Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

for

Chemoselective Reduction of *α,β*-Unsaturated Ketones to Allylic Alcohols Under Catalyst-Free Conditions

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1. General information

All chemicals were purchased from Adamas Reagent, Ltd, Energy chemical company, J&K Scientific Ltd, Alfa Aesa chemical company and so forth. Unless otherwise stated, all experiments were conducted in a seal tube under air atmosphere. Reactions were monitored by TLC or GC-MS analysis. Flash column chromatography was performed over silica gel (200-300 mesh).

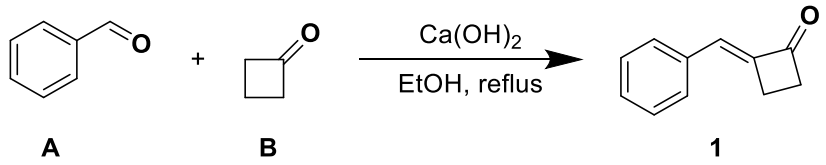
^{1}H-NMR and ^{13}C-NMR spectra were recorded in $CDCl_3$ on a Bruker Avance 500 spectrometer (500 MHz ^{1}H, 125 MHz ^{13}C) at room temperature. Chemical shifts were reported in ppm on the scale relative to $CDCl_3$ (δ = 7.26 for ^{1}H-NMR , δ = 77.00 for ^{13}C-NMR) or DMSO-d6 (δ = 2.50 for ^{1}H-NMR, δ = 39.60 for ^{13}C-NMR) as an internal reference. High resolution mass spectra were recorded using Q-TOF time-of-flight mass spectrometer. Coupling constants (*J*) were reported in Hertz (Hz).

The starting materials Acyclic α, β-Unsaturated Ketones were purchased from commercial suppliers. Other *Exo*-α, β-Unsaturated Cyclobutanones were synthetized according to methods reported by previous literatures.1 Exocyclic α, β-Unsaturated Pentanones were prepared according to the known methods reported by previous literatures.2

1 J. Li, Y. -F. Lu, Y. Zhu, Y. Nie, J. -F. Shen, Y. -G. Liu, D. -L. Liu, and W.-B Zhang, *Org. Lett*. **2019**, *21*, 4331.

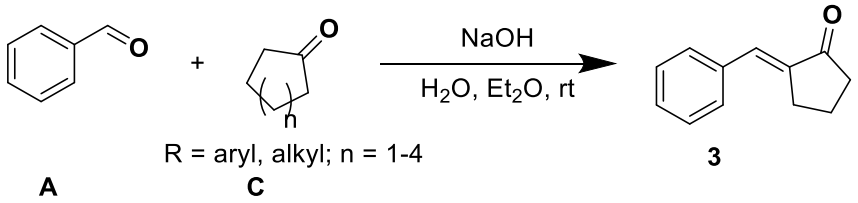
2 L. Zhang, M. Wang, M. -X. Zhou, Z. F. Zhang, M. Muraoka, W. B. Zhang, *Asian J. Org. Chem* 10.1002/ajoc.201900294

2.General procedure for the synthesis of 1



To a 100 mL round-bottomed flask was added 10 mol% of $Ca(OH)_2$, aldehyde A (20.0 mmol, 1.0 equiv), cyclobutanone B (60.0 mmol, 3.0 equiv) and anhydrous EtOH (40 mL) sequentially. The reaction was then heated at reflux under an N_2 atmosphere for 24 hours. The solvent was evaporated in vacuo to afford crude product, which was then purified by column chromatography (SiO_2, PE/EtOAc = 40/1) to afford pure four-membered exo-α,β-unsaturated cyclobutanone **1a** to **1r**. performed according to method reported by previous literature[1].

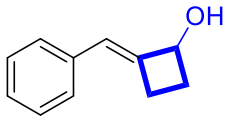
3.General procedure for the synthesis of 3



A mixture of an aldehyde **A** (10 mmol), cycloalkanone **C** (12 mmol), diethyl ether (15 mL), and 1N NaOH solution (15 mL) was stirred at room temperature for 24 h. The mixture was diluted with 50 mL of EtOAc. The aqueous layer was separated and extracted with EtOAc (3 × 20 mL). The combined organic phase was washed with water (3 × 20 mL) and dried over Na_2SO_4. The solvent was evaporated, and the residue was purified by silica gel column chromatography with PE/EtOAc (50/1) to give the corresponding cycloalkanone **3a** to **3n**, performed according to method reported by previous literature[2].

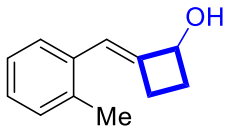
4. Characterization data for products

(*E*)-2-benzylidenecyclobutan-1-ol (2a)



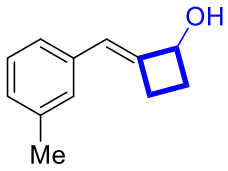
Compound **2a** (CAS: 2097787-96-5) : colorless oil (94% yield, 30.1 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.33 (t, *J* = 7.6 Hz, 2H), 7.27 (d, *J* = 7.2 Hz, 2H), 7.22 (t, *J* = 7.3 Hz, 1H), 6.44 (q, *J* = 2.4 Hz, 1H), 4.85 (d, *J* = 7.8 Hz, 1H), 2.82 – 2.74 (m, 1H), 2.67 (ddd, *J* = 15.9, 7.9, 2.8 Hz, 1H), 2.54 – 2.47 (m, 2H), 1.97 (tdd, *J* = 10.7, 9.0, 7.1 Hz, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 147.9, 136.9, 128.5, 127.8, 126.7, 120.1, 72.1, 31.9, 25.2.

(*E*)-2-(2-methylbenzylidene)cyclobutan-1-ol (2b)



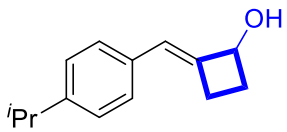
Compound **2b** (CAS: 2376476-72-9) : white solid (94% yield, 32.7 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.27 – 7.23 (m, 1H), 7.20 – 7.11 (m, 3H), 6.61 (q, *J* = 2.4 Hz, 1H), 4.86 (d, *J* = 8.2 Hz, 1H), 2.74 – 2.59 (m, 2H), 2.50 (dtd, *J* = 10.9, 8.2, 3.9 Hz, 1H), 2.36 (s, 3H), 2.13 (dd, *J* = 8.7, 2.0 Hz, 1H), 1.94 (tdd, *J* = 10.6, 9.2, 7.2 Hz, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 148.2, 135.7, 135.1, 130.3, 127.5, 126.8, 125.7, 117.3, 72.3, 31.9, 24.9, 19.9.

(*E*)-2-(3-methylbenzylidene)cyclobutan-1-ol (2c)



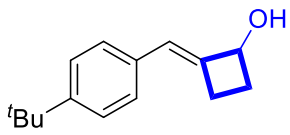
Compound **2c** (CAS: 2376476-80-9) : colorless oil (94% yield, 32.9 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.22 (t, *J* = 7.8 Hz, 1H), 7.10 – 7.06 (m, 2H), 7.04 (d, *J* = 7.6 Hz, 1H), 6.40 (q, *J* = 2.4 Hz, 1H), 4.84 (tt, *J* = 7.5, 2.7 Hz, 1H), 2.84 – 2.76 (m, 1H), 2.67 (dtd, *J* = 15.8, 8.9, 2.7 Hz, 1H), 2.51 (dd, *J* = 11.5, 8.9, 8.0, 3.7 Hz, 1H), 2.35 (s, 3H), 2.15 – 2.03 (m, 1H), 1.96 (tdd, *J* = 10.7, 9.0, 7.1 Hz, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 147.7, 137.9, 136.8, 128.6, 128.4, 127.5, 124.9, 120.1, 72.2, 31.9, 25.2, 21.5.

(*E*)-2-(4-isopropylbenzylidene)cyclobutan-1-ol (2d)



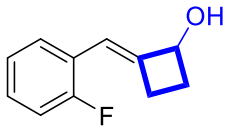
Compound **2d**: colorless oil (95% yield, 38.4 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.20 (s, 4H), 6.42 (q, *J* = 2.4 Hz, 1H), 4.85 (d, *J* = 7.3 Hz, 1H), 2.90 (hept, *J* = 6.8 Hz, 1H), 2.79 (ddtd, J = 16.0, 10.0, 3.5, 2.3 Hz, 1H), 2.66 (dtd, J = 15.8, 8.9, 2.7 Hz, 1H), 2.54 – 2.46 (m, 1H), 2.32 (s, 1H), 2.00 – 1.90 (m, 1H), 1.26 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (125 MHz, Chloroform-d) δ 147.5, 146.9, 134.4, 127.8, 126.5, 119.93, 72.2, 33.9, 31.9, 25.1, 23.9. HRMS (ESI, m/z) calcd for $C_{14}H_{18}O$ [M+H]+: 203.1430; found: 203.1427.

(*E*)-2-(4-(tert-butyl)benzylidene)cyclobutan-1-ol (2e)



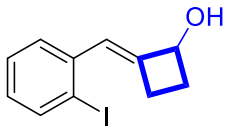
Compound **2e** (CAS: 2376476-98-9): colorless oil (85% yield, 36.7 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.41 – 7.36 (m, 2H), 7.24 (d, *J* = 8.4 Hz, 2H), 6.44 (q, *J* = 2.4 Hz, 1H), 4.87 (t, *J* = 7.6 Hz, 1H), 2.92 – 2.76 (m, 1H), 2.69 (dtd, *J* = 15.8, 9.0, 2.8 Hz, 1H), 2.53 (m, *J* = 11.3, 8.9, 7.9, 3.6 Hz, 1H), 2.23 (s, 1H), 1.97 (m, *J* = 11.1, 10.4, 9.0, 7.0 Hz, 1H), 1.35 (s, 9H). ^{13}C NMR (125 MHz, Chloroform-d) δ 149.7, 147.0, 134.0, 127.6, 125.4, 119.8, 72.2, 34.5, 31.9, 31.3, 25.1.

(*E*)-2-(2-fluorobenzylidene)cyclobutan-1-ol (2f)



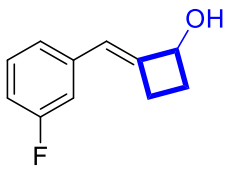
Compound **2f**: colorless oil (60% yield, 21.4 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.29 (td, *J* = 7.7, 1.8 Hz, 1H), 7.18 (tdd, *J* = 7.4, 5.1, 1.8 Hz, 1H), 7.11 – 7.00 (m, 2H), 6.62 (q, *J* = 2.5 Hz, 1H), 4.85 (d, *J* = 8.1 Hz, 1H), 2.68 (m, *J* = 32.9, 15.9, 7.8, 3.0 Hz, 2H), 2.50 (ddt, *J* = 14.6, 8.4, 3.7 Hz, 1H), 2.25 (s, 1H), 1.95 (tdd, *J* = 10.7, 9.2, 7.1 Hz, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 159.5 (*J* = 246.2 Hz), 150.0, 128.7 (*J* = 3.7 Hz), 128.2 (*J* = 7.5 Hz), 124.4 (*J* = 13.7 Hz), 123.8 (*J* = 3.75 Hz), 115.5 (*J* = 22.5), 111.9, 72.2, 31.6, 25.0. HRMS (ESI, m/z) calcd for $C_{11}H_{11}FO$ [M+H]+: 179.0867; found: 179.0866.

(*E*)-2-(2-iodobenzylidene)cyclobutan-1-ol (2g)



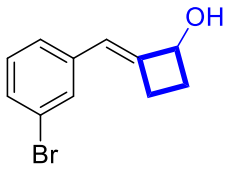
Compound **2g**: colorless oil (60% yield, 34.3 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.85 (t, *J* = 6.6 Hz, 1H), 7.29 – 7.24 (m, 2H), 6.92 – 6.86 (m, 1H), 6.60 (t, *J* = 2.1 Hz, 1H), 4.85 (d, *J* = 8.0 Hz, 1H), 2.55 (m, *J* = 57.6, 12.9, 7.1, 3.6 Hz, 4H), 2.00 – 1.90 (m, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 150.5, 139.6, 139.1, 128.3, 128.2, 128.0, 123.7, 100.1, 72.1, 31.7, 24.5. HRMS (ESI, m/z) calcd for $C_{11}H_{11}IO$ [M+H]+: 286.9927; found:286.9929.

(*E*)-2-(3-fluorobenzylidene)cyclobutan-1-ol (2h)



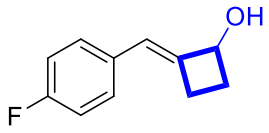
Compound **2h**: colorless oil (65% yield, 23.1 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.30 – 7.25 (m, 1H), 7.02 (dt, *J* = 7.8, 1.1 Hz, 1H), 6.97 – 6.87 (m, 2H), 6.40 (d, *J* = 2.5 Hz, 1H), 4.84 (d, *J* = 8.1 Hz, 1H), 2.82 – 2.73 (m, 1H), 2.70 – 2.61 (m, 1H), 2.56 – 2.49 (m, 1H), 2.12 (d, *J* = 7.9 Hz, 1H), 1.97 (tdd, *J* = 10.7, 9.0, 7.1 Hz, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 162.9 (*J* = 243.7 Hz), 149.4, 139.0 (*J* = 7.5 Hz), 129.8 (*J* = 7.5 Hz), 123.6 (*J* = 2.5 Hz), 119.1, 114.2 (*J* = 21.2 Hz), 113.5 (*J* = 20 Hz), 72.1, 31.8, 25.1. HRMS (ESI, m/z) calcd for $C_{11}H_{11}FO$ [M+H]+: 179.0867; found: 179.0866.

(*E*)-2-(3-bromobenzylidene)cyclobutan-1-ol (2i)



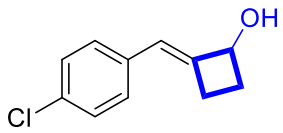
Compound **2i**: colorless oil (67% yield, 32.0 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.39 (s, 1H), 7.32 (dt, *J* = 6.7, 2.2 Hz, 1H), 7.17 (d, *J* = 6.5 Hz, 2H), 6.35 (q, *J* = 2.4 Hz, 1H), 4.84 (d, *J* = 7.8 Hz, 1H), 2.80 – 2.72 (m, 1H), 2.66 (dtd, *J* = 15.9, 9.0, 2.8 Hz, 1H), 2.56 – 2.48 (m, 1H), 2.11 (d, *J* = 8.3 Hz, 1H), 1.97 (tdd, *J* = 10.6, 9.0, 7.1 Hz, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 149.6, 138.9, 130.6, 129.9, 129.6, 126.3, 122.6, 118.8, 72.1, 31.8, 25.1. HRMS (ESI, m/z) calcd for $C_{11}H_{11}BrO$ [M+H]+: 239.0066; found: 239.0063.

(*E*)-2-(4-fluorobenzylidene)cyclobutan-1-ol (2j)



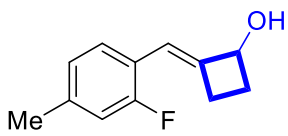
Compound **2j**: colorless oil (95% yield, 33.8 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.23 – 7.16 (m, 2H), 7.00 (t, *J* = 8.7 Hz, 2H), 6.38 (q, *J* = 2.4 Hz, 1H), 4.83 (s, 1H), 2.71 (dd, *J* = 10.2, 2.8 Hz, 1H), 2.65 – 2.59 (m, 1H), 2.52 – 2.46 (m, 1H), 1.96 (dd, J = 9.1, 7.0 Hz, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ161.6 (*J* = 245 Hz), 147.3 (*J* = 2.5 Hz), 133.0 (*J* = 3.7 Hz), 129.2 (*J* = 7.5 Hz), 119.0, 115.3 (*J* = 21.2 Hz), 72.0, 31.8, 25.0. HRMS (ESI, m/z) calcd for $C_{11}H_{11}FO$ [M+H]+: 179.0867; found: 179.0868.

(*E*)-2-(4-chlorobenzylidene)cyclobutan-1-ol (2k)



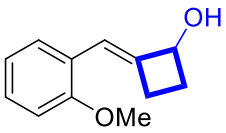
Compound **2k** (CAS: 2376476-98-9): colorless oil (95% yield, 36.9 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.27 (d, *J* = 8.5 Hz, 2H), 7.16 (d, *J* = 8.5 Hz, 2H), 6.37 (q, *J* = 2.4 Hz, 1H), 4.87 – 4.79 (m, 1H), 2.78 – 2.68 (m, 1H), 2.63 (dtd, *J* = 15.9, 9.0, 2.7 Hz, 1H), 2.54 – 2.46 (m, 1H), 2.27 (d, *J* = 8.0 Hz, 1H), 1.97 (tdd, *J* = 10.7, 9.0, 7.1 Hz, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 148.6, 135.3, 132.3, 129.0, 128.6, 119.0, 72.1, 31.8, 25.1.

(*E*)-2-(2-fluoro-4-methylbenzylidene)cyclobutan-1-ol (2l)



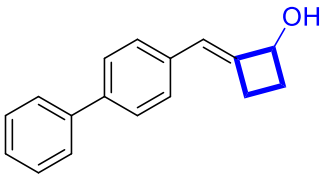
Compound **2l**: colorless oil (66% yield, 25.3 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.06 (dd, *J* = 7.5, 2.2 Hz, 1H), 6.96 (ddd, *J* = 7.6, 5.0, 2.2 Hz, 1H), 6.91 (dd, *J* = 10.1, 8.3 Hz, 1H), 6.58 (d, *J* = 2.5 Hz, 1H), 4.84 (td, *J* = 7.7, 6.3, 3.8 Hz, 1H), 2.75 – 2.58 (m, 2H), 2.53 – 2.45 (m, 1H), 2.30 (s, 3H), 2.29 (s, 1H), 1.94 (tdd, *J* = 10.6, 9.2, 7.1 Hz, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 158.2 (*J* = 245 Hz), 149.7 (*J* = 2.5 Hz), 133.0(*J* = 3.7 Hz), 129.1 (*J* = 3.7 Hz), 128.6 (*J* = 7.5 Hz), 123.9 (*J* = 12.5 Hz), 115.1 (*J* = 21.2 Hz), 112.0 (*J* = 5 Hz), 72.2, 31.5, 25.0 (*J* = 1.2 Hz), , 20.8. HRMS (ESI, m/z) calcd for $C_{12}H_{13}FO$ [M+H]+: 193.1023; found: 193.1029.

(*E*)-2-(2-methoxybenzylidene)cyclobutan-1-ol (2m)



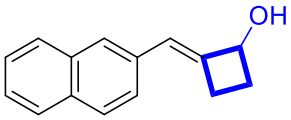
Compound **2m**: colorless oil (70% yield, 26.6 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.26 – 7.24 (m, 1H), 7.20 (td, *J* = 7.8, 1.7 Hz, 1H), 6.94 – 6.86 (m, 2H), 6.78 (q, *J* = 2.4 Hz, 1H), 4.84 (s, 1H), 3.84 (s, 3H), 2.75 – 2.59 (m, 2H), 2.53 – 2.45 (m, 1H), 2.12 – 2.06 (m, 1H), 1.97 – 1.87 (m, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 156.5, 147.8, 128.3, 128.0, 125.5, 120.3, 114.0, 110.7, 72.4, 55.4, 32.0, 25.0. HRMS (ESI, m/z) calcd for $C_{12}H_{14}O_2$ [M+H]+: 191.1067; found: 191.1065.

(*E*)-2-([1,1'-biphenyl]-4-ylmethylene)cyclobutan-1-ol (2n)



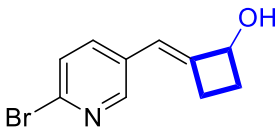
Compound **2n** (CAS: 2376477-00-6): colorless oil (78% yield, 36.8 mg);^{1}H NMR (500 MHz, Chloroform-d) δ 7.63 – 7.56 (m, 4H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.37 – 7.33 (m, 3H), 6.48 (q, *J* = 2.4 Hz, 1H), 4.88 (tt, *J* = 7.5, 2.6 Hz, 1H), 2.89 – 2.79 (m, 1H), 2.71 (dtd, *J* = 15.9, 9.0, 2.8 Hz, 1H), 2.59 – 2.50 (m, 1H), 1.99 (tdd, *J* = 10.7, 9.1, 7.1 Hz, 2H). ^{13}C NMR (125 MHz, Chloroform-d) δ 148.1, 140.8, 139.4, 135.9, 128.8, 128.2, 127.3, 127.1, 126.9, 119.7, 72.3, 31.9, 25.3.

(*E*)-2-(naphthalen-2-ylmethylene)cyclobutan-1-ol (2o)



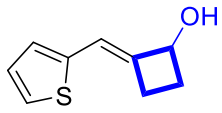
Compound **2o** (CAS: 2376477-04-0): colorless oil (72% yield, 30.2 mg);^{1}H NMR (500 MHz, Chloroform-d) δ 7.82 – 7.76 (m, 3H), 7.68 (d, *J* = 1.7 Hz, 1H), 7.49 – 7.42 (m, 3H), 6.59 (q, *J* = 2.4 Hz, 1H), 4.90 (s, 1H), 2.94 – 2.85 (m, 1H), 2.82 – 2.72 (m, 1H), 2.59 – 2.50 (m, 1H), 2.23 (s, 1H), 2.00 (tdd, *J* = 10.7, 9.0, 7.1 Hz, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 148.4, 134.4, 133.6, 132.3, 127.9, 127.9, 127.6, 126.8, 126.2, 125.9, 125.7, 120.3, 72.3, 31.9, 25.3.

(*E*)-2-((6-bromopyridin-3-yl)methylene)cyclobutan-1-ol (2p)



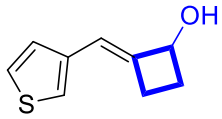
Compound **2p**: colorless oil (55% yield, 26.3 mg);^{1}H NMR (500 MHz, Chloroform-d) δ 8.21 (d, *J* = 2.1 Hz, 1H), 7.41 – 7.37 (m, 2H), 6.33 (q, *J* = 2.4 Hz, 1H), 4.85 (d, *J* = 8.2 Hz, 1H), 2.79 (s, 1H), 2.68 – 2.58 (m, 2H), 2.51 (dtd, *J* = 11.3, 8.4, 3.9 Hz, 1H), 2.02 – 1.97 (m, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 151.8, 149.1, 139.4, 136.9, 131.9, 127.8, 115.3, 71.8, 31.5, 25.0. HRMS (ESI, m/z) calcd for $C_{10}H_{10}BrNO$ [M+H]+: 240.0019; found:

(*E*)-2-(thiophen-2-ylmethylene)cyclobutan-1-ol (2q)



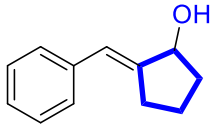
Compound **2q**: colorless oil (84% yield, 27.9 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.23 (d, *J* = 5.1 Hz, 1H), 7.00 (t, *J* = 4.3 Hz, 1H), 6.92 (d, *J* = 3.5 Hz, 1H), 6.66 (s, 1H), 4.83 (dt, *J* = 7.1, 3.5 Hz, 1H), 2.82 (ddd, *J* = 19.2, 8.1, 5.0 Hz, 1H), 2.58 – 2.49 (m, 2H), 2.14 – 2.05 (m, 1H), 1.99 – 1.91 (m, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 146.3, 140.8, 127.0, 125.6, 124.9, 114.2, 71.8, 31.3, 24.8. HRMS (ESI, m/z) calcd for $C_9H_{10}OS$ [M+H]+: 167.0525; found: 167.0532.

2-(thiophen-3-ylmethyl)cyclobutan-1-ol (2r)



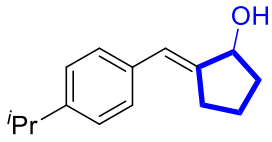
Compound **2r**: colorless oil (78% yield, 25.9 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.28 (dd, *J* = 4.9, 3.0 Hz, 1H), 7.12 – 7.00 (m, 2H), 6.47 (q, *J* = 2.4 Hz, 1H), 4.82 (t, *J* = 7.6 Hz, 1H), 2.76 (ddq, *J* = 16.2, 9.8, 3.2 Hz, 1H), 2.59 (dtd, *J* = 15.6, 8.9, 2.7 Hz, 1H), 2.54 – 2.43 (m, 1H), 2.12 (s, 1H), 1.97 – 1.91 (m, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 146.6, 138.4, 127.1, 125.5, 121.9, 114.7, 71.9, 31.4, 24.8. HRMS (ESI, m/z) calcd for $C_9H_{10}OS$ [M+H]+: 167.0525; found: 167.0532.

(*E*)-2-benzylidenecyclopentan-1-ol (4a)



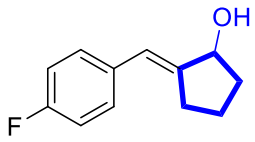
Compound **4a** (CAS: 100121-37-7): colorless oil (73% yield, 25.4 mg);^{1}H NMR (500 MHz, Chloroform-d) δ 7.38 – 7.32 (m, 4H), 7.24 – 7.19 (m, 1H), 6.58 (q, *J* = 2.4 Hz, 1H), 4.60 (ddt, *J* = 5.4, 3.6, 1.7 Hz, 1H), 2.74 (dddt, *J* = 13.6, 8.4, 4.4, 2.8 Hz, 1H), 2.59 (m, *J* = 17.2, 8.3, 5.4, 2.6 Hz, 1H), 2.03 – 1.92 (m, 2H), 1.79 – 1.73 (m, 1H), 1.72 (d, *J* = 8.8 Hz, 1H), 1.69 – 1.62 (m, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 147.8, 137.8, 128.4, 128.3, 126.6, 123.7, 34.9, 29.4, 22.6.

(*E*)-2-(4-isopropylbenzylidene)cyclopentan-1-ol (4b)



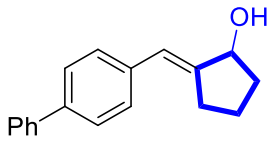
Compound **4b**: colorless oil (76% yield, 32.8 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.30 (d, *J* = 8.2 Hz, 2H), 7.21 (d, *J* = 8.2 Hz, 2H), 6.56 (s, 1H), 4.58 (d, *J* = 3.6 Hz, 1H), 2.90 (p, *J* = 6.9 Hz, 1H), 2.79 – 2.69 (m, 1H), 2.58 (dtd, *J* = 17.6, 5.7, 2.9 Hz, 1H), 2.02 – 1.88 (m, 2H), 1.79 – 1.70 (m, 1H), 1.66 (t, *J* = 5.5 Hz, 1H), 1.62 (dd, *J* = 11.4, 5.0 Hz, 1H), 1.26 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (125 MHz, Chloroform-d) δ 147.4, 146.9, 135.3, 128.4, 126.4, 123.6, 34.9, 33.8, 29.4, 23.9, 22.6. HRMS (ESI, m/z) calcd for $C_{15}H_{20}O$ [M+H]+: 217.1587; found: 217.1579.

(*E*)-2-(4-fluorobenzylidene)cyclopentan-1-ol (4c)



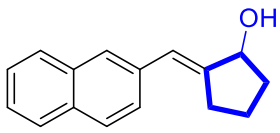
Compound **4c** (CAS: 2087452-28-4): colorless oil (74% yield, 28.4 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.31 (dd, *J* = 8.6, 5.6 Hz, 2H), 7.02 (t, *J* = 8.7 Hz, 2H), 6.54 (d, *J* = 2.2 Hz, 1H), 4.58 (t, *J* = 5.7 Hz, 1H), 2.68 (m, *J* = 16.9, 8.4, 6.0, 2.3 Hz, 1H), 2.53 (m, *J* = 17.0, 8.2, 5.3, 2.6 Hz, 1H), 2.05 – 1.89 (m, 2H), 1.78 – 1.72 (m, 1H), 1.69 (s, 1H), 1.67 – 1.60 (m, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 161.5 (*J* = 245 Hz), 147.3 (*J* = 1.2 Hz), 133.9 (*J* = 3.7 Hz), 129.9 (*J* = 7.5 Hz), 122.6, 115.2 (*J* = 21.1 Hz), 77.3, 34.9, 29.2, 22.5.

(*E*)-2-([1,1'-biphenyl]-4-ylmethylene)cyclopentan-1-ol (4d)



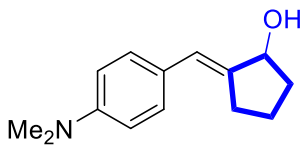
Compound **4d** (CAS: 2097787-89-6): colorless oil (64% yield, 32.0 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.63 – 7.58 (m, 4H), 7.47 – 7.43 (m, 4H), 7.37 – 7.33 (m, 1H), 6.63 (d, *J* = 2.1 Hz, 1H), 4.63 (t, *J* = 5.1 Hz, 1H), 2.78 (ddt, *J* = 13.3, 6.6, 2.3 Hz, 1H), 2.64 (m, *J* = 11.7, 8.7, 5.3, 2.6 Hz, 1H), 2.00 (ddt, *J* = 16.0, 12.0, 6.2 Hz, 2H), 1.81 – 1.68 (m, 2H), 1.68 (s, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 148.0, 140.7, 139.3, 136.8, 128.9, 128.8, 127.3, 127.0, 126.9, 123.3, 34.9, 29.5, 22.6.

(*E*)-2-(naphthalen-2-ylmethylene)cyclopentan-1-ol (4e)



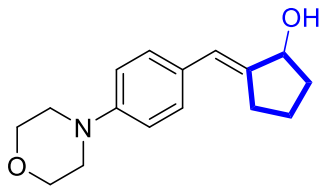
Compound **4e** (CAS: 2087452-34-2): colorless oil (60% yield, 26.9 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.83 – 7.79 (m, 4H), 7.52 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.46 (tt, *J* = 7.1, 5.3 Hz, 2H), 6.74 (q, *J* = 2.3 Hz, 1H), 4.65 (t, *J* = 5.6 Hz, 1H), 2.85 (m, *J* = 19.5, 8.4, 6.0, 2.3 Hz, 1H), 2.70 (m, *J* = 17.2, 8.3, 5.3, 2.6 Hz, 1H), 2.06 – 1.95 (m, 2H), 1.82 – 1.76 (m, 1H), 1.72 (d, *J* = 5.9 Hz, 1H), 1.68 (ddd, *J* = 12.1, 5.8, 2.7 Hz, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 148.3, 135.4, 133.5, 132.2, 128.0, 127.8, 127.6, 127.2, 126.8, 126.1, 125.8, 123.8, 34.9, 29.5, 22.6.

(*E*)-2-(4-(dimethylamino)benzylidene)cyclopentan-1-ol (4f)



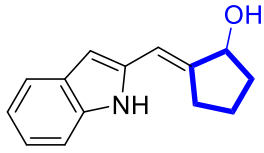
Compound **4f**: colorless oil (46% yield, 20.0 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.31 – 7.27 (m, 2H), 6.73 – 6.70 (m, 2H), 6.49 (q, *J* = 2.3 Hz, 1H), 4.60 – 4.55 (m, 1H), 2.96 (s, 6H), 2.76 – 2.69 (m, 1H), 2.55 (m, *J* = 17.3, 8.7, 6.2, 2.6 Hz, 1H), 2.02 – 1.95 (m, 1H), 1.93 – 1.86 (m, 1H), 1.75 (ddd, *J* = 12.1, 8.5, 6.1 Hz, 2H), 1.70 – 1.62 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-d) δ 149.2, 143.6, 129.5, 126.5, 123.8, 112.3, 40.5, 34.9, 29.4, 22.9. HRMS (ESI, m/z) calcd for $C_{14}H_{19}ON$ [M+H]+: 218.1539; found: 218.1534.

(*E*)-2-(4-morpholinobenzylidene)cyclopentan-1-ol (4g)



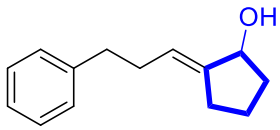
Compound **4g**: colorless oil (77% yield, 39.9 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.28 (d, *J* = 8.8 Hz, 2H), 6.87 (d, *J* = 8.8 Hz, 2H), 6.49 (q, *J* = 2.3 Hz, 1H), 4.56 (t, *J* = 5.3 Hz, 1H), 3.86 – 3.84 (m, 4H), 3.17 – 3.14 (m, 4H), 2.74 – 2.66 (m, 1H), 2.54 (m, *J* = 17.2, 8.5, 5.9, 2.6 Hz, 1H), 2.00 (s, 1H), 1.92 (ddt, *J* = 25.1, 12.9, 6.4 Hz, 2H), 1.73 (ddt, *J* = 11.9, 8.4, 6.4 Hz, 1H), 1.63 (dq, *J* = 12.8, 6.7 Hz, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 149.7, 145.2, 129.8, 129.4, 123.2, 115.3, 77.4, 66.8, 49.1, 34.9, 29.4, 22.7. HRMS (ESI, m/z) calcd for $C_{16}H_{21}NO_2$ [M+H]+: 260.1645; found: 260.1643.

(*E*)-2-((1H-indol-2-yl)methylene)cyclopentan-1-ol (4h)



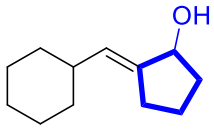
Compound **4h**: colorless oil (95% yield, 40.5 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 8.12 (s, 1H), 7.59 (d, *J* = 7.9 Hz, 1H), 7.30 (d, *J* = 9.0 Hz, 1H), 7.16 (t, *J* = 7.6 Hz, 1H), 7.10 (t, *J* = 7.4 Hz, 1H), 6.55 – 6.47 (m, 2H), 4.62 (d, *J* = 5.9 Hz, 1H), 2.75 (ddt, *J* = 17.4, 9.0, 2.3 Hz, 1H), 2.63 (dtt, *J* = 15.3, 6.4, 2.9 Hz, 1H), 2.02 (ddd, *J* = 10.5, 7.6, 4.4 Hz, 2H), 1.79 (ddd, *J* = 8.2, 6.1, 2.0 Hz, 1H), 1.65 (ddd, *J* = 14.2, 7.0, 5.2 Hz, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 147.5, 136.1, 136.0, 128.9, 122.2, 120.5, 120.0, 114.2, 110.7, 102.5, 35.3, 29.8, 22.2. HRMS (ESI, m/z) calcd for $C_{14}H_{15}ON$ [M+H]+: 214.1226; found: 214.1223.

(*E*)-2-(3-phenylpropylidene)cyclopentan-1-ol (4i)



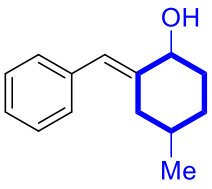
Compound **4i** (CAS: 1542149-57-4): colorless oil (52% yield, 21.0 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.31 – 7.26 (m, 2H), 7.21 – 7.17 (m, 3H), 5.58 (ddd, *J* = 8.9, 4.6, 1.7 Hz, 1H), 4.37 (d, *J* = 4.9 Hz, 1H), 2.69 (t, *J* = 7.8 Hz, 2H), 2.35 – 2.31 (m, 2H), 2.31 – 2.25 (m, 1H), 2.14 – 2.06 (m, 1H), 1.84 – 1.77 (m, 2H), 1.63 – 1.57 (m, 2H), 1.44 (s, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 146.7, 142.0, 128.5, 128.3, 125.8, 123.2, 35.6, 35.5, 31.2, 26.9, 21.9.

(*E*)-2-(cyclohexylmethylene)cyclopentan-1-ol (4j)



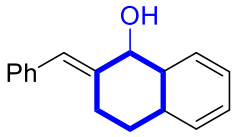
Compound **4j** (CAS: 1824873-18-8): colorless oil (80% yield, 28.8 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 5.36 (d, *J* = 8.9 Hz, 1H), 4.35 (t, *J* = 4.9 Hz, 1H), 2.42 – 2.31 (m, 1H), 2.24 – 2.14 (m, 1H), 2.04 (td, *J* = 11.1, 7.7 Hz, 1H), 1.88 – 1.77 (m, 2H), 1.74 – 1.67 (m, 2H), 1.62 (dt, *J* = 10.1, 3.8 Hz, 5H), 1.53 (s, 1H), 1.32 – 1.14 (m, 4H), 1.06 (ddt, *J* = 15.5, 12.3, 6.1 Hz, 2H). ^{13}C NMR (125 MHz, Chloroform-d) δ 144.0, 130.2, 75.7, 38.5, 35.5, 32.8, 32.7, 26.7, 26.1, 25.9, 22.1.

(*E*)-2-benzylidene-4-methylcyclohexan-1-ol (4k)



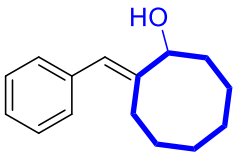
Compound **4k**: colorless oil (90% yield, 36.4 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.33 (dd, *J* = 8.1, 7.0 Hz, 2H), 7.24 – 7.19 (m, 3H), 6.57 (s, 1H), 4.17 (ddd, *J* = 11.0, 4.8, 1.9 Hz, 1H), 2.90 (dd, *J* = 9.9, 2.4 Hz, 1H), 2.17 (ddt, *J* = 12.2, 4.8, 3.7 Hz, 1H), 1.86 – 1.81 (m, 1H), 1.77 (s, 1H), 1.57 – 1.52 (m, 2H), 1.45 (tdd, *J* = 12.4, 10.9, 3.9 Hz, 1H), 1.26 (ddt, *J* = 10.7, 9.4, 3.7 Hz, 1H), 0.92 (d, *J* = 6.0 Hz, 3H). ^{13}C NMR (125 MHz, Chloroform-d) δ 144.0, 137.9, 128.9, 128.1, 126.1, 118.9, 73.1, 36.7, 36.4, 33.8, 33.1, 21.8. HRMS (ESI, m/z) calcd for $C_{14}H_{18}O$ [M+H]+: 203.1430; found: 203.1433.

(*E*)-2-benzylidene-1,2,3,4-tetrahydronaphthalen-1-ol (4l)



Compound **4l** (CAS: 6261-31-0): colorless oil (87% yield, 41.4 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.54 – 7.50 (m, 1H), 7.41 – 7.38 (m, 2H), 7.35 – 7.33 (m, 2H), 7.31 – 7.27 (m, 3H), 7.20 – 7.17 (m, 1H), 6.77 (s, 1H), 5.20 (s, 1H), 3.07 – 3.01 (m, 1H), 2.88 (d, *J* = 3.4 Hz, 1H), 2.86 – 2.81 (m, 2H), 2.38 (s, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 140.9, 138.8, 137.4, 137.4, 128.9, 128.3, 127.9, 126.7, 126.6, 125.4, 73.9, 29.9, 24.2.

(*E*)-2-benzylidenecyclooctan-1-ol (4m)

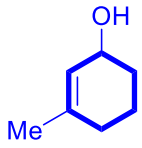


Compound **4m** (CAS: 94410-41-0): colorless oil (87% yield, 37.6 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.34 (d, *J* = 4.4 Hz, 4H), 7.23 (q, *J* = 4.4 Hz, 1H), 6.55 (s, 1H), 4.26 (dd, *J* = 10.0, 4.4 Hz, 1H), 2.59 – 2.53 (m, 1H), 2.47 (ddd, *J* = 14.5, 10.4, 4.5 Hz, 1H), 1.96 (ddd, *J* = 14.7, 7.6, 3.9 Hz, 1H), 1.72 – 1.56 (m, 6H), 1.47 (ddd, *J* = 15.3, 10.4, 4.4 Hz, 4H). ^{13}C NMR (125 MHz, Chloroform-d) δ 146.3, 137.7, 128.6, 128.3, 127.9, 126.5, 32.8, 26.9, 26.1, 25.7, 25.7, 22.9.

cyclohex-2-en-1-ol (4n)

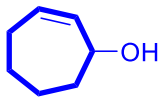
Compound **4n** (CAS: 822-67-3): colorless oil (90% yield, 17.6 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 5.86 – 5.80 (m, 1H), 5.77 – 5.71 (m, 1H), 4.19 (s, 1H), 2.06 – 1.92 (m, 2H), 1.90 – 1.84 (m, 1H), 1.77 – 1.68 (m, 1H), 1.61 (m, *J* = 10.9, 6.7, 4.8, 2.5 Hz, 2H), 1.24 (s, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 130.6, 129.8, 65.5, 32.0, 25.0, 18.9.

3-methylcyclohex-2-en-1-ol (4o)



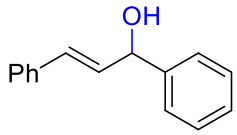
Compound **4o** (CAS: 21378-21-2): colorless oil (70% yield, 15.7 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 5.47 (dq, *J* = 3.4, 1.7 Hz, 1H), 4.15 (d, *J* = 4.5 Hz, 1H), 1.96 – 1.84 (m, 2H), 1.80 – 1.69 (m, 3H), 1.66 (s, 3H), 1.54 (ddd, *J* = 8.2, 4.7, 1.8 Hz, 2H). ^{13}C NMR (125 MHz, Chloroform-d) δ 138.6, 124.3, 65.8, 31.7, 30.1, 23.6, 19.0.

cyclohept-2-en-1-ol (4p)



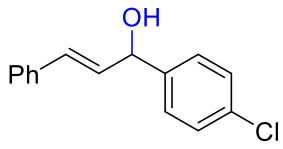
Compound **4p** (CAS: 4096-38-2): colorless oil (41% yield, 9.2 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 5.72 – 5.67 (m, 2H), 4.35 (dd, *J* = 9.5, 2.8 Hz, 1H), 2.32 (d, *J* = 9.2 Hz, 1H), 2.14 (dd, *J* = 12.9, 5.6 Hz, 1H), 2.02 – 1.96 (m, 1H), 1.88 (ddd, *J* = 9.9, 6.1, 3.7 Hz, 1H), 1.83 (dt, *J* = 12.2, 2.8 Hz, 1H), 1.67 – 1.61 (m, 1H), 1.59 – 1.50 (m, 2H), 1.32 (dtt, *J* = 14.4, 7.8, 3.7 Hz, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 137.9, 129.9, 72.0, 36.7, 28.6, 26.9, 26.7.

(*E*)-1,3-diphenylprop-2-en-1-ol (6a)



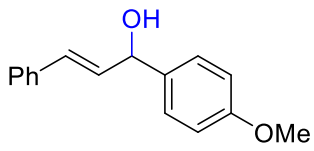
Compound **6a** (CAS: 4663-33-6): colorless oil (94% yield, 39.5 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.46 (d, *J* = 8.1 Hz, 2H), 7.40 (td, *J* = 8.0, 1.8 Hz, 4H), 7.33 (td, *J* = 8.1, 6.8, 3.9 Hz, 3H), 7.29 – 7.25 (m, 1H), 6.70 (dd, *J* = 15.8, 1.2 Hz, 1H), 6.41 (dd, *J* = 15.8, 6.5 Hz, 1H), 5.39 (d, *J* = 6.5 Hz, 1H), 2.36 (s, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 142.9, 136.6, 131.6, 130.6, 128.7, 128.6, 127.8, 126.7, 126.4, 75.1.

(*E*)-1-(4-chlorophenyl)-3-phenylprop-2-en-1-ol (6b)



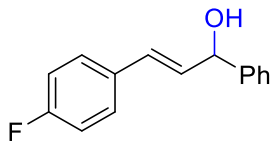
Compound **6b** (CAS: 166823-887): colorless oil (93% yield, 45.4 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.40 – 7.37 (m, 2H), 7.37 – 7.34 (m, 4H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.29 – 7.26 (m, 1H), 6.66 (dd, *J* = 15.8, 1.2 Hz, 1H), 6.32 (dd, *J* = 15.8, 6.7 Hz, 1H), 5.34 (d, *J* = 6.7 Hz, 1H), 2.42 (s, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 141.2, 136.3, 133.5, 131.1, 131.1, 128.7, 128.7, 128.0, 127.8, 126.7, 74.5.

(*E*)-1-(4-methoxyphenyl)-3-phenylprop-2-en-1-ol (6c)



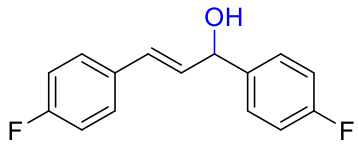
Compound **6c** (CAS: 13677-45-7)**:** colorless oil (86% yield, 41.3 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.41 – 7.30 (m, 6H), 7.25 (d, *J* = 5.7 Hz, 1H), 6.91 (d, *J* = 8.7 Hz, 2H), 6.67 (dd, *J* = 15.9, 1.2 Hz, 1H), 6.39 (dd, *J* = 15.8, 6.4 Hz, 1H), 5.34 (d, *J* = 6.3 Hz, 1H), 3.81 (s, 3H), 2.24 (s, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 159.3, 136.7, 135.1, 131.8, 130.2, 128.6, 127.7, 126.63, 114.1, 74.6, 55.3.

(*E*)-3-(4-fluorophenyl)-1-phenylprop-2-en-1-ol (6d)



Compound **6d** (CAS: 166823-97-8): colorless oil (91% yield, 41.5 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.36 – 7.32 (m, 4H), 7.21 (d, *J* = 7.9 Hz, 2H), 7.01 (t, *J* = 8.7 Hz, 2H), 6.63 (dd, *J* = 15.9, 1.2 Hz, 1H), 6.31 (dd, *J* = 15.9, 6.4 Hz, 1H), 5.33 (d, *J* = 6.4 Hz, 1H), 2.42 (s, 1H), 2.38 (s, 3H). ^{13}C NMR (125 MHz, Chloroform-d) δ 162.4 (*J* = 245 Hz), 139.9, 137.6, 131.6, 129.4, 129.1, 128.2 (*J* = 8.8 Hz), 126.4, 115.5 (*J* = 21.3 Hz), 74.9, 21.2.

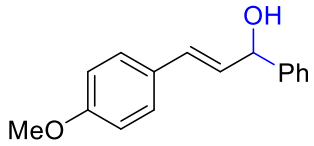
(*E*)-1,3-bis(4-fluorophenyl)prop-2-en-1-ol (6e)



Compound **6e** (CAS: 166823-99-0): colorless oil (83% yield, 40.8 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.40 – 7.30 (m, 4H), 7.08 – 6.97 (m, 4H), 6.60 (d, *J* = 15.8 Hz, 1H), 6.25 (dd, *J* = 15.8, 6.5 Hz, 1H), 5.32 (d, *J* = 6.5 Hz, 1H), 2.60 (s, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 162.5 (*J* = 245 Hz), 162.4 (*J* = 245 Hz), 138.5, 132.6, 131.1, 129.6, 128.2 (*J* = 8,8 Hz), 128.1(*J* = 8.8 Hz), 115.6 (*J* = 21.3 Hz), 115.5 (*J* = 21.3 Hz), 74.4.

