Supplementary Material (ESI) for Chemical Communications

Supporting Informations

Magnetic Nanoparticle-Supported Hoveyda-Grubbs Catalysts for Ring-Closing Metathesis Reactions

Chao Che, *[a] Wenzhao Li, [a] Shengyue Lin, [a] Jiwei Chen, [a] Jie Zheng, [a] Jiun-chen Wu, [a] Qunxiong Zheng,[a] Guoqing Zhang, [a] Zhen Yang*[b] and Biwang Jiang*[a]

[a] Nano-micro Material Research Center, School of Chemical Biology and Biotechnology, Shenzhen Graduate School of Peking University, Shenzhen University Town, Shenzhen 518055, Tel: 86-755-26032537, Jiangbw@szpku.edu.cn and [b] Laboratory of Chemical Genomics, School of Chemical Biology and Biotechnology, Shenzhen Graduate School of Peking University, Shenzhen University Town, Shenzhen 518055, China, Tel: 86-755-26032971, zyang@pku.edu.cn

Table of contents

1. General methods

Unless noted otherwise, all oxygen and moisture-sensitive reactions were executed in flame-dried glasswares under a positive pressure of dry argon or nitrogen. The moisture sensitive solutions and anhydrous solvents were transferred *via* standard syringe or cannula. All commercial available reagents were used as received, and solvents were dried by standard methods under nitrogen atmosphere: THF and diethyl ether were distilled over Na/benzophenone; CH_2Cl_2, Et_3N and pyridine were distilled from CaH_2. Purification of products was conducted by flash column chromatography on silica gel (200-300 mesh) purchased from Qing Dao Hai Yang Chemical Industry Co. ^{1}H NMR spectra were recorded on a Bruker 500 MHz and 300 MHz spectrometer using residual solvent (δ ($CDCl_3$) = 7.26) as internal standard. All the coupling constants are reported in Hz. ^{13}C NMR spectra were recorded on the same instruments, and chemical shifts were measured relative to solvent resonances (δ ($CDCl_3$) = 77.0). High-resolution mass spectra were obtained on a qadrupole time-of-flight (QqTOF) mass spectrometer. The particle size and morphology of the samples were determined by transmission electronic microscopy (TEM, JEOL, JEM-200CX, 200 kV). Elemental analysis of ruthenium was determined by ICP-AES.

2. Synthesis of iron oxide magnetic nanoparticles

2-1: Synthesis of Iron–oleate complex

Magnetic nanoparticles were produced by the thermal decomposition. Typically, 5.4 g of iron chloride ($FeCl_3$•$6H_2O$, 20 mmol) and sodium oleate (18.2 g, 60 mmol) was dissolved in a mixture solvent composed of 40 mL ethanol, 30 mL distilled water and 70 mL hexane. The resulting solution was heated to 70 °C and kept stirring at that temperature for 4 hours. When the reaction was completed, the upper organic layer containing the iron–oleate complex was washed three times with deionized water in a separatory funnel. After washing, hexane was evaporated off, resulting in iron–oleate complex in a waxy solid form.

2-2: Synthesis of supermanetic iron oxide nanoparticles

The iron-oleate complex (36 g, 40 mmol) synthesized as described above and oleic acid (5.7 g, 20 mmol) were dissolved in 200 g of 1-octadecene at room temperature. The reaction mixture was heated to 320 °C with a constant heating rate of 3 °C/min, and then kept at that temperature for 30 min. When the reaction temperature reached 320 °C, a severe reaction occurred and the initial transparent solution became turbid and brownish black. The resulting solution containing the nanocrystals was then cooled to room temperature, and 500 mL solution of acetone/hexane (4/1, v/v) was added to the solution to precipitate the nanocrystals. The nanocrystals were separated by

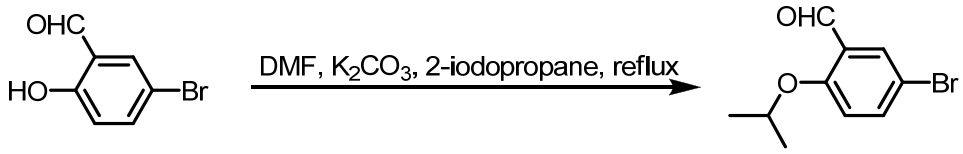
centrifugation and washed with acetone/hexane mixture to remove 1-octadecene.

3. Surface modification of iron oxide nanoparticles with APTS

Iron oxide magnetic nanoparticles (1.0 g) were refluxed with 3-aminopropyl- triethoxysilane (APTS, 4 mL, 55 mmol) in dry toluene (6.0 mL) for 3 days. The solids were separated by applying an external magnetic field and washed with dry toluene 4 times followed with dry acetone for 2 times. The resulting product was dried under vacuum.

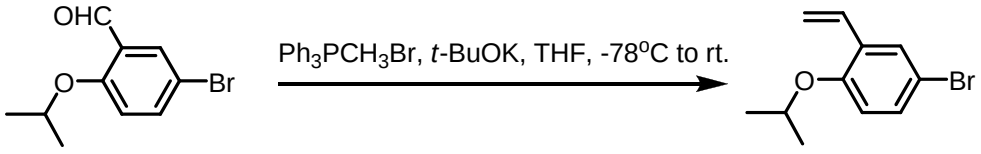
4. Synthesis of Hoveyda's *o*-isopropoxy styrene ligand

4-1: Synthesis of 5-bromo-2-isopropoxybenzaldehyde[1]



To a solution of 5-bromosalicylaldehyde (20 mmol, 4.02 g) in DMF (50 mL) was added K_2CO_3 (60 mmol, 8.28 g), leading to precipitation of a bright yellow solid. 2-Iodopropane (26mmol, 4.42 g) was then added dropwise to the mixture with a syringe. The mixture was refluxed for 3h, at which point TLC analysis showed complete consumption of the starting phenol. The reaction mixture was then diluted with 100 mL each of water and a saturated ammonium chloride solution and transferred to a separatory funnel. The aqueous layer was extracted three times with 400 mL of Et_2O. The combined organic layers were then washed three times with 200 mL saturated brine to remove residual DMF. The product solution was then dried over anhydrous sodium sulfate, filtered, and concentrated to a crude oil. Flash chromatography (*n*-hexane/Et_2O, 20/1) gave the desired product as colorless oil. (4.66 g, 96%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.39 (*s*, 1H), 7.91 (*d*, *J* = 2.7 Hz, 1H), 7.60 (*dd*, *J* = 2.7, 9.0 Hz, 1H), 6.88 (*d*, *J* = 9.0 Hz, 1H), 4.61-4,69 (*m*, 1H), 1.39 (*d*, *J* = 6.0 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 188.7, 159.4, 138.1, 130.9, 126.9, 115.9, 113.1, 71.6, 21.9; HRMS (*m/z*) calc. for $C_{10}H_{12}O_2Br(+)$ 240.9864, found 240.9848.

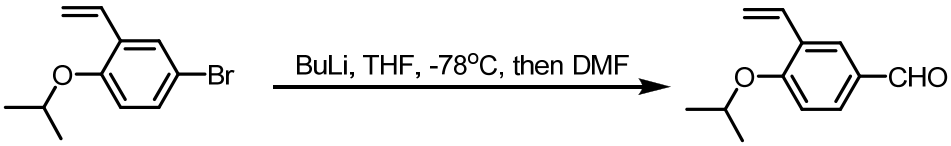
4-2: Synthesis of 4-bromo-1-isopropoxy-2-vinylbenzene[1]



A 250 mL round-bottom flask was charged with methyl triphenylphosphonium bromide (19.8 mmol, 7.07 g, 1.2 equiv) and THF (60 mL). To the resulting white suspension was added a solution of

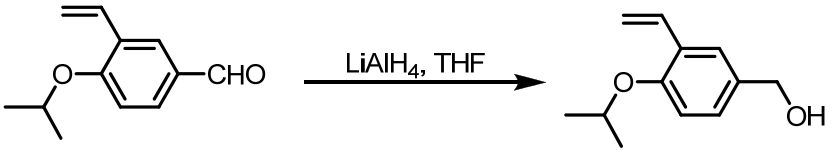
potassium *tert*-butoxide (19.8 mmol, 2.22g, 1.2equiv) in THF (50 mL) through a cannula. The reaction mixture truned yellow in color upon formation of the ylide. This mixture was stirred for 2 h at room temperature and then cooled to -78°C. A THF solution (50 mL) of 5-bromo-2-isopropoxybenzaldehyde(16.5 mmol, 4.0 g) was then added dropwise via cannula. The reaction mixture was warmed slowly to the room temperature and stirred for another 4 h. 100 mL of water were then added, and the aqueous layer was extracted three times with 250 mL of Et_2O. The organic layer was washed with 100 mL of saturated brine, dried over Na_2SO_4, filtered, and concentrated. The resulting crude oil was purified through flash chromatography (*n*-hexane/Et_2O, 40/1) to afford the desired product (3.42 g, 86%) as colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.57 (*d*, *J* = 2.4 Hz, 1H), 7.26 (*dd*, *J* = 2.4, 8.7 Hz, 1H), 6.97(*dd*, *J* = 11.1, 17.7 Hz, 1H), 6.76 (*d*, *J* = 8.7 Hz, 1H), 5.74 (*dd*, *J* = 0.9, 17.7 Hz, 1H), 5.52 (*dd*, *J* = 0.9, 11.1 Hz, 1H), 4.45-4,53 (*m*, 1H), 1.34 (*d*, *J* = 6.0 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 154.1 131.1, 130.7, 129.9, 129.1, 115.8, 115.2, 113.0, 71.2, 22.0; HRMS (*m/z*) calc. for $C_{11}H_{14}OBr(+)$ 241.0228, found 241.0219.

4-3: Synthesis of 4-isopropoxy-3-vinylbenzaldehyde[1]



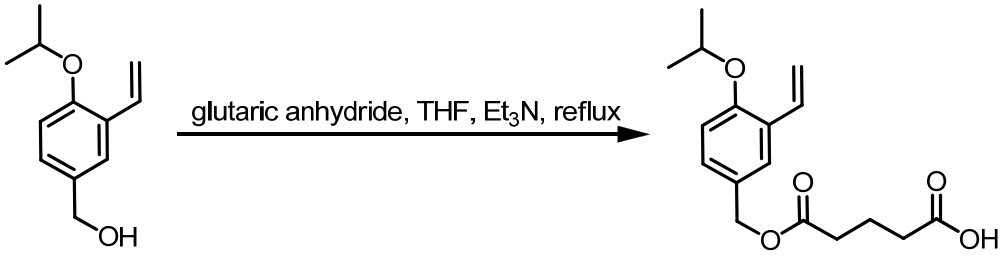
A 250 mL flame-dried flask was charged 80 mL of anhydrous THF and 4-bromo-1-isopropoxy-2-vinylbenzene (3.2 g, 13.3 mmol). When cooled to -78°C, the solution was added *n*-BuLi (2.86 M in hexanes, 14.6 mmol, 5.1 mL, 1.1 equiv) through a syringe. The resulting yellow solution was stirred at this temperature for 30 min, at which point DMF (16.0 mmol, 1.17 g, 1.2 equiv) was introduced dropwise from a syringe. After removing the cold bath, the reaction mixture was allowed to warm slowly to the room temperature and stirred for another 4 h. The reaction was cooled to 0°C and quenched with 10 mL of water. The mixture was transferred to a separatory funnel, diluted with 200 mL of Et_2O and washed twice with 100 mL of saturated brine. The organic layers were dried over Na_2SO_4, filtered, and concentrated to yellow oil. Purification by flash chromatography (*n*-hexane/Et_2O, 4/1) delivered the desired product as colorless oil (2.2 g, 87%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.88 (*s*, 1H), 8.0 (*s*, 1H), 7.76 (*d*, *J* = 8.7 Hz, 1H), 6.95-7.08 (*m*, 2H), 5.87 (*d*, *J* = 17.7 Hz, 1H), 5.35 (*d*, *J* = 11.4 Hz, 1H), 4.66-4,74 (*m*, 1H), 1.41 (*d*, *J* = 5.7 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.0, 160.1, 131.1, 130.8, 129.2, 128.4, 128.0, 115.7, 112.6, 70.9, 21.9; HRMS (*m/z*) calc. for $C_{12}H_{15}O_2(+)$ 191.1072, found 191.1070.

4-4: Synthesis of (4-isopropoxy-3-vinylphenyl)methanol[1]



To a solution of 4-isopropoxy-3-vinylbenzaldehyde (2.0 g, 10.5 mmol) in anhydrous THF (20 mL) was added $LiAlH_4$ (418 mg, 11 mmol) at 0°C. The mixture was then stirred at room temperature for 2 h, at which point TLC analysis showed the complete consumption of the starting aldehyde. Then, the reaction mixture was poured into a 250 ml beaker and diluted with Et_2O (50 mL), to which 10 drops of water was added slowly with stirring the mixture. The resulting white suspension was filtered through celite and rinsed with Et_2O. After removing the solvent, the residue was purified by flash chromatography (*n*-hexane/ethyl acetate, 4/1) to give the desired product (1.97 g, 98%) as colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 (*d*, *J* = 1.8 Hz, 1H), 7.18 (*dd*, *J* = 2.1, 8.4 Hz, 1H), 7.06 (*dd, J* = 11.4, 17.7 Hz, 1H), 6.85 (d, *J* = 8.4 Hz, 1H), 5.77 (*dd*, *J* = 1.5, 17.7 Hz, 1H), 5.35 (*dd*, *J* = 1.5, 11.4 Hz, 1H), 4.49-4,60 (*m*, 3H), 1.35 (*d*, *J* = 6.0 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 154.7, 132.9, 131.7, 127.9, 127.7, 125.6, 114.3, 114.2, 71.0, 65.1, 22.1; HRMS (*m/z*) calc. for $C_{12}H_{17}O_2$(+) 193.1229, found 193.1228.

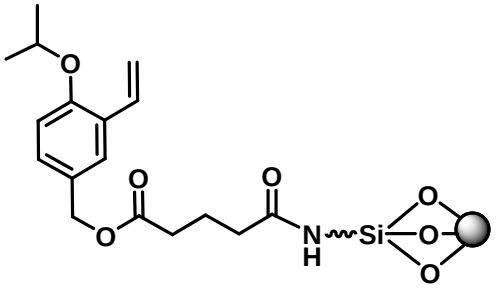
4-5: Synthesis of 5-(4-isopropoxy-3-vinylbenzyloxy)-5-oxopentanoic acid（1）[2]



A 100 mL round-bottom flask was charged 50 mL of anhydrous THF, (4-isopropoxy-3-vinylphenyl)methanol (1.80 g, 9.4 mmol) and glutaric anhydride (1.61 g, 14.1 mmol, 1.5 equiv). To the solution was added dropwise Et_3N (1.52 g, 15.04 mmol, 1.6equiv) via a syringe. The reaction mixture was then refluxed under nitrogen overnight. After cooling to room temperature, the mixture was diluted with 20 mL of water, and transferred to a separatory funnel. The aqueous layer was extracted three times with ethyl acetate (150 mL). The combined organic layers were dried over anhydrous Na_2SO_4. After removing the solvent, the residue was purified by flash chromatography (*n*-hexane/ethyl acetate, 2/1) to give the desired product (2.2 g, 76%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46 (*s*, 1H), 7.21 (*d*, *J* = 8.4 Hz, 1H), 7.04 (*dd, J* = 11.1, 17.7 Hz, 1H), 6.87 (d, *J* = 8.4 Hz, 1H), 5.78 (*d*, *J* = 17.7 Hz, 1H), 5.35 (*d*, *J* = 11.1 Hz, 1H), 5.05 (*s*, 2H), 4.50-4,58 (*m*, 1H), 2.43 (*t*, *J* = 7.2 Hz, 4H), 1.94-2.02 (m, 2H), 1.35 (*d*, *J* = 6.0 Hz, 6H); ^{13}C NMR (75 MHz,

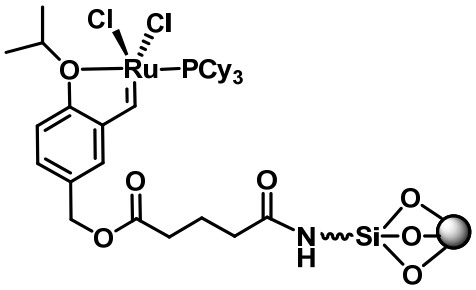
$CDCl_3$) δ 179.0, 172.7, 155.2, 131.5, 129.1, 127.8, 127.7, 127.1, 114.4, 113.9, 70.8, 66.2, 33.1, 32.9, 22.1, 19.7; HRMS (*m/z*) calc. for $C_{17}H_{22}O_5Na(+)$ 329.1365, found 329.1378.

5. Immobilization of Hoveyda-type ligand on the Fe_2O_3 nanoparticles (2)



To a solution of 5-(4-isopropoxy-3-vinylbenzyloxy)-5-oxopentanoic acid (0.40 g, 1.31 mmol) in 20 mL of DMF was added dicyclohexylcarbodimide (288 mg, 1.40 mmol), DMAP(50 mg) and TsOH (20 mg). This solution was stirred at room temperature for 1 h. Then, amino-functionalized Fe_2O_3 manetic nanoparticles (200 mg) were added to the first solution in one portion. The resulting solution mixture was stirred at 70°C for 48 h. After the reaction, the immobilized ligand was magnetically separated through using an external magnet. Typically, the magnetic nanoparticles were magnetized to one side of the flask by a magnet, and the reaction mixture was drawn out via a syringe. The 20 mL CH_2Cl_2 was then added and stirred for a minute. After the magnetic separation, the washing CH_2Cl_2 were drawn out. The above process was repeated another two times and the magnetic nanoparticle was dried under vacuum.

6. Preparation of magnetic Hoveyda-Grubbs catalyst (3).

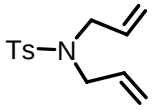


The magnetic nanoparticles (400 mg) prepared in the section **6**, and CuCl (20 mg, 0.20 mmol) was charged to a 25 mL tube-flask. In the glove box, $RuCl_2$-(=CHPh)$(PCy_3)_2$ (170 mg, 0.2 mmol) was weighed to the above tube flask, and the flask was vacuumed and charged with N_2 for three times. Then, 15 mL of CH_2Cl_2 was added and the reaction mixture was stirred at 40°C for 12 h before magnetic separation. Typically, the magnetic nanoparticle catalysts were magnetized to one side of the flask by a magnet, and the reaction mixture was drawn out via a syringe. The 5 mL CH_2Cl_2 was then added and stirred for a minute. After the magnetic separation, the washing CH_2Cl_2 were drawn

out. The above process was repeated another two times and the magnetic nanoparticle catalyst was dried under vacuum.

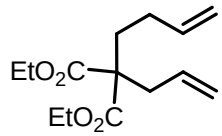
7. Synthesis of substrates for ring-closing metathesis

7-1: Synthesis of N,N-diallyl-4-methylbenzenesulfonamide (4)[3]



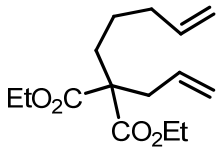
Triethylamine (1.09 g, 10.68 mmol) and tosyl chloride (2.0 g, 10.48 mmol) were added to a solution of diallylamine (1.0 g, 10.29 mmol) in 35 mL of CH_2Cl_2. The mixture was stirred at 20°C overnight. The organic phase was washed twice by a 10% aqueous solution of $KHSO_4$ (30 mL) and twice by a saturated aqueous solution of $NaHCO_3$ (30 mL), then by water (30 mL). The organic phase was dried over Na_2SO_4, concentrated under vacuum and purified through flash chromatography to give the product (95%) as a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72 (*d*, *J* = 8.1 Hz, 2 H), 7.28 (*d*, *J* = 8.1 Hz, 2 H), 5.54-5.68 (*m*, 2 H), 5.16 (*dd*, *J* = 1.2, 13.2 Hz, 4 H), 3.80 (*d*, *J* = 6.0 Hz, 4 H), 2.43 (*s*, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.2, 137.4, 132.6, 129.6, 127.2, 118.9, 49.3, 21.5; HRMS (*m/z*) calc. for $C_{13}H_{17}NO_2NaS(+)$ 274.0878, found 274.0871.

7-2: Synthesis of diethyl 2-allyl-2-(but-3-enyl)malonate (9)[4]



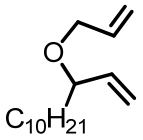
Alkylation of allylmalonate was carried out according to the previously reported procedure. Sodium metal (0.23 g, 10 mmol) was added to absolute ethanol (10 mL). When the sodium had completely reacted, diethyl allylmalonate (2.0 g, 10 mmol) was slowly added, and then 4-bromo-1-butene (1.62 g, 12 mmol) was added dropwise and the reaction mixture was refluxed for 3 h. The mixture was then acidified with glacial acetic acid, filtered and concentrated under reduced pressure. The resulting residue was purified by flash chromatography to give the desired product (1.82 g, 72%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.59-5.78 (*m*, 2 H), 4.93-5.11 (*m*, 4 H), 4.16 (*q*, *J* = 7.0 Hz, 4 H), 2.65 (*d*, *J* = 7.5 Hz, 2 H), 1.95 (*s*, 4 H), 1.22 (*t*, *J* = 7.0 Hz, 6 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.1, 137.5, 132.3, 118.9, 114.9, 61.1, 57.1, 36.9, 31.4, 28.2, 14.1; HRMS (*m/z*) calc. for $C_{14}H_{22}O_4Na(+)$ 277.1416, found 277.1423.

7-3: Synthesis of diethyl 2-allyl-2-(pent-4-enyl)malonate (11)[4]



The compound was prepared in a manner similar to that of **7-2** using 5-bromopent-1-ene. The compound was obtained in 78% yield as colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.58-5.77 (*m*, 2 H), 4.93-5.09 (*m*, 4 H), 4.16 (*q*, *J* = 7.0 Hz, 4 H), 2.63 (*d*, *J* = 7.5 Hz, 2 H), 2.01-2.05 (*m*, 2 H), 1.84-1.87 (*m*, 2 H), 1.21-1.32 (*m*, 8 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.3, 138.2, 132.7, 118.8, 115.0, 61.2, 57.4, 37.1, 33.8, 31.8, 23.3, 14.2; HRMS (*m/z*) calc. for $C_{15}H_{24}O_4Na$(+) 291.1572, found 291.1578.

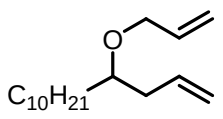
7-4: Synthesis of 3-(allyloxy)tridec-1-ene (6)



Undecanal (8.50 g, 50 mmol) was dissolved under N_2 in dry THF (250 mL), cooled to 0^{o}C and treated with vinylmagnesium bromide (1M solution in THF, 60 mL, 60 mmol). The mixture was stirred for 2 h at 0^{o}C and worked up. After removal of all volatiles in vacuo, the residue was chromatographed on silica gel (*n*-hexanes/EtOAc, 5/1) to yield carbinols.

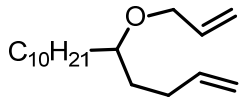
A 50% suspension of NaH in mineral oil (2.4 g, equivalent to ca. 50 mmol of active hydride) was washed three times under N_2 with dry hexane. Dry THF (30 mL) was then added, followed by a solution of the above carbinol (1.98 g, 10 mmol) in dry THF (20 mL). The solution was stirred at room temperature for 30 min. Allyl bromide (1.80 g, 15 mmol) was then added dropwise, followed by tetrabutyl ammonium bromide (370 mg, 1.0 mmol). The reaction mixture was then heated overnight at reflux. Work-up and column chromatography on silica gel (*n*-hexanes/EtOAc, 30/1) furnished the expected ethers (1.61 g, 68%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.84-5.92 (*m*, 1H), 5.61-5.68 (*m*, 1H), 5.11-5.25 (*m*, 4H), 4.0 (*dd*, *J* = 5.5, 12.5 Hz, 1H), 3.80 (*dd*, *J* = 6.0, 12.5 Hz, 1H), 3.62-3.66 (*m*, 1H), 1.44-1.61 (*m*, 2H), 1.23 (*s*, 16H), 0.87 (*t*, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 139.4, 135.4, 116.7, 116.5, 80.8, 69.2, 35.6, 32.0, 29.7, 29.6, 29.4, 25.4, 22.8, 14.1; HRMS (*m/z*) calc. for $C_{16}H_{31}O$(+) 239.2375, found 239.2368.

7-5: Synthesis of 4-(allyloxy)tetradec-1-ene (7)



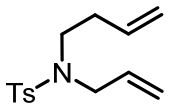
The compound was prepared in a manner similar to that of **7-4** using allylmagnesium bromide which was generated from allyl bromide by means of the standard procedure. The compound was obtained as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.79-5.95 (*m*, 2H), 5.03-5.28 (*m*, 4H), 3.95-4.05 (*m*, 2H), 3.32-3.37 (*m*, 1H), 2.27 (*dd*, *J* = 5.6, 11.1 Hz, 2H), 1.45-1.49 (*m*, 2H), 1.26 (*s*, 16H), 0.87 (*t*, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 135.7, 135.3, 116.7, 116.3, 78.7, 70.1, 38.5, 33.9, 32.0, 29.8, 29.7, 29.4, 25.4, 22.7, 14.1; HRMS (*m/z*) calc. for $C_{17}H_{33}O(+)$ 253.2531, found 253.2513.

7-6：Synthesis of 5-(allyloxy)pentadec-1-ene (12)



The compound was prepared in a manner similar to that of **7-4** using homoallylmagnesium bromide which was generated from 4-bromo-1-butene by means of the standard procedure. The compound was obtained as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.77-5.90 (*m*, 2H), 5.22 (*d*, *J* = 17.0 Hz, 1H), 5.12 (*d*, *J* = 10.5 Hz, 1H), 5.01 (*d*, *J* = 17.0 Hz, 1H), 4.91 (*d*, *J* = 10.5 Hz, 1H), 3.94 (*t*, *J* = 5.0 Hz, 2H), 3.25-3.31 (*m*, 1H), 2.08-2.18 (*m*, 2H), 1.41-1.57 (*m*, 4H), 1.26 (*s*, 16H), 0.87 (*t*, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 138.9, 135.8, 116.3,114.4, 78.6, 70.0, 34.0, 33.3, 32.0, 29.9, 29.8 29.7, 29.4, 25.4, 22.7, 20.2, 14.1; HRMS (*m/z*) calc. for $C_{18}H_{35}O(+)$ 267.2688, found 267.2679.

7-7: Synthesis of N-allyl-N-(but-3-enyl)-4-methylbenzenesulfonamide (8)[5]

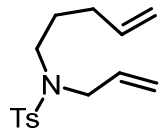


Triethylamine (1.21 g, 12 mmol) and tosyl chloride (2.3 g, 12 mmol) were added to a solution of allylamine (0.68 g, 12 mmol) in 40 mL of CH_2Cl_2. The mixture was stirred at room temperature for 5 h. After the reaction, the reaction mixture was diluted with 100 mL Et_2O, transferred to the separatory funnel, and washed with water and saturated brine. The organic layer was dried over Na_2SO_4 and concentrated. The resulting residue was purified by column chromatography (*n*-hexane/ EtOAc, 30/1) to give the *N*-allyl-*p*-toluenesulfonamide.

To a solution of *N*-allyl-*p*-toluenesulfonamide (211 mg, 1.0 mmol) and K_2CO_3 (414 mg, 3 mmol) in CH_3CN (12 mL) was added 4-bromo-1-butene (243 mg, 1.8 mmol), and the mixture was refluxed for 8 hours. The solution was filtrated through a celite pad and the filtrate was concentrated under reduced pressure. The crude residue was subjected to column chromatography (*n*-hexanes/EtOAc, 10/1) on silica gel to give the desired product (222 mg, 84%) as a colorless oil.

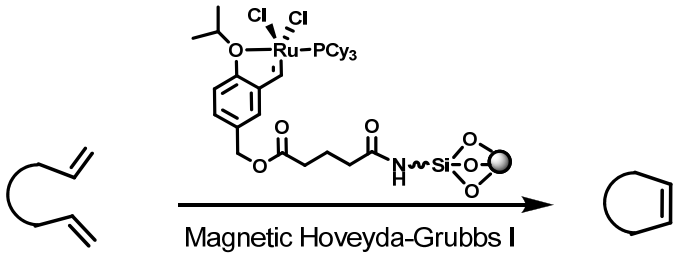
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.68 (*d*, *J* = 7.9 Hz, 2 H), 7.28 (*d*, *J* = 7.9 Hz, 2 H), 5.58-5.72 (*m*, 2 H), 4.99-5.18 (*m*, 4 H), 3.79 (*d*, *J* = 6.2 Hz, 2 H), 3.17 (*t*, *J* = 7.4 Hz, 2 H), 2.41 (*s*, 3 H), 2.25 (*q*, *J* = 7.0 Hz, 2 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.2, 137.4, 134.8, 133.4, 129.7, 127.3, 118.7, 117.0, 50.8, 46.8, 32.9, 21.5; HRMS (*m/z*) calc. for $C_{14}H_{19}NO_2NaS(+)$ 288.1034, found 288.1026.

7-8: Synthesis of N-allyl-N-(pent-4-enyl)-4-methylbenzenesulfonamide (10)[6]



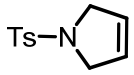
The compound was prepared in a manner similar to that of **7-7** using 5-bromopent-1-ene. The compound was obtained as a colorless oil in 82% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.67 (*d*, *J* = 8.2 Hz, 2 H), 7.27 (*d*, *J* = 8.2 Hz, 2 H), 5.59-5.77 (*m*, 2 H), 4.94-5.17 (*m*, 4 H), 3.78 (*d*, *J* = 6.4 Hz, 2 H), 3.10 (*t*, *J* = 7.5 Hz, 2 H), 2.41 (*s*, 3 H), 1.99-2.04 (*m*, 2 H), 1.58-1.64 (*m*, 2 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.2, 137.6, 137.4, 133.5, 129.7, 127.3, 118.7, 115.2, 50.8, 47.0, 30.8, 27.5, 21.5; HRMS (*m/z*) calc. for $C_{15}H_{22}NO_2S(+)$ 280.1371, found 280.1380.

8. General methods for ring-closing metathesis reaction catalyzed by magnetic Hoveyda-Grubbs catalyst



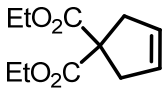
To a tube flask containing magnetic nanoparticles supported Hoveyda-Grubbs I catalyst (500 mg) was added a solution of diene (0.5 mmol) in the CH_2Cl_2 (5 mL) via a syringe. The reaction mixture was stirred at 40^{o}C for 1-12 h. After cooling to the room temperature, the reaction mixture was magnetically separated by an external magnet. The magnetic catalyst was kept in the flask, and the solution was drawn out through a syringe. The flask was then allowed to be washed with CH_2Cl_2 for twice, and the combined solution was concentrated under vacuum to give the pure RCM product. The second run of the RCM using the recycled catalyst was conducted in exactly the same way as described for the first cycle. This reaction was repeated for eight more cycles, each time using the catalyst from previous cycle.

