

Supporting Information

One simple Ir/hydrosilane catalytic system for chemoselective isomerization of 2-substituted allyl ethers

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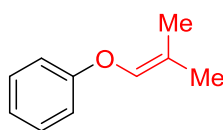
I. General information

All air or moisture sensitive reactions were conducted in oven-dried glassware under nitrogen atmosphere using dry solvents. Flash column chromatography was performed over silica gel (200-300 mesh) purchased from Qingdao Puke Co., China. Silanes and common organic chemicals were purchased from commercial suppliers, such as Sigma-Aldrich® and J&K® Scientific Ltd., and used as received. Iridium complexes were purchased from Strem® Chemicals, Inc. ¹H and ¹³C NMR spectra were collected on a Bruker AV 400 MHz NMR spectrometer using residue solvent peaks as an internal standard (¹H NMR: CDCl₃ at 7.26 ppm, ¹³C NMR: CDCl₃ at 77.0 ppm). Mass spectra were collected on a Thermo Scientific GC/MS ISQ7000 system, or a Xevo G2 Qtof mass spectrometer.

II. Iridium-catalyzed isomerization of 2-substituted allyl ethers

General Procedure.

In a glove box, to an oven-dried 5-mL vial were added the alkene (0.50 mmol), the silane (0.25 mmol), $[\text{Ir}(\mu\text{-Cl})(\text{cod})]_2$ (0.01 mmol, 2 mol %), and DCM (1.0 mL). The vial was capped and removed from the glove box. The reaction mixture was stirred at room temperature for 6 h, and then concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (eluent: 0→20% EtOAc in petroleum ether) to give the desired product.

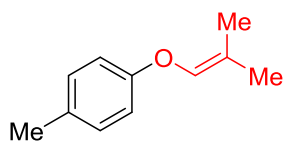


2a, 78%

((2-Methylprop-1-en-1-yl)oxy)benzene (2a) was prepared as colorless oil according to the General Procedure in 78% yield (57.8 mg).¹

¹H NMR (400 MHz, CDCl_3) δ 7.25-7.18 (m, 2 H), 6.95-6.88 (m, 3 H), 6.13 (t, J = 1.2 Hz, 1 H), 1.65 (s, 3 H), 1.62 (s, 3 H).

¹³C NMR (100 MHz, CDCl_3) δ 157.8, 135.1, 129.5, 121.8, 117.7, 115.8, 19.5, 15.2.

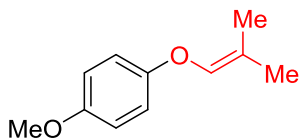


2b, 83%

1-Methyl-4-((2-methylprop-1-en-1-yl)oxy)benzene (2b) was prepared as colorless oil according to the General Procedure in 83 % yield (67.3 mg).²

¹H NMR (400 MHz, CDCl_3) δ 7.09 (d, J = 8.0 Hz, 2 H), 6.87 (d, J = 8.0 Hz, 2 H), 6.17 (s, 1 H), 2.30 (s, 3 H), 1.72 (s, 3 H), 1.68 (s, 3 H).

¹³C NMR (100 MHz, CDCl_3) δ 155.7, 135.5, 131.2, 129.9, 117.1, 115.6, 20.5, 19.5, 15.1

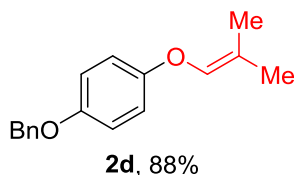


2c, 90%

1-Methoxy-4-((2-methylprop-1-en-1-yl)oxy)benzene (2c) was prepared as colorless oil according to the General Procedure in 90% yield (80.2 mg).³

¹H NMR (400 MHz, CDCl₃) δ 6.90 (d, *J* = 8.8 Hz, 2 H), 6.82 (d, *J* = 8.8 Hz, 2 H), 6.13 (d, *J* = 0.8 Hz, 1 H), 3.77 (s, 3 H), 1.72 (s, 3 H), 1.67 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃) δ 154.7, 151.9, 136.1, 116.8, 116.5, 114.6, 55.7, 19.4, 15.1.

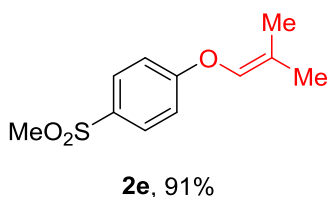


1-(Benzyloxy)-4-((2-methylprop-1-en-1-yl)oxy)benzene (2d) was prepared as white solid according to the General Procedure in 88% yield (111.9 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.44-7.30 (m, 5 H), 6.90 (s, 4 H), 6.14-6.11 (m, 1 H), 5.02 (s, 2 H), 1.71 (d, *J* = 0.8 Hz, 3 H), 1.67 (d, *J* = 0.8 Hz, 3 H).

¹³C NMR (100 MHz, CDCl₃) δ 153.8, 152.1, 137.1, 136.0, 128.5, 127.9, 127.4, 116.8, 116.6, 115.7, 70.6, 19.4, 15.1.

HRMS *m/z* (CI) calcd. for C₁₇H₁₉O₂ (M+H)⁺ 255.1386, found 255.1342.

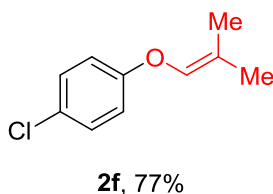


1-((2-Methylprop-1-en-1-yl)oxy)-4-(methylsulfonyl)benzene (2e) was prepared as white solid according to the General Procedure in 91% yield (103.0 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.87 (d, *J* = 8.8 Hz, 2 H), 7.10 (d, *J* = 8.8 Hz, 2 H), 6.24 (s, 1 H), 3.04 (s, 3 H), 1.73 (s, 3 H), 1.72 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃) δ 161.8, 133.6, 133.3, 129.5, 121.0, 115.9, 44.8, 19.4, 15.2.

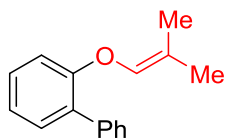
HRMS *m/z* (CI) calcd. for C₁₁H₁₅O₃S (M+H)⁺ 227.0743, found 227.0755.



1-Chloro-4-((2-methylprop-1-en-1-yl)oxy)benzene (2f) was prepared as colorless oil according to the General Procedure in 77% yield (70.3 mg).³

¹H NMR (400 MHz, CDCl₃) δ 7.27-7.21 (m, 2 H), 6.92-6.86 (m, 2 H), 6.16-6.13 (m, 1 H), 1.70 (d, *J* = 1.2 Hz, 3 H), 1.69 (d, *J* = 1.2 Hz, 3 H).

¹³C NMR (100 MHz, CDCl₃) δ 156.4, 134.8, 129.4, 126.7, 118.6, 117.0, 19.4, 15.2.



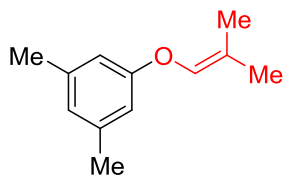
2g, 76%

2-((2-Methylprop-1-en-1-yl)oxy)-1,1'-biphenyl (2g) was prepared as colorless oil according to the General Procedure in 76 % yield (85.2 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.58-7.54 (m, 2 H), 7.43-7.27 (m, 5 H), 7.11-7.02 (m, 2 H), 6.19-6.16 (m, 1H), 1.66 (s, 6 H).

¹³C NMR (100 MHz, CDCl₃) δ 154.6, 138.1, 135.5, 131.0, 130.9, 129.5, 128.5, 127.9, 126.9, 122.1, 117.4, 115.0, 19.4, 15.3.

HRMS *m/z* (CI) calcd. for C₁₆H₁₇O (M+H)⁺ 225.1280, found 225.1294.

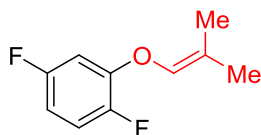


2h, 66%

1,3-Dimethyl-5-((2-methylprop-1-en-1-yl)oxy)benzene (2h) was prepared as colorless oil according to the General Procedure in 66% yield (58.2 mg).⁴

¹H NMR (400 MHz, CDCl₃) δ 6.65 (s, 1 H), 6.60 (s, 2 H), 6.17 (t, *J* = 1.2 Hz, 1 H), 2.29 (s, 6 H), 1.71 (d, *J* = 0.4 Hz, 3 H), 1.69 (d, *J* = 0.8 Hz, 3 H).

¹³C NMR (100 MHz, CDCl₃) δ 157.8, 139.3, 135.3, 123.6, 117.2, 113.5, 21.3, 19.5, 15.1.



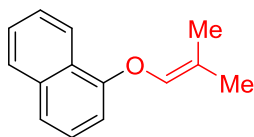
2i, 60%

1,4-difluoro-2-((2-methylprop-1-en-1-yl)oxy)benzene (2i) was prepared as colorless oil according to the General Procedure in 60% yield (55.3 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.06-6.90 (m, 1 H), 6.77-6.71 (m, 1 H), 6.66-6.58 (m, 1 H), 6.14 (t, *J* = 1.2 Hz, 1 H), 1.74 (d, *J* = 0.8 Hz, 3 H), 1.71 (d, *J* = 1.2 Hz, 3 H).

¹³C NMR (100 MHz, CDCl₃) δ 134.8, 134.3, 129.5, 128.0, 120.4, 116.52 (dd, *J* = 20.4, 9.9 Hz), 107.82 (dd, *J* = 23.8, 6.7 Hz), 104.27 (d, *J* = 27.5 Hz), 19.4, 15.2.

HRMS *m/z* (CI) calcd. for C₁₀H₁₁F₂O (M+H)⁺ 185.0779, found 185.0801.



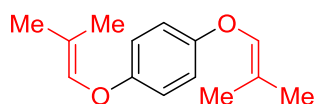
2j, 60%

1-((2-Methylprop-1-en-1-yl)oxy)naphthalene (2j) was prepared as pale yellow oil according to the General Procedure in 60% yield (59.5 mg).

¹H NMR (400 MHz, CDCl₃) δ 8.32-8.27 (m, 1 H), 7.82-7.76 (m, 1 H), 7.50-7.45 (m, 3 H), 7.34 (t, *J* = 7.6 Hz, 1 H), 6.90 (d, *J* = 7.6 Hz, 1 H), 6.38-6.35 (m, 1 H), 1.82 (s, 3 H), 1.74 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃) δ 153.5, 135.2, 134.6, 127.5, 126.4, 125.7, 125.5, 125.4, 121.9, 121.4, 118.2, 107.5, 19.5, 15.3.

HRMS *m/z* (CI) calcd. for C₁₄H₁₅O (M+H)⁺ 199.1124, found 199.1131.



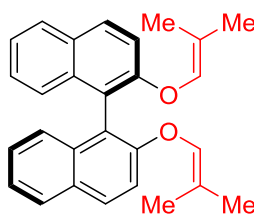
2k, 72%

1,4-Bis((2-methylprop-1-en-1-yl)oxy)benzene (2k) was prepared as white solid according to the General Procedure in 72% yield (78.6 mg).

¹H NMR (400 MHz, CDCl₃) δ 6.90 (s, 4 H), 6.14 (t, *J* = 1.2 Hz, 2 H), 1.72 (d, *J* = 0.8 Hz, 6 H), 1.68 (d, *J* = 1.2 Hz, 6 H).

¹³C NMR (100 MHz, CDCl₃) δ 152.8, 135.9, 116.9, 116.7, 19.5, 15.1.

HRMS *m/z* (CI) calcd. for C₁₄H₁₉O₂ (M+H)⁺ 219.1386, found 219.1402.



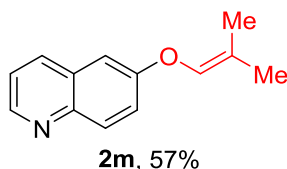
2l, 90%

7,7'-Bis((2-methylprop-1-en-1-yl)oxy)-1,1'-binaphthalene (2l) was prepared as white solid according to the General Procedure in 90% yield (177.5 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.84-7.75 (m, 4 H), 7.33-7.30 (m, 2 H), 7.28-7.22 (m, 2 H), 7.18-7.11 (m, 4 H), 6.05 (t, *J* = 1.6 Hz, 2 H), 1.39 (s, 6 H), 1.20 (s, 6 H).

¹³C NMR (100 MHz, CDCl₃) δ 153.1, 136.1, 134.1, 129.8, 129.3, 127.8, 126.3, 125.6, 123.9, 120.0, 116.9, 116.8, 19.2, 14.8.

HRMS *m/z* (CI) calcd. for C₂₈H₂₇O₂ (M+H)⁺ 395.2012, found 395.2002.

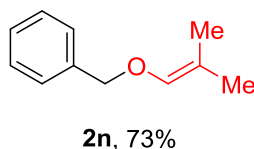


6-((2-Methylprop-1-en-1-yl)oxy)quinoline (2m) was prepared as pale yellow oil according to the General Procedure in 57 % yield (56.8 mg).

¹H NMR (400 MHz, CDCl₃) δ 8.80-8.77 (m, 1 H), 8.05-8.02 (m, 2 H), 7.46-7.44 (m, 1 H), 7.38-7.32 (m, 1 H), 7.19 (s, 1 H), 6.33 (s, 1 H), 1.76 (s, 6 H).

¹³C NMR (100 MHz, CDCl₃) δ 155.8, 148.4, 144.8, 134.9, 134.7, 131.1, 129.1, 122.0, 121.4, 119.3, 108.6, 19.5, 15.3.

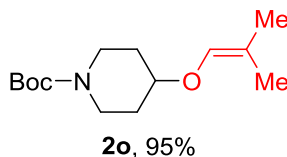
HRMS *m/z* (CI) calcd. for C₁₃H₁₄NO (M+H)⁺ 200.1075, found 200.1088.



(((2-Methylprop-1-en-1-yl)oxy)methyl)benzene (2n) was prepared as colorless oil according to the General Procedure in 73% yield (59.2 mg).⁵

¹H NMR (400 MHz, CDCl₃) δ 7.37-7.26 (m, 5 H), 5.88 (s, 1 H), 4.73 (s, 2 H), 1.65 (s, 3 H), 1.53 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃) δ 139.7, 138.1, 128.4, 127.6, 127.3, 111.2, 73.3, 19.5, 15.1.

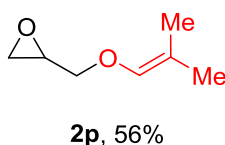


Tert-Butyl 4-((2-methylprop-1-en-1-yl)oxy)piperidine-1-carboxylate (2o) was prepared as colorless oil according to the General Procedure in 95% yield (121.3 mg).

¹H NMR (400 MHz, CDCl₃) δ 5.82-5.79 (m, 1 H), 3.78-3.71 (m, 1 H), 3.63-3.56 (m, 2 H), 3.33-3.26 (m, 2 H), 1.81-1.73 (m, 2 H), 1.66-1.58 (m, 2 H), 1.62 (d, *J* = 0.8 Hz, 3 H), 1.55 (d, *J* = 0.8 Hz, 3 H), 1.45 (s, 9 H).

¹³C NMR (100 MHz, CDCl₃) δ 154.8, 138.0, 111.9, 79.4, 75.0, 31.0, 28.4, 19.5, 15.1.

HRMS *m/z* (CI) calcd. for C₁₄H₂₅NO₃Na (M+Na)⁺ 278.1732, found 278.1740.

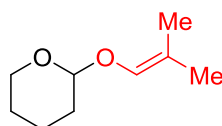


2-(((2-Methylprop-1-en-1-yl)oxy)methyl)oxirane (2p) was prepared as colorless oil according to the General Procedure in 56% yield (35.9 mg).

¹H NMR (400 MHz, CDCl₃) δ 5.84 (t, *J* = 1.2 Hz, 1 H), 3.95-3.90 (m, 1 H), 3.67-3.61 (m, 1 H), 3.21-3.15 (m, 1 H), 2.84-2.80 (m, 1 H), 2.67-2.62 (m, 1 H), 1.63 (s, 3 H), 1.55 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃) δ 139.8, 111.4, 71.9, 50.7, 44.2, 19.4, 14.9.

HRMS *m/z* (CI) calcd. for C₇H₁₂O₂K (M+K)⁺ 167.0474, found 167.0501.

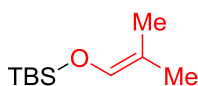


2q, 66%

2-((2-Methylprop-1-en-1-yl)oxy)tetrahydro-2H-pyran (2q) was prepared as colorless oil according to the General Procedure in 66% yield (51.5 mg).⁶

¹H NMR (400 MHz, CDCl₃) δ 6.00 (t, *J* = 1.2 Hz, 1 H), 4.84 (t, *J* = 3.2 Hz, 1 H), 3.88-3.81 (m, 1 H), 3.57-3.51 (m, 1 H), 1.95-1.68 (m, 4 H), 1.66 (s, 3 H), 1.64-1.58 (m, 2 H), 1.57 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃) δ 137.0, 111.8, 98.3, 62.1, 29.9, 25.3, 19.6, 19.0, 15.0.



2r, 90%

tert-Butyldimethyl((2-methylprop-1-en-1-yl)oxy)silane (2r) was prepared as colorless oil according to the General Procedure in 90% yield (83.8 mg).⁷

¹H NMR (400 MHz, CDCl₃) δ 6.04-6.01 (m, 1 H), 1.60 (s, 3 H), 1.53 (s, 3 H), 0.92 (s, 9 H), 0.11 (s, 6 H).

¹³C NMR (100 MHz, CDCl₃) δ 133.5, 113.3, 25.7, 19.3, 18.2, 14.7, -5.3.



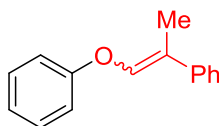
2s, 66%

1-Benzyl-4-(((2-methylprop-1-en-1-yl)oxy)methyl)-1H-1,2,3-triazole (2s) was prepared as colorless oil according to the General Procedure in 66 % yield (80.3 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.38 (s, 1 H), 7.32-7.28 (m, 3 H), 7.22-7.18 (m, 2 H), 5.84 (s, 1 H), 5.45 (s, 2 H), 4.75 (s, 2 H), 1.48 (s, 2 H), 1.45 (s, 2 H).

¹³C NMR (100 MHz, CDCl₃) δ 145.5, 139.3, 134.5, 129.1, 128.7, 128.1, 122.3, 112.1, 65.0, 54.2, 19.4, 15.0.

HRMS *m/z* (CI) calcd. for C₁₄H₁₈N₃O (M+H)⁺ 244.1450, found 244.1436.



2u, 76%, *E/Z* = 4:1

(1-Phenoxyprop-1-en-2-yl)benzene (2u) was prepared as colorless oil according to the General Procedure in 76% yield (79.9 mg, *E/Z* = 4:1).

Z-2u

¹H NMR (400 MHz, CDCl₃) δ 7.32 (d, *J* = 7.2 Hz, 2 H), 7.28-7.21 (m, 4 H), 7.19-7.13 (m, 1 H), 6.99 (q, *J* = 2 Hz, 3 H), 6.77 (d, *J* = 1.2 Hz, 1 H), 2.07 (d, *J* = 1.2 Hz, 3 H).

¹³C NMR (100 MHz, CDCl₃) δ 157.6, 139.8, 139.0, 129.6, 128.4, 126.7, 125.5, 122.7, 120.6, 116.4, 13.0.

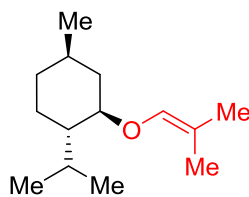
HRMS *m/z* (CI) calcd. for C₁₅H₁₅O (M+H)⁺ 211.1124, found 211.1058.

E-2u

¹H NMR (400 MHz, CDCl₃) δ 7.56-7.51 (m, 2 H), 7.29-7.22 (m, 4 H), 7.18-7.14 (m, 1 H), 7.01-6.97 (m, 3 H), 6.47 (d, *J* = 1.6 Hz, 1 H), 1.99 (d, *J* = 1.2 Hz, 3 H).

¹³C NMR (100 MHz, CDCl₃) δ 157.5, 137.7, 129.6, 128.1, 127.7, 126.8, 122.7, 117.6, 116.5, 18.5.

HRMS *m/z* (CI) calcd. for C₁₅H₁₅O (M+H)⁺ 211.1124, found 211.1078.

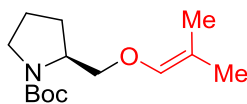


2v, 89%
(from **Menthol**)

(1S,2R,4R)-1-Isopropyl-4-methyl-2-((2-methylprop-1-en-1-yl)oxy)cyclohexane (2v) was prepared as colorless oil according to the General Procedure in 89% yield (93.6 mg).⁸

¹H NMR (400 MHz, CDCl₃) δ 5.84 (t, *J* = 1.2 Hz, 1 H), 3.31-3.22 (m, 1 H), 2.22-2.12 (m, 1 H), 2.03-1.96 (m, 1 H), 1.68-1.61 (m, 2 H), 1.60 (s, 3 H), 1.54 (s, 3 H), 1.40-1.25 (m, 3 H), 1.04-0.95 (m, 2 H), 0.92-0.87 (q, *J* = 1.2 Hz, 6 H), 0.78 (d, *J* = 7.2 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 139.2, 80.7, 47.8, 41.5, 34.5, 31.6, 25.8, 23.5, 22.2, 20.8, 19.6, 16.3, 15.1.



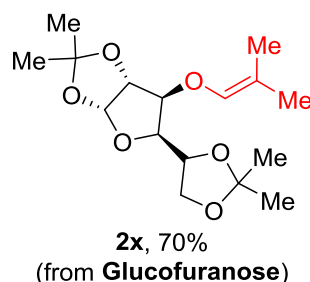
2w, 66%
(from **Prolinol**)

(S)-tert-Butyl 2-(((2-methylprop-1-en-1-yl)oxy)methyl)pyrrolidine-1-carboxylate (2w) was prepared as colorless oil according to the General Procedure in 66% yield (84.5 mg).

¹H NMR (400 MHz, CDCl₃) δ 5.82-5.77 (m, 1 H), 3.95-3.53 (m, 3 H), 3.36-3.28 (m, 2 H), 1.95-1.80 (m, 4 H), 1.62-1.57 (m, 3 H), 1.53 (s, 3 H), 1.47 (s, 9 H).

¹³C NMR (100 MHz, CDCl₃) δ 154.4, 140.5, 140.1, 110.4, 109.7, 79.3, 79.1, 72.2, 71.5, 56.4, 46.8, 46.4, 28.6, 28.5, 23.7, 22.8, 19.3, 14.9.

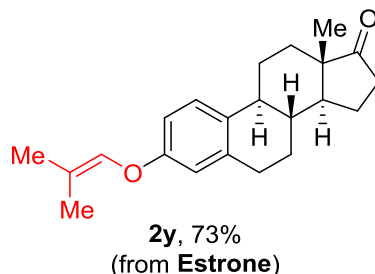
HRMS *m/z* (CI) calcd. for C₁₄H₂₅NO₃Na (M+Na)⁺ 278.1732, found 278.1735.



(3a*R*,5*R*,6*S*,6a*R*)-5-((*R*)-2,2-Dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyl-6-((2-methylprop-1-en-1-yl)oxy)tetrahydrofuro[2,3-*d*][1,3]dioxole (2x) was prepared as colorless oil according to the General Procedure in 70% yield (110.0 mg).⁹

¹H NMR (400 MHz, CDCl₃) δ 5.91-5.87 (m, 1 H), 5.88 (s, 1 H), 4.54-4.52 (m, 1 H), 4.35-4.33 (m, 1 H), 4.20-4.00 (m, 4 H), 1.57 (s, 3 H), 1.55 (s, 3 H), 1.50 (s, 3 H), 1.44 (s, 3 H), 1.34 (s, 3 H), 1.31 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃) δ 138.2, 113.1, 111.9, 109.0, 105.2, 82.7, 82.6, 80.7, 72.4, 67.1, 26.8, 26.8, 26.2, 25.3, 19.4, 15.1.

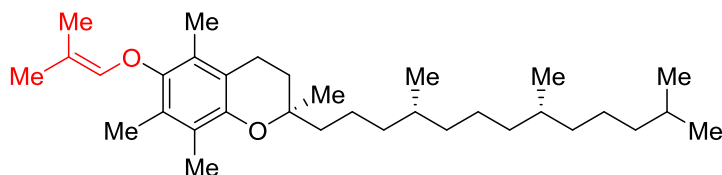


(8*R*,9*S*,13*S*,14*S*)-13-Methyl-3-((2-methylprop-1-en-1-yl)oxy)-7,8,9,11,12,13,15,16-octahydro-6*H*-cyclopenta[*a*]phenanthren-17(14*H*)-one (2y) was prepared as white solid according to the General Procedure in 73% yield (118.4 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.22-7.17 (m, 1 H), 6.79-6.67 (m, 2 H), 6.17 (t, *J* = 1.6 Hz, 1 H), 2.91-2.87 (m, 2 H), 2.53-2.45 (m, 1 H), 2.42-2.35 (m, 1 H), 2.27-2.20 (m, 1 H), 2.18-1.92 (m, 4 H), 1.71 (d, *J* = 0.8 Hz, 3 H), 1.68 (d, *J* = 1.2 Hz, 3 H), 1.63-1.41 (m, 6 H), 0.90 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃) δ 155.7, 137.8, 135.2, 133.2, 126.3, 117.2, 115.7, 113.3, 50.3, 47.9, 43.9, 38.2, 35.8, 31.5, 29.5, 26.4, 25.8, 21.5, 19.4, 15.1, 13.8.

HRMS *m/z* (CI) calcd. for C₂₂H₂₉O₂ (M+H)⁺ 325.2168, found 325.2176.



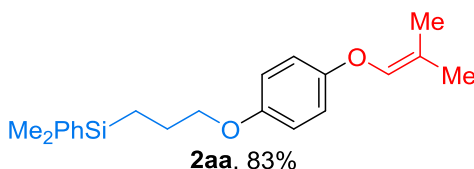
2z, 70%
(from **Vitamin E**)

(R)-2,5,7,8-Tetramethyl-6-((2-methylprop-1-en-1-yl)oxy)-2-((4R,8R)-4,8,12-trimethyltridecyl)chroman (2z) was prepared as pale yellow oil according to the General Procedure in 70% yield (170.0 mg).

¹H NMR (400 MHz, CDCl₃) δ 5.77 (t, *J* = 1.6 Hz, 1 H), 2.61-2.56 (m, 2 H), 2.13 (s, 3 H), 2.10 (s, 3 H), 2.08 (s, 3 H), 1.81 (s, 3 H), 1.87-1.71 (m, 3 H), 1.59 (s, 3 H), 1.57-1.50 (m, 3 H), 1.46-1.34 (m, 4 H), 1.30-1.06 (m, 16 H), 0.89-0.83 (m, 12 H).

¹³C NMR (100 MHz, CDCl₃) δ 148.1, 147.4, 140.4, 127.8, 125.8, 122.8, 117.4, 109.7, 74.8, 40.0, 39.4, 37.5, 37.4, 37.3, 32.8, 32.7, 28.0, 24.8, 24.4, 23.9, 22.7, 22.6, 21.0, 20.6, 19.7, 19.7, 19.4, 15.0, 12.7, 11.8, 11.7.

HRMS *m/z* (CI) calcd. for C₃₃H₅₇O₂ (M+H)⁺ 485.4359, found 485.4309.



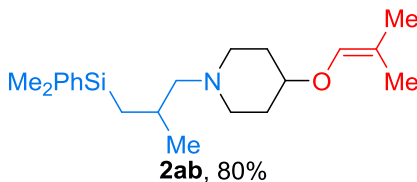
2aa, 83%

Dimethyl(3-(4-((2-methylprop-1-en-1-yl)oxy)phenoxy)propyl)(phenyl)silane (2aa) was prepared as colorless oil according to the General Procedure in 83% yield (141.4 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.46-7.42 (m, 2 H), 7.29-7.26 (m, 2.5 H), 7.20 (s, 0.5 H), 6.83-6.77 (m, 2 H), 6.72-6.68 (m, 2 H), 6.05 (t, *J* = 1.6 Hz, 1 H), 3.78 (t, *J* = 6.8 Hz, 2 H), 1.75-1.65 (m, 2 H), 1.64 (s, 3 H), 1.60 (d, *J* = 0.8 Hz, 3 H), 0.81-0.75 (m, *J* = 8.8 Hz, 3 H), 0.20 (d, *J* = 8.8 Hz, 6 H).

¹³C NMR (100 MHz, CDCl₃) δ 154.2, 151.8, 136.2, 133.5, 128.9, 127.8, 116.8, 116.4, 115.3, 23.9, 19.5, 15.1, 11.7, -3.1.

HRMS *m/z* (CI) calcd. for C₂₁H₂₉O₂Si (M+H)⁺ 341.1937, found 341.1966.



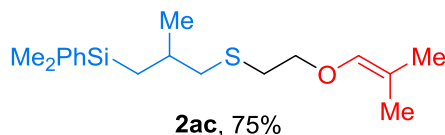
2ab, 80%

1-(3-(Dimethyl(phenyl)silyl)-2-methylpropyl)-4-((2-methylprop-1-en-1-yl)oxy)piperidine (2ab) was prepared as colorless oil according to the General Procedure in 80% yield (138.2 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.54-7.49 (m, 2 H), 7.33 (t, *J* = 3.2 Hz, 3 H), 5.80 (s, 1 H), 3.55 (s, 1 H), 2.55 (s, 2 H), 2.03 (s, 4 H), 1.80 (s, 3 H), 1.64-1.53 (m, 4 H), 0.864 (d, *J* = 6.0 Hz, 3 H), 0.30 (s, 3 H), 0.29 (s, 3 H).

^{13}C NMR (100 MHz, CDCl_3) δ 138.3, 133.5, 128.7, 127.7, 111.2, 68.3, 51.1, 31.5, 27.1, 21.8, 21.4, 19.6, 15.1, -1.8, -2.1.

HRMS m/z (CI) calcd. for $\text{C}_{21}\text{H}_{36}\text{NOSi}$ ($\text{M}+\text{H}$) $^+$ 346.2566, found 346.2553.

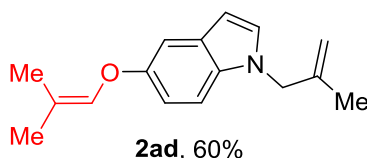


Dimethyl(2-methyl-3-((2-methylprop-1-en-1-yl)oxy)ethylthio)propyl(phenyl)silane (2ac) was prepared as colorless oil according to the General Procedure in 75% yield (121.0 mg).

^1H NMR (400 MHz, CDCl_3) δ 7.51 (s, 2 H), 7.35 (s, 3 H), 5.76 (s, 1 H), 3.73 (t, J = 7.2 Hz, 2 H), 2.60 (m, 2 H), 2.49-2.39 (m, 2 H), 1.60 (s, 3 H), 1.54 (s, 4 H), 1.00-0.89 (m, 2 H), 0.96 (d, J = 7.2 Hz, 3 H), 0.31 (s, 6 H).

^{13}C NMR (100 MHz, CDCl_3) δ 139.6, 133.5, 128.9, 127.8, 71.3, 43.7, 31.9, 30.2, 23.5, 22.3, 19.5, 15.0, -2.0, -2.2.

HRMS m/z (CI) calcd. for $\text{C}_{18}\text{H}_{31}\text{OSSi}$ ($\text{M}+\text{H}$) $^+$ 323.1865, found 323.1888.

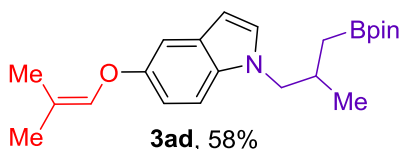


1-(2-Methylallyl)-5-((2-methylprop-1-en-1-yl)oxy)-1H-indole (2ad) was prepared as colorless oil according to the General Procedure in 60 % yield (72.9 mg).

^1H NMR (400 MHz, CDCl_3) δ 7.17-7.09 (m, 2 H), 6.98 (d, J = 3.0 Hz, 1 H), 6.85-6.81 (m, 1 H), 6.34 (d, J = 3.0 Hz, 1 H), 6.16 (s, 1 H), 4.83 (s, 1 H), 4.63 (s, 1 H), 4.52 (s, 2 H), 1.68 (s, 3H), 1.62 (s, 3 H), 1.57 (s, 3 H).

^{13}C NMR (100 MHz, CDCl_3) δ 152.1, 141.2, 136.9, 132.5, 129.1, 129.0, 115.7, 112.6, 112.3, 110.2, 106.2, 100.9, 52.7, 19.8, 19.5, 15.1.

HRMS m/z (CI) calcd. for $\text{C}_{16}\text{H}_{20}\text{NO}$ ($\text{M}+\text{H}$) $^+$ 242.1545, found 242.1574.



1-(2-Methyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propyl)-5-((2-methylprop-1-en-1-yl)oxy)-1H-indole(3ad) was prepared as colorless oil according to the General Procedure in 58 % yield (107.3 mg).

^1H NMR (400 MHz, CDCl_3) δ 7.34-7.29 (m, 1 H), 7.16 (s, 1 H), 7.07 (d, J = 2.8 Hz, 1 H), 6.93-6.88 (m, 1 H), 6.38 (d, J = 2.8 Hz, 1 H), 6.24 (s, 1 H), 4.07-4.00 (m, 1 H), 3.84-3.76 (m, 1 H), 2.35-2.18 (m, 1 H), 1.76 (s, 3 H), 1.70 (s, 3 H), 1.26 (s, 12 H), 0.90 (d, J = 6.4 Hz, 3 H), 0.88-0.81 (m, 1 H), 0.76-0.68 (m, 1 H).

¹³C NMR (100 MHz, CDCl₃) δ 151.9, 137.0, 132.6, 129.4, 128.8, 115.5, 112.2, 110.4, 106.1, 100.2, 83.2, 54.6, 30.85, 24.9, 24.8, 20.2, 19.5, 15.1.

HRMS *m/z* (CI) calcd. for C₂₂H₃₃BNO₃ (M+H)⁺ 370.2553, found 370.2568.

III. Deuterium labeling experiment

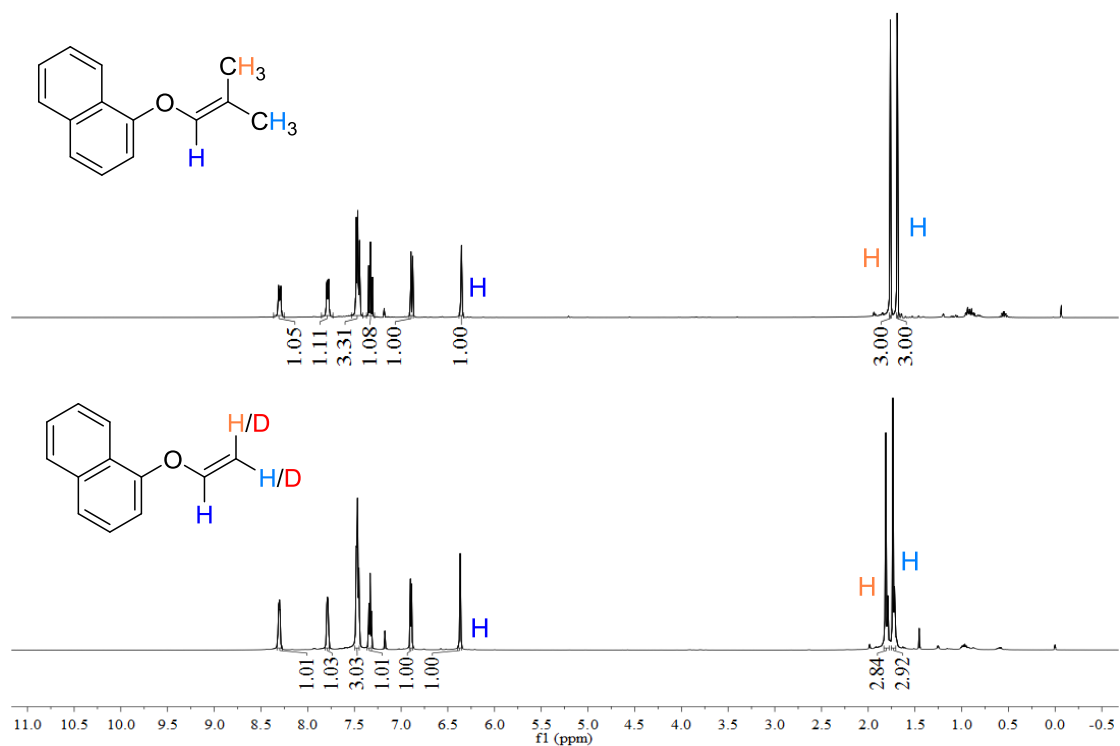


Figure S1. ^1H NMR spectra of **2j** and **2j-d**.

