

Supporting Information for

A Quick Access to the Spirotricyclic Core Analogue of Mangicol A by a Rh(I)-Catalyzed Tandem Pauson-Khand/[4+2] Cycloaddition

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I. Analytical Instruments:

NMR: Varian 300 MHz, and Bruker 600 MHz.

HPLC: Waters 600 Pump and Waters 486 Tunable Absorbance Detector, Chiralcel OD, OD-H, or Chiralpak AD-H column.

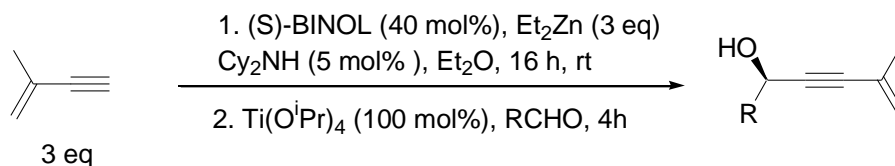
Polarimeter: Jasco Digital Polarimeter P-2000.

High resolution mass spectra were obtained by EI [70-VSE(C)] or ESI (Q-ToF) analysis and the [M] or [MH⁺] was observed

II. General Data:

All commercial chemicals were used as received unless otherwise noted. Cy₂NH was distilled prior to use. [Rh(CO)₂Cl]₂ was purchased and stored in dry nitrogen atmosphere. THF was distilled over sodium and benzophenone under nitrogen atmosphere. Diethyl ether were dried by passing through activated alumina columns under nitrogen. All aldehydes were distilled from 4 Å molecular sieves prior to use and stored under nitrogen atmosphere. All the NMR spectra were obtained in CDCl₃ unless indicated otherwise.

III. Synthesis and Characterization of the Optically Active Propargylic Alcohols (*R*)-1a,b and (*R*)-12



General procedure for the (S)-BINOL Catalyzed Enantioselective Enyne Additions. Under nitrogen 2-methyl-1-buten-3-yne (0.75 mmol, 3 equiv) was weighed into a tared flask and dissolved

in Et₂O (3 mL). (*S*)-BINOL (>99% ee, 28.8 mg, 0.025 mmol, 40 mol%), Cy₂NH (2.5 μL, 0.0125 mmol, 5 mol%), Et₂Zn (76.9 μL, 0.5 mmol, 3 equiv) were added, and the reaction mixture was stirred for 16 h. Then, Ti(O^{*i*}Pr)₄ (74 μL, 0.25 mmol, 100 mol%) was added followed by the addition of an aldehyde (0.25 mmol, 1 equiv). The resulting solution was stirred for 3 h during which the aldehyde was judged to be consumed by TLC. The reaction was quenched with saturated aqueous ammonium chloride (5 mL), and extracted three times with CH₂Cl₂. The organic layer was dried with sodium sulfate and concentrated by rotary evaporation. The resultant oil was purified by flash column chromatography on silica gel, eluted with hexanes/ethyl acetate (4-9% ethyl acetate) to give the product.

2-Methylnona-1,8-dien-3-yn-5-ol, (R)-1a. Colorless oil, 75% yield. 82~90% ee determined by HPLC analysis: Chiralpak AD-H column, 98:2 hexanes : ^{*i*}PrOH, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_{major} = 13.7, t_{minor} = 12.7. [α]_D²³ = -14.4 (c = 1.50, CHCl₃). ¹H NMR (300 MHz, CDCl₃) δ 5.84 (m, 1H), 5.28 (s, 1H), 5.22 (s, 1H), 5.07 (m, 1H), 4.99 (m, 1H), 4.51 (t, 1H, J = 6.6 Hz), 2.22 (m, 2H), 1.94 (br, 1H), 1.88 (m, 3H), 1.82 (m, 2H). ¹³C NMR (75 MHz, CDCl₃) δ 137.9, 126.4, 122.4, 115.5, 89.0, 86.5, 62.5, 37.0, 29.7, 23.6. HRMS (ESI) for C₁₀H₁₅O (MH⁺) Calcd: 151.1123, Found: 151.1120.

2,6,6-Trimethylnona-1,8-dien-3-yn-5-ol, (R)-1b. Colorless oil, 85% yield. 89% ee determined by HPLC analysis: Chiralpak AD-H column, 98:2 hexanes : ^{*i*}PrOH, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_{major} = 12.0, t_{minor} = 11.0. [α]_D²³ = -16.5 (c = 1.9, CHCl₃). ¹H NMR (300 MHz, CDCl₃) δ 5.85 (m, 1H), 5.28 (d, 1H, J = 0.3 Hz), 5.22 (m, 1H), 5.09 (m, 1H), 5.05 (s, 1H), 5.18 (d, 1H, J = 6.3 Hz), 2.14 (m, 2H), 1.91 (d, 1H, J = 6.3 Hz), 1.89 (s, 3H), 0.98 (s, 3H), 0.97 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 135.2, 126.6, 122.2, 117.9, 87.9, 87.5, 70.7, 43.0, 39.1, 23.7, 22.9, 22.8. HRMS (ESI) for C₁₂H₁₉O (MH⁺) Calcd: 179.1436, Found: 179.1438.

2-Methyl-9-(trimethylsilyl)nona-1-en-3,8-diyn-5-ol, (R)-12. Colorless oil, 83% yield. 88% ee determined by HPLC analysis: Chiralpak AD-H column, 97:3 hexanes : ^{*i*}PrOH, flow rate = 1.0 mL/min, λ = 225 nm, retention time: t_{major} = 8.3, t_{minor} = 7.4. [α]_D²⁴ = -25.4 (c = 0.88, CHCl₃). ¹H NMR (300 MHz, CDCl₃) δ 5.29 (s, 1H), 5.23 (m, 1H), 4.63 (q, 1H, J = 6.0 Hz), 2.53-2.33 (m, 2H), 2.11 (d, 1H, J = 5.7 Hz), 1.93 (q, 2H, J = 6.9 Hz), 1.88 (t, 3H, J = 1.2 Hz), 0.14 (s, 9H). ¹³C NMR (75 MHz, CDCl₃) δ 126.3, 122.6, 106.3, 88.4, 86.7, 85.7, 62.0, 36.6, 23.6, 16.2, 0.3. HRMS (ESI) for C₁₃H₂₁OSi (MH⁺) Calcd: 221.1362, Found: 221.1362.

IV. Preparation and Characterization of the Optically Active Triene-yne (*R*)-2a-c and Diene-diyne (*R*)-13

(R)-5-(Allyloxy)-2-methylnona-1,8-dien-3-yne, (R)-2a. Under nitrogen, KOH powder (134.4 mg, 2.4 mmol, 2 equiv) was weighted and dissolved in DMSO (5 mL) in around bottom flask. The optical active propargylic alcohol (*R*)-1a (83~90% ee) (180 mg, 1.2 mmol) was added into the solution followed by allyl bromide (0.16 ml, 1.8 mmol, 1.5 equiv). Then, the mixture was stirred at room temperature until the reaction was determined to be complete by TLC (2 h). The reaction mixture was loaded directly onto silica gel column eluted with hexane to give (*R*)-2a as a colorless liquid in 81% yield (184 mg). [α]_D²³ = 79.8 (c = 0.41, CHCl₃). ¹H NMR (300 MHz, CDCl₃) δ 5.99-5.75 (m, 2H), 5.33-5.17 (m, 4H), 5.08-4.96 (m, 2H), 4.28-4.18 (m, 2H), 3.96 (dd, 1H, J = 12.6, 6.3 Hz), 2.23 (q, 2H, J = 7.5 Hz), 1.89 (d, 3H, J = 0.6 Hz), 1.84 (m, 2H). ¹³C NMR

(75 MHz, CDCl₃) δ 138.0, 134.8, 126.6, 122.1, 117.5, 115.3, 87.4, 87.3, 69.8, 68.7, 35.1, 29.7, 23.7. HRMS (EI) for C₁₃H₁₈O (M) Calcd: 190.1358, Found: 190.1353.

(R)-5-(Allyloxy)-2,6,6-trimethylnona-1,8-dien-3-yne, (R)-2b. By following the same procedure used to synthesize (R)-2a, compound (R)-2b was prepared as a colorless liquid in 72% yield (128 mg) from the optical active propargylic alcohol (R)-1b (178 mg, 1 mmol), KOH powder (112 mg, 2 mmol, 2 equiv) and allyl bromide (0.13 mL, 1.5 mmol, 1.5 equiv). $[\alpha]_D^{24} = 54.7$ (c = 0.73, CHCl₃). ¹H NMR (300 MHz, CDCl₃) δ 5.98-5.75 (m, 2H), 5.34-5.15 (m, 4H), 5.06 (s, 1H), 5.02 (m, 1H), 4.28 (m, 1H), 3.91 (m, 1H), 3.85 (s, 1H), 2.16 (m, 1H), 1.90 (m, 3H), 1.00 (s, 3H), 0.97 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 135.2, 135.0, 126.8, 121.8, 117.6, 117.0, 88.2, 86.3, 76.9, 70.1, 43.4, 38.7, 23.8, 23.5, 23.2. HRMS (EI) for C₁₅H₂₂O (M) Calcd: 218.1671, Found: 218.1664.

(R)-5-(Allyloxy)-8-methylnona-8-en-1,6-diyne-1-yl)trimethylsilane, (R)-13. Under nitrogen the optical active propargylic alcohol (R)-12 (330 mg, 1.5 mmol) was dissolved in dry THF (7.5 mL) and cooled to -78 °C. A solution of ⁿBuLi (2.5 M in hexane) (0.6 mL, 1.5 mmol, 1 equiv) was added and the mixture was stirred for 10 min. Allyl bromide (1.04 mL, 12 mmol, 8 equiv) was then added, followed by the addition of DMSO (0.21 mL, 3 mmol, 2 equiv). The reaction flask was allowed to warm to room temperature over ~12 h. Upon consumption of the starting material, the reaction was quenched with saturated aqueous ammonium chloride (10 mL), extracted three times with CH₂Cl₂, dried with sodium sulfate, and concentrated by rotary evaporation. The resultant oil was purified by flash column chromatography on silica gel eluted with hexanes to give (R)-13 as a colorless oil in 72% yield (281 mg). $[\alpha]_D^{23} = 27.9$ (c = 1.71, CHCl₃). ¹H NMR (300 MHz, CDCl₃) δ 5.92 (m, 1H), 5.33-5.16 (m, 4H), 4.32 (t, 1H, J = 6.0 Hz), 4.23 (ddt, 1H, J = 12.6, 5.1, 1.5 Hz), 3.97 (ddt, 1H, J = 12.6, 6.3, 1.2 Hz), 2.40 (m, 2H), 1.94 (m, 2H), 1.88 (m, 3H), 0.13 (s, 9H). ¹³C NMR (75 MHz, CDCl₃) δ 134.6, 126.5, 122.4, 117.5, 106.5, 87.4, 86.7, 85.2, 69.9, 67.7, 34.9, 23.7, 16.2, 0.3. HRMS (ESI) for C₁₆H₂₅O (MH⁺) Calcd: 261.1675, Found: 261.1673.

V. Experimental Procedure for the Rh(I)-Catalyzed Tandem Pauson-Khand/[4+2] Cycloaddition and Characterization of the Products

General procedure. Under nitrogen, a triene-yne or diene-diyne (0.25 mmol) and [Rh(CO)₂Cl]₂ (9.75 mg, 0.10 equiv) were weighed into a tared two-necked round bottom flask and dissolved in DCE (5 mL). The flask was fitted with reflux condenser fit with a septum and the side arm of the flask was also fitted with septum. The solution was bubbled with CO gas for 2 minutes through the side arm fitted with septum and a vent needle in the septum of the reflux condenser. Then, the solution was placed under CO atmosphere by using a balloon. After the reaction mixture was heated at 65 °C to reflux temperature for 26 – 72 h, it was cooled to room temperature and the CO was released cautiously in the hood. The reaction mixture was concentrated and the crude product was purified by column chromatography on silica gel.

Characterizations of the Products 3a,b, 4b and 14.

3a: Colorless oil, 75% yield. 90% ee determined by HPLC analysis: Chiralpak AD-H column, 95:5 hexanes : ¹PrOH, flow rate = 1.0 mL/min, $\lambda = 254$ nm, retention time: $t_{\text{major}} = 15.8$, $t_{\text{minor}} =$

13.3. $[\alpha]_D^{23} = -85.2$ ($c = 0.96$, CHCl_3). $^1\text{H NMR}$ (600 MHz, CDCl_3) δ 4.15 (d, 1H, $J = 4.2$ Hz), 4.02 (dd, 1H, $J = 9.0, 4.8$ Hz), 3.78 (d, 1H, $J = 9.0$ Hz), 2.58 (dd, 1H, $J = 18.6, 9.6$ Hz), 2.47 (m, 1H), 2.20 (dd, 1H, $J = 18.6, 7.2$ Hz), 2.19 (m, 1H), 2.16 (s, 3H), 2.11-2.03 (m, 3H), 1.85 (m, 1H), 1.76 (ddt, 1H, $J = 13.8, 7.8, 1.2$ Hz), 1.59-1.54 (m, 2H), 1.53-1.47 (m, 1H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) δ 205.9, 148.7, 134.5, 90.7, 73.7, 62.5, 46.2, 43.8, 41.9, 33.1, 32.6, 31.4, 27.9, 19.8. HRMS (EI) for $\text{C}_{14}\text{H}_{18}\text{O}_2$ (M) Calcd: 218.1307, Found: 218.1321

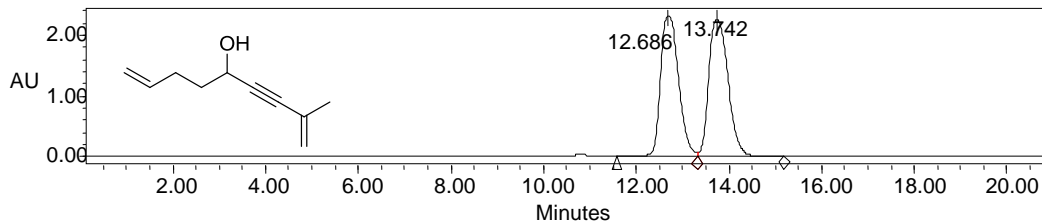
3b: white solid, 56% yield. 83% ee determined by HPLC analysis: Chiralpak AD-H column, 95:5 hexanes : $^i\text{PrOH}$, flow rate = 1.0 mL/min, $\lambda = 254$ nm, retention time: $t_{\text{major}} = 16.3$, $t_{\text{minor}} = 13.2$. $[\alpha]_D^{23} = -52.6$ ($c = 0.40$, CHCl_3). mp 83-84 °C. $^1\text{H NMR}$ (600 MHz, CDCl_3) δ 4.15 (d, 1H, $J = 4.2$ Hz), 4.02 (dd, 1H, $J = 9.0, 4.8$ Hz), 3.78 (d, 1H, $J = 9.0$ Hz), 2.58 (dd, 1H, $J = 18.6, 9.6$ Hz), 2.47 (m, 1H), 2.20 (dd, 1H, $J = 18.6, 7.2$ Hz), 2.19 (m, 1H), 2.16 (s, 3H), 2.11-2.03 (m, 3H), 1.85 (m, 1H), 1.76 (ddt, 1H, $J = 13.8, 7.8, 1.2$ Hz), 1.59-1.54 (m, 2H), 1.53-1.47 (m, 1H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) δ 205.9, 148.7, 134.5, 90.7, 73.7, 62.5, 46.2, 43.8, 41.9, 33.1, 32.6, 31.4, 27.9, 19.8. HRMS (ESI) for $\text{C}_{16}\text{H}_{23}\text{O}_2$ (MH+) Calcd: 247.1698, Found: 247.1690.

4b: Colorless oil, 68% yield. $^1\text{H NMR}$ (600 MHz, CDCl_3) δ 5.83 (m, 1H), 5.21 (m, 1H), 5.09-5.02 (m, 3H), 4.59 (s, 1H), 4.35 (dd, 1H, $J = 7.8, 0.6$ Hz), 3.25 (dd, 1H, $J = 11.1, 7.5$ Hz), 3.19 (m, 1H), 2.66 (dd, 1H, $J = 18.0, 6.6$ Hz), 2.20 (dd, 1H, $J = 13.2, 7.8$ Hz), 2.12 (ddd, 1H, $J = 18.0, 3.6, 0.6$ Hz), 2.00 (dd, 1H, $J = 13.2, 7.2$ Hz), 1.93 (t, 3H, $J = 1.2$ Hz), 0.93 (s, 3H), 0.91 (s, 3H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) δ 207.3, 176.9, 139.9, 136.6, 134.4, 117.9, 117.2, 82.5, 71.4, 43.4, 43.2, 39.3, 39.2, 23.3, 23.2, 21.8. HRMS (ESI) for $\text{C}_{16}\text{H}_{23}\text{O}_2$ (MH+) Calcd: 247.1698, Found: 247.1694.