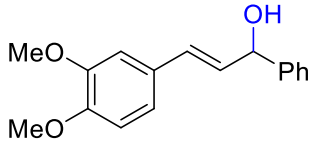
x

(*E*)-3-(4-methoxyphenyl)-1-phenylprop-2-en-1-ol (6f)



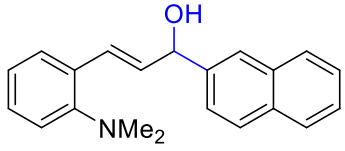
Compound **6f** (CAS: 39212-13-0): colorless oil (95% yield, 45.6 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.46 – 7.42 (m, 2H), 7.38 (dd, *J* = 8.3, 6.7 Hz, 2H), 7.34 – 7.28 (m, 3H), 6.88 – 6.84 (m, 2H), 6.62 (d, *J* = 15.7 Hz, 1H), 6.26 (dd, *J* = 15.8, 6.8 Hz, 1H), 5.35 (d, *J* = 5.2 Hz, 1H), 3.81 (s, 3H), 2.32 (s, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 159.4, 143.1, 130.2, 129.5, 129.4, 128.6, 127.9, 127.7, 126.3, 114.0, 75.3, 55.3.

(*E*)-3-(3,4-dimethoxyphenyl)-1-phenylprop-2-en-1-ol (6g)



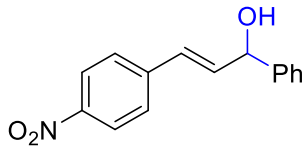
Compound **6g** (CAS: 338953-03-0): colorless oil (80% yield, 43.2 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.46 – 7.42 (m, 2H), 7.37 (t, *J* = 7.6 Hz, 2H), 7.30 (d, *J* = 7.2 Hz, 1H), 6.94 – 6.90 (m, 2H), 6.80 (d, *J* = 8.1 Hz, 1H), 6.60 (dd, *J* = 15.7, 1.2 Hz, 1H), 6.25 (dd, *J* = 15.8, 6.7 Hz, 1H), 5.36 (d, *J* = 6.6 Hz, 1H), 3.86 (s, 6H), 2.33 (s, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 149.1, 149.1, 143.0, 130.5, 129.7, 129.7, 128.6, 127.7, 126.3, 120.0, 111.2, 109.0, 75.2, 55.9, 55.8.

(*E*)-3-(2-(dimethylamino)phenyl)-1-(naphthalen-2-yl)prop-2-en-1-ol (6h)



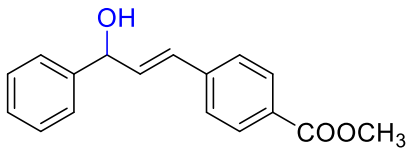
Compound **6i**: yellow solid (85% yield, 51.5 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.93 – 7.90 (m, 1H), 7.86 – 7.82 (m, 3H), 7.56 (dd, *J* = 8.5, 1.8 Hz, 1H), 7.52 – 7.46 (m, 2H), 7.44 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.25 – 7.19 (m, 1H), 7.09 (dd, *J* = 16.0, 1.2 Hz, 1H), 7.03 (dd, *J* = 8.2, 1.2 Hz, 1H), 6.98 (td, *J* = 7.5, 1.2 Hz, 1H), 6.40 (dd, *J* = 16.0, 6.9 Hz, 1H), 5.60 (d, *J* = 6.8 Hz, 1H), 2.73 (s, 6H), 2.32 (s, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 151.9, 140.6, 133.4, 133.0, 131.0, 130.6, 128.9, 128.4, 128.4, 128.1, 127.7, 127.3, 126.2, 125.9, 124.8, 124.6, 122.4, 118.1, 44.7. HRMS (ESI, m/z) calcd for $C_{21}H_{21}ON$ [M+H]+: 304.1696; found: 304.1700.

(*E*)-3-(4-nitrophenyl)-1-phenylprop-2-en-1-ol (6i)



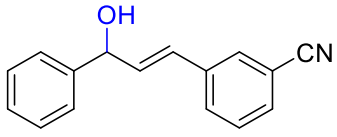
Compound **6h** (CAS: 17245-24-8): yellow solid (98% yield, 49.9 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 8.16 (d, *J* = 8.7 Hz, 2H), 7.50 (d, *J* = 8.7 Hz, 2H), 7.45 – 7.37 (m, 4H), 7.34 (d, *J* = 6.9 Hz, 1H), 6.78 (d, *J* = 15.9 Hz, 1H), 6.56 (dd, *J* = 15.9, 5.8 Hz, 1H), 5.46 – 5.41 (m, 1H), 2.25 (d, *J* = 3.3 Hz, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 147.0, 143.2, 142.1, 136.3, 128.9, 128.3, 127.8, 127.1, 126.4, 124.0, 74.7.

methyl (E)-4-(3-hydroxy-3-phenylprop-1-en-1-yl)benzoate (6j)



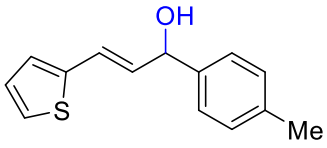
Compound **6v** (CAS: 2490144-85-7): yellow oil (88% yield, 47.1 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.96 (dd, J = 8.4, 1.9 Hz, 2H), 7.48 – 7.40 (m, 4H), 7.40 – 7.24 (m, 4H), 6.72 (d, J = 15.8 Hz, 1H), 6.49 (dd, J = 15.9, 6.2 Hz, 1H), 5.40 (d, J = 5.4 Hz, 1H), 3.90 (s, 3H), 2.37 (s, 1H). ^{13}C NMR (126 MHz, Chloroform-d) δ 166.9, 142.5, 141.1, 134.2, 129.9, 129.8, 129.3, 128.7, 128.0, 126.5, 126.4, 74.9, 52.1.

(E)-3-(3-hydroxy-3-phenylprop-1-en-1-yl)benzonitrile (6k)



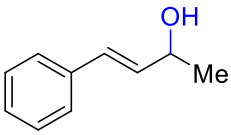
Compound **6u** (CAS: 2451060-11-8): yellow oil (80% yield, 37.6 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.64 – 7.57 (m, 2H), 7.50 (dt, J = 7.7, 1.5 Hz, 1H), 7.43 – 7.33 (m, 6H), 6.68 (dd, J = 15.8, 1.4 Hz, 1H), 6.44 (dd, J = 15.9, 6.0 Hz, 1H), 5.40 (d, J = 5.9 Hz, 1H), 2.30 (s, 1H). ^{13}C NMR (126 MHz, Chloroform-d) δ 142.3, 137.9, 134.4, 130.9, 130.7, 130.1, 129.4, 128.8, 128.2, 127.8, 126.4, 118.7, 112.8, 74.7.

(*E*)-3-(thiophen-2-yl)-1-(p-tolyl)prop-2-en-1-ol (6l)



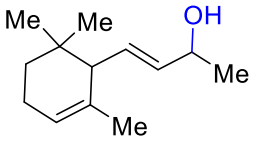
Compound **6j** (CAS: 30461-43-9): colorless oil (86% yield, 39.6 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.32 (d, *J* = 8.1 Hz, 2H), 7.21 (d, *J* = 7.9 Hz, 2H), 7.17 (d, *J* = 4.6 Hz, 1H), 6.98 (d, *J* = 4.7 Hz, 2H), 6.80 (d, *J* = 15.7 Hz, 1H), 6.24 (dd, *J* = 15.7, 6.3 Hz, 1H), 5.30 (d, *J* = 6.3 Hz, 1H), 2.42 (s, 1H), 2.39 (s, 3H). ^{13}C NMR (125 MHz, Chloroform-d) δ 141.9, 139.7, 137.6, 131.4, 129.4, 127.4, 127.4, 126.4, 126.1, 124.5, 123.5, 74.6, 71.1, 21.2.

(*E*)-4-phenylbut-3-en-2-ol (6m)



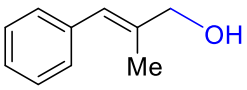
Compound **6k** (CAS: 17488-65-2): colorless oil (95% yield, 28.1 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.41 – 7.36 (m, 2H), 7.35 – 7.29 (m, 2H), 7.27 – 7.23 (m, 1H), 6.57 (d, *J* = 15.3 Hz, 1H), 6.27 (dd, *J* = 15.9, 6.3 Hz, 1H), 4.49 (p, *J* = 6.5 Hz, 1H), 2.03 (s, 1H), 1.38 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR (125 MHz, Chloroform-d) δ 136.8, 133.7, 129.4, 128.6, 127.6, 126.5, 68.9, 23.4.

(*E*)-4-(2,6,6-trimethylcyclohex-2-en-1-yl)but-3-en-2-ol (6n)



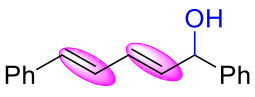
Compound **6m** (CAS: 25312-34-9): colorless oil (87% yield, 33.7 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 5.49 (ddd, *J* = 15.3, 6.4, 1.9 Hz, 1H), 5.43 – 5.36 (m, 2H), 4.27 (p, *J* = 6.3 Hz, 1H), 2.06 (d, *J* = 9.2 Hz, 1H), 1.97 (d, *J* = 1.8 Hz, 2H), 1.71 (s, 1H), 1.56 (dq, *J* = 9.7, 1.9 Hz, 3H), 1.43 – 1.37 (m, 1H), 1.24 (dd, *J* = 6.4, 1.8 Hz, 3H), 1.17 – 1.12 (m, 1H), 0.87 (d, *J* = 2.9 Hz, 3H), 0.79 (d, *J* = 11.2 Hz, 3H). ^{13}C NMR (125 MHz, Chloroform-d) δ 136.0, 135.9, 134.0, 133.9, 131.2, 131.1, 121.0, 120.9, 68.9, 68.8, 53.9, 53.9, 31.9, 31.9, 31.6, 31.5, 27.5, 27.5, 26.9, 23.6, 23.6, 23.0, 22.8, 22.8.

(*E*)-2-methyl-3-phenylprop-2-en-1-ol (6o)



Compound **6l** (CAS: 1504-55-8): colorless oil (95% yield, 28.1 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.35 (t, *J* = 7.6 Hz, 2H), 7.29 (d, *J* = 7.0 Hz, 2H), 7.23 (t, *J* = 7.1 Hz, 1H), 6.53 (s, 1H), 4.20 (s, 2H), 1.91 (s, 3H), 1.82 (s, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 137.7, 137.6, 128.9, 128.2, 126.5, 125.0, 69.0, 15.3.

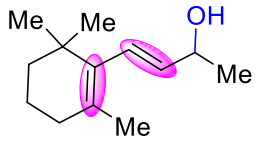
(2*E*,4*E*)-1,5-diphenylpenta-2,4-dien-1-ol (6p)



Compound **6n** (CAS: 58926-22-0): colorless oil (85% yield, 40.1 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.45 – 7.37 (m, 6H), 7.33 (td, *J* = 7.2, 6.8, 5.3 Hz, 3H), 7.27 – 7.22 (m, 1H), 6.80 (dd, *J* = 15.6, 10.5 Hz, 1H), 6.65 – 6.43 (m, 2H), 6.01 (dd, *J* = 15.1, 6.6 Hz, 1H), 5.32 (d, *J*

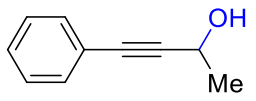
= 6.6 Hz, 1H), 2.25 (s, 1H). ^{13}C NMR (125 MHz, Chloroform-d) δ 142.9, 137.1, 135.6, 133.3, 131.0, 128.7, 128.1, 127.8, 127.7, 126.4, 126.4, 74.9.

(*E*)-4-(2,6,6-trimethylcyclohex-1-en-1-yl)but-3-en-2-ol (6q)



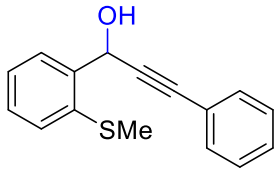
Compound **6o** (CAS: 472-80-0): colorless oil (93% yield, 36.1 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 6.04 (d, *J* = 15.9 Hz, 1H), 5.48 (dd, *J* = 16.0, 6.7 Hz, 1H), 4.36 (pd, *J* = 6.4, 1.2 Hz, 1H), 1.99 – 1.95 (m, 2H), 1.91 (s, 1H), 1.66 (d, *J* = 1.0 Hz, 3H), 1.62 – 1.57 (m, 2H), 1.46 – 1.42 (m, 2H), 1.31 (d, *J* = 6.4 Hz, 3H), 0.99 (d, *J* = 2.0 Hz, 6H). ^{13}C NMR (125 MHz, Chloroform-d) δ 137.7, 136.7, 128.7, 127.4, 69.4, 39.4, 33.9, 32.7, 28.7, 28.7, 23.6, 21.3, 19.2.

4-phenylbut-3-yn-2-ol (6r)



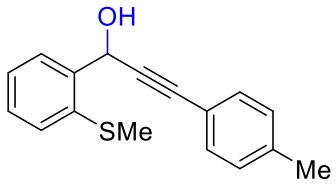
Compound **6p** (CAS: 5876-76-6): colorless oil (88% yield, 25.7 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.46 – 7.40 (m, 2H), 7.30 (dd, *J* = 5.2, 2.0 Hz, 3H), 4.76 (dt, *J* = 10.3, 5.2 Hz, 1H), 2.24 (s, 1H), 1.56 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (125 MHz, Chloroform-d) δ 131.7, 128.4, 128.3, 122.6, 91.0, 84.0, 58.8, 24.4.

1-(2-(methylthio)phenyl)-3-phenylprop-2-yn-1-ol (6s)



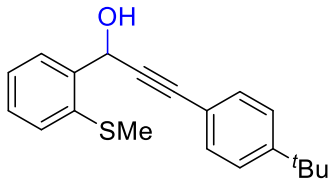
Compound **6q** (CAS: 2033072-02-3): colorless oil (90% yield, 45.7 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.76 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.50 – 7.47 (m, 2H), 7.39 – 7.36 (m, 1H), 7.32 (td, *J* = 5.3, 2.3 Hz, 4H), 7.26 (td, *J* = 7.5, 1.5 Hz, 1H), 6.07 (d, *J* = 5.1 Hz, 1H), 3.06 (s, 1H), 2.52 (s, 3H). ^{13}C NMR (125 MHz, Chloroform-d) δ 139.5, 136.6, 131.8, 129.1, 128.6, 128.4, 128.3, 127.4, 126.2, 122.6, 88.5, 86.9, 63.1, 17.3.

1-(2-(methylthio)phenyl)-3-(p-tolyl)prop-2-yn-1-ol (6t)



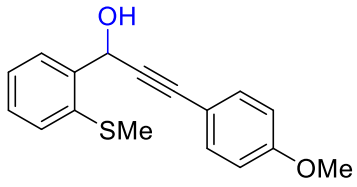
Compound **6r** (CAS: 2703050-03-5): colorless oil (52.5% yield, 131 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.76 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.39 – 7.36 (m, 3H), 7.33 (td, *J* = 7.5, 1.6 Hz, 1H), 7.27 – 7.24 (m, 1H), 7.12 (d, *J* = 7.9 Hz, 2H), 6.06 (s, 1H), 3.04 (s, 1H), 2.52 (s, 3H), 2.35 (s, 3H). ^{13}C NMR (125 MHz, Chloroform-d) δ 139.6, 138.7, 136.6, 131.7, 129.1, 129.0, 128.4, 127.4, 126.2, 119.5, 87.8, 87.0, 63.1, 21.5, 17.3.

3-(4-(tert-butyl)phenyl)-1-(2-(methylthio)phenyl)prop-2-yn-1-ol (6u)



Compound **6s** (CAS: 2703050-04-6): colorless oil (92% yield, 49.3 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.77 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.45 – 7.41 (m, 2H), 7.39 – 7.32 (m, 4H), 7.27 – 7.23 (m, 1H), 6.07 (s, 1H), 3.05 (s, 1H), 2.52 (d, *J* = 1.2 Hz, 3H), 1.32 (s, 9H). ^{13}C NMR (125 MHz, Chloroform-d) δ 151.8, 139.6, 136.6, 131.5, 129.0, 128.4, 127.5, 126.2, 125.3, 119.5, 87.8, 87.0, 63.1, 34.8, 31.2, 17.3.

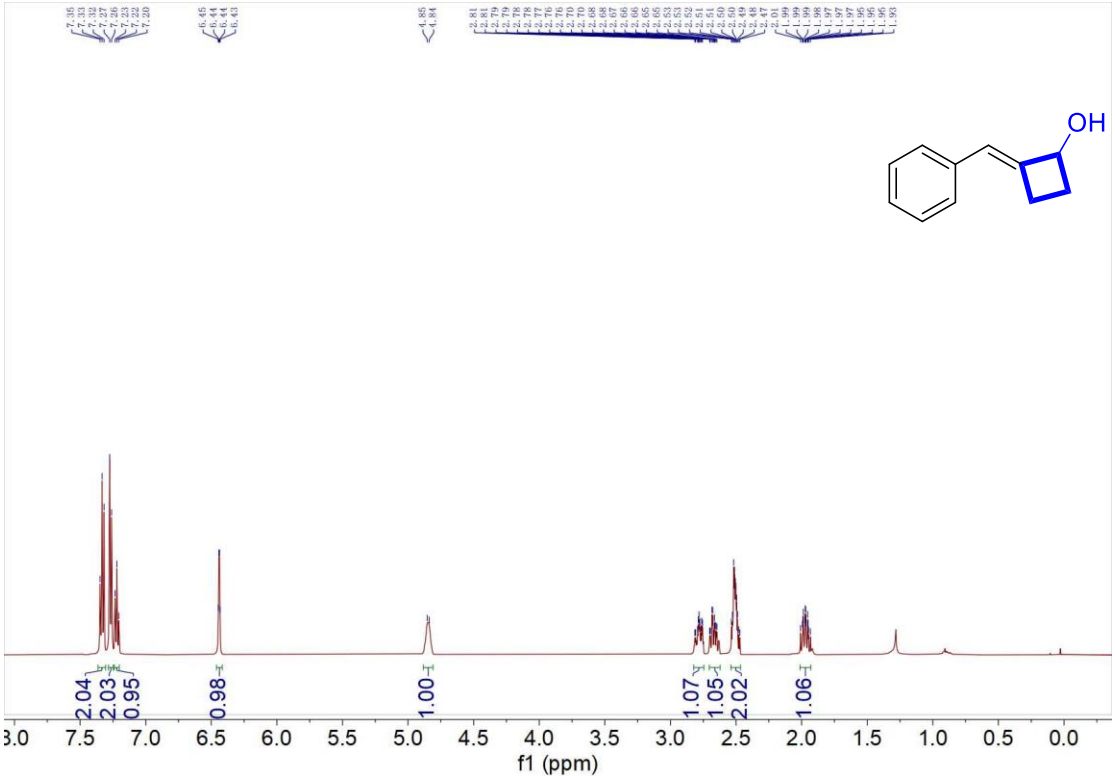
3-(4-methoxyphenyl)-1-(2-(methylthio)phenyl)prop-2-yn-1-ol (6v)

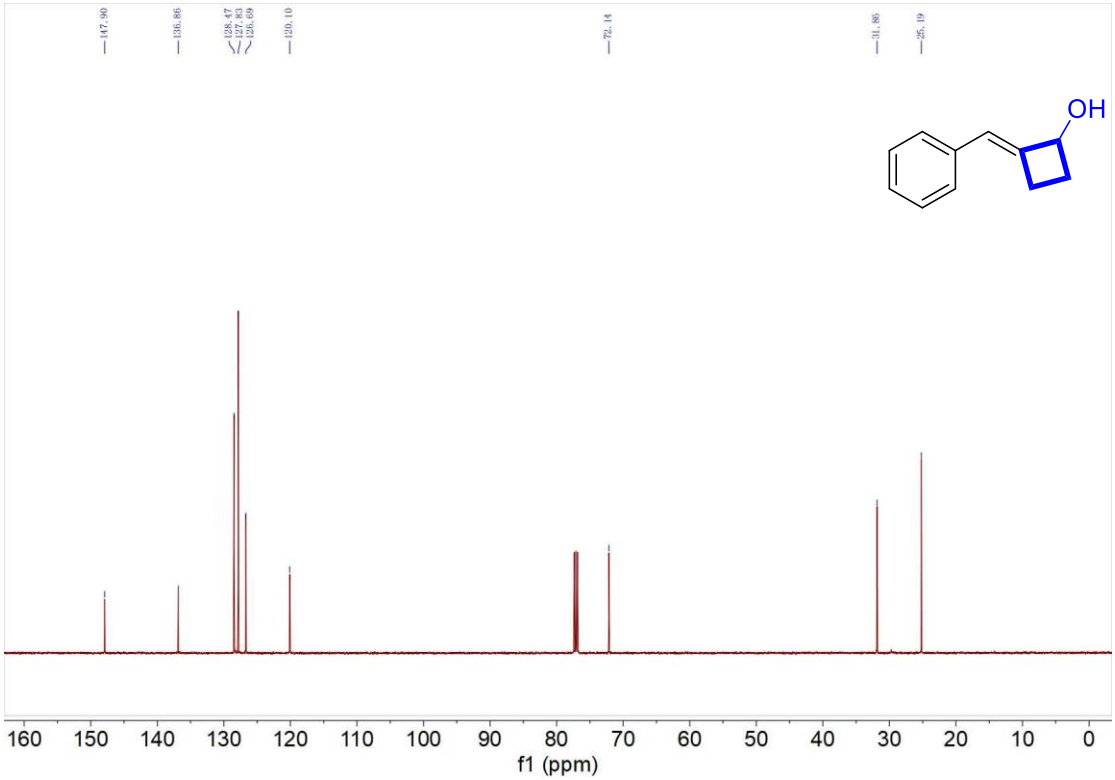


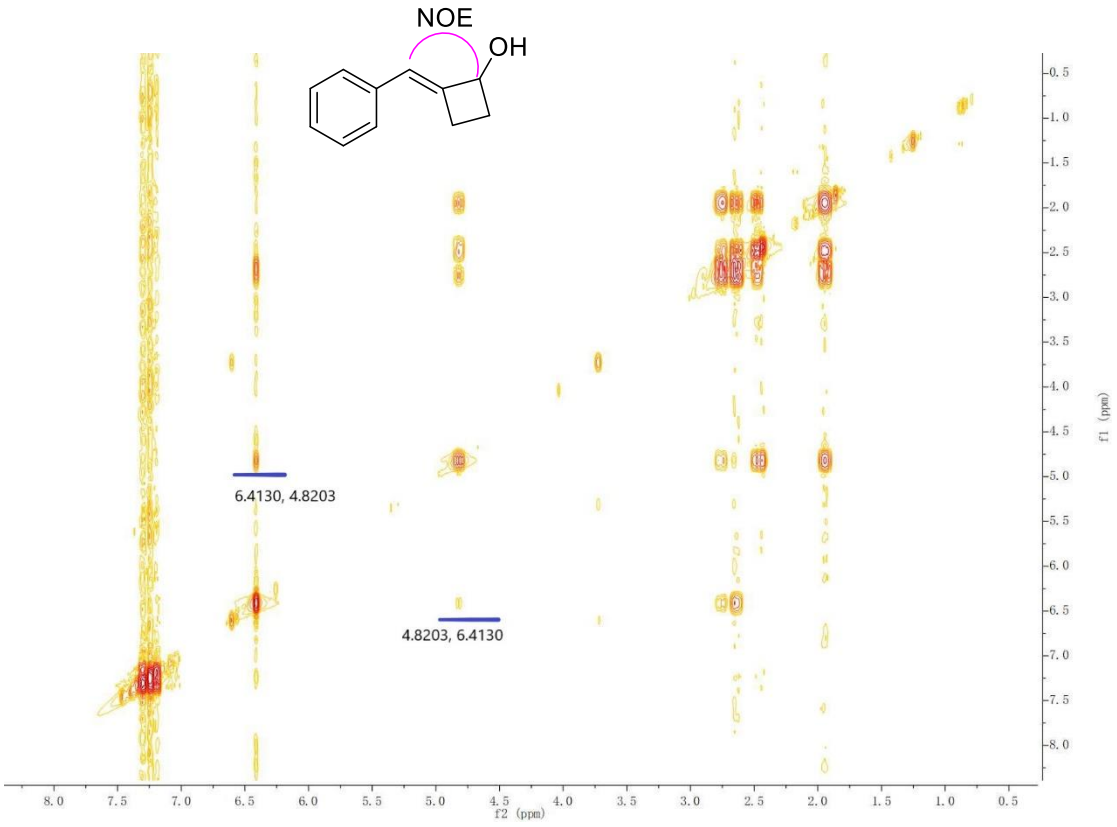
Compound **6t** (CAS: 2033072-03-4): colorless oil (85% yield, 48.3 mg); ^{1}H NMR (500 MHz, Chloroform-d) δ 7.76 (dd, *J* = 7.7, 1.5 Hz, 1H), 7.43 – 7.39 (m, 2H), 7.34 (ddd, *J* = 17.0, 7.5, 1.5 Hz, 2H), 7.26 – 7.22 (m, 1H), 6.83 (d, *J* = 8.8 Hz, 2H), 6.05 (s, 1H), 3.79 (s, 3H), 3.11 (s, 1H), 2.51 (s, 3H). ^{13}C NMR (125 MHz, Chloroform-d) δ 159.8, 139.7, 136.6, 133.3, 129.0, 128.3, 127.4, 126.1, 114.7, 113.9, 87.1, 86.8, 63.1, 55.3, 17.3.

5. NMR spectroscopic data

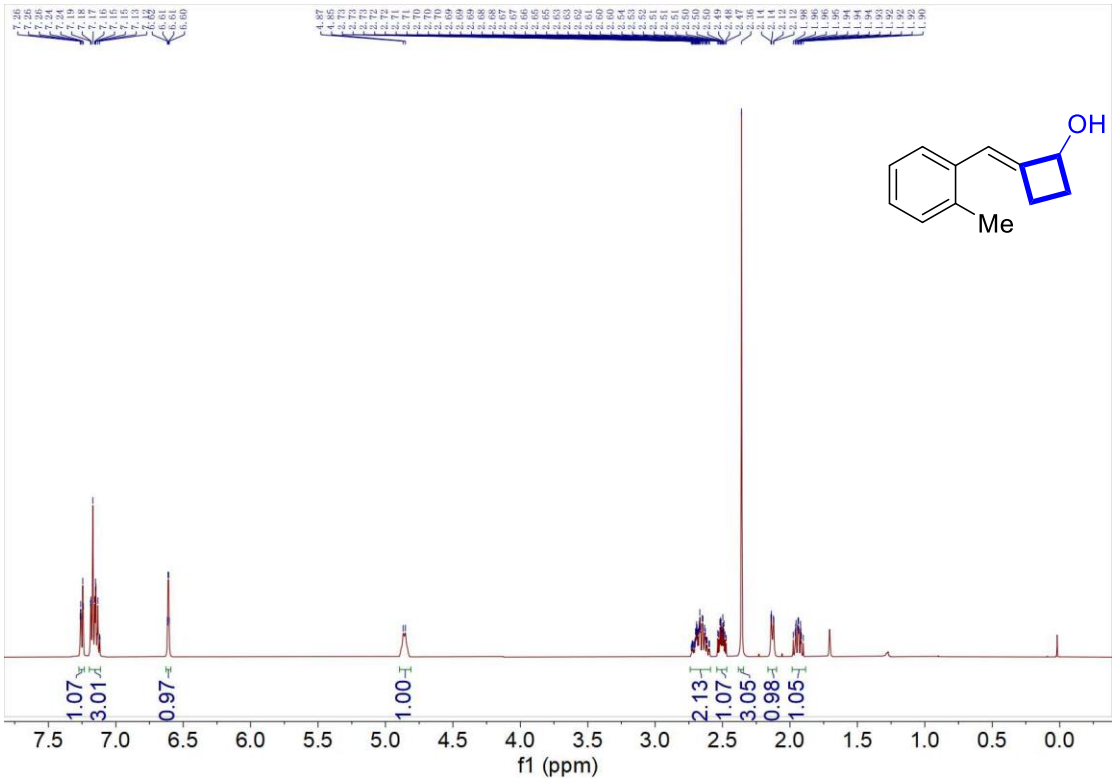
(*E*)-2-benzylidenecyclobutan-1-ol (2a)

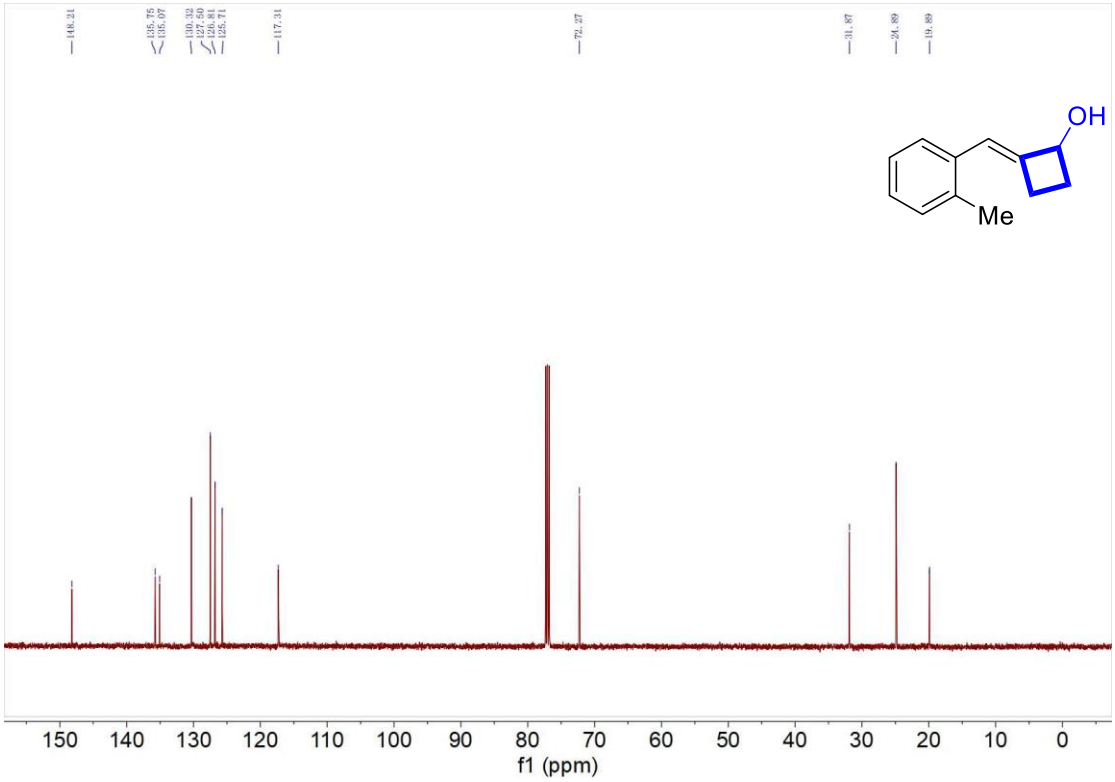




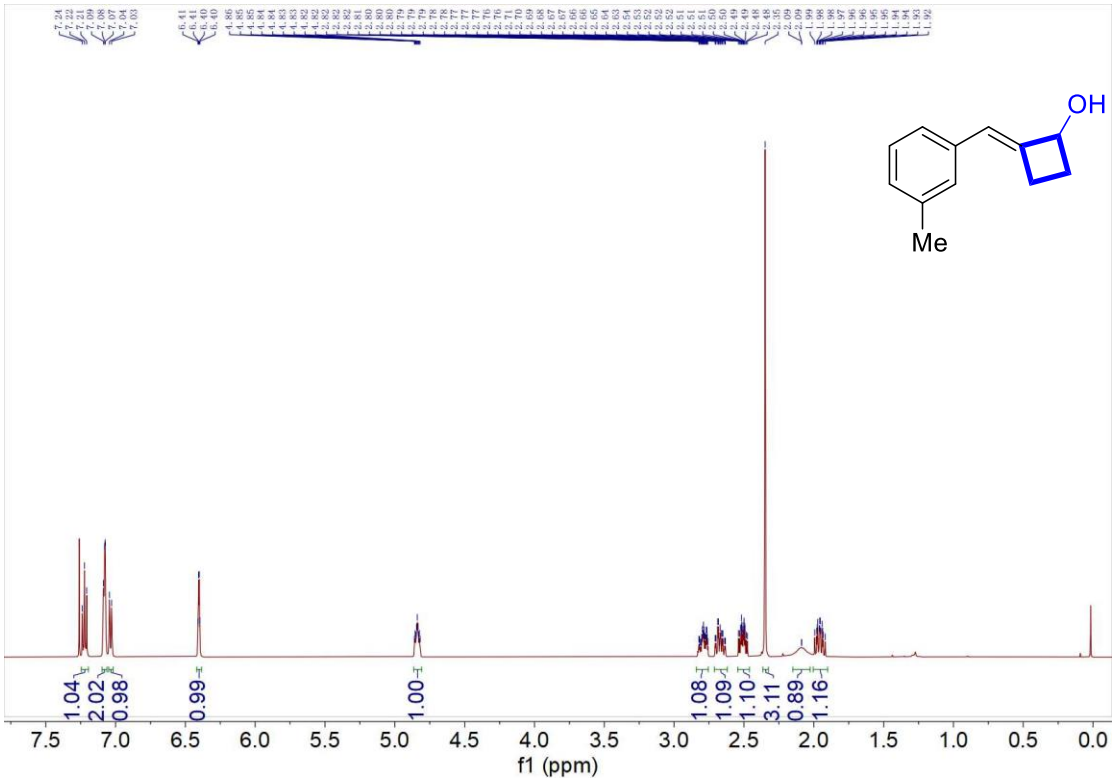

NOE
OH
6.4130, 4.8203
4.8203, 6.4130
f2 (ppm)
f1 (ppm)

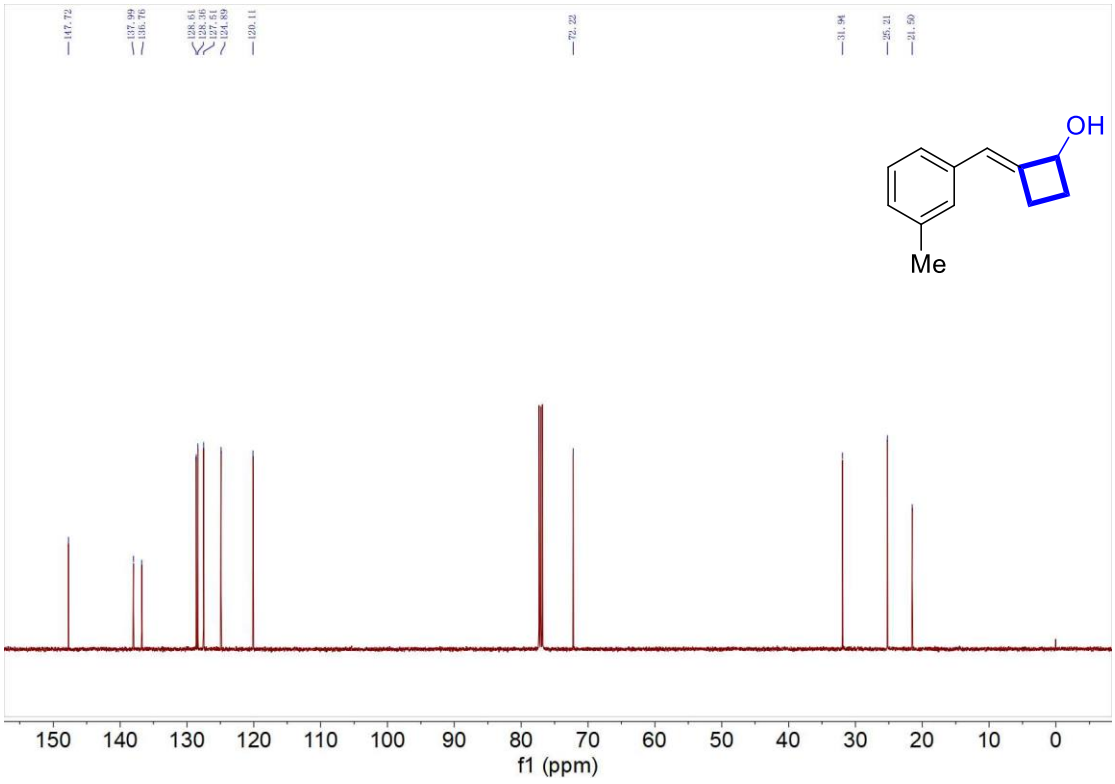
(*E*)-2-(2-methylbenzylidene)cyclobutan-1-ol (2b)



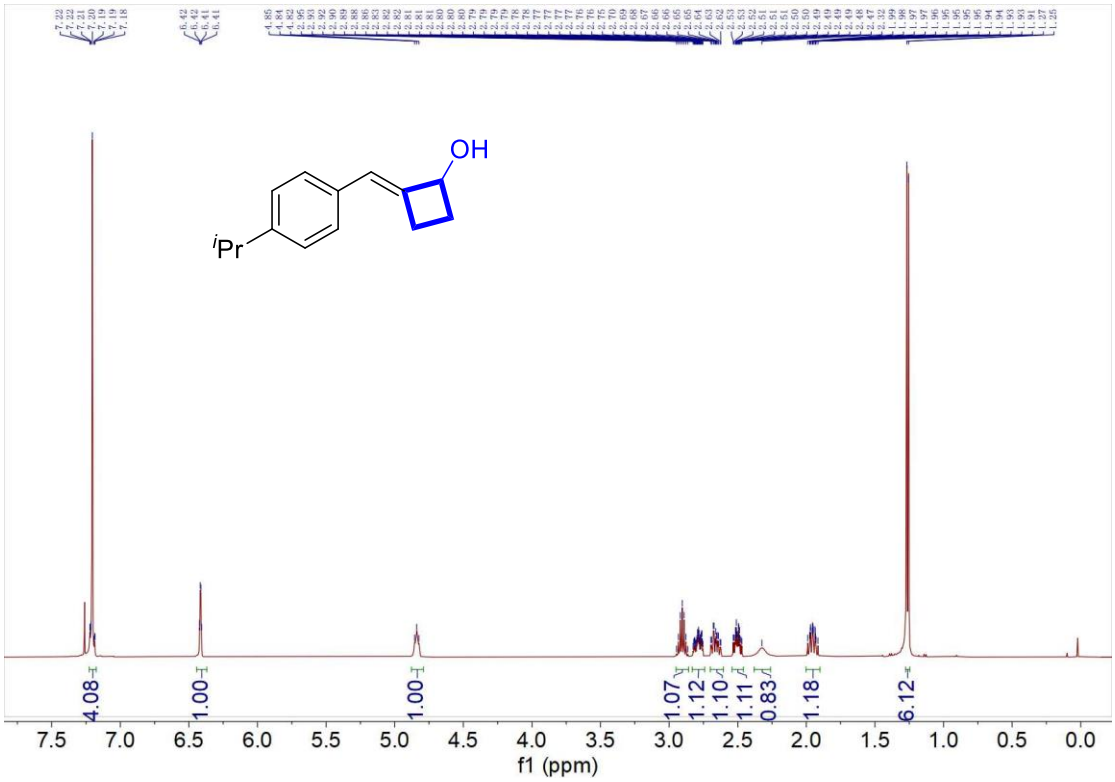


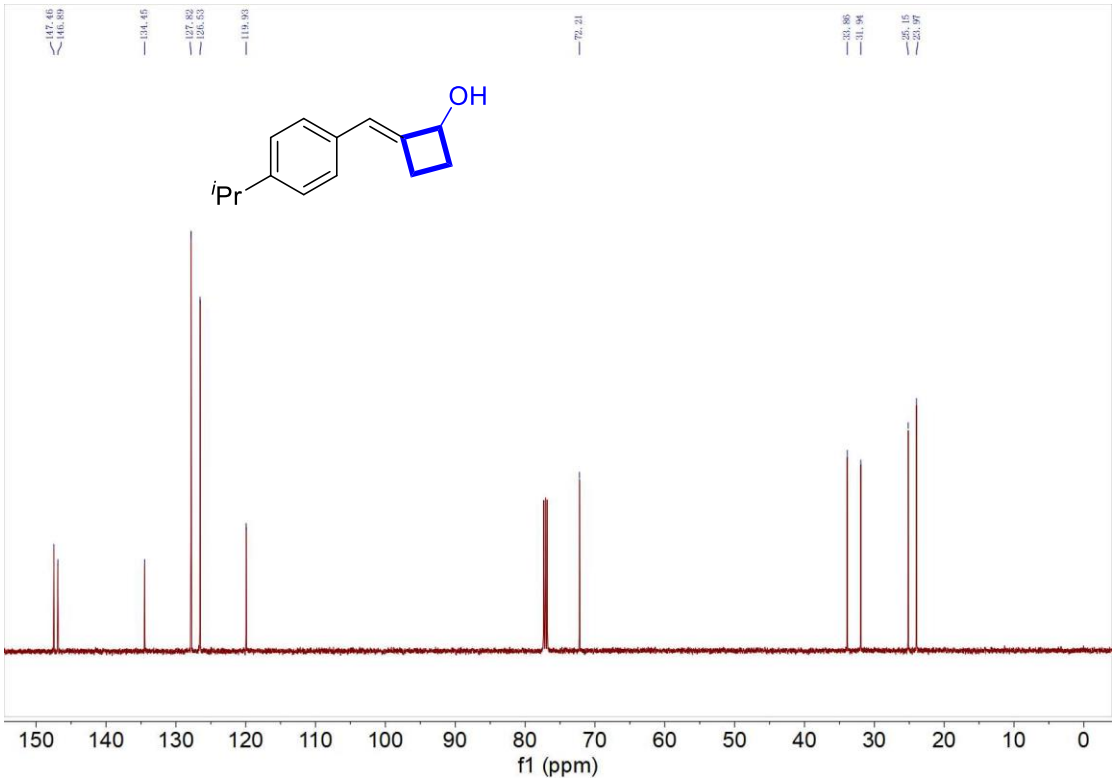
(*E*)-2-(3-methylbenzylidene)cyclobutan-1-ol (2c)



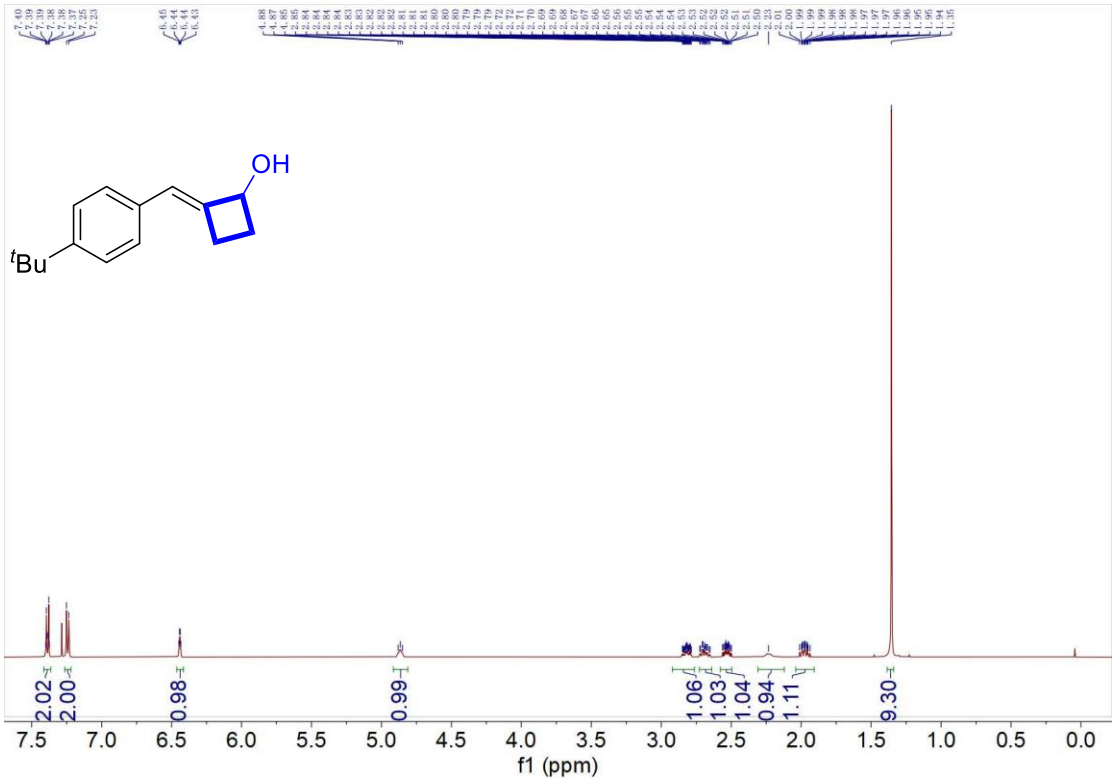


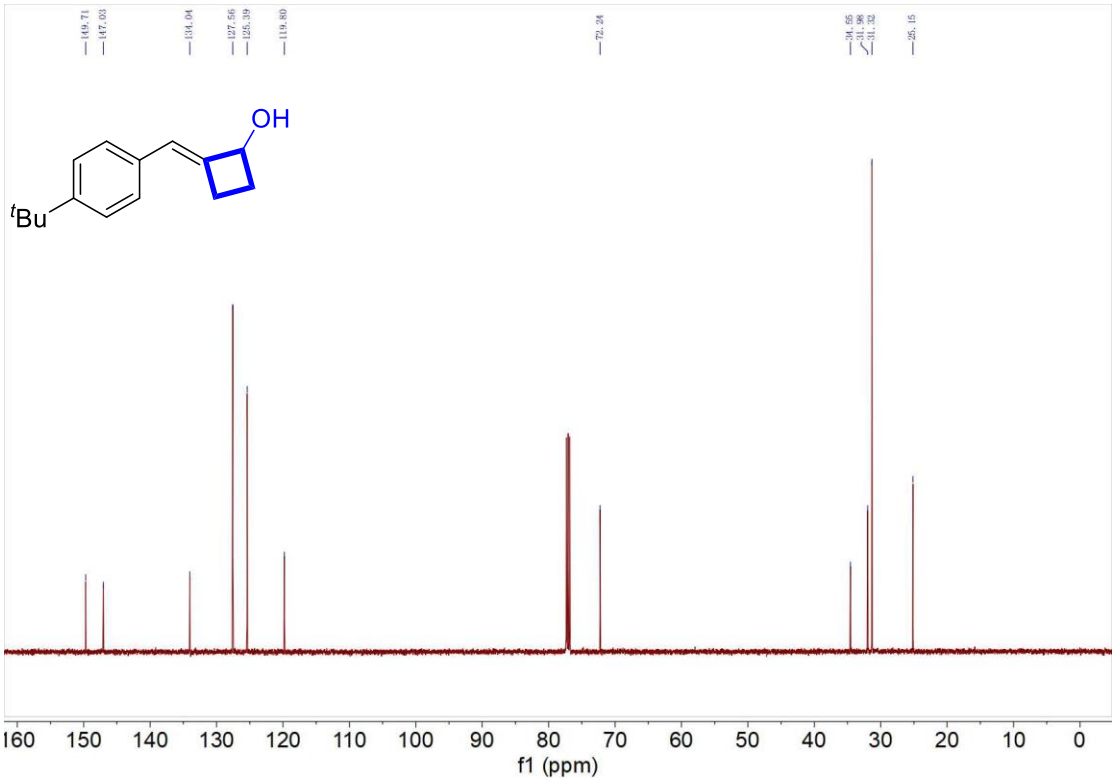
(*E*)-2-(4-isopropylbenzylidene)cyclobutan-1-ol (2d)



