Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Rhodium(III)-catalyzed Cyanation of Vinylic C-H Bonds : N-Cyano-N- phenyl-p-toluenesulfonamide as cyanation reagent

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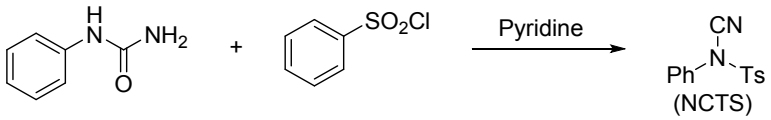
I. General Remark:

All solvents were obtained from commercial suppliers and used without further purification. $[RhCp^*(CH_3CN)_3](SbF_6)_2$ was prepared according to the literature[1]. Analytical TLC was done on pre-coated silica gel plates. Column chromatography was conducted with 300-400 mesh silica gel. ^1H NMR spectra were recorded on 400 MHz spectrometers. Chemical shifts of ^1H NMR spectra were reported in parts per million relative to tetramethylsilane (δ = 0). The following abbreviations were used to describe peak splitting patterns when appropriate: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublet. Coupling constants, *J*, were reported in hertz unit (Hz). ^{13}C NMR spectra were recorded on 101 MHz spectrometers. Chemical shifts were reported in parts per million relative to tetramethylsilane (δ = 0). High-resolution mass spectra (HRMS) were recorded on a BRUKER VPEXII spectrometer with EI and ESI mode unless otherwise stated.

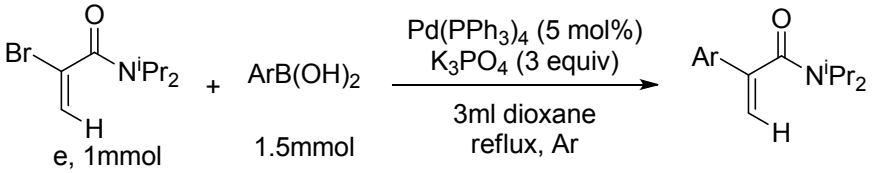
II. General Procedure for the Preparation of Starting Materials

A. Synthesis of *N*-cyano-*N*-phenyl-*p*-toluenesulfonamide (NCTS)[2]:



B. General procedure for the preparation of acrylamide.

Acrylamides (1**a**, 1**b**, 1**o** and **e**) was synthesized following the known literature procedures[3] and the other acrylamides synthesized from 2-Bromo-N,N-diisopropylacrylamide(e) and arylboronic acids by Suzuki reactions. To a 1,4-dioxane solution (3mL/mmol) of the 2-Bromo-N,N-diisopropylacrylamide (1 mmol) were added potassium phosphate (K_3PO_4, 3 equiv), the boronic acid $ArB(OH)_2$ (1.5 equiv), and $Pd(PPh_3)_4$ (0.05 equiv) at 20 °C. The reaction mixture was heated and stirred at reflux for 6 h. The reaction mixture was then allowed to cool to ambient temperature, and diethyl ether (10mL/mmol) was added. The precipitate was filtered off, washed with diethyl ether, and the filtrate was concentrated in vacuo. The residue was purified by chromatography (silica gel, petroleum ether/EtOAc) to give the aryl-substituted diisopropylacrylamide.



C. General procedure for the preparation of O-methyl oximes and Pyridines.

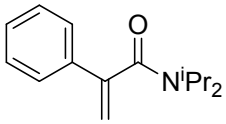
O-methyl oximes(**4a**, **4d**) was synthesized following the known literature procedures[4].
Pyridines(**4b**, **4c**) was synthesized following the known literature procedures[5].

III. General Procedure for Rhodium-Catalyzed C-H Cyanation

A 10mL Schlenk tube equipped with a magnetic stirrer was charged with $[Cp^*Rh(CH3CN)_3](SbF_6)_2$ (10 mol%), NaOAc (20 mol%), Ag_2CO_3 (20 mol%), N-cyano-N-phenyl-p-toluenesulfonamide **2** (0.2 mmol). The tube was evacuated and backfilled with argon for three times. Then acrylamide or O-methyl oxime (0.1 mmol) in DCE (0.5 mL) was added. After addition of all substrates, the reaction mixture was stirred and heated at 120°C for 24h. Then reaction was cooled to room temperature. Solvent and volatile reagents were removed by rotary evaporation and the residue was purified by flash column chromatography on silica gel to give the target product.

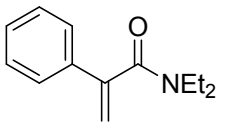
IV. NMR Spectra of Products

N,N-diisopropyl-2-phenylacrylamide (1a):



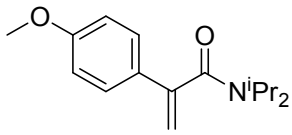
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 – 7.41 (m, 2H), 7.43 – 7.25 (m, 3H), 5.62 (s, 1H), 5.25 (s, 1H), 3.98 (dt, *J* = 13.4, 6.7 Hz, 1H), 3.43 (dt, *J* = 13.6, 6.8 Hz, 1H), 1.54 (d, *J* = 6.8 Hz, 6H), 1.01 (d, *J* = 6.7 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.05, 146.73, 135.87, 128.69, 128.39, 125.62, 111.51, 50.65, 45.56, 20.44, 20.38.

N,N-diethyl-2-phenylacrylamide (1b):



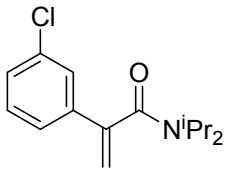
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (m, 2H), 7.39 – 7.29 (m, 3H), 5.69 (s, 1H), 5.32 (s, 1H), 3.51 (q, *J* = 7.1 Hz, 1H), 3.23 (q, *J* = 7.1 Hz, 1H), 1.22 (t, *J* = 7.1 Hz, 6H), 1.00 (t, *J* = 7.1 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.21, 145.48, 135.72, 128.75, 128.50, 125.57, 112.96, 42.75, 38.76, 13.97, 12.78.

N,N-diisopropyl-2-(4-methoxyphenyl)acrylamide (1c):



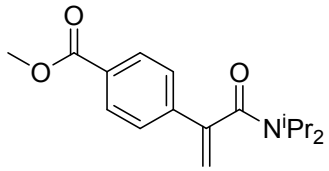
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 – 7.30 (m, 2H), 7.11 – 6.61 (m, 2H), 5.50 (s, 1H), 5.13 (s, 1H), 3.99 (dt, *J* = 13.3, 6.7 Hz, 1H), 3.81 (s, 3H), 3.43 (dt, *J* = 13.6, 6.8 Hz, 1H), 1.54 (d, *J* = 6.8 Hz, 6H), 1.02 (d, *J* = 6.7 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.46, 159.78, 145.88, 128.41, 126.93, 114.06, 109.57, 55.30 50.72, 45.61, 20.45, 20.42.
HRMS calcd for $C_{16}H_{23}NO_2[Na^+]$: 284.1626; found: 284.1621.

2-(3-chlorophenyl)-N,N-diisopropylacrylamide (1d):



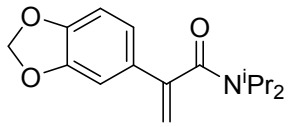
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 1.1 Hz, 1H), 7.37 – 7.20 (m, 3H), 5.64 (s, 1H), 5.31 (s, 1H), 3.94 (dt, *J* = 13.3, 6.7 Hz, 1H), 3.44 (dt, *J* = 13.6, 6.8 Hz, 1H), 1.54 (d, *J* = 6.8 Hz, 6H), 1.03 (d, *J* = 6.7 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 169.49, 145.12, 137.60, 134.72, 130.01, 128.50, 125.67, 123.89, 113.12, 50.86, 45.81, 20.36.
HRMS calcd for $C_{15}H_{20}NOCl$ $[Na^+]$: 288.1131; found: 288.1129.

methyl 4-(3-(diisopropylamino)-3-oxoprop-1-en-2-yl)benzoate (1e):



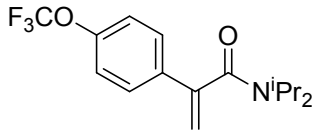
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, *J* = 8.2 Hz, 2H), 7.52 (d, *J* = 8.2 Hz, 2H), 5.73 (s, 1H), 5.38 (s, 1H), 4.10 – 3.77 (m, 4H), 3.66 – 3.27 (m, 1H), 1.54 (d, *J* = 6.7 Hz, 6H), 1.01 (d, *J* = 6.5 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.47, 167.74, 146.94, 141.28, 141.28, 131.14, 130.98, 126.68, 53.25, 51.85, 46.80, 21.46.
HRMS calcd for $C_{17}H_{23}NO_3[Na^+]$: 312.1576 ; found: 312.1574.

2-(benzo[d][1,3]dioxol-5-yl)-N,N-diisopropylacrylamide (1f):



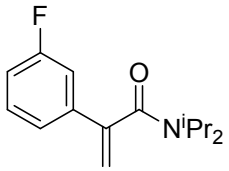
^{1}H NMR (400 MHz, $CDCl_3$) δ 6.97 (s, 1H), 6.90 (d, *J* = 8.1 Hz, 1H), 6.78 (d, *J* = 8.1 Hz, 1H), 5.97 (s, 2H), 5.49 (s, 1H), 5.16 (s, 1H), 3.99 (dt, *J* = 13.3, 6.7 Hz, 1H), 3.44 (dt, *J* = 13.6, 6.8 Hz, 1H), 1.53 (d, *J* = 6.8 Hz, 6H), 1.04 (d, *J* = 6.7 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.26, 148.11, 147.88, 145.75, 130.04, 119.94, 110.39, 108.36, 105.69, 101.25, 50.81, 45.73, 20.39.
HRMS calcd for $C_{16}H_{21}NO_3[Na^+]$: 298.1419; found: 298.1416.

N,N-diisopropyl-2-(4-(trifluoromethoxy)phenyl)acrylamide (1g):



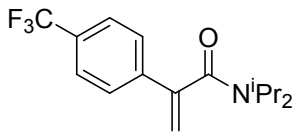
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 – 7.43 (m, 2H), 7.20 (d, *J* = 8.7 Hz, 2H), 5.61 (s, 1H), 5.29 (s, 1H), 3.97 (dt, *J* = 13.3, 6.6 Hz, 1H), 3.44 (dt, *J* = 13.6, 6.8 Hz, 1H), 1.53 (d, *J* = 6.8 Hz, 6H), 1.03 (d, *J* = 6.7 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 169.56, 149.20, 145.25, 134.61, 127.14, 121.13, 120.42 (q, *J* =255.7 Hz), 112.41, 50.78, 45.72, 20.42.
^{19}F NMR (376 MHz, $CDCl_3$) δ -57.84.
HRMS calcd for $C_{16}H_{20}NO_2F_3[Na^+]$: 338.1344; found: 338.1340.

2-(3-fluorophenyl)-4-isopropyl-5-methylhex-1-en-3-one (1h):



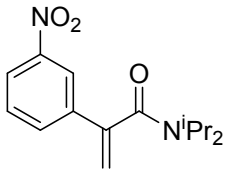
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (m, 1H), 7.22 (d, *J* = 7.9 Hz, 1H), 7.20 – 7.13 (m, 1H), 7.00 (m, 1H), 5.64 (s, 1H), 5.30 (s, 1H), 3.96 (dt, *J* = 13.3, 6.7 Hz, 1H), 3.44 (dt, *J* = 13.6, 6.8 Hz, 1H), 1.54 (d, *J* = 6.8 Hz, 6H), 1.03 (d, *J* = 6.7 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 169.53, 163.04 (d, *J* = 246.0 Hz), 145.43 (d, *J* = 2.4 Hz), 138.08 (d, *J* = 7.6 Hz), 130.28 (d, *J* = 8.3 Hz), 121.44 (d, *J* = 2.9 Hz), 115.36 (d, *J* = 21.3 Hz), 112.87, 112.50 (d, *J* = 22.5 Hz), 50.82, 45.77, 20.38.
^{19}F NMR (376 MHz, $CDCl_3$) δ -112.59.
HRMS calcd for $C_{15}H_{20}NOF$ $[Na^+]$: 272.1427; found: 272.1422.

N,N-diisopropyl-2-(4-(trifluoromethyl)phenyl)acrylamide (1i):



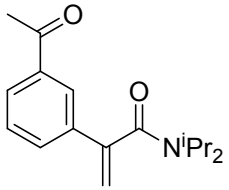
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 8.3 Hz, 2H), 7.57 (d, *J* = 8.4 Hz, 2H), 5.72 (s, 1H), 5.38 (s, 1H), 3.95 (dt, *J* = 13.2, 6.6 Hz, 1H), 3.45 (dt, *J* = 13.4, 6.7 Hz, 1H), 1.54 (d, *J* = 6.8 Hz, 6H), 1.03 (d, *J* = 6.6 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 169.30, 145.39, 139.38, 130.35 (q, *J* = 32.6 Hz), 125.97, 125.76 (q, *J* = 3.7 Hz), 124.02 (q, *J* =270 Hz), 113.91, 50.84, 45.80, 20.44 , 20.40.
^{19}F NMR (376 MHz, $CDCl_3$) δ -62.66.
HRMS calcd for $C_{16}H_{20}NOF_3$ [Na^+]: 322.1395; found: 322.1398.

N,N-diisopropyl-2-(3-nitrophenyl)acrylamide (1j):



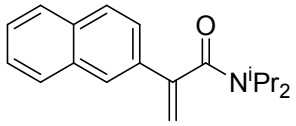
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 (t, *J* = 1.8 Hz, 1H), 8.18 (dd, *J* = 8.2, 1.3 Hz, 1H), 7.79 (d, *J* = 7.8 Hz, 1H), 7.55 (t, *J* = 8.0 Hz, 1H), 5.79 (s, 1H), 5.44 (s, 1H), 3.96 (dt, *J* = 13.2, 6.6 Hz, 1H), 3.48 (dt, *J* = 13.5, 6.7 Hz, 1H), 1.56 (d, *J* = 6.7 Hz, 6H), 1.06 (d, *J* = 6.6 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 168.84, 148.66, 144.33, 137.61, 131.56, 129.82, 123.16, 120.52, 114.29, 50.97, 45.96, 20.51, 20.39.
HRMS calcd for $C_{15}H_{20}N_2O_3$[Na^+]: 299.1372; found: 299.1368.

2-(3-acetylphenyl)-N,N-diisopropylacrylamide (1k):



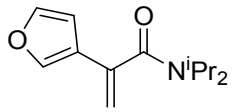
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (t, *J* = 1.7 Hz, 1H), 7.95 – 7.84 (m, 1H), 7.67 (ddd, *J* = 7.8, 1.7, 1.1 Hz, 1H), 7.47 (t, *J* = 7.8 Hz, 1H), 5.72 (s, 1H), 5.35 (s, 1H), 3.97 (dt, *J* = 13.3, 6.6 Hz, 1H), 3.46 (dt, *J* = 13.6, 6.8 Hz, 1H), 2.61 (s, 3H), 1.56 (d, *J* = 6.8 Hz, 6H), 1.02 (d, *J* = 6.7 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 197.77, 169.55, 145.70, 137.54, 136.41, 130.13, 129.07, 128.23, 125.48, 112.76, 50.80, 45.71, 26.68, 20.42.
HRMS calcd for $C_{17}H_{23}NO_2$[Na^+]: 296.1626; found: 296.1626.

N,N-diisopropyl-2-(naphthalen-2-yl)acrylamide (1l):



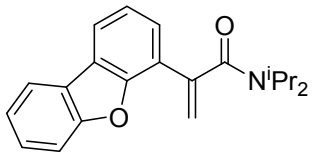
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 – 7.73 (m, 4H), 7.63 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.53 – 7.33 (m, 2H), 5.76 (s, 1H), 5.36 (s, 1H), 4.03 (dt, *J* = 13.4, 6.7 Hz, 1H), 3.47 (dt, *J* = 13.6, 6.8 Hz, 1H), 1.60 (d, *J* = 6.8 Hz, 6H), 1.00 (d, *J* = 6.7 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.16, 146.54, 133.32, 133.21, 133.05, 128.47, 128.39, 127.61, 126.38 (2C), 125.24, 123.07, 112.01, 50.79, 45.69, 20.49, 20.42.
HRMS calcd for $C_{19}H_{23}NO[Na^+]$: 304.1677; found: 304.1672.

2-(furan-3-yl)-N,N-diisopropylacrylamide (1m):



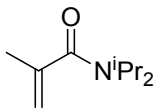
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (s, 1H), 7.39 (t, *J* = 1.6 Hz, 1H), 6.62 – 6.52 (m, 1H), 5.39 (s, 1H), 5.09 (s, 1H), 4.23 – 3.88 (m, 1H), 3.88 – 3.22 (m, 1H), 1.52 (d, *J* = 6.2 Hz, 6H), 1.11 (d, *J* = 6.0 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 169.57, 143.69, 140.31, 137.98, 122.99, 110.04, 107.57, 50.73, 45.65, 20.62, 20.44.
HRMS calcd for $C_{13}H_{19}NO_2[Na^+]$: 244.1313; found: 244.1309.

2-(dibenzo[b,d]furan-4-yl)-N,N-diisopropylacrylamide (1n):



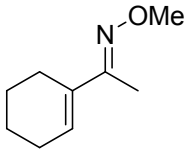
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.6 Hz, 1H), 7.90 (dd, *J* = 7.6, 1.0 Hz, 1H), 7.59 (d, *J* = 8.2 Hz, 1H), 7.47 (m, 2H), 7.35 (m, 2H), 6.34 (s, 1H), 5.64 (s, 1H), 4.36 – 3.83 (m, 1H), 3.48 (m, 1H), 1.60 (d, *J* = 6.8 Hz, 6H), 1.03 (d, *J* = 6.7 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 169.94, 155.99, 153.25, 142.21, 127.31, 125.94, 125.06, 123.83, 122.98, 122.95, 120.90, 120.68, 120.62, 116.63, 111.75, 50.87, 45.76, 20.43, 20.33.
HRMS calcd for $C_{21}H_{23}NO_2[Na^+]$: 344.1626; found: 344.1628.

N,N-diisopropylmethacrylamide (1o):



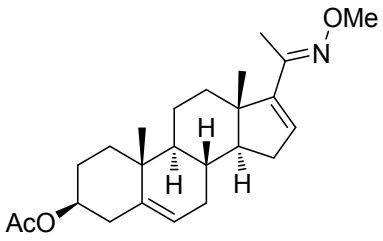
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.01 (d, *J* = 1.4 Hz, 1H), 4.92 (d, *J* = 1.0 Hz, 1H), 4.11 (s, 1H), 3.41 (s, 1H), 2.06 – 1.83 (m, 3H), 1.30 (d, *J* = 84.8 Hz, 12H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 172.07, 142.94, 112.51, 50.28, 45.27, 20.54.

(E)-1-(cyclohex-1-en-1-yl)ethanone O-methyl oxime (4a):



^{1}H NMR (400 MHz, $CDCl_3$) δ 6.12 (m, 1H), 3.88 (s, 3H), 2.36 – 2.23 (m, 2H), 2.17 (m, 2H), 1.93 (s, 3H), 1.82 – 1.29 (m, 4H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 156.08, 134.89, 129.01, 61.52, 25.98, 24.50, 22.46, 22.20, 10.33.

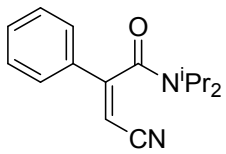
(3S,8R,9S,10R,13S,14S)-17-((E)-1-(methoxyimino)ethyl)-10,13-dimethyl-2,3,4,7,8,9,10,11,12,13,14,15-dodecahydro-1H-cyclopenta[a]phenanthren-3-yl acetate (4d):



^{1}H NMR (400 MHz, $CDCl_3$) δ 6.00 (s, 1H), 5.40 (d, *J* = 3.7 Hz, 1H), 4.91 – 4.41 (m, 1H), 3.88 (s, 3H), 2.51 (d, *J* = 12.4 Hz, 1H), 2.34 (d, *J* = 6.1 Hz, 2H), 2.25 – 2.15 (m, 1H), 2.04 (s, 3H), 2.04 – 1.82 (m, 7H), 1.65 (m, 15.6, 9.3 Hz, 6H), 1.50 – 1.33 (m, 2H), 1.21 – 1.10 (m, 1H), 1.06 (s, 3H), 0.97 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.56, 152.17, 151.68, 140.13, 131.88, 122.34, 73.96, 61.66, 57.01, 50.40, 46.70, 38.17, 36.90, 36.79, 35.63, 31.61 (2C), 30.30, 27.78, 21.47, 20.87, 19.23, 15.86, 11.76.
HRMS calcd for $C_{24}H_{35}NO_2[Na^+]$: 408.2515; found: 408.2511.

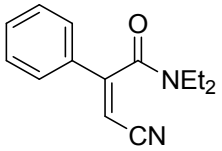
III. General Procedure for Rhodium-Catalyzed C-H Cyanation and NMR Spectra of Products

(Z)-3-cyano-N,N-diisopropyl-2-phenylacrylamide (3a):



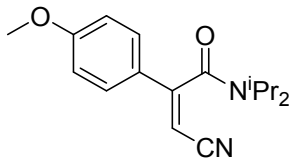
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 – 7.35 (m, 5H), 5.76 (s, 1H), 3.75 (dt, *J* = 13.3, 6.6 Hz, 1H), 3.53 (dt, *J* = 13.7, 6.8 Hz, 1H), 1.61 (d, *J* = 6.8 Hz, 6H), 1.12 (s, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 165.31, 158.51, 132.32, 131.43, 129.32, 126.30, 116.37, 92.32), 51.45, 46.31, 20.67, 20.17.
HRMS calcd for $C_{16}H_{20}N_2O[Na^+]$: 279.1473; found: 279.1466.

(Z)-3-cyano-N,N-diethyl-2-phenylacrylamide (3b):



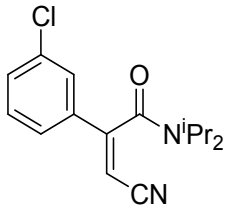
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 – 7.34 (m, 5H), 5.83 (s, 1H), 3.60 (q, *J* = 7.1 Hz, 2H), 3.26 (q, *J* = 7.1 Hz, 2H), 1.30 (t, *J* = 7.2 Hz, 6H), 1.10 (t, *J* = 7.2 Hz, 6H) (Note: Because of a trace of impurities in petroleum ether, the shape of peak is irregular.)
^{13}C NMR (101 MHz, $CDCl_3$) δ 165.65, 157.98, 132.41, 131.51, 129.39, 126.33, 116.08, 93.87, 42.92, 39.03, 13.90, 12.57.
HRMS calcd for $C_{14}H_{16}N_2O$ $[Na^+]$: 251.1160; found: 251.1163.

(Z)-3-cyano-N,N-diisopropyl-2-(4-methoxyphenyl)acrylamide (3c):



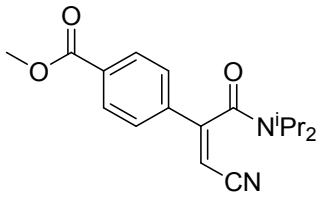
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.9 Hz, 2H), 6.93 (d, *J* = 8.9 Hz, 2H), 5.62 (s, 1H), 3.85 (s, 3H), 3.76 (dt, *J* = 13.2, 6.6 Hz, 1H), 3.53 (dt, *J* = 13.6, 6.8 Hz, 1H), 1.61 (d, *J* = 6.8 Hz, 6H), 1.52 – 0.85 (m, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 165.66, 162.17, 157.83, 128.10, 124.73, 116.81, 114.70, 89.56, 55.49, 51.45, 46.31, 20.74, 20.20.
HRMS calcd for $C_{17}H_{22}N_2O_2[Na^+]$: 309.1579; found: 309.1578.

(Z)-2-(3-chlorophenyl)-3-cyano-N,N-diisopropylacrylamide (3d):



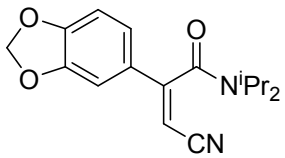
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (s, 1H), 7.47 – 7.42 (m, 1H), 7.38 (m, 2H), 5.76 (s, 1H), 3.72 (dt, *J* = 13.2, 6.6 Hz, 1H), 3.54 (dt, *J* = 13.6, 6.8 Hz, 1H), 1.60 (d, *J* = 6.8 Hz, 6H), 1.14 (s, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 164.67, 157.03, 135.45, 134.16, 131.34, 130.62, 126.21, 124.59, 115.90, 93.82, 51.56, 46.46, 20.74, 20.13.
HRMS calcd for $C_{16}H_{19}N_2OCl$ [Na^+]: 313.1084; found: 313.1079.

(Z)-methyl 4-(1-cyano-3-(diisopropylamino)-3-oxoprop-1-en-2-yl)benzoate (3e):



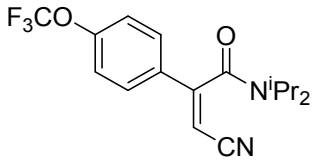
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, *J* = 8.5 Hz, 2H), 7.58 (d, *J* = 8.5 Hz, 2H), 5.84 (s, 1H), 3.94 (s, 3H), 3.71 (dt, *J* = 13.2, 6.6 Hz, 1H), 3.54 (dt, *J* = 13.6, 6.8 Hz, 1H), 1.61 (d, *J* = 6.8 Hz, 6H), 1.33 – 0.96 (m, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 166.02, 164.73, 157.39, 136.44, 132.47, 130.47, 126.32, 115.88, 94.55, 52.49, 51.56, 46.47, 20.71, 20.14.
HRMS calcd for $C_{18}H_{22}N_2O_3$[Na^+]: 337.1528; found: 337.1530.

(Z)-2-(benzo[d][1,3]dioxol-5-yl)-3-cyano-N,N-diisopropylacrylamide (3f):



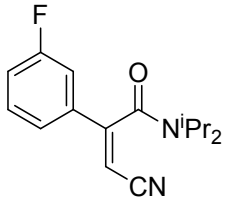
^{1}H NMR (400 MHz, $CDCl_3$) δ 6.94 (dd, *J* = 8.2, 1.7 Hz, 1H), 6.90 (d, *J* = 1.6 Hz, 1H), 6.76 (d, *J* = 8.2 Hz, 1H), 5.97 (s, 2H), 5.51 (s, 1H), 3.67 (dt, *J* = 13.2, 6.6 Hz, 1H), 3.45 (dt, *J* = 13.6, 6.8 Hz, 1H), 1.53 (d, *J* = 6.8 Hz, 6H), 1.10 (s, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 164.39, 156.85, 149.49, 147.70, 125.46, 121.11, 115.58, 107.81, 104.61, 100.94, 89.36, 50.45, 45.31, 20.04, 19.14.
HRMS calcd for $C_{17}H_{20}N_2O_3$[Na^+]: 323.1372; found: 323.1377.

(Z)-3-cyano-N,N-diisopropyl-2-(4-(trifluoromethoxy)phenyl)acrylamide (3g):



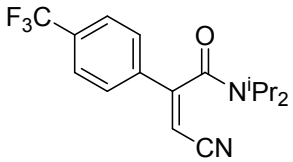
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.8 Hz, 2H), 7.28 (d, *J* = 9.0 Hz, 2H), 5.73 (s, 1H), 3.74 (dt, *J* = 13.2, 6.6 Hz, 1H), 3.54 (dt, *J* = 13.6, 6.8 Hz, 1H), 1.61 (d, *J* = 6.8 Hz, 6H), 1.15 (s, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 164.83 (s), 156.86 (s), 151.28 (s), 130.91 (s), 128.07 (s), 121.37 (s), 120.28 (q, *J*=257Hz), 115.98 (s), 93.27 (s), 51.58 (s), 46.49 (s), 21.07 (s), 20.16 (s).
^{19}F NMR (376 MHz, $CDCl_3$) δ -57.72.
HRMS calcd for $C_{17}H_{19}N_2O_2F_3[Na^+]$: 363.1296; found: 363.1293.

(Z)-3-cyano-2-(3-fluorophenyl)-N,N-diisopropylacrylamide (3h):



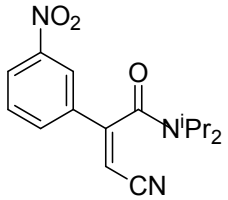
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 – 7.35 (m, 1H), 7.29 (d, *J* = 7.8 Hz, 1H), 7.24 – 7.11 (m, 2H), 5.76 (s, 1H), 3.73 (dt, *J* = 13.2, 6.6 Hz, 1H), 3.54 (dt, *J* = 13.6, 6.8 Hz, 1H), 1.61 (d, *J* = 6.8 Hz, 6H), 1.14 (s, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 164.75, 163.02 (d, *J* = 248.4 Hz), 157.22 (d, *J* = 2.6 Hz), 134.55 (d, *J* = 7.7 Hz), 131.06 (d, *J* = 8.3 Hz), 122.21 (d, *J* = 3.0 Hz), 118.40 (d, *J* = 21.3 Hz), 115.93, 113.20 (d, *J* = 23.2 Hz), 93.79, 51.57, 46.46, 20.74, 20.14.
^{19}F NMR (376 MHz, $CDCl_3$) δ -110.65 – -110.91 (m).
HRMS calcd for $C_{16}H_{19}N_2OF[Na^+]$: 297.1379; found: 297.1381.

(Z)-3-cyano-N,N-diisopropyl-2-(4-(trifluoromethyl)phenyl)acrylamide (3i):



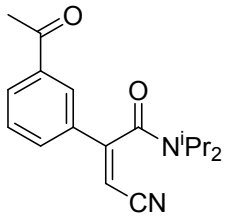
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.3 Hz, 2H), 7.63 (d, *J* = 8.3 Hz, 2H), 5.84 (s, 1H), 3.72 (dt, *J* = 13.2, 6.6 Hz, 1H), 3.55 (dt, *J* = 13.6, 6.8 Hz, 1H), 1.61 (d, *J* = 6.8 Hz, 6H), 1.14 (s, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 164.56, 156.93, 135.83, 132.92 (q, *J* = 33.0 Hz), 126.70, 126.35 (q, *J* = 3.7 Hz), 123.5 (q, *J* =271.0 Hz), 115.73, 94.89, 51.62, 46.52, 20.77, 20.13.
^{19}F NMR (376 MHz, $CDCl_3$) δ -63.07.
HRMS calcd for $C_{17}H_{19}N_2OF_3[Na^+]$: 347.1347; found: 347.1344.

(Z)-5-isopropyl-6-methyl-3-(3-nitrophenyl)-4-oxohept-2-enenitrile (3j):



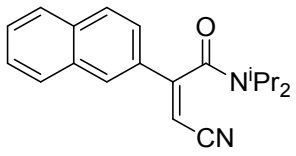
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 (s, 1H), 8.33 (dd, *J* = 8.1, 1.7 Hz, 1H), 7.85 (d, *J* = 7.7 Hz, 1H), 7.66 (t, *J* = 8.0 Hz, 1H), 5.90 (s, 1H), 3.74 (dt, *J* = 13.2, 6.6 Hz, 1H), 3.57 (dt, *J* = 13.6, 6.8 Hz, 1H), 1.63 (d, *J* = 6.8 Hz, 6H), 1.17 (s, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 164.17, 155.80, 148.81, 134.20, 131.98, 130.60, 125.70, 121.14, 115.47, 95.35, 51.77, 46.68, 20.87, 20.13.
HRMS calcd for $C_{16}H_{19}N_3O_3[Na^+]$: 324.1324; found: 324.1327.

