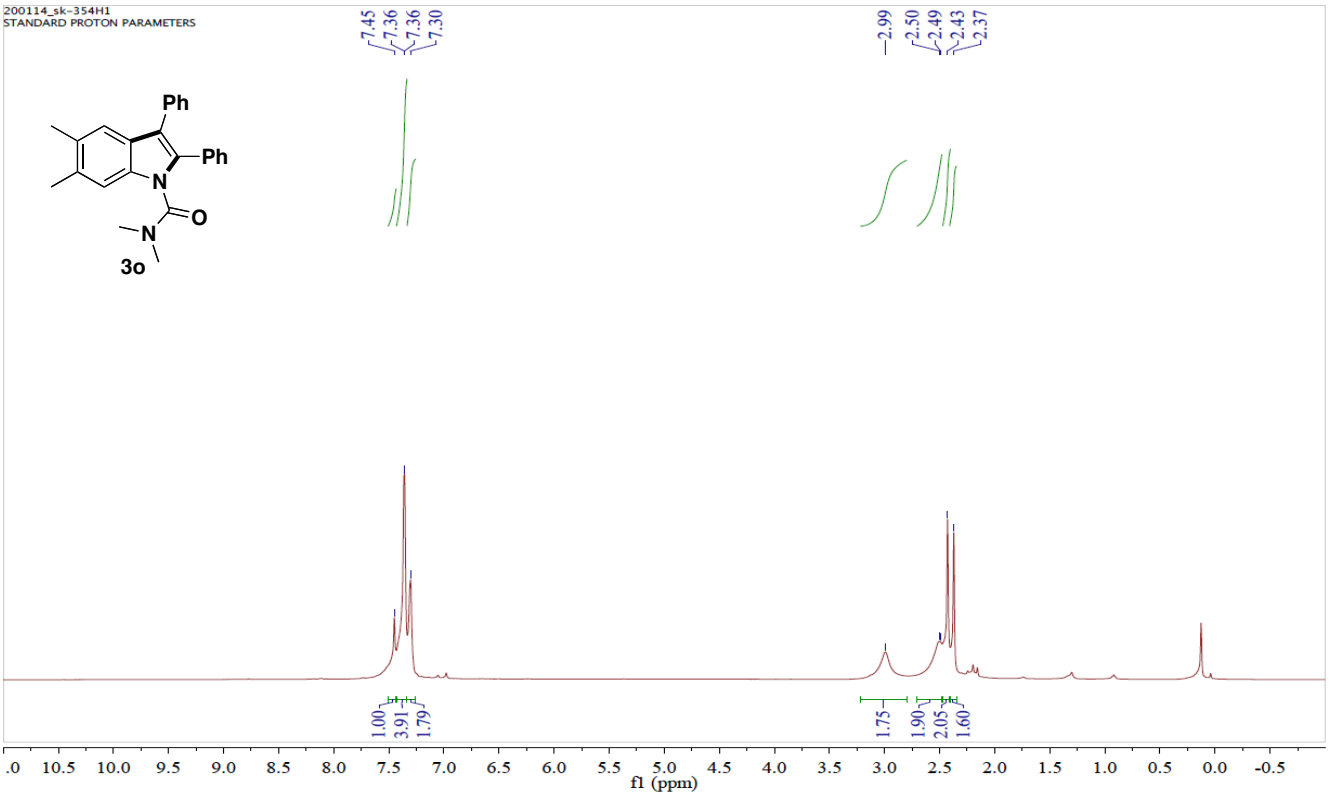

020914_sk-489BC13
STANDARD CARBON PARAMETERS
MeOOC
Ph
Ph
N
O
N
3p
138.13
136.15
133.27
130.71
130.19
129.58
128.67
128.50
127.93
126.93
125.10
123.81
122.72
118.48
167.79
153.38
111.21
77.41
77.16
76.91
60.48
52.05
38.04
36.55
21.14
14.26
fl (ppm)

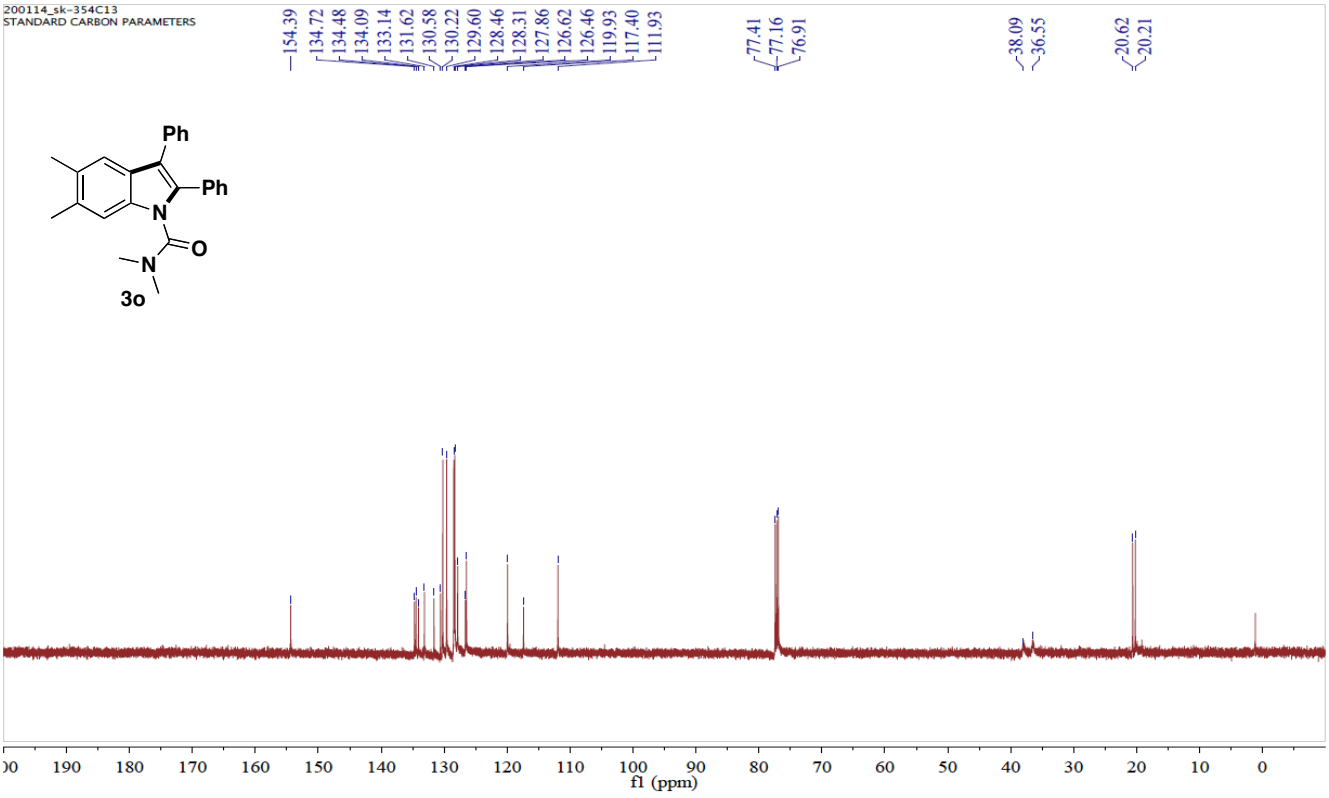

200114_sk-352C13
STANDARD PROTON PARAMETERS
7.46
7.43
7.41
7.34
7.30
7.16
7.15
2.98
2.54
2.45
Ph
Ph
N
O
N
3m
1.00
1.03
7.04
0.94
2.45
2.54
3.00
fl (ppm)

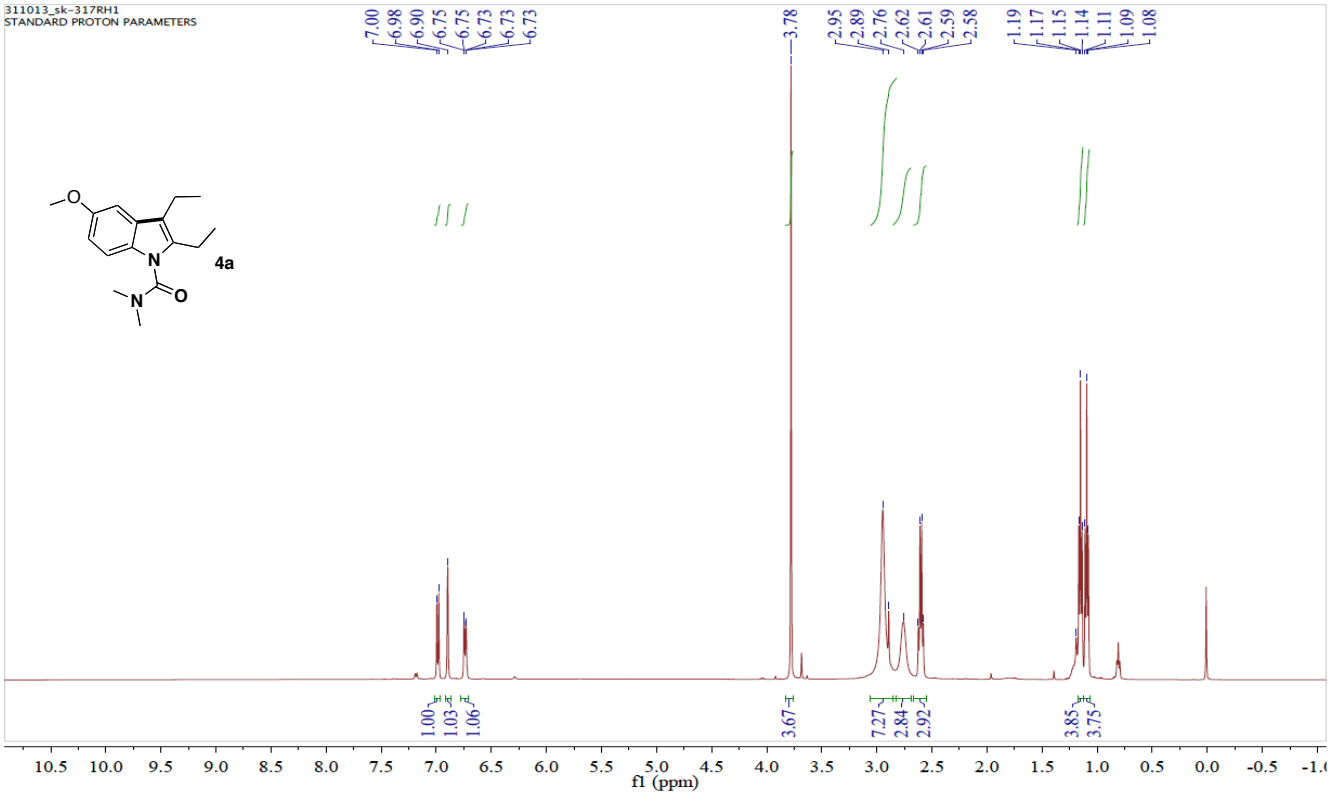

060314_sk-352REC13
STANDARD CARBON PARAMETERS
154.25
135.09
134.30
134.17
131.51
131.25
130.30
129.67
128.58
128.51
128.37
128.04
126.56
125.37
119.58
117.59
111.27
77.41
77.16
76.91
37.95
36.59
21.61
Ph
Ph
N
O
N
3m
fl (ppm)

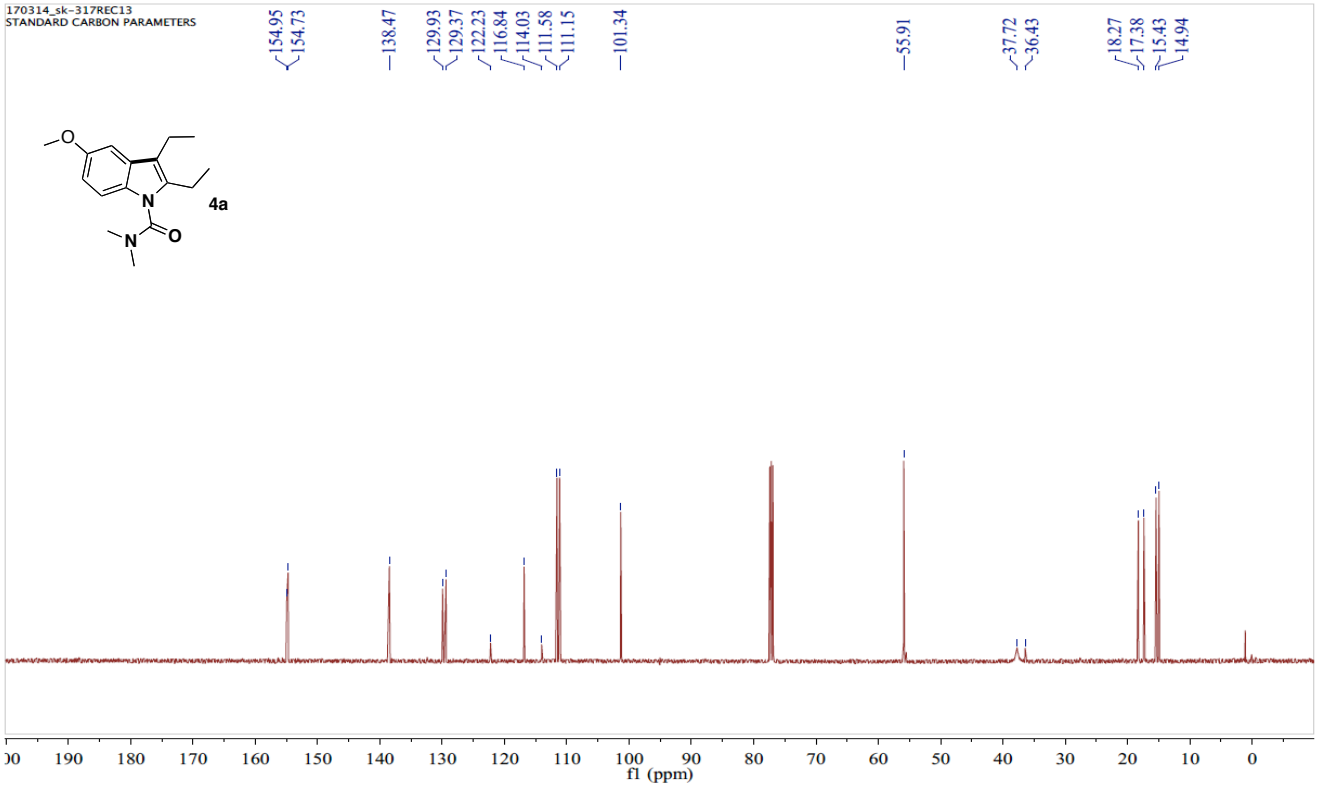

200114_sk-353H1
STANDARD PROTON PARAMETERS
7.57
7.55
7.33
7.33
7.30
7.29
7.29
7.27
7.26
7.12
7.06
7.05
2.99
2.50
Ph
Ph
N
O
N
3n
1.00
11.12
0.50
1.08
2.89
6.18
fl (ppm)

