Supporting Information

A Maitland-Japp Inspired Synthesis of Dihydropyran-4-ones and their Stereoselective Conversion to Functionalised Tetrahydropyran-4-ones

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1. General Experimental

All reactions were carried out under N_2 unless otherwise specified. IR analyses were carried out on a ThermoNicolet IR100 FT-IR spectrometer using NaCl plates. Nuclear magnetic resonance spectra were recorded on a Jeol ECX-400 MHz, Jeol ECS-400 MHz or Bruker AV-500 MHz spectrometer at ambient temperature; chemical shifts are quoted in parts per million (ppm) and were referenced as follows: chloroform-d, δ_H 7.26 ppm, δ_C 77.0 ppm; benzene-d, δ_H 7.15 ppm, δ_C 128.02 ppm. Mass spectrometry was performed by the University of York mass spectrometry service using ESI ionization. Microanalysis was performed by the microanalytical unit at the University of York. Melting points were determined using a Stuart SMP3 apparatus and remain uncorrected. Optical rotations were recorded at ambient temperature using a JASCO DIP-370 digital polarimeter. Thin layer chromatography was performed on glass-backed plates coated with Merck Silica gel 60 F254, and developed using ultraviolet light and ethanolic anisaldehyde, acidic aqueous ceric ammonium molybdate or basic aqueous potassium permanganate. Liquid chromatography was performed using forced flow (flash column) with the solvent systems indicated. The stationary phase was silica gel 60 (220-240 mesh) supplied by Fluorochem or silica gel Merck TLC grade 11695 supplied by Sigma-Aldrich. Dry solvents were obtained from a PureSlov PS-MD-7 solvent tower or distilled as follows: dichloromethane and toluene from calcium hydride, THF from sodium/benzophenone ketyl, diethyl ether from lithium aluminium hydride. All other solvents and reagents were used as received from commercial suppliers.

2. Experimental Procedures and Compound Characterisation

Methyl 6-ethyl-2-methyl-4-oxo-5,6-dihydro-2H-pyran-3-carboxylate (10b).
Oil; υ_{max} (film) 2928, 2903, 2840, 1705, 1690, 1652, 1568, 1413, 1378, 1325, 1289, 1246, 1197, 1154, 1123, 1100, 1065, 1023 cm^{-1}; δ_H (400MHz, C_6D_6) 3.55 (3H, s), 3.46 (1H, m), 1.94 (1H, dd, J = 16.2, 4.0 Hz), 1.88 (3H, s), 1.86 (1H, dd, J = 16.2, 13.1 Hz), 1.16 (1H, m), 0.99 (1H, m) and 0.53 (3H, t, J = 7.3 Hz) ppm; δ_C (100 MHz, C_6D_6) 187.0, 175.2, 166.5, 113.2, 80.0, 51.6, 40.4, 27.2, 19.6 and 8.9 ppm; *m/z* (ESI+) 221 $(M + Na)^+$. (Found 221.0784 $(M + Na)^+$. $C_{10}H_{14}NaO_4$ requires 221.0784).

Methyl 2-methyl-4-oxo-6-propyl-5,6-dihydro-2H-pyran-3-carboxylate (10c).
Oil; υ_{max} (film) 2960, 2875, 1772, 1700, 1684, 1576, 1559, 1457, 1399, 1340, 1265, 1207, 1084 cm^{-1}; δ_H (400MHz, C_6D_6) 3.50 (1H, m), 3.47 (3H, s), 1.88 (1H, dd, J = 16.5, 3.7 Hz), 1.80 (3H, s), 1.79 (1H, dd, J = 16.5, 13.1 Hz), 1.14-0.77 (4H, m) and 0.54 (3H, t, J = 7.3 Hz) ppm; δ_C (100 MHz, C_6D_6) 187.0, 175.2, 166.5, 113.3, 78.8, 51.6, 40.9, 36.1, 19.6, 18.0 and 13.7 ppm; *m/z* (ESI+) 235 $(M + Na)^+$, 213 $(M + H)^+$, 181 $(M - CH_3OH)^+$. (Found 235.0937 $(M + Na)^+$. $C_{11}H_{16}NaO_4$ requires 235.0941).

Methyl 6-isopropyl-2-methyl-4-oxo-5,6-dihydro-2H-pyran-3-carboxylate (10d).
Oil; υ_{max} (film) 2966, 1734, 1684, 1594, 1436, 1399, 1347, 1266, 1108, 1086 cm^{-1}; δ_H (400MHz, C_6D_6) 3.52 (3H, s), 3.30 (1H, m), 1.92 (1H, d, J = 1.8 Hz), 1.92 (1H, d, J = 9.2 Hz), 1.84 (3H, s), 1.30 (1H, m), 0.53 (3H, d,

J = 7.0 Hz) and 0.47 (3H, d, *J* = 7.0 Hz) ppm; δ_C (100 MHz, C_6D_6) 187.0, 175.1, 166.3, 113.0, 83.1, 51.4, 37.8, 31.2, 19.3, 17.2 and 16.9 ppm; *m/z* (ESI+) 235 $(M + Na)^+$, 213 $(M + H)^+$, 181 $(M - CH_3OH)^+$. (Found 235.0945 $(M + Na)^+$. $C_{11}H_{16}NaO_4$ requires 235.0941).

Methyl 6-(furan-2-yl)-2-methyl-4-oxo-5,6-dihydro-2H-pyran-3-carboxylate (10e).
Oil; υ_{max} (film) 3165, 2950, 2925, 1705, 1667, 1580, 1381, 1339 cm^{-1}; δ_H (400MHz, C_6D_6) 6.86 (1H, dd, *J* = 1.8, 0.8 Hz), 5.86 (1H, d, *J* = 3.3 Hz), 5.84 (1H, dd, *J* = 3.3, 1.8 Hz), 4.56 (1H, dd, *J* = 12.5, 3.9 Hz), 3.48 (3H, s), 2.51 (1H, dd, *J* = 16.6, 12.5 Hz), 2.13 (1H, dd, *J* = 16.6, 3.9 Hz) and 1.78 (3H, s) ppm; δ_C (100 MHz, C_6D_6) 185.6, 174.4, 165.9, 150.0, 143.2, 113.3, 110.3, 109.5, 72.8, 51.4, 38.4 and 19.3 ppm; *m/z* (ESI+) 259 $(M + Na)^+$, 237 $(M + H)^+$, 205 $(M - CH_3OH)^+$. (Found 259.0577 $(M + Na)^+$. $C_{12}H_{12}NaO_5$ requires 259.0577).

Methyl 6-(benzyloxymethyl)-2-methyl-4-oxo-5,6-dihydro-2H-pyran-3-carboxylate (10f).
Oil; υ_{max} (film) 3031, 2952, 2925, 2858, 1737, 1601 cm^{-1}; δ_H (400 MHz, C_6D_6) 7.36-7.06 (5H, m), 4.16 (1H, d, *J* = 12.0 Hz), 4.11 (1H, d, *J* = 12.0 Hz), 3.79 (1H, dddd, *J* = 13.5, 5.0, 3.5, 3.5 Hz), 3.54 (3H, s), 3.03 (1H, dd, *J* = 10.5, 3.5 Hz), 2.97 (1H, dd, *J* = 10.5, 5.0 Hz), 2.26 (1H, dd, *J* = 16.5, 13.5 Hz), 1.99 (1H, dd, *J* = 16.5, 3.5, Hz) and 1.87 (3H, s) ppm; δ_C (100 MHz, C_6D_6) 186.6, 175.1, 166.4, 138.2, 128.6, 128.1, 127.9, 113.3, 78.0, 73.2, 70.4, 51.7, 37.4 and 19.6 ppm; *m/z* (ESI+) 313 $(M + Na)^+$, 291 $(M + H)^+$. (Found 291.1228 $(M + H)^+$. $C_{16}H_{19}O_5$ requires 291.1227).

Methyl 6-(2-(benzyloxy)ethyl)-2-methyl-4-oxo-5,6-dihydro-2H-pyran-3-carboxylate (10g).
Oil; υ_{max} (film) 2906, 2880, 2822, 1706, 1652, 1570, 1413, 1377, 1326, 1246, 1182, 1064 cm^{-1}; δ_H (400 MHz, C_6D_6) 7.18-7.05 (5H, m, Ar), 4.17 (1H, d, *J* = 12.3 Hz, H-12), 4.12 (1H, d, *J* = 12.3 Hz, H-12), 4.02 (1H, m, H-6), 3.51 (3H, s, H-9), 3.14 (1H, m, H-11), 3.06 (1H, m, H-11), 2.06 (1H, dd, *J* = 16.5, 3.5 Hz, H-5_{eq}), 1.92 (1H, dd, *J* = 16.5, 13.2 Hz, H-5_{ax}), 1.83 (3H, s, H-7), 1.54 (1H, m, H-10) and 1.37 (1H, m, H-10) ppm; δ_C (100 MHz, C_6D_6) 186.8 (C-4), 175.2 (C-2), 166.5 (C-8), 138.8 (Ar), 128.6 (Ar), 127.8 (Ar), 113.3 (C-3), 76.6 (C-6), 73.0 (C-12), 65.2 (C-11), 51.7 (C-9), 40.9 (C-5), 34.5 (C-10) and 19.7 (C-7) ppm; *m/z* (ESI+) 327 $(M + Na)^+$, 305 $(M + H)^+$. (Found 305.1386 $(M + H)^+$. $C_{17}H_{21}O_5$ requires 305.1384).

Isopropyl 2-methyl-4-oxo-6-phenyl-5,6-dihydro-2H-pyran-3-carboxylate (10h).
Oil; Mp: 58 – 59 °C; υ_{max} (film) 2981, 1729, 1675, 1602 cm^{-1}; δ_H (400 MHz, C_6D_6) 7.02-7.06 (3H, m), 6.88-6.91 (2H, m), 5.27 (1H, sep, *J* = 6.5 Hz), 4.58 (1H, dd, *J* = 14.0, 4.0 Hz), 2.31 (1H, dd, *J* = 16.5, 14.0 Hz), 2.19 (1H, dd, *J* = 16.5, 4.0 Hz), 1.93 (3H, s), 1.23 (3H, d, *J* = 6.5 Hz), 1.22 (3H, d, *J* = 6.5 Hz) ppm; δ_C (100 MHz, C_6D_6) 184.7, 172.1, 163.5, 136.2, 126.9, 126.7, 124.5, 112.5, 78.7, 66.6, 40.6, 20.0, 19.9, 17.5 ppm. *m/z* (ESI+) 297 $(M + Na)^+$, 275 $(M + H)^+$, 233 $(M - C_3H_7)^+$. (Found 275.1275 $(M + H)^+$. $C_{16}H_{19}O_4$ requires 275.1278).

Isopropyl 6-cyclohexyl-2-methyl-4-oxo-5,6-dihydro-2H-pyran-3-carboxylate (10i).
Pale white solid; Mp: 48.2 – 49.3 °C. υ_{max} 2927, 2854, 1730, 1679, 1602, 1450, 1400, 1269, 1080 cm^{-1}; δ_H (400MHz, C_6D_6) 5.26 (1H, sept, *J* = 6.1 Hz), 3.43 (1H, ddd, *J* = 8.6, 8.6, 6.1 Hz), 2.02 (1H, d, *J* = 1.5 Hz), 1.99 (1H, s), 1.92 (3H, s), 1.54-1.44 (4H, m), 1.21 (6H, d, *J* = 6.1 Hz), 1.09 (1H, m), 1.00-0.89 (4H, m) and 0.70-0.60 (2H, m) ppm; δ_C (100 MHz, C_6D_6) 187.5, 174.2, 165.9, 114.1, 82.9, 68.4, 41.1, 38.3, 27.8, 26.4, 25.9, 21.9 and 19.4 ppm; *m/z* (ESI+) 303 $(M + Na)^+$, 281 $(M + H)^+$, 221 $(M - C_3H_7O)^+$. (Found 303.1563 $(M + Na)^+$. $C_{16}H_{24}NaO_4$ requires 303.1567). Anal. Calcd. for $C_{16}H_{24}O_4$: C, 68.55; H, 8.63. Found C, 68.62; H, 8.62.

Isopropyl 2-methyl-4-oxo-6-propyl-5,6-dihydro-2H-pyran-3-carboxylate (10j).
Oil; υ_{max} (film) 2964, 2936, 2875, 1729, 1678, 1601, 1466, 1400, 1338, 1266, 1080 cm^{-1}; δ_H (400MHz, C_6D_6) 5.24 (1H, sept, *J* = 6.1 Hz), 3.61 (1H, m), 1.96 (1H, dd, *J* = 16.3, 4.2 Hz), 1.92-1.88 (4H, m), 1.21 (3H, d, *J* = 6.1 Hz), 1.19 (3H, d, *J* = 6.1 Hz), 1.19-0.81 (4H, m) and 0.63 (3H, t, *J* = 7.3 Hz) ppm; δ_C (100 MHz, C_6D_6) 187.1, 174.0, 165.6, 114.1, 78.7, 68.4, 40.9, 36.2, 21.9, 21.8, 19.4, 18.0 and 13.7 ppm; *m/z* (ESI+) 263 $(M + Na)^+$, 241 $(M + H)^+$, 199 $(M - C_3H_7)^+$, 181 $(M - C_3H_7O)^+$. (Found 241.1437 $(M + H)^+$. $C_{13}H_{21}O_4$ requires 241.1434).

(*E*)-Isopropyl 2-methyl-4-oxo-6-(prop-1-enyl)- 5,6-dihydro-2H-pyran-3-carboxylate (10k).
Pale yellow solid; Mp: 46.6 – 47.7 °C. υ_{max} 2980, 2939, 1728, 1677, 1598, 1398, 1368, 1264, 1055, 968 cm^{-1}; δ_H (400MHz, C_6D_6) 5.30-5.19 (2H, m), 5.10 (1H, ddq, *J* = 15.2, 7.0, 1.3 Hz), 4.09 (1H, m), 2.09 (1H, s), 2.07 (1H, d, *J* = 2.4 Hz), 1.93 (3H, s), 1.30 (3H, dd, *J* = 7.0, 1.3 Hz), 1.20 (3H, d, *J* = 6.0 Hz) and 1.19 (3H, d, *J* = 6.0 Hz) ppm; δ_C (100 MHz, C_6D_6) 189.7, 173.9, 165.5, 130.4, 128.2, 114.2, 79.4, 68.4, 41.1, 21.9, 21.8, 19.5 and 17.5 ppm; *m/z* (ESI+) 261 $(M + Na)^+$, 239 $(M + H)^+$, 197 $(M - C_3H_7)^+$, 179 $(M - C_3H_7O)^+$. (Found 239.1281 $(M + H)^+$. $C_{13}H_{19}O_4$ requires 239.1278). Anal. Calcd. for $C_{13}H_{18}O_4$: C, 65.53; H, 7.61. Found C, 65.48; H, 7.60.

General procedure for the synthesis of 2-phenyl dihydropyrans

N,N-dimethylbenzamide dimethyl acetal (1.14 mL, 6.13 mmol) was added to a stirred solution of δ-hydroxy-β-ketoester (0.61 mmol) in dry toluene (3.2 mL) at room temperature. The solution was stirred at room temperature and monitored by TLC. Upon completion of the reaction, the solvent was removed *in vacuo*. Purification by flash column chromatography (petroleum ether – ethyl acetate) afforded the product.

Isopropyl 2-phenyl-6-propyl-4-oxo-5,6-dihydro-2H-pyran-3-carboxylate (10l).
Yellow solid; Mp: 80.9 – 81.2 °C. υ_{max} 2963, 2935, 2874, 1725, 1668, 1591, 1567, 1380, 1254, 1104, 1069 cm^{-1}; δ_H (400MHz, C_6D_6) 7.70-7.68 (2H, m) 7.06-7.02 (3H, m), 5.14 (1H, sept, *J* = 6.2 Hz), 3.77 (1H, m), 2.02 (1H, d, *J* = 2.2 Hz), 2.00 (1H, d, *J* = 8.8 Hz), 1.31-1.13 (2H, m), 1.06 (3H, d, *J* = 6.2 Hz), 1.05-0.91 (2H, m), 0.96 (3H, d, *J* = 6.2 Hz) and 0.66 (3H, t, *J* = 7.3 Hz) ppm; δ_C (100 MHz, C_6D_6) 188.0, 169.3, 165.3, 134.0, 131.0, 128.3, 114.2, 78.9, 68.3, 40.7, 36.1, 21.4, 21.1, 18.0 and 13.5 ppm; *m/z* (ESI+) 325 $(M + Na)^+$, 303 $(M + H)^+$, 243 $(M - C_3H_7O)^+$. (Found 325.1411 $(M + Na)^+$. $C_{18}H_{22}NaO_4$ requires 325.1410). Anal. Calcd. for $C_{18}H_{22}O_4$: C, 71.50; H, 7.33. Found C, 71.23; H, 7.40.

(*E*)-Isopropyl 2-phenyl-6-(prop-1-enyl)-4-oxo-5,6-dihydro-2H-pyran-3-carboxylate (10m).
Pale orange solid. Mp: 102.7 – 104.7 °C. υ_{max} 3059, 2981, 2920, 2852, 1725, 1668, 1590, 1566, 1379, 1263, 1104, 1047 cm^{-1}; δ_H (400MHz, C_6D_6) 7.72-7.69 (2H, m), 6.99-6.95 (3H, m), 5.32 (1H, dqd, *J* = 15.3, 6.4, 0.9 Hz), 5.19 (1H, ddq, *J* = 15.3, 6.4, 1.2 Hz), 5.14 (1H, sept, *J* = 6.4 Hz), 4.23 (1H, m), 2.17 (1H, d, *J* = 1.8 Hz), 2.15 (1H, s), 1.32 (3H, dd, *J* = 6.4, 1.2 Hz), 1.05 (3H, d, *J* = 6.4 Hz) and 0.96 (3H, d, *J* = 6.4 Hz) ppm; δ_C (100 MHz, C_6D_6) 187.7, 169.2, 165.3, 133.8, 131.1, 130.6, 128.4, 114.3, 79.5, 68.4, 40.8, 21.4, 21.1 and 17.3 ppm; *m/z* (ESI+) 323 $(M + Na)^+$, 301 $(M + H)^+$. (Found 301.1434 $(M + H)^+$. $C_{18}H_{21}O_4$ requires 301.1434). Anal. Calcd. for $C_{18}H_{20}O_4$: C, 71.98; H, 6.71. Found C, 73.30; H, 7.72.

Isopropyl 6-isopropyl-2-phenyl-4-oxo-5,6-dihydro-2H-pyran-3-carboxylate (10n).
Oil; υ_{max} (film) 3056, 2878, 1721, 1667, 1601, 1565, 1491, 1450, 1386, 1341, 1315, 1266, 1148, 1106, 1071, 967, 937, 897, 841, 823 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 7.63-7.38 (5H, m), 4.99 (1H, sept, *J* = 6.2 Hz), 4.38 (1H, ddd, *J* = 14.1, 6.3, 3.3 Hz), 2.65 (1H, dd, *J* = 16.4, 14.1 Hz), 2.55 (1H, dd, *J* = 16.4, 3.3 Hz), 2.08 (1H, d sept, *J* = 6.8, 6.6 Hz), 1.16 (3H, d, *J* = 6.2 Hz), 1.11 (3H, d, *J* = 6.6 Hz), 1.06 (3H, d, *J* = 6.8 Hz), 0.90 (3H, d, *J* = 6.2 Hz) ppm; δ_C (400 MHz, $CDCl_3$) 189.7, 172.5, 165.5, 133.5, 131.9, 128.6, 128.5, 112.8, 84.4, 68.8, 38.5, 32.1, 21.8, 21.1 and 18.1 ppm; *m/z* (ESI+) 325 $(M + Na)^+$, 303 $(M + H)^+$. (Found 325.1411 $(M + Na)^+$. $C_{18}H_{22}NaO_4$ requires 325.1410).

Methyl 6-(2-(benzyloxy)ethyl)-2-phenyl-4-oxo-5,6-dihydro-2H-pyran-3-carboxylate (10o).
Oil; υ_{max} (film) 3055, 2868, 2306, 1729, 1669, 1590, 1564, 1435, 1384, 1345, 1100, 1065, 1030, 896 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 7.52-7.31 (10H, m), 4.89 (1H, m), 4.56 (2H, m), 3.80-3.62 (2H, m), 3.62 (3H, s), 2.65 (2H, m), 2.25-2.03 (2H, m) ppm; δ_C (400 MHz, $CDCl_3$) 192.6, 175.9, 170.2, 141.6, 136.7, 135.7, 132.2, 132.0, 131.5, 131.5, 115.9, 77.0, 68.9, 55.9, 44.6, 38.3 ppm; *m/z* (ESI+) 389 $(M + Na)^+$, 367 $(M + H)^+$. (Found 389.1359 $(M + Na)^+$. $C_{22}H_{22}NaO_5$ requires 389.1359).

Methyl 6-cyclohexyl-2-phenyl-4-oxo-5,6-dihydro-2H-pyran-3-carboxylate (10p).
Oil; υ_{max} (film) 3055, 2933, 2857, 2306, 1729, 1667, 1590, 1564, 1423, 1384, 1342, 1120, 1058, 896 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 7.56-7.40 (5H, m), 4.38(1H, ddd, *J* = 13.7, 6.1, 3.4 Hz), 3.62 (3H, s), 2.65 (1H, dd, *J* = 16.5,

13.7 Hz), 2.56 (1H, dd, *J*= 16.5, 3.4 Hz), 2.05-1.20 (11H, m) ppm; δ_C (400 MHz, $CDCl_3$) 189.6, 172.8, 166.6, 133.1, 132.0, 128.5, 128.3, 111.85, 83.8, 52.2, 41.4, 38.4, 28.2, 26.1, 25.7 ppm; *m/z* (ESI+) 337 $(M + Na)^+$, 315 $(M + H)^+$. (Found 337.1410 $(M + Na)^+$. $C_{19}H_{22}NaO_4$ requires 337.1410).

General procedure for orthoester cyclisations (themal heating)

A mixture of δ-hydroxy-β-ketoester (0.27 mmol), orthoester (0.28 mmol) and acetic anhydride (0.05 mL, 0.53 mmol) in toluene (2 mL) was heated at reflux for 1 hour. The mixture was then cooled and the solvent removed *in vacuo*. Flash column chromatography (petroleum ether – diethyl ether) gave the product.

General procedure for orthoester cyclisations (microwave heating)

A solution of δ-hydroxy-β-ketoester (0.13 mmol), orthoester (0.26 mmol) and acetic anhydride (0.044 mL, 0.43 mmol) in toluene (1.00 mL) was heated in microwave (100 pressure, 110 ^oC, 150 watt) for 15 minutes. The mixture was then cooled and the solvent removed *in vacuo*. Flash column chromatography (petroleum ether – ethyl acetate) gave the products.

Methyl 2-butyl-4-oxo-6-propyl-5,6-dihydro-2H-pyran-3-carboxylate (10q).
Oil; υ_{max} (film) 2914, 2890, 2829, 1701, 1652, 1567, 1439, 1413, 1368, 1323, 1243, 1190, 1151, 1060 cm^{-1}; δ_H (400MHz, C_6D_6) 3.63 (1H, m), 3.56 (3H, s), 2.45-2.30 (2H, m), 2.00 (1H, dd, J = 16.3, 4.4 Hz), 1.93 (1H, dd, J = 16.3, 12.5 Hz), 1.58-1.46 (2H, m), 1.26-0.88 (6H, m), 0.80 (3H, t, J = 7.3 Hz) and 0.64 (3H, t, J = 7.1 Hz) ppm; δ_C (100 MHz, C_6D_6) 187.5, 177.6, 166.7, 113.5, 78.8, 51.7, 41.0, 36.2, 33.1, 29.3, 22.6, 18.1, 13.8 and 13.7 ppm; m/z (ESI+) 277 $(M + Na)^+$, 255 $(M + H)^+$. (Found 277.1412 $(M + Na)^+$. $C_{14}H_{22}NaO_4$ requires 277.1410).

Methyl 2-butyl-6-isopropyl-4-oxo-5,6-dihydro-2H-pyran-3-carboxylate (10r).
Oil; υ_{max} (film) 2917, 2831, 1704, 1651, 1561, 1443, 1415, 1370, 1327, 1245, 1201, 1151, 1089, 1060 cm^{-1}; δ_H (400MHz, C_6D_6) 3.56 (3H, s), 3.34 (1H, m), 2.46-2.29 (2H, m), 1.99 (1H, s), 1.97 (1H, d, *J* = 5.1 Hz), 1.57-1.46 (2H, m), 1.36 (1H, m), 1.25-1.16 (2H, m), 0.80 (3H, t, *J* = 7.3 Hz), 0.60 (3H, d, *J* = 6.8 Hz) and 0.53 (3H, d, *J* = 6.8 Hz) ppm; δ_C (100 MHz, C_6D_6) 187.8, 177.8, 166.7, 113.4, 83.4, 51.8, 38.3, 33.1, 31.6, 29.2, 22.5, 17.6, 17.2 and 13.9 ppm; *m/z* (ESI+) 277 $(M + Na)^+$, 255 $(M + H)^+$. (Found 277.1407 $(M + Na)^+$. $C_{14}H_{22}NaO_4$ requires 277.1410).

Methyl 6-phenyl-2-butyl-4-oxo-5,6-dihydro-2H-pyran-3-carboxylate (10s).
Oil; υ_{max} (film) 2961, 2924, 2863, 1722, 1674, 1592, 1581, 1384, 1260, 1219, 1158, 1068, 756, 698 cm-1; δ_H (400MHz, $CDCl_3$) 7.44-7.34 (5H, m), 5.41 (1H, dd, *J* = 16.0, 4.0 Hz), 3.84 (3H, s), 2.88 (1H, dd, *J* = 16.0, 12.0 Hz), 2.71 (1H, dd, *J* = 16.0, 4.0 Hz), 2.55 (2H, t, *J* = 8.0 Hz), 1.68- 1.60 (2H, m), 1.42-1.33 (2H, m), 0.91 (3H, t, J = 8.0 Hz) ppm; δ_C (100 MHz, $CDCl_3$) 188.4, 180.6, 166.7, 137.4, 80.0, 52.4, 42.4, 33.6, 29.7, 17.6, 13.8 ppm; *m/z* (ESI^+) 311 $(M + Na)^+$, 289 $(M + H)^+$. (Found 311.1256 $(M + Na)^+$. $C_{17}H_{20}NaO_4$ requires 311.1259).

Methyl 6-(benzyloxymethyl)-2-butyl-4-oxo-5,6-dihydro-2H-pyran-3-carboxylate (10t).
Oil. υ_{max} (film) 2955, 2926, 2869, 1718, 1672, 1686, 1387, 1341, 1208, 1066, 804, 737, 698 cm^{-1}; δ_H (400MHz, $CDCl_3$) 7.31-7.24 (5H, m), 4.62 (1H, d, *J* = 12.12 Hz), 4.58 (1H, d, *J* = 12.12 Hz), 4.57- 4.53 (1H, m), 3.80 (3H, s), 3.73 (1H, dd, *J* = 10.9, 3.7 Hz), 3.68 (1H, dd, *J* = 10.9, 5.0 Hz), 2.72 (1H, dd, *J* = 16.72, 13.6 Hz), 2.51 (1H, dd, *J* = 16.7, 3.5 Hz), 2.49 (2H, t, *J* = 7.6 Hz,), 1.65-1.57 (2H, m), 1.42- 1.32 (2H, m), 0.92 (3H, t, *J* = 8.0 Hz) ppm; δ_C (100 MHz, $CDCl_3$) 188.4, 179.8, 166.7, 137.4, 128.5- 127.1, 112.34, 78.10, 73.5, 70.4, 52.2, 37.5, 33.4, 29.1, 22.50, 13.8 ppm; *m/z* (ESI^+) 355 $(M + Na)^+$, 333 $(M + H)^+$. (Found 355.1515 $(M + Na)^+$. $C_{19}H_{24}NaO_5$ requires 355.1521).

General Procedure for L-Selectride® reduction of dihydropyrans

A 1.0 M solution of L-Selectride® in THF (0.04 mL, 0.04 mmol) was added to a stirred solution of DHP (0.04 mmol) in THF (1 mL) at -78 °C. The mixture was stirred for 1 hour, then partitioned between Et_2O (10 mL) and

saturated aqueous $NHCl_4$ (10 mL). The aqueous layer was washed with Et_2O (10 mL) and the combined organic extracts were washed with brine (20 mL), dried over $MgSO_4$ and concentrated *in vacuo*. Purification by flash column chromatography (petroleum ether – diethyl ether or cyclohexane – ethyl acetate) afforded the product.

Methyl 2-methyl-4-oxo-6-phenyl-tetrahydro-2H-pyran-3-carboxylate (11a).
Oil (keto : enol 1 : 0.34); υ_{max} (film) 2954, 2933, 2873, 1748, 1717, 1653 cm^{-1}; nOe: H-2 - H-6 2.2%; δ_H (400 MHz, $CDCl_3$) 12.08 (1H, s, *enol*), 7.42-7.29 (5H, m, *enol*), 7.29-7.42 (5H, m), 4.75 (1H, dd, *J* = 11.5, 3.0 Hz), 4.67 (1H, m, *enol*), 4.57 (1H, dd , *J* = 10.5, 3.0, *enol*), 4.21 (1H, dq, *J* = 10.5, 6.0 Hz), 3.81 (3H, s), 3.35 (1H, d, *J* = 10.5 Hz), 3.18 (3H, s, *enol*), 2.72 (1H, dd, *J* = 14.5, 3.0 Hz), 2.61 (1H, dd, *J* = 14.5, 11.5 Hz) 2.61 (1H, m, *enol*), 2.47 (1H, dd, *J* = 17.0, 3.0 Hz, *enol*), 1.46 (3H, d, *J* = 6.0 Hz, *enol*) and 1.43 (3H, d, *J* = 6.0 Hz) ppm; δ_C (100 MHz, $CDCl_3$) 201.6, 170.0 (*enol*), 168.5, 141.0, 140.1, 128.7, 128.5, 128.3, 127.9, 126.0, 125.9, 125.6, 78.6, 77.2, 75.3, 74.0, 70.0, 64.3, 52.2, 51.4 (*enol*), 48.6, 36.9, 22.2 (*enol*) and 20.9 ppm; *m/z* (ESI+) 249 $(M + H)^+$. (Found 249.1113 $(M + H)^+$. $C_{14}H_{17}O_4$ requires 249.1121).

Methyl 6-ethyl-2-methyl-4-oxo-tetrahydro-2H-pyran-3-carboxylate (11b).
Oil (keto : enol 1 : 0.10); υ_{max} (film) 2929, 2893, 2835, 1720, 1691, 1416, 1318, 1292, 1243, 1203, 1178, 1113, 995 cm^{-1}; δ_H (400MHz, C_6D_6) 12.64 (1H, s, *enol*), 4.46 (1H, m, *enol*), 3.91 (1H, dq, *J* = 10.0, 6.1 Hz), 3.41 (3H, s), 3.25 (3H, s, *enol*), 3.04 (1H, m, *enol*), 2.98 (1H, m), 2.97 (1H, d, *J* = 10.0 Hz) 2.19-1.93 (2H, m, *enol*), 2.02 (1H, dd, *J* = 14.6, 2.1 Hz), 1.65 (1H, dd, *J* = 14.6, 12.5 Hz), 1.48 (3H, d, *J* = 5.5 Hz, *enol*), 1.31 (1H, m, *enol*), 1.27 (1H, m), 1.19 (3H, d, *J* = 6.1 Hz), 1.10 (1H, m, *enol*), 1.08 (1H, m), 0.79 (3H, t, *J* = 7.3 Hz, *enol*) and 0.68 (3H, t, *J* = 7.3 Hz) ppm; δ_C (100 MHz, C_6D_6) 201.3, 168.7, 77.8, 74.9, 64.6, 51.6, 46.5, 29.2, 20.8 and 9.5 ppm; *m/z* (ESI+) 223 $(M + Na)^+$. (Found 223.0942 $(M + Na)^+$. $C_{10}H_{16}NaO_4$ requires 223.0941).

Methyl 2-methyl-4-oxo-6-propyl-tetrahydro-2H-pyran-3-carboxylate (11c).
Oil (keto : enol 1 : 0.13); υ_{max} (film) 2959, 2933, 2873, 1747, 1718, 1657, 1618, 1438, 1335, 1261, 1126, 1035 cm^{-1}; nOe: H-2 – H-6 1.8%, H-3 – H-5_{ax} 0.7%, H-3 – H-7 0.9%, *enol* H-2 – H-6 1%; δ_H (400MHz, C_6D_6) 12.64 (1H, s, *enol*), 4.46 (1H, m, *enol*), 3.91 (1H, dq, *J* = 10.4, 5.8 Hz), 3.41 (3H, s), 3.25 (3H, s, *enol*), 3.16 (1H, m, *enol*), 3.10 (1H, m), 3.00 (1H, dd, *J* = 10.4, 0.9 Hz), 2.10 (1H, m, *enol*), 2.04 (1H, dd, *J* = 14.0, 2.4 Hz), 1.96 (1H, m, *enol*), 1.68 (1H, ddd, *J* = 14.0, 11.6, 0.9 Hz), 1.47 (3H, d, *J* = 6.1 Hz, *enol*), 1.33-0.98 (4H, m), 1.33-0.98 (4H, m, *enol*), 1.19 (3H, d, *J* = 6.1 Hz), 0.79 (3H, t, *J* = 7.0 Hz, *enol*) and 0.72 (3H, d, *J* = 7.0 Hz) ppm; δ_C (100 MHz, C_6D_6) 201.4, 171.7, 171.5, 168.8, 102.5 (*enol*), 77.4, 76.6, 75.1, 72.2 (*enol*), 68.9 (*enol*), 64.8, 51.8, 51.0 (*enol*), 47.0, 38.6, 38.0 (*enol*), 35.7 (*enol*), 22.7, 21.0, 18.8 (*enol*), 18.7, 14.3 (*enol*) and 14.2 ppm; *m/z* (ESI+) 237 $(M + Na)^+$, 215 $(M + H)^+$, 171. (Found 237.1095 $(M + Na)^+$. $C_{11}H_{18}NaO_4$ requires 237.1097).

Methyl 6-isopropyl-2-methyl-4-oxo-tetrahydro-2H-pyran-3-carboxylate (11d).
Oil (keto : enol 1 : 0.13); υ_{max} (film) 3365, 3043, 2988, 2915, 2833, 1721, 1691, 1453, 1416, 1322, 1246, 1124, 1102, 1019 cm^{-1}; nOe: H-2 – H-6 1.7%, H-3 – H-5_{ax} 0.6%, H-3 – H-7 0.9%, *enol* H-2 – H-6 1%; δ_H (400MHz, C_6D_6) 12.62 (1H, s, *enol*), 4.42 (1H, m, *enol*), 3.89 (1H, dq, *J* = 10.5, 5.8 Hz), 3.42 (3H, s), 3.27 (3H, s, *enol*), 2.97 (1H, dd, *J* = 10.5, 0.6 Hz), 2.84 (1H, ddd, *J* = 11.7, 5.8, 2.4 Hz), 2.83 (1H, m, *enol*), 2.15 (1H, m, *enol*), 2.07 (1H, dd, *J* = 14.0, 2.4 Hz), 1.99 (1H, m, *enol*), 1.72 (1H, ddd, *J* = 14.0, 11.7, 0.6 Hz), 1.56 (1H, m, *enol*), 1.47-1.37 (1H, m), 1.46 (3H, d, *J* = 6.7 Hz, *enol*), 1.18 (3H, d, *J* = 5.8 Hz), 0.88 (3H, d, *J* = 6.7 Hz, *enol*), 0.73 (3H, d, *J* = 7.0 Hz), 0.71 (3H, d, *J* = 6.7 Hz, *enol*) and 0.63 (3H, d, *J* = 7.0 Hz) ppm; δ_C (100 MHz, C_6D_6) 201.9, 171.9, 171.8, 168.9, 102.4 (*enol*), 81.4, 77.4 (*enol*), 75.0, 70.0 (*enol*), 64.7, 51.7, 51.0 (*enol*), 44.0, 43.3, 33.3, 32.9 (*enol*), 22.6 (*enol*), 21.0, 17.9 (*enol*), 17.4 (*enol*), 17.3 and 17.2 ppm; *m/z* (ESI+) 237 $(M + Na)^+$, 215 $(M + H)^+$, 171. (Found 237.1096 $(M + Na)^+$. $C_{11}H_{18}NaO_4$ requires 237.1097).

Methyl 6-(furan-2-yl)-2-methyl-4-oxo-tetrahydro-2H-pyran-3-carboxylate (11e).
Oil (keto : enol 1 : 0.20); υ_{max} (film) 3394, 3097, 2989, 2909, 1702, 1636, 1594, 1417, 1333, 1249, 1179, 1135, 1078 cm^{-1}; nOe H-2 – H-6 2.2%, H-3 – H-5_{ax} 0.8%, H-3 – H-7 0.9%, *enol* H-2 – H-6 1.0%; δ_H (400MHz, C_6D_6) 12.62 (1H, s, *enol*), 7.04 (1H, m, *enol*), 6.98 (1H, m), 6.06-6.00 (2H, m, *enol*), 5.98-5.92 (2H, m), 4.53 (1H, m, *enol*), 4.27 (1H, m, *enol*), 4.15 (1H, dd, *J* = 11.9, 2.9 Hz), 3.99 (1H, dq, *J* = 10.5, 6.1 Hz), 3.38 (3H, s), 3.22 (3H, s, *enol*), 3.02 (1H, d, *J* = 10.5Hz), 2.77 (1H, m, *enol*), 2.38 (1H, dd, *J* = 14.3, 11.9 Hz), 2.31 (1H, dd, *J* =

14.3, 2.9 Hz), 2.24 (1H, m, *enol*), 1.45 (1H, d, *J* = 6.1 Hz, *enol*) and 1.17 (3H, d, *J* = 6.1 Hz) ppm; δ_C (100 MHz, C_6D_6) 200.2, 171.7, 170.5, 168.6, 154.0 (*enol*), 153.0, 142.8, 142.6 (*enol*), 110.6, 110.5, 107.9, 107.5, 102.3 (*enol*), 75.0, 71.9, 70.3 (*enol*), 68.2 (*enol*), 64.5, 51.8, 51.1 (*enol*), 44.7, 33.7 (*enol*), 22.4 (*enol*) and 20.9 ppm; *m/z* (ESI+) 261 $(M + Na)^+$. (Found 261.0733 $(M + Na)^+$. $C_{12}H_{14}NaO_5$ requires 261.0733).

Methyl 6-(benzyloxymethyl)-2-methyl-4-oxo-tetrahydro-2H-pyran-3-carboxylate (11f).
Oil (keto : enol 1 : 0.15); υ_{max} (film) 2881, 2817, 1719, 1692, 1430, 1317, 1243, 1179, 1084 cm^{-1}; nOe: H-2 – H-6 2.6%, H-3 – $H\text{-}5_{ax}$ 1.0%, H-3 – H-7 1.3%; δ_H (400MHz, C_6D_6) 7.24-7.06 (5H, m), 7.24-7.06 (5H, m, *enol*), 4.47 (1H, m, *enol*), 4.34-1.26 (2H, m, *enol*), 4.25 (1H, d, *J* = 13.1 Hz) , 4.22 (1H, d, *J* = 13.1 Hz), 3.93 (1H, dq, *J* = 10.5, 5.8 Hz), 3.50 (1H, m, *enol*), 3.37 (3H, s), 3.37 (1H, m), 3.22 (3H, s, *enol*), 3.13 (1H, s), 3.13 (1H, s, *enol*), 3.12 (1H, s), 3.12 (1H, s, *enol*), 2.98 (1H, d, *J* = 10.5 Hz), 2.30 (1H, m, *enol*), 2.15 (1H, dd, *J* = 14.5, 2.8 Hz), 2.06 (1H, m, *enol*), 2.04 (1H, dd, *J* = 14.5, 11.5 Hz), 1.46 (3H, d, *J* = 6.2 Hz, *enol*) and 1.18 (3H, d, *J* = 5.8 Hz) ppm; δ_C (100 MHz, C_6D_6) 201.1, 171.6, 170.9, 168.5, 138.7, 128.4, 128.3, 128.1, 102.1 (*enol*), 76.0, 75.1, 73.4, 73.3, 72.6, 72.2, 71.8, 69.8, 64.4, 51.6, 50.8 (*enol*), 43.4, 32.5 (*enol*), 25.0 (*enol*) and 20.8 ppm; *m/z* (ESI+) 315 $(M + Na)^+$, 310, 293 $(M + H)^+$, 275. (Found 293.1385 $(M + H)^+$. $C_{16}H_{21}O_5$ requires 293.1384).

Methyl 6-(2-(benzyloxy)ethyl)-2-methyl-4-oxo-tetrahydro-2H-pyran-3-carboxylate (11g).
Oil (keto : enol 1 : 0.19); υ_{max} (film) 3420, 3030, 2925, 2863, 1745, 1718, 1656, 1617, 1452, 1337, 1261, 1197, 1101, 1041 cm^{-1}; nOe: H-2 – H-6 2%; δ_H (400MHz, C_6D_6) 12.63 (1H, s), 7.29-7.05 (5H, m), 7.29-7.05 (5H, m, *enol*), 4.45 (1H, m, *enol*), 4.30-4.25 (2H, m, *enol*), 4.25 (1H, d, *J* = 12.2 Hz), 4.20 (1H, d, *J* = 12.2 Hz), 3.91 (1H, dq, *J* = 10.5, 6.1Hz), 3.54 (1H, m, *enol*), 3.45 (1H, m), 3.40 (3H, s), 3.39-3.23 (2H, m), 3.39-3.23 (2H, m, *enol*), 3.23 (3H, s, *enol*), 2.97 (1H, dd, *J* = 10.5, 0.9 Hz), 2.16 (1H, m, *enol*), 2.11 (1H, dd, *J* = 14.3, 2.4 Hz), 2.01 (1H, m, *enol*), 1.72 (1H, ddd, *J* = 14.3, 11.6, 0.9 Hz), 1.60 (1H, m), 1.60 (1H, m, *enol*), 1.47 (1H, m), 1.47 (1H, m, *enol*), 1.46 (3H, d, *J* = 6.1 Hz, *enol*) and 1.16 (3H, d, *J* = 6.1 Hz) ppm; δ_C (100 MHz, C_6D_6) 201.0, 171.8, 171.2, 168.6, 139.1, 128.6, 138.5, 127.9, 102.2 (*enol*), 74.8, 74.5, 74.1, 73.0, 69.7, 69.5, 66.4, 66.0, 64.5, 51.6, 50.8 (*enol*), 46.8, 36.5, 36.0, 35.5, 22.4 (*enol*) and 20.8 ppm; *m/z* (ESI+) 329 $(M + Na)^+$, 307 $(M + H)^+$, 284 $(M + Na - OCH_3)^+$, 255 $(M + Na - CO_2Me, - CH_3)^+$. (Found 307.1545 $(M + H)^+$. $C_{17}H_{23}O_5$ requires 307.1540).

Isopropyl 2-methyl-4-oxo-6-phenyl-tetrahydro-2H-pyran-3-carboxylate (11h).
Oil; υ_{max} (film) 2981, 2935, 2875, 1738, 1714 cm^{-1}; nOe: H-2 –H-6 3.2 %; δ_H (400 MHz, $CDCl_3$) 7.30-7.41 (5H, m), 5.16 (1H, sept, *J* = 6.0 Hz), 4.76 (1H, dd, *J* = 11.5, 3.0 Hz), 4.20 (1H, dq, *J* = 10.5, 6.0 Hz), 3.28 (1H, d, *J* = 10.5 Hz), 2.70 (1H, dd, *J* = 14.5, 3.0 Hz), 2.59 (1H, dd, *J* = 14.5, 11.5 Hz) 1.43 (3H, d, *J* = 6.0 Hz) and 1.30 (6H, app t, *J* = 6.0 Hz) ppm; δ_C (100 MHz, $CDCl_3$) 201.8, 167.6, 140.2, 128.7, 128.3, 125.6, 78.6, 75.3, 68.9, 64.4, 48.7, 21.8, 21.7 and 20.8 ppm. *m/z* (ESI+) 299 $(M + Na)^+$, 277 $(M + H)^+$. (Found 299.1254 $(M + Na)^+$. $C_{16}H_{20}NaO_4$ requires 299.1254).

(*E*)-Isopropyl 2-methyl-4-oxo-6-(prop-1-enyl)-tetrahydro-2H-pyran-3-carboxylate (11k).
Oil (keto : enol 1 : 0.15); υ_{max} (film) 2979, 2927, 2856, 1739, 1717, 1373, 1264, 1105, 965 cm^{-1}; nOe: H-2 – H-6 2.1%; δ_H (400MHz, C_6D_6) 12.87 (1H, s, *enol*), 5.58-5.37 (2H, m, *enol*), 5.33 (1H, dqd, *J* = 15.3, 6.1, 0.9 Hz), 5.21 (1H, ddq, *J* = 15.3, 6.1, 1.5 Hz), 5.15 (1H, sept, *J* = 6.1 Hz), 5.02 (1H, sept, *J* = 6.4 Hz, *enol*), 4.56 (1H, m, *enol*), 4.02 (1H, dq, *J* = 10.7, 6.1 Hz), 3.69 (1H, m, *enol*), 3.63 (1H, m), 3.01 (1H, dd, *J* = 10.7, 0.8 Hz), 2.32 (1H, m), 2.15 (1H, dd, *J* = 14.0, 2.4 Hz), 2.08 (1H, m, *enol*), 1.87 (1H, ddd, *J* = 14.0, 11.6, 0.8 Hz), 1.55 (3H, d, *J* = 6.1 Hz, *enol*), 1.40 (3H, dd, *J* = 6.1, 1.5 Hz), 1.27 (3H, d, *J* = 6.1 Hz), 1.23 (3H, d, *J* = 6.1 Hz, *enol*), 1.13 (3H, d, *J* = 6.4 Hz), 1.13 (3H, d, *J* = 6.4 Hz, *enol*), 1.07 (3H, d, *J* = 6.4 Hz) and 1.07 (3H, d, *J* = 6.4 Hz, *enol*) ppm; δ_C (100 MHz, C_6D_6) 200.8, 167.5, 130.5, 127.0, 76.9, 74.5, 68.4, 64.4, 46.8, 21.5, 20.6 and 17.3 ppm; *m/z* (ESI+) 263 $(M + Na)^+$, 197 $(M - C_3H_7)^+$. (Found 263.1248 $(M + Na)^+$. $C_{13}H_{20}NaO_4$ requires 263.1254).

Isopropyl 4-oxo-2-phenyl-6-propyl-tetrahydro-2H-pyran-3-carboxylate (11l).
Oil; υ_{max} (film) 3488, 2961, 2933, 2873, 1661, 1620, 1595, 1448, 1370, 1301, 1176, 1097 cm^{-1}; δ_H (400MHz, C_6D_6) 7.34-7.31 (2H, m), 7.08-7.00 (3H,m), 5.03 (1H, d, *J* = 2.1 Hz), 4.92-4.86 (2H, m), 3.62 (1H, m), 2.02 (1H, ddd, *J* = 13.6, 7.4, 2.1 Hz), 1.89 (1H, ddd, *J* = 13.6, 10.7, 9.0 Hz), 1.48 (1H, m), 1.31 (1H, m), 1.22-1.13

(2H, m), 0.78 (3H, d, J = 6.3 Hz), 0.73 (3H, t, J = 7.2 Hz) and 0.67 (3H, d, J = 6.3 Hz) ppm; δ_C (100 MHz, C_6D_6) 169.7, 165.9, 137.4, 129.4, 129.3, 127.7, 107.8, 76.5, 67.6, 64.6, 36.7, 36.2, 21.6, 21.0, 18.8 and 14.0 ppm; *m/z* (ESI+) 327 $(M + Na)^+$, 287 $(M + Na - C_3H_7)^+$. (Found 327.1578 $(M + Na)^+$. $C_{18}H_{24}NaO_4$ requires 327.1567).

(*E*)-Isopropyl 4-oxo-2-phenyl-6-(prop-1-enyl)-tetrahydro-2H-pyran-3-carboxylate (11m).
Oil; υ_{max} (film) 3489, 2979, 2931, 2877, 1664, 1621, 1595, 1413, 1336, 1300, 1175, 1095 cm^{-1}; nOe: H-2 – H-6 0.9%; δ_H (400MHz, C_6D_6) 7.37-7.30 (2H, m), 7.08-6.99 (3H, m), 5.49-5.46 (2H, m), 4.94-4.85 (3H, m), 4.15 (1H, m), 2.14 (1H, ddd, J = 13.7, 7.1, 2.6 Hz), 2.06 (1H, ddd, J = 13.7, 10.6, 8.6 Hz), 1.39 (3H, d, J = 4.8 Hz), 0.77 (3H, d, J = 6.2 Hz) and 0.67 (3H, d, J = 6.2 Hz) ppm; δ_C (100 MHz, C_6D_6) 169.5, 165.5, 137.3, 129.7, 129.4, 129.3, 129.1, 127.7, 107.8, 77.1, 67.6, 64.3, 36.5, 21.5, 21.0 and 17.6 ppm; *m/z* (ESI+) 325 $(M + Na)^+$, 285 $(M + Na - C_3H_5)^+$, 225 $(M + Na - C_3H_5, - C_3H_7OH)^+$. (Found 325.1396 $(M + Na)^+$. $C_{18}H_{22}NaO_4$ requires 325.1410).

Methyl 2-butyl-4-oxo-6-propyl-tetrahydro-2H-pyran-3-carboxylate (11q).
Oil; υ_{max} (film) 2913, 2888, 2826, 1721, 1692, 1594, 1440, 1416, 1320, 1243, 1197, 1174, 1111, 1022, 981 cm^{-1}; nOe: H-2 – H-6 2.3%; δ_H (400MHz, C_6D_6) 3.87 (1H, m), 3.44 (3H, s), 3.13 (1H, dd, J = 10.6, 0.7 Hz), 3.10 (1H, m), 2.07 (1H, dd, J = 13.9, 1.8 Hz), 1.71 (1H, m), 1.58-0.87 (10H, m), 0.84 (3H, t, J = 7.3 Hz) and 0.73 (3H, t, J = 7.5 Hz) ppm; δ_C (100 MHz, C_6D_6) 201.5, 168.8, 78.4, 76.4, 63.4, 51.6, 47.2, 38.4, 35.1, 27.8, 22.7, 18.7, 14.1 and 14.0 ppm; *m/z* (ESI+) 279 $(M + Na)^+$. (Found 279.1571 $(M + Na)^+$. $C_{14}H_{24}NaO_4$ requires 279.1567).

Methyl 2-butyl-6-isopropyl-4-oxo-tetrahydro-2H-pyran-3-carboxylate (11r).
Oil; υ_{max} (film) 2914, 2829, 1721, 1691, 1634, 1593, 1443, 1416, 1322, 1290, 1247, 1199, 1105, 1048, 1021 cm^{-1}; nOe: H-2 – H-6 2.6%, H-3 – H-5_{ax} 0.8%; δ_H (400MHz, C_6D_6) 3.84 (1H, m), 3.45 (3H, s), 3.09 (1H, dd, J = 10.7, 0.9 Hz), 2.79 (1H, ddd, J = 11.6, 6.1, 2.4 Hz), 2.13 (1H, dd, J = 14.0, 2.4 Hz), 1.73 (1H, ddd, J = 14.0, 11.6, 0.9 Hz), 1.62-1.15 (7H, m), 0.84 (3H, t, J = 7.3 Hz), 0.77 (3H, d, J = 6.7 Hz) and 0.61 (3H, d, J = 6.7 Hz) ppm; δ_C (100 MHz, C_6D_6) 201.8, 168.8, 81.3, 78.2, 63.4, 51.6, 44.4, 35.1, 33.3, 27.9, 26.7, 18.0 and 14.1 ppm; *m/z* (ESI+) 279 $(M + Na)^+$. (Found 279.1564 $(M + Na)^+$. $C_{14}H_{24}NaO_4$ requires 279.1567).

Synthesis of Civet constituent

2-(2-(Benzyloxy)ethyl)-6-methyl-dihydro-2H-pyran-4(3H)-one (12)
A solution of THP **11g** (0.21 g, 0.68 mmol) in DMF (3.66 mL) and H_2O (0.08 mL) was submitted to 200 W microwave radiation in a sealed tube at 160 °C for 10 minutes. The solution was cooled to rt and taken up in EtOAc (30 mL). The mixture was washed with H_2O (2 x 20 mL). The aqueous layer was extracted with EtOAc (30 mL) and the combined organic extracts were washed with brine (20 mL), dried over $MgSO_4$ and concentrated *in vacuo* to give **12** (0.17 g, 100 %) as a pale yellow oil. υ_{max}/cm^{-1} (film) 2863, 1721, 1453, 1373, 1327, 1274, 1204, 1157, 1093, 1028, 740, 699 cm^{-1}; δ_H (400 MHz, C_6D_6) 7.27-7.05 (5H, m), 4.27 (1H, d, J = 12.1 Hz), 4.23 (1H, d, J = 12.1 Hz), 3.49 (1H, m), 3.42 (1H, m), 3.31 (1H, m), 3.18 (1H, dqd, J = 11.4, 6.2, 2.6 Hz), 2.16 (1H, ddd, J = 14.7, 2.2, 2.2 Hz), 2.04 (1H, ddd, J = 14.3, 2.6, 2.2 Hz), 1.82, (1H, dd, J = 14.7, 11.7 Hz), 1.77-1.68 (2H, m), 1.54 (1H, m) and 0.93 (3H, d, J = 6.2 Hz) ppm; δ_C (100 MHz, C_6D_6) 204.9, 139.2, 128.3, 127.0, 74.0, 73.0, 72.9, 66.3, 49.3, 47.5, 36.9, 22.0 ppm; *m/z* (ESI+) 271 $(M + Na)^+$, 249 $(M + H)^+$. (Found 249.1490 $(M + H)^+$. $C_{15}H_{21}O_3$ requires 249.1485).

2-(9-Methyl-8-oxa-1,4-dithiaspiro[4.5]decan-7-yl)ethanol (13)
Boron trifluoride diethyl etherate (0.03 mL, 0.26 mmol) was added to a stirred solution of THP **12** (0.07 g, 0.26 mmol) and 1,2-ethanedithiol (0.03 mL, 0.39 mmol) in CH_2Cl_2 (5 mL) at rt. The mixture was stirred for 20 minutes then taken up in Et_2O (80 mL), washed with brine (20 mL), dried over $MgSO_4$ and concentrated *in vacuo* to give an orange oil which was then dissolved in CH_2Cl_2 (11.5 mL). A 2M solution of boron trichloride dimethyl sulfide in CH_2Cl_2 (1.52 mL, 3 mmol) was added to the solution at rt. The mixture was stirred for 1h then quenched with sat. aq. $NaHCO_3$ (10 mL), and the phases separated. The aqueous layer was extracted with

CH_2Cl_2 (6 x 15 mL); the combined organic extracts were dried over $MgSO_4$ and concentrated *in vacuo* to give **13** (0.06 g, 100%) as a yellow oil. υ_{max}/cm^{-1} (film) 3432, 2926, 2860, 1426, 1372, 1323, 1277, 1151, 1099, 1056 cm^{-1}; δ_H (400 MHz, C_6D_6) 3.74 (1H, m), 3.70-3.54 (3H, m), 2.78-2.70 (4H, m), 2.29 (1H, br s), 1.91 (2H, m), 1.79 (1H, dd, *J* = 13.4, 10.8 Hz), 1.68 (1H, dd, *J* = 13.5, 10.6 Hz), 1.60 (1H, m), 1.35 (1h, m) and 0.93 (3H, d, *J* = 6.2 Hz) ppm; δ_C (100 MHz, C_6D_6) 76.6, 72.9, 65.7, 60.9, 49.5, 48.0, 39.1, 38.3, 37.7 and 21.5 ppm; *m/z* (ESI+) 257 $(M + Na)^+$, 235 $(M + H)^+$. (Found 235.0816 $(M + H)^+$. $C_{10}H_{19}O_2S_2$ requires 235.0821).

2-(6-Methyltetrahydro-2H-pyran-2-yl)ethanol (14).
A mixture of **13** (0.02 g, 0.09 mmol) in EtOH (5 mL) and Raney Ni (0.6g) was heated to 50 °C under an atmosphere of H_2 for 45h, after which time it was filtered through Celite with MeOH concentrated *in vacuo* to give **14** (0.01 g, 68 %). δ_H (400 MHz, $CDCl_3$) 3.79-3.76 (2H, m), 3.58 (1H, m), 3.47 (1H, m), 1.84-1.44 (6H, m), 1.35-1.14 (2H, m) and 1.16 (3H, d, *J* = 5.9 Hz) ppm; δ_C (100 MHz, C_6D_6) 78.4, 74.0, 61.5, 33.0, 31.2, 23.4 and 22.1 ppm. Spectroscopic data were in agreement with the literature.[1]

2-(6-Methyltetrahydro-2H-pyran-2-yl)acetic acid (15).
A solution of Jones reagent (0.08 mL, 0.22 mmol) was added to a stirred solution of **14** (0.015 g, 0.1 mmol) in acetone (8mL) at 0 °C. The mixture was stirred for 0.5h at this temperature then warmed to rt and stirred for a further 1.5h. The reaction was quenched with propan-2-ol (2 mL) and the solvent was removed *in vacuo*. The turquoise residue was dissolved in H_2O (8 mL) and acidified to pH 1 with 2M HCl. The solution was extracted with Et_2O (8 x 20 mL), and the combined organic extracts were dried over $MgSO_4$ and concentrated *in vacuo* to give the Civet constituent **15** (0.010 g, 63 %). δ_H (400 MHz, $CDCl_3$) 3.77 (1H, m), 3.55 (1H, m), 2.57 (1H, dd, *J* = 15.9, 7.6 Hz), 2.51 (1H, dd, *J* = 15.9, 4.9 Hz), 1.84 (1H, m), 1.67-1.48 (3H, m), 1.33-1.14 (2H, m) and 1.20 (3H, d, *J* = 6.1 Hz) ppm; δ_C (100 MHz, $CDCl_3$) 174.1, 74.7, 73.9, 41.0, 32.7, 30.7, 23.1 and 22.0 ppm. Spectroscopic data were in agreement with the literature.[1]

Methyl 2,3-dimethyl-4-oxo-6-propyl-tetrahydro-2H-pyran-3-carboxylate (16b).
Oil; υ_{max} (film) 2913, 2830, 1717, 1687, 1432, 1356, 1308, 1246, 1084, 1013 cm^{-1}; nOe: H-2 – H-6 1.8%, $H\text{-}5_{ax}$ – H-10 1.1%; δ_H (400MHz, C_6D_6) 4.16 (1H, q, *J* = 6.1 Hz), 3.41 (3H, s), 3.21 (1H, m), 1.96-1.94 (2H, m), 1.30 (3H, s), 1.37-1.20 (2H, m), 1.18-1.08 (1H, m), 1.01 (3H, d, *J* = 6.1 Hz), 0.98-0.80 (1H, m) and 0.74 (3H, t, *J* = 7.3 Hz) ppm; δ_C (100 MHz, C_6D_6) 205.5, 171.4, 76.6, 76.3, 62.6, 51.8, 43.3, 38.4, 18.5, 16.1, 14.0 and 13.8 ppm; *m/z* (ESI+) 251 $(M + Na)^+$, 229 $(M + H)^+$, 185 $(M - C_3H_7)^+$. (Found 229.1424 $(M + H)^+$. $C_{12}H_{21}O_4$ requires 229.1434).

Methyl 6-isopropyl-2,3-dimethyl-4-oxo-tetrahydro-2H-pyran-3-carboxylate (16d).
Oil; υ_{max} (film) 2915, 1716, 1688, 1431, 1355, 1250, 1088 cm^{-1}; nOe: H-2 – H-6 2.3%, $H\text{-}5_{ax}$ – H-10 0.9%; δ_H (400MHz, C_6D_6) 4.14 (1H, q, *J* = 6.1 Hz), 3.42 (3H, s), 2.96 (1H, ddd, *J* = 9.5, 5.5, 5.5 Hz), 2.00-1.98 (2H, m), 1.43 (1H, m), 1.28 (3H, s), 1.01 (3H, d, *J* = 6.1 Hz),0.76 (3H, d, *J* = 6.9 Hz) and 0.64 (3H, d, *J* = 6.9 Hz) ppm; δ_C (100 MHz, C_6D_6) 205.8, 171.4, 81.4, 76.2, 62.6, 51.8, 40.2, 33.1, 17.9, 16.0 and 13.7 ppm; *m/z* (ESI+) 283, 261, 251 $(M + Na)^+$, 229 $(M + H)^+$, 185 $(M - C_3H_7)^+$. (Found 229.1427 $(M + H)^+$. $C_{12}H_{21}O_4$ requires 229.1434).

Methyl 6-(furan-2-yl)-2,3-dimethyl-4-oxo-tetrahydro-2H-pyran-3-carboxylate (16e).
Oil; υ_{max} (film) 3101, 3012, 2942, 2907, 1716, 1431, 1364, 1325, 1253, 1076, 728 cm^{-1}; nOe: H-2 – H-6 3.0%, $H\text{-}5_{ax}$ – H-10 0.9%; δ_H (400MHz, C_6D_6) 7.00 (1H, s), 5.99 (1H, dd, *J* = 3.3, 1.7 Hz), 5.96 (1H, d, *J* = 3.3 Hz), 4.30 (1H, dd, *J* = 12.3, 3.0 Hz), 4.24 (1H, q, *J* = 6.3 Hz), 3.39 (3H, s), 2.71 (1H, dd, *J* = 15.0, 12.3 Hz), 2.24 (1H, dd, *J* = 15.0, 3.0 Hz), 1.31 (3H, s) and 1.01 (3H, d, *J* = 6.3 Hz) ppm; δ_C (100 MHz, C_6D_6) 204.3, 171.1, 153.0, 142.7, 110.3, 107.7, 76.4, 71.9, 62.6, 51.8, 41.0, 16.0 and 13.7 ppm; *m/z* (ESI+) 307, 275 $(M + Na)^+$, 253 $(M + H)^+$. (Found 253.1066 $(M + H)^+$. $C_{13}H_{17}O_5$ requires 253.1071).

Methyl 6-(benzyloxymethyl)-2,3-dimethyl-4-oxo-tetrahydro-2H-pyran-3-carboxylate (16f).
Oil; υ_{max} (film) 3010, 2941, 2907, 2875, 2823, 1713, 1689, 1431, 1248, 1085 cm^{-1}; δ_H (400MHz, C_6D_6) 7.25-7.05 (5H, m), 4.29 (1H, d, *J* = 12.2 Hz), 4.24 (1H, d, *J* = 12.2 Hz), 4.18 (1H, q, *J* = 6.3 Hz), 3.48 (1H, m), 3.39 (3H, s), 3.16-3.14 (2H, m), 2.36 (1H, dd, *J* = 15.3, 11.6 Hz), 2.06 (1H, dd, *J* = 15.3, 3.0 Hz), 1.30 (3H, s) and

1.02 (3H, d, *J* = 6.3 Hz) ppm; δ_C (100 MHz, C_6D_6) 205.3, 171.2, 138.0, 128.6, 127.9, 127.7, 76.5, 76.2, 73.4, 72.2, 62.6, 51.8, 39.7, 16.1 and 13.7 ppm; *m/z* (ESI+) 361, 329 $(M + Na)^+$. (Found 329.1347 $(M + Na)^+$. $C_{17}H_{22}NaO_5$ requires 329.1359).

Isopropyl 2,3-dimethyl-4-oxo-6-propyl-tetrahydro-2H-pyran-3-carboxylate (16j).
Oil; υ_{max} (film) 2981, 2938, 2875, 1737, 1714, 1455, 1376, 1265, 1100 cm^{-1}; nOe: H-2 – H-6 2.2%; δ_H (400MHz, C_6D_6) 5.14 (1H, sept, *J* = 5.7 Hz), 4.22 (1H, q *J* = 6.2 Hz), 3.24 (1H, m), 1.97-1.94 (2H, m), 1.32 (3H, s), 1.12 (3H, d, *J* = 6.2 Hz), 1.08 (3H, d, *J* = 6.2 Hz), 1.06 (3H, d, *J* = 6.2 Hz), 1.36-1.22 (2H, m) and 1.16-0.98 (2H, m) and 0.74 (3H, t, *J* = 7.1 Hz) ppm; δ_C (100 MHz, C_6D_6) 205.3, 170.1, 76.3, 76.1, 68.1, 62.1, 43.1, 38.1, 21.4, 18.3, 15.7, 13.7 and 13.6 ppm; *m/z* (ESI+) 293, 279 $(M + Na)^+$. (Found 279.1561 $(M + Na)^+$. $C_{14}H_{24}NaO_4$ requires 279.1567).

Methyl 2-butyl-6-isopropyl-3-methyl-4-oxo-tetrahydro-2H-pyran-3-carboxylate (16r).
Oil; υ_{max} (film) 2912, 2831, 1717, 1688, 1440, 1358, 1275, 1249, 1176, 1084 cm^{-1}; nOe: H-2 – H-6 2.7%; δ_H (400MHz, C_6D_6) 4.00 (1H, dd, *J* = 9.8, 2.1 Hz), 3.45 (3H, s), 2.87 (1H, m), 2.05 (1H, dd, *J* = 15.0, 3.4 Hz), 1.98 (1H, dd, *J* = 15.0, 11.3 Hz), 1.60-1.11 (7H, m), 1.32 (3H, s), 0.84 (3H, t, *J* = 7.3 Hz), 0.80 (3H, d, *J* = 6.7 Hz) and 0.63 (3H, d, *J* = 6.7 Hz) ppm; δ_C (100 MHz, C_6D_6) 205.9, 171.5, 81.7, 80.3, 62.7, 51.8, 41.0, 33.4, 30.6, 28.9, 22.6, 18.0, 17.9, 14.3 and 14.1 ppm; *m/z* (ESI+) 293 $(M + Na)^+$, 271 $(M + H)^+$. (Found 293.1717 $(M + Na)^+$. $C_{15}H_{26}NaO_4$ requires 293.1723).

Methyl 2-butyl-6-(benzyloxymethyl)-4-oxo-5,6-dihydro-2H-pyran-3-carboxylate (16t).
Light yellow oil; υ_{max} (film) 3955, 2913, 2849, 1738, 1712, 1454, 1260, 1089, 1016, 795, 736, 697, 552 cm-1; δ_H (400MHz, C_6D_6) 7.27-7.17 (4H, m), 7.14-7.08 (1H, m), 4.31 (1H, d, *J* = 12.3 Hz), 4.27 (1H, d, *J* = 12.3 Hz), 4.08 (1H, dd, *J* = 9.5, 2.2 Hz), 3.51-3.45 (1H, m), 3.43 (3H, s), 3.17 (2H, m), 2.37 (1H, dd, *J* = 15.1, 11.8 Hz), 2.10 (1H, dd, *J* = 15.1, 3.1 Hz), 1.63-1.50 (2H, m), 1.34 (3H, s), 1.27-1.15 (4H, m), 0.84 (3H, t, *J* = 8.3 Hz) ppm; δ_C (100 MHz, C_6D_6) 204.9, 171.0, 138.6, 128.3, 80.2, 76.2, 72.7, 71.9, 62.2, 51.3, 39.9, 30.6, 28.5, 22.6, 14.05, 13.8 ppm; *m/z* (ESI^+) 371 $(M + Na)^+$. (Found 371.1828 $(M + Na)^+$. $C_{20}H_{28}NaO_5$ requires 371.1829).

Isopropyl 4-methoxy-2-phenyl-6-propyl-5,6-dihydro-2H-pyran-3-carboxylate (17l).
Oil; υ_{max} (film) 2961, 2931, 2874, 1695, 1628, 1597, 1463, 1371, 1280, 1176, 1139, 1088 cm^{-1}; nOe: H-2 – H-6 0.5%; δ_H (400MHz, C_6D_6) 7.52-7.48 (2H, m), 7.11-7.05 (3H, m), 4.98 (1H, sept, *J* = 6.2 Hz), 4.50 (1H, dd, *J* = 6.2, 4.6 Hz), 4.02 (1H, m), 3.27 (3H, s), 2.01 (1H, m), 1.76 (1H, ddd, *J* = 15.2, 6.2, 3.8 Hz), 1.65 (1H, ddd, *J* = 15.2, 5.3, 4.6 Hz), 1.49-1.19 (3H, m), 0.93 (3H, d, *J* = 6.2 Hz), 0.79 (3H, t, *J* = 7.1 Hz) and 0.74 (3H, d, *J* = 6.2 Hz) ppm; δ_C (100 MHz, C_6D_6) 167.9, 160.5, 137.4, 129.2, 129.1, 107.9, 75.4, 71.4, 67.1, 56.7, 34.8, 30.3, 21.8, 21.1, 19.9 and 14.0 ppm; *m/z* (ESI+) 341 $(M + Na)^+$, 287 $(M + Na - C_2H_6)^+$. (Found 341.1719 $(M + Na)^+$. $C_{19}H_{26}NaO_4$ requires 341.1723).

(*E*)-Isopropyl 4-methoxy-2-phenyl-6-(prop-1-enyl)- 5,6-dihydro-2H-pyran-3-carboxylate (17m).
Oil; υ_{max} (film) 2979, 2931, 2822, 1694, 1629, 1449, 1371, 1279, 1075, 973 cm^{-1}; δ_H (400MHz, C_6D_6) 7.53-7.51 (2H, m), 7.07-7.05 (3H, m), 6.17 (1H, ddq, *J* = 15.0, 8.3, 1.5 Hz), 5.49 (1H, dq, *J* = 15.0, 6.6 Hz), 4.99 (1H, sept, *J* = 6.2 Hz), 4.55 (1H, dd, *J* =5.1, 4.0 Hz), 4.50 (1H, ddd, *J* = 8.3, 5.1, 4.0 Hz), 3.26 (3H, s), 1.91 (1H, ddd, *J* = 14.3, 4.0, 4.0 Hz), 1.74 (1H, ddd, *J* = 14.3, 5.1, 5.1 Hz), 1.45 (3H, dd, *J* = 6.6, 1.5 Hz), 0.94 (3H, d, *J* = 6.2 Hz) and 0.75 (3H, d, *J* = 6.2 Hz) ppm; δ_C (100 MHz, C_6D_6) 167.7, 160.5, 137.0, 129.5, 128.9, 128.5, 128.3, 107.8, 76.3, 70.9, 66.9, 56.4, 31.3, 21.6, 20.9 and 17.3 ppm; *m/z* (ESI+) 401, 339 $(M + Na)^+$, 285, 225. (Found 339.1568 $(M + Na)^+$. $C_{19}H_{24}NaO_4$ requires 339.1567).

Synthesis of the Model A-Ring of Lasonolide A
(2R,3S,4R,6R)-6-(benzyloxymethyl)-2-butyl-3-(hydroxymethyl)-3-methyl-tetrahydro-2H-pyran-4-ol (18).
A solution of **16t** (0.033 g, 0.09 mmol) in THF (3.0 mL) was added slowly and under N_2 to a stirred suspension of $LiAlH_4$ (0.009 g, 0.24 mmol) in THF (3.0 mL) at 0 °C. When the reaction was shown to be complete by TLC the $LiAlH_4$ was quenched by the sequential dropwise addition of 5 mL of H_2O, 15 mL of 15% of NaOH solution followed with 3x5 mL of H_2O. The resulting slurry was dried over $MgSO_4$, filtered and concentrated *in vacuo.*

The resulting oil was subjected to flash column chromatography (hexane – ethyl acetate) to afford **18** as a colourless oil, 0.015 g, (51%). υ max/cm^{-1} (film) 3369, 2927, 2857, 1453, 1375, 1113, 1086, 1025, 733, 696, 665 cm^{-1}; δH (400MHz, $CDCl_3$) 7.34-7.22 (4H, m, Ar), 7.31-7.27 (1H, m, Ar), 4.60 (1H, d, *J* = 12.0 Hz, H-9), 4.56 (1H, d, *J* = 12.0 Hz, H-9), 3.93 (1H, dd, *J* = 11.5, 4.8 Hz, H- 4), 3.69 (1H, d, *J* = 10.4 Hz, H-11), 3.52 (1H, d, *J* = 10.4 Hz, H-11), 3.61-3.58 (1H, m, H-6), 3.55 (1H, dd, *J* = 12.0, 6.5 Hz, H-8), 3.48 (1H, dd, *J* = 12.0, 6.5 Hz, H-8), 3.02 (1H, dd, *J* = 10.2, 1.4 Hz, H-2), 2.41 (1H, br s, OH), 1.75 (1H, ddd, *J* = 12.4, 4.8, 2.0 Hz, H-5_{eq}), 1.48 (1H, dd, *J* = 12.4, 12.0, 11.5 Hz, H-5_{ax}), 1.42-1.35 (2H, m, H-7), 1.33-1.25 (2H, m, H-14), 0.94 (3H, s, H-12) and 0.89 (3H, t, *J* = 7.1 Hz, H-15) ppm; δC (100 MHz, $CDCl_3$) 138.5 (Ar), 128.4 (Ar), 127.7 (Ar), 80.3 (C-2), 75.6 (C-6), 74.3 (C-4), 73.4 (C-9), 73.2 (C-8), 69.8 (C-11), 42.8 (C-3), 33.3 (C-5), 29.8 (C-13), 22.8 (C-14), 14.2 (C-15) and 9.1 (C-12) ppm; (*m/z* (ESI^+) 345 $(M + Na)^+$. (Found 345.2042 $(M + Na)^+$. $C_{19}H_{30}NaO_4$ requires 345.2036).

(2R,3S,4S,6R)-methyl 6-(benzyloxymethyl)-2-butyl-4-hydroxy-3-methyl-tetrahydro-2H-pyran-3-carboxylate (19).

A 1.0 M solution of L-Selectride® in THF (0.47 mL, 2.19 mmol) was added to a stirred solution of **16t** (0.064 g, 0.18 mmol) in THF (9.00 mL) at -78 °C. The mixture was stirred for 1 hour at -78 °C followed by 1 hour at room temperature until complete by TLC. The reaction was partitioned between Et_2O (45.0 mL) and sat. aq. $NHCl_4$ (45.0 mL). The aqueous layer was washed with Et_2O (45.0 mL) and the combined organic extracts were washed with brine (100.0 mL), dried over $MgSO_4$ and concentrated *in vacuo*. Purification by flash column chromatography (toluene-methanol) afforded **19** as a light yellow oil, 0.044 g (69%); υ_{max} (film) 3462, 2953, 2926, 2856, 1731, 1454, 1264, 1101, 1042, 734, 697 cm^{-1} ; δ_H (400MHz, C_6D_6) 7.32-7.30 (2H, m, Ar), 7.20-7.18 (2H, m, Ar), 7.11-7.07 (1H, m, Ar), 4.42 (1H, d, *J* = 12.9 Hz, H-9), 4.39 (1H, d, *J* = 12.9 Hz, H-9), 4.30 (1H, dd, *J* = 6.9, 4.4 Hz, H-2), 4.19-4.13 (1H, m, H-6), 3.84 (1H, dd, *J* = 5.7, 2.7 Hz, H- 4), 3.44 (1H, dd, *J* = 10.1, 5.2 Hz, H-8), 3.37 (1H, dd, *J* = 10.1, 4.5 Hz, H-8), 3.26 (3H, s, H-11), 2.69 (1H, br s, OH), 1.79-1.72 (1H, m, H-7), 1.48-44 (1H, m, H-7), 1.66 (1H, ddd, *J* = 14.0, 12.0, 2.7 Hz, H-5_{ax}), 1.46 (1H, ddd, *J* = 14.0, 5.7, 2.4 Hz, H-5_{eq}), 1.55-1.50 (2H, m, H-13), 1.38-1.29 (2H, m, H-14), 1.17 (3H, s, H-12) and 0.90 (3H, t, *J* = 7.3 Hz, H-15) ppm; δ_C (100 MHz, C_6D_6) 195.0, 175.5, 139.1, 128.2, 127.3, 75.2, 73.4, 73.1, 71.6, 71.4, 51.1, 49.5, 31.2, 31.0, 29.1, 22.8, 14.6 and 14.0 ppm; *m/z* (ESI^+) 373 $(M + Na)^+$. (Found 373.1980 $(M + Na)^+$. $C_{20}H_{30}NaO_5$ requires 373.1985).

(2R,3S,4S,6R)-6-(benzyloxymethyl)-2-butyl-3-(hydroxymethyl)-3-methyl-tetrahydro-2H-pyran-4-ol (20).

A solution of **19** (0.027 g, 0.08 mmol) in THF (3.0 mL) was added slowly and under N_2 to a stirred suspension of $LiAlH_4$ (0.006 g, 0.15 mmol) in THF (3.0 mL) at 0 °C. When the reaction was shown to be complete by TLC the $LiAlH_4$ was quenched by the sequential dropwise addition of 5 mL of H_2O, 15 mL of 15% of NaOH solution followed with 3x5 mL of H_2O. The resulting slurry was dried over $MgSO_4$, filtered and concentrated *in vacuo.* The resulting oil was subjected to flash column chromatography (hexane – ethyl acetate) to afford **20** as a colourless oil, 0.011 g, (52%); υ_{max} (film) 3351, 2924, 2857, 1453, 1377, 1092, 1028, 732, 697, 613 cm^{-1}; δ_H (400MHz, $CDCl_3$) 7.34-7.33 (4H, m, Ar), 7.31-7.27 (1H, m, Ar), 4.61 (1H, d, *J* = 12.4 Hz, H-9), 4.58 (1H, d, *J* = 12.4 Hz, H-9), 3.99 (1H, dddd, *J* = 12.0, 5.6, 4.4, 3.0 Hz, H-6), 3.93 (1H, dd, *J* = 5.6, 3.0 Hz, H- 4), 3.91 (1H, dd, *J* = 8.0, 3.6 Hz, H-2), 3.63 (1H, d, *J* = 12.6 Hz, H-11), 3.59 (1H, d, *J* = 12.6 Hz, H-11), 3.56 (1H, dd, *J* = 10.4, 5.6 Hz, H-8), 3.48 (1H, dd, *J* = 10.4, 4.4 Hz, H-8), 2.86 (2H, br s, OH), 1.80 (1H, ddd, *J* = 14.4, 12.0, 3.0 Hz, H-5_{ax}), 1.50 (1H, ddd, *J* = 14.4, 5.6, 2.8 Hz, H-5_{eq}), 1.63-1.58 (2H, m, H-14), 1.43-1.25 (4H, m, H-7, H-13), 0.90 (3H, t, *J* = 7.0 Hz, H-15) and 0.78 (3H, s, H-12) ppm; δ_C (100 MHz, $CDCl_3$) 138.6, 128.4, 127.7, 127.6, 75.3, 75.1, 73.4 , 73.3, 71.6, 70.1, 40.5, 33.5, 29.0, 28.7, 22.9, 15.7 and 14.3 ppm; (*m/z* (ESI^+) 345 $(M + Na)^+$. (Found 345.2044 $(M + Na)^+$. $C_{19}H_{30}$ NaO_4 requires 345.2036).

Methyl 4-hydroxy-2,2-dimethyl-6-propyl-5,6-dihydro-2H-pyran-3-carboxylate (25b).

Oil ($keto_{eq}$: enol : $keto_{ax}$ 2.4 : 3 : 1); υ_{max} (film) 3364, 2914, 2889, 1725, 1694, 1620, 1417, 1359, 1323, 1269, 1200, 1114, 1053, 1023 cm^{-1}; δ_H (400 MHz, C_6D_6) 13.29 (1H, s), 3.54-3.43 (1H, m), 3.54-3.43 (1H, m, *keto*$_{eq}$), 3.54-3.43 (1H, m, *keto*$_{ax}$), 3.38 (3H, s, *keto*$_{eq}$), 3.38 (1H, s, *keto*$_{eq}$), 3.25 (3H, s), 3.21 (1H, d, *J* = 1.3 Hz, *keto*$_{ax}$), 3.17 (3H, s, *keto*$_{ax}$), 3.02 (1H, dd, *J* = 14.1, 11.2 Hz, *keto*$_{ax}$), 2.16 (1H, m, *keto*$_{ax}$), 2.14 (1H, dd, *J* = 17.6, 10.6

Hz), 2.01 (1H, dd, *J* = 15.1, 3.1 Hz, *keto*$_{eq}$), 1.99 (1H, dd, *J* = 17.6, 3.1 Hz), 1.67 (1H, dd, *J* = 15.1, 11.4 Hz, *keto*$_{eq}$), 1.57 (3H, s), 1.44 (3H, s), 1.44-0.96 (4H, m), 1.44-0.96 (4H, m, *keto*$_{eq}$), 1.44-0.96 (4H, m, *keto*$_{ax}$), 1.38 (3H, s, *keto*$_{eq}$), 1.30 (3H, s, *keto*$_{eq}$), 0.92-0.74 (3H, m), 0.92-0.74 (3H, m, *keto*$_{eq}$), 0.92-0.74 (3H, m, *keto*$_{ax}$), 0.86 (3H, s, *keto*$_{ax}$) and 0.77 (3H, s, *keto*$_{ax}$) ppm; δ_C (100 MHz, C_6D_6) 201.8 (*keto*), 172.6, 172.5, 168.4 (*keto*$_{eq}$), 168.2 (*keto*$_{ax}$), 105.4, 77.2, 76.0 (*keto*$_{ax}$), 73.3, 70.6 (*keto*$_{ax}$), 70.5 (*keto*$_{eq}$), 67.4 (*keto*$_{eq}$), 66.4, 66.2 (*keto*$_{ax}$), 51.6 (*keto*$_{eq}$ & *keto*$_{ax}$), 51.1, 47.2 (*keto*$_{eq}$), 45.6 (*keto*$_{ax}$), 39.0, 38.1, 36.3, 30.1, 29.6 (*keto*$_{eq}$), 28.0 (*keto*$_{ax}$), 25.9, 25.2 (*keto*$_{ax}$), 21.8 (*keto*$_{eq}$), 19.0, 18.8, 14.3 and 14.2 ppm; *m/z* (ESI+) 251 $(M + Na)^+$. (Found 251.1253 $(M + Na)^+$. $C_{12}H_{20}NaO_4$ requires 251.1254).

Methyl 4-hydroxy-6-isopropyl-2,2-dimethyl-5,6-dihydro-2H-pyran-3-carboxylate (25c).
Oil (keto$_{eq}$: enol : keto$_{ax}$ 2.1 : 2.95 : 1); υ_{max} (film) 2916, 2831, 1725, 1693, 1621, 1584, 1418, 1322, 1257, 1208, 1113, 1050, 843 cm^{-1}; δ_H (400 MHz, C_6D_6) 13.29 (1H, s), 3.38 (3H, s, *keto*$_{eq}$), 3.36 (1H, s, *keto*$_{eq}$), 3.33-3.24 (1H, m, *keto*$_{eq}$), 3.33-3.24 (1H, m, *keto*$_{ax}$), 3.24 (3H, s), 3.21 (1H, d, *J* = 1.2 Hz, *keto*$_{ax}$), 3.16 (3H, s, *keto*$_{ax}$) 3.15 (1H, m), 3.04 (1H, dd, *J* = 14.0, 11.3 Hz, *keto*$_{ax}$), 2.21 (1H, m, *keto*$_{ax}$), 2.18 (1H, dd, *J* = 19.5, 11.0 Hz), 2.06 (1H, m, *keto*$_{eq}$), 2.05 (1H, m), 1.70 (1H, dd, *J* = 13.1, 11.3 Hz, *keto*$_{eq}$), 1.56 (3H, s), 1.57-1.49 (1H, m), 1.57-1.49 (1H, m, *keto*$_{ax}$), 1.43 (3H, s), 1.42 (1H, m, *keto*$_{eq}$), 1.37 (3H, s, *keto*$_{eq}$), 1.28 (3H, s, *keto*$_{eq}$), 1.18 (3H, s, *keto*$_{ax}$), 0.93 (3H, d, *J* = 6.7 Hz), 0.88 (3H, d, *J* = 6.7 Hz, *keto*$_{ax}$), 0.84 (3H, s, *keto*$_{ax}$), 0.77 (3H, d, J = 6.7 Hz, *keto*$_{eq}$), 0.76 (3H, d, *J* = 6.7 Hz, *keto*$_{ax}$), 0.72 (3H, d, *J* = 6.7 Hz) and 0.62 (3H, d, J = 6.7 Hz, *keto*$_{eq}$) ppm; δ_C (100 MHz, C_6D_6) 203.3 (*keto*$_{ax}$), 202.0 (*keto*$_{eq}$), 172.6, 172.4, 168.3, 168.0, 105.2, 76.9, 75.6, 75.4 (*keto*$_{ax}$), 75.3 (*keto*$_{eq}$), 73.1, 71.6, 67.1 (*keto*$_{eq}$), 66.0 (*keto*$_{ax}$), 51.4 (*keto*$_{eq}$), 51.4 (*keto*$_{ax}$), 50.9, 44.2 (*keto*$_{eq}$), 42.4 (*keto*$_{ax}$), 33.5, 33.5 (*keto*$_{ax}$), 33.4 (*keto*$_{eq}$), 32.9, 29.8, 29.3 (*keto*$_{eq}$), 27.7 (*keto*$_{ax}$), 25.6, 25.1 (*keto*$_{ax}$), 21.7 (*keto*$_{eq}$), 18.5, 18.1 (*keto*$_{ax}$), 18.1 (*keto*$_{eq}$), 18.0, 17.8 (*keto*$_{ax}$) and 17.8 (*keto*$_{eq}$) ppm; *m/z* (ESI+) 251 $(M + Na)^+$. (Found 251.1250 $(M + Na)^+$. $C_{12}H_{20}NaO_4$ requires 251.1254).

Methyl 6-(furan-2-yl)-4-hydroxy-2,2-dimethyl-5,6-dihydro-2H-pyran-3-carboxylate (25d).
Oil (keto$_{eq}$: enol : keto$_{ax}$ 2.42 : 3.72 : 1); υ_{max} (film) 2929, 2909, 2885, 2810, 1724, 1694, 1621, 1585, 1419, 1357, 1328, 1262, 1134, 1111, 1043 cm^{-1}; δ_H (400 MHz, C_6D_6) 13.27 (1H, s), 7.04-6.99 (1H, m), 7.04-6.99 (1H, m, *keto*$_{eq}$), 7.04-6.99 (1H, m, *keto*$_{ax}$), 6.11-5.95 (2H, m), 6.11-5.95 (2H, m, *keto*$_{eq}$), 6.11-5.95 (2H, m, *keto*$_{ax}$), 4.68-4.61 (1H, m), 4.68-4.61 (1H, m, *keto*$_{eq}$), 4.68-4.61 (1H, m, *keto*$_{ax}$), 3.68 (1H, dd, *J* = 14.3, 11.6 Hz, *keto*$_{ax}$), 3.46 (1H, s, *keto*$_{eq}$), 3.36 (1H, s, *keto*$_{ax}$), 3.34 (3H, s, *keto*$_{eq}$), 3.20 (3H, s), 3.15 (3H, s, *keto*$_{ax}$), 2.82 (1H, dd, *J* = 14.0, 11.0 Hz), 2.44 (1H, m, *keto*$_{ax}$), 2.43 (1H, ddd, *J* = 14.0, 11.6, 0.6 Hz, *keto*$_{eq}$), 2.28 (1H, dd, *J* = 14.0, 3.1 Hz), 2.25 (1H, dd, *J* = 14.0, 3.4 Hz, *keto*$_{eq}$), 1.55 (3H, s), 1.48 (3H, s), 1.40 (3H, s, *keto*$_{eq}$), 1.27 (3H, s, *keto*$_{eq}$), 1.17 (3H, s, *keto*$_{ax}$) and 0.85 (3H, s, *keto*$_{ax}$) ppm; δ_C (100 MHz, C_6D_6) 171.3, 154.3, 153.5, 142.5, 142.3, 110.4, 110.3, 107.5, 107.1, 77.6, 74.2, 67.2 (*keto*$_{ax}$), 67.0 (*keto*$_{eq}$), 66.6 (*keto*$_{ax}$), 66.5 (*keto*$_{eq}$), 63.1, 51.5, 50.9, 44.7 (*keto*$_{eq}$), 43.3 (*keto*$_{ax}$), 33.9, 29.6, 29.1 (*keto*$_{eq}$), 27.6 (*keto*$_{ax}$), 25.7, 24.8 (*keto*$_{ax}$) and 21.3 (*keto*$_{eq}$) ppm; *m/z* (ESI+) 275 $(M + Na)^+$. (Found 275.0889 $(M + Na)^+$. $C_{13}H_{16}NaO_5$ requires 275.0890).

Methyl 6-(benzyloxymethyl)-4-hydroxy-2,2-dimethyl-5,6-dihydro-2H-pyran-3-carboxylate (25e).
Oil (keto$_{eq}$: enol : keto$_{ax}$ 2.3 : 2.3 : 1); υ_{max} (film) 2931, 2891, 2864, 2822, 1724, 1693, 1619, 1584, 1418, 1322, 1263, 1199, 1116, 1101, 1081, 1048 cm^{-1}; δ_H (400 MHz, C_6D_6) 13.27 (1H, s), 7.27-7.06 (5H, m), 7.27-7.06 (5H, m, *keto*$_{eq}$), 7.27-7.06 (5H, m, *keto*$_{ax}$), 4.37-4.25 (2H, m), 4.37-4.25 (2H, m, *keto*$_{eq}$), 4.37-4.25 (2H, m, *keto*$_{ax}$), 3.88-3.83 (1H, m), 3.88-3.83 (1H, m, *keto*$_{ax}$), 3.77 (1H, m, *keto*$_{eq}$), 3.48-3.38 (1H, m), 3.48-3.38 (1H, m, *keto*$_{eq}$), 3.48-3.38 (1H, m, *keto*$_{ax}$), 3.35 (3H, s), 3.30-3.13 (1H, m), 3.30-3.13 (1H, m, *keto*$_{eq}$), 3.30-3.13 (1H, m, *keto*$_{ax}$), 3.30-3.13 (1H, m, *keto*$_{eq}$), 3.30-3.13 (1H, m, *keto*$_{ax}$), 3.30-3.13 (1H, m, *keto*$_{ax}$), 3.22 (3H, s, *keto*$_{eq}$), 3.15 (3H, s, *keto*$_{ax}$), 2.33 (1H, dd, *J* = 17.4, 11.0 Hz), 2.29 (1H, m, *keto*$_{ax}$), 2.18-2.11 (1H, m), 2.18-2.11 (1H, m, *keto*$_{eq}$), 2.06 (1H, ddd, *J* = 14.0, 10.7, 0.6 Hz, *keto*$_{eq}$), 1.56 (3H, s), 1.44 (3H, s), 1.37 (3H, s), 1.28 (3H, s), 1.18 (3H, s, *keto*$_{ax}$) and 0.83 (3H, s, *keto*$_{ax}$) ppm; δ_C (100 MHz, C_6D_6) 202.3 (*keto*$_{ax}$), 201.4 (*keto*$_{eq}$), 172.2, 171.8, 168.1, 167.9, 139.0, 138.8, 128.6, 128.5, 128.5, 127.9, 127.7, 105.2, 77.4, 76.2, 73.6, 73.5 (*keto*$_{ax}$), 73.4, 73.4, 73.0, 72.7, 70.6, 70.4 (*keto*$_{eq}$), 67.0, 66.6, 66.0, 51.4, 50.9, 43.4 (*keto*$_{eq}$), 42.0 (*keto*$_{ax}$), 32.9, 29.8, 29.2, 27.7 (*keto*$_{ax}$), 25.7, 24.8 (*keto*$_{ax}$) and 21.4 ppm; *m/z* (ESI+) 329 $(M + Na)^+$, 307 $(M + H)^+$. (Found 307.1536 $(M + Na)^+$. $C_{17}H_{23}O_5$ requires 307.1540).

Methyl 6-(2-(benzyloxy)ethyl)-4-hydroxy-2,2-dimethyl-5,6-dihydro-2H-pyran-3-carboxylate (25f).
Oil ($keto_{eq}$: enol : $keto_{ax}$ 2.37 : 4.19 : 1); υ_{max} (film) 3583, 3034, 3030, 2927, 2862, 1751, 1718, 1646, 1608, 1440, 1359, 1286, 1218 cm^{-1}; δ_H (400 MHz, C_6D_6) 13.28 (1H, s) 7.28-7.10 (5H, m), 7.28-7.10 (5H, m, *keto*$_{eq}$), 7.28-7.10 (5H, m, *keto*$_{ax}$), 4.32-4.20 (2H, m), 4.32-4.20 (2H, m, *keto*$_{eq}$), 4.32-4.20 (2H, m, *keto*$_{ax}$), 3.90 (1H, m, *keto*$_{ax}$), 3.87-3.79 (1H, m), 3.87-3.79 (1H, m, *keto*$_{eq}$), 3.55-3.25 (2H, m), 3.55-3.25 (2H, m, *keto*$_{eq}$), 3.55-3.25 (2H, m, *keto*$_{ax}$), 3.55-3.25 (1H, m, *keto*$_{eq}$), 3.37 (3H, s, *keto*$_{eq}$), 3.23 (3H, s), 3.20 (1H, d, J = 1.3 Hz, *keto*$_{ax}$), 3.15 (3H, s, *keto*$_{ax}$), 3.04 (1H, dd, J = 14.1, 11.5 Hz, *keto*$_{ax}$), 2.24 (1H, m, *keto*$_{ax}$), 2.17 (1H, dd, J = 17.8, 10.8 Hz), 2.09-2.02 (1H, m), 2.09-2.02 (1H, m, *keto*$_{eq}$), 1.89-1.42 (2H, m), 1.89-1.42 (2H, m, *keto*$_{eq}$), 1.89-1.42 (2H, m, *keto*$_{ax}$), 1.89-1.42 (1H, m, *keto*$_{eq}$), 1.56 (3H, s), 1.43 (3H, s), 1.42 (3H, s, *keto*$_{ax}$), 1.38 (3H, s, *keto*$_{eq}$), 1.27 (3H, s, *keto*$_{eq}$) and 1.18 (3H, s, *keto*$_{ax}$) ppm; δ_C (100 MHz, C_6D_6) 201.3 (*keto*), 172.4, 172.2, 168.2, 139.3, 139.1, 128.6, 128.5, 127.9, 127.7, 105.2, 77.1, 75.9, 73.3 (*keto*), 173.1, 173.1 (*keto*), 68.0 (*keto*$_{ax}$), 67.1, 66.5, 66.2, 66.0, 63.8, 51.4 (*keto*$_{ax}$), 50.9, 47.0 (*keto*$_{eq}$), 37.0, 36.1, 29.9, 29.3 (*keto*$_{eq}$), 27.8 (*keto*$_{ax}$), 25.8 and 21.6 (*keto*$_{eq}$) ppm; *m/z* (ESI+) 343 $(M + Na)^+$, 321 $(M + H)^+$. (Found 343.1517 $(M + Na)^+$. $C_{18}H_{24}NaO_5$ requires 343.1516).