(Z)-2-(3-acetylphenyl)-3-cyano-N, N-diisopropylacrylamide (3k):



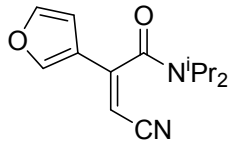
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (s, 1H), 8.04 (d, *J* = 7.7 Hz, 1H), 7.71 (d, *J* = 7.9 Hz, 1H), 7.56 (t, *J* = 7.8 Hz, 1H), 5.86 (s, 1H), 3.74 (dt, *J* = 13.2, 6.6 Hz, 1H), 3.56 (dt, *J* = 13.6, 6.8 Hz, 1H), 2.63 (s, 3H), 1.63 (d, *J* = 6.8 Hz, 6H), 1.14 (s, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 196.91, 164.87, 157.34, 137.93, 133.00, 130.95, 130.50, 129.76, 125.96, 115.95, 93.70, 51.60, 46.48, 26.65, 20.76, 20.14.
HRMS calcd for $C_{18}H_{22}N_2O_2[Na^+]$: 321.1579; found: 321.1578.

(Z)-3-cyano-N, N-diisopropyl-2-(naphthalen-2-yl) acrylamide (3l):



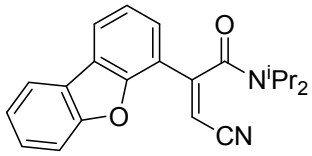
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (s, 1H), 7.93 – 7.80 (m, 3H), 7.56 (m, 3H), 5.87 (s, 1H), 3.93 – 3.69 (m, 1H), 3.67 – 3.34 (m, 1H), 1.67 (d, *J* = 6.8 Hz, 6H), 1.12 (s, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 165.43, 158.44, 134.45, 132.98, 129.61, 129.34, 129.00, 128.09, 127.81, 127.72, 127.20, 122.09, 116.49, 92.49, 51.57, 46.44, 20.77, 20.26.
HRMS calcd for $C_{20}H_{22}N_2O_2[Na^+]$: 329.1630; found: 329.1628.

(Z)-3-cyano-2-(furan-3-yl)-N,N-diisopropylacrylamide (3m):



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 1.8 Hz, 1H), 6.62 (d, *J* = 1.8 Hz, 1H), 5.85 (s, 1H), 5.46 (s, 1H), 4.03 (s, 1H), 3.48 (s, 1H), 1.51 (d, *J* = 5.8 Hz, 6H), 1.15 (d, *J* = 5.6 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 167.42, 147.27, 135.65, 133.32, 122.52, 116.63, 111.52, 110.90, 50.98, 46.11, 20.62, 20.37.
HRMS calcd for $C_{14}H_{18}N_2O_2[Na^+]$: 269.1266; found: 269.1269.

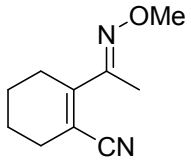
(Z)-3-(dibenzo[b,d]furan-4-yl)-5-isopropyl-6-methyl-4-oxohept-2-enenitrile (3n):



^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (dd, *J* = 7.6, 1.2 Hz, 1H), 8.01 – 7.96 (m, 1H), 7.65 (d, *J* = 8.3 Hz, 1H), 7.58 – 7.50 (m, 1H), 7.48 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.41 (m, 2H), 3.86 (dt, *J* = 13.2, 6.6 Hz, 1H), 3.59 (dt, *J* = 13.7, 6.8 Hz, 1H), 1.68 (d, *J* = 6.8 Hz, 6H), 1.15 (s, 6H).

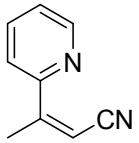
^{13}C NMR (101 MHz, $CDCl_3$) δ 165.21, 155.91, 154.06, 153.80, 128.07, 127.36, 125.85, 123.73, 123.63, 123.36, 123.07, 120.89, 117.45, 116.77, 111.94, 96.67, 51.65 , 46.50, 20.76, 20.22.
HRMS calcd for $C_{22}H_{22}N_2O_2[Na^+]$: 369.1579; found: 369.1583.

(E)-2-(1-(methoxyimino)ethyl)cyclohex-1-enecarbonitrile (5a):



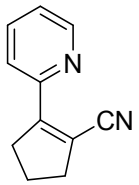
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.95 (s, 3H), 2.35 (dt, *J* = 6.0, 3.0 Hz, 4H), 2.05 (s,3H), 1.69 (s,4H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 154.82, 150.72, 118.86, 109.79, 61.97, 28.43, 27.90, 21.14, 21.10, 13.55.
HRMS calcd for $C_{10}H_{14}N_2O[Na^+]$: 201.1004; found: 201.0999.

(Z)-3-(pyridin-2-yl)but-2-enenitrile, 5b



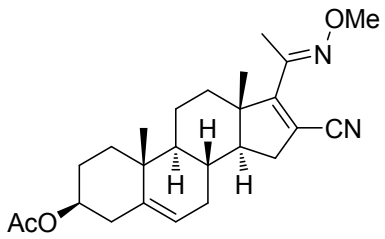
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.71 (d, *J* = 4.4 Hz, 1H), 7.78 (m, 2H), 7.34 (d, *J* = 4.9 Hz, 1H), 5.58 (s, 1H), 2.38 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 159.40 (s), 154.86 (s), 149.51 (s), 136.74 (s), 124.33 (s), 122.55 (s), 117.33 (s), 97.08 (s), 22.92 (s).
HRMS calcd for $C_9H_8N_2$ [Na^+]: 167.0585; found: 167.0581.

2-(pyridin-2-yl)cyclopent-1-enecarbonitrile, 5c

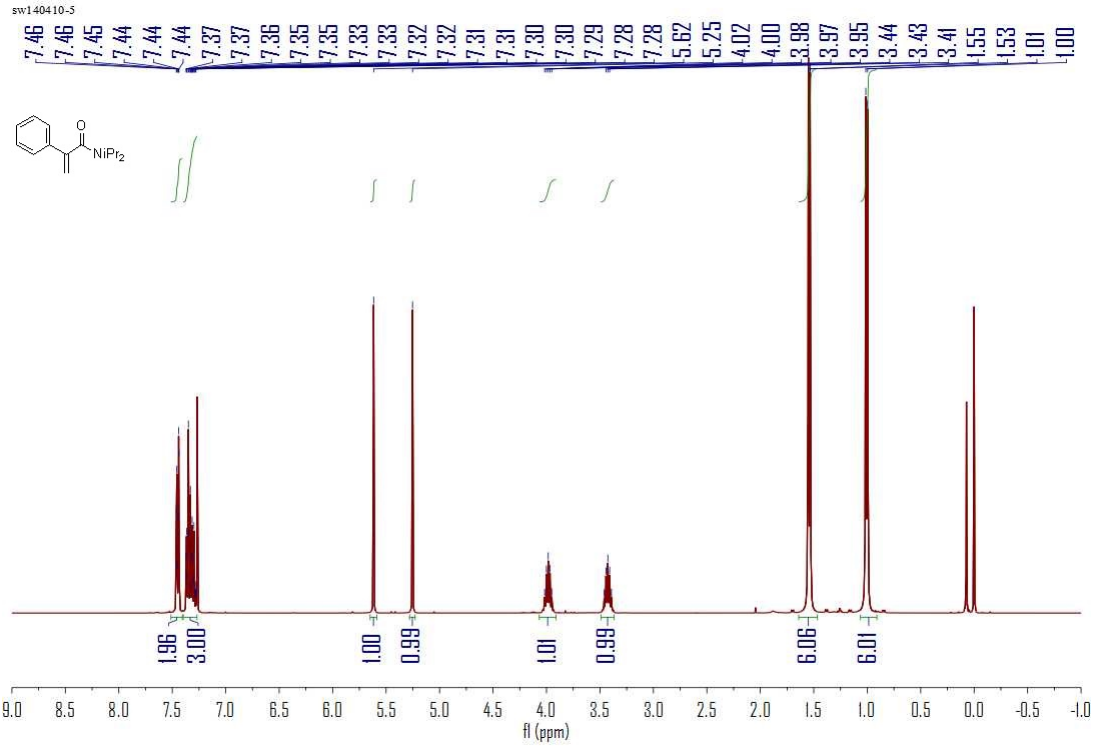


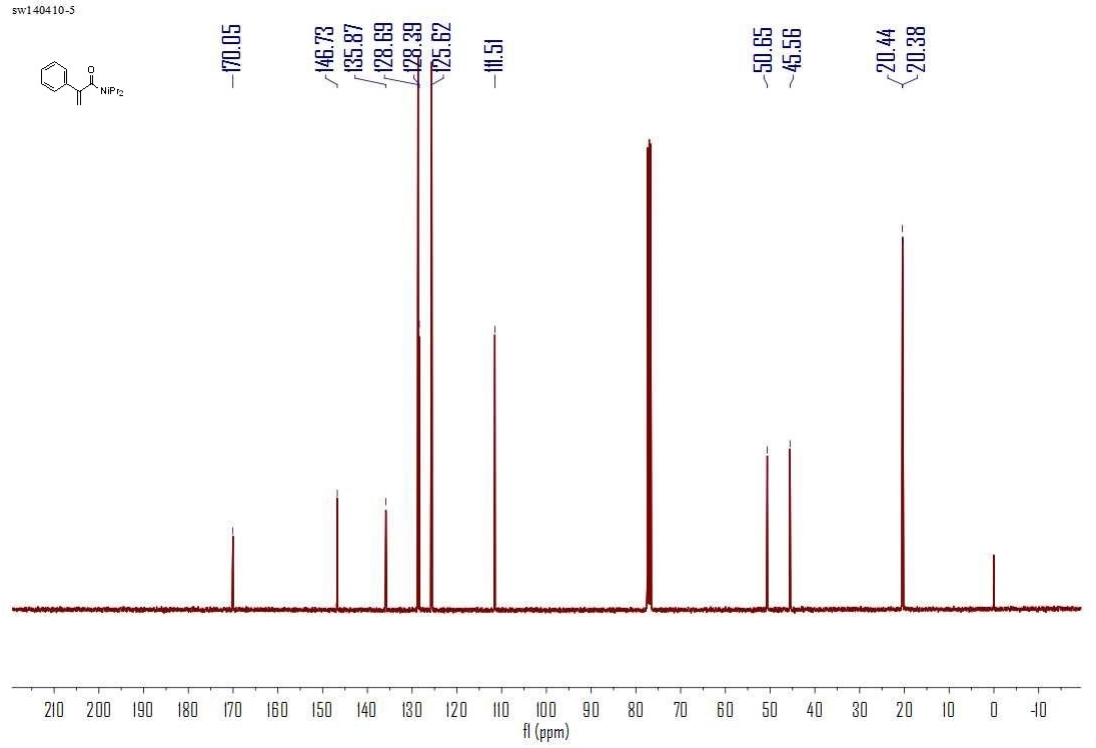
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.62 (d, *J* = 4.3 Hz, 1H), 7.78 (d, *J* = 7.9 Hz, 1H), 7.68 (m, 1H), 7.21 (m, 1H), 3.06 – 2.94 (m, 2H), 2.87 – 2.74 (m, 2H), 2.10 – 1.96 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 157.86 (s), 151.82 (s), 149.60 (s), 136.57 (s), 123.92 (s), 122.21 (s), 117.62 (s), 109.90 (s), 36.75 (s), 35.19 (s), 22.35 (s).
HRMS calcd for $C_{11}H_{10}N_2$ [Na^+]: 193.0742; found: 193.0739.

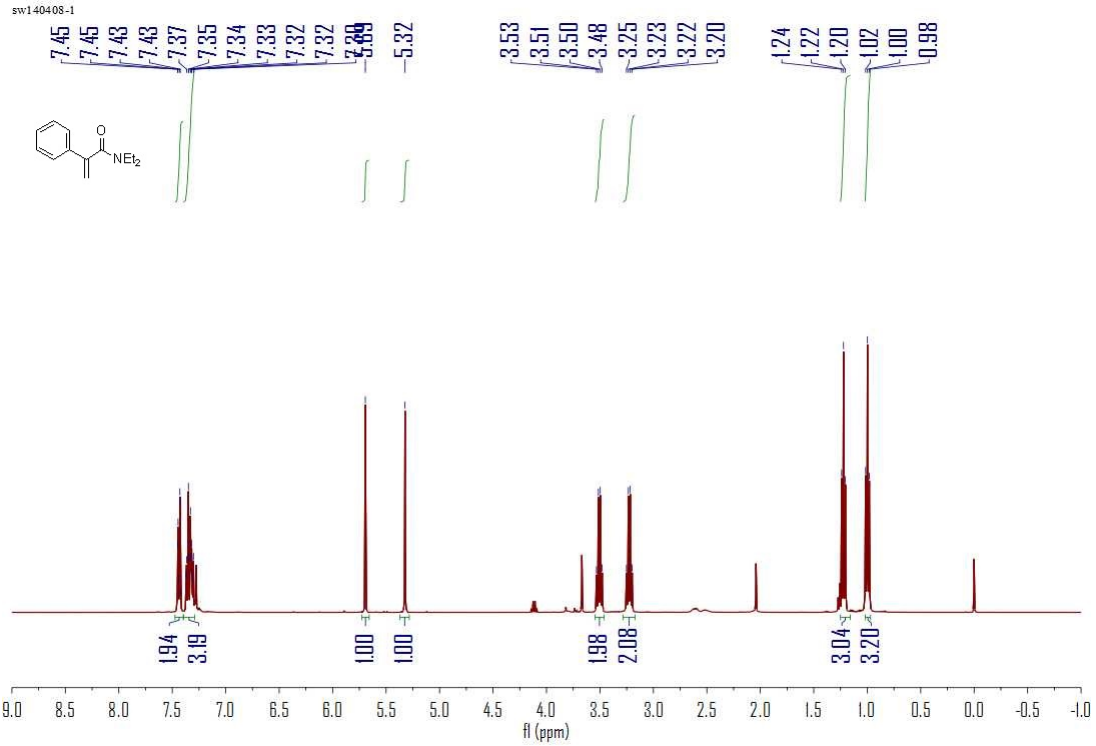
(3S,8R,9S,10R,13S,14S)-16-cyano-17-((E)-1-(methoxyimino)ethyl)-10,13-dimethyl-2,3,4,7,8,9,10,11,12,13,14,15-dodecahydro-1H-cyclopenta[a]phenanthren-3-yl acetate (5d):

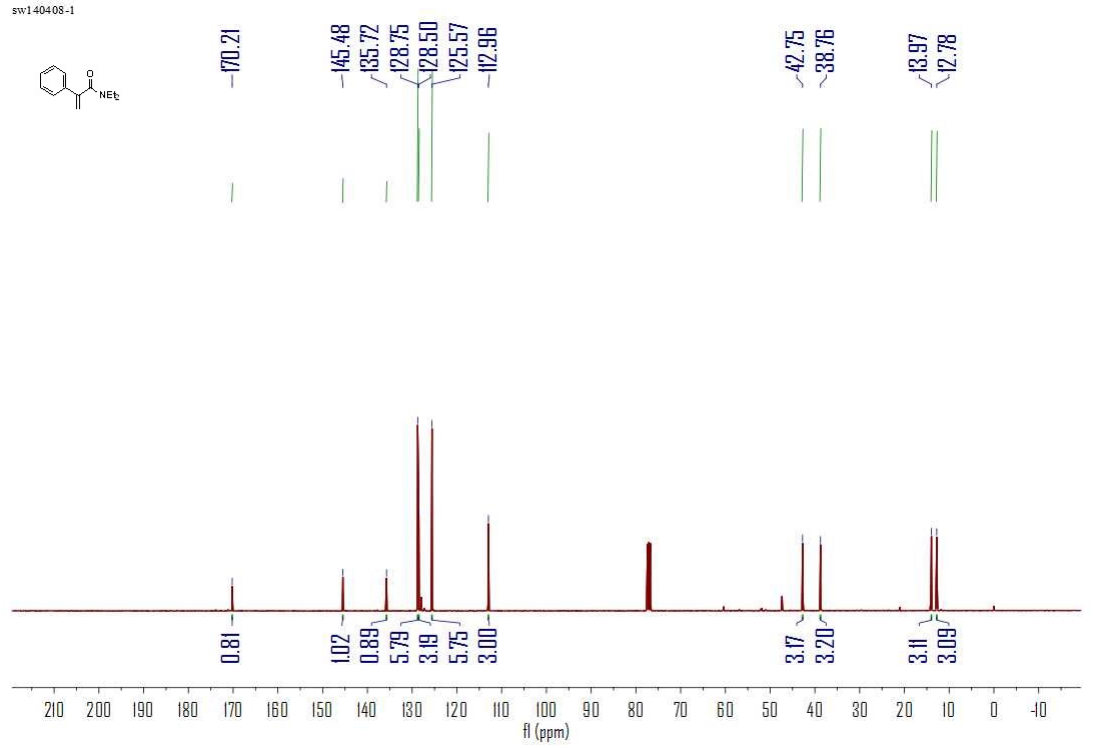


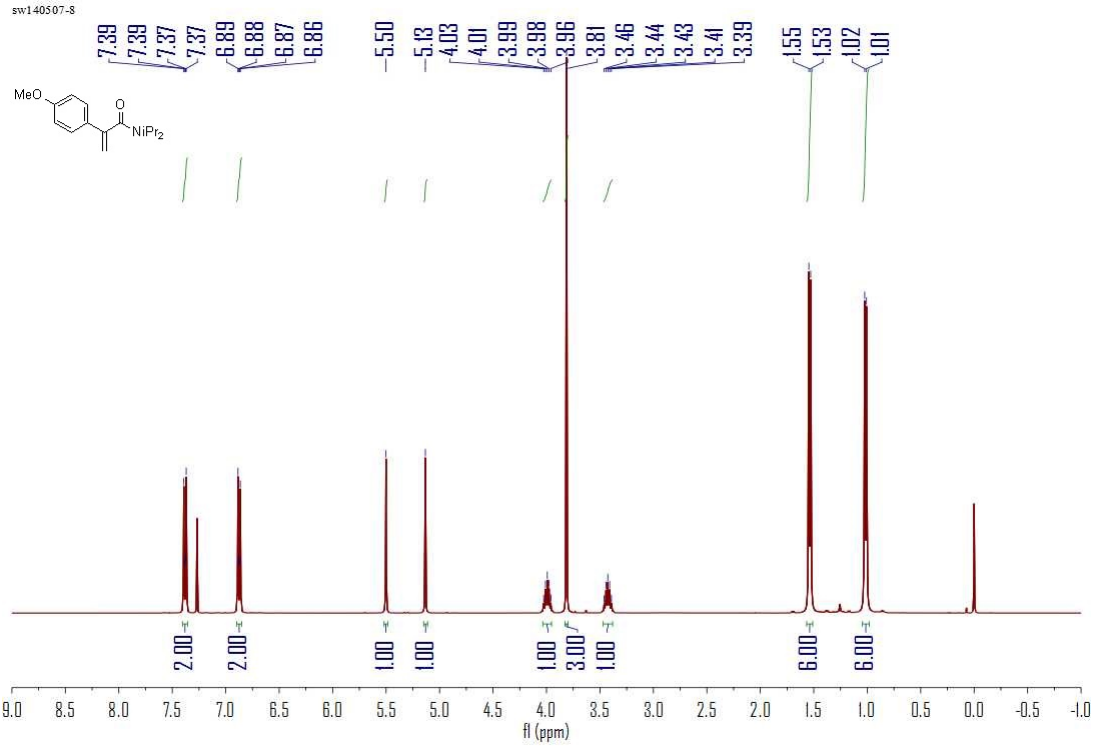
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.39 (d, *J* = 4.5 Hz, 1H), 4.60 (m, 1H), 3.96 (s, 3H), 2.49 (dd, *J* = 15.4, 6.6 Hz, 1H), 2.41 – 2.26 (m, 4H), 2.19 (s, 3H), 2.04 (s, 3H), 1.99 (s, 1H), 1.93 – 1.78 (m, 2H), 1.62 (m, 6H), 1.39 (m, 1H), 1.22 – 1.08 (m, 2H), 1.05 (s, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.51, 165.00 (2C), 150.80, 140.12, 121.76, 110.98, 73.71, 62.36, 55.67, 49.83, 49.49, 38.04, 36.81, 36.66, 35.68, 34.93, 31.22, 30.18, 27.68, 21.42, 20.67, 19.19, 15.98, 13.29.
HRMS calcd for $C_{25}H_{34}N_2O_3$[Na^+]: 433.2467; found: 433.2461.

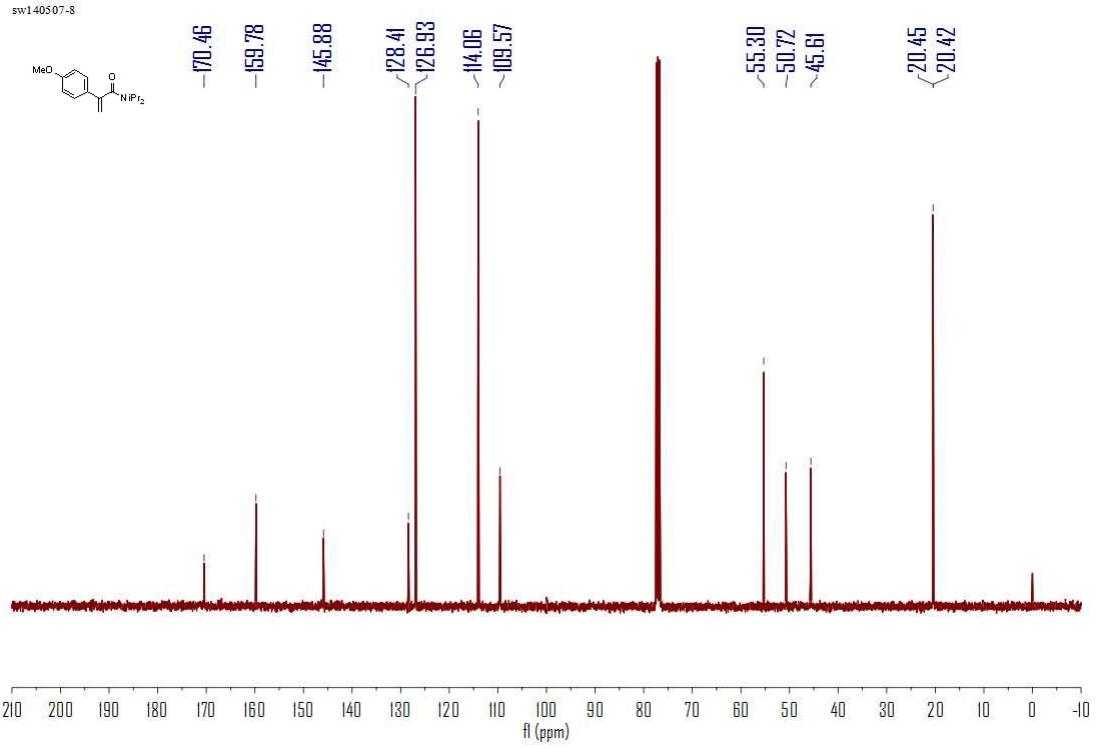

sw140410-5
7.46
7.46
7.45
7.44
7.44
7.44
7.37
7.37
7.36
7.35
7.35
7.33
7.33
7.32
7.32
7.31
7.31
7.30
7.30
7.29
7.28
7.28
5.62
5.25
4.02
4.00
3.98
3.97
3.95
3.44
3.43
3.41
1.55
1.53
1.01
1.00
NiPr2
1.96
3.00
1.00
0.99
1.01
0.99
6.06
6.01
f1 (ppm)

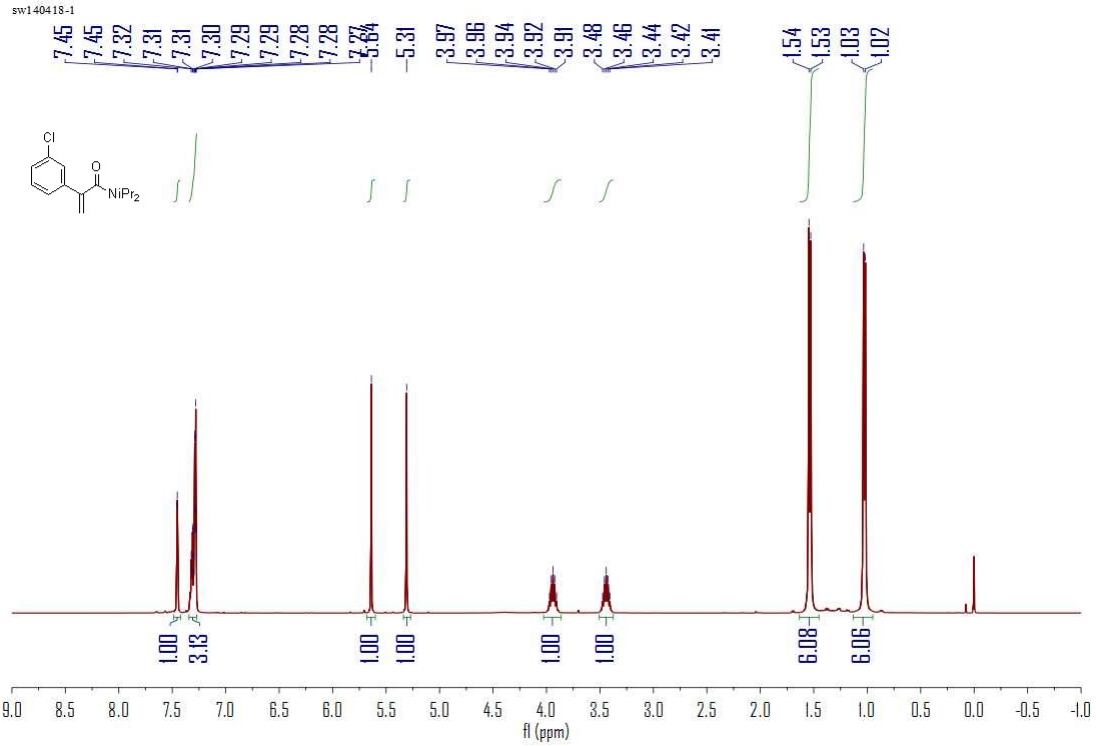

sw140410-5
NiPr2
170.05
146.73
135.87
128.69
128.39
125.62
111.51
50.65
45.56
20.44
20.38
f1 (ppm)

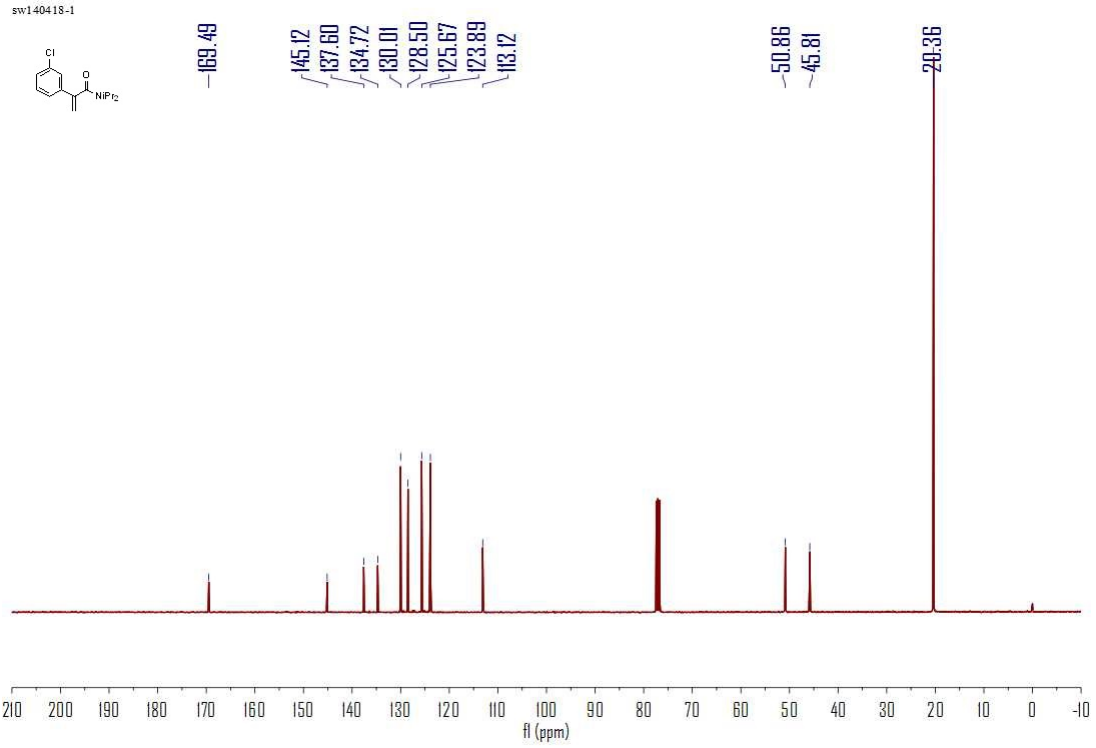

sw140408-1
7.45
7.45
7.43
7.43
7.37
7.35
7.34
7.33
7.32
7.32
5.32
3.53
3.51
3.50
3.48
3.25
3.23
3.22
3.20
1.24
1.22
1.20
1.02
1.00
0.98
NEt2
1.94
3.19
1.00
1.00
1.98
2.08
3.04
3.20
fl (ppm)

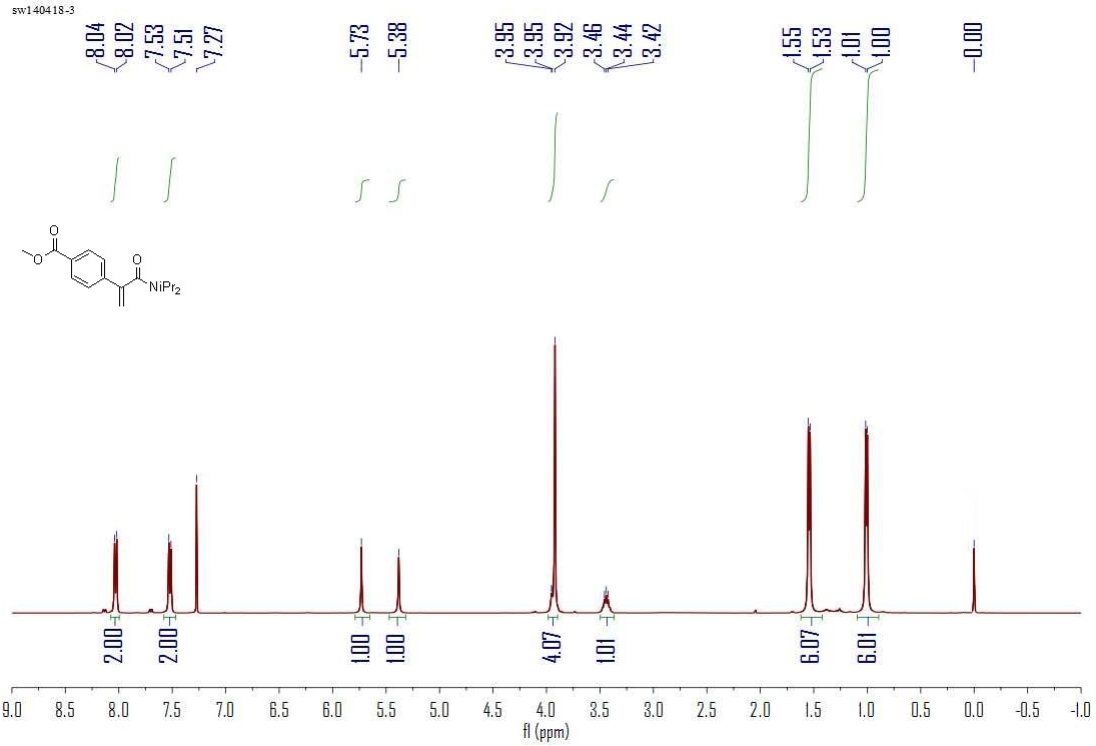

sw140408-1
170.21
145.48
135.72
128.75
128.50
125.57
112.96
42.75
38.76
13.97
12.78
NEt2
0.81
1.02
0.89
5.79
3.19
5.75
3.00
3.17
3.20
3.11
3.09
fl (ppm)