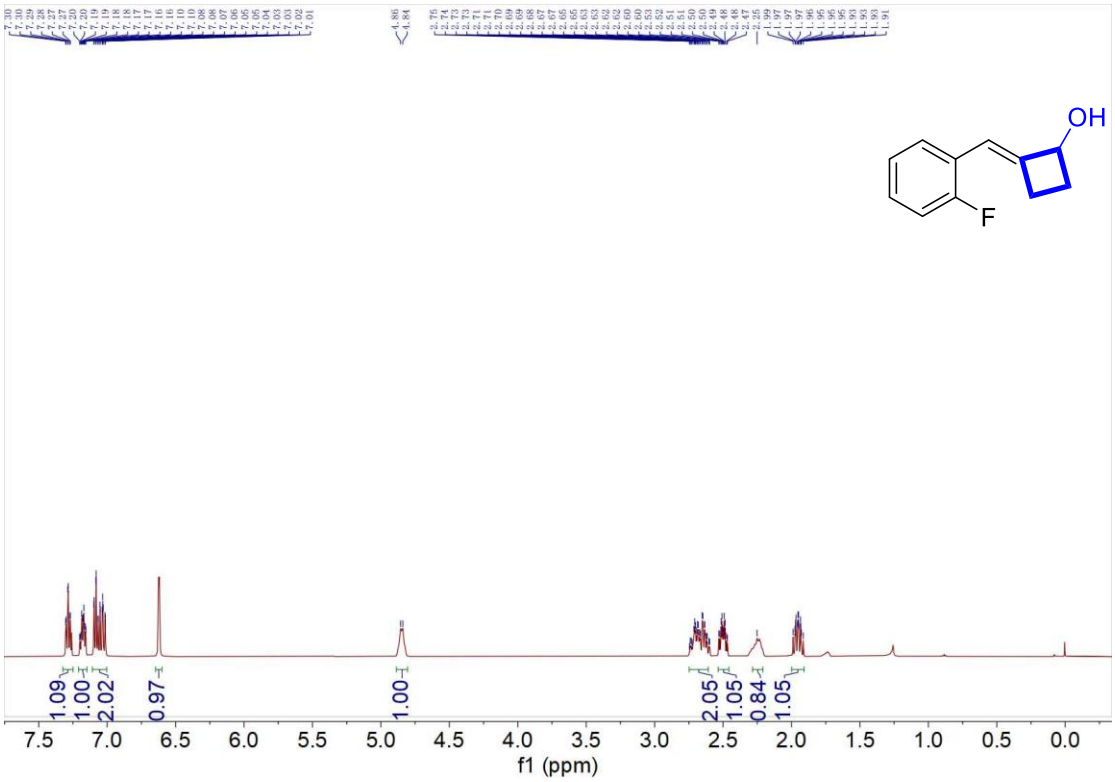


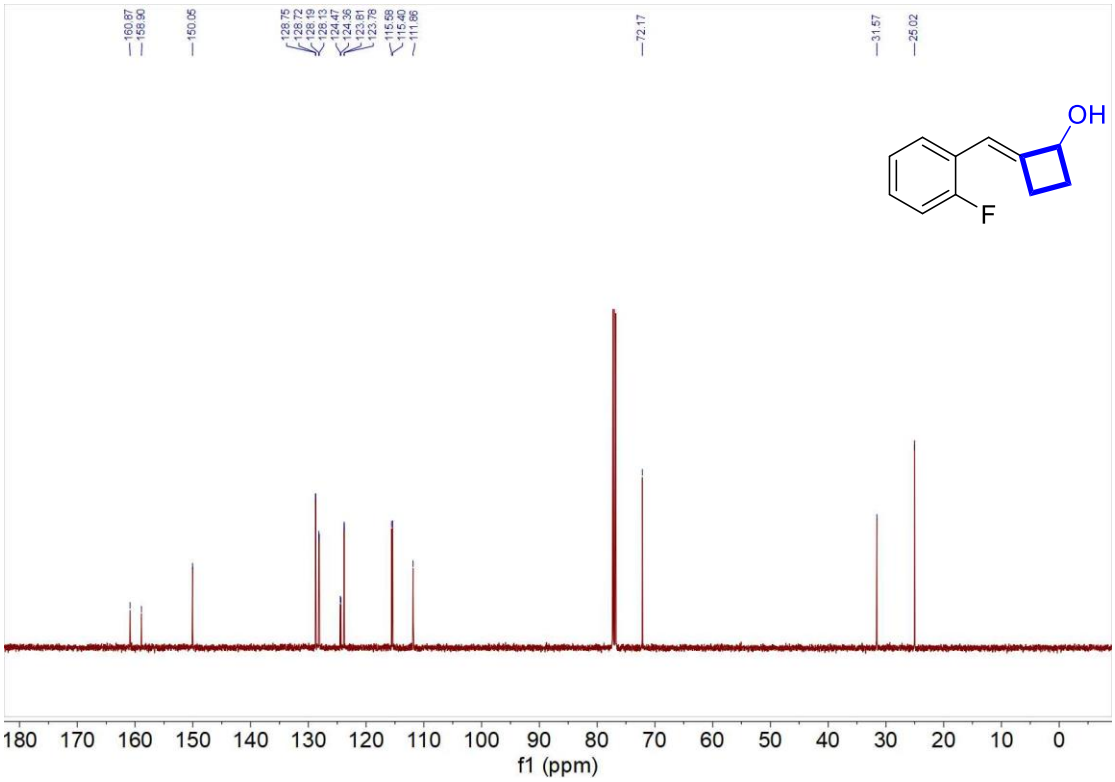
(*E*)-2-(4-(tert-butyl)benzylidene)cyclobutan-1-ol (2e)



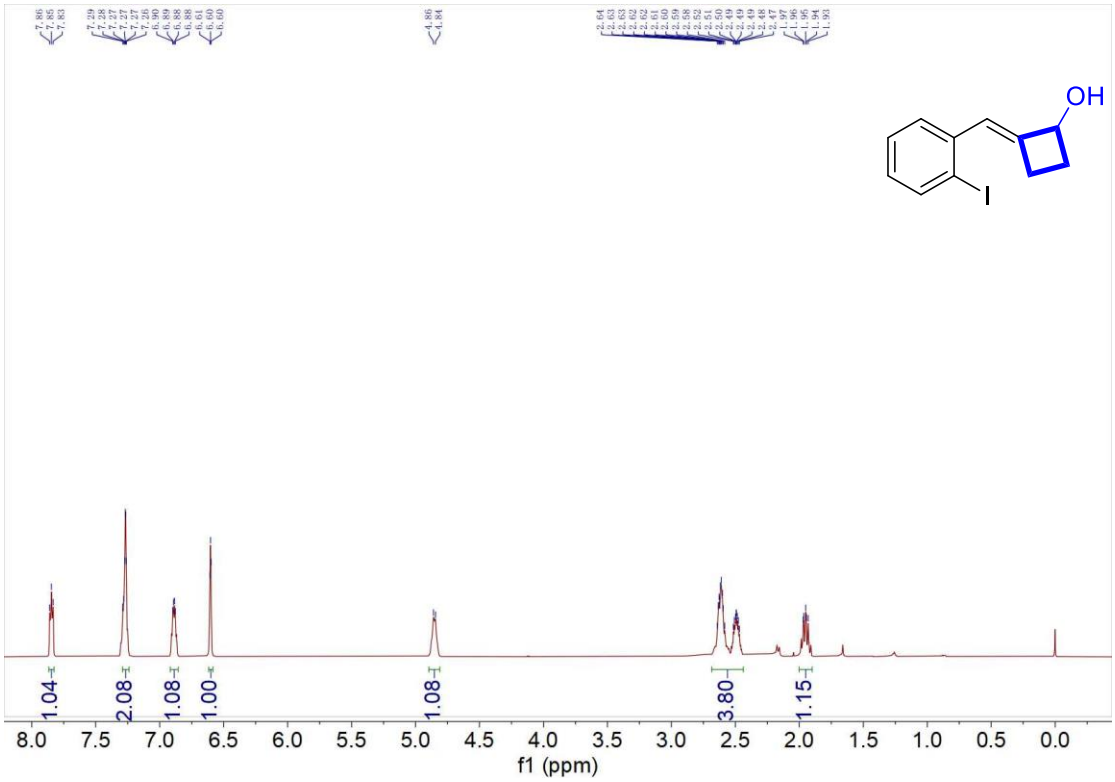


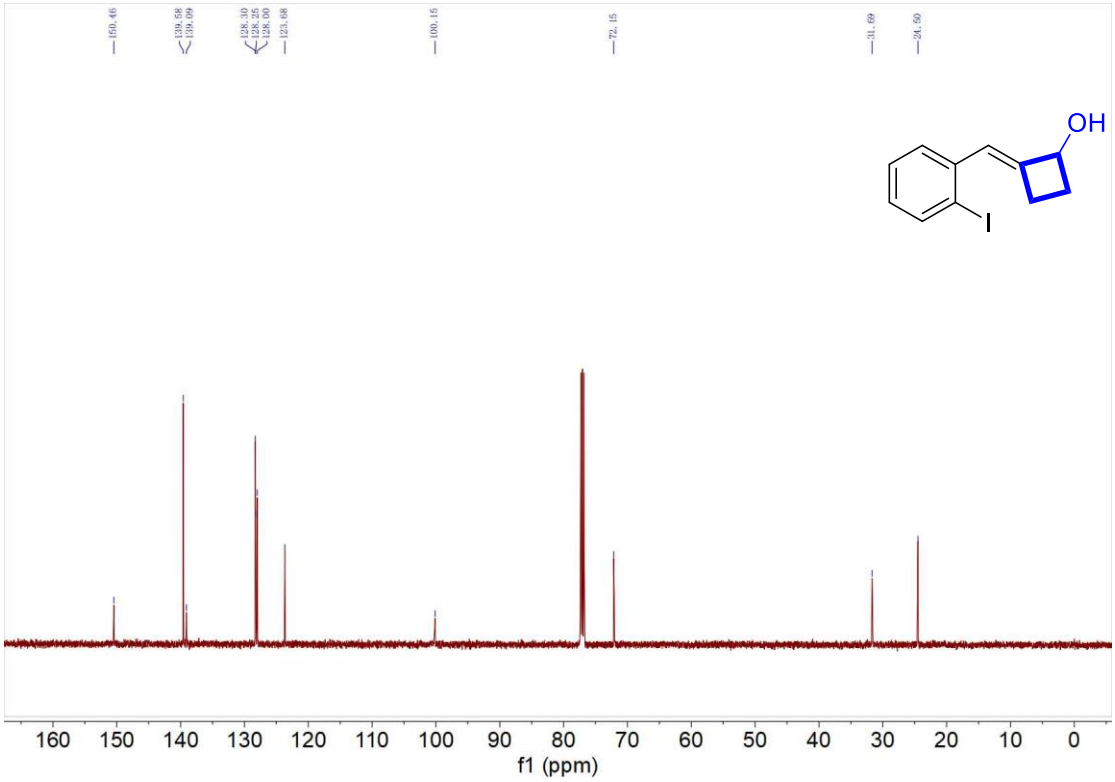
(*E*)-2-(2-fluorobenzylidene)cyclobutan-1-ol (2f)



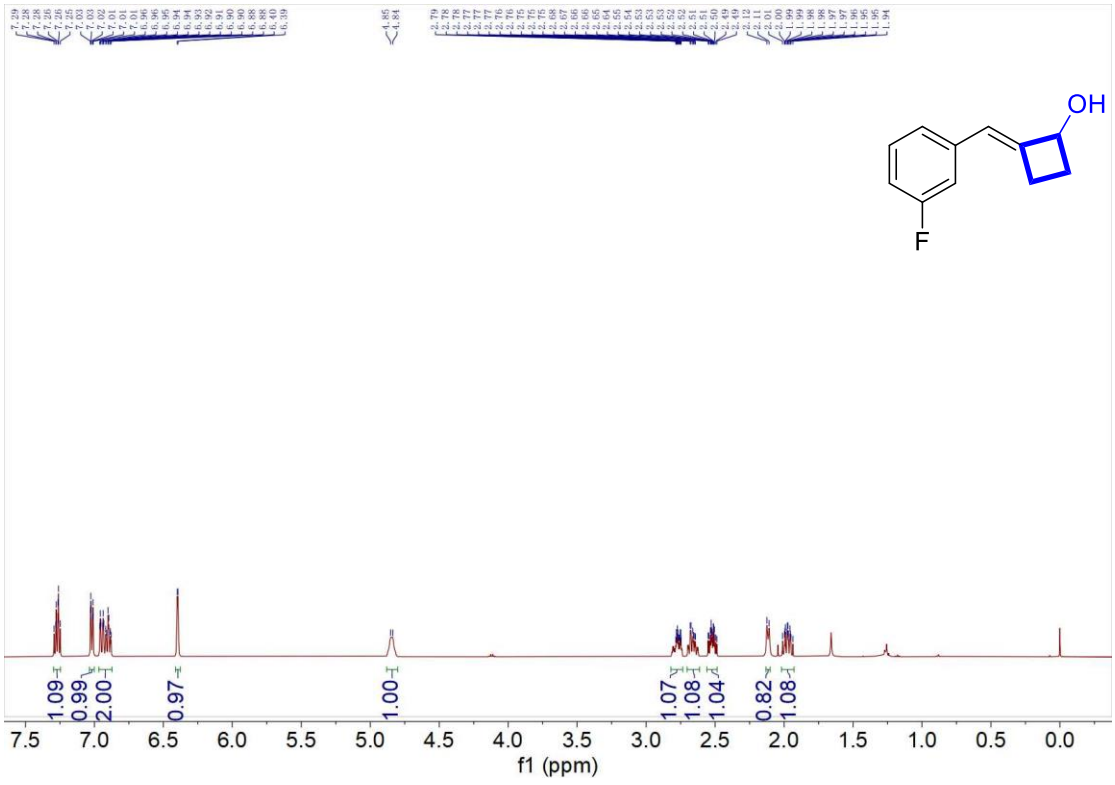


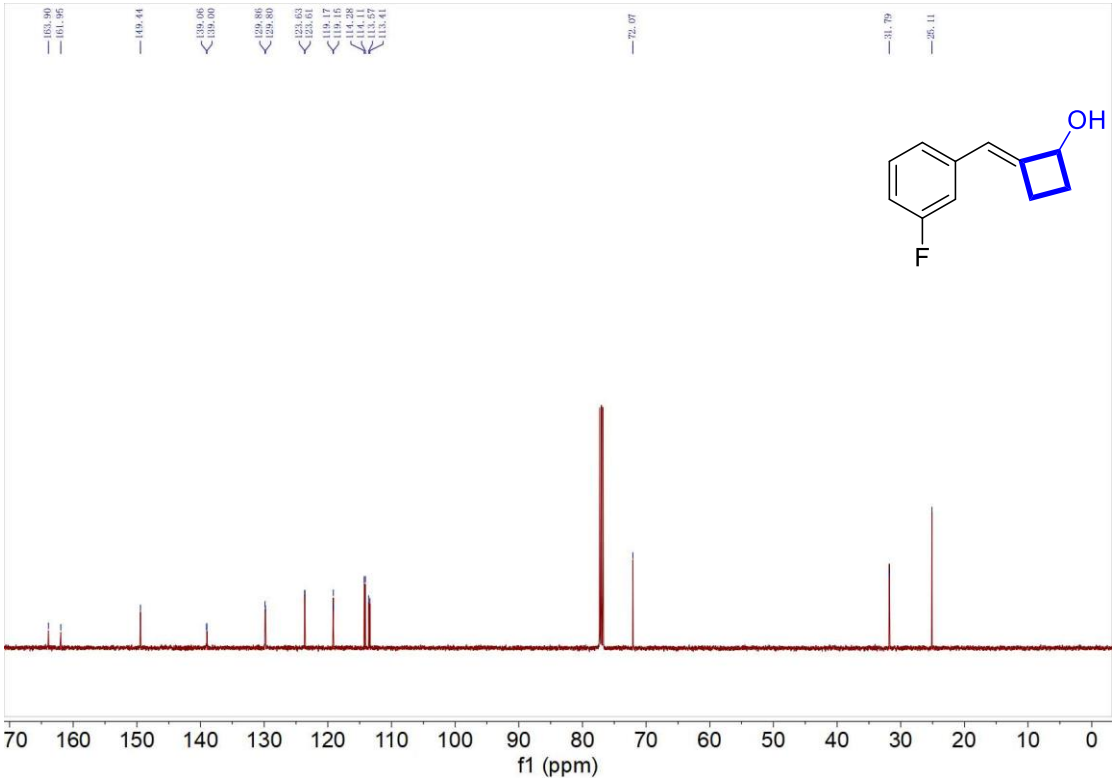
(*E*)-2-(2-iodobenzylidene)cyclobutan-1-ol (2g)



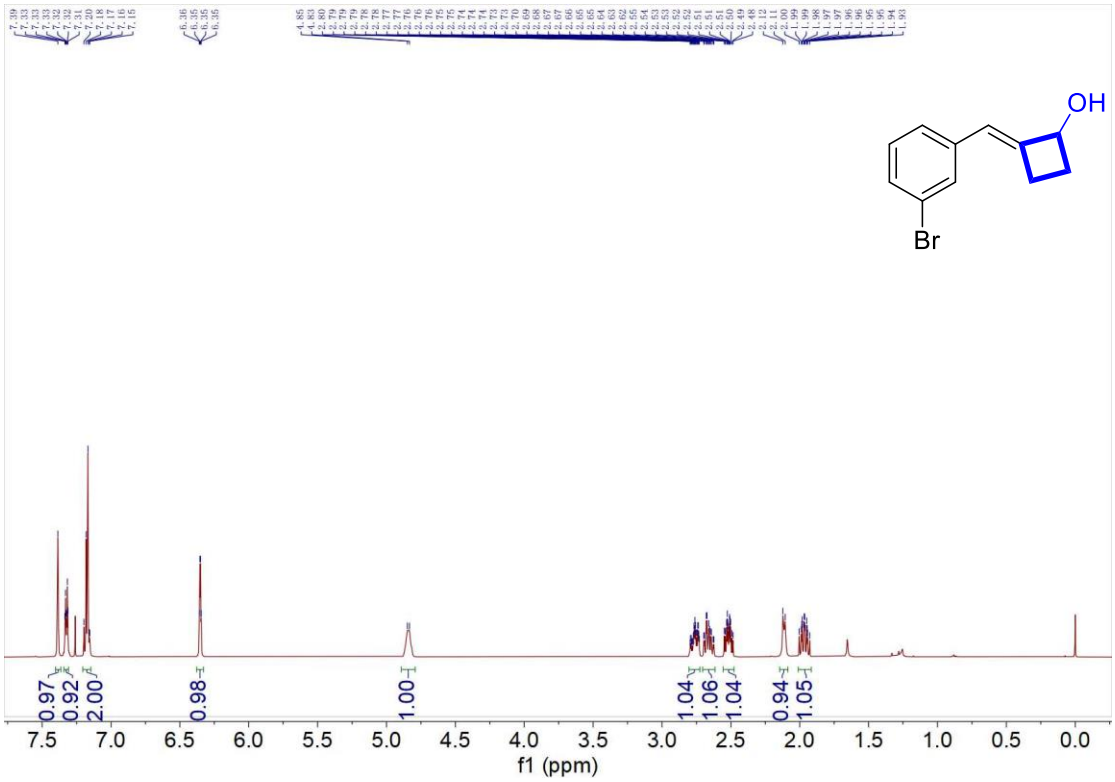


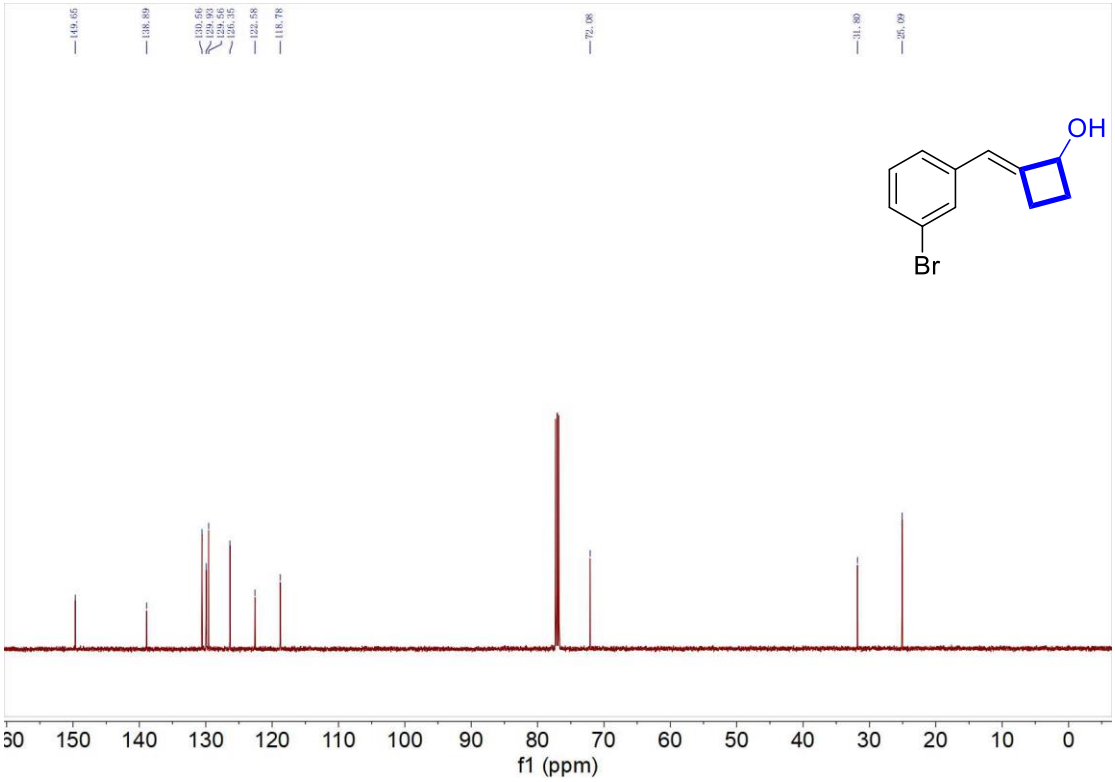
(*E*)-2-(3-fluorobenzylidene)cyclobutan-1-ol (2h)



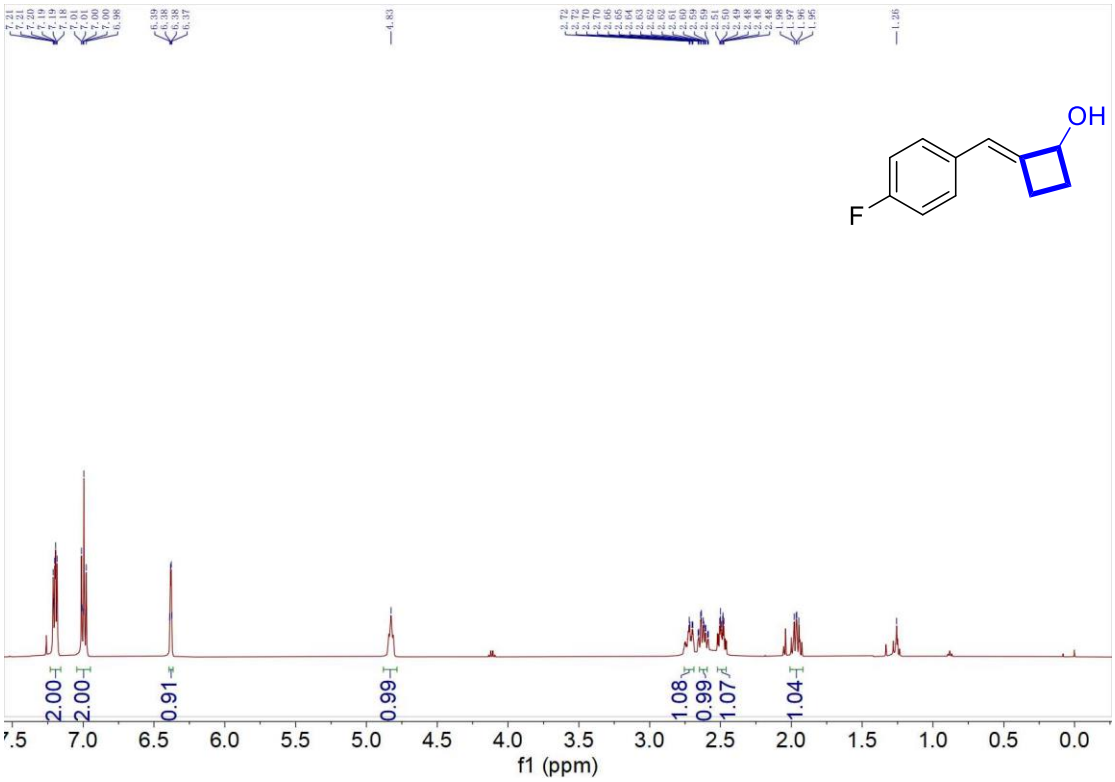


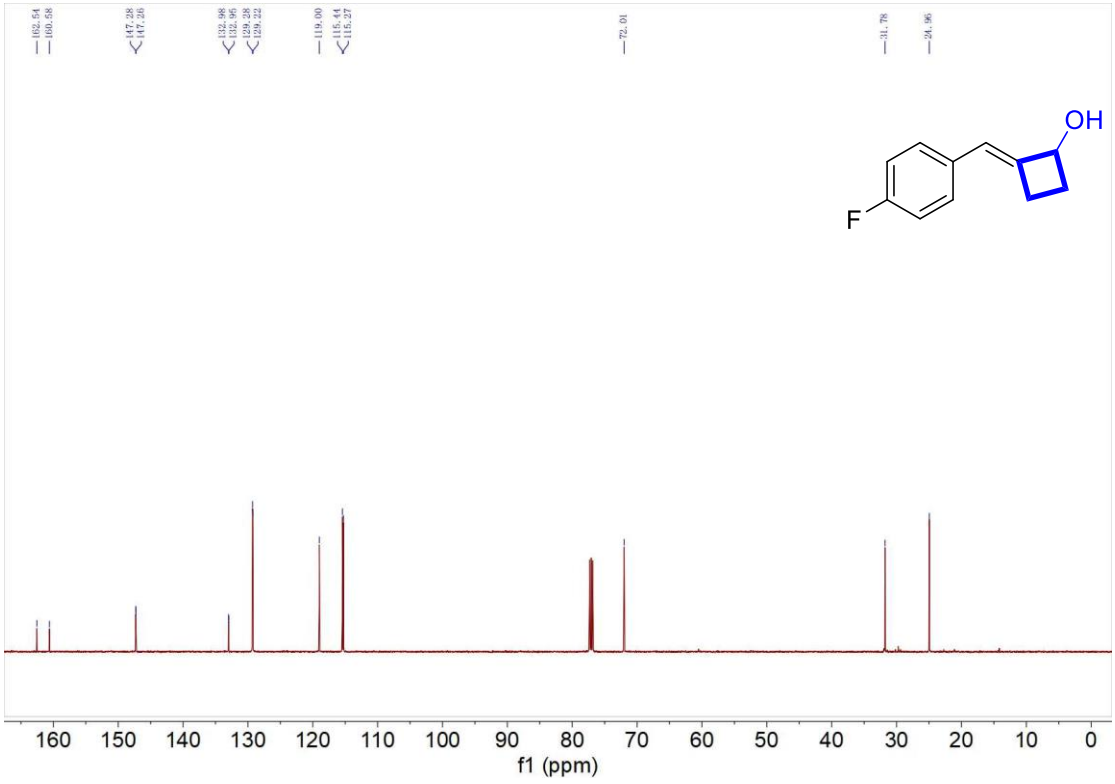
(*E*)-2-(3-bromobenzylidene)cyclobutan-1-ol (2i)



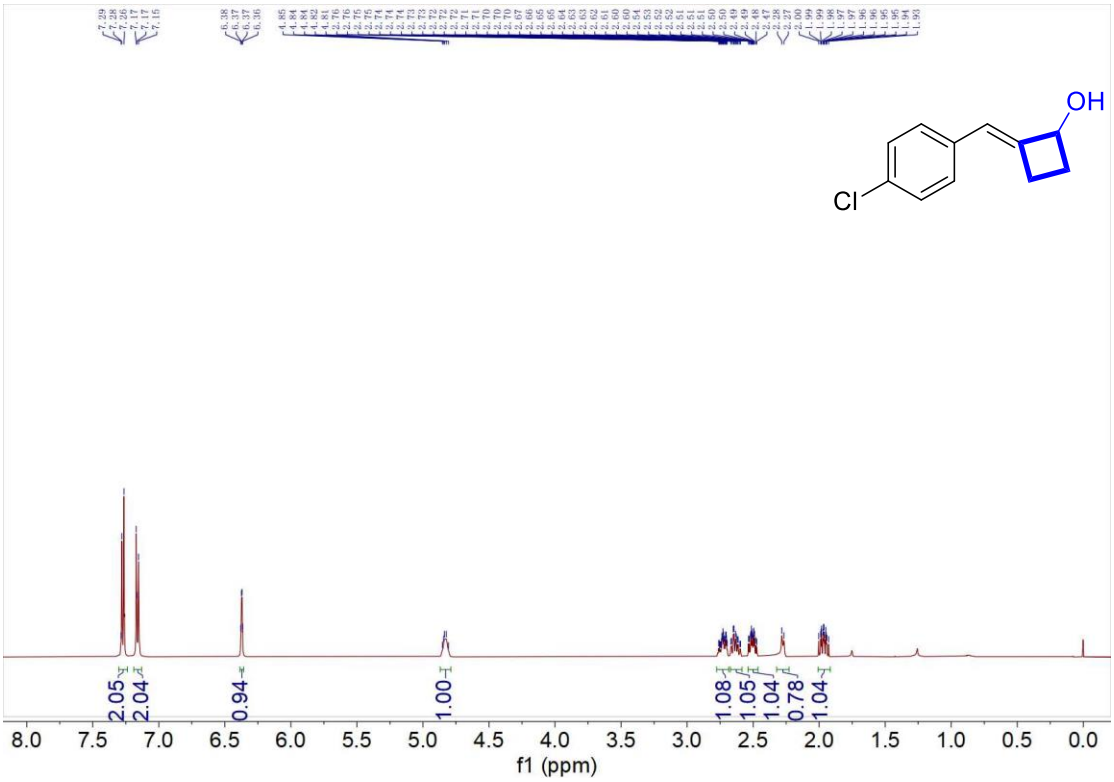


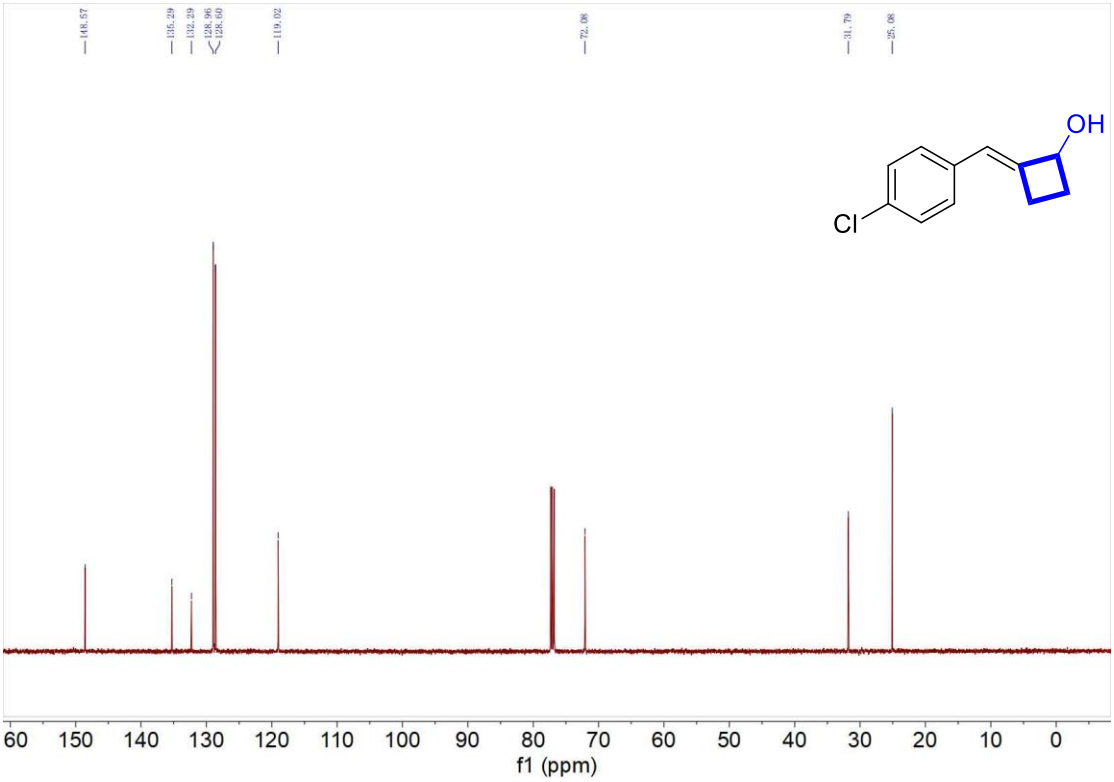
(*E*)-2-(4-fluorobenzylidene)cyclobutan-1-ol (2j)



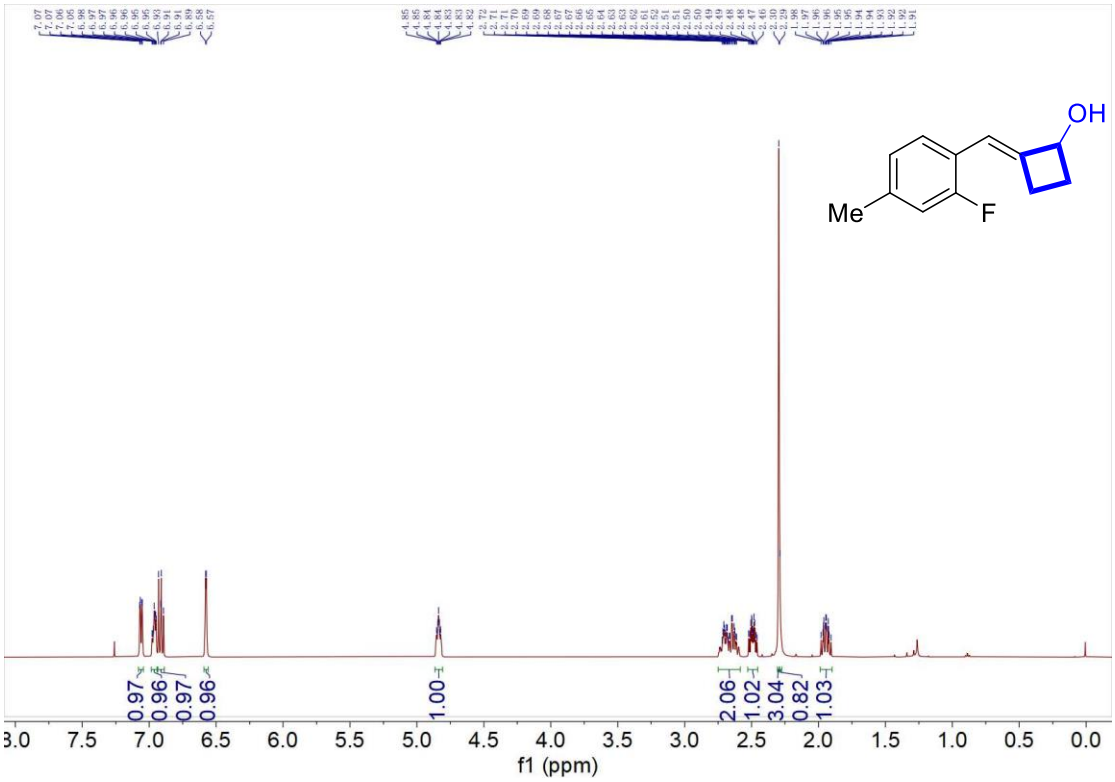


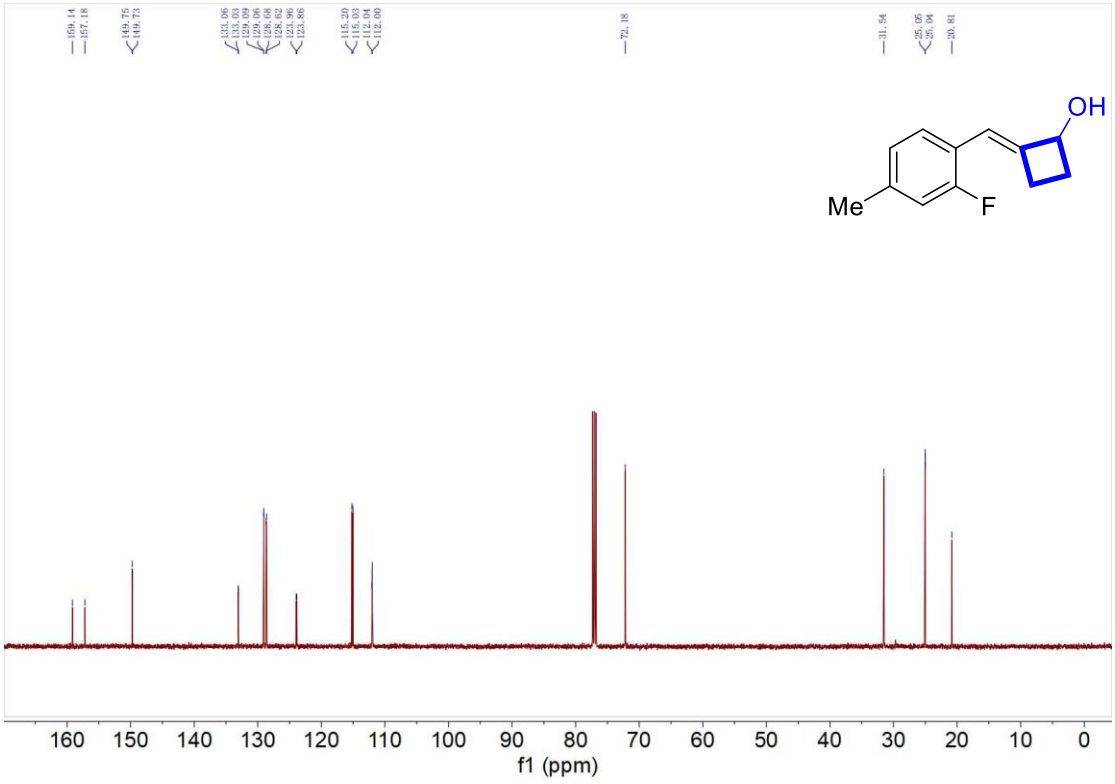
(*E*)-2-(4-chlorobenzylidene)cyclobutan-1-ol (2k)



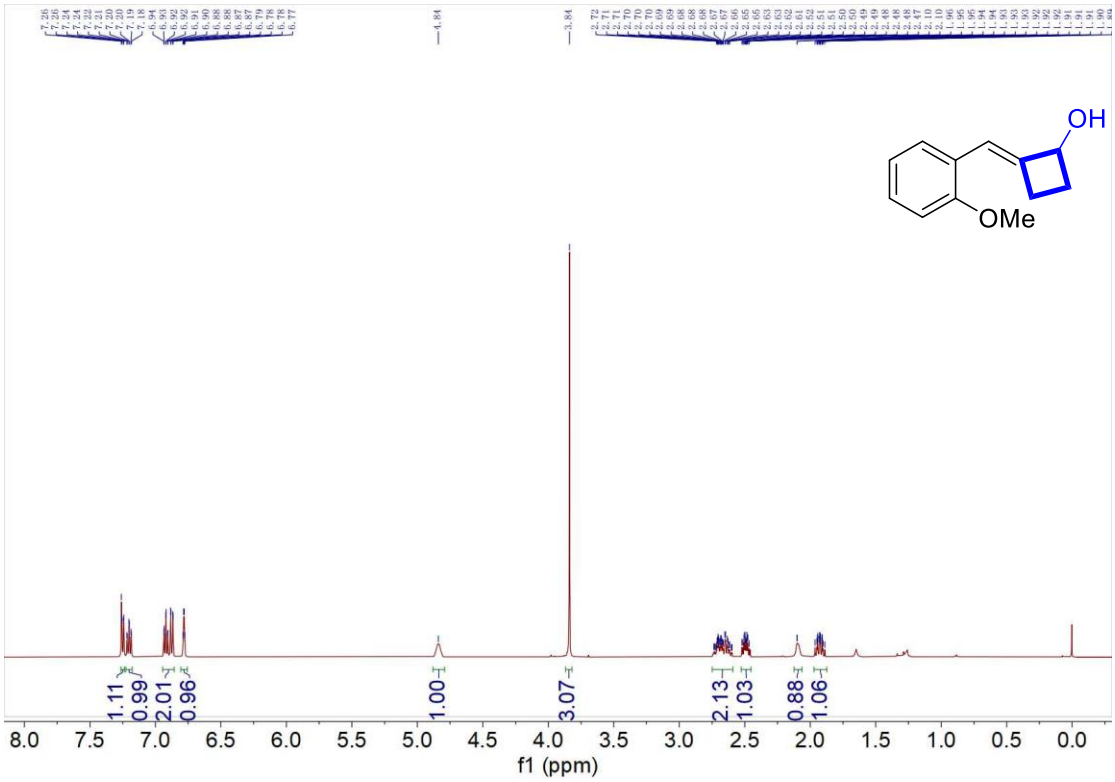


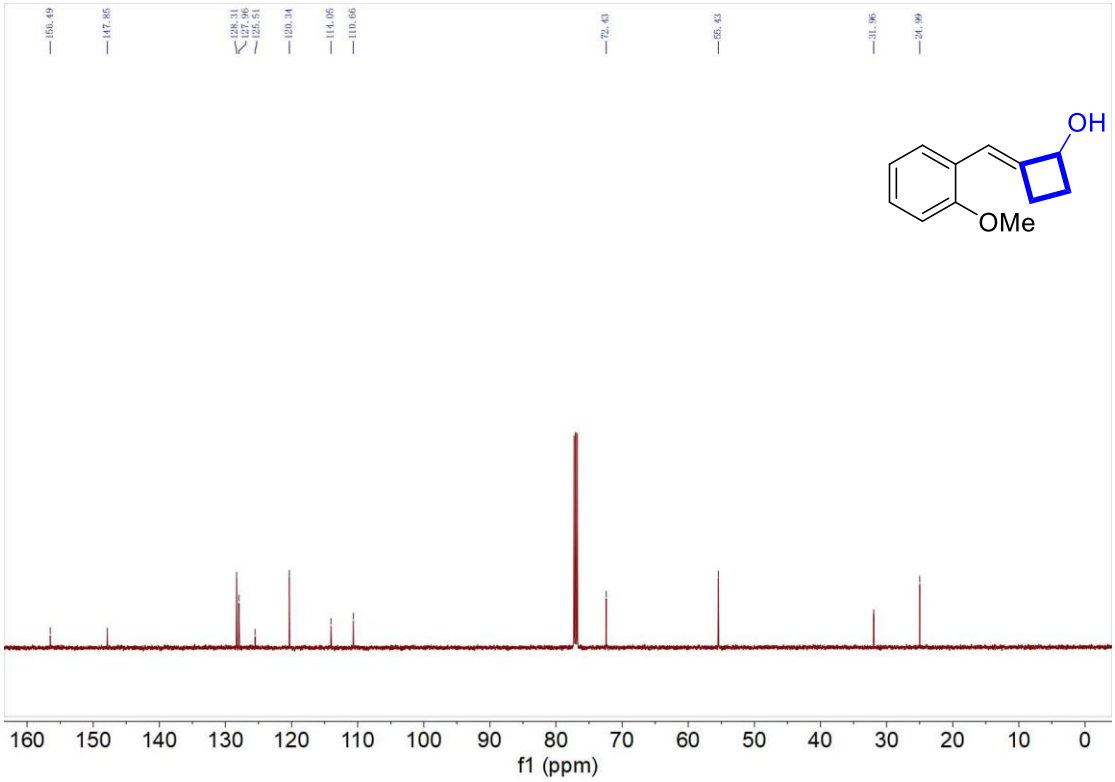
(*E*)-2-(2-fluoro-4-methylbenzylidene)cyclobutan-1-ol (2l)