Methyl 2-butyl-4-hydroxy-2-methyl-6-phenyl-5,6-dihydro-2H-pyran-3-carboxylate (25g).
Oil ($keto_{eq}$: enol : $keto_{ax}$ 0.85 : 6.35 : 1); υ_{max} (film) 3044, 3022, 2988, 2911, 2827, 1712, 1692, 1619, 1583, 1474, 1454, 1420, 1356, 1315, 1255, 1201, 1047 cm^{-1}; nOe: H-6 – H-8 4%, *keto*$_{eq}$ H-3 – H-5_{ax} 1.6%; δ_H (400 MHz, C_6D_6) 13.37 (1H, s), 7.43-7.41 (1H, m, *keto*$_{eq}$), 7.43-7.41 (1H, m, *keto*$_{ax}$), 7.31-7.29 (2H, m), 7.18-7.04 (3H, m), 7.18-7.04 (4H, m, *keto*$_{eq}$), 7.18-7.04 (4H, m, *keto*$_{ax}$), 4.61 (2H, dd, J = 11.6, 3.1 Hz, *keto*$_{eq}$ &), 4.56 (1H, dd, J = 10.8, 3.6 Hz), 3.56 (1H, s, *keto*$_{eq}$), 3.46 (1H, dd, J = 14.0, 11.6 Hz, *keto*$_{ax}$), 3.40 (4H, s, *keto*$_{eq}$ & *keto*$_{ax}$), 3.27 (3H, s), 3.22 (3H, s, *keto*$_{ax}$), 2.50 (1H, dd, J = 17.4, 10.8 Hz), 2.47 (1H, m, *keto*$_{ax}$), 2.41 (1H, m, *keto*$_{eq}$), 2.36 (1H, dd, J = 17.4, 3.6 Hz), 2.09 (1H, dd, J = 13.4, 11.6 Hz, *keto*$_{eq}$), 2.03-1.95 (1H, m), 2.03-1.95 (1H, m, *keto*$_{eq}$), 2.03-1.95 (1H, m, *keto*$_{ax}$), 1.83-1.76 (1H, m), 1.83-1.76 (1H, m, *keto*$_{eq}$), 1.83-1.76 (1H, m, *keto*$_{ax}$), 1.60 (3H, s), 1.56-1.07 (4H, m), 1.56-1.07 (7H, m, *keto*$_{eq}$), 1.56-1.07 (7H, m, *keto*$_{ax}$), 0.83 (3H, t, J = 7.3 Hz) and 0.79-0.72 (6H, m, *keto*$_{eq}$ & *keto*$_{ax}$) ppm; δ_C (100 MHz, C_6D_6) 202.0 (*keto*), 200.7 (*keto*), 172.5, 171.1, 168.1 (*keto*), 168.0 (*keto*), 142.1, 142.0, 141.5, 128.7, 128.6, 128.1, 127.9, 126.6, 126.1, 106.3, 79.8 (*keto*), 78.6 (*keto*), 75.8, 73.0 (*keto*), 72.4 (*keto*), 68.1, 68.0 (*keto*$_{eq}$), 65.8 (*keto*$_{ax}$), 51.6 (*keto*), 51.0, 48.7 (*keto*$_{eq}$), 47.0 (*keto*$_{ax}$), 37.4, 36.7, 36.4, 31.4, 26.1, 25.9, 25.7, 25.0, 24.3, 24.1, 23.5, 23.2, 23.0, 14.3, 14.2 (*keto*), and 14.1 (*keto*) ppm; *m/z* (ESI+) 327 $(M + Na)^+$, 305 $(M + H)^+$. (Found 305.1746 $(M + H)^+$. $C_{18}H_{25}O_4$ requires 305.1747).

Methyl 2-butyl-4-hydroxy-2-methyl-6-propyl-5,6-dihydro-2H-pyran-3-carboxylate (25h).
Oil ($keto_{eq}$: enol : $keto_{ax}$ 1 : 5. 9 : 1); υ_{max} (film) 2918, 2889, 2827, 1724, 1693, 1618, 1583, 1419, 1355, 1324, 1265, 1243, 1200, 1113, 1051, 1023 cm^{-1}; nOe: H-6 – H-8 2.8%, *keto*$_{eq}$ H-3 H-5_{ax} 0.12%; δ_H (400 MHz, C_6D_6) 13.41 (1H, s), 3.47-3.40 (1H, m), 3.47-3.40 (1H, m, *keto*$_{eq}$), 3.47-3.40 (1H, m, *keto*$_{ax}$), 3.46 (1H, m, *keto*$_{eq}$), 3.38 (3H, s, *keto*), 3.32 (1H, d, J = 1.2 Hz, *keto*$_{ax}$), 3.25 (3H, s), 3.19 (3H, s, *keto*), 3.07 (1H, dd, J = 14.0, 11.3 Hz, *keto*$_{ax}$), 2.19 (1H, m, *keto*$_{ax}$), 2.15 (1H, dd, J = 17.4, 10.7 Hz), 2.02 (1H, dd, J = 17.4, 3.4 Hz), 2.07-1.91 (1H, m), 2.07-1.91 (1H, m, *keto*$_{eq}$), 2.07-1.91 (1H, m, *keto*$_{ax}$), 2.07-1.91 (1H, m, *keto*$_{eq}$), 1.74-1.65 (1H, m), 1.74-1.65 (1H, m, *keto*$_{eq}$), 1.74-1.65 (1H, m, *keto*$_{ax}$), 1.74-1.65 (1H, m, *keto*$_{eq}$), 1.55 (3H, s), 1.31 (3H, s, *keto*), 1.18 (3H, s, *keto*), 1.60-0.74 (8H, m), 1.60-0.74 (8H, m, *keto*$_{eq}$), 1.60-0.74 (8H, m, *keto*$_{ax}$), 0.95 (3H, t, J = 7.3 Hz), 0.80 (3H, t, J = 7.0 Hz), 0.80 (6H, t, J = 7.0 Hz, *keto*$_{eq}$) and 0.80 (6H, t, J = 7.0 Hz, *keto*$_{ax}$) ppm; δ_C (100 MHz, C_6D_6) 202.9 (*keto*), 201.4 (*keto*), 172.6, 172.2, 168.2 (*keto*), 168.1 (*keto*), 106.4, 79.1 (*keto*), 77.9 (*keto*), 74.9, 70.0 (*keto*), 68.2 (*keto*$_{eq}$), 66.0 (*keto*$_{ax}$), 65.7 (C-6), 51.4 (*keto*), 50.9, 47.2 (*keto*$_{eq}$), 45.4 (*keto*$_{ax}$), 39.1, 39.0, 36.6, 36.1, 31.4, 26.1, 25.8, 25.7, 24.9, 24.2, 24.1, 23.6, 23.2, 23.1, 19.0, 18.9, 18.8, 14.5 and 14.2 ppm; *m/z* (ESI+) 293 $(M + Na)^+$, 271 $(M + H)^+$, 171. (Found 293.1715 $(M + Na)^+$. $C_{15}H_{26}NaO_4$ requires 293.1723).

Methyl 4-hydroxy-2-methyl-6-phenyl-2-vinyl-5,6-dihydro-2H-pyran-3-carboxylate (25i).
Oil ($keto_{eq}$: enol : $keto_{ax}$ 0.06 : 1 : 0.06); υ_{max} (film) 3040, 3020, 2986, 2933, 2907, 1693, 1620, 1584, 1420, 1254, 1202, 1048 cm^{-1}; nOe: H-6 – H-7 0.44%, H-6 – H-8 0.76%, *keto*$_{ax}$ H-3 – H-8 1.8%, *keto*$_{eq}$ H-3 – H-5_{ax} 1.2%, H-5_{ax} – H-9 0.3%; δ_H (400 MHz, C_6D_6) 13.40 (1H, s), 7.30-7.00 (5H, m), 7.30-7.00 (5H, m, *keto*$_{eq}$), 7.30-7.00 (5H, m, *keto*$_{ax}$), 6.50 (1H, dd, J = 17.7, 11.0 Hz, *keto*$_{eq}$), 5.94 (1H, dd, J = 17.4, 10.4 Hz), 5.39 (1H, dd, J = 18.0, 11.0 Hz, *keto*$_{ax}$), 5.28 (1H, dd, J = 17.7, 1.2 Hz, *keto*$_{eq}$), 5.12 (1H, dd, J = 11.0, 1.2 Hz, *keto*$_{ax}$), 5.07-4.96

(2H, m), 5.07-4.96 (1H, m, *keto$_{eq}$*), 5.07-4.96 (1H, m, *keto$_{ax}$*), 4.90-4.88 (2H, m, *keto*), 4.70 (1H, dd, *J* = 11.3, 3.4 Hz, *keto$_{eq}$*), 4.59 (1H, dd, *J* = 11.3, 3.4 Hz), 3.74 (1H, d, *J* = 1.2 Hz, *keto$_{ax}$*), 3.48 (1H, s, *keto$_{eq}$*), 3.35 (1H, m, *keto$_{ax}$*) 3.34 (3H, s, *keto*), 3.20 (3H, s), 3.14 (3H, s, *keto*), 2.47 (1H, dd, *J* = 17.7, 11.3 Hz), 2.43 (1H, m, *keto$_{ax}$*), 2.28 (1H, m, *keto$_{eq}$*), 2.27 (1H, dd, *J* = 17.7, 3.4 Hz), 2.04 (1H, ddd, *J* = 13.7, 11.6, 0.9 Hz, *keto$_{eq}$*), 1.67 (3H, s), 1.45 (3H, s, *keto$_{eq}$*) and 1.27 (3H, s, *keto$_{ax}$*) ppm; δ_C (100 MHz, C_6D_6) 173.2, 172.4, 141.8, 141.6, 128.6, 127.9, 126.2, 114.4, 102.1, 76.6, 68.8, 51.0, 37.4 and 29.1 ppm; *m/z* (ESI+) 297 $(M + Na)^+$, 275 $(M + H)^+$. (Found 275.1281 $(M + H)^+$. $C_{16}H_{19}O_4$ requires 275.1278).

Methyl 4-hydroxy-2-methyl-6-propyl-2-vinyl-5,6-dihydro-2H-pyran-3-carboxylate (25j).
Oil (keto$_{eq}$: enol : keto$_{ax}$ 0.05 : 1 : 0.05); υ_{max} (film) 2913, 2889, 2829, 1621, 1584, 1420, 1328, 1202, 1049 cm^{-1}; nOe: H-6 – H-7 0.5%, H-6 – H-8 0.7%; δ_H (400 MHz, C_6D_6) 13.46 (1H, s), 6.51 (1H, dd, *J* = 17.7, 11.0 Hz, *keto*), 5.95 (1H, dd, *J* = 19.8, 8.2 Hz), 5.42 (1H, dd, *J* = 18.0, 11.0 Hz, *keto*), 5.30 (1H, dd, *J* = 17.7, 1.2 Hz, *keto*), 5.09-4.89 (2H, m), 5.09-4.89 (3H, m, *keto*), 3.78 (1H, m, *keto*), 3.71 (1H, d, *J* = 1.2 Hz, *keto$_{ax}$*), 3.64 (1H, m, *keto*), 3.54 (1H, m), 3.44 (1H, s, *keto$_{eq}$*), 3.37 (3H, s, *keto*), 3.26 (3H, s), 3.17 (3H, s, *keto*), 2.98 (1H, dd, *J* = 14.3, 11.3 Hz, *keto$_{ax}$*), 2.15 (1H, dd, *J* = 17.5, 11.0 Hz), 2.13 (1H, m, *keto$_{ax}$*), 2.00 (1H, dd, *J* = 17.5, 3.1 Hz), 1.98 (1H, m, *keto$_{eq}$*), 1.72 (1H, *keto$_{eq}$*), 1.66 (3H, s), 1.50-1.08 (4H, m), 1.50-1.08 (14H, m, *keto*), 0.97-0.72 (6H, m, *keto*) and 0.79 (3H, t, *J* = 7.0 Hz) ppm; δ_C (100 MHz, C_6D_6) 173.6, 172.5, 142.0, 114.3, 102.1, 75.7, 66.3, 50.9, 37.9, 36.0, 29.1, 18.7 and 14.1 ppm; *m/z* (ESI+) 263 $(M + Na)^+$, 241 $(M + H)^+$. (Found 263.1247 $(M + Na)^+$. $C_{13}H_{20}NaO_4$ requires 263.1254).

Methyl 4-hydroxy-6-isopropyl-2-methyl-2-vinyl-5,6-dihydro-2H-pyran-3-carboxylate (25k).
Oil (keto$_{eq}$: enol : keto$_{ax}$ 0.06 : 1 : 0.06); υ_{max} (film) 3037, 2913, 1694, 1621, 1585, 1420, 1344, 1255, 1205, 1048 cm^{-1}; nOe: H-6 – H-7 0.4%, H-6 – H-8 0.4%, *keto$_{ax}$* H-3 – H-8 1.1%, *keto$_{eq}$* H-3 – H-5$_{ax}$ 0.4% *keto$_{eq}$* H-3 – H9 0.5%; δ_H (400 MHz, C_6D_6) 13.46 (1H, s), 6.55 (1H, m, *keto$_{eq}$*), 5.91 (1H, dd, *J* = 17.4, 10.7 Hz), 5.41 (1H, dd, *J* = 18.0, 11.0 Hz, *keto$_{ax}$*), 5.29 (1H, dd, *J* = 17.7, 1.2 Hz, *keto*), 5.16-4.89 (2H, m), 5.16-4.89 (3H, m, *keto*), 3.71 (1H, d, *J* = 1.2 Hz, *keto$_{ax}$*), 3.58 (1H, m, *keto*), 3.43 (1H, m, *keto*), 3.41 (1H, s, *keto$_{eq}$*), 3.38 (3H, s, *keto*), 3.28 (1H, m), 3.25 (3H, s), 3.18 (3H, s, *keto*), 3.01 (1H, dd, *J* = 14.3, 11.6 Hz, *keto$_{ax}$*), 2.21 (1H, dd, *J* = 17.4, 11.0 Hz), 2.19 (1H, m, *keto$_{ax}$*), 2.06 (1H, m, *keto$_{eq}$*), 2.05 (1H, dd, *J* = 17.4, 3.4 Hz), 1.76 (1H, dd, *J* = 13.1, 11.6 Hz, *keto$_{eq}$*), 1.65 (3H, s), 1.55 (1H, m), 1.43 (3H, s, *keto$_{eq}$*), 1.36-1.28 (2H, m, *keto*), 1.27 (3H, s, *keto$_{ax}$*), 0.91 (3H, d, *J* = 7.0Hz), 0.98-0.84 (12H, m, *keto*) and 0.71, (3H, d, *J* = 7.0 Hz) ppm; δ_C (100 MHz, C_6D_6) 173.8, 172.5, 142.0, 114.6, 102.0, 75.7, 71.4, 50.9, 33.3, 32.9, 28.9, 18.5 and 18.0 ppm; *m/z* (ESI+) 263 $(M +Na)^+$, 241 $(M + H)^+$, 209. (Found 263.1252 $(M + Na)^+$. $C_{13}H_{20}NaO_4$ requires 263.1254).

Methyl 6-(furan-2-yl)-4-hydroxy-2-methyl-2-vinyl-5,6-dihydro-2H-pyran-3-carboxylate (25l).
Oil (keto$_{eq}$: enol : keto$_{ax}$ 0.09 : 1 : 0.06); υ_{max} (film) 2911, 2886, 2829, 1694, 1622, 1587, 1420, 1389, 1357, 1334, 1312, 1259, 1199, 1163, 1036 cm^{-1}; nOe: H-6 – H-8 0.5%; δ_H (400 MHz, C_6D_6) 13.43 (1H, s), 7.04 (1H, dd, *J* = 1.5, 0.8 Hz), 7.01-6.99 (2H, m, *keto*), 6.48 (1H, m, *keto$_{eq}$*), 6.11-5.93 (1H, m), 6.11-5.93 (3H, m), 6.11-5.93 (3H, m), 5.49-5.42 (1H, m, *keto$_{ax}$*), 5.49-5.42 (1H, m, *keto$_{eq}$*), 5.24 (1H, dd, *J* = 17.4, 1.5 Hz), 5.18 (1H, dd, *J* = 11.0, 1.1 Hz, *keto$_{ax}$*), 5.10 (1H, dd, *J* = 10.4, 1.5 Hz), 4.99 (1H, d, *J* = 18.1 Hz, *keto$_{eq}$*), 4.95 (1H, dd, *J* = 11.9, 3.5 Hz, *keto$_{ax}$*), 4.89 (1H, d, *J* = 11.0 Hz, *keto$_{ax}$*), 4.81 (1H, dd, *J* = 11.9, 2.9 Hz, *keto$_{eq}$*), 4.73 (1H, dd, *J* = 11.6, 3.1 Hz), 3.71 (1H, d, *J* = 1.3 Hz, *keto$_{eq}$*), 3.63 (1H, dd, *J* = 14.5, 11.9 Hz, *keto$_{ax}$*), 3.52 (1H, s, *keto$_{ax}$*), 3.34 (3H, s, *keto$_{ax}$*), 3.21 (3H, s), 3.16 (3H, s, *keto$_{eq}$*), 2.87 (1H, dd, *J* = 17.6, 11.6 Hz), 2.52-2.40 (1H, m, *keto$_{ax}$*), 2.52-2.40 (1H, m, *keto$_{eq}$*), 2.27 (1H, m, *keto$_{eq}$*), 2.25 (1H, dd, *J* = 17.6, 3.1 Hz) and 1.64 (9H, s) ppm; δ_C (100 MHz, C_6D_6) 172.6, 172.2, 154.0, 142.5, 141.3, 114.6, 110.4, 107.4, 102.2, 76.7, 63.1, 51.0, 33.7 and 29.0 ppm; *m/z* (ESI+) 287 $(M + Na)^+$. (Found 287.0917 $(M + Na)^+$. $C_{14}H_{16}NaO_5$ requires 287.0890).

Methyl 6-(benzyloxymethyl)-4-hydroxy-2-methyl-2-vinyl-5,6-dihydro-2H-pyran-3-carboxylate (25m).
Oil (keto$_{eq}$: enol : keto$_{ax}$ 0.1 : 1 : 0.07); υ_{max} (film) 2984, 2908, 2886, 2818, 1694, 1620, 1584, 1420, 1388, 1346, 1259, 1201, 1079, 1046 cm^{-1}; nOe: H-6 – H-8 0.5%; δ_H (400 MHz, C_6D_6) 13.42 (1H, s), 7.25-7.19 (2H, m), 7.25-7.19 (4H, m, *keto*), 7.17-7.12 (2H, m), 7.17-7.12 (4H, m, *keto*), 7.10-7.05 (1H, m), 7.10-7.05 (2H, m, *keto*), 6.44 (1H, dd, *J* = 17.7, 11.0 Hz, *keto$_{eq}$*), 5.98 (1H, dd, *J* = 17.4, 11.0 Hz), 5.40 (1H, m, *keto-d*), 5.19 (1H, dd, *J* = 17.4, 1.5 Hz), 5.18 (1H, m, *keto$_{ax}$*), 5.14 (1H, m, *keto$_{eq}$*), 5.07 (1H, dd, *J* = 11.0, 1.5 Hz), 4.96 (1H, d, *J* =

17.7 Hz, *keto*$_{ax}$), 4.87 (1H, d, *J* = 11.0 Hz, *keto*$_{eq}$), 4.35-4.26 (2H, m), 4.35-4.26 (4H, m, *keto*), 4.06 (1H, m, *keto*$_{eq}$), 3.96 (1H, m, *keto*$_{ax}$), 3.91 (1H, m), 3.69 (1H, d, *J* = 1.5 Hz, *keto*$_{eq}$), 3.47-3.33 (1H, m, *keto*$_{ax}$), 3.47-3.33 (1H, m), 3.47-3.33 (2H, m, *keto*), 3.26-3.11 (1H, m), 3.26-3.11 (2H, m, *keto*), 3.20 (3H, s), 3.15 (6H, s, *keto*), 3.08 (1H, dd, *J* = 14.6, 11.6 Hz, *keto*$_{ax}$), 2.34 (1H, dd, *J* = 17.7, 11.0 Hz), 2.23 (1H, m, *keto*$_{ax}$), 2.11-2.08 (2H, m, *keto*$_{eq}$), 2.07 (1H, dd, *J* = 17.7, 3.4 Hz), 1.65 (3H, s) and 1.63 (6H, s, *keto*) ppm; δ_C (100 MHz, C_6D_6) 173.1, 172.3, 141.4, 139.0, 128.5, 127.7, 114.4, 102.2, 76.1, 73.4, 72.7, 66.6, 50.9, 32.7 and 29.1 ppm; *m/z* (ESI+) 341 $(M + Na)^+$, 319 $(M + H)^+$. (Found 319.1538 $(M + H)^+$. $C_{18}H_{23}O_5$ requires 319.1540).

Methyl 6-(2-(benzyloxy)ethyl)-4-hydroxy-2-methyl-2-vinyl-5,6-dihydro-2H-pyran-3-carboxylate (25n).
Oil (keto$_{eq}$: enol : keto$_{ax}$ 0.07 : 1 : 0.07); υ_{max} (film) 2984, 2905, 2817, 1694, 1620, 1584, 1420, 1343, 1266, 1248, 1202, 1082, 1046 cm^{-1}; nOe: H-6 – H-8 0.4%; δ_H (400 MHz, C_6D_6) 13.44 (1H, s), 7.30-7.22 (2H, m), 7.30-7.22 (4H, m, *keto*), 7.19-7.15 (2H, m), 7.19-7.15 (4H, m, *keto*), 7.11-7.07 (1H, m), 7.11-7.07 (2H, m, *keto*), 6.50 (1H, dd, *J* = 17.7, 11.0 Hz, *keto*), 5.94 (1H, dd, *J* = 17.7, 10.4 Hz), 5.42 (1H, dd, *J* = 17.7, 11.0 Hz, *keto*), 5.32 (1H, dd, *J* = 17.7, 1.2 Hz, *keto*), 5.14 (1H, m, *keto*), 5.06 (2H, m), 4.98 (1H, d, *J* = 17.7 Hz, *keto*), 4.88 (1H, d, *J* = 11.0 Hz, *keto*), 4.32-4.19 (2H, m), 4.32-4.19 (4H, m, *keto*), 4.12 (1H, m, *keto*), 4.02 (1H, m, *keto*), 3.86 (1H, m), 3.70 (1H, d, J = 1.2 Hz, *keto*$_{eq}$), 3.51-3.43 (1H, m), 3.51-3.43 (2H, m, *keto*), 3.39-3.33 (1H, m, *keto*$_{ax}$), 3.39-3.33 (2H, m), 3.39-3.33 (4H, m, *keto*), 3.23 (3H, s), 3.16 (6H, s, *keto*), 3.00 (1H, dd, *J* = 14.3, 11.6 Hz, *keto*$_{ax}$), 2.22 (1H, m, *keto*$_{ax}$), 2.20 (1H, dd, *J* = 17.7, 11.0 Hz), 2.04 (1H, dd, *J* = 17.7, 7.3 Hz), 2.02 (1H, m, *keto*$_{eq}$), 1.77-1.57 (1H, m, *keto*$_{eq}$), 1.77-1.57 (1H, m), 1.77-1.57 (2H, m, *keto*), 1.65 (3H, s) and 1.65 (6H, s, *keto*) ppm; δ_C (100 MHz, C_6D_6) 173.4, 172.4, 141.8, 139.2, 128.5, 127.6, 114.4, 102.1, 75.8, 73.0, 66.4, 64.0, 50.9, 36.0, 35.9 and 29.0 ppm; *m/z* (ESI+) 355 $(M + Na)^+$, 333 $(M + H)^+$. (Found 333.1696 $(M + H)^+$. $C_{19}H_{25}O_5$ requires 333.1697).

Methyl 4-hydroxy-2-methyl-2,6-diphenyl-5,6-dihydro-2H-pyran-3-carboxylate (25o).
Oil; υ_{max} (film) 3040, 3014, 2986, 2938, 2906, 2861, 1712, 1692, 1621, 1471, 1420, 1347, 1254, 1045, 942 cm^{-1}; nOe: H-6 – Ph 0.9%; δ_H (400 MHz, C_6D_6) 13.74 (1H, s), 7.51-7.48 (2H, m), 7.25-7.05 (8H, m), 4.36 (1H, dd, *J* = 11.3, 3.7 Hz), 3.22 (3H, s), 2.68 (1H, dd, *J* = 17.8, 11.3 Hz), 2.40 (1H, dd, *J* = 17.8, 3.8 Hz) and 2.10 (3H, s) ppm; δ_C (100 MHz, C_6D_6) 173.5, 172.9, 145.4, 141.7, 128.6, 128.5, 128.2, 127.7, 127.6, 126.2, 103.3, 78.0, 68.6, 51.0, 36.9 and 29.9 ppm; *m/z* (ESI+) 347 $(M + Na)^+$. (Found 347.1253 $(M + Na)^+$. $C_{20}H_{20}NaO_4$ requires 347.1254).

Methyl 4-hydroxy-2-methyl-2-phenyl-6-propyl-5,6-dihydro-2H-pyran-3-carboxylate (25p).
Oil; υ_{max} (film) 3371, 2912, 2887, 2829, 1712, 1692, 1619, 1584, 1419, 1243, 1144, 1050 cm^{-1}; nOe: H-6 – Ph 0.7%; δ_H (400 MHz, C_6D_6) 13.64 (1H, s), 7.41-7.38 (2H, m), 7.18-7.06 (3H, m), 3.13 (3H, s), 3.13 (1H, m), 2.21 (1H, dd, *J* = 18.6, 10.4 Hz), 1.99 (1H, dd, *J* = 18.6, 3.1 Hz), 1.98 (3H, s), 1.37-1.18 (2H, m), 1.06-0.92 and 0.59 (3H, t, *J* = 6.7 Hz) ppm; δ_C (100 MHz, C_6D_6) 173.9, 172.9, 145.6, 128.6, 127.4, 103.4, 77.2, 66.2, 50.9, 37.8, 36.0, 29.8, 18.5 and 13.8 ppm; *m/z* (ESI+) 313 $(M + Na)^+$, 291 $(M + H)^+$, 172 $(M - CO_2Me - C_3H_7)^+$. (Found 291.1581 $(M + H)^+$. $C_{17}H_{23}O_4$ requires 291.1591).

Methyl 4-hydroxy-6-isopropyl-2-methyl-2-phenyl-5,6-dihydro-2H-pyran-3-carboxylate (25q).
Oil; υ_{max} (film) 2912, 2832, 1620, 1584, 1420, 1365, 1345, 1256, 1045 cm^{-1}; nOe: H-6 – Ph 0.7% δ_H (400 MHz, C_6D_6) 13.66 (1H, s), 7.40-7.37 (2H, m), 7.19-7.15 (2H, m), 7.09 (1H, m), 3.15 (3H, s), 2.87 (1H, m), 2.29 (1H, dd, *J* = 17.7, 11.6 Hz), 2.08 (1H, dd, *J* = 17.7, 3.4 Hz), 1.98 (3H, s), 1.42 (1H, m), 0.77 (3H, d, *J* = 6.7 Hz) and 0.52 (3H, d, *J* = 6.7 Hz) ppm; δ_C (100 MHz, C_6D_6) 174.1, 172.9, 145.4, 127.9, 127.9, 127.4, 103.3, 77.2, 71.6, 50.9, 33.5, 32.9, 29.8, 18.4 and 17.8 ppm; *m/z* (ESI+) 313 $(M + Na)^+$. (Found 313.1412 $(M + Na)^+$. $C_{17}H_{22}NaO_4$ requires 313.1410).

Methyl 6-(furan-2-yl)-4-hydroxy-2-methyl-2-phenyl-5,6-dihydro-2H-pyran-3-carboxylate (25r).
Oil; υ_{max} (film) 3012, 2985, 2939, 2907, 2871, 1693, 1621, 1586, 1476, 1421, 1393, 1359, 1334, 1312, 1262, 1199, 1134, 1043, 1028 cm^{-1}; nOe: H-6 – Ph 0.9% δ_H (400 MHz, C_6D_6) 13.64 (1H, s), 7.47-7.44 (2H, m), 7.20-7.07 (3H, m), 6.94 (1H, dd, *J* = 1.8, 0.7 Hz), 5.92 (1H, dd, *J* = 3.3, 1.8 Hz), 5.88 (1H, d, *J* = 3.3 Hz), 4.34 (1H, dd, *J* = 11.7, 3.3 Hz), 3.11 (3H, s), 2.95 (1H, dd, *J* = 17.9, 11.7 Hz), 2.23 (1H, dd, *J* = 17.9, 3.3 Hz) and 1.97

(3H, s) ppm; δ_C (100 MHz, C_6D_6) 173.0, 172.8, 153.8, 145.1, 142.4, 127.9, 127.7, 110.3, 107.3, 103.4, 78.1, 63.1, 51.0, 33.5 and 29.8 ppm; *m/z* (ESI+) 337 $(M + Na)^+$. (Found 337.1047 $(M + Na)^+$. $C_{18}H_{18}NaO_5$ requires 337.1046).

Methyl 6-(benzyloxymethyl)-4-hydroxy-2-methyl-2-phenyl-5,6-dihydro-2H-pyran-3-carboxylate (25s).
Oil; υ_{max} (film) 2985, 2904, 2859, 2817, 1619, 1584, 1421, 1346, 1325, 1261, 1202, 1082, 1043 cm^{-1}; nOe: H-6 – Ph 1% δ_H (400 MHz, C_6D_6) 13.70 (1H, s), 7.50-7.46 (2H, m), 7.24-7.11 (8H, m), 4.22 (2H, s), 3.57 (1H, m), 3.26 (1H, dd, *J* = 10.1, 5.5 Hz), 3.17 (3H, s), 3.13 (1H, dd, *J* = 10.1, 4.6 Hz), 2.51 (1H, dd, *J* = 18.0, 11.3 Hz), 2.15 (1H, dd, *J* = 18.0, 3.4 Hz) and 2.02 (3H, s) ppm; δ_C (100 MHz, C_6D_6) 173.6, 172.8, 145.2, 138.9, 128.5, 128.1, 127.6, 127.6, 127.5, 103.4, 77.5, 73.1, 72.5, 66.4, 51.0, 32.5 and 29.8 ppm; *m/z* (ESI+) 391 $(M + Na)^+$, 369 $(M + H)^+$. (Found 369.1690 $(M + H)^+$. $C_{22}H_{25}O_5$ requires 369.1697).

Methyl 4-acetoxy-2,2-dimethyl-6-propyl-5,6-dihydro-2H-pyran-3-carboxylate (26b).
Oil; υ_{max} (film) 2957, 2933, 2872, 1767, 1722, 1435, 1364, 1278, 1238, 1191, 1058 cm^{-1}; δ_H (400 MHz, C_6D_6) 3.62 (1H, m), 3.31 (3H, s), 2.30 (1H, dd, J = 17.2, 10.6 Hz), 1.84 (1H, dd, J = 17.2, 3.3 Hz), 1.77 (3H, s), 1.59 (3H, s), 1.55 (3H, s), 1.50-1.33 (4H, m) and 0.81 (3H, t, J = 7.1 Hz) ppm; δ_C (100 MHz, C_6D_6) 167.6, 165.7, 151.2, 126.0, 74.3, 67.3, 51.2, 37.8, 35.0, 28.7, 26.0, 20.4, 18.8 and 14.1 ppm; m/z (ESI+) 293 $(M + Na)^+$, 271 $(M + H)^+$. (Found 293.1358 $(M + Na)^+$. $C_{14}H_{22}NaO_5$ requires 293.1359).

Methyl 4-acetoxy-6-isopropyl-2,2-dimethyl-5,6-dihydro-2H-pyran-3-carboxylate (26c).
Oil; υ_{max} (film) 2915, 2831, 1740, 1697, 1413, 1347, 1315, 1227, 1201, 1175, 1042 cm^{-1}; δ_H (400 MHz, C_6D_6) 3.32 (1H, m), 3.32 (3H, s), 2.36 (1H, dd, *J* = 16.8, 10.6 Hz), 1.90 (1H, dd, *J* = 16.8, 3.3 Hz), 1.78 (3H, s), 1.58 (3H, s), 1.56 (1H, m), 1.54 (3H, s), 0.93 (3H, d, *J* = 7.0 Hz) and 0.71 (3H, d, *J* = 7.0 Hz) ppm; δ_C (100 MHz, C_6D_6) 167.6, 165.6, 151.6, 126.0, 74.3, 72.6, 51.1, 32.9, 32.6, 28.6, 25.8, 20.5, 18.6 and 18.0 ppm; *m/z* (ESI+) 293 $(M + Na)^+$. (Found 293.1353 $(M + Na)^+$. $C_{14}H_{22}NaO_5$ requires 293.1359).

Methyl 4-acetoxy-6-(furan-2-yl)-2,2-dimethyl-5,6-dihydro-2H-pyran-3-carboxylate (26d).
Oil; υ_{max} (film) 2934, 2892, 1739, 1696, 1414, 1344, 1301, 1248, 1218, 1192, 1158, 1036 cm^{-1}; δ_H (400 MHz, C_6D_6) 7.03 (1H, dd, *J* = 1.8, 0.9 Hz), 6.09 (1H, m), 6.01 (1H, dd, *J* = 3.1, 1.8 Hz), 4.80 (1H, dd, *J* = 11.0, 3.4 Hz), 3.28 (3H, s), 2.99 (1H, dd, *J* = 17.1, 11.0 Hz), 2.12 (1H, dd, *J* = 17.1, 3.4 Hz), 1.73 (3H, s), 1.63 (3H, s) and 1.54 (3H, s) ppm; δ_C (100 MHz, C_6D_6) 167.5, 165.3, 154.1, 150.4, 142.4, 126.0, 110.4, 107.4, 75.3, 64.0, 51.2, 33.0, 28.4, 25.9 and 20.3 ppm; *m/z* (ESI+) 317 $(M + Na)^+$. (Found 317.0992 $(M + Na)^+$. $C_{15}H_{18}NaO_6$ requires 317.0996).

Methyl 4-acetoxy-6-(benzyloxymethyl)-2,2-dimethyl-5,6-dihydro-2H-pyran-3-carboxylate (26e).
Oil; υ_{max} (film) 2934, 2891, 2864, 2822, 1740, 1696, 1431, 1413, 1343, 1310, 1291, 1223, 1194, 1174, 1158, 1085, 1041 cm^{-1}; δ_H (400 MHz, C_6D_6) 7.25-7.23 (2H, m), 7.17-7.13 (2H, m), 7.07 (1H, m), 4.33 (1H, d, *J* = 12.2 Hz), 4.29 (1H, d, *J* = 12.2 Hz), 4.00 (1H, m), 3.34 (1H, dd, *J* = 10.1, 5.5 Hz), 3.30 (3H, s), 3.25 (1H, dd, *J* = 10.1, 5.2 Hz), 2.50 (1H, dd, *J* = 17.4, 10.4 Hz), 1.99 (1H, dd, *J* = 17.4, 3.4 Hz), 1.74 (3H, s), 1.59 (3H, s) and 1.53 (3H, s) ppm; δ_C (100 MHz, C_6D_6) 167.6, 165.5, 150.9, 139.0, 128.5, 127.8, 127.7, 126.0, 74.7, 73.5, 72.9, 67.4, 51.1, 32.0, 28.5, 25.9 and 20.4 ppm; *m/z* (ESI+) 371 $(M + Na)^+$. (Found 371.1453 $(M + Na)^+$. $C_{19}H_{24}NaO_6$ requires 371.1465).

Methyl 4-acetoxy-6-(2-(benzyloxy)ethyl)-2,2-dimethyl-5,6-dihydro-2H-pyran-3-carboxylate (26f).
Oil; υ_{max} (film) 2984, 2887, 2819, 1739, 1696, 1431, 1413, 1347, 1222, 1193, 1172, 1084, 1042 cm^{-1}; δ_H (400 MHz, C_6D_6) 7.30-7.05 (5H, m), 4.31-4.25 (2H, m), 3.97 (1H, m), 3.47 (1H, m), 3.36 (1H, m), 3.30 (3H, s), 2.35 (1H, dd, J = 17.6, 10.1 Hz), 1.85 (1H, dd, J = 17.6, 2.8 Hz), 1.77-1.62 (2H, m), 1.75 (3H, s), 1.59 (3H, s) and 1.55 (3H, s) ppm; δ_C (100 MHz, C_6D_6) 167.5, 165.6, 151.2, 139.3, 128.5, 127.9, 127.6, 126.0, 74.5, 73.1, 66.5, 64.9, 51.1, 36.0, 35.0, 28.6, 26.0 and 20.4 ppm; m/z (ESI+) 385 $(M + Na)^+$, 363 $(M + H)^+$. (Found 385.1615 $(M + Na)^+$. $C_{20}H_{26}NaO_6$ requires 385.1622).

Methyl 4-acetoxy-2-butyl-2-methyl-6-phenyl-5,6-dihydro-2H-pyran-3-carboxylate (26g).
Oil; υ_{max} (film) 2910, 2826, 1740, 1696, 1430, 1413, 1348, 1324, 1300, 1235, 1201, 1175, 1151, 1080, 1035 cm^{-1}; nOe: H-6 – H-8 4.9%; δ_H (400 MHz, C_6D_6) 7.30-7.27 (2H, m), 7.17-7.12 (2H, m), 7.09 (1H, m), 4.76 (1H, dd, *J* = 10.7, 3.4 Hz), 3.37 (3H, s), 2.62 (1H, dd, *J* = 17.4, 10.7 Hz), 2.23 (1H, dd, *J* = 17.4, 3.4 Hz), 2.19-2.03 (2H, m), 1.77 (3H, s), 1.65 (3H, s), 1.53-1.29 (2H, m), 1.27-1.17 (2H, m) and 0.80 (3H, t, *J* = 7.3 Hz) ppm; δ_C (100 MHz, C_6D_6) 167.7, 165.8, 150.4, 141.9, 128.6, 127.0, 126.2, 77.1, 69.5, 51.3, 37.0, 36.4, 25.5, 25.3, 23.3, 20.4 and 14.2 ppm; *m/z* (ESI+) 369 $(M + Na)^+$. (Found 369.1660 $(M + Na)^+$. $C_{20}H_{26}NaO_5$ requires 369.1672).

Methyl 4-acetoxy-2-butyl-2-methyl-6-propyl-5,6-dihydro-2H-pyran-3-carboxylate (26h).
Oil; υ_{max} (film) 2912, 2890, 1741, 1697, 1440, 1413, 1349, 1306, 1258, 1234, 1202, 1177, 1153, 1111, 1033 cm^{-1}; nOe: H-6 – H-8 4.8%; δ_H (400 MHz, C_6D_6) 3.61 (1H, m), 3.33 (3H, s), 2.26 (1H, dd, *J* = 17.4, 10.1 Hz), 2.12 (1H, m), 1.92 (1H, m), 1.89 (1H, dd, *J* = 17.4, 3.7 Hz), 1.77 (3H, s), 1.55 (3H, s), 1.53-1.12 (8H, m), 0.89 (3H, t, *J* = 7.3 Hz) and 0.80 (3H, t, *J* = 7.3 Hz) ppm; δ_C (100 MHz, C_6D_6) 167.7, 165.9, 150.5, 127.1, 76.2, 66.9, 51.2, 38.3, 36.7, 34.7, 25.5, 25.1, 23.4, 20.4, 19.0, 14.4 and 14.2 ppm; *m/z* (ESI+) 335 $(M + Na)^+$. (Found 335.1818 $(M + Na)^+$. $C_{17}H_{28}NaO_5$ requires 335.1829).

Methyl 4-acetoxy-2-methyl-6-phenyl-2-vinyl-5,6-dihydro-2H-pyran-3-carboxylate (26i).
Oil; υ_{max} (film) 2986, 2937, 2906, 1740, 1697, 1413, 1348, 1306, 1240, 1187, 1033 cm^{-1}; nOe: H-6 – H-8 0.8%; δ_H (400 MHz, C_6D_6) 7.26-7.23 (2H, m), 7.16-7.11 (2H, m), 7.07 (1H, m), 6.10 (1H, dd, *J* = 17.4, 10.6 Hz), 5.48 (1H, dd, *J* = 17.4, 1.3 Hz), 5.17 (1H, dd, *J* = 10.6, 1.3 Hz), 4.84 (1H, dd, *J* = 10.8, 3.5 Hz), 3.31 (3H, s), 2.58 (1H, dd, *J* = 17.4, 10.8 Hz), 2.19 (1H, dd, *J* = 17.4, 3.5 Hz), 1.77 (3H, s) and 1.76 (3H, s) ppm; δ_C (100 MHz, C_6D_6) 167.7, 165.2, 153.0, 141.6, 140.7, 128.6, 127.8, 126.3, 123.2, 116.4, 77.8, 69.7, 51.2, 36.7, 28.0 and 20.4 ppm; *m/z* (ESI+) 339 $(M + Na)^+$. (Found 339.1190 $(M + Na)^+$. $C_{18}H_{20}NaO_5$ requires 339.1203).

Methyl 4-acetoxy-2-methyl-6-propyl-2-vinyl-5,6-dihydro-2H-pyran-3-carboxylate (26j).
Oil; υ_{max} (film) 2913, 2889, 2830, 1740, 1697, 1413, 1346, 1309, 1213, 1189, 1151, 1041 cm^{-1}; δ_H (400 MHz, C_6D_6) 6.06 (1H, dd, *J* = 17.4, 10.6 Hz), 5.45 (1H, dd, *J* = 17.4, 1.5 Hz), 5.18 (1H, dd, *J* = 10.6, 1.5 Hz), 3.73 (1H, m), 3.30 (3H, s), 2.21 (1H, dd, *J* = 17.2, 10.6 Hz), 1.86 (1H, dd, *J* = 17.2, 3.3 Hz), 1.79 (3H, s), 1.69 (3H, s), 1.52-1.33 (2H, m), 1.27-1.12 (2H, m) and 0.80 (3H, t, *J* = 7.2 Hz) ppm; δ_C (100 MHz, C_6D_6) 167.7, 165.3, 153.2, 141.0, 123.2, 116.2, 77.0, 67.2, 51.1, 37.8, 35.0, 28.0, 20.5, 18.7 and 14.2 ppm; *m/z* (ESI+) 305 $(M + Na)^+$. (Found 305.1349 $(M + Na)^+$. $C_{15}H_{22}NaO_5$ requires 305.1359).

Methyl 4-acetoxy-6-isopropyl-2-methyl-2-vinyl-5,6-dihydro-2H-pyran-3-carboxylate (26k).
Oil; υ_{max} (film) 2914, 2832, 1740, 1697, 1413, 1348, 1318, 1293, 1276, 1186, 1040 cm^{-1}; nOe: H-6 – H-8 0.5%; δ_H (400 MHz, C_6D_6) 6.02 (1H, dd, *J* = 17.4, 10.6 Hz), 5.46 (1H, dd, *J* = 17.4, 1.5 Hz), 5.18 (1H, dd, *J* = 10.6, 1.5 Hz), 3.48 (1H, m), 3.30 (3H, s), 2.29 (1H, dd, *J* = 17.2, 10.8 Hz), 1.94 (1H, dd, *J* = 17.2, 3.5 Hz), 1.80 (3H, s), 1.68 (3H, s), 1.59 (1H, m), 0.92 (3H, d, *J* = 6.8 Hz) and 0.71 (3H, d, *J* = 6.8 Hz) ppm; δ_C (100 MHz, C_6D_6) 167.7, 165.3, 153.4, 141.0, 123.1, 116.5, 76.9, 72.2, 51.1, 32.9, 32.4, 27.8, 20.5, 18.5 and 17.9 ppm; *m/z* (ESI+) 305 $(M + Na)^+$. (Found 305.1351 $(M + Na)^+$. $C_{15}H_{22}NaO_5$ requires 305.1359).

Methyl 4-acetoxy-6-(furan-2-yl)-2-methyl-2-vinyl-5,6-dihydro-2H-pyran-3-carboxylate (26l).
Oil; υ_{max} (film) 2910, 2883, 2812, 1740, 1697, 1627, 1412, 1346, 1304, 1246, 1214, 1183, 1146, 1030 cm^{-1}; δ_H (400 MHz, C_6D_6) 7.03 (1H, dd, *J* = 1.8, 0.9 Hz), 6.12 (1H, dd, *J* = 17.4, 10.7 Hz), 6.07 (1H, ddd, *J* = 3.4, 0.9, 0.9 Hz), 6.01 (1H, dd, *J* = 3.4, 1.8 Hz), 5.59 (1H, dd, *J* = 17.4, 1.2 Hz), 5.21 (1H, dd, *J* = 10.7, 1.2 Hz), 4.93 (1H, dd, *J* = 11.3, 3.4 Hz), 3.27 (3H, s), 2.95 (1H, dd, *J* = 17.4, 11.3 Hz), 2.12 (1H, dd, *J* = 17.4, 3.4 Hz), 1.76 (3H, s) and 1.68 (3H, s) ppm; δ_C (100 MHz, C_6D_6) 167.6, 165.0, 153.7, 152.5, 142.5, 140.4, 123.2, 116.7, 110.4, 107.6, 77.9, 63.8, 51.2, 32.9, 27.8 and 20.4 ppm; *m/z* (ESI+) 329 $(M + Na)^+$. (Found 329.0986 $(M + Na)^+$. $C_{16}H_{18}NaO_6$ requires 329.0996).

Methyl 4-acetoxy-6-(benzyloxymethyl)-2-methyl-2-vinyl-5,6-dihydro-2H-pyran-3-carboxylate (26m).
Oil; υ_{max} (film) 2984, 2935, 2886, 2818, 1740, 1695, 1629, 1430, 1413, 1345, 1312, 1293, 1238, 1215, 1187, 1149, 1080, 1039, 1008 cm^{-1}; δ_H (400 MHz, C_6D_6) 7.25-7.23 (2H, m), 7.17-7.12 (2H, m), 7.08 (1H, m), 6.09

(1H, dd, *J* = 17.4, 10.7 Hz), 5.55 (1H, dd, *J* = 17.4, 1.2 Hz), 5.18 (1H, dd, *J* = 10.7, 1.2 Hz), 4.32 (1H, d, *J* = 13.3 Hz), 4.29 (1H, d, *J* = 13.3 Hz), 4.11 (1H, m), 3.42 (1H, dd, *J* = 10.1, 5.8 Hz), 3.28 (3H, s), 3.24 (1H, dd, *J* = 10.1, 4.6 Hz), 2.44 (1H, dd, *J* = 17.4, 10.7 Hz), 1.98 (1H, dd, *J* = 17.4, 3.7 Hz), 1.75 (3H, s) and 1.68 (3H, s) ppm; δ_C (100 MHz, C_6D_6) 167.6, 165.1, 152.9, 140.5, 138.9, 128.5, 127.7, 123.2, 116.5, 77.3, 73.4, 72.6, 67.3, 51.2, 31.8, 27.9 and 20.4 ppm; *m/z* (ESI+) 383 $(M + Na)^+$ 361 $(M + H)^+$. (Found 361.1644 $(M + H)^+$. $C_{20}H_{25}O_6$ requires 361.1646).

Methyl 4-acetoxy-6-(2-(benzyloxy)ethyl)-2-methyl-2-vinyl-5,6-dihydro-2H-pyran-3-carboxylate (26n).
Oil; υ_{max} (film) 2985, 2936, 2887, 2817, 1740, 1696, 1430, 1413, 1347, 1292, 1215, 1188, 1150, 1084, 1039 cm^{-1}; δ_H (400 MHz, C_6D_6) 7.28-7.25 (2H, m), 7.19-7.15 (2H, m), 7.08 (1H, m), 6.05 (1H, dd, *J* = 17.4, 10.7 Hz), 5.45 (1H, dd, *J* = 17.4, 1.2 Hz), 5.18 (1H, dd, *J* = 10.7, 1.2 Hz), 4.26 (2H, s), 4.03 (1H, m), 3.46 (1H, m), 3.36 (1H, m), 3.29 (3H, s), 2.29 (1H, dd, *J* = 17.4, 10.7 Hz), 1.91 (1H, dd, *J* = 17.4, 3.7 Hz), 1.79 (1H, m), 1.77 (3H, s), 1.66 (1H, m) and 1.64 (3H, s) ppm; δ_C (100 MHz, C_6D_6) 167.7, 165.2, 153.0, 140.8, 139.2, 128.5, 127.6, 123.1, 116.3, 77.0, 73.0, 66.4, 65.0, 51.1, 36.0, 35.0, 27.9 and 20.5 ppm; *m/z* (ESI+) 397 $(M + Na)^+$, 375 $(M + H)^+$. (Found 375.1804 $(M + H)^+$. $C_{21}H_{27}O_6$ requires 375.1802).

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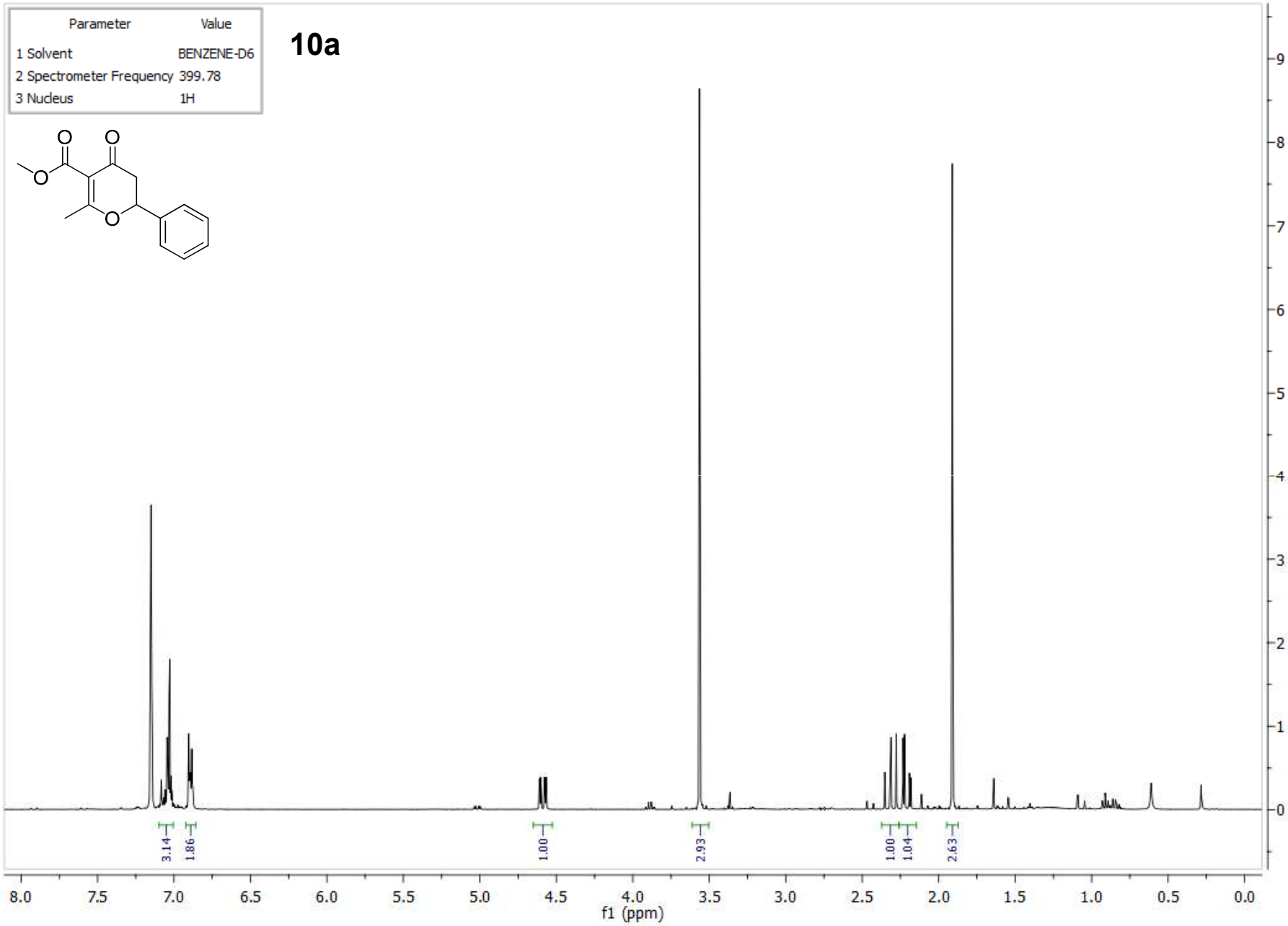
3. NMR Data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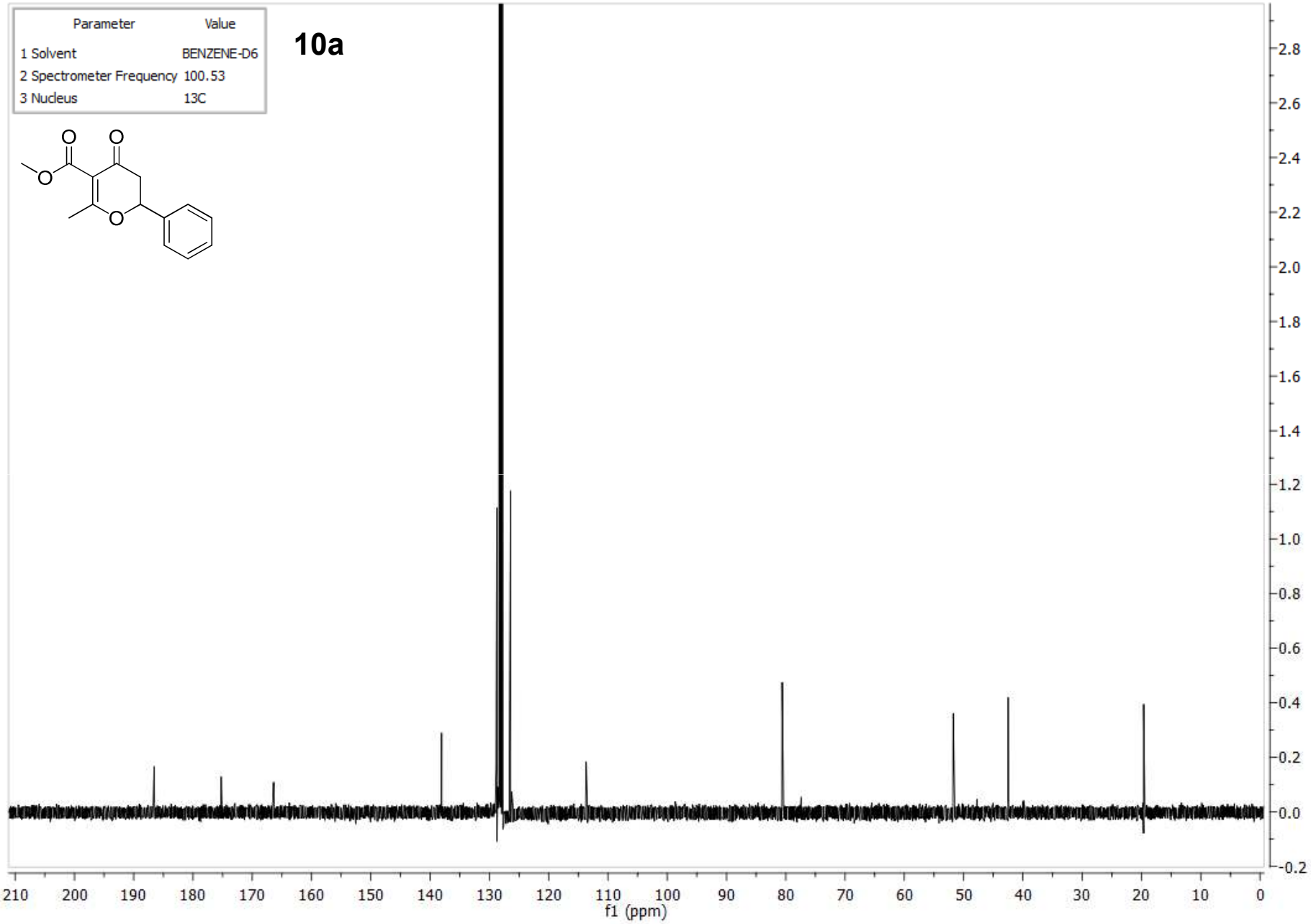
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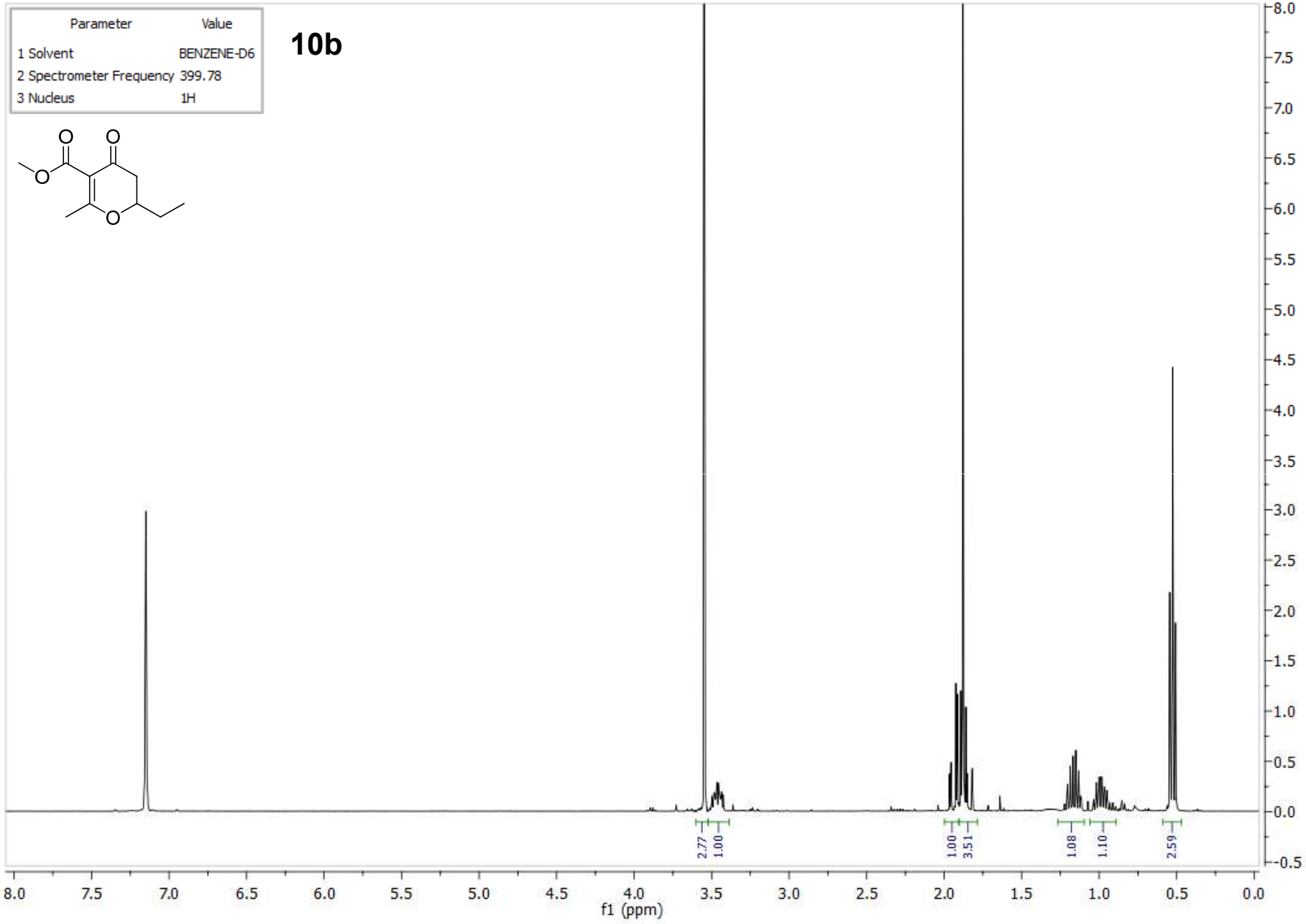
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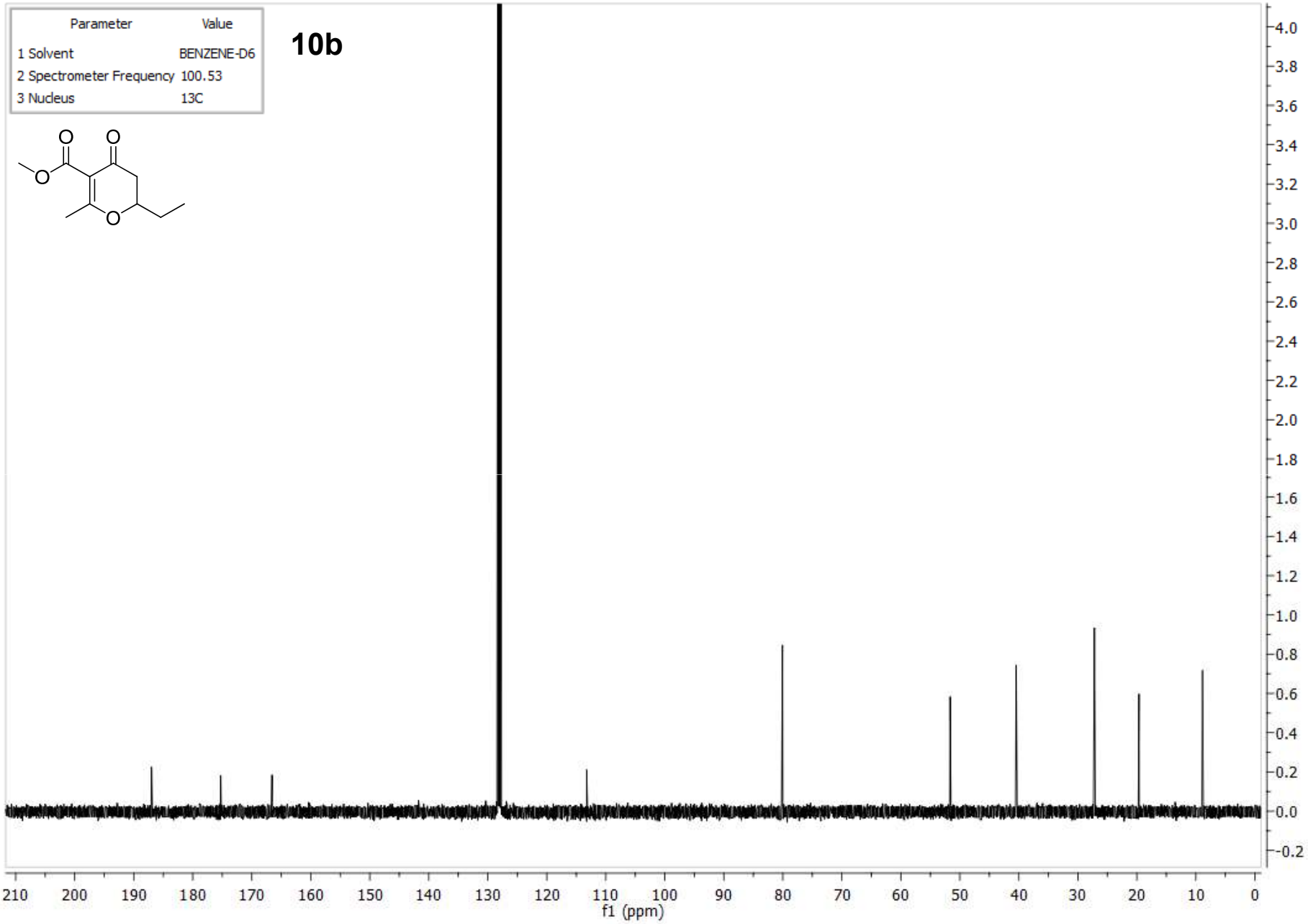
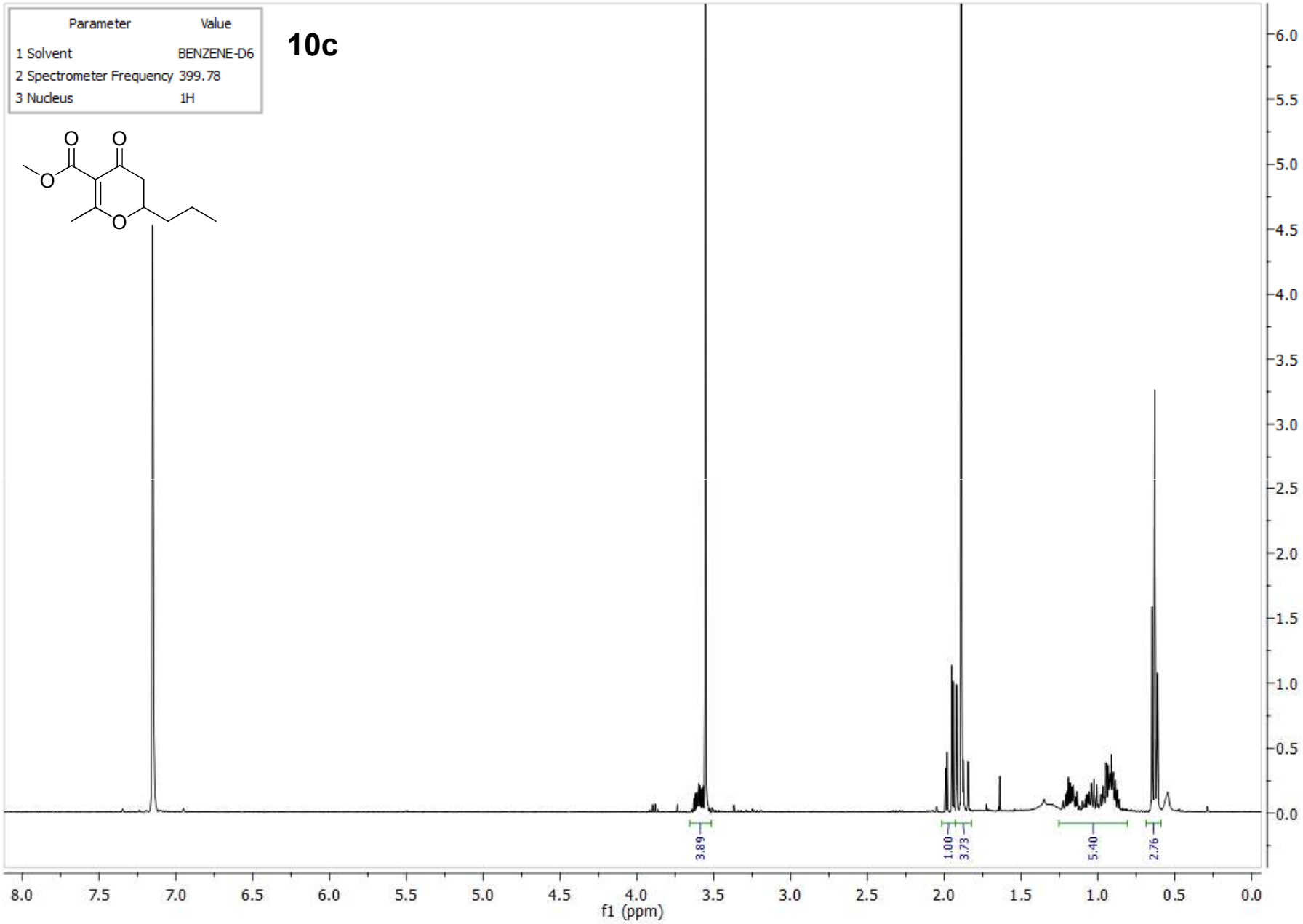
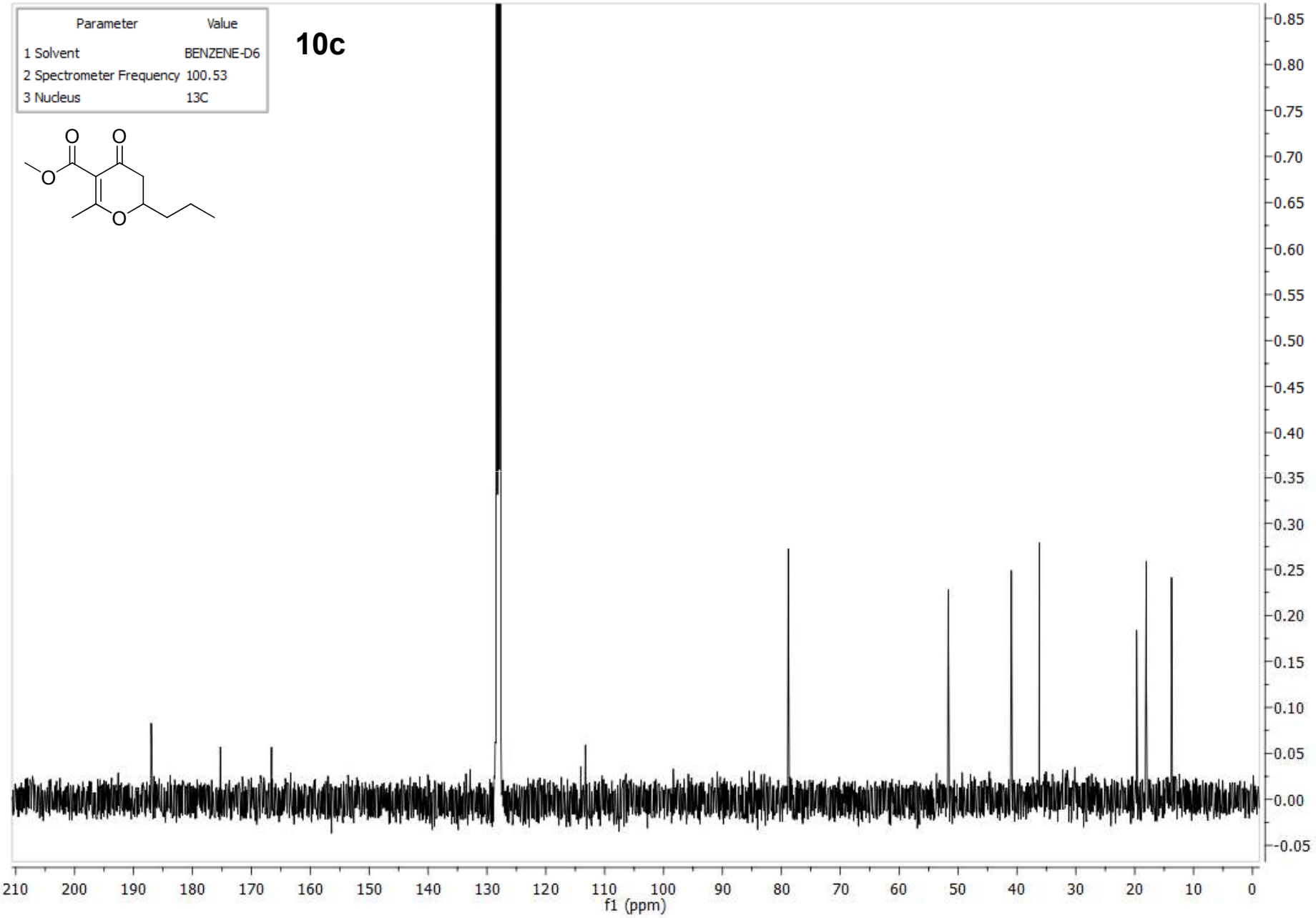
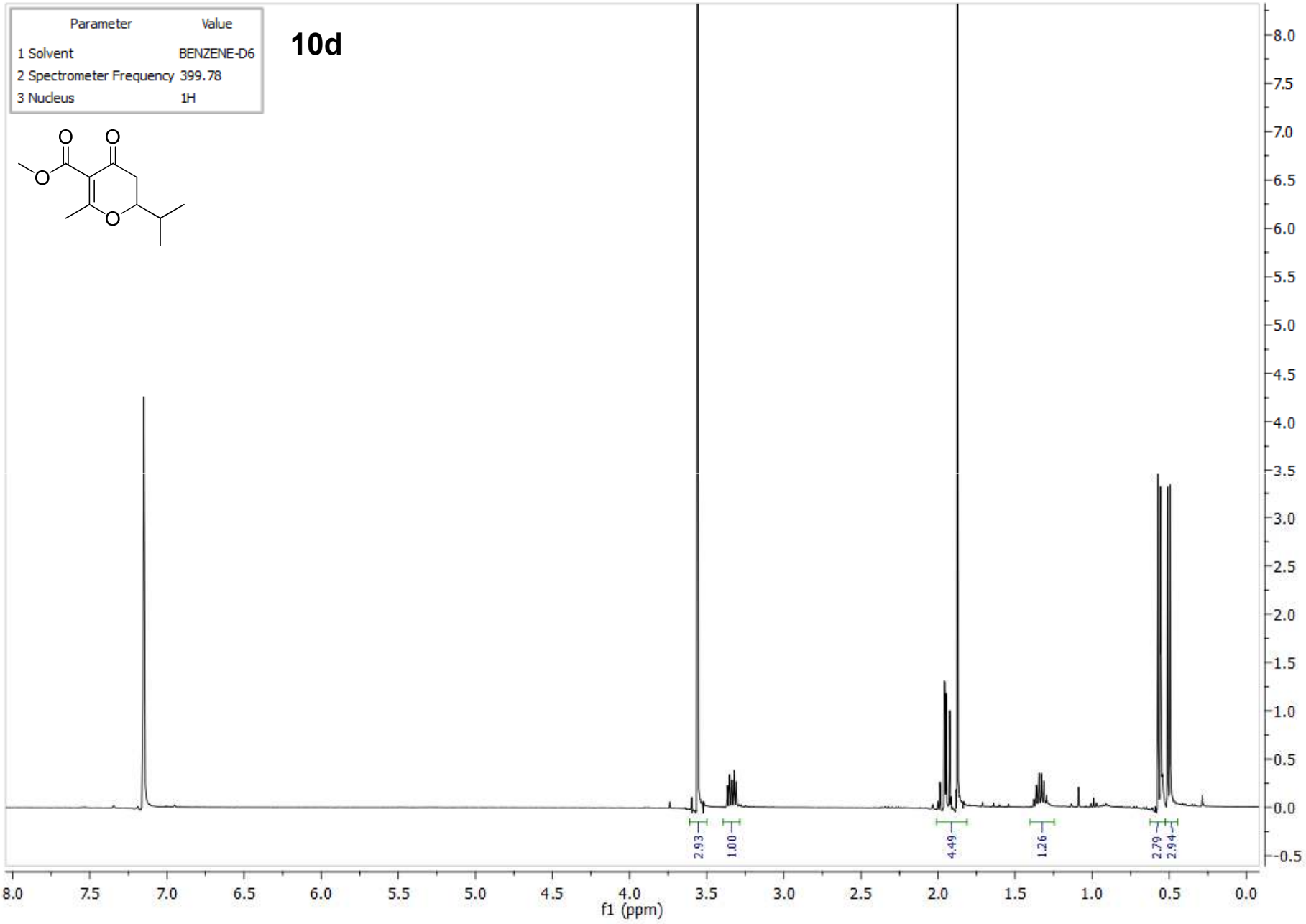
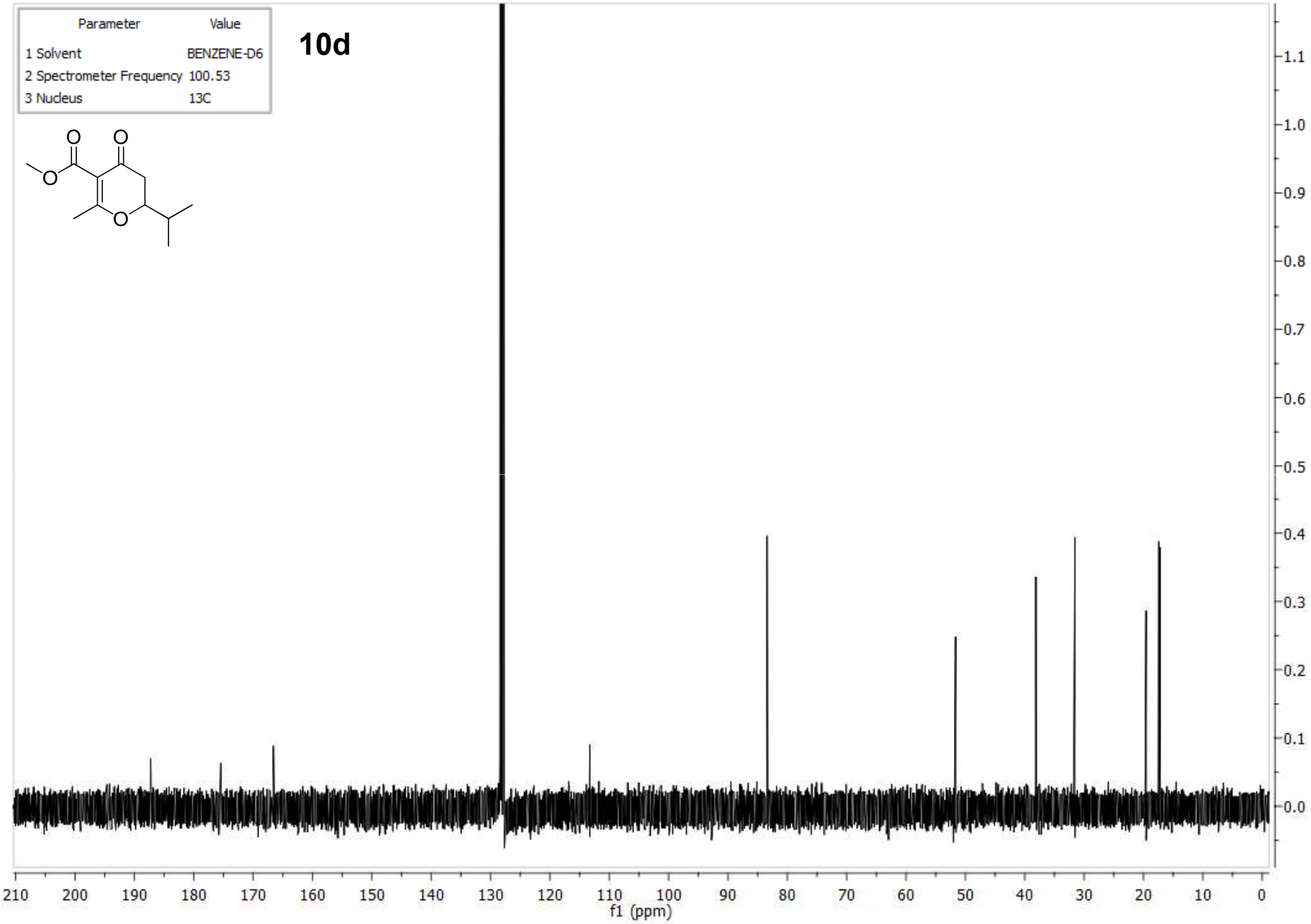
2.59
f1 (ppm)

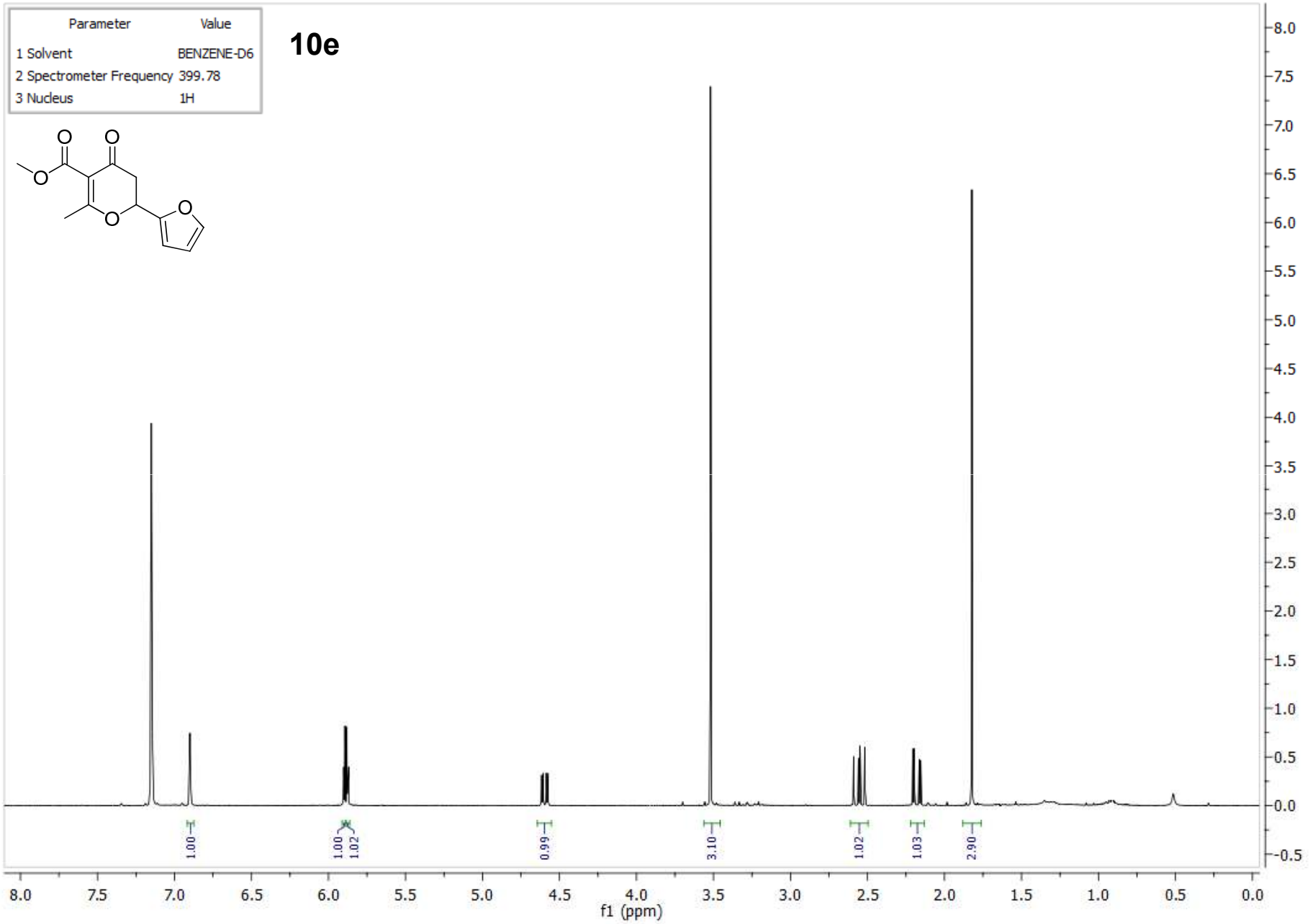

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
10b
f1 (ppm)

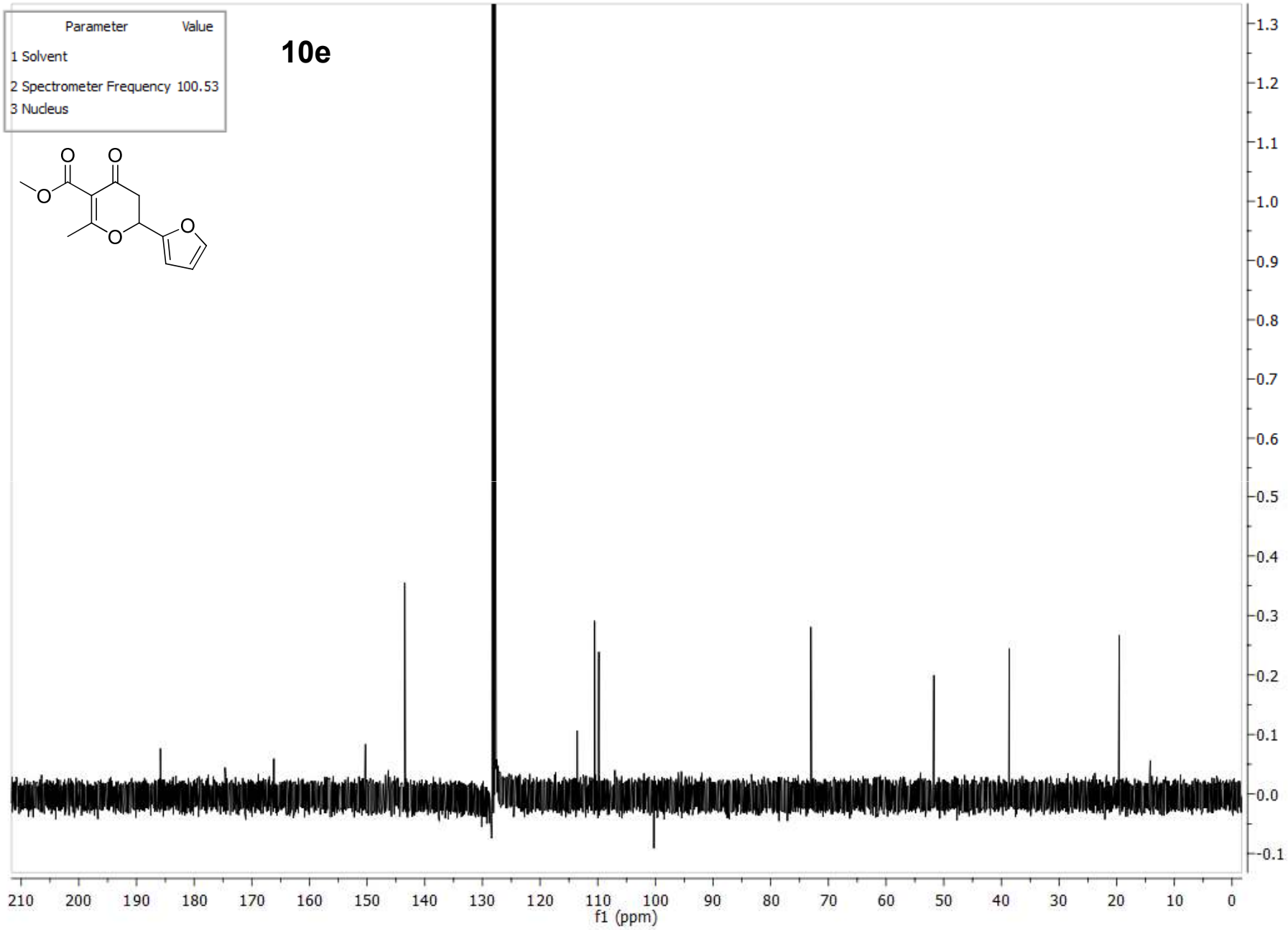

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
10c
O
O
O
O
3.89
1.00
3.73
5.40
2.76
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

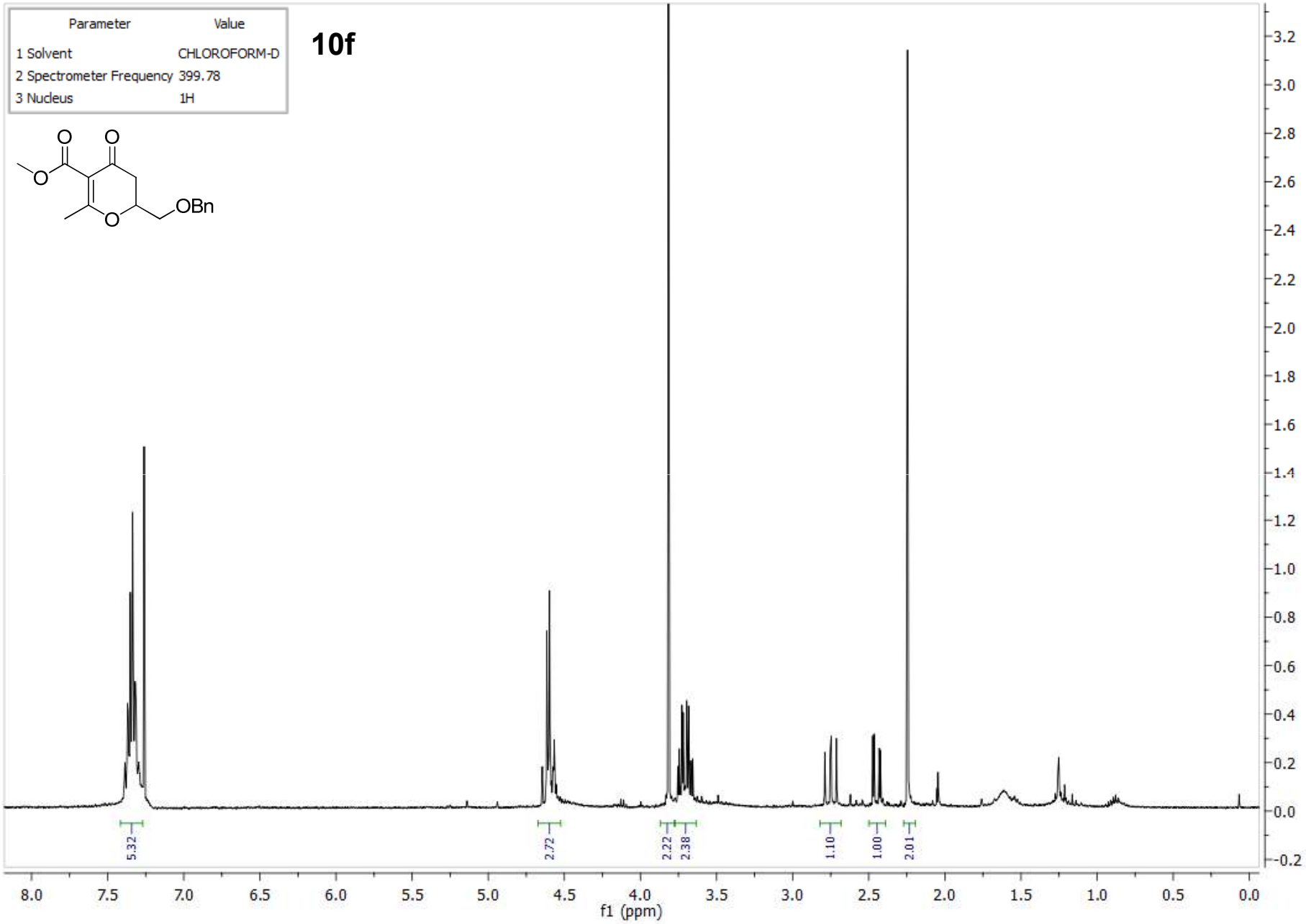

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
10c
O
O
O
O
0.85
0.80
0.75
0.70
0.65
0.60
0.55
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
-0.05
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

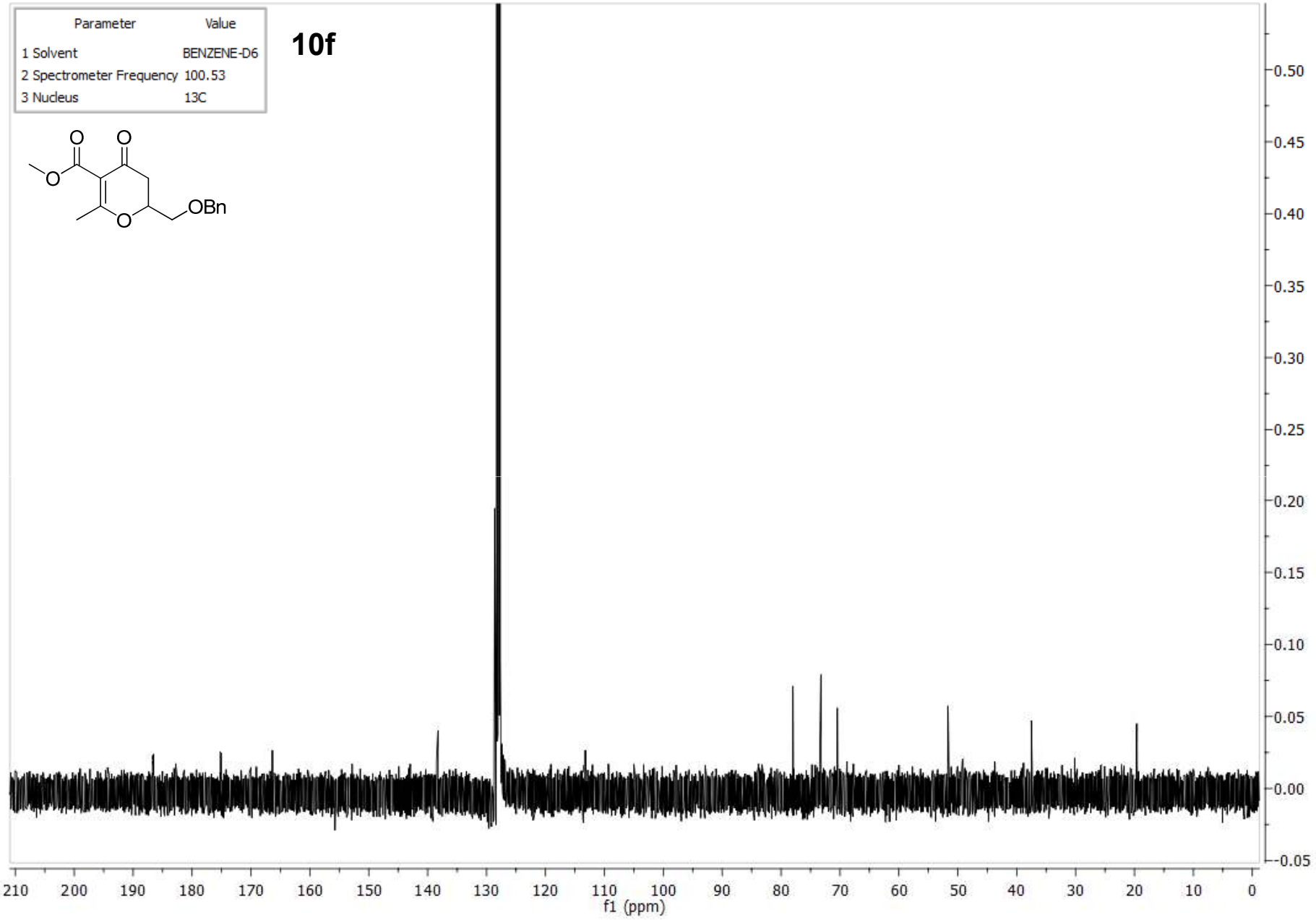

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
10d
2.93
1.00
4.49
1.26
2.79
2.94
f1 (ppm)

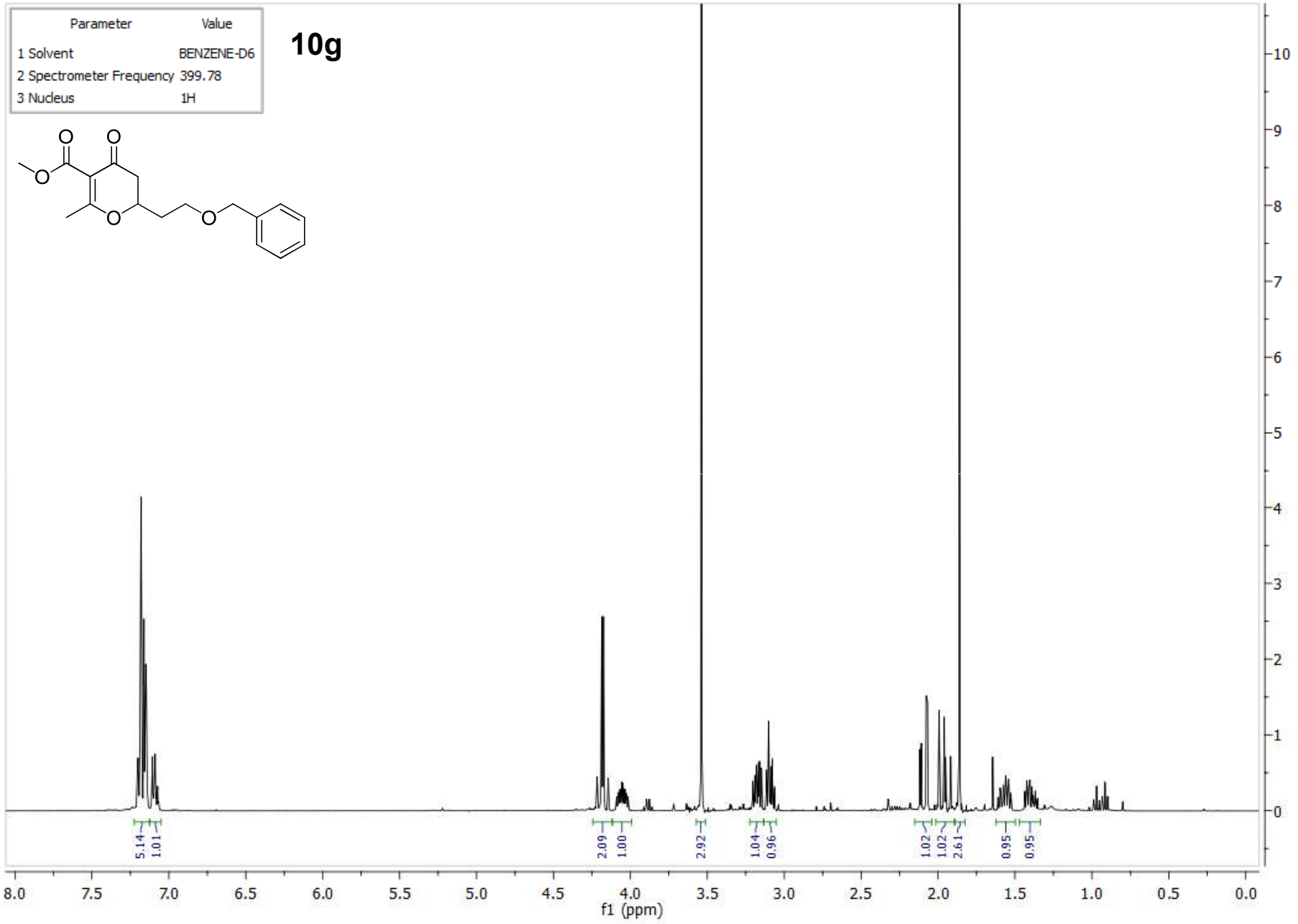

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
10d
O
O
O
O
1.1
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

