# Chlorosulfonic acid supported diethylamine ionic liquid catalyzed green synthesis of novel 2-mercaptonaphthalen-1-yl)methyl)-3-hydroxy-5,5-dimethylcyclohex-2-enones under neat conditions

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**2-((2-bromophenyl)(2-mercaptonaphthalen-1-yl)methyl)-3-hydroxy-5,5-dimethylcyclohex-2-enone (4a)**. Yield 90%; white solid; mp 180-182 °C. IR (KBr): ν= 3431, 3050, 2615, 1706, 1575 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 10.21 (s, 1H), 7.88 (s, 1H), 7.79-7.73 (m, 3H), 7.60 (d, *J* = 8.0Hz, 1H), 7.57-7.55 (m, 1H), 7.47-7.43 (m, 2H), 7.42 (d, *J* = 7.3 Hz, 1H), 7.27-7.24 (m, 1H), 7.14-7.11 (m, 1H), 6.40 (s, 1H), 2.44 (d, *J* = 16.1 Hz, 1H), 2.27 (d, *J* = 16.2 Hz, 1H), 2.14 (d, *J* = 16.2 Hz, 1H), 2.06 (d, *J* = 16.2 Hz, 1H), 0.96 (s, 3H), 0.72 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.6, 173.8, 136.9, 133.7, 133.5, 132.5, 130.2, 129.6, 1294.4, 128.9, 127.8, 127.6, 127.4, 126.6, 126.4, 125.1, 110.9, 50.1, 47.9, 43.5, 31.6, 28.1, 27.6 ppm. HRMS (ESI, m/z): calcd for $C_{25}H_{23}BrO_2S$ ($M+H^+$) 467.4179; found: 467.3956.

**2-((2-fluorophenyl)(2-mercaptonaphthalen-1-yl)methyl)-3-hydroxy-5,5-dimethylcyclohex-2-enone (4b).** Yield 89%; white solid; mp 157-159 °C. IR (KBr): ν= 3401, 3045, 2619, 1725, 1570 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 9.89 (s, 1H), 7.86 (s, 1H), 7.79-7.73 (m, 3H), 7.52-7.47 (m, 3H), 7.44-7.38 (m, 1H), 7.30-7.25 (m, 1H), 7.12-7.07 (m, 2H), 6.40 (s, 1H), 2.42 (d, *J* = 16.1 Hz, 1H), 2.22 (d, *J* = 16.2 Hz, 1H), 2.14 (d, *J* = 16.2 Hz, 1H), 2.04 (d, *J* = 16.2 Hz, 1H), 0.95 (s, 3H), 0.68 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.6, 173.8, 160.0 (*J* = 204.1), 133.4, 132.4, 130.2, 129.7, 129.6, 129.2, 128.8, 127.7, 127.6, 127.4, 126.6, 125.3, 124.3, 116.0, 115.8, 110.5, 50.1, 42.9, 40.6, 31.8, 28.8, 27.8 ppm. HRMS (ESI, m/z): calcd for $C_{25}H_{23}FO_2S$ ($M+H^+$) 406.1402; found: 406.1429.

**3-hydroxy-2-((2-mercaptonaphthalen-1-yl)(o-tolyl)methyl)-5,5-dimethylcyclohex-2-enone (4c).** Yield 90%; white solid; mp 116-118 °C. IR (KBr): ν= 3395, 2985, 2698, 1698, 1574 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 10.0 (s, 1H), 7.82 (s, 1H), 7.79-7.73 (m, 3H), 7.49-7.43 (m, 3H), 7.32 (d, *J* = 8.1 Hz, 1H), 7.23-7.16 (m, 3H), 6.27 (s, 1H), 2.69 (s, 3H), 2.45 (d, *J* = 16.1 Hz, 1H), 2.23 (d, *J* = 16.2 Hz, 1H), 2.15 (d, *J* = 16.1 Hz, 1H), 2.06 (d, *J* = 16.2 Hz, 1H), 0.97 (s, 3H), 0.70 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.8, 173.6, 137.1, 133.5, 131.1, 130.7, 129.0, 128.8, 128.1, 127.6, 127.4, 127.2, 127.1, 126.6, 126.5, 126.3, 111.4, 50.3, 44.1, 43.5, 31.7, 28.2, 27.5, 19.2 ppm. HRMS (ESI, m/z): calcd for $C_{26}H_{26}O_2S$ ($M+H^+$) 402.1653; found: 402.1712.

**3-hydroxy-2-((2-mercaptonaphthalen-1-yl)(2-methoxyphenyl)methyl)-5,5-dimethylcyclohex-2-enone (4d).** Yield 91%; white solid; mp 128-130 °C. IR (KBr): ν= 3439, 3058, 2615, 1706, 1565 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 9.90 (s, 1H), 7.81 (s, 1H), 7.79-7.73 (m, 3H), 7.49-7.43 (m, 4H), 7.32 (d, *J* = 8.1 Hz, 1H), 7.23-7.16 (m, 2H), 6.44 (s, 1H), 3.95 (s, 3H), 2.45 (d, *J* = 16.1 Hz, 1H), 2.23 (d, *J* = 16.2 Hz, 1H), 2.15 (d, *J* = 16.1 Hz, 1H), 2.06 (d, *J* = 16.2 Hz, 1H), 0.95 (s, 3H), 0.69 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.7, 173.1, 156.8, 133.4, 132.9, 131.5, 129.2, 129.1, 128.8, 128.5, 127.6, 127.5, 127.3, 126.4, 126.0, 120.9, 111.4, 111.2, 55.8, 50.4, 43.5, 41.8, 31.5, 28.2, 27.4 ppm. HRMS (ESI, m/z): calcd for $C_{26}H_{26}O_3S$ ($M+H^+$) 418.1602; found: 418.1702.

**2-((3-bromophenyl)(2-mercaptonaphthalen-1-yl)methyl)-3-hydroxy-5,5-dimethylcyclohex-2-enone (4e).**Yield 88%; white solid; mp 145-147 °C. IR (KBr): ν= 3450, 3050, 2600, 1715, 1590 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 9.54 (s, 1H), 7.83 (s, 1H), 7.79-7.77 (m, 3H),7.75-7.71 (m, 1H), 7.60 (d, *J* = 7.6 Hz, 1H), 7.48-7.45 (m, 2H), 7.41-7.38 (m, 2H), 7.25-7.18 (q, 1H), 6.20 (s, 1H), 2.42 (d, *J* = 16.1 Hz, 1H), 2.16 (d, *J* = 16.2 Hz, 2H), 2.01 (d, *J* = 16.2 Hz, 1H), 0.95 (s, 3H), 0.59 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.8, 173.7, 140.1, 133.4, 132.5, 131.0, 130.9, 130.2, 129.9, 129.7, 128.9, 128.4, 127.4, 126.7, 126.6, 126.5, 122.7, 111.0, 50.1, 45.7, 43.3, 31.6, 28.2, 27.3 ppm. HRMS (ESI, m/z): calcd for $C_{25}H_{23}BrO_2S$ ($M+H^+$) 467.4179; found: 467.4110.

**2-((3-fluorophenyl)(2-mercaptonaphthalen-1-yl)methyl)-3-hydroxy-5,5-dimethylcyclohex-2-enone (4f).** Yield 90%; white solid; mp 151-153 °C. IR (KBr): ν= 3420, 3050, 2619, 1704, 1570 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 9.56 (s, 1H), 7.82 (s, 1H), 7.78-7.71 (m, 3H), 7.47-7.43 (m, 3H), 7.29-7.25 (m, 2H), 7.18 (d, *J* = 8.0 Hz, 1H), 6.98-6.94 (m, 1H), 6.24 (s, 1H), 2.40 (d, *J* = 16.1 Hz, 1H), 2.16 (d, *J* = 16.2 Hz, 2H), 2.02 (d, *J* = 16.2 Hz, 1H), 0.95 (s, 3H), 0.60 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.9, 173.7, 162.9 (d, *J* = 245.4), 140.4, 133.4, 132.4, 130.3, 130.2, 130.1, 129.6, 128.8, 127.6, 127.4, 126.6, 126.4, 123.7, 115.1, 114.8, 114.6, 111.3, 50.1, 45.7, 43.3, 31.6, 28.2, 27.3 ppm. HRMS (ESI, m/z): calcd $C_{25}H_{23}FO_2S$ ($M+H^+$) 406.1402; found: 406.1430.

**3-hydroxy-2-((2-mercaptonaphthalen-1-yl)(3-nitrophenyl)methyl)-5,5-dimethylcyclohex-2-enone (4g).** Yield 89%; white solid; mp 167-169 °C. IR (KBr): ν= 33801, 2990, 2665, 1740, 1570 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 9.68 (s, 1H), 8.33 (s, 1H), 8.14 (dd, *J* = 2.2, 8.2 Hz, 1H), 7.85 (s, 1H), 7.85-7.72 (m, 4H), 7.51-7.47 (m, 4H), 6.26 (s, 1H), 2.40 (d, *J* = 16.1 Hz, 1H), 2.23 (d, *J* = 16.2 Hz, 2H), 2.04 (d, *J* = 16.2 Hz, 1H), 0.97 (s, 3H), 0.66 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.8, 173.6, 140.2, 133.5, 132.5, 130.3, 129.9, 129.8, 128.9, 127.6, 127.4, 126.7, 126.5, 123.7, 114.9, 114.7, 111.2, 50.1, 45.7, 43.3, 31.6, 28.3, 27.3 ppm. HRMS (ESI, m/z): calcd for $C_{25}H_{23}NO_4S$ ($M+H^+$) 433.1347; found: 433.1295.

**3-hydroxy-2-((2-mercaptonaphthalen-1-yl)(m-tolyl)methyl)-5,5-dimethylcyclohex-2-enone (4h).** Yield 94%; white solid; mp 138-140 °C. IR (KBr): ν= 3380, 3078, 2610, 1709, 1545 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 9.69 (s, 1H), 7.80 (s, 1H), 7.77-7.72 (m, 3H), 7.46 (d, *J* = 7.2 Hz, 3H), 7.31-7.29 (m, 2H), 7.21-7.18 (m, 1H), 7.05 (d, *J* = 7.7 Hz, 1H), 6.19 (s, 1H), 2.39 (d, *J* = 16.1 Hz, 1H), 2.33 (s, 3H), 2.25 (d, *J* = 16.2 Hz, 1H), 2.17 (d, *J* = 16.2 Hz, 1H), 2.02 (d, *J* = 16.2 Hz, 1H), 0.97 (s, 3H), 0.68 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.8, 173.6, 137.0, 135.6, 133.5, 132.4, 131.1, 130.7, 128.9, 128.8, 127.6, 127.3, 127.2, 127.1, 126.6, 126.4, 126.3,

111.3, 50.3, 44.1, 43.5, 31.6, 28.1, 27.5, 19.2 ppm. HRMS (ESI, m/z): calcd for $C_{26}H_{26}O_2S$ ($M+H^+$) 402.1653; found: 402.1625.

**2-((4-bromophenyl)(2-mercaptonaphthalen-1-yl)methyl)-3-hydroxy-5,5-dimethylcyclohex-2-enone (4i).**Yield 92%; white solid; mp 189-191 °C. IR (KBr): ν= 3421, 3050, 2615, 1715, 1589 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 9.48 (s, 1H), 7.83 (s, 1H), 7.79-7.71 (m, 3H), 7.50-7.44 (m, 5H), 7.33 (d, *J* = 8.2 Hz, 2H), 6.20 (s, 1H), 2.39 (d, *J* = 16.1 Hz, 1H), 2.17 (d, *J* = 16.2 Hz, 1H), 2.12 (d, *J* = 16.2 Hz, 1H), 2.01 (d, *J* = 16.2 Hz, 1H), 0.94 (s, 3H), 0.60 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.8, 173.6, 136.8, 133.5, 132.5, 131.9, 130.0, 129.7, 128.9, 127.6, 127.4, 126.7, 126.5, 126.5, 121.8, 111.1, 50.1, 45.6, 43.3, 31.6, 28.2, 27.4 ppm. HRMS (ESI, m/z): calcd for $C_{25}H_{23}BrO_2S$ ($M+H^+$) 467.4179; found: 467.4111.

**2-((4-chlorophenyl)(2-mercaptonaphthalen-1-yl)methyl)-3-hydroxy-5,5-dimethylcyclohex-2-enone (4j).** Yield 91%; white solid; mp 168-170 °C. IR (KBr): ν= 3388, 2989, 2655, 1720, 1575 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 9.48 (s, 1H), 7.83 (s, 1H), 7.79-7.72 (m, 3H), 7.50-7.45 (m, 3H), 7.40 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 6.22 (s, 1H), 2.39 (d, *J* = 16.1 Hz, 1H), 2.14 (d, *J* = 16.2 Hz, 2H), 2.00 (d, *J* = 16.2 Hz, 1H), 0.94 (s, 3H), 0.60 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.8, 173.5, 136.3, 133.6, 133.4, 130.0, 129.6, 129.3, 128.9, 128.8, 127.6, 127.5, 127.4, 126.7, 126.5, 111.3, 50.1, 45.6, 43.3, 31.6, 28.2, 27.4 ppm. HRMS (ESI, m/z): calcd for $C_{25}H_{23}ClO_2S$ ($M+H^+$) 422.9669; found: 422.9600.

**2-((4-fluorophenyl)(2-mercaptonaphthalen-1-yl)methyl)-3-hydroxy-5,5-dimethylcyclohex-2-enone (4k).** Yield 93%; white solid; mp 139-141 °C. IR (KBr): ν= 3430, 3040, 2615, 1700, 1570 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 9.58 (s, 1H), 7.82 (s, 1H), 7.79-7.72 (m, 3H), 7.47-7.44 (m, 5H), 7.04-6.99 (m, 2H), 6.23 (s, 1H), 2.40 (d, *J* = 16.1 Hz, 1H), 2.15 (d, *J* = 16.2 Hz, 2H), 2.02 (d, *J* = 16.2 Hz, 1H), 0.95 (s, 3H), 0.61 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.8, 173.4, 162.0 (d, *J* = 250.1 Hz), 133.4, 132.4, 130.2, 129.7, 129.6, 129.5, 128.8, 127.6, 127.5, 127.4, 126.6, 126.4, 115.8, 115.5, 111.5, 50.1, 45.5, 43.3, 31.6, 28.2, 27.3 ppm. HRMS (ESI, m/z): calcd for $C_{25}H_{23}FO_2S$ ($M+H^+$) 406.1402; found: 406.13901.

**3-hydroxy-2-((2-mercaptonaphthalen-1-yl)(4-nitrophenyl)methyl)-5,5-dimethylcyclohex-2-enone (4l).** Yield 90%; white solid; mp 163-165 °C. IR (KBr): ν= 3452, 3033, 2644, 1719, 1601 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 9.48 (s, 1H), 8.20 (d, *J* = 8.2 Hz, 2H), 7.86 (s, 1H), 7.81-7.74 (m, 3H), 7.63 (d, *J* = 8.0 Hz, 2H), 7.51-7.45 (m, 3H), 6.28 (s, 1H), 2.42 (d, *J* = 16.1 Hz, 1H), 2.21 (d, *J* = 16.2 Hz, 1H), 2.16 (d, *J* = 16.2 Hz, 1H), 2.00 (d, *J* = 16.2 Hz, 1H), 0.95 (s, 3H), 0.64 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.3, 173.7, 147.2, 145.8, 133.4, 132.5, 129.9, 129.7, 129.0, 128.9, 127.6, 127.4, 126.8, 126.7, 123.9, 111.3, 50.1, 45.9, 43.3, 31.7, 28.5, 27.3 ppm. HRMS (ESI, m/z): calcd for $C_{25}H_{23}NO_4S$ ($M+H^+$) 433.1347; found: 433.1332.

**3-hydroxy-2-((2-mercaptonaphthalen-1-yl)(p-tolyl)methyl)-5,5-dimethylcyclohex-2-enone (4m).** Yield 93%; white solid; mp 185-187 °C. IR (KBr): ν= 3434, 3001, 2617, 1709, 1570 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 9.46 (s, 1H), 7.82 (s, 1H), 7.78-7.71 (m, 3H), 7.47-7.44 (m, 3H), 7.35(d, *J* = 7.6 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 6.27 (s, 1H), 2.38 (d, *J* = 16.1 Hz, 1H), 2.32 (s, 3H), 2.14 (d, *J* = 16.2 Hz, 2H), 2.00 (d, *J* = 16.2 Hz, 1H), 0.95 (s, 3H), 0.59 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.7, 173.3, 137.6, 134.4, 133.5, 132.4, 130.5, 129.5, 129.4, 128.7,

127.8, 127.6, 127.4, 126.6, 126.3, 111.5, 50.2, 45.8, 43.3, 31.6, 28.2, 27.3, 21.0 ppm. HRMS (ESI, m/z): calcd for $C_{26}H_{26}O_2S$ ($M+H^+$) 402.1653; found: 402.1650.

**3-hydroxy-2-((4-isopropylphenyl)(2-mercaptonaphthalen-1-yl)methyl)-5,5-dimethylcyclohex-2-enone (4n).** Yield 91%; white solid; mp 138-140 °C. IR (KBr): ν= 3422, 3011, 2633, 1741, 1555 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 9.51 (s, 1H), 7.82 (s, 1H), 7.86-7.72 (m, 3H), 7.48-7.45 (m, 3H), 7.38 (d, *J* = 7.8 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 6.28 (s, 1H), 2.94-2.83 (m, 1H), 2.40 (d, *J* = 16.1 Hz, 1H), 2.14 (d, *J* = 16.2 Hz, 2H), 2.00 (d, *J* = 16.2 Hz, 1H), 1.23 (d, *J* = 6.9 Hz, 6H), 0.96 (s, 3H), 0.58 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.7, 173.3, 148.5, 134.6, 133.5, 132.4, 130.5, 129.4, 128.7, 127.9, 127.6, 127.4, 126.9, 126.3, 111.4, 50.2, 45.8, 43.3, 33.7, 31.6, 28.3, 27.3, 23.8 ppm. HRMS (ESI, m/z): calcd for $C_{28}H_{30}O_2S$ ($M+H^+$) 430.1966; found: 430.1930.

2-**((4-(dimethylamino)phenyl)(2-mercaptonaphthalen-1-yl)methyl)-3-hydroxy-5,5-dimethyl-cyclohex-2-enone (4o).** Yield 92%; white solid; mp 146-148 °C. IR (KBr): ν= 3430, 3050, 2619, 1706, 1575 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 9.52 (s, 1H), 7.80 (s, 1H), 7.76-7.71 (m, 3H), 7.68-7.66 (m, 3H), 7.33 (d, *J* = 8.1Hz, 2H), 6.68 (d, *J* = 8.0 Hz, 2H), 6.26 (s, 1H), 2.92 (s, 6H), 2.55 (d, *J* = 16.1 Hz, 1H), 2.38 (d, *J* = 16.1 Hz, 1H), 2.13 (d, *J* = 16.1 Hz, 1H), 2.00 (d, *J* = 16.1 Hz, 1H), 0.95 (s, 3H), 0.58 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.6, 173.0, 150.0, 138.5, 133.4, 132.2, 131.0, 130.3, 129.1, 128.8, 128.5, 127.5, 127.4, 127.3, 126.4, 126.2, 126.1, 126.0, 124.4, 111.2, 50.2, 45.7, 43.2, 40.3, 31.5, 28.2, 27.3 ppm. HRMS (ESI, m/z): calcd for $C_{27}H_{29}NO_2S$ ($M+H^+$) 431.1919; found: 431.1800.

**2-((4-ethoxyphenyl)(2-mercaptonaphthalen-1-yl)methyl)-3-hydroxy-5,5-dimethylcyclohex-2-enone (4p).** Yield 91%; white solid; mp 142-144 °C. IR (KBr): ν= 3395, 3052, 2595, 1719, 1562 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 9.53 (s, 1H), 7.81 (s, 1H), 7.78-7.71 (m, 3H), 7.47-7.44 (m, 3H), 7.37 (d, *J* = 8.4 Hz, 2H), 6.85 (d, *J* = 8.8 Hz, 2H), 6.25 (s, 1H), 4.03-3.98 (q, 2H), 2.39 (d, *J* = 16.1 Hz, 1H), 2.14 (d, *J* = 16.2 Hz, 2H), 2.00 (d, *J* = 16.2 Hz, 1H), 1.40 (t, *J* = 6.9 Hz, 3H), 0.95 (s, 3H), 0.59 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.7, 173.2, 158.3, 133.5, 132.3, 130.6, 129.3, 129.1, 128.7, 127.6, 127.5, 127.4, 126.5, 126.3, 114.7, 116.6, 63.3, 50.2, 45.3, 43.3, 31.6, 28.2, 27.3, 14.7 ppm. HRMS (ESI, m/z): calcd for $C_{27}H_{28}O_3S$ ($M+H^+$) 432.1759; found: 432.1745.

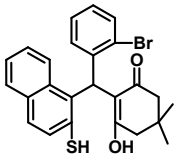
**2-((2,5-dimethylphenyl)(2-mercaptonaphthalen-1-yl)methyl)-3-hydroxy-5,5-dimethylcyclohex-2-enone (4q).** Yield 91%; white solid; mp 159-161 °C. IR (KBr): ν= 3423, 3050, 2656, 1700, 1559 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 10.06 (s, 1H), 7.82 (s, 1H), 7.79-7.73 (m, 3H), 7.47-7.44 (m, 3H), 7.10 (t, *J* = 6.9 Hz, 2H), 7.00 (d, *J* = 8.1Hz, 1H), 6.23 (s, 1H), 2.63 (s, 3H), 2.47 (d, *J* = 16.1 Hz, 1H), 2.29 (s, 3H), 2.23 (d, *J* = 16.2 Hz, 1H), 2.16 (d, *J* = 16.2 Hz, 1H), 2.06 (d, *J* = 16.2 Hz, 1H), 0.98 (s, 3H), 0.70 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.7, 173.5, 135.8, 135.4, 133.8, 133.5, 132.3, 131.0, 130.8, 128.9, 128.8, 127.7, 127.6, 127.3, 127.2, 126.6, 126.2, 111.3, 50.2, 44.1, 43.4, 31.7, 28.1, 27.4, 21.2, 18.7. HRMS (ESI, m/z): calcd for $C_{27}H_{28}O_2S$ ($M+H^+$) 416.1810; found: 416.1801.