8-1: Spectra data for 1-Tosyl-2,5-dihydro-1*H*-pyrrole (4p)[3]



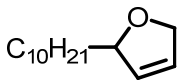
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.71 (*d*, *J* = 8.1 Hz, 2H), 7.30 (*d*, *J* = 8.1 Hz, 2H), 5.65 (*s*, 2H), 4.13 (*d*, *J* = 4.5 Hz, 4H), 2.42 (*s*, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.4, 134.2, 129.7, 127.4, 125.4, 54.8, 21.5; HRMS (*m/z*) calc. for $C_{11}H_{13}NO_2NaS$ (+) 246.0565, found 246.0565.

8-2: Spectra data for Diethyl cyclopent-3-ene-1,1-dicarboxylate (5p)[7]



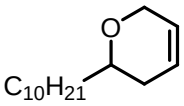
^{1}H NMR (300 MHz, $CDCl_3$) δ 5.61 (*s*, 2H), 4.19 (*q*, *J* = 7.2 Hz, 4H), 3.02 (*s*, 4H), 1.26 (*t*, *J* = 7.2 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ172.2, 127.8, 61.5, 58.8, 40.8, 14.0; HRMS (*m/z*) calc. for $C_{11}H_{16}O_4Na$(+) 235.0946, found 235.0954.

8-3: Spectra data for 2-Decyl-2,5-dihydrofuran (6p)



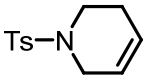
^{1}H NMR (300 MHz, $CDCl_3$) δ 5.77-5.88 (*m*, 2H), 4.79-4.83 (*m*, 1H), 4.59-4.65 (*m*, 2H), 1.50-1.55 (*m*, 2H), 1.26 (*s*, 16H), 0.87 (*t*, *J* = 6.9 Hz, 3H) ; ^{13}C NMR (75 MHz, $CDCl_3$) δ 129.8, 126.2, 86.1, 74.9, 36.0, 31.9, 30.9, 29.7, 29.6, 29.3, 25.2, 22.6, 14.1; HRMS (*m/z*) calc. for $C_{14}H_{27}O$(+) 211.2062, found 211.2047.

8-4: Spectra data for 2-Decyl-3,6-dihydro-2H-pyran (7p)



^{1}H NMR (300 MHz, $CDCl_3$) δ 5.72-5.79 (*m*, 2H), 4.18 (*s*, 2H), 3.48 (*m*, 1H), 1.99 (2H), 1.26-1.44 (*m*, 18H), 0.87 (*t*, *J* = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 126.3, 124.3, 73.7, 65.9, 36.0, 31.9, 31.0, 29.7, 29.6, 29.3, 25.4, 22.6, 14.1; HRMS (*m/z*) calc. for $C_{15}H_{29}O$(+) 225.2218, found 225.2223.

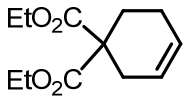
8-5: Spectra data for 1-Tosyl-1,2,3,6-tetrahydropyridine (8p)[5]



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 (*d*, *J* = 8.2 Hz, 2H), 7.31 (*d*, *J* = 8.2 Hz, 2H), 5.74-5.77 (*m*, 1H),

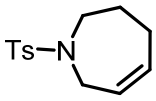
5.60-5.63 (*m*, 1H), 3.58 (*t, J* = 2.7 Hz, 2H), 3.18 (*t*, *J* = 5.7 Hz, 2H), 2.43 (*s*, 3H), 2.21 (*t*, *J* = 5.7 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.5, 133.9, 129.7, 127.8, 125.2, 122.9, 44.8, 42.7, 25.4, 21.5; HRMS (*m/z*) calc. for $C_{12}H_{15}NO_2NaS(+)$ 260.0721, found 260.0707.

8-6: Spectra data for Diethyl cyclohex-3-ene-1,1-dicarboxylate (9p)[4]



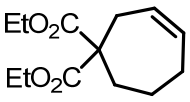
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.67 (*s*, 2H), 4.16-4.21 (*m*, 4H), 2.56 (*s*, 2H), 2.10-2.15 (*m*, 3H), 1.26 (*t*, *J* = 7.1 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.7, 126.2, 124.1, 61.3, 53.1, 30.5, 27.5, 22.4, 14.1; HRMS (*m/z*) calc. for $C_{12}H_{18}O_4Na(+)$ 249.1103, found 249.1105.

8-7: Spectra data for 1-Tosyl-2,3,4,7-tetrahydro-1*H*-azepine (10p)[6]



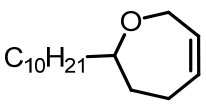
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.68 (*d*, *J* = 8.2 Hz, 2H), 7.28 (*d*, *J* = 8.2 Hz, 2H), 5.73-5.78 (*m*, 1H), 5.62-5.66 (*m*, 1H), 3.83 (*d, J* = 3.9 Hz, 2H), 3.40 (*t*, *J* = 6.0 Hz, 2H), 2.42 (*s*, 3H), 2.15-2.20 (*m*, 2H), 1.75-1.83 (*m*, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.1, 136.6, 132.9, 129.6, 127.3, 126.7, 49.7, 46.5, 29.8, 26.9, 21.5; HRMS (*m/z*) calc. for $C_{13}H_{17}NO_2NaS(+)$ 274.0878, found 274.0878.

8-8: Spectra data for Diethyl cyclohept-3-ene-1,1-dicarboxylate (11p)[4]



^{1}H NMR (300 MHz, $CDCl_3$) δ 5.63-5.87 (*m*, 2H), 4.16 (*q*, *J* = 6.9 Hz, 4H), 2.64 (*d*, *J* = 6.0 Hz, 2H), 2.11-2.25 (*m*, 4H), 1.61-1.65(*m*, 2H), 1.22 (*t*, *J* = 6.9 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.7, 134.0, 127.1, 61.1, 56.1, 36.6, 32.3, 28.3, 22.7, 14.0; HRMS (*m/z*) calc. for $C_{13}H_{20}O_4Na(+)$ 263.1259, found 263.1266.

8-9: Spectra data for 2-Decyl-2,3,4,7-tetrahydrooxepine (12p)



^{1}H NMR (300 MHz, $CDCl_3$) δ 5.63-5.88 (*m*, 2H), 4.24-4.31(*m*, 1H), 3.99-4.06 (*m*, 1H), 3.51-4.58 (*m*, 1H), 2.12-2.36 (*m*, 2H), 1.85-1.92 (*m*, 1H), 1.53-1.60 (*m*, 1H), 1.26 (*s*, 18H), 0.88 (*t*, *J* = 6.9 Hz, 3H);

^{13}C NMR (75 MHz, $CDCl_3$) δ 131.9, 130.0, 81.2, 66.8, 36.1, 34.0, 31.9, 29.7, 29.6, 29.3, 26.1, 25.8, 22.7, 14.1; HRMS (*m/z*) calc. for $C_{16}H_{31}O(+)$ 239.2375, found 239.2364.

9. Characterization of magnetic nanoparticles

9-1: Magnetic measurements

Magnetic characteristics were measured on Magnetic Properties Measurement System-5 (MPMS, Quantum Design) at the College of Chemistry and Molecular Engineering, Peking University. The following quantities were measured: saturated induced magnetization (in a field of 7 T) in the temperature range 5–300K (cooling–heating run), remanent magnetization from 5 to 300K after previous cooling in zero field (ZFC), magnetic susceptibility (as a complex quantity, its in-phase and out of phase component) from 5 to 300K at the field intensity 4 Oe (320 A/m) and frequencies 0.1, 1, 10, 100, 500 and 1000 Hz, hysteresis loops in the range -1 to +1 T at 5, 100, 150 and 300 K. All the quantities were measured in the same sample.

9-2: Transmission electron microscopy (TEM)

TEM analyses were conducted on a Hitachi 800 transmission electron microscope at an acceleration of voltage of 200 kV. The sample for TEM analysis was prepared by placing dispersed particle solutions on copper grids coated with thin films of carbon successively.

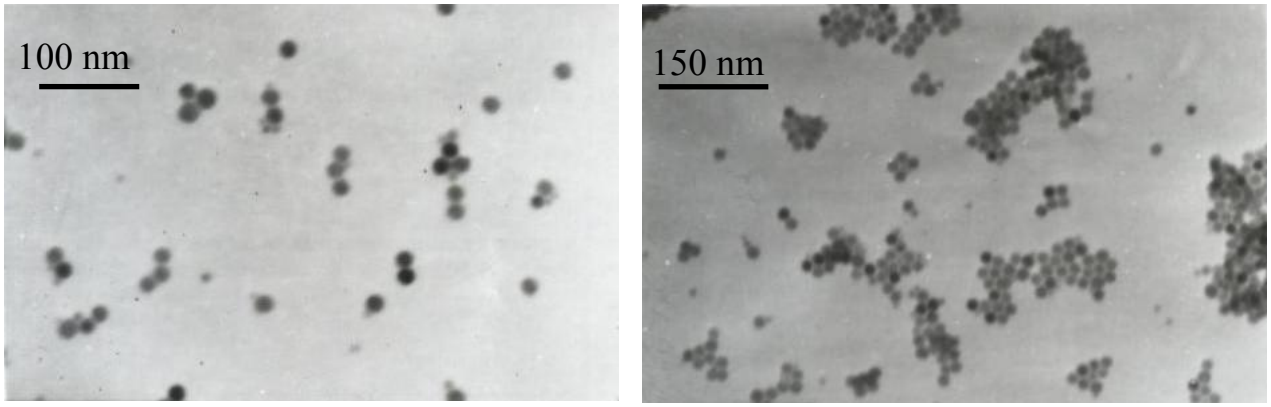


Figure 1 TEM micrographs of iron oxide nanoparticles

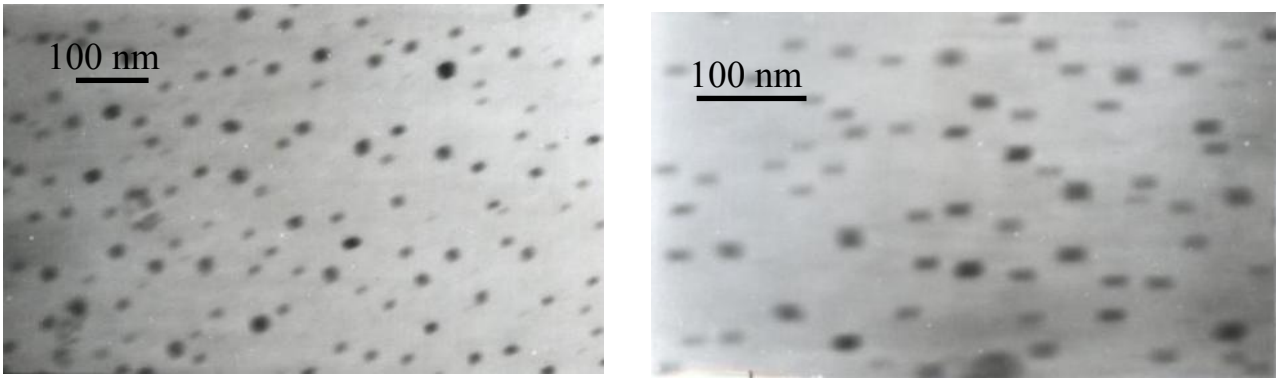


Figure 2 TEM micrographs of iron oxide nanoparticle supported Hoveyda-Grubbs Catalyst

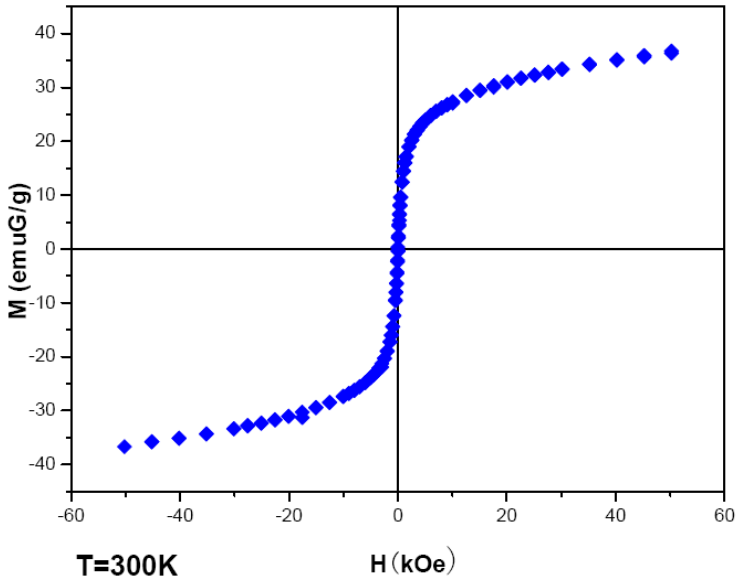


Figure 3 Magnetization curves of magentic nanoparticle supported Hoveyda-Grubbs Catalyst

Reference:

1. J. S. Kingsbury, S. B. Garber, J. M. Giftos, B. L. Gray, M. M. Okamoto, R. A. Farrer, J. T. Fourkas and A. H. Hoveyda, *Angew. Chem. Int. Ed.*, 2001, **40**, 4251.

2. Q.Yao and A. R. Motta, *Tetrahedron Lett.*, 2004, ***45***, 2447

3. S. Varray, R. Lazaro, J. Martinez and F. Lamaty, *Organometallics*, 2003, **22**, 2426.

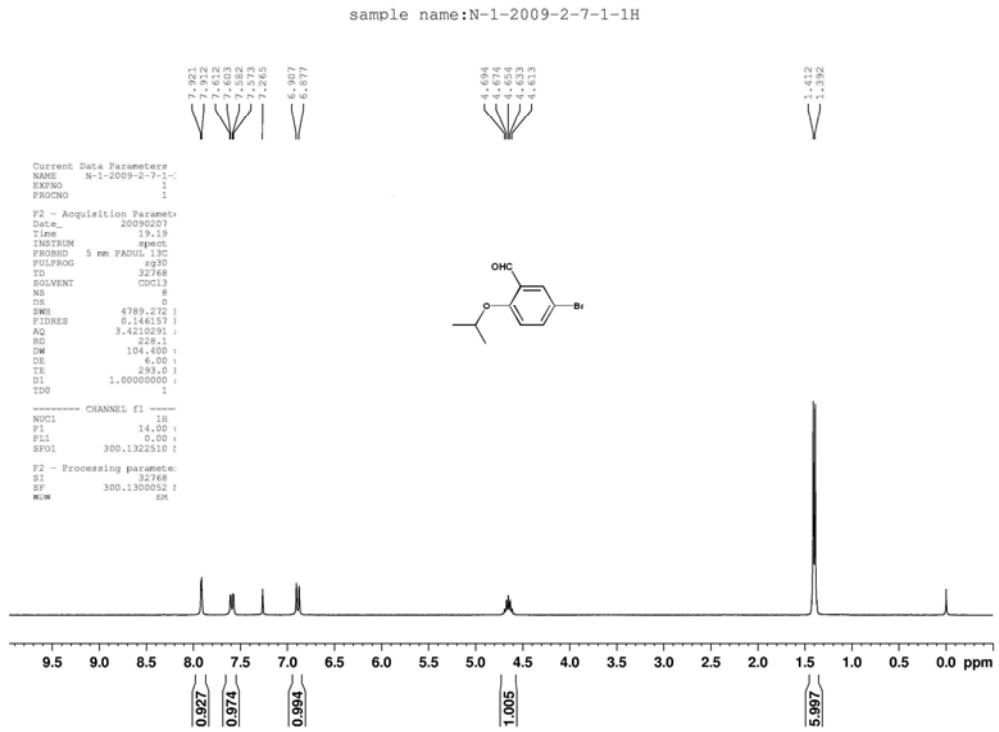
4. P. E. Romero, W. E. Piers and R. McDonald, *Angew. Chem. Int. Ed.*, 2004, **43**, 6161.

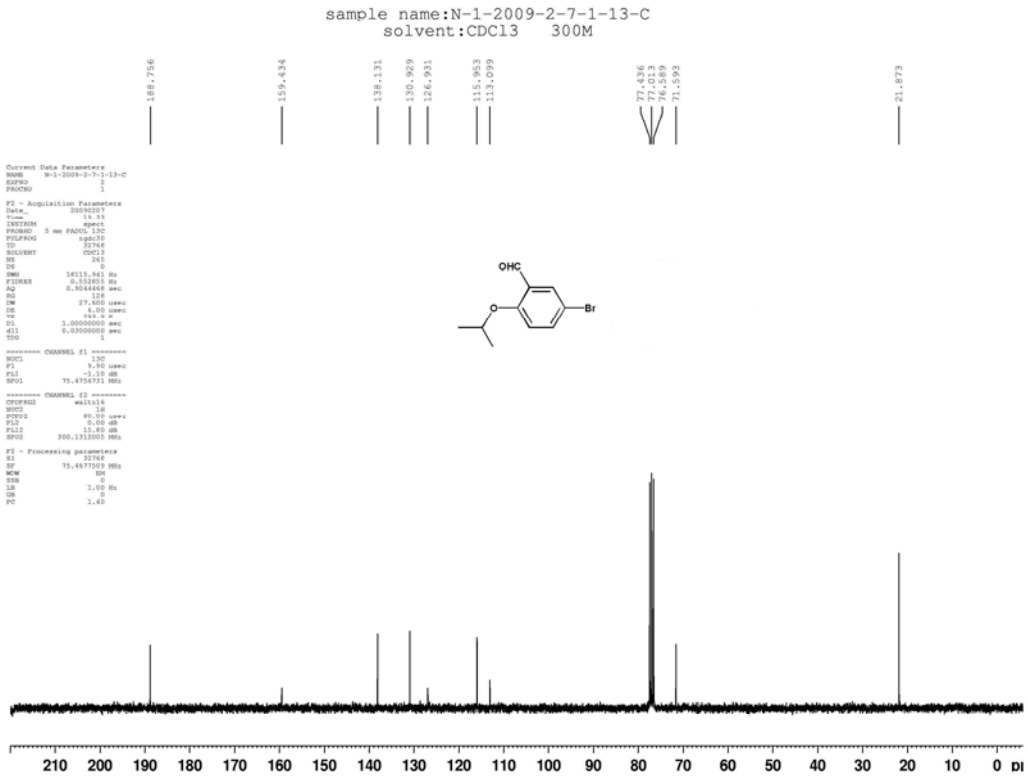
5. R. Akiyama and S. Kobayashi, *Angew. Chem. Int. Ed.*,2002, **41**, 2602.

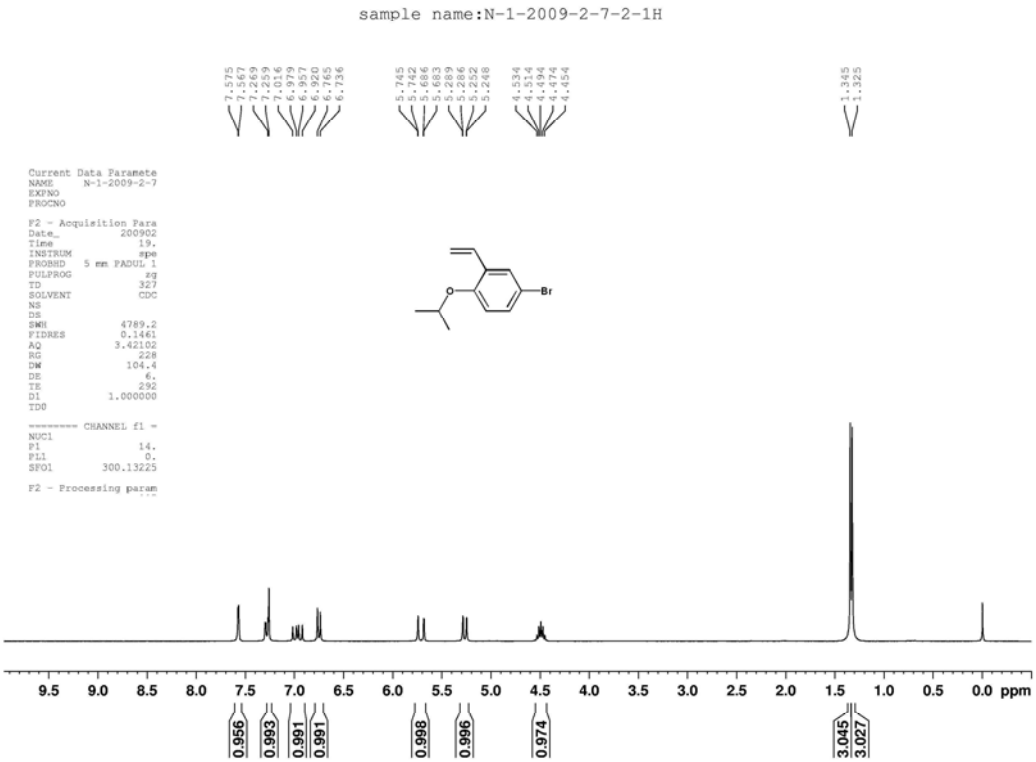
6. J. S. Kingsbury, J. P. A. Harrity, P. J. Bonitatebus and A. H. Hoveyda, *J. Am. Chem. Soc.*, 1999, **121**, 791.

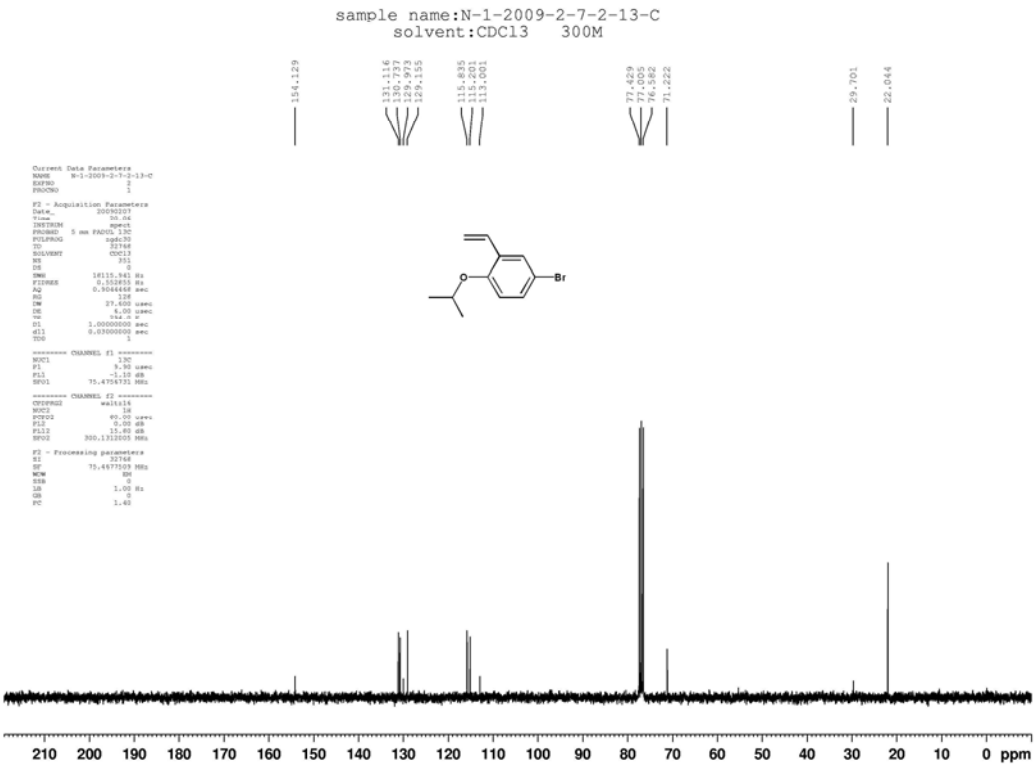
7. W. A. Nugent, J. Feldman and J. C. Calabrese, *J. Am. Chem. Soc.*,1995, **117**, 8992.

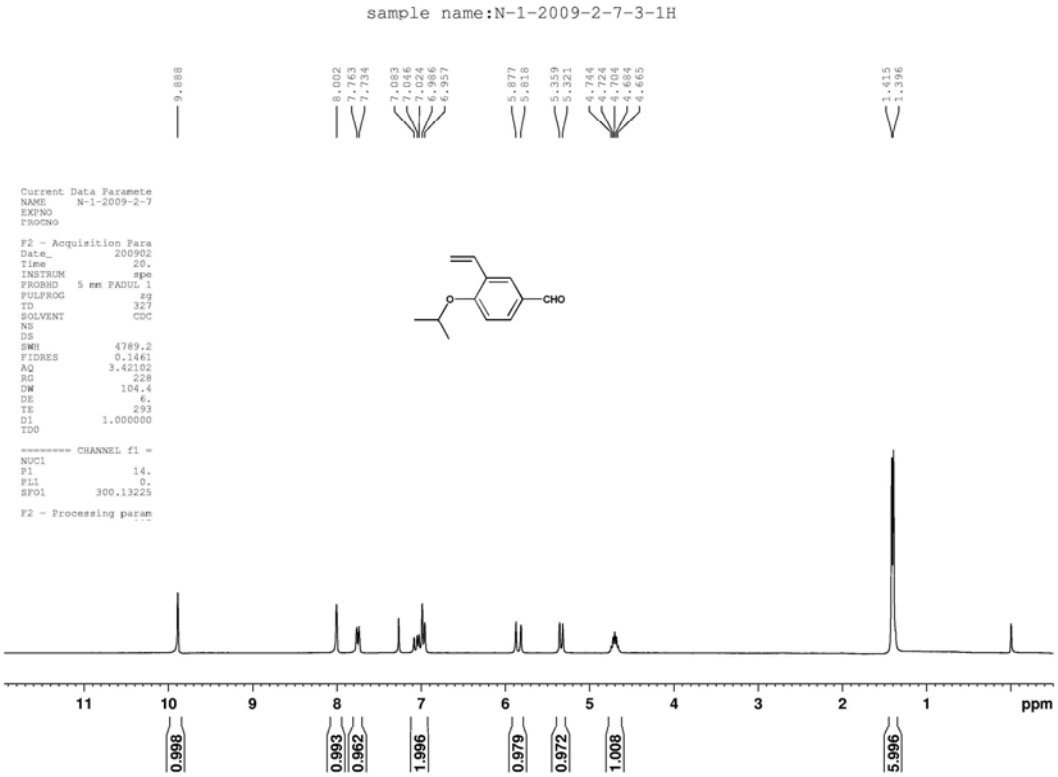
10. ^{1}HNMR and ^{13}CNMR Spectra:

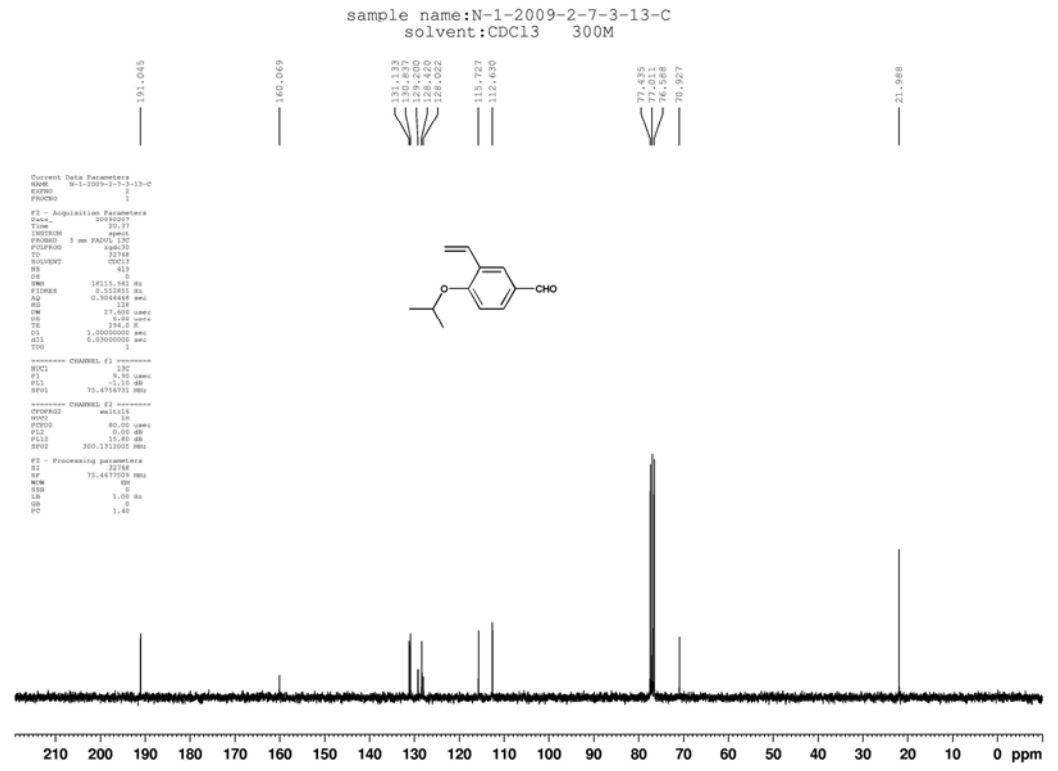


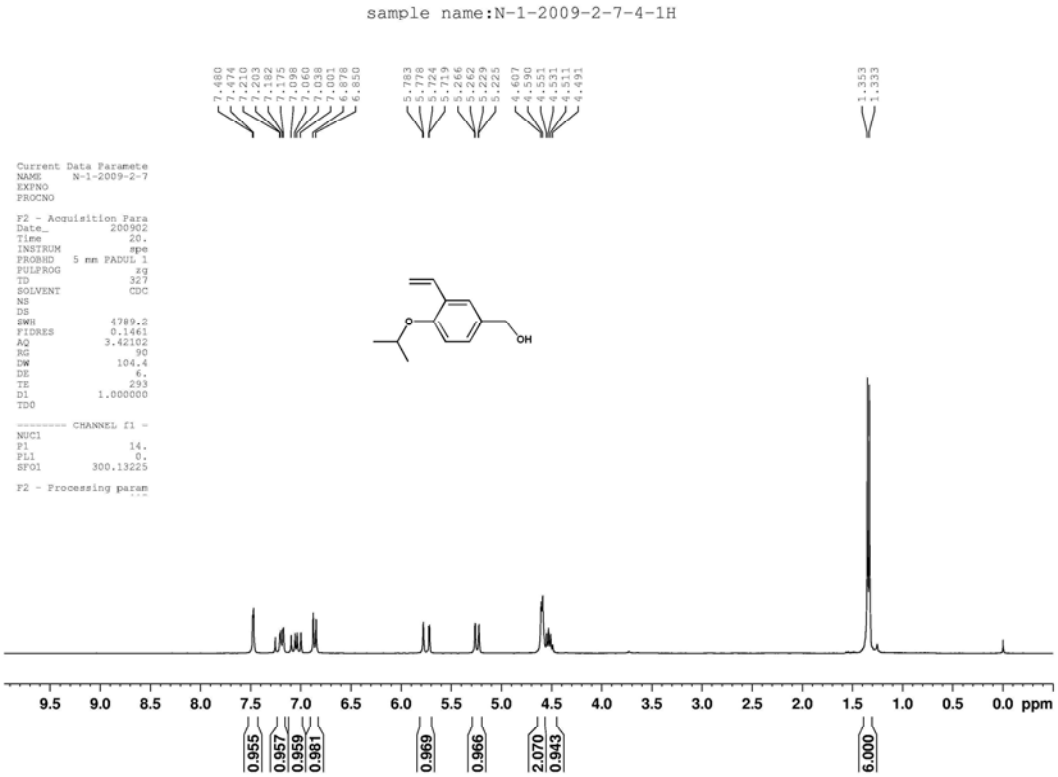


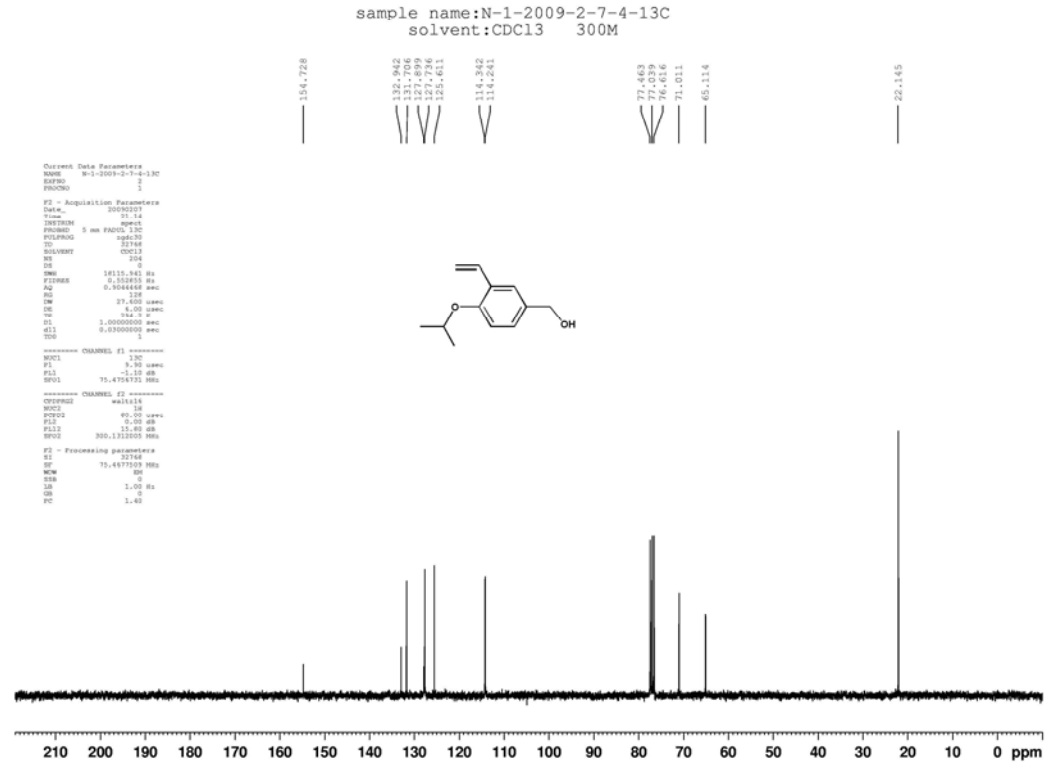

sample name:N-1-2009-2-7-2-1H
7.575
7.567
7.269
7.259
7.016
6.979
6.957
6.920
6.765
6.736
5.745
5.742
5.686
5.683
5.289
5.286
5.252
5.248
4.534
4.514
4.494
4.474
4.454
1.345
1.325
Br
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.956
0.993
0.991
0.991
0.998
0.996
0.974
3.045
3.027