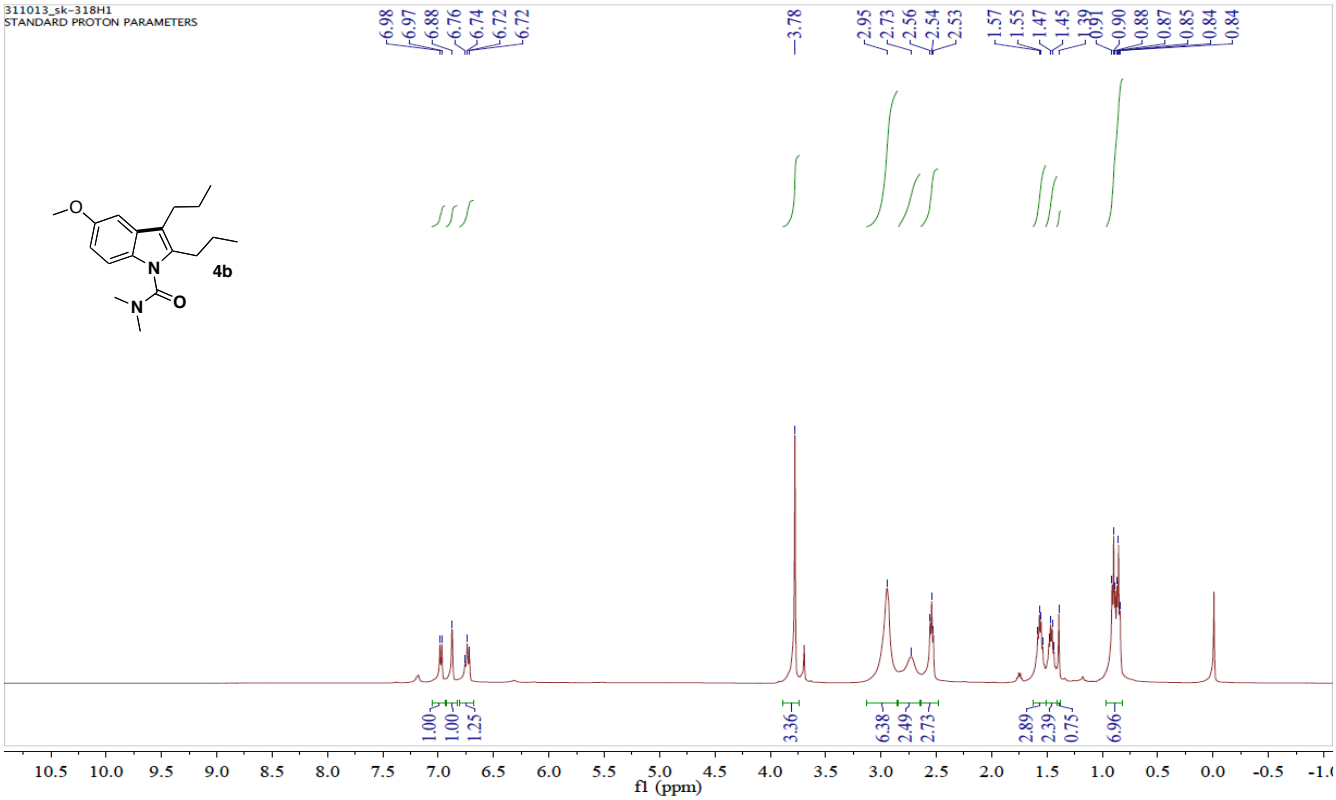

070314_sk-353REC13
STANDARD CARBON PARAMETERS
154.33
136.15
134.27
134.24
133.97
131.49
130.18
129.62
128.48
128.38
127.99
126.53
126.19
123.51
119.64
117.72
111.45
77.41
77.16
76.91
38.09
36.49
21.92
Ph
Ph
N
O
N
3n
fl (ppm)

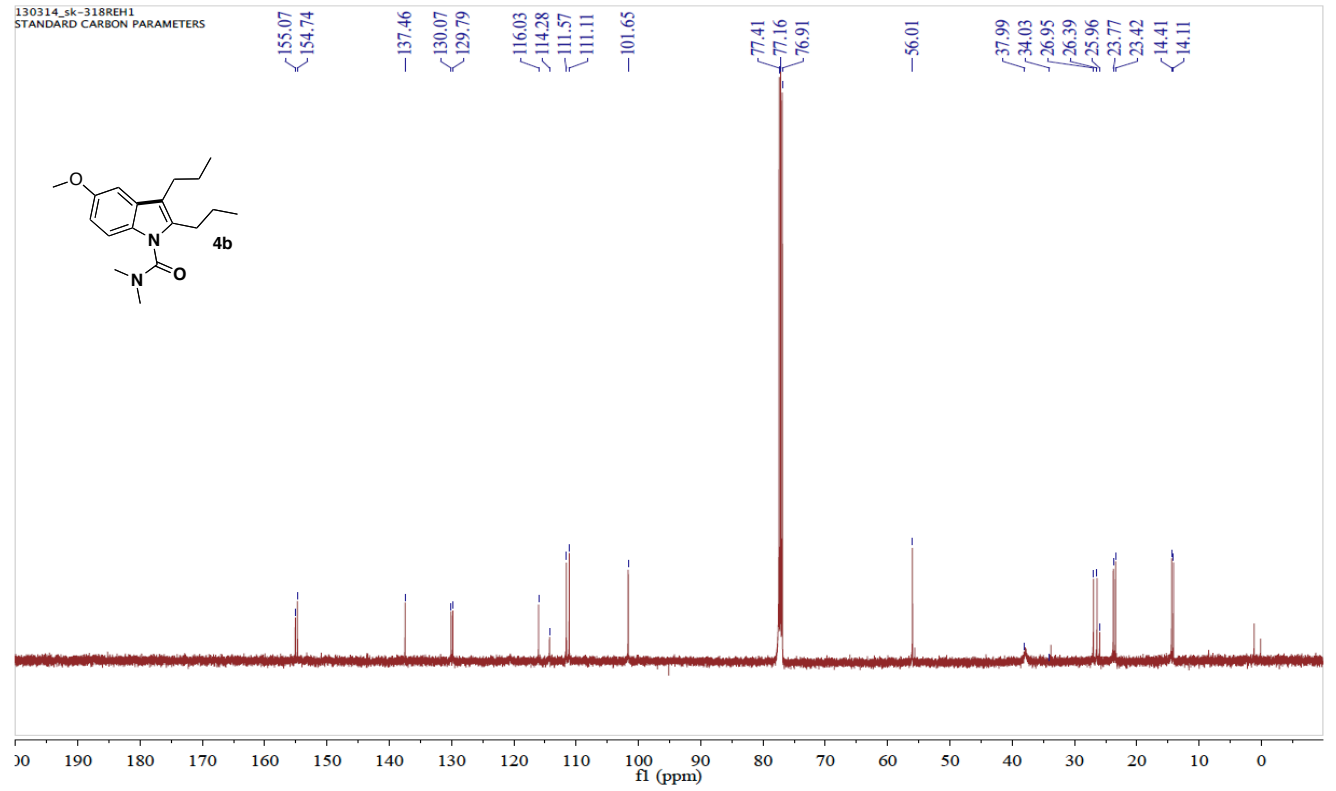

200114_sk-354H1
STANDARD PROTON PARAMETERS
7.45
7.36
7.36
7.30
2.99
2.50
2.49
2.43
2.37
Ph
Ph
N
O
N
3o
1.00
3.91
1.79
1.75
1.90
2.05
1.60
f1 (ppm)

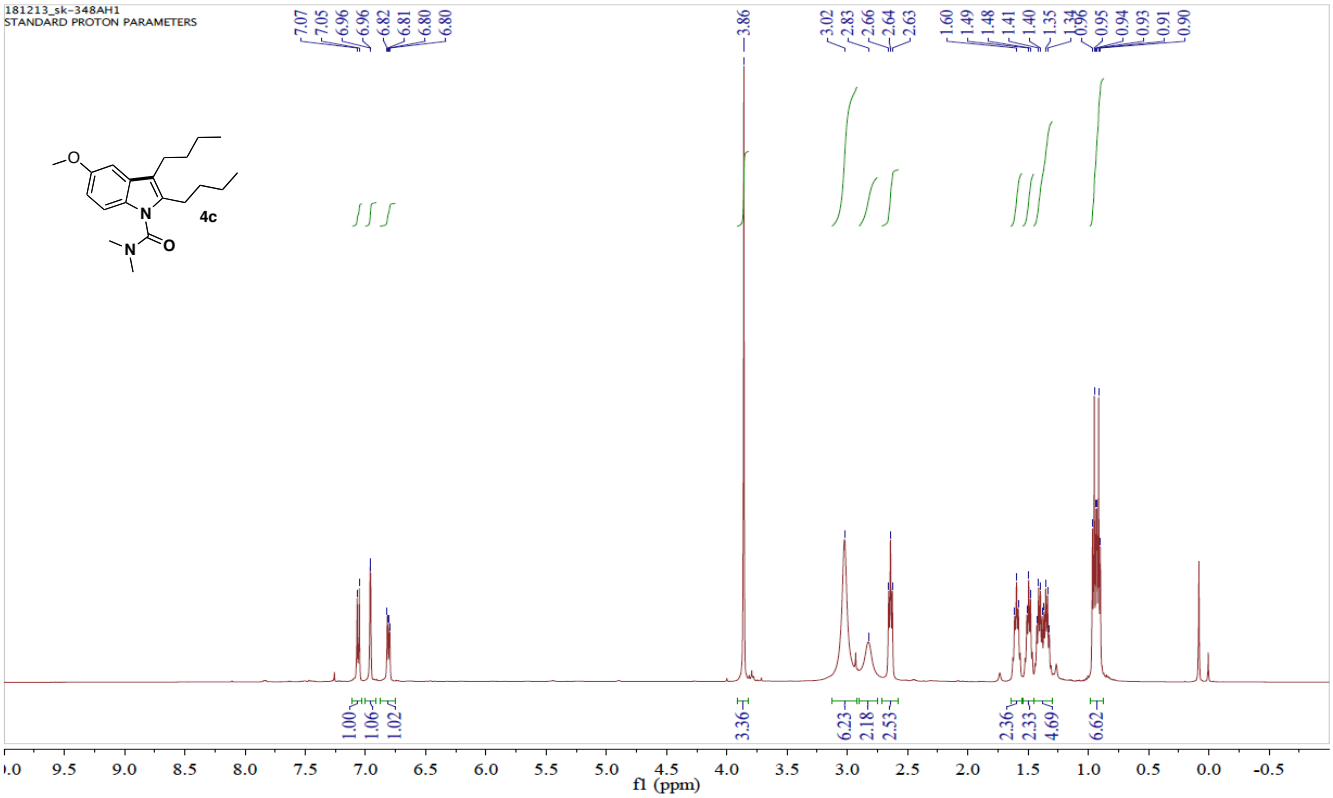

200114_sk-354C13
STANDARD CARBON PARAMETERS
154.39
134.72
134.48
134.09
133.14
131.62
130.58
130.22
129.60
128.46
128.31
127.86
126.62
126.46
119.93
117.40
111.93
77.41
77.16
76.91
38.09
36.55
20.62
20.21
Ph
Ph
N
O
N
3o
f1 (ppm)

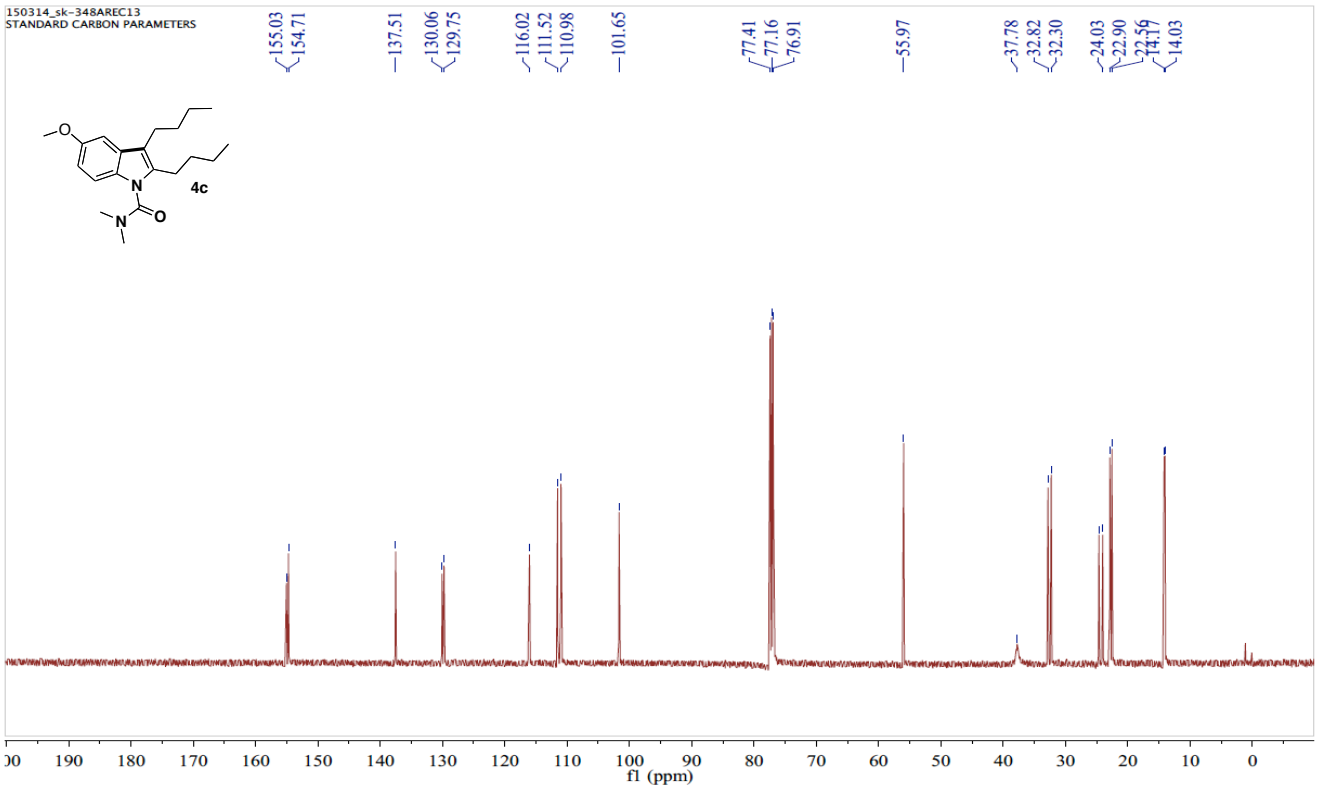

311013_sk-317RH1
STANDARD PROTON PARAMETERS
7.00
6.98
6.90
6.75
6.75
6.73
6.73
6.73
3.78
2.95
2.89
2.76
2.62
2.61
2.59
2.58
1.19
1.17
1.15
1.14
1.11
1.09
1.08
4a
1.00
1.03
1.06
3.67
7.27
2.84
2.92
3.85
3.75
fl (ppm)

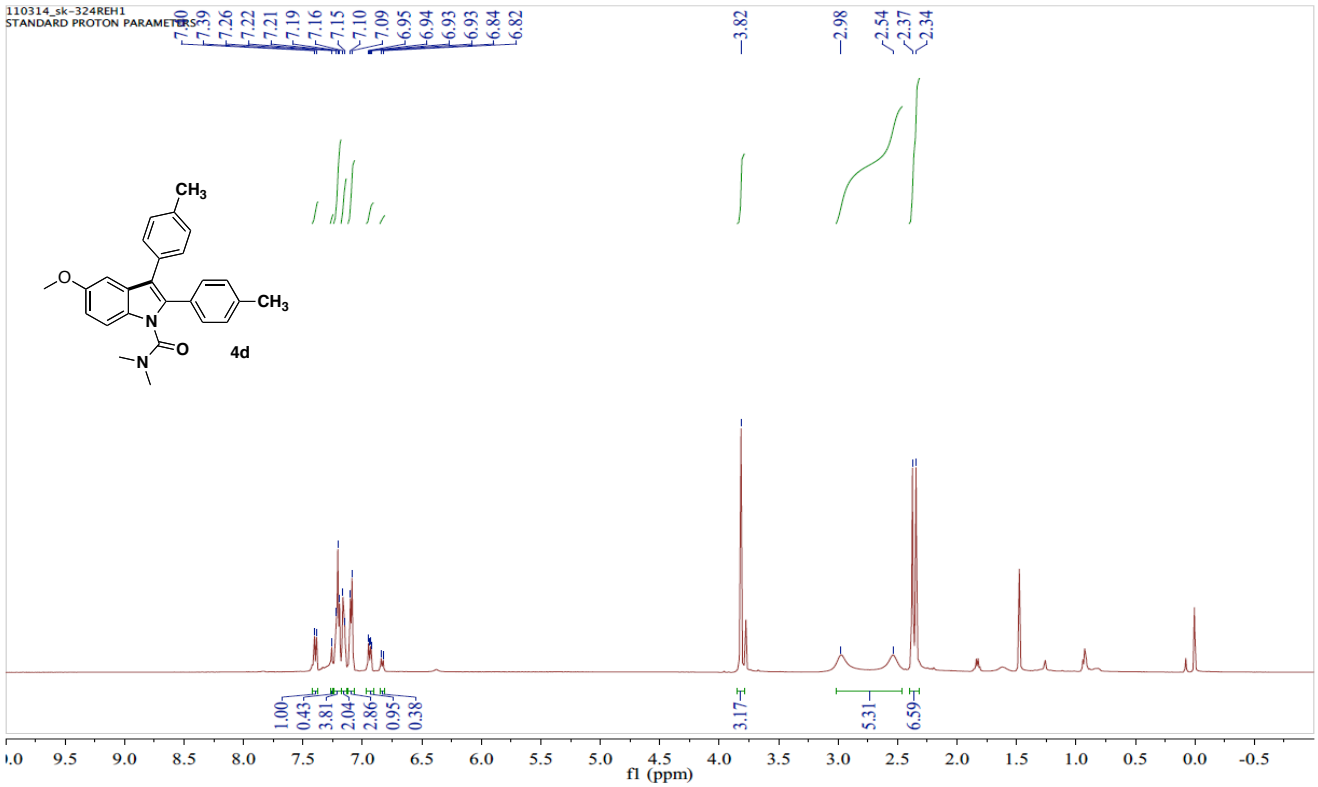

170314_sk-317REC13
STANDARD CARBON PARAMETERS
154.95
154.73
138.47
129.93
129.37
122.23
116.84
114.03
111.58
111.15
101.34
55.91
37.72
36.43
18.27
17.38
15.43
14.94
4a
fl (ppm)

