SUPPORTING INFROMATION

Intramolecular Imidoylative Heck Reaction: Synthesis of Cyclic Ketoimines from Functionalized Isocyanide

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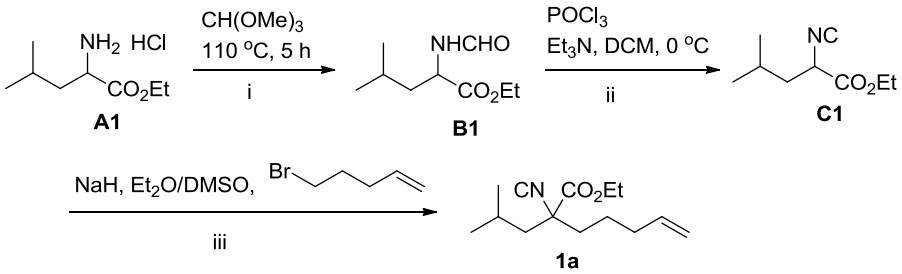
Contents

I. General Information

^{1}H NMR (400 MHz) and ^{13}C NMR (125 MHz) were registered on 400 M and 500 M spectrometers, respectively. Chemical shifts were reported in units (ppm) by assigning TMS resonance in the ^{1}H spectrum as 0.00 ppm, $CDCl_3$ resonance in the ^{13}C spectrum as 77.0 ppm. All coupling constants (*J* values) were reported in Hertz (Hz). NMR analysis was carried out at 298 K unless noted otherwise. HRMS was obtained on an ESI-LC-MS/MS spectrometer.

II. Preparation of Starting Materials

ethyl 2-isobutyl-2-isocyanohept-6-enoate (1a): [1,2]



(i) A 250 mL round bottom flask charged with 100 mL of $CH(OMe)_3$ (solvent) and **A1** (10 mmol, 1.95 g) was heated at 110 ^{o}C for 5 h. The solvent was removed under reduced pressure to afford the crude product **B1** for the next step without further purification.

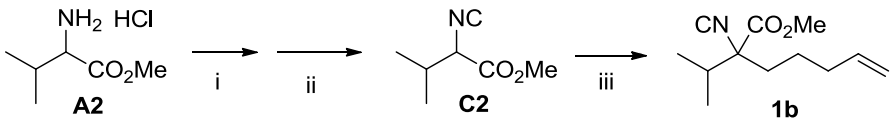
(ii) To a solution of crude **B1** (10 mmol, 1.0 equiv) and Et_3N (4 equiv) in 100 mL of dry DCM was added $POCl_3$ (1.2 equiv) dropwise in 30 min at -20 ^{o}C. The reaction mixture was stirred for 4 h. Then 10 mL of H_2O was added dropwise to the reaction mixture carefully at -20 ^{o}C. The crude reaction mixture was extracted with DCM (100 mL × 3) and washed with brine (100 mL). The organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography (petroleum ether : EtOAc = 8 : 1) to afford the product **C1** as colorless liquid in 85% yield.

(iii) An oven-dried 100 mL round bottom flask charged with a stir-bar and NaH (60%) (10 mmol, 2 equiv) was vacuumed and refilled with Ar for 3 times. A mixture solution of DMSO (2 mL) and Et_2O (50 mL) was added to the flask using a syringe. Then 5 mmol of **C1** (1.0 equiv) was added to the mixture dropwise in 10 min at room temperature and the reaction mixture was stirred for 30 min. Two equivalents of

5-bromopent-1-ene was added with a syringe and the reaction mixture was stirred for 30 min. Then 2 mL of H_2O was added dropwise to the reaction mixture carefully at room temperature. The crude reaction mixture was extracted with DCM (20 mL × 3) and washed with brine (20 mL). The organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography (petroleum ether : EtOAc = 16 : 1) to afford the product **1a** as colorless liquid in 91% yield. (new compound)

^{1}H NMR (400 MHz, $CDCl_3$): δ 5.78-5.68 (m, 1H), 5.02-4.95 (m, 2H), 4.25-4.20 (m, 2H), 2.07-2.01 (m, 2H), 1.92-1.81 (m, 3H), 1.75-1.65 (m, 3H), 1.30 (t, *J* = 7.2 Hz, 3H), 0.98 (d, *J* = 6.8 Hz, 3H), 0.84 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.3, 159.6, 137.4, 115.4, 67.6, 62.4, 47.1, 40.1, 32.9, 25.0, 23.7, 23.1, 22.3, 14.0; HRMS: calcd for $C_{14}H_{24}NO_2$ (M^++H) 238.1802; found 238.1804.

methyl 2-isocyano-2-isopropylhept-6-enoate (1b):



(i) A 250 mL round bottom flask charged with 50 mL of $CH(OMe)_3$ (solvent) and **A2** (5 mmol, 835 mg) was heated at 110 ^{o}C for 5 h. The solvent was removed under reduced pressure to afford the crude product **B2** for the next step without further purification.

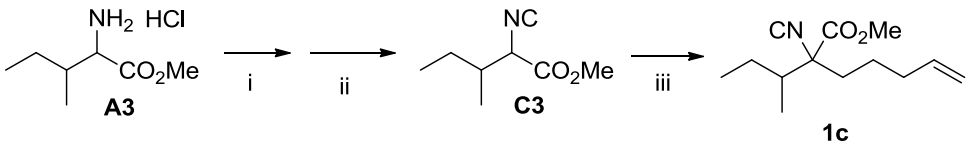
(ii) To a solution of crude **B2** (5 mmol, 1.0 equiv) and Et_3N (4 equiv) in 50 mL of dry DCM was added $POCl_3$ (1.2 equiv) dropwise in 30 min at -20 ^{o}C. The reaction mixture was stirred for 4 h. Then 10 mL of H_2O was added dropwise to the reaction mixture carefully at -20 ^{o}C. The crude reaction mixture was extracted with DCM (100 mL × 3) and washed with brine (100 mL). The organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography (petroleum ether : EtOAc = 8 : 1) to afford the product **C2** as colorless liquid in 78% yield.

(iii) An oven-dried 100 mL round bottom flask charged with a stir-bar and NaH (60%) (4 mmol, 2 equiv) was vacuumed and refilled with Ar for 3 times. A mixture solution of DMSO (1 mL) and Et_2O (20 mL) was added to the flask using a syringe. Then 2 mmol of **C2** (1.0 equiv) was added to the mixture dropwise in 10 min at room temperature and the reaction mixture was stirred for 30 min. Two equivalents of

5-bromopent-1-ene was added with a syringe and the reaction mixture was stirred for 30 min. Then 2 mL of H_2O was added dropwise to the reaction mixture carefully at room temperature. The crude reaction mixture was extracted with DCM (20 mL × 3) and washed with brine (20 mL). The organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography (petroleum ether : EtOAc = 16 : 1) to afford the product **1b** as colorless liquid in 87% yield. (new compound)

^{1}H NMR (400 MHz, $CDCl_3$): δ 5.74-5.66 (m, 1H), 5.00-4.92 (m, 2H), 3.76 (s, 3H), 2.13-2.01 (m, 3H), 1.81-1.77 (m, 2H), 1.66-1.59 (m, 1H), 1.23-1.16 (m, 1H), 1.01 (d, *J* = 6.8 Hz, 3H), 0.90 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.5, 159.5, 137.4, 115.4, 53.0, 36.6, 35.6, 33.0, 23.6, 17.8, 16.3; HRMS: calcd for $C_{12}H_{20}NO_2$ (M^++H) 210.1489; found 210.1490.

methyl 2-(sec-butyl)-2-isocyanohept-6-enoate (1c):



(i) A 250 mL round bottom flask charged with 50 mL of $CH(OMe)_3$ (solvent) and **A3** (5 mmol, 905 mg) was heated at 110 ^{o}C for 5 h. The solvent was removed under reduced pressure to afford the crude product **B3** for the next step without further purification.

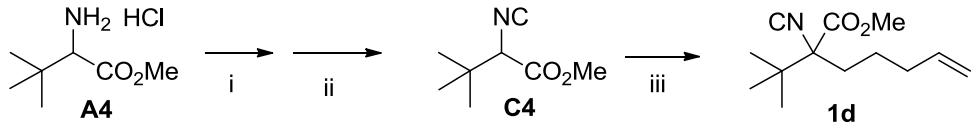
(ii) To a solution of crude **B3** (5 mmol, 1.0 equiv) and Et_3N (4 equiv) in 50 mL of dry DCM was added $POCl_3$ (1.2 equiv) dropwise in 30 min at -20 ^{o}C. The reaction mixture was stirred for 4 h. Then 10 mL of H_2O was added dropwise to the reaction mixture carefully at -20 ^{o}C. The crude reaction mixture was extracted with DCM (100 mL × 3) and washed with brine (100 mL). The organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography (petroleum ether : EtOAc = 8 : 1) to afford the product **C3** as colorless liquid in 89% yield.

(iii) An oven-dried 100 mL round bottom flask charged with a stir-bar and NaH (60%) (4 mmol, 2 equiv) was vacuumed and refilled with Ar for 3 times. A mixture solution of DMSO (1 mL) and Et_2O (20 mL) was added to the flask using a syringe. Then 2 mmol of **C3** (1.0 equiv) was added to the mixture dropwise in 10 min at room temperature and the reaction mixture was stirred for 30 min. Two equivalents of

5-bromopent-1-ene was added with a syringe and the reaction mixture was stirred for 30 min. Then 2 mL of H_2O was added dropwise to the reaction mixture carefully at room temperature. The crude reaction mixture was extracted with DCM (20 mL × 3) and washed with brine (20 mL). The organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography (petroleum ether : EtOAc = 16 : 1) to afford the product **1c** as colorless liquid in 76% yield. (new compound)

^{1}H NMR (400 MHz, $CDCl_3$): δ 5.77-5.70 (m, 1H), 5.03-4.95 (m, 2H), 3.78 (s, 3H), 2.08-2.03 (m, 2H), 1.89-1.70 (m, 3H), 1.69-1.62 (m, 1H), 1.32-1.17 (m, 1H), 1.01 (d, *J* = 6.8 Hz, 2H), 0.96-0.88 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.8, 159.4, 137.5, 115.4, 73.3, 53.0, 42.1, 36.8, 33.1, 25.0, 23.5, 12.5, 11.8; HRMS: calcd for $C_{13}H_{22}NO_2$ (M^++H) 224.1645; found 224.1649.

methyl 2-(tert-butyl)-2-isocyanohept-6-enoate (1d):



(i) A 250 mL round bottom flask charged with 50 mL of $CH(OMe)_3$ (solvent) and **A4** (5 mmol, 905 mg) was heated at 110 ^{o}C for 5 h. The solvent was removed under reduced pressure to afford the crude product **B4** for the next step without further purification.

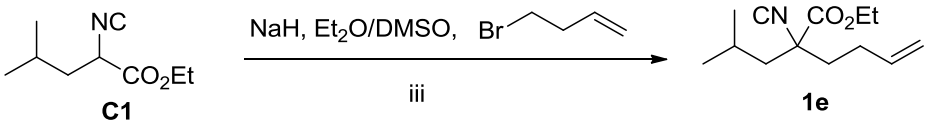
(ii) To a solution of crude **B4** (5 mmol, 1.0 equiv) and Et_3N (4 equiv) in 50 mL of dry DCM was added $POCl_3$ (1.2 equiv) dropwise in 30 min at -20 ^{o}C. The reaction mixture was stirred for 4 h. Then 10 mL of H_2O was added dropwise to the reaction mixture carefully at -20 ^{o}C. The crude reaction mixture was extracted with DCM (100 mL × 3) and washed with brine (100 mL). The organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography (petroleum ether : EtOAc = 8 : 1) to afford the product **C4** as colorless liquid in 65% yield.

(iii) An oven-dried 100 mL round bottom flask charged with a stir-bar and NaH (60%) (4 mmol, 2 equiv) was vacuumed and refilled with Ar for 3 times. A mixture solution of DMSO (1 mL) and Et_2O (20 mL) was added to the flask using a syringe. Then 2 mmol of **C4** (1.0 equiv) was added to the mixture dropwise in 10 min at room temperature and the reaction mixture was stirred for 30 min. Two equivalents of

5-bromopent-1-ene was added with a syringe and the reaction mixture was stirred for 30 min. Then 2 mL of H_2O was added dropwise to the reaction mixture carefully at room temperature. The crude reaction mixture was extracted with DCM (20 mL × 3) and washed with brine (20 mL). The organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography (petroleum ether : EtOAc = 16 : 1) to afford the product **1d** as colorless liquid in 51% yield. (new compound)

^{1}H NMR (400 MHz, $CDCl_3$): δ 5.82-5.72 (m, 1H), 5.05-4.97 (m, 2H), 3.79 (s, 3H), 2.11-1.99 (m, 3H), 1.75-1.64 (m, 2H), 1.28-1.16 (m, 1H), 1.07 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.1, 159.8, 138.0, 115.6, 76.1, 53.1, 38.3, 33.5, 32.9, 25.9, 24.6; HRMS: calcd for $C_{13}H_{22}NO_2$ (M^++H) 224.1645; found 224.1642.

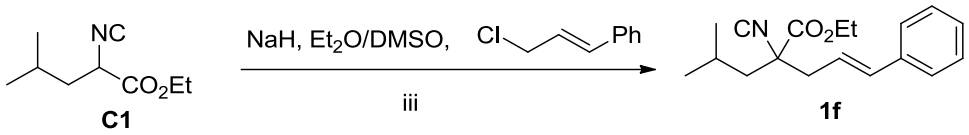
ethyl 2-isobutyl-2-isocyanohex-5-enoate (1e):



(iii) An oven-dried 100 mL round bottom flask charged with a stir-bar and NaH (60%) (4 mmol, 2 equiv) was vacuumed and refilled with Ar for 3 times. A mixture solution of DMSO (1 mL) and Et_2O (20 mL) was added to the flask using a syringe. Then 2 mmol of **C1** (1.0 equiv) was added to the mixture dropwise in 10 min at room temperature and the reaction mixture was stirred for 30 min. Two equivalents of 4-bromobut-1-ene was added with a syringe and the reaction mixture was stirred for 30 min. Then 2 mL of H_2O was added dropwise to the reaction mixture carefully at room temperature. The crude reaction mixture was extracted with DCM (20 mL × 3) and washed with brine (20 mL). The organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography (petroleum ether : EtOAc = 16 : 1) to afford the product **1e** as colorless liquid in 91% yield. (new compound)

^{1}H NMR (400 MHz, $CDCl_3$): δ 5.77-5.70 (m, 1H), 5.07-4.97 (m, 2H), 4.26-4.20 (m, 2H), 2.32-2.30 (m, 1H), 2.02-1.90 (m, 2H), 1.88-1.81 (m, 3H), 1.75-1.67 (m, 1H), 1.31 (t, *J* = 7.2 Hz, 3H), 1.00 (d, *J* = 6.4 Hz, 3H), 0.86 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.1, 159.8, 136.1, 116.0, 67.3, 62.5, 47.3, 39.9, 28.2, 25.0, 23.7, 22.3, 14.0; HRMS: calcd for $C_{13}H_{22}NO_2$ (M^++H) 224.1645; found 224.1647.

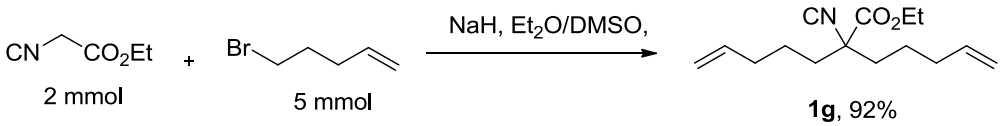
(E)-ethyl 2-isobutyl-2-isocyano-5-phenylpent-4-enoate (1f):



(iii) An oven-dried 100 mL round bottom flask charged with a stir-bar and NaH (60%) (4 mmol, 2 equiv) was vacuumed and refilled with Ar for 3 times. A mixture solution of DMSO (1 mL) and Et_2O (20 mL) was added to the flask using a syringe. Then 2 mmol of **C1** (1.0 equiv) was added to the mixture dropwise in 10 min at room temperature and the reaction mixture was stirred for 30 min. Two equivalents of (E)-(3-chloroprop-1-en-1-yl)benzene was added with a syringe and the reaction mixture was stirred for 30 min. Then 2 mL of H_2O was added dropwise to the reaction mixture carefully at room temperature. The crude reaction mixture was extracted with DCM (20 mL × 3) and washed with brine (20 mL). The organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography (petroleum ether : EtOAc = 16 : 1) to afford the product **1f** as yellow oil in 57% yield. (new compound)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37-7.23 (m, 5H), 6.51 (d, *J* = 16.0 Hz, 1H), 6.21-6.13 (m, 1H), 4.23 (q, *J* = 6.8 Hz, 2H), 2.80-2.64 (m, 2H), 1.97-1.89 (m, 2H), 1.83-1.79 (m, 1H), 1.28 (t, *J* = 6.8 Hz, 3H), 1.02 (d, J = 6.0 Hz, 3H), 0.89 (d, *J* = 6.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.2, 160.4, 136.9, 136.0, 128.9, 128.2, 126.8, 121.5, 67.9, 62.9, 46.8, 44.6, 25.4, 24.0, 22.6, 14.4; HRMS: calcd for $C_{18}H_{24}NO_2$ (M^++H) 286.1802; found 286.1798.

ethyl 2-isocyano-2-(pent-4-en-1-yl)hept-6-enoate (1g):

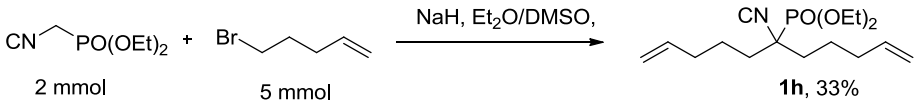


(iii) An oven-dried 100 mL round bottom flask charged with a stir-bar and NaH (60%) (6 mmol, 3 equiv) was vacuumed and refilled with Ar for 3 times. A mixture solution of DMSO (1 mL) and Et_2O (20 mL) was added to the flask using a syringe. Then 2 mmol of ethyl 2-isocyanoacetate (1.0 equiv) was added to the mixture dropwise in 10 min at room temperature and the reaction mixture was stirred for 30 min. Three equivalents of 5-bromopent-1-ene was added with a syringe and the reaction mixture was stirred for 30 min. Then 2 mL of H_2O was added dropwise to the

reaction mixture carefully at room temperature. The crude reaction mixture was extracted with DCM (20 mL × 3) and washed with brine (20 mL). The organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography (petroleum ether : EtOAc = 16 : 1) to afford the product **1g** as colorless liquid in 92% yield. (new compound)

^{1}H NMR (400 MHz, $CDCl_3$): δ 5.77-5.67 (m, 2H), 5.01-4.94 (m, 4H), 4.23 (q, *J* = 7.2 Hz, 2H), 2.07-2.02 (m, 4H), 1.92-1.85 (m, 2H), 1.78-1.59 (m, 4H), 1.37-1.27 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.8, 159.1, 137.4, 115.4, 68.3, 62.5, 38.5, 33.0, 23.2, 14.1; HRMS: calcd for $C_{15}H_{24}NO_2$ (M^++H) 250.1802; found 250.1802.

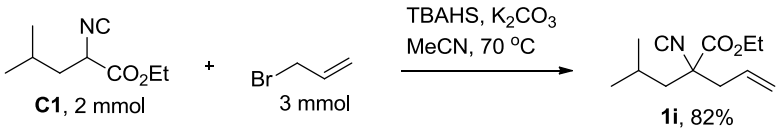
diethyl (6-isocyanoundeca-1,10-dien-6-yl)phosphonate (1h)



(iii) An oven-dried 100 mL round bottom flask charged with a stir-bar and NaH (60%) (6 mmol, 3 equiv) was vacuumed and refilled with Ar for 3 times. A mixture solution of DMSO (1 mL) and Et_2O (20 mL) was added to the flask using a syringe. Then 2 mmol of diethyl (isocyanomethyl)phosphonate (1.0 equiv) was added to the mixture dropwise in 10 min at room temperature and the reaction mixture was stirred for 30 min. Three equivalents of 5-bromopent-1-ene was added with a syringe and the reaction mixture was stirred for 30 min. Then 2 mL of H_2O was added dropwise to the reaction mixture carefully at room temperature. The crude reaction mixture was extracted with DCM (20 mL × 3) and washed with brine (20 mL). The organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography (petroleum ether : EtOAc = 8 : 1) to afford the product **1h** as colorless liquid in 33% yield. (new compound)

^{1}H NMR (400 MHz, $CDCl_3$): δ 5.82-5.72 (m, 2H), 5.05-4.97 (m, 4H), 4.27-4.19 (m, 4H), 2.10-2.05 (m, 4H), 1.93-1.75 (m, 4H), 1.65-1.54 (m, 4H), 1.36 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ159.6, 138.0, 115.8, 64.4 (d, *J* = 7.4 Hz), 62.0 (d, *J* = 155.4 Hz), 34.6, 33.7, 23.1 (d, *J* = 5.4 Hz), 16.8 (d, J = 5.6 Hz); HRMS: calcd for $C_{16}H_{29}NO_3P$ (M^++H) 314.1880; found 314.1885.

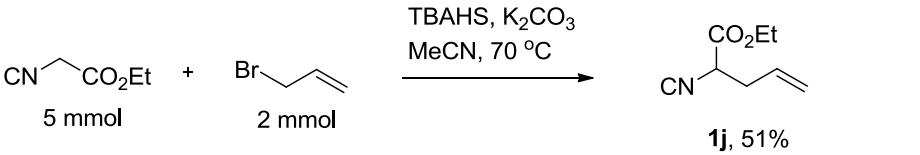
ethyl 2-isobutyl-2-isocyanopent-4-enoate (1i):



Compound **1i** was prepared according to similar route of literature reported methods[3]: To a solution of **C1** (2 mmol) in acetonitrile (20 mL) were added K_2CO_3 (8 mmol), TBAHS (0.2 mmol), and 3-bromoprop-1-ene (3 mmol). The mixture was heated at 70 ^oC until the reaction was completed, monitoring with TLC. Then the mixture was cooled and the solvent was removed and reduced pressure. H_2O (10 mL) and ethyl acetate (20 mL) were added into the mixture. The organic phase was seperated and the aqueous phase was extacted with ethyl acetate (20.0 mL × 3). The combined organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography (petroleum ether : EtOAc = 8 : 1) to afford **1i** as colorless liquid in 82% yield. (new compound)

^1H NMR (400 MHz, $CDCl_3$): δ 5.84-5.74 (m, 1H), 5.24-5.17 (m, 2H), 4.27-4.20 (m, 2H), 2.65-2.47 (m, 2H), 1.94-1.72 (m, 3H), 1.31 (t, *J* = 7.2 Hz, 3H), 1.01 (d, *J* = 6.4 Hz, 3H), 0.87 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.9, 159.8, 130.0, 120.9, 67.3, 62.5, 46.5, 44.9, 25.0, 23.6, 22.3, 14.1; HRMS: calcd for $C_{12}H_{20}NO_2$ (M^++H) 210.1489; found 210.1485.

ethyl 2-isocyanopent-4-enoate (1j):



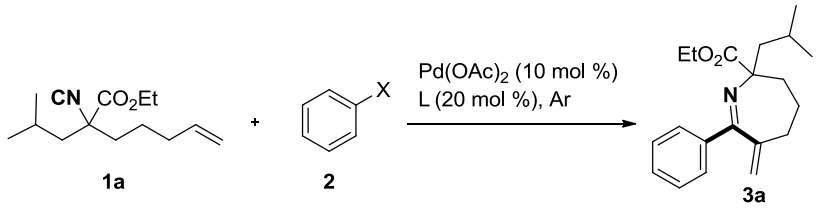
To a solution of ethyl 2-isocyanoacetate (5 mmol, 2.5 equiv) in acetonitrile (20 mL) were added K_2CO_3 (10 mmol), TBAHS (0.2 mmol), and 3-bromoprop-1-ene (2 mmol). The mixture was heated at 70 ^oC until the reaction was completed, monitoring with TLC. Then the mixture was cooled and the solvent was removed and reduced pressure. H_2O (10 mL) and ethyl acetate (20 mL) were added into the mixture. The organic phase was seperated and the aqueous phase was extacted with ethyl acetate (20.0 mL × 3). The combined organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography (petroleum ether : EtOAc = 8 : 1) to afford **1i** as colorless liquid in 51% yield. (known compound)[4]

^1H NMR (400 MHz, $CDCl_3$): δ 5.81-5.71 (m, 1H), 5.24-5.19 (m, 2H), 4.30-4.20 (m, 3H), 2.69-2.55 (m, 2H), 1.27 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ

166.1, 160.4, 130.4, 120.4, 62.7, 56.3, 37.0, 14.0; HRMS: calcd for $C_8H_{12}NO_2$ (M^++H) 154.0863; found 154.0861.

III. Imidoylative Heck Reaction

Table 1. Detailed Information of Conditions Optimization [a]

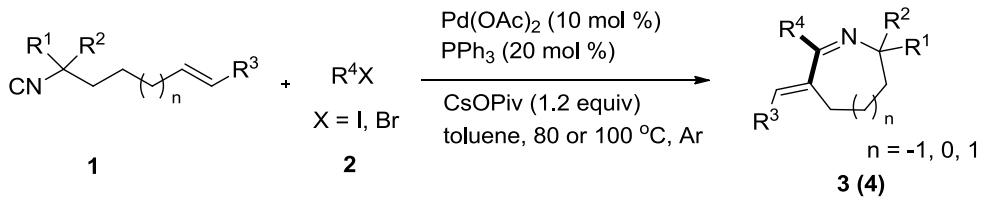


entry	solvent	base (1.2 equiv)	ligand (20 mol %)	temp (^{o}C)	yield (%)[b]
1	toluene	Na_2CO_3	PPh_3	80	nd
2[c]	toluene	Na_2CO_3	PPh_3	80	18
3[c]	toluene	K_2CO_3	PPh_3	80	18
4[c]	toluene	Cs_2CO_3	PPh_3	80	30
5[c]	toluene	CsOPiv	PPh_3	80	70
6[c]	toluene	Et_3N	PPh_3	80	25
7[c]	DMSO	CsOPiv	PPh_3	80	70
8[c]	dioxane	CsOPiv	PPh_3	80	33
9[c]	CH_3CN	CsOPiv	PPh_3	80	40
10[c,e]	toluene	CsOPiv	PPh_3	80	81 (79)[f]
11[c,e]	toluene	CsOPiv	dppb (10 mol %)	80	75
12[c,e]	toluene	CsOPiv	BINAP (10 mol %)	80	trace
13[c,e,g]	toluene	CsOPiv	PPh_3	80	78
14[c,e,h]	toluene	CsOPiv (1.0 equiv)	PPh_3	80	70
15[c,e,i]	toluene	CsOPiv	PPh_3	80	85 (change)
16[c,j]	toluene	CsOPiv	PPh_3	80	56[f]
17[c,e]	toluene	CsOPiv	PPh_3	70	45[f]
18[c,e,k]	toluene	CsOPiv	PPh_3	70	0
19[d,e]	toluene	CsOPiv	PPh_3	100	85 (82)[f]

[a] Reaction conditions: **1a** (0.2 mmol), **2** (0.30 mmol), $Pd(OAc)_2$ (0.02 mmol, 10 mol %), PPh_3 (0.04 mmol, 20 mol %), base (0.24 mmol), Ar. A solution of **1a** in solvent (1 mL) was added slowly within 1 h. [b] NMR yield with 1-iodo-4-methoxybenzene as an internal standard. [c] X = I. [d] X = Br. [e] A solution of **1a** in toluene (1 mL) was added via a syringe pump within 1 h. [f] Isolated yield. [g] One equivalent of H2O was added in the reaction. [h] One equivalent of base was used. [i] The ratio of 1a and 2 was altered. [j] 5 mol % of $Pd(OAc)_2$ and 10 mol % of PPh_3. [k] Uner air atmosphere.

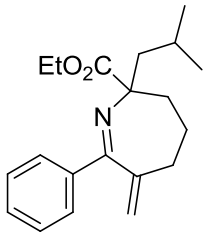
The reaction conditions were screened with ethyl 2-isobutyl-2-isocyanohept-6-enoate **1a** and iodobenzene as model substrates. However, a reaction of premixed reactants under normal Heck conditions ($Pd(OAc)_2$, PPh_3, Na_2CO_3 in toluene) yielded only a messy mixture (entry 1, Table 1). When a solution of isocyanide **1a** in toluene was introduced to the reaction mixture slowly, the desired product, ethyl 2-isobutyl-6-methylene-7-phenyl-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate **3a**, was formed in 18% NMR yield (entry 2, Table 1). Screening of bases revealed that CsOPiv was the base of choice for this imidoylative Heck reaction, and **3a** could be obtained in 70% yield (entries 3-6). The reaction was equally efficient in DMSO, but less efficient in dioxane or CH_3CN (entries 7-9). The yield of **3a** was increased to 81% when a solution of **1a** in 1 mL of toluene was added via a syringe pump during 1 h (entry 10). Dppb was also an efficient ligand (entry 11). Other phosphine ligands such as BINAP were tested as well, but resulted in trace product (entry 12). Considering that PPh_3 was an easily available and inexpensive ligand, it was chosen in the reaction. When 1.0 equivalent of H_2O was added in the reaction, 78% NMR yield of product was generated, indicating that the reaction was well tolerated in moisture (entry 13). When using 1.0 equivalent of base, the yield was decreased to 70% (entry 14). If the ratio between the isocyanide and the aryl iodide are altered, the yield was similar (entry 15). Considering that aryl halides are less inexpensive than isocyanide, we used it in excess amount. The yield decreased to 45% at 70 ^oC (entry 16) and 56% with 5 mol % of $Pd(OAc)_2$ and 10 mol % of PPh_3 (entry 17). No product was generated under air atmosphere (entry 18). When using bromobenzene as the arylating reagent, the same product **3a** was generated in 85% yield at elevated temperature (100 ^oC, entry 19).

General procedure:



An oven-dried 25 mL Schlenk tube charged with $Pd(OAc)_2$ (0.02 mmol, 4.5 mg), PPh_3 (0.04 mmol, 10.5 mg) and CsOPiv (0.24 mmol, 56.0 mg) was vacuumed and refilled with Ar for 3 times. Then a solution of **2** (0.3 mmol, 38.4 μL) in 1.0 mL of toluene was added via a syringe and the tube was placed in an oil-bath at 80 °C for aryl iodide or 100 °C for aryl bromide. A solution of **1** (0.2 mmol) in 1.0 mL of toluene was introduced with a syringe pump to the reaction mixture during 1 h. The crude reaction mixture was extracted with DCM (20 mL × 3) and washed with brine (20 mL). The organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography to afford the corresponding cyclic imines.

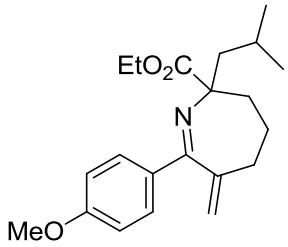
IV. Characterization Data



ethyl 2-isobutyl-6-methylene-7-phenyl-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**3a**)

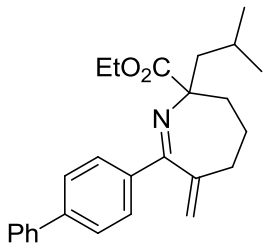
Prepared from ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol, 1.0 equiv) and iodobenzene (61 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 30 : 1) furnished the product **3a** as colorless oil (50 mg, 0.16 mmol, 79% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86-7.83 (m, 2H), 7.39-7.33 (m, 3H), 5.40 (d, *J* = 1.2 Hz, 1H), 4.87 (d, *J* = 1.2 Hz, 1H), 4.19-4.03 (m, 2H), 2.41-2.36 (m, 1H), 2.20-2.12 (m, 2H), 2.05-1.96 (m, 2H), 1.89-1.81 (m, 3H), 1.74-1.64 (m, 1H), 1.24 (t, *J* = 7.2 Hz, 3H), 1.02 (d, *J* = 6.8 Hz, 3H), 0.93 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.4, 170.6, 146.8, 140.6, 129.8, 128.7, 127.9, 118.5, 69.9, 60.5, 51.7, 35.0, 34.0, 25.7, 24.7, 24.7, 23.8, 14.1; HRMS: calcd for $C_{20}H_{28}NO_2$

(M^++H) 314.2115; found 314.2113.



ethyl 2-isobutyl-7-(4-methoxyphenyl)-6-methylene-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**3b**)

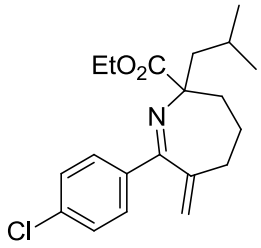
Prepared from ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol, 1.0 equiv) and 1-iodo-4-methoxybenzene (70 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 15 : 1) furnished the product **3b** as colorless oil (54 mg, 0.16 mmol, 79% yield). (new compound). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.82-7.81 (m, 2H), 6.88-6.86 (m, 2H), 5.38 (d, *J* = 1.0 Hz, 1H), 4.86 (d, *J* = 1.0 Hz, 1H), 4.18-4.02 (m, 2H), 3.82 (d, *J* = 6.0 Hz, 3H), 2.37-2.35 (m, 1H), 2.12-2.11 (m, 2H), 2.03-1.94 (m, 2H), 1.86-1.81 (m, 3H), 1.71-1.61 (m, 1H), 1.24 (t, *J* = 7.5 Hz, 3H), 1.01 (d, *J* = 6.5 Hz, 3H), 0.92 (d, *J* = 6.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.6, 170.1, 161.2, 146.8, 133.2, 130.2, 118.2, 113.2, 69.6, 60.4, 55.3, 51.8, 35.1, 34.0, 25.9, 24.7, 24.7, 23.8, 14.1; HRMS: calcd for $C_{21}H_{30}NO_3$ (M^++H) 344.2220; found 344.2221.



ethyl 7-([1,1'-biphenyl]-4-yl)-2-isobutyl-6-methylene-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**3c**)

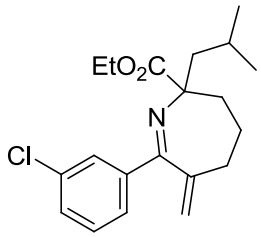
Prepared from ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol, 1.0 equiv) and 4-iodo-1,1'-biphenyl (84 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 30 : 1) furnished the product **3c** as colorless oil (60 mg, 0.15 mmol, 77% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94-7.92 (m, 2H), 7.63-7.58 (m, 4H), 7.47-7.43 (m, 2H), 7.38-7.34 (m, 1H), 5.44 (d, *J* = 0.8 Hz, 1H), 4.93 (d, *J* = 0.8 Hz,

1H), 4.20-4.05 (m, 2H), 2.43-2.40 (m, 1H), 2.19-2.13 (m, 2H), 2.07-1.98 (m, 2H), 1.90-1.84 (m, 3H), 1.76-1.72 (m, 1H), 1.25 (t, *J* = 7.2 Hz, 3H), 1.03 (d, *J* = 6.4 Hz, 3H), 0.95 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.4, 170.2, 146.7, 142.6, 140.8, 139.5, 129.2, 128.8, 127.5, 127.1, 126.7, 118.5, 70.0, 60.5, 51.8 35.1, 34.1, 25.8, 24.7, 24.7, 23.8, 14.1; HRMS: calcd for $C_{26}H_{32}NO_2$ (M^++H) 390.2428; found 390.2430.



ethyl 7-(4-chlorophenyl)-2-isobutyl-6-methylene-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**3d**)

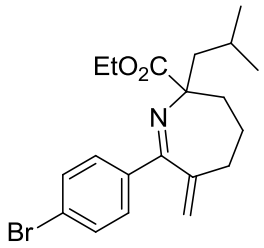
Prepared from ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol, 1.0 equiv) and 1-chloro-4-iodobenzene (71 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 30 : 1) furnished the product **3d** as colorless oil (54 mg, 0.16 mmol, 78% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.4 Hz, 2H), 5.41 (s, 1H), 4.85 (d, *J* = 0.8 Hz, 1H), 4.20-4.02 (m, 2H), 2.40-2.34 (m, 1H), 2.18-2.10 (m, 2H), 2.03-1.93 (m, 2H), 1.88-1.80 (m, 3H), 1.73-1.67 (m, 1H), 1.23 (t, *J* = 7.2 Hz, 3H), 1.00 (d, *J* = 6.4 Hz, 3H), 0.92 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.1, 169.4, 146.4, 139.0, 136.0, 130.0, 128.1, 118.8, 70.0, 60.5, 51.5, 35.0, 34.0, 25.7, 24.7, 24.7, 23.7, 14.1; HRMS: calcd for $C_{20}H_{27}ClNO_2$ (M^++H) 348.1725; found 348.1722.



ethyl 7-(3-chlorophenyl)-2-isobutyl-6-methylene-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**3e**)

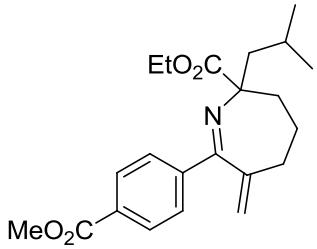
Prepared from ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol, 1.0

equiv) and 1-chloro-3-iodobenzene (71 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 30 : 1) furnished the product **3e** as colorless oil (69 mg, 0.19 mmol, 99% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (t, *J* = 1.6 Hz, 1H), 7.71 (d, *J* = 7.6 Hz, 1H), 7.37-7.34 (m, 1H), 7.30-7.26 (m, 1H), 5.42 (d, *J* = 0.8 Hz, 1H), 4.88 (d, *J* = 0.8 Hz, 1H), 4.20-4.04 (m, 2H), 2.39-2.35 (m, 1H), 2.17-2.10 (m, 2H), 2.02-1.94 (m, 2H), 1.89-1.80 (m, 3H), 1.73-1.69 (m, 1H), 1.24 (t, *J* = 6.8 Hz, 3H), 1.00 (d, *J* = 6.4 Hz, 3H), 0.92 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.1, 169.2, 146.2, 142.4, 134.0, 129.8, 129.1, 128.7, 126.9, 119.0, 70.1, 60.6, 51.5, 35.0, 34.0, 25.6, 24.7, 24.6, 23.7, 14.1; HRMS: calcd for $C_{20}H_{27}ClNO_2$ (M^++H) 348.1725; found 348.1723.



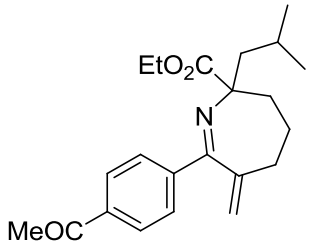
ethyl 7-(4-bromophenyl)-2-isobutyl-6-methylene-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**3f**)

Prepared from ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol, 1.0 equiv) and 1-bromo-4-iodobenzene (84 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 30 : 1) furnished the product **3f** as colorless oil (69 mg, 0.18 mmol, 90% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, *J* = 8.4 Hz, 2H), 7.47 (d, *J* = 8.4 Hz, 2H), 5.41 (s, 1H), 4.85 (d, *J* = 0.8 Hz, 1H), 4.20-4.02 (m, 2H), 2.40-2.34 (m, 1H), 2.18-2.10 (m, 2H), 2.03-1.93 (m, 2H), 1.89-1.79 (m, 3H), 1.73-1.67 (m, 1H), 1.23 (t, *J* = 6.8 Hz, 3H), 1.00 (d, *J* = 6.4 Hz, 3H), 0.92 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.1, 169.5, 146.3, 139.4, 131.0, 130.3, 124.5, 118.9, 70.0, 60.5, 51.5, 35.0, 34.0, 25.7, 24.7, 24.6, 23.7, 14.1; HRMS: calcd for $C_{20}H_{27}BrNO_2$ (M^++H) 392.1220; found 392.1222.



ethyl 7-(4-bromophenyl)-2-isobutyl-6-methylene-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**3g**)

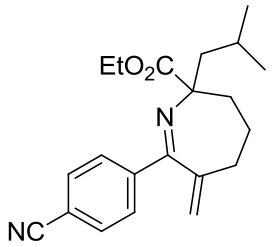
Prepared from ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol, 1.0 equiv) and methyl 4-iodobenzoate (78 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 15 : 1) furnished the product **3g** as yellow oil (57 mg, 0.15 mmol, 77% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01 (d, *J* = 8.4 Hz, 2H), 7.88 (d, *J* = 8.4 Hz, 2H), 5.42 (s, 1H), 4.85 (s, 1H), 4.19-4.04 (m, 2H), 3.93 (s, 3H), 2.42-2.36 (m, 1H), 2.21-2.11 (m, 2H), 2.03-1.94 (m, 2H), 1.89-1.80 (m, 3H), 1.80-1.72 (m, 1H), 1.23 (t, *J* = 6.8 Hz, 3H), 1.00 (d, *J* = 6.4 Hz, 3H), 0.92 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.0, 169.8, 166.9, 146.4, 144.8, 131.1, 129.2, 128.7, 119.1, 70.3, 60.6, 52.1, 51.5, 35.0, 34.0, 25.6, 24.7, 24.6, 23.7, 14.1; HRMS: calcd for $C_{22}H_{30}NO_4$ (M^++H) 372.2169; found 372.2171.



ethyl 7-(4-acetylphenyl)-2-isobutyl-6-methylene-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**3h**)

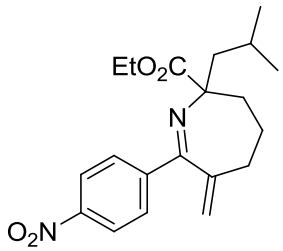
Prepared from ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol, 1.0 equiv) and 1-(4-iodophenyl)ethanone (74 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 15 : 1) furnished the product **3h** as yellow oil (70 mg, 0.19 mmol, 99% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94-7.89 (m, 4H), 5.43 (d, *J* = 1.2 Hz, 1H), 4.85 (d, *J* = 1.2 Hz, 1H), 4.19-4.04 (m, 2H), 2.60 (s, 3H), 2.41-2.36 (m, 1H), 2.19-2.10 (m, 2H), 2.03-1.94 (m, 2H), 1.90-1.81 (m, 3H), 1.74-1.70 (m, 1H), 1.23 (t, *J* = 7.2 Hz, 3H), 1.00 (d, *J* = 6.4 Hz, 3H), 0.92 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (125 MHz,

$CDCl_3$): δ 197.8, 174.0, 169.7, 146.4, 144.8, 137.8, 128.9, 127.9, 119.0, 70.3, 60.6, 51.5, 35.0, 34.0, 26.7, 25.6, 24.7, 24.6, 23.7, 14.1; HRMS: calcd for $C_{22}H_{30}NO_3$ (M^++H) 356.2220; found 356.2220.



ethyl 7-(4-cyanophenyl)-2-isobutyl-6-methylene-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**3i**)

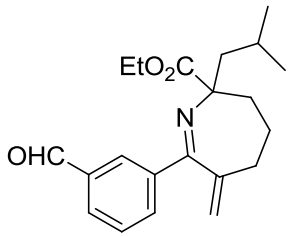
Prepared from ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol, 1.0 equiv) and 4-iodobenzonitrile (69 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 15 : 1) furnished the product **3i** as yellow oil (67 mg, 0.19 mmol, 99% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J* = 8.4 Hz, 2H), 6.30 (d, *J* = 8.4 Hz, 2H), 5.45 (d, *J* = 0.8 Hz, 1H), 4.85 (d, *J* = 0.8 Hz, 1H), 4.19-4.05 (m, 2H), 2.40-2.36 (m, 1H), 2.18-2.10 (m, 2H), 2.00-1.78 (m, 5H), 1.74-1.67 (m, 1H), 1.23 (t, *J* = 7.2 Hz, 3H), 0.99 (d, *J* = 6.4 Hz, 3H), 0.91 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.7, 168.9, 145.9, 144.6, 131.7, 129.2, 119.4, 118.7, 113.2, 70.5, 60.7, 51.3, 35.0, 34.0, 25.5, 24.7, 24.6, 23.6, 14.1; HRMS: calcd for $C_{21}H_{27}N_2O_2$ (M^++H) 339.2067; found 339.2071.



ethyl 2-isobutyl-6-methylene-7-(4-nitrophenyl)-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**3j**)

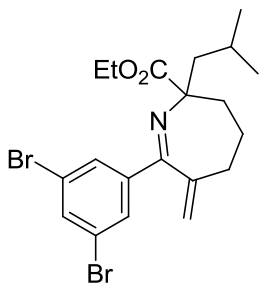
Prepared from ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol, 1.0 equiv) and 1-iodo-4-nitrobenzene (74 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 15 : 1) furnished the product **3j** as yellow oil (69 mg, 0.19 mmol, 97% yield). (new

compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.21-8.17 (m, 2H), 8.00-7.96 (m, 2H), 5.47 (d, *J* = 0.8 Hz, 1H), 4.87 (d, *J* = 0.8 Hz, 2H), 4.20-4.06 (m, 2H), 2.41-2.38 (m, 1H), 2.18-2.11 (m, 2H), 2.02-1.82 (m, 5H), 1.76-1.71 (m, 1H), 1.24 (t, *J* = 7.2 Hz, 3H), 1.00 (d, *J* = 6.4 Hz, 3H), 0.92 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.6, 168.6, 148.6, 146.3, 146.0, 129.5, 123.1, 119.5, 70.6, 60.7, 51.2, 35.0, 34.0, 25.5, 24.7, 24.6, 23.6, 14.1; HRMS: calcd for $C_{20}H_{27}N_2O_4$ (M^++H) 359.1965; found 359.1963.



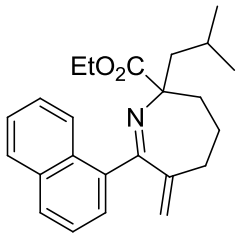
ethyl 7-(3-formylphenyl)-2-isobutyl-6-methylene-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**3k**)

Prepared from ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol, 1.0 equiv) and 3-iodobenzaldehyde (70 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 15 : 1) furnished the product **3k** as colorless oil (68 mg, 0.19 mmol, 99% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.0 (s, 1H), 8.29 (t, *J* = 1.2 Hz, 1H), 8.16 (ddd, *J* = 8.0, 1.2, 1.2 Hz, 1H), 7.91 (ddd, *J* = 8.0, 1.2, 1.2 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 1H), 5.46 (s, 1H), 4.89 (d, *J* = 0.8 Hz, 1H), 4.20-4.05 (m, 2H), 2.43-2.38 (m, 1H), 2.21-2.11 (m, 2H), 2.03-1.95 (m, 2H), 1.90-1.81 (m, 3H), 1.75-1.71 (m, 1H), 1.24 (t, *J* = 7.2 Hz, 3H), 1.00 (d, *J* = 6.4 Hz, 3H), 0.93 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.2, 174.0, 169.3, 146.1, 141.6, 136.2, 134.5, 130.5, 130.5, 128.7, 119.3, 70.2, 60.6, 51.4, 35.0, 34.0, 25.6, 24.7, 24.6, 23.7, 14.1; HRMS: calcd for $C_{21}H_{28}NO_3$ (M^++H) 342.2064; found 342.2065.



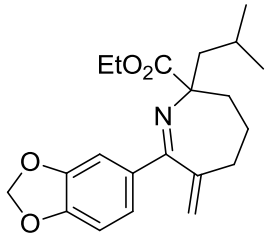
ethyl 7-(3,5-dibromophenyl)-2-isobutyl-6-methylene-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**3l**)

Prepared from ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol, 1.0 equiv) and 1,3-dibromo-5-iodobenzene (108 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 30 : 1) furnished the product **3l** as yellow oil (79 mg, 0.17 mmol, 84% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89 (d, *J* = 2.0 Hz, 2H), 7.67 (t, *J* = 1.6 Hz, 1H), 5.45 (s, 1H), 4.89 (s, 1H), 4.19-4.05 (m, 2H), 2.39-2.33 (m, 1H), 2.16-2.10 (m, 2H), 1.98-1.78 (m, 5H), 1.71-1.65 (m, 1H), 1.24 (t, *J* = 7.2 Hz, 3H), 0.99 (d, *J* = 6.4 Hz, 3H), 0.92 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.8, 167.8, 145.6, 143.9, 135.1, 130.5, 122.6, 119.6, 70.3, 60.8, 51.3, 34.9, 33.9, 25.6, 24.7, 24.7, 23.8, 14.2; HRMS: calcd for $C_{20}H_{26}Br_2NO_2$ (M^++H) 470.0325; found 470.0323.



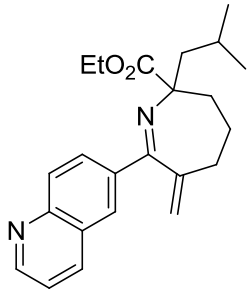
ethyl 2-isobutyl-6-methylene-7-(naphthalen-1-yl)-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**3m**)

Prepared from ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol, 1.0 equiv) and 1-iodonaphthalene (76 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 30 : 1) furnished the product **3m** as colorless oil (43 mg, 0.17 mmol, 59% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.33-8.31 (m, 1H), 7.84-7.82 (m, 2H), 7.51-7.42 (m, 4H), 5.27 (s, 1H), 4.83 (s, 1H), 4.26-4.15 (m, 2H), 2.65-2.59 (m, 1H), 2.49-2.42 (m, 1H), 2.34-2.30 (m, 1H), 2.10-2.04 (m, 1H), 2.03-2.00 (m, 2H), 1.97-1.84 (m, 3H), 1.31 (t, *J* = 7.6 Hz, 3H), 0.95-0.92 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.2, 171.7, 148.7, 141.4, 133.9, 131.5, 128.8, 128.1, 127.0, 126.6, 125.9, 125.6, 124.8, 120.8, 70.8, 60.8, 52.7, 35.3, 34.2, 24.9, 24.7, 24.4, 23.6, 14.1; HRMS: calcd for $C_{24}H_{30}NO_2$ (M^++H) 364.2271; found 364.2273.



ethyl 7-(benzo[*d*][1,3]dioxol-5-yl)-2-isobutyl-6-methylene-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**3n**)

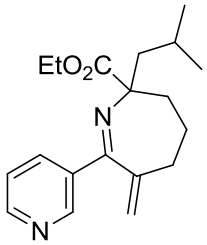
Prepared from ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol, 1.0 equiv) and 5-bromobenzo[*d*][1,3]dioxole (49 mg, 0.3 mmol, 1.5 equiv) according to the procedure A. Column chromatography purification (EtOAc : petroleum ether 15 : 1) furnished the product **3n** as colorless oil (79 mg, 0.13 mmol, 66% yield). (new compound). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.52 (d, *J* = 1.0 Hz, 1H), 7.35 (dd, *J* = 6.4, 1.5 Hz, 1H), 6.81 (d, *J* = 6.4 Hz, 1H), 6.02 (d, *J* = 1.0 Hz, 2H), 5.42 (s, 1H), 4.91 (s, 1H), 4.23-4.05 (m, 2H), 2.41-2.36 (m, 1H), 2.18-2.15 (m, 2H), 2.06-1.98 (m, 3H), 1.89-1.85 (m, 2H), 1.74-1.70 (m, 1H), 1.27 (t, *J* = 7.0 Hz, 3H), 1.04 (d, *J* = 7.0 Hz, 3H), 0.96 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.5, 169.8, 149.3, 147.6, 146.7, 135.1, 123.9, 118.4, 108.6, 107.3, 101.3, 70.0, 60.5, 51.8, 35.1, 34.0, 25.9, 24.7, 24.7, 23.8, 14.1; HRMS: calcd for $C_{21}H_{28}NO_4$ (M^++H) 358.2013; found 358.2011



ethyl 2-isobutyl-6-methylene-7-(quinolin-6-yl)-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**3o**)

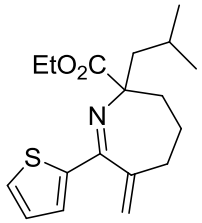
Prepared from ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol, 1.0 equiv) and 6-iodoquinoline (77 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 15 : 1) furnished the product **3o** as yellow oil (72 mg, 0.19 mmol, 99% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.92 (dd, *J* = 4.0, 1.6 Hz, 1H), 8.37 (dd, *J* = 8.4, 1.2 Hz, 1H), 8.20-8.18 (m, 2H), 8.09 (d, *J* = 8.8 Hz, 1H), 7.41 (dd, *J* = 4.4, 4.0

Hz, 1H), 5.50 (s, 1H), 4.94 (d, *J* = 1.2 Hz, 1H), 4.22-4.06 (m, 2H), 2.47-2.42 (m, 1H), 2.25-2.15 (m, 2H), 2.09-2.00 (m, 2H), 1.93-1.86 (m, 3H), 1.78-1.73 (m, 1H), 1.25 (t, *J* = 7.2 Hz, 3H), 1.05 (d, *J* = 6.4 Hz, 3H), 0.96 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.2, 169.8, 151.0, 149.2, 146.4, 138.4, 136.9, 129.4, 129.1, 128.8, 127.6, 121.2, 118.9, 70.2, 60.6, 51.6, 35.0, 34.0, 25.8, 24.7, 24.7, 23.8, 14.1; HRMS: calcd for $C_{23}H_{29}N_2O_2$ (M^++H) 365.2224; found 365.2223.