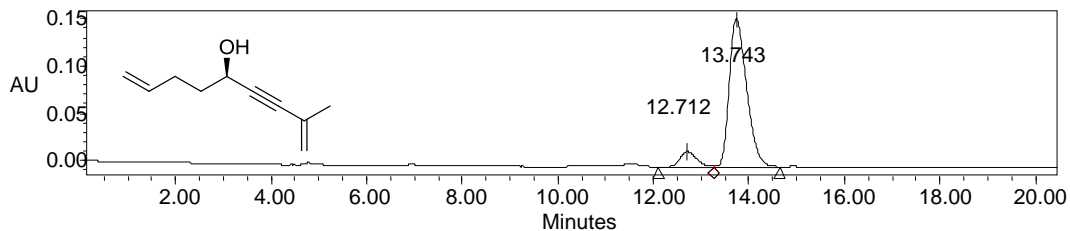
14: White solid, 36% yield. $[\alpha]_D^{24} = 103.9$ ($c = 0.35$, CHCl_3). mp 122-123 °C. $^1\text{H NMR}$ (600 MHz, CDCl_3) δ 4.21 (dd, 1H, $J = 9.0, 6.6$ Hz), 4.15 (dd, 1H, $J = 6.6, 3.0$ Hz), 3.97 (d, 1H, $J = 9.6$ Hz), 2.69 (m, 1H), 2.56 (m, 1H), 2.33 (m, 1H), 2.32 (s, 3H), 2.27 (t, 1H, $J = 7.2$ Hz), 2.02-1.97 (m, 3H), 1.78 (dt, 1H, $J = 13.2, 3.0$ Hz), 1.51 (m, 1H), 0.23 (s, 9H). $^{13}\text{C NMR}$ (150 MHz, CDCl_3) δ 200.2, 186.1, 151.8, 139.3, 133.9, 84.6, 77.6, 61.8, 42.6, 36.8, 33.9, 32.8, 29.5, 19.4, -0.7. HRMS (ESI) for $\text{C}_{17}\text{H}_{25}\text{O}_2\text{Si}$ (MH+) Calcd: 289.1624, Found: 289.1628.

VI. HPLC Plots for Determination of Enantiomeric Excess

2-methylnona-1,8-dien-3-yn-5-ol, 1a

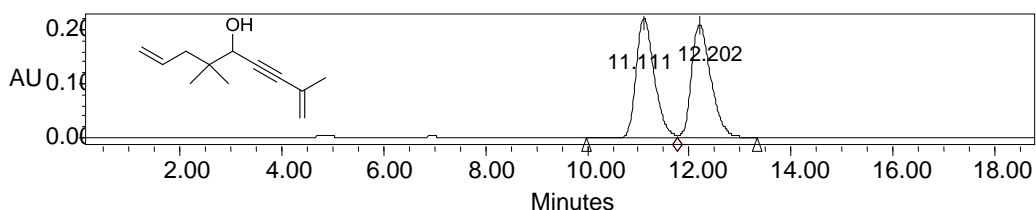


	Name	Retention Time	Area	% Area
1		12.686	62916750	49.25
2		13.742	64831953	50.75

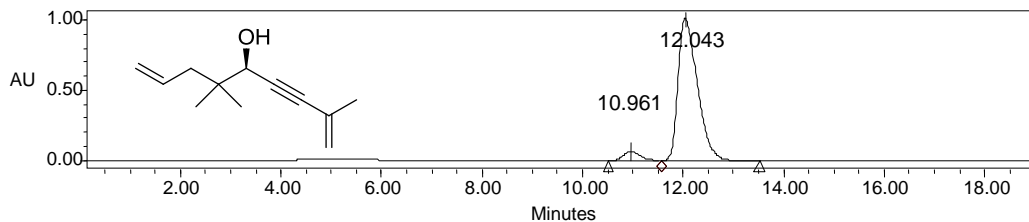


	Name	Retention Time	Area	% Area
1		12.712	377640	8.81
2		13.743	3909191	91.19

2,6,6-trimethylnona-1,8-dien-3-yn-5-ol, 1b

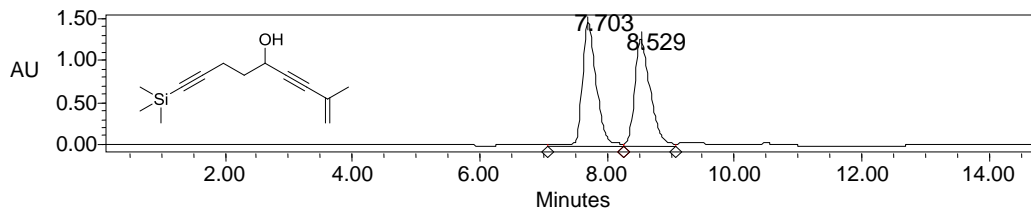


	Name	Retention Time	Area	% Area
1		11.111	5522473	49.84
2		12.202	5557302	50.16

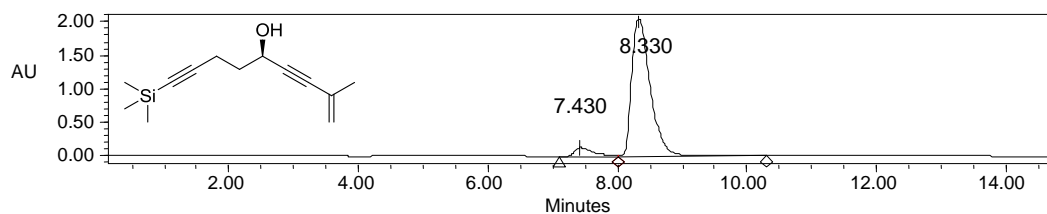


	Name	Retention Time	Area	% Area
1		10.961	1590088	5.50
2		12.043	27307507	94.50

2-methyl-9-(trimethylsilyl)nona-1-en-3,8-diyn-5-ol, (R)-12

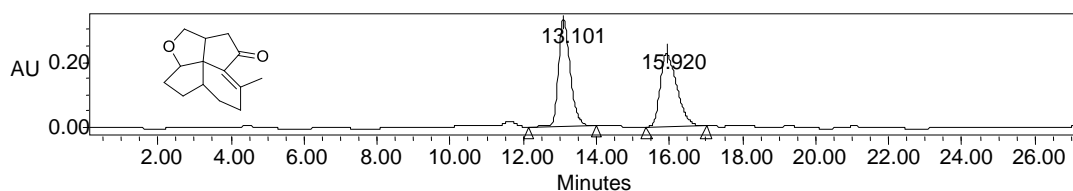


	Name	Retention Time	Area	% Area
1		7.703	22234903	50.66
2		8.529	21654310	49.34

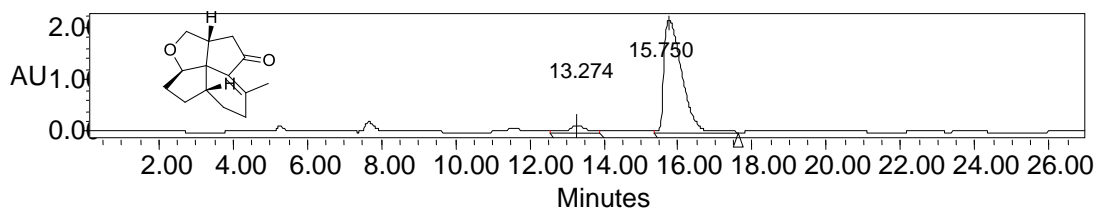


	Name	Retention Time	Area	% Area
1		7.430	2724854	6.21
2		8.330	41181586	93.79

Compound 3a:

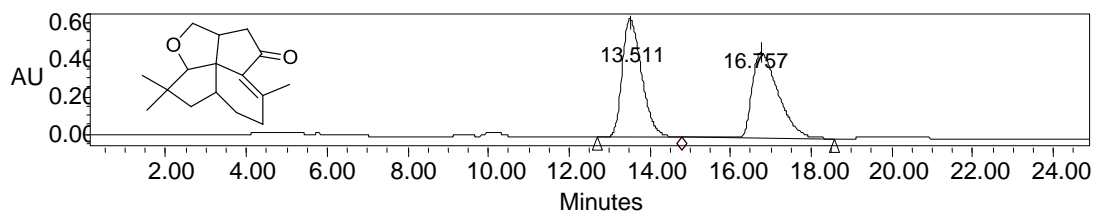


	Name	Retention Time	Area	% Area
1		13.101	7226370	50.39
2		15.920	7114325	49.61

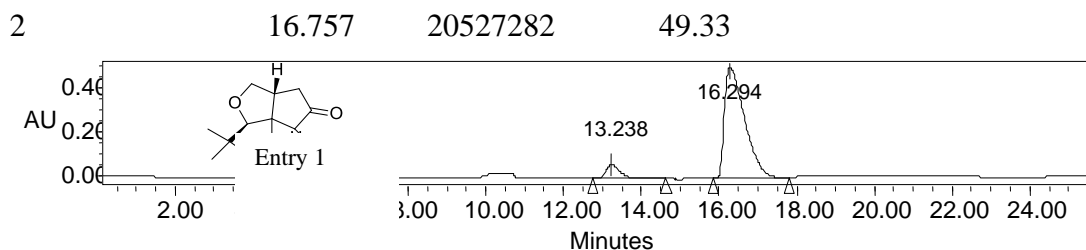


	Name	Retention Time	Area	% Area
1		13.274	3679251	4.96
2		15.750	70464451	95.04

Compound 3b:



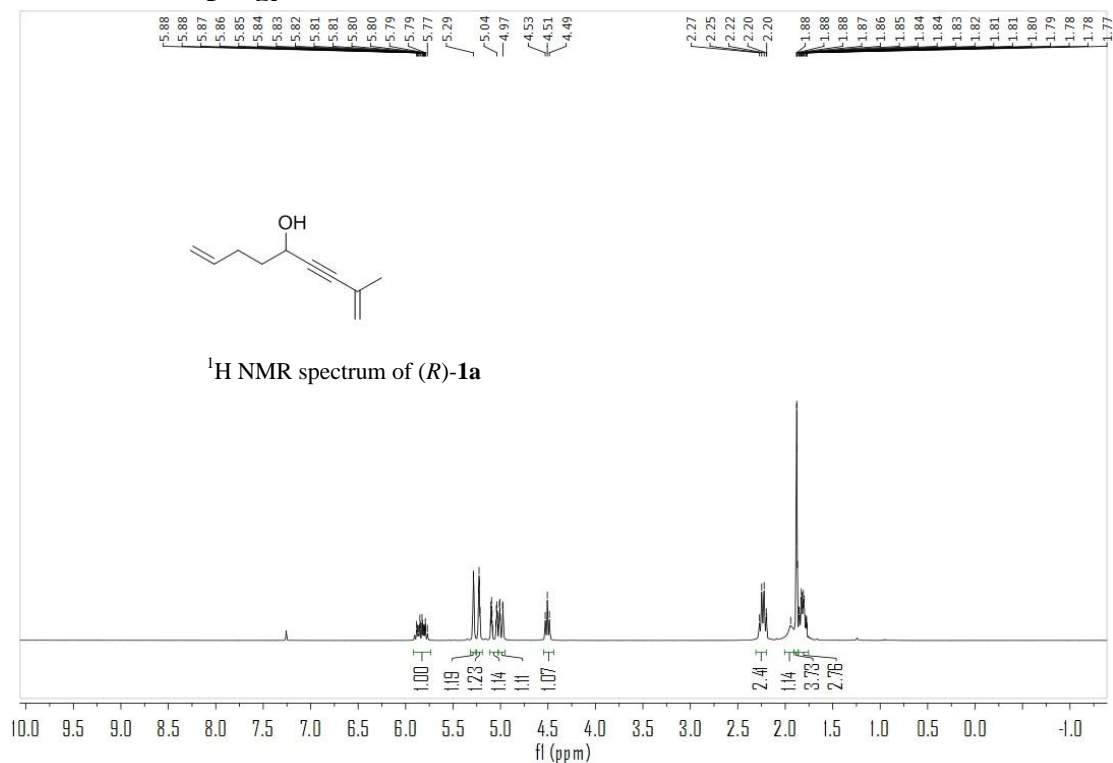
	Name	Retention Time	Area	% Area
1		13.511	21085797	50.67

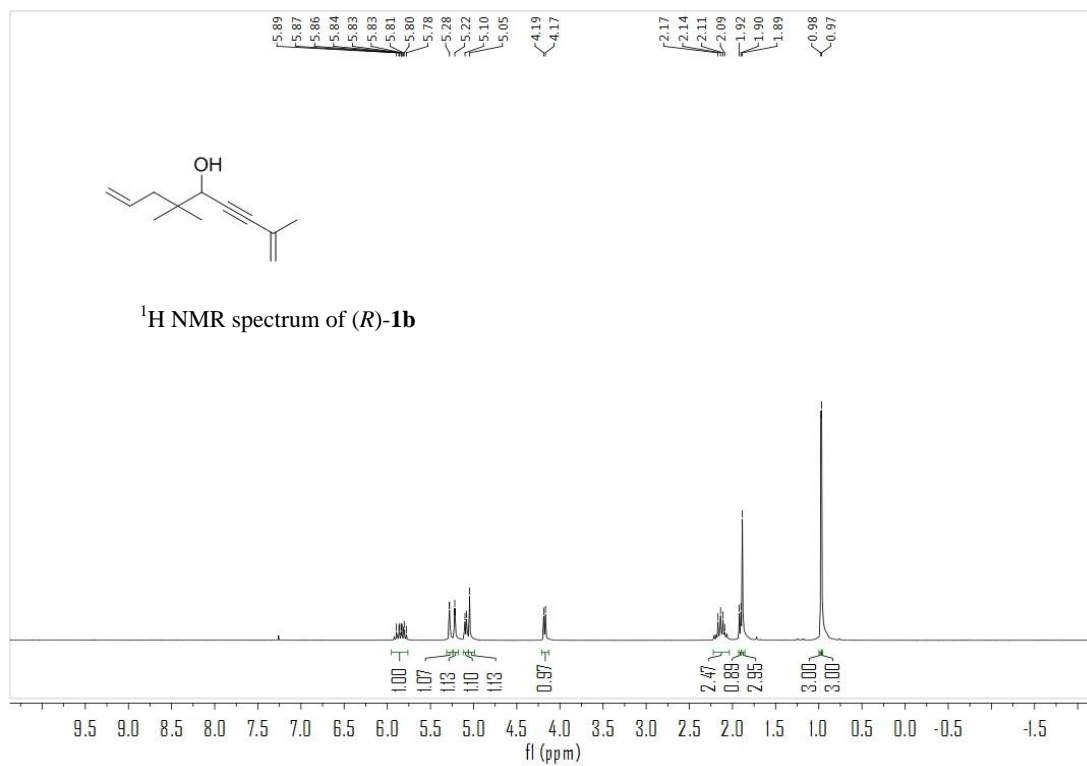
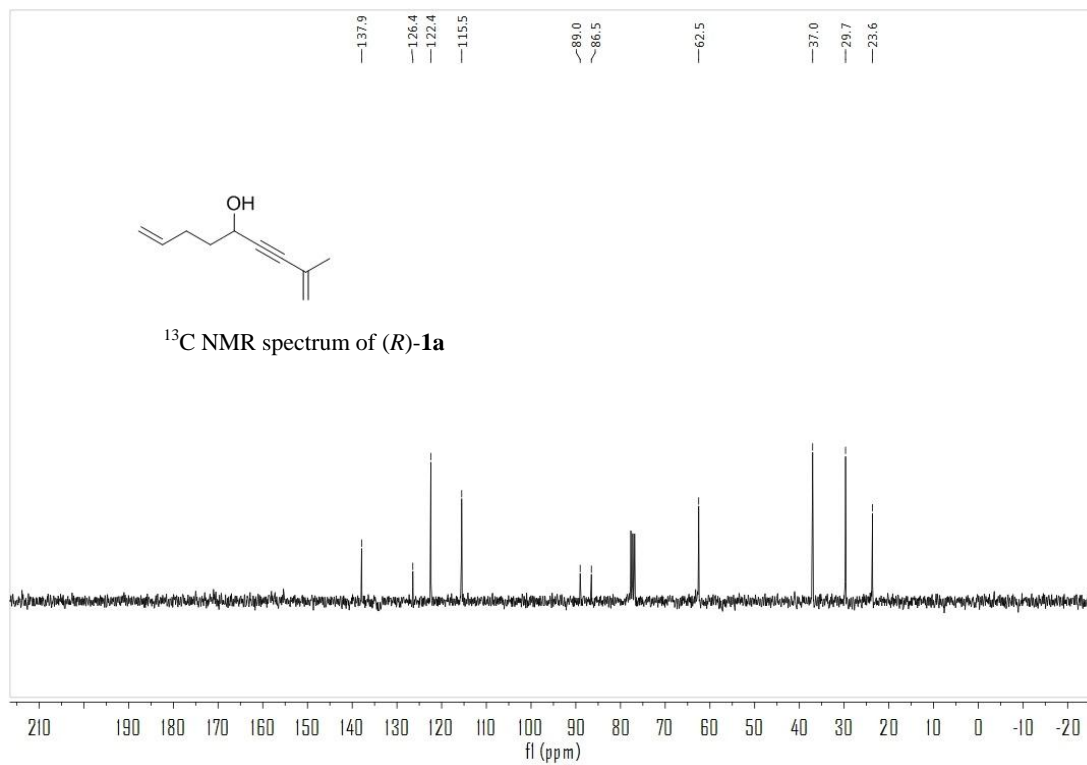