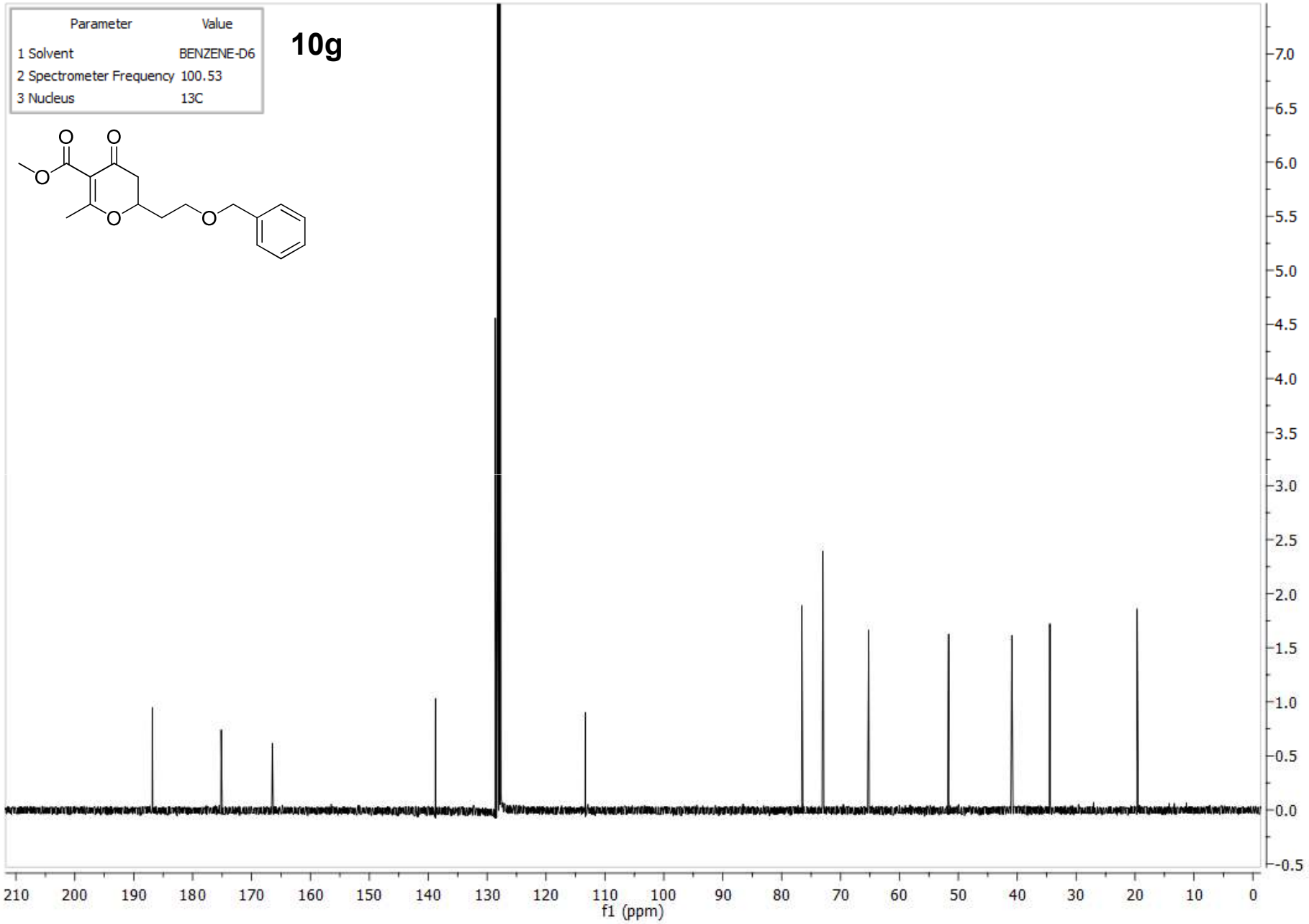

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
10e
O
O
O
O
O
1.00
1.00
1.02
0.99
3.10
1.02
1.03
2.90
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

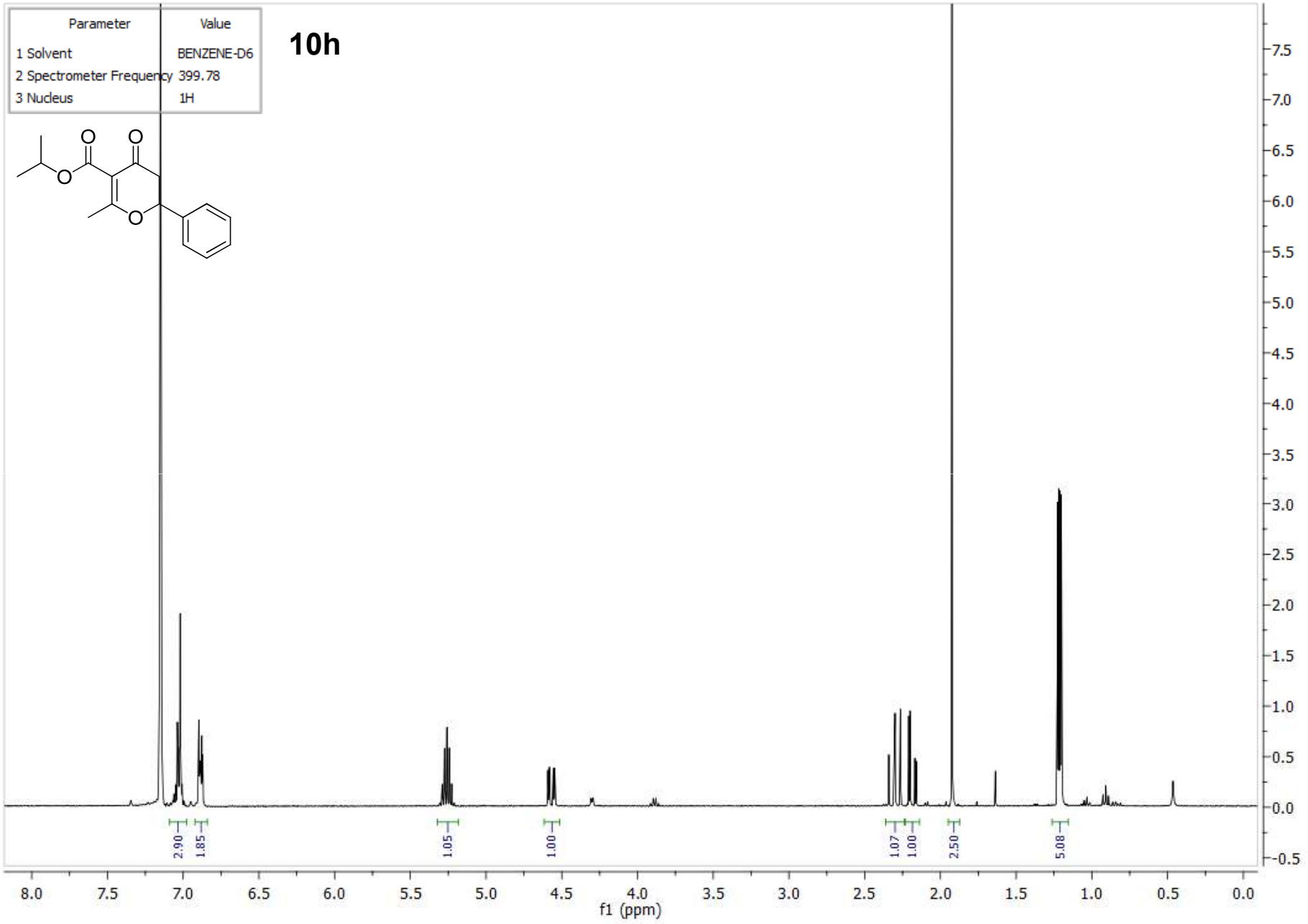

Parameter Value
1 Solvent
2 Spectrometer Frequency 100.53
3 Nucleus
10e
f1 (ppm)

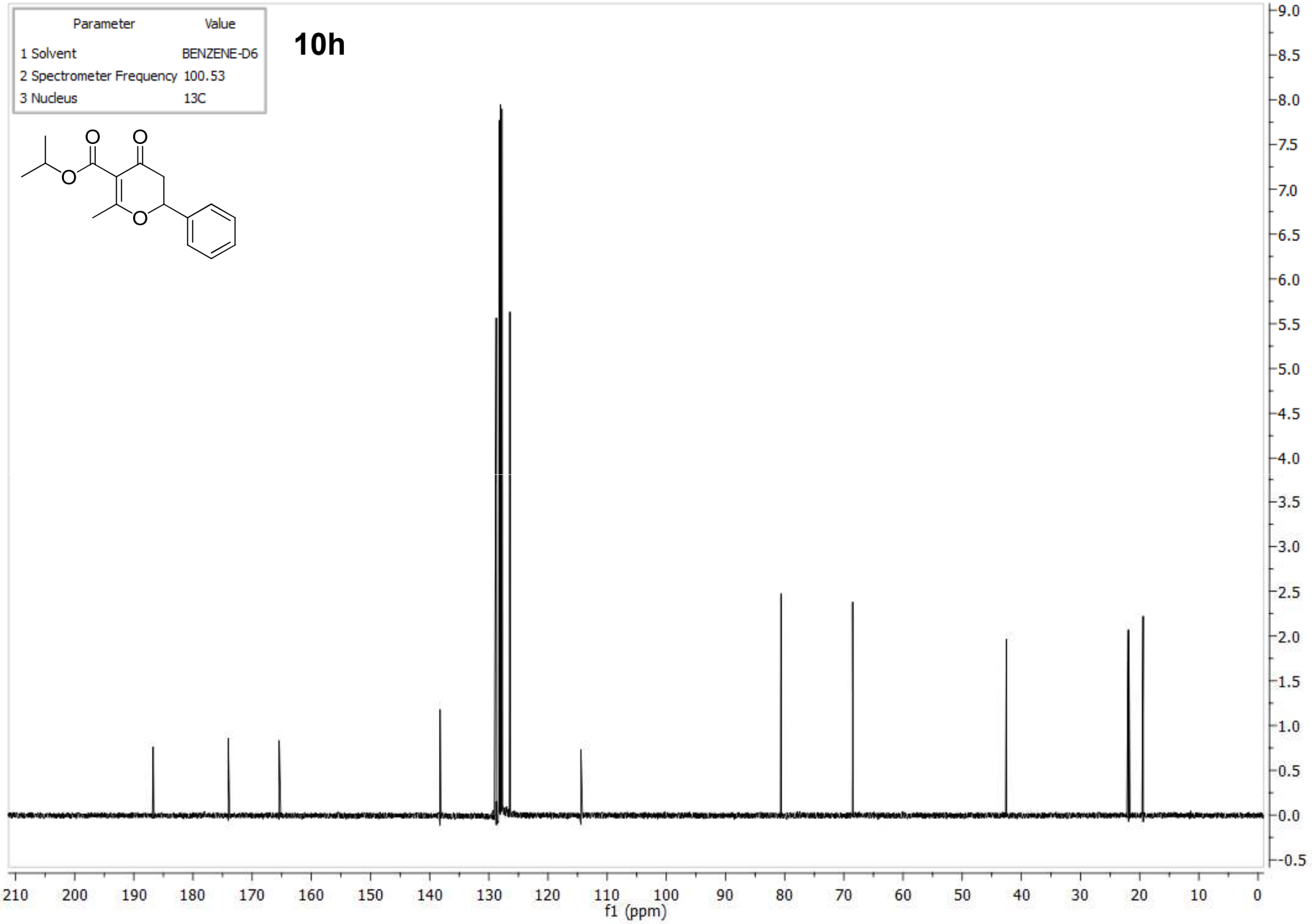

Parameter Value
1 Solvent CHLOROFORM-D
2 Spectrometer Frequency 399.78
3 Nucleus 1H
10f
OBn
5.32
2.72
2.22
2.38
1.10
1.00
2.01
f1 (ppm)

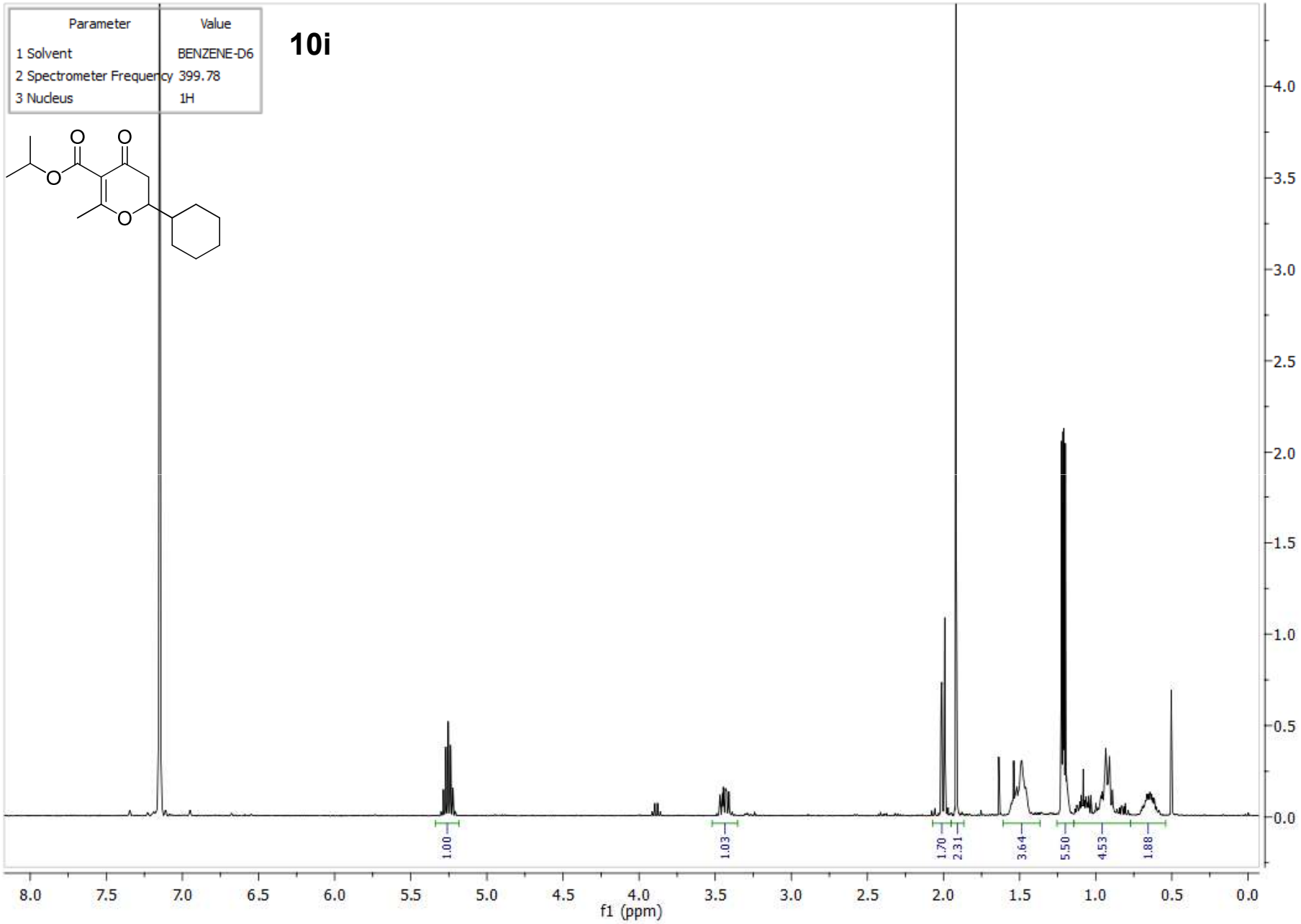

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
10f
O
O
O
O
OBn
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
-0.05
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

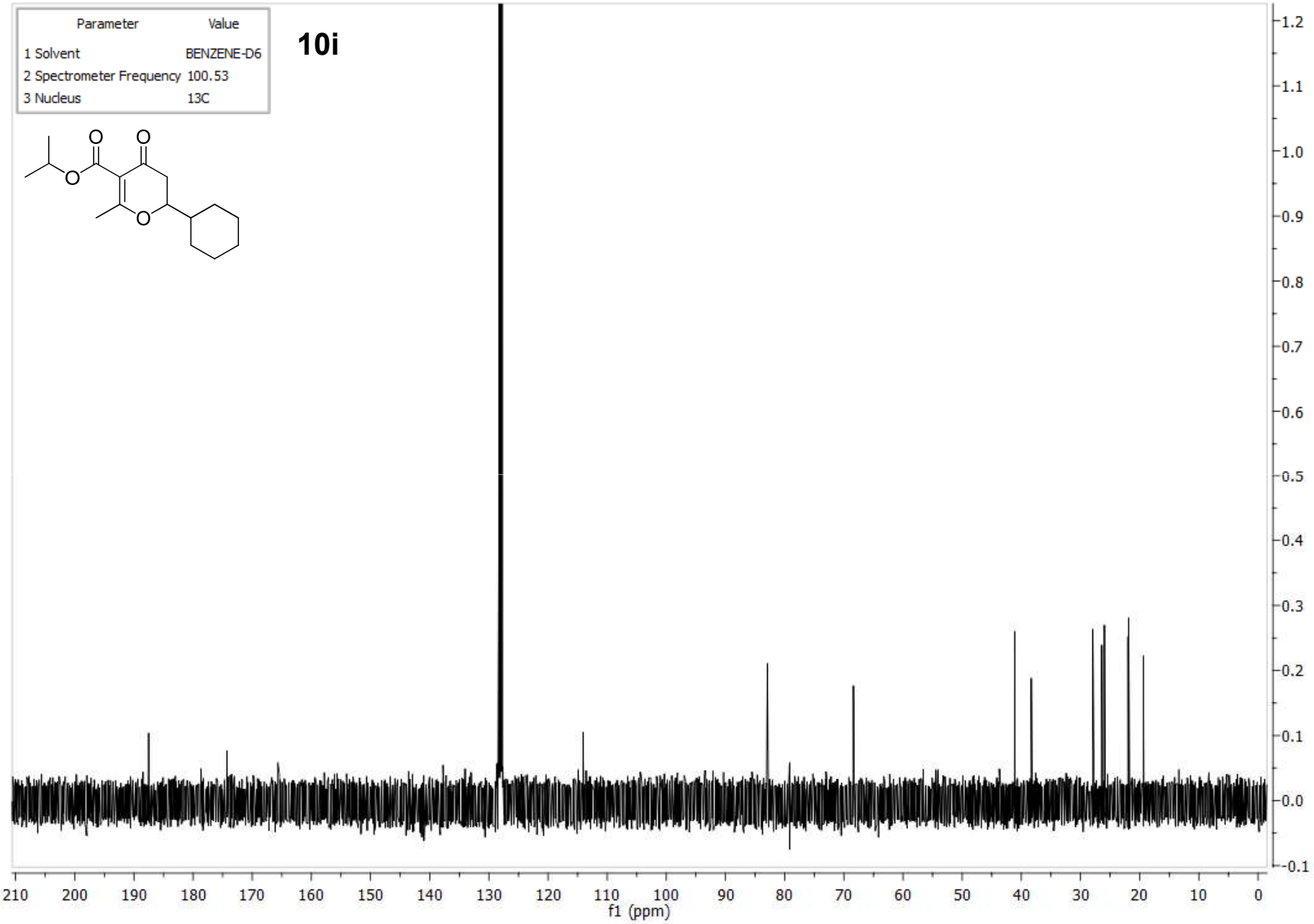

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
10g
O
O
O
O
O
5.14
1.01
2.09
1.00
2.92
1.04
0.96
1.02
1.02
2.61
0.95
0.95
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
10
9
8
7
6
5
4
3
2
1
0

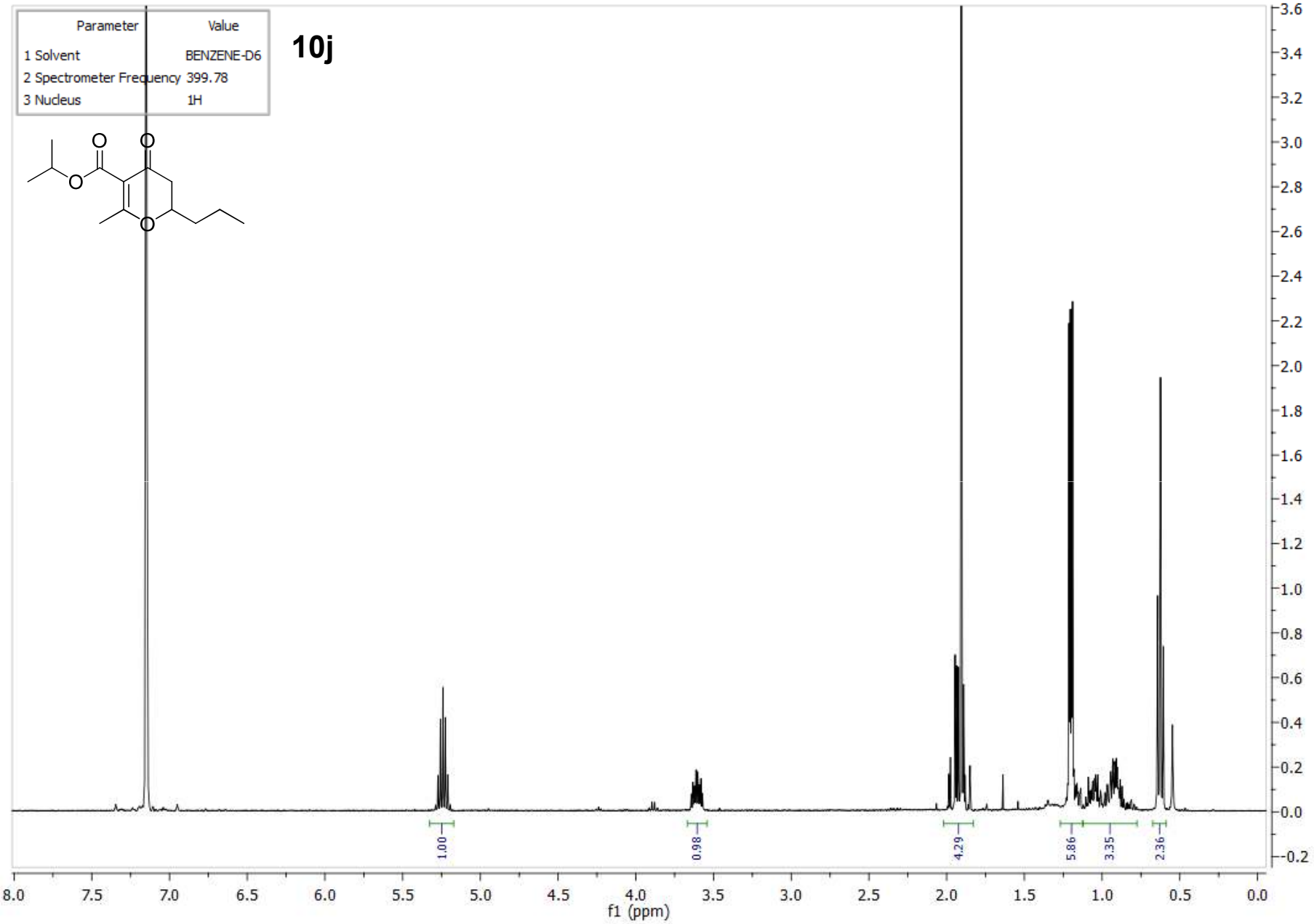

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
10g
O
O
O
O
O
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

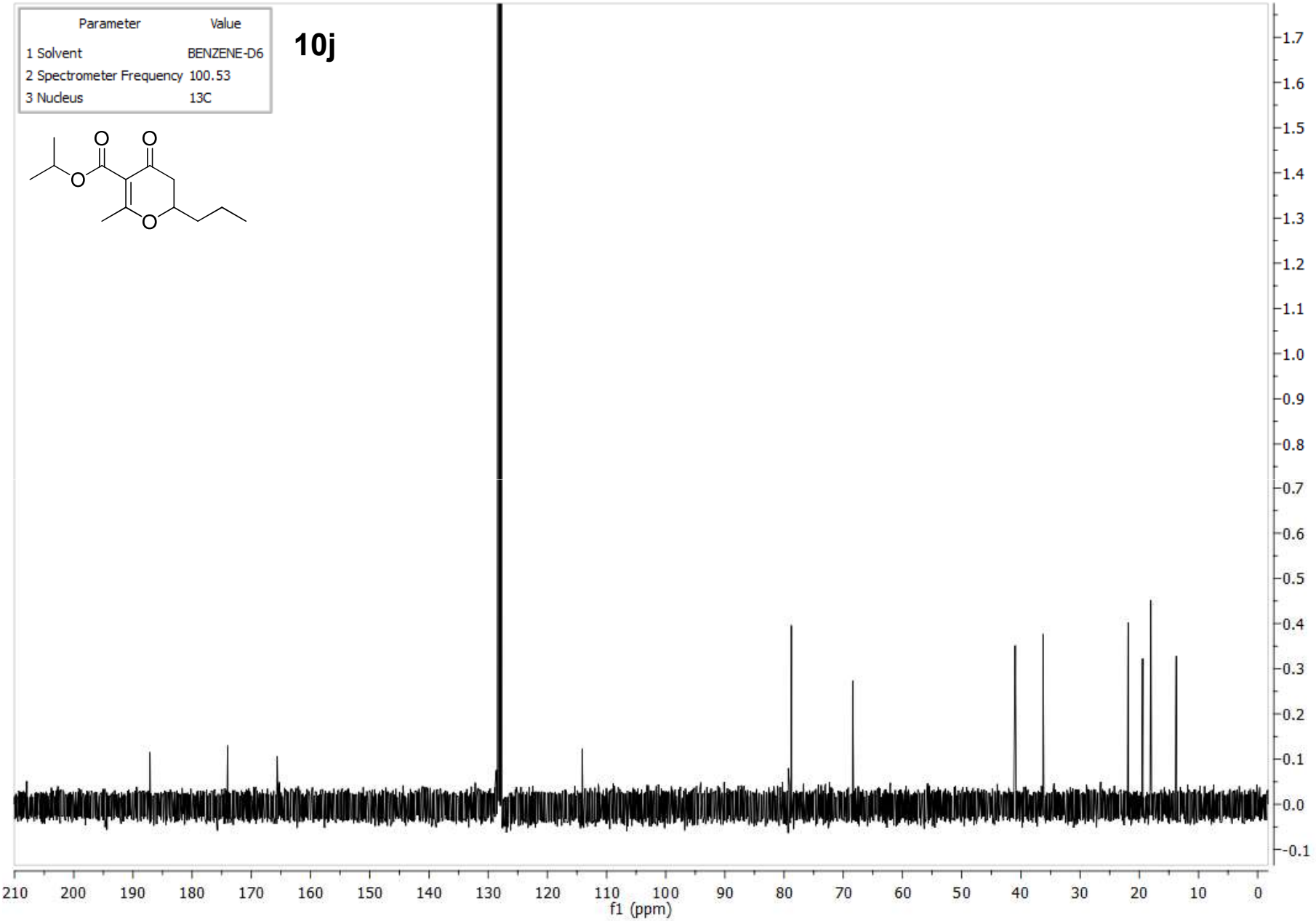

Parameter
Value
1 Solvent
BENZENE-D6
2 Spectrometer Frequency
399.78
3 Nucleus
1H
10h
O
O
O
O
2.90
1.85
1.05
1.00
1.07
1.00
2.50
5.08
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5

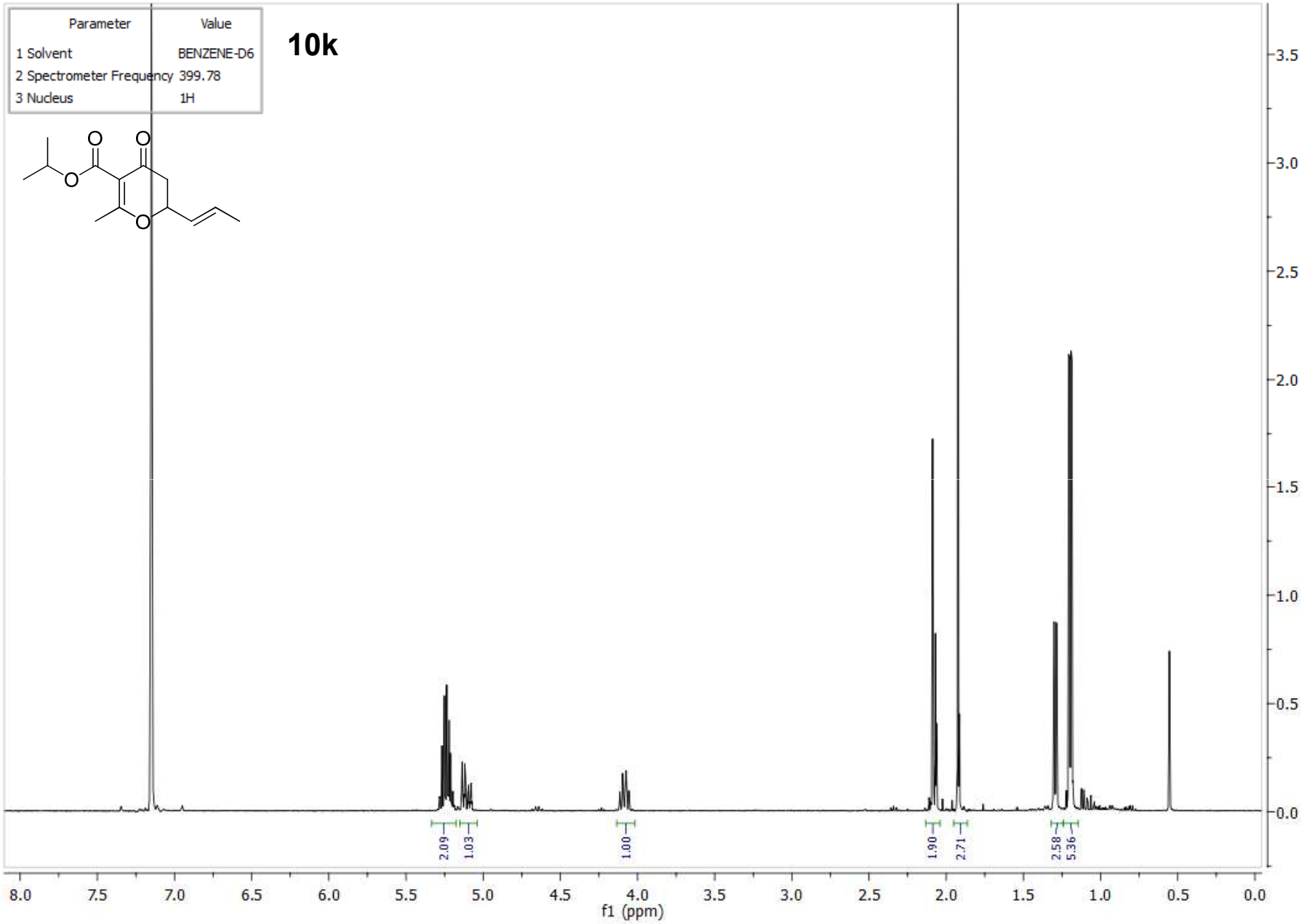

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
10h
O
O
O
O
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

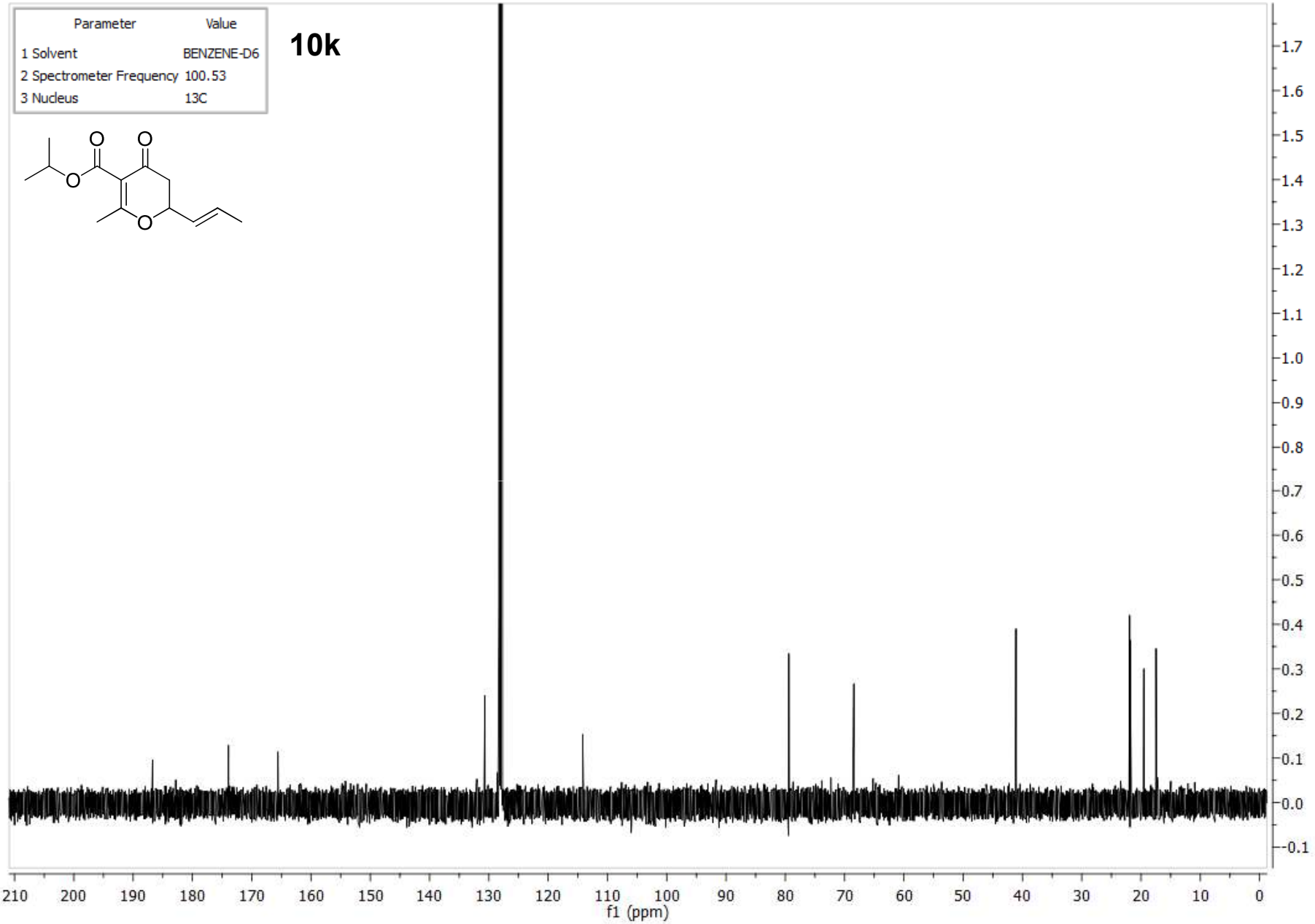

Parameter
Value
1 Solvent
BENZENE-D6
2 Spectrometer Frequency
399.78
3 Nucleus
1H
10i
1.00
1.03
1.70
2.31
3.64
5.50
4.53
1.88
f1 (ppm)

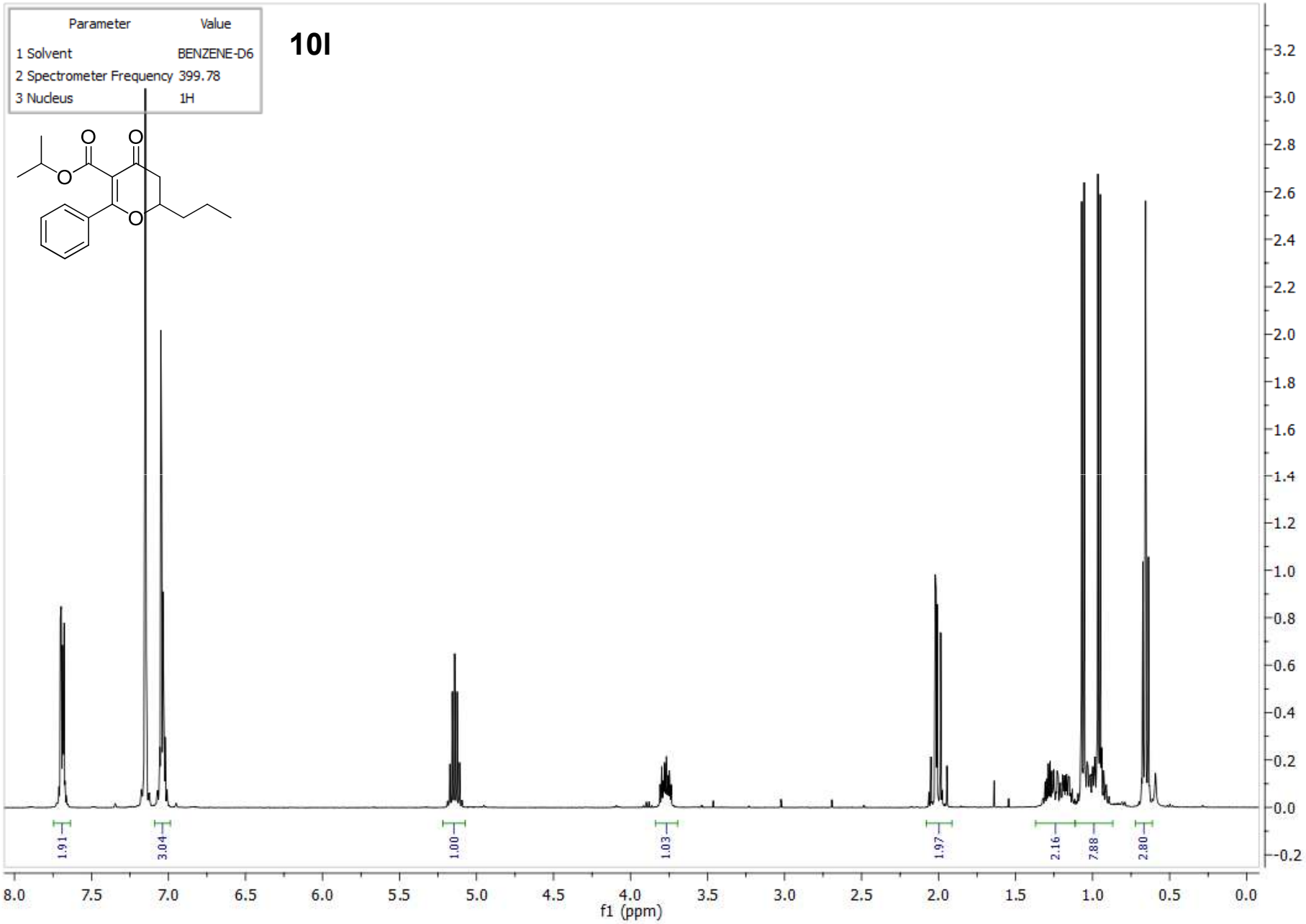

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
10i
O
O
O
O
1.2
1.1
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
-0.1
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

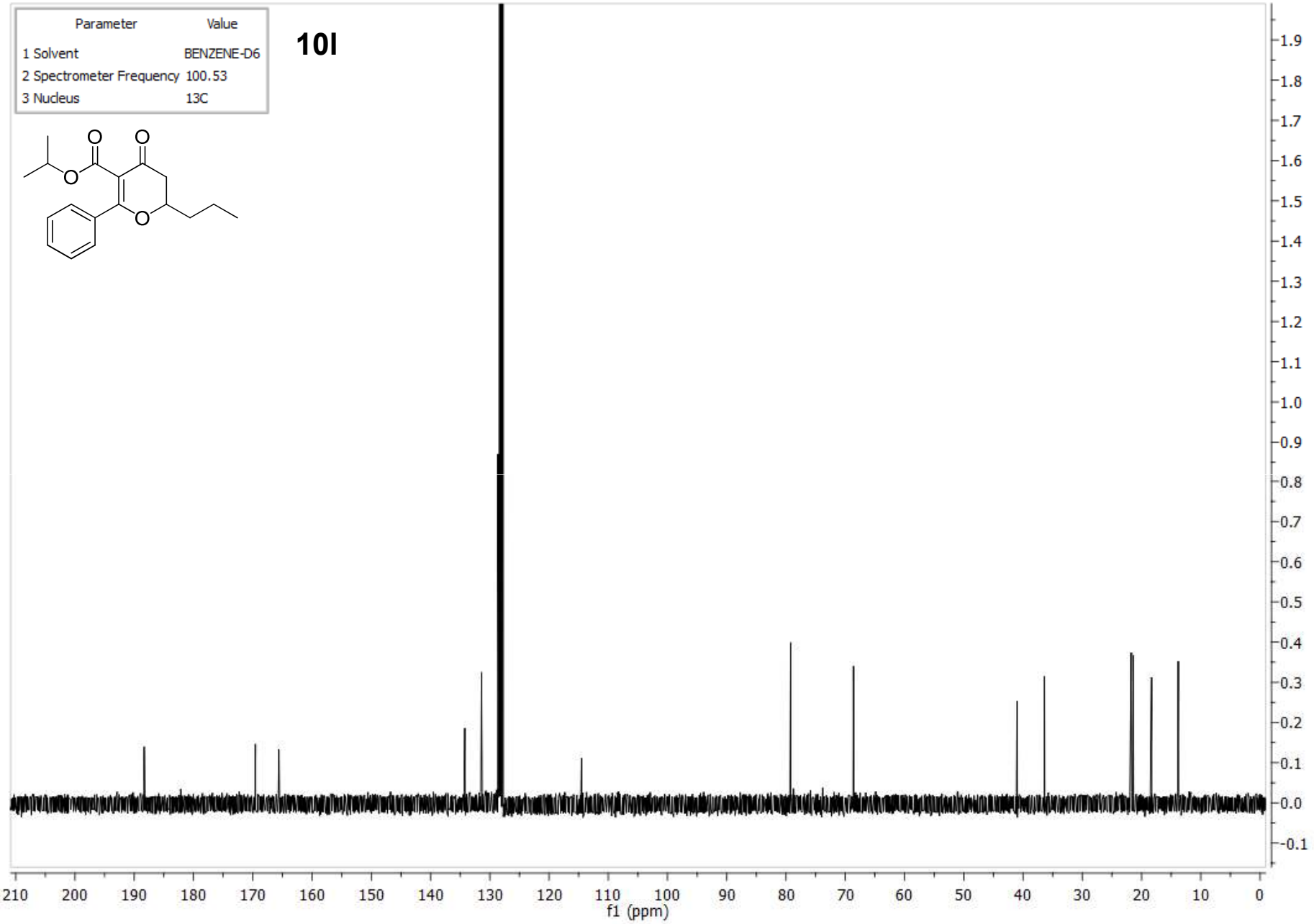

Parameter
Value
1 Solvent
BENZENE-D6
2 Spectrometer Frequency
399.78
3 Nucleus
1H
10j
1.00
0.98
4.29
5.86
3.35
2.36
f1 (ppm)

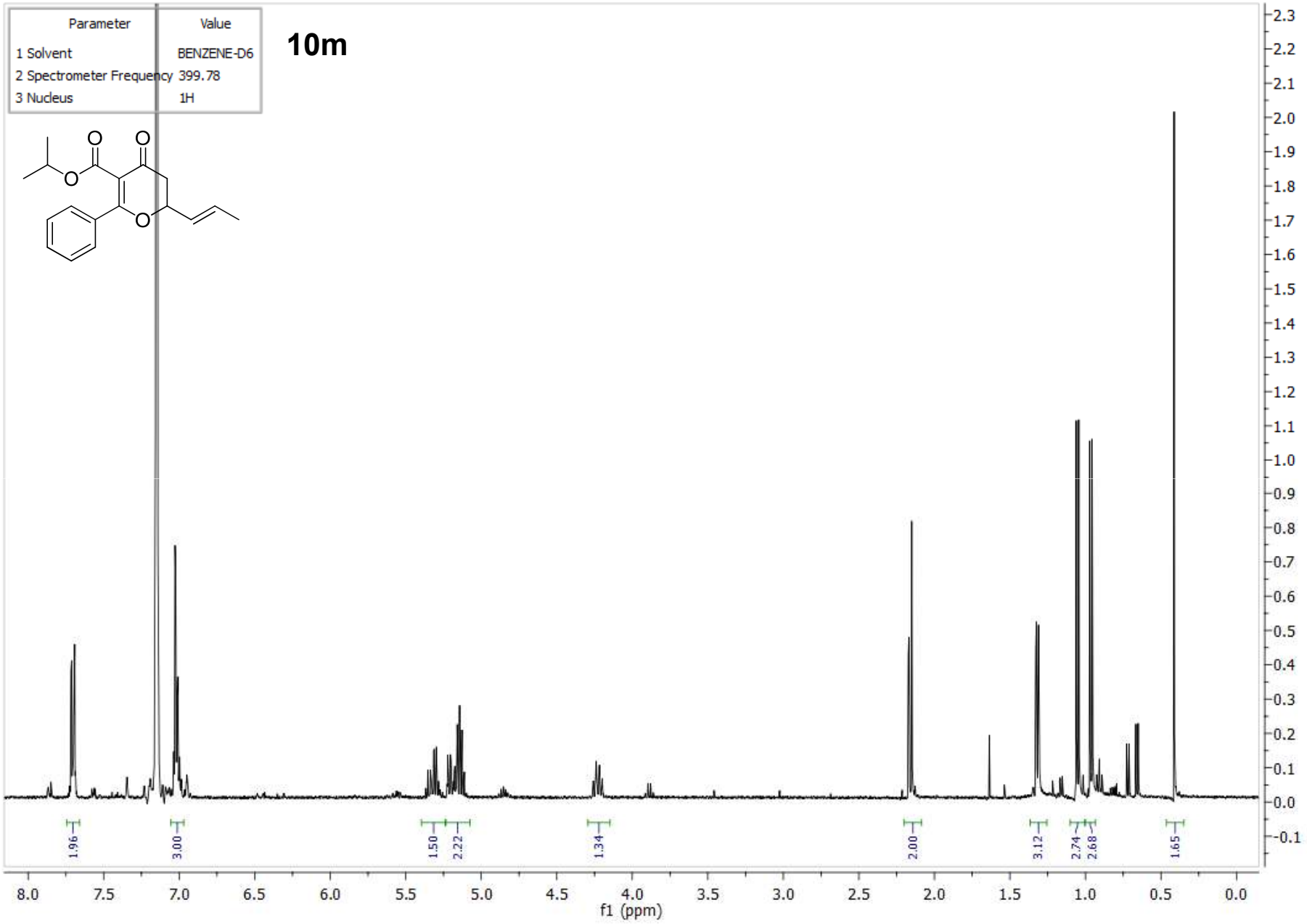

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
10j
f1 (ppm)

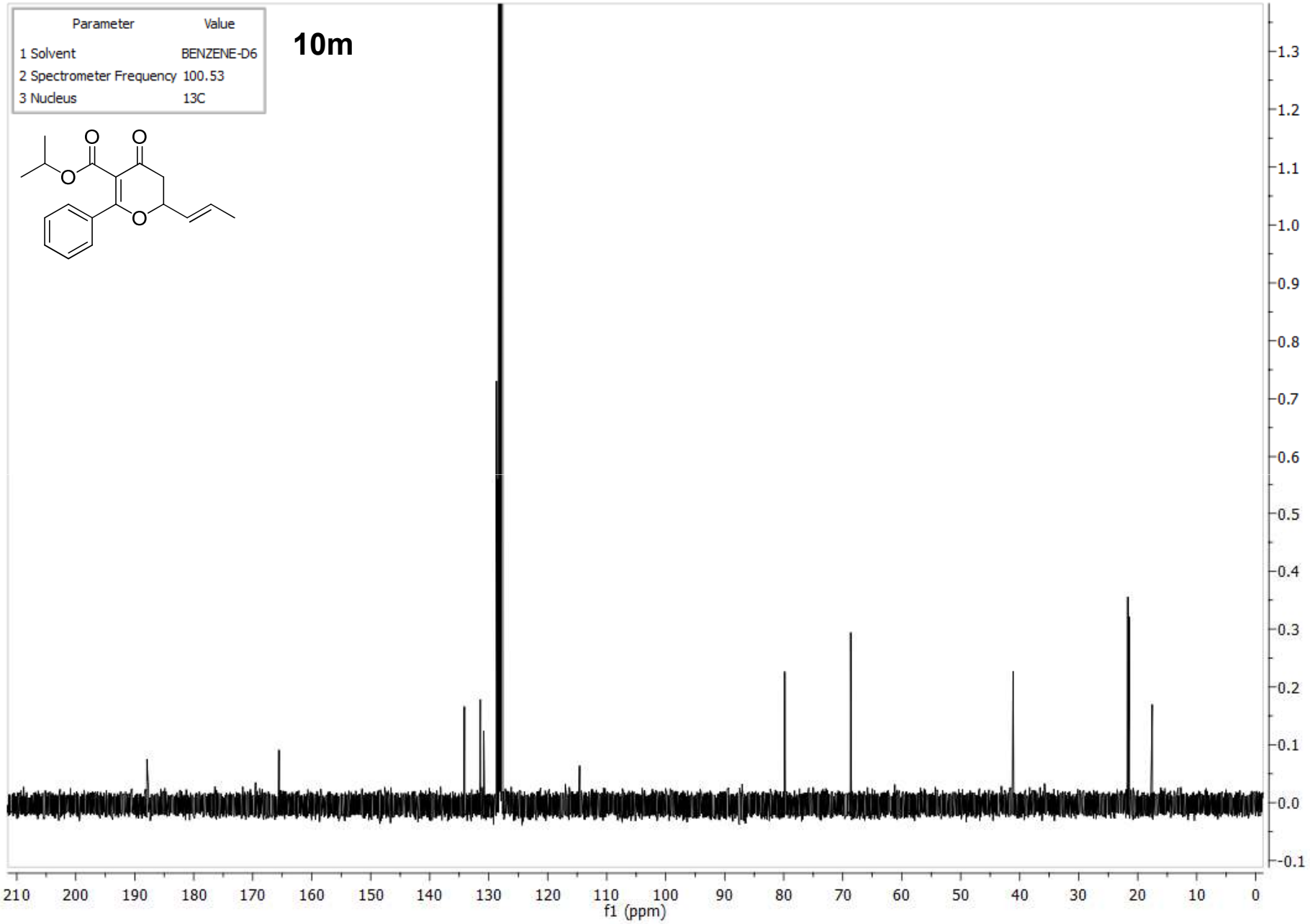

Parameter
Value
1 Solvent
BENZENE-D6
2 Spectrometer Frequency
399.78
3 Nucleus
1H
10k
O
O
O
O
2.09
1.03
1.00
1.90
2.71
2.58
5.36
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

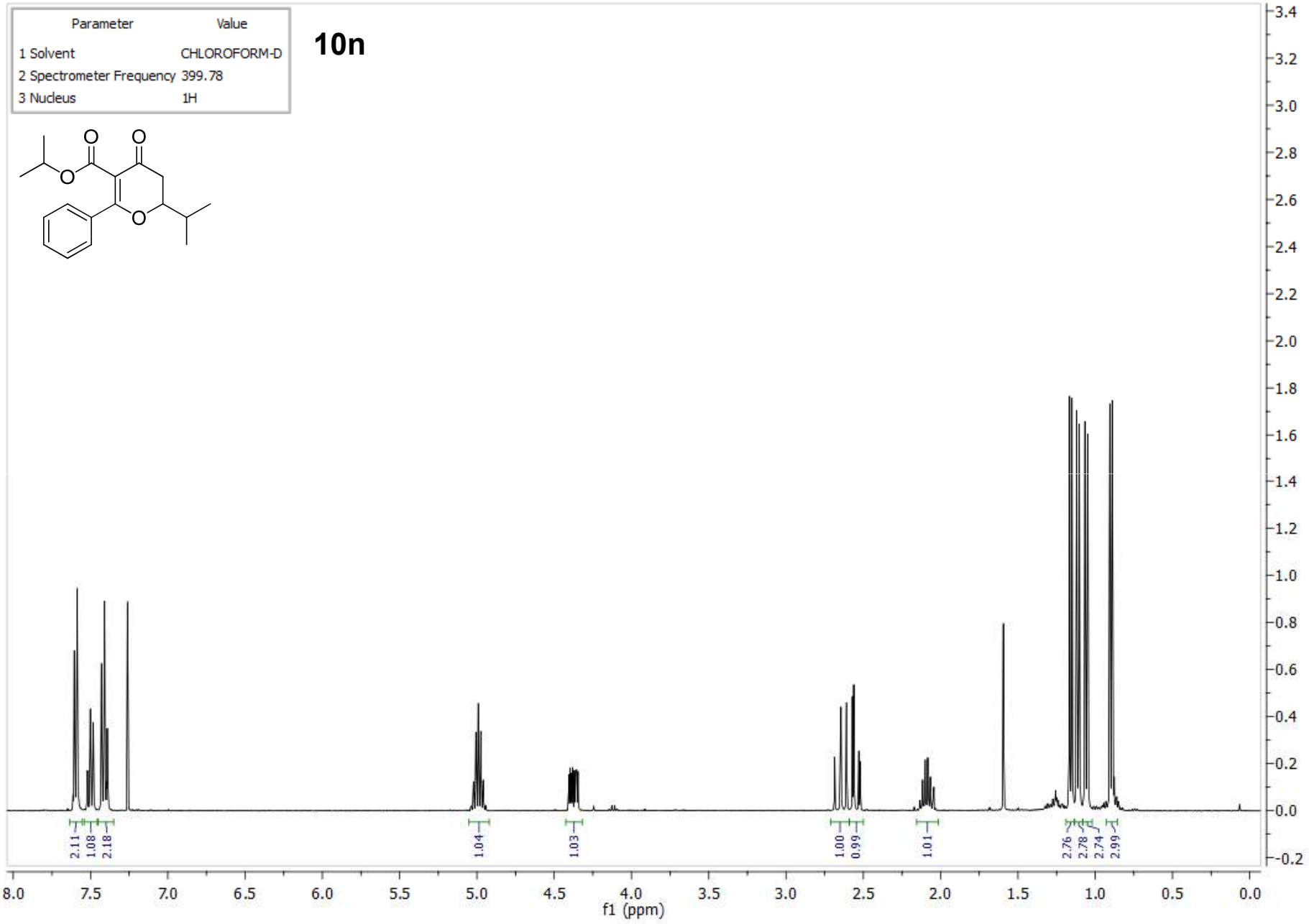

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
10k
f1 (ppm)

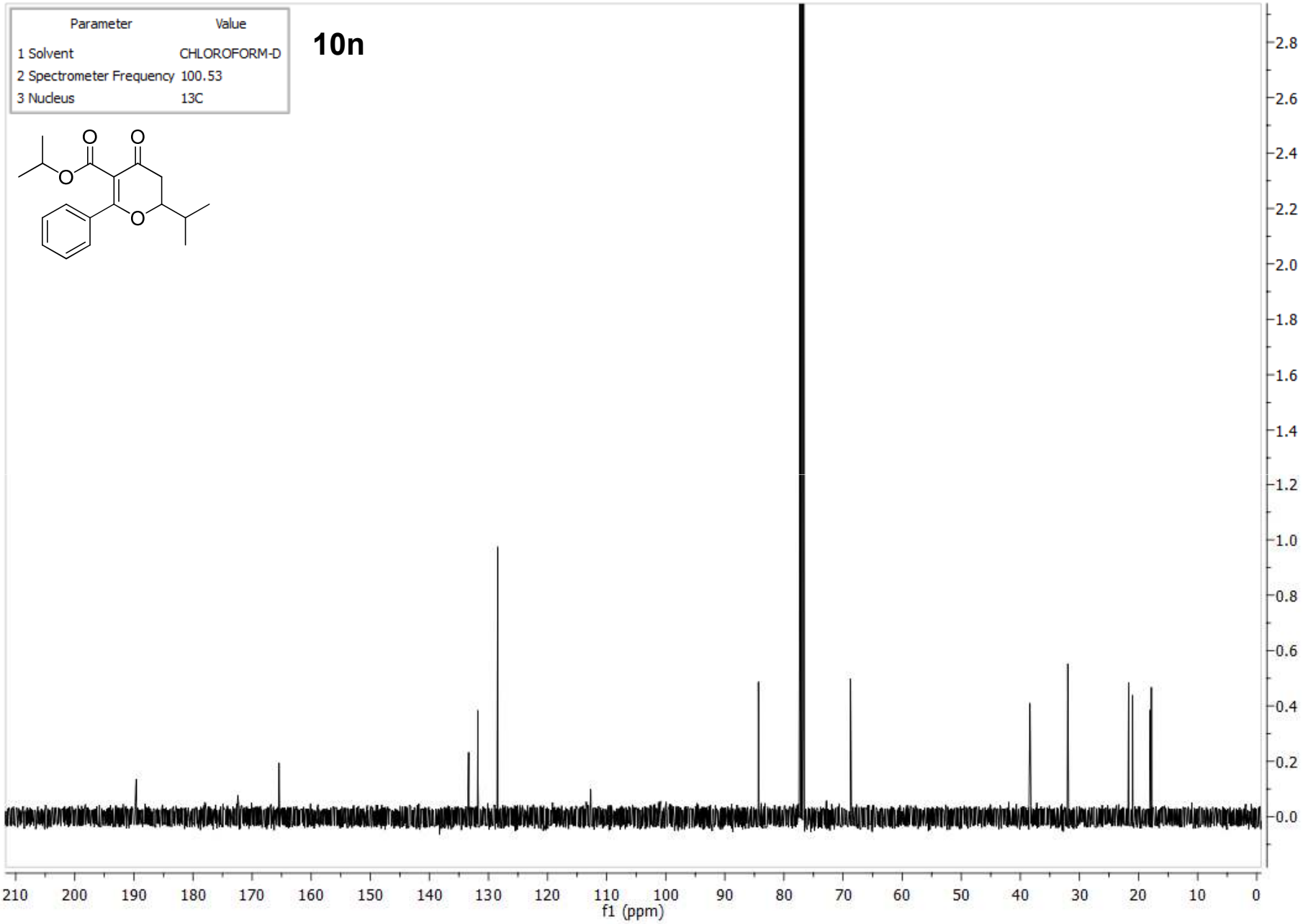

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
10l
O
O
O
O
1.91
3.04
1.00
1.03
1.97
2.16
7.88
2.80
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
3.2
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
-0.2

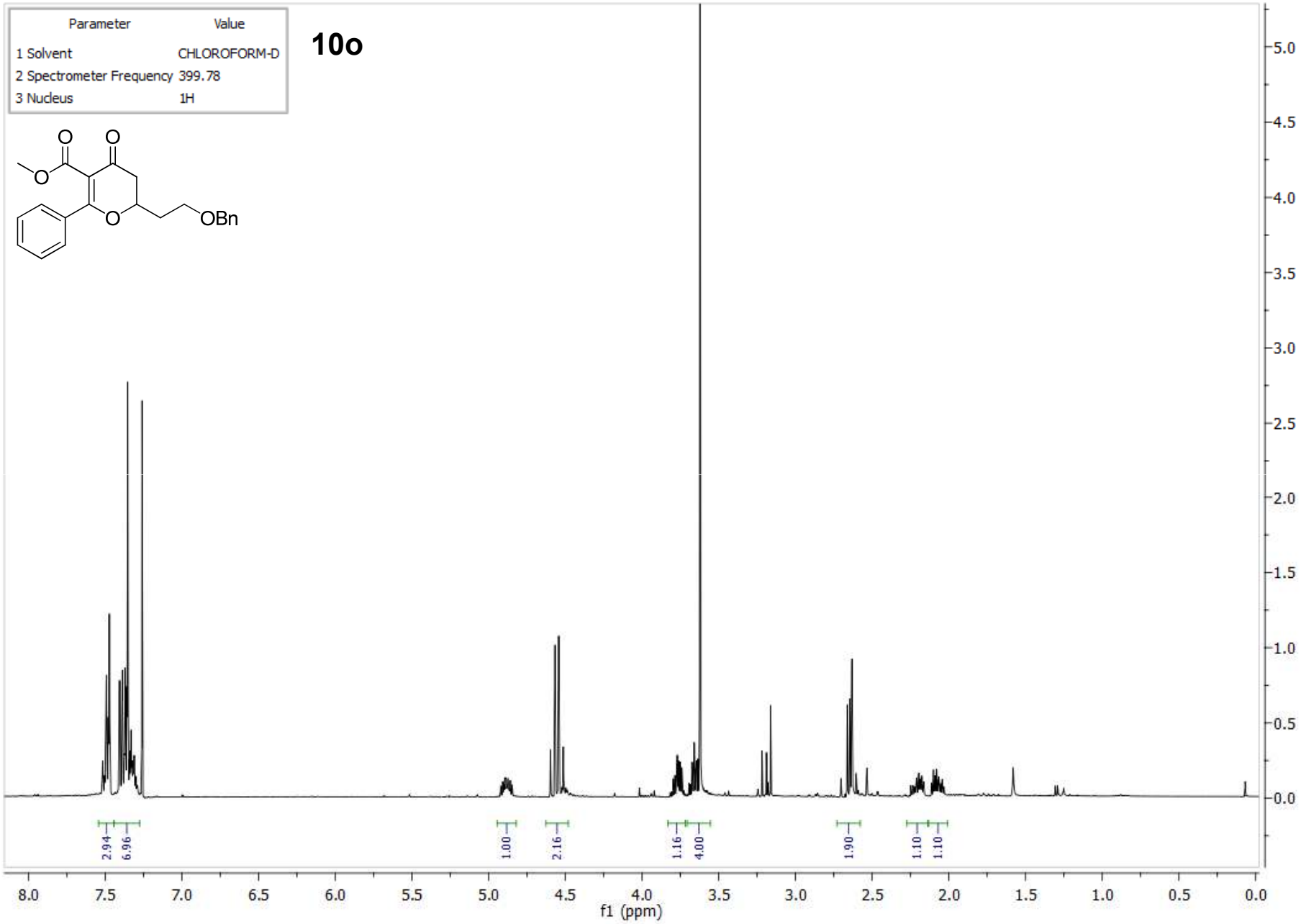

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
10l
O
O
O
O
1.9
1.8
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
-0.1
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

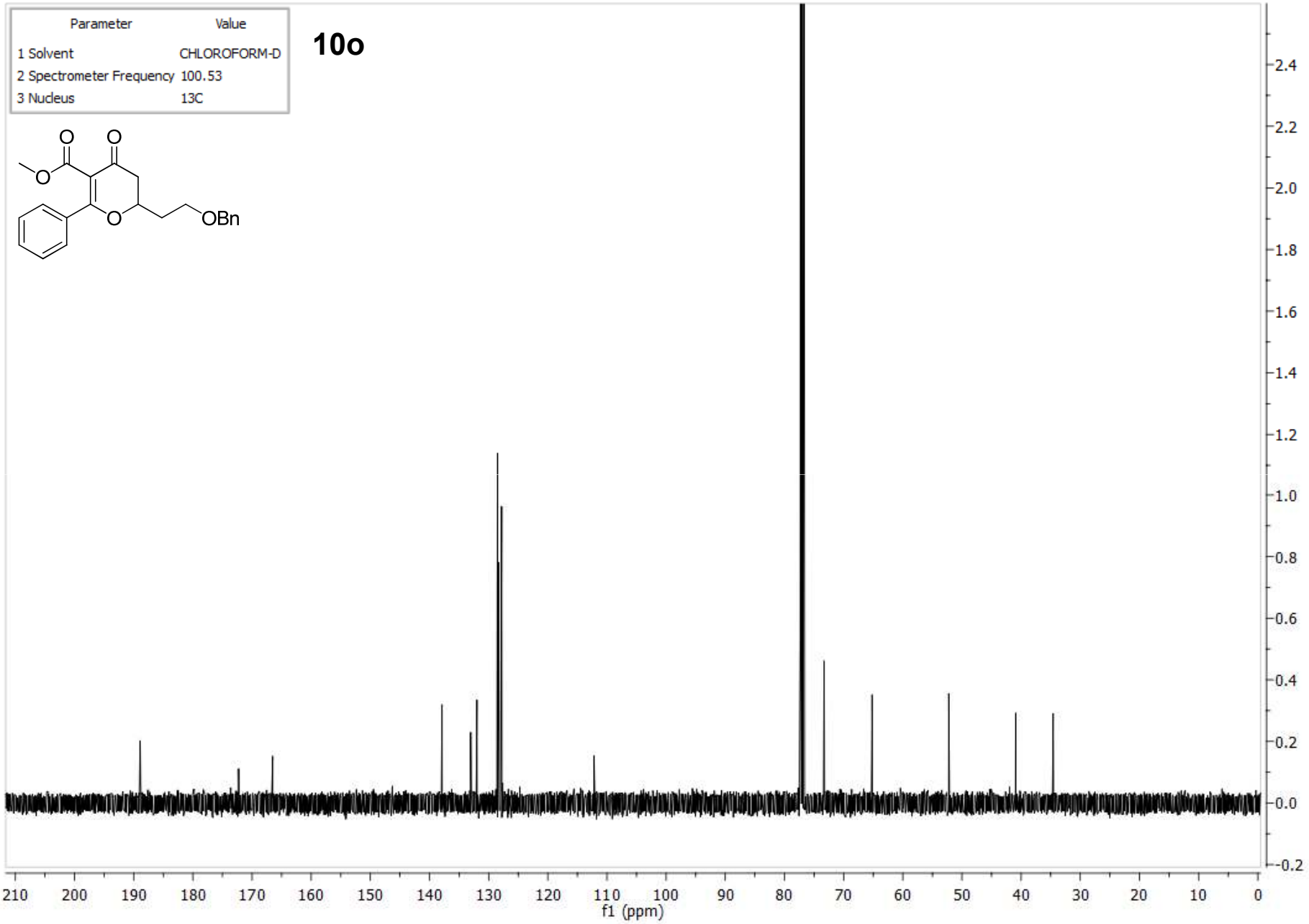

Parameter
Value
1 Solvent
BENZENE-D6
2 Spectrometer Frequency
399.78
3 Nucleus
1H
10m
O
O
O
O
1.96
3.00
1.50
2.22
1.34
2.00
3.12
2.74
2.68
1.65
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
2.3
2.2
2.1
2.0
1.9
1.8
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
-0.1

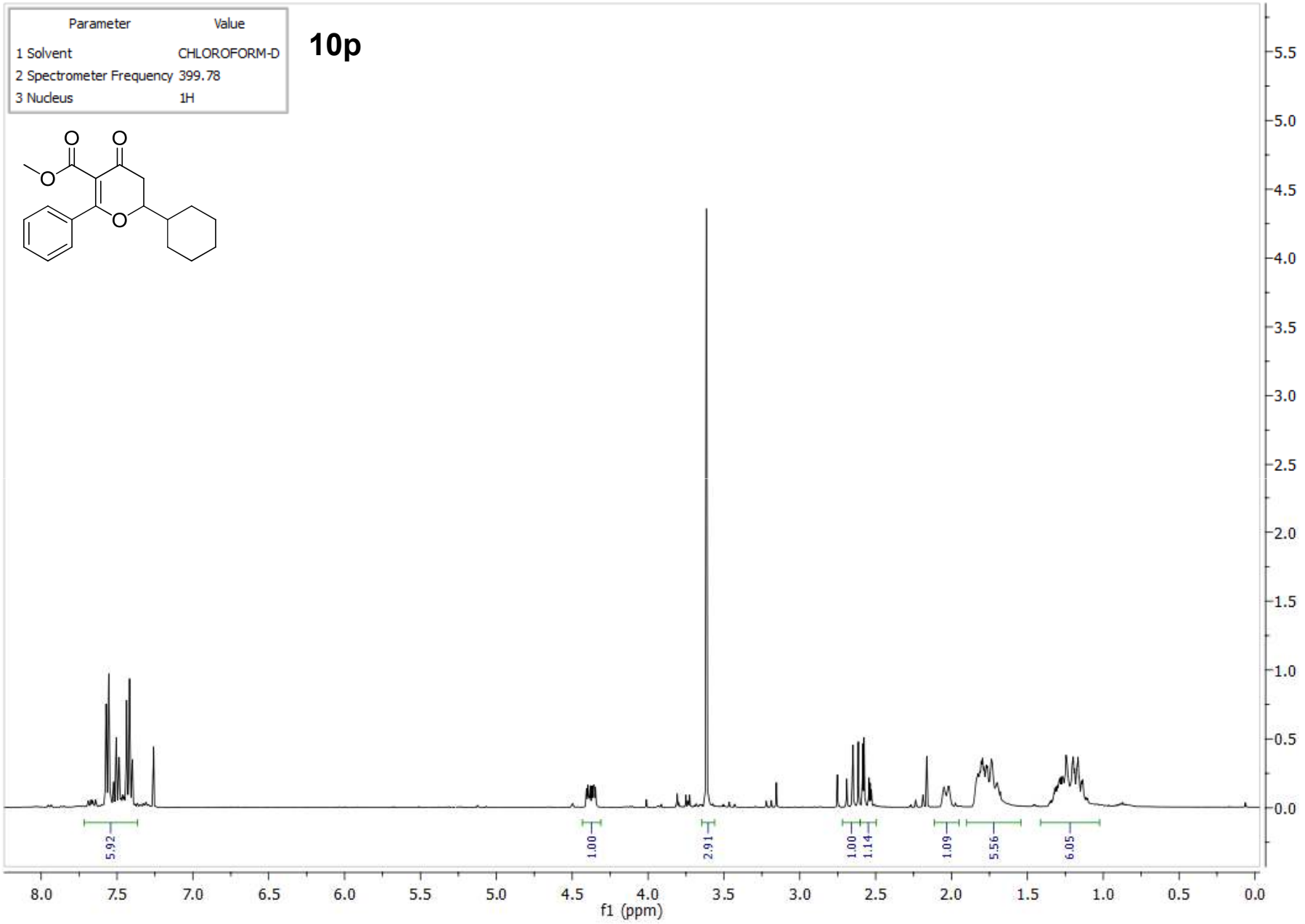

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
10m
O
O
O
O
1.3
1.2
1.1
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
-0.1
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

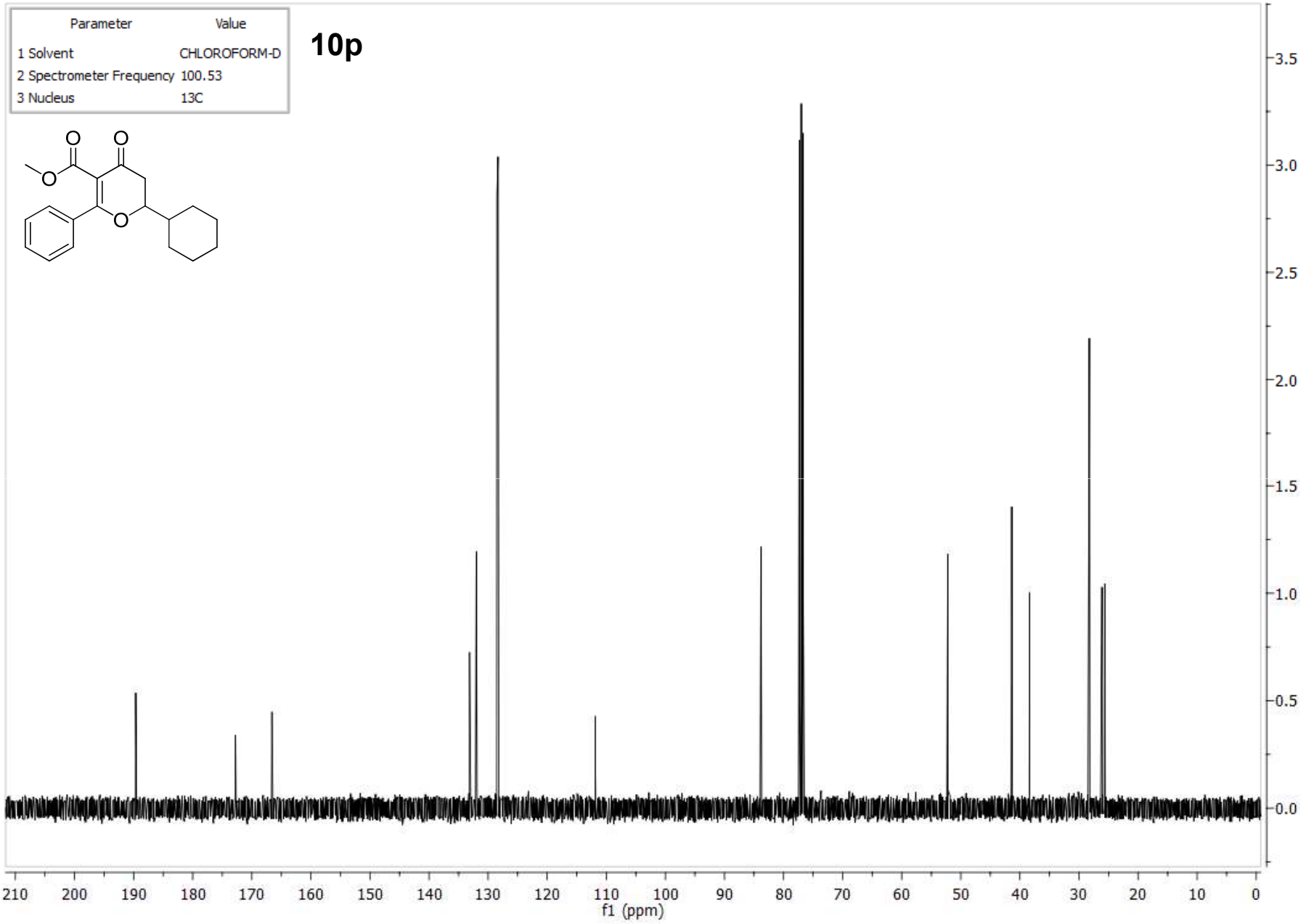

Parameter Value
1 Solvent CHLOROFORM-D
2 Spectrometer Frequency 399.78
3 Nucleus 1H
10n
2.11
1.08
2.18
1.04
1.03
1.00
0.99
1.01
2.76
2.78
2.74
2.99
f1 (ppm)

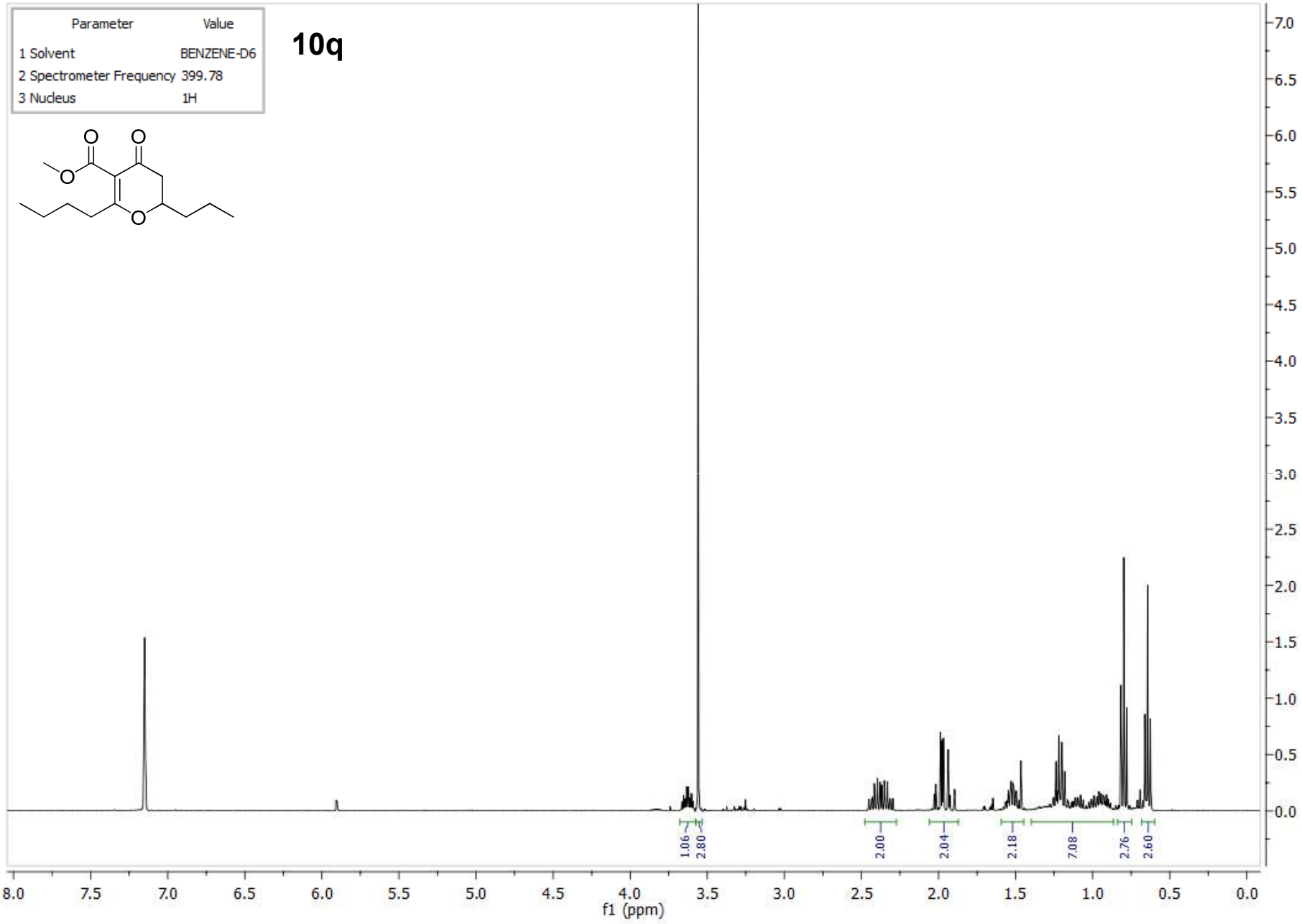

Parameter Value
1 Solvent CHLOROFORM-D
2 Spectrometer Frequency 100.53
3 Nucleus 13C
10n
O
O
O
O
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

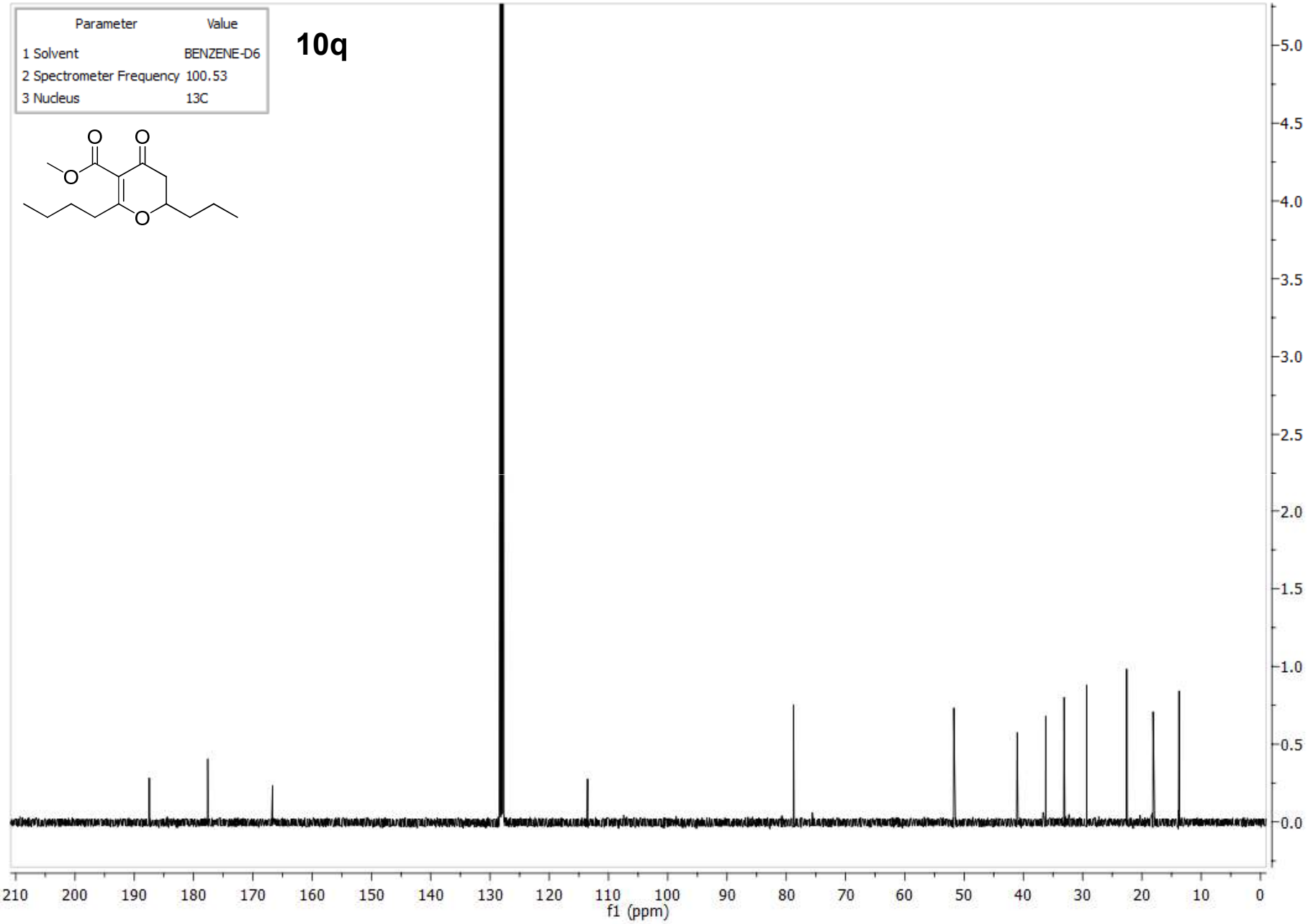

Parameter Value
1 Solvent CHLOROFORM-D
2 Spectrometer Frequency 399.78
3 Nucleus 1H
10o
O
O
O
O
OBn
2.94
6.96
1.00
2.16
1.16
4.00
1.90
1.10
1.10
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

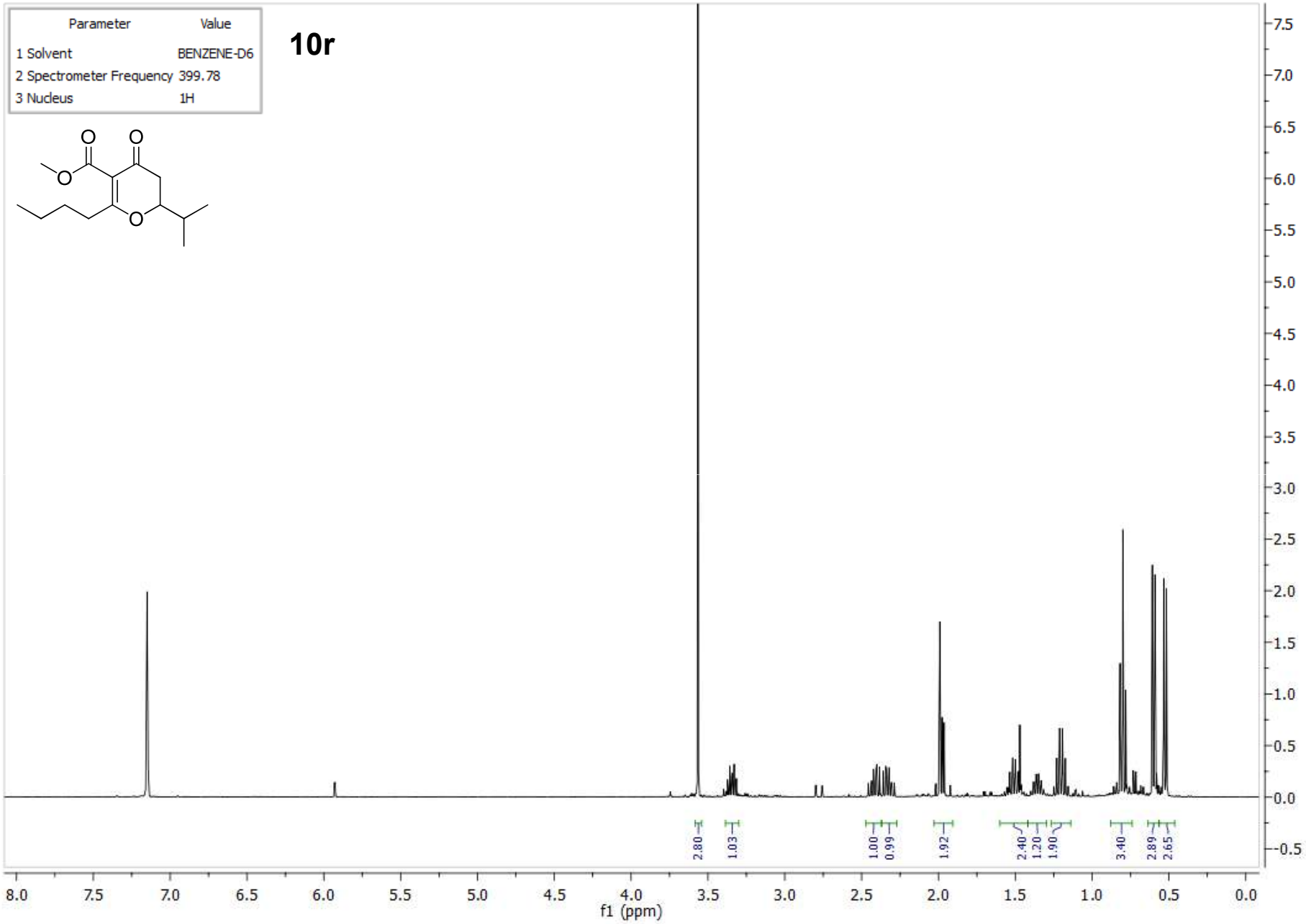

Parameter Value
1 Solvent CHLOROFORM-D
2 Spectrometer Frequency 100.53
3 Nucleus 13C
10o
O
O
O
O
OBn
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
-0.2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

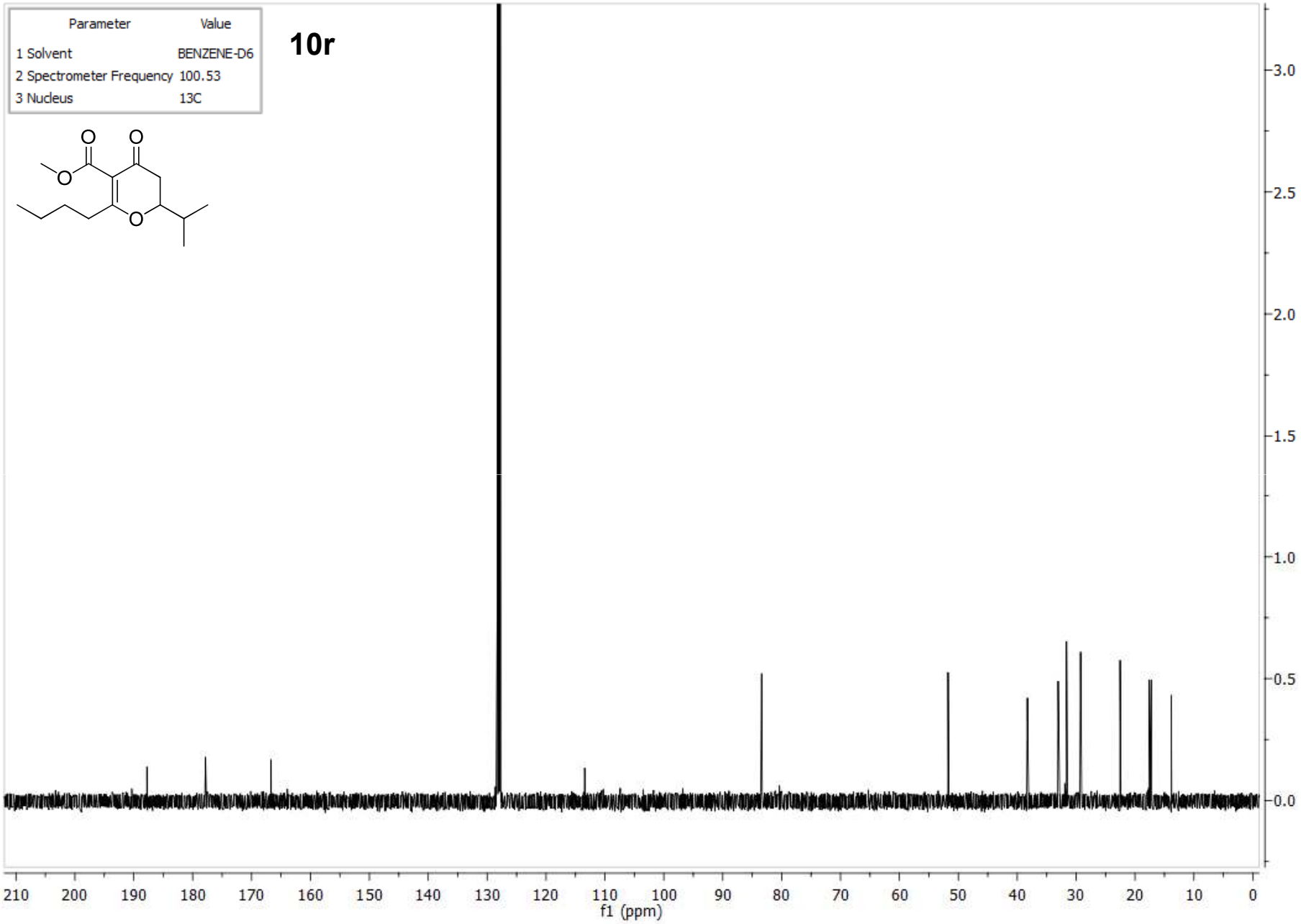

Parameter Value
1 Solvent CHLOROFORM-D
2 Spectrometer Frequency 399.78
3 Nucleus 1H
10p
O
O
O
O
5.92
1.00
2.91
1.00
1.14
1.09
5.56
6.05
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

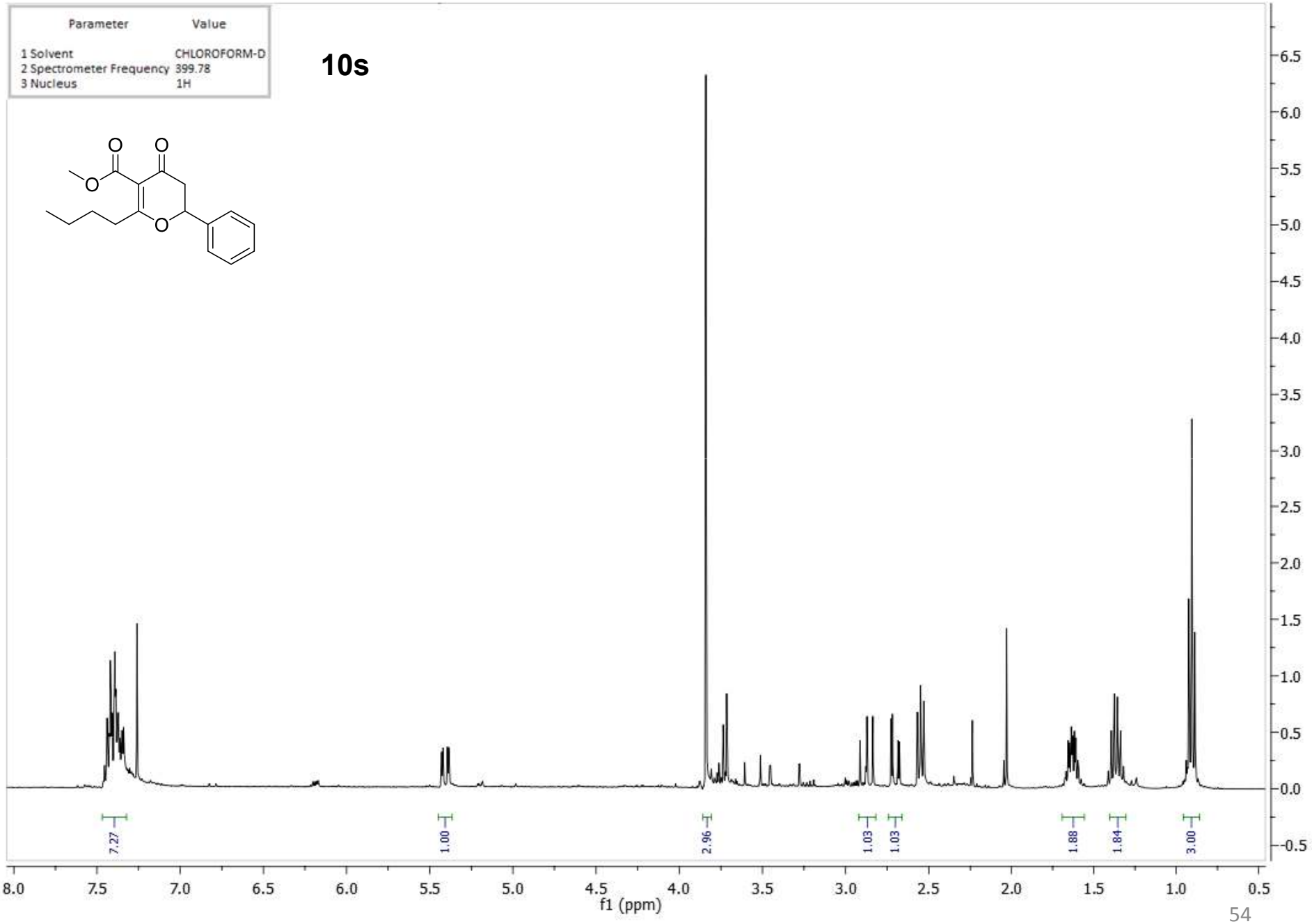

Parameter Value
1 Solvent CHLOROFORM-D
2 Spectrometer Frequency 100.53
3 Nucleus 13C
10p
O
O
O
O
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