2-**(benzo[d][1,3]dioxol-5-yl(2-mercaptonaphthalen-1-yl)methyl)-3-hydroxy-5,5-dimethyl-cyclohex-2-enone (4r).** Yield 94%; white solid; mp 183-185 °C. IR (KBr): ν= 3385, 3046, 2635, 1732, 1578 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 9.96 (s, 1H), 7.76 (s, 1H), 7.74-7.68 (m, 3H),

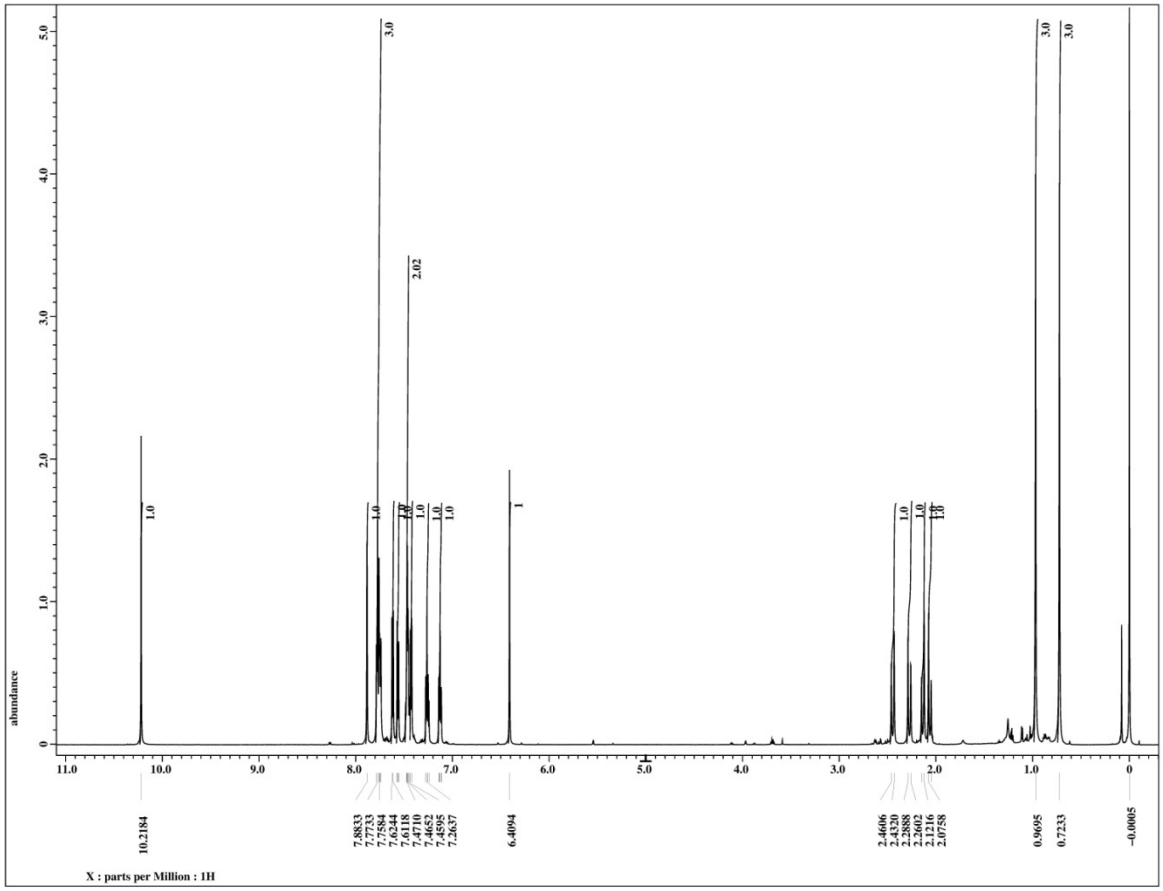
7.44-7.42 (m, 3H), 7.09 (s, 1H), 6.99 (d, $J$ = 8.1 Hz, 1H), 6.73 (d, $J$ = 7.6 Hz, 1H), 6.05 (s, 1H), 5.91 (s, 2H), 2.38 (d, $J$ = 16.1 Hz, 1H), 2.26 (d, $J$ = 16.2 Hz, 1H), 2.19 (d, $J$ = 16.1 Hz, 1H), 2.10 (d, $J$ = 16.2 Hz, 1H), 0.98 (s, 3H), 0.78 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.3, 172.1, 147.3, 146.3, 133.3, 133.0, 131.7, 128.1, 127.9, 127.3, 127.0, 126.2, 125.6, 120.9, 113.4, 108.4, 107.7, 100.7, 50.1, 45.5, 43.4, 31.5, 28.1, 27.5ppm. HRMS (ESI, m/z): calcd for $C_{26}H_{24}O_4S$ ($M+H^+$) 432.1395; found: 432.1300.

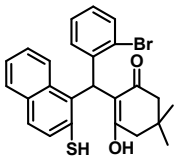
**3-hydroxy-2-((2-mercaptonaphthalen-1-yl)(phenyl)methyl)-5,5-dimethylcyclohex-2-enone (4s).** Yield 90%; white solid; mp 168-170 °C. IR (KBr): ν= 3423, 3023, 2675, 1709, 1586 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 9.55 (s, 1H), 7.83 (s, 1H), 7.78-7.71 (m, 3H), 7.48-7.42 (m, 5H), 7.34 (d, $J$ = 6.9 Hz, 2H), 7.27 (d, $J$ = 8.0 Hz, 1H), 6.29 (s, 1H), 2.39 (d, $J$ = 16.1 Hz, 1H), 2.15 (d, $J$ = 16.2 Hz, 2H), 2.01 (d, $J$ = 16.2 Hz, 1H), 0.94 (s, 3H), 0.59 (s, 3H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.8, 173.4, 137.7, 133.4, 132.3, 130.7, 129.3, 128.7, 128.6, 127.9, 127.7, 127.6, 127.5, 127.3, 126.5, 126.2, 111.6, 50.1, 46.0, 43.3, 31.6, 28.2, 27.3 ppm. HRMS (ESI, m/z): calcd for $C_{25}H_{24}O_2S$ ($M+H^+$) 388.1497; found: 388.1485.

**2,2'-(1,4-phenylenebis((2-mercaptonaphthalen-1-yl)methylene))bis(3-hydroxy-5,5-dimethylcyclohex-2-enone)** (4t). Yield 90%; white solid; mp 213-215 °C. IR (KBr): ν= 3423, 3023, 2675, 1709, 1586 $cm^{-1}$. $^1$H-NMR (400 MHz, $CDCl_3$): δ 9.51 (s, 1H), 9.47 (s, 1H), 7.83 (s, 2H), 7.79-7.73 (m, 6H), 7.49-7.44 (m, 10H), 6.27 (d, $J$ = 2.7 Hz, 2H), 2.41 (d, $J$ = 16.1 Hz, 2H), 2.14 (dd, $J$ = 3.6, 16.2 Hz, 4H), 2.00 (d, $J$ = 16.2 Hz, 2H), 0.96 (s, 6H), 0.58 (s, 6H) ppm. $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 196.9, 173.4, 137.7, 133.4, 132.3, 130.7, 129.3, 128.7, 128.6, 127.9, 127.7, 127.6, 127.5, 127.3, 126.5, 126.2, 111.2, 50.1, 46.0, 43.3, 31.6, 28.2, 27.3 ppm. HRMS (ESI, m/z): calcd for $C_{44}H_{42}O_4S_2$ ($M+H^+$) 698.2524; found: 698.2012.

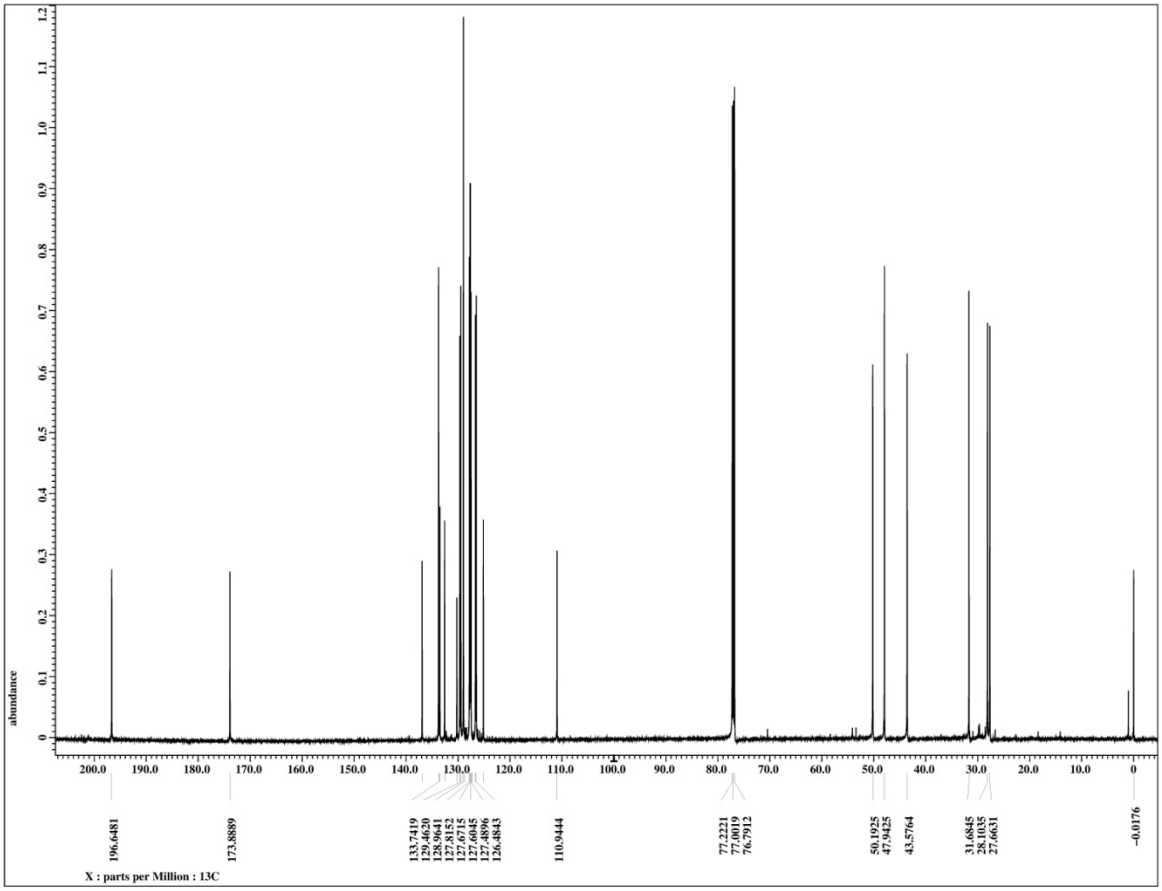

Br
O
SH
OH

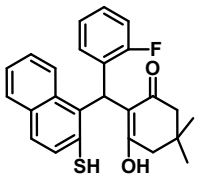
4a-$^{1}$H-NMR


abundance
X : parts per Million : 1H
10.2184
7.8833
7.7733
7.7584
7.6344
7.6118
7.4710
7.4652
7.4595
7.2637
6.4094
2.4606
2.4320
2.2888
2.2602
2.1216
2.0758
0.9695
0.7233
-0.0005

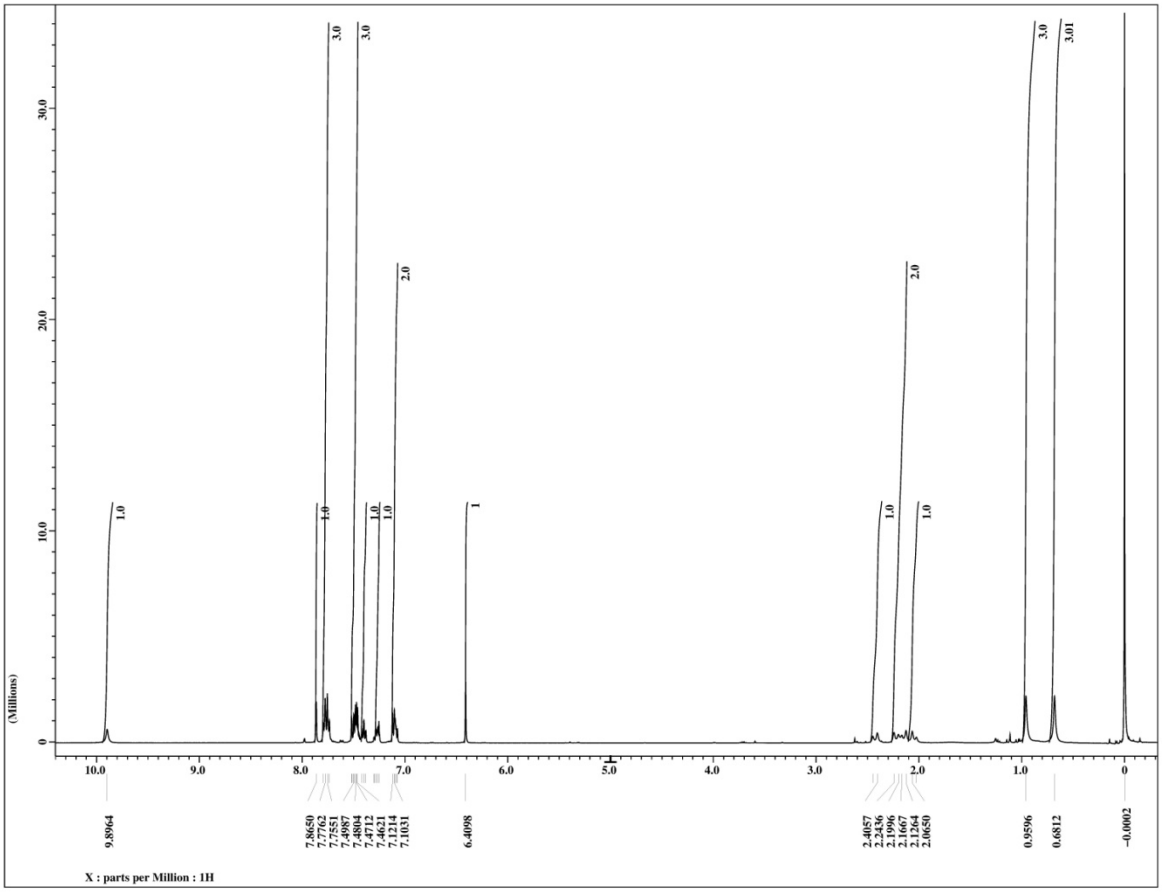

Br
O
SH
OH

4a-$^{13}$C-NMR


abundance
196.6481
173.8889
133.7419
129.4620
128.9641
127.8152
127.6715
127.6045
127.4896
126.4843
110.9444
77.2221
77.0019
76.7912
50.1925
47.9425
43.5764
31.6845
28.1035
27.6631
-0.0176
X : parts per Million : 13C


F
O
SH
OH

4b-$^{1}$H-NMR


(Millions)
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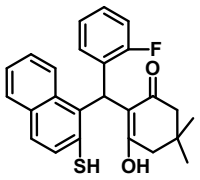
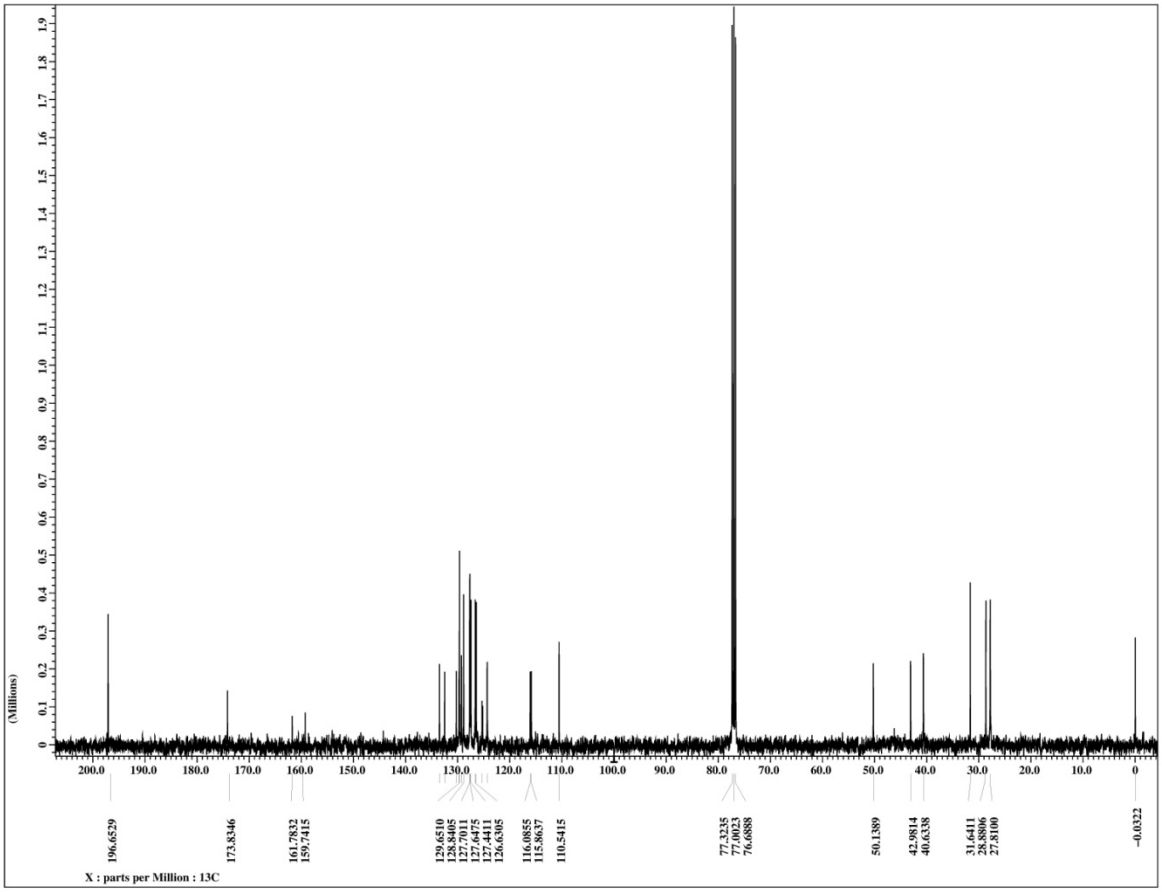
20.0
10.0
0
3.0
3.0
2.0
1.0
1.0
1.0
1.0
1
1.0
2.0
1.0
3.0
3.01
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.8964
7.8650
7.7762
7.7551
7.4987
7.4804
7.4712
7.4643
7.4611
7.1214
7.1031
6.4098
2.4057
2.3436
2.1996
2.1667
2.1264
2.0650
0.9596
0.6812
-0.0002
X : parts per Million : 1H


F
O
SH
OH

4b-$^{13}$C-NMR


(Millions)
X : parts per Million : 13C
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173.8346
161.7832
159.7415
129.6510
129.4015
127.7011
127.6475
127.4411
126.6305
116.0855
115.8657
110.5415
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76.6888
50.1389
42.9814
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31.6411
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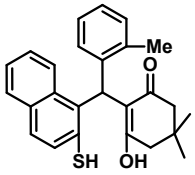
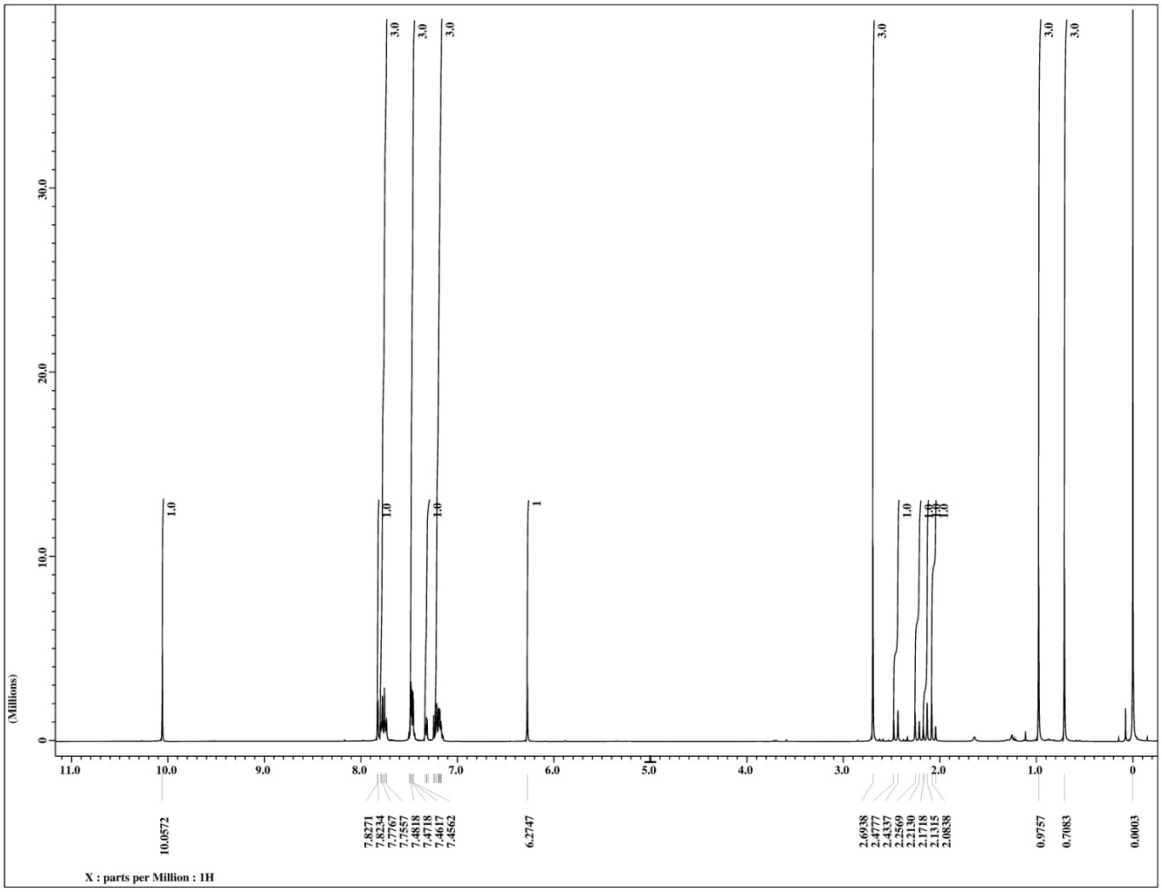
27.8100
-0.0322


Me
O
SH
OH

4c-$^{1}$H-NMR


(Millions)
X : parts per Million : 1H
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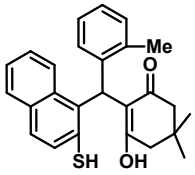
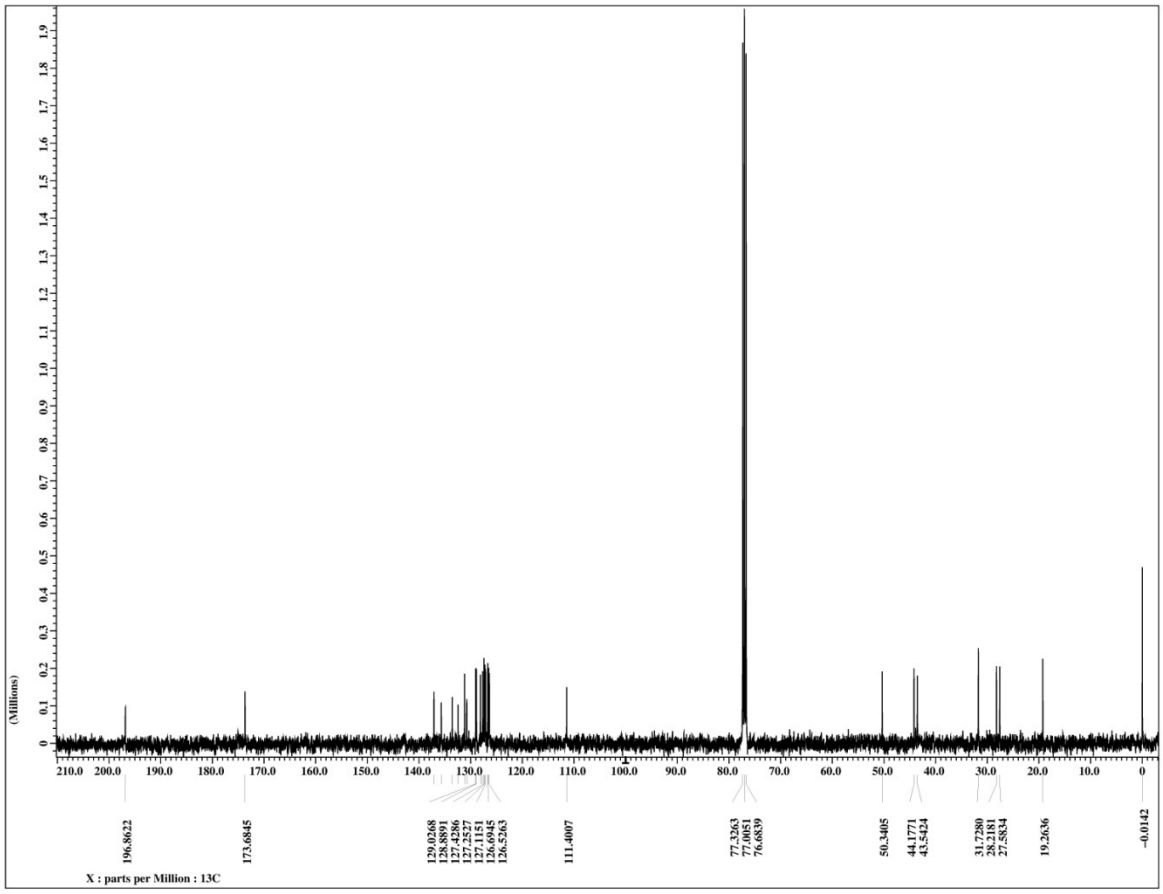
7.7767
7.7557
7.4818
7.4718
7.4617
7.4562
6.2747
2.6938
2.4777
2.4437
2.2560
2.2130
2.1718
2.1315
2.0838
0.9757
0.7083
0.0003