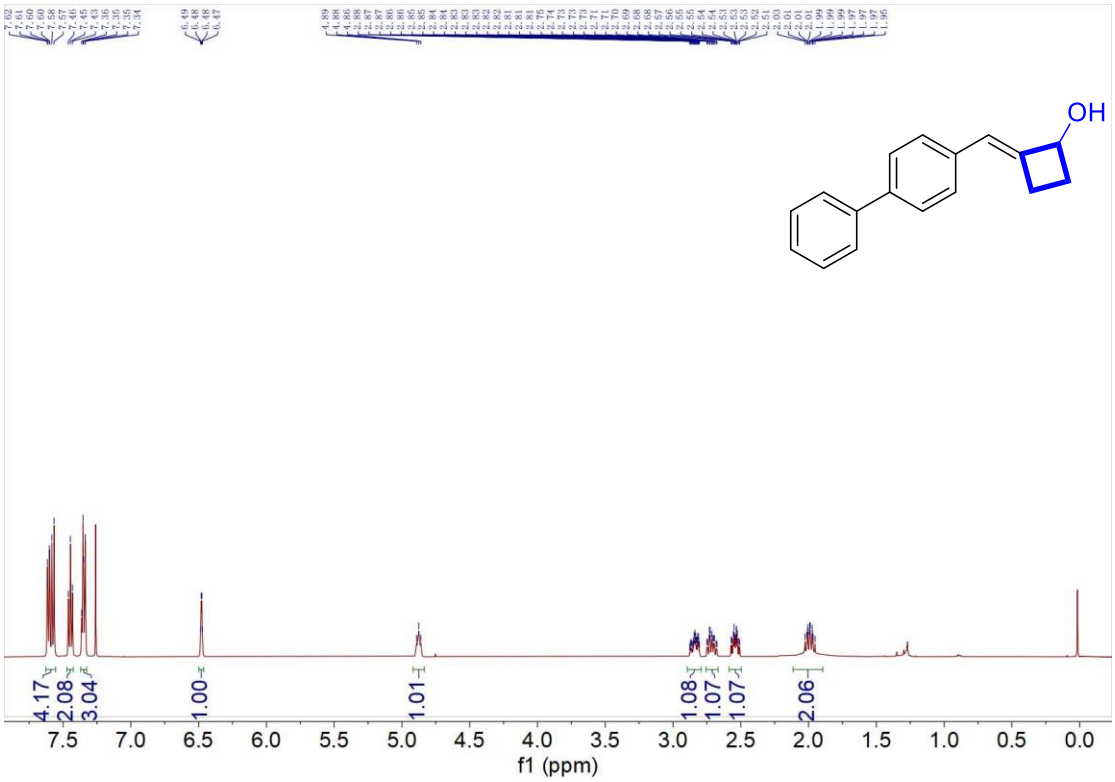


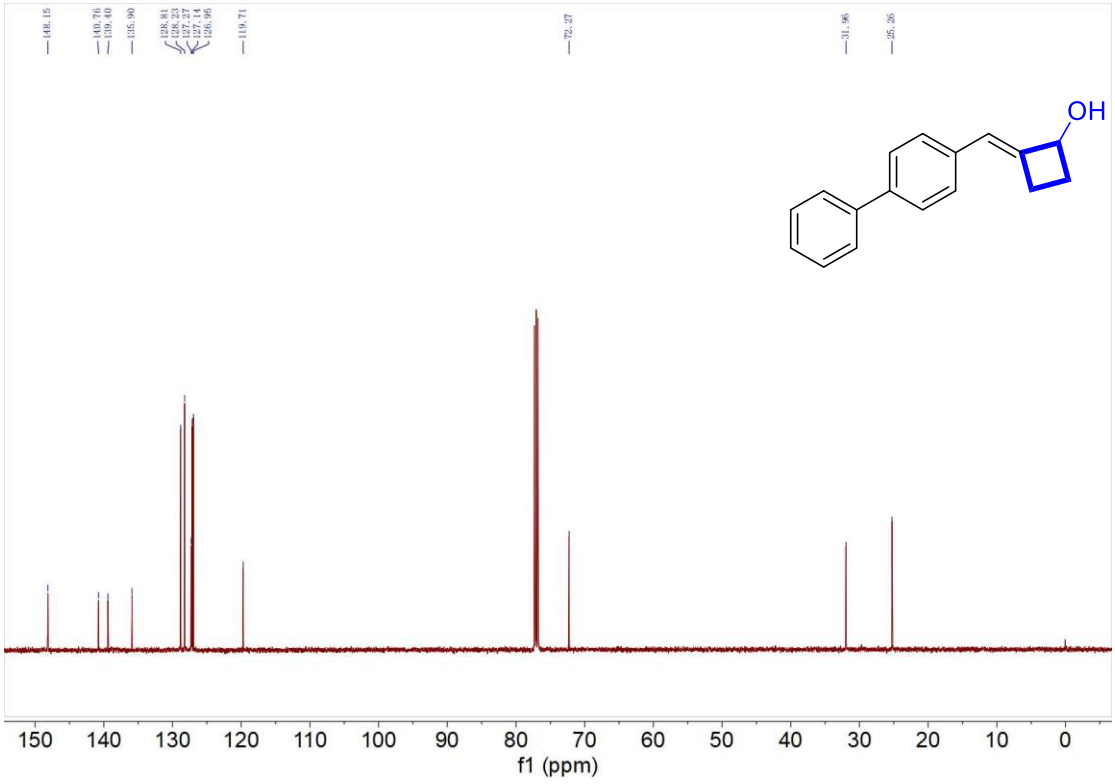
(*E*)-2-(2-methoxybenzylidene)cyclobutan-1-ol (2m)



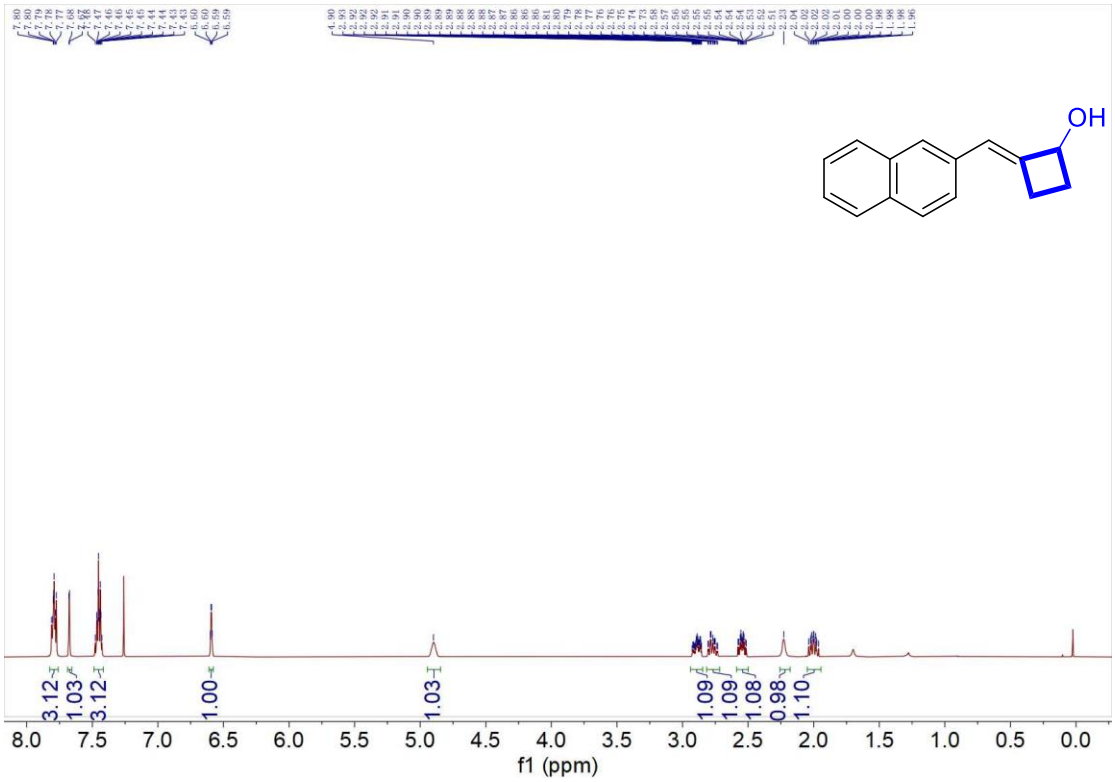


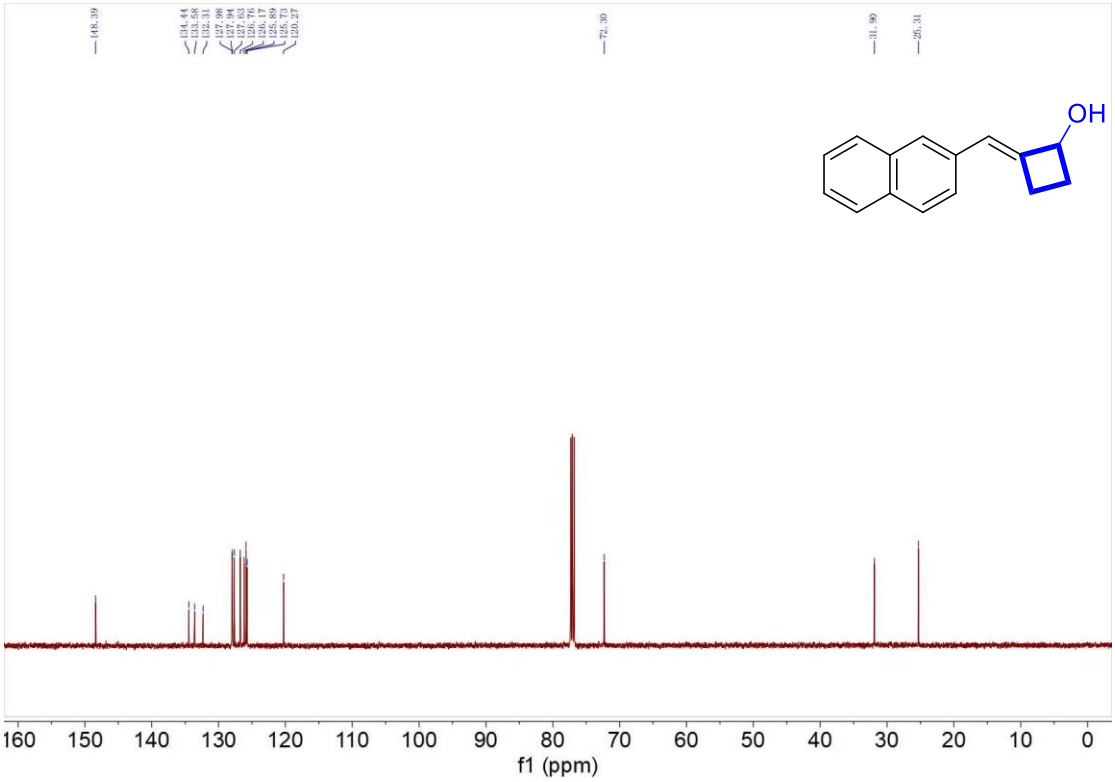
(*E*)-2-([1,1'-biphenyl]-4-ylmethylene)cyclobutan-1-ol (2n)



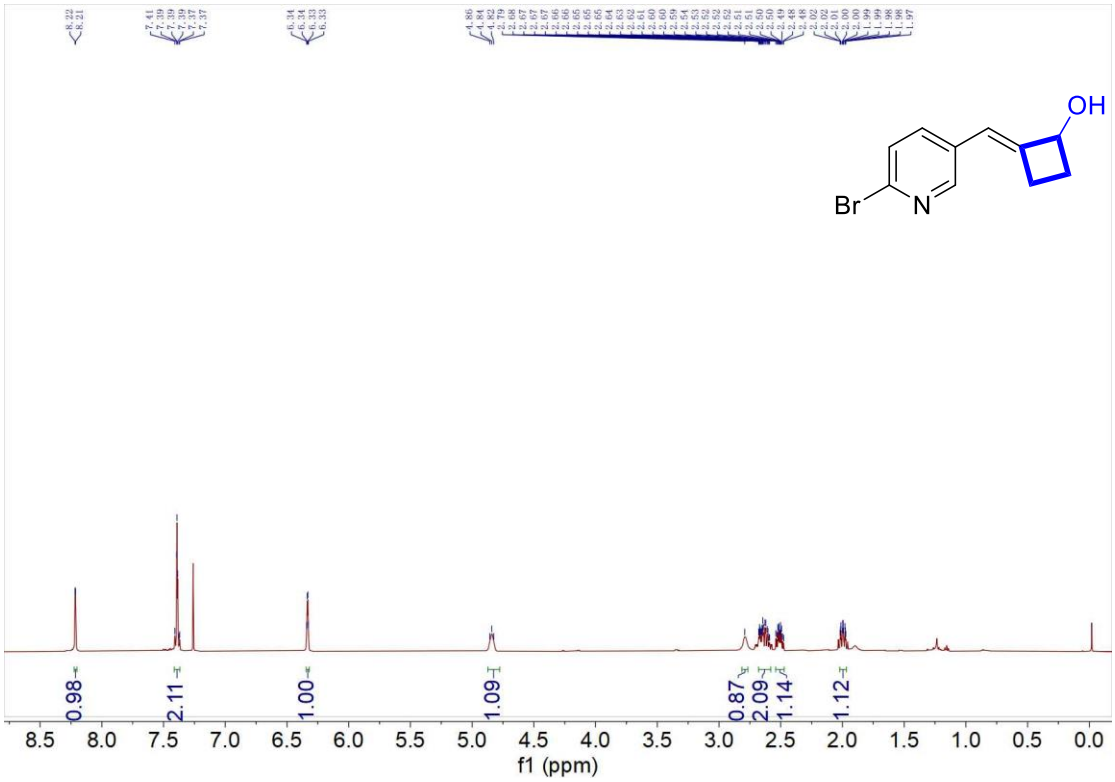


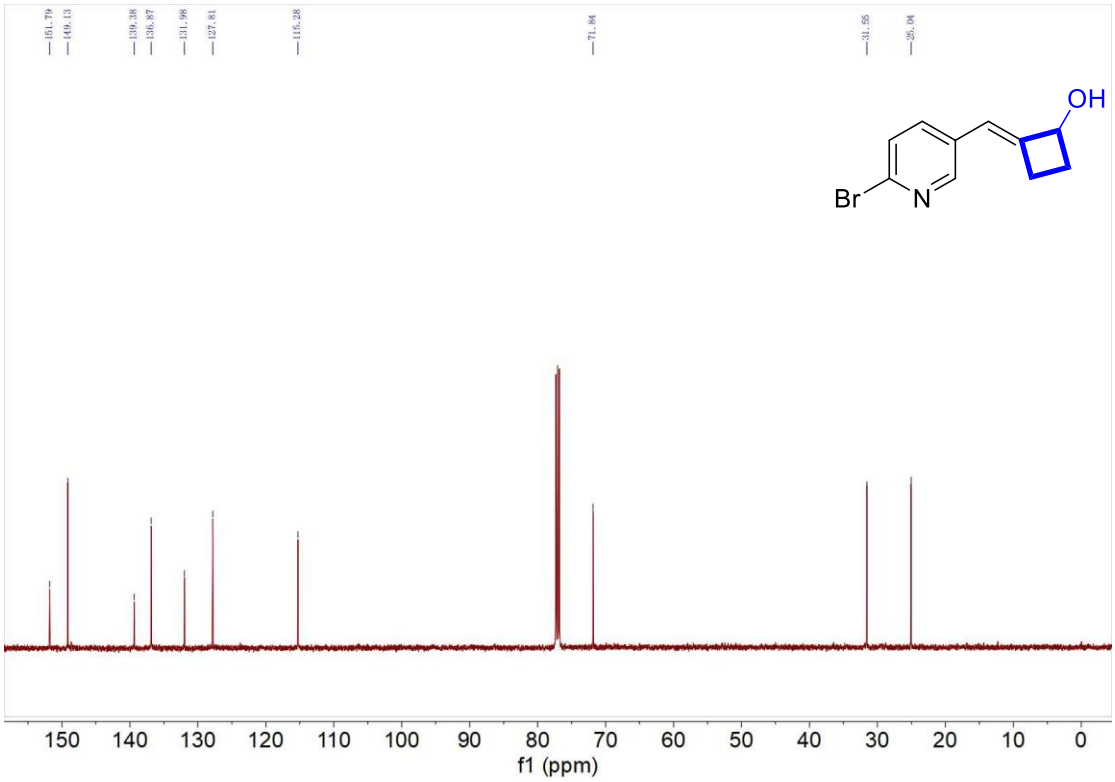
(*E*)-2-(naphthalen-2-ylmethylene)cyclobutan-1-ol (2o)



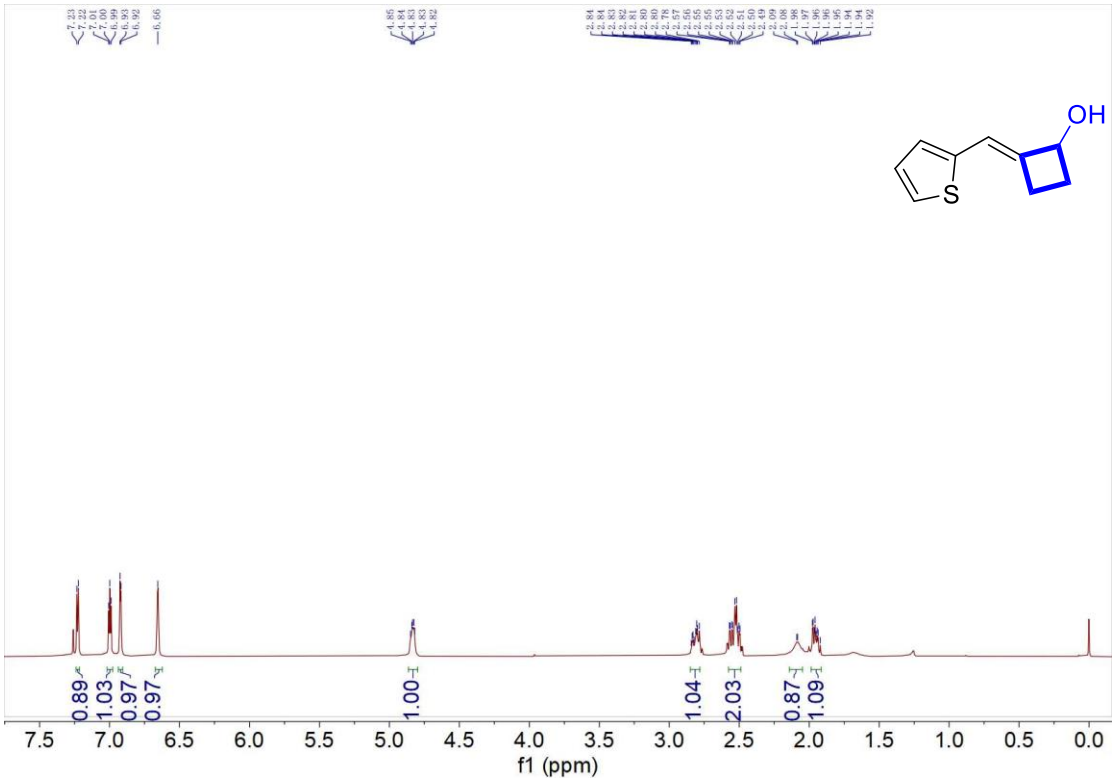


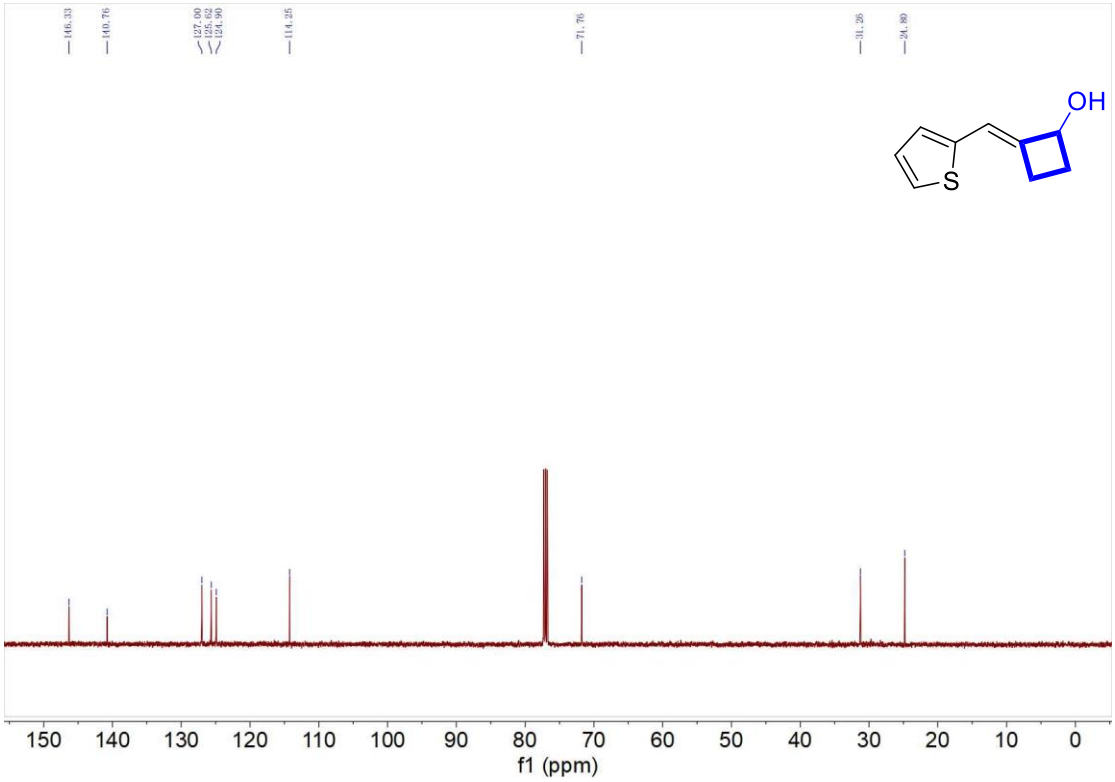
(*E*)-2-((6-bromopyridin-3-yl)methylene)cyclobutan-1-ol (2p)



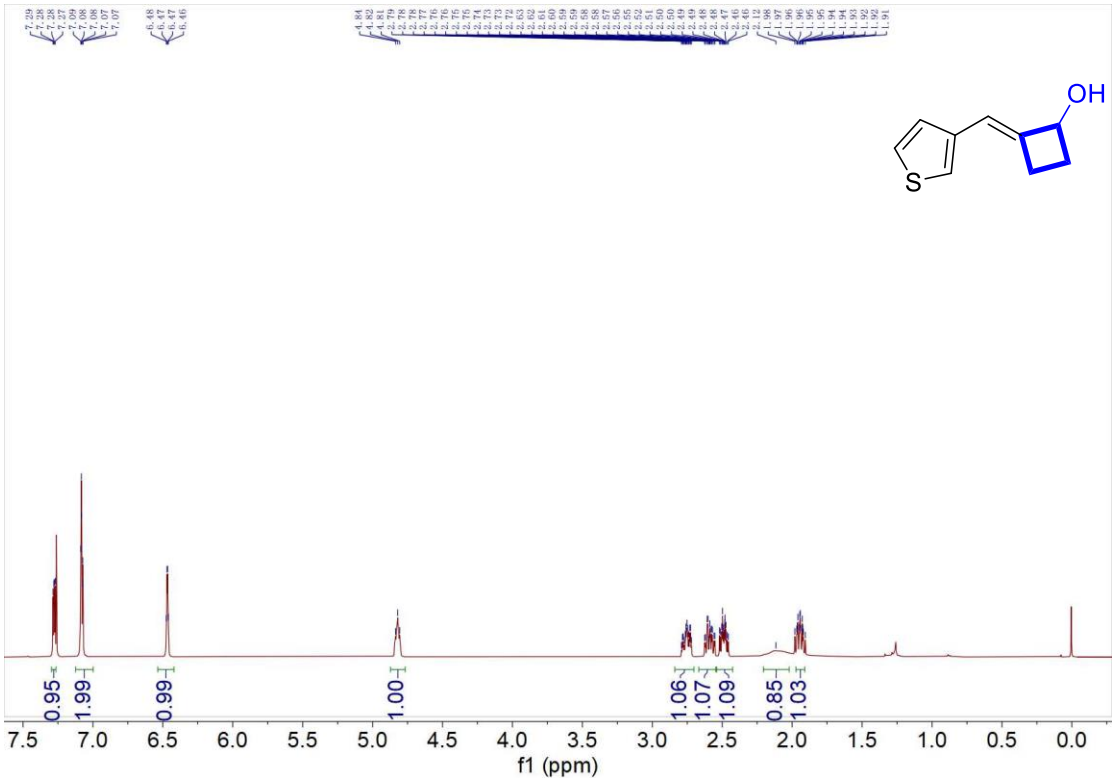


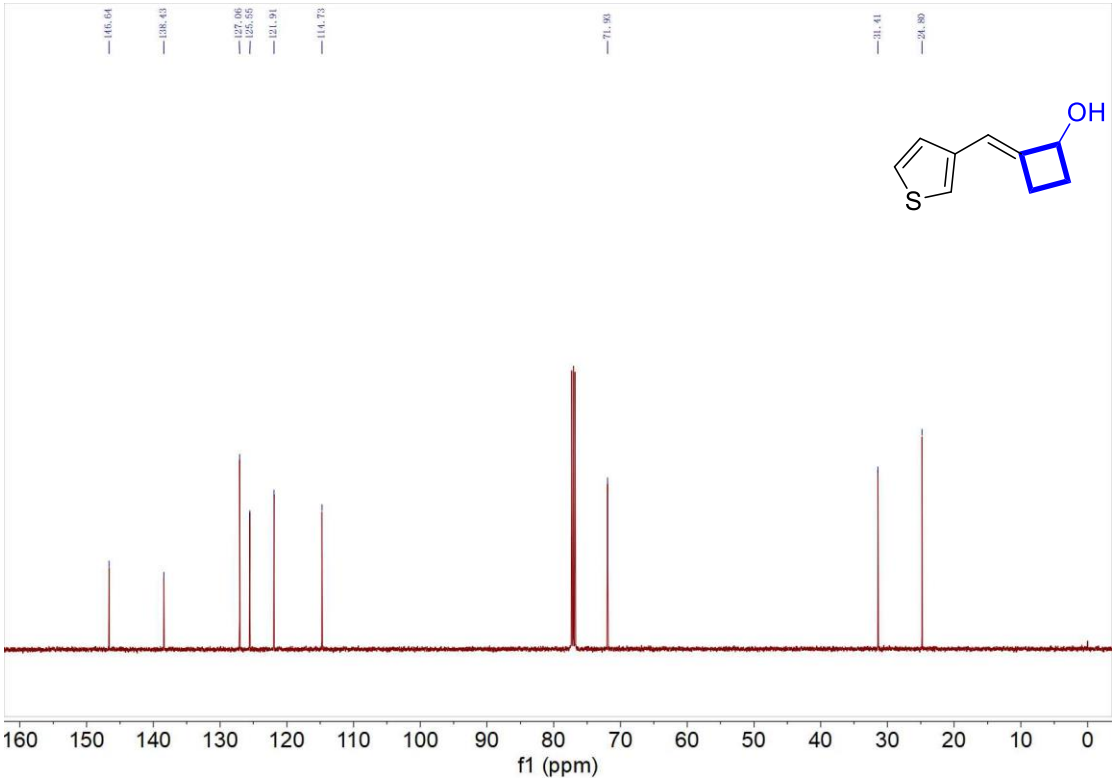
(*E*)-2-(thiophen-2-ylmethylene)cyclobutan-1-ol (2q)



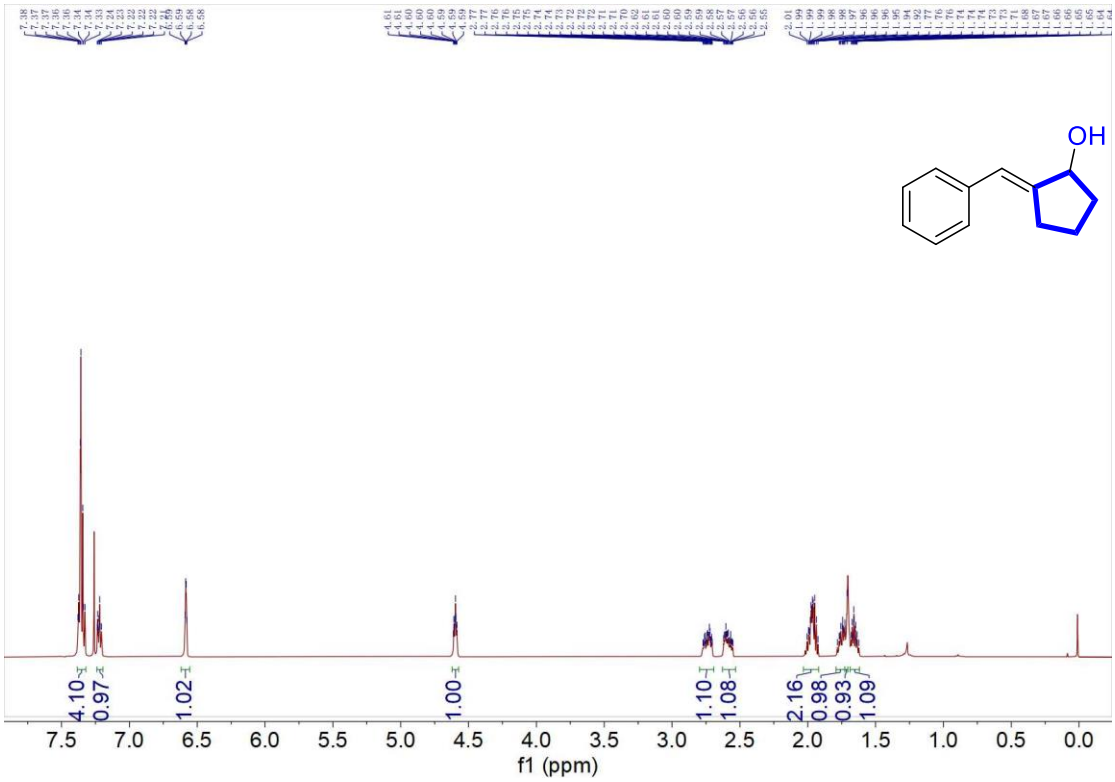


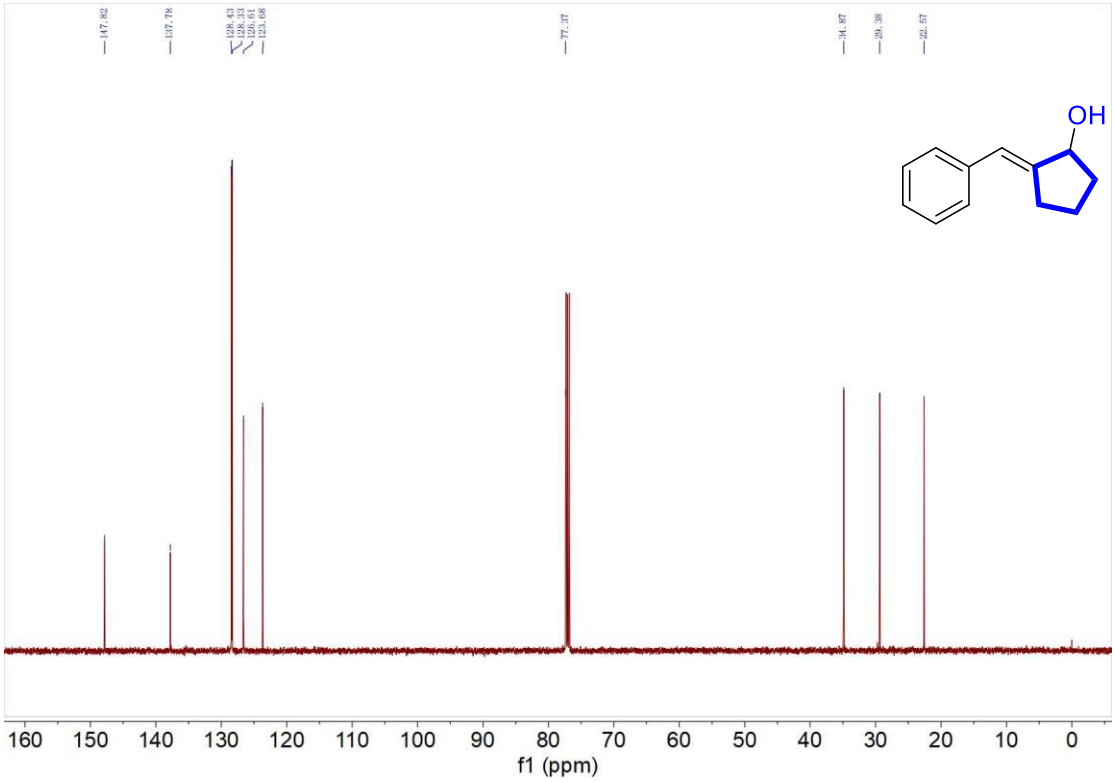
2-(thiophen-3-ylmethyl)cyclobutan-1-ol (2r)

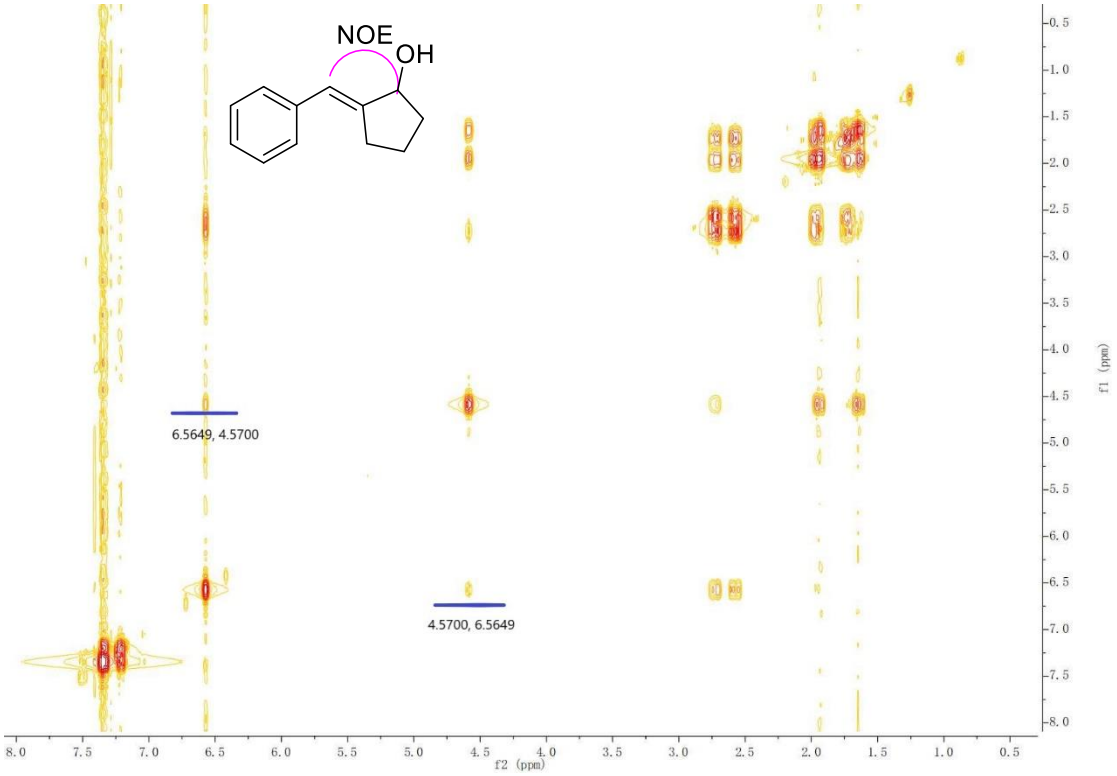




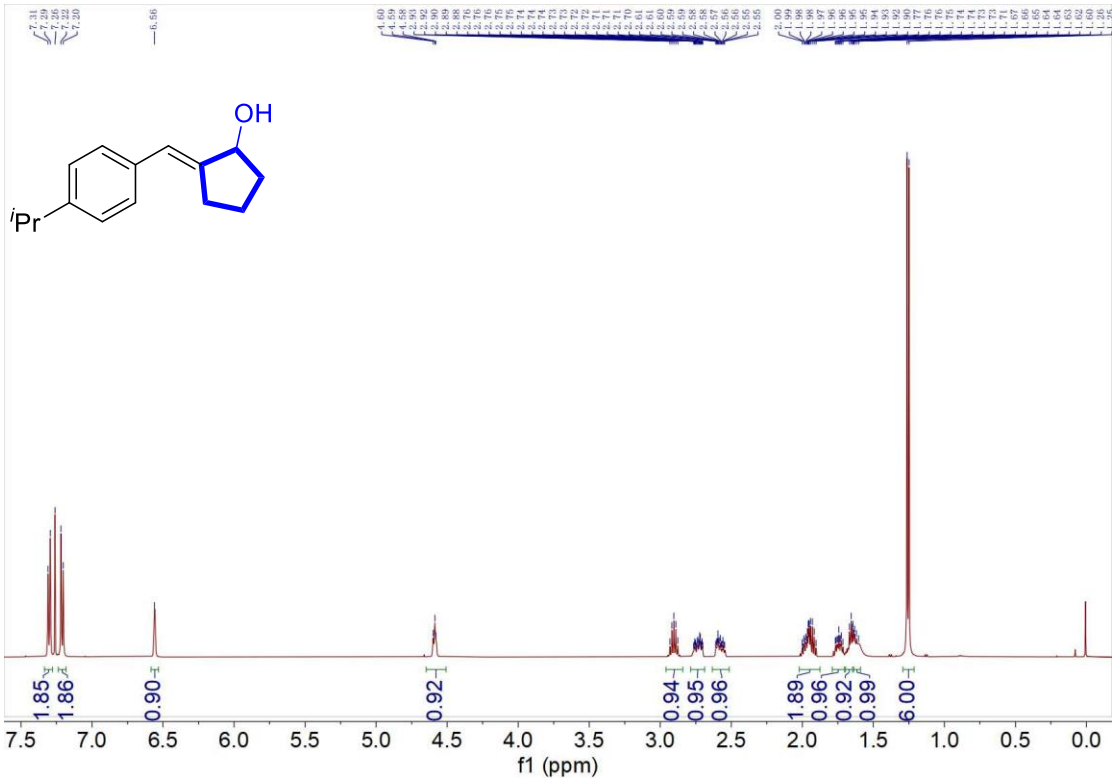
(*E*)-2-benzylidenecyclopentan-1-ol (4a)

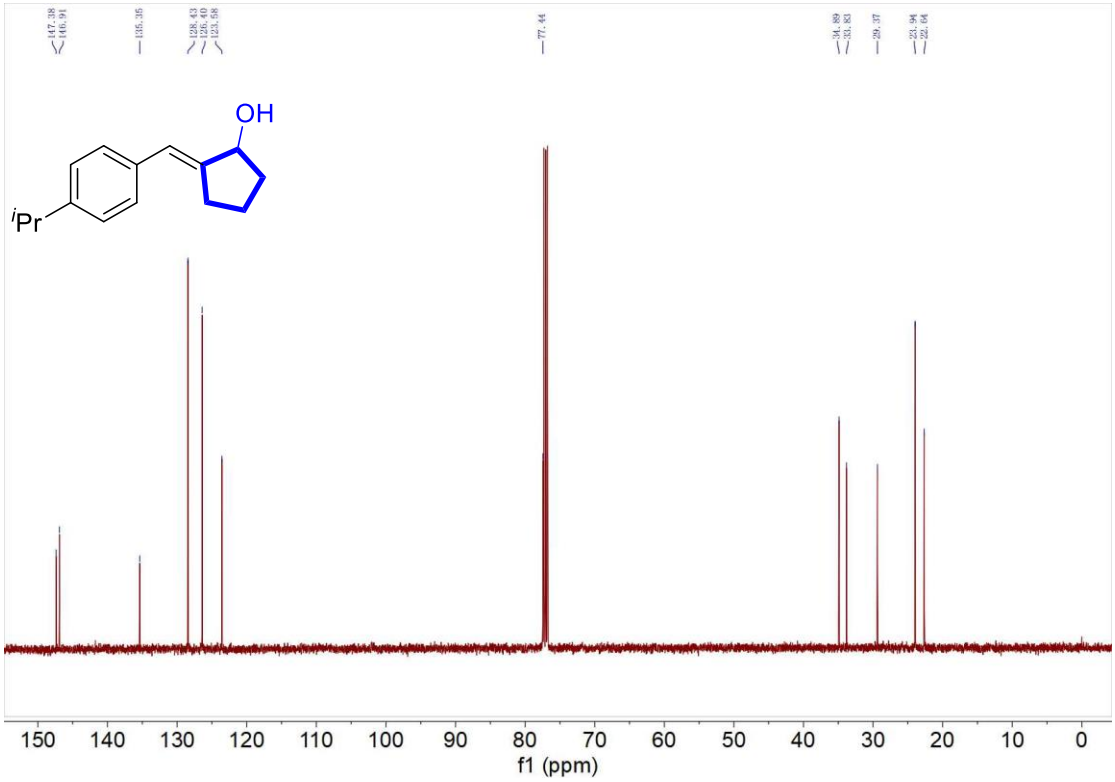




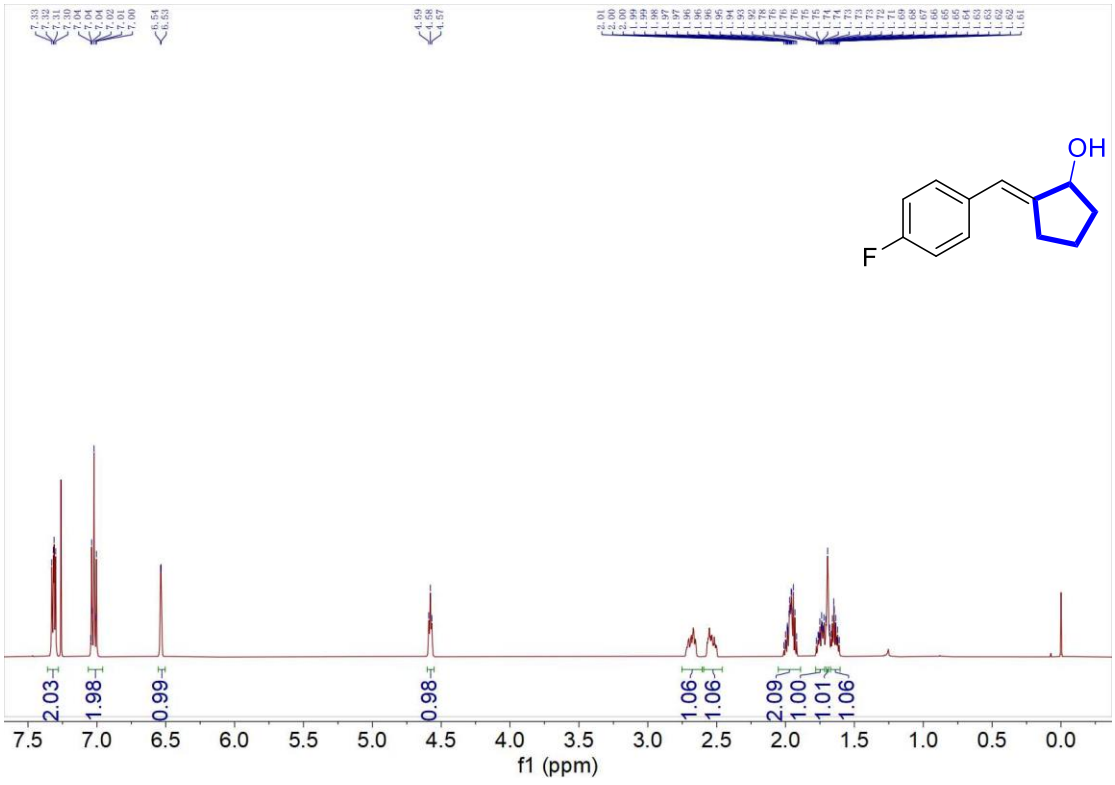

NOE
OH
6.5649, 4.5700
4.5700, 6.5649
f2 (ppm)
f1 (ppm)

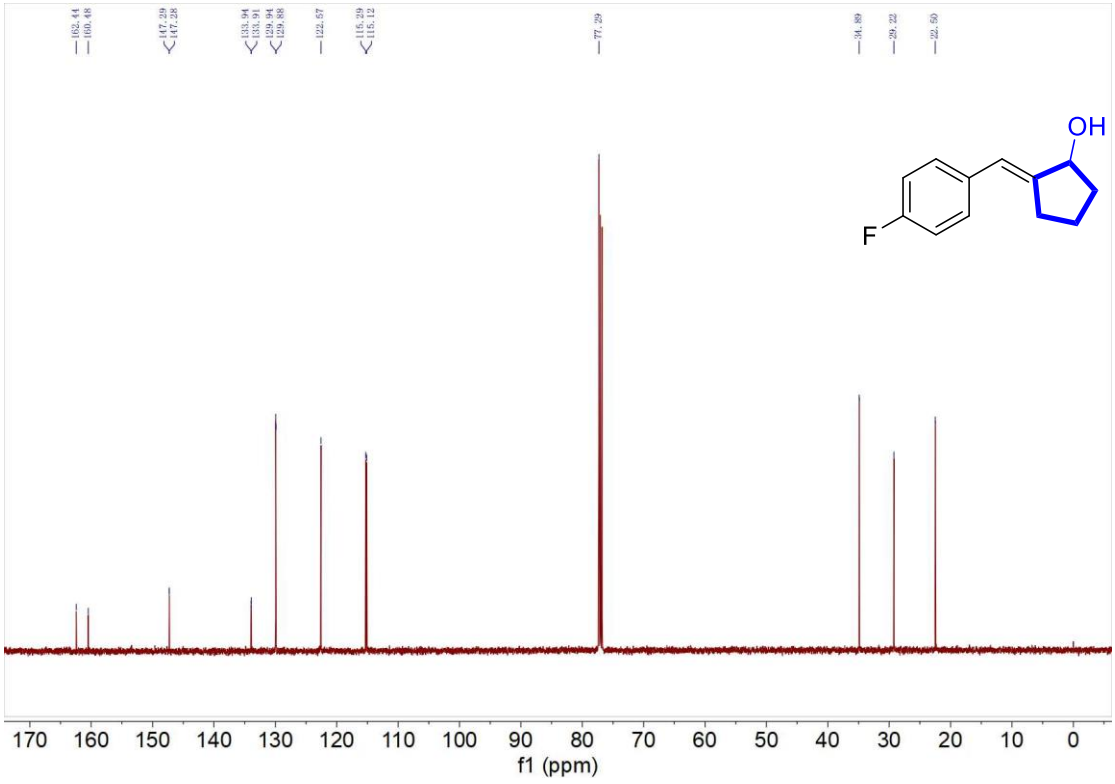
(*E*)-2-(4-isopropylbenzylidene)cyclopentan-1-ol (4b)