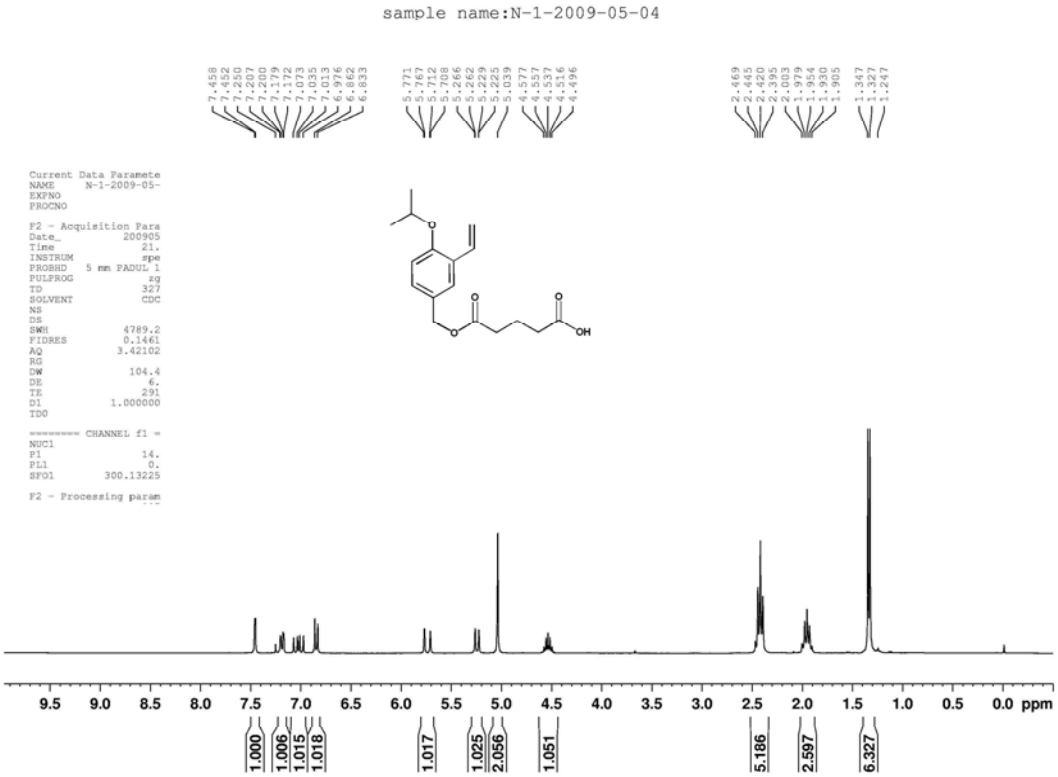

sample name:N-1-2009-2-7-2-13-C
solvent:CDCl3 300M
154.129
131.116
130.737
129.973
129.155
115.835
115.201
113.001
77.429
77.005
76.582
71.222
29.701
22.044
Br
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

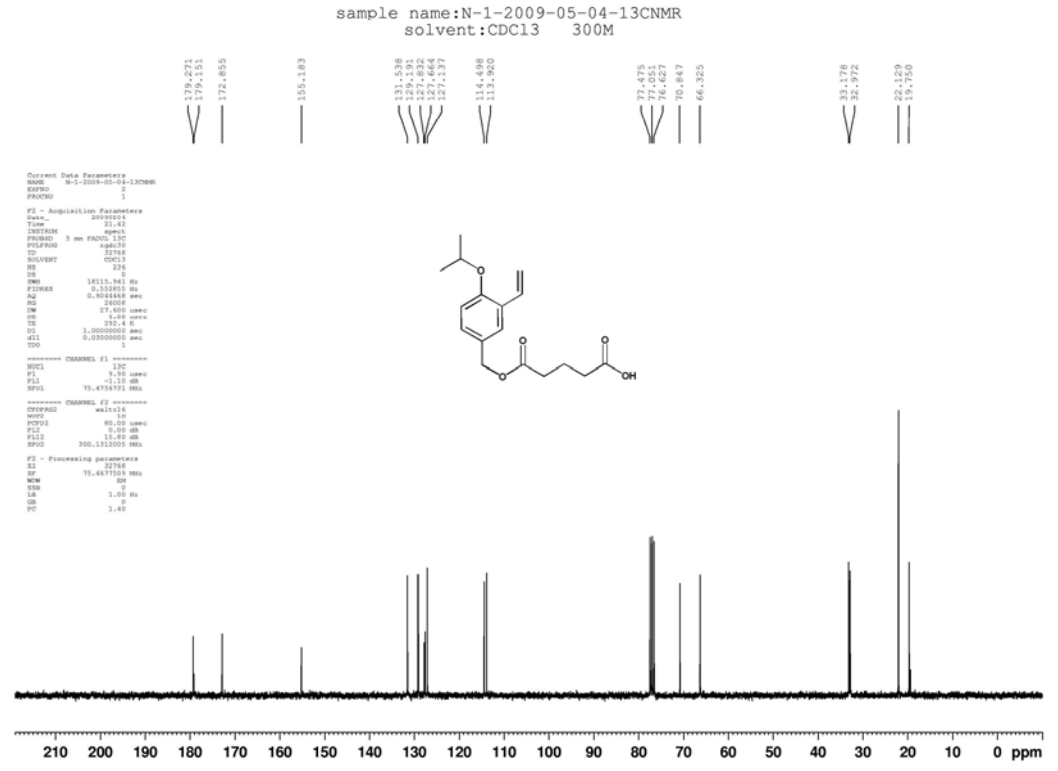

sample name:N-1-2009-2-7-3-1H
9.888
8.002
7.763
7.734
7.083
7.046
7.024
6.986
6.957
5.877
5.818
5.359
5.321
4.744
4.724
4.704
4.684
4.665
1.415
1.396
CHO
11
10
9
8
7
6
5
4
3
2
1
ppm
0.998
0.993
0.962
1.996
0.979
0.972
1.008
5.996

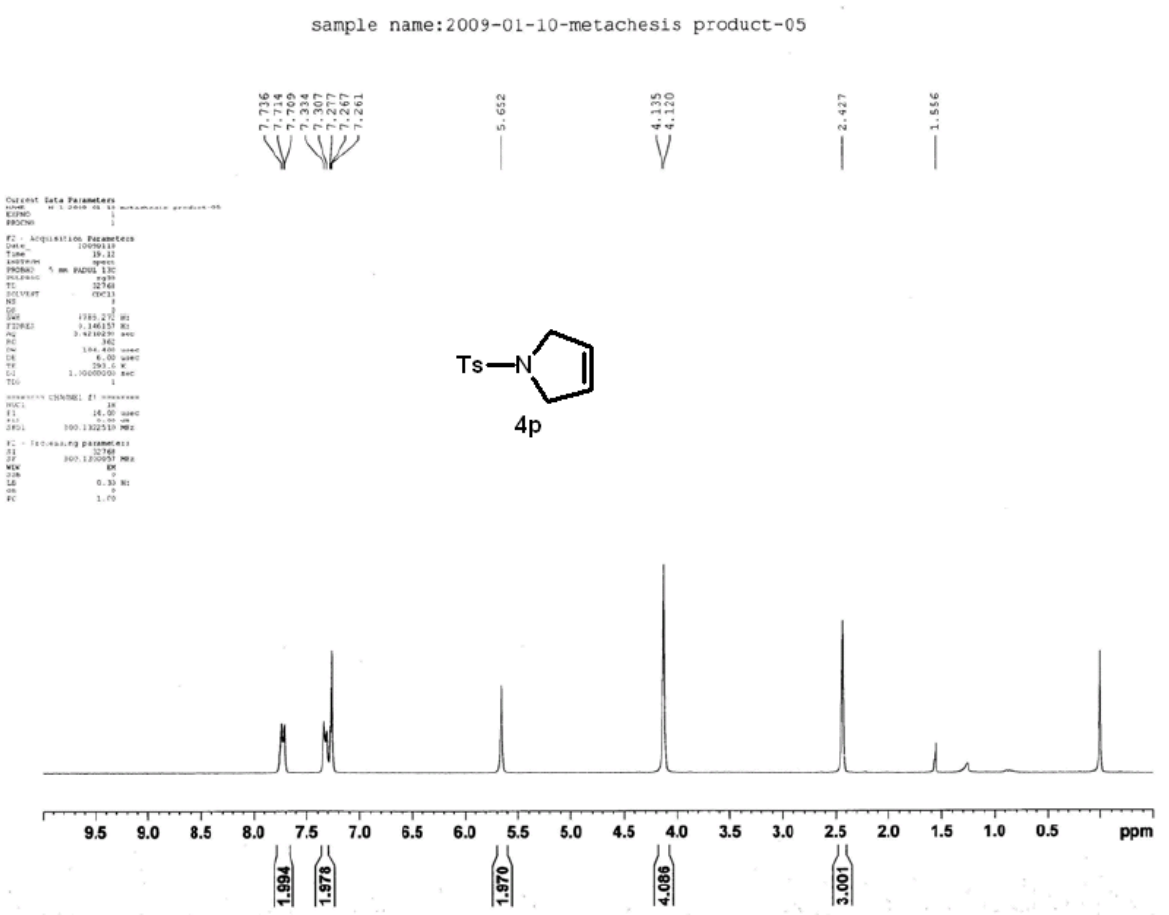

sample name:N-1-2009-2-7-3-13-C
solvent:CDCl3 300M
191.045
160.069
131.133
130.837
129.200
128.420
128.022
115.727
112.630
77.435
77.011
76.588
70.927
21.988
CHO
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

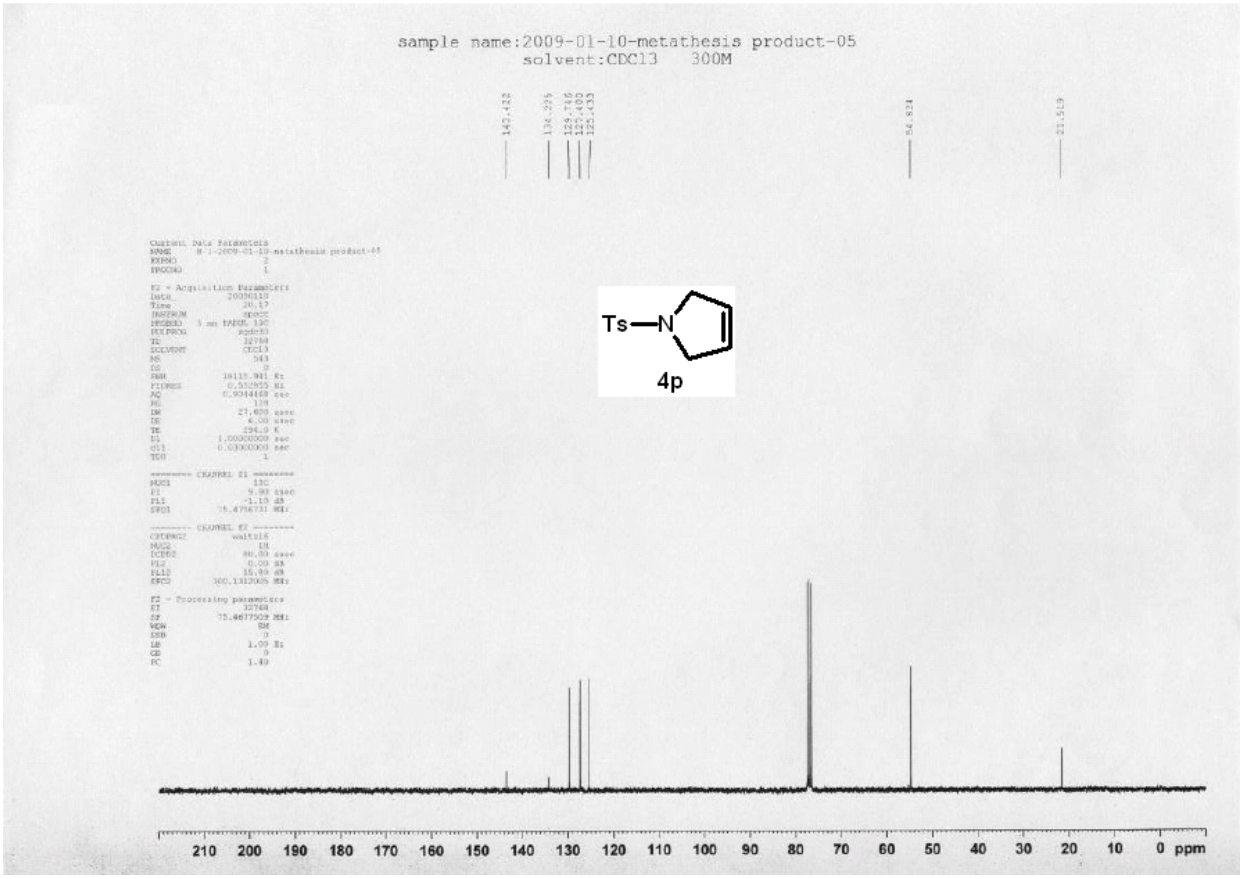

sample name:N-1-2009-2-7-4-1H
7.480
7.474
7.210
7.203
7.182
7.175
7.098
7.060
7.038
7.001
6.878
6.850
5.783
5.778
5.724
5.719
5.266
5.262
5.229
5.225
4.607
4.590
4.551
4.531
4.511
4.491
1.353
1.333
OH
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.955
0.957
0.959
0.981
0.969
0.966
2.070
0.943
6.000

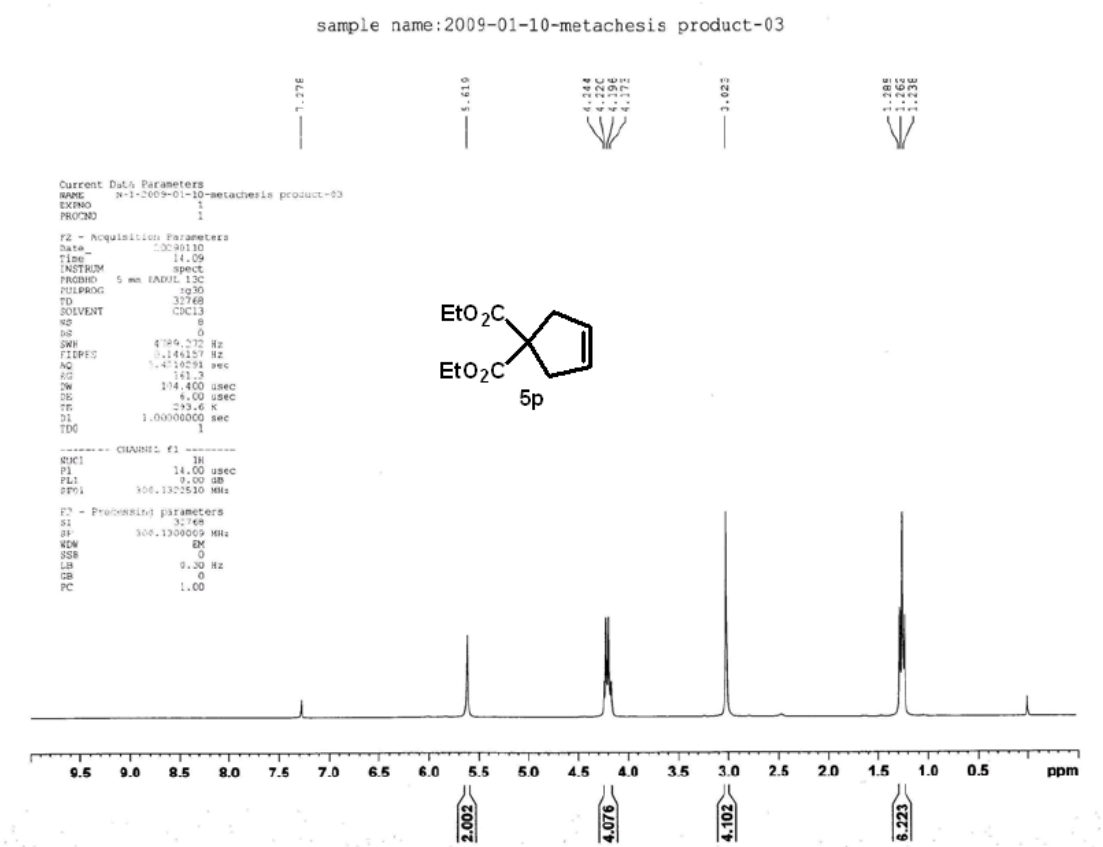

sample name:N-1-2009-2-7-4-13C
solvent:CDCl3 300M
154.728
132.942
131.796
127.899
127.736
125.611
114.342
114.241
77.463
77.039
76.616
71.011
65.114
22.145
OH
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

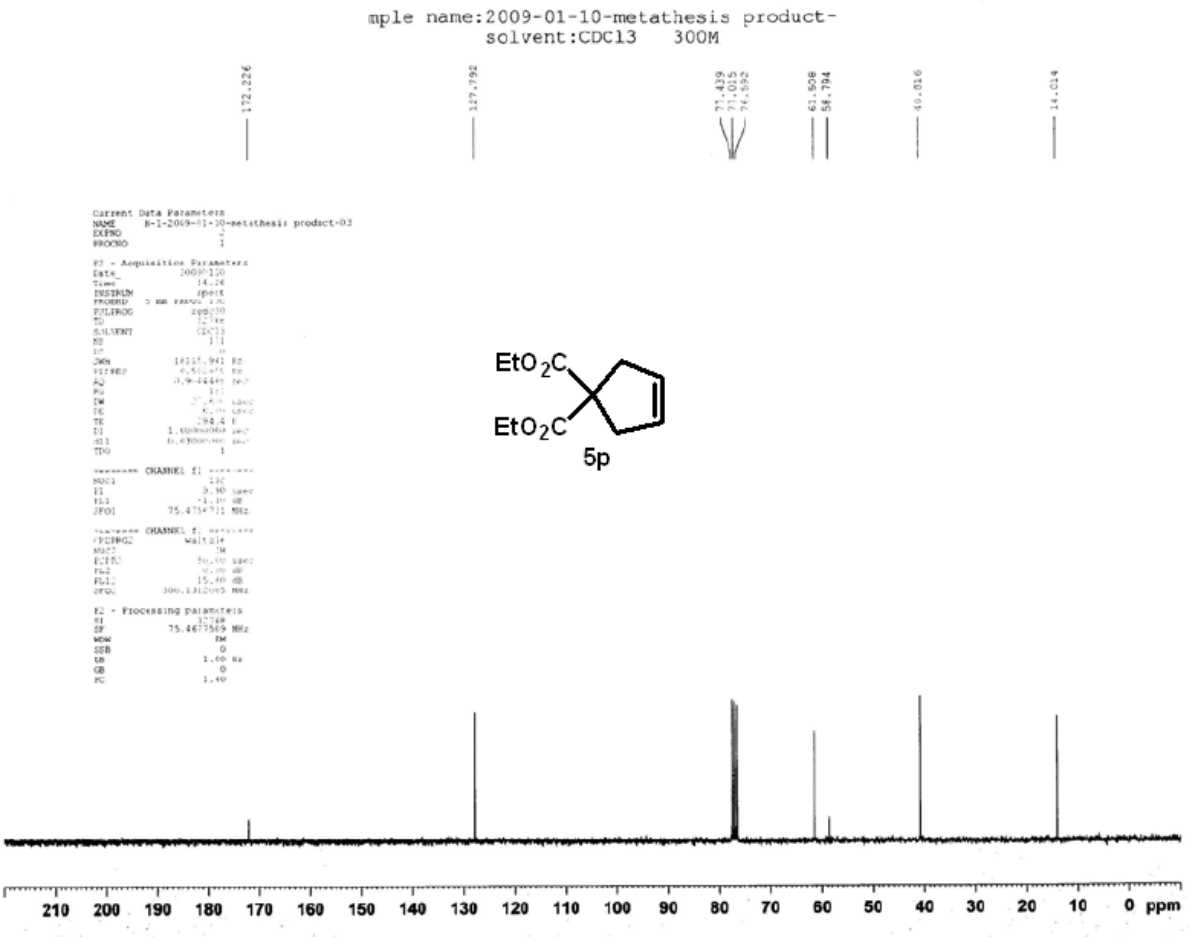

sample name:N-1-2009-05-04
7.458
7.452
7.250
7.207
7.200
7.179
7.172
7.073
7.035
7.013
6.976
6.862
6.833
5.771
5.767
5.712
5.708
5.266
5.262
5.229
5.225
5.039
4.577
4.557
4.537
4.516
4.496
2.469
2.445
2.420
2.395
2.003
1.979
1.954
1.930
1.905
1.347
1.327
1.247
OH
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.000
1.006
1.015
1.018
1.017
1.025
2.056
1.051
5.186
2.597
6.327

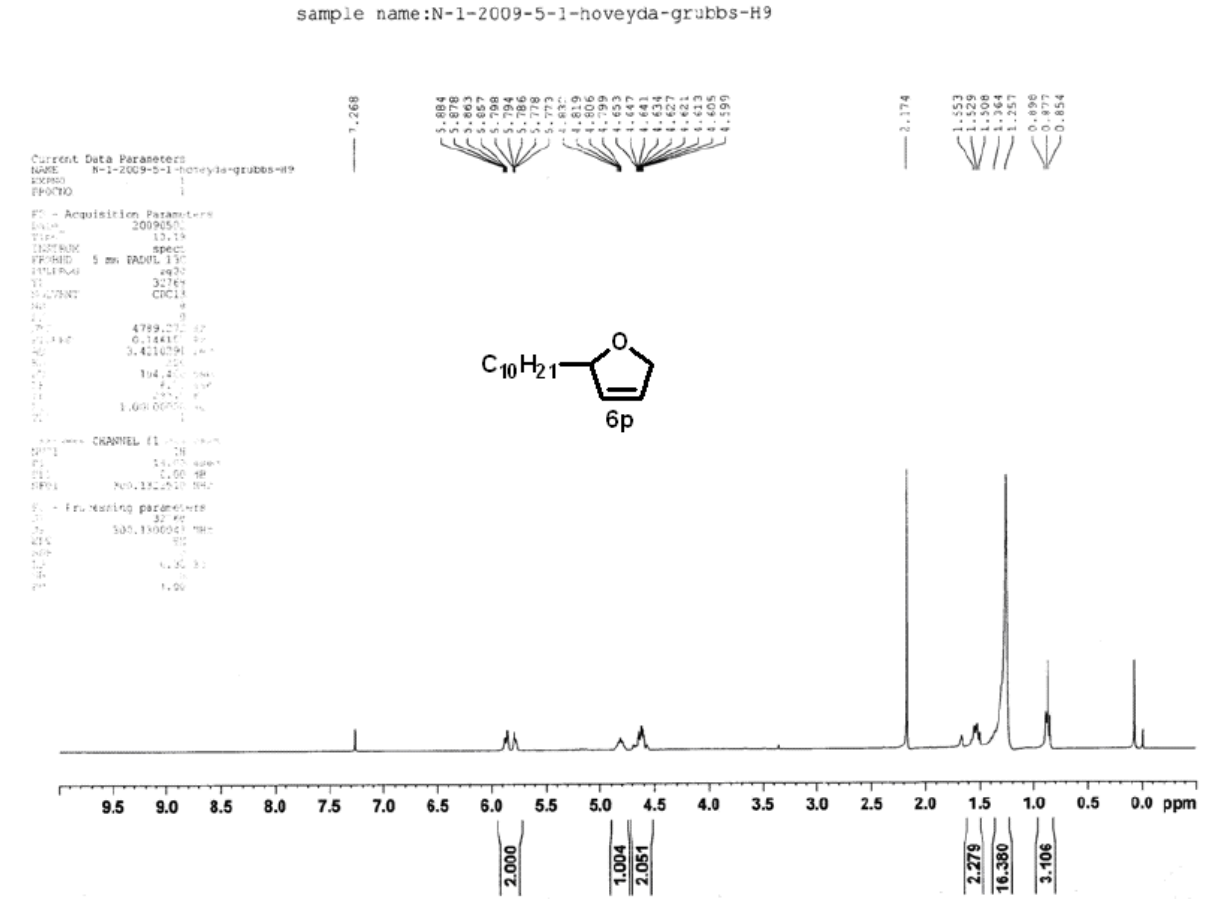

sample name:N-1-2009-05-04-13CNMR
solvent:CDCl3 300M
179.271
179.151
172.855
155.183
131.538
129.351
127.852
127.664
127.137
114.498
113.920
77.475
77.051
76.627
70.847
66.325
33.178
32.972
22.129
19.750
OH
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

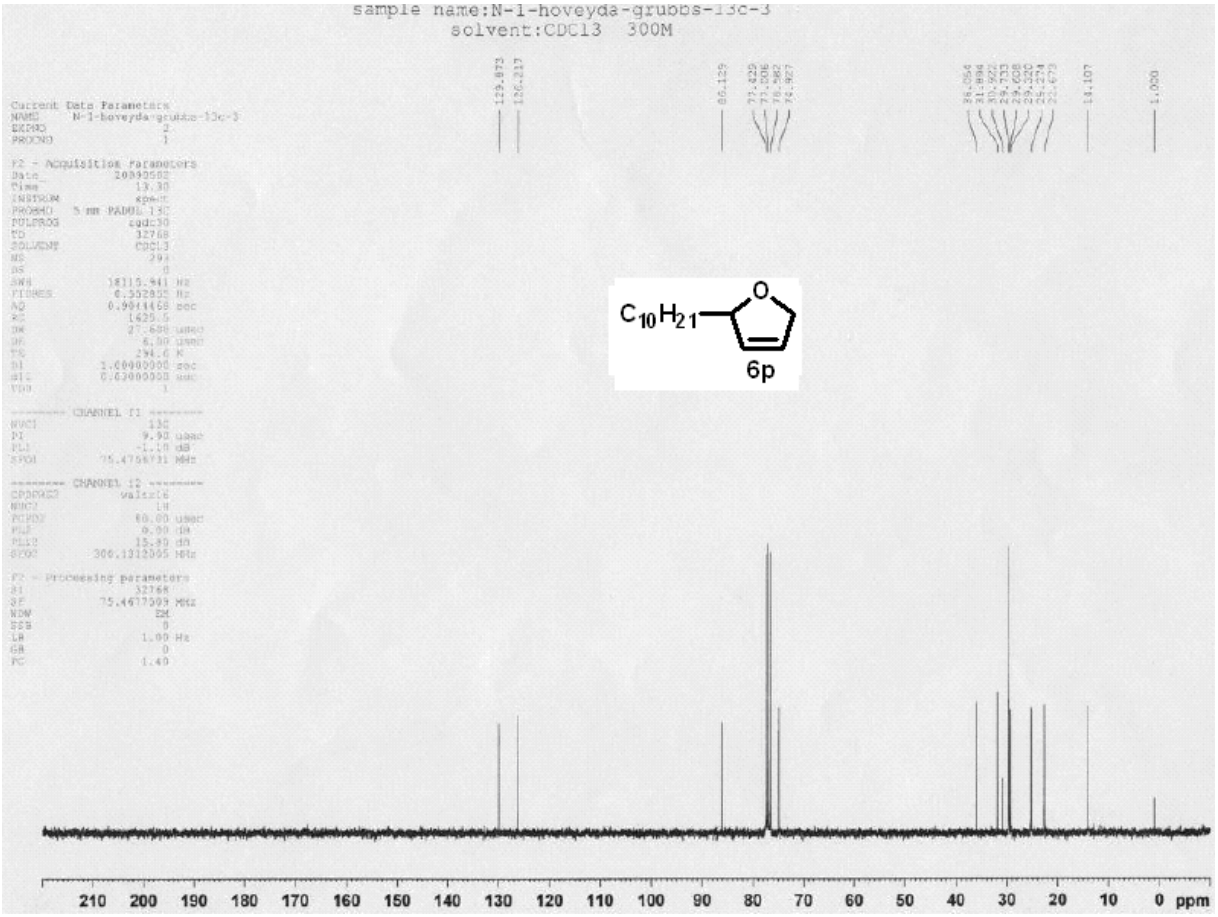

sample name:2009-01-10-metachesis product-05
7.736
7.714
7.709
7.334
7.307
7.277
7.267
7.261
5.652
4.135
4.120
2.427
1.556
Ts—N
4p
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.994
1.978
1.970
4.086
3.001

