Supporting Informations

Palladium Catalyzed Hydroamidocarbonylation of 1,3-dienes

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Contents

General Remarks

All chemicals were purchased from Sigma-Aldrich, Strem, Acros, TCI or Alfa Aesar and used as received unless stated otherwise. Solvents (Anhydrous and under inert atmosphere) were collected from the solvent purification system and used under standard schlenk technique. 1,3-Butadiene is obtained from Evonik Industry. Isoprene was purchased from Acros and stored under -28°C under Argon atmosphere. NMR spectra were recorded on Bruker Avance 300 and Bruker ARX 400 spectrometers. Chemical shifts (ppm) are given relative to solvent: references for $CDCl_3$ were 7.26 ppm (^{1}H NMR) and 77.00 ppm (^{13}C NMR). Multiplets were assigned as s (singlet), d (doublet), t (triplet), q (quartet), p (pentet) dd (doublet of doublet), m (multiplet) and br. s (broad singlet). Unless otherwise stated, yields refer to isolated yields, estimated to be >95% pure according to ^{1}H-NMR spectroscopy or GC. GC-yields were calculated using isooctane as internal standard. All measurements were carried out at room temperature unless otherwise stated. Electron impact (EI) mass spectra were recorded on AMD 402 mass spectrometer (70 eV). High resolution mass spectra (HRMS) were recorded on Agilent 6210. The data are given as mass units per charge (m/z). Gas chromatography analysis was performed on an Agilent HP-7890A instrument with a FID detector and HP-5 capillary column (polydimethylsiloxane with 5% phenyl groups, 30 m, 0.32 mm i.d., 0.25 µm film thickness) using argon as carrier gas. The products were isolated from the reaction mixture by column chromatography on silica gel 60, 0.063-0.2 mm, 70-230 mesh (Merck).

General Procedure for the optimization and the scope

A 4 mL screw-cap glass vial was charged with palladium salt, ligand, solid substrates and an oven-dried stirring bar under air. The vial was closed by PTFE/white rubber septa and phenolic cap and connected with atmosphere with a needle. Then the vial was evacuated under vacuum and recharged with argon for three times. Then, toluene (2 ml) is added via syringe, followed by the addition of isoprene and amide (in case of liquid) by micro syringe. The vial was fixed in an alloy plate and put into Paar 4560 series autoclave (300 mL) under argon atmosphere. At room temperature, the fixed autoclave is washed with carbon monoxide for three times before 40 bar of carbon monoxide was charged. The autoclave was placed on a heating plate equipped with magnetic stirring and an aluminum block. The reaction is allowed to heat at 100°C for 20 hours. After the reaction finishes, the autoclave is cooled to room temperature. The CO pressure was carefully released. Isooctane (100 µL) was added in to the reaction mixture as internal standard. The mixture was analyzed by GC. After removal of solvent

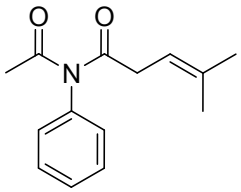
under reduced pressure, pure product was obtained by column chromatography on silica gel (eluent: pentane/ethyl acetate = 10:1).

General Procedure for Reactions With Butadiene

In a 25 mL HASTELLOY® autoclave equipped with thermometer sensor, a pressure sensor and a mechanical stirring bar, Palladium, Xantphos were added. Then the autoclave was closed. Next, the autoclave was evacuated under vacuum and recharged with argon under schlenk line for three times. Then autoclave is again opened and connected to argon flow. After toluene (10 ml) and amide were injected by syringe under argon flow, the autoclave is closed again. Afterwards, the autoclave is cooled down with an acetone-dry ice bath and about 1.1 g of 1.3-butadiene is slowed added into the autoclave while stirring (checked by balance). Further, after the autoclave is warmed up to room temperature, carbon monoxide is purged into the autoclave up to 40 bar. The reaction is allowed to be heated in a heating block to certain temperature for 20 hours. The gas consumption was monitored by Paar 4848 gas controller. After the reaction, the autoclave was cooled to room temperature and the pressure was carefully released. The mixture was diluted with acetone and analysed by gas chromatography. Pure product was obtained by column chromatography over silica gel (eluent: pentane/ethyl acetate = 10:1).

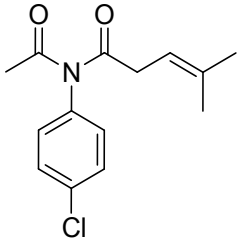
Characterization Data

N-acetyl-4-methyl-N-phenylpent-3-enamide



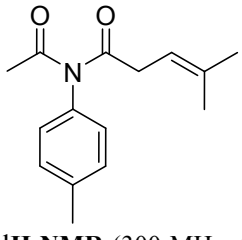
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.59 – 7.35 (m, 3H), 7.24 – 7.00 (m, 2H), 5.27 (tt, *J* = 6.9, 1.4 Hz, 1H), 3.18 (d, *J* = 6.9 Hz, 2H), 2.37 (s, 3H), 1.72 (d, *J* = 1.3 Hz, 3H), 1.48 (s, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 174.6, 173.2, 139.1, 135.9, 129.8, 128.9, 128.8, 115.8, 37.9, 27.2, 25.7, 18.1.
HRMS (EI): Calcd. for $C_{14}H_{17}O_2N_1$ [M]+: 231.12538, Found: 231.12593.

N-acetyl-N-(4-chlorophenyl)-4-methylpent-3-enamide



^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.57 – 7.35 (m, 2H), 7.16 – 6.93 (m, 2H), 5.32 – 5.14 (m, 1H), 3.17 (d, *J* = 6.9 Hz, 2H), 2.37 (s, 3H), 1.85 – 1.64 (m, 3H), 1.49 (s, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 174.26, 172.98, 137.53, 136.18, 134.88, 130.28, 130.00, 115.56, 37.89, 27.11, 25.72, 18.10.
HRMS (EI): Calcd. for $C_{14}H_{16}O_2Cl_1N_1$ [M]+:265.08641; Found:265.08674. Calcd. for $C_{14}H_{16}O_2Cl_1N_1$ [M]+:267.08346; Found:267.08412.

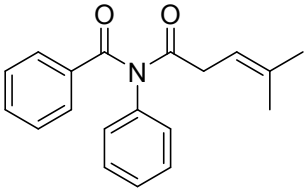
N-acetyl-4-methyl-N-(p-tolyl)pent-3-enamide



^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.23 (d, *J* = 8.0 Hz, 2H), 7.05 – 6.90 (m, 2H), 5.26 (tt, *J* = 7.0, 1.5 Hz, 1H), 3.16 (d, *J* = 6.9 Hz, 2H), 2.36 (s, 3H), 2.34 (s, 3H), 1.72 – 1.67 (m, 3H), 1.47 (s, 3H).

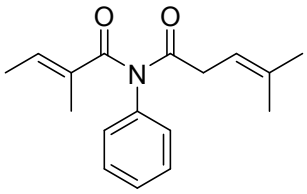
^{13}C NMR (75 MHz, $CDCl_3$) δ = 174.6, 173.3, 138.7, 136.4, 135.6, 130.4, 128.5, 116.0, 37.8, 27.1, 25.7, 21.2, 18.1.
HRMS (EI): Calcd. for $C_{15}H_{19}O_2N_1$ [M]+:245.14103; Found:245.14145.

N-(4-methylpent-3-enoyl)-*N*-phenylbenzamide



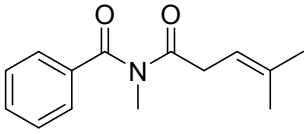
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.59 – 7.48 (m, 2H), 7.38 – 7.14 (m, 6H), 7.10 – 7.01 (m, 2H), 5.35 – 5.22 (m, 1H), 3.32 (ddt, *J* = 8.3, 2.2, 1.1 Hz, 2H), 1.63 (d, *J* = 1.4 Hz, 3H), 1.47 (s, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 175.5, 173.0, 139.1, 136.4, 135.0, 132.1, 129.4, 129.1, 128.5, 128.3, 128.1, 116.2, 37.2, 25.7, 18.2.
HRMS (EI): Calcd. for $C_{19}H_{19}O_2N_1$ [M]+: 293.14103 ; Found: 293.14104 .

4-Methyl-N-(2-methylbut-2-enoyl)-N-phenylpent-3-enamide



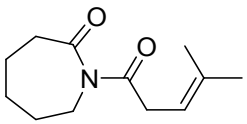
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.37 – 7.23 (m, 2H), 7.28 – 7.17 (m, 1H), 7.09 – 6.97 (m, 2H), 6.37 – 6.20 (m, 1H), 5.22 (dddt, *J* = 7.1, 5.7, 2.9, 1.4 Hz, 1H), 3.28 – 3.00 (m, 2H), 1.72 – 1.63 (m, 3H), 1.68 – 1.60 (m, 3H), 1.65 – 1.56 (m, 3H), 1.45 (s, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 175.1, 174.9, 139.1, 136.02, 135.95, 133.8, 129.3, 128.1, 127.9, 116.3, 36.9, 25.7, 18.1, 14.2, 13.0.
HRMS (EI): Calcd. for $C_{17}H_{21}O_2N_1$ [M]+: 271.15668 ; Found: 271.15630.

N-methyl-N-(4-methylpent-3-enoyl)benzamide



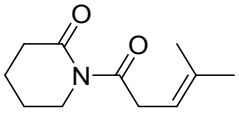
^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.63 – 7.57 (m, 2H), 7.56 – 7.49 (m, 1H), 7.48 – 7.40 (m, 2H), 5.38 – 5.21 (m, 1H), 3.34 (d, *J* = 6.9 Hz, 2H), 3.18 (s, 3H), 1.68 (s, 3H), 1.52 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ = 175.6, 174.1, 135.7, 135.3, 132.4, 128.8, 128.5, 116.5, 37.3, 34.4, 25.7, 18.1.
HRMS (EI): Calcd. for $C_{14}H_{17}O_2N_1$ [M]+:231.12538; Found:231.12601.

1-(4-Methylpent-3-enoyl)azepan-2-one



^{1}H NMR (300 MHz, $CDCl_3$) δ = 5.38 – 5.26 (m, 1H), 3.91 – 3.82 (m, 2H), 3.64 – 3.47 (m, 2H), 2.78 – 2.64 (m, 2H), 1.85 – 1.48 (m, 12H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 177.7, 174.9, 135.0, 116.9, 43.4, 39.9, 38.5, 29.2, 28.5, 25.8, 23.7, 18.2.
HRMS (EI): Calcd. for $C_{11}H_{17}O_2N_1$ [M]+: 195.12527; Found:195.12538.

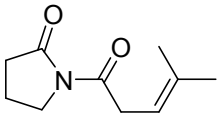
1-(4-Methylpent-3-enoyl)piperidin-2-one



^{1}H NMR (300 MHz, $CDCl_3$) δ = 5.42 – 5.24 (m, 1H), 3.67 (t, 2H), 3.58 (d, *J* = 7.0 Hz, 2H), 2.60 – 2.44 (m, 2H), 1.85 – 1.72 (m, 4H), 1.72 – 1.65 (m, 3H), 1.59 (s, 3H).

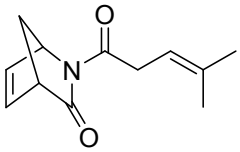
^{13}C NMR (75 MHz, $CDCl_3$) δ = 175.6, 173.3, 134.9, 116.8, 44.0, 38.9, 34.9, 25.7, 22.4, 20.3, 18.1.
HRMS (EI): Calcd. for $C_{11}H_{17}O_2N_1$ [M]+:195.12538; Found:195.12491 .

1-(4-Methylpent-3-enoyl)pyrrolidin-2-one



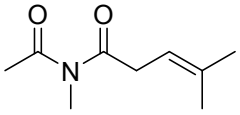
^{1}H NMR (300 MHz, $CDCl_3$) δ = 5.46 – 5.25 (m, 1H), 3.95 – 3.70 (m, 2H), 3.67 – 3.37 (m, 2H), 2.66 – 2.35 (m, 2H), 2.15 – 1.91 (m, 2H), 1.73 (s, 3H), 1.64 (s, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 175.3, 172.9, 135.5, 115.7, 115.6, 45.6, 36.2, 33.7, 25.8, 25.7, 18.2, 18.2, 17.2.
HRMS (EI): Calcd. for $C_{10}H_{15}O_2N_1$ [M]+:181.10973; Found:181.10964.

2-(4-Methylpent-3-enoyl)-2-azabicyclo[2.2.1]hept-5-en-3-one



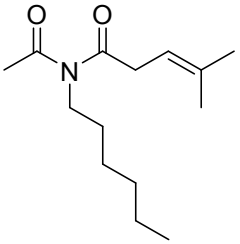
^{1}H NMR (300 MHz, $CDCl_3$) δ = δ 6.89 (ddd, *J* = 5.3, 2.4, 0.6 Hz, 1H), 6.65 (ddd, *J* = 5.2, 3.2, 1.5 Hz, 1H), 5.39 – 5.19 (m, 2H), 3.64 – 3.35 (m, 3H), 2.30 (dt, *J* = 8.6, 1.7 Hz, 1H), 2.19 (dt, *J* = 8.7, 1.5 Hz, 1H), 1.84 – 1.67 (m, 3H), 1.63 (s, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 177.5, 171.6, 140.5, 138.1, 135.6, 115.6, 60.4, 54.7, 54.7, 35.4, 25.8, 18.2.
HRMS (EI): Calcd. for $C_{12}H_{15}O_2N_1$ [M]+: 205.10973; Found: 205.10955 .

N-acetyl-N,4-dimethylpent-3-enamide



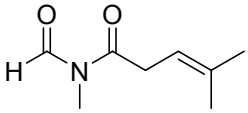
^{1}H NMR (300 MHz, $CDCl_3$) δ = 5.38 – 5.19 (m, 1H), 3.36 (d, *J* = 6.8 Hz, 2H), 3.18 (s, 3H), 2.38 (s, 3H), 1.72 (d, *J* = 1.4 Hz, 3H), 1.61 (s, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 174.8, 173.4, 135.8, 115.8, 37.5, 31.5, 26.8, 25.7, 18.1.
HRMS (EI): Calcd. for $C_9H_{15}O_2N_1$ [M]+:169.10973; Found:169.10987.

N-acetyl-N-hexyl-4-methylpent-3-enamide



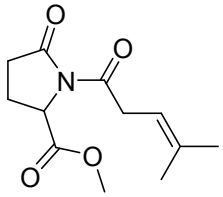
^{1}H NMR (300 MHz, $CDCl_3$) δ 5.30 (t, *J* = 6.9 Hz, 1H), 3.60 (t, *J* = 7.9 Hz, 2H), 3.37 (d, *J* = 6.9 Hz, 2H), 2.37 (s, 3H), 1.73 (s, 3H), 1.62 (s, 3H), 1.59 – 1.43 (m, 2H), 1.35 – 1.19 (m, 6H), 0.85 (t, *J* = 5.7 Hz, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 174.9, 173.3, 135.6, 116.2, 44.7, 37.4, 31.4, 29.3, 26.6, 26.6, 25.7, 22.5, 18.1, 14.0.
HRMS (EI): Calcd. for $C_{14}H_{25}O_2N1$ [M]+: 239.18798 ; Found: 239.18811 .

N-formyl-N,4-dimethylpent-3-enamide



^{1}H NMR (400 MHz, $CDCl_3$) δ = 9.15 (s, 1H), 5.31 – 5.13 (m, 1H), 3.31 (d, *J* = 6.7 Hz, 1H), 3.04 (s, 3H), 1.70 (s, 3H), 1.61 (s, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ = 173.3, 162.7, 137.0, 114.5, 35.0, 25.6, 18.1.
HRMS (EI): Calcd. for $C_8H_{13}O_2N_1$ [M]+:155.09408; Found:155.09387 .

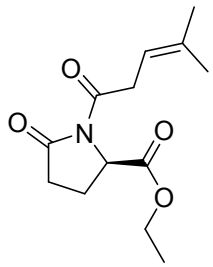
N-formyl-N,4-dimethylpent-3-enamide



^{1}H NMR (300 MHz, $CDCl_3$) δ = 5.35 – 5.17 (m, 1H), 4.68 (dd, *J* = 9.5, 2.8 Hz, 1H), 3.69 (d, *J* = 2.9 Hz, 3H), 3.63 – 3.51 (m, 2H), 2.82 – 2.41 (m, 2H), 2.39 – 2.17 (m, 1H), 2.15 – 1.93 (m, 1H), 1.67 (s, 3H), 1.58 (s, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 174.3, 172.6, 171.5, 135.7, 115.1, 57.9, 52.6, 36.0, 31.9, 25.7, 21.3, 18.1.

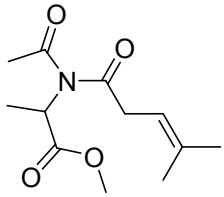
HRMS (EI): Calcd. for $C_{12}H_{17}O_4N_1$ [M]+: 239.11521; Found: 239.11477.

Ethyl (R)-1-(4-methylpent-3-enoyl)-5-oxopyrrolidine-2-carboxylate



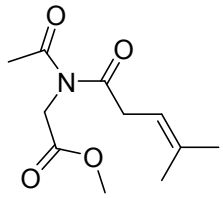
^{1}H NMR (300 MHz, $CDCl_3$) δ = 5.41 – 5.24 (m, 1H), 4.69 (dd, *J* = 9.5, 2.7 Hz, 1H), 4.17 (q, *J* = 7.1 Hz, 2H), 3.62 (d, *J* = 6.8 Hz, 2H), 2.69 (dt, *J* = 17.8, 10.0 Hz, 1H), 2.59 – 2.45 (m, 1H), 2.41 – 2.20 (m, 1H), 2.11 – 1.94 (m, 1H), 1.70 (s, 3H), 1.60 (s, 3H), 1.23 (t, *J* = 7.2 Hz, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 174.3, 172.6, 171.0, 135.8, 115.2, 61.6, 58.1, 36.0, 32.0, 25.7, 21.3, 18.2, 14.0.
HRMS (EI): Calcd. for $C_{13}H_{19}O_4N_1$ [M]+: 253.13086 ; Found: 253.13081 .

Methyl *N*-acetyl-*N*-(4-methylpent-3-enoyl)alaninate



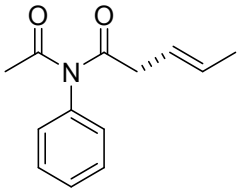
^{1}H NMR (300 MHz, $CDCl_3$) δ = δ 5.38 – 5.18 (m, 1H), 4.51 (q, *J* = 6.9 Hz, 1H), 3.67 (s, 3H), 3.41 – 3.27 (m, 2H), 2.37 (s, 3H), 1.73 (d, *J* = 1.4 Hz, 3H), 1.61 (d, *J* = 1.2 Hz, 3H), 1.50 (d, *J* = 6.9 Hz, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 174.4, 172.8, 170.7, 136.3, 115.4, 54.2, 52.5, 37.7, 26.6, 25.7, 18.1, 15.5.

Methyl *N*-acetyl-N-(4-methylpent-3-enoyl)glycinate



^{1}H NMR (300 MHz, $CDCl_3$) δ = 5.35 – 5.18 (m, 1H), 4.39 (s, 2H), 3.71 (s, 3H), 3.32 (d, *J* = 6.8 Hz, 2H), 2.38 (s, 3H), 1.70 (s, 3H), 1.58 (s, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 174.1, 172.8, 169.1, 136.2, 115.4, 52.5, 45.7, 37.3, 26.3, 25.7, 18.0.
HRMS (EI): Calcd. for $C_{12}H_{17}O_4N_1$ [M]+: 239.11521 ; Found: 239.11577 .

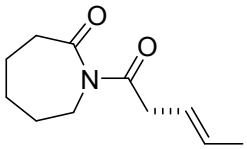
N-acetyl-N-phenylpent-3-enamide



Trans isomer:
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.55 – 7.36 (m, 3H), 7.22 – 7.07 (m, 2H), 5.91 – 5.26 (m, 2H), 3.22 – 3.10 (m, 2H), 2.36 (s, 3H), 1.74 – 1.61 (m, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 174.5 , 173.2 , 139.0 , 129.8 , 128.9 (3C) 122.8 , 42.0 , 27.2 , 18.0 .

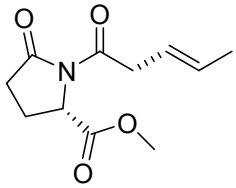
Cis isomer:
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.55 – 7.36 (m, 3H), 7.22 – 7.07 (m, 2H), 5.91 – 5.26 (m, 2H), 3.32 – 3.24 (m, 2H), 2.33 (s, 3H), 1.53 – 1.48 (m, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 174.1 , 173.2 , 139.0 , 129.8 , 128.9 (2C), 127.8 , 121.8, 36.7, 27.1, 13.1.
HRMS (EI): Calcd. for $C_{13}H_{15}O_2N_1$ [M]+: 217.10973; Found: 217.11032.

1-(Pent-3-enoyl)azepan-2-one



Trans isomer: (73%)
^{1}H NMR (300 MHz, $CDCl_3$) δ = 5.73 – 5.47 (m, 2H), 3.99 – 3.81 (m, 2H), 3.62 – 3.53 (m, 2H), 2.77 – 2.64 (m, 2H), 1.87 – 1.55 (m, 9H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 177.6 , 174.8 , 128.8 , 124.0 , 43.3 , 42.7 , 39.8 , 29.2 , 28.5 , 23.7 , 18.0 .
Cis isomer: (27%)
^{1}H NMR (300 MHz, $CDCl_3$) δ = 5.73 – 5.47 (m, 2H), 3.99 – 3.81 (m, 2H), 3.70 – 3.61 (m, 2H), 2.77 – 2.64 (m, 2H), 1.87 – 1.55 (m, 9H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 177.6 , 174.5 , 127.1 , 122.9 , 43.3 , 39.8 , 37.4 , 29.2 , 28.5 , 23.7 , 13.1 .
HRMS (EI): Calcd. for $C_{11}H_{17}O_2N_1$ [M]+:195.12538 Found: 195.12527.

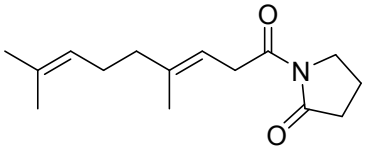
Methyl (S,E)-5-oxo-1-(pent-3-enoyl)pyrrolidine-2-carboxylate



E/Z mixture
^{1}H NMR (400 MHz, $CDCl_3$) δ = 5.73 – 5.50 (m, 2H), 4.81 – 4.68 (m, 1H), 3.75 (d, *J* = 0.8 Hz, 3H), 3.73 – 3.60 (m, 2H), 2.80 – 2.64 (m, 1H), 2.63 – 2.48 (m, 1H), 2.41 – 2.21 (m, 1H), 2.15 – 1.96 (m, 1H), 1.71 – 1.61 (m, 3H).

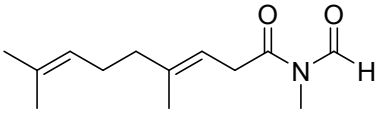
Major isomer ^{13}C NMR (101 MHz, $CDCl_3$) δ = 174.3 , 172.7 , 171.6 , 129.8 , 122.2 , 57.9 , 52.7 , 40.2 , 32.0 , 21.4 , 18.1 .
Minor Isomer ^{13}C NMR (101 MHz, $CDCl_3$) δ = 174.3 , 172.2 , 171.6 , 127.9 , 121.1 , 57.9 , 52.7 , 34.9 , 32.0 , 21.4 , 13.2 .
HRMS (EI): Calcd. for $C_{11}H_{15}O_4N_1$ [M]+:225.09956 Found: 225.09956.

1-(4,8-Dimethylnona-3,7-dienoyl)pyrrolidin-2-one



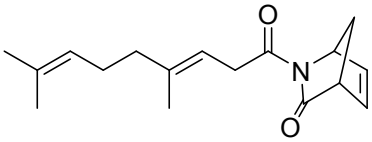
^{1}H NMR (300 MHz, $CDCl_3$) δ = 5.41 – 5.26 (m, 1H), 5.14 – 4.91 (m, 1H), 3.85 – 3.67 (m, 2H), 3.57 (d, *J* = 6.9 Hz, 2H), 2.54 (t, *J* = 8.1 Hz, 2H), 2.15 – 1.88 (m, 6H), 1.78 – 1.55 (m, 6H), 1.55 – 1.48 (m, 3H).
Major Isomer:
^{13}C NMR (75 MHz, $CDCl_3$) δ = 175.2, 172.7, 138.9, 131.4, 124.0, 115.5, 45.5, 39.6, 36.1, 33.7, 26.5, 25.7, 17.6, 17.2, 16.6.
Minor Isomer:
^{13}C NMR (75 MHz, $CDCl_3$) δ = 175.2, 172.8, 139.0, 131.7, 124.0, 116.3, 45.5, 39.6, 35.9, 32.3, 26.3, 23.5, 17.6, 17.2, 16.6.
HRMS (EI): Calcd. for $C_{15}H_{23}O_2N_1$ [M]+: 249.17233 ; Found: 249.17250 .

N-formyl-N,4,8-trimethylnona-3,7-dienamide



^{1}H NMR (300 MHz, $CDCl_3$) δ = 9.19 (s, 1H), 5.35 – 5.21 (m, 2H), 5.13 – 4.99 (m, 1H), 3.35 (d, *J* = 6.9 Hz, 3H), 3.18 – 3.02 (m, 5H), 2.05 (s, 3H), 1.75 (q, *J* = 1.3 Hz, 5H), 1.66 (s, 4H), 1.59 (s, 5H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 173.3, 162.7, 140.7, 132.4, 123.5, 115.0, 34.8, 32.3, 26.6, 26.1, 25.7, 23.4, 17.7.
HRMS (EI): Calcd. for $C_{13}H_{21}O_2N_1$ [M]+: 223.15668 ; Found: 223.15705 .

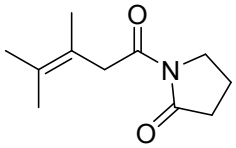
2-(4,8-Dimethylnona-3,7-dienoyl)-2-azabicyclo[2.2.1]hept-5-en-3-one



^{1}H NMR (300 MHz, $CDCl_3$) δ = 6.99 – 6.80 (m, 1H), 6.71 – 6.55 (m, 1H), 5.41 – 5.30 (m, 1H), 5.29 – 5.22 (m, 0H), 5.13 – 5.01 (m, 1H), 3.58 – 3.45 (m, 2H), 3.45 – 3.36 (m, 1H), 2.35 – 2.16 (m, 2H), 2.12 – 1.95 (m, 4H), 1.77 – 1.54 (m, 10H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 177.4, 171.5, 140.5, 139.2, 139.2, 138.1, 131.8, 131.5, 124.1, 124.0, 116.2, 115.4, 60.4, 54.7, 54.7, 39.7, 35.4, 35.1, 32.3, 26.6, 26.4, 25.7, 23.5, 17.7, 17.7, 16.6. (mixture of cis 31% and trans 69%)

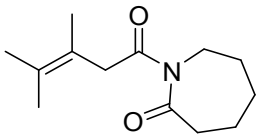
HRMS (EI): Calcd. for $C_{17}H_{23}O_2N_1$ [M]+: 273.17233; Found: 273.17228.

1-(3,4-Dimethylpent-3-enoyl)pyrrolidin-2-one



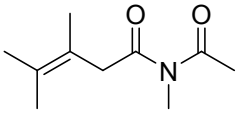
^{1}H NMR (300 MHz, $CDCl_3$) δ = ^{1}H NMR (300 MHz, Chloroform-*d*) δ 3.80 (t, *J* = 7.1 Hz, 2H), 3.65 (s, 2H), 2.59 (t, *J* = 8.1 Hz, 2H), 2.13 – 1.95 (m, 2H), 1.69 (s, 3H), 1.67 – 1.63 (m, 3H), 1.64 – 1.57 (m, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 175.5, 172.7, 128.4, 120.8, 45.6, 42.2, 33.8, 20.7, 20.6, 17.2.
HRMS (EI): Calcd. for $C_{11}H_{17}O_2N_1$ [M]+: 195.12538 ; Found: 195.12543 .

1-(3,4-Dimethylpent-3-enoyl)azepan-2-one



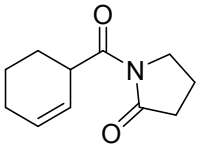
^{1}H NMR (300 MHz, $CDCl_3$) δ = 3.86 – 3.68 (m, 2H), 3.51 (s, 2H), 2.70 – 2.48 (m, 2H), 1.81 – 1.44 (m, 15H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 177.7, 174.7, 127.7, 122.0, 44.3, 43.3, 39.7, 29.1, 28.6, 23.7, 20.6, 20.5, 19.2.

N-acetyl-N,3,4-trimethylpent-3-enamide



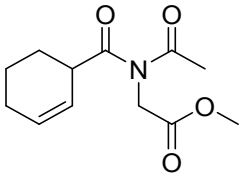
^{1}H NMR (300 MHz, $CDCl_3$) δ = δ 3.37 (s, 2H), 3.16 (s, 3H), 2.37 (s, 3H), 1.67 (s, 3H), 1.63 (s, 3H), 1.59 (s, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = ^{13}C NMR (75 MHz, $CDCl_3$) δ = 174.6, 173.4, 128.6, 120.8, 43.4, 31.4, 26.8, 20.6, 18.9.
HRMS (EI): Calcd. for $C_{10}H_{17}O_2N_1$ [M]+:183.12538; Found:183.12483 .

N-acetyl-N,3,4-trimethylpent-3-enamide



^{1}H NMR (300 MHz, $CDCl_3$) δ =5.98 – 5.78 (m, 1H), 5.73 – 5.56 (m, 1H), 4.32 – 4.18 (m, 1H), 3.79 (t, *J* = 7.1 Hz, 2H), 2.59 (t, *J* = 8.1 Hz, 2H), 2.35 – 1.49 (m, 8H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 175.9, 174.9, 129.7, 124.3, 46.0, 41.6, 34.0, 25.4, 24.6, 20.8, 17.2.

Methyl *N*-acetyl-*N*-(cyclohex-2-ene-1-carbonyl)glycinate



^{1}H NMR (300 MHz, $CDCl_3$) δ = 5.94 – 5.76 (m, 1H), 5.66 – 5.45 (m, 1H), 4.39 (s, 2H), 3.70 (s, 3H),3.75-3.65 (m, 1H), 2.33 (s, 3H), 2.22 – 1.48 (m, 6H).
^{13}C NMR (75 MHz, $CDCl_3$) δ = 177.5, 172.9, 169.2, 130.4, 123.8, 52.6, 46.1, 42.7, 26.2, 25.7, 24.6, 20.6.