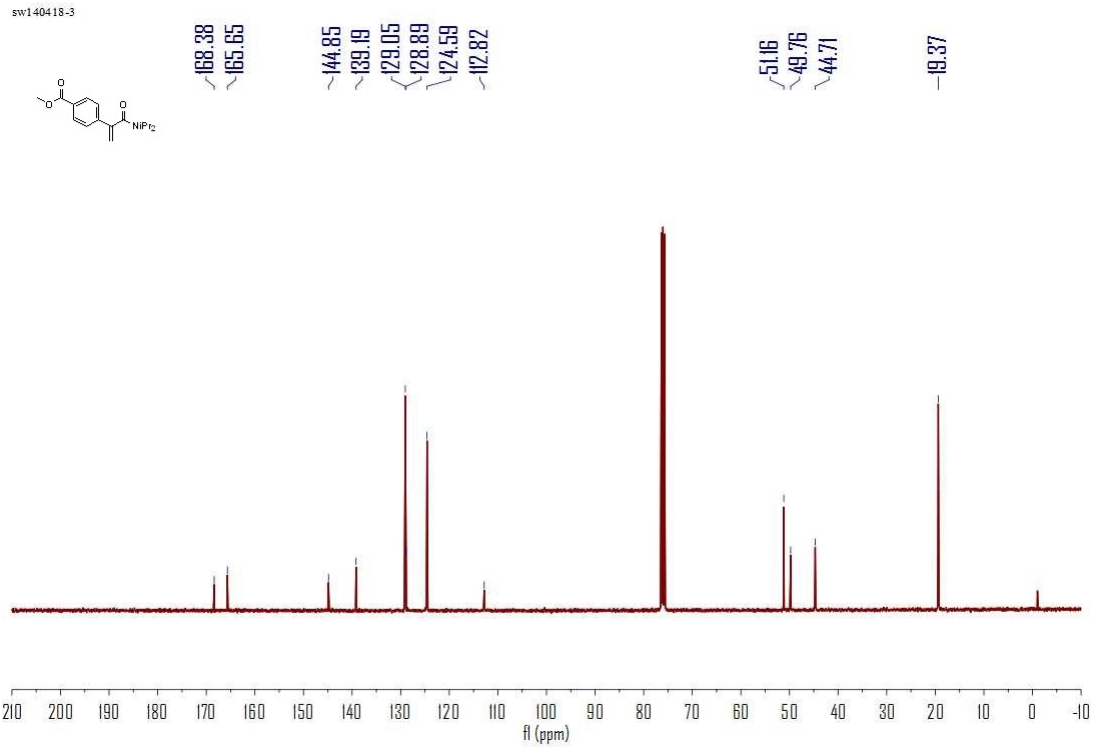

sw140507-8
7.39
7.39
7.37
7.37
6.89
6.88
6.87
6.86
5.50
5.13
4.03
4.01
3.99
3.98
3.96
3.81
3.46
3.44
3.43
3.41
3.39
1.55
1.53
1.02
1.01
MeO
O
NiPr2
2.00
2.00
1.00
1.00
1.00
3.00
1.00
6.00
6.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

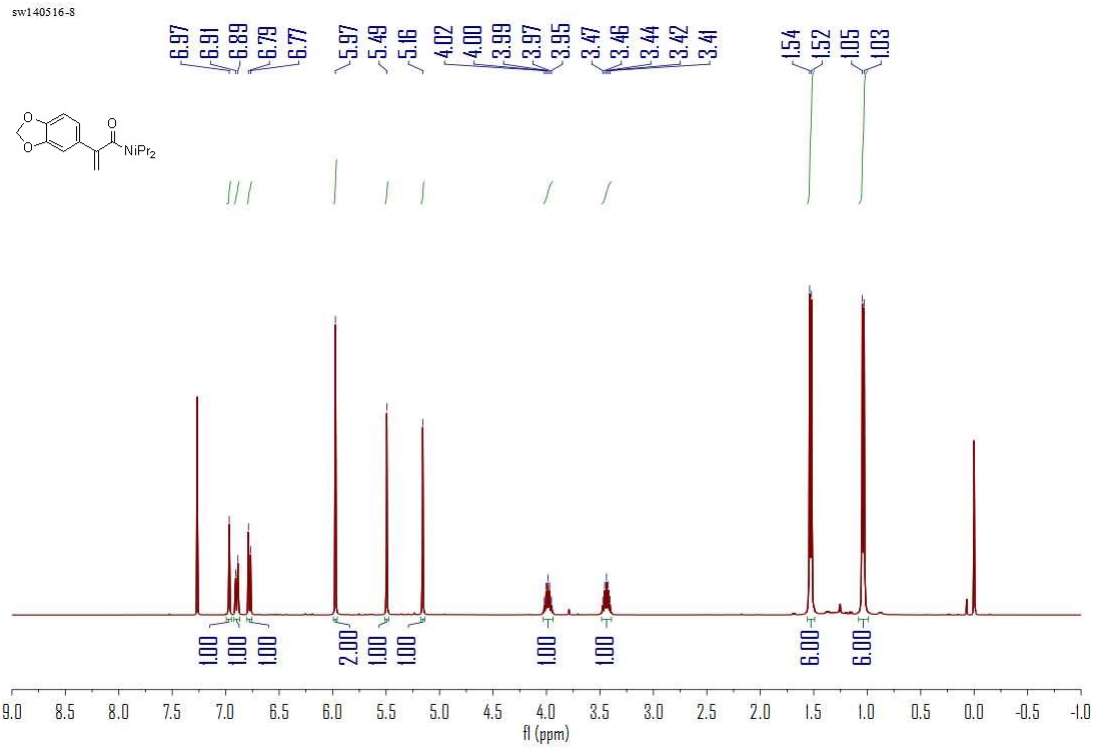

sw140507-8
170.46
159.78
145.88
128.41
126.93
114.06
109.57
55.30
50.72
45.61
20.45
20.42
MeO
O
NiPr2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

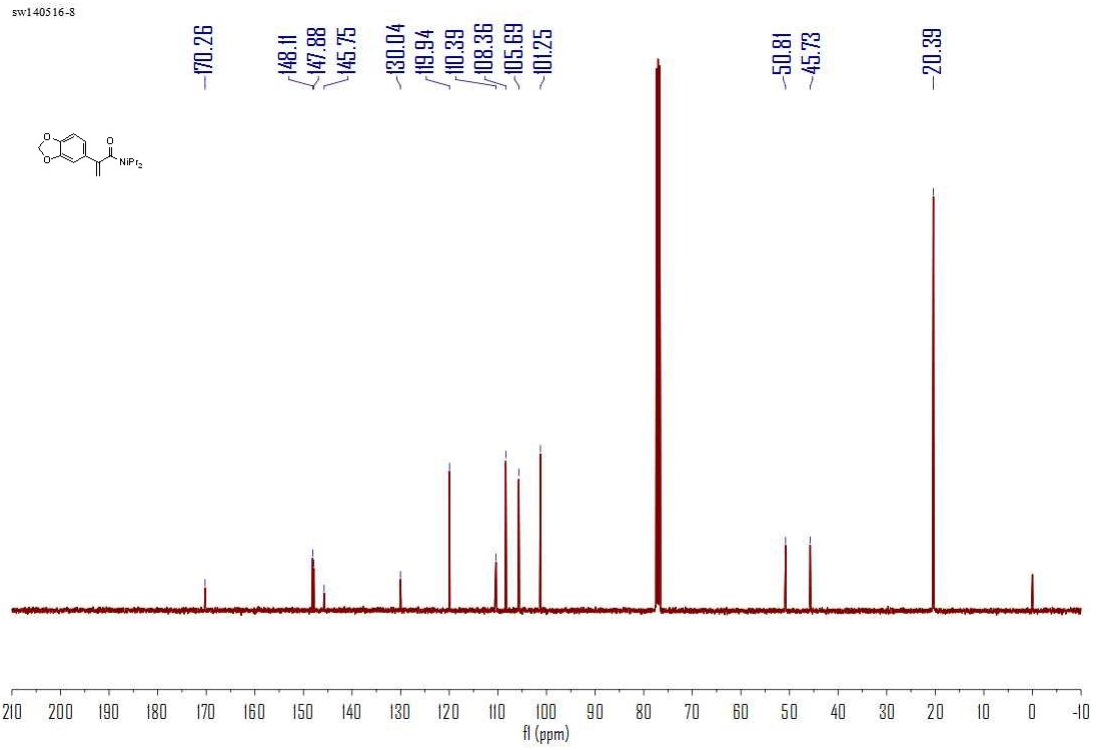

sw140418-1
7.45
7.45
7.32
7.31
7.31
7.30
7.29
7.29
7.28
7.28
7.27
5.64
5.31
3.97
3.96
3.94
3.92
3.91
3.48
3.46
3.44
3.42
3.41
1.54
1.53
1.03
1.02
Cl
O
NiPr2
1.00
3.13
1.00
1.00
1.00
1.00
6.08
6.06
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

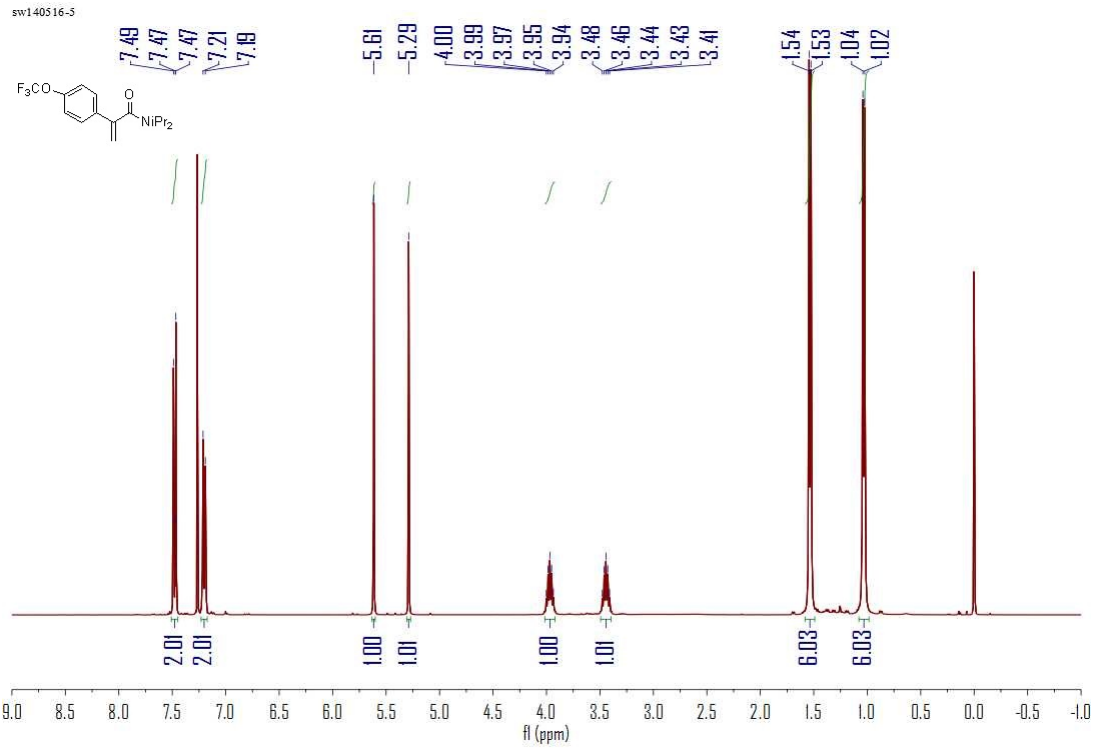

sw140418-1
Cl
O
NiPr2
169.49
145.12
137.60
134.72
130.01
128.50
125.67
123.89
113.12
50.86
45.81
28.36
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

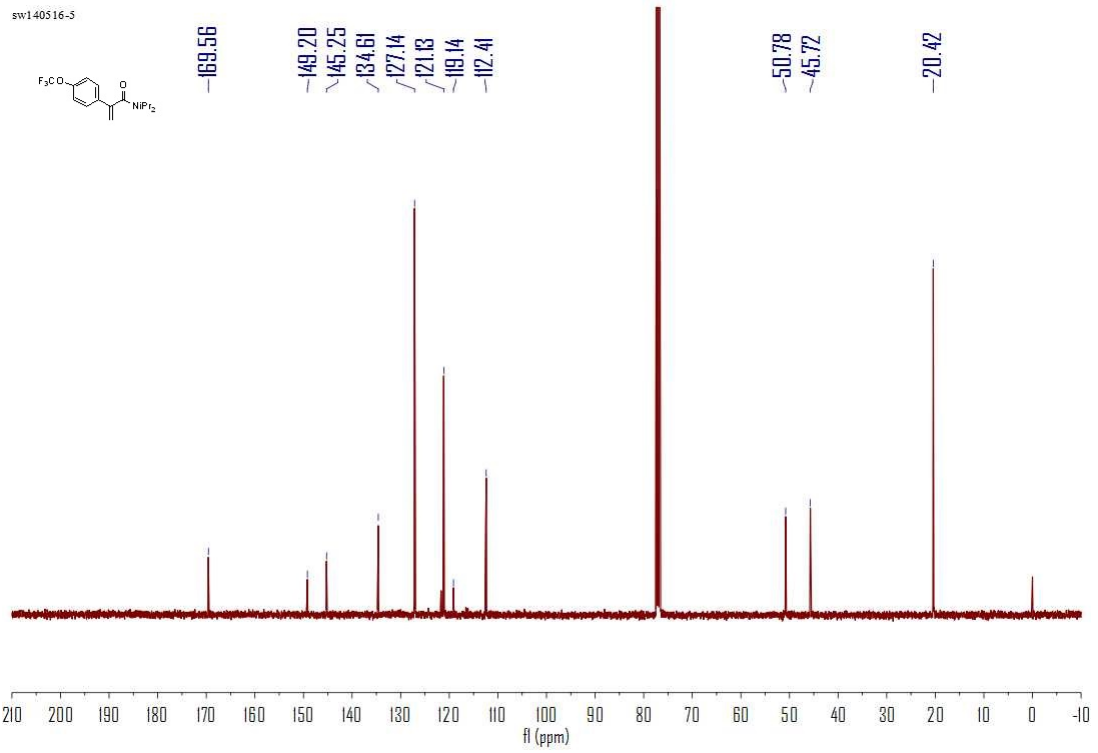

sw140418-3
8.04
8.02
7.53
7.51
7.27
5.73
5.38
3.95
3.95
3.92
3.46
3.44
3.42
1.55
1.53
1.01
1.00
0.00
NiPr2
2.00
2.00
1.00
1.00
4.07
1.01
6.07
6.01
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


sw140418-3
168.38
165.65
144.85
139.19
129.05
128.89
124.59
112.82
51.16
49.76
44.71
19.37
NiPr2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

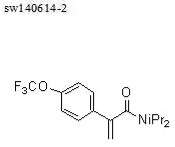

sw140516-8
6.97
6.91
6.89
6.79
6.77
5.97
5.49
5.16
4.02
4.00
3.99
3.97
3.95
3.47
3.46
3.44
3.42
3.41
1.54
1.52
1.05
1.03
NiPr2
1.00
1.00
1.00
2.00
1.00
1.00
1.00
1.00
6.00
6.00
f1 (ppm)


sw140516-8
170.26
148.11
147.88
145.75
130.04
119.94
110.39
108.36
105.69
101.25
50.81
45.73
20.39
NiPr2
f1 (ppm)

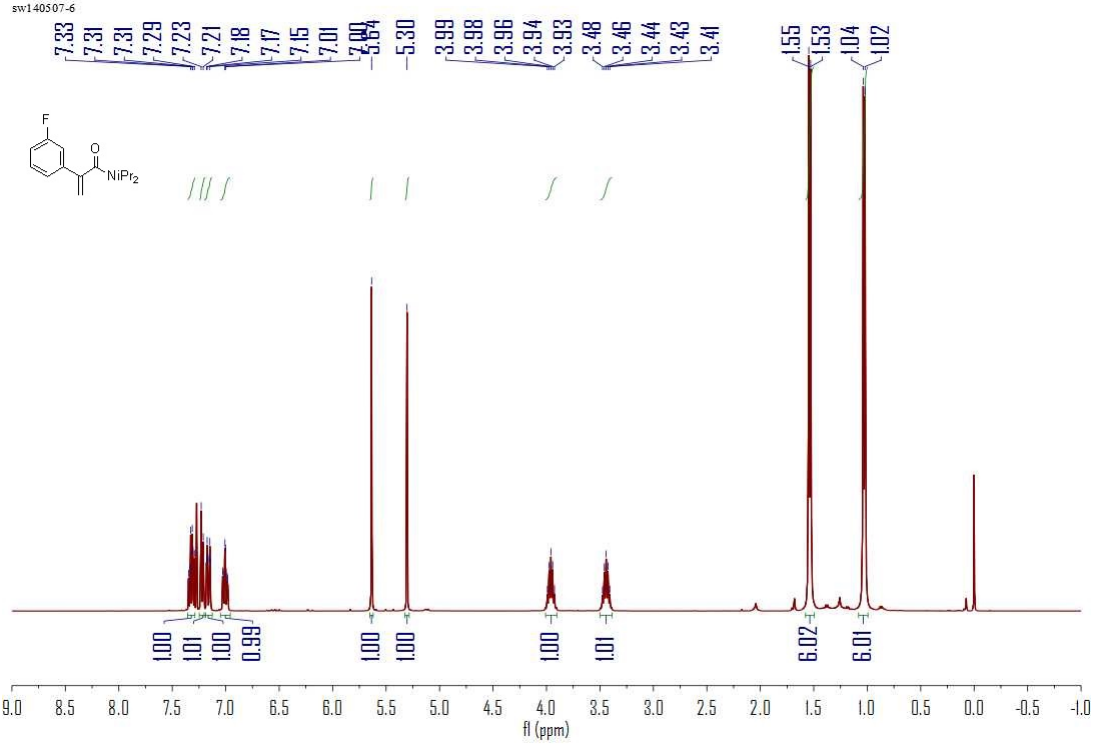

sw140516-5
7.49
7.47
7.47
7.21
7.19
5.61
5.29
4.00
3.99
3.97
3.95
3.94
3.48
3.46
3.44
3.43
3.41
1.54
1.53
1.04
1.02
F3CO
O
NiPr2
2.01
2.01
1.00
1.01
1.00
1.01
6.03
6.03
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

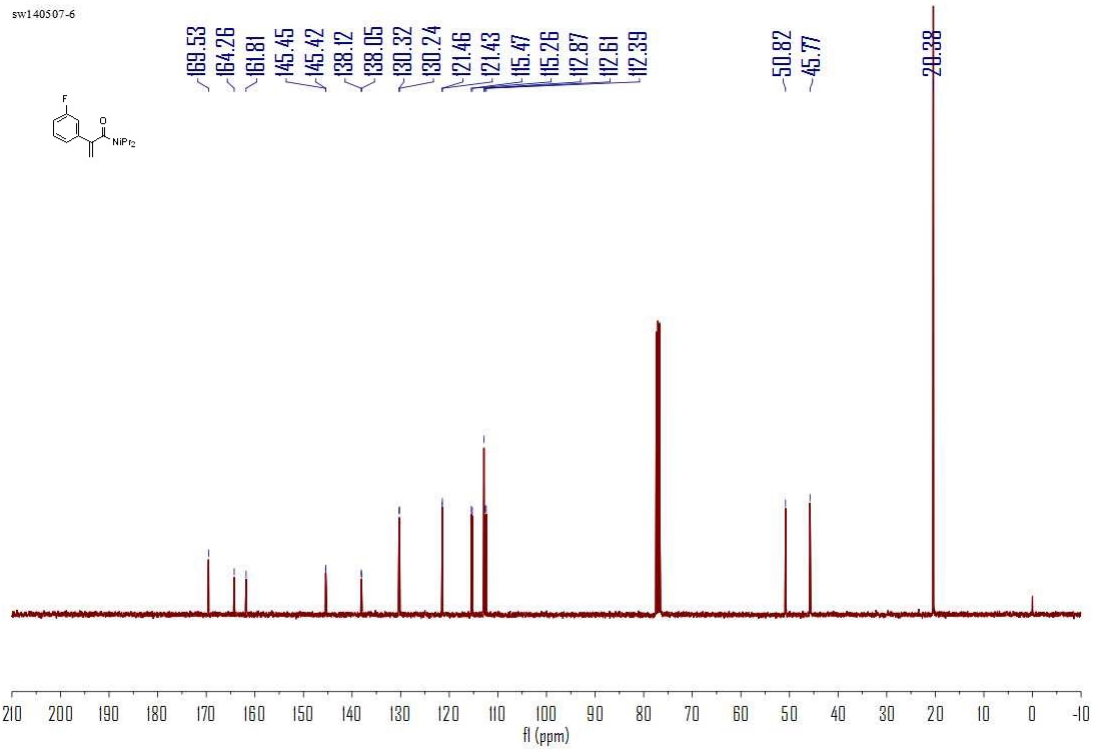

sw140516-5
169.56
149.20
145.25
134.61
127.14
121.83
119.14
112.41
50.78
45.72
20.42
F3CO
O
NiPr2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

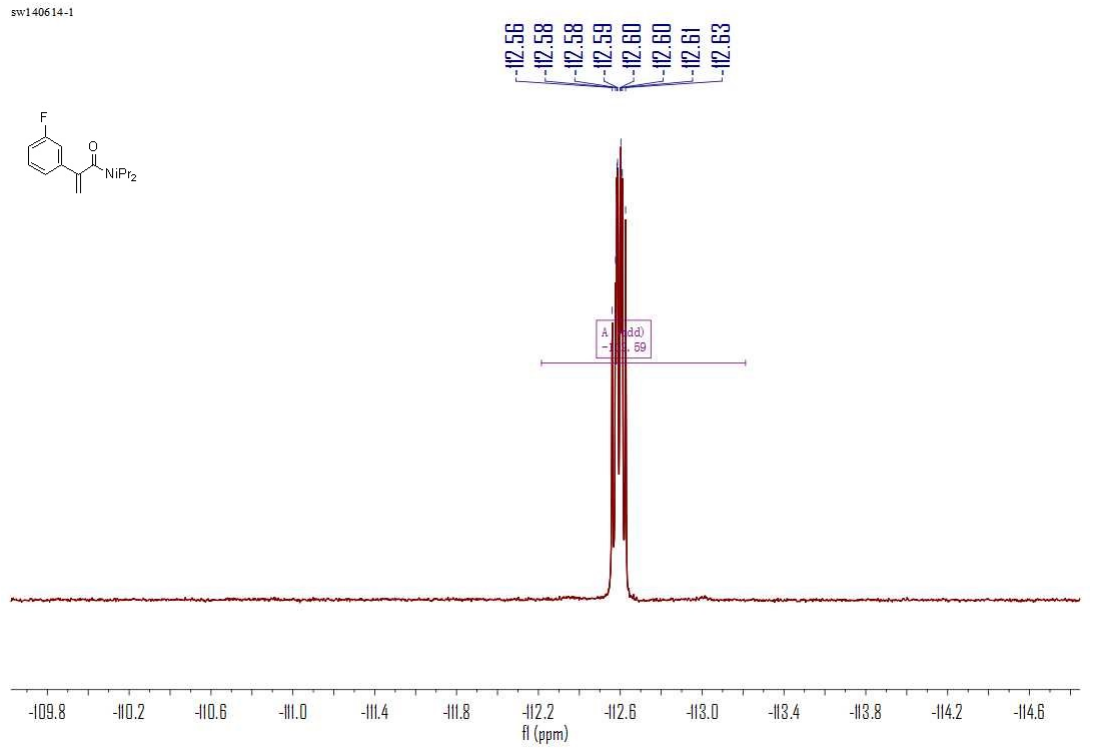
sw140614-2

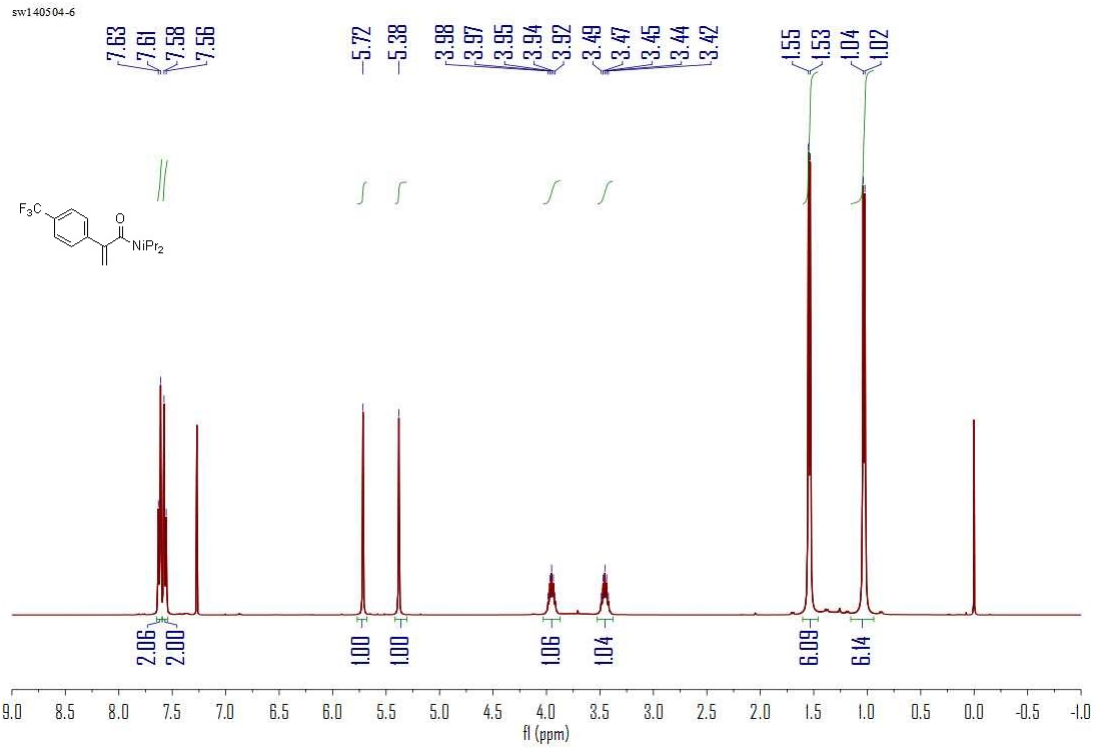


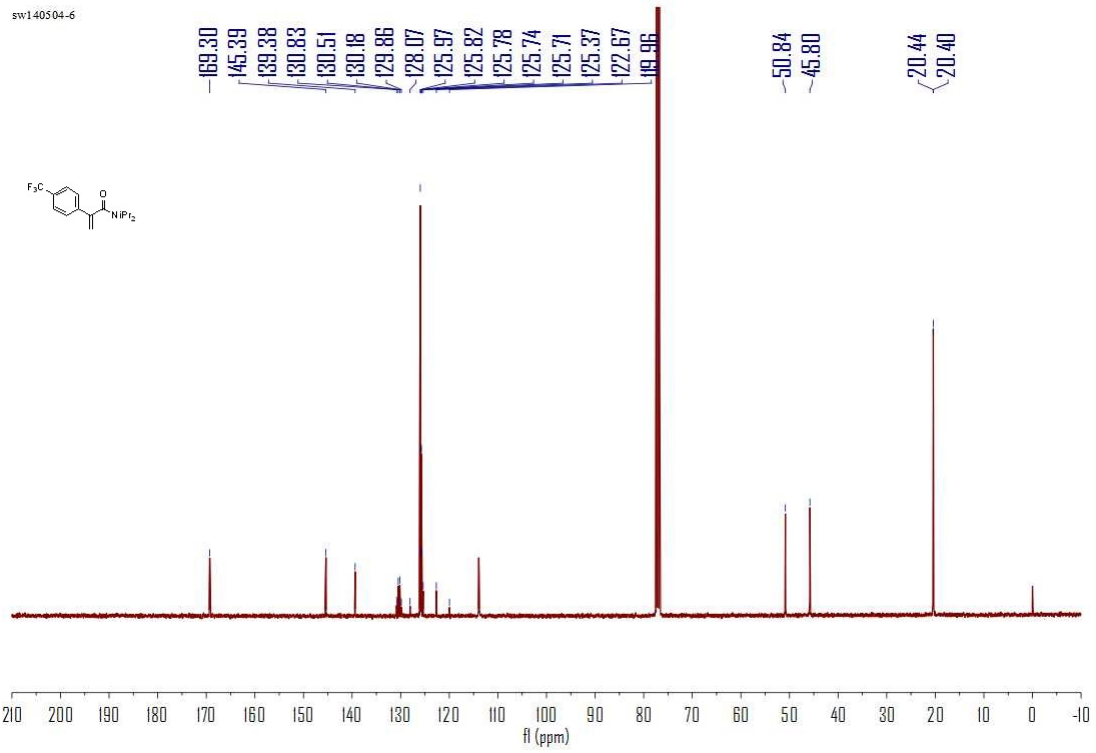
sw140507-6



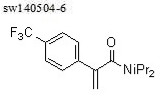

sw140507-6
169.53
164.26
161.81
145.45
145.42
138.12
138.05
130.32
130.24
121.46
121.43
115.47
115.26
112.87
112.61
112.39
50.82
45.77
20.38
F
O
NiPr2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


sw140614-1
112.56
112.58
112.58
112.59
112.60
112.60
112.61
112.63
F
O
NiPr2
-109.8 -110.2 -110.6 -111.0 -111.4 -111.8 -112.2 -112.6 -113.0 -113.4 -113.8 -114.2 -114.6
f1 (ppm)

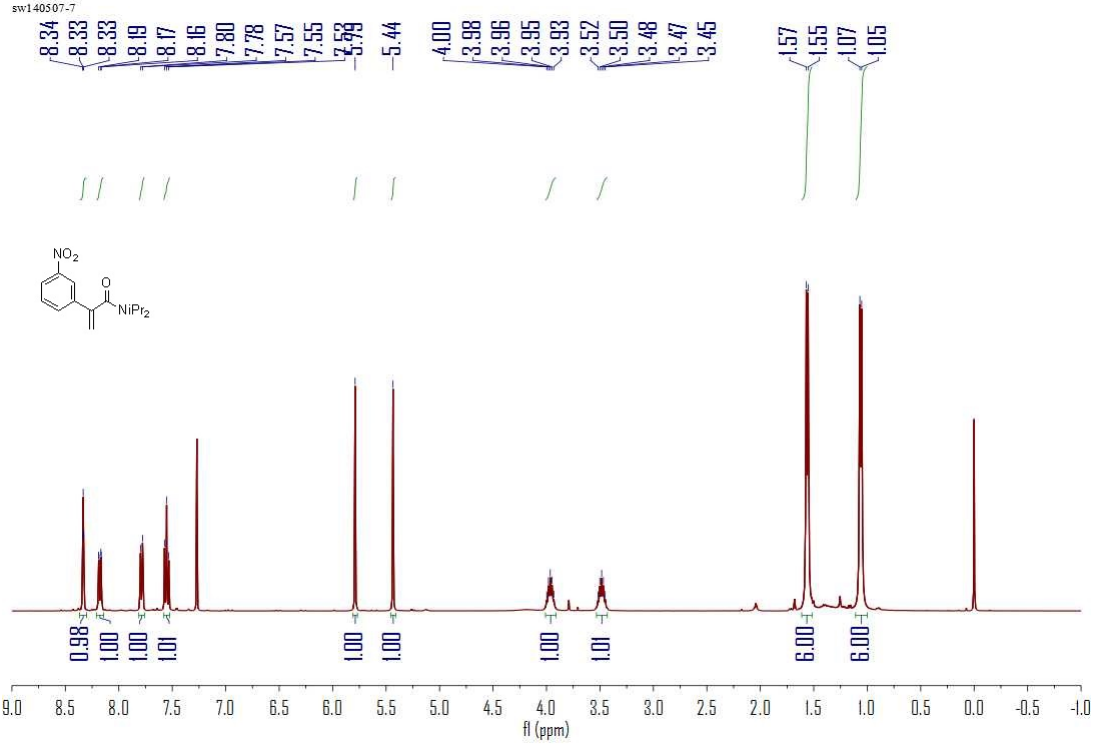

sw140504-6
7.63
7.61
7.58
7.56
5.72
5.38
3.98
3.97
3.95
3.94
3.92
3.49
3.47
3.45
3.44
3.42
1.55
1.53
1.04
1.02
F3C
O
NiPr2
2.06
2.00
1.00
1.00
1.06
1.04
6.09
6.14
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

