**Supplementary materials**

# Thioether and ether furofuran lignans: Semisynthesis, reaction mechanism and inhibitory effect against α–glucosidase and free radicals

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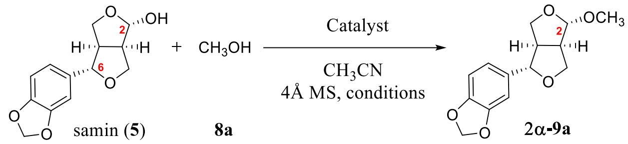
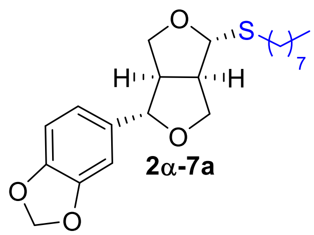
**Contents**

**Table S1**. Optimization of Reaction Conditions[a].



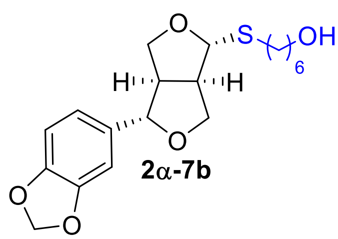

| entry | catalyst (10 mol %) | conditions | yield (%)[b] |
|---|---|---|---|
| 1 | $AlCl_3$ | rt, 2 h | 96 |
| 2 | $AlCl_3$ | 70 °C, 1.5 h | 96 |
| 3 | $BF_3 \cdot Et_2O$ | rt, 2 h | 95 |
| 4 | $BF_3 \cdot Et_2O$ | 70 °C, 1.5 h | 96 |
| 5 | $CH_3CO_2H$ | rt, 24 h | no reaction |
| 6 | $CH_3CO_2H$ | 70 °C, 24 h | no reaction |
| 7 | HCl | rt, 2 h | 95 |
| 8 | HCl | 70 °C, 1.5 h | 95 |
| 9 | $CF_3CO_2H$ | rt, 2 h | 96 |
| 10 | $CF_3CO_2H$ | 70 °C, 1.5 h | 96 |
| 11 | Amberlyst-15[c] | rt, 2 h | 98 |
| 12 | Amberlyst-15[c] | 70 °C, 1.5 h | 98 |

[a]Reaction conditions: **5** (1.0 mmol), $CH_3OH$ (2.0 mmol), and catalyst were mixed in $CH_3CN$ (0.1 M for **5**). The reaction mixture was stirred in the indicated conditions. [b]Isolated yield. [c]200 mg/mmol of **5**.



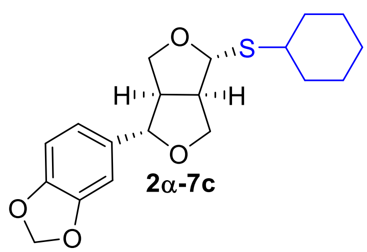

**2α-7a**

1-Octanethiol samin (**2α-7a**). Yield: quant., yellow oil; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 6.86 – 6.75 (m, 3H, H-2′, H-5′ and H-6′), 5.95 (s, 2H, H-7′), 5.38 (s, 1H, H-2), 4.38 (m, 2H, H-6 and H-8), 4.15 (dd, $J$ = 9.3, 5.9 Hz, 1H, H-4), 3.83 (d, $J$ = 9.4 Hz, 1H, H-4), 3.60 (m, 1H, H-8), 2.96 (m, 1H, H-1), 2.77 (m, 1H, H-5), 2.75 (m, 1H, H-1″), 2.55 (m, 1H, H-1″), 1.62 – 1.27 (m, 12H, H-$CH_2$), 0.88 (t, 3H, H-$CH_3$); $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 148.0, 147.3, 134.6, 119.6, 108.2, 106.6, 101.1, 89.0, 86.9, 73.4, 68.3, 53.3, 53.2, 31.8, 31.1, 29.8, 29.2, 29.1, 29.0, 22.6, 14.0; HRESIMS m/z 401.1759 $[M+Na]^+$ (calcd for $C_{21}H_{30}NaO_4S$, 401.1763).



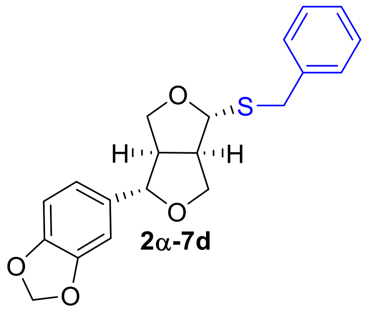

**2α-7b**

6-Mercapto-1-hexanol samin (**2α-7b**). Yield: quant., yellow oil; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 6.86 – 6.75 (m, 3H), 5.94 (s, 2H), 5.26 (s, 1H), 4.42 – 4.32 (m, 2H), 4.12 (dd, $J$ = 9.2, 6.0 Hz, 1H), 3.84 (d, $J$ = 9.4 Hz, 1H), 3.65 – 3.52 (m, 3H), 2.97 (m, 1H), 2.81 – 2.73 (m, 1H), 2.67 (m, 1H), 2.55 (m, 1H), 1.70 – 1.25 (m, 8H); $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 148.0, 147.3, 134.5, 119.6, 108.2, 106.6, 101.1, 89.0, 86.9, 73.4, 68.3, 62.9, 53.3, 53.2, 32.6, 31.0, 29.7, 28.6, 25.3; HRESIMS *m/z* 389.1395 $[M+Na]^+$ (calcd for $C_{19}H_{26}NaO_5S$ , 389.1399).



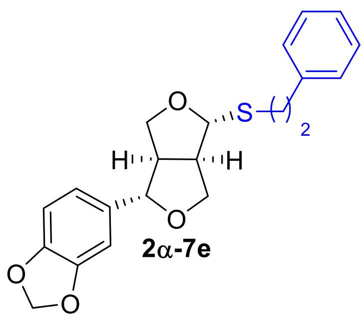

**2α-7c**

Cyclohexanethiol samin (**2α-7c**). Yield: 95%, yellow oil; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 6.86 – 6.75 (m, 3H), 5.95 (s, 2H), 5.38 (s, 1H), 4.42 – 4.35 (m, 2H), 4.15 (dd, $J$ = 9.3, 5.9 Hz, 1H), 3.83 (d, $J$ = 9.4 Hz, 1H), 3.60 (m, 1H), 2.96 (m, 1H), 2.76 (m, 1H), 2.12 – 1.17 (m, 11H); $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 148.0, 147.3, 134.6, 119.6, 108.8, 106.6, 101.1, 87.9, 86.9, 73.5, 68.2, 53.4, 53.3, 43.4, 34.0, 33.8, 26.1, 26.0, 25.8; HRESIMS *m/z* 371.1297 $[M+Na]^+$ (calcd for $C_{19}H_{24}NaO_4S$ , 371.1293).



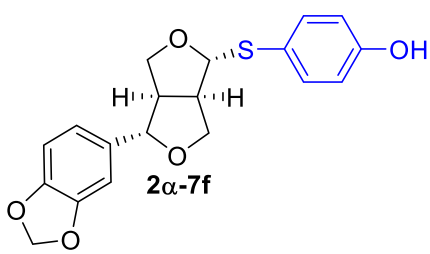

**2α-7d**

*Benzyl mercaptan samin (**2α-7d**)*. Yield: 93%, yellow oil; [1]H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.24 (m, 5H), 6.86 – 6.76 (m, 3H), 5.95 (s, 2H), 5.12 (s, 1H), 4.37 – 4.31 (m, 2H), 4.18 (dd, *J* = 9.4, 5.9 Hz, 1H), 3.91 – 3.85 (m, 2H), 3.71 (d, *J* = 13.5 Hz, 1H), 3.51 (dd, *J* = 8.9, 7.4 Hz, 1H), 2.96 (m, 1H), 2.79 (m, 1H); [13]C NMR (100 MHz, $CDCl_3$) δ 148.0, 147.4, 138.2, 134.5, 128.9, 128.6, 127.0, 119.6, 108.2, 106.6, 101.1, 87.5, 86.9, 73.4, 68.4, 53.3, 52.8, 34.8; HRESIMS m/z 379.0974 $[M+Na]^+$ (calcd for $C_{20}H_{20}NaO_4S$, 379.0980).



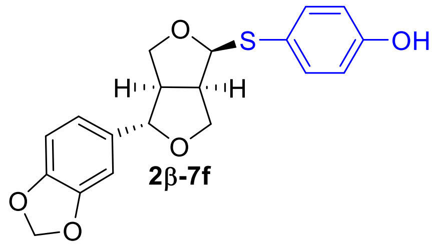

**2α-7e**

*Phenylethyl mercaptan samin (**2α-7e**)*. Yield: 96%, yellow oil; [1]H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.20 (m, 5H), 6.87 – 6.76 (m, 3H), 5.95 (s, 2H), 5.23 (s, 1H), 4.40 – 4.35 (m, 2H), 4.12 (dd, *J* = 9.4, 5.9 Hz, 1H), 3.85 (d, *J* = 9.4 Hz, 1H), 3.56 (m, 1H), 3.01 – 2.73 (m, 5H); [13]C NMR (100 MHz, $CDCl_3$) δ 148.2, 147.5, 140.6, 134.7, 128.7, 128.6, 126.5, 119.8, 108.3, 106.7, 101.2, 89.3, 87.0, 73.6, 68.4, 53.5, 53.3, 36.6, 32.6; HRESIMS m/z 393.1149 $[M+Na]^+$ (calcd for $C_{21}H_{22}NaO_4S$, 393.1136).



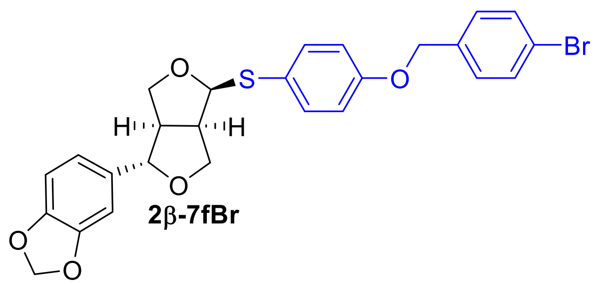

**2α-7f**

*4-Mercaptophenol samin (**2α-7f**)*. Yield: 99%, white powder; [1]H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 8.5 Hz, 2H), 6.87 – 6.71 (m, 5H), 5.95 (s, 2H), 5.37 (s, 1H), 4.44 (t, *J* = 8.7 Hz, 1H), 4.37 (d, *J* = 7.5 Hz, 1H), 4.28 (dd, *J* = 9.4, 5.9 Hz, 1H), 3.93 (d, *J* = 9.4 Hz, 1H), 3.61 (dd, *J* = 8.9, 7.1 Hz, 1H), 3.11 (m, 1H), 2.83 (m, 1H); [13]C NMR (100 MHz, $CDCl_3$) δ 156.1, 148.2, 147.6, 135.2, 134.4, 133.1, 119.9, 116.2, 108.4, 106.7, 101.2, 93.2, 87.0, 73.5, 68.7, 53.3, 53.0; HRESIMS *m/z* 381.0771 $[M+Na]^+$ (calcd for $C_{19}H_{18}NaO_5S$, 381.0773).



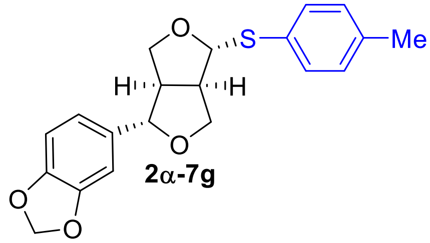
**2β-7f**

*4-Mercaptophenol samin (**2β-7f**)*. Yield: 34%, white powder; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, $J$ = 8.6 Hz, 2H), 6.86 – 6.75 (m, 5H), 5.95 (s, 2H), 5.09 (d, $J$ = 6.1 Hz, 1H), 4.54 (d, $J$ = 6.7 Hz, 1H), 4.21 (dd, $J$ = 9.4, 8.1 Hz, 1H), 4.06-3.99 (m, 2H), 3.81 (dd, $J$ = 9.5, 6.7 Hz, 1H), 3.36 (m, 1H), 2.84 (m, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 155.9, 148.1, 147.4, 134.8, 134.6, 125.6, 119.7, 116.3, 108.3, 106.7, 101.2, 90.9, 86.8, 70.6, 69.2, 54.0, 50.7; HRESIMS *m/z* 381.0770 $[M+Na]^+$ (calcd for $C_{19}H_{18}NaO_5S$, 381.0773).



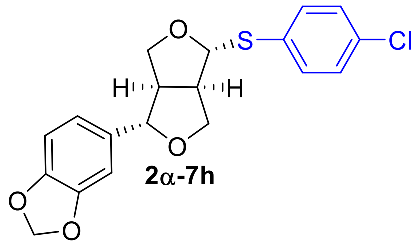
**2β-7fBr**

*Preparation of **2β-7fBr***: A solution of **2β-7f** (1 mmol) in acetonitrile (15 mL) was treated with 4-bromobenzylbromide (1 mmol) and $K_2CO_3$ (3 mmol). After stirring at 70ºC for 4 h, the reaction mixture was evaporated to dryness and purified by silica gel column (10%EtOAc-hexane) to give the product as a white solid. This product was further recrystallized using MeOH to afford **2β-7fBr** (80%) as a white crystal; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.50 (dd, $J$ = 11.3, 8.6 Hz, 4H), 7.29 (d, $J$ = 8.2 Hz, 2H), 6.91 (d, $J$ = 8.7 Hz, 2H), 6.85 (s, 1H), 6.82-6.76 (m, 2H), 5.95 (s, 2H), 5.11 (d, $J$ = 6.1 Hz, 1H), 5.00 (s, 2H), 4.54 (d, $J$ = 6.7 Hz, 1H), 4.21 (t, $J$ = 8.7 Hz, 1H), 4.02 (dd, $J$ = 17.2, 9.7 Hz, 2H), 3.81 (dd, $J$ = 9.4, 6.7 Hz, 1H), 3.38 (dd, $J$ = 15.7, 8.0 Hz, 1H), 2.88-2.82 (m, 1H); HRESIMS *m/z* 549.0349, 551.0329 $[M+Na]^+$ (calcd for $C_{26}H_{23}{}^{79}BrNaO_5S$, 549.0347 and $C_{26}H_{23}{}^{81}BrNaO_5S$, 551.0327).



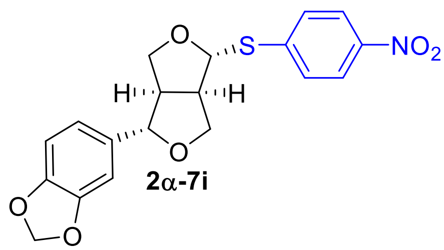
**2α-7g**

*4-Methylthiophenol samin (**2α-7g**)*. Yield: 97%, white powder; $^1$H NMR (400 MHz, $CDCl_3$) δ 7.40 (d, $J$ = 7.7 Hz, 2H), 7.13 (d, $J$ = 7.8 Hz, 2H), 6.88 – 6.76 (m, 3H), 5.96 (s, 2H), 5.47 (s, 1H), 4.44 (t, $J$ = 8.7 Hz, 1H), 4.36 (d, $J$ = 7.5 Hz, 1H), 4.26 (dd, $J$ = 9.4, 5.9 Hz, 1H), 3.92 (d, $J$ = 9.5 Hz, 1H), 3.61 (m, 1H), 3.12 (m, 1H), 2.83 (m, 1H), 2.33 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 148.2, 147.5, 137.7, 134.6, 132.3, 131.2, 129.8, 119.8, 108.3, 106.7, 101.2, 92.5, 87.0, 73.5, 68.7, 53.4, 53.3, 21.2; HRESIMS m/z 379.0987 $[M+Na]^+$ (calcd for $C_{20}H_{20}NaO_4S$ , 379.0980).



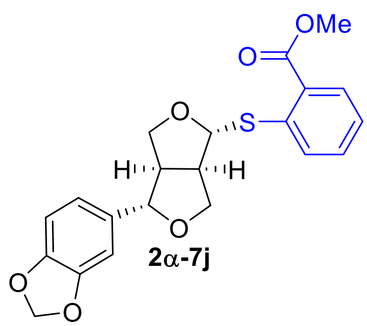
**2α-7h**

*4-Chlorothiophenol samin* (***2α-7h***). Yield: 98%, yellow oil; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.40 (d, *J* = 8.4 Hz, 2H), 7.25 (d, J = 8.4 Hz, 2H), 6.84 – 6.73 (m, 3H), 5.93 (s, 2H), 5.46 (s, 1H), 4.42 (t, *J* = 8.7 Hz, 1H), 4.34 (d, *J* = 7.5 Hz, 1H), 4.21 (dd, *J* = 9.5, 5.9 Hz, 1H), 3.91 (d, *J* = 9.6 Hz, 1H), 3.59 (dd, *J* = 9.0, 7.1 Hz, 1H), 3.09 (m, 1H), 2.81 (m, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 148.0, 147.4, 134.3, 133.5, 133.4, 132.8, 129.0, 119.6, 108.2, 106.5, 101.1, 92.0, 86.9, 73.3, 68.7, 53.2, 53.1.



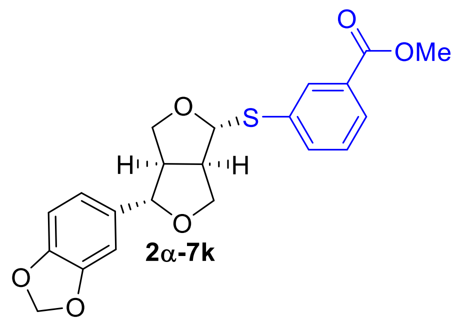
**2α-7i**

*4-nitrothiophenol samin (**2α-7i**)*. Yield: 83%, yellow powder; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 8.13 (d, *J* = 8.8 Hz, 2H), 7.59 (d, *J* = 8.8 Hz, 2H), 6.87 – 6.75 (m, 3H), 5.95 (s, 2H), 5.73 (s, 1H), 4.48 (t, *J* = 8.8 Hz, 1H), 4.41 (d, *J* = 7.5 Hz, 1H), 4.21 (dd, *J* = 9.7, 5.8 Hz, 1H), 3.99 (d, *J* = 9.6 Hz, 1H), 3.67 (dd, *J* = 9.1, 7.0 Hz, 1H), 3.16 (m, 1H), 2.88 (m, 1H).; $^{13}$C NMR (100 MHz, $CDCl_3$) δ 148.2, 147.6, 146.2, 145.8, 134.2, 128.8, 124.0, 119.8, 108.4, 106.6, 101.3, 90.6, 87.0, 73.4, 69.2, 53.3, 53.2.



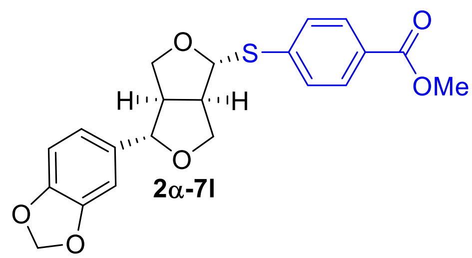
**2α-7j**

*Methyl thiosalicylate samin (**2α-7j**)*. Yield: 78%, white powder; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.93 (dd, *J* = 7.8, 1.1 Hz, 1H), 7.85 (d, *J* = 8.1 Hz, 1H), 7.47 (m, 1H), 7.23 (dd, *J* = 14.2, 6.9 Hz, 1H), 6.88 – 6.75 (m, 3H), 5.95 (s, 2H), 5.68 (s, 1H), 4.48 – 4.41 (m, 2H), 4.23 (dd, *J* = 9.5, 5.8 Hz, 1H), 3.99 – 3.89 (m, 4H), 3.70 (dd, *J* = 9.1, 6.8 Hz, 1H), 3.24 (m, 1H), 2.87 (m, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 167.0, 148.1, 147.4, 139.9, 134.3, 132.4, 130.9, 128.8, 128.4, 125.1, 119.6, 108.2, 106.5, 101.1, 89.8, 86.8, 73.5, 68.9, 53.3, 53.0, 52.2; HRESIMS m/z 423.0884 $[M+Na]^+$ (calcd for $C_{21}H_{20}NaO_6S$ , 423.0878).



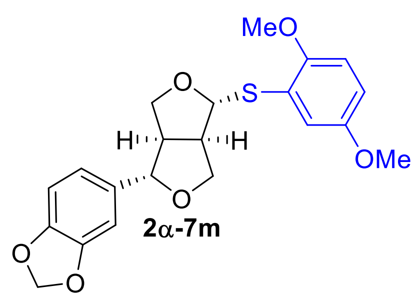

2α-7k

*Methyl 3-mercaptobenzoate samin (**2α-7k**)*. Yield: 85%, white powder; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 8.16 (s, 1H), 7.92 (d, *J* = 7.7 Hz, 1H), 7.68 (d, *J* = 7.8 Hz, 1H), 7.38 (t, *J* = 7.8 Hz, 1H), 6.88 – 6.75 (m, 3H), 5.95 (s, 2H), 5.58 (s, 1H), 4.46 (t, *J* = 8.7 Hz, 1H), 4.38 (d, *J* = 7.5 Hz, 1H), 4.26 (dd, *J* = 9.5, 5.9 Hz, 1H), 3.96 (d, *J* = 9.6 Hz, 1H), 3.92 (s, 3H), 3.64 (dd, *J* = 9.0, 7.1 Hz, 1H), 3.14 (m, 1H), 2.85 (m, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.5, 148.1, 147.4, 135.7, 135.5, 134.3, 132.2, 130.9, 130.8, 128.9, 128.3, 119.6, 108.2, 106.5, 101.1, 91.8, 86.8, 73.3, 68.7, 53.2, 52.2; HRESIMS m/z 423.0884 $[M+Na]^+$ (calcd for $C_{21}H_{20}NaO_6S$ , 423.0878).



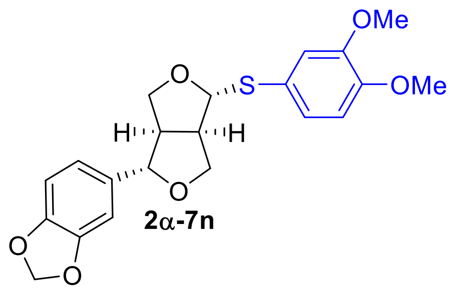

2α-7l

*Methyl 4-mercaptobenzoate samin (**2α-7l**)*. Yield: 90%, white powder; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.4 Hz, 2H), 7.53 (d, *J* = 8.4 Hz, 2H), 6.88 – 6.76 (m, 3H), 5.96 (s, 2H), 5.68 (s, 1H), 4.47 (t, *J* = 8.7 Hz, 1H), 4.40 (d, *J* = 7.5 Hz, 1H), 4.23 (m, 1H), 3.97 (d, *J* = 9.6 Hz, 1H), 3.91 (s, 3H), 3.67 (dd, *J* = 9.0, 7.1 Hz, 1H), 3.15 (m, 1H), 2.86 (m, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.7, 148.1, 147.5, 142.1, 134.2, 129.9, 129.0, 128.8, 119.7, 108.2, 106.5, 101.1, 90.8, 86.9, 73.4, 68.9, 53.2, 53.1, 52.1; HRESIMS m/z 423.0884 $[M+Na]^+$ (calcd for $C_{21}H_{20}NaO_6S$ , 423.0878).