Me
O
SH
OH

4c-$^{13}$C-NMR


(Millions)
X : parts per Million : 13C
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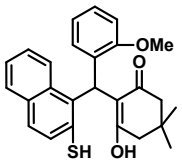
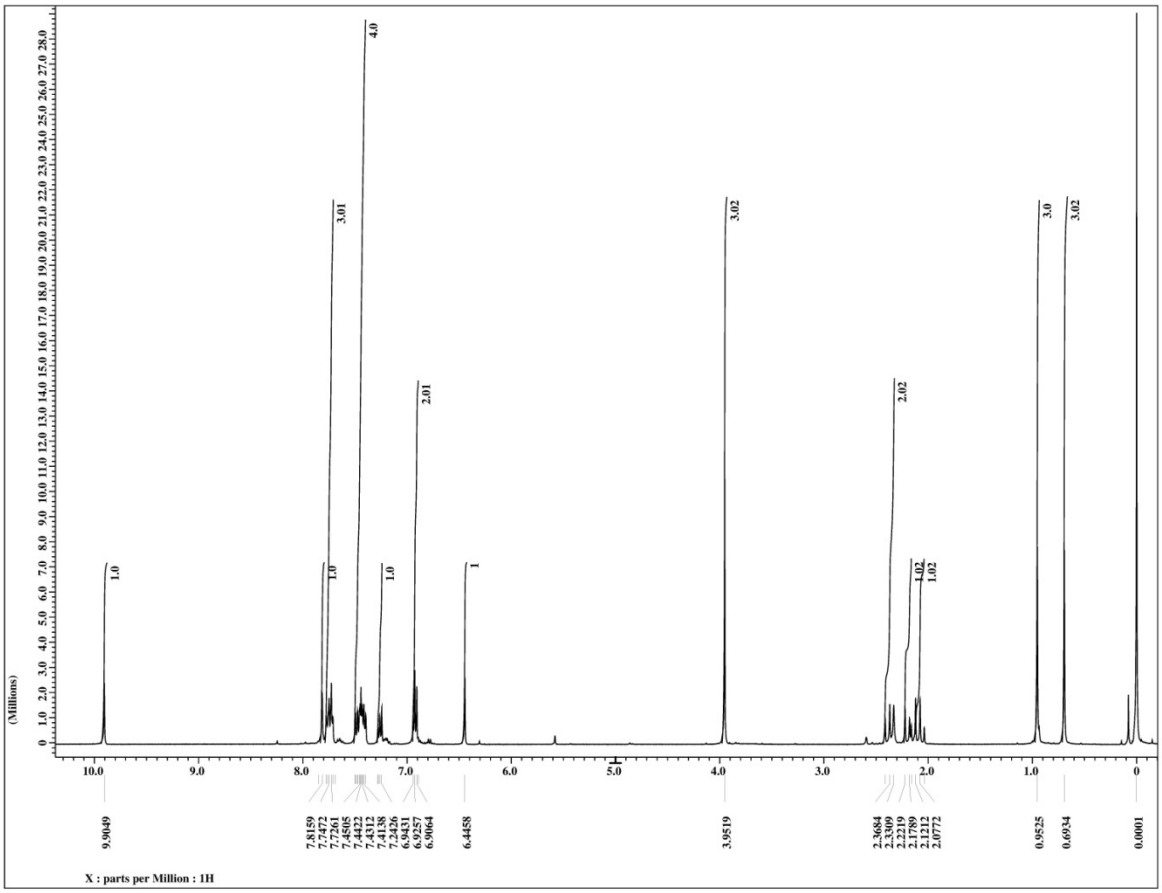
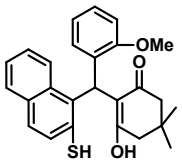
173.6845
129.0268
128.8891
127.4286
127.2527
127.1151
126.6945
126.5263
111.4007
77.3263
77.0045
76.6839
50.3405
44.1771
43.5424
31.7280
28.2181
27.5834
19.2636
-0.0142

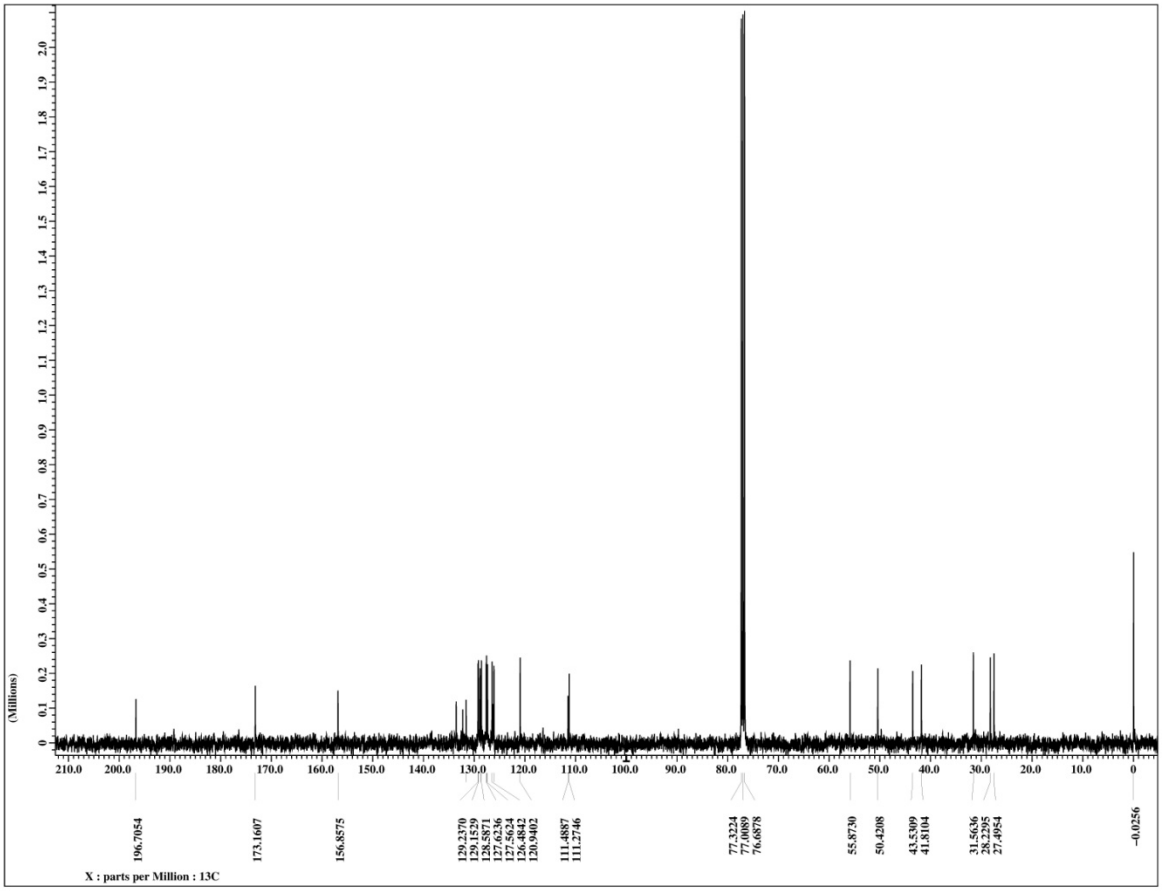



4d-$^{1}$H-NMR




OMe
O
SH
OH

4d-$^{13}$C-NMR


(Millions)
X : parts per Million : 13C
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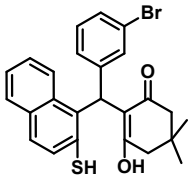
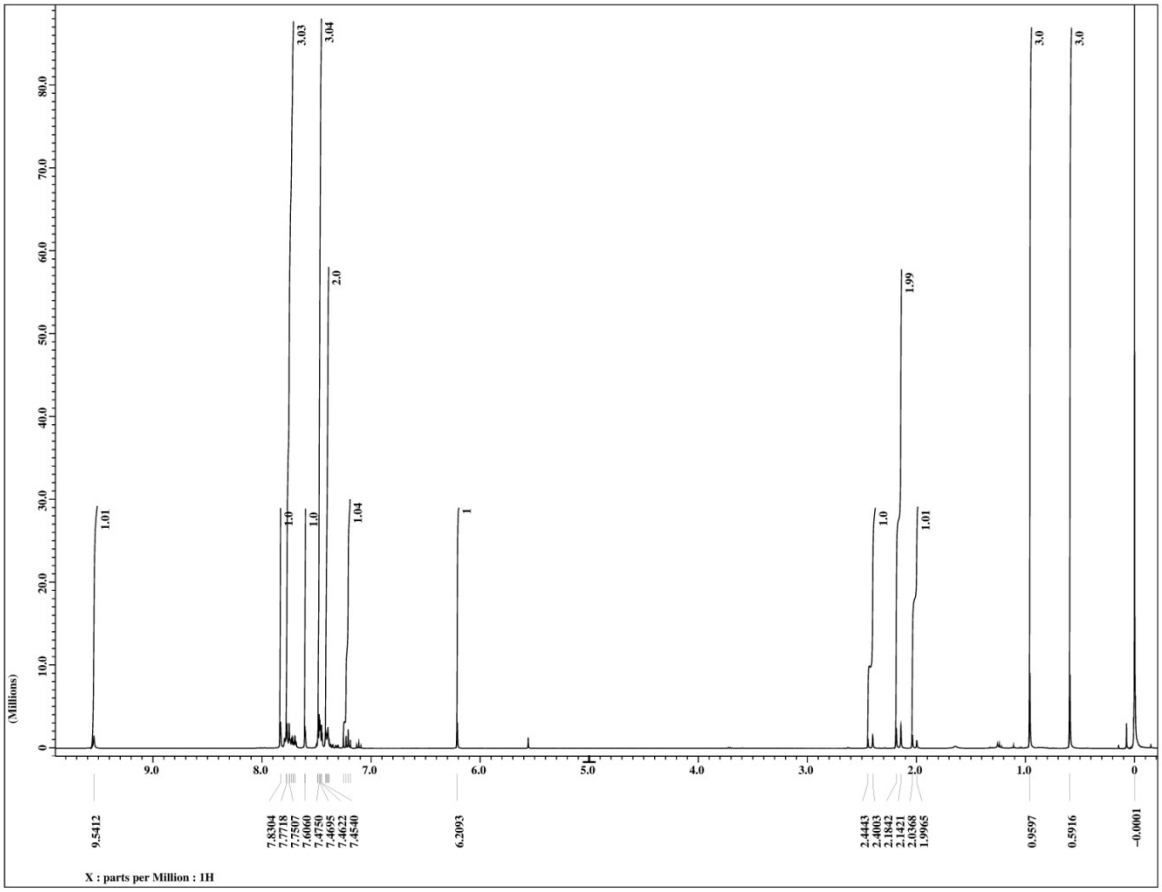
129.2370
129.1529
128.5871
127.6236
127.5624
126.4612
120.9402
111.4887
111.2746
77.3224
77.0049
76.6878
55.8730
50.4208
43.5309
41.8104
31.5636
28.2295
27.4954
-0.0256


Br
O
SH
OH

4e-$^{1}$H-NMR


(Millions)
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50.0
40.0
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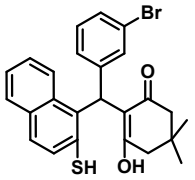
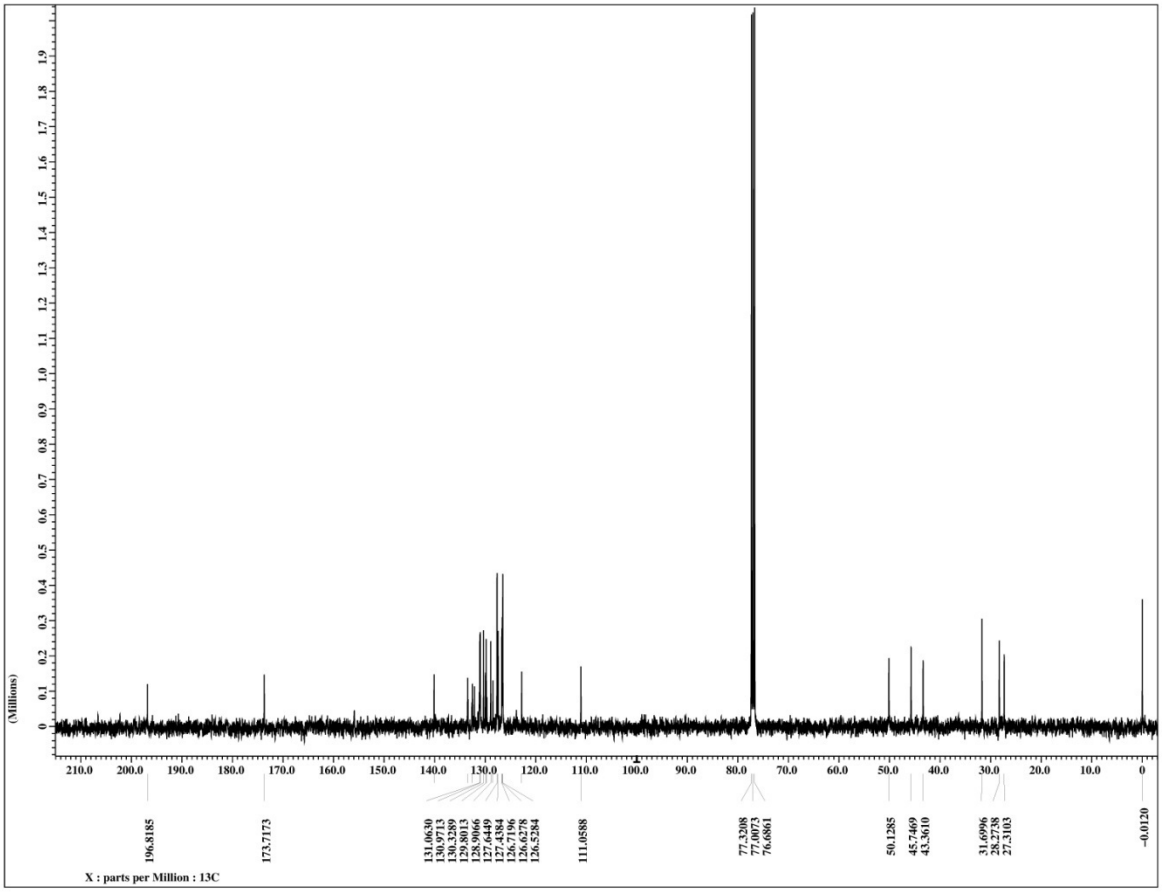
20.0
10.0
0
1.01
3.03
3.04
2.0
1.04
1.0
1.0
1
1.0
1.99
1.01
3.0
3.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.5412
7.8304
7.7738
7.7507
7.6060
7.4750
7.4695
7.4622
7.4540
6.2093
2.4443
2.4003
2.1842
2.1431
2.1368
1.9965
0.9597
0.5916
-0.0001
X : parts per Million : 1H


Br
O
SH
OH

4e-$^{13}$C-NMR


(Millions)
X : parts per Million : 13C
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131.0630
130.9713
130.2030
129.8013
128.9066
127.6449
127.4384
126.7196
126.6278
126.5284
111.0588
77.3208
77.0073
76.6861
50.1285
45.7469
43.3610
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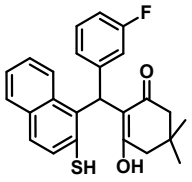
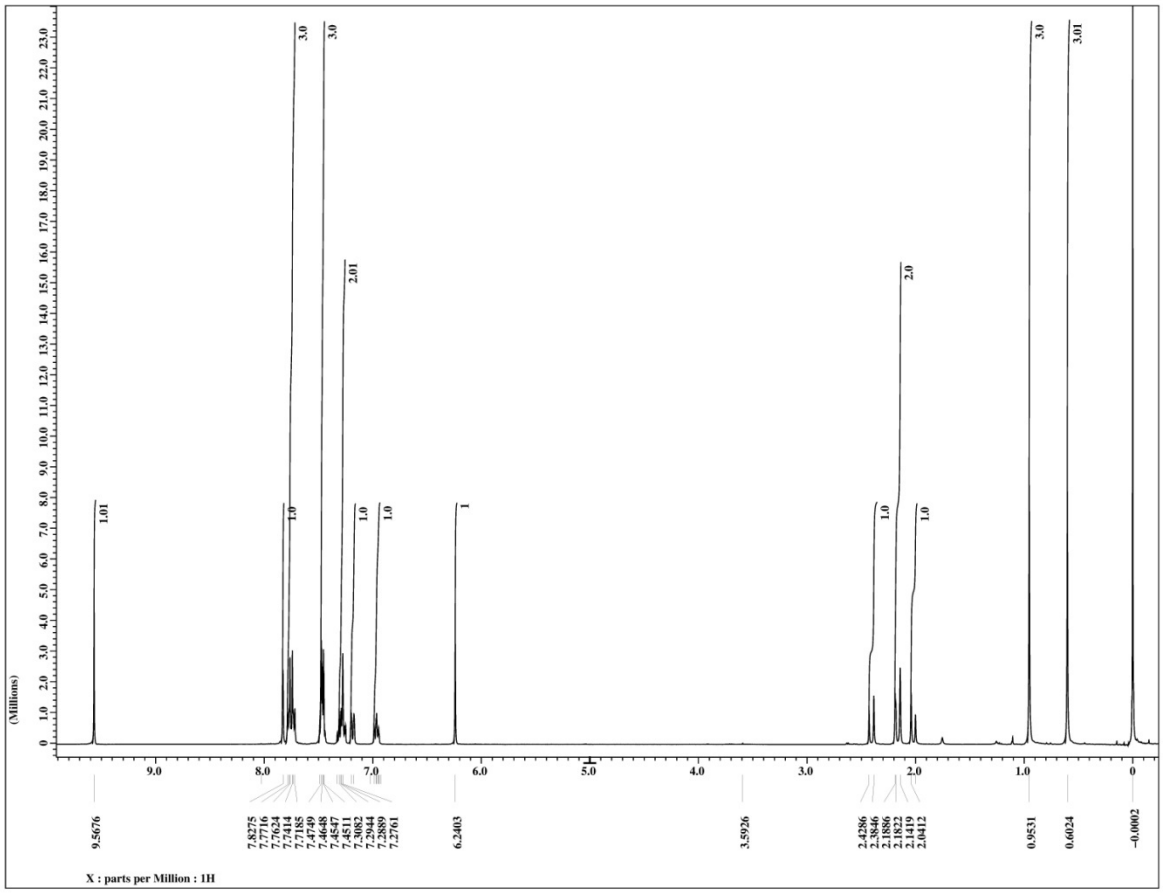
27.3103
-0.0120


F
O
SH
OH

4f-$^{1}$H-NMR


(Millions)
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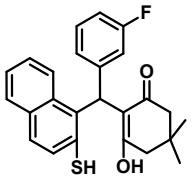
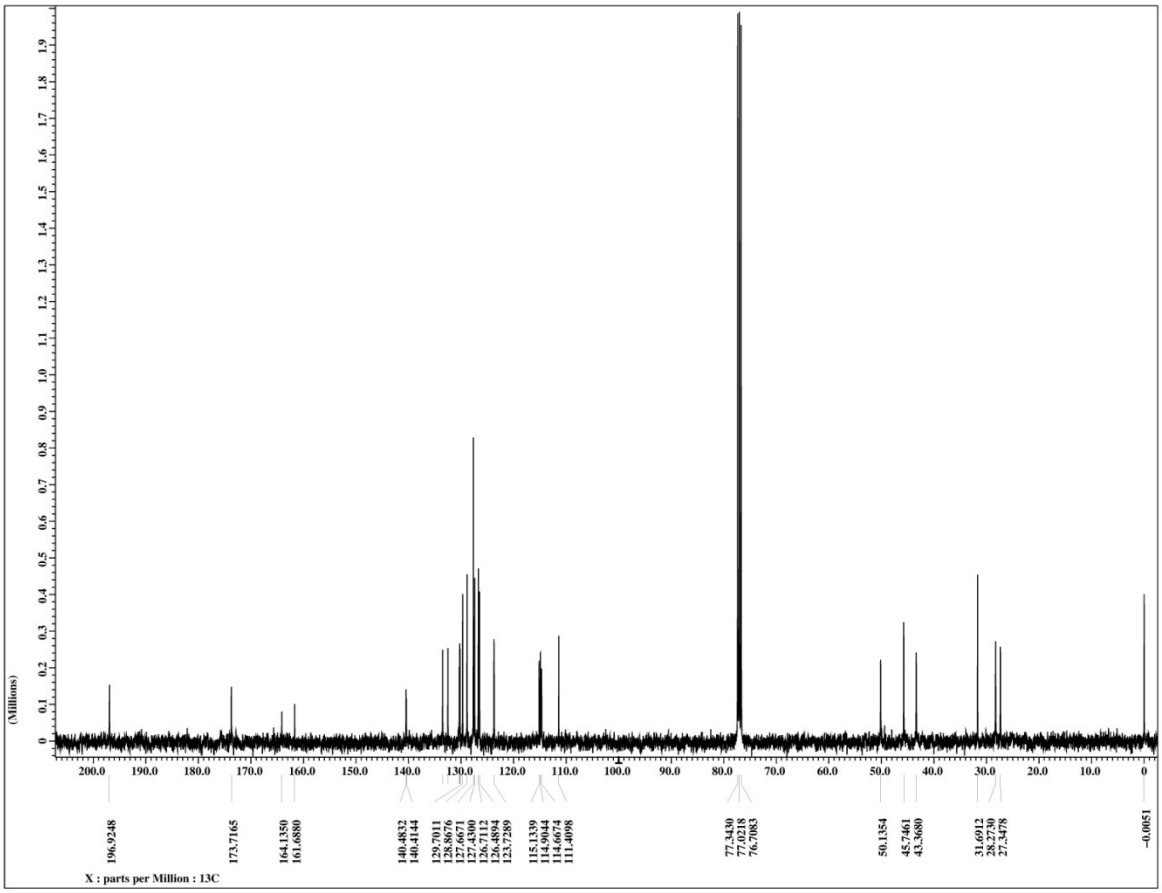
3.0
2.01
1.0
1.0
1
1.0
2.0
1.0
3.0
3.01
9.5676
7.8275
7.7716
7.7624
7.7414
7.7195
7.4749
7.4648
7.4547
7.4511
7.3082
7.2844
7.2809
7.2761
6.2403
3.5926
2.4286
2.3846
2.1956
2.1822
2.1419
2.0412
0.9531
0.6024
-0.0002
X : parts per Million : 1H


F
O
SH
OH

4f-$^{13}$C-NMR


(Millions)
X : parts per Million : 13C
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161.6880
140.4832
140.4144
129.7011
128.8676
127.6671
127.4300
126.7112
126.4894
123.7289
115.1339
114.9044
114.6674
111.4098
77.3430
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50.1354
45.7461
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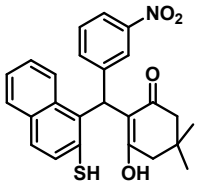
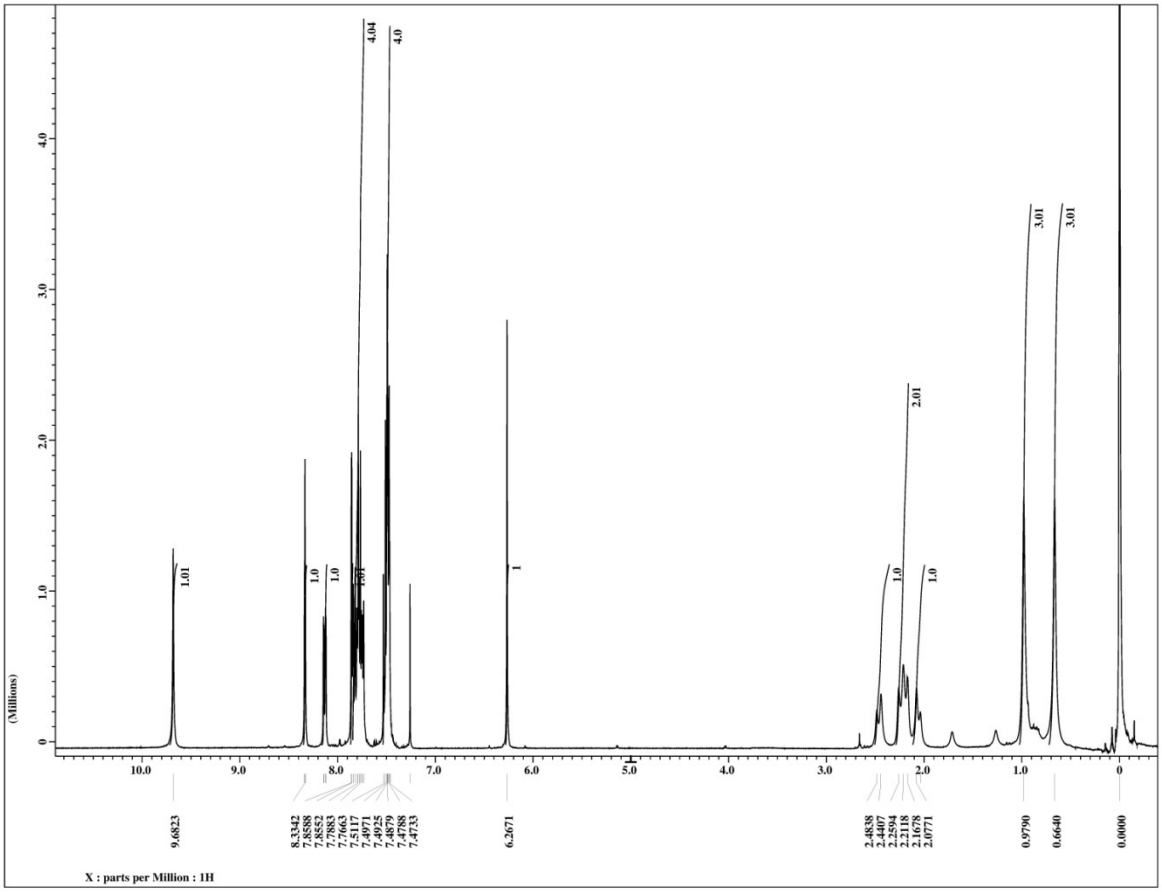
31.6912
28.2730
27.3478
-0.0051