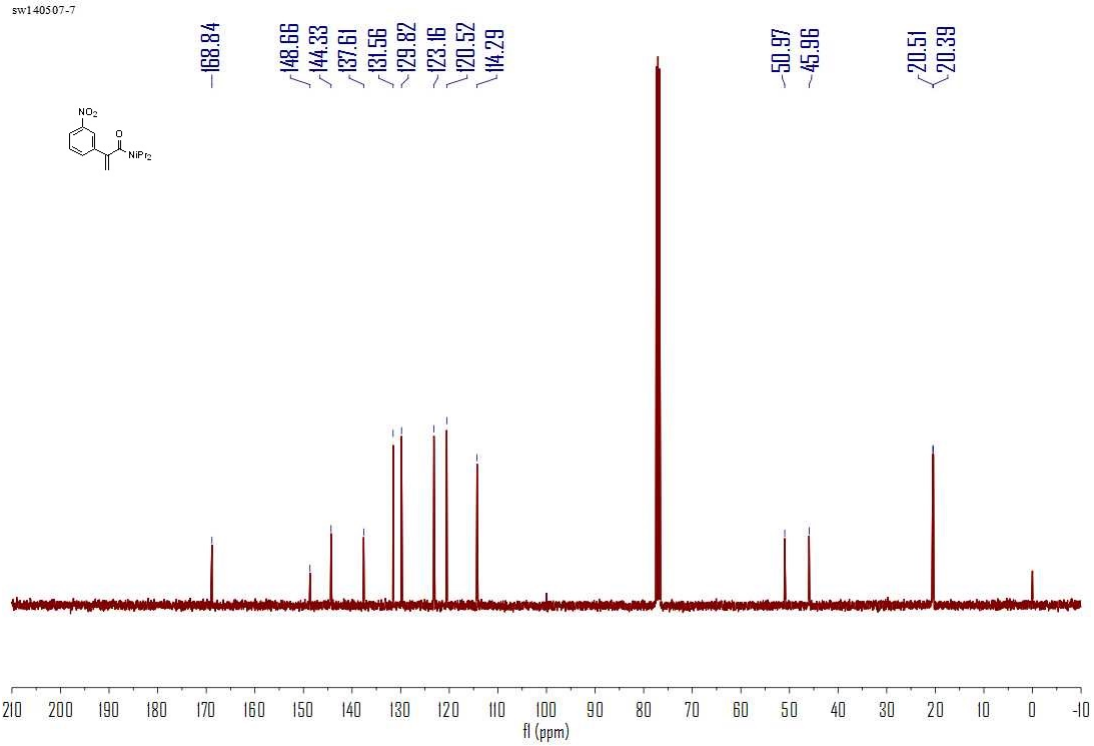

sw140504-6
169.30
145.39
139.38
130.83
130.51
130.18
129.86
128.07
125.97
125.82
125.78
125.74
125.71
125.37
122.67
118.96
50.84
45.80
20.44
20.40
F3C
O
NiPr2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

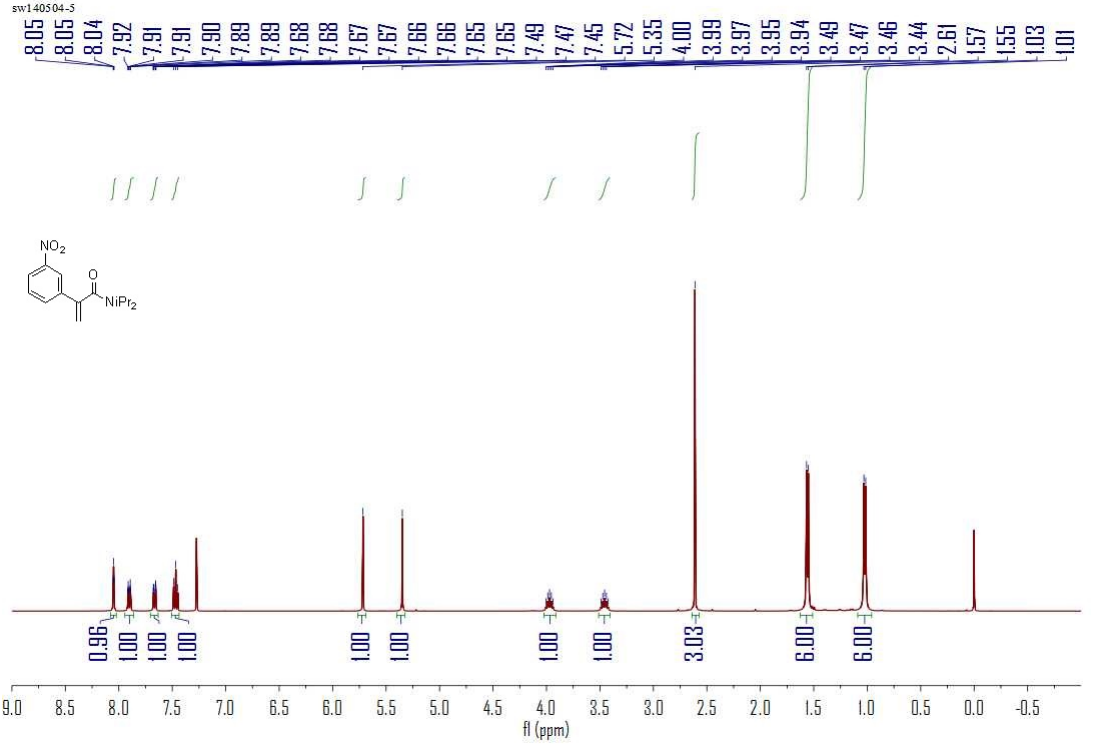
sw140504-6

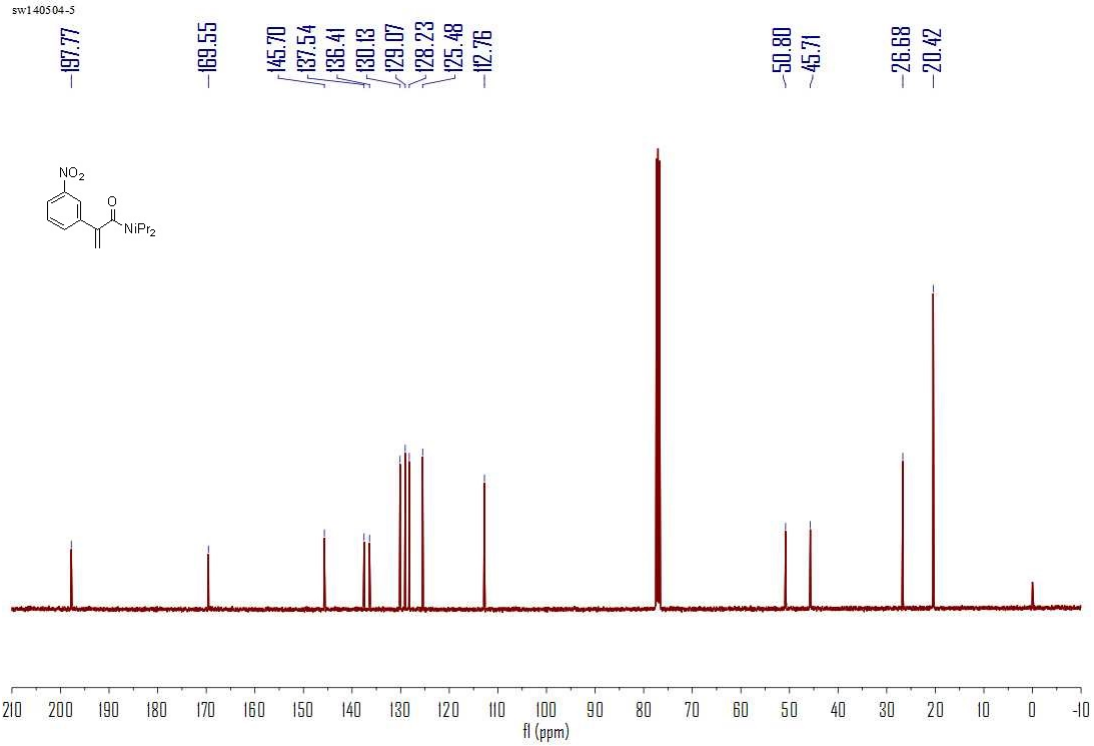


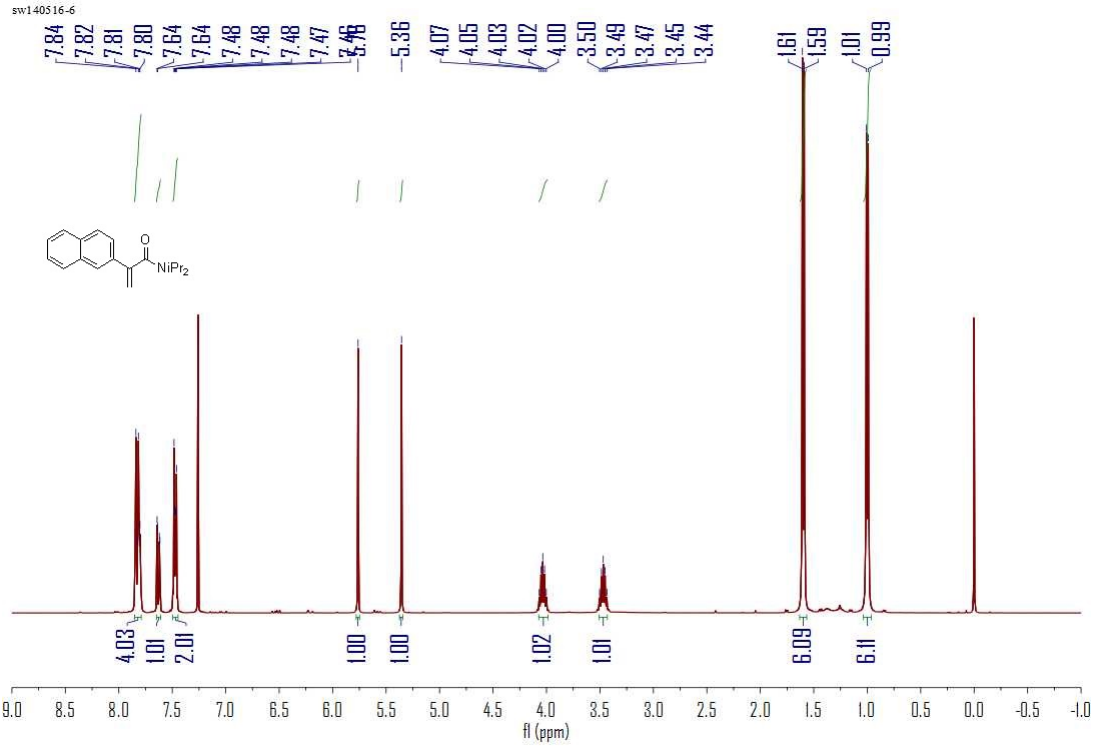
sw140507-7



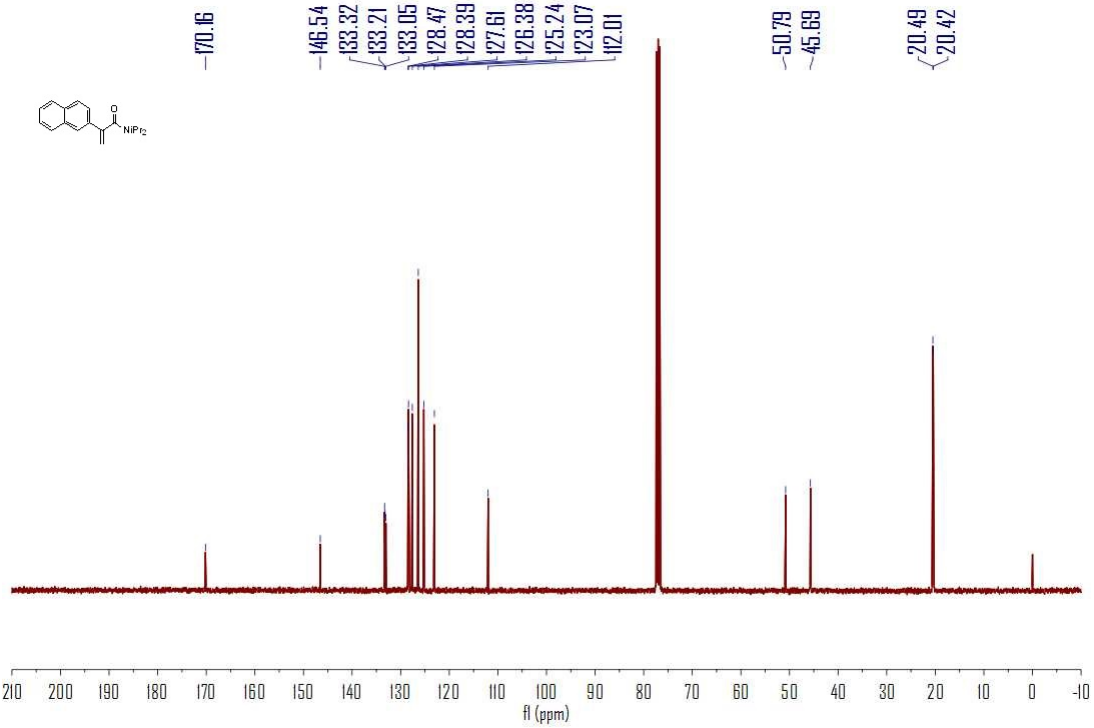

sw140507-7
168.84
148.66
144.33
137.61
131.56
129.82
123.16
120.52
114.29
50.97
45.96
20.51
20.39
NO2
O
NiPr2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


sw140504-5
8.05 8.05 8.04 7.92 7.91 7.91 7.90 7.89 7.89 7.68 7.68 7.67 7.67 7.66 7.66 7.65 7.65 7.49 7.47 7.45 5.72 5.35 4.00 3.99 3.97 3.95 3.94 3.49 3.47 3.46 3.44 2.61 1.57 1.55 1.03 1.01
NO2
O
NiPr2
0.96 1.00 1.00 1.00 1.00 1.00 1.00 1.00 3.03 6.00 6.00
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)

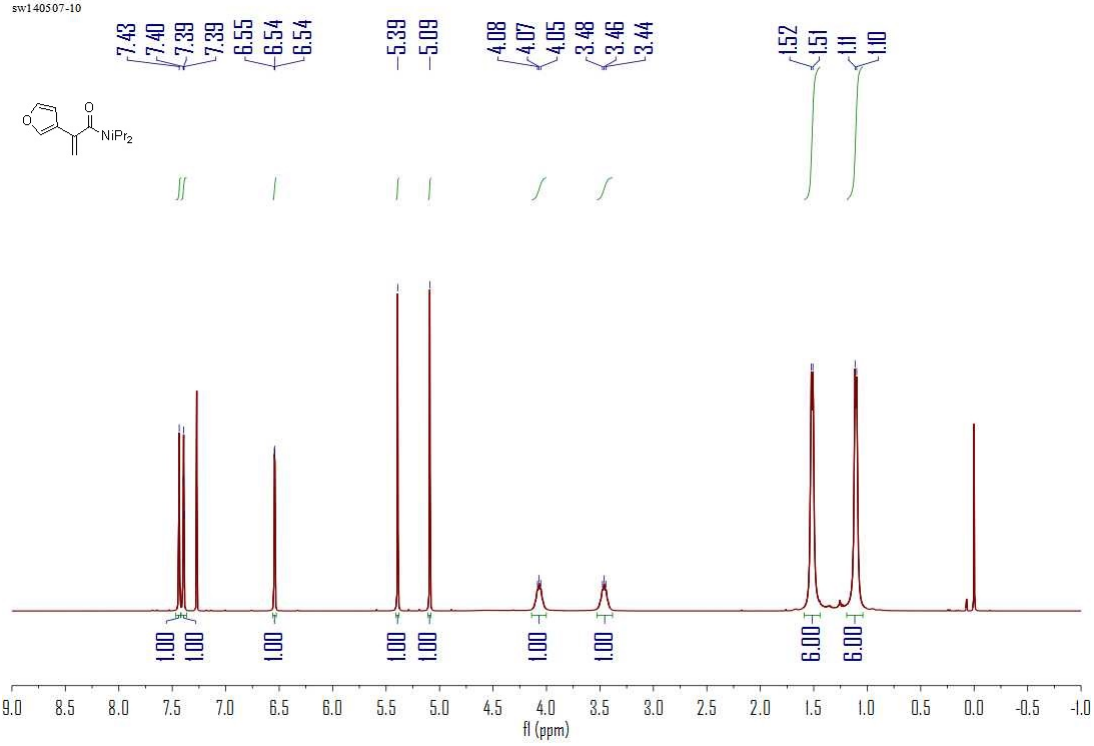

sw140504-5
197.77
169.55
145.70
137.54
136.41
130.13
129.07
128.23
125.48
112.76
50.80
45.71
26.68
20.42
NO2
O
NiPr2
f1 (ppm)

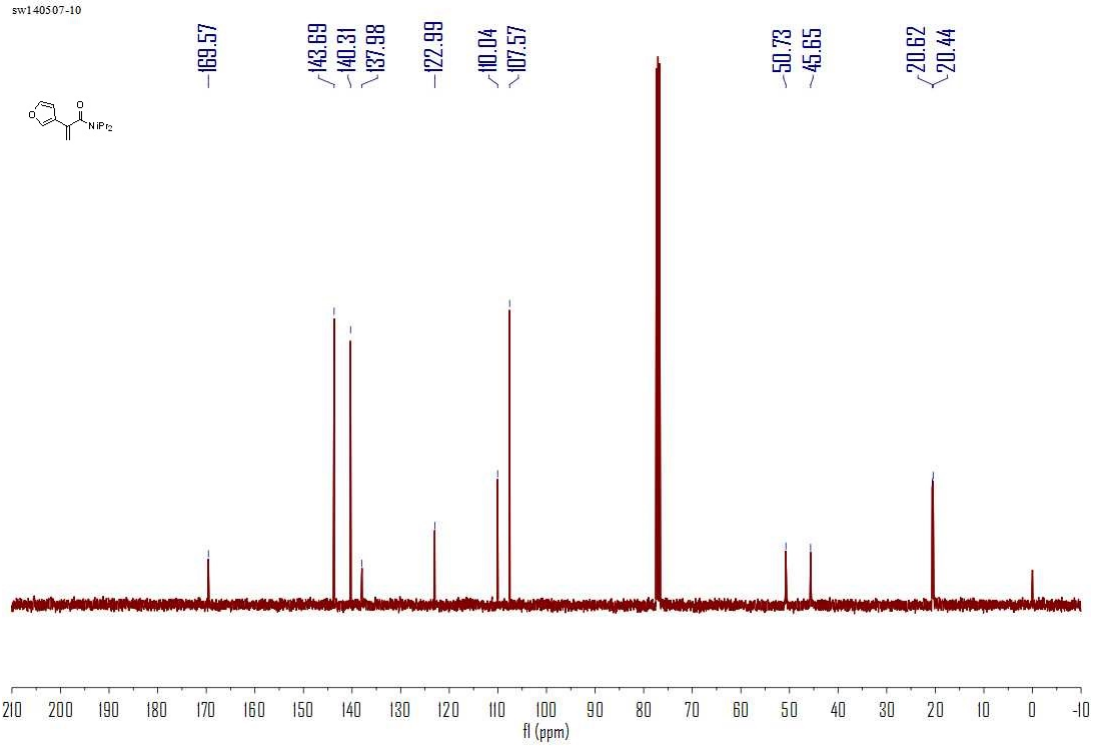

sw140516-6
7.84
7.82
7.81
7.80
7.64
7.64
7.48
7.48
7.48
7.47
5.36
4.07
4.05
4.03
4.02
4.00
3.50
3.49
3.47
3.45
3.44
1.61
1.59
1.01
0.99
O
NiPr2
4.03
1.01
2.01
1.00
1.00
1.02
1.01
6.09
6.11
f1 (ppm)

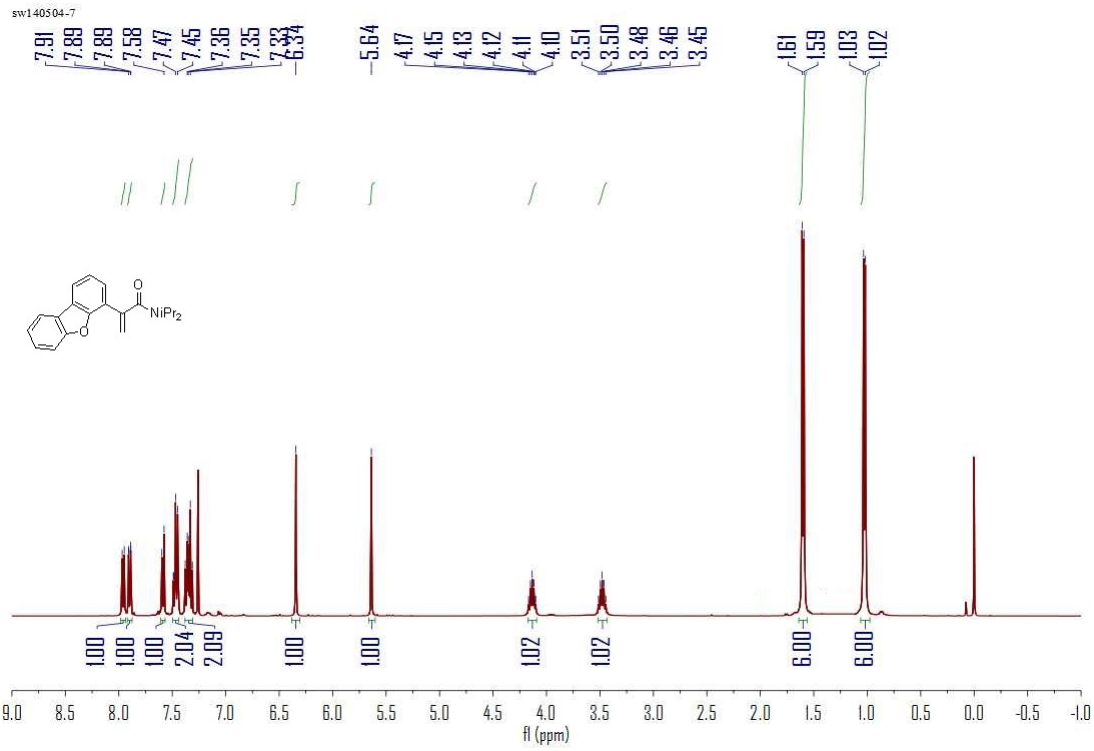
sw140516-6

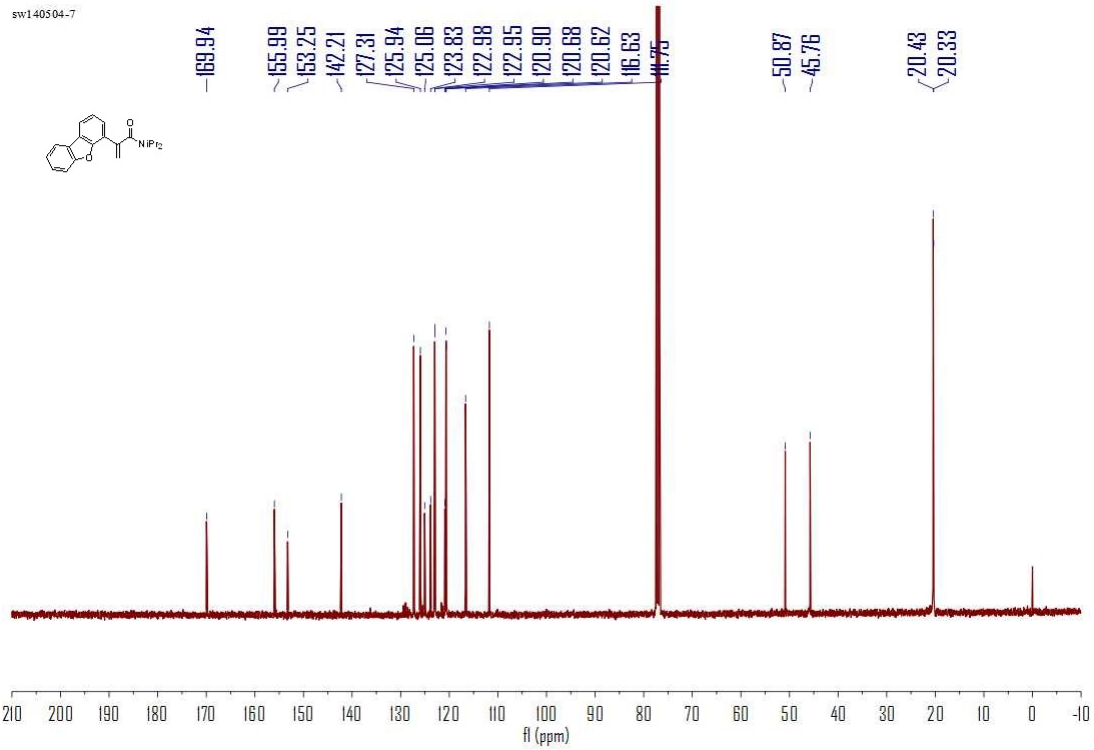


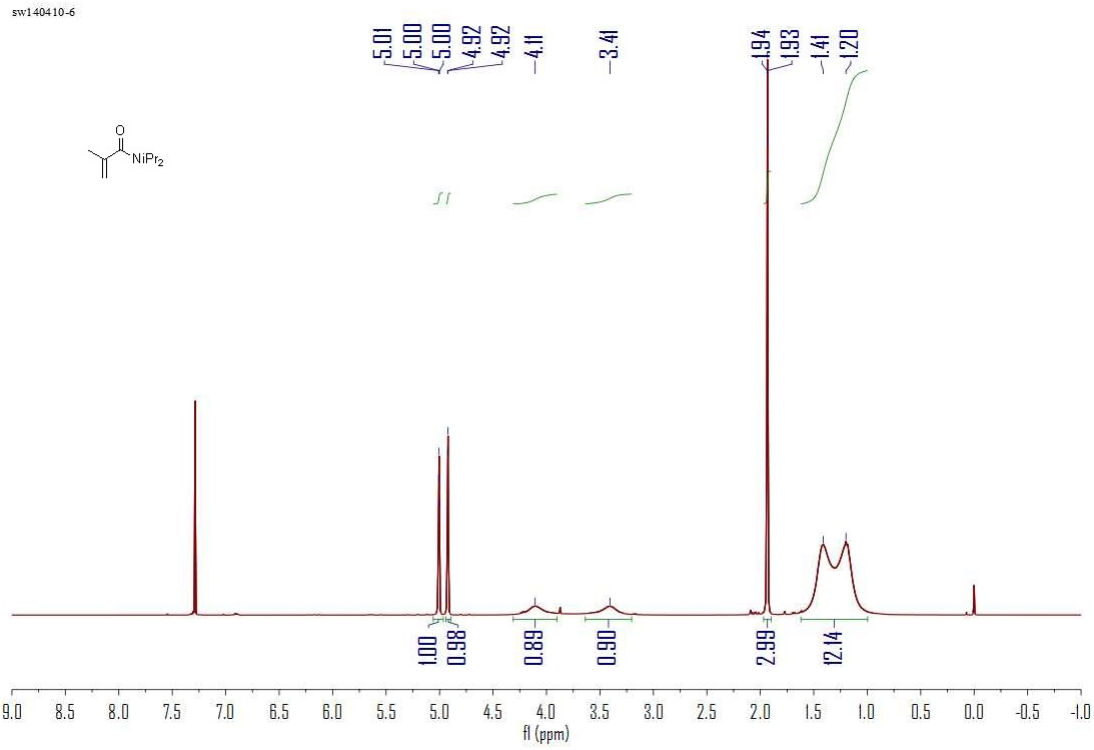
sw140507-10

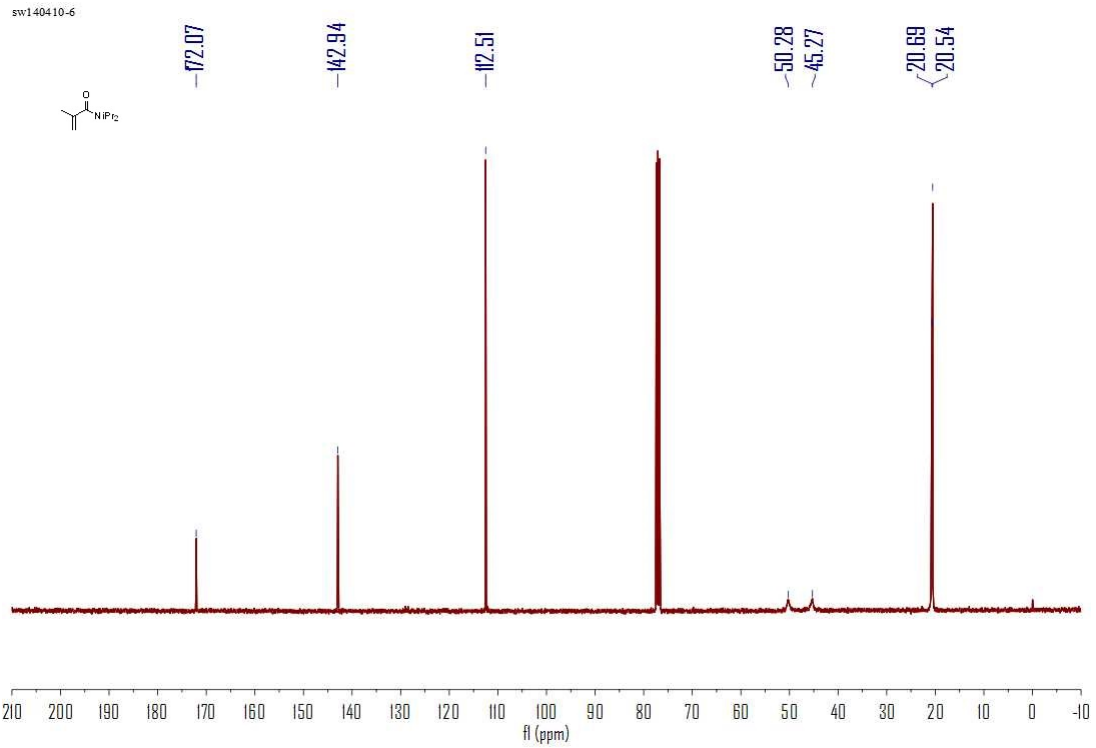


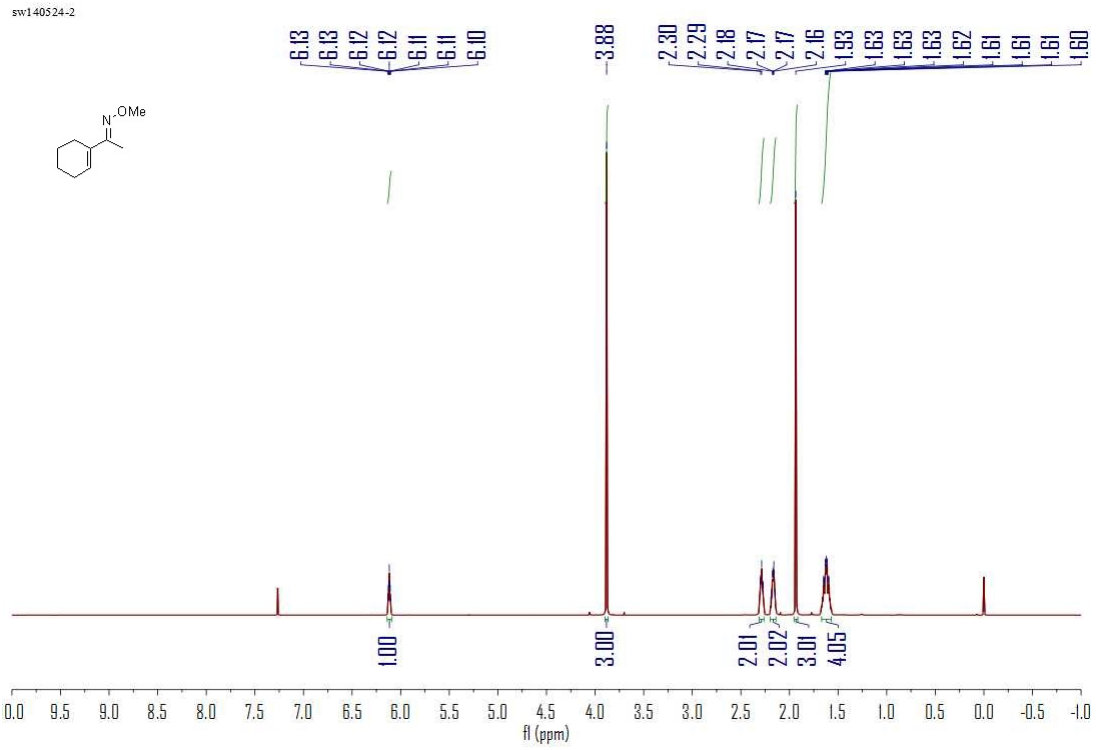

sw140507-10
169.57
143.69
140.31
137.98
122.99
110.04
107.57
50.73
45.65
20.62
20.44
NiPr2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