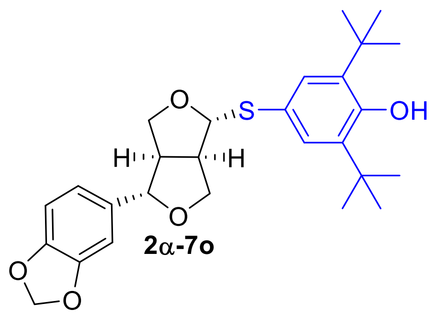

2α-7m

*2,5-Dimethoxythiophenol samin (**2α-7m**)*. Yield: 87%, colorless oil; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.17 (d, *J* = 2.6 Hz, 1H), 6.87 – 6.74 (m, 5H), 5.94 (s, 2H), 5.68 (s, 1H), 4.44 (t, *J* = 8.7 Hz, 1H), 4.37 (d, *J* = 7.6 Hz, 1H), 4.23 (dd, *J* = 9.5, 5.9 Hz, 1H), 3.91 (d, *J* = 9.5 Hz, 1H), 3.84 (s, 3H, H-$OCH_3$), 3.77 (s, 3H, H-$OCH_3$), 3.64 (dd, *J* = 8.9, 7.2 Hz, 1H), 3.17 (m, 1H), 2.84 (m, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 153.9, 152.1, 148.0, 147.4, 134.4, 124.0, 119.6, 118.0, 112.9, 111.7, 108.2, 106.6, 101.1, 89.8, 86.9, 73.4, 68.8, 56.5, 55.8, 53.3, 53.2; HRESIMS m/z 425.1048 $[M+Na]^+$ (calcd for $C_{21}H_{22}NaO_6S$ , 425.1035).



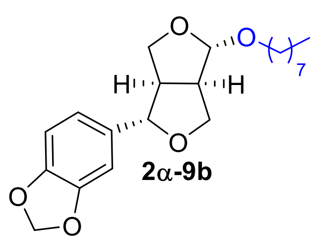
2α-7n

*3,4-Dimethoxythiophenol samin (**2α-7n**)*. Yield: 87%, colorless oil; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.09 (dd, *J* = 8.3, 1.8 Hz, 1H), 7.05 (d, *J* = 1.6 Hz, 1H) 6.81 (m, 4H), 5.95 (s, 2H), 5.42 (s, 1H), 4.44 (t, *J* = 8.7 Hz, 1H), 4.37 (d, *J* = 7.5 Hz, 1H), 4.27 (dd, *J* = 9.4, 5.9 Hz, 1H), 3.93 (d, *J* = 9.5 Hz, 1H), 3.89 (s, 3H, H-$OCH_3$), 3.87 (s, 3H, H-$OCH_3$), 3.62 (dd, *J* = 8.9, 7.1 Hz, 1H), 3.11 (m, 1H), 2.82 (m, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 149.3, 149.1, 148.0, 147.4, 134.4, 125.8, 125.5, 119.6, 116.1, 111.6, 108.2, 106.5, 101.9, 92.9, 86.8, 73.4, 68.6, 56.0, 56.0, 53.3, 53.0; HRESIMS m/z 425.1048 $[M+Na]^+$ (calcd for $C_{21}H_{22}NaO_6S$ , 425.1035).



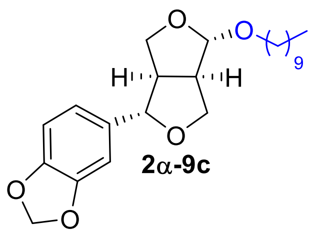
2α-7o

*2,6-Di-tert-butyl-4-mercaptophenol samin (**2α-7o**)*. Yield: 89%, yellow oil; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.33 (s, 2H), 6.87 – 6.75 (m, 3H), 5.95 (s, 2H), 5.39 (s, 1H), 4.43 (t, *J* = 8.7 Hz, 1H), 4.37 (d, *J* = 7.5 Hz, 1H), 4.29 (dd, *J* = 9.4, 5.9 Hz, 1H), 3.93 (d, *J* = 9.4 Hz, 1H), 3.61 (m, 1H), 3.11 (m, 1H), 2.81 (m, 1H), 1.43 (s, 18H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 154.2, 148.2, 147.5, 136.7, 130.4, 127.9, 119.8, 108.3, 106.7, 101.2, 93.1, 86.98, 73.5, 68.5, 53.5, 53.2, 34.5, 30.3; HRESIMS m/z 493.2024 $[M+Na]^+$ (calcd for $C_{27}H_{34}NaO_5S$ , 493.2025).



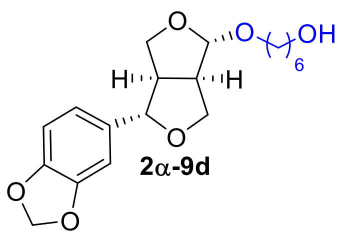
2α-9b

*Octanol samin (**2α-9b**)*. Yield: quant., pale yellow oil; $^{1}$H NMR (400 MHz, $CDCl_3$) δ 6.85 (d, *J* = 0.8 Hz, 1H), 6.80 (dd, *J* = 8.0, 1.2 Hz, 1H), 6.78 (d, *J* = 7.6 Hz, 1H), 5.94 (s, 2H), 4.93 (s, 1H), 4.38 (d, *J* = 8.8 Hz, 1H), 4.34 (t, *J* = 7.2 Hz, 1H), 3.97 (dd, *J* = 8.8, 6.0 Hz, 1H), 3.85 (d, *J* = 8.8 Hz, 1H), 3.65 (m, 1H), 3.55 (dd, *J* = 8.8, 7.6 Hz, 1H), 3.37 (m, 1H), 3.04 (q, *J* = 8.8 Hz, 1H), 2.81 (q, *J* = 9.2 Hz, 1H), 1.59-1.54 (m, 4H), 1.27 (overlap, 8H), 0.87 (t, *J* = 6.8 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 148.1, 147.4, 134.9, 119.8, 108.3, 107.6, 106.7, 101.2, 87.2, 71.6, 69.0, 67.6, 53.2, 52.9, 31.9, 29.8, 29.5, 29.4, 26.3, 22.8, 14.2; HRESIMS *m/z* 385.1997 $[M+Na]^+$ (calcd for $C_{21}H_{30}NaO_5$, 385.1991).



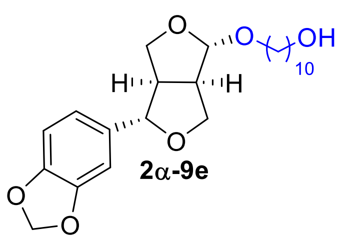
**2α-9c**

*Decanol samin (**2α-9c**)*. Yield: quant., colorless oil; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 6.85 (d, *J* = 1.6 Hz, 1H), 6.81-6.75 (m, 2H), 5.94 (s, 2H), 4.93 (s, 1H), 4.38 (d, *J* = 8.8 Hz, 1H), 4.35 (t, *J* = 7.6 Hz, 1H), 3.97 (dd, *J* = 9.2, 6.0 Hz, 1H), 3.85 (d, *J* = 8.4 Hz, 1H), 3.65 (m, 1H), 3.55 (dd, *J* = 8.8, 7.6 Hz, 1H), 3.36 (m, 1H), 3.04 (q, *J* = 8.8 Hz, 1H), 2.81 (m, 1H), 1.60-1.52 (m, 4H), 1.26 (overlap, 12H), 0.87 (t, *J* = 6.8 Hz, 3H); $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 148.1, 147.4, 134.9, 119.8, 108.3, 107.6, 106.7, 101.2, 87.2, 71.6, 69.0, 67.6, 53.3, 52.9, 32.0, 29.8, 29.7, 29.7, 29.6, 29.4, 26.3, 22.8, 14.2; HRESIMS m/z 413.2306 $[M+Na]^+$ (calcd for $C_{23}H_{34}NaO_5$, 413.2304).



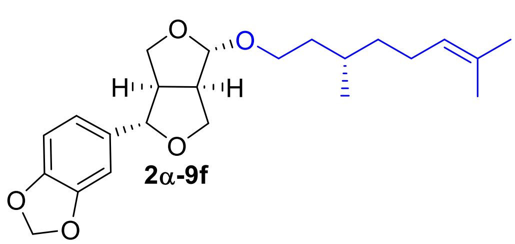
**2α-9d**

*1,6-Hexanediol samin (**2α-9d**)*. Yield: quant., colorless oil; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 6.85 (s, 1H), 6.81-6.76 (m, 2H), 5.94 (s, 2H), 4.93 (s, 1H), 4.38 (d, *J* = 8.8 Hz, 1H), 4.35 (t, *J* = 8.0 Hz, 1H), 3.96 (dd, *J* = 9.2, 6.0 Hz, 1H), 3.85 (d, *J* = 8.8 Hz, 1H), 3.69-3.62 (m, 3H), 3.55 (dd, *J* = 8.8, 8.0 Hz, 1H), 3.38 (m, 1H), 3.03 (q, *J* = 8.4 Hz, 1H), 2.81 (q, *J* =8.8 Hz, 1H), 1.59-1.56 (m, 2H), 1.43-1.33 (m, 6H); $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 148.1, 147.4, 134.9, 119.8, 108.3, 107.6, 106.7, 101.2, 87.2, 71.5, 69.1, 67.4, 64.6, 53.2, 52.9, 32.8, 29.7, 28.7, 25.9; HRESIMS *m/z* 373.1630 $[M+Na]^+$ (calcd for $C_{19}H_{26}NaO_6$, 373.1627).



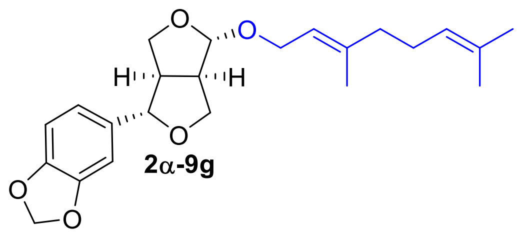
**2α-9e**

*1,10-Decanediol samin (**2α-9e**)*. Yield: quant., colorless oil; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 6.85 (s, 1H), 6.81-6.75 (m, 2H), 5.94 (s, 2H), 4.93 (s, 1H), 4.38 (d, *J* = 8.8 Hz, 1H), 4.35 (t, *J* = 7.6 Hz, 1H), 3.97 (dd, *J* = 8.8, 6.0 Hz, 1H), 3.85 (d, *J* = 8.8 Hz, 1H), 3.68-3.62 (m, 3H), 3.55 (dd, *J* = 8.8, 7.6 Hz, 1H, H-8), 3.37 (m, 1H), 3.04 (q, *J* = 8.4 Hz, 1H), 2.82 (q, *J* =8.8 Hz, 1H), 1.57-1.54 (m, 2H), 1.28-1.25 (m, 14H); $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 148.1, 147.4, 134.9, 119.8, 108.3, 107.6, 106.7, 101.2, 87.2, 71.6, 69.1, 67.6, 63.2, 53.2, 52.9, 32.9, 29.8, 29.8, 29.7, 29.6, 29.5, 26.3, 25.9; HRESIMS m/z 429.2261 $[M+Na]^+$ (calcd for $C_{23}H_{34}NaO_6$, 429.2253).



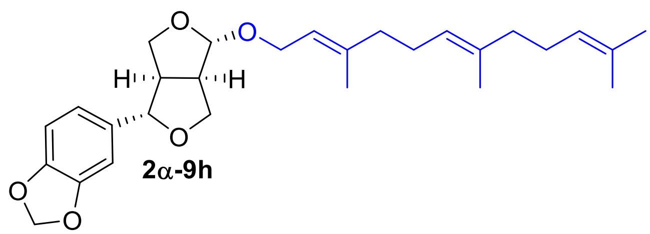
**2α-9f**

*β–Citronellol samin (**2α-9f**)*. Yield: 90%, colorless oil; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 6.85 (s, 1H), 6.81-6.76 (m, 2H), 5.94 (s, 2H), 5.09 (t, *J* = 6.8 Hz, 1H), 4.93 (s, 1H), 4.38 (d, *J* = 8.8 Hz, 1H), 4.35 (t, *J* = 7.2 Hz, 1H), 3.97 (dd, *J* = 9.8, 6.0 Hz, 1H), 3.85 (d, *J* = 8.8 Hz, 1H), 3.70 (m, 1H), 3.55 (dd, *J* = 8.8, 7.6 Hz, 1H), 3.40 (m, 1H), 3.03 (q, *J* = 9.2 Hz, 1H), 2.81 (q, *J* = 6.8 Hz, 1H), 2.03-1.91 (m, 2H), 1.68 (s, 3H), 1.59 (s, 3H), 1.54 (m, 1H), 1.41-1.30 (m, 3H), 1.16 (m, 1H), 0.89 (d, *J* = 6.4 Hz, 3H); $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 148.1, 147.4, 135.0, 131.3, 124.9, 119.8, 108.3, 107.5, 106.7, 101.2, 87.2, 71.6, 69.1, 65.9, 53.3, 53.0, 37.4, 36.8, 29.8, 25.8, 25.6, 19.8, 17.8; HRESIMS m/z 411.2155 $[M+Na]^+$ (calcd for $C_{23}H_{32}NaO_5$, 411.2147).



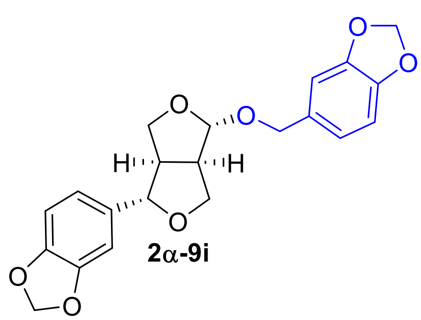
**2α-9g**

*Geraniol samin (**2α-9g**)*. Yield: 89%, colorless oil; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 6.85 (s, 1H), 6.81-6.75 (m, 2H), 5.94 (s, 2H), 5.33 (t, *J* = 6.4 Hz, 1H), 5.09 (m, 1H), 4.99 (s, 1H), 4.38 (d, *J* = 9.2 Hz, 1H), 4.35 (dd, *J* = 8.4, 7.2 Hz, 1H), 4.17 (dd, *J* = 12.0, 6.8 Hz, 1H), 4.02-3.97 (m, 2H), 3.87 (d, *J* = 8.8 Hz, 1H), 3.55 (dd, *J* = 9.2, 7.6 Hz, 1H), 3.06 (m, 1H), 2.82 (m, 1H), 2.10-2.04 (m, 4H), 1.68 (s, 6H), 1.60 (s, 3H); $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 148.1, 147.4, 141.1, 134.9, 131.8, 124.1, 120.2, 119.8, 108.3, 106.8, 106.7, 101.2, 87.2, 71.6, 69.1, 63.6, 53.3, 53.0, 39.8, 26.5, 25.8, 17.8, 16.6; HRESIMS m/z 409.1992 $[M+Na]^+$ (calcd for $C_{23}H_{30}NaO_5$, 409.1991).



**2α-9h**

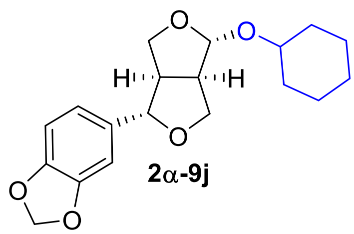
*trans,trans-Farnesol samin (**2α-9h**)*. Yield: 84%, colorless oil; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 6.85 (s, 1H), 6.79-6.75 (m, 2H), 5.94 (s, 2H), 5.33 (m, 1H), 5.10-5.07 (m, 2H), 4.98 (s, 1H), 4.38 (d, *J* = 8.8 Hz, 1H), 4.34 (dd, *J* = 9.6, 7.6 Hz, 1H), 4.18 (m, 1H), 4.01-3.98 (m, 2H), 3.86 (d, *J* = 8.8 Hz, 1H), 3.54 (dd, *J* = 8.8, 8.8 Hz, 1H), 3.06 (m, 1H), 2.81 (m, 1H), 2.12-1.95 (m, 8H), 1.69 (s, 3H), 1.67 (s, 3H), 1.59 (s, 6H); $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 148.1, 147.4, 141.1, 135.5, 134.9, 131.4, 124.5, 123.9, 120.2, 119.8, 108.3, 106.7, 106.7, 101.2, 87.1, 71.5, 69.1, 63.6, 53.2, 53.0, 39.8, 39.7, 26.9, 26.4, 25.8, 17.8, 16.6, 16.1; HRESIMS m/z 477.2617 $[M+Na]^+$ (calcd for $C_{28}H_{38}NaO_5$, 477.2617).



**2α-9i**

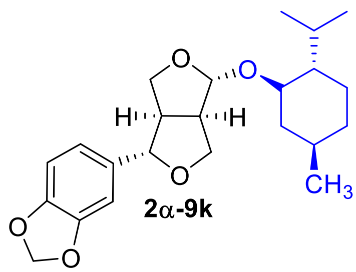
*Piperonyl alcohol samin (**2α-9i**)*. Yield: 45%, white solid; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 6.80 (m, 6H), 5.95 (d, *J* = 1.0 Hz, 4H), 5.02 (s, 1H), 4.60 (d, *J* = 11.4 Hz, 1H), 4.42 – 4.31 (m, 3H), 4.03 (dd, *J* = 9.1, 6.0 Hz, 1H), 3.90 (d, *J* = 9.0 Hz, 1H), 3.57 – 3.51 (m, 1H), 3.09 (dd, *J* = 16.9, 8.7 Hz, 1H), 2.83 (dd, *J* = 15.5, 6.7 Hz, 1H); $^{13}C$ NMR (100

MHz, $CDCl_3$) δ 148.1, 134.8, 131.6, 121.9, 119.8, 108.9, 108.3, 106.7, 106.5, 101.2, 101.2, 87.2, 71.5, 69.3, 68.9, 53.2, 52.9.



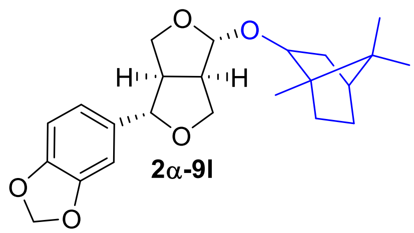
**2α-9j**

*Cyclohexanol samin (**2α-9j**)*. Yield: 93%, colorless oil; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 6.85 (d, $J$ = 0.8 Hz, 1H), 6.81-6.75 (m, 2H), 5.94 (s, 2H), 4.93 (s, 1H), 4.37 (d, $J$ = 8.8 Hz, 1H), 4.34 (dd, $J$ = 8.0, 5.6 Hz, 1H), 4.01 (dd, $J$ = 8.8, 6.0 Hz, 1H), 3.83 (d, $J$ = 8.8 Hz, 1H), 3.55 (m, 2H), 3.02 (q, $J$ = 8.4 Hz, 1H), 2.83 (m, 1H), 1.89-1.52 (m, 10H); $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 148.1, 147.4, 135.0, 119.8, 108.3, 106.8, 105.6, 101.2, 87.3, 74.9, 71.6, 69.0, 53.5, 53.0, 34.0, 32.0, 25.8, 24.5, 24.4; HRESIMS m/z 355.1527 $[M+Na]^+$ (calcd for $C_{19}H_{24}NaO_5$, 355.1521).



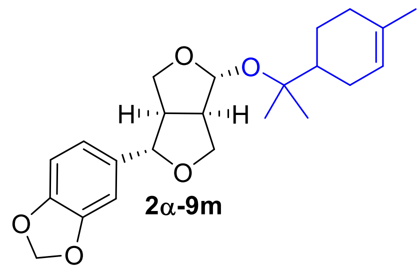
**2α-9k**

*α-Terpeneol samin (**2α-9k**)*. Yield: 89%, colorless oil; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 6.84 (s, 1H), 6.81-6.75 (m, 2H), 5.94 (s, 2H), 5.01 (s, 1H), 4.38 (d, $J$ = 8.8 Hz, 1H), 4.35 (dd, $J$ = 8.4, 6.4 Hz, 1H), 4.05 (dd, $J$ = 9.2, 6.4 Hz, 1H), 3.84 (d, $J$ = 8.8 Hz, 1H), 3.54 (dd, $J$ = 9.2, 7.6 Hz, 1H), 3.31 (ddd, $J$ = 10.4, 10.0, 4.4 Hz, 1H), 3.04 (q, $J$ = 8.0 Hz, 1H), 2.83 (q, $J$ = 8.8 Hz, 1H), 2.13-2.04 (m, 2H), 1.63-1.59 (m, 3H), 1.40 (m, 1H), 1.17 (m, 1H), 1.01-0.94 (m, 2H), 0.91 (d, $J$ = 4.8 Hz, 3H), 0.89 (d, $J$ = 4.4 Hz, 3H), 0.78 (d, $J$ = 6.8 Hz, 3H); $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 148.1, 147.4, 135.1, 119.8, 109.4, 108.3, 106.7, 101.2, 87.3, 79.7, 71.7, 69.1, 53.4, 52.9, 48.9, 43.6, 34.5, 31.9, 25.9, 23.5, 22.4, 21.3, 16.5; HRESIMS m/z 411.2143 $[M+Na]^+$ (calcd for $C_{23}H_{32}NaO_5$, 411.2147).



**2α-9l**

*Borneol samin (**2α-9l**)*. Yield: 90%, colorless oil; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 6.85 (s, 1H), 6.82-6.76 (m, 2H), 5.95 (s, 2H), 4.95 (s, 1H), 4.38 (d, $J$ = 8.8 Hz, 1H), 4.34 (dd, $J$ = 12.8, 8.0 Hz, 1H), 3.98 (dd, $J$ = 8.8, 6.0 Hz, 1H), 3.82 (d, $J$ = 8.8 Hz, 1H), 3.76 (brd, $J$ = 8.0 Hz, 1H), 3.53 (dd, $J$ = 8.8, 7.6 Hz, 1H), 3.07 (q, $J$ = 8.4 Hz, 1H), 2.84 (q, $J$ = 9.2 Hz, 1H), 2.22 (m, 1H), 1.89 (m, 1H), 1.68-1.58 (m, 2H), 1.22-1.15 (m, 2H), 1.01 (dd, $J$ = 13.6, 3.2 Hz, 1H), 0.85 (s, 3H), 0.84 (s, 3H), 0.83 (s, 3H); $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 148.1, 147.4, 135.1, 119.8, 109.2, 108.3, 106.8, 101.2, 87.2, 84.0, 71.7, 69.0, 53.3, 53.1, 49.4, 47.5, 45.2, 37.9, 28.4, 26.9, 19.9, 18.9, 14.0; HRESIMS m/z 409.1992 $[M+Na]^+$ (calcd for $C_{23}H_{30}NaO_5$, 409.1991).