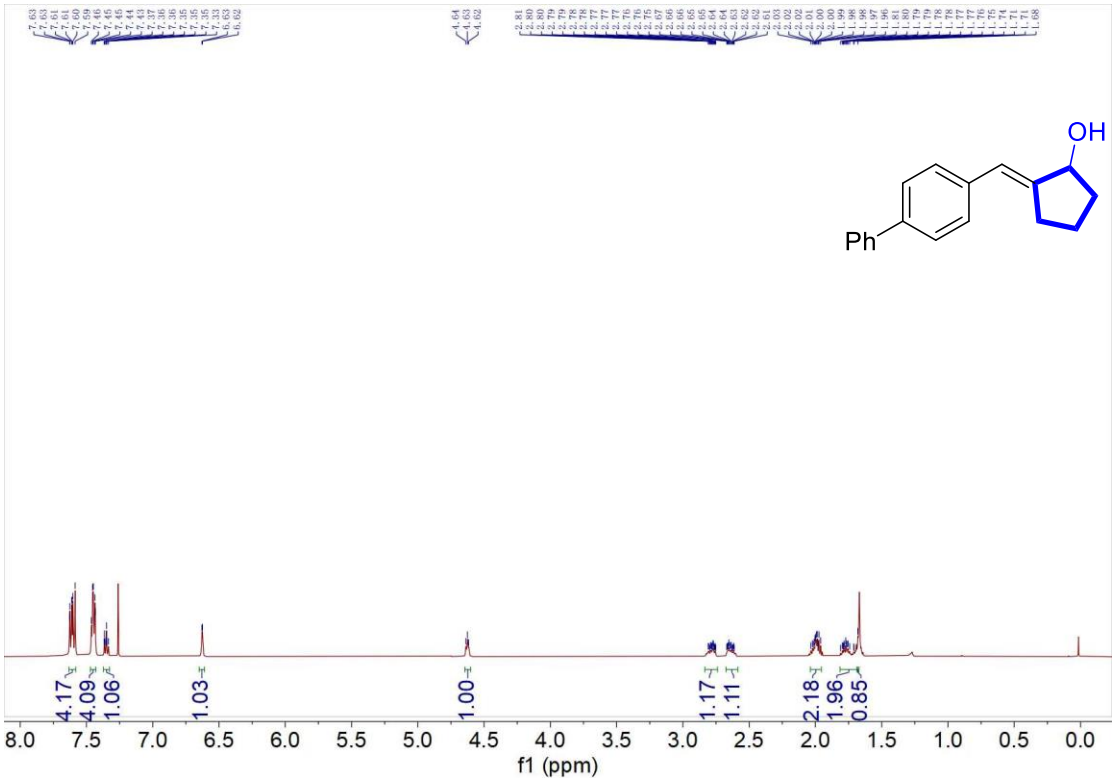


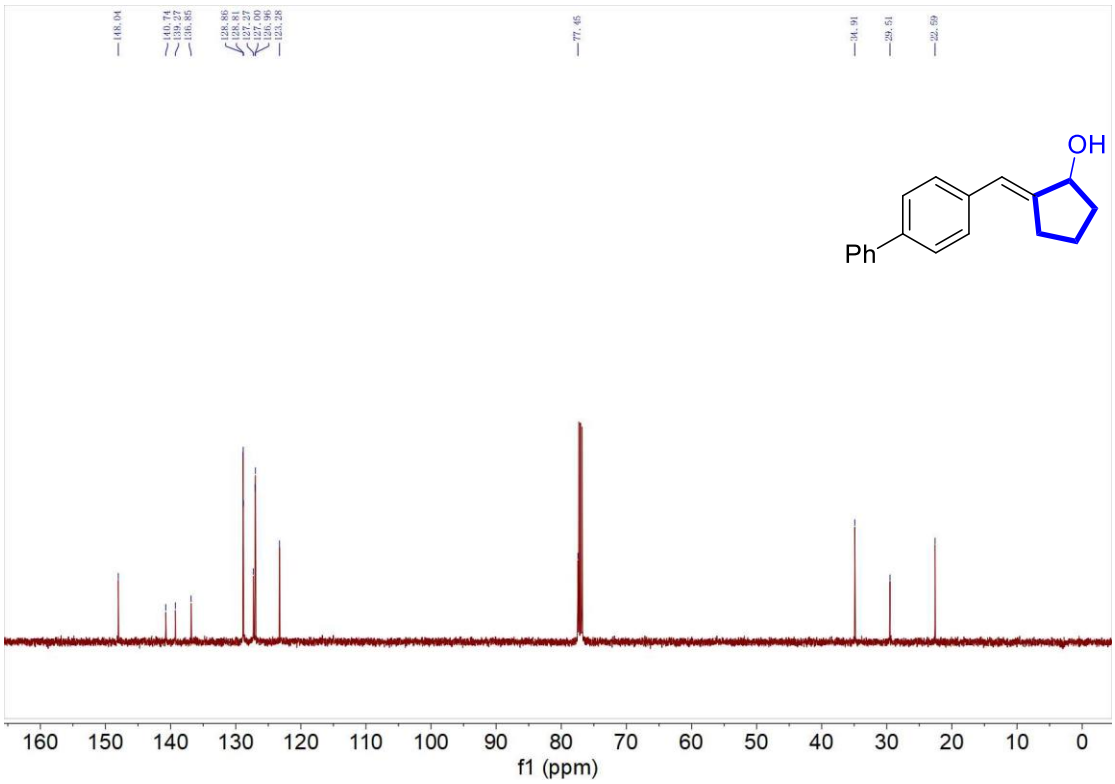
(*E*)-2-(4-fluorobenzylidene)cyclopentan-1-ol (4c)



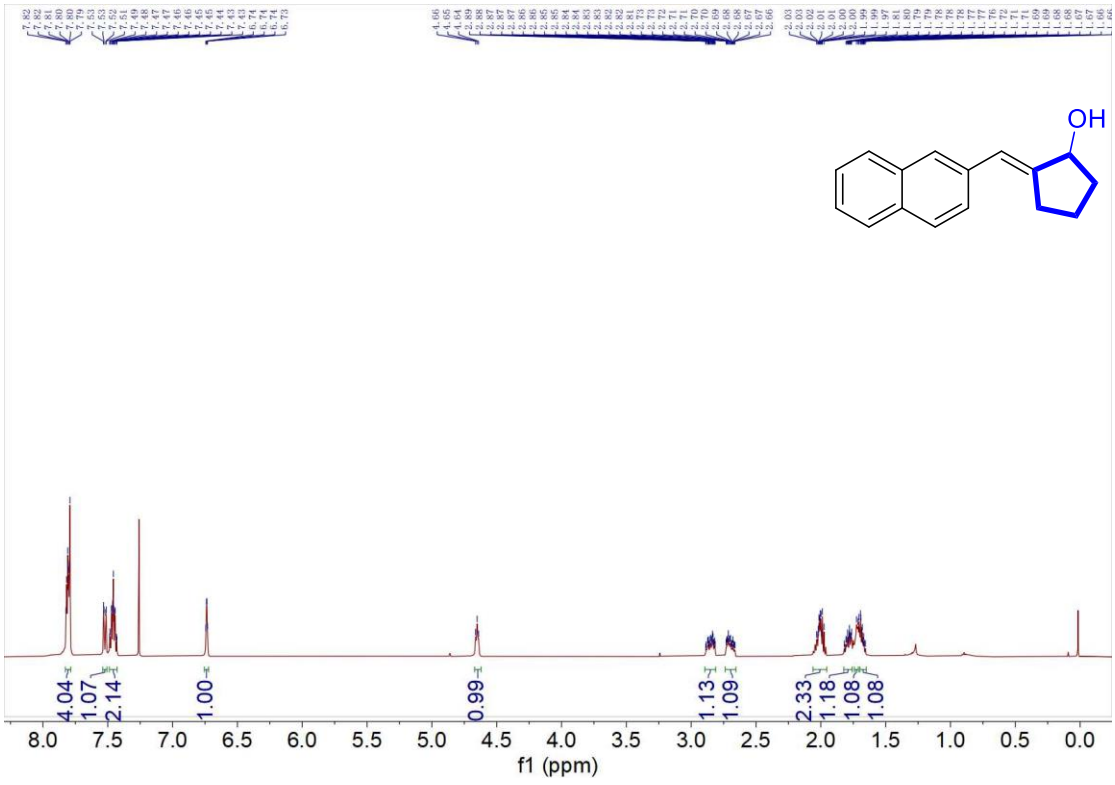


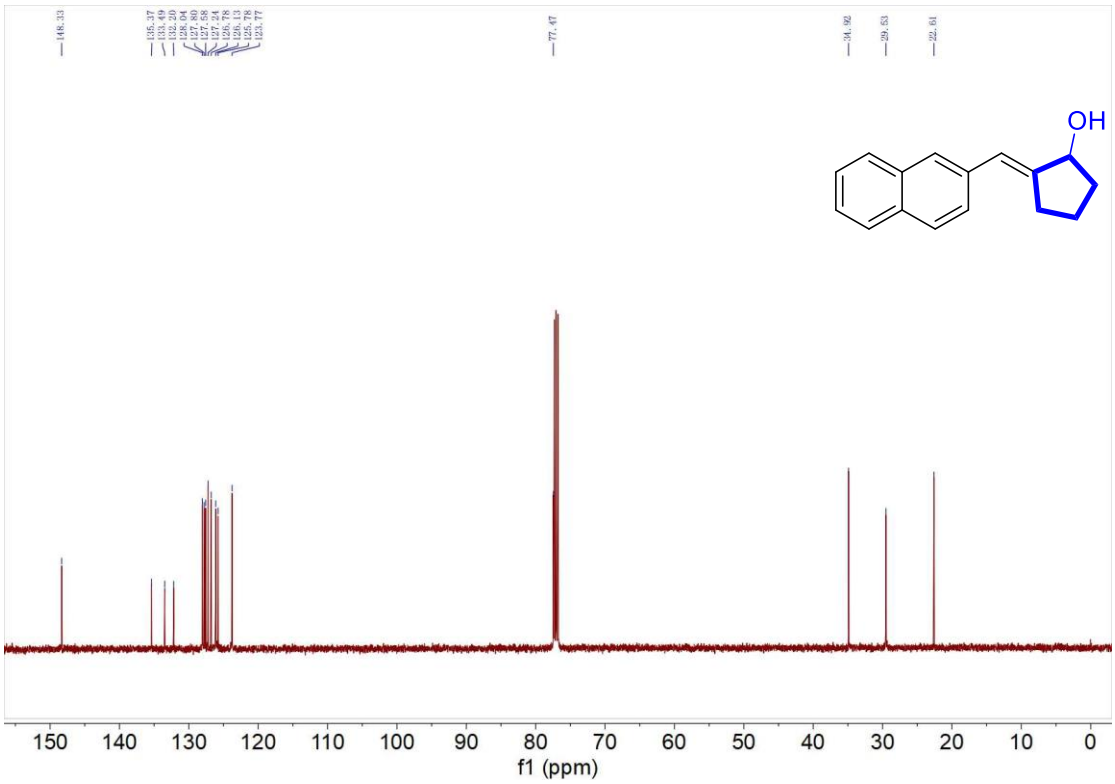
(*E*)-2-([1,1'-biphenyl]-4-ylmethylene)cyclopentan-1-ol (4d)



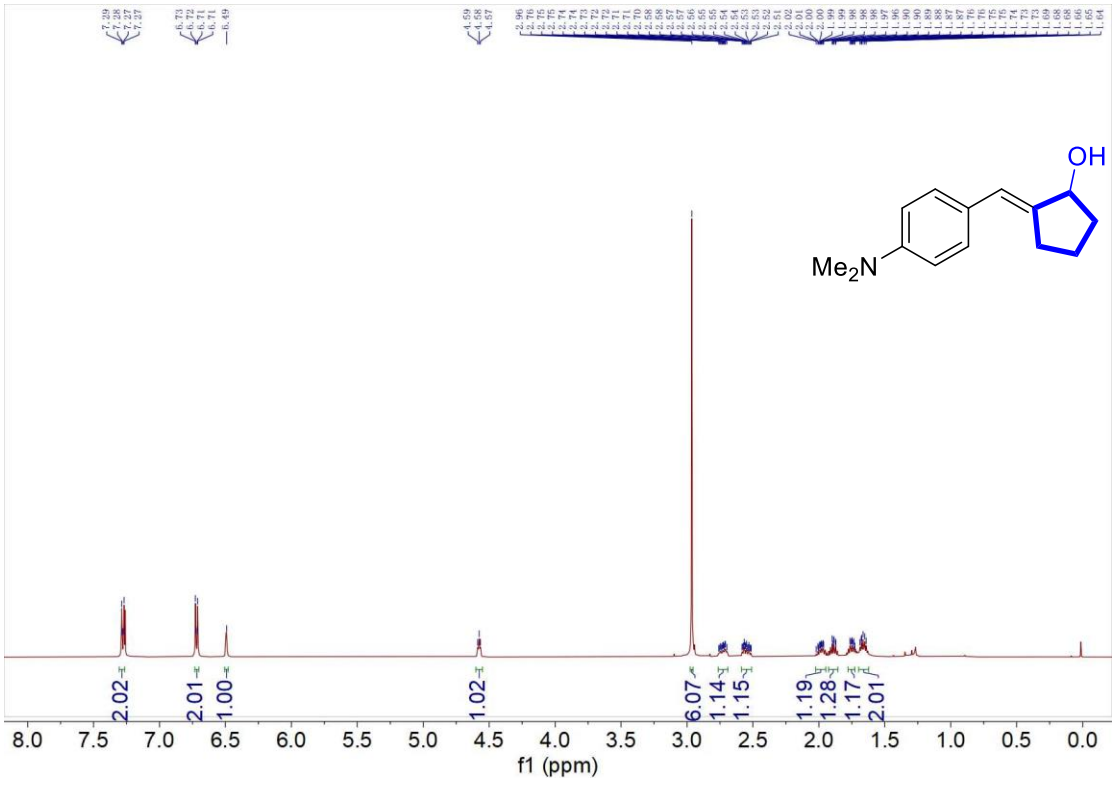


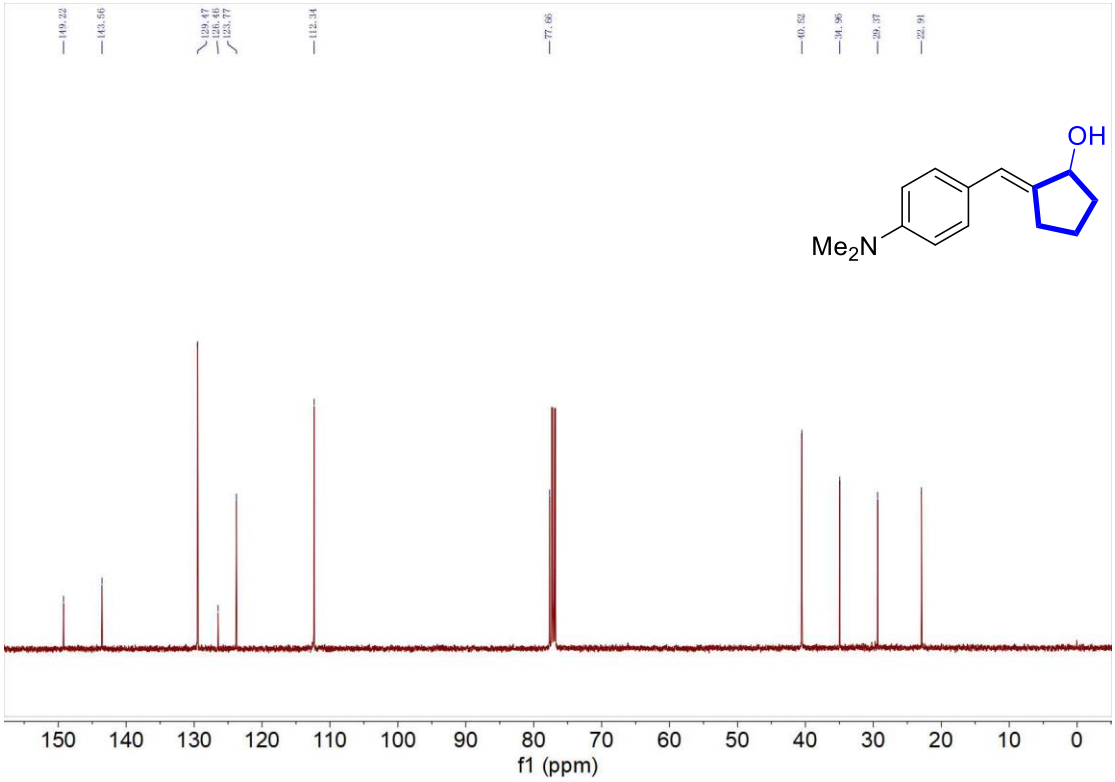
(*E*)-2-(naphthalen-2-ylmethylene)cyclopentan-1-ol (4e)



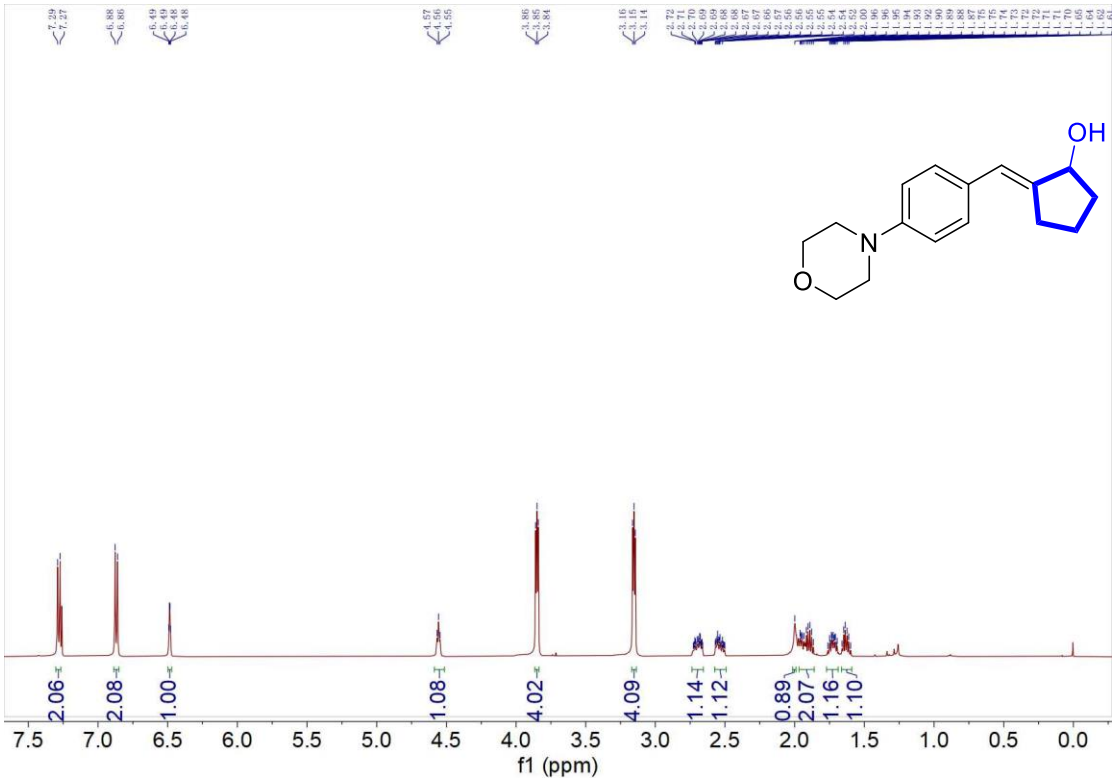


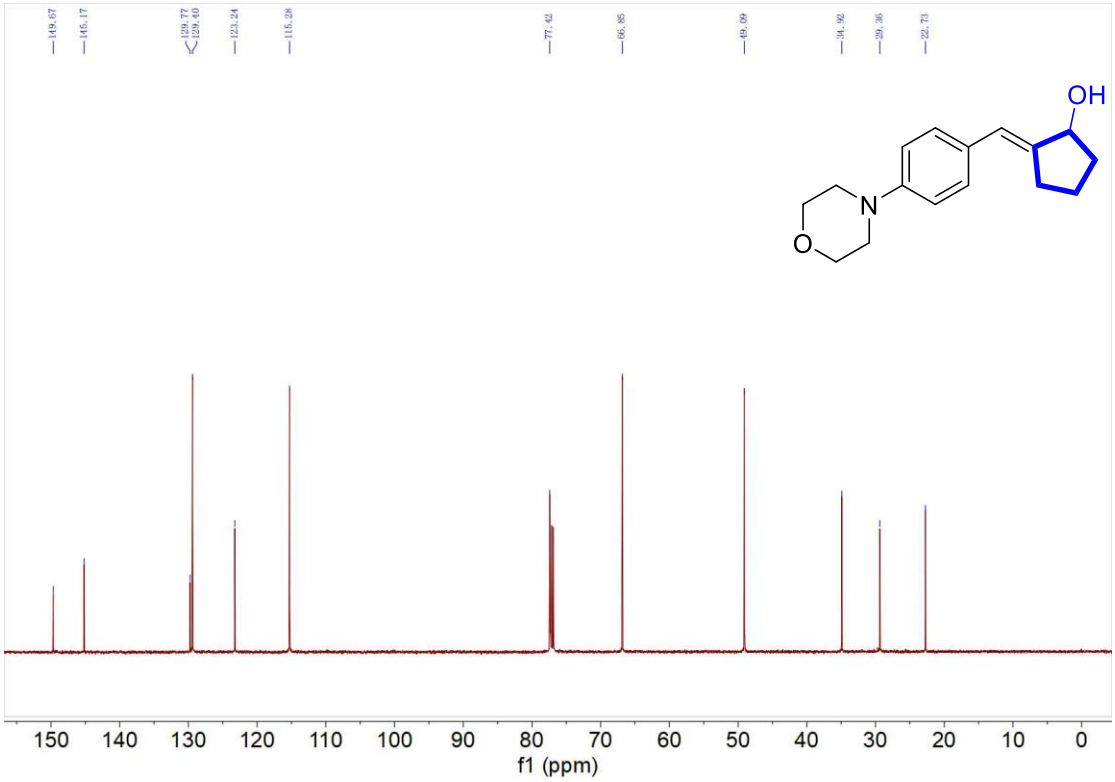
(*E*)-2-(4-(dimethylamino)benzylidene)cyclopentan-1-ol (4f)



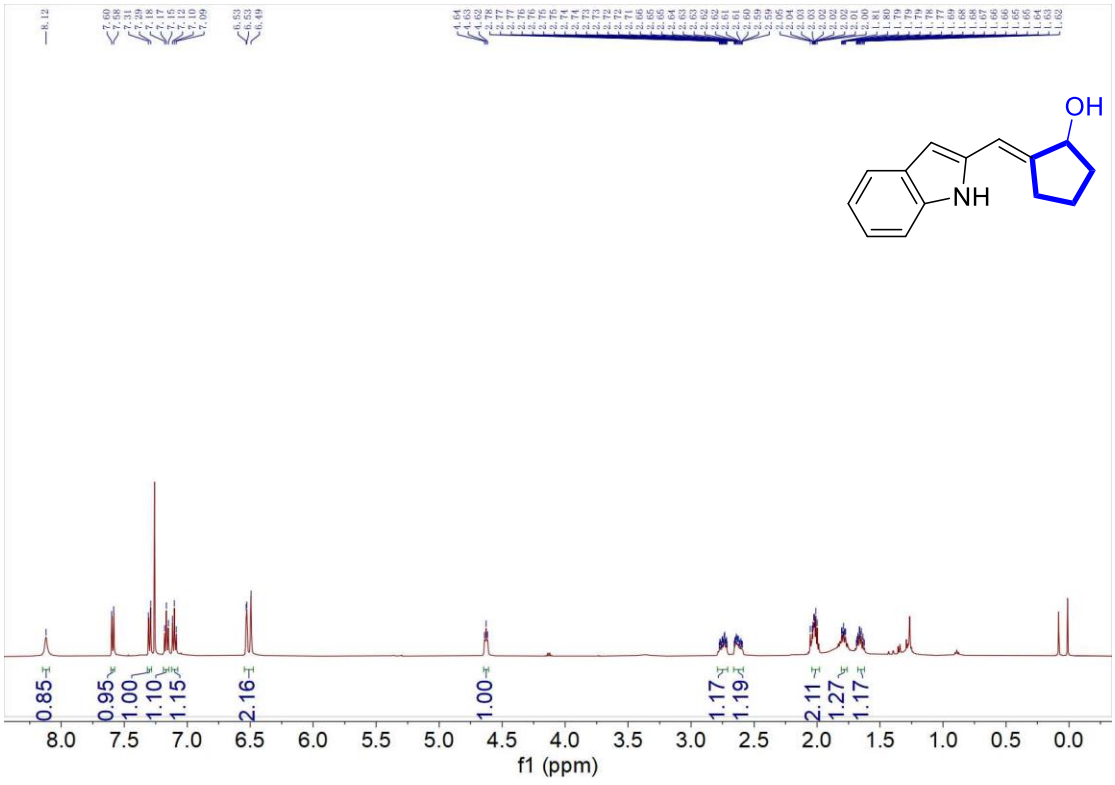


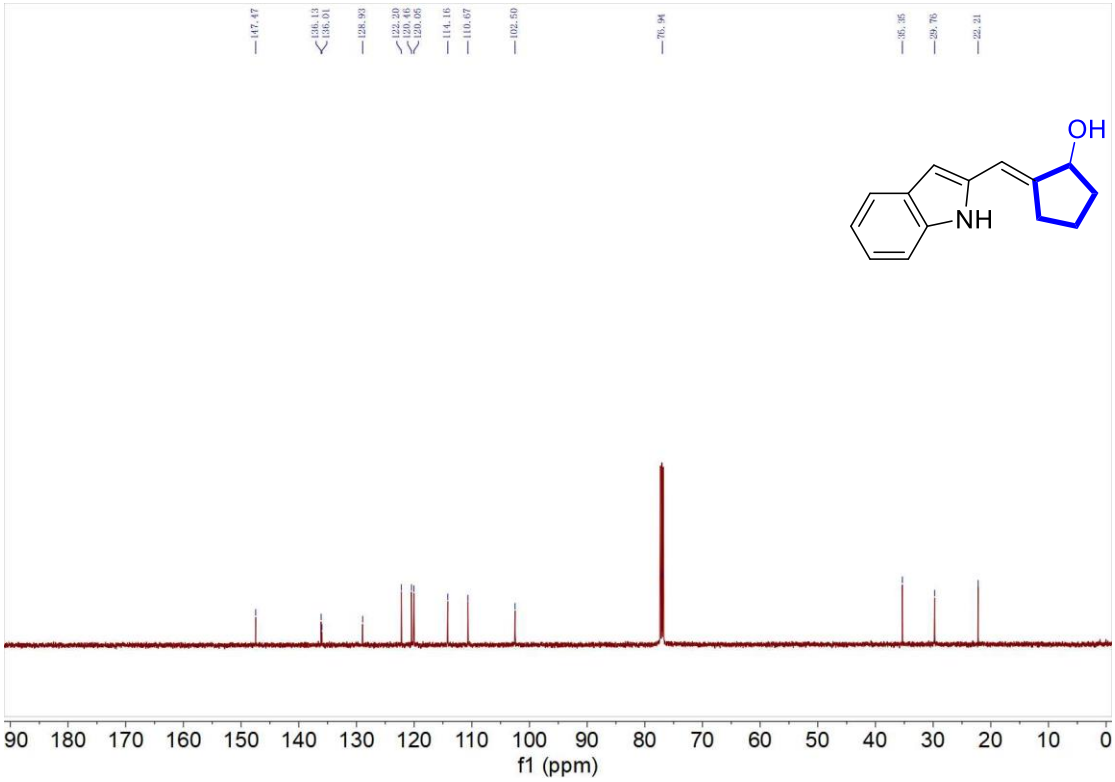
(*E*)-2-(4-morpholinobenzylidene)cyclopentan-1-ol (4g)



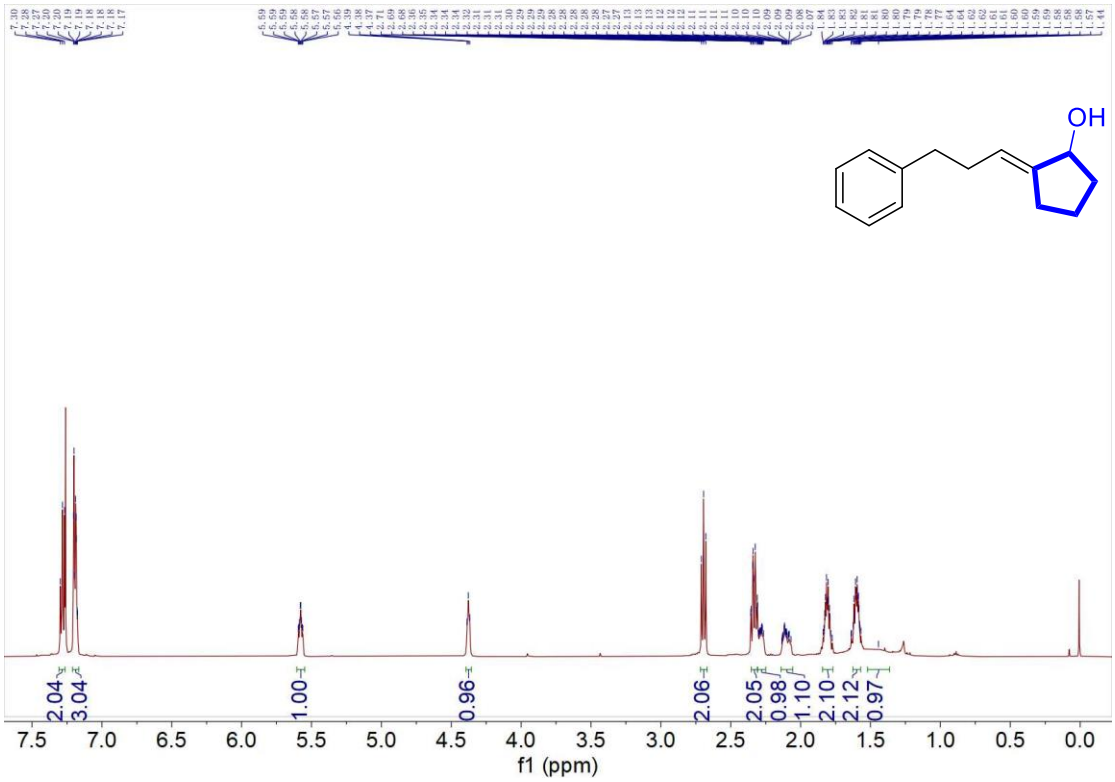


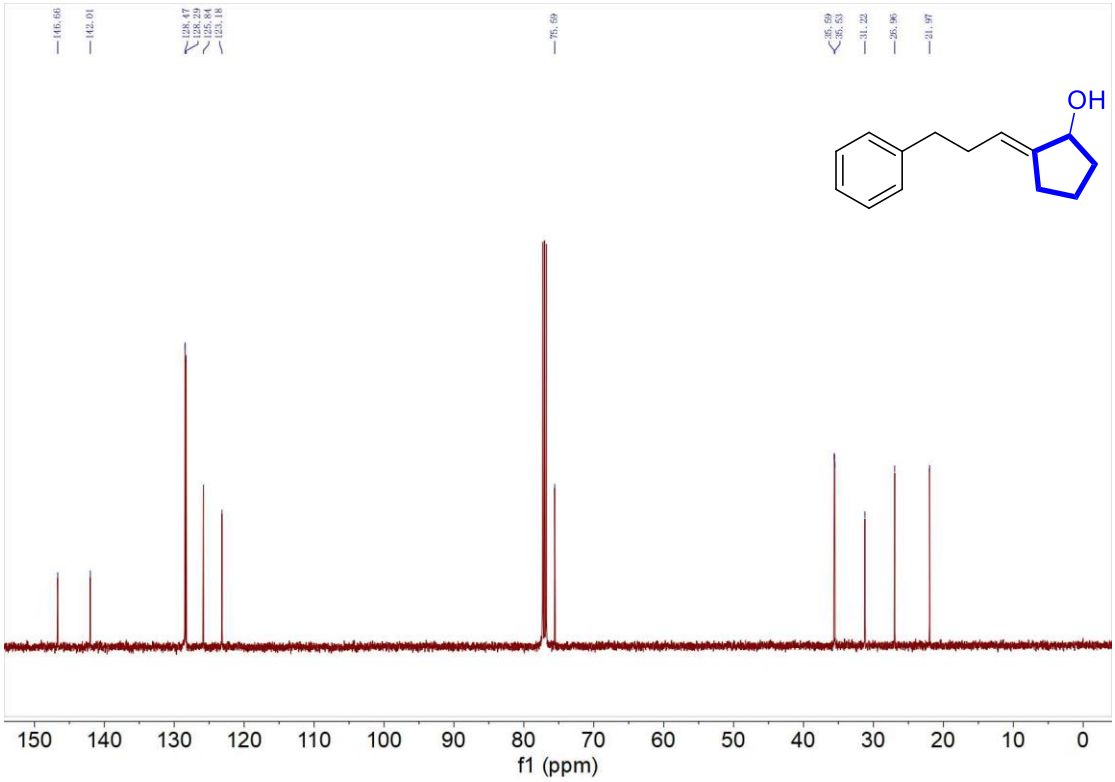
(*E*)-2-((1H-indol-2-yl)methylene)cyclopentan-1-ol (4h)



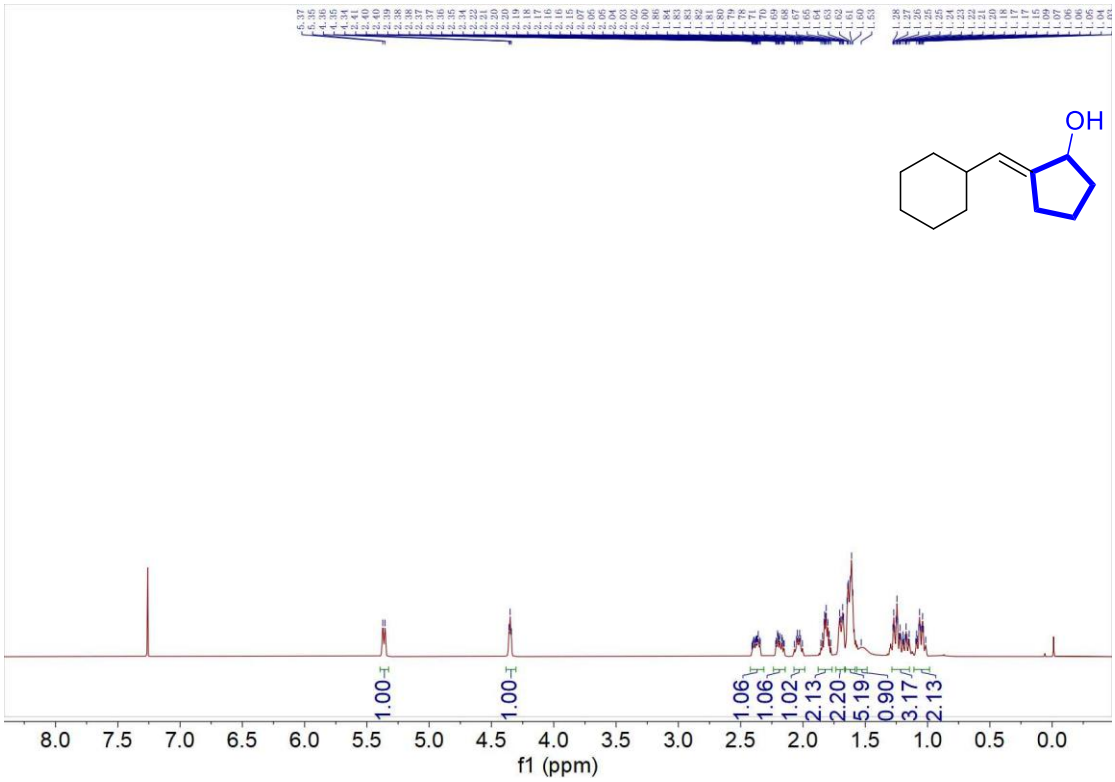


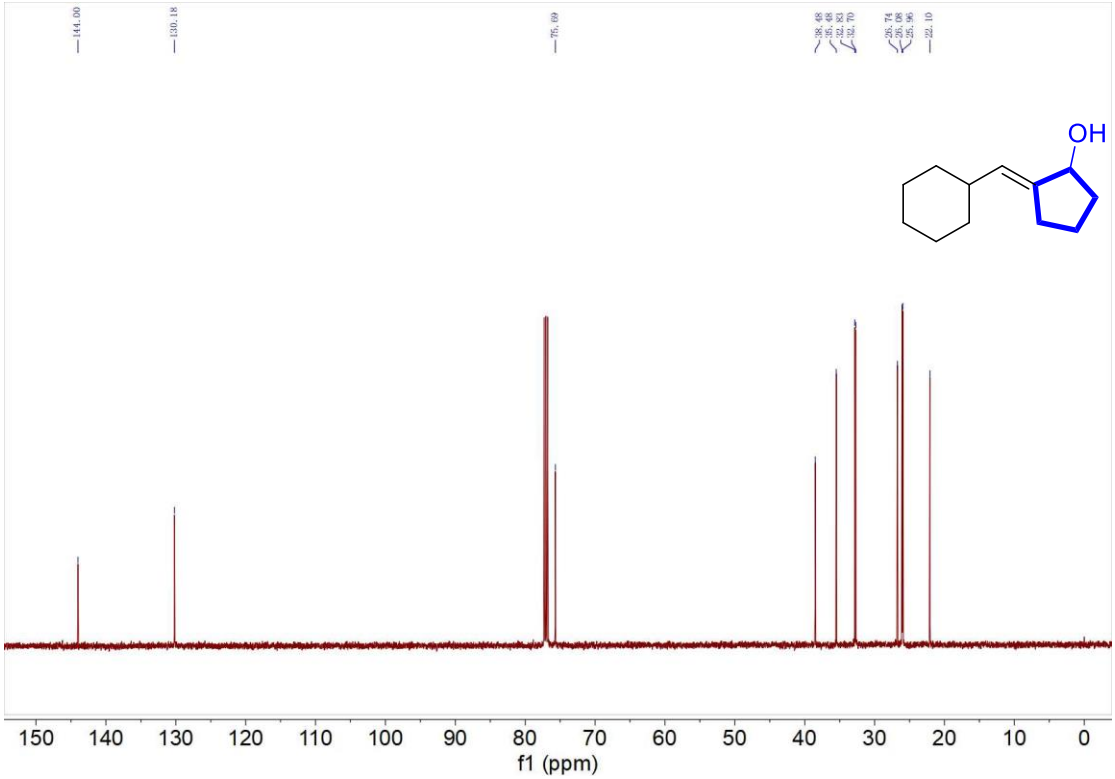
(*E*)-2-(3-phenylpropylidene)cyclopentan-1-ol (4i)



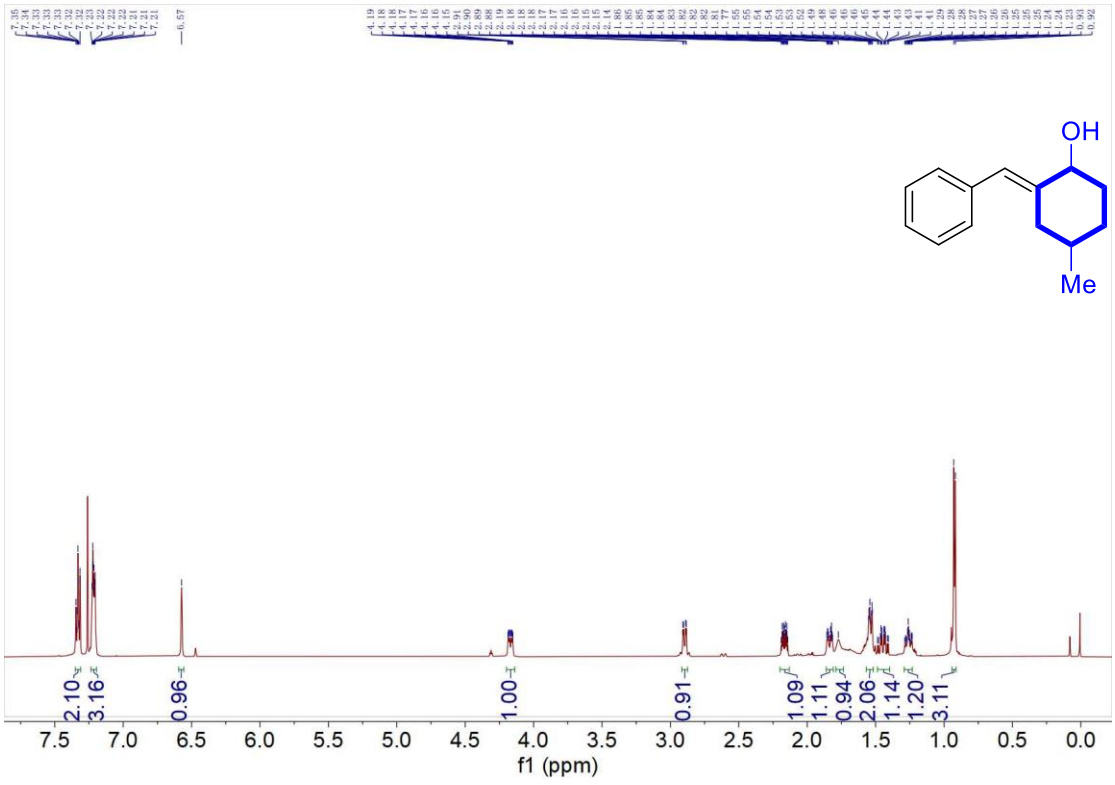


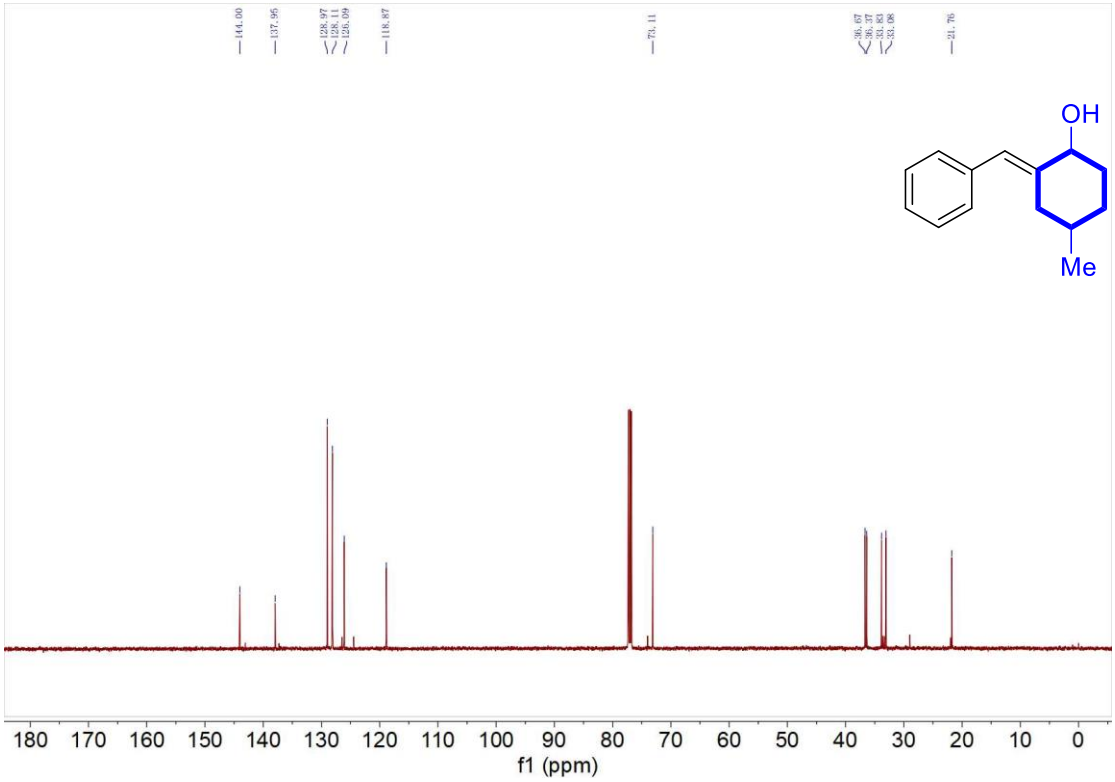
(*E*)-2-(cyclohexylmethylene)cyclopentan-1-ol (4j)



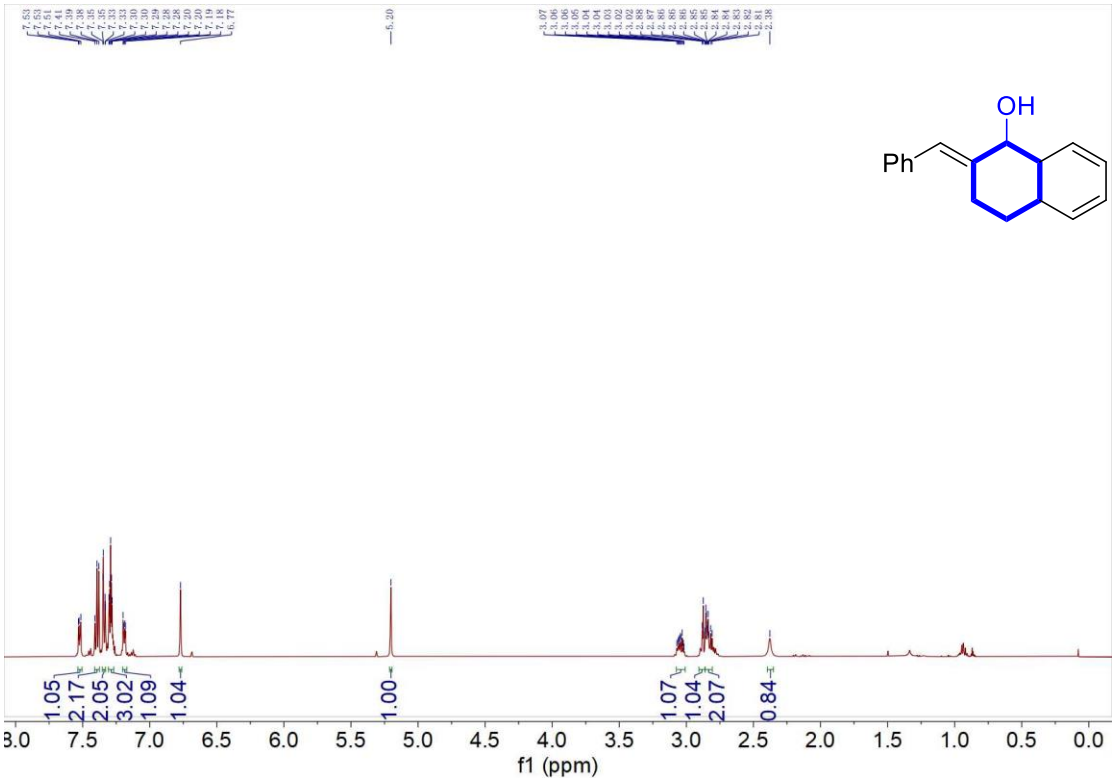


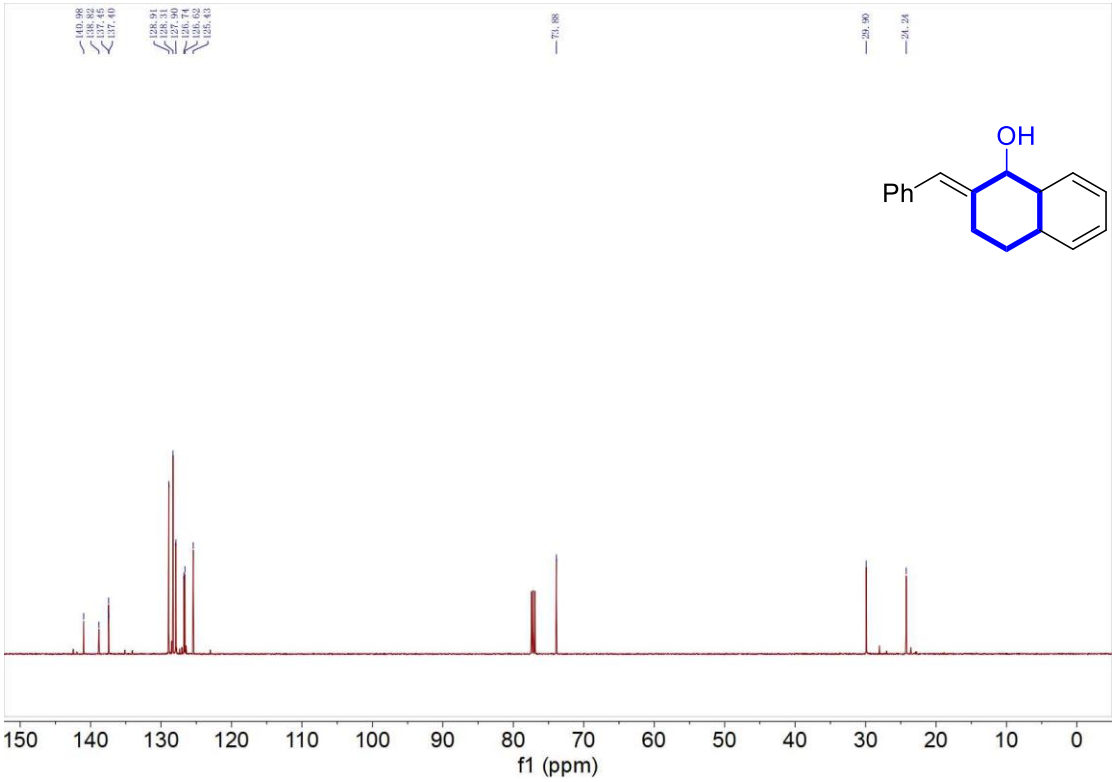
(*E*)-2-benzylidene-4-methylcyclohexan-1-ol (4k)