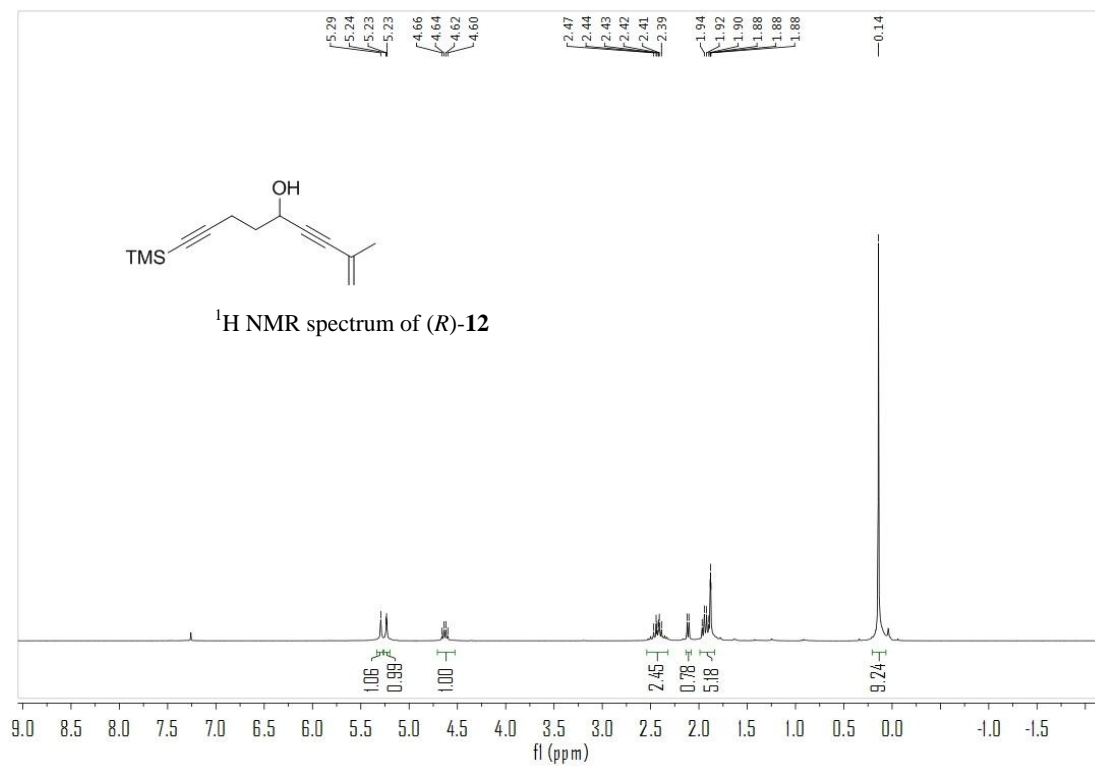
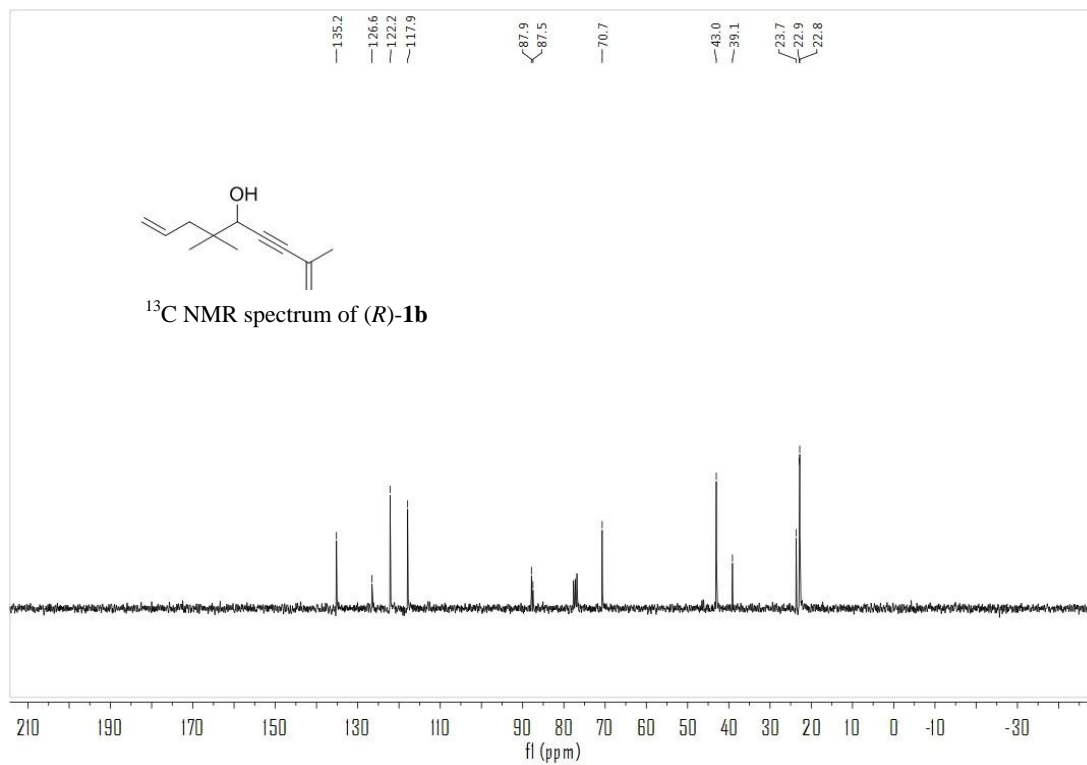
	Name	Retention Time	Area	% Area
1		13.238	1657793	8.39
2		16.294	18089845	91.61

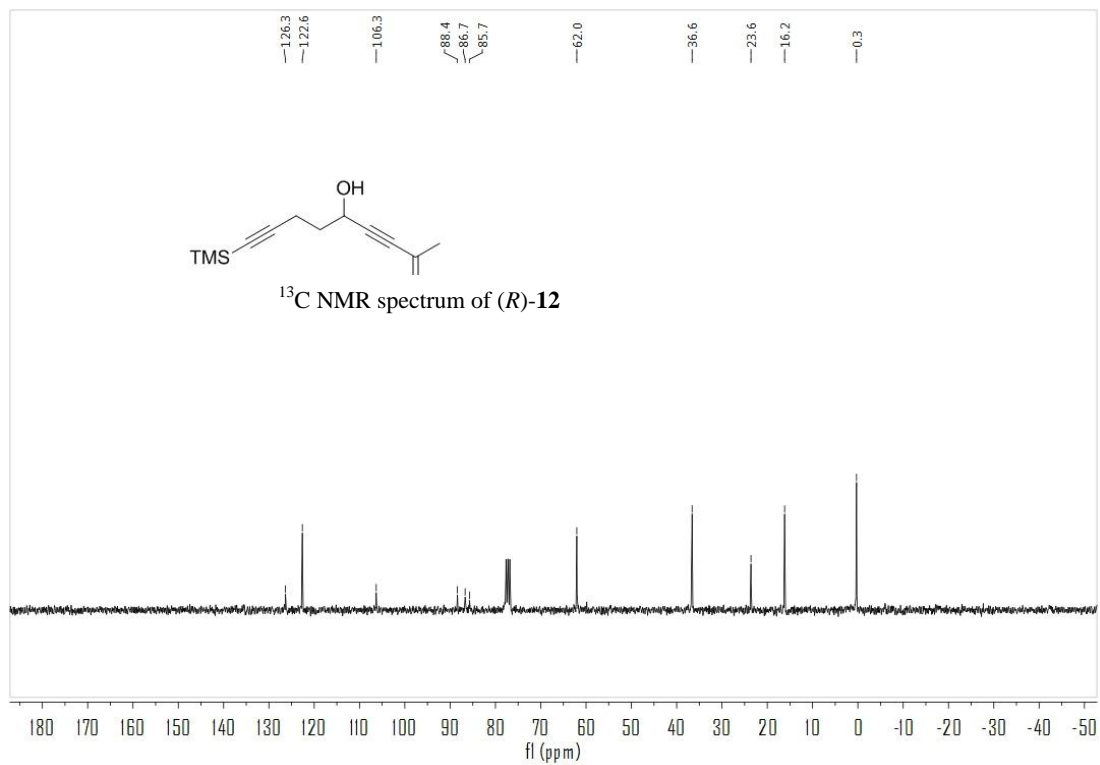
VII. ^1H NMR and ^{13}C NMR Spectra (All the NMR spectra were obtained in CDCl_3 unless indicated otherwise.)

1. Chiral Propargylic Alcohols

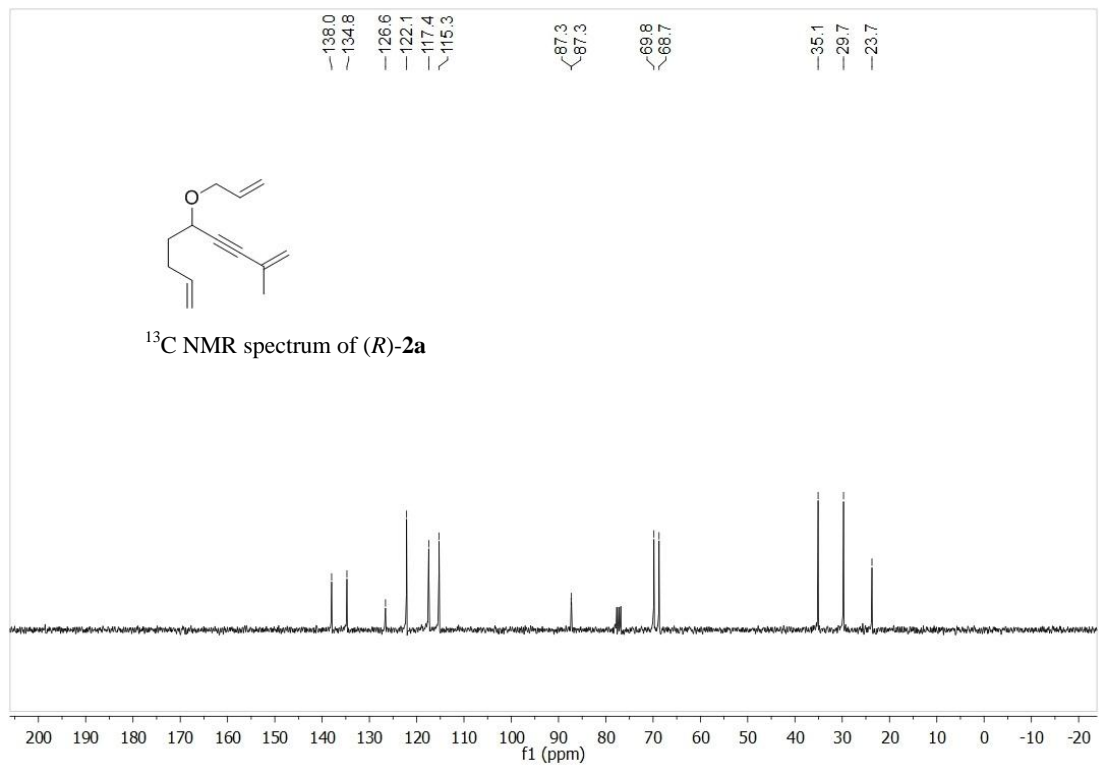
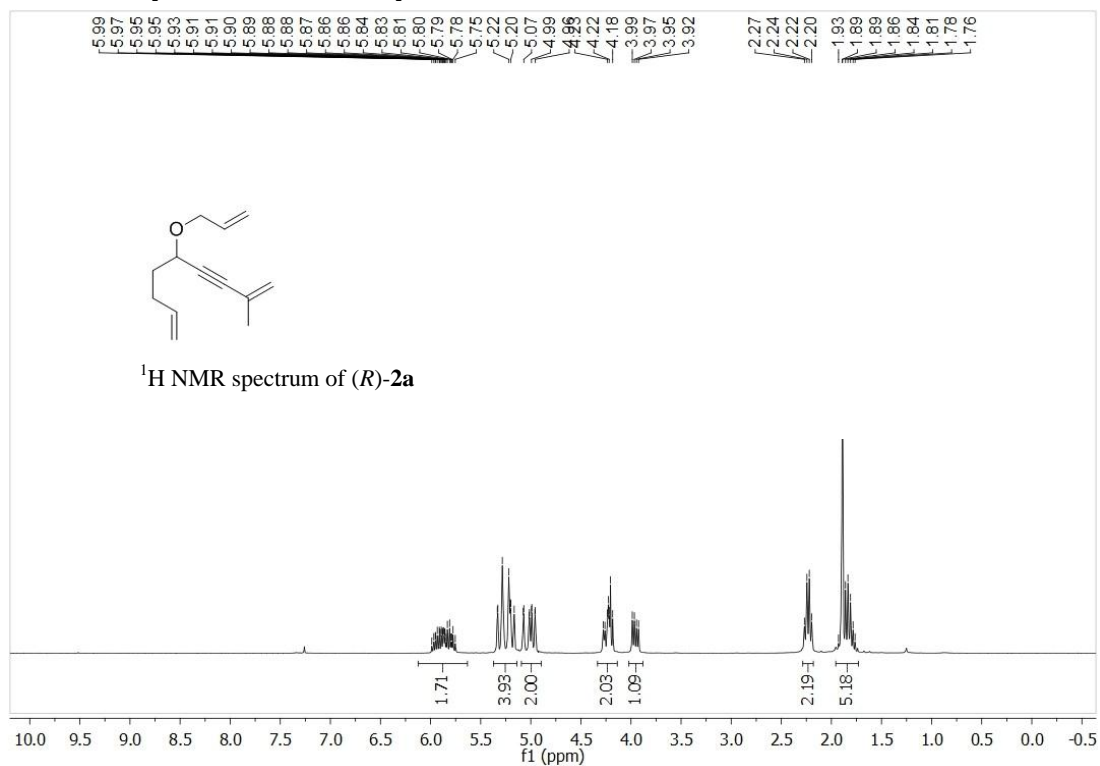


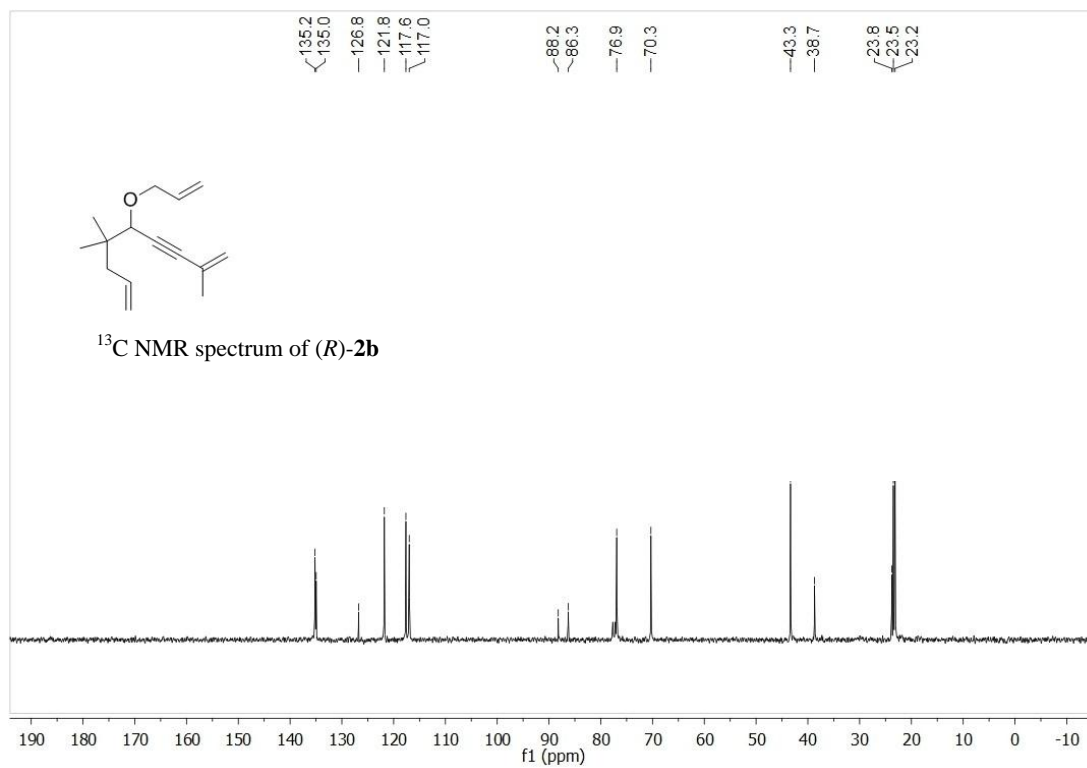
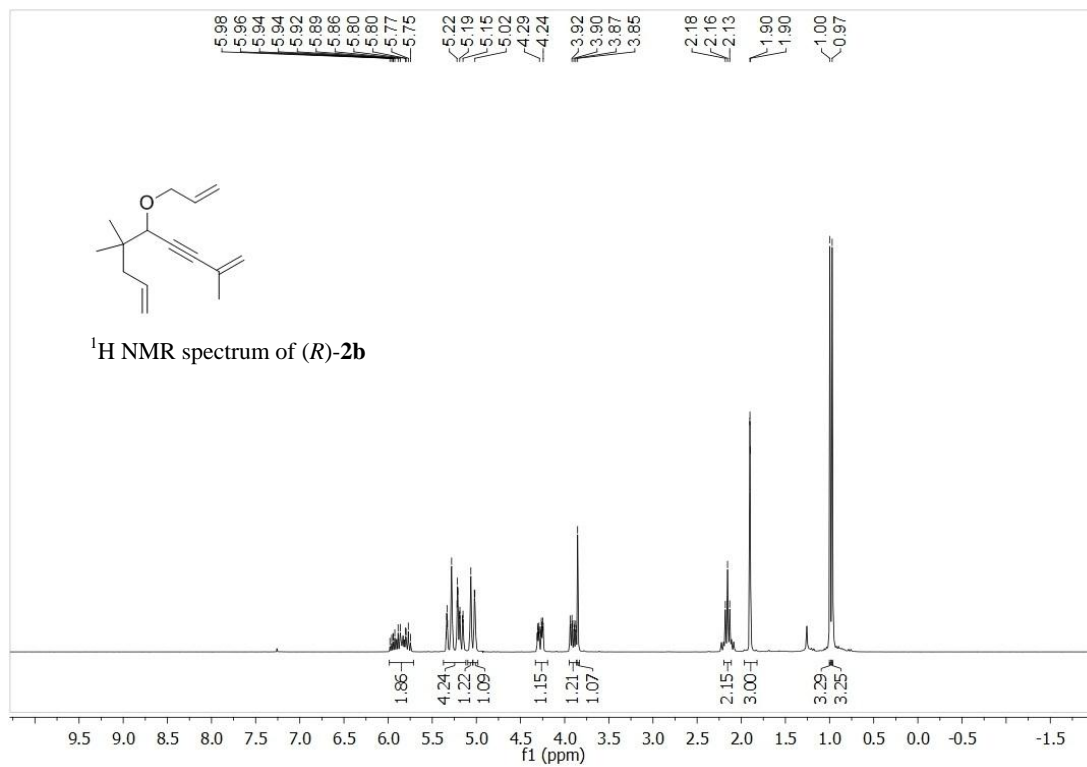