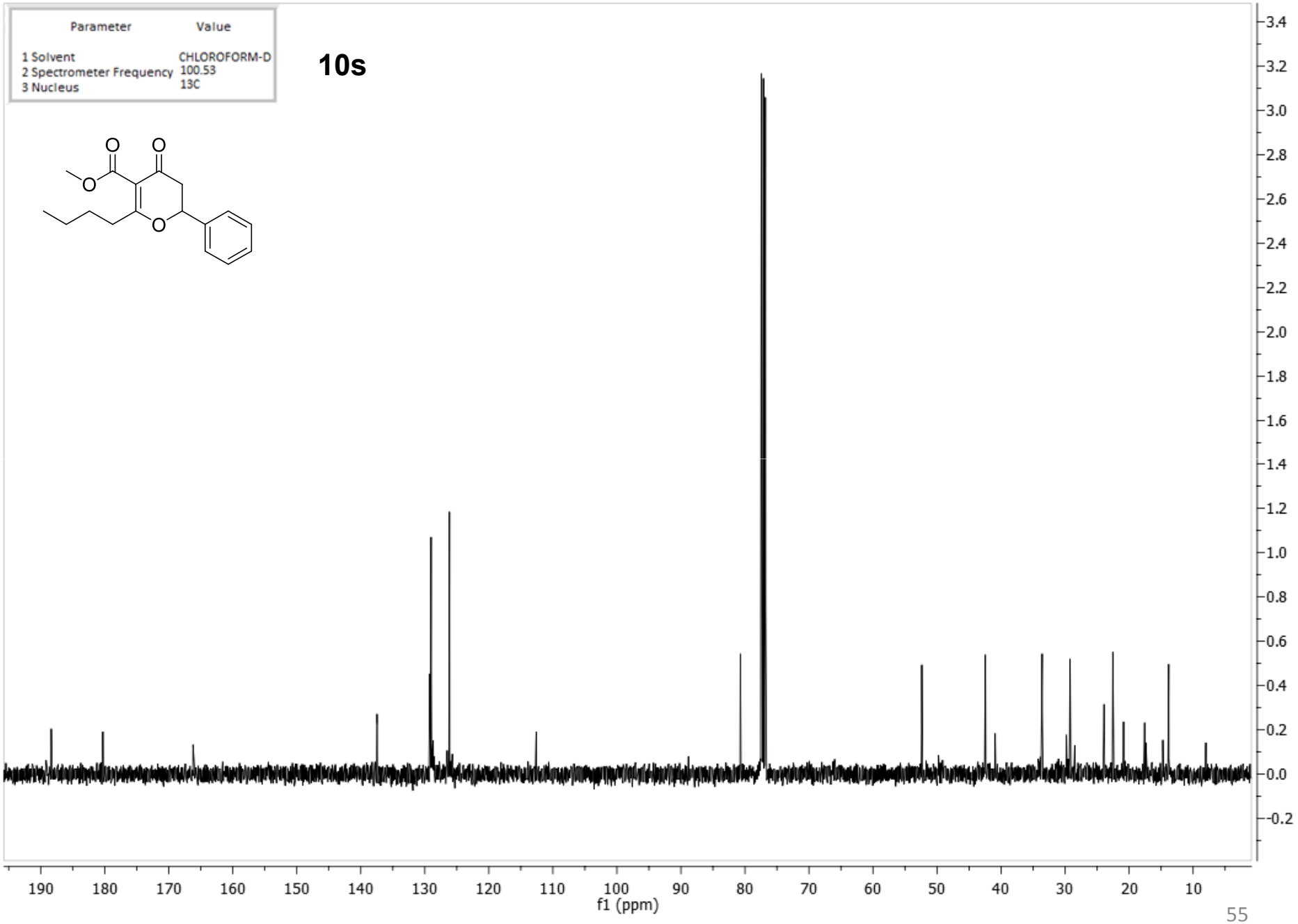

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
10q
1.06
2.80
2.00
2.04
2.18
7.08
2.76
2.60
f1 (ppm)

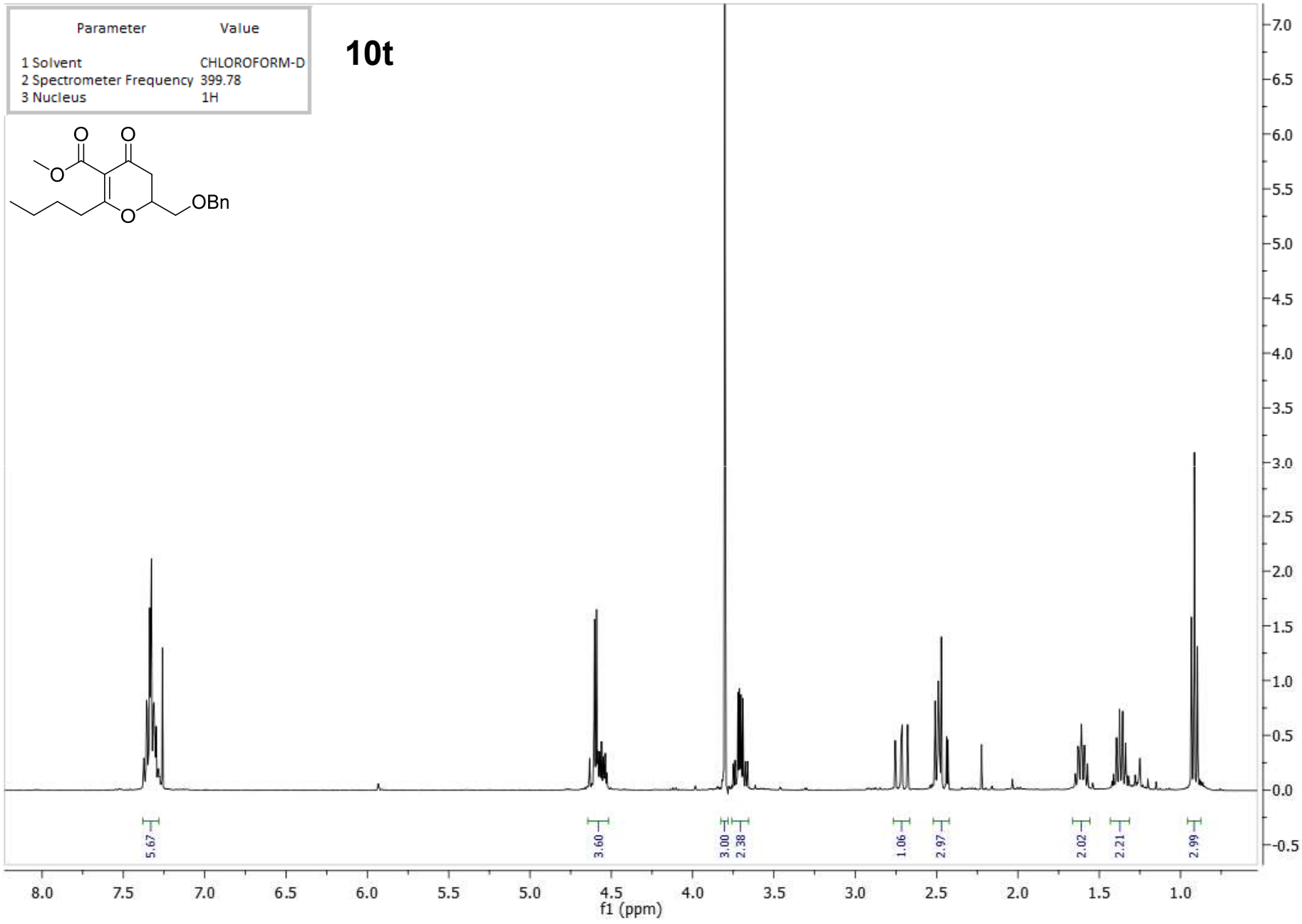

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
10q
O
O
O
O
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
10r
2.80
1.03
1.00
0.99
1.92
2.40
1.20
1.90
3.40
2.89
2.65
f1 (ppm)


Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
10r
O
O
O
O
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

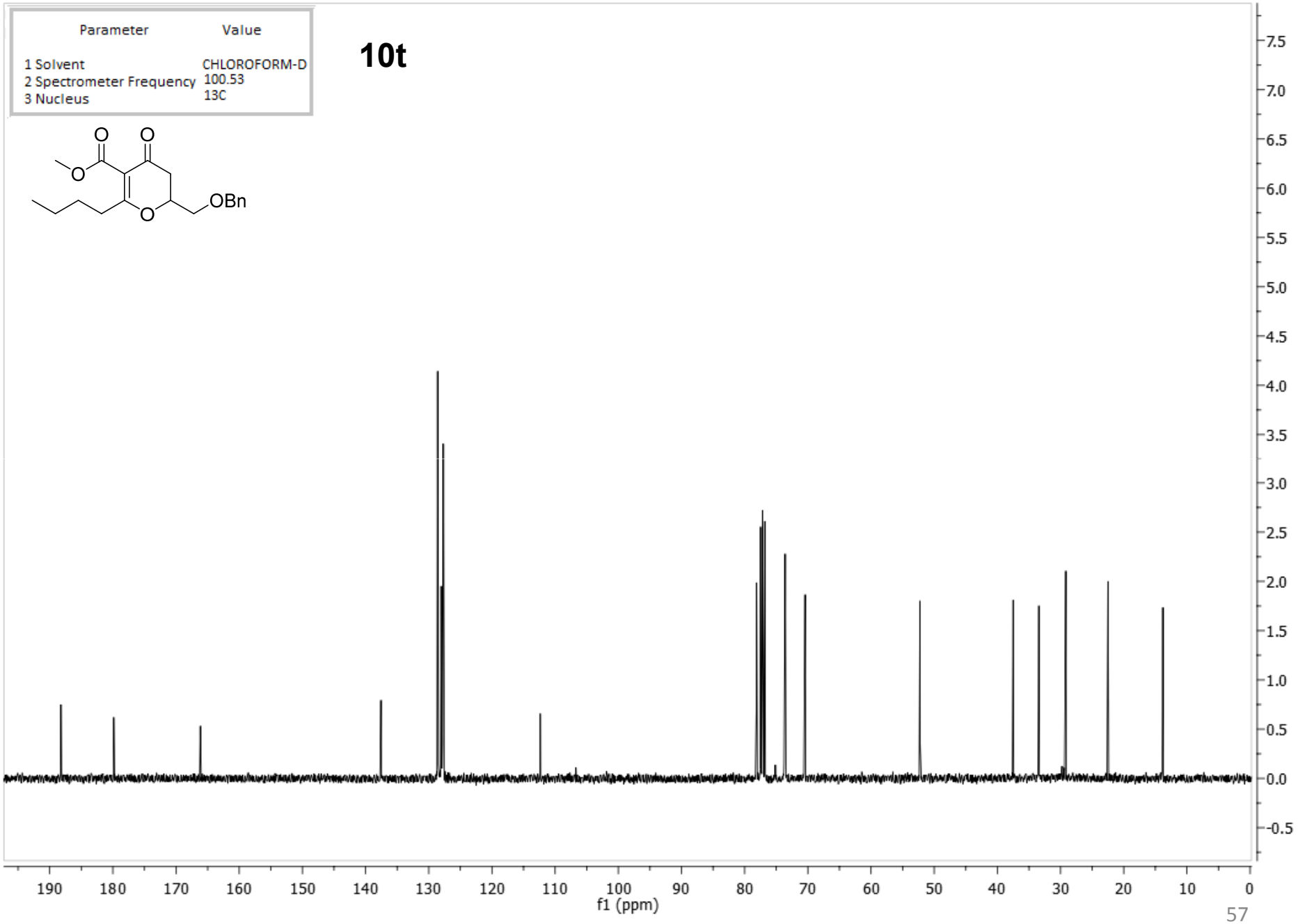

Parameter Value
1 Solvent CHLOROFORM-D
2 Spectrometer Frequency 399.78
3 Nucleus 1H
10s
O
O
O
O
7.27
1.00
2.96
1.03
1.03
1.88
1.84
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5

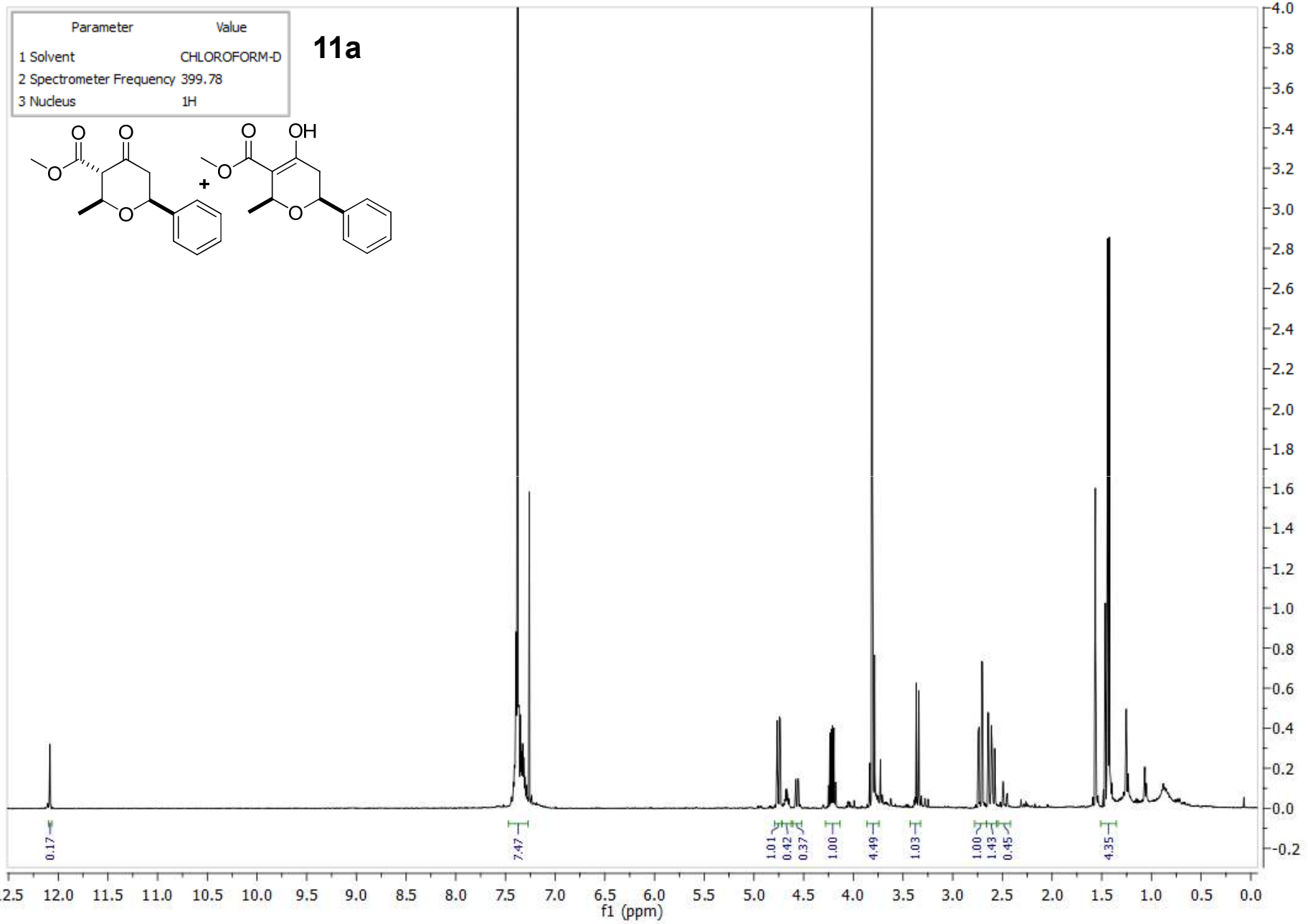

Parameter Value
1 Solvent CHLOROFORM-D
2 Spectrometer Frequency 100.53
3 Nucleus 13C
10s
f1 (ppm)

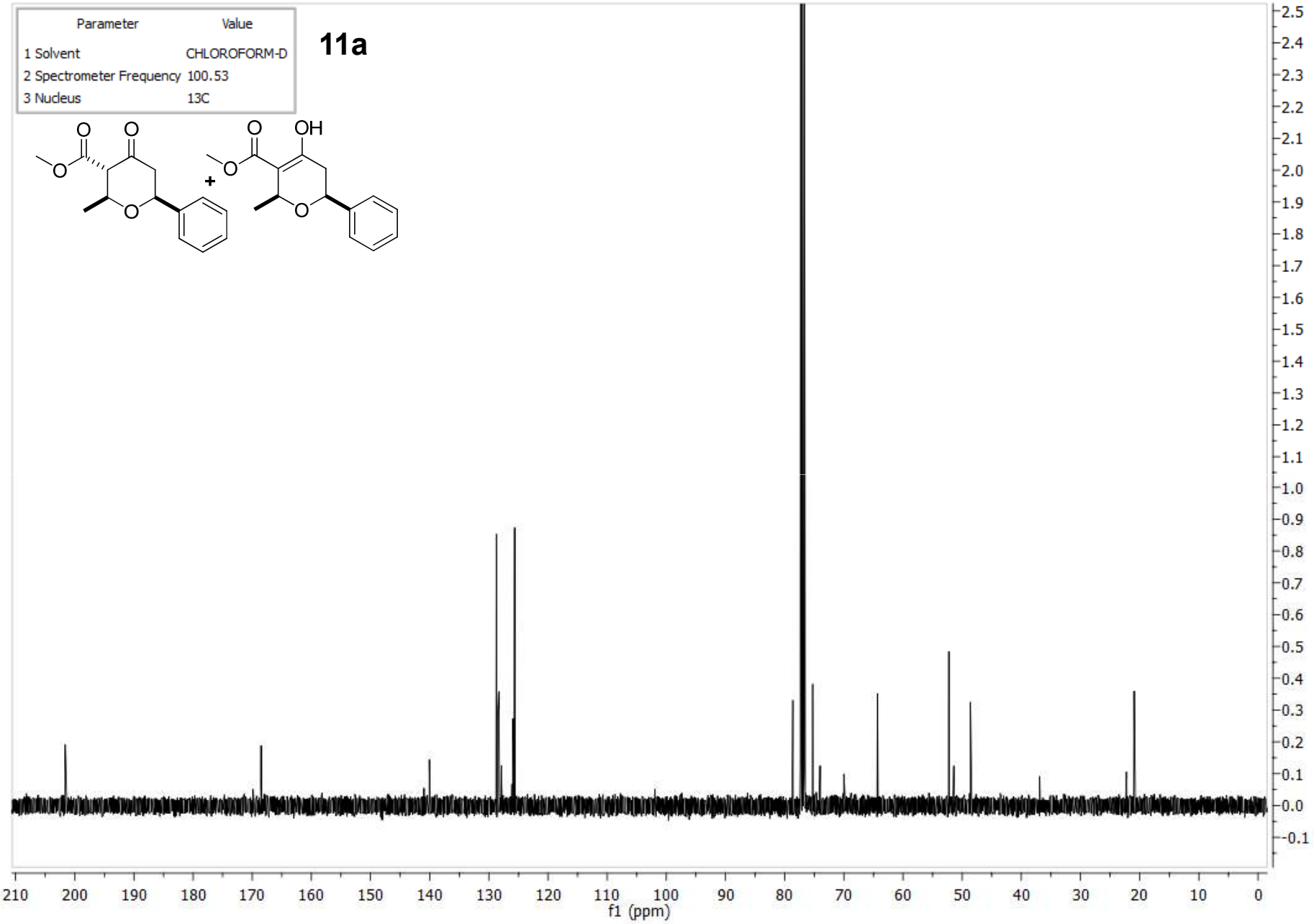

Parameter Value
1 Solvent CHLOROFORM-D
2 Spectrometer Frequency 399.78
3 Nucleus 1H
10t
OBn
5.67
3.60
3.00
2.38
1.06
2.97
2.02
2.21
2.99
f1 (ppm)

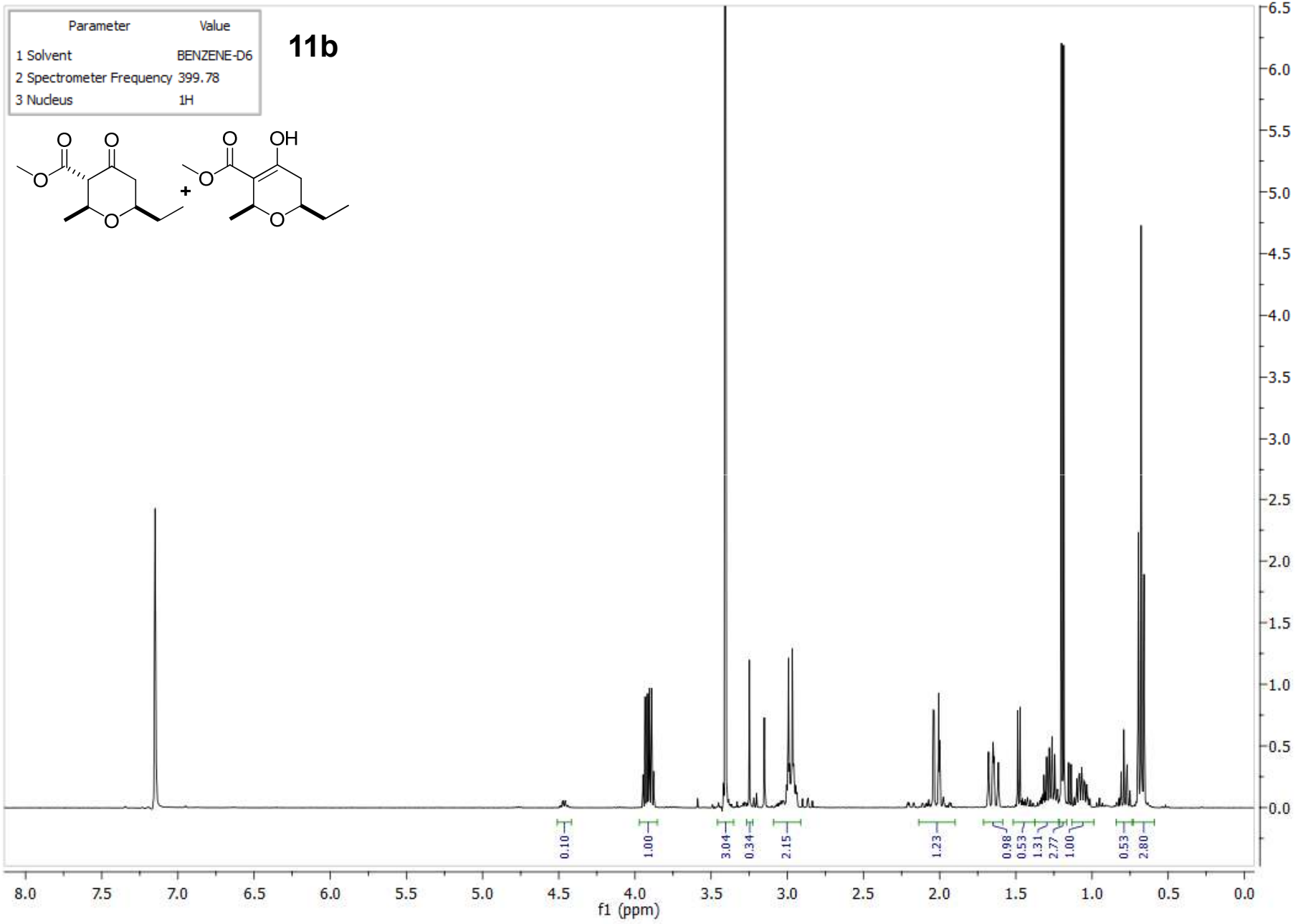
Parameter	Value
1 Solvent	CHLOROFORM-D
2 Spectrometer Frequency	100.53
3 Nucleus	13C

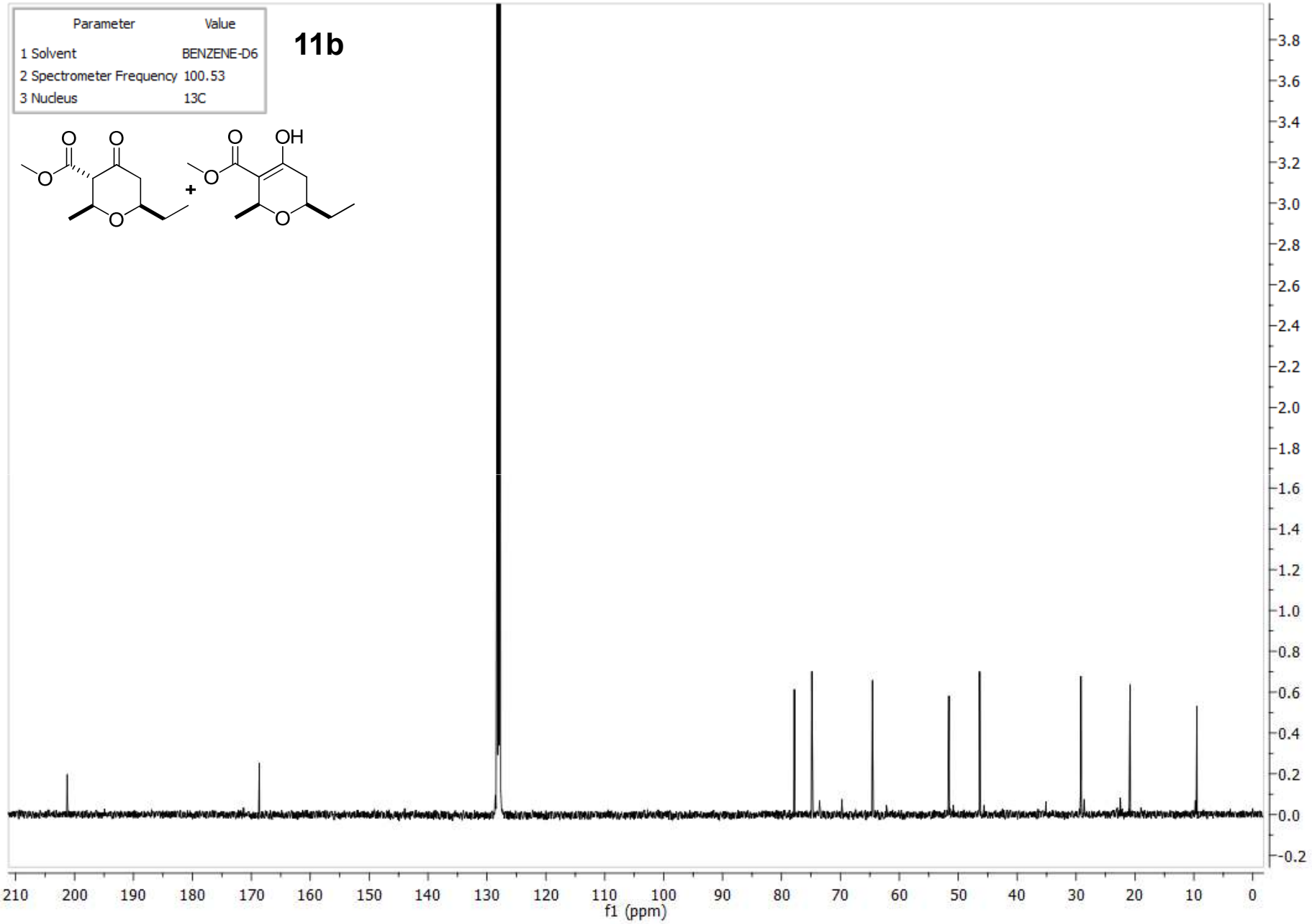
10t

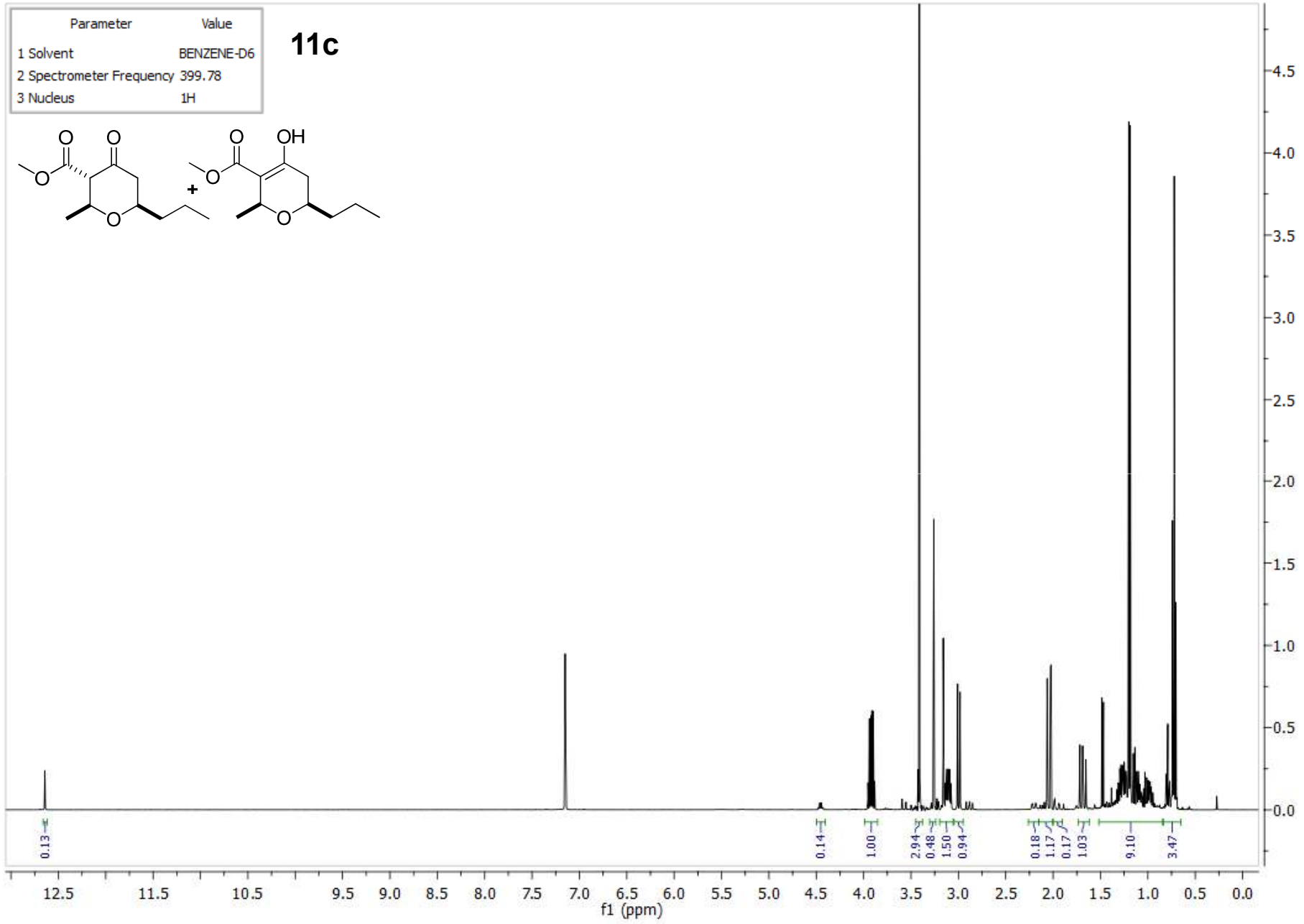


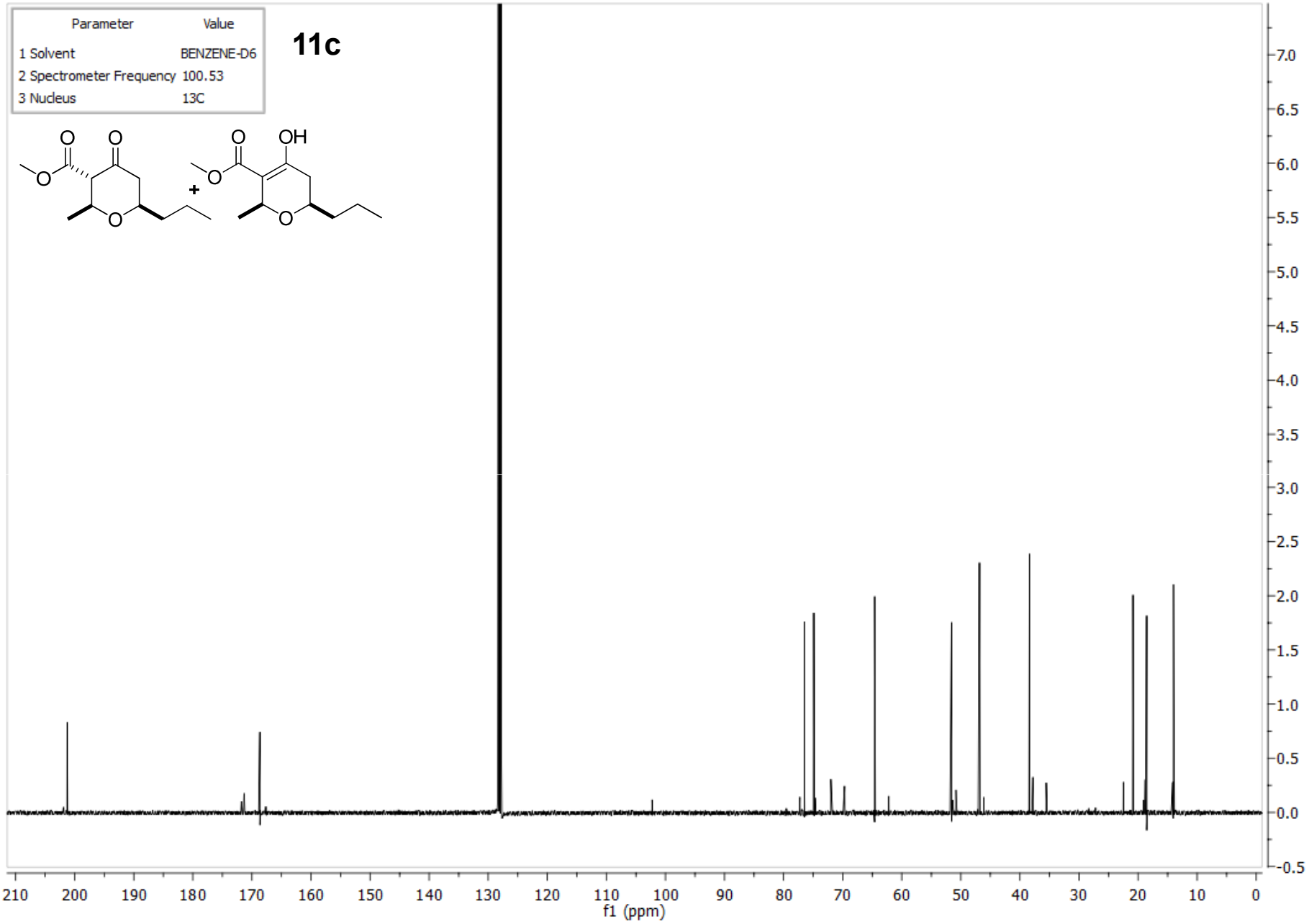

Parameter Value
1 Solvent CHLOROFORM-D
2 Spectrometer Frequency 399.78
3 Nucleus 1H
11a
O
O
O
O
O
OH
+
0.17
7.47
1.01
0.42
0.37
1.00
4.49
1.03
1.00
1.43
0.45
4.35
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6 1.4 1.2 1.0 0.8 0.6 0.4 0.2 0.0 -0.2

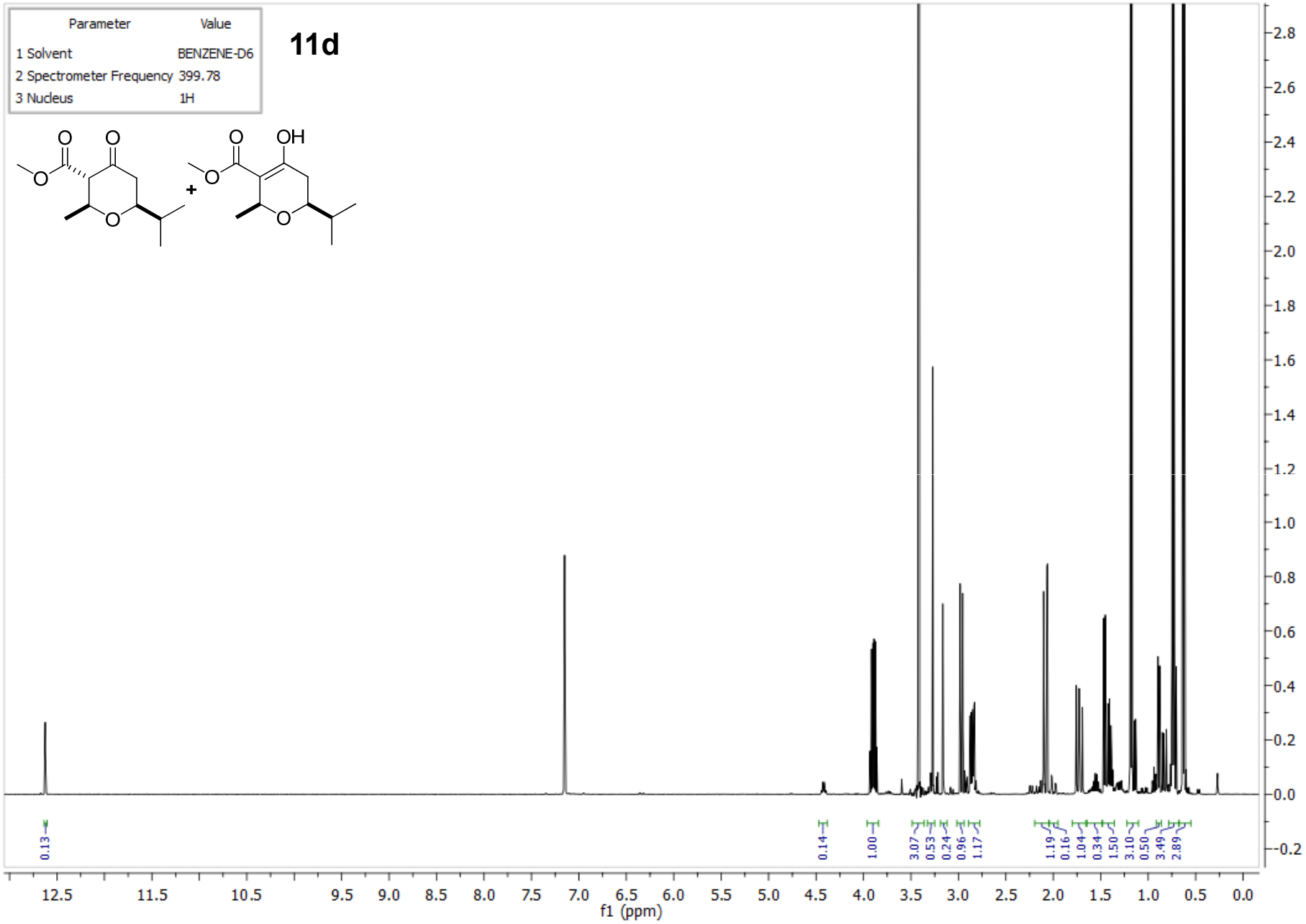

Parameter Value
1 Solvent CHLOROFORM-D
2 Spectrometer Frequency 100.53
3 Nucleus 13C
11a
O
O
O
O
O
OH
+
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
-0.1
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

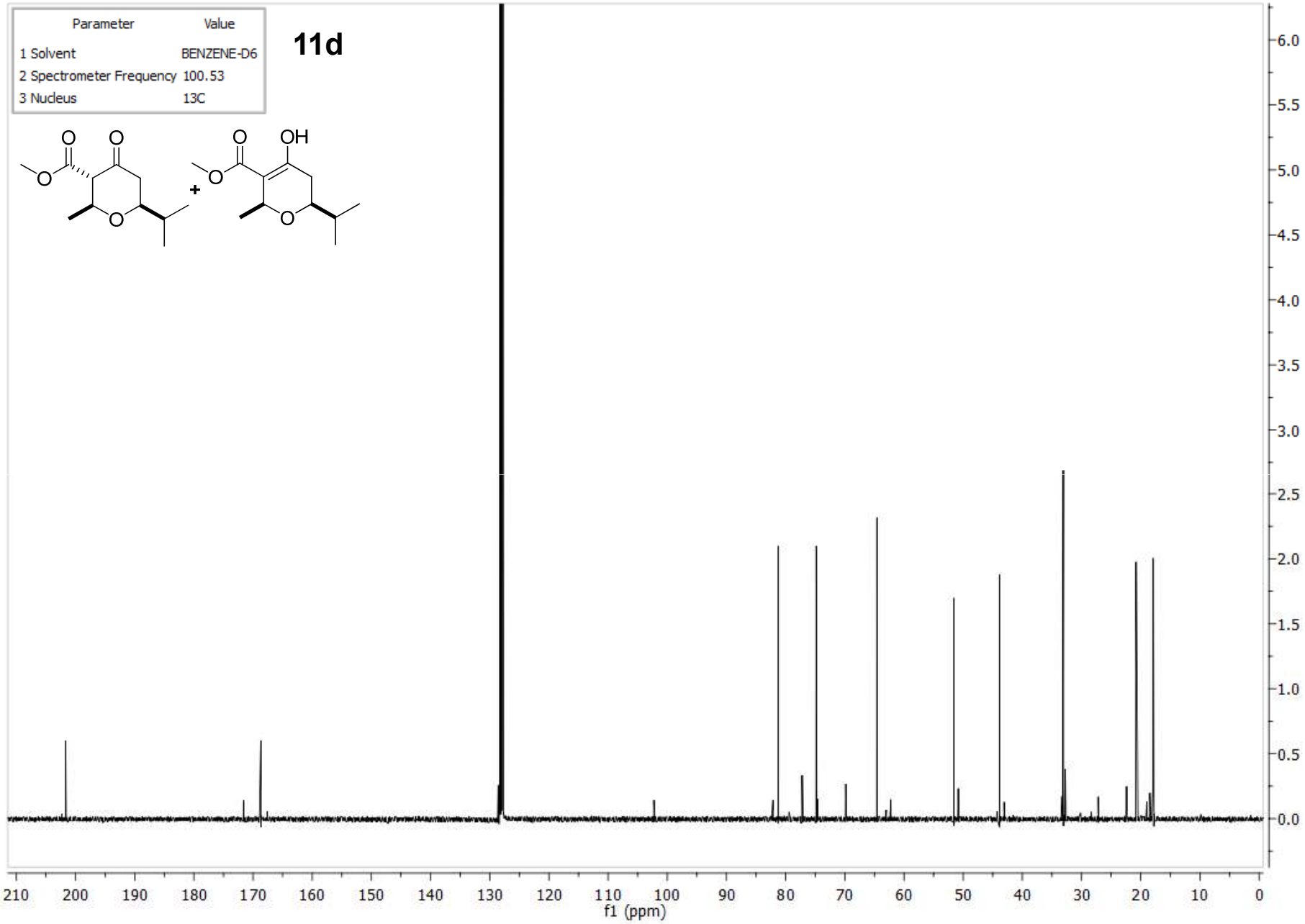

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
11b
O
O
O
O
+
O
OH
O
0.10
1.00
3.04
0.34
2.15
1.23
0.98
0.53
1.31
2.77
1.00
0.53
2.80
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

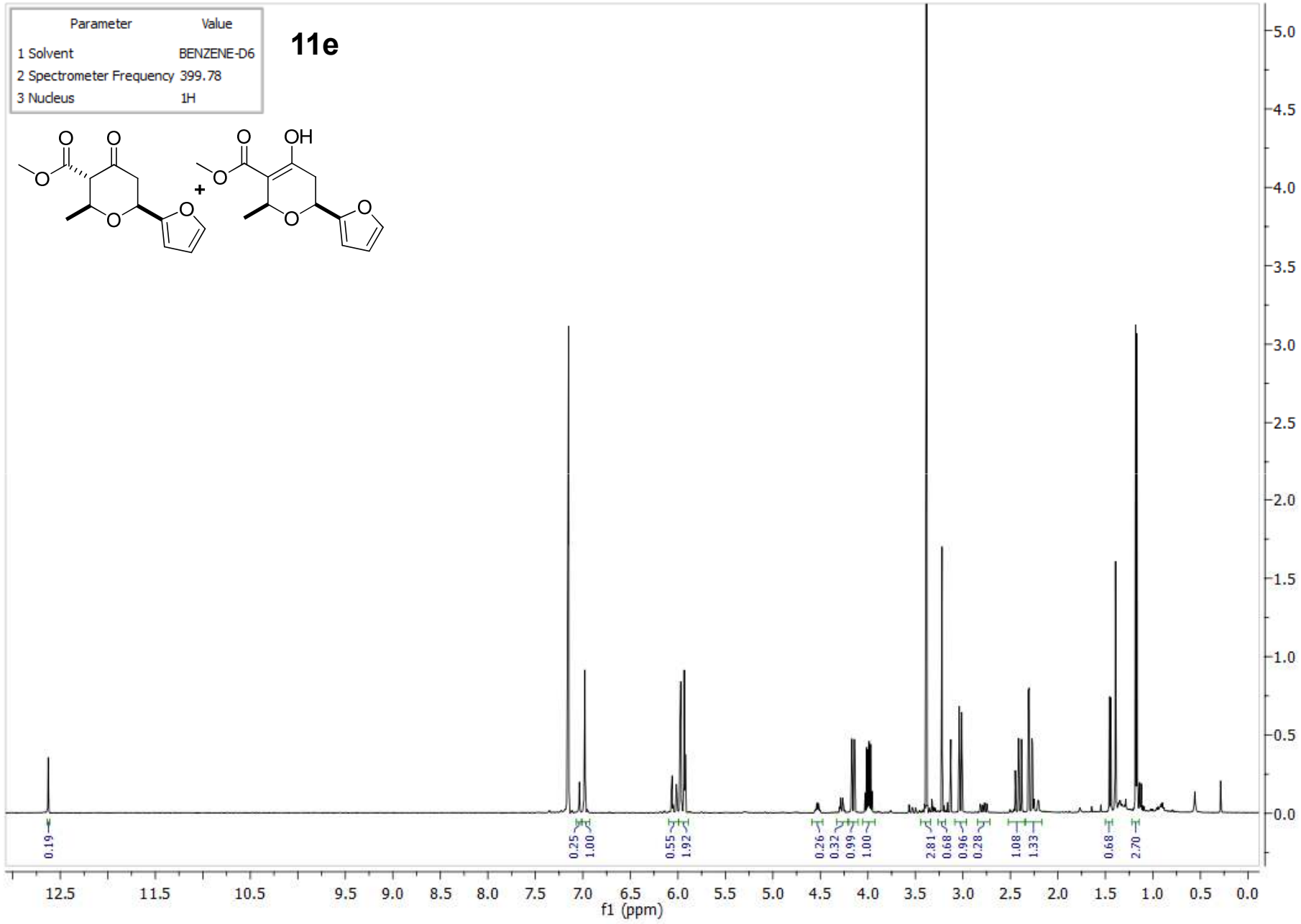

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
11b
O
O
O
O
+
O
OH
O
3.8
3.6
3.4
3.2
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
-0.2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

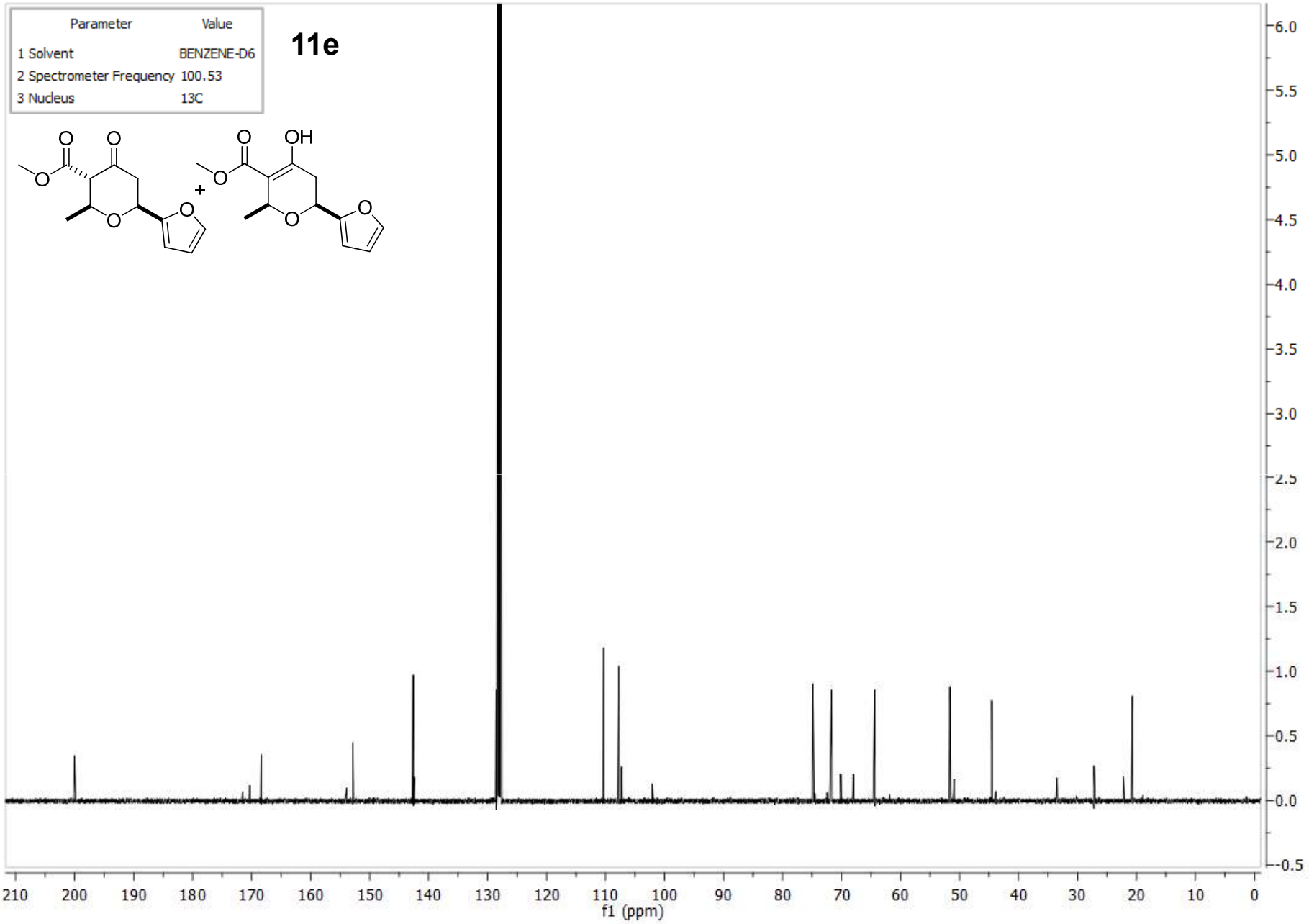

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
11c
O
O
O
O
OH
+
0.13
0.14
1.00
2.94
0.48
1.50
0.94
0.18
1.17
0.17
1.03
9.10
3.47
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

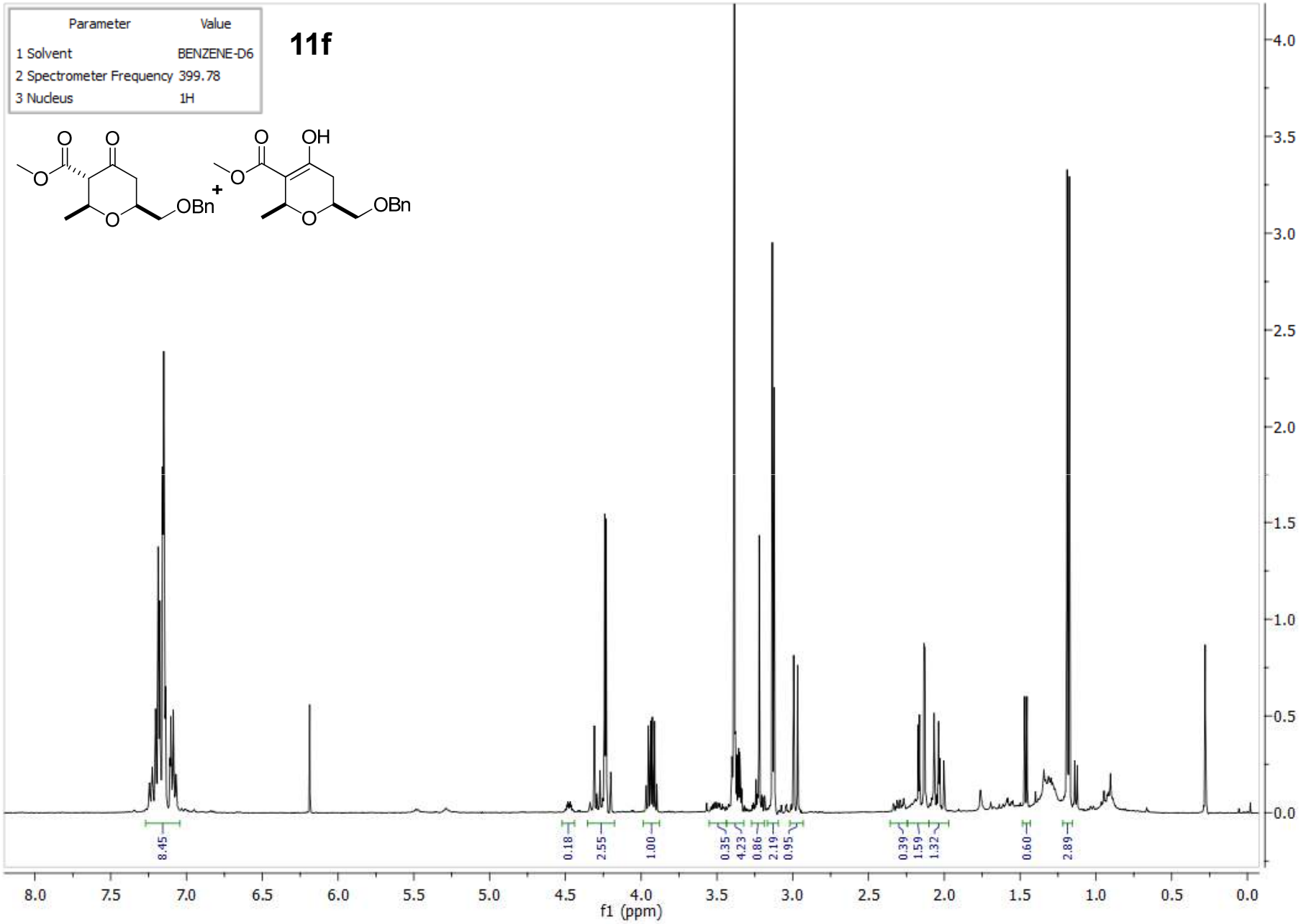

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
11c
O
O
O
O
O
OH
+
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

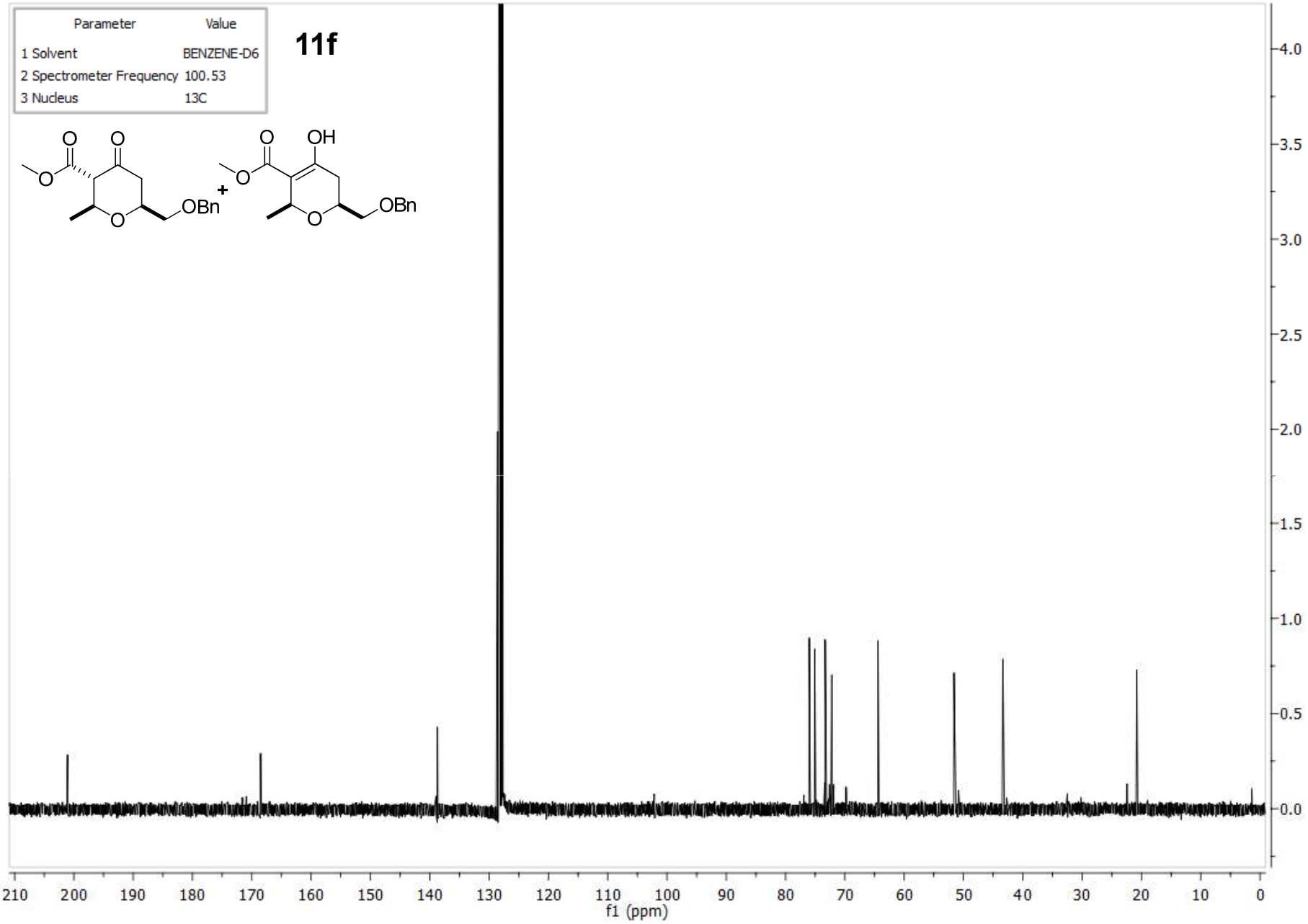

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
11d
O
O
O
O
+
O
OH
O
0.13
0.14
1.00
3.07
0.53
0.24
0.96
1.17
1.19
0.16
1.04
0.34
1.50
3.10
0.50
3.49
2.89
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
-0.2

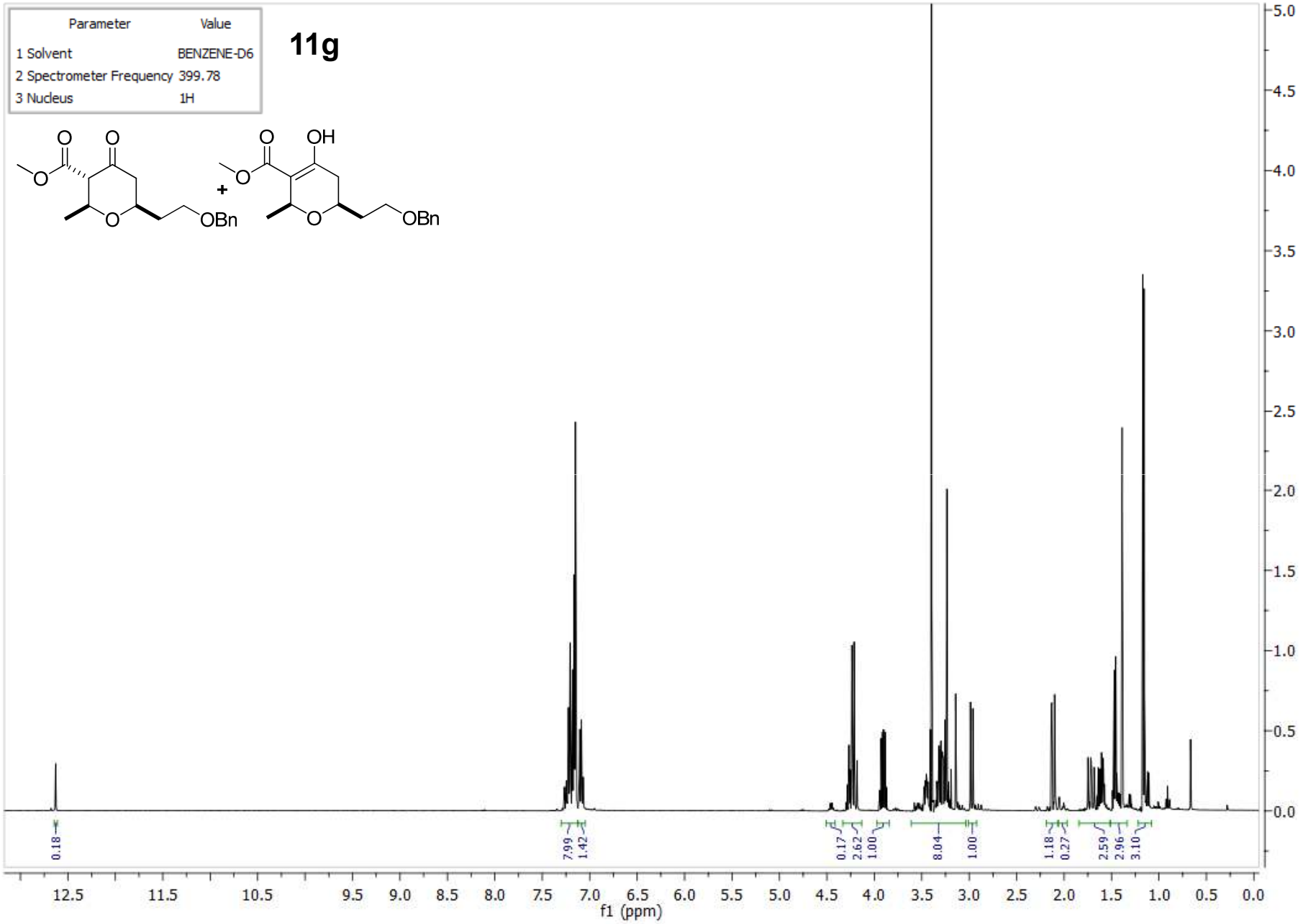

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
11d
O
O
O
O
+
O
OH
O
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

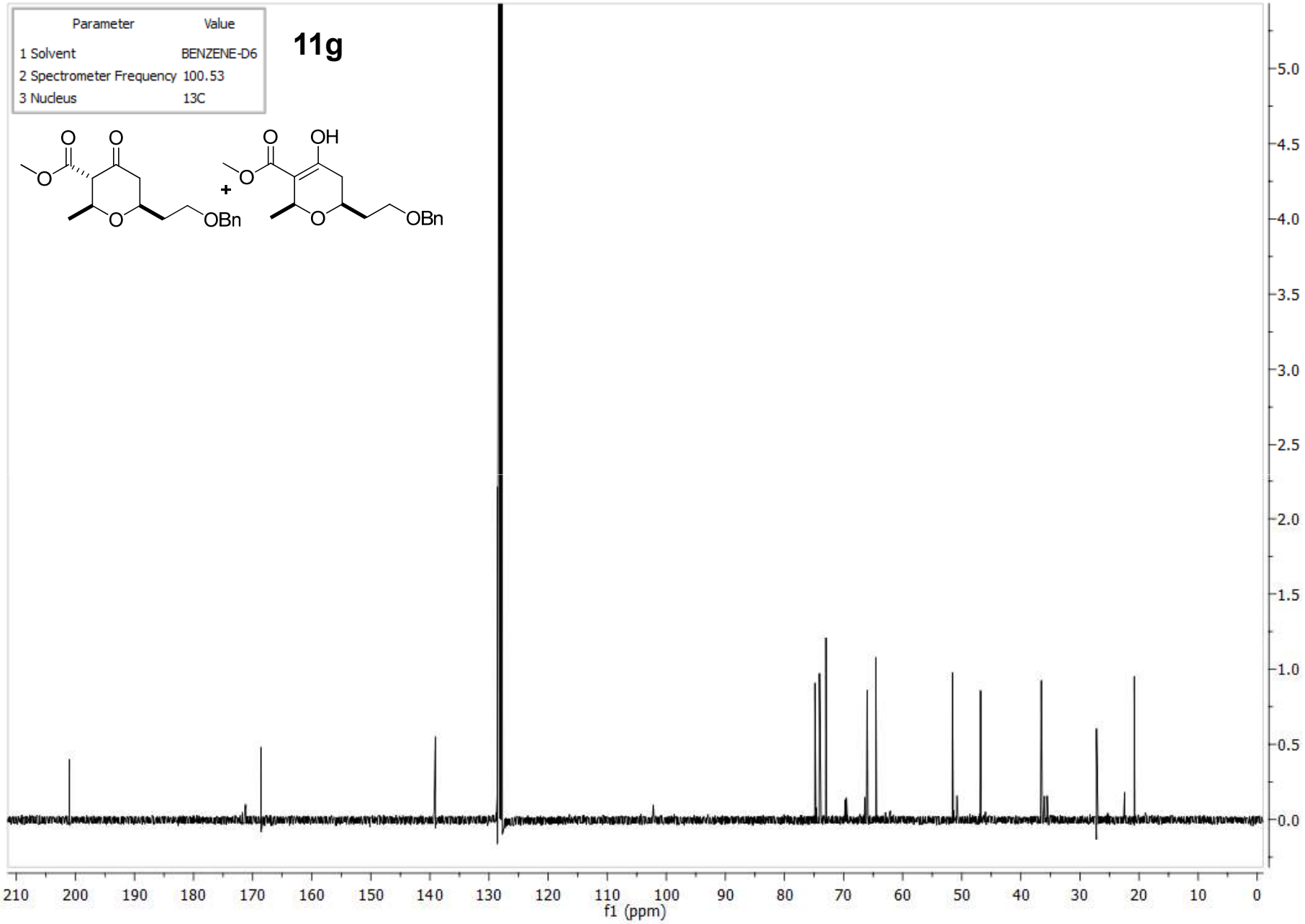

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
11e
OH
0.19
0.25
1.00
0.55
1.92
0.26
0.32
0.99
1.00
2.81
0.68
0.96
0.28
1.08
1.33
0.68
2.70
f1 (ppm)

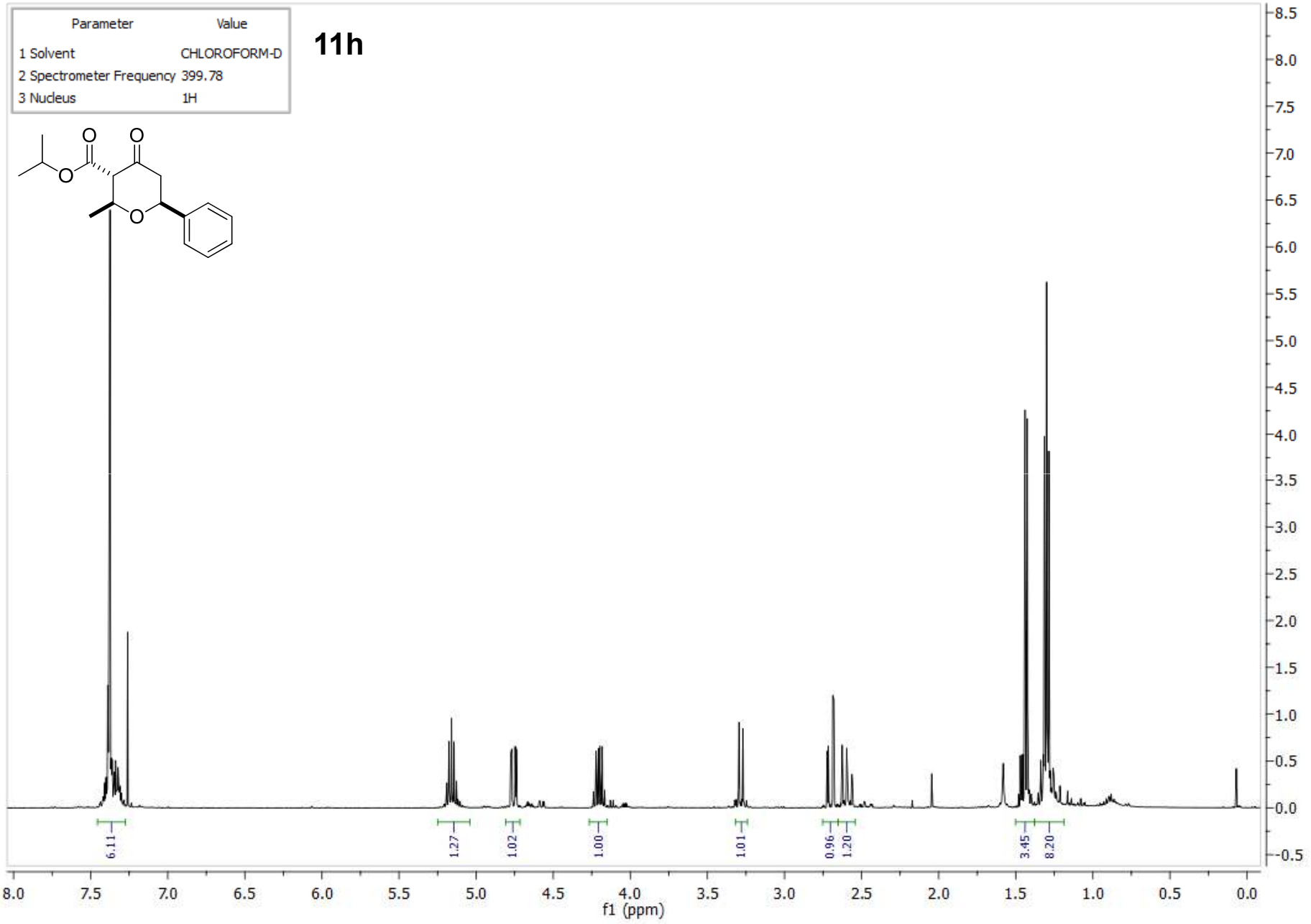

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
11e
O
O
O
O
+
O
OH
O
O
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

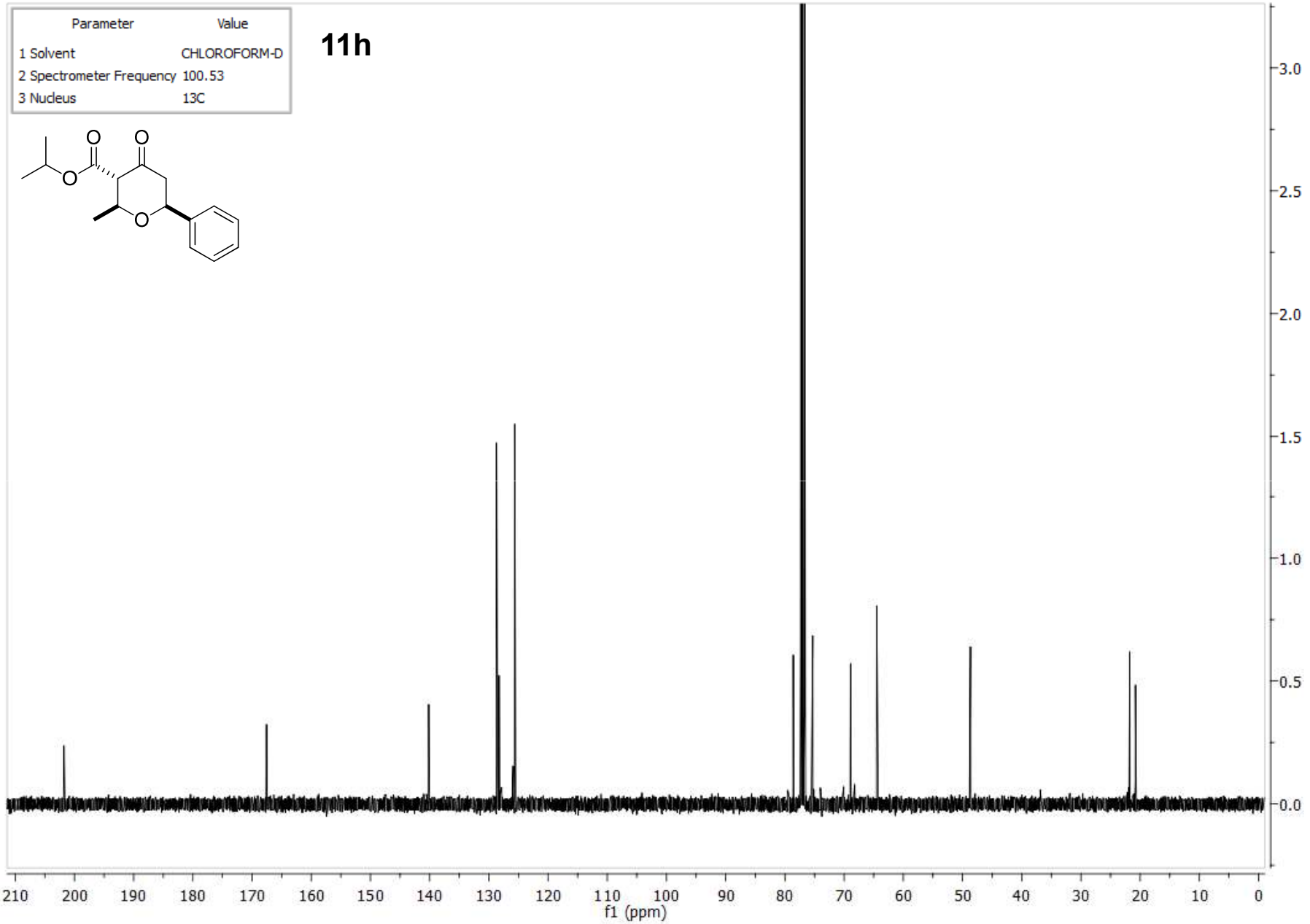

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
11f
OBn
OH
OBn
8.45
0.18
2.55
1.00
0.35
4.23
0.86
2.19
0.95
0.39
1.59
1.32
0.60
2.89
f1 (ppm)

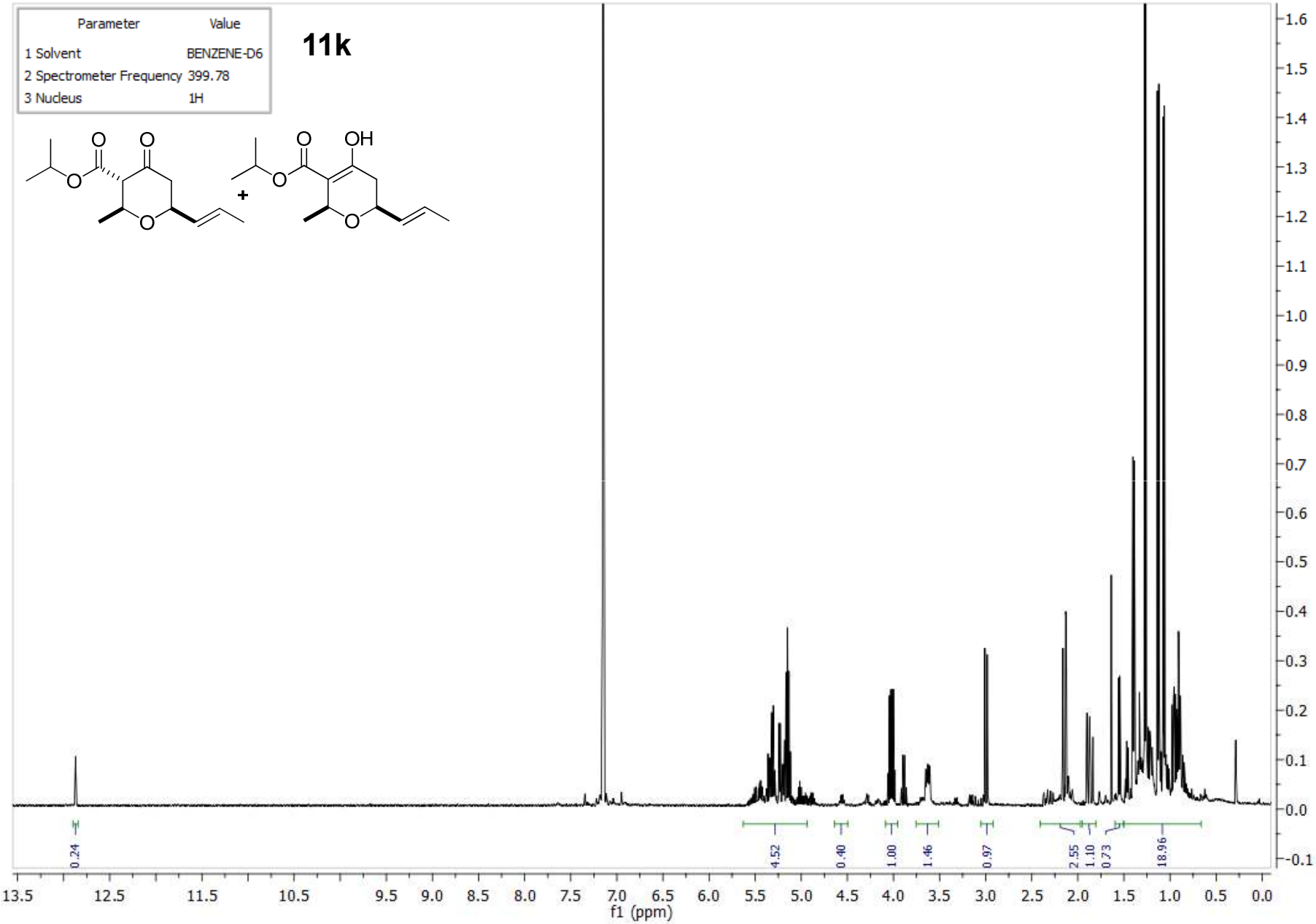

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
11f
OBn
OH
OBn
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

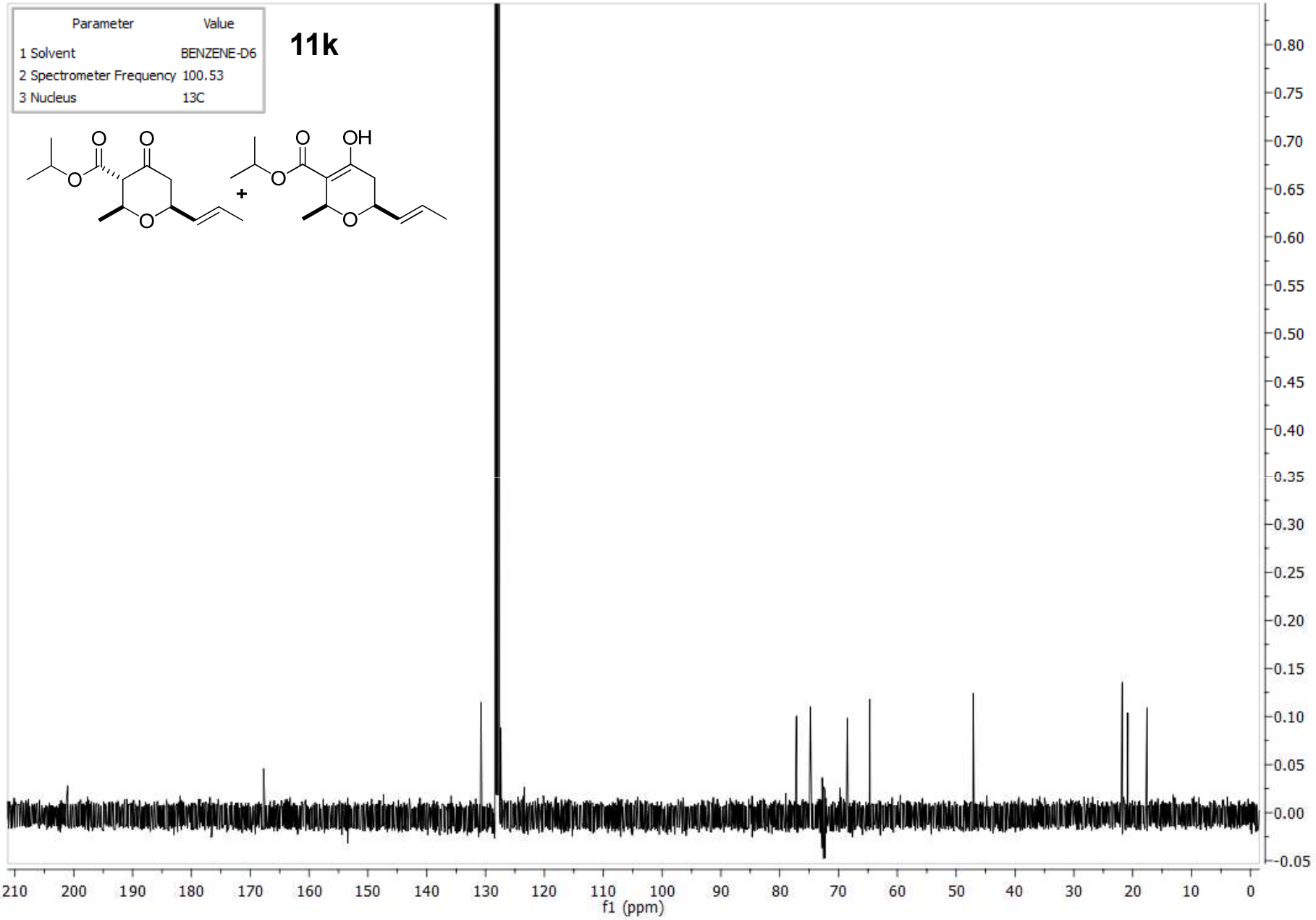

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
11g
OBn
OH
f1 (ppm)

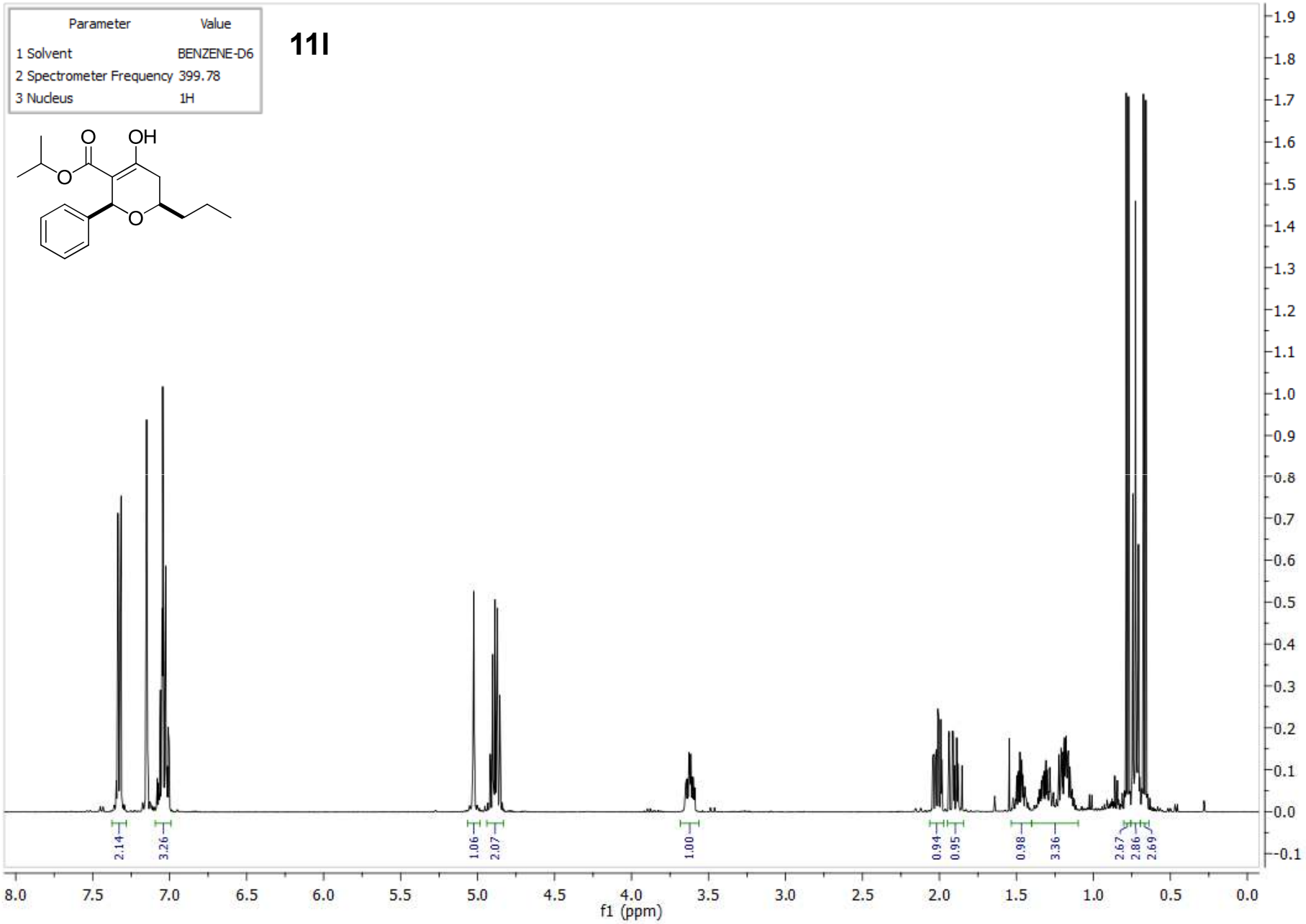

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
11g
O
O
O
OBn
+
O
OH
O
OBn
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

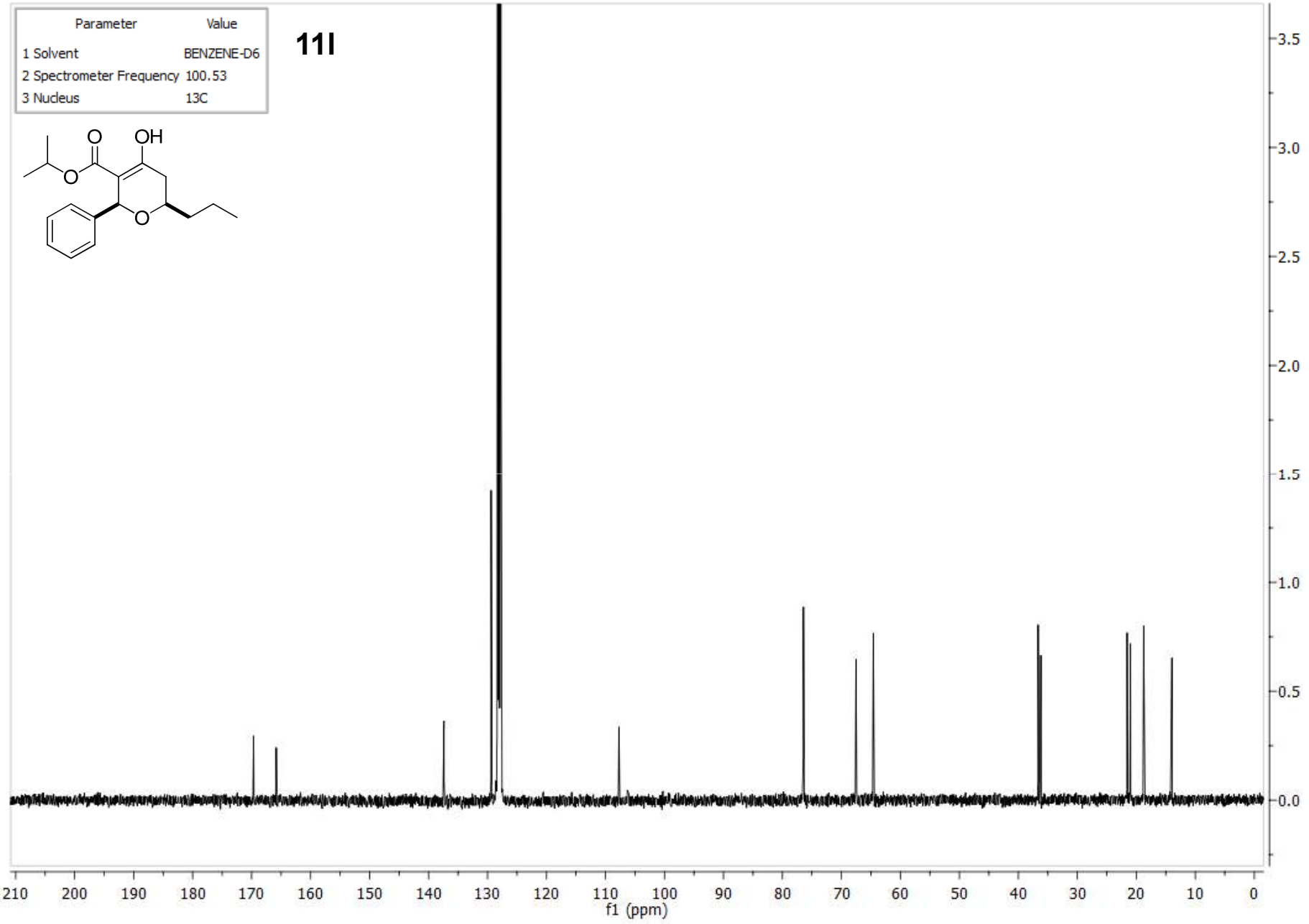

Parameter Value
1 Solvent CHLOROFORM-D
2 Spectrometer Frequency 399.78
3 Nucleus 1H
11h
O
O
O
O
6.11
1.27
1.02
1.00
1.01
0.96
1.20
3.45
8.20
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

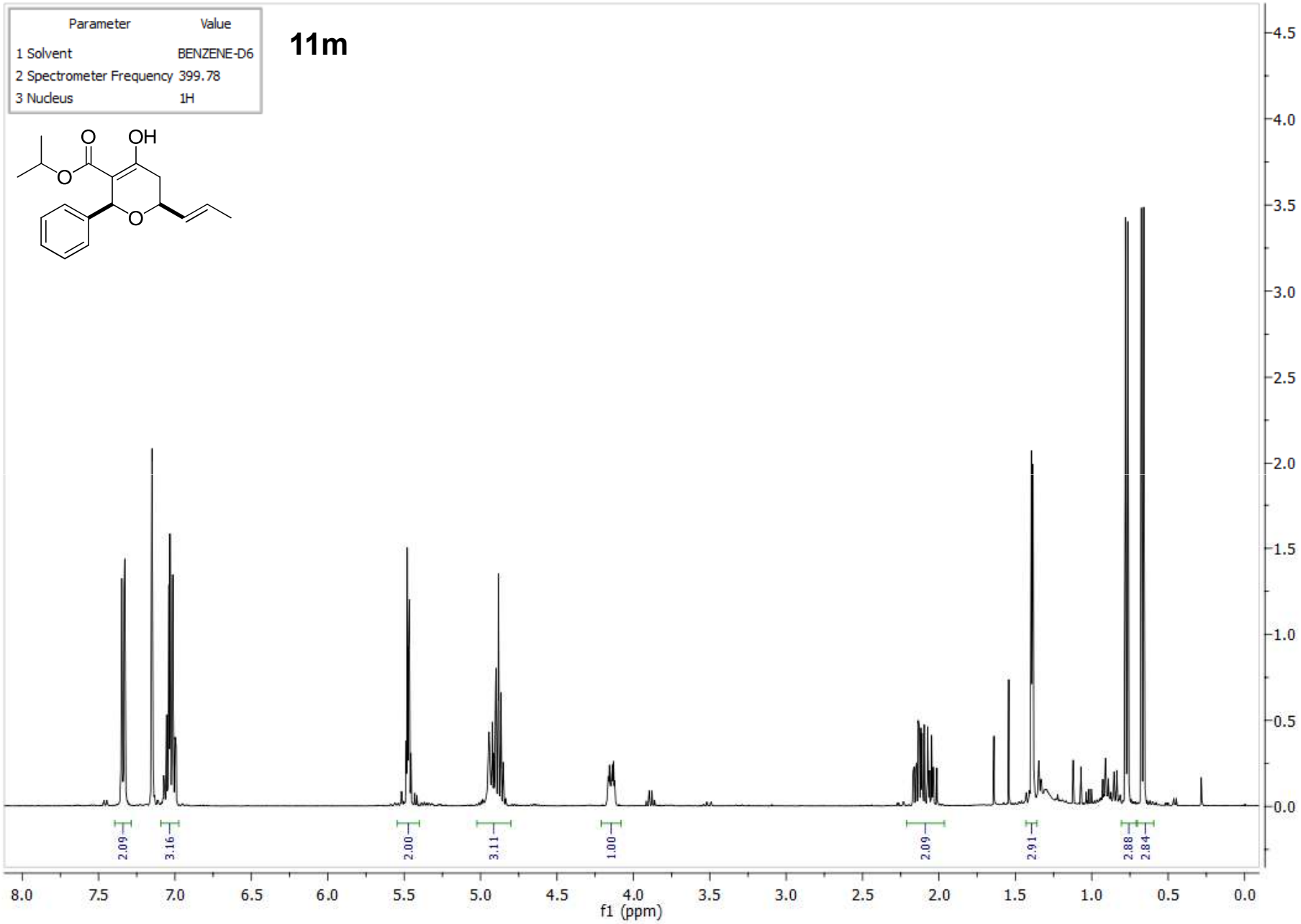

Parameter Value
1 Solvent CHLOROFORM-D
2 Spectrometer Frequency 100.53
3 Nucleus 13C
11h
O
O
O
O
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

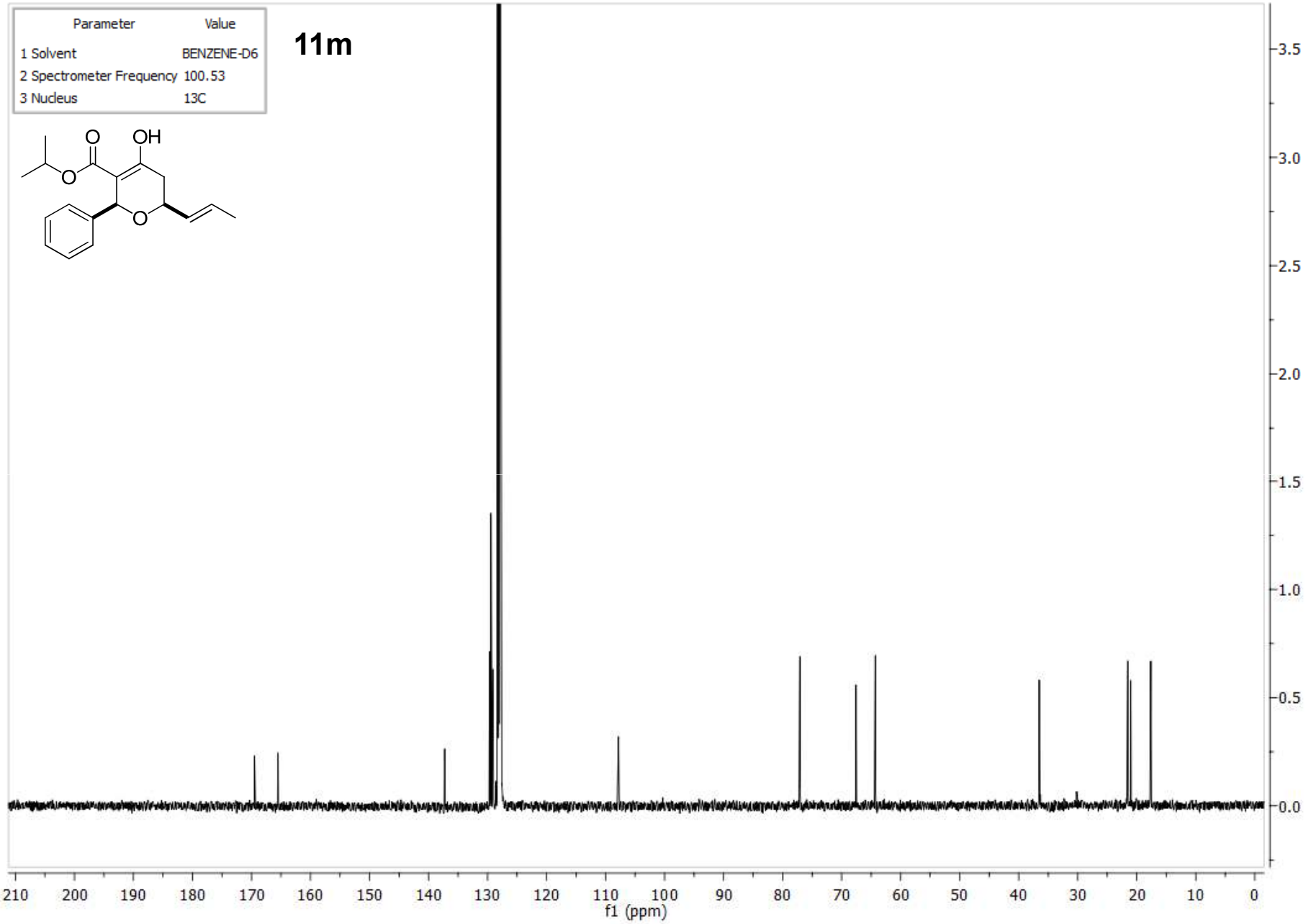

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
11k
O
O
O
O
OH
+
0.24
4.52
0.40
1.00
1.46
0.97
2.55
1.10
0.73
18.96
13.5 12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
1.6 1.5 1.4 1.3 1.2 1.1 1.0 0.9 0.8 0.7 0.6 0.5 0.4 0.3 0.2 0.1 0.0 -0.1