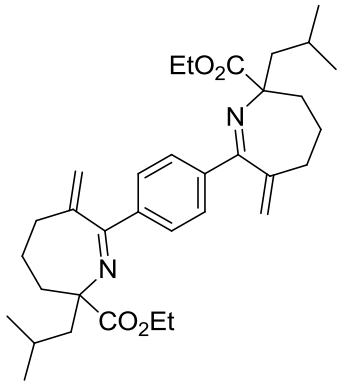
ethyl 2-isobutyl-6-methylene-7-(pyridin-3-yl)-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**3p**)

Prepared from ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol, 1.0 equiv) and 3-iodopyridine (62 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 15 : 1) furnished the product **3p** as yellow oil (55 mg, 0.17 mmol, 87% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.00 (d, *J* = 1.6 Hz, 1H), 8.62 (dd, *J* = 4.8, 1.6 Hz, 1H), 8.15 (ddd, *J* = 8.0, 2.0, 2.0 Hz, 1H), 7.32-7.28 (m, 1H), 5.48 (d, *J* = 0.8 Hz, 1H), 4.92 (d, *J* = 0.8 Hz, 1H), 4.21-4.07 (m, 2H), 2.44-2.39 (m, 1H), 2.22-2.13 (m, 2H), 2.05-1.72 (m, 6H), 1.26 (t, *J* = 7.2 Hz, 3H), 1.01 (d, *J* = 6.4 Hz, 3H), 0.93 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.9, 168.5, 150.6, 150.3, 146.1, 136.2, 135.9, 122.9, 119.5, 70.4, 60.7, 51.5, 35.2, 34.1, 25.6, 24.7, 24.7, 23.6, 14.1; HRMS: calcd for $C_{19}H_{27}N_2O_2$ (M^++H) 315.2067; found 315.2067.



ethyl
2-isobutyl-6-methylene-7-(thiophen-2-yl)-3,4,5,6-tetrahydro-2H-azepine-2-carboxylate (**3q**)

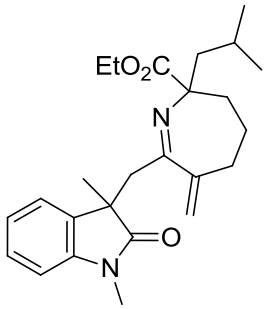
Prepared from ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol, 1.0 equiv) and 2-iodothiophene (63 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 15 : 1) furnished the product **3q** as colorless oil (35 mg, 0.11 mmol, 55% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37-7.36 (m, 1H), 7.28-7.26 (m, 1H), 7.00-6.98 (m, 1H), 5.40 (s, 1H), 5.11 (d, *J* = 1.2 Hz, 1H), 4.18-4.05 (m, 2H), 2.35-2.30 (m, 1H), 2.13-2.07 (m, 2H), 2.00-1.91 (m, 1H), 1.90-1.80 (m, 3H), 1.77-1.64 (m, 1H), 1.23 (t, *J* = 7.2 Hz, 3H), 1.01 (d, *J* = 6.4 Hz, 3H), 0.91 (d, *J* = 6.4 Hz, 3H) ; ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.2, 165.2, 148.1, 145.9, 123.0, 129.5, 127.0, 118.3, 70.1, 60.6, 51.6, 35.6, 33.9, 26.0, 24.8, 24.7, 23.8, 14.2; HRMS: calcd for $C_{18}H_{26}NO_2S$ (M^++H) 320.1679; found 320.1680.



diethyl 7,7'-(1,4-phenylene)bis(2-isobutyl-6-methylene-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate) (**3r**)

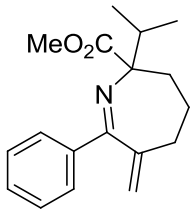
Procedure: An oven-dried 25 mL Schlenk tube charged with $Pd(OAc)_2$ (0.02 mmol, 4.5 mg), PPh_3 (0.04 mmol, 10.5 mg), 1,4-diiodobenzene (0.1 mmol, 33 mg) and CsOPiv (0.24 mmol, 56.0 mg) was vacuumed and refilled with Ar for 3 times. Then the tube was placed in an oil-bath at 80 °C. A solution of ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol) in 1.0 mL of toluene was added dropwise with a syringe pump within 1 h. The crude reaction mixture was extracted with DCM (20 mL × 3) and washed with brine (20 mL). The organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography to afford **3r** as colorless oil (42 mg, 0.15 mmol, 76% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 (s, 4H), 5.39 (s, 2H), 4.85 (s, 2H), 4.19-4.00 (m, 4H), 2.41-2.35 (m, 2H), 2.19-2.12 (m, 4H), 2.06-1.94 (m, 4H), 1.88-1.79 (m, 6H), 1.74-1.68 (m, 2H), 1.22 (t, *J* = 7.2 Hz, 6H), 1.00 (d, *J* = 6.4 Hz,

6H), 0.92 (d, J = 6.4 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.4, 170.3, 146.7, 141.9, 128.3, 118.7, 70.1, 60.5, 51.8, 35.1, 34.0, 25.7, 24.7, 24.7, 23.8, 14.1; HRMS: calcd for $C_{34}H_{49}N_2O_4$ (M^++H) 549.3687; found 549.3685.



ethyl 7-((1,3-dimethyl-2-oxoindolin-3-yl)methyl)-2-isobutyl-6-methylene-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**3s**)

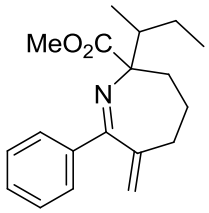
Prepared from ethyl 2-isobutyl-2-isocyanohept-6-enoate (47.4 mg, 0.2 mmol, 1.0 equiv) and *N*-(2-iodophenyl)-*N*-methylmethacrylamide (91 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 10 : 1) furnished the product **3s** as yellow solid (50 mg, 0.12 mmol, 61% yield). (new compound). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.22-7.17 (m, 2H), 7.00-6.98 (m, 1H), 6.79-6.76 (m, 1H), 5.03-5.01 (m, 1H), 4.92-4.91 (m, 1H), 4.06-3.90 (m, 2H), 3.32-3.28 (m, 1H), 3.19-3.17 (m, 2H), 3.03-2.98 (m, 1H), 1.84-1.82 (m, 1H), 1.69-1.54 (m, 4H), 1.52-1.32 (m, 6H), 1.26-1.10 (m, 5H), 0.80-0.68 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 180.4, 174.4, 168.5, 148.4, 143.8, 134.0, 127.3, 122.8, 121.8, 115.7, 107.6, 69.4, 60.4, 49.0, 48.6, 46.9, 34.4, 32.9, 26.2, 25.7, 24.3, 24.3, 24.0, 23.5, 14.1; HRMS: calcd for $C_{25}H_{35}N_2O_3$ (M^++H) 411.2642; found 411.2645.



methyl 2-isopropyl-6-methylene-7-phenyl-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**4a**)

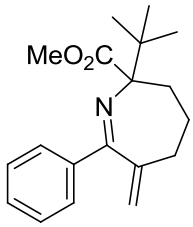
Prepared from methyl 2-isocyano-2-isopropylhept-6-enoate (42 mg, 0.2 mmol, 1.0 equiv) and iodobenzene (61 mg, 0.3 mmol, 1.5 equiv) according to the general

procedure. Column chromatography purification (EtOAc : petroleum ether 30 : 1) furnished the product **4a** as colorless oil (56 mg, 0.19 mmol, 99% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (dd, *J* = 8.0, 1.6 Hz, 2H), 7.42-7.26 (m, 3H), 5.39 (s, 1H), 4.87 (d, *J* = 1.2 Hz, 1H), 3.60 (s, 3H), 2.45-2.39 (m, 1H), 2.22-2.04 (m, 3H), 1.86-1.79 (m, 2H), 1.71-1.64 (m, 1H), 1.07 (d, *J* = 6.4 Hz, 3H), 1.04 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.2, 170.9, 146.2, 140.2, 129.8, 128.6, 127.8, 118.0, 72.6, 51.1, 40.2, 33.6, 31.8, 26.2, 18.4, 16.9; HRMS: calcd for $C_{18}H_{24}NO_2$ (M^++H) 286.1802; found 286.1800.



methyl 2-(sec-butyl)-6-methylene-7-phenyl-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**4b**)

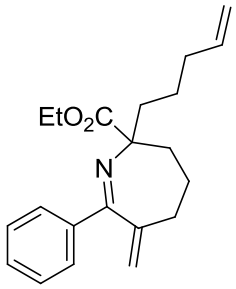
Prepared from methyl 2-(sec-butyl)-2-isocyanohept-6-enoate (45 mg, 0.2 mmol, 1.0 equiv) and iodobenzene (61 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 30 : 1) furnished the product **4b** as colorless oil (55 mg, 0.18 mmol, 92% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90-7.88 (m, 2H), 7.40-7.33 (m, 3H), 5.38 (s, 1H), 4.86 (d, *J* = 1.2 Hz, 1H), 3.60 (s, 3H), 2.44-2.38 (m, 1H), 2.15-2.07 (m, 2H), 1.94-1.64 (m, 5H), 1.40-1.21 (m, 1H), 1.07-0.94 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.3, 170.7, 146.2, 140.1, 129.8, 128.6, 127.8, 117.9, 73.2, 51.1, 47.3, 33.4, 31.4, 25.8, 24.8, 13.0, 12.7; HRMS: calcd for $C_{19}H_{26}NO_2$ (M^++H) 300.1958; found 300.1955.



methyl 2-(*tert*-butyl)-6-methylene-7-phenyl-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**4c**)

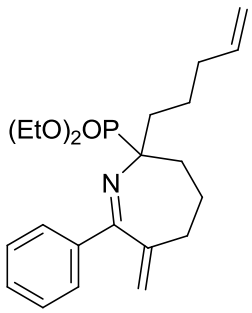
Prepared from methyl 2-(*tert*-butyl)-2-isocyanohept-6-enoate (45 mg, 0.2 mmol,

1.0 equiv) and iodobenzene (61 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 30 : 1) furnished the product **4c** as colorless solid (50 mg, 0.16 mmol, 83% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95-7.91 (m, 2H), 7.43-7.35 (m, 3H), 5.36 (s, 1H), 4.78 (s, 1H), 3.54 (s, 3H), 2.40-2.29 (m, 2H), 2.05-1.80 (m, 3H), 1.53-1.49 (m, 1H), 1.12 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.1, 169.9, 146.4, 139.8, 129.8, 128.4, 127.9, 116.7, 74.3, 50.6, 39.9, 33.5, 28.4, 26.5, 26.1; HRMS: calcd for $C_{19}H_{26}NO_2$ (M^++H) 300.1958; found 300.1958.



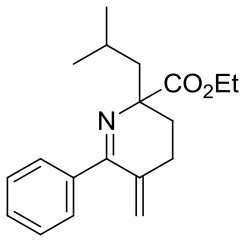
ethyl 6-methylene-2-(pent-4-en-1-yl)-7-phenyl-3,4,5,6-tetrahydro-2*H*-azepine-2-carboxylate (**4d**)

Prepared from ethyl 2-isocyano-2-(pent-4-en-1-yl)hept-6-enoate (50 mg, 0.2 mmol, 1.0 equiv) and iodobenzene (61 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 30 : 1) furnished the product **4d** as colorless oil (59 mg, 0.16 mmol, 83% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (dd, *J* = 8.0, 1.2 Hz, 2H), 7.39-7.33 (m, 3H), 5.88-5.81 (m, 1H), 5.39 (s, 1H), 5.06-4.95 (m, 2H), 4.86 (d, *J* = 1.2 Hz, 1H), 4.18-4.03 (m, 2H), 2.43-2.39 (m, 1H), 2.19-2.08 (m, 4H), 2.00-1.93 (m, 2H), 1.87-1.79 (m, 3H), 1.72-1.60 (m, 1H), 1.50-1.40 (m, 1H), 1.22 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.9, 171.2, 146.7, 140.5, 138.9, 129.9, 128.7, 127.9, 118.7, 114.4, 69.9, 60.5, 43.3, 34.0, 34.0, 25.9, 23.6, 14.2; HRMS: calcd for $C_{21}H_{28}NO_2$ (M^++H) 326.2115; found 326.2119.



diethyl (6-methylene-2-(pent-4-en-1-yl)-7-phenyl-3,4,5,6-tetrahydro-2H-azepin-2-yl) phosphonate (**4e**)

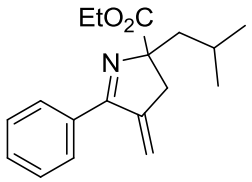
Prepared from diethyl (6-isocyanoundeca-1,10-dien-6-yl)phosphonate (63 mg, 0.2 mmol, 1.0 equiv) and iodobenzene (61 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 15 : 1) furnished the product **4e** as yellow oil (63 mg, 0.16 mmol, 81% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 (d, *J* = 6.8 Hz, 2H), 7.37-7.30 (m, 3H), 5.78-5.74 (m, 1H), 5.42 (s, 1H), 5.00-4.91 (m, 3H), 4.27-4.14 (m, 4H), 2.55-2.52 (m, 1H), 2.37-2.25 (m, 1H), 2.20-1.79 (m, 7H), 1.60-1.49 (m, 2H), 1.34-1.26 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.8 (d, *J* = 24.3 Hz), 146.7, 141.0, 139.0, 130.1, 129.0, 128.1, 119.5, 114.9, 68.4 (d, *J* = 169.5 Hz), 63.1 (d, *J* = 7.5 Hz), 35.5, 34.6, 33.5, 31.2 (d, *J* = 153.3 Hz), 30.0, 29.7, 23.8 (d, *J* = 3.1 Hz), 23.2 (d, *J* = 12.4 Hz), 16.9 (t, *J* = 6.3 Hz); HRMS: calcd for $C_{22}H_{33}NO_3P$ (M^++H) 390.2193; found 390.2186.



ethyl 2-isobutyl-5-methylene-6-phenyl-2,3,4,5-tetrahydropyridine-2-carboxylate (**4f**)

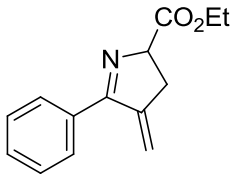
Prepared from ethyl 2-isobutyl-2-isocyanohex-5-enoate (45 mg, 0.2 mmol, 1.0 equiv) and iodobenzene (61 mg, 0.3 mmol, 1.5 equiv) according to the procedure but in THF as a solvent. Column chromatography purification (EtOAc : petroleum ether 30 : 1) furnished the product **4f** as colorless oil (46 mg, 0.15 mmol, 77% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.49-7.47 (m, 2H), 7.36-7.35 (m, 3H), 5.38 (s, 1H), 5.25 (s, 1H), 4.23-4.12 (m, 2H), 2.54-2.51 (m, 2H), 2.25-2.19 (m, 1H), 1.99-1.73 (m, 4H), 1.27 (t, *J* = 6.8 Hz, 3H), 0.97 (d, *J* = 6.4 Hz, 3H), 0.95 (d, *J* = 6.4

Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.3, 167.3, 139.8, 137.6, 128.9, 128.7, 127.8, 121.8, 66.9, 61.0, 48.5, 31.2, 26.4, 24.7, 24.4, 24.0, 14.2; HRMS: calcd for $C_{19}H_{26}NO_2$ (M^++H) 300.1958; found 300.1960.



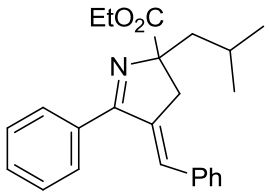
ethyl 2-isobutyl-4-methylene-5-phenyl-3,4-dihydro-2*H*-pyrrole-2-carboxylate (**4g**)

Prepared from ethyl 2-isobutyl-2-isocyanopent-4-enoate (42 mg, 0.2 mmol, 1.0 equiv) and iodobenzene (61 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 30 : 1) furnished the product **4g** as colorless oil (36 mg, 0.12 mmol, 63% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.66-7.63 (m, 2H), 7.47-7.40 (m, 3H), 5.47 (t, *J* = 2.4 Hz, 1H), 5.42 (t, *J* = 2.4 Hz, 1H), 4.29-4.17 (m, 2H), 3.27 (dt, *J* = 16.8, 2.4 Hz, 1H), 2.75 (dt, *J* = 16.8, 2.4 Hz, 1H), 1.99-1.93 (m, 1H), 1.88-1.79 (m, 2H), 1.30 (t, *J* = 6.8 Hz, 3H), 0.96 (d, *J* = 6.0 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.0, 172.3, 148.1, 133.4, 130.0, 128.8, 128.3, 112.6, 79.3, 61.2, 47.7, 40.6, 25.1, 24.1, 23.8, 14.2; HRMS: calcd for $C_{18}H_{24}NO_2$ (M^++H) 286.1802; found 286.1804.



ethyl 4-methylene-5-phenyl-3,4-dihydro-2*H*-pyrrole-2-carboxylate (**4h**)

Prepared from ethyl 2-isocyanopent-4-enoate (30 mg, 0.2 mmol, 1.0 equiv) and iodobenzene (61 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 30 : 1) furnished the product **4h** as yellow oil (33 mg, 0.14 mmol, 72% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): 7.67-7.65 (m, 2H), 7.46-7.40 (m, 3H), 5.51-5.47 (m, 2H), 4.87 (t, *J* = 6.4 Hz, 1H), 4.30-4.22 (m, 2H), 3.06-3.03 (m, 2H), 1.32 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.8, 172.0, 147.8, 133.1, 130.2, 128.7, 128.4, 112.9, 71.0, 61.4, 34.3, 14.2; HRMS: calcd for $C_{14}H_{16}NO_2$ (M^++H) 230.1176; found 230.1174.



(*E*)-ethyl 4-benzylidene-2-isobutyl-5-phenyl-3,4-dihydro-2*H*-pyrrole-2-carboxylate (**4i**)

Prepared from (*E*)-ethyl 2-isobutyl-2-isocyano-5-phenylpent-4-enoate (57 mg, 0.2 mmol, 1.0 equiv) and iodobenzene (61 mg, 0.3 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether 30 : 1) furnished the product **4i** as yellow oil (70 mg, 0.19 mmol, 97% yield). (new compound). ^{1}H NMR (400 MHz, $CDCl_3$): 7.66-7.65 (m, 2H), 7.48-7.37 (m, 7H), 7.32-7.25 (m, 1H), 6.80 (t, *J* = 2.4 Hz, 1H), 4.31-4.18 (m, 2H), 3.55 (dd, *J* = 17.2, 2.8 Hz, 1H), 3.02 (dd, *J* = 17.2, 2.8 Hz, 1H), 1.97-1.93 (m, 2H), 1.91-1.82 (m, 1H), 1.31 (t, *J* = 6.8 Hz, 3H), 0.98-0.95 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.5, 174.5, 140.9, 136.8, 134.2, 130.1, 129.7, 129.4, 129.3, 129.0, 128.7, 128.6, 81.2, 61.7, 48.4, 27.4, 25.4, 24.4, 24.2, 14.5; HRMS: calcd for $C_{24}H_{28}NO_2$ (M^++H) 362.2115; found 362.2118.

V. References

(1) Casabona, D.; Jiménez, A.; Cativiela C. *Tetrahedron*. **2007**, *63*, 5056.

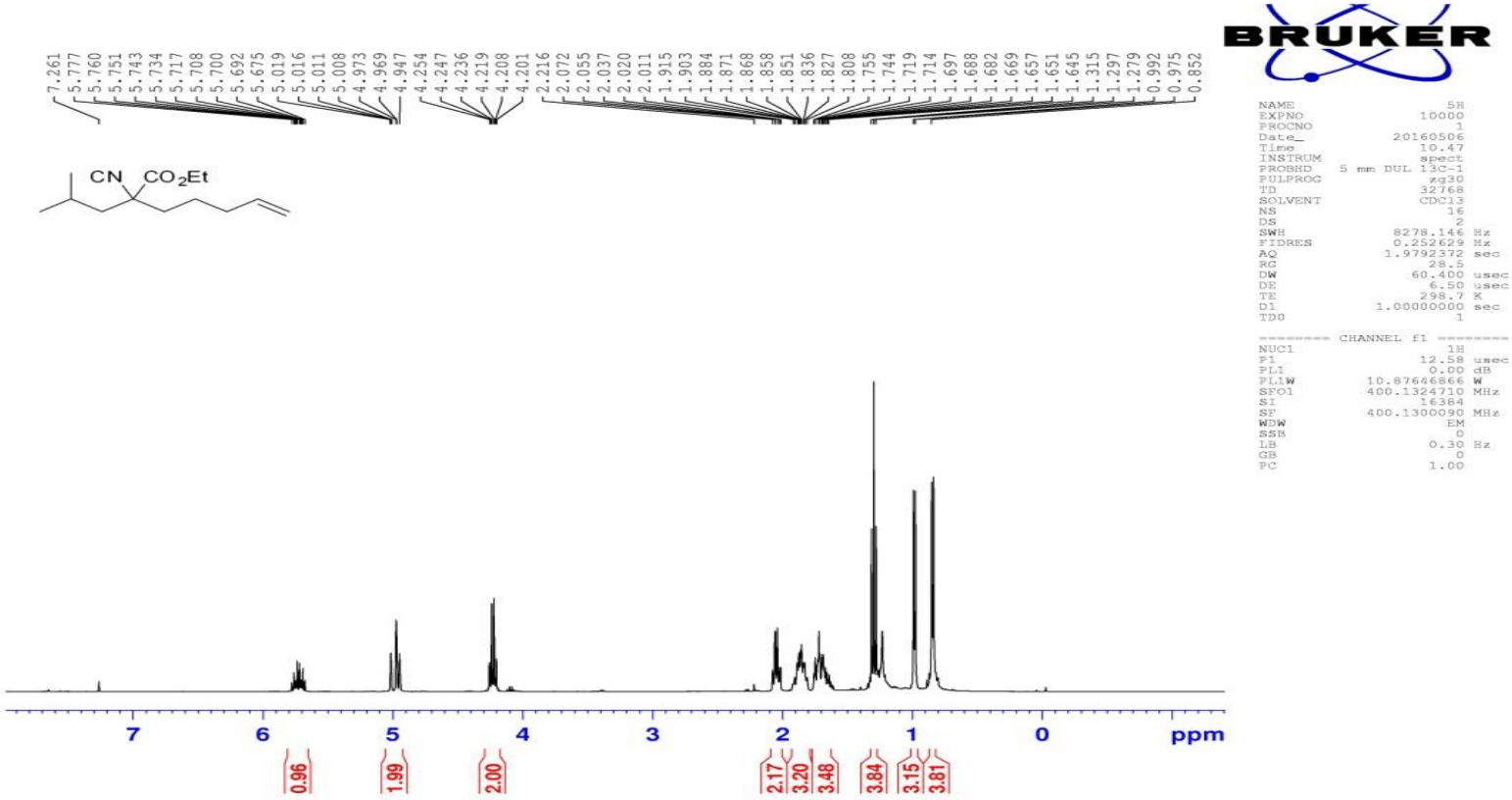
(2) Gulevich, A. V.; Zhdanko, A. G.; Orru, R. V. A.; Nenajdenko, V. G. *Chem. Rev.* **2010**, *110*, 5235.

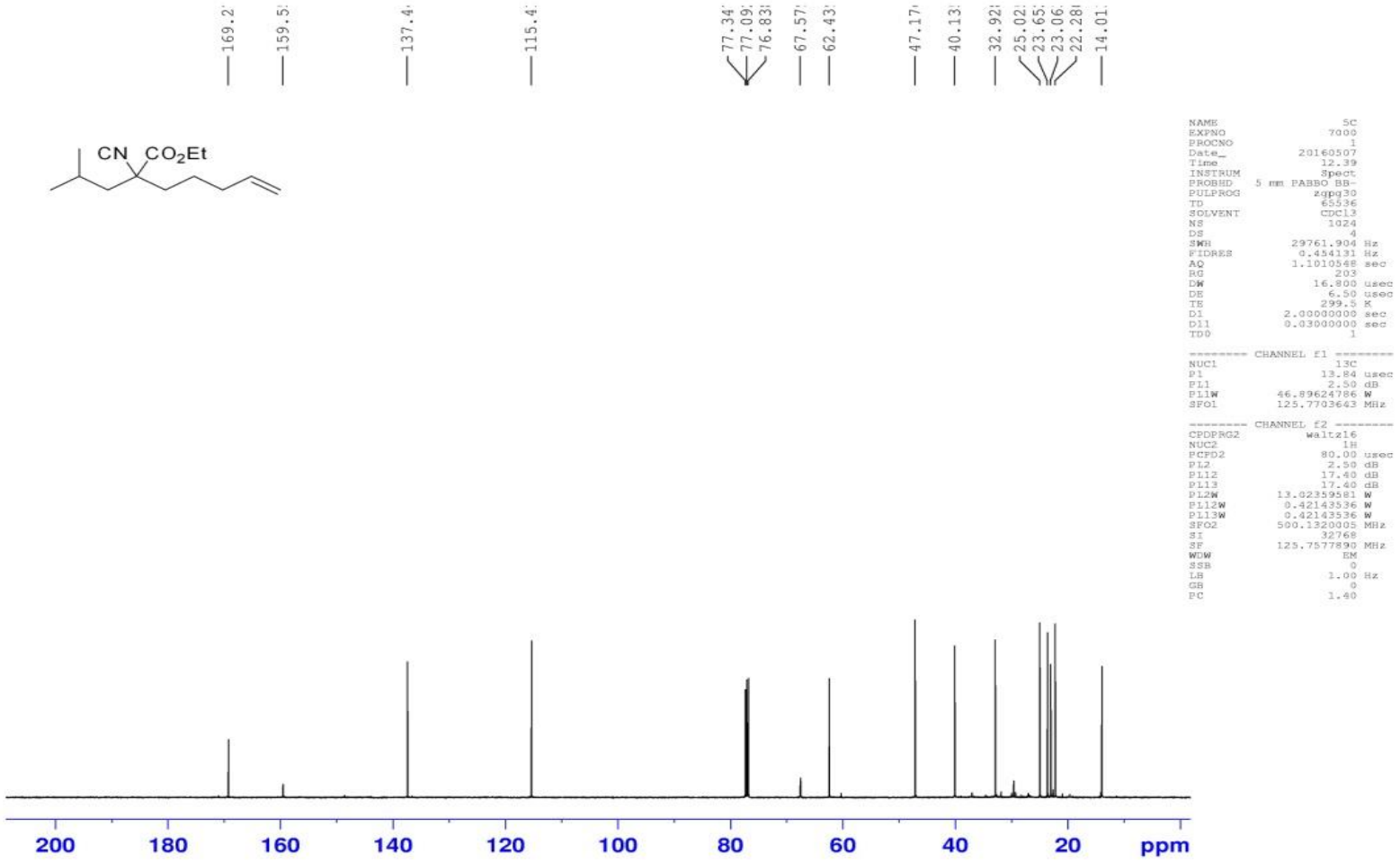
(3) Ito, Y.; Higuchi, N.; Murakami, M. *Tetrahedron Lett.* **1988**, *29*, 5151.

(4) Banfi, L.; Basso, A.; Guanti, G.; Riva, R. *Tetrahedron Lett*. **2003**, *44*, 7655.

VI. Copies of ^{1}H and ^{13}C NMR Spectra

1a





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FIDRES	0.454131 Hz
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RG	203
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DE	6.50 usec
TE	299.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
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PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
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NUC2	1H
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PL12	17.40 dB
PL13	17.40 dB
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PL12W	0.42143536 W
PL13W	0.42143536 W
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SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
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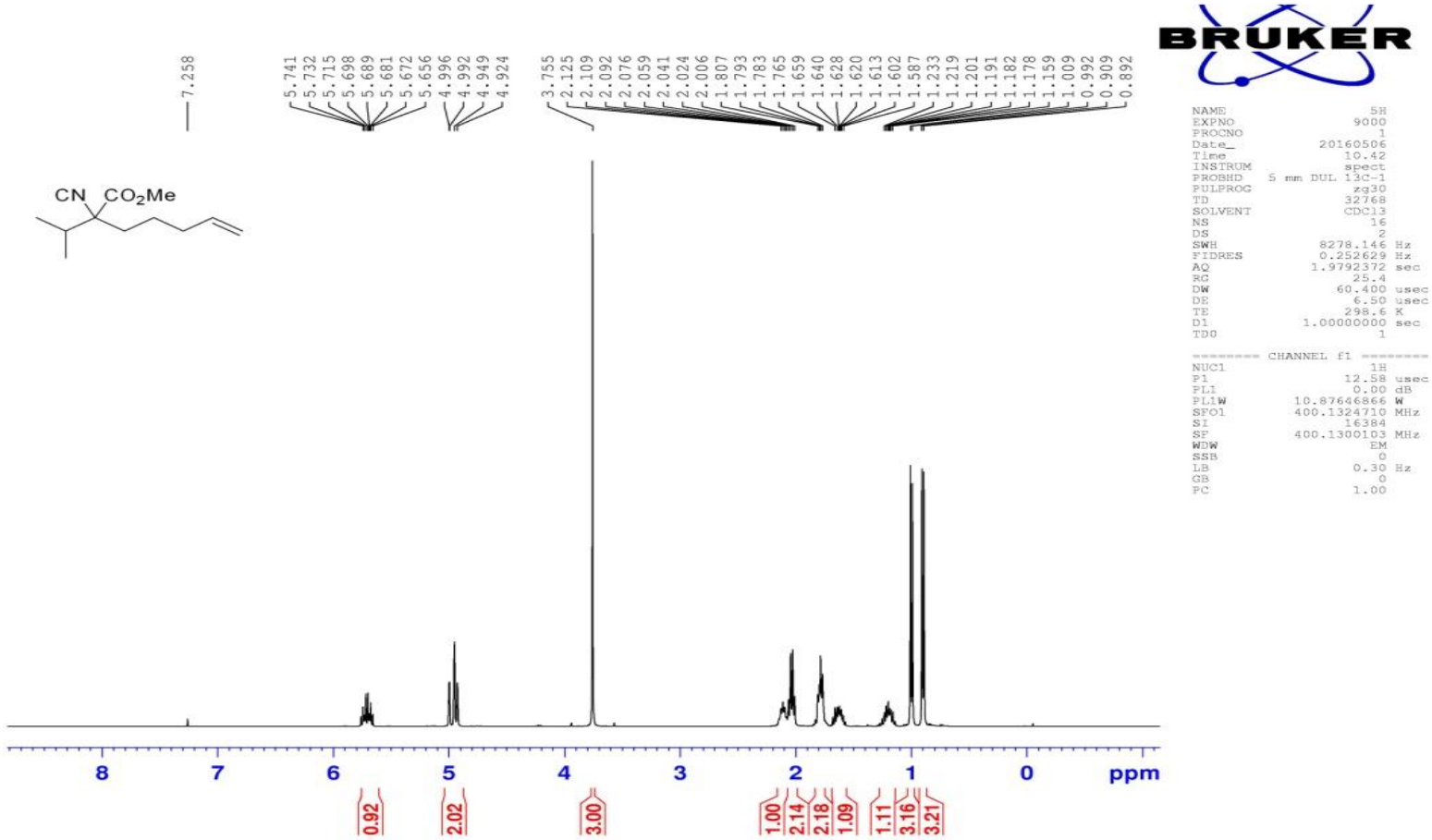
1b



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SOLVENT	CDCl3	
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DS	2	
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FIDRES	0.252629	Hz
AQ	1.9792372	sec
RG	25.4	
DW	60.400	usec
DE	6.50	usec
TE	298.6	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
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P1	12.58	usec
PL1	0.00	dB
PL1W	10.87646866	W
SFO1	400.1324710	MHz
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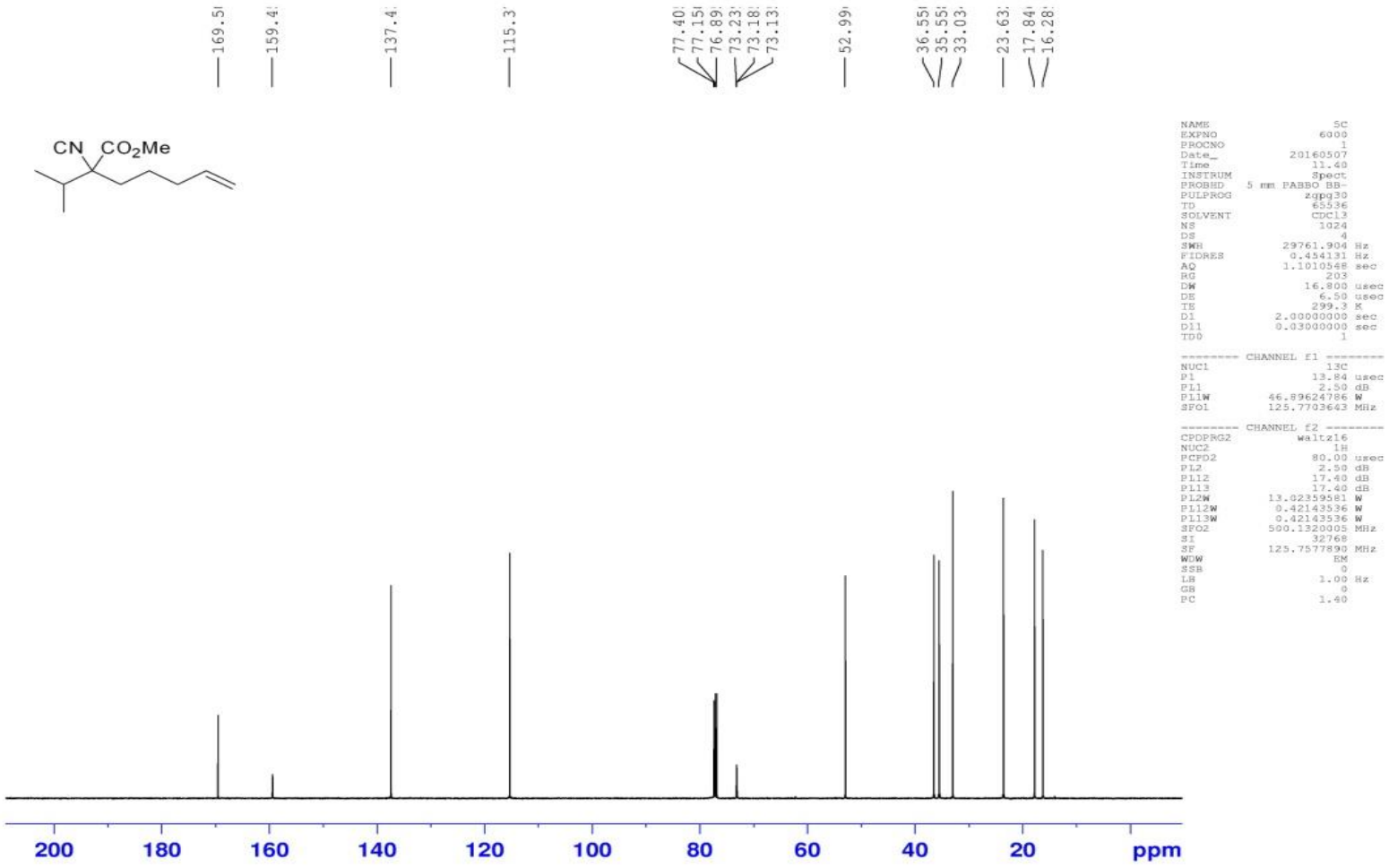

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FIDRES	0.454131 Hz
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RG	203
DW	16.800 usec
DE	6.50 usec
TE	299.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
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NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
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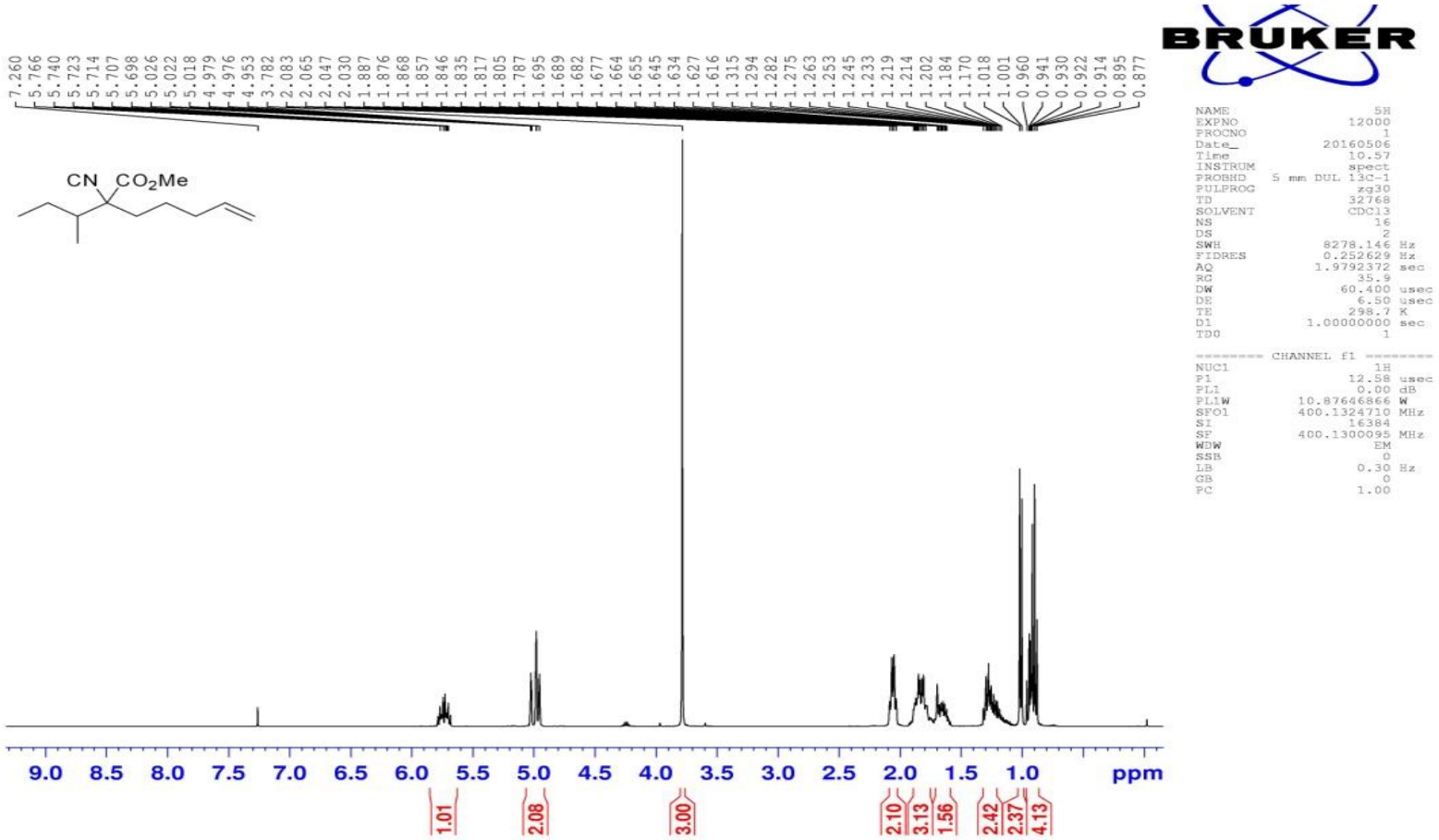
1c



BRUKER

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SOLVENT	CDCl3	
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DS	2	
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FIDRES	0.252629	Hz
AQ	1.9792372	sec
RG	35.9	
DW	60.400	usec
DE	6.50	usec
TE	298.7	K
D1	1.00000000	sec
TD0	1	
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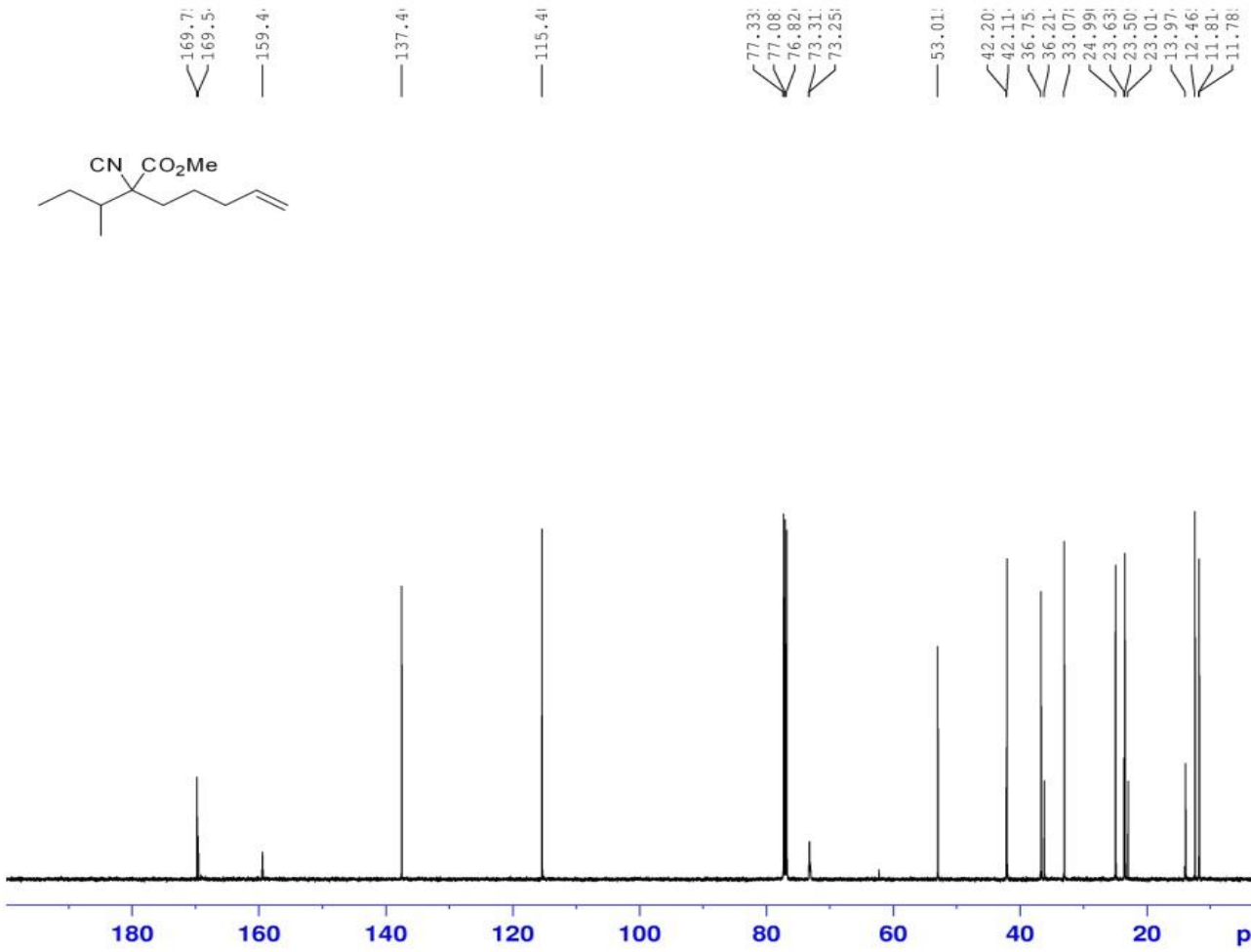

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FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	299.4 K
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D11	0.03000000 sec
TD0	1
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P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
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CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
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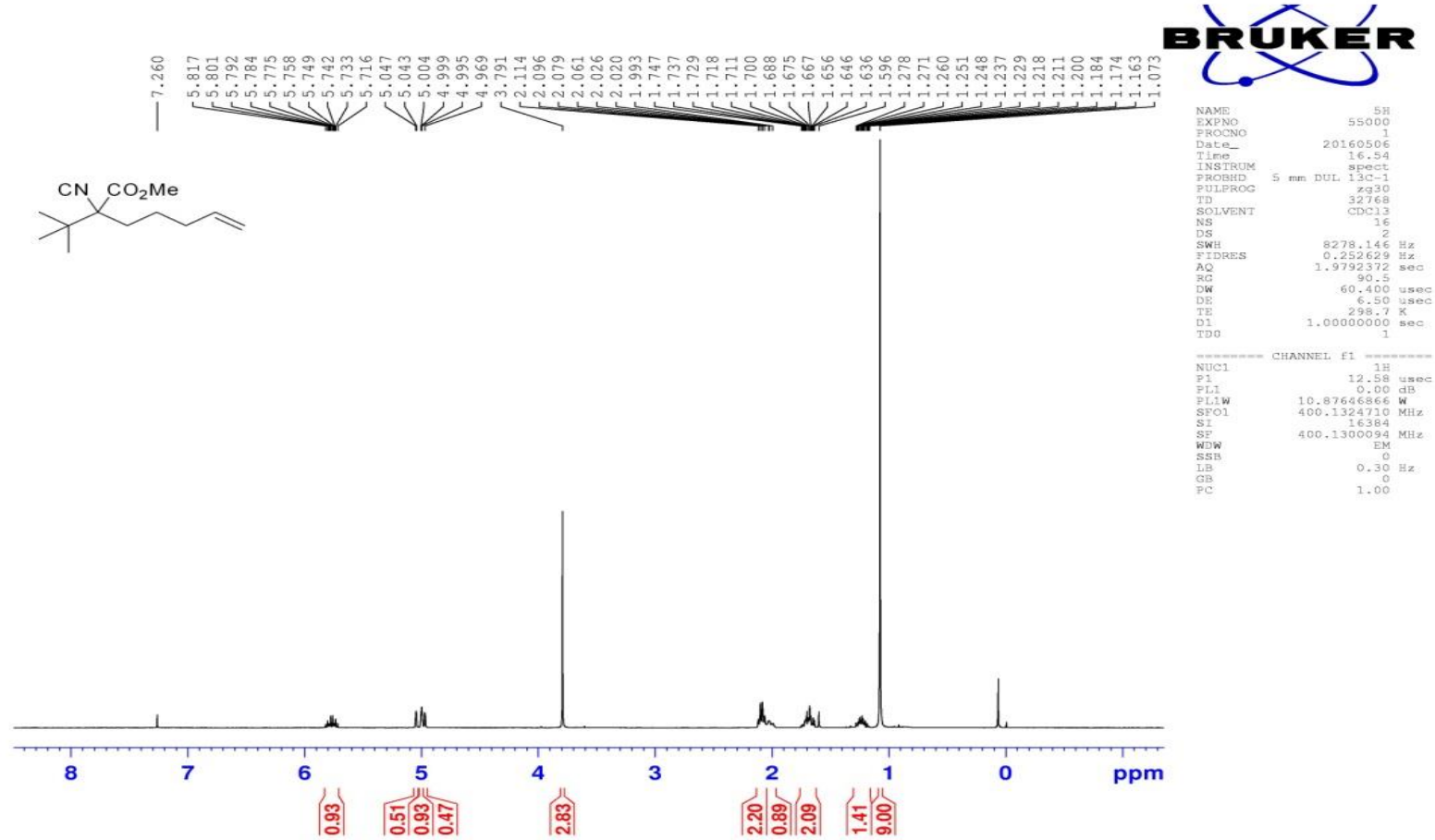
1d



BRUKER

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FIDRES	0.252629 Hz
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RG	90.5
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TE	298.7 K
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PL1W	10.87646866 W
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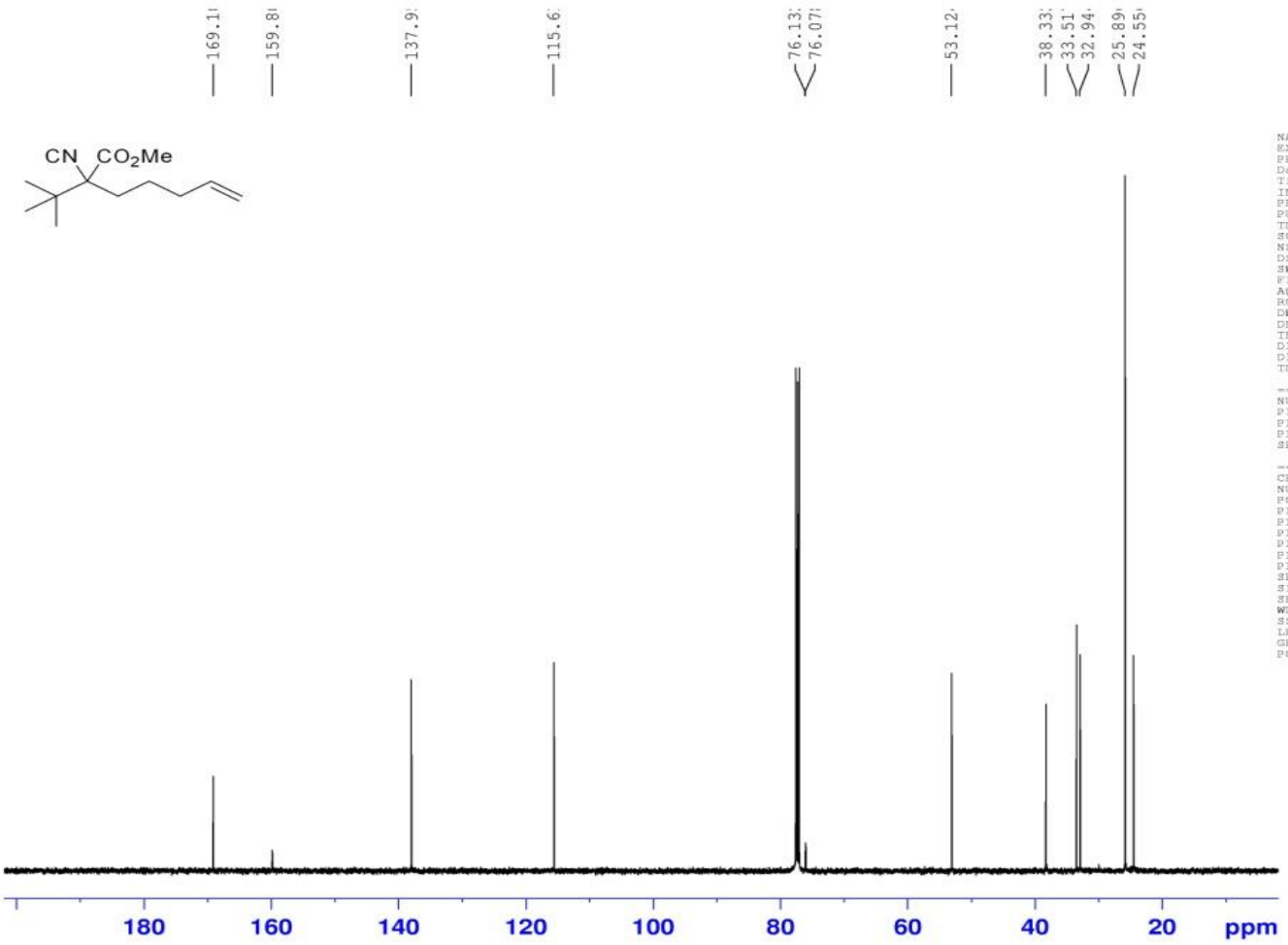