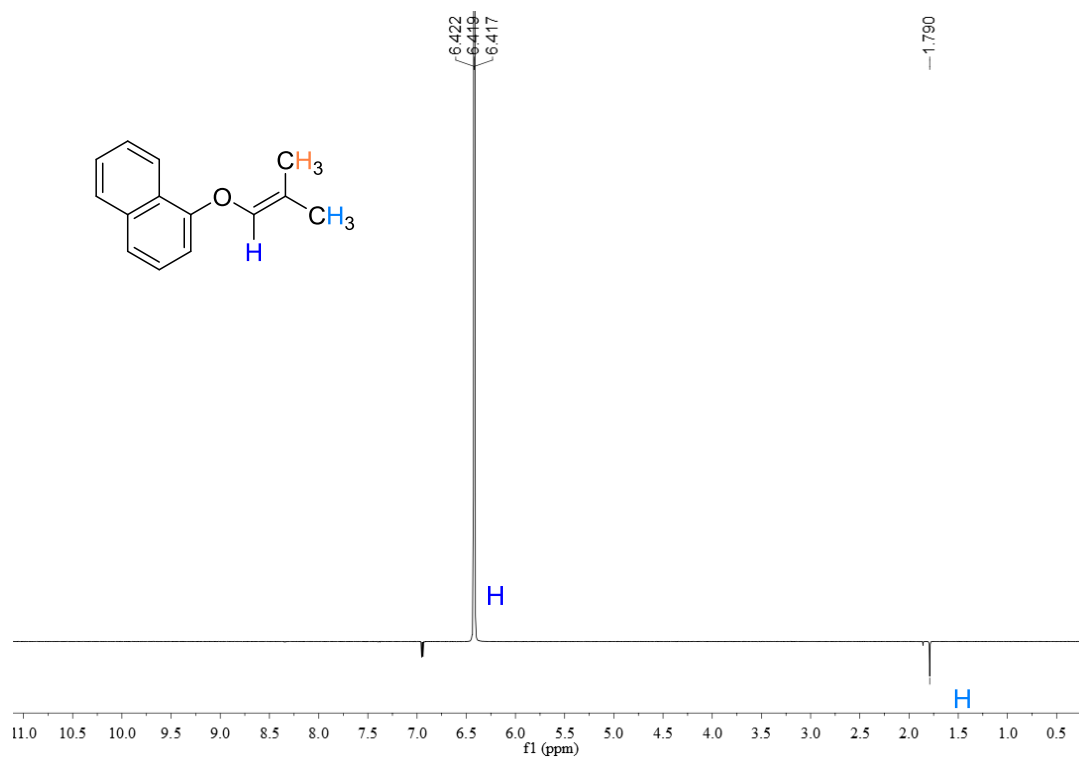
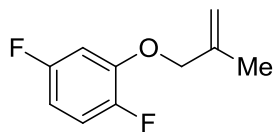


Figure S2. ^1H - ^1H Correlation and 1D NOESY NMR spectra of **2j**.

IV. New substrates

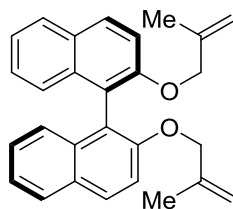


1,4-Difluoro-2-((2-methylallyl)oxy)benzene (1i) was synthesized from 2,5-difluorophenol and 3-bromo-2-methylpropene according to reported procedure.¹⁰

¹H NMR (400 MHz, CDCl₃) δ 7.05-6.96 (m, 1 H), 6.72-6.65 (m, 1 H), 6.61-6.53 (m, 1 H), 5.11 (s, 1 H), 5.02 (s, 1 H), 4.48 (s, 2 H), 1.84 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃) δ 159.8, 157.4, 150.2, 147.8, 147.3, 139.9, 116.3, 116.2, 116.1, 116.0, 113.5, 106.8, 106.8, 106.6, 106.5, 103.3, 103.0, 73.1, 19.2.

HRMS m/z (CI) calcd. for C₁₀H₁₀F₂OK (M+K)⁺ 223.1683, found 223.2065.

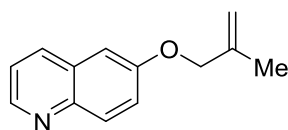


7,7'-Bis((2-methylallyl)oxy)-1,1'-binaphthalene (1l) was synthesized from (*R*)-(+)-1,1'-bi(2-naphthol) and 3-bromo-2-methylpropene according to reported procedure.¹⁰

¹H NMR (400 MHz, CDCl₃) δ 7.93 (s, 1 H), 7.91 (s, 1 H), 7.86 (s, 1 H), 7.84 (s, 1 H), 7.39 (s, 1 H), 7.37 (s, 1 H), 7.33-7.27 (m, 2 H), 7.23-7.14 (m, 4 H), 4.69 (d, J = 7.2 Hz, 4 H), 4.38 (s, 4 H), 1.41 (s, 6 H).

¹³C NMR (100 MHz, CDCl₃) δ 154.1, 141.2, 134.2, 129.2, 129.1, 127.8, 126.1, 125.5, 123.5, 120.2, 115.4, 111.7, 72.8, 18.9.

HRMS m/z (CI) calcd. for C₂₈H₂₆O₂Na (M+Na)⁺ 417.1831, found 417.1828.

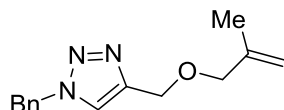


6-((2-Methylallyl)oxy)quinoline (1m) was synthesized from 6-hydroxyquinoline and 3-bromo-2-methylpropene according to reported procedure.¹⁰

¹H NMR (400 MHz, CDCl₃) δ 8.77-8.74 (m, 1 H), 8.04-7.98 (m, 2 H), 7.42-7.38 (m, 1 H), 7.35-7.30 (m, 1 H), 7.07 (d, J = 3.2 Hz, 1 H), 5.15 (s, 1 H), 5.03 (s, 1 H), 4.55 (s, 2 H), 1.87 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃) δ 156.8, 148.0, 144.4, 140.4, 134.8, 130.8, 129.2, 122.5, 121.3, 113.0, 106.4, 71.9, 19.4.

HRMS m/z (CI) calcd. for C₁₃H₁₄NO (M+H)⁺ 200.1075, found 200.1067.

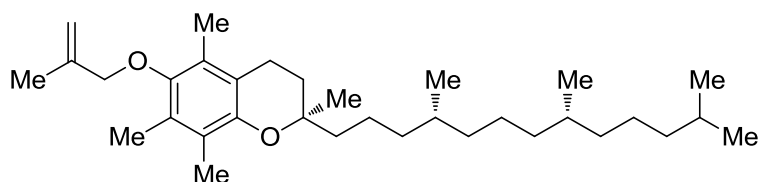


1-Benzyl-4-((2-methylallyl)oxy)methyl-1H-1,2,3-triazole (1s) was synthesized from intermediate (1-benzyl-1H-1,2,3-triazol-4-yl)methanol and 3-bromo-2-methylpropene according to reported procedure,⁹ and the intermediate was synthesized from benzyl azide and propargyl alcohol according to reported procedure.¹¹

¹H NMR (400 MHz, CDCl₃) δ 7.38 (s, 1 H), 7.30-7.28 (m, 3 H), 7.21-1.87 (m, 2 H), 5.44 (s, 2 H), 4.89 (s, 1 H), 4.83 (s, 1 H), 4.52 (s, 2 H), 3.88 (s, 2 H), 1.65 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 145.7, 141.7, 134.5, 129.0, 128.7, 128.1, 122.2, 112.6, 74.4, 63.4, 54.1, 19.4.

HRMS m/z (CI) calcd. for C₁₄H₁₈N₃O (M+H)⁺ 244.1450, found 244.1460.

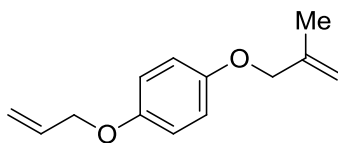


(R)-2,5,7,8-Tetramethyl-6-((2-methylallyl)oxy)-2-((4R,8R)-4,8,12-trimethyltridecyl)chroman (1z) was synthesized from vitamin E and 3-bromo-2-methylpropene according to reported procedure.¹⁰

¹H NMR (400 MHz, CDCl₃) δ 5.18 (s, 1 H), 4.97 (s, 1 H), 4.06 (s, 2 H), 2.58 (t, J = 5.6 Hz, 2 H), 2.18 (s, 3 H), 2.13 (s, 3 H), 2.09 (s, 3 H), 1.89 (s, 3 H), 1.86-1.70 (m, 2 H), 1.57-1.49 (m, 3 H), 1.48-1.31 (m, 4 H), 1.29-1.00 (m, 16 H), 0.88 (s, 3 H), 0.87-0.84 (m, 9 H).

¹³C NMR (100 MHz, CDCl₃) δ 148.2, 147.8, 142.0, 127.9, 125.9, 122.8, 117.5, 111.8, 76.4, 74.8, 40.0, 39.4, 37.5, 37.4, 37.3, 32.8, 32.7, 31.3, 28.0, 24.8, 24.4, 23.9, 22.7, 22.6, 21.0, 20.7, 19.8, 19.8, 19.7, 12.7, 11.8, 11.8..

HRMS m/z (CI) calcd. for C₃₃H₅₇O₂ (M+H)⁺ 485.4359, found 485.4352.

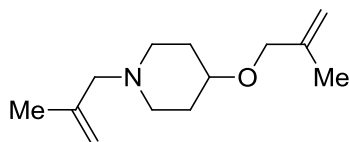


1-(Allyloxy)-4-((2-methylallyl)oxy)benzene (1aa) was synthesized from hydroquinone and 3-bromo-2-methylpropene according to reported procedure.¹⁰

¹H NMR (400 MHz, CDCl₃) δ 6.90 (s, 4 H), 6.08-6.02 (m, 1 H), 5.42 (d, J = 1.2 Hz, 0.5 H), 5.38 (d, J = 1.2 Hz, 0.5 H), 5.29 (d, J = 1.2 Hz, 0.5 H), 5.26 (d, J = 1.2 Hz, 0.5 H), 5.08 (s, 1 H), 4.98 (s, 3 H), 4.49 (d, J = 5.2 Hz, 2 H), 4.38 (s, 2 H), 1.83 (s, 3 H) .

¹³C NMR (100 MHz, CDCl₃) δ 153.1, 152.8, 141.2, 133.6, 117.4, 115.7, 115.6, 112.5, 72.4, 69.5, 19.4.

HRMS m/z (CI) calcd. for C₁₃H₁₇O₂ (M+H)⁺ 205.7071, found 205.7056.

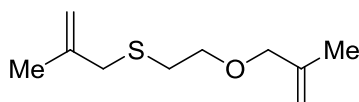


1-(2-Methylallyl)-4-((2-methylallyl)oxy)piperidine (1ab) was synthesized from 4-hydroxypiperidine and 3-bromo-2-methylpropene according to reported procedure.¹⁰

¹H NMR (400 MHz, CDCl₃) δ 4.97 (s, 4 H), 4.87 (s, 2 H), 4.83 (s, 1 H), 3.90 (s, 2 H), 3.32 (s, 1 H), 2.85 (s, 2 H), 2.69(2.69, *J* = 5.2 Hz, 2 H), 2.04 (s, 2 H), 1.87 (s, 2 H), 1.74 (s, 6 H), 1.61 (t, *J* = 9.6 Hz, 2 H) .

¹³C NMR (100 MHz, CDCl₃) δ 143.2, 142.9, 112.5, 111.6, 71.7, 65.3, 51.3, 31.4, 20.9, 19.5.

HRMS *m/z* (CI) calcd. for C₁₃H₂₄NO₂ (M+H)⁺ 210.1858, found 210.1843.

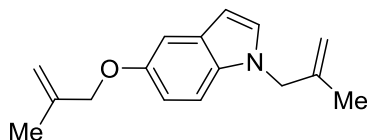


(2-Methylallyl)(2-((2-methylallyl)oxy)ethyl)sulfane (1ac) was synthesized from 2-mercaptoethanol and 3-bromo-2-methylpropene according to reported procedure.¹⁰

¹H NMR (400 MHz, CDCl₃) δ 4.96 (d, *J* = 3.2 Hz, 1 H), 4.89 (s, 1 H), 4.85 (d, *J* = 1.2 Hz, 1 H), 4.83 (d, *J* = 1.2 Hz, 1 H), 3.14 (s, 2 H), 2.63 (t, *J* = 3.2 Hz, 2 H), 1.82 (3, 3 H), 1.74 (s, 3 H) .

¹³C NMR (100 MHz, CDCl₃) δ 142.1, 141.3, 113.6, 112.2, 74.9, 69.2, 39.7, 30.2, 20.6, 19.4.

HRMS *m/z* (CI) calcd. for C₁₀H₁₉OS (M+H)⁺ 187.1176, found 187.1186.



1-(2-Methylallyl)-5-((2-methylallyl)oxy)-1H-indole (1ad) was synthesized from 5-Hydroxyindole and 3-bromo-2-methylpropene according to reported procedure.¹⁰

¹H NMR (400 MHz, CDCl₃) δ 7.20 (d, *J* = 8.8 Hz, 1 H), 7.10 (s, 1 H), 7.05 (d, *J* = 2.8 Hz, 1 H), 6.90 (d, *J* = 8.8 Hz, 1 H), 6.42 (d, *J* = 2.8 Hz, 1 H), 5.13 (s, 1 H), 4.98 (s, 1 H), 4.90 (s, 1 H), 4.72 (s, 1 H), 4.60 (s, 2 H), 4.47 (s, 2 H), 1.86 (s, 3 H), 1.66 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃) δ 153.2, 141.7, 141.3, 128.8, 112.6, 112.4, 112.3, 110.3, 104.0, 100.8, 72.7, 52.7, 19.8, 19.5.

HRMS *m/z* (CI) calcd. for C₁₆H₂₀NO (M+H)⁺ 242.1545, found 242.1569.

V. ^1H NMR and ^{13}C NMR Spectra

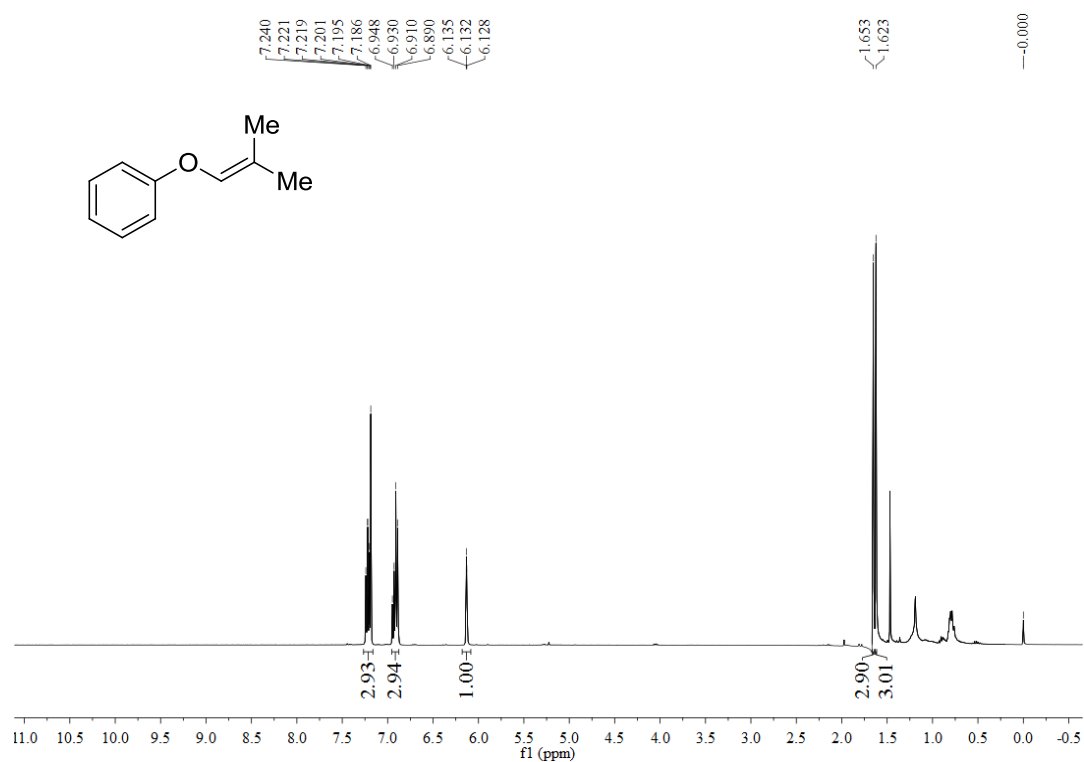


Figure S3. ^1H NMR spectra of **2a**.

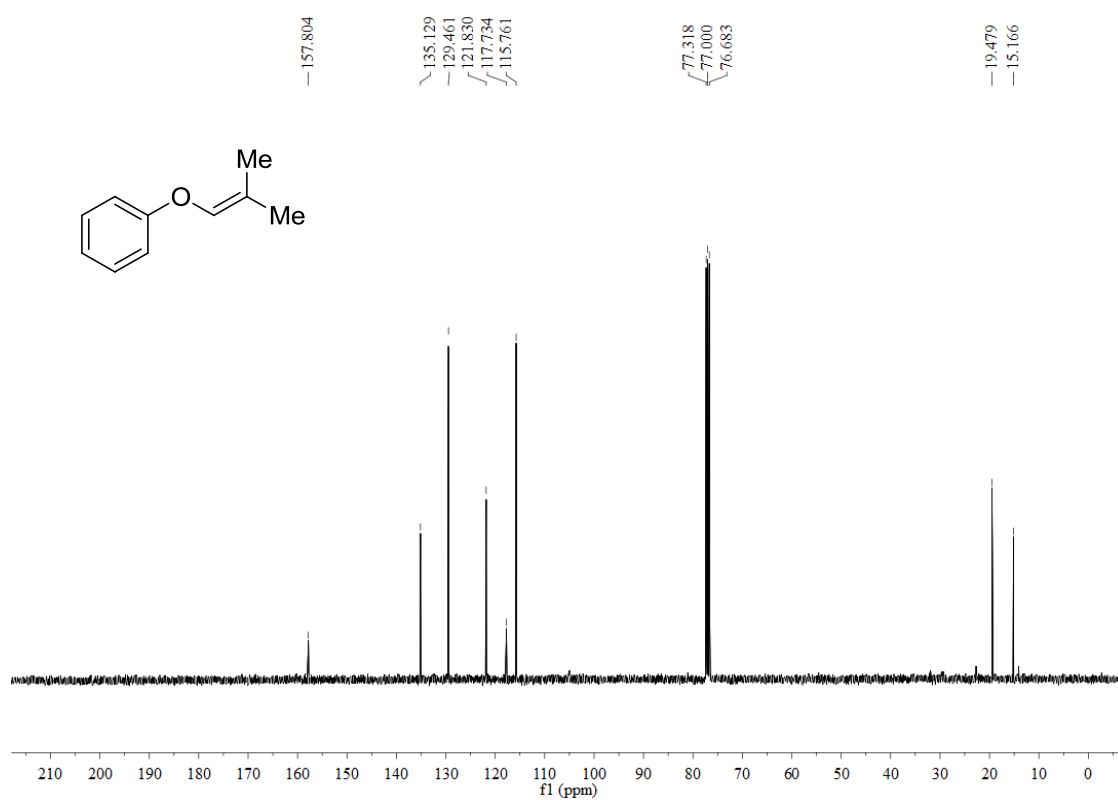


Figure S4. ^{13}C NMR spectra of **2a**.

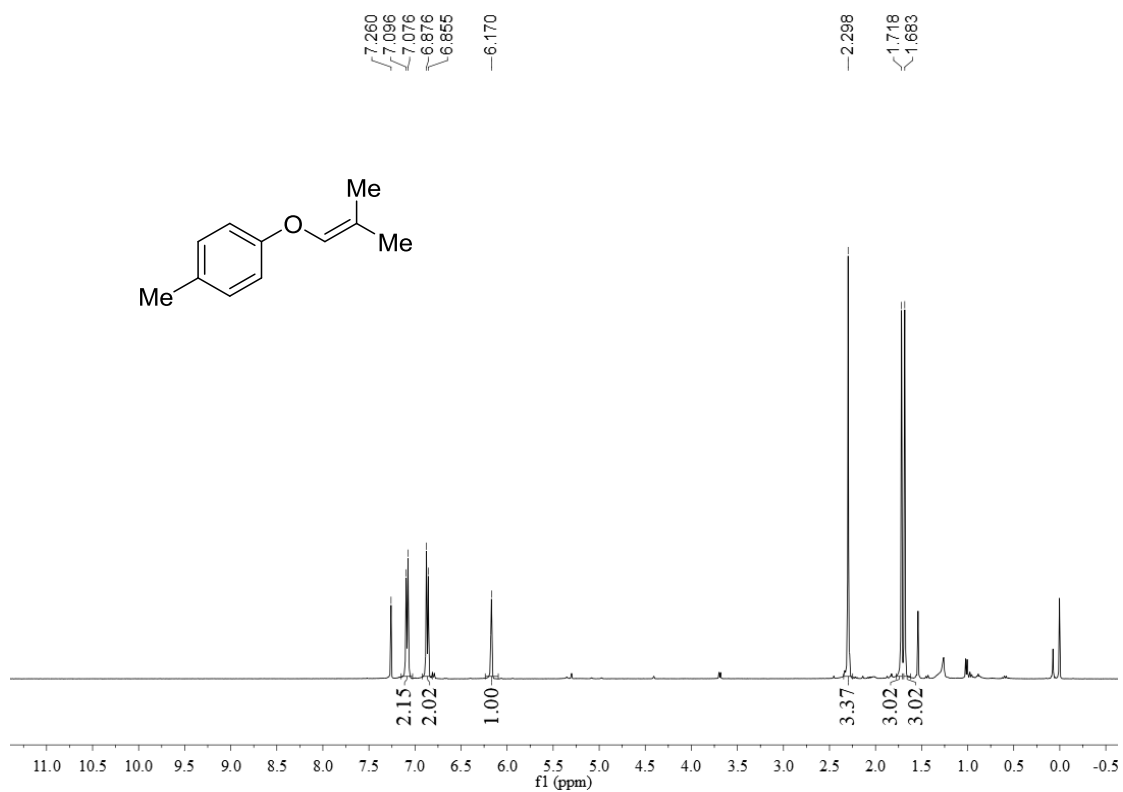


Figure S5. ¹H NMR spectra of **2b**.

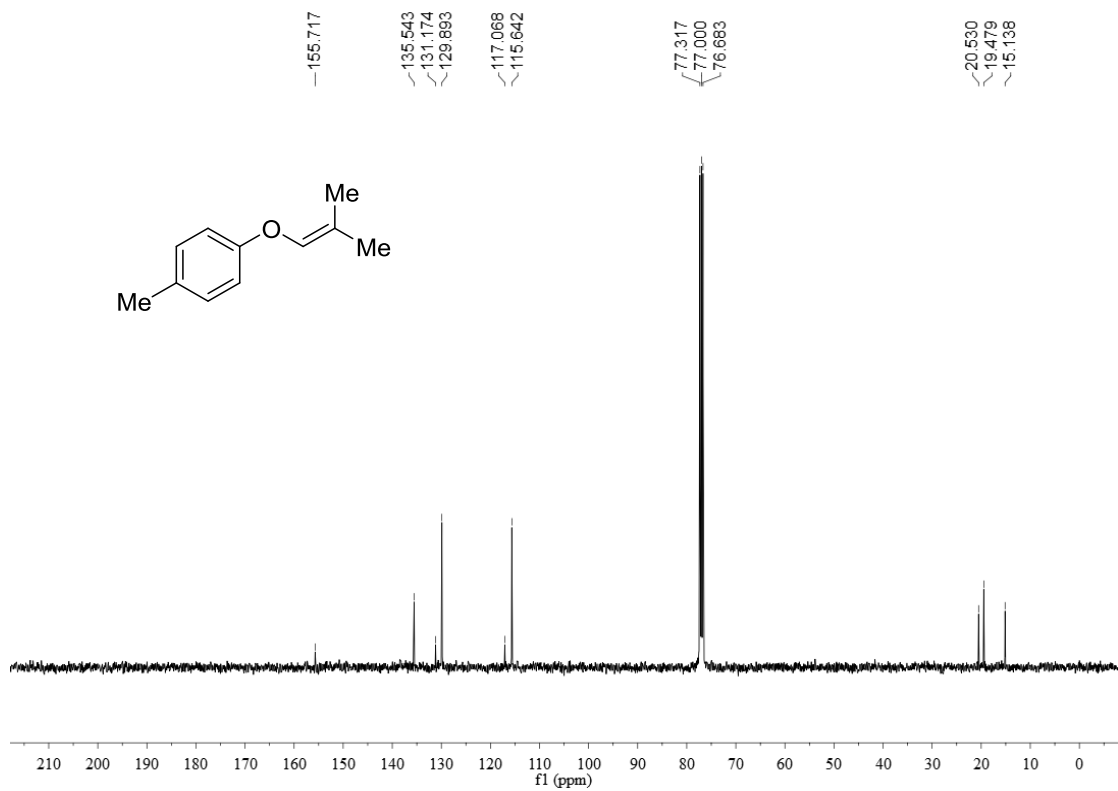


Figure S6. ¹³C NMR spectra of **2b**.

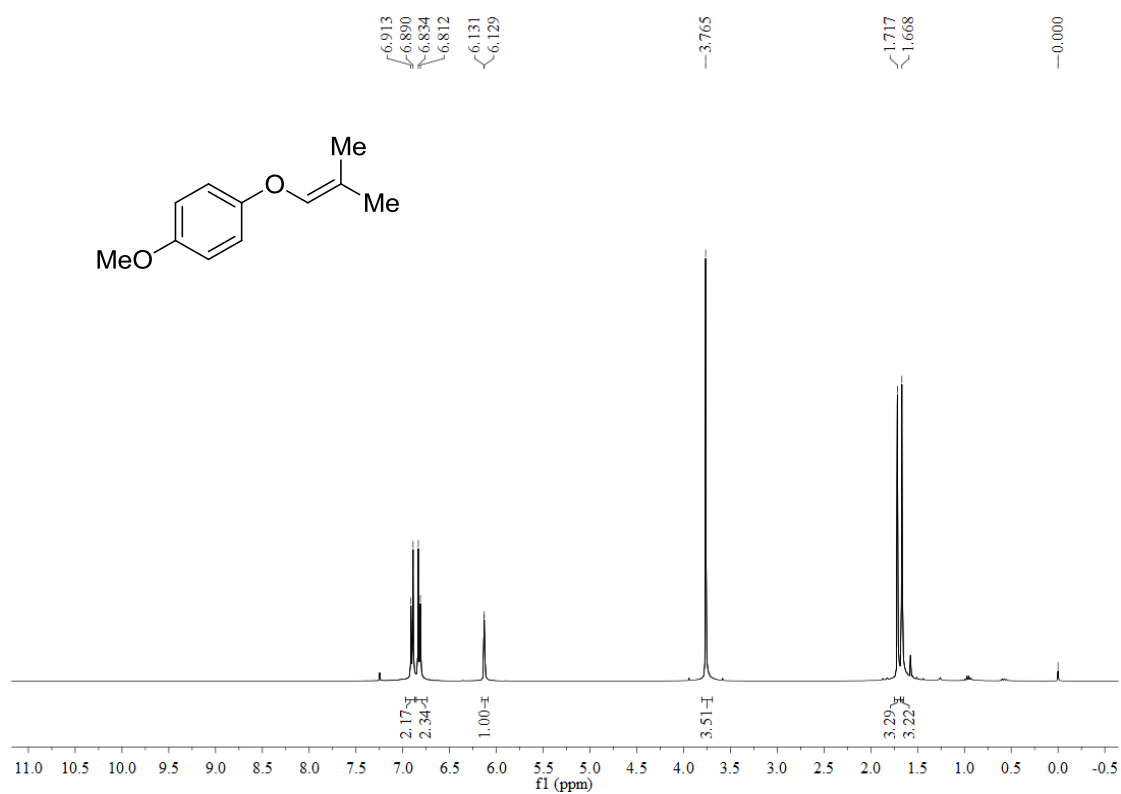


Figure S7. ¹H NMR spectra of **2c**.

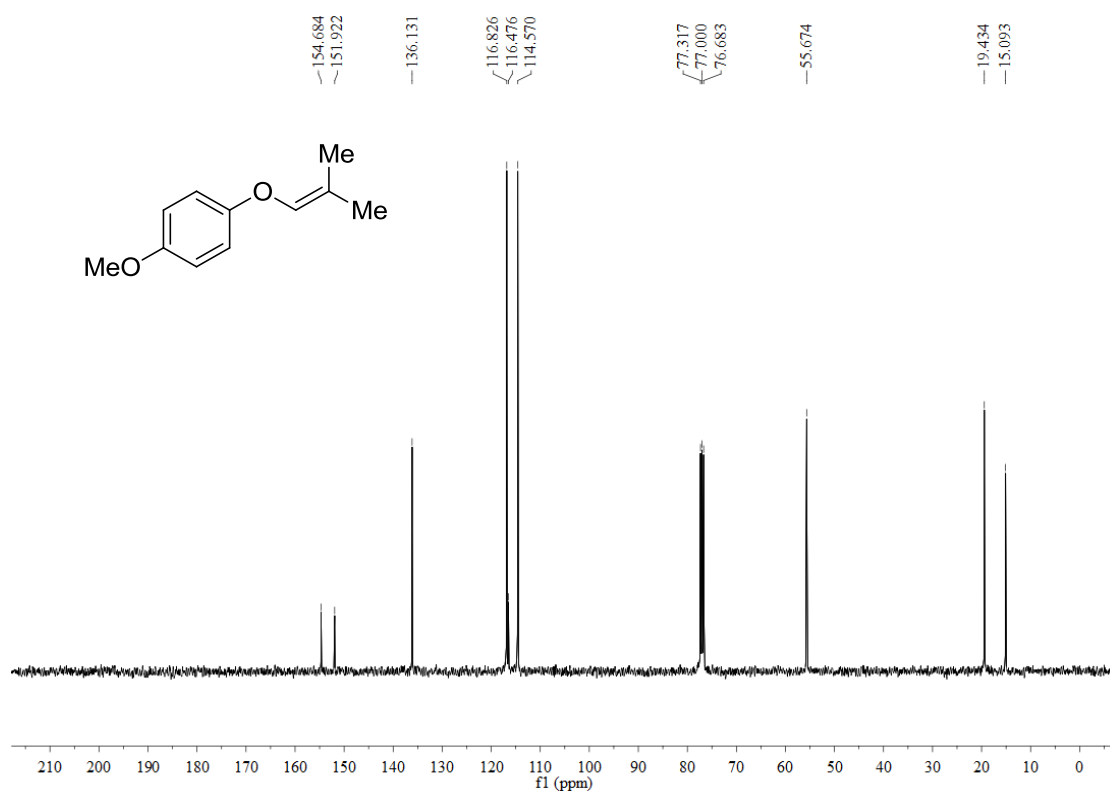


Figure S8. ¹³C NMR spectra of **2c**.

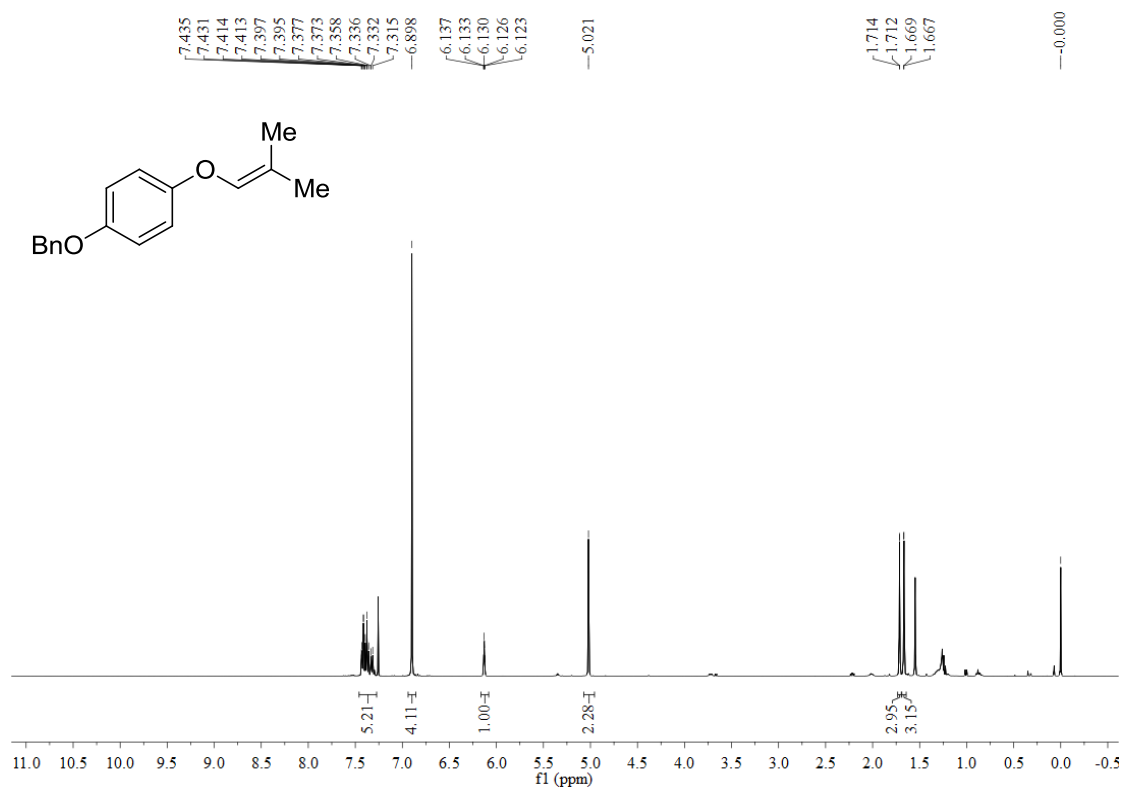


Figure S9. ¹H NMR spectra of **2d**.