NO2
O
SH
OH

4g-$^{1}$H-NMR


(Millions)
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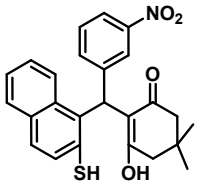
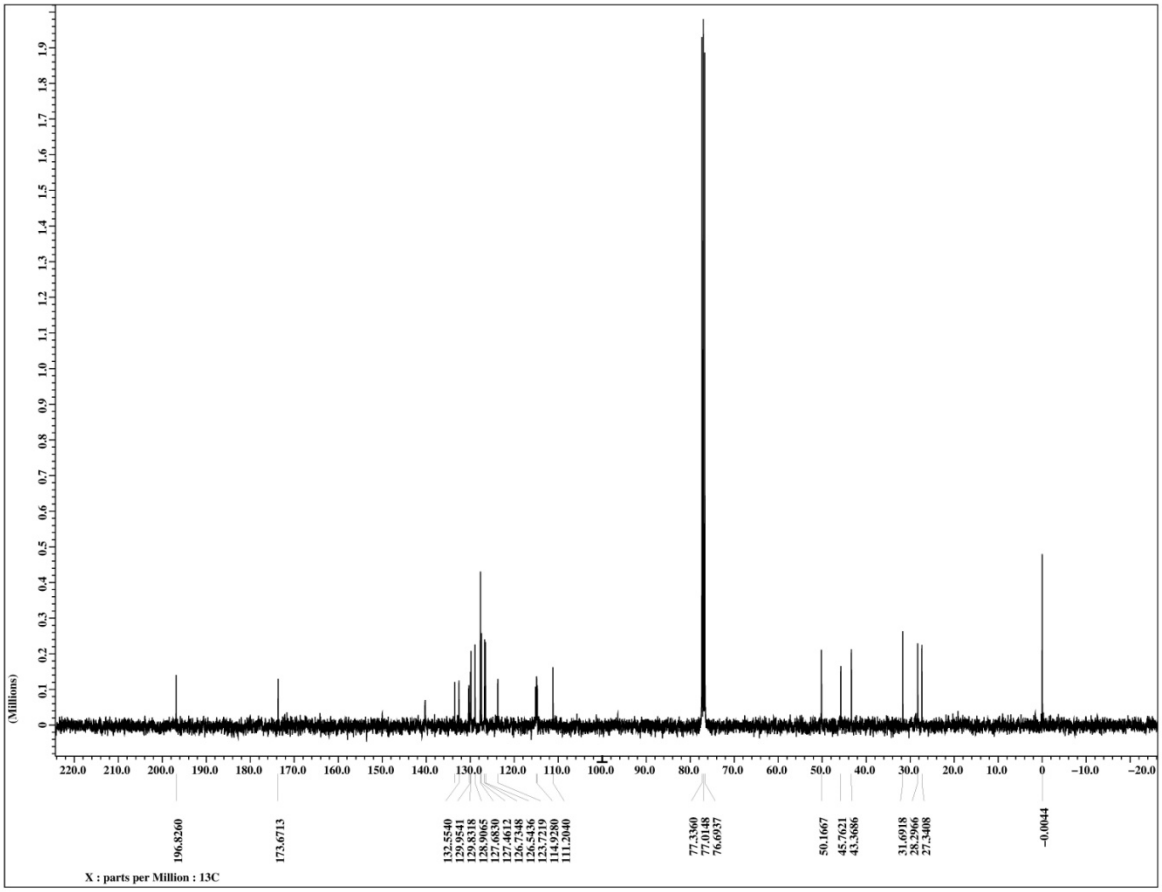
2.0
1.0
0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
1.01
1.0
1.0
1.01
4.04
4.0
1
1.0
2.01
1.0
3.01
3.01
9.6823
8.3342
7.8588
7.8552
7.7883
7.7663
7.5417
7.4971
7.4925
7.4879
7.4788
7.4733
6.2671
2.4838
2.4407
2.2594
2.2118
2.1678
2.0771
0.9790
0.6640
0.0000
X : parts per Million : 1H


NO2
O
SH
OH

4g-$^{13}$C-NMR


(Millions)
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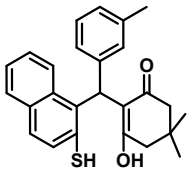
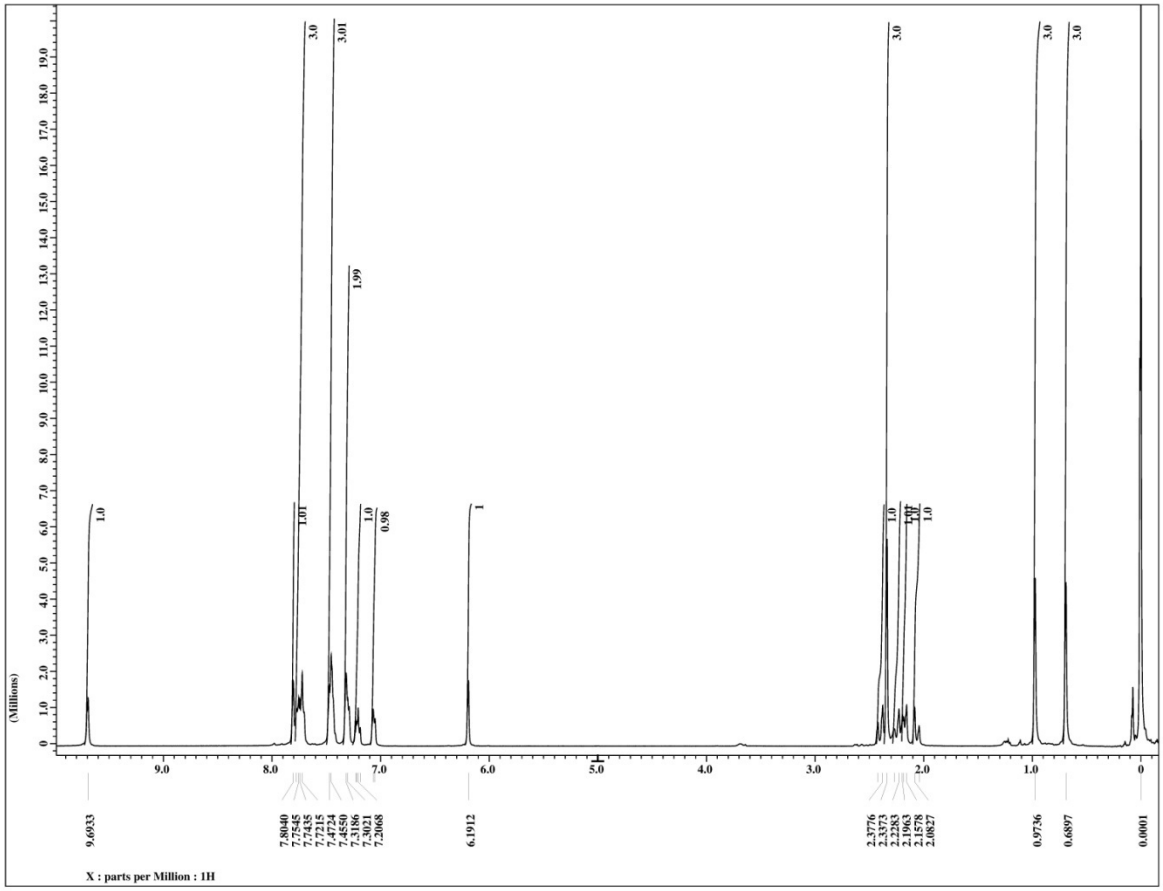
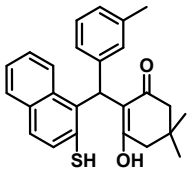
132.5540
129.9541
129.8318
128.9065
127.6830
127.4612
126.7348
126.5436
123.7219
114.9280
111.2040
77.3360
77.0148
76.6937
50.1667
45.7621
43.3686
31.6918
28.7966
27.2408
-0.0044
X : parts per Million : 13C

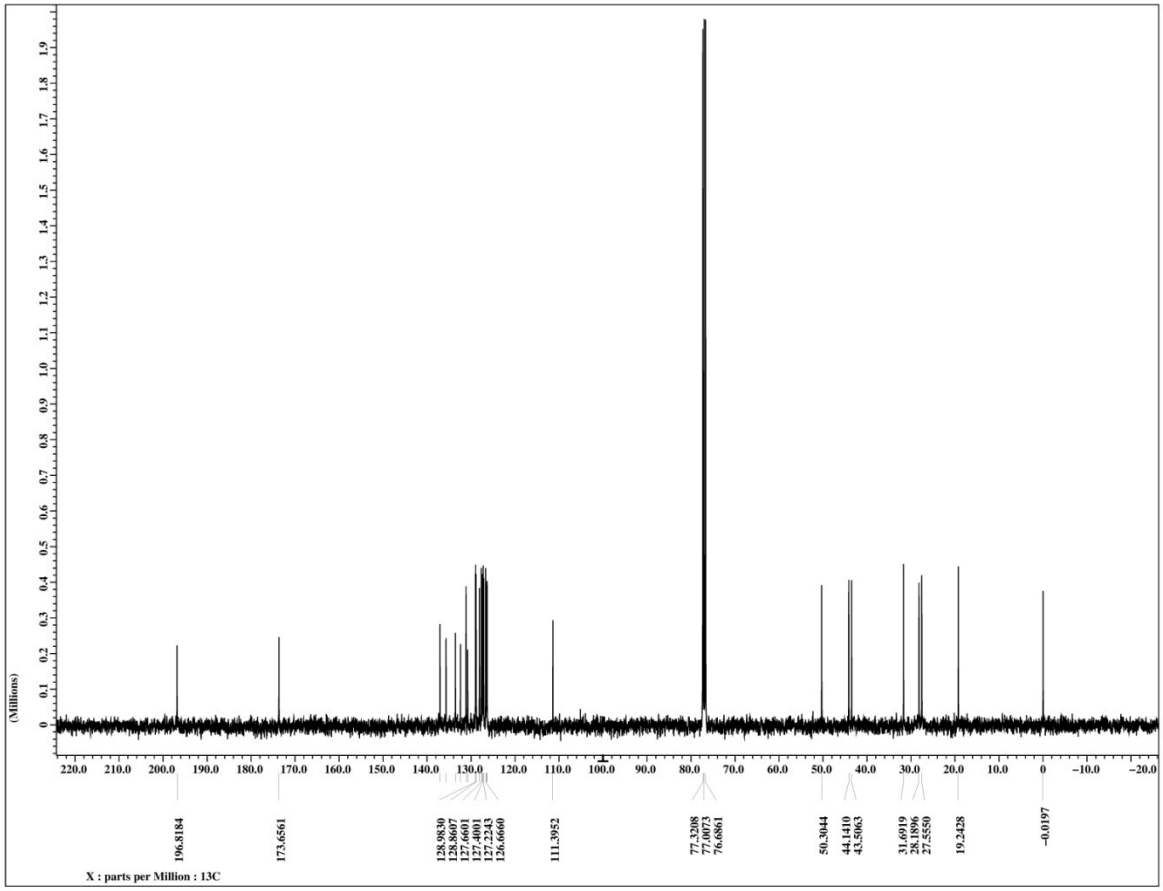

O
SH
OH

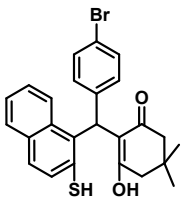
4h-$^{1}$H-NMR


(Millions)
X : parts per Million : 1H
9.6933
7.8040
7.7545
7.7435
7.7215
7.4724
7.4550
7.3156
7.3021
7.2068
6.1912
2.3776
2.3373
2.2283
2.1963
2.1578
2.0827
0.9736
0.6897
0.0001

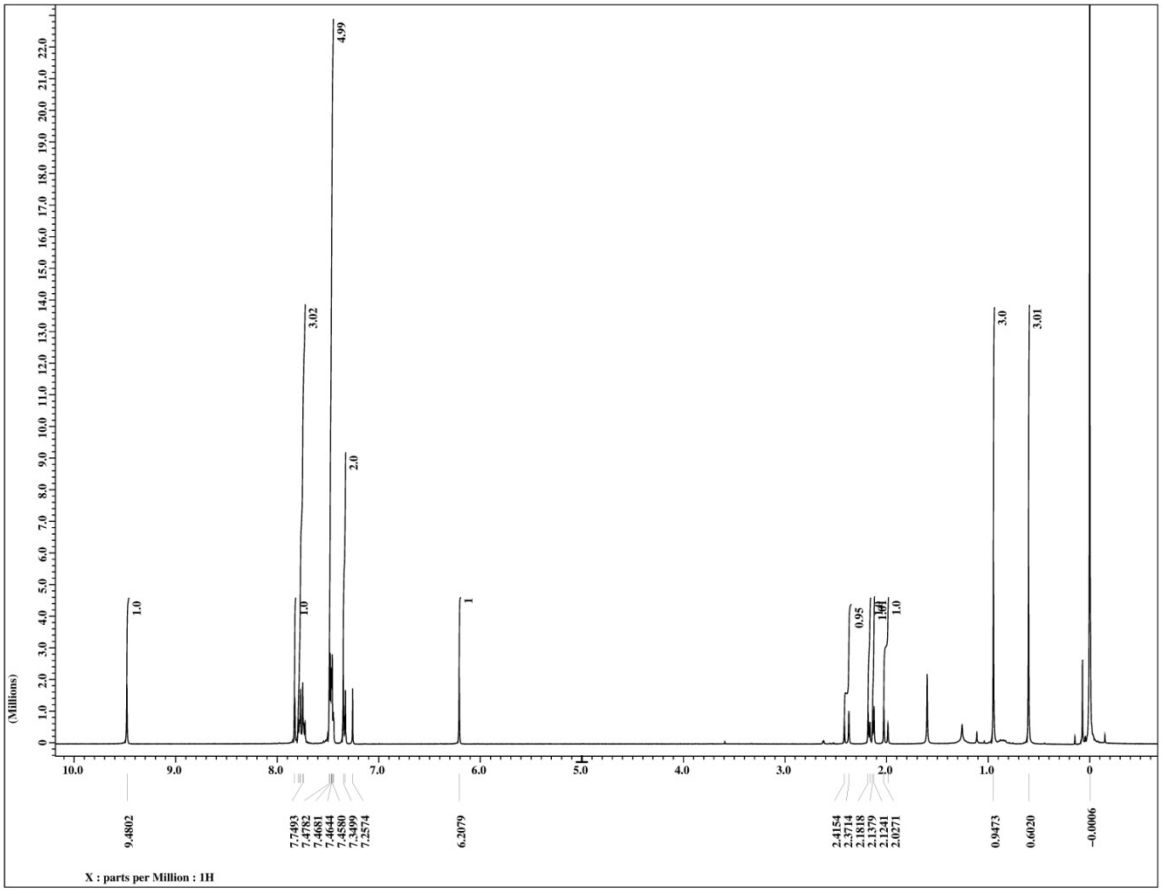

O
SH
OH

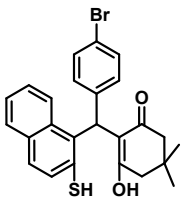
4h-$^{13}$C-NMR


(Millions)
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
1.1
1.2
1.3
1.4
1.5
1.6
1.7
1.8
1.9
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
−10.0
−20.0
196.8184
173.6561
128.9830
128.6677
127.6601
127.4001
127.2243
126.6660
111.3952
77.3208
77.0073
76.6861
50.3044
44.1410
43.5063
31.6919
28.1896
27.5550
19.2428
−0.0197
X : parts per Million : 13C

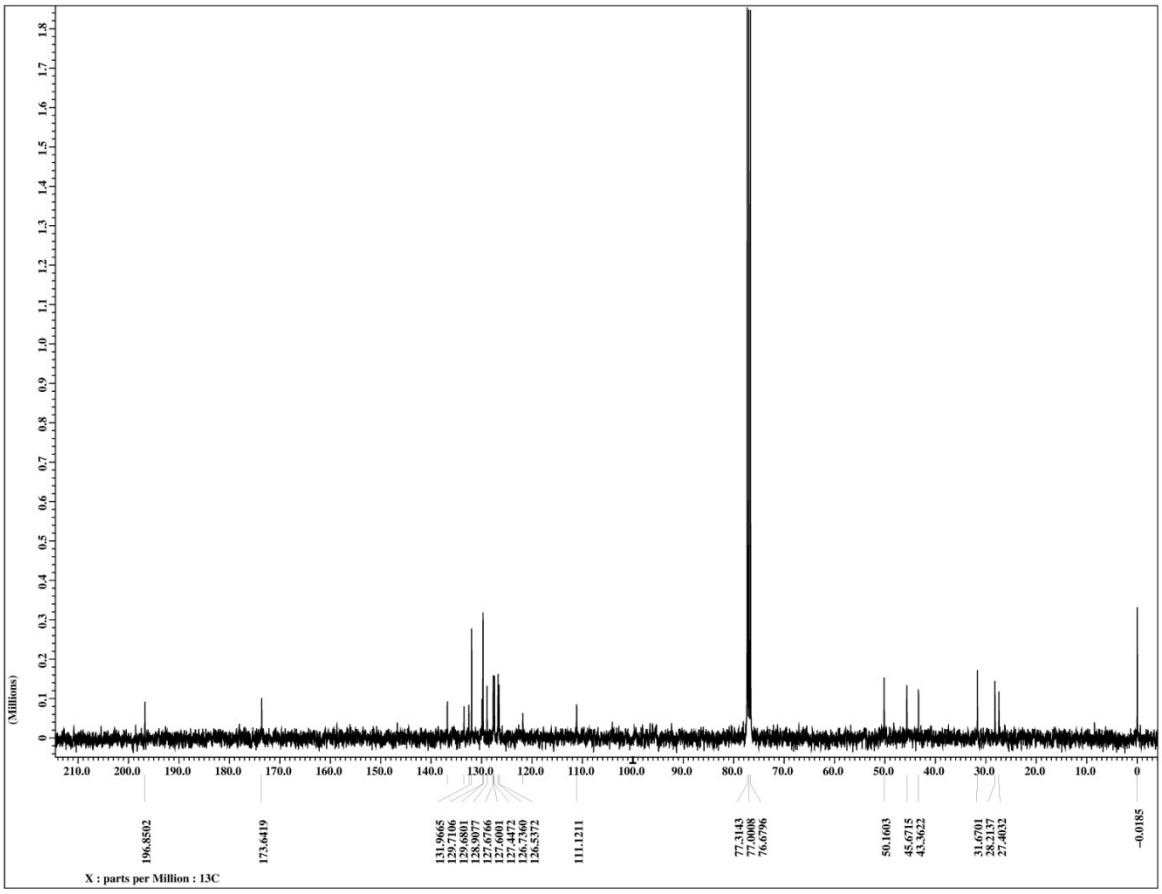

Br
O
SH
OH

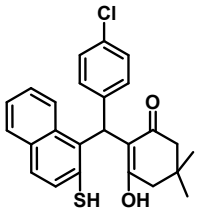
4i-$^{1}$H-NMR


(Millions)
4.99
3.02
2.0
1.0
1
0.95
3.0
3.01
9.4802
7.7493
7.4782
7.4681
7.4644
7.4580
7.3490
7.2574
6.2079
2.4154
2.3714
2.1818
2.1379
2.1241
2.0271
0.9473
0.6020
−0.0006
X : parts per Million : 1H

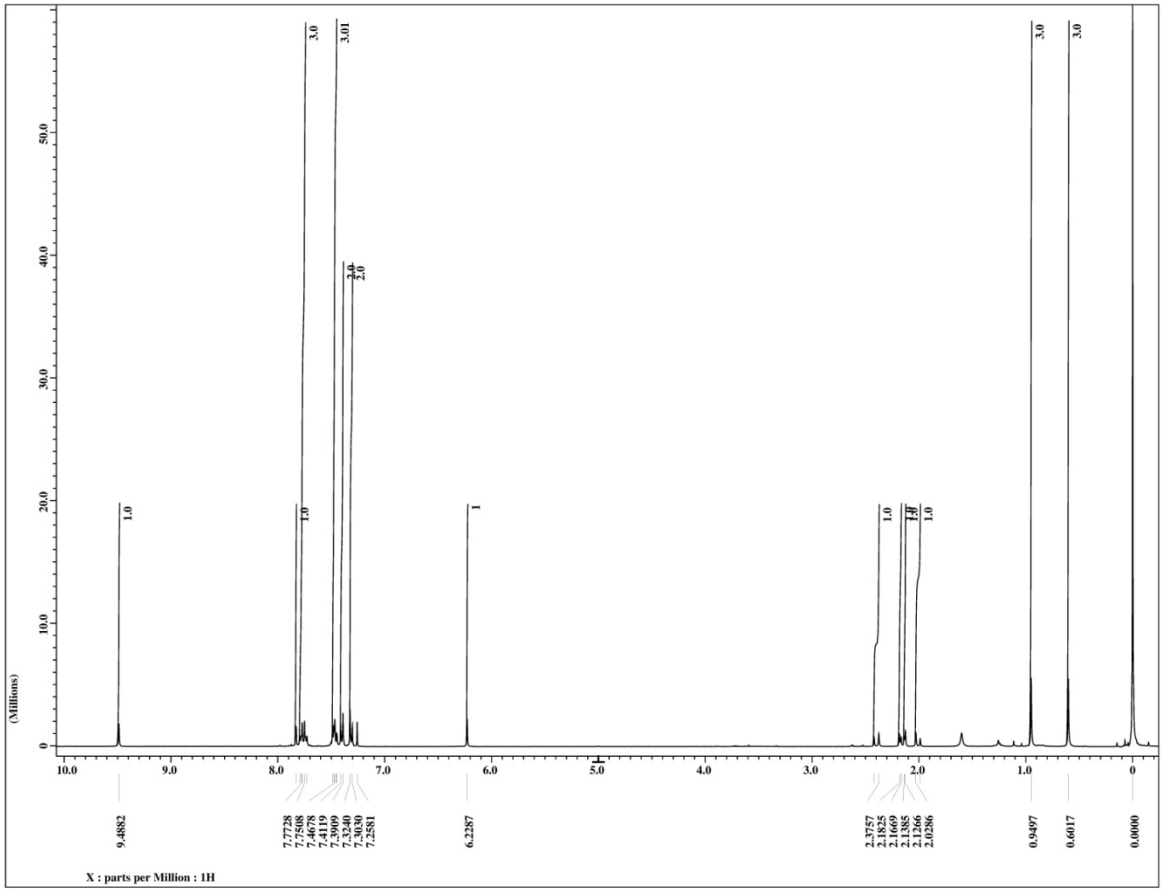

Br
O
SH
OH

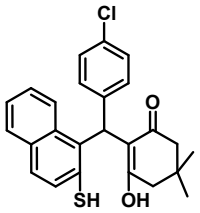
4i-$^{13}$C-NMR


(Millions)
X : parts per Million : 13C
196.8502
173.6419
131.9665
129.7016
129.6891
128.9077
127.6766
127.6001
127.4472
126.7360
126.5372
111.1211
77.3143
77.0008
76.6796
50.1603
45.6715
43.3622
31.6701
28.2137
27.4032
-0.0185