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RG	203
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DE	6.50 usec
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NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
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NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
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PL12W	0.42143536 W
PL13W	0.42143536 W
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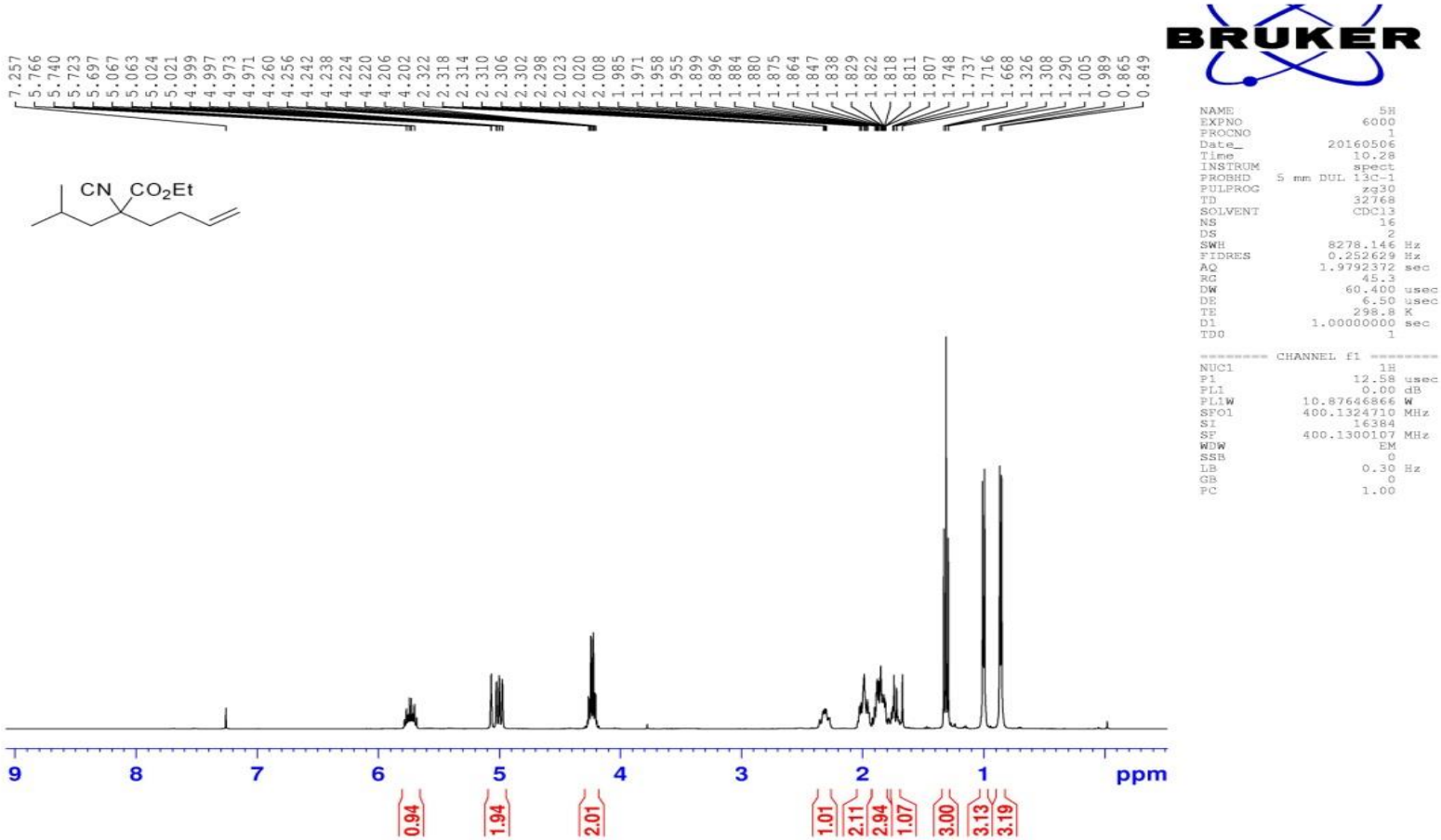
1e



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TE	298.8	K
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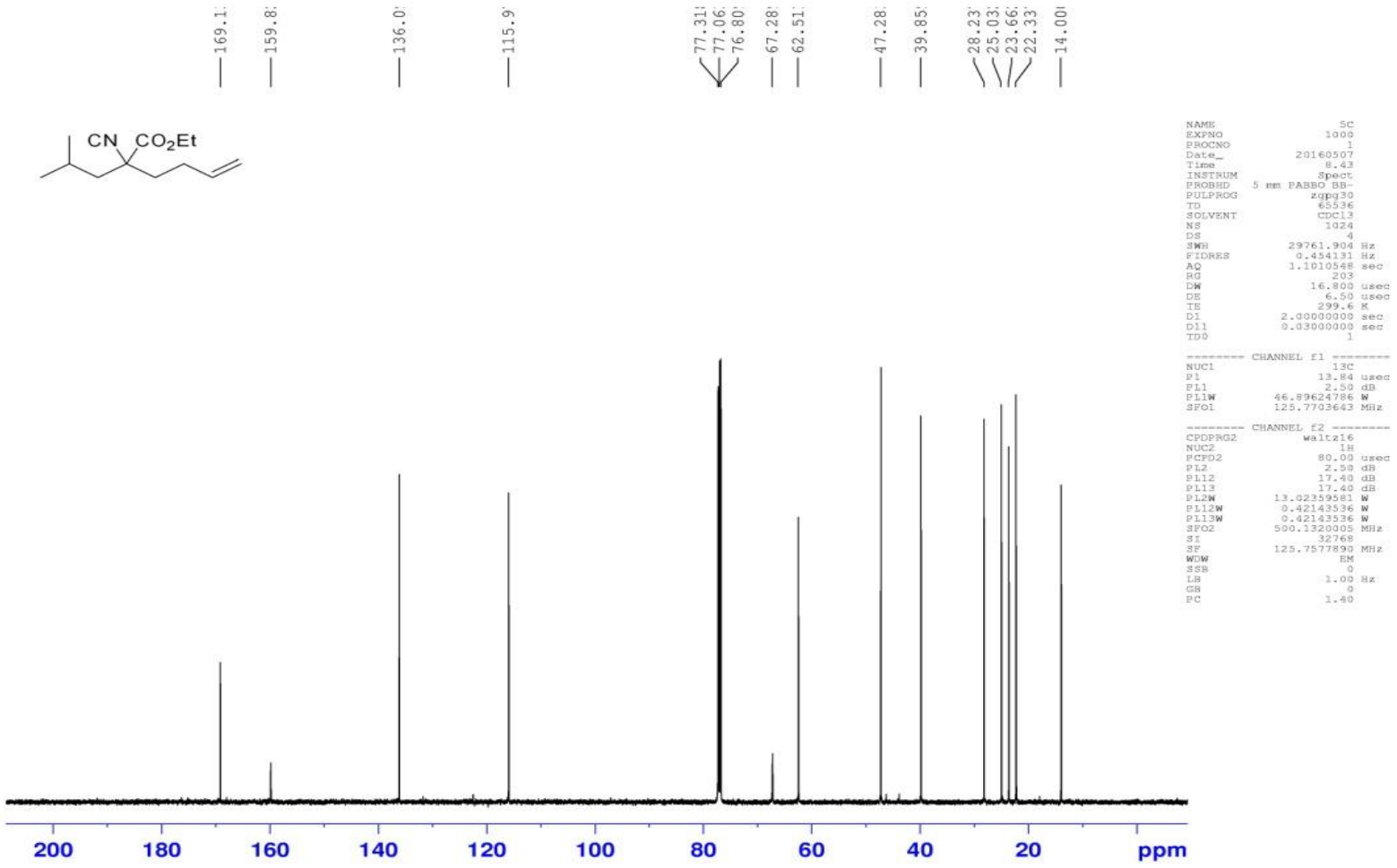

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RG	203
DW	16.800 usec
DE	6.50 usec
TE	299.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
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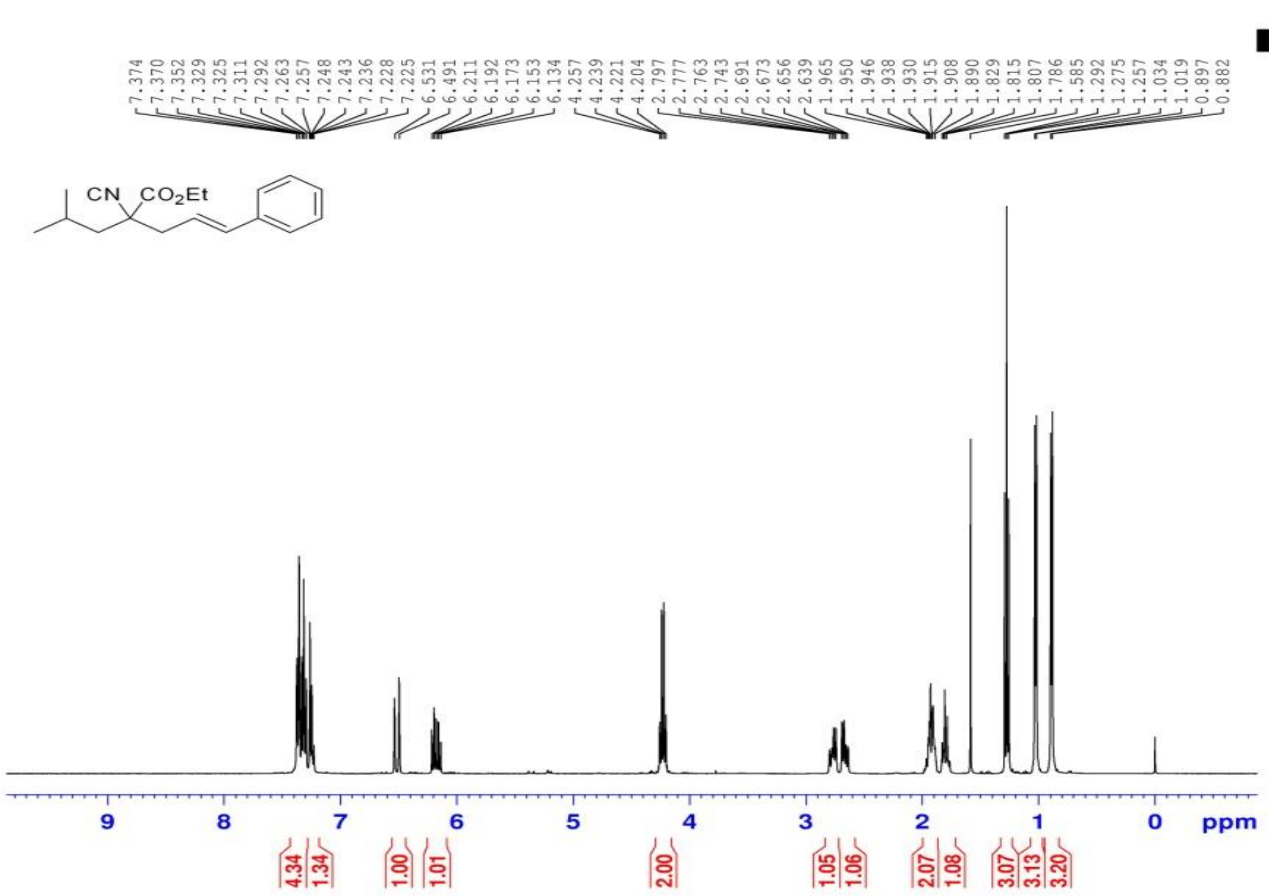
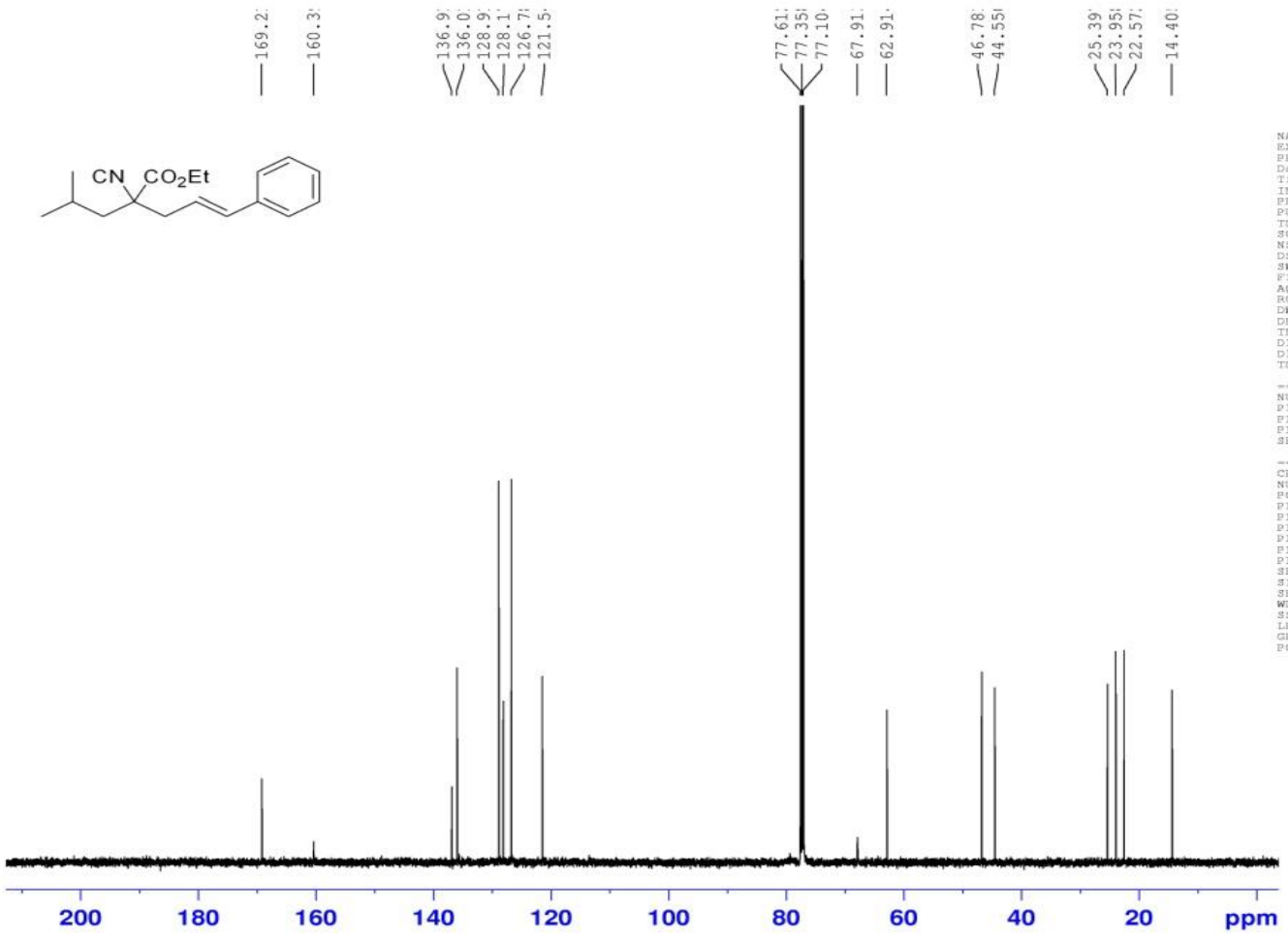
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1f

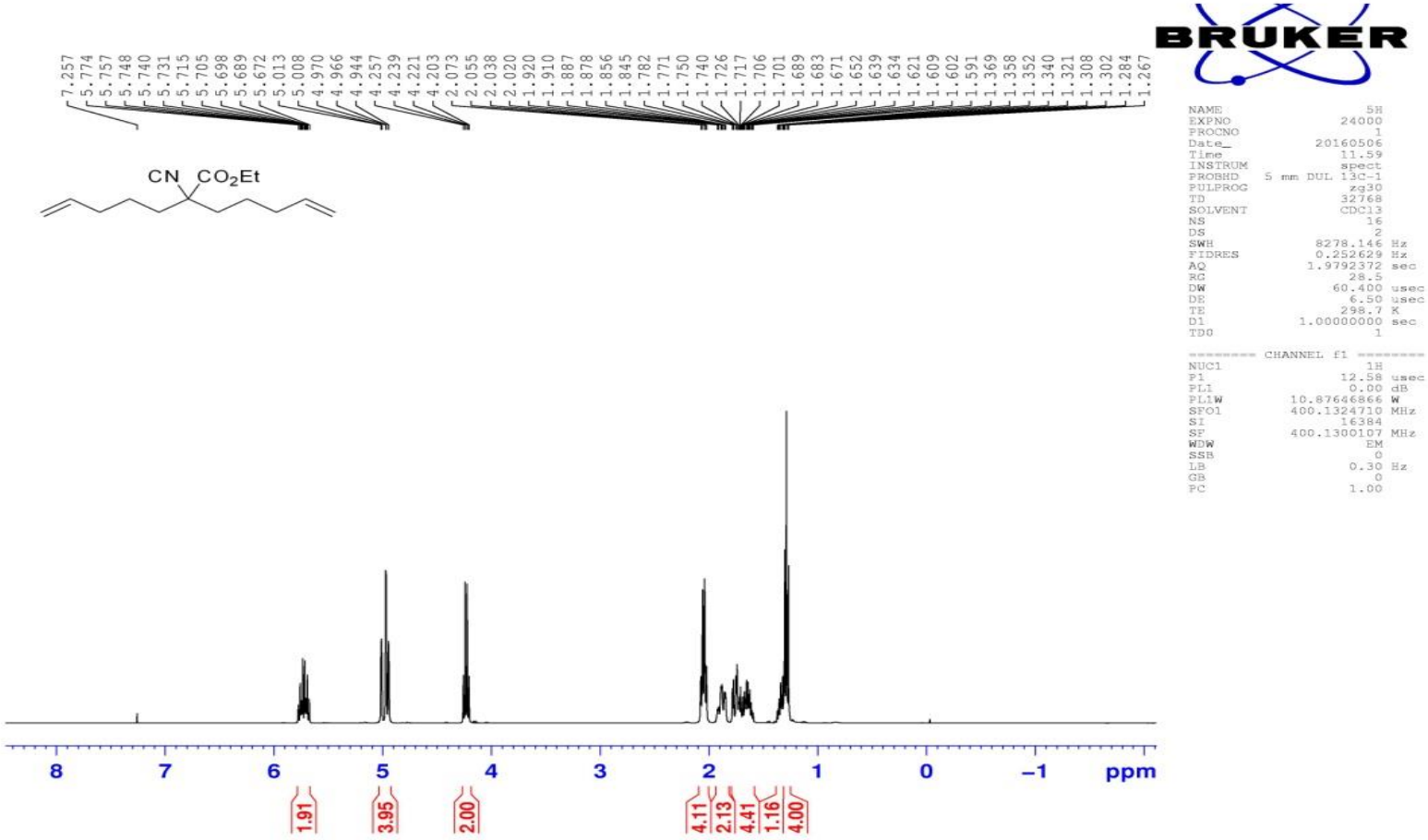


NAME	5H	
EXPNO	29000	
PROCNO	1	
Date_	20160506	
Time	13.30	
INSTRUM	spect	
PROBHD	5 mm DUL 13C-1	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8278.146	Hz
FIDRES	0.252629	Hz
AQ	1.9792372	sec
RG	128	
DW	60.400	usec
DE	6.50	usec
TE	298.9	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	12.58	usec
PL1	0.00	dB
PL1W	10.87646866	W
SFO1	400.1324710	MHz
SI	16384	
SF	400.1300104	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

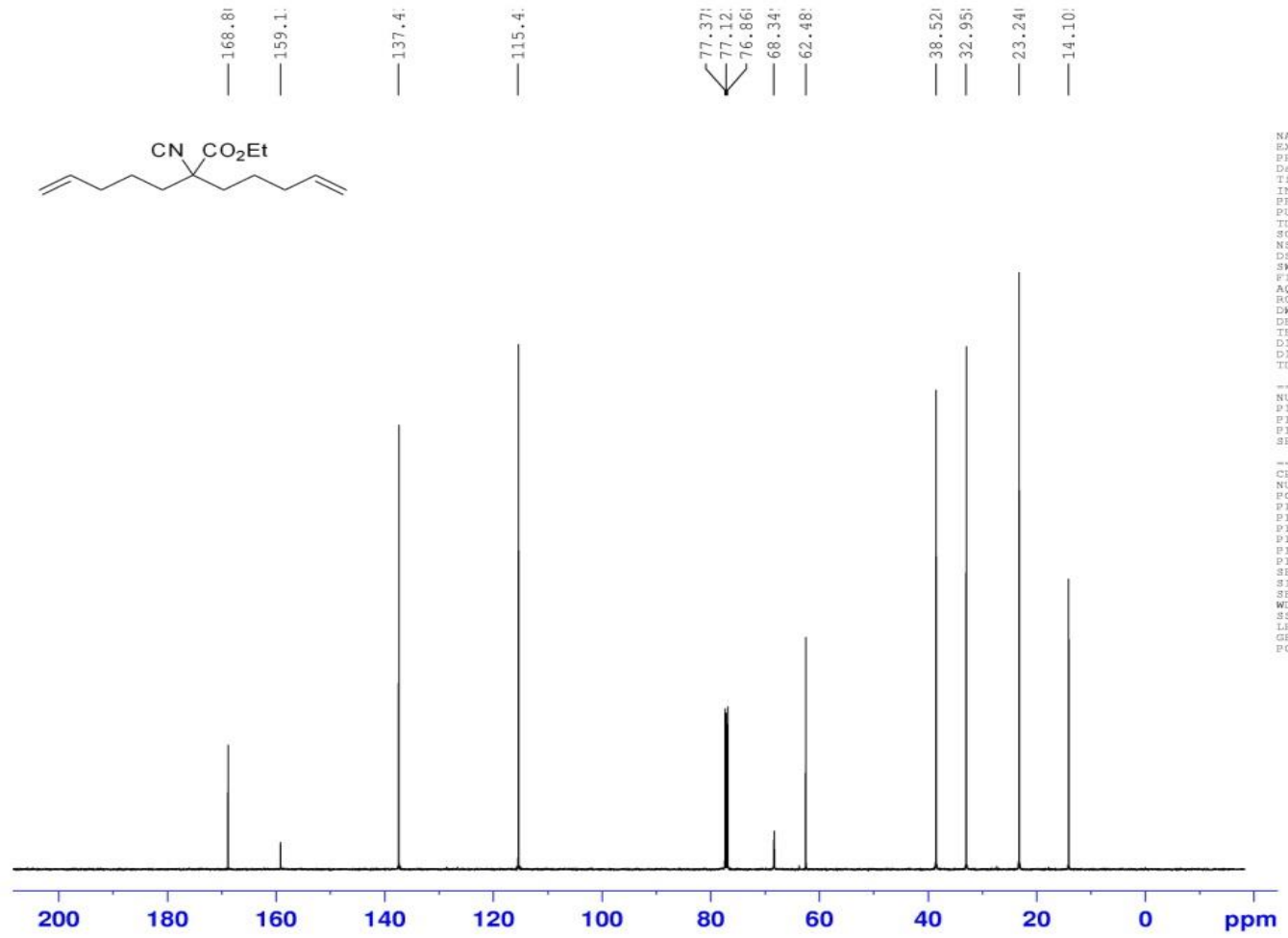



```
NAME                  5C
EXPNO               17000
PROCNO                  1
Date_            20160508
Time                3.48
INSTRUM             Spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG                   203
DW               16.800 usec
DE                6.50 usec
TE               299.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 13.84 usec
PL1                2.50 dB
PL1W       46.89624786 W
SFO1       125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                2.50 dB
PL12              17.40 dB
PL13              17.40 dB
PL2W       13.02359581 W
PL12W       0.42143536 W
PL13W       0.42143536 W
SFO2       500.1320005 MHz
SI                32768
SF         125.7577488 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

1g

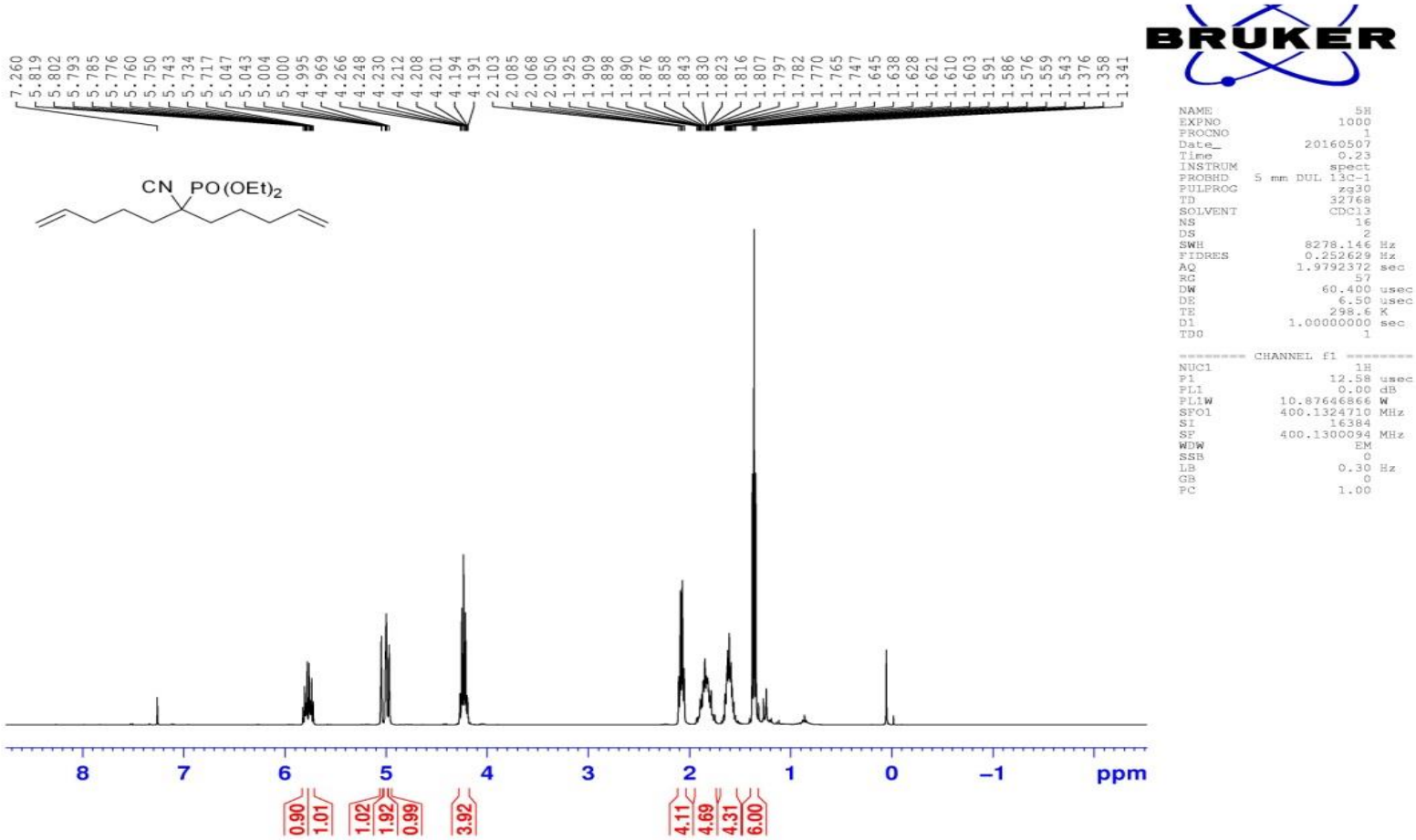



```
NAME                  5H
EXPNO                 24000
PROCNO                1
Date_                 20160506
Time                  11.59
INSTRUM               spect
PROBHD     5 mm DUL 13C-1
PULPROG               zg30
TD                    32768
SOLVENT               CDCl3
NS                    16
DS                    2
SWH                   8278.146 Hz
FIDRES                0.252629 Hz
AQ                    1.9792372 sec
RG                    28.5
DW                    60.400 usec
DE                    6.50 usec
TE                    298.7 K
D1                    1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                    12.58 usec
PL1                   0.00 dB
PL1W                  10.87646866 W
SFO1                  400.1324710 MHz
SI                    16384
SF                    400.1300107 MHz
WDW                   EM
SSB                   0
LB                    0.30 Hz
GB                    0
PC                    1.00
```



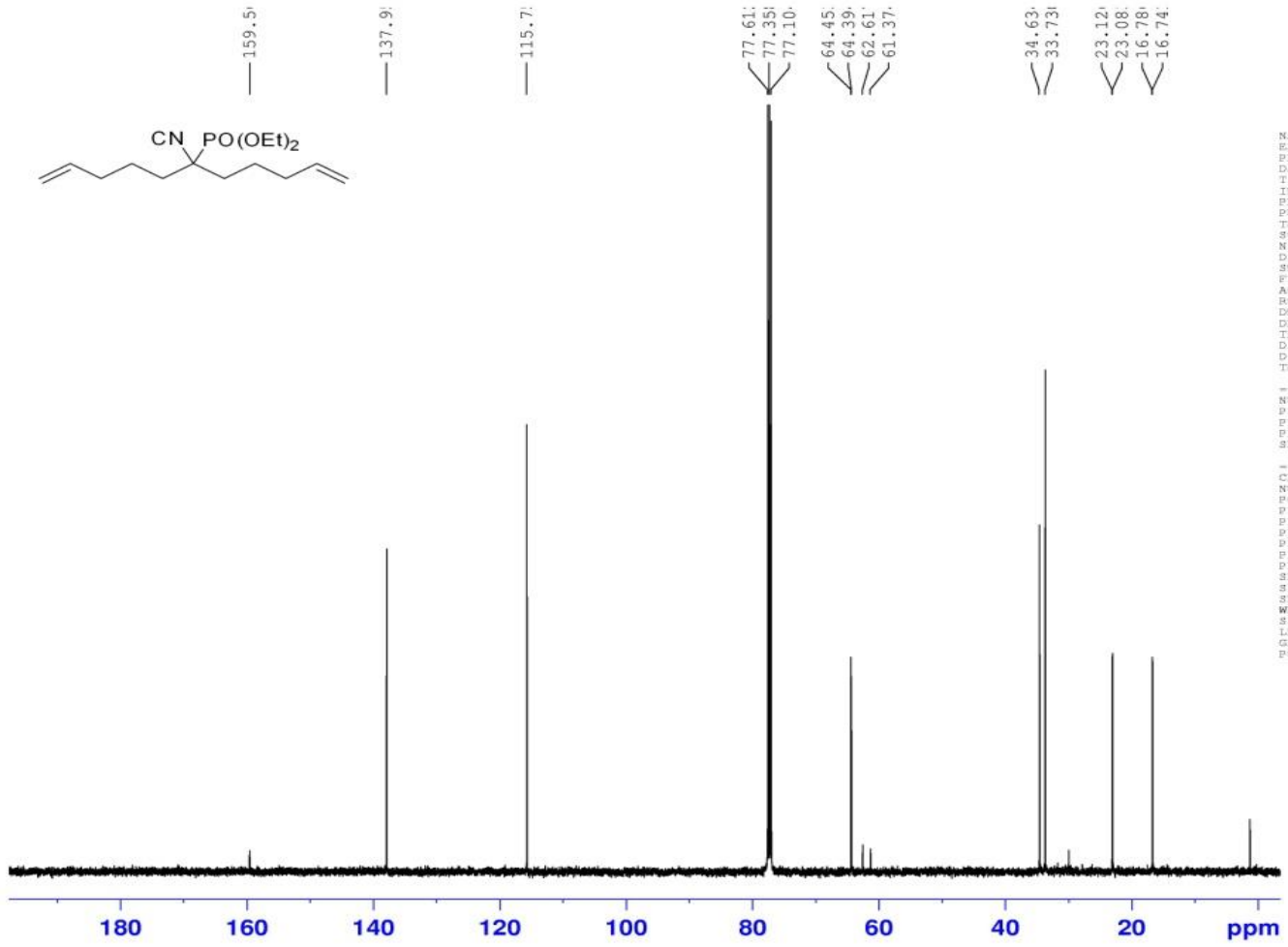
NAME	5C
EXPNO	8000
PROCNO	1
Date_	20160507
Time	13.37
INSTRUM	3peot
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	299.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
--------	CHANNEL f1 --------
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
--------	CHANNEL f2 --------
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1h



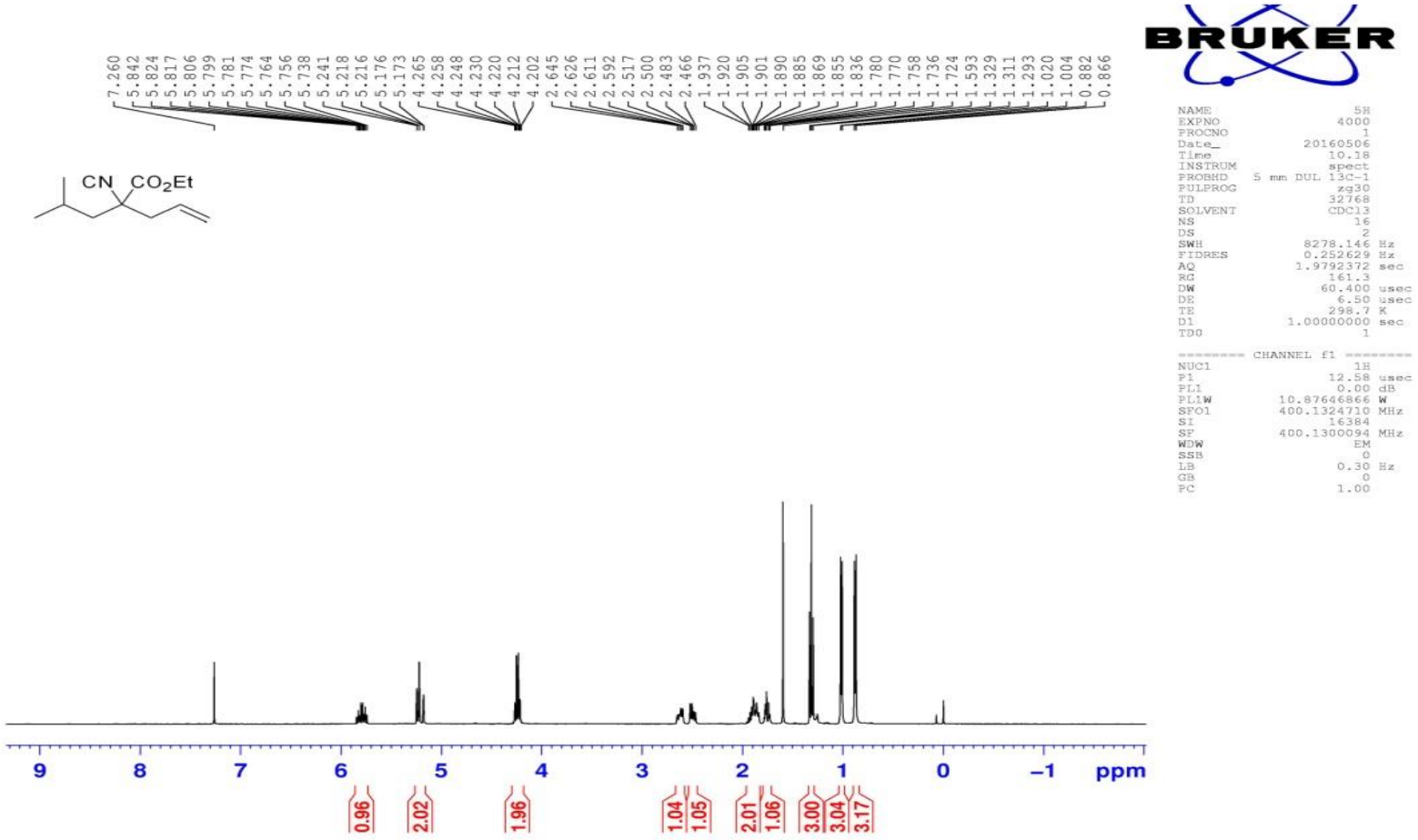
BRUKER

NAME	5H	
EXPNO	1000	
PROCNO	1	
Date_	20160507	
Time	0.23	
INSTRUM	spect	
PROBHD	5 mm DUL 13C-1	
PULPROG	zg30	
TD	32768	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8278.146	Hz
FIDRES	0.252629	Hz
AQ	1.9792372	sec
RG	57	
DW	60.400	usec
DE	6.50	usec
TE	298.6	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	12.58	usec
PL1	0.00	dB
PL1W	10.87646866	W
SFO1	400.1324710	MHz
SI	16384	
SF	400.1300094	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

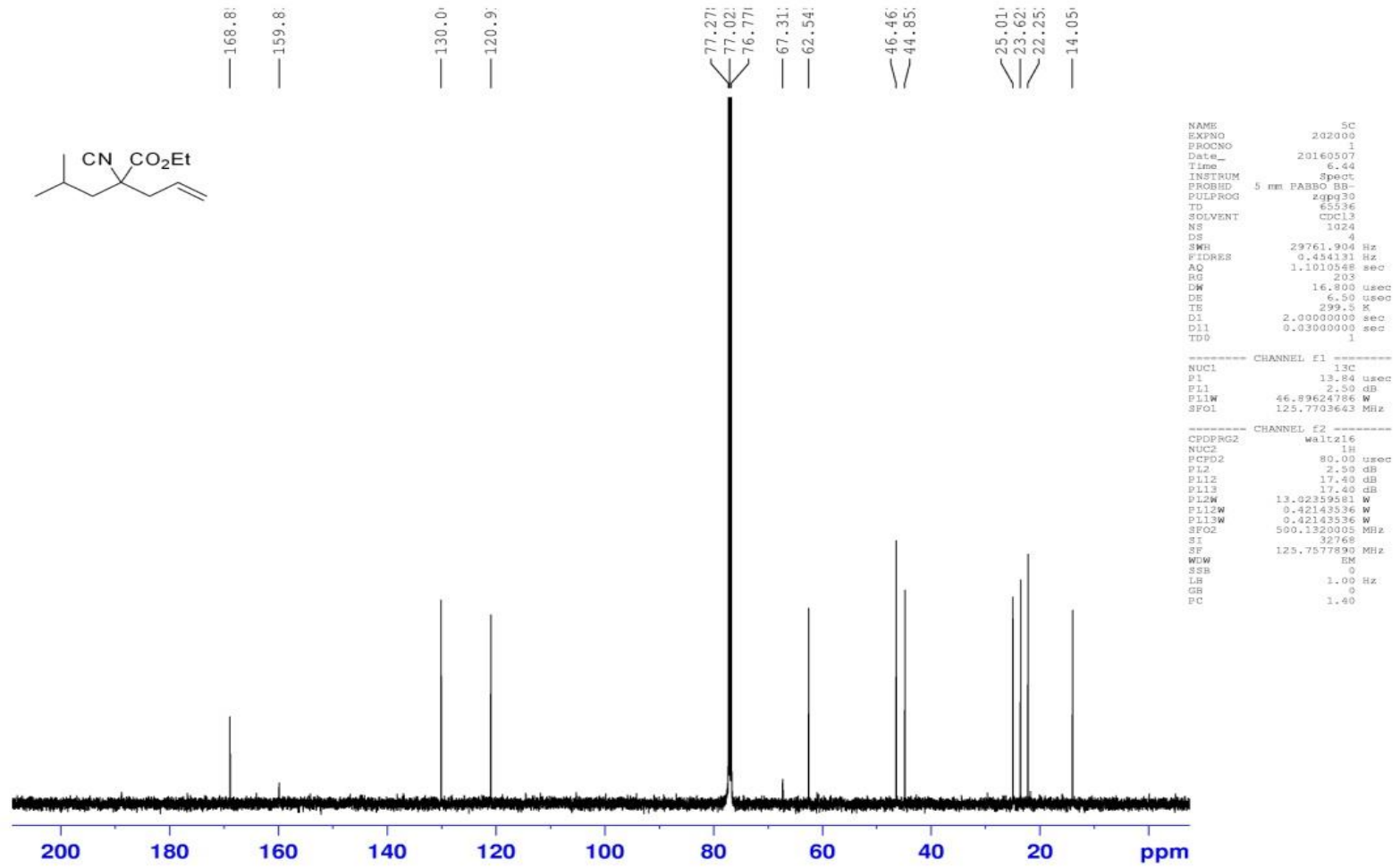


NAME	5C
EXPNO	2000
PROCNO	1
Date_	20160508
Time	18.18
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	299.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577488 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1i

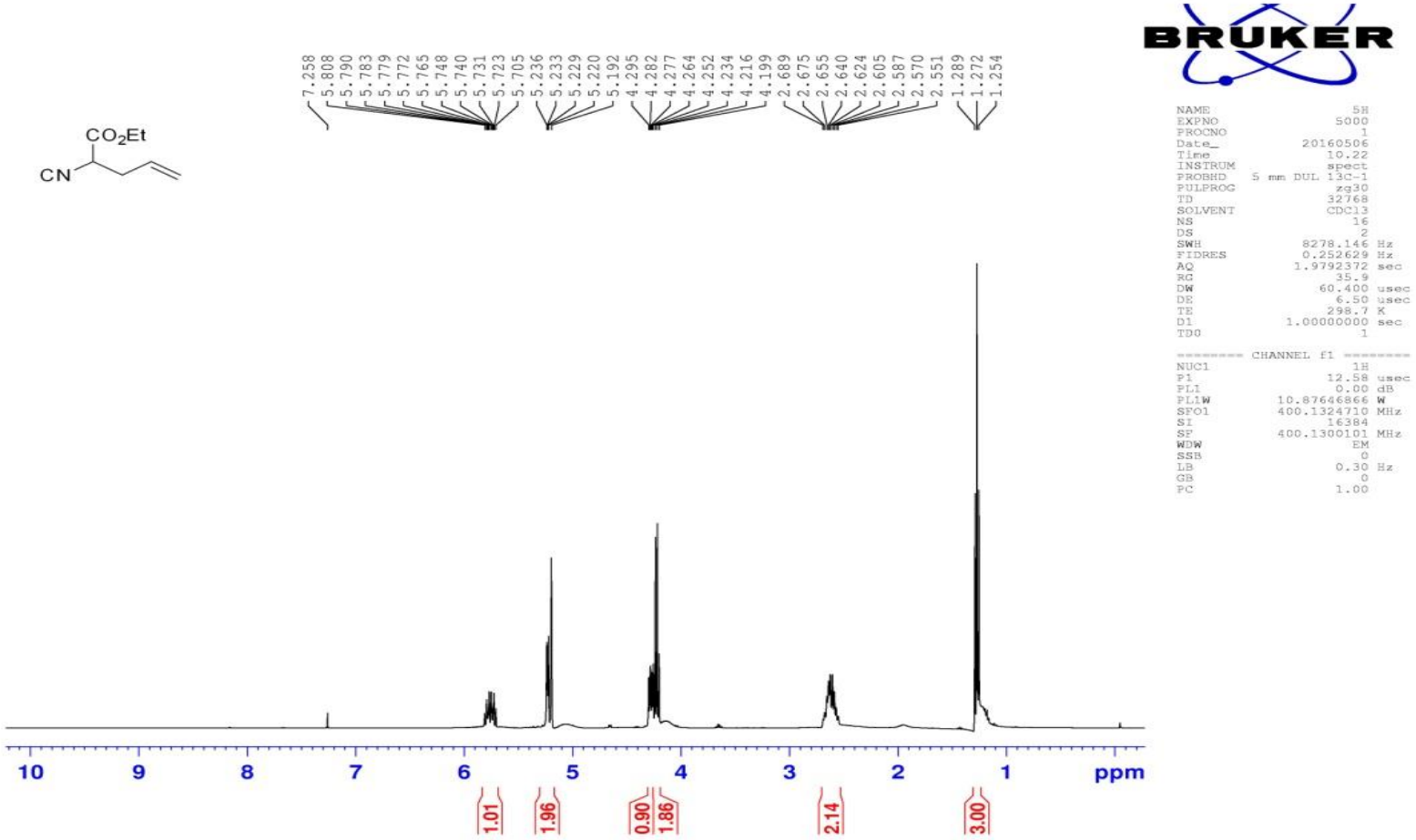


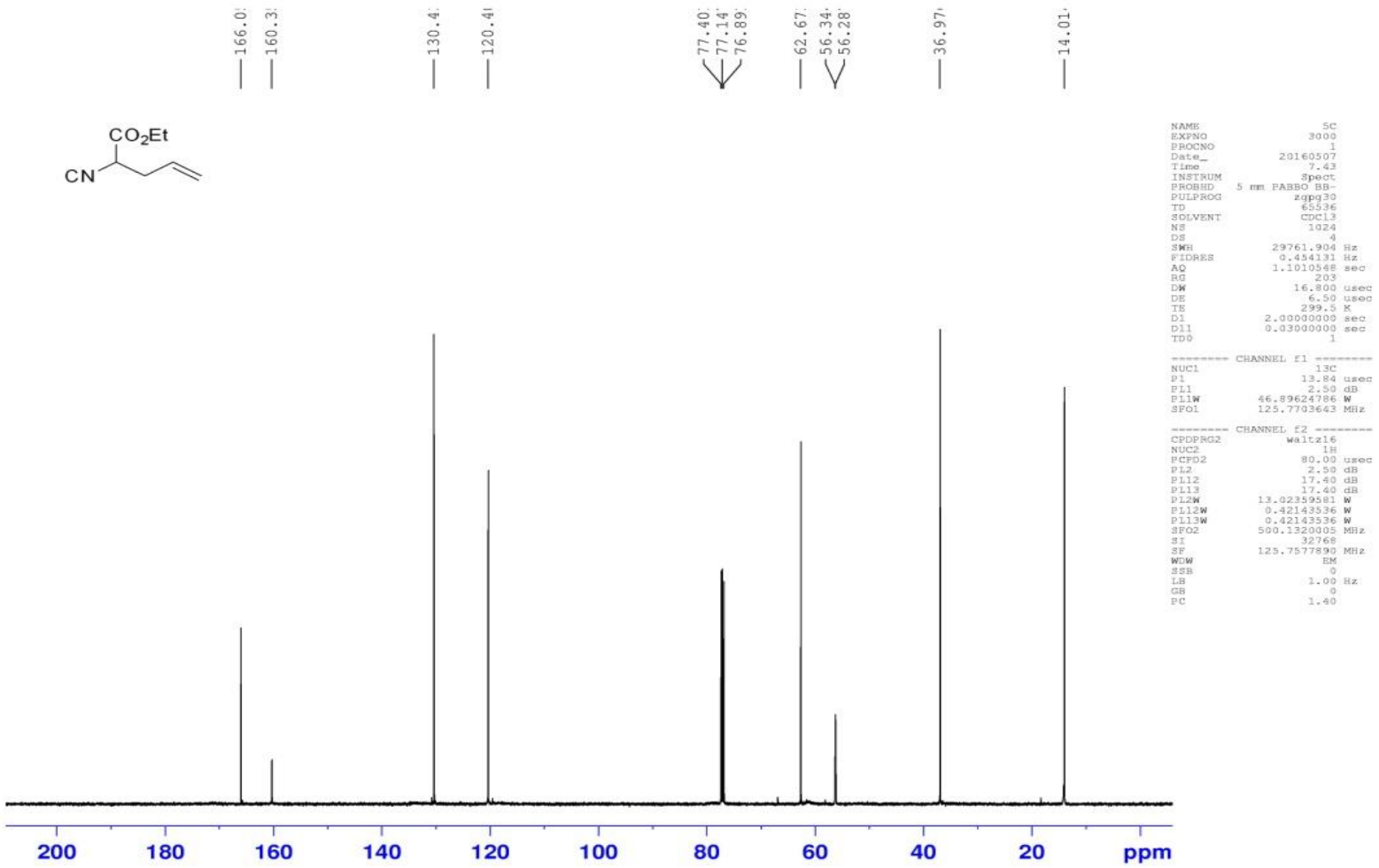

```
NAME                  5H
EXPNO               4000
PROCNO                 1
Date_           20160506
Time               10.18
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8278.146 Hz
FIDRES        0.252629 Hz
AQ            1.9792372 sec
RG                161.3
DW                60.400 usec
DE                 6.50 usec
TE                298.7 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                12.58 usec
PL1                0.00 dB
PL1W       10.87646866 W
SFO1       400.1324710 MHz
SI                16384
SF         400.1300094 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



NAME	SC
EXPNO	202000
PROCNO	1
Date_	20160507
Time	6.44
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	299.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42142536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