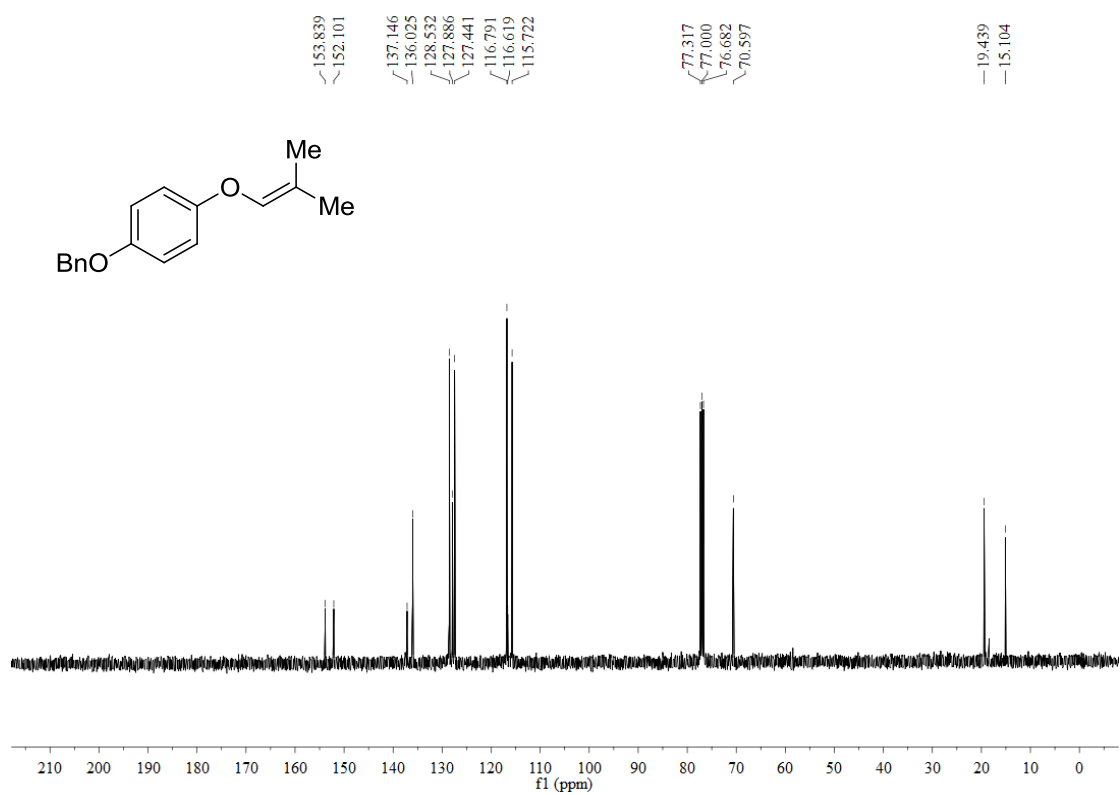


Figure S10. ¹³C NMR spectra of **2d**.

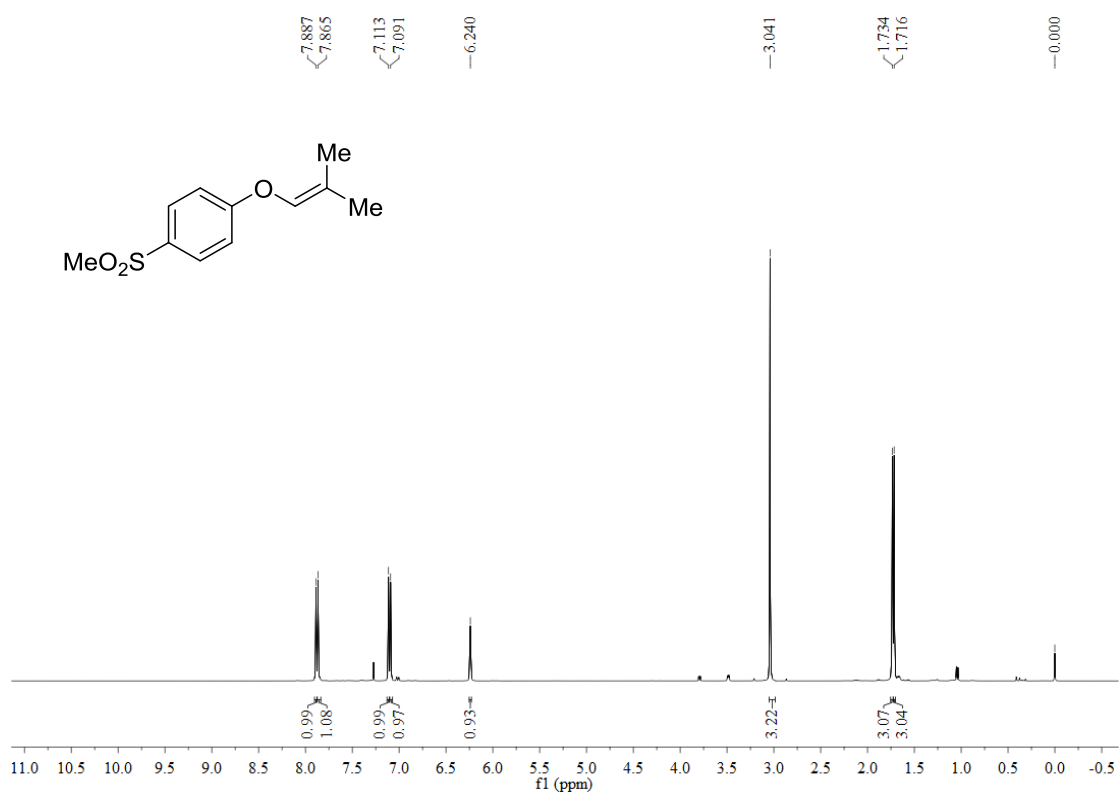


Figure S11. ¹H NMR spectra of **2e**.

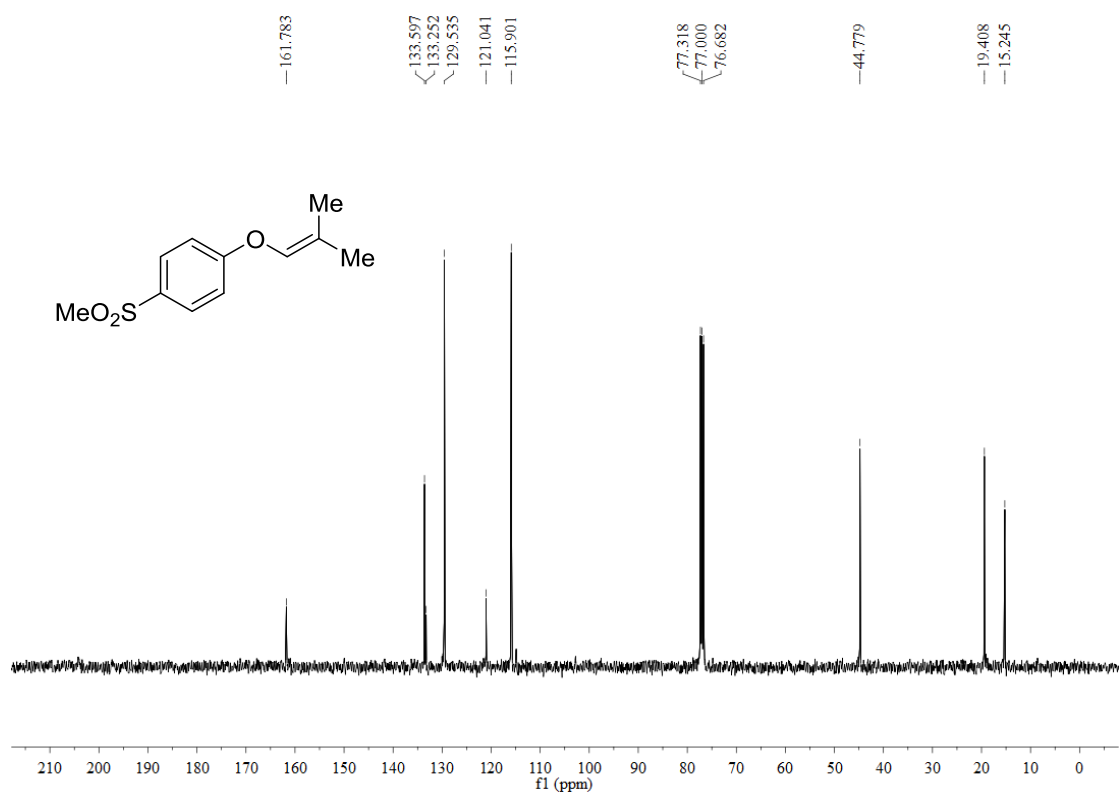


Figure S12. ¹³C NMR spectra of **2e**.

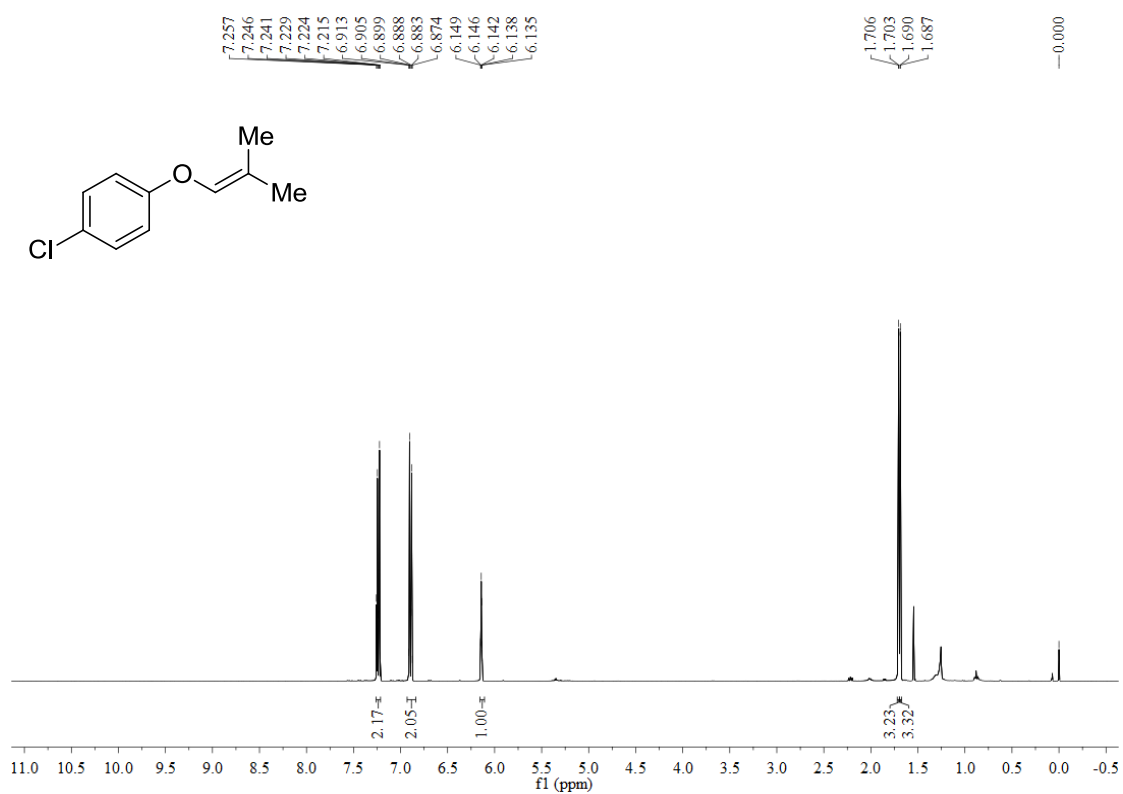


Figure S13. ¹H NMR spectra of **2f**.

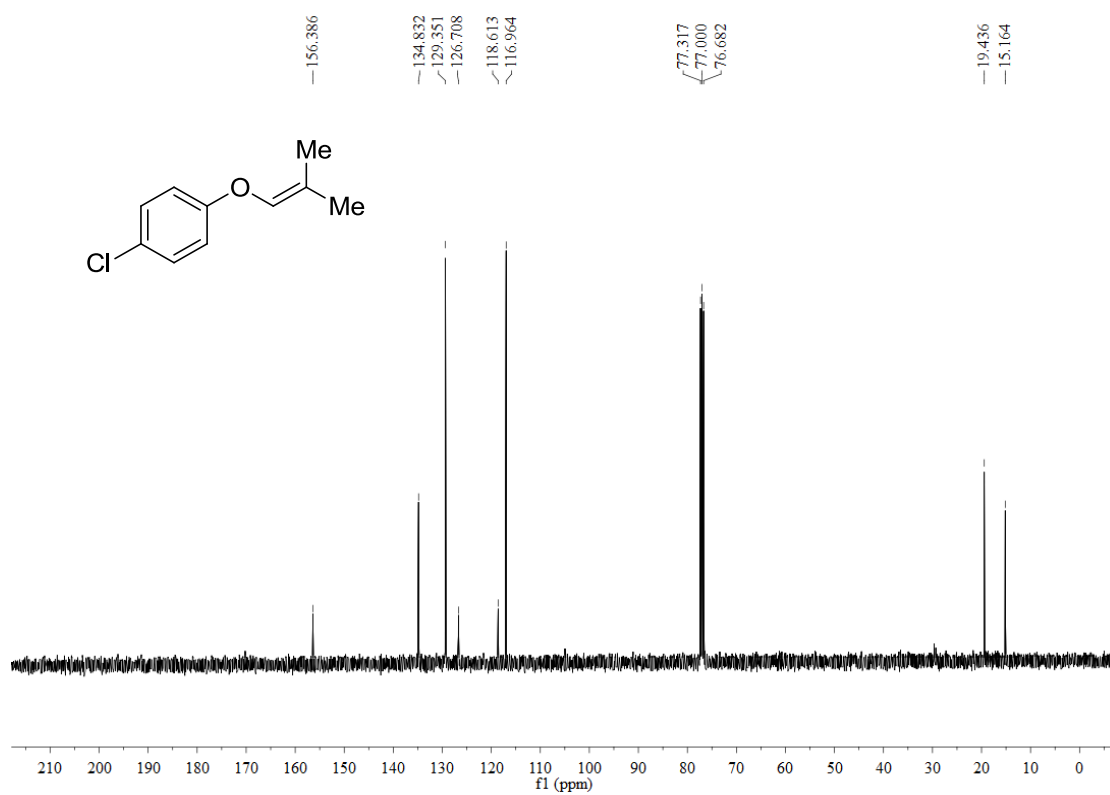


Figure S14. ¹³C NMR spectra of **2f**.

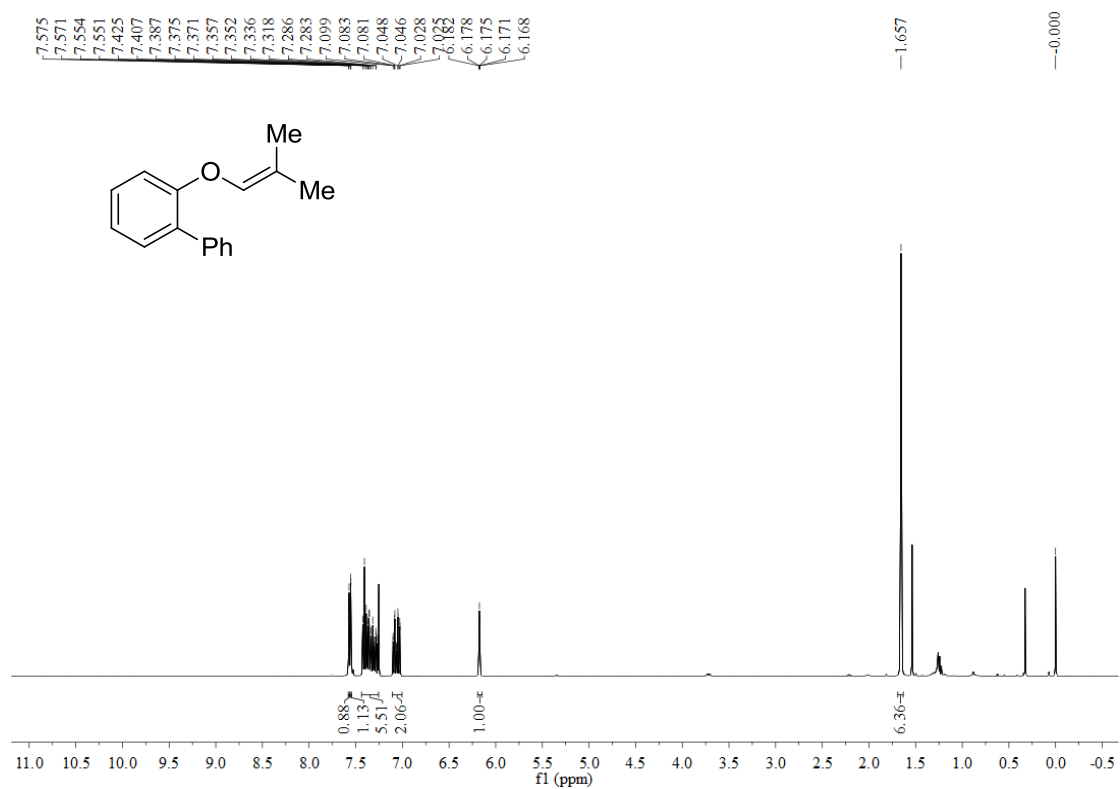


Figure S15. ¹H NMR spectra of **2g**.

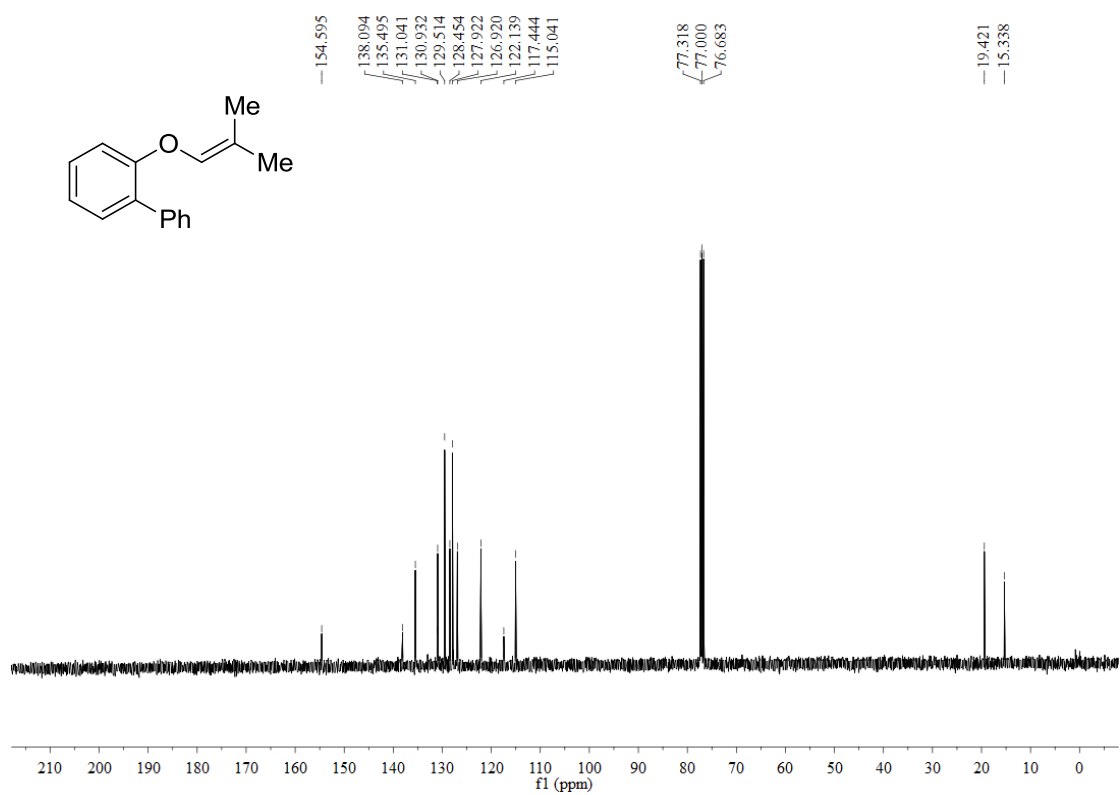


Figure S16. ¹³C NMR spectra of **2g**.

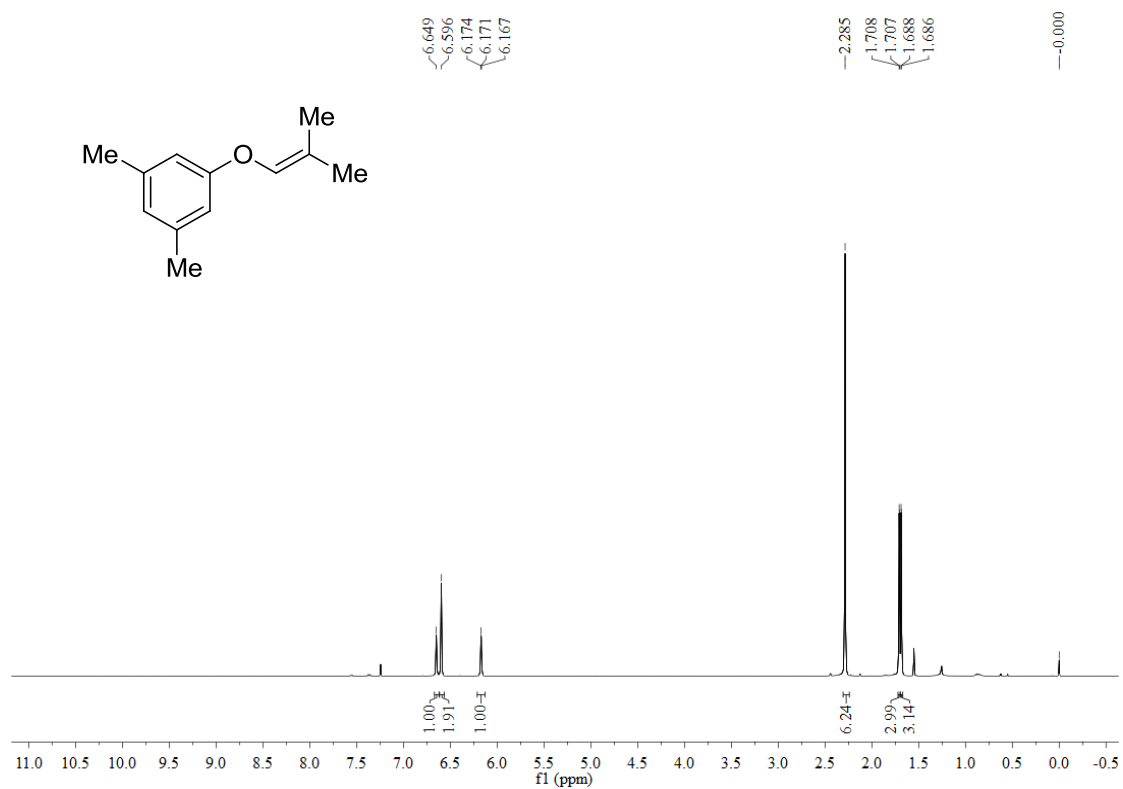


Figure S17. ¹H NMR spectra of **2h**.

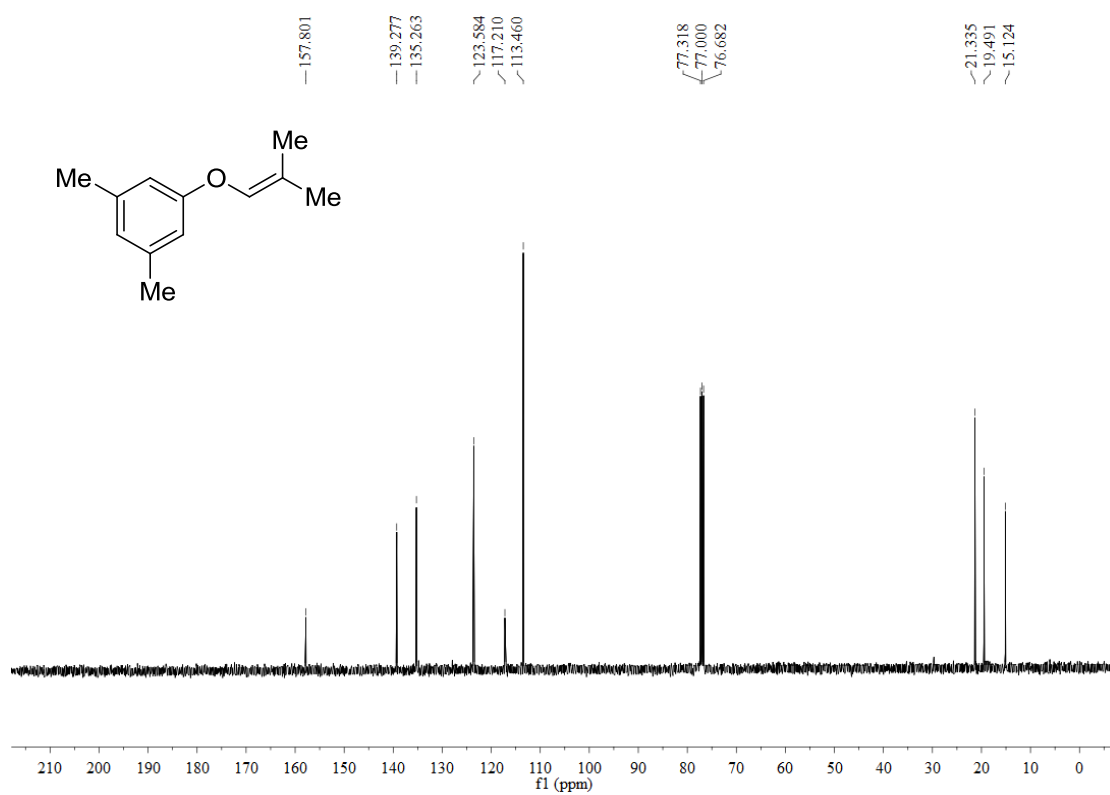


Figure S18. ¹³C NMR spectra of **2h**.

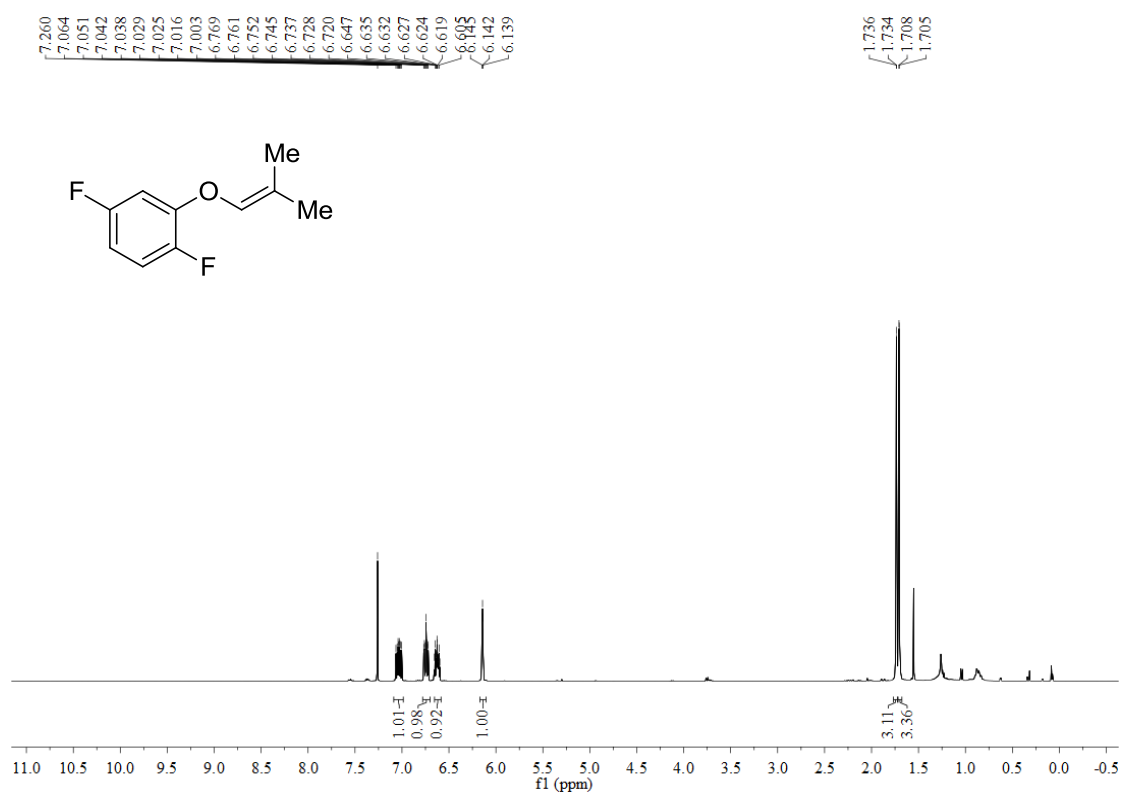


Figure S19. ¹H NMR spectra of **2i**.

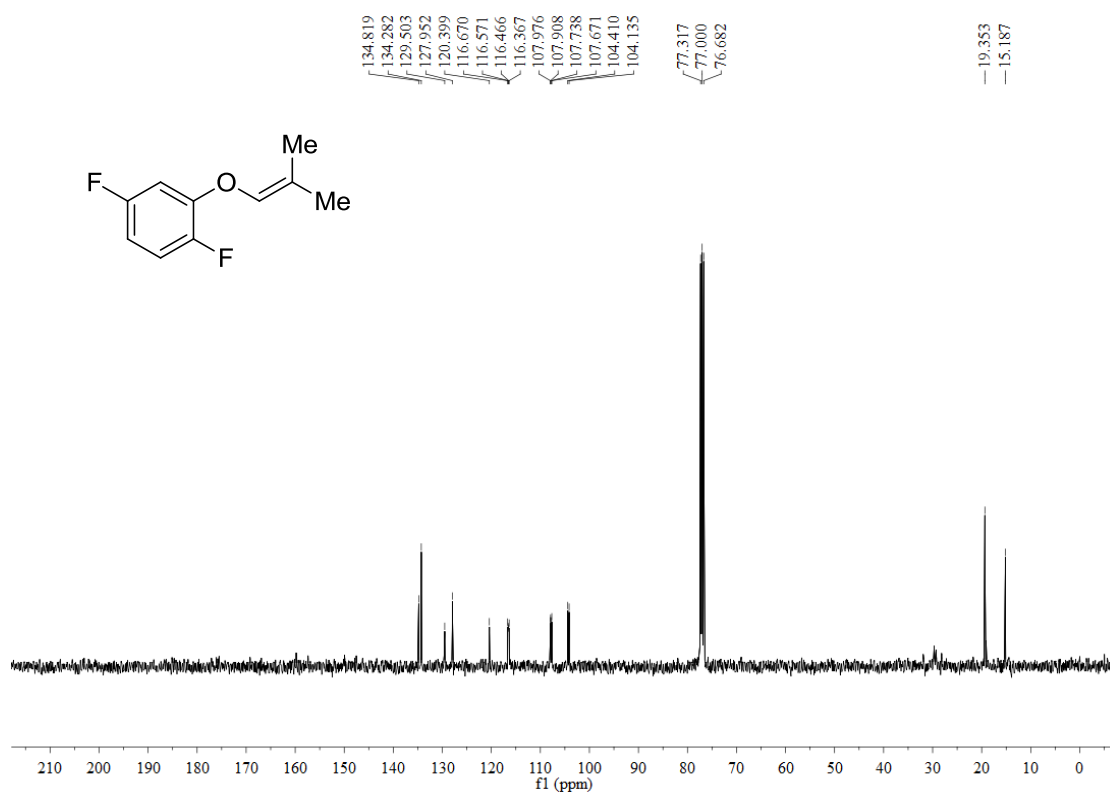


Figure S20. ¹³C NMR spectra of **2i**.

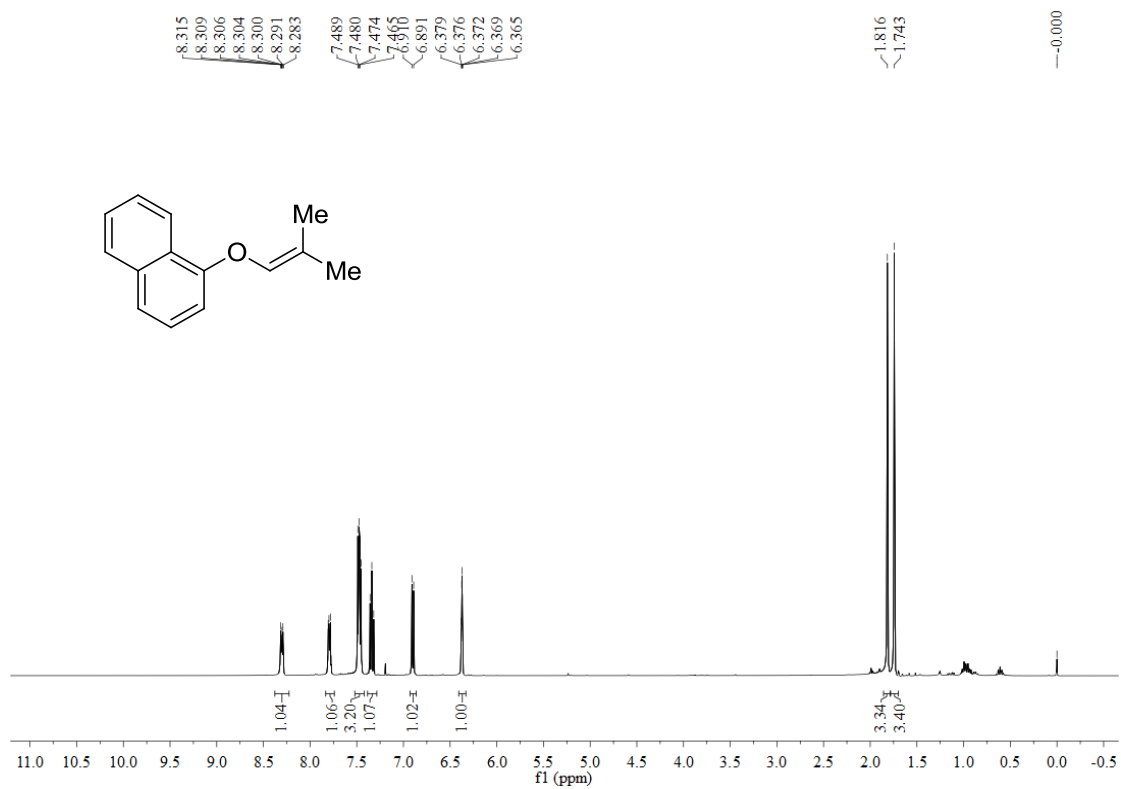


Figure S21. ¹H NMR spectra of **2j**.