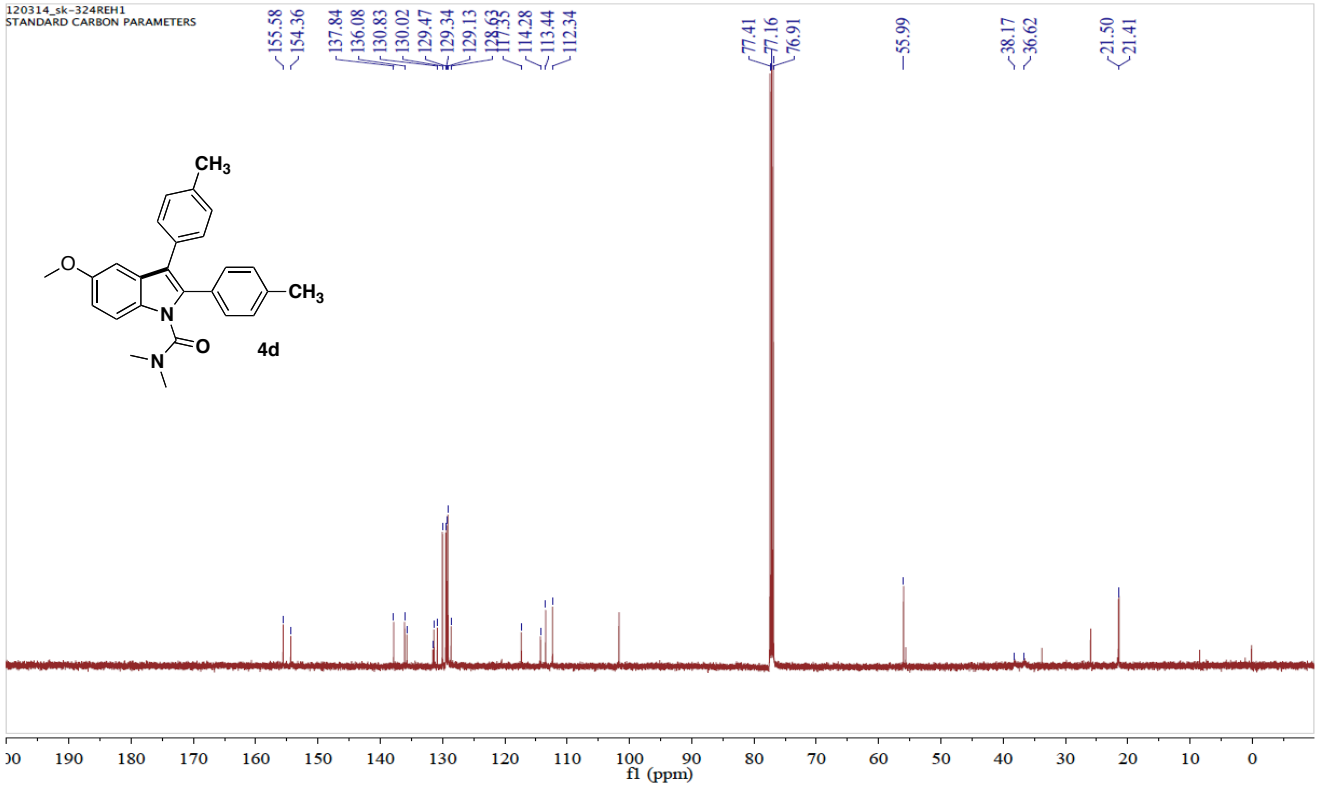

311013_sk-318H1
STANDARD PROTON PARAMETERS
4b
fl (ppm)

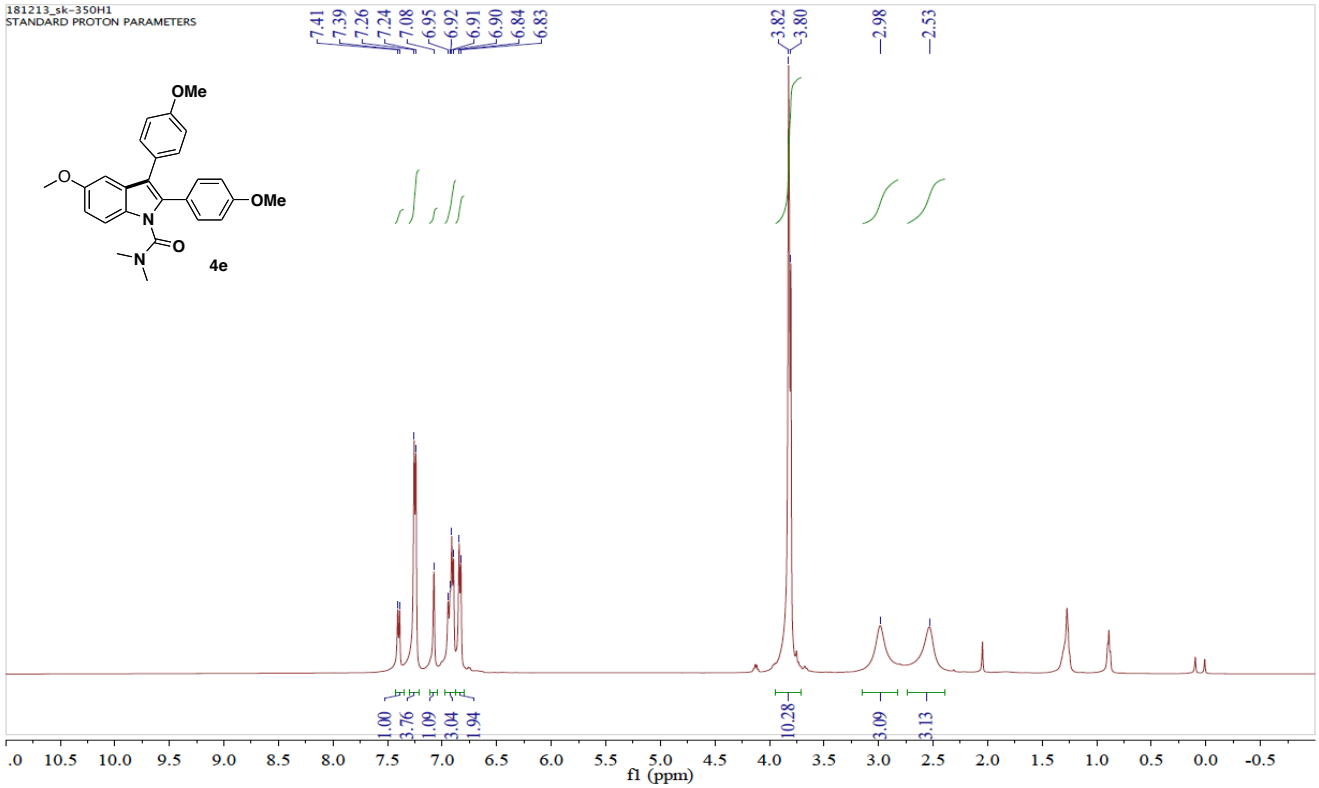

130314_sk-318REH1
STANDARD CARBON PARAMETERS
4b
fl (ppm)

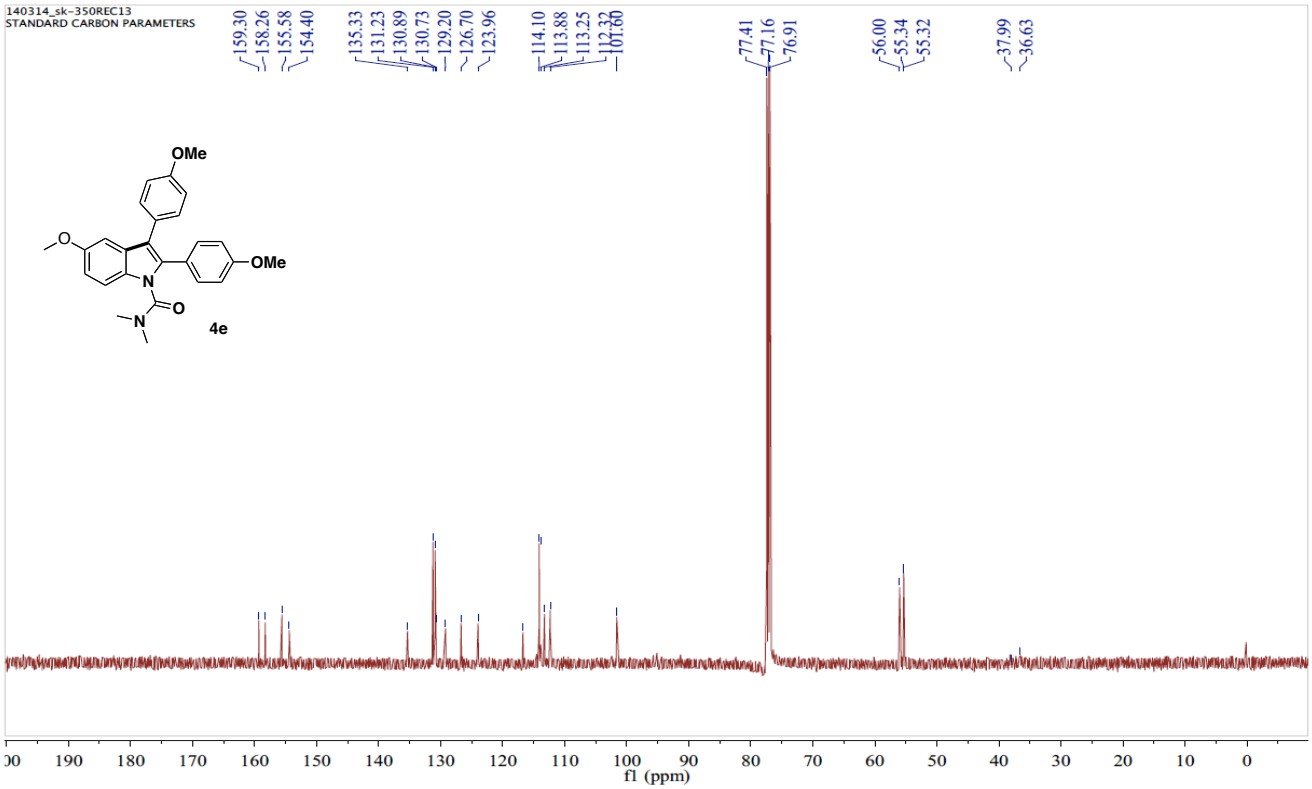

181213_sk-348AH1
STANDARD PROTON PARAMETERS
7.07
7.05
6.96
6.96
6.82
6.81
6.80
6.80
3.86
3.02
2.83
2.66
2.64
2.63
1.60
1.49
1.48
1.41
1.40
1.35
1.34
0.96
0.95
0.94
0.93
0.91
0.90
4c
1.00
1.06
1.02
3.36
6.23
2.18
2.53
2.36
2.33
4.69
6.62
f1 (ppm)

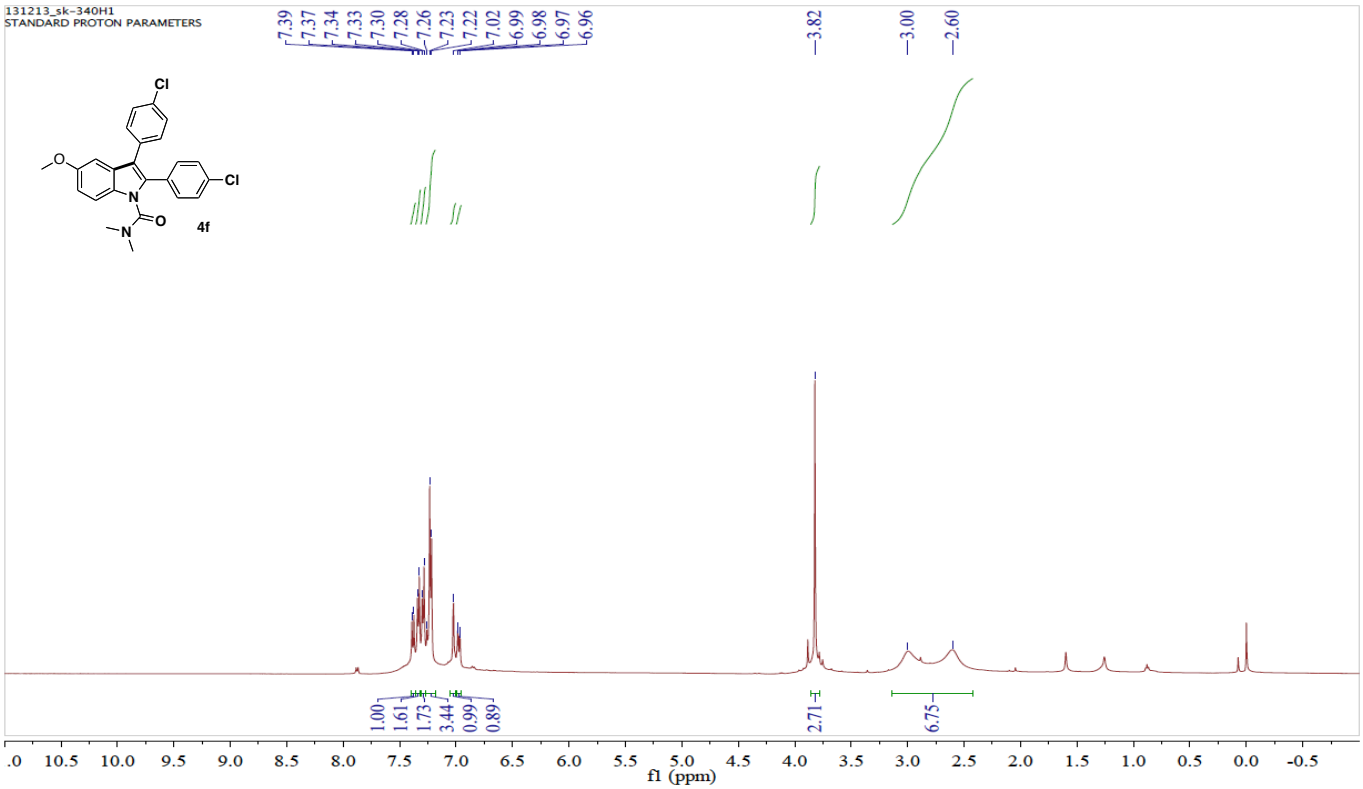

150314_sk-348AREC13
STANDARD CARBON PARAMETERS
155.03
154.71
137.51
130.06
129.75
116.02
111.52
110.98
101.65
77.41
77.16
76.91
55.97
37.78
32.82
32.30
24.03
22.90
14.17
14.03
4c
f1 (ppm)