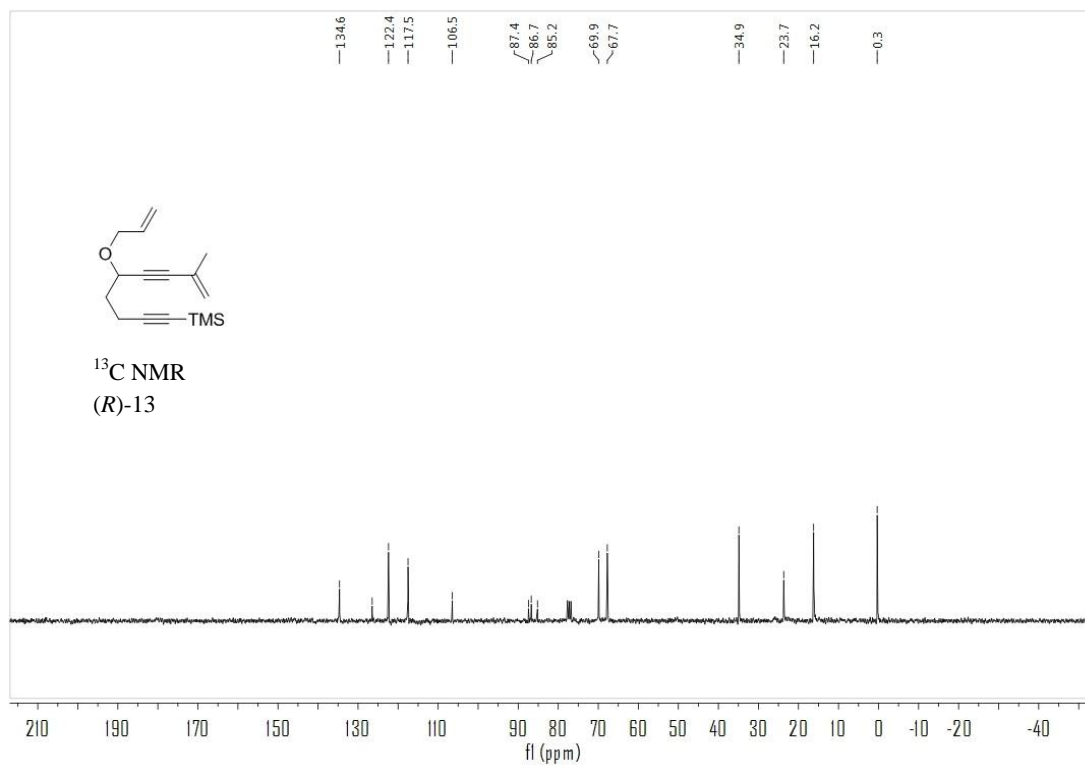
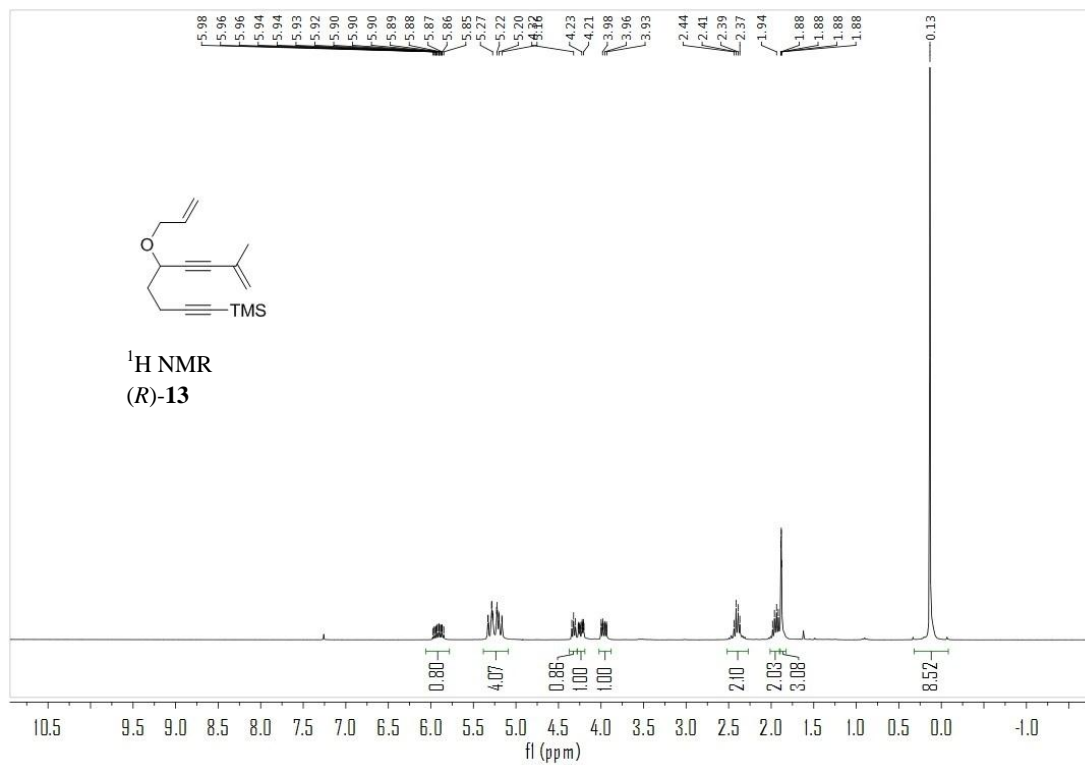




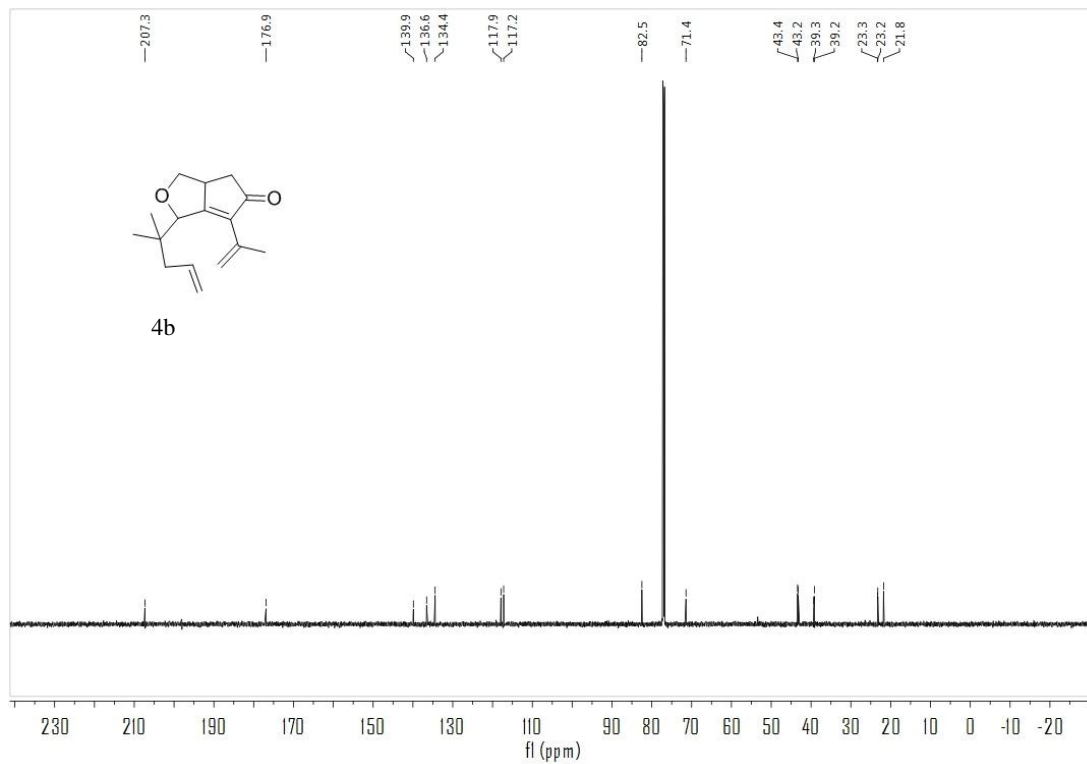
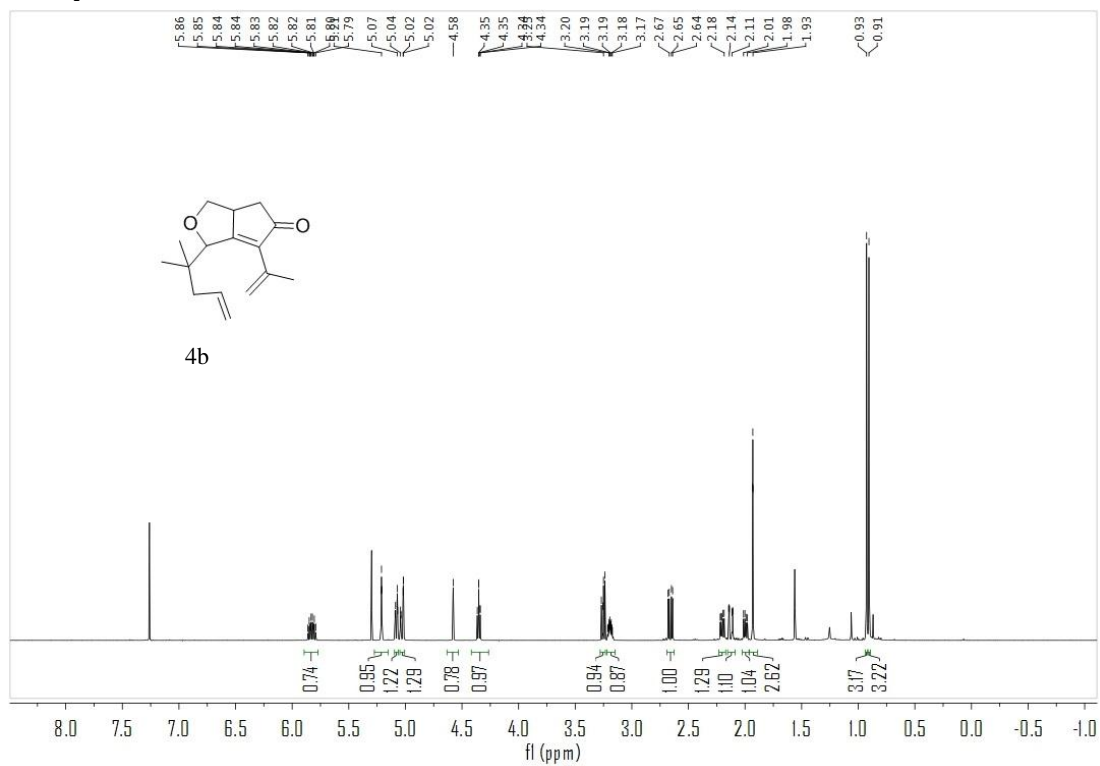
2. Triene-yne and Diene-diyne

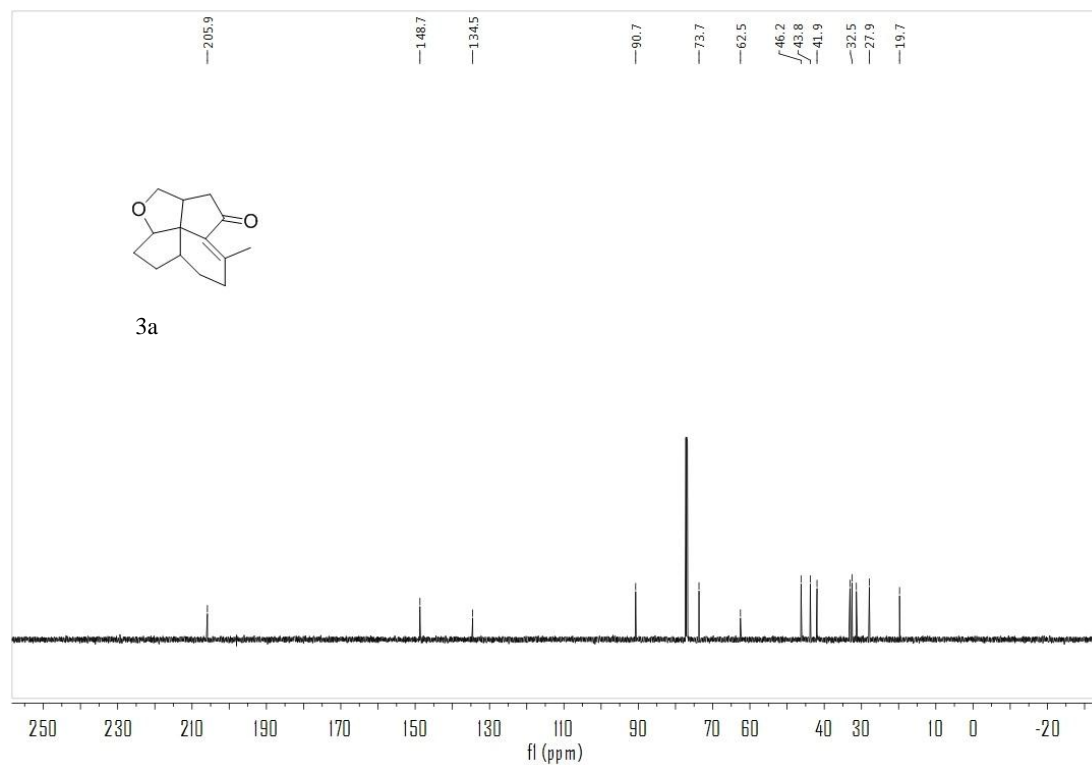
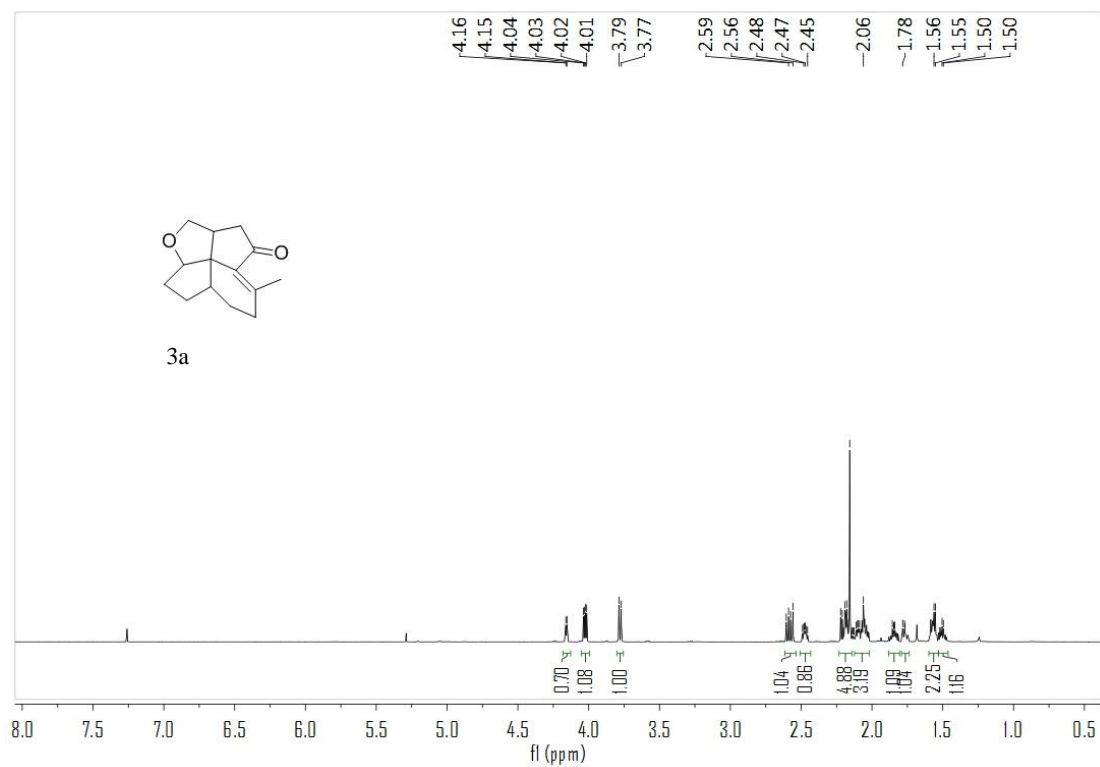