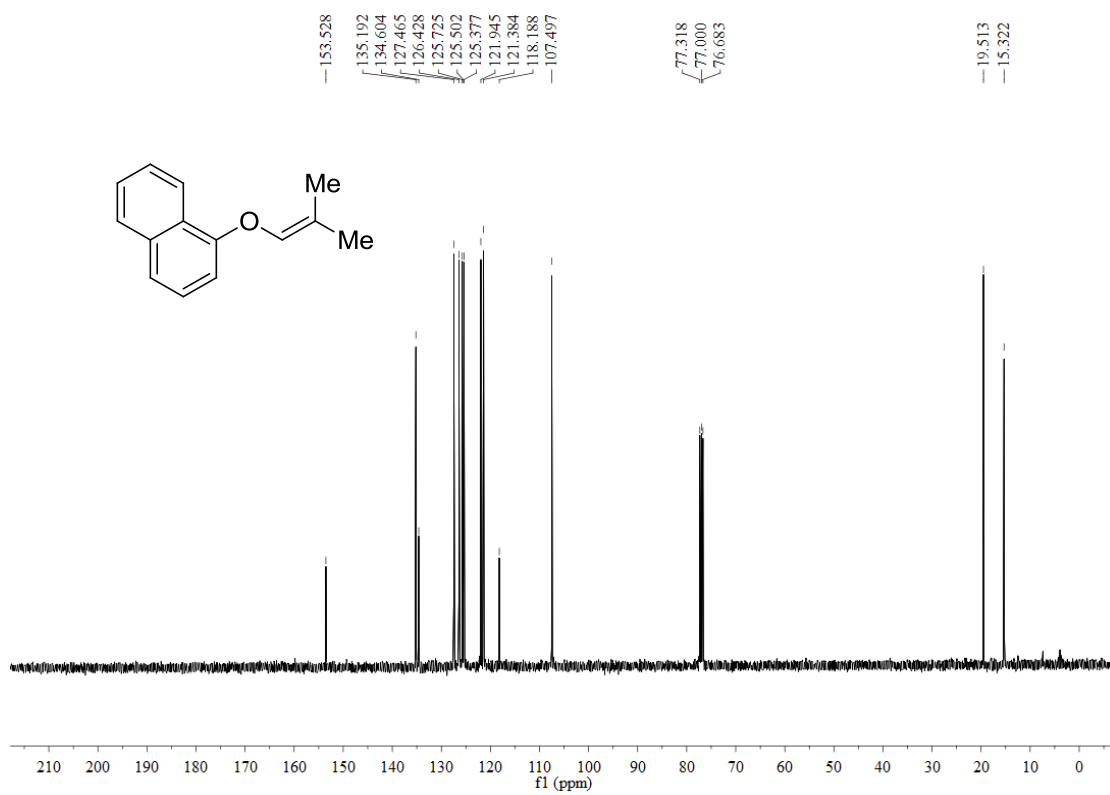


Figure S22. ¹³C NMR spectra of **2j**.

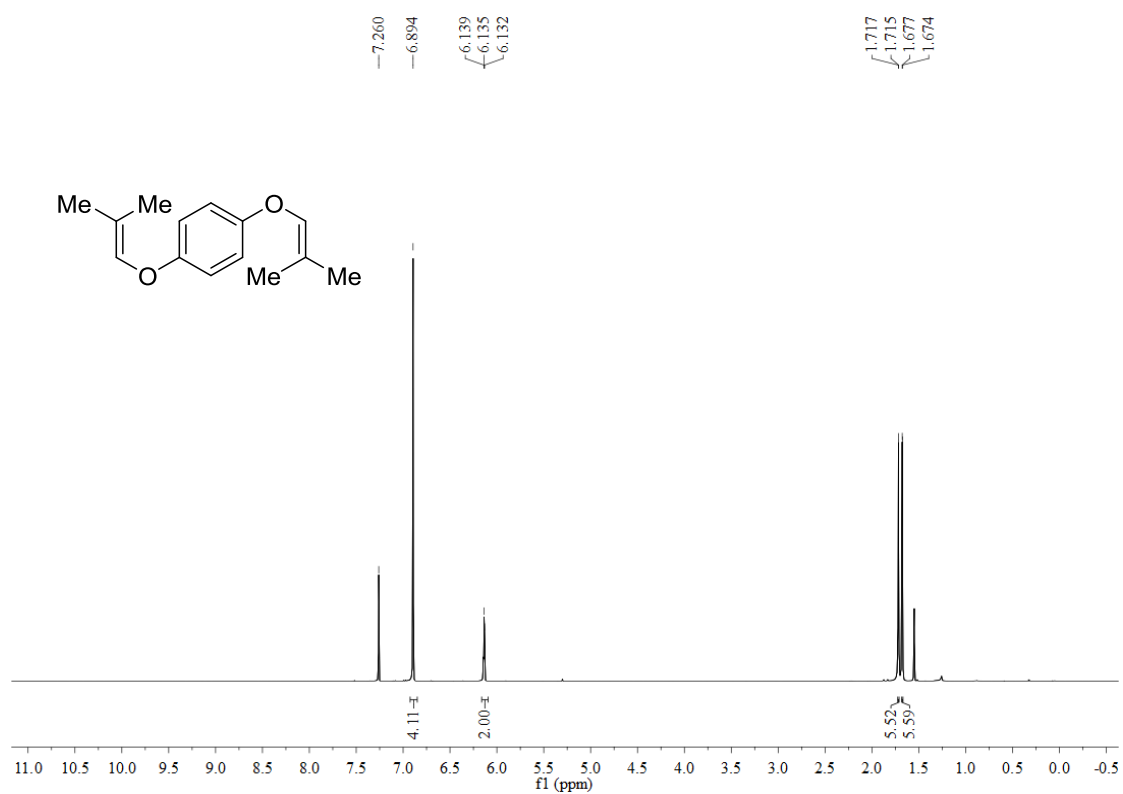


Figure S23. ¹H NMR spectra of **2k**.

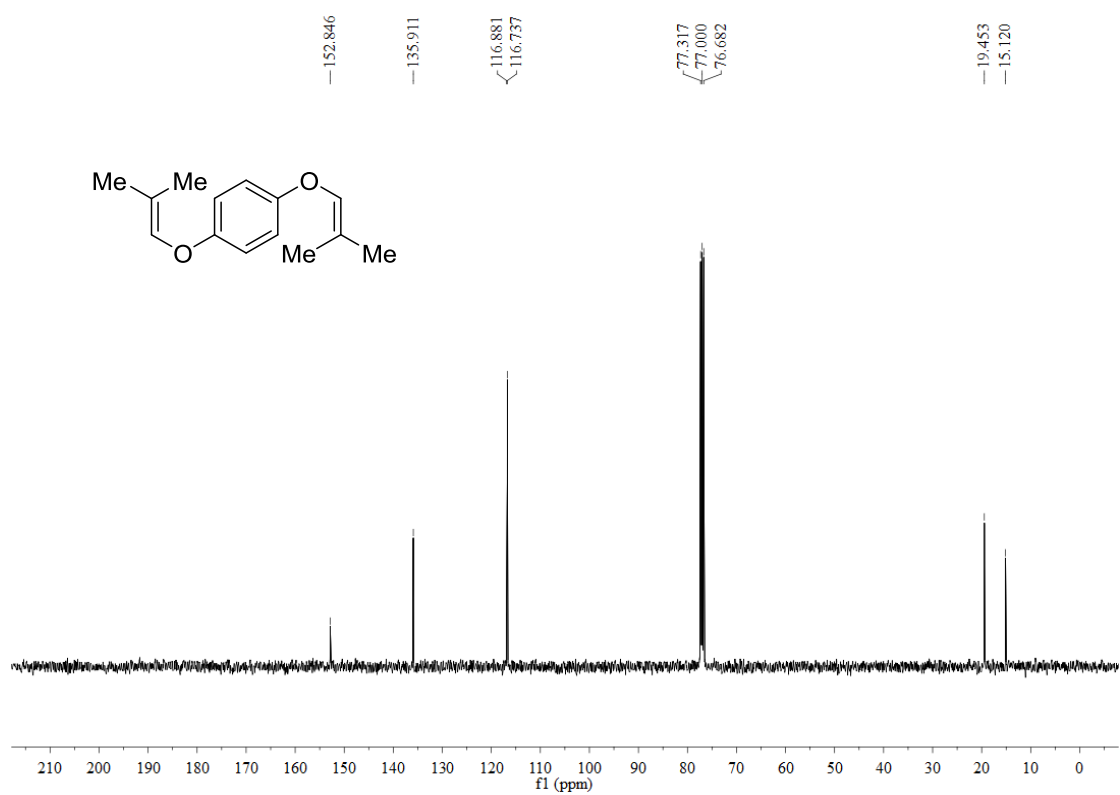


Figure S24. ¹³C NMR spectra of **2k**.

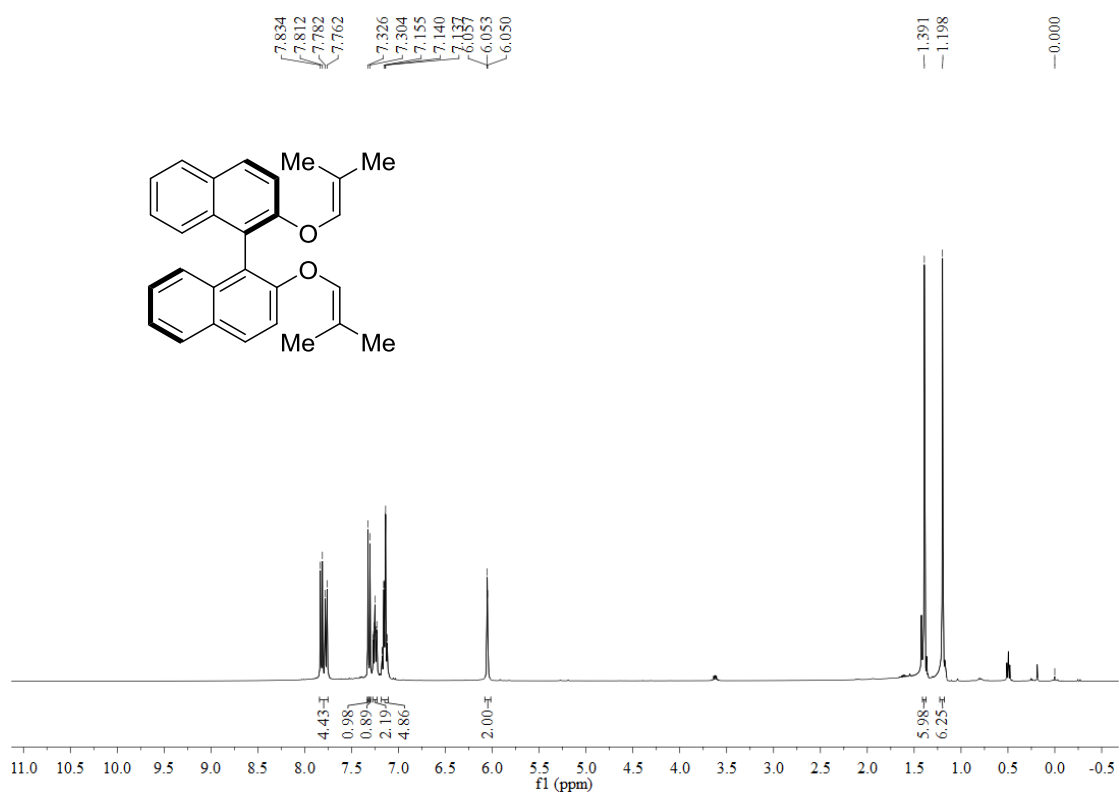


Figure S25. ¹H NMR spectra of 2I.

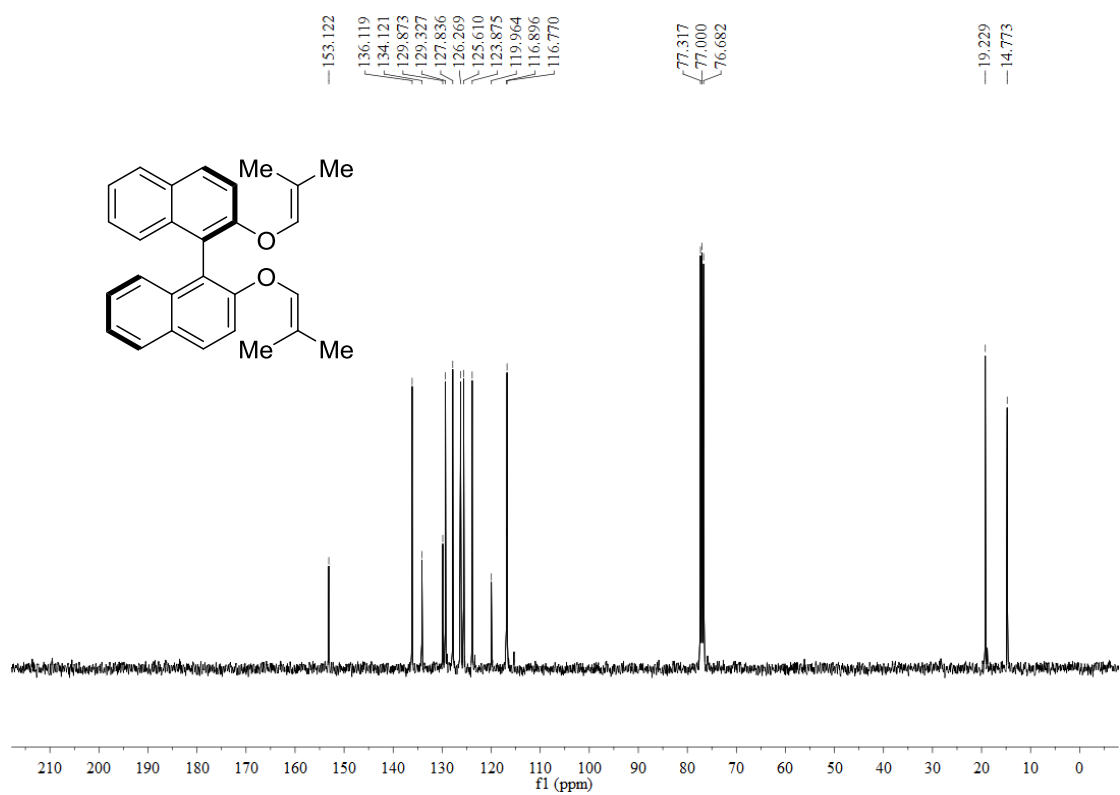


Figure S26. ¹³C NMR spectra of 2I.

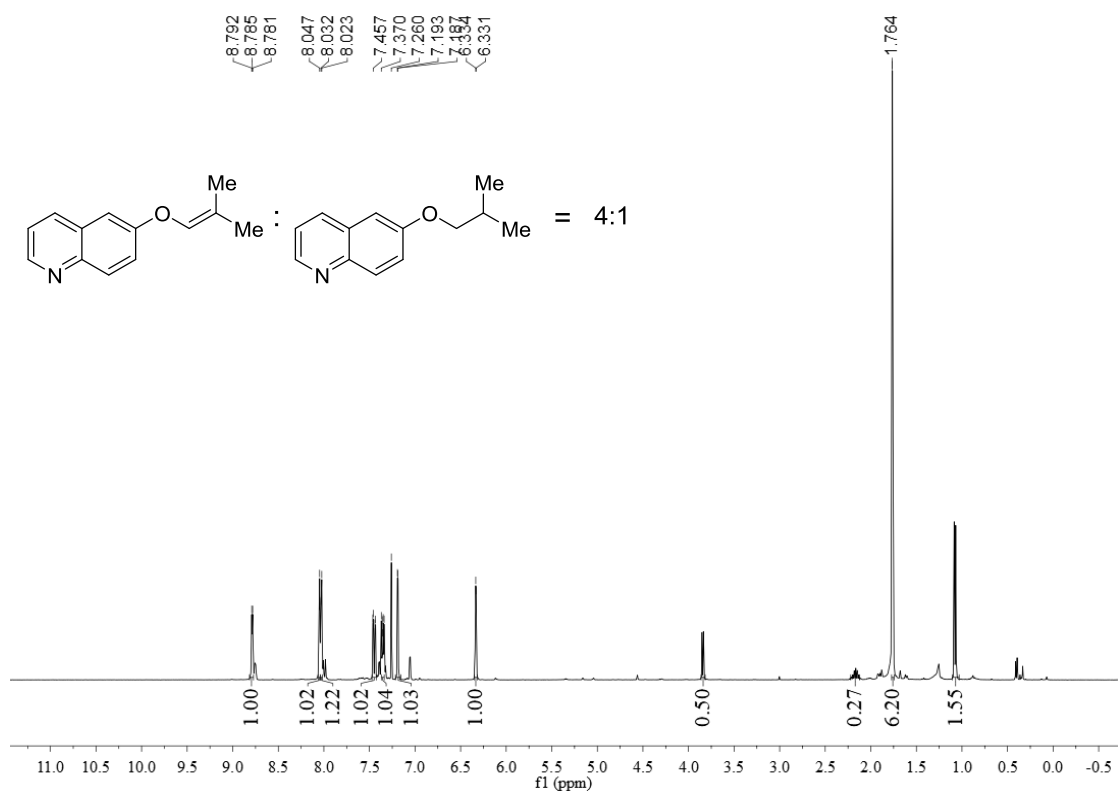


Figure S27. ^1H NMR spectra of **2m** & **3m** (4:1).

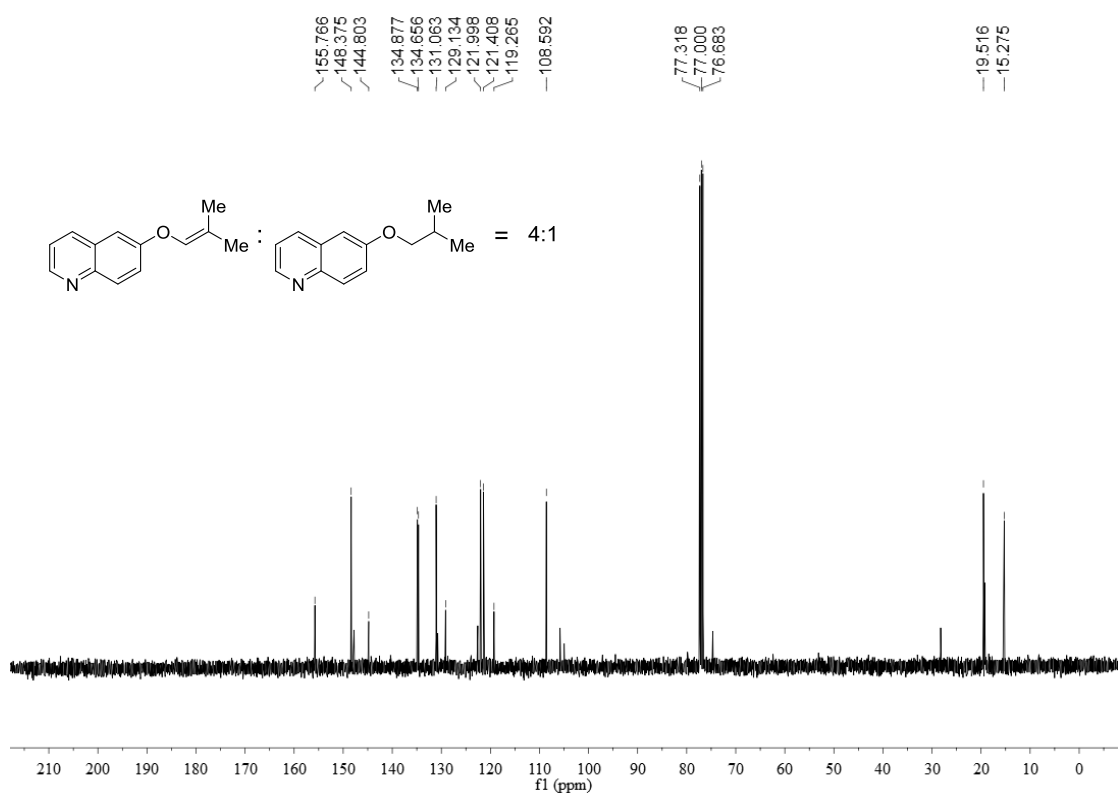


Figure S28. ^{13}C NMR spectra of **2m** & **3m**.

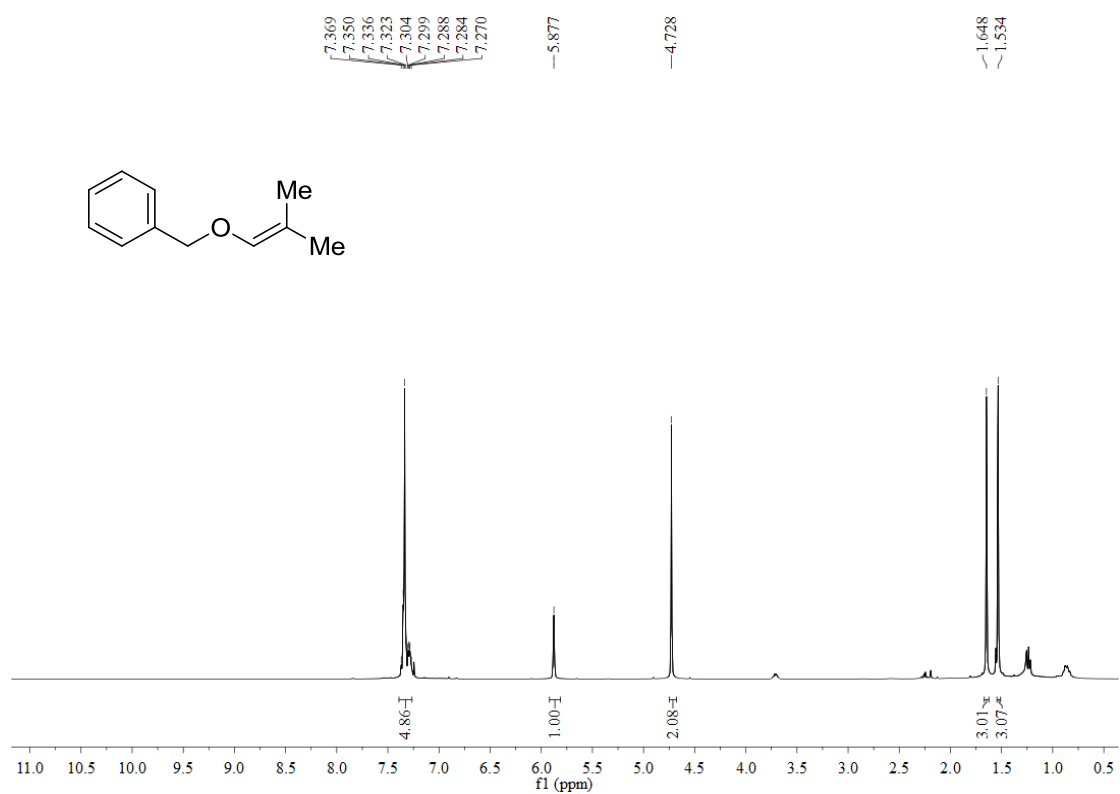


Figure S29. ¹H NMR spectra of 2n.

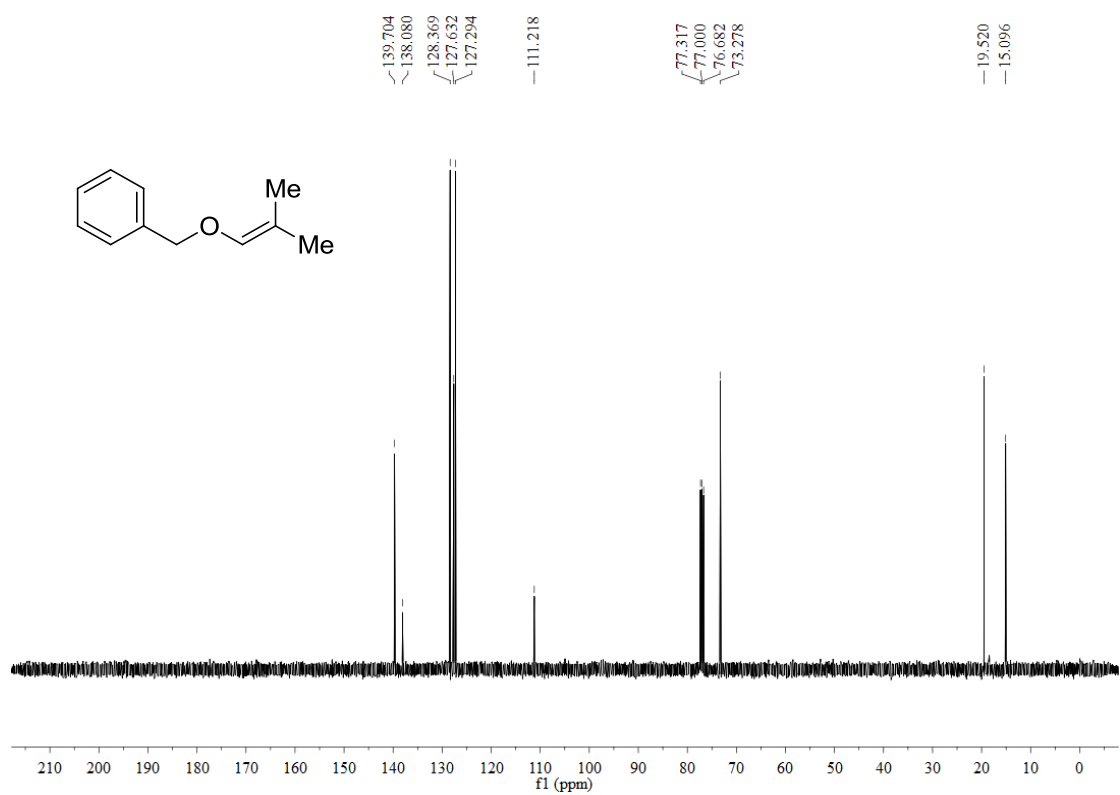


Figure S30. ¹³C NMR spectra of 2n.

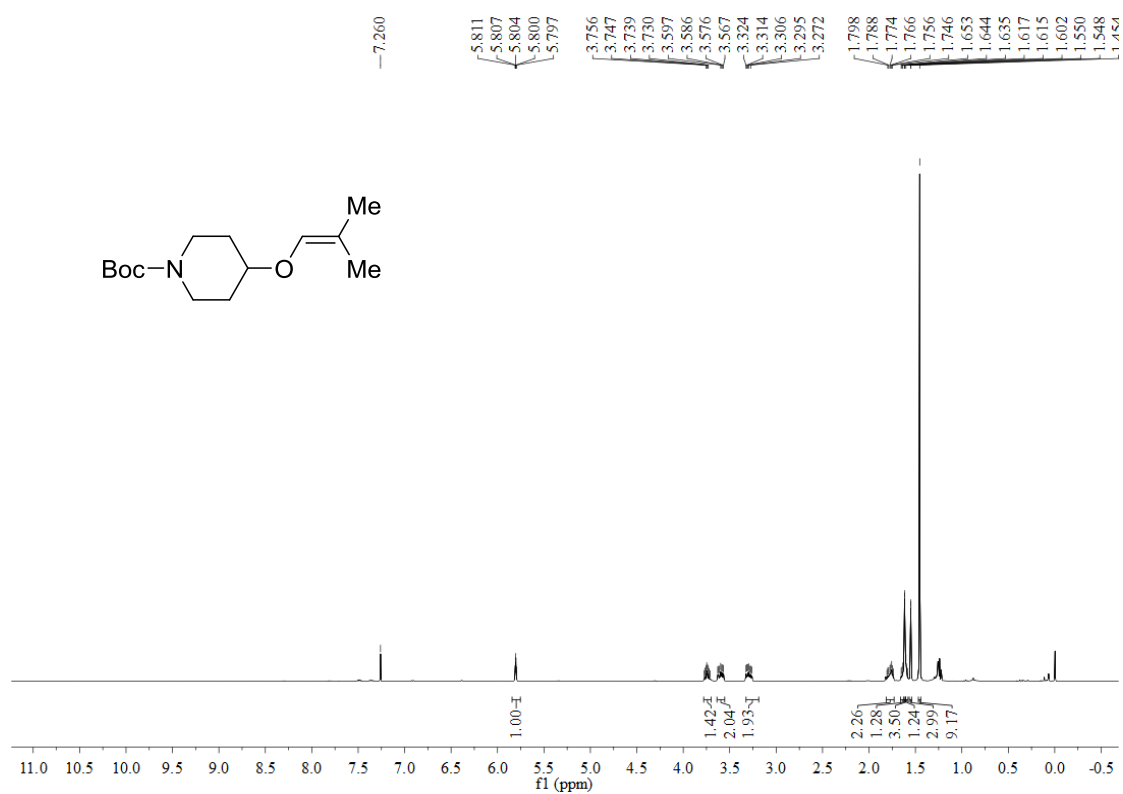


Figure S31. ¹H NMR spectra of **2o**.

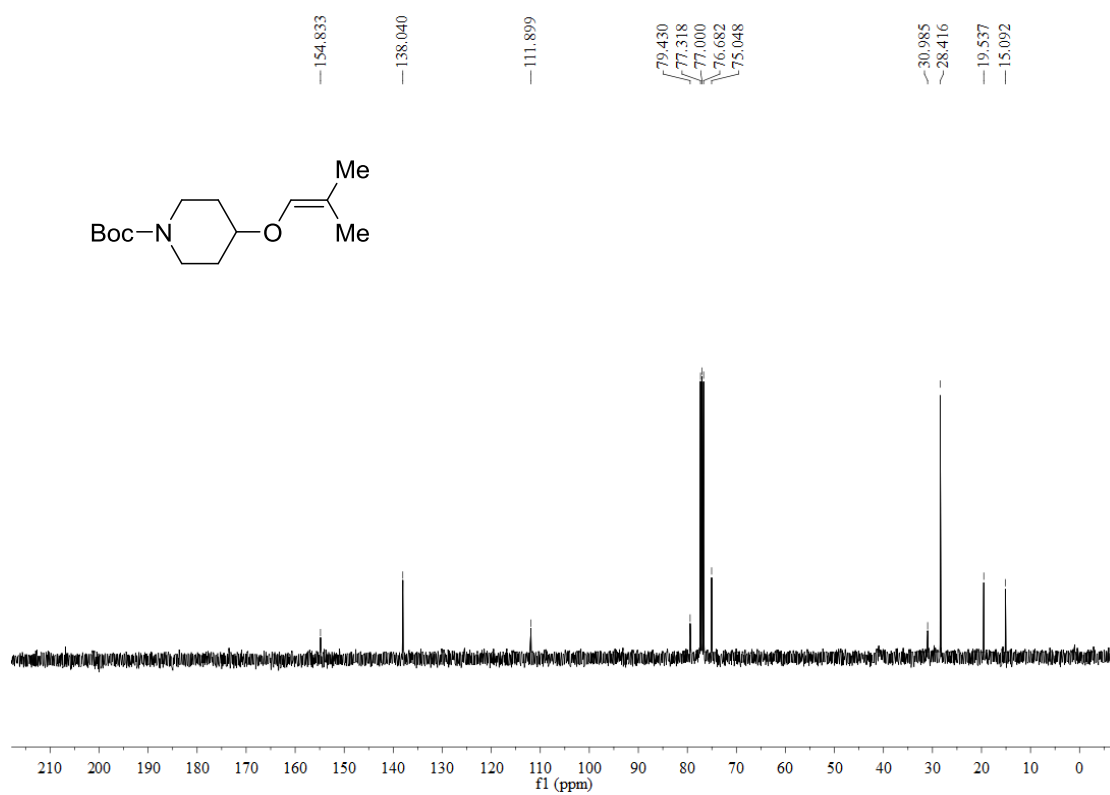


Figure S32. ¹³C NMR spectra of **2o**.

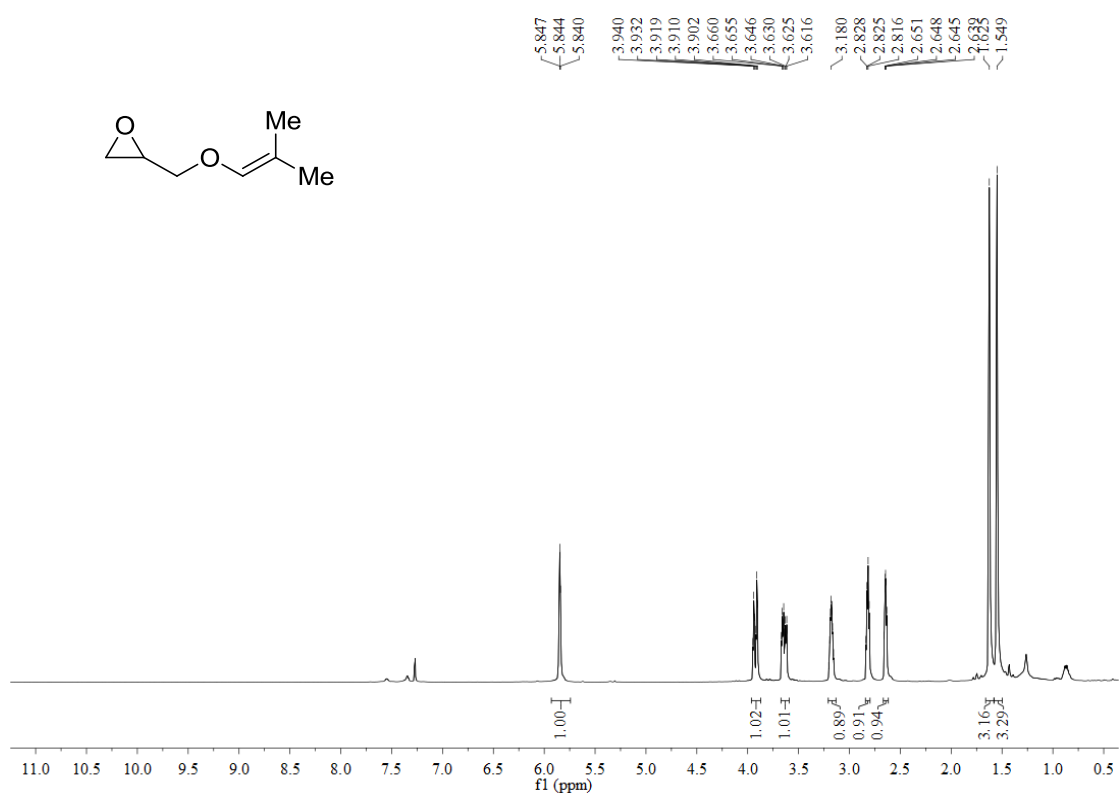


Figure S33. ¹H NMR spectra of **2p**.

