Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supporting information

for

Sustainable synthesis of 1, 8- Dioxooctahydroxanthenes in deep eutectic solvents (DES)

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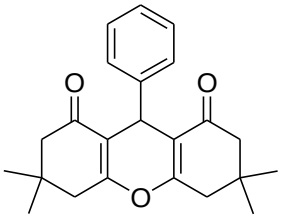
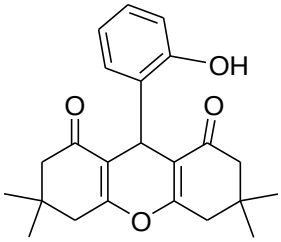
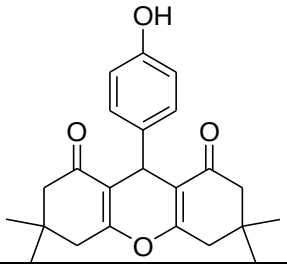
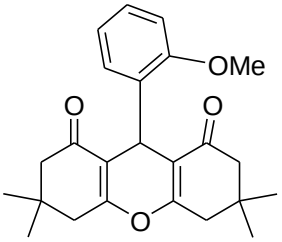
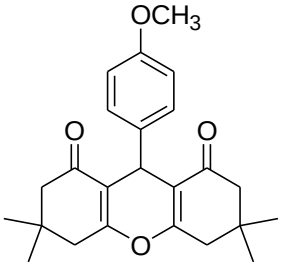
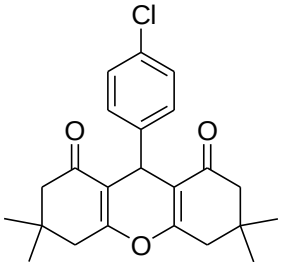
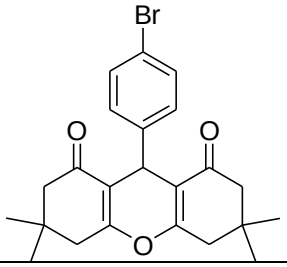
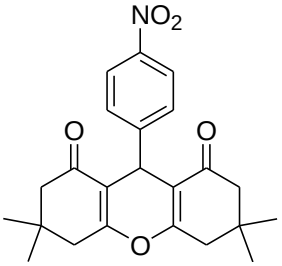
- **Methods used**

  GC-MS analysis was carried out on a 1200 L single Quadruple, Varian Gas Chromatograph model with mass detector. ^{1}H and ^{13}C NMR Spectra were recorded on Bruker 400 MHz instrument with TMS as internal standard in $CDCl_3$ (IIRBS, MG University, Kottayam). Absorption spectra were recorded using Thermo Scientific Evolusion 220 spectrophotometer in 200-900 nm range. Flourescence measurements were carried out using Horiba Flourolog 3 (FL-1057). The capacitance was measured using a HP 4277A LCZ meter operating with a frequency of 10 kHz.

Structures of the synthesised products

			
X1	X2	X3	X4
			
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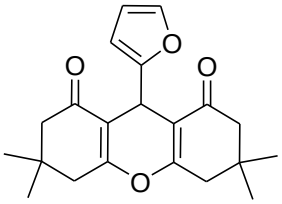
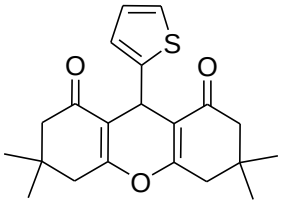
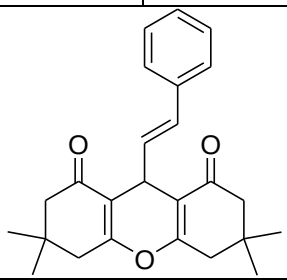
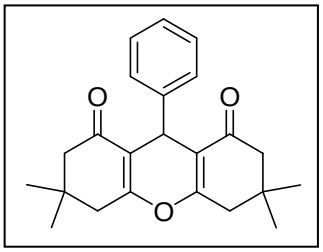
			
X9	X10	X11	

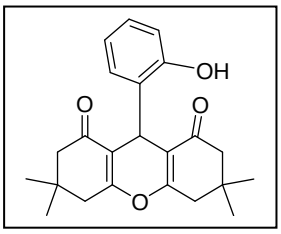
Spectral data of the synthesized products

1. 3,3,6,6-Tetramethyl-9-phenyl-1,8-dioxooctahydroxanthene (X1)[1]



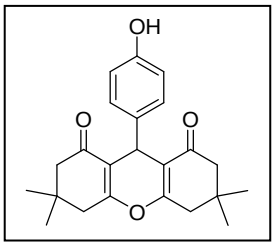
Off white solid (96 %), Melting point 199–201 °C; GC-MS (M^+): 350; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 1.01 (s, 6H), 1.12 (s, 6H), 2.29 – 2.15 (m, 4H), 2.48 (s, 4H), 4.76 (s, 1H), 7.11 (t, 1H), 7.23 (t, 2H), 7.30 (t, *J* = 6.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 27.3, 29.26, 32.19, 40.87, 50.74, 115.66, 126.35, 128.03, 128.37, 144.08, 162.25, 196.38.

2. 3,3,6,6-Tetramethyl-9-(2-hydroxyphenyl)-1,8-dioxooctahydroxanthene (X2)[2]



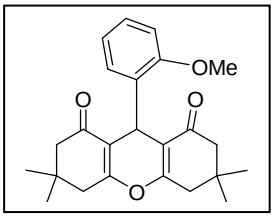
Pale yellow solid (92 %) Melting point 206–208 °C; GC-MS (M^+): 366; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 0.98 (d, 6H), 1.01 (s, 3H), 1.11 (s, 3H), 1.96 (q, 2H), 2.27–2.58 (m, 6H), 4.68 (s, 1H), 6.98 (d, 3H), 7.11–7.15 (m, 1H), 10.44 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 26.60, 27.11, 27.66, 29.17, 29.68, 30.97, 32.20, 41.53, 43.19, 49.96, 50.59, 111.04, 115.69, 118.28, 124.39, 124.48, 127.42, 128.00, 150.97, 168.84, 170.66, 196.50, 200.55.

3. 3,3,6,6-Tetramethyl-9-(4-hydroxyphenyl)-1,8-dioxooctahydroxanthene (X3)[1]



Off white solid (93 %), Melting point 252–254 °C; GC-MS (M^+): 366; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 0.97 (s, 6H), 1.07 (s, 6H), 2.09-2.25 (m, 4H), 2.44 (s, 4H), 4.64 (s, 1H), 6.51 (d, *J* = 7.4 Hz, 2H), 7.04 (d, *J* = 7.4 Hz, 2H), 7.17 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 27.4, 29.2, 30.9, 32.3, 40.8, 50.7, 115.3, 115.9, 129.3, 135.4, 154.8, 162.5, 197.4.

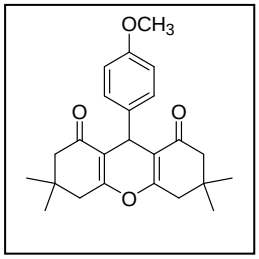
4. 3,3,6,6-Tetramethyl-9-(2-Methoxyphenyl)-1,8-dioxooctahydroxanthene (X4)[3]



Off white solid (96 %), Melting point 230–231 °C; GC-MS (M^+): 380, ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 0.96 (s, 6H), 1.11 (s, 6H), 2.12-2.25 (m, 4H), 2.37-2.50 (m, 4H), 3.79 (s, 3H), 4.87 (s, 1H), 6.77 (d, *J* = 8.1 Hz, 1H), 6.88 (d, 1H), 7.12 (t, 1H), 7.43 – 7.44 (d, 1H); ^{13}C

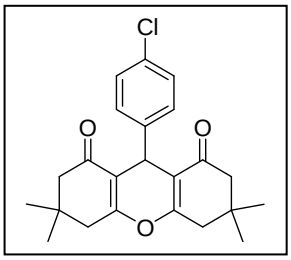
NMR (100 MHz, $CDCl_3$) δ (ppm) 26.76, 29.49, 32.11, 40.97, 50.79, 55.14, 110.65, 113.76, 120.36, 127.75, 132.07, 157.59, 162.86, 196.54.

5. 3,3,6,6-Tetramethyl-9-(4-Methoxyphenyl)-1,8-dioxooctahydroxanthene (X5)[1]



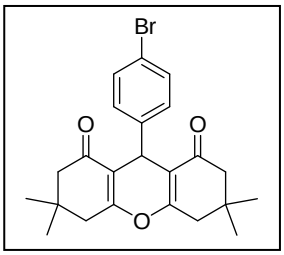
White solid (88 %), Melting point 243–245 ℃; GC-MS (M^+): 380, ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 1.01 (s, 6H), 1.12 (s, 6H), 2.18 (d, *J* = 16.4 Hz, 2H), 2.25 (d, *J* = 16.4 Hz, 2H), 2.48 (s, 4H), 3.75 (s, 3H), 4.72 (s, 1H), 6.77 (d, *J* = 8.8 Hz, 2H), 7.22 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 27.3, 29.3, 30.9, 32.2, 40.9, 50.8, 55.1, 113.5, 115.8, 129.3, 136.5, 157.9, 162.1, 196.5.

6. 3,3,6,6-Tetramethyl-9-(4-chlorophenyl)-1,8-dioxooctahydroxanthene (X6)[1]



White solid (88 %), Melting point 229–231 ℃; GC-MS (M^+): 384, ^{1}H NMR (500 MHz, DMSO-*d6*): δ (ppm) 0.90 (s, 6H), 1.04 (s, 6H), 2.09 (d, *J* = 16.1 Hz, 2H), 2.27 (d, 2H), 2.52 (d, 2H), 2.57 (d, 2H), 4.51 (s, 1H), 7.19 (d, 2H), 7.29 (d, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 27.3, 29.3, 31.5, 32.2, 40.8, 50.7, 115.2, 128.2, 129.8.2, 131.9, 142.8, 162.5, 196.4.

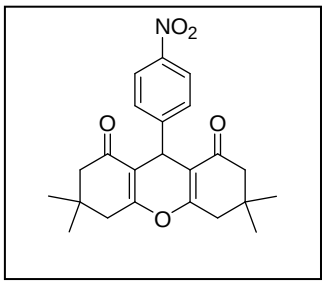
7. 3,3,6,6-Tetramethyl-9-(4-bromophenyl)-1,8-dioxooctahydroxanthene (X7)[1]



White solid (88 %), Melting point 242–244 ℃; GC-MS (M^+): 429, ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (s, 6H), 0.99 (s, 6H), 2.09 (q, *J* = 16.3 Hz, 4H), 2.35 (s, 4H), 4.58 (s, 1H), 7.07

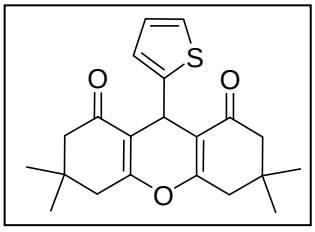
(d, *J* = 8.4 Hz, 2H), 7.23 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 27.48, 29.47, 32.40, 41.03, 50.87, 115.37, 120.43, 130.37, 131.35, 143.41, 162.68, 196.59.

8. 3,3,6,6-Tetramethyl-9-(4-nitrophenyl)-1,8-dioxooctahydroxanthene (X8)[1]



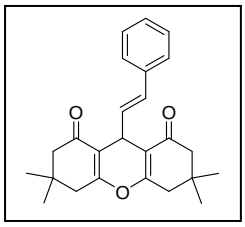
Yellow solid (90 %), Melting point 224–226 °C; GC-MS (M^+): 395, ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 0.99 (s, 6H), 1.12 (s, 6H), 2.16 (d, 2H), 2.26 (d, 2H), 2.51 (t, 4H), 4.83 (s, 1H), 7.48 (d, 2H), 8.08 (2H, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 27.7, 29.6, 32.6, 32.8, 41.2, 51.0, 114.9, 123.8, 129.8, 146.8, 152.0, 163.5, 196.7.

9. 3,3,6,6-Tetramethyl-9-(2-Thienyl)-1,8-dioxooctahydroxanthene (X10)[4]

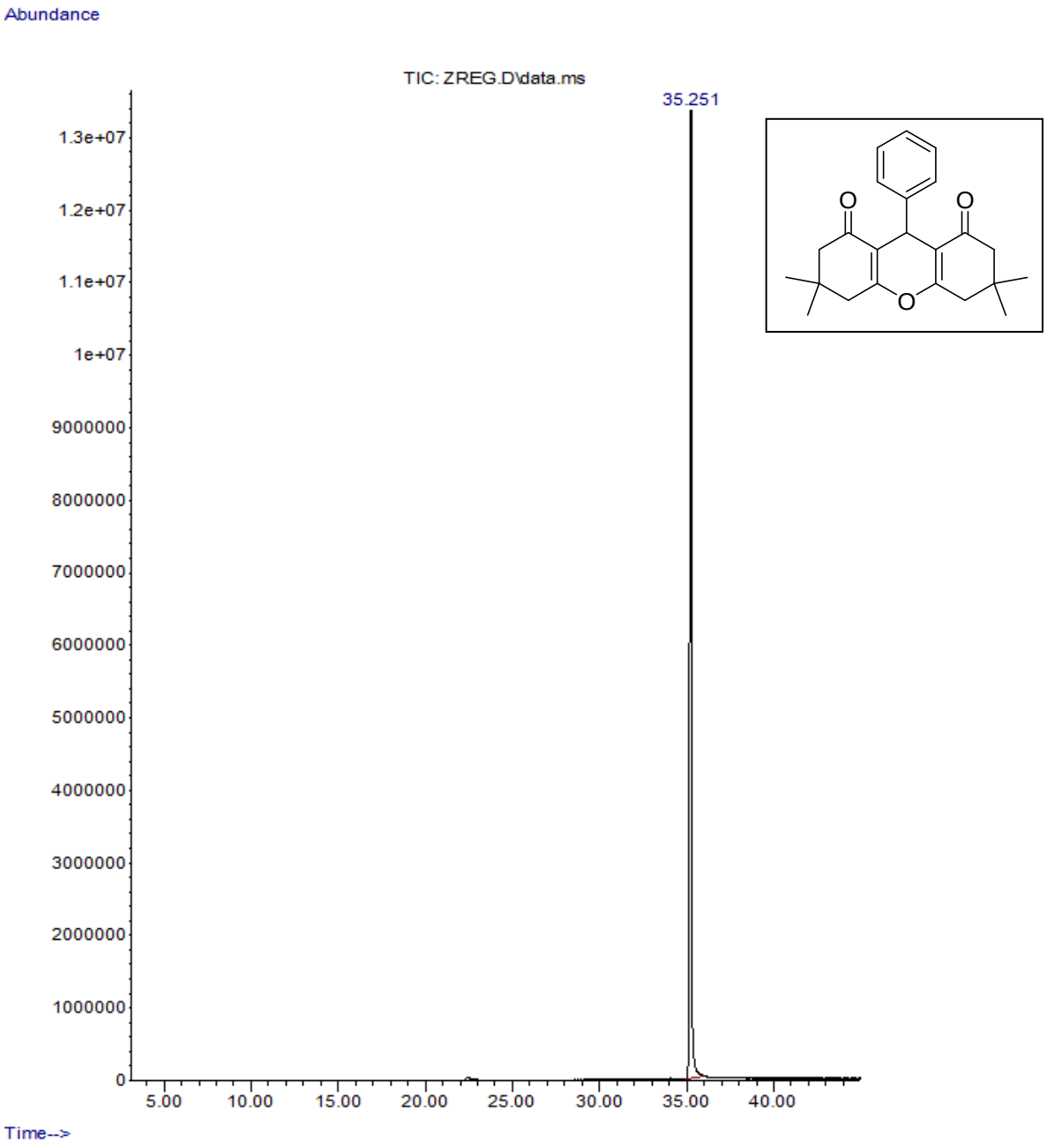


Black solid (90); Melting point 161-162 ºC; GC-MS (M^+): 356, ^{1}H NMR (400 MHz, $CDCl_3$) δ1.02 (s, 6H), 1.08 (s, 6H), 2.23 (s, 4H), 2.43 (s, 4H), 5.11 (s, 1H), 6.80 (m, 1H), 6.93 (d, 1H), 6.99 (d, 1H), ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.55, 27.54, 29.53, 32.32, 41.04, 50.93, 115.45, 123.57, 125.49, 126.93, 148.33, 162.91, 196.50.