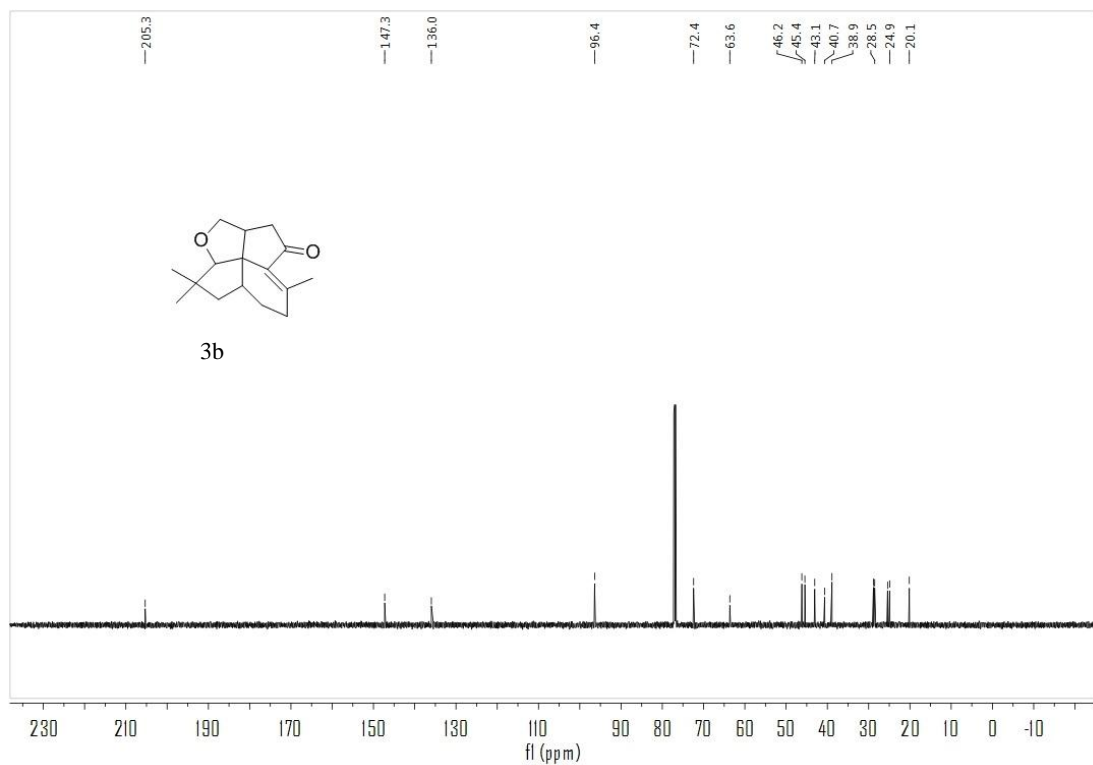
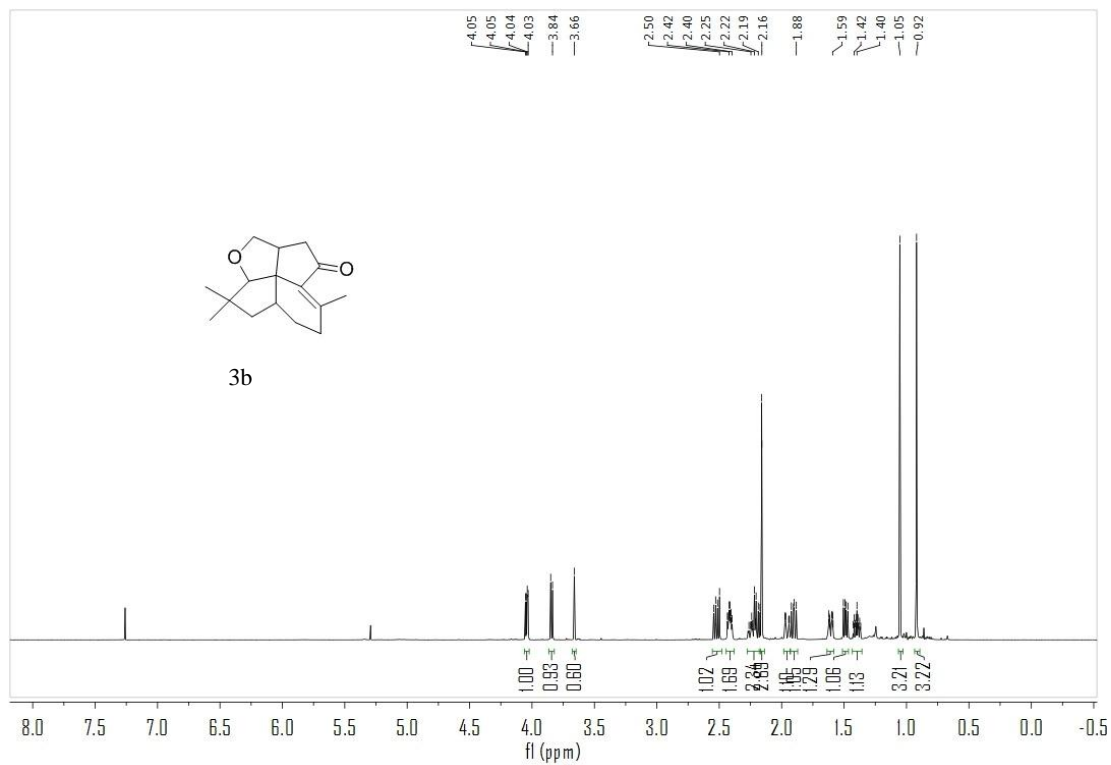


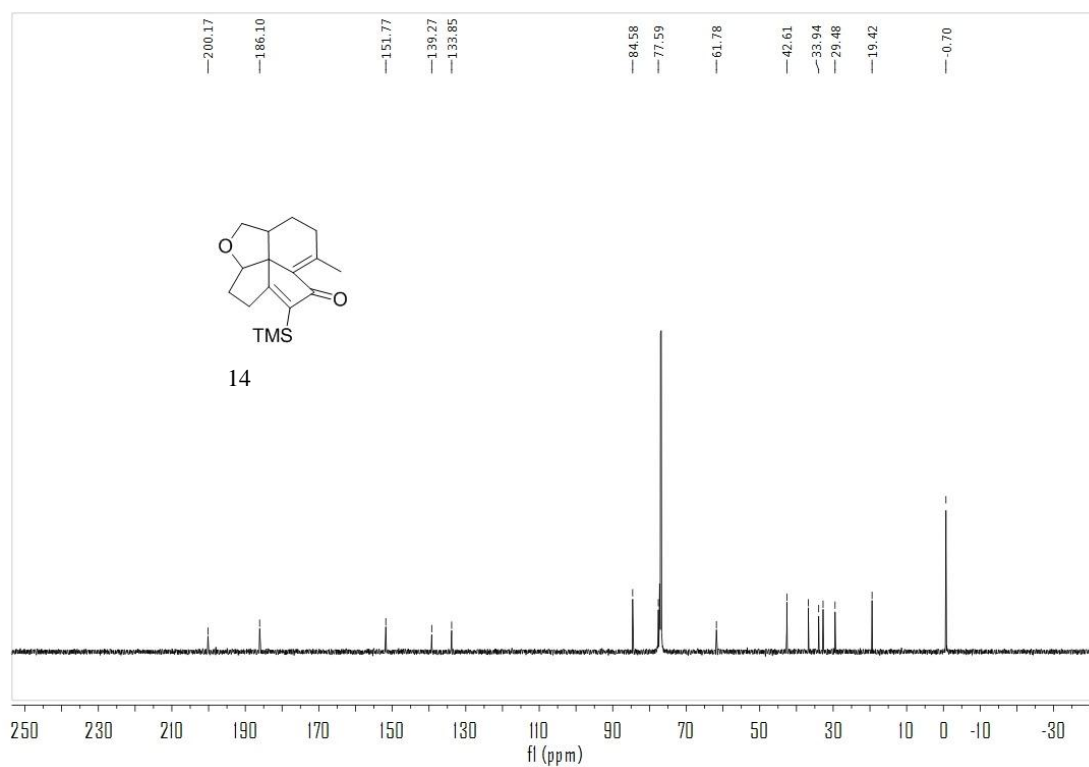
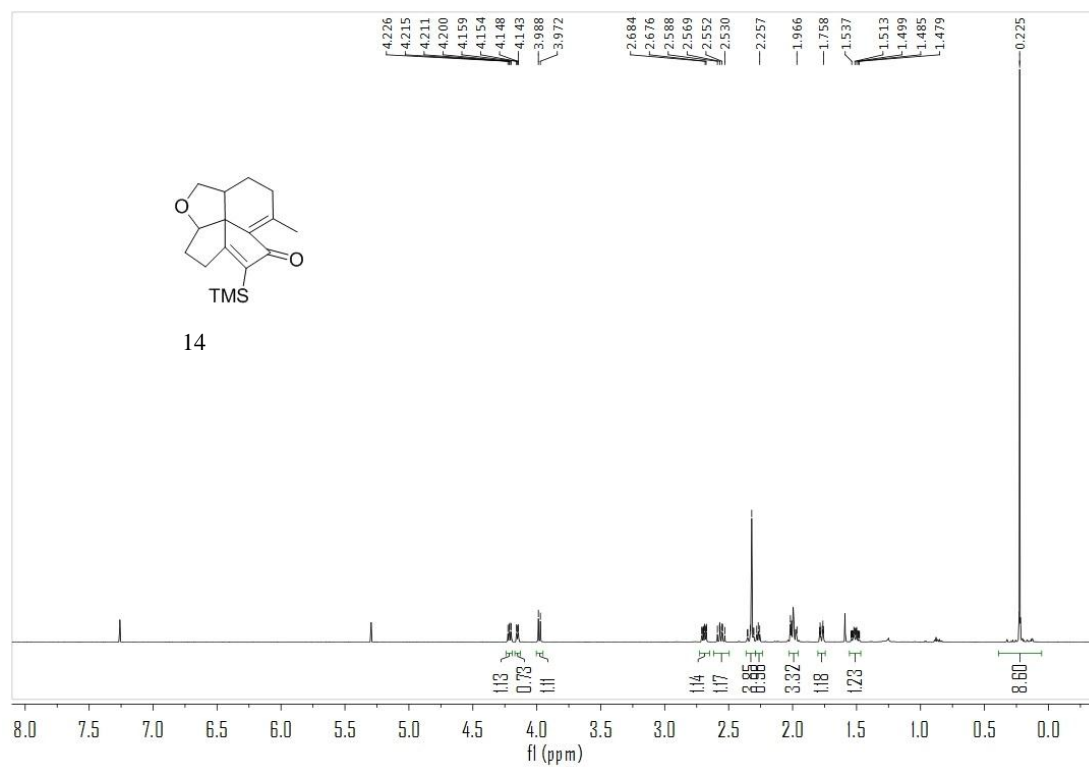