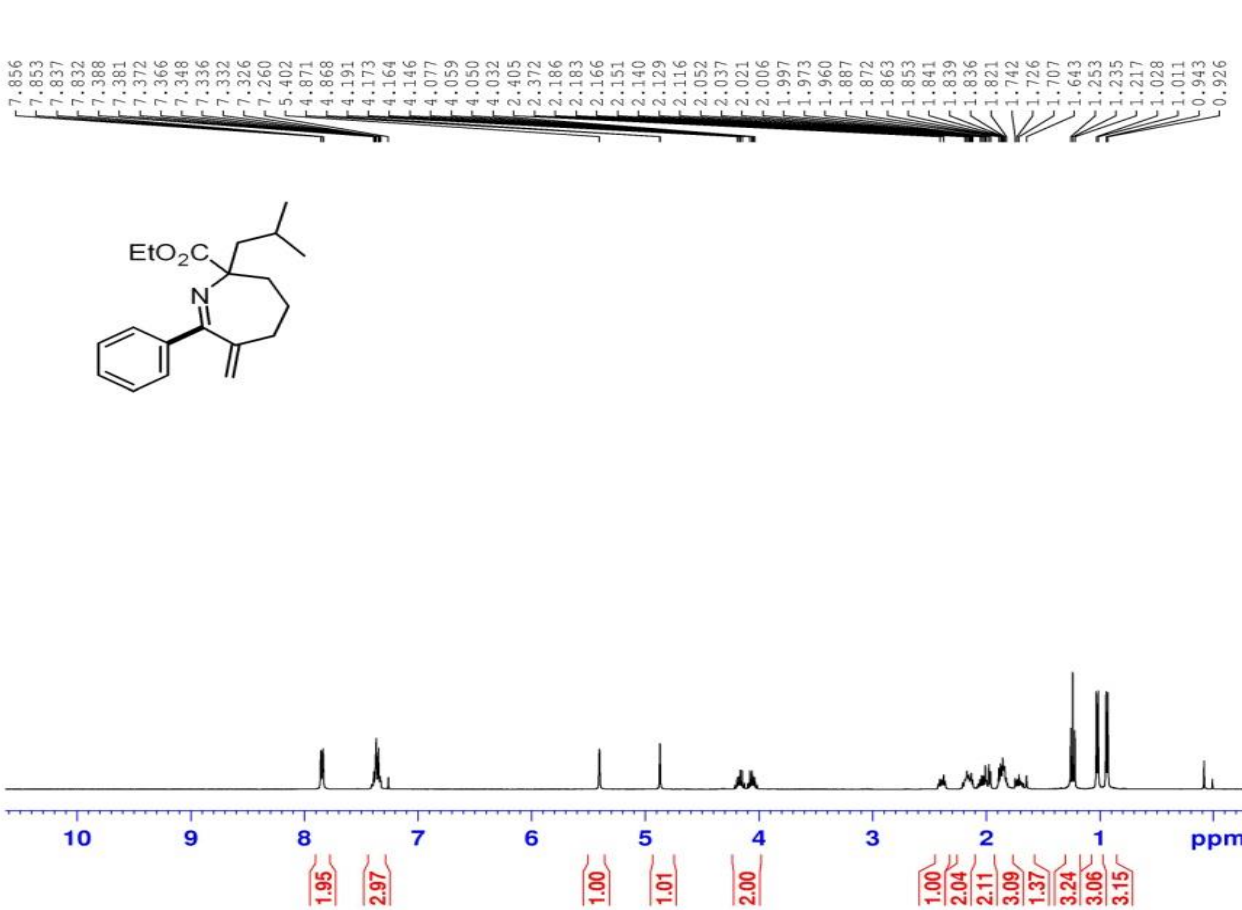
1j


7.258
5.808
5.790
5.783
5.779
5.772
5.765
5.748
5.740
5.731
5.723
5.705
5.236
5.233
5.229
5.220
5.192
4.295
4.282
4.277
4.264
4.252
4.234
4.216
4.199
2.689
2.675
2.655
2.640
2.624
2.605
2.587
2.570
2.551
1.289
1.272
1.254
CO2Et
CN
BRUKER
NAME 5H
EXPNO 5000
PROCNO 1
Date_ 20160506
Time 10.22
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 35.9
DW 60.400 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 16384
SF 400.1300101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.01
1.96
0.90
1.86
2.14
3.00



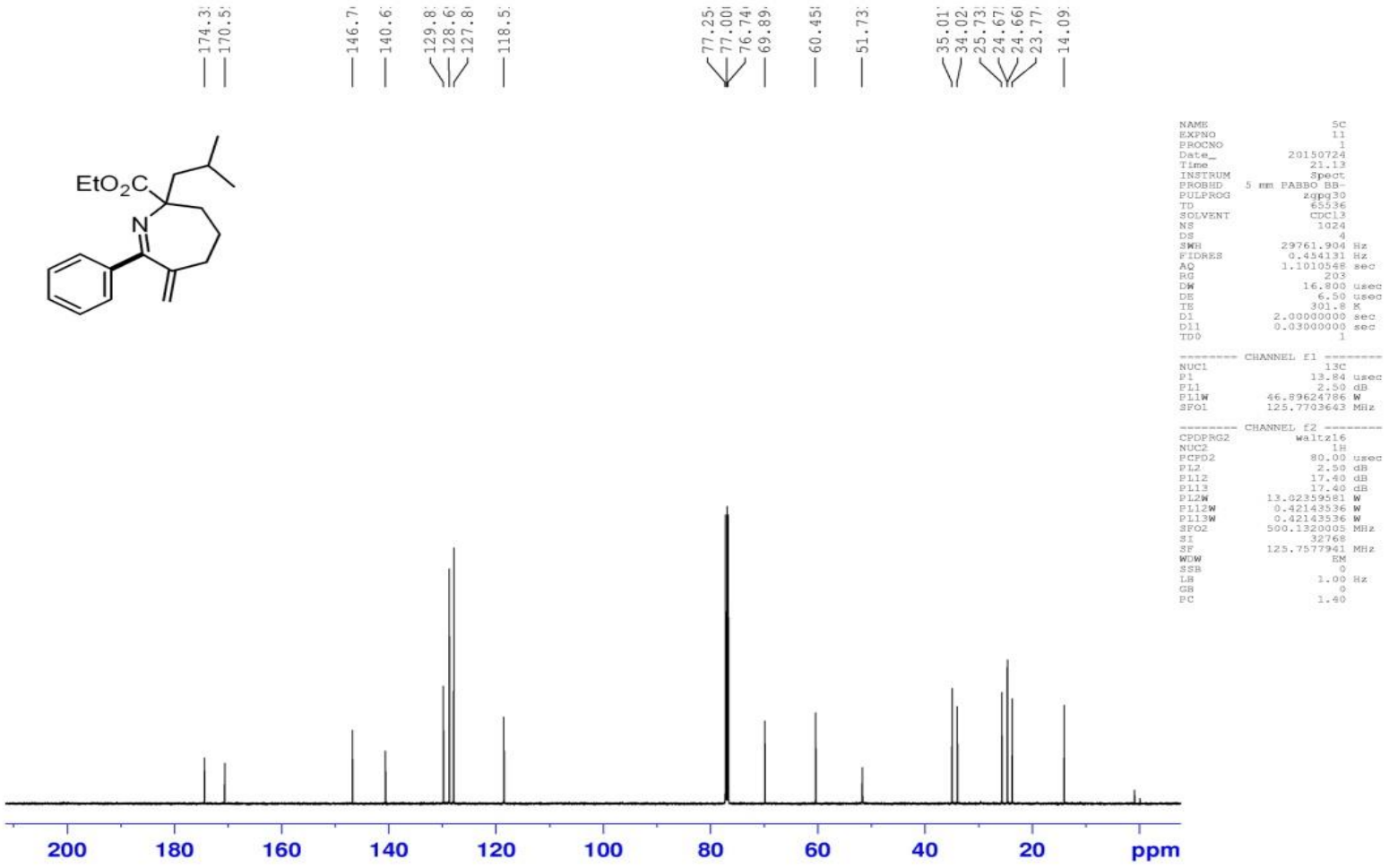
NAME	SC
EXPNO	3000
PROCNO	1
Date_	20160507
Time	7.43
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	299.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

3a



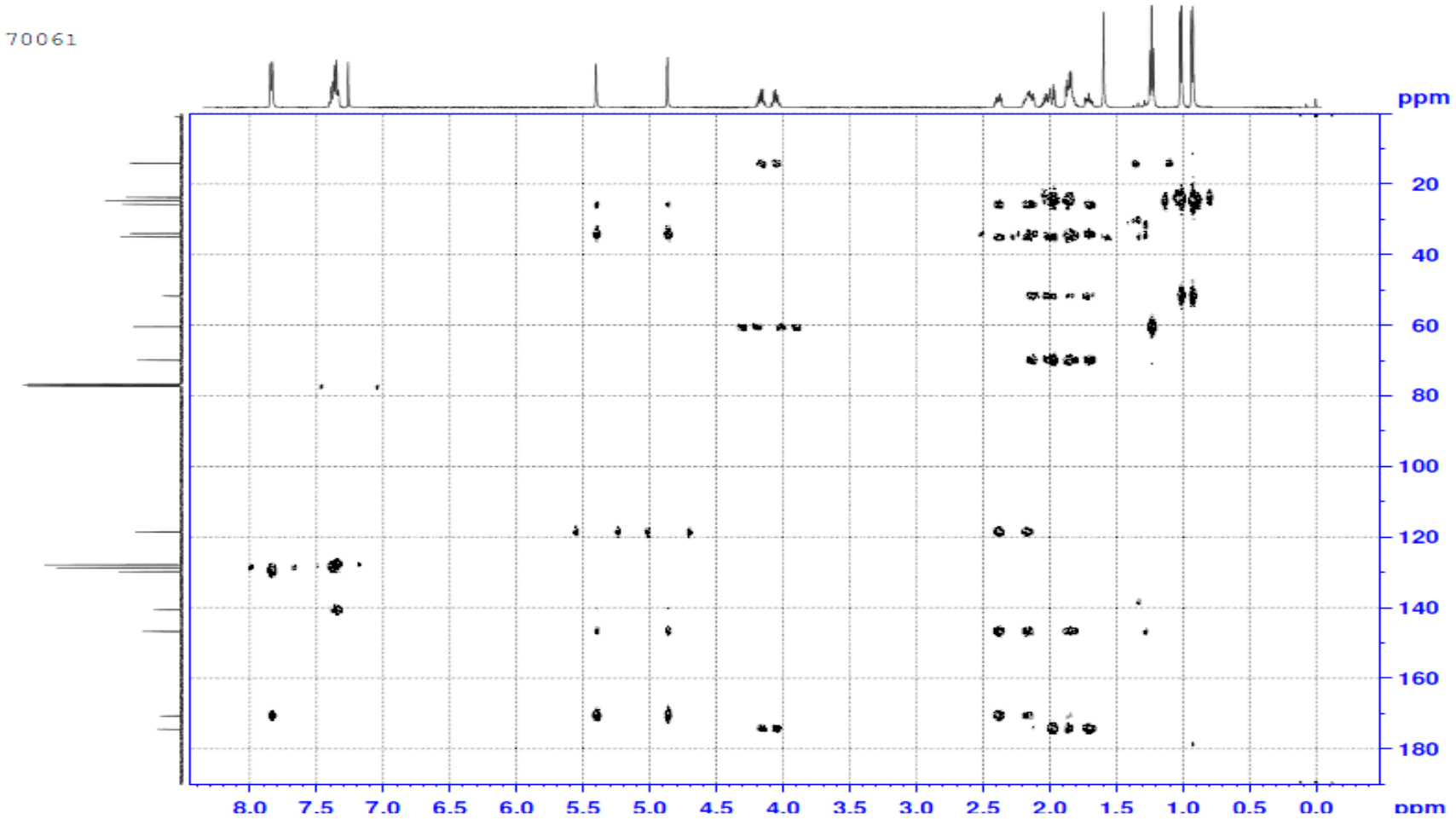
BRUKER

NAME	0007211
EXPNO	65
PROCNO	1
Date_	20150723
Time	18.16
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	71.8
DW	60.400 usec
DE	6.50 usec
TE	300.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300093 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

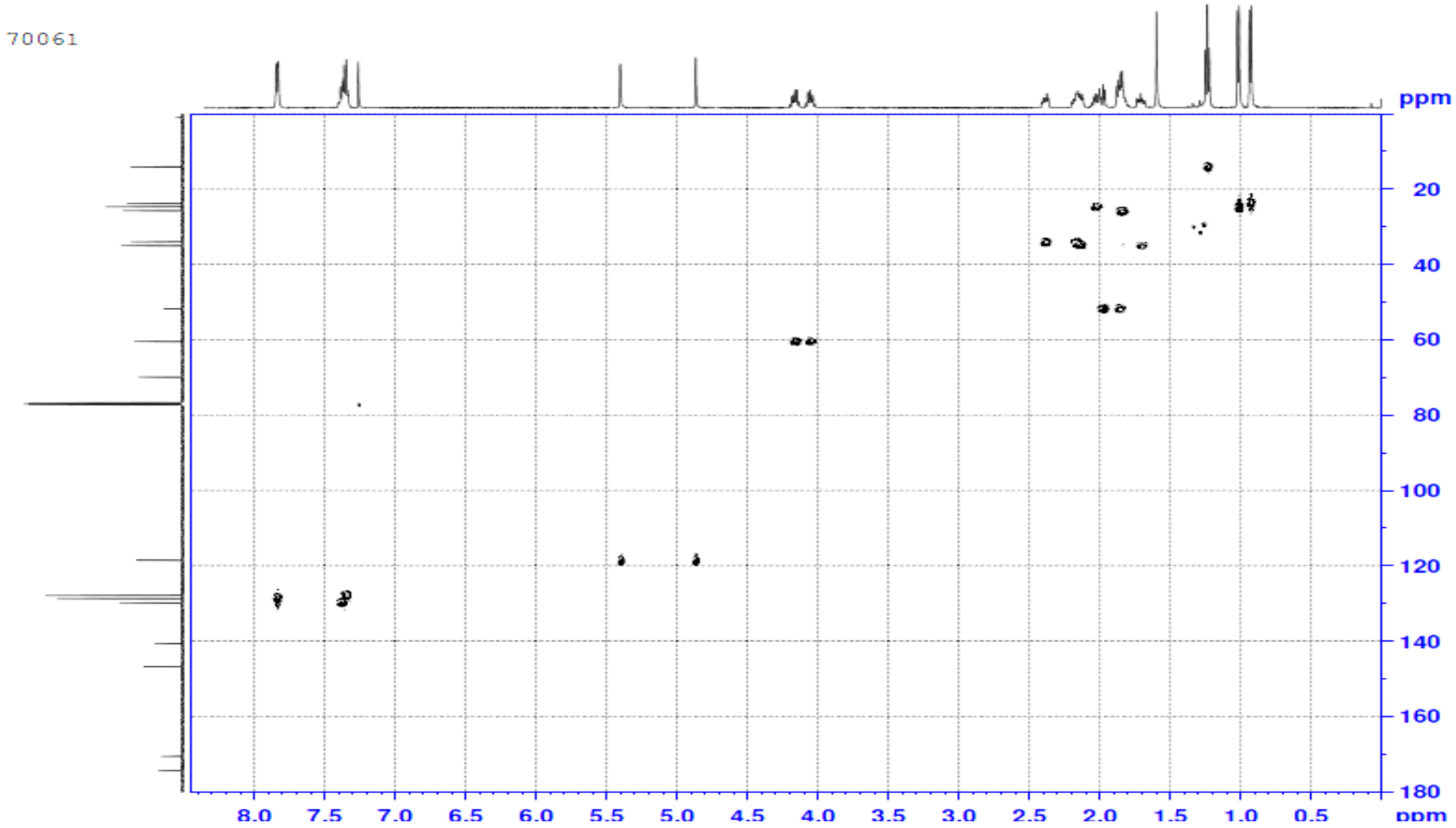


NAME	5C	
EXPNO	11	
PROCNO	1	
Date_	20150724	
Time	21.13	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	1024	
DS	4	
SWH	29761.904	Hz
FIDRES	0.454131	Hz
AQ	1.1010548	sec
RG	203	
DW	16.800	usec
DE	6.50	usec
TE	301.8	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	13.84	usec
PL1	2.50	dB
PL1W	46.89624786	W
SFO1	125.7703643	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	2.50	dB
PL12	17.40	dB
PL13	17.40	dB
PL2W	13.02359581	W
PL12W	0.42143536	W
PL13W	0.42143536	W
SFO2	500.1320005	MHz
SI	32768	
SF	125.7577941	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

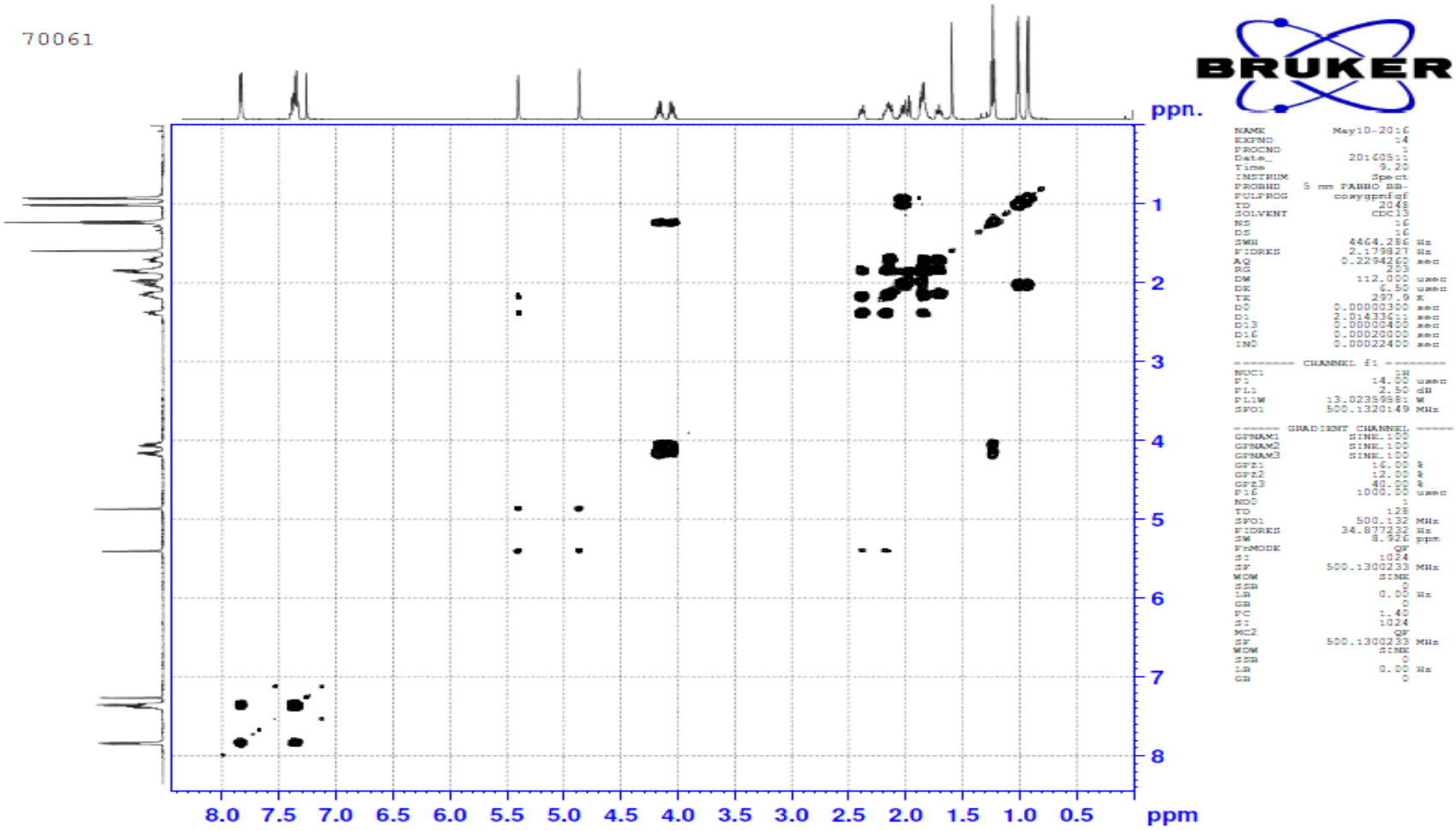
3a-HMBC


70061
ppm
20
40
60
80
100
120
140
160
180
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

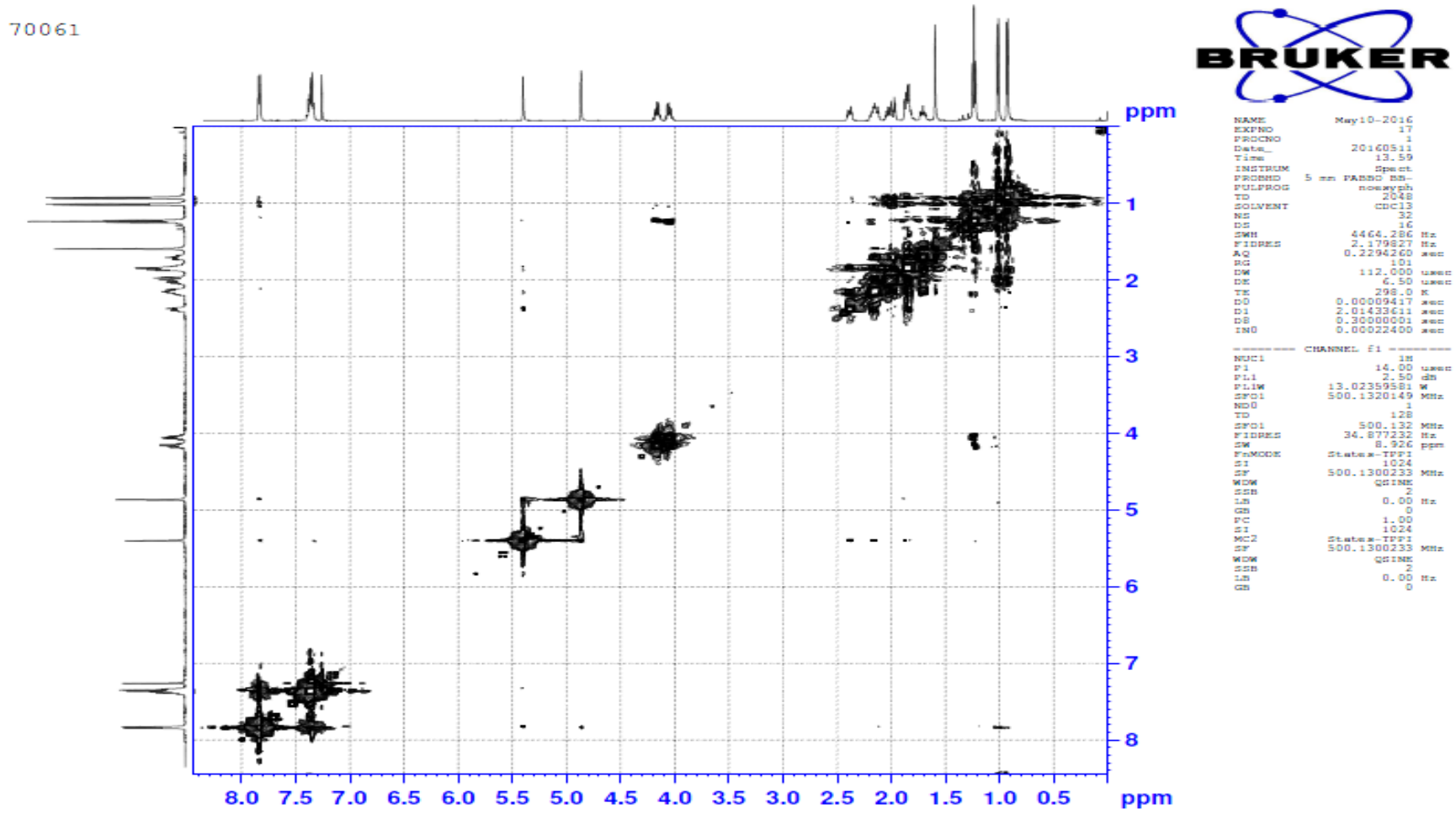
3a-HSQC


70061
ppm
20
40
60
80
100
120
140
160
180
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

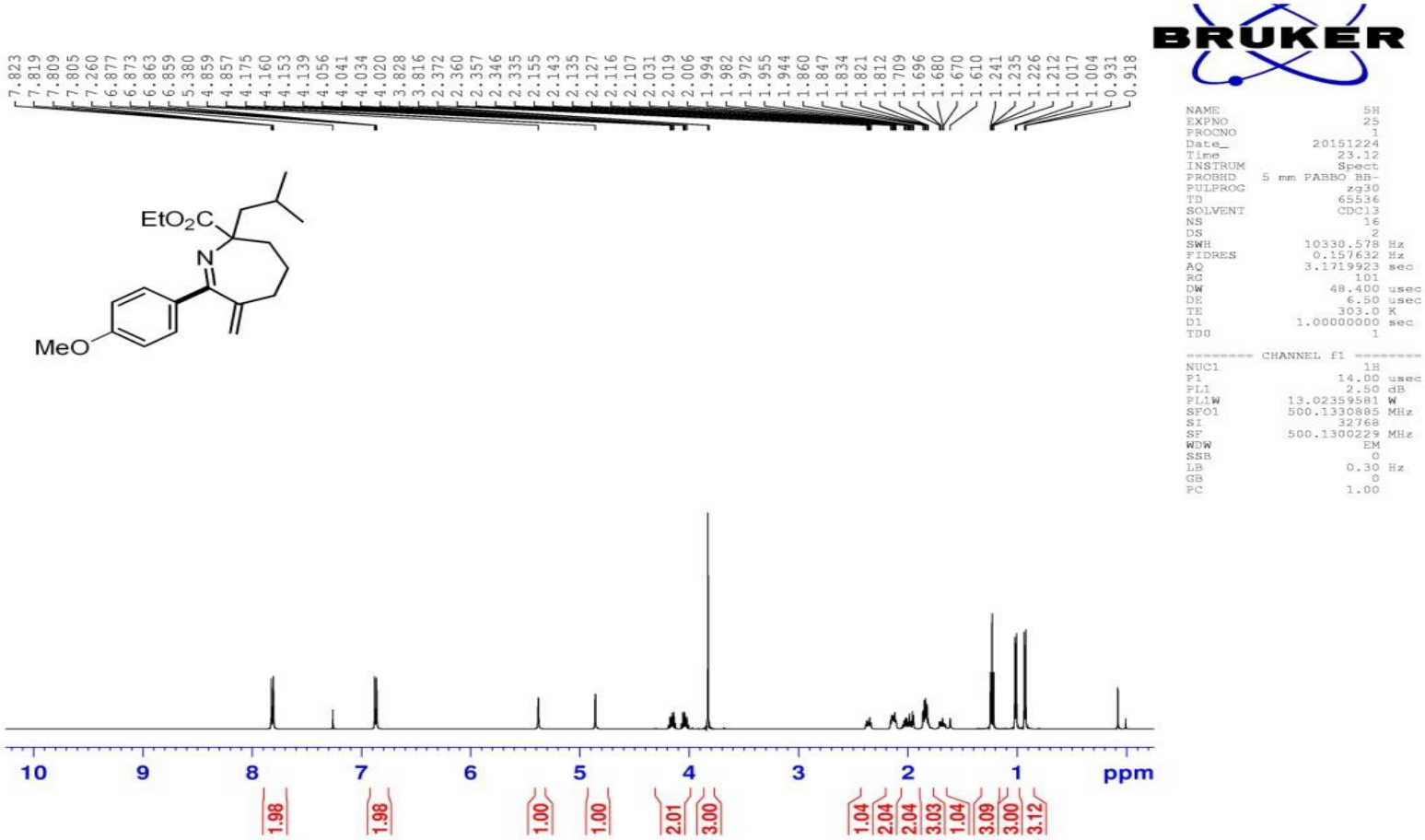
3a-COSY


70061
ppn.
1
2
3
4
5
6
7
8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
ppm
BRUKER
NAME May10-2016
EXPNO 14
PROCNO 1
Date_ 20160511
Time 9.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG coxyqpndqf
TD 2048
SOLVENT CDCl3
NS 16
DS 16
SWH 4464.286 Hz
FIDRES 2.179827 Hz
AQ 0.2294260 sec
RG 203
DW 112.000 usec
DE 6.50 usec
TE 297.9 K
D0 0.00000300 sec
D1 2.0143341 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00022400 sec
CHANNEL f1
NUC1 1H
P1 14.00 usec
PL1 2.50 dB
PLW1 13.02359581 W
SFO1 500.1320149 MHz
GRADIENT CHANNEL
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPZ1 16.00 %
GPZ2 12.00 %
GPZ3 40.00 %
P16 1000.00 usec
NDD 1
TD 128
SFO1 500.132 MHz
FIDRES 34.877222 Hz
SW 8.926 ppm
FnMODE QF
S1 1024
SF 500.1300233 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
S2 1024
MC2 QF
SF 500.1300233 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

3a-NOE

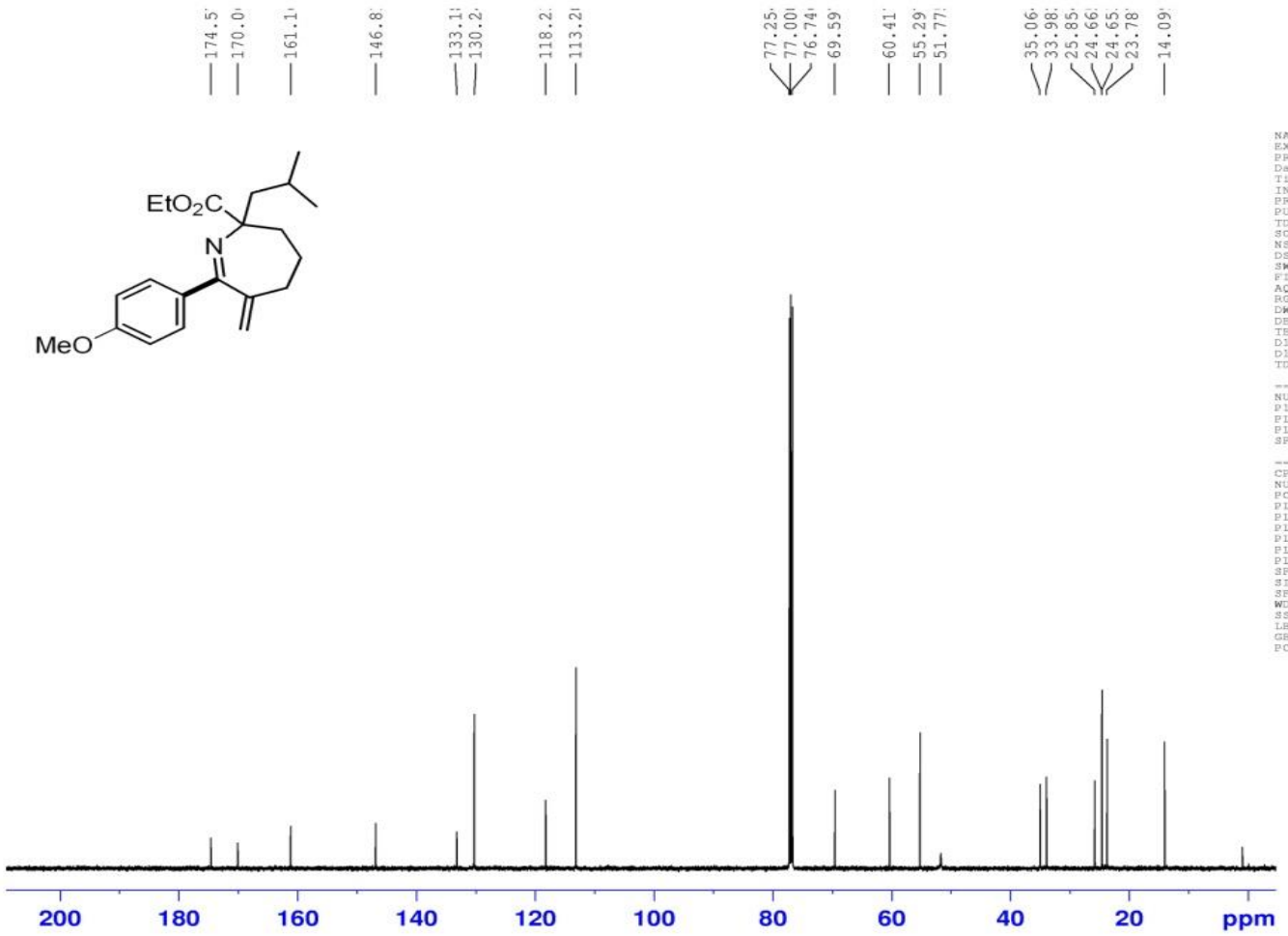

70061
ppm
1
2
3
4
5
6
7
8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
ppm
BRUKER
NAME May10-2016
EXPNO 17
PROCNO 1
Date_ 20160511
Time 13.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG noesyph
TD 2048
SOLVENT CDCl3
NS 32
DS 16
SWH 4464.286 Hz
FIDRES 2.179827 Hz
AQ 0.2294260 sec
RG 101
DW 112.000 usec
DE 6.50 usec
TE 298.0 K
D0 0.00009417 sec
D1 2.01433611 sec
D8 0.30000001 sec
IN0 0.00022400 sec
CHANNEL f1
NUC1 1H
P1 14.00 usec
PL1 2.50 dB
PLIW 13.02359581 W
SFO1 500.1320149 MHz
ND0 1
TD 128
SFO1 500.132 MHz
FIDRES 34.877232 Hz
SW 8.926 ppm
FnMODE States-TPPI
SI 1024
SF 500.1300233 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.00
SI 1024
MC2 States-TPPI
SF 500.1300233 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