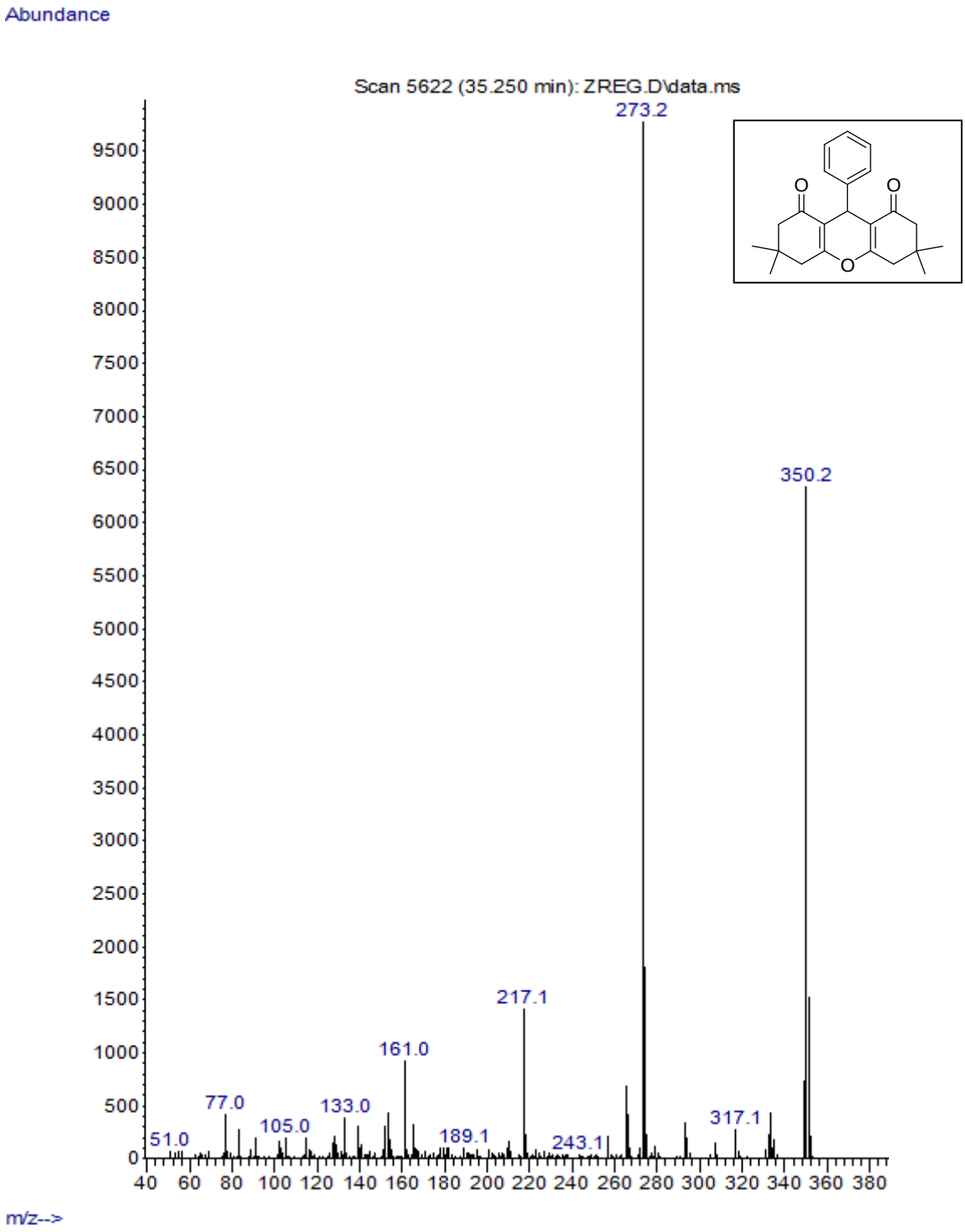
10. 3,3,6,6-Tetramethyl-9-(2- phenylethylene)-1,8-dioxooctahydroxanthene (X11)[5]



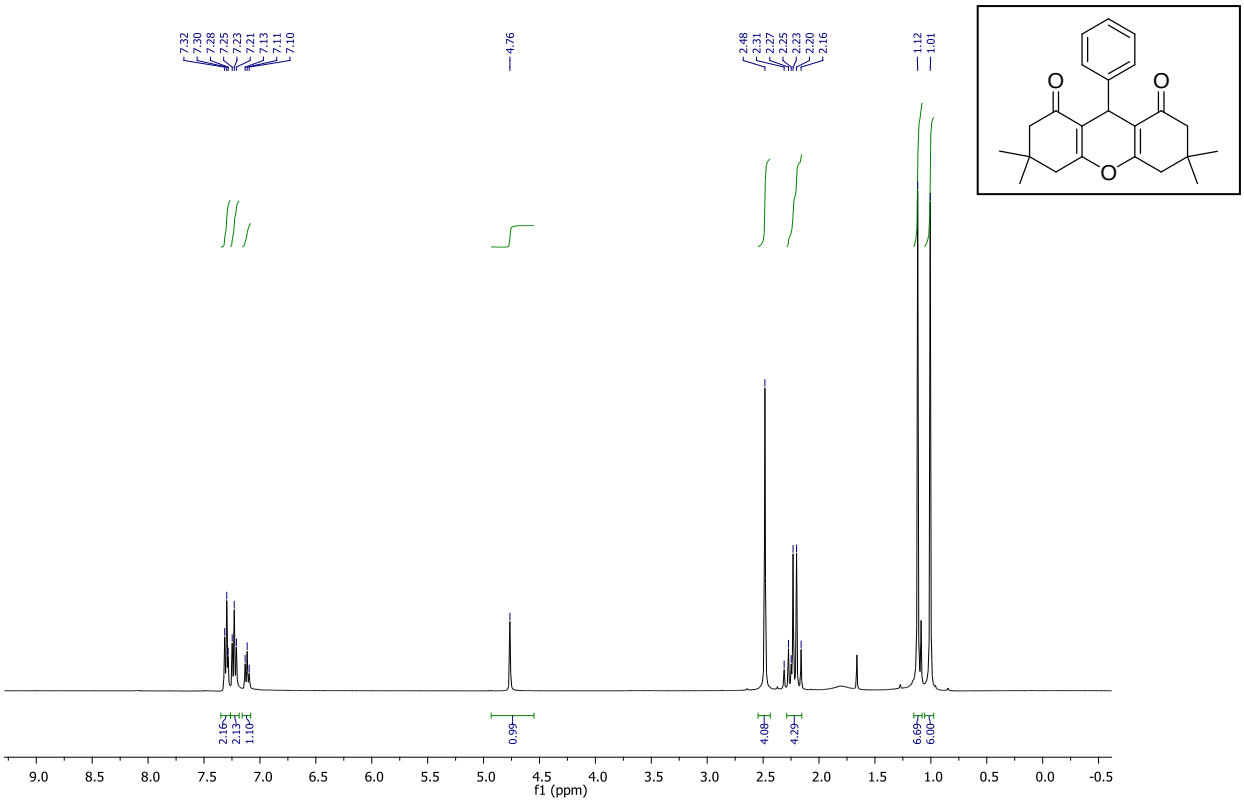
Pale yellow solid (88 %); Melting point 175– 177 °C; GC-MS (M^+): 376, ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 1.12 (s, 12H), 2.30 (d, 4H), 2.44 (d, 4H), 4.41 (s, 1H), 6.24-6.32 (m, 2H), 7.15 – 7.28 (m, 5H), ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 27.59, 29.24, 32.21, 40.94, 50.84, 114.51, 126.35, 127.11, 128.29, 130.44, 131.31, 137.25, 163.11, 196.62.



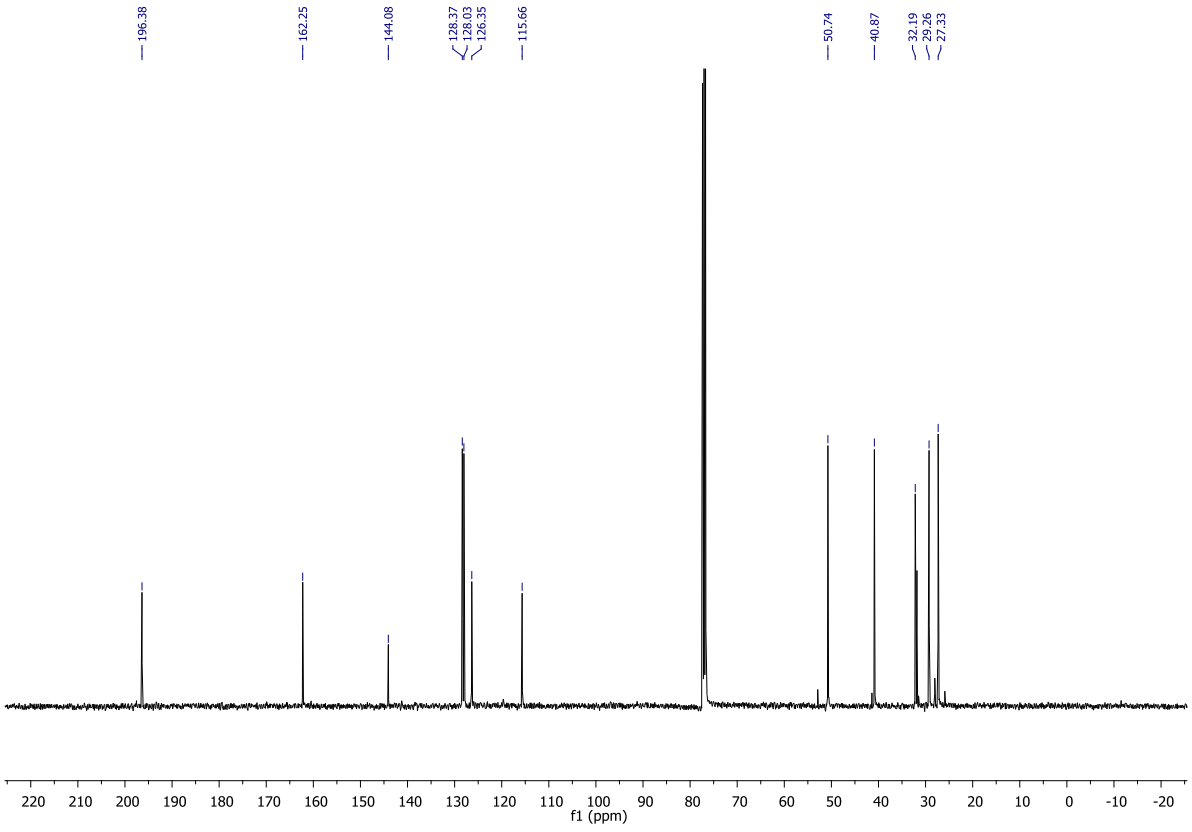
Gas chromatogram of 3,3,6,6-Tetramethyl-9-phenyl-1,8-dioxooctahydroxanthene (X1)



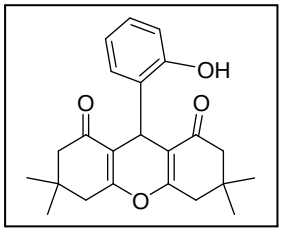
Mass spectrum of 3,3,6,6-Tetramethyl-9-phenyl-1,8-dioxooctahydroxanthene (X1)

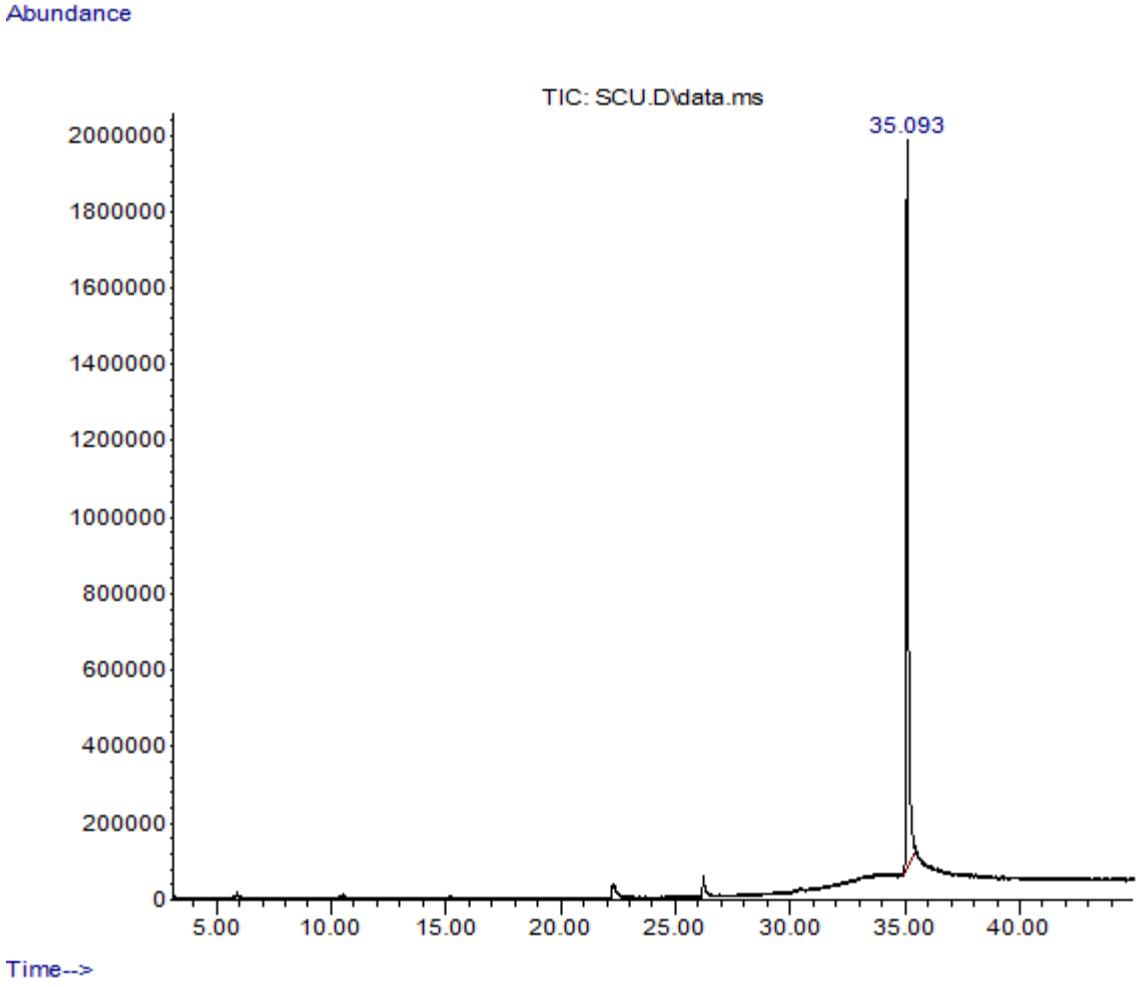


^{1}H NMR spectrum of 3,3,6,6-Tetramethyl-9-phenyl-1,8-dioxooctahydroxanthene (X1)