3. Cycloaddition Products



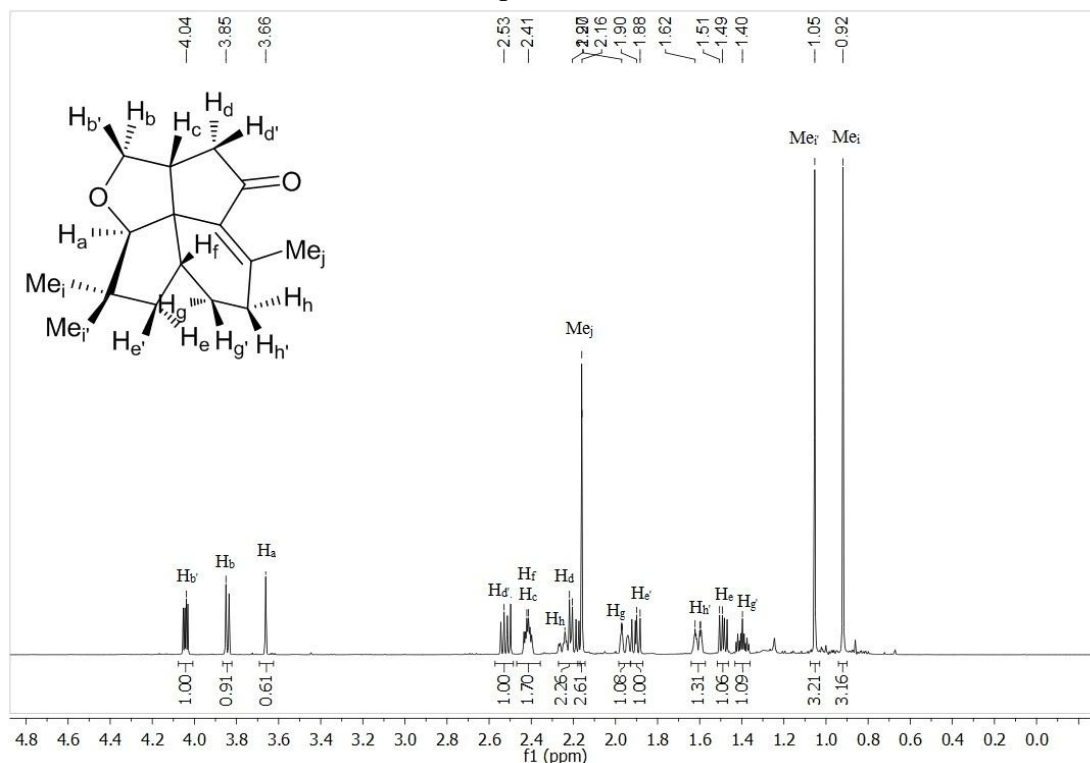


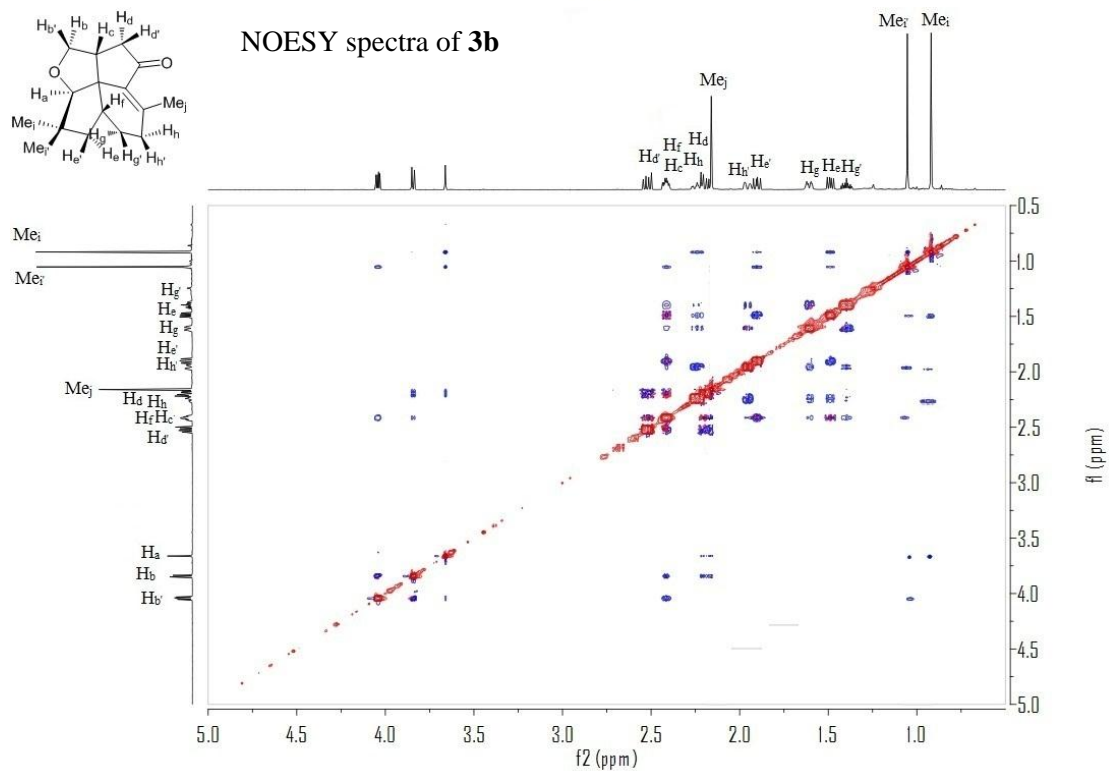
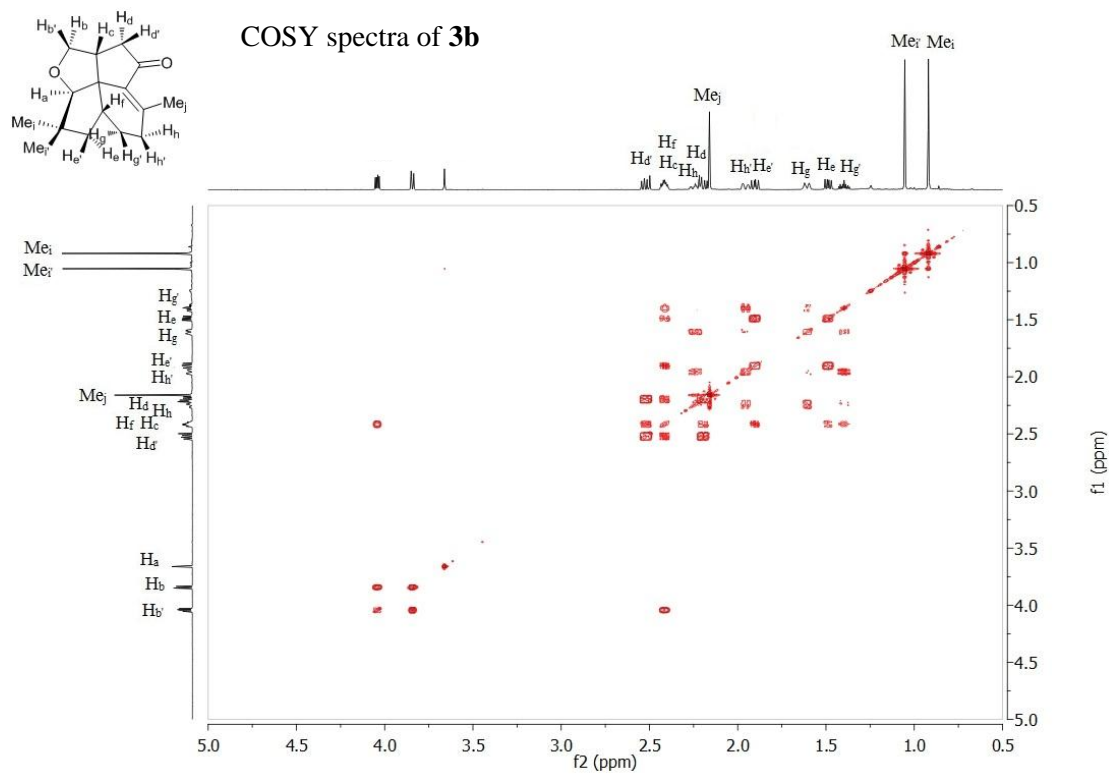


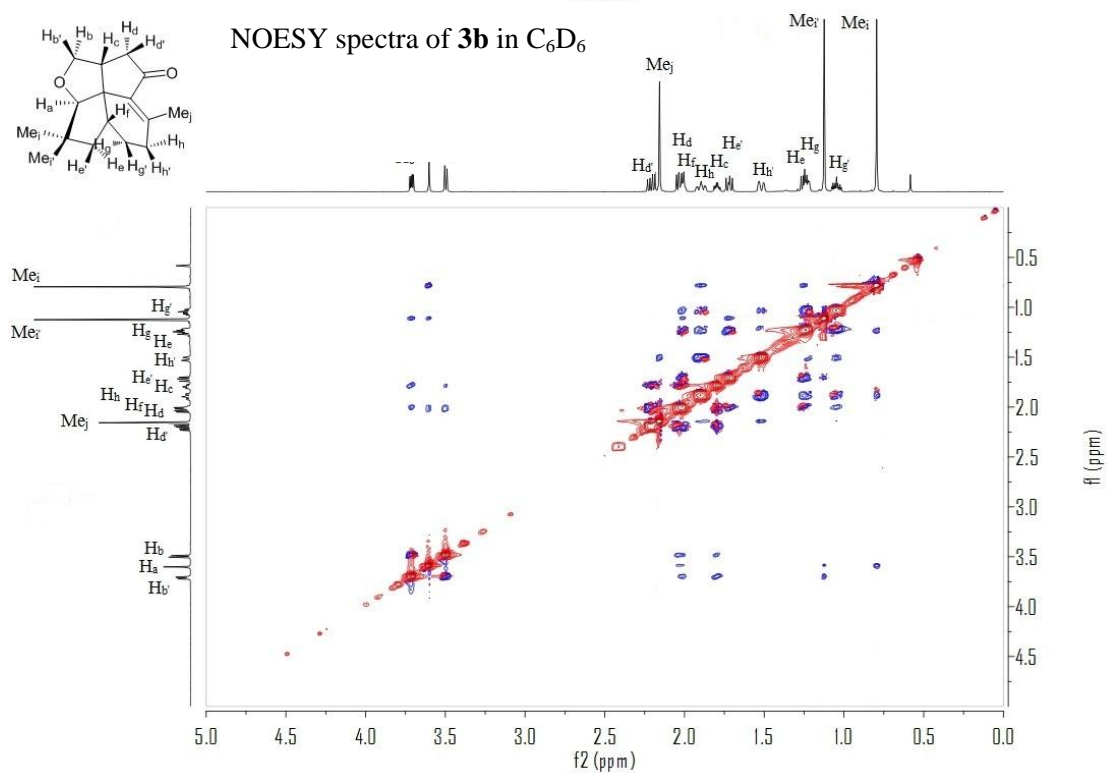
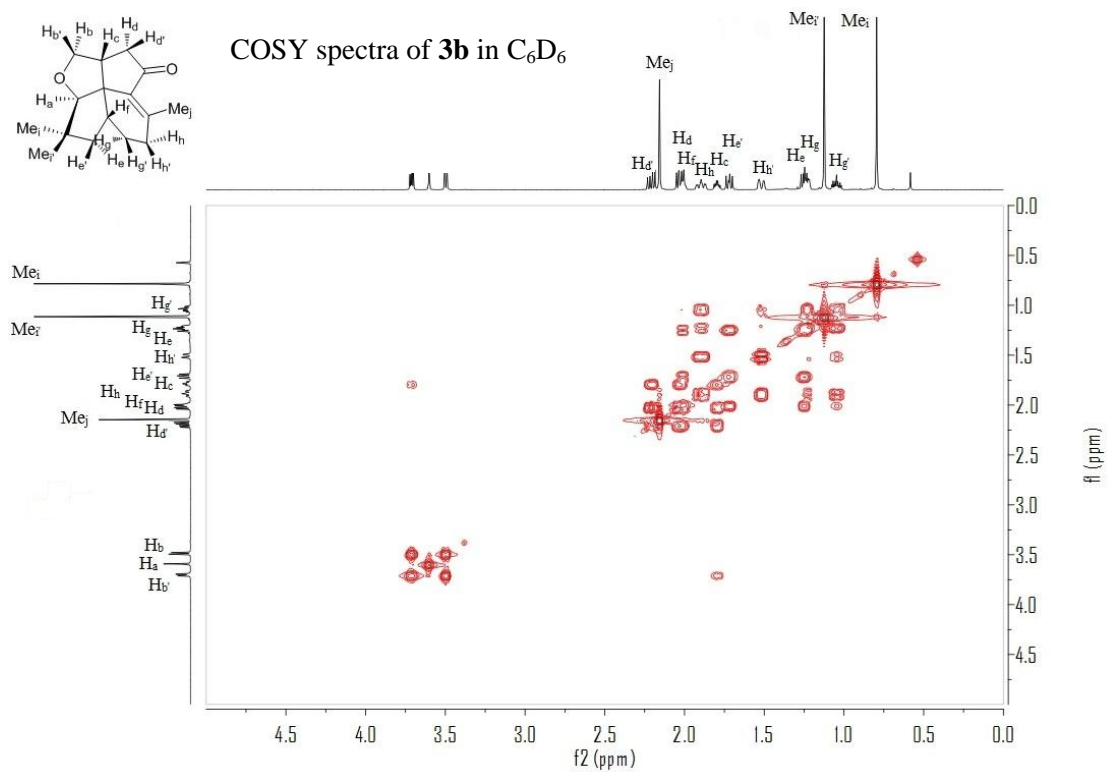


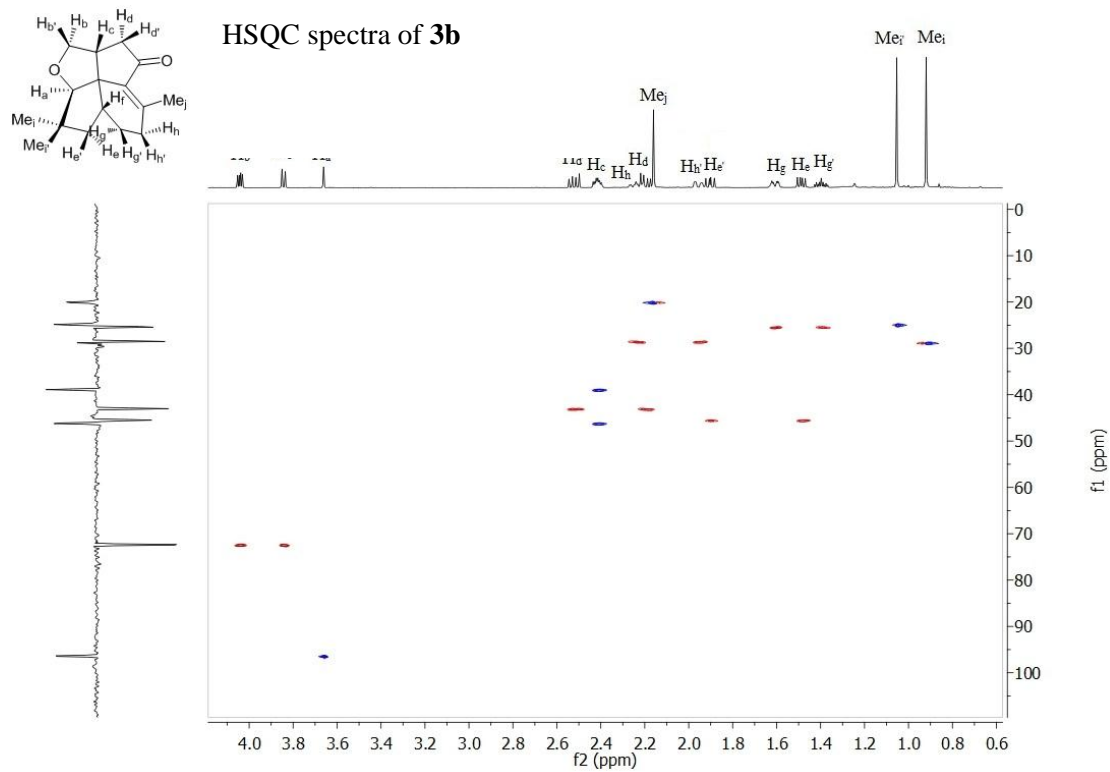
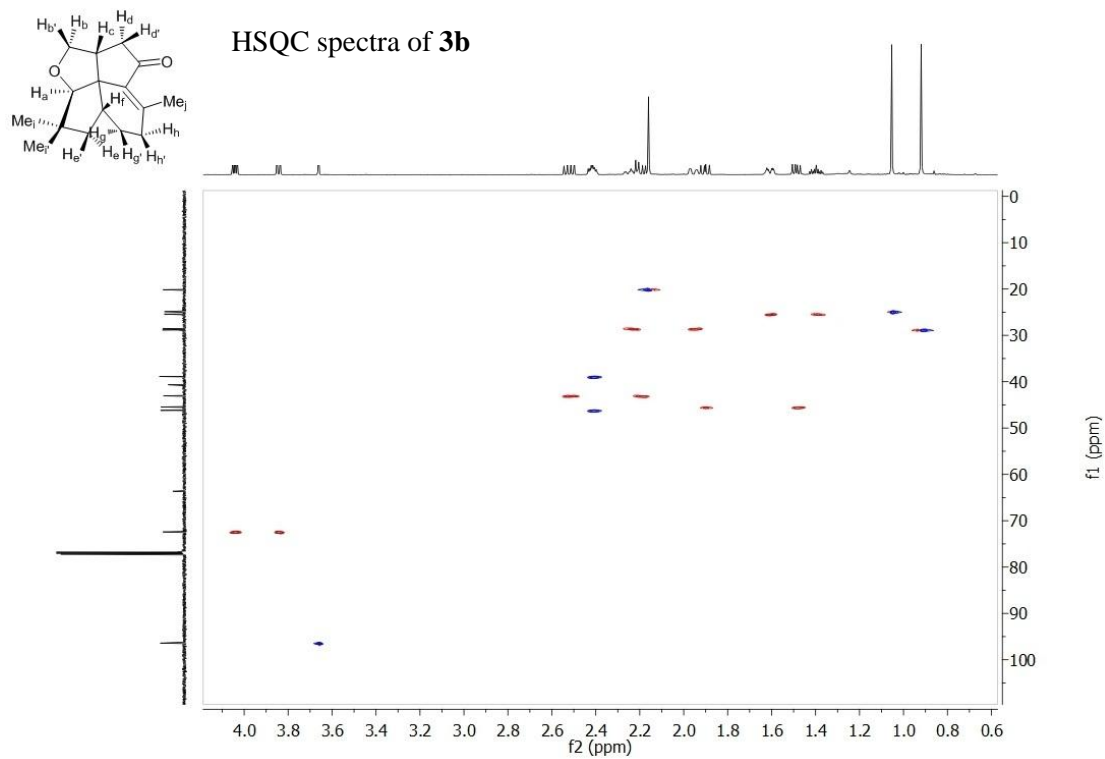
VIII. Determination of the Structure of the Cycloaddition Product **3b** by NMR Analyses