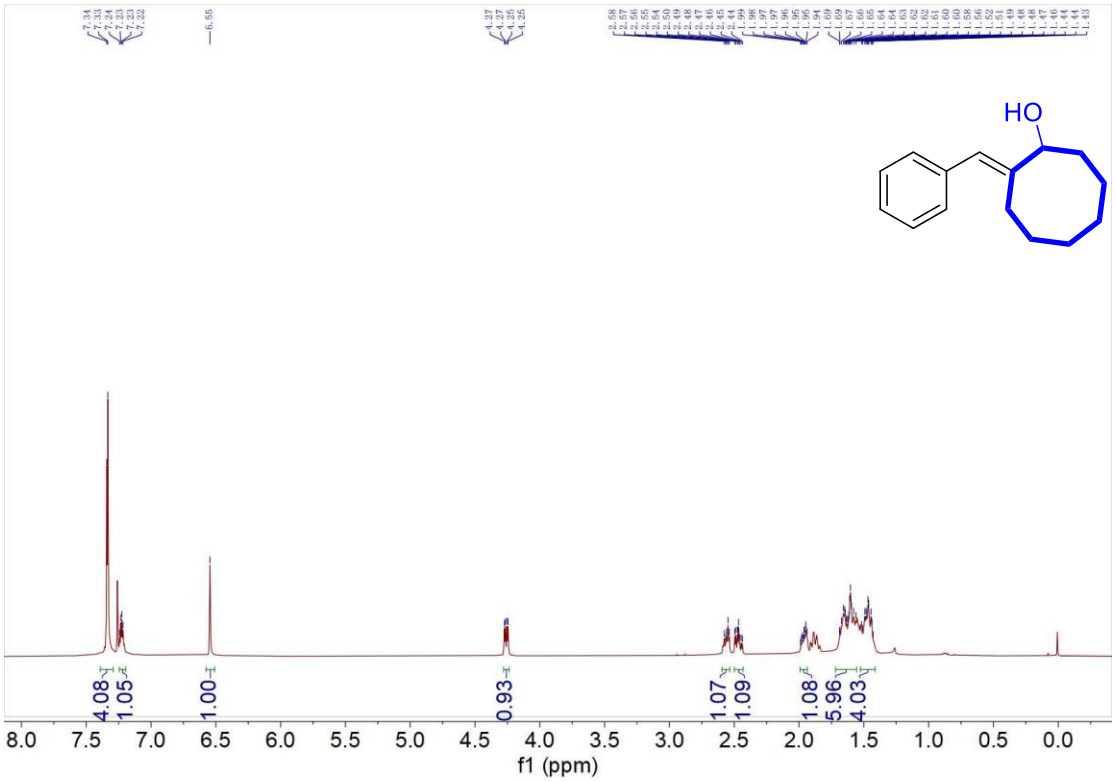


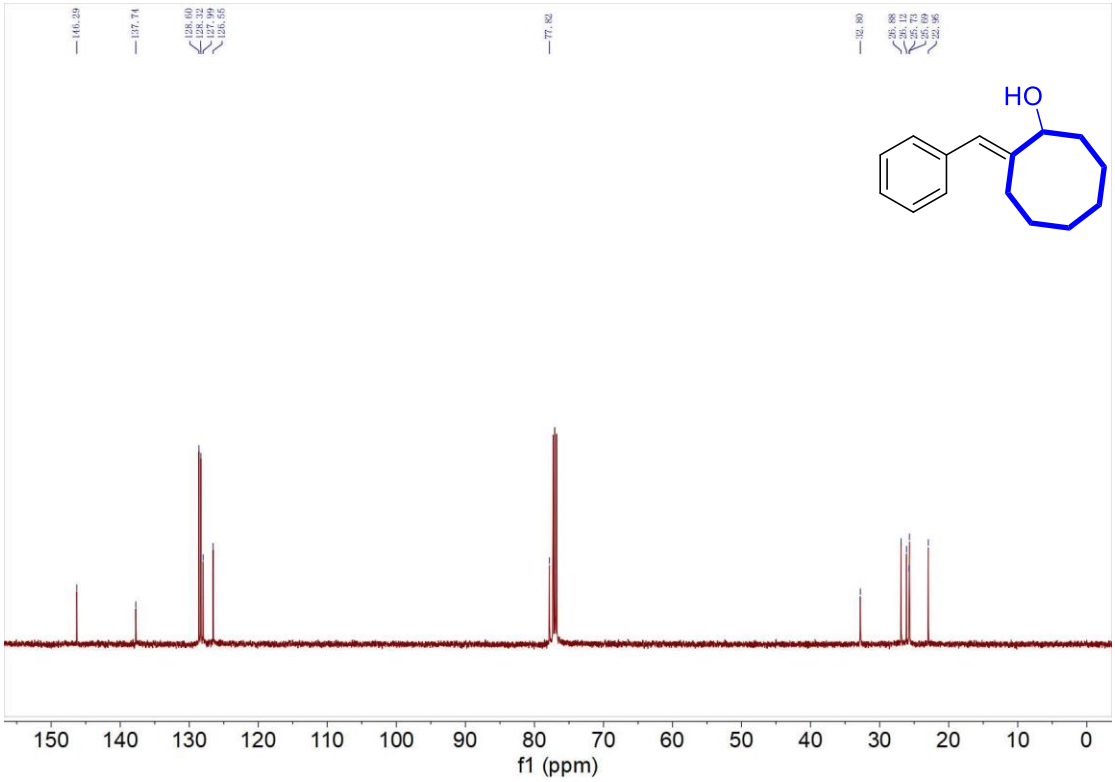
(*E*)-2-benzylidene-1,2,3,4-tetrahydronaphthalen-1-ol (4l)



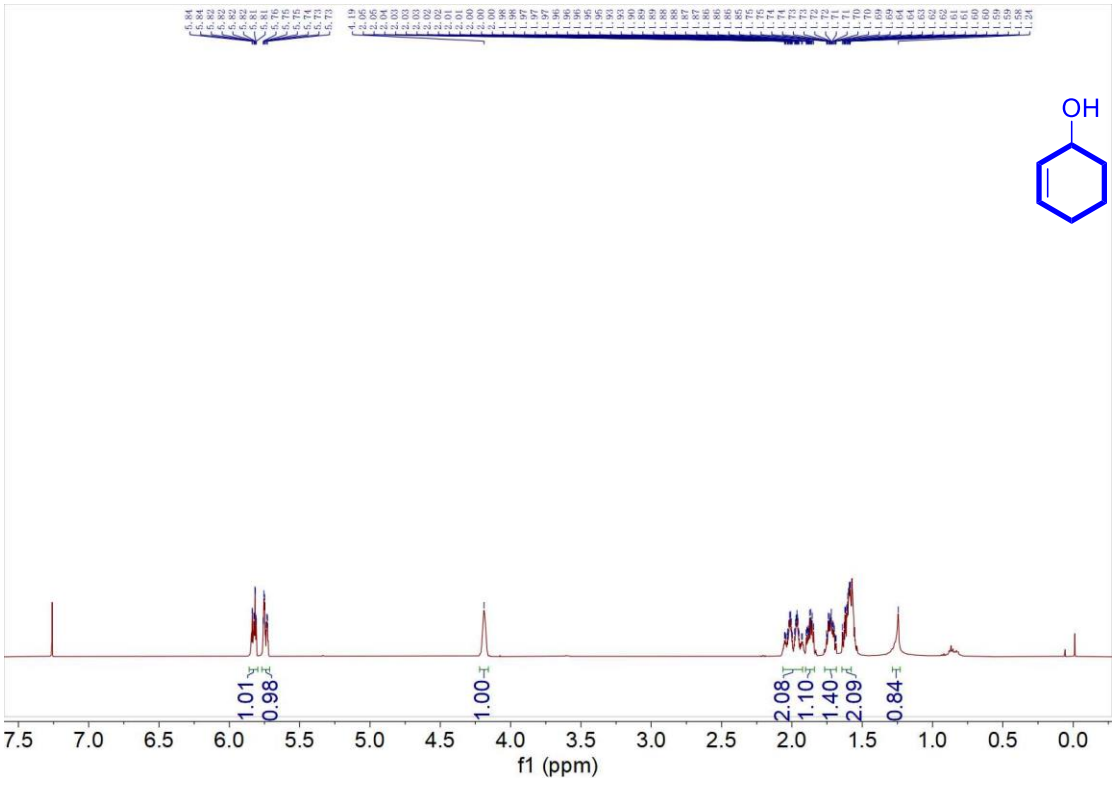


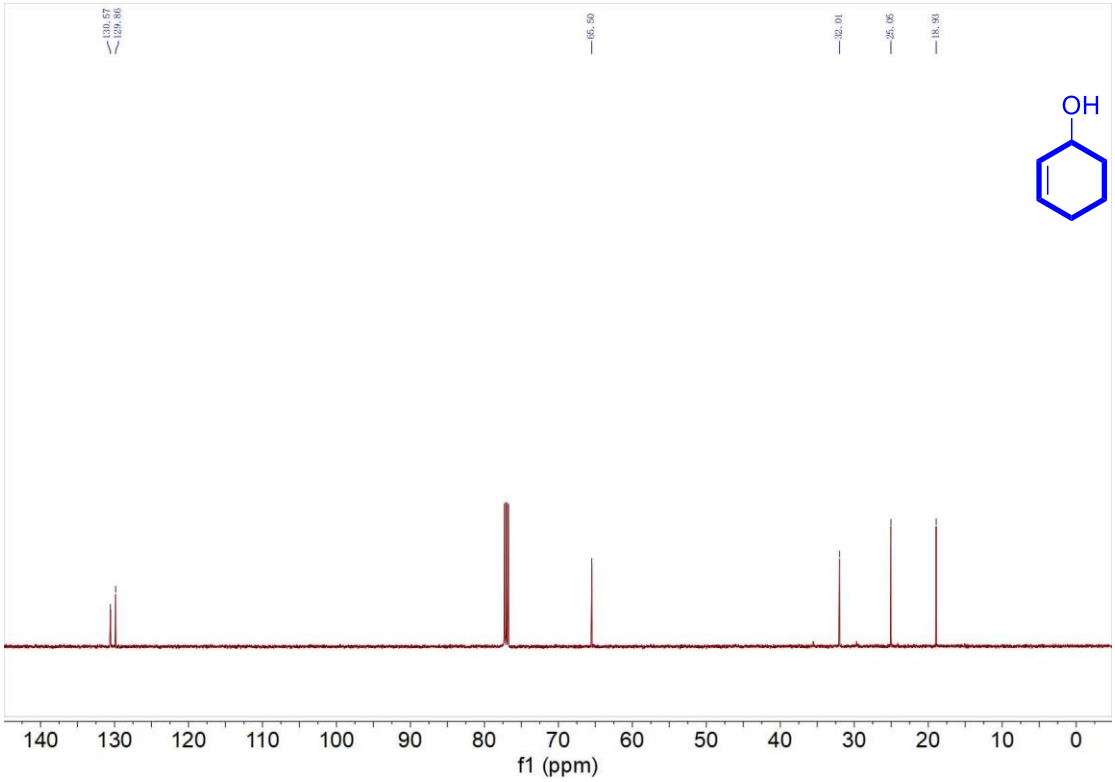
(*E*)-2-benzylidenecyclooctan-1-ol (4m)



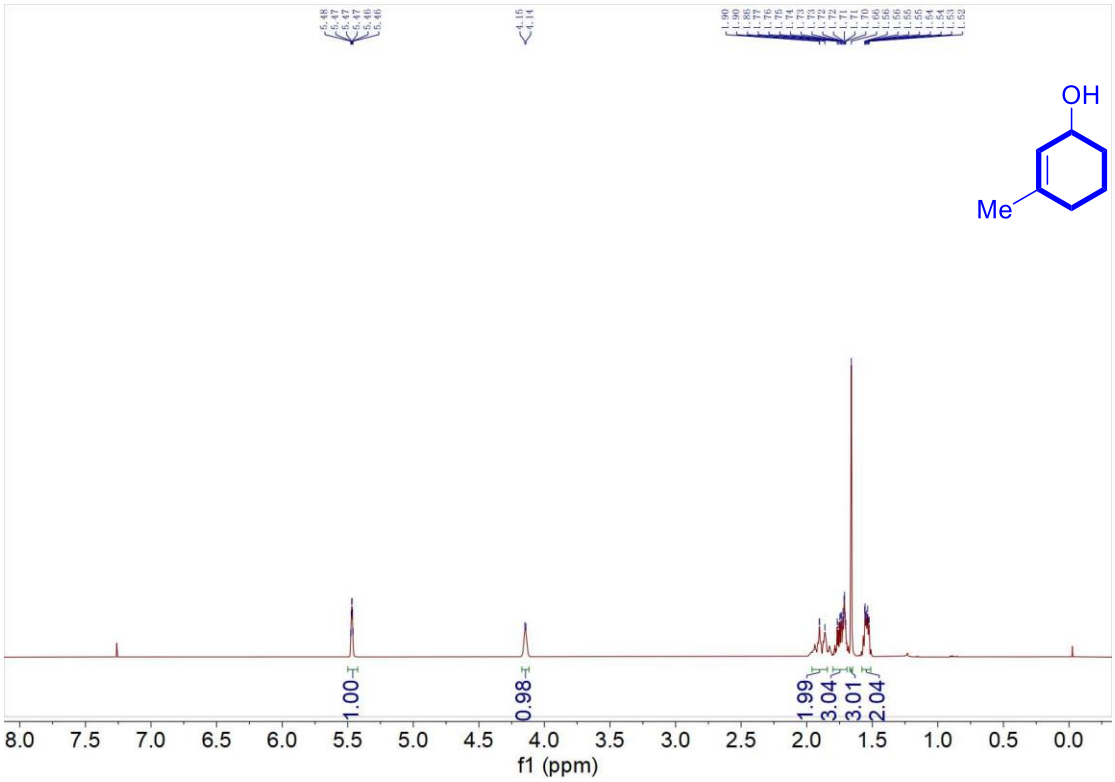


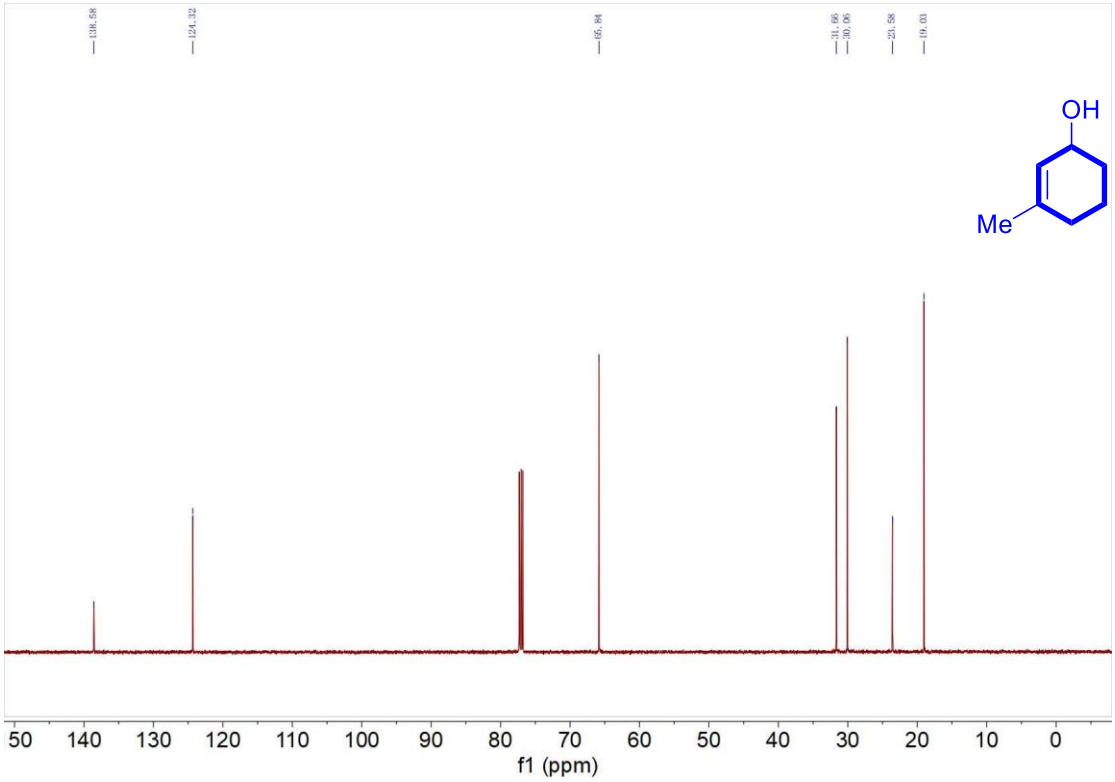
cyclohex-2-en-1-ol (4n)



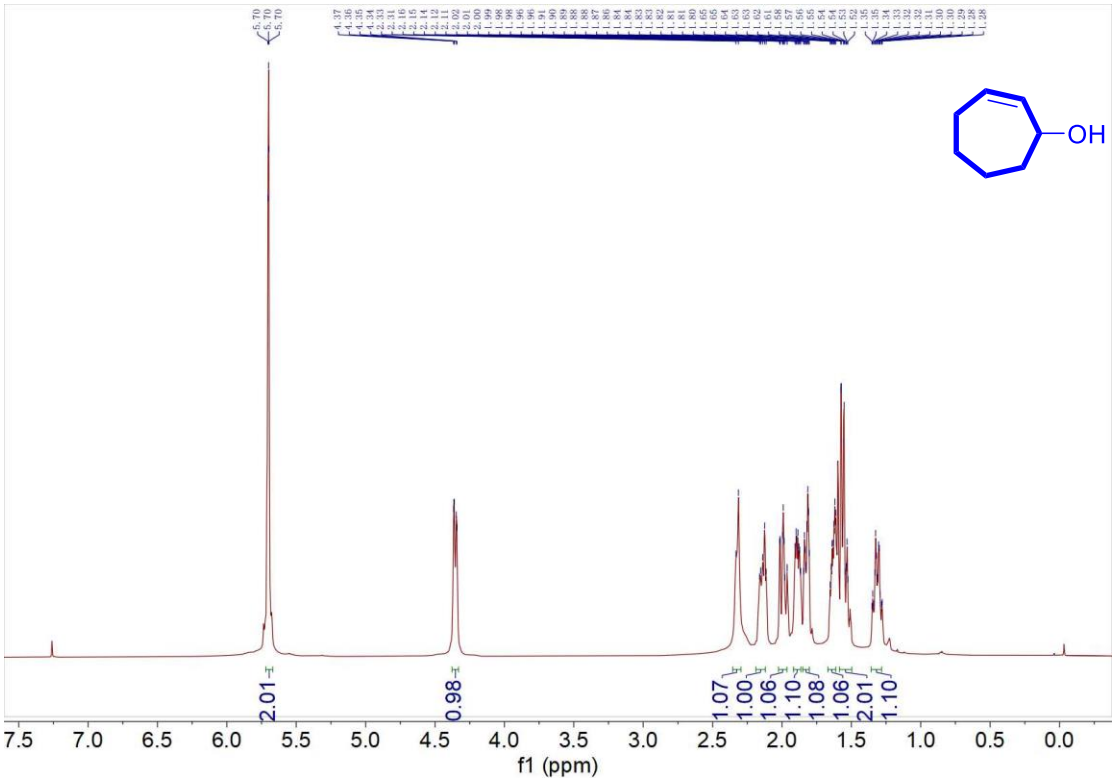


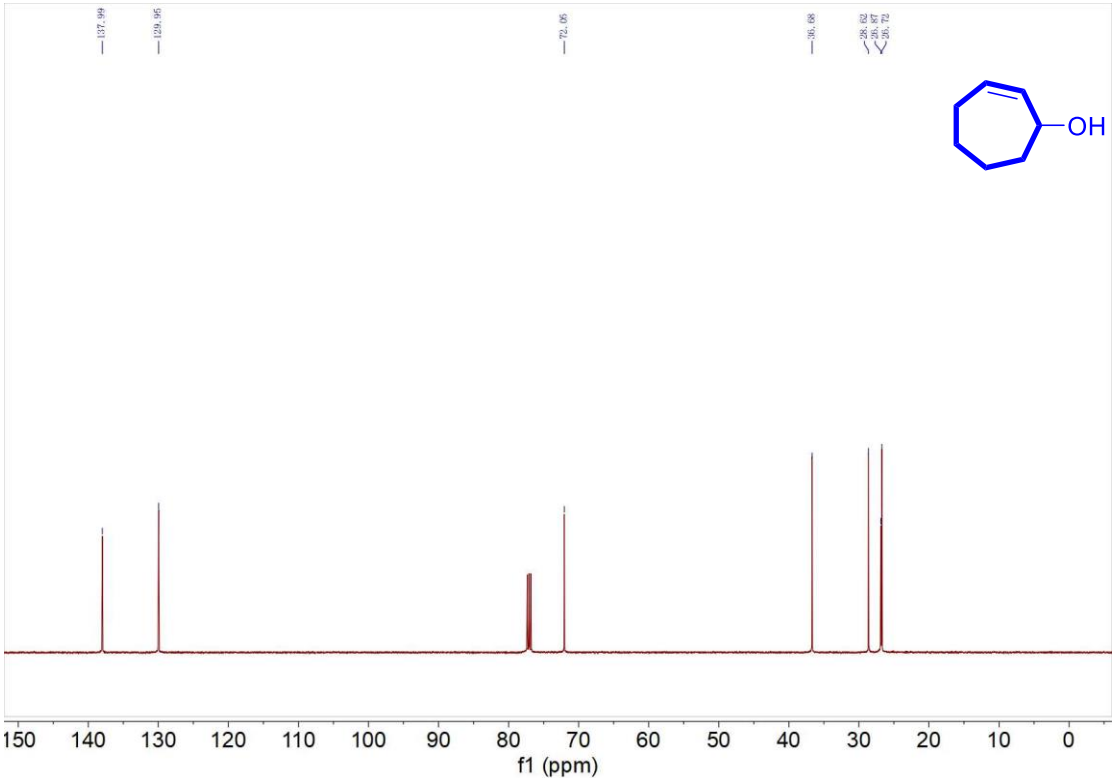
3-methylcyclohex-2-en-1-ol (4o)



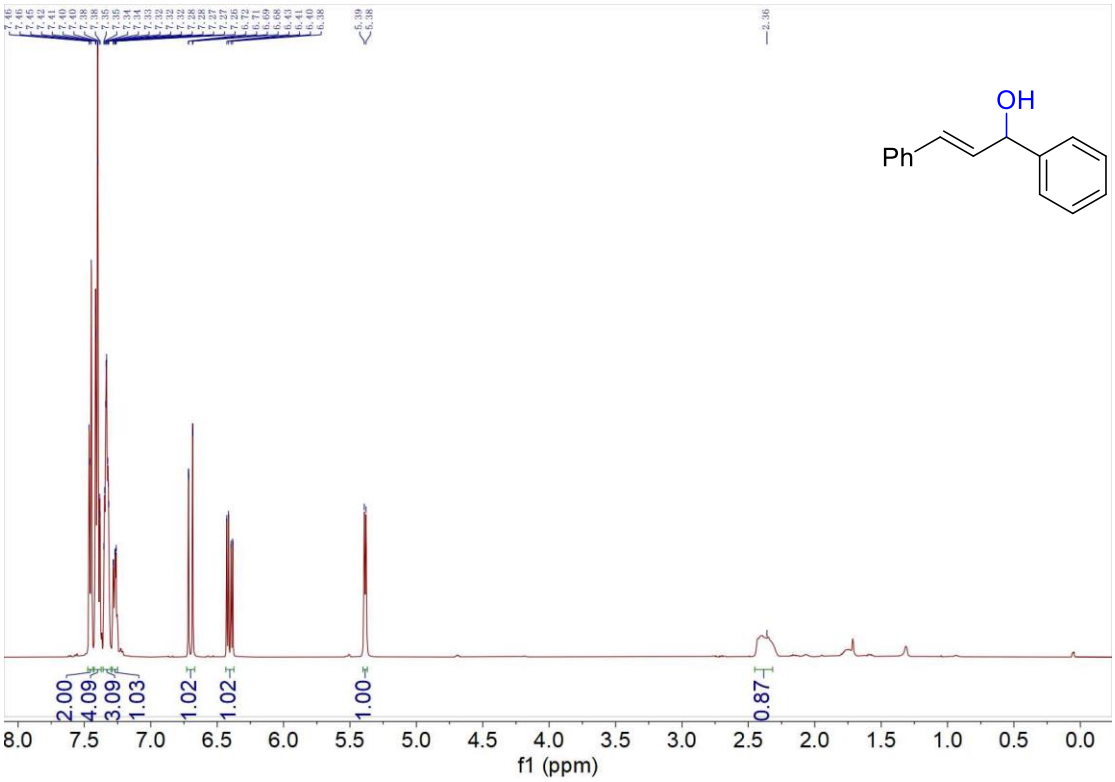


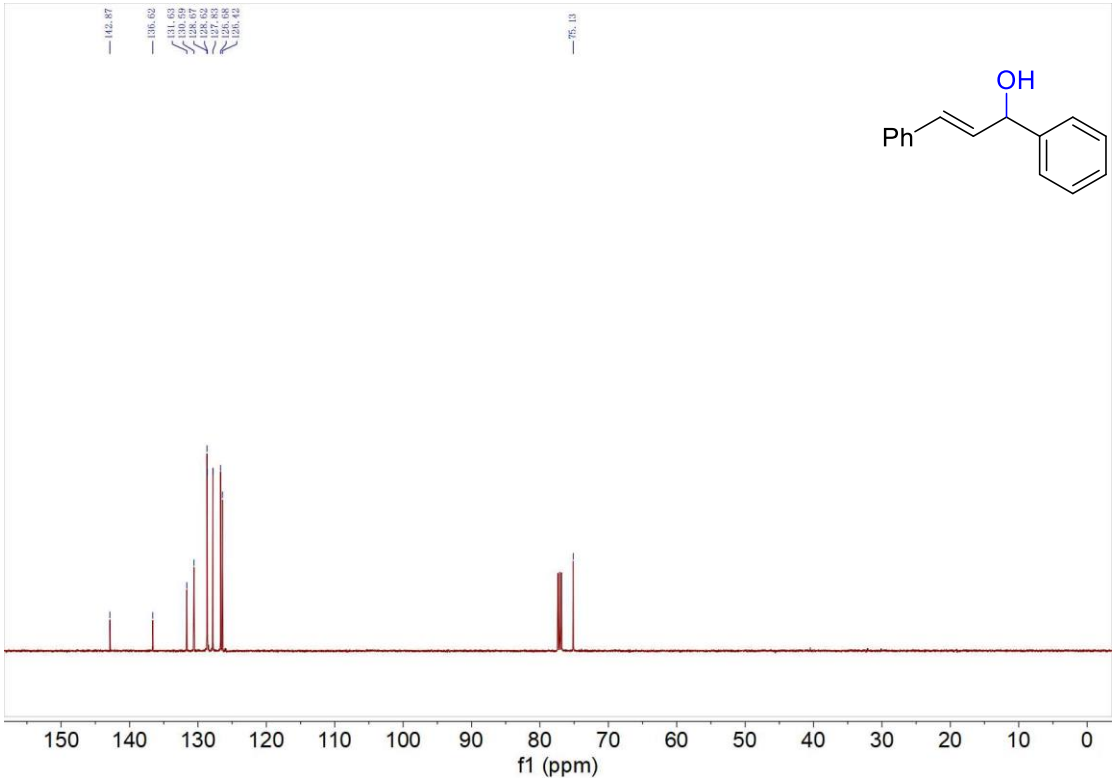
cyclohept-2-en-1-ol (4p)



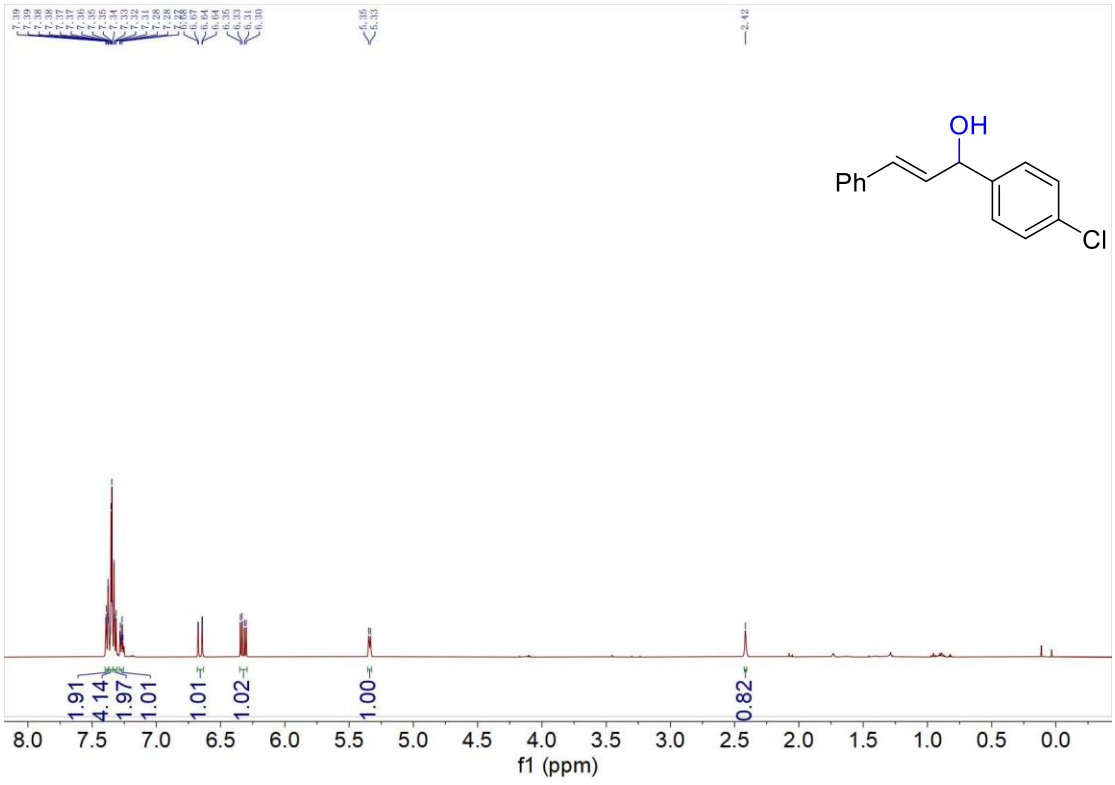


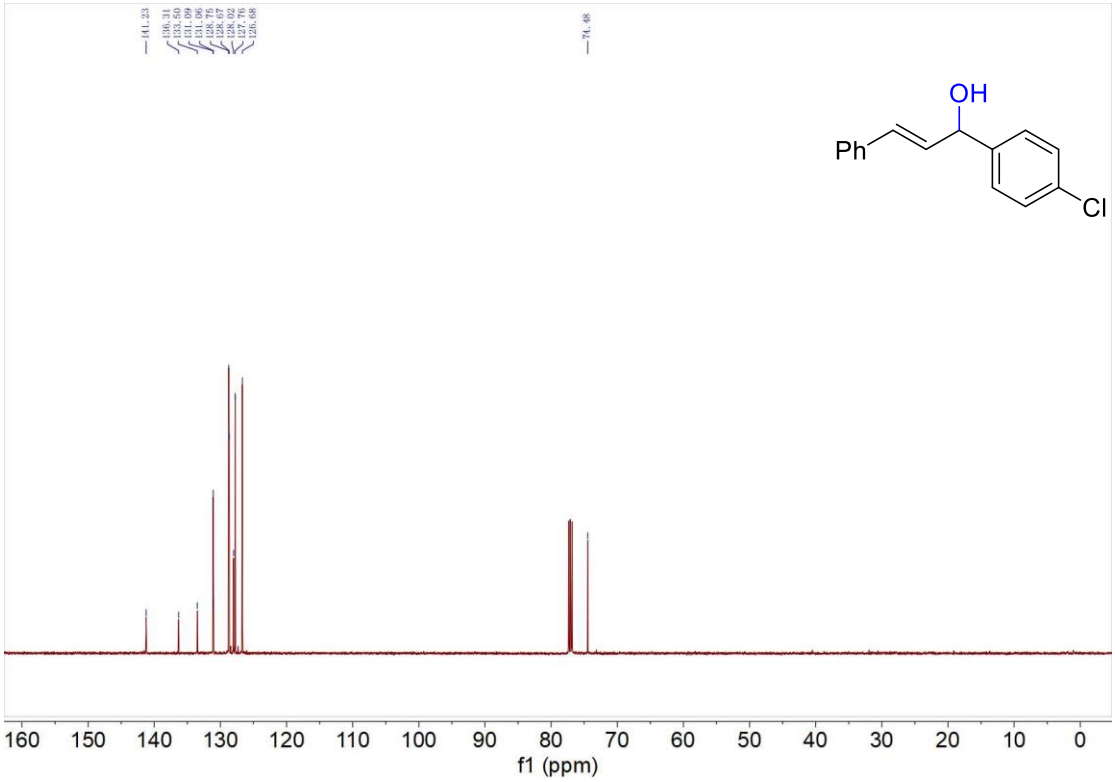
(*E*)-1,3-diphenylprop-2-en-1-ol (6a)



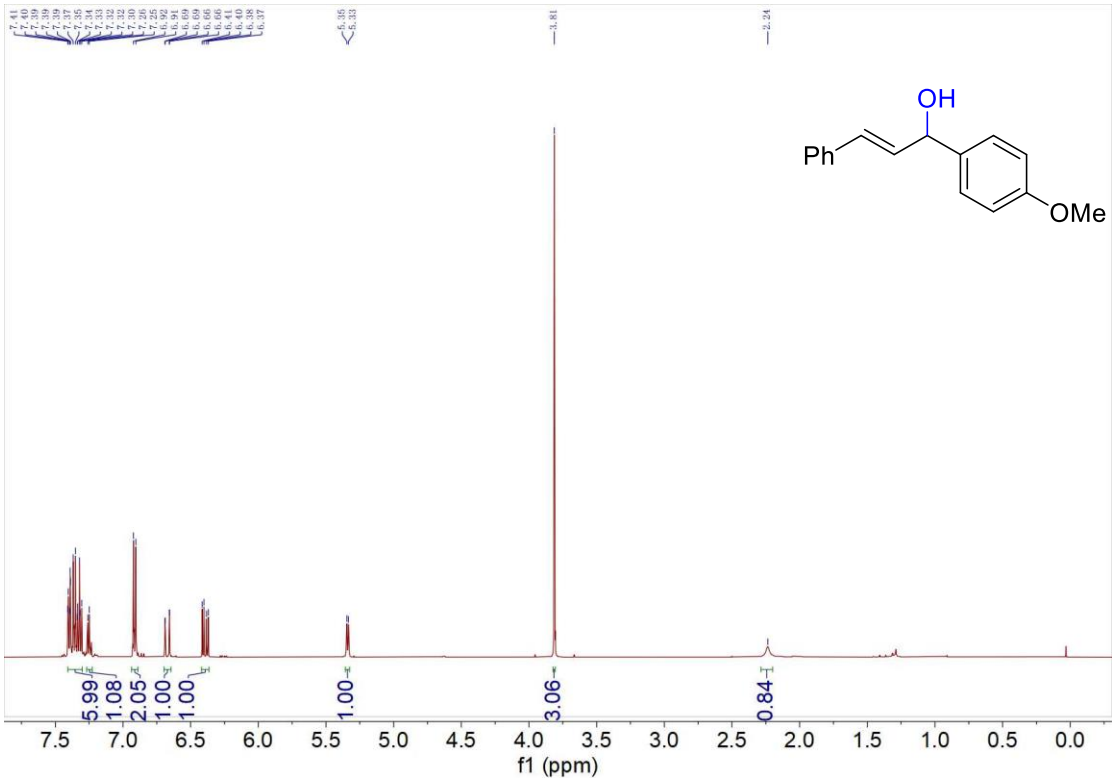


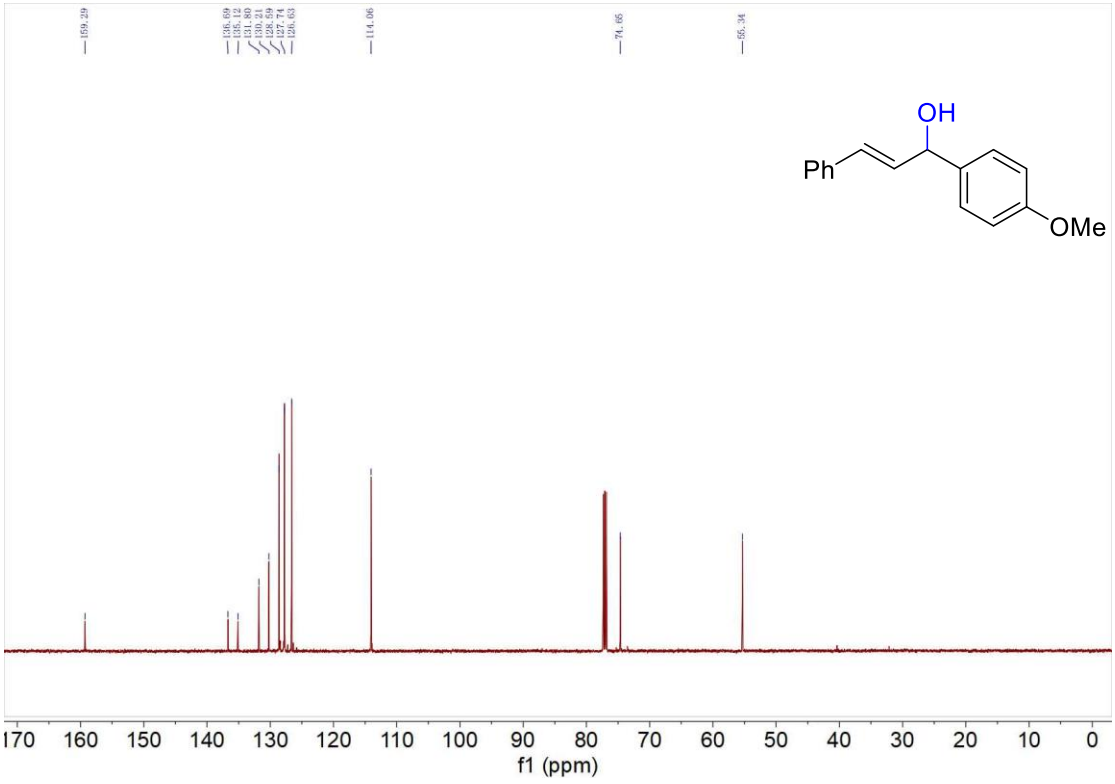
(*E*)-1-(4-chlorophenyl)-3-phenylprop-2-en-1-ol (6b)



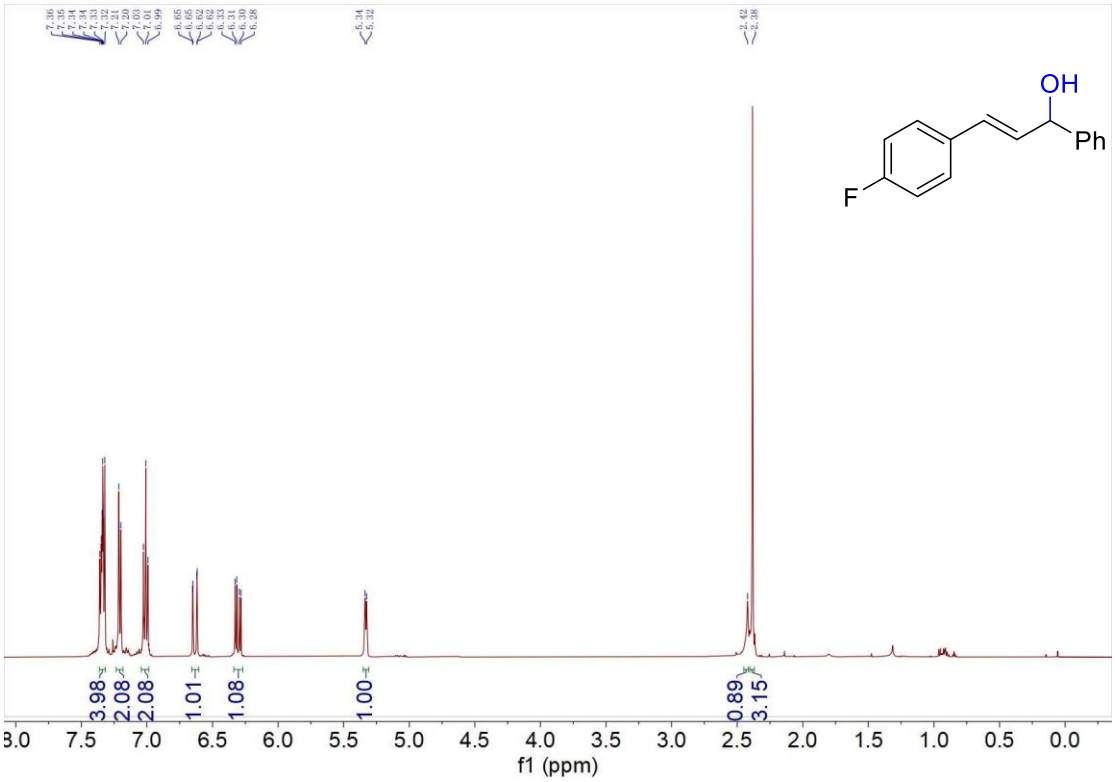


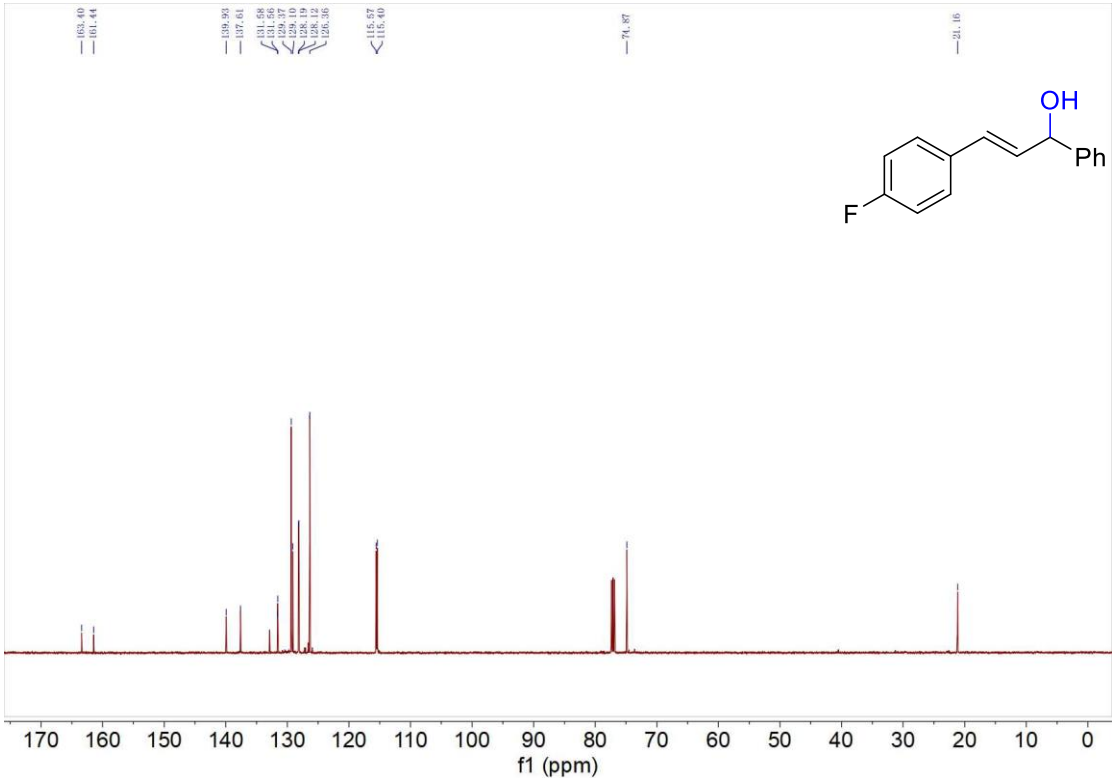
(*E*)-1-(4-methoxyphenyl)-3-phenylprop-2-en-1-ol (6c)