Chiral HPLC Spectrum for Compound 3aq

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                                                  Inj Volume : 0.2 µl
Acq. Method     : C:\CHEM32\2\METHODS\FISCHER.M
Last changed    : 8/14/2014 12:42:11 PM
                  (modified after loading)
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Last changed    : 8/14/2014 12:46:13 PM
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Method Info     : Cellulose 2, Heptan/Isoprop 95:5,  Fluß: 1ml/min

Additional Info : Peak(s) manually integrated
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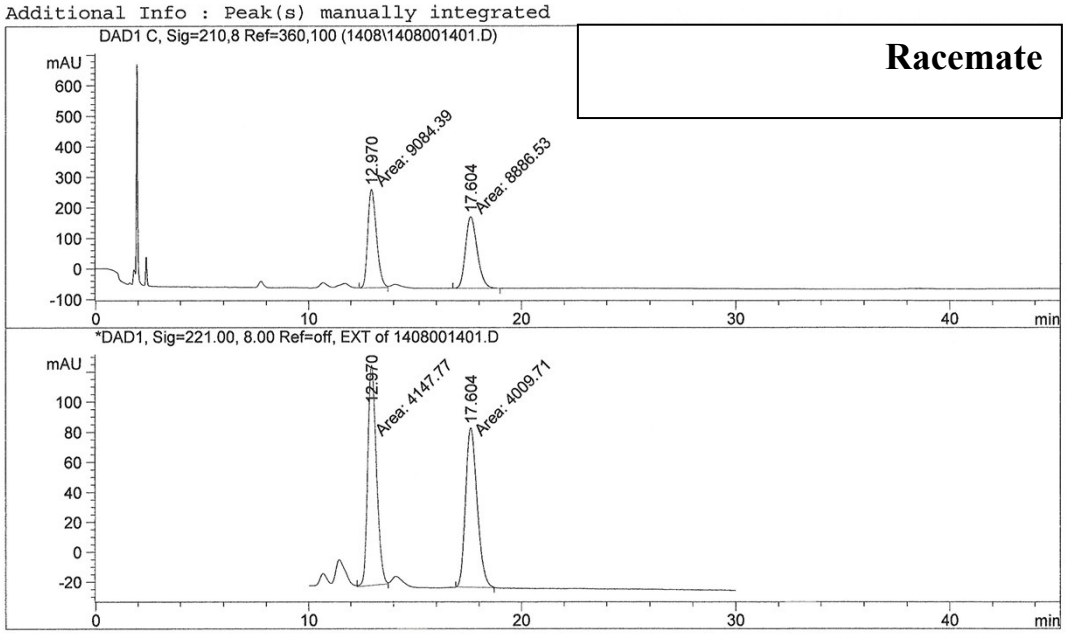
Racemate

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==========================================================================
                        Area Percent Report
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Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
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Totals :                  1.79709e4   556.98082
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50,49
49,51

Data File D:\HPCHEM\2\DATA\1408\1408001403.D
Sample Name: LHQ784B

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Injection Date  : 8/14/2014 1:24:26 PM                  Inj :   1
                                                  Inj Volume : 0.2 µl
Acq. Method     : C:\CHEM32\2\METHODS\FISCHER.M
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                  (modified after loading)
Analysis Method : C:\CHEM32\2\METHODS\FISCHER.M
Last changed    : 8/14/2014 2:31:35 PM
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Method Info     : Cellulose 2, Heptan/Isoprop 95:5,  Fluß: 1ml/min

Additional Info : Peak(s) manually integrated
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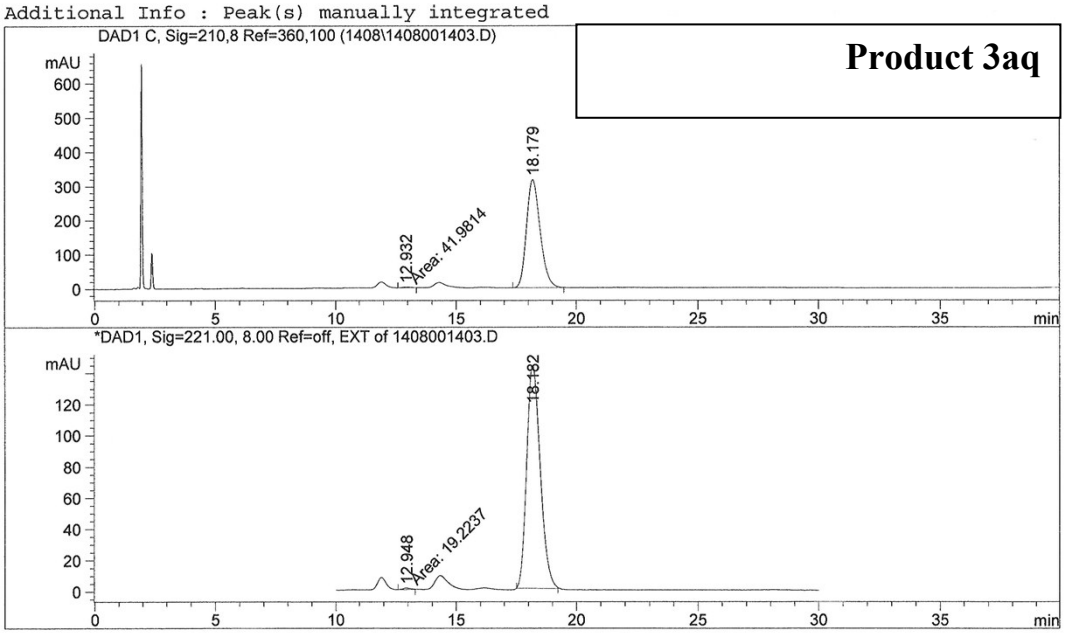
Product 3aq

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Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
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   2  18.179 BB    0.6130 1.24302e4   315.94415  99.6634

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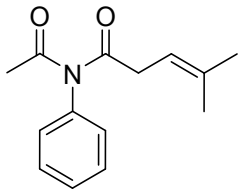
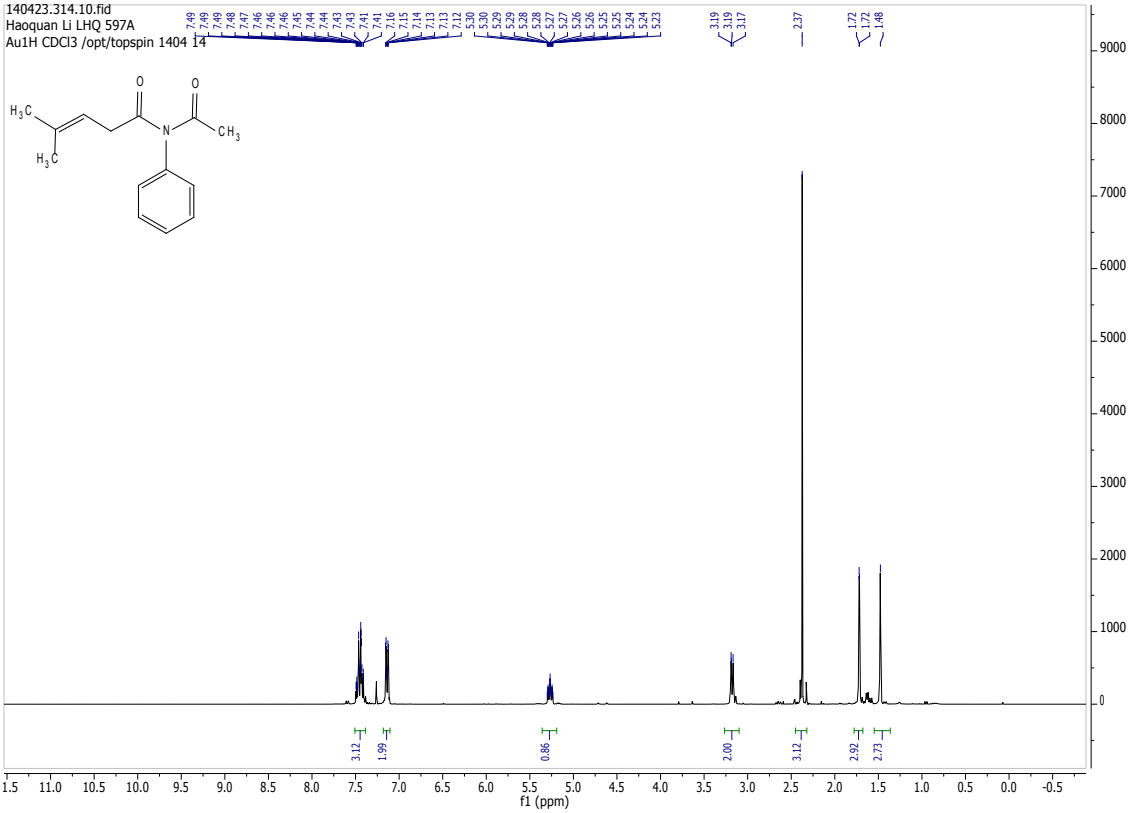
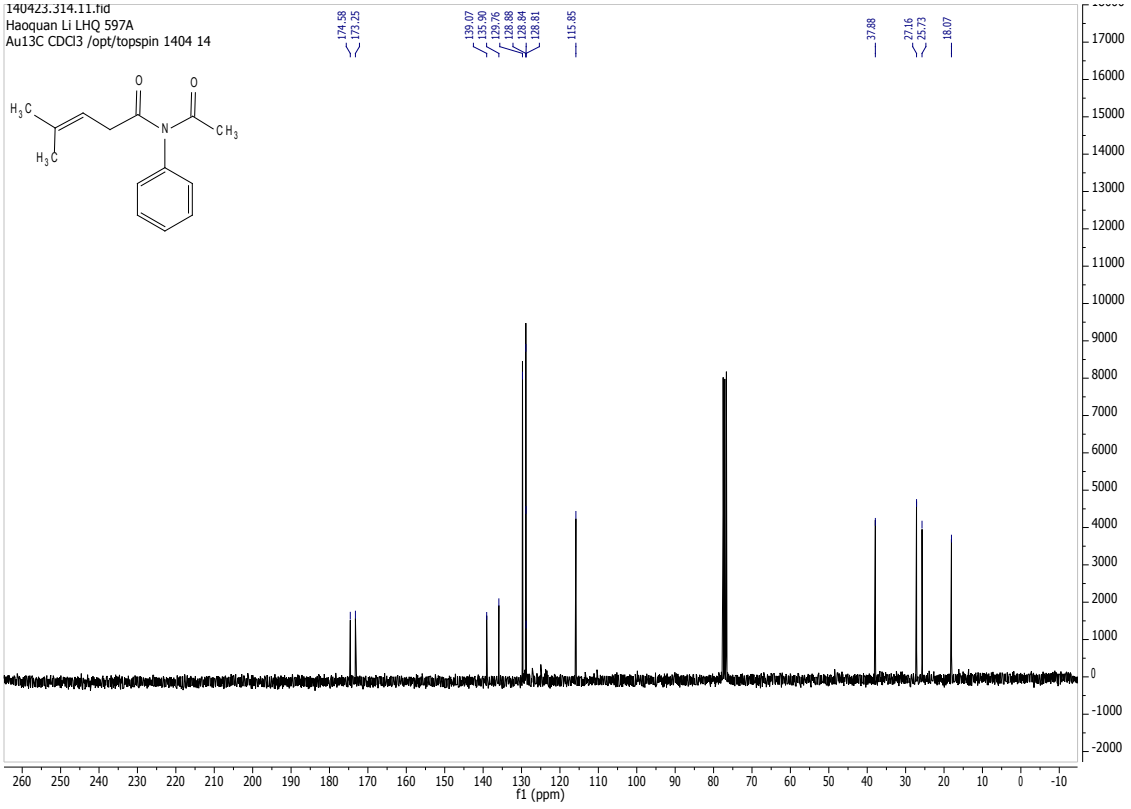
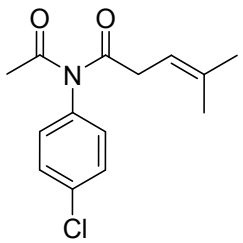
0,30
99,70

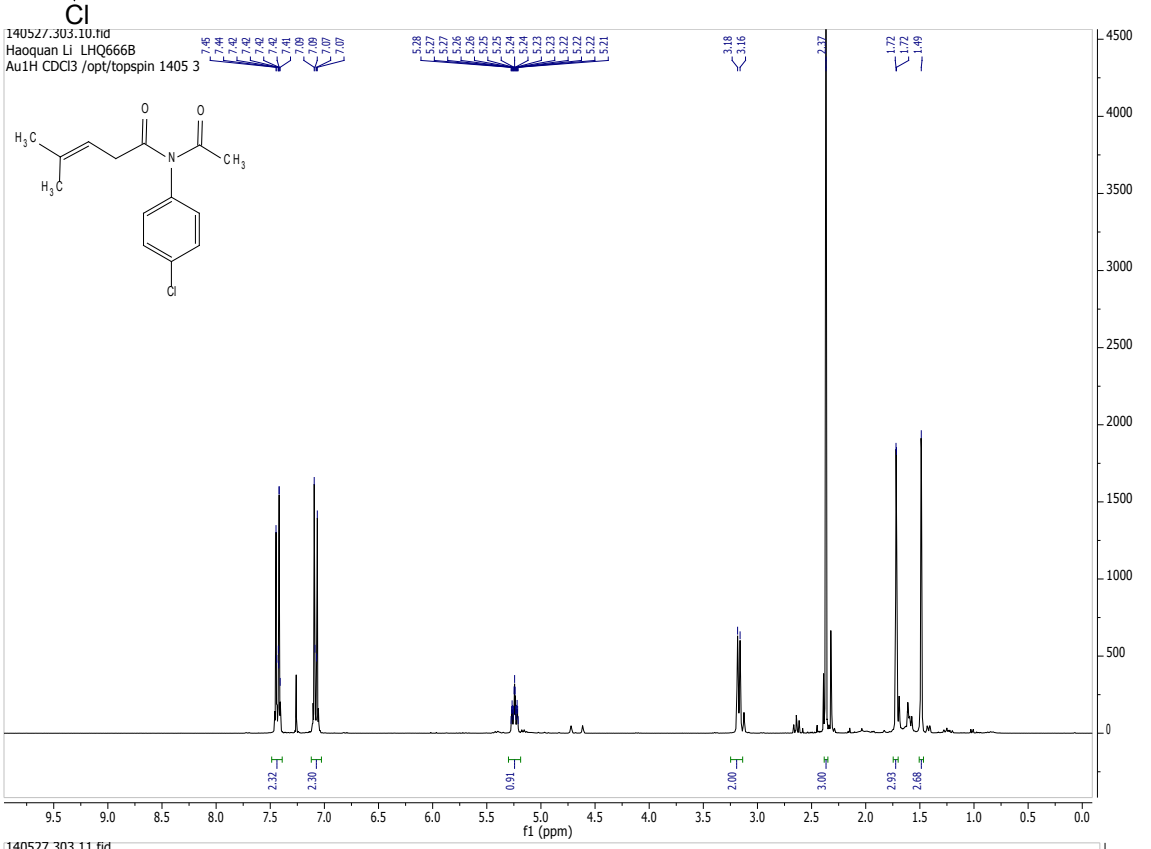
NMR Spectrum







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Haoquan Li LHQ666B
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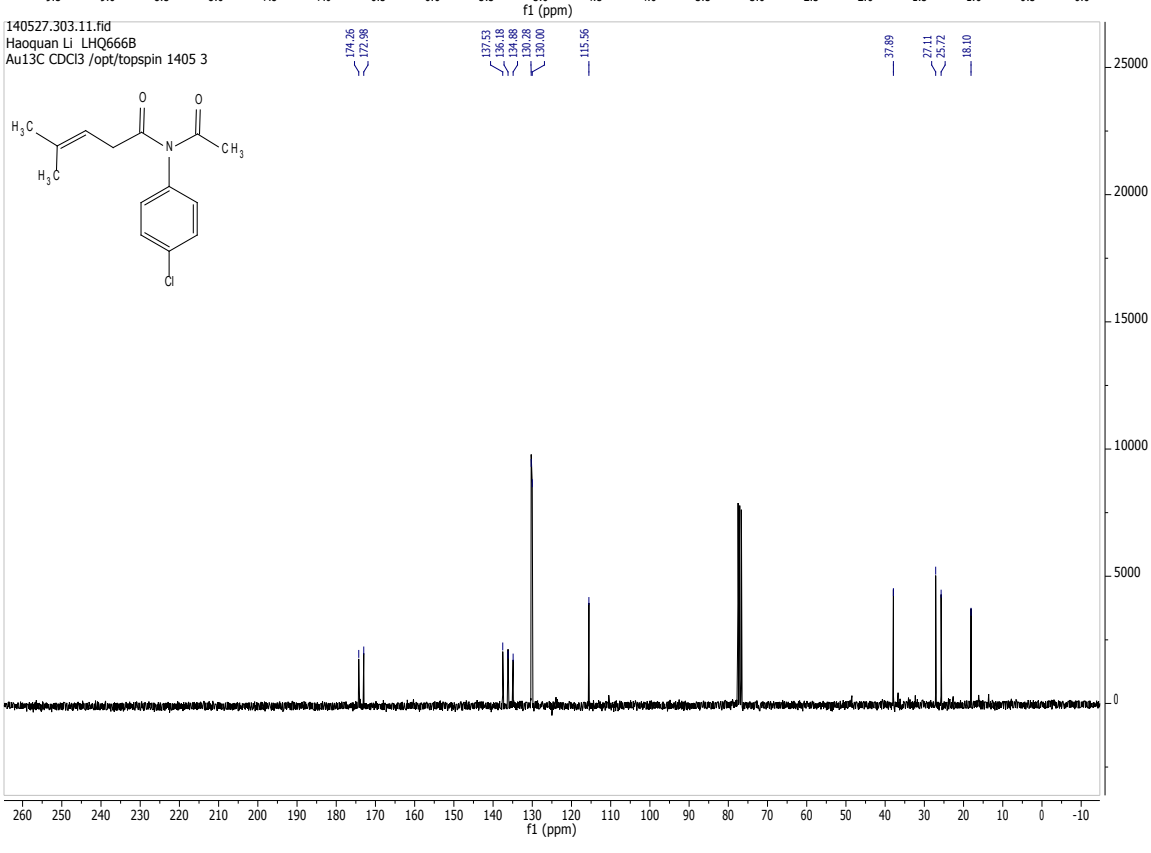
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Haoquan Li LHQ666B
Au13C CDCl3 /opt/topspin 1405 3
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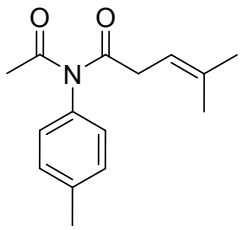

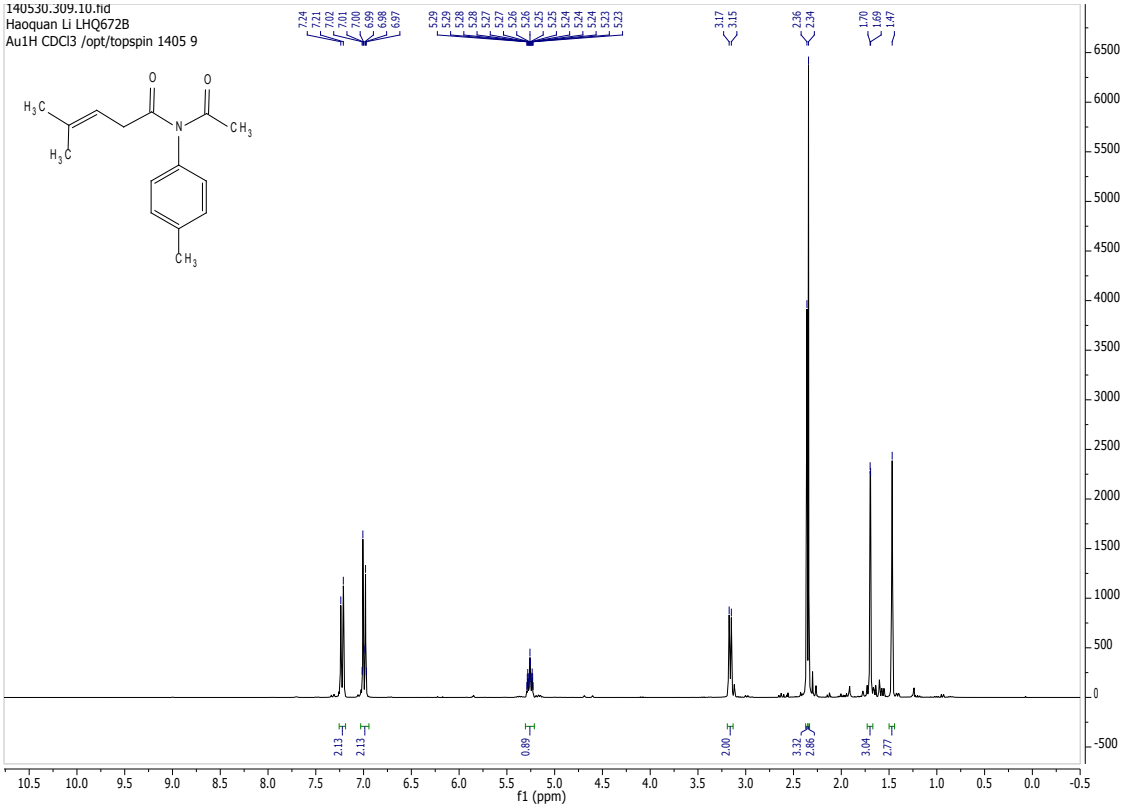
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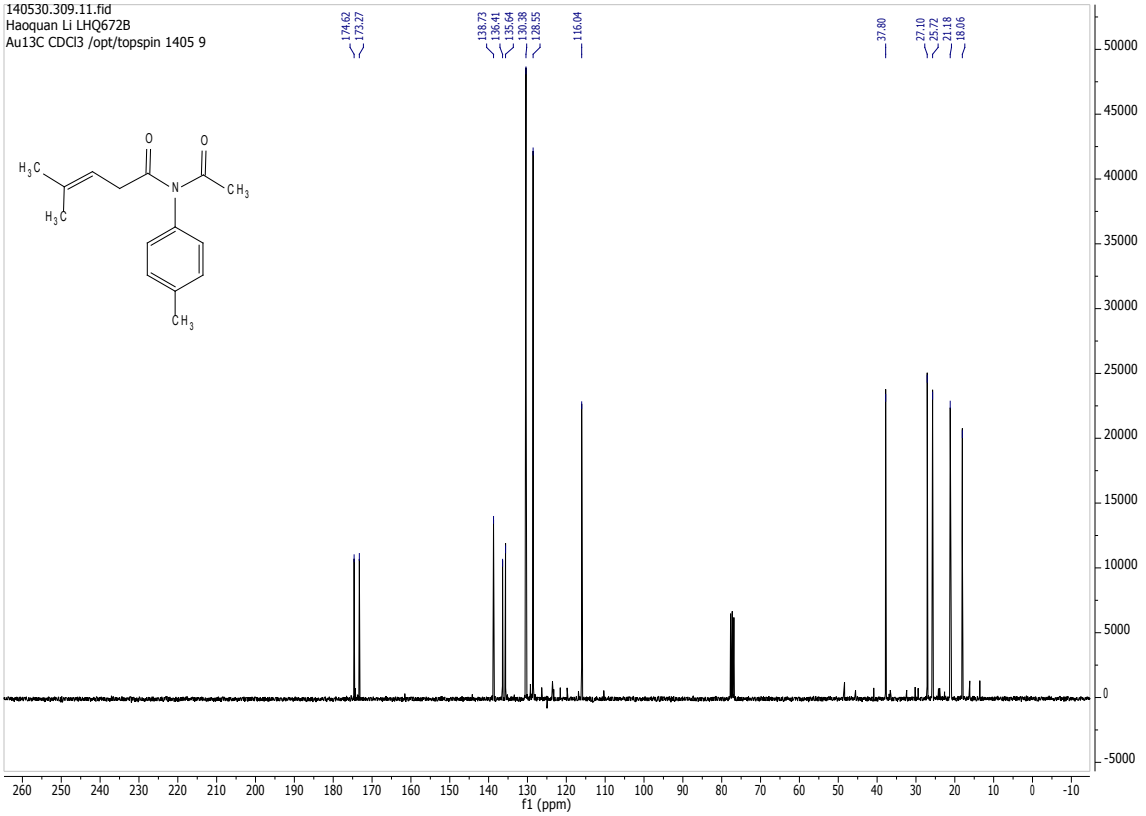
f1 (ppm)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Au13C CDCl3 /opt/topspin 1405 9
f1 (ppm)


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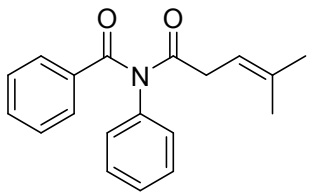
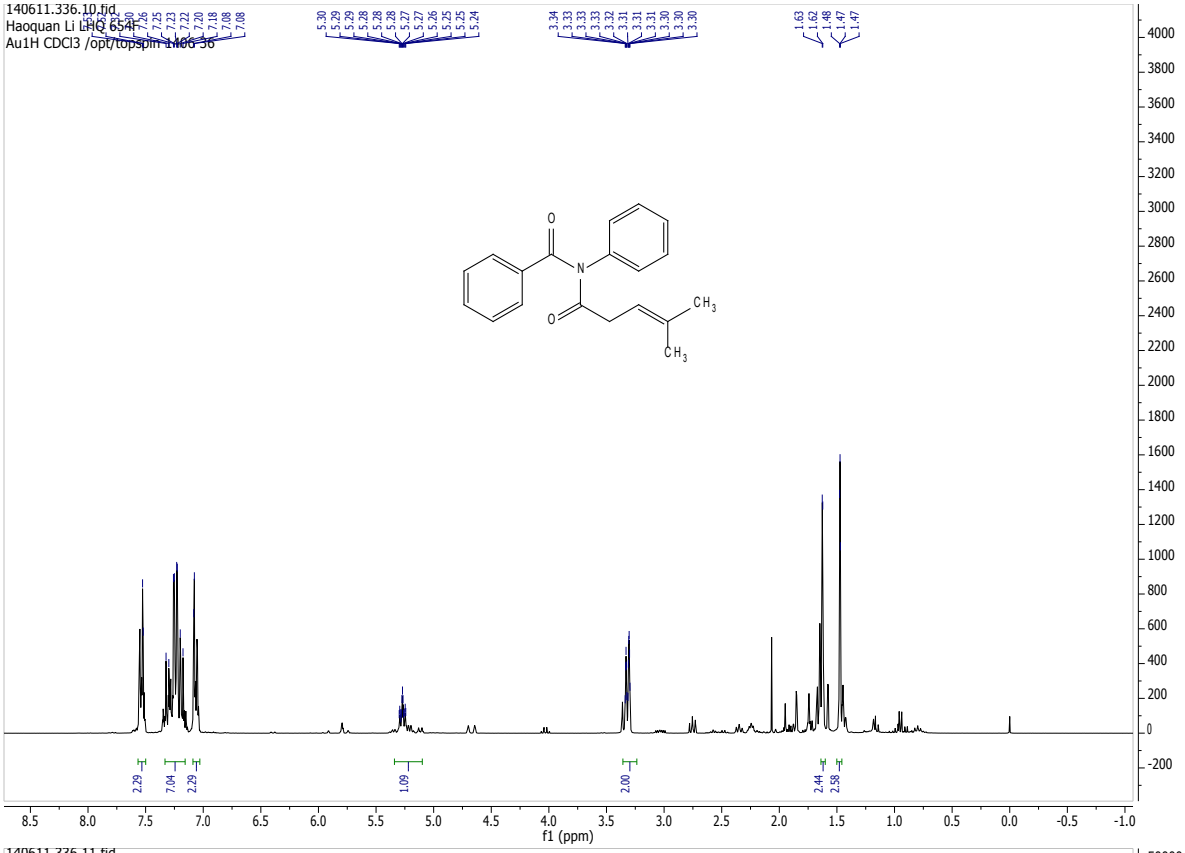
f1 (ppm)

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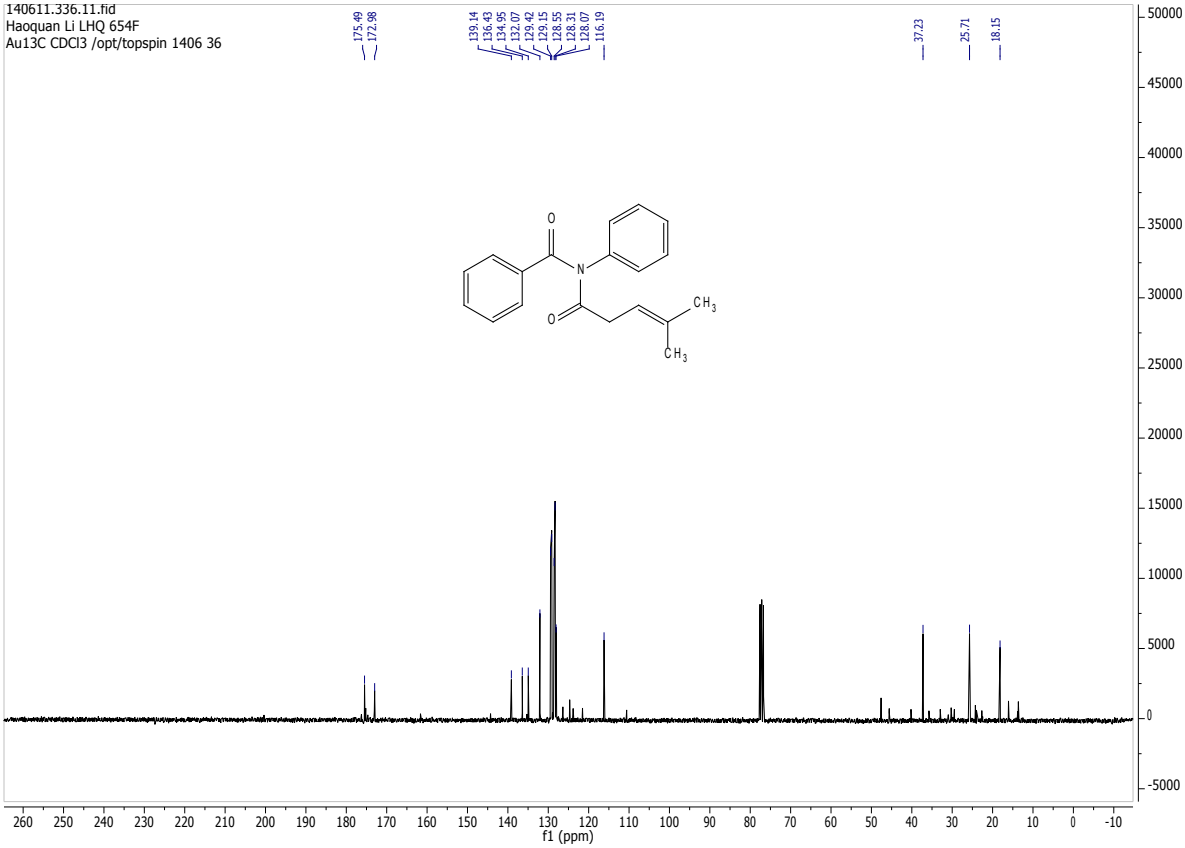
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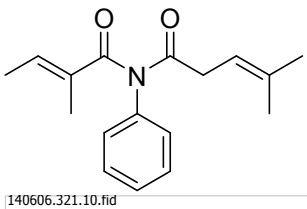
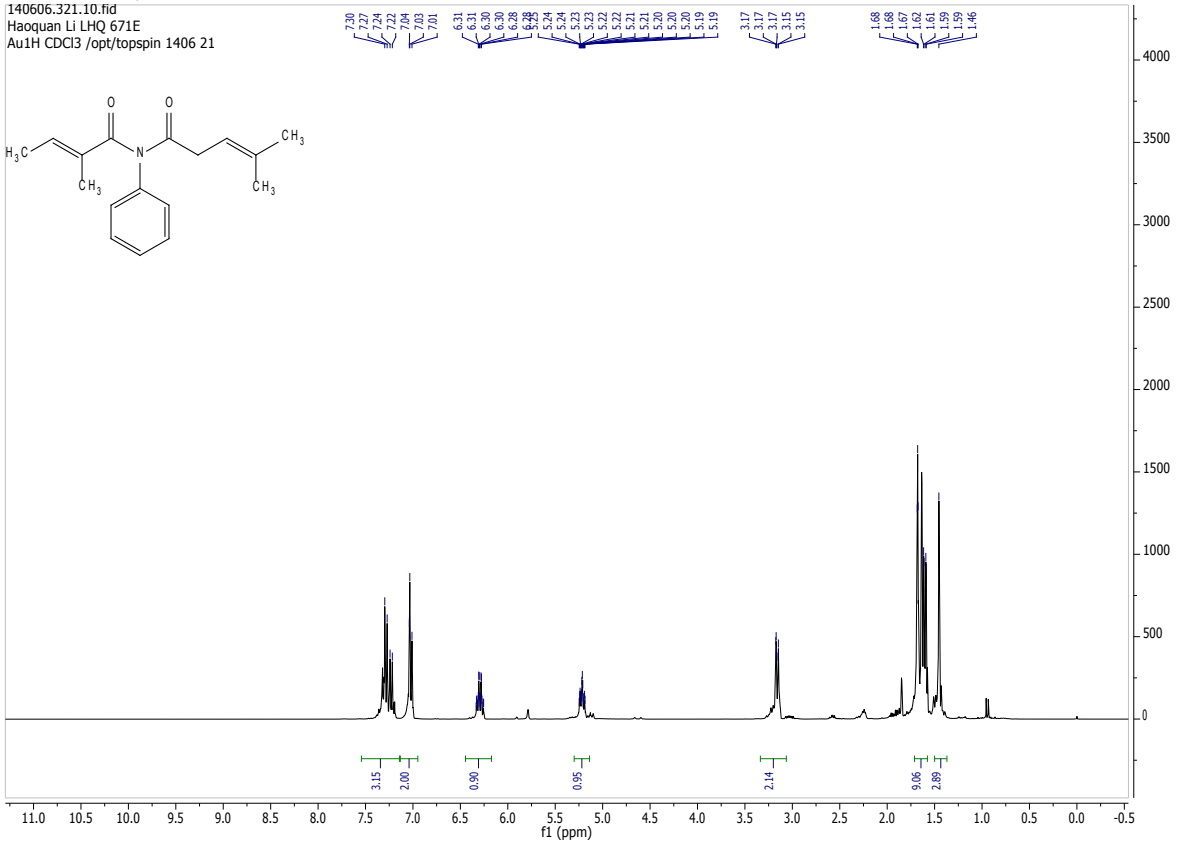
f1 (ppm)