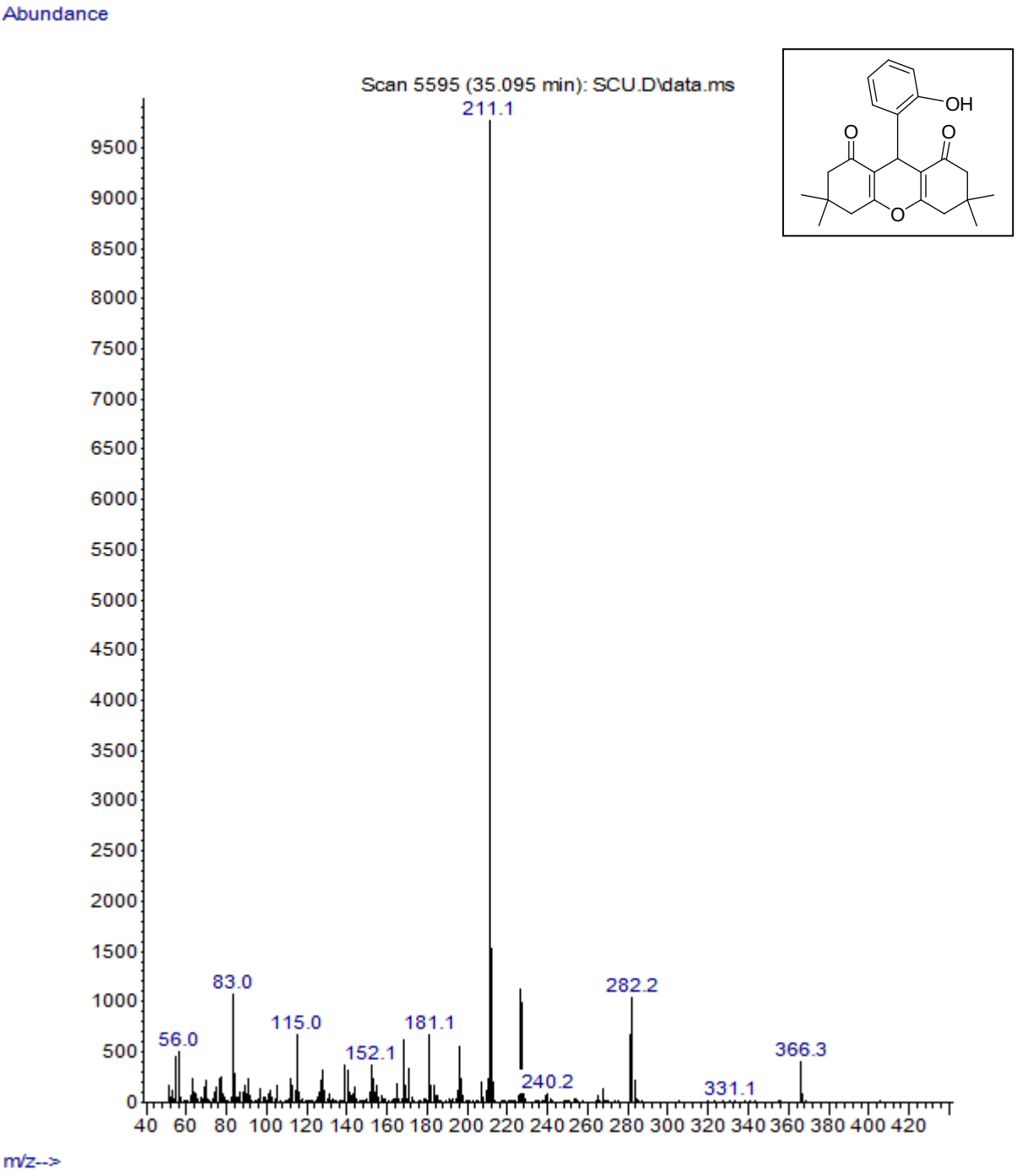


^{13}C NMR spectrum of3,3,6,6-Tetramethyl-9-phenyl-1,8-dioxooctahydroxanthene (X1)

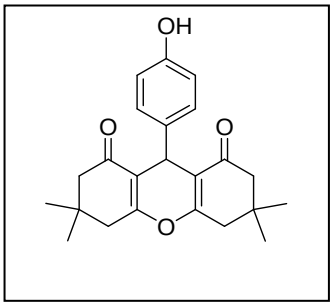


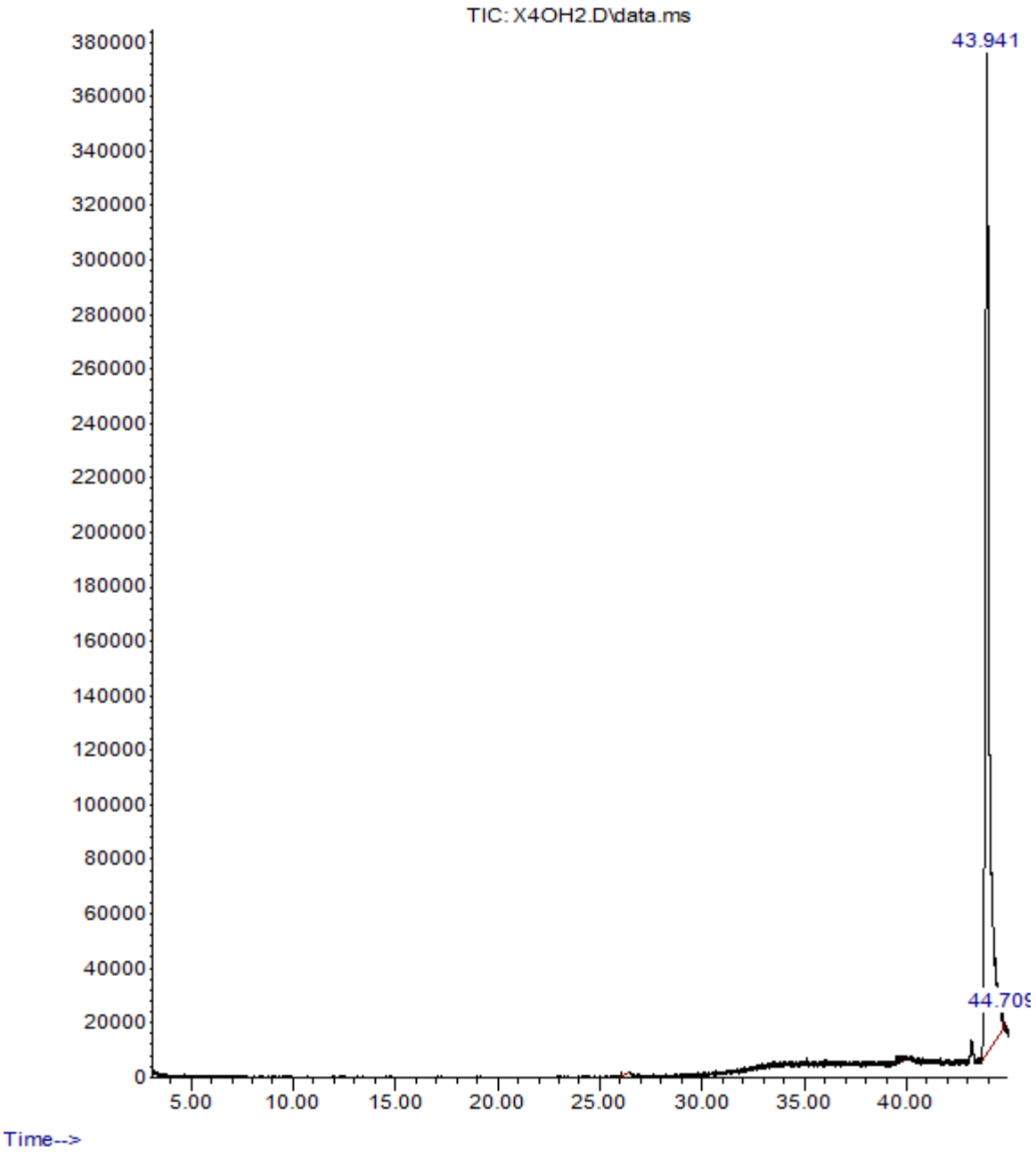


Gas chromatogram of 3,3,6,6-Tetramethyl-9-(2-hydroxyphenyl)-1,8 dioxooctahydroxanthene (X2)

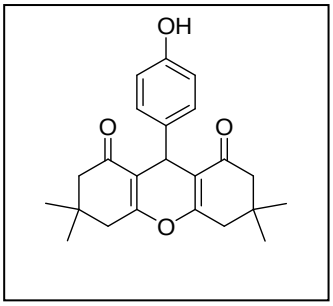


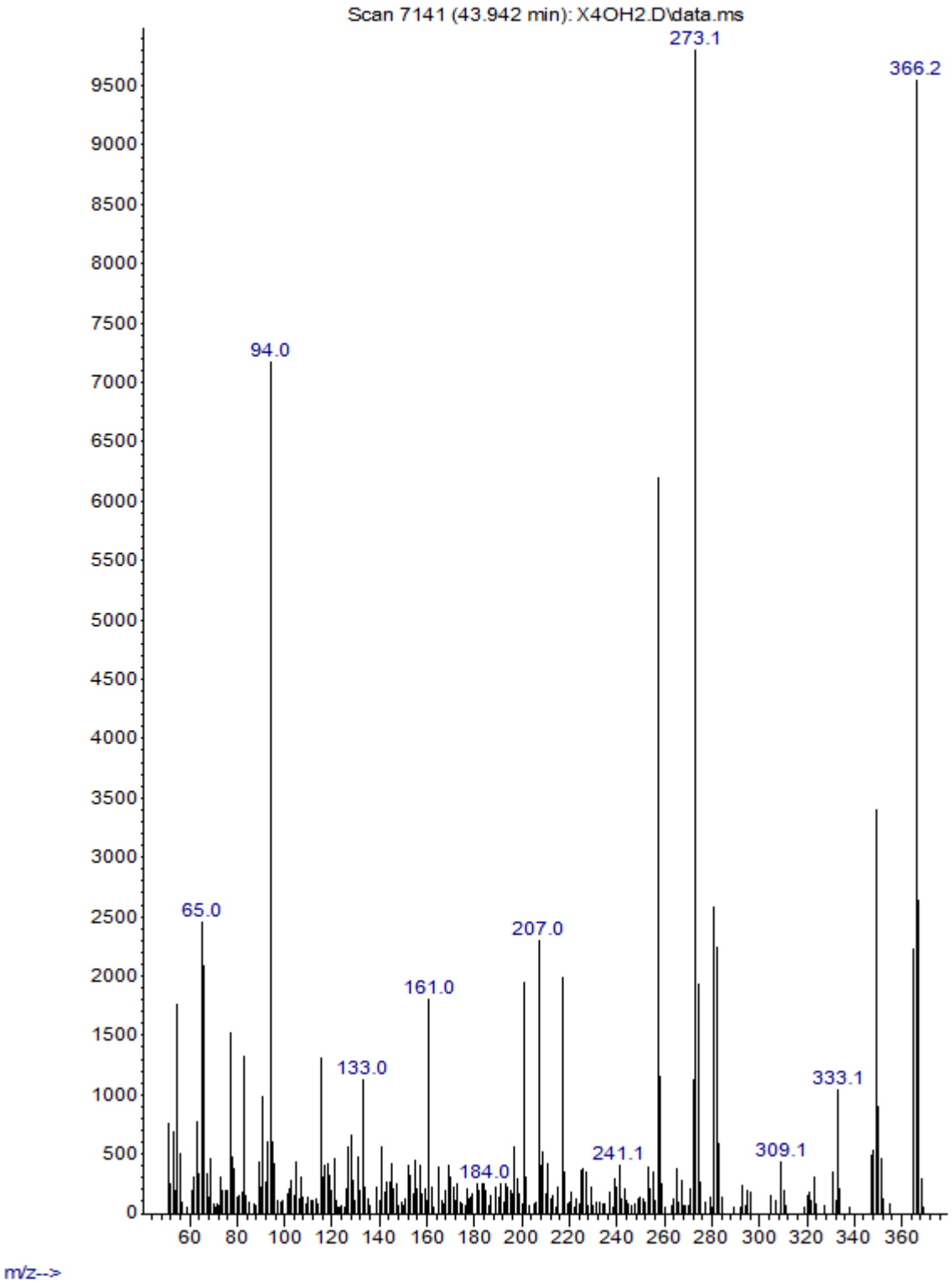
Mass spectrum of 3,3,6,6-Tetramethyl-9-(2-hydroxyphenyl)-1,8 dioxooctahydroxanthene (X2)



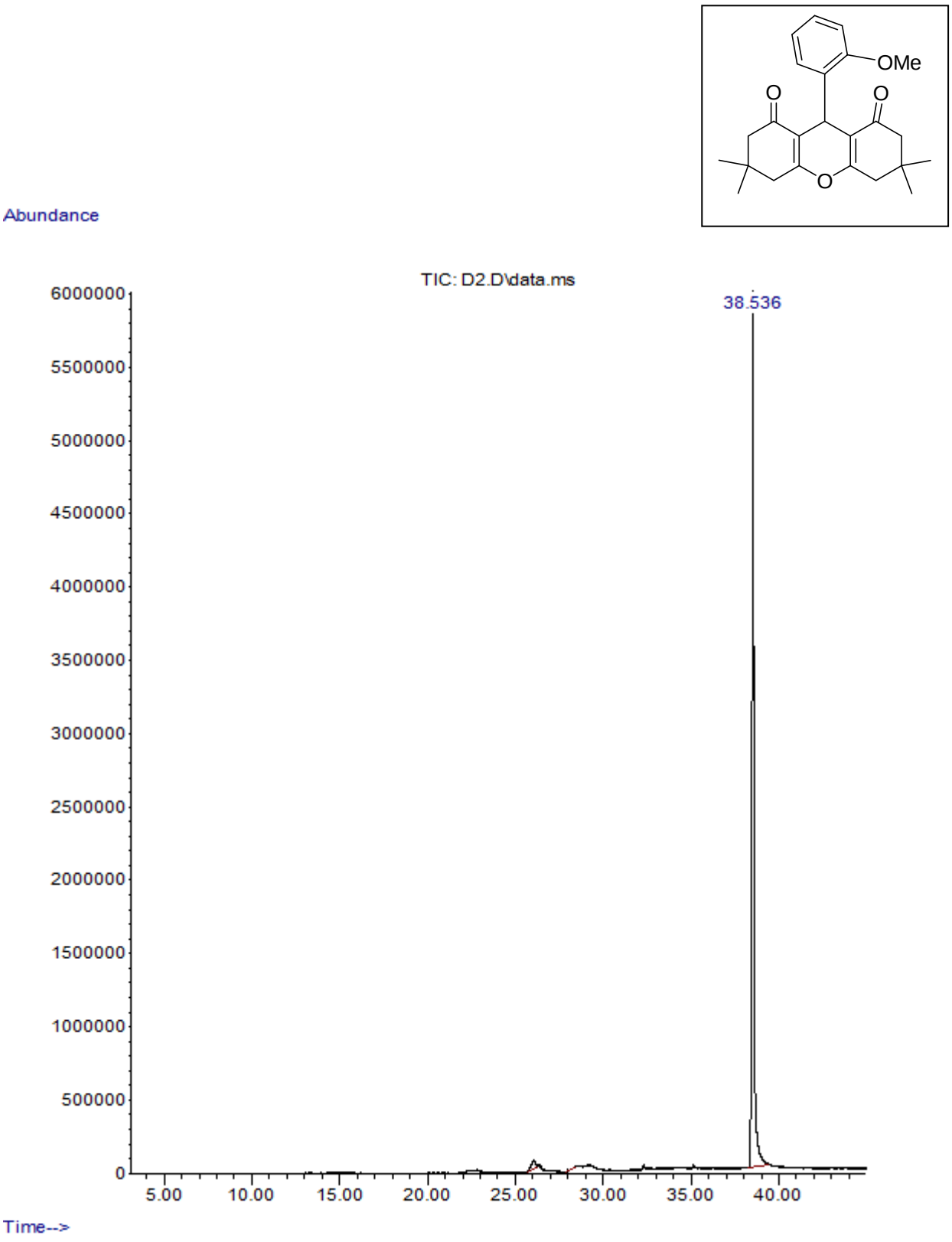


Gas chromatogram of 3,3,6,6-Tetramethyl-9-(4-hydroxyphenyl)-1,8-dioxooctahydroxanthene (X3)