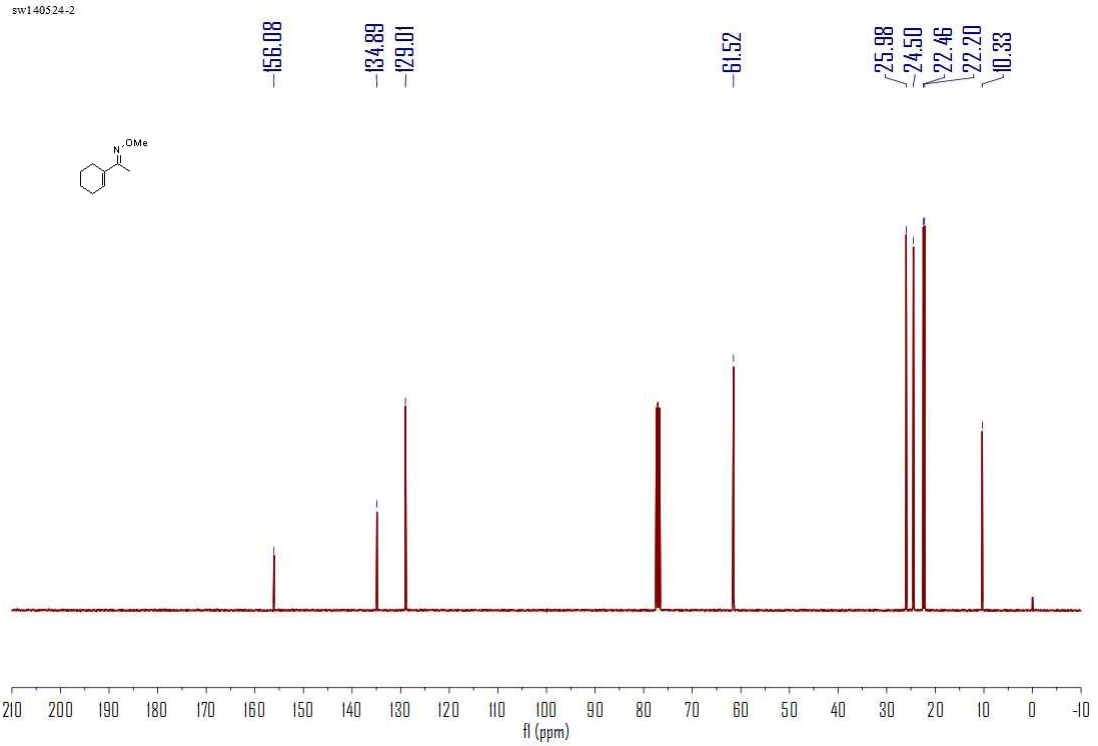

sw140504-7
7.91
7.89
7.89
7.58
7.47
7.45
7.36
7.35
7.33
6.34
5.64
4.17
4.15
4.13
4.12
4.11
4.10
3.51
3.50
3.48
3.46
3.45
1.61
1.59
1.03
1.02
NiPr2
1.00 1.00 1.00 2.04 2.09 1.00 1.00 1.02 1.02 6.00 6.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

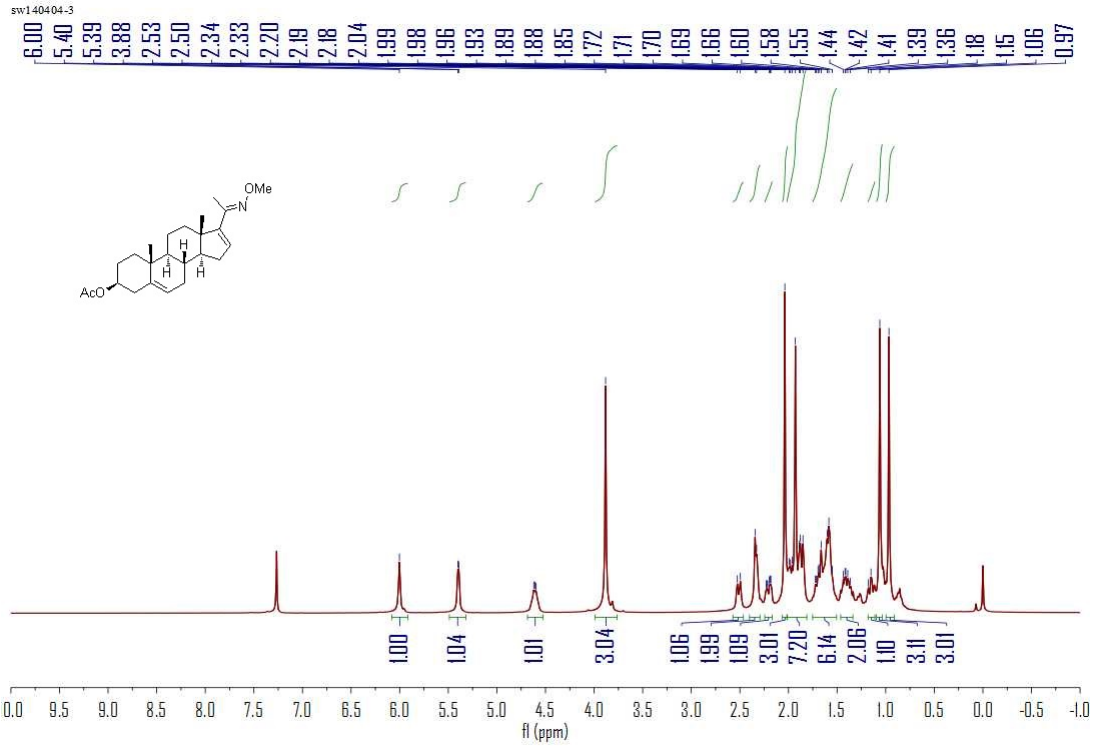

sw140504-7
169.94
155.99
153.25
142.21
127.31
125.94
125.06
123.83
122.98
122.95
120.90
120.68
120.62
116.63
50.87
45.76
20.43
20.33
O
NiPr2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

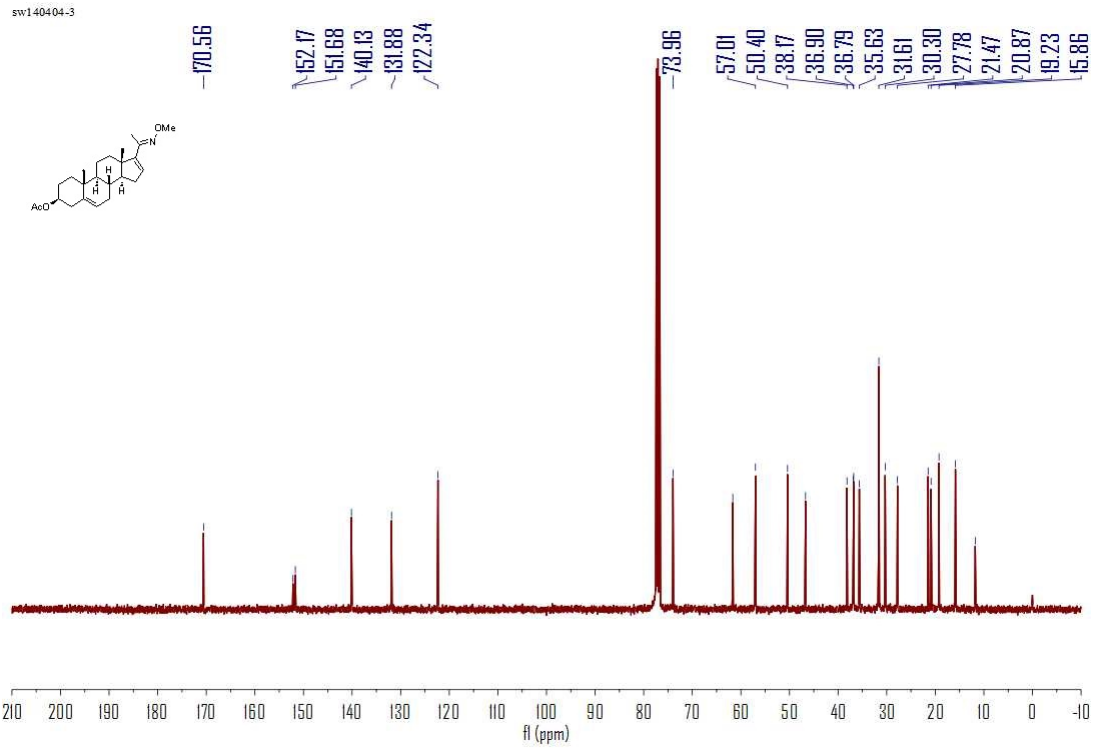

sw140410-6
5.01
5.00
5.00
4.92
4.92
4.11
3.41
1.94
1.93
1.41
1.20
O
NiPr2
1.00
0.98
0.89
0.90
2.99
12.14
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

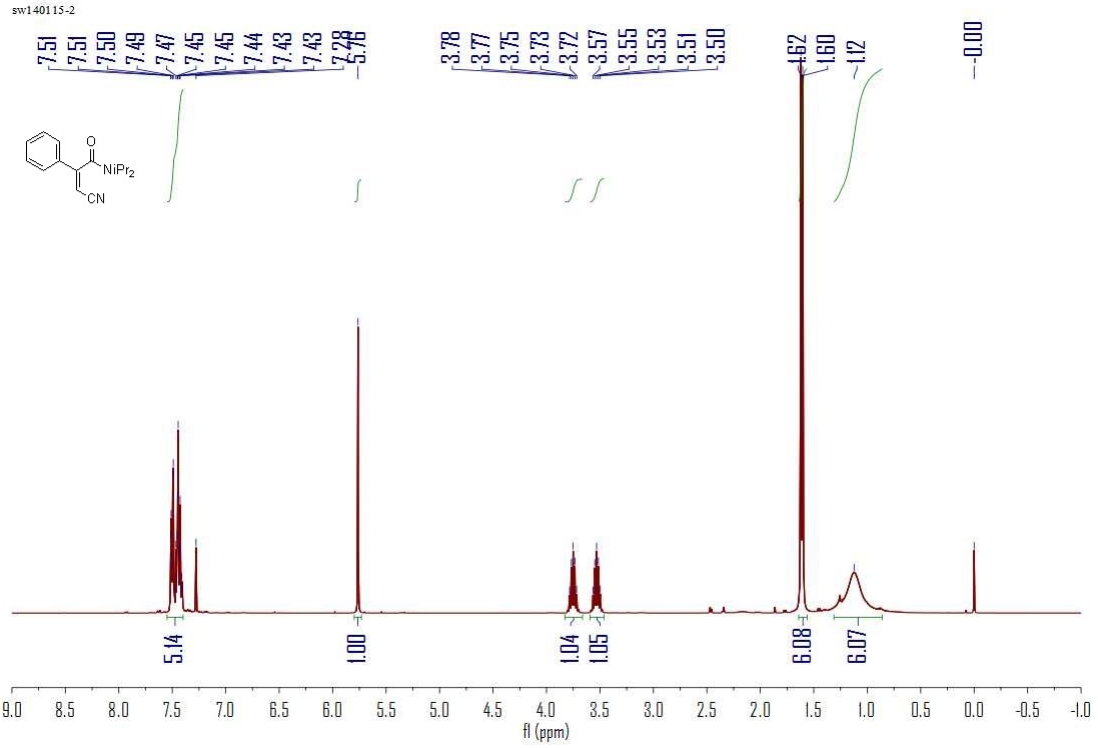

sw140410-6
172.07
142.94
112.51
50.28
45.27
20.69
20.54
f1 (ppm)


sw140524-2
6.13
6.13
6.12
6.12
6.11
6.11
6.10
3.88
2.30
2.29
2.18
2.17
2.17
2.16
1.93
1.63
1.63
1.63
1.62
1.61
1.61
1.61
1.60
1.00
3.00
2.01
2.02
3.01
4.05
f1 (ppm)

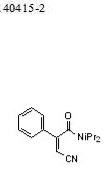

sw140524-2
156.08
134.89
129.01
61.52
25.98
24.50
22.46
22.20
10.33
N
OMe
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


sw140404-3
6.00
5.40
5.39
3.88
2.53
2.50
2.34
2.33
2.20
2.19
2.18
2.04
1.99
1.98
1.96
1.93
1.89
1.88
1.85
1.72
1.71
1.70
1.69
1.66
1.60
1.58
1.55
1.44
1.42
1.41
1.39
1.36
1.18
1.15
1.06
0.97
OMe
N
H
H
H
AcO
1.00
1.04
1.01
3.04
1.06
1.99
1.09
3.01
7.20
6.14
2.06
1.10
3.11
3.01
0.0 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 -1.0
f1 (ppm)

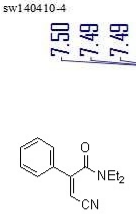

sw140404-3
170.56
152.17
151.68
140.13
131.88
122.34
73.96
57.01
50.40
38.17
36.90
36.79
35.63
31.61
30.30
27.78
21.47
20.87
19.23
15.86
OMe
AcO
f1 (ppm)


sw140115-2
7.51
7.51
7.50
7.49
7.47
7.45
7.45
7.44
7.43
7.43
7.28
5.76
3.78
3.77
3.75
3.73
3.72
3.57
3.55
3.53
3.51
3.50
1.62
1.60
1.12
0.00
NiPr2
CN
5.14
1.00
1.04
1.05
6.08
6.07
f1 (ppm)

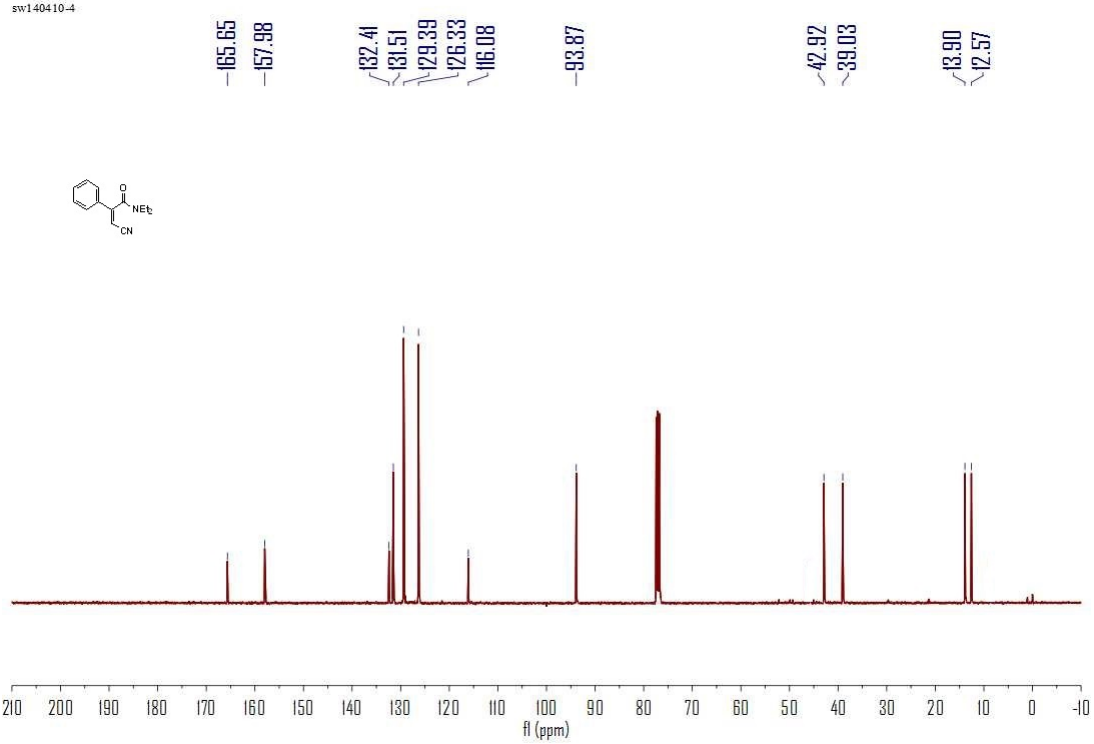
sw140415-2



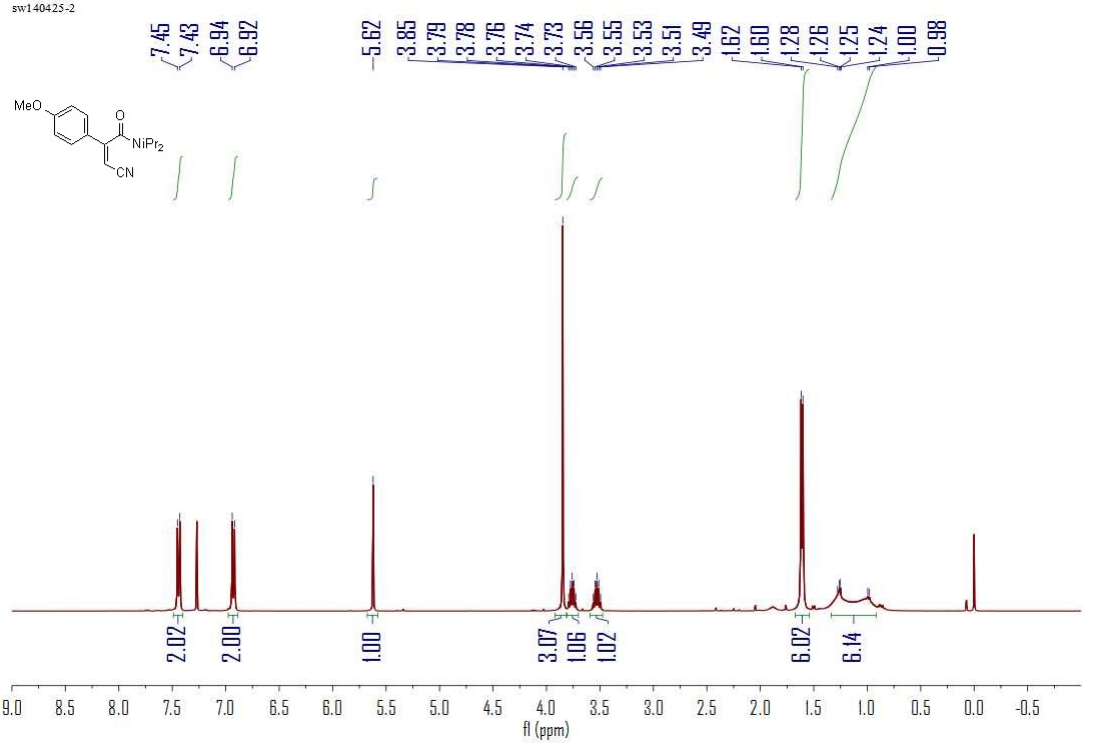
sw140410-4

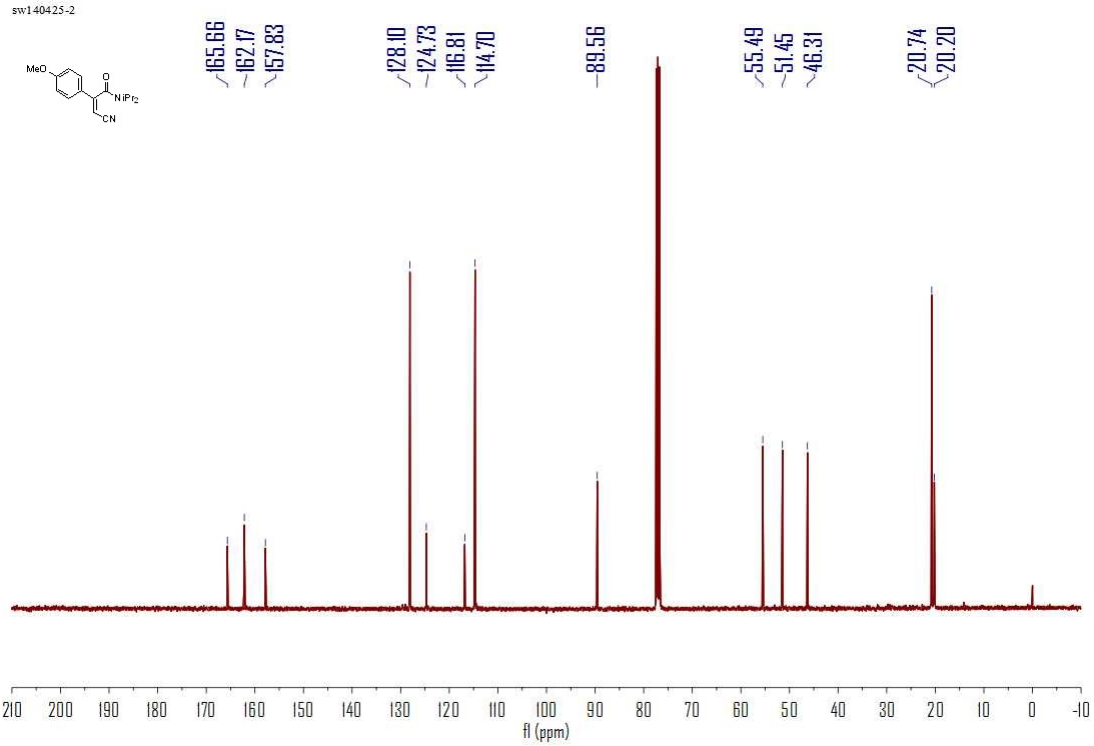


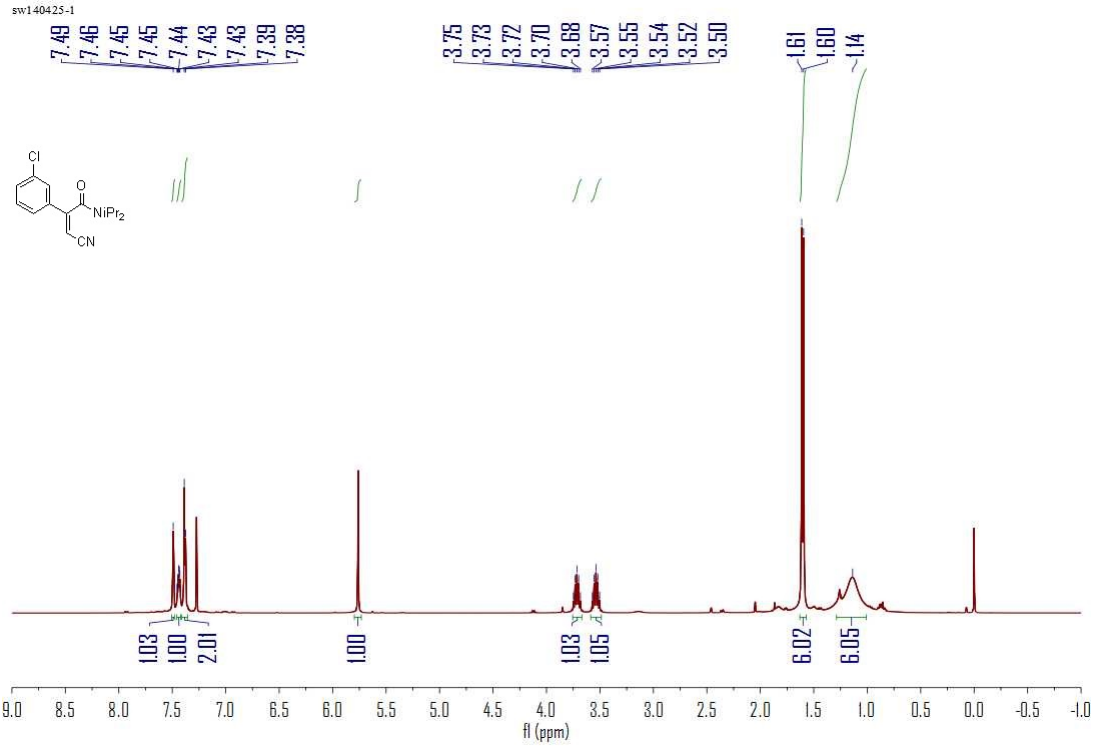
sw140410-4

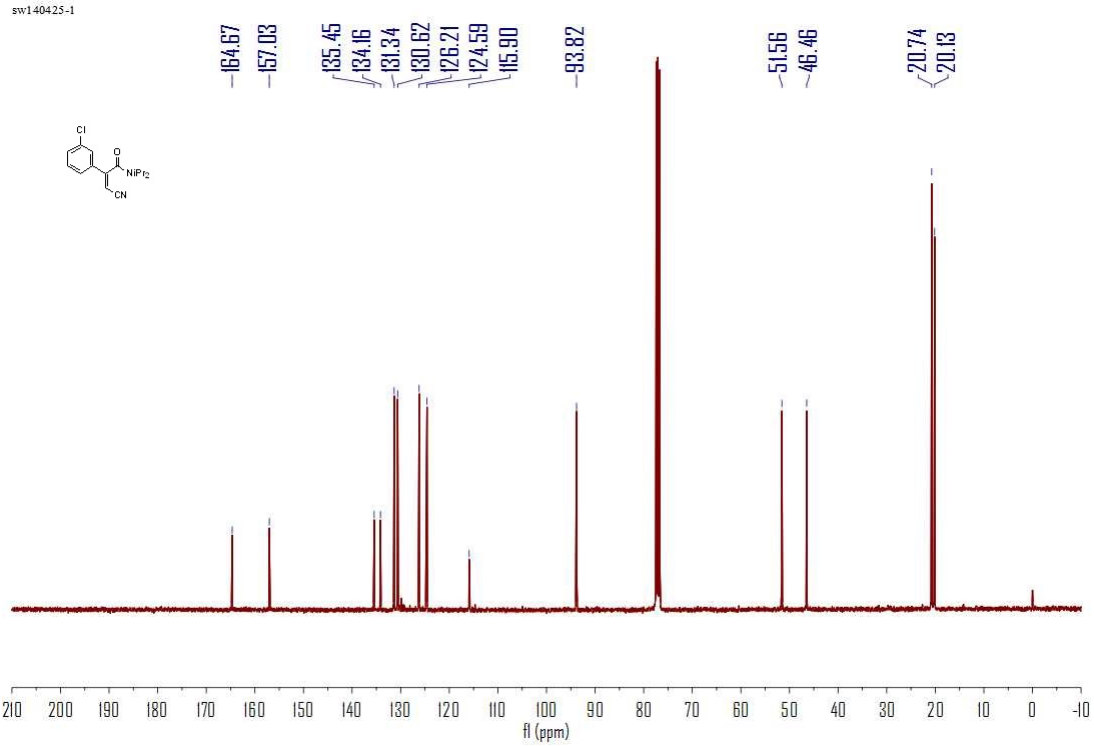


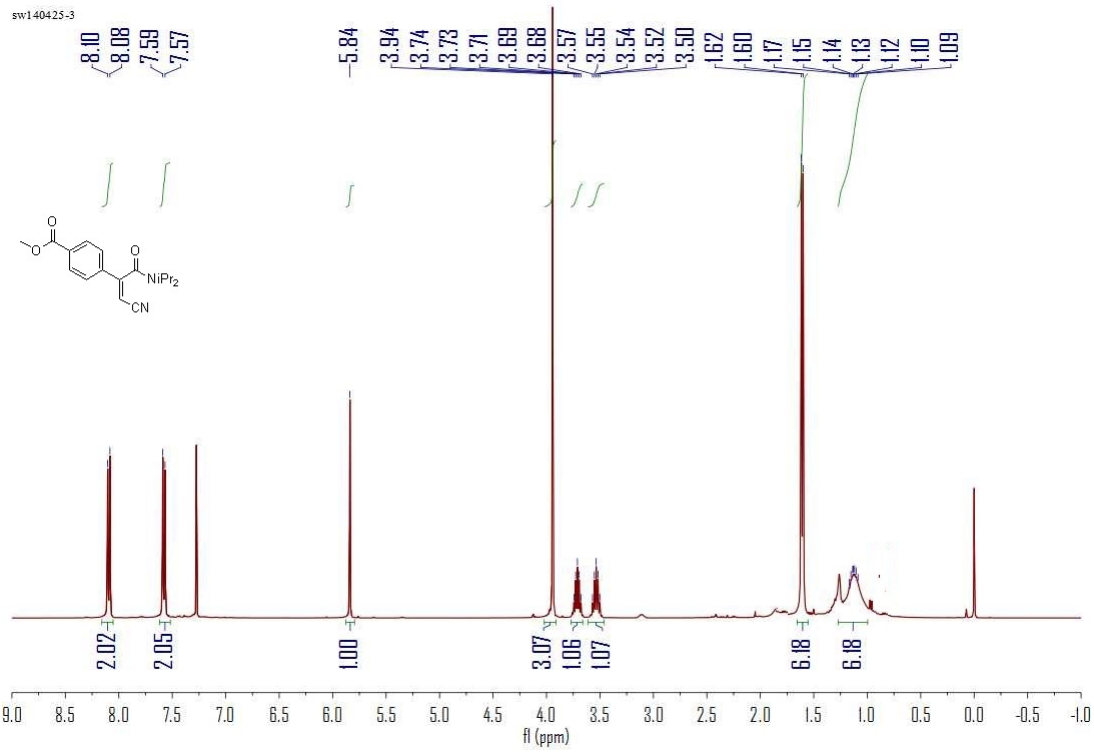
sw140425-2

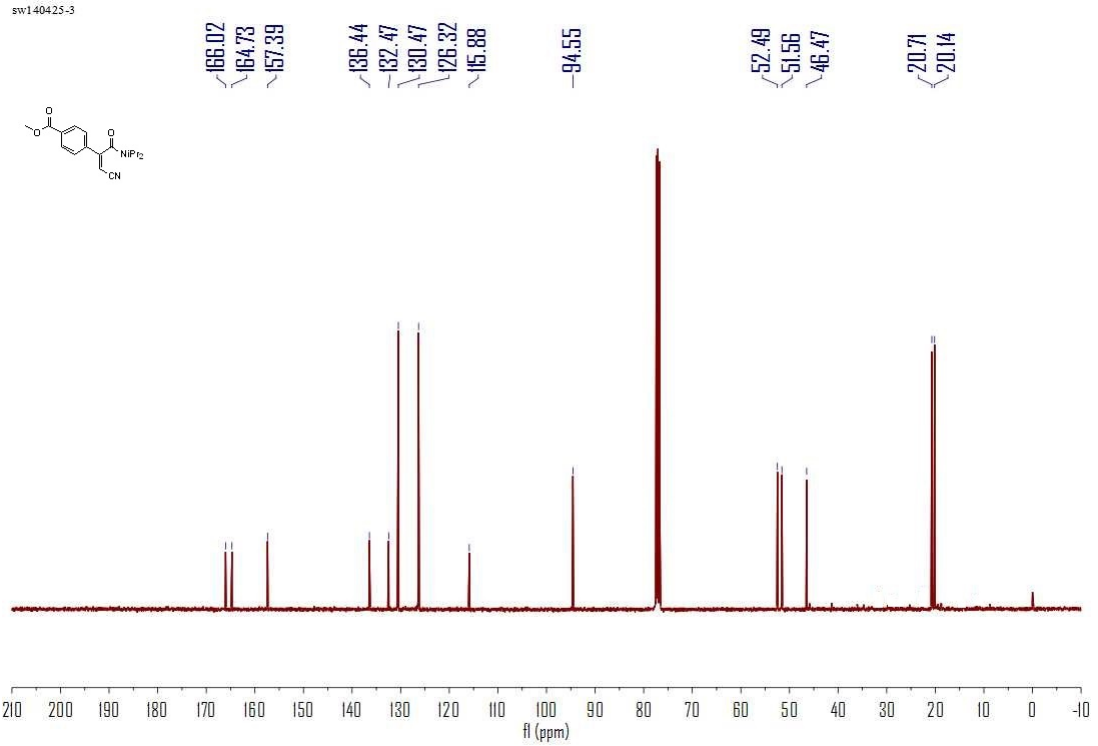


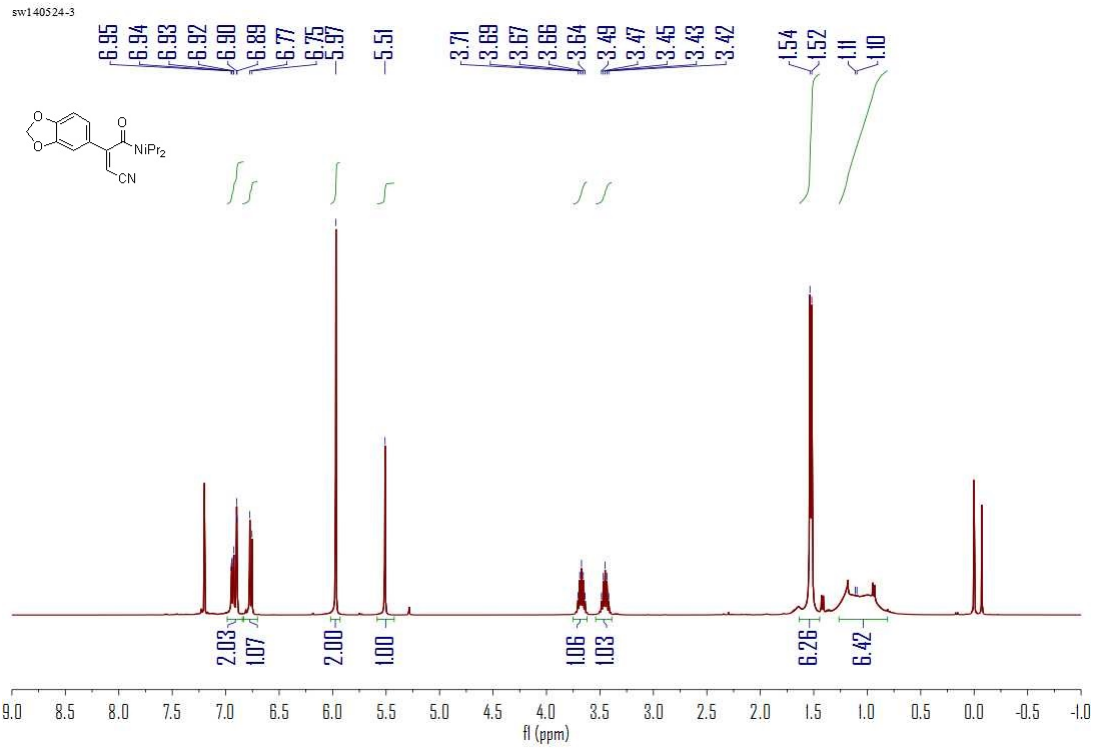

sw140425-2
MeO
O
NiPr2
CN
165.66
162.17
157.83
128.10
124.73
116.81
114.70
89.56
55.49
51.45
46.31
20.74
20.20
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