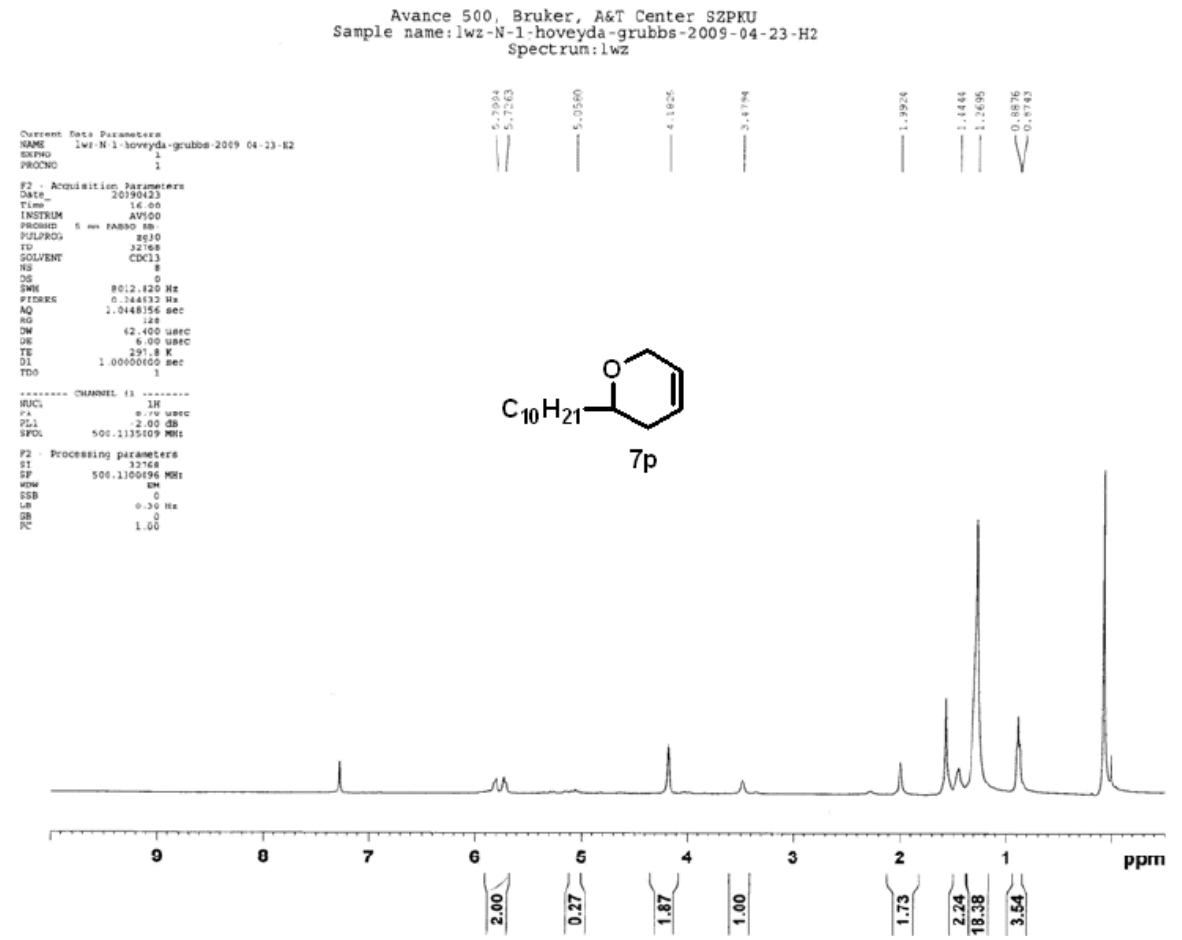

sample name:2009-01-10-metathesis product-05
solvent:CDCl3 300M
142.438
136.295
129.746
125.400
125.433
54.824
21.519
Ts—N
4p
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

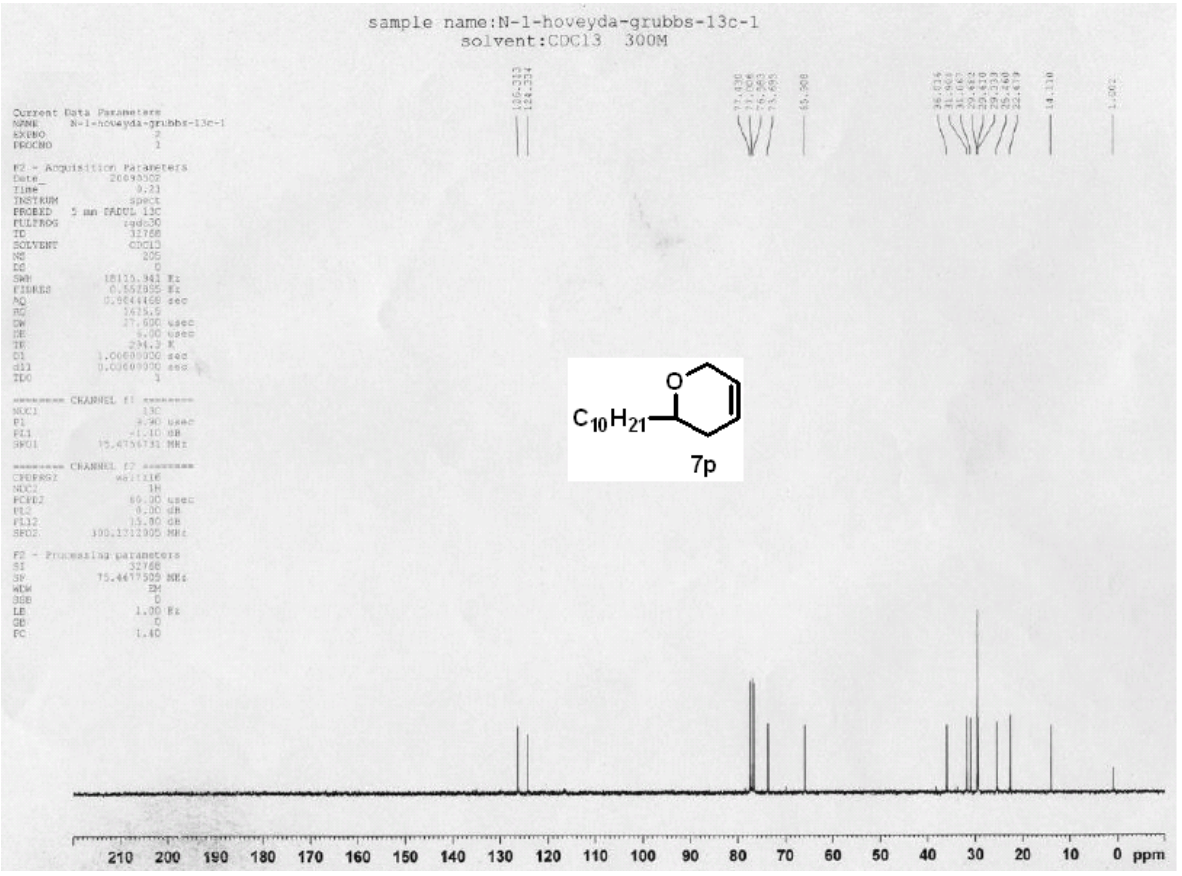

sample name:2009-01-10-metachesis product-03
7.278
5.619
4.244
4.226
4.208
4.190
4.172
3.023
1.288
1.262
1.236
Current Data Parameters
NAME N-1-2009-01-10-metachesis product-03
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090110
Time_ 14.09
INSTRUM spect
PROBHD 5 mm PABBL 13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 161.3
DW 104.400 usec
DE 6.00 usec
TE 293.6 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 14.00 usec
PL1 9.00 dB
SFO1 300.1320510 MHz
F2 - Processing parameters
SI 32768
SF 300.1300009 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
EtO2C
EtO2C
5p
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.002
4.076
4.102
6.223

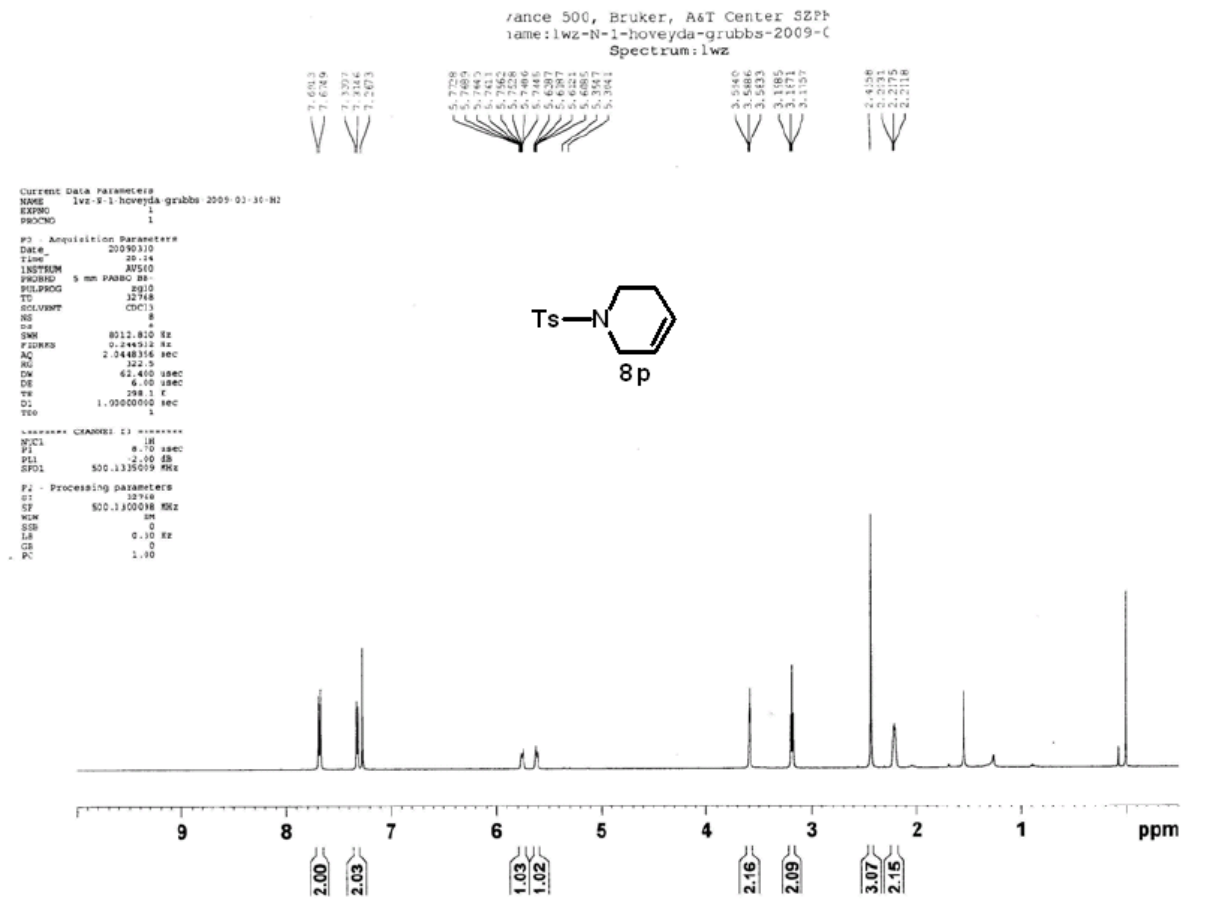

mple name:2009-01-10-metathesis product-
solvent:CDCl3 300M
172.226
127.792
77.439
77.015
76.592
61.508
58.794
40.816
14.014
Current Data Parameters
NAME N-1-2009-01-10-metathesis product-03
EXPNO
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090120
Time_ 14.24
INSTRUM spect
PULPROG zgpg30
SOLVENT CDCl3
NS 114
DW 16.640 usec
TE 298.4 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 13C
P1 9.90 usec
PL1 -1.00 dB
SFO1 75.4754711 MHz
F2 - Processing parameters
SI 32768
SF 75.4677569 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
EtO2C
EtO2C
5p
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

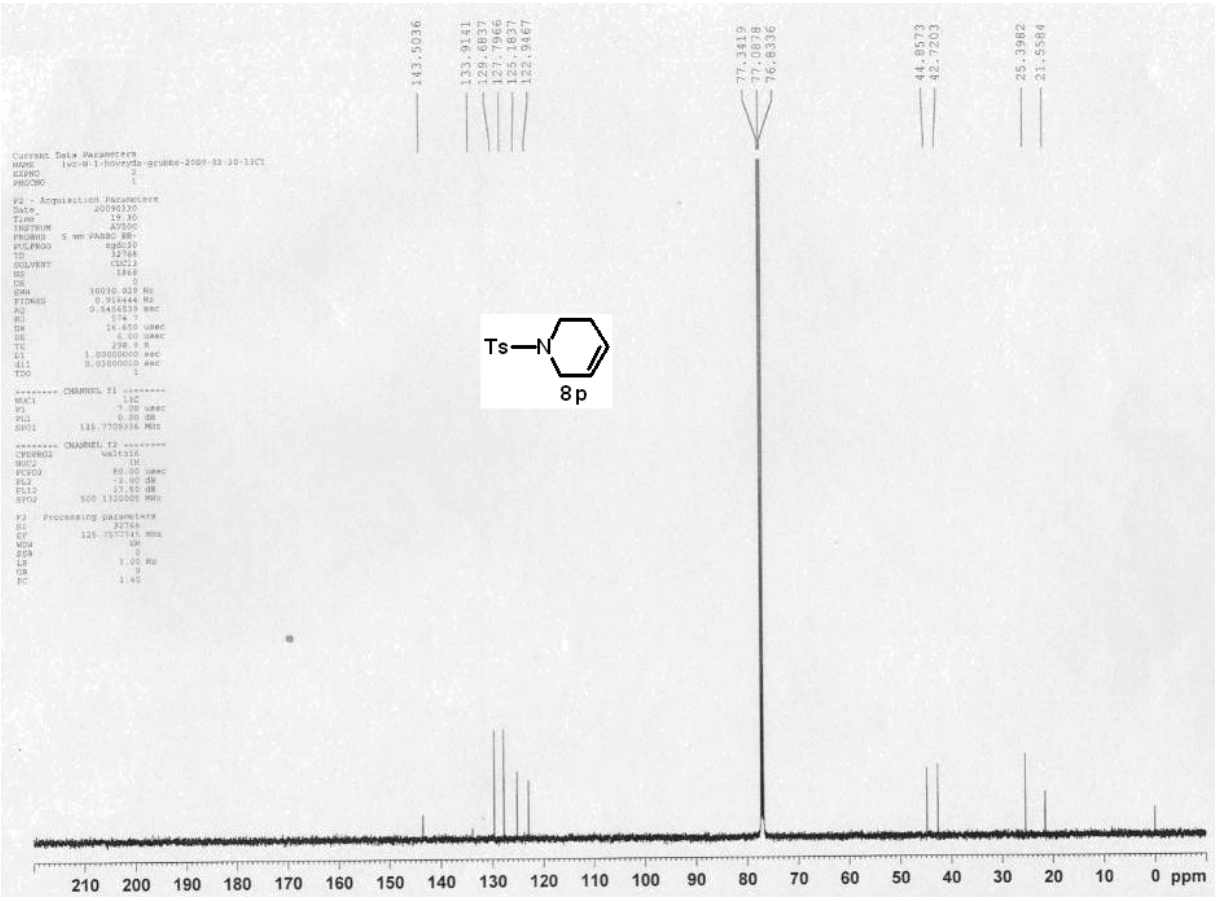

sample name:N-1-2009-5-1-hoveyda-grubbs-H9
$C_{10}H_{21}$
6p
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.000
1.004
2.051
2.279
16.380
3.106

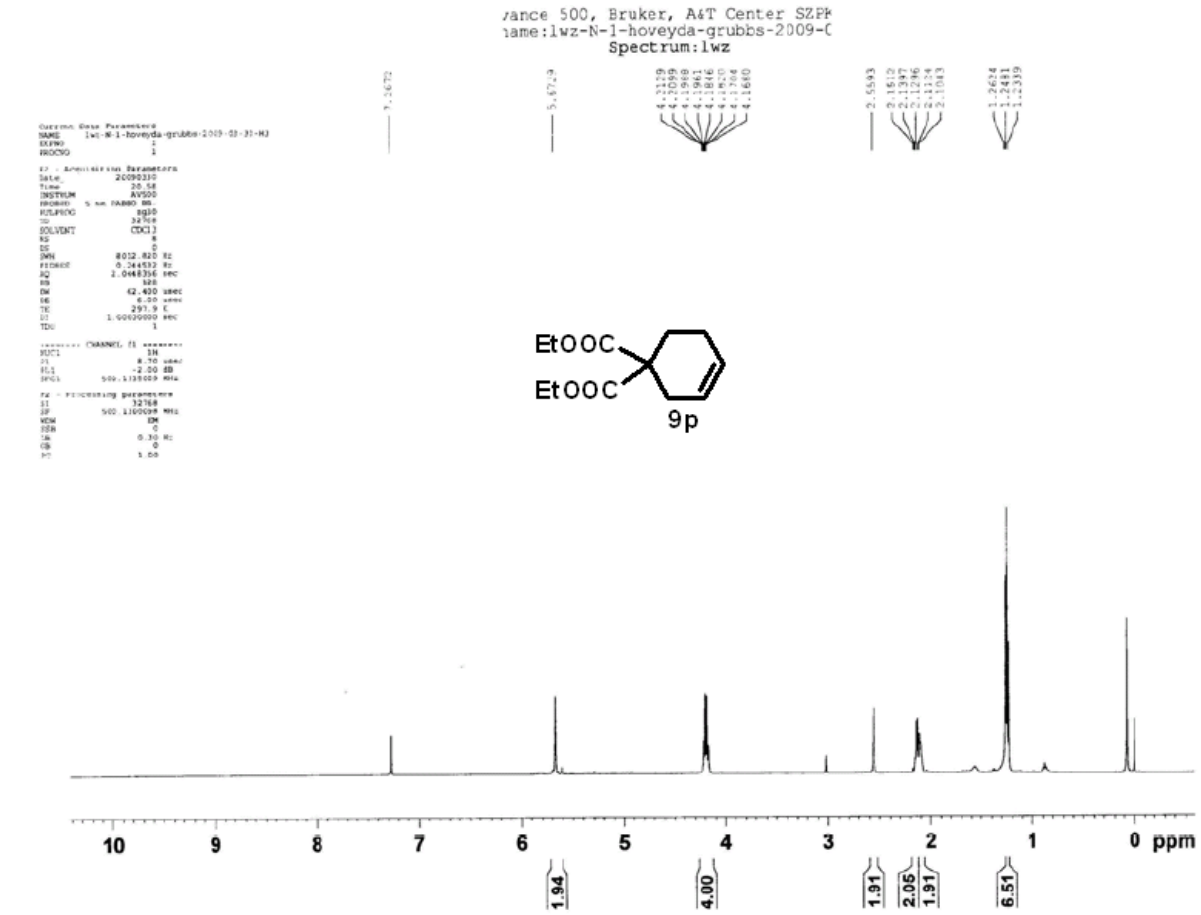

sample name:N-1-hoveyda-grubbs-13c-3
solvent:CDCl3 300M
$C_{10}H_{21}$
6p
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

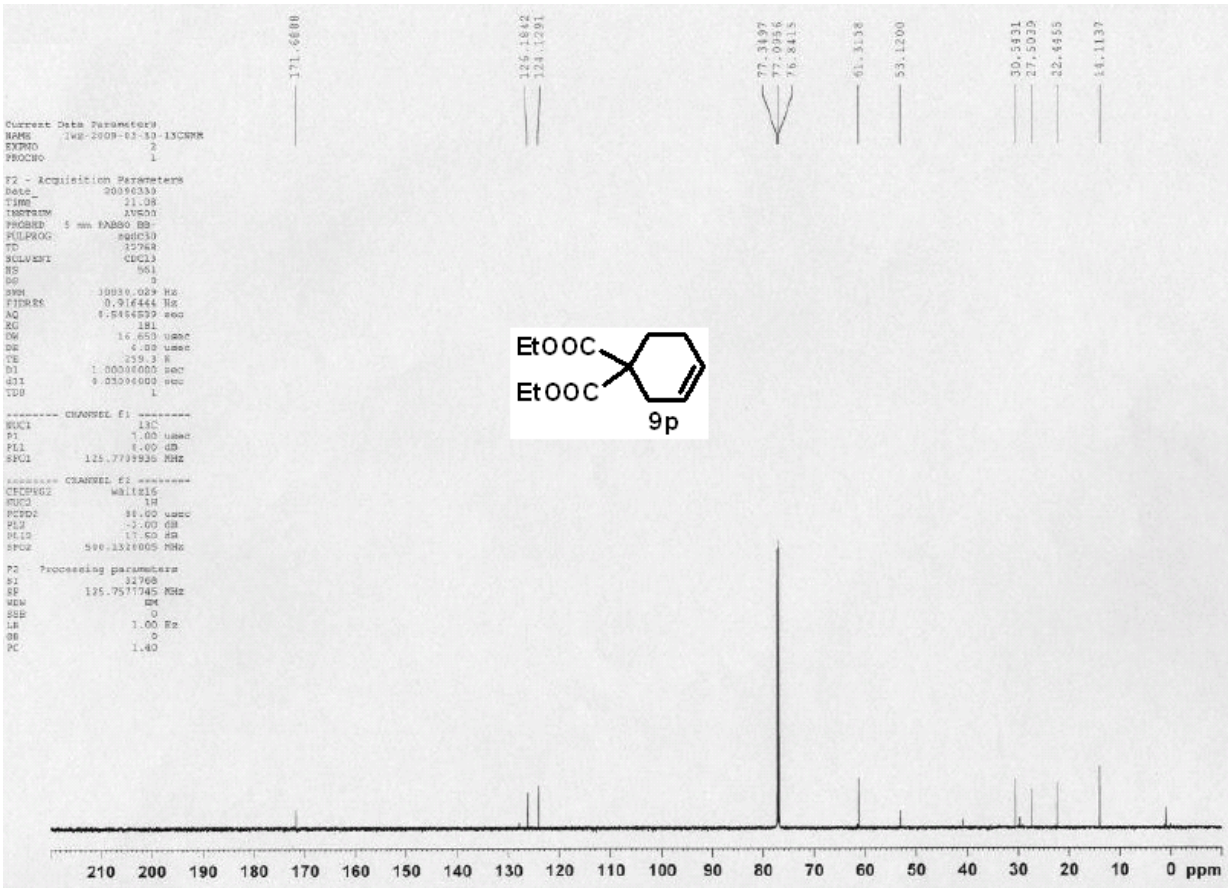

Avance 500, Bruker, A&T Center SZPKU
Sample name:lwz-N-1-hoveyda-grubbs-2009-04-23-H2
Spectrum:lwz
5.7994
5.7763
5.0560
4.1825
3.4794
1.9924
1.4444
1.2695
0.8876
0.8745
C10H21
O
7p
9
8
7
6
5
4
3
2
1
ppm
2.00
0.27
1.87
1.00
1.73
2.24
18.38
3.54

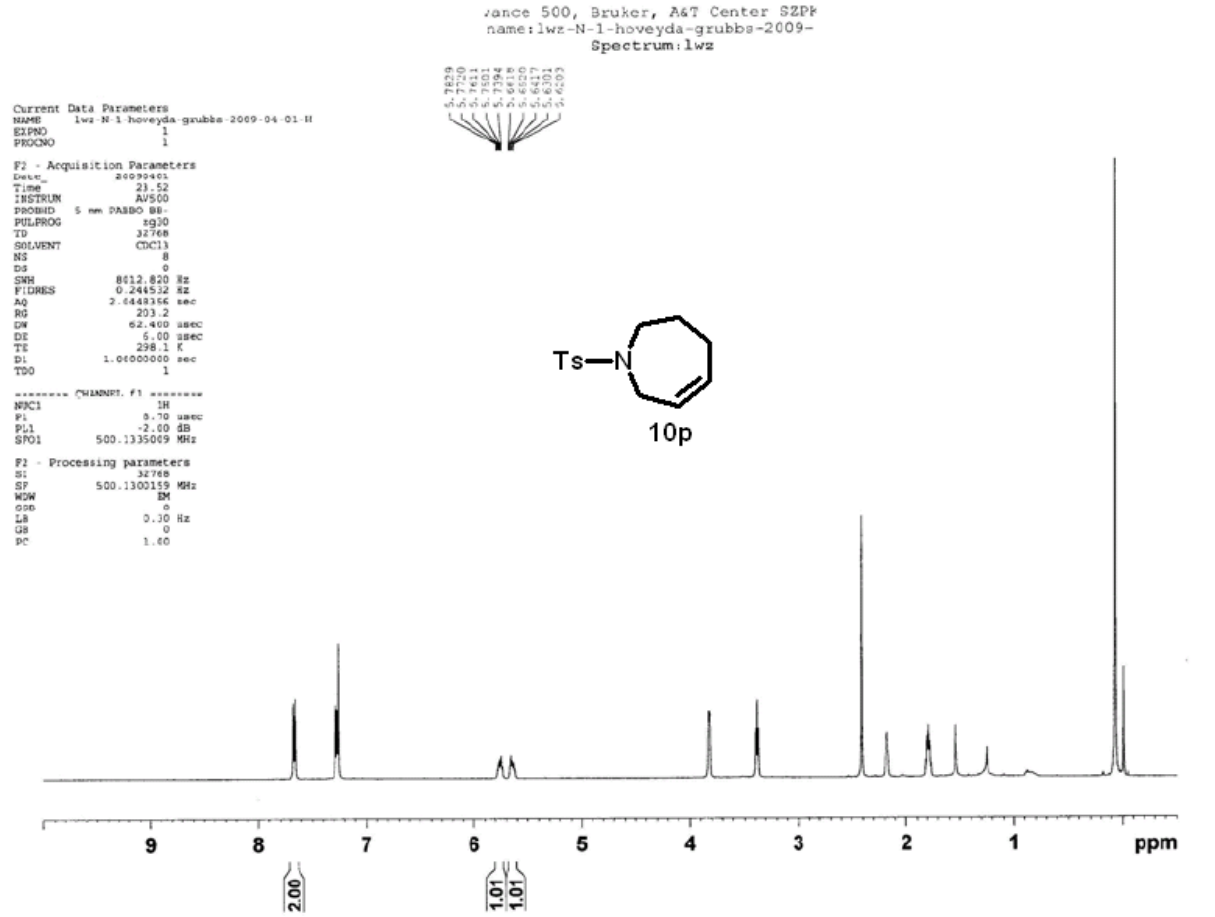

sample name:N-1-hoveyda-grubbs-13c-1
solvent:CDCl3 300M
C10H21
O
7p
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

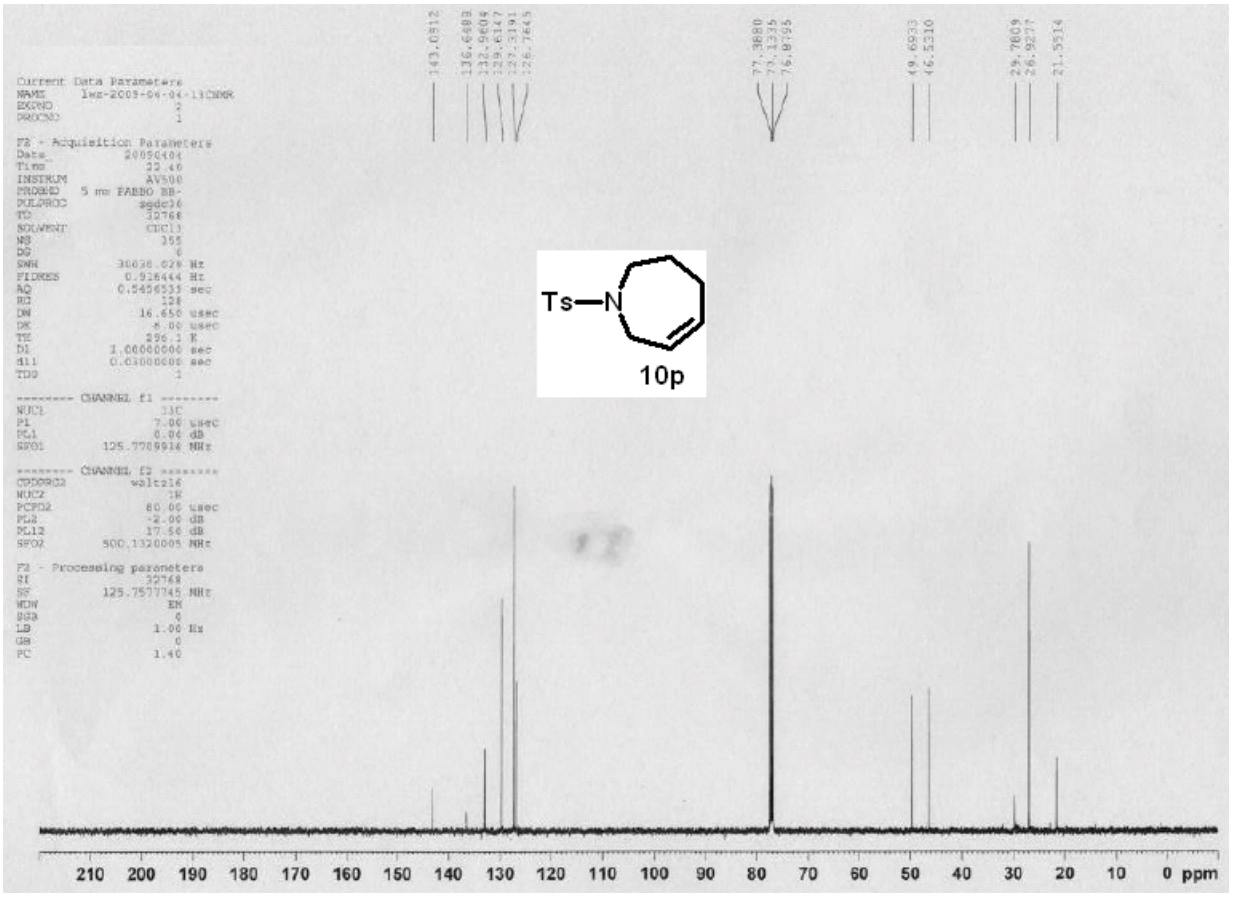

Spectrum:1wz
Ts—N
8p
9
8
7
6
5
4
3
2
1
ppm
2.00
2.03
1.03
1.02
2.16
2.09
3.07
2.15