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Haoquan Li LHQ 671E
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5.23
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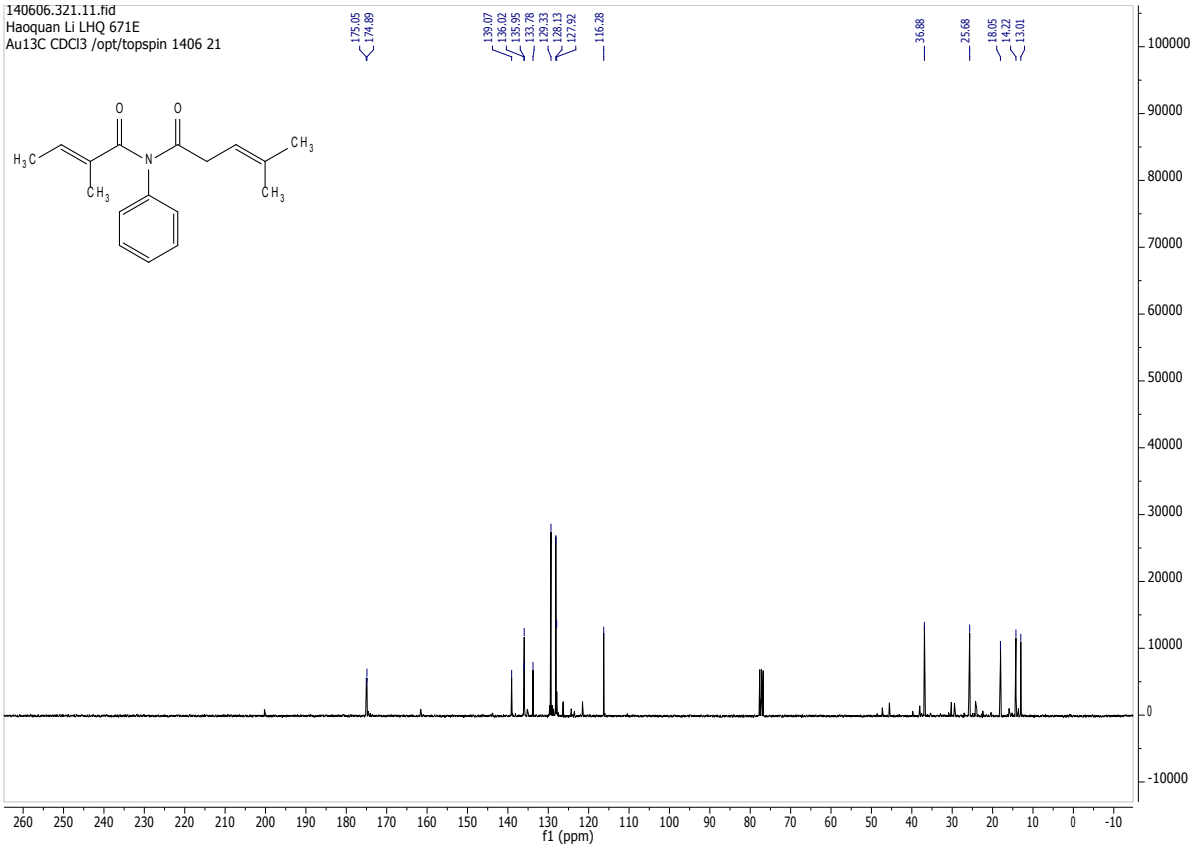
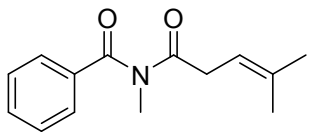
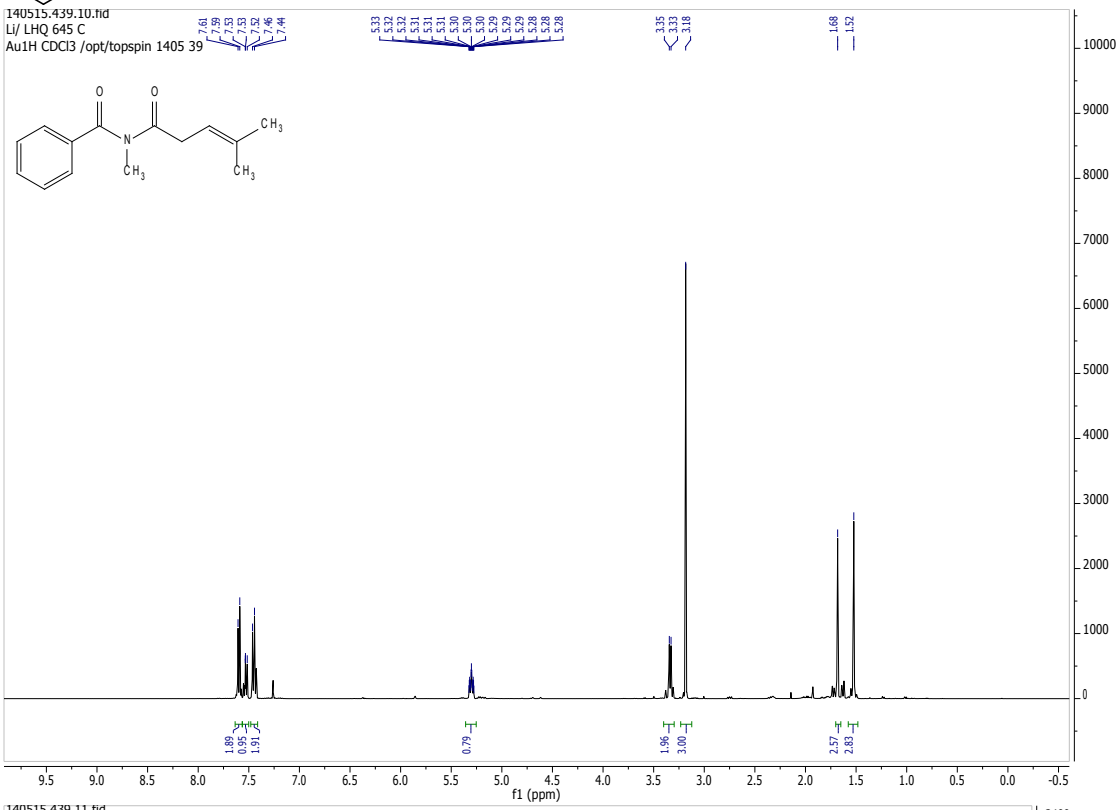
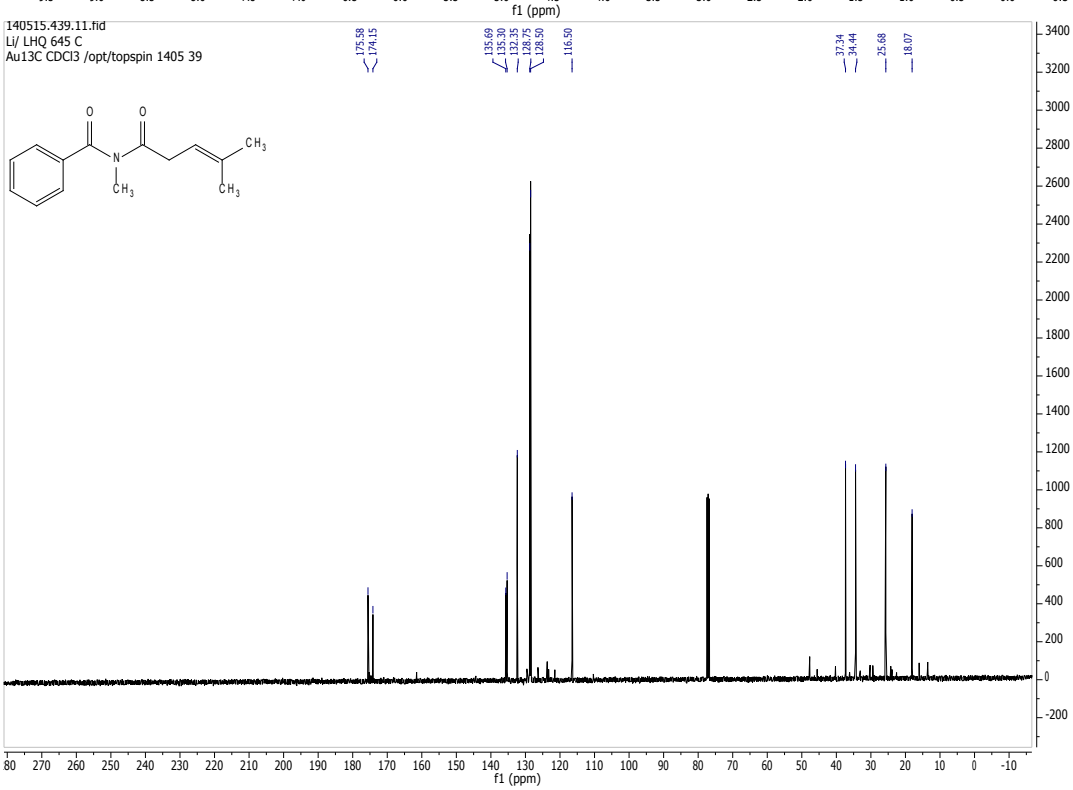
5.19
3.17
3.15
1.68
1.67
1.62
1.61
1.59
1.46
3.15
2.00
0.90
0.95
2.14
9.06
2.89
f1 (ppm)


140606.321.11.fid
Haoquan Li LHQ 671E
Au13C CDCl3 /opt/topspin 1406 21
175.05
174.89
139.07
136.02
135.95
133.78
129.33
128.13
127.92
116.28
36.88
25.68
18.05
14.22
13.01
f1 (ppm)




140515.439.10.fid
Li/ LHQ 645 C
Au1H CDCl3 /opt/topspin 1405 39
7.61
7.59
7.53
7.53
7.52
7.46
7.44
5.33
5.32
5.32
5.31
5.31
5.31
5.30
5.30
5.30
5.29
5.29
5.28
5.28
5.28
3.35
3.33
3.18
1.68
1.52
1.89
0.95
1.91
0.79
1.96
3.00
2.57
2.83
f1 (ppm)


140515.439.11.fid
Li/ LHQ 645 C
Au13C CDCl3 /opt/topspin 1405 39
175.58
174.15
135.69
133.30
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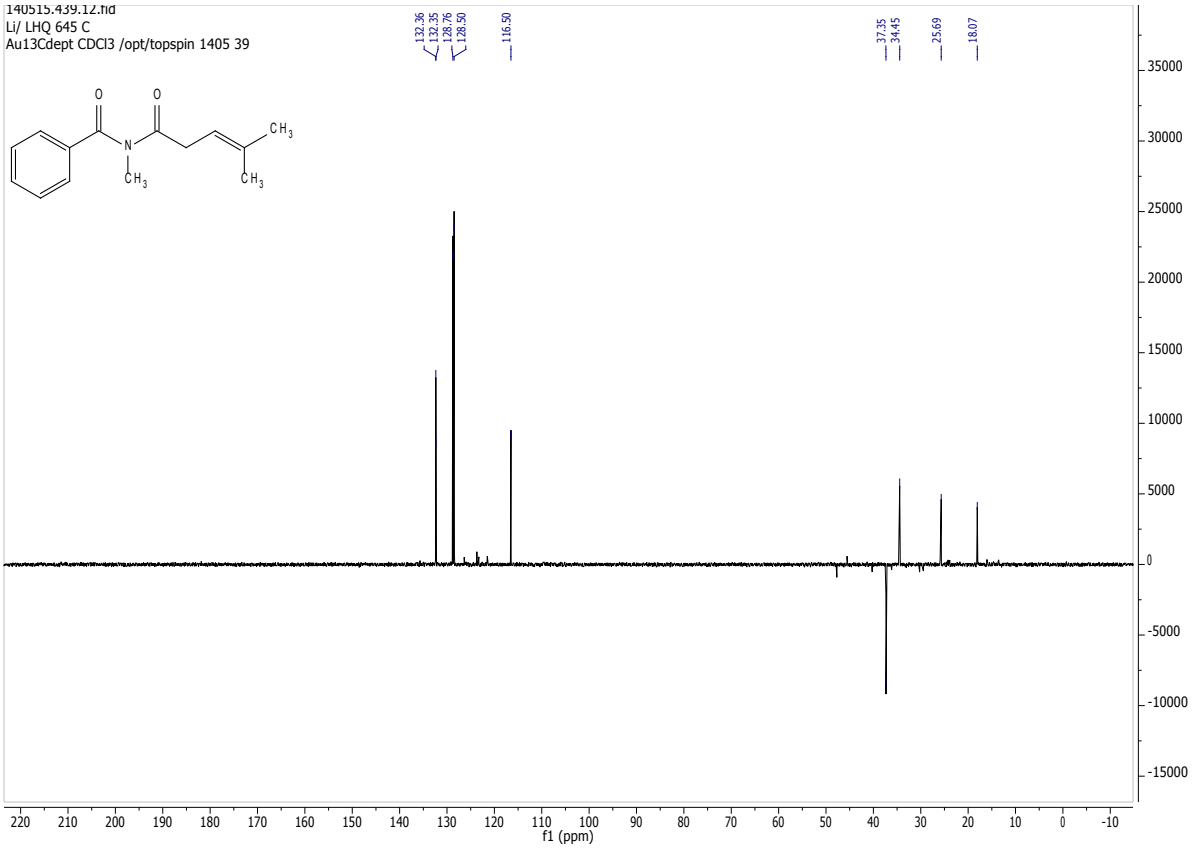
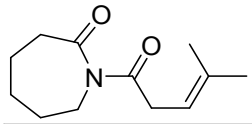
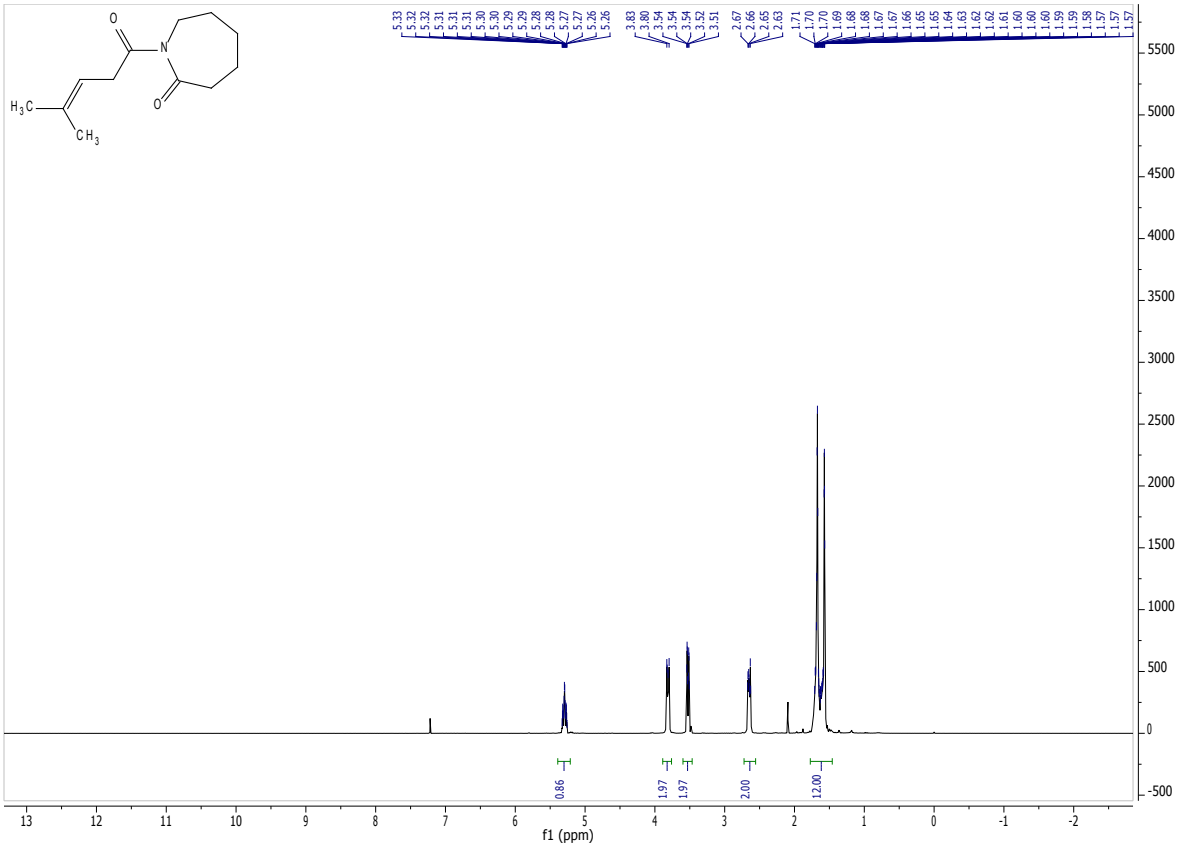
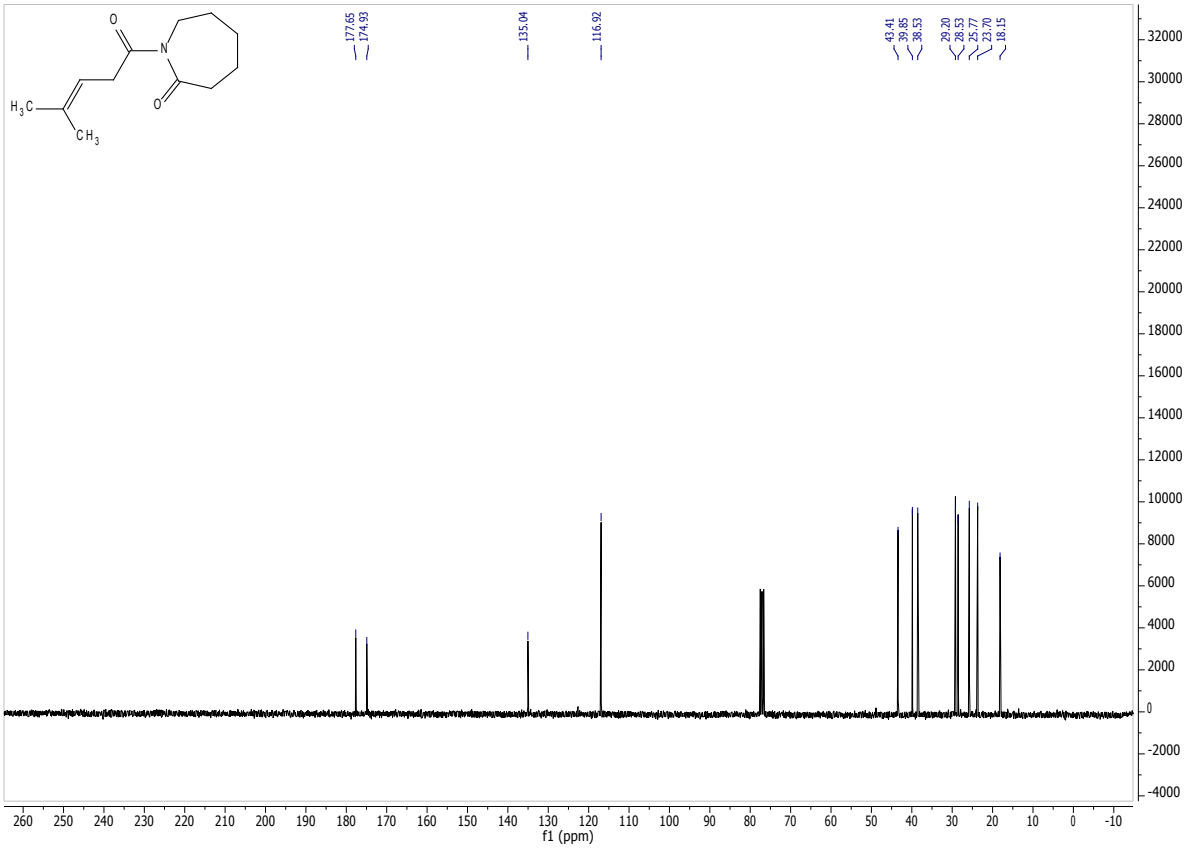
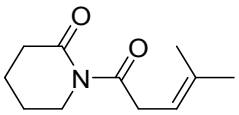
128.75
128.50
116.50
37.34
34.44
25.68
18.07
f1 (ppm)

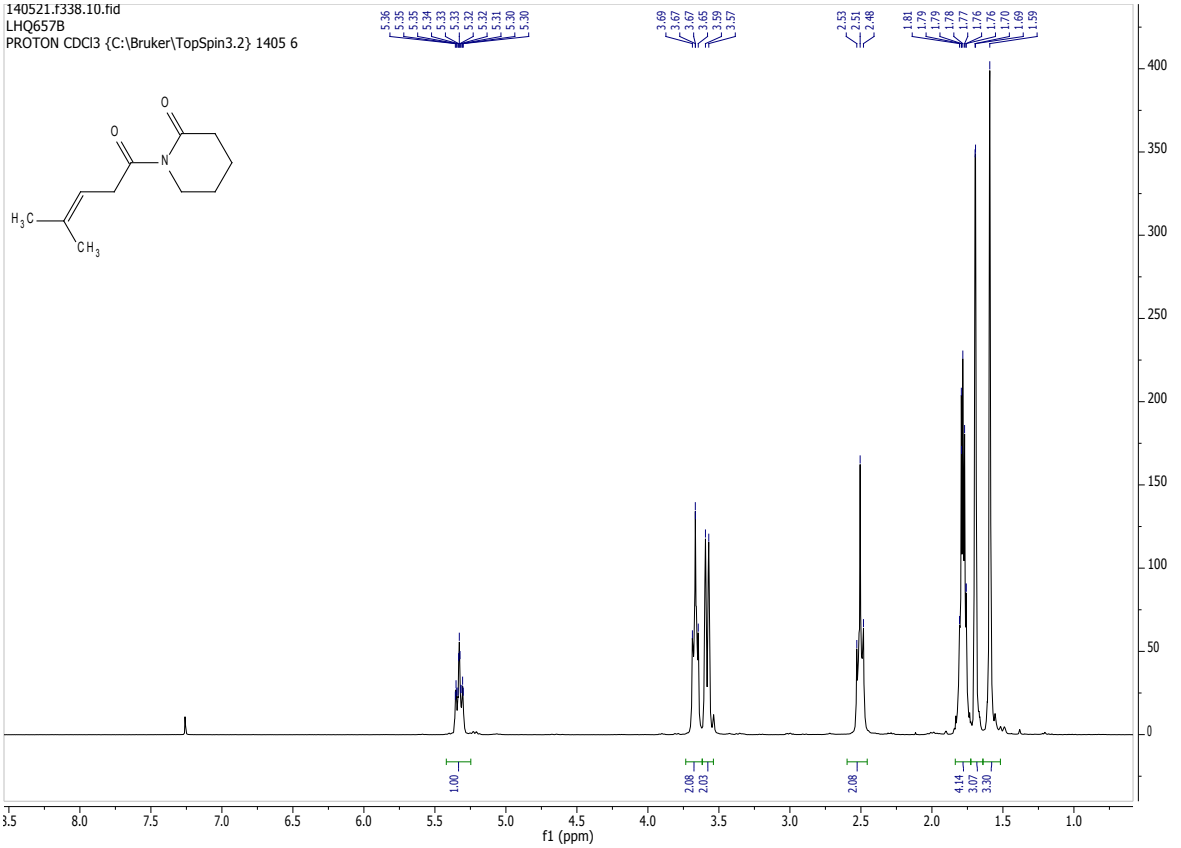

140515.439.12.fid
Li/ LHQ 645 C
Au13Cdept CDCl3 /opt/topspin 1405 39
132.36
132.35
128.76
128.50
116.50
37.35
34.45
25.69
18.07
O
O
N
CH3
CH3
CH3
35000
30000
25000
20000
15000
10000
5000
0
-5000
-10000
-15000
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)




5.33 5.32 5.32 5.31 5.31 5.31 5.30 5.30 5.29 5.29 5.28 5.28 5.27 5.27 5.26 5.26
3.83 3.80 3.54 3.54 3.54 3.52 3.51
2.67 2.66 2.65 2.63
1.71 1.70 1.70 1.69 1.68 1.68 1.67 1.67 1.66 1.65 1.64 1.63 1.62 1.61 1.60 1.60 1.60 1.59 1.59 1.58 1.57 1.57 1.57
0.86
1.97
1.97
2.00
12.00
f1 (ppm)


177.65
174.93
135.04
116.92
43.41
39.85
38.53
29.20
28.53
25.77
23.70
18.15
f1 (ppm)




140521.f338.10.fid
LHQ657B
PROTON CDCl3 {C:\Bruker\TopSpin3.2} 1405 6
5.36
5.35
5.35
5.34
5.33
5.33
5.32
5.32
5.31
5.30
5.30
3.69
3.67
3.67
3.65
3.59
3.57
2.53
2.51
2.48
1.81
1.79
1.79
1.78
1.77
1.76
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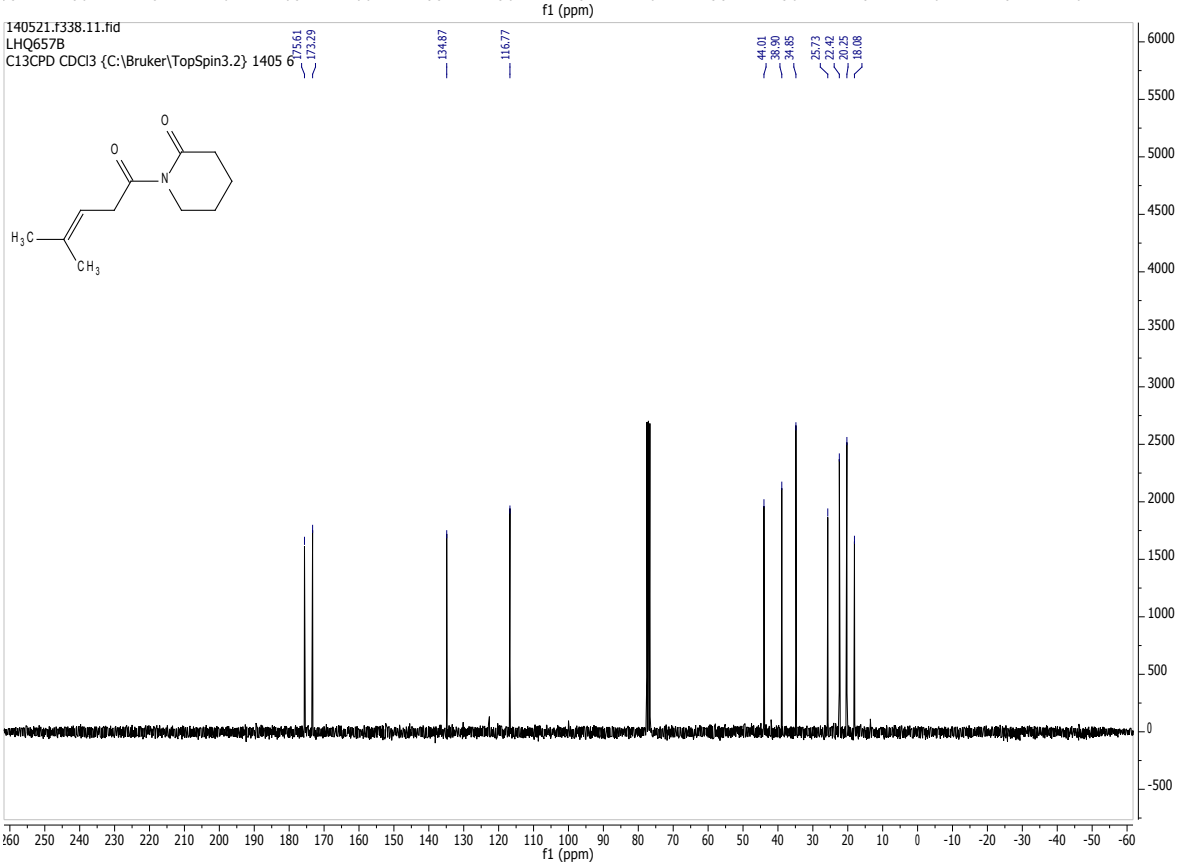
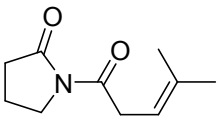
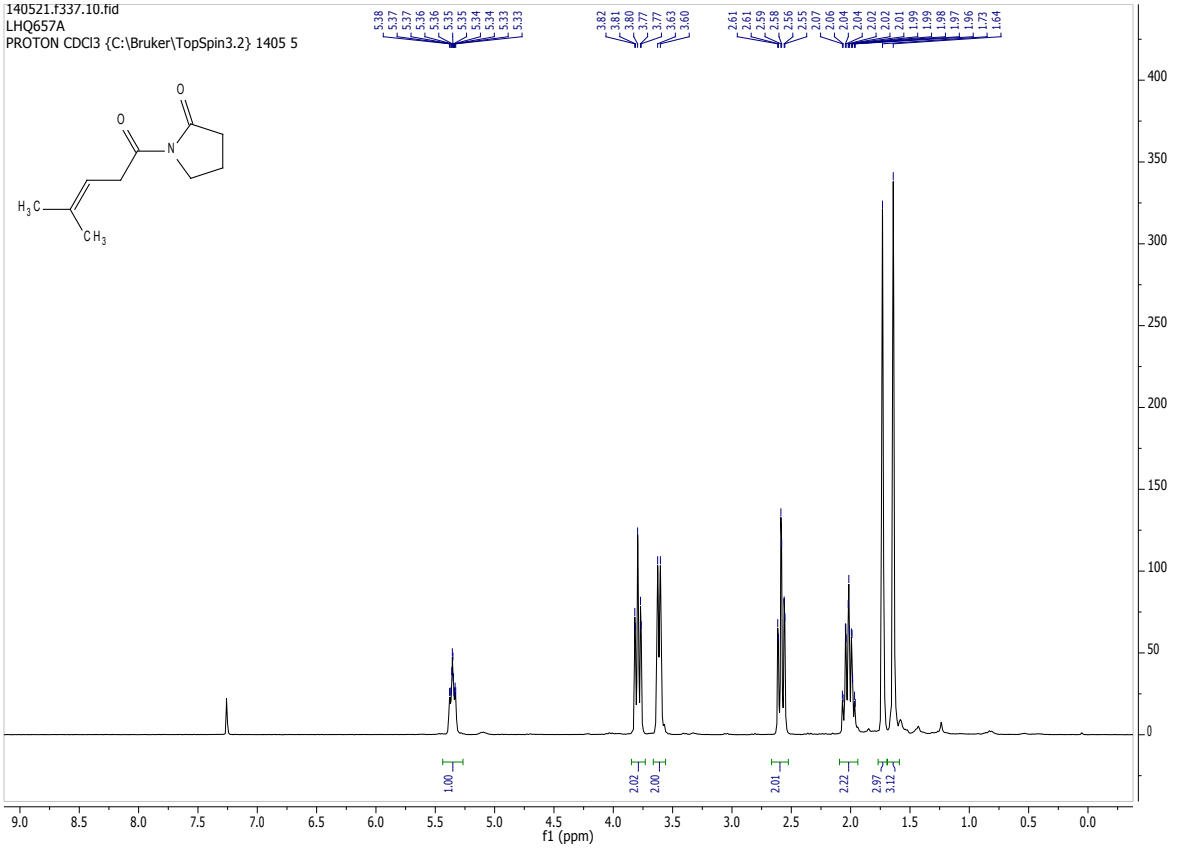
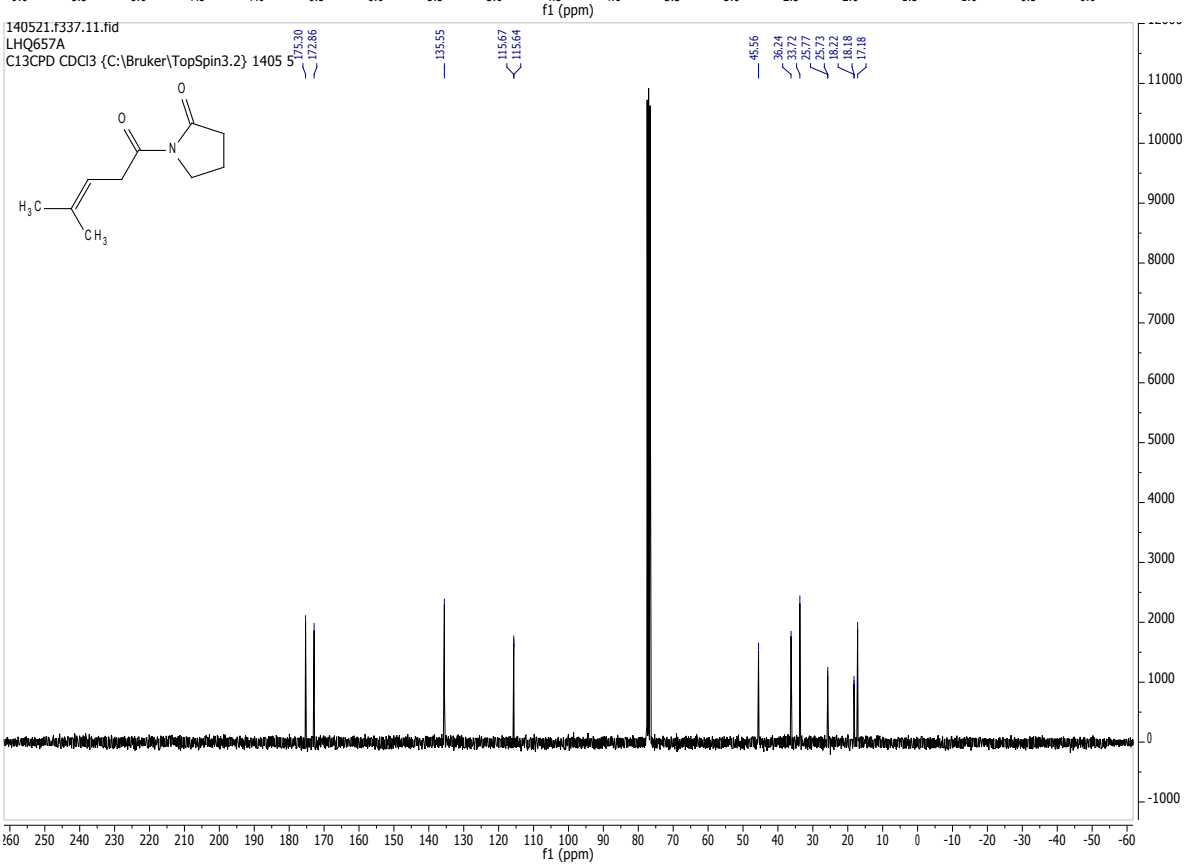
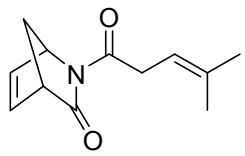
2.03
2.08
4.14
3.07
3.30
f1 (ppm)