**2α-9m**

*α-Terpineol samin (**2α-9m**)*. Yield: 81%, colorless oil; [1]H NMR (400 MHz, $CDCl_3$) δ 6.84 (d, *J* = 1.6 Hz, 1H), 6.80-6.77 (m, 2H), 5.94 (s, 2H), 5.37 (brs, 1H), 5.24 (s, 1H), 4.36 (d, *J* = 5.6 Hz, 1H), 4.33 (dd, *J* = 6.0, 4.0 Hz, 1H), 4.05 (dd, *J* = 9.2, 6.0 Hz, 1H), 3.80 (d, *J* = 8.8 Hz, 1H), 3.57 (dd, *J* = 8.8, 7.2 Hz, 1H), 2.95 (m, 1H), 2.83 (m, 1H), 2.04-1.96 (m, 4H), 1.83-1.81 (m, 3H), 1.64 (s, 3H), 1.57 (s, 3H), 1.19 (s, 3H); [13]C NMR (100 MHz, $CDCl_3$) δ 148.1, 147.4, 135.3, 134.1, 121.0, 119.7, 108.3, 106.7, 102.3, 101.2, 87.2, 78.5, 71.8, 69.1, 54.2, 53.2, 44.2, 31.2, 26.9, 24.3, 24.0, 23.6, 23.5; HRESIMS m/z 409.1992 $[M+Na]^+$ (calcd for $C_{23}H_{30}NaO_5$, 409.1991)