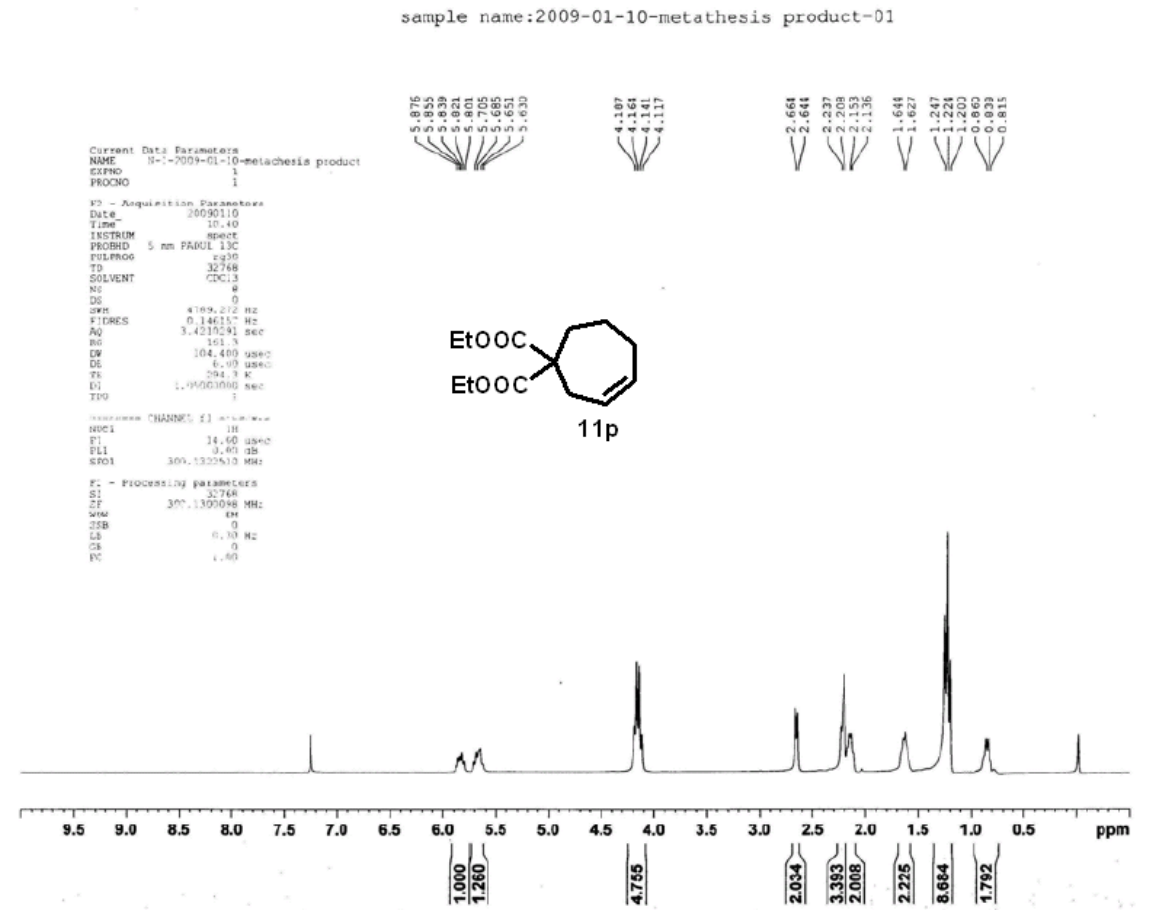

143.5036
133.9141
129.6837
127.7966
125.1837
122.9467
77.3419
77.0878
76.8336
44.8573
42.7203
25.3982
21.5584
Ts—N
8p
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

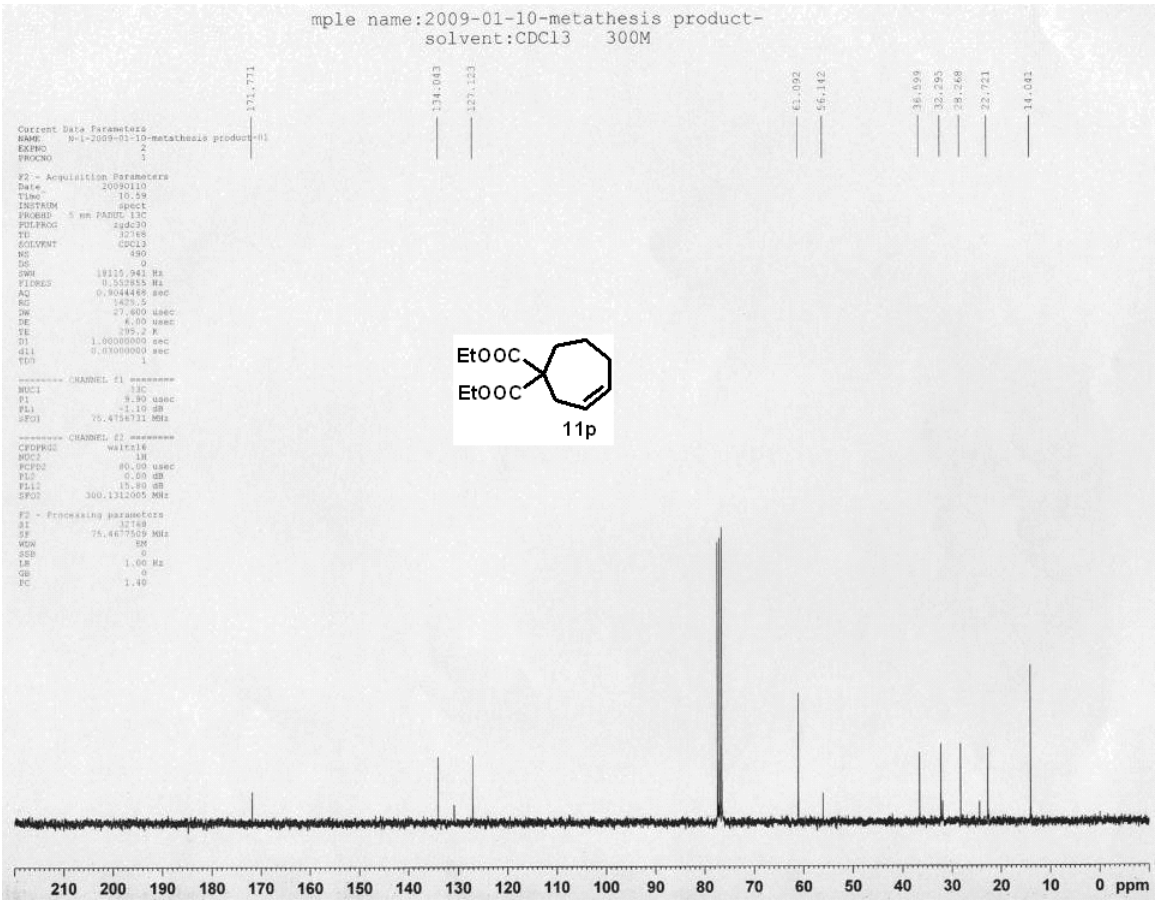

Spectrum:lwz
EtOOC
EtOOC
9p
10 9 8 7 6 5 4 3 2 1 0 ppm
1.94
4.00
1.91
2.05
1.91
6.51

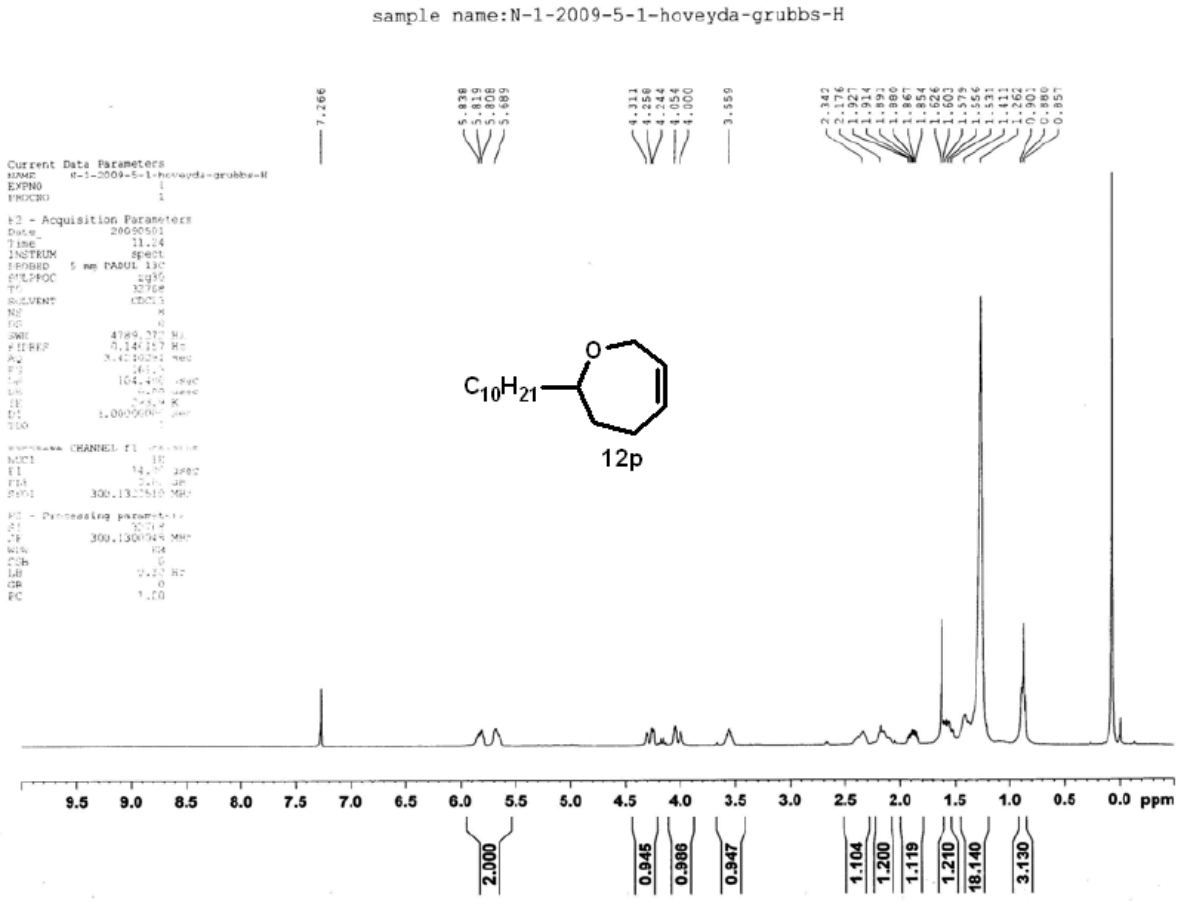

EtOOC
EtOOC
9p
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

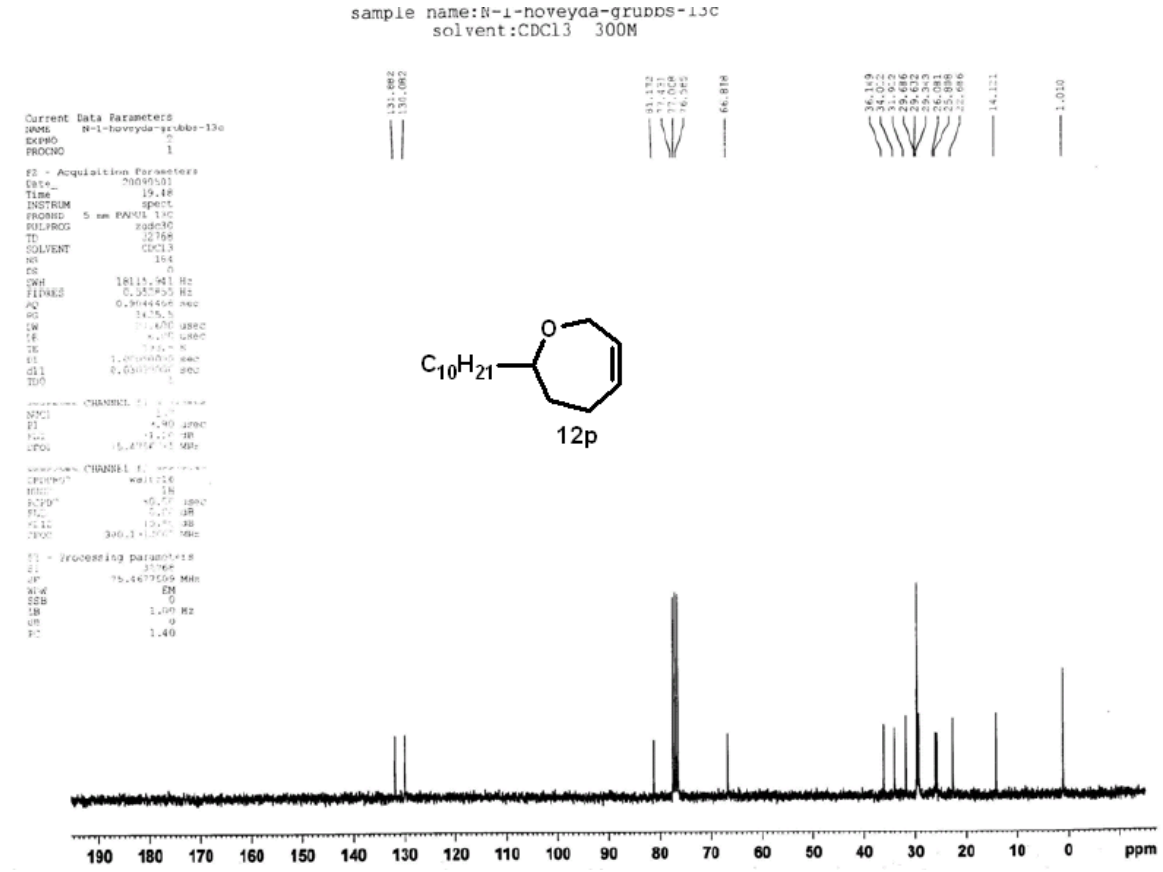

vance 500, Bruker, A&T Center SZPK
name:lwz-N-1-hoveyda-grubbs-2009-
Spectrum:lwz
Ts—N
10p
9
8
7
6
5
4
3
2
1
ppm
2.00
1.01
1.01


Ts—N
10p
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm


sample name:2009-01-10-metathesis product-01
5.876
5.855
5.839
5.821
5.801
5.705
5.685
5.651
5.630
4.107
4.164
4.141
4.117
2.664
2.641
2.237
2.208
2.153
2.136
1.644
1.627
1.247
1.221
1.200
0.869
0.839
0.815
EtOOC
EtOOC
11p
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.000
1.260
4.755
2.034
3.393
2.008
2.225
8.684
1.792

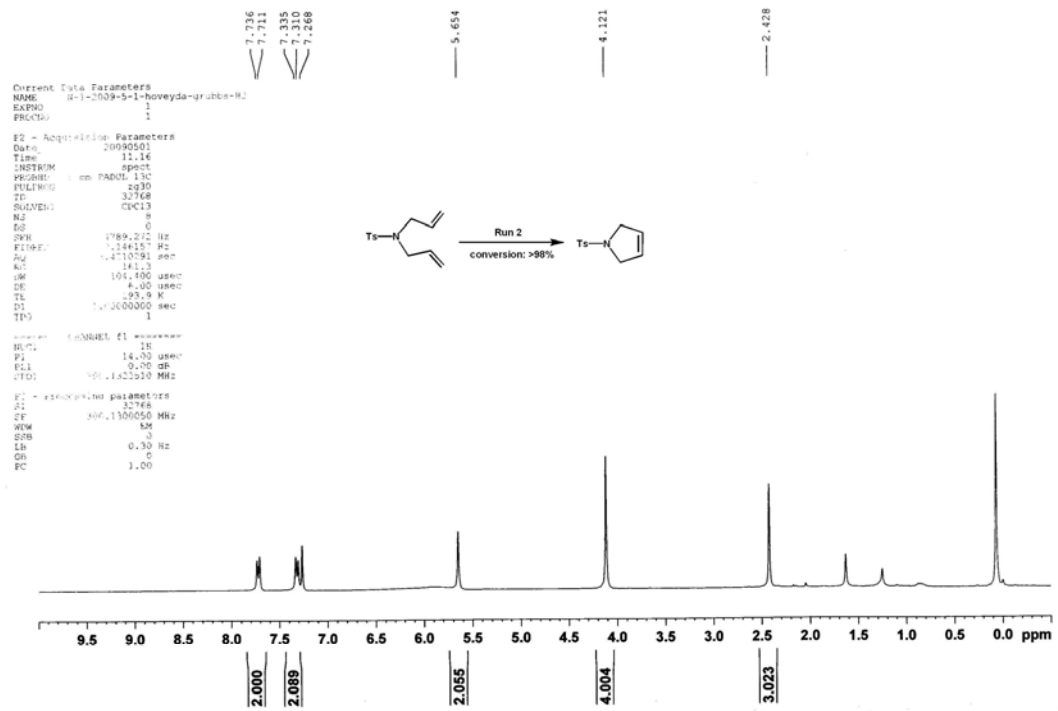

mple name:2009-01-10-metathesis product-
solvent:CDCl3 300M
171.771
134.043
127.123
61.092
56.142
36.599
32.295
28.268
22.721
14.041
EtOOC
EtOOC
11p
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


sample name:N-1-2009-5-1-hoveyda-grubbs-H
7.266
5.838
5.819
5.808
5.689
4.311
4.256
4.244
4.054
4.000
3.559
2.342
2.176
1.927
1.914
1.893
1.880
1.867
1.854
1.626
1.603
1.579
1.556
1.531
1.111
1.262
0.901
0.880
0.857
O
C10H21
12p
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.000
0.945
0.986
0.947
1.104
1.200
1.119
1.210
18.140
3.130

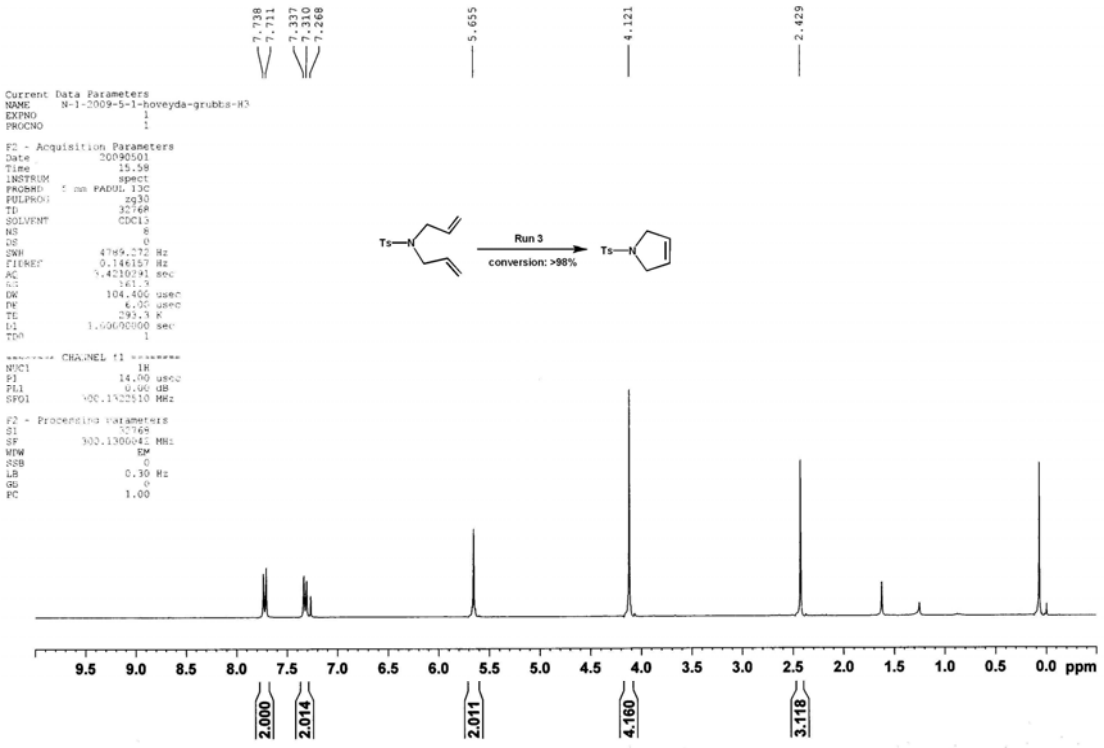

sample name:N-1-hoveyda-grubbs-13c
solvent:CDCl3 300M
131.662
130.062
93.172
77.431
77.004
76.585
66.814
36.149
34.022
31.672
29.636
29.615
29.343
26.081
25.898
22.686
14.121
1.010
O
C10H21
12p
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{1}H NMR Spectra for the selected products 4p and 10p in RCM Reactions with recyclable Magnetic Catalyst 3

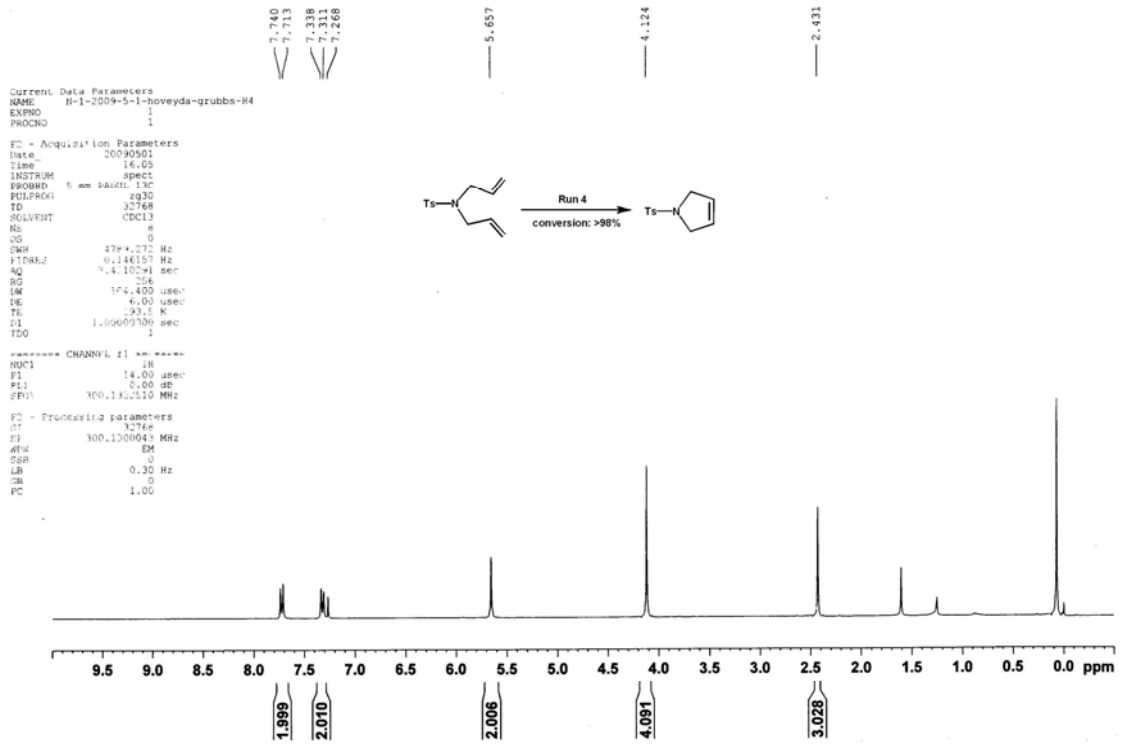
sample name:N-1-2009-5-1-hoveyda-grubbs-H2



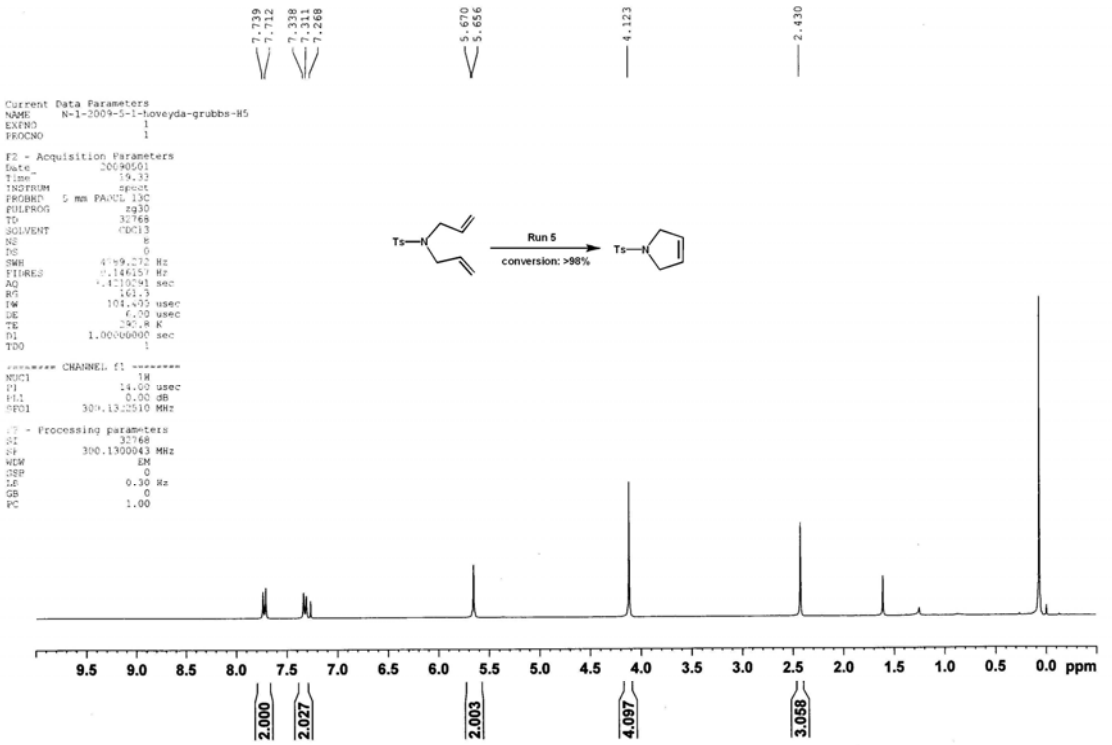
sample name:N-1-2009-5-1-hoveyda-grubbs-H3



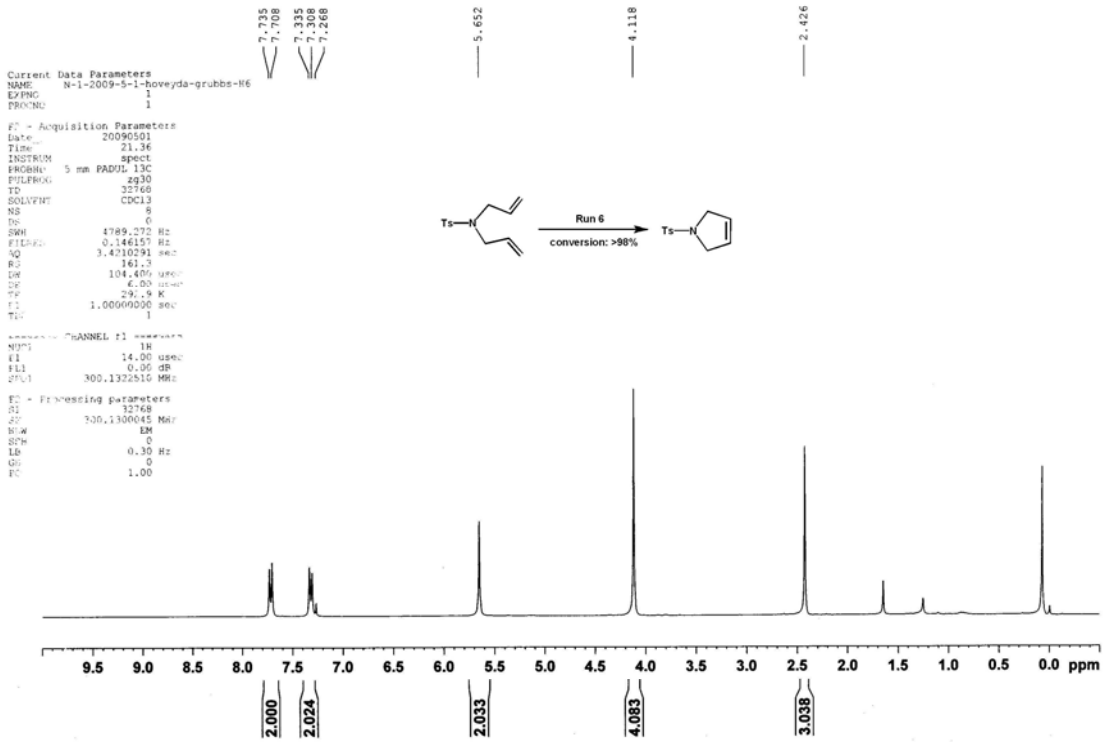
sample name:N-1-2009-5-1-hoveyda-grubbs-H4