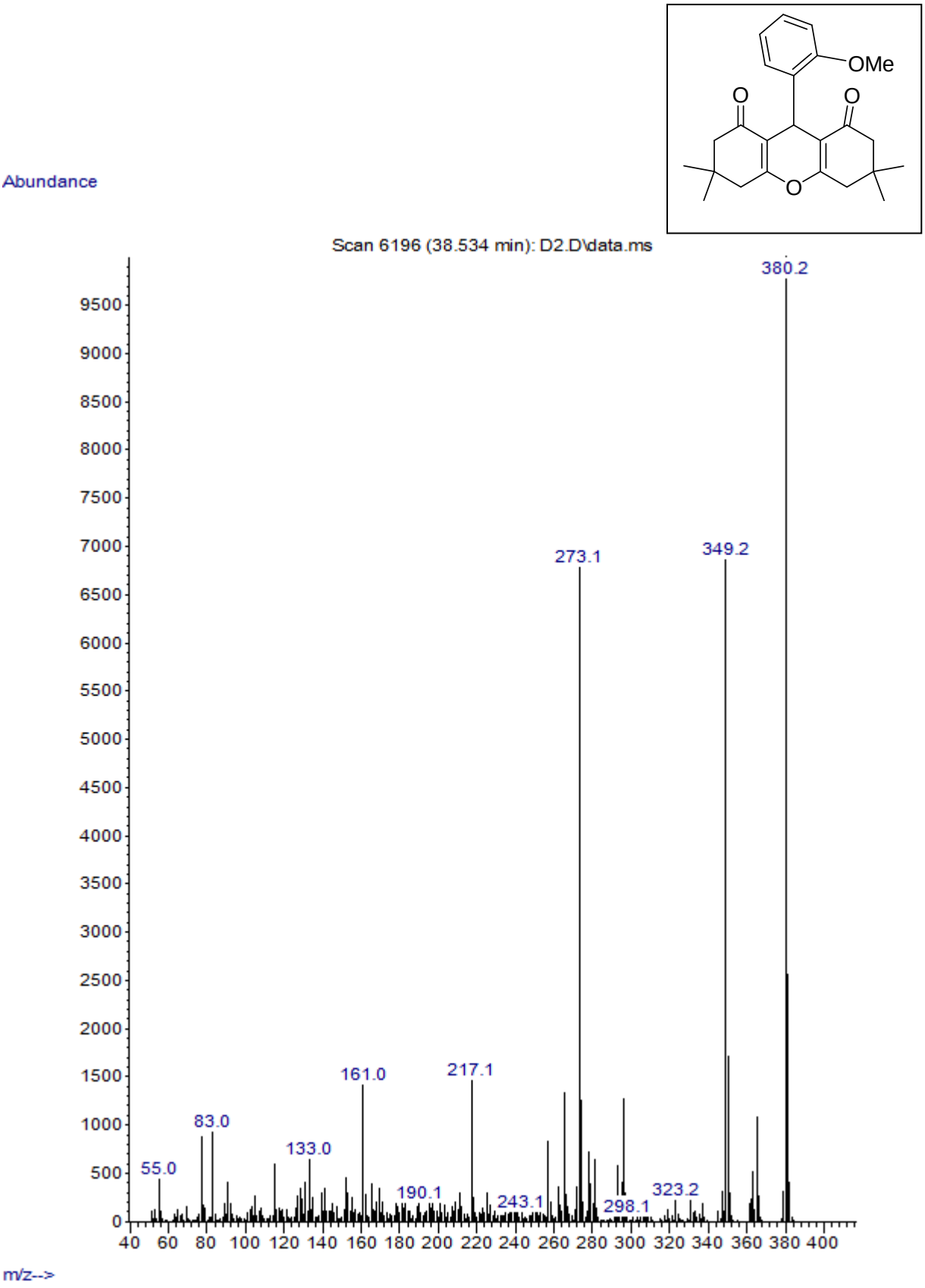




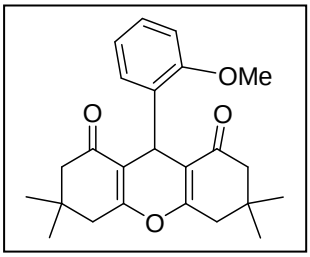
Mass spectrum of 3,3,6,6-Tetramethyl-9-(4-hydroxyphenyl)-1,8-dioxooctahydroxanthene (X3)

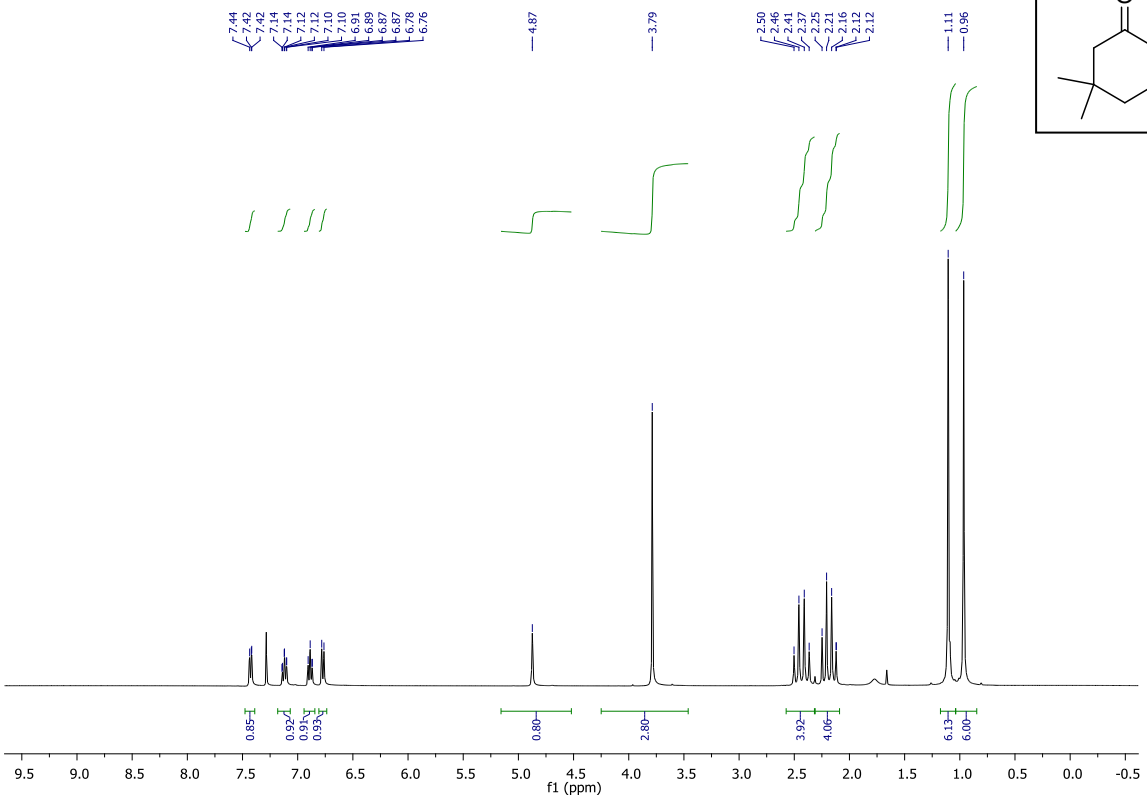


Gas chromatogram of 3,3,6,6-Tetramethyl-9-(2-Methoxyphenyl)-1,8-dioxooctahydroxanthene (X4)

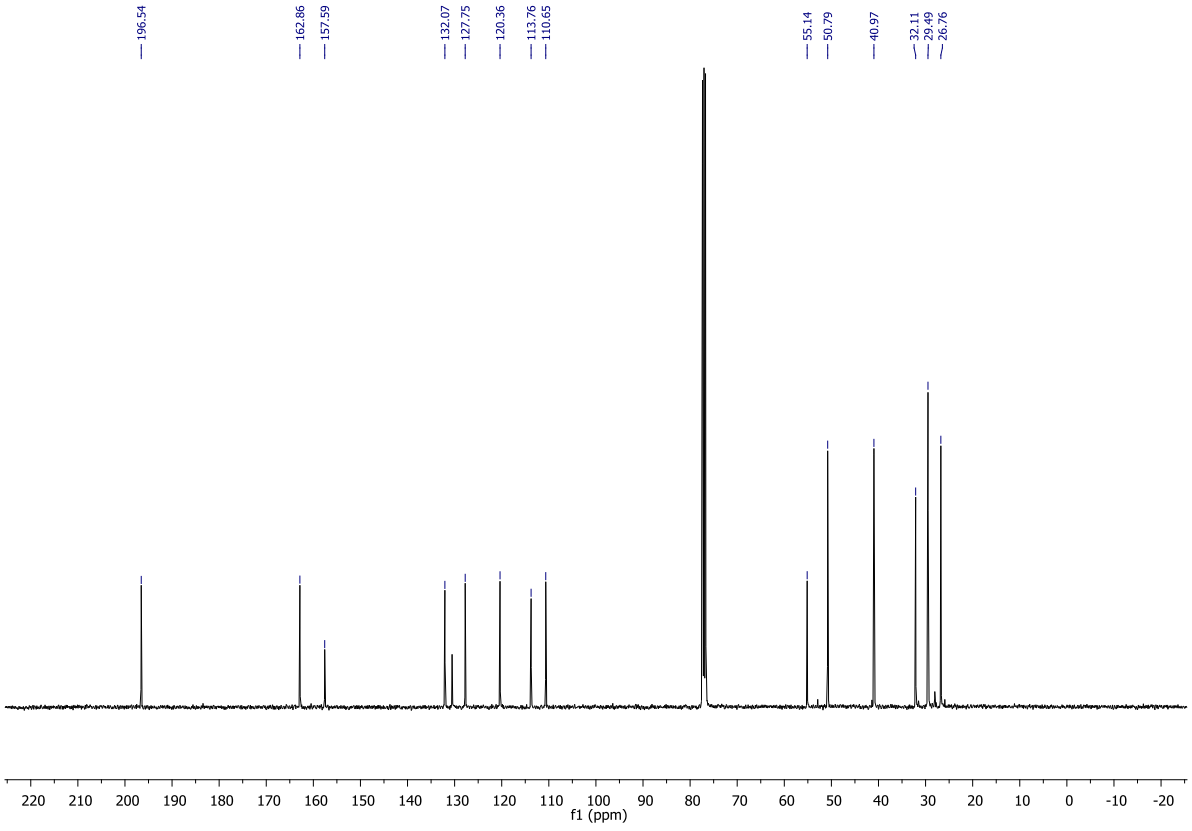


Mass spectrum of 3,3,6,6-Tetramethyl-9-(2-Methoxyphenyl)-1,8-dioxooctahydroxanthene (X4)

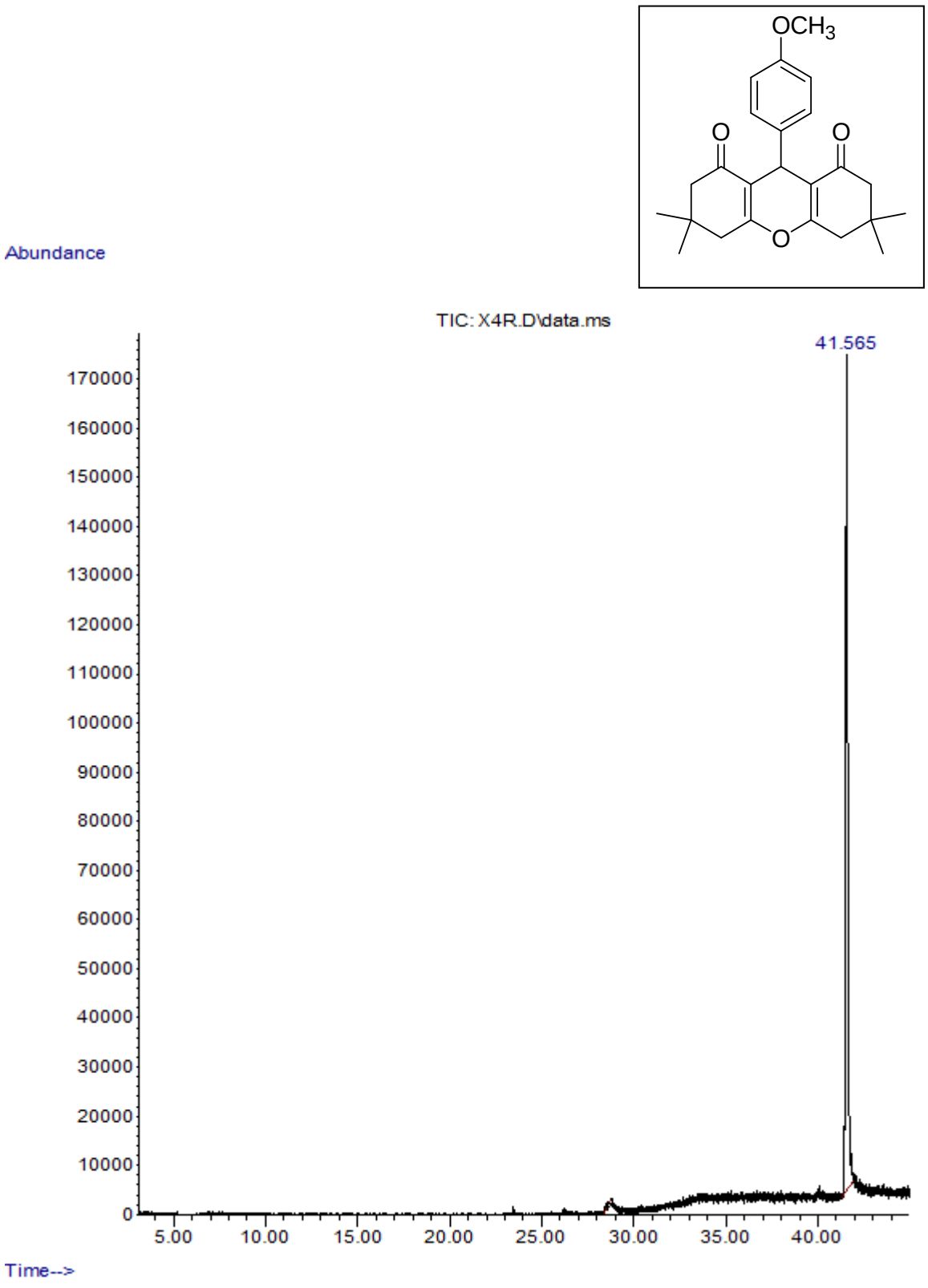




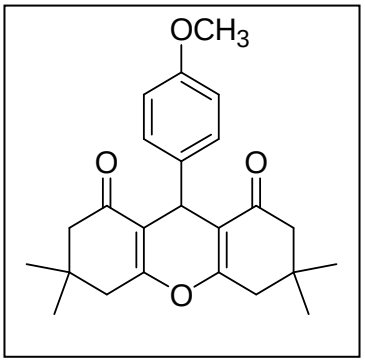
^{1}H NMR spectrum of 3,3,6,6-Tetramethyl-9-(2-Methoxyphenyl)-1,8-dioxooctahydroxanthene (X4)