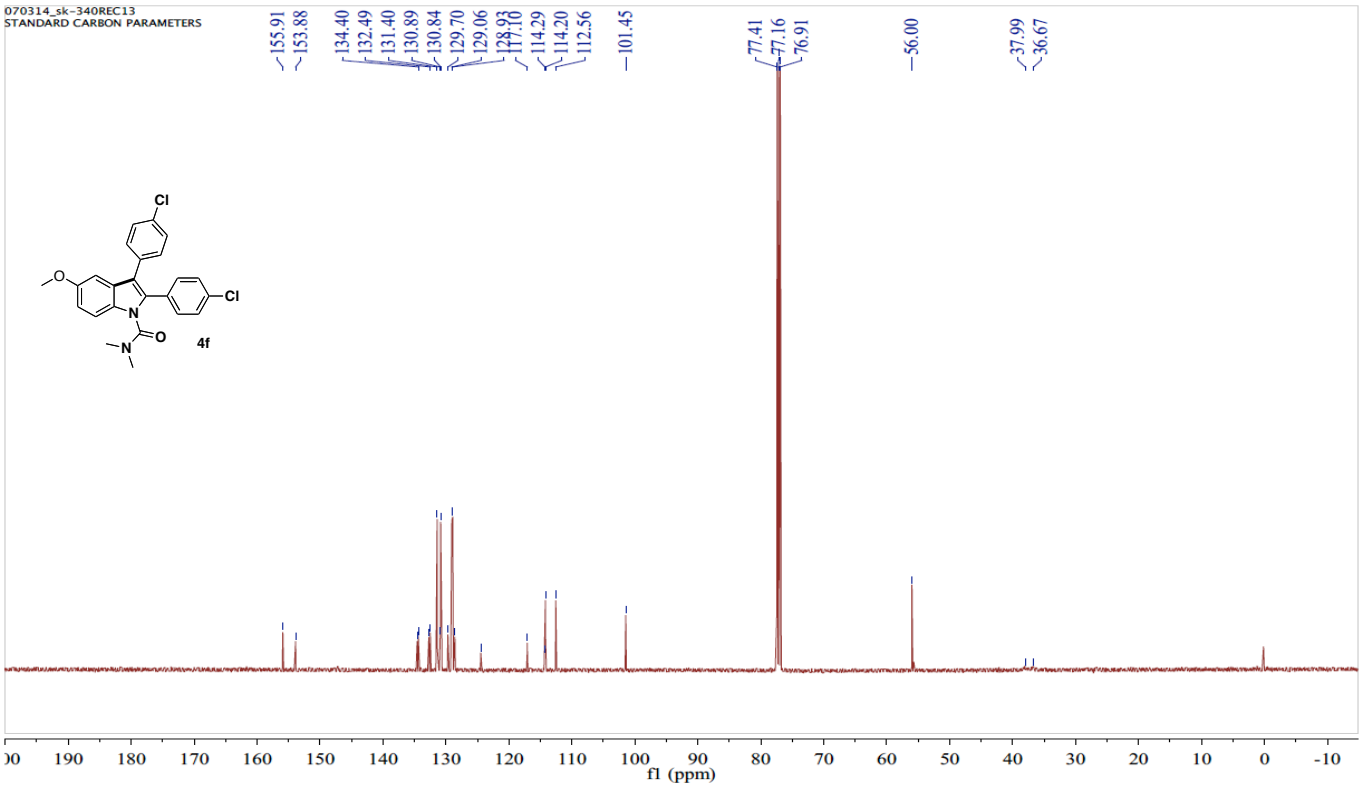

110314_sk-324REH1
STANDARD PROTON PARAMETERS
7.40
7.39
7.26
7.22
7.21
7.19
7.16
7.15
7.10
7.09
6.95
6.94
6.93
6.84
6.82
3.82
2.98
2.54
2.37
2.34
CH3
CH3
O
N
O
N
4d
1.00
0.43
3.81
2.04
2.86
0.95
0.38
3.17
5.31
6.59
f1 (ppm)

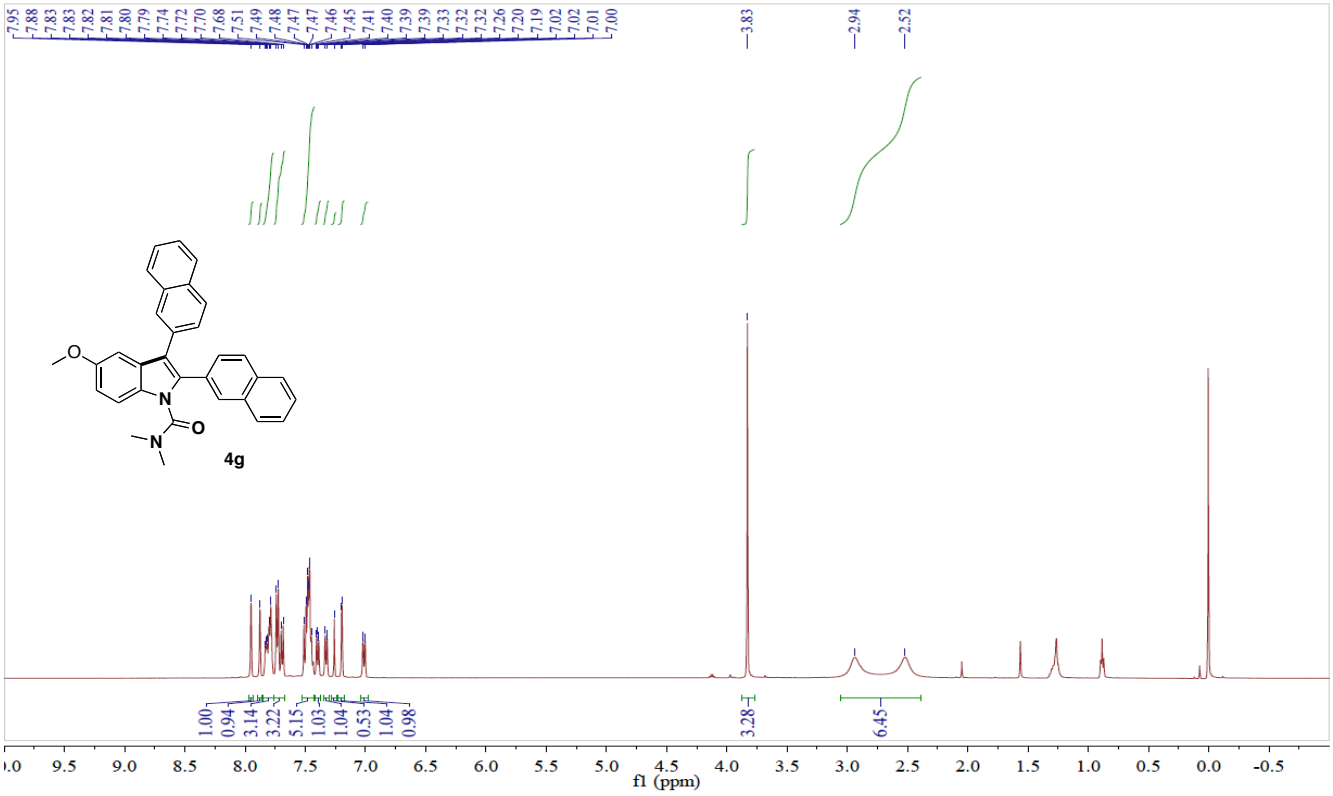

120314_sk-324REH1
STANDARD CARBON PARAMETERS
155.58
154.36
137.84
136.08
130.83
130.02
129.47
129.34
129.13
128.63
119.35
114.28
113.44
112.34
77.41
77.16
76.91
55.99
38.17
36.62
21.50
21.41
CH3
CH3
O
N
O
N
4d
f1 (ppm)

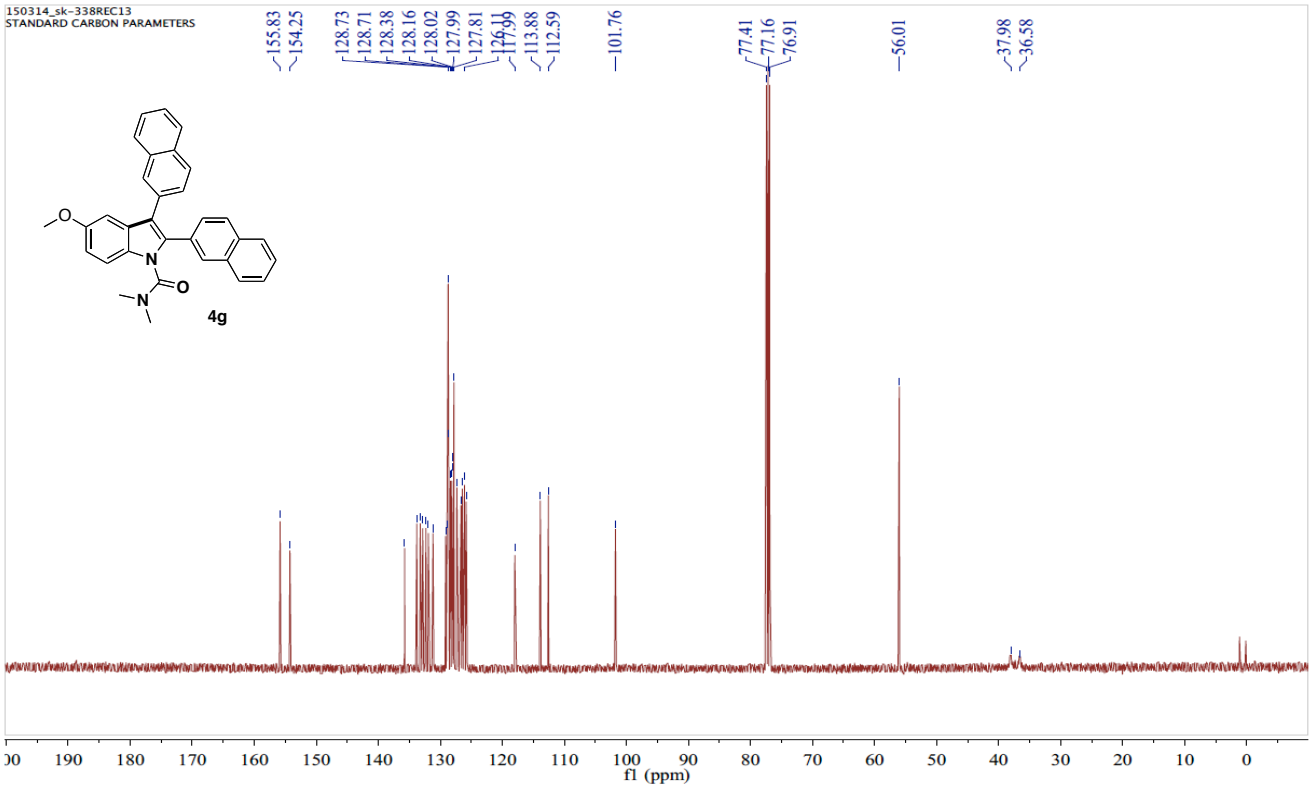

181213_sk-350H1
STANDARD PROTON PARAMETERS
7.41
7.39
7.26
7.24
7.08
6.95
6.92
6.91
6.90
6.84
6.83
3.82
3.80
2.98
2.53
OMe
OMe
4e
1.00
3.76
1.09
3.04
1.94
10.28
3.09
3.13
f1 (ppm)

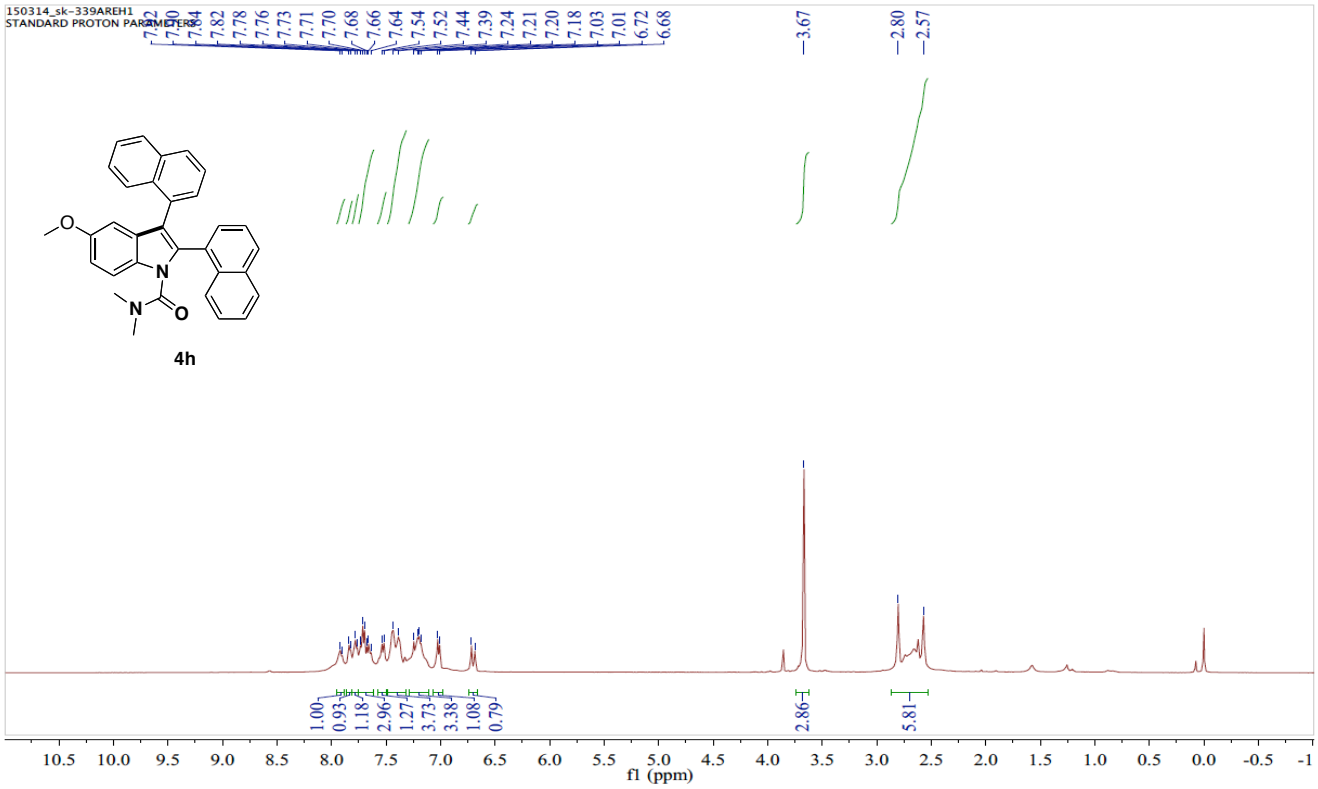

140314_sk-350REC13
STANDARD CARBON PARAMETERS
159.30
158.26
155.58
154.40
135.33
131.23
130.89
130.73
129.20
126.70
123.96
114.10
113.88
113.25
112.32
101.60
77.41
77.16
76.91
56.00
55.34
55.32
37.99
36.63
OMe
OMe
4e
f1 (ppm)