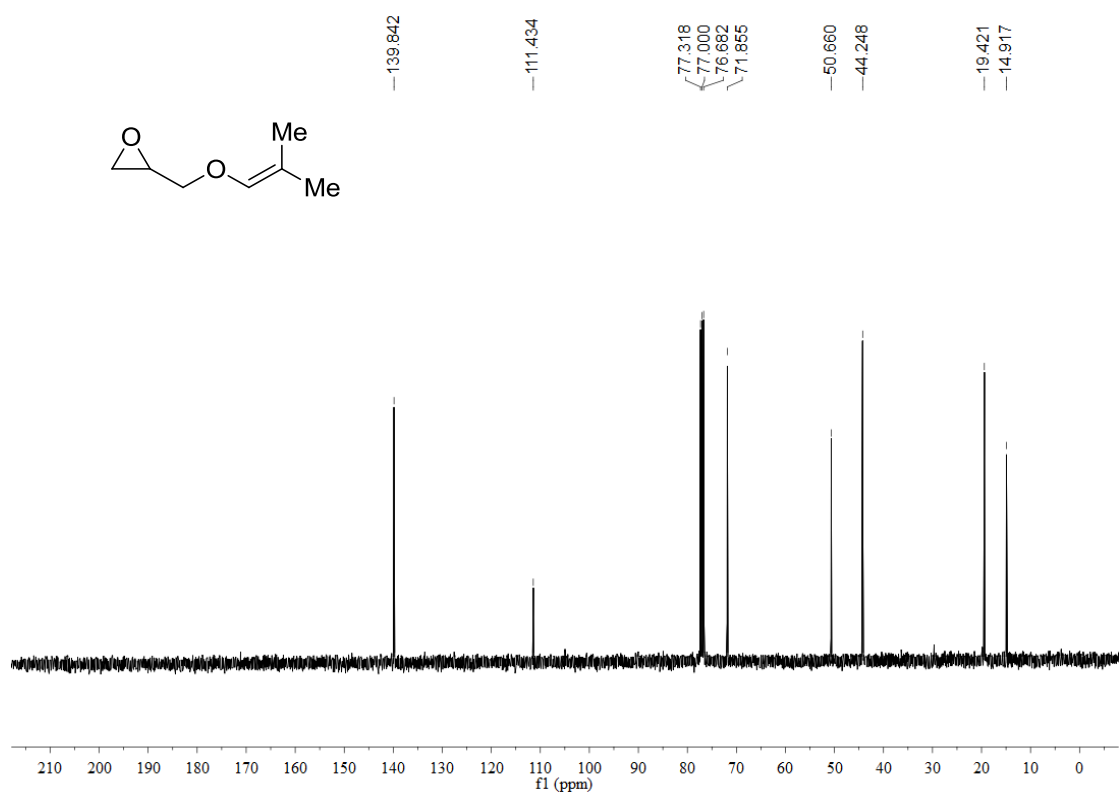


Figure S34. ¹³C NMR spectra of **2p**.

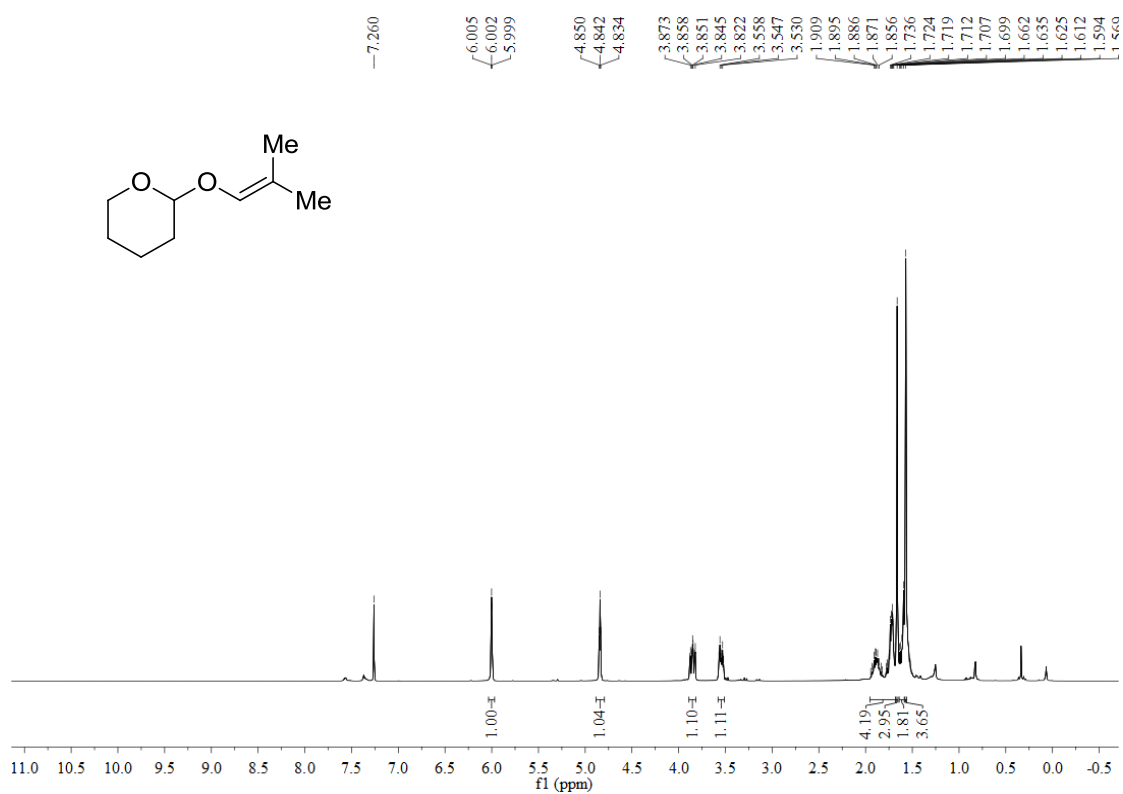


Figure S35. ¹H NMR spectra of 2q.

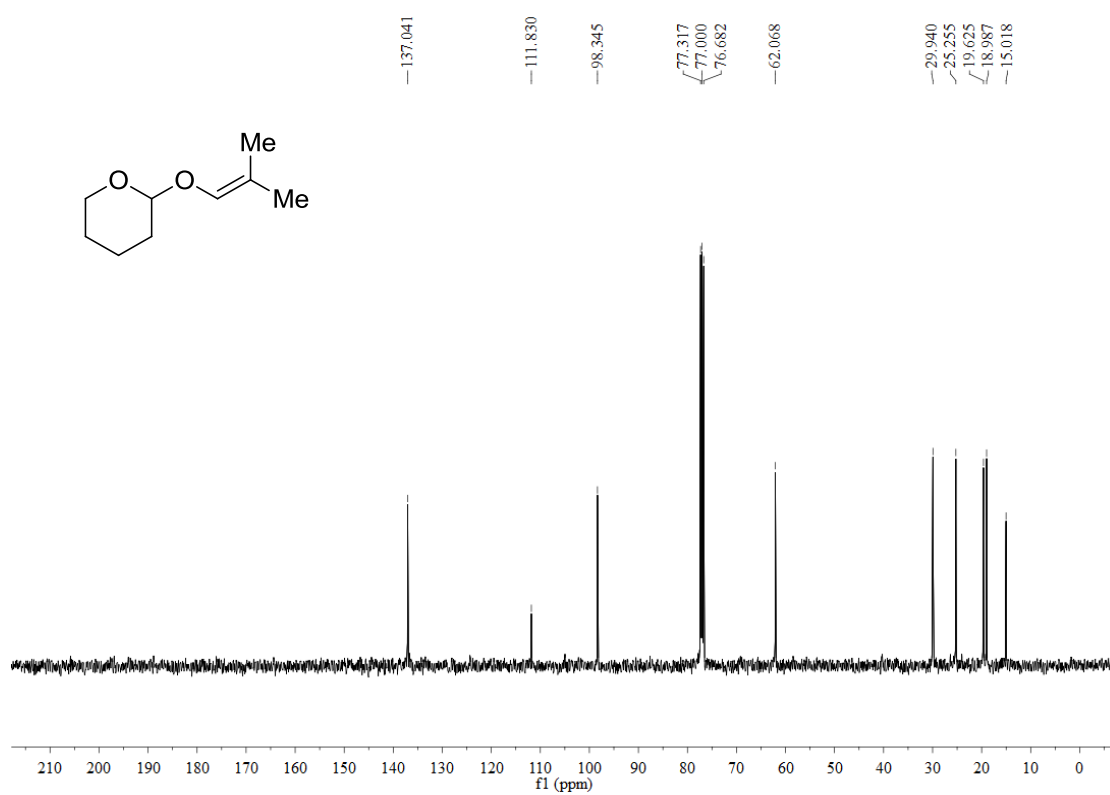


Figure S36. ¹³C NMR spectra of 2q.

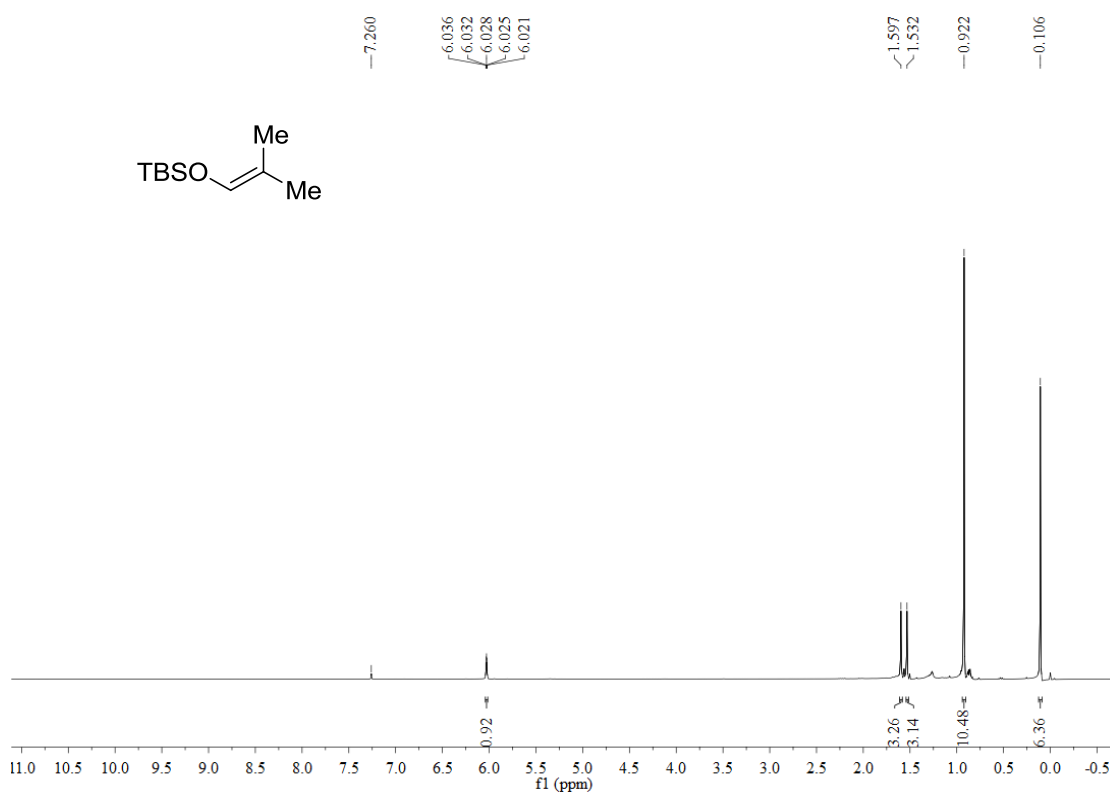


Figure S37. ^1H NMR spectra of **2r**.

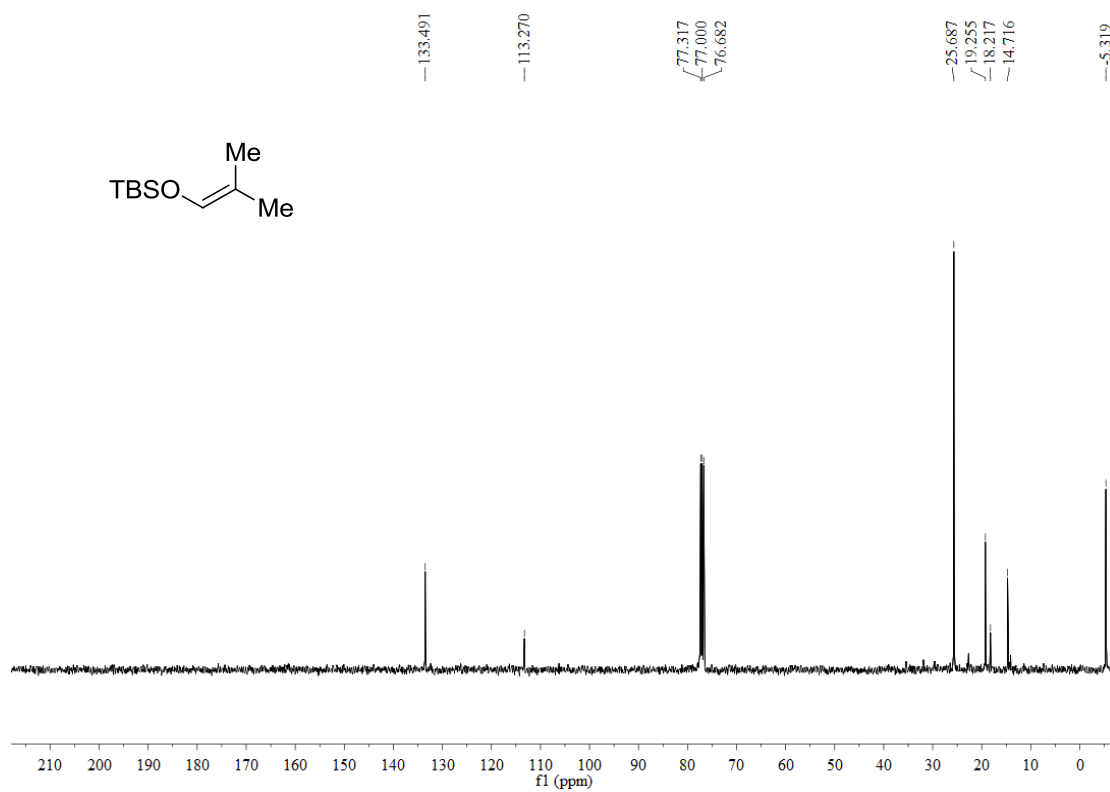


Figure S38. ^{13}C NMR spectra of **2r**.

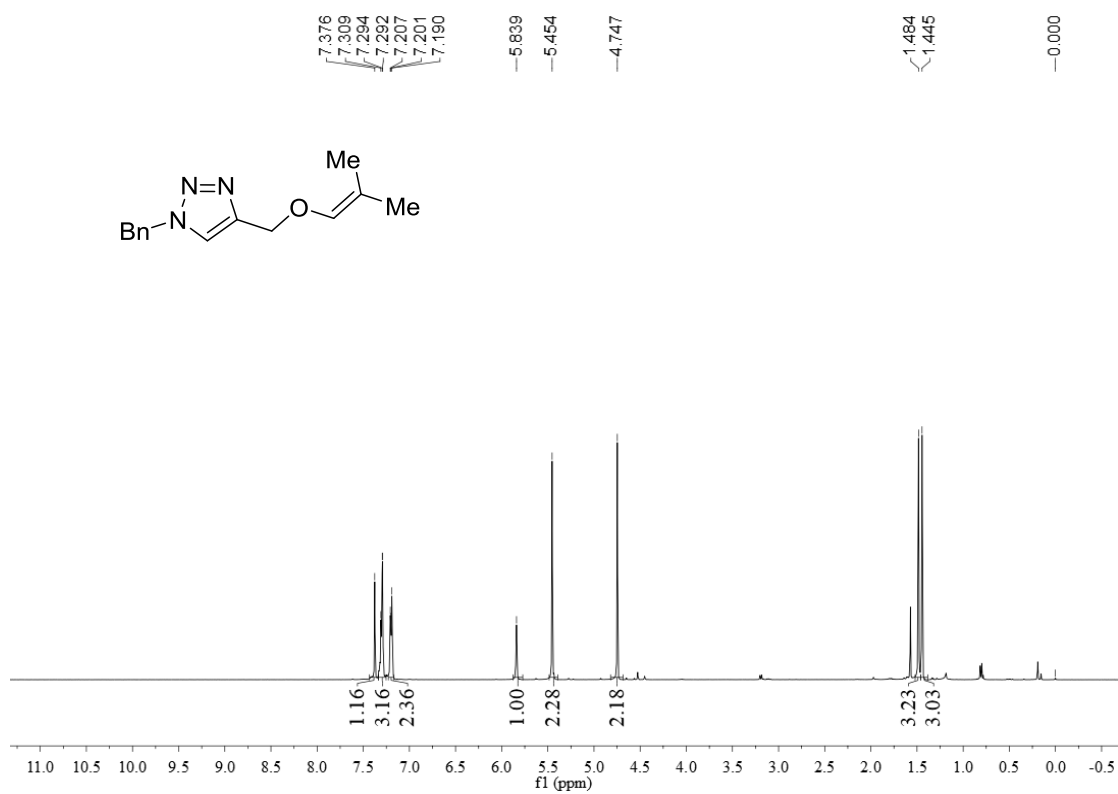


Figure S39. ^1H NMR spectra of 2s.

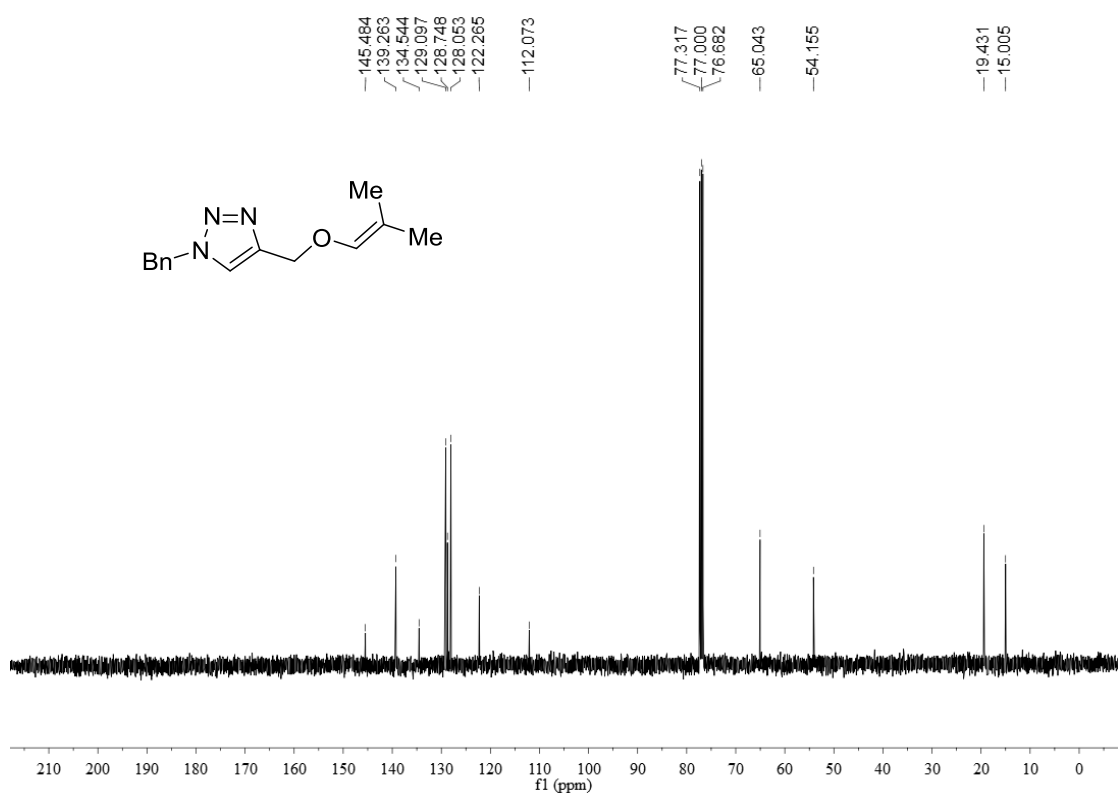


Figure S40. ^{13}C NMR spectra of 2s.

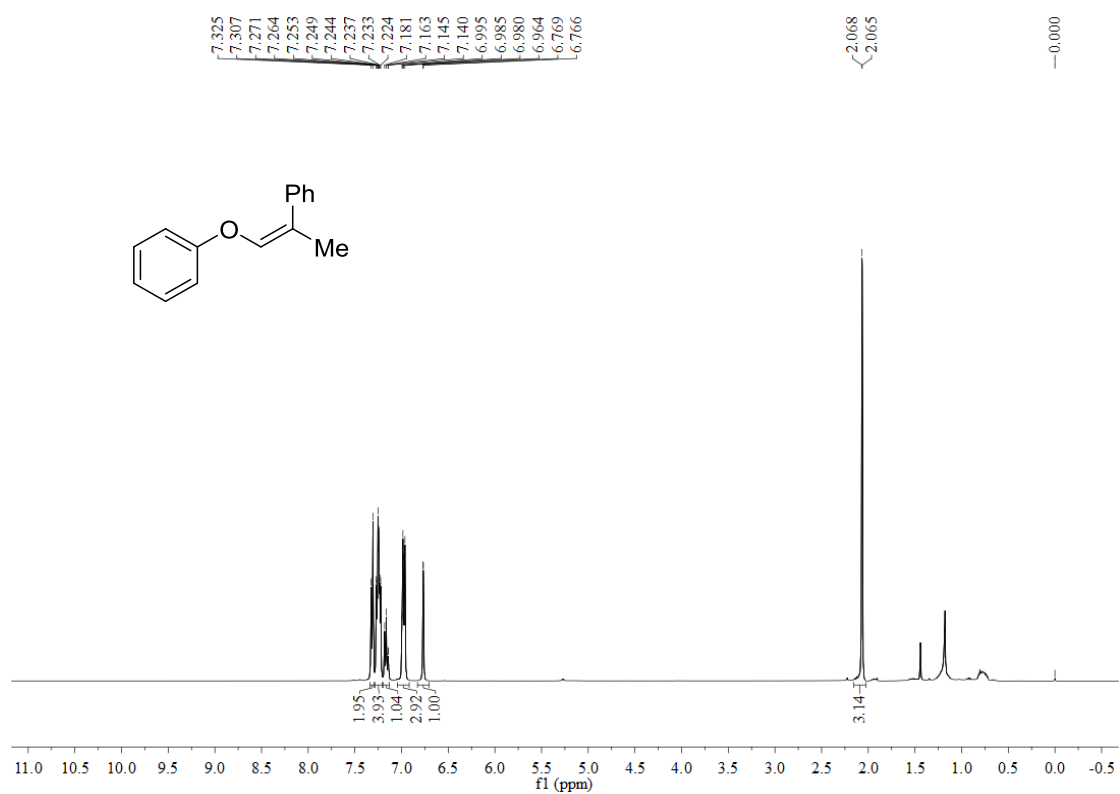


Figure S41. ¹H NMR spectra of **Z-2u**.

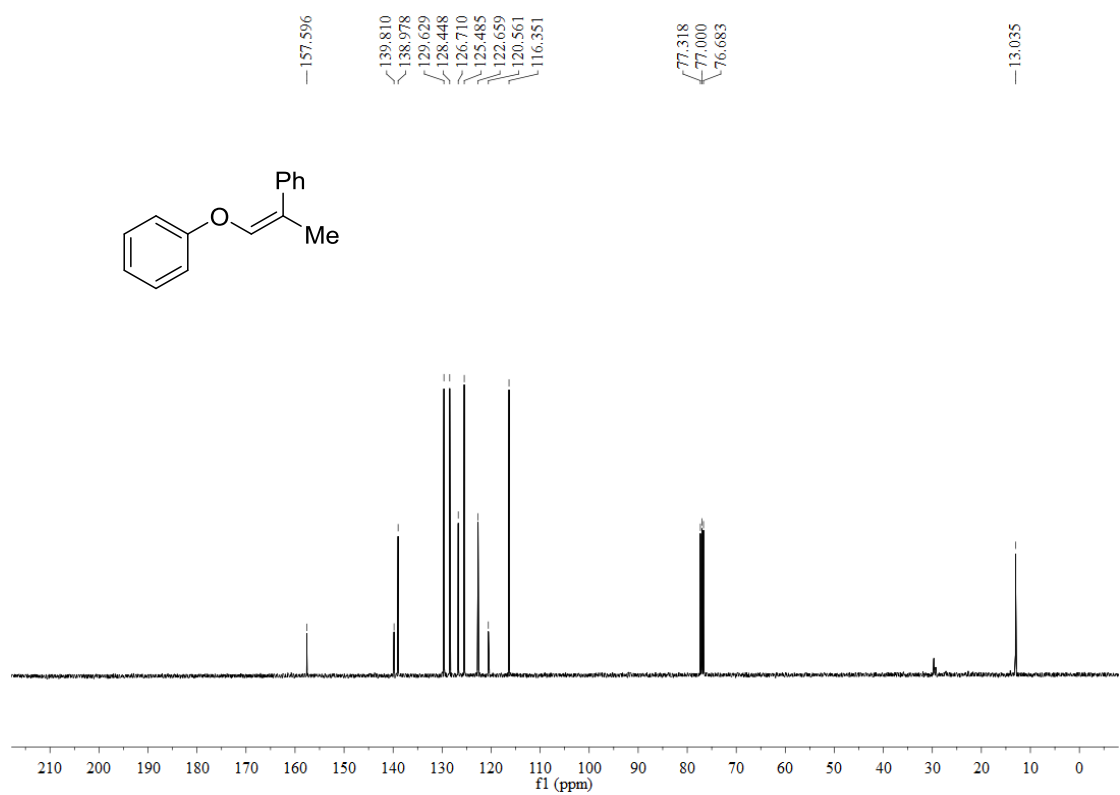


Figure S42. ¹³C NMR spectra of **Z-2u**.

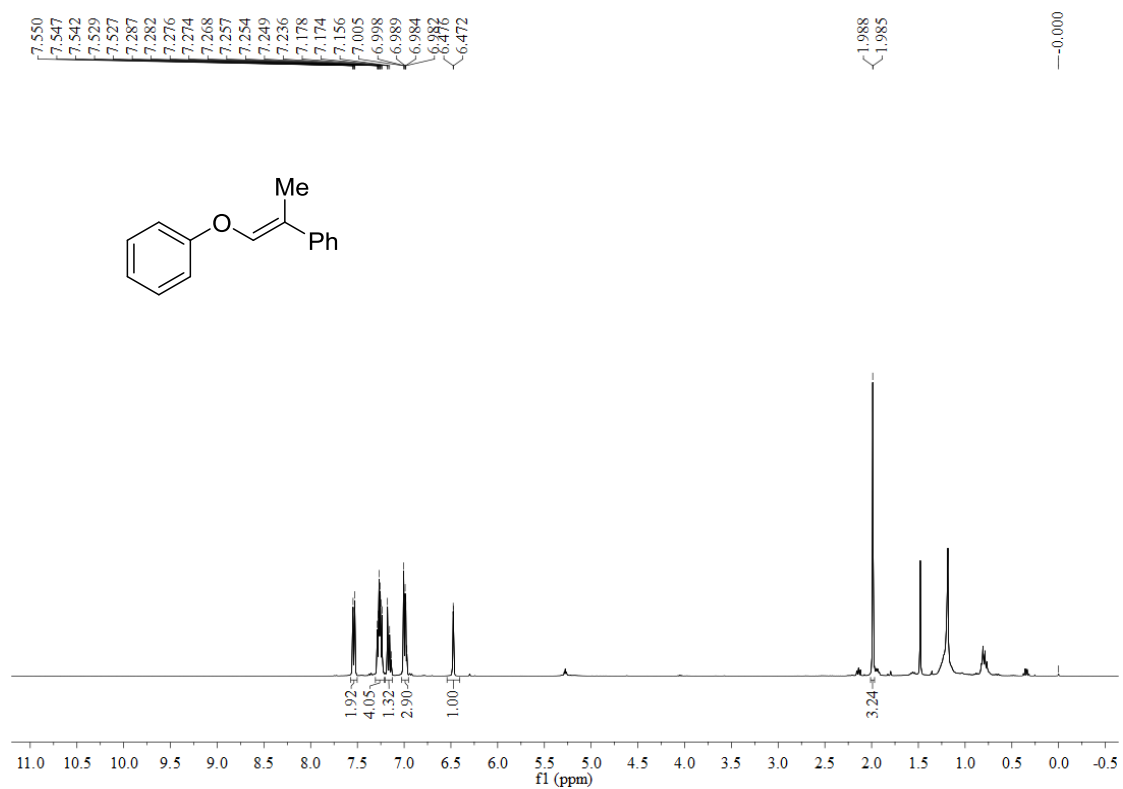


Figure S43. ¹H NMR spectra of *E*-2u.

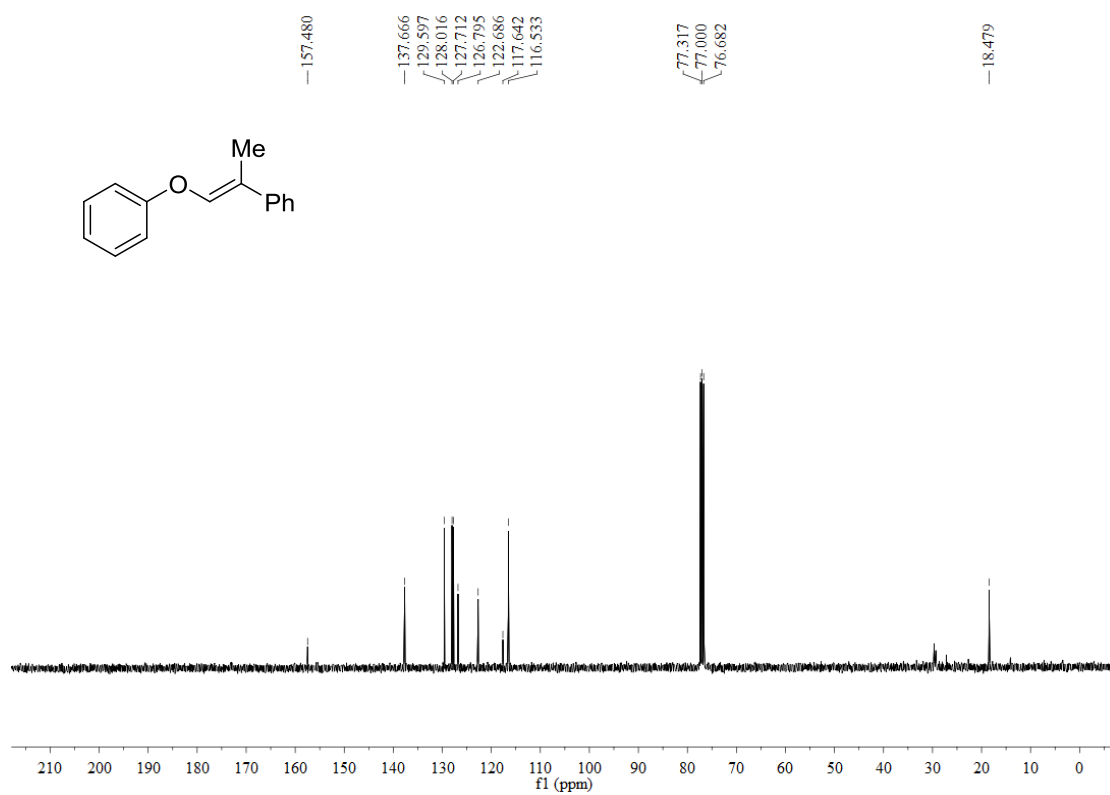


Figure S44. ¹³C NMR spectra of *E*-2u.

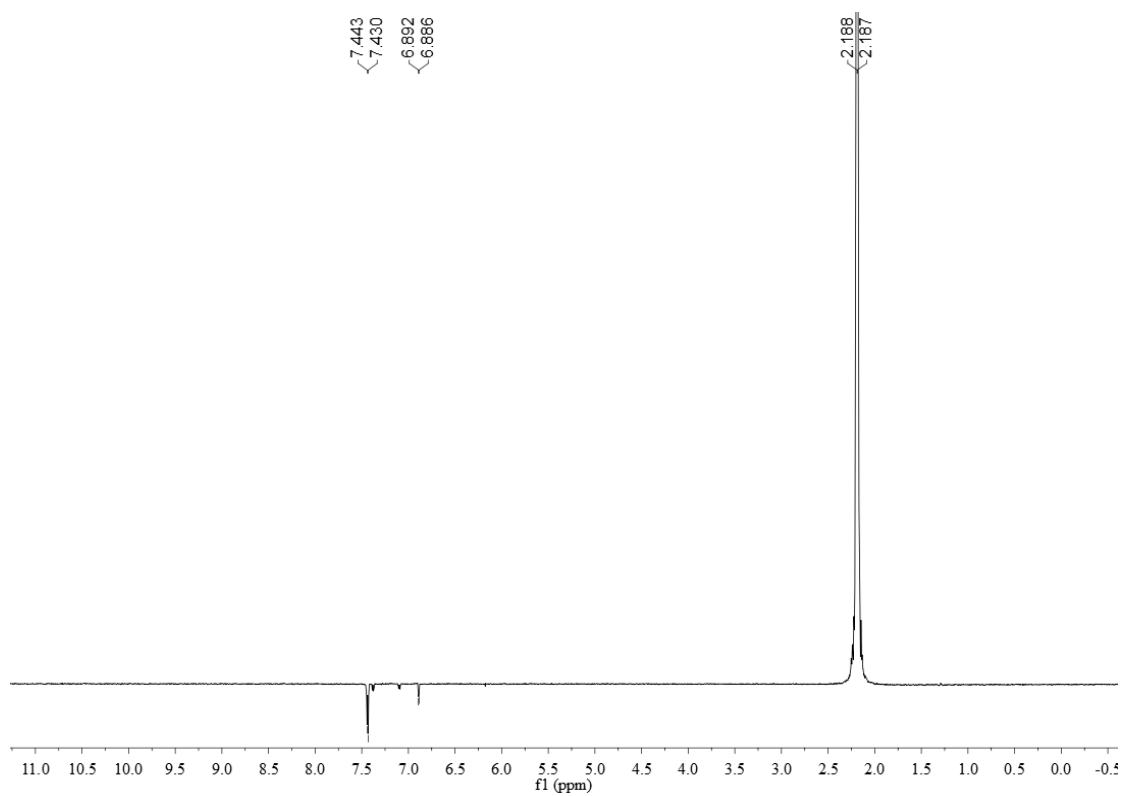


Figure S45. 1D NOESY of Z-2u.

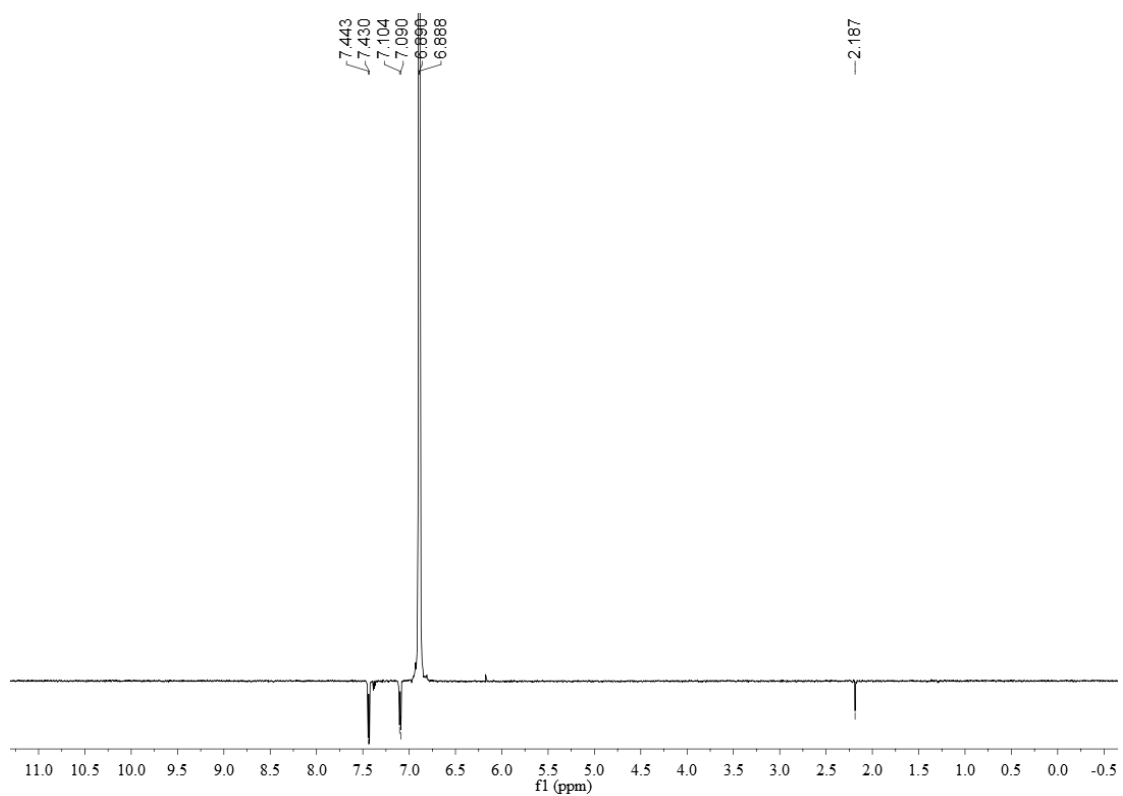


Figure S46. 1D NOESY of Z-2u.