sw140425-1
Cl
O
NiPr2
CN
7.49
7.46
7.45
7.45
7.44
7.43
7.43
7.39
7.38
3.75
3.73
3.72
3.70
3.68
3.57
3.55
3.54
3.52
3.50
1.61
1.60
1.14
1.03
1.00
2.01
1.00
1.03
1.05
6.02
6.05
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 -1.0
f1 (ppm)

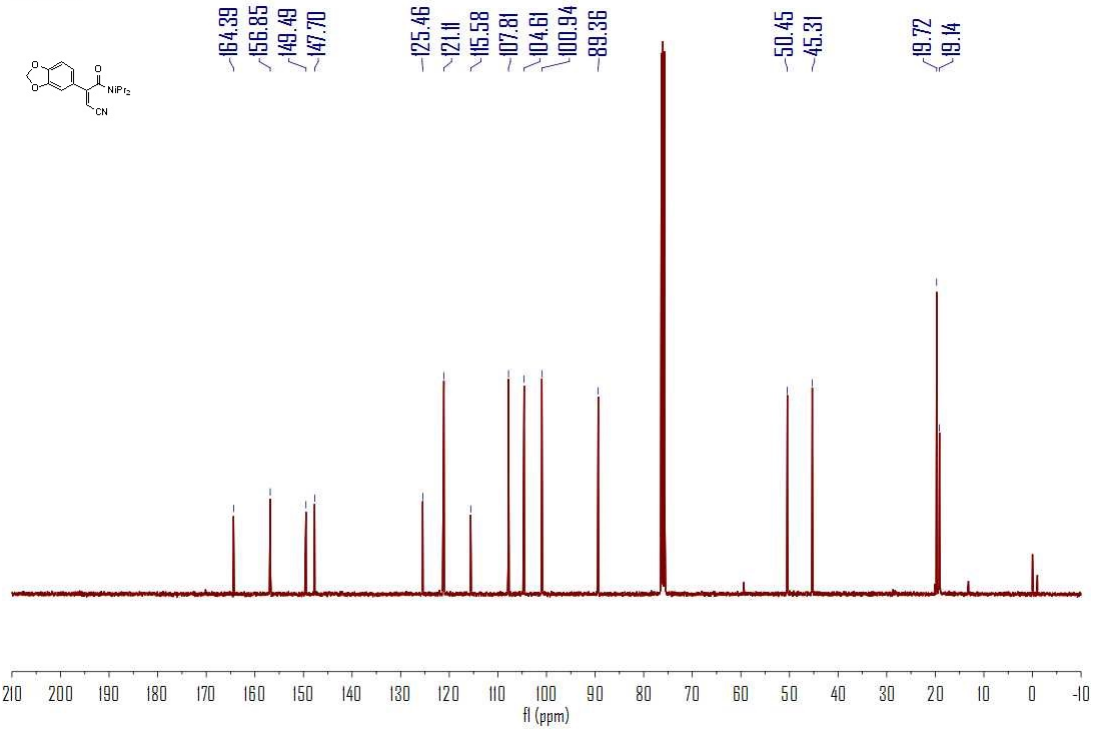

sw140425-1
164.67
157.03
135.45
134.16
131.34
130.62
126.21
124.59
115.90
93.82
51.56
46.46
20.74
20.13
Cl
O
NiPr2
CN
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


sw140425-3
8.10
8.08
7.59
7.57
5.84
3.94
3.74
3.73
3.71
3.69
3.68
3.57
3.55
3.54
3.52
3.50
1.62
1.60
1.17
1.15
1.14
1.13
1.12
1.10
1.09
O
O
NiPr2
CN
2.02
2.05
1.00
3.07
1.06
1.07
6.18
6.18
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 -1.0
f1 (ppm)

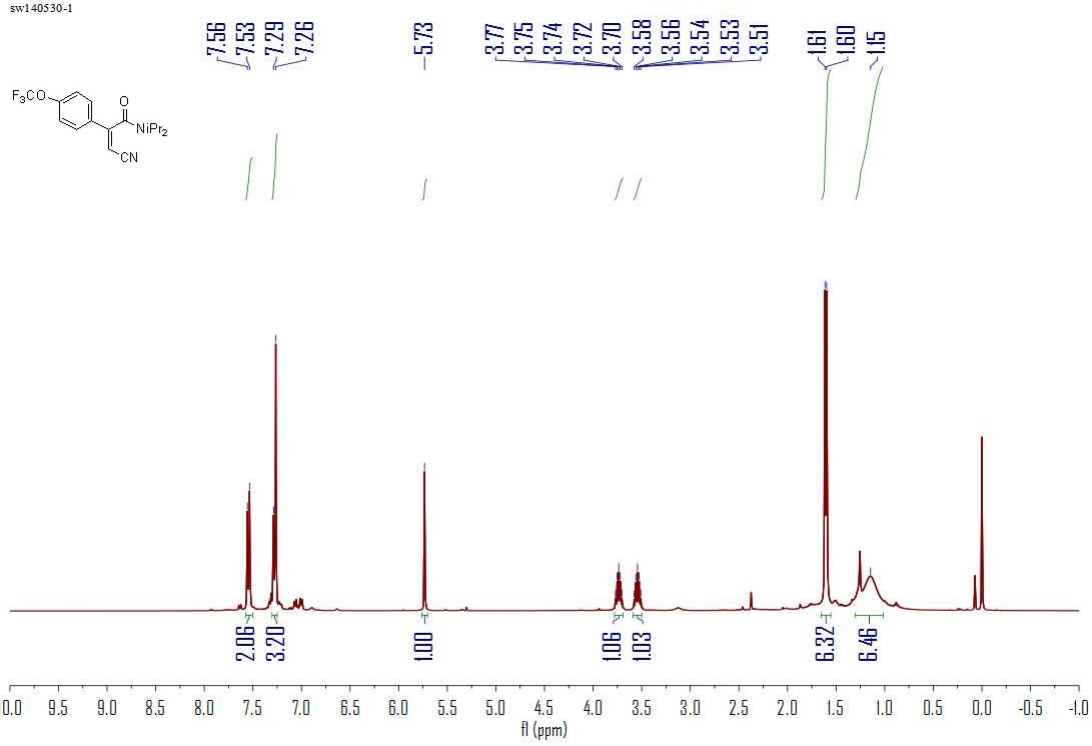

sw140425-3
166.02
164.73
157.39
136.44
132.47
130.47
126.32
115.88
94.55
52.49
51.56
46.47
20.71
20.14
NiPr2
CN
f1 (ppm)

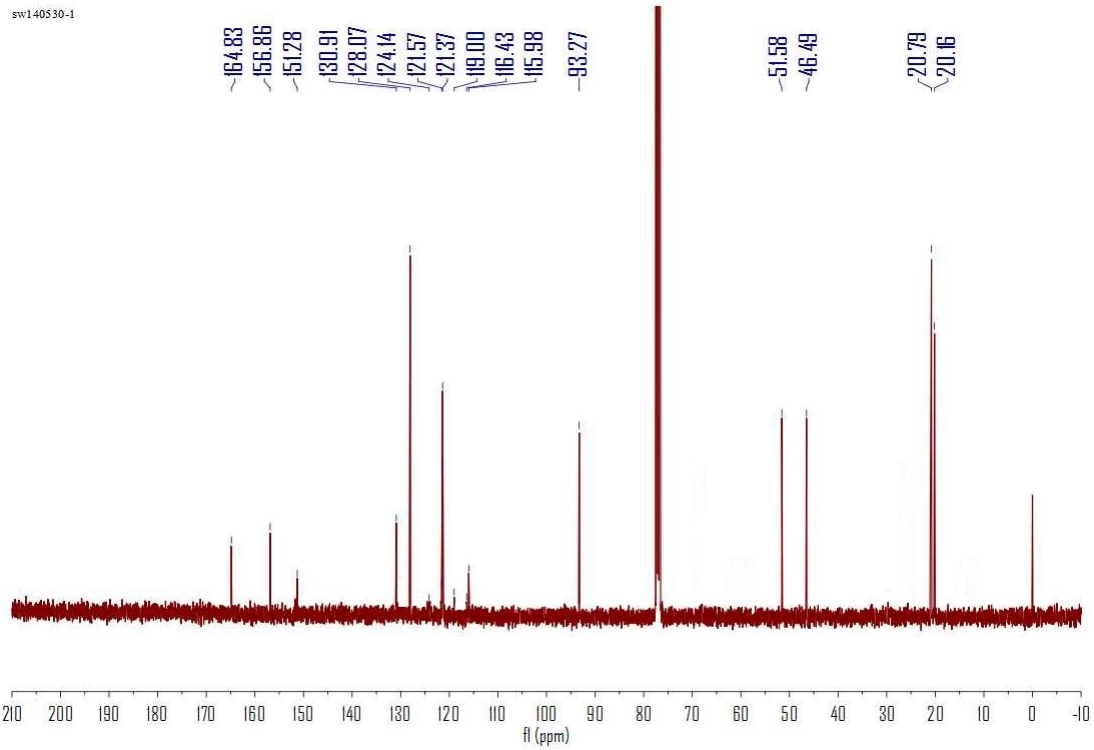

sw140524-3
6.95
6.94
6.93
6.92
6.90
6.89
6.77
6.75
5.97
5.51
3.71
3.69
3.67
3.66
3.64
3.49
3.47
3.45
3.43
3.42
1.54
1.52
1.11
1.10
NiPr2
CN
2.03
1.07
2.00
1.00
1.06
1.03
6.26
6.42
f1 (ppm)

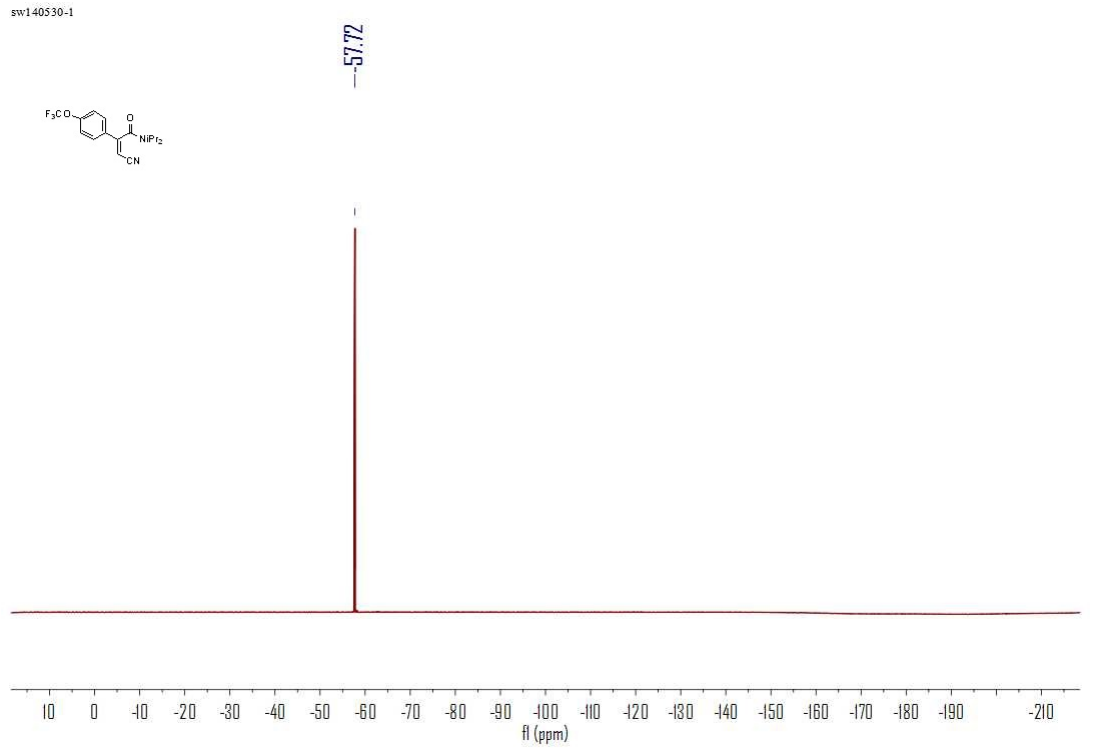
sw140521-5

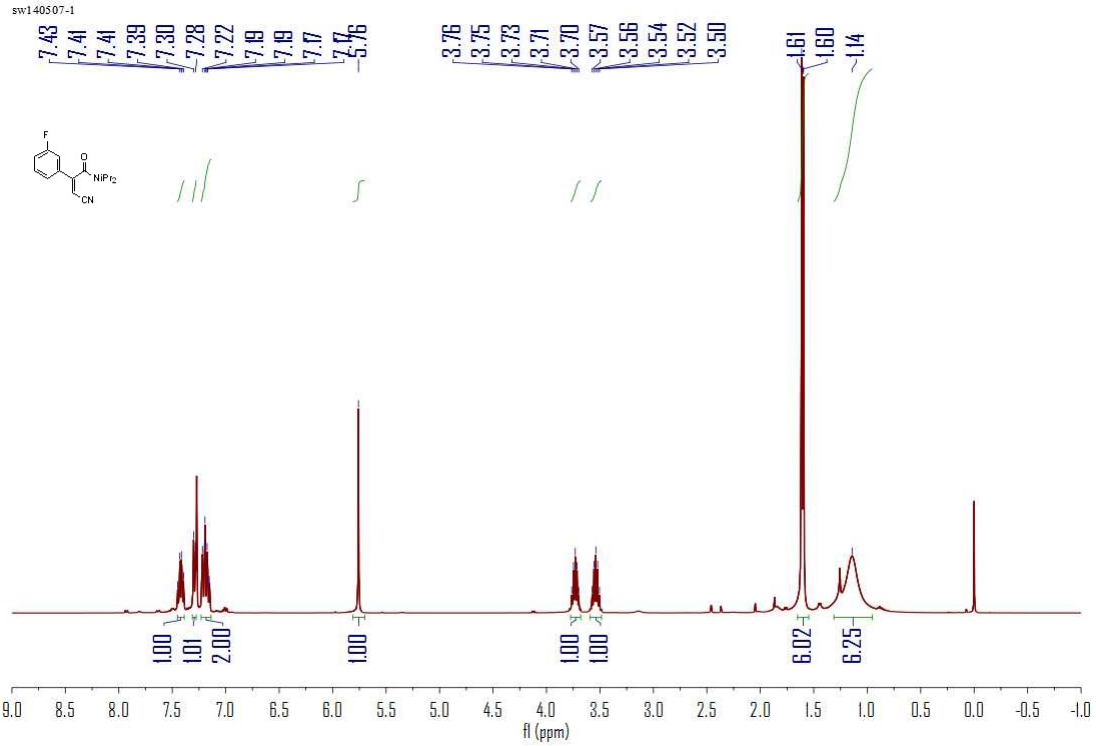


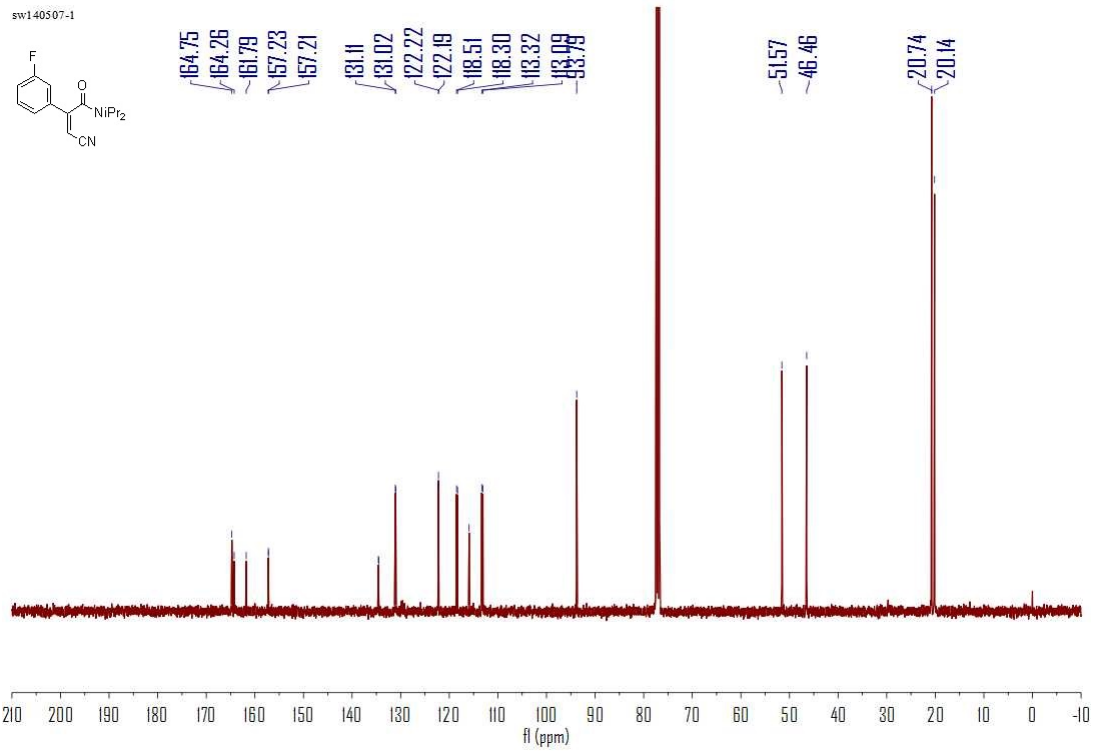
sw140530-1




sw140530-1
164.83
156.86
151.28
130.91
128.07
124.14
121.57
121.37
119.00
116.43
115.98
93.27
51.58
46.49
20.79
20.16
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

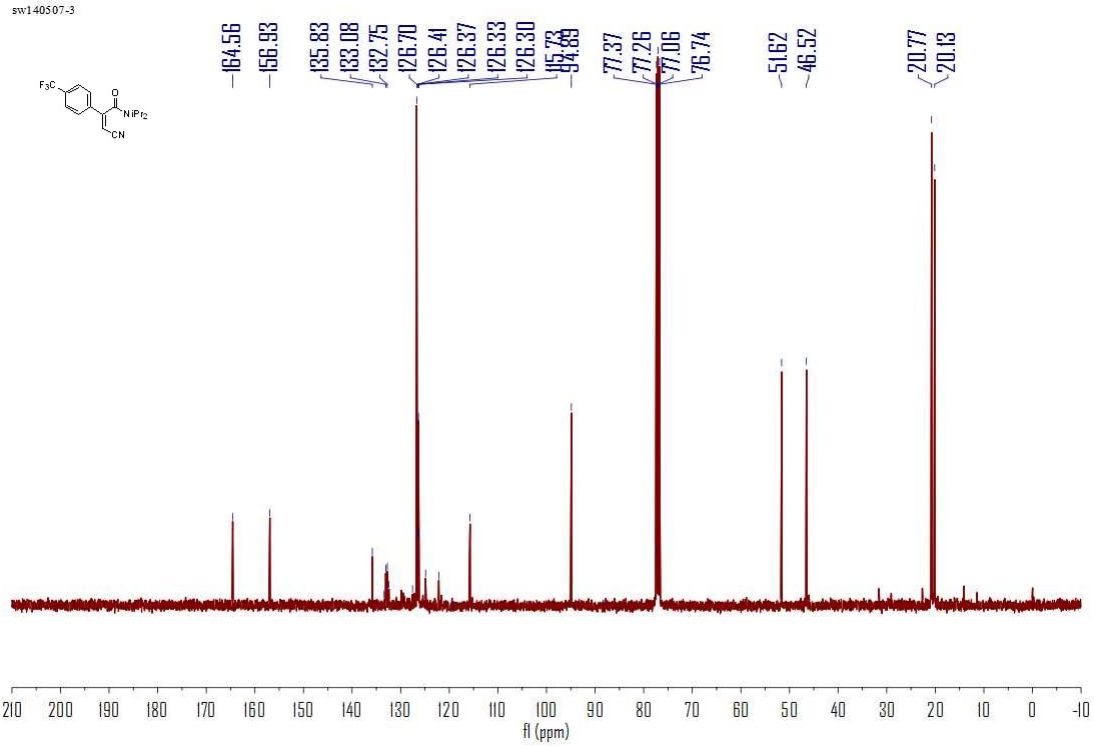

sw140530-1
57.72
F3CO
O
NiPr2
CN
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -210
f1 (ppm)

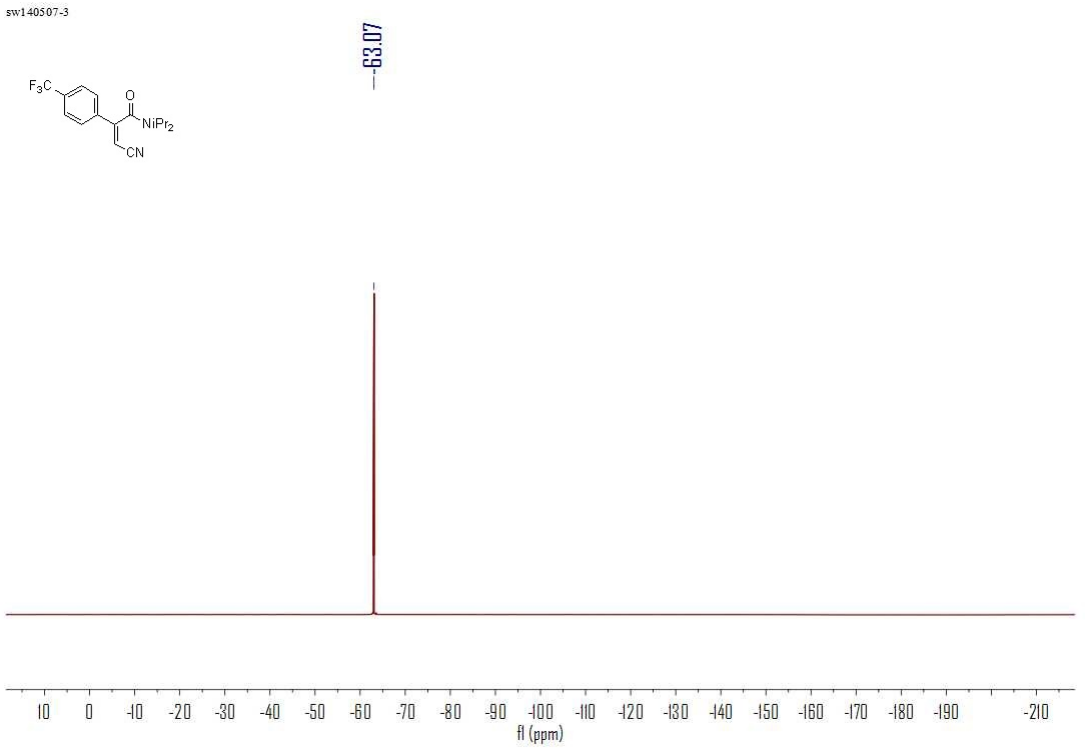

sw140507-1
7.43
7.41
7.41
7.39
7.30
7.28
7.22
7.19
7.19
7.17
5.76
3.76
3.75
3.73
3.71
3.70
3.57
3.56
3.54
3.52
3.50
1.61
1.60
1.14
F
O
NiPr2
CN
1.00
1.01
2.00
1.00
1.00
1.00
6.02
6.25
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

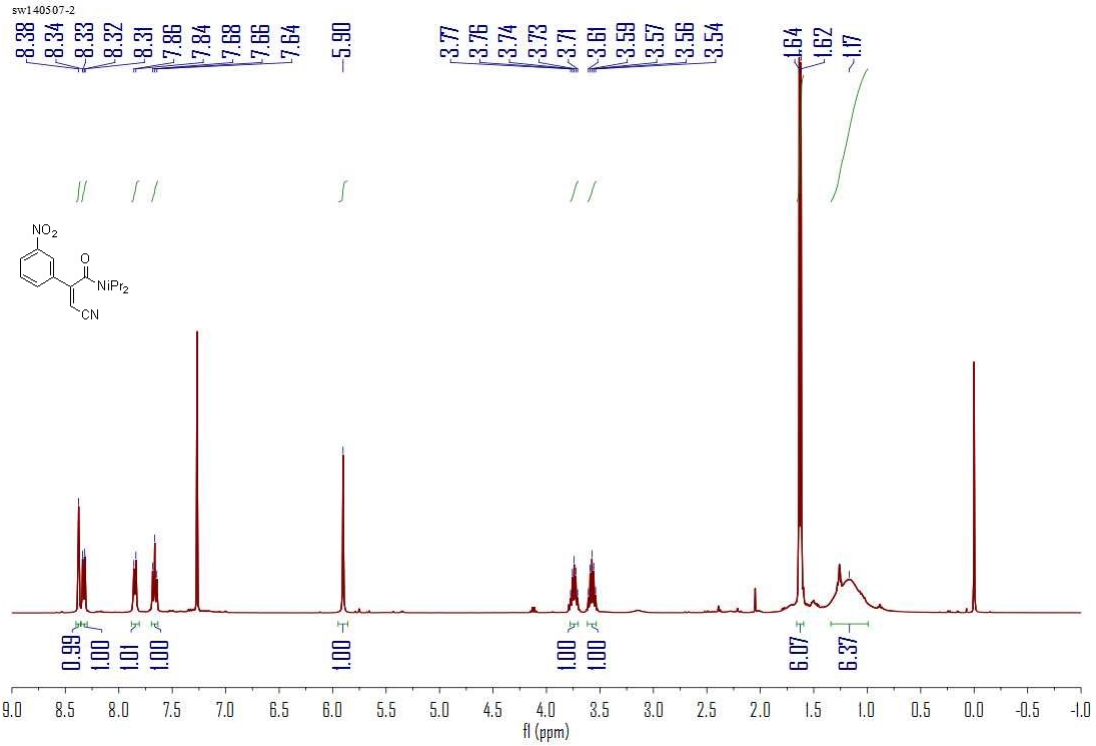

sw140507-1
164.75
164.26
161.79
157.23
157.21
131.11
131.02
122.22
122.19
118.51
118.30
113.32
93.79
51.57
46.46
20.74
20.14
F
O
NiPr2
CN
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