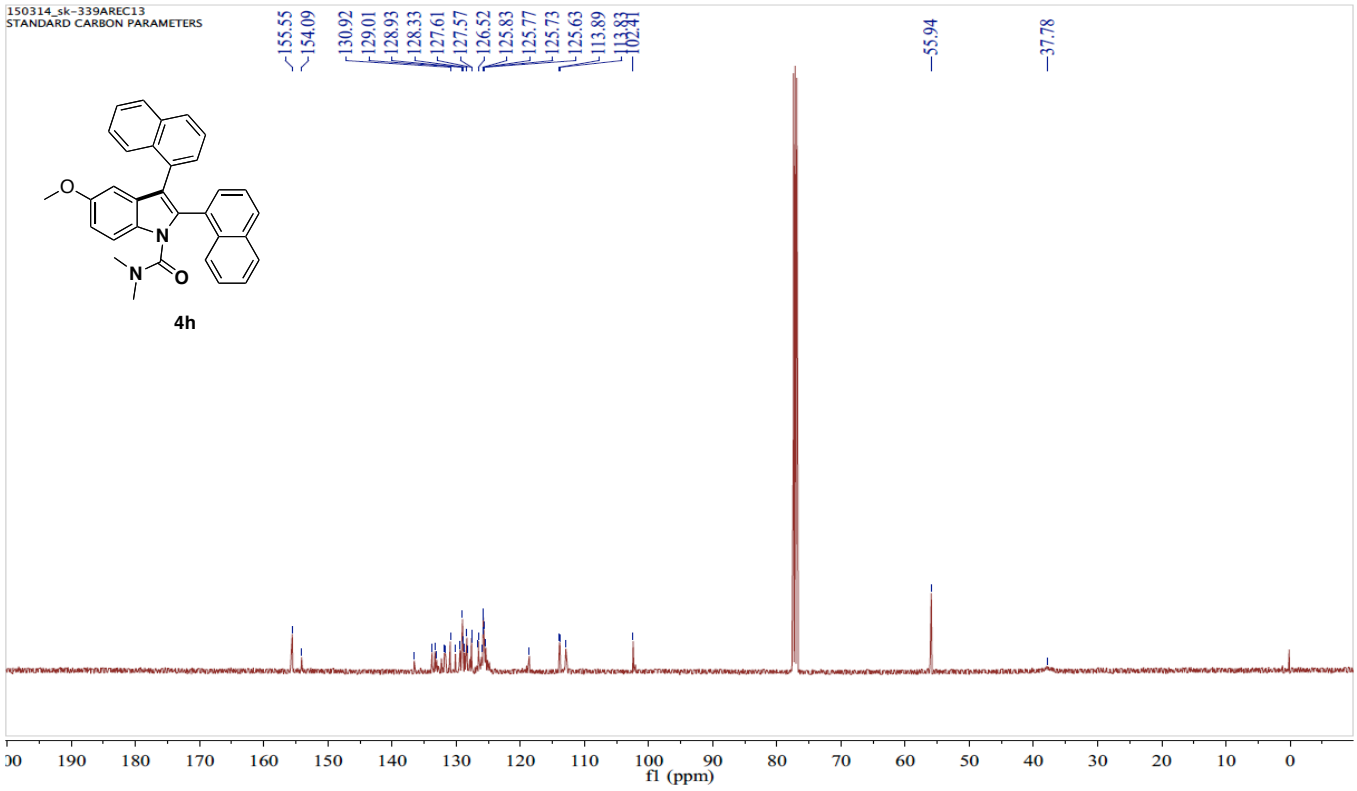

131213_sk-340H1
STANDARD PROTON PARAMETERS
7.39
7.37
7.34
7.33
7.30
7.28
7.26
7.23
7.22
7.02
6.99
6.98
6.97
6.96
3.82
3.00
2.60
Cl
Cl
O
N
O
N
4f
1.00
1.61
1.73
3.44
0.99
0.89
2.71
6.75
f1 (ppm)

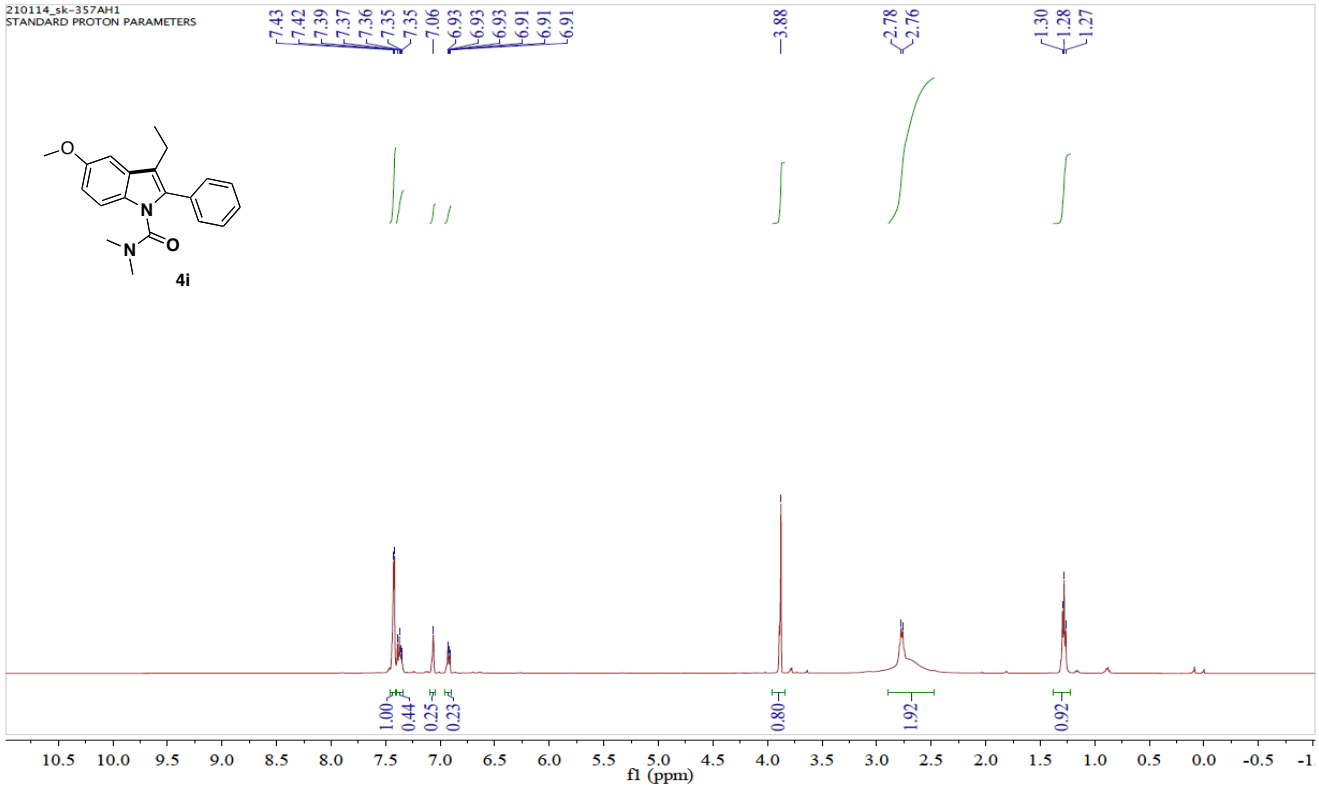

070314_sk-340REC13
STANDARD CARBON PARAMETERS
155.91
153.88
134.40
132.49
131.40
130.89
130.84
129.70
129.06
128.93
117.10
114.29
114.20
112.56
101.45
77.41
77.16
76.91
56.00
37.99
36.67
Cl
Cl
O
N
O
N
4f
f1 (ppm)

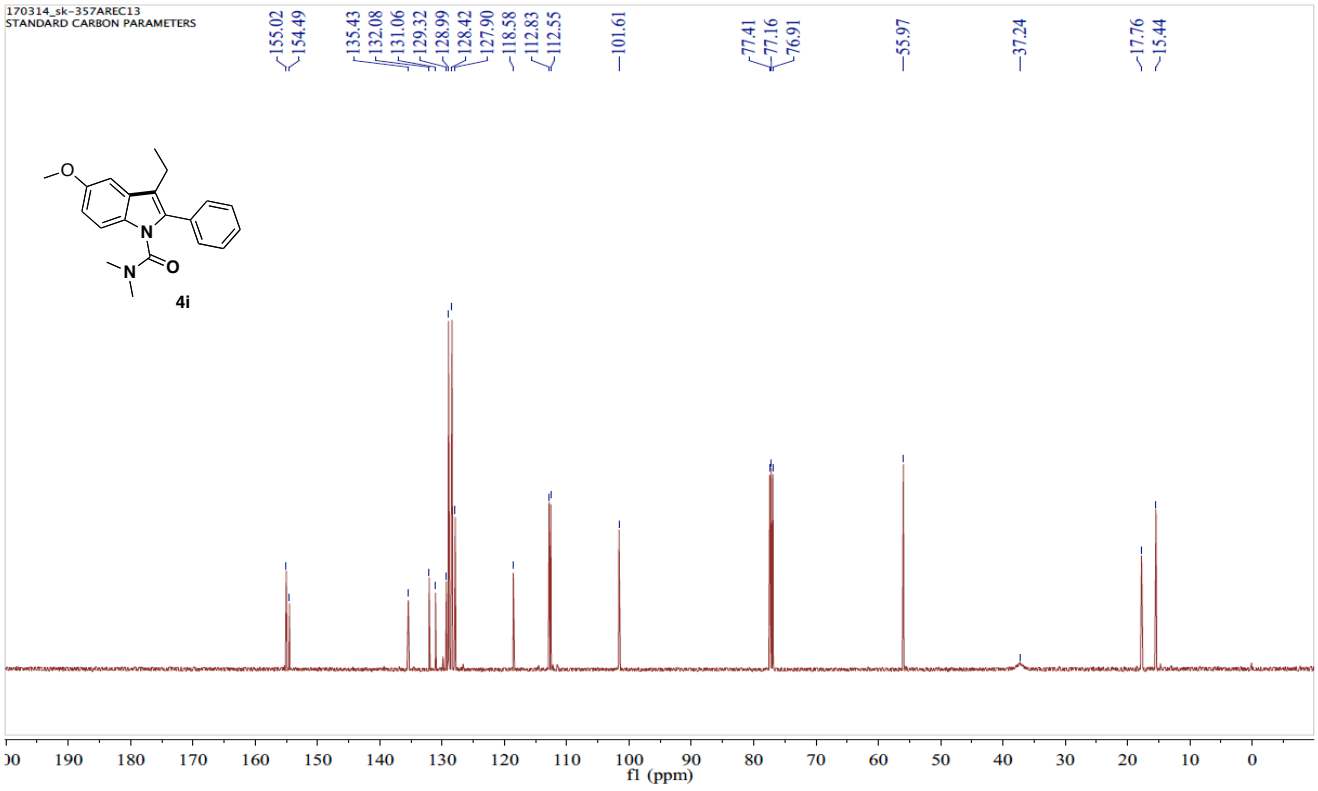

7.95
7.88
7.83
7.83
7.82
7.81
7.80
7.79
7.74
7.72
7.70
7.68
7.51
7.49
7.48
7.47
7.47
7.46
7.45
7.41
7.40
7.39
7.39
7.33
7.32
7.32
7.26
7.20
7.19
7.02
7.02
7.01
7.00
3.83
2.94
2.52
4g
1.00
0.94
3.14
3.22
5.15
1.03
1.04
0.53
1.04
0.98
3.28
6.45
f1 (ppm)

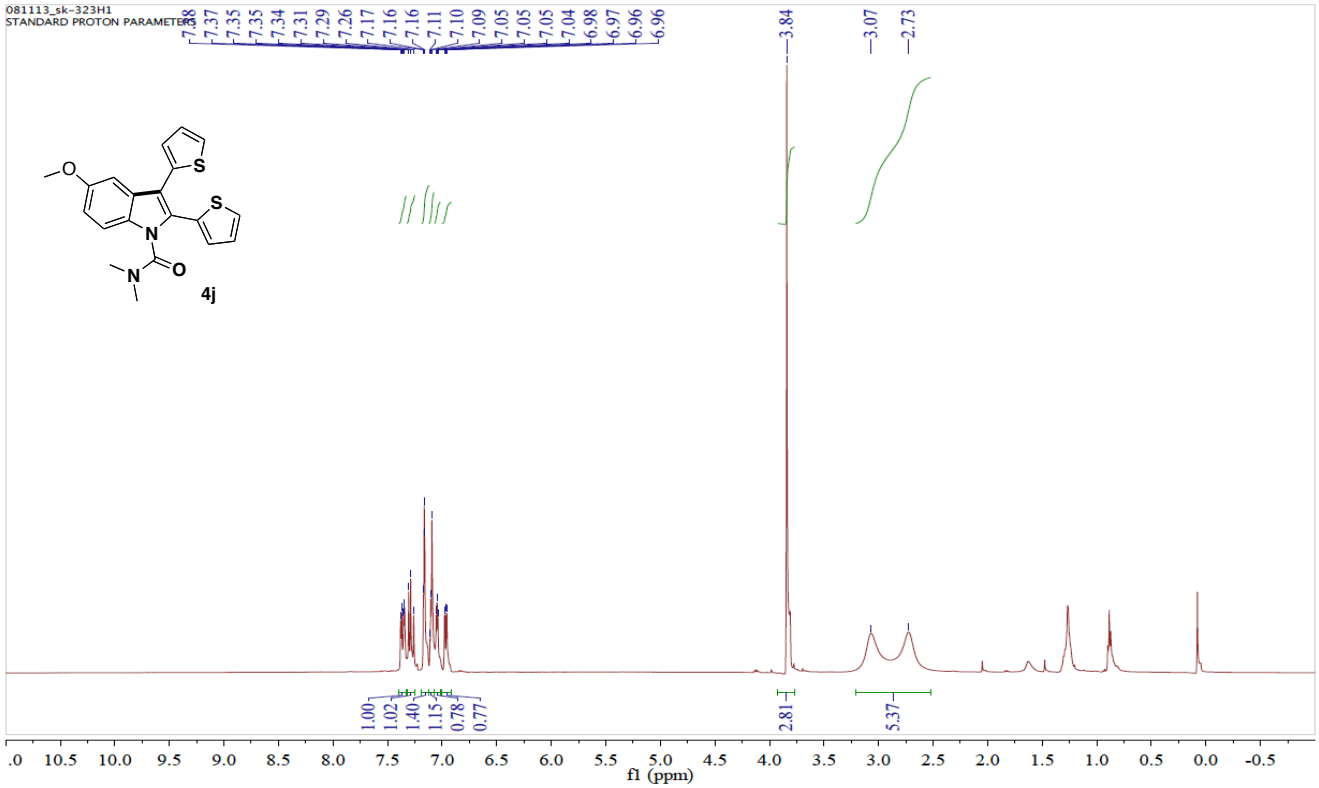

150314_sk-338REC13
STANDARD CARBON PARAMETERS
155.83
154.25
128.73
128.71
128.38
128.16
128.02
127.99
127.81
113.88
112.59
101.76
77.41
77.16
76.91
56.01
37.98
36.58
4g
f1 (ppm)