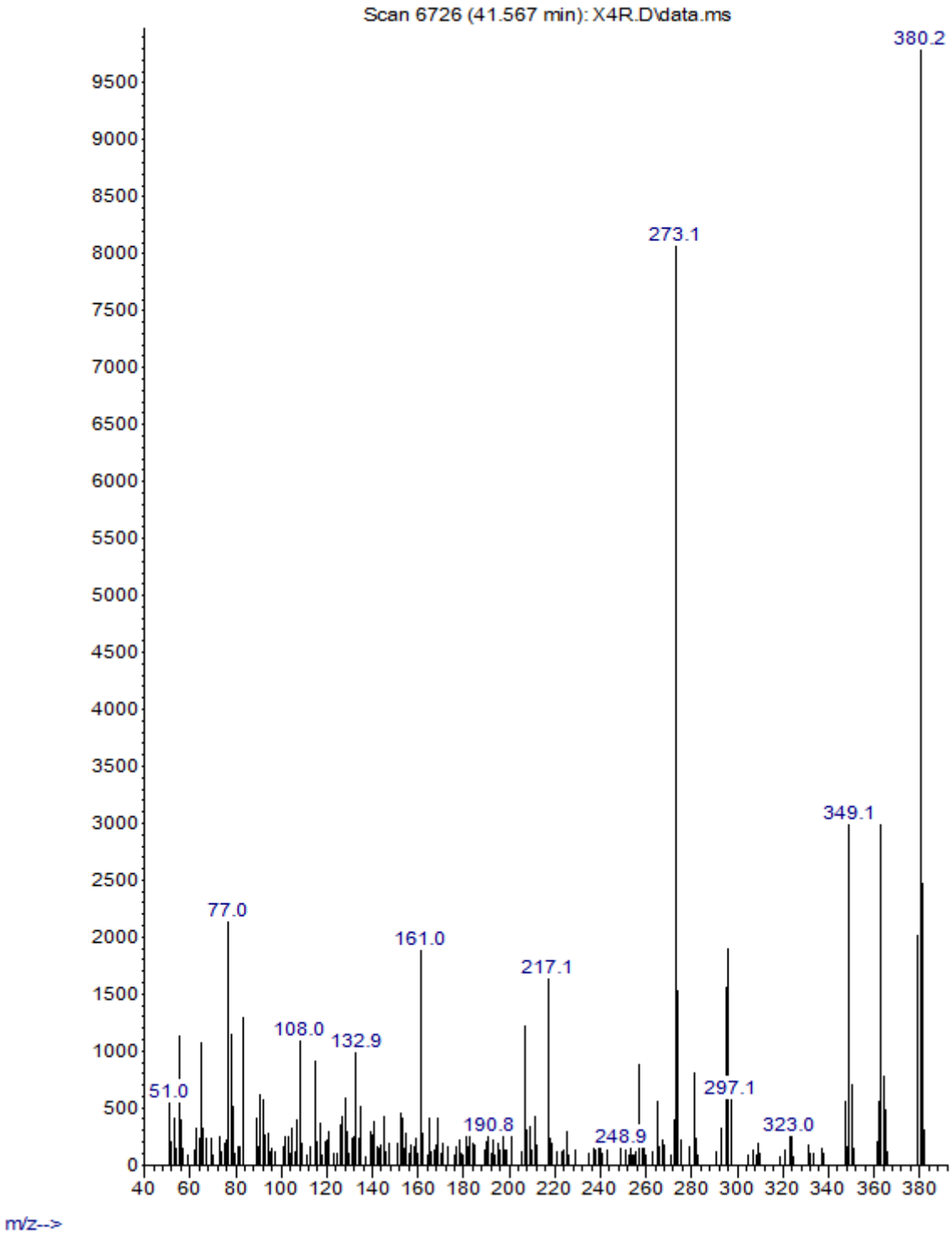


^{13}C NMR spectrum of 3,3,6,6-Tetramethyl-9-(2-Methoxyphenyl)-1,8-dioxooctahydroxanthene (X4)

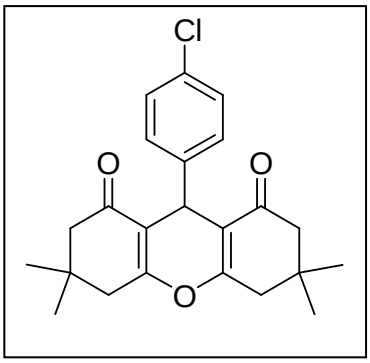


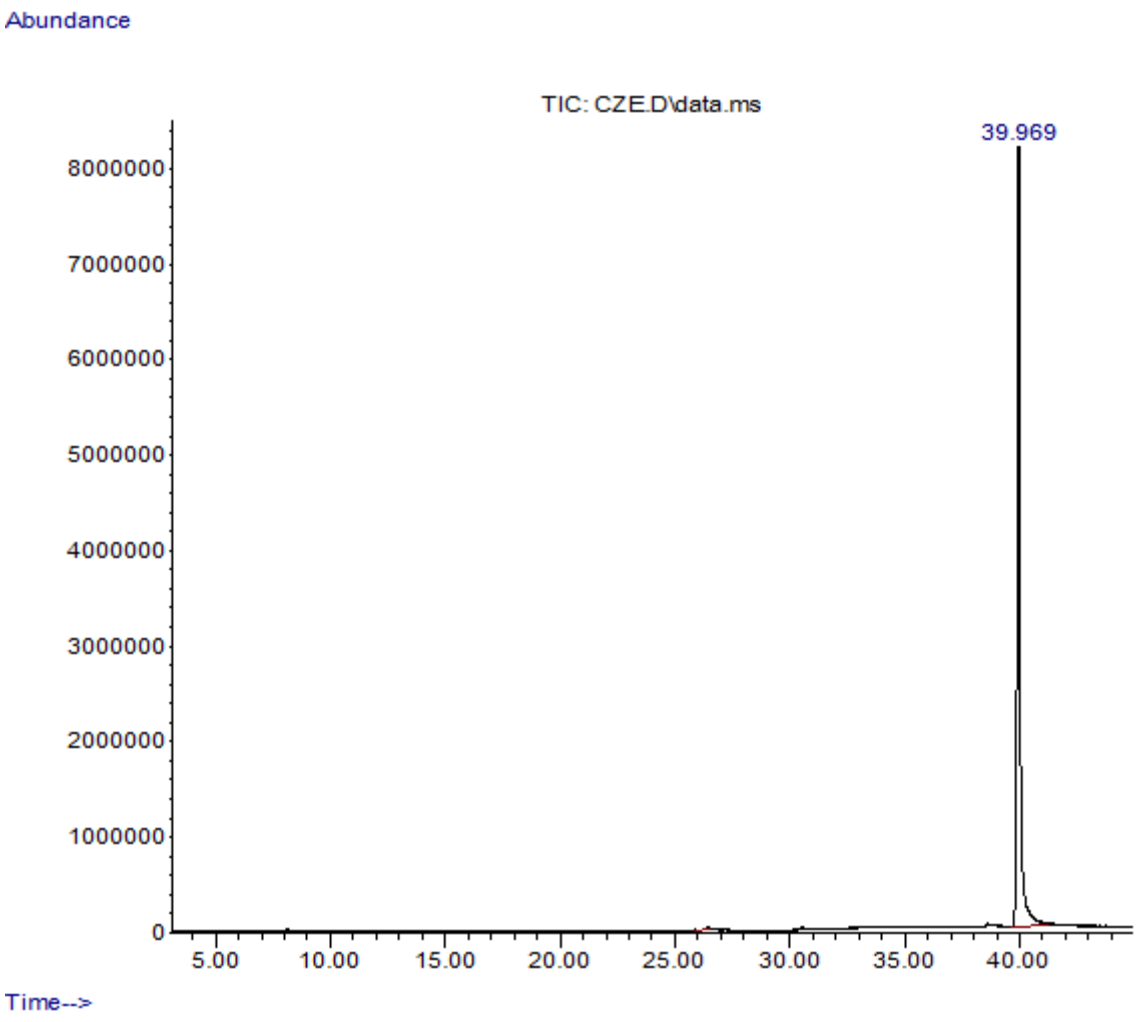
Gas chromatogram of 3,3,6,6-Tetramethyl-9-(4-Methoxyphenyl)-1,8-dioxooctahydroxanthene (X5)



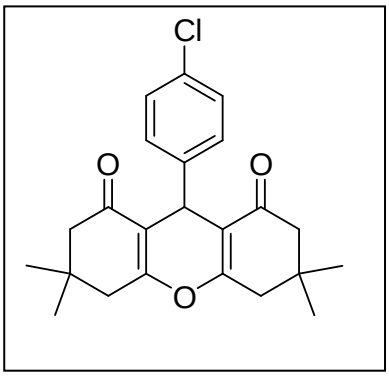


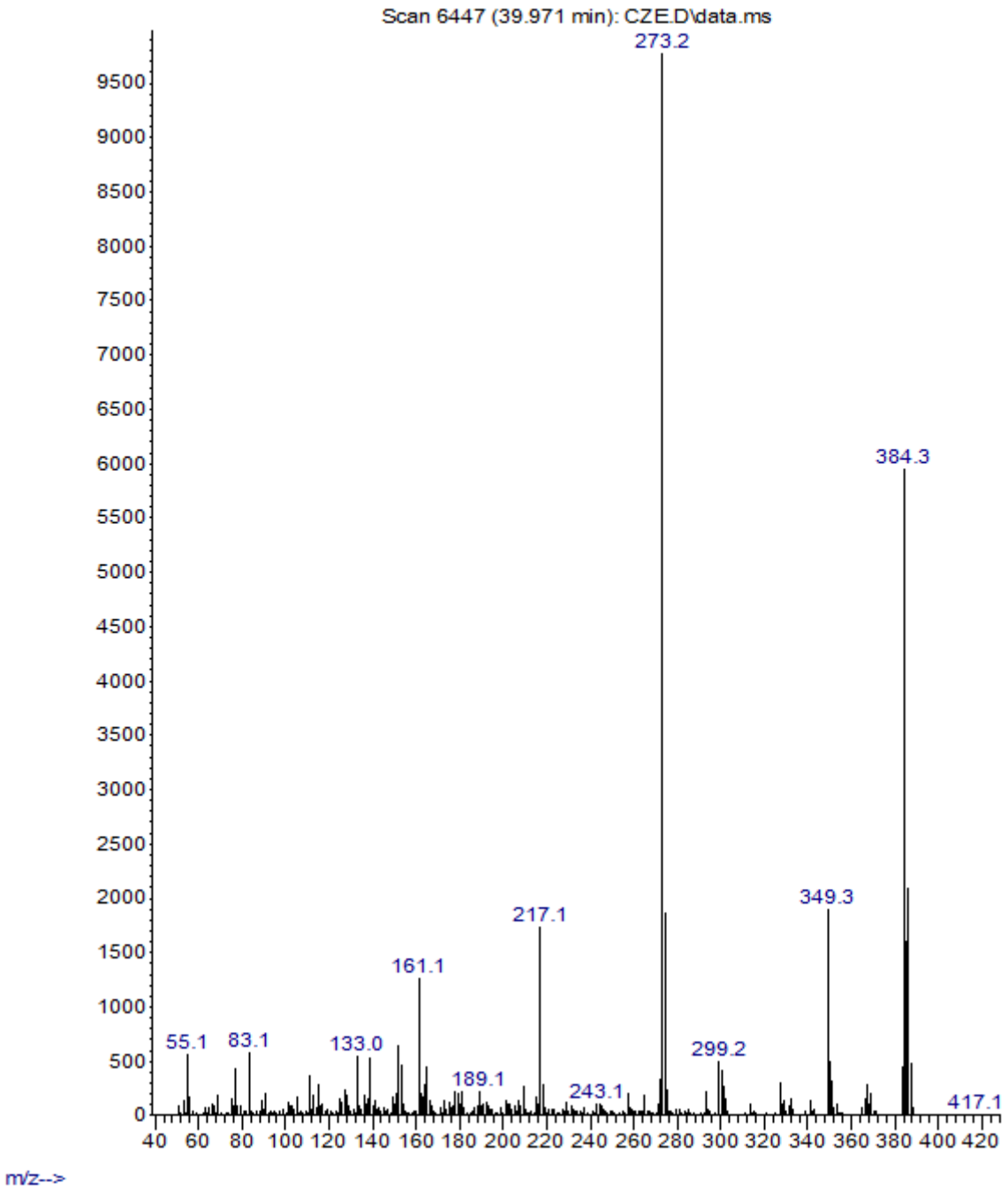
Mass spectrum of 3,3,6,6-Tetramethyl-9-(4-Methoxyphenyl)-1,8-dioxooctahydroxanthene (X5)



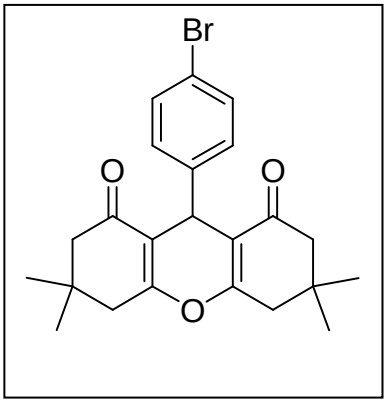


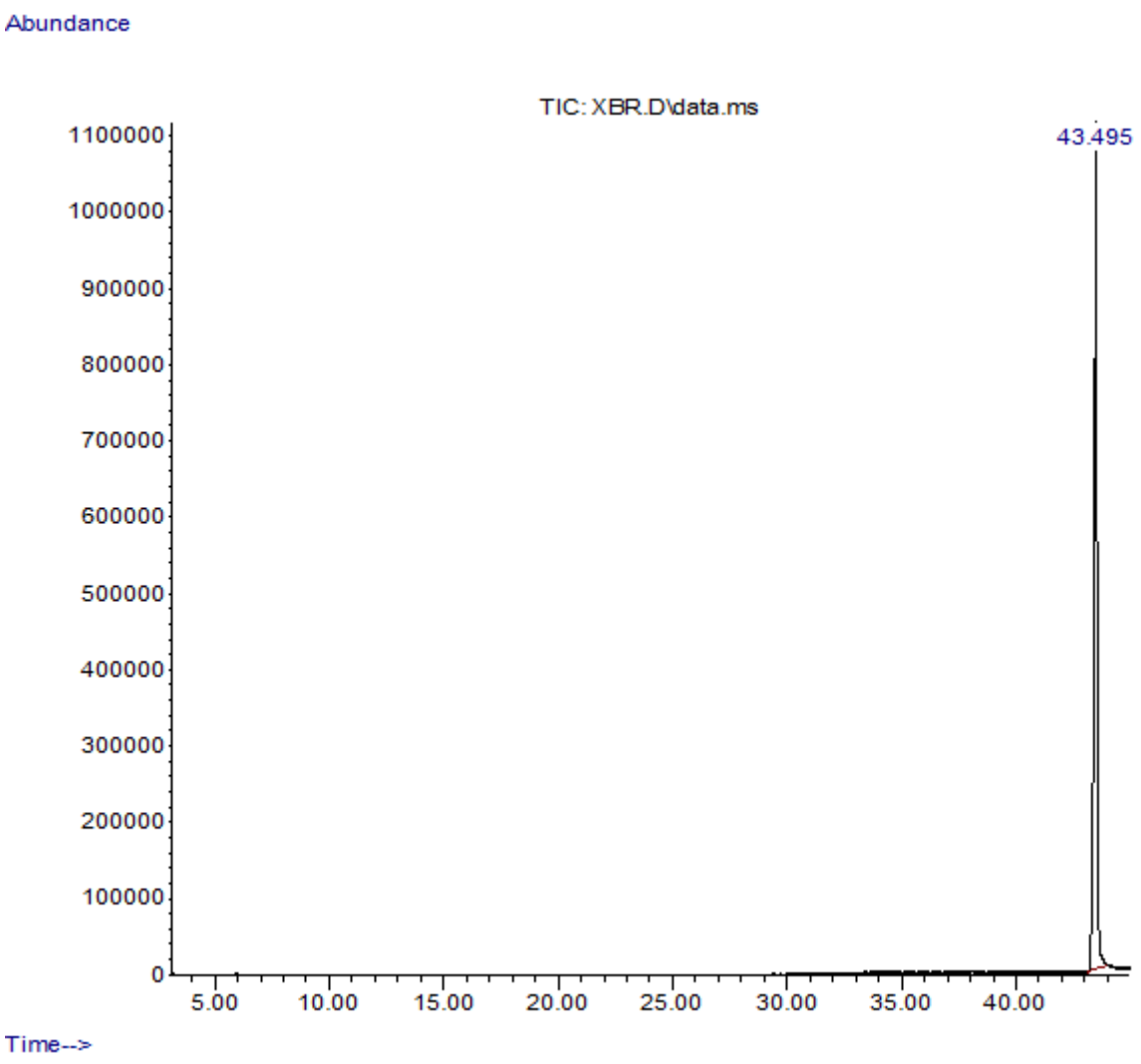
Gas chromatogram of 3,3,6,6-Tetramethyl-9-(4-chlorophenyl)-1,8-dioxooctahydroxanthene (X6)