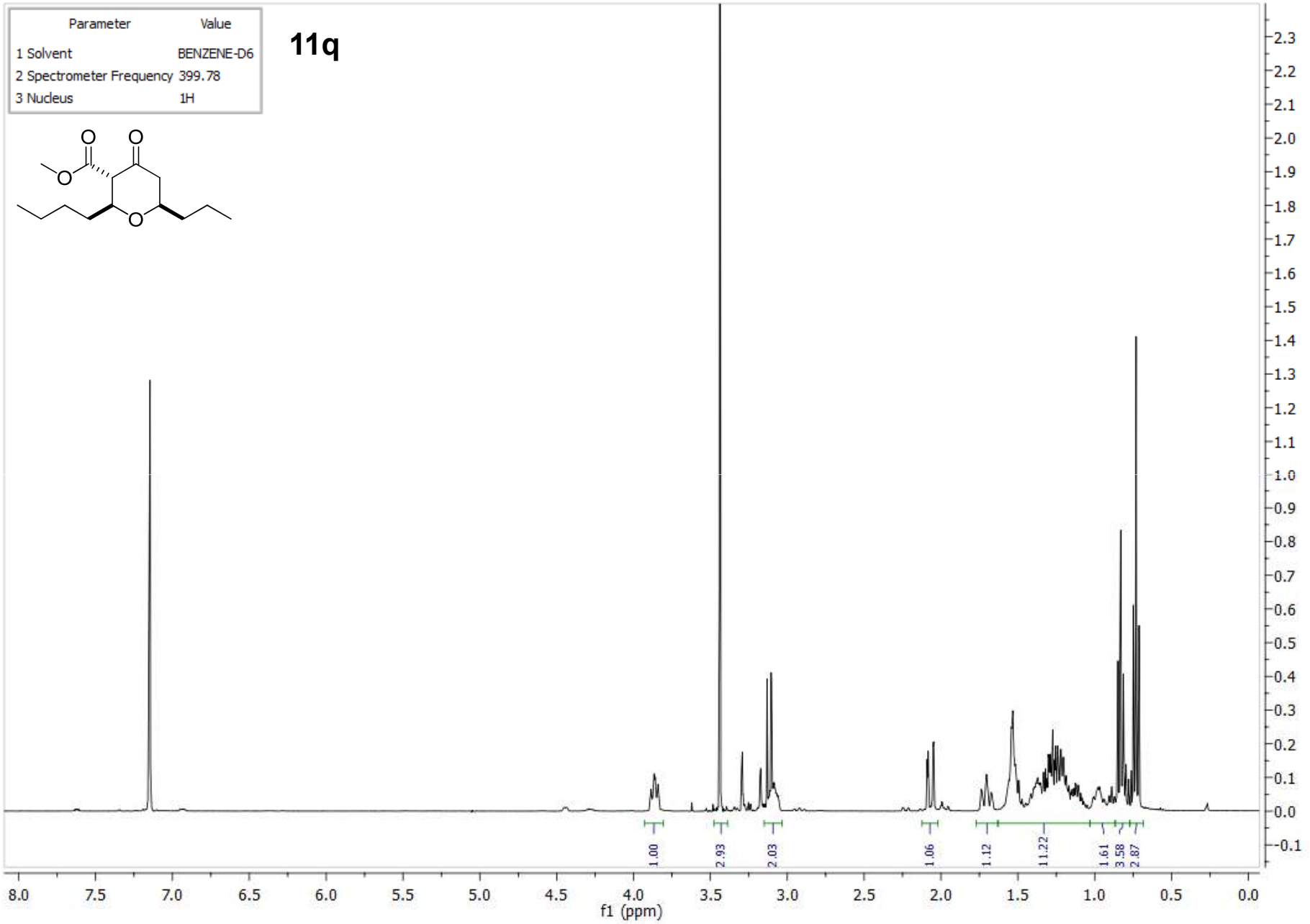

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
11k
O
O
O
O
O
OH
+
0.80
0.75
0.70
0.65
0.60
0.55
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
-0.05
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

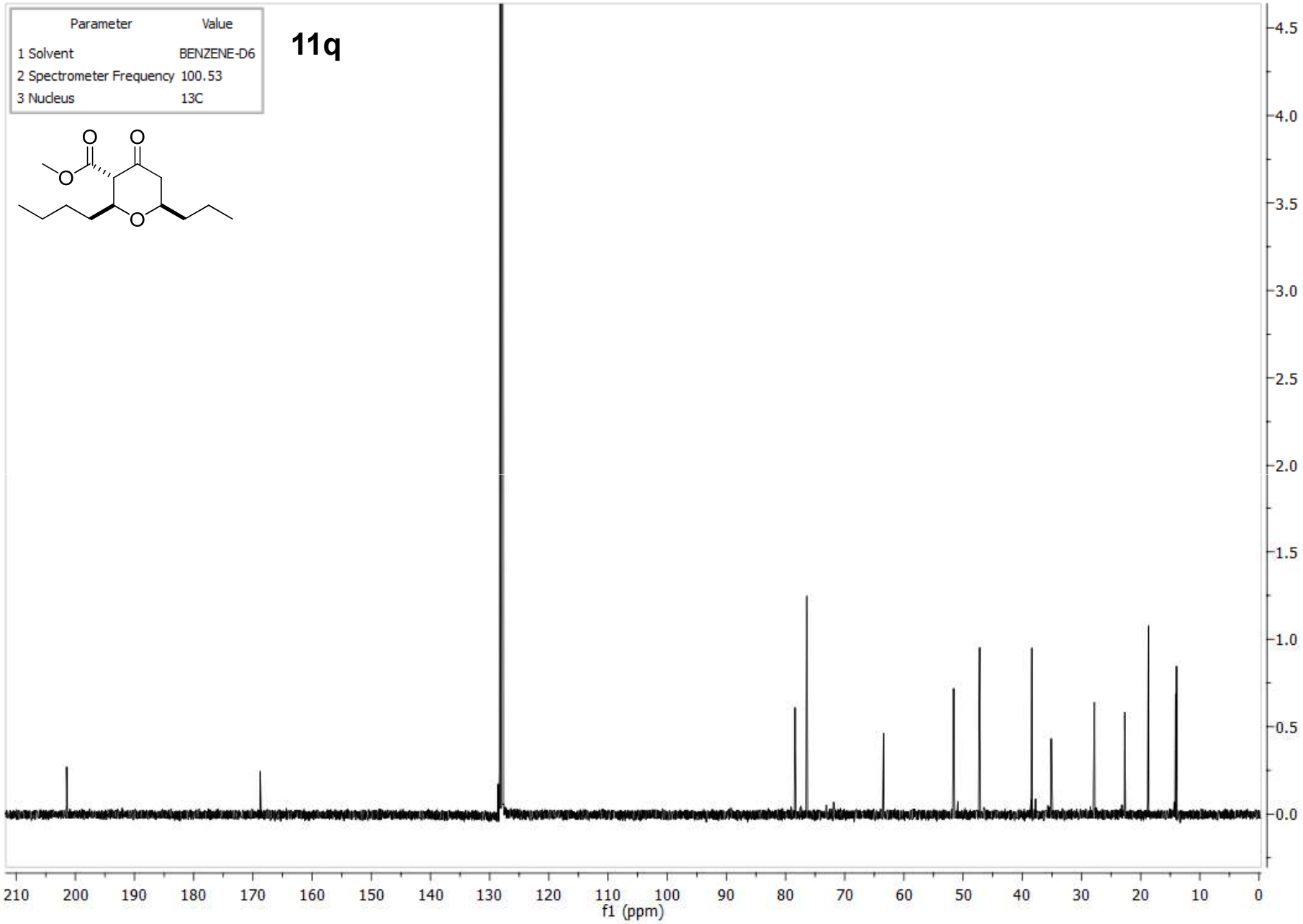

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
11l
O
OH
O
O
2.14
3.26
1.06
2.07
1.00
0.94
0.95
0.98
3.36
2.67
2.86
2.69
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
1.9
1.8
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
-0.1

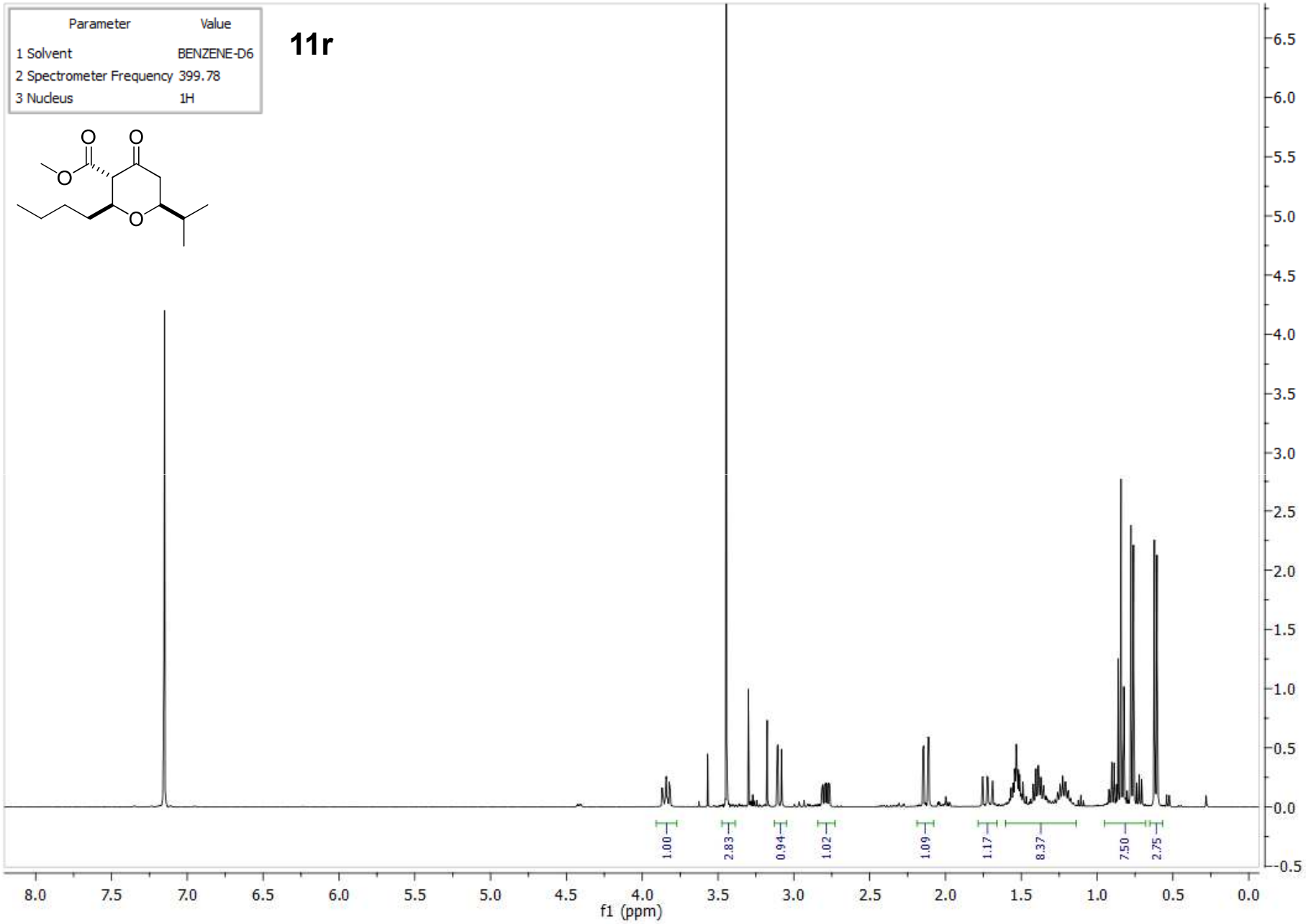

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
11l
O
OH
O
O
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

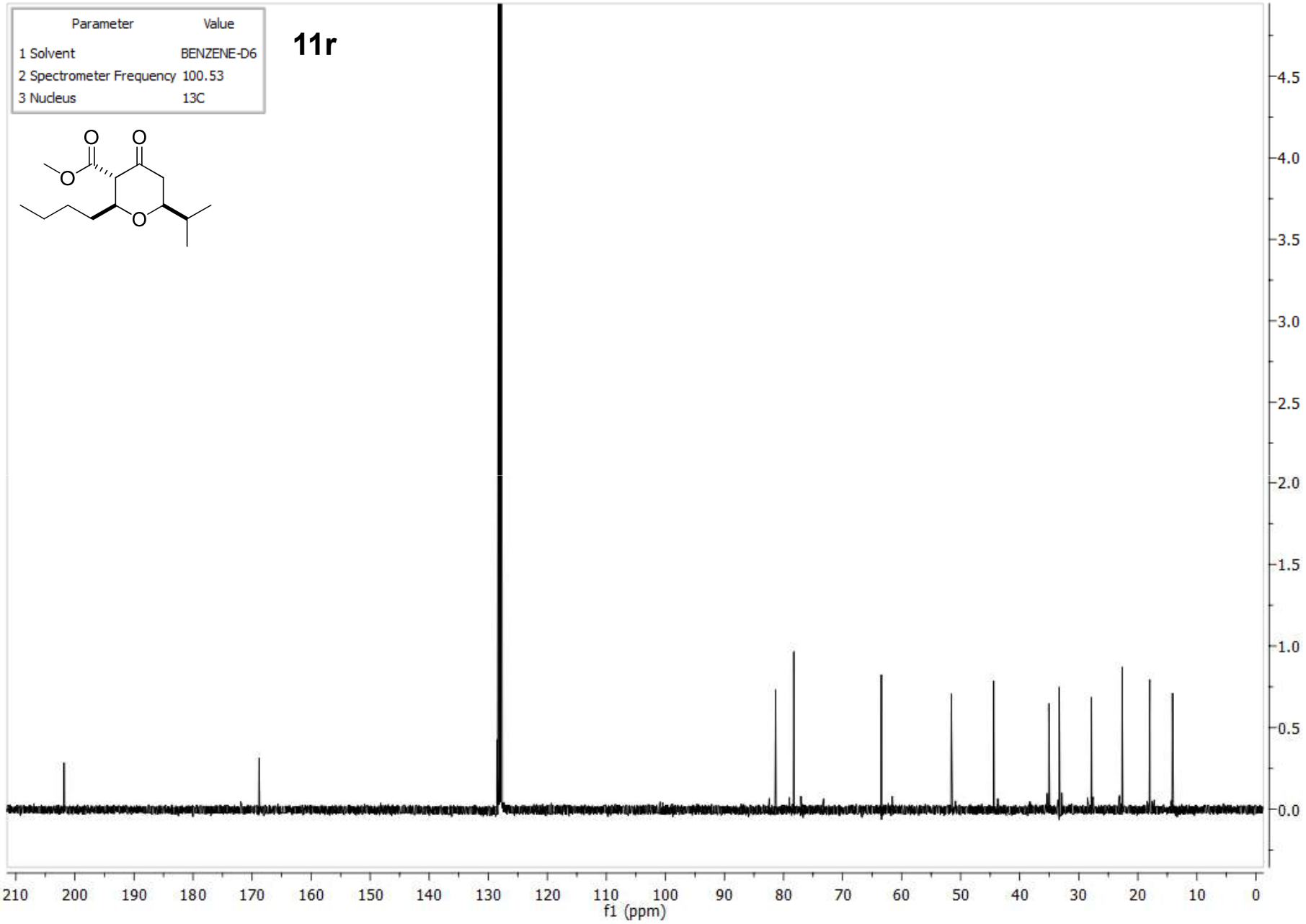

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
11m
O
OH
O
O
2.09
3.16
2.00
3.11
1.00
2.09
2.91
2.88
2.84
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

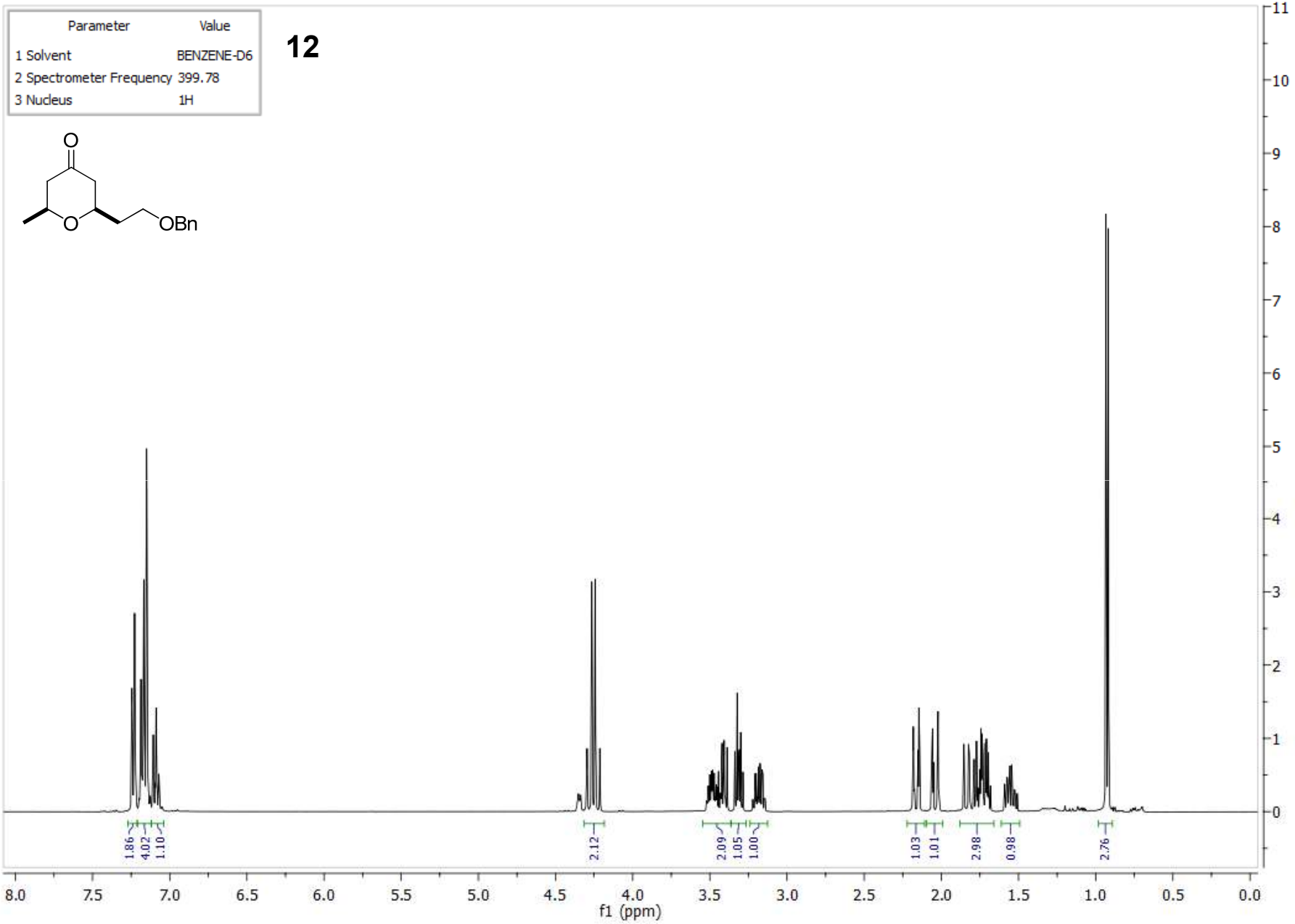

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
11m
O
OH
O
O
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

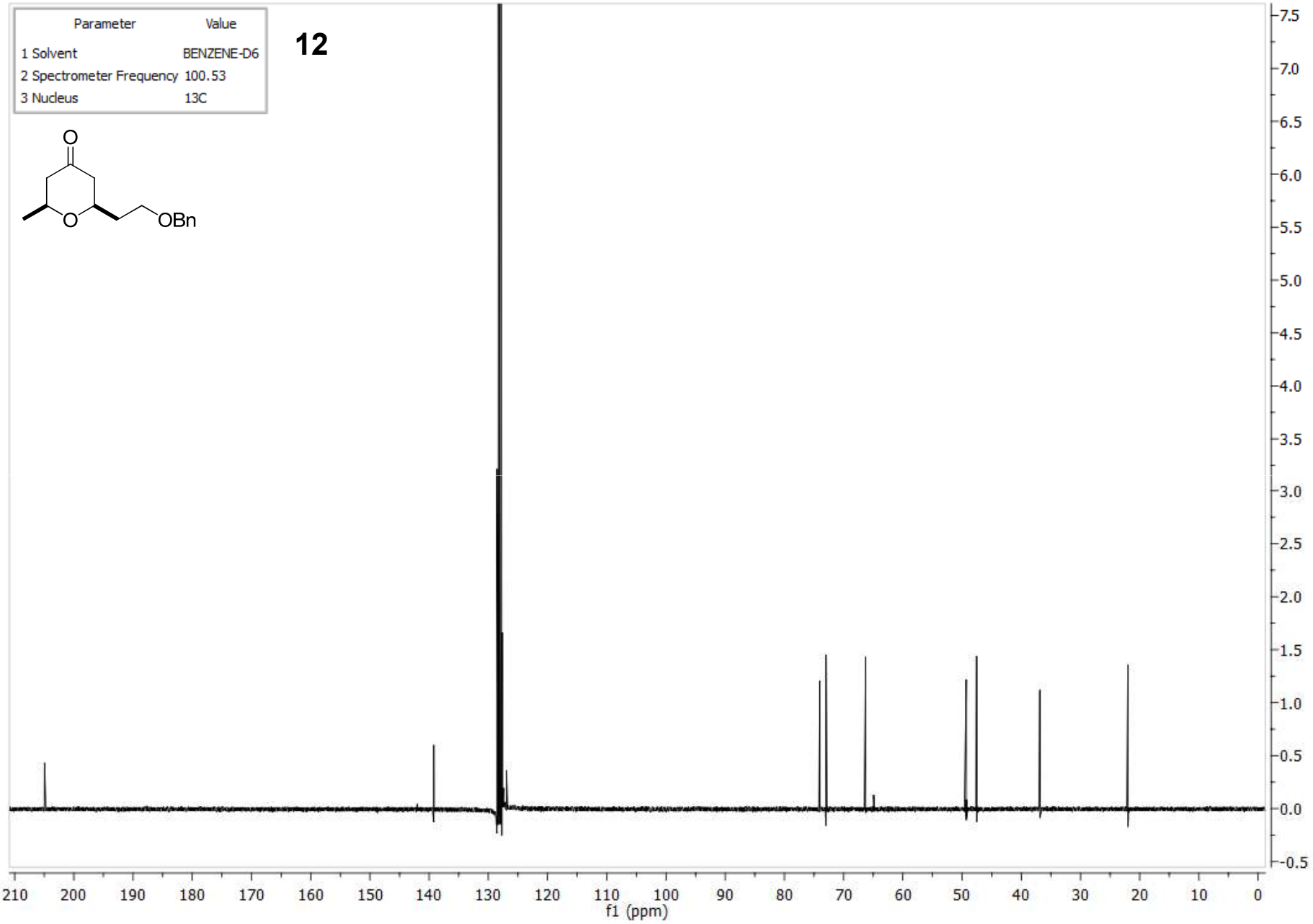

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
11q
1.00
2.93
2.03
1.06
1.12
11.22
1.61
3.58
2.87
f1 (ppm)

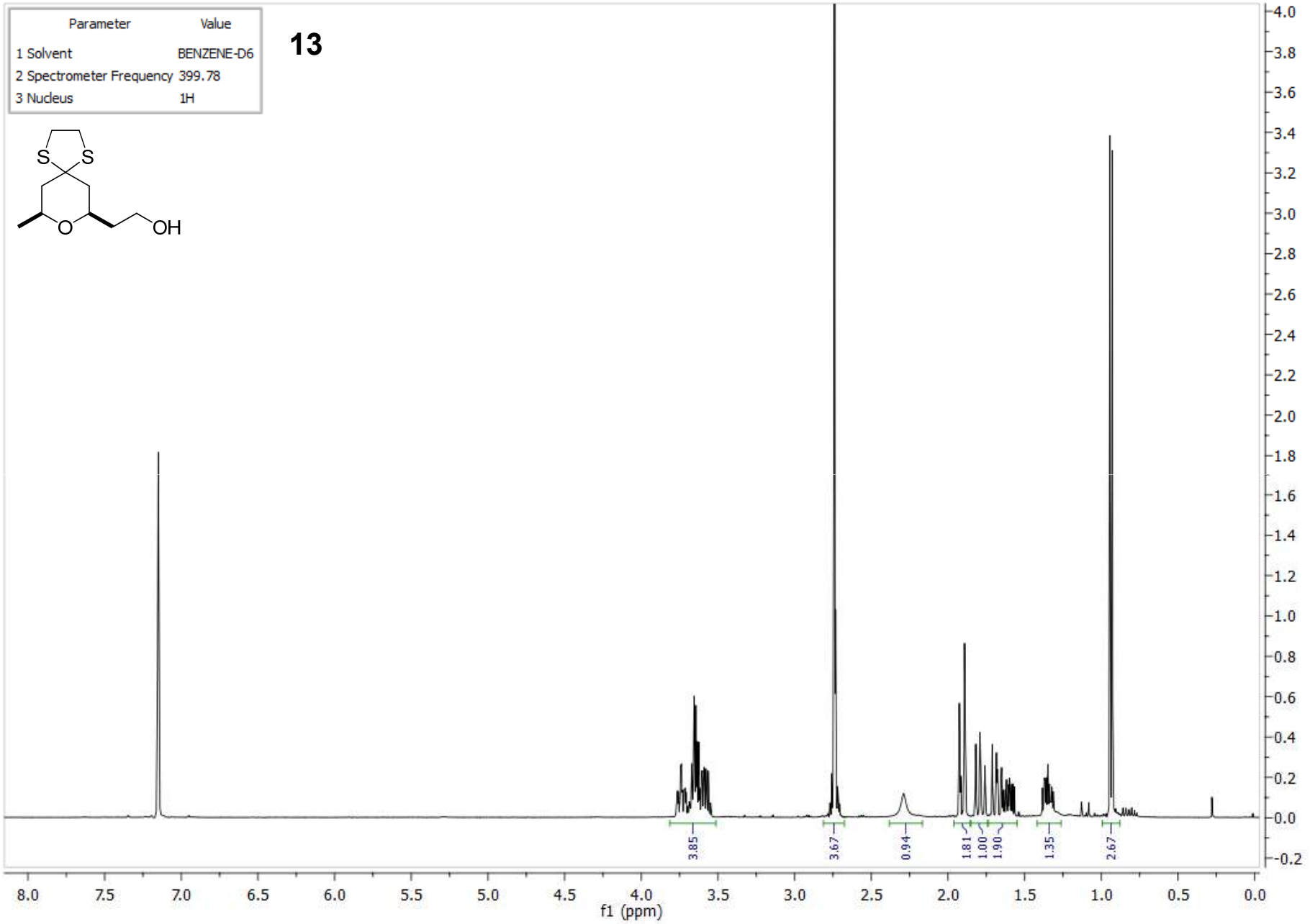

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
11q
O
O
O
O
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

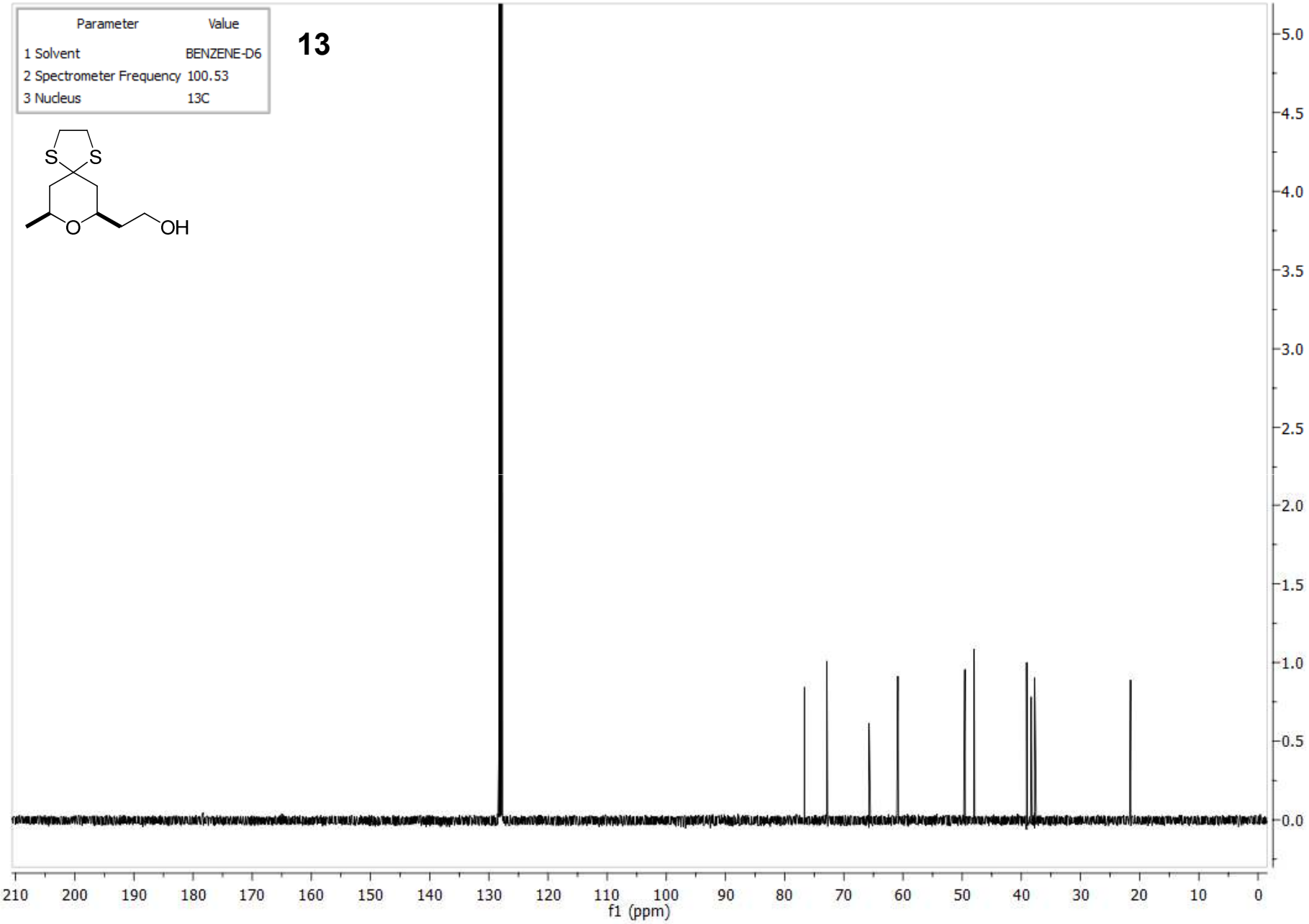

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
11r
1.00
2.83
0.94
1.02
1.09
1.17
8.37
7.50
2.75
f1 (ppm)

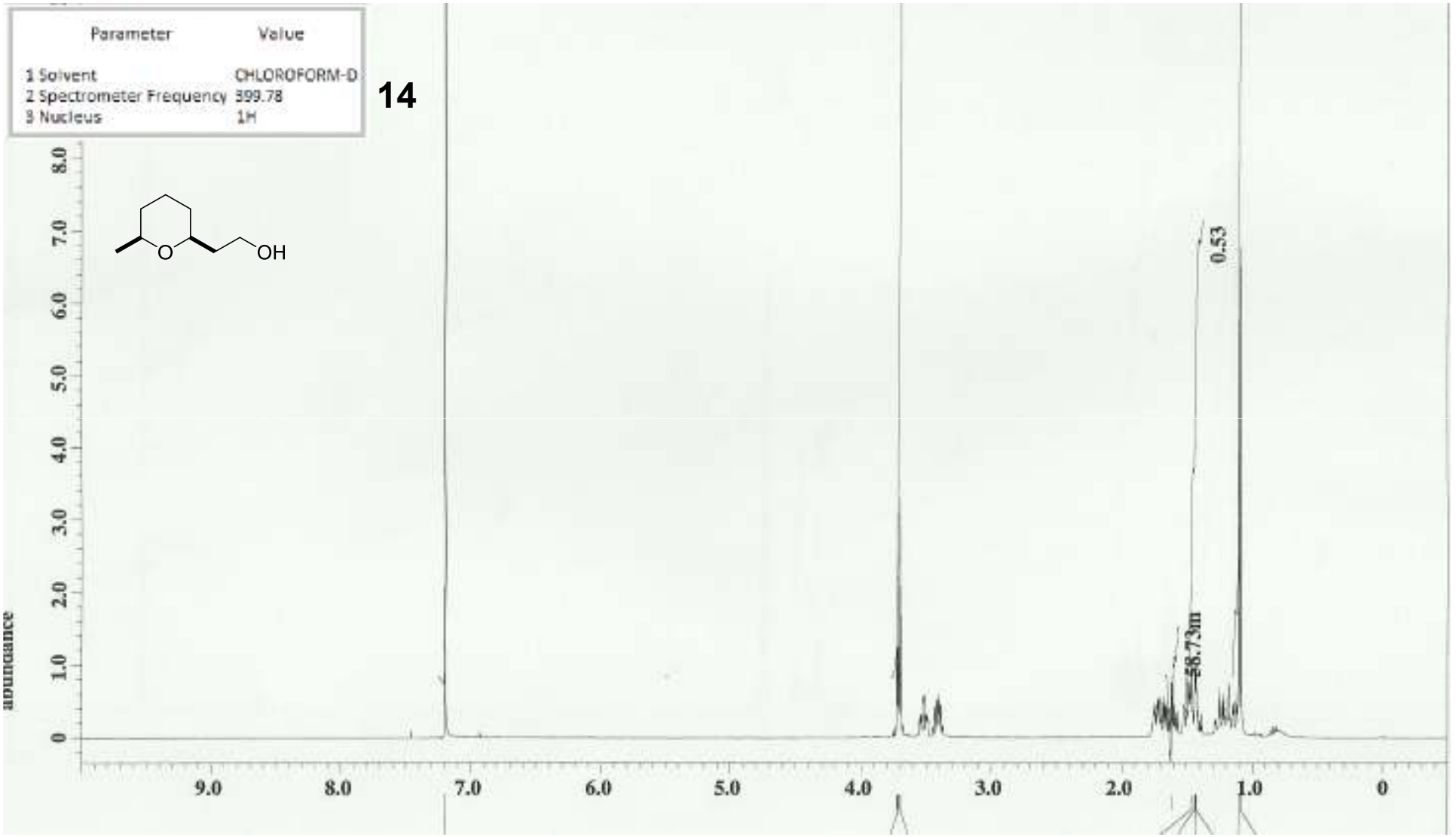

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
11r
O
O
O
O
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

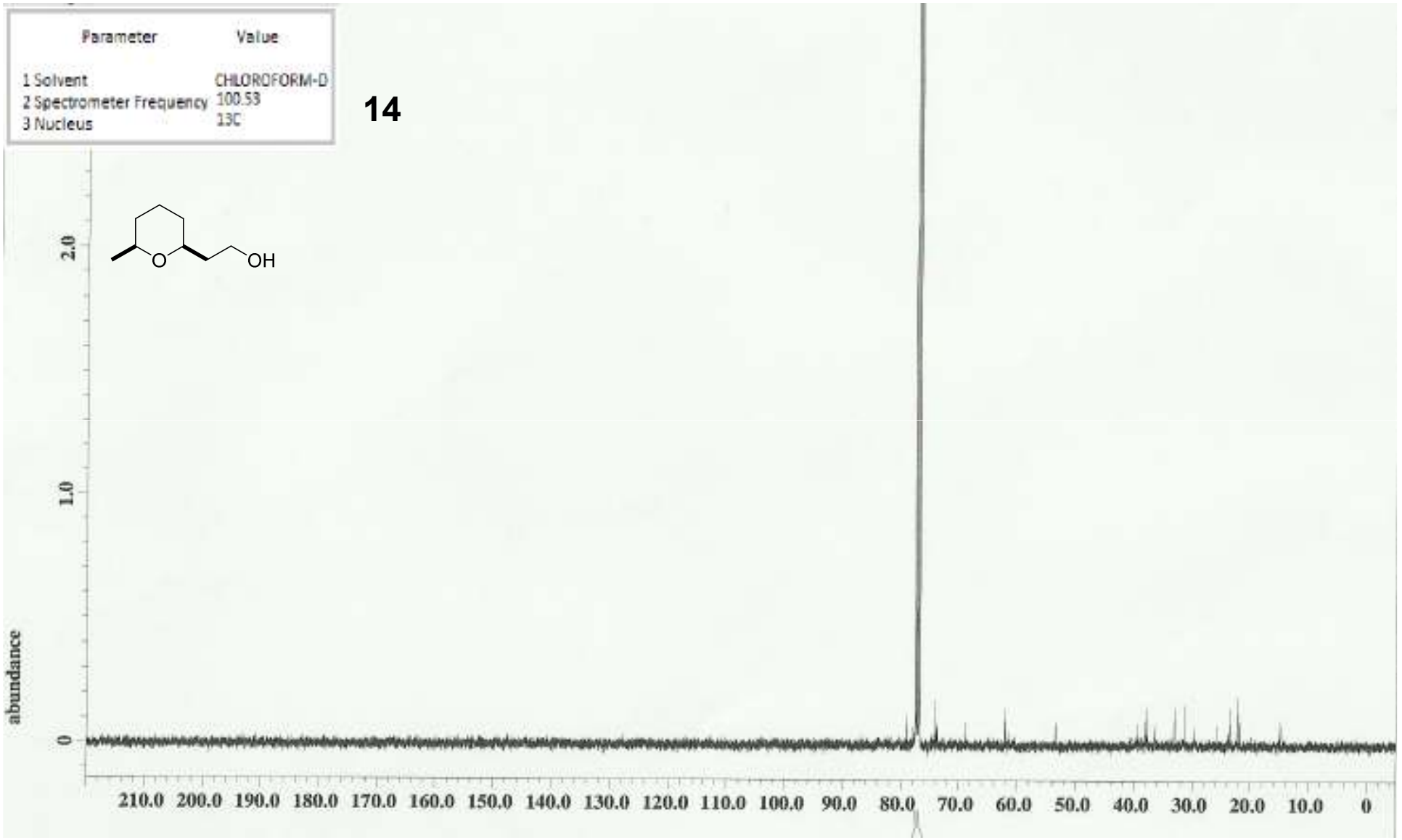

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
12
O
O
OBn
1.86
4.02
1.10
2.12
2.09
1.05
1.00
1.03
1.01
2.98
0.98
2.76
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
11
10
9
8
7
6
5
4
3
2
1
0


Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
12
O
O
OBn
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
13
S
S
O
OH
3.85
3.67
0.94
1.81
1.00
1.90
1.35
2.67
f1 (ppm)

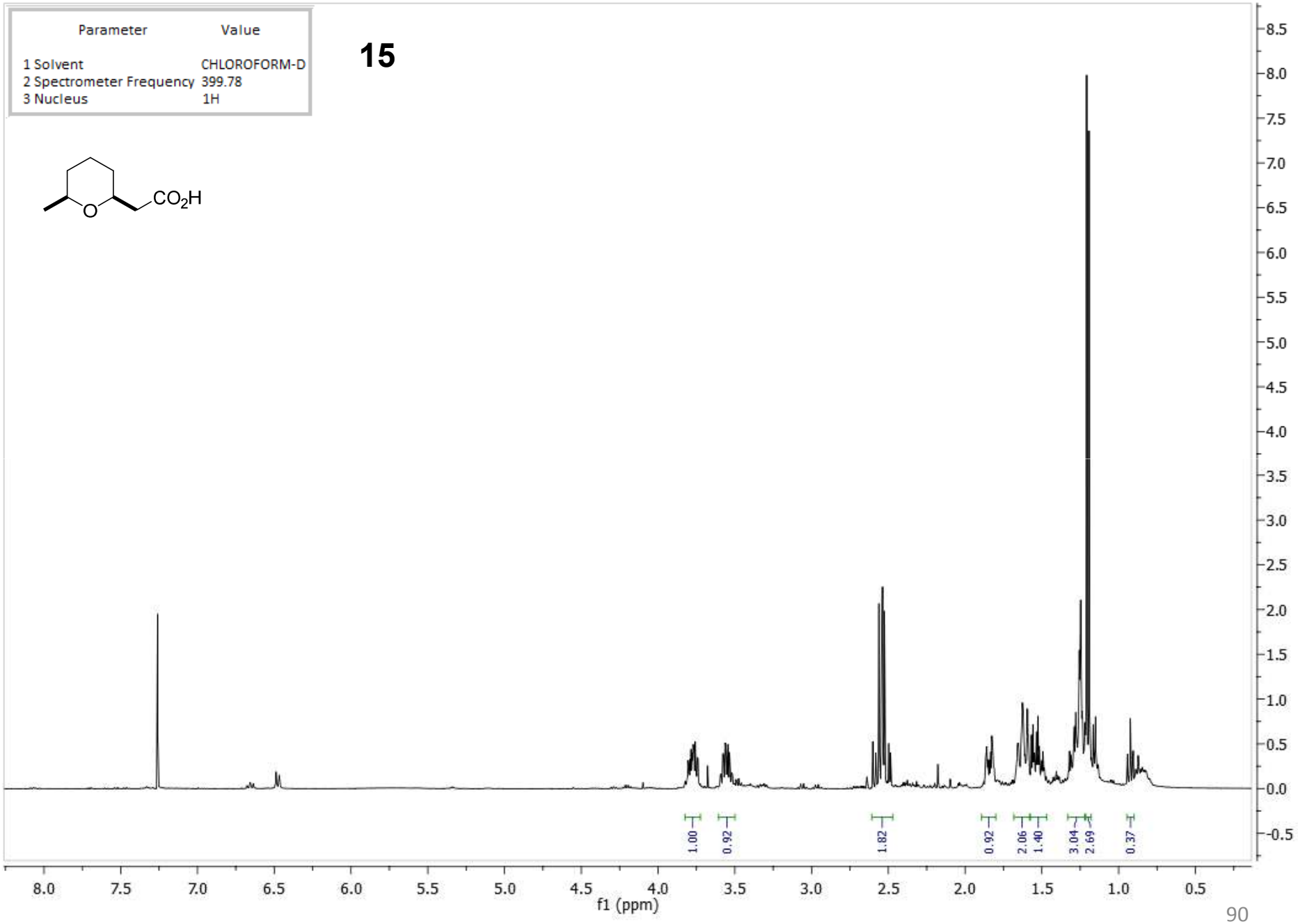

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
13
S
S
O
OH
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Parameter Value
1 Solvent CHLOROFORM-D
2 Spectrometer Frequency 399.78
3 Nucleus 1H
14
O
OH
0.53
8.73m
abundance
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0


Parameter Value
1 Solvent CHLOROFORM-D
2 Spectrometer Frequency 100.53
3 Nucleus 13C
14
O
OH
abundance
2.0
1.0
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

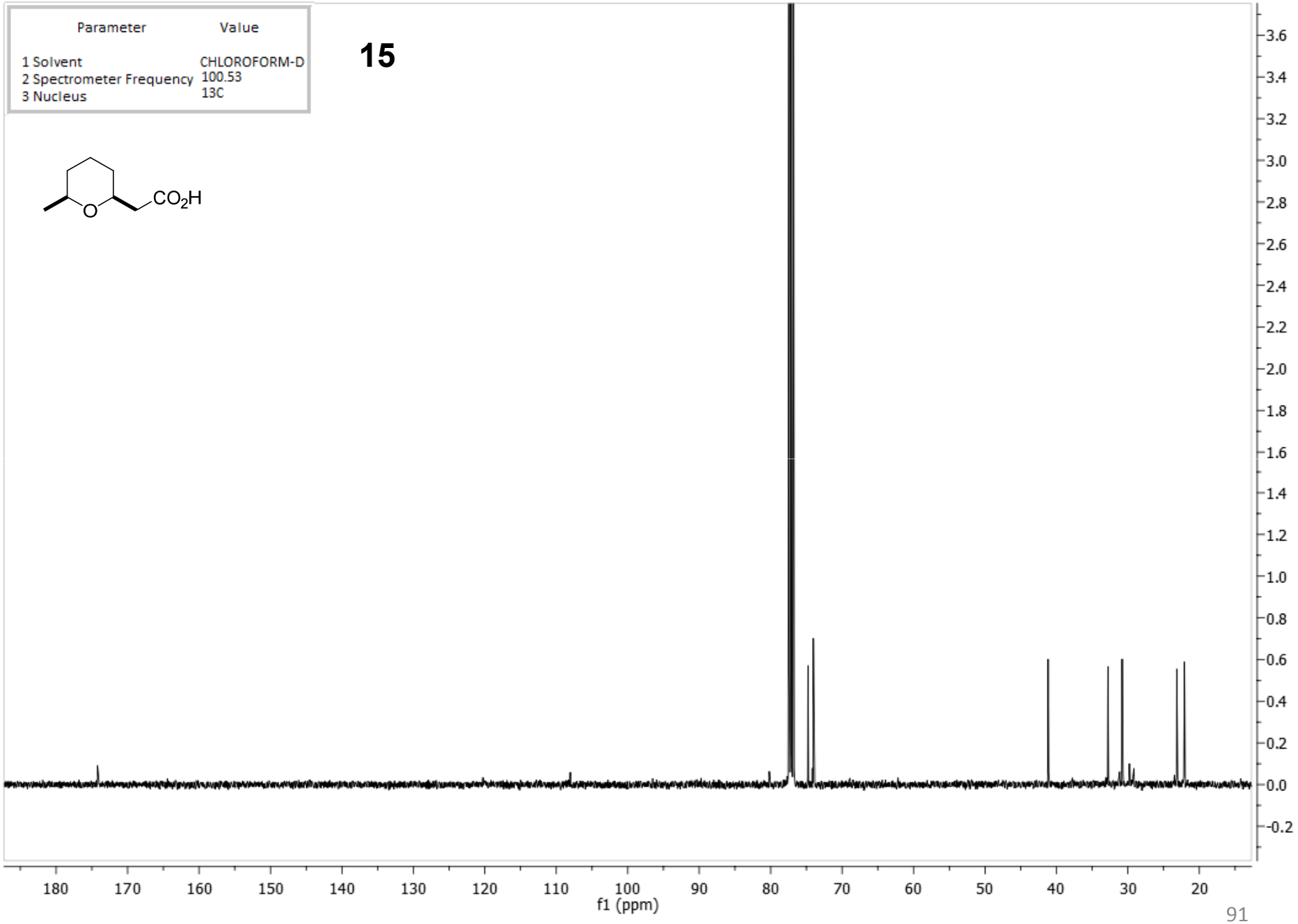
Parameter	Value
1 Solvent	CHLOROFORM-D
2 Spectrometer Frequency	399.78
3 Nucleus	1H

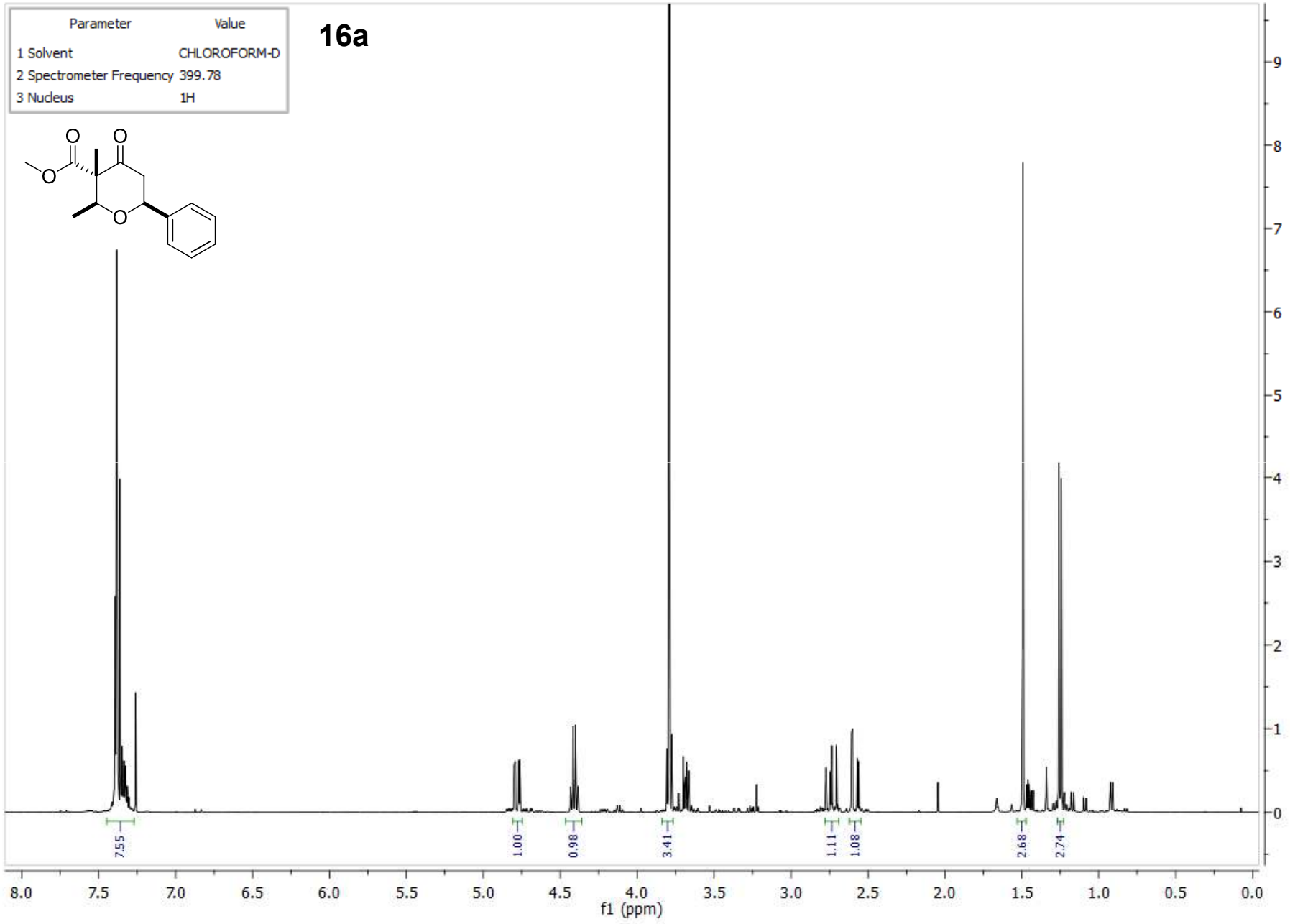
15



Parameter	Value
1 Solvent	CHLOROFORM-D
2 Spectrometer Frequency	100.53
3 Nucleus	13C

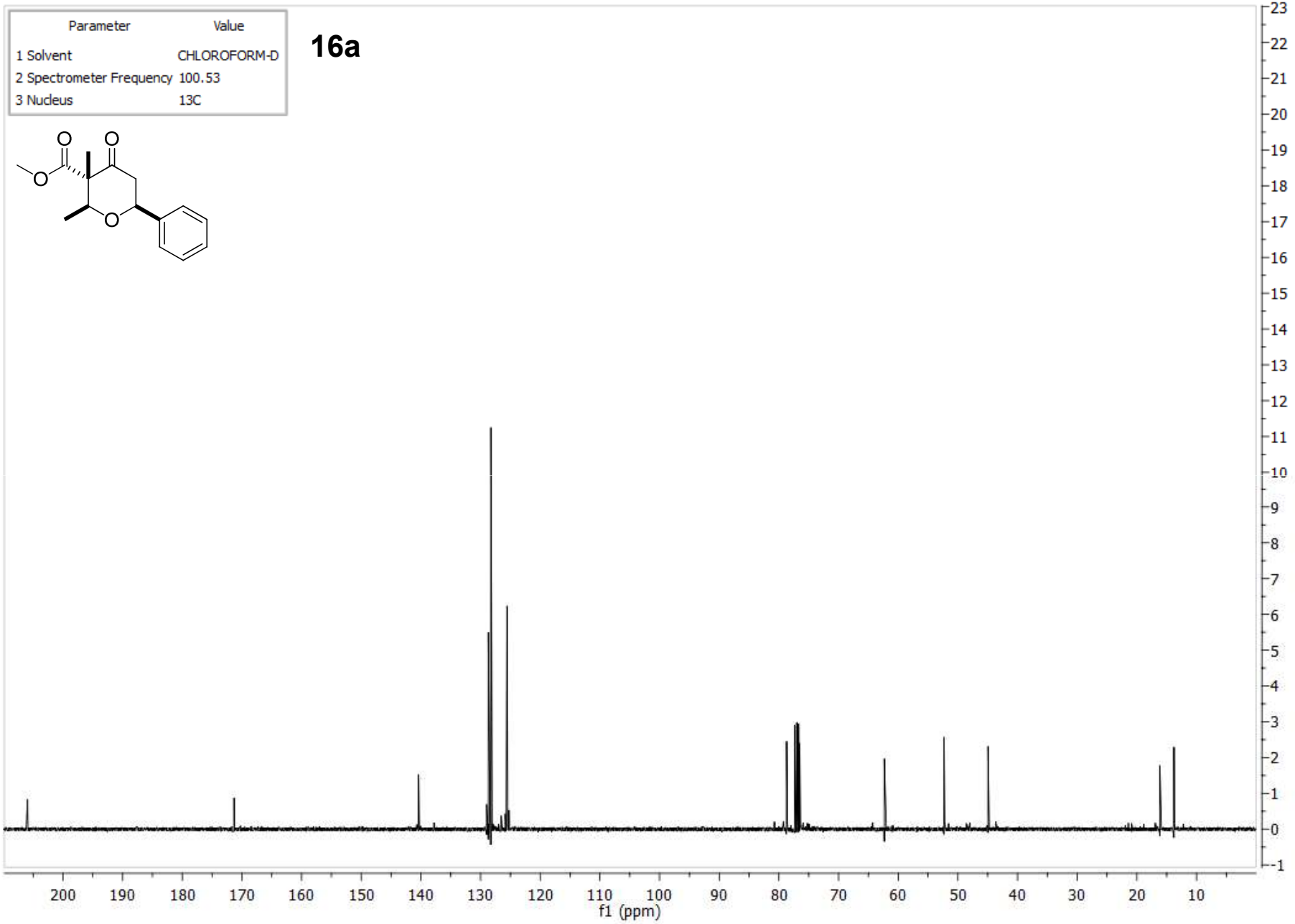
15

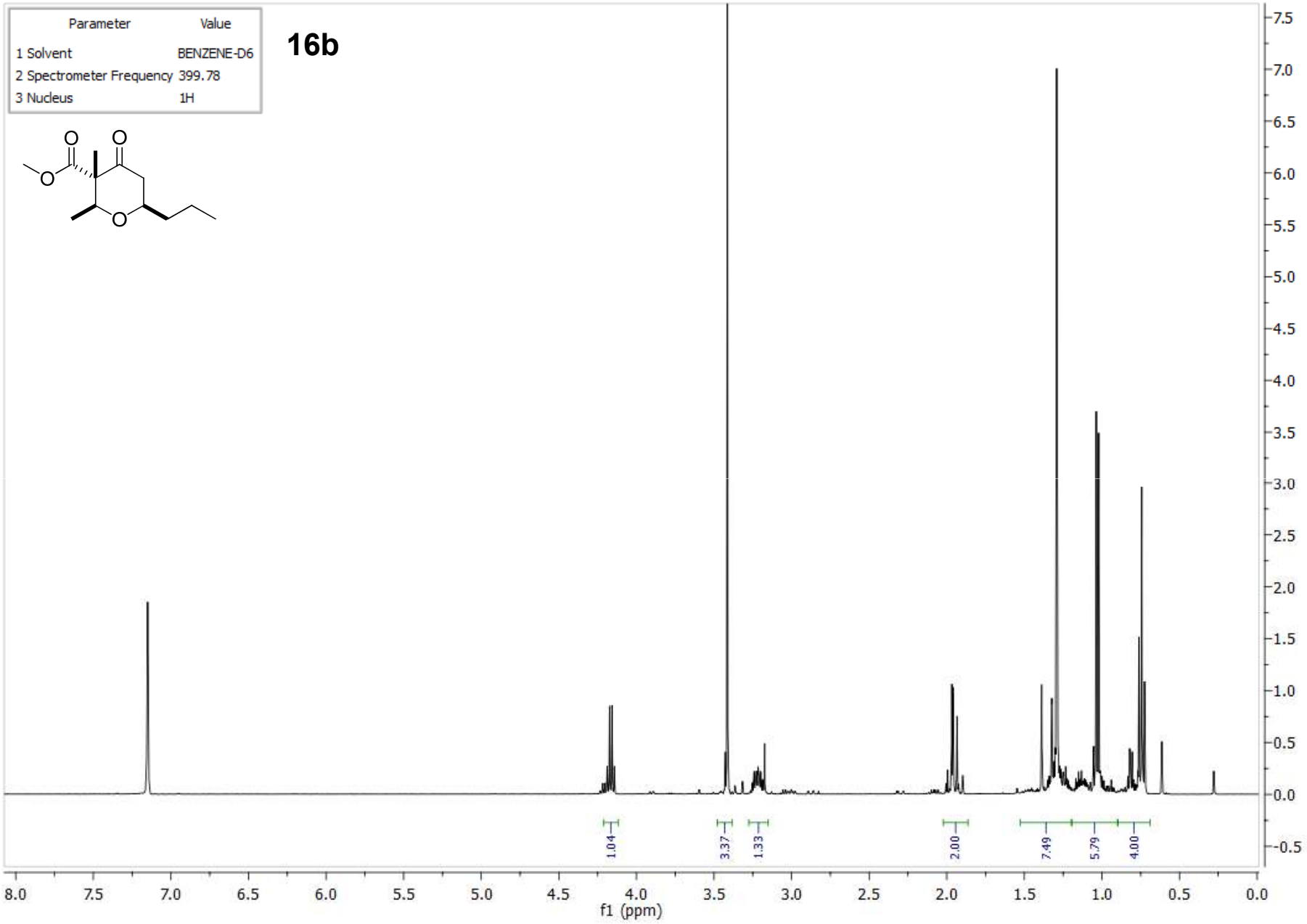


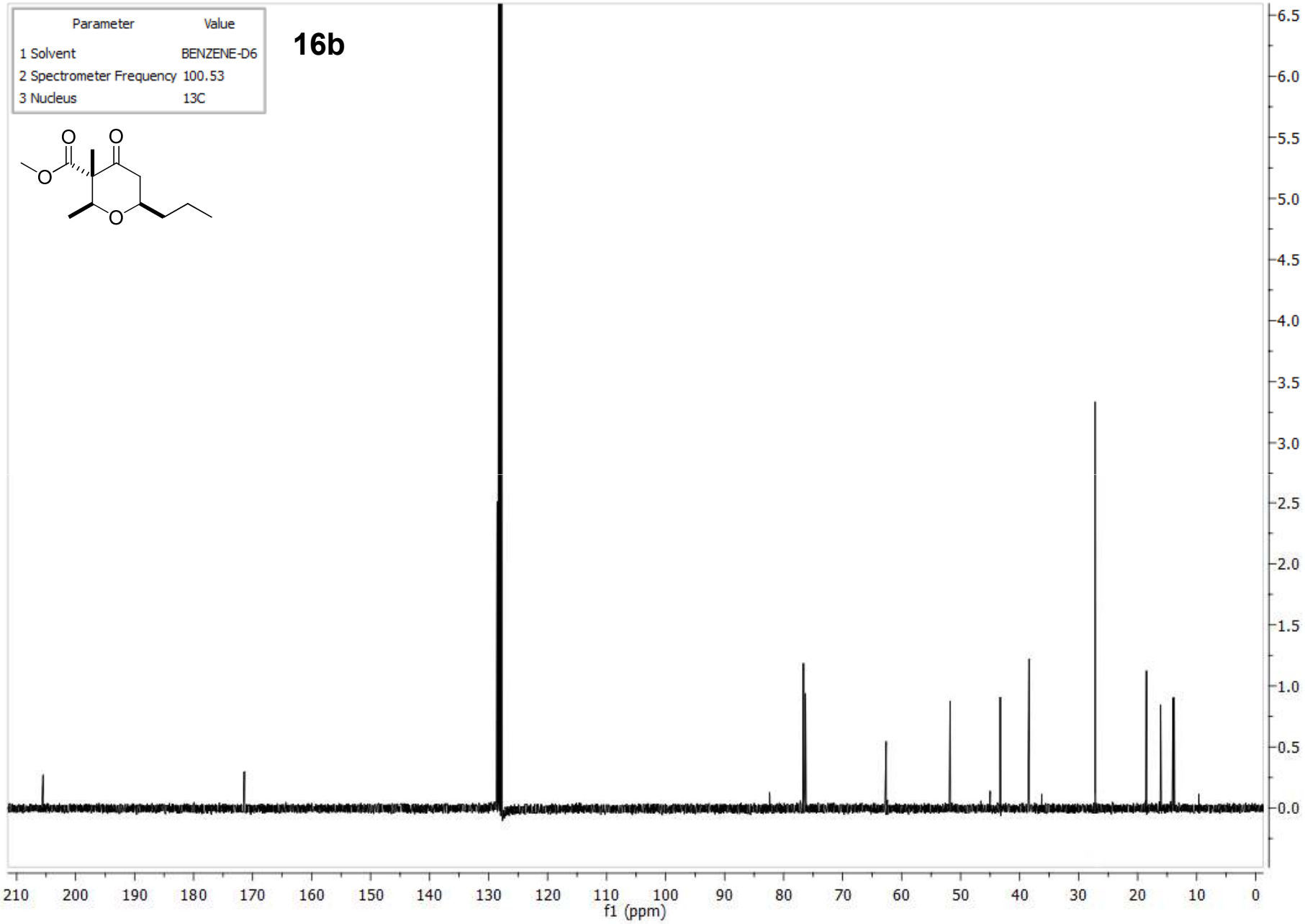

Parameter Value
1 Solvent CHLOROFORM-D
2 Spectrometer Frequency 399.78
3 Nucleus 1H
16a
7.55
1.00
0.98
3.41
1.11
1.08
2.68
2.74
f1 (ppm)

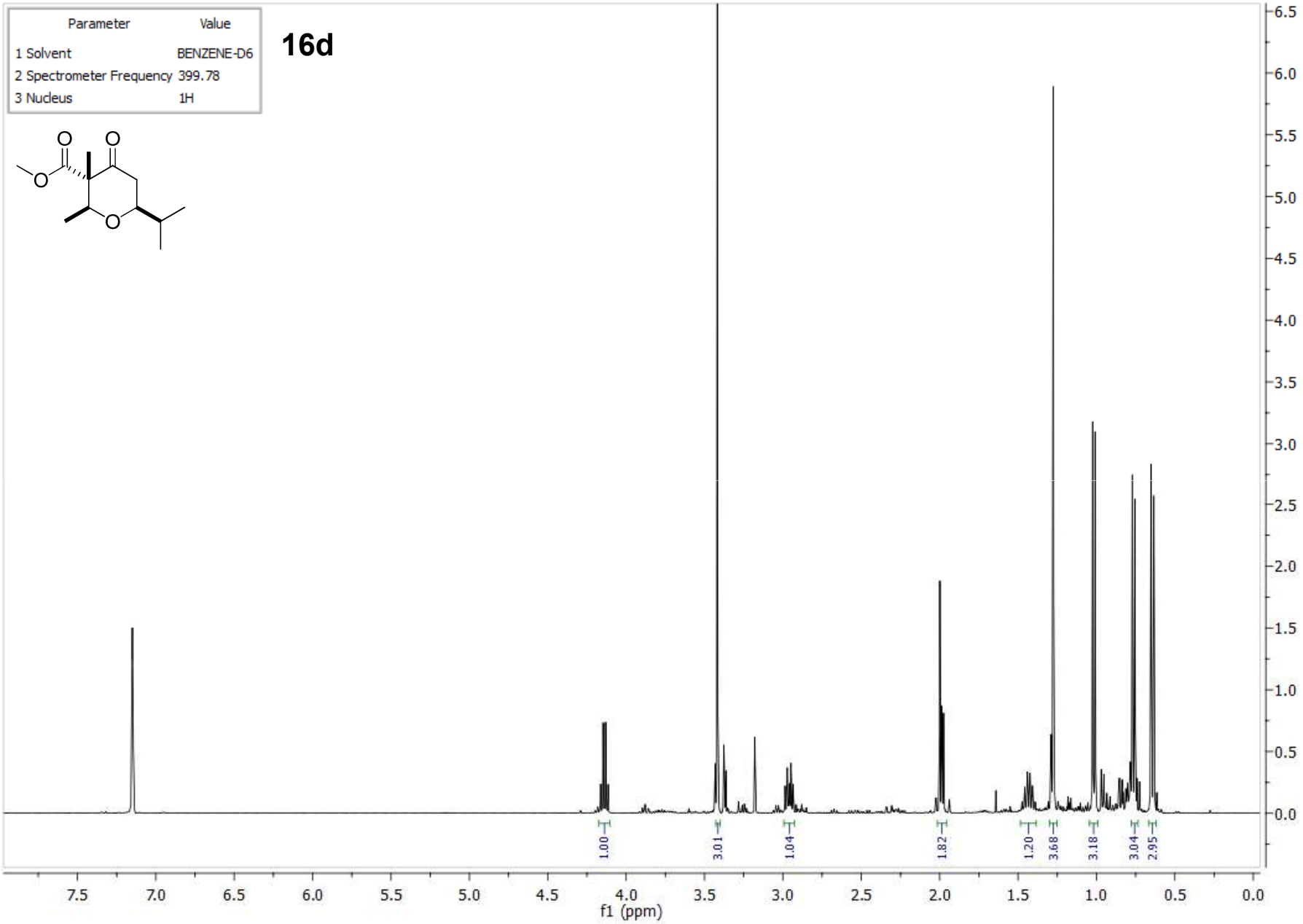
Parameter	Value
1 Solvent	CHLOROFORM-D
2 Spectrometer Frequency	100.53
3 Nucleus	13C

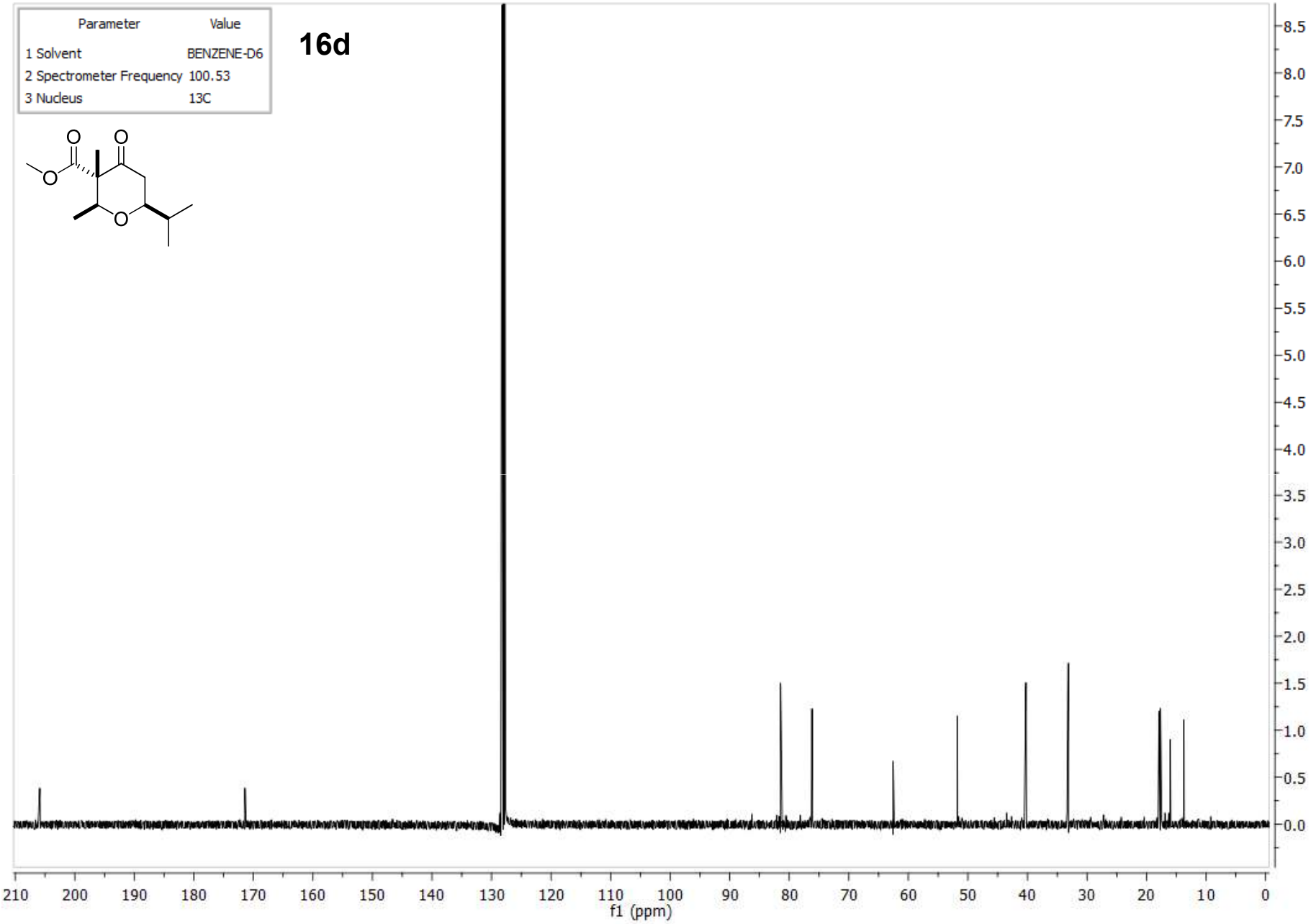
16a

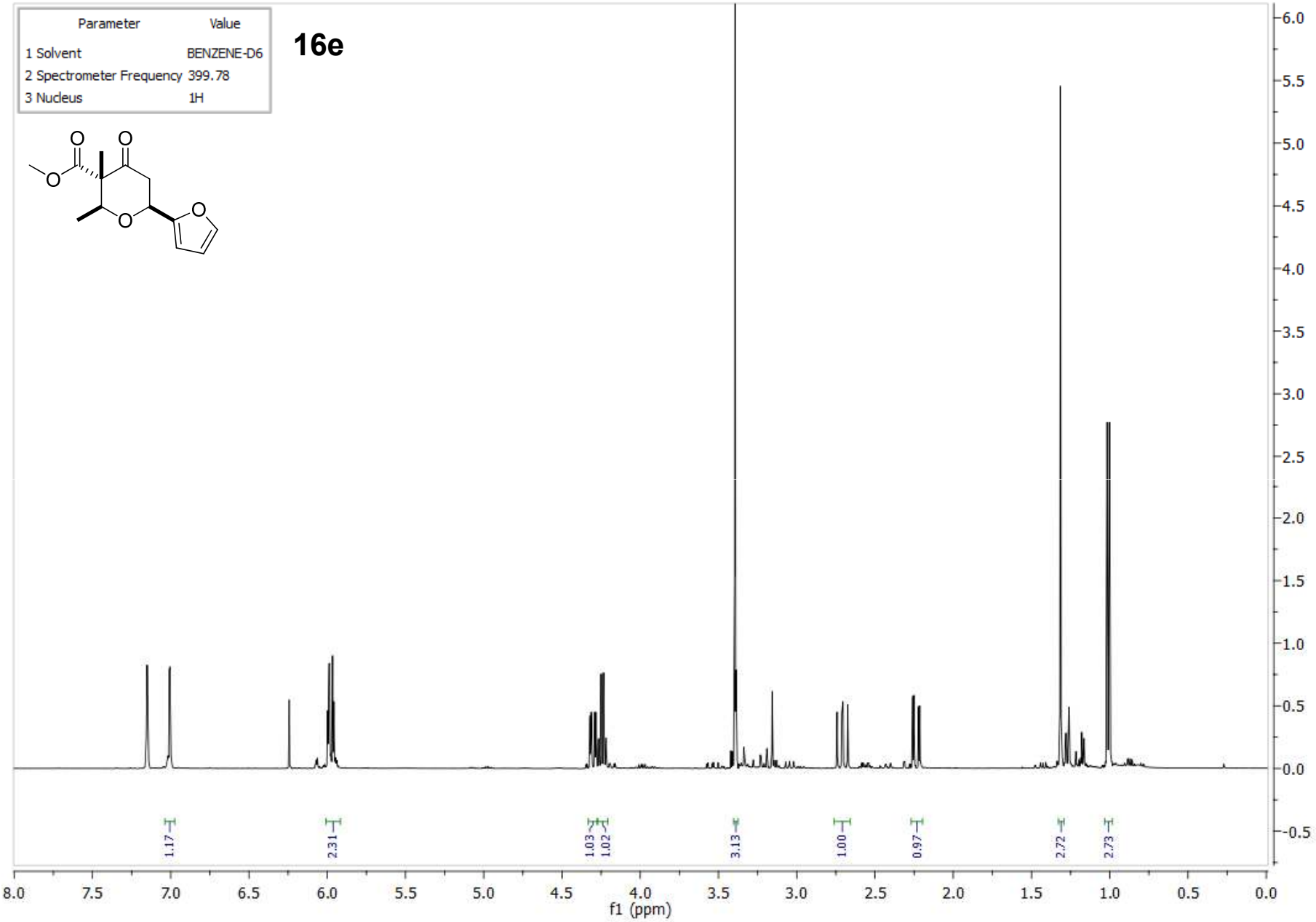


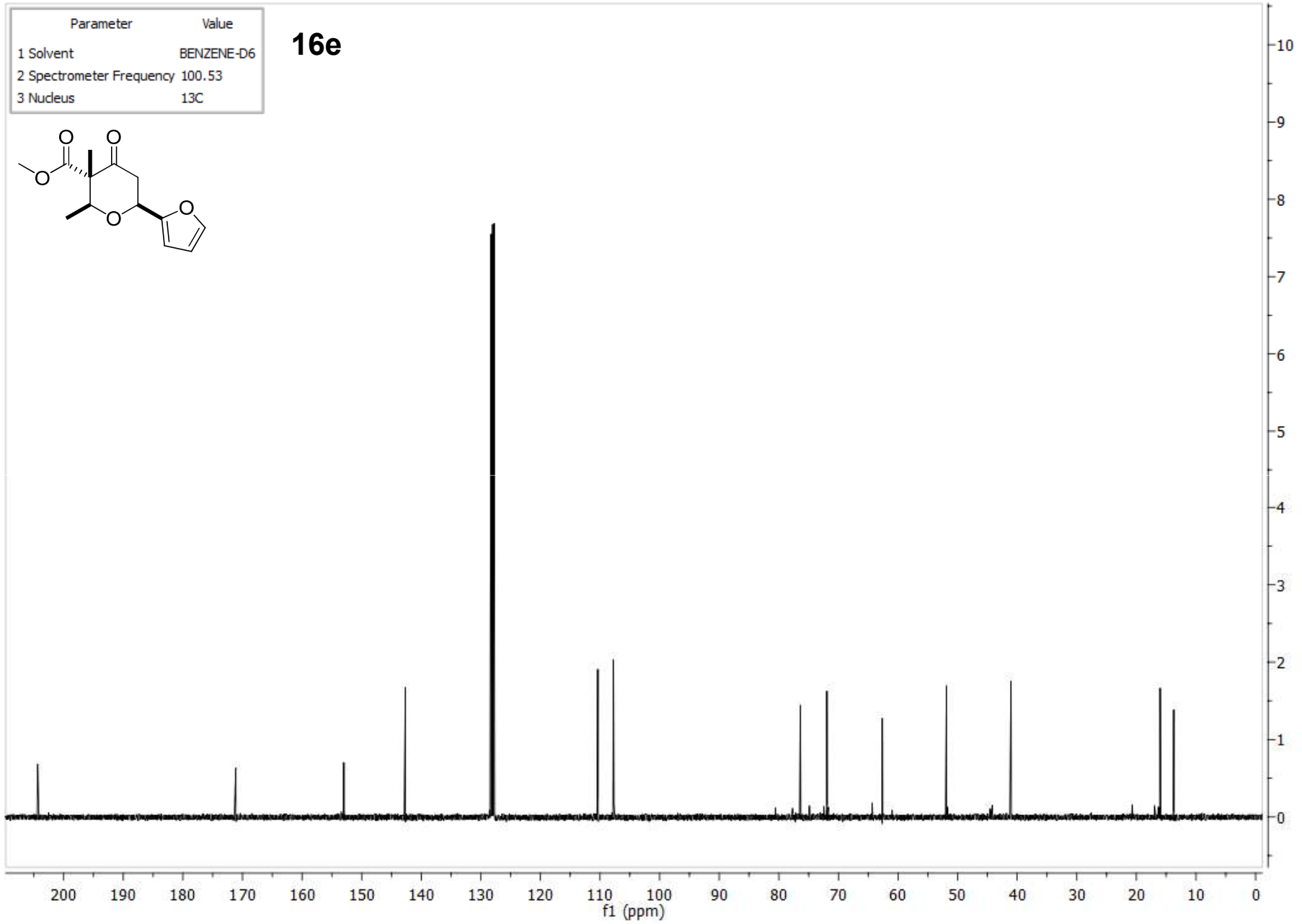

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
16b
1.04
3.37
1.33
2.00
7.49
5.79
4.00
f1 (ppm)

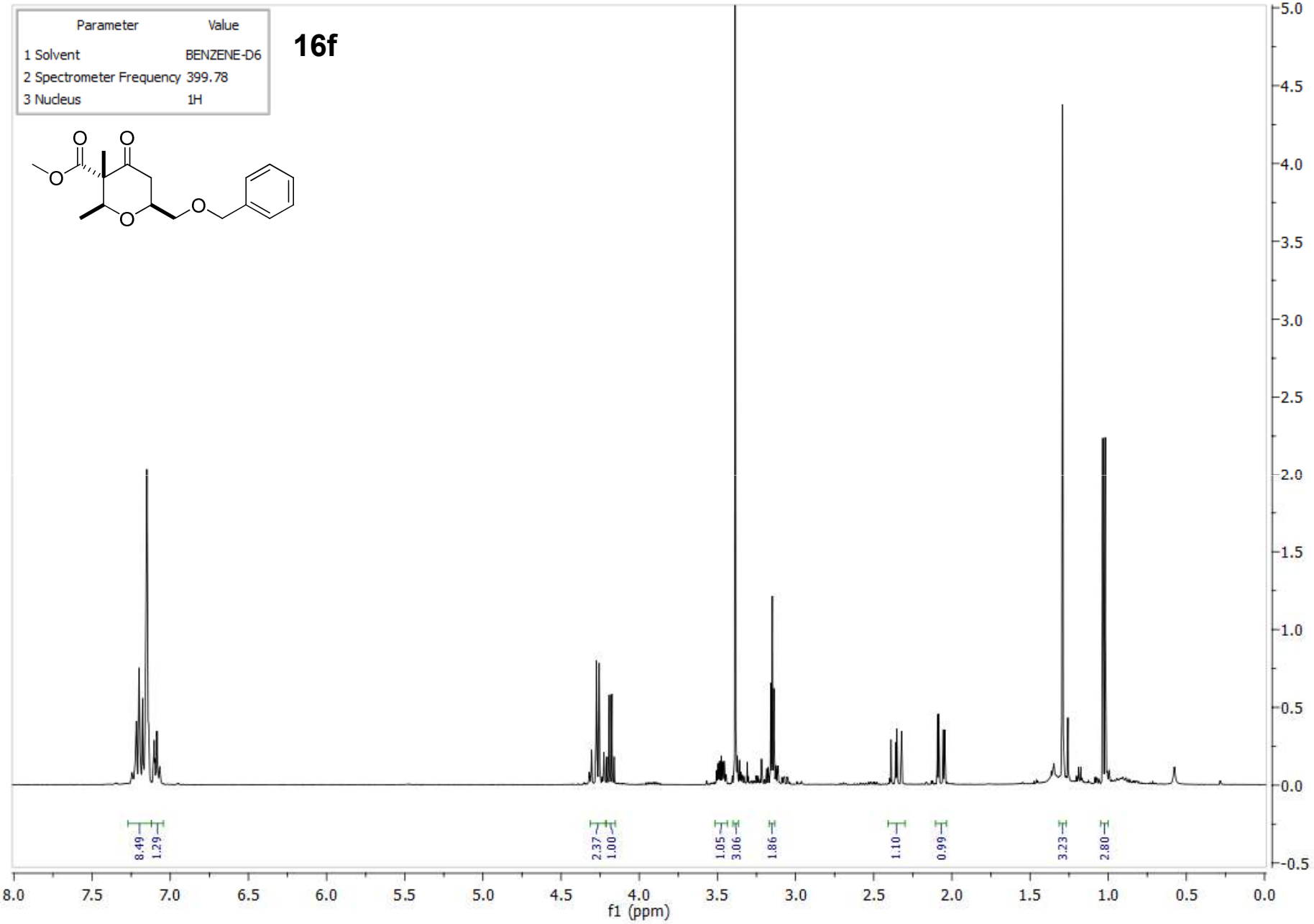

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
16b
O
O
O
O
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

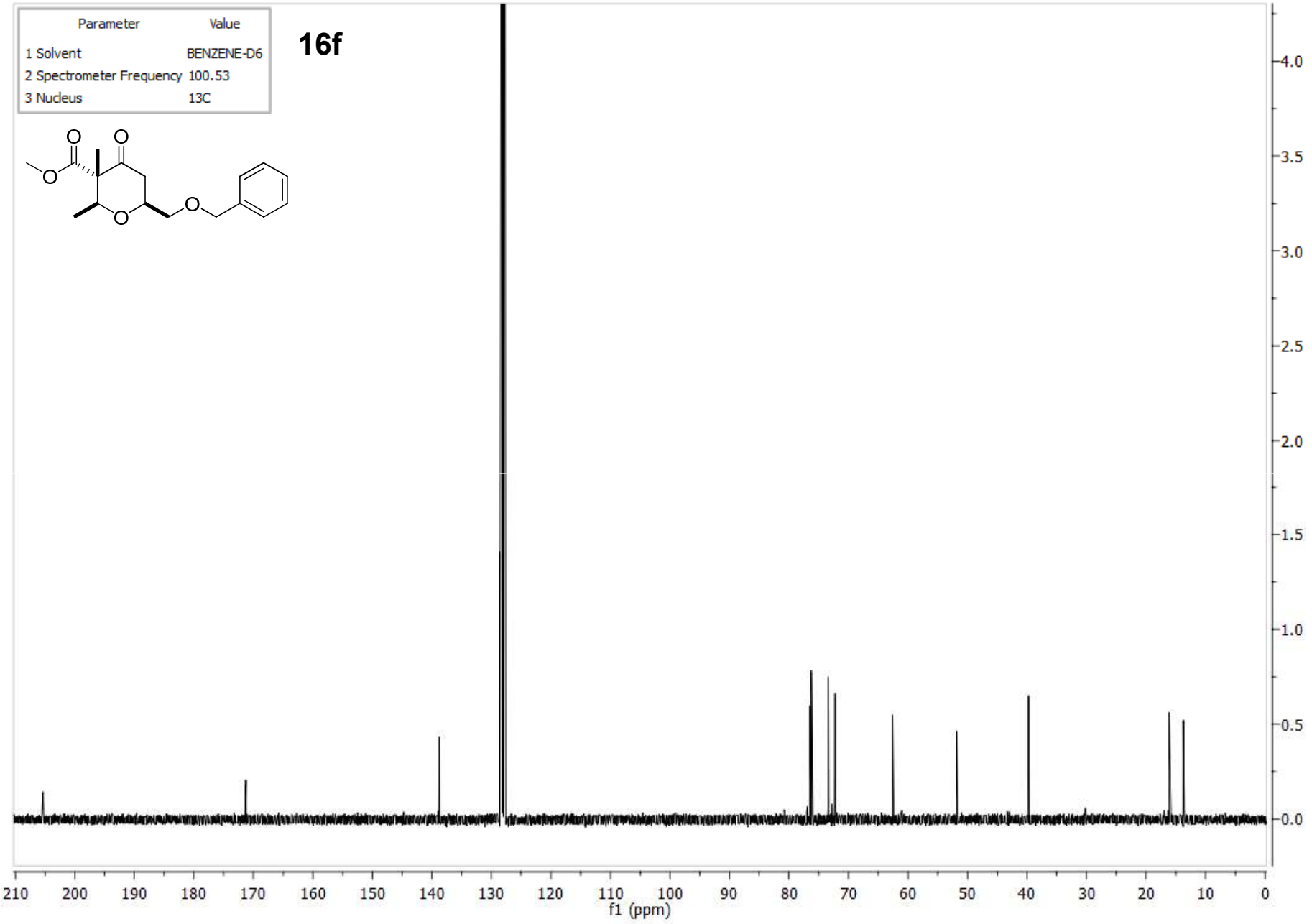

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
16d
1.00
3.01
1.04
1.82
1.20
3.68
3.18
3.04
2.95
f1 (ppm)

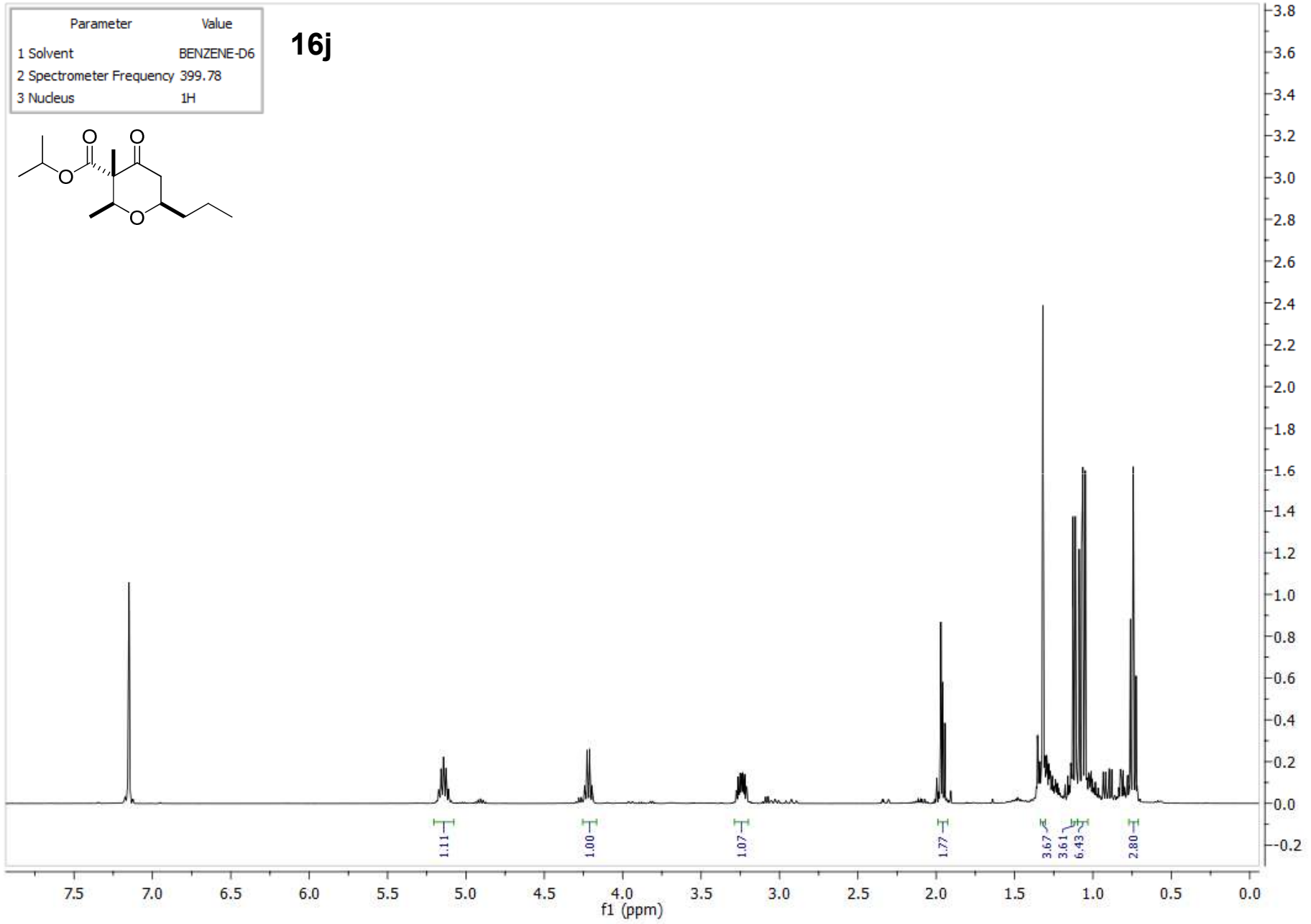

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
16d
f1 (ppm)

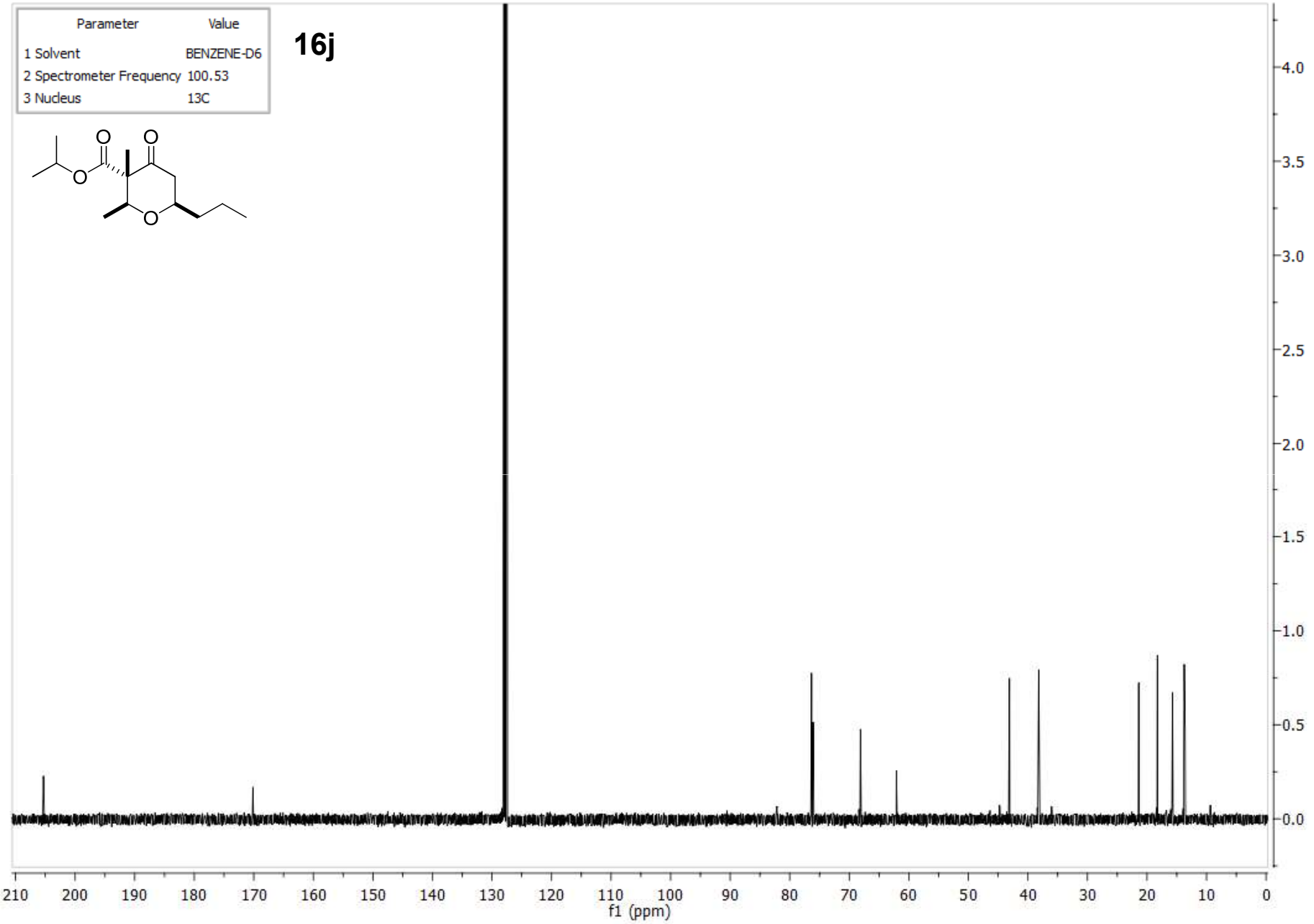

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
16e
1.17
2.31
1.03
1.02
3.13
1.00
0.97
2.72
2.73
f1 (ppm)