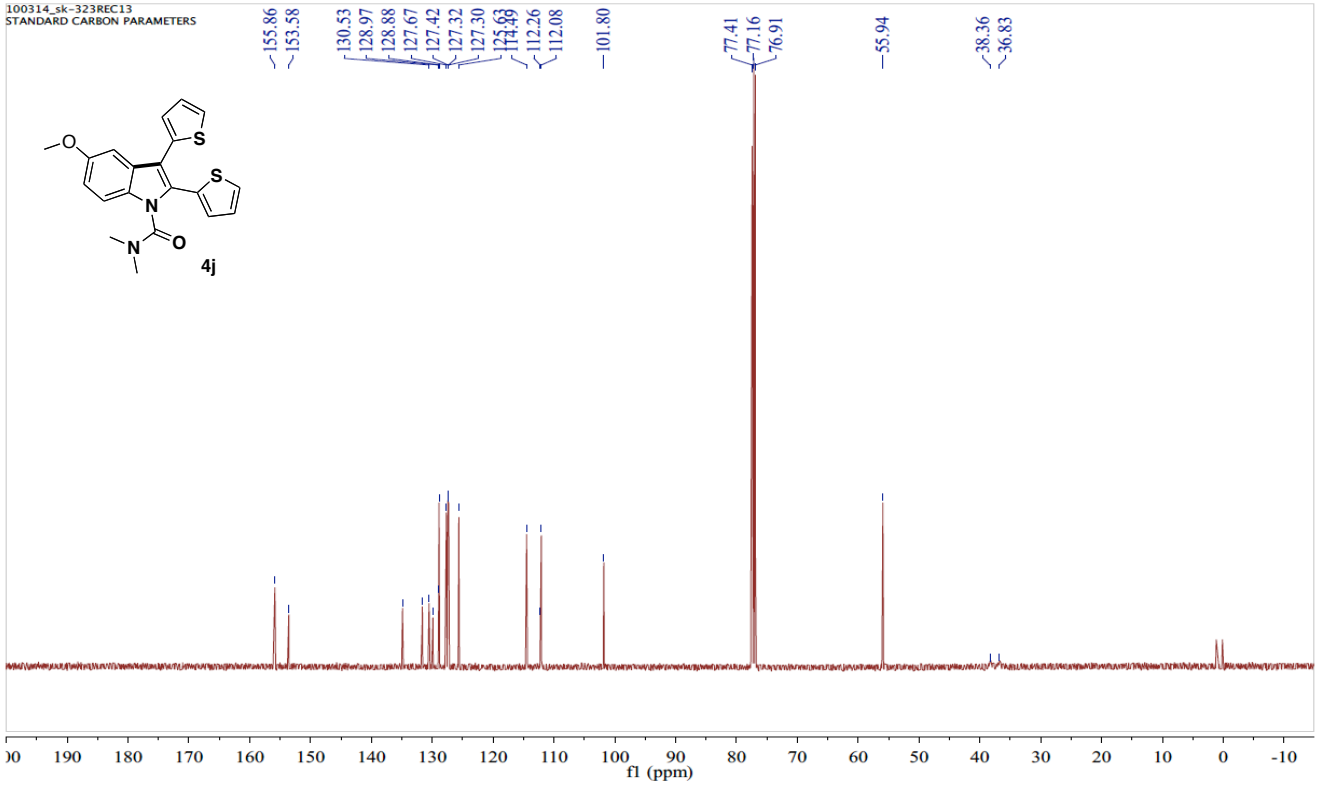

150314_sk-339AREH1
STANDARD PROTON PARAMETERS
7.82
7.80
7.84
7.82
7.78
7.76
7.73
7.71
7.70
7.68
7.66
7.64
7.54
7.52
7.44
7.39
7.24
7.21
7.20
7.18
7.03
7.01
6.72
6.68
3.67
2.80
2.57
4h
1.00
0.93
1.18
2.96
1.27
3.73
3.38
1.08
0.79
2.86
5.81
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

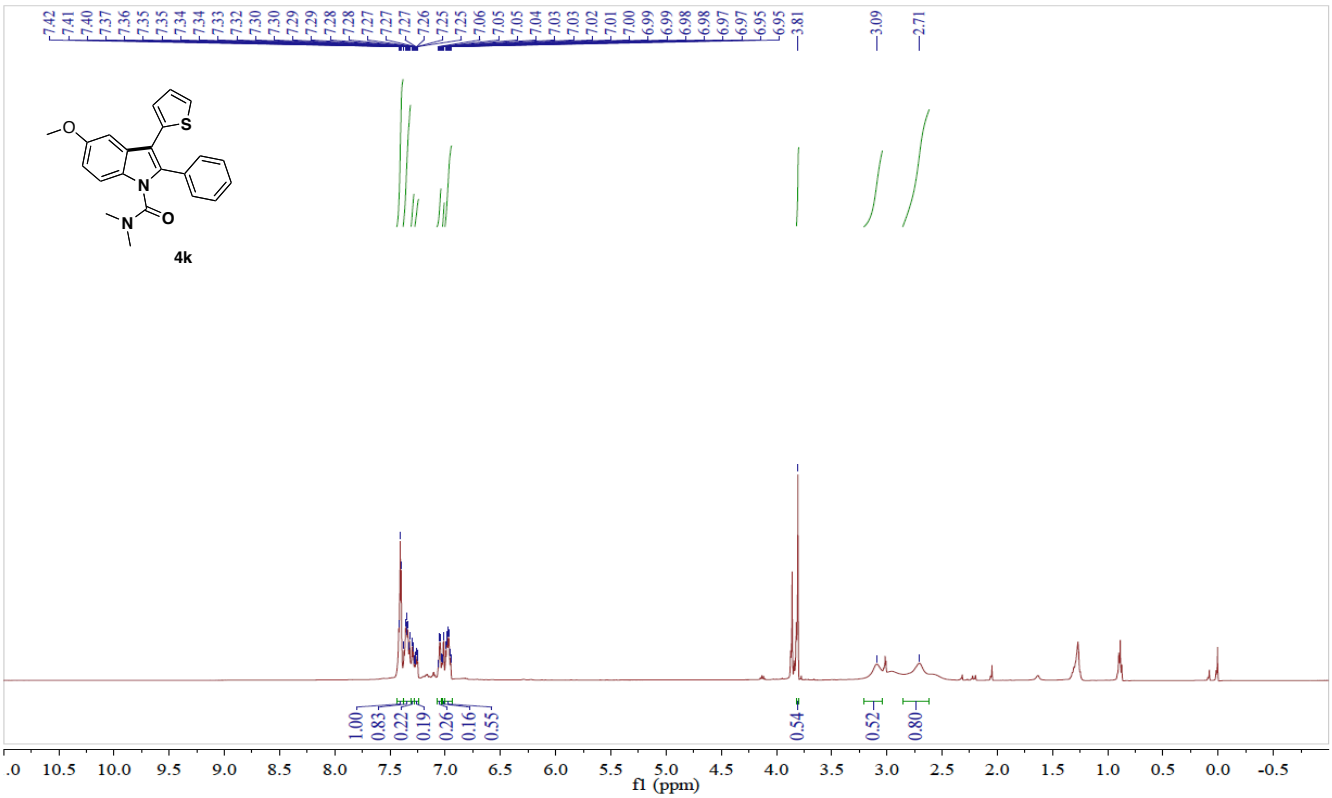

150314_sk-339AREC13
STANDARD CARBON PARAMETERS
155.55
154.09
130.92
129.01
128.93
128.33
127.61
127.57
126.52
125.83
125.77
125.73
125.63
113.89
55.94
37.78
4h
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

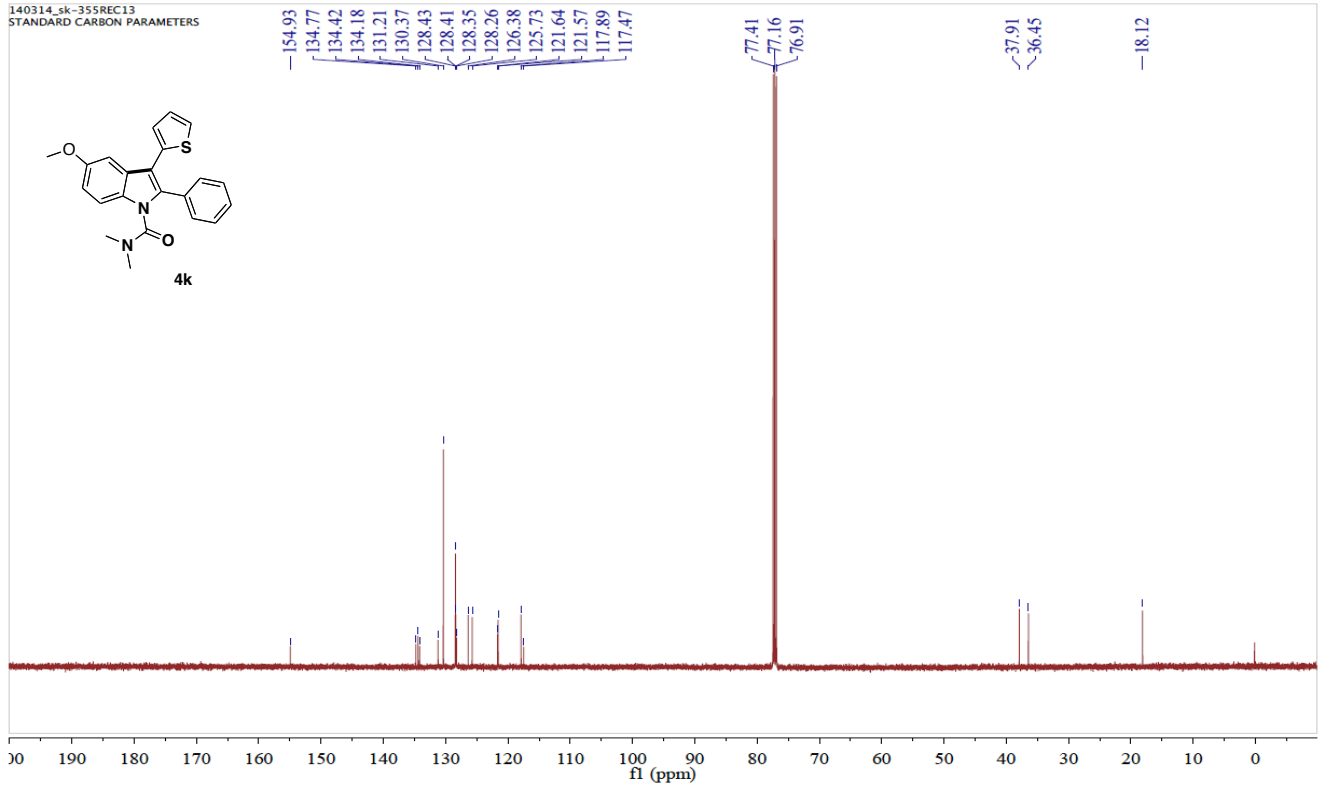

210114_sk-357AH1
STANDARD PROTON PARAMETERS
7.43
7.42
7.39
7.37
7.36
7.35
7.35
7.06
6.93
6.93
6.93
6.91
6.91
6.91
3.88
2.78
2.76
1.30
1.28
1.27
4i
1.00
0.44
0.25
0.23
0.80
1.92
0.92
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

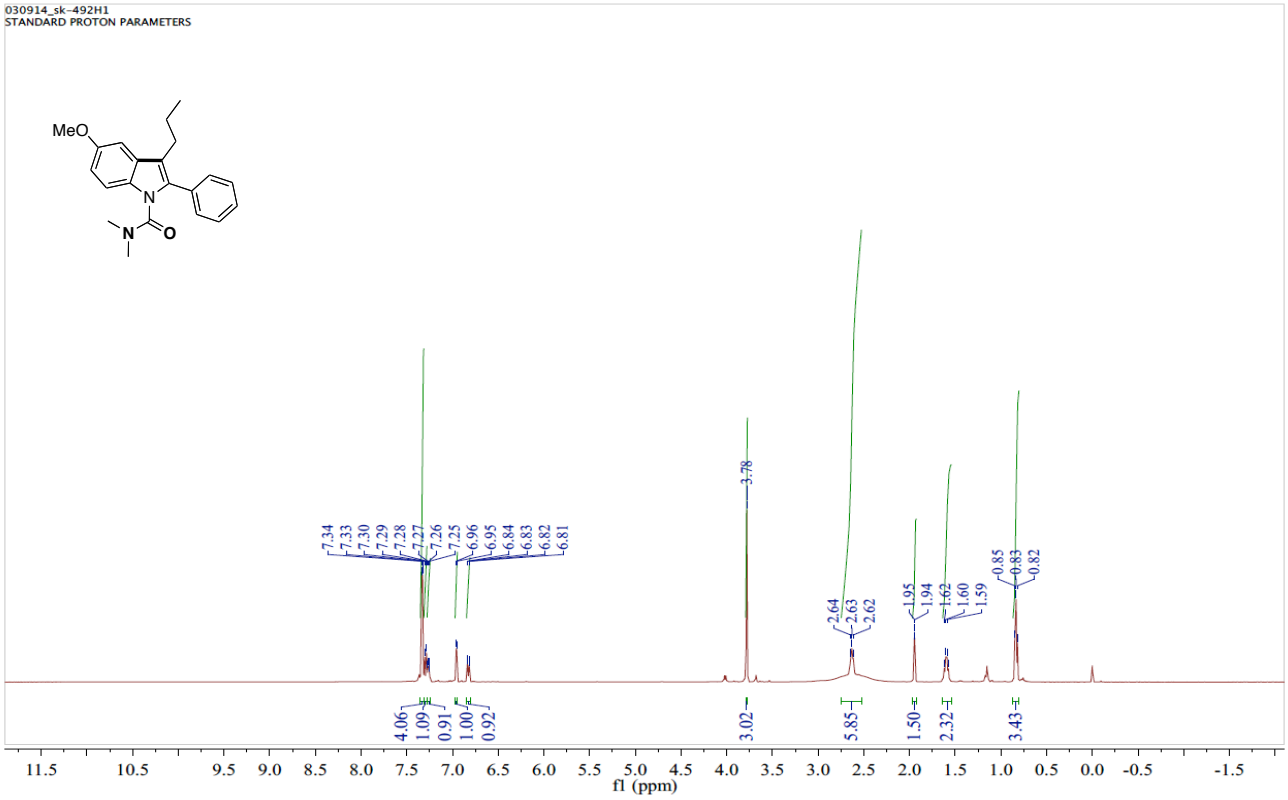

170314_sk-357AREC13
STANDARD CARBON PARAMETERS
155.02
154.49
135.43
132.08
131.06
129.32
128.99
128.42
127.90
118.58
112.83
112.55
101.61
77.41
77.16
76.91
55.97
37.24
17.76
15.44
4i
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

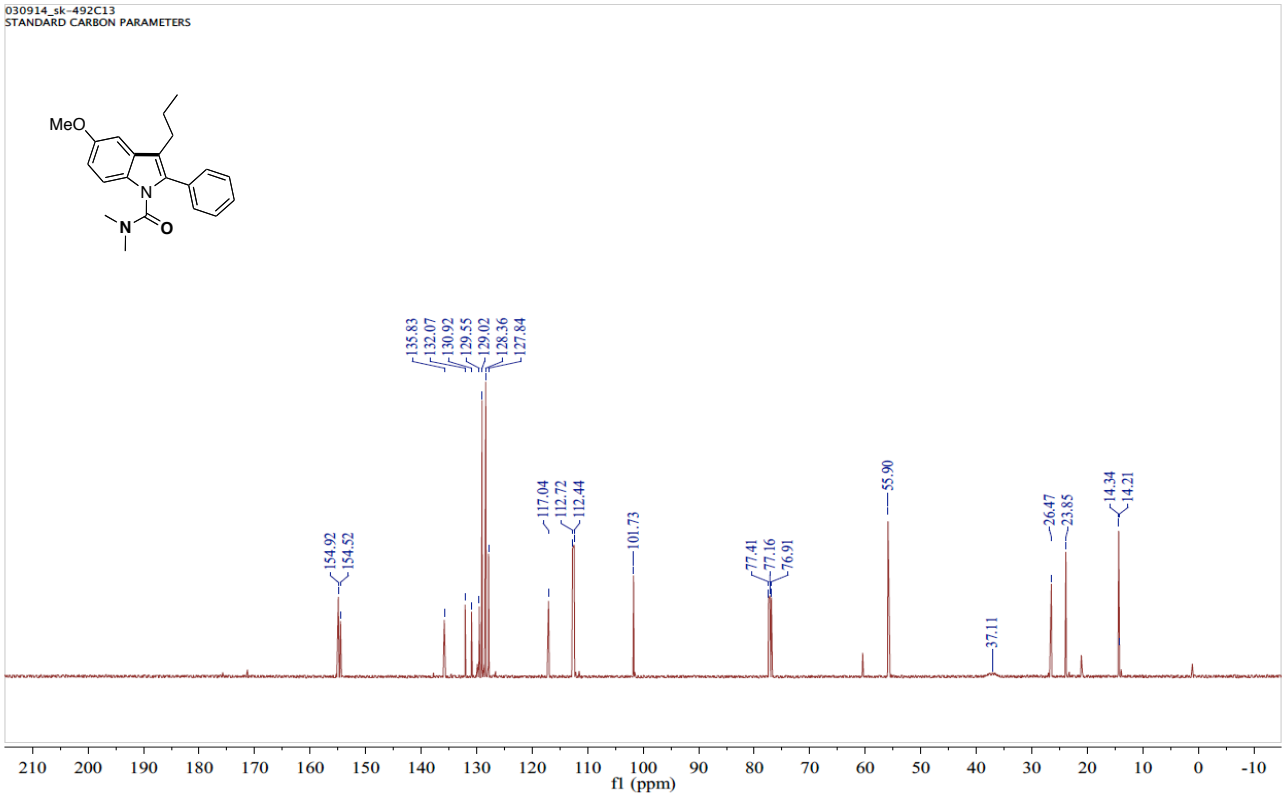

081113_sk-323H1
STANDARD PROTON PARAMETERS
7.38
7.37
7.35
7.35
7.34
7.31
7.29
7.26
7.17
7.16
7.16
7.11
7.10
7.09
7.05
7.05
7.05
7.04
6.98
6.97
6.96
6.96
3.84
3.07
2.73
4j
1.00
1.02
1.40
1.15
0.78
0.77
2.81
5.37
f1 (ppm)