3b



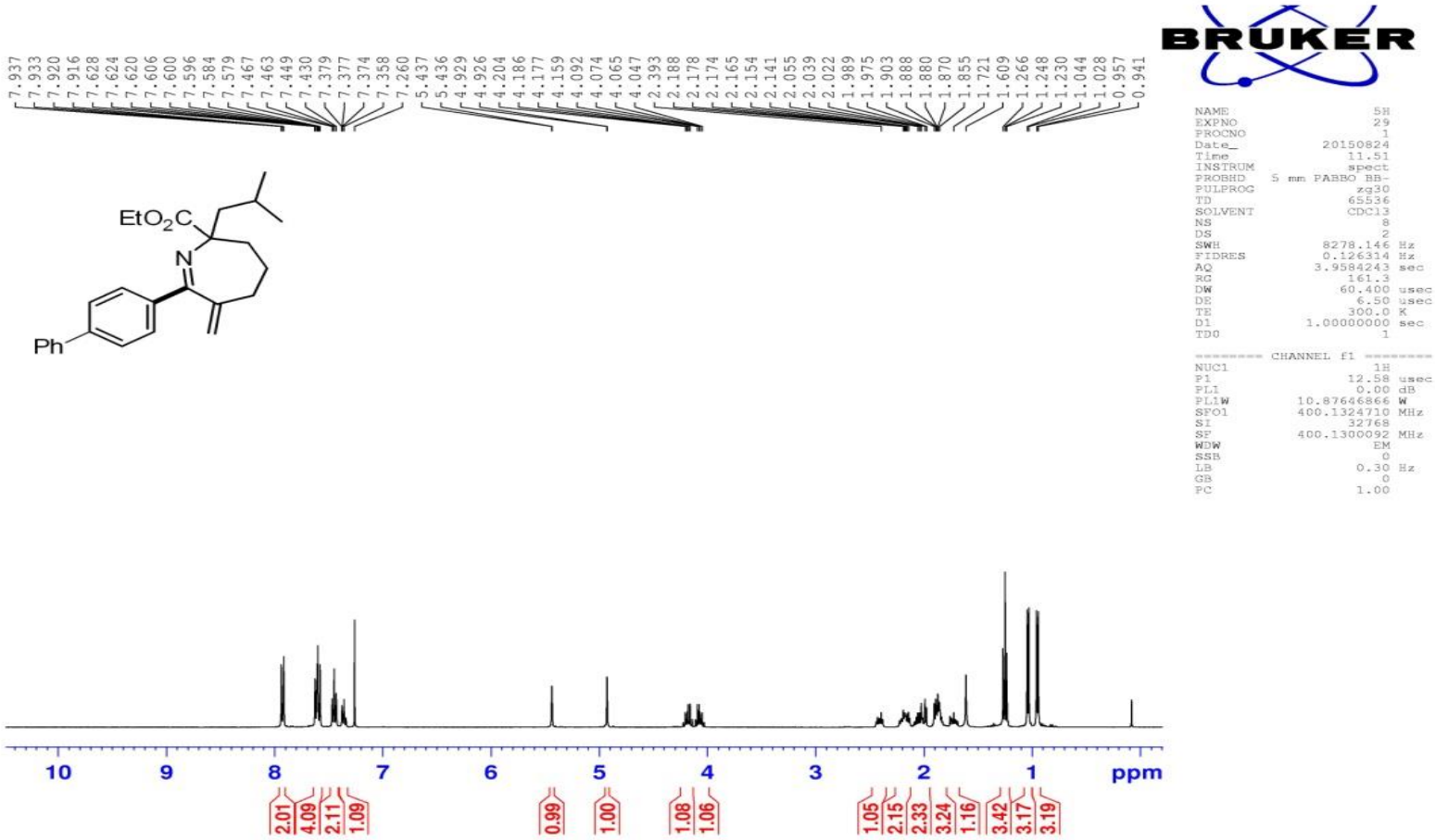
BRUKER

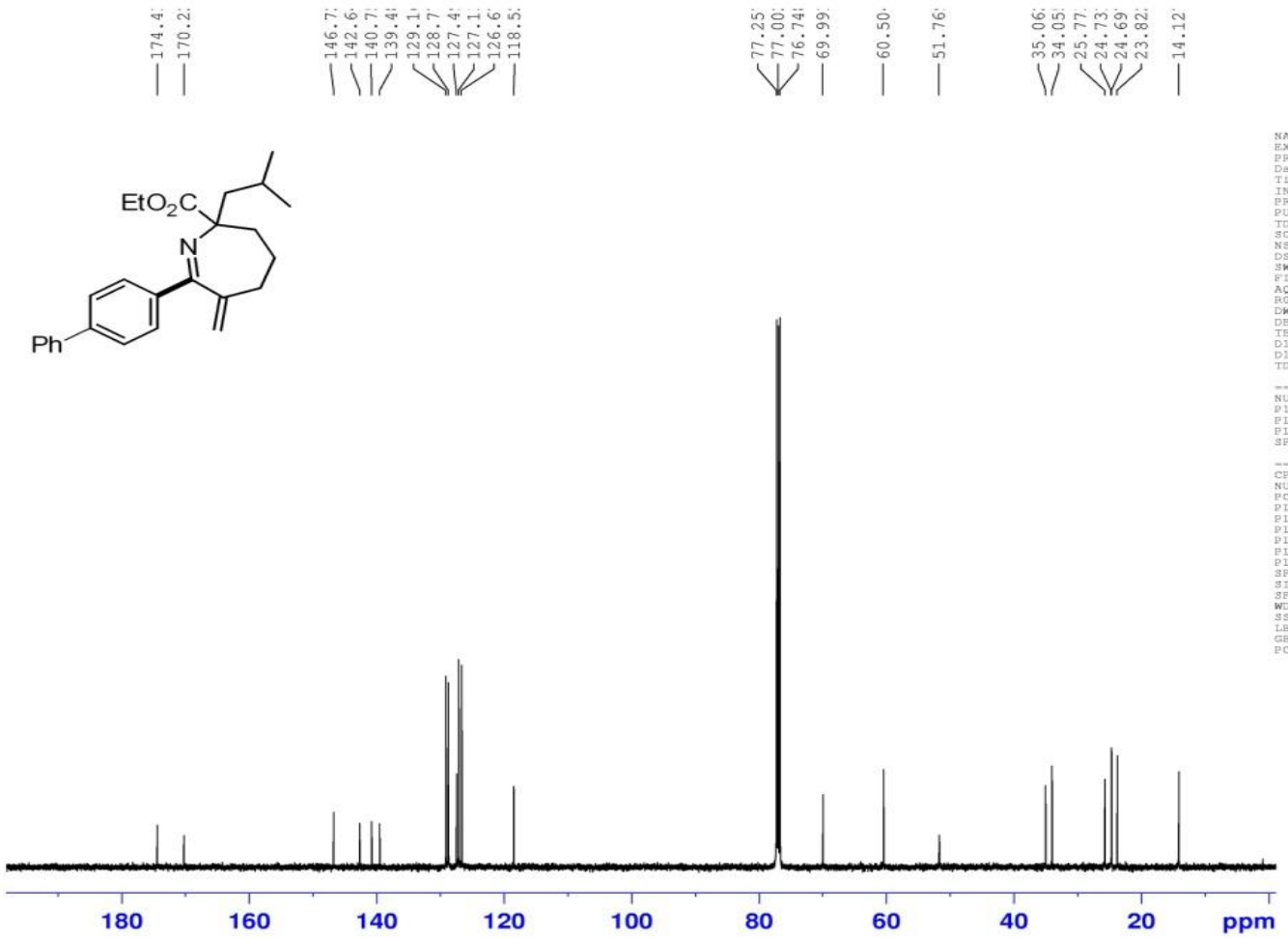
NAME	5H	
EXPNO	25	
PROCNO	1	
Date_	20151224	
Time	23.12	
INSTRUM	Spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	10330.578	Hz
FIDRES	0.157632	Hz
AQ	3.1719923	sec
RG	101	
DW	48.400	usec
DE	6.50	usec
TE	303.0	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	14.00	usec
PL1	2.50	dB
PL1W	13.02359581	W
SFO1	500.1330885	MHz
SI	32768	
SF	500.1300229	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	




```
NAME                  5C
EXPNO                 55
PROCNO                 1
Date_           20151225
Time                9.45
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG                  203
DW               16.800 usec
DE                6.50 usec
TE               294.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                13.84 usec
PL1                2.50 dB
PL1W       46.89624786 W
SFO1      125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             80.00 usec
PL2                2.50 dB
PL12              17.40 dB
PL13              17.40 dB
PL2W       13.02359581 W
PL12W       0.42143536 W
PL13W       0.42143536 W
SFO2      500.1320005 MHz
SI                32768
SF        125.7577959 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

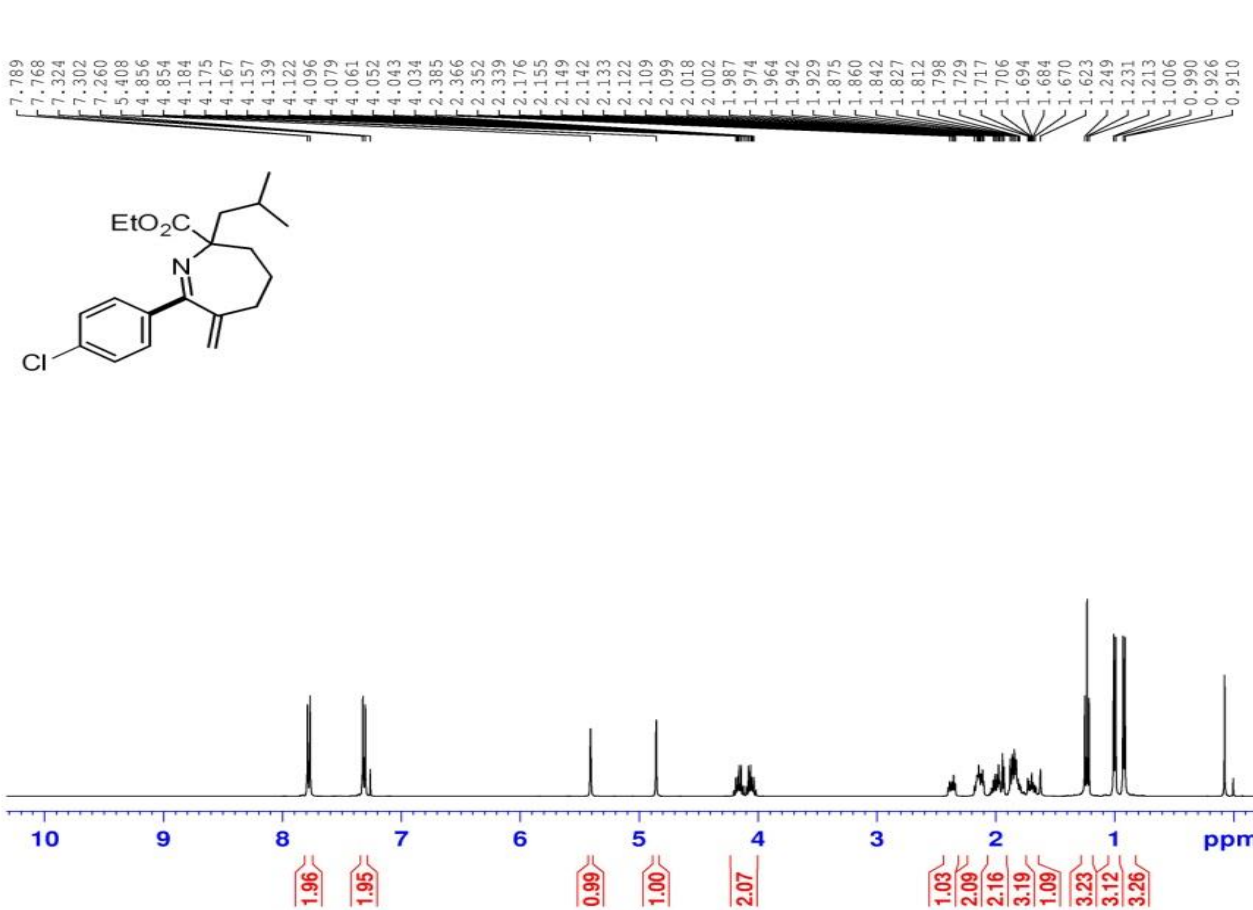
3c


7.937
7.933
7.920
7.916
7.628
7.624
7.620
7.606
7.600
7.596
7.584
7.579
7.467
7.463
7.449
7.430
7.379
7.377
7.374
7.358
7.260
5.437
5.436
4.929
4.926
4.204
4.186
4.177
4.159
4.092
4.074
4.065
4.047
2.393
2.188
2.178
2.174
2.165
2.154
2.141
2.055
2.039
2.022
1.989
1.975
1.903
1.888
1.880
1.870
1.855
1.721
1.609
1.266
1.248
1.230
1.044
1.028
0.957
0.941
EtO2C
N
Ph
BRUKER
NAME 5H
EXPNO 29
PROCNO 1
Date_ 20150824
Time 11.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 161.3
DW 60.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
2.01
4.09
2.11
1.09
0.99
1.00
1.08
1.06
1.05
2.15
2.33
3.24
1.16
3.42
3.17
3.19



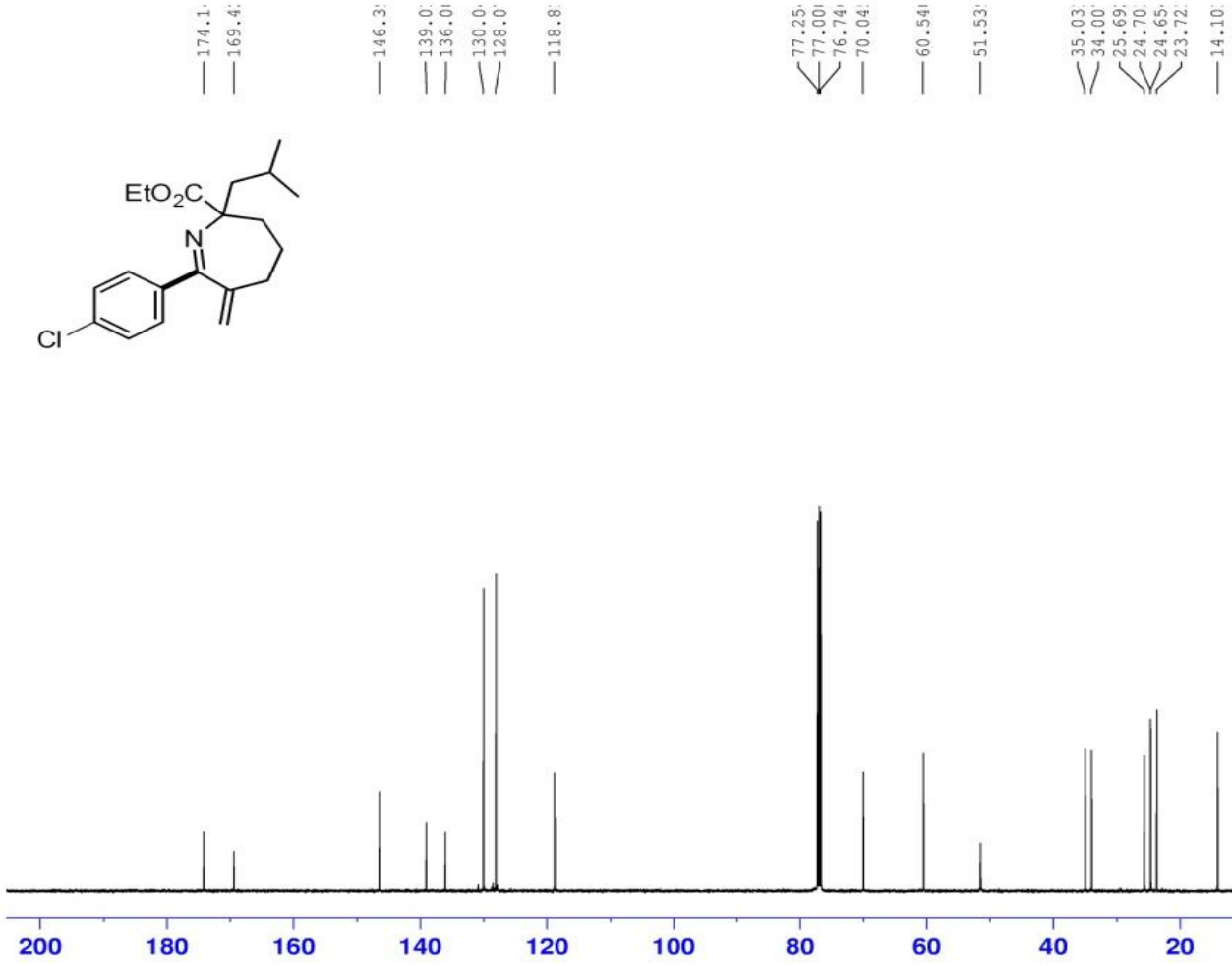
NAME	5C	
EXPNO	20	
PROCNO	1	
Date_	20150825	
Time	1.32	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	1024	
DS	4	
SWH	29761.904	Hz
FIDRES	0.454131	Hz
AQ	1.1010548	sec
RG	203	
DW	16.800	usec
DE	6.50	usec
TE	303.4	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	13.84	usec
PL1	2.50	dB
PL1W	46.89624786	W
SFO1	125.7703643	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	2.50	dB
PL12	17.40	dB
PL13	17.40	dB
PL2W	13.02359581	W
PL12W	0.42143536	W
PL13W	0.42143536	W
SFO2	500.1320005	MHz
SI	32768	
SF	125.7577915	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

3d



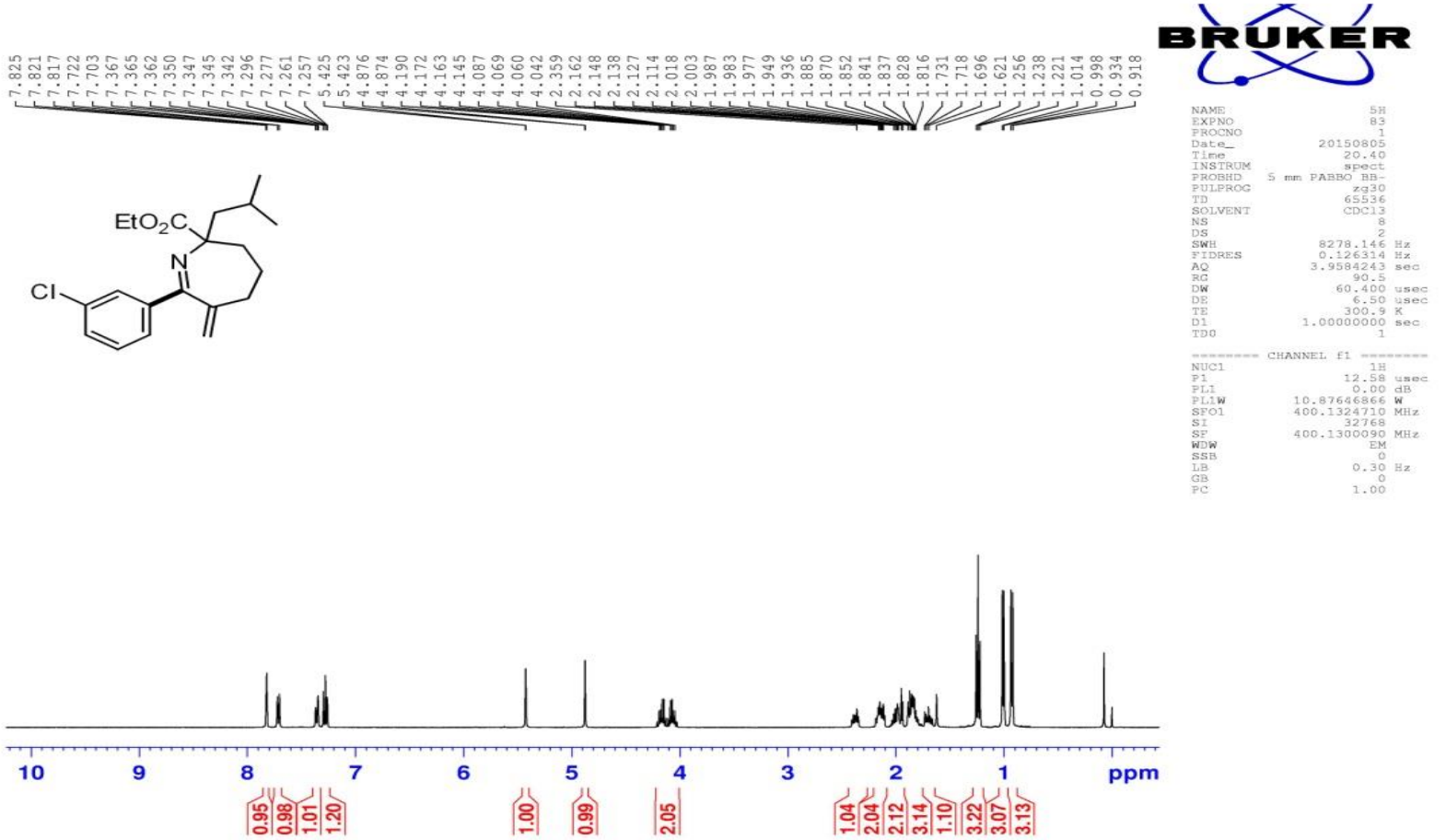
BRUKER

NAME	5H
EXPNO	311
PROCNO	1
Date_	20150803
Time	15.43
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	71.8
DW	60.400 usec
DE	6.50 usec
TE	300.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300093 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



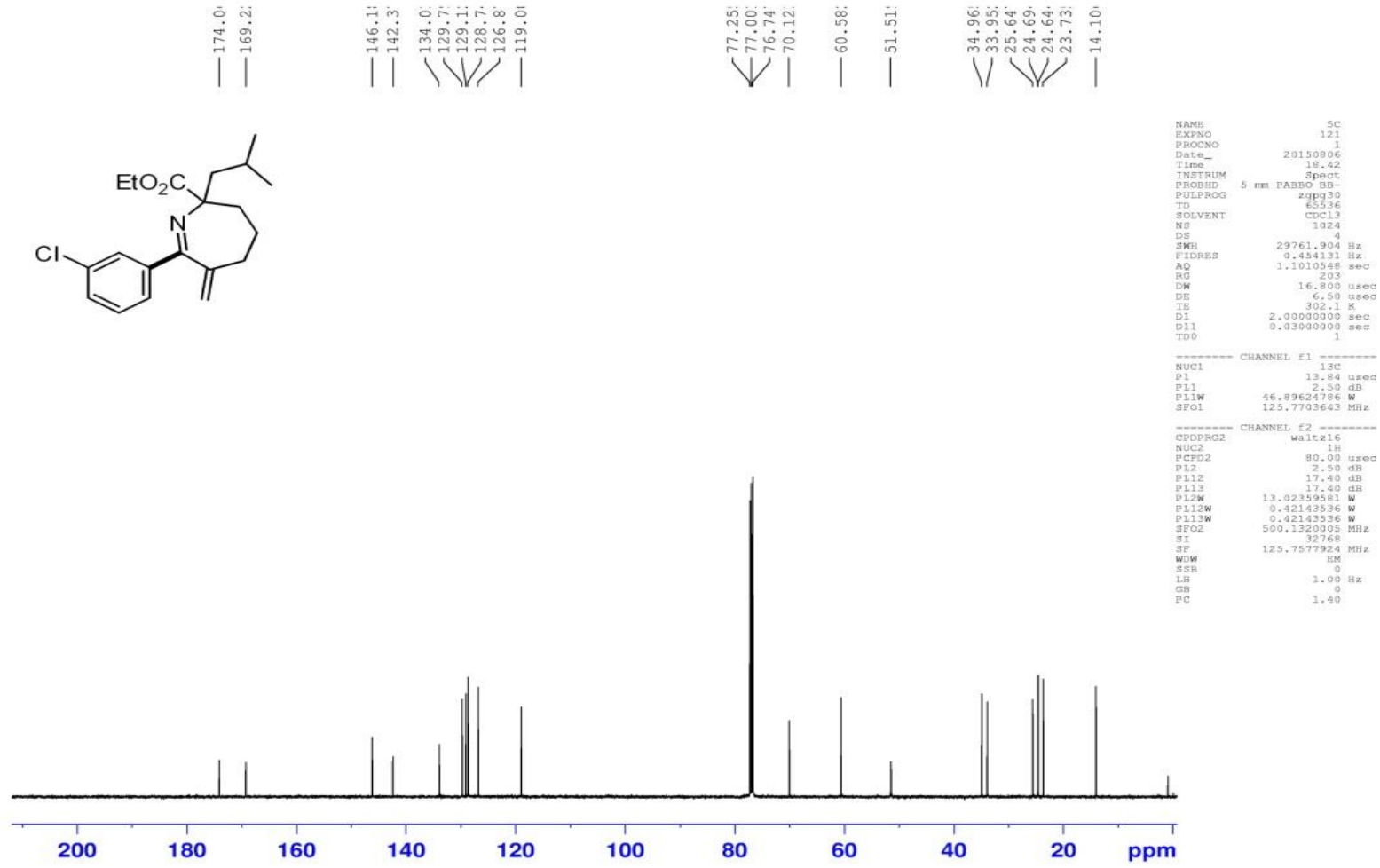
NAME	SC
EXPNO	7
PROCNO	1
Date_	20150805
Time	6.13
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1600
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	302.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577922 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

3e

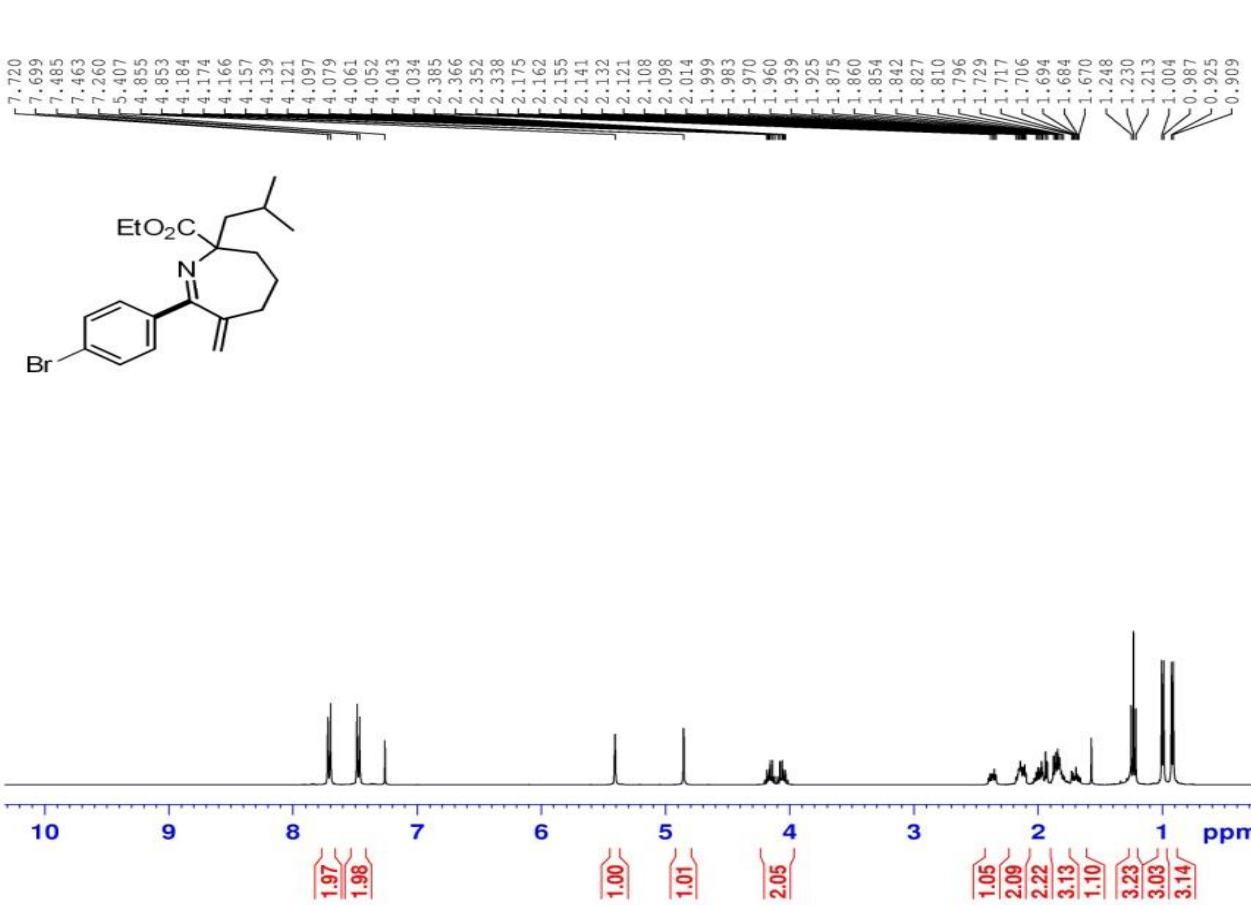


BRUKER

NAME	5H
EXPNO	83
PROCNO	1
Date_	20150805
Time	20.40
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	90.5
DW	60.400 usec
DE	6.50 usec
TE	300.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300090 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

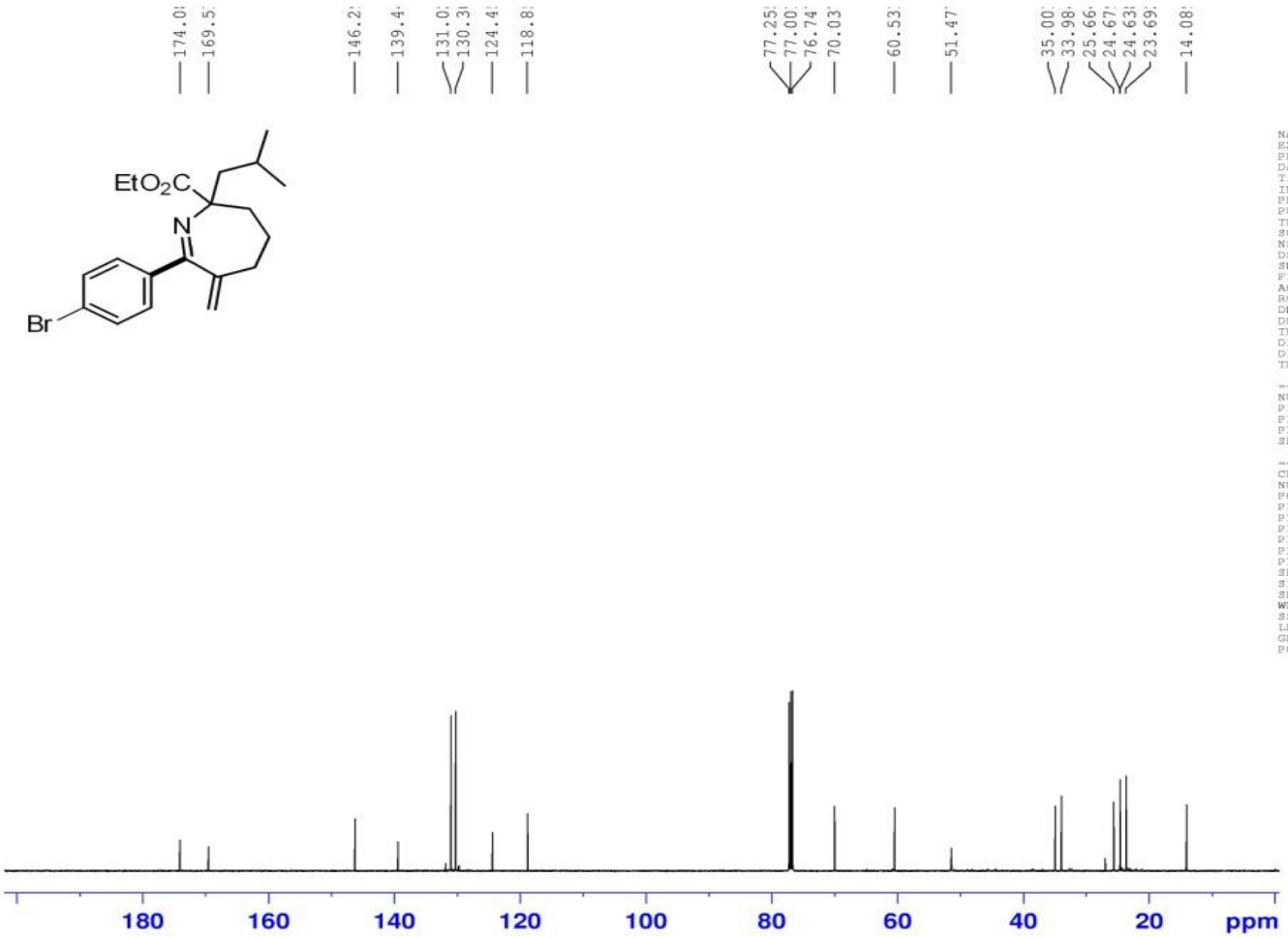

174.0
169.2
146.1
142.3
134.0
129.7
129.1
128.7
126.8
119.0
77.25
77.00
76.74
70.12
60.58
51.51
34.96
33.95
25.64
24.69
24.64
23.73
14.10
EtO2C
N
Cl
NAME 5C
EXPNO 121
PROCNO 1
Date_ 20150806
Time 18.42
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 302.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577924 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
ppm

3f

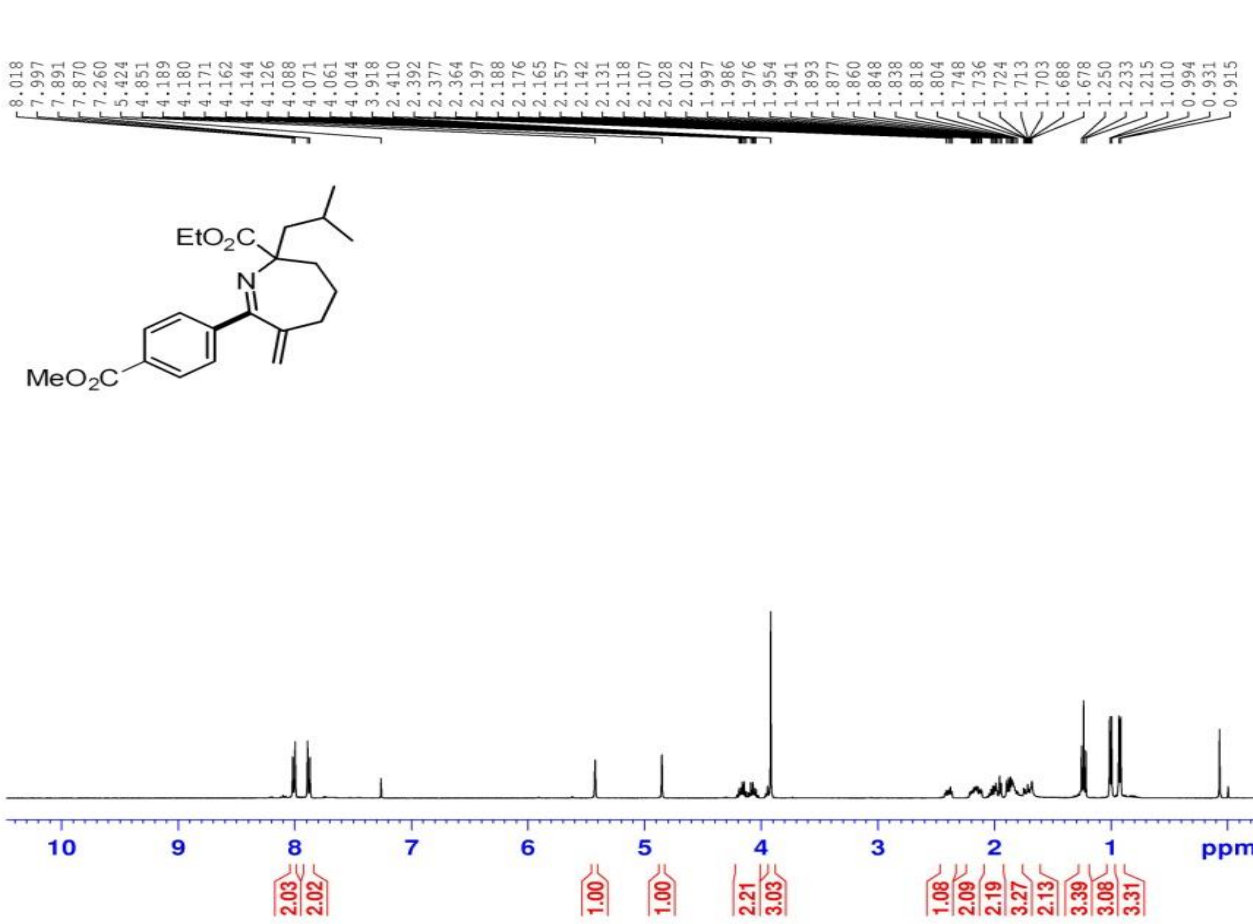


BRUKER

NAME	5H
EXPNO	3000
PROCNO	1
Date_	20150808
Time	9.30
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	128
DW	60.400 usec
DE	6.50 usec
TE	301.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300094 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

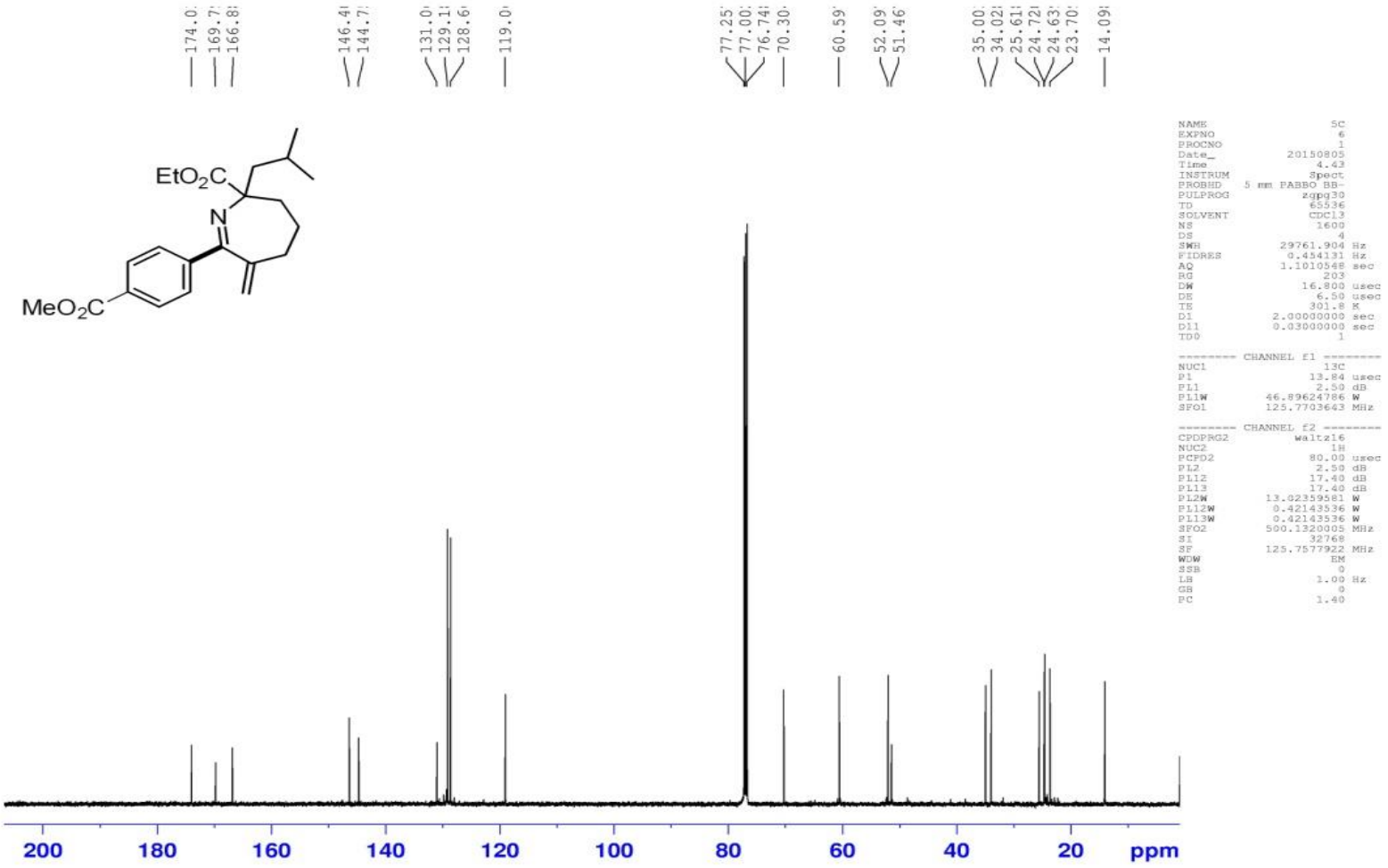

174.0
169.5
146.2
139.4
131.0
130.3
124.4
118.8
77.25
77.00
76.74
70.03
60.53
51.47
35.00
33.98
25.66
24.67
24.63
23.69
14.08
EtO2C
N
Br
NAME SC
EXPNO 14
PROCNO 1
Date_ 20151201
Time 9.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577959 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180
160
140
120
100
80
60
40
20
ppm

3g



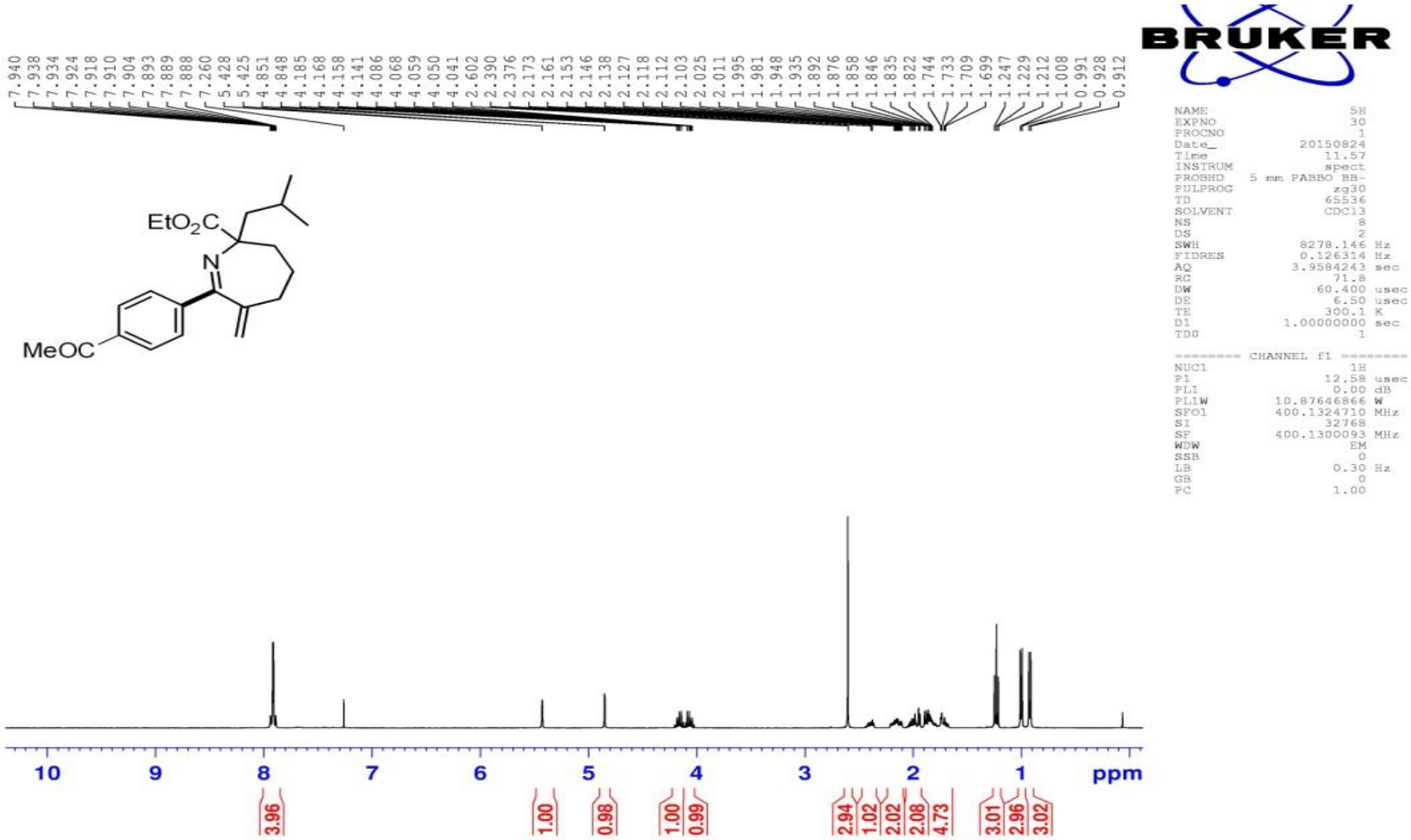
BRUKER

NAME	5H
EXPNO	300
PROCNO	1
Date_	20150803
Time	15.37
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	90.5
DW	60.400 usec
DE	6.50 usec
TE	300.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300093 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00




```
NAME                  SC
EXPNO                  6
PROCNO                 1
Date_           20150805
Time                4.43
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT             CDCl3
NS                   1600
DS                      4
SWH             29761.904 Hz
FIDRES          0.454131 Hz
AQ              1.1010548 sec
RG                    203
DW                16.800 usec
DE                 6.50 usec
TE                301.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 13.84 usec
PL1                 2.50 dB
PL1W           46.89624786 W
SFO1          125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2              80.00 usec
PL2                 2.50 dB
PL12               17.40 dB
PL13               17.40 dB
PL2W           13.02359581 W
PL12W           0.42143536 W
PL13W           0.42143536 W
SFO2          500.1320005 MHz
SI                   32768
SF            125.7577922 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

3h


7.940
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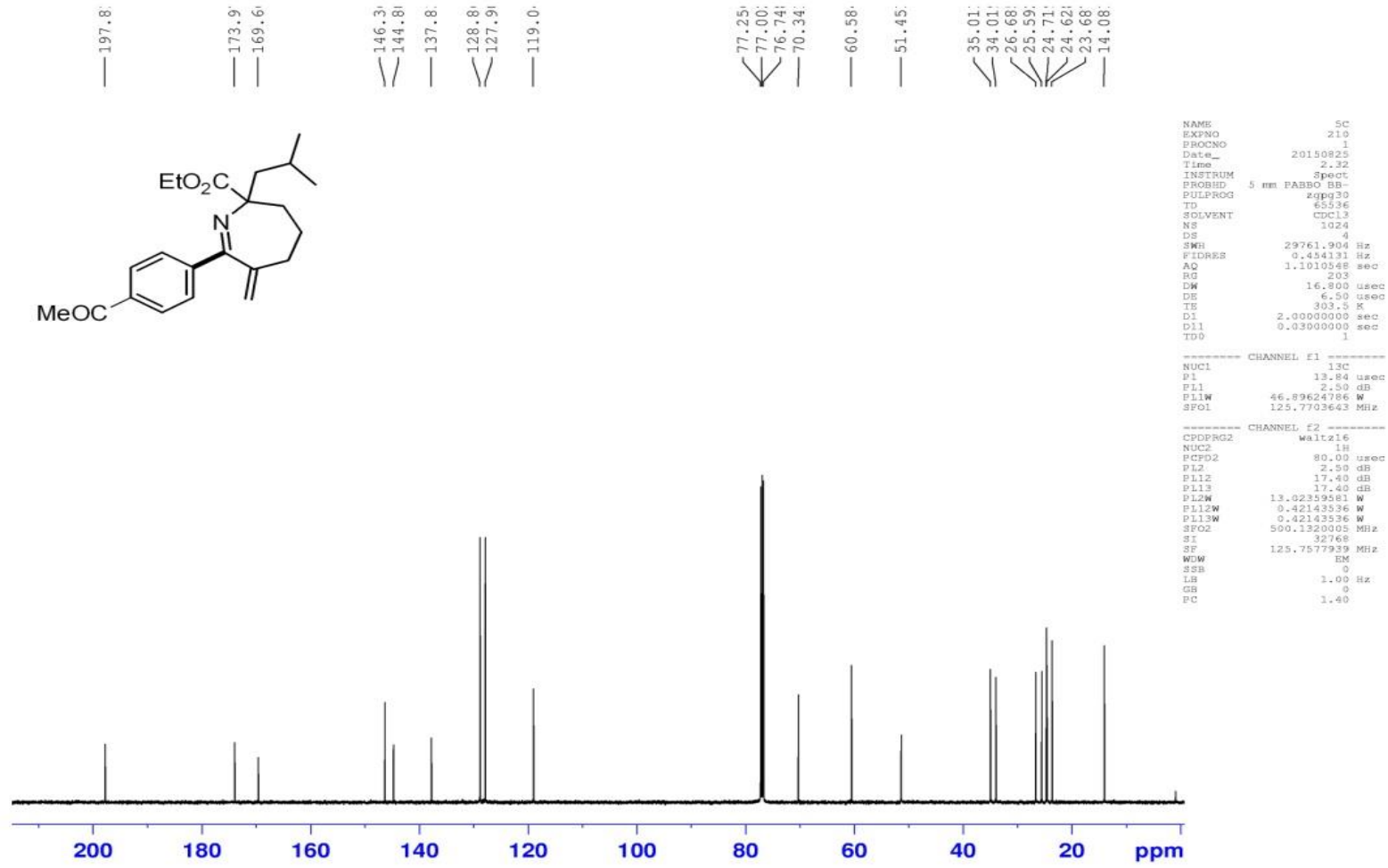
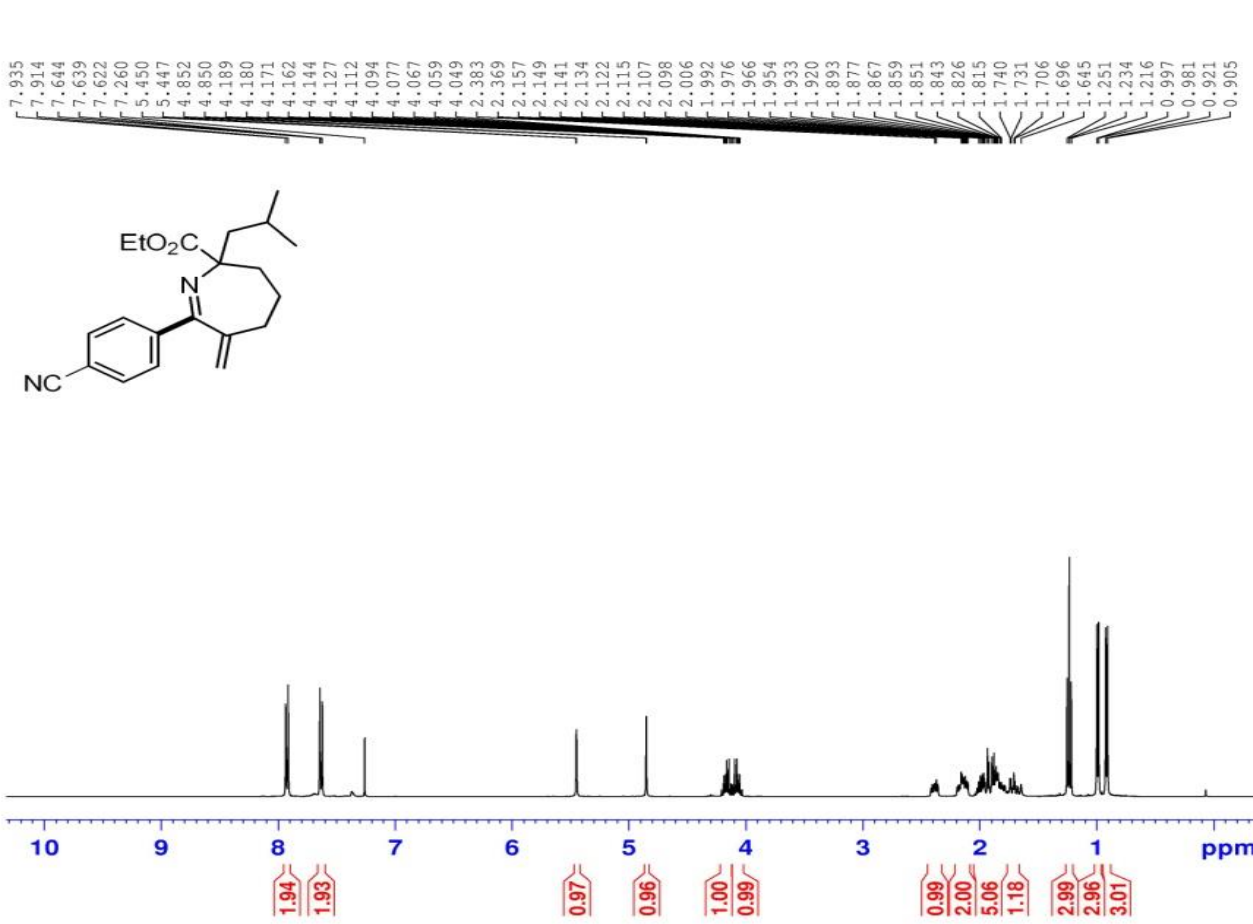
7.934
7.924
7.918
7.910
7.904
7.893
7.889
7.888
7.260
5.428
5.425
4.851
4.848
4.185
4.168
4.158
4.141
4.086
4.068
4.059
4.050
4.041
2.602
2.390
2.376
2.173
2.161
2.153
2.146
2.138
2.127
2.118
2.112
2.103
2.025
2.011
1.995
1.981
1.948
1.935
1.892
1.876
1.858
1.846
1.835
1.822
1.744
1.733
1.709
1.699
1.247
1.229
1.212
1.008
0.991
0.928
0.912
EtO2C
N
MeOC
BRUKER
NAME 5H
EXPNO 30
PROCNO 1
Date_ 20150824
Time 11.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400 usec
DE 6.50 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
3.96
1.00
0.98
1.00
0.99
2.94
1.02
2.02
2.08
4.73
3.01
2.96
3.02



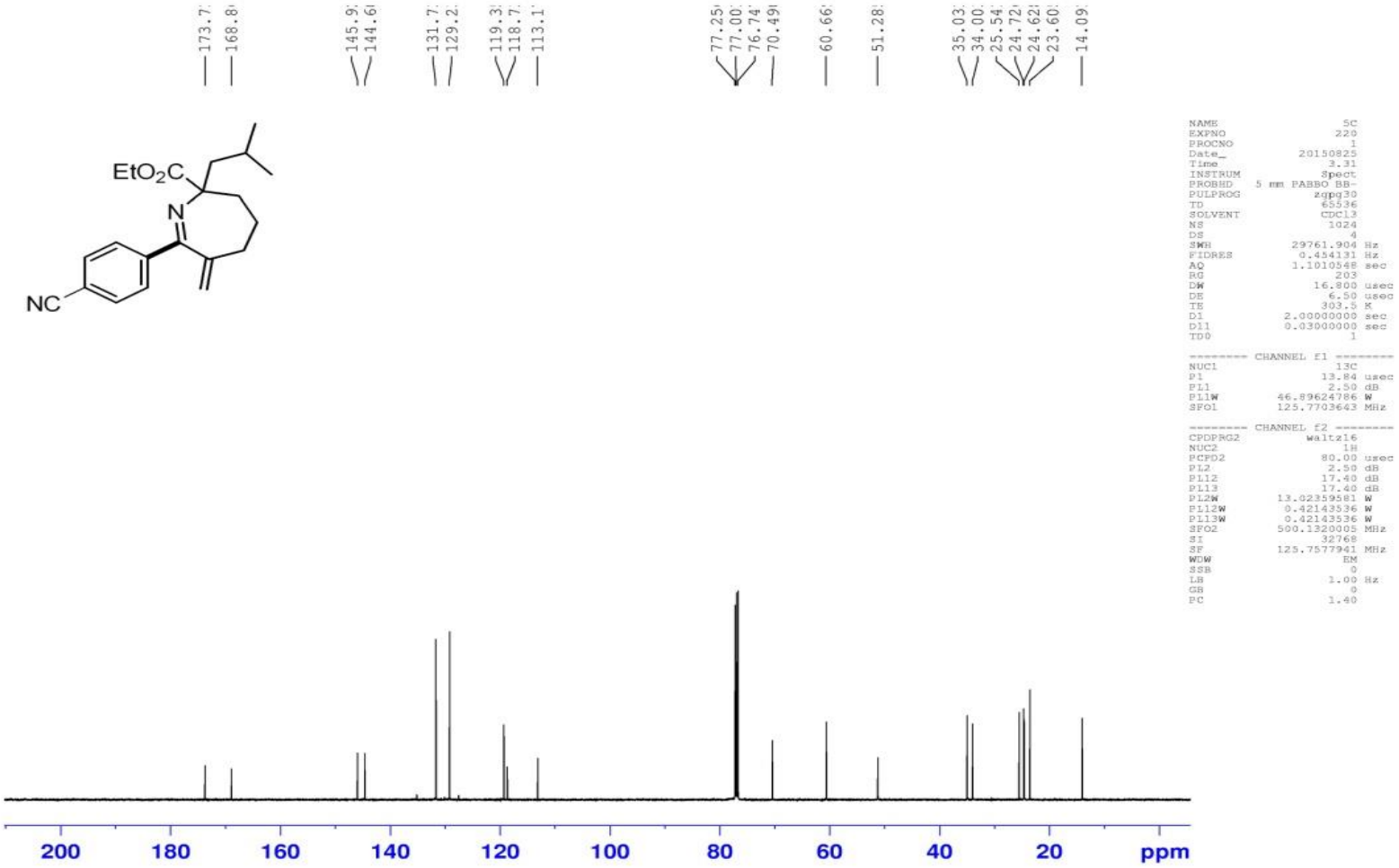
NAME	5C
EXPNO	210
PROCNO	1
Date_	20150825
Time	2.32
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	303.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577939 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

3i



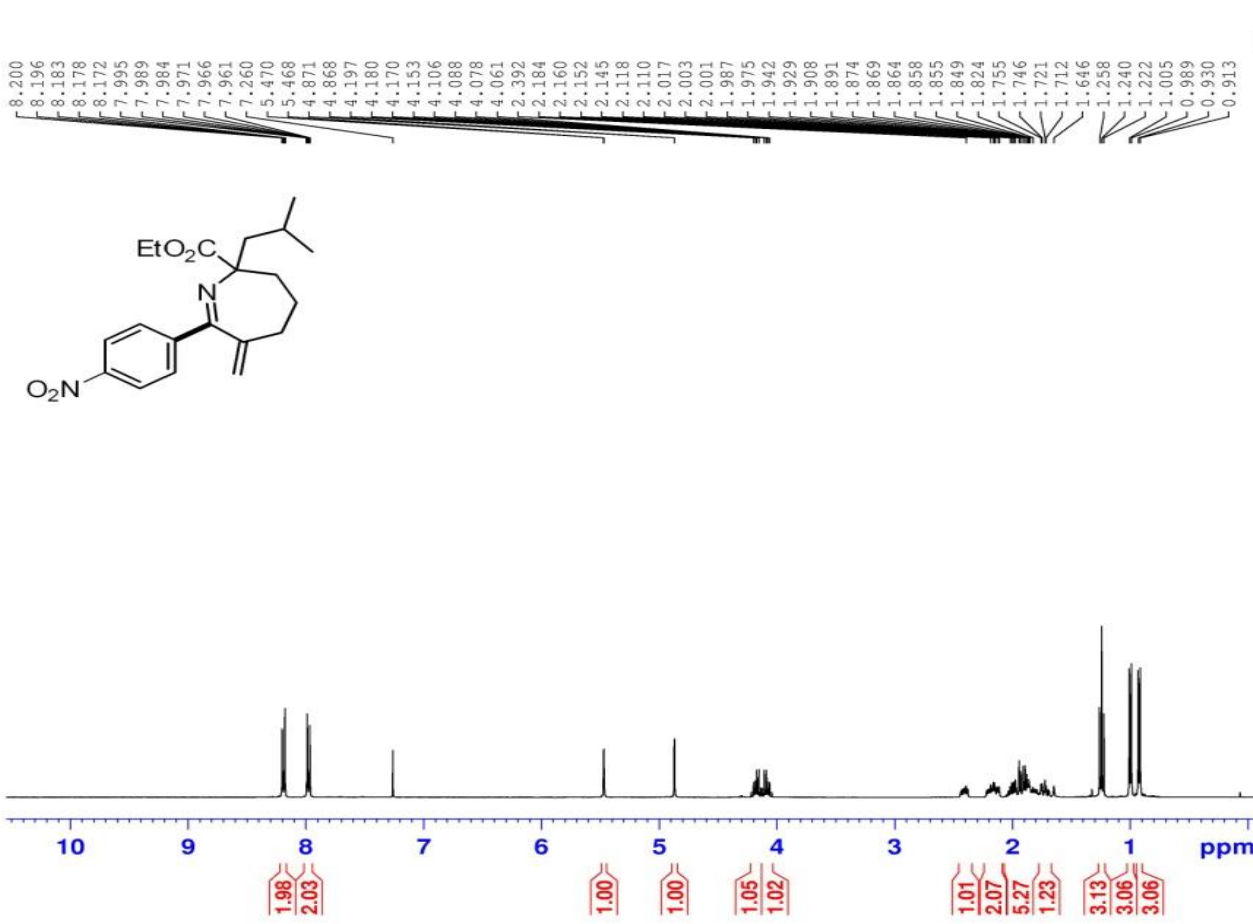
BRUKER

NAME	5H
EXPNO	31
PROCNO	1
Date_	20150824
Time	12.02
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	71.8
DW	60.400 usec
DE	6.50 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300093 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



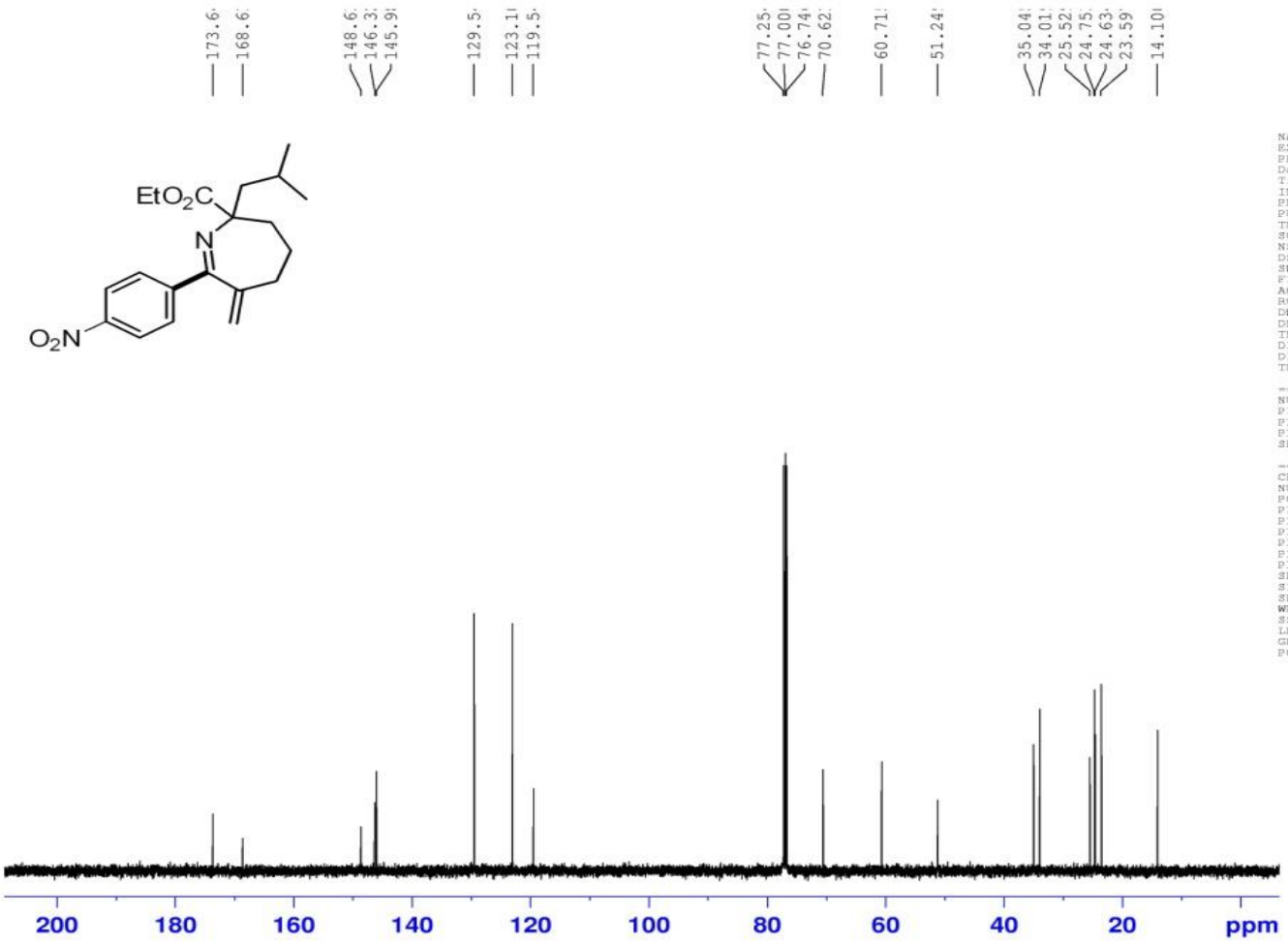
NAME	5C
EXPNO	229
PROCNO	1
Date_	20150825
Time	3.31
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	303.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577941 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

3j



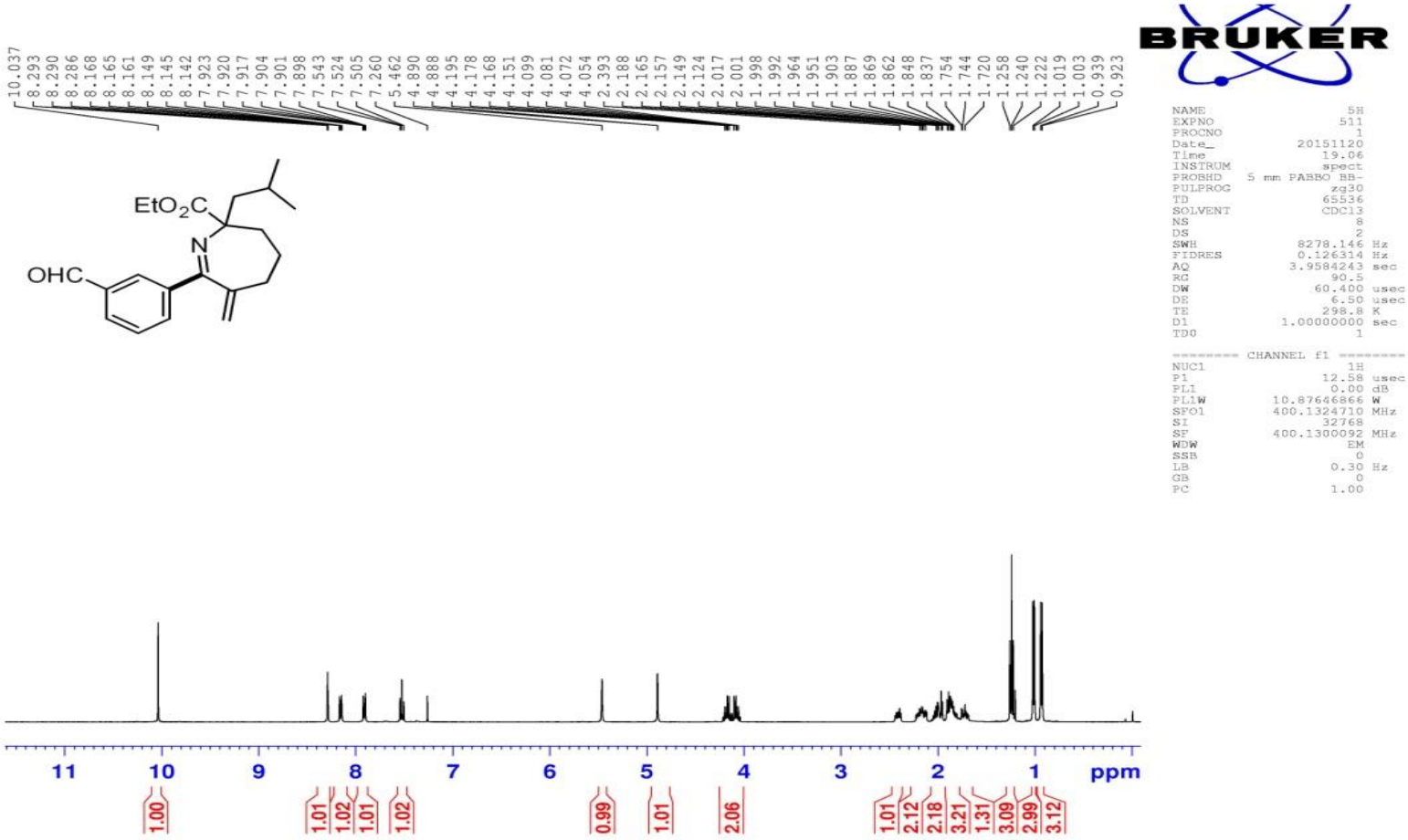
BRUKER

NAME	5H
EXPNO	321000
PROCNO	1
Date_	20150824
Time	12.08
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	71.8
DW	60.400 usec
DE	6.50 usec
TE	300.1 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300092 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



NAME	SC
EXPNO	23
PROCNO	1
Date_	20150825
Time	15.17
INSTRUM	3peot
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	89
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	302.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577941 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