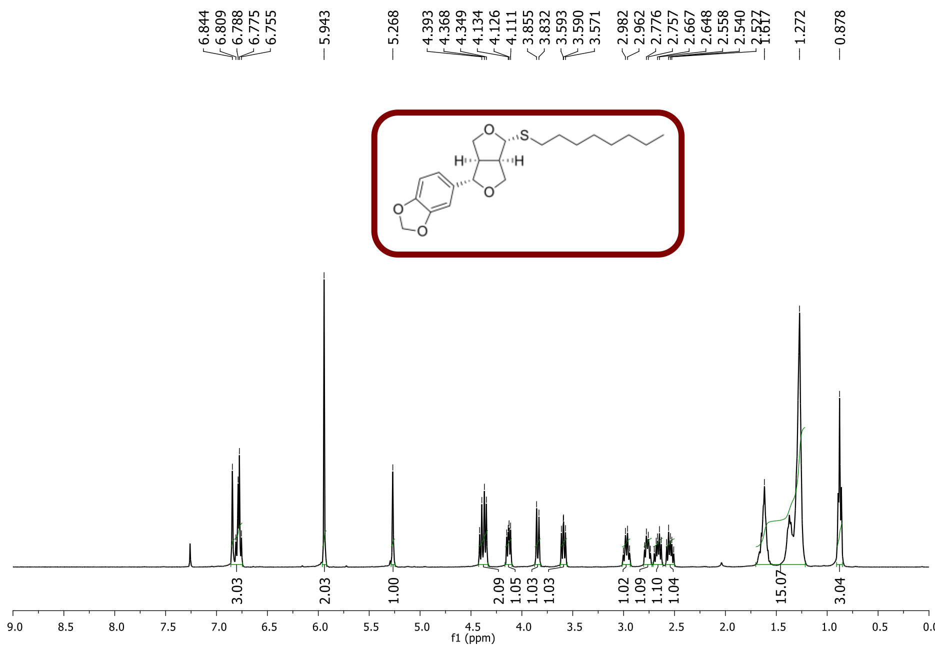



**Figure S1.** $^{1}$H NMR spectrum of **2α-7a** (CDCl$_3$)

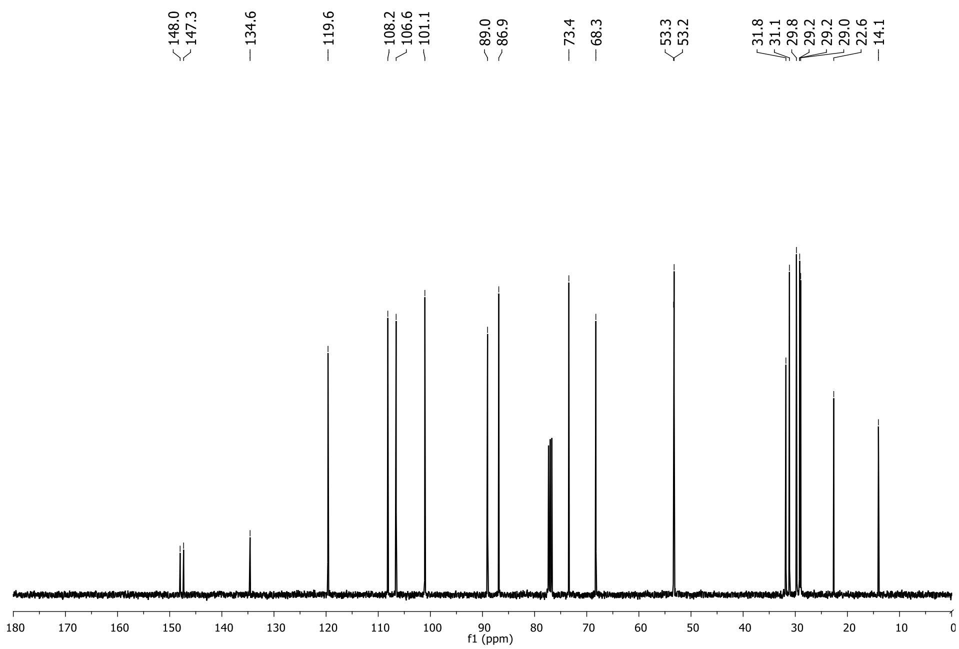



**Figure S2.** $^{13}$C NMR spectrum of **2α-7a** (CDCl$_3$)

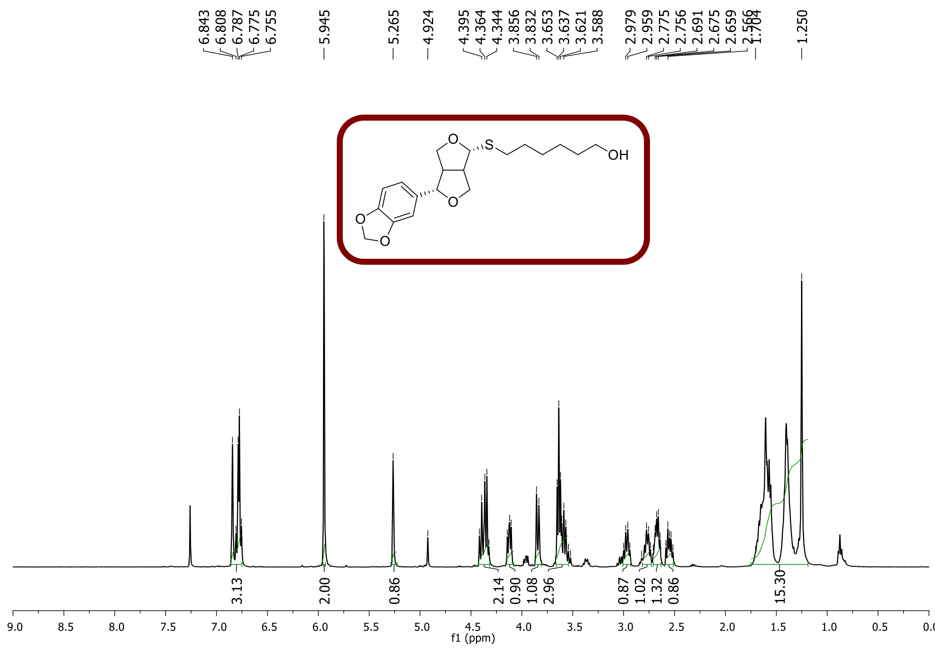


**Figure S3.** $^{1}$H NMR spectrum of **2α-7b** ($CDCl_3$)

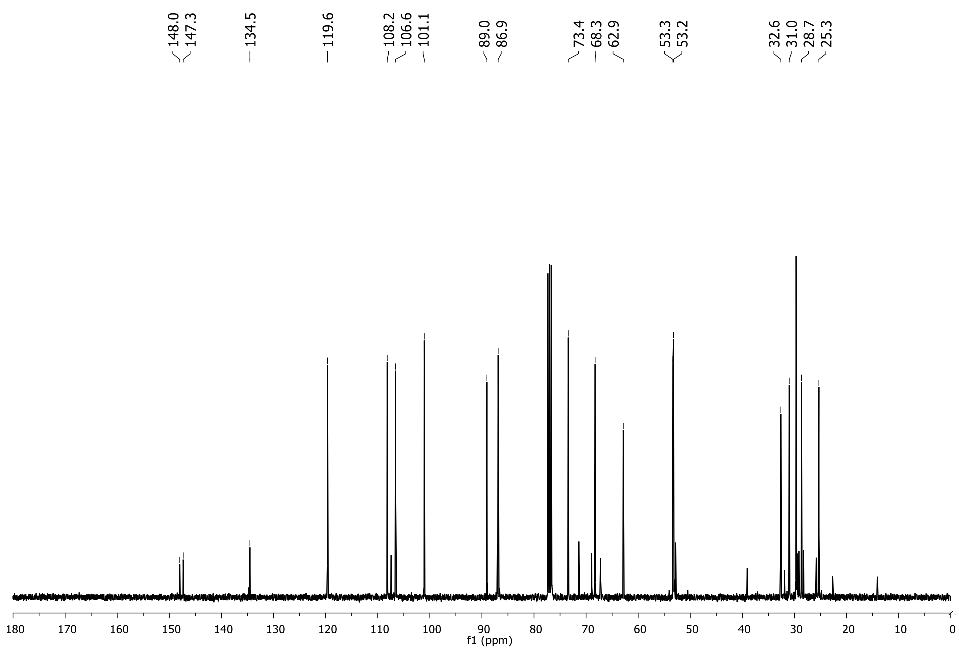


**Figure S4.** $^{13}$C NMR spectrum of **2α-7b** ($CDCl_3$)

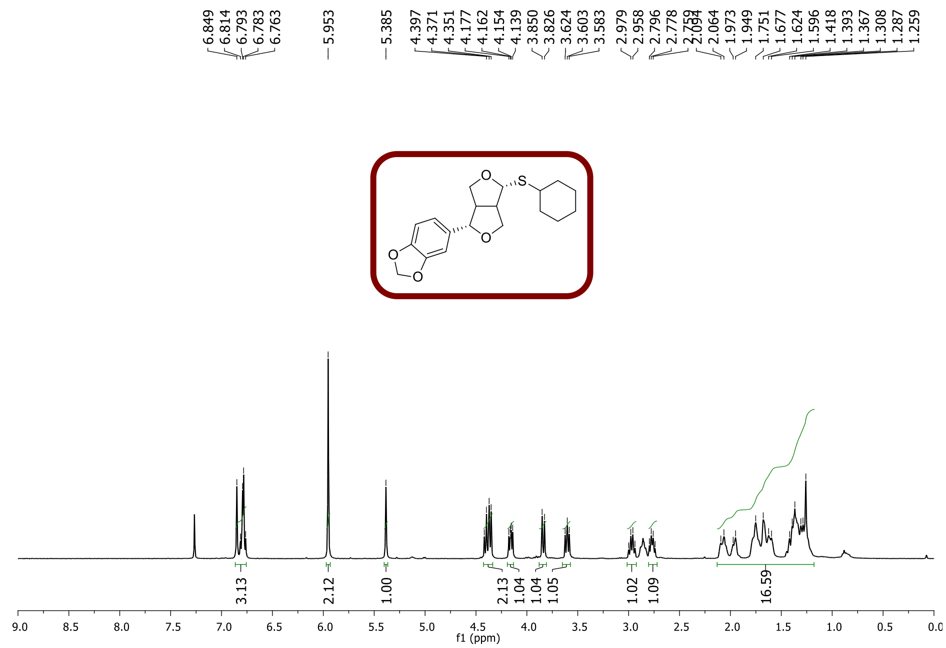



**Figure S5.** $^{1}$H NMR spectrum of **2α-7c** ($CDCl_3$)

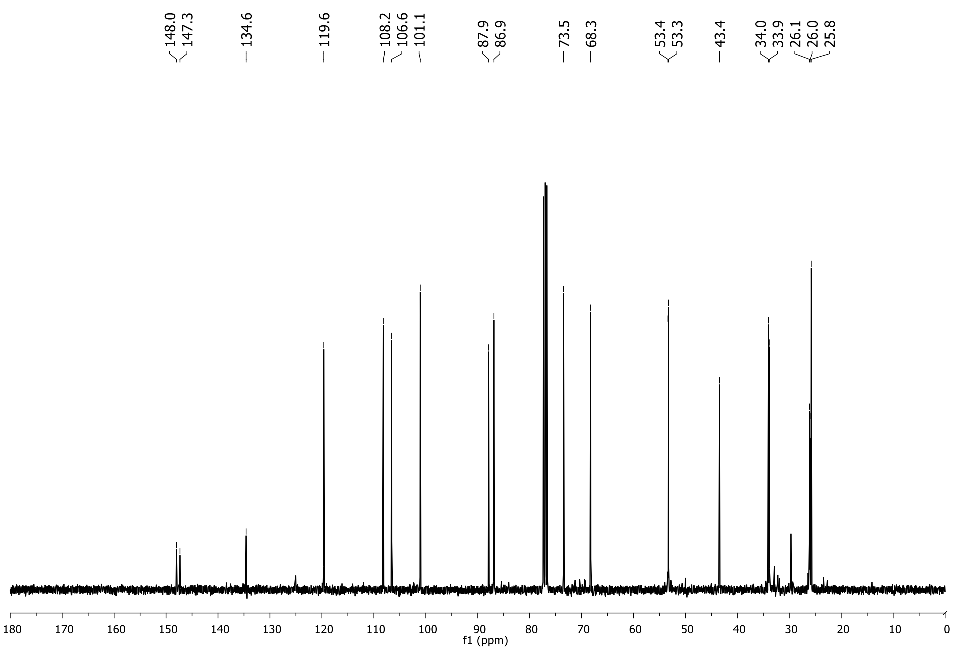



**Figure S6.** $^{13}$C NMR spectrum of **2α-7c** ($CDCl_3$)

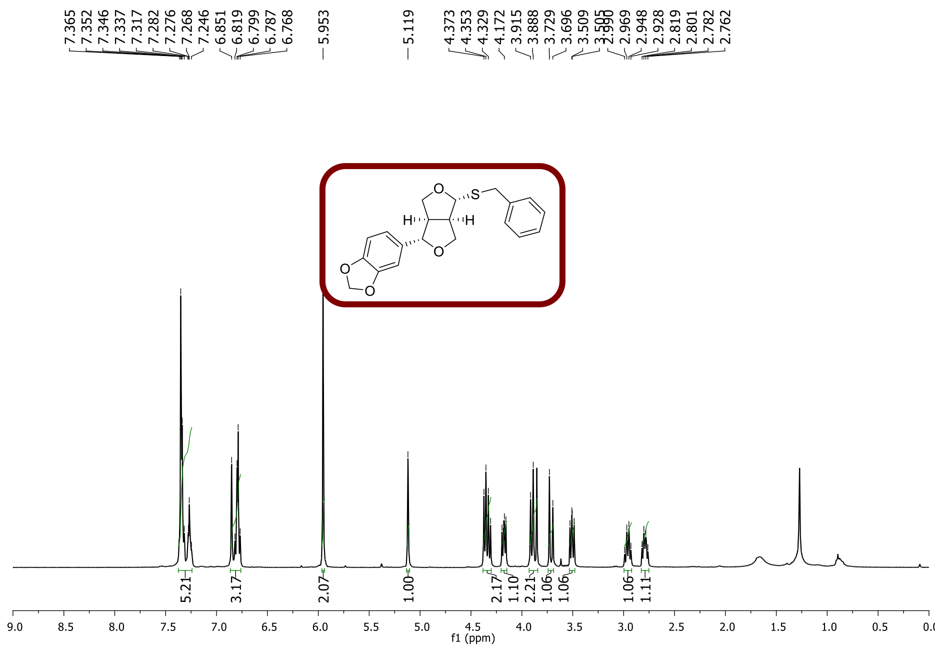



**Figure S7.** $^{1}$H NMR spectrum of **2α-7d** ($CDCl_3$)

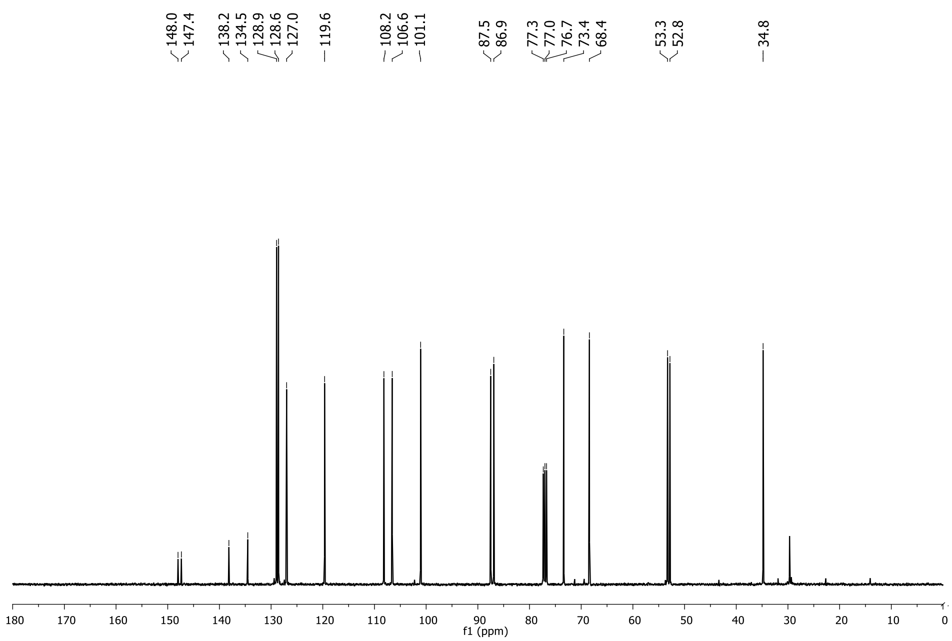



**Figure S8.** $^{13}$C NMR spectrum of **2α-7d** ($CDCl_3$)

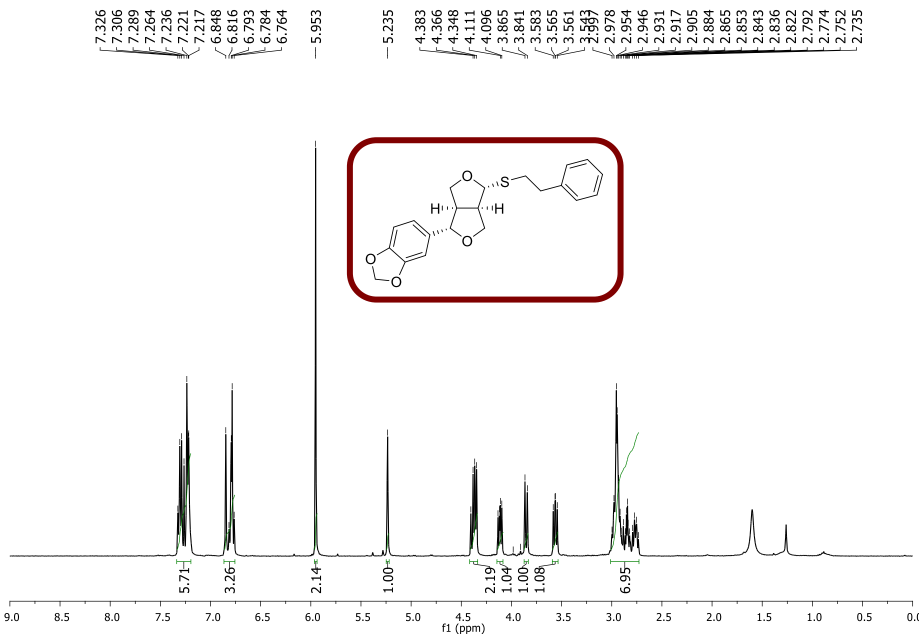


**Figure S9.** $^{1}H$ NMR spectrum of **2α-7e** ($CDCl_3$)

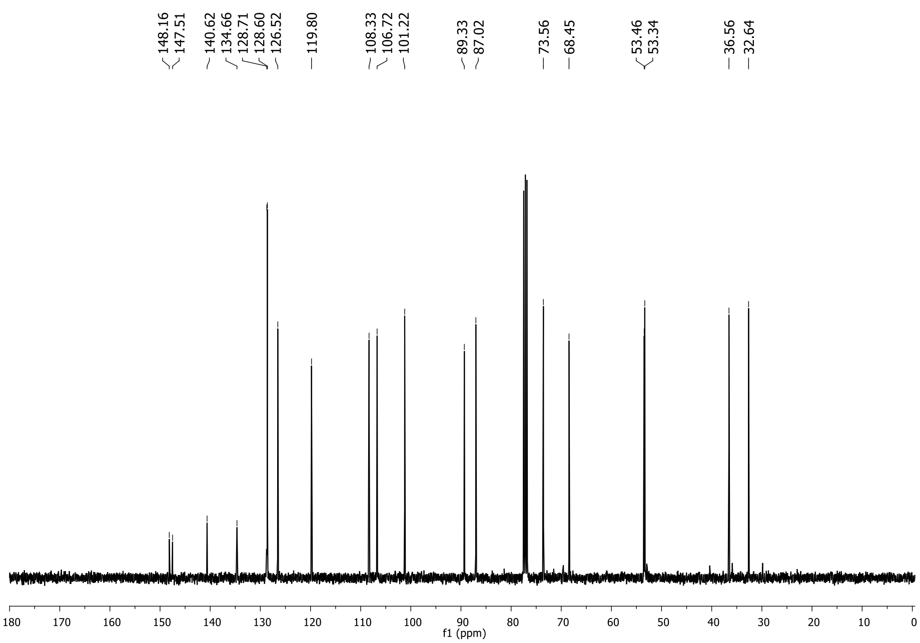


**Figure S10.** $^{13}C$ NMR spectrum of **2α-7e** ($CDCl_3$)

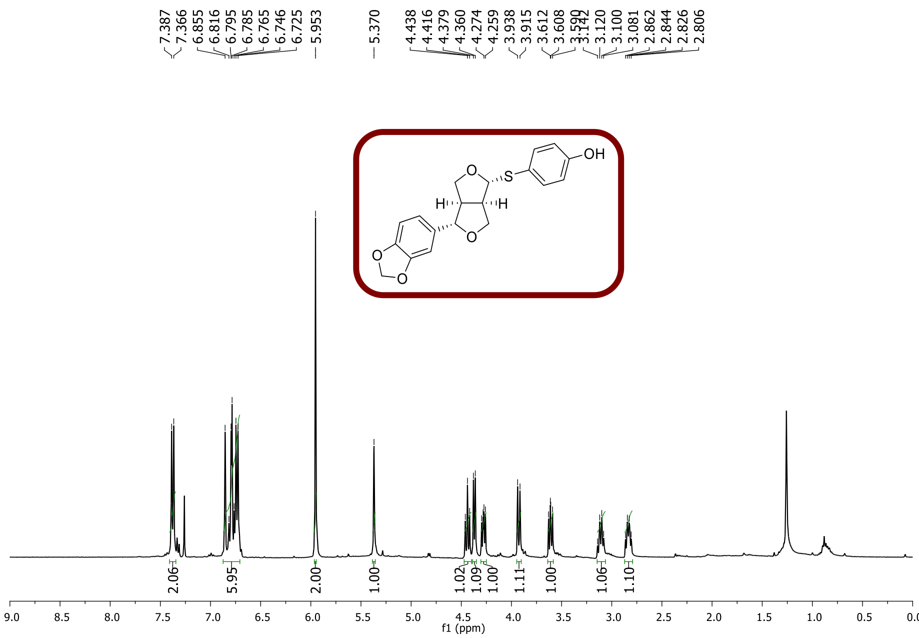


**Figure S11.** $^{1}$H NMR spectrum of **2α-7f** ($CDCl_3$)

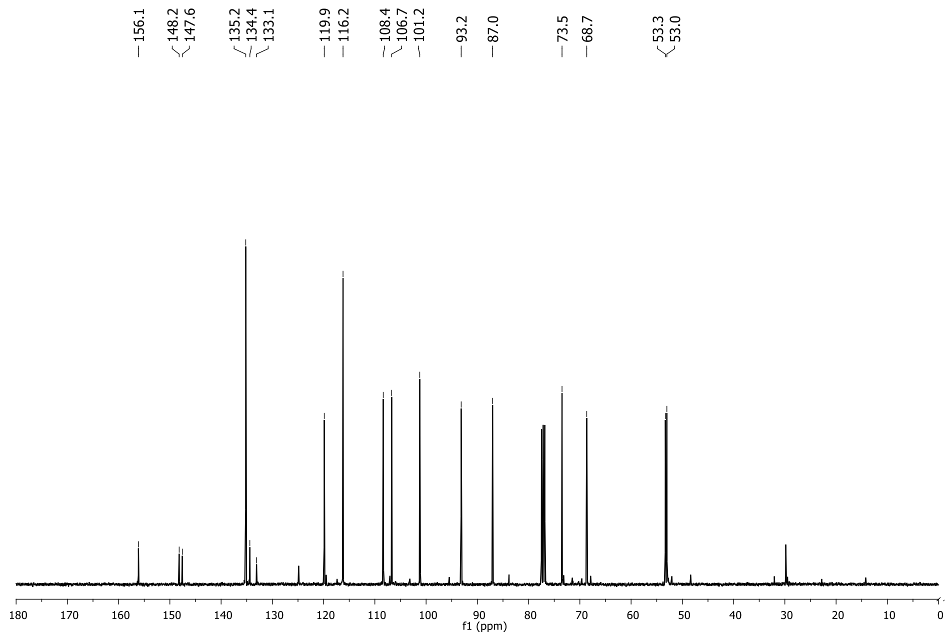


**Figure S12.** $^{13}$C NMR spectrum of **2α-7f** ($CDCl_3$)

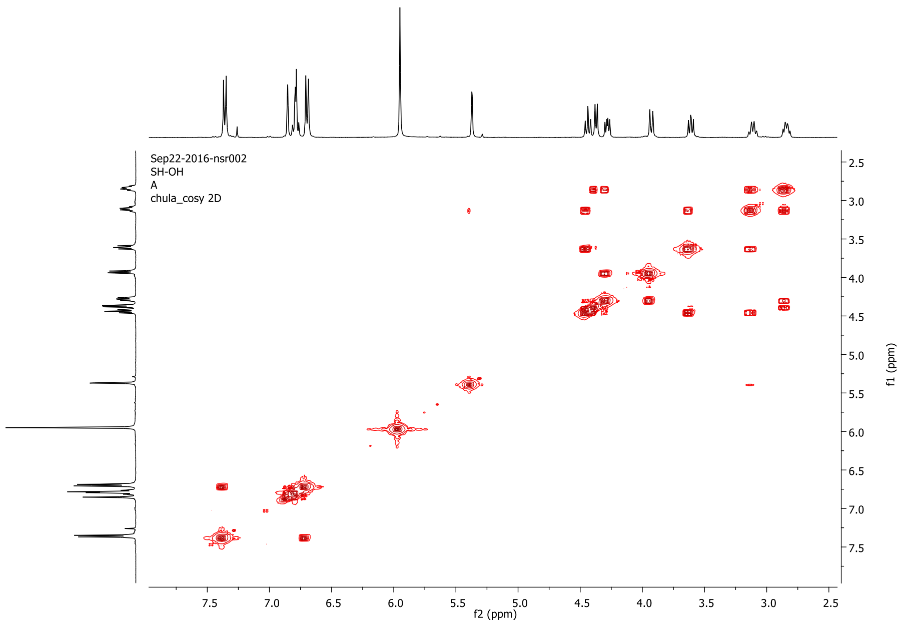



**Figure S13.** COSY spectrum of **2α-7f** ($CDCl_3$)

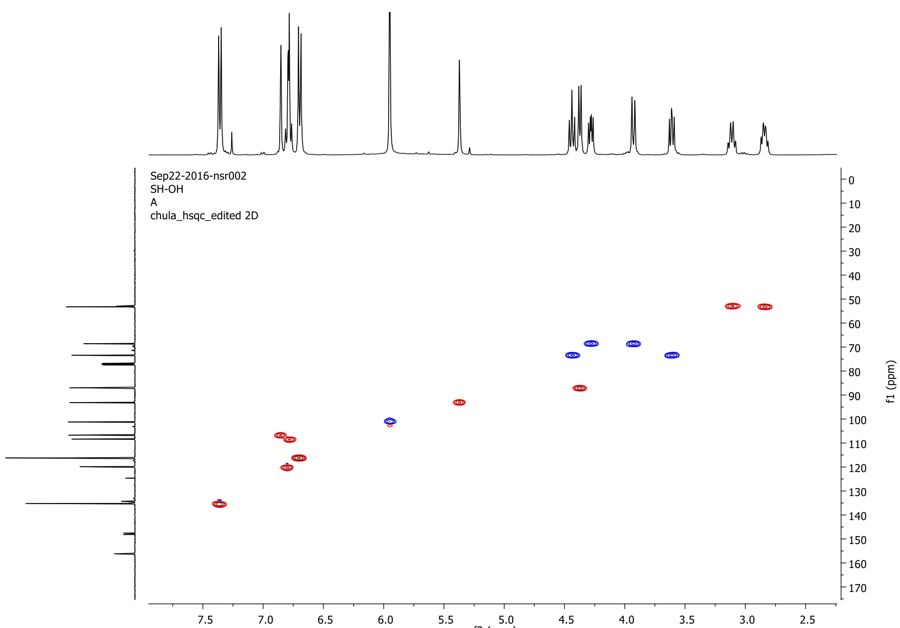



**Figure S14.** HSQC spectrum of **2α-7f** ($CDCl_3$)

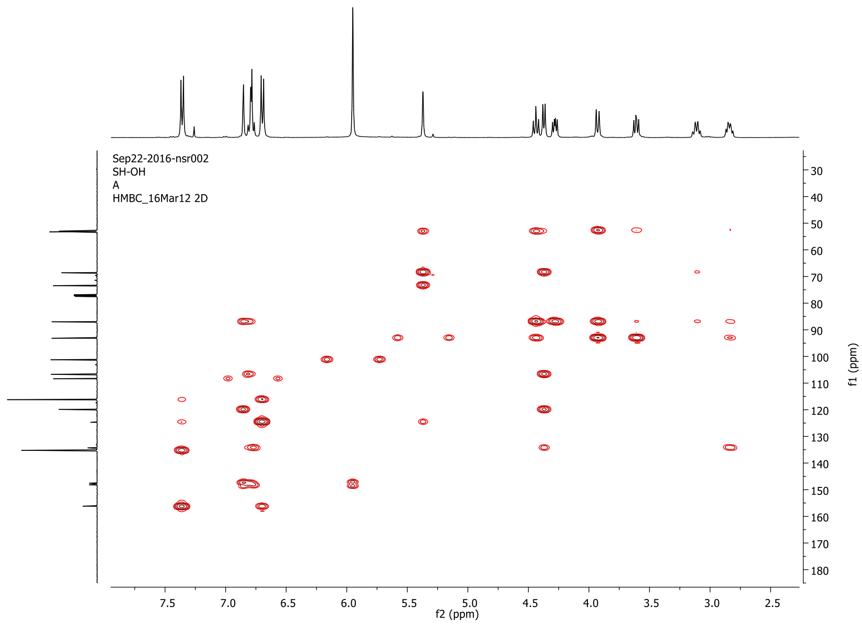



**Figure S15.** HMBC spectrum of **2α-7f** ($CDCl_3$)

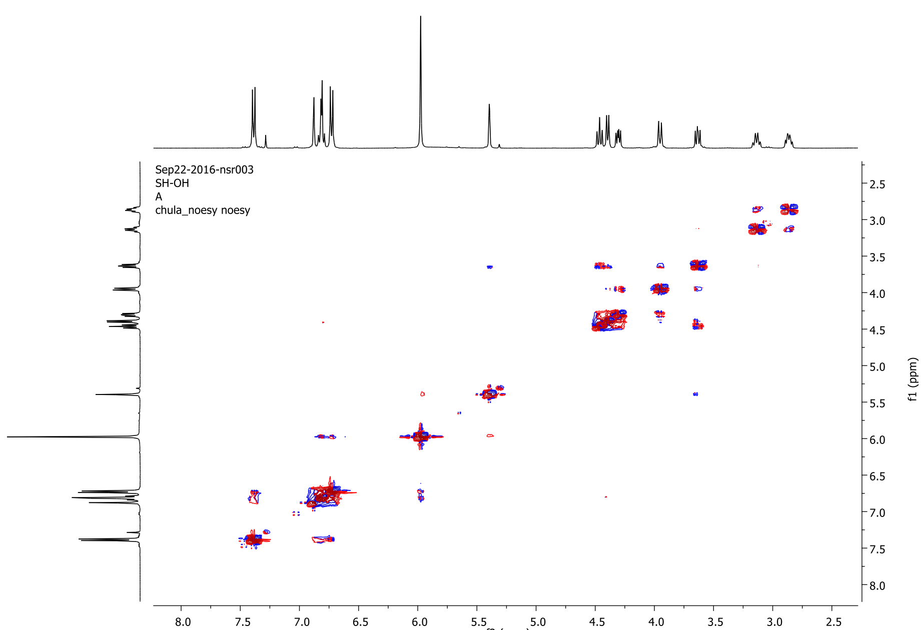



**Figure S16.** NOESY spectrum of **2α-7f** ($CDCl_3$)

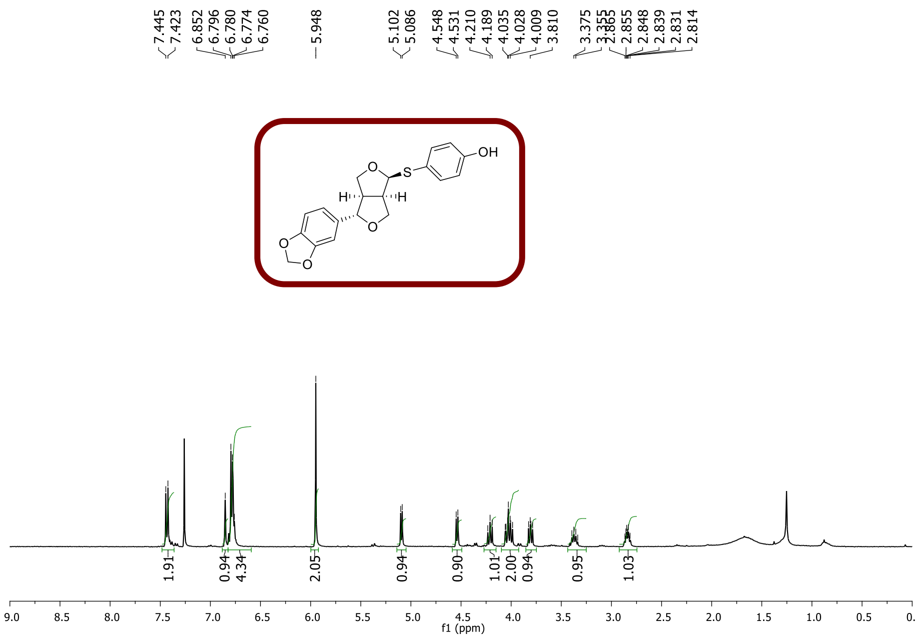