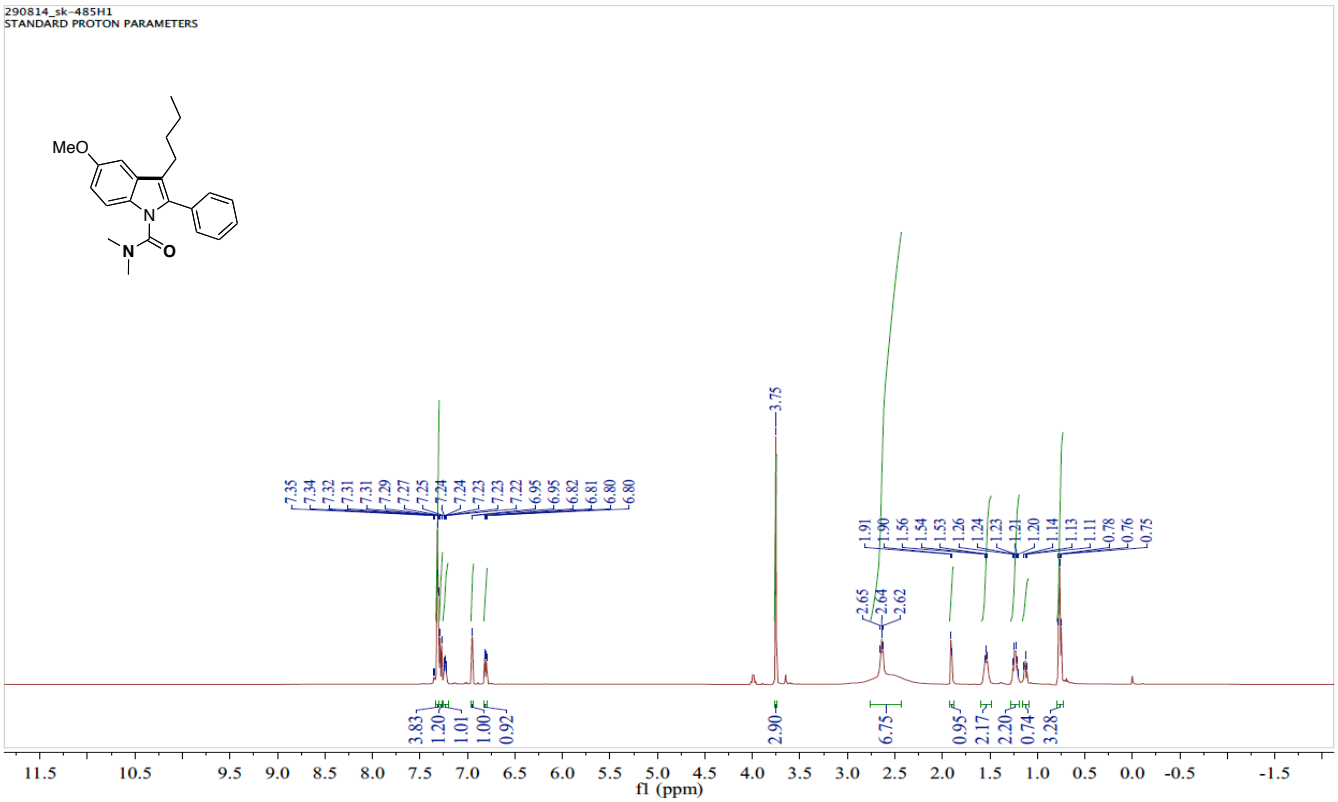

100314_sk-323REC13
STANDARD CARBON PARAMETERS
155.86
153.58
130.53
128.97
128.88
127.67
127.42
127.32
127.30
125.63
114.49
112.26
112.08
101.80
77.41
77.16
76.91
55.94
38.36
36.83
4j
f1 (ppm)

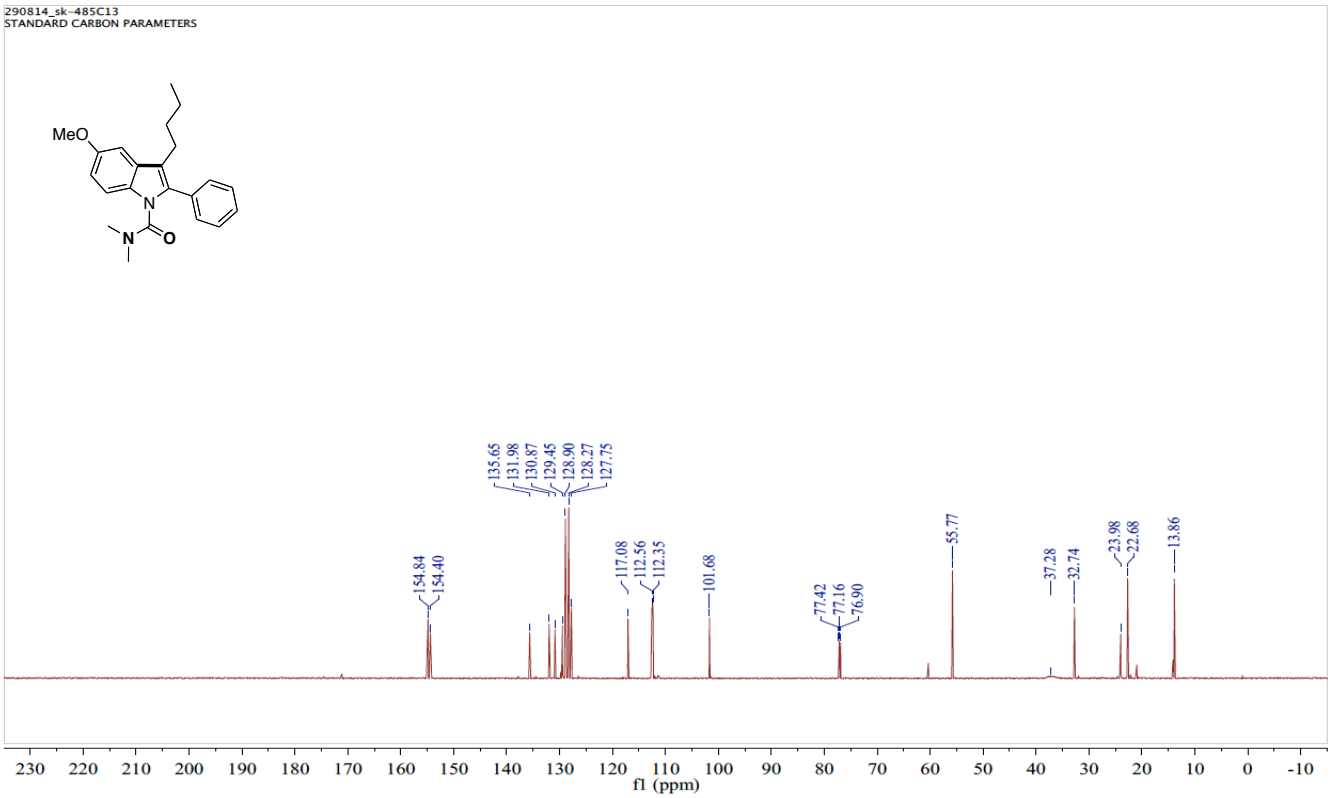

7.42
7.41
7.40
7.37
7.36
7.35
7.35
7.34
7.34
7.33
7.32
7.30
7.30
7.29
7.29
7.28
7.28
7.27
7.27
7.27
7.26
7.25
7.25
7.06
7.05
7.05
7.04
7.03
7.03
7.02
7.01
7.00
6.99
6.99
6.98
6.98
6.97
6.97
6.95
6.95
3.81
3.09
2.71
4k
1.00
0.83
0.22
0.19
0.26
0.16
0.55
0.54
0.52
0.80
fl (ppm)

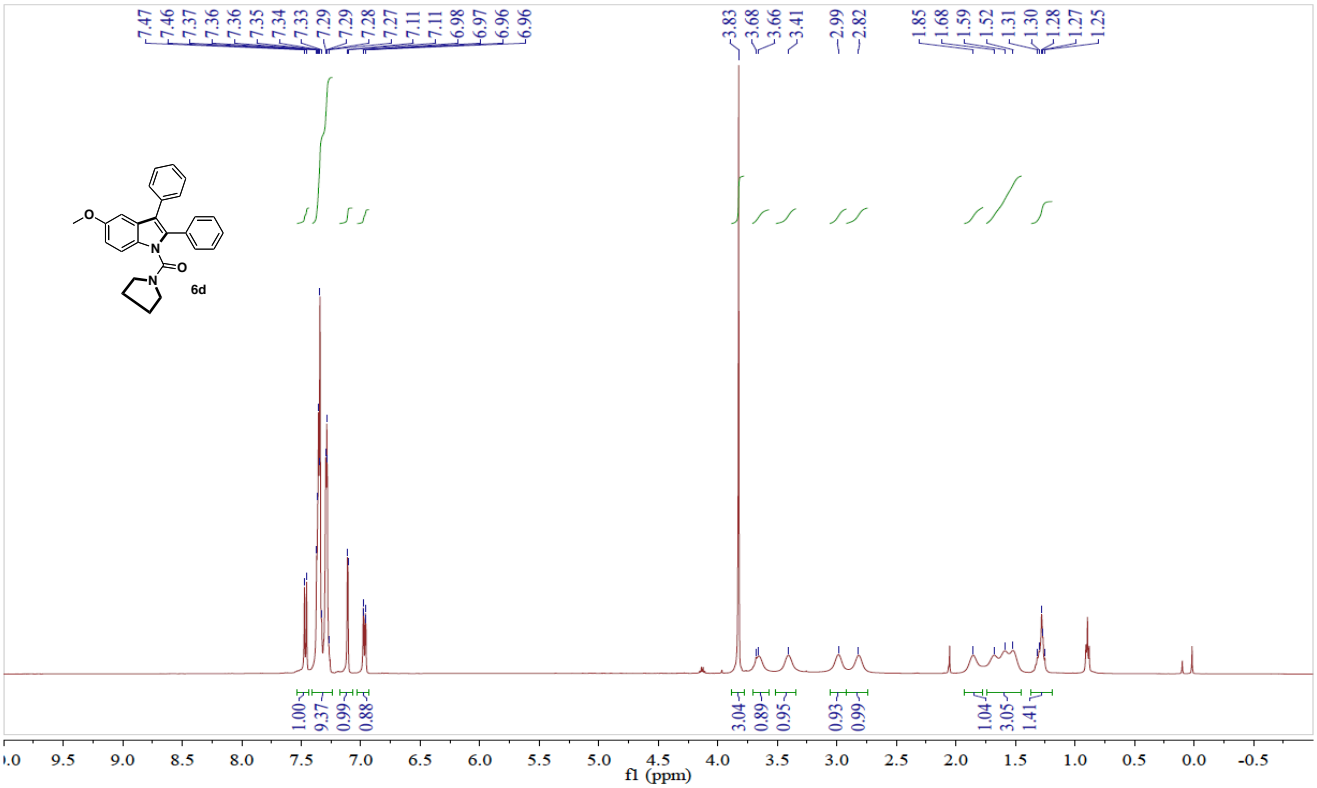

140314_sk-355REC13
STANDARD CARBON PARAMETERS
154.93
134.77
134.42
134.18
131.21
130.37
128.43
128.41
128.35
128.26
126.38
125.73
121.64
121.57
117.89
117.47
77.41
77.16
76.91
37.91
36.45
18.12
4k
fl (ppm)

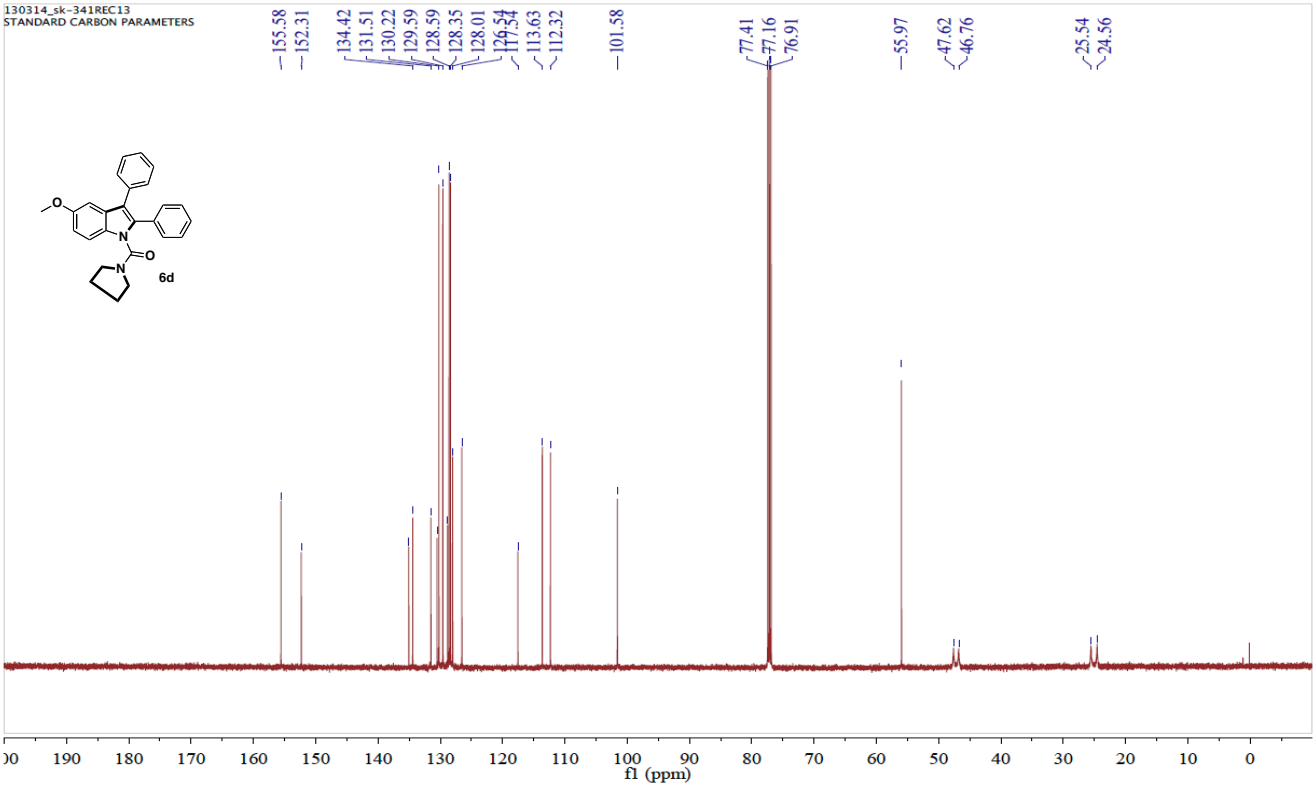

030914_sk-492H1
STANDARD PROTON PARAMETERS
MeO
N
O
7.34
7.33
7.30
7.29
7.28
7.27
7.26
7.25
6.96
6.95
6.84
6.83
6.82
6.81
3.78
2.64
2.63
2.62
1.95
1.94
1.62
1.60
1.59
0.85
0.83
0.82
4.06
1.09
0.91
1.00
0.92
3.02
5.85
1.50
2.32
3.43
f1 (ppm)