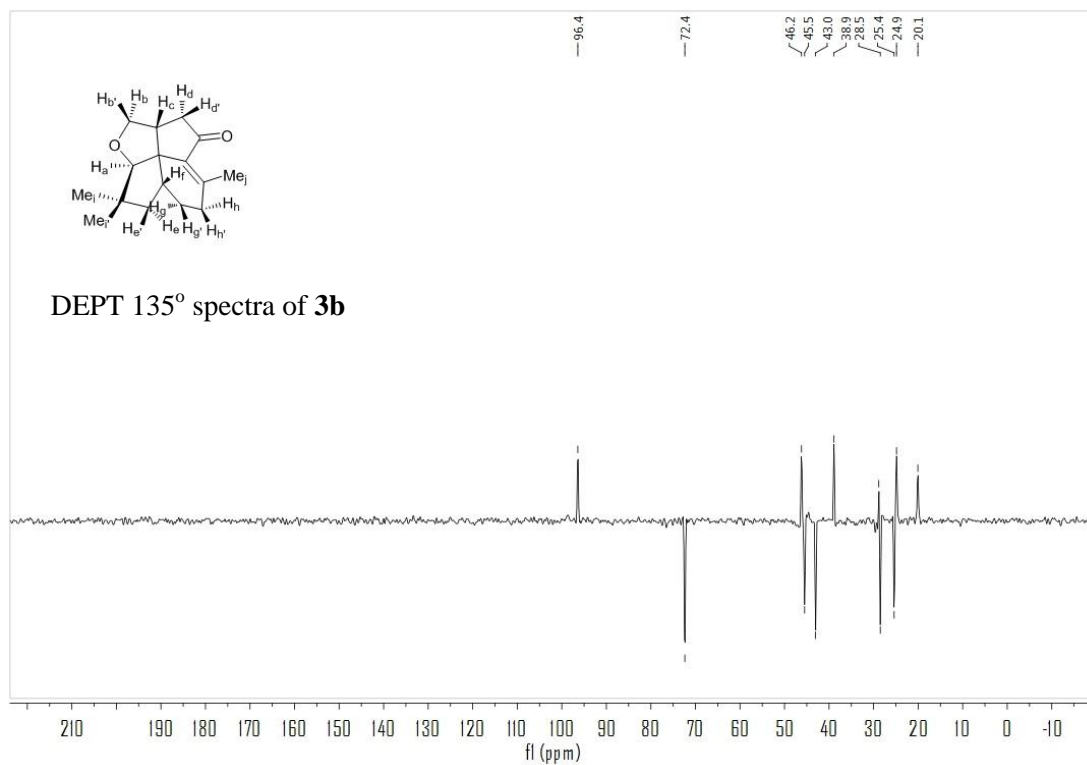
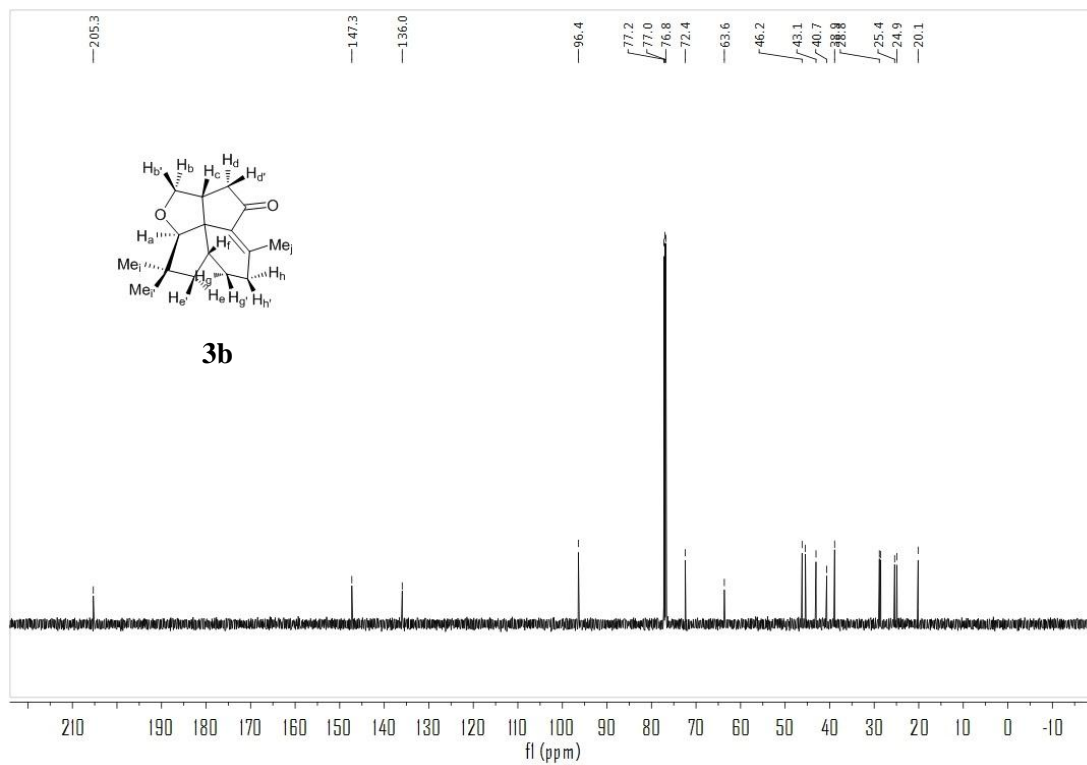
The structure of **3b** was established by various ^1H and ^{13}C NMR spectroscopic analyses including COSY, NOESY, HSQC 2D NMR and DEPT 135° . The ^1H NMR signal assignments for **3b** are shown below. The resonances at δ 4.04 and 3.85 were assigned to be $\text{H}_{\text{b}'}$ / H_{b} on the basis of COSY correlation, HSQC and DEPT 135° . The signal at δ 0.92 was assigned to be $\text{Me}_{\text{i}'}$ since it showed stronger NOE effect with the proton at δ 3.66 (H_{a}) than the signal at δ 1.05 which was assigned to be Me_{i} . The signal at δ 4.04 was assigned to be $\text{H}_{\text{b}'}$, because of its NOE effect with the signal at δ 1.05 ($\text{Me}_{\text{i}'}$). This allowed the signal at δ 3.85 to be assigned to H_{b} . The signal at δ 2.41 was assigned to H_{c} on the basis of the COSY correlation with $\text{H}_{\text{b}'}$ (δ 4.04), DEPT 135° and HSQC. The configuration of H_{c} was assigned to be *up* because of its stronger NOE interaction with $\text{H}_{\text{b}'}$ than with H_{b} . Tracing the COSY and NOE correlations of H_{c} allowed the assignment of H_{d} and $\text{H}_{\text{d}'}$ at δ 2.20 and 2.52 in which H_{d} was at δ 2.20 on the basis of the observed NOE effect with H_{b} . In support of this assignment, NOE effect was also observed between H_{a} and H_{d} . The signal at δ 1.90 was assigned to be $\text{H}_{\text{e}'}$, because of its stronger NOE effect with $\text{Me}_{\text{i}'}$ than with Me_{i} . The signal at δ 1.48 was assigned to H_{e} because of its stronger NOE effect with Me_{i} than with $\text{Me}_{\text{i}'}$. The signal of H_{f} was found to overlap with that of H_{c} at δ 2.41 as supported by its COSY correlation with $\text{H}_{\text{b}}/\text{H}_{\text{e}'}$, and the HSQC and DEPT 135° experiments. The NOE effect between $\text{Me}_{\text{i}'}$ and H_{f} supported the *up* configuration of H_{f} . This assignment was confirmed by changing the NMR solvent from CDCl_3 to C_6D_6 which separated the signal of H_{c} from H_{f} in the ^1H NMR spectrum and showed NOE effect between $\text{H}_{\text{b}'}$ and H_{f} , and between H_{c} and H_{f} in the NOESY spectrum.