3k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7.917
7.904
7.901
7.898
7.543
7.524
7.505
7.260
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4.072
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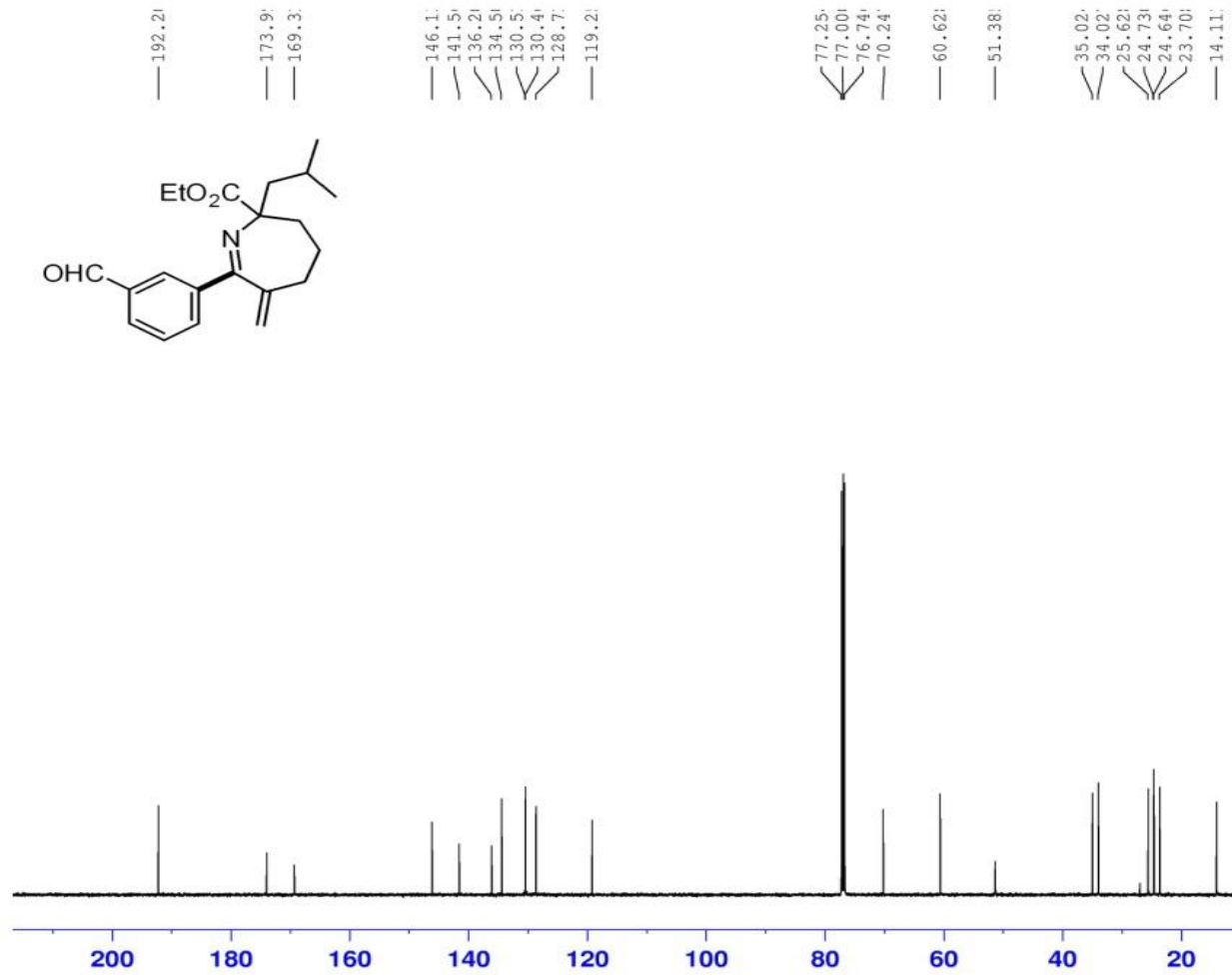
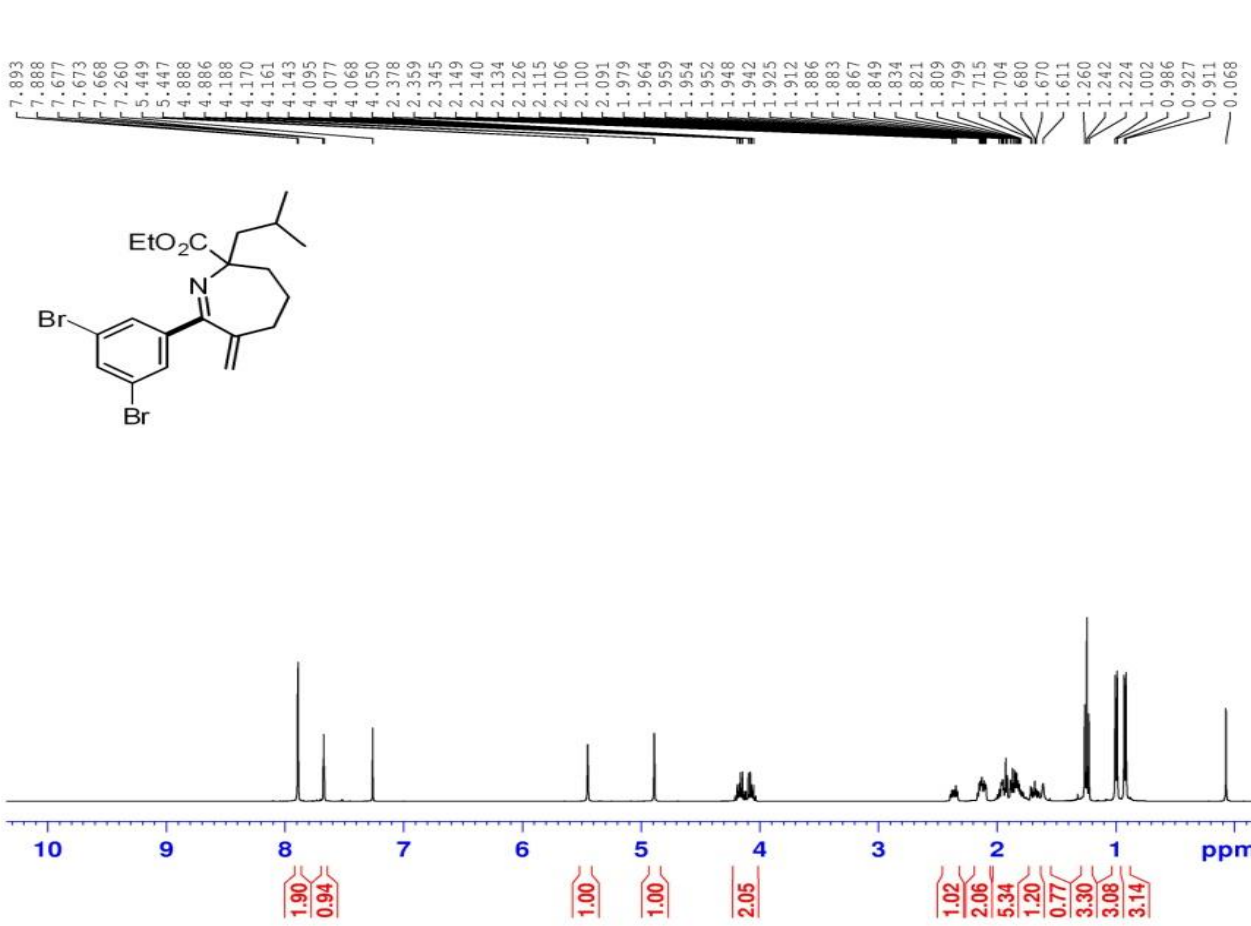
2.393
2.188
2.165
2.157
2.149
2.124
2.017
2.001
1.998
1.992
1.964
1.951
1.903
1.887
1.869
1.862
1.848
1.837
1.754
1.744
1.720
1.258
1.240
1.222
1.019
1.003
0.939
0.923
EtO2C
N
OHC
BRUKER
NAME 5H
EXPNO 511
PROCNO 1
Date_ 20151120
Time 19.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 6.50 usec
TE 298.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.01
1.02
1.01
1.02
0.99
1.01
2.06
1.01
2.12
2.18
3.21
1.31
3.09
2.99
3.12



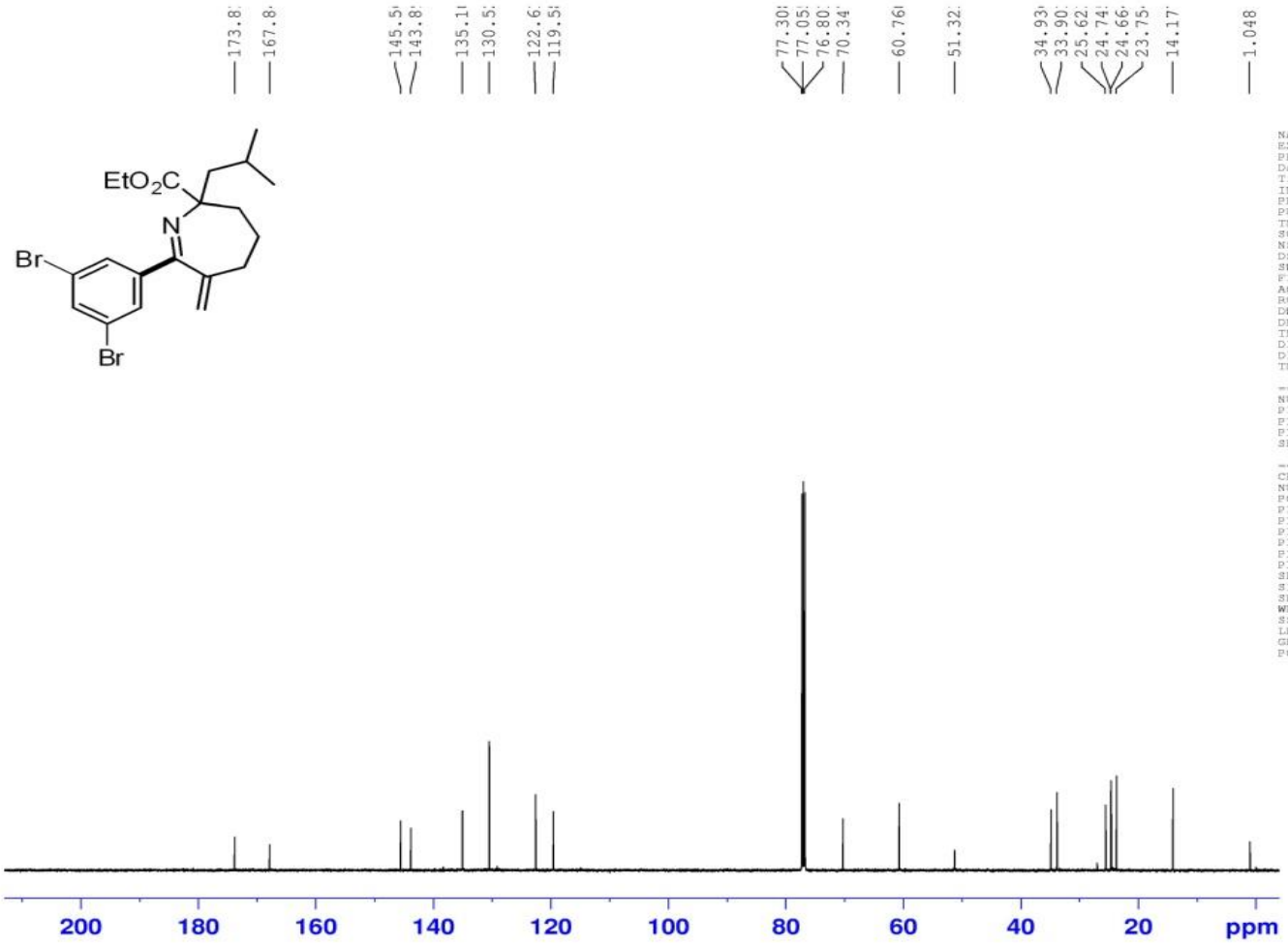
NAME	5C
EXPNO	29
PROCNO	1
Date_	20151121
Time	10.24
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	299.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577940 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

3l



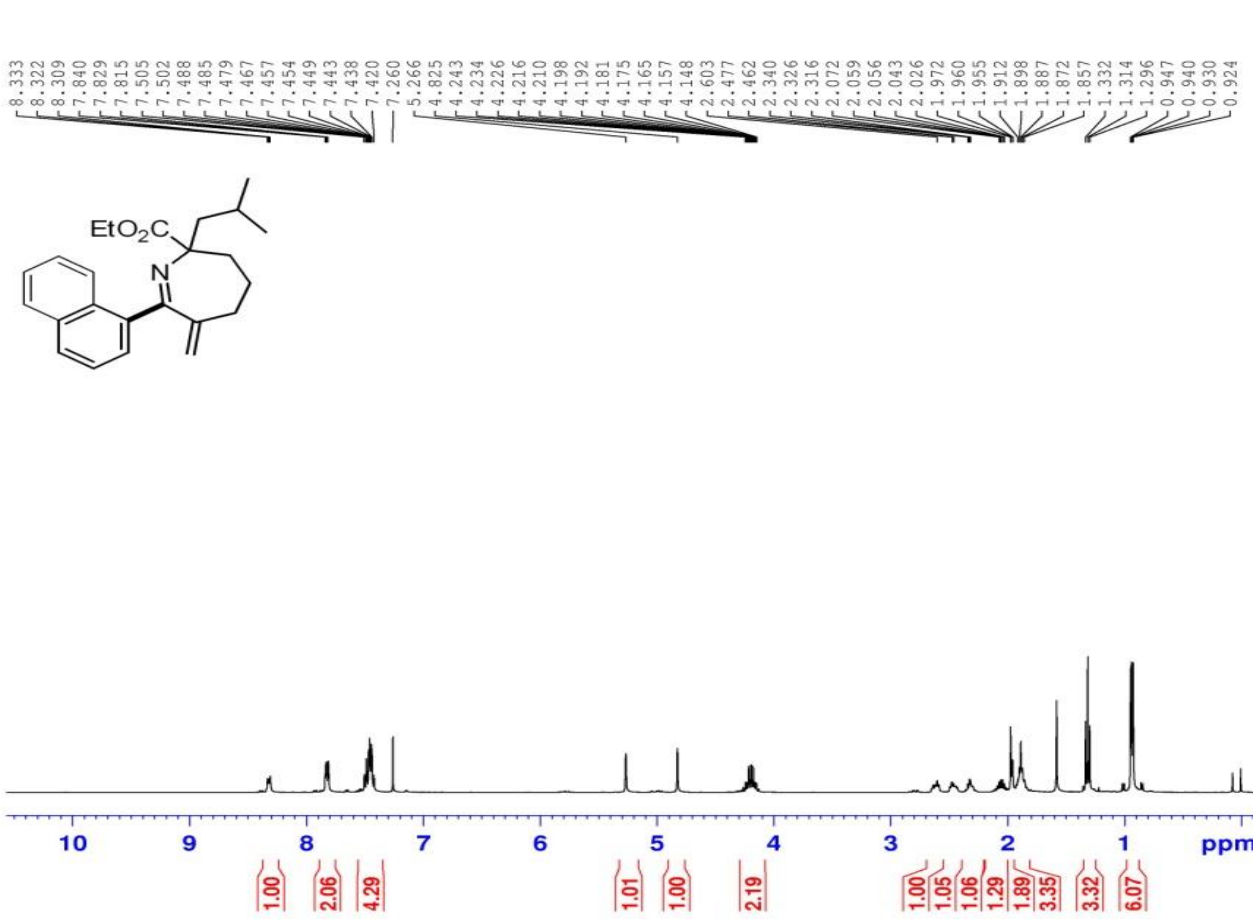
BRUKER

NAME	5H
EXPNO	21
PROCNO	1
Date_	20150828
Time	14.51
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	90.5
DW	60.400 usec
DE	6.50 usec
TE	299.4 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300093 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



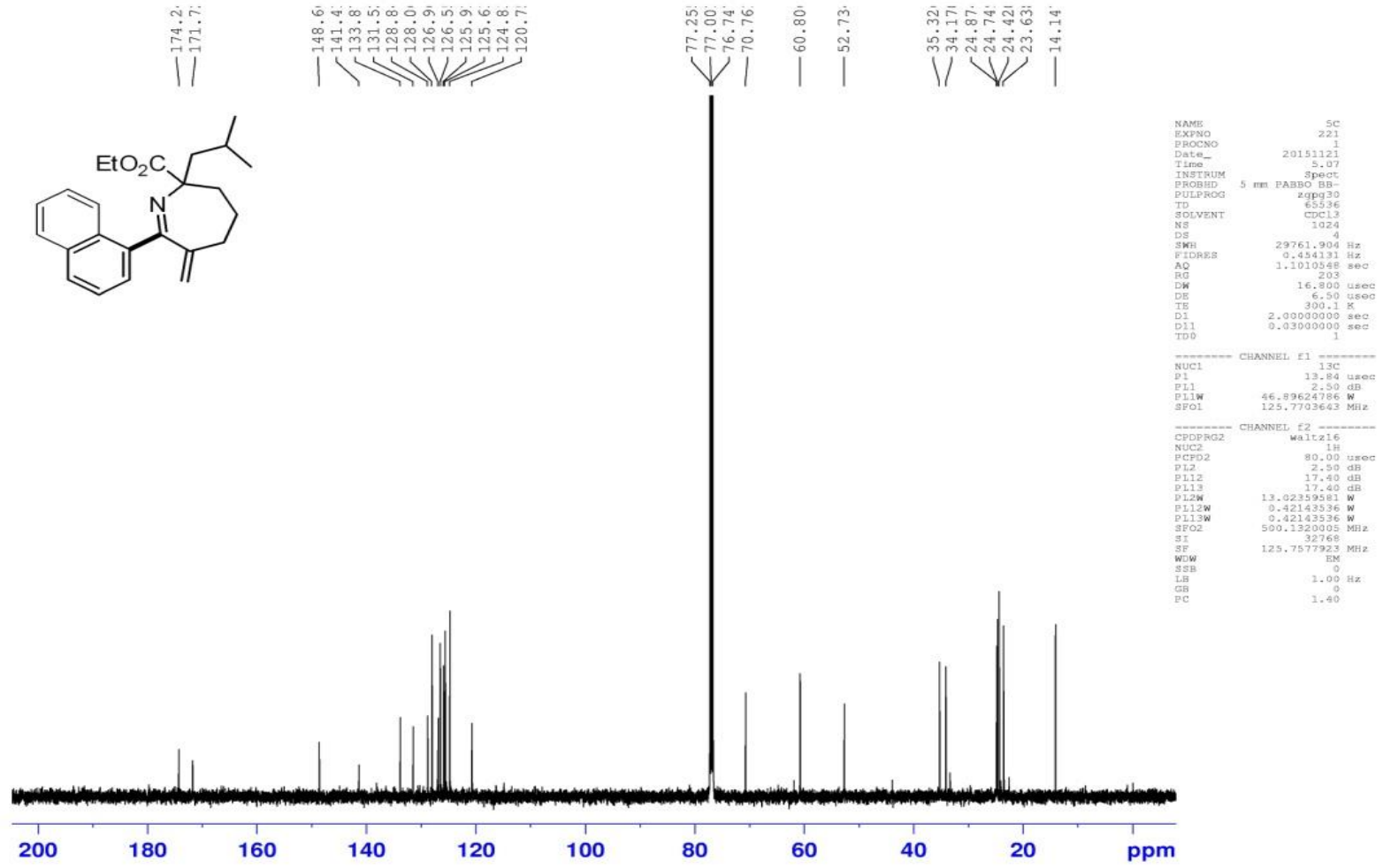
NAME	SC
EXPNO	100
PROCNO	1
Date_	20151226
Time	17.08
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	294.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

3m



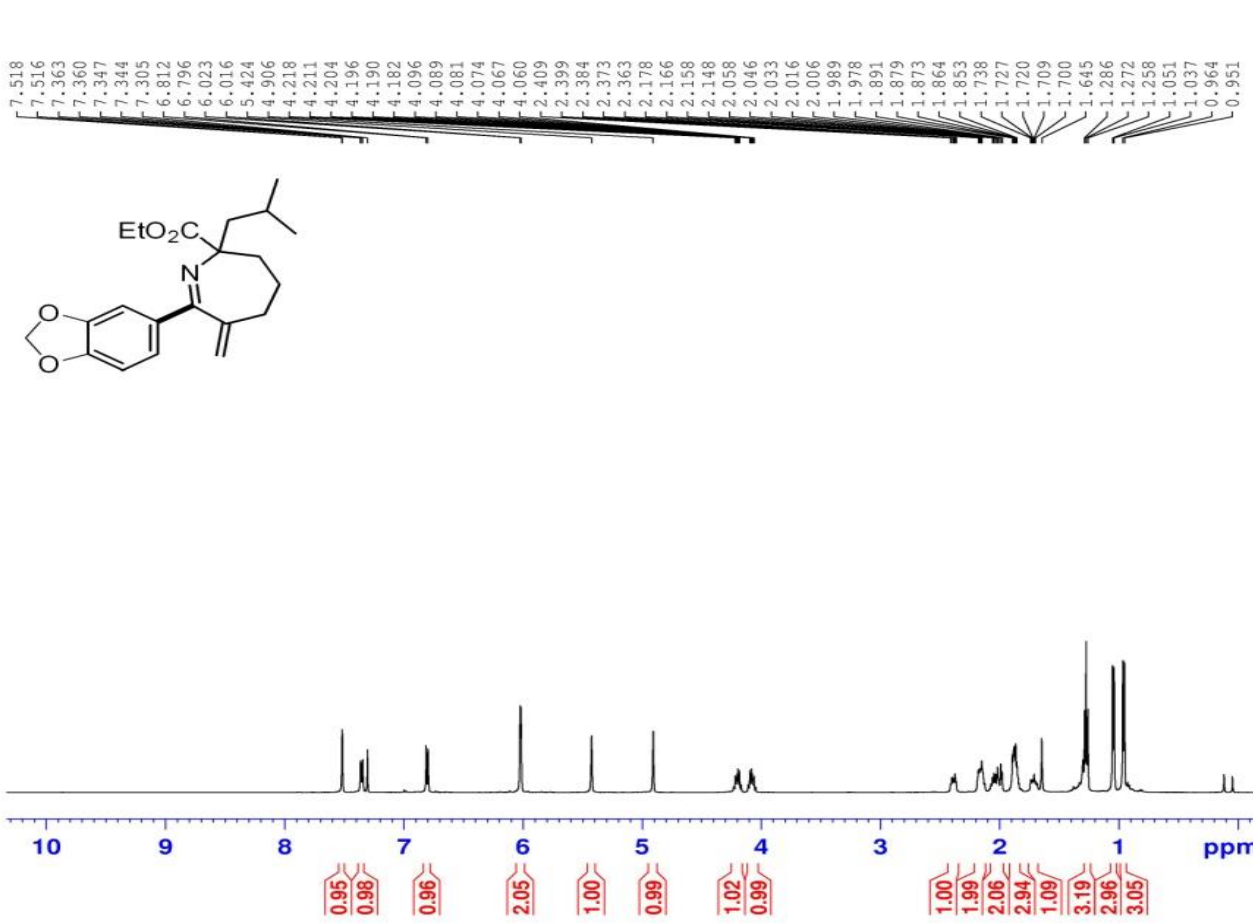
BRUKER

NAME	5H
EXPNO	32
PROCNO	1
Date_	20151120
Time	14.58
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	228.1
DW	60.400 usec
DE	6.50 usec
TE	298.6 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300094 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



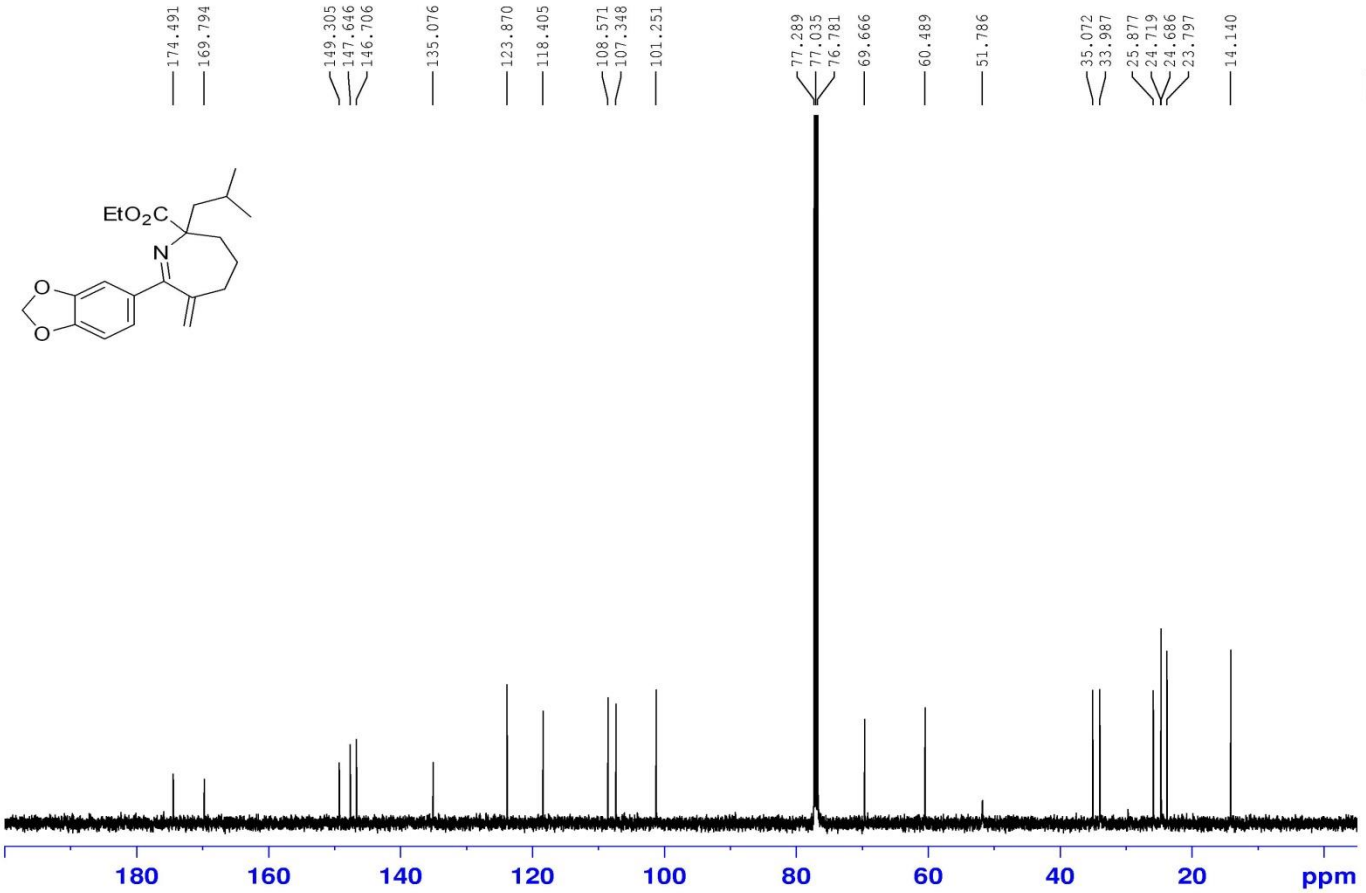
NAME	5C
EXPNO	221
PROCNO	1
Date_	20151121
Time	5.07
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	300.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577923 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

3n



BRUKER

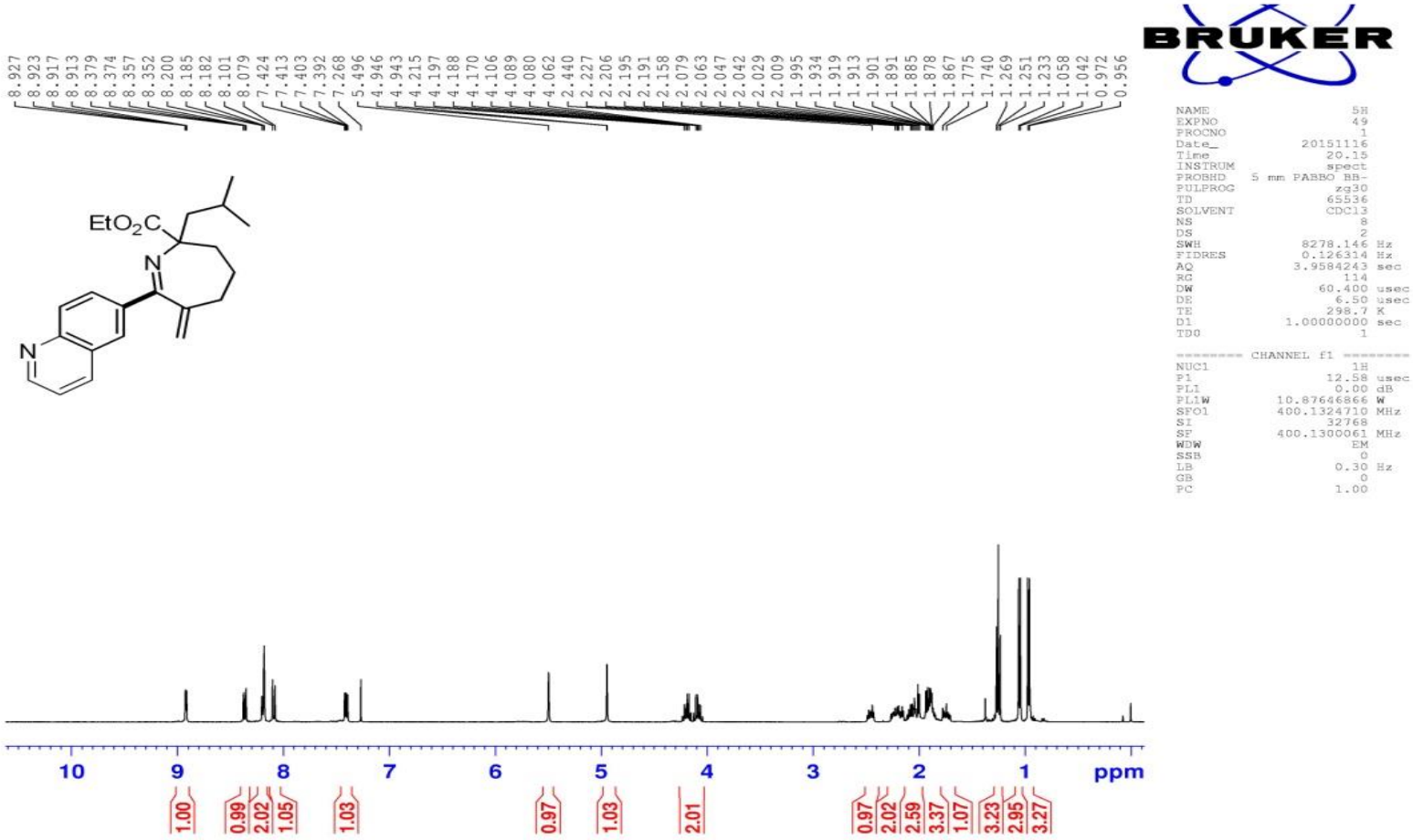
NAME	5H	
EXPNO	500	
PROCNO	1	
Date_	20160303	
Time	17.33	
INSTRUM	Spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	10330.578	Hz
FIDRES	0.157632	Hz
AQ	3.1719923	sec
RG	114	
DW	48.400	usec
DE	6.50	usec
TE	298.1	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	14.00	usec
PL1	2.50	dB
PL1W	13.02359581	W
SFO1	500.1330885	MHz
SI	32768	
SF	500.1300000	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	



BRUKER

NAME	5C
EXPNO	600
PROCNO	1
Date_	20160303
Time	18.02
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	427
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	298.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

3o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8.185
8.182
8.101
8.079
7.424
7.413
7.403
7.392
7.268
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4.215
4.197
4.188
4.170
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4.080
4.062
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BRUKER
NAME 5H
EXPNO 49
PROCNO 1
Date_ 20151116
Time 20.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 114
DW 60.400 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.00
0.99
2.02
1.05
1.03
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1.03
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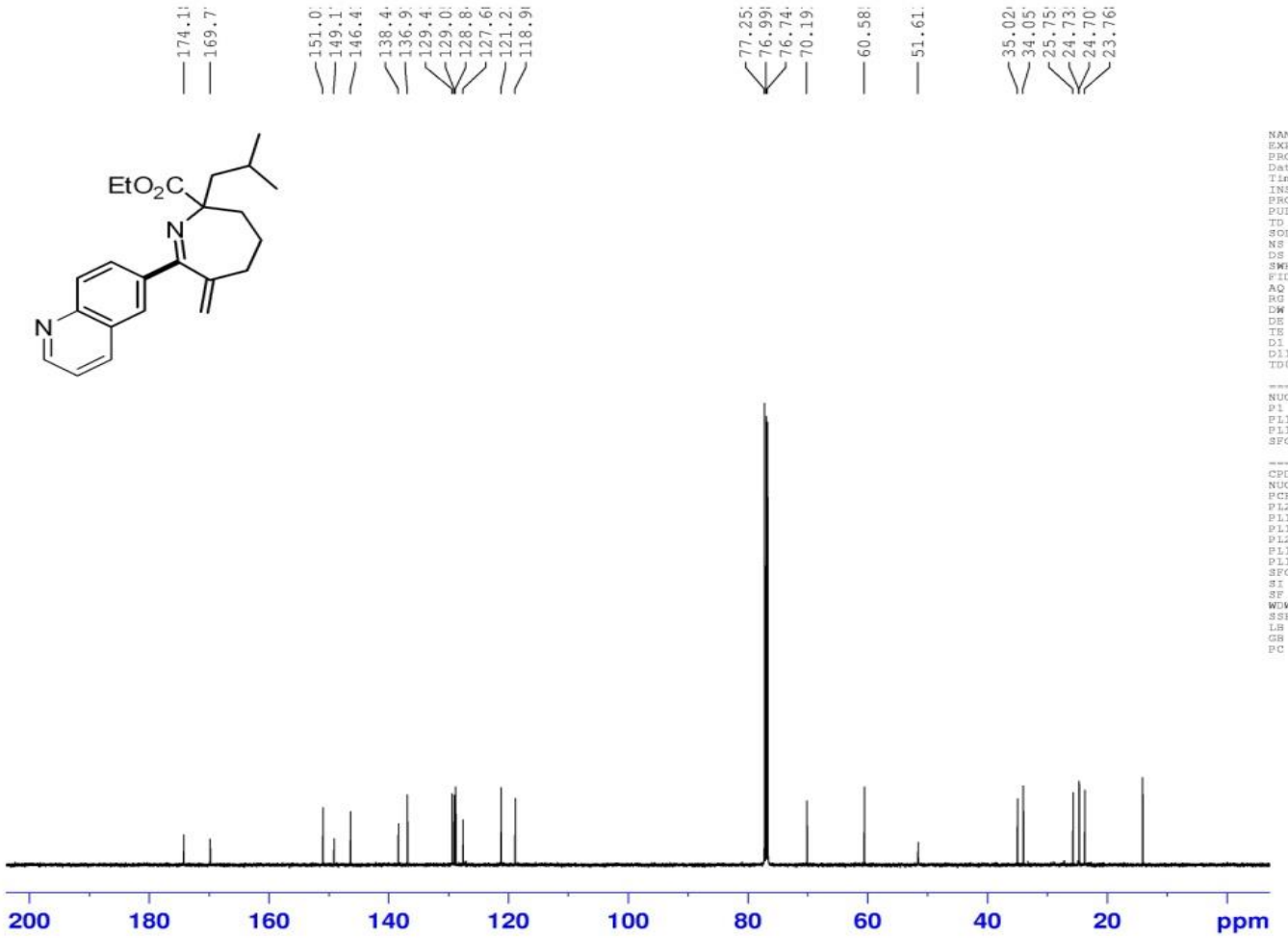
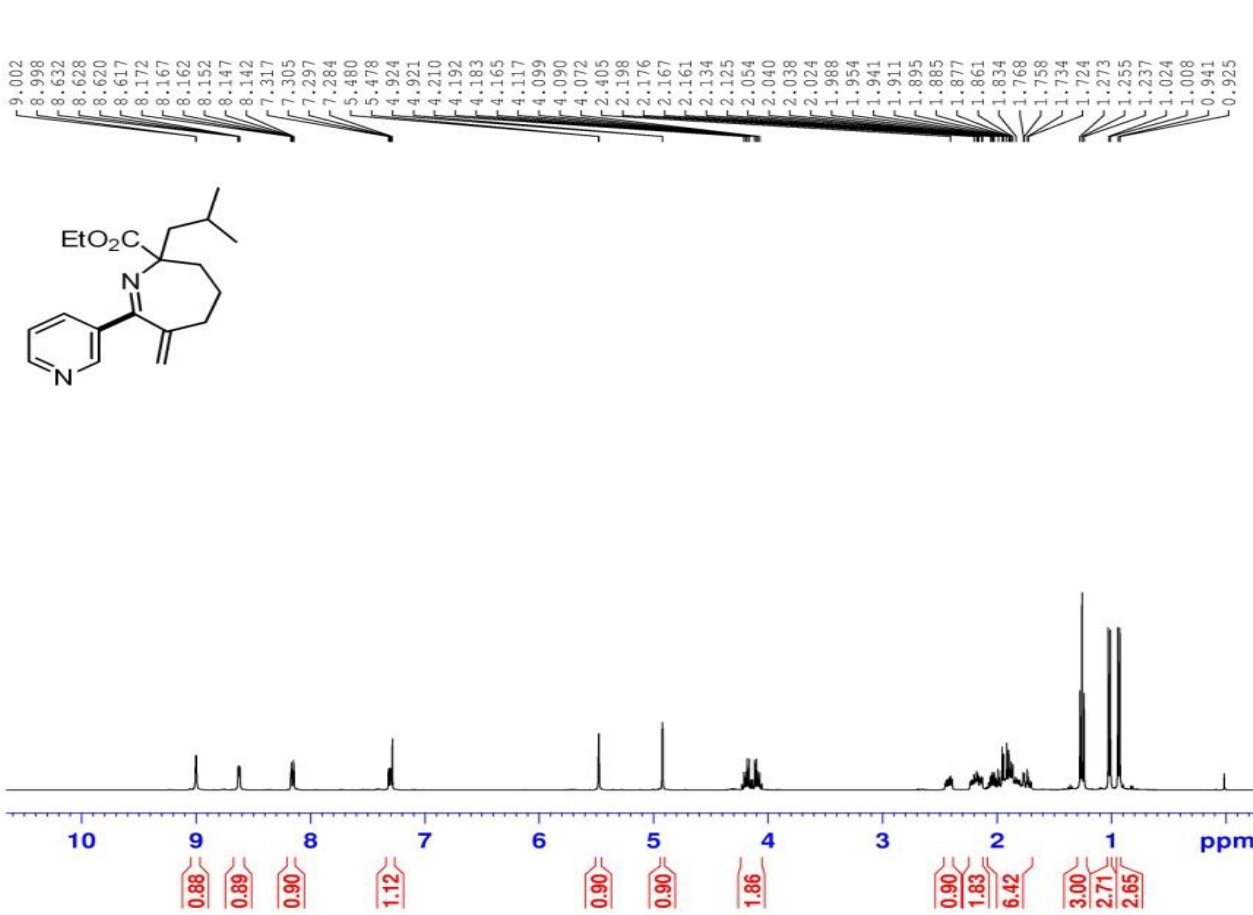
0.97
2.02
2.59
3.37
1.07
3.23
2.95
3.27



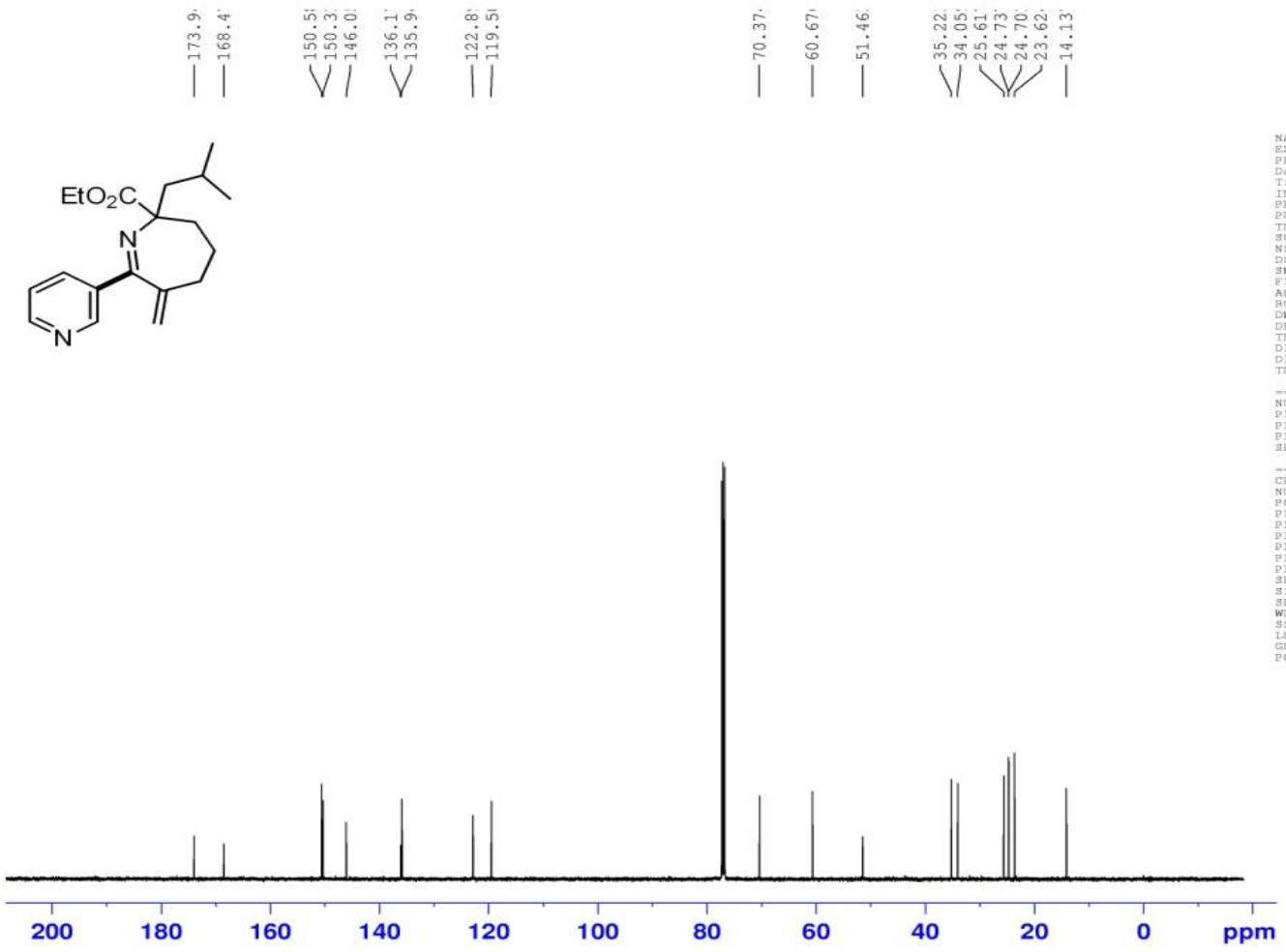
NAME	5C
EXPNO	150
PROCNO	1
Date_	20151117
Time	10.30
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	299.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577939 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

3p



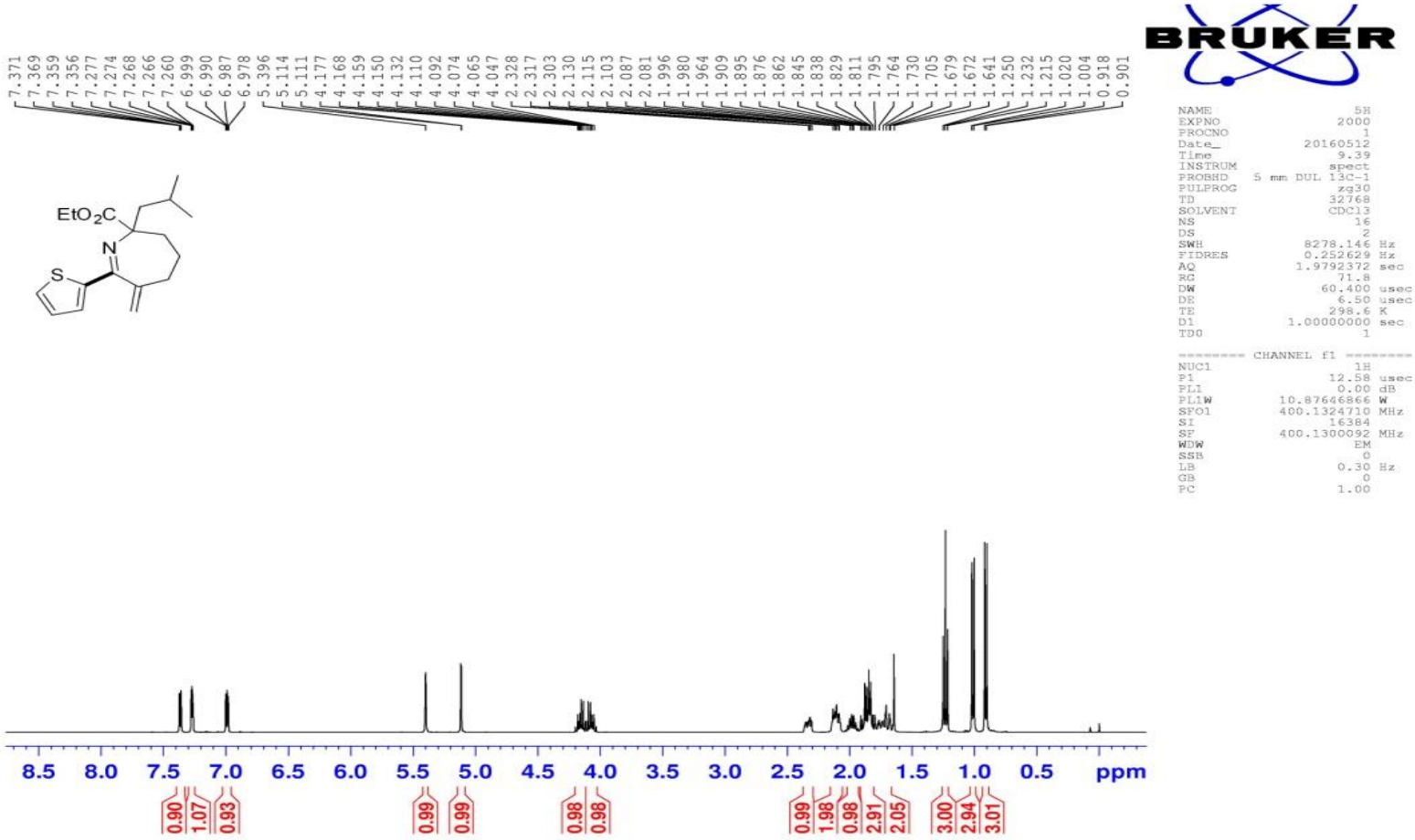
BRUKER

NAME	5H	
EXPNO	35	
PROCNO	1	
Date_	20151117	
Time	15.52	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	8	
DS	2	
SWH	8278.146	Hz
FIDRES	0.126314	Hz
AQ	3.9584243	sec
RG	90.5	
DW	60.400	usec
DE	6.50	usec
TE	298.8	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	12.58	usec
PL1	0.00	dB
PL1W	10.87646866	W
SFO1	400.1324710	MHz
SI	32768	
SF	400.1300000	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	



NAME	SC
EXPNO	90
PROCNO	1
Date_	20151118
Time	8.21
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	300.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