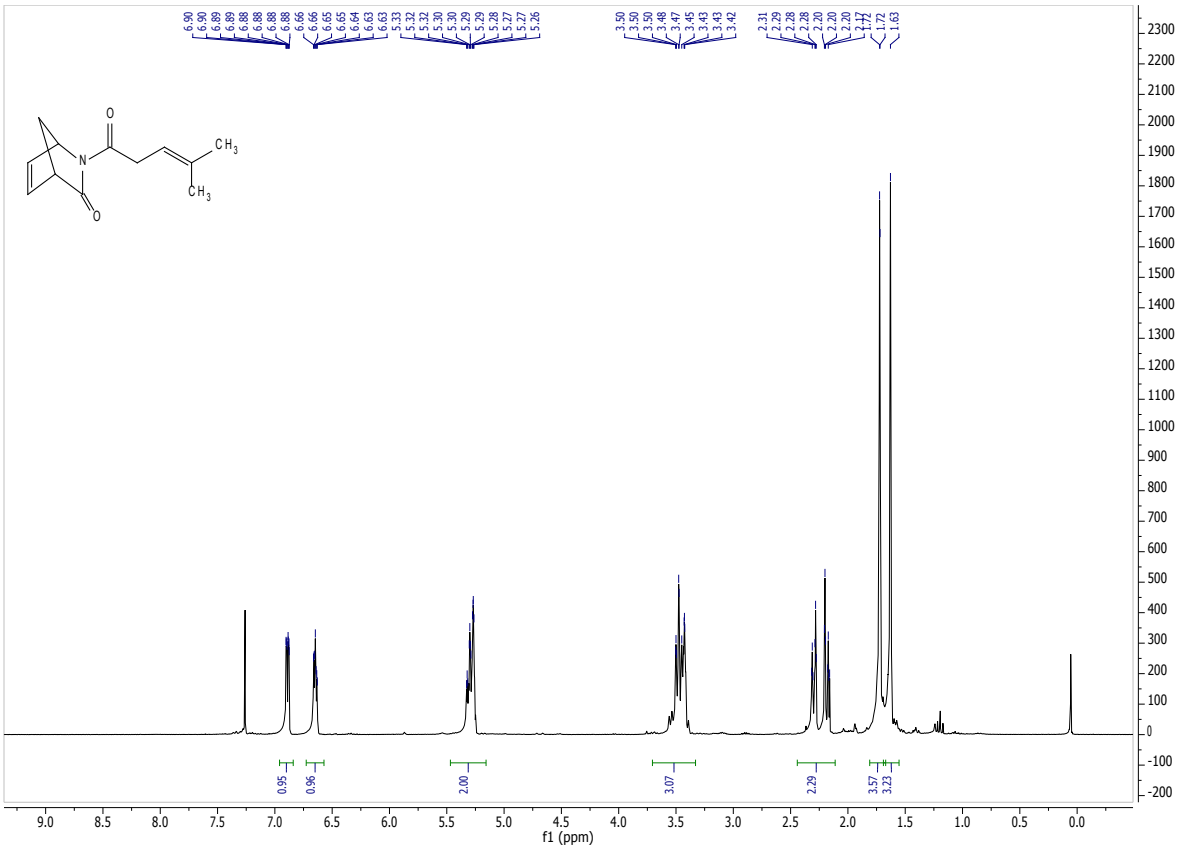

140521.f338.11.fid
LHQ657B
C13CPD CDCl3 {C:\Bruker\TopSpin3.2} 1405 6
175.61
173.29
134.87
116.77
44.01
38.90
34.85
25.73
22.42
20.25
18.08
f1 (ppm)

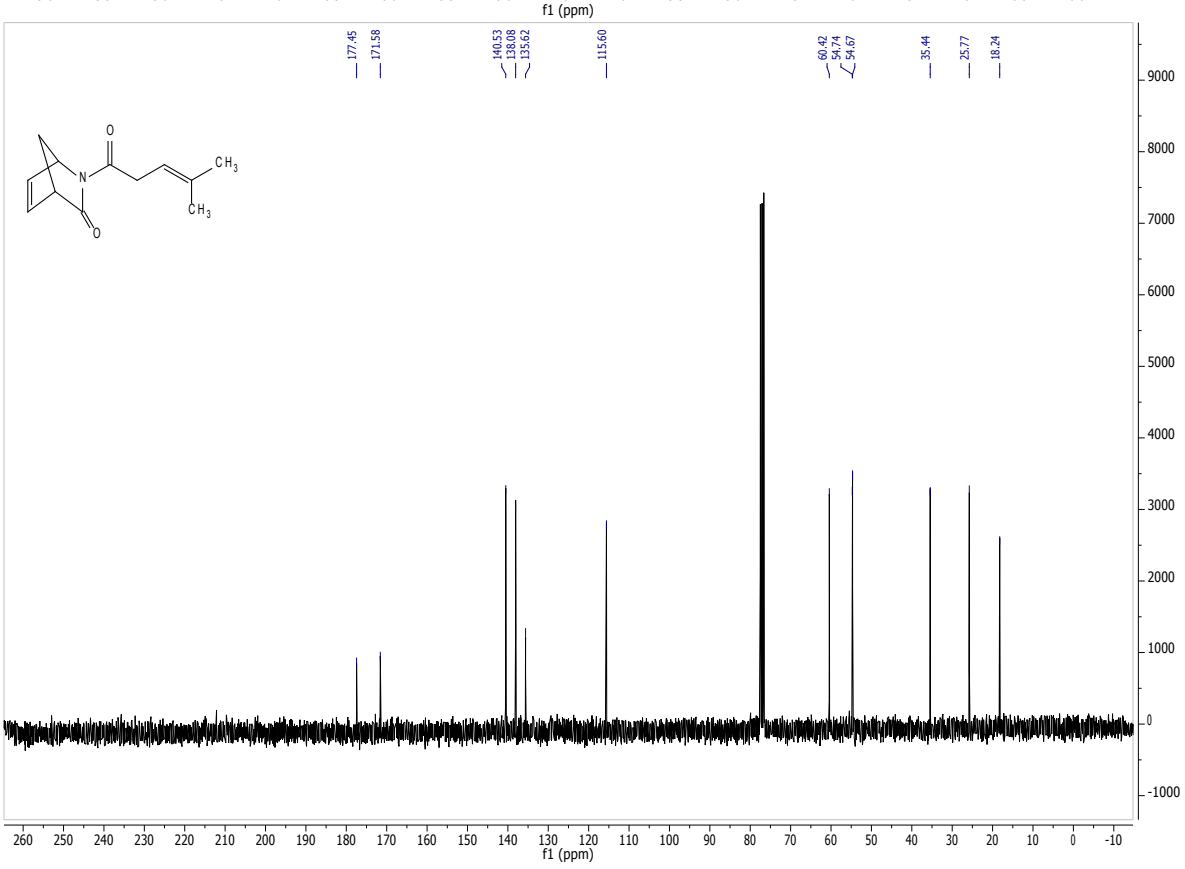


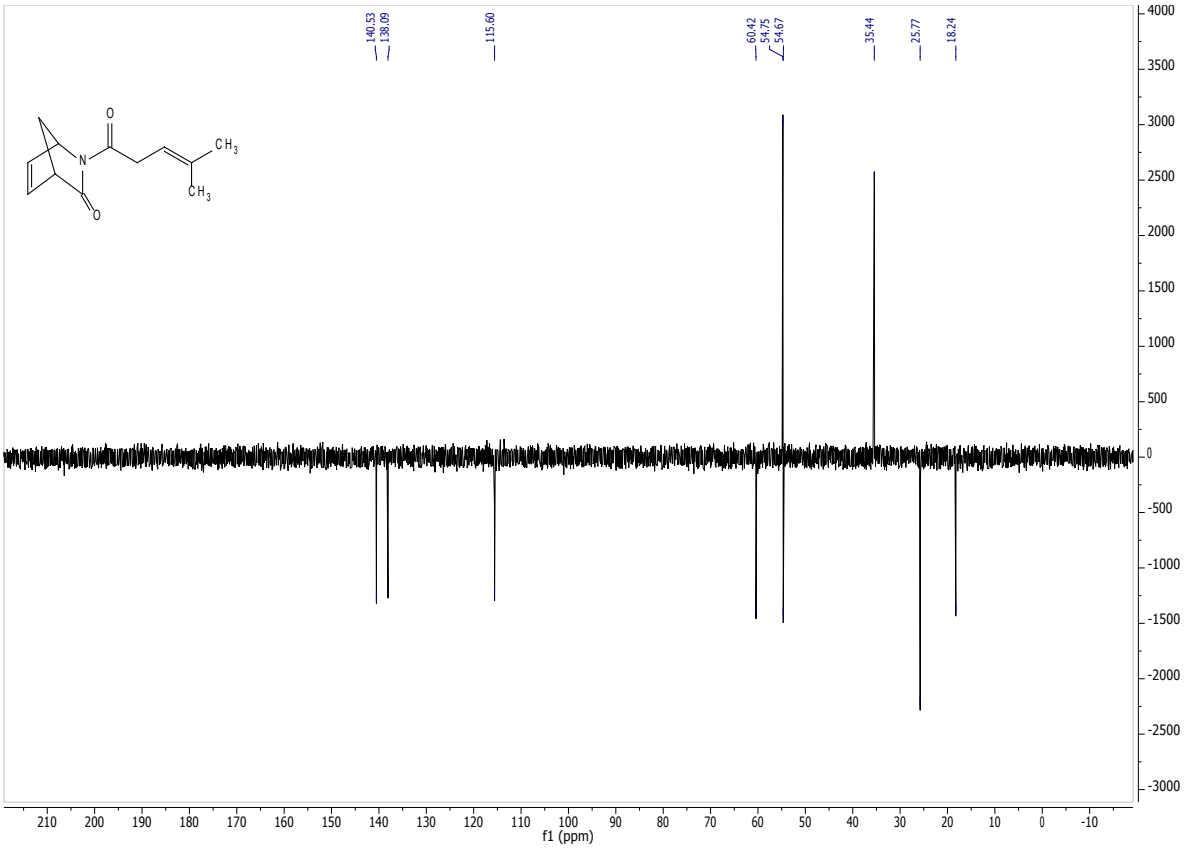

140521.f337.10.fid
LHQ657A
PROTON CDCl3 {C:\Bruker\TopSpin3.2} 1405 5
5.38 5.37 5.37 5.36 5.36 5.35 5.35 5.34 5.34 5.33 5.33
3.82 3.81 3.80 3.77 3.77 3.63 3.60
2.61 2.61 2.59 2.58 2.56 2.55 2.07 2.06 2.04 2.04 2.02 2.02 2.01 1.99 1.99 1.98 1.97 1.96 1.73 1.64
1.00 2.02 2.00 2.01 2.22 2.97 3.12
f1 (ppm)

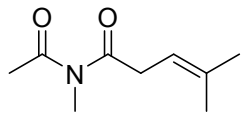

140521.f337.11.fid
LHQ657A
C13CPD CDCl3 {C:\Bruker\TopSpin3.2} 1405 5
175.30 172.86 135.55 115.67 115.64 45.56 36.24 33.72 25.77 25.73 18.22 17.18
f1 (ppm)

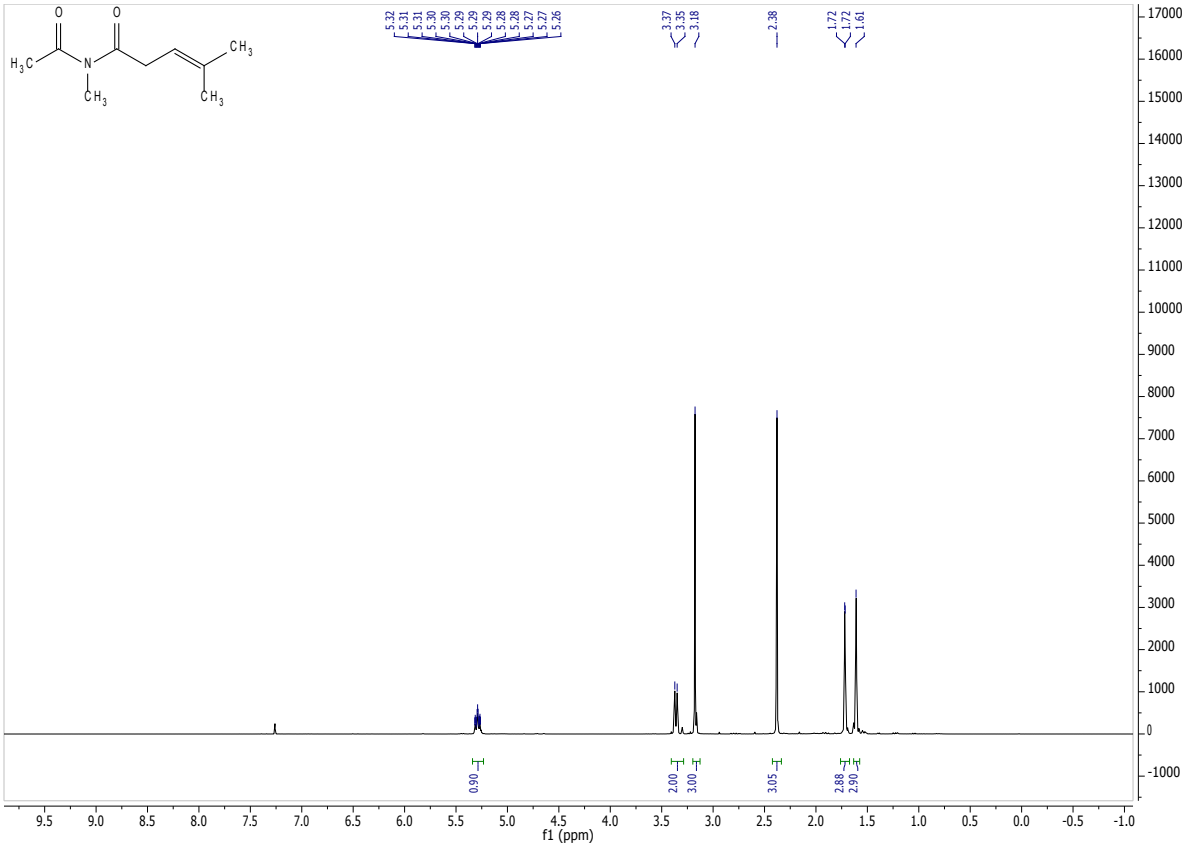


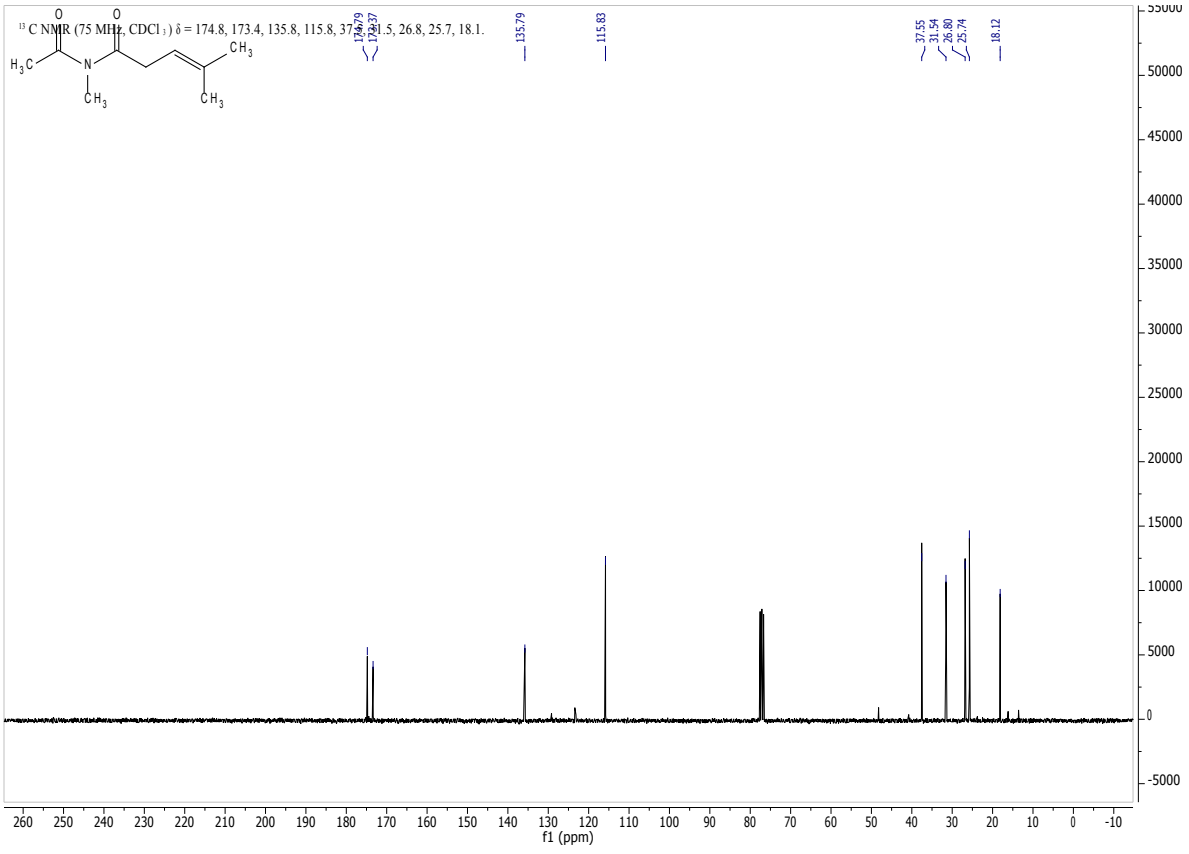

6.90 6.90 6.89 6.89 6.88 6.88 6.88 6.88 6.66 6.66 6.65 6.65 6.64 6.63 6.63 5.33 5.32 5.32 5.30 5.30 5.29 5.29 5.28 5.27 5.27 5.26
3.50 3.50 3.50 3.48 3.47 3.45 3.43 3.43 3.42
2.31 2.29 2.28 2.28 2.20 2.20 2.20 2.17 1.72 1.71 1.63
0.95 0.96 2.00 3.07 2.29 3.57 3.23
f1 (ppm)

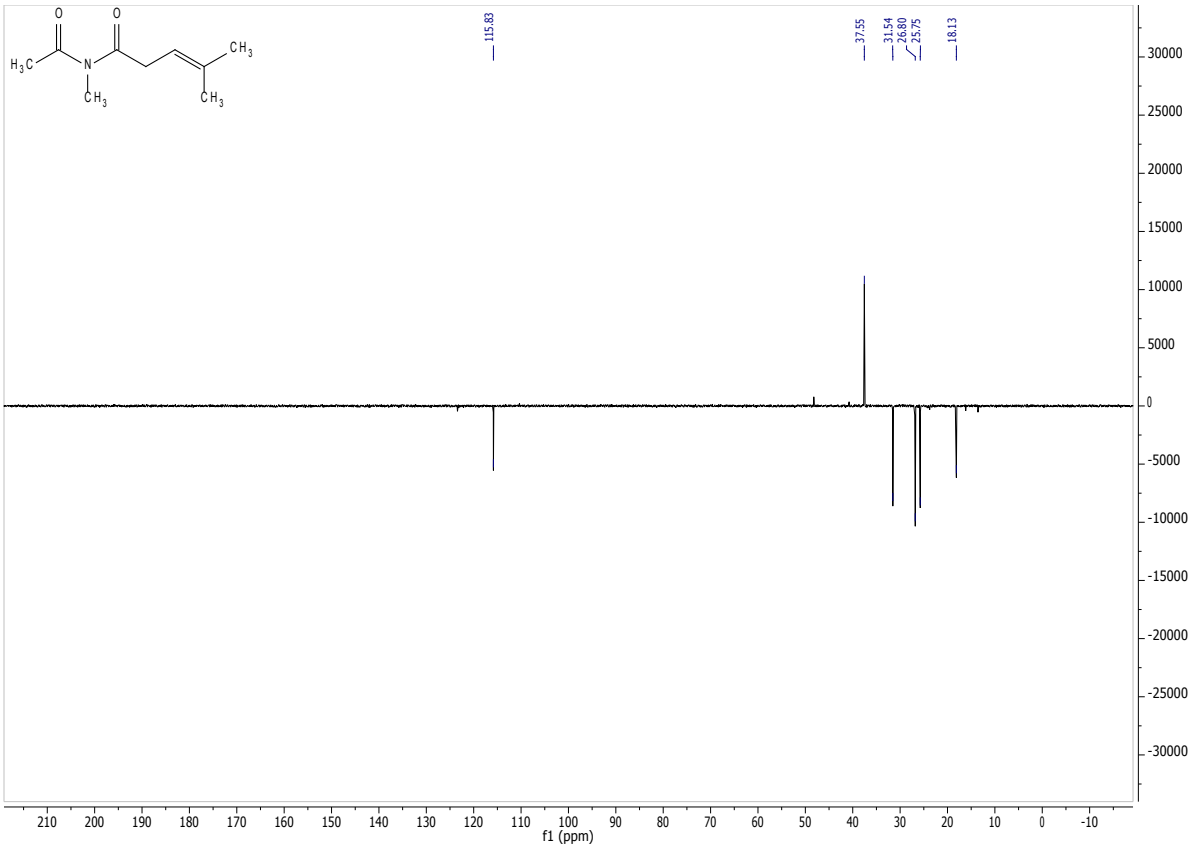

177.45 171.58 140.53 138.08 135.62 115.60 60.42 54.74 54.67 35.44 25.77 18.24
f1 (ppm)

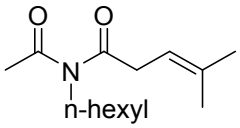

140.53
138.09
115.60
60.42
54.75
54.67
35.44
25.77
18.24
O
N
O
CH3
CH3
4000
3500
3000
2500
2000
1500
1000
500
0
-500
-1000
-1500
-2000
-2500
-3000
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

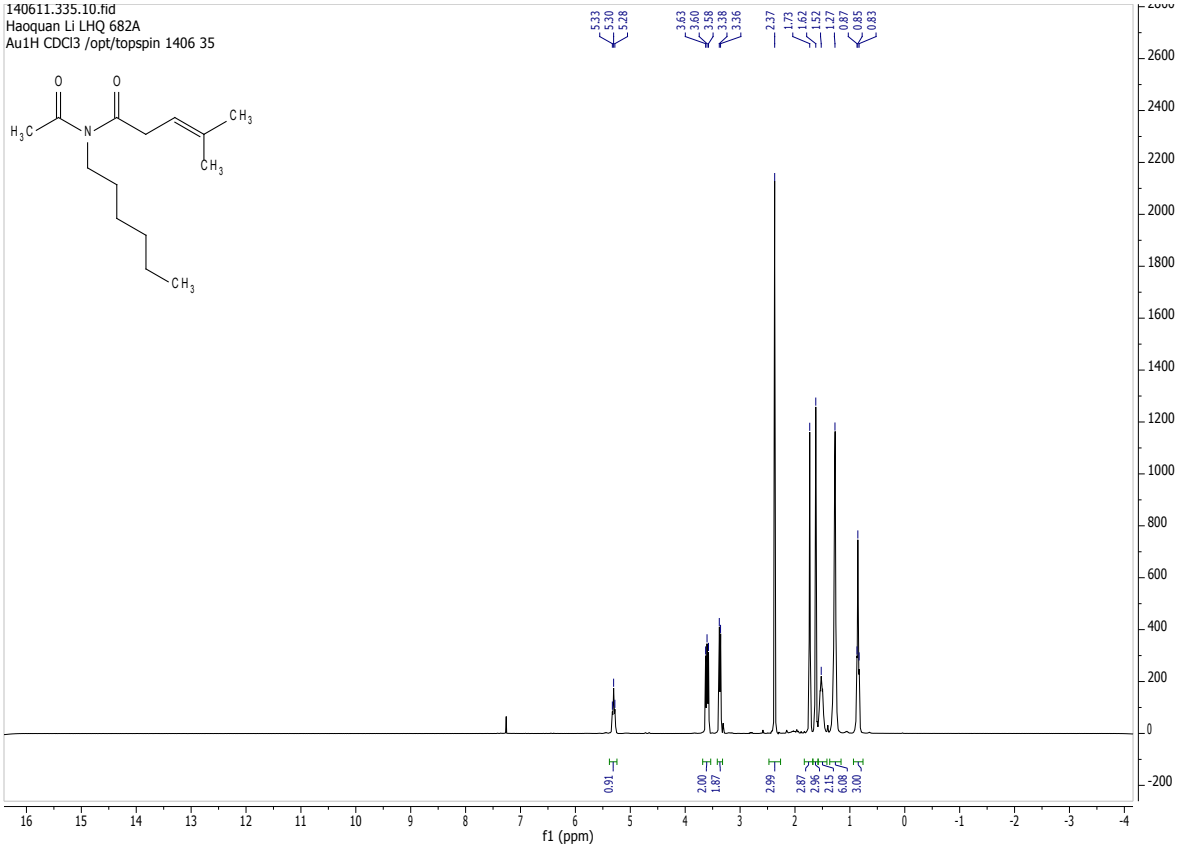


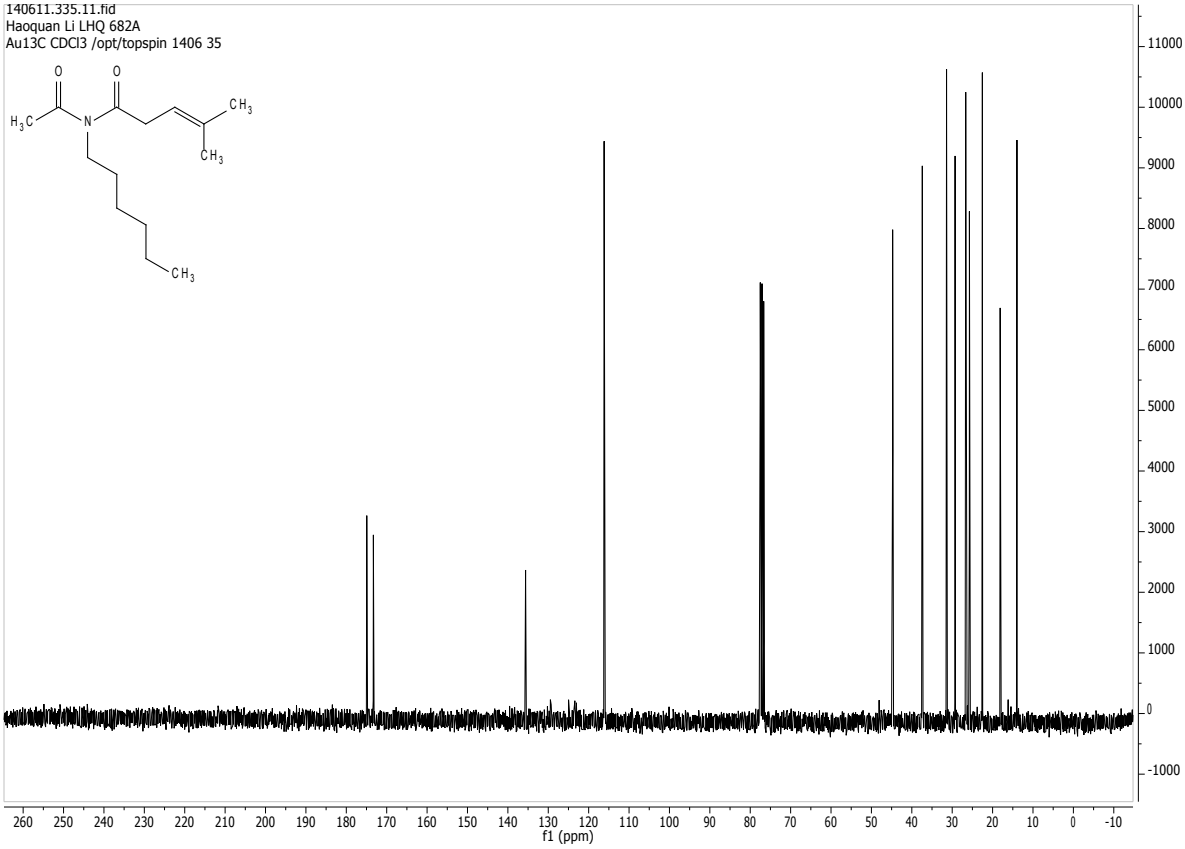

5.32
5.31
5.31
5.30
5.30
5.29
5.29
5.29
5.28
5.28
5.27
5.27
5.26
3.37
3.35
3.18
2.38
1.72
1.72
1.61
0.90
2.00
3.00
3.05
2.88
2.90
f1 (ppm)