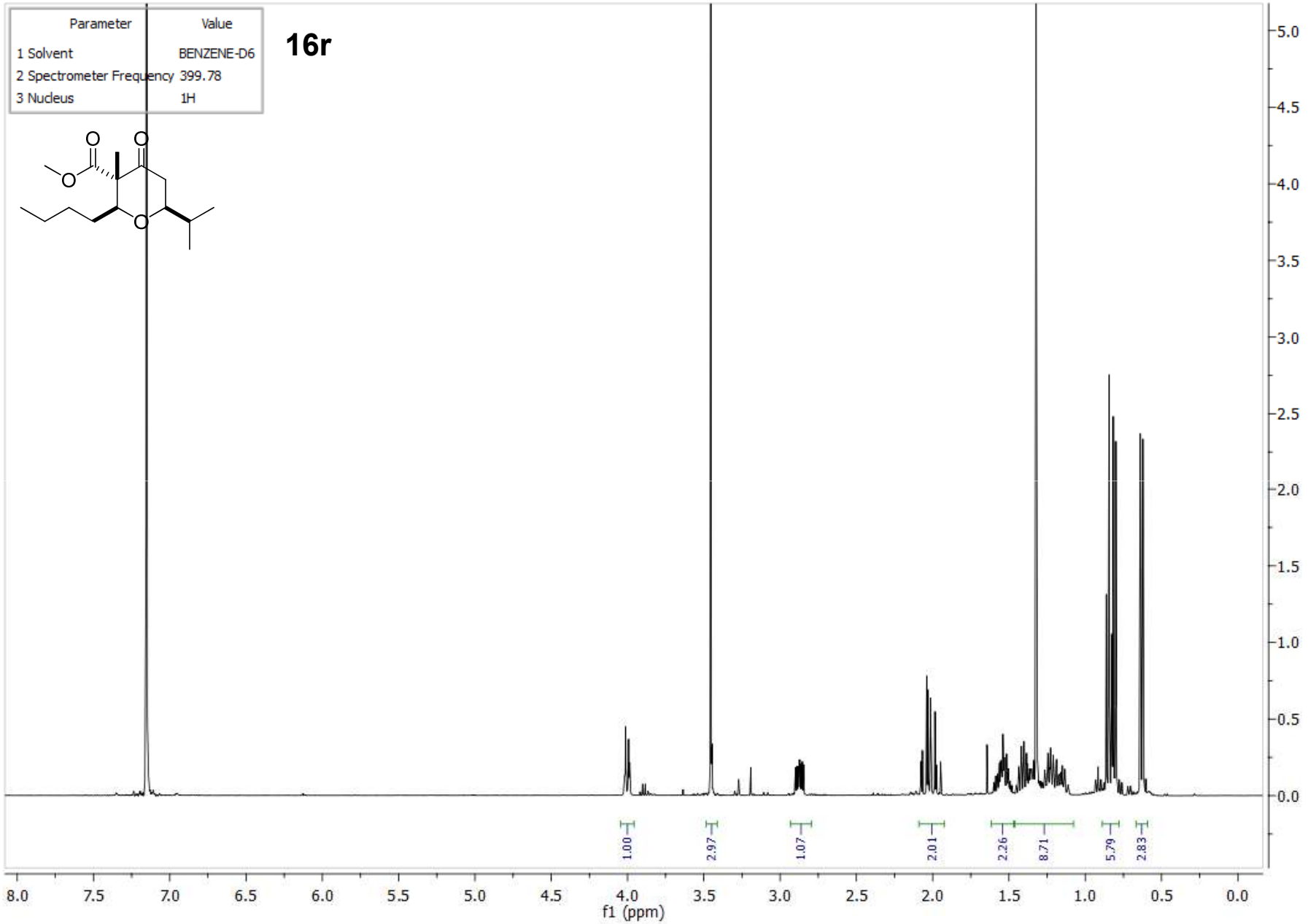

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
16e
O
O
O
O
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
10 9 8 7 6 5 4 3 2 1 0

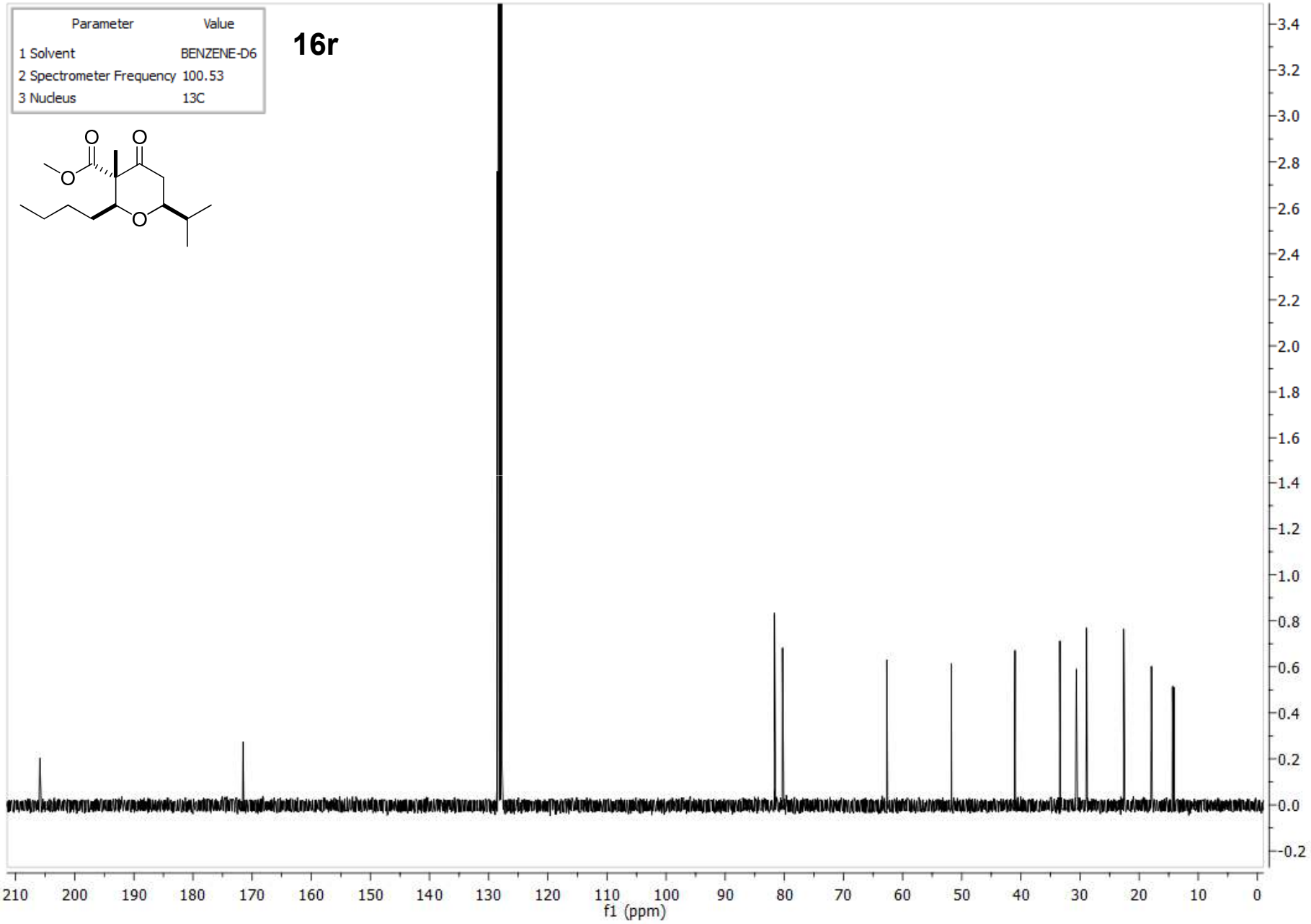

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
16f
8.49
1.29
2.37
1.00
1.05
3.06
1.86
1.10
0.99
3.23
2.80
f1 (ppm)

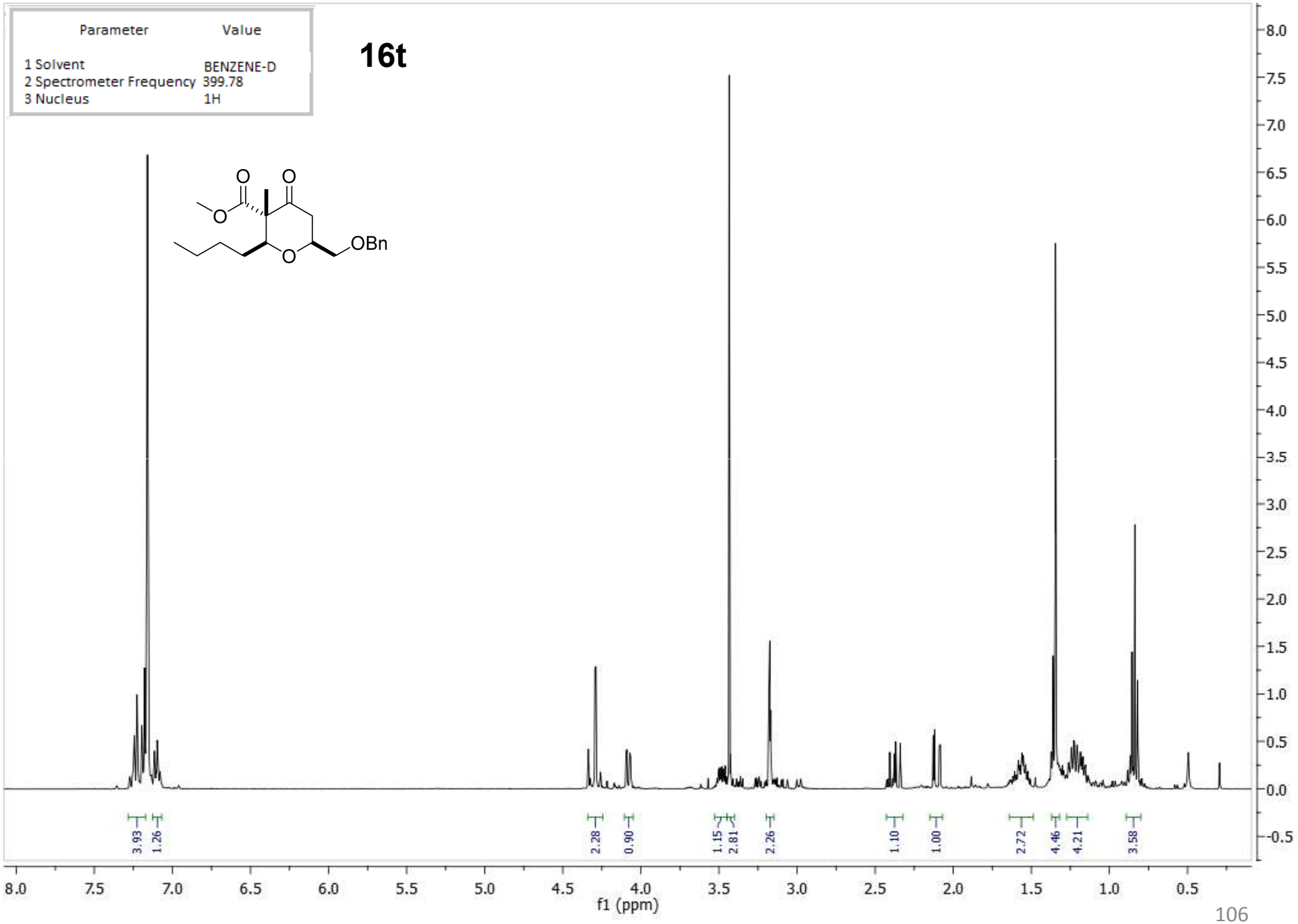

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
16f
O
O
O
O
O
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

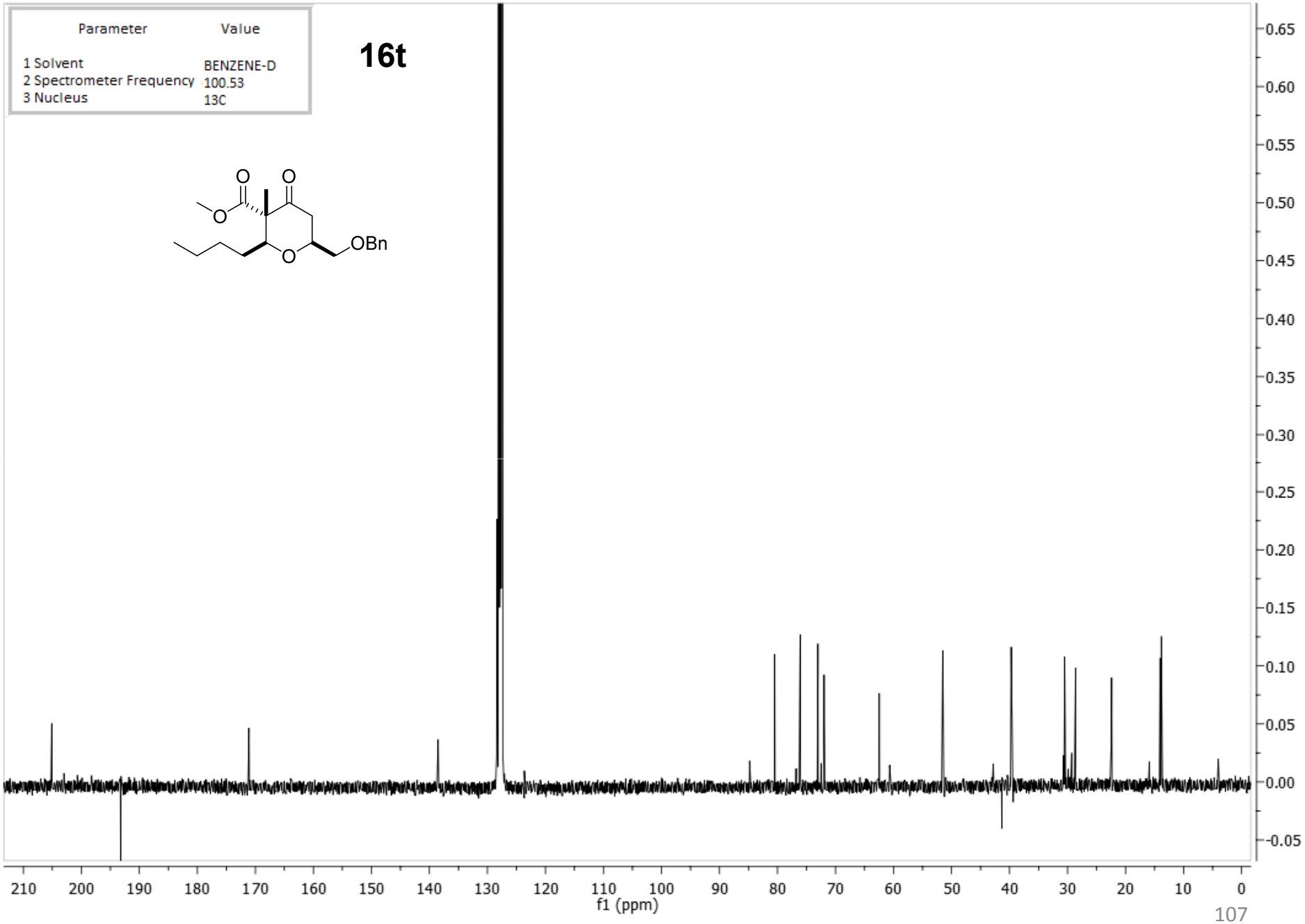

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
16j
1.11
1.00
1.07
1.77
3.67
3.61
6.43
2.80
f1 (ppm)

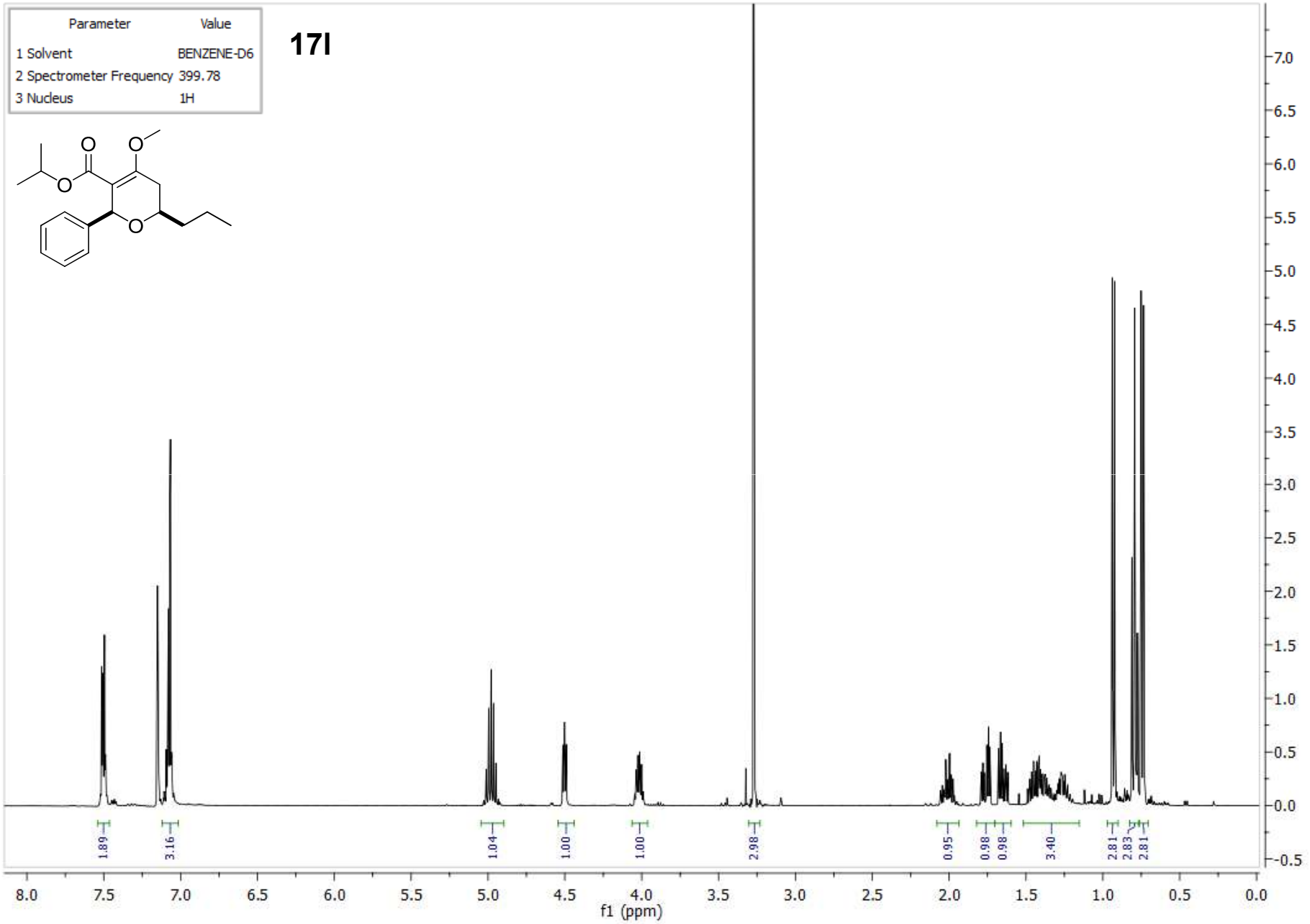

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
16j
O
O
O
O
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

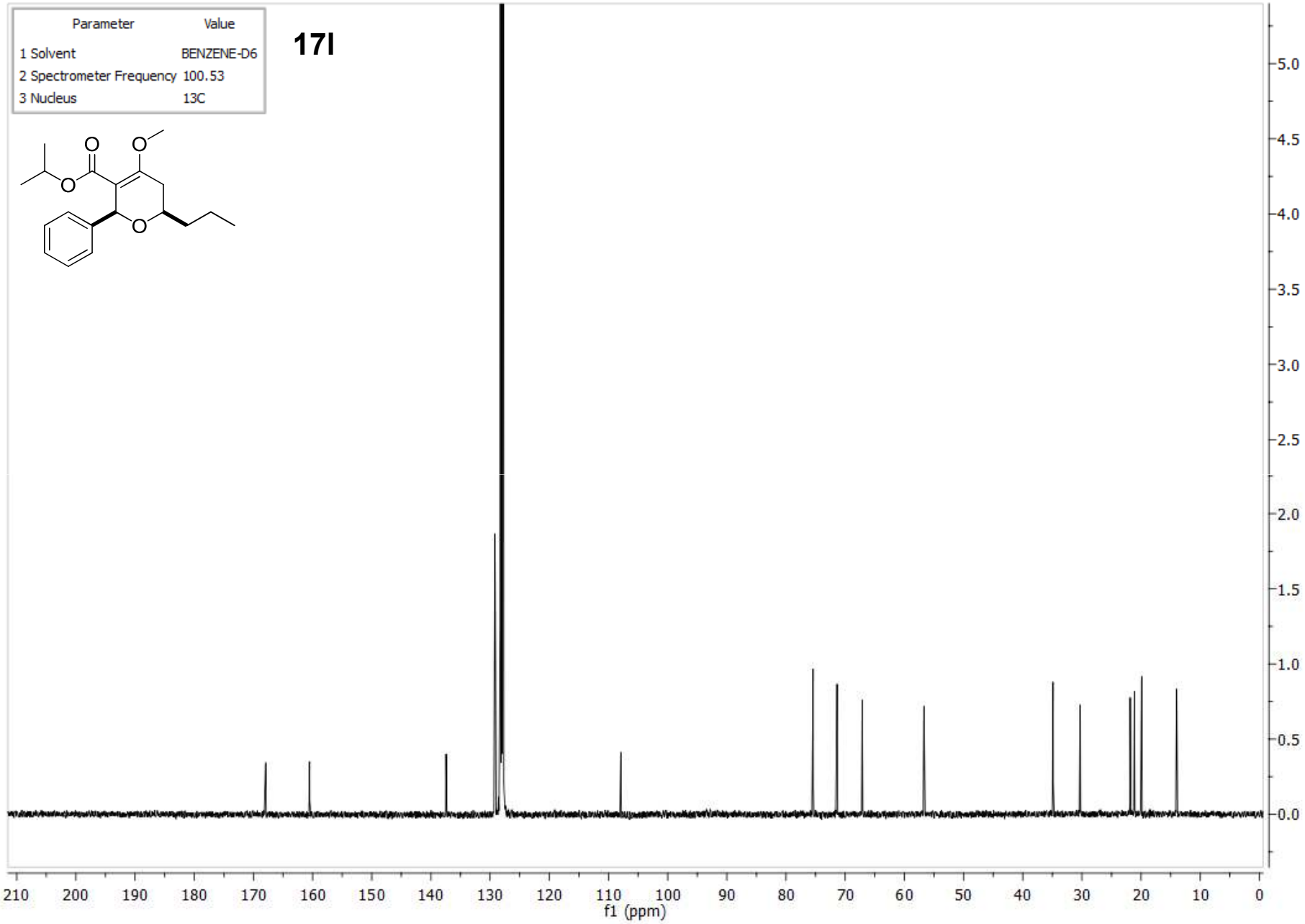

Parameter
Value
1 Solvent
BENZENE-D6
2 Spectrometer Frequency
399.78
3 Nucleus
1H
16r
1.00
2.97
1.07
2.01
2.26
8.71
5.79
2.83
f1 (ppm)

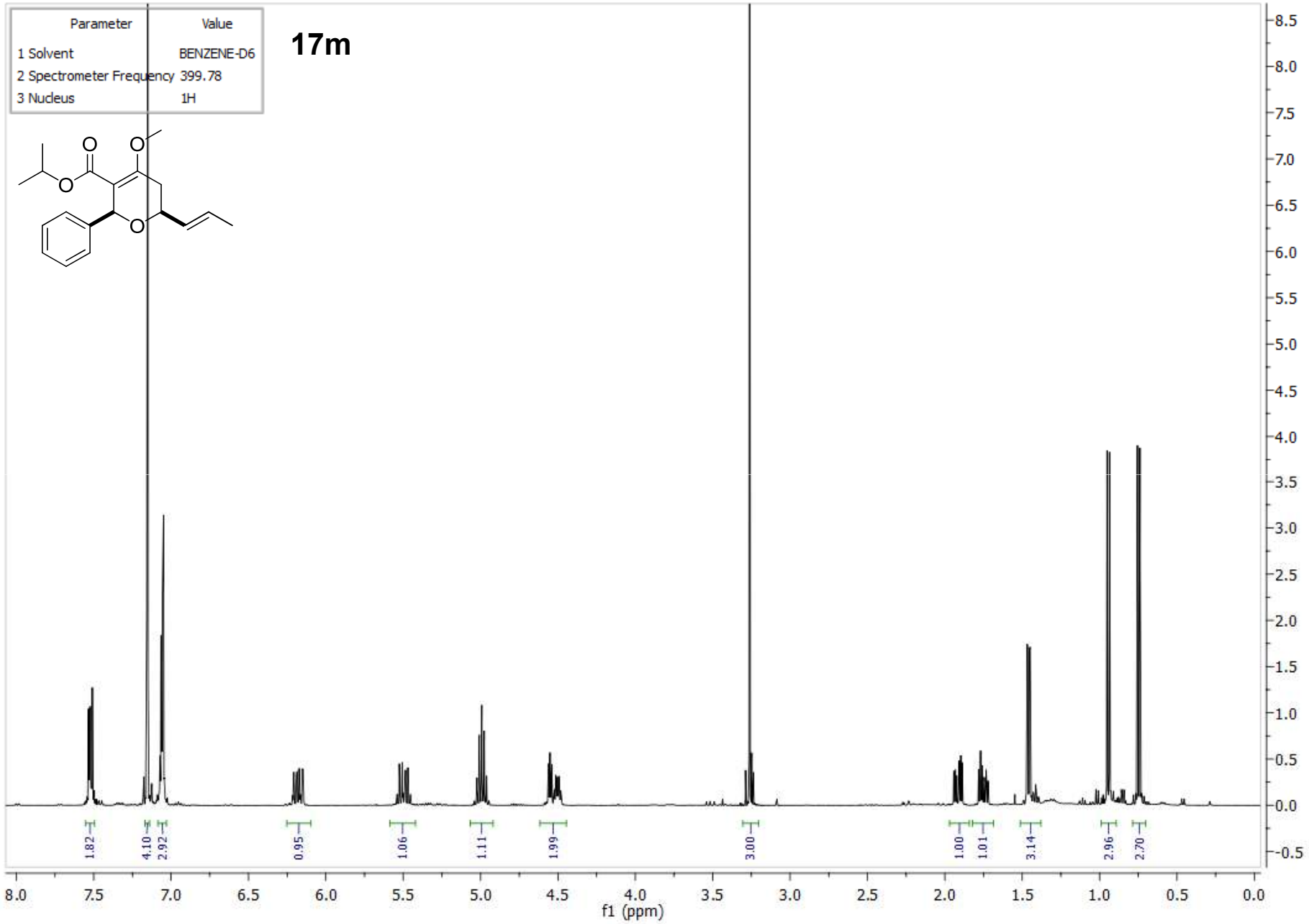

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
16r
O
O
O
O
3.4
3.2
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
-0.2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

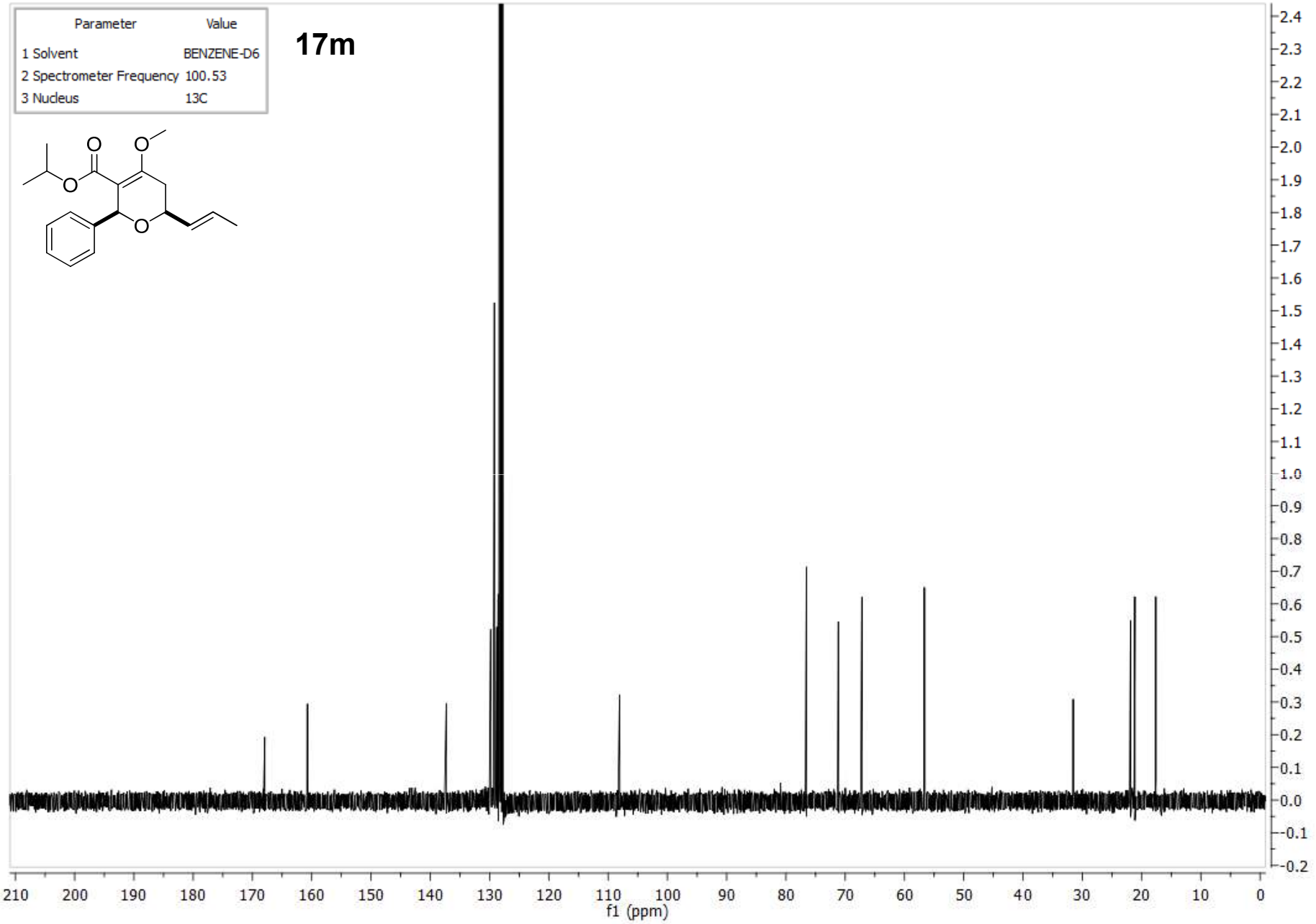

Parameter Value
1 Solvent BENZENE-D
2 Spectrometer Frequency 399.78
3 Nucleus 1H
16t
O
O
O
O
OBn
3.93
1.26
2.28
0.90
1.15
2.81
2.26
1.10
1.00
2.72
4.46
4.21
3.58
f1 (ppm)

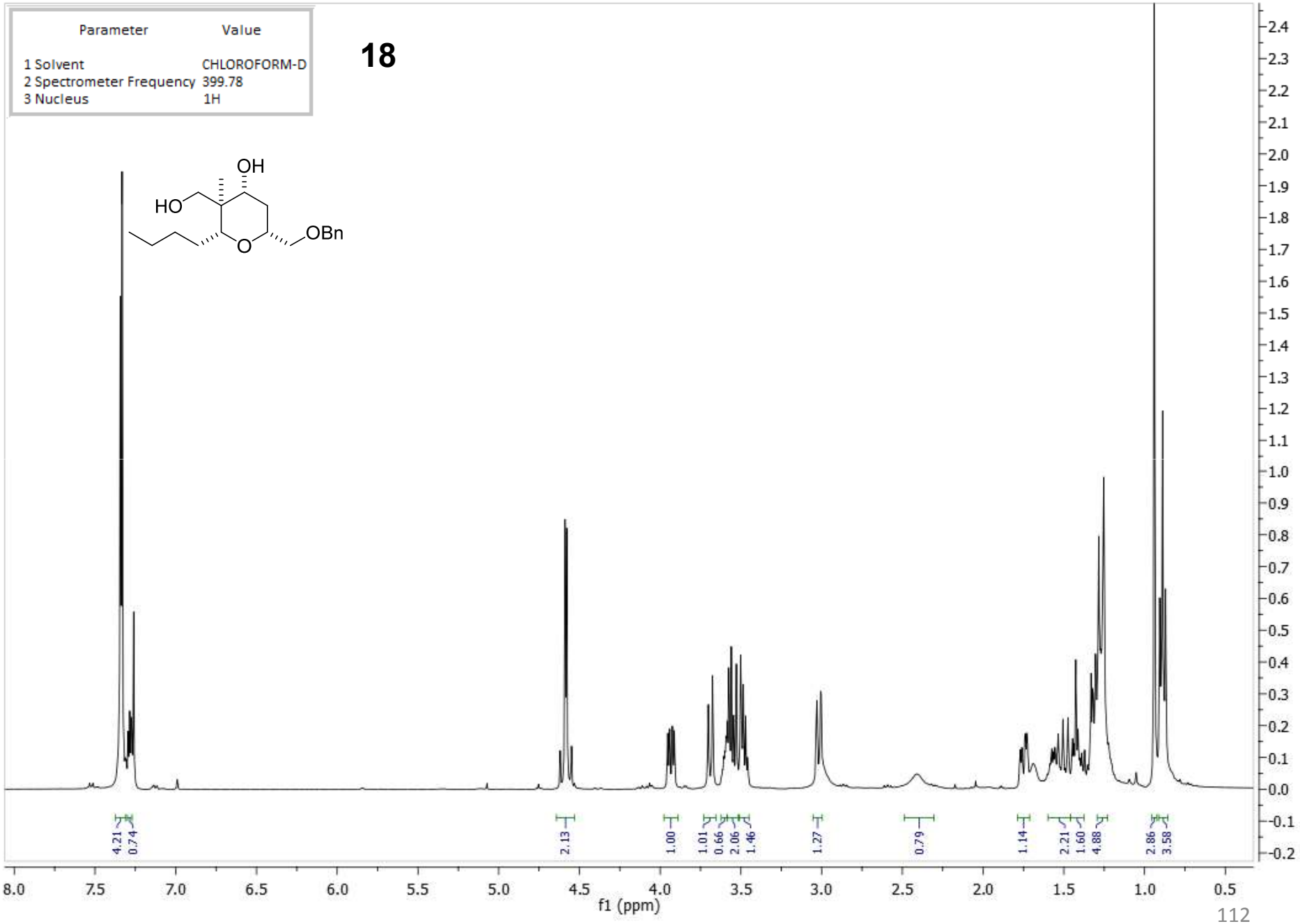

Parameter
Value
1 Solvent
BENZENE-D
2 Spectrometer Frequency
100.53
3 Nucleus
13C
16t
OBn
f1 (ppm)

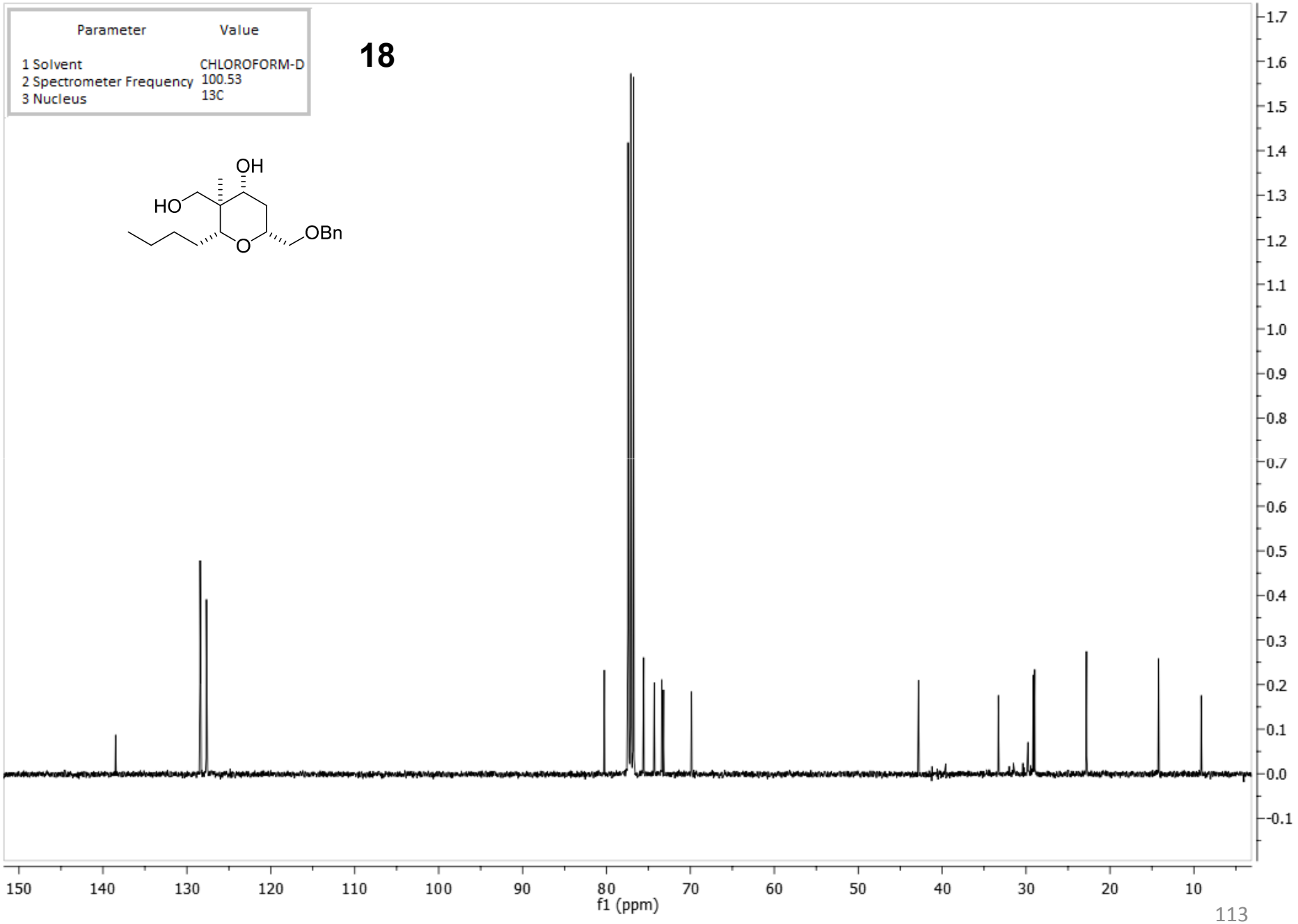

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
17l
O
O
O
O
1.89
3.16
1.04
1.00
1.00
2.98
0.95
0.98
0.98
3.40
2.81
2.83
2.81
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5

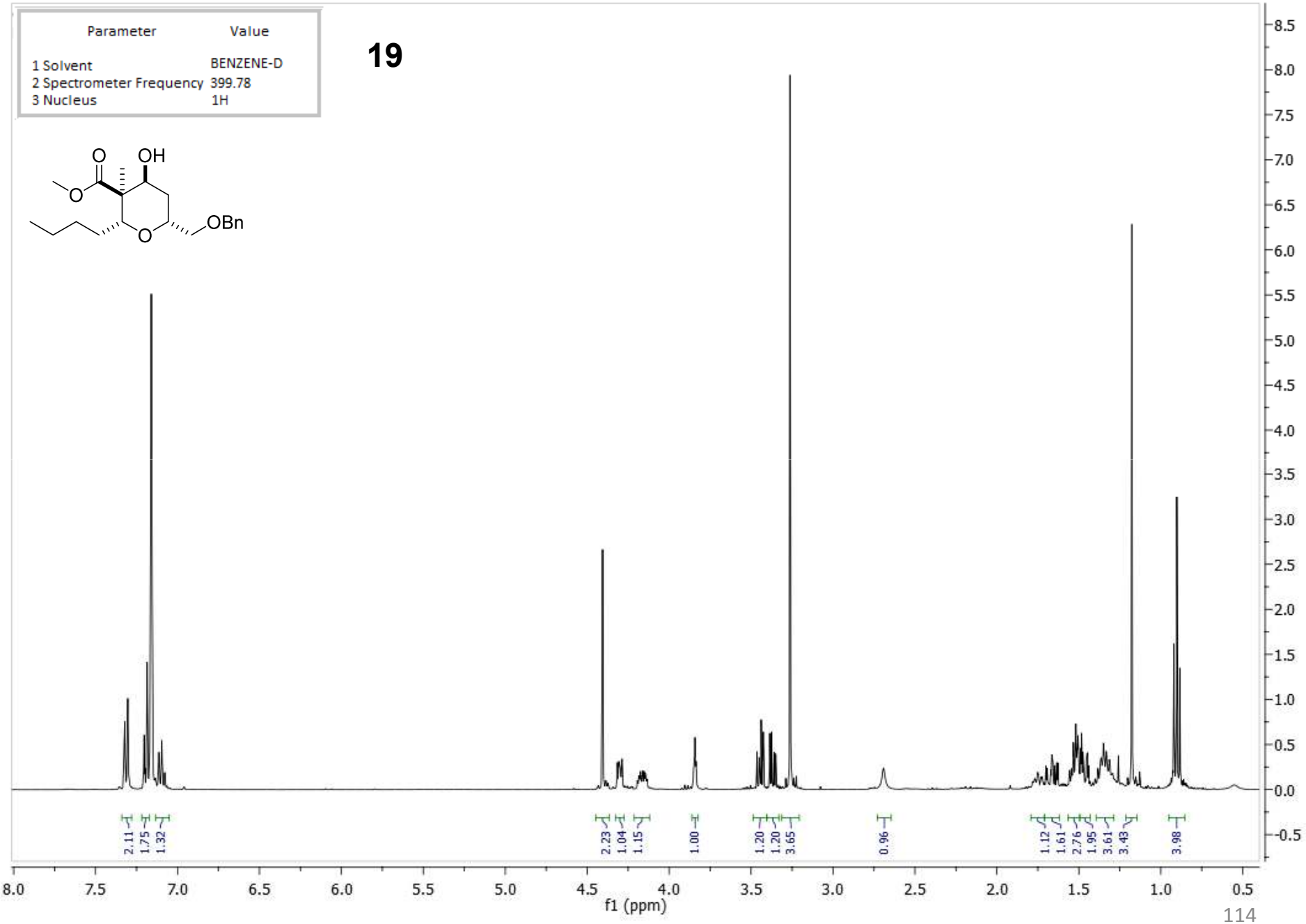

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
17l
O
O
O
O
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

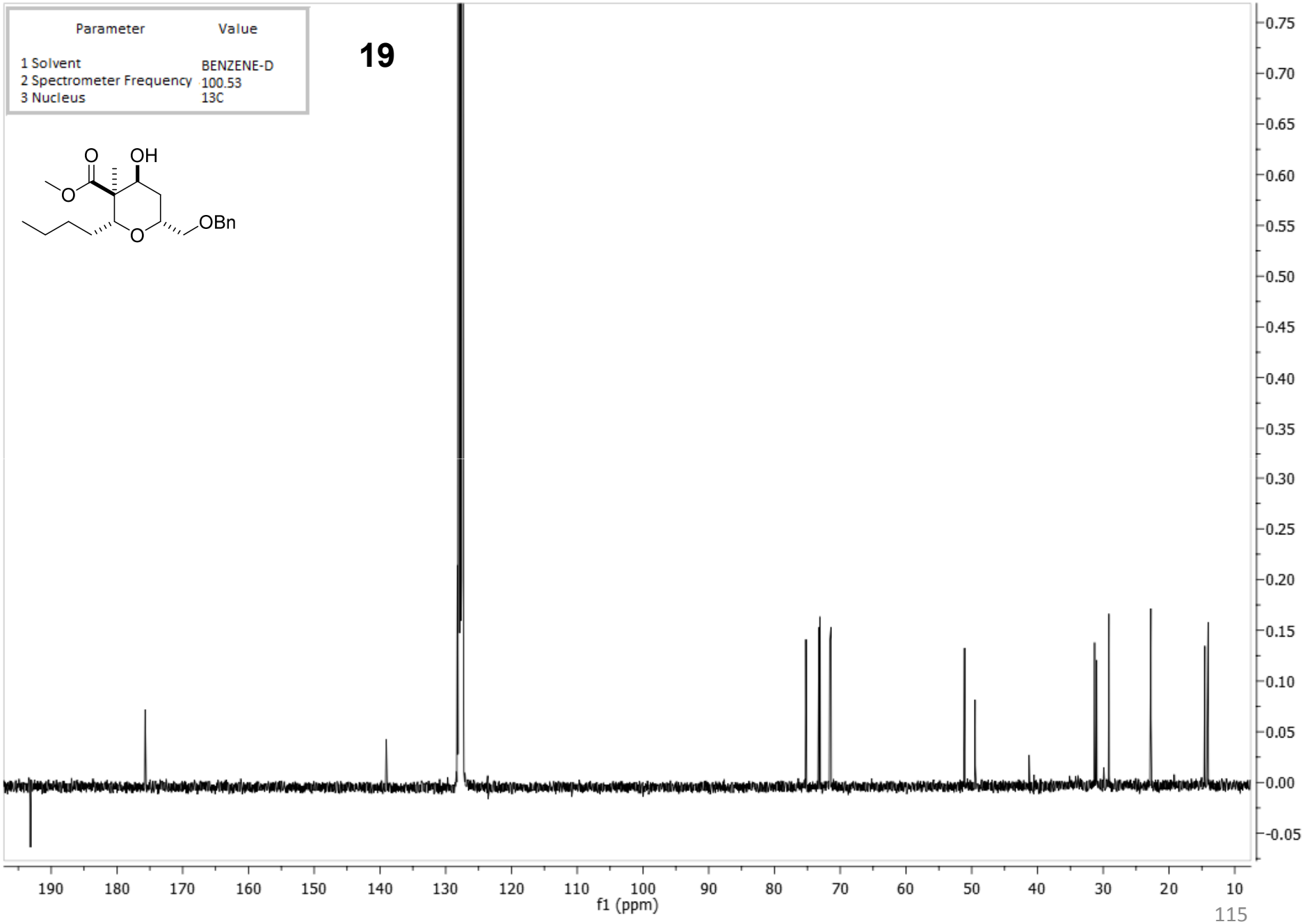

Parameter
Value
1 Solvent
BENZENE-D6
2 Spectrometer Frequency
399.78
3 Nucleus
1H
17m
1.82
4.10
2.92
0.95
1.06
1.11
1.99
3.00
1.00
1.01
3.14
2.96
2.70
f1 (ppm)


Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
17m
f1 (ppm)


Parameter Value
1 Solvent CHLOROFORM-D
2 Spectrometer Frequency 399.78
3 Nucleus 1H
18
OH
HO
OBn
O
4.21
0.74
2.13
1.00
1.01
0.66
2.06
1.46
1.27
0.79
1.14
2.21
1.60
4.88
2.86
3.58
f1 (ppm)

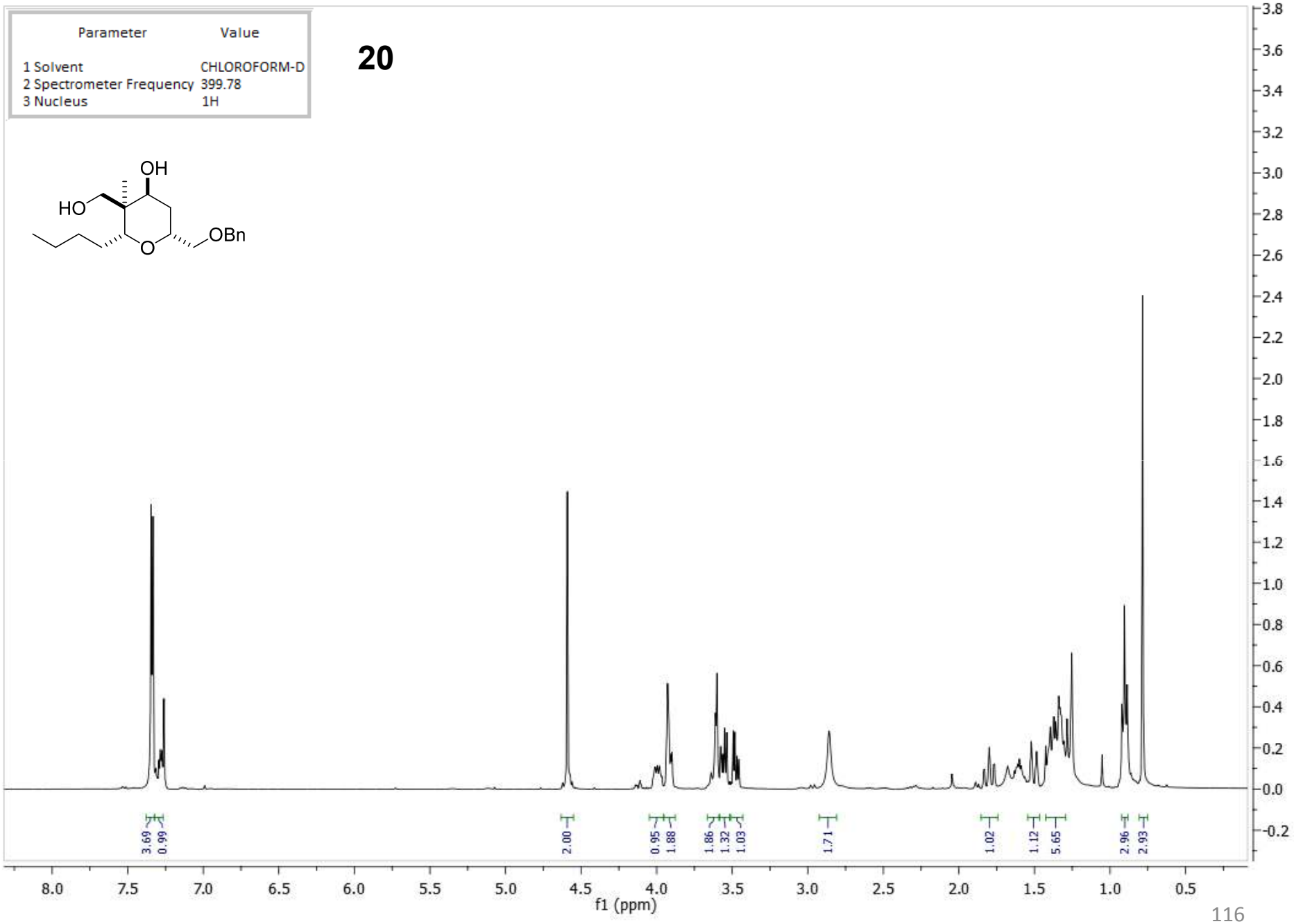

Parameter
Value
1 Solvent
CHLOROFORM-D
2 Spectrometer Frequency
100.53
3 Nucleus
13C
18
OH
HO
OBn
O
f1 (ppm)

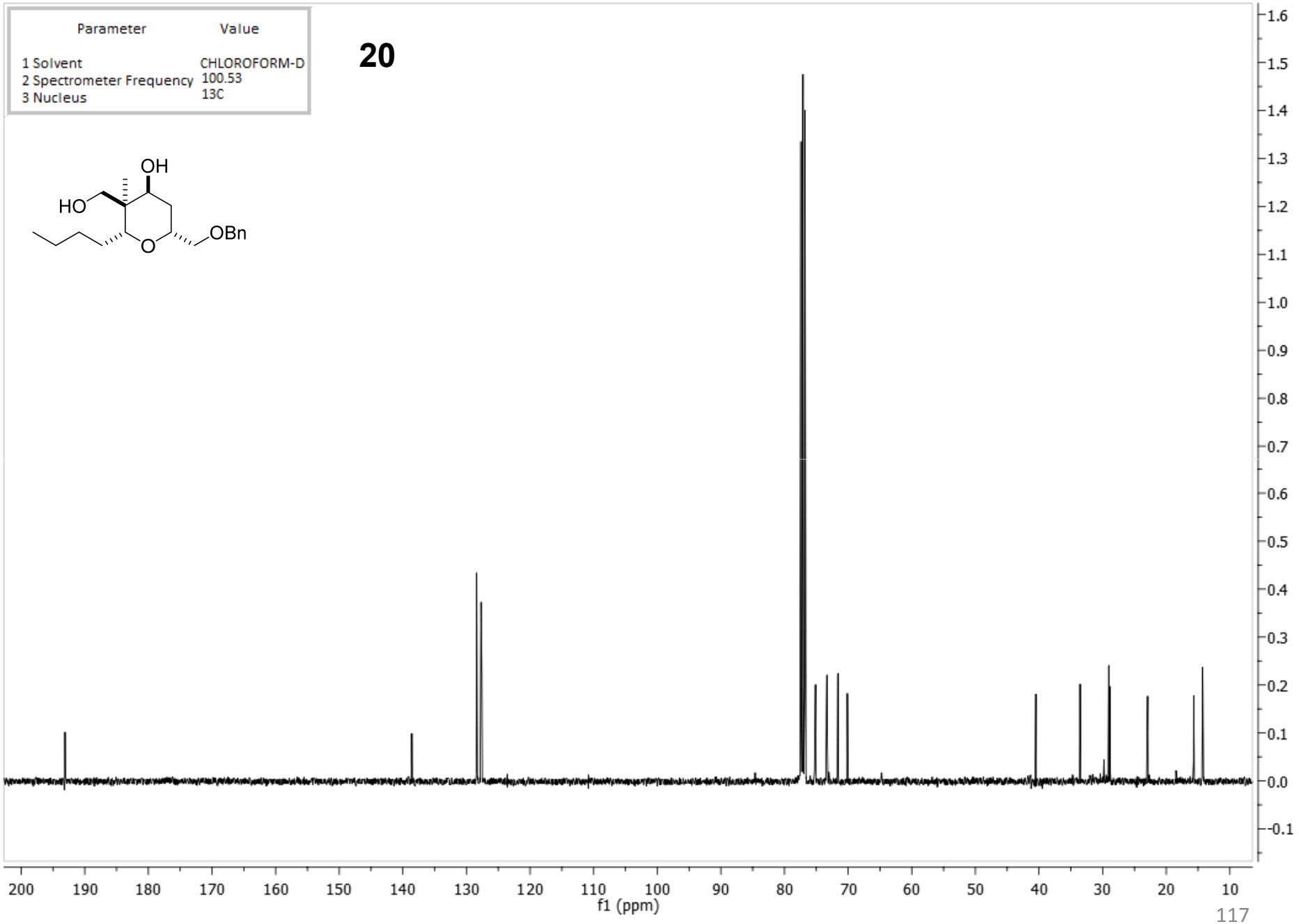

Parameter Value
1 Solvent BENZENE-D
2 Spectrometer Frequency 399.78
3 Nucleus 1H
19
O
OH
O
OBn
O
2.11
1.75
1.32
2.23
1.04
1.15
1.00
1.20
1.20
3.65
0.96
1.12
1.61
2.76
1.95
3.61
3.43
3.98
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

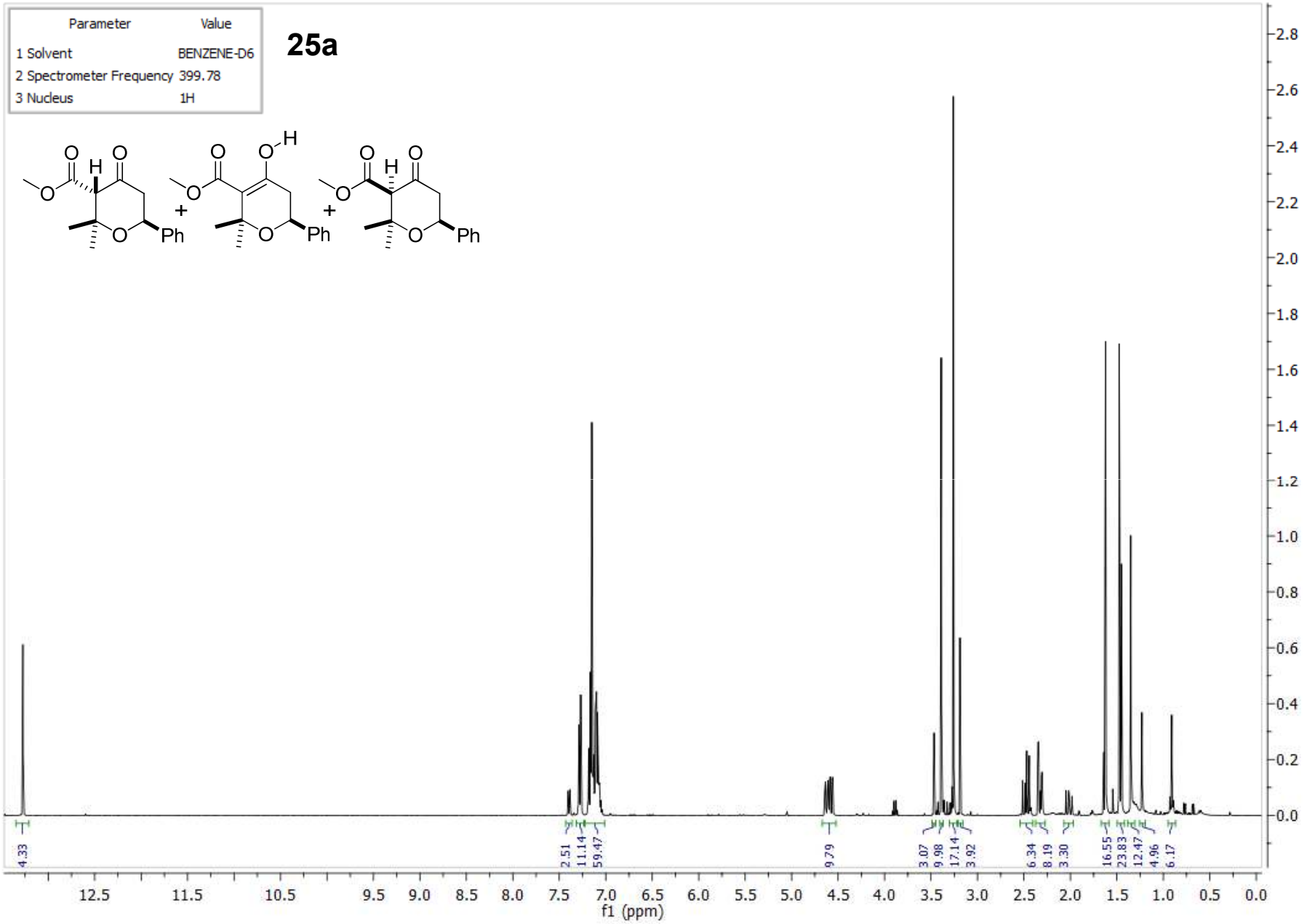

Parameter
Value
1 Solvent
BENZENE-D
2 Spectrometer Frequency
100.53
3 Nucleus
13C
19
O
OH
O
OBn
O
0.75
0.70
0.65
0.60
0.55
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
-0.05
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

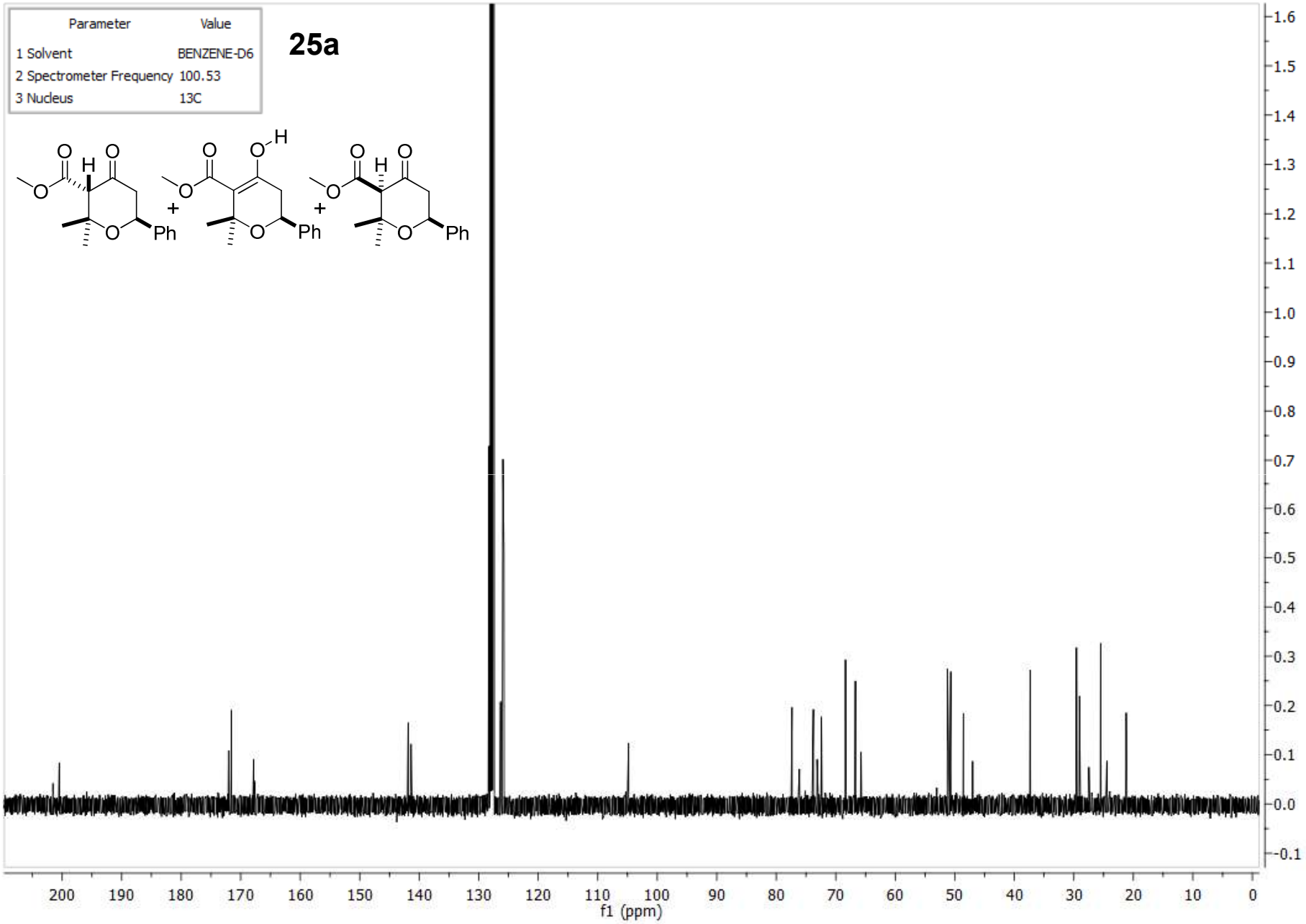
Parameter	Value
1 Solvent	CHLOROFORM-D
2 Spectrometer Frequency	399.78
3 Nucleus	1H

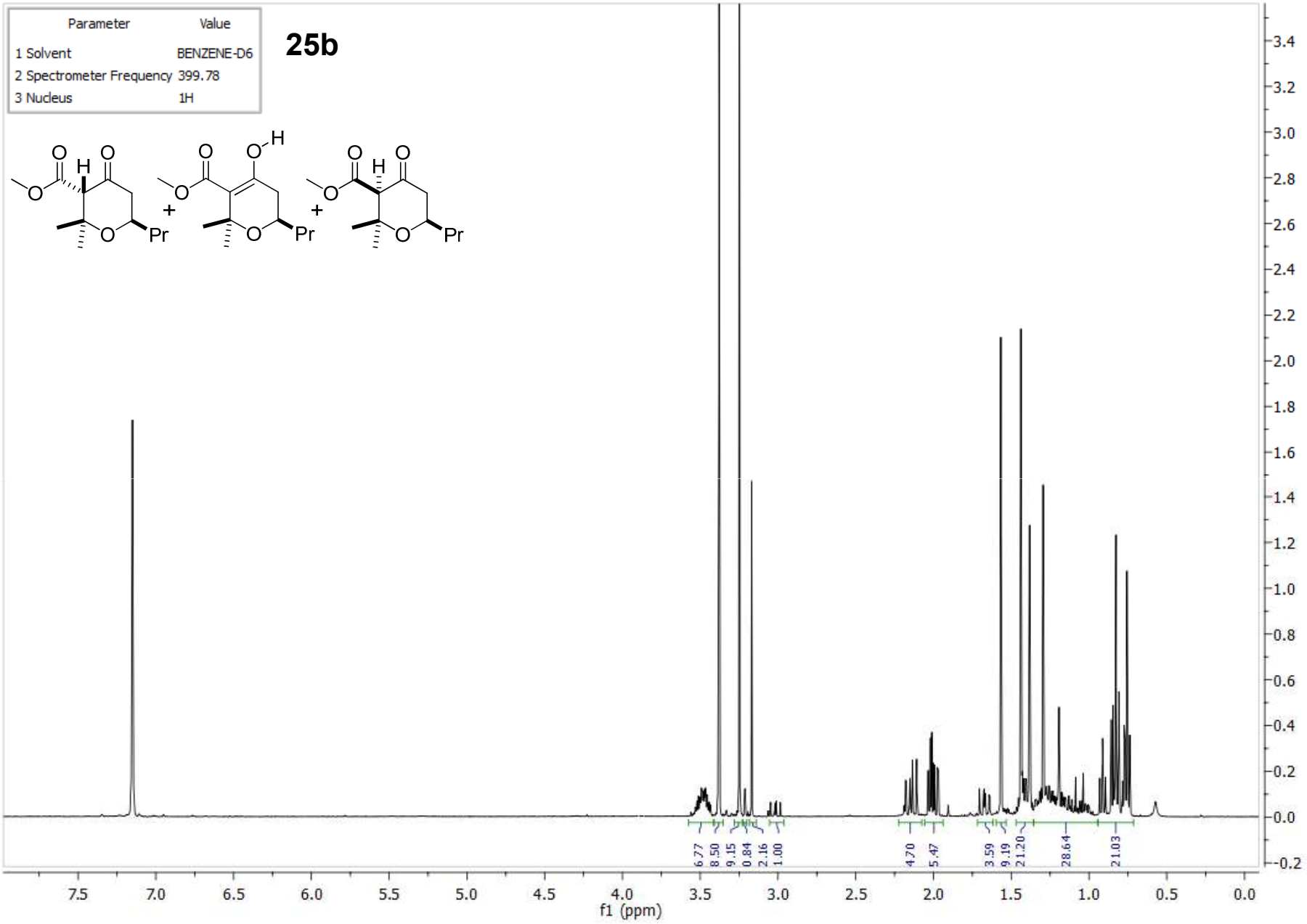
20

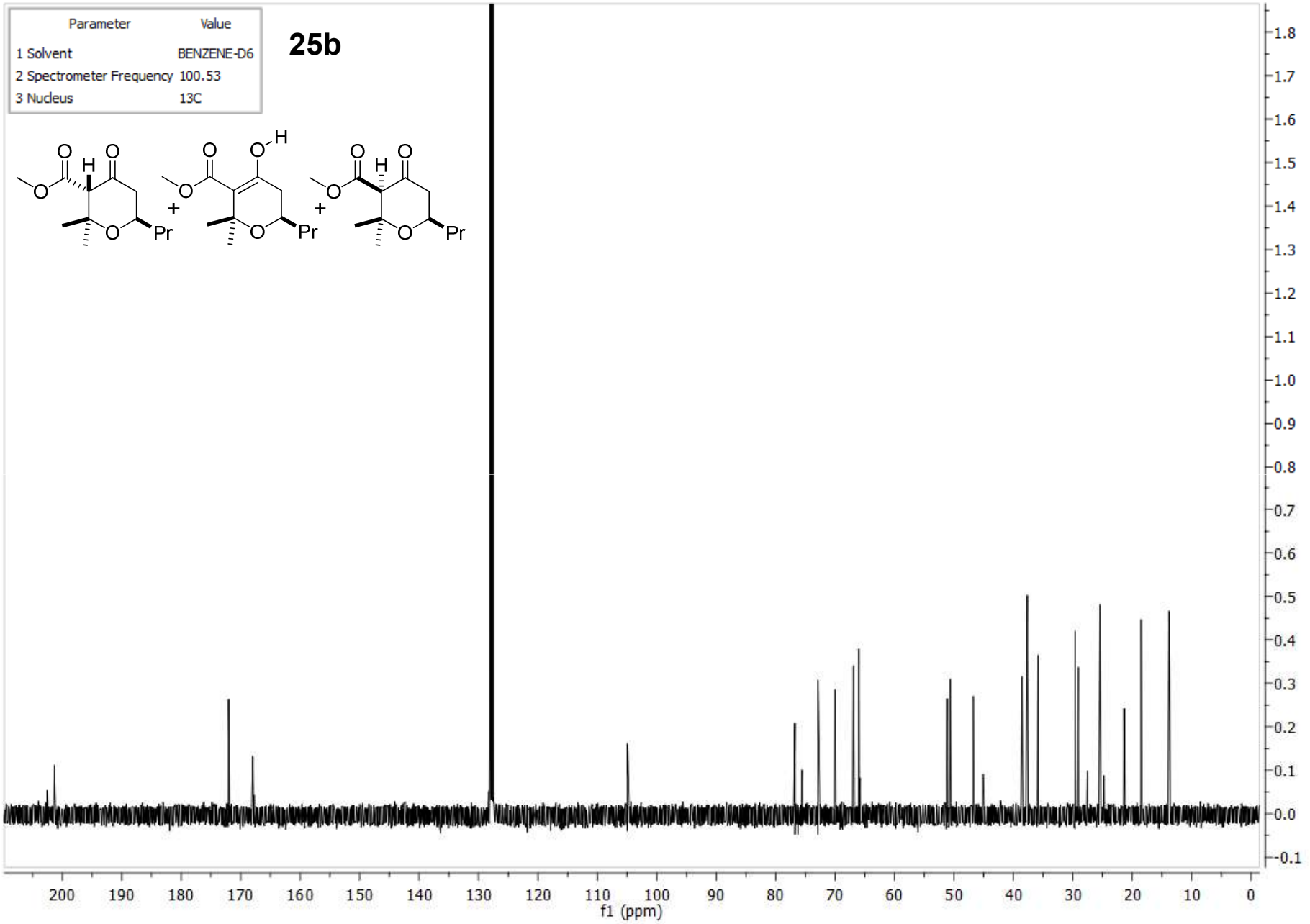


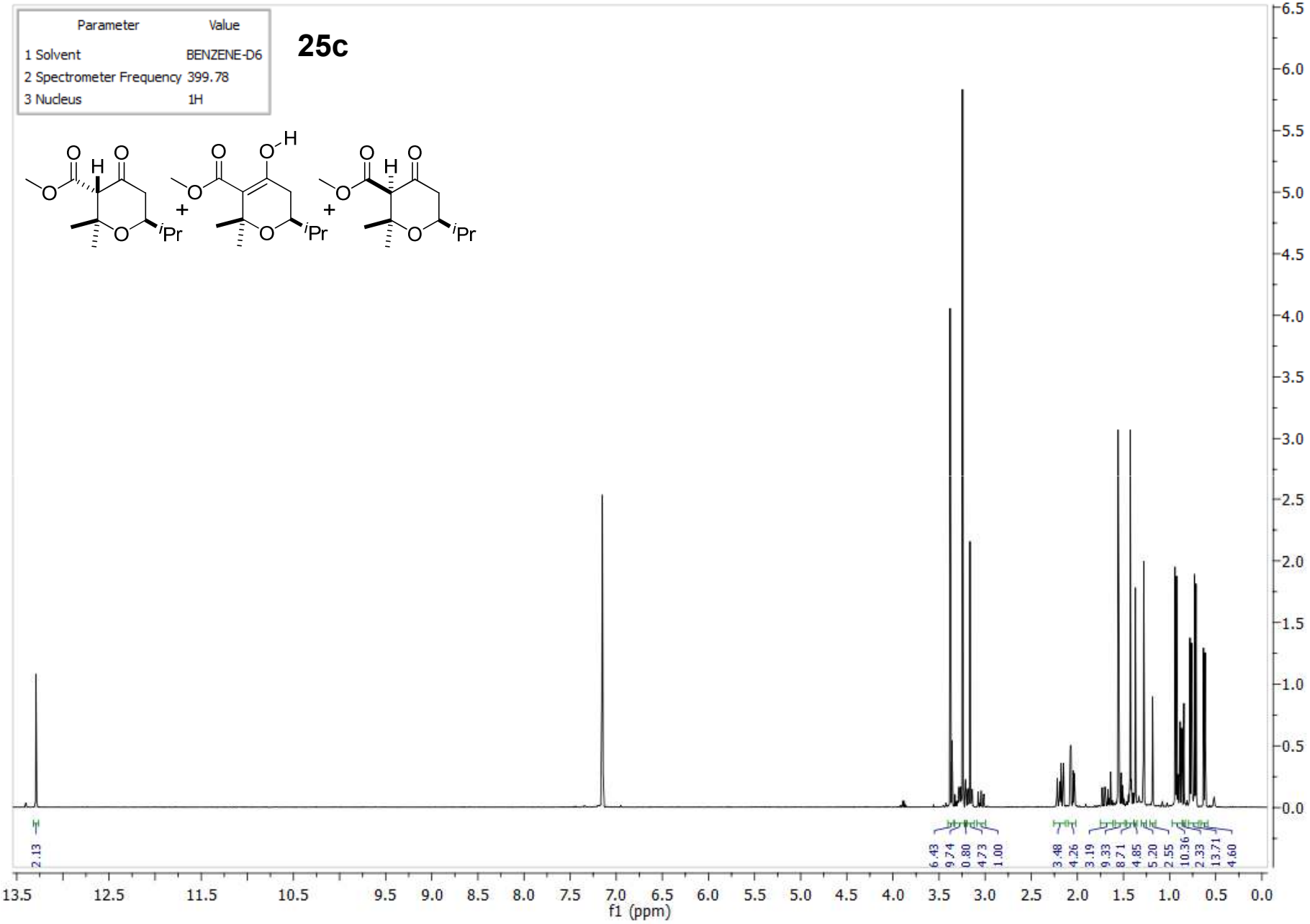

Parameter
Value
1 Solvent
CHLOROFORM-D
2 Spectrometer Frequency
100.53
3 Nucleus
13C
20
OH
HO
OBn
O
f1 (ppm)

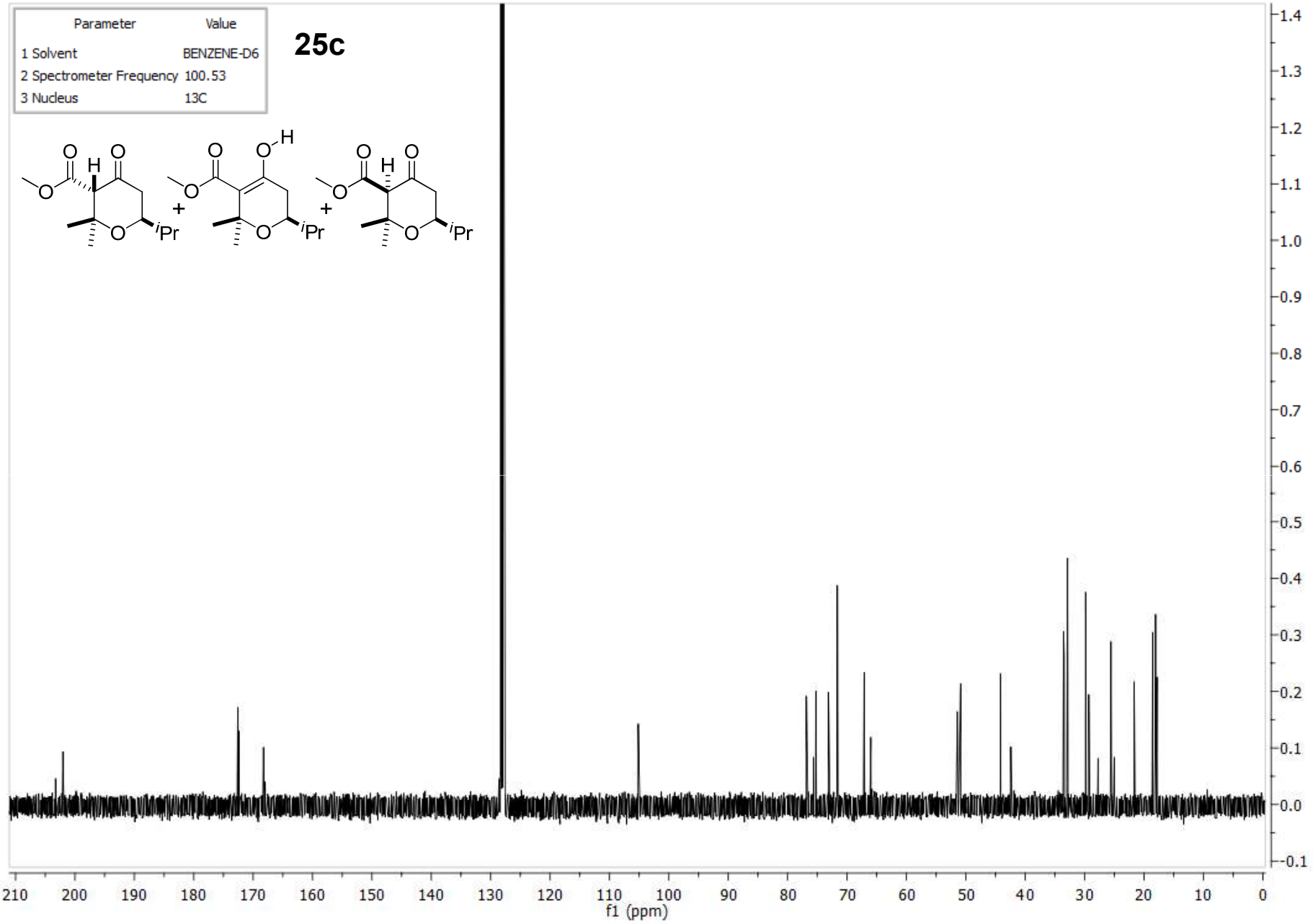

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
25a
Ph
Ph
Ph
4.33
2.51
11.14
59.47
9.79
3.07
9.98
17.14
3.92
6.34
8.19
3.30
16.55
23.83
12.47
4.96
6.17
f1 (ppm)

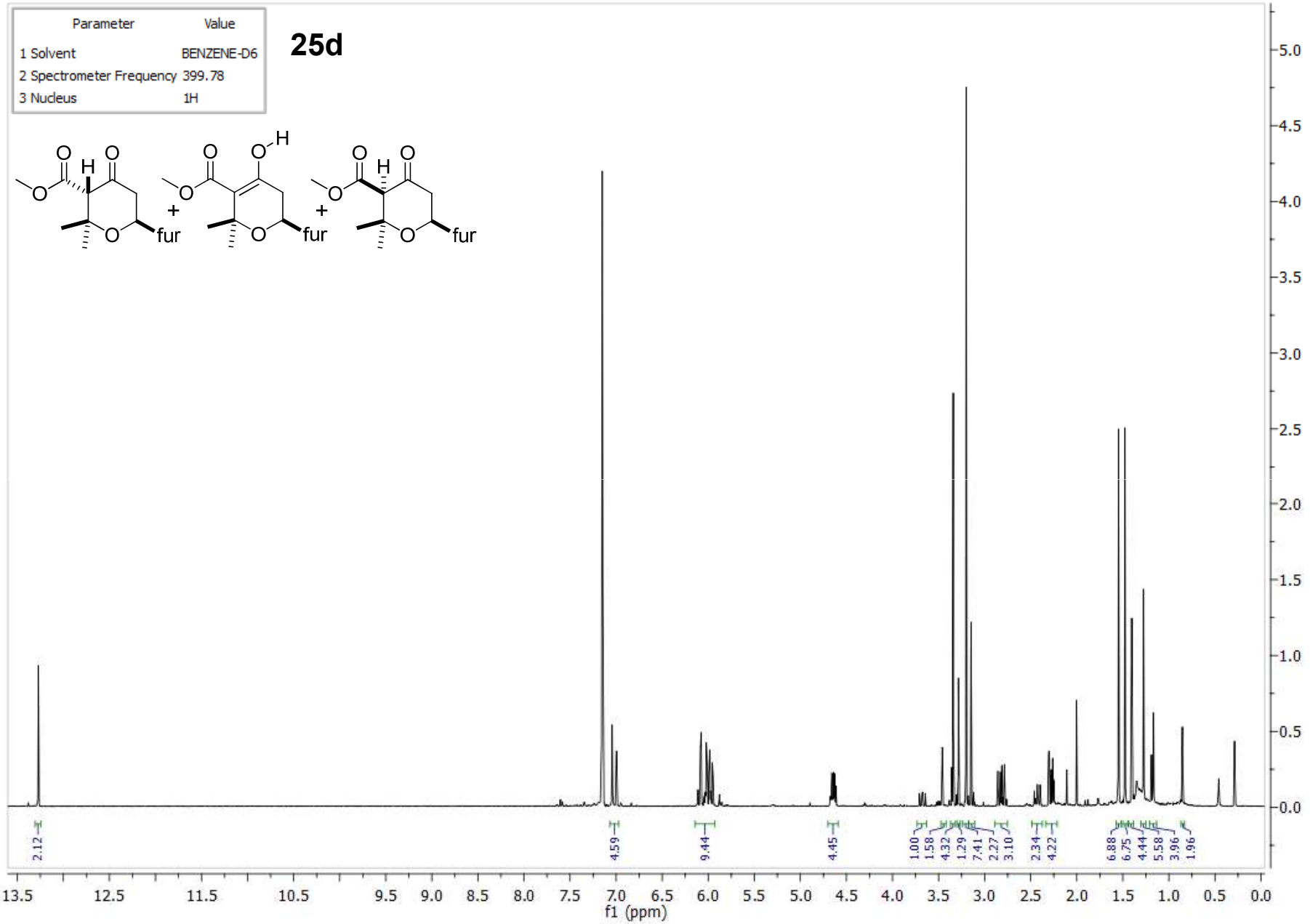

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
25a
Ph
Ph
Ph
f1 (ppm)

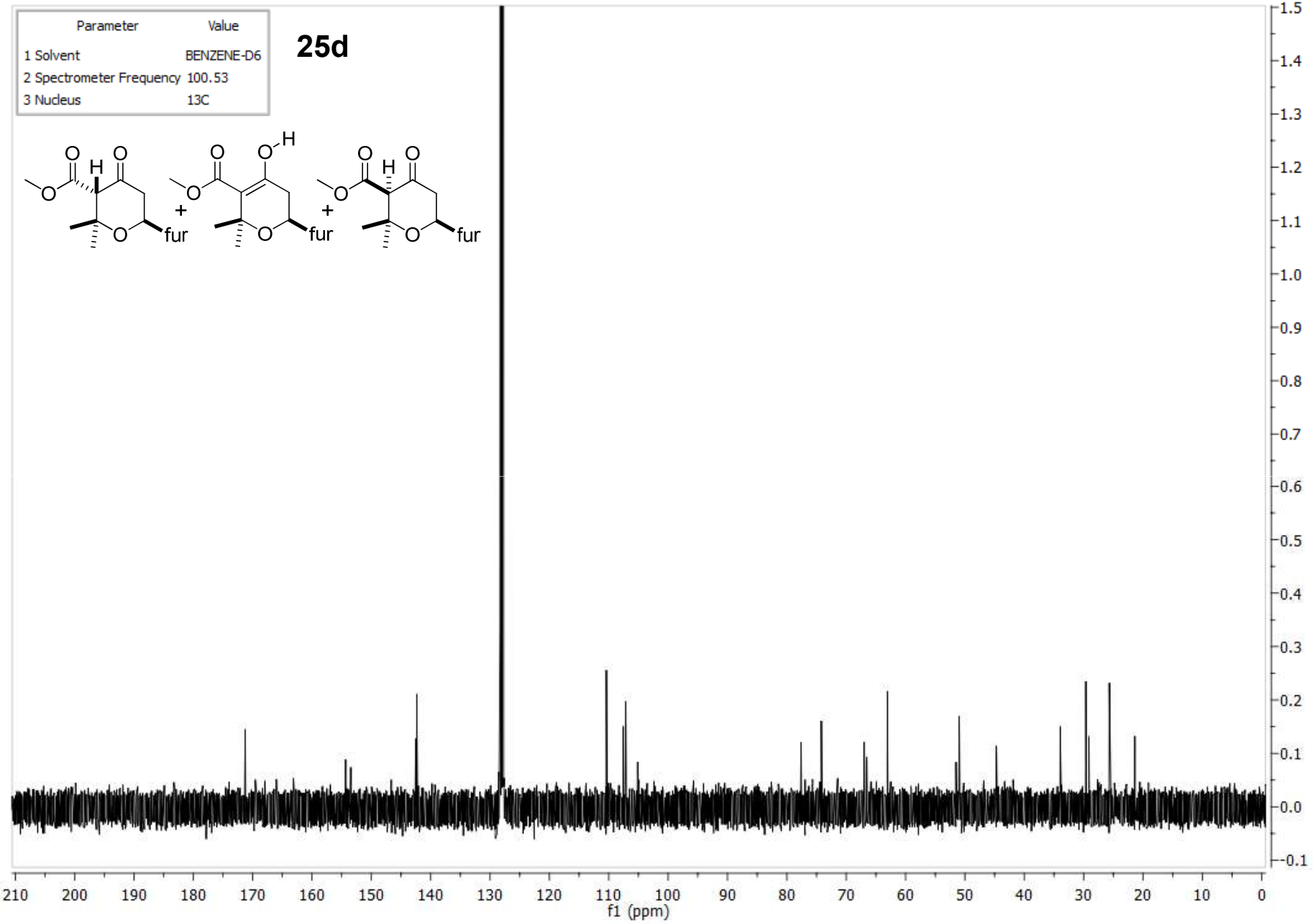

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
25b
Pr
Pr
Pr
6.77
8.50
9.15
0.84
2.16
1.00
4.70
5.47
3.59
9.19
21.20
28.64
21.03
f1 (ppm)

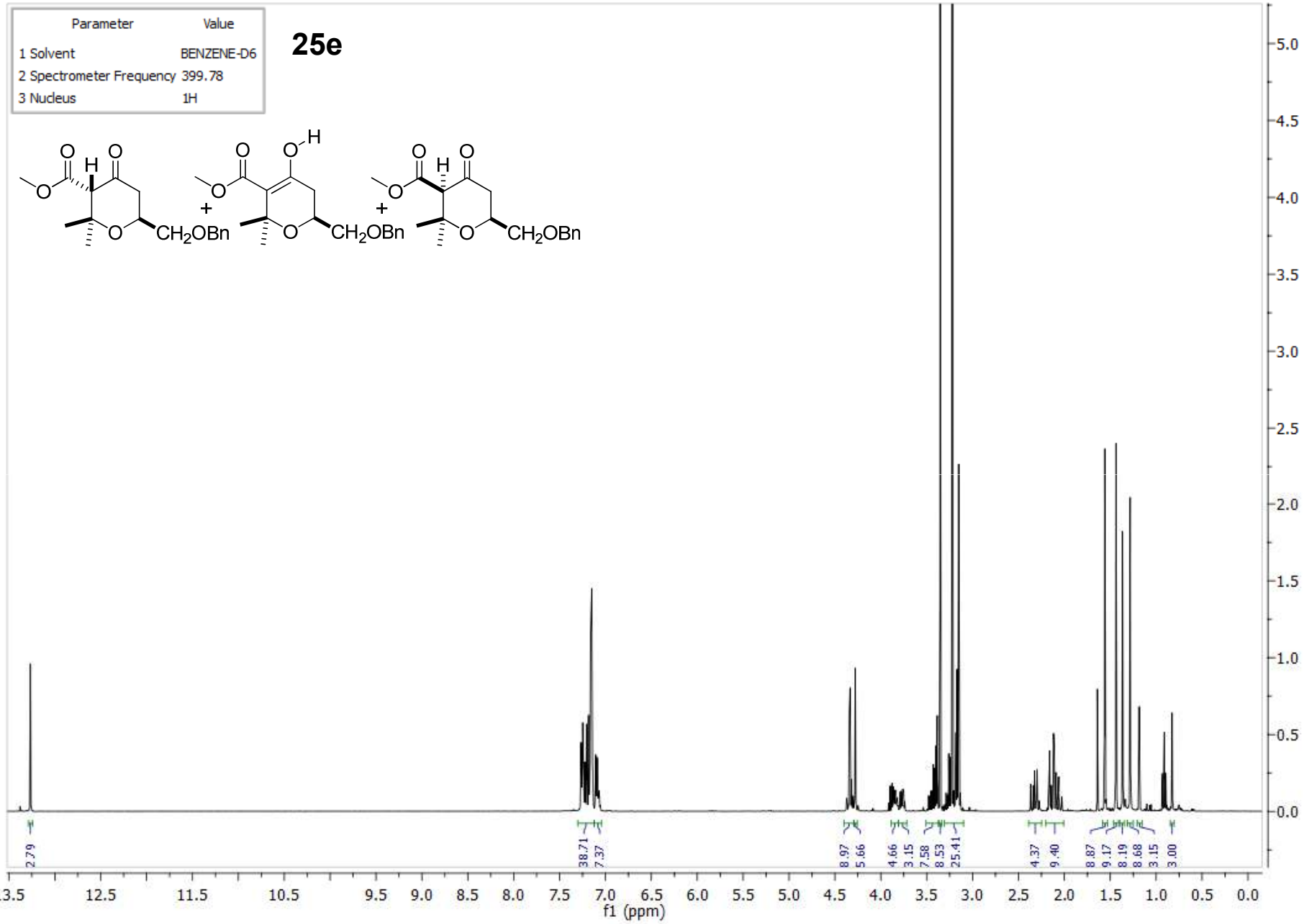

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
25b
Pr
Pr
Pr
1.8
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
-0.1
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

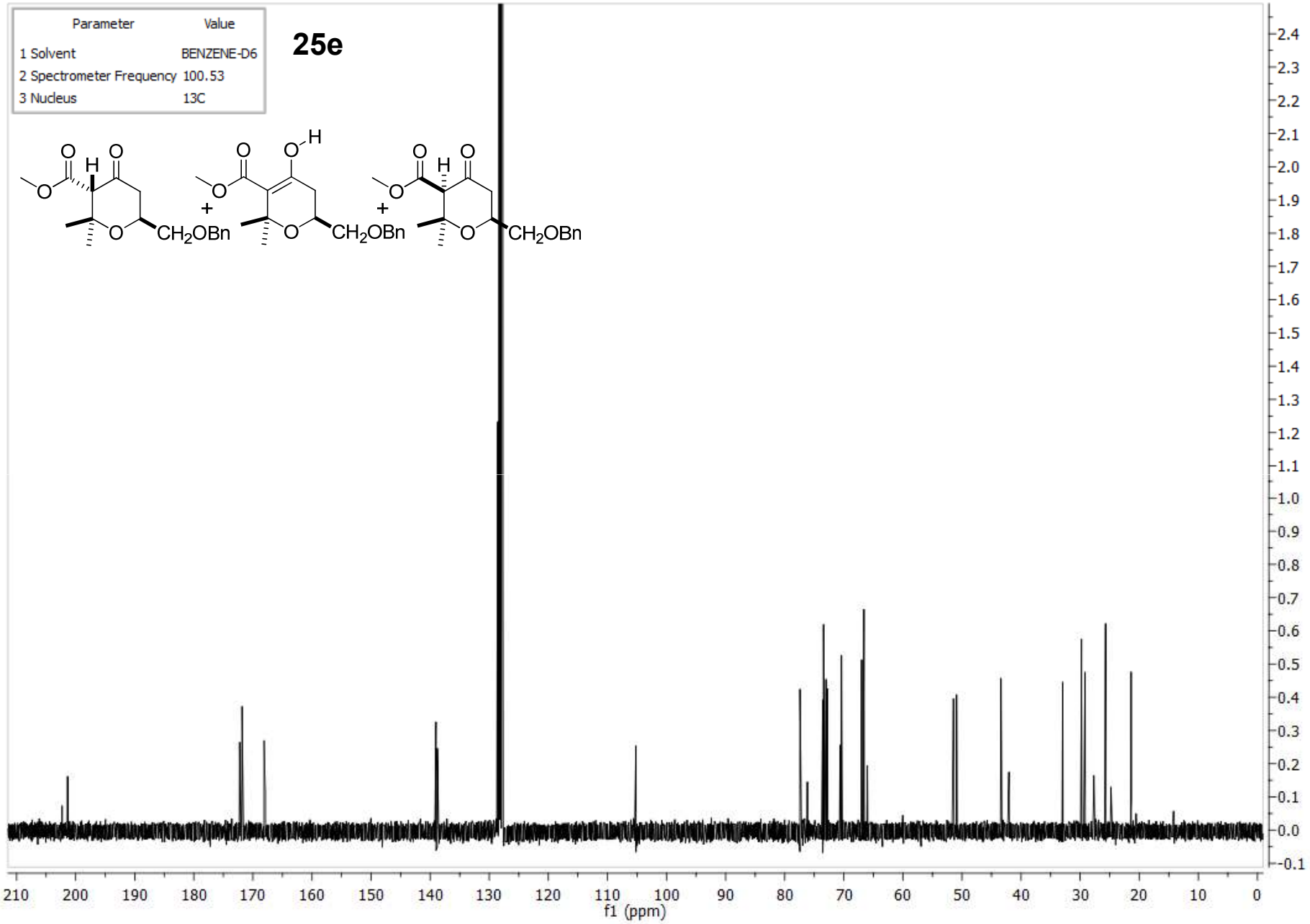

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
25c
iPr
iPr
iPr
2.13
6.43
9.74
0.80
4.73
1.00
3.48
4.26
3.19
9.33
8.71
4.85
5.20
2.55
10.36
2.33
13.71
4.60
f1 (ppm)

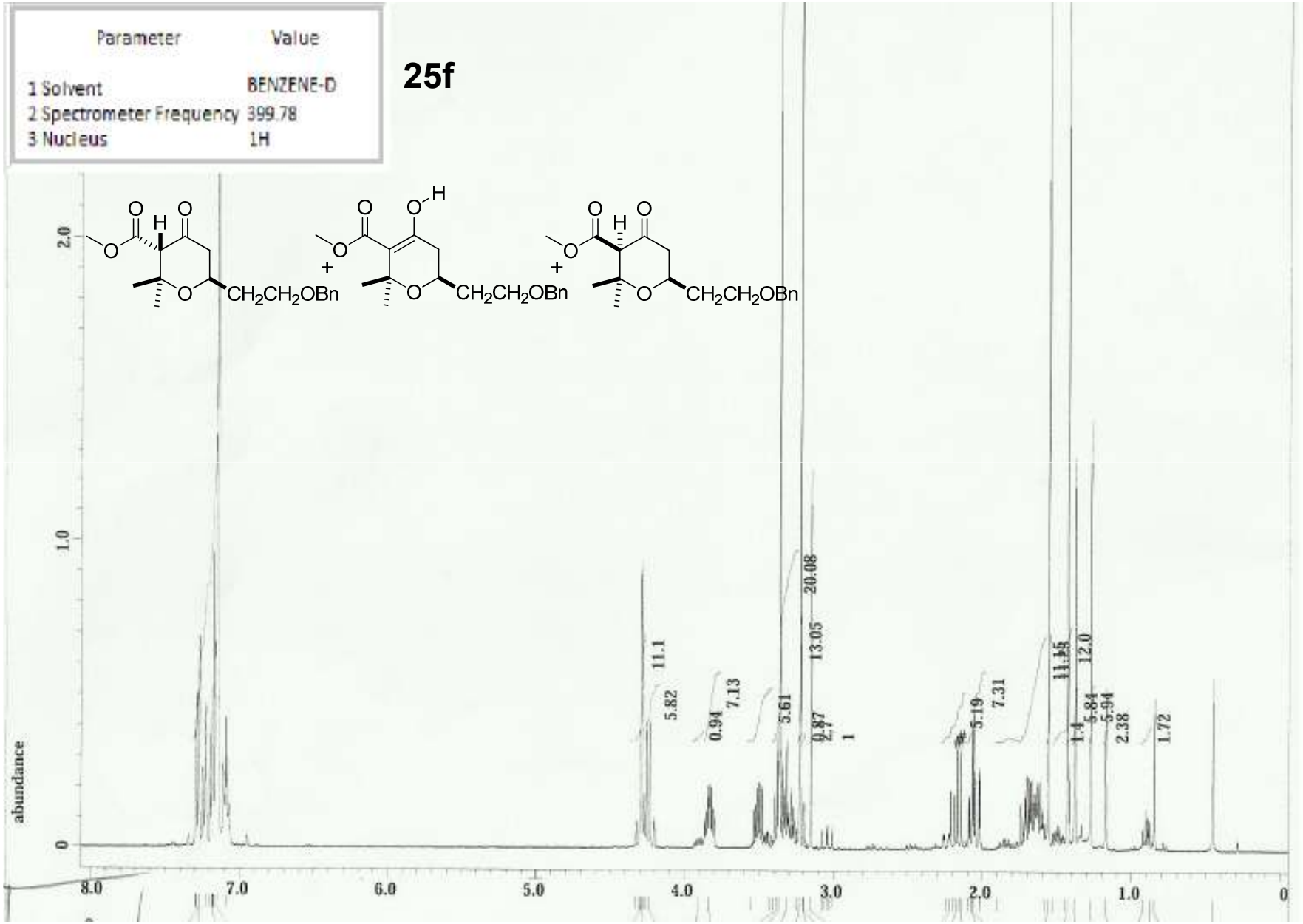

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
25c
iPr
iPr
iPr
f1 (ppm)