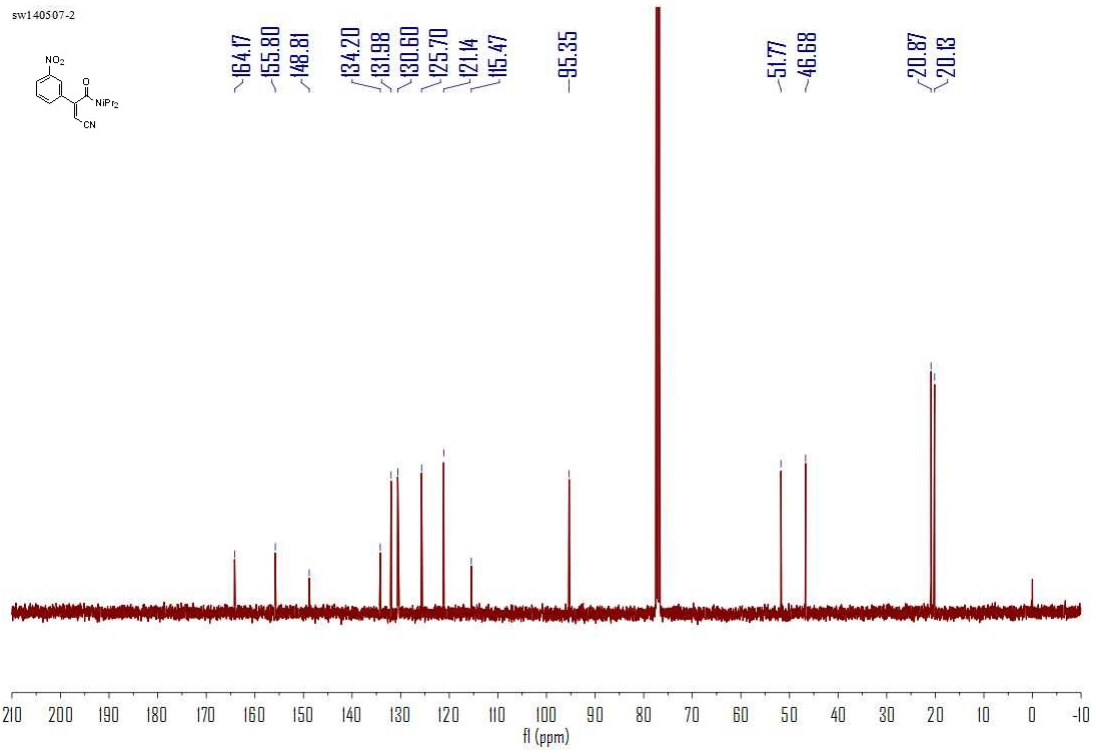
sw140507-1

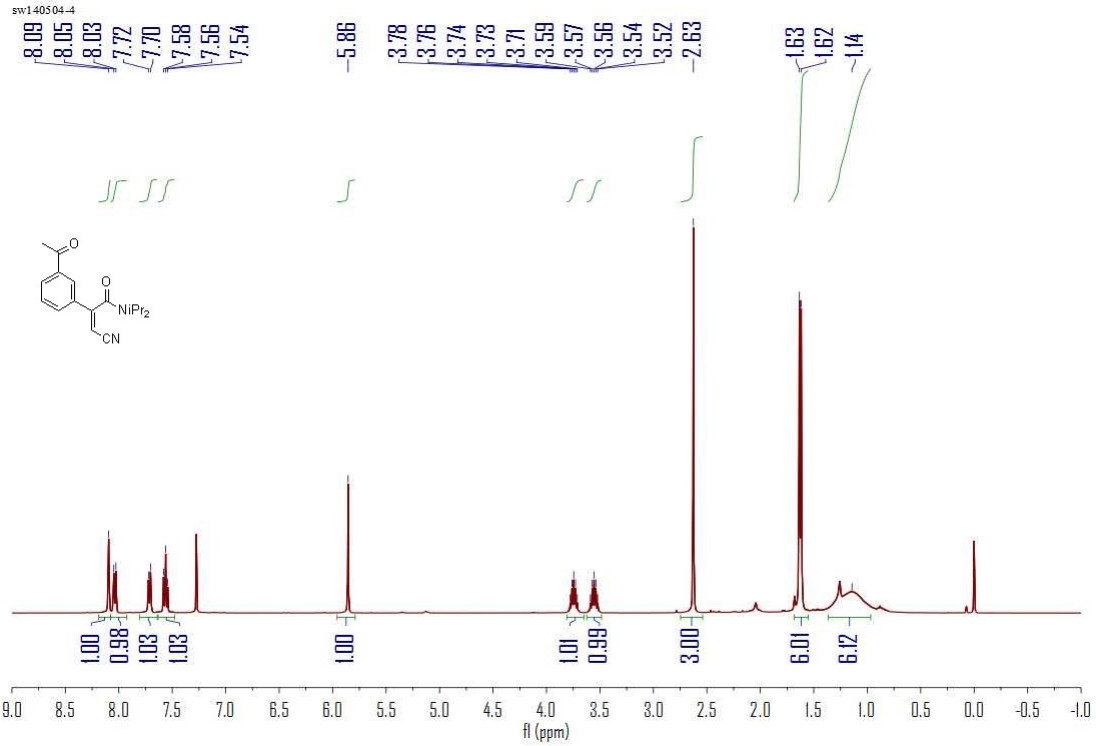
sw140507-3

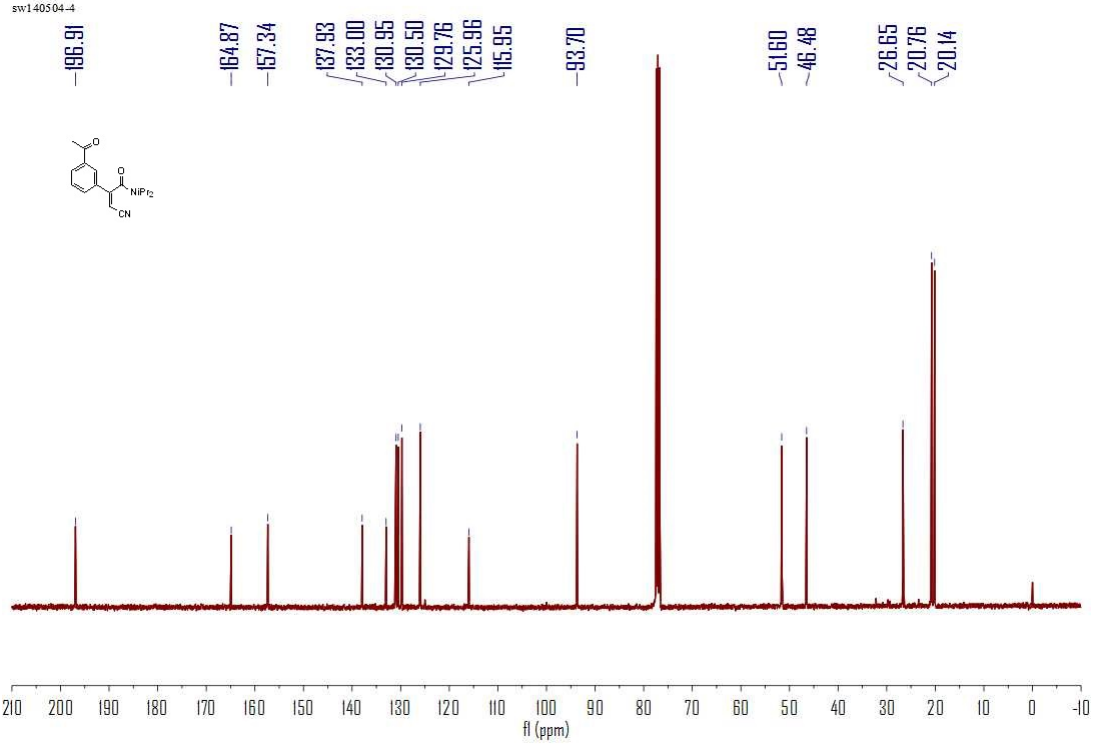

sw140507-3
164.56
156.93
135.83
133.08
132.75
126.70
126.41
126.37
126.33
126.30
115.73
94.88
77.37
77.26
77.06
76.74
51.62
46.52
20.77
20.13
F3C
O
NiPr2
CN
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

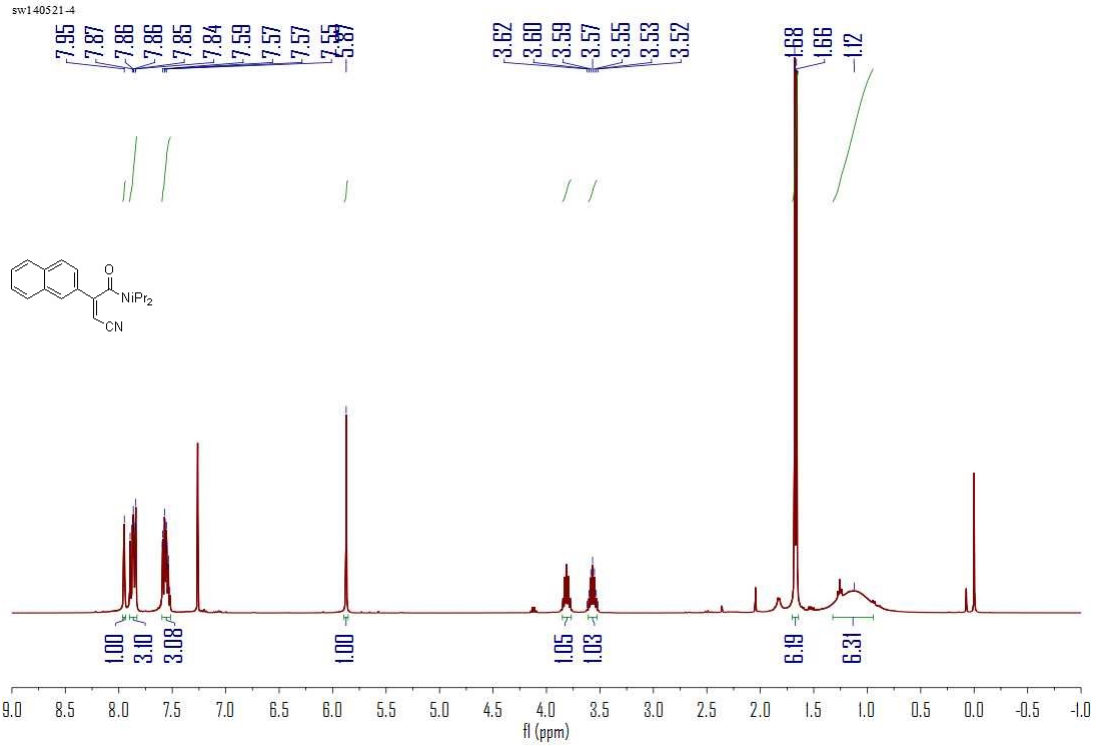

sw140507-3
-63.07
F3C
O
NiPr2
CN
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -210
f1 (ppm)


sw140507-2
8.38
8.34
8.33
8.32
8.31
7.86
7.84
7.68
7.66
7.64
5.90
3.77
3.76
3.74
3.73
3.71
3.61
3.59
3.57
3.56
3.54
1.64
1.62
1.17
NO2
O
NiPr2
CN
0.99
1.00
1.01
1.00
1.00
1.00
1.00
6.07
6.37
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 -1.0
f1 (ppm)


sw140507-2
NO2
O
NiPr2
CN
164.17
155.80
148.81
134.20
131.98
130.60
125.70
121.14
115.47
95.35
51.77
46.68
20.87
20.13
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


sw140504-4
8.09
8.05
8.03
7.72
7.70
7.58
7.56
7.54
5.86
3.78
3.76
3.74
3.73
3.71
3.59
3.57
3.56
3.54
3.52
2.63
1.63
1.62
1.14
O
O
NiPr2
CN
1.00
0.98
1.03
1.03
1.00
1.01
0.99
3.00
6.01
6.12
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
-1.0
f1 (ppm)


sw140504-4
196.91
164.87
157.34
137.93
133.00
130.95
130.50
129.76
125.96
115.95
93.70
51.60
46.48
26.65
20.76
20.14
O
O
NiPr2
CN
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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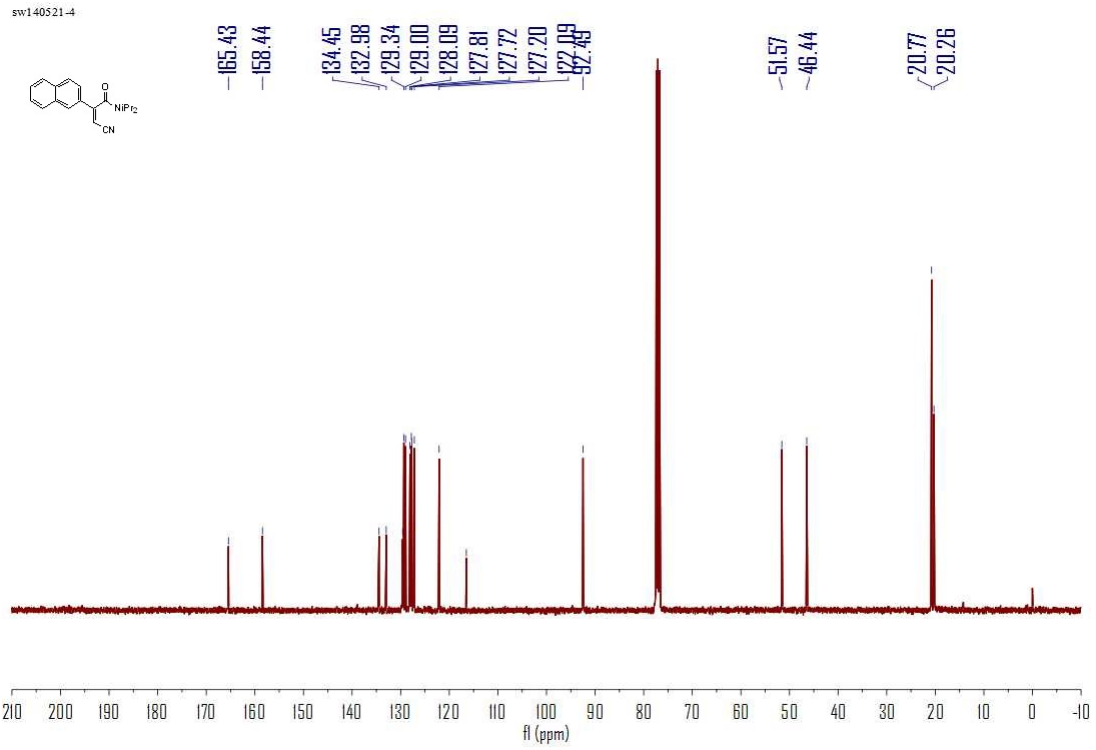
7.87
7.86
7.86
7.85
7.84
7.59
7.57
7.57
7.55
5.87
3.62
3.60
3.59
3.57
3.55
3.53
3.52
1.68
1.66
1.12
NiPr2
CN
1.00
3.10
3.08
1.00
1.05
1.03
6.19
6.31
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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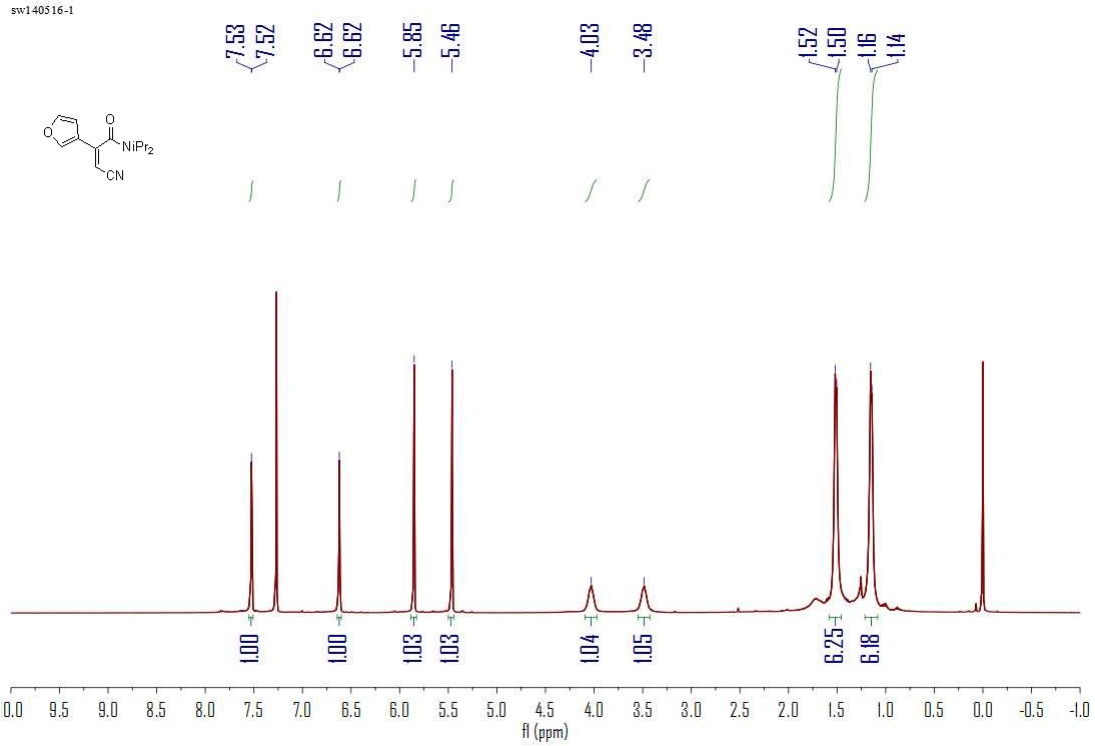
158.44
134.45
132.98
129.34
129.00
128.09
127.81
127.72
127.20
122.09
92.49
51.57
46.44
20.77
20.26
NiPr2
CN
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.52
6.62
6.62
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5.46
4.03
3.48
1.52
1.50
1.16
1.14
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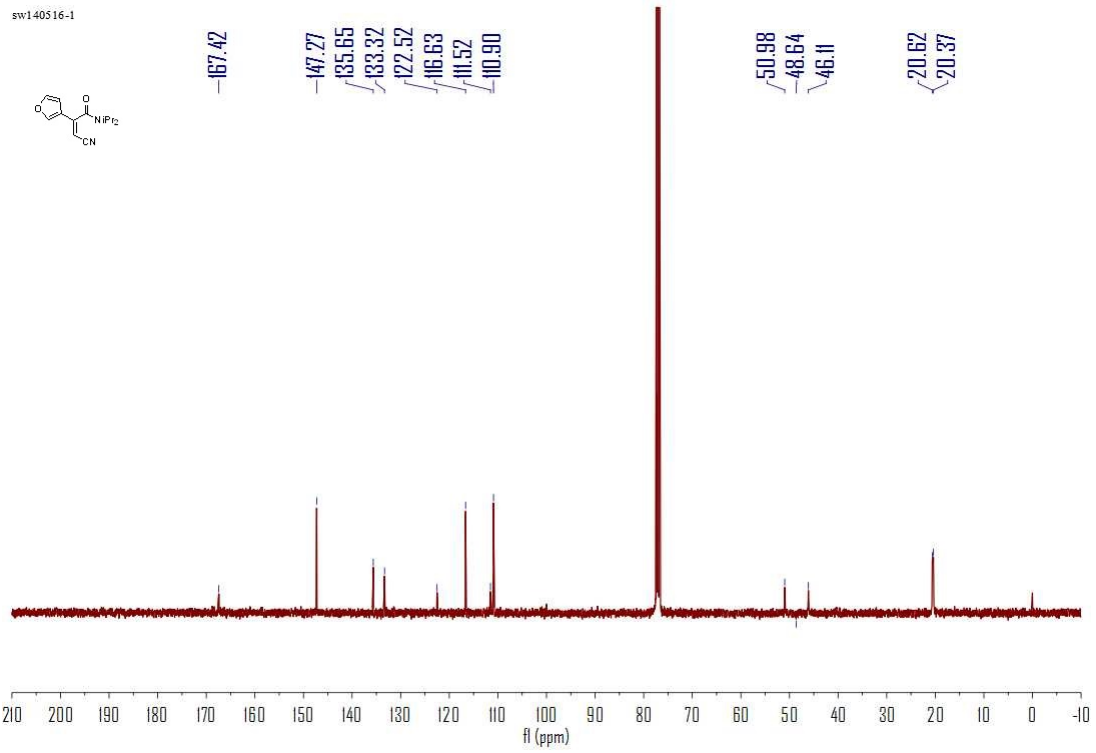
CN
1.00
1.00
1.03
1.03
1.04
1.05
6.25
6.18
fl (ppm)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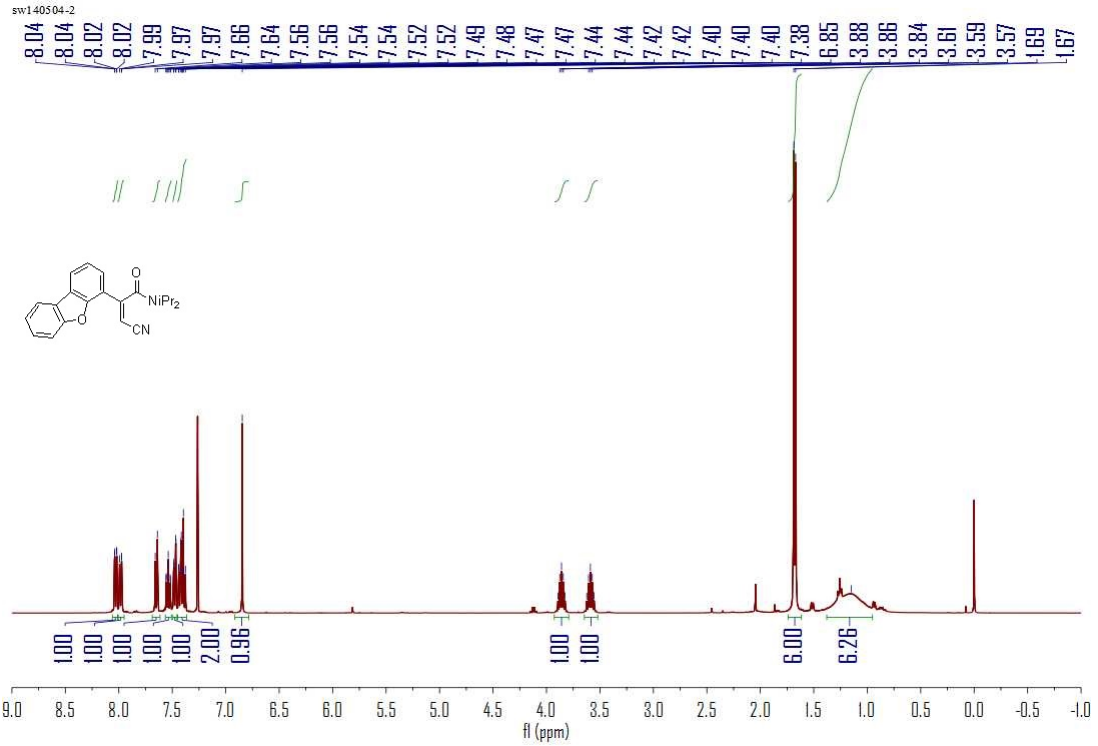
20.62
20.37
NiPr2
CN
fl (ppm)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8.04
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8.02
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7.97
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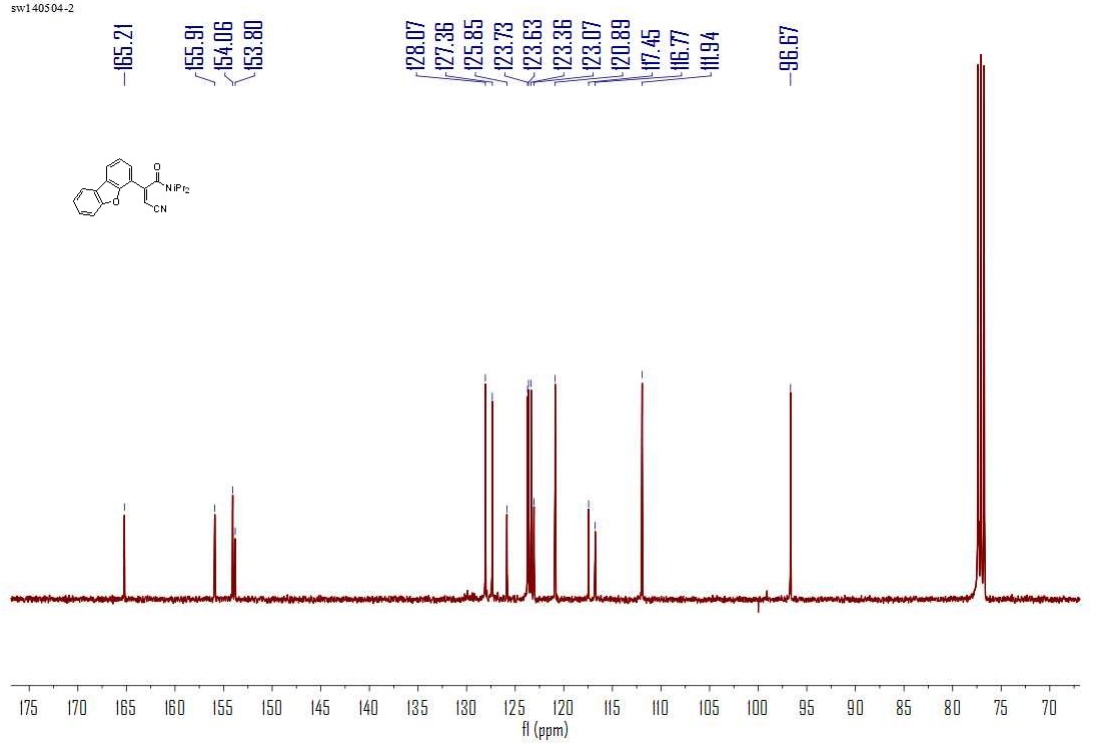
7.64
7.56
7.56
7.54
7.54
7.52
7.52
7.49
7.48
7.47
7.47
7.44
7.44
7.42
7.42
7.40
7.40
7.40
7.38
6.85
3.88
3.86
3.84
3.61
3.59
3.57
1.69
1.67
O
NiPr2
CN
1.00
1.00
1.00
1.00
1.00
2.00
0.96
1.00
1.00
6.00
6.26
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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154.06
153.80
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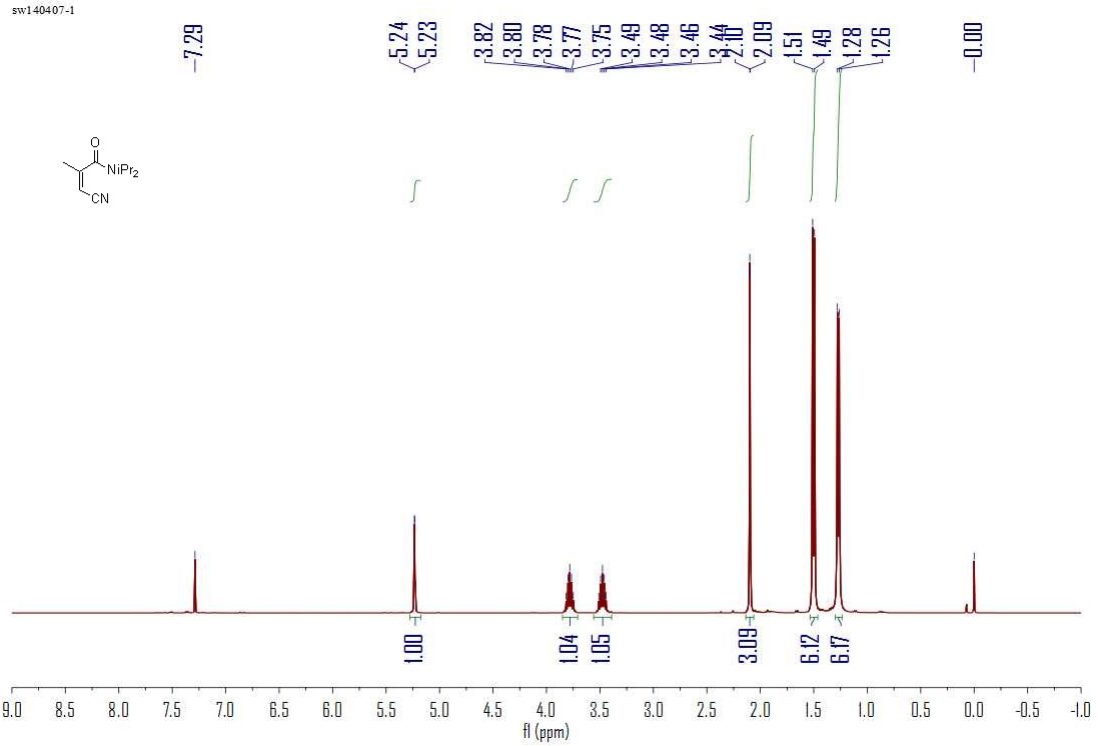
125.85
123.73
123.63
123.36
123.07
120.89
117.45
116.77
111.94
96.67
O
NiPr2
CN
175
170
165
160
155
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
f1 (ppm)


sw140407-1
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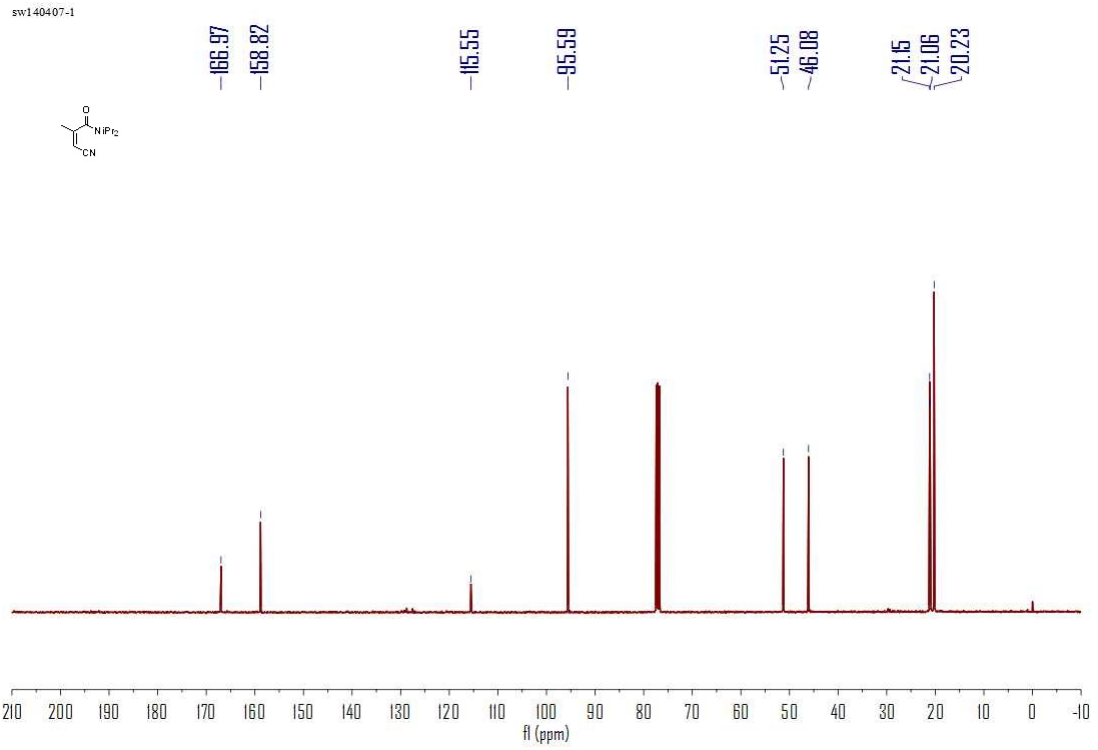
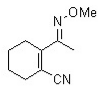
5.24
5.23
3.82
3.80
3.78
3.77
3.75
3.49
3.48
3.46
3.44
2.10
2.09
1.51
1.49
1.28
1.26
0.00
NiPr2
CN
1.00
1.04
1.05
3.09
6.12
6.17
f1 (ppm)