**Figure S17.** $^{1}H$ NMR spectrum of **2β-7f** ($CDCl_3$)

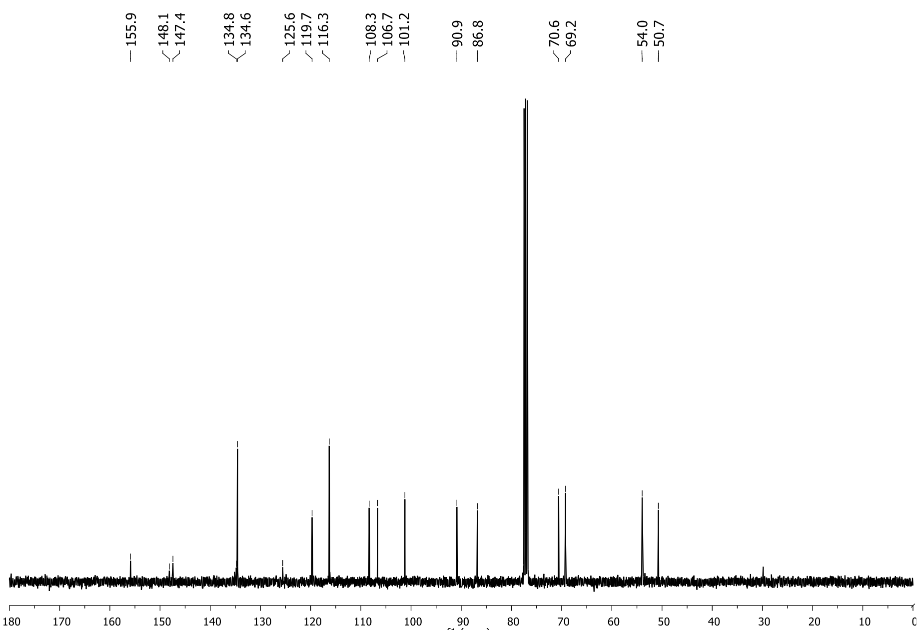



**Figure S18.** $^{13}C$ NMR spectrum of **2β-7f** ($CDCl_3$)

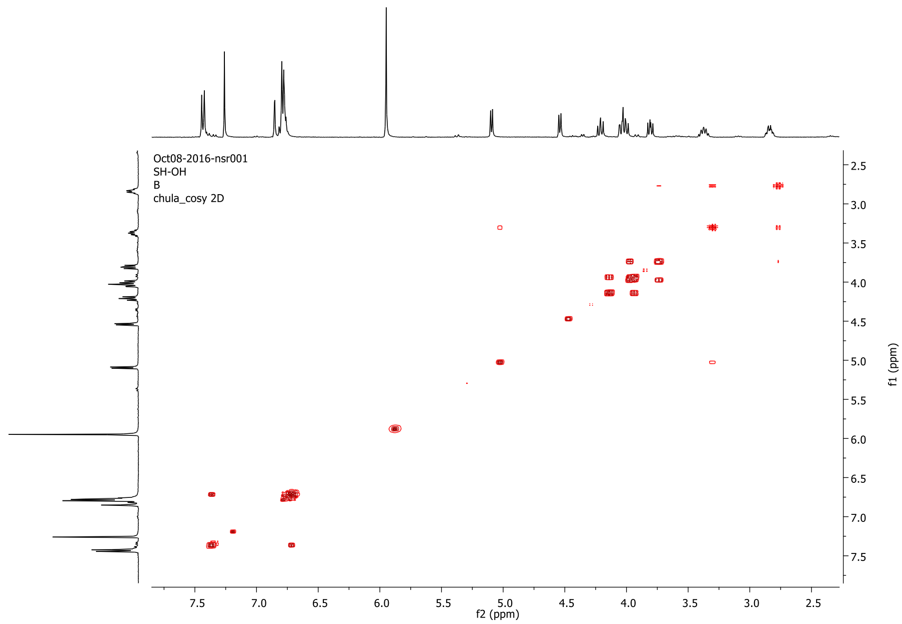



**Figure S19.** COSY spectrum of **2β-7f** ($CDCl_3$)

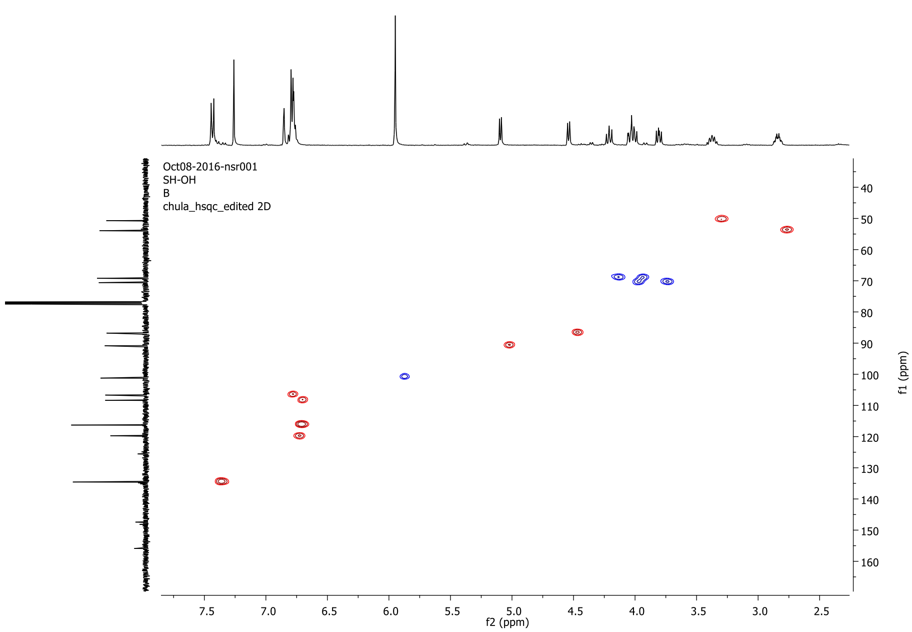



**Figure S20.** HSQC spectrum of **2β-7f** ($CDCl_3$)

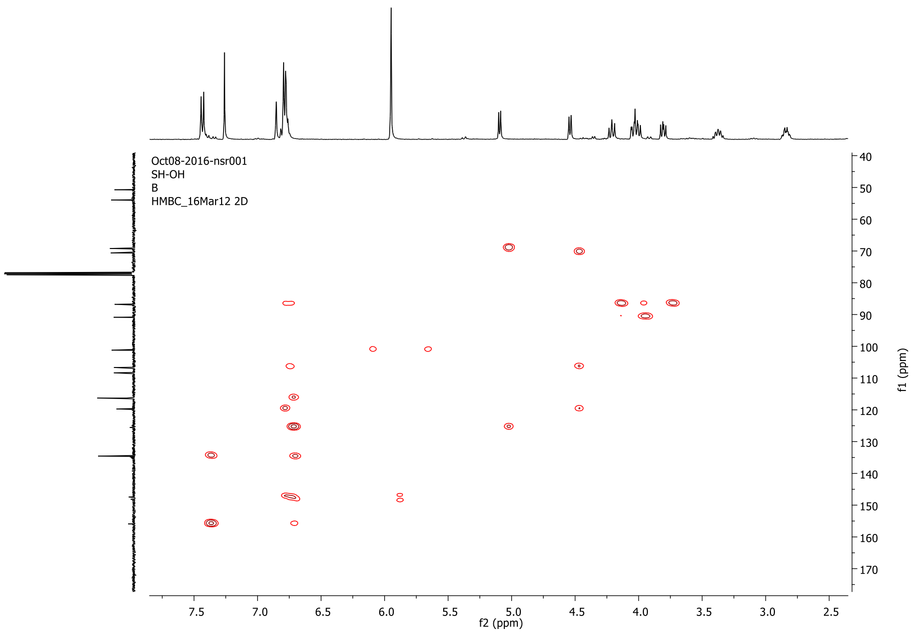



**Figure S21.** HMBC spectrum of **2β-7f** ($CDCl_3$)

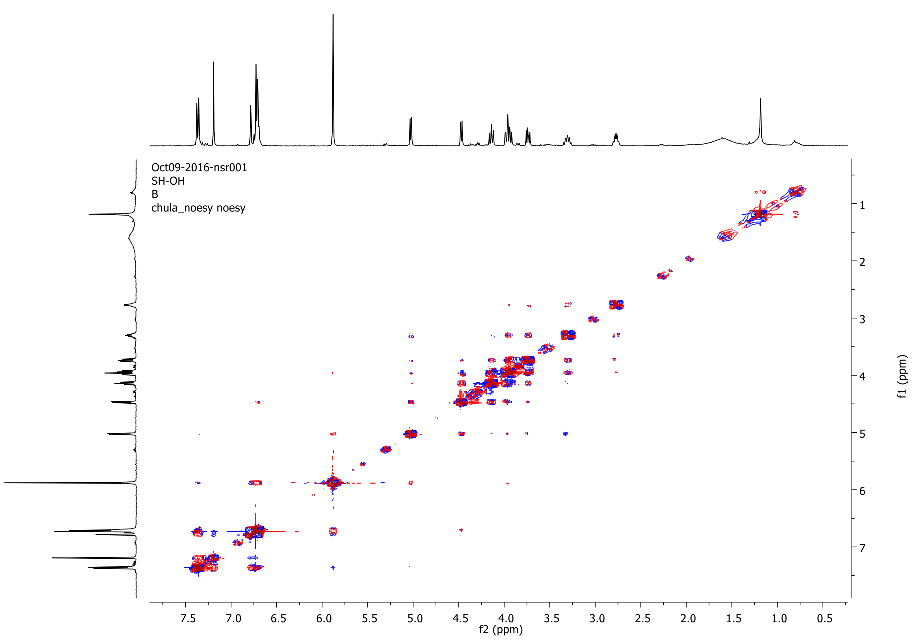



**Figure S22.** NOESY spectrum of **2β-7f** ($CDCl_3$)

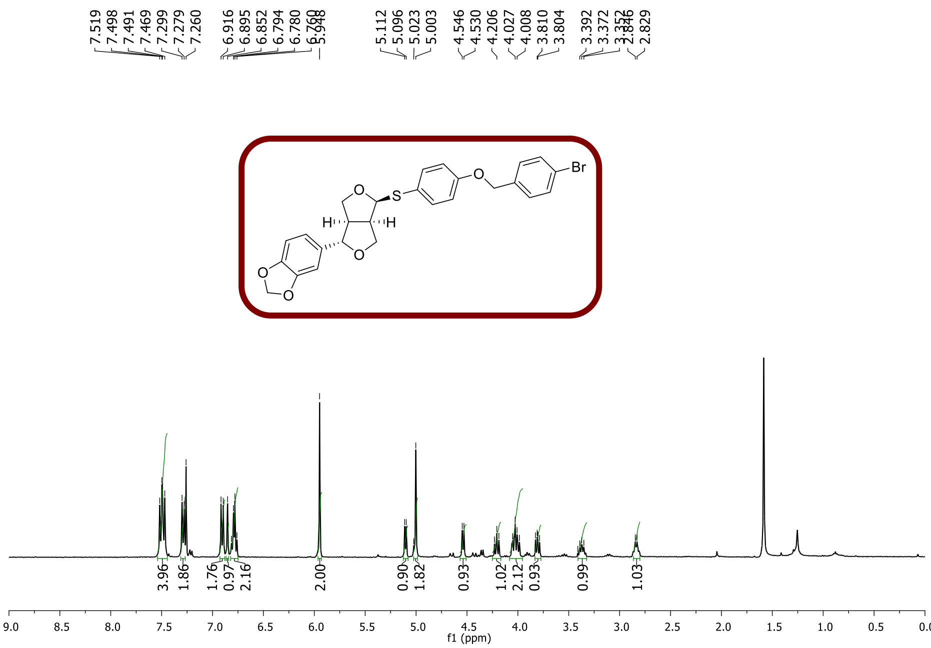


**Figure S23.** $^{1}$H NMR spectrum of **2β-7fBr** ($CDCl_3$)

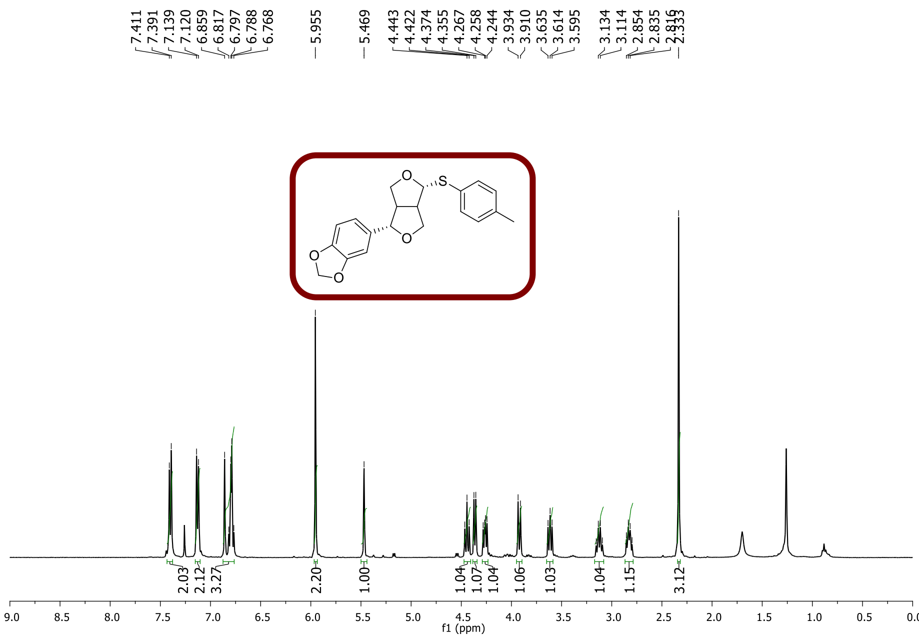



**Figure S24.** $^{1}$H NMR spectrum of **2α-7g** ($CDCl_3$)

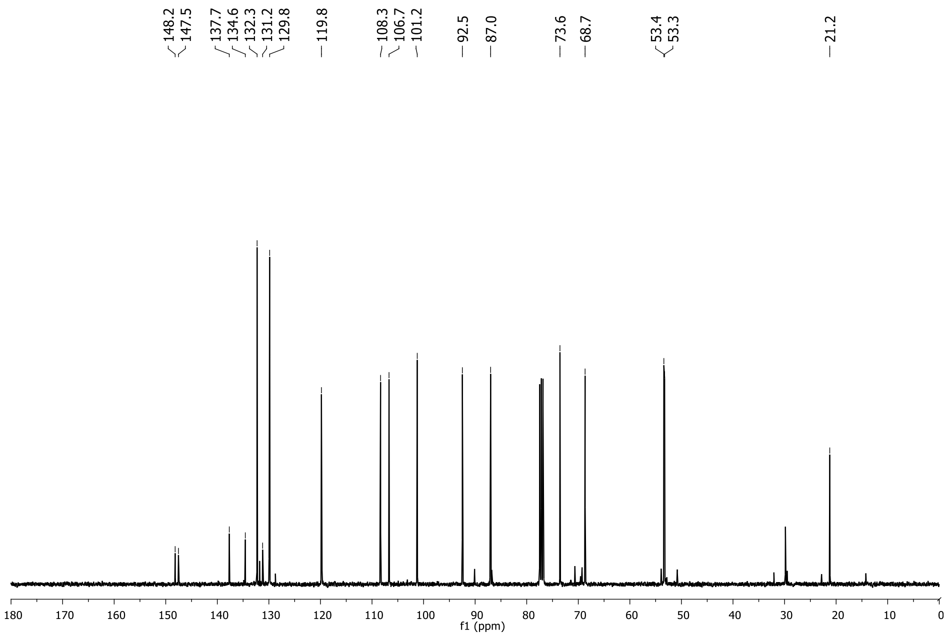



**Figure S25.** $^{13}$C NMR spectrum of **2α-7g** ($CDCl_3$)

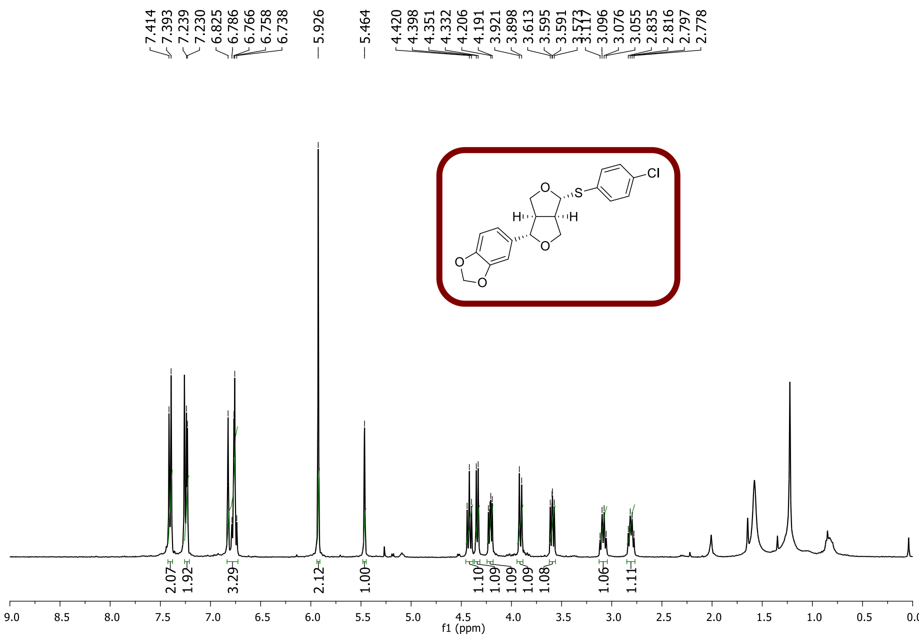


**Figure S26.** $^{1}H$ NMR spectrum of **2α-7h** ($CDCl_3$)

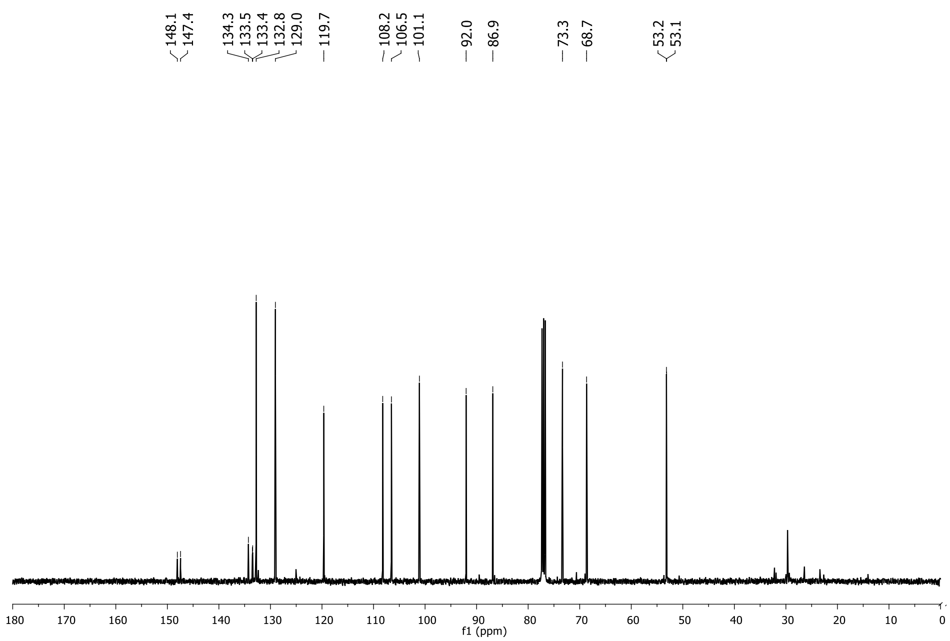


**Figure S27.** $^{13}C$ NMR spectrum of **2α-7h** ($CDCl_3$)

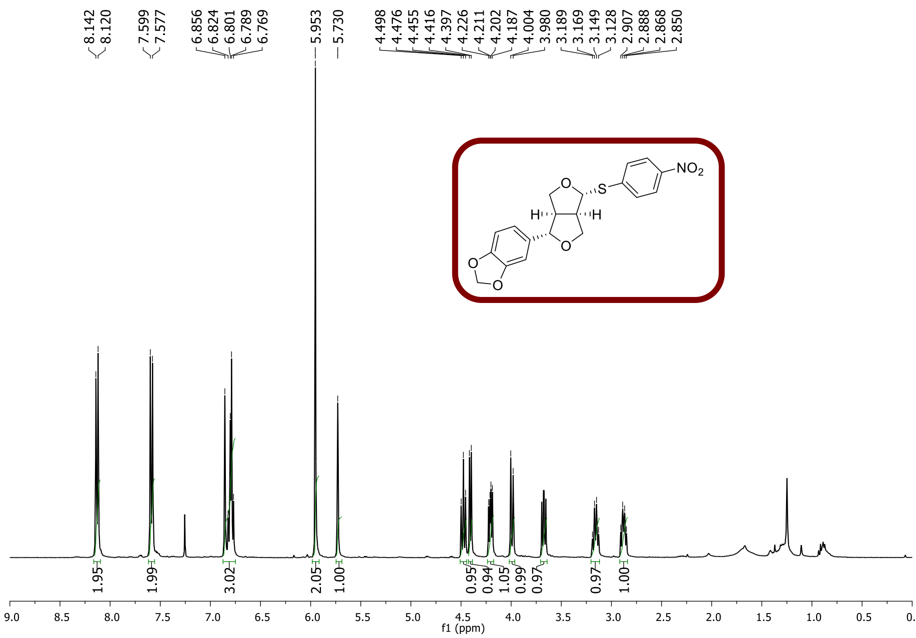



**Figure S28.** $^{1}H$ NMR spectrum of **2α-7i** ($CDCl_3$)

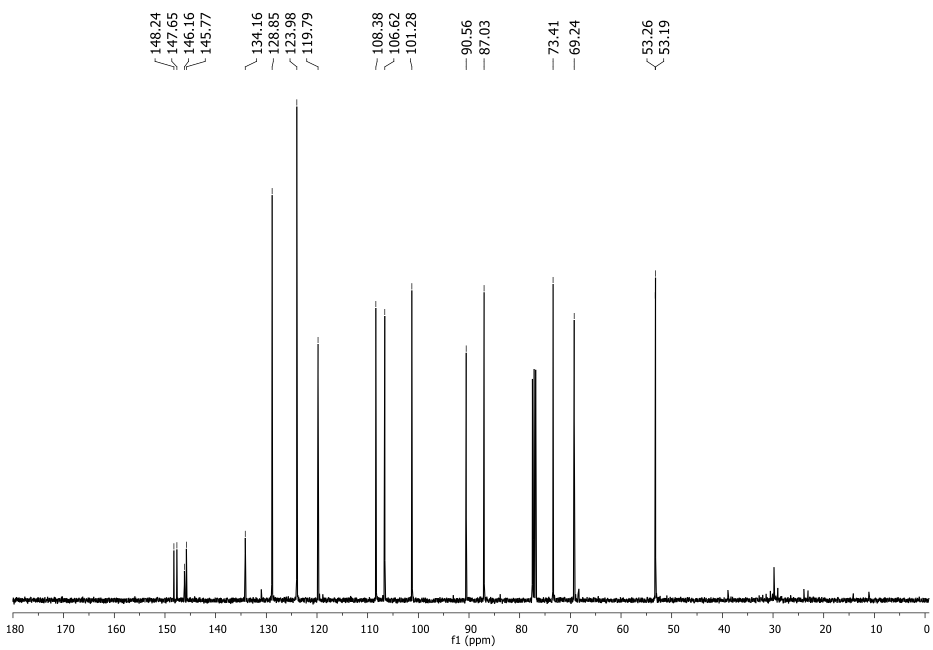



**Figure S29.** $^{13}C$ NMR spectrum of **2α-7i** ($CDCl_3$)

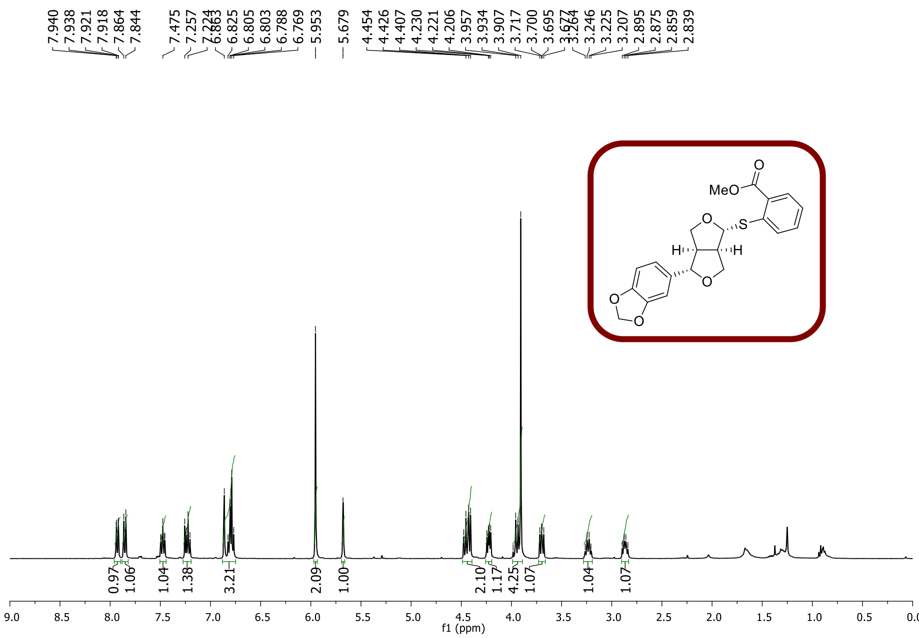


**Figure S30.** $^{1}H$ NMR spectrum of **2α-7j** ($CDCl_3$)

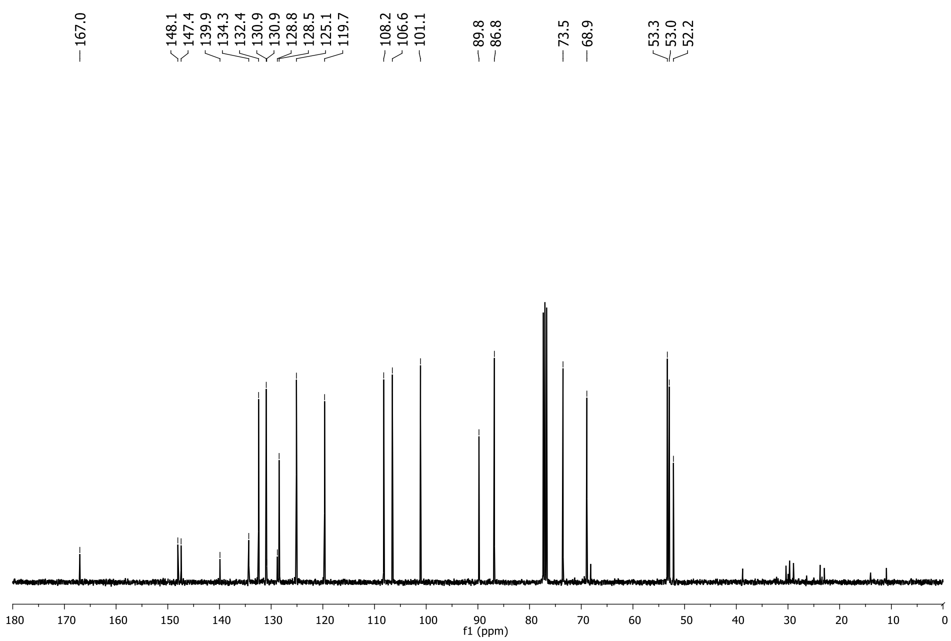


**Figure S31.** $^{13}C$ NMR spectrum of **2α-7j** ($CDCl_3$)

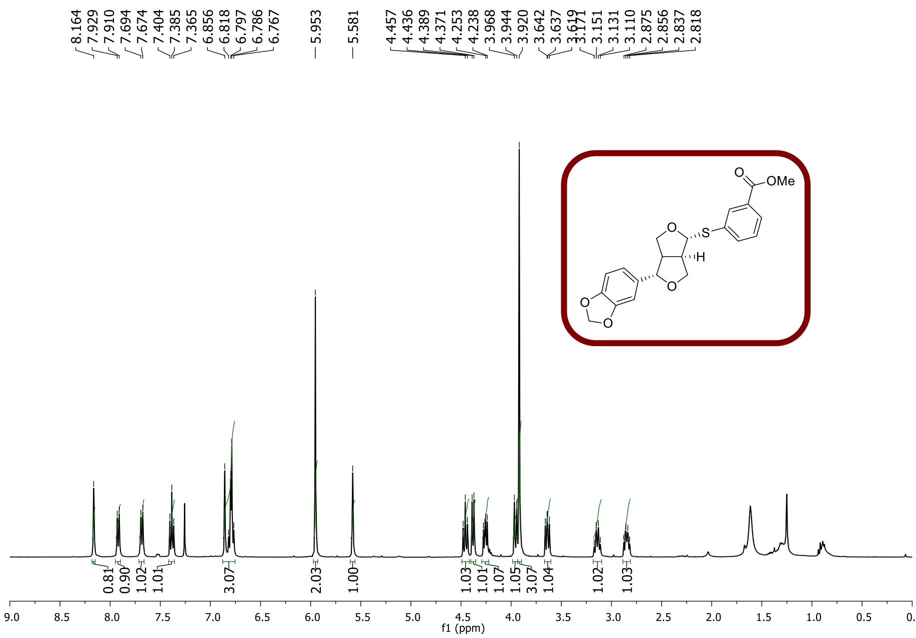


**Figure S32.** $^{1}H$ NMR spectrum of **2α-7k** ($CDCl_3$)

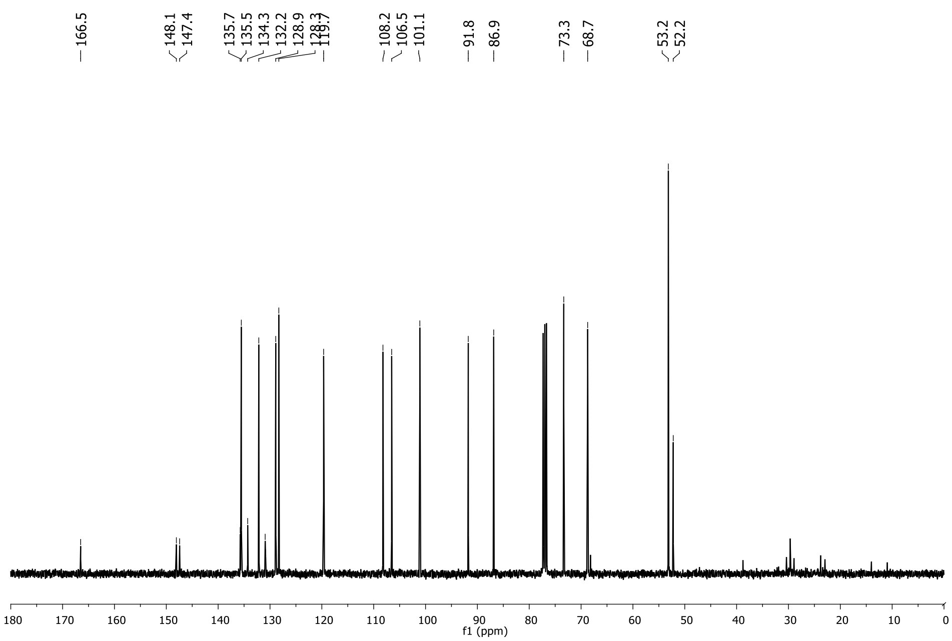