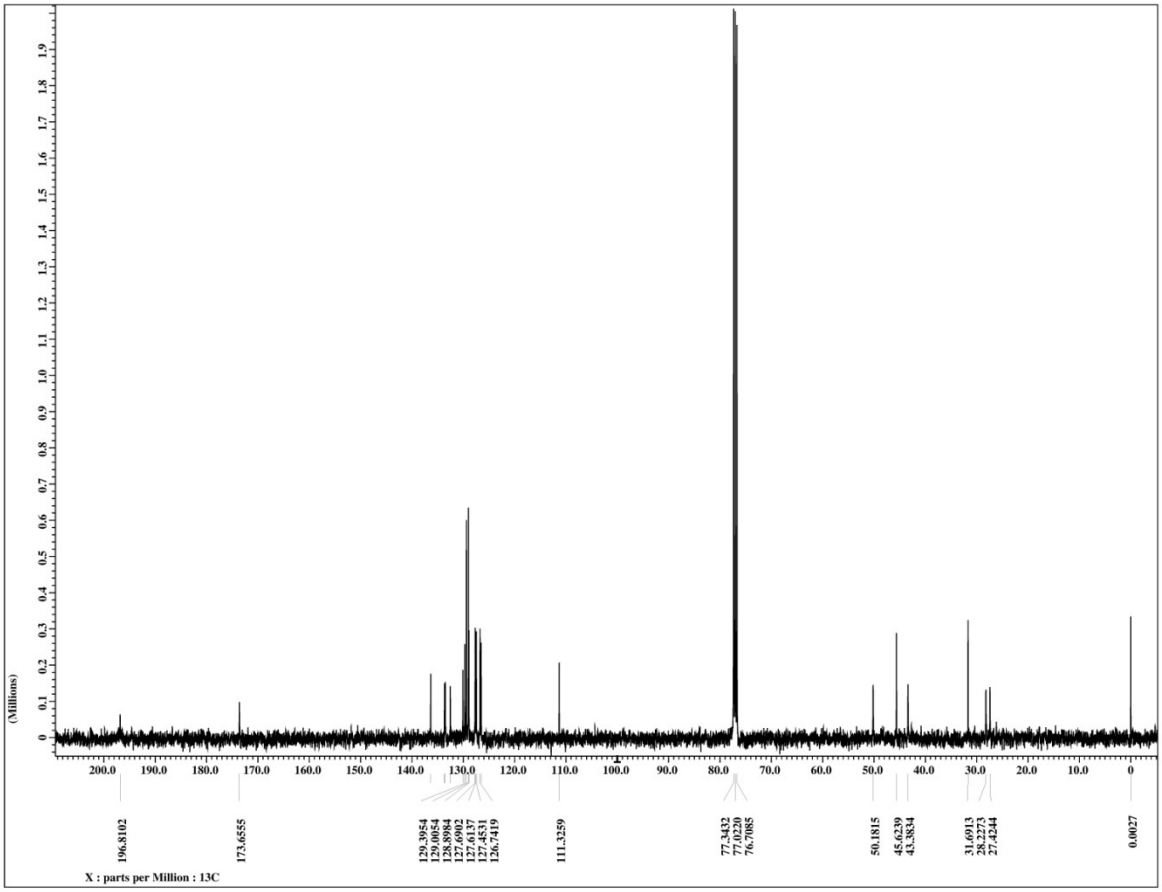

Cl
O
SH
OH

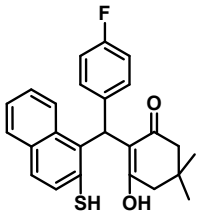
4j-$^{1}$H-NMR


(Millions)
X : parts per Million : 1H
9.4882
7.7728
7.7598
7.4678
7.4119
7.3909
7.3240
7.3030
7.2581
6.2287
2.3757
2.1825
2.1669
2.1385
2.1266
2.0286
0.9497
0.6017
0.0000

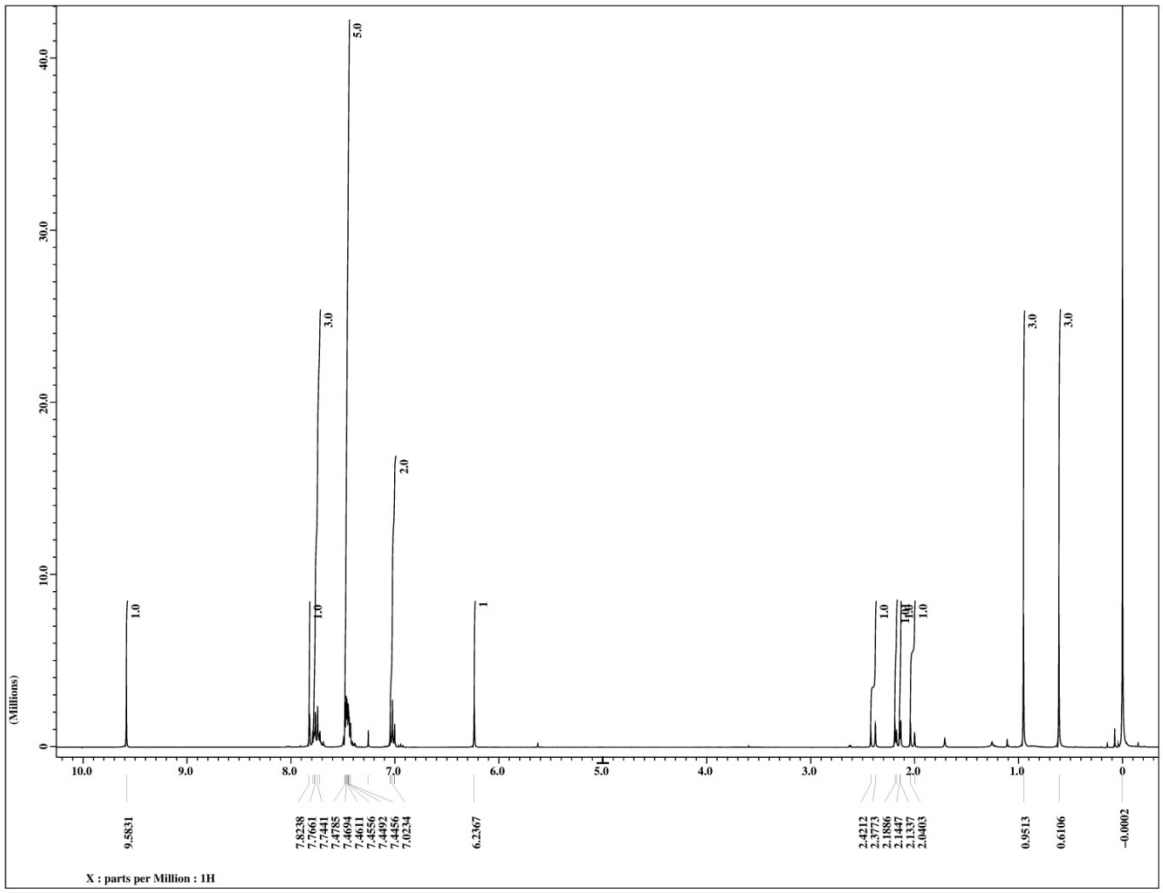

Cl
O
SH
OH

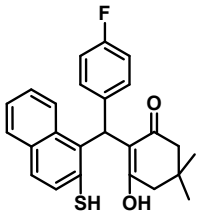
4j-$^{13}$C-NMR


(Millions)
X : parts per Million : 13C
196.8102
173.6555
129.3954
129.0054
128.8984
127.6902
127.6137
127.4531
126.7419
111.3259
77.3432
77.0220
76.7085
50.1815
45.6239
43.3834
31.6913
28.2273
27.4244
0.0027

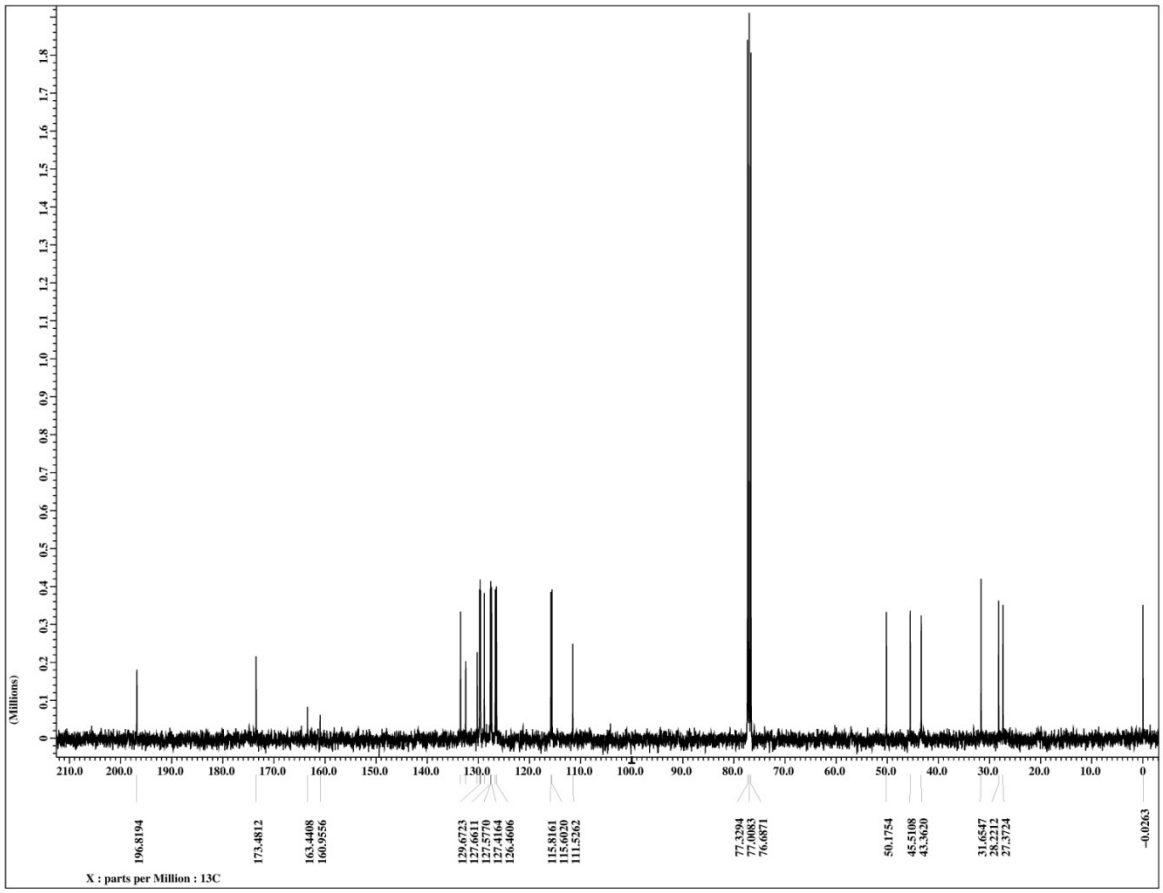

F
O
SH
OH

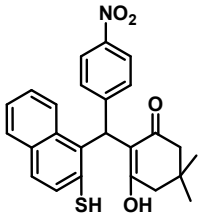
4k-$^{1}$H-NMR


(Millions)
40.0
30.0
20.0
10.0
0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
H
1.0
3.0
5.0
2.0
1
3.0
3.0
9.5831
7.8238
7.7661
7.7441
7.4785
7.4694
7.4611
7.4556
7.4493
7.4456
7.0234
6.2367
2.4212
2.3773
2.1936
2.1447
2.1337
2.0403
0.9513
0.6106
-0.0002
X : parts per Million : 1H

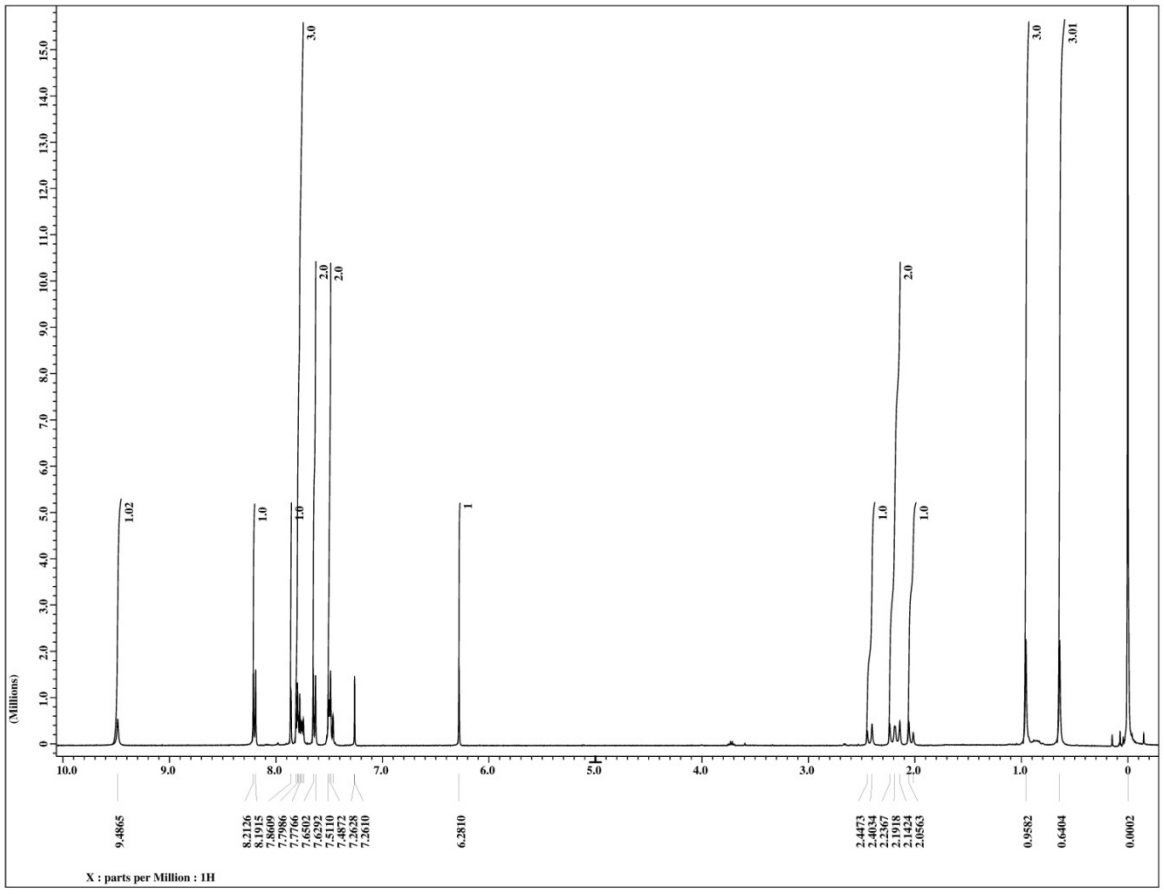

F
O
SH
OH

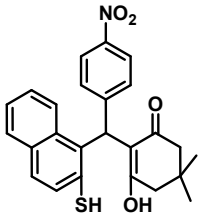
4k-$^{13}$C-NMR


(Millions)
X : parts per Million : 13C
196.8194
173.4812
163.4408
160.9556
129.6723
127.6611
127.5770
127.4164
126.4606
115.8161
115.6010
111.5262
77.3294
77.0083
76.6871
50.1754
45.5108
43.3620
31.6547
28.2212
27.3724
-0.0263

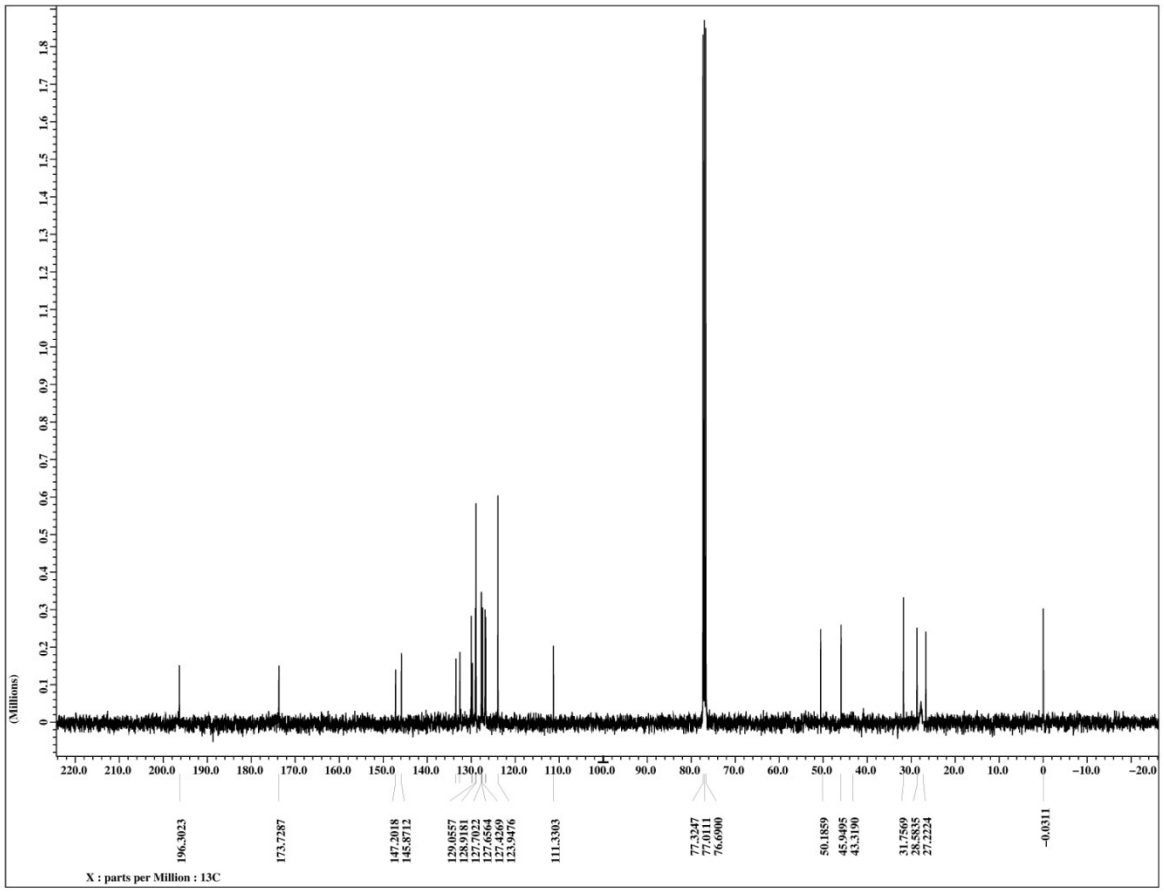

NO2
O
SH
OH

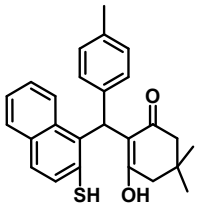
4l-$^{1}$H-NMR


(Millions)
X : parts per Million : 1H
9.4865
8.2126
8.1915
7.9680
7.7986
7.7766
7.6502
7.6292
7.5110
7.4872
7.2628
7.2610
6.2810
2.4473
2.4014
2.2367
2.1918
2.1424
2.0563
0.9582
0.6404
0.0002

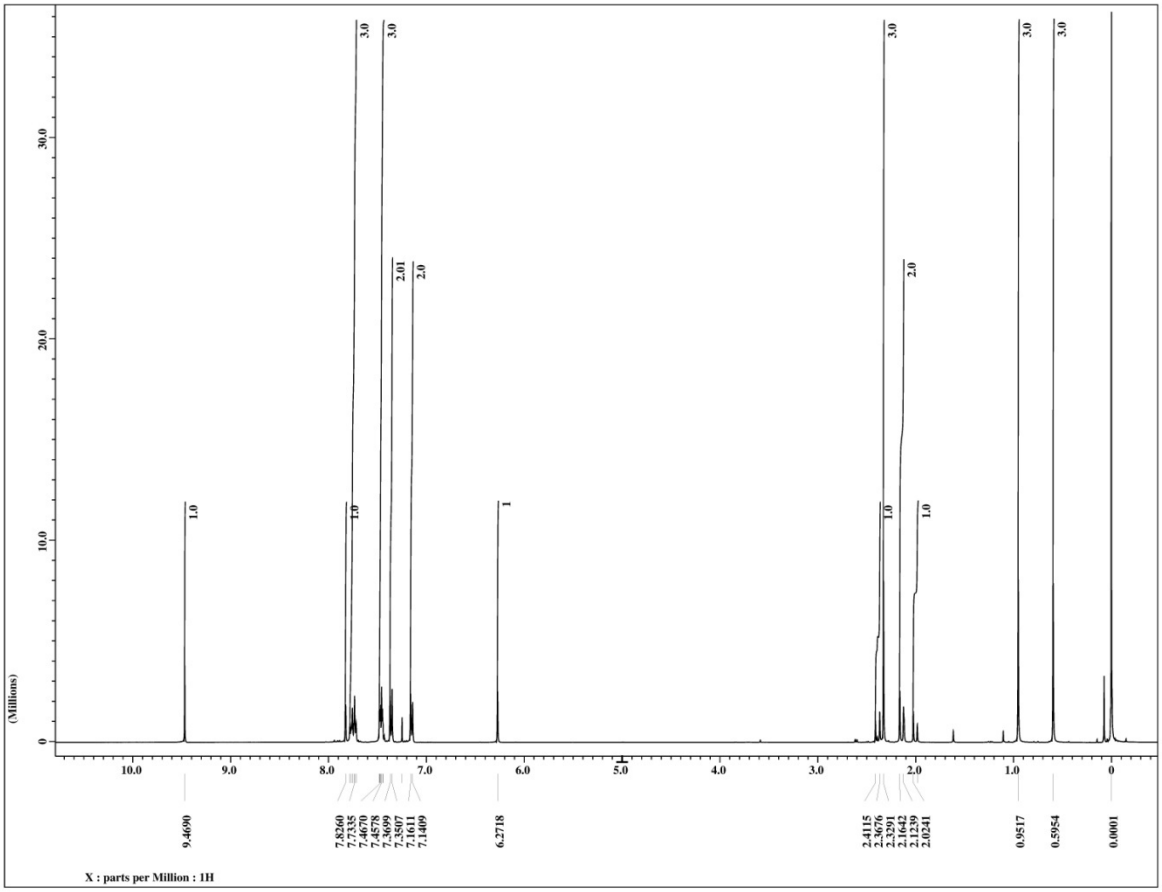

NO2
O
SH
OH

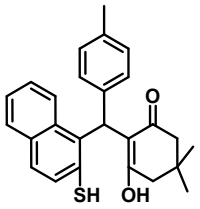
4l-$^{13}$C-NMR


(Millions)
X : parts per Million : 13C
196.3023
173.7287
147.2018
145.8712
129.0557
128.9181
127.7022
127.6564
127.4269
123.9476
111.3303
77.3247
77.0111
76.6900
50.1859
45.9495
43.3190
31.7569
28.5835
27.2224
-0.0311