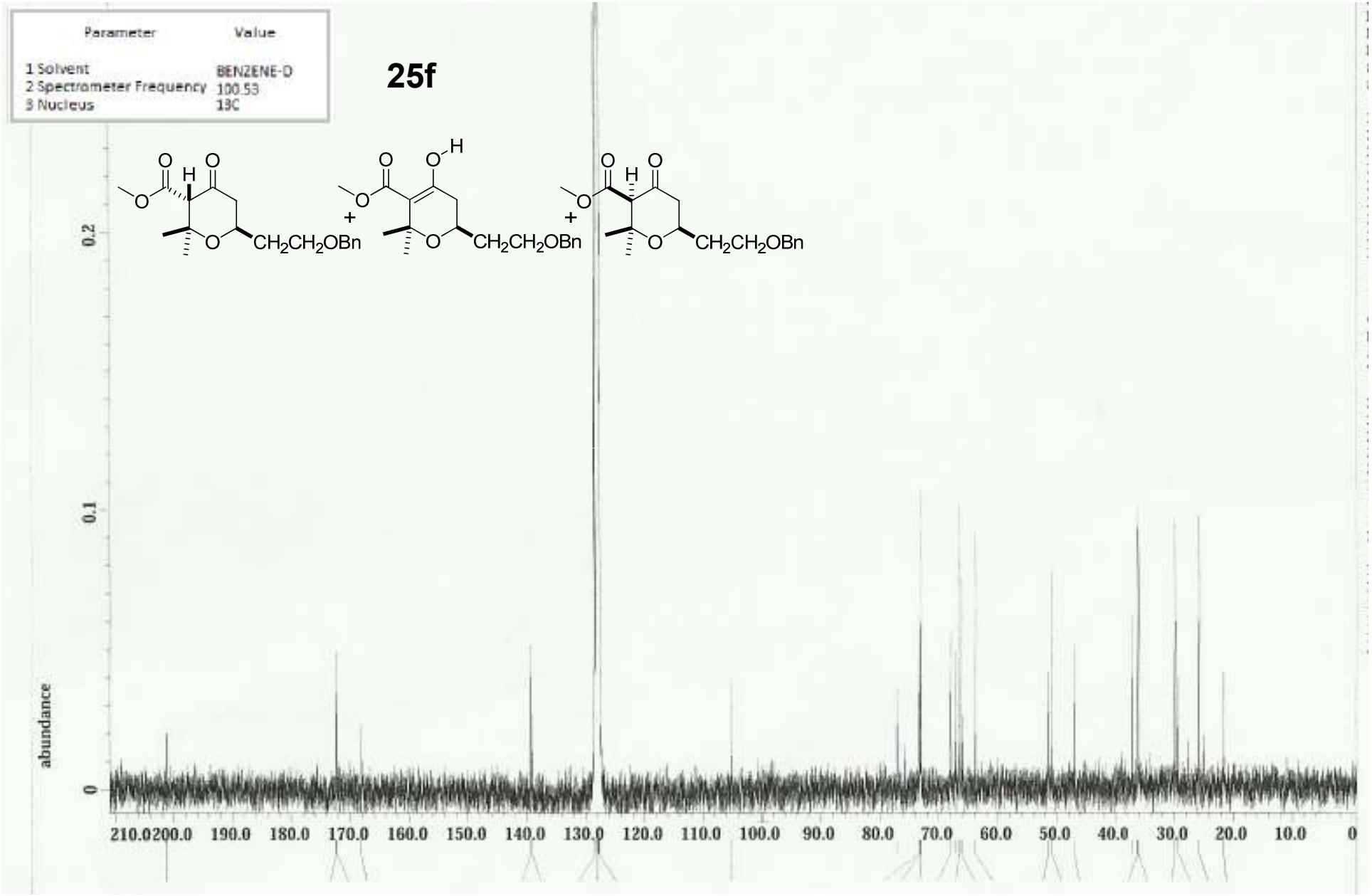

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
25d
fur
fur
fur
2.12
4.59
9.44
4.45
1.00
1.58
4.32
1.29
7.41
2.27
3.10
2.34
4.22
6.88
6.75
4.44
5.58
3.96
1.96
f1 (ppm)

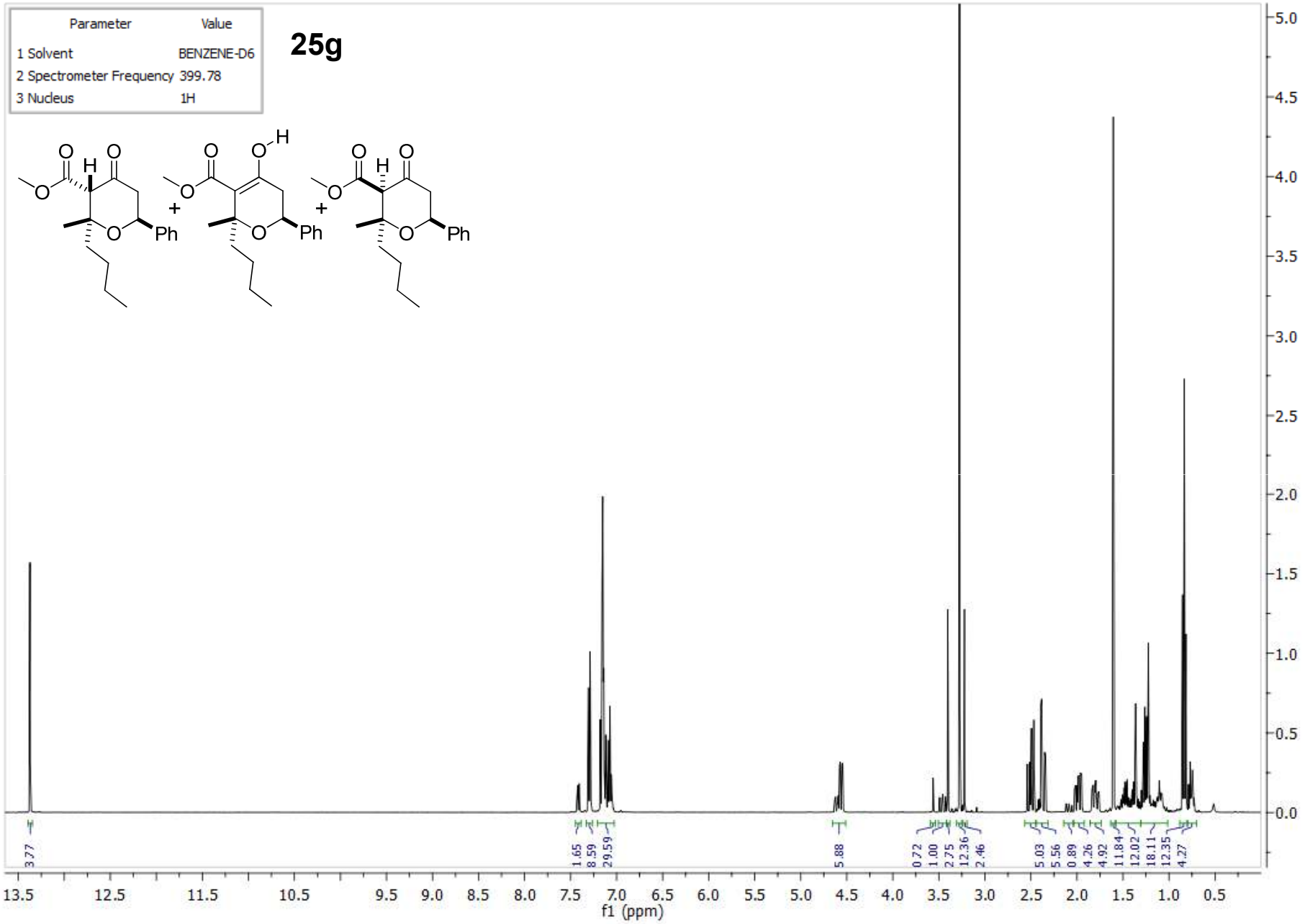

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
25d
fur
fur
fur
f1 (ppm)

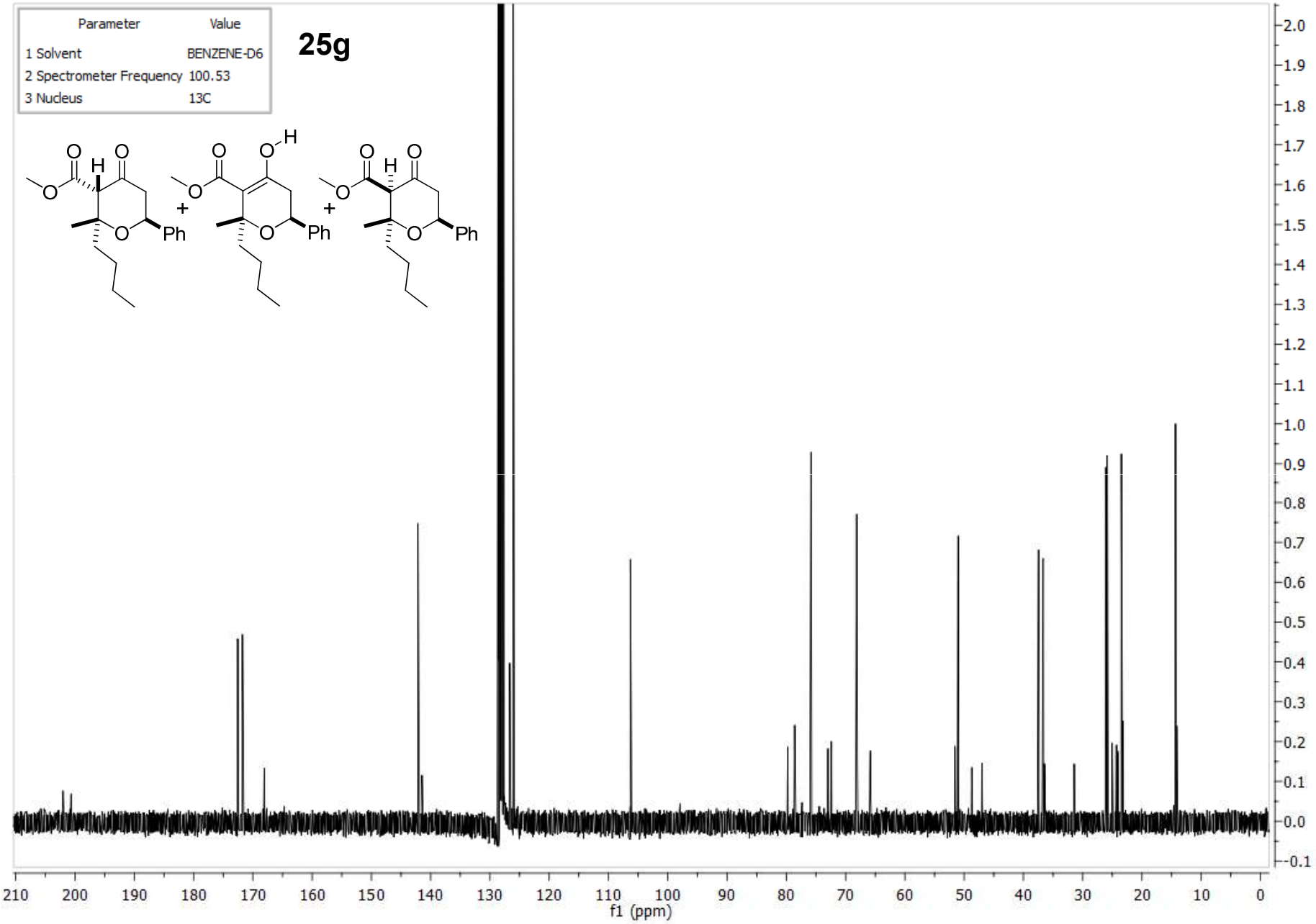

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
25e
CH2OBn
CH2OBn
CH2OBn
2.79
38.71
7.37
8.97
5.66
4.66
3.15
7.58
8.53
25.41
4.37
9.40
8.87
9.17
8.19
8.68
3.15
3.00
f1 (ppm)

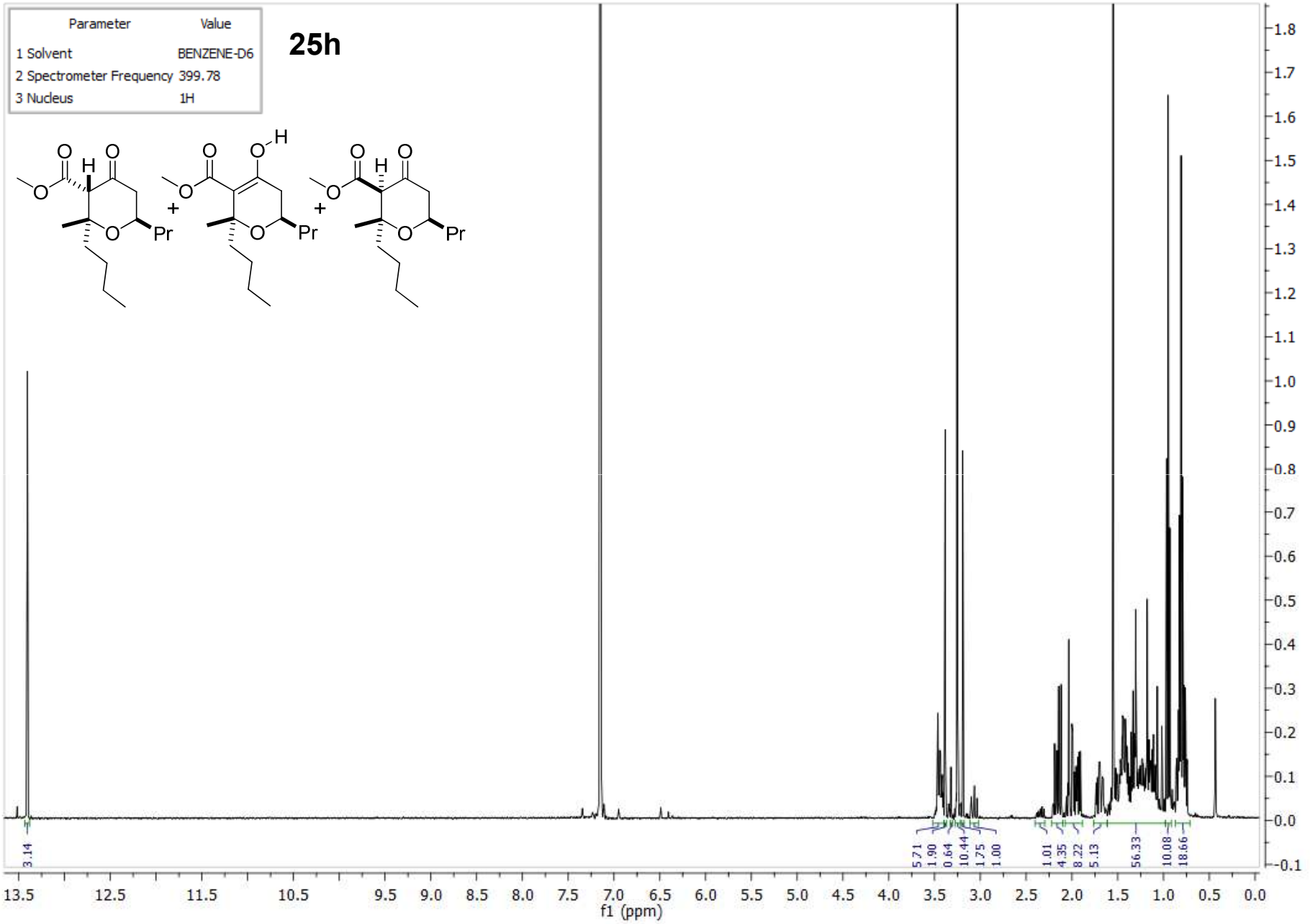

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
25e
CH2OBn
CH2OBn
CH2OBn
f1 (ppm)

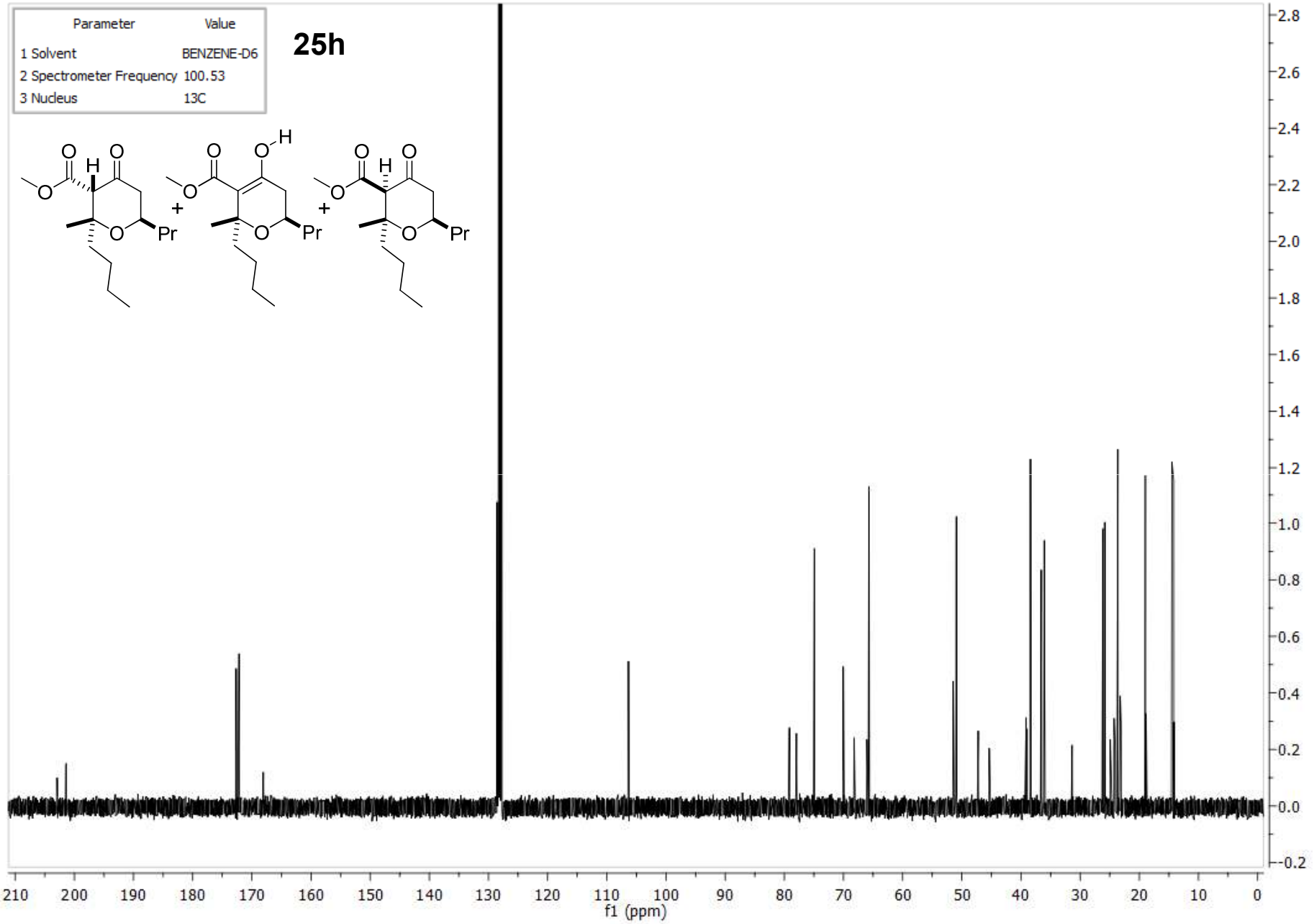

Parameter Value
1 Solvent BENZENE-D
2 Spectrometer Frequency 399.78
3 Nucleus 1H
25f
CH2CH2OBn
CH2CH2OBn
CH2CH2OBn
abundance
11.1
5.82
0.94
7.13
5.61
20.08
13.05
5.19
7.31
12.0
5.84
5.94
2.38
1.72
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0

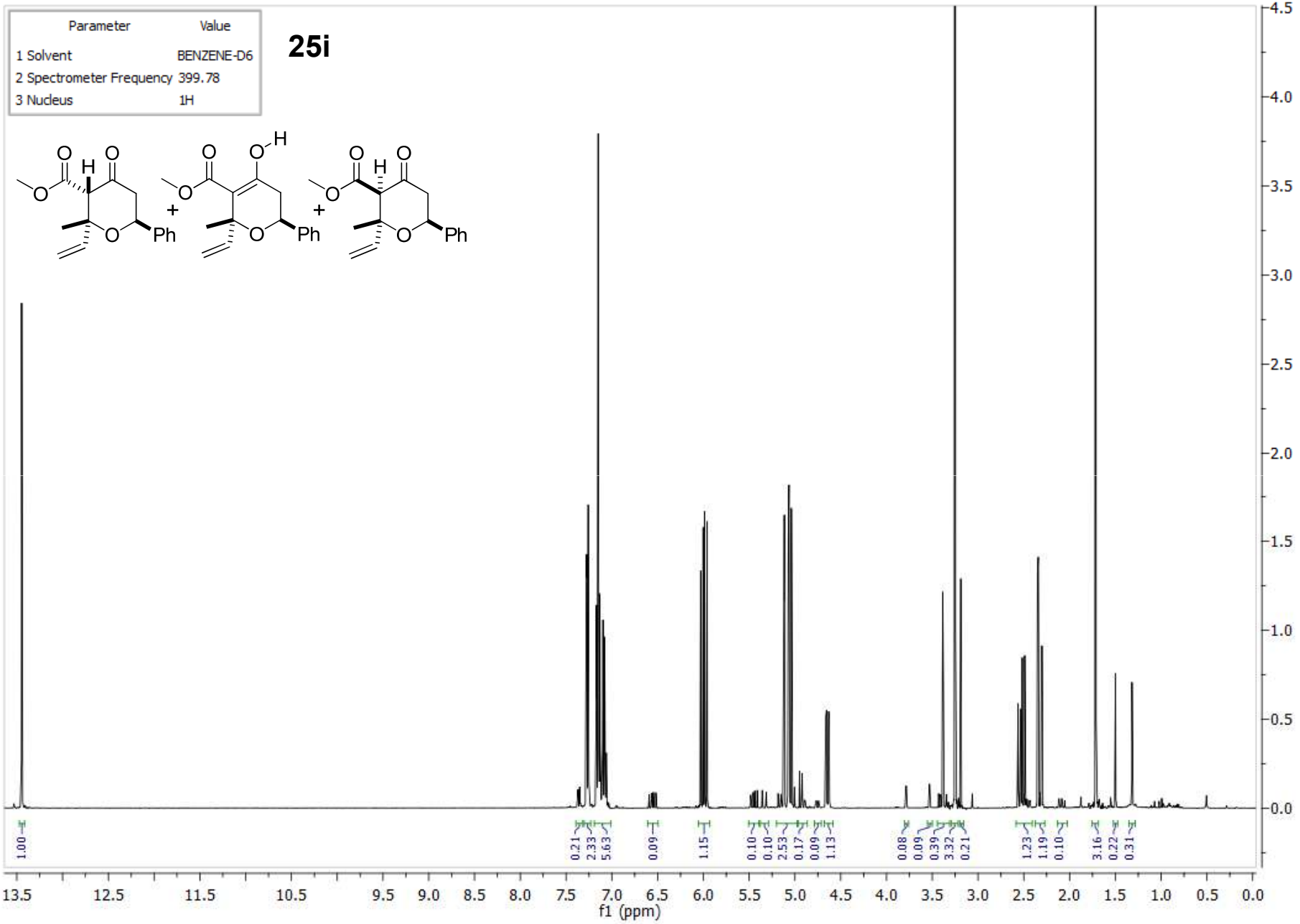

Parameter Value
1 Solvent BENZENE-D
2 Spectrometer Frequency 100.53
3 Nucleus 13C
25f
CH2CH2OBn
CH2CH2OBn
CH2CH2OBn
abundance
0.2
0.1
0
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0

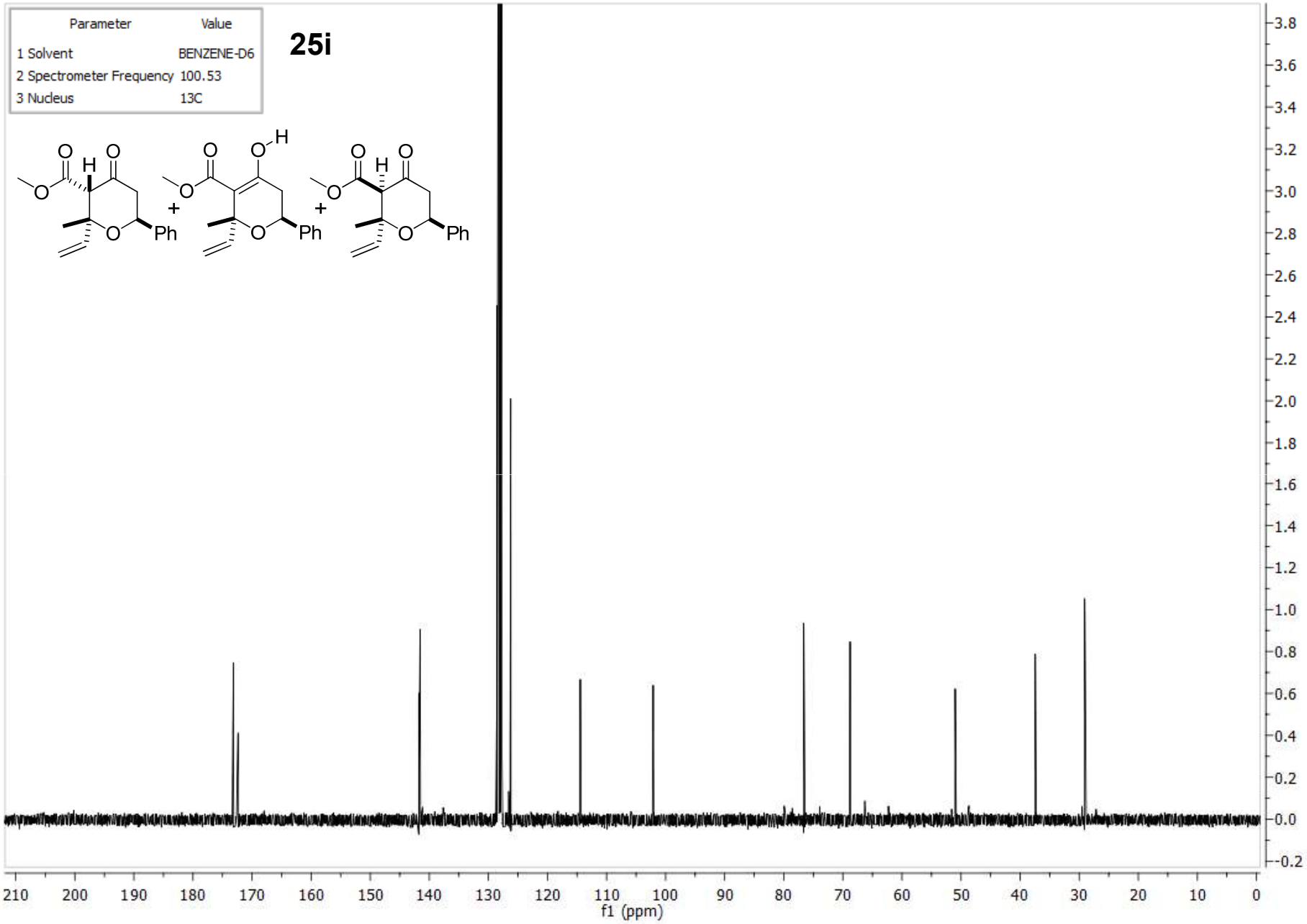

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
25g
Ph
Ph
Ph
3.77
1.65
8.59
29.59
5.88
0.72
1.00
2.75
12.36
2.46
5.03
5.56
0.89
4.26
4.92
11.84
12.02
18.11
12.35
4.27
f1 (ppm)

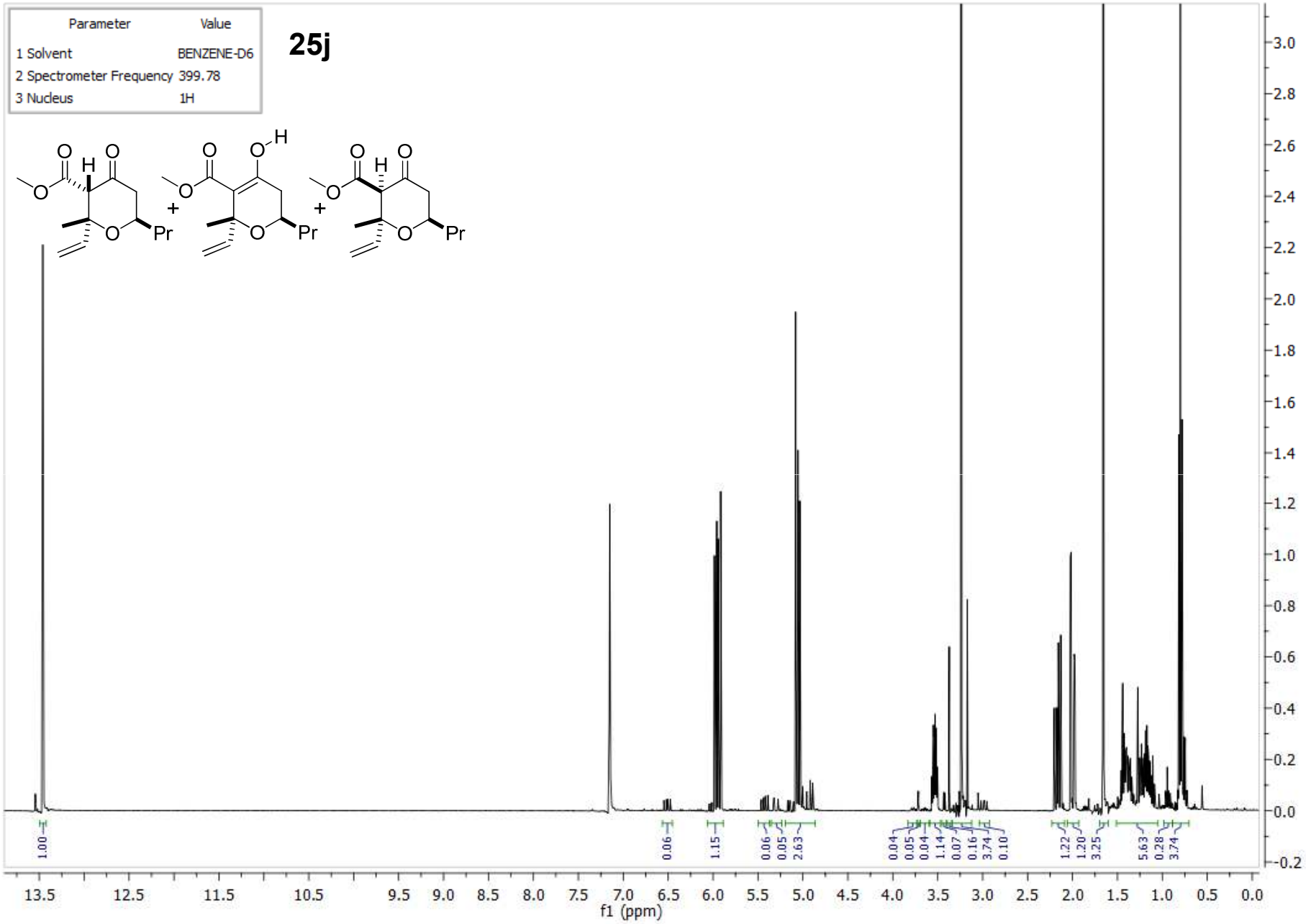

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
25g
Ph
Ph
Ph
f1 (ppm)

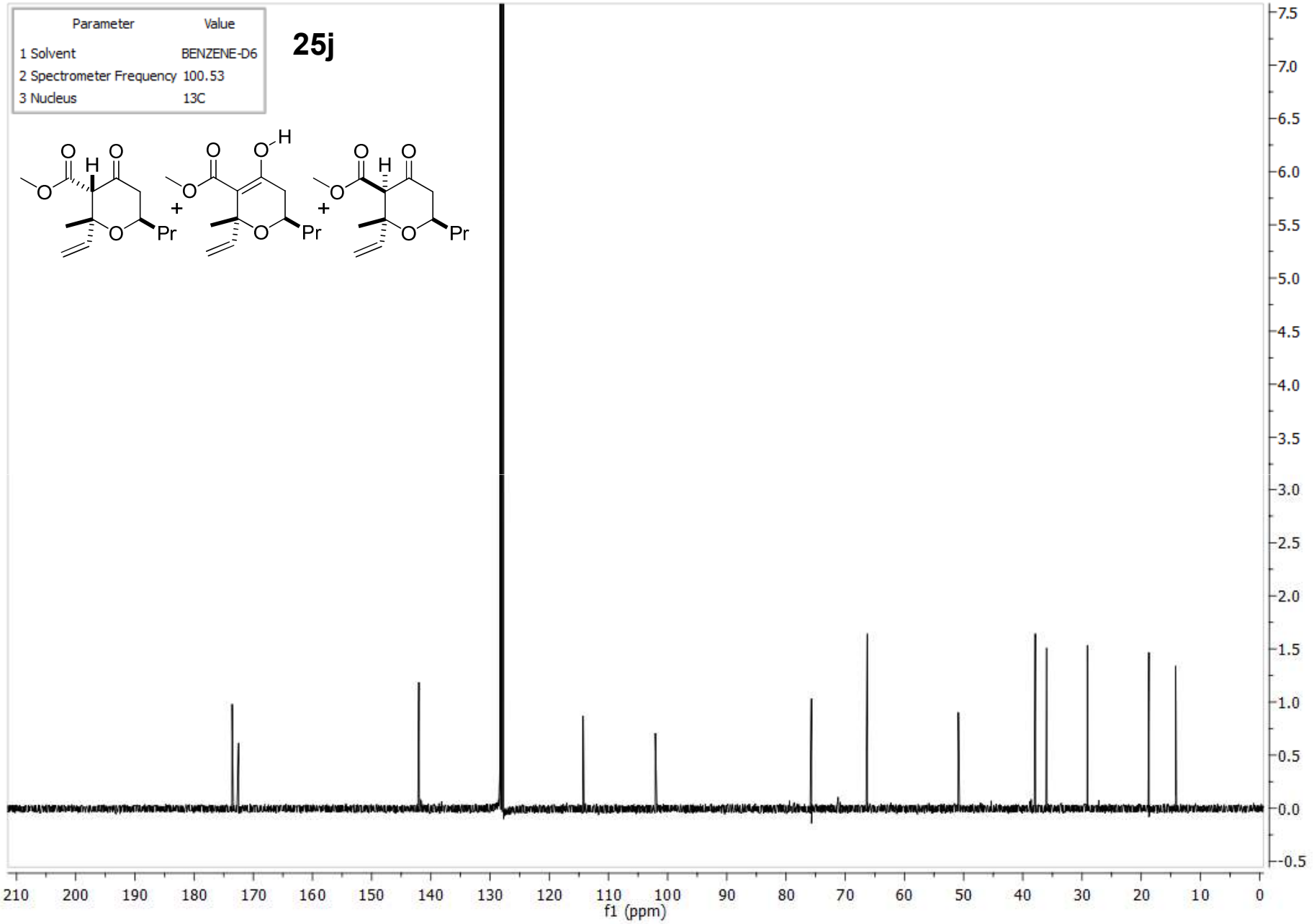

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
25h
Pr
Pr
Pr
3.14
5.71
1.90
0.64
10.44
1.75
1.00
1.01
4.35
8.22
5.13
56.33
10.08
18.66
f1 (ppm)

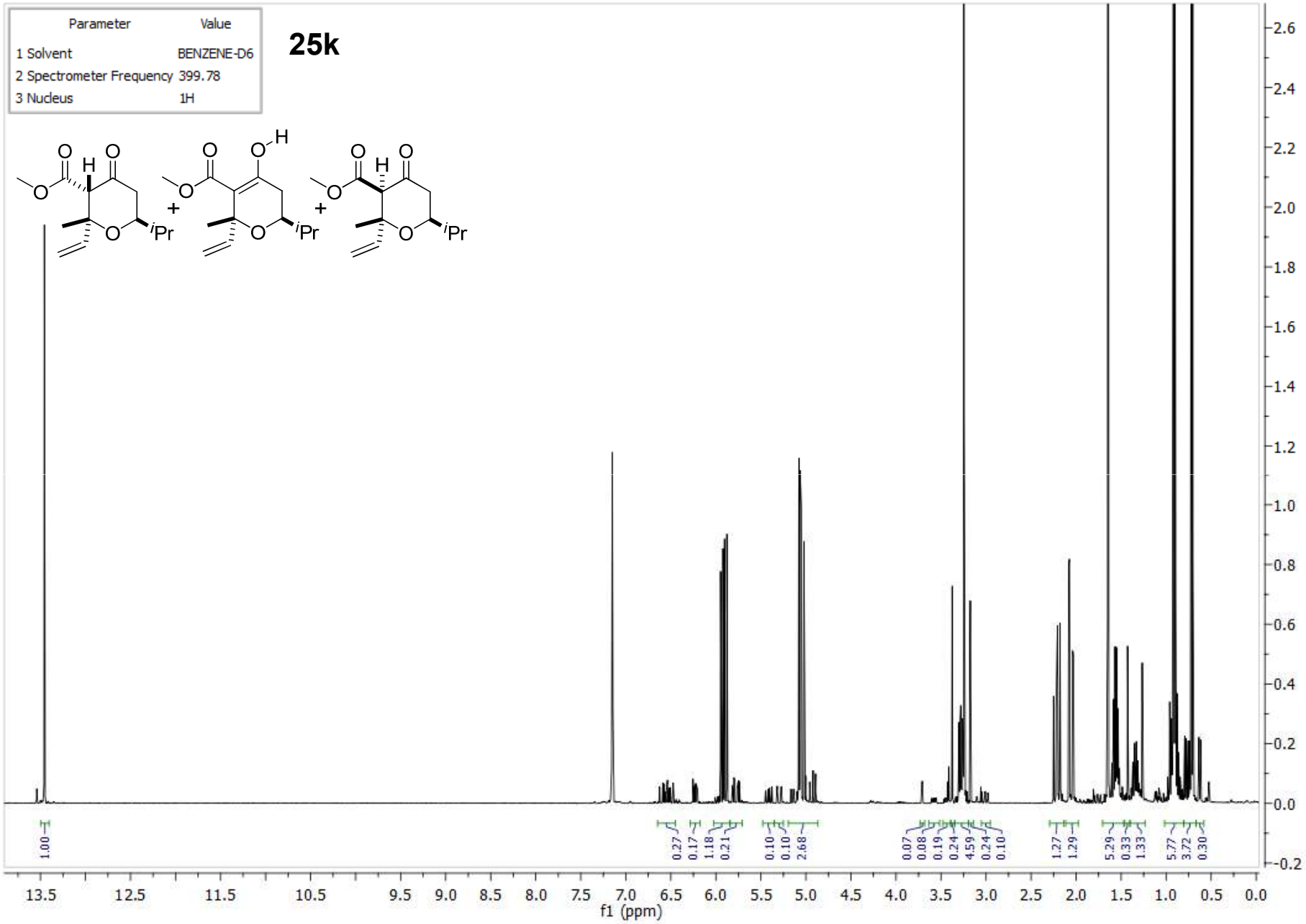

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
25h
Pr
Pr
Pr
f1 (ppm)

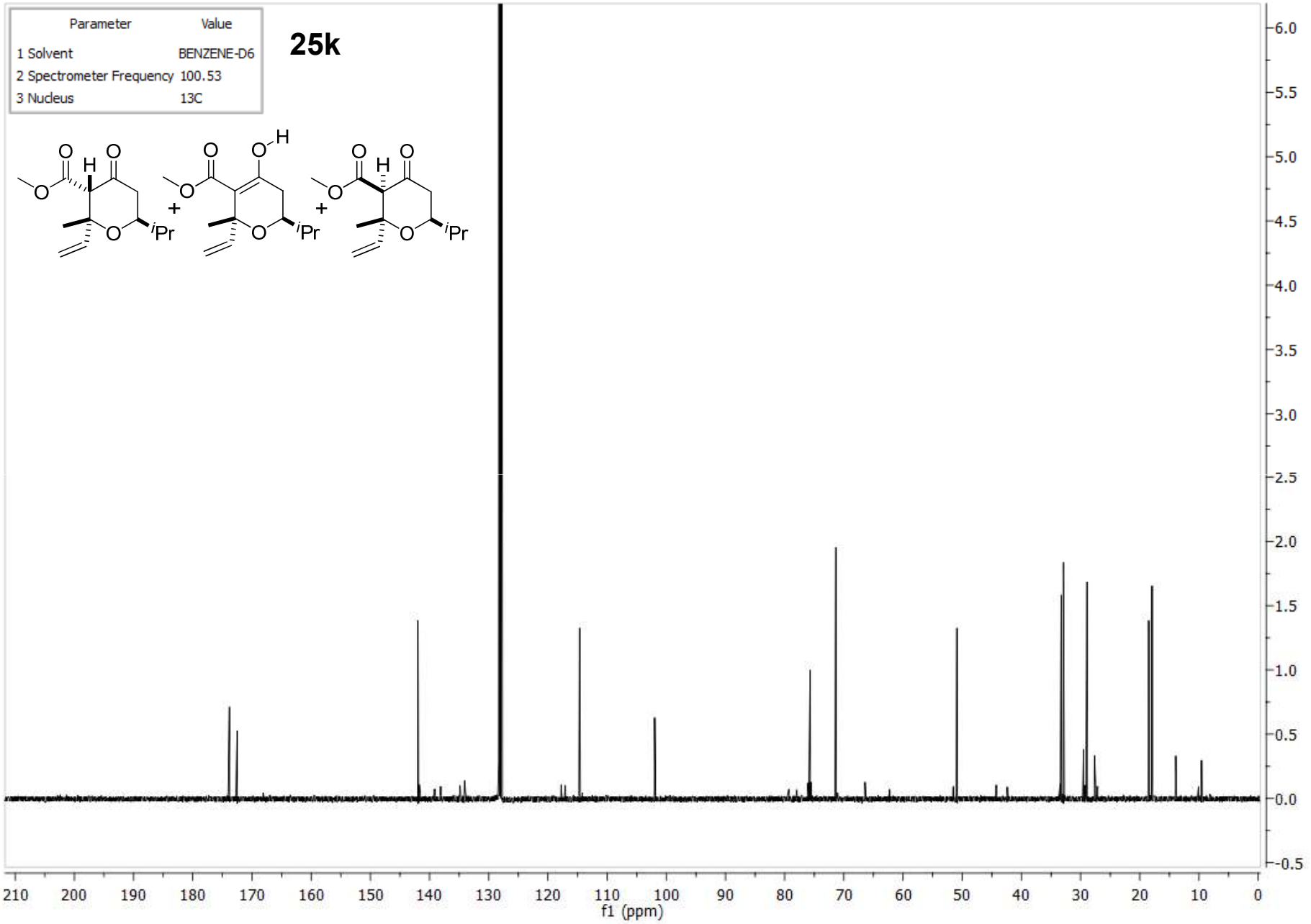

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
25i
Ph
Ph
Ph
1.00
0.21
2.33
5.63
0.09
1.15
0.10
0.10
2.53
0.17
0.09
1.13
0.08
0.09
0.39
3.32
0.21
1.23
1.19
0.10
3.16
0.22
0.31
f1 (ppm)

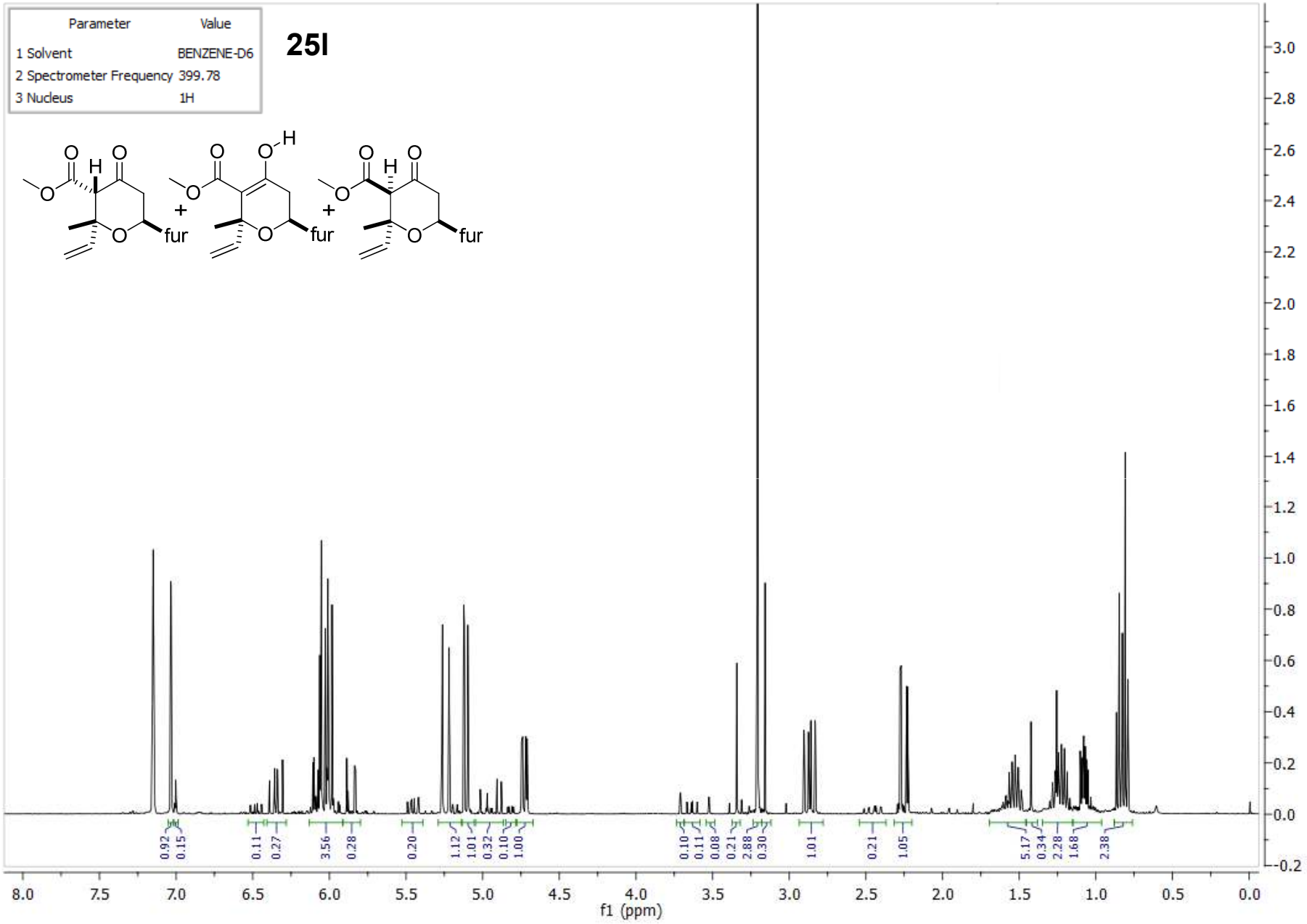

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
25i
Ph
Ph
Ph
f1 (ppm)

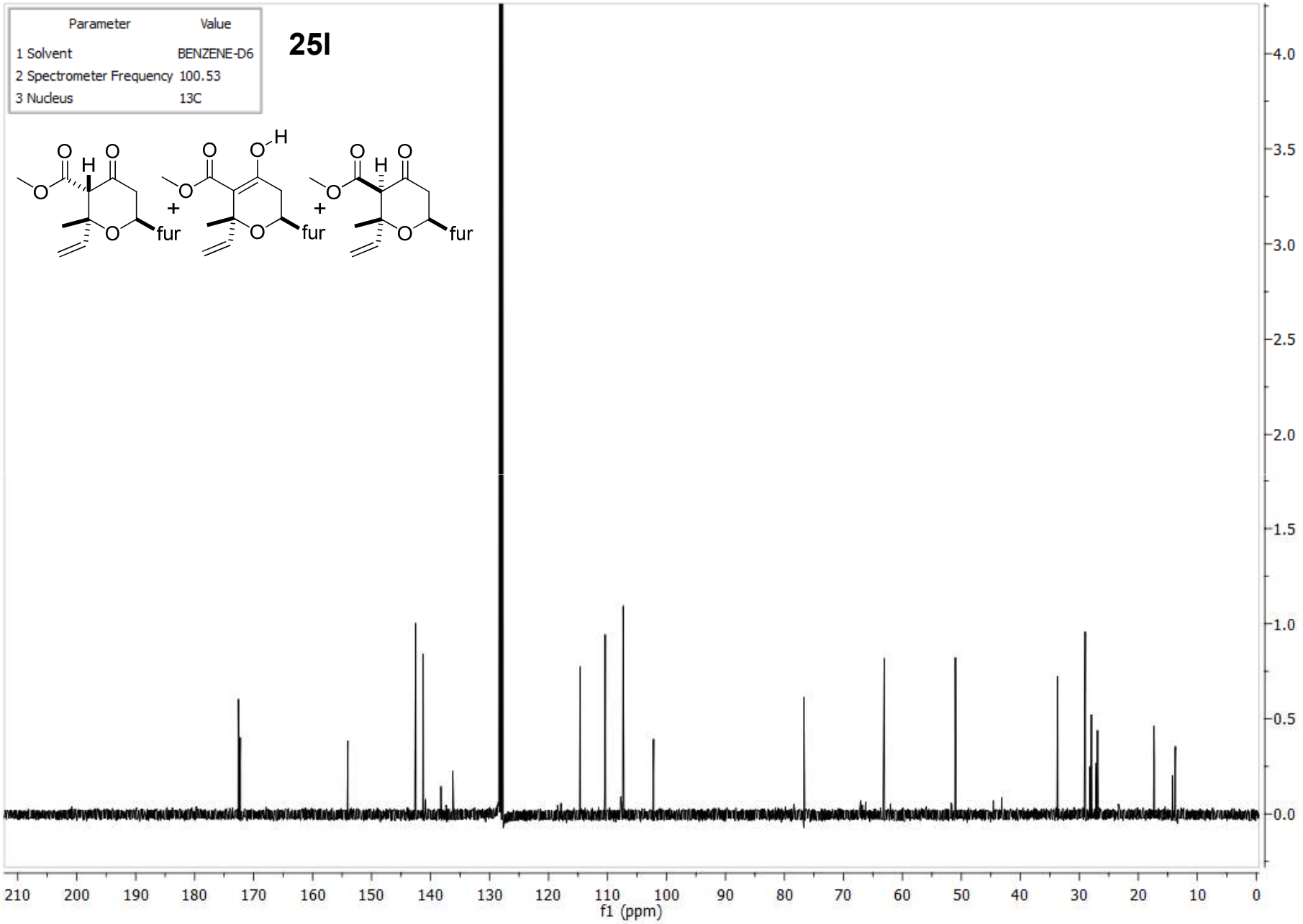

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
25j
Pr
Pr
Pr
1.00
0.06
1.15
0.06
0.05
2.63
0.04
0.05
0.04
1.14
0.07
0.16
3.74
0.10
1.22
1.20
3.25
5.63
0.28
3.74
f1 (ppm)

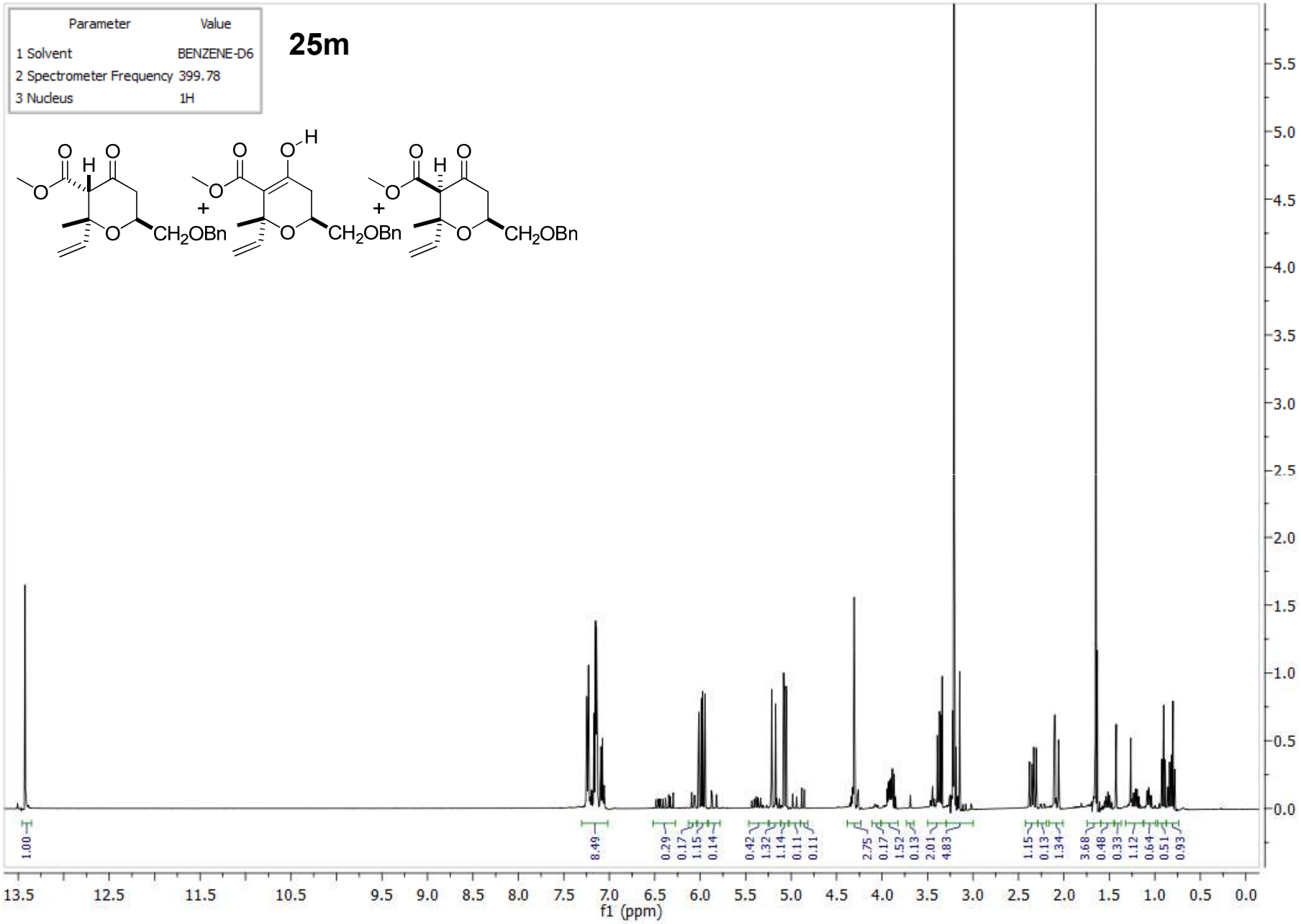

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
25j
Pr
Pr
Pr
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

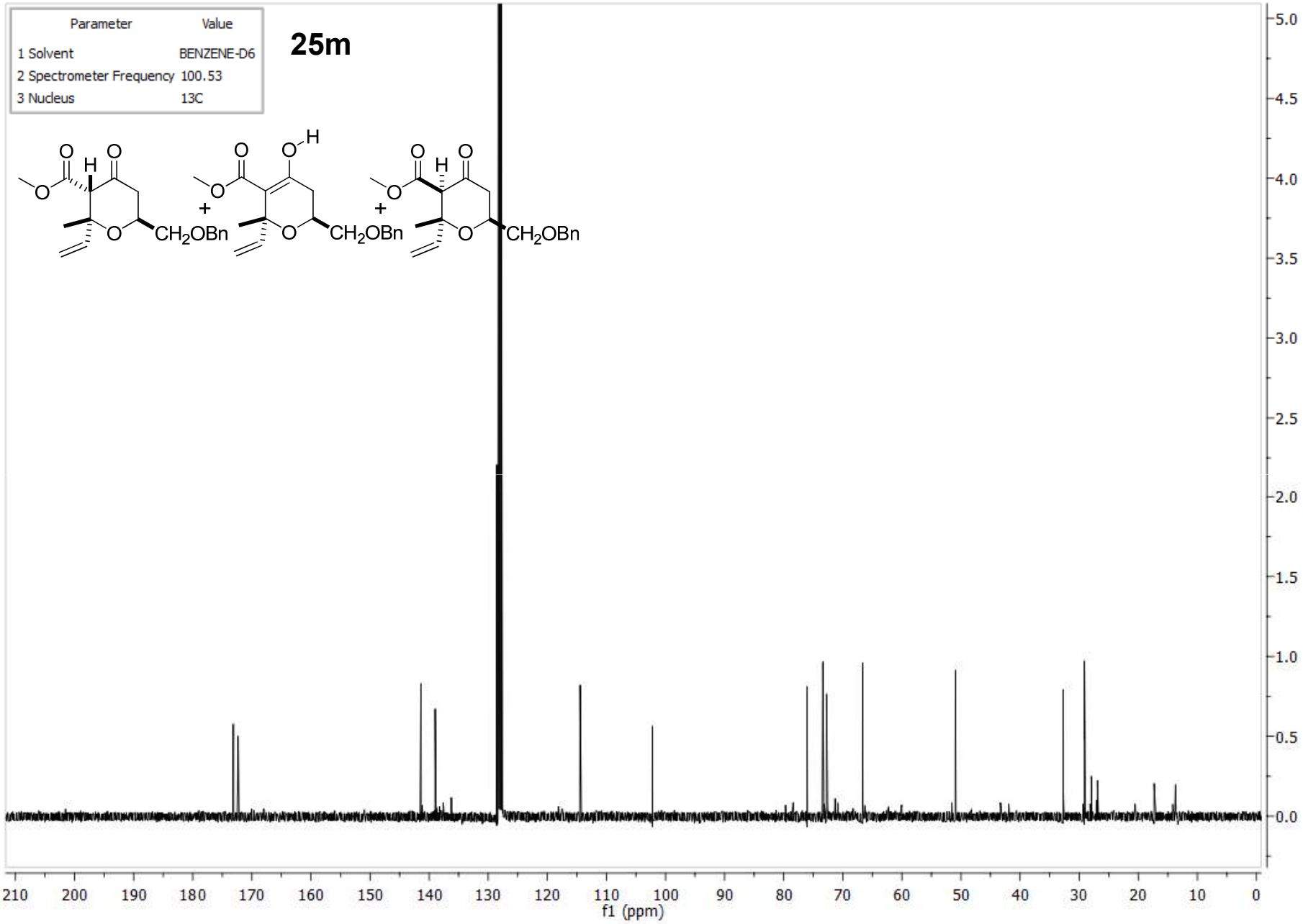

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
25k
iPr
iPr
iPr
1.00
0.27
0.17
1.18
0.21
0.10
0.10
2.68
0.07
0.08
0.19
0.24
4.59
0.24
0.10
1.27
1.29
5.29
0.33
1.33
5.77
3.72
0.30
f1 (ppm)


Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
25k
iPr
iPr
iPr
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
25l
fur
fur
fur
0.92 0.15 0.11 0.27 3.56 0.28 0.20 1.12 1.01 0.32 0.10 1.00 0.10 0.11 0.08 0.21 2.88 0.30 1.01 0.21 1.05 5.17 0.34 2.28 1.68 2.38
f1 (ppm)

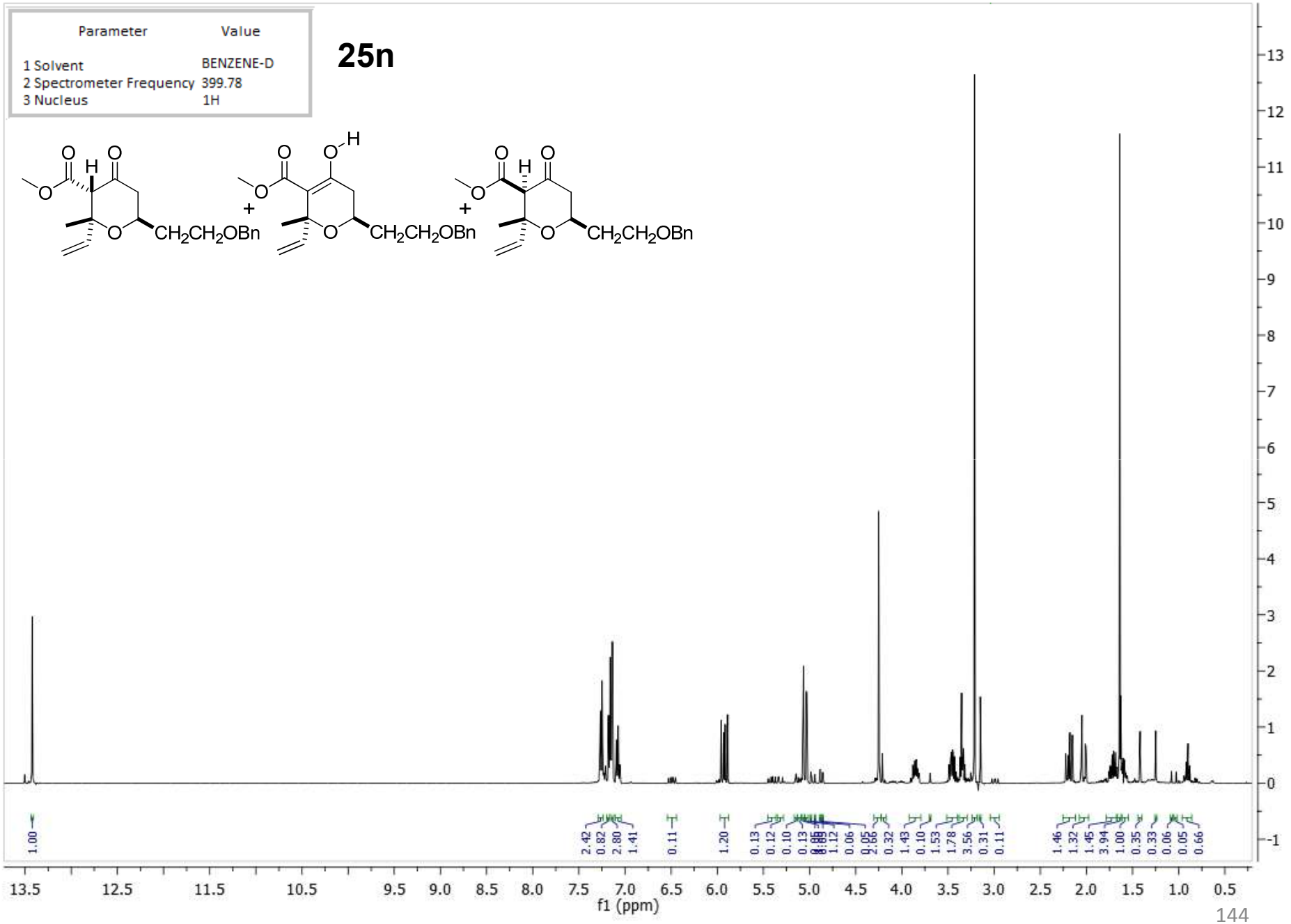

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
25l
fur
fur
fur
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

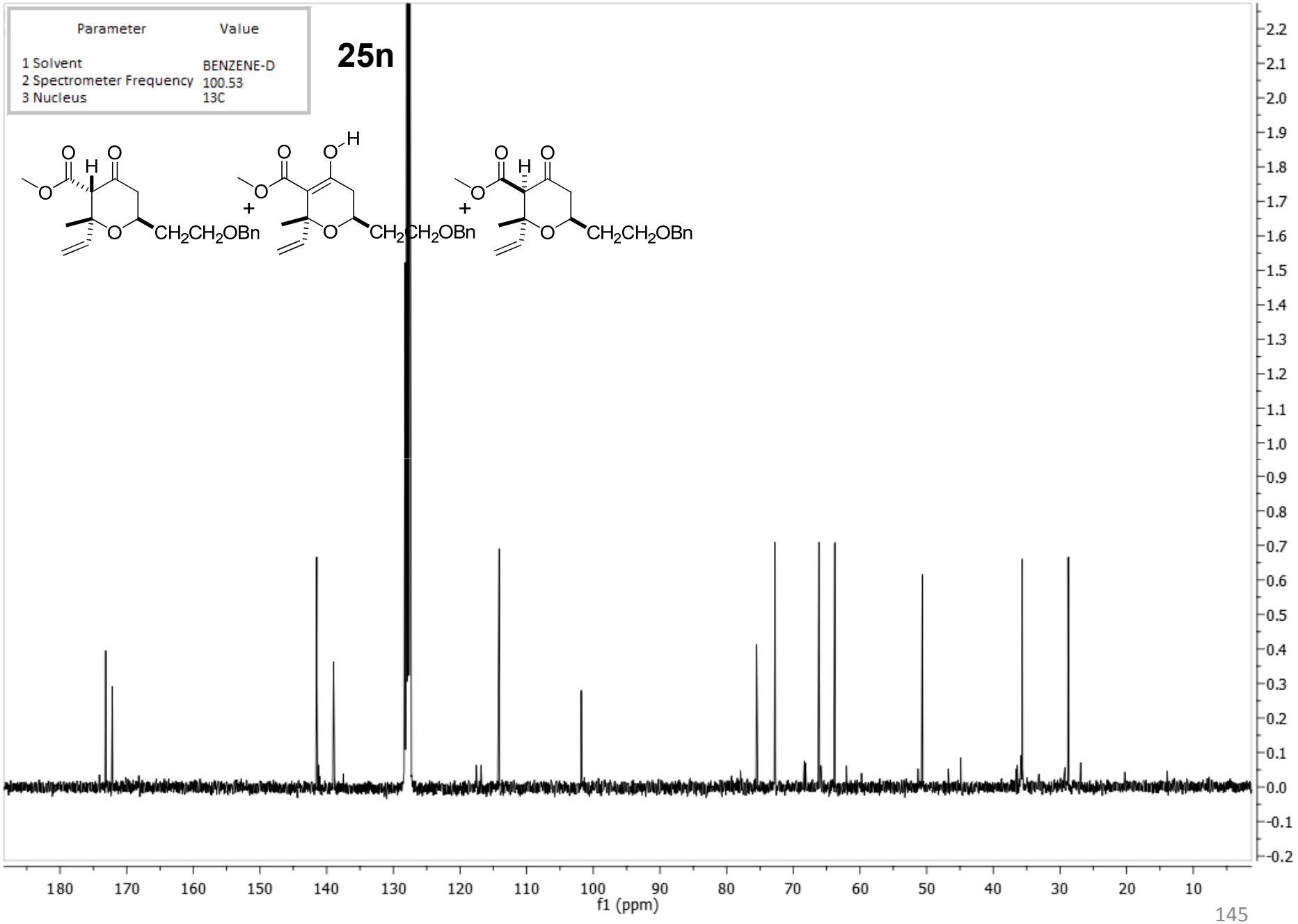

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
25m
CH2OBn
CH2OBn
CH2OBn
1.00
8.49
0.29
0.17
1.15
0.14
0.42
1.32
1.14
0.11
0.11
2.75
0.17
1.52
0.13
2.01
4.83
1.15
0.13
1.34
3.68
0.48
0.33
1.12
0.64
0.51
0.93
f1 (ppm)


Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
25m
CH2OBn
CH2OBn
CH2OBn
f1 (ppm)

Parameter	Value
1 Solvent	BENZENE-D
2 Spectrometer Frequency	399.78
3 Nucleus	1H

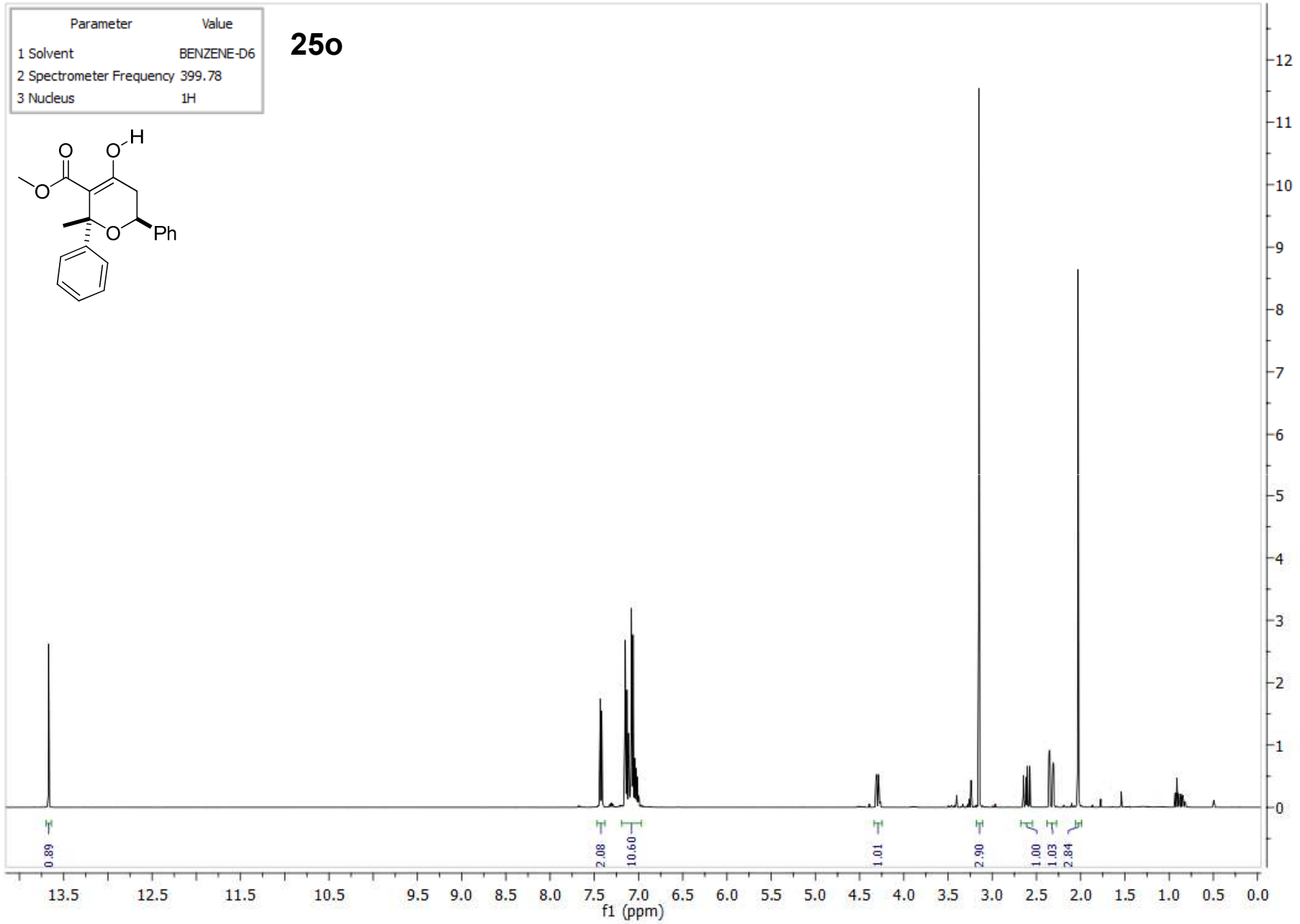
25n

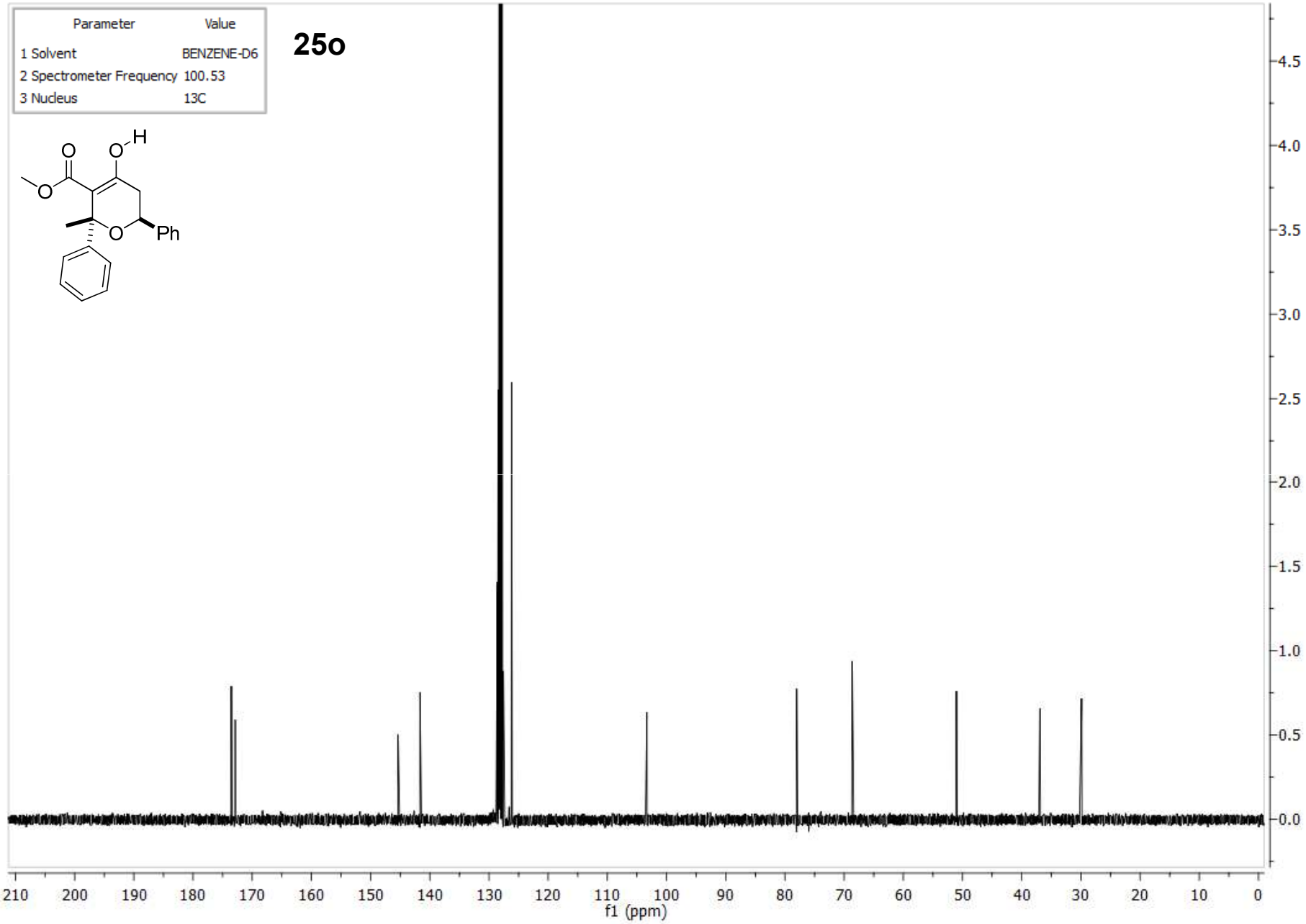


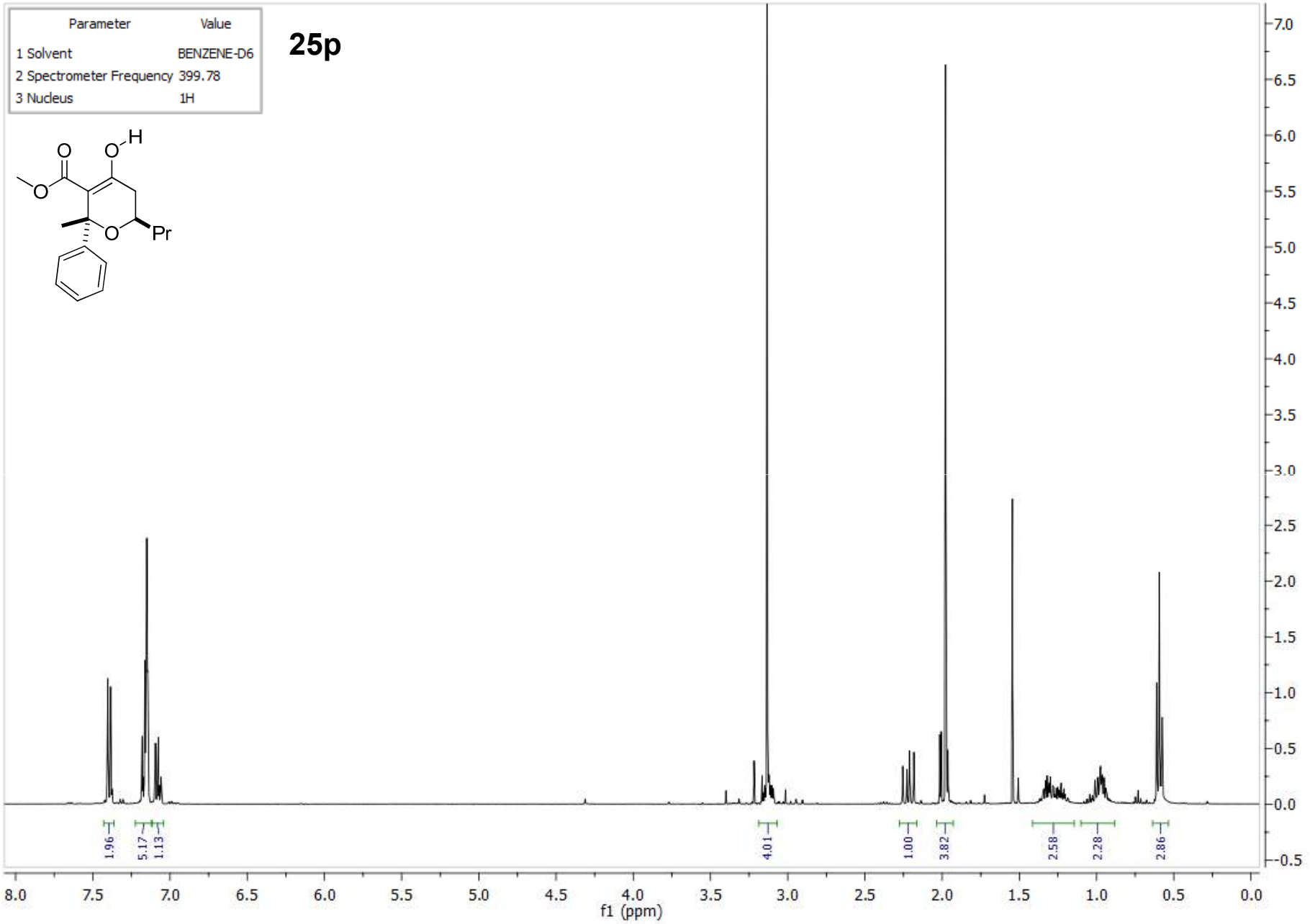

Parameter
Value
1 Solvent
BENZENE-D
2 Spectrometer Frequency
100.53
3 Nucleus
13C
25n
CH2CH2OBn
CH2CH2OBn
CH2CH2OBn
f1 (ppm)

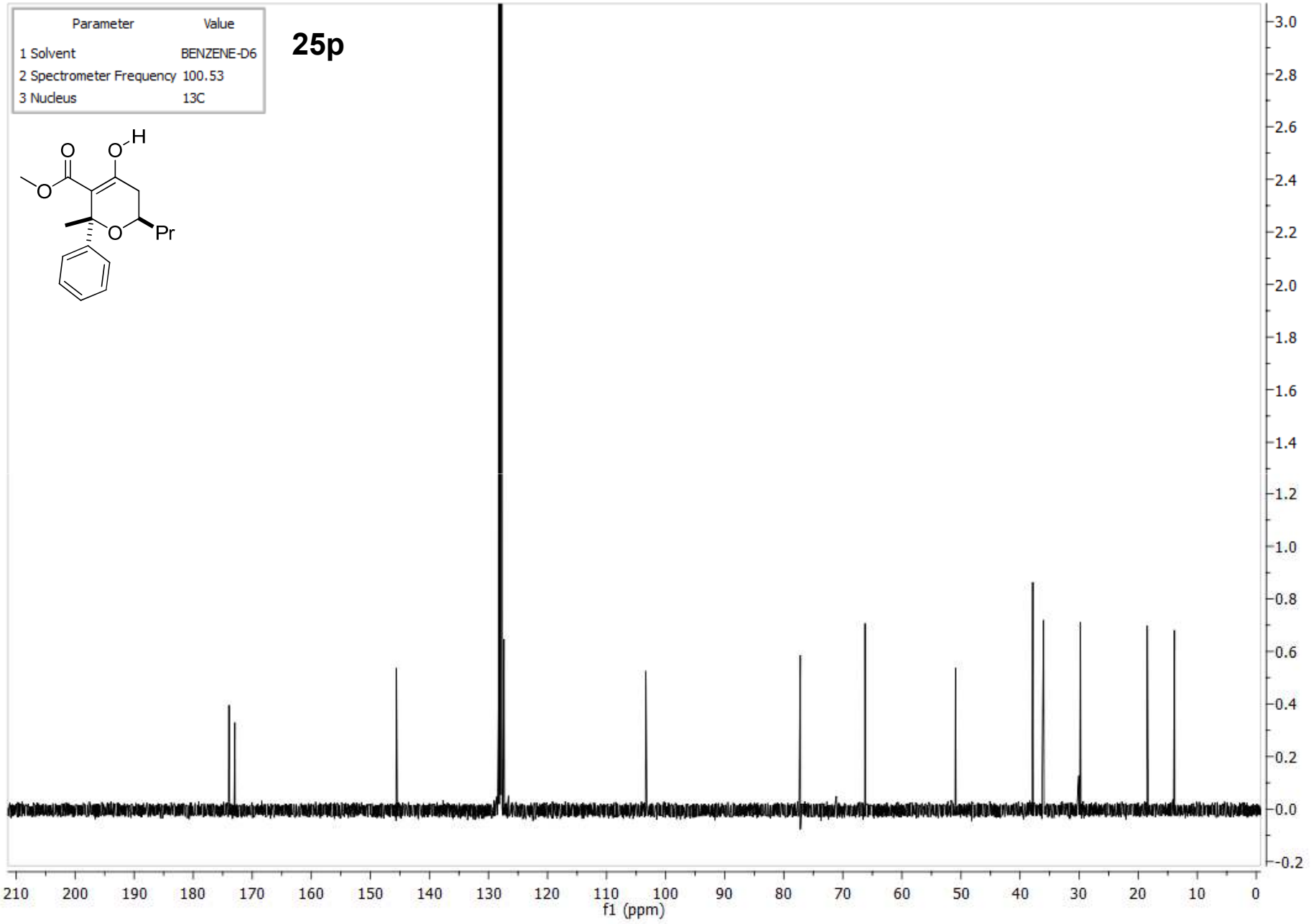
Parameter	Value
1 Solvent	BENZENE-D6
2 Spectrometer Frequency	399.78
3 Nucleus	1H

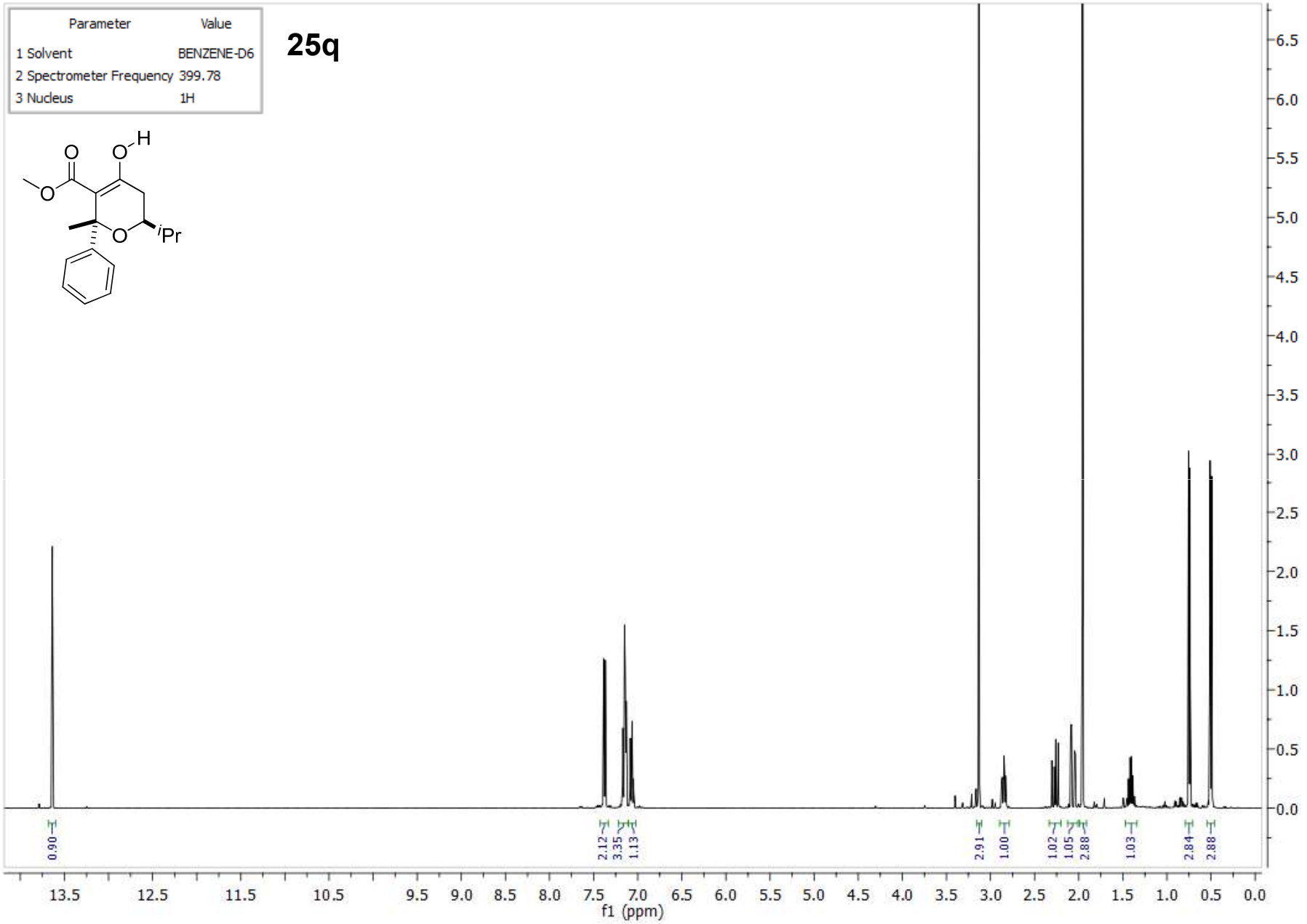
25o

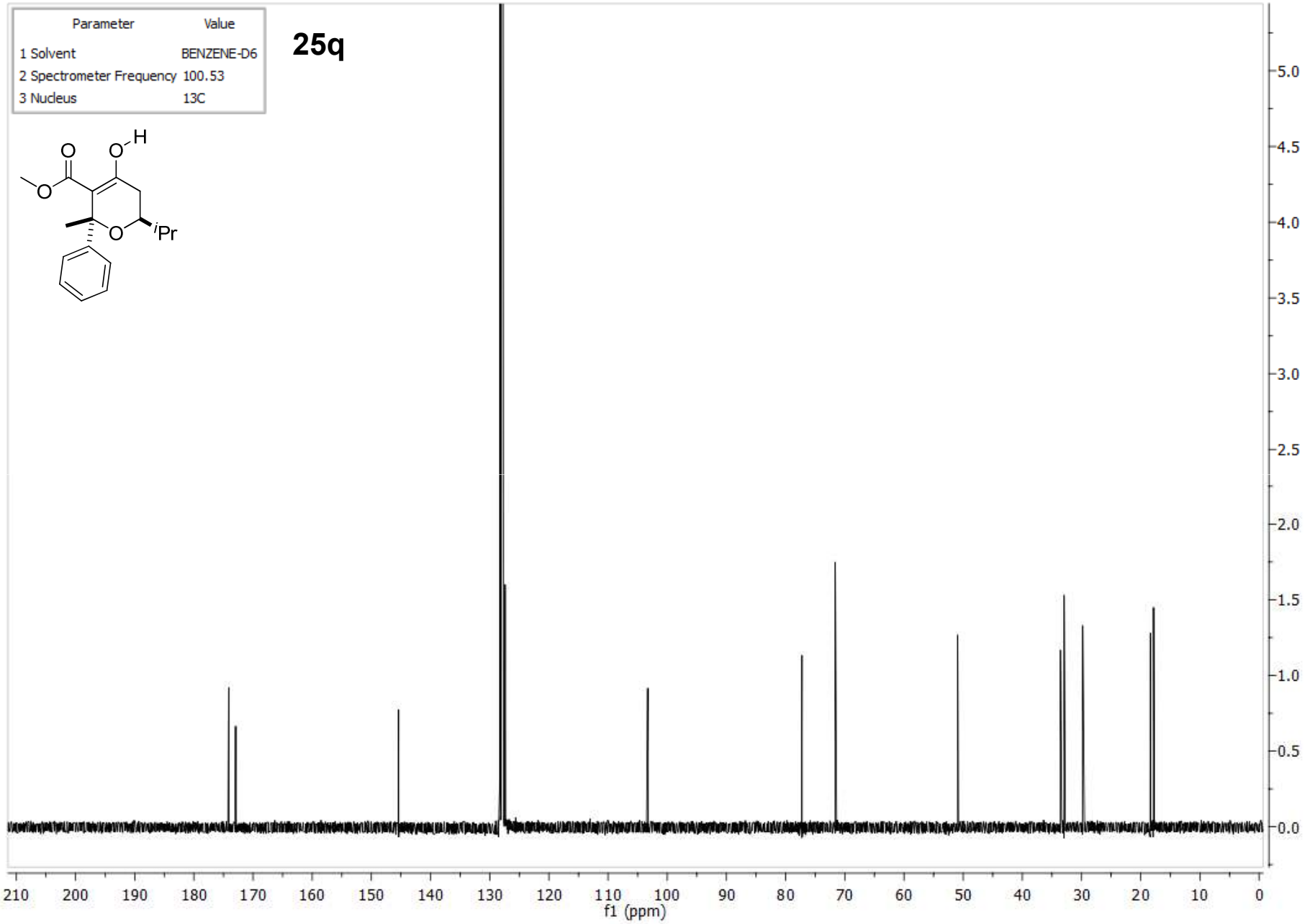


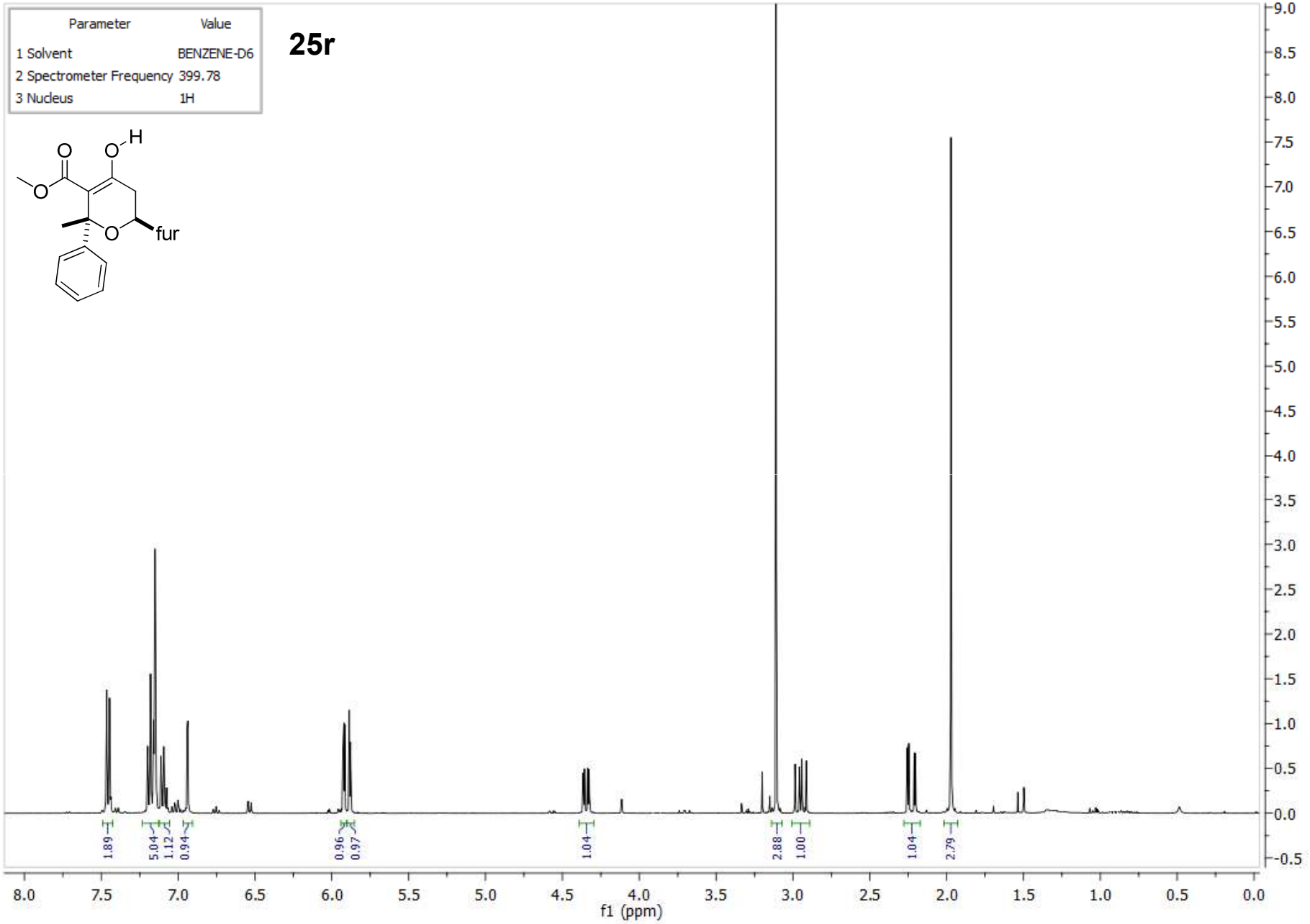

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
25o
O
O
O
H
O
Ph
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