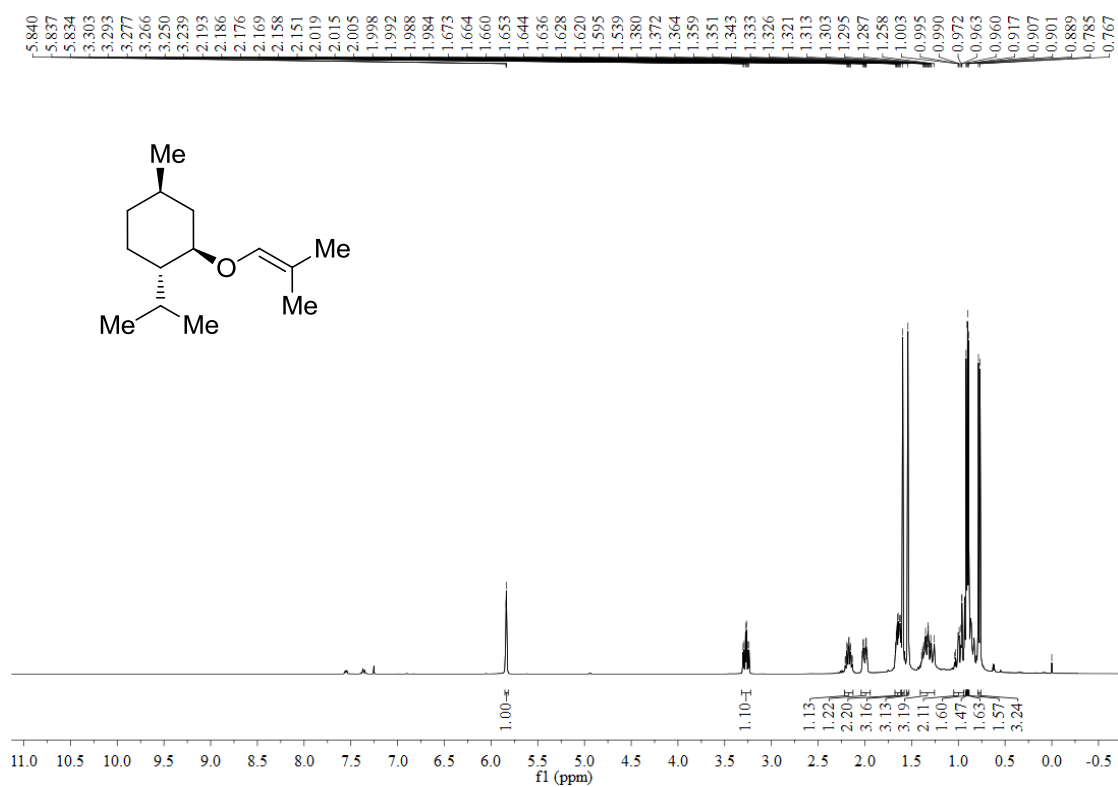


Figure S47. ¹H NMR spectra of **2v**.

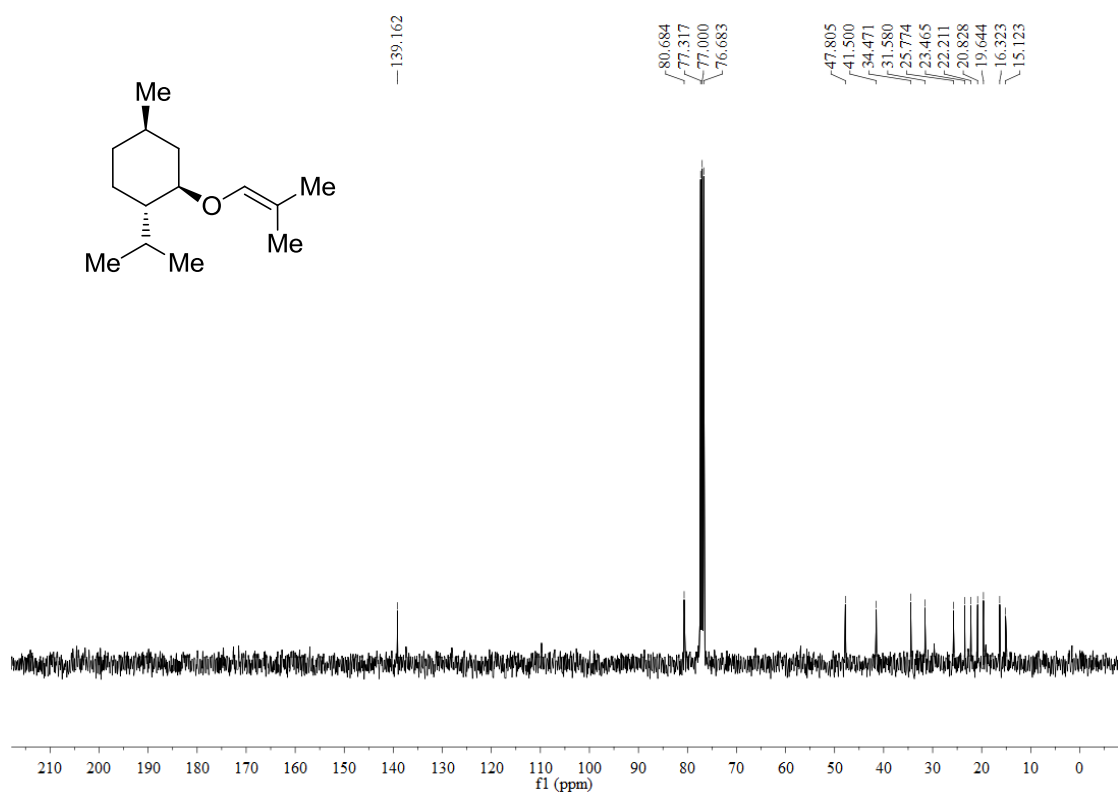


Figure S48. ¹³C NMR spectra of **2v**.

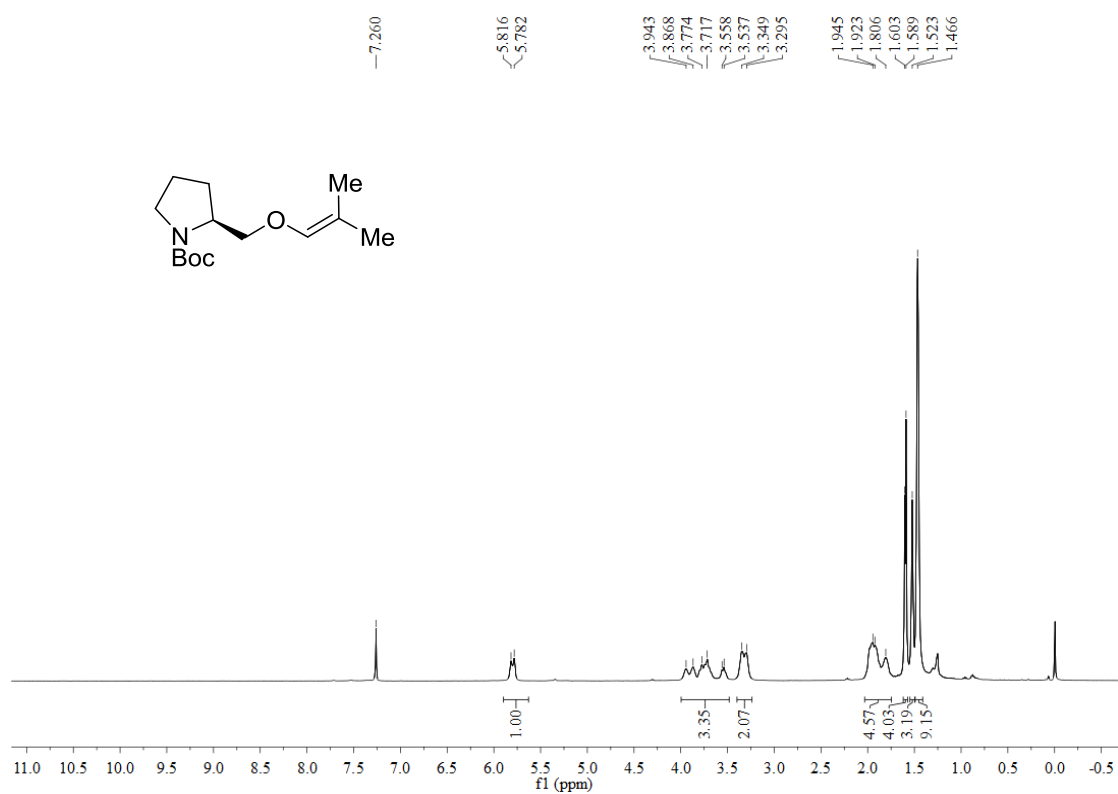


Figure S49. ¹H NMR spectra of **2w**.

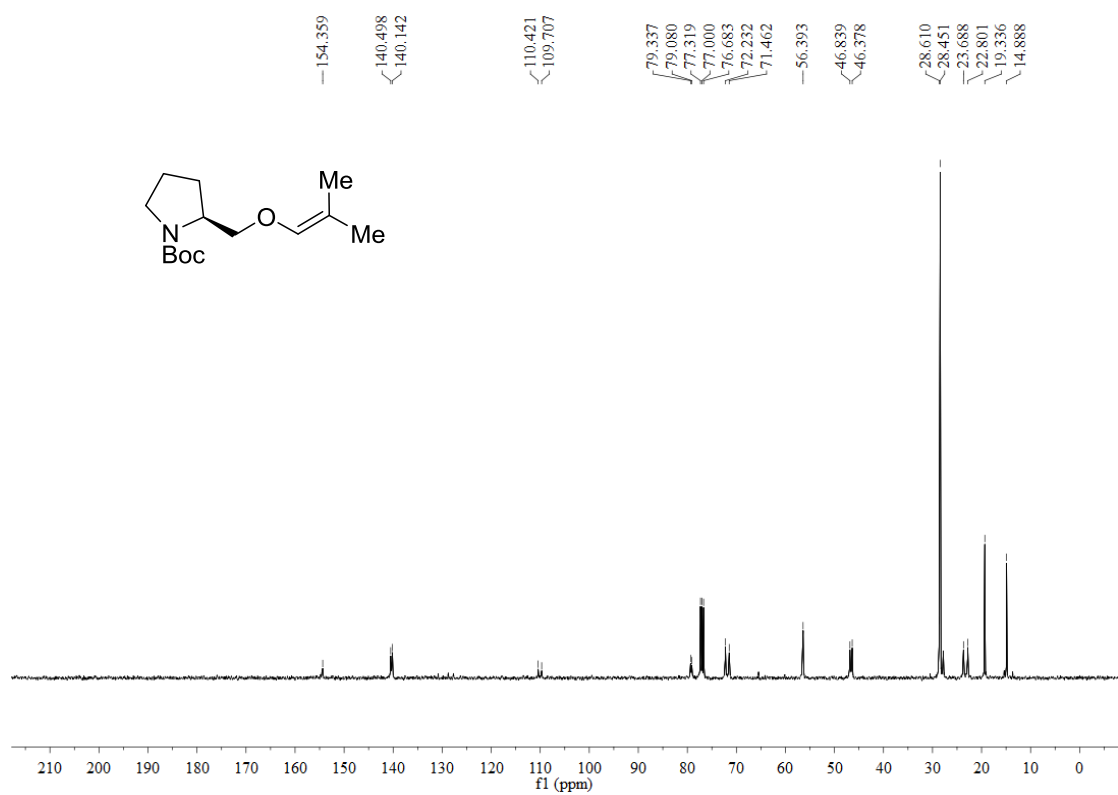


Figure S50. ¹³C NMR spectra of **2w**.

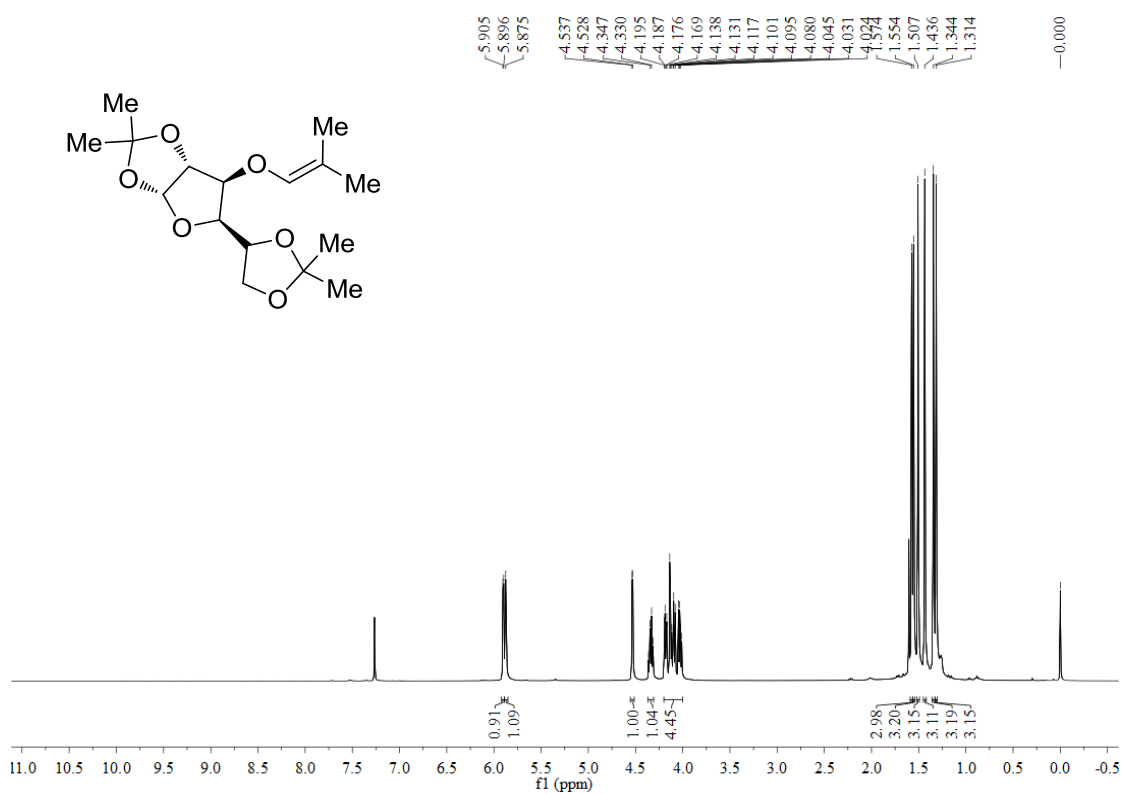


Figure S51. ^1H NMR spectra of **2x**.

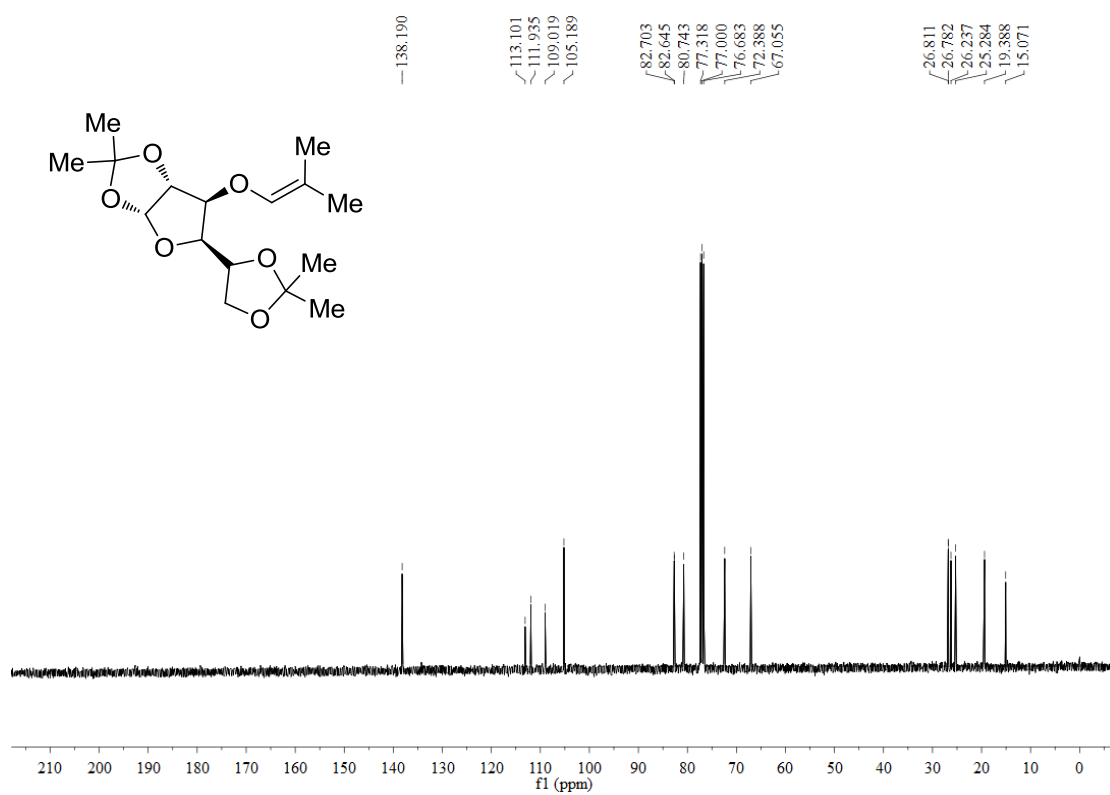


Figure S52. ^{13}C NMR spectra of **2x**.

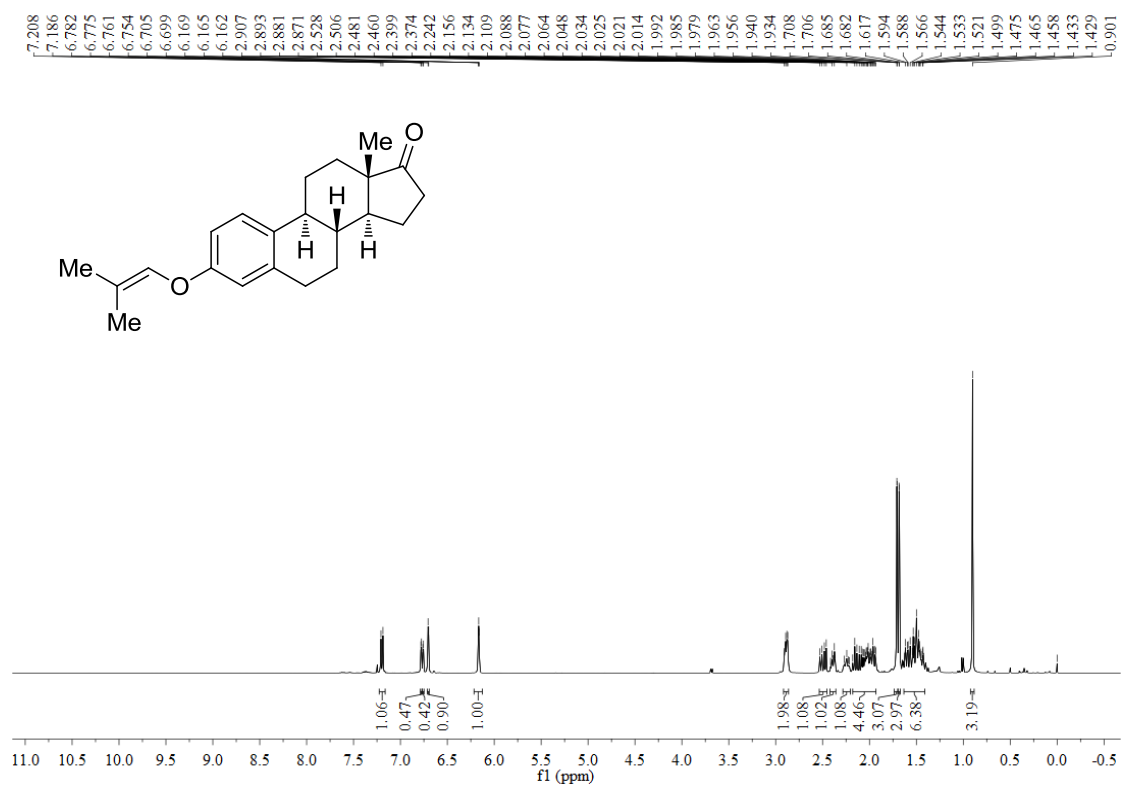


Figure S53. ¹H NMR spectra of **2y**.

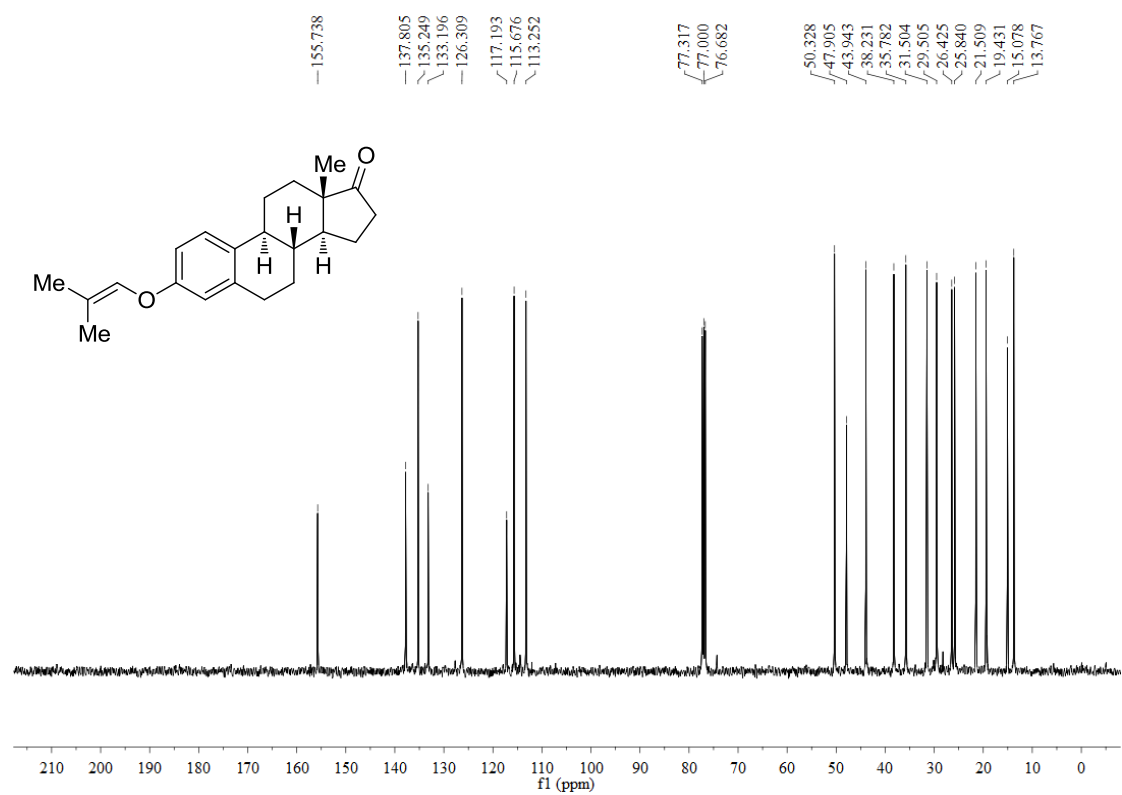


Figure S54. ¹³C NMR spectra of **2y**.

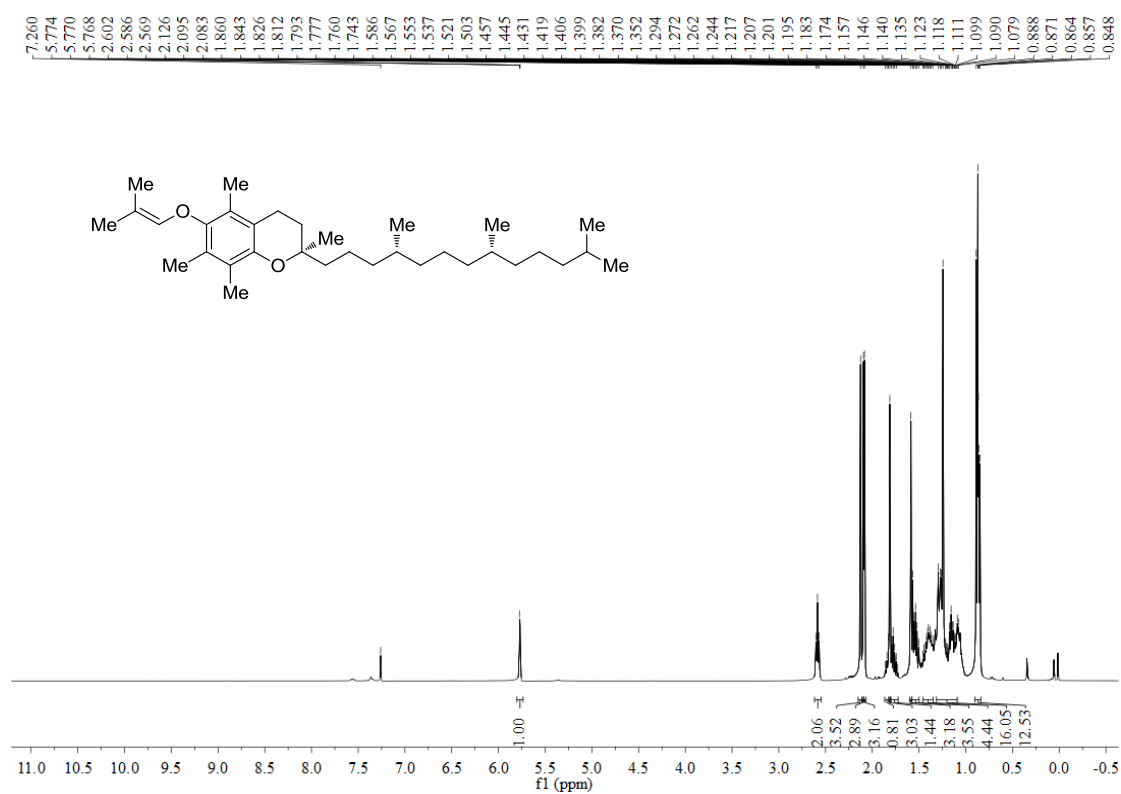


Figure S55. ^1H NMR spectra of **2z**.

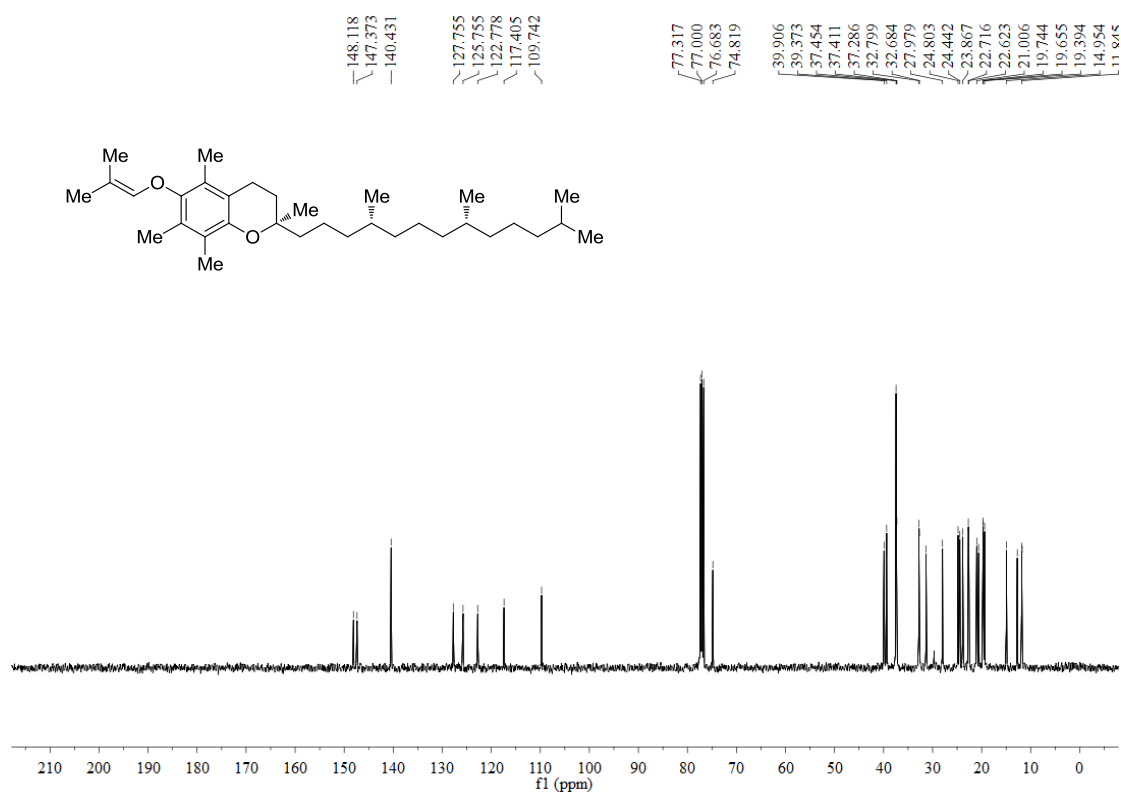


Figure S56. ^{13}C NMR spectra of **2z**.

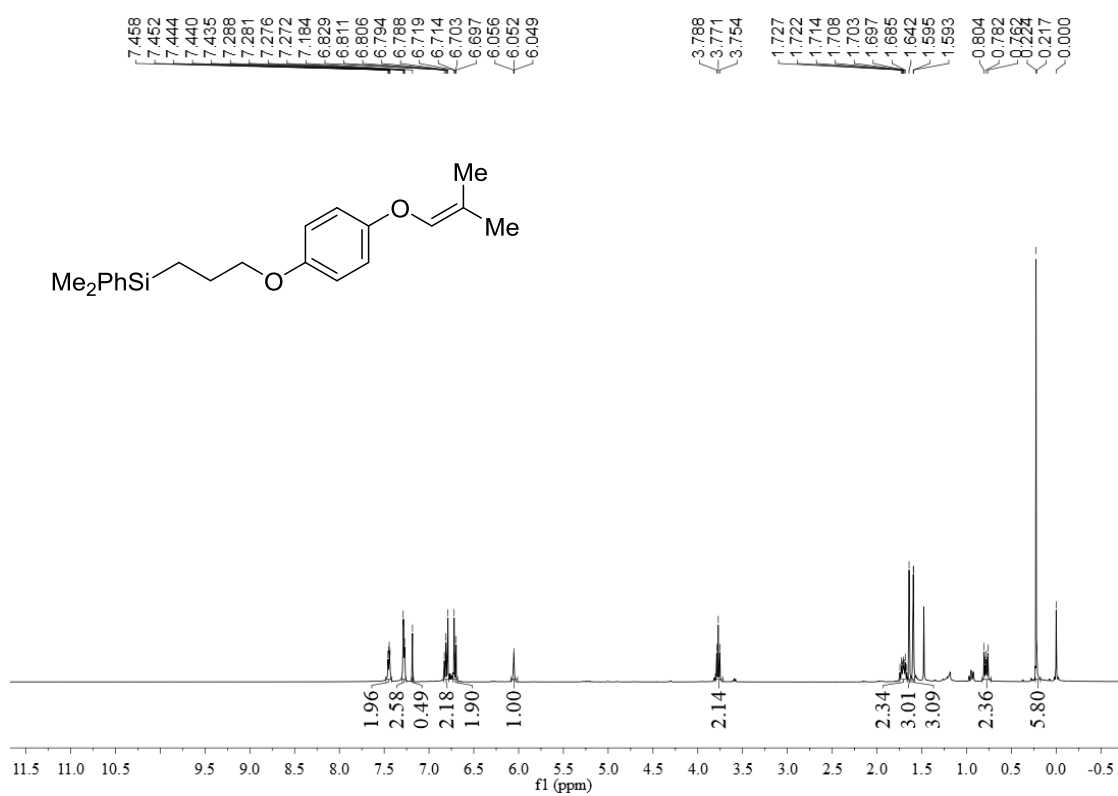


Figure S57. ¹H NMR spectra of **2aa**.

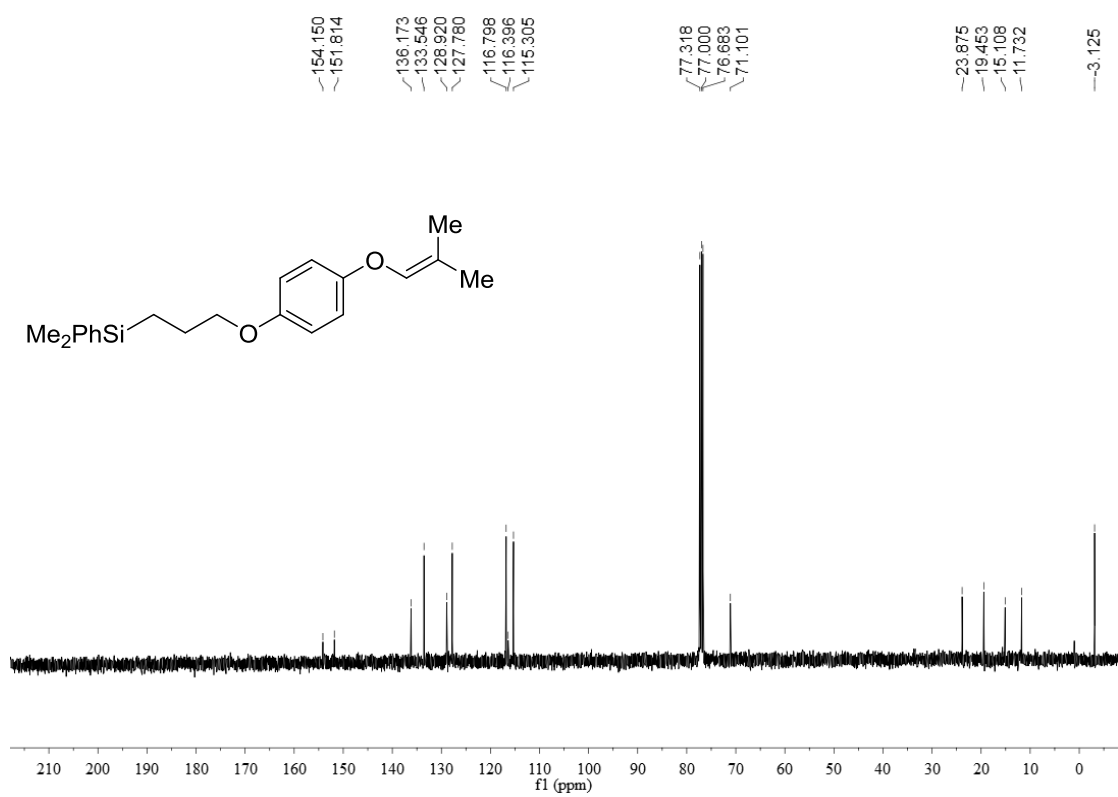


Figure S58. ¹³C NMR spectra of **2aa**.