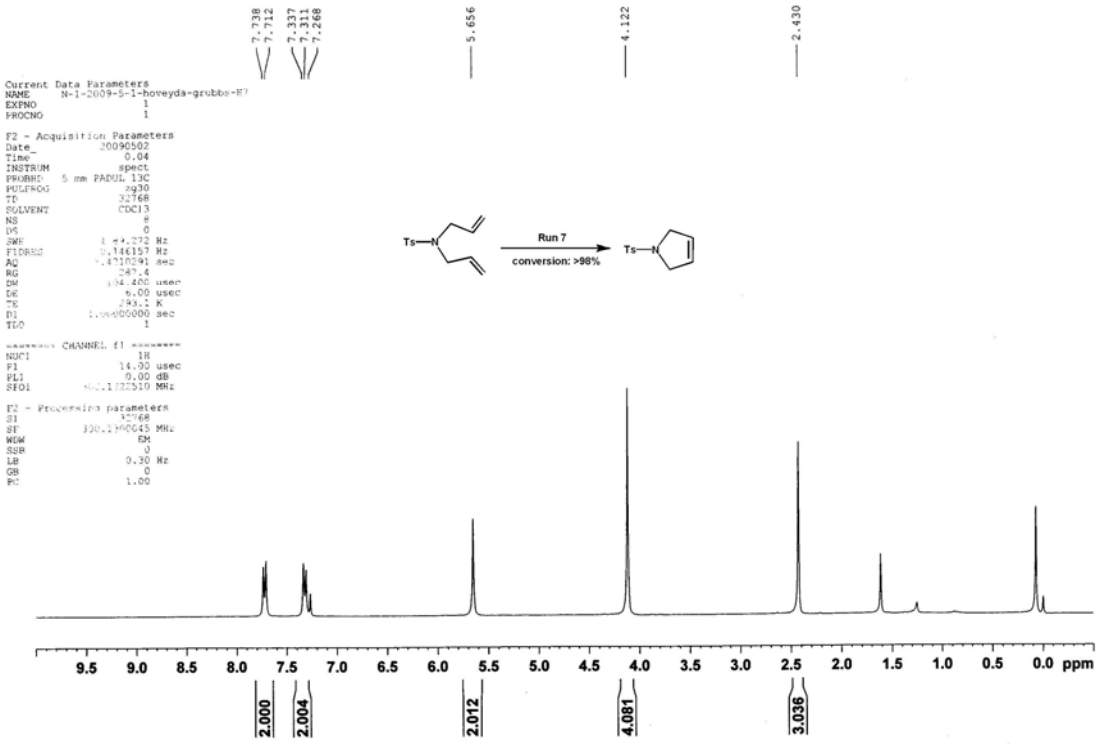
sample name:N-1-2009-5-1-hoveyda-grubbs-H5



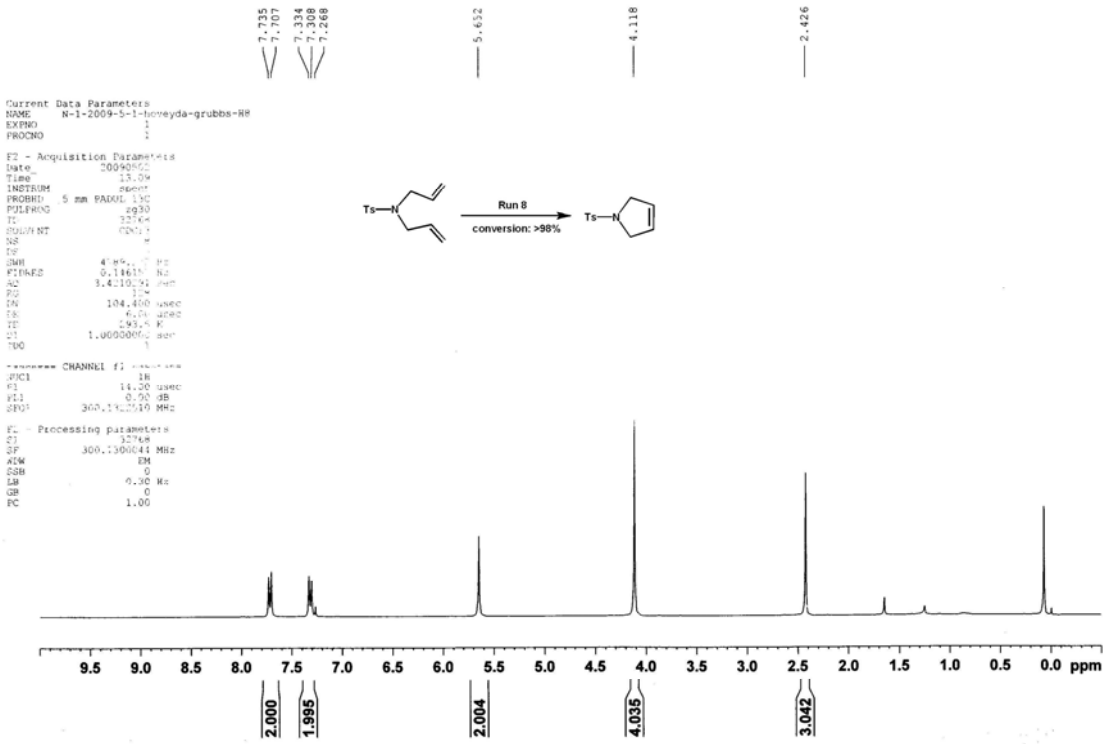
sample name:N-1-2009-5-1-hoveyda-grubbs-H6



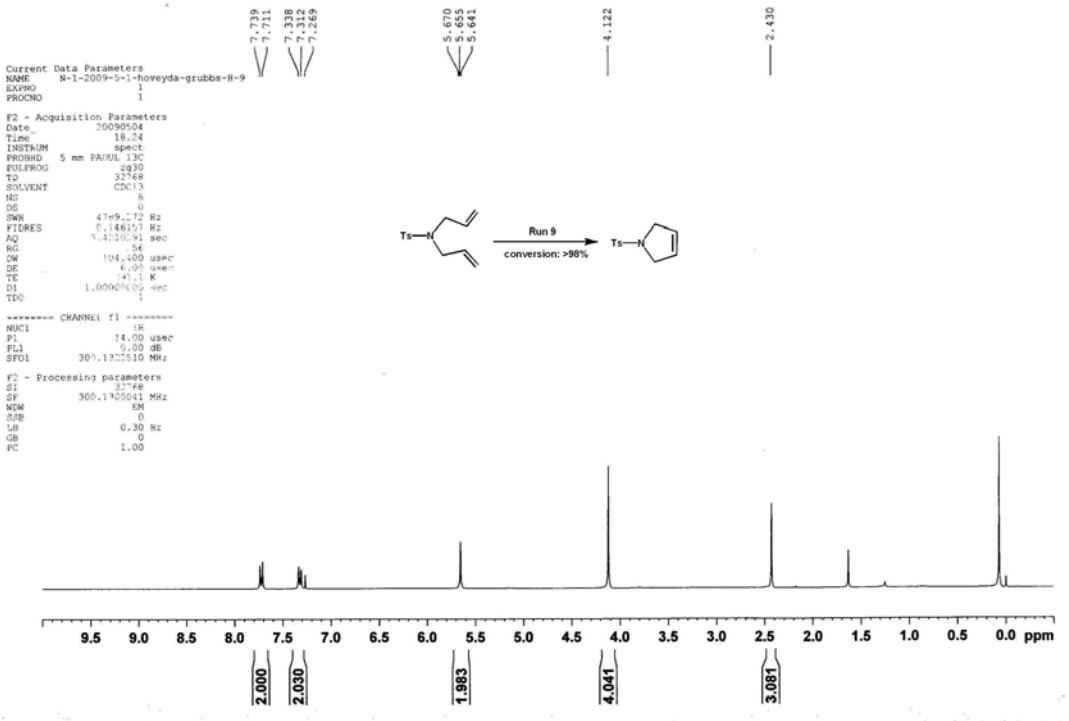
sample name:N-1-2009-5-1-hoveyda-grubbs-H7



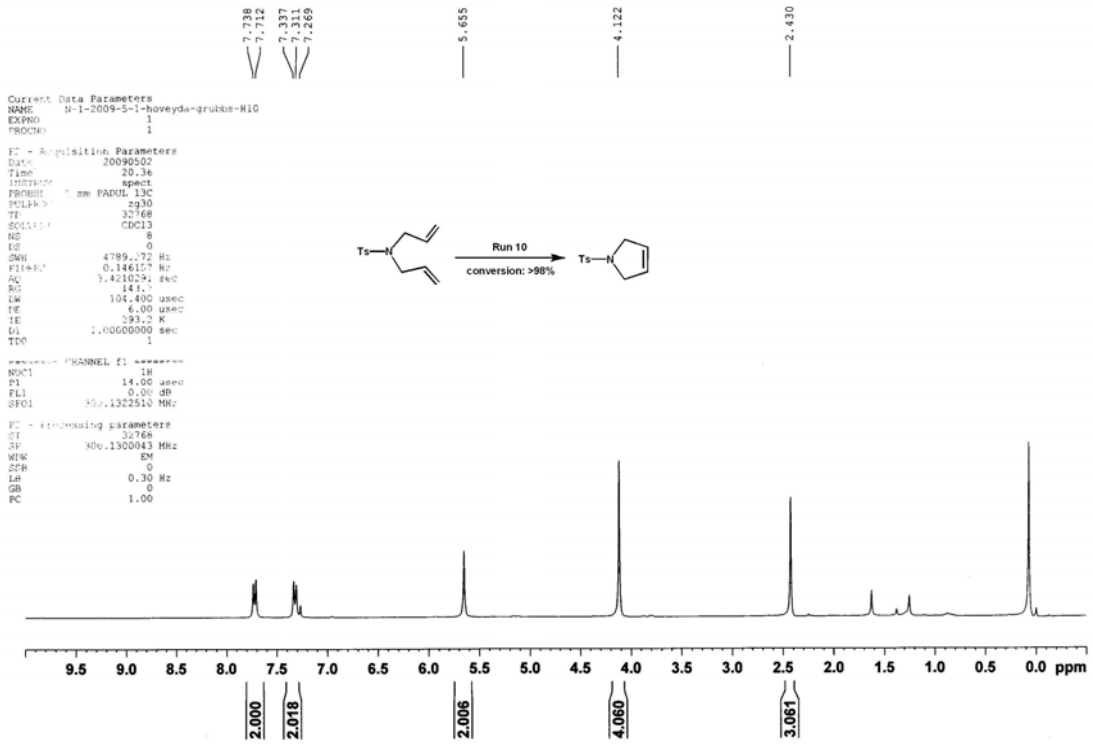
sample name:N-1-2009-5-1-hoveyda-grubbs-H8



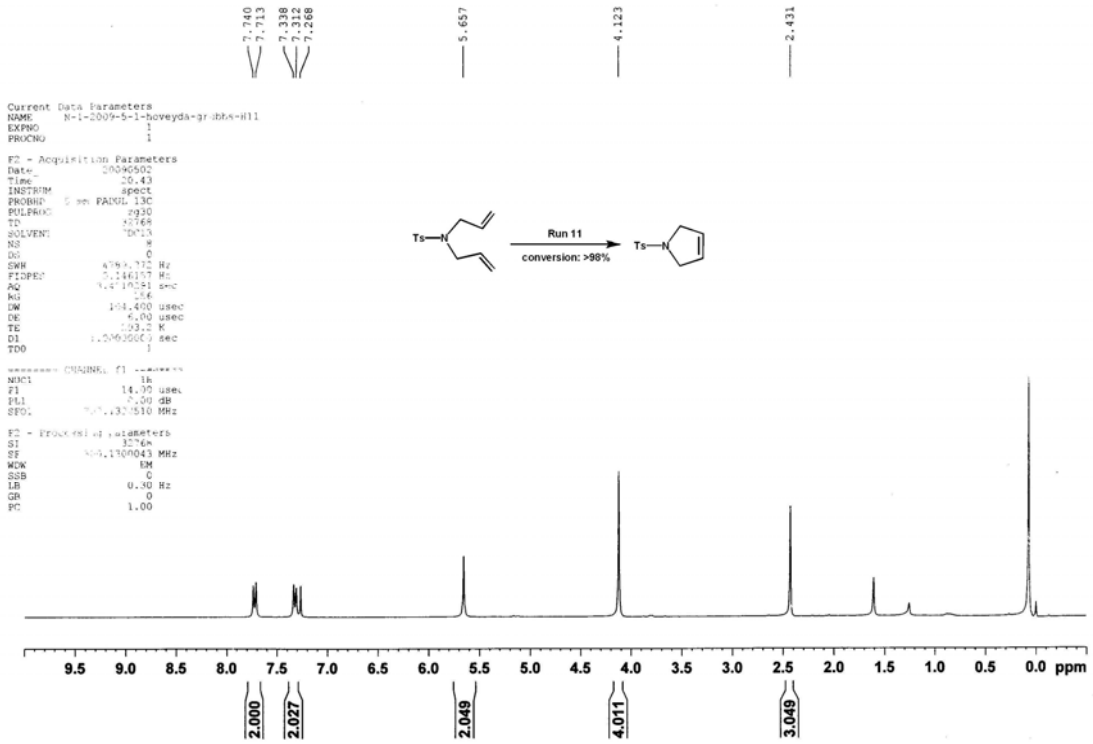
sample name:N-1-2009-5-1-hoveyda-grubbs-H-9



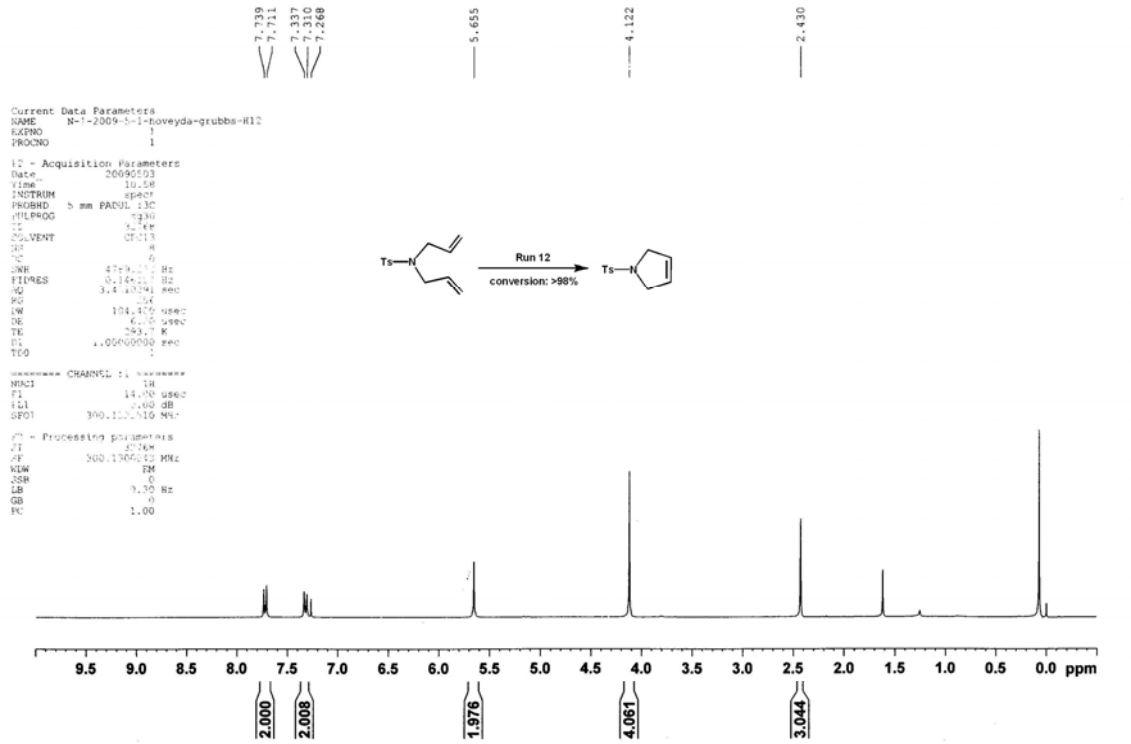
sample name:N-1-2009-5-1-hoveyda-grubbs-H10



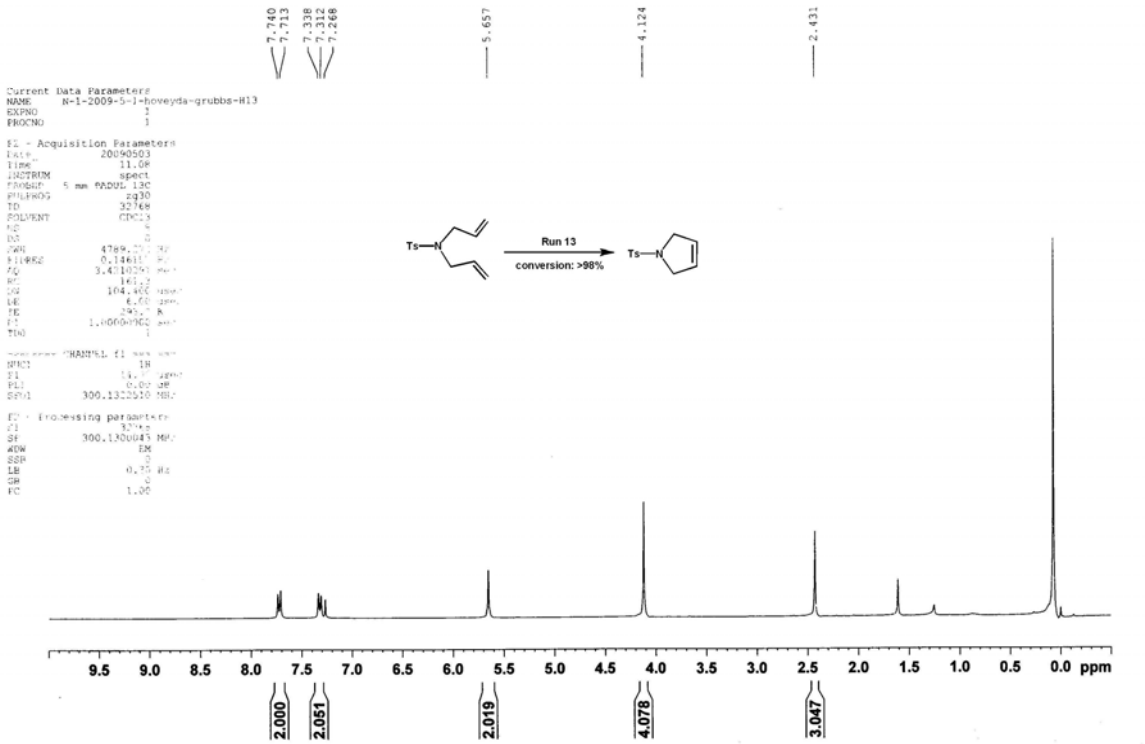
sample name:N-1-2009-5-1-hoveyda-grubbs-H11

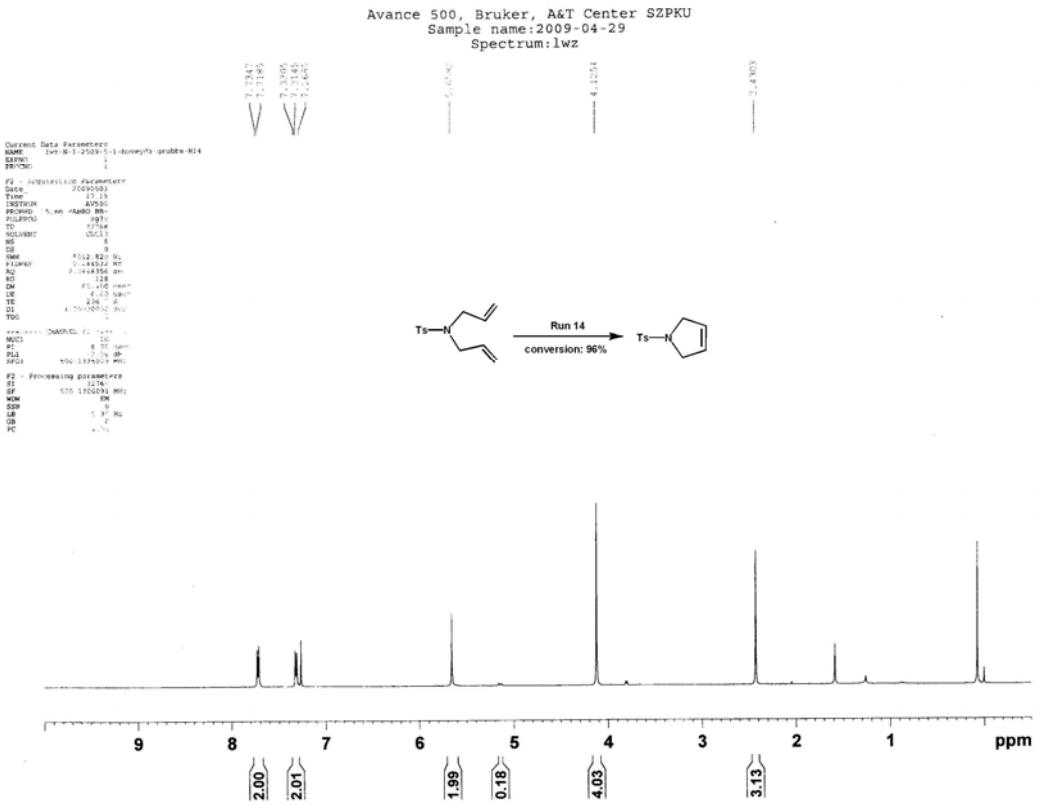


sample name:N-1-2009-5-1-hoveyda-grubbs-H12

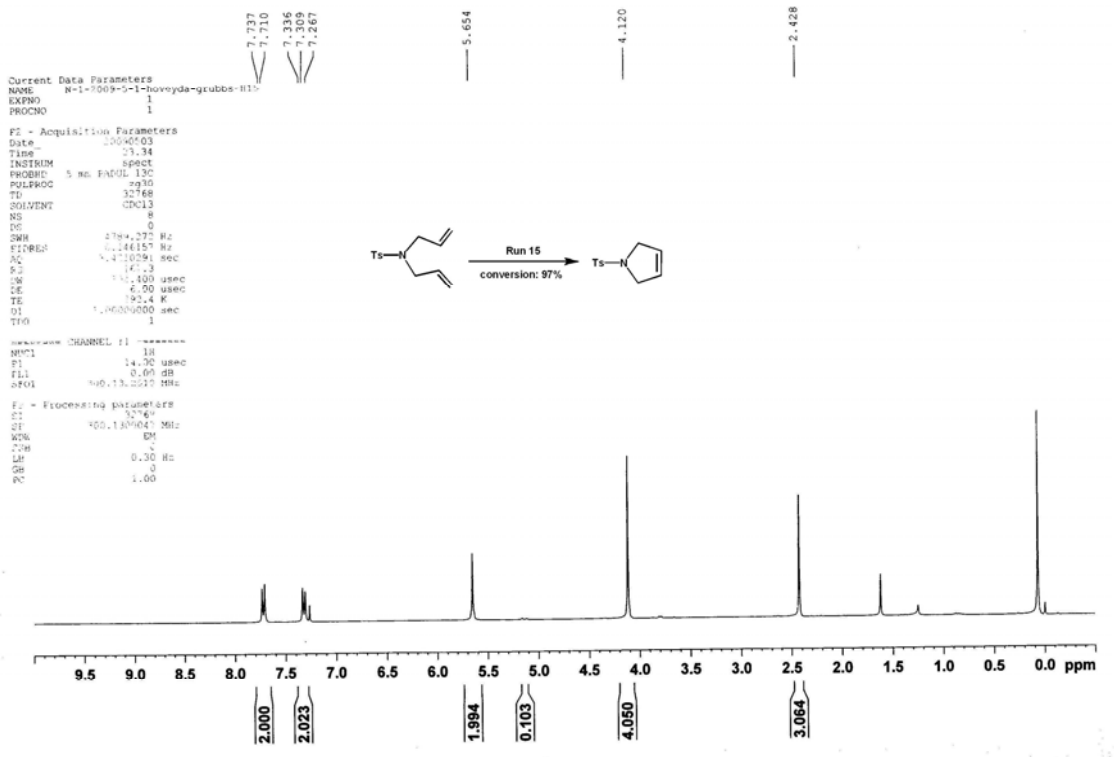


sample name:N-1-2009-5-1-hoveyda-grubbs-H13

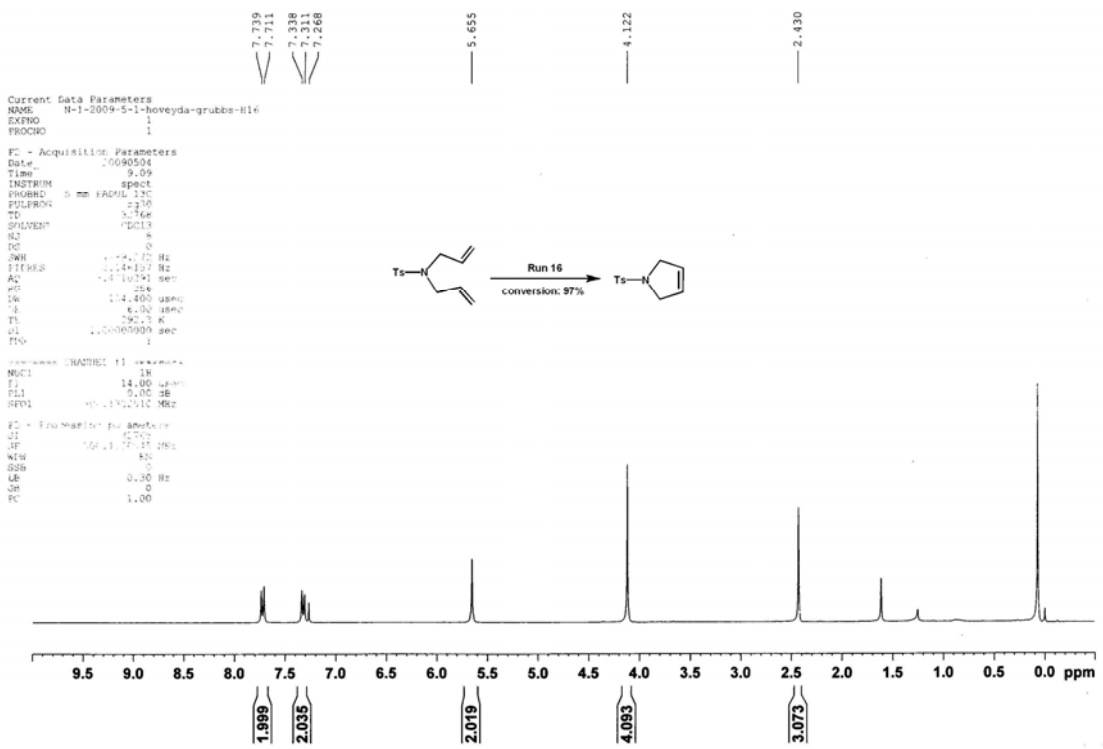




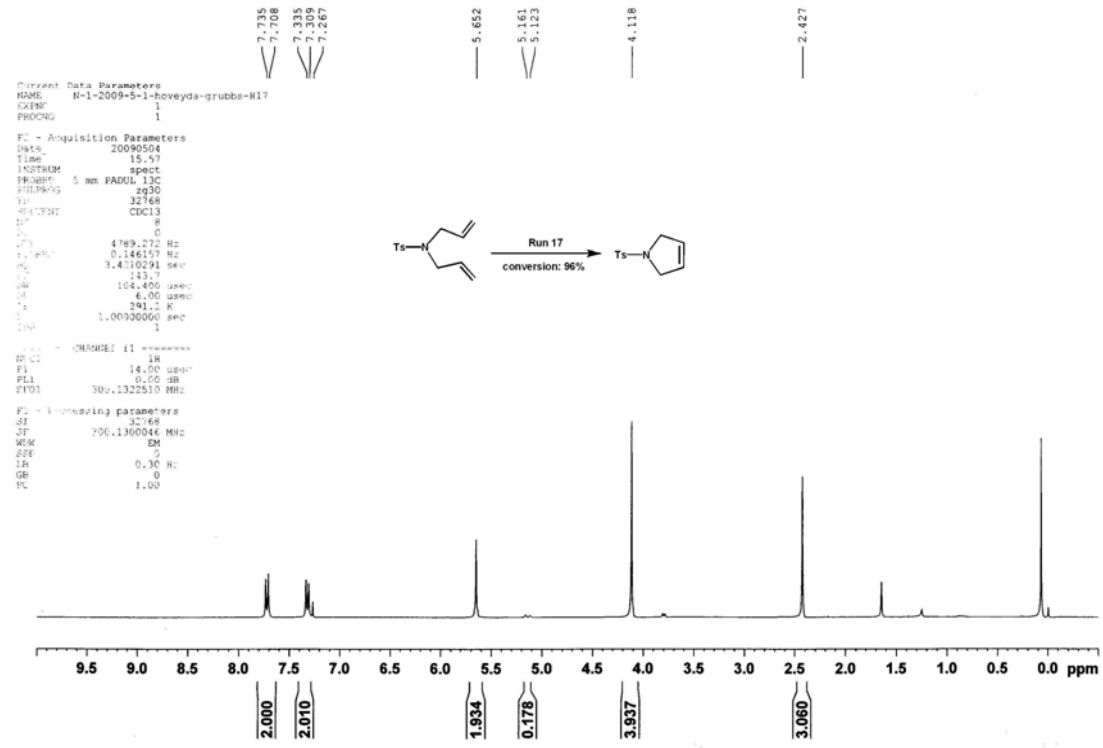
sample name:N-1-2009-5-1-hoveyda-grubbs-H15



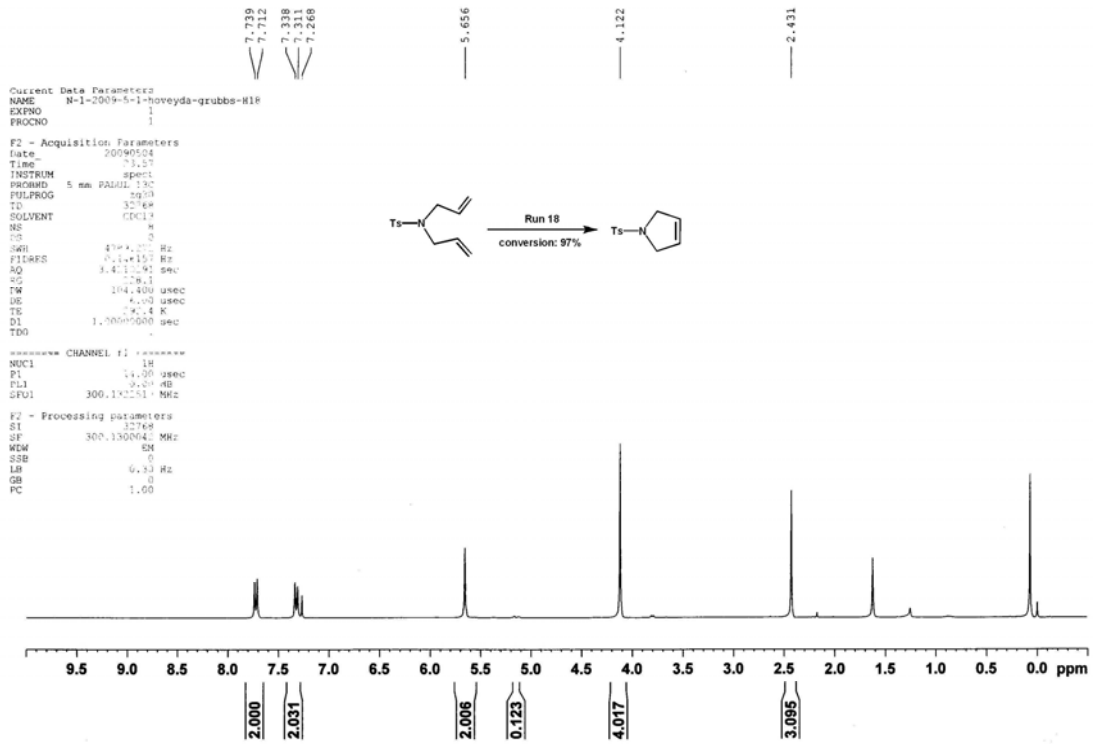
sample name:N-1-2009-5-1-hoveyda-grubbs-H16