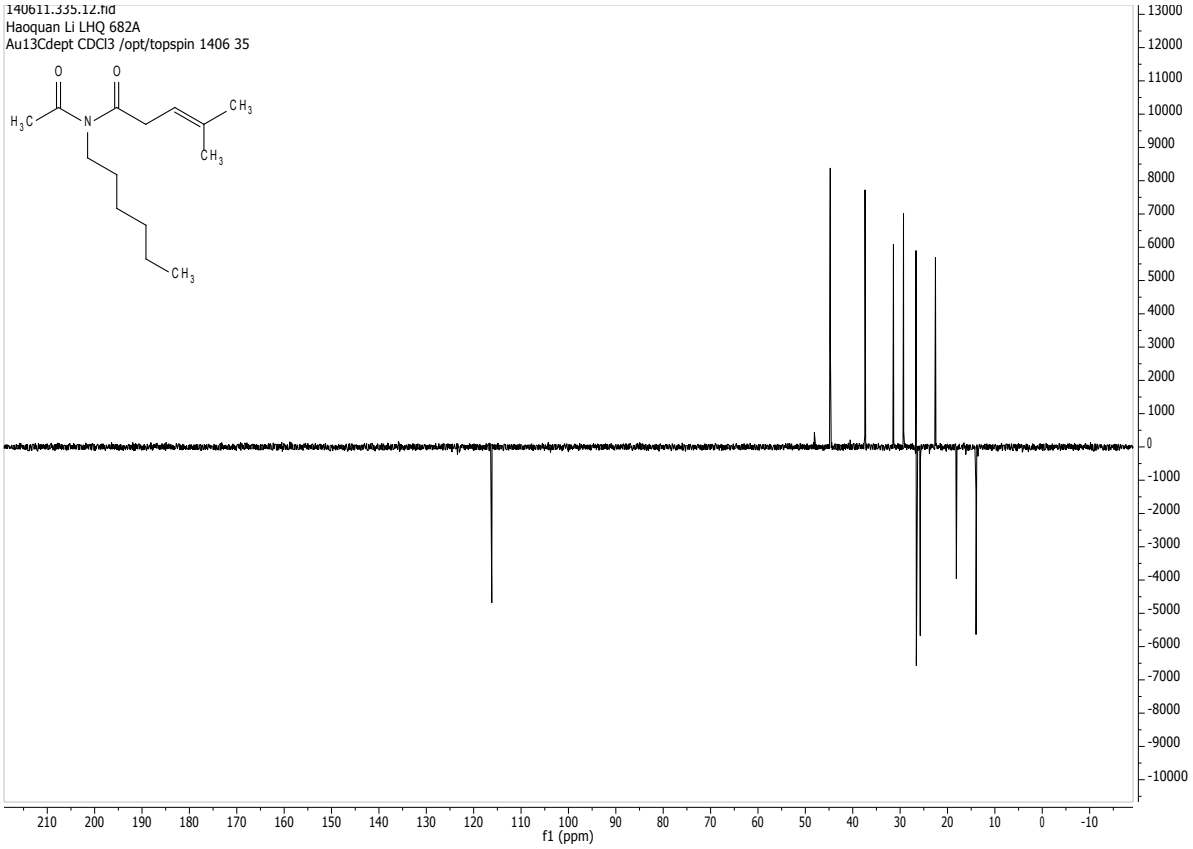

13 C NMR (75 MHz, CDCl 3) δ = 174.8, 173.4, 135.8, 115.8, 37.6, 31.5, 26.8, 25.7, 18.1.
174.79
173.37
135.79
115.83
37.55
31.54
26.80
25.74
18.12
f1 (ppm)

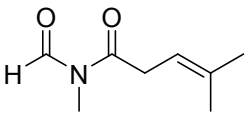

O
O
H3C
N
CH3
CH3
CH3
115.83
37.55
31.54
26.80
25.75
18.13
30000
25000
20000
15000
10000
5000
0
-5000
-10000
-15000
-20000
-25000
-30000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

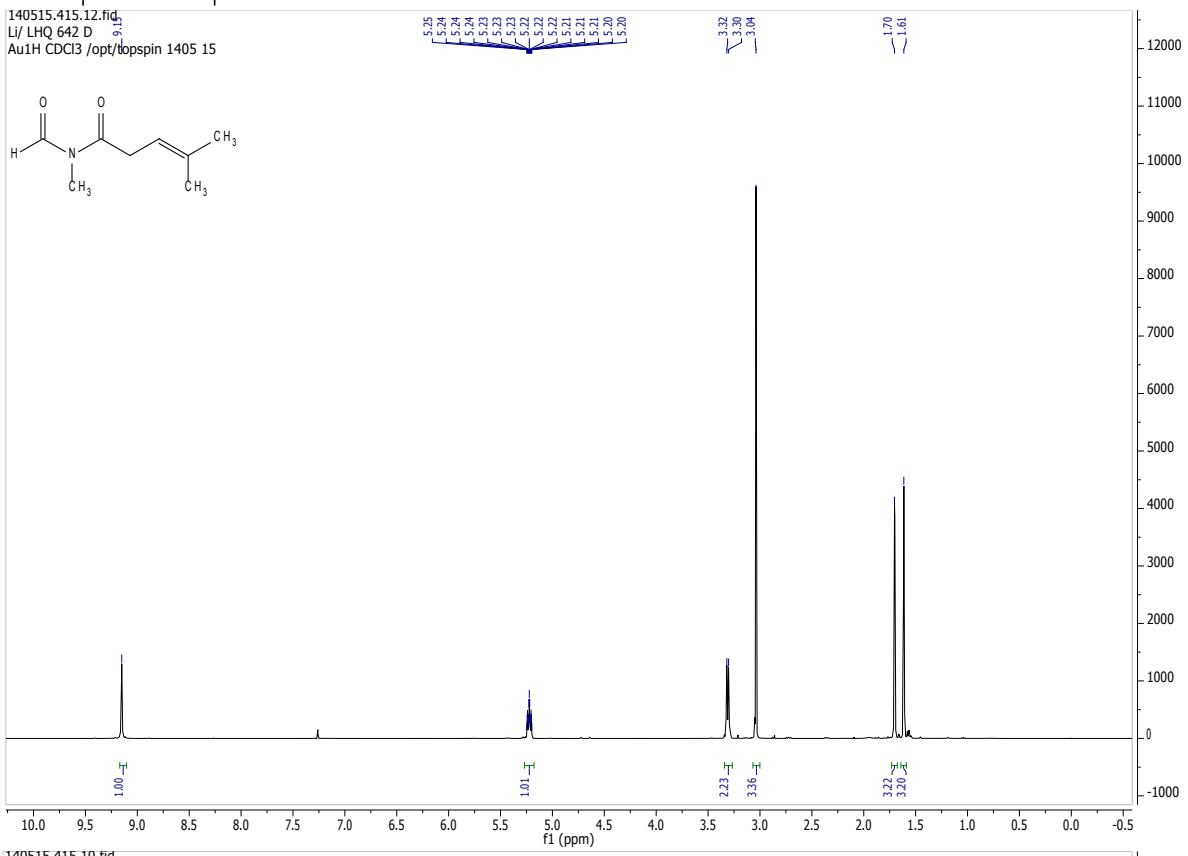

O
O
N
n-hexyl

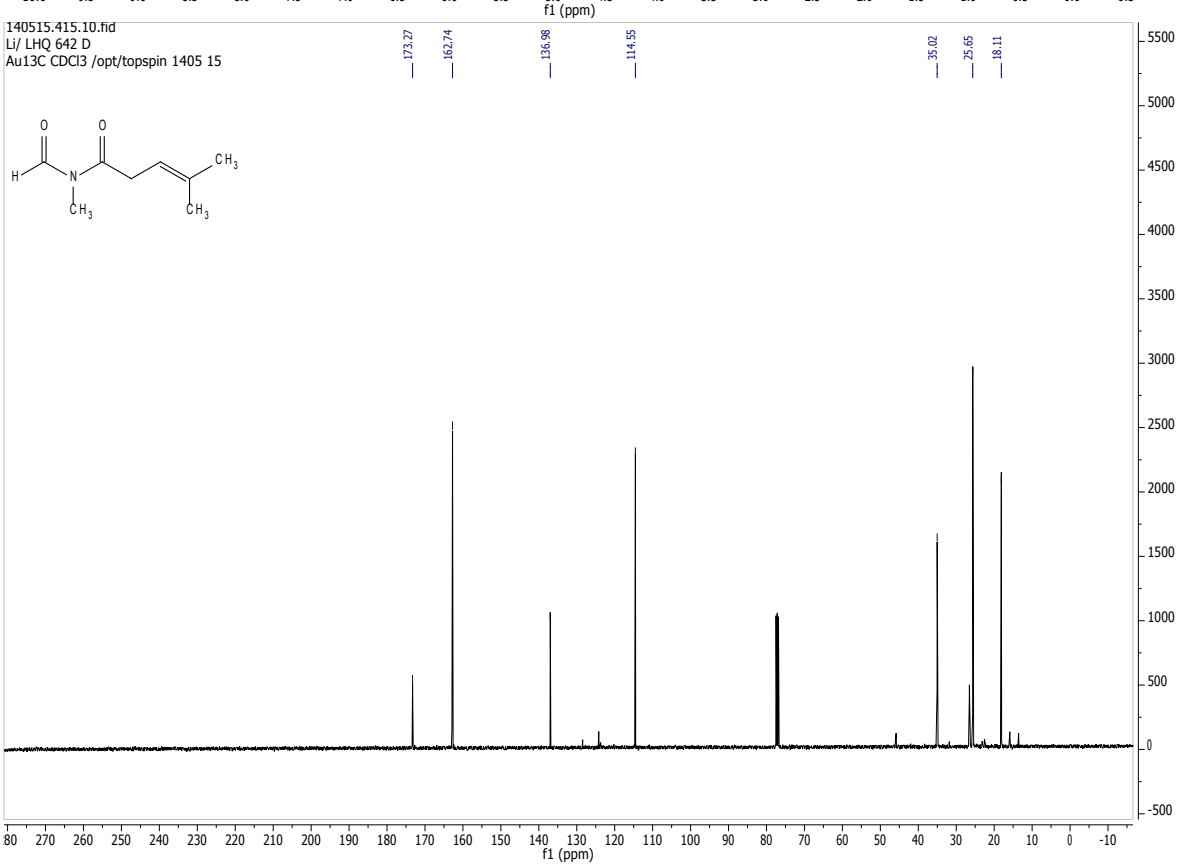

140611.335.10.fid
Haoquan Li LHQ 682A
Au1H CDCl3 /opt/topspin 1406 35
5.33
5.30
5.28
3.63
3.60
3.58
3.38
3.36
2.37
1.73
1.62
1.52
1.27
0.87
0.85
0.83
0.91
2.00
1.87
2.99
2.87
2.96
2.15
6.08
3.00
f1 (ppm)

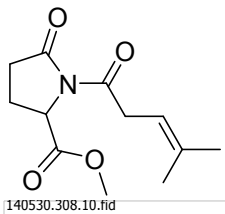

140611.335.11.fid
Haoquan Li LHQ 682A
Au13C CDCl3 /opt/topspin 1406 35
f1 (ppm)

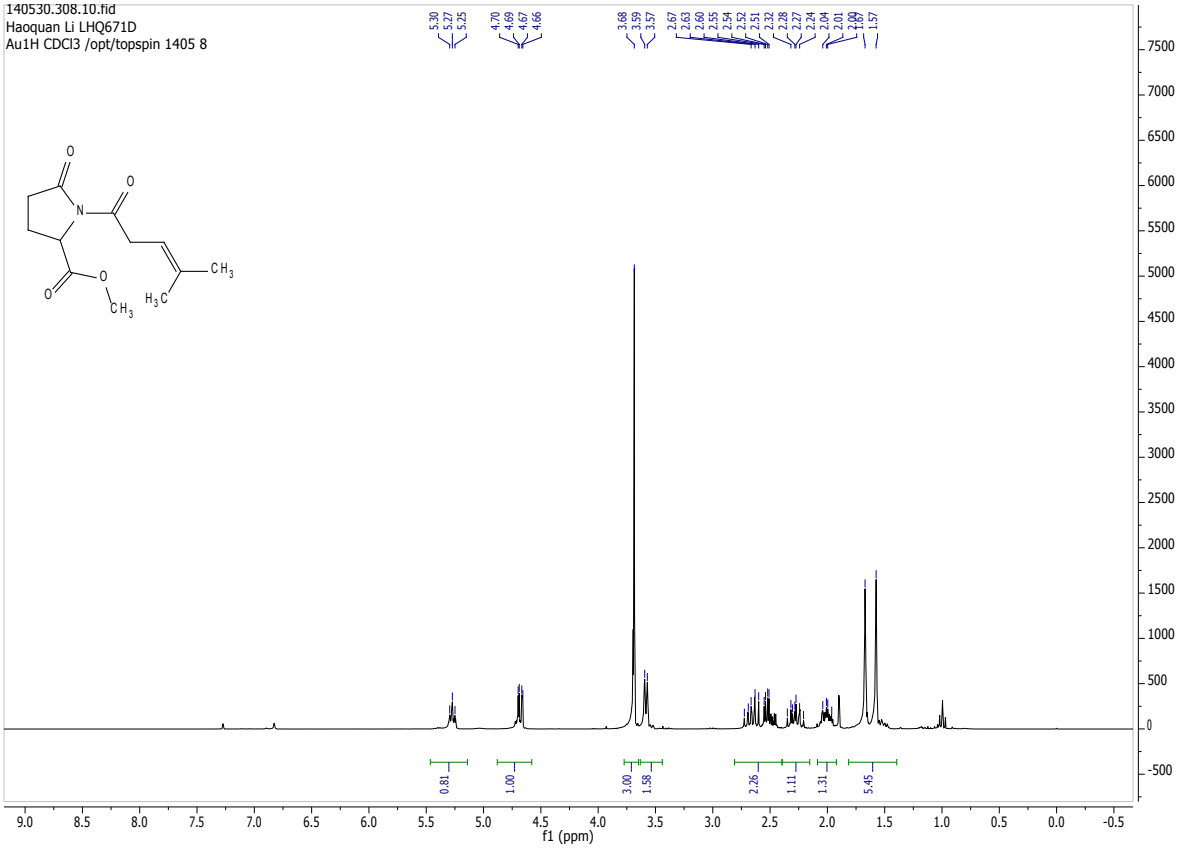

140611.335.12.fid
Haoquan Li LHQ 682A
Au13Cdept CDCl3 /opt/topspin 1406 35
O
O
H3C
N
CH3
CH3
CH3
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
-3000
-4000
-5000
-6000
-7000
-8000
-9000
-10000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

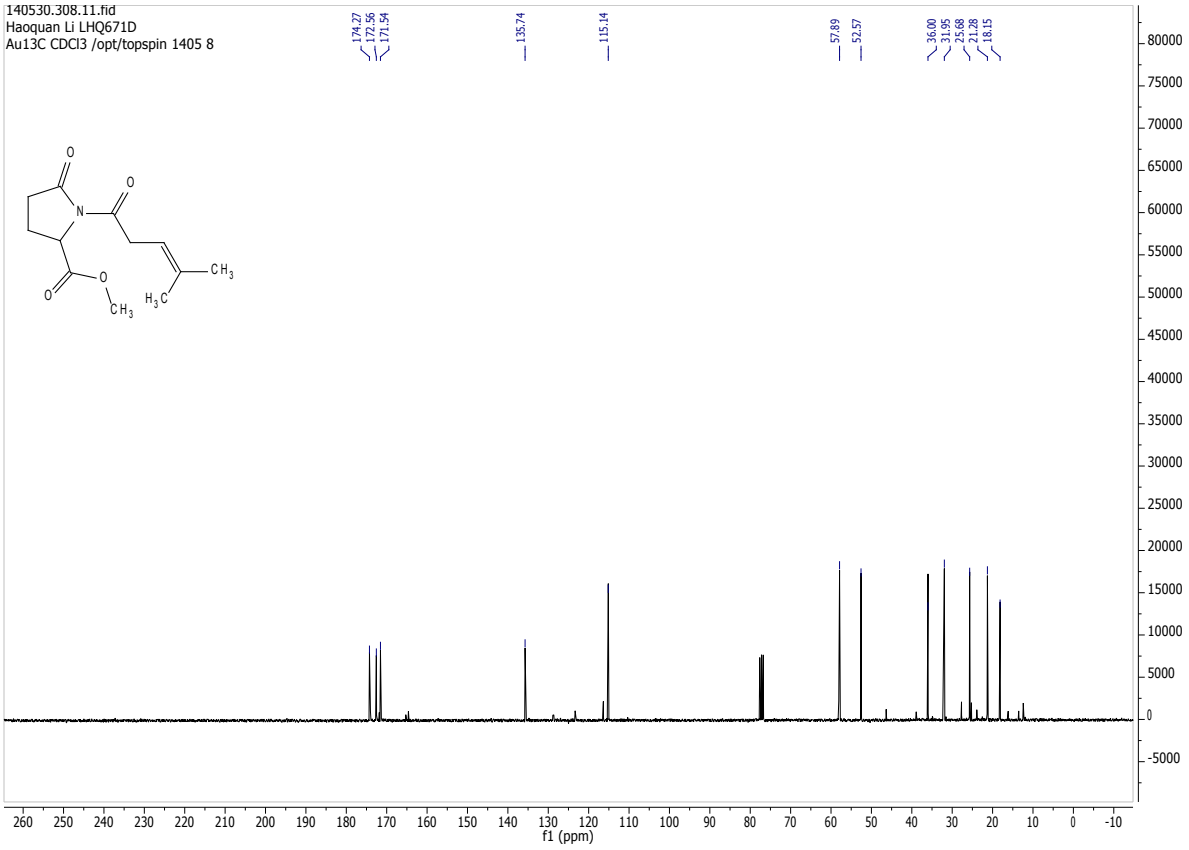


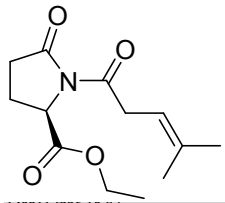

140515.415.12.fid
Li/ LHQ 642 D
Au1H CDCl3 /opt/topspin 1405 15
9.11
5.25
5.24
5.24
5.24
5.23
5.23
5.23
5.22
5.22
5.22
5.21
5.21
5.21
5.20
5.20
3.32
3.30
3.04
1.70
1.61
1.00
1.01
2.23
3.36
3.22
3.20
f1 (ppm)

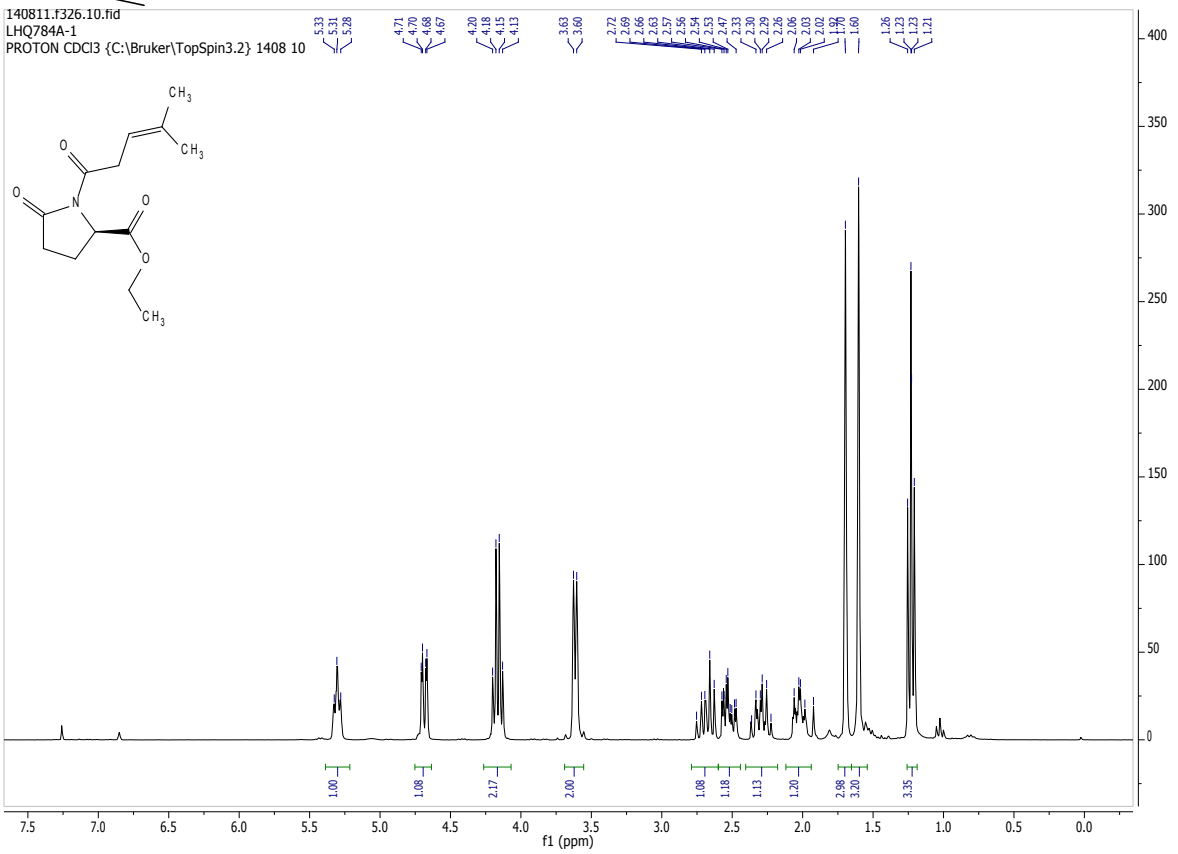

140515.415.10.fid
Li/ LHQ 642 D
Au13C CDCl3 /opt/topspin 1405 15
173.27
162.74
136.98
114.55
35.02
25.65
18.11
f1 (ppm)

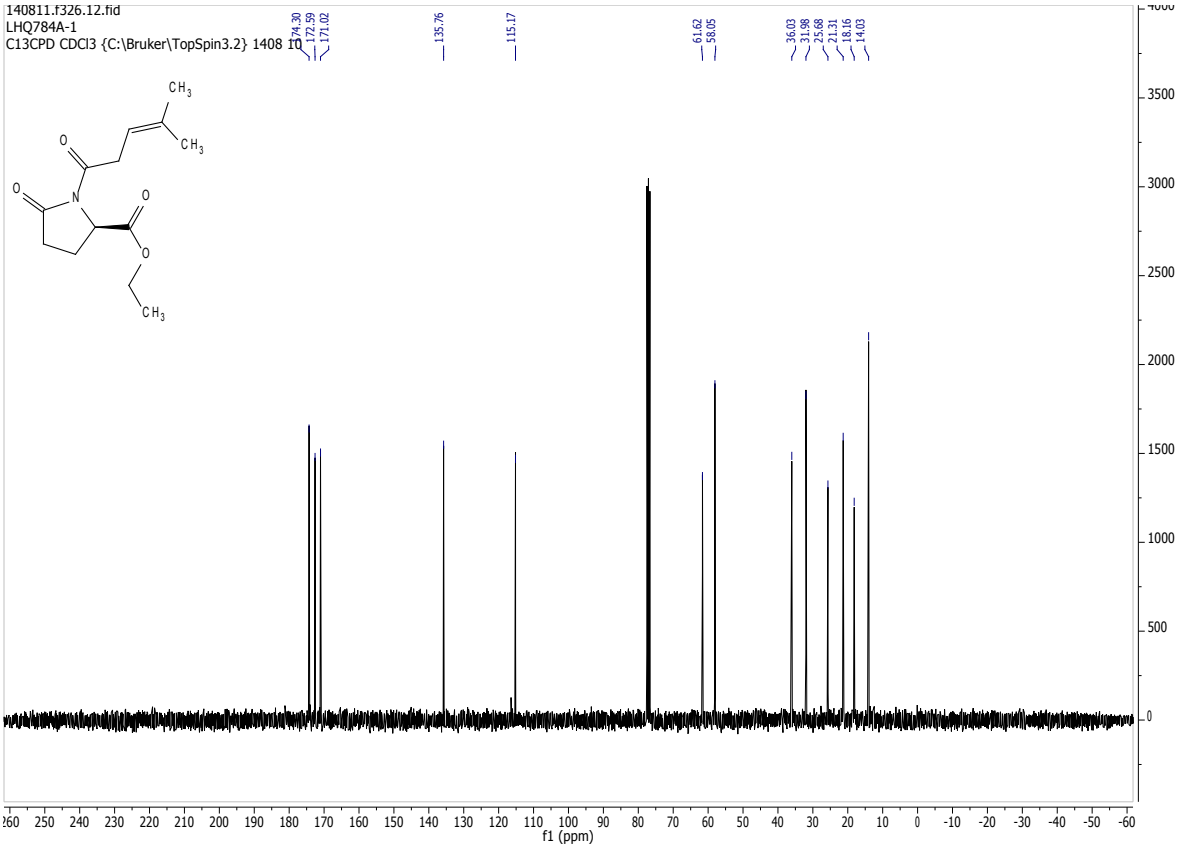


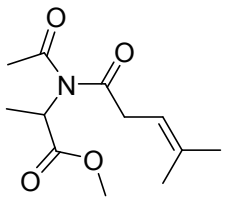

140530.308.10.fid
Haoquan Li LHQ671D
Au1H CDCl3 /opt/topspin 1405 8
5.30
5.27
5.25
4.70
4.69
4.67
4.66
3.68
3.59
3.57
2.67
2.63
2.60
2.55
2.54
2.52
2.51
2.32
2.28
2.27
2.24
2.04
2.01
1.89
1.87
1.57
0.81
1.00
3.00
1.58
2.26
1.11
1.31
5.45
f1 (ppm)

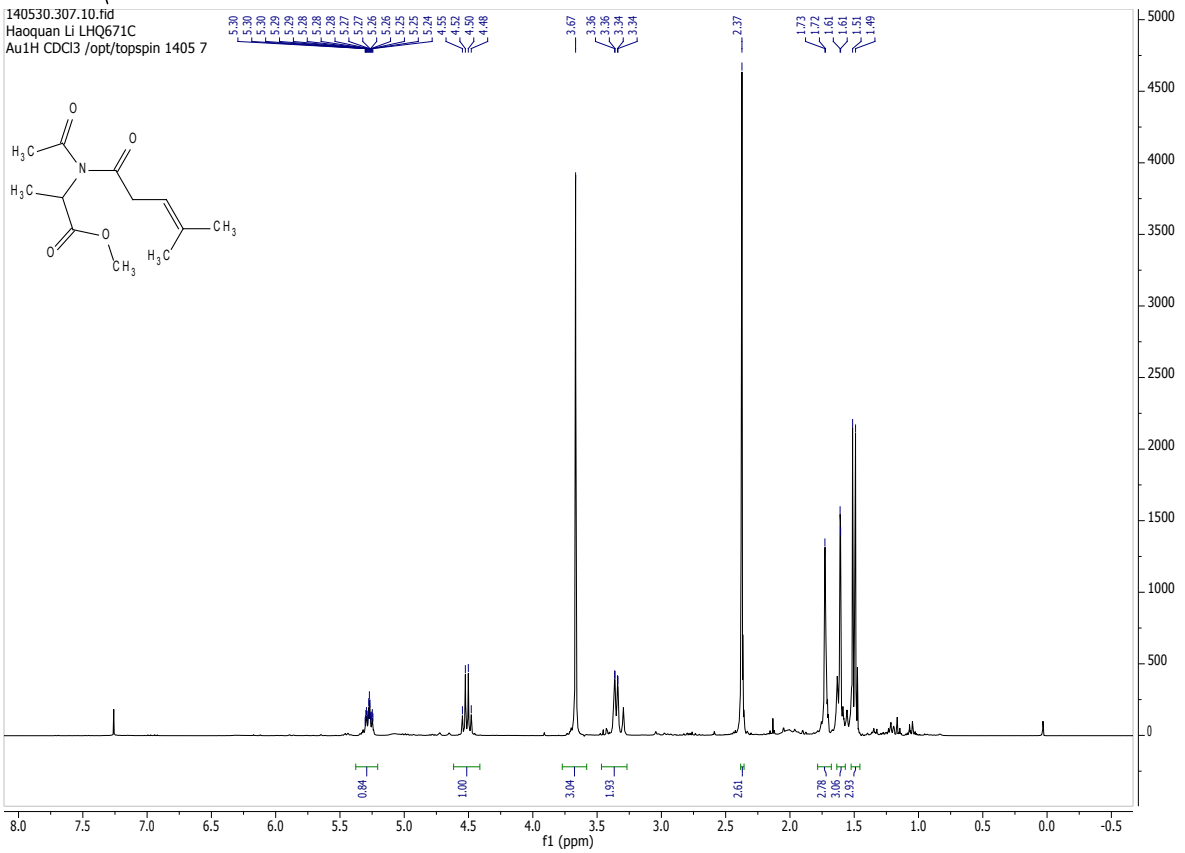

140530.308.11.fid
Haoquan Li LHQ671D
Au13C CDCl3 /opt/topspin 1405 8
174.27
172.56
171.54
135.74
115.14
57.89
52.57
36.00
31.95
25.68
21.28
18.15
f1 (ppm)