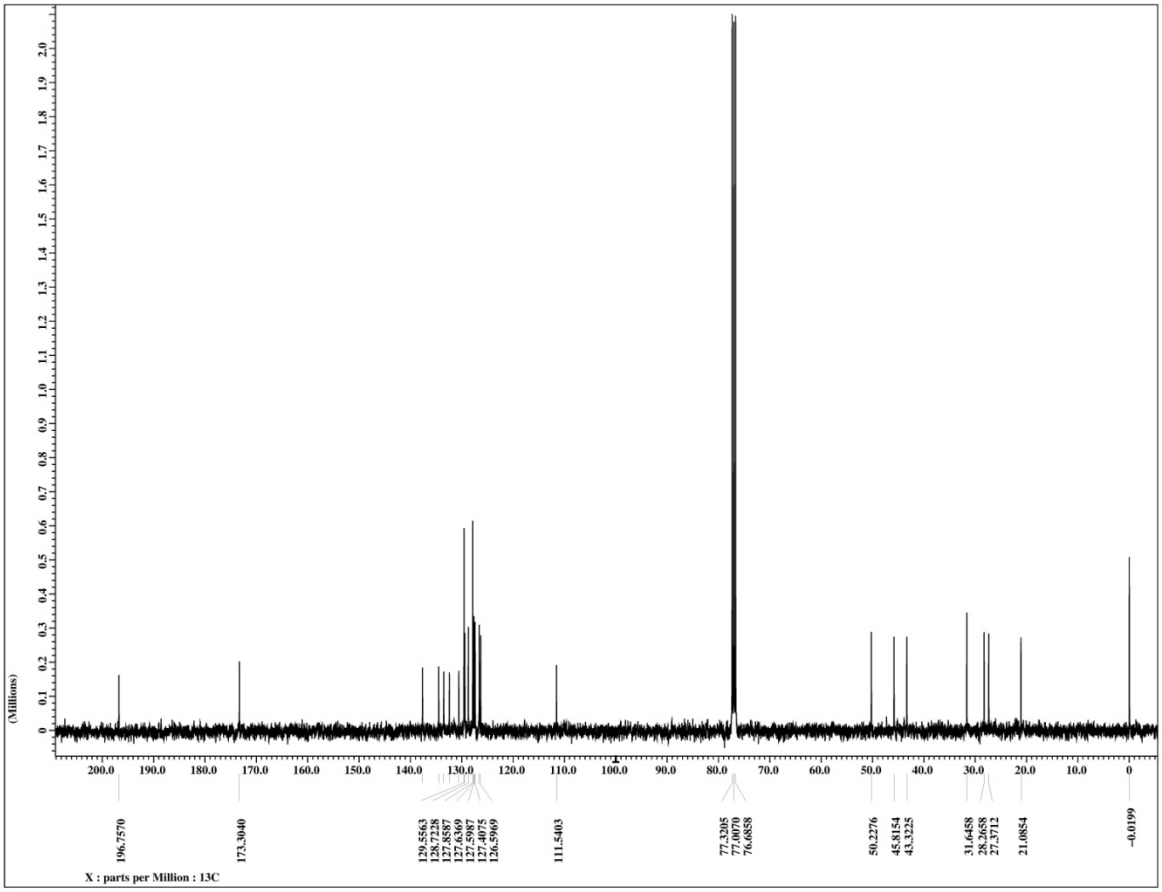

O
SH
OH

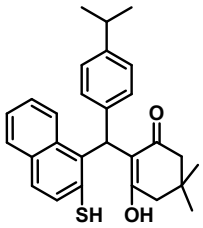
4m-$^{1}$H-NMR


(Millions)
30.0
20.0
10.0
0
3.0
3.0
2.01
2.0
1.0
1.0
1
1.0
3.0
2.0
1.0
3.0
3.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.4690
7.8260
7.7335
7.4670
7.4578
7.3699
7.3507
7.1611
7.1469
6.2718
2.4115
2.3676
2.3291
2.1642
2.1239
2.0241
0.9517
0.5954
0.0001
X : parts per Million : 1H

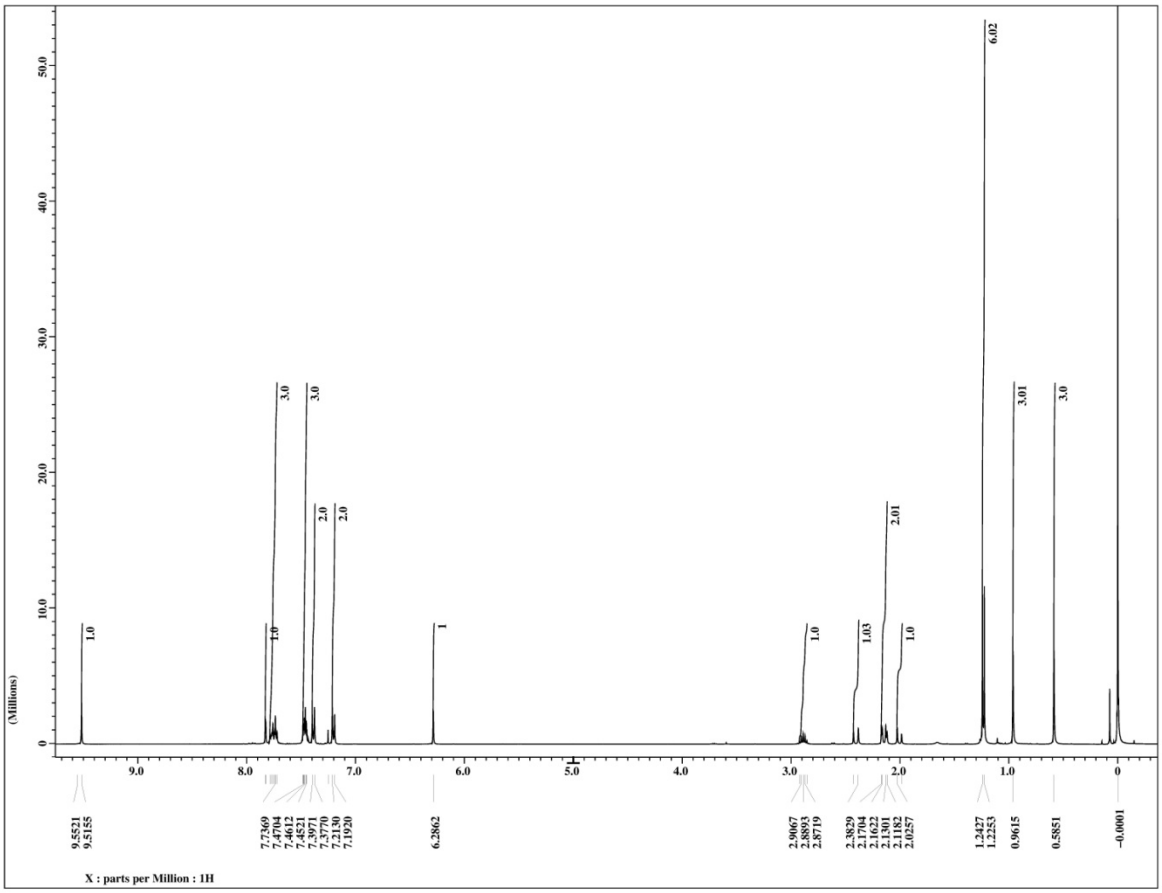

O
SH
OH

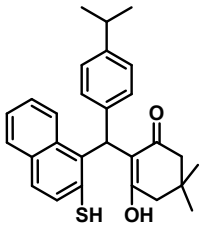
4m-$^{13}$C-NMR


(Millions)
X : parts per Million : 13C
196.7570
173.3040
129.5563
128.7228
127.8587
127.6369
127.5987
127.4075
126.5969
111.5403
77.3205
77.0070
76.6858
50.2276
45.8154
43.3225
31.6458
28.3658
27.3712
21.0854
-0.0199

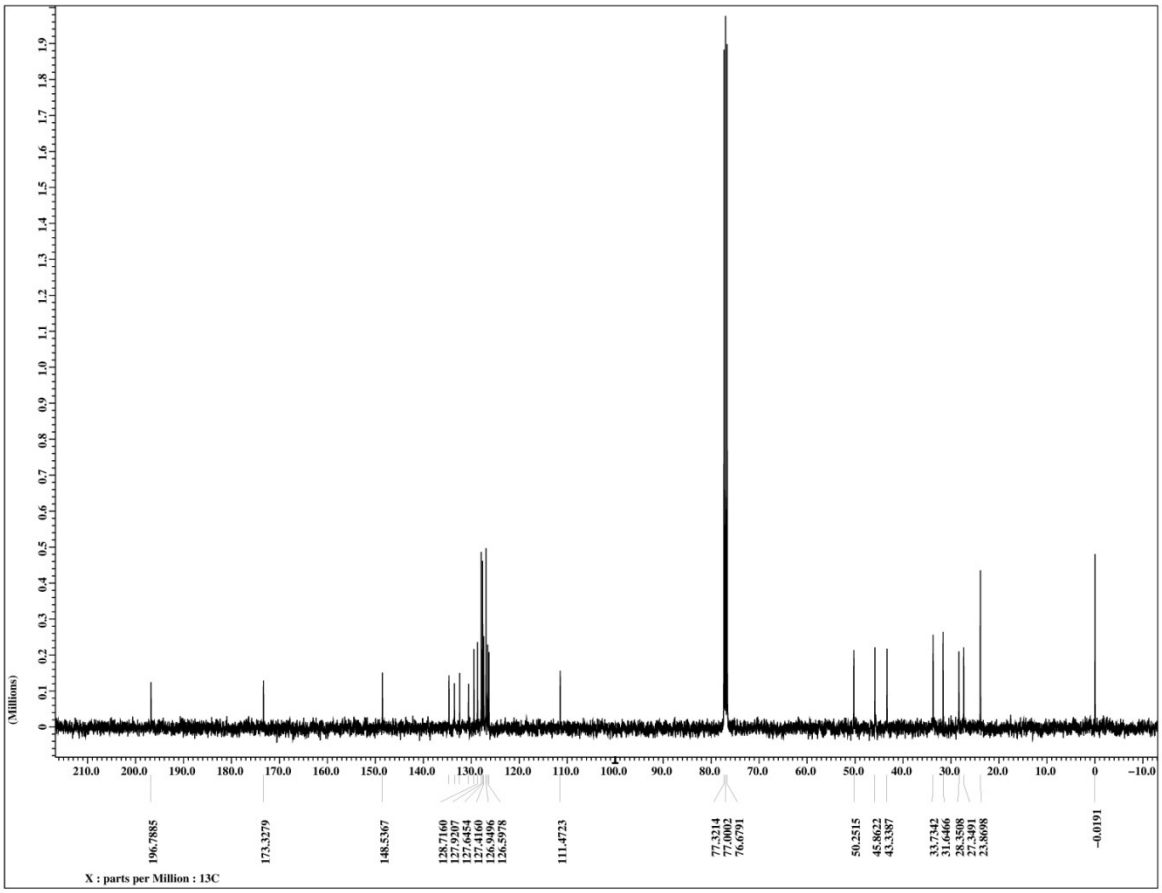

O
SH
OH

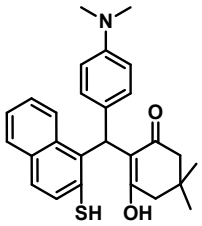
4n-$^{1}$H-NMR


(Millions)
50.0
40.0
30.0
20.0
10.0
0
1.0
3.0
3.0
2.0
2.0
1.0
1
1.0
1.03
2.01
1.0
6.02
3.01
3.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.5521
9.5155
7.7369
7.4704
7.4612
7.4521
7.3977
7.3770
7.2130
7.1920
6.2862
2.9067
2.8893
2.8719
2.3829
2.1704
2.1622
2.1301
2.1182
2.0257
1.2427
1.2253
0.9615
0.5851
−0.0001
X : parts per Million : 1H

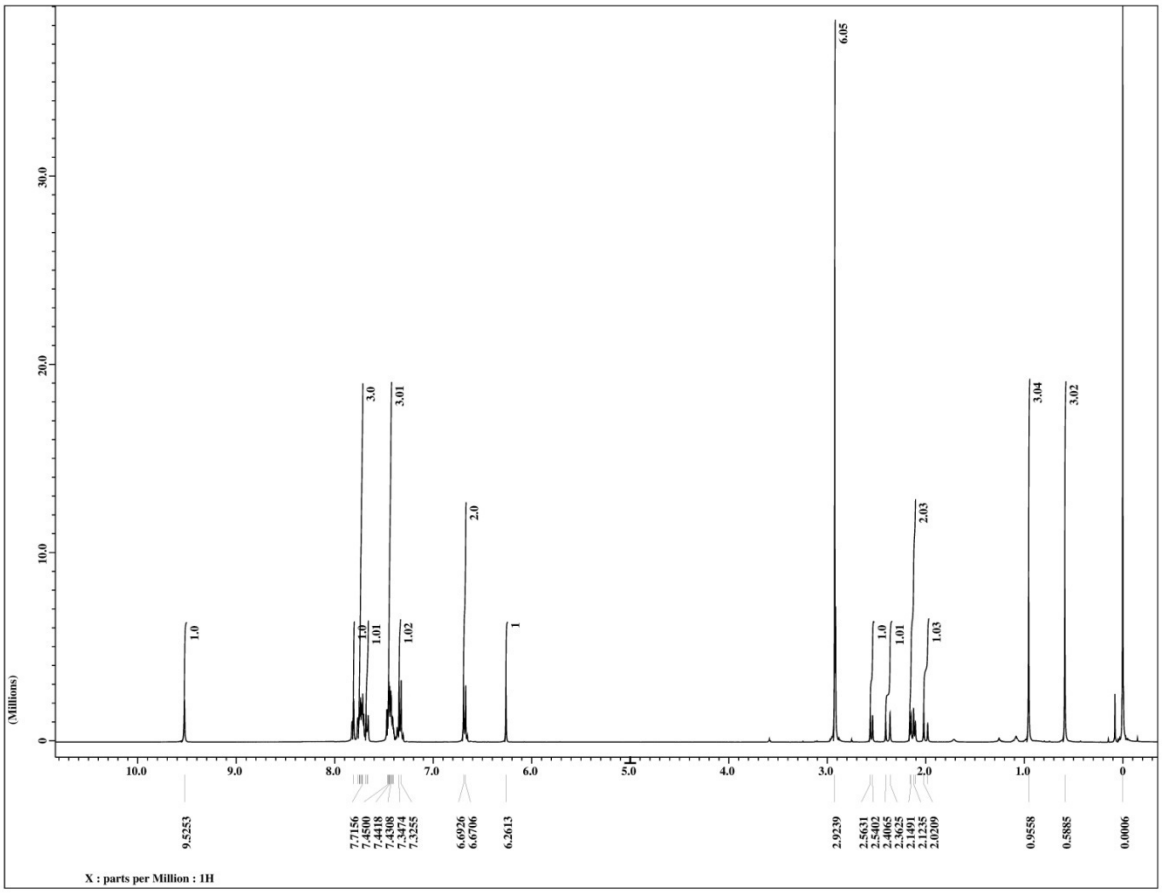

O
SH
OH

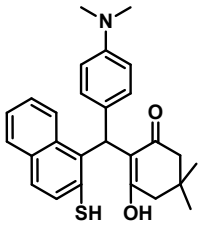
4n-$^{13}$C-NMR


(Millions)
X : parts per Million : 13C
196.7885
173.3279
148.5367
128.7160
127.9207
127.6484
127.4160
126.9496
126.5978
111.4723
77.3214
77.0002
76.6791
50.2515
45.8622
43.3387
33.7342
31.6466
28.3508
27.3491
23.8698
-0.0191

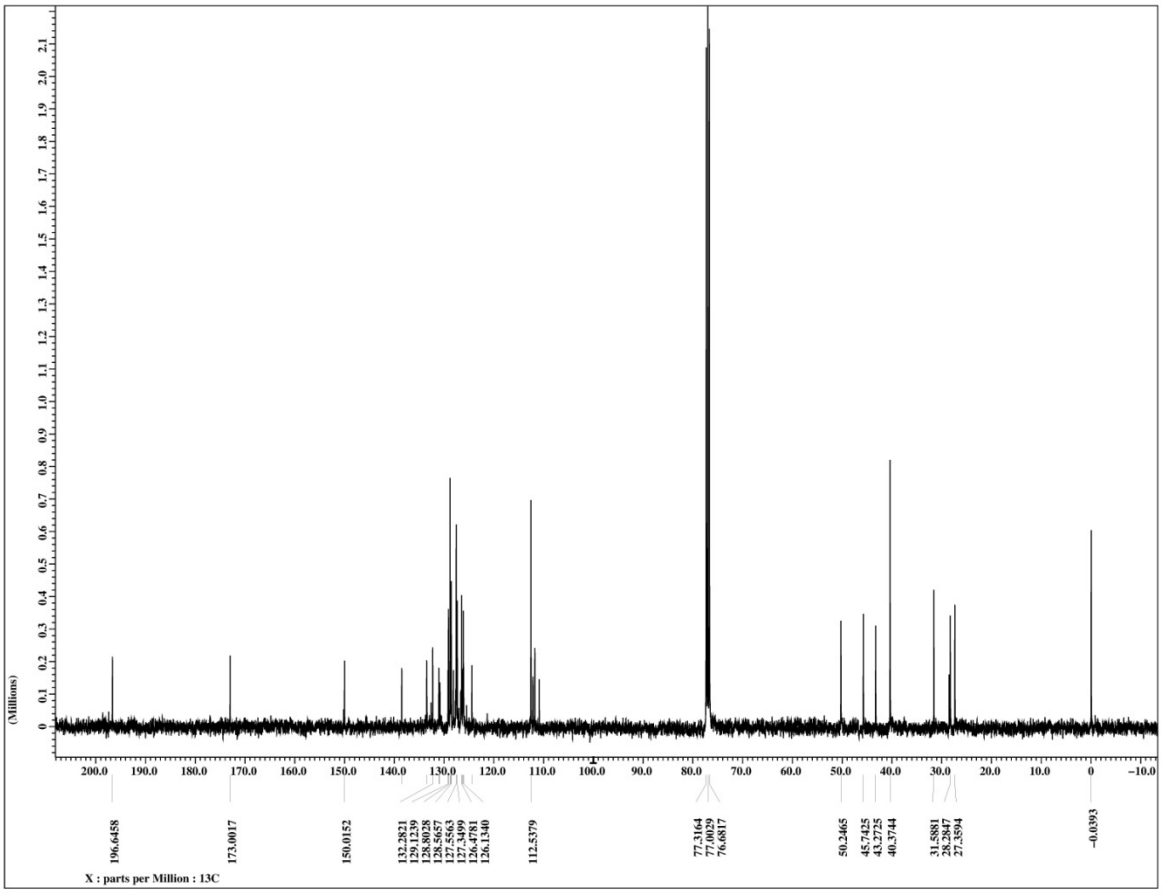

N
O
SH
OH

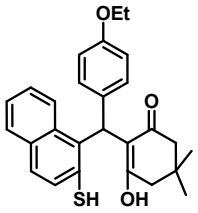
4o-$^{1}$H-NMR


(Millions)
30.0
20.0
10.0
0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
1.0
3.0
1.0
1.01
3.01
1.02
2.0
1
6.05
1.0
1.01
2.03
1.03
3.04
3.02
9.5253
7.7156
7.4500
7.4418
7.4308
7.3474
7.3255
6.6926
6.6706
6.2613
2.9239
2.5631
2.5402
2.4065
2.3825
2.1491
2.1235
2.0209
0.9558
0.5885
0.0006
X : parts per Million : 1H

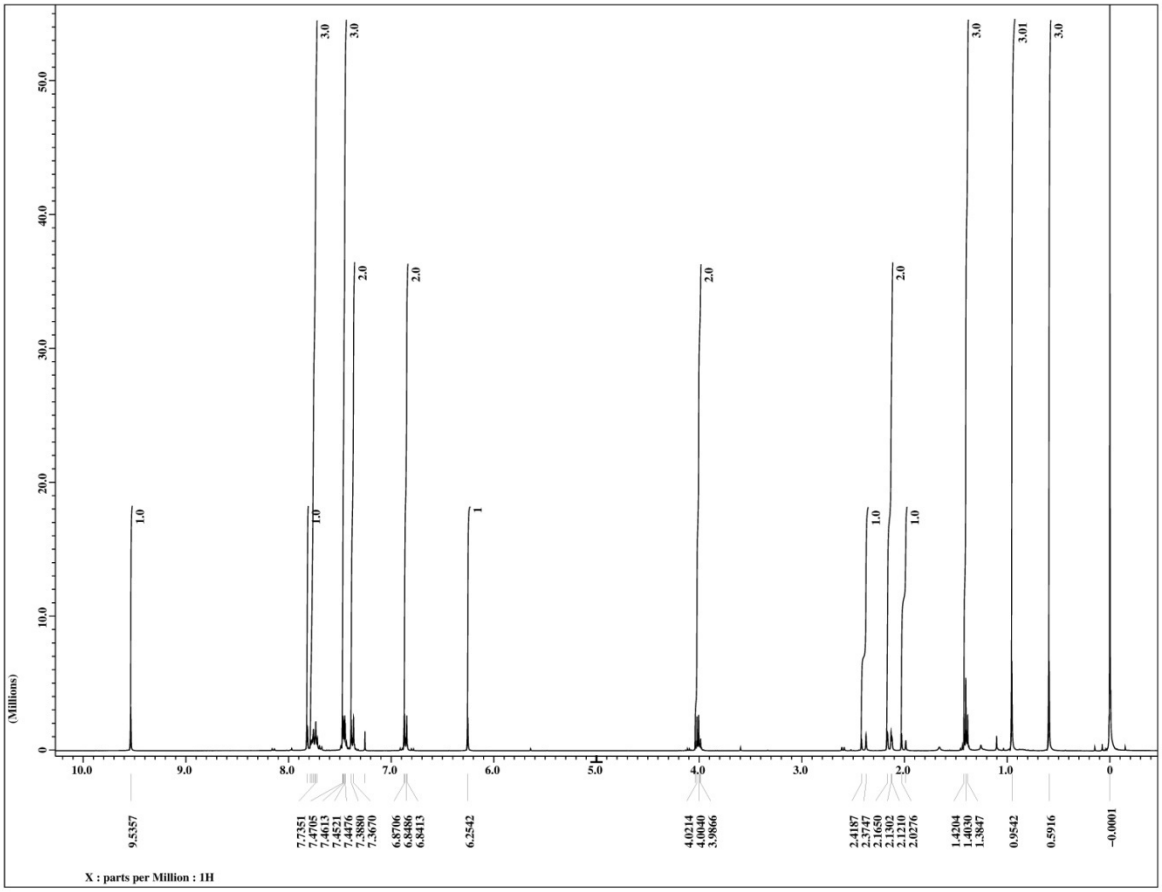

N
O
SH
OH

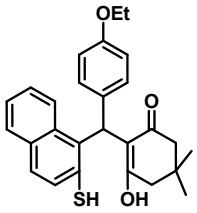
4o-$^{13}$C-NMR


(Millions)
X : parts per Million : 13C
196.6458
173.0017
150.0152
132.2821
129.1239
128.8028
128.4657
127.5563
127.3499
126.4781
126.1340
112.5379
77.3164
77.0029
76.6817
50.2465
45.7425
43.2725
40.3744
31.5881
28.2847
27.3594
-0.0393

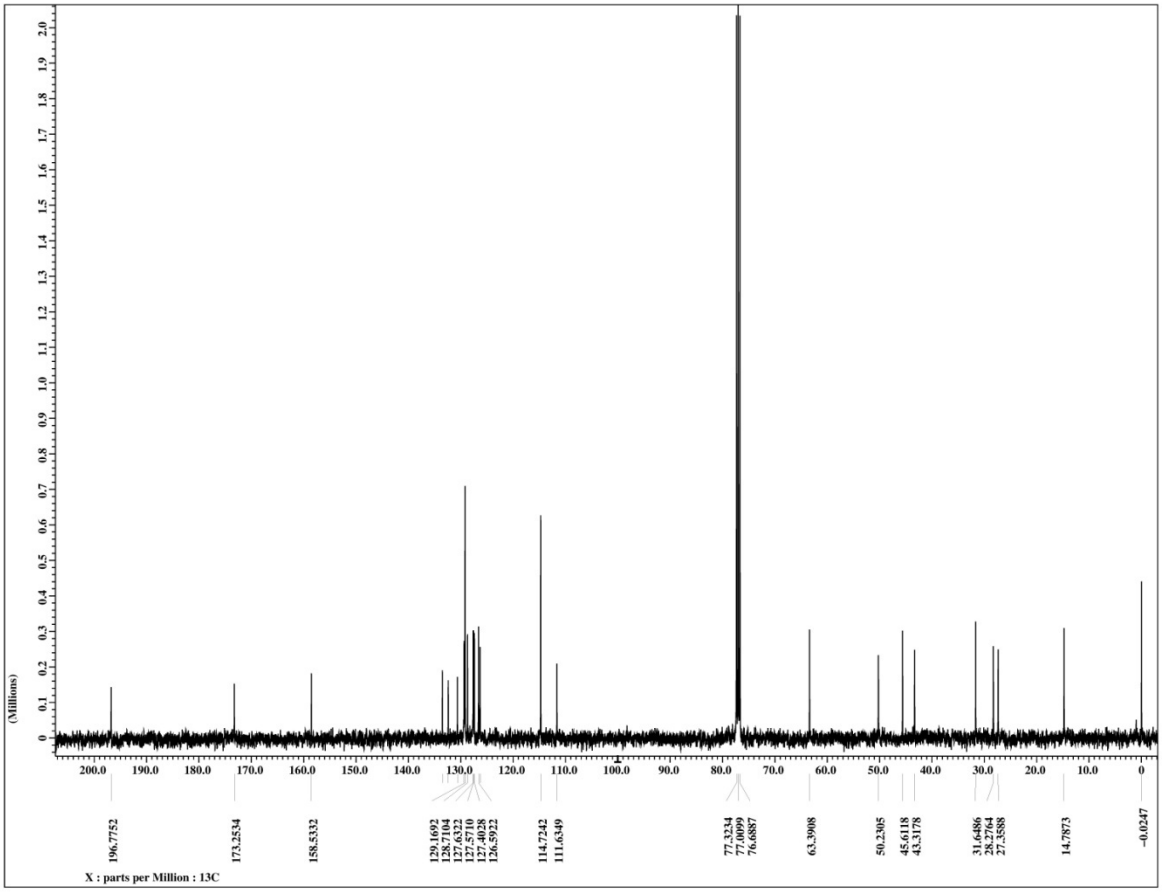

OEt
O
SH
OH

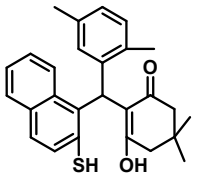
4p-$^{1}$H-NMR


(Millions)
X : parts per Million : 1H
9.5357
7.7351
7.4705
7.4613
7.4521
7.4476
7.3880
7.3670
6.8706
6.8486
6.8413
6.2542
4.0214
4.0040
3.9866
2.4187
2.3747
2.1650
2.1302
2.1210
2.0276
1.4204
1.4030
1.3847
0.9542
0.5916
-0.0001