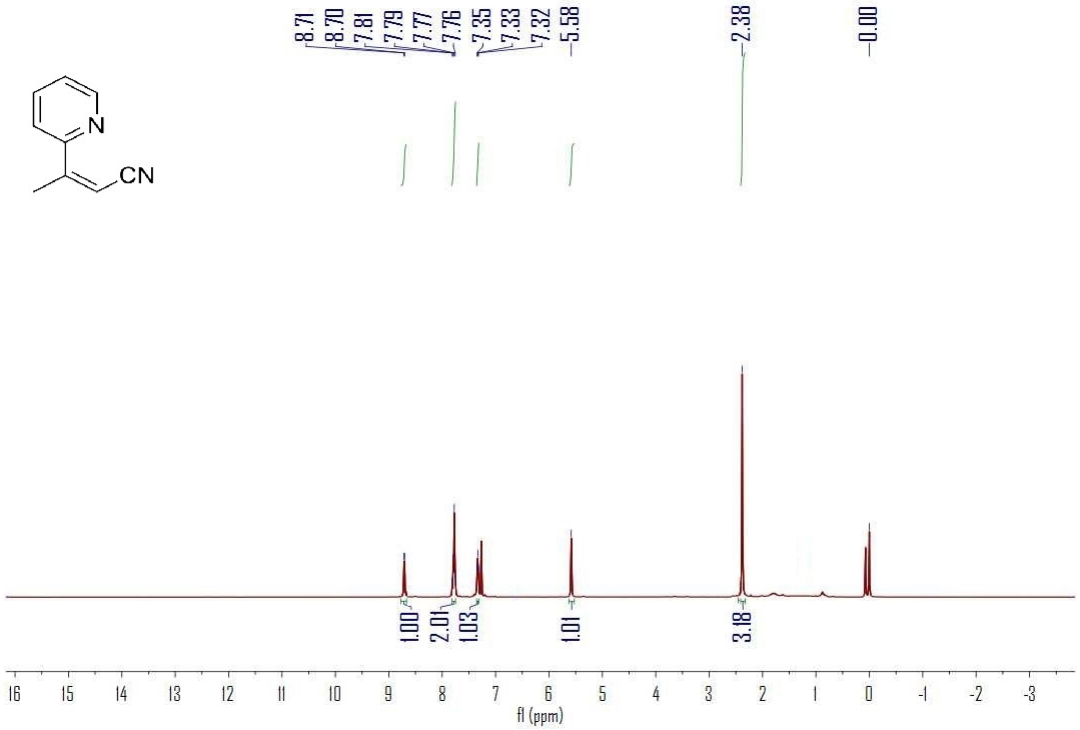

sw140407-1
166.97
158.82
115.55
95.59
51.25
46.08
21.15
21.06
20.23
NiPr2
CN
f1 (ppm)

sw140710-1

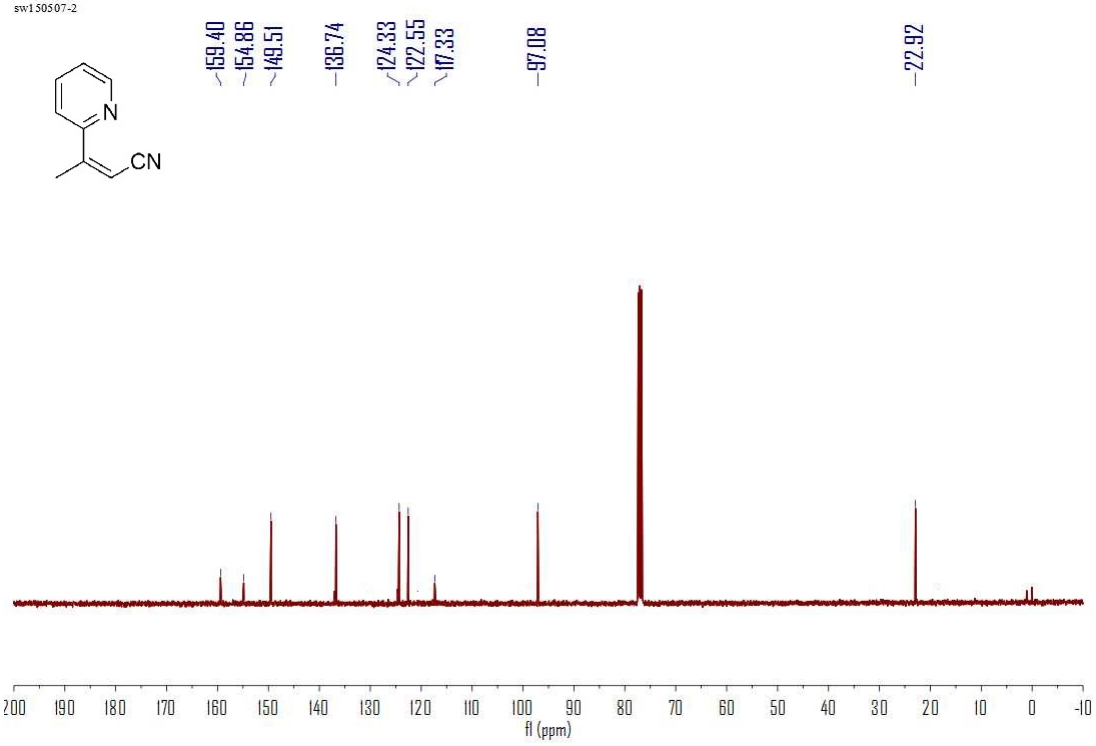


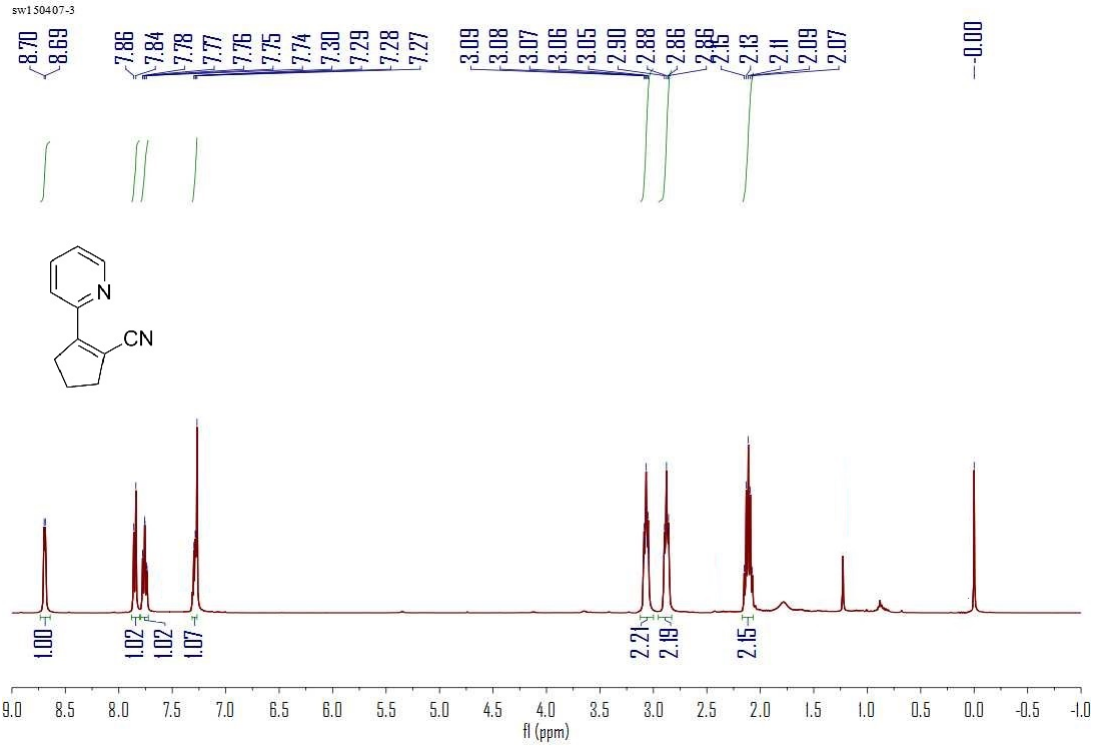
sw140524-1

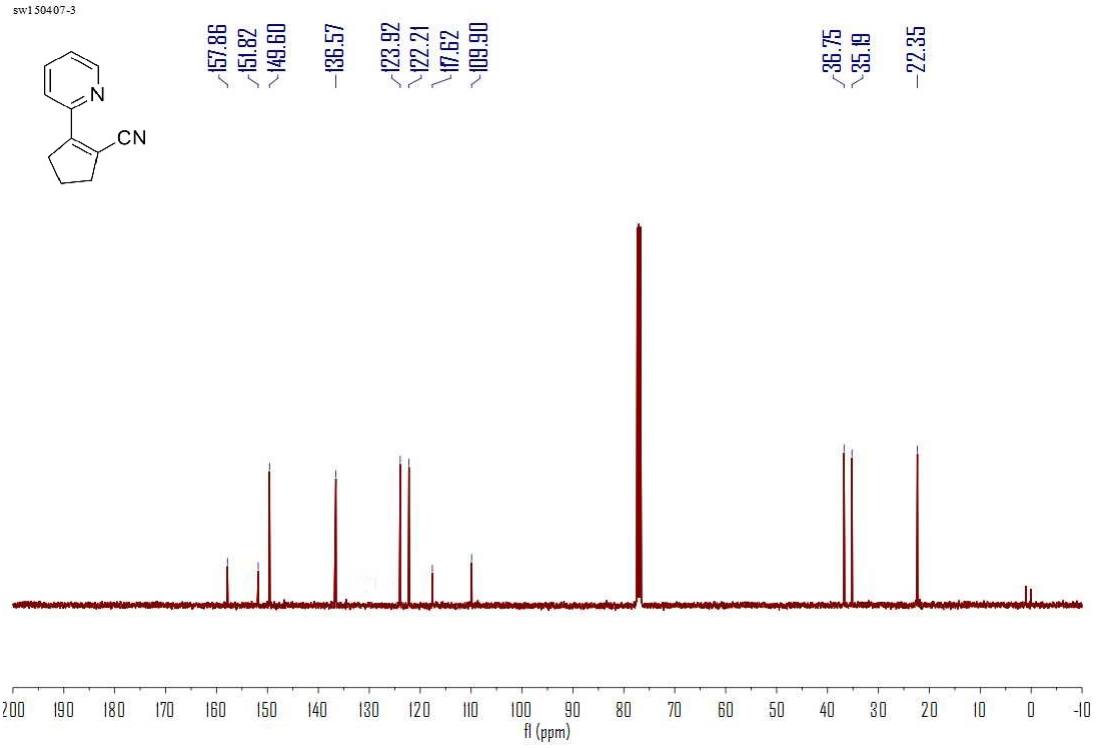
sw150507-2

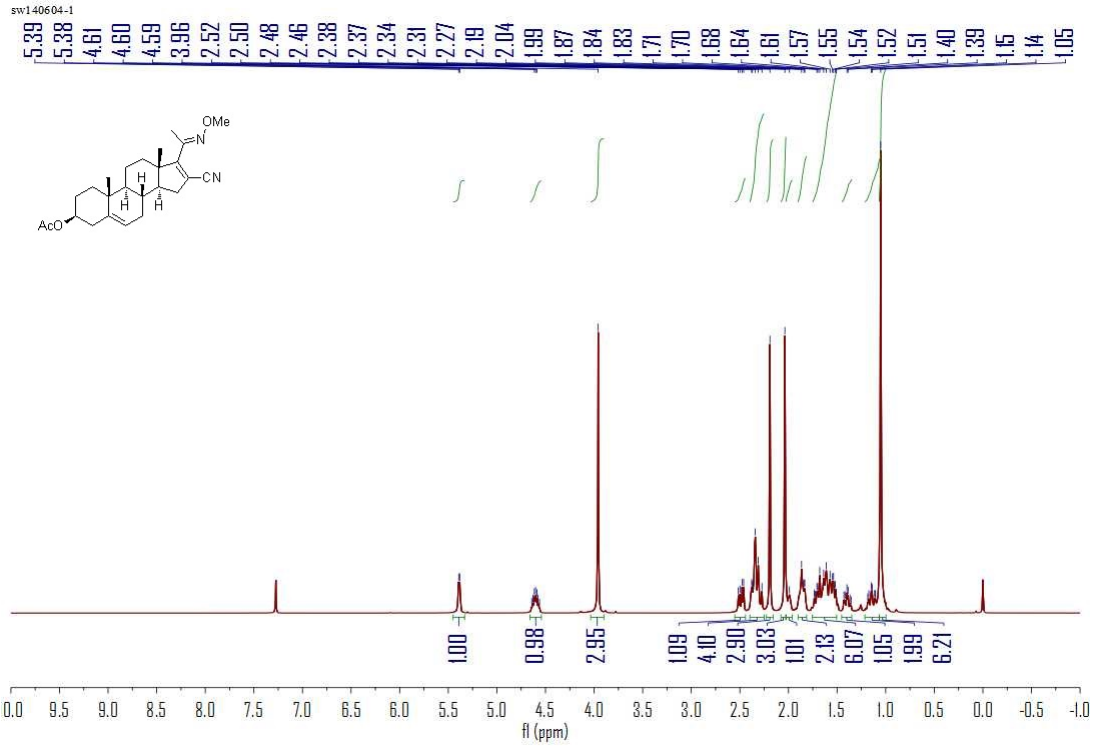


sw150507-2




sw150407-3
8.70
8.69
7.86
7.84
7.78
7.77
7.76
7.75
7.74
7.30
7.29
7.28
7.27
3.09
3.08
3.07
3.06
3.05
2.90
2.88
2.86
2.15
2.13
2.11
2.09
2.07
0.00
N
CN
1.00
1.02
1.02
1.07
2.21
2.19
2.15
f1 (ppm)


sw150407-3
157.86
151.82
149.60
136.57
123.92
122.21
117.62
109.90
36.75
35.19
22.35
N
CN
f1 (ppm)


sw140604-1
5.39
5.38
4.61
4.60
4.59
3.96
2.52
2.50
2.48
2.46
2.38
2.37
2.34
2.31
2.27
2.19
2.04
1.99
1.87
1.84
1.83
1.71
1.70
1.68
1.64
1.61
1.57
1.55
1.54
1.52
1.51
1.40
1.39
1.15
1.14
1.05
OMe
N
CN
H
H
AcO
1.00
0.98
2.95
1.09
4.10
2.90
3.03
1.01
2.13
6.07
1.05
1.99
6.21
0.0
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
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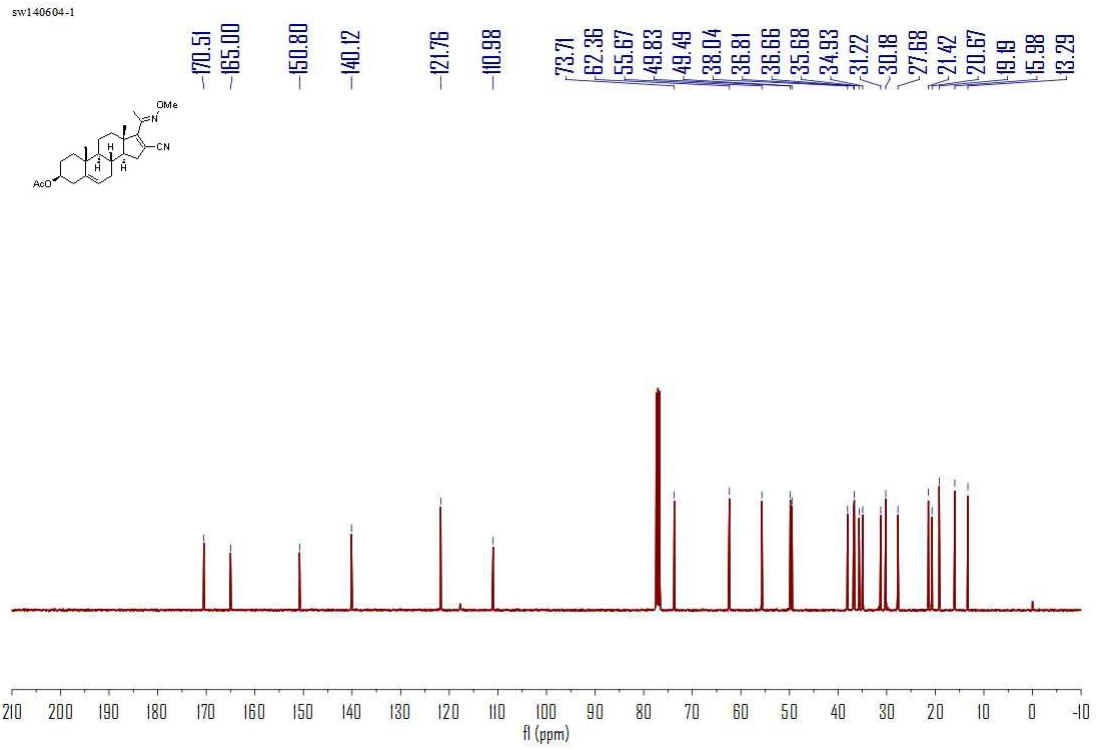
-1.0
f1 (ppm)


sw140604-1
170.51
165.00
150.80
140.12
121.76
110.98
73.71
62.36
55.67
49.83
49.49
38.04
36.81
36.66
35.68
34.93
31.22
30.18
27.68
21.42
20.67
19.19
15.98
13.29
OMe
N
CN
H
H
AcO
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

V. X-ray Crystal data

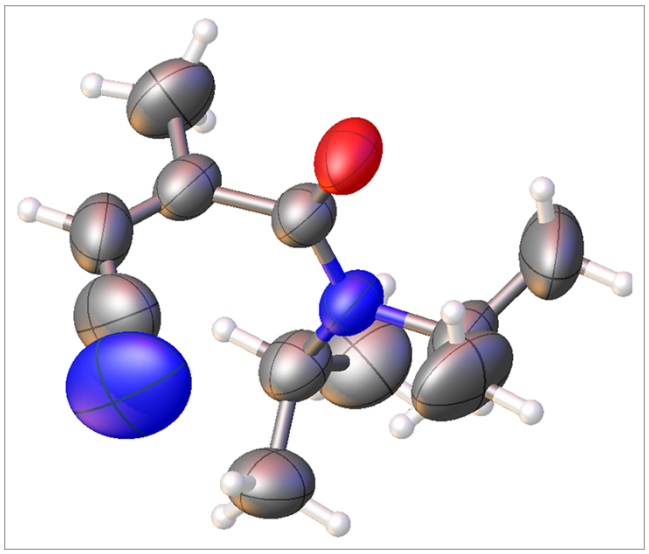


Table 1 Crystal data and structure refinement for SW140328-1.

Identification code	SW140328-1
Empirical formula	$C_{11}H_{18}N_2O$
Formula weight	194.27
Temperature/K	291(2)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	7.3992(4)
b/Å	9.2492(7)
c/Å	18.3176(11)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1253.60(14)
Z	4
ρ_{calc}mg/mm^3	1.029
m/mm^{-1}	0.527
F(000)	424.0
Crystal size/mm^3	0.36 × 0.3 × 0.21
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection	10.716 to 138.7°

Index ranges	$-8 \leqslant h \leqslant 5$, $-11 \leqslant k \leqslant 11$, $-22 \leqslant l \leqslant 20$
Reflections collected	2813
Independent reflections	2016 [R_{int} = 0.0185, R_{sigma} = 0.0302]
Data/restraints/parameters	2016/0/133
Goodness-of-fit on F^2	1.065
Final R indexes [I>=2σ (I)]	R_1 = 0.0457, wR_2 = 0.1288
Final R indexes [all data]	R_1 = 0.0624, wR_2 = 0.1672
Largest diff. peak/hole / e Å^{-3}	0.12/-0.11
Flack parameter	1.6(5)

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for SW140328-1. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O1	-5846(3)	-138(3)	-729.1(14)	92.8(9)
N1	-8665(3)	433(3)	-1128.2(14)	73.2(8)
C2	-7499(4)	-252(4)	-693.8(17)	69.1(8)
C3	-8224(4)	-1268(4)	-129.7(17)	75.8(9)
C5	-8540(5)	-2666(5)	-293(3)	96.2(13)
C7	-10639(4)	317(5)	-1025(2)	83.1(10)
C4	-8398(7)	-706(6)	628.3(19)	113.7(15)
C6	-8356(9)	-3196(5)	-1010(3)	121.0(16)
N2	-8251(13)	-3627(6)	-1583(4)	198(3)
C10	-7980(6)	1345(6)	-1728(2)	102.5(14)
C8	-11571(6)	-403(6)	-1653(2)	110.8(14)
C11	-6978(8)	447(9)	-2284(2)	149(3)
C9	-11480(7)	1804(6)	-850(3)	130.2(18)
C12	-6923(9)	2650(6)	-1456(4)	149(2)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for SW140328-1. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\cdots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	66.4(13)	108(2)	103.9(17)	42.5(16)	-5.7(11)	-1.6(13)
N1	70.4(15)	81.5(17)	67.6(13)	19.2(14)	-4.7(11)	0.2(13)
C2	66.0(17)	75.6(19)	65.7(15)	11.3(16)	-2.9(13)	1.1(15)
C3	68.4(17)	93(2)	66.6(17)	18.9(17)	-2.4(14)	1.3(19)
C5	89(2)	87(3)	112(3)	35(2)	-6(2)	-11(2)
C7	71.9(19)	94(2)	83(2)	16(2)	-1.3(15)	10.8(18)
C4	136(4)	137(4)	68(2)	8(2)	4(2)	6(3)
C6	151(4)	85(3)	127(4)	-13(3)	-16(4)	7(3)
N2	281(9)	136(4)	176(5)	-65(4)	-32(5)	50(5)
C10	88(3)	129(3)	90(2)	53(3)	-10(2)	-7(2)
C8	84(2)	142(4)	106(3)	7(3)	-21(2)	-7(3)
C11	131(4)	238(7)	78(2)	46(4)	17(3)	2(5)
C9	111(3)	136(4)	144(4)	7(3)	1(3)	44(3)
C12	142(5)	119(4)	187(5)	81(4)	-40(4)	-40(4)

Table 4 Bond Lengths for SW140328-1.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	C2	1.229(4)	C5	C6	1.407(7)
N1	C2	1.334(4)	C7	C8	1.498(6)
N1	C7	1.476(4)	C7	C9	1.544(6)
N1	C10	1.474(4)	C6	N2	1.125(7)
C2	C3	1.496(4)	C10	C11	1.510(8)
C3	C5	1.348(5)	C10	C12	1.523(7)
C3	C4	1.488(5)			

Table 5 Bond Angles for SW140328-1.

Atom	Atom	Atom	Angle/	Atom	Atom	Atom	Angle/
C2	N1	C7	121.9(3)	C3	C5	C6	121.6(4)
C2	N1	C10	119.6(3)	N1	C7	C8	112.9(3)
C10	N1	C7	118.5(3)	N1	C7	C9	111.1(4)
O1	C2	N1	124.9(3)	C8	C7	C9	111.7(4)
O1	C2	C3	116.6(3)	N2	C6	C5	178.3(8)

N1	C2	C3	118.5(3)	N1	C10	C11	110.9(4)
C5	C3	C2	120.7(3)	N1	C10	C12	112.8(4)
C5	C3	C4	121.9(4)	C11	C10	C12	113.9(5)
C4	C3	C2	117.1(4)				

Table 6 Torsion Angles for SW140328-1.

A	B	C	D	Angle/	A	B	C	D	Angle/
O1	C2	C3	C5	-91.1(4)	C7	N1	C2	O1	-176.0(4)
O1	C2	C3	C4	82.9(5)	C7	N1	C2	C3	5.6(5)
N1	C2	C3	C5	87.5(4)	C7	N1	C10	C11	-116.2(4)
N1	C2	C3	C4	-98.6(4)	C7	N1	C10	C12	114.8(5)
C2	N1	C7	C8	-115.7(4)	C4	C3	C5	C6	-179.3(4)
C2	N1	C7	C9	117.8(4)	C10	N1	C2	O1	3.5(6)
C2	N1	C10	C11	64.3(5)	C10	N1	C2	C3	-174.9(4)
C2	N1	C10	C12	-64.8(5)	C10	N1	C7	C8	64.8(5)
C2	C3	C5	C6	-5.7(6)	C10	N1	C7	C9	-61.7(5)

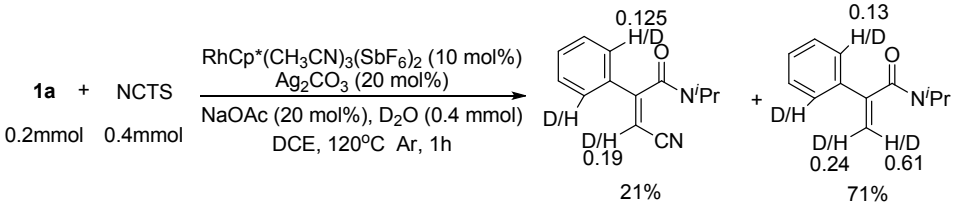
Table 7 Hydrogen Atom Coordinates (Å×10⁴) and Isotropic Displacement Parameters (Å²×10³) for SW140328-1.

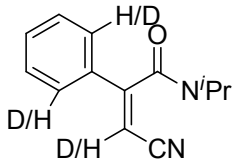
Atom	*x*	*y*	*z*	U(eq)
H5	-8888	-3297	76	115
H7	-10834	-297	-596	100
H4A	-7240	-379	797	171
H4B	-8829	-1461	942	171
H4C	-9237	86	634	171
H10	-9048	1729	-1978	123
H8A	-10973	-1298	-1764	166
H8B	-11528	219	-2072	166
H8C	-12807	-592	-1526	166
H11A	-7690	-384	-2410	223
H11B	-5844	138	-2083	223
H11C	-6763	1017	-2714	223
H9A	-11491	2388	-1284	195
H9B	-10777	2276	-480	195
H9C	-12695	1674	-679	195

H12A	-5789	2336	-1257	224
H12B	-7606	3137	-1084	224
H12C	-6707	3300	-1855	224

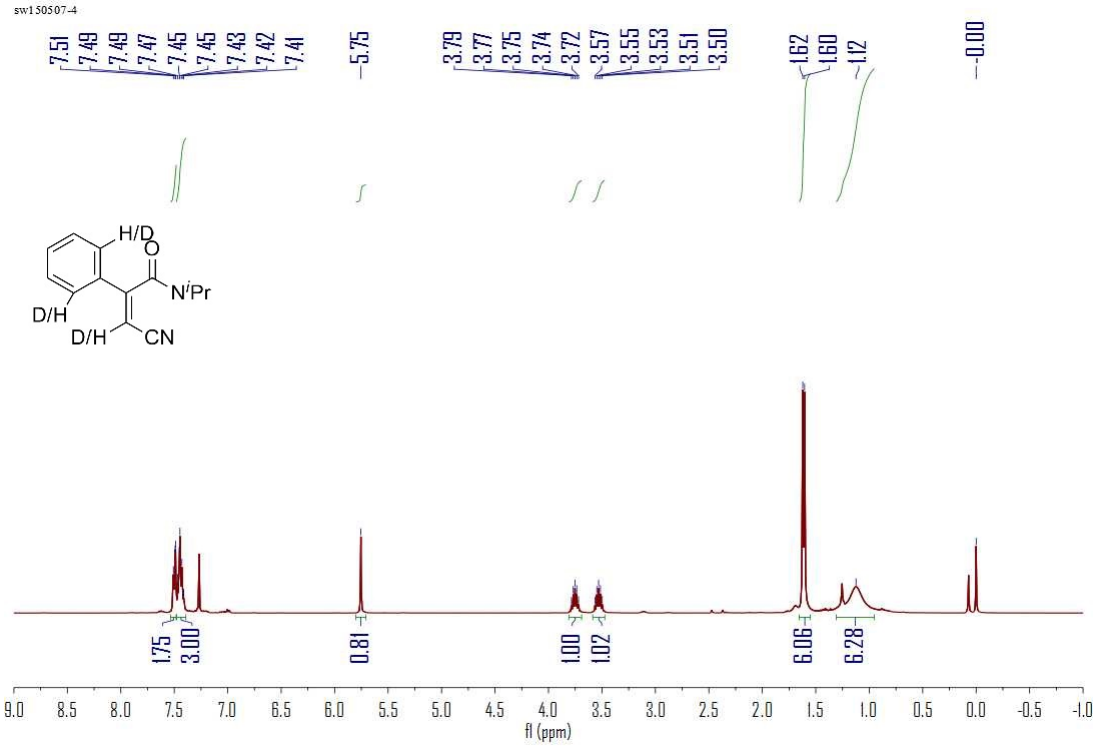
VI. Deuterium Exchange Experiments

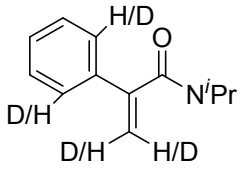
A 10mL Schlenk tube equipped with a magnetic stirrer was charged with $[Cp^*Rh(CH3CN)_3](SbF_6)_2$ (10 mol%), NaOAc (20 mol%), Ag_2CO_3 (20 mol%), N-cyano-N-phenyl-p-toluenesulfonamide **2** (0.4 mmol). The tube was evacuated and backfilled with argon for three times. Then acrylamide **1a** (0.2 mmol) and D_2O (0.4mmol) in DCE (1 mL) was added. After addition of all substrates, the reaction mixture was stirred and heated at 120°C for 1h. Then reaction was cooled to room temperature. Solvent and volatile reagents were removed by rotary evaporation and the residue was purified by flash column chromatography on silica gel to give starting material **1a** and product **3a**.



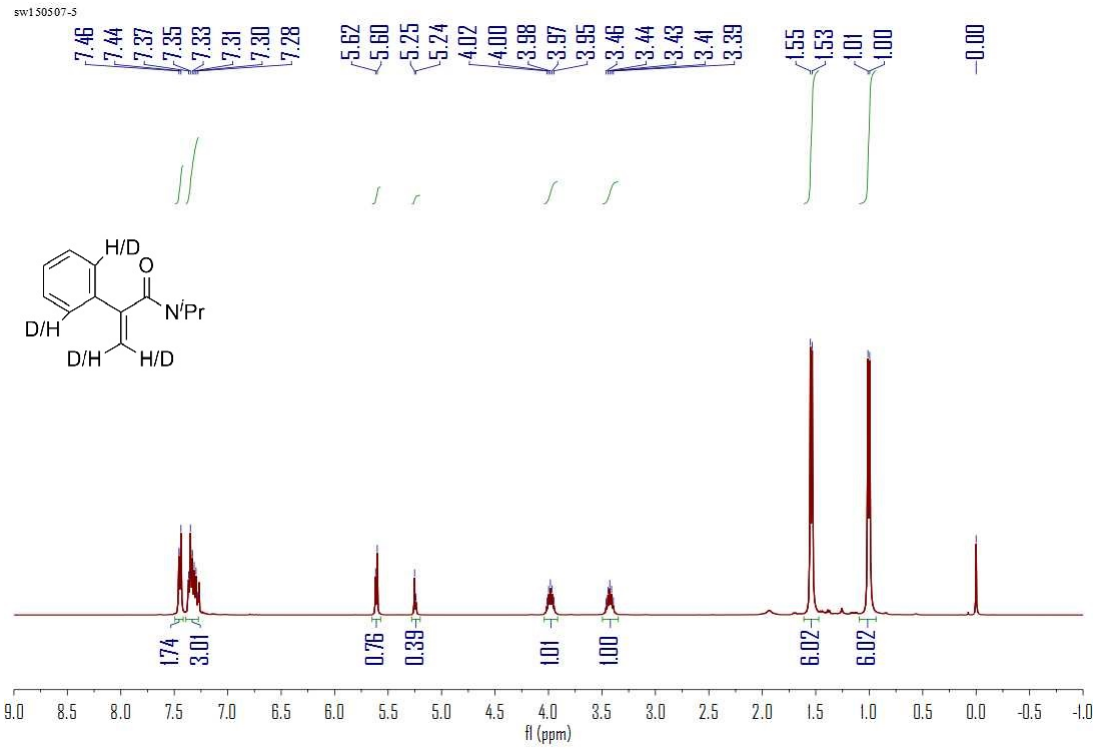


^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 – 7.48 (m, 1.75H), 7.48 – 7.39 (m, 3H), 5.75 (s, 0.81H), 3.75 (dt, *J* = 13.2, 6.6 Hz, 1H), 3.53 (dt, *J* = 13.6, 6.8 Hz, 1H), 1.61 (d, *J* = 6.8 Hz, 6H), 1.12 (s, 6H).





^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 7.5 Hz, 1.74H), 7.32 (m, 3H), 5.61 (m, 0.76H), 5.25 (m, 0.39H), 3.98 (dt, *J* = 13.3, 6.6 Hz, 1H), 3.43 (dt, *J* = 13.5, 6.8 Hz, 1H), 1.54 (d, *J* = 6.8 Hz, 6H), 1.00 (d, *J* = 6.7 Hz, 6H).



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