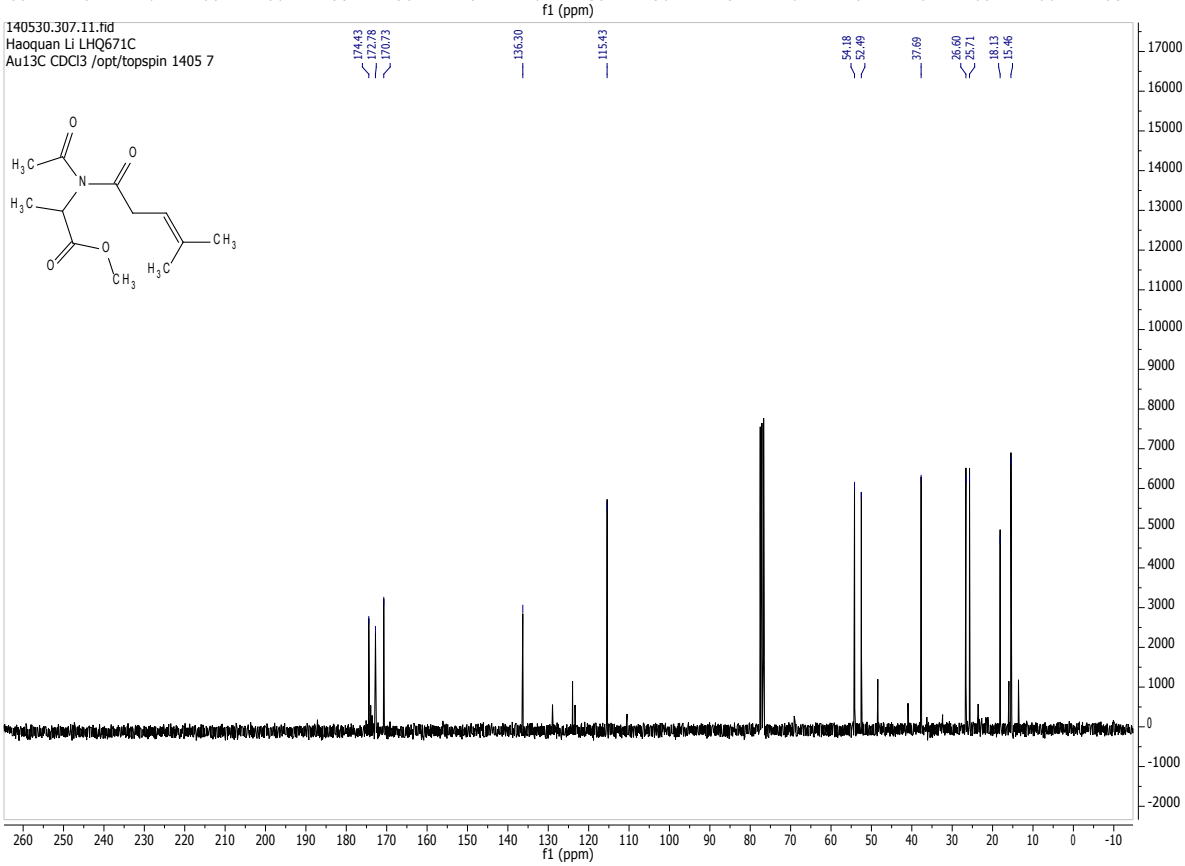


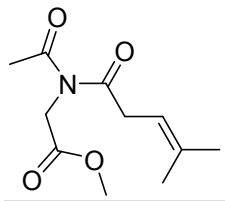

140811.f326.10.fid
LHQ784A-1
PROTON CDCl3 {C:\Bruker\TopSpin3.2} 1408 10
5.33
5.31
5.28
4.71
4.70
4.68
4.67
4.20
4.18
4.15
4.13
3.63
3.60
2.72
2.69
2.66
2.63
2.57
2.56
2.54
2.53
2.47
2.33
2.30
2.29
2.26
2.06
2.03
2.02
1.92
1.70
1.60
1.26
1.23
1.23
1.21
1.00
1.08
2.17
2.00
1.08
1.18
1.13
1.20
2.98
3.20
3.35
f1 (ppm)

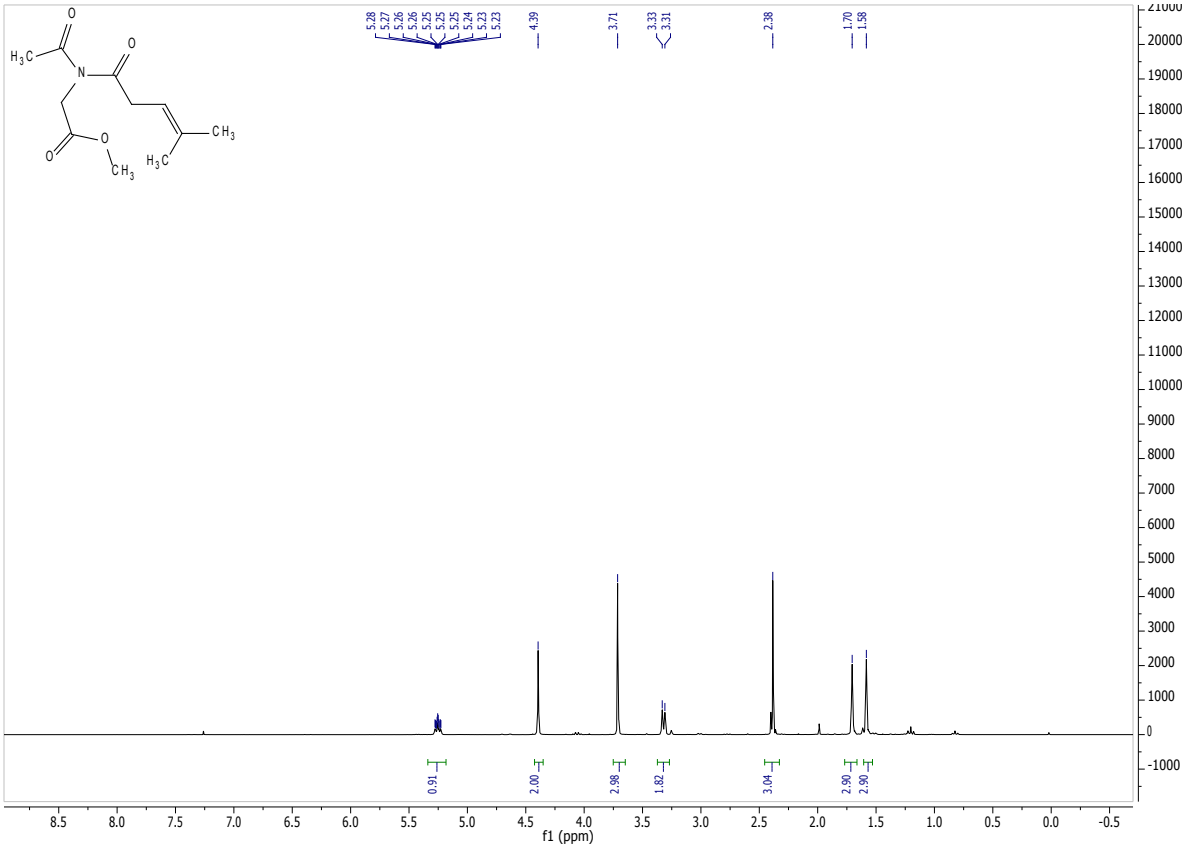

140811.f326.12.fid
LHQ784A-1
C13CPD CDCl3 {C:\Bruker\TopSpin3.2} 1408 10
174.30
172.59
171.02
135.76
115.17
61.62
58.05
36.03
31.98
25.68
21.31
18.16
14.03
f1 (ppm)

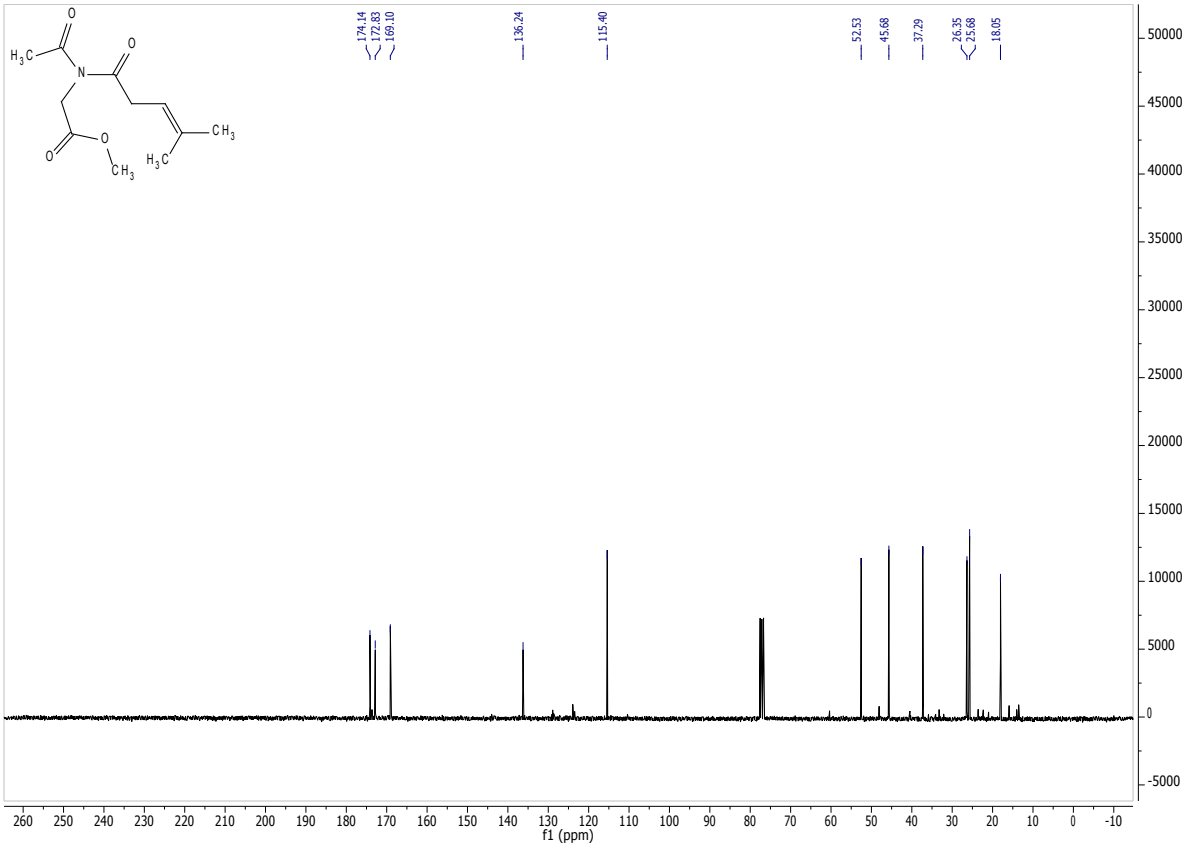


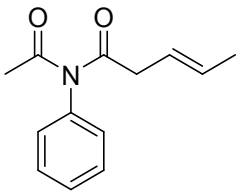

140530.307.10.fid
Haoquan Li LHQ671C
Au1H CDCl3 /opt/topspin 1405 7
5.30
5.30
5.30
5.29
5.29
5.28
5.28
5.28
5.27
5.27
5.26
5.26
5.25
5.25
5.24
4.55
4.52
4.50
4.48
3.67
3.36
3.36
3.34
3.34
2.37
1.73
1.72
1.61
1.61
1.51
1.49
0.84
1.00
3.04
1.93
2.61
2.78
3.06
2.93
f1 (ppm)

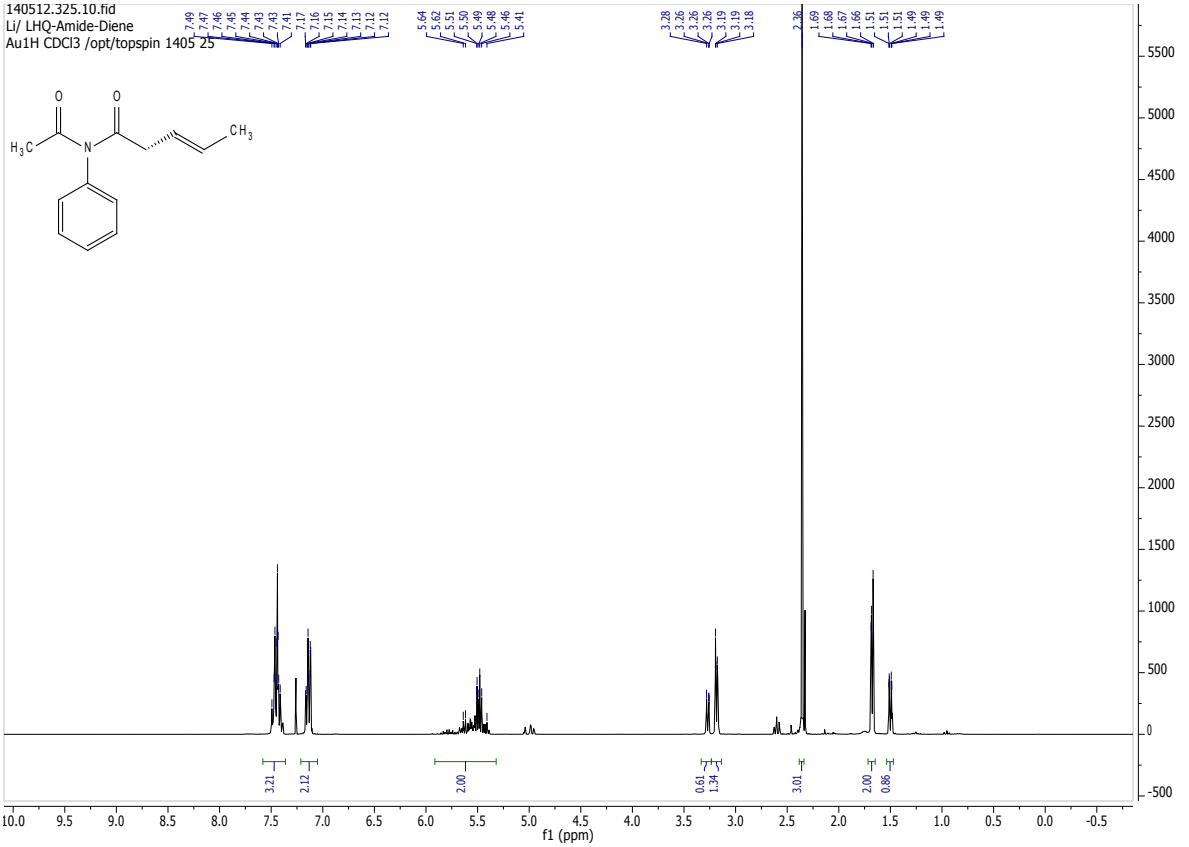

140530.307.11.fid
Haoquan Li LHQ671C
Au13C CDCl3 /opt/topspin 1405 7
174.43
172.78
170.73
136.30
115.43
54.18
52.49
37.69
26.60
25.71
18.13
15.46
f1 (ppm)

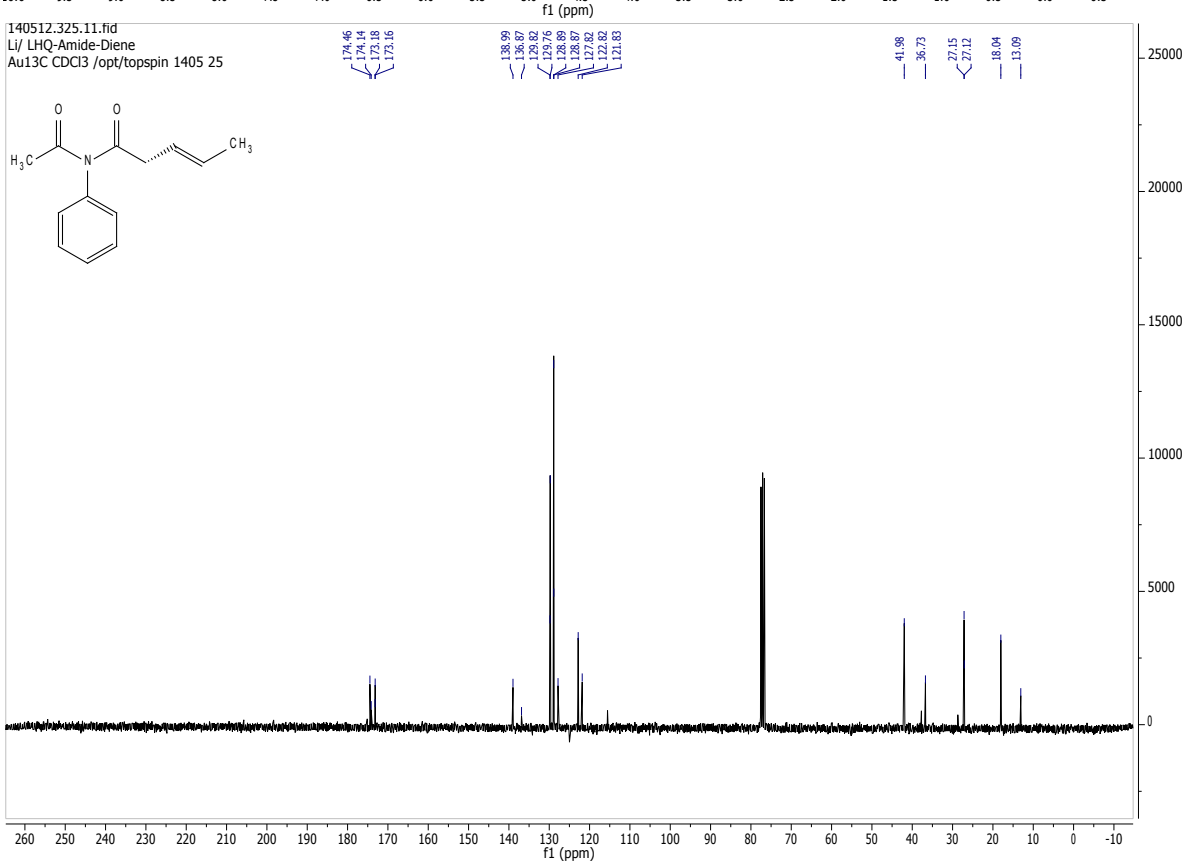


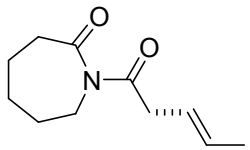

5.28
5.27
5.26
5.26
5.25
5.25
5.25
5.24
5.23
5.23
4.39
3.71
3.33
3.31
2.38
1.70
1.58
0.91
2.00
2.98
1.82
3.04
2.90
2.90
f1 (ppm)

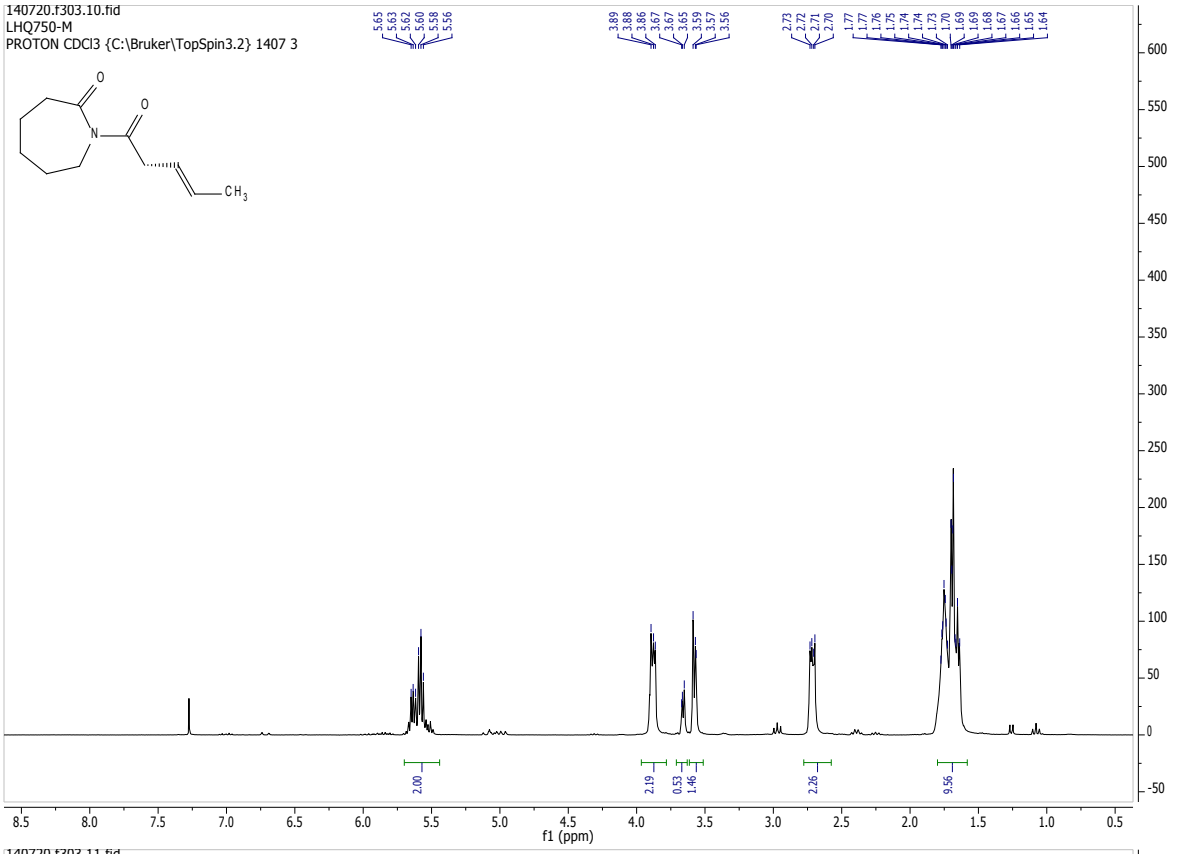

174.14
172.83
169.10
136.24
115.40
52.53
45.68
37.29
26.35
25.68
18.05
f1 (ppm)

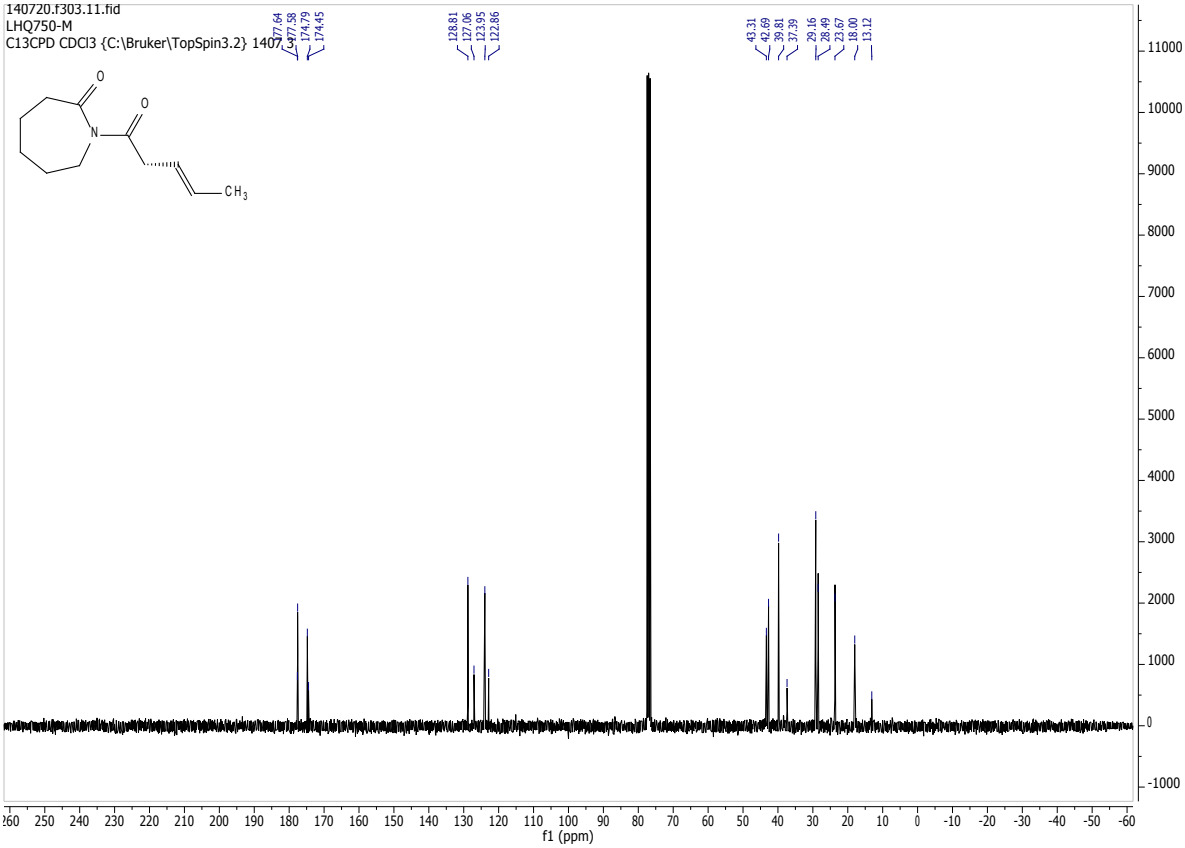


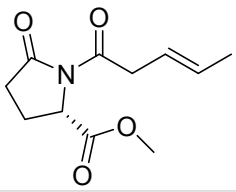

140512.325.10.fid
Li/ LHQ-Amide-Diene
Au1H CDCl3 /opt/topspin 1405 25
f1 (ppm)

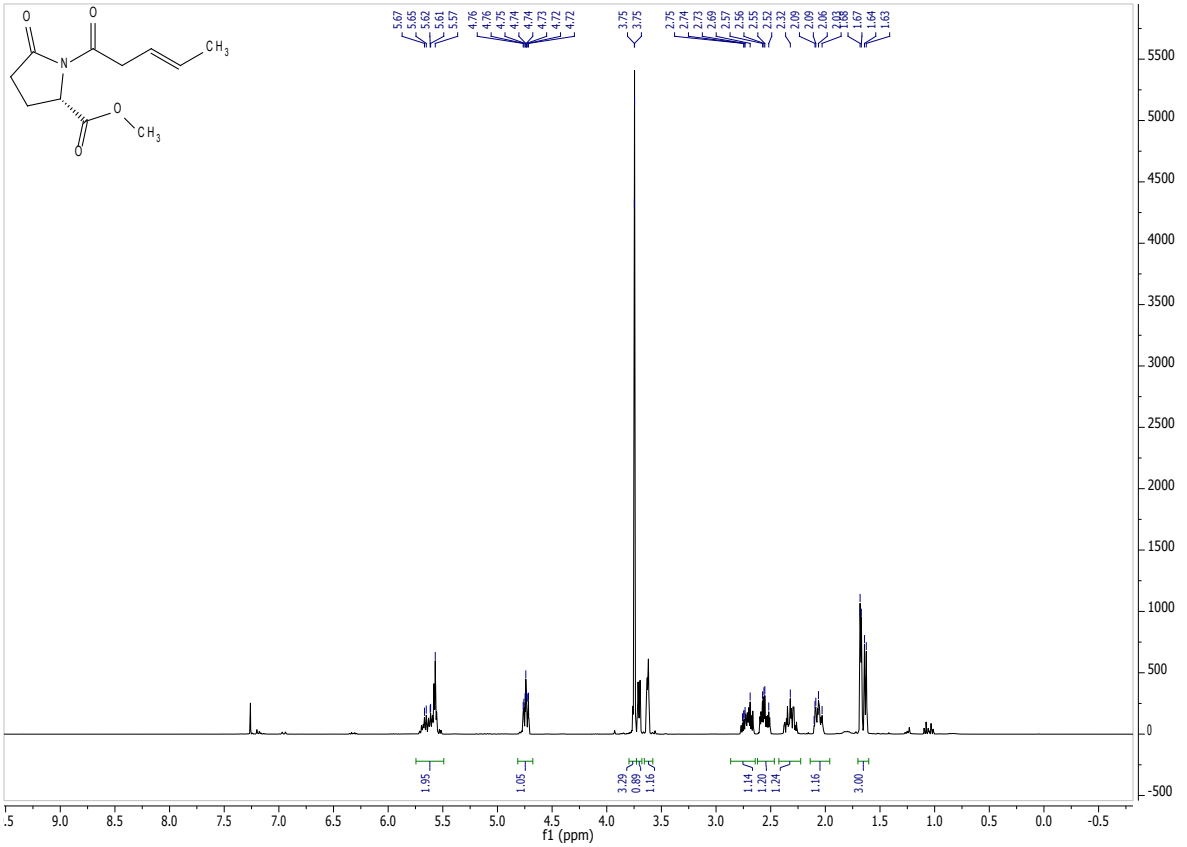

140512.325.11.fid
Li/ LHQ-Amide-Diene
Au13C CDCl3 /opt/topspin 1405 25
f1 (ppm)

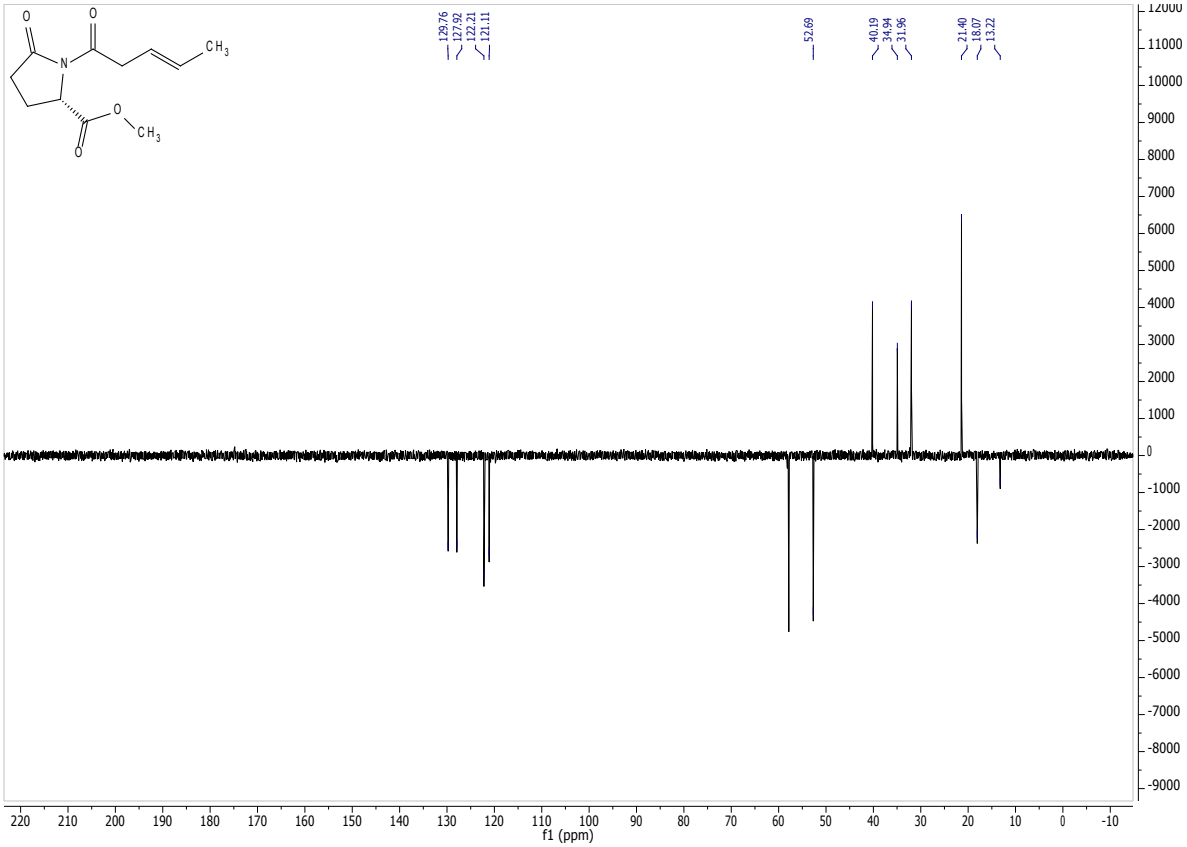


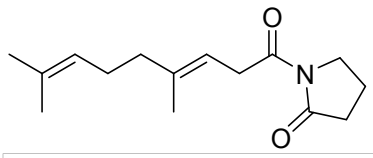

140720.f303.10.fid
LHQ750-M
PROTON CDCl3 {C:\Bruker\TopSpin3.2} 1407 3
5.65
5.63
5.62
5.60
5.58
5.56
3.89
3.88
3.86
3.67
3.67
3.65
3.59
3.57
3.56
2.73
2.72
2.71
2.70
1.77
1.77
1.76
1.75
1.74
1.74
1.73
1.70
1.69
1.69
1.68
1.67
1.66
1.65
1.64
2.00
2.19
0.53
1.46
2.26
9.56
f1 (ppm)