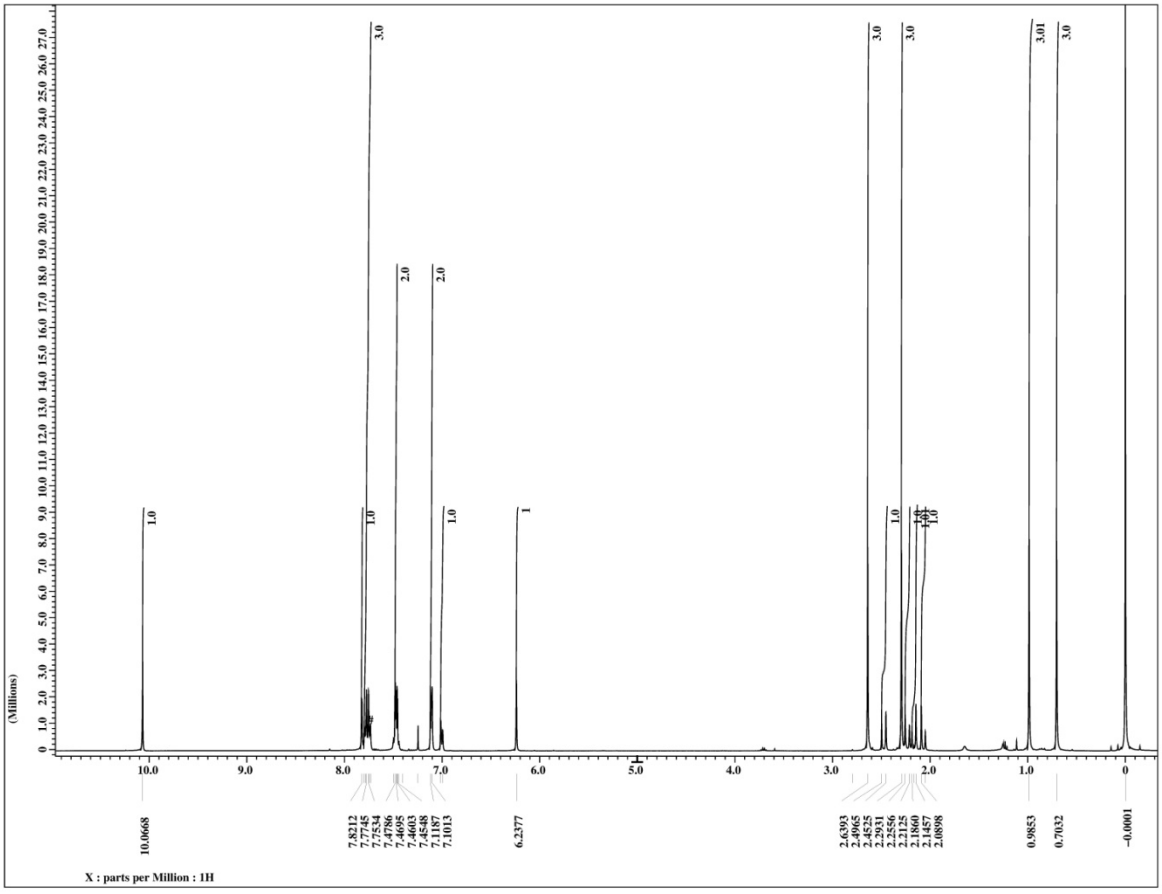

OEt
O
SH
OH

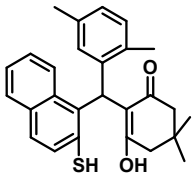
4p-$^{13}$C-NMR


(Millions)
X : parts per Million : 13C
196.7752
173.2534
158.5332
129.1692
128.7104
127.6322
127.5710
127.4028
126.5922
114.7342
111.6349
77.3234
77.0099
76.6887
63.3908
50.2305
45.6118
43.3178
31.6486
28.2764
27.3588
14.7873
-0.0247

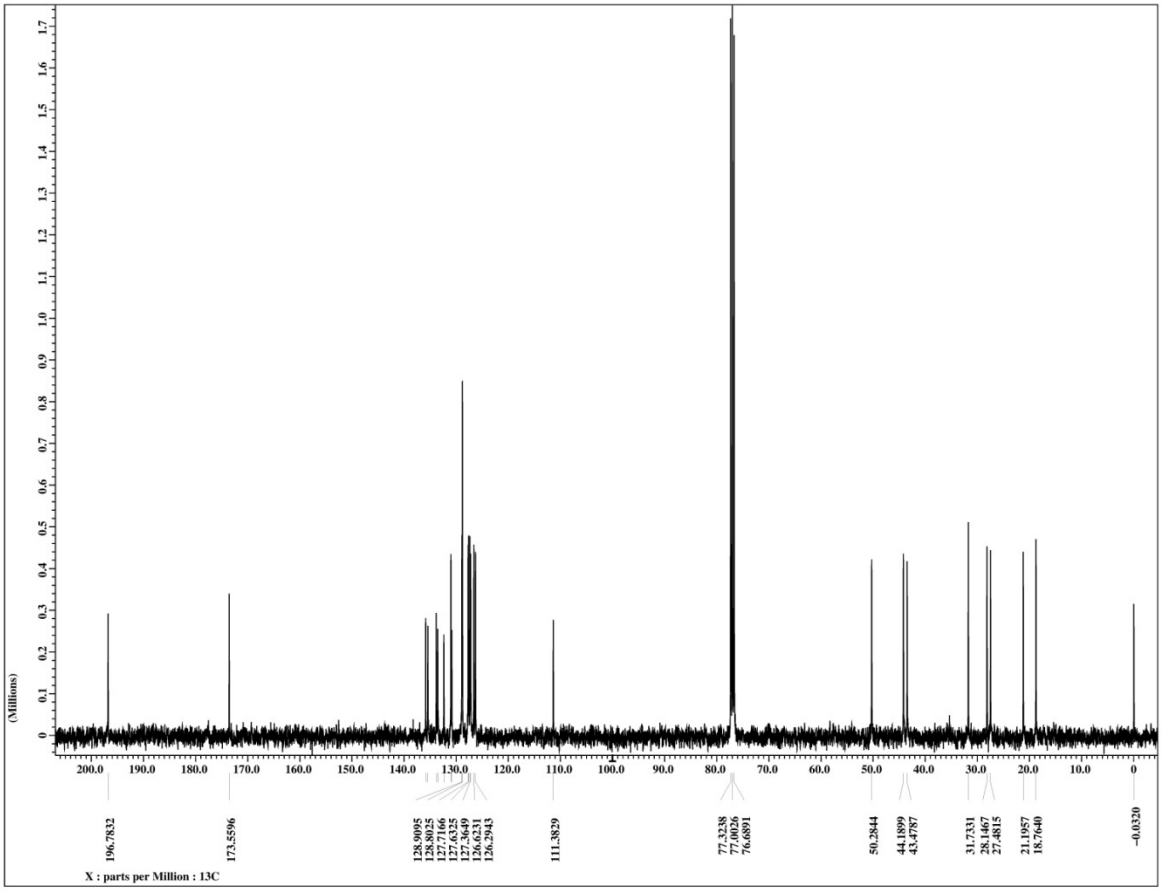

O
SH
OH

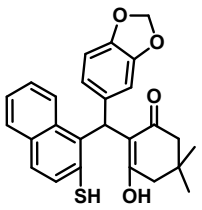
4q-$^{1}$H-NMR


(Millions)
10.0668
7.8212
7.7745
7.7534
7.4786
7.4695
7.4603
7.4548
7.1187
7.1013
6.2377
2.6393
2.4965
2.4525
2.2931
2.2556
2.2125
2.1860
2.1457
2.0898
0.9853
0.7032
-0.0001
X : parts per Million : 1H

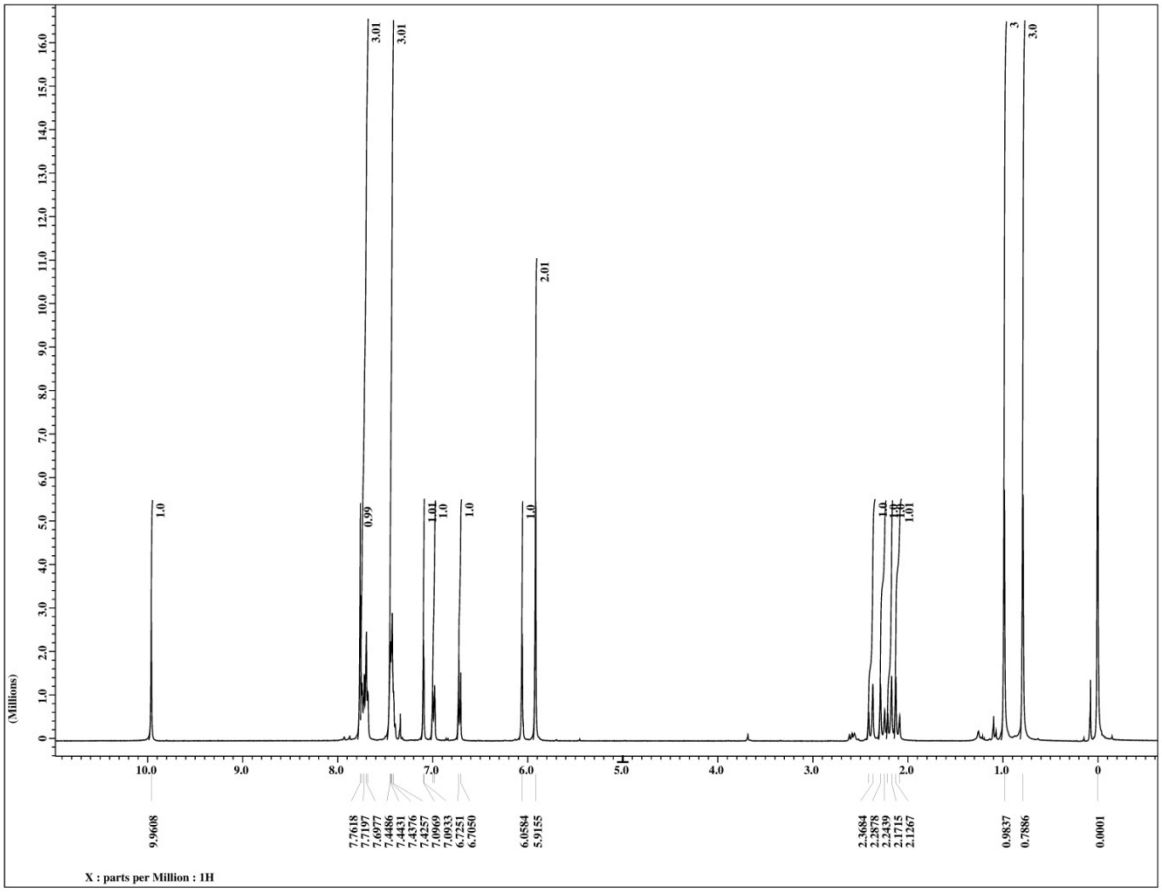

O
SH
OH

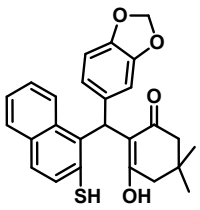
4q-$^{13}$C-NMR


(Millions)
X : parts per Million : 13C
196.7832
173.5596
128.9095
128.8025
127.7166
127.6325
127.3649
126.6231
126.2943
111.3829
77.3238
77.0026
76.6891
50.2844
44.1899
43.4787
31.7331
29.1467
27.4815
21.1957
18.7640
-0.0320

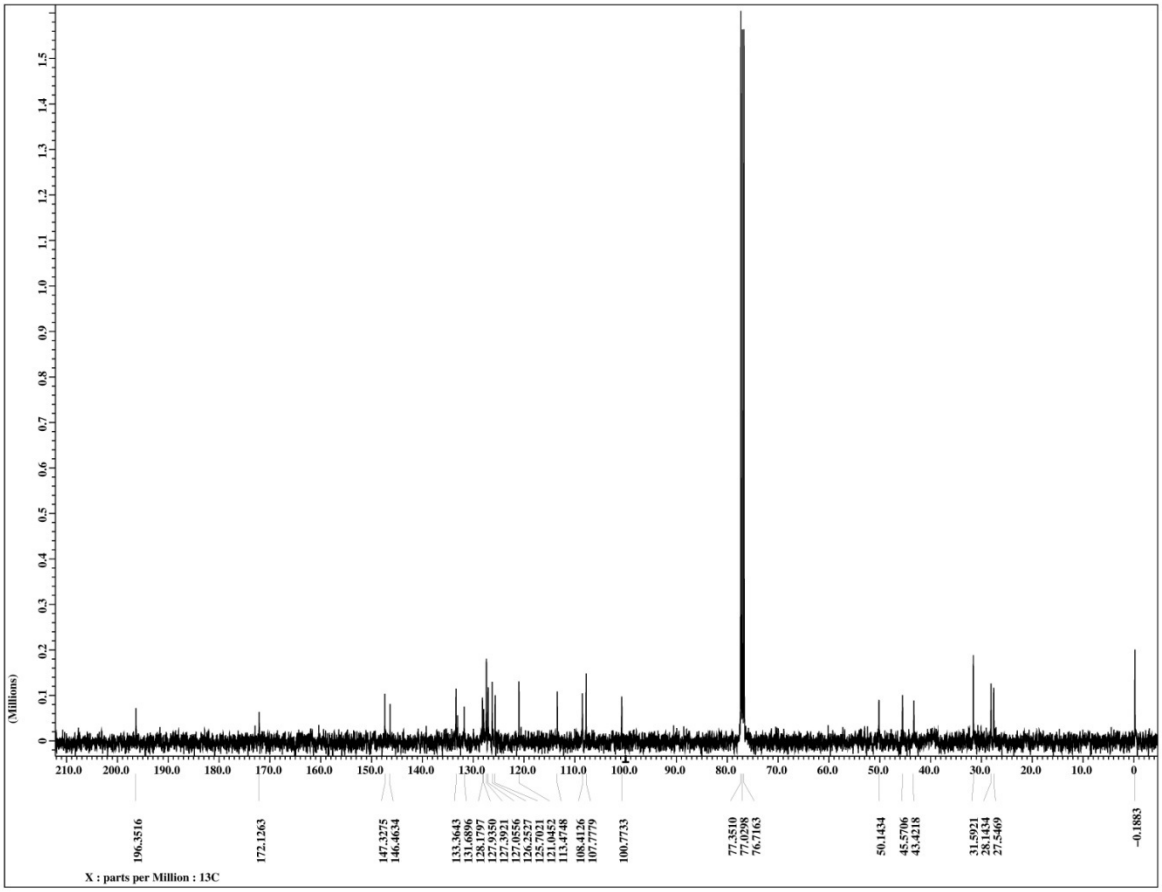



4r-$^{1}$H-NMR




O
O
O
SH
OH

4r-$^{13}$C-NMR


(Millions)
X : parts per Million : 13C
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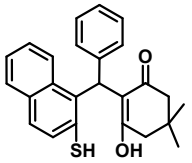
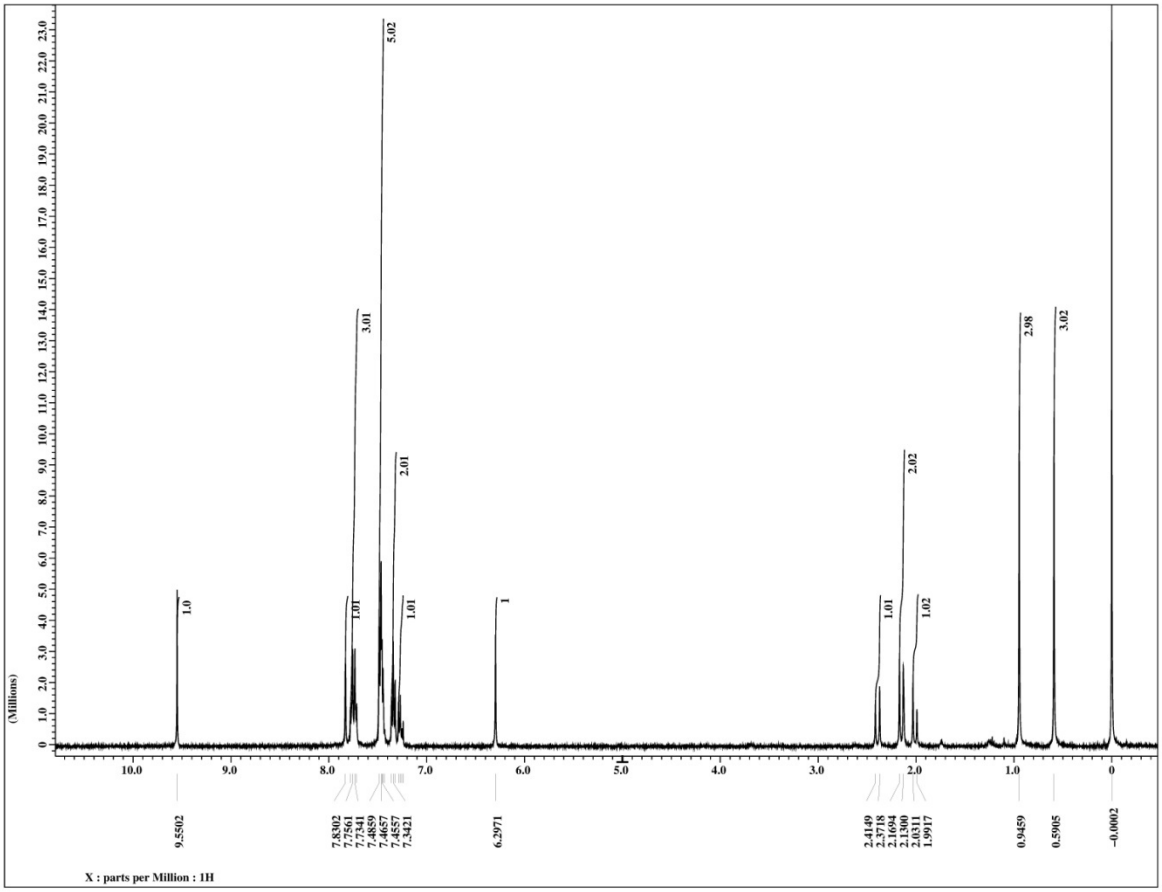
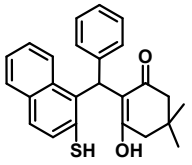
172.1263
147.3275
146.4634
133.3643
131.6896
129.7097
129.3350
127.3921
127.0556
126.2527
125.7021
123.0452
113.4748
108.4126
107.7779
100.7733
77.3510
77.0298
76.7163
50.1434
45.5706
43.4218
31.5921
28.1434
27.5469
-0.1883

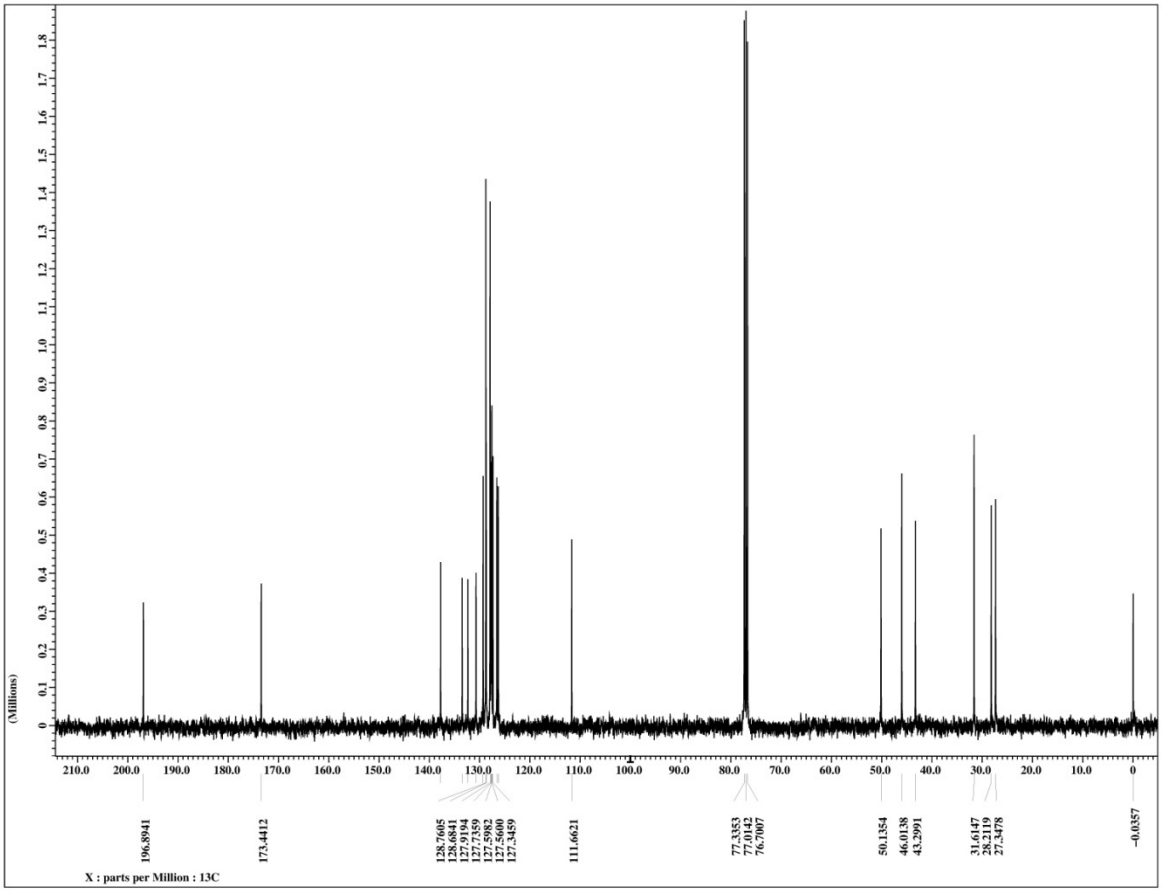

O
SH
OH

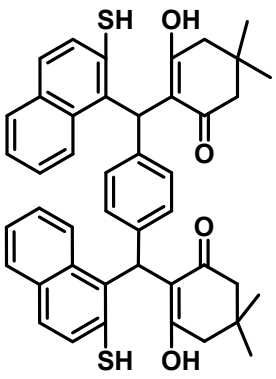
4s-$^{1}$H-NMR


(Millions)
1.0
1.01
3.01
5.02
2.01
1.01
1
1.01
2.02
1.02
2.98
3.02
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.5502
7.8302
7.7561
7.7341
7.4859
7.4657
7.4557
7.3421
6.2971
2.4149
2.3718
2.1694
2.1300
2.0311
1.9917
0.9459
0.5905
-0.0002
X : parts per Million : 1H