**Figure S33.** $^{13}C$ NMR spectrum of **2α-7k** ($CDCl_3$)

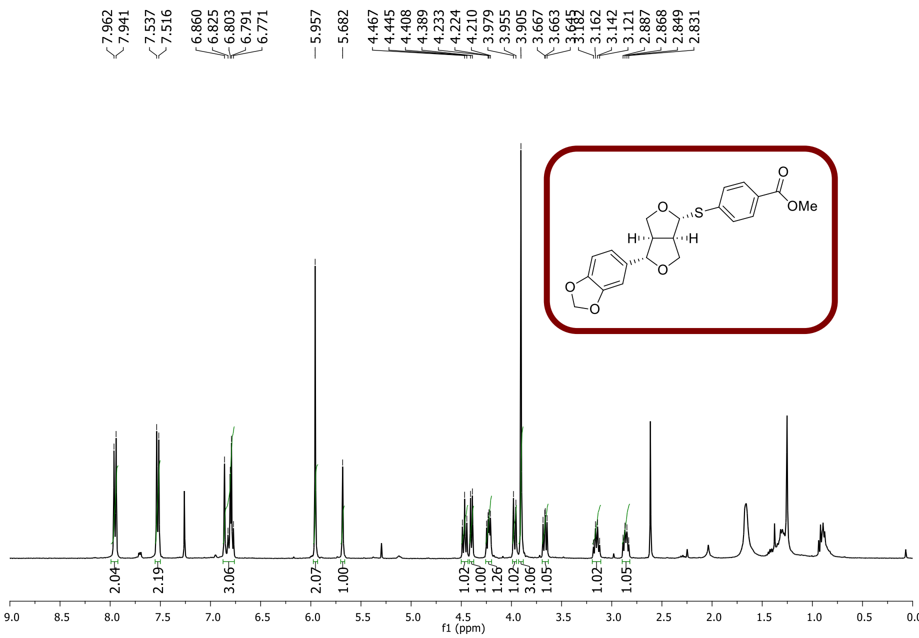


**Figure S34.** $^{1}H$ NMR spectrum of **2α-7l** ($CDCl_3$)

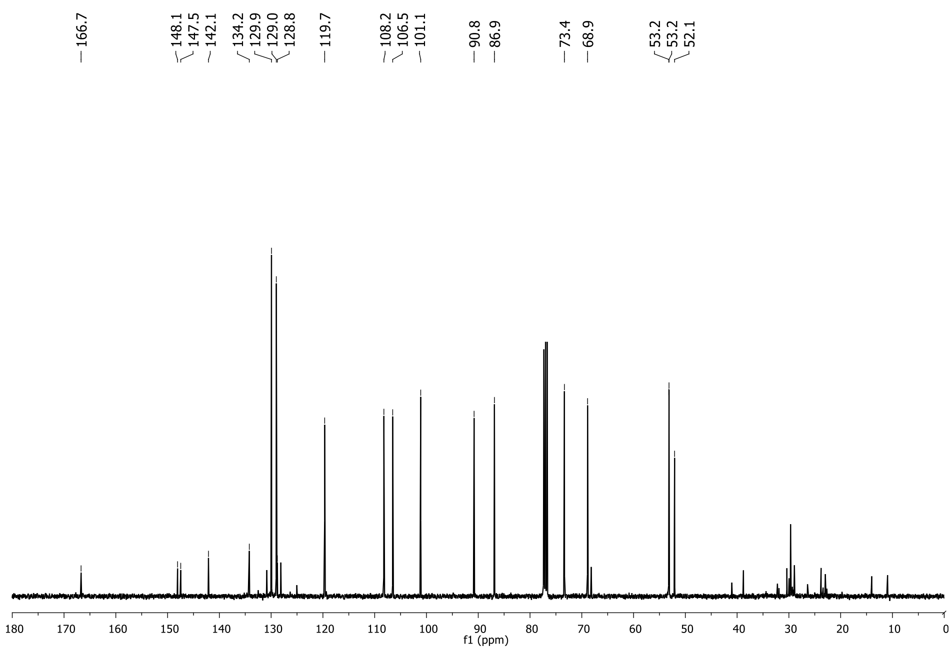


**Figure S35.** $^{13}C$ NMR spectrum of **2α-7l** ($CDCl_3$)

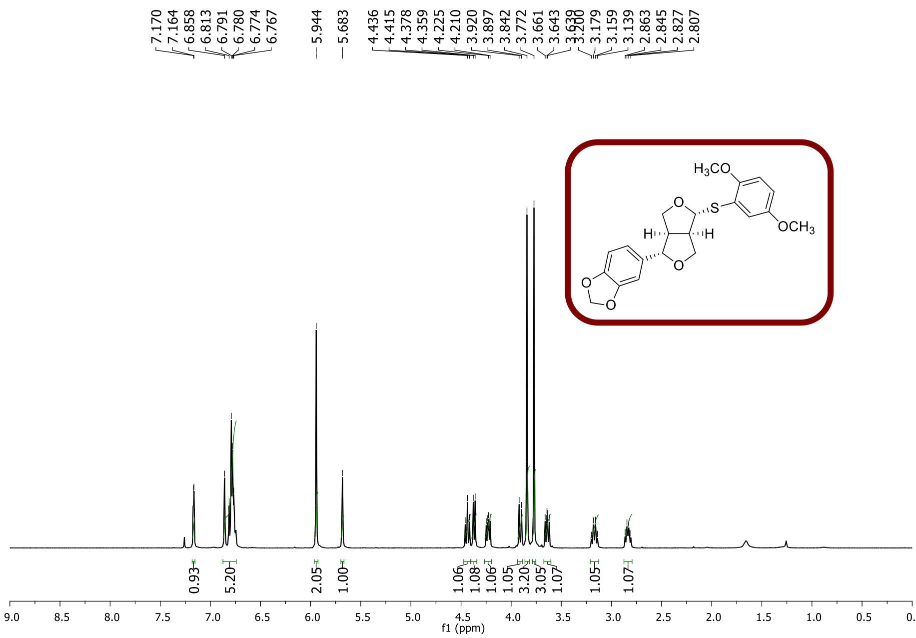



**Figure S36.** $^{1}H$ NMR spectrum of **2α-7m** ($CDCl_3$)

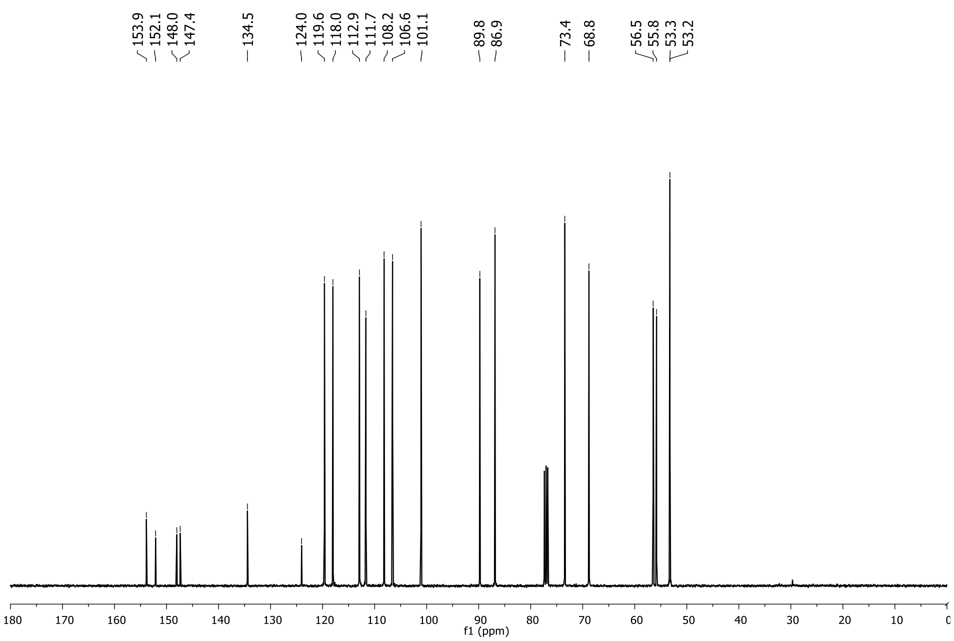



**Figure S37.** $^{13}C$ NMR spectrum of **2α-7m** ($CDCl_3$)

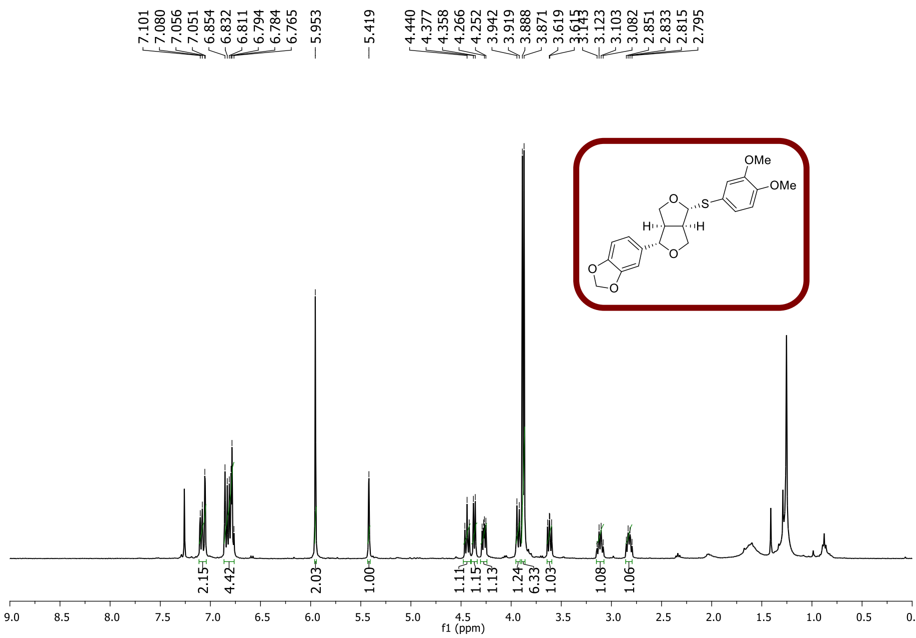



**Figure S38.** $^{1}H$ NMR spectrum of **2α-7n** ($CDCl_3$)

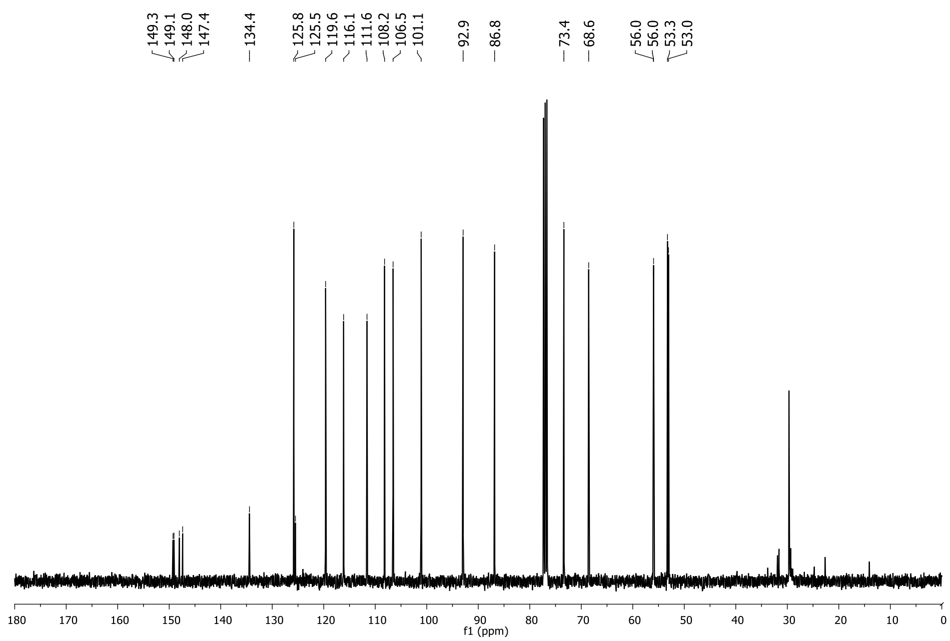



**Figure S39.** $^{13}C$ NMR spectrum of **2α-7n** ($CDCl_3$)

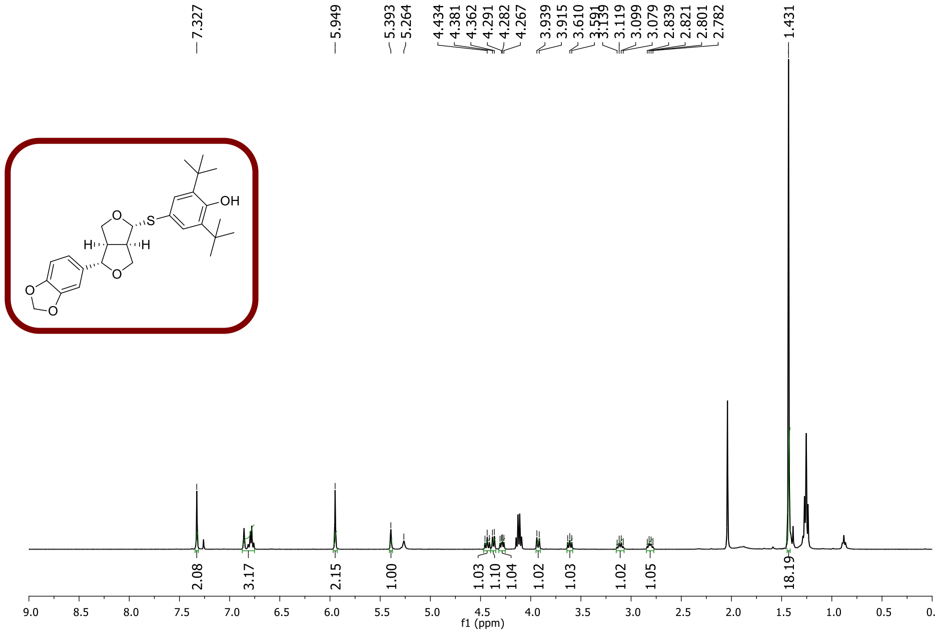



**Figure S40.** $^{1}H$ NMR spectrum of **2α-7o** ($CDCl_3$)

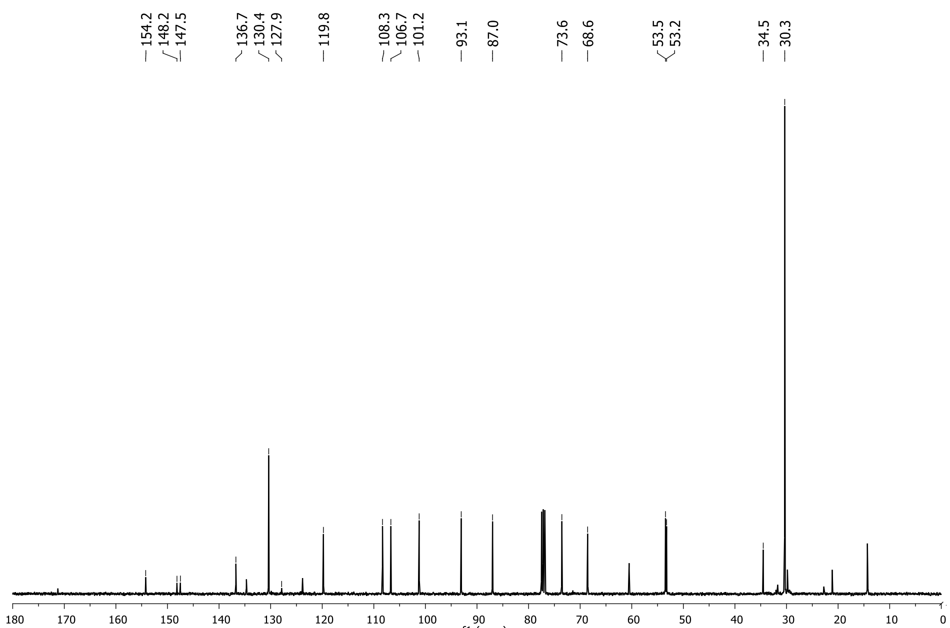



**Figure S41.** $^{13}C$ NMR spectrum of **2α-7o** ($CDCl_3$)

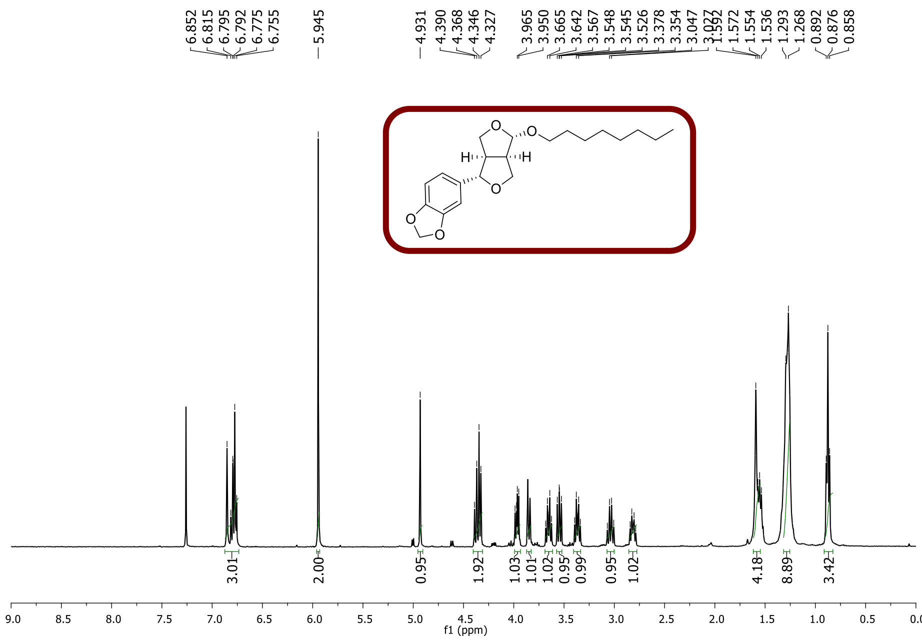


**Figure S42.** $^{1}H$ NMR spectrum of **2α-9b** ($CDCl_3$)

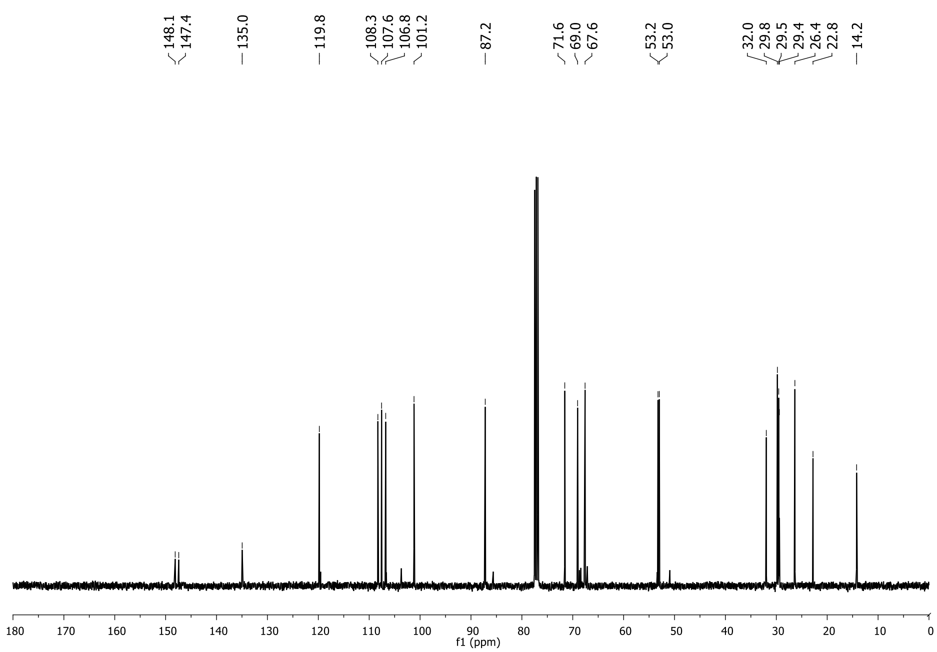


**Figure S43.** $^{13}C$ NMR spectrum of **2α-9b** ($CDCl_3$)

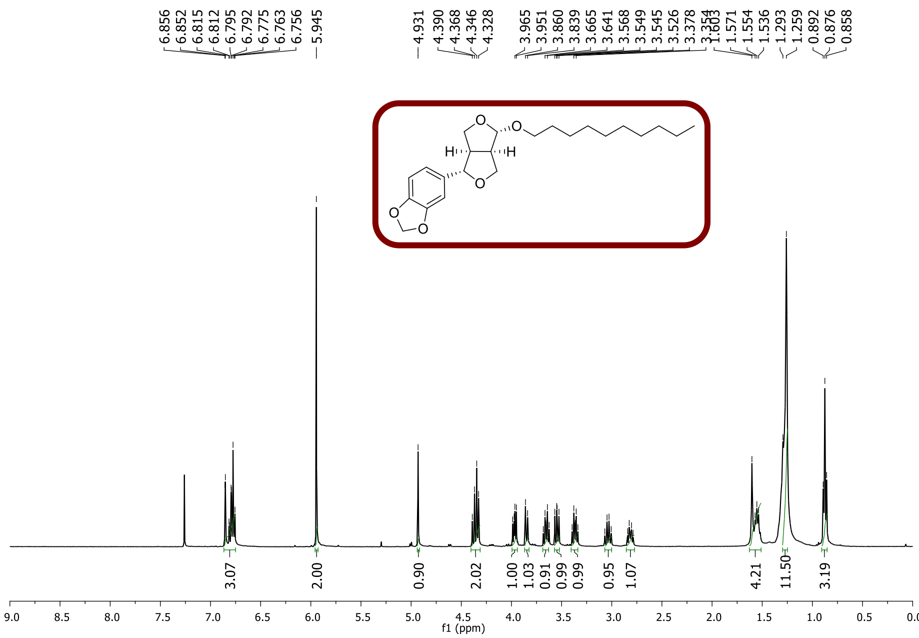


**Figure S44.** $^{1}H$ NMR spectrum of **2α-9c** ($CDCl_3$)

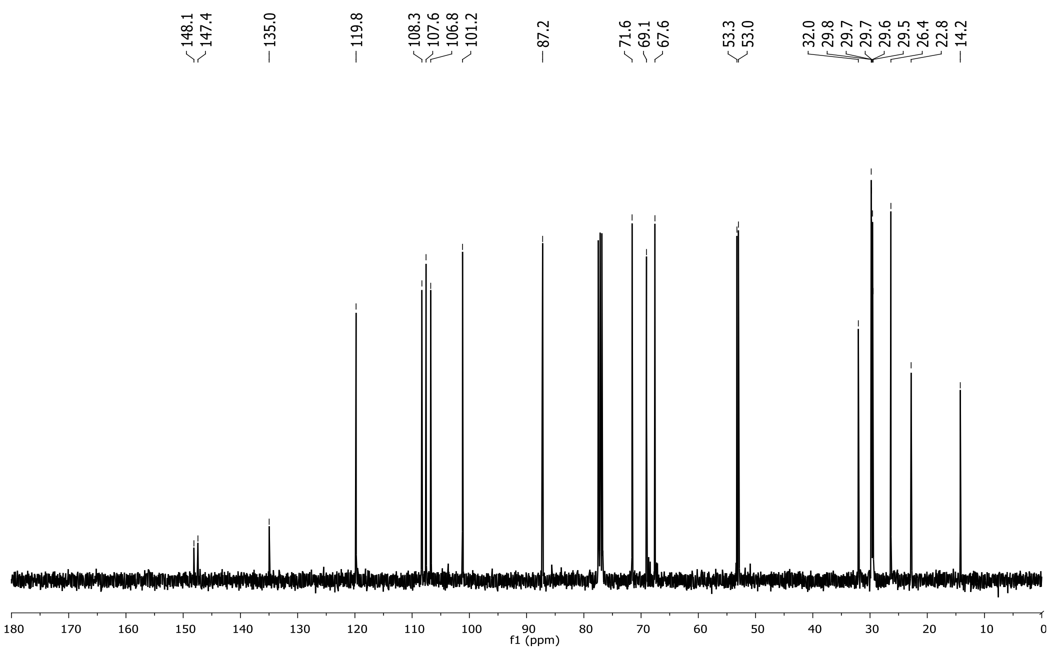


**Figure S45.** $^{13}C$ NMR spectrum of **2α-9c** ($CDCl_3$)

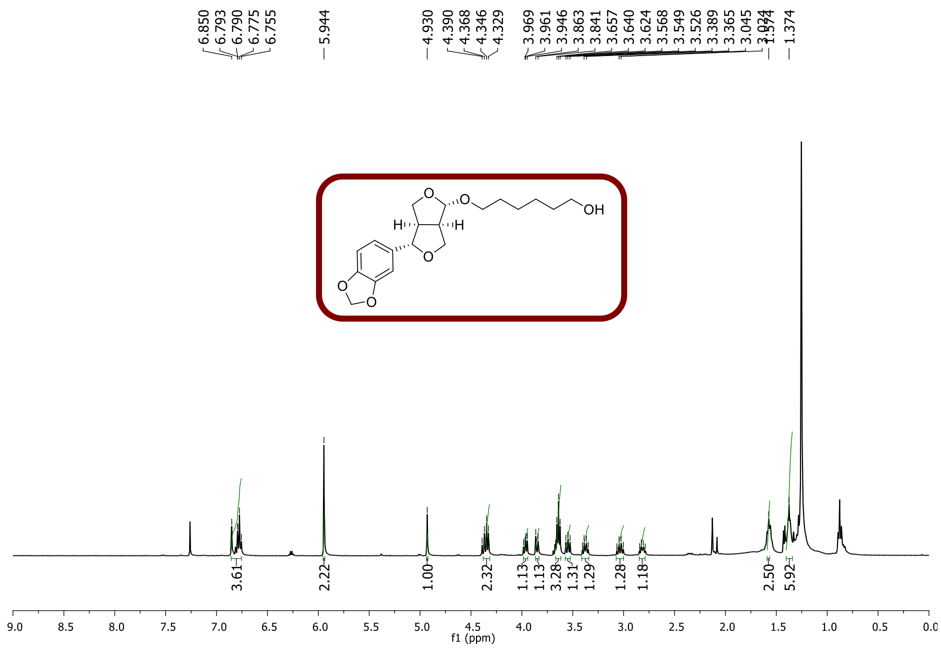



**Figure S46.** $^{1}$H NMR spectrum of **2α-9d** ($CDCl_3$)

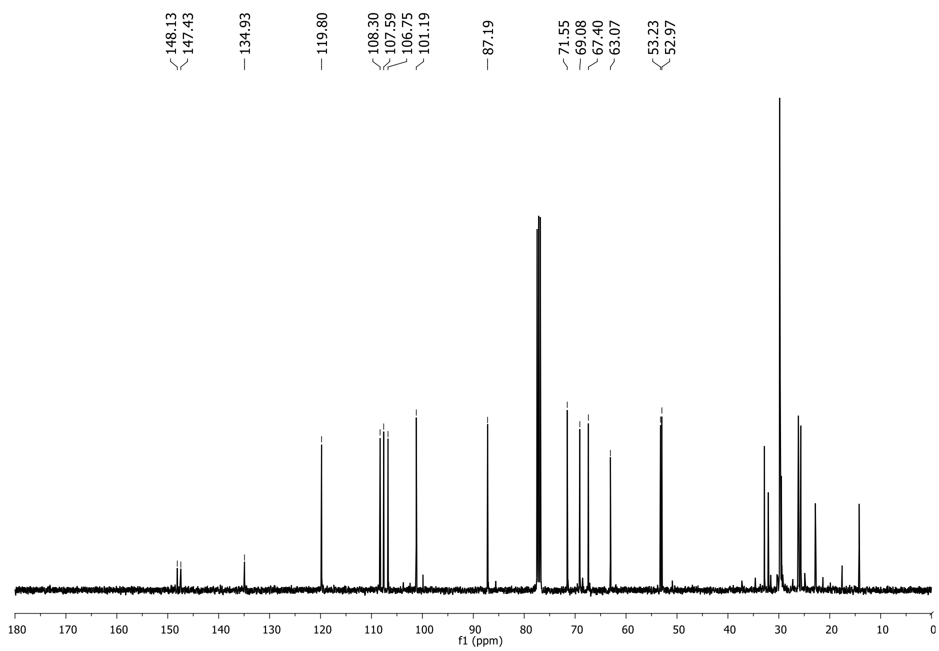



**Figure S47.** $^{13}$C NMR spectrum of **2α-9d** ($CDCl_3$)

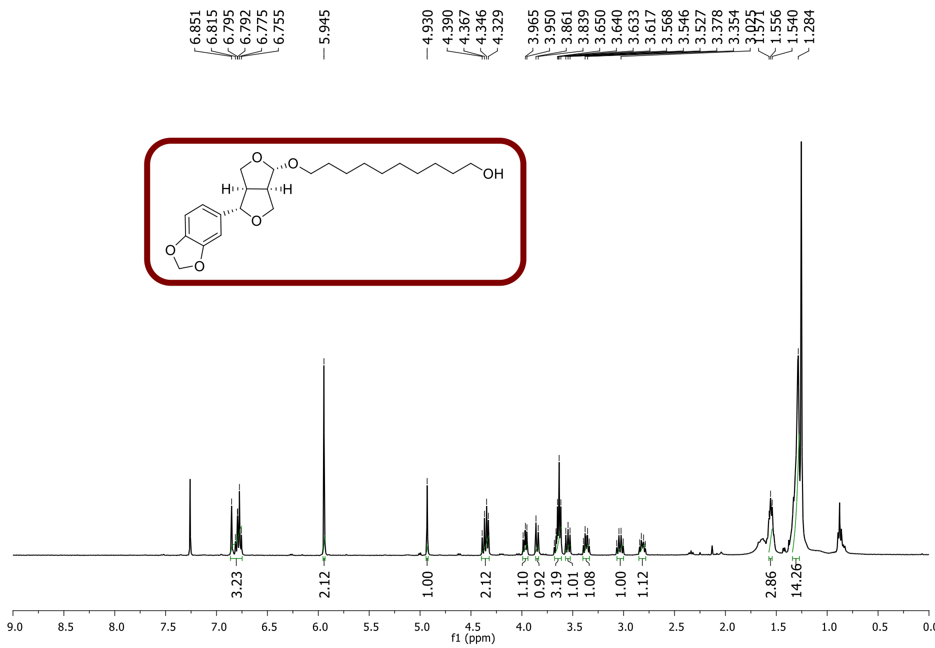



**Figure S48.** $^{1}$H NMR spectrum of **2β-9e** ($CDCl_3$)

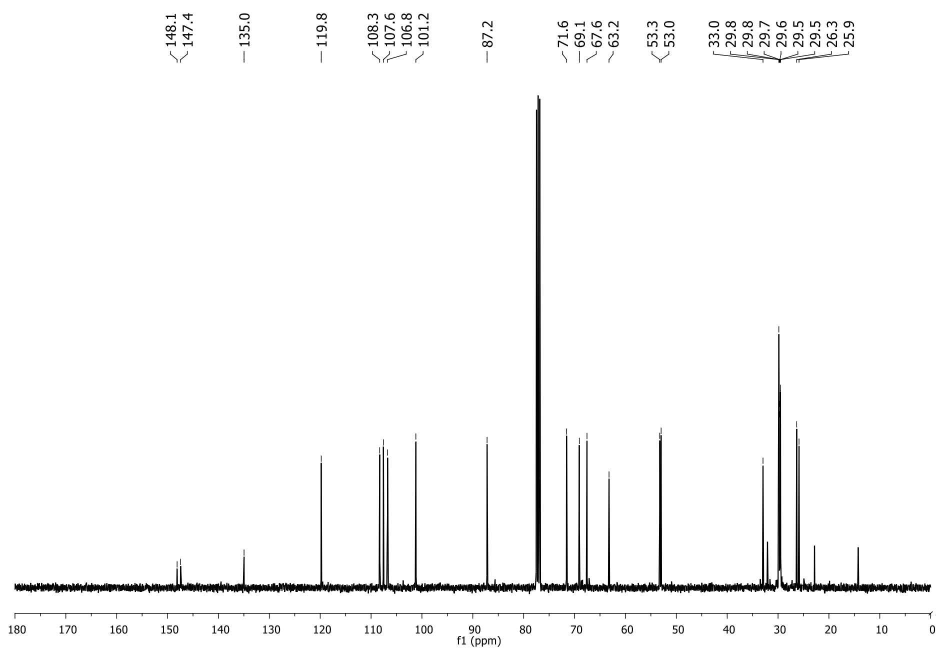