3q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7.356
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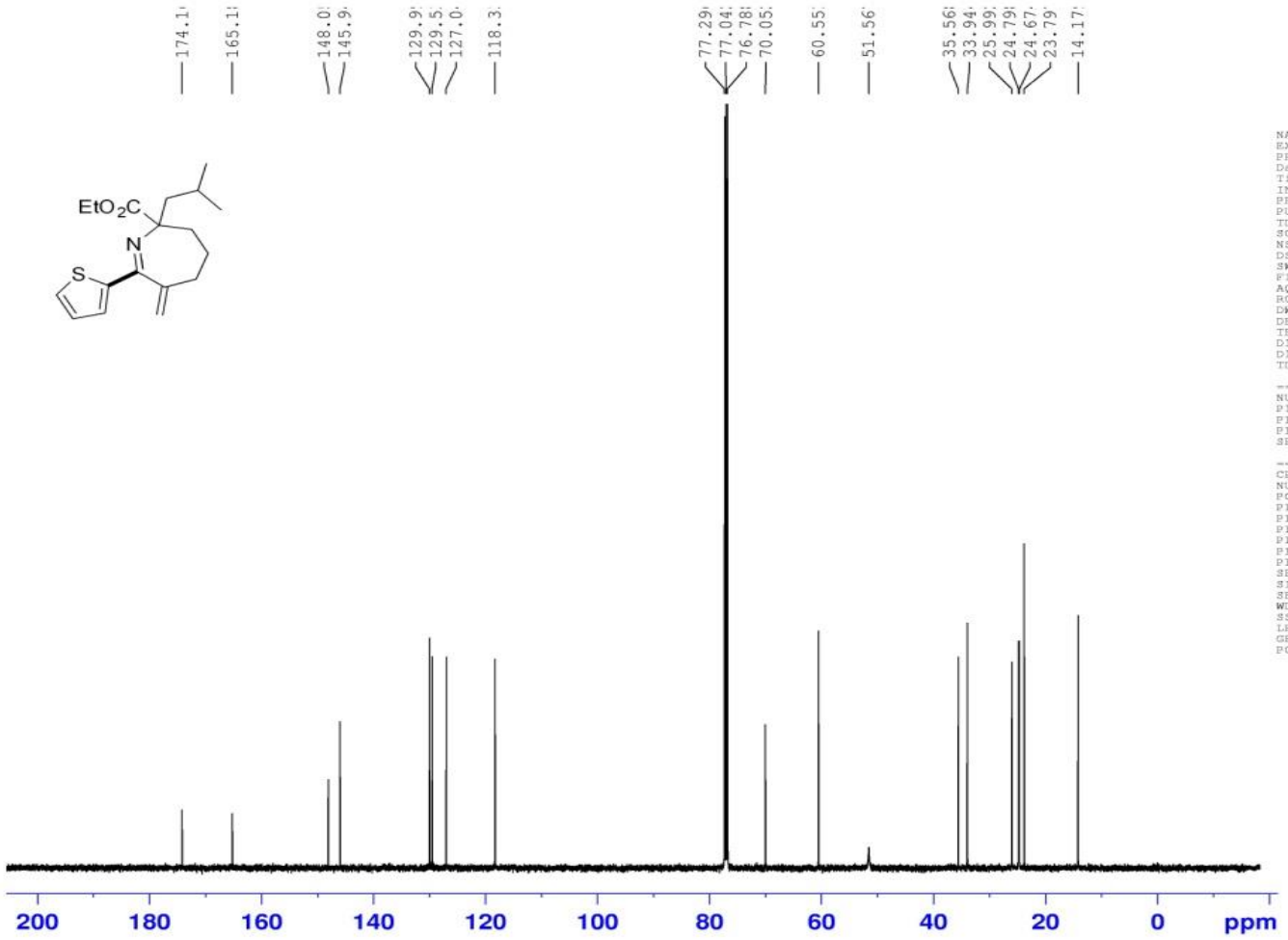
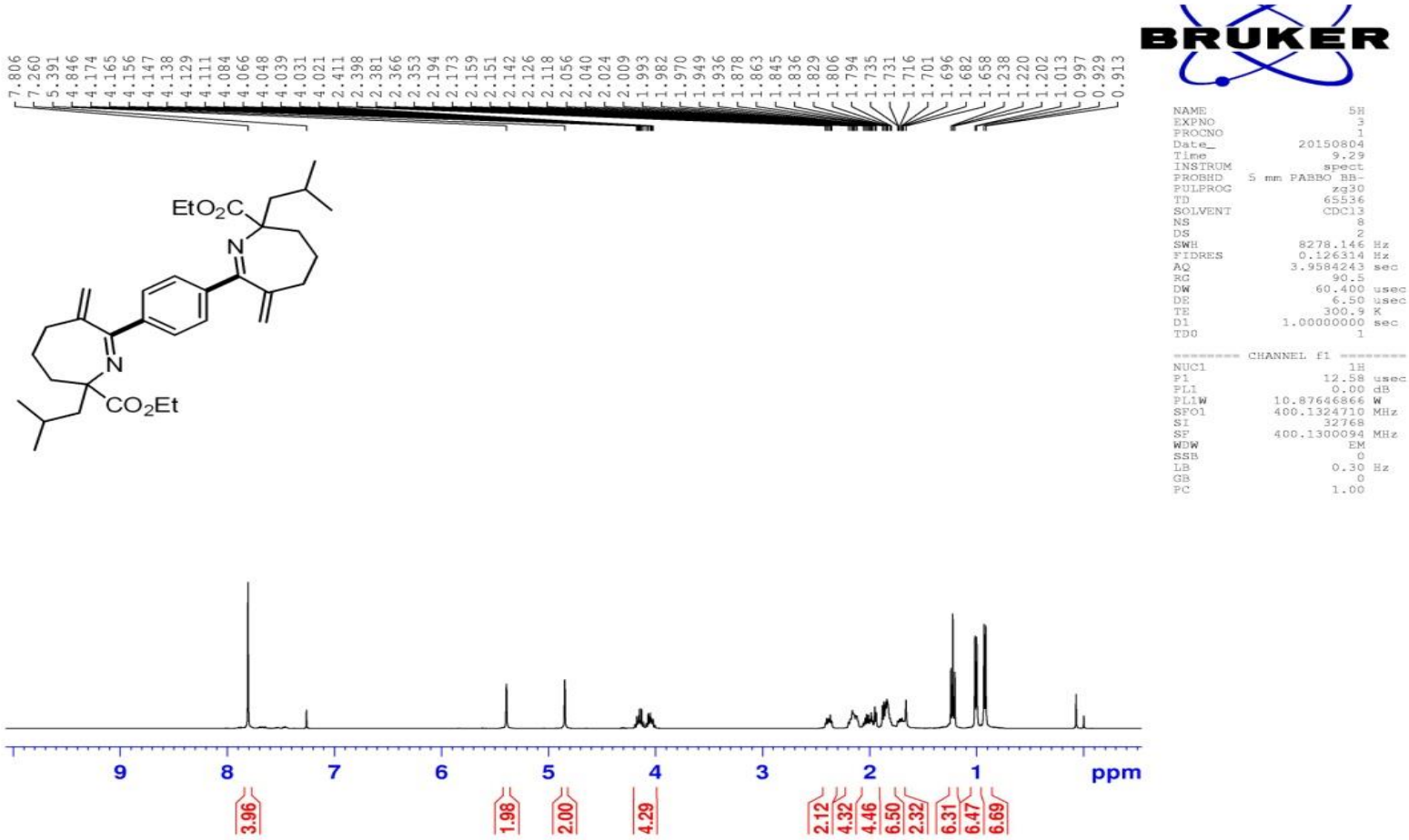
7.274
7.268
7.266
7.260
6.999
6.990
6.987
6.978
5.396
5.114
5.111
4.177
4.168
4.159
4.150
4.132
4.110
4.092
4.074
4.065
4.047
2.328
2.317
2.303
2.130
2.115
2.103
2.087
2.081
1.996
1.980
1.964
1.909
1.895
1.876
1.862
1.845
1.838
1.829
1.811
1.795
1.764
1.730
1.705
1.679
1.672
1.641
1.250
1.232
1.215
1.020
1.004
0.918
0.901
EtO2C
N
S
BRUKER
NAME 5H
EXPNO 2000
PROCNO 1
Date_ 20160512
Time 9.39
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 71.8
DW 60.400 usec
DE 6.50 usec
TE 298.6 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 16384
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.90
1.07
0.93
0.99
0.99
0.98
0.98
0.99
1.98
0.98
2.91
2.05
3.00
2.94
3.01



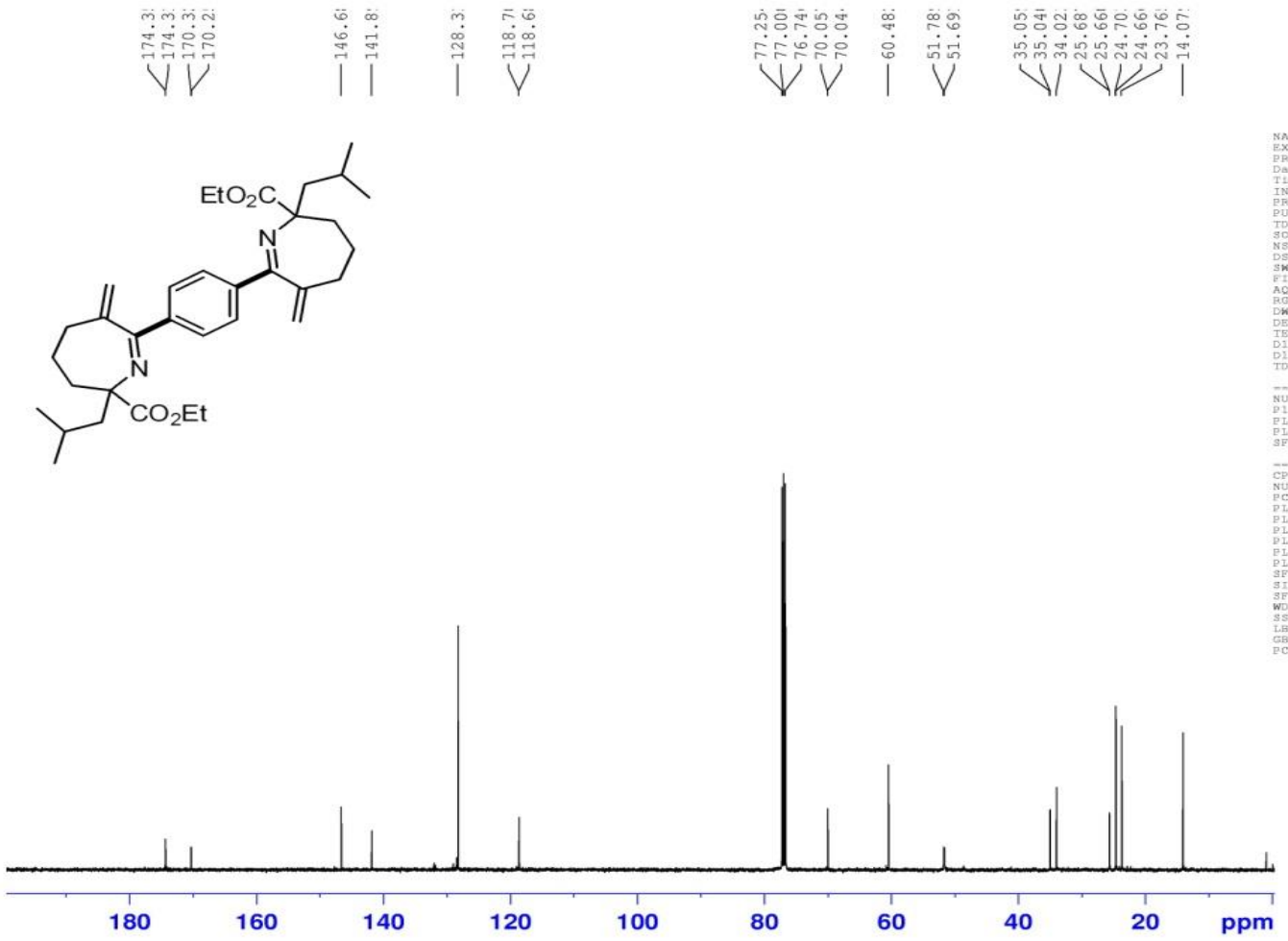
NAME	SC
EXPNO	101000
PROCNO	1
Date_	20160513
Time	4.58
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	299.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

3r



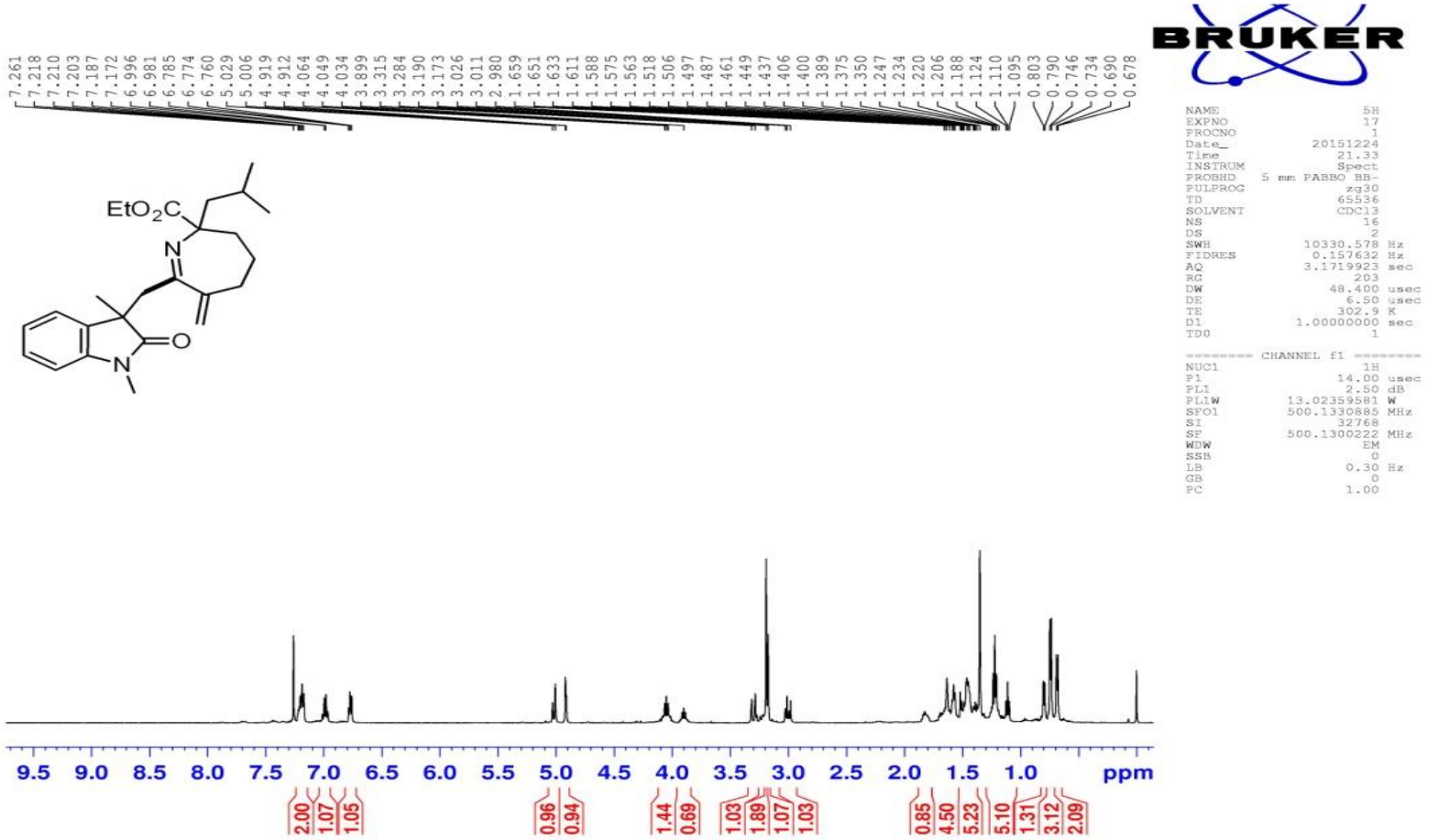
BRUKER

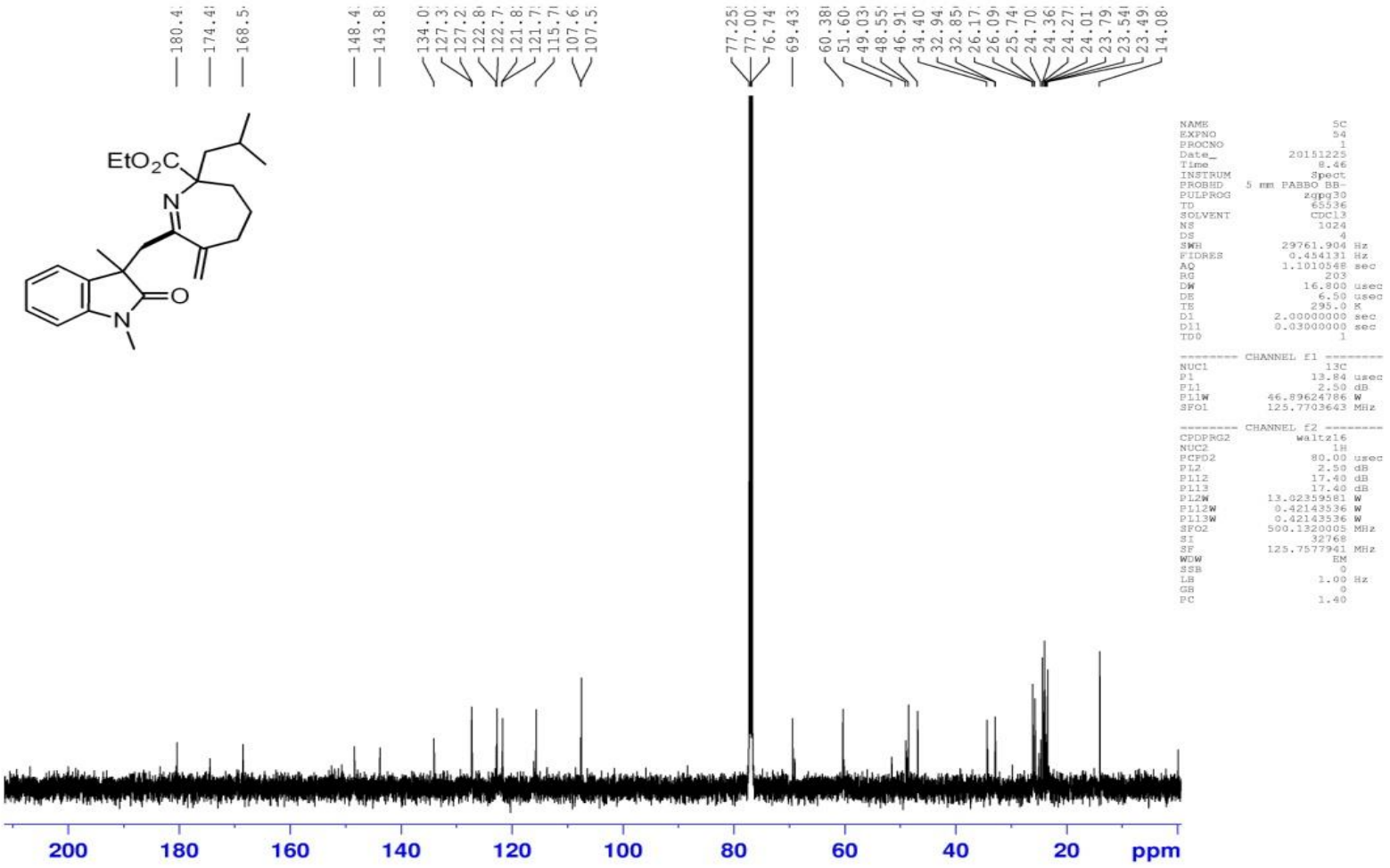
NAME	5H
EXPNO	3
PROCNO	1
Date_	20150804
Time	9.29
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	90.5
DW	60.400 usec
DE	6.50 usec
TE	300.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300094 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