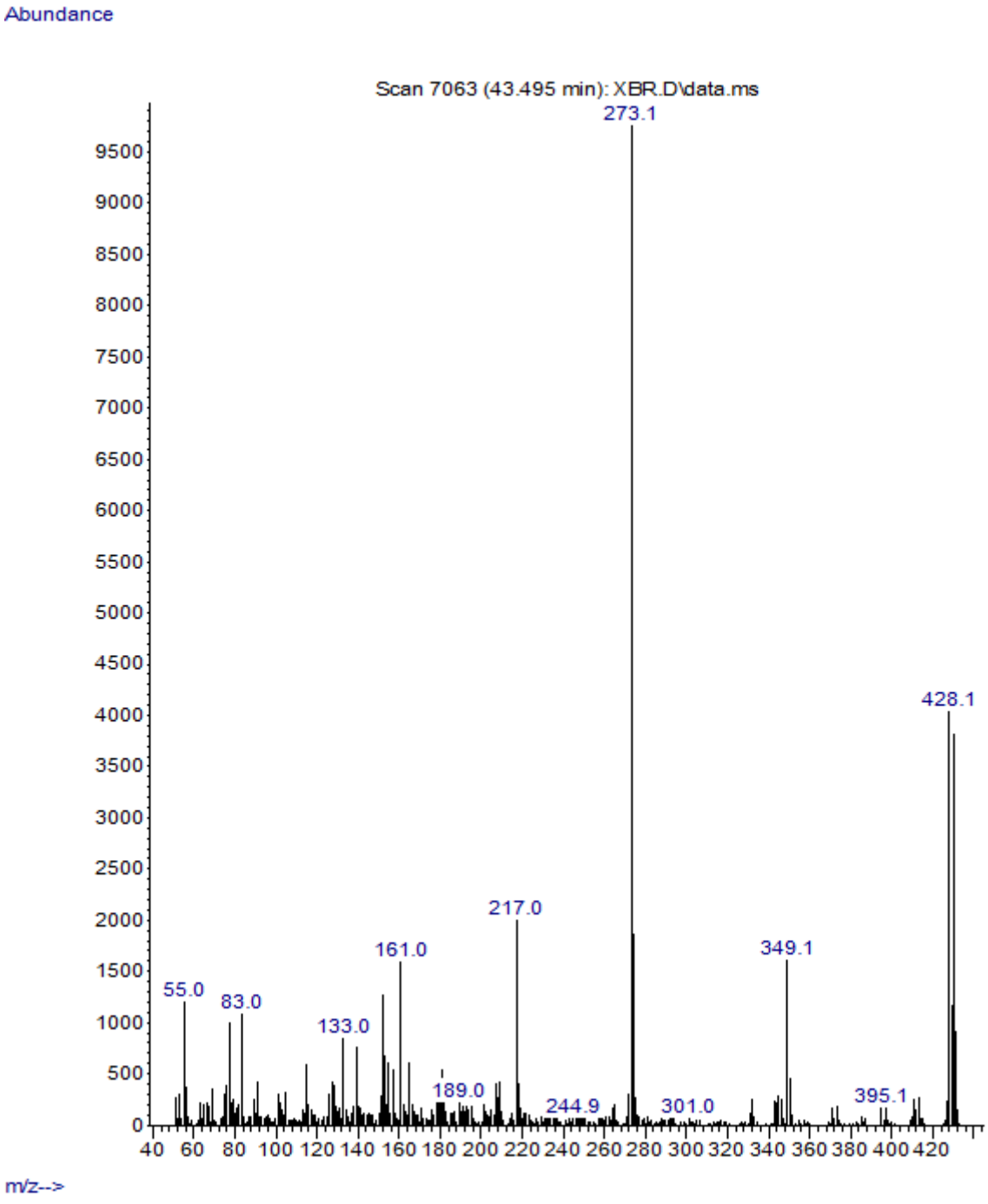


Mass spectrum of 3,3,6,6-Tetramethyl-9-(4-chlorophenyl)-1,8-dioxooctahydroxanthene (X6)

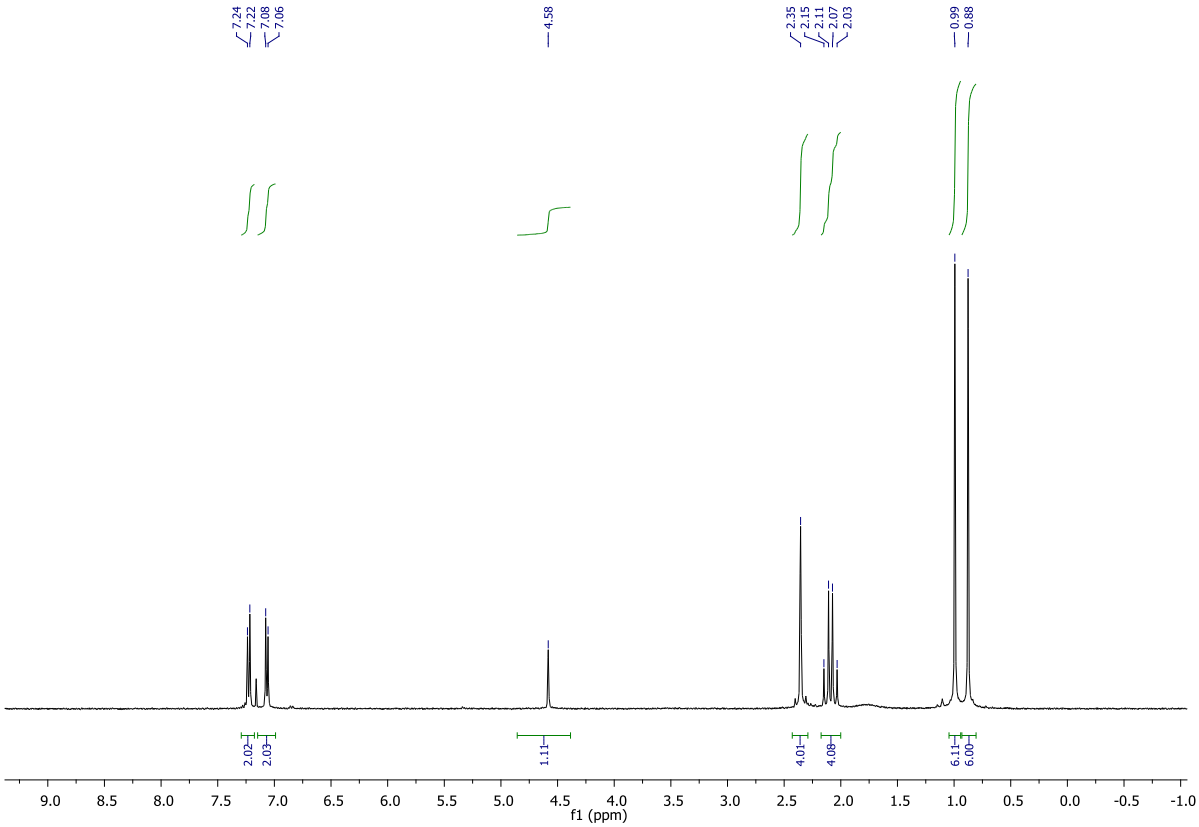




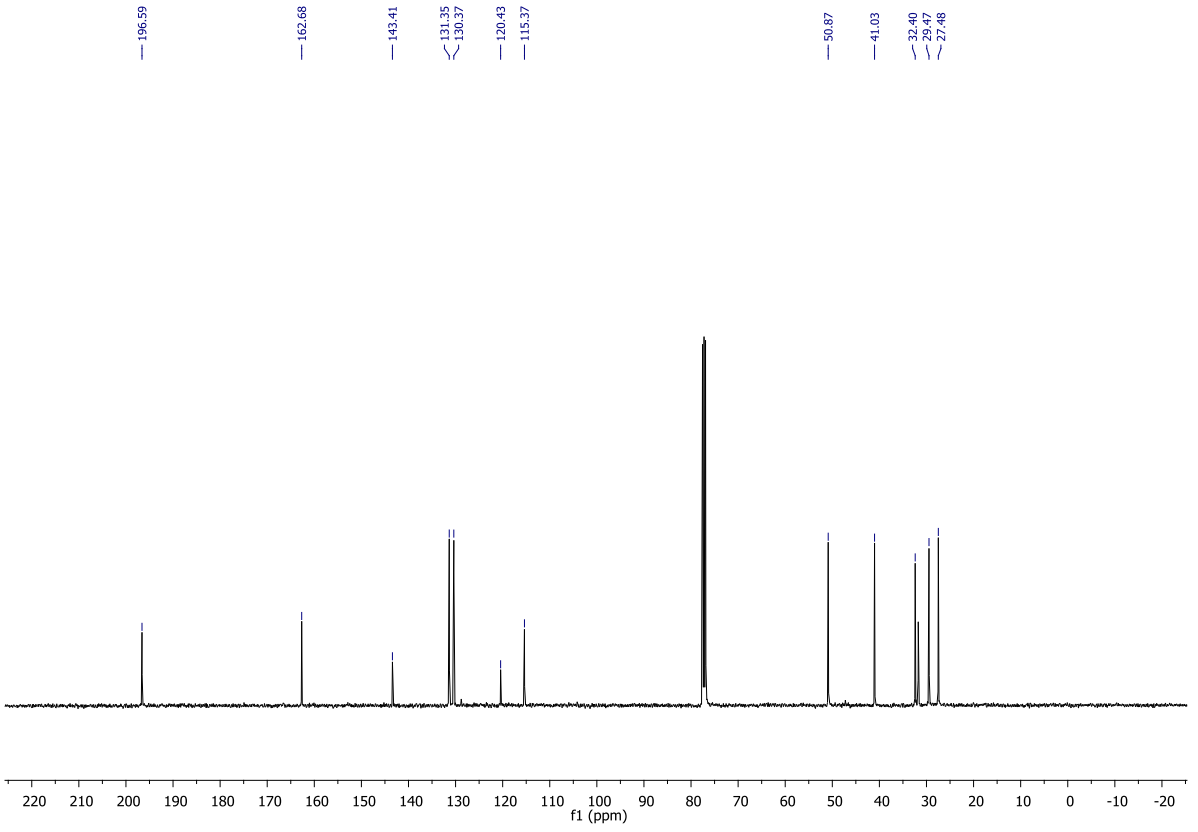
Gas chromatogram of 3,3,6,6-Tetramethyl-9-(4-bromophenyl)-1,8-dioxooctahydroxanthene (X7)



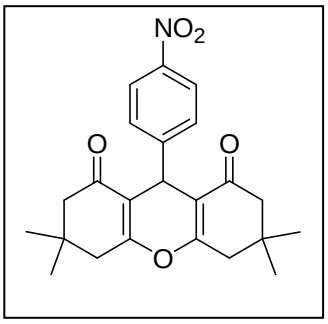
Mass spectrum of 3,3,6,6-Tetramethyl-9-(4-bromophenyl)-1,8-dioxooctahydroxanthene (X7)

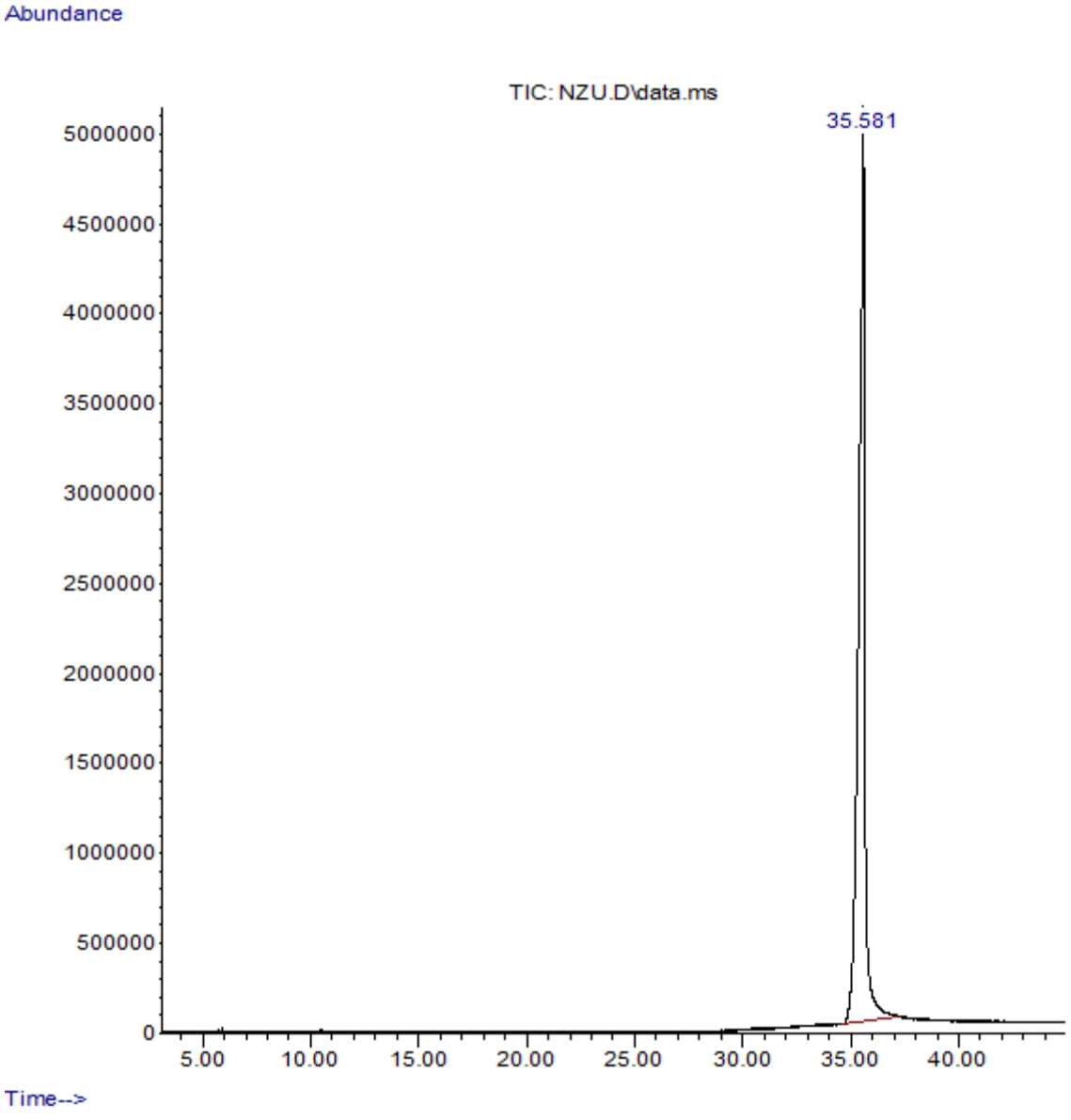


^{1}H NMR spectrum of 3,3,6,6-Tetramethyl-9-(4-bromophenyl)-1,8-dioxooctahydroxanthene (X7)