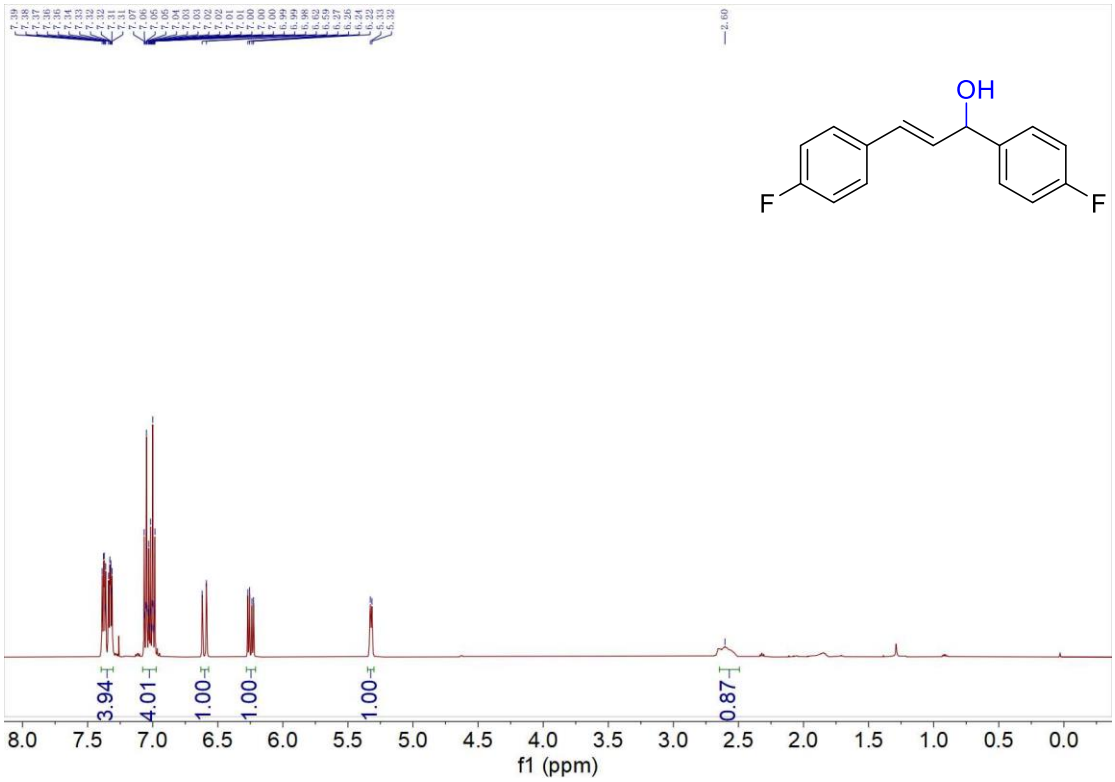


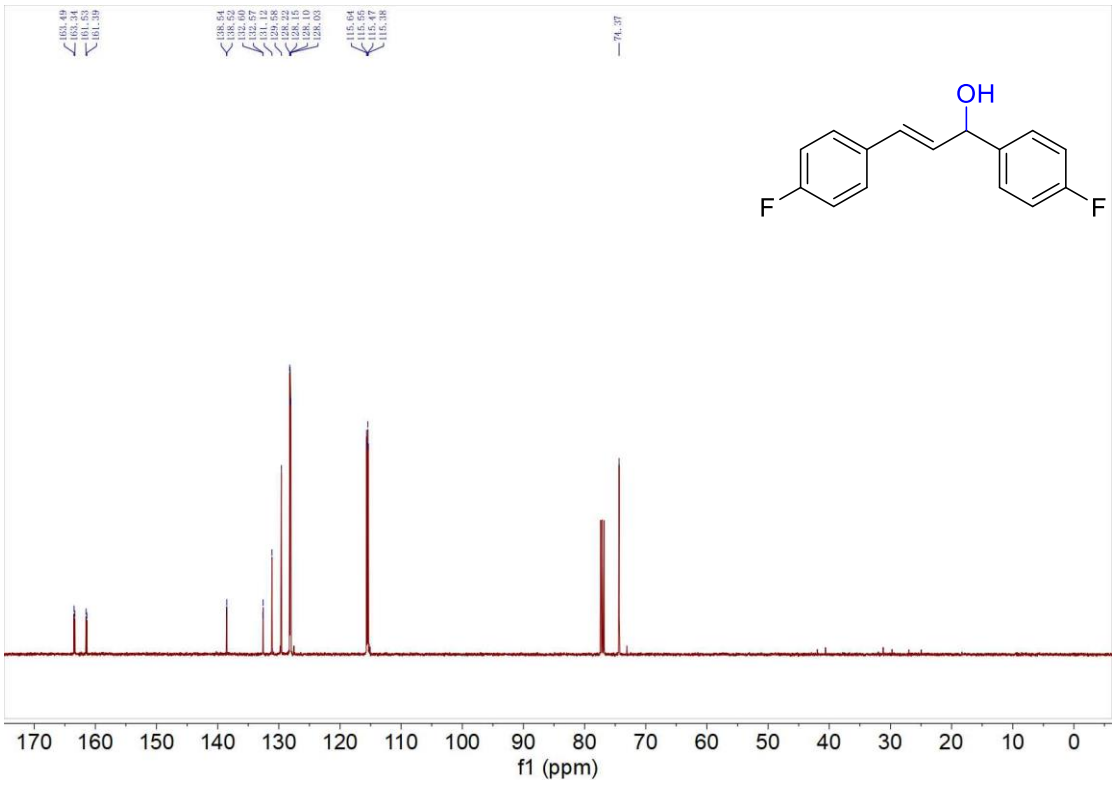
(*E*)-3-(4-fluorophenyl)-1-phenylprop-2-en-1-ol (6d)



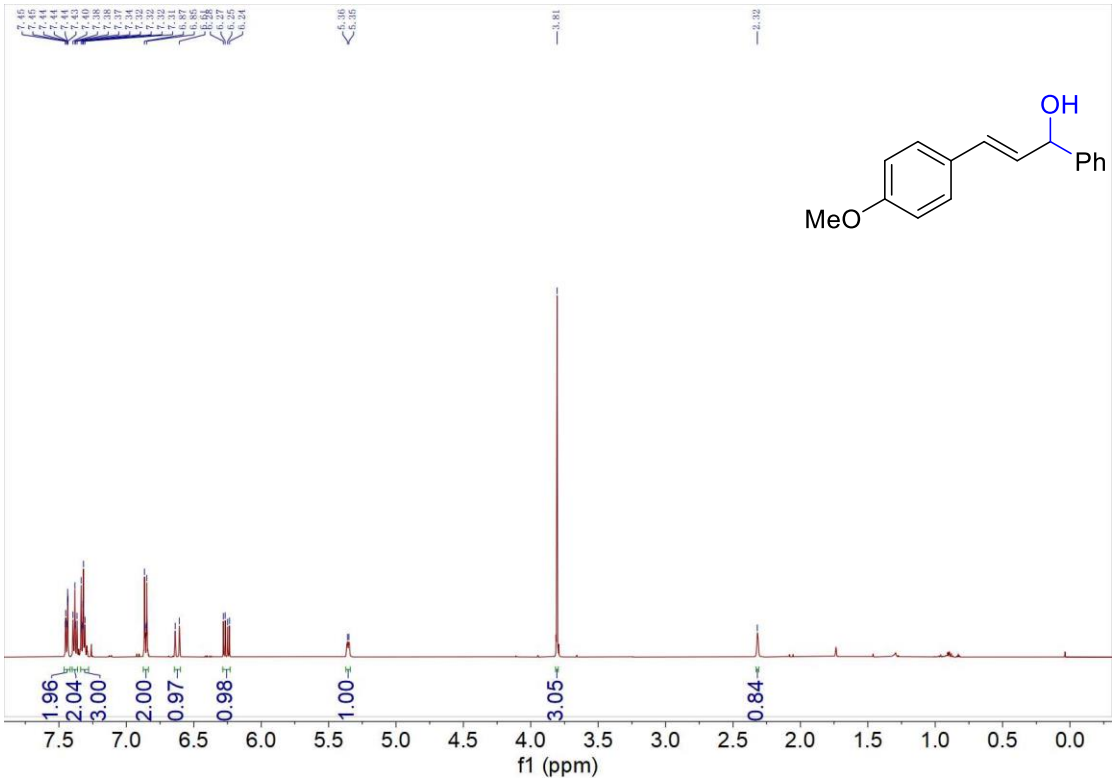


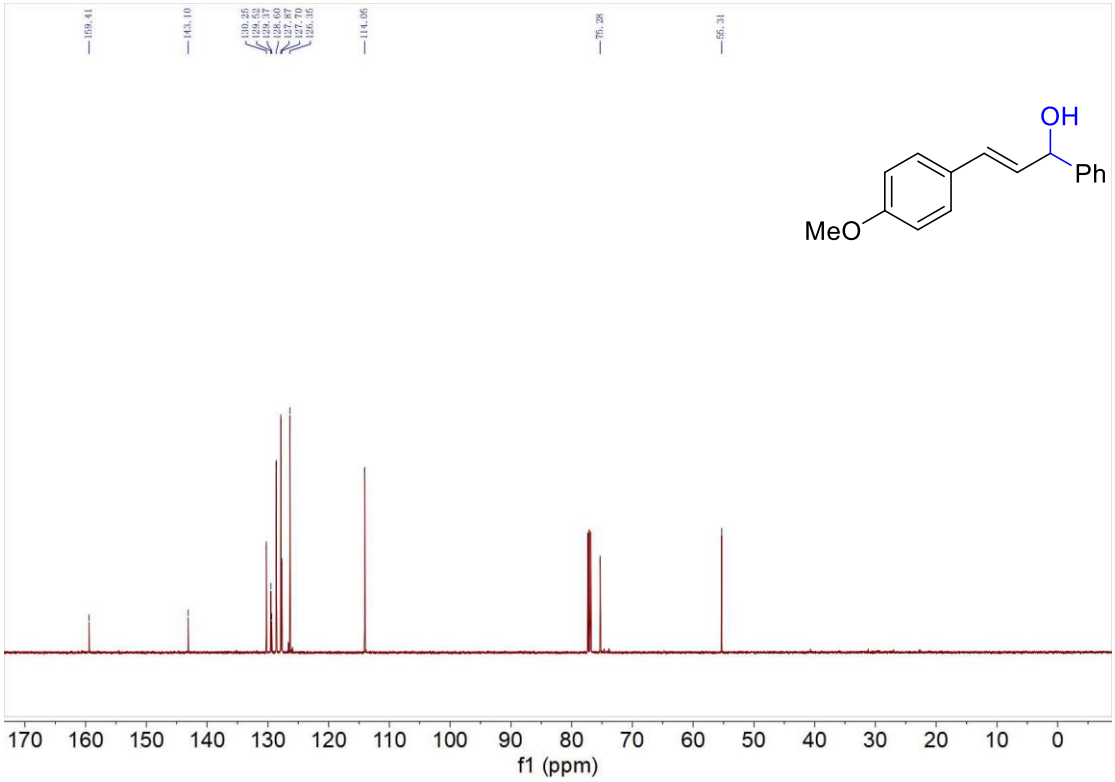
(*E*)-1,3-bis(4-fluorophenyl)prop-2-en-1-ol (6e)





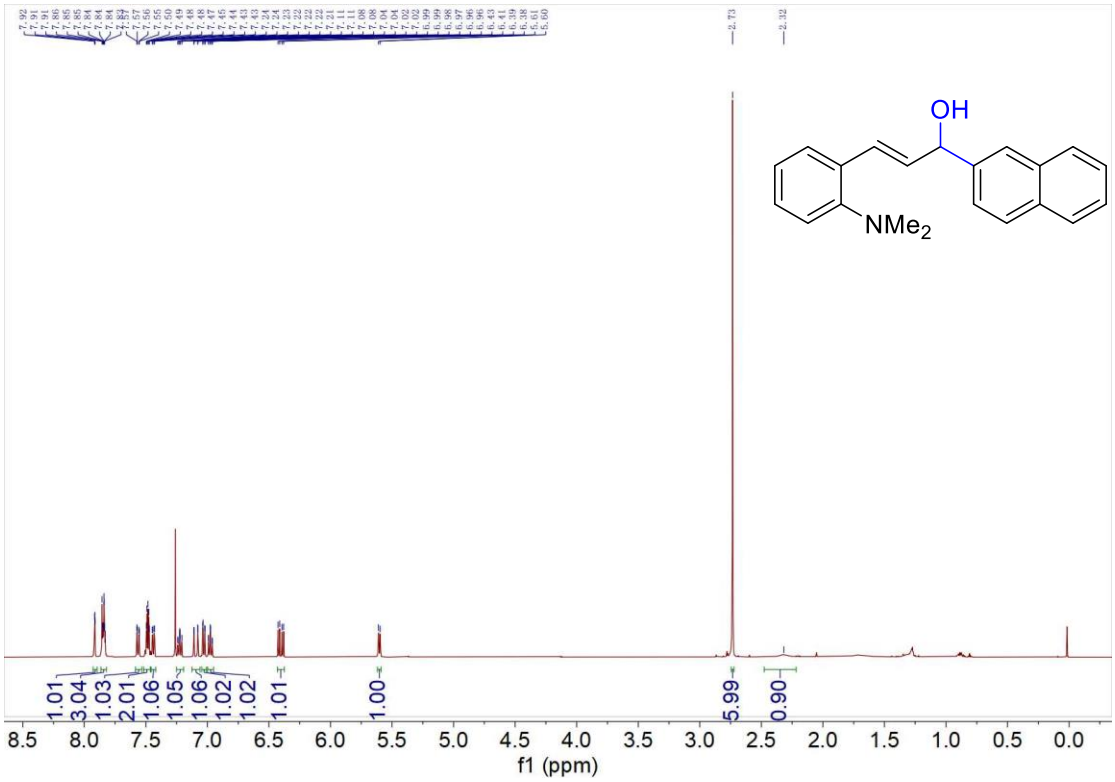
(*E*)-3-(4-methoxyphenyl)-1-phenylprop-2-en-1-ol (6f)

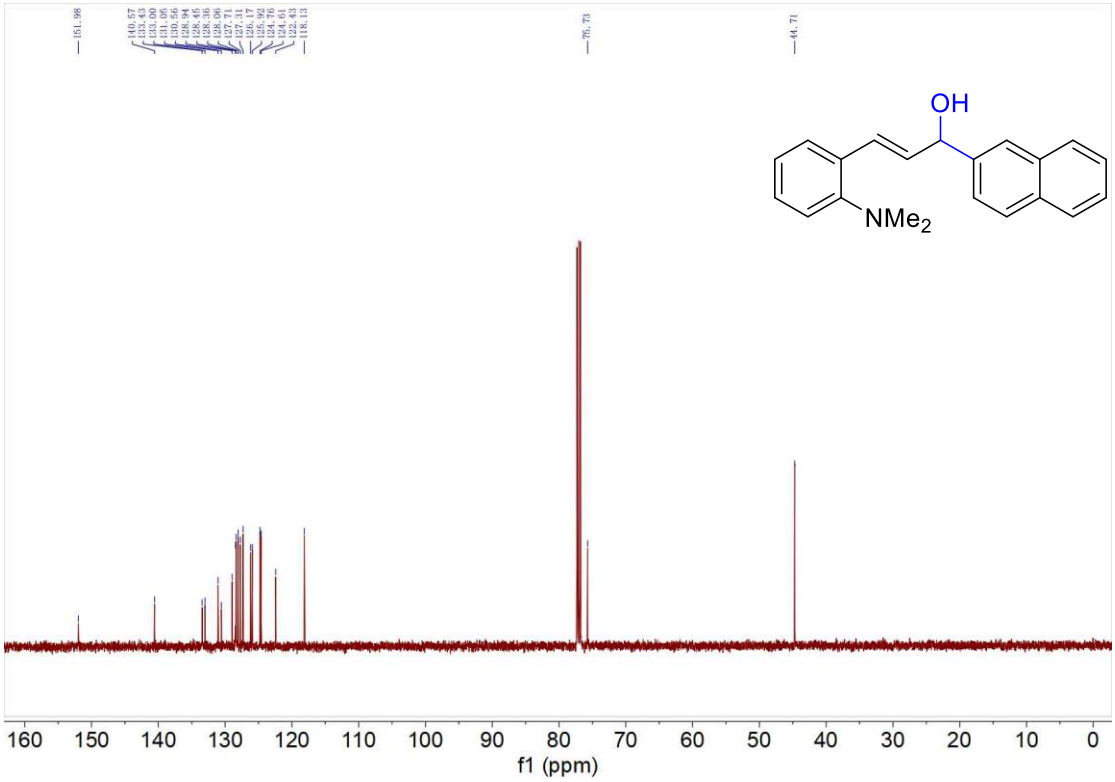




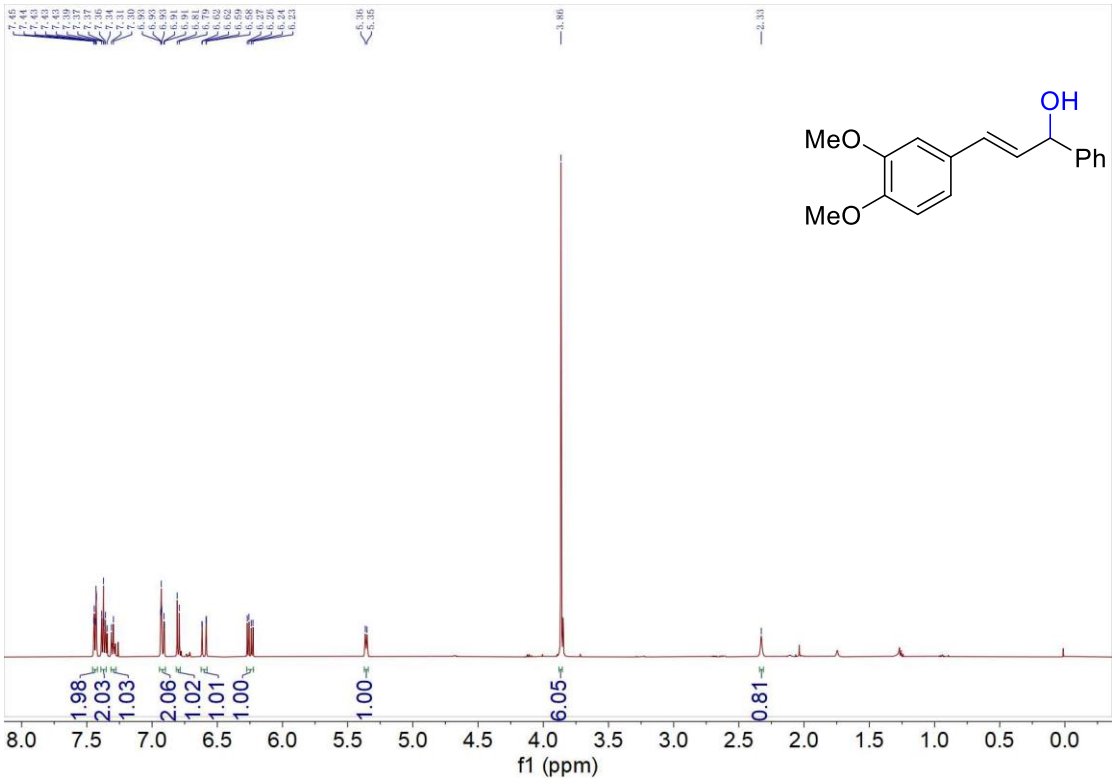
(*E*)-3-(2-(dimethylamino)phenyl)-1-(naphthalen-2-yl)prop-2-en-1-ol

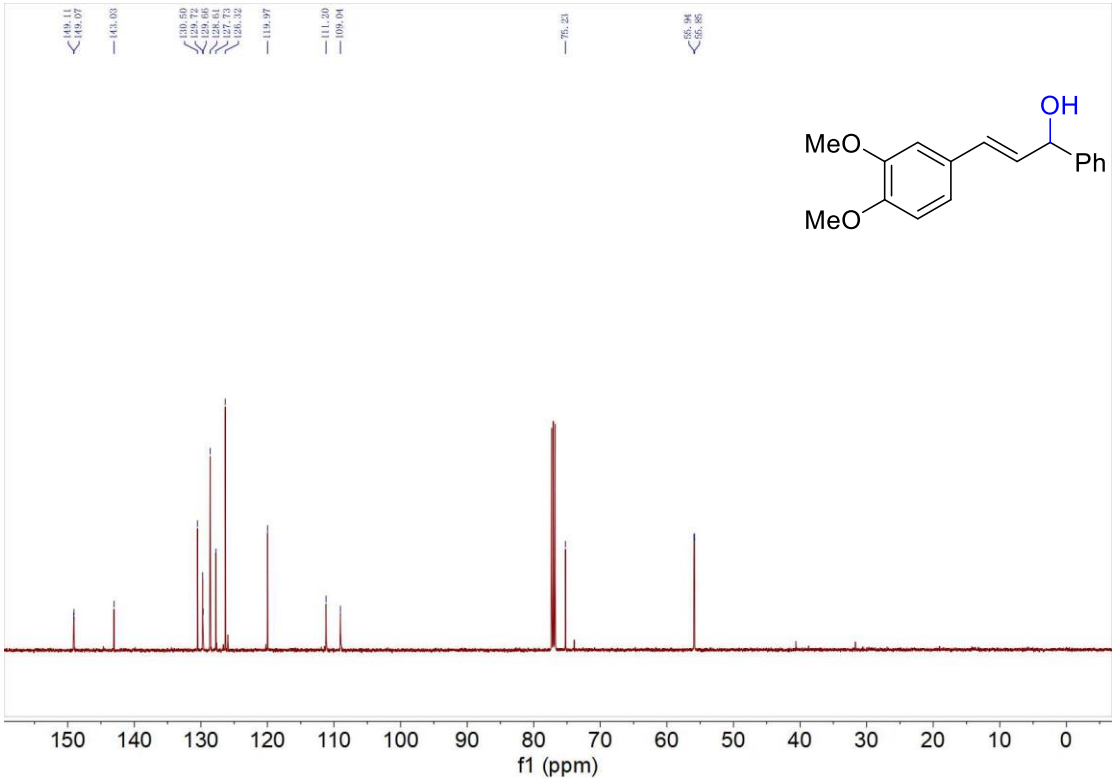
(6g)



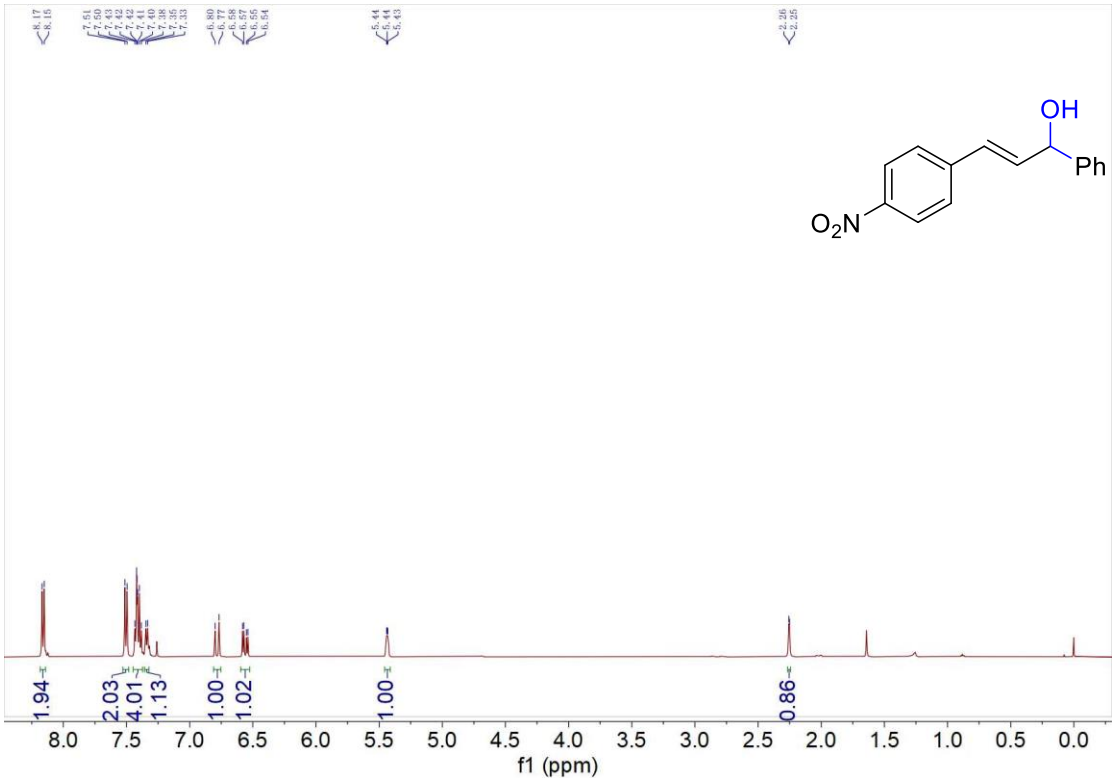


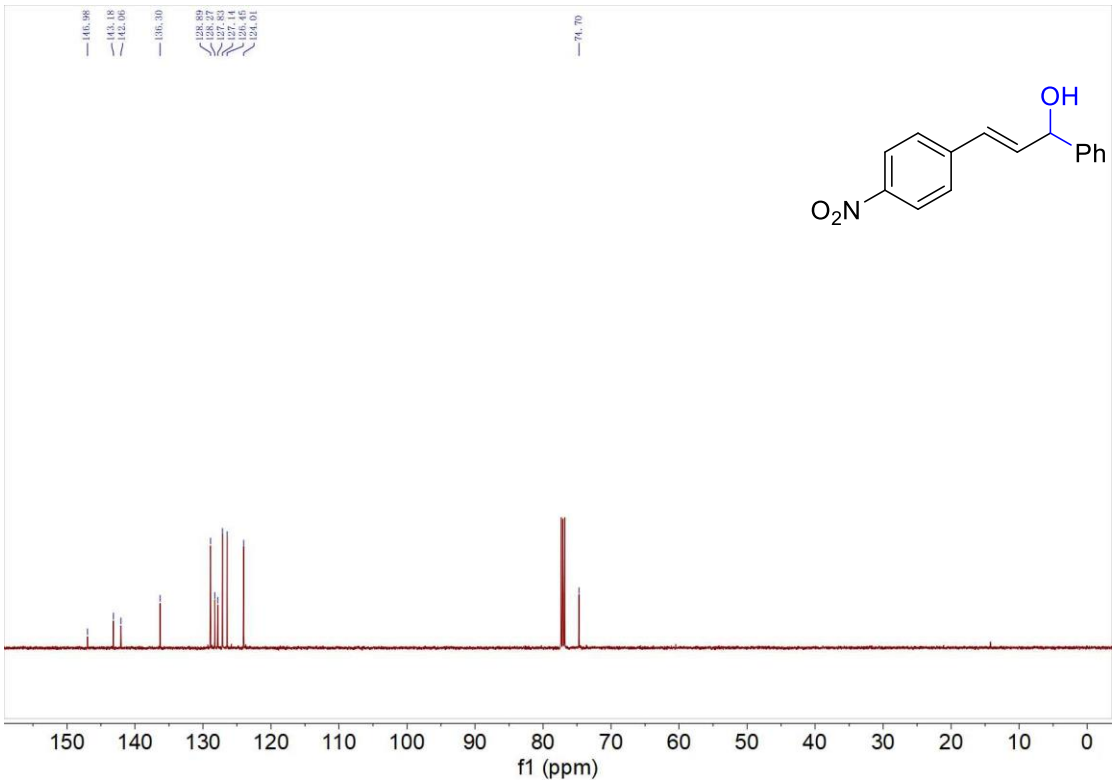
(*E*)-3-(3,4-dimethoxyphenyl)-1-phenylprop-2-en-1-ol (6h)



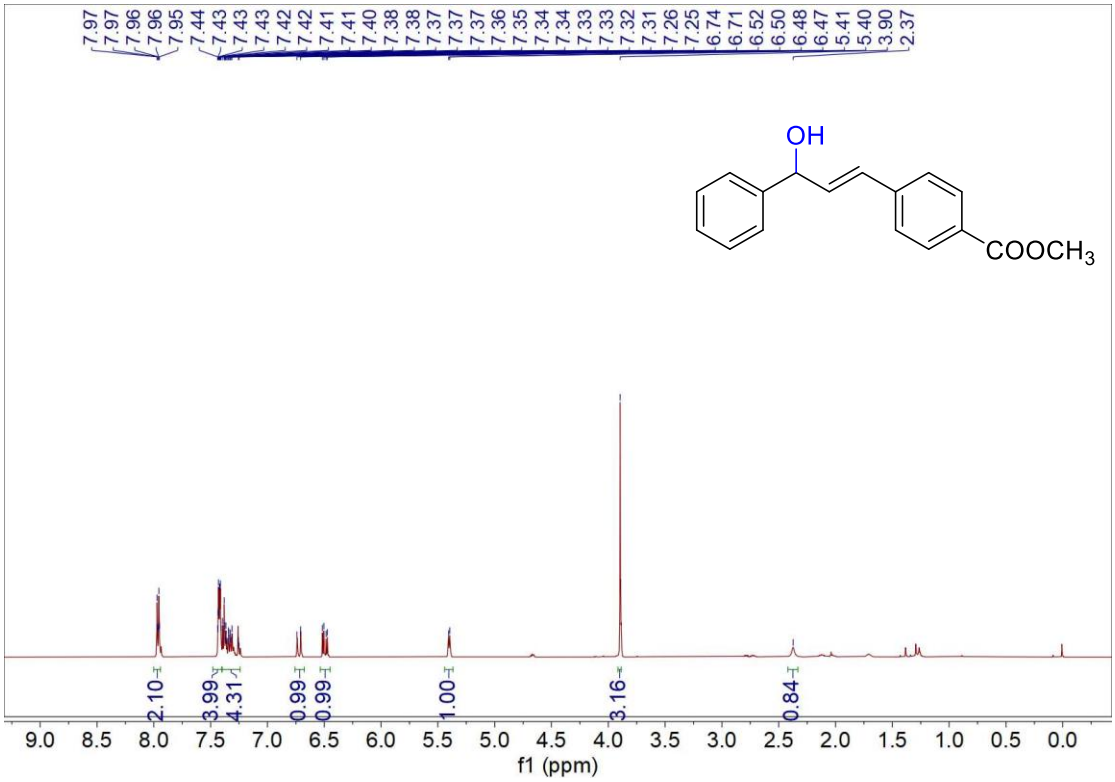


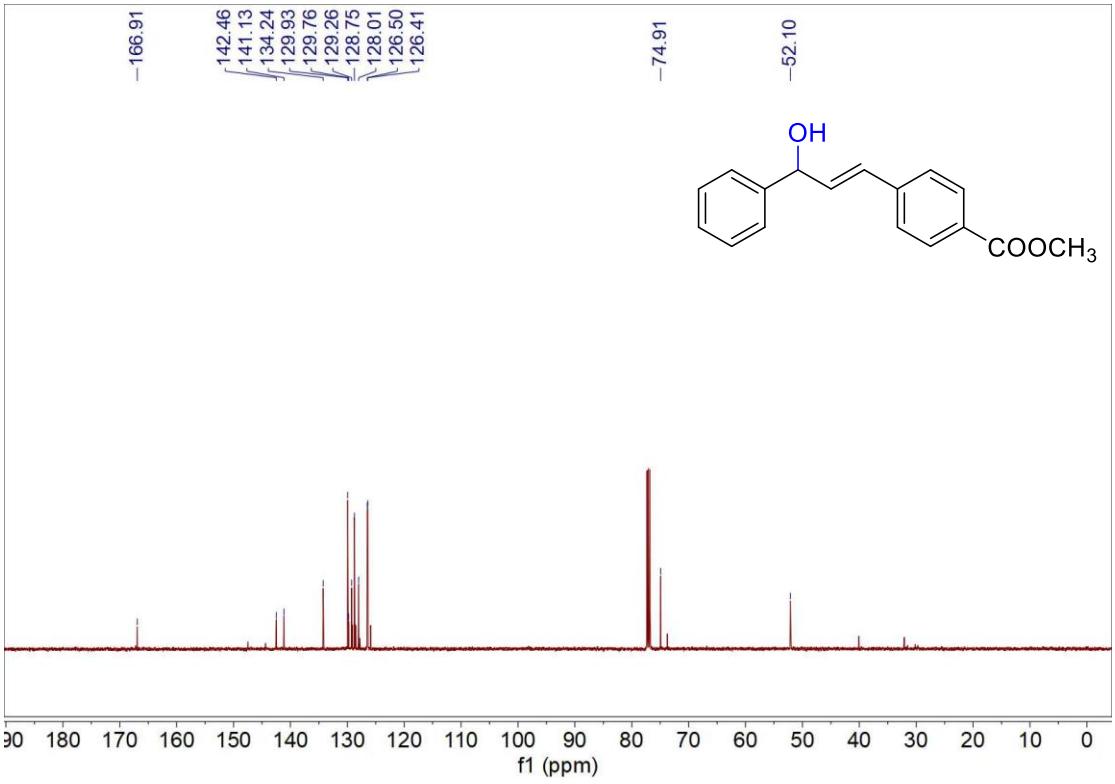
(*E*)-3-(4-nitrophenyl)-1-phenylprop-2-en-1-ol (6i)



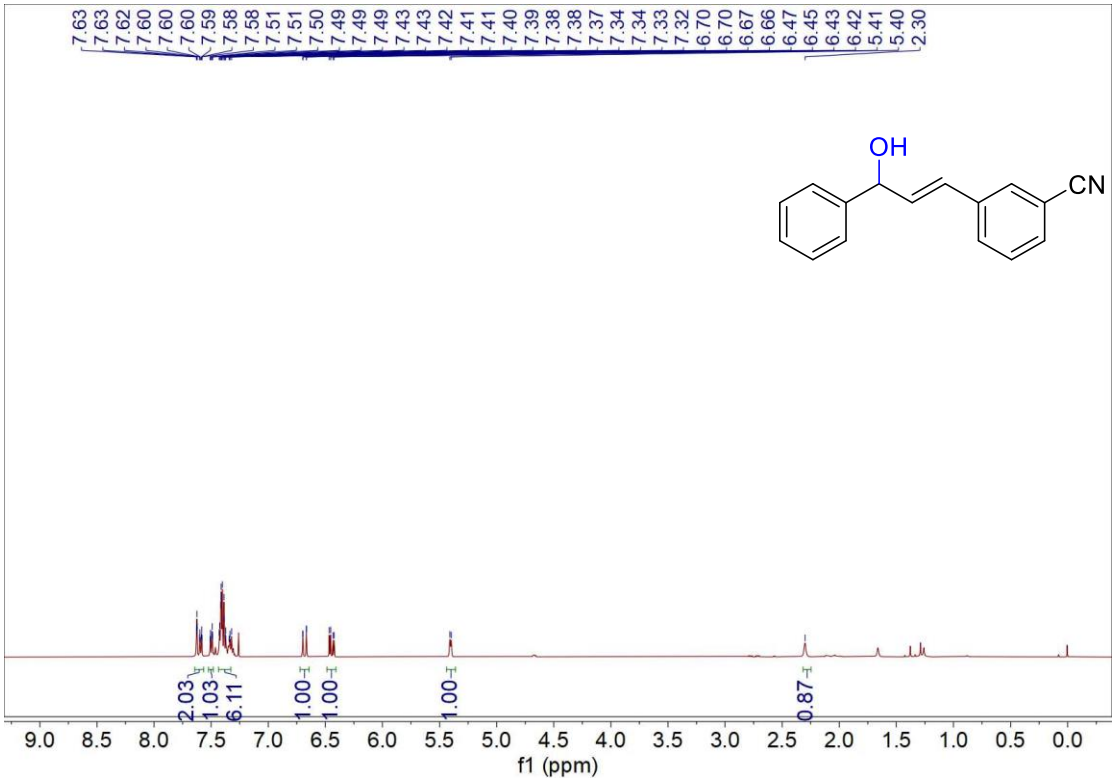


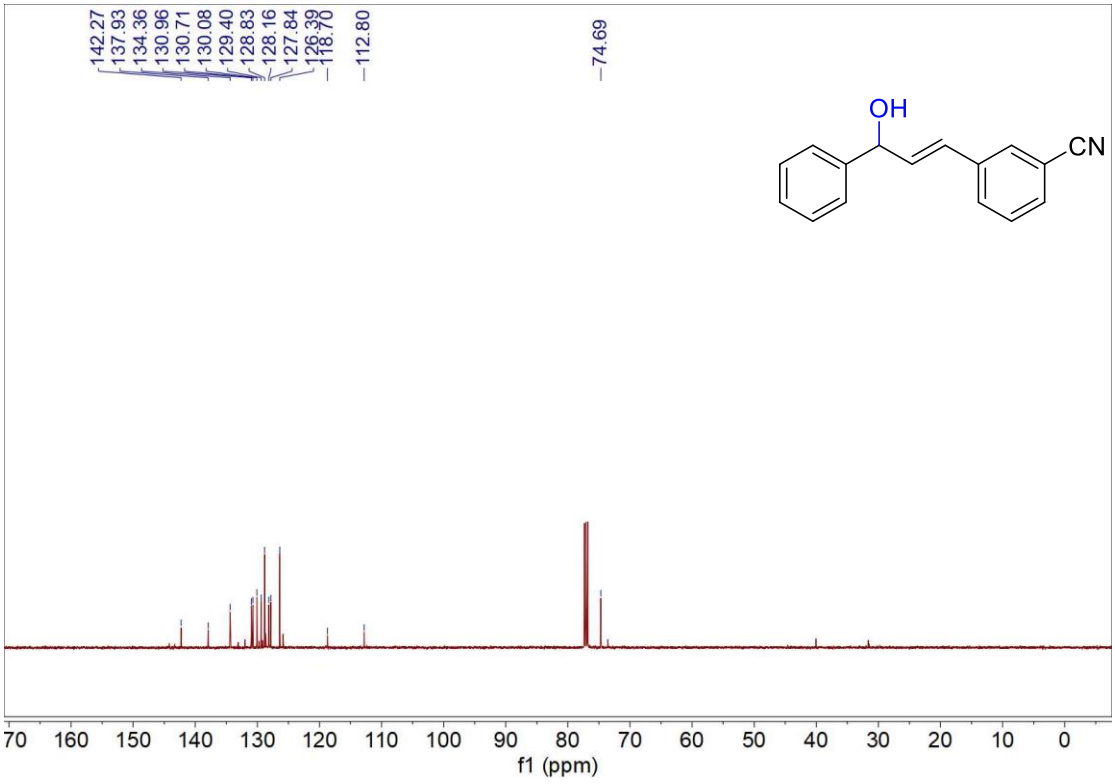
methyl (E)-4-(3-hydroxy-3-phenylprop-1-en-1-yl)benzoate (6j)



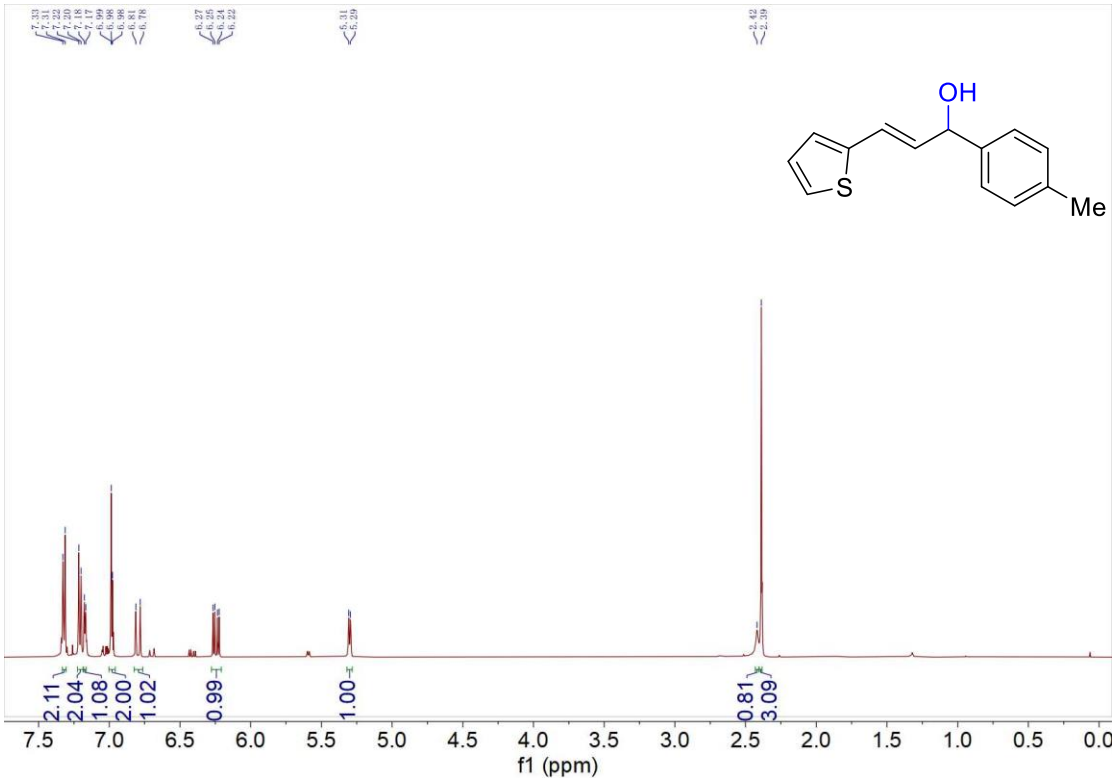


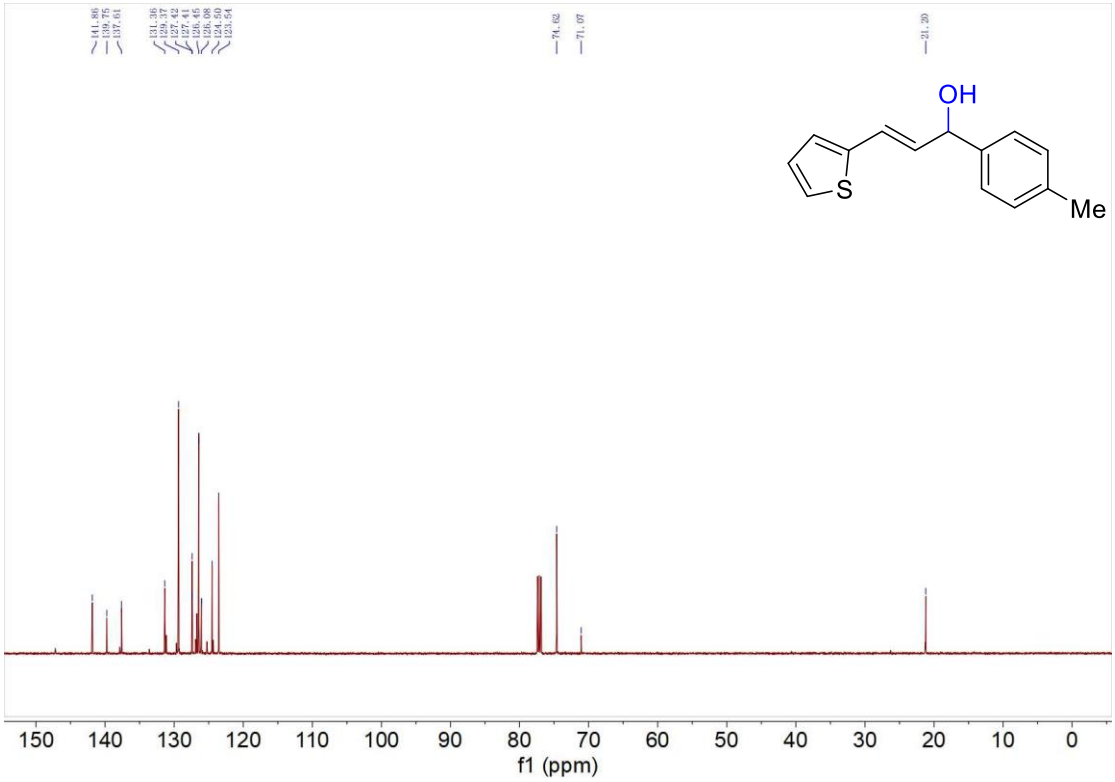
(E)-3-(3-hydroxy-3-phenylprop-1-en-1-yl)benzonitrile (6k)



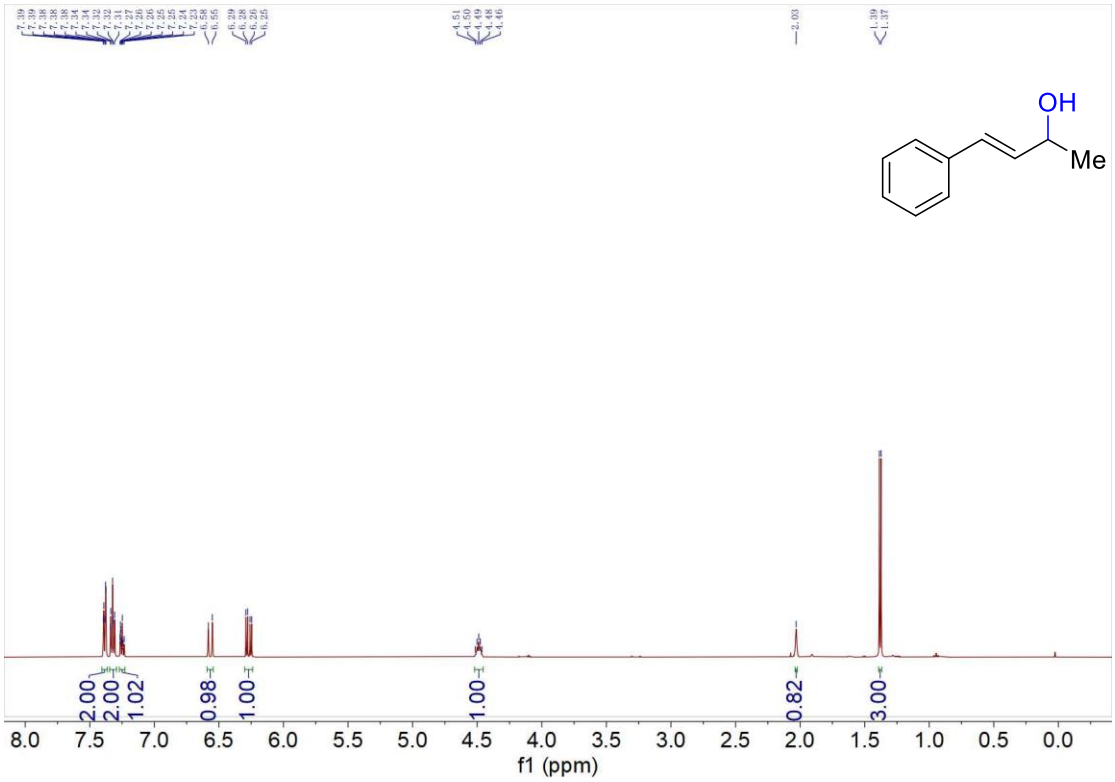


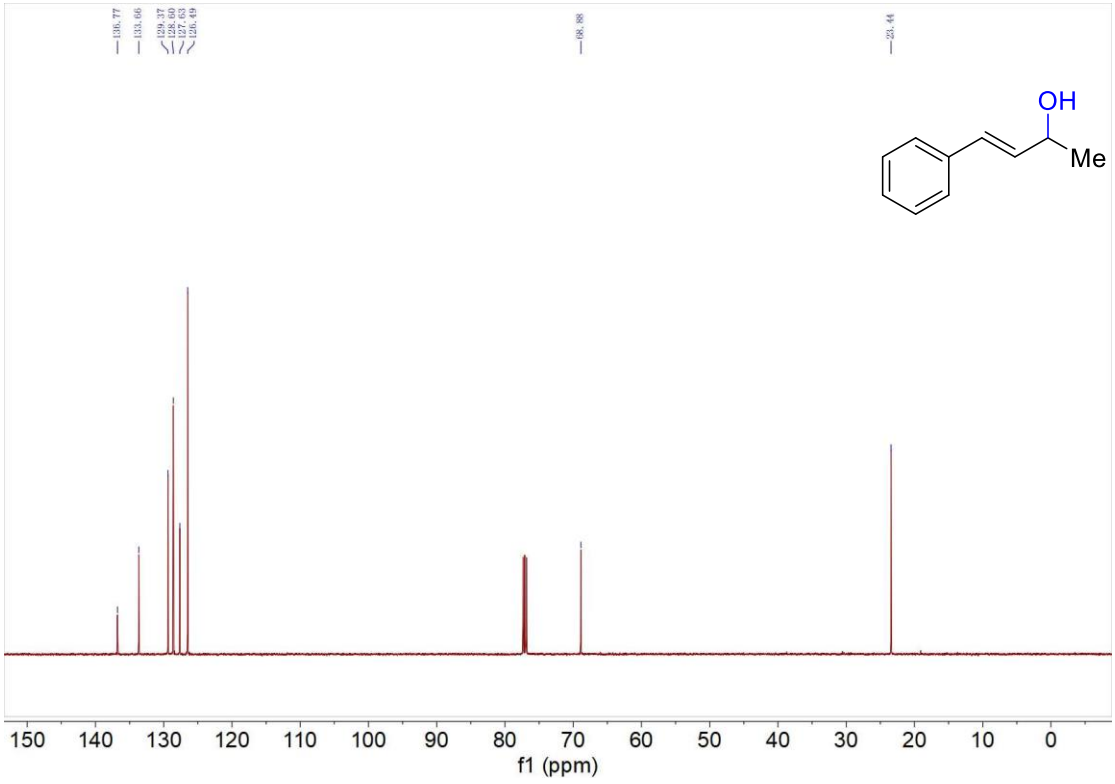
(*E*)-3-(thiophen-2-yl)-1-(p-tolyl)prop-2-en-1-ol (6l)