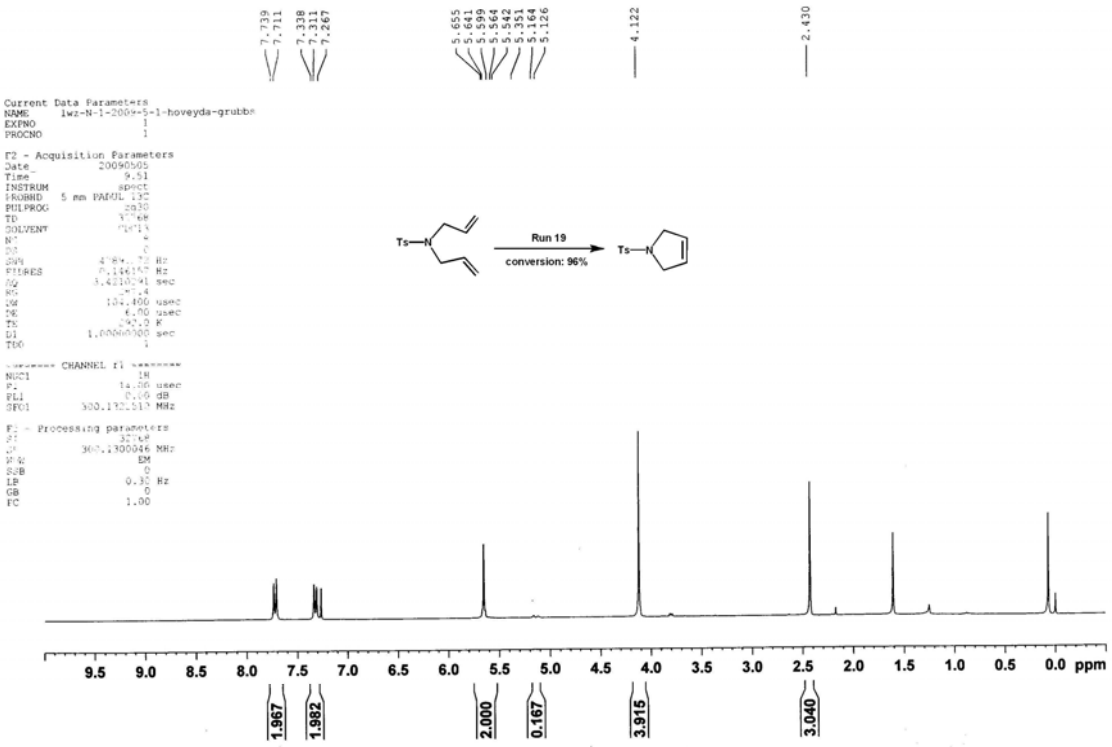
sample name:N-1-2009-5-1-hoveyda-grubbs-H17



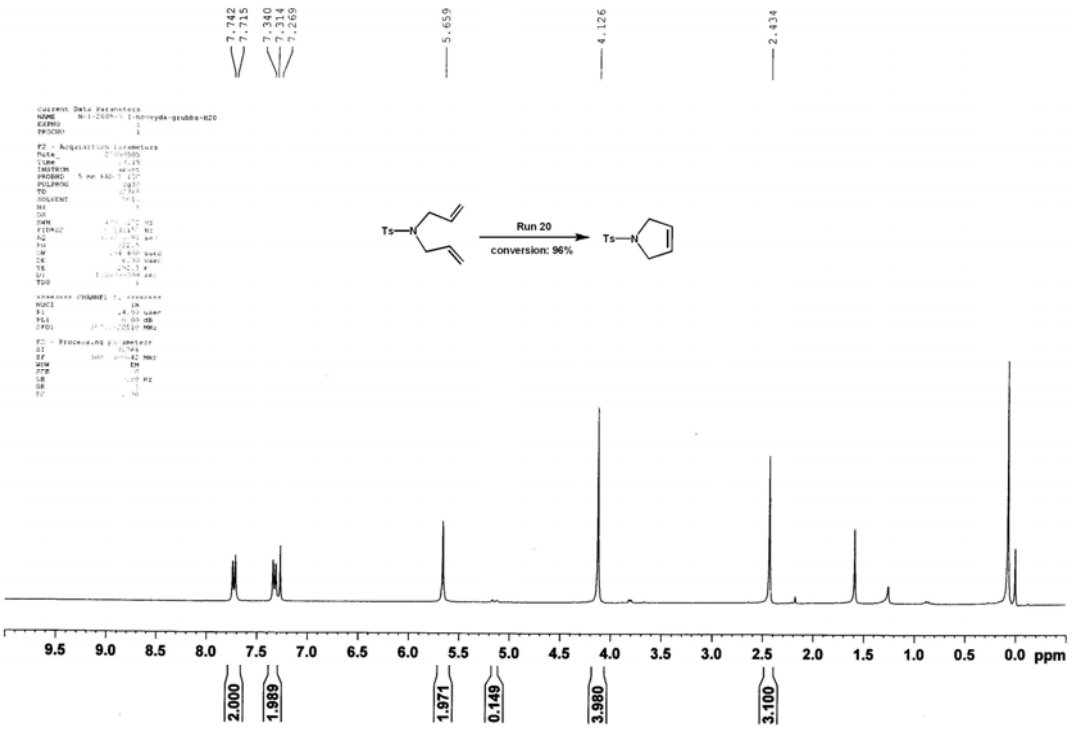
sample name:N-1-2009-5-1-hoveyda-grubbs-H18



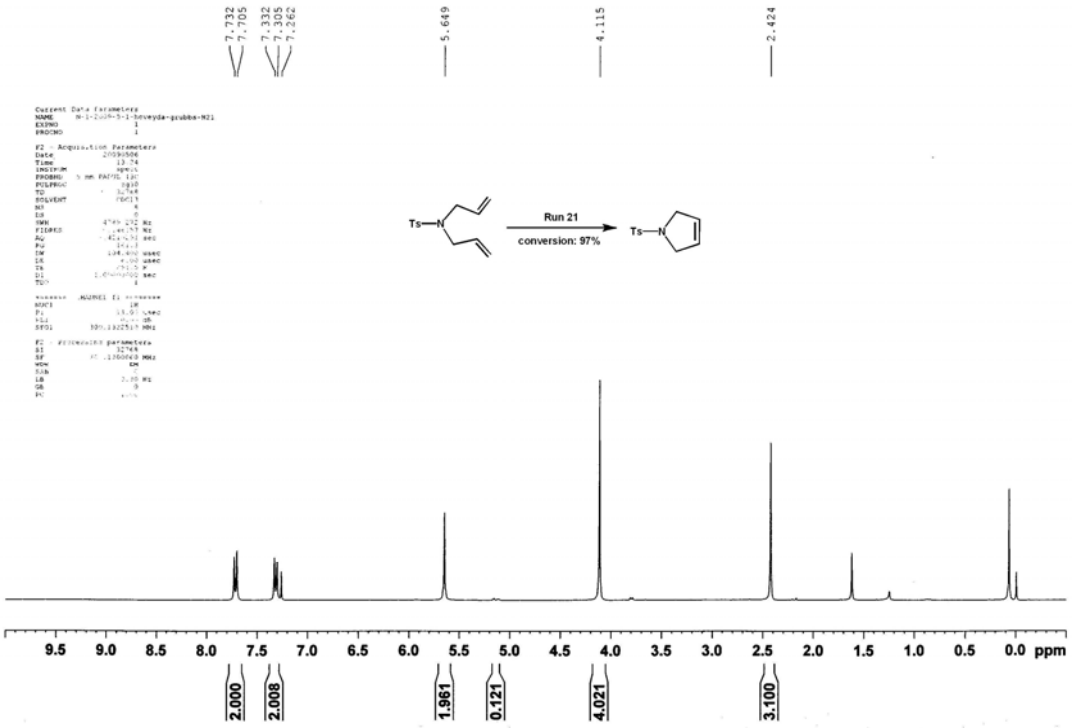
sample name:N-1-2009-5-1-hoveyda-grubbs-H19



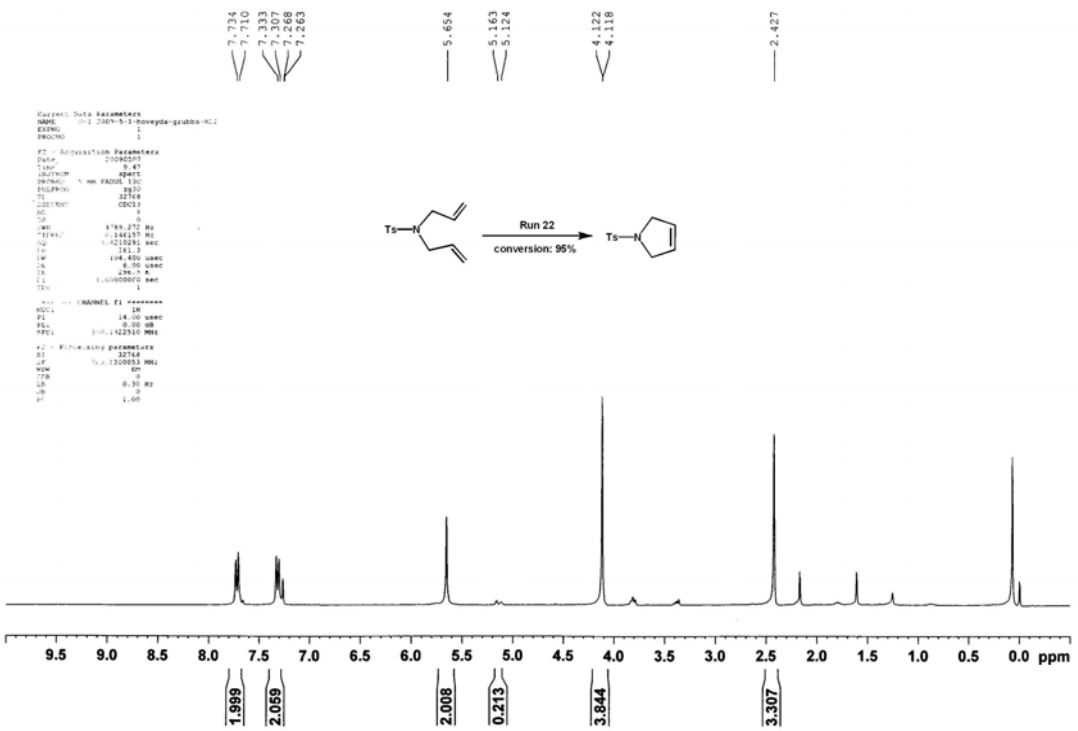
sample name:N-1-2009-5-1-hoveyda-grubbs-H20



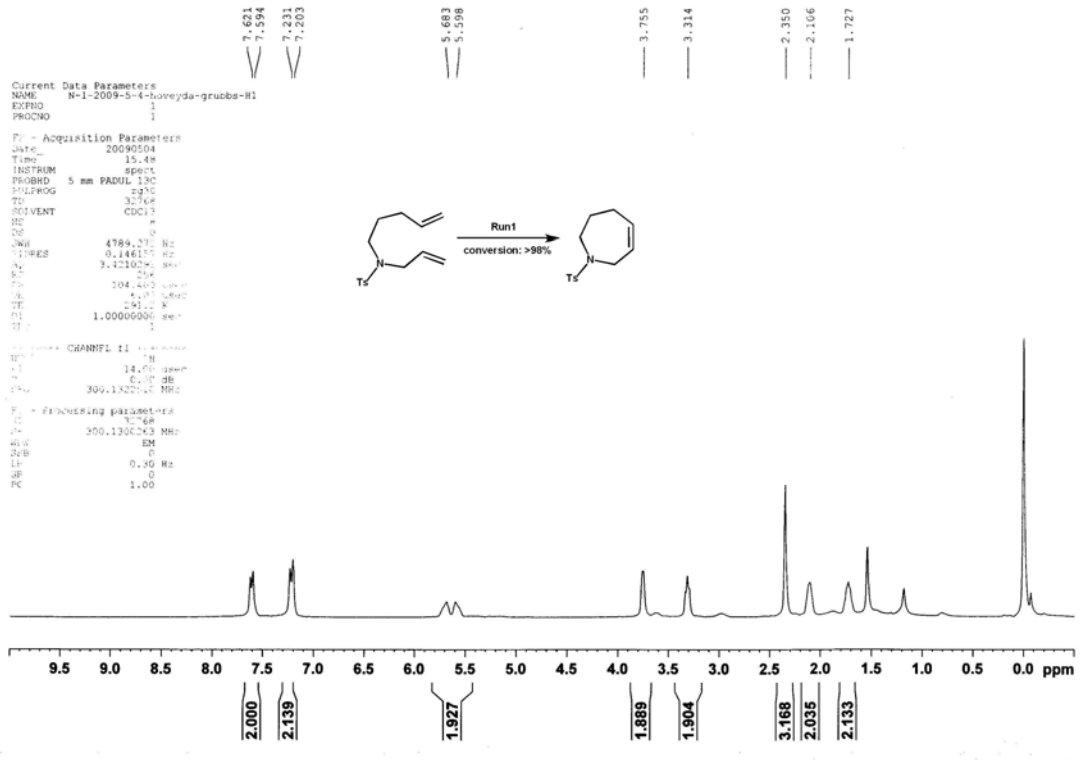
sample name:N-1-2009-5-1-hoveyda-grubbs-H21

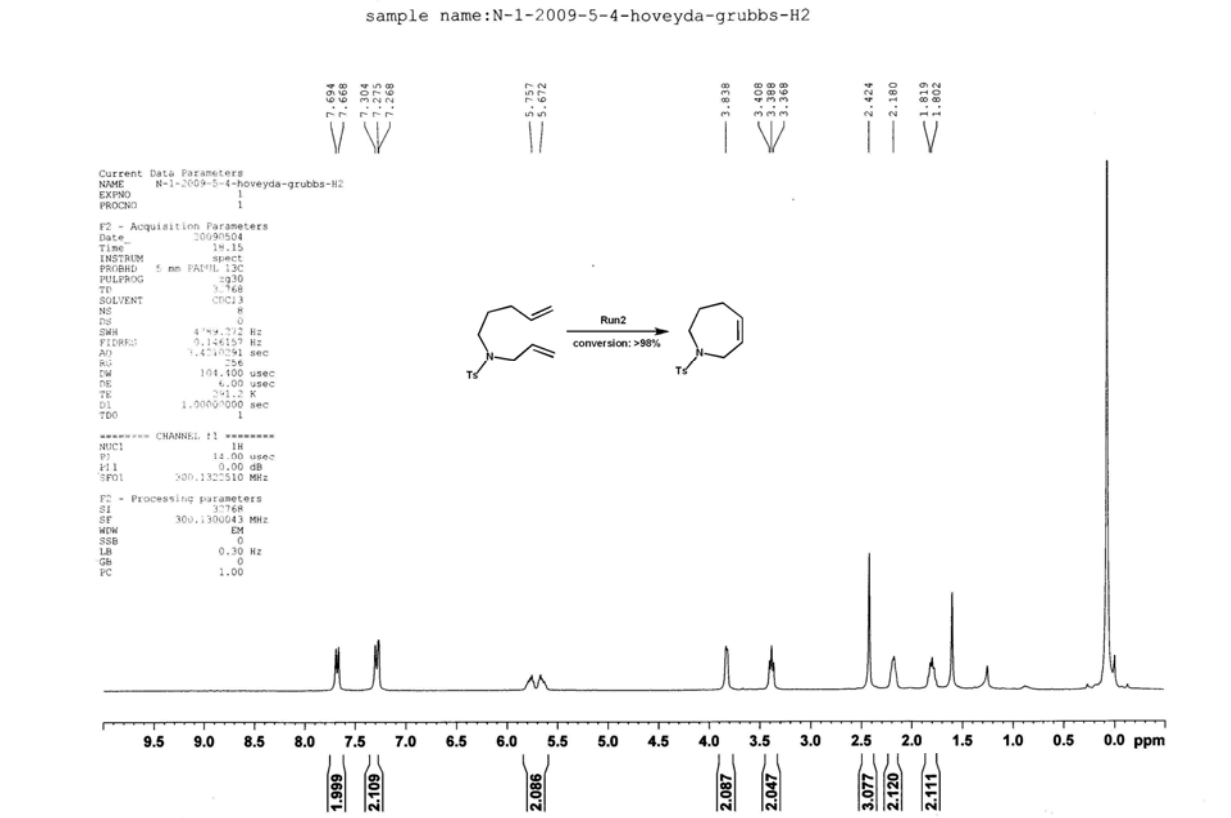


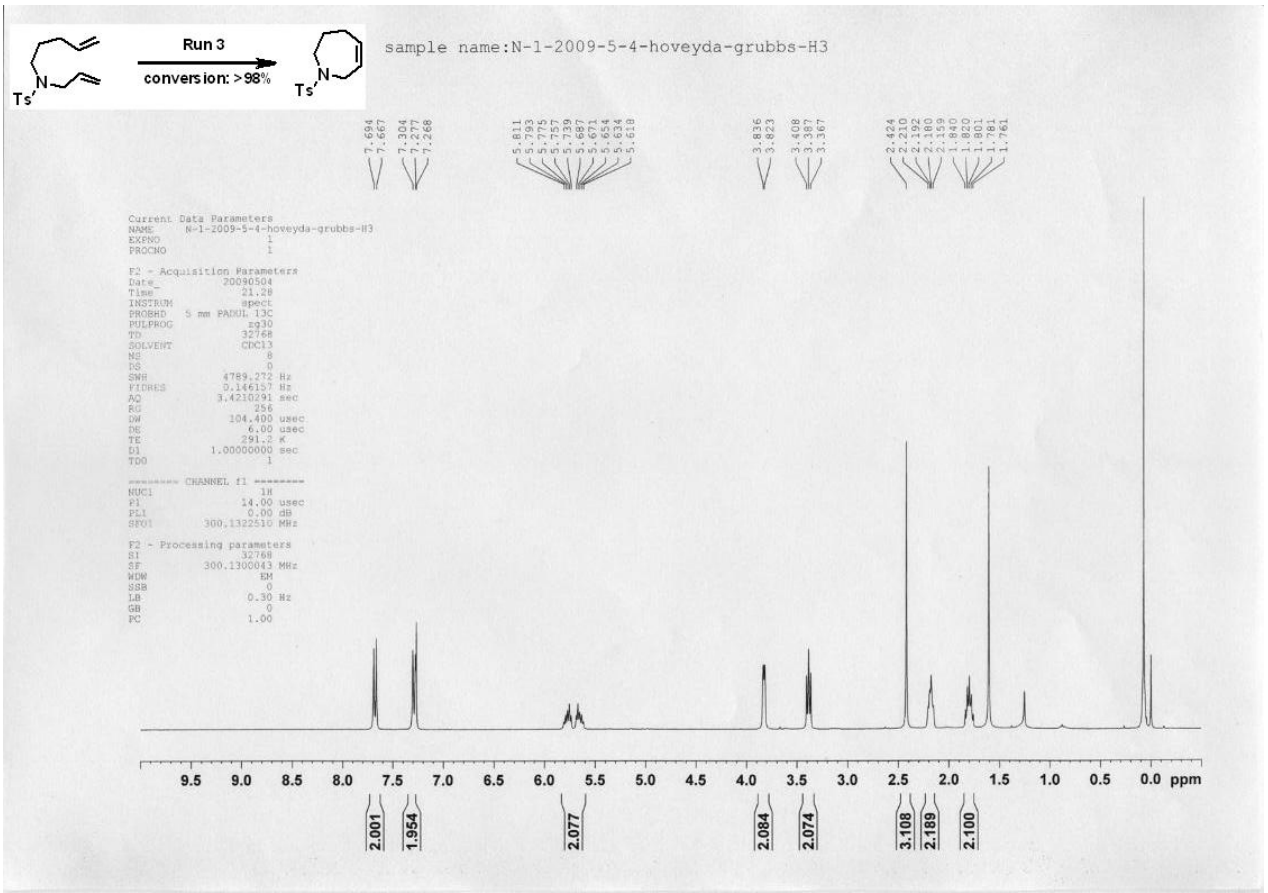
sample name:N-1-2009-5-1-hoveyda-grubbs-H22



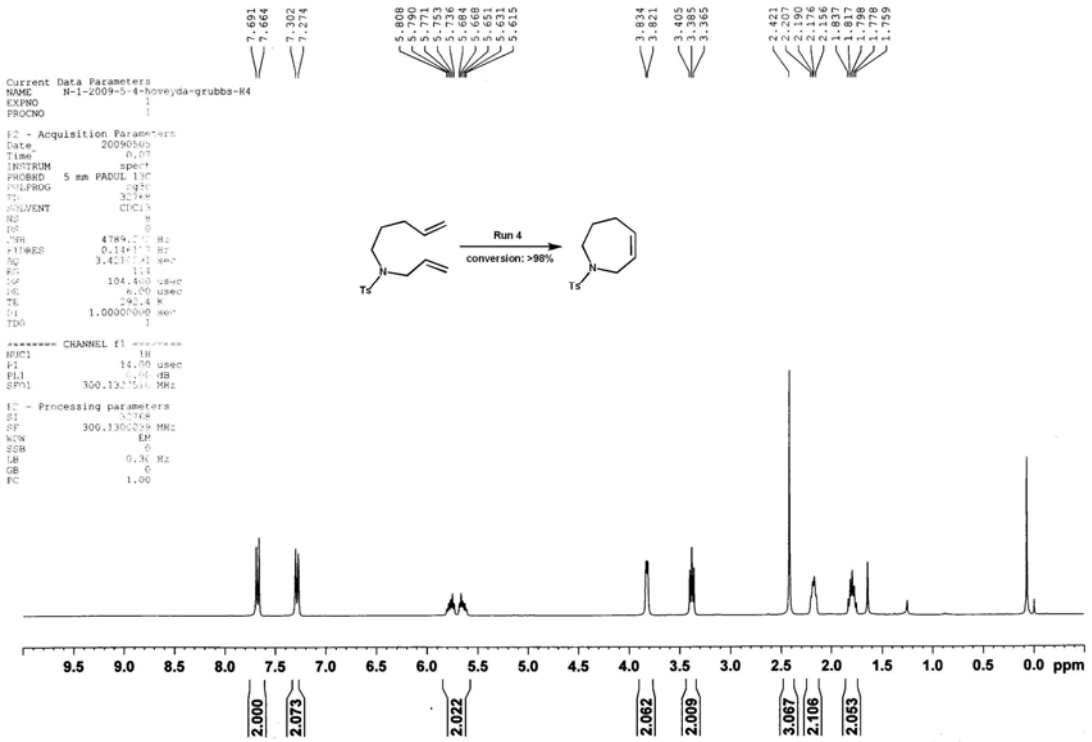
sample name:N-1-2009-5-4-hoveyda-grubbs-H1



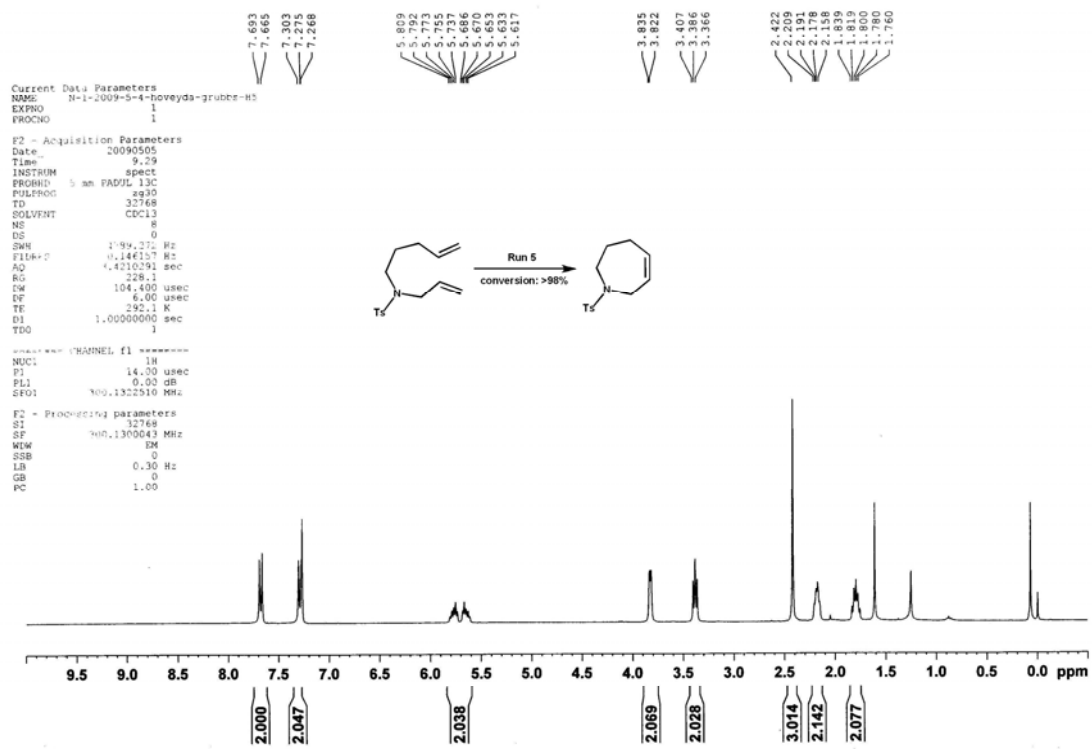

sample name:N-1-2009-5-4-hoveyda-grubbs-H2
Run2
conversion: >98%
Ts
Current Data Parameters
NAME N-1-2009-5-4-hoveyda-grubbs-H2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090504
Time 14.15
INSTRUM spect
PROBHD 5 mm PABBL 13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 256
DW 104.400 usec
DE 6.00 usec
TE 291.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 300.1322510 MHz
F2 - Processing parameters
SI 32768
SF 300.1300043 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.694
7.668
7.304
7.275
7.268
5.757
5.672
3.838
3.408
3.388
3.368
2.424
2.180
1.819
1.802
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.999
2.109
2.086
2.087
2.047
3.077
2.120
2.111


Run 3
conversion: >98%
Ts
sample name:N-1-2009-5-4-hoveyda-grubbs-H3
Current Data Parameters
NAME N-1-2009-5-4-hoveyda-grubbs-H3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090504
Time 21.28
INSTRUM spect
PROBHD 5 mm PABBL 13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 256
DW 104.400 usec
DE 6.00 usec
TE 291.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 300.1322510 MHz
F2 - Processing parameters
SI 32768
SF 300.1300043 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.694
7.667
7.304
7.277
7.268
5.811
5.793
5.775
5.757
5.739
5.687
5.671
5.654
5.634
5.618
3.836
3.823
3.408
3.387
3.367
2.424
2.210
2.192
2.180
2.159
1.840
1.820
1.801
1.781
1.761
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.001
1.954
2.077
2.084
2.074
3.108
2.189
2.100

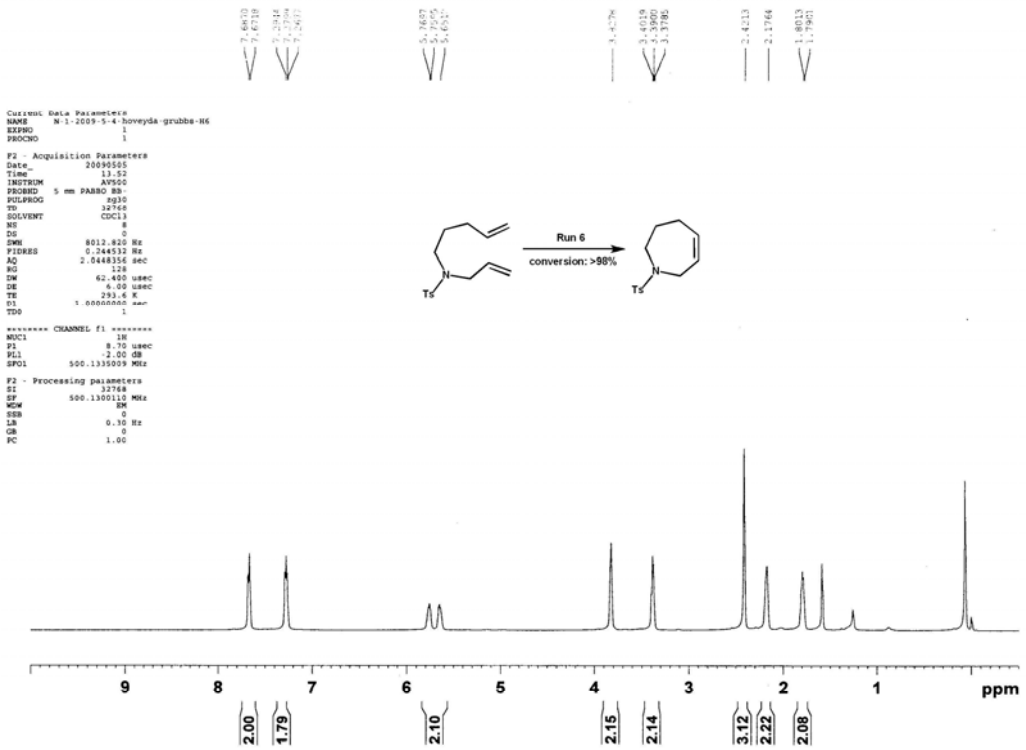
sample name:N-1-2009-5-4-hoveyda-grubbs-H4