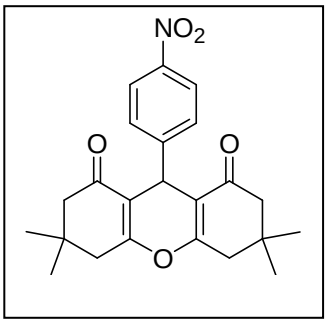


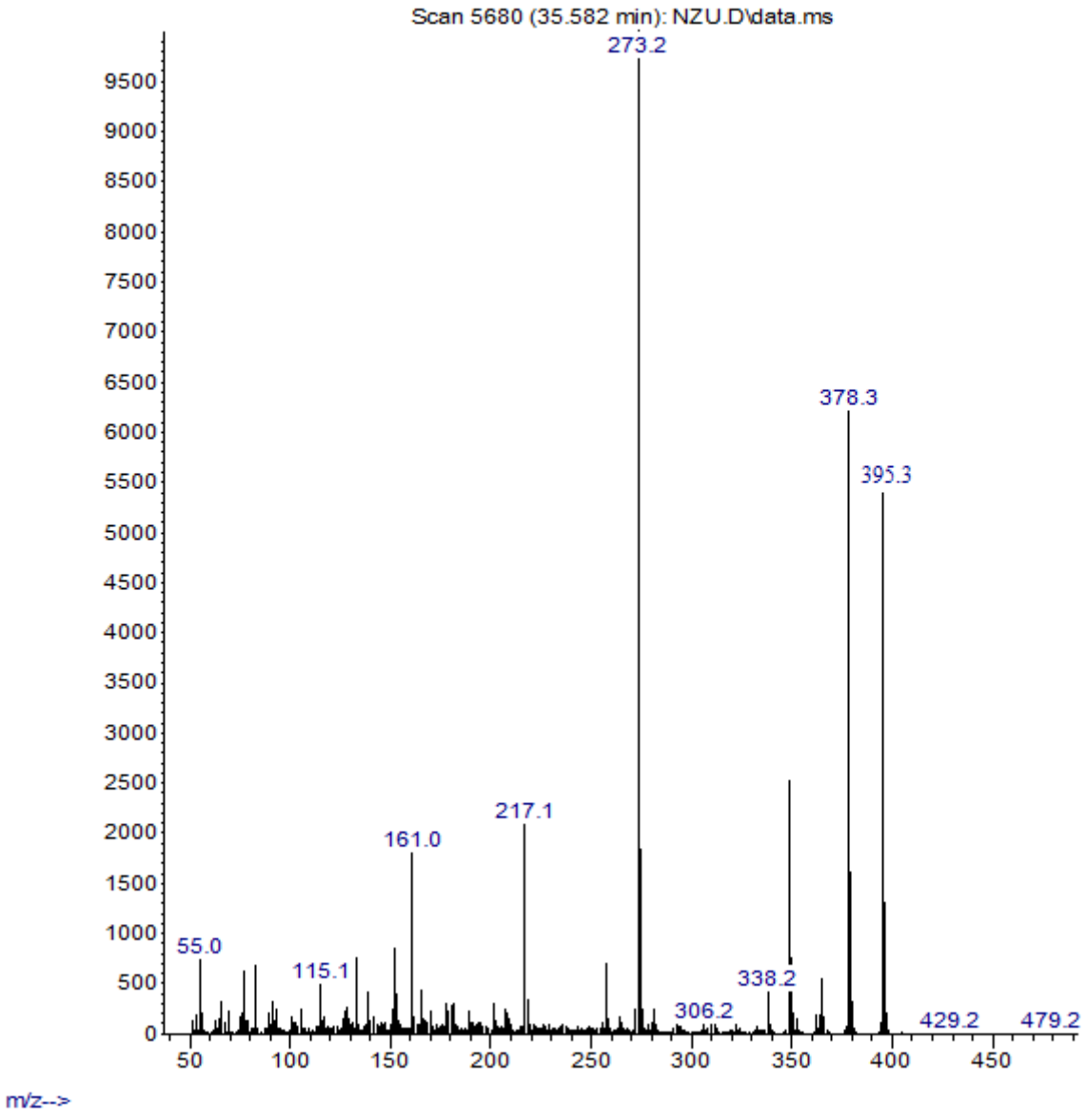
^{13}C NMR spectrum of 3,3,6,6-Tetramethyl-9-(4-bromophenyl)-1,8-dioxooctahydroxanthene (X7)



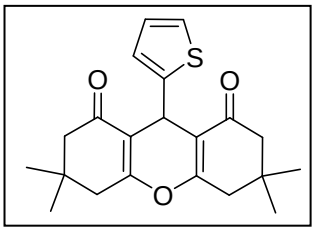


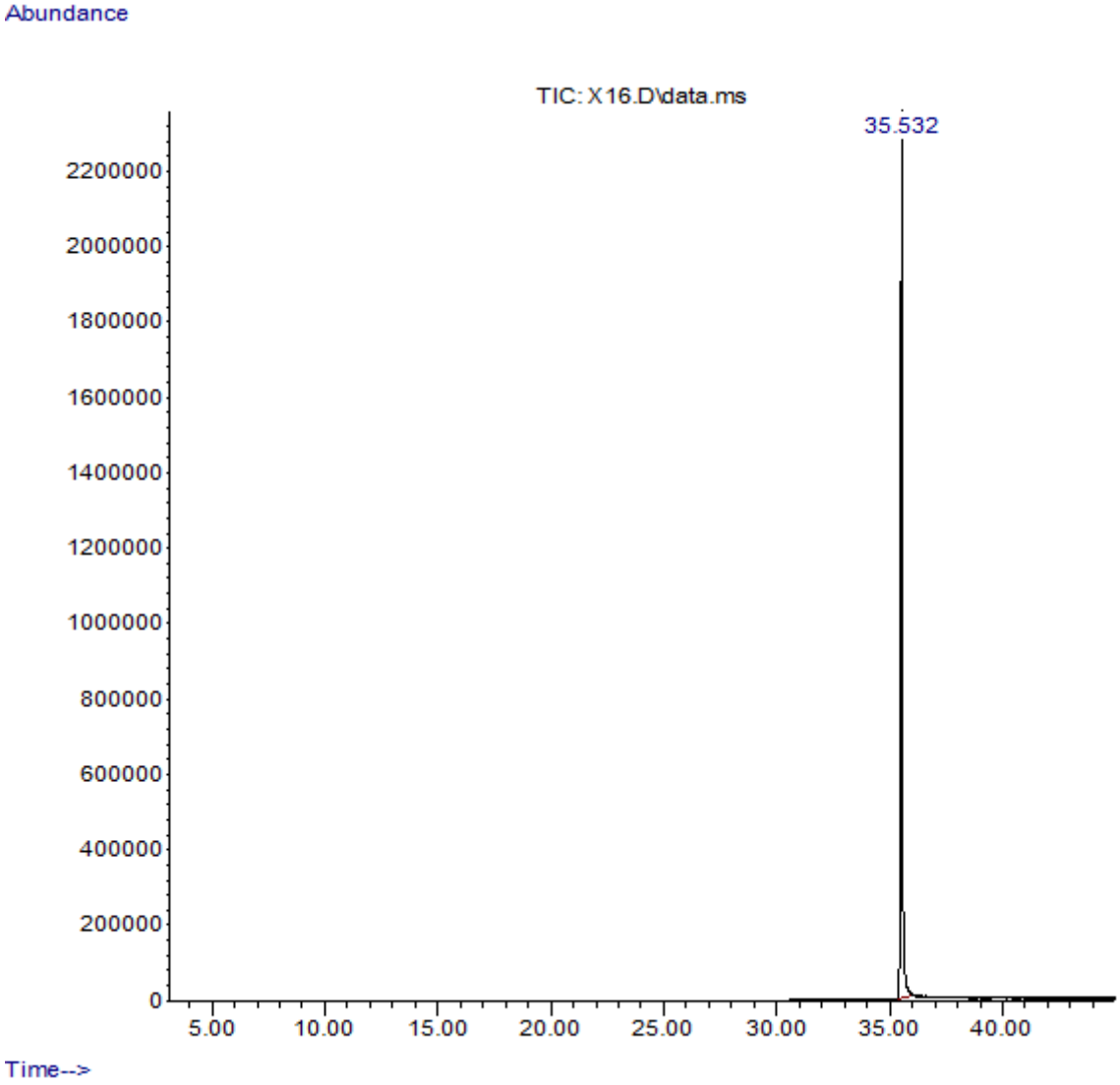
Gas chromatogram of 3,3,6,6-Tetramethyl-9-(4-nitrophenyl)-1,8-dioxooctahydroxanthene (X8)



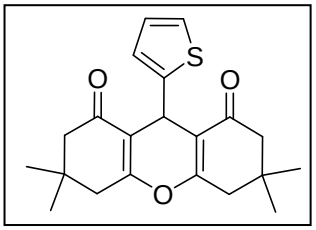


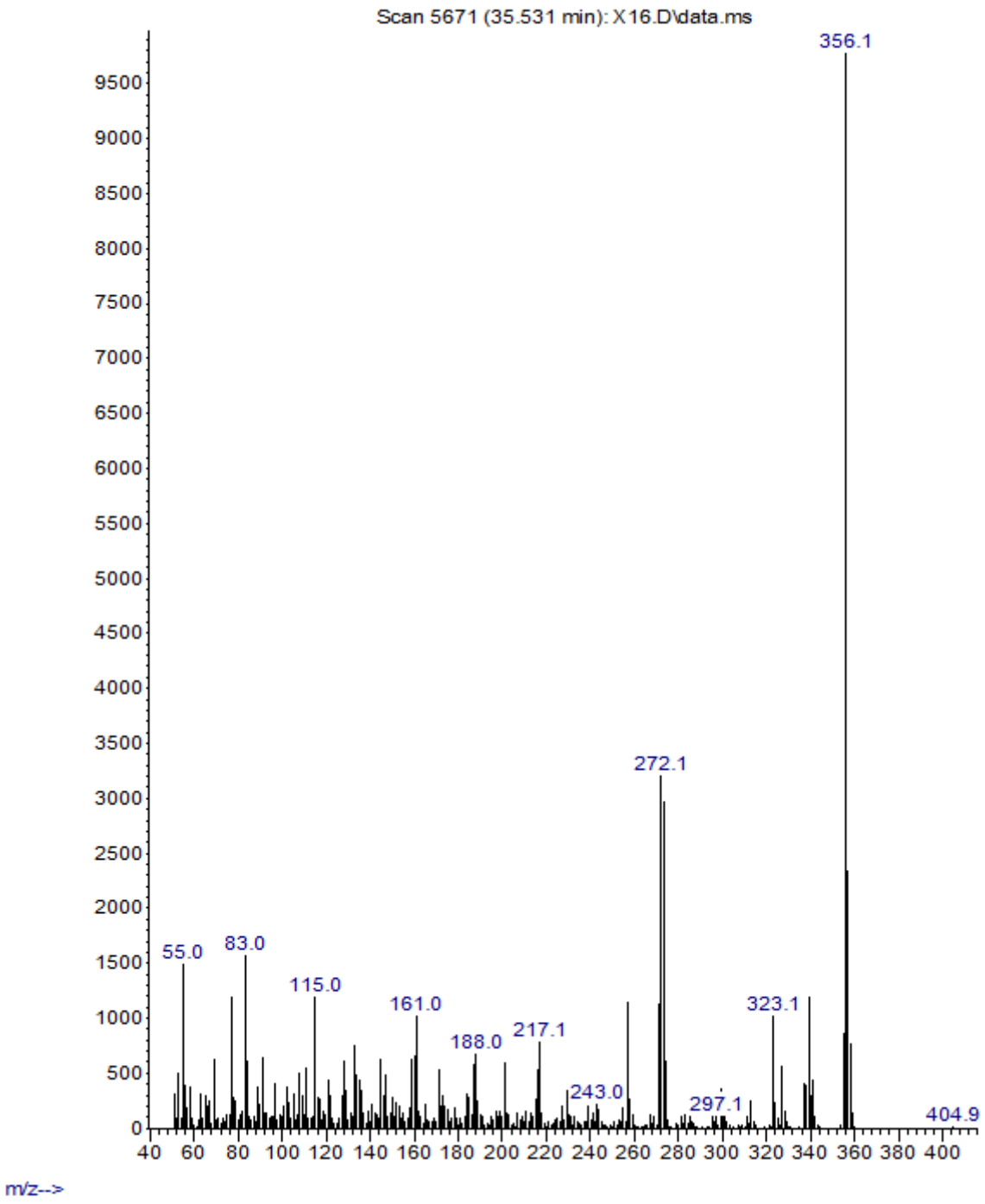
Mass spectrum of 3,3,6,6-Tetramethyl-9-(4-nitrophenyl)-1,8-dioxooctahydroxanthene (X8)



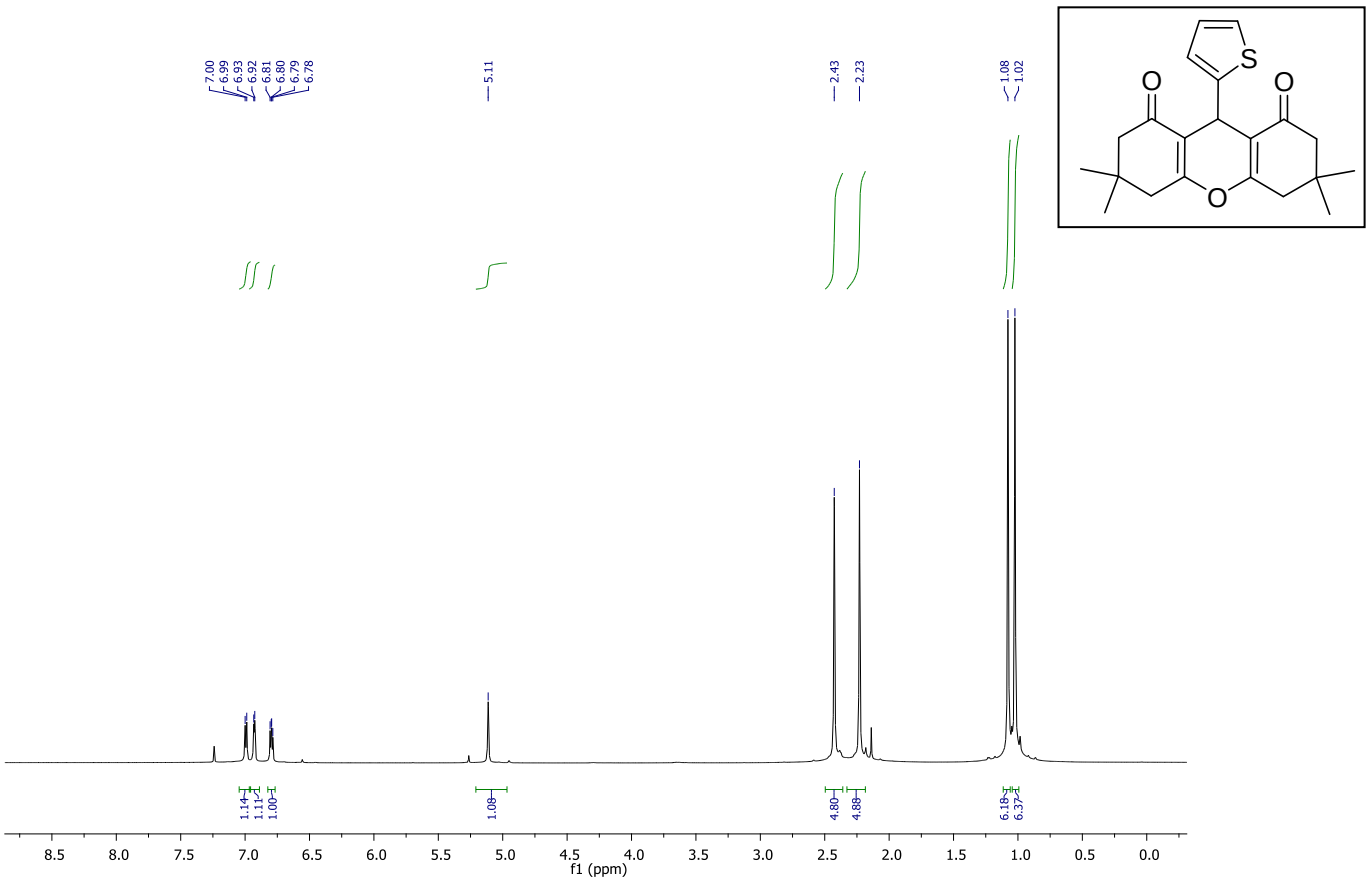


Gas chromatogram of 3,3,6,6-Tetramethyl-9-(2-Thienyl)-1,8-dioxooctahydroxanthene (X10)