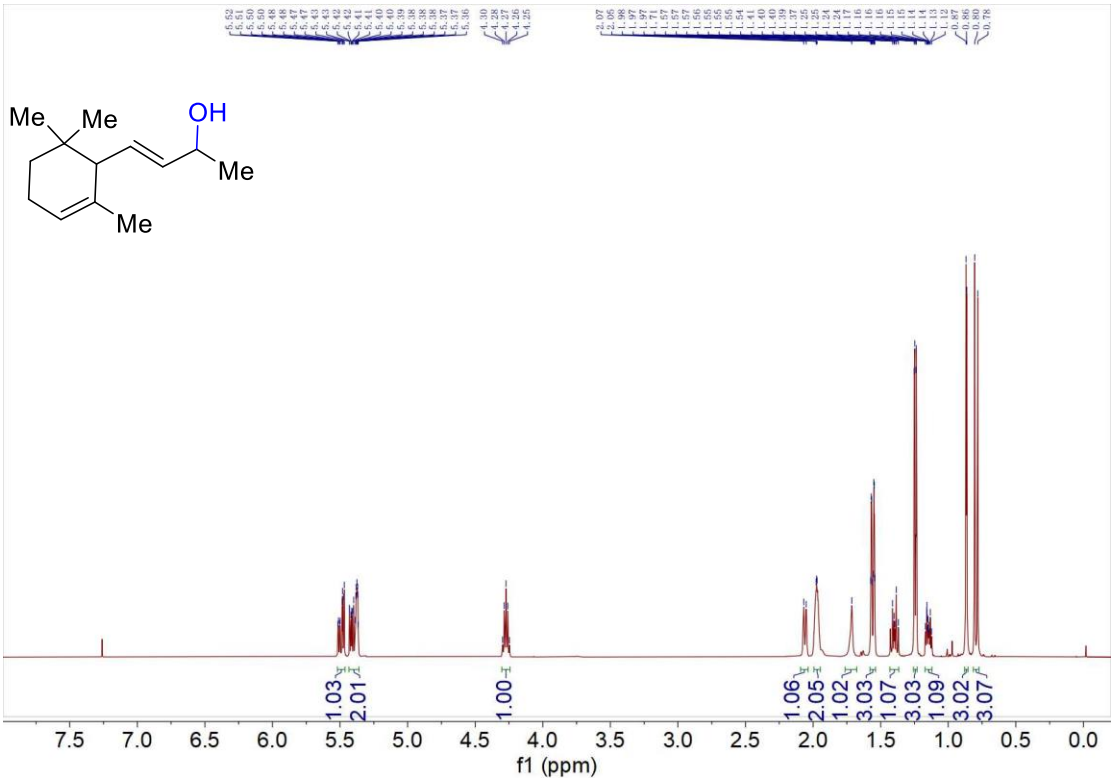


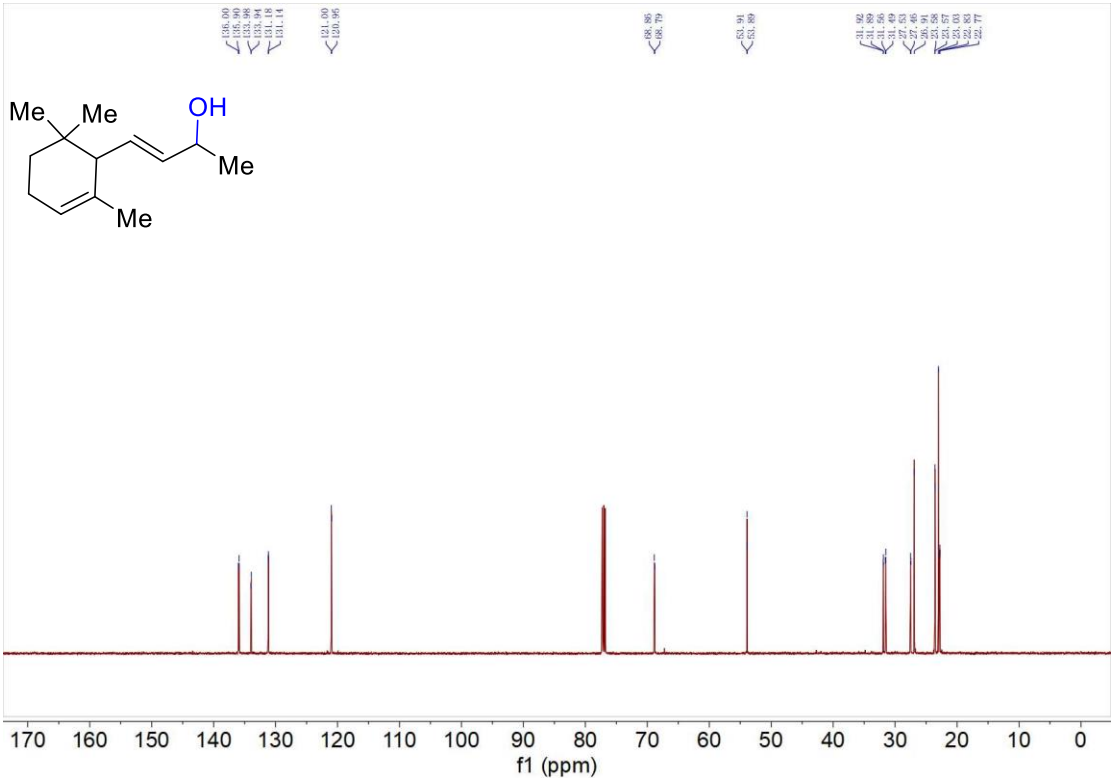
(*E*)-4-phenylbut-3-en-2-ol (6m)



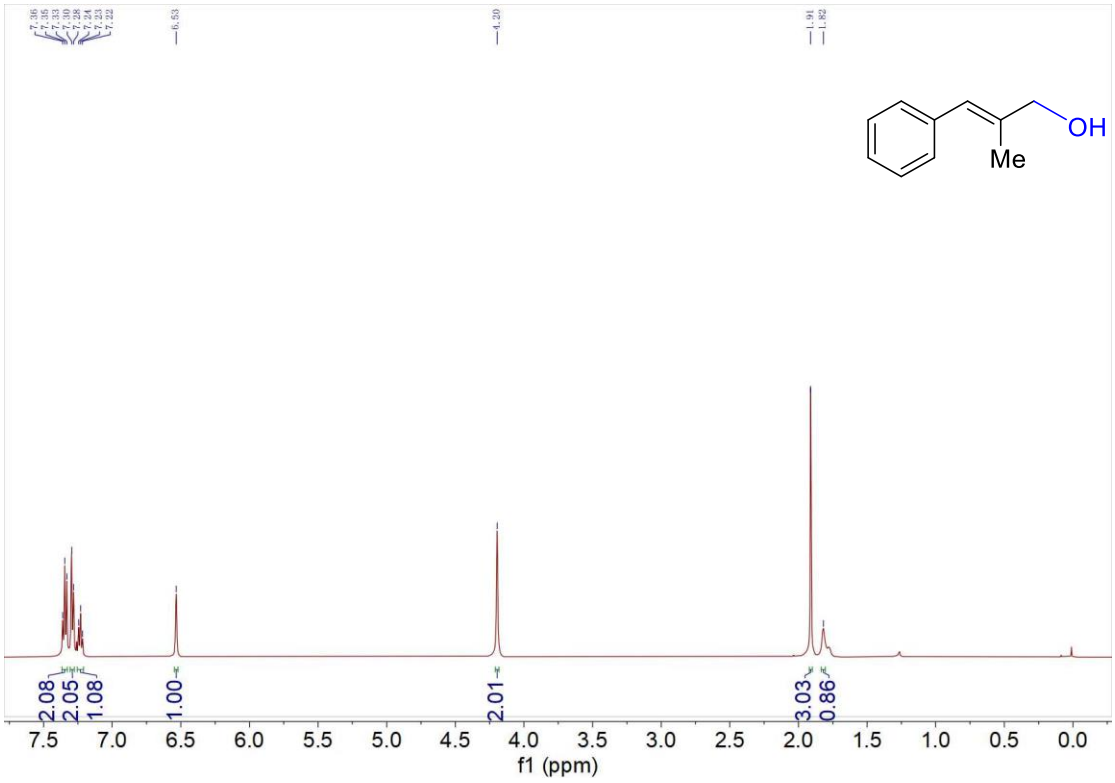


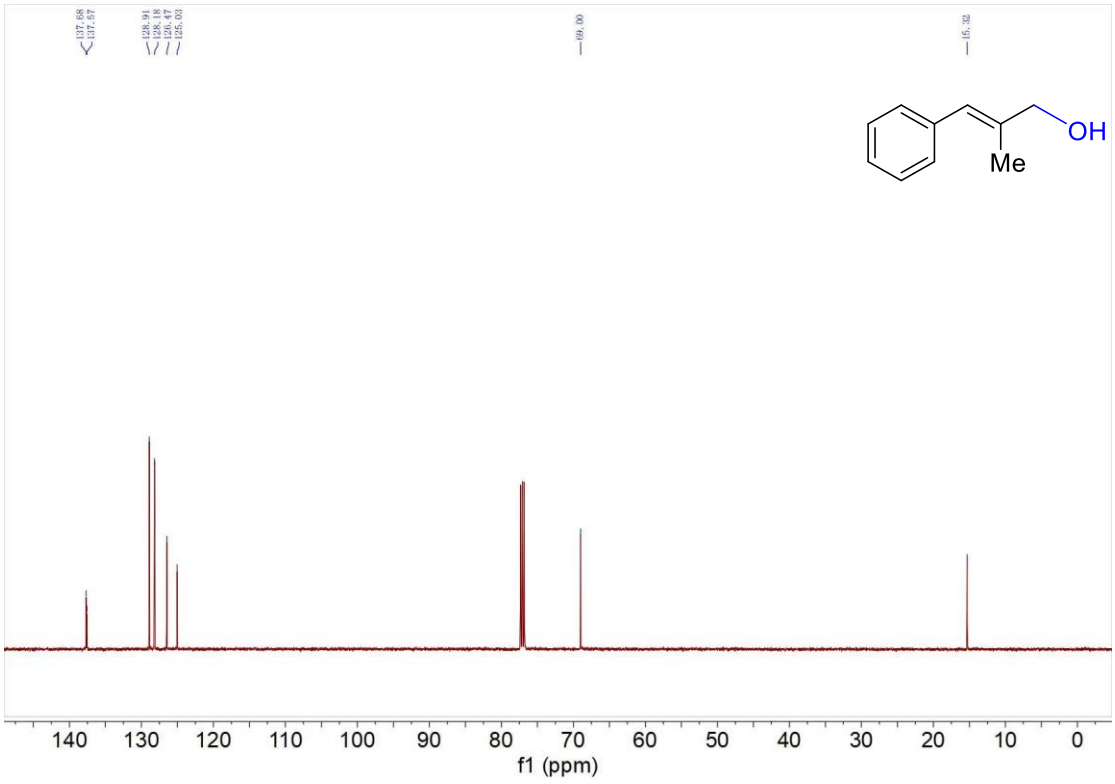
(*E*)-4-(2,6,6-trimethylcyclohex-2-en-1-yl)but-3-en-2-ol (6n)



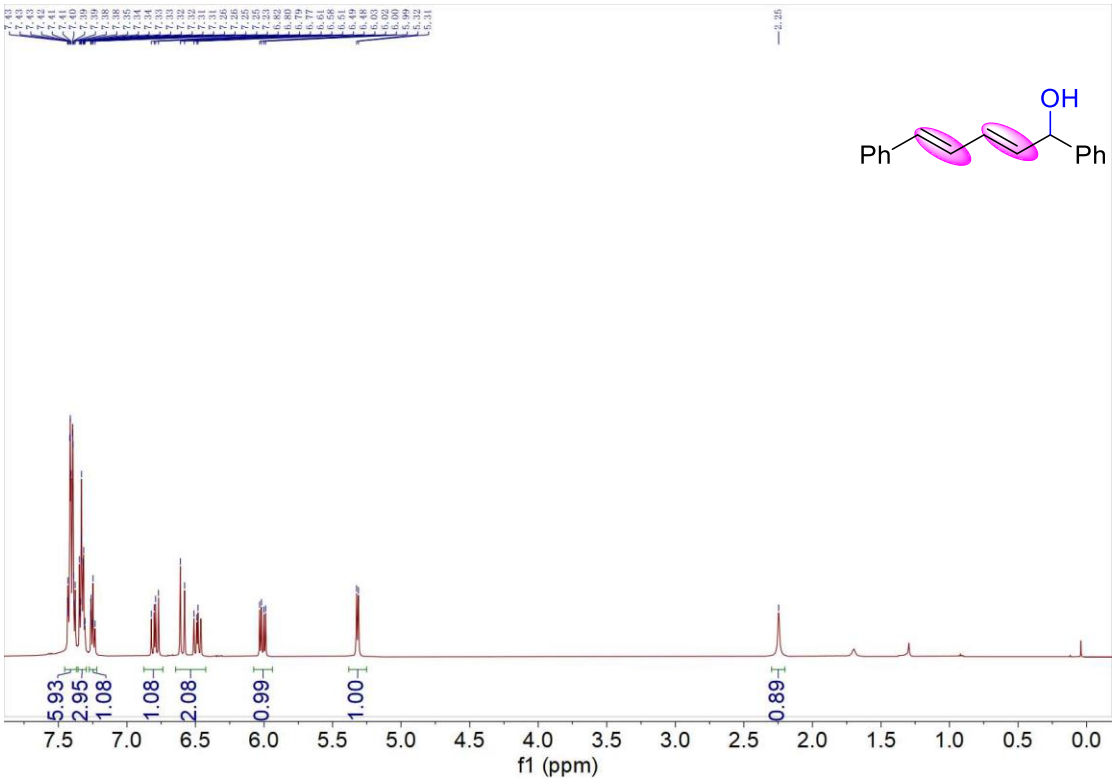


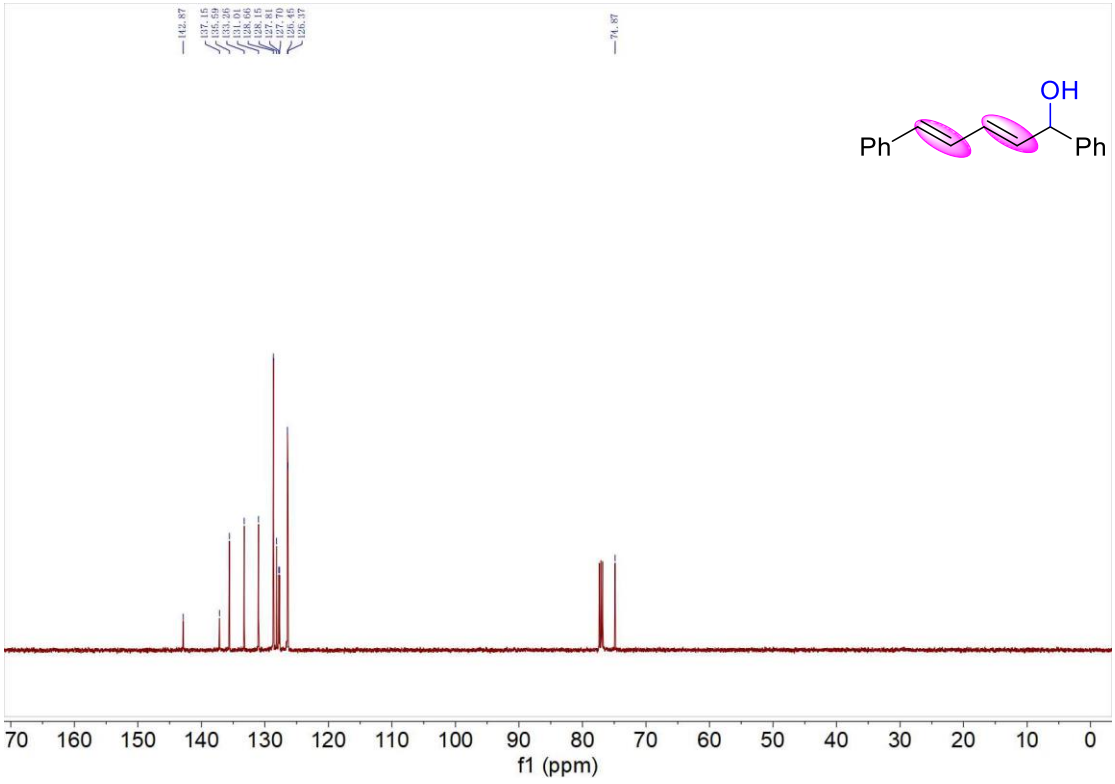
(*E*)-2-methyl-3-phenylprop-2-en-1-ol (6o)



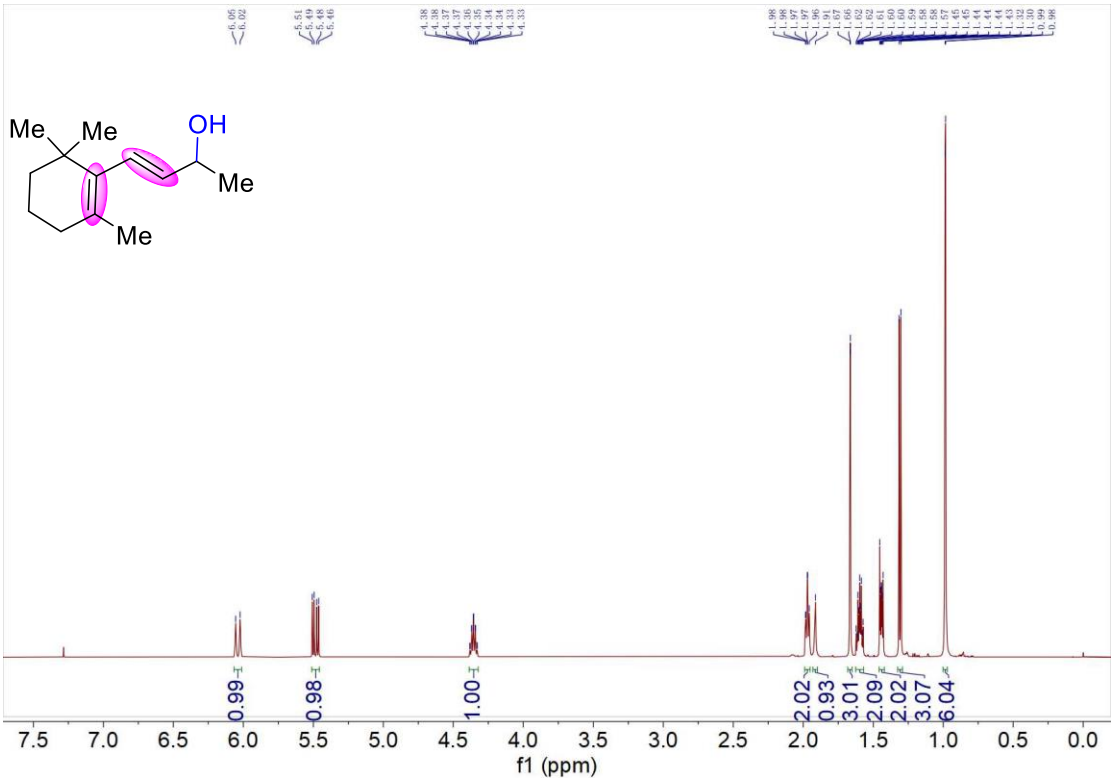


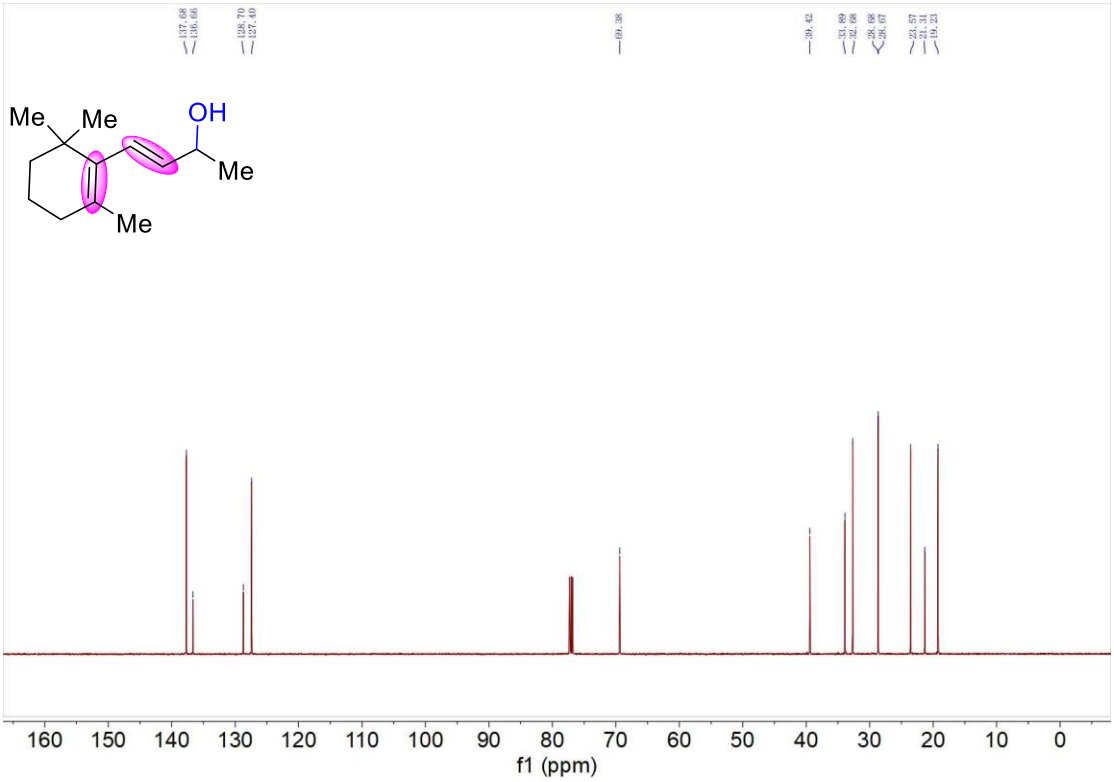
(2*E*,4*E*)-1,5-diphenylpenta-2,4-dien-1-ol (6p)



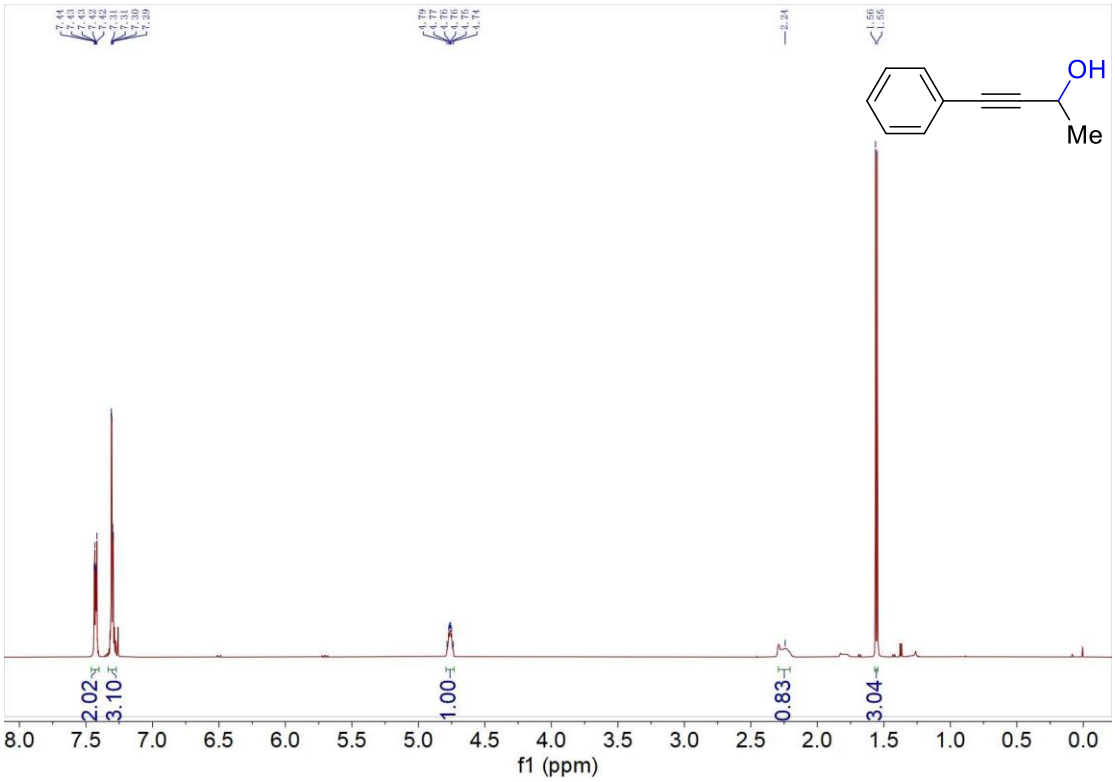


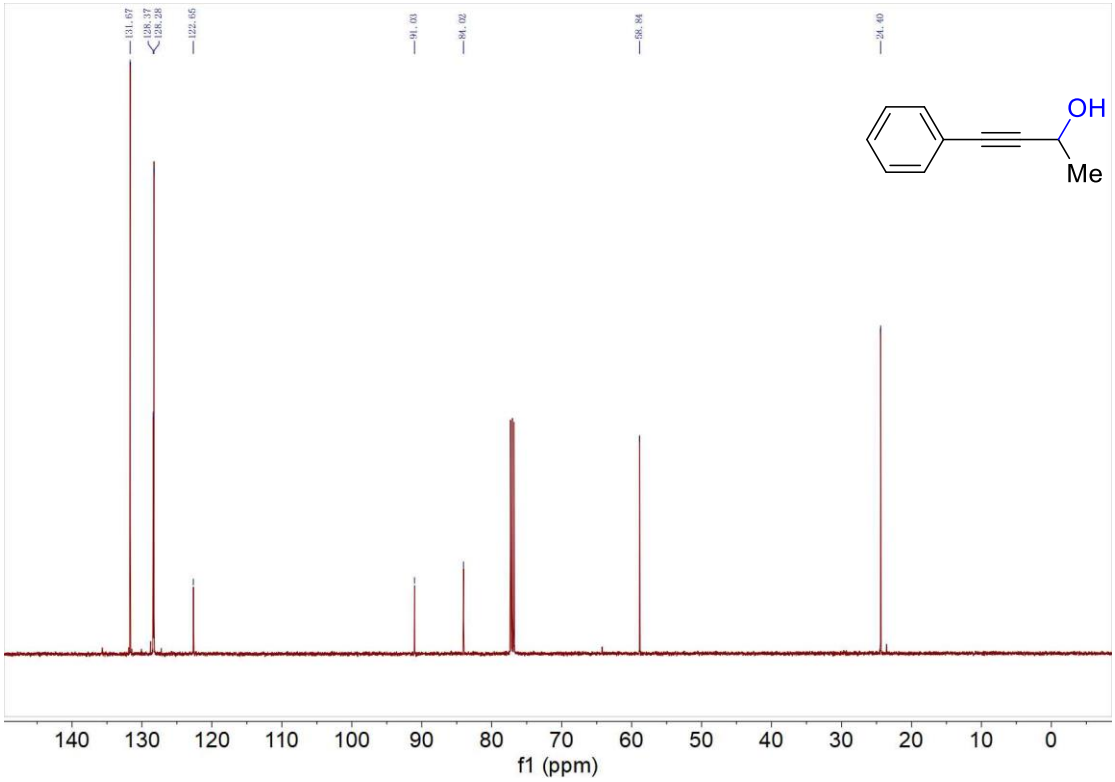
(*E*)-4-(2,6,6-trimethylcyclohex-1-en-1-yl)but-3-en-2-ol (6q)



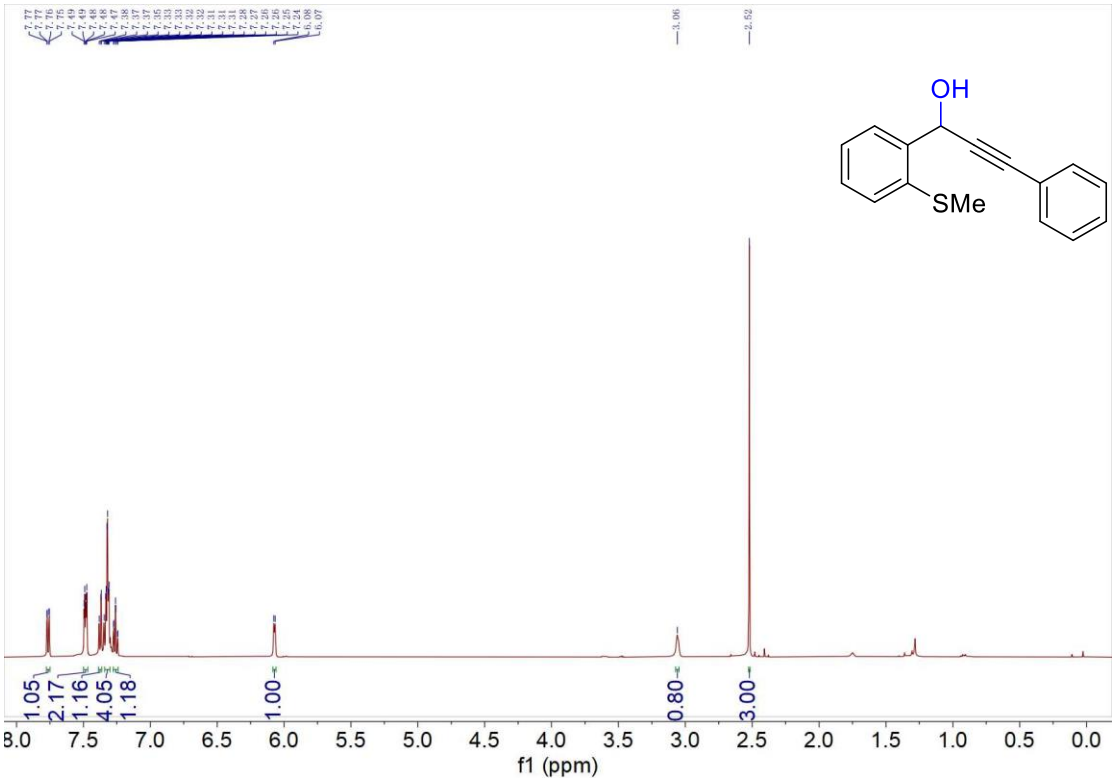


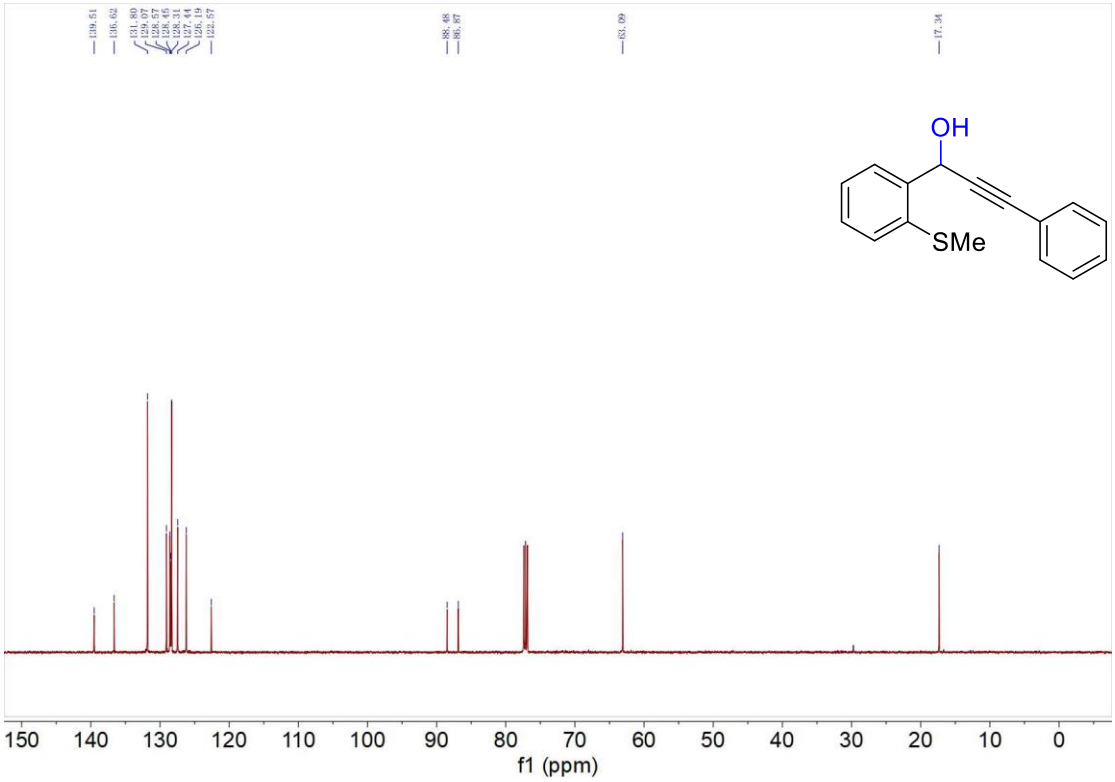
4-phenylbut-3-yn-2-ol (6r)



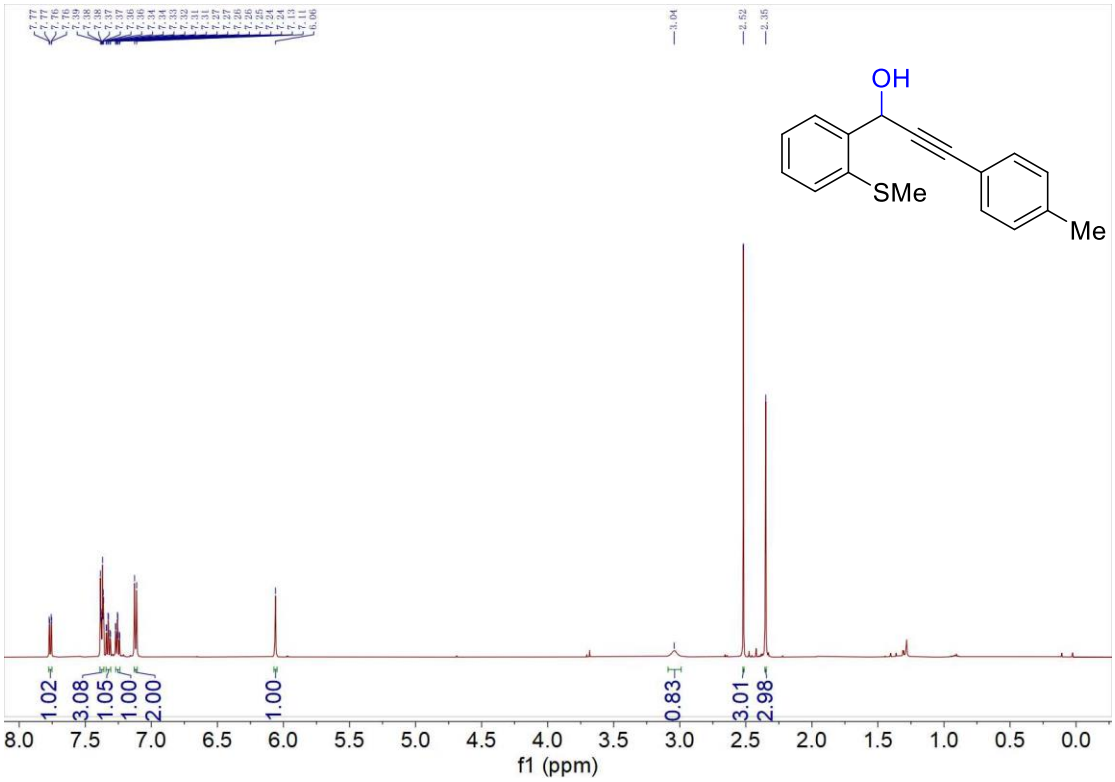


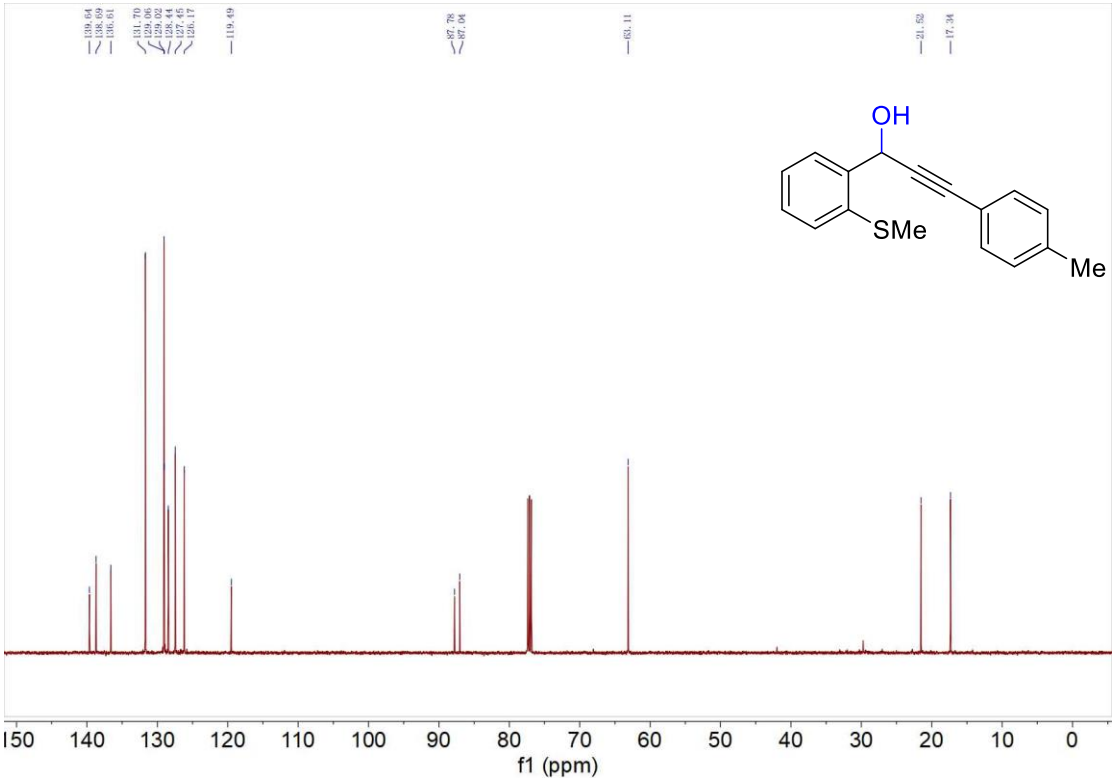
1-(2-(methylthio)phenyl)-3-phenylprop-2-yn-1-ol (6s)



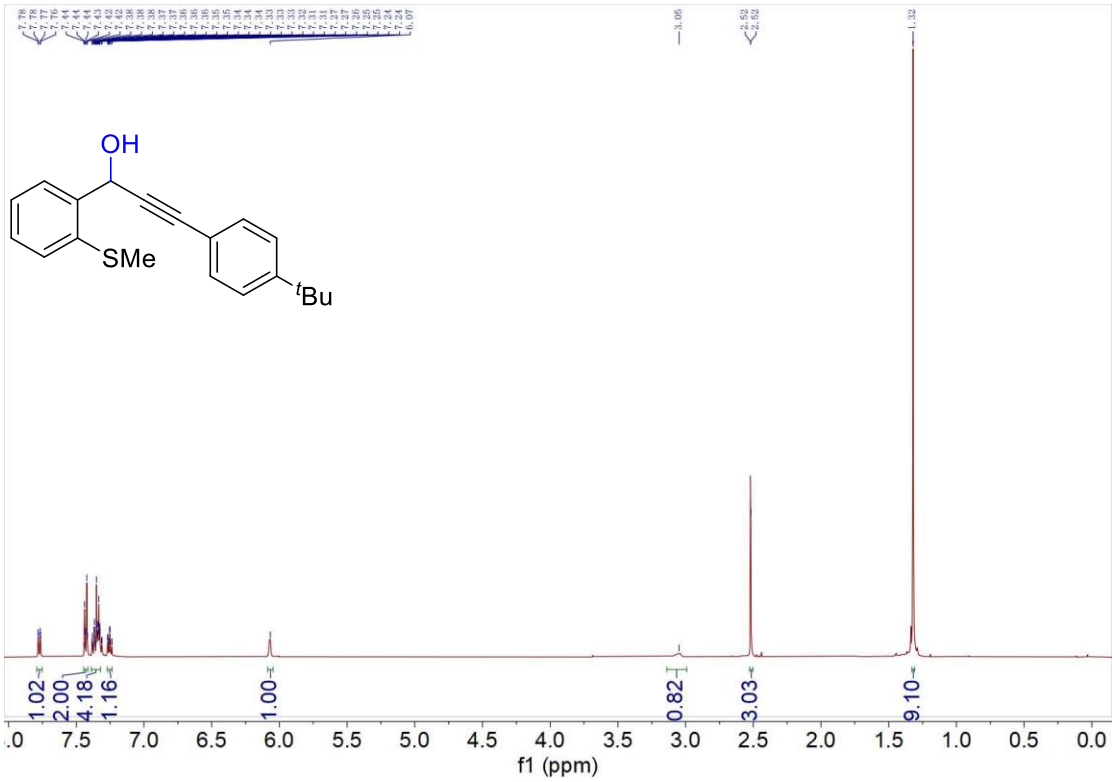


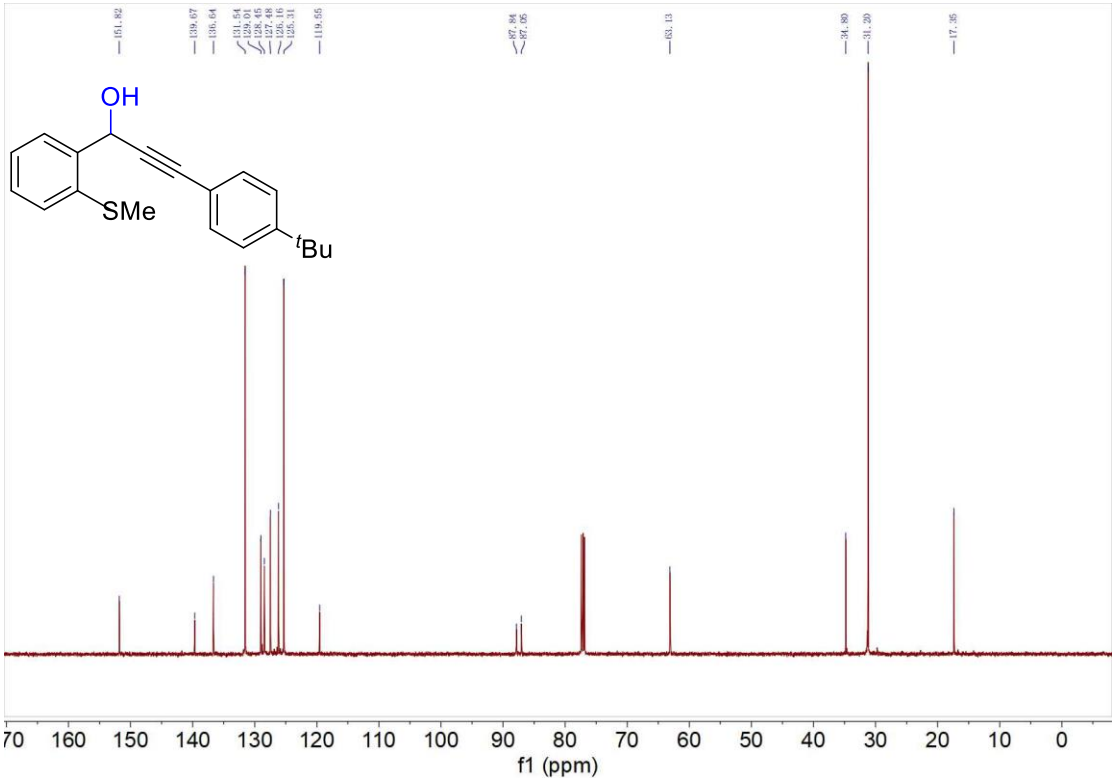
1-(2-(methylthio)phenyl)-3-(p-tolyl)prop-2-yn-1-ol (6t)





3-(4-(tert-butyl)phenyl)-1-(2-(methylthio)phenyl)prop-2-yn-1-ol (6u)





3-(4-methoxyphenyl)-1-(2-(methylthio)phenyl)prop-2-yn-1-ol (6v)