**Figure S49.** $^{13}$C NMR spectrum of **2β-9e** ($CDCl_3$)

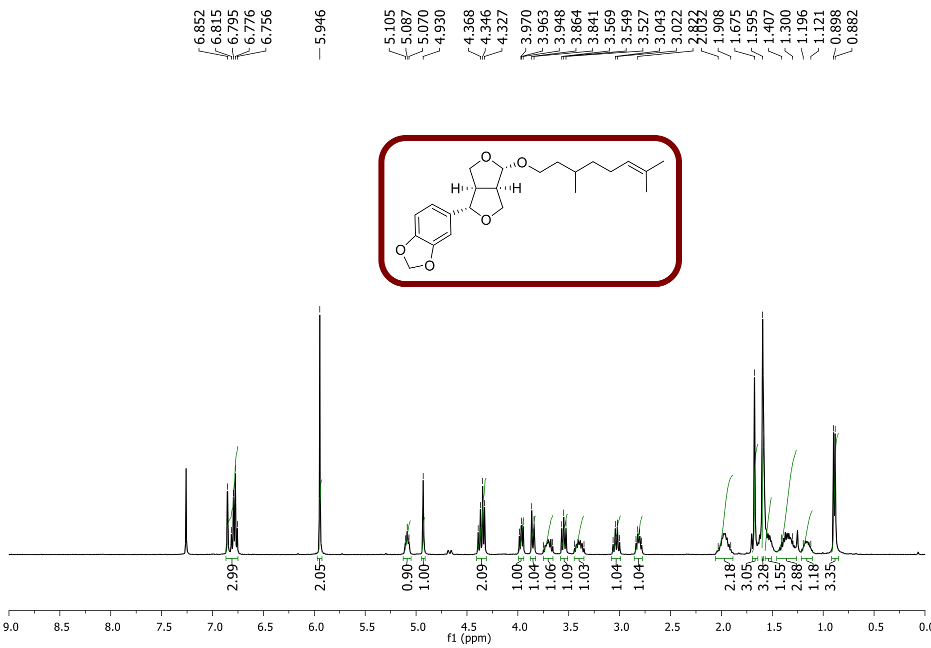


**Figure S50.** $^{1}$H NMR spectrum of **2α-9f** ($CDCl_3$)

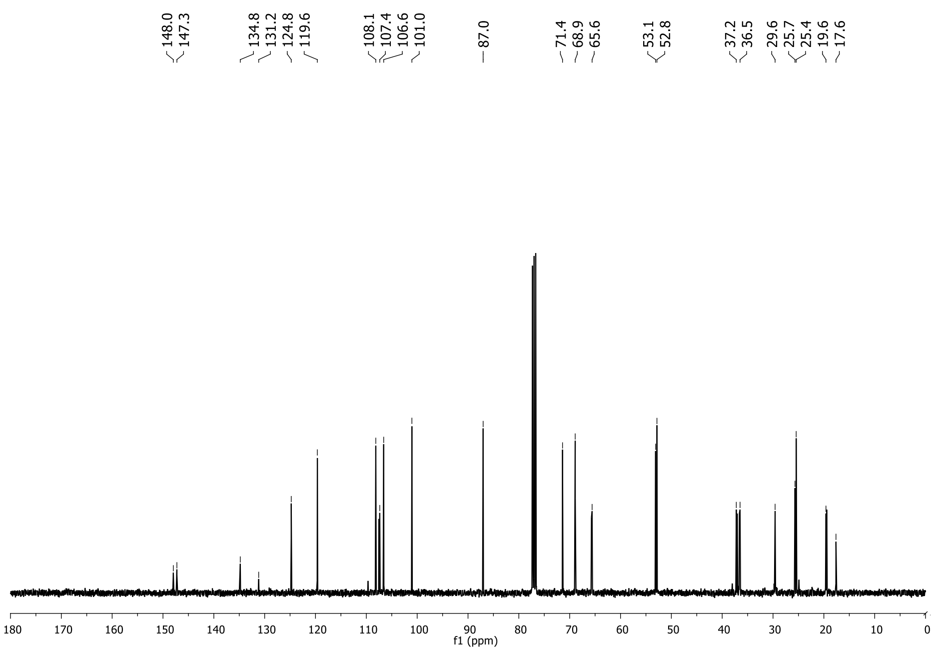


**Figure S51.** $^{13}$C NMR spectrum of **2α-9f** ($CDCl_3$)

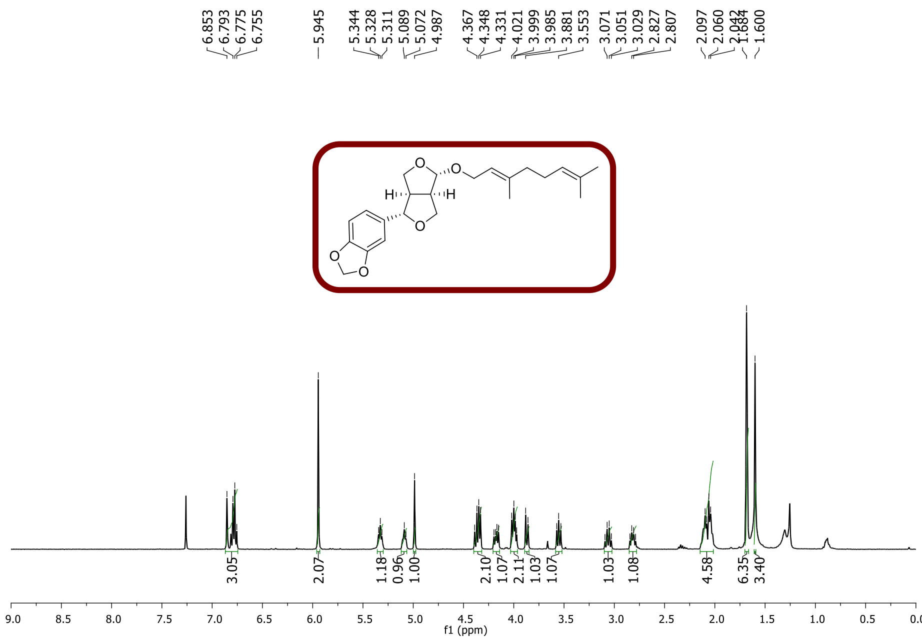


**Figure S52.** $^{1}H$ NMR spectrum of **2α-9g** ($CDCl_3$)

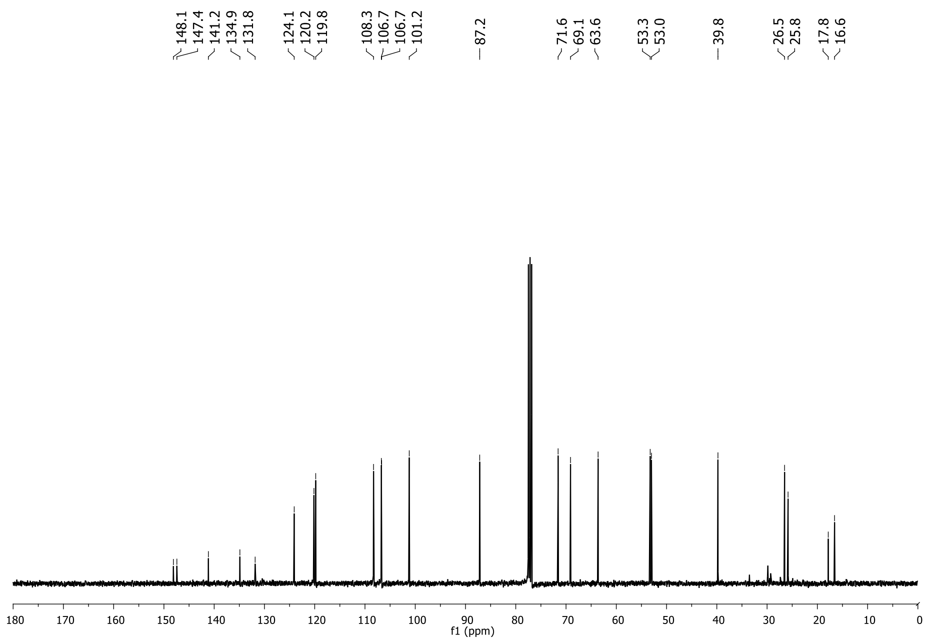


**Figure S53.** $^{13}C$ NMR spectrum of **2α-9g** ($CDCl_3$)

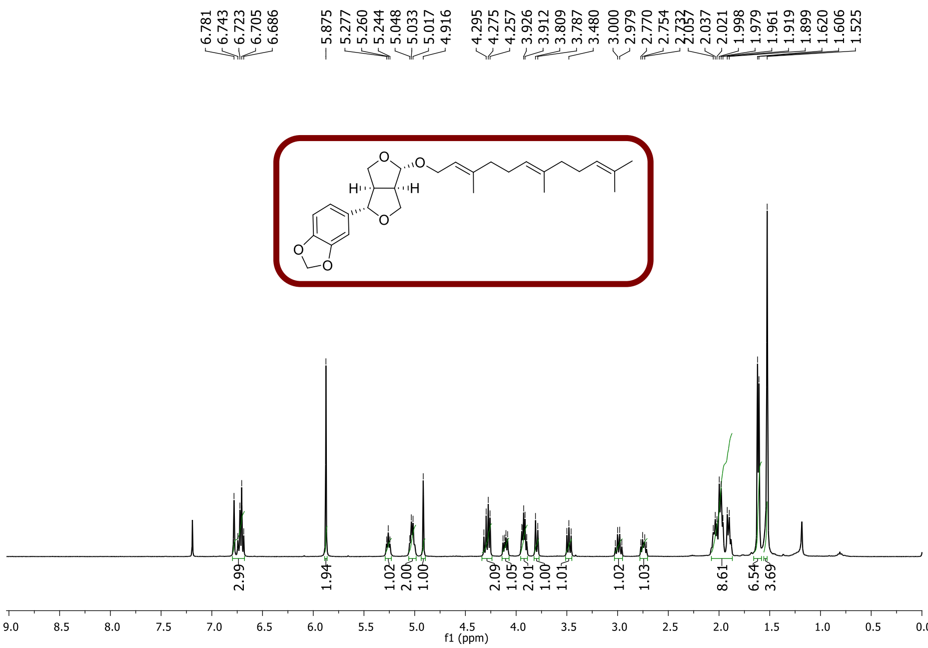


**Figure S54.** $^{1}$H NMR spectrum of **2α-9h** ($CDCl_3$)

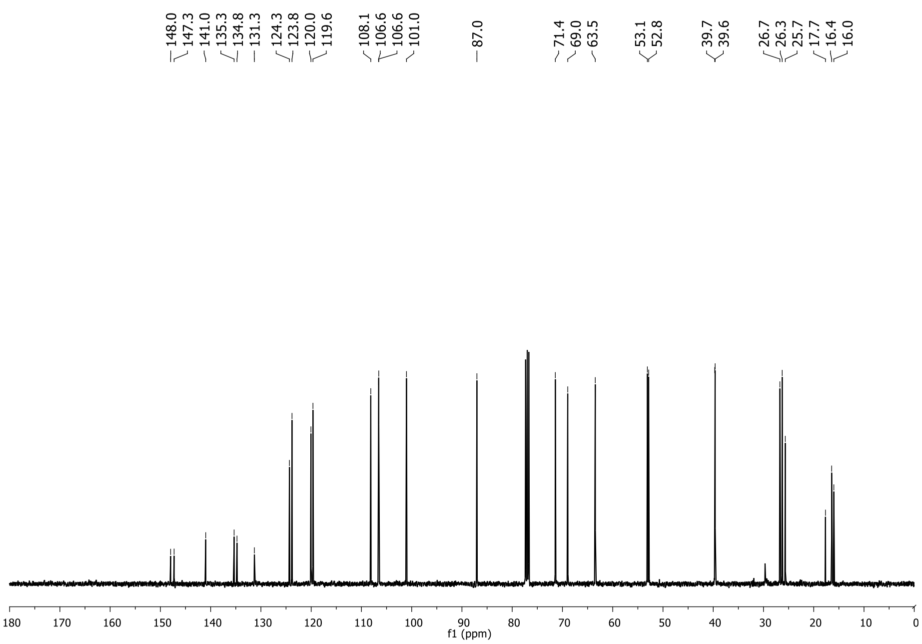


**Figure S55.** $^{13}$C NMR spectrum of **2α-9h** ($CDCl_3$)

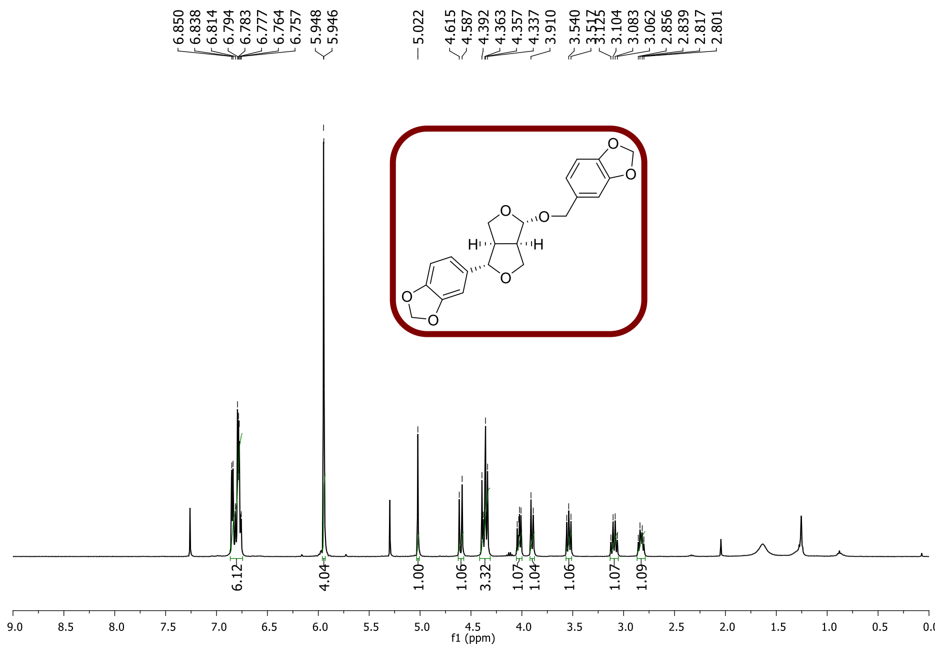


**Figure S56.** $^{1}H$ NMR spectrum of **2α-9i** ($CDCl_3$)

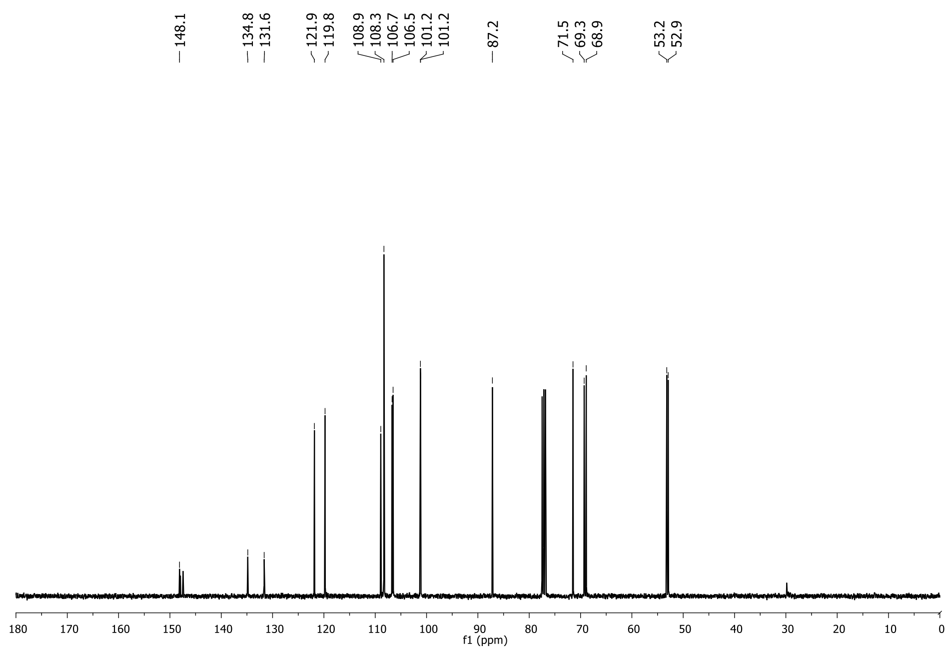


**Figure S57.** $^{13}C$ NMR spectrum of **2α-9i** ($CDCl_3$)

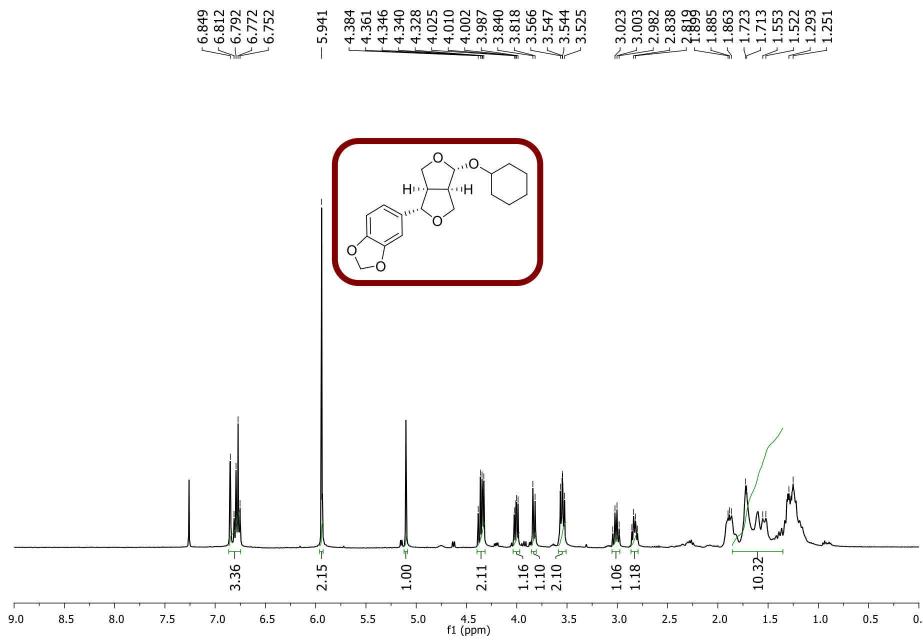



**Figure S58.** $^{1}H$ NMR spectrum of **2α-9j** ($CDCl_3$)

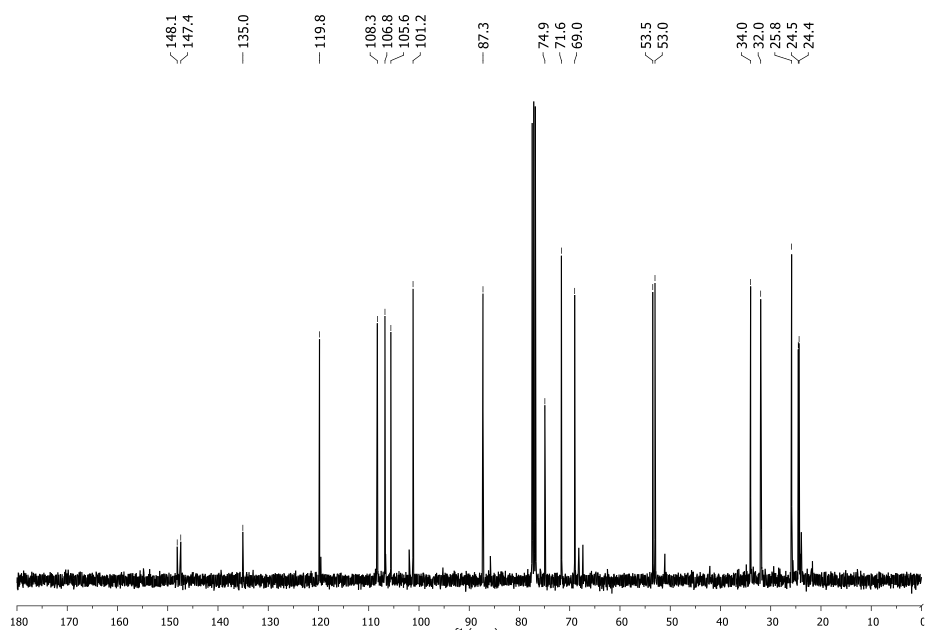



**Figure S59.** $^{13}C$ NMR spectrum of **2α-9j** ($CDCl_3$)

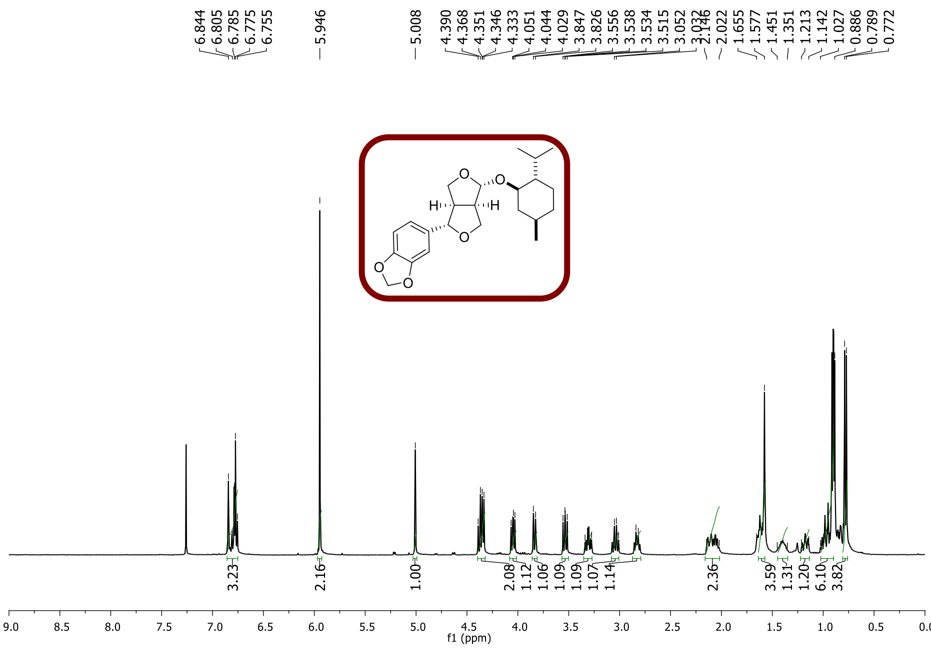


**Figure S60.** $^{1}H$ NMR spectrum of **2α-9k** ($CDCl_3$)

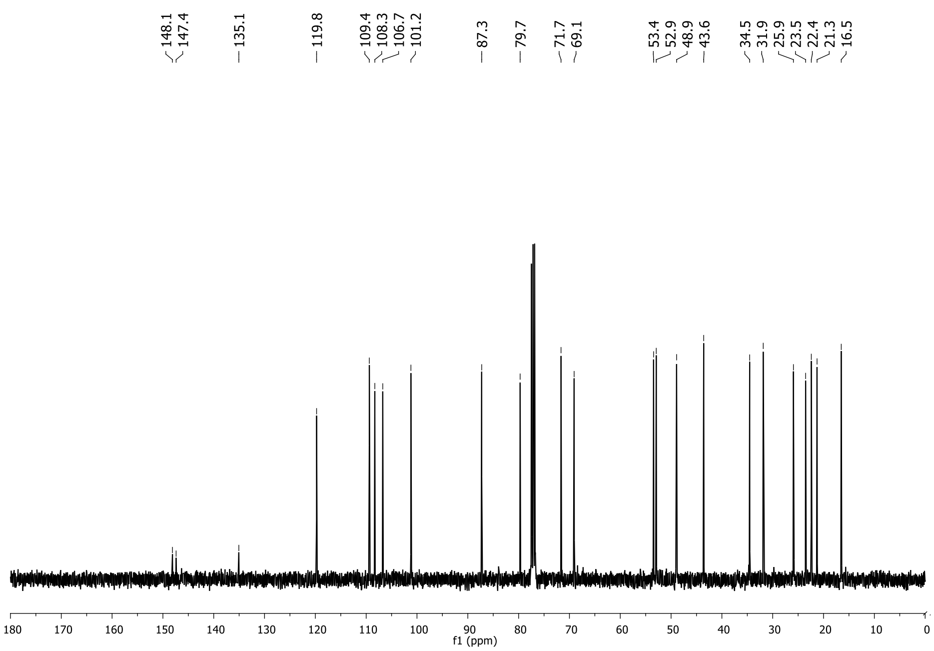


**Figure S61.** $^{13}C$ NMR spectrum of **2α-9k** ($CDCl_3$)

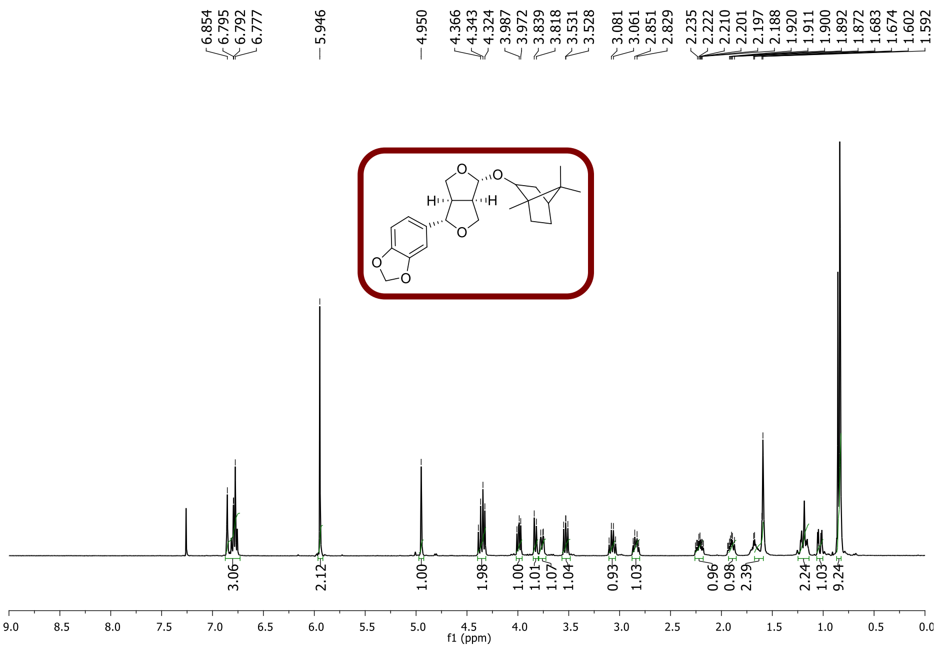


**Figure S62.** $^1$H NMR spectrum of **2α-9l** ($CDCl_3$)

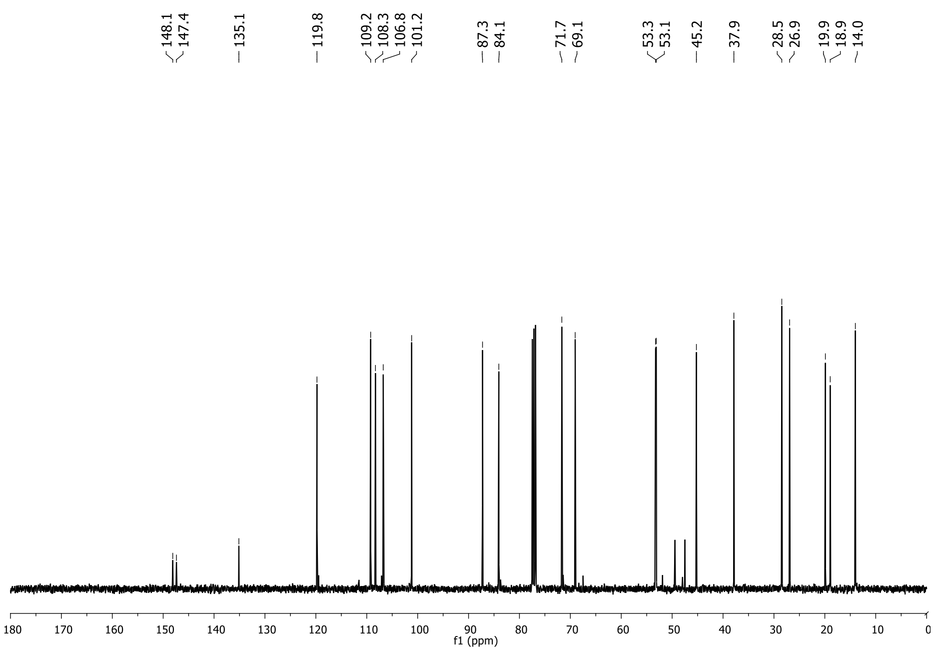


**Figure S63.** $^{13}$C NMR spectrum of **2α-9l** ($CDCl_3$)

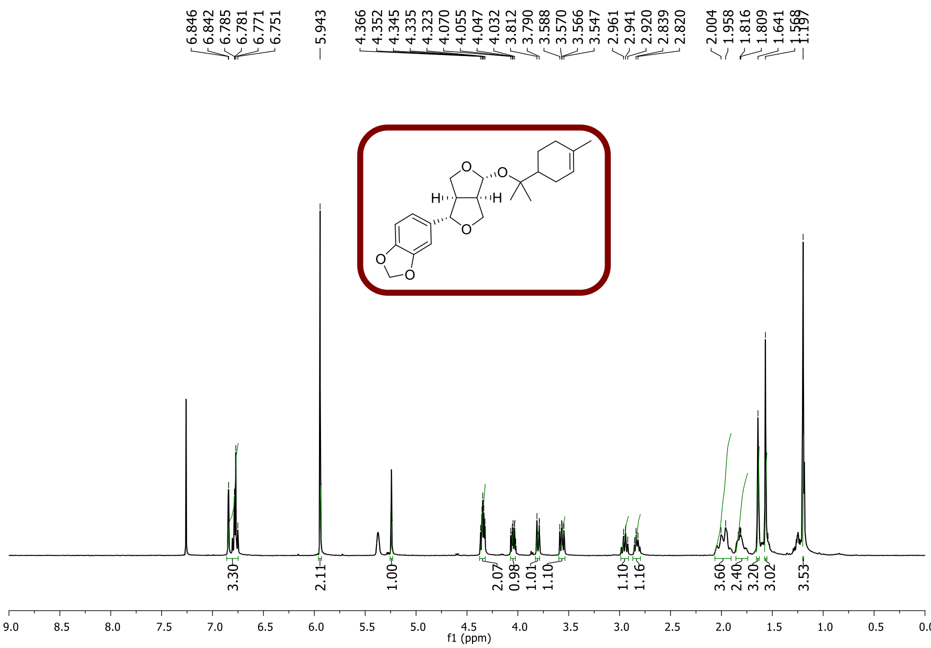



**Figure S64.** $^{1}$H NMR spectrum of **2α-9m** ($CDCl_3$)

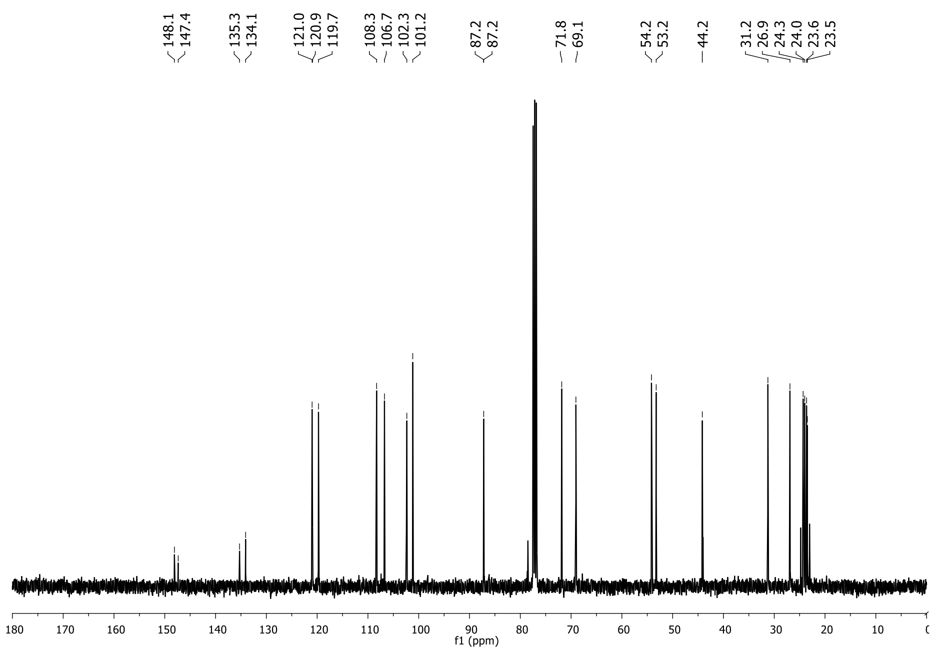