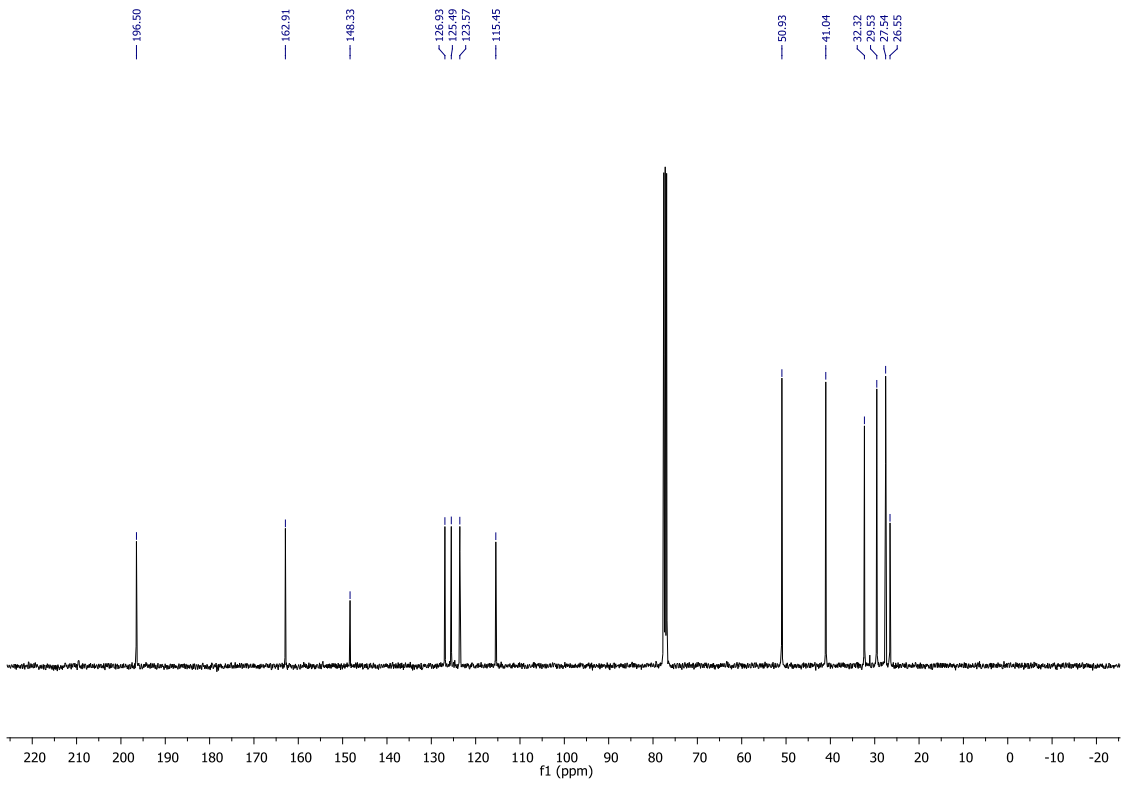




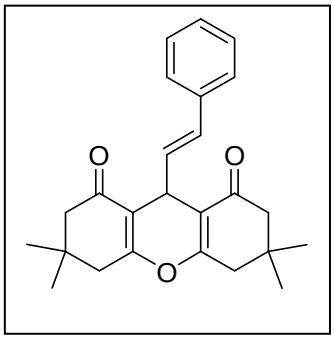
Mass spectrum of 3,3,6,6-Tetramethyl-9-(2-Thienyl)-1,8-dioxooctahydroxanthene (X10)

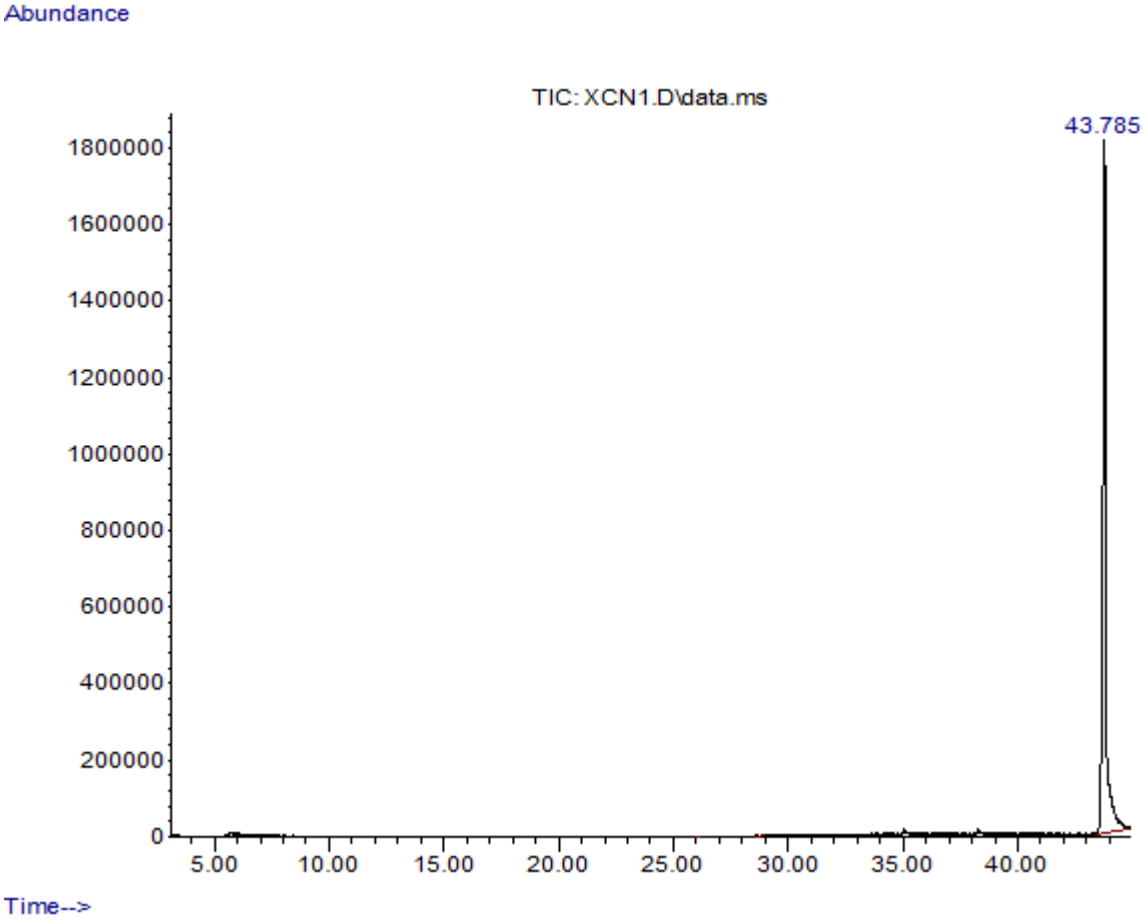


^{1}H NMR spectrum of 3,3,6,6-Tetramethyl-9-(2-Thienyl)-1,8-dioxooctahydroxanthene (X10)

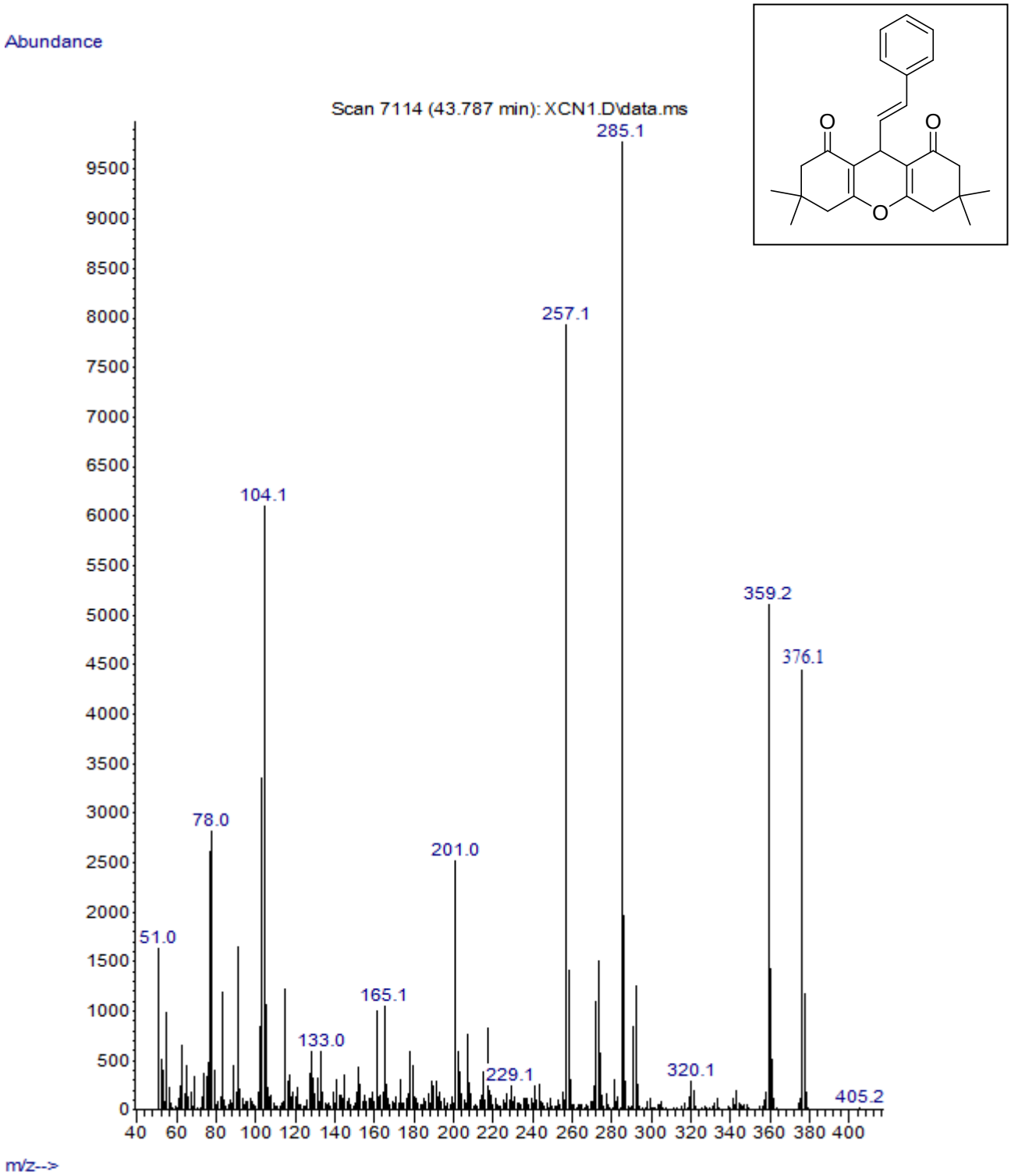


^{13}C NMR spectrum of 3,3,6,6-Tetramethyl-9-(2-Thienyl)-1,8-dioxooctahydroxanthene (X10)

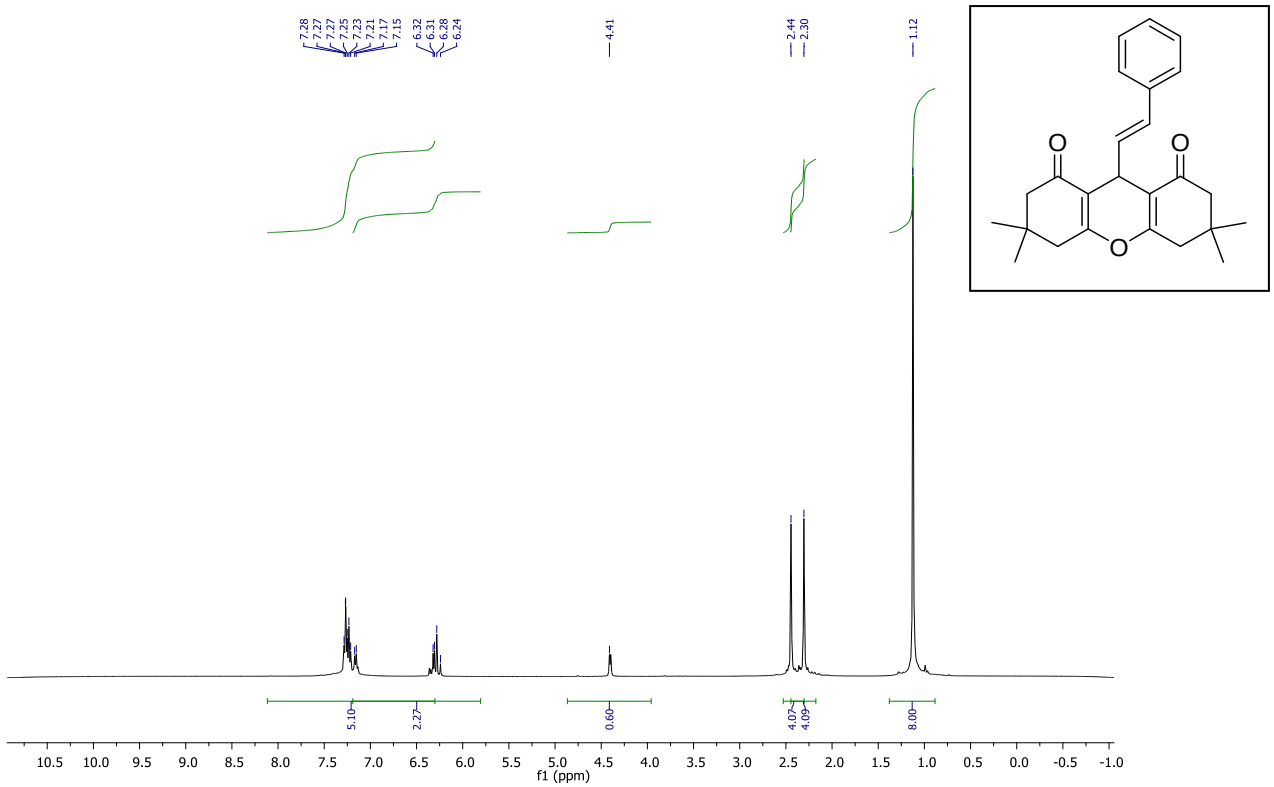




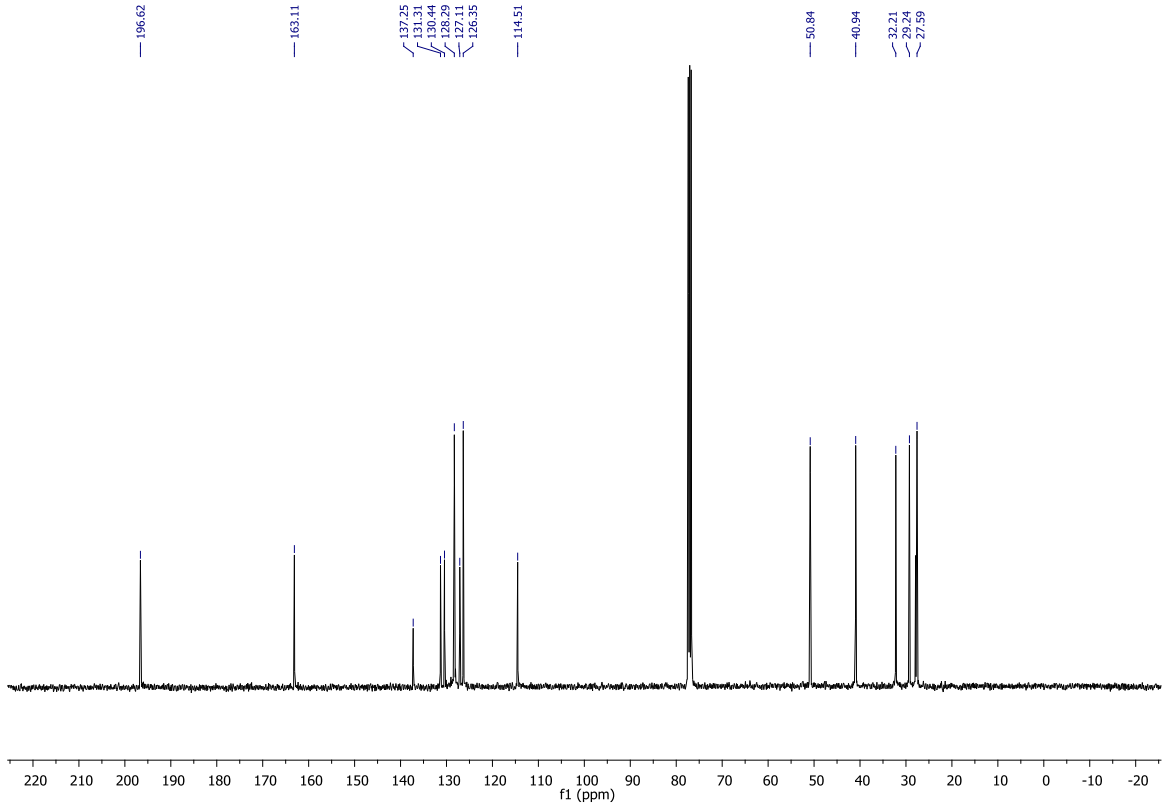
Gas chromatogram of 3,3,6,6-Tetramethyl-9-(2- phenylethylene)-1,8-dioxooctahydroxanthene (X11)



Mass spectrum of 3,3,6,6-Tetramethyl-9-(2- phenylethylene)-1,8-dioxooctahydroxanthene (X11)



^{1}H NMR spectrum of 3,3,6,6-Tetramethyl-9-(2- phenylethylene)-1,8-dioxooctahydroxanthene (X11)



^{13}C NMR spectrum of 3,3,6,6-Tetramethyl-9-(2- phenylethylene)-1,8-dioxooctahydroxanthene (X11)

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