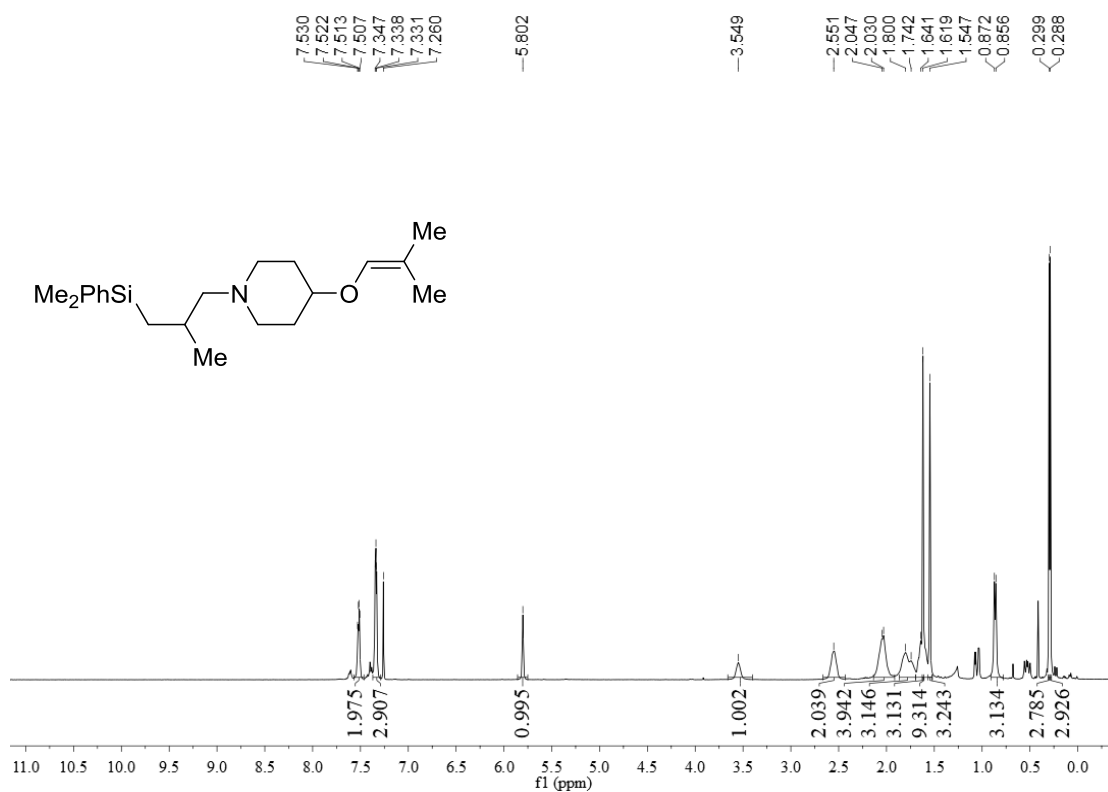


Figure S59. ¹H NMR spectra of **2ab**.

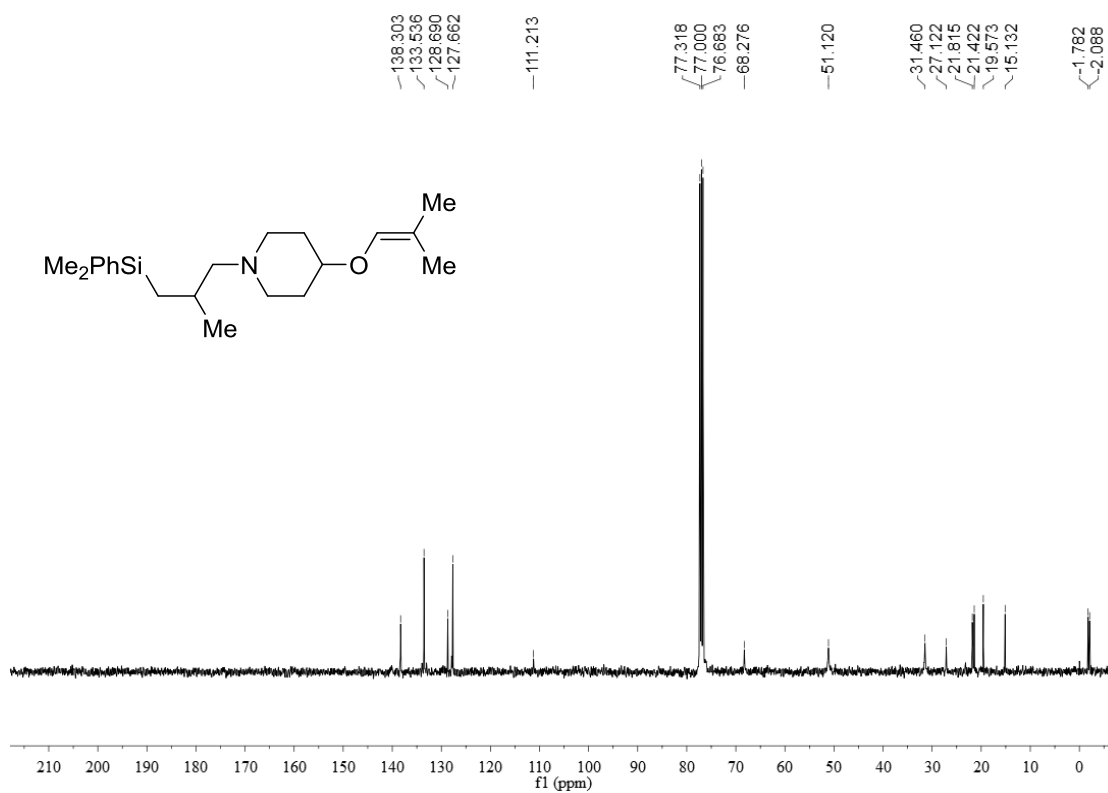


Figure S60. ¹³C NMR spectra of **2ab**.

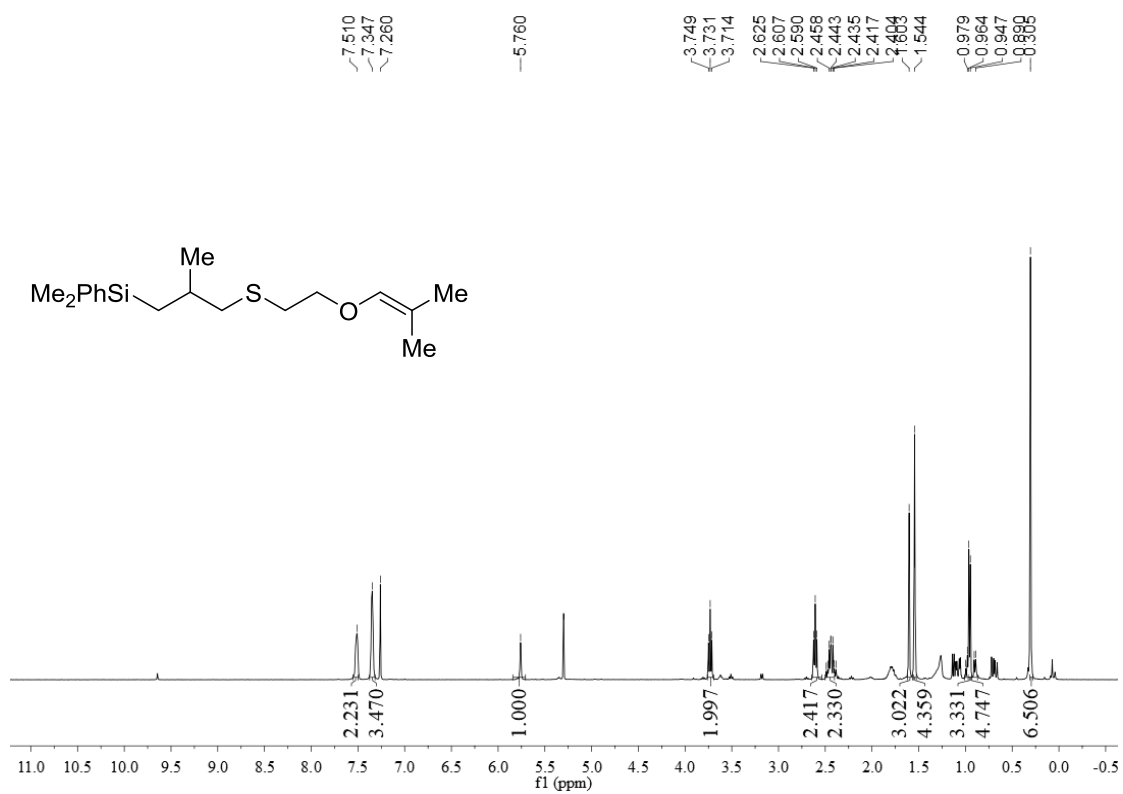


Figure S61. ¹H NMR spectra of **2ac**.

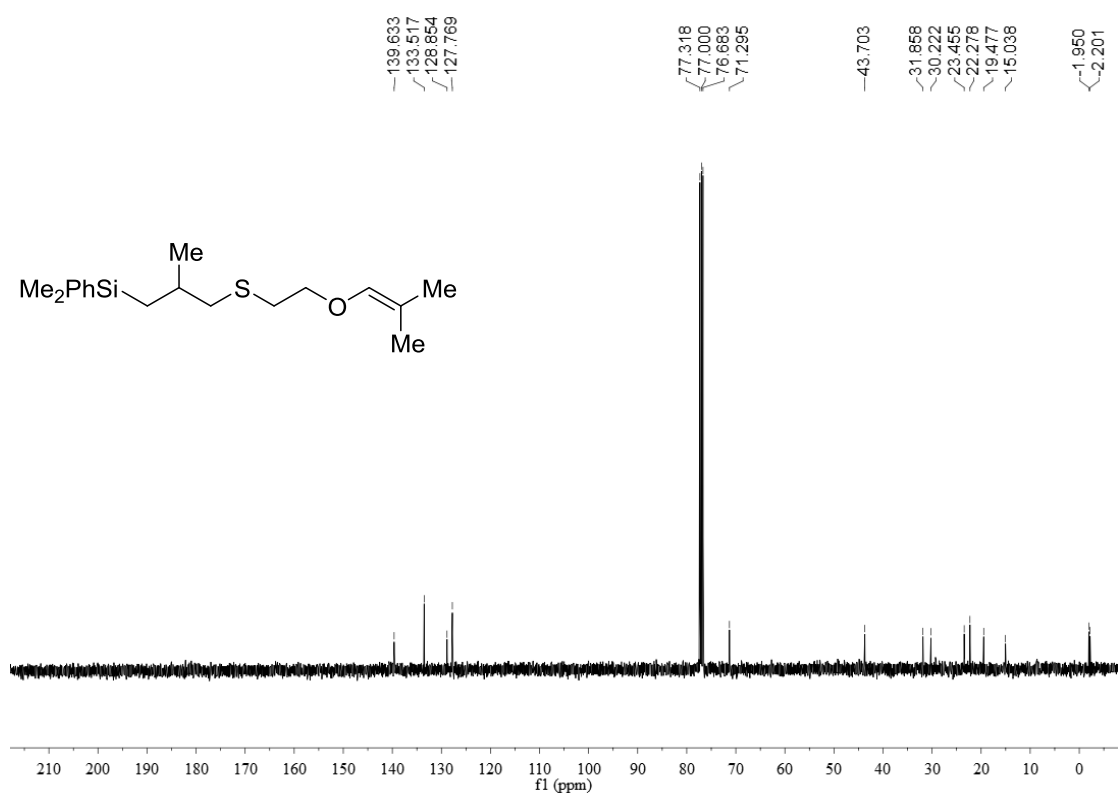


Figure S62. ¹³C NMR spectra of **2ac**.

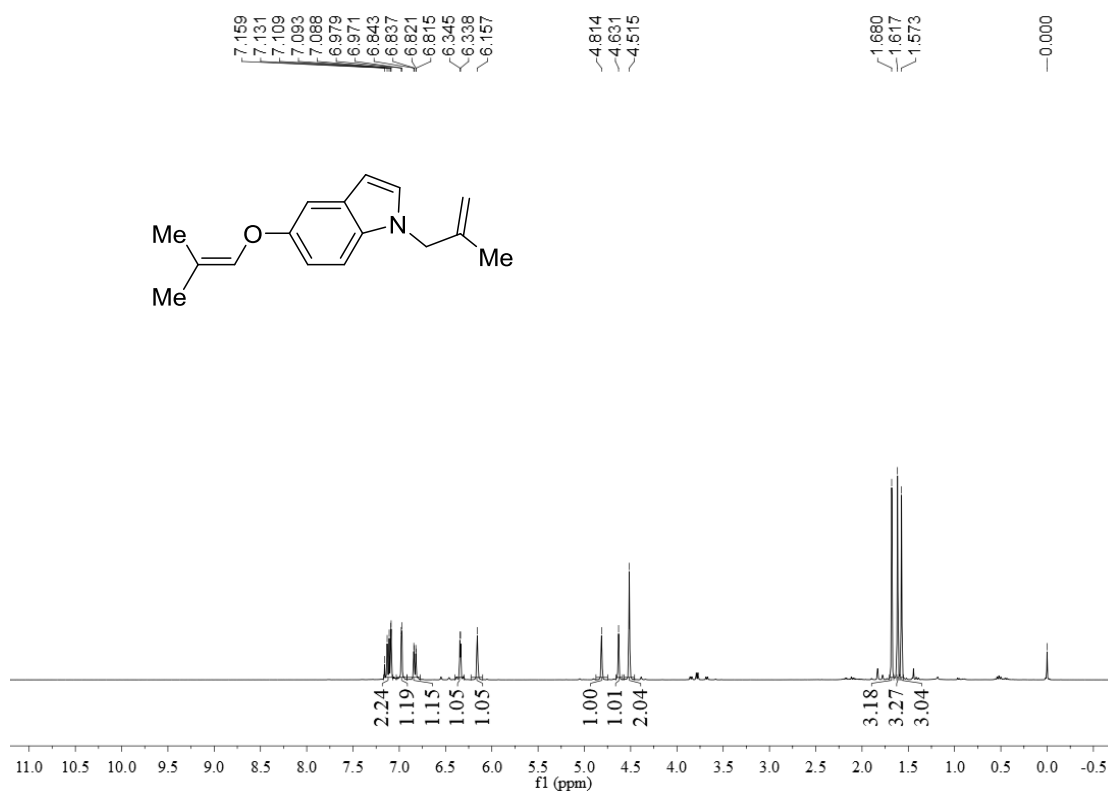


Figure S63. ¹H NMR spectra of 2ad.

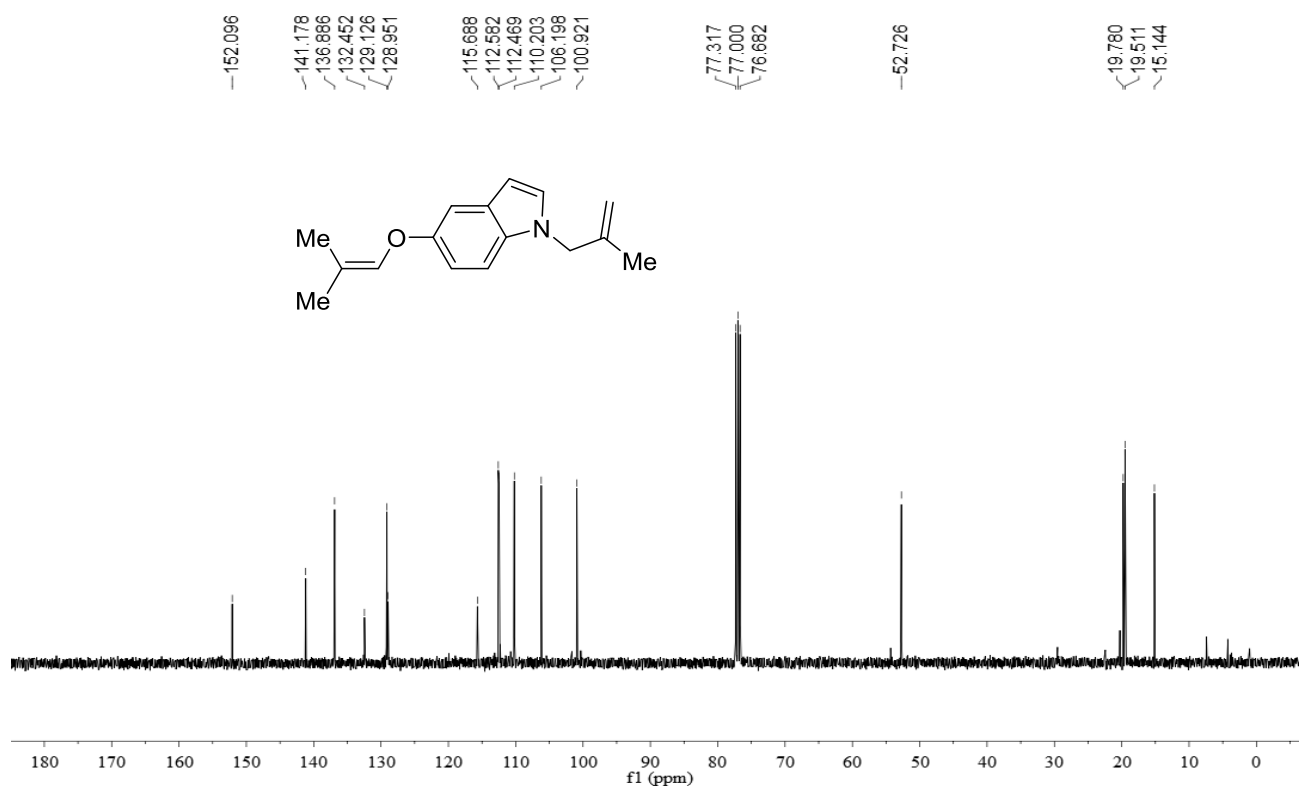


Figure S64. ¹³C NMR spectra of 2ad.

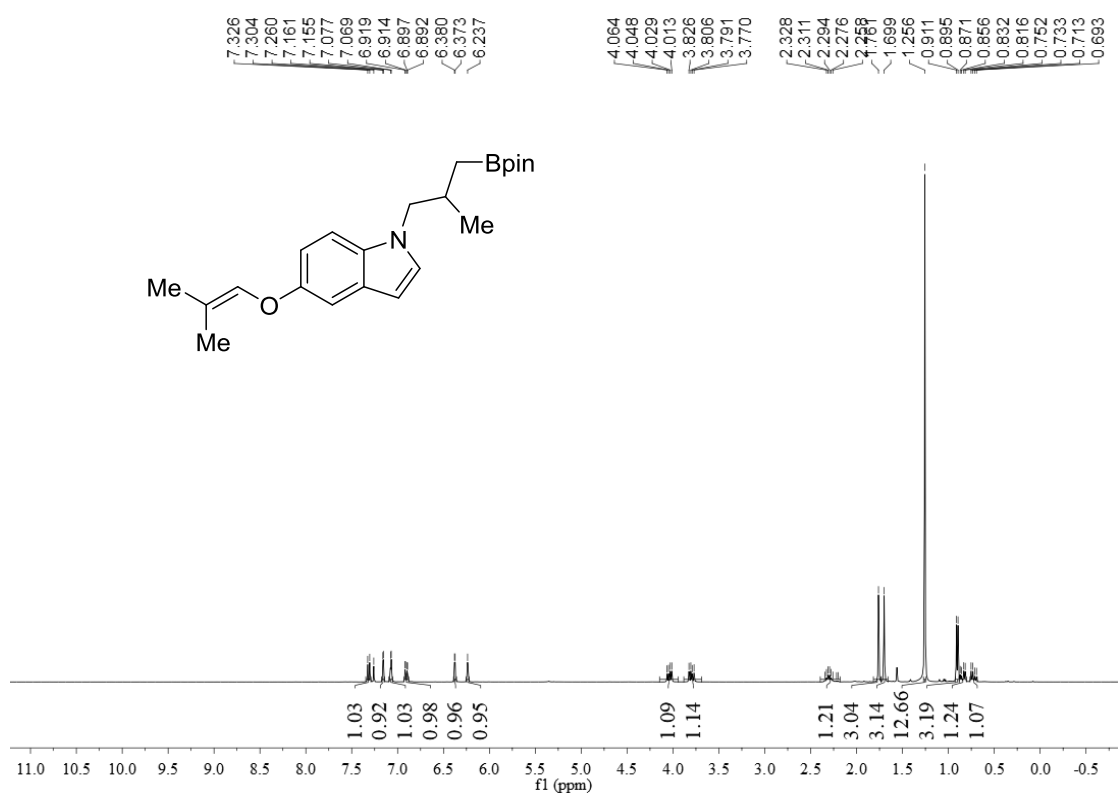


Figure S65. ¹H NMR spectra of **3ad**.

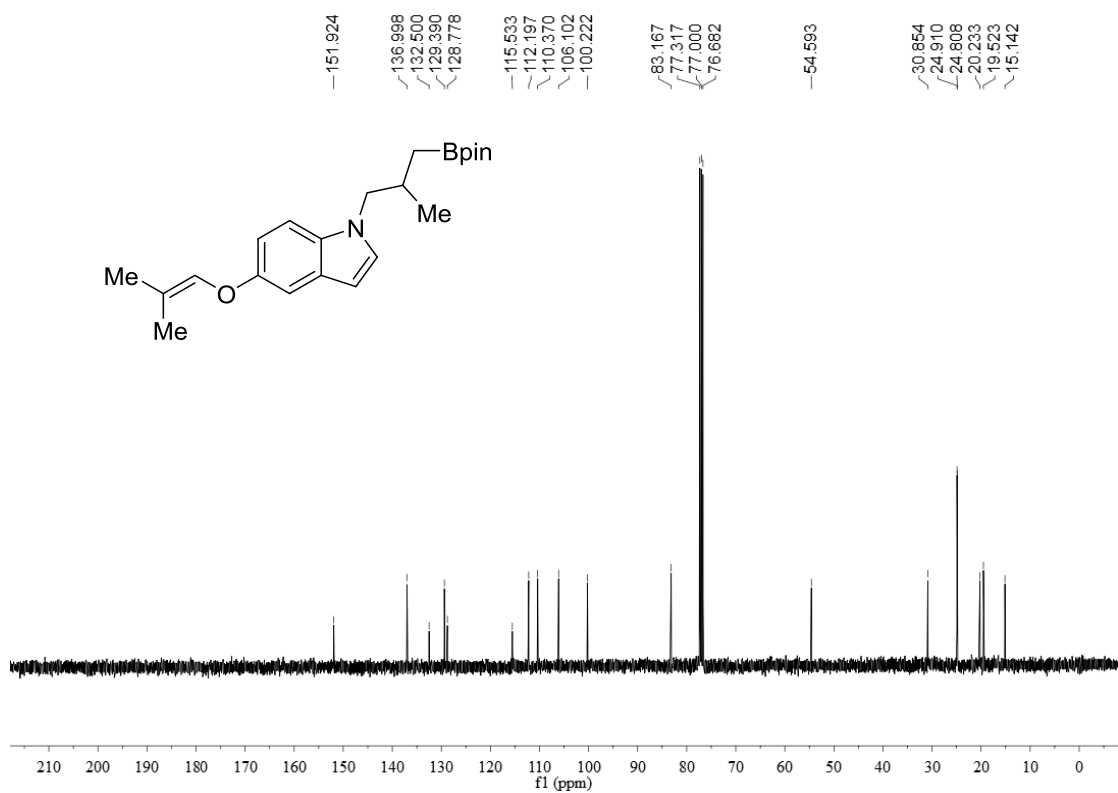


Figure S66. ¹³C NMR spectra of **3ad**.

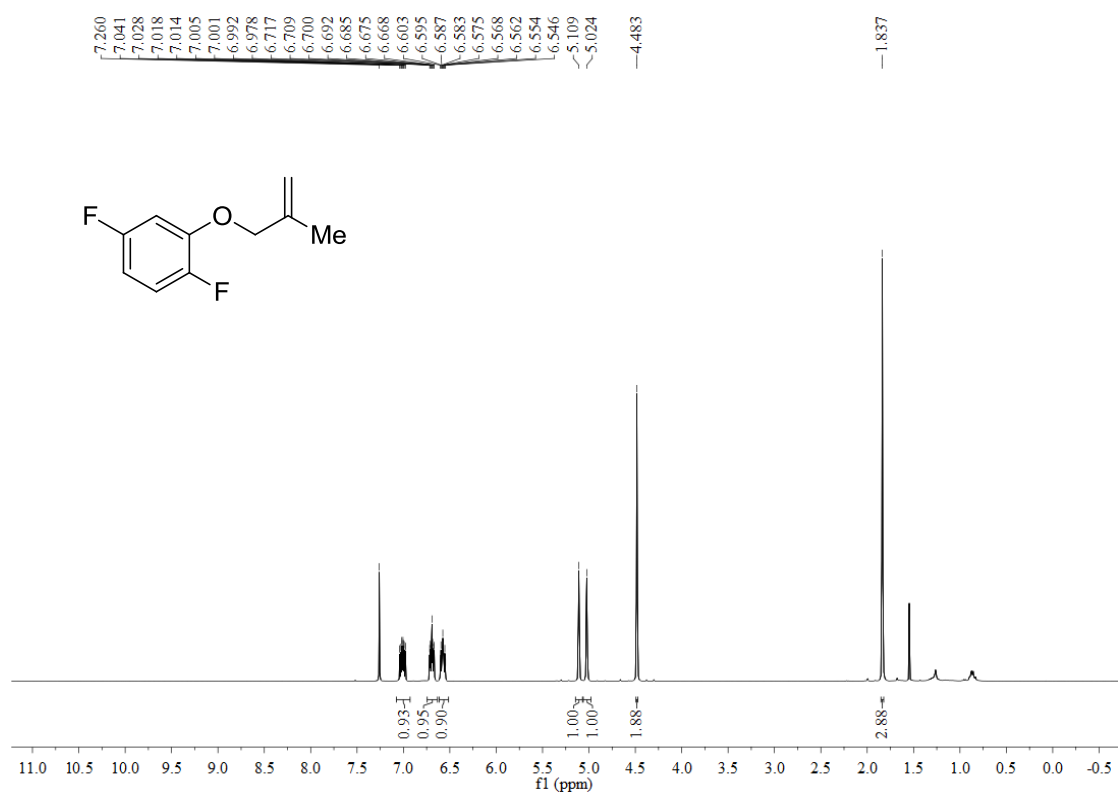


Figure S67. ¹H NMR spectra of **1i**.

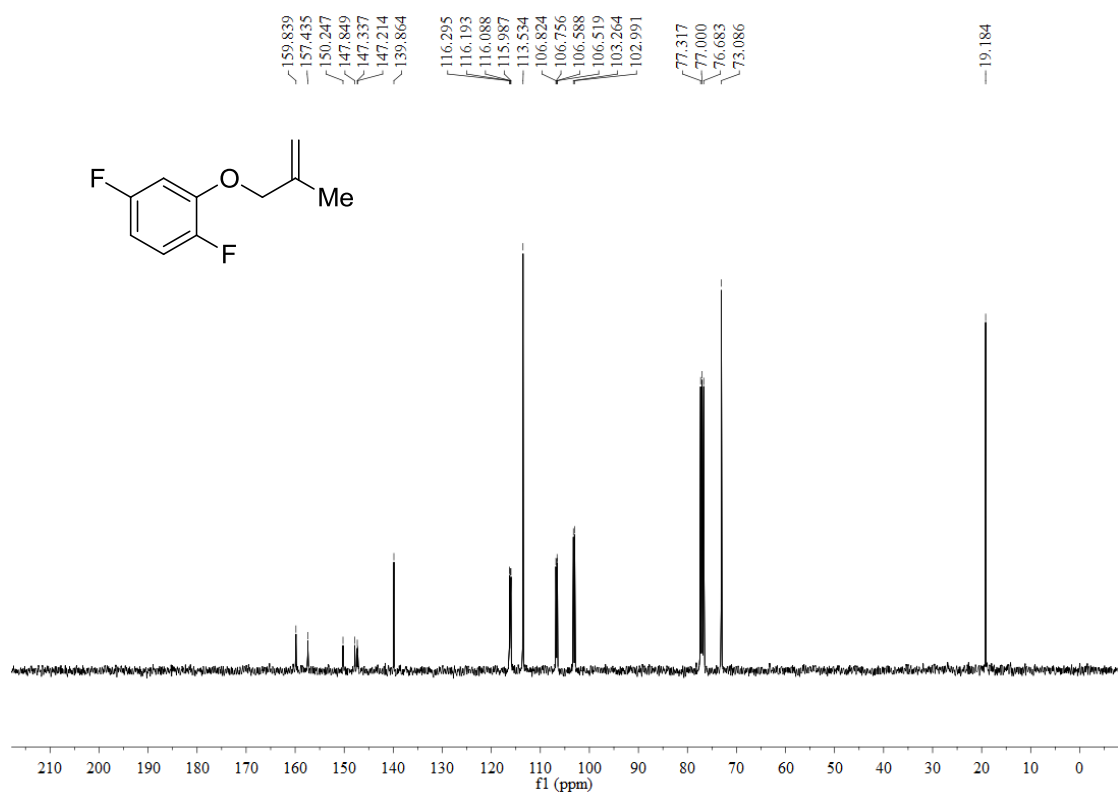


Figure S68. ¹³C NMR spectra of **1i**.

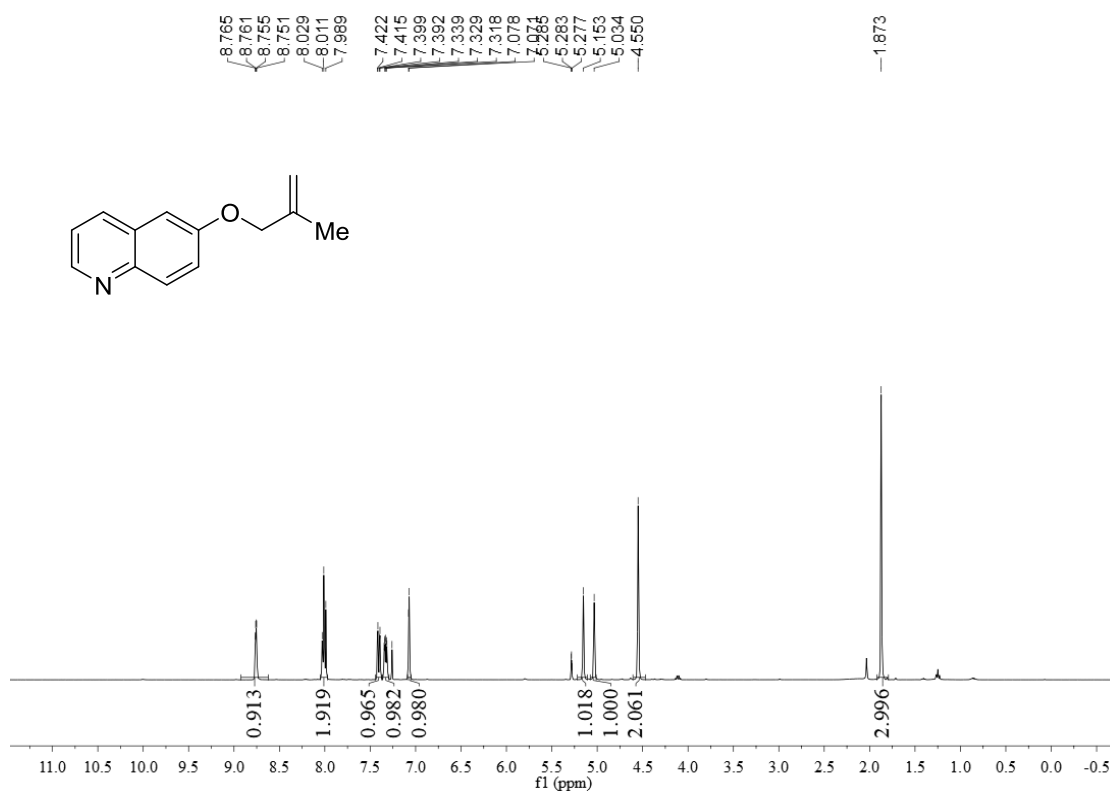


Figure S71. ¹H NMR spectra of **1m**.

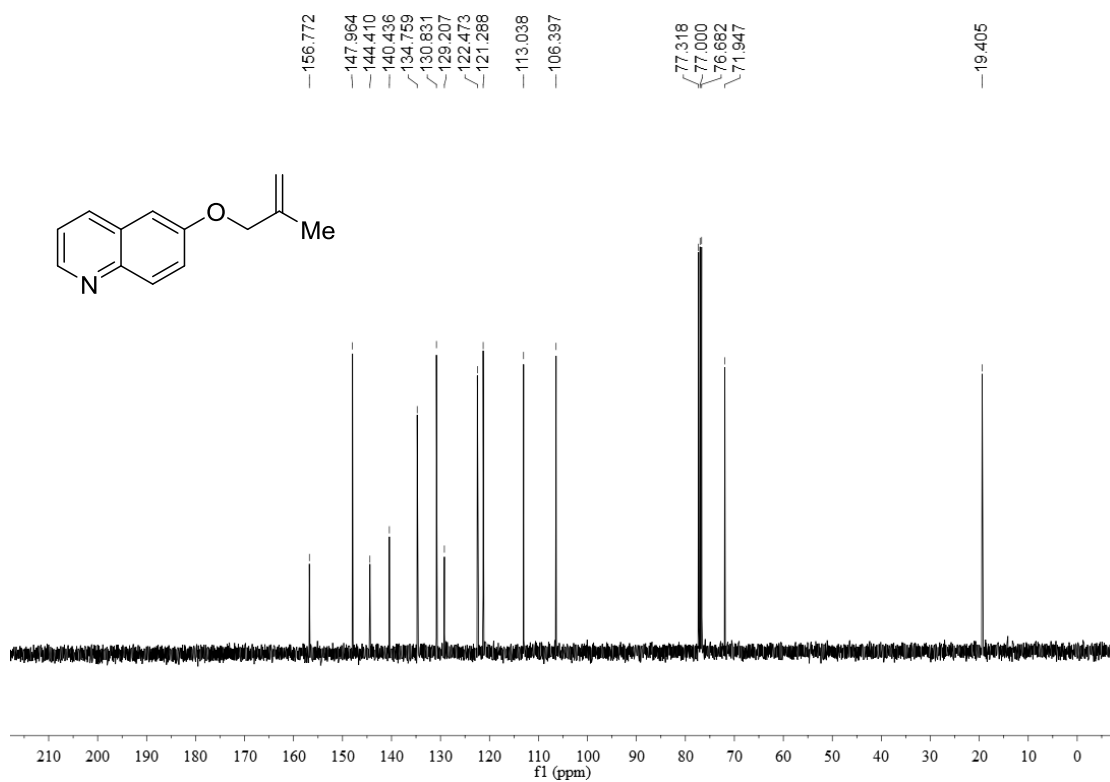


Figure S72. ¹³C NMR spectra of **1m**.