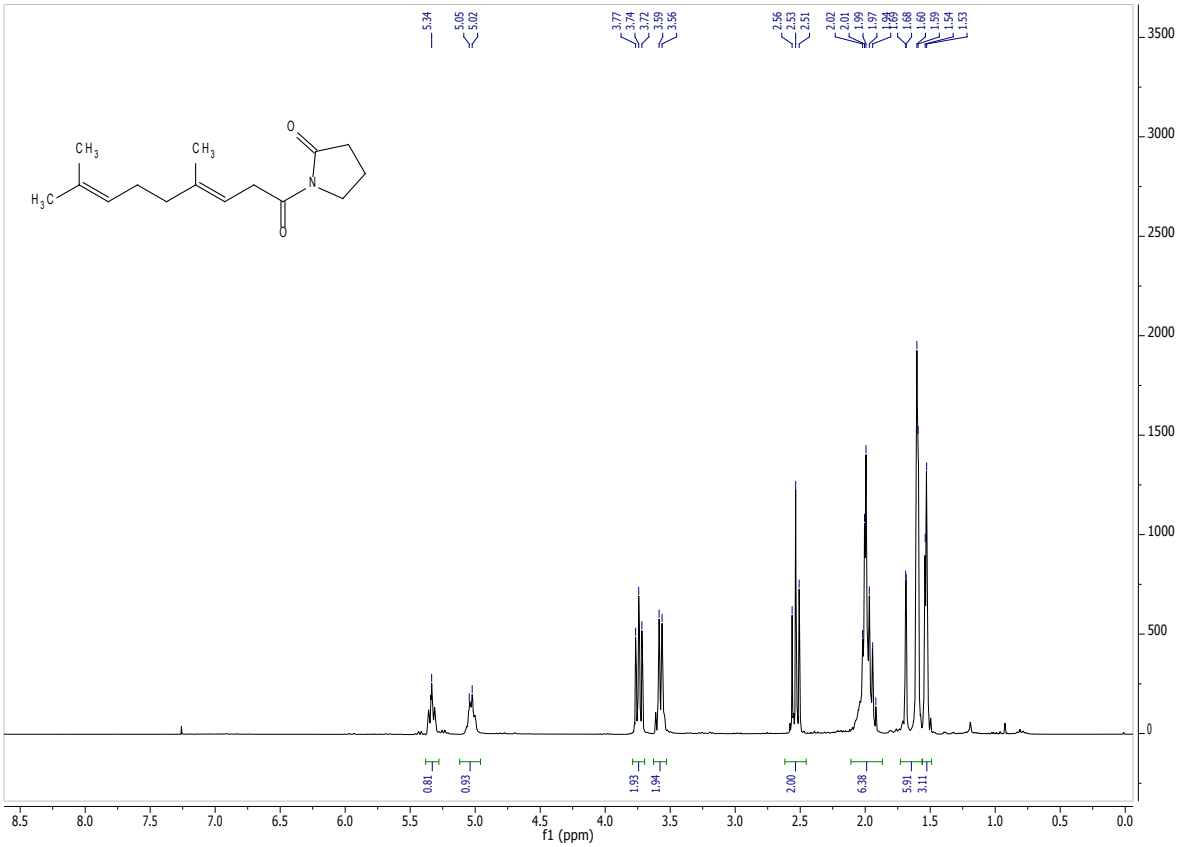

140720.f303.11.fid
LHQ750-M
C13CPD CDCl3 {C:\Bruker\TopSpin3.2} 1407 3
177.64
177.58
174.79
174.45
128.81
127.06
123.95
122.86
43.31
42.69
39.81
37.39
29.16
28.49
23.67
18.00
13.12
f1 (ppm)

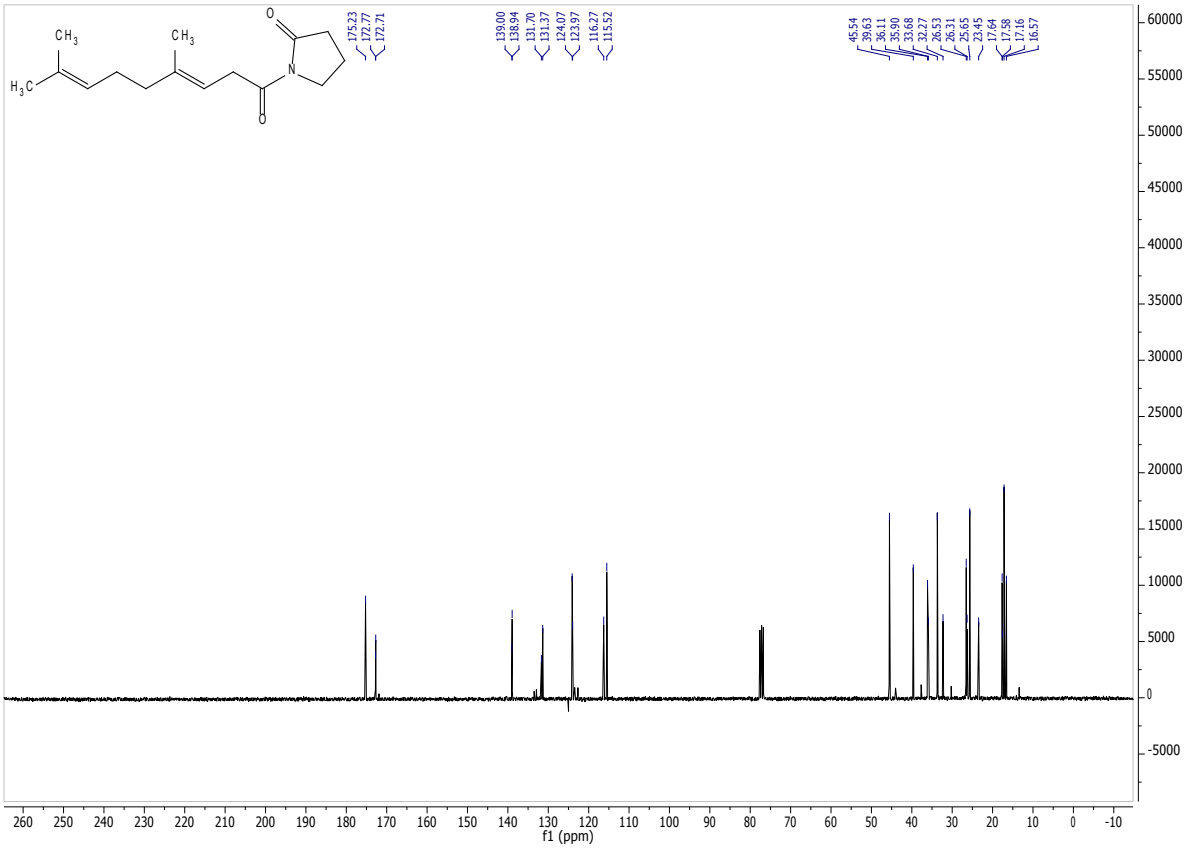


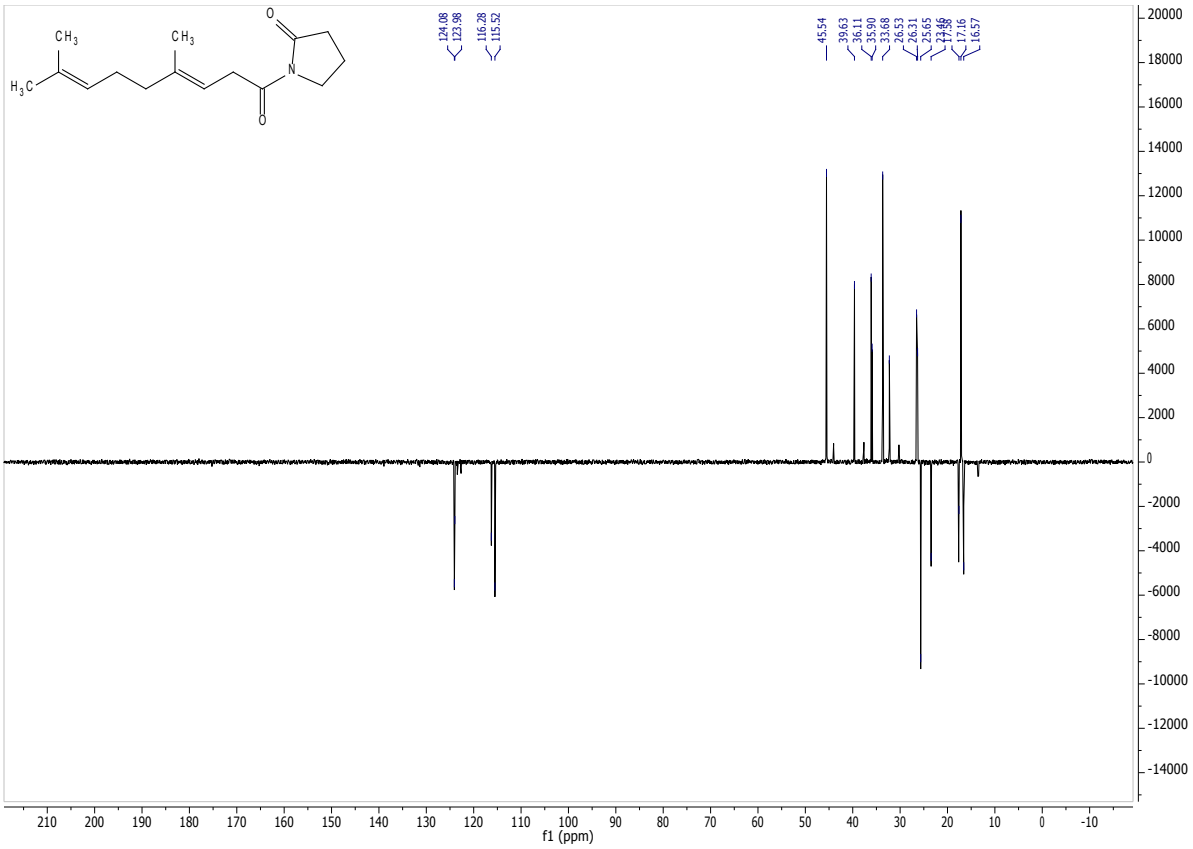

5.67
5.65
5.62
5.61
5.57
4.76
4.76
4.75
4.74
4.74
4.73
4.72
4.72
3.75
3.75
2.75
2.74
2.73
2.69
2.57
2.56
2.55
2.32
2.32
2.09
2.09
2.06
2.03
1.68
1.67
1.64
1.63
1.95
1.05
3.29
0.89
1.16
1.14
1.20
1.24
1.16
3.00
f1 (ppm)

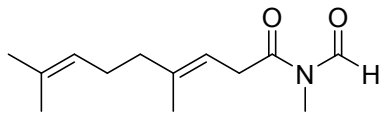

129.76
127.92
122.21
121.11
52.69
40.19
34.94
31.96
21.40
18.07
13.22
f1 (ppm)

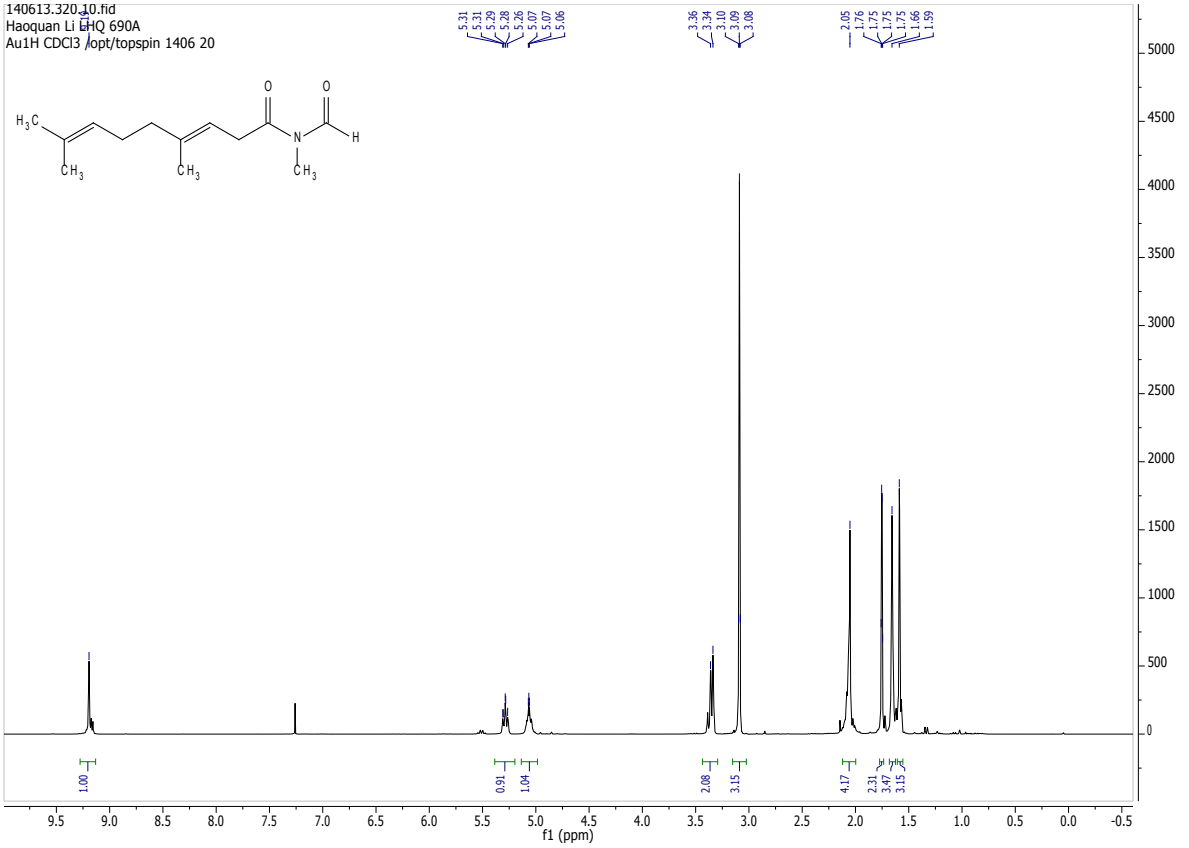


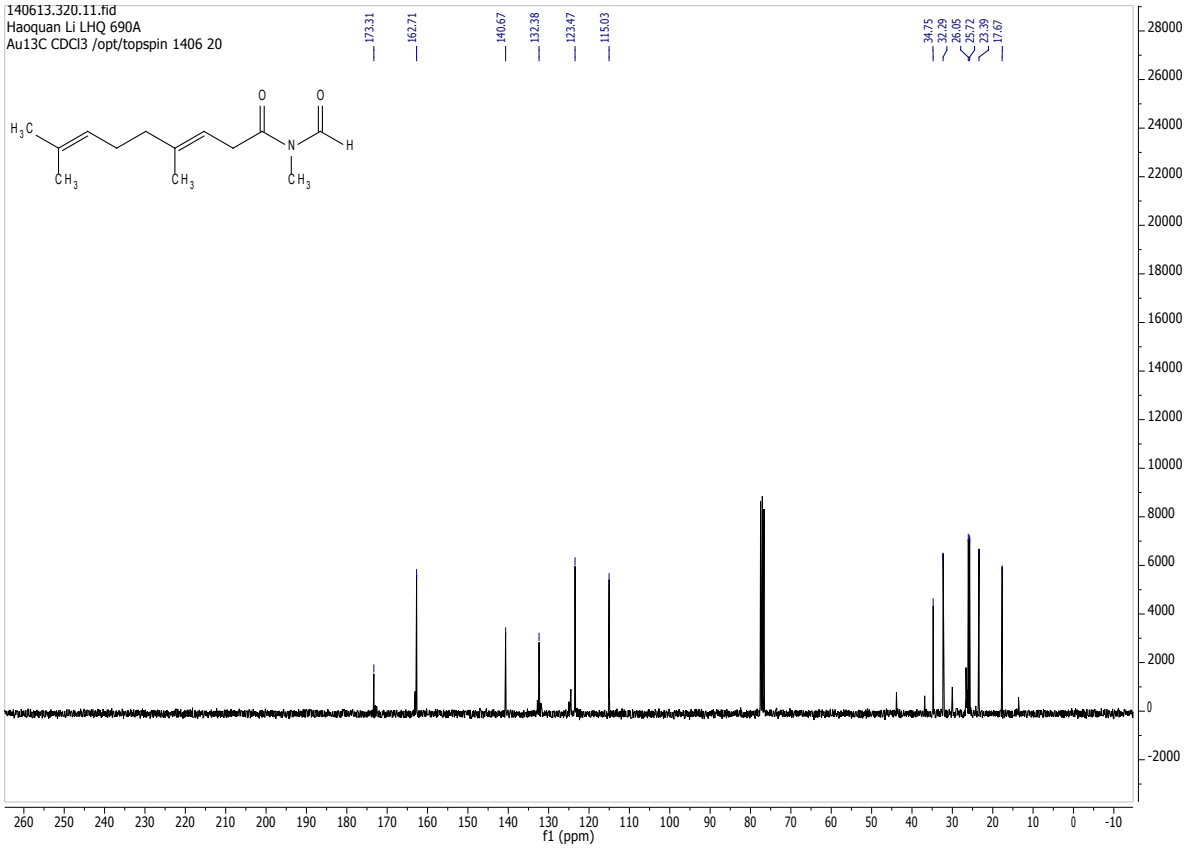

5.34
5.05
5.02
3.77
3.74
3.72
3.59
3.56
2.56
2.53
2.51
2.02
2.01
1.99
1.97
1.94
1.69
1.68
1.60
1.59
1.54
1.53
0.81
0.93
1.93
1.94
2.00
6.38
5.91
3.11
f1 (ppm)

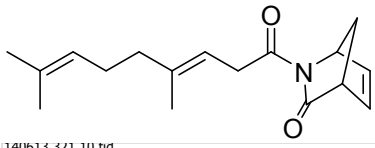

175.23
172.77
172.71
139.00
138.94
131.70
131.37
124.07
123.97
116.27
115.52
45.54
39.63
36.11
35.90
33.68
32.27
26.53
26.31
25.65
23.45
17.64
17.58
17.16
16.57
f1 (ppm)

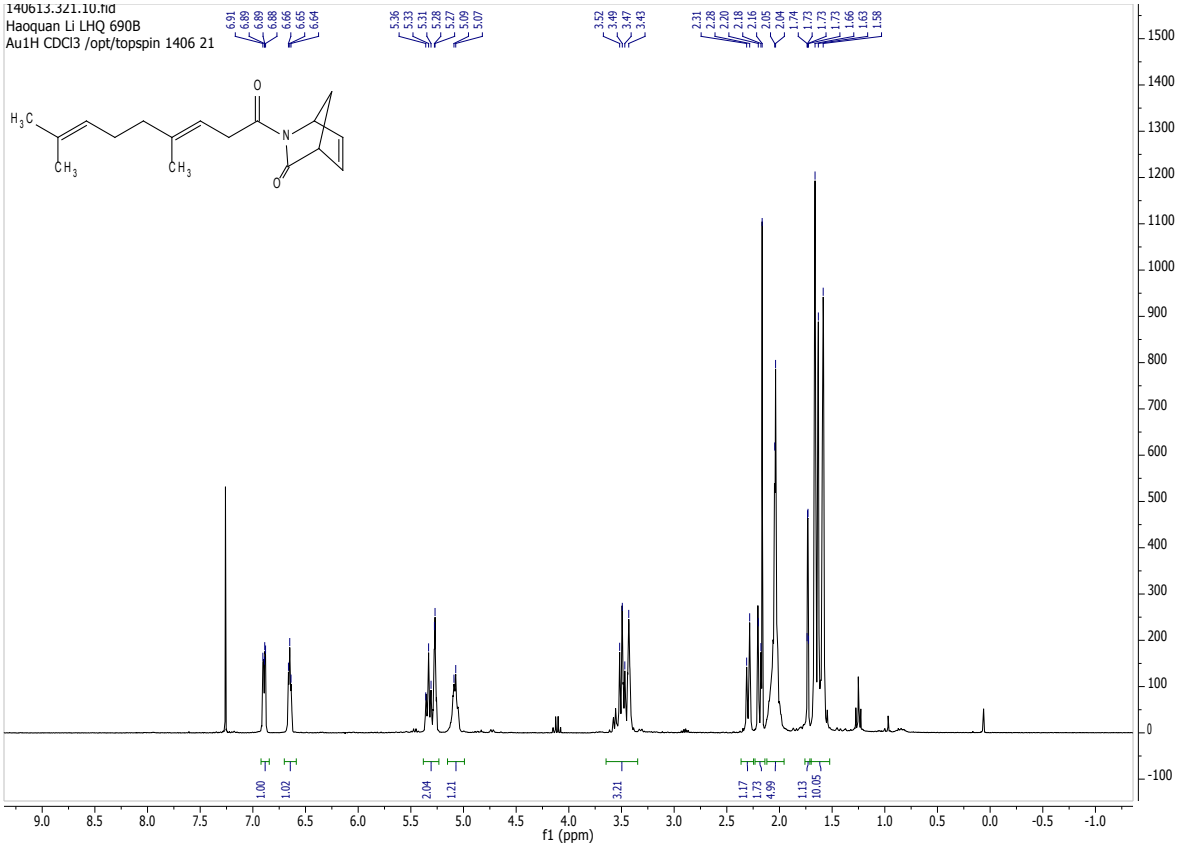

CH3
CH3
H3C
O
N
O
124.08
123.98
116.28
115.52
45.54
39.63
36.11
35.90
33.68
26.53
26.31
25.65
17.16
16.57
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
-4000
-6000
-8000
-10000
-12000
-14000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

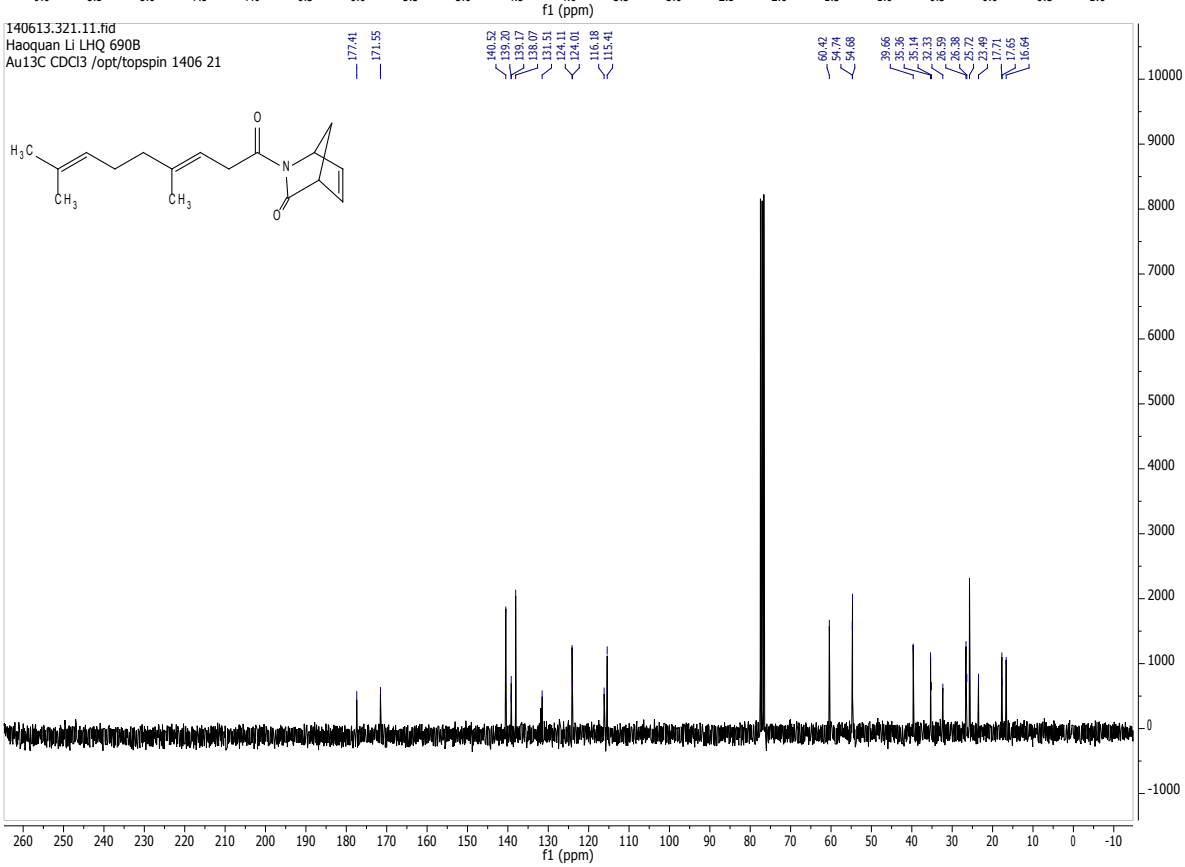


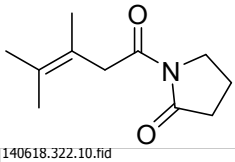

140613.320.10.fid
Haoquan Li LHQ 690A
Au1H CDCl3 /opt/topspin 1406 20
5.31
5.31
5.29
5.28
5.26
5.07
5.07
5.06
3.36
3.34
3.10
3.09
3.08
2.05
1.76
1.75
1.75
1.75
1.66
1.59
1.00
0.91
1.04
2.08
3.15
4.17
2.31
3.47
3.15
f1 (ppm)

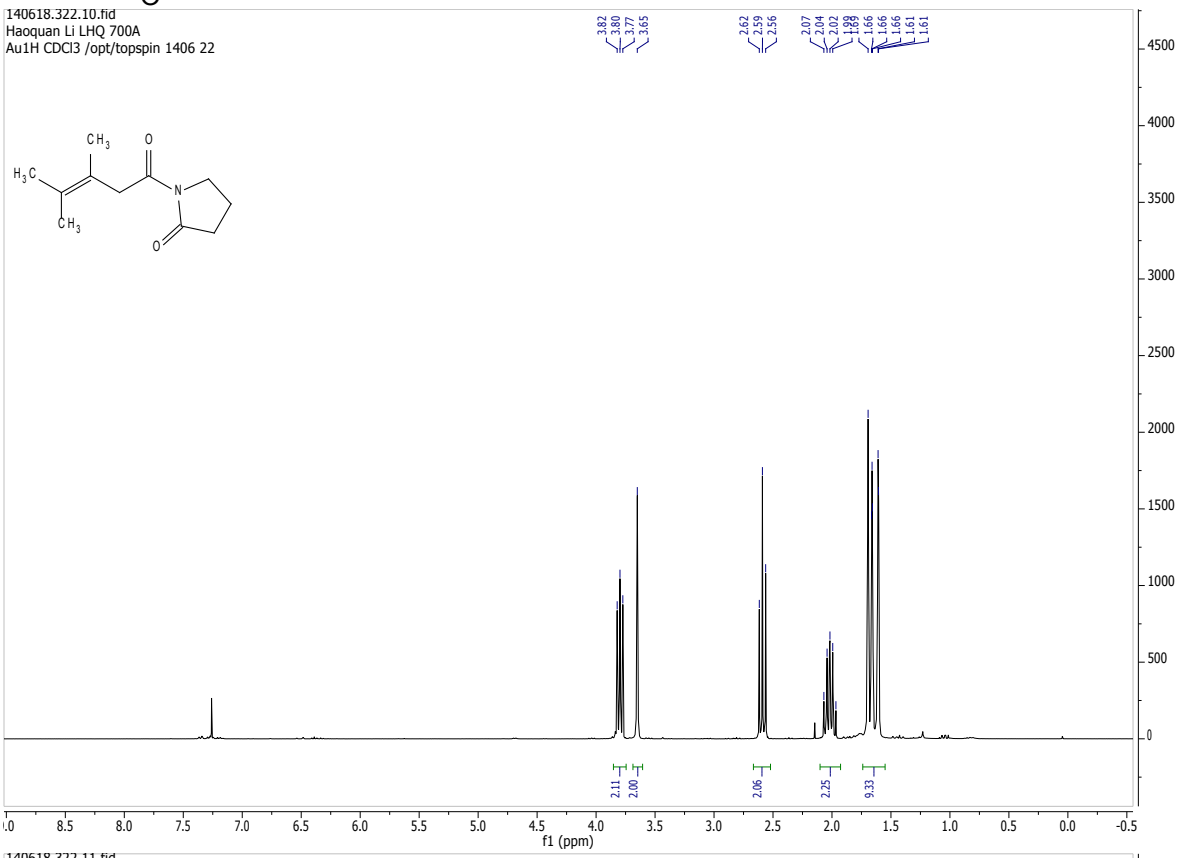

140613.320.11.fid
Haoquan Li LHQ 690A
Au13C CDCl3 /opt/topspin 1406 20
173.31
162.71
140.67
132.38
123.47
115.03
34.75
32.29
26.05
25.72
23.39
17.67
f1 (ppm)

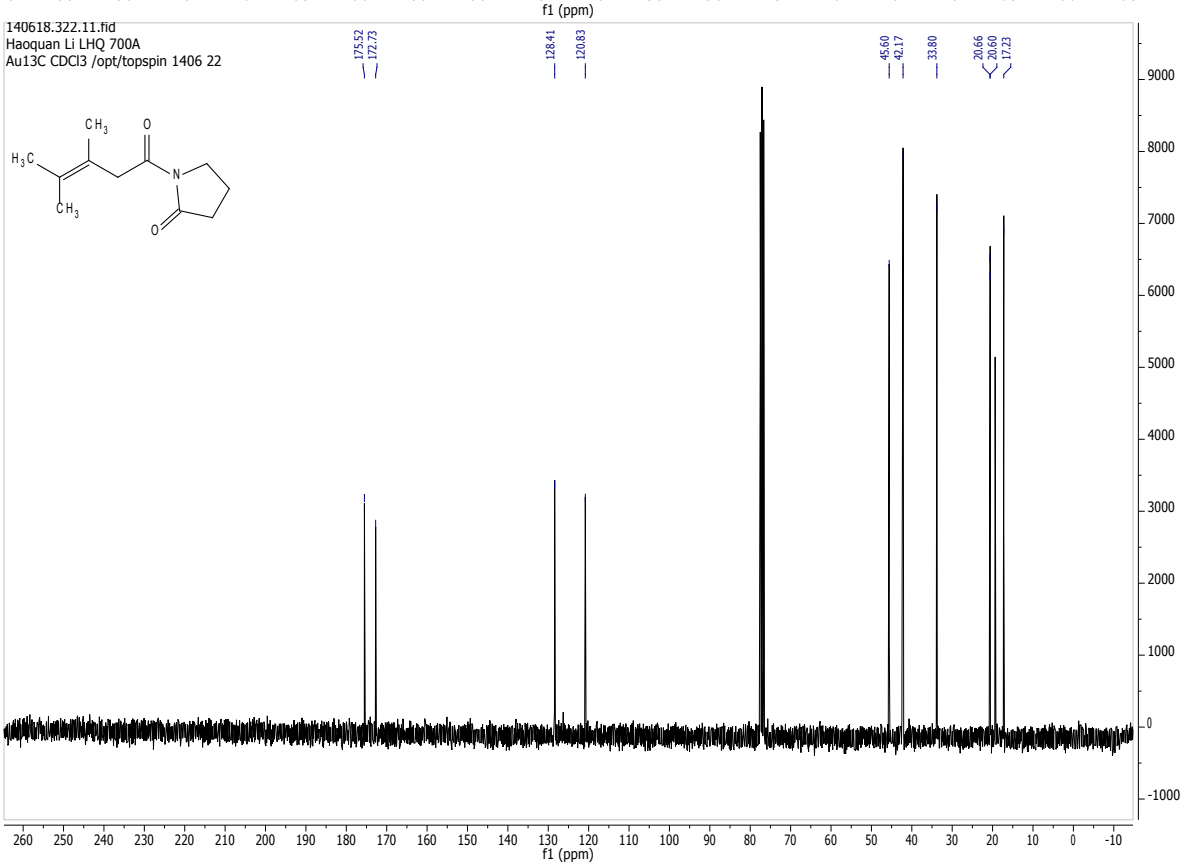


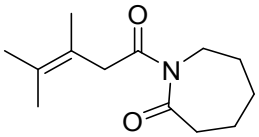

140613.321.10.fid
Haoquan Li LHQ 690B
Au1H CDCl3 /opt/topspin 1406 21
6.91 6.89 6.89 6.88 6.66 6.65 6.64
5.36 5.33 5.31 5.28 5.27 5.09 5.07
3.52 3.49 3.47 3.43
2.31 2.28 2.20 2.18 2.16 2.05 2.04 1.74 1.73 1.73 1.73 1.66 1.63 1.58
1.00 1.02 2.04 1.21 3.21 1.17 1.73 4.99 1.13 10.05
f1 (ppm)