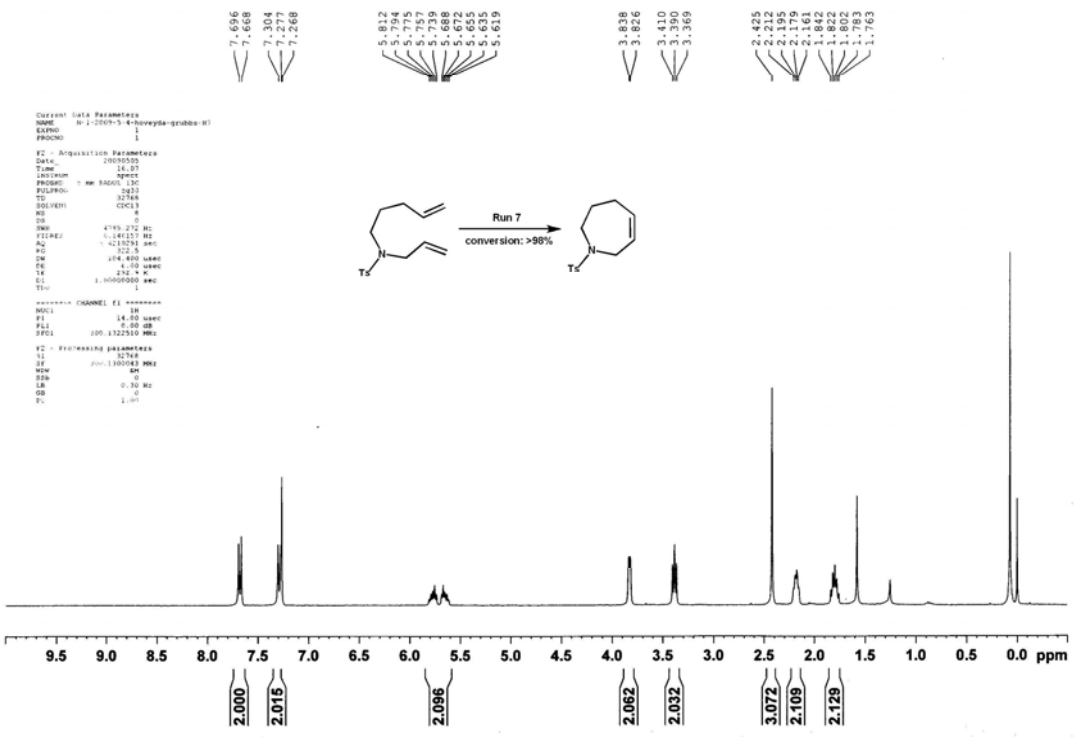
sample name:N-1-2009-5-4-hoveyda-grubbs-H5



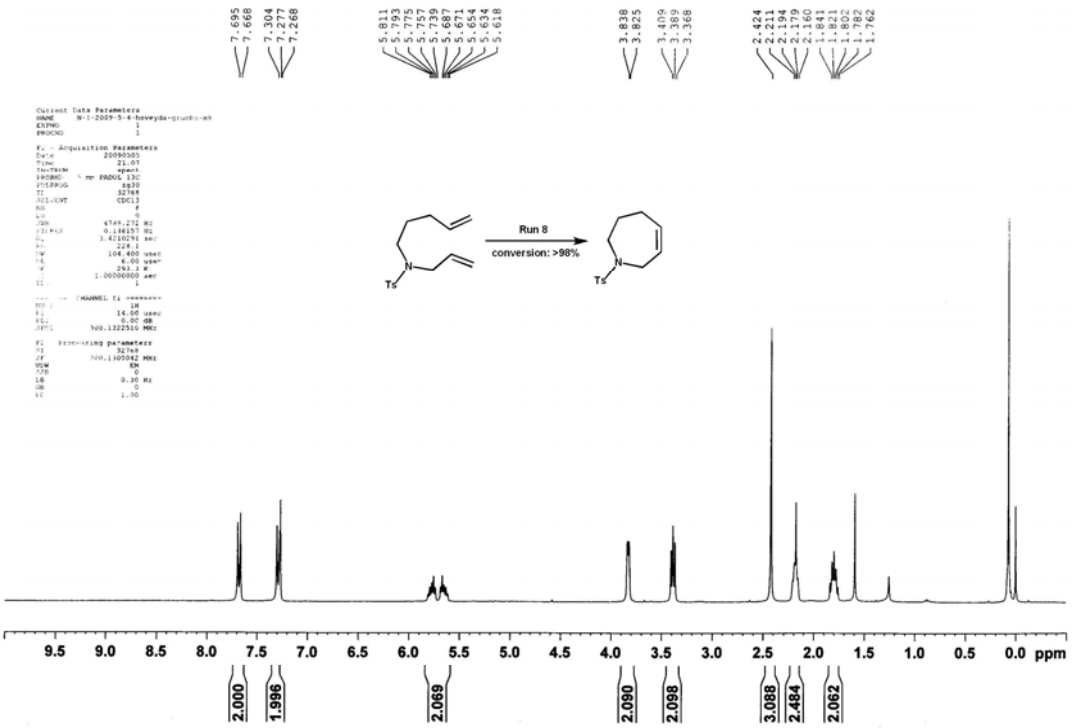
Sample name:N-1-2009-5-4-hoveyda-grubbs-H6



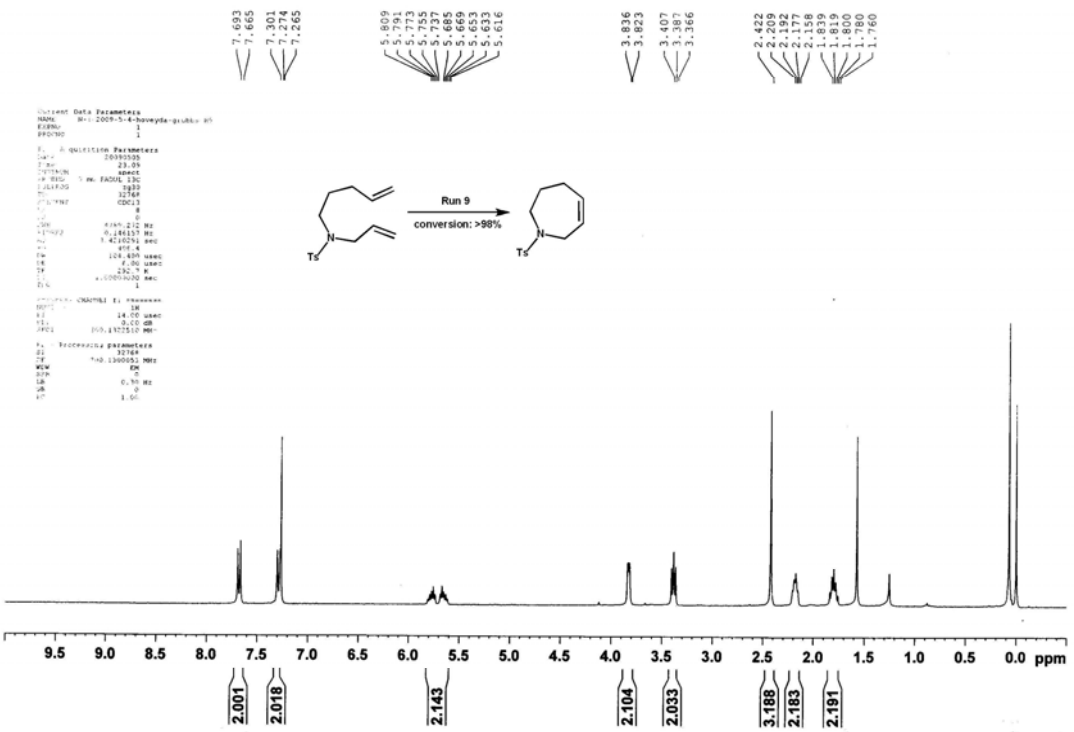
sample name:N-1-2009-5-4-hoveyda-grubbs-H7



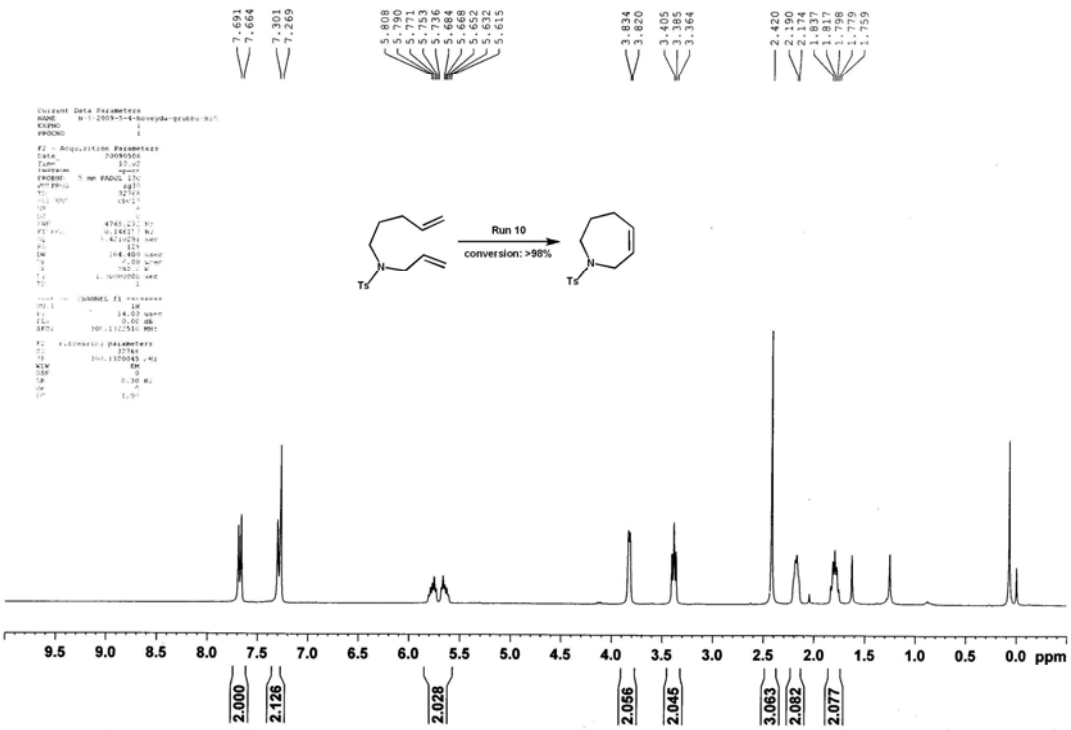
sample name:N-1-2009-5-4-hoveyda-grubbs-H78



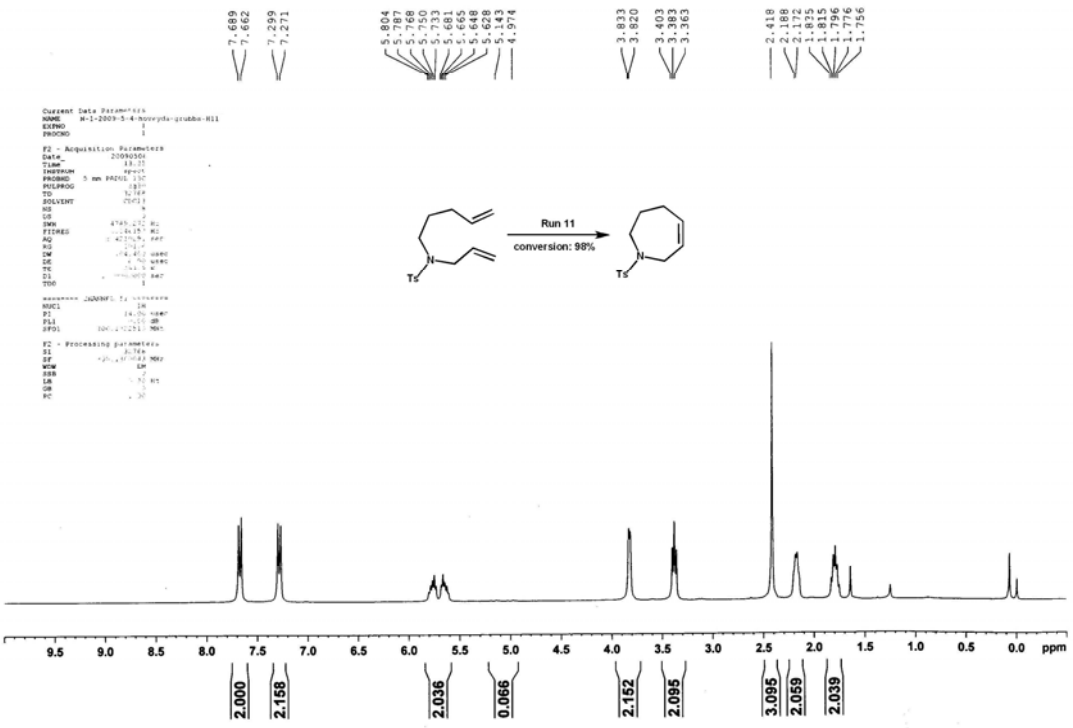
sample name:N-1-2009-5-4-hoveyda-grubbs-H9



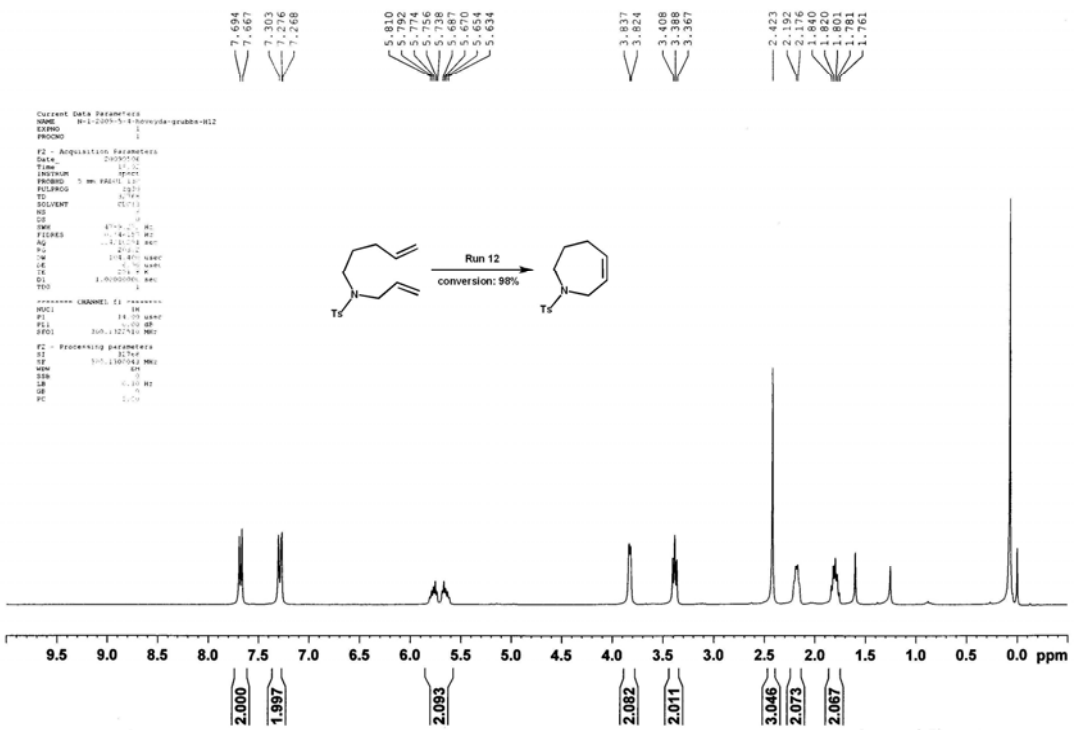
sample name:N-1-2009-5-4-hoveyda-grubbs-H10



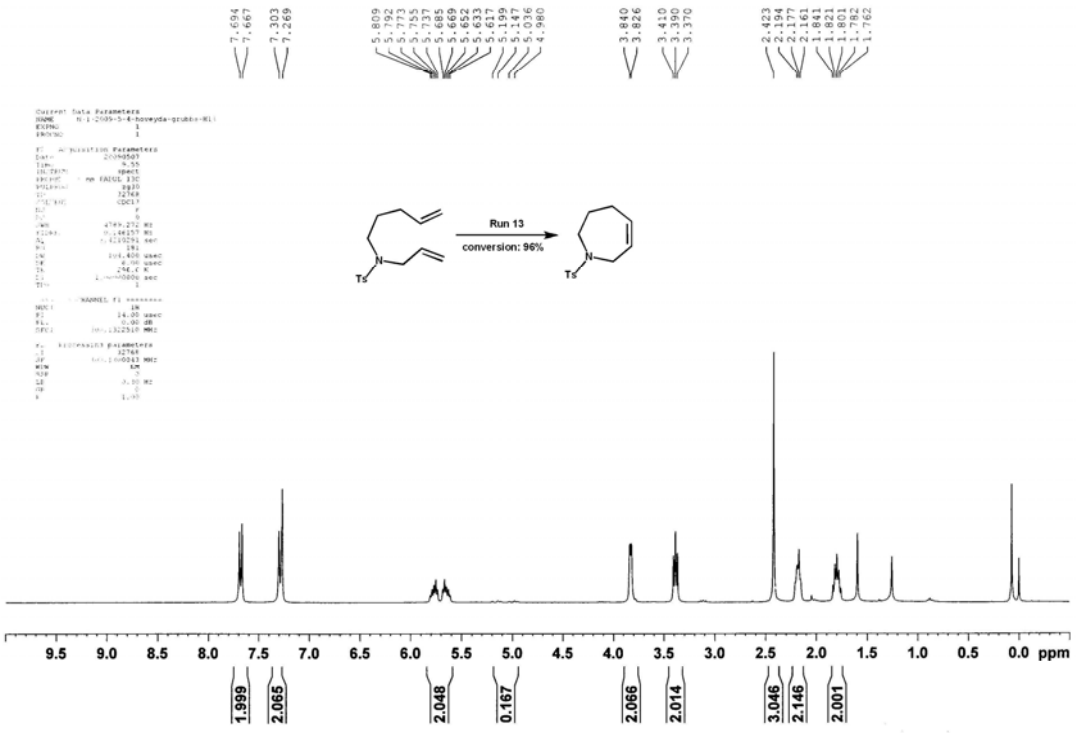
sample name:N-1-2009-5-4-hoveyda-grubbs-H11



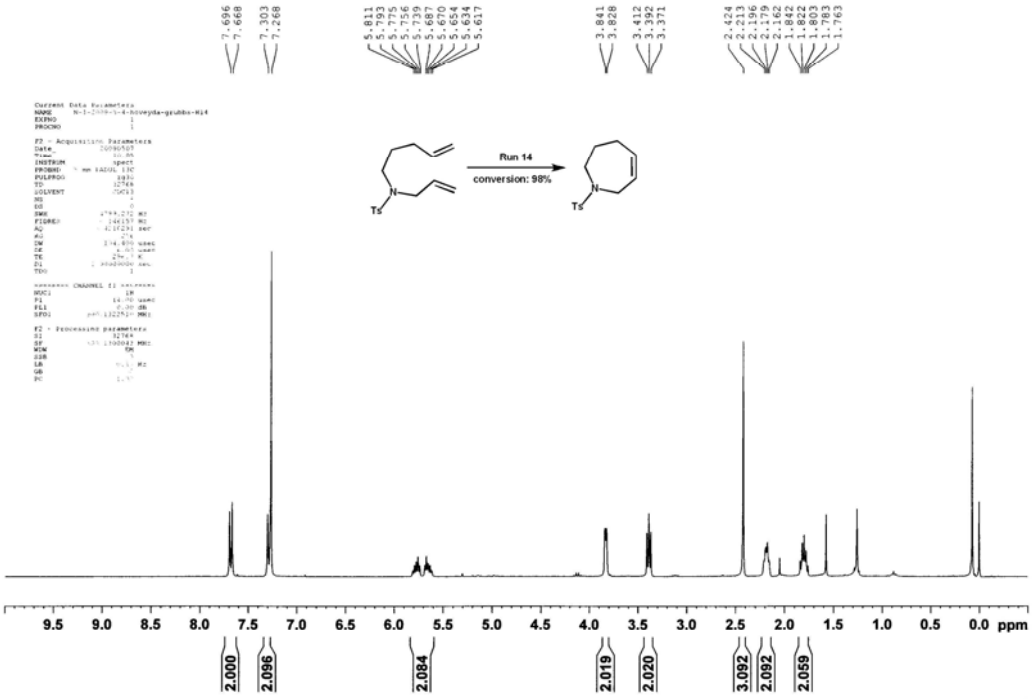
sample name:N-1-2009-5-4-hoveyda-grubbs-H12



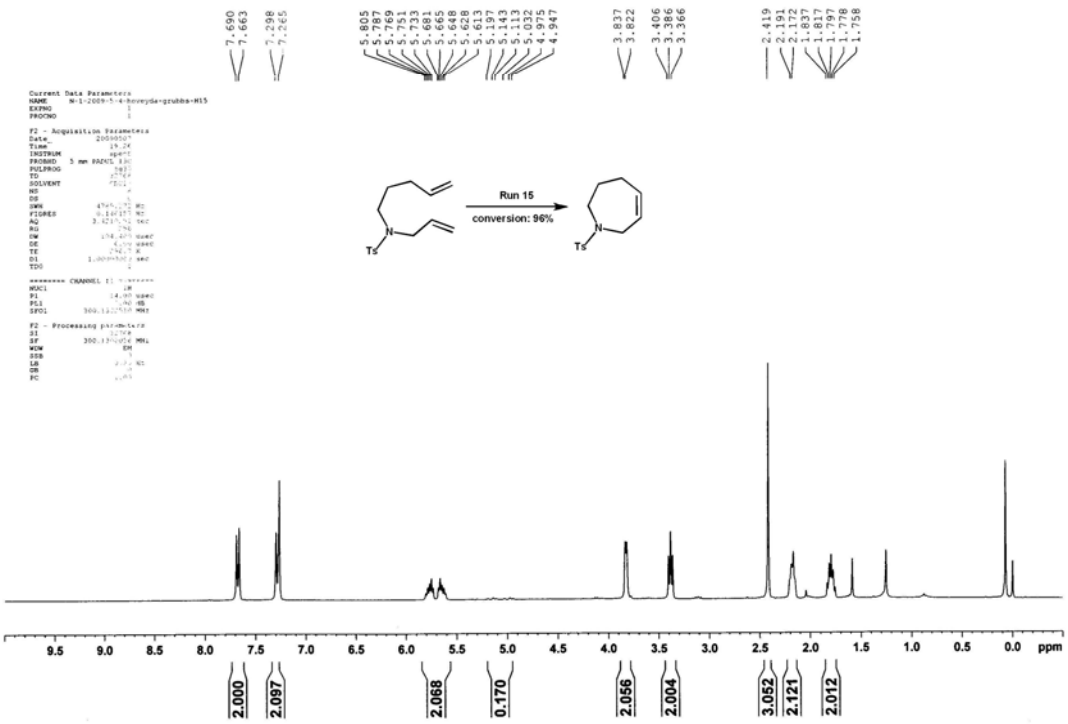
sample name:N-1-2009-5-4-hoveyda-grubbs-H13



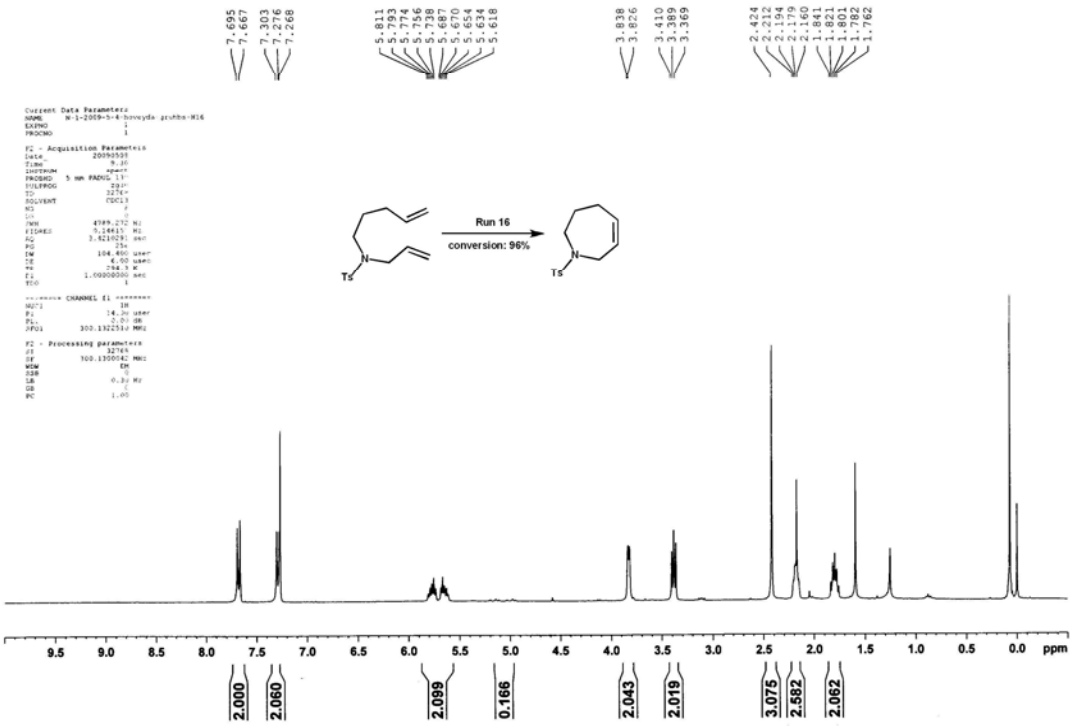
sample name:N-1-2009-5-4-hoveyda-grubbs-H14



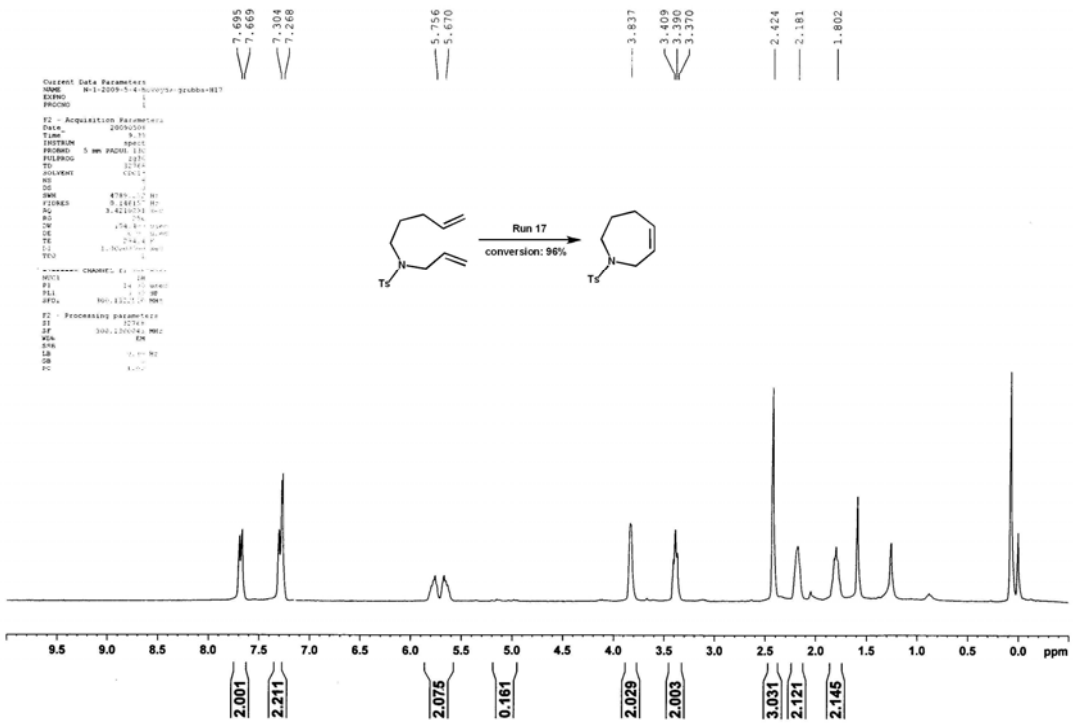
sample name:N-1-2009-5-4-hoveyda-grubbs-H15

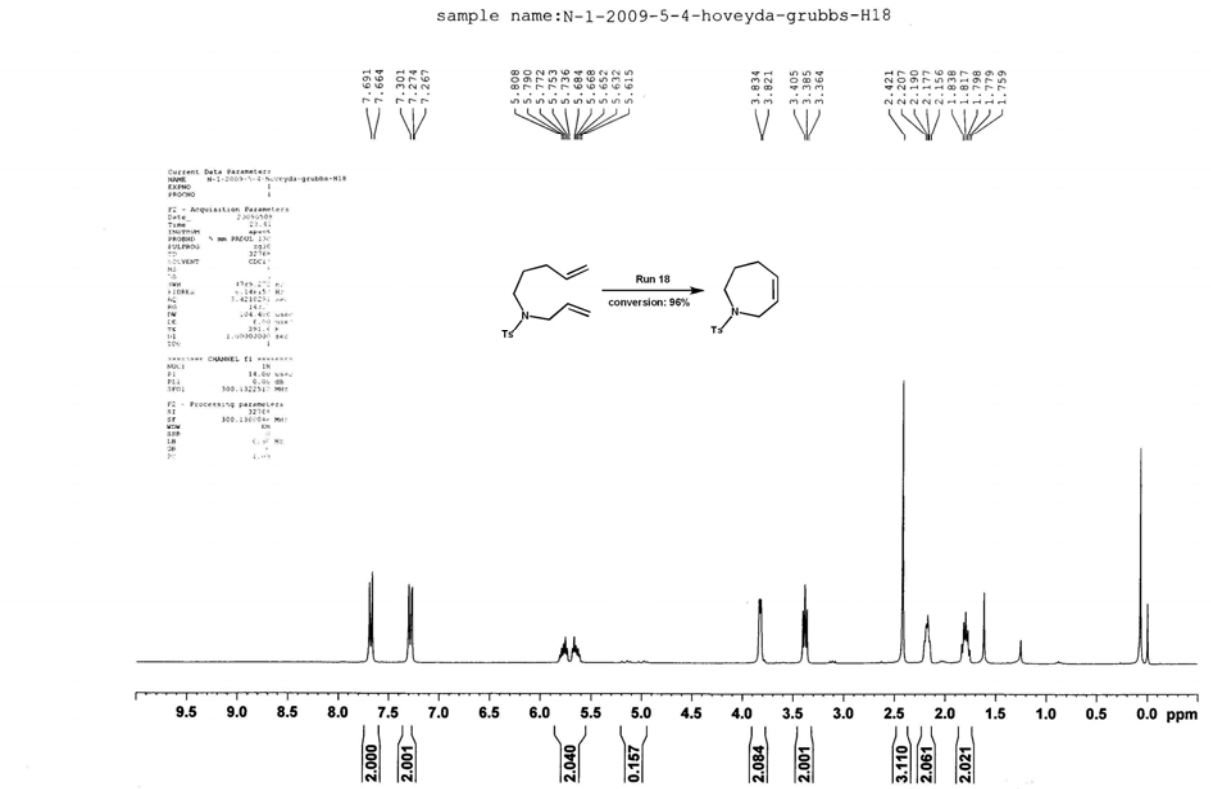


sample name:N-1-2009-5-4-hoveyda-grubbs-H16



sample name:N-1-2009-5-4-hoveyda-grubbs-H17




sample name:N-1-2009-5-4-hoveyda-grubbs-H18
7.691
7.664
7.301
7.274
7.267
5.808
5.790
5.772
5.753
5.736
5.684
5.668
5.652
5.632
5.615
3.834
3.821
3.405
3.385
3.364
2.421
2.407
2.190
2.177
2.156
1.838
1.817
1.798
1.779
1.759
Run 18
conversion: 96%
Ts
N
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.000
2.001
2.040
0.157
2.084
2.001
3.110
2.061
2.021