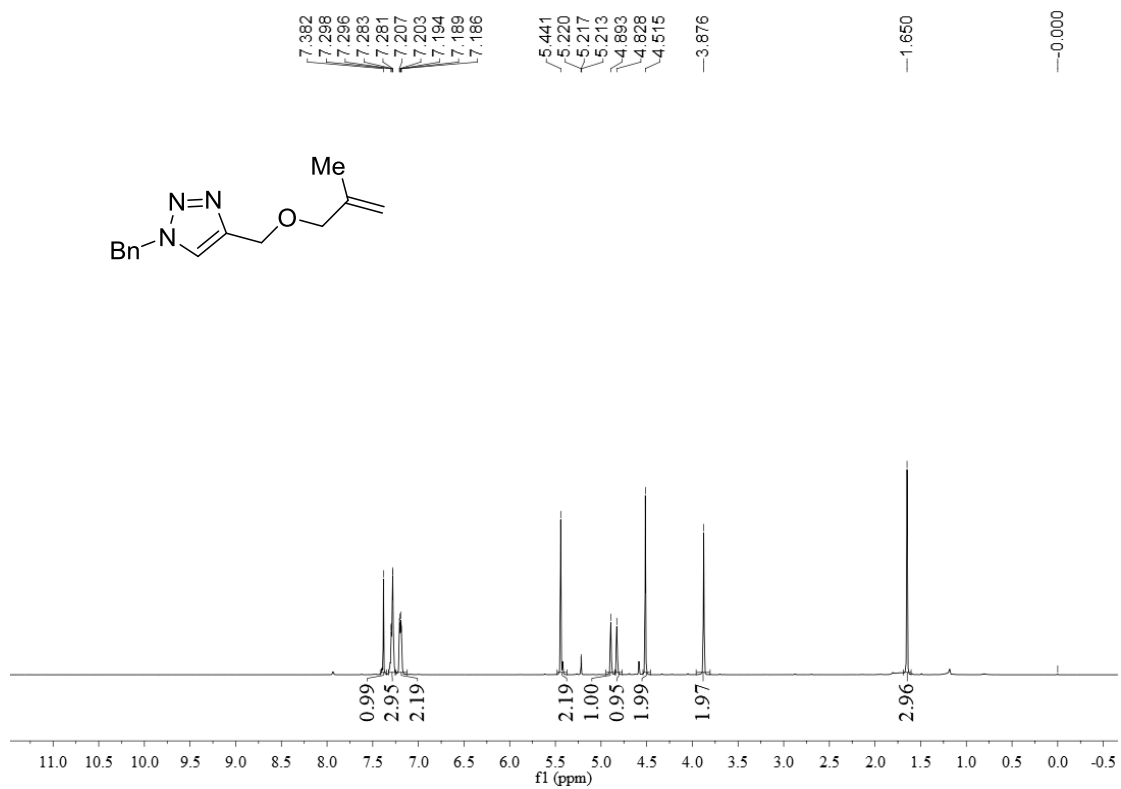


Figure S73. ¹H NMR spectra of **1s**.

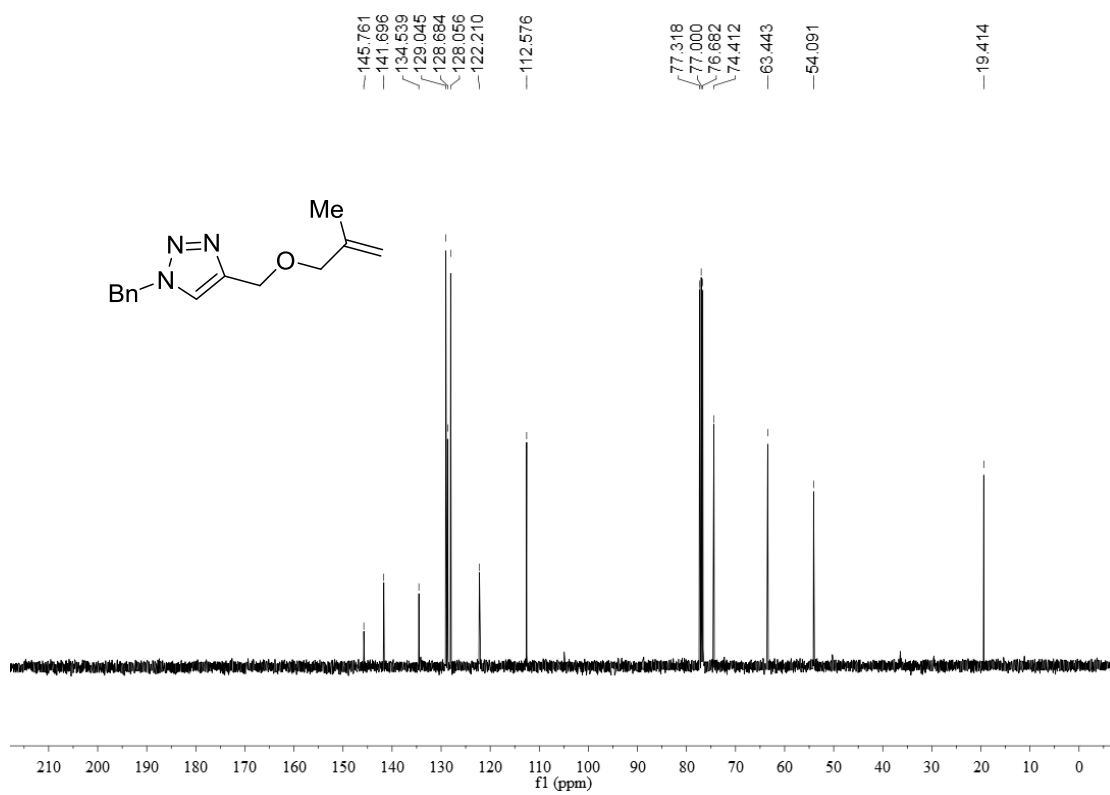


Figure S74. ¹³C NMR spectra of **1s**.

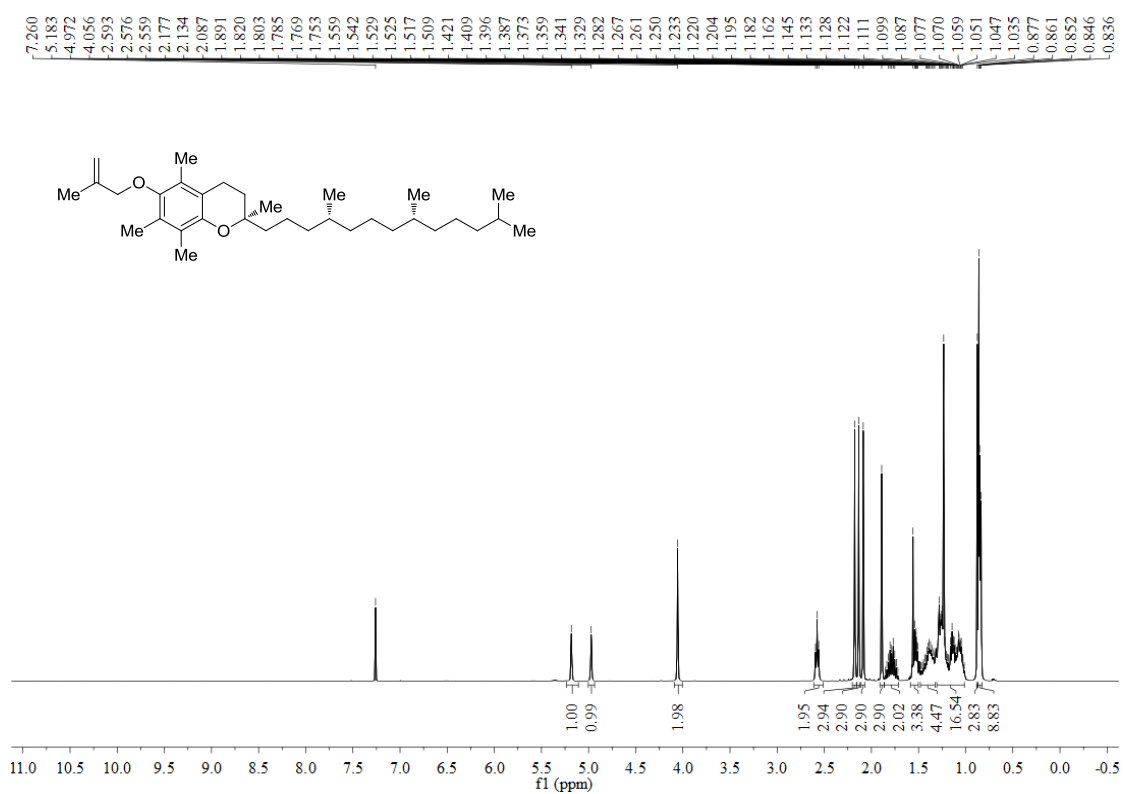


Figure S75. ¹H NMR spectra of **1z**.

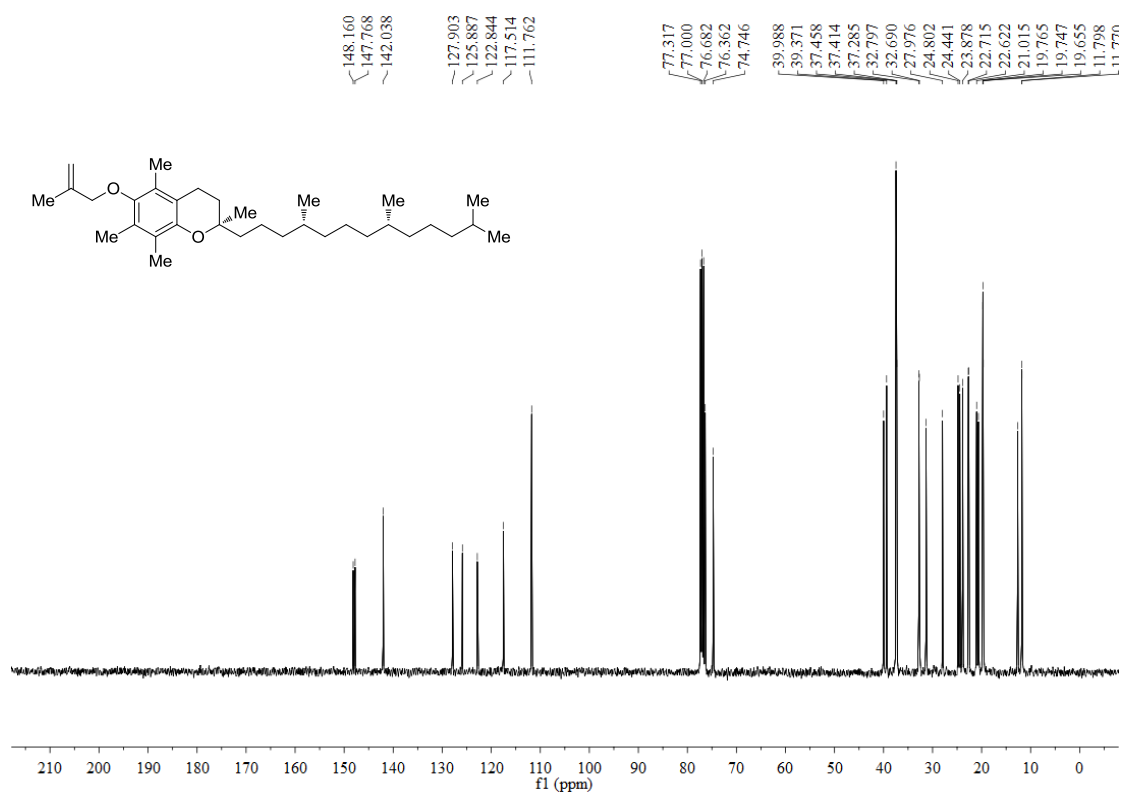


Figure S76. ¹³C NMR spectra of **1z**.

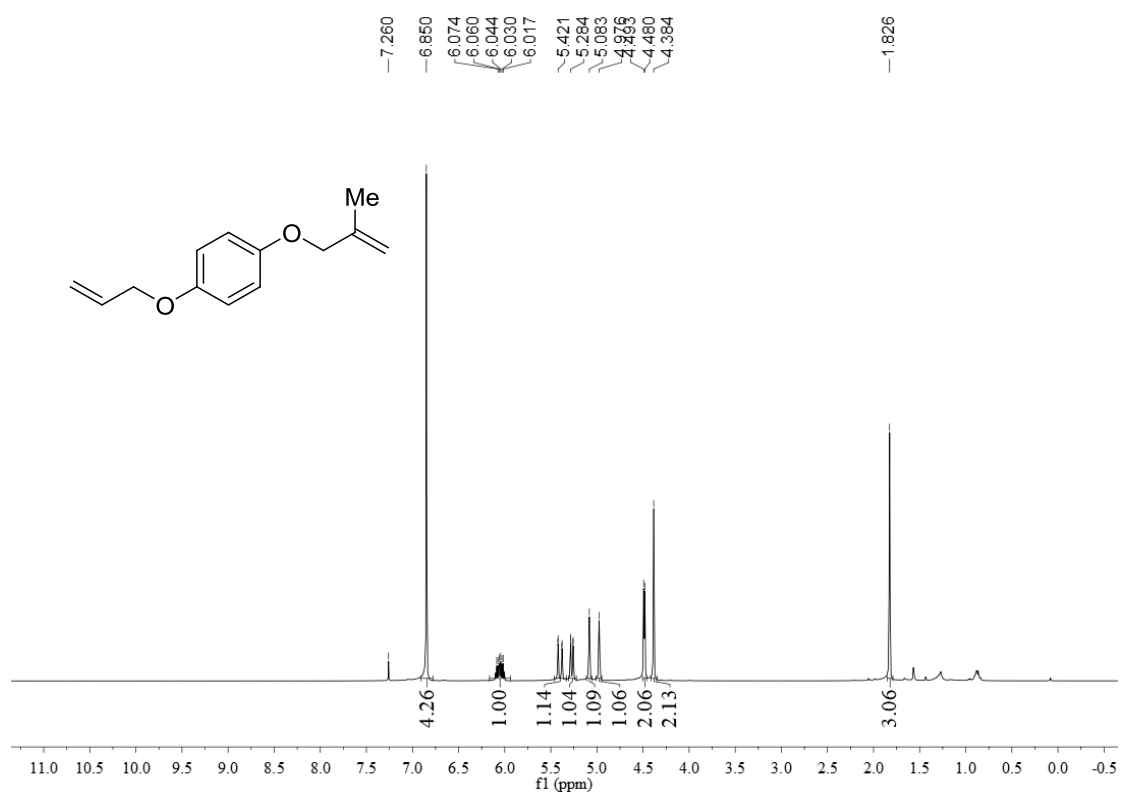


Figure S77. ¹H NMR spectra of **1aa**.

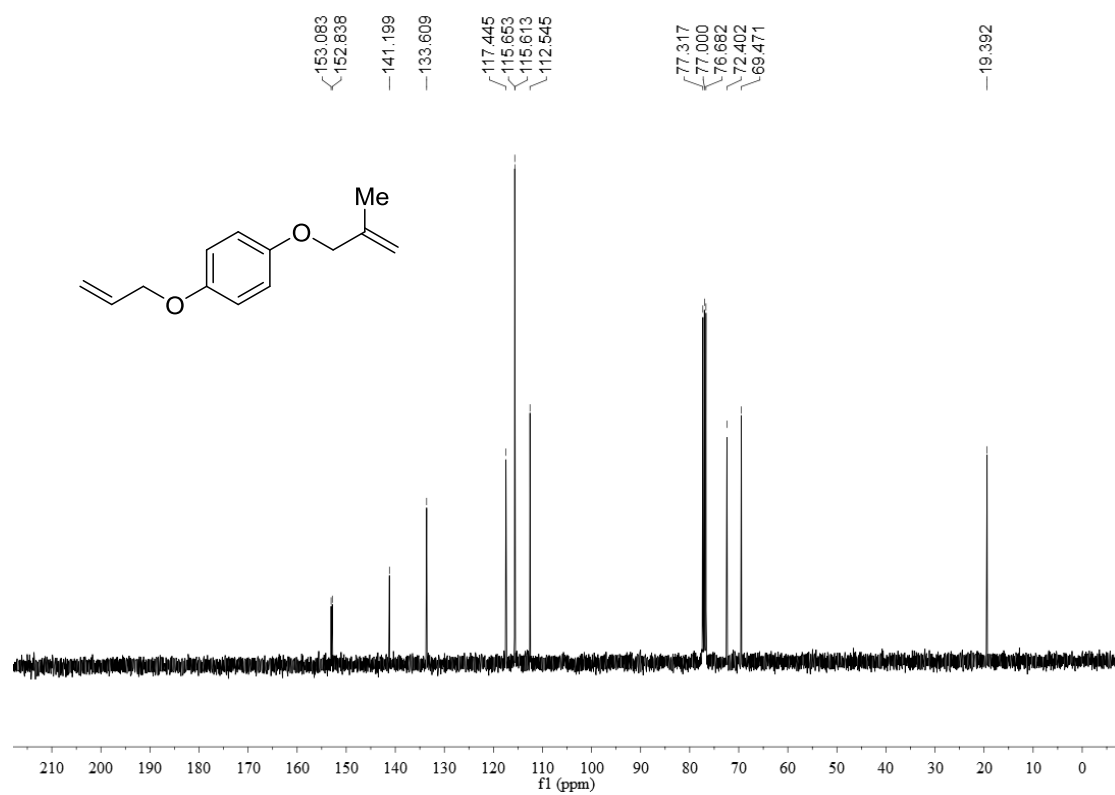


Figure S78. ¹³C NMR spectra of **1aa**.

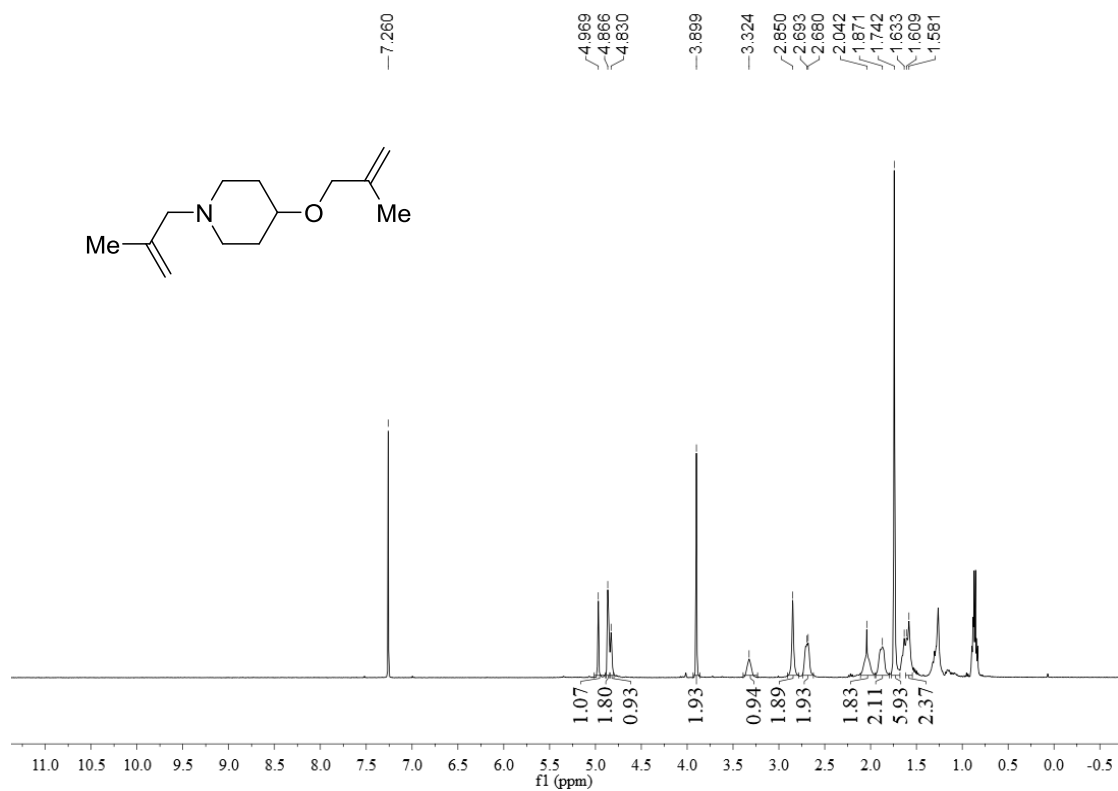


Figure S79. ¹H NMR spectra of **1ab**.

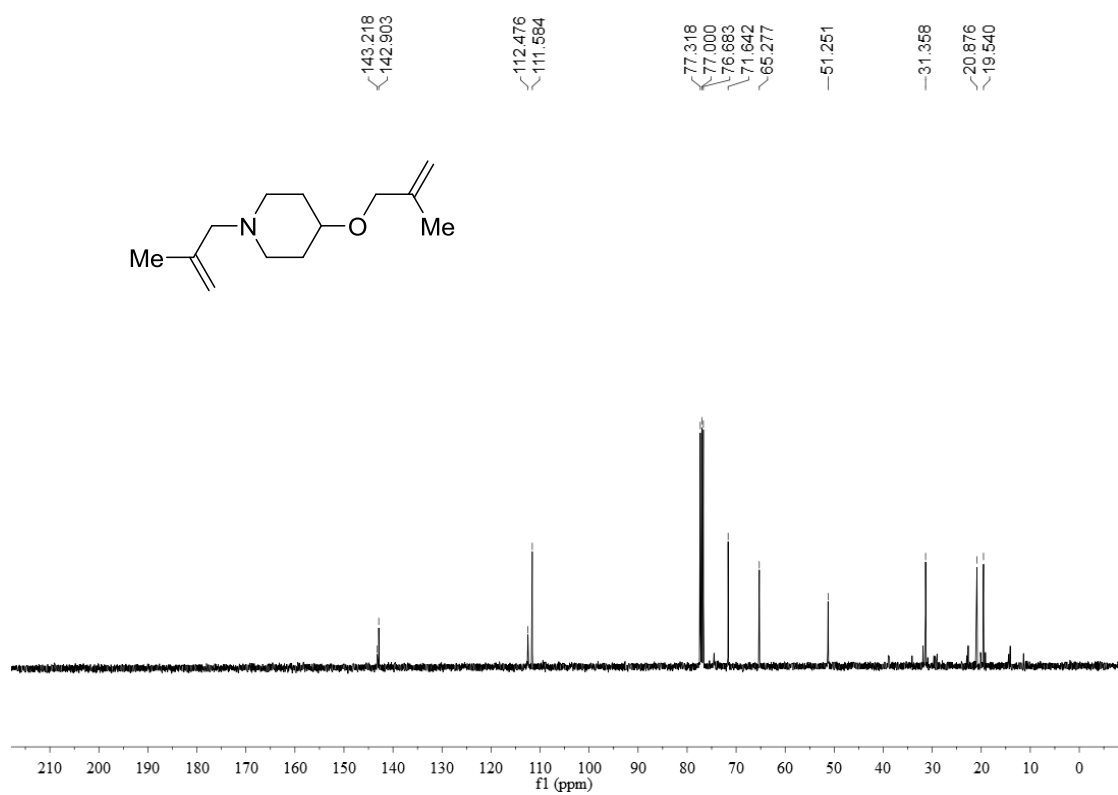


Figure S80. ¹³C NMR spectra of **1ab**.

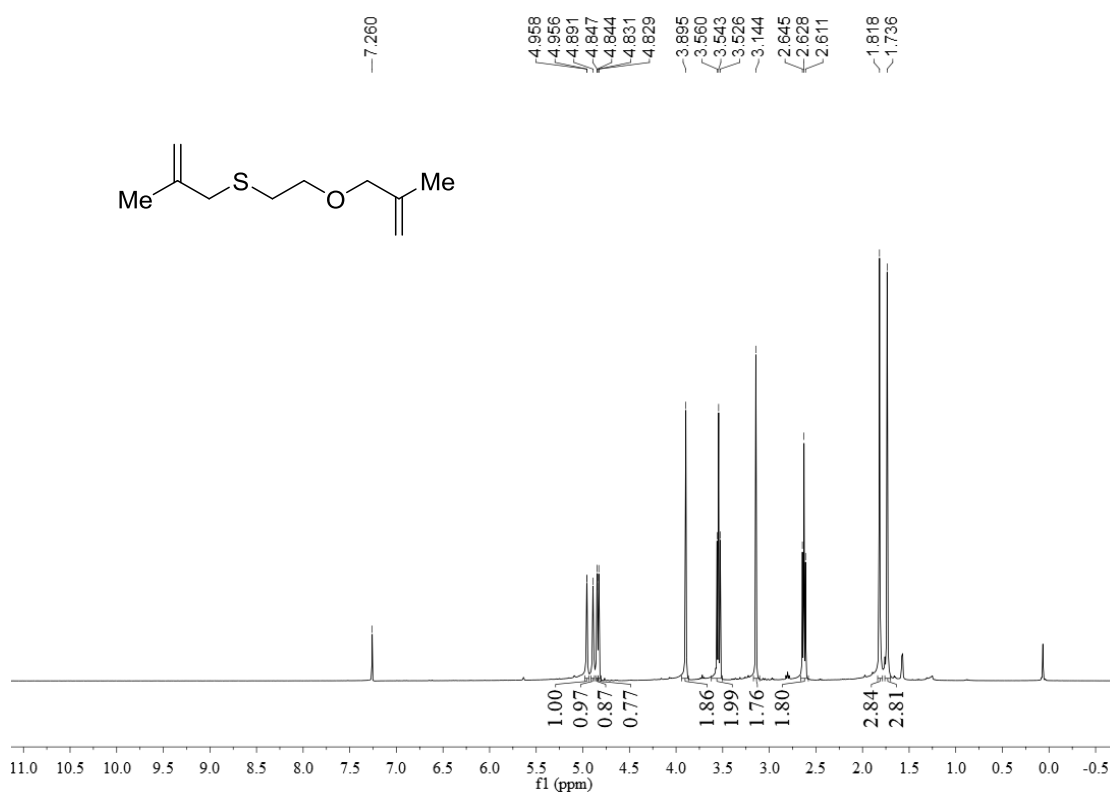


Figure S81. ¹H NMR spectra of **1ac**.

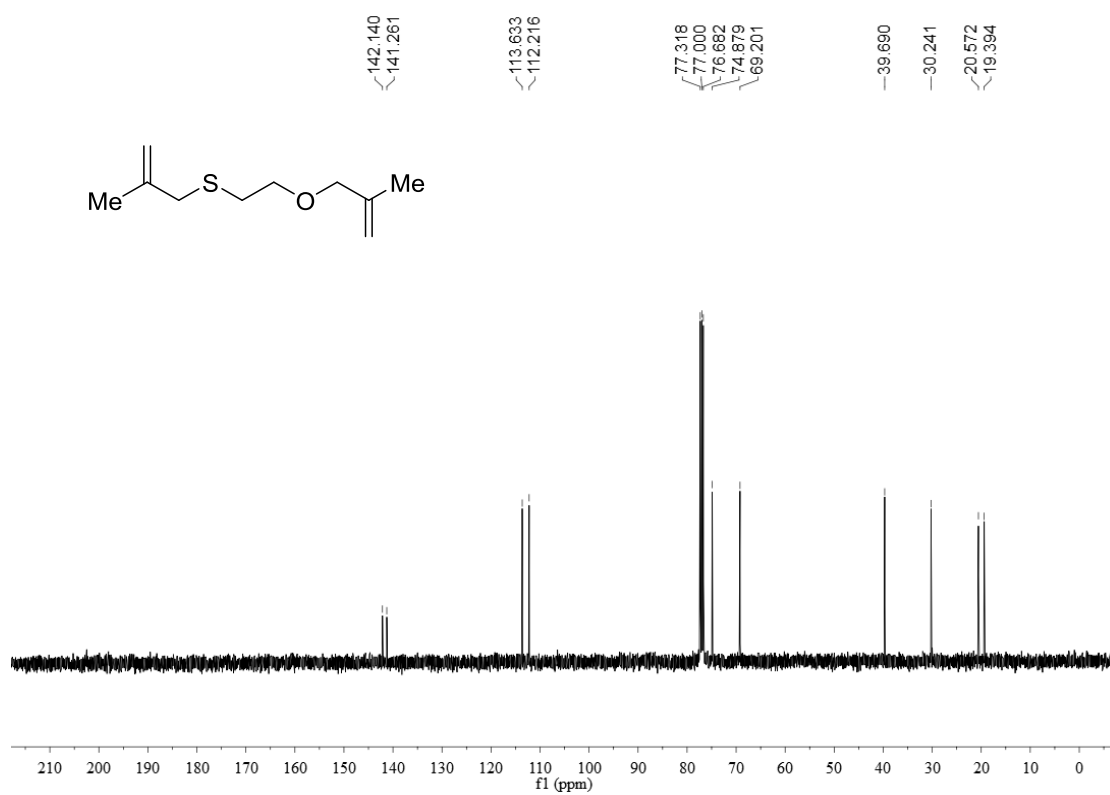


Figure S82. ¹³C NMR spectra of **1ac**.

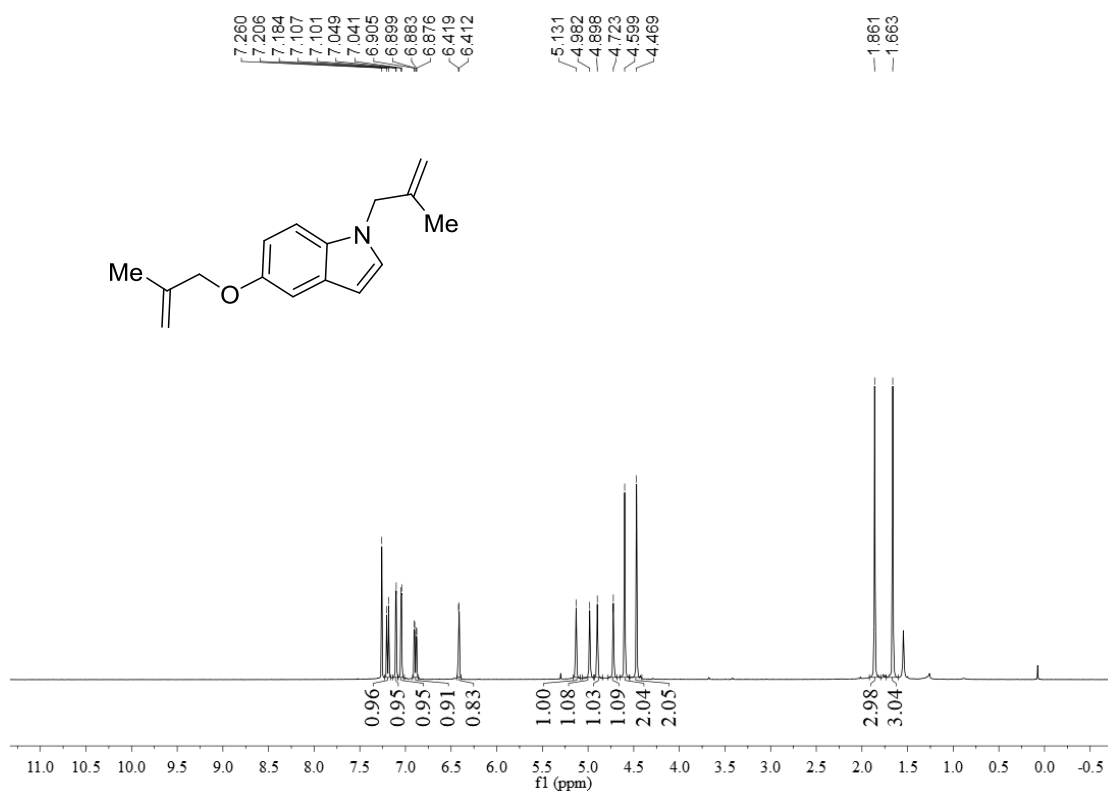


Figure S83. ¹H NMR spectra of **1ad**.

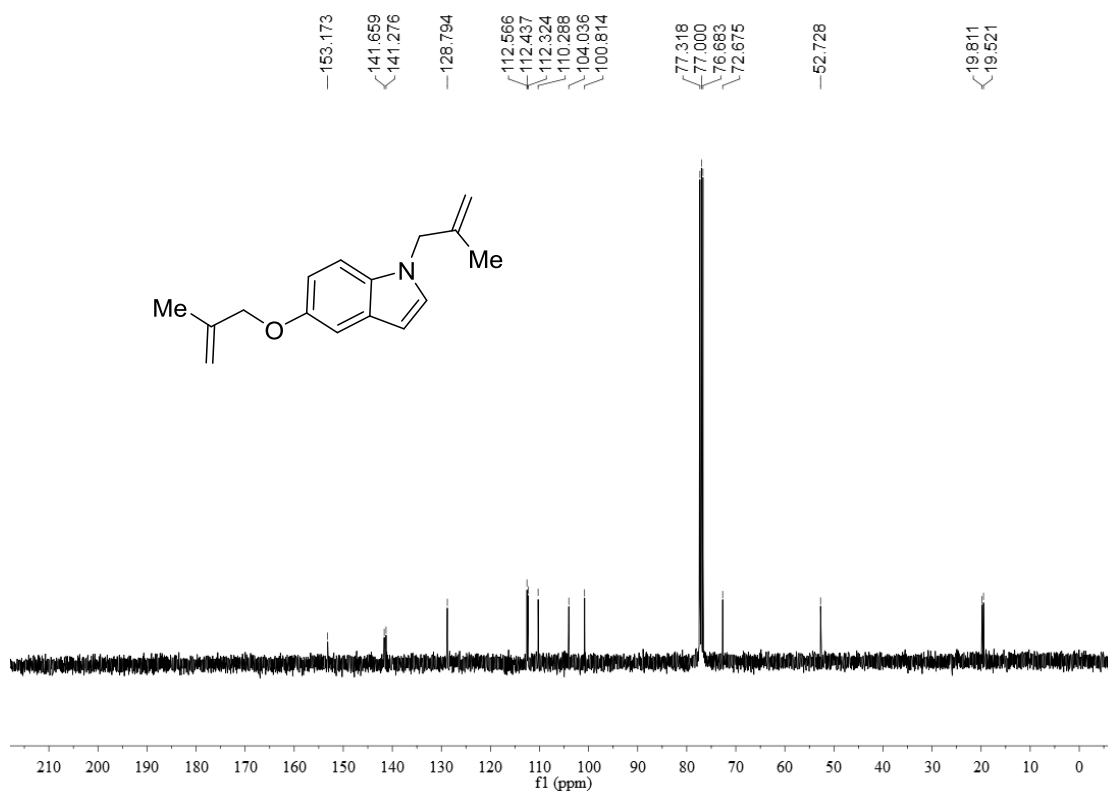


Figure S84. ¹³C NMR spectra of **1ad**.

VI. Supplementary references

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