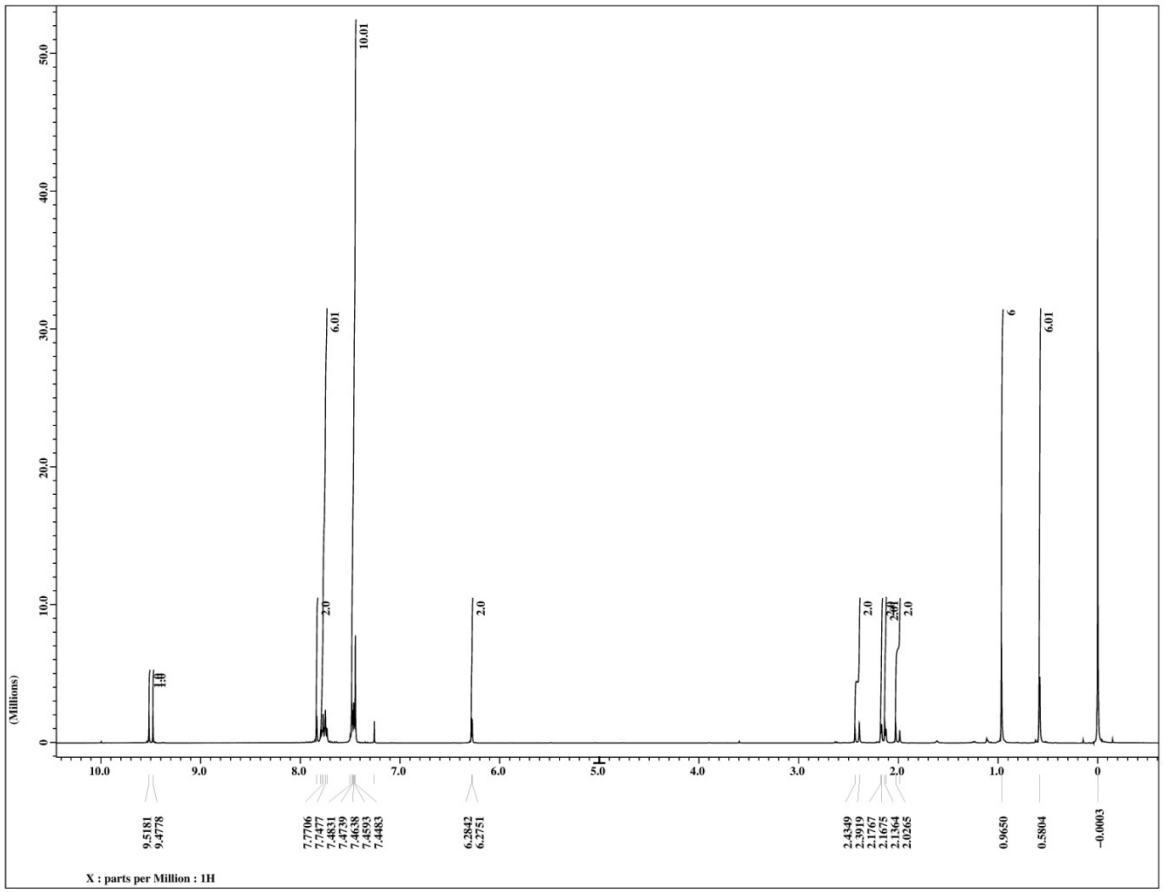

O
SH
OH

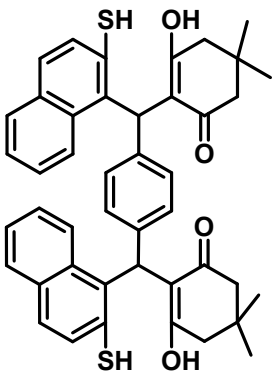
4s-$^{13}$C-NMR


(Millions)
X : parts per Million : 13C
196.8941
173.4412
128.7605
128.6641
128.5194
127.7359
127.5982
127.5600
127.3459
111.6621
77.3353
77.0142
76.7007
50.1354
46.0138
43.2991
31.6147
28.2119
27.3478
-0.0357

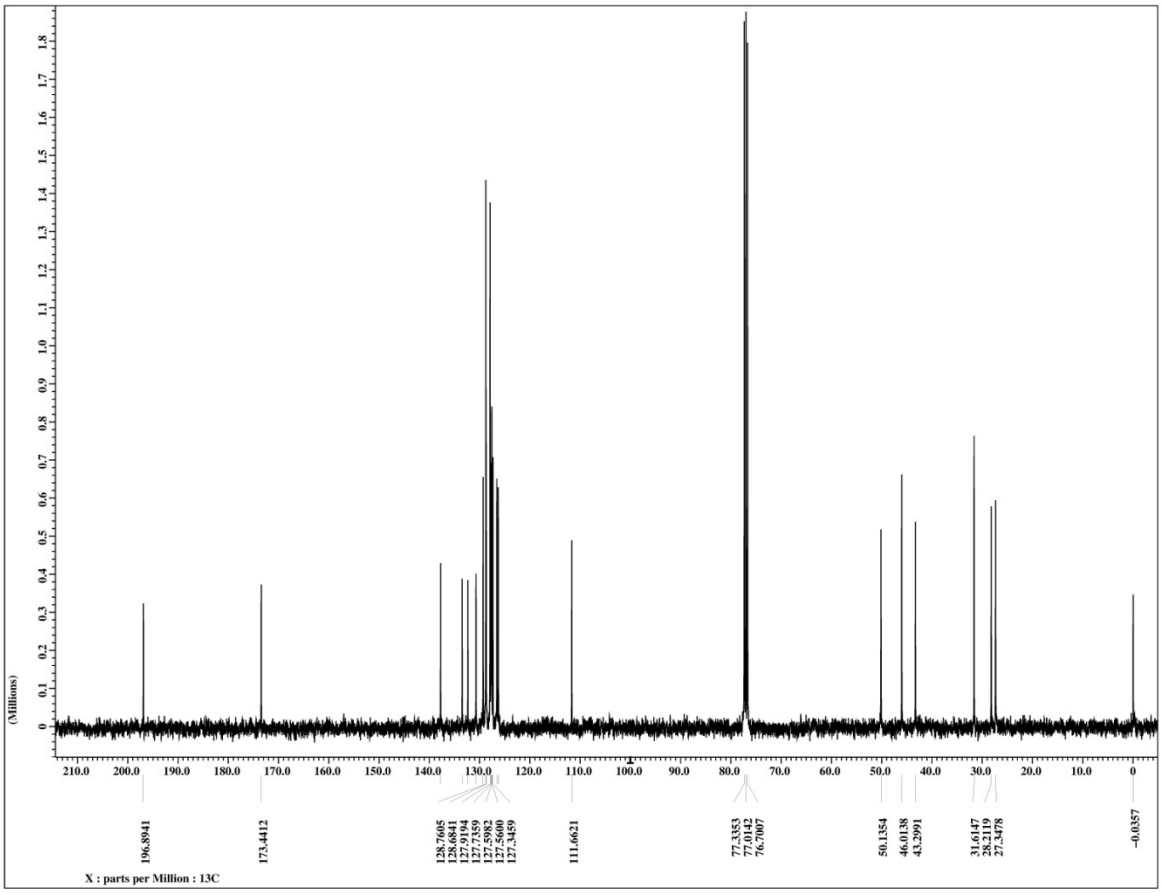

SH
OH
O
O
SH
OH

4t-$^{1}$H-NMR


(Millions)
50.0
40.0
30.0
20.0
10.0
0
10.01
6.01
2.0
1.0
6
6.01
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.5181
9.4778
7.7706
7.7477
7.4831
7.4739
7.4638
7.4593
7.4483
6.2842
6.2751
2.4349
2.3919
2.1767
2.1675
2.1364
2.0265
0.9650
0.5804
−0.0003
X : parts per Million : 1H


SH
OH
O
O
SH
OH

4t-$^{13}$C-NMR


(Millions)
X : parts per Million : 13C
196.8941
173.4412
128.7605
128.6641
128.3194
127.7359
127.5982
127.5600
127.3459
111.6621
77.3353
77.0142
76.7007
50.1354
46.0138
43.2991
31.6147
28.2119
27.3478
-0.0357

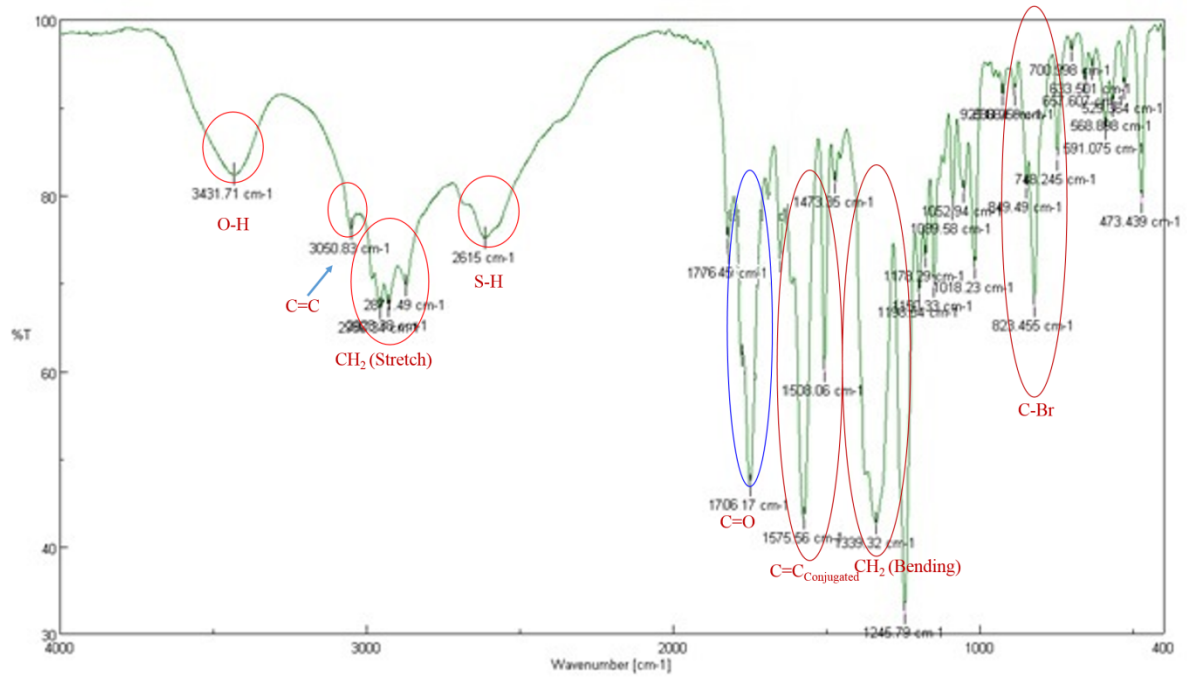



**Figure 4**: FT-IR spectrum of compound **4a**

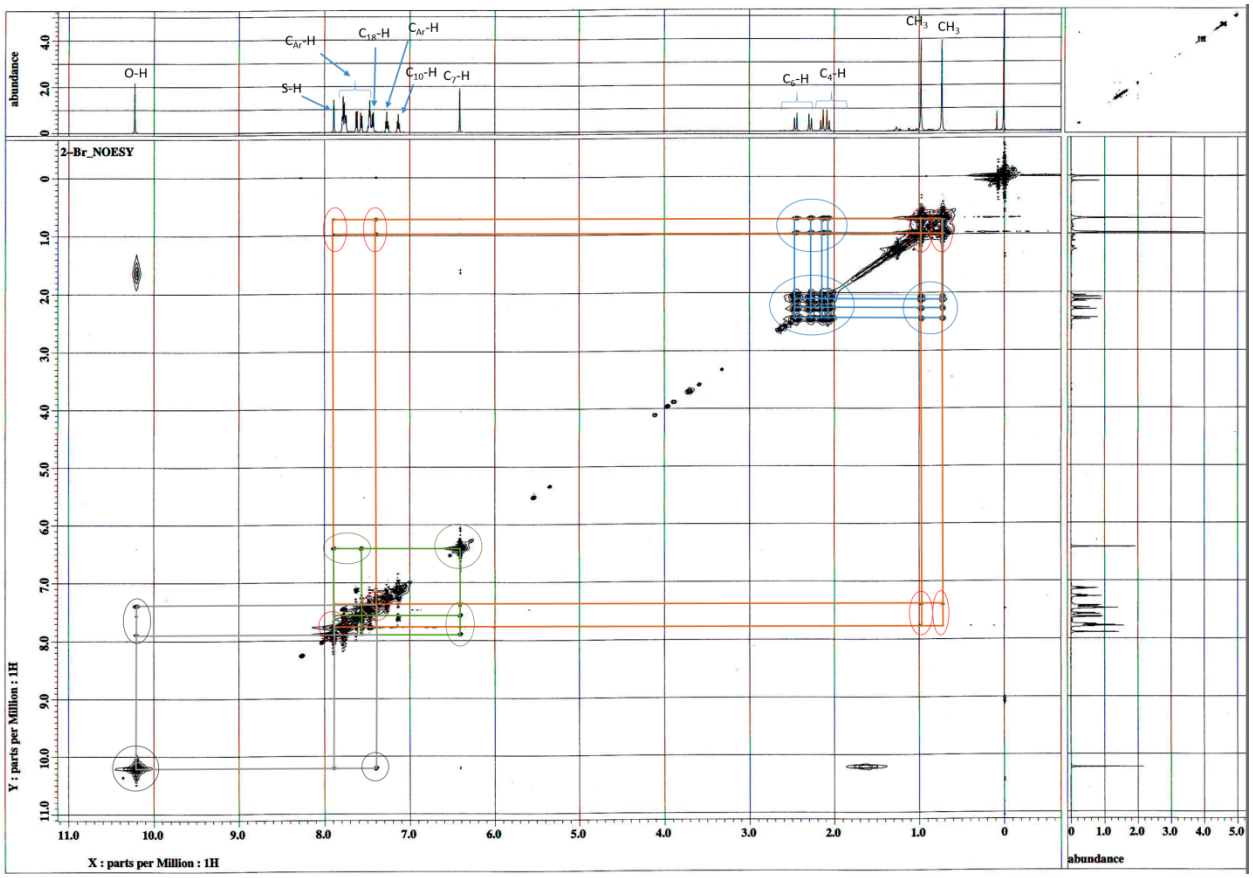



**Figure 5**: 2D-$^{1}$H-$^{1}$H NOESY spectrum of compound **4a** in DMSO-$d_6$.

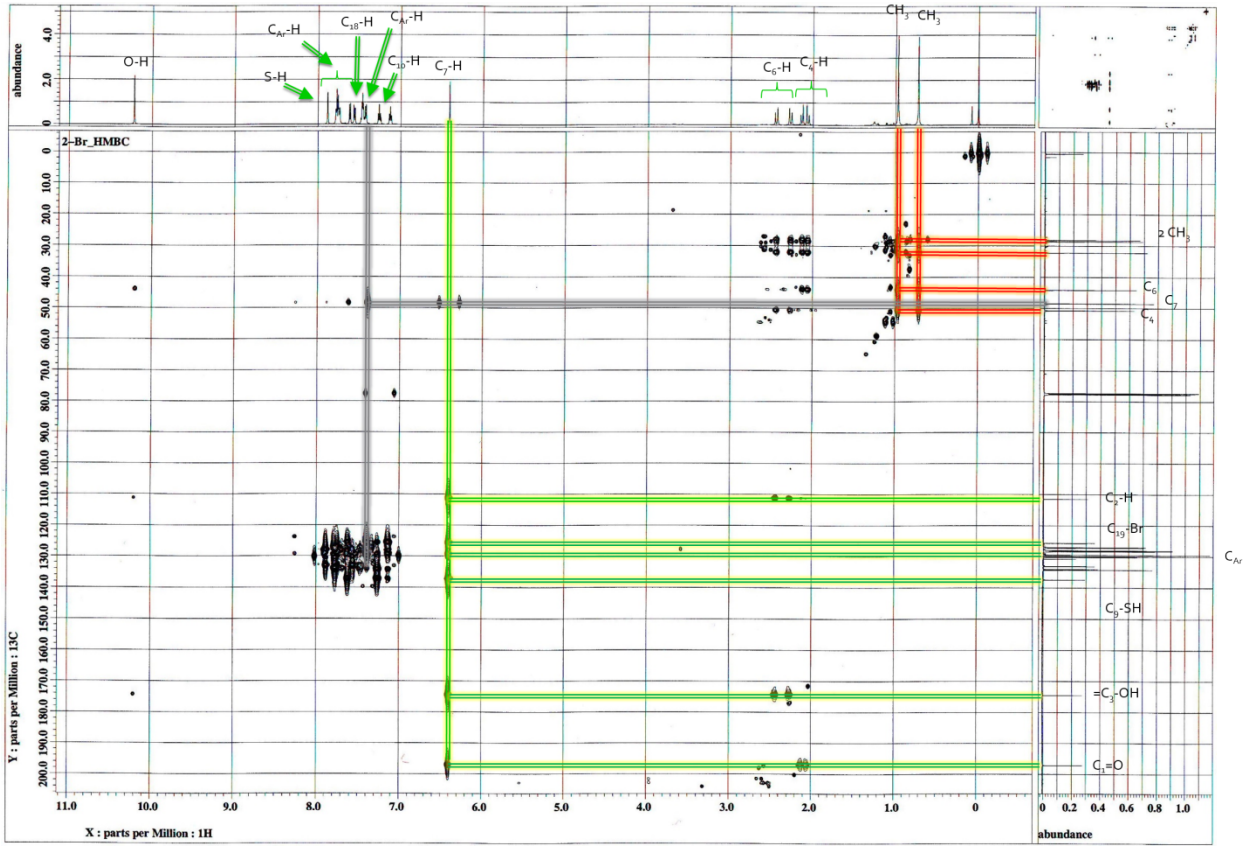



**Figure 6**: 2D-$^{1}$H-$^{13}$C HMQC spectrum of compound **4a** in DMSO-$d_6$.

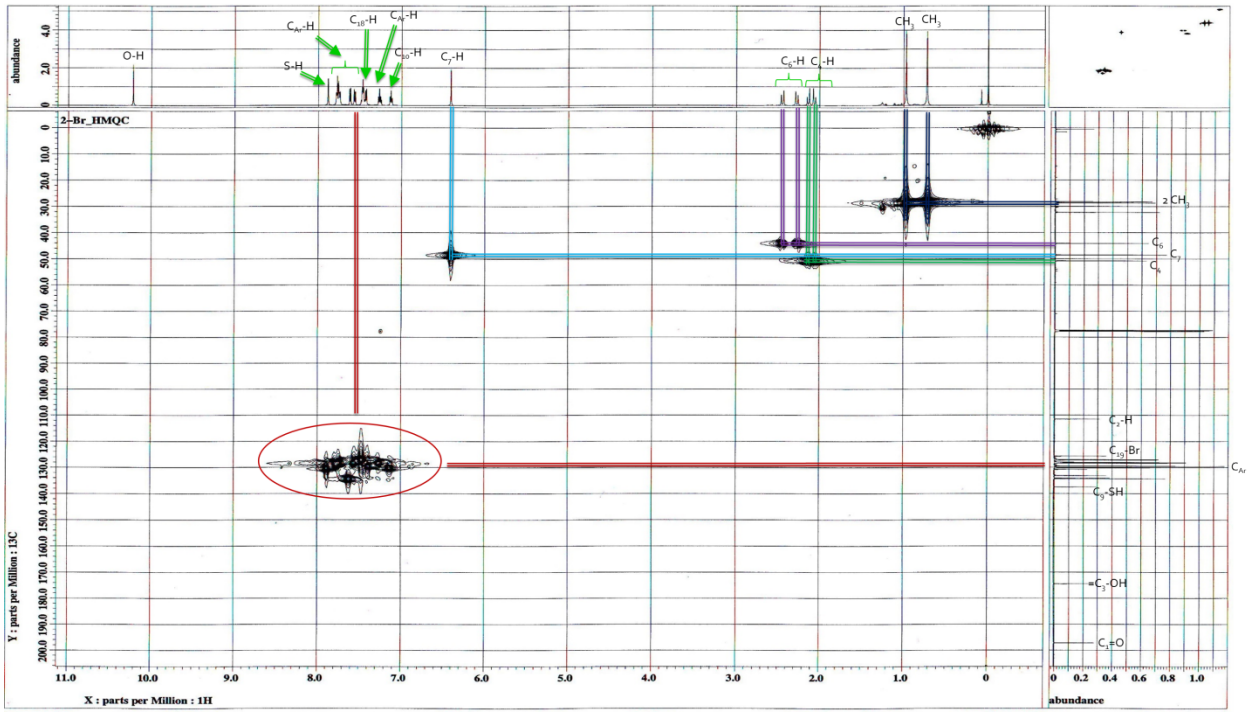



**Figure 7**: 2D-$^{1}$H-$^{13}$C HMBC spectrum of compound **4a** in DMSO-$d_6$.

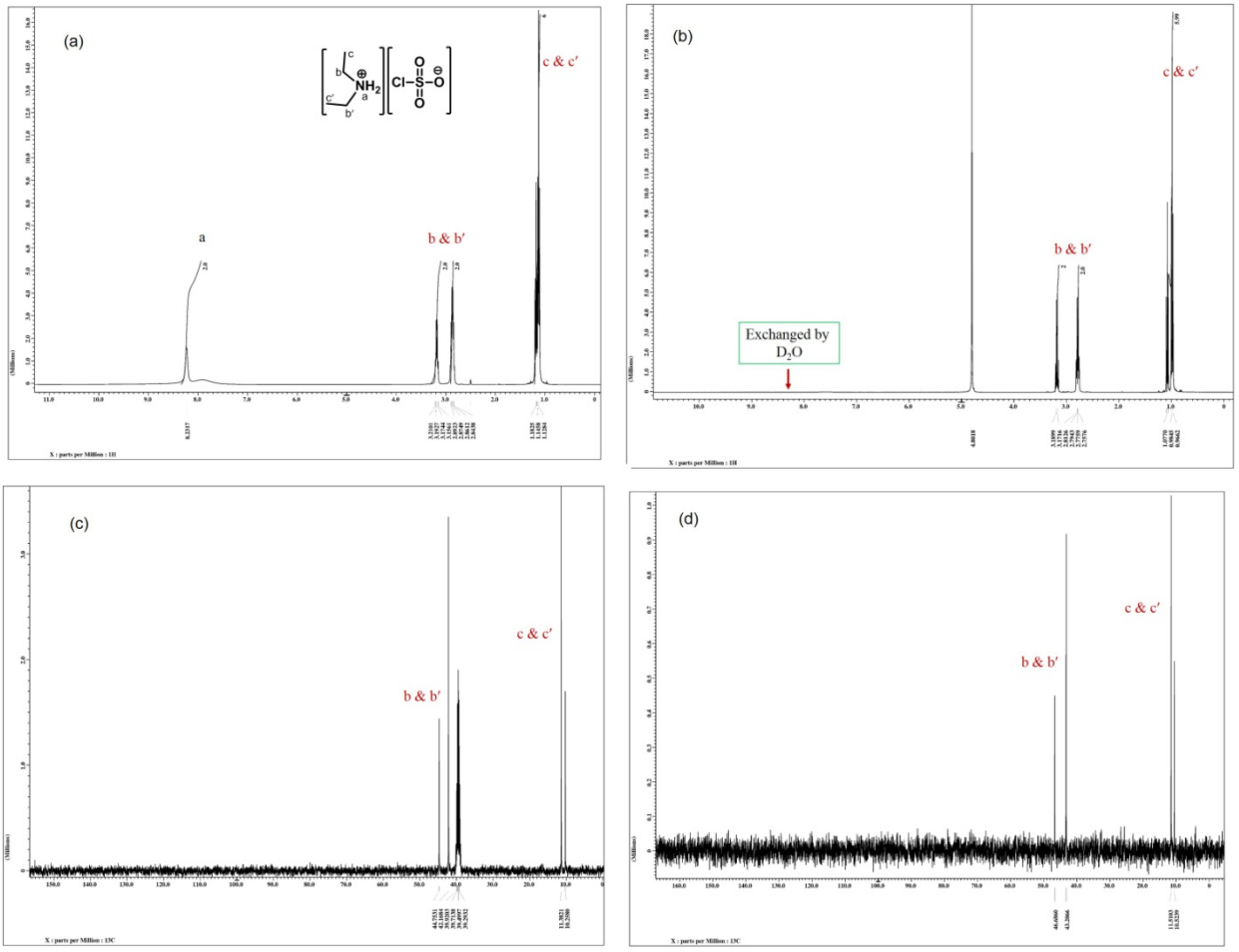



**Figure 2.** $^{1}$H and $^{13}$CNMR spectra of DEACSA IL measured in $D_2O$ [(a) and (c)] and DMSO-$d_6$ [(b) and (d)].