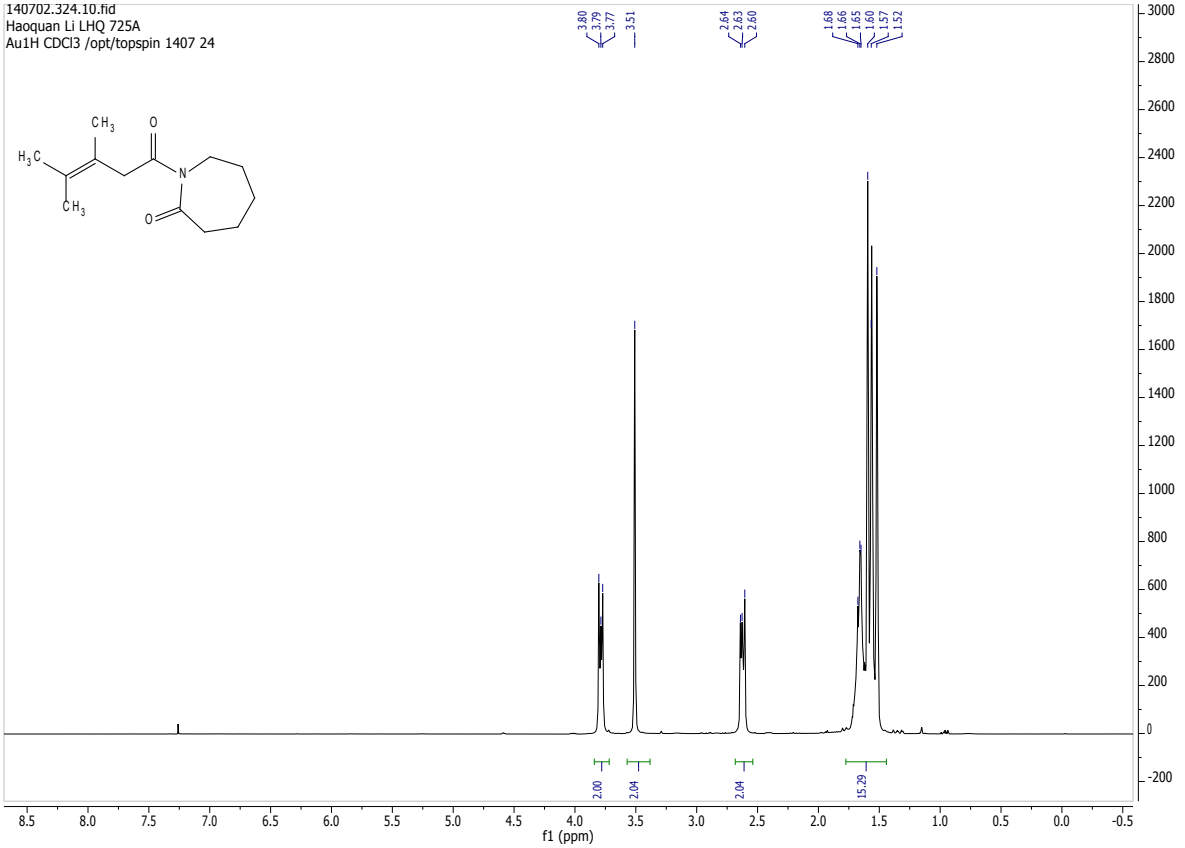

140613.321.11.fid
Haoquan Li LHQ 690B
Au13C CDCl3 /opt/topspin 1406 21
177.41 171.55 140.52 139.20 139.17 138.07 131.51 124.11 124.01 116.18 115.41 60.42 54.74 54.68 39.66 35.36 35.14 32.33 26.59 26.38 25.72 23.49 17.71 17.65 16.64
f1 (ppm)

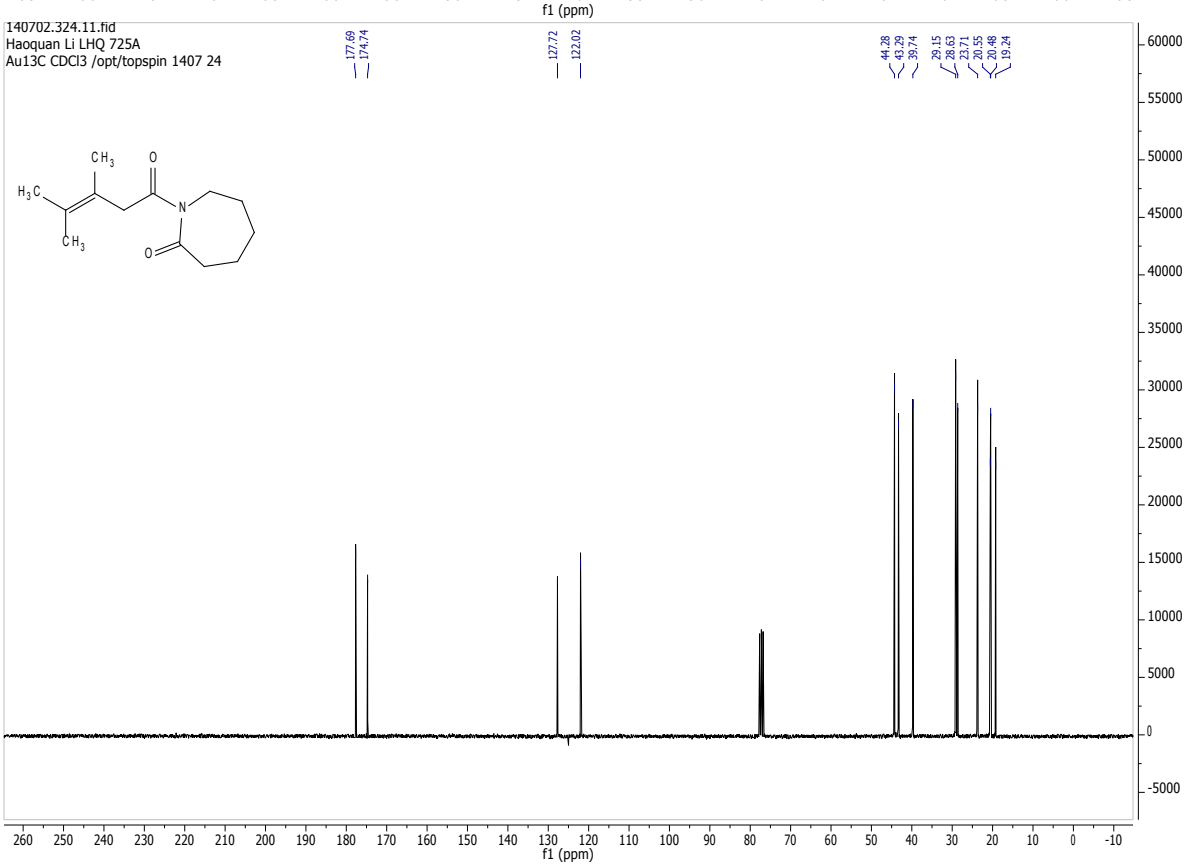


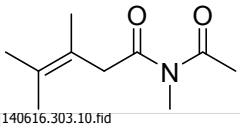

140618.322.10.fid
Haoquan Li LHQ 700A
Au1H CDCl3 /opt/topspin 1406 22
3.82
3.80
3.77
3.65
2.62
2.59
2.56
2.07
2.04
2.02
1.99
1.89
1.66
1.66
1.66
1.61
1.61
2.11
2.00
2.06
2.25
9.33
f1 (ppm)

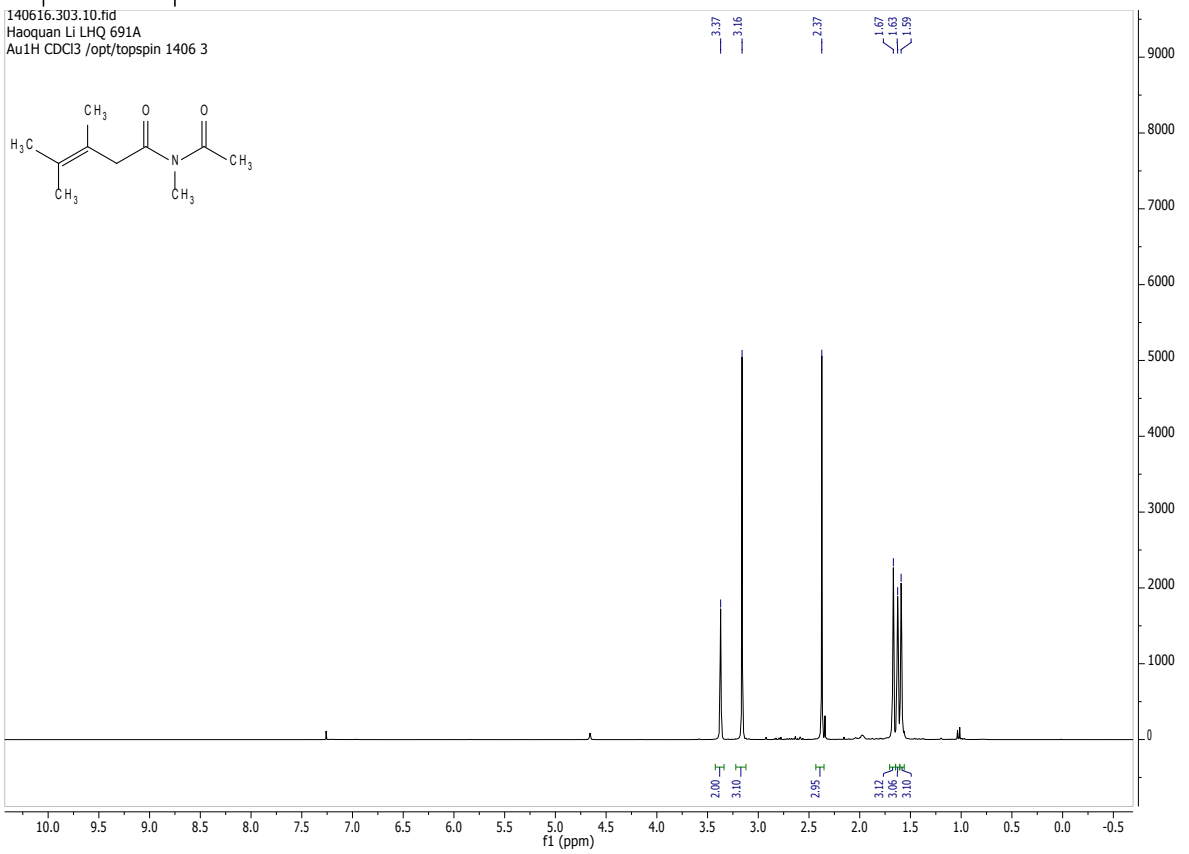

140618.322.11.fid
Haoquan Li LHQ 700A
Au13C CDCl3 /opt/topspin 1406 22
175.52
172.73
128.41
120.83
45.60
42.17
33.80
20.66
20.60
17.23
f1 (ppm)

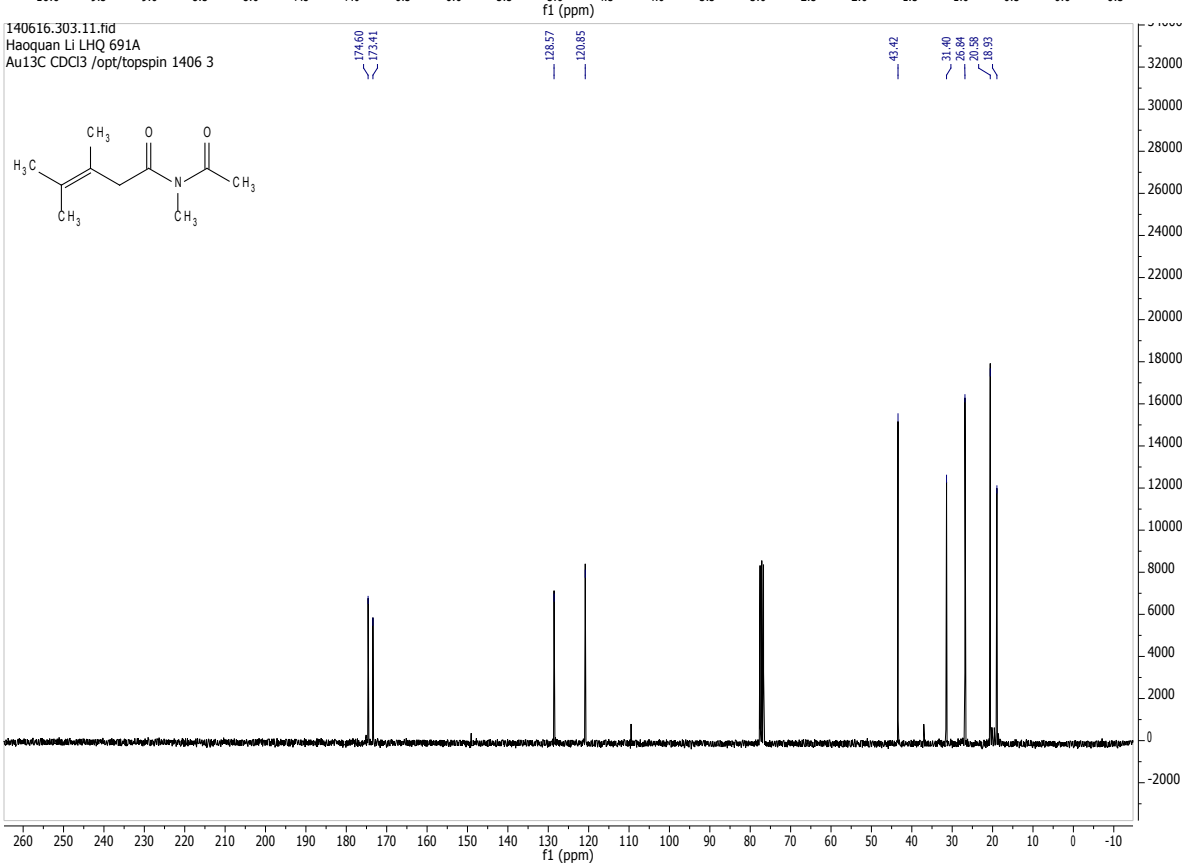


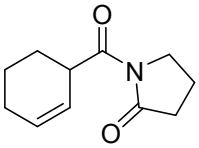

140702.324.10.fid
Haoquan Li LHQ 725A
Au1H CDCl3 /opt/topspin 1407 24
3.80
3.79
3.77
3.51
2.64
2.63
2.60
1.68
1.66
1.65
1.60
1.57
1.52
2.00
2.04
2.04
15.29
f1 (ppm)

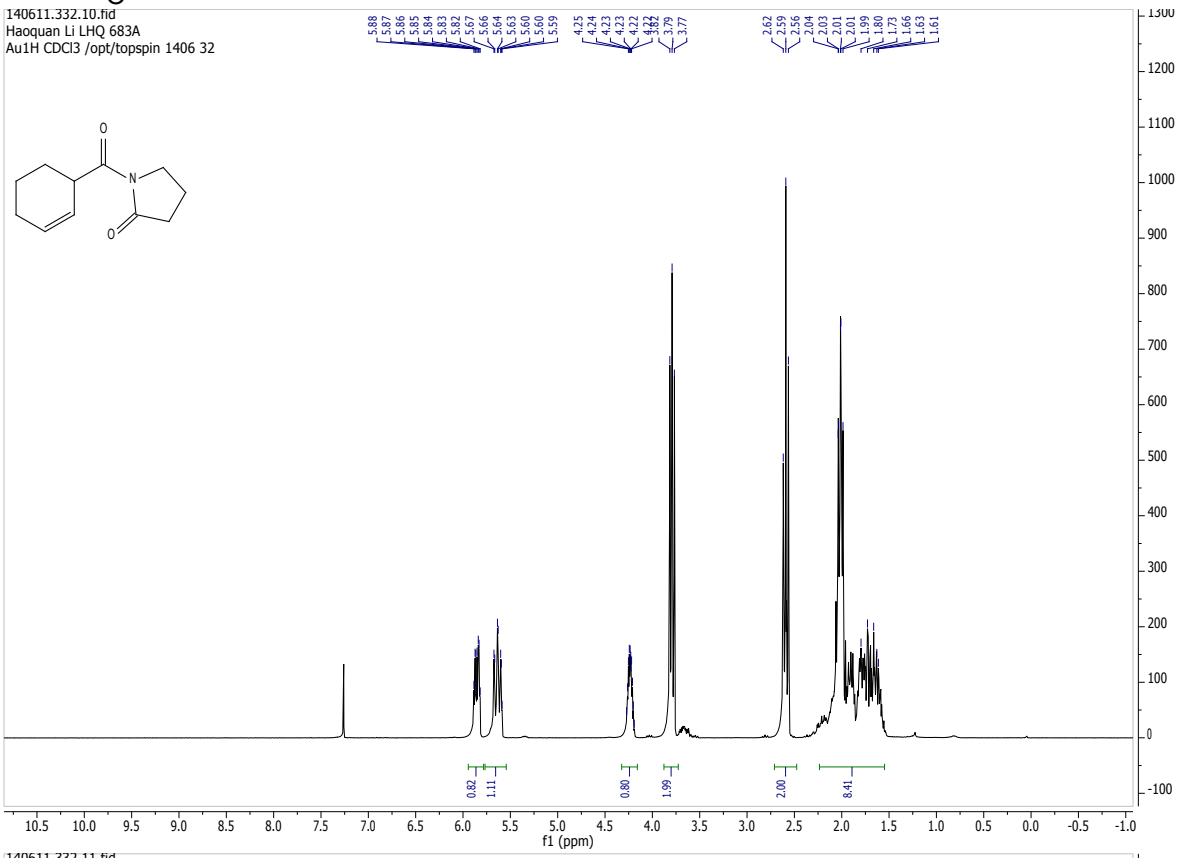

140702.324.11.fid
Haoquan Li LHQ 725A
Au13C CDCl3 /opt/topspin 1407 24
177.69
174.74
127.72
122.02
44.28
43.29
39.74
29.15
28.63
23.71
20.55
20.48
19.24
f1 (ppm)

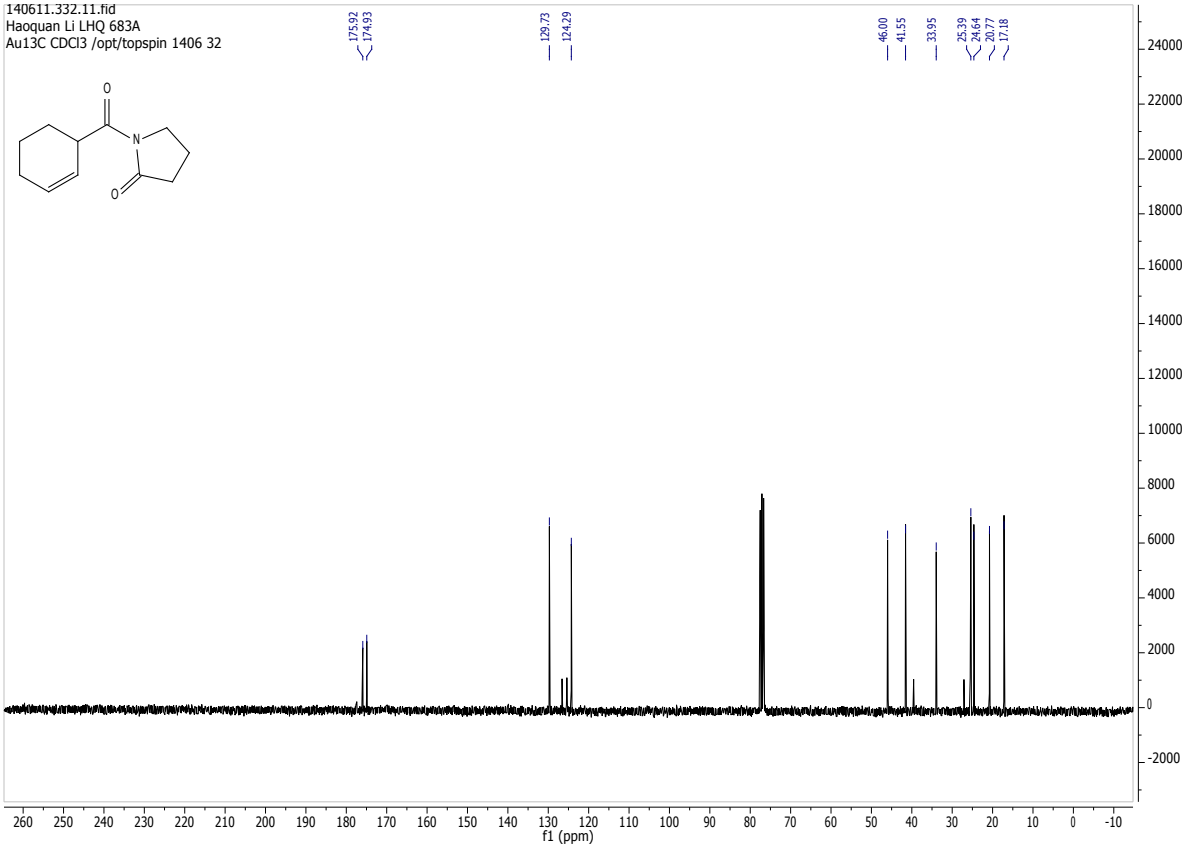


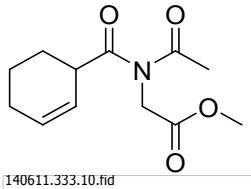

140616.303.10.fid
Haoquan Li LHQ 691A
Au1H CDCl3 /opt/topspin 1406 3
3.37
3.16
2.37
1.67
1.63
1.59
2.00
3.10
2.95
3.12
3.06
3.10
f1 (ppm)

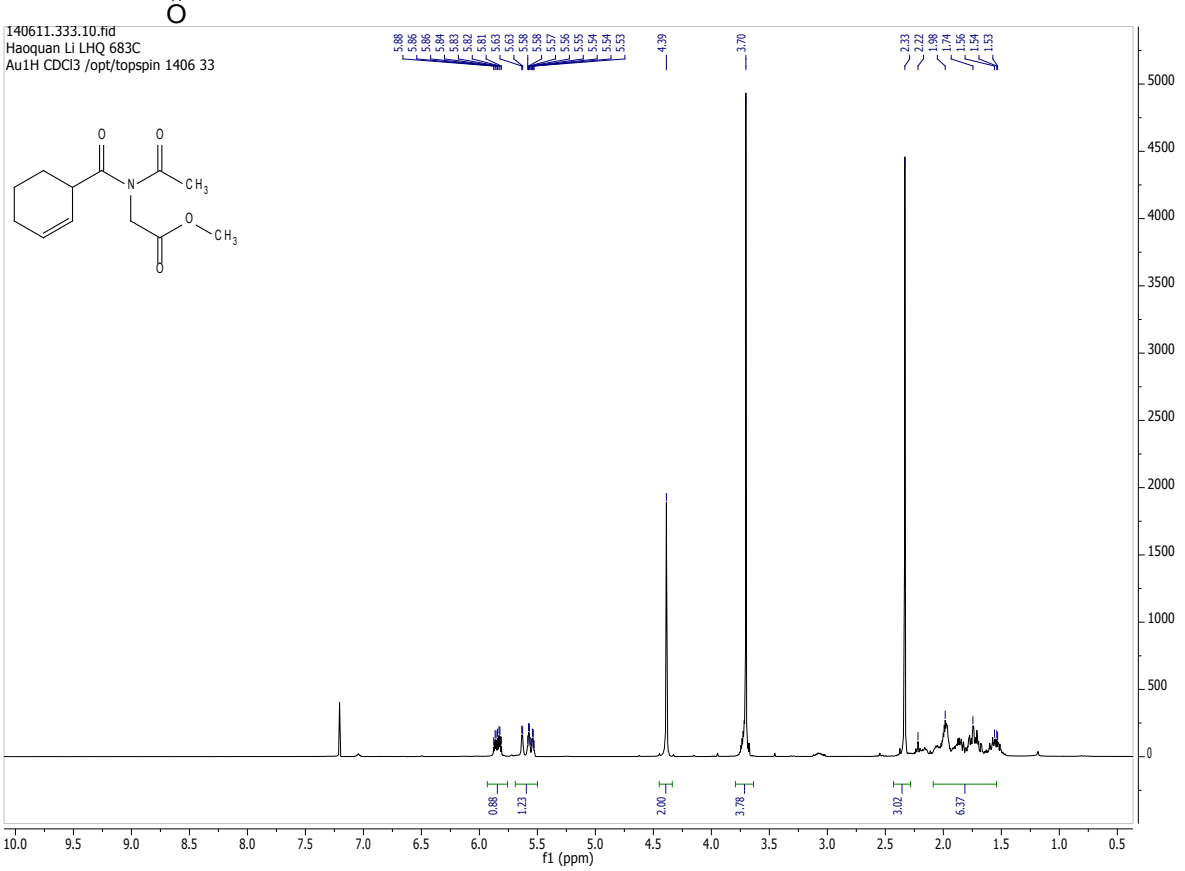

140616.303.11.fid
Haoquan Li LHQ 691A
Au13C CDCl3 /opt/topspin 1406 3
174.60
173.41
128.57
120.85
43.42
31.40
26.84
20.58
18.93
f1 (ppm)

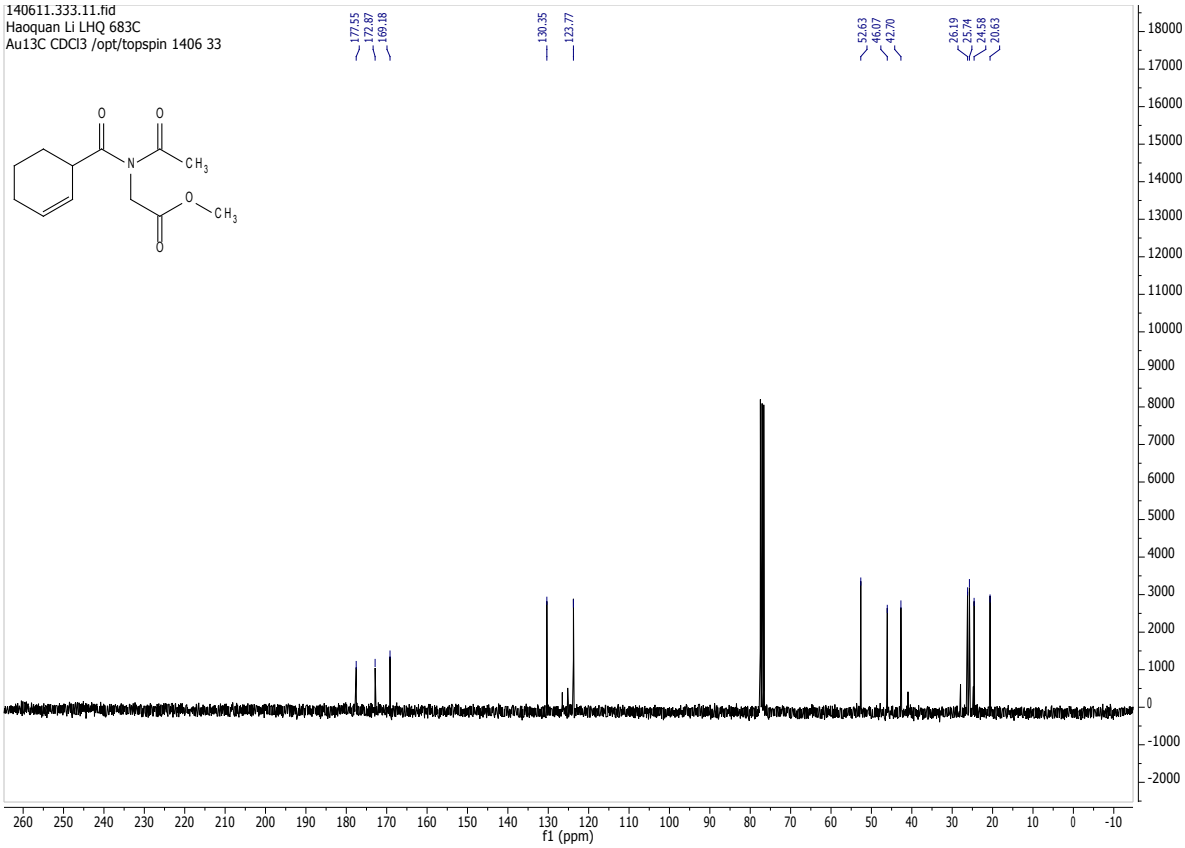



140611.332.10.fid
Haoquan Li LHQ 683A
Au1H CDCl3 /opt/topspin 1406 32
5.88 5.87 5.86 5.85 5.84 5.83 5.82 5.67 5.66 5.64 5.63 5.60 5.60 5.59
4.25 4.24 4.23 4.23 4.22 4.22 3.79 3.77
2.62 2.59 2.56 2.04 2.03 2.01 2.01 1.99 1.80 1.77 1.66 1.63 1.61
0.82 1.11 0.80 1.99 2.00 8.41
f1 (ppm)


140611.332.11.fid
Haoquan Li LHQ 683A
Au13C CDCl3 /opt/topspin 1406 32
175.92 174.93 129.73 124.29 46.00 41.55 33.95 25.39 24.64 20.77 17.18
f1 (ppm)




140611.333.10.fid
Haoquan Li LHQ 683C
Au1H CDCl3 /opt/topspin 1406 33
5.88
5.86
5.86
5.84
5.83
5.82
5.81
5.63
5.63
5.58
5.58
5.57
5.56
5.55
5.54
5.54
5.53
4.39
3.70
2.33
2.22
1.98
1.74
1.56
1.54
1.53
0.88
1.23
2.00
3.78
3.02
6.37
f1 (ppm)


140611.333.11.fid
Haoquan Li LHQ 683C
Au13C CDCl3 /opt/topspin 1406 33
177.55
172.87
169.18
130.35
123.77
52.63
46.07
42.70
26.19
25.74
24.58
20.63
f1 (ppm)