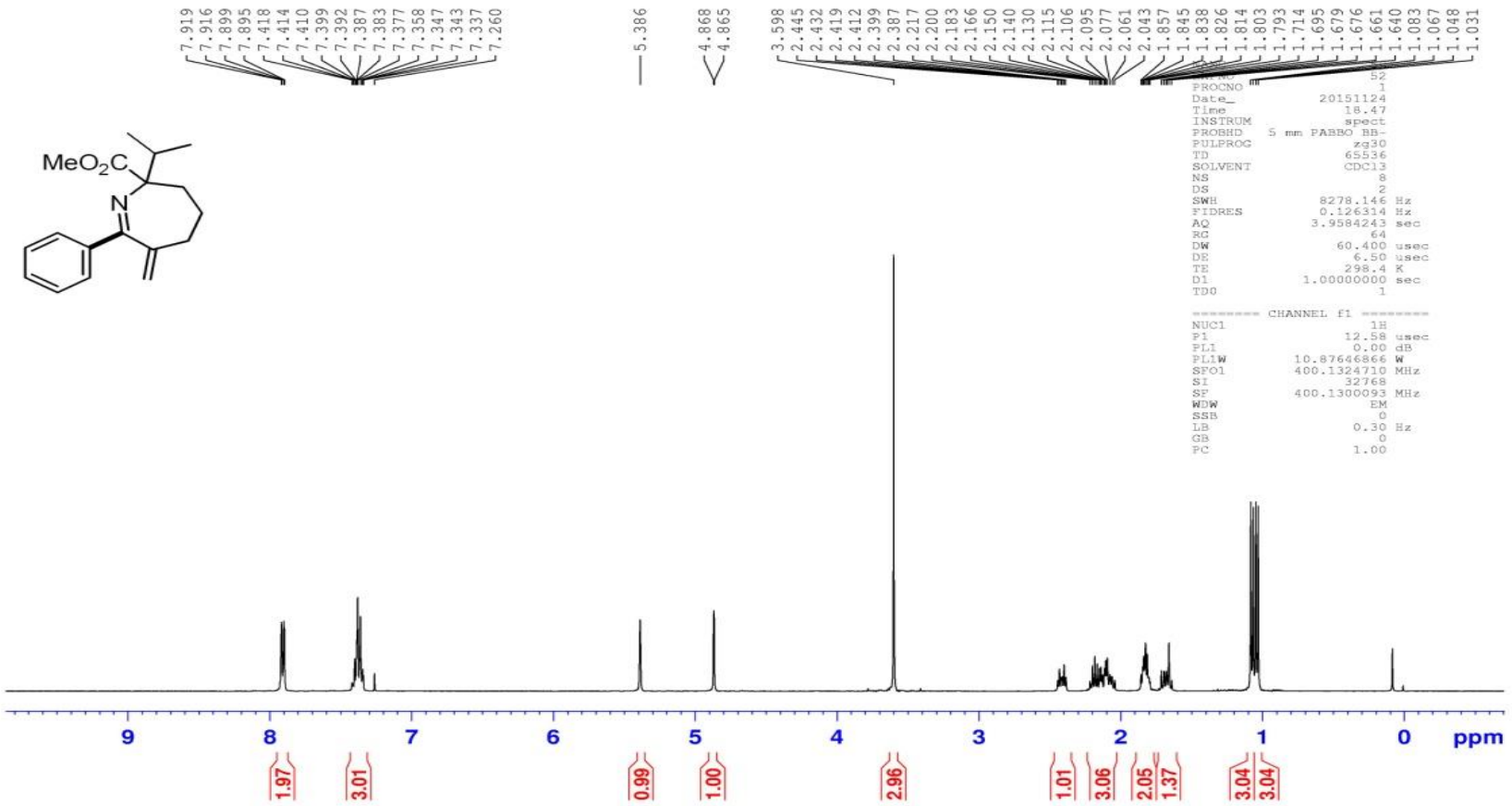

```
NAME                  5C
EXPNO                 15
PROCNO                 1
Date_           20150805
Time                14.56
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1094
DS                    4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG                  203
DW               16.800 usec
DE                6.50 usec
TE               302.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                13.84 usec
PL1                2.50 dB
PL1W         46.89624786 W
SFO1        125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             80.00 usec
PL2                2.50 dB
PL12              17.40 dB
PL13              17.40 dB
PL2W         13.02359581 W
PL12W         0.42143536 W
PL13W         0.42143536 W
SFO2        500.1320005 MHz
SI                32768
SF          125.7577922 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

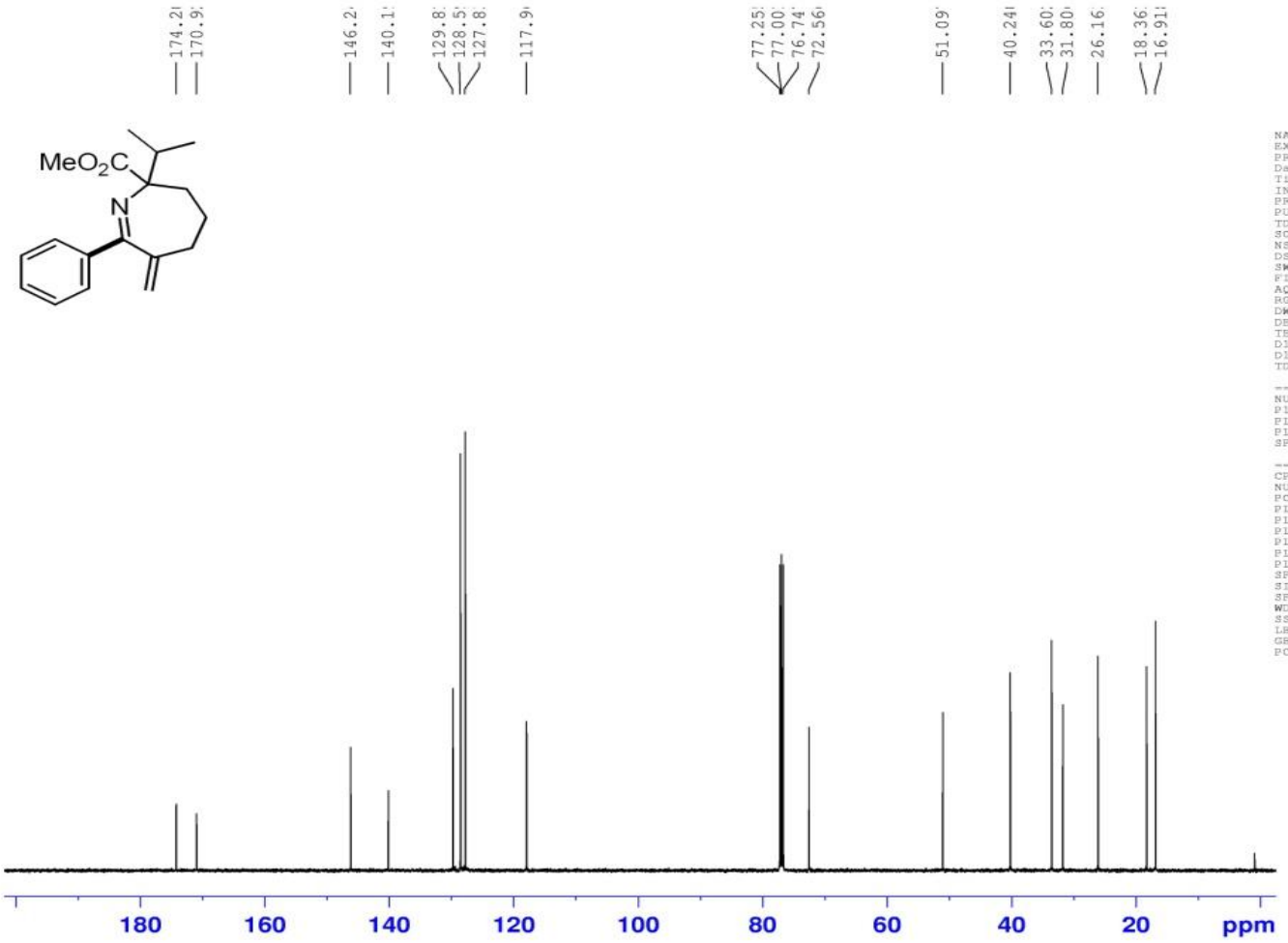
3s


7.261
7.218
7.210
7.203
7.187
7.172
6.996
6.981
6.785
6.774
6.760
5.029
5.006
4.919
4.912
4.064
4.049
4.034
3.899
3.315
3.284
3.190
3.173
3.026
3.011
2.980
1.659
1.651
1.633
1.611
1.588
1.575
1.563
1.518
1.506
1.497
1.487
1.461
1.449
1.437
1.406
1.400
1.389
1.375
1.350
1.247
1.234
1.220
1.206
1.188
1.124
1.110
1.095
0.803
0.790
0.746
0.734
0.690
0.678
EtO2C
N
O
N
NAME 5H
EXPNO 17
PROCNO 1
Date_ 20151224
Time 21.33
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 203
DW 48.400 usec
DE 6.50 usec
TE 302.9 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.00 usec
PL1 2.50 dB
PL1W 13.02359581 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300222 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.00 1.07 1.05 0.96 0.94 1.44 0.69 1.03 1.89 1.07 1.03 0.85 4.50 5.23 5.10 1.31 3.12 2.09


180.4
174.4
168.5
148.4
143.8
134.0
127.3
127.2
122.8
122.7
121.8
121.7
115.7
107.6
107.5
77.25
77.00
76.74
69.43
60.38
51.60
49.03
48.55
46.91
34.40
32.94
32.85
26.17
26.09
25.74
24.70
24.36
24.27
24.01
23.79
23.54
23.49
14.08
EtO2C
N
O
N
NAME 5C
EXPNO 54
PROCNO 1
Date_ 20151225
Time 8.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577941 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
ppm

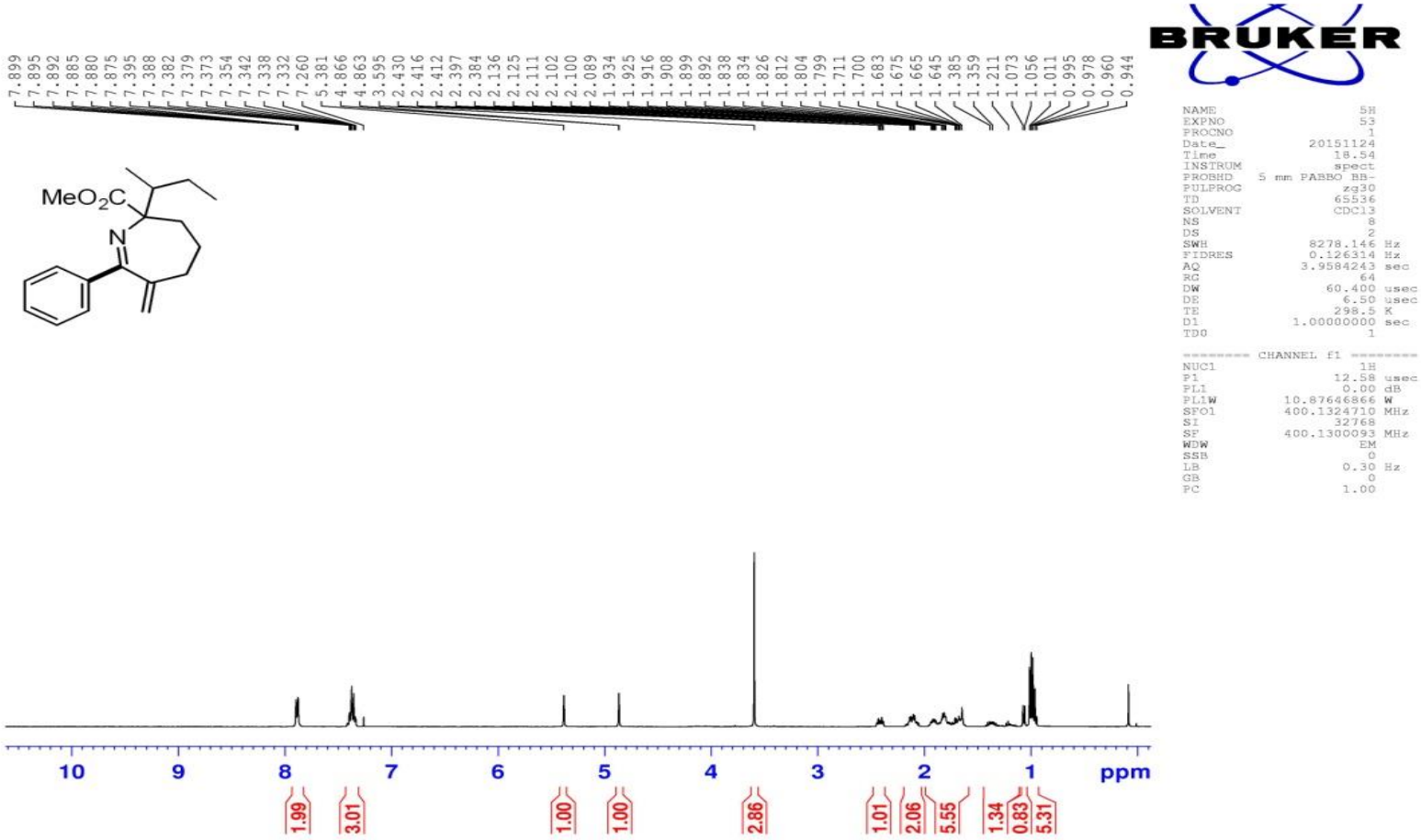
4a

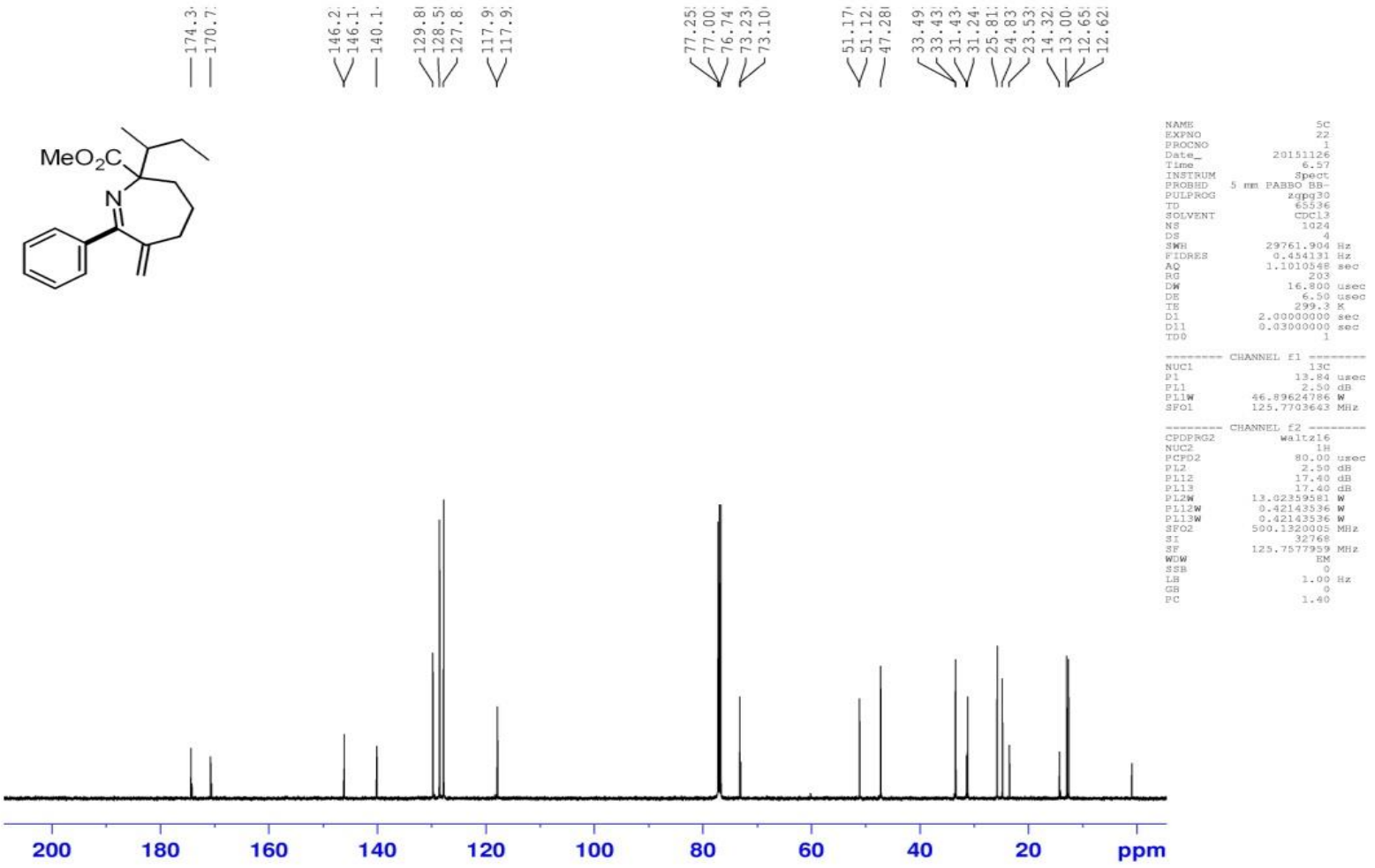

7.919
7.916
7.899
7.895
7.418
7.414
7.410
7.399
7.392
7.387
7.383
7.377
7.358
7.347
7.343
7.337
7.260
5.386
4.868
4.865
3.598
2.445
2.432
2.419
2.412
2.399
2.387
2.217
2.200
2.183
2.166
2.150
2.140
2.130
2.115
2.106
2.095
2.077
2.061
2.043
1.857
1.845
1.838
1.826
1.814
1.803
1.793
1.714
1.695
1.679
1.676
1.661
1.640
1.083
1.067
1.048
1.031
MeO2C
N
PROCNO 1
Date_ 20151124
Time 18.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 64
DW 60.400 usec
DE 6.50 usec
TE 298.4 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.97
3.01
0.99
1.00
2.96
1.01
3.06
2.05
1.37
3.04
3.04



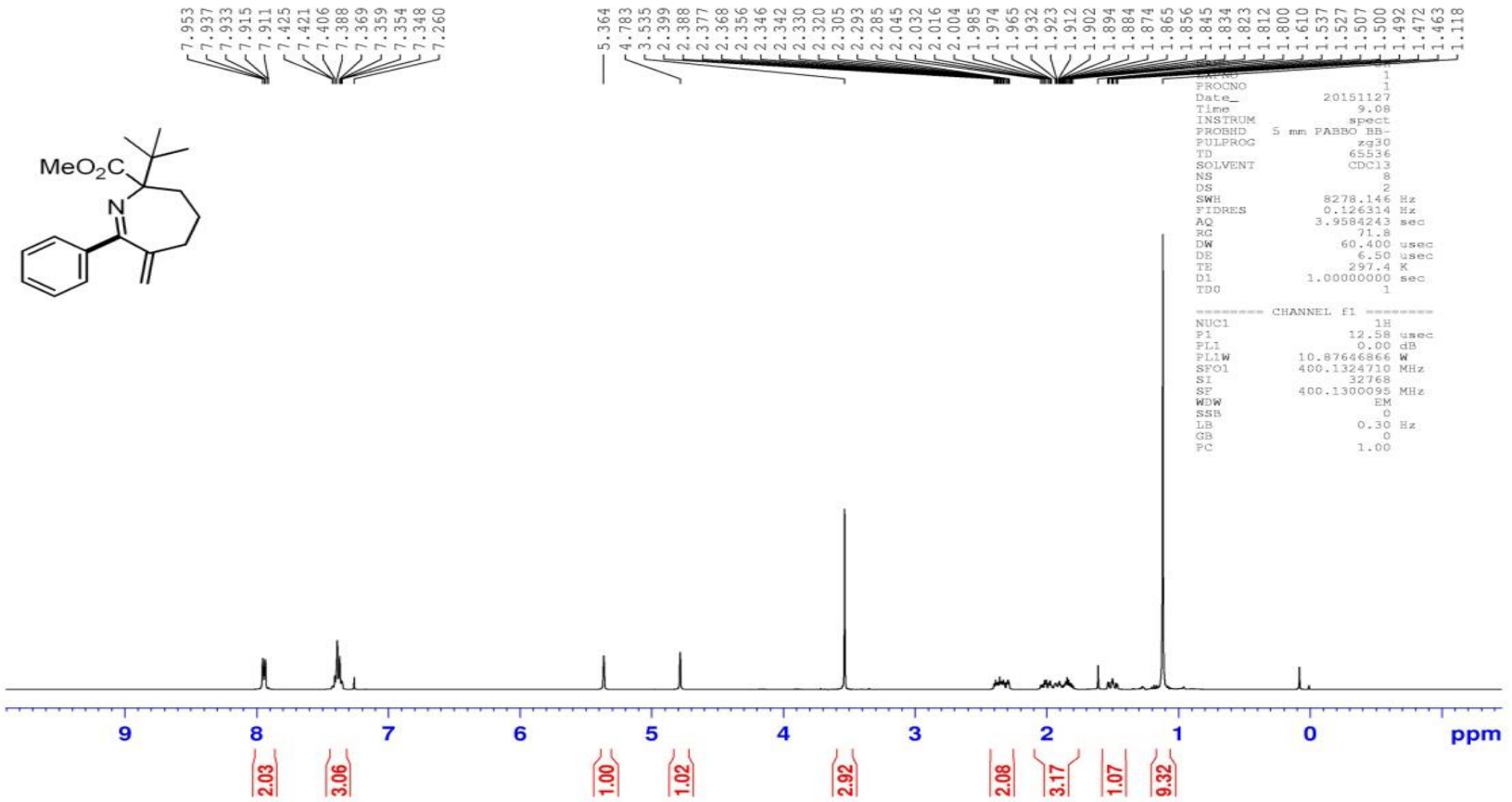
NAME	SC
EXPNO	21
PROCNO	1
Date_	20151126
Time	5.57
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	299.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577967 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

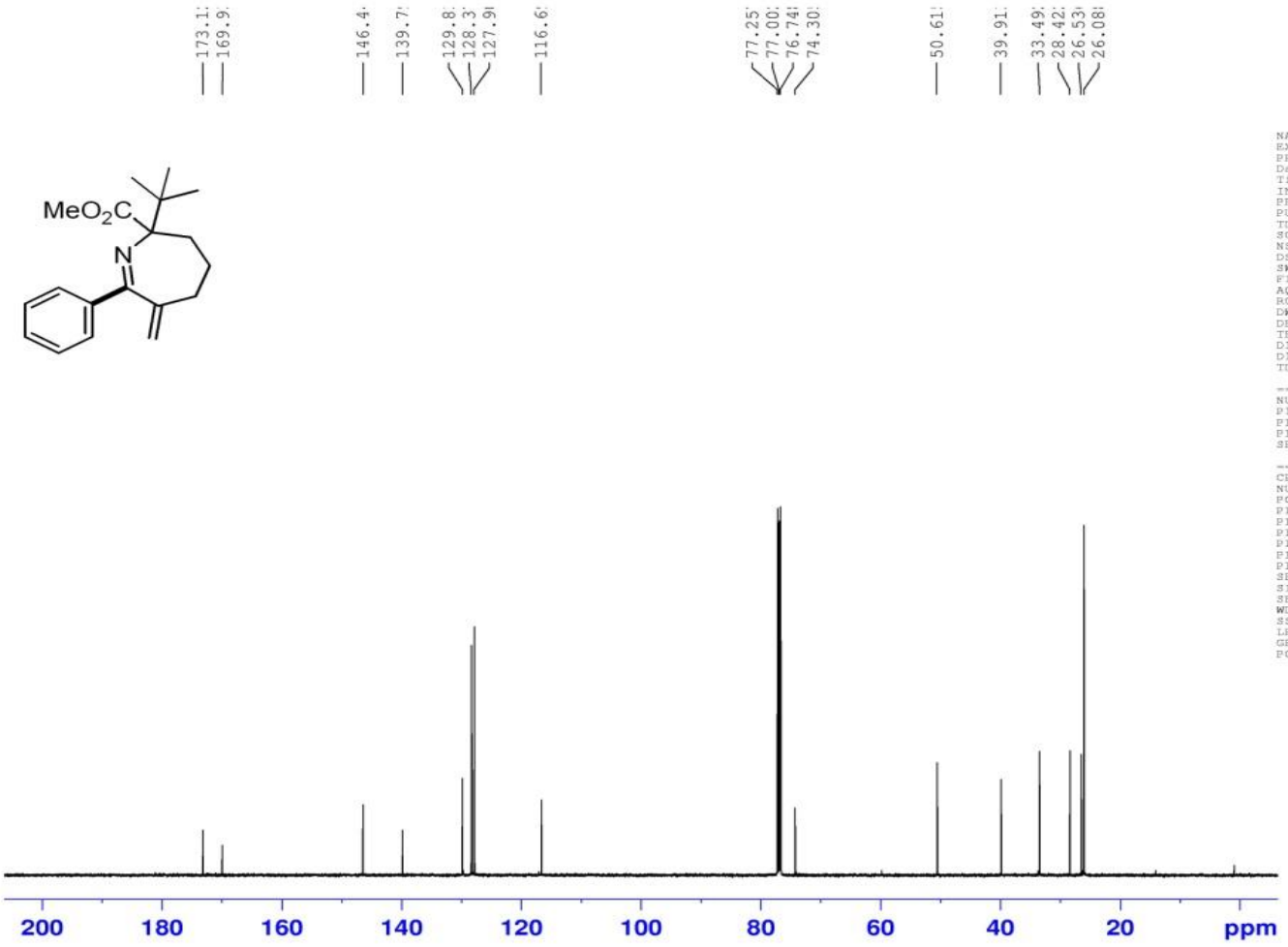
4b


7.899
7.895
7.892
7.885
7.880
7.875
7.395
7.388
7.382
7.379
7.373
7.354
7.342
7.338
7.332
7.260
5.381
4.866
4.863
3.595
2.430
2.416
2.412
2.397
2.384
2.136
2.125
2.111
2.102
2.100
2.089
1.934
1.925
1.916
1.908
1.899
1.892
1.838
1.834
1.826
1.812
1.804
1.799
1.711
1.700
1.683
1.675
1.665
1.645
1.385
1.359
1.211
1.073
1.056
1.011
0.995
0.978
0.960
0.944
MeO2C
N
BRUKER
NAME 5H
EXPNO 53
PROCNO 1
Date_ 20151124
Time 18.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 64
DW 60.400 usec
DE 6.50 usec
TE 298.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.99
3.01
1.00
1.00
2.86
1.01
2.06
5.55
1.34
0.83
5.31


174.3
170.7
146.2
146.1
140.1
129.8
128.5
127.8
117.9
117.9
77.25
77.00
76.74
73.23
73.10
51.17
51.12
47.28
33.49
33.43
31.43
31.24
25.81
24.83
23.53
14.32
13.00
12.65
12.62
MeO2C
N
NAME 5C
EXPNO 22
PROCNO 1
Date_ 20151126
Time 6.57
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577959 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
ppm

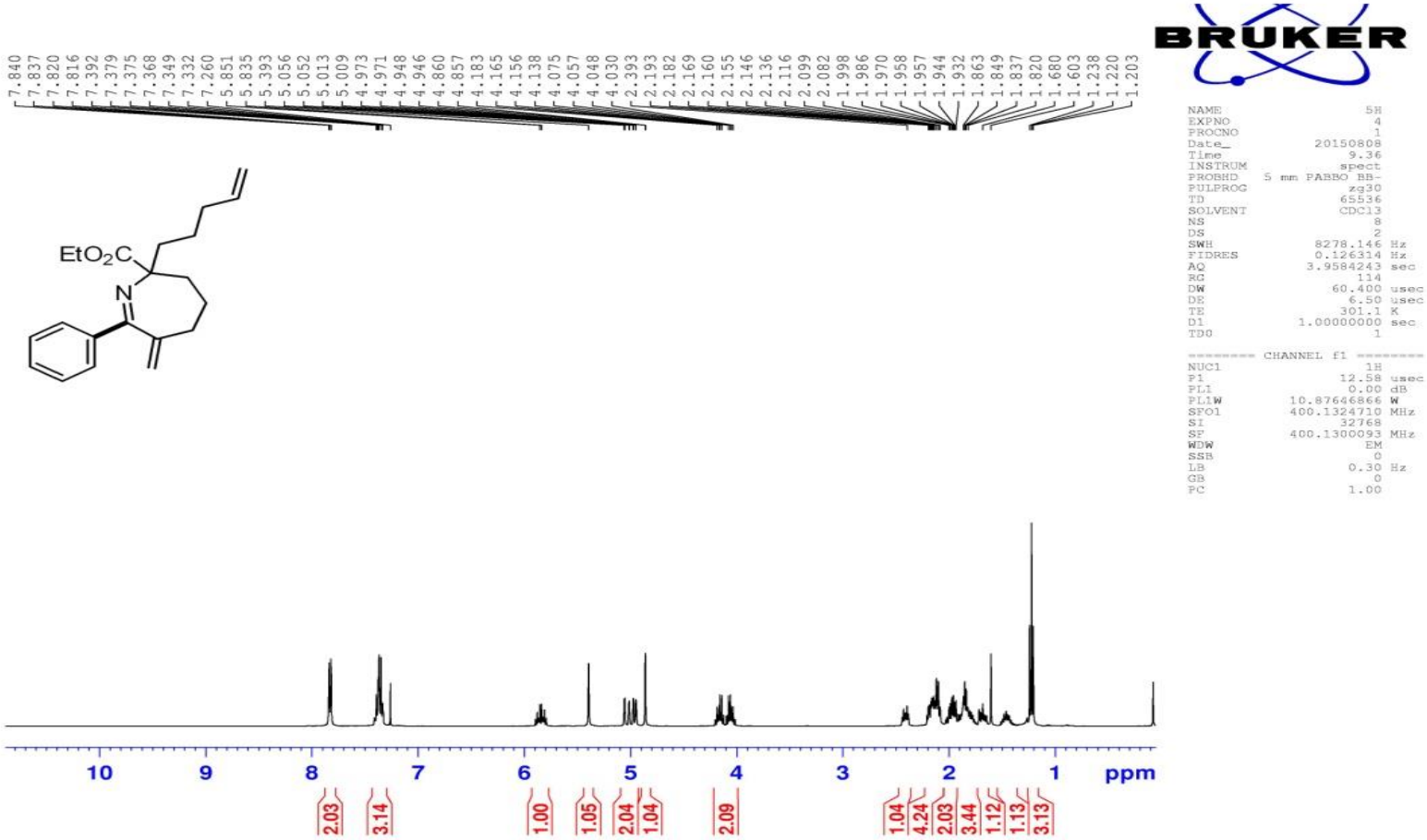
4c

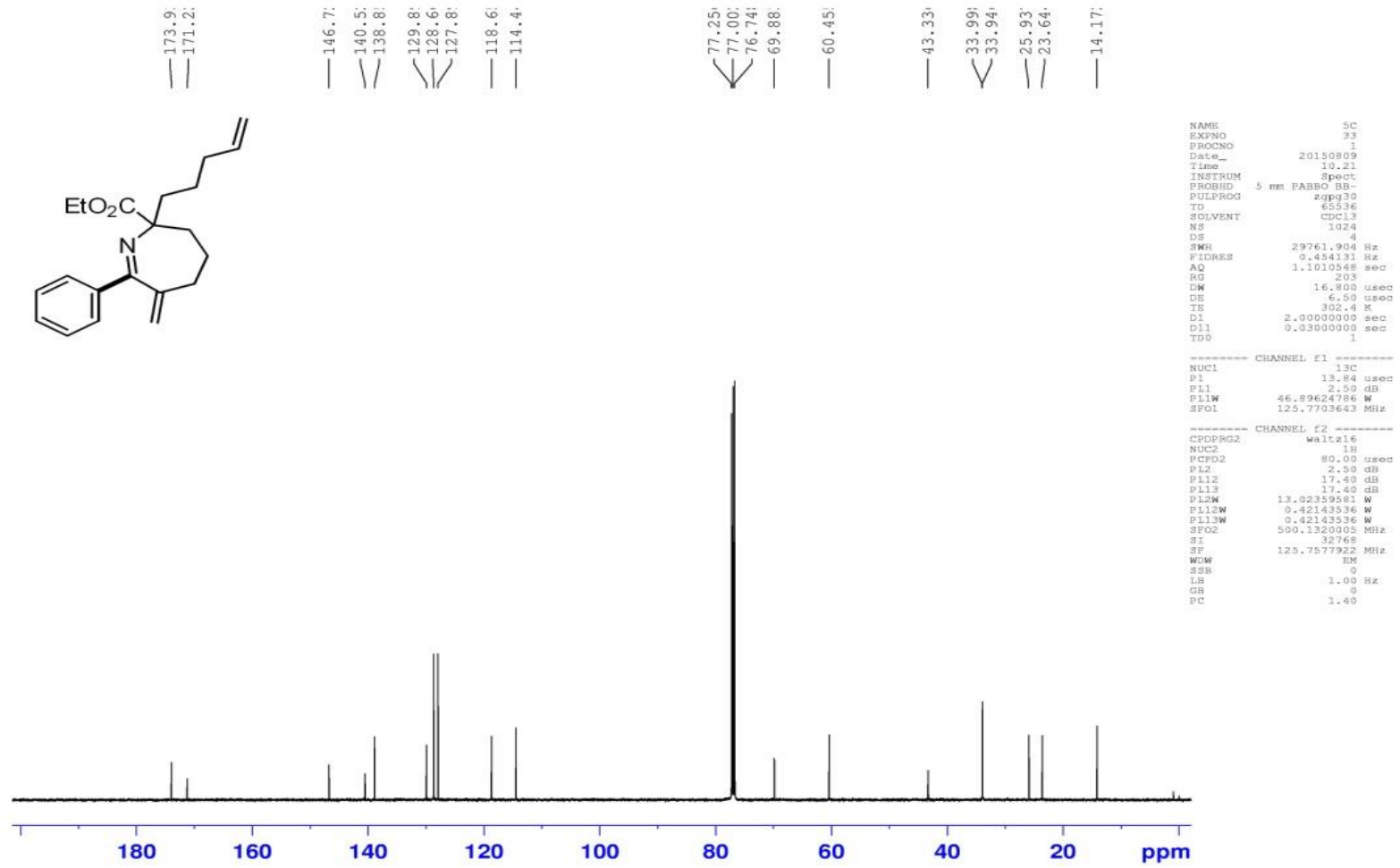

7.953
7.937
7.933
7.915
7.911
7.425
7.421
7.406
7.388
7.369
7.359
7.354
7.348
7.260
5.364
4.783
3.535
2.399
2.388
2.377
2.368
2.356
2.346
2.342
2.330
2.320
2.305
2.293
2.285
2.045
2.032
2.016
2.004
1.985
1.974
1.965
1.932
1.923
1.912
1.902
1.894
1.884
1.874
1.865
1.856
1.845
1.834
1.823
1.812
1.800
1.610
1.537
1.527
1.507
1.500
1.492
1.472
1.463
1.118
MeO2C
N
PROCNO 1
Date_ 20151127
Time 9.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
2.03
3.06
1.00
1.02
2.92
2.08
3.17
1.07
9.32



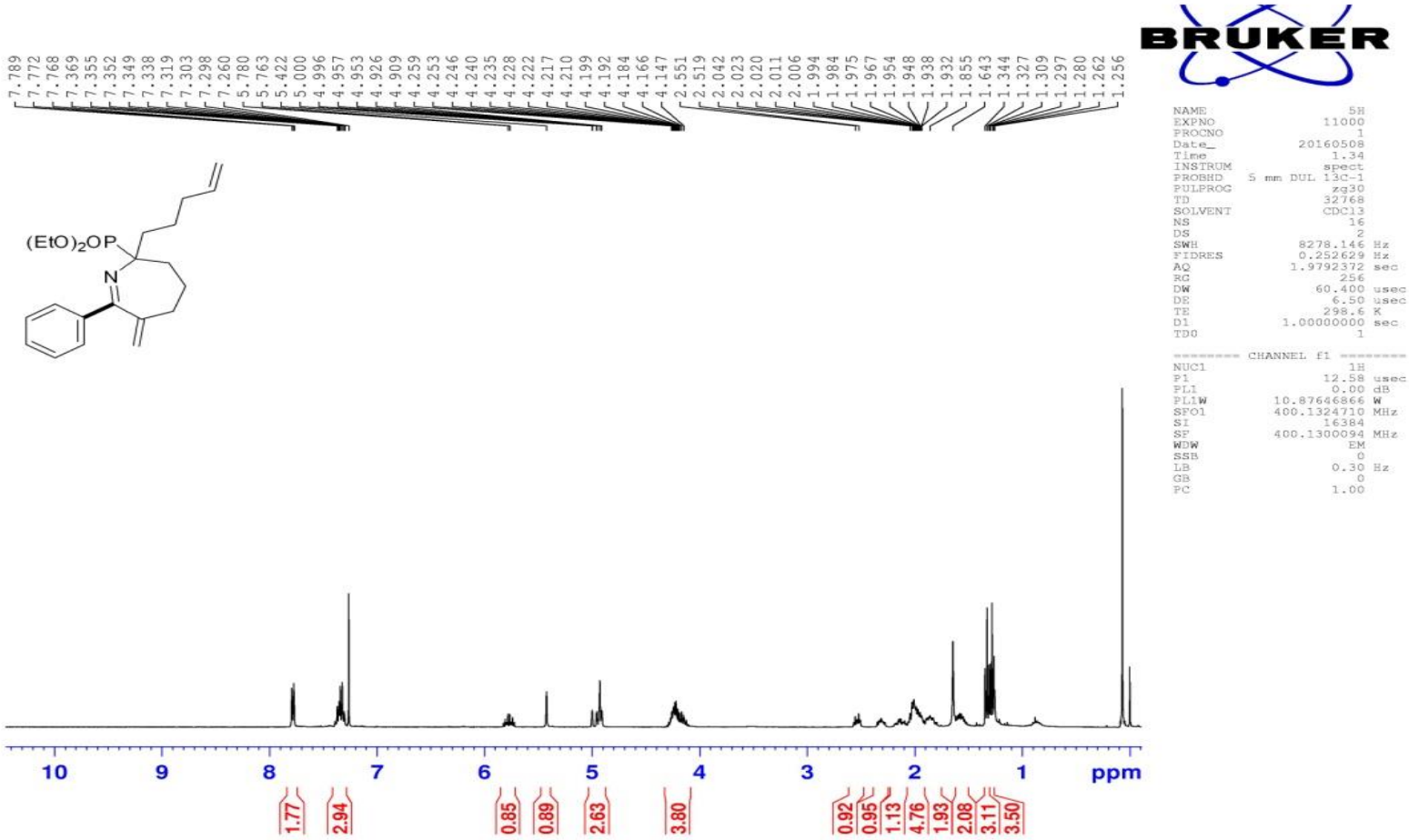
NAME	5C
EXPNO	1
PROCNO	1
Date_	20151127
Time	18.11
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	298.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577942 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

4d


7.840
7.837
7.820
7.816
7.392
7.379
7.375
7.368
7.349
7.332
7.260
5.851
5.835
5.393
5.056
5.052
5.013
5.009
4.973
4.971
4.948
4.946
4.860
4.857
4.183
4.165
4.156
4.138
4.075
4.057
4.048
4.030
2.393
2.193
2.182
2.169
2.160
2.155
2.146
2.136
2.116
2.099
2.082
1.998
1.986
1.970
1.958
1.957
1.944
1.932
1.863
1.849
1.837
1.820
1.680
1.603
1.238
1.220
1.203
EtO2C
N
BRUKER
NAME 5H
EXPNO 4
PROCNO 1
Date_ 20150808
Time 9.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 114
DW 60.400 usec
DE 6.50 usec
TE 301.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
2.03
3.14
1.00
1.05
2.04
1.04
2.09
1.04
4.24
2.03
3.44
1.12
1.13
3.13

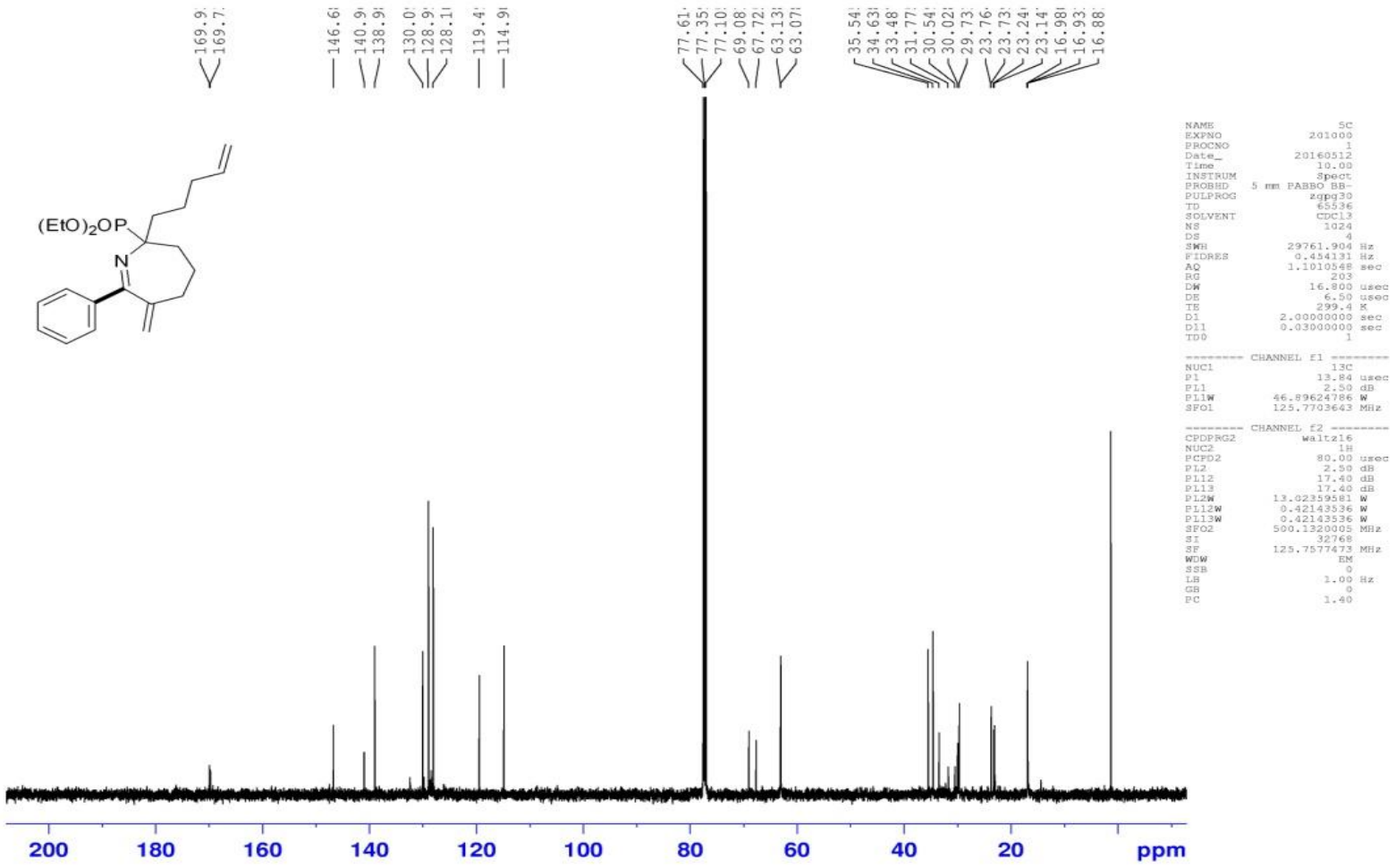

173.9
171.2
146.7
140.5
138.8
129.8
128.6
127.8
118.6
114.4
77.25
77.00
76.74
69.88
60.45
43.33
33.99
33.94
25.93
23.64
14.17
EtO2C
N
NAME SC
EXPNO 33
PROCNO 1
Date_ 20150809
Time 10.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 302.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577922 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180
160
140
120
100
80
60
40
20
ppm

4e

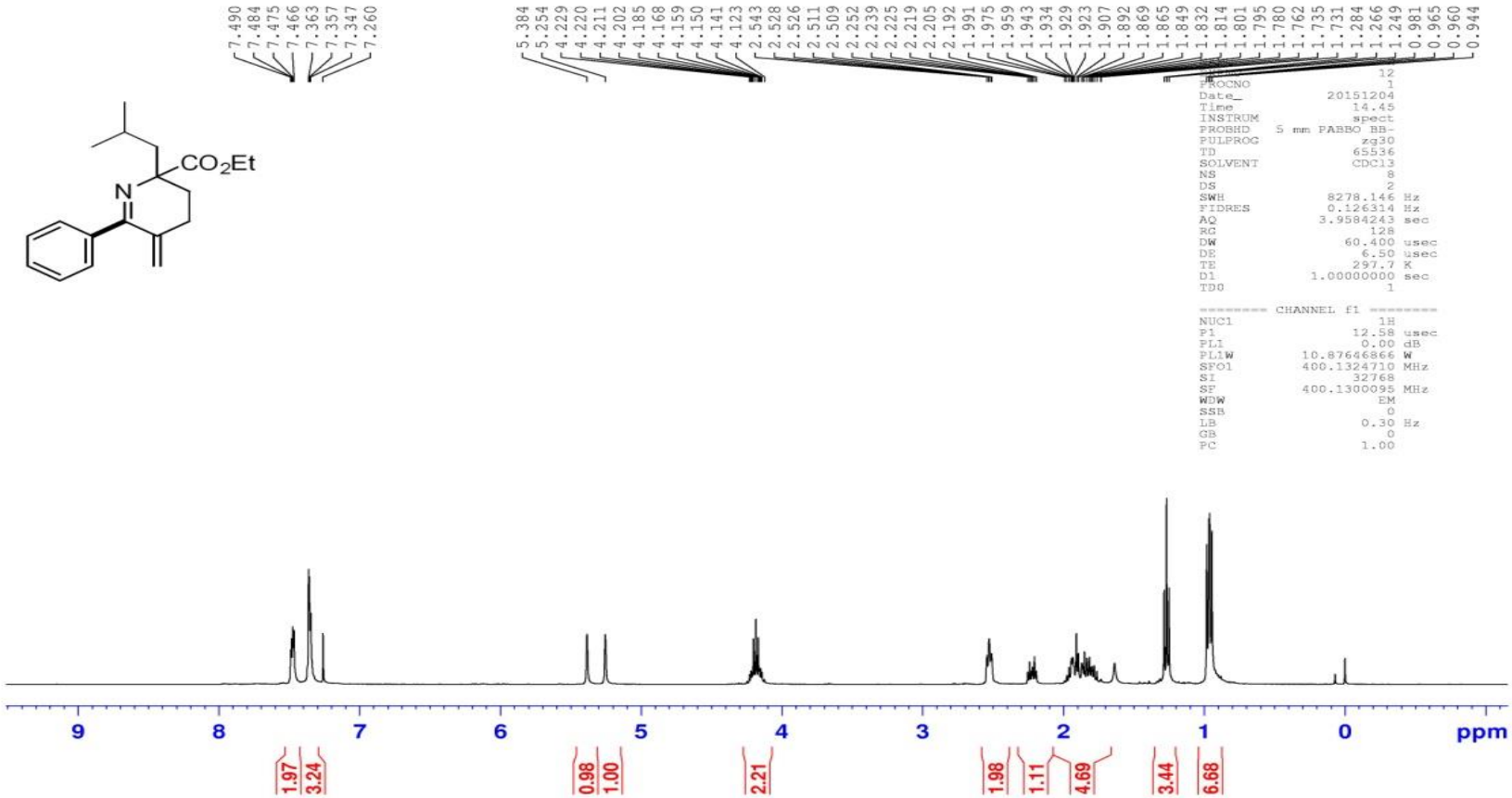


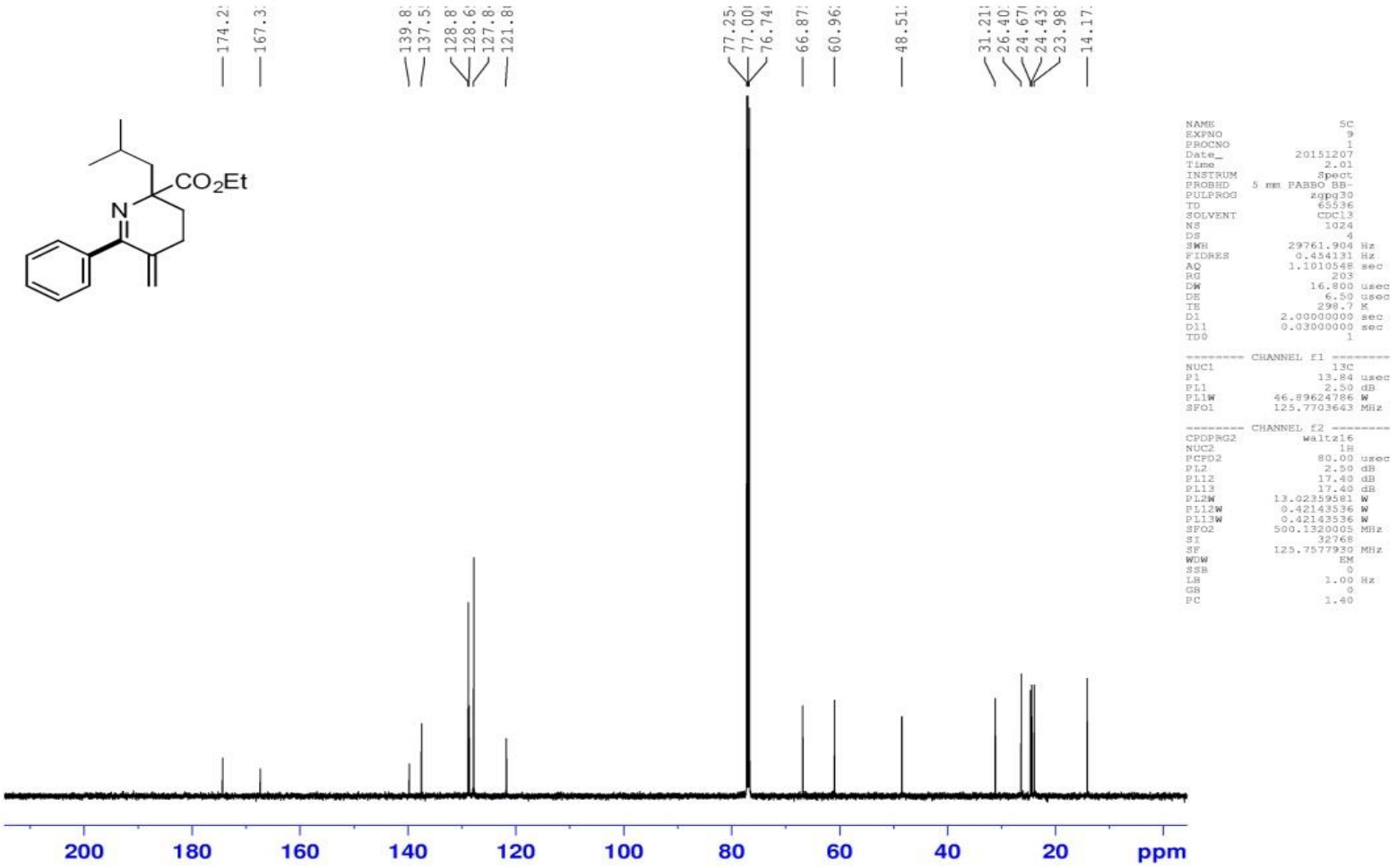
BRUKER

NAME	5H
EXPNO	11000
PROCNO	1
Date_	20160508
Time	1.34
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.252629 Hz
AQ	1.9792372 sec
RG	256
DW	60.400 usec
DE	6.50 usec
TE	298.6 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	16384
SF	400.1300094 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00


169.9
169.7
146.6
140.9
138.9
130.0
128.9
128.1
119.4
114.9
77.61
77.35
77.10
69.08
67.72
63.13
63.07
35.54
34.63
33.48
31.77
30.54
30.02
29.73
23.76
23.73
23.24
23.14
16.98
16.93
16.88
(EtO)2OP
N
NAME 5C
EXPNO 201009
PROCNO 1
Date_ 20160512
Time 10.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 299.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577473 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
ppm

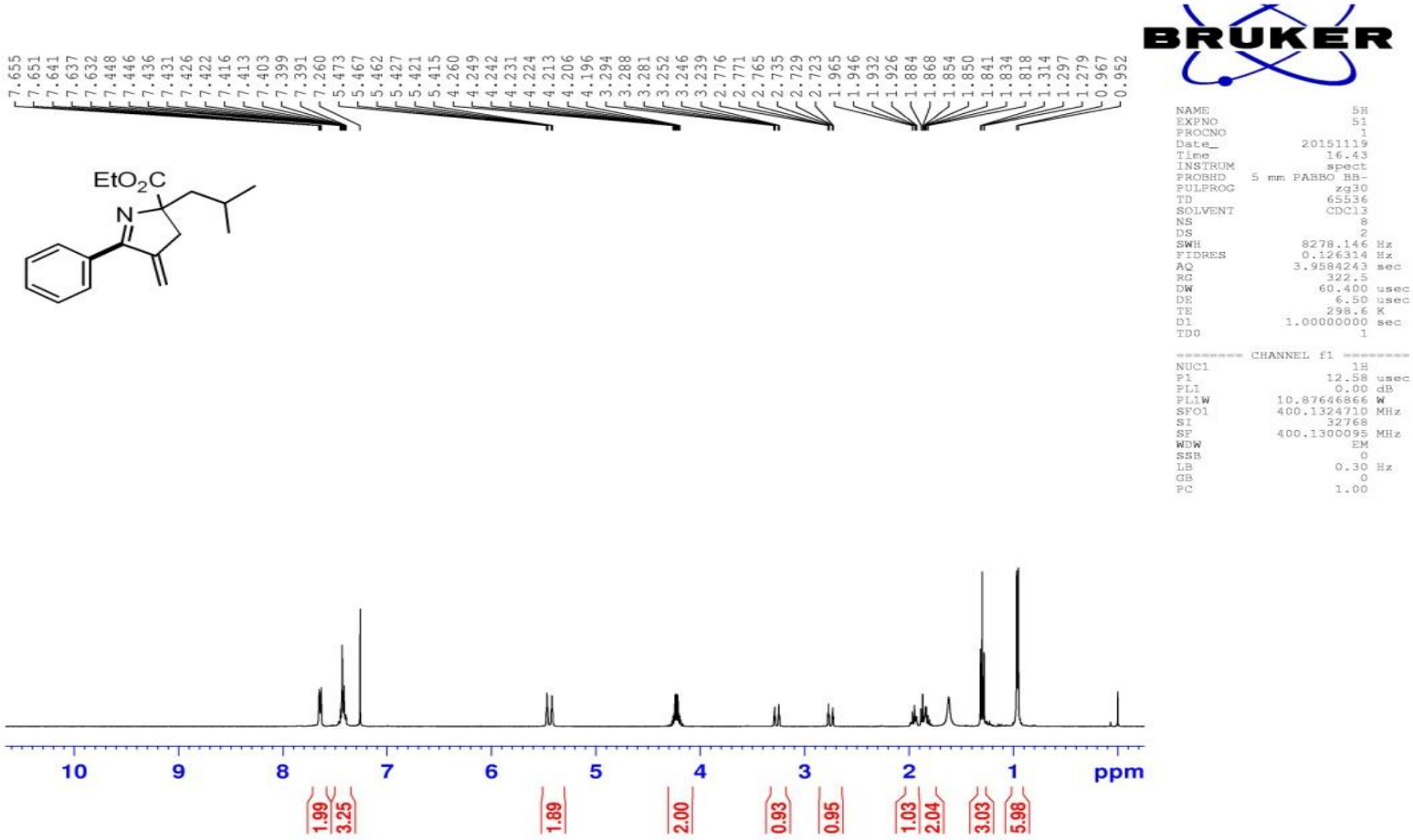
4f

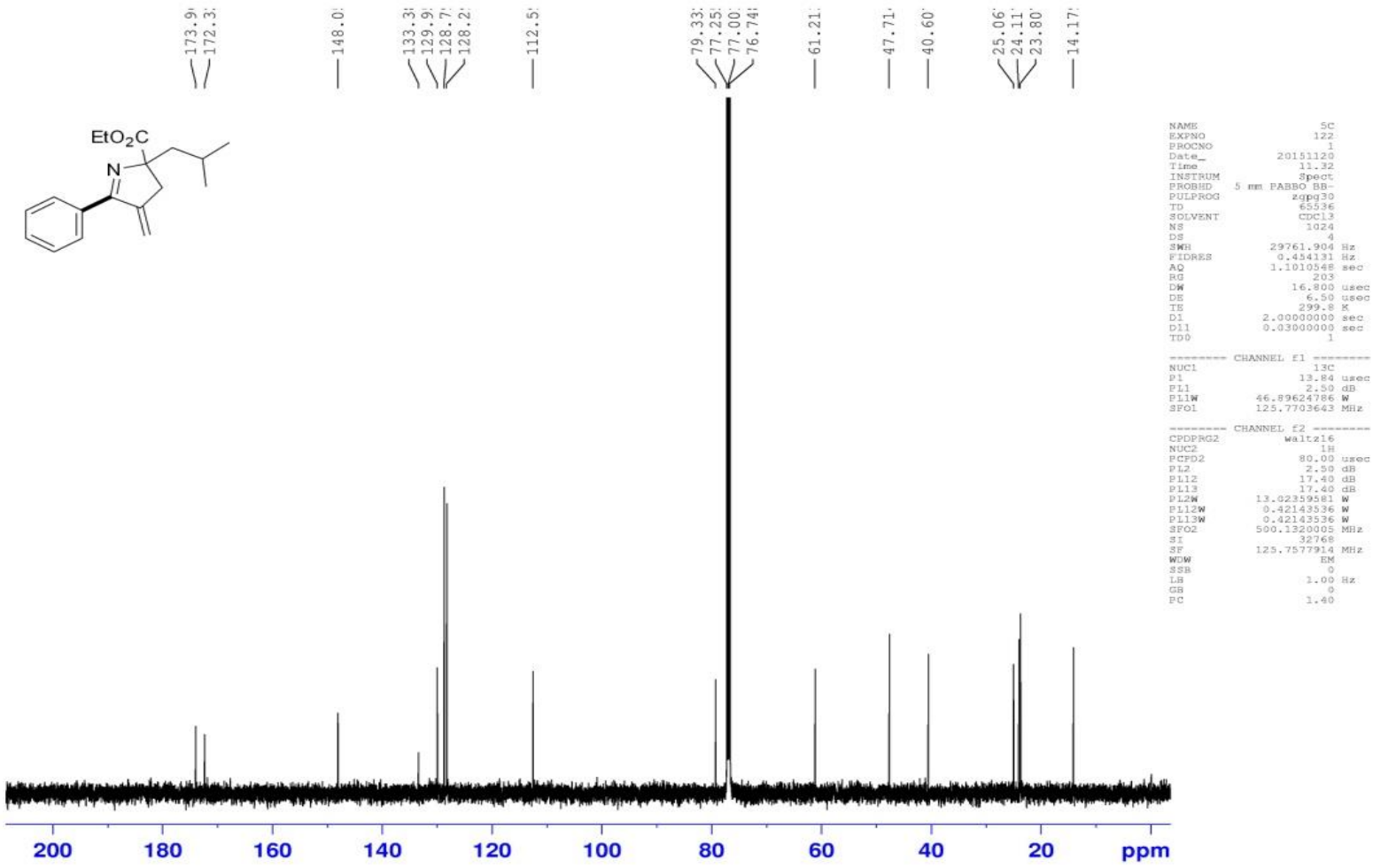

7.490
7.484
7.475
7.466
7.363
7.357
7.347
7.260
5.384
5.254
4.229
4.220
4.211
4.202
4.185
4.168
4.159
4.150
4.141
4.123
2.543
2.528
2.526
2.511
2.509
2.252
2.239
2.225
2.219
2.205
2.192
1.991
1.975
1.959
1.943
1.934
1.929
1.923
1.907
1.892
1.869
1.865
1.849
1.832
1.814
1.801
1.795
1.780
1.762
1.735
1.731
1.284
1.266
1.249
0.981
0.965
0.960
0.944
CO2Et
N
PROCNO 1
Date_ 20151204
Time 14.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DW 60.400 usec
DE 6.50 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.97
3.24
0.98
1.00
2.21
1.98
1.11
4.69
3.44
6.68



NAME	SC
EXPNO	9
PROCNO	1
Date_	20151207
Time	2.01
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	298.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577930 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

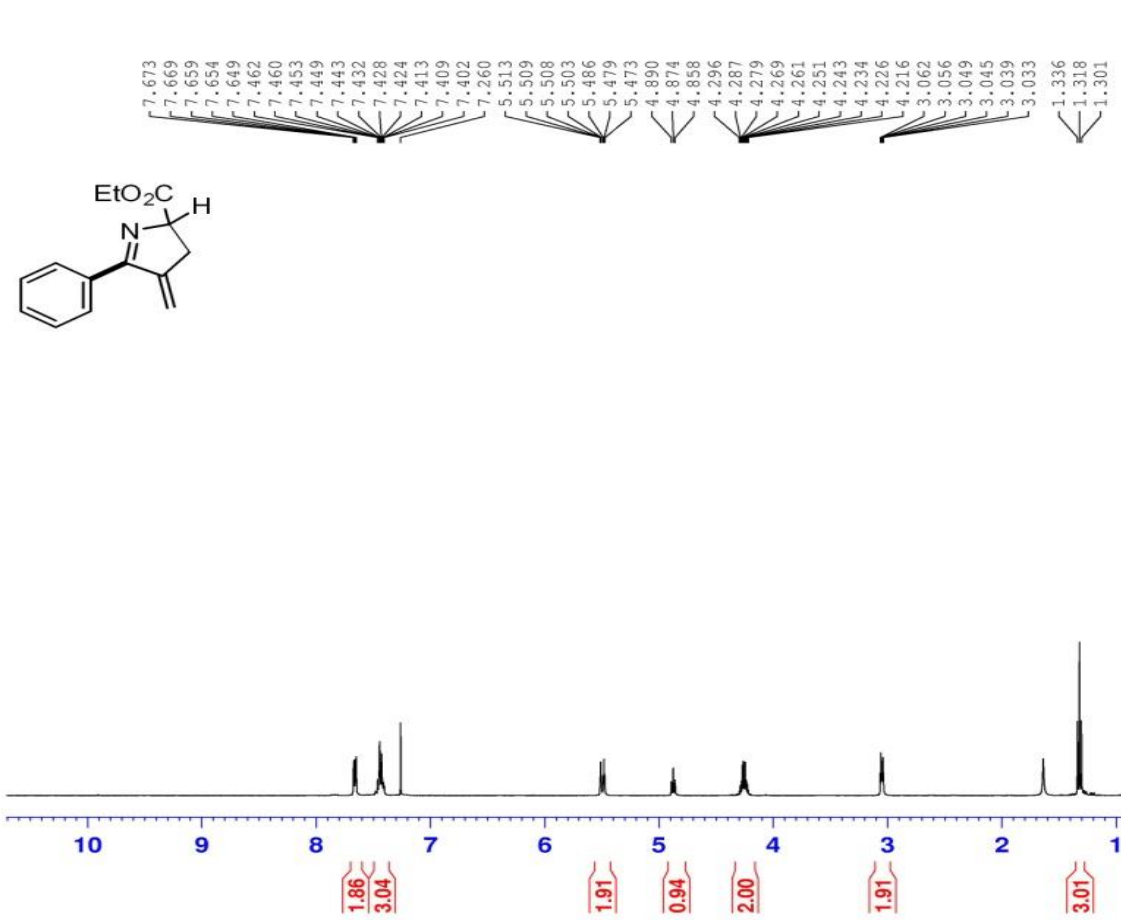
4g


7.655
7.651
7.641
7.637
7.632
7.448
7.446
7.436
7.431
7.426
7.422
7.416
7.413
7.403
7.399
7.391
7.260
5.473
5.467
5.462
5.427
5.421
5.415
4.260
4.249
4.242
4.231
4.224
4.213
4.206
4.196
3.294
3.288
3.281
3.252
3.246
3.239
2.776
2.771
2.765
2.735
2.729
2.723
1.965
1.946
1.932
1.926
1.884
1.868
1.854
1.850
1.841
1.834
1.818
1.314
1.297
1.279
0.967
0.952
EtO2C
N
BRUKER
NAME 5H
EXPNO 51
PROCNO 1
Date_ 20151119
Time 16.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.50 usec
TE 298.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.99
3.25
1.89
2.00
0.93
0.95
1.03
2.04
3.03
5.98



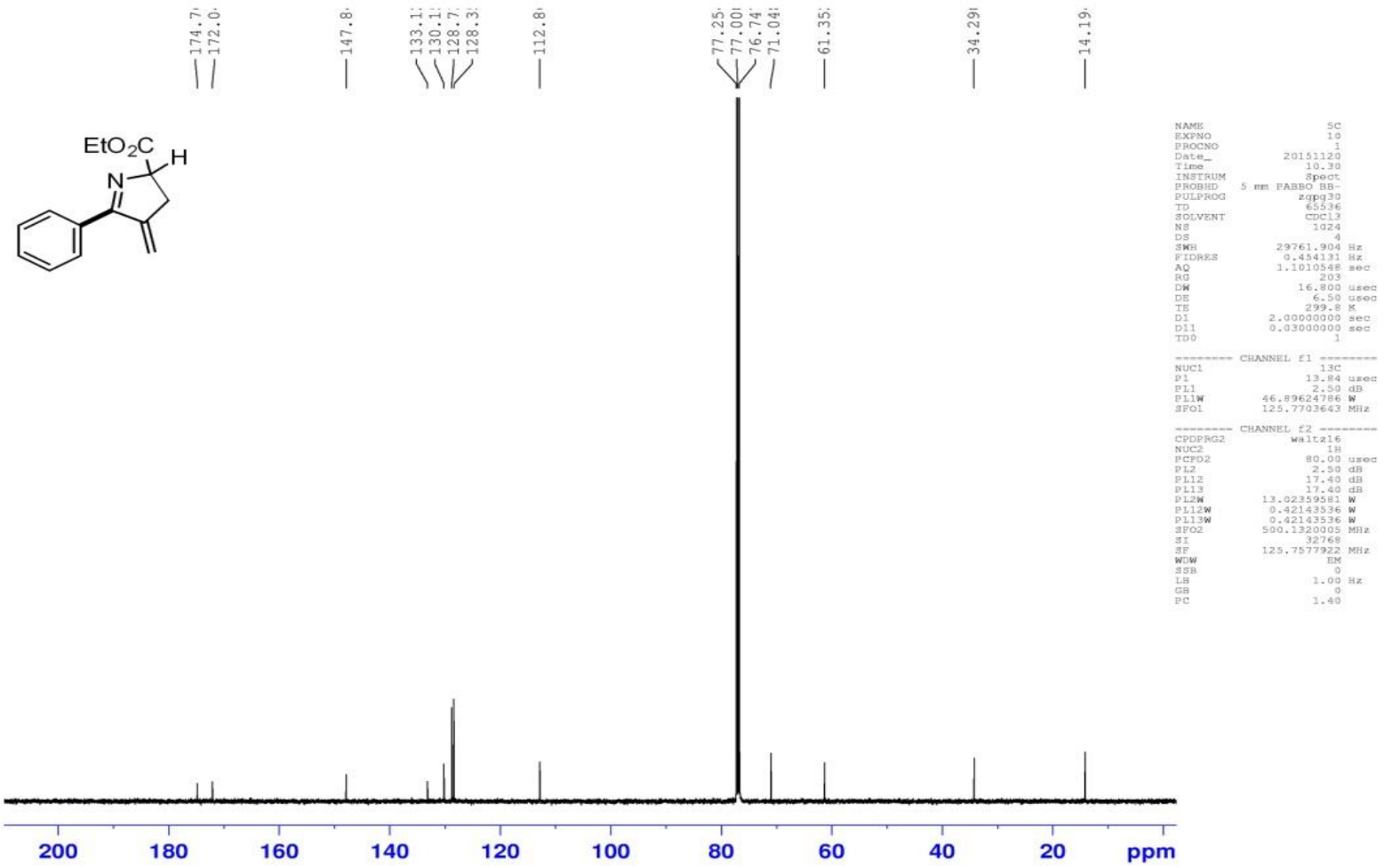
NAME	5C
EXPNO	122
PROCNO	1
Date_	20151120
Time	11.32
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	299.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577914 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

4h

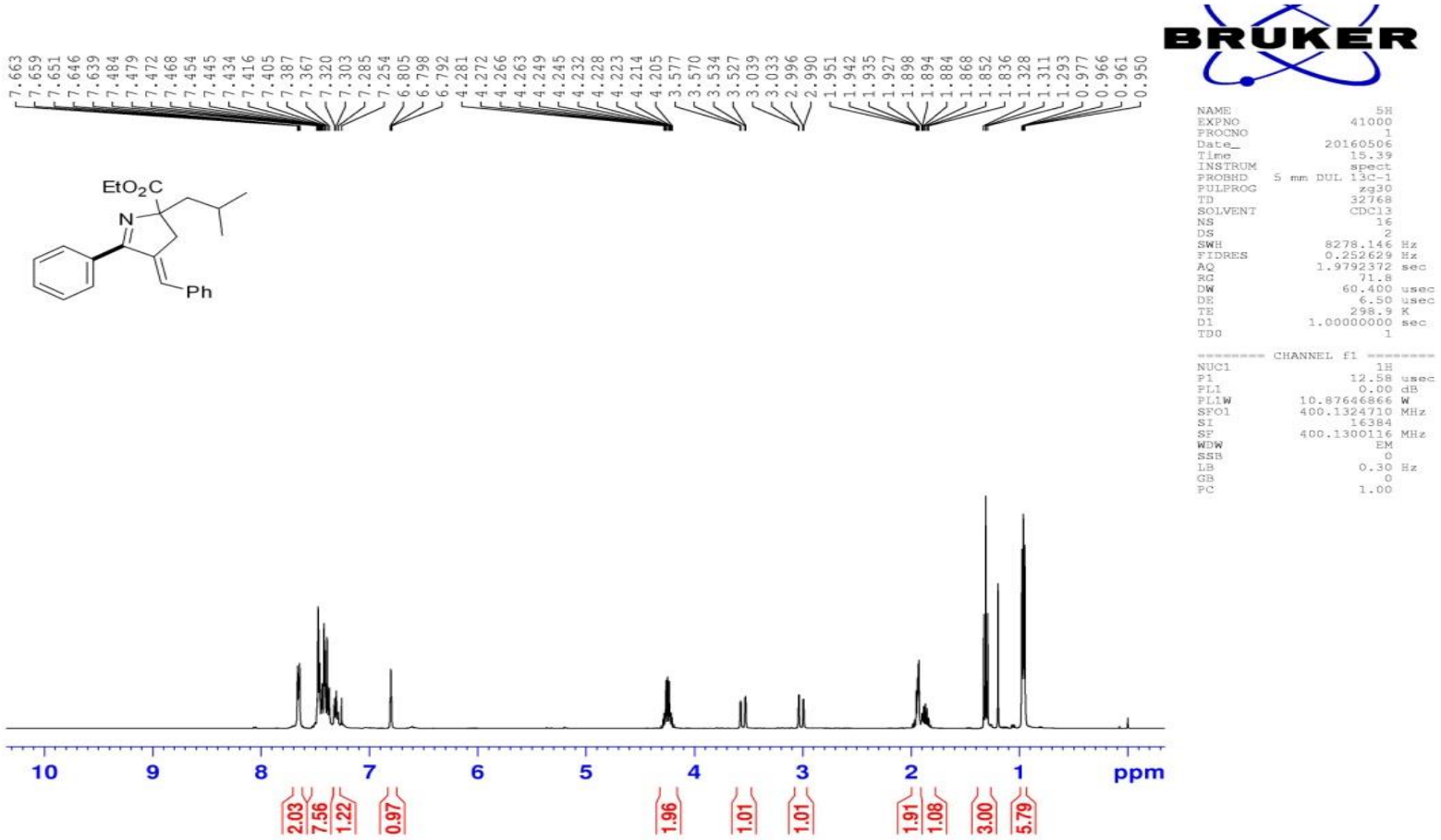


BRUKER

NAME	5H	
EXPNO	41	
PROCNO	1	
Date_	20151119	
Time	15.18	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	8	
DS	2	
SWH	8278.146	Hz
FIDRES	0.126314	Hz
AQ	3.9584243	sec
RG	322.5	
DW	60.400	usec
DE	6.50	usec
TE	298.8	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	12.58	usec
PL1	0.00	dB
PL1W	10.87646866	W
SFO1	400.1324710	MHz
SI	32768	
SF	400.1300095	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

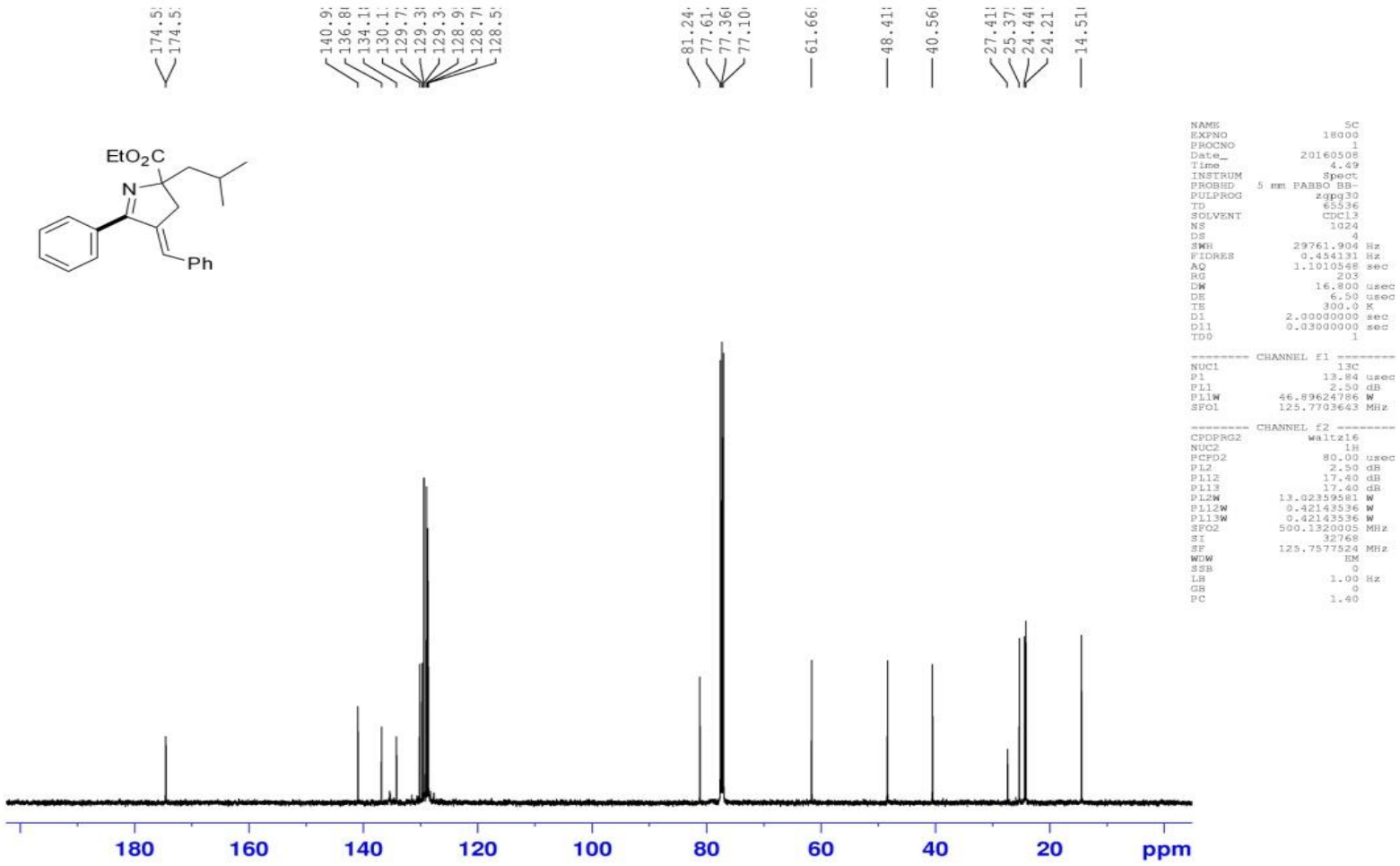

174.7
172.0
147.8
133.1
130.1
128.7
128.3
112.8
77.25
77.00
76.74
71.04
61.35
34.29
14.19
EtO2C
H
N
NAME 5C
EXPNO 10
PROCNO 1
Date_ 20151120
Time 10.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 299.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577922 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
ppm

4i



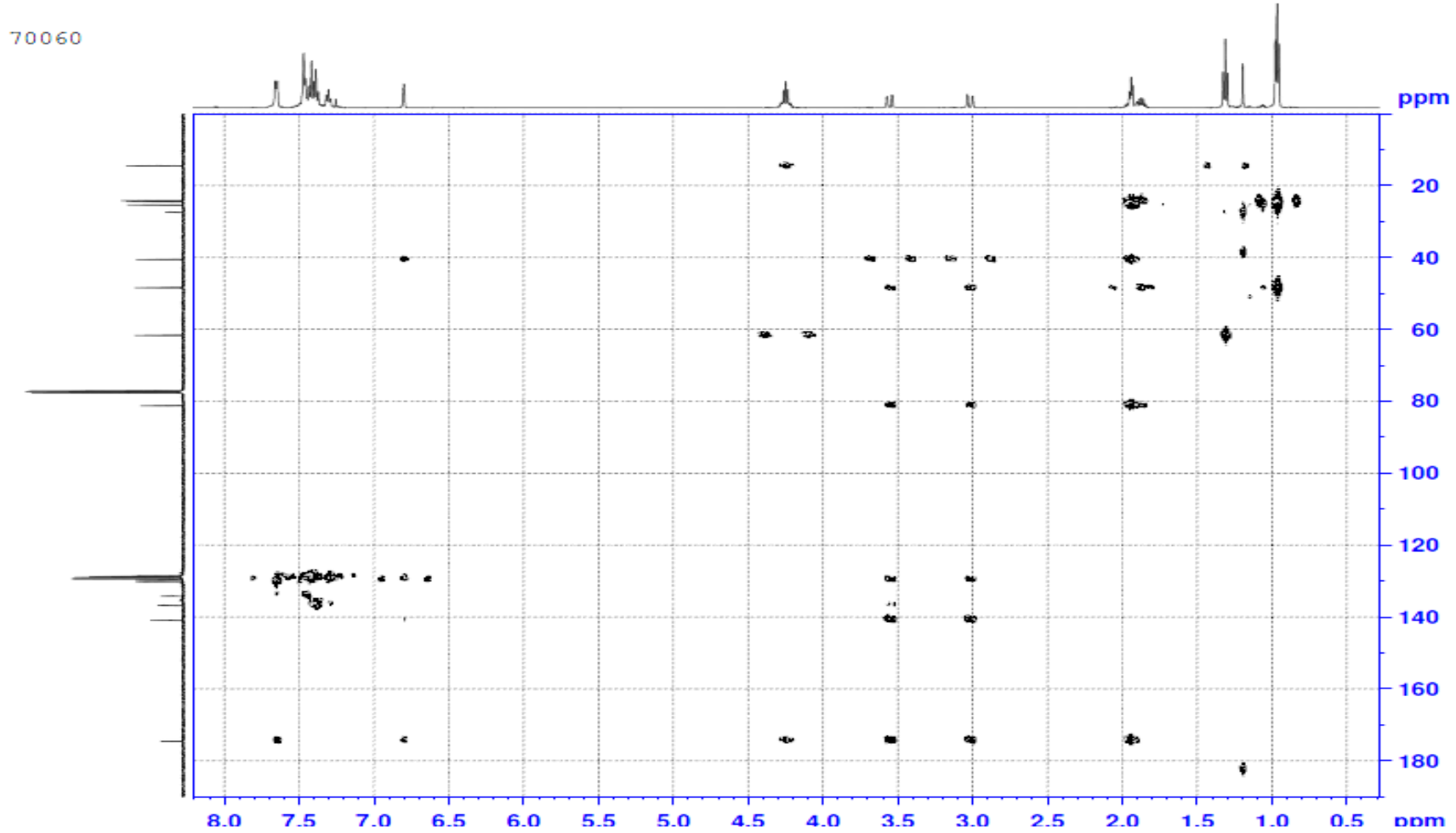
BRUKER

NAME	5H
EXPNO	41000
PROCNO	1
Date_	20160506
Time	15.39
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.252629 Hz
AQ	1.9792372 sec
RG	71.8
DW	60.400 usec
DE	6.50 usec
TE	298.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	16384
SF	400.1300116 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

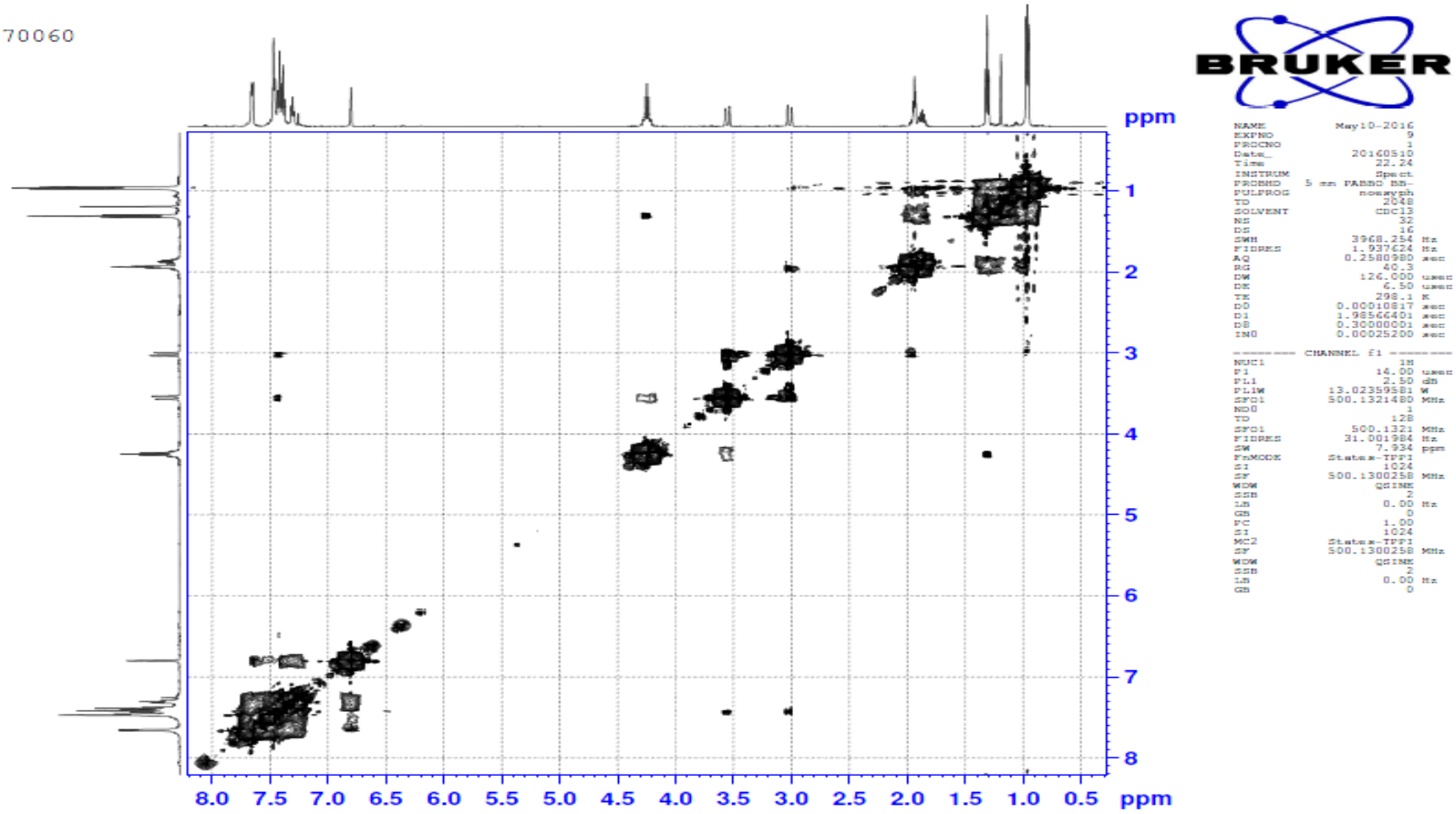


NAME	5C
EXPNO	18000
PROCNO	1
Date_	20160508
Time	4.49
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577524 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

4i-HMBC


70060
ppm
20
40
60
80
100
120
140
160
180
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

4i-NOE


70060
ppm
1
2
3
4
5
6
7
8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
BRUKER
NAME May10-2016
EXPNO 9
PROCNO 1
Date_ 20160510
Time 22.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG noesyph
TD 2048
SOLVENT CDCl3
NS 32
DS 16
SWH 3968.254 Hz
FIDRES 1.937624 Hz
AQ 0.2580980 sec
RG 40.5
DW 126.000 usec
DE 6.50 usec
TE 298.1 K
D0 0.00010817 sec
D1 1.98566401 sec
D8 0.30000001 sec
IN0 0.00025200 sec
CHANNEL f1
NUC1 1H
P1 14.00 usec
PL1 2.50 dB
PL1W 13.02395581 W
SFO1 500.1321480 MHz
ND0 1
TD 128
SFO1 500.1321 MHz
FIDRES 31.001984 Hz
SW 7.934 ppm
FnMODE States-TPPI
SI 1024
SF 500.1300258 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.00
SI 1024
MC2 States-TPPI
SF 500.1300258 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0