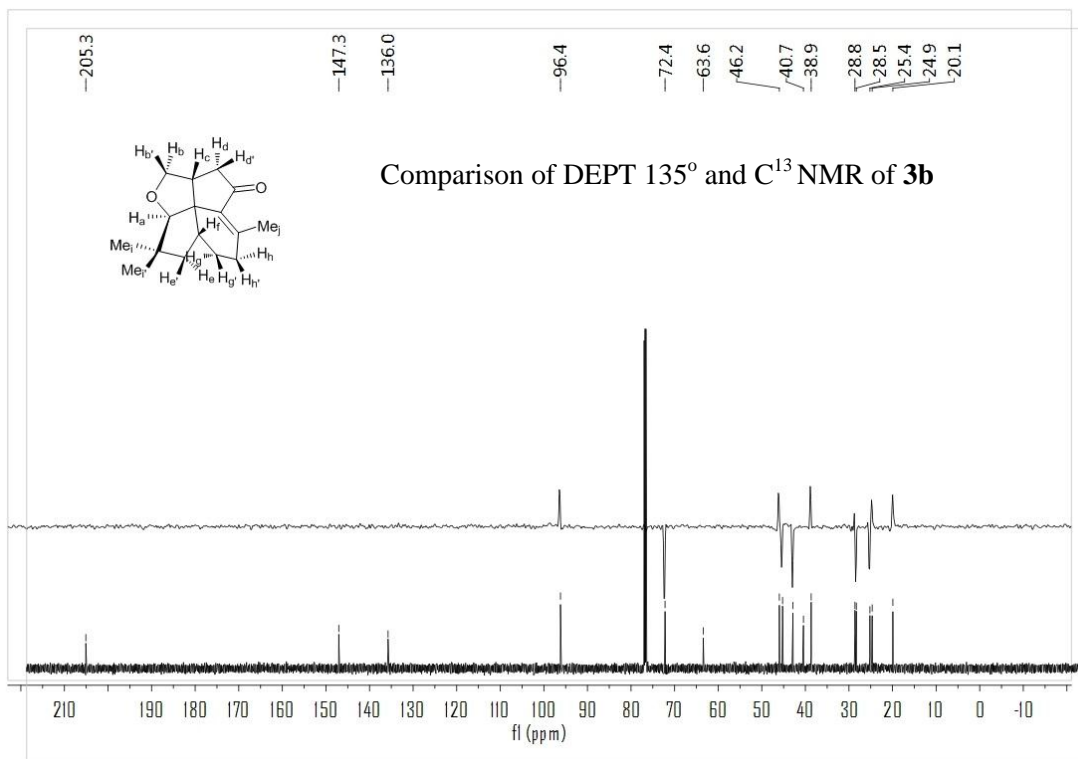




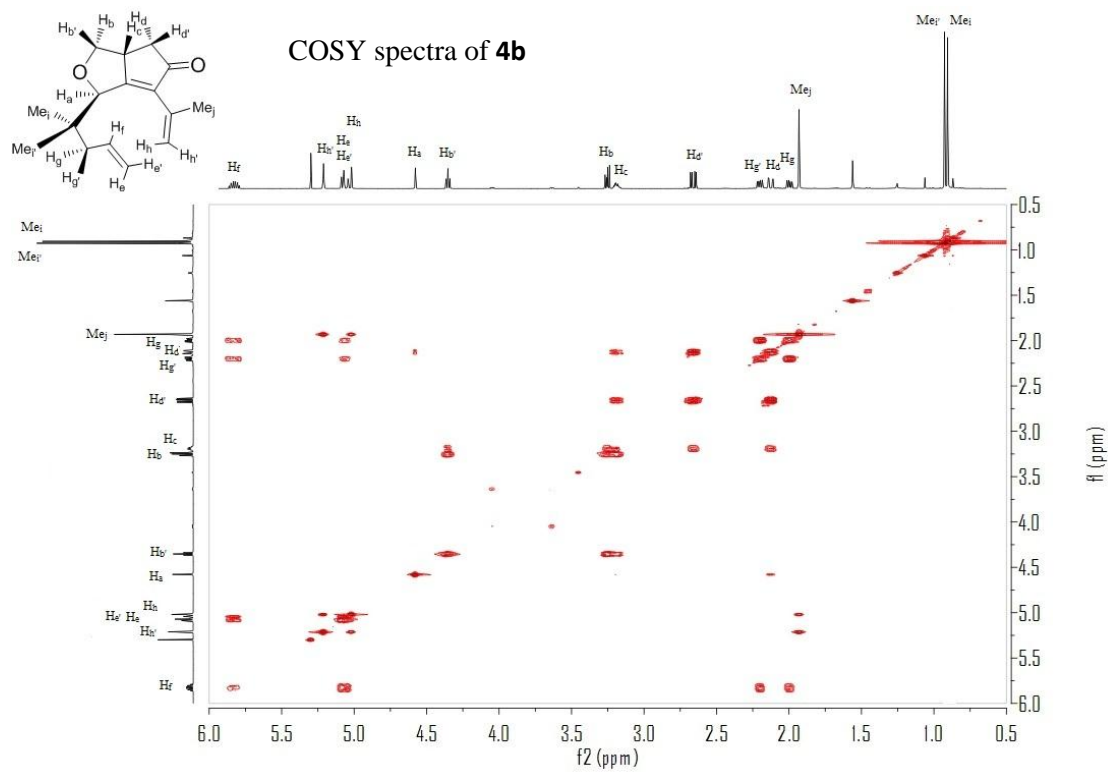


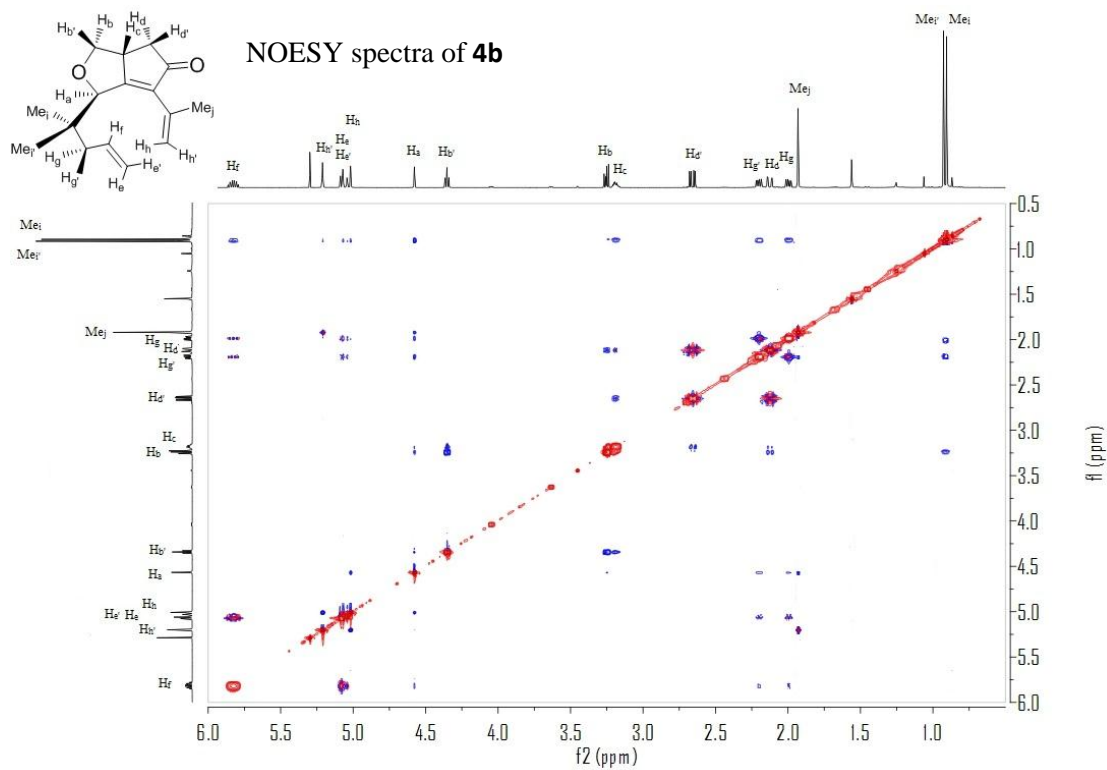




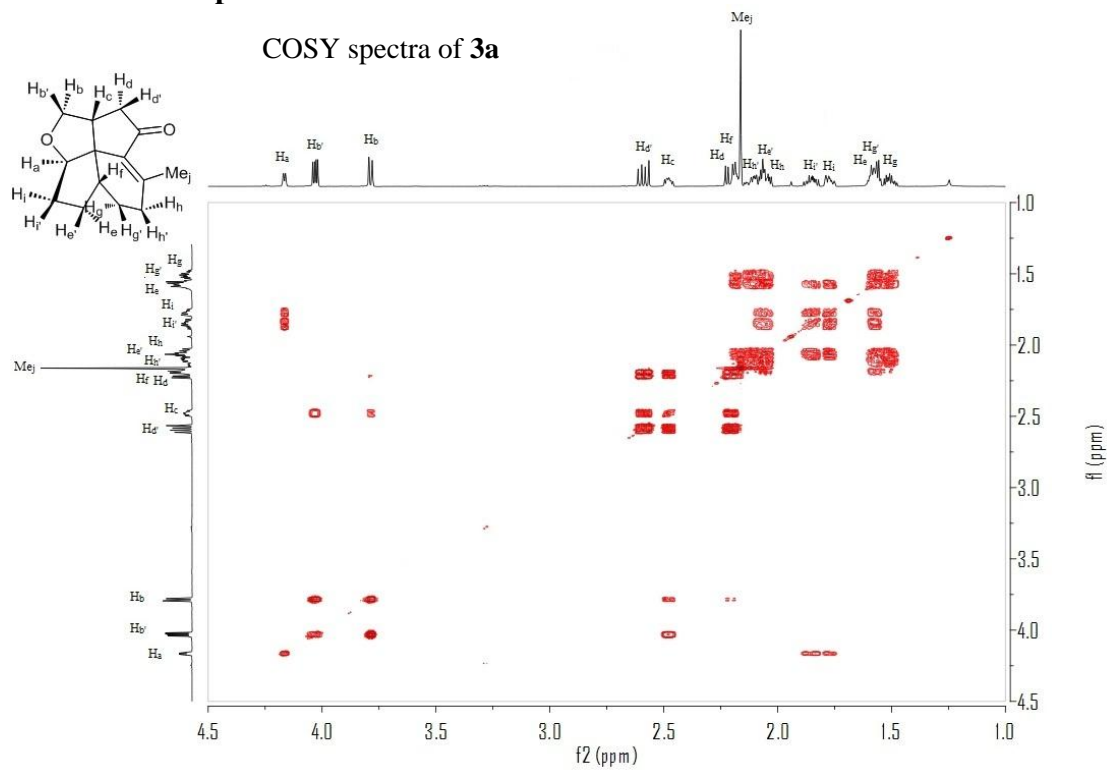


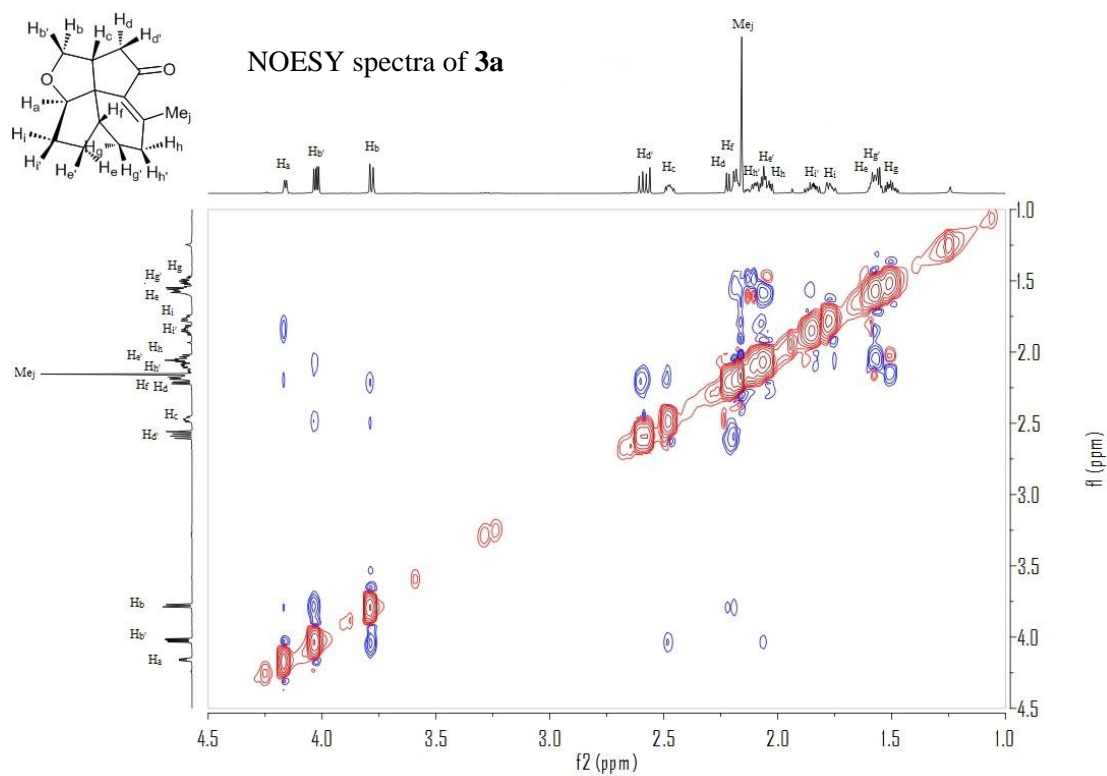
IX. 2D NMR Spectra of **4b**



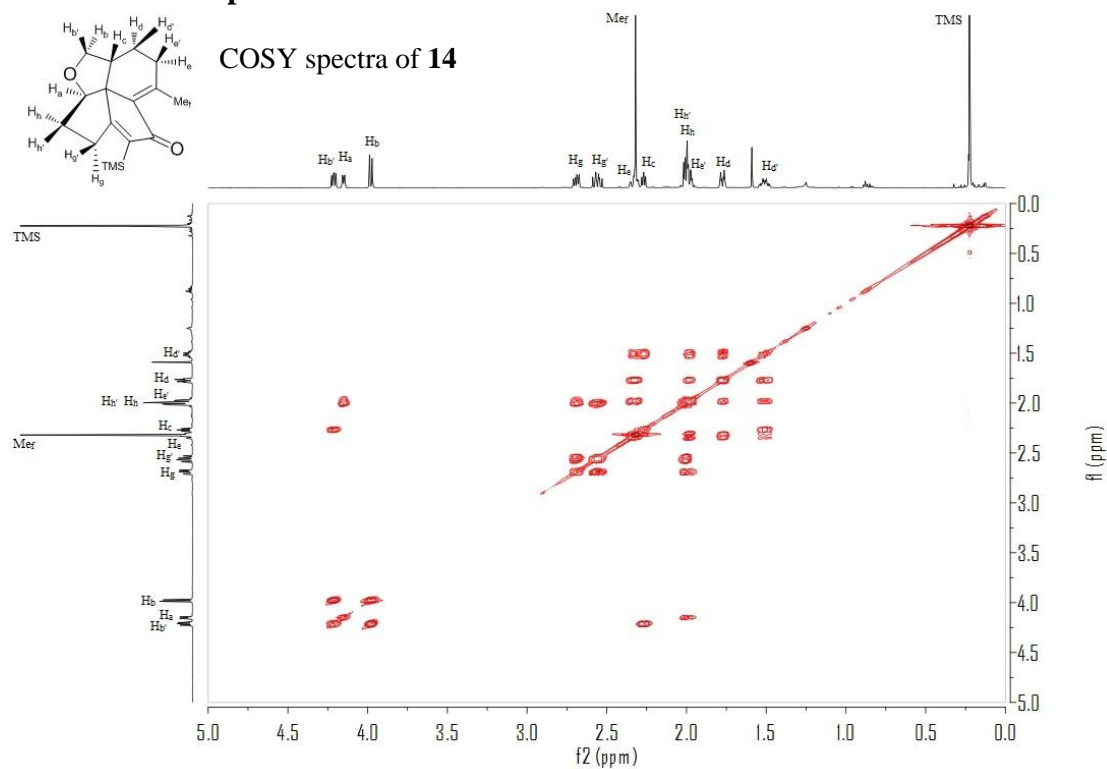


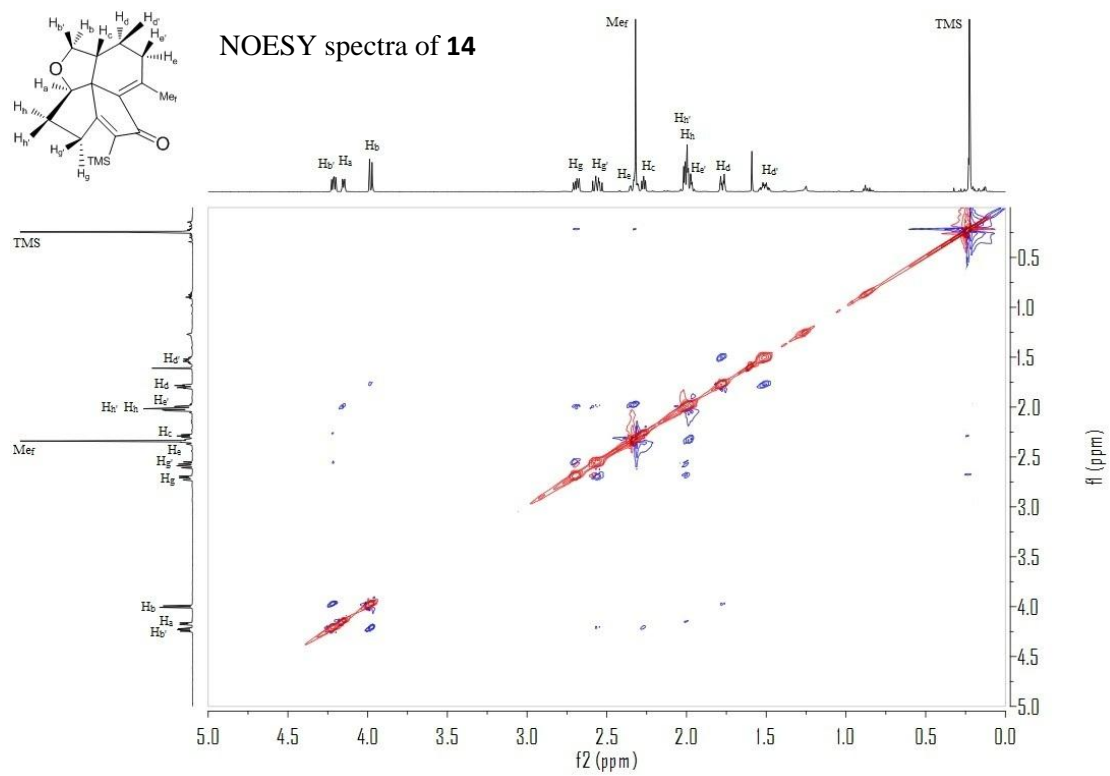
XI. 2D NMR Spectra of 3a





XII. 2D NMR Spectra of 14





XIII. X-Ray Analysis Data for Compound 14-

Table 1. Crystal data and structure refinement for C₁₇H₂₄O₂Si.

Empirical formula	C ₁₇ H ₂₄ O ₂ Si
Formula weight	288.45
Temperature	153(2) K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	P2(1)2(1)2(1)
Unit cell dimensions	a = 9.1005(3) Å b = 12.8251(5) Å c = 13.4859(5) Å
Volume	1574.0(1) Å ³
Z	4
Density (calculated)	1.217 Mg/m ³
Absorption coefficient	0.149 mm ⁻¹
F(000)	624
Crystal size	0.42 x 0.31 x 0.22 mm ³
Theta range for data collection	3.41 to 37.08°.
Index ranges	-14<=h<=15, -19<=k<=21, -22<=l<=22
Reflections collected	38547
Independent reflections	7644 [R(int) = 0.0189]
Completeness to theta = 37.08°	96.7 %
Absorption correction	Empirical
Max. and min. transmission	0.9680 and 0.9401
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	7644 / 0 / 185
Goodness-of-fit on F ²	0.874
Final R indices [I>2sigma(I)]	R1 = 0.0283, wR2 = 0.0955

R indices (all data)	R1 = 0.0308, wR2 = 0.1000
Absolute structure parameter	0.06(5)
Largest diff. peak and hole	0.376 and -0.184 e.Å ⁻³

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å²x 10³) for C₁₇H₂₄O₂Si. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Si(1)	10002(1)	9598(1)	7722(1)	20(1)
O(1)	4201(1)	7342(1)	6215(1)	30(1)
O(2)	7456(1)	9406(1)	9356(1)	25(1)
C(1)	8289(1)	8786(1)	7760(1)	18(1)
C(2)	7636(1)	8182(1)	7067(1)	19(1)
C(3)	7810(1)	8086(1)	5969(1)	30(1)
C(4)	6257(1)	8442(1)	5632(1)	29(1)
C(5)	5166(1)	8170(1)	6478(1)	21(1)
C(6)	5054(1)	6419(1)	6336(1)	32(1)
C(7)	5878(1)	6566(1)	7319(1)	22(1)
C(8)	4950(1)	6186(1)	8202(1)	27(1)
C(9)	3922(1)	7026(1)	8622(1)	26(1)
C(10)	4862(1)	7910(1)	8996(1)	20(1)
C(11)	5935(1)	8226(1)	8381(1)	16(1)
C(12)	7250(1)	8870(1)	8611(1)	17(1)
C(13)	4619(1)	8304(1)	10028(1)	27(1)
C(14)	6136(1)	7774(1)	7361(1)	16(1)
C(15)	11080(1)	9376(1)	8879(1)	35(1)
C(16)	9423(1)	10990(1)	7654(1)	28(1)

C(17) 11171(1) 9260(1) 6630(1) 34(1)

Table 3. Bond lengths [\AA] and angles [$^\circ$] for $\text{C}_{17}\text{H}_{24}\text{O}_2\text{Si}$.

Si(1)-C(16)	1.8640(8)
Si(1)-C(15)	1.8654(8)
Si(1)-C(17)	1.8678(8)
Si(1)-C(1)	1.8756(6)
O(1)-C(5)	1.4232(9)
O(1)-C(6)	1.4258(12)
O(2)-C(12)	1.2308(7)
C(1)-C(2)	1.3511(8)
C(1)-C(12)	1.4914(8)
C(2)-C(3)	1.4937(9)
C(2)-C(14)	1.5150(8)
C(3)-C(4)	1.5527(12)
C(4)-C(5)	1.5518(10)
C(5)-C(14)	1.5670(8)
C(6)-C(7)	1.5349(11)
C(7)-C(8)	1.5395(10)
C(7)-C(14)	1.5676(8)
C(8)-C(9)	1.5358(12)
C(9)-C(10)	1.5070(10)
C(10)-C(11)	1.3439(8)
C(10)-C(13)	1.4967(9)
C(11)-C(12)	1.4873(8)
C(11)-C(14)	1.5039(8)

C(16)-Si(1)-C(15)	109.59(4)
C(16)-Si(1)-C(17)	110.17(4)
C(15)-Si(1)-C(17)	108.92(5)
C(16)-Si(1)-C(1)	107.36(3)
C(15)-Si(1)-C(1)	109.26(3)
C(17)-Si(1)-C(1)	111.50(3)
C(5)-O(1)-C(6)	104.80(6)
C(2)-C(1)-C(12)	107.20(5)
C(2)-C(1)-Si(1)	131.65(5)
C(12)-C(1)-Si(1)	120.51(4)
C(1)-C(2)-C(3)	133.32(6)
C(1)-C(2)-C(14)	114.40(5)
C(3)-C(2)-C(14)	109.04(5)
C(2)-C(3)-C(4)	99.76(6)
C(5)-C(4)-C(3)	107.55(5)
O(1)-C(5)-C(4)	112.28(5)
O(1)-C(5)-C(14)	107.14(5)
C(4)-C(5)-C(14)	105.70(5)
O(1)-C(6)-C(7)	105.21(5)
C(6)-C(7)-C(8)	111.16(6)
C(6)-C(7)-C(14)	103.06(5)
C(8)-C(7)-C(14)	111.54(5)
C(9)-C(8)-C(7)	113.42(5)
C(10)-C(9)-C(8)	107.81(6)
C(11)-C(10)-C(13)	125.40(6)
C(11)-C(10)-C(9)	115.63(5)
C(13)-C(10)-C(9)	118.77(6)
C(10)-C(11)-C(12)	128.53(5)

C(10)-C(11)-C(14)	122.48(5)
C(12)-C(11)-C(14)	107.91(5)
O(2)-C(12)-C(11)	127.11(6)
O(2)-C(12)-C(1)	124.88(6)
C(11)-C(12)-C(1)	107.98(4)
C(11)-C(14)-C(2)	102.47(4)
C(11)-C(14)-C(5)	120.04(5)
C(2)-C(14)-C(5)	101.34(4)
C(11)-C(14)-C(7)	113.33(5)
C(2)-C(14)-C(7)	117.85(5)
C(5)-C(14)-C(7)	102.06(5)

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for $\text{C}_{17}\text{H}_{24}\text{O}_2\text{Si}$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Si(1)	16(1)	21(1)	24(1)	3(1)	-2(1)	-2(1)
O(1)	31(1)	38(1)	23(1)	1(1)	-9(1)	-13(1)
O(2)	28(1)	27(1)	20(1)	-8(1)	-4(1)	-4(1)
C(1)	16(1)	20(1)	18(1)	1(1)	-1(1)	-1(1)
C(2)	18(1)	22(1)	16(1)	-1(1)	1(1)	-2(1)
C(3)	29(1)	44(1)	16(1)	-5(1)	6(1)	-9(1)
C(4)	36(1)	36(1)	16(1)	5(1)	-2(1)	-9(1)
C(5)	24(1)	24(1)	17(1)	1(1)	-4(1)	-3(1)
C(6)	48(1)	27(1)	23(1)	-6(1)	-2(1)	-14(1)
C(7)	28(1)	17(1)	22(1)	-2(1)	1(1)	-1(1)
C(8)	36(1)	20(1)	25(1)	3(1)	1(1)	-6(1)

C(9)	24(1)	31(1)	23(1)	0(1)	3(1)	-10(1)
C(10)	19(1)	25(1)	15(1)	0(1)	1(1)	0(1)
C(11)	18(1)	18(1)	14(1)	-1(1)	-1(1)	-1(1)
C(12)	18(1)	17(1)	17(1)	-1(1)	-2(1)	0(1)
C(13)	26(1)	38(1)	16(1)	-2(1)	3(1)	2(1)
C(14)	18(1)	17(1)	14(1)	-1(1)	-1(1)	-1(1)
C(15)	26(1)	42(1)	38(1)	13(1)	-14(1)	-8(1)
C(16)	28(1)	23(1)	32(1)	3(1)	0(1)	-1(1)
C(17)	23(1)	34(1)	44(1)	-4(1)	11(1)	-2(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for $\text{C}_{17}\text{H}_{24}\text{O}_2\text{Si}$.

	x	y	z	U(eq)
H(3A)	8021	7360	5766	36
H(3B)	8589	8554	5713	36
H(4A)	6251	9202	5504	35
H(4B)	5973	8077	5015	35
H(5)	4594	8802	6679	26
H(6A)	5754	6336	5779	39
H(6B)	4413	5796	6364	39
H(7)	6840	6189	7300	27
H(8A)	4353	5581	7988	32
H(8B)	5617	5946	8735	32
H(9A)	3247	7279	8099	31
H(9B)	3326	6735	9171	31

H(13A)	5343	8847	10178	40
H(13B)	4728	7728	10499	40
H(13C)	3627	8596	10081	40
H(15A)	11435	8655	8893	53
H(15B)	10453	9504	9457	53
H(15C)	11920	9854	8895	53
H(16A)	8723	11138	8189	42
H(16B)	8955	11124	7012	42
H(16C)	10286	11441	7727	42
H(17A)	12136	9591	6701	50
H(17B)	10698	9510	6022	50
H(17C)	11294	8502	6594	50