**Figure S65.** $^{13}$C NMR spectrum of **2α-9m** ($CDCl_3$)

**Table S2.** Crystal data and refinement parameters of compounds **2α-7f** and **2β-7fBr**.

| | **2α-7f** | **2β-7fBr** |
|---|---|---|
| Crystal habit | Thin plate, colorless | Thin plate, colorless |
| Crystal size ($mm^3$) | 0.01×0.24×0.34 | 0.01×0.18×0.20 |
| Empirical formula | $C_{19}H_{18}O_5S$ | $C_{26}H_{23}BrO_5S$ |
| *FW* | 358.40 | 527.41 |
| Crystal system | Monoclinic | Triclinic |
| Space group | $P2_1$ (no. 4) | $P1$ (no. 1) |
| $a$ [Å] | 6.1485(2) | 6.7501(13) |
| $b$ [Å] | 7.6475(2) | 8.8663(18) |
| $c$ [Å] | 17.9288(4) | 20.864(4) |
| $\alpha$ [º] | 90 | 95.858(5) |
| $\beta$ [º] | 90.711(1) | 94.023(5) |
| $\gamma$ [º] | 90 | 111.089(5) |
| $V$ [Å$^3$] | 842.96(4) | 1151.3(4) |
| $Z$ | 2 | 2 |
| $\rho_{calcd}$ [Mg m$^{-3}$] | 1.412 | 1.521 |
| $\mu$ [mm$^{-1}$] | 1.948 | 1.912 |
| $F(000)$ | 376 | 540 |
| Diffractometer | Bruker X8 Prospector KAPPA CCD | Bruker X8 APEX II KAPPA CCD |
| Radiation (λ, Å) | CuK$\alpha$ (1.54178) | MoK$\alpha$ (0.71073) |
| Resolution (Å) | 0.83 | 0.83 |
| Temperature (K) | 296(2) | 296(2) |
| $2\theta_{max}$ | 136.8 | 50.6 |
| Completeness (%) | 99.6 | 99.7 |
| Reflns collected/ unique/ > $2\sigma(I)$ | 6974 / 3010 / 2417 | 20477 / 8319 / 3229 |
| $R_{int}$ | 0.0522 | 0.0943 |
| Data / parameters | 3010 / 227 | 8319 / 595 |
| Goodness on fit | 1.100 | 0.921 |
| $R_1$,[a] $wR2$[b] $[I > 2\sigma(I)]$ | 0.0558, 0.1403 | 0.0584, 0.0827 |
| $R_1$, $wR_2$ [all data] | 0.0672, 0.1535 | 0.2047, 0.1200 |
| Flack parameter ($x$) | 0.040(19) | 0.026(12) |
| Δρ (e Å$^{-3}$) | -0.39, 0.21 | -0.28, 0.28 |

[a] $R = \sum ||F_o| - |F_c|| / \sum |F_o|$; [b] $wR = \sum \{w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2\}^{1/2}$.