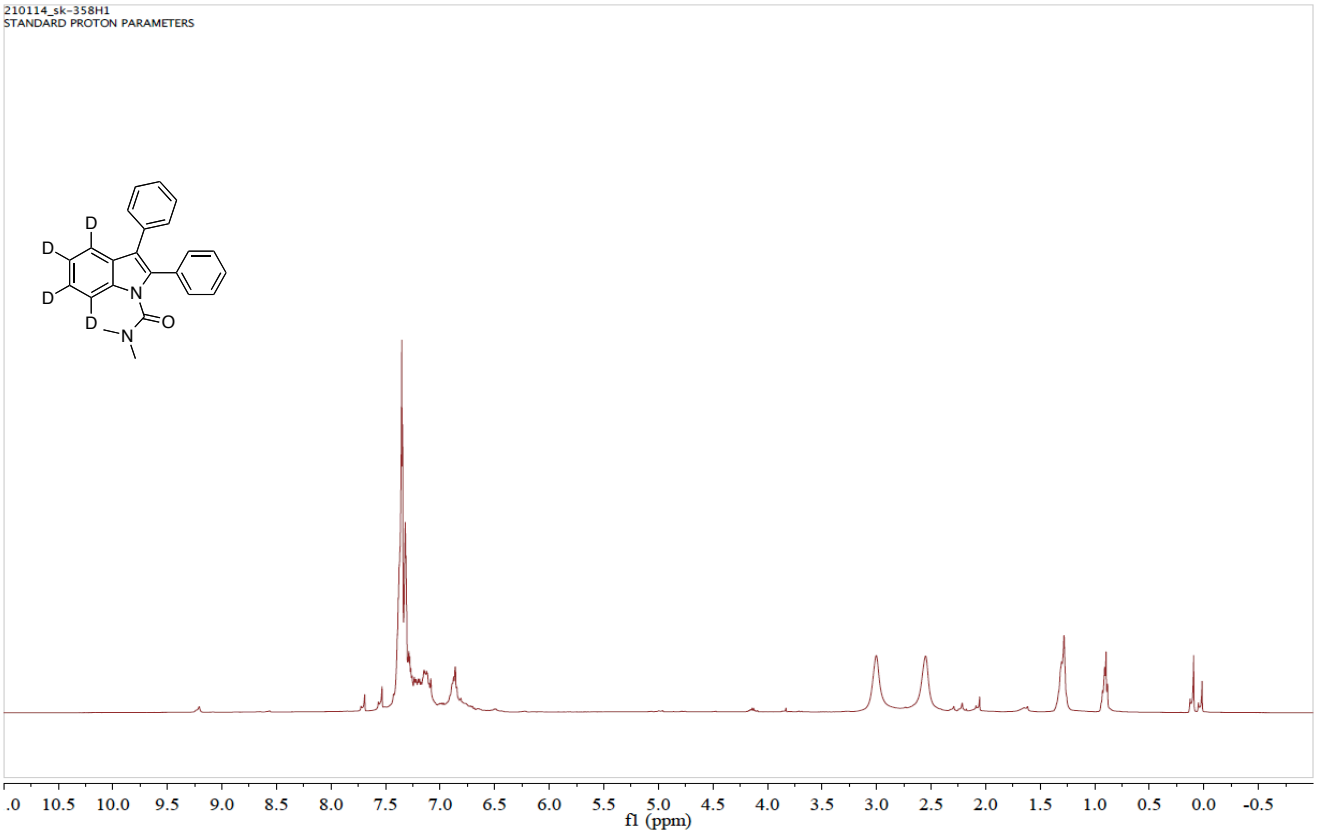

030914_sk-492C13
STANDARD CARBON PARAMETERS
MeO
N
O
135.83
132.07
130.92
129.55
129.02
128.36
127.84
117.04
112.72
112.44
101.73
77.41
77.16
76.91
55.90
37.11
26.47
23.85
14.34
14.21
154.92
154.52
f1 (ppm)

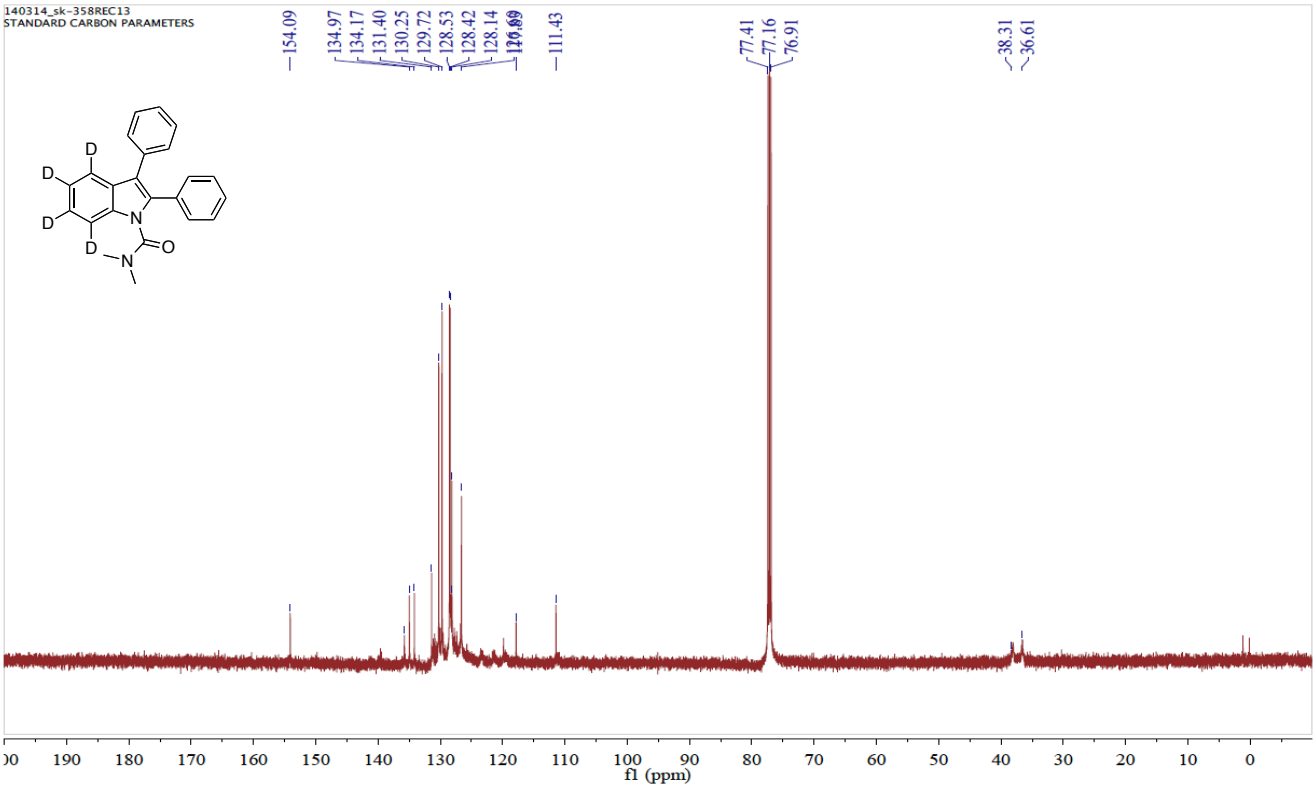

290814_sk--485H1
STANDARD PROTON PARAMETERS
MeO
N
O
7.35
7.34
7.32
7.31
7.31
7.29
7.27
7.25
7.24
7.24
7.23
7.23
7.22
6.95
6.95
6.82
6.81
6.80
6.80
3.75
2.65
2.64
2.62
1.91
1.90
1.56
1.54
1.53
1.26
1.24
1.23
1.21
1.20
1.14
1.13
1.11
0.78
0.76
0.75
3.83
1.20
1.01
1.00
0.92
2.90
6.75
0.95
2.17
2.20
0.74
3.28
fl (ppm)

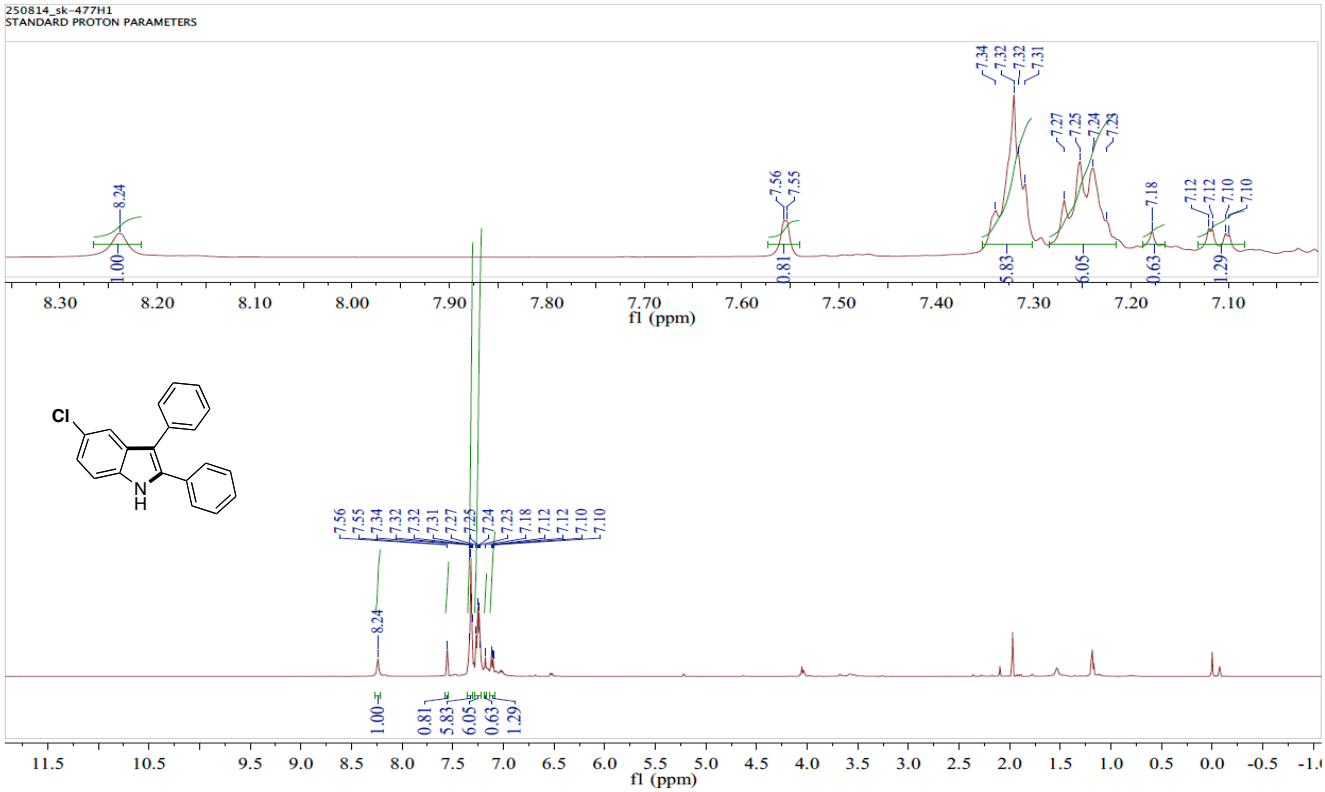

290814_sk--485C13
STANDARD CARBON PARAMETERS
MeO
N
O
135.65
131.98
130.87
129.45
128.90
128.27
127.75
154.84
154.40
117.08
112.56
112.35
101.68
77.42
77.16
76.90
55.77
37.28
32.74
23.98
22.68
13.86
fl (ppm)

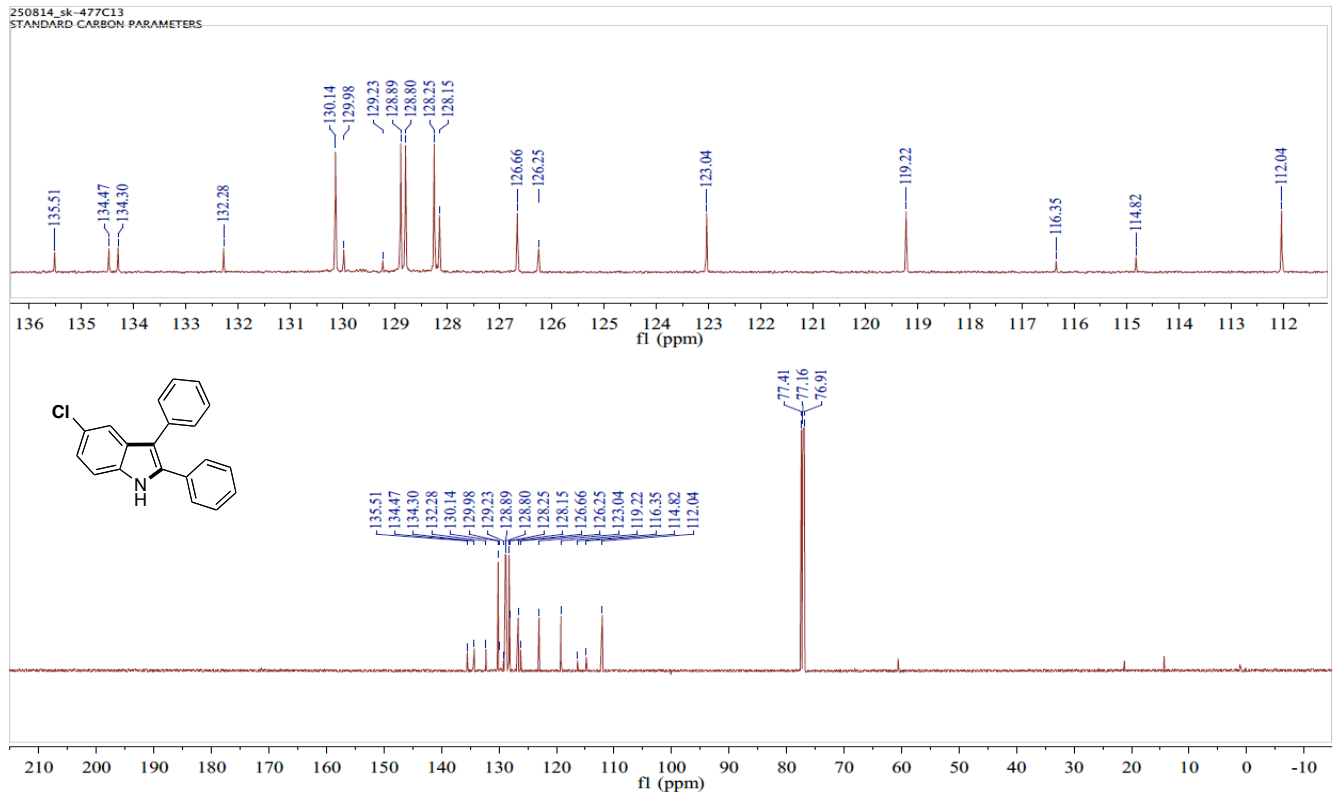

7.47
7.46
7.37
7.36
7.36
7.35
7.34
7.33
7.29
7.29
7.28
7.27
7.11
7.11
6.98
6.97
6.96
6.96
3.83
3.68
3.66
3.41
2.99
2.82
1.85
1.68
1.59
1.52
1.31
1.30
1.28
1.27
1.25
6d
1.00
9.37
0.99
0.88
3.04
0.89
0.95
0.93
0.99
1.04
3.05
1.41
f1 (ppm)


130314_sk-341REC13
STANDARD CARBON PARAMETERS
155.58
152.31
134.42
131.51
130.22
129.59
128.59
128.35
128.01
126.54
117.54
113.63
112.32
101.58
77.41
77.16
76.91
55.97
47.62
46.76
25.54
24.56
6d
f1 (ppm)


210114_sk-358H1
STANDARD PROTON PARAMETERS
D
D
D
D
N
N
O
.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 -0.5
f1 (ppm)


140314_sk-358REC13
STANDARD CARBON PARAMETERS
154.09
134.97
134.17
131.40
130.25
129.72
128.53
128.42
128.14
111.43
77.41
77.16
76.91
38.31
36.61
D
D
D
D
N
N
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


250814_sk-477H1
STANDARD PROTON PARAMETERS
fl (ppm)


250814_sk-477C13
STANDARD CARBON PARAMETERS
fl (ppm)