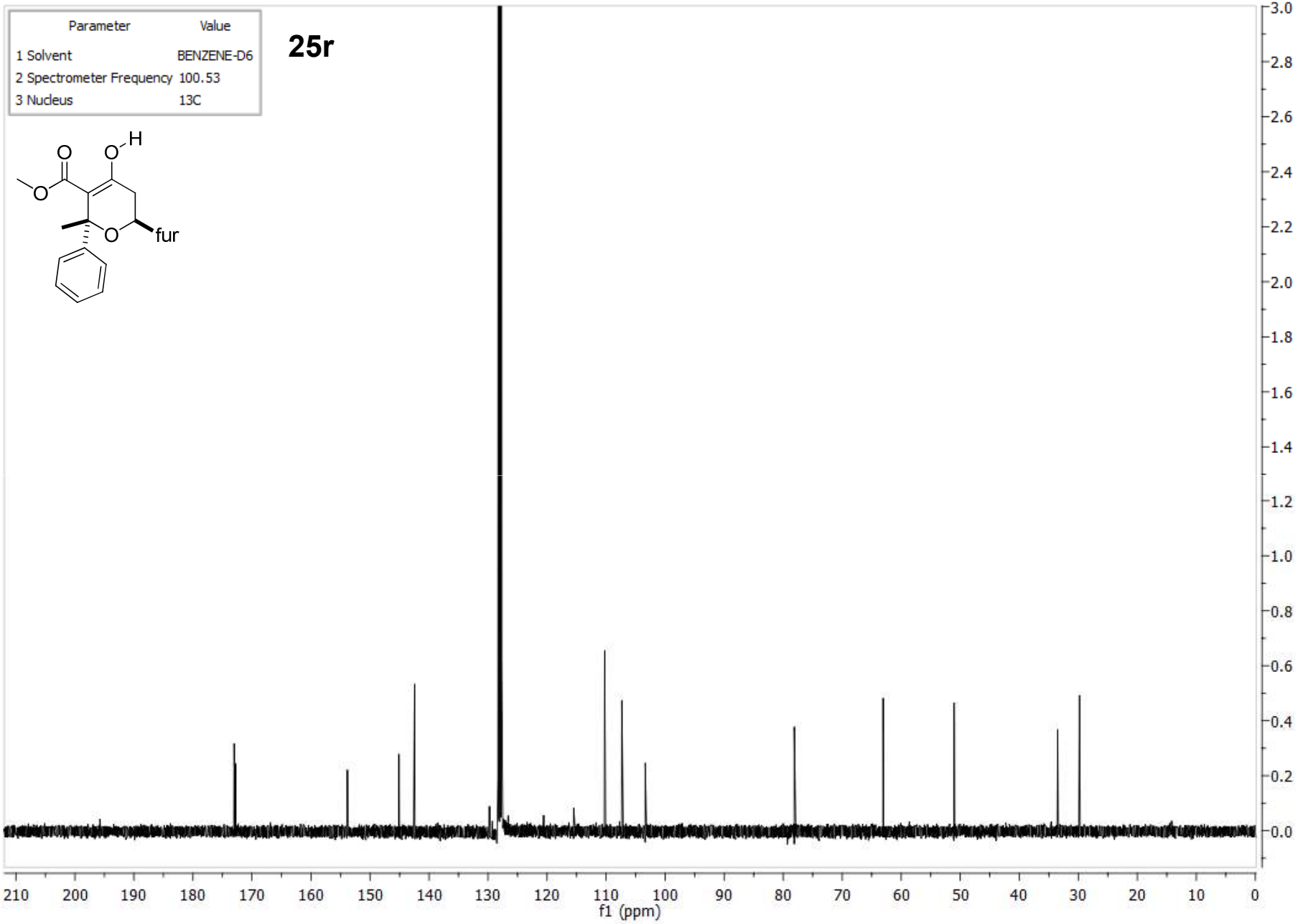

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
25p
O
O
O
H
O
Pr
1.96
5.17
1.13
4.01
1.00
3.82
2.58
2.28
2.86
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

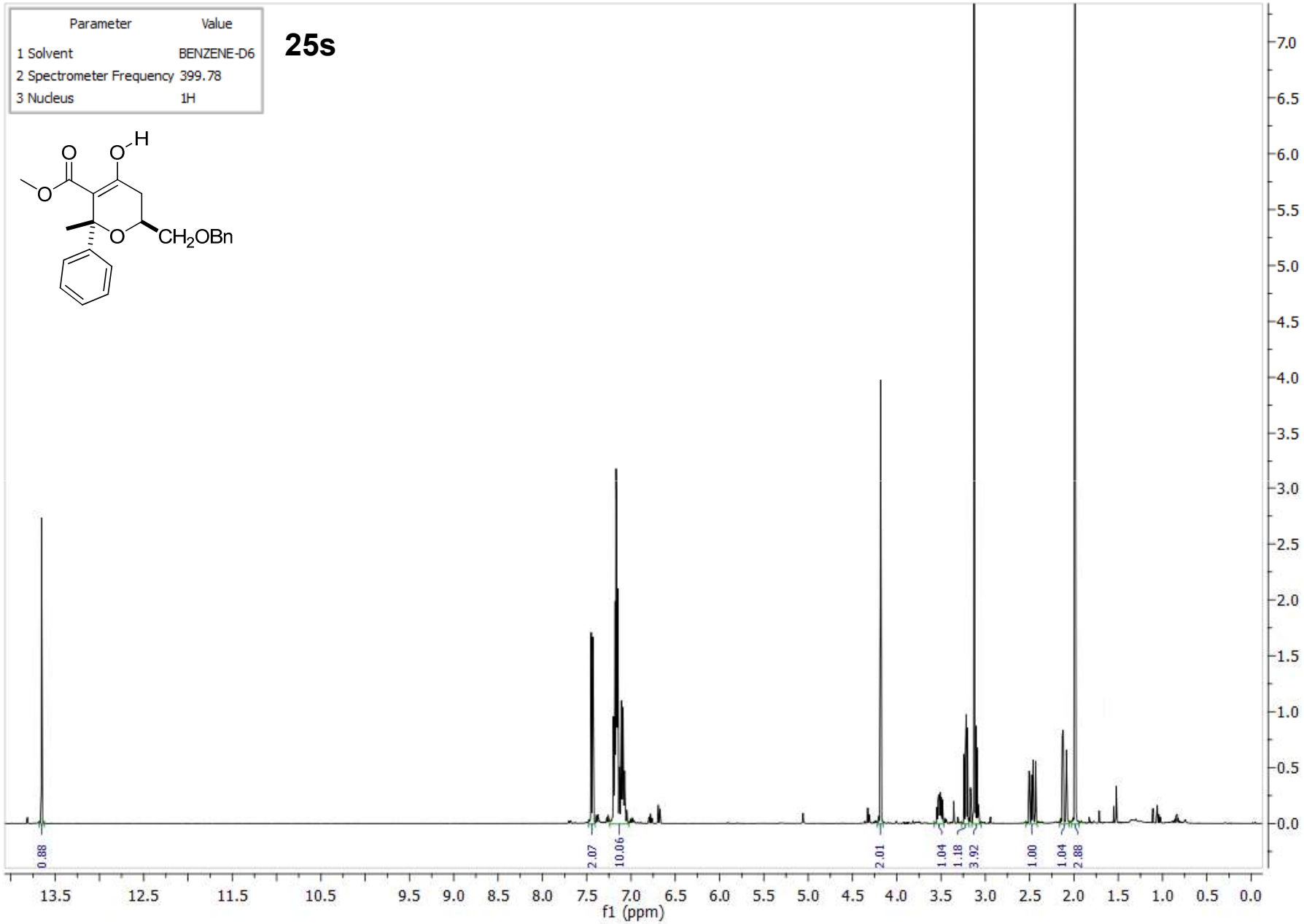

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
25p
O
O
O
H
O
Pr
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
-0.2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

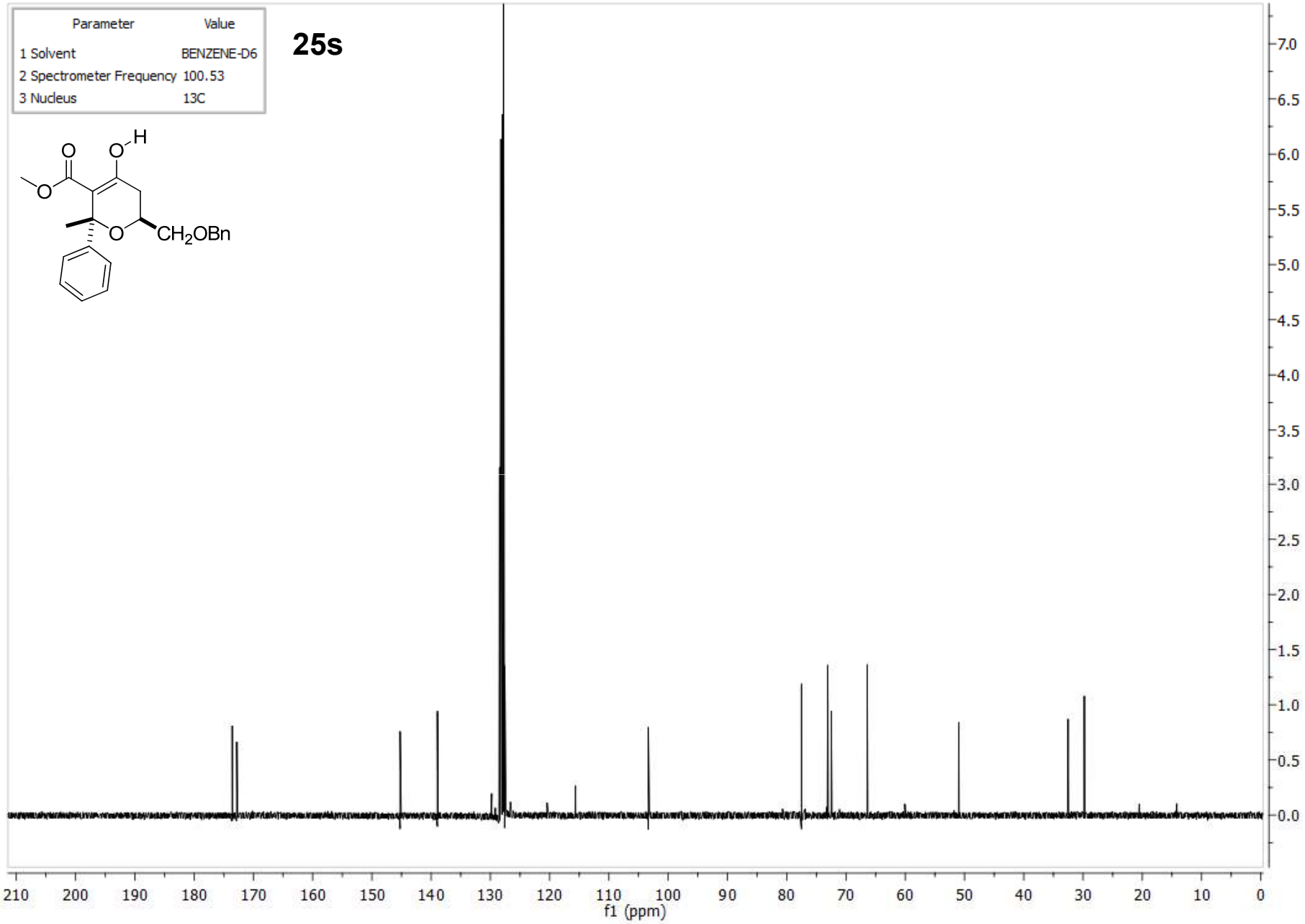

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
25q
O
O
H
O
O
iPr
0.90
2.12
3.35
1.13
2.91
1.00
1.02
1.05
2.88
1.03
2.84
2.88
13.5 12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

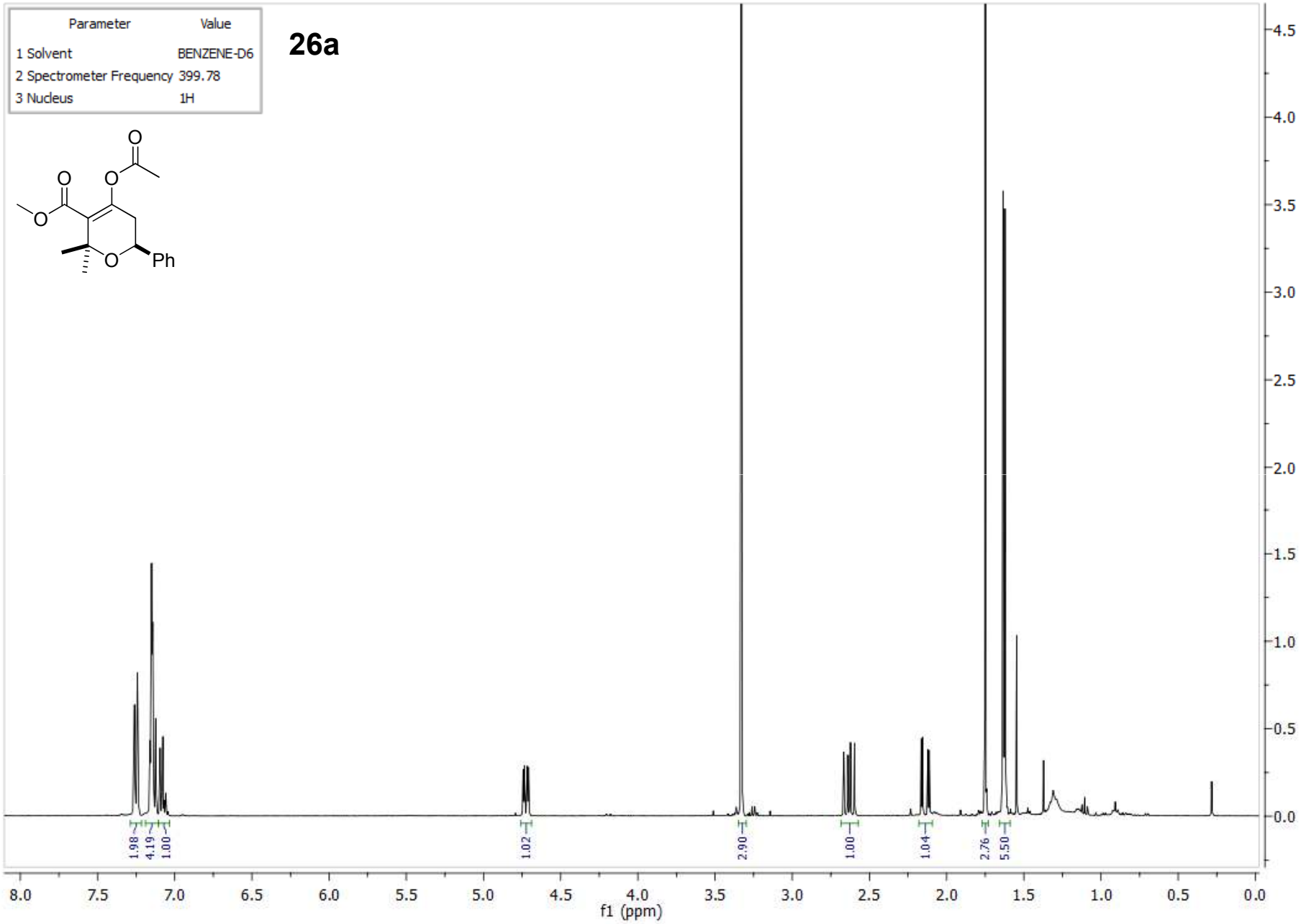

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
25q
O
O
O
H
O
iPr
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

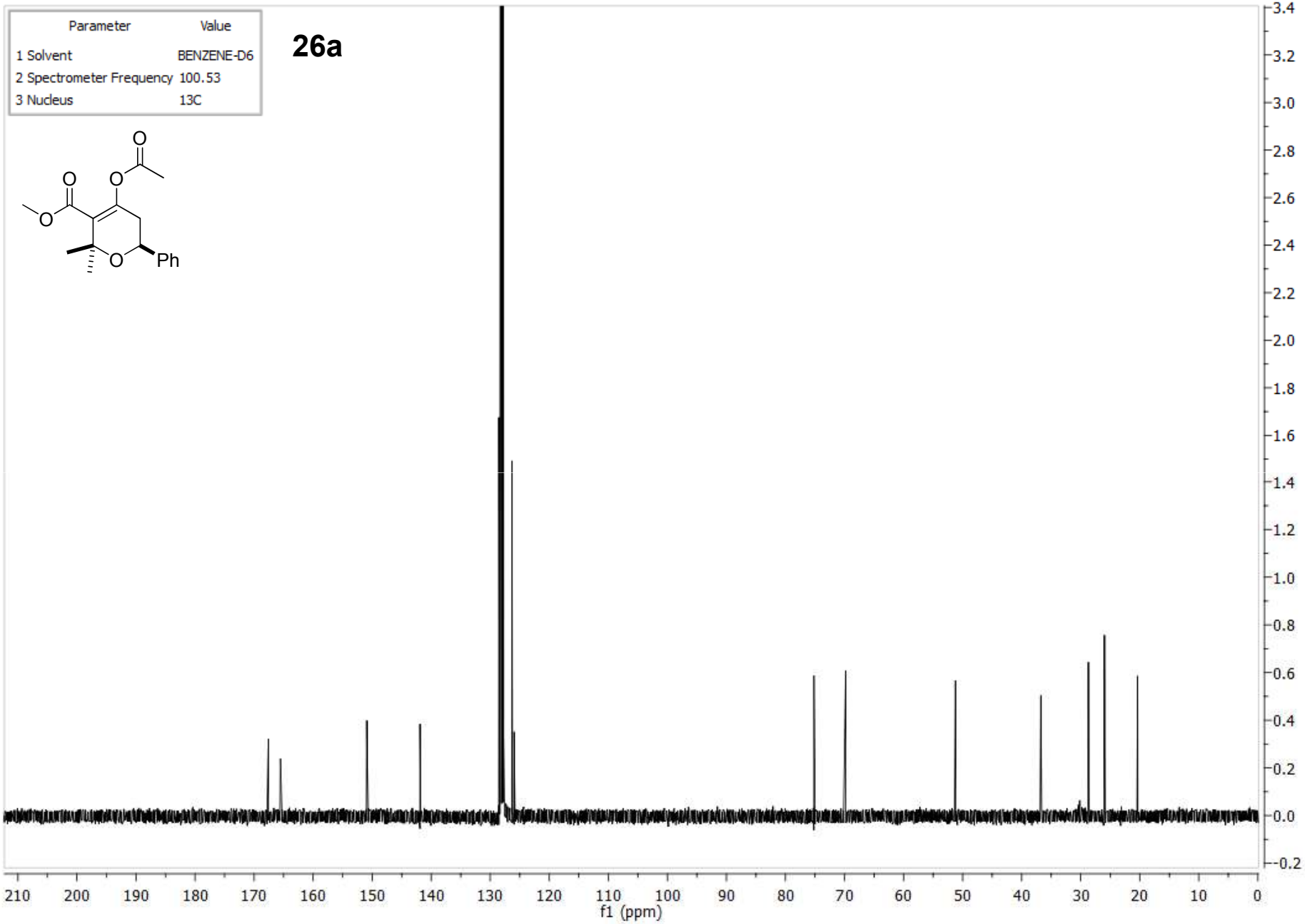

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
25r
O
O
O
H
O
fur
1.89
5.04
1.12
0.94
0.96
0.97
1.04
2.88
1.00
1.04
2.79
f1 (ppm)

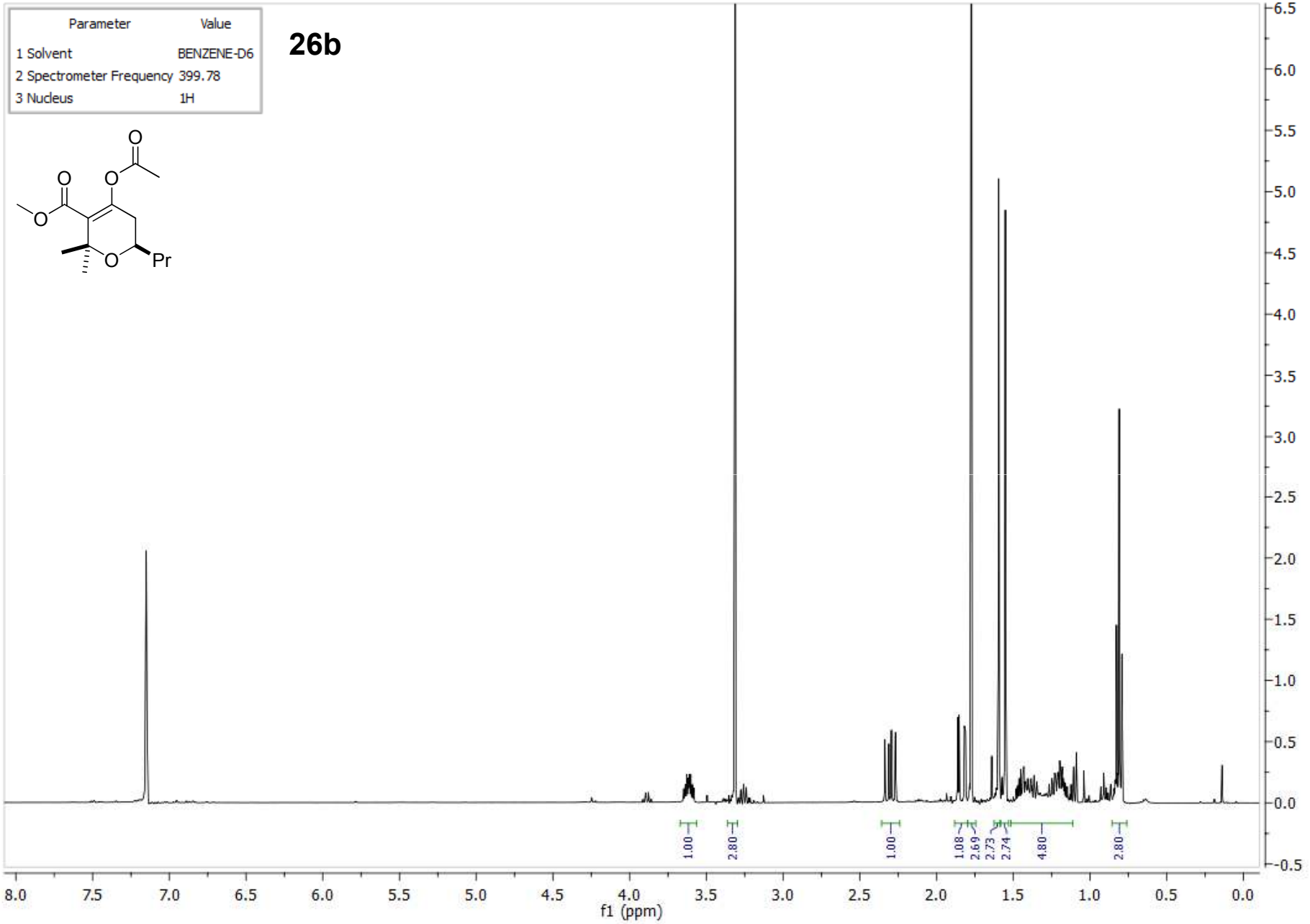

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
25r
O
O
O
H
O
fur
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

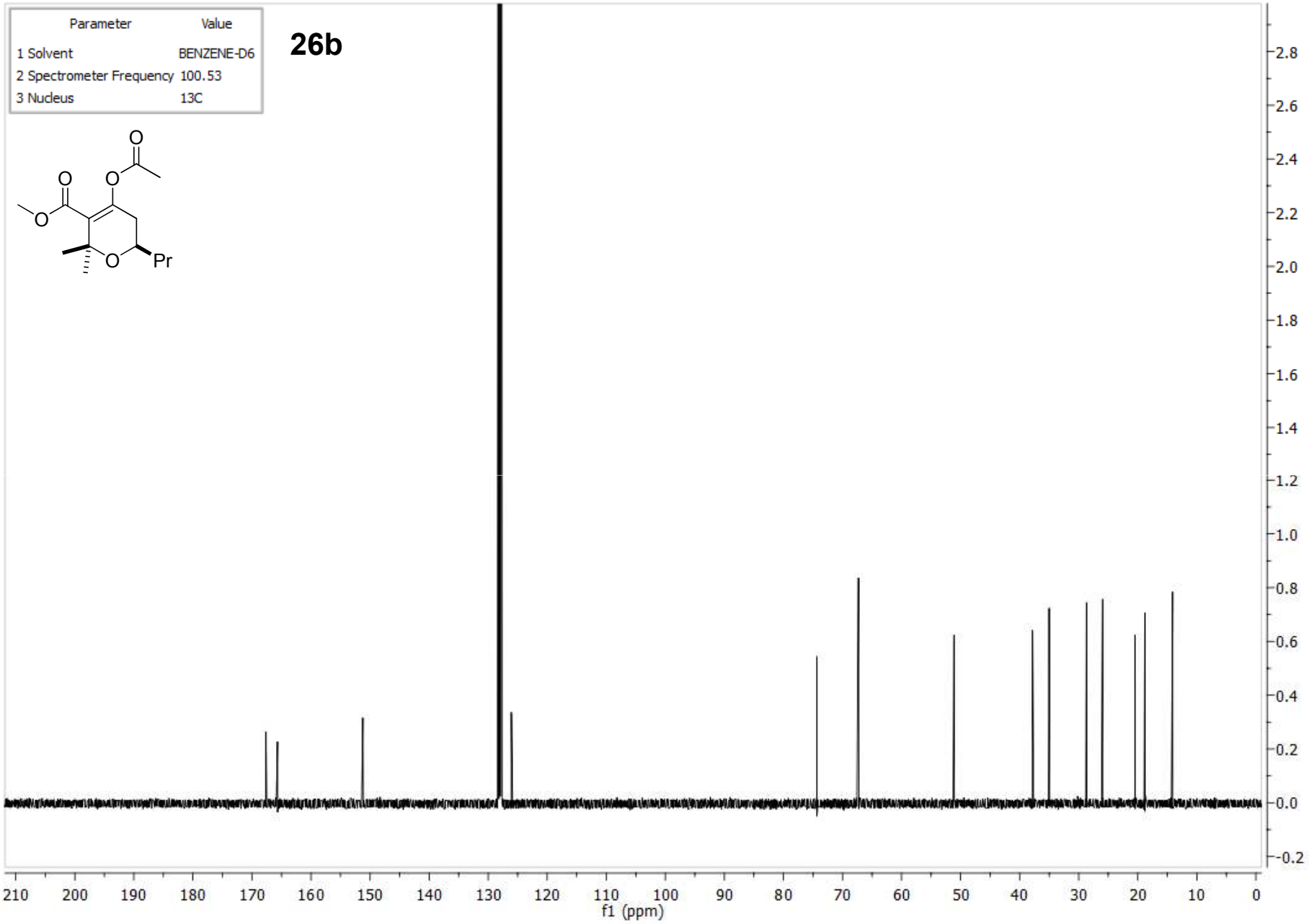

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
25s
O
O
H
O
O
CH2OBn
0.88
2.07
10.06
2.01
1.04
1.18
3.92
1.00
1.04
2.88
13.5 12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

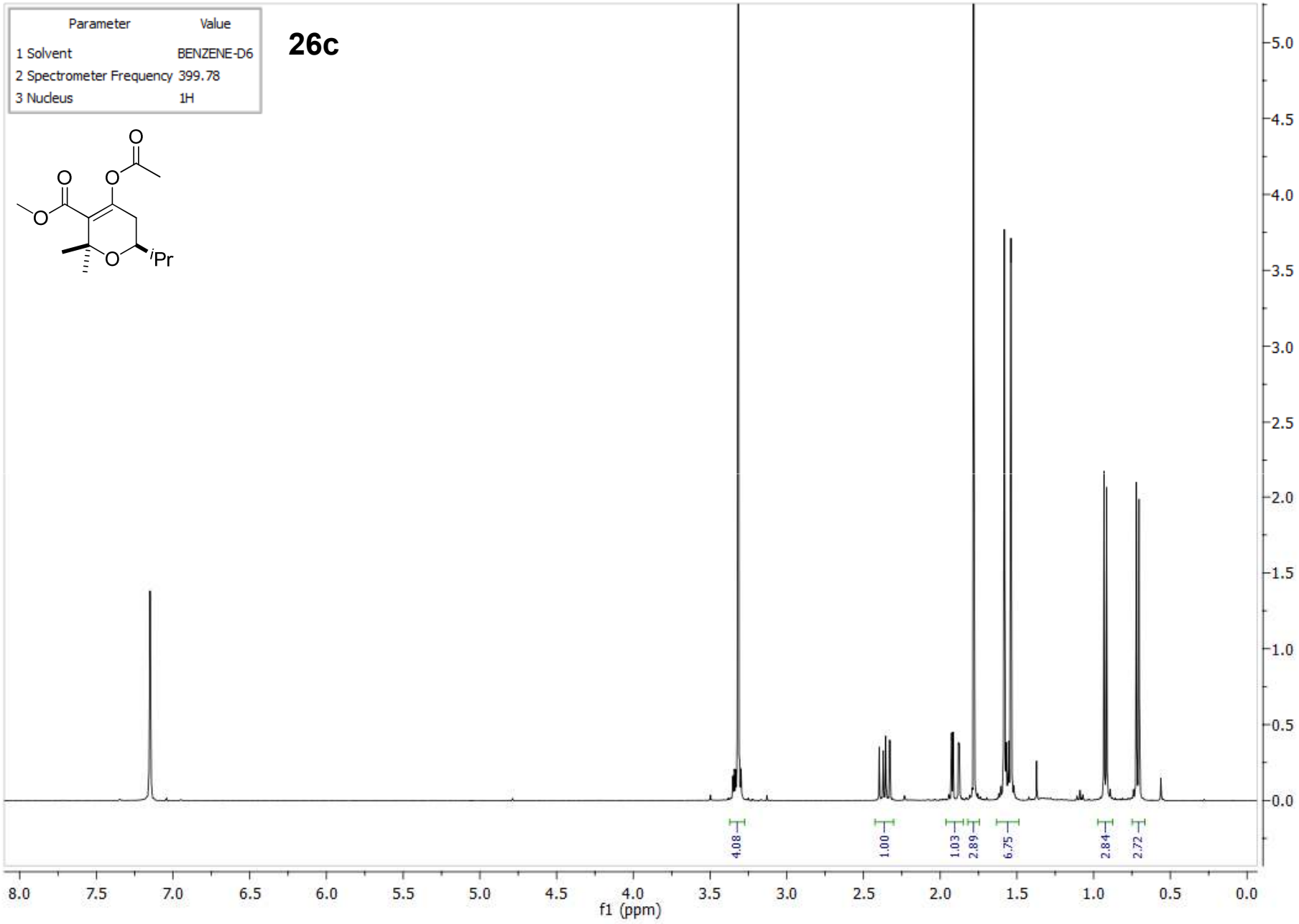

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
25s
O
O
O
H
O
CH2OBn
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

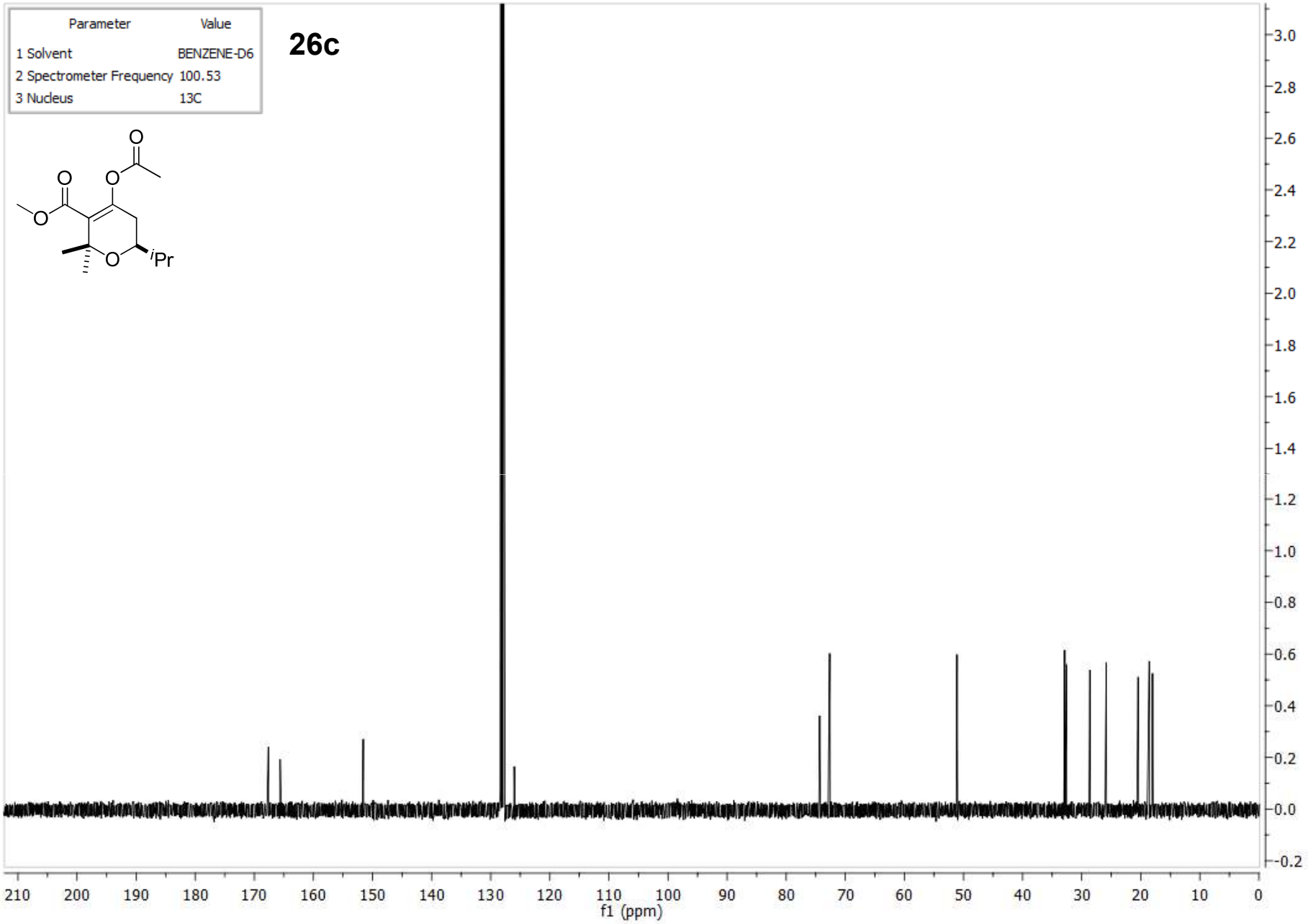

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
26a
O
O
O
O
O
Ph
1.98
4.19
1.00
1.02
2.90
1.00
1.04
2.76
5.50
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

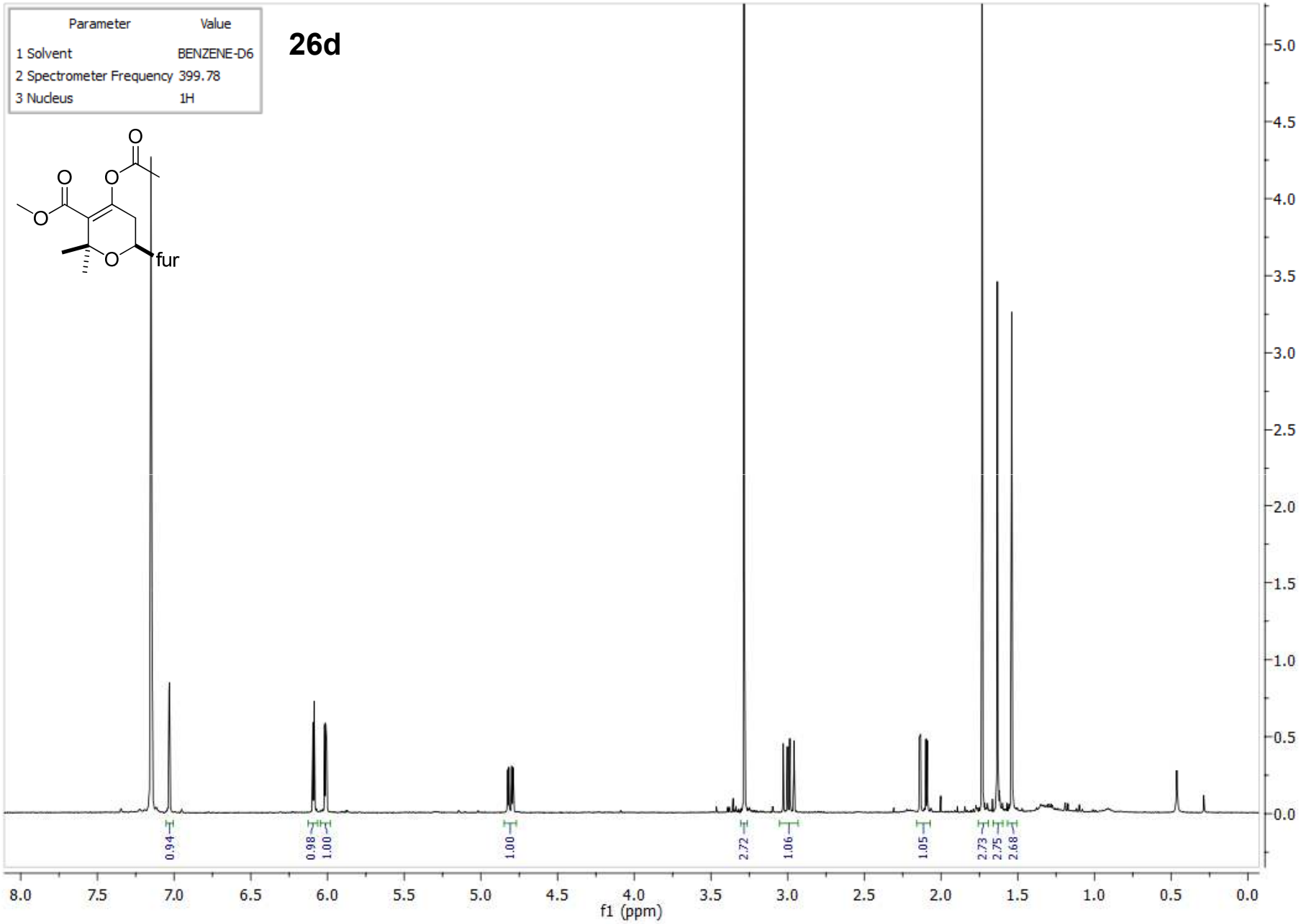

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
26a
O
O
O
O
O
Ph
3.4
3.2
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
-0.2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

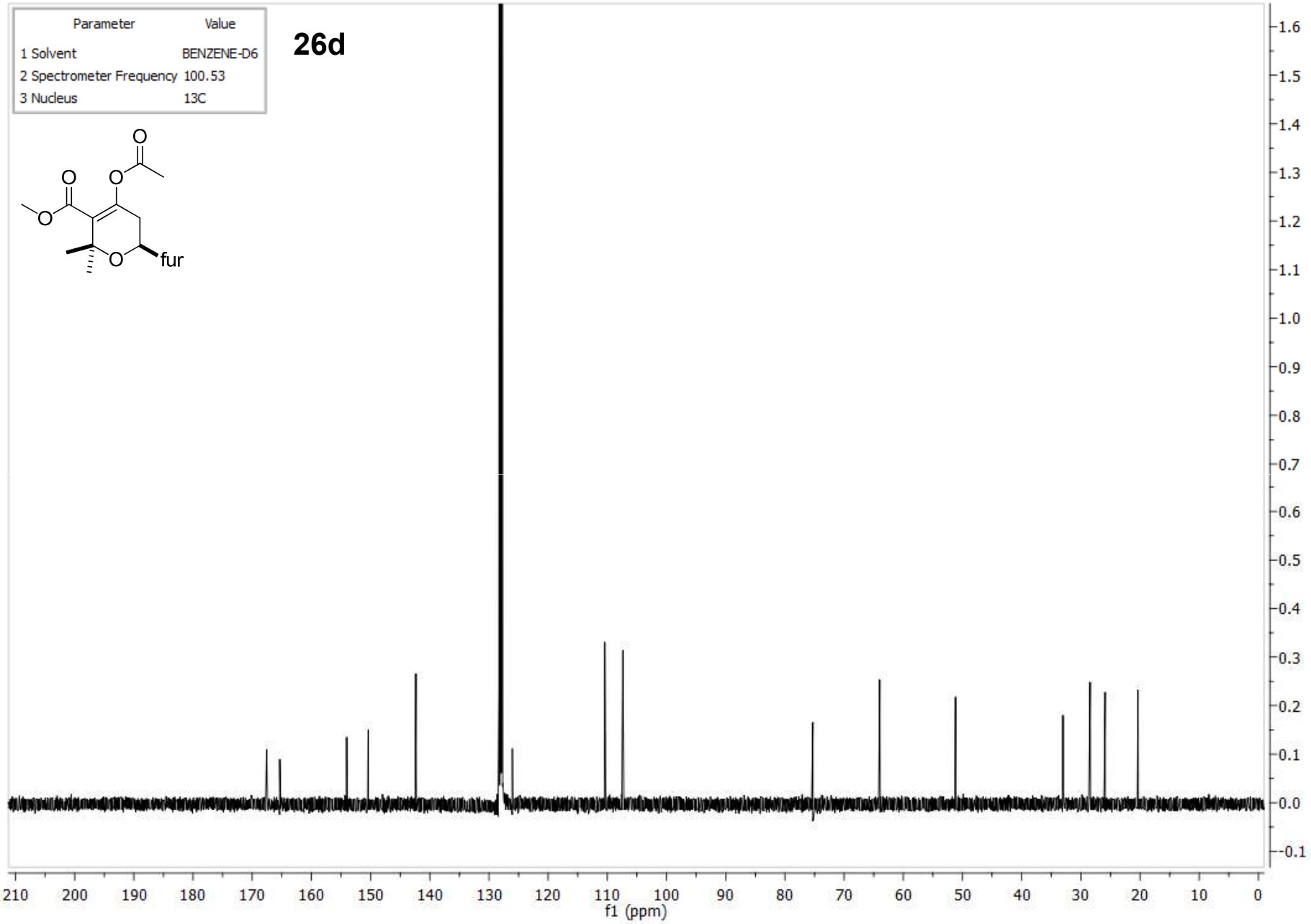

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
26b
O
O
O
O
O
Pr
1.00
2.80
1.00
1.08
2.69
2.73
2.74
4.80
2.80
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5

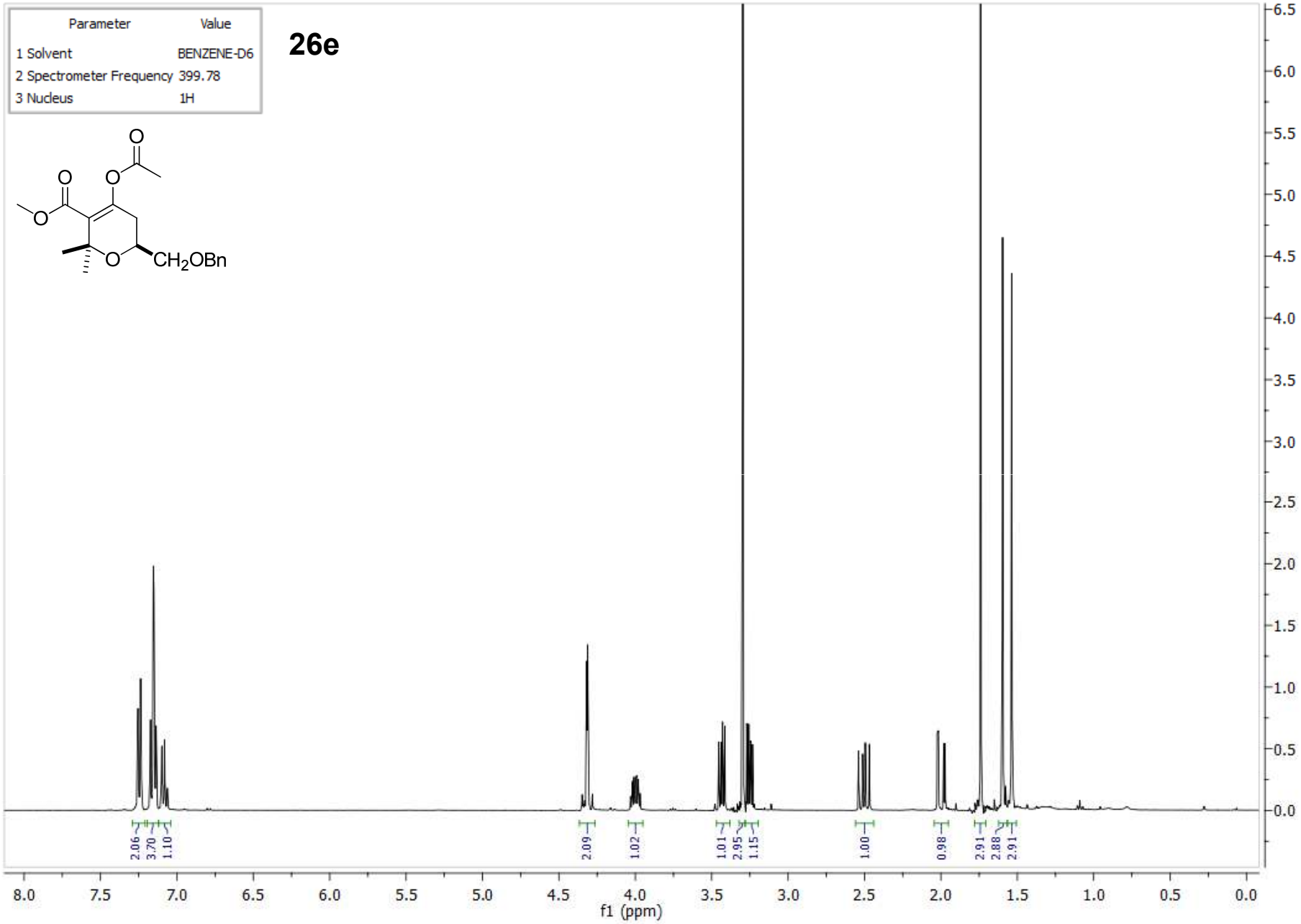

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
26b
O
O
O
O
Pr
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
-0.2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

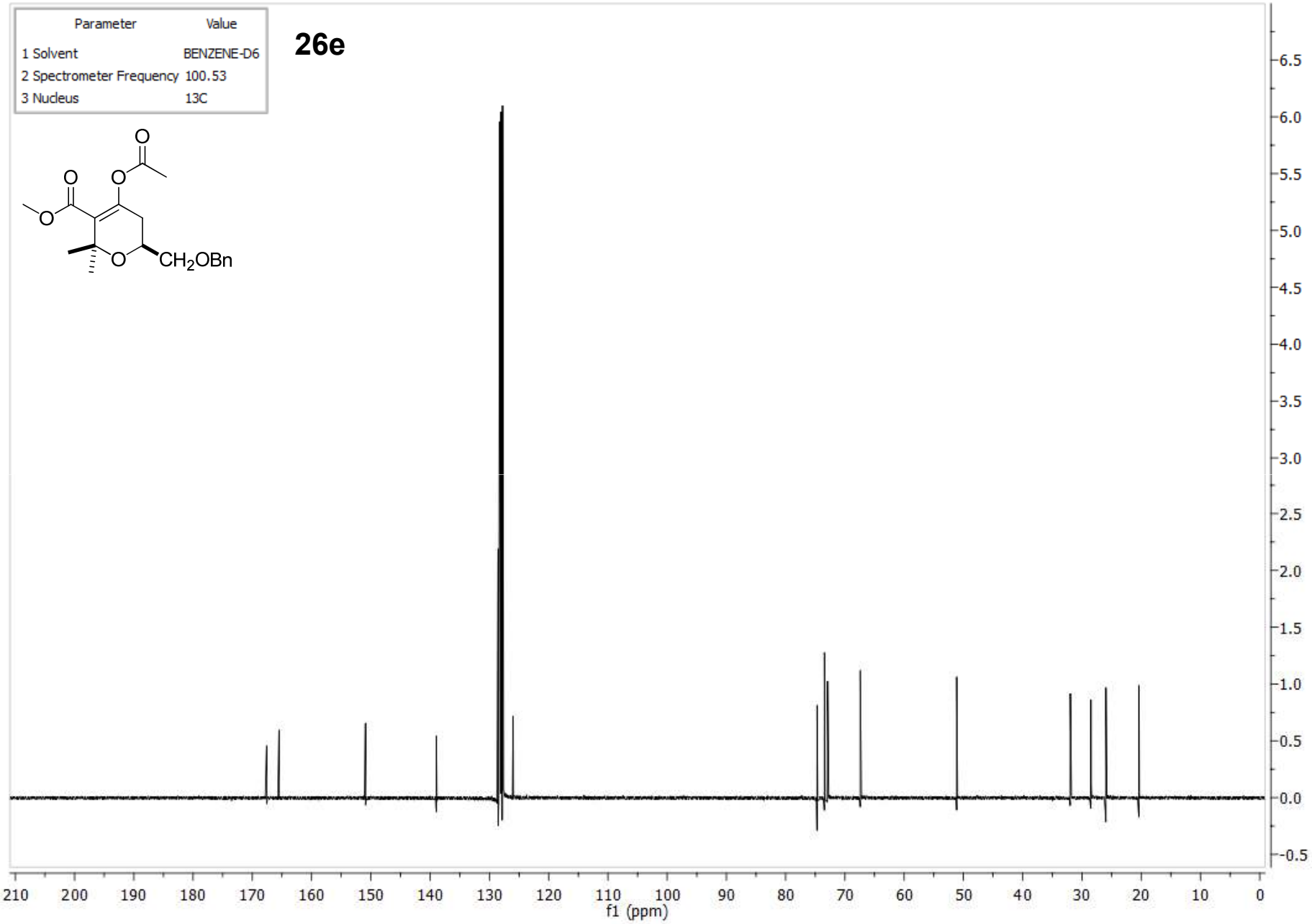

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
26c
O
O
O
O
O
iPr
4.08
1.00
1.03
2.89
6.75
2.84
2.72
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

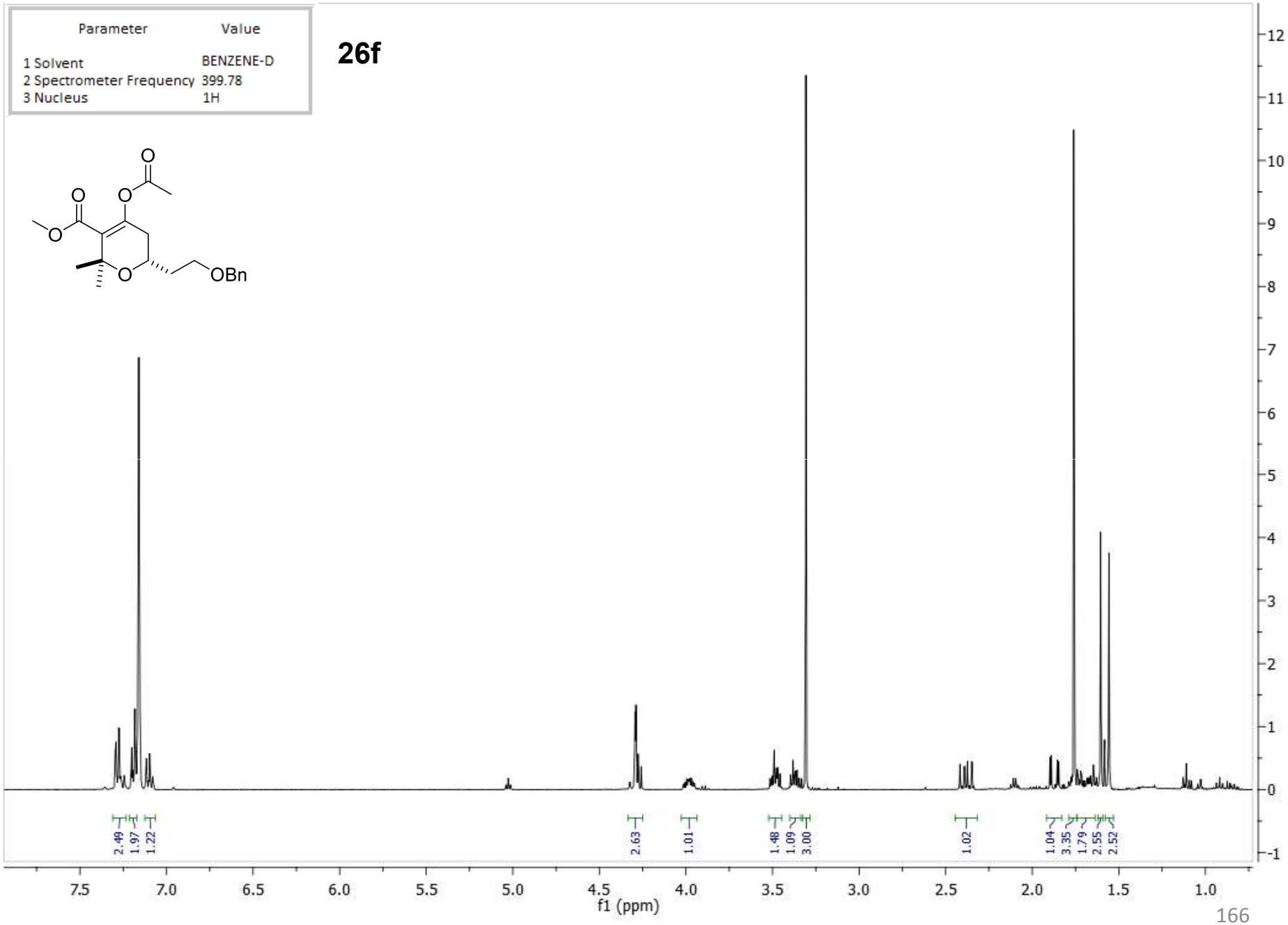

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
26c
O
O
O
O
iPr
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
-0.2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

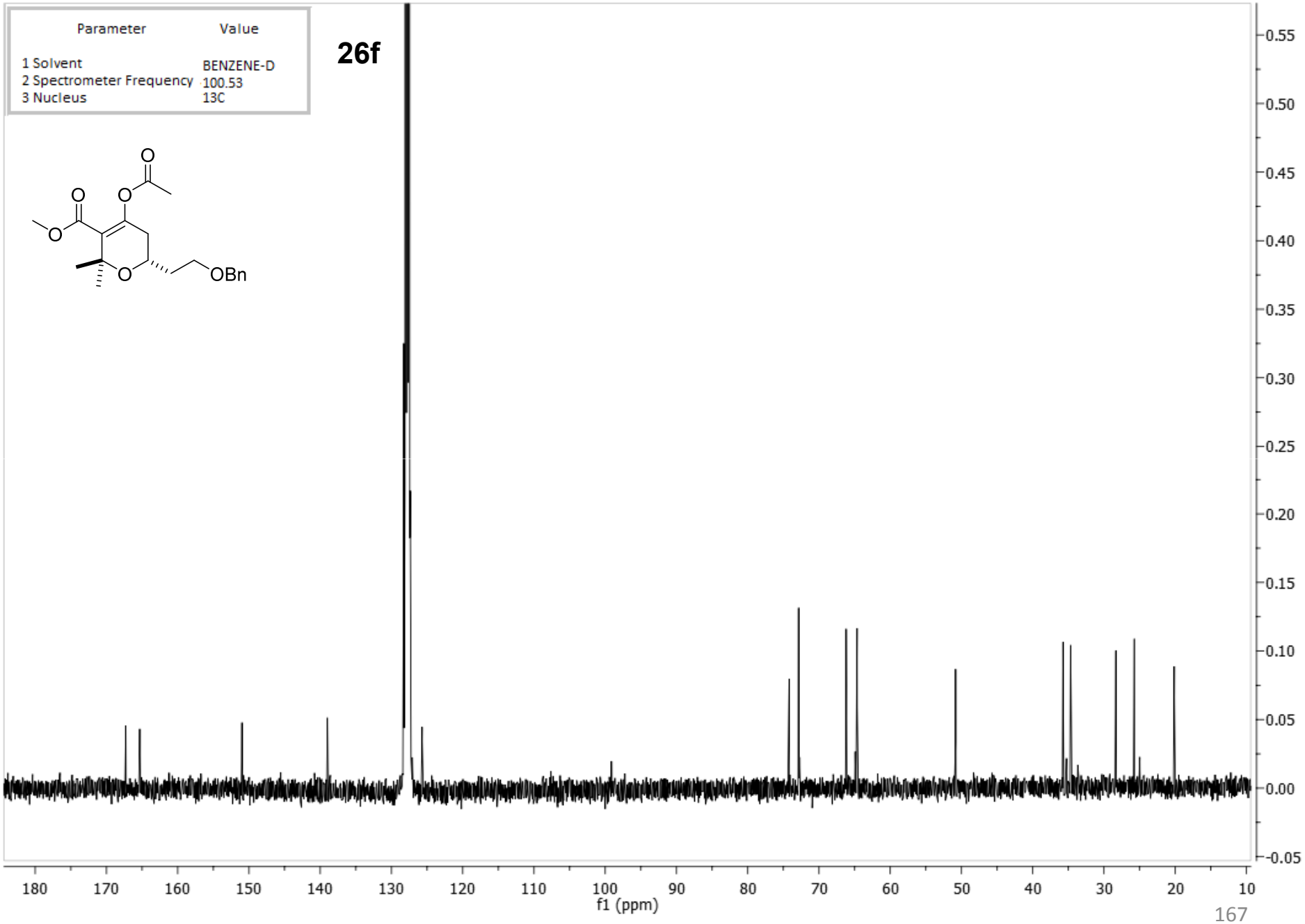

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
26d
fur
0.94
0.98
1.00
1.00
2.72
1.06
1.05
2.73
2.75
2.68
f1 (ppm)

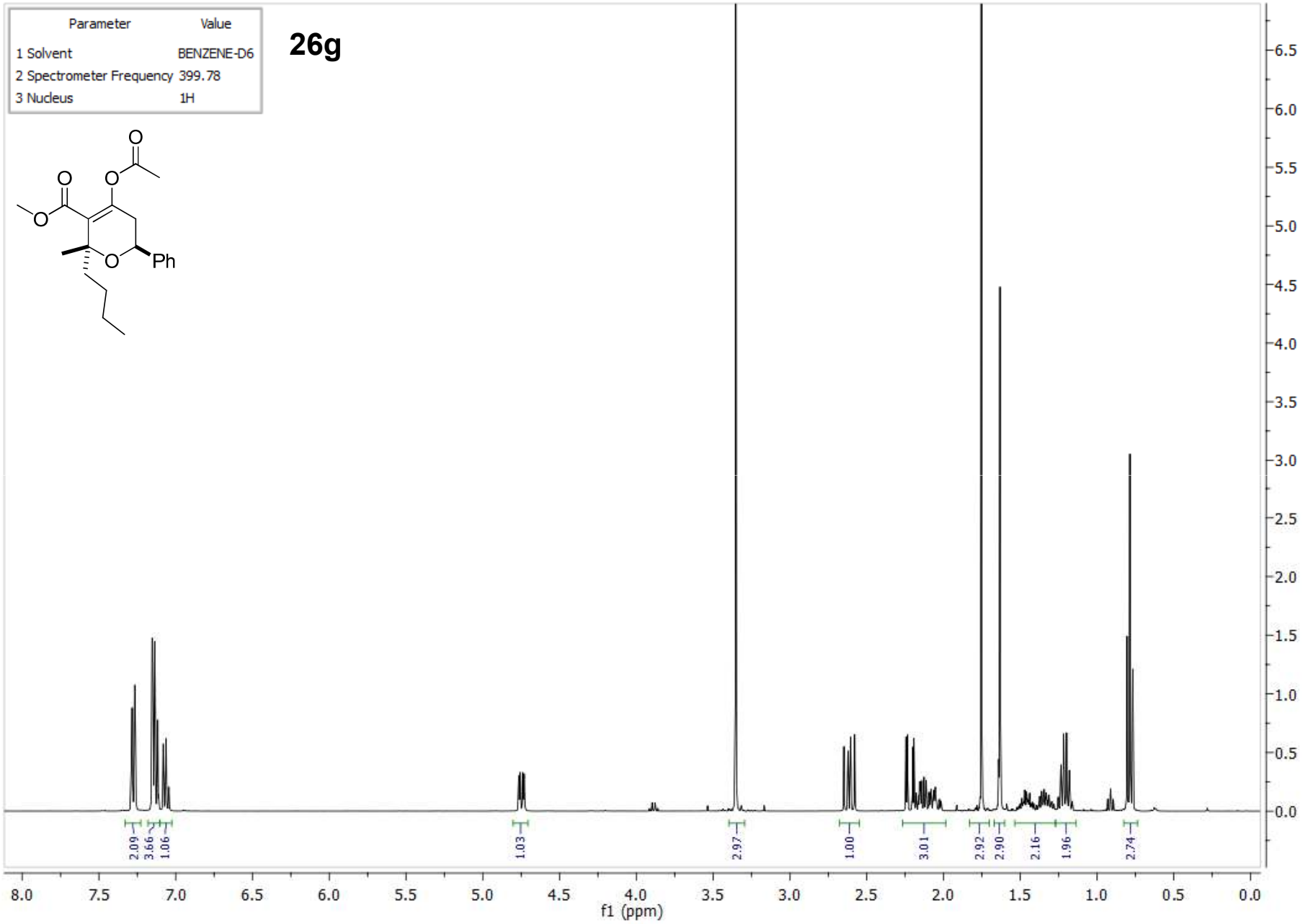

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
26d
O
O
O
O
fur
1.6
1.5
1.4
1.3
1.2
1.1
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
-0.1
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

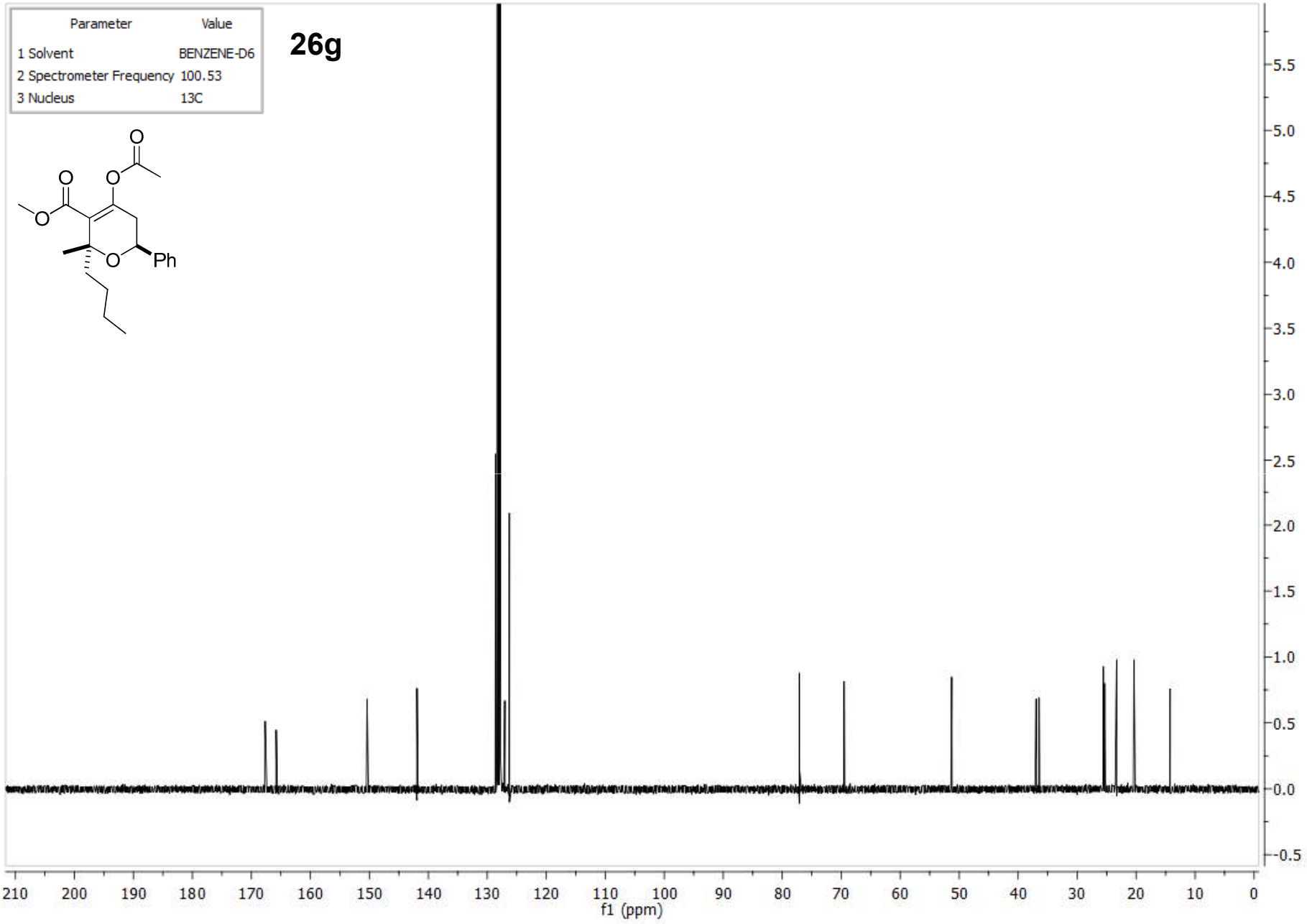

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
26e
O
O
O
O
O
CH2OBn
2.06
3.70
1.10
2.09
1.02
1.01
2.95
1.15
1.00
0.98
2.91
2.88
2.91
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

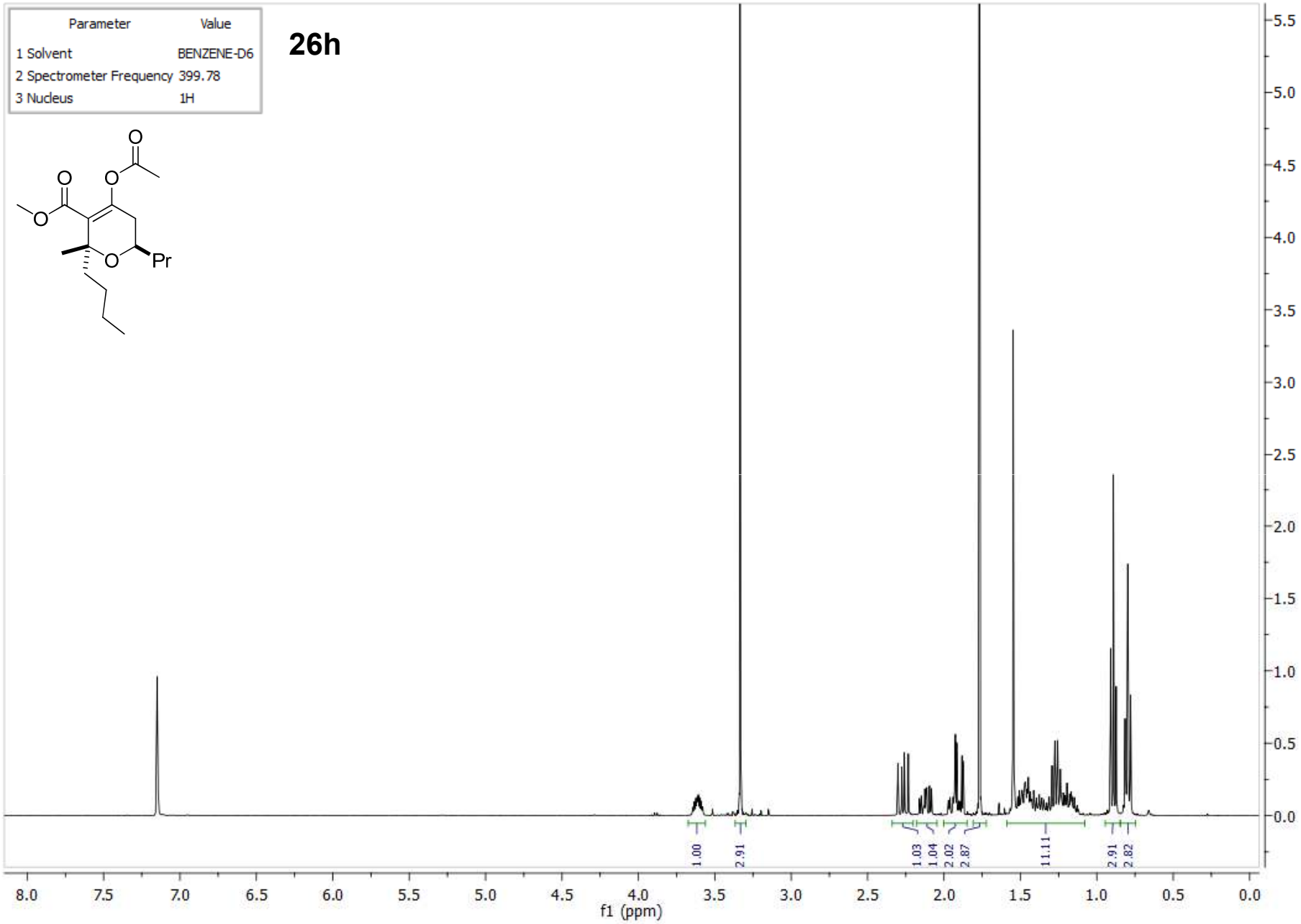

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
26e
O
O
O
O
CH2OBn
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

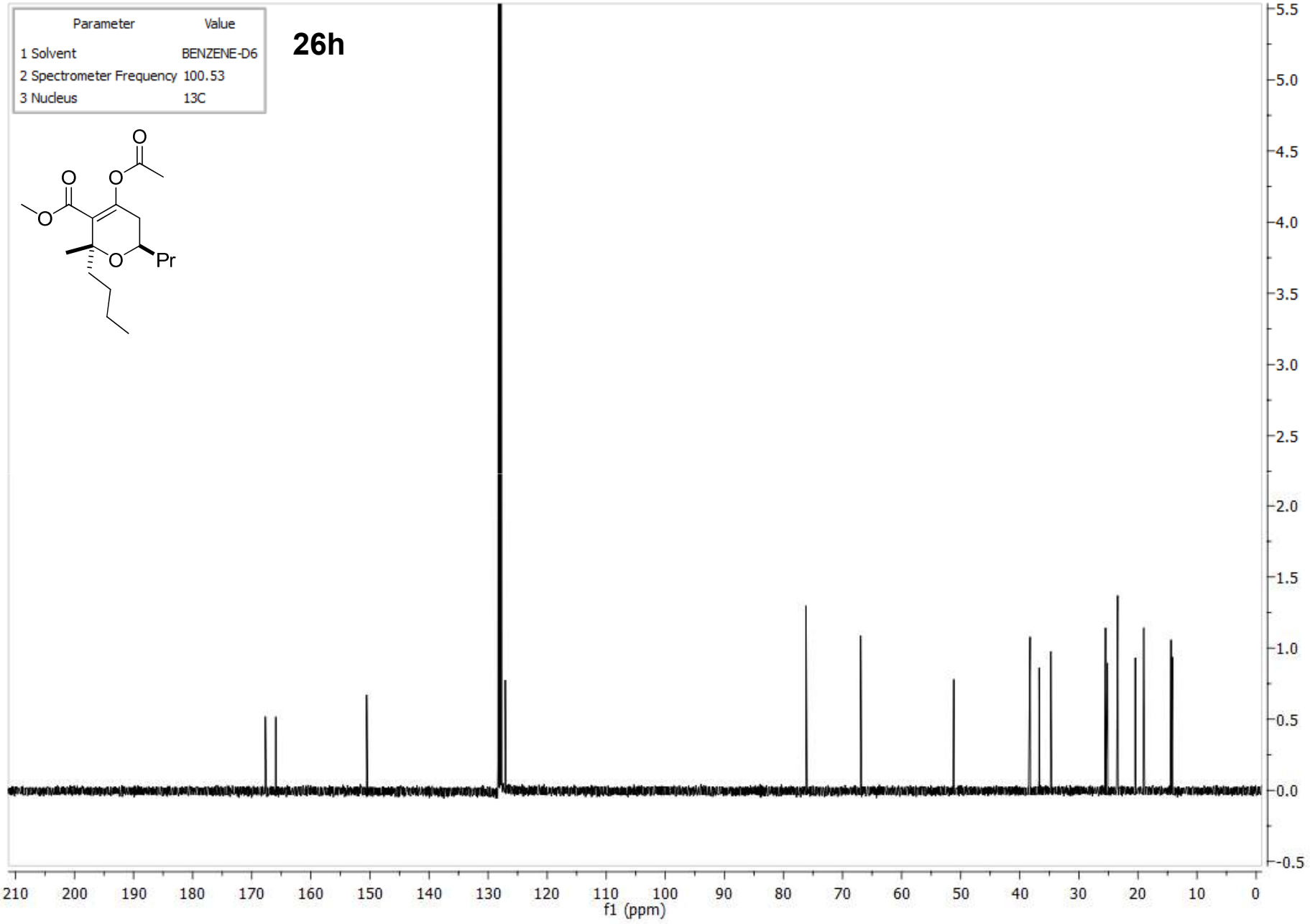

Parameter Value
1 Solvent BENZENE-D
2 Spectrometer Frequency 399.78
3 Nucleus 1H
26f
OBn
2.49
1.97
1.22
2.63
1.01
1.48
1.09
3.00
1.02
1.04
3.35
1.79
2.55
2.52
f1 (ppm)

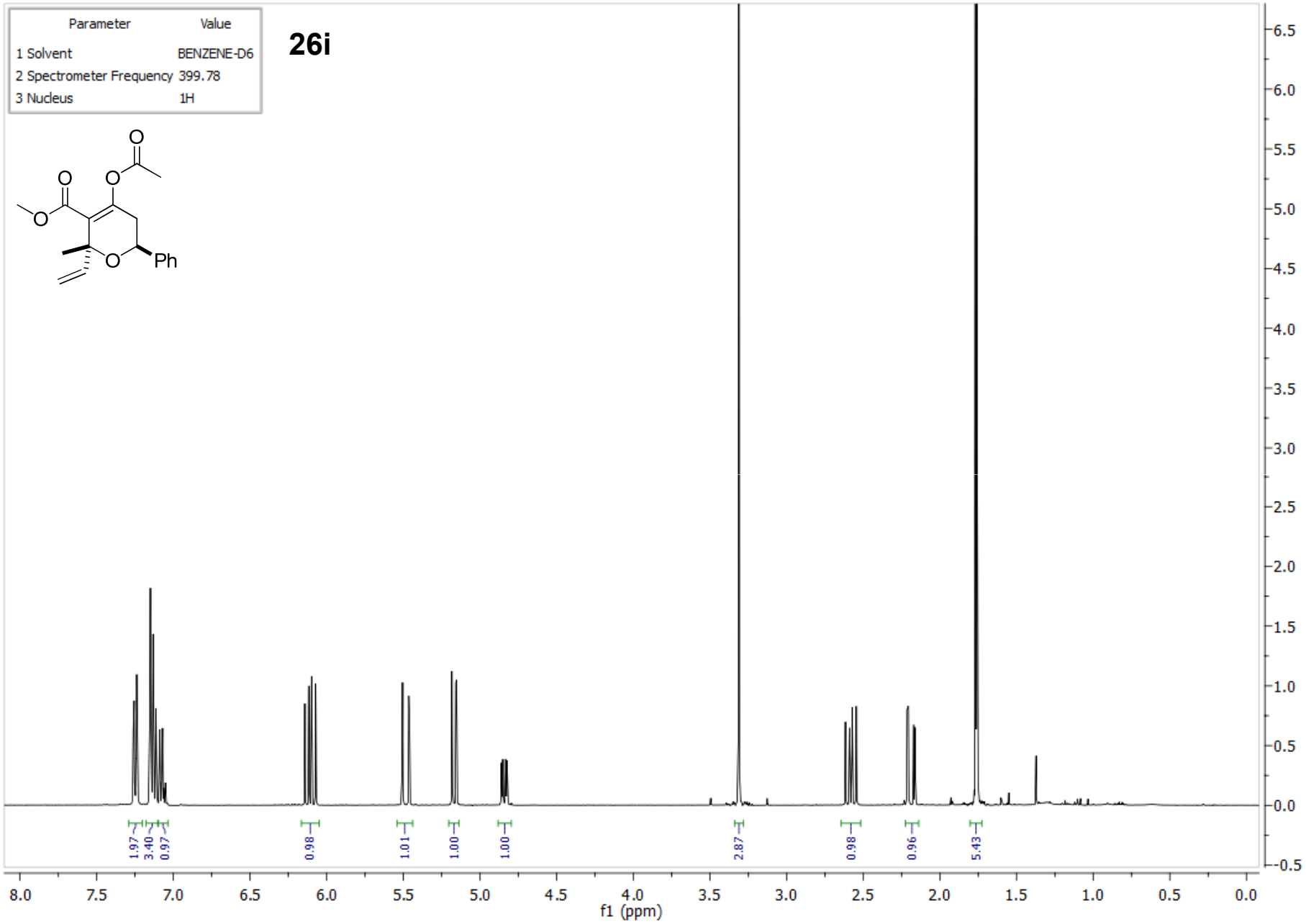

Parameter
Value
1 Solvent
BENZENE-D
2 Spectrometer Frequency
100.53
3 Nucleus
13C
26f
OBn
f1 (ppm)

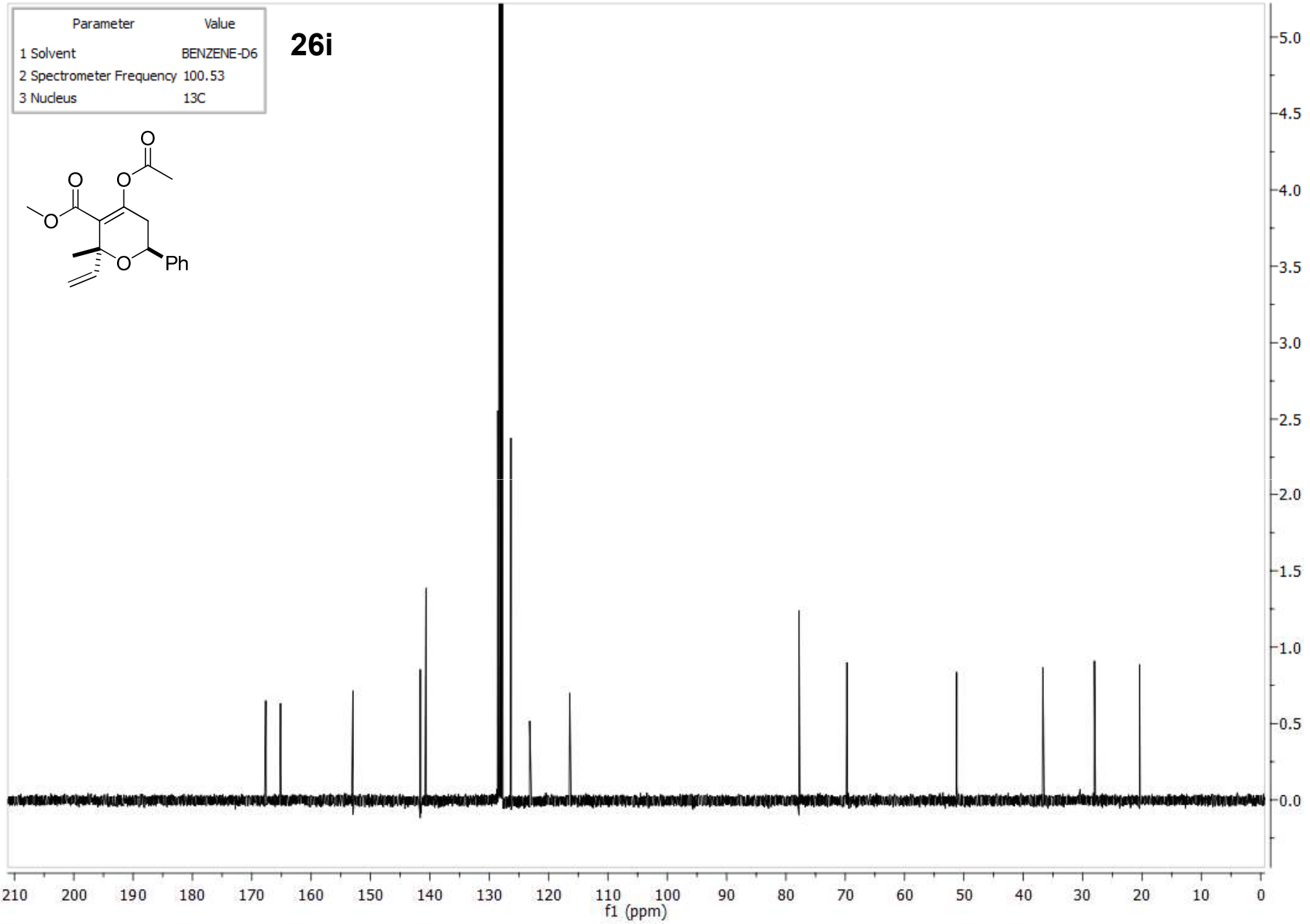

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
26g
O
O
O
O
O
Ph
2.09
3.66
1.06
1.03
2.97
1.00
3.01
2.92
2.90
2.16
1.96
2.74
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

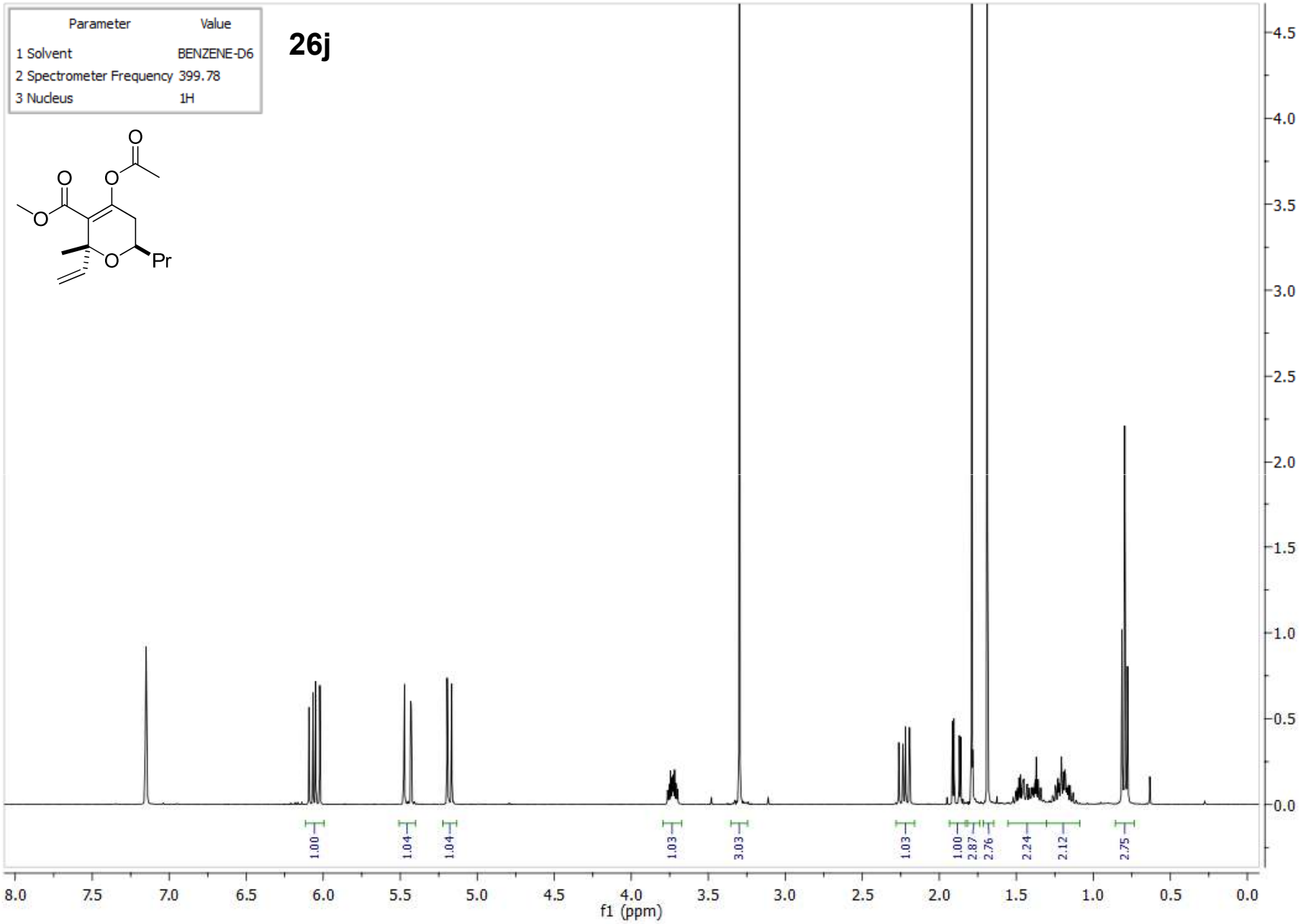

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
26g
O
O
O
O
O
Ph
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

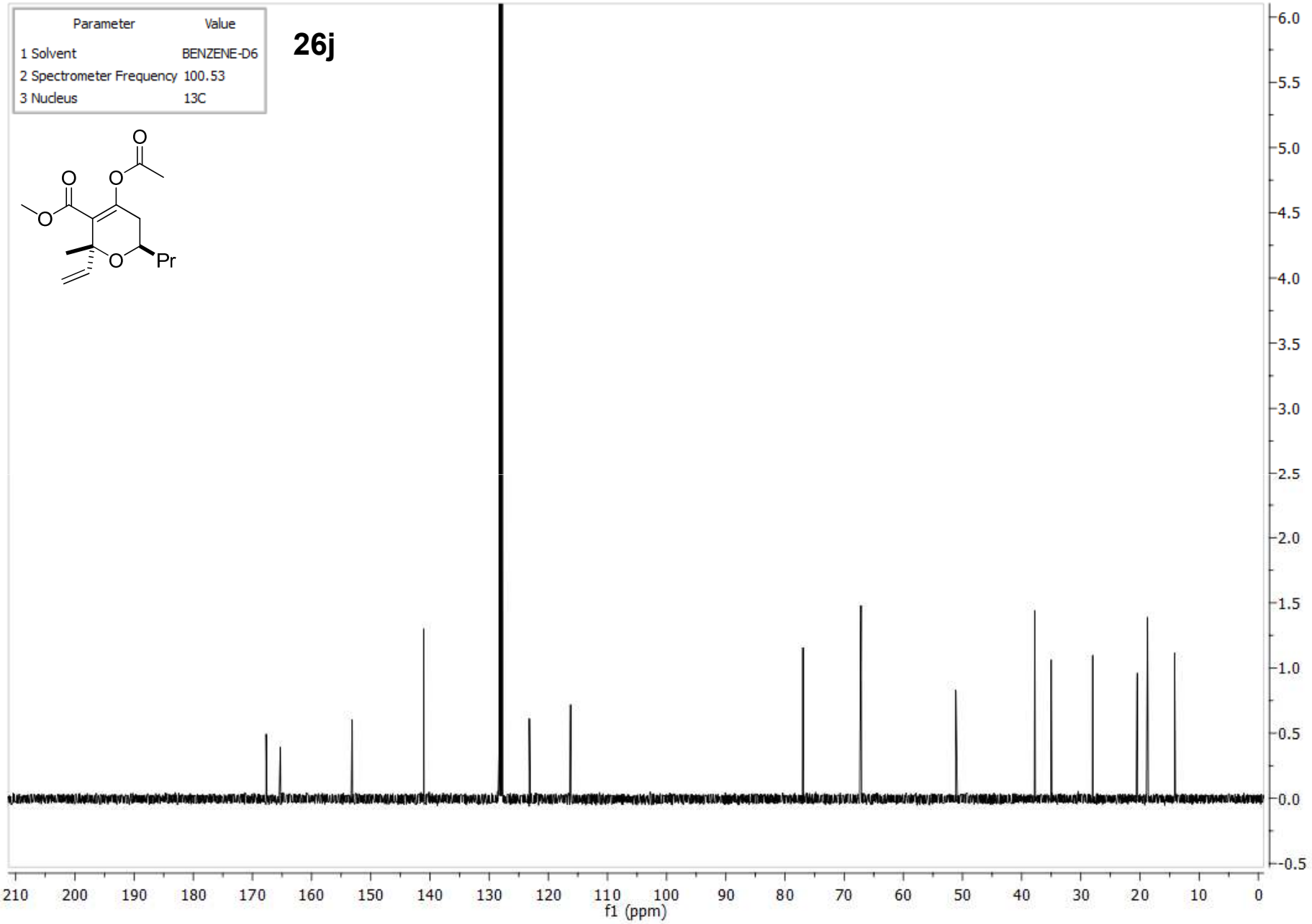

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
26h
O
O
O
O
Pr
1.00
2.91
1.03
1.04
2.02
2.87
11.11
2.91
2.82
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

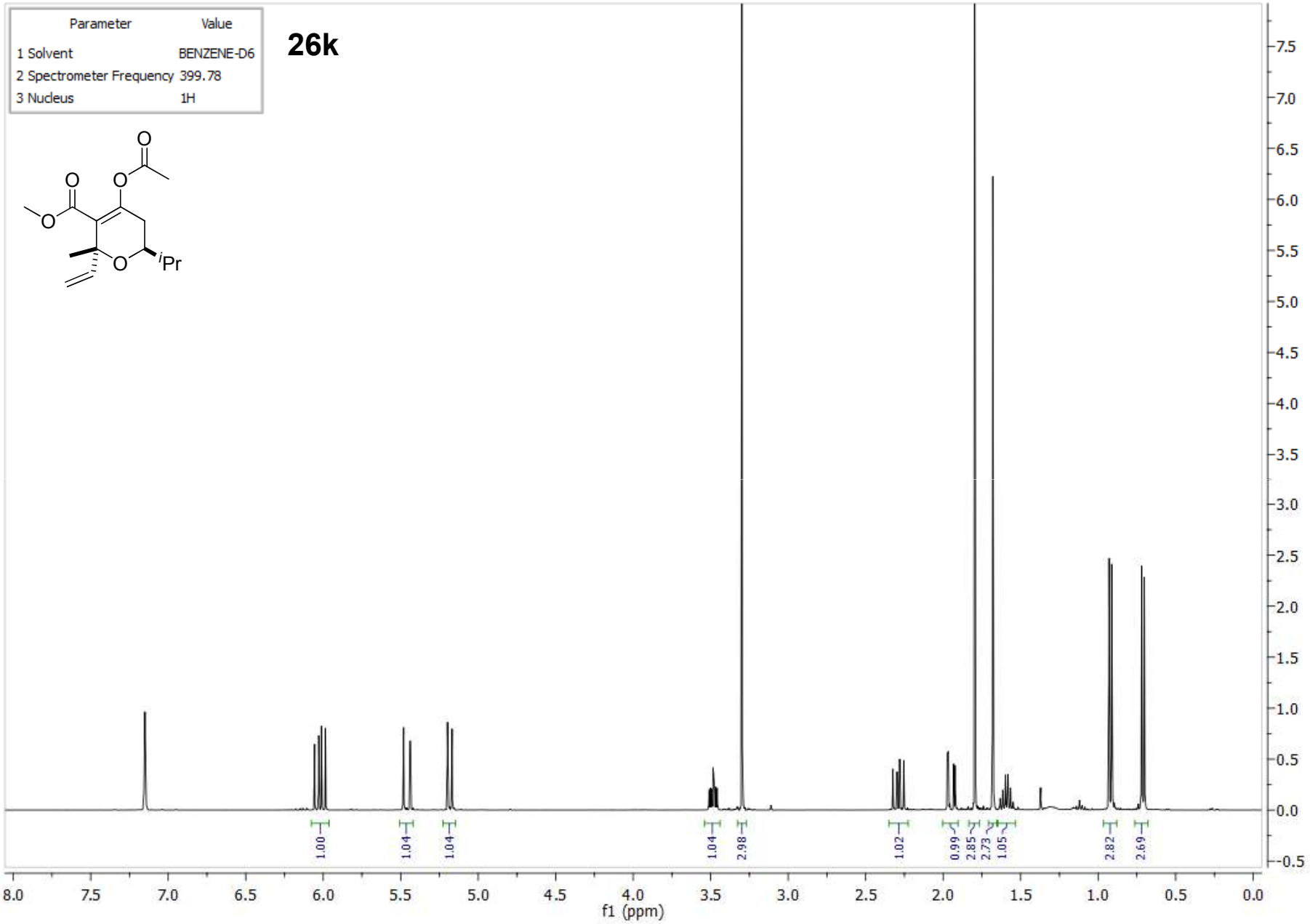

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
26h
O
O
O
O
Pr
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

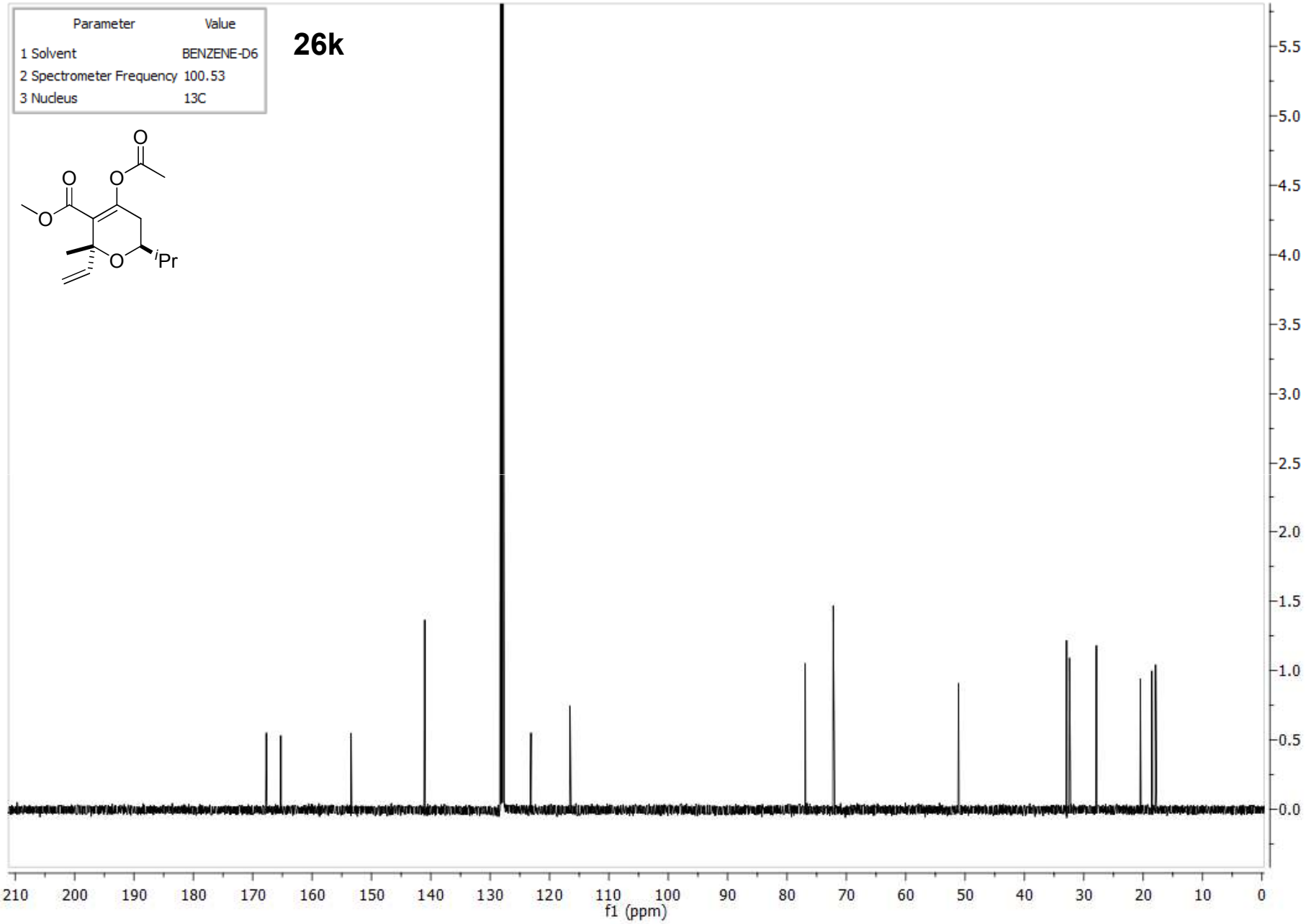

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
26i
O
O
O
O
Ph
1.97
3.40
0.97
0.98
1.01
1.00
1.00
2.87
0.98
0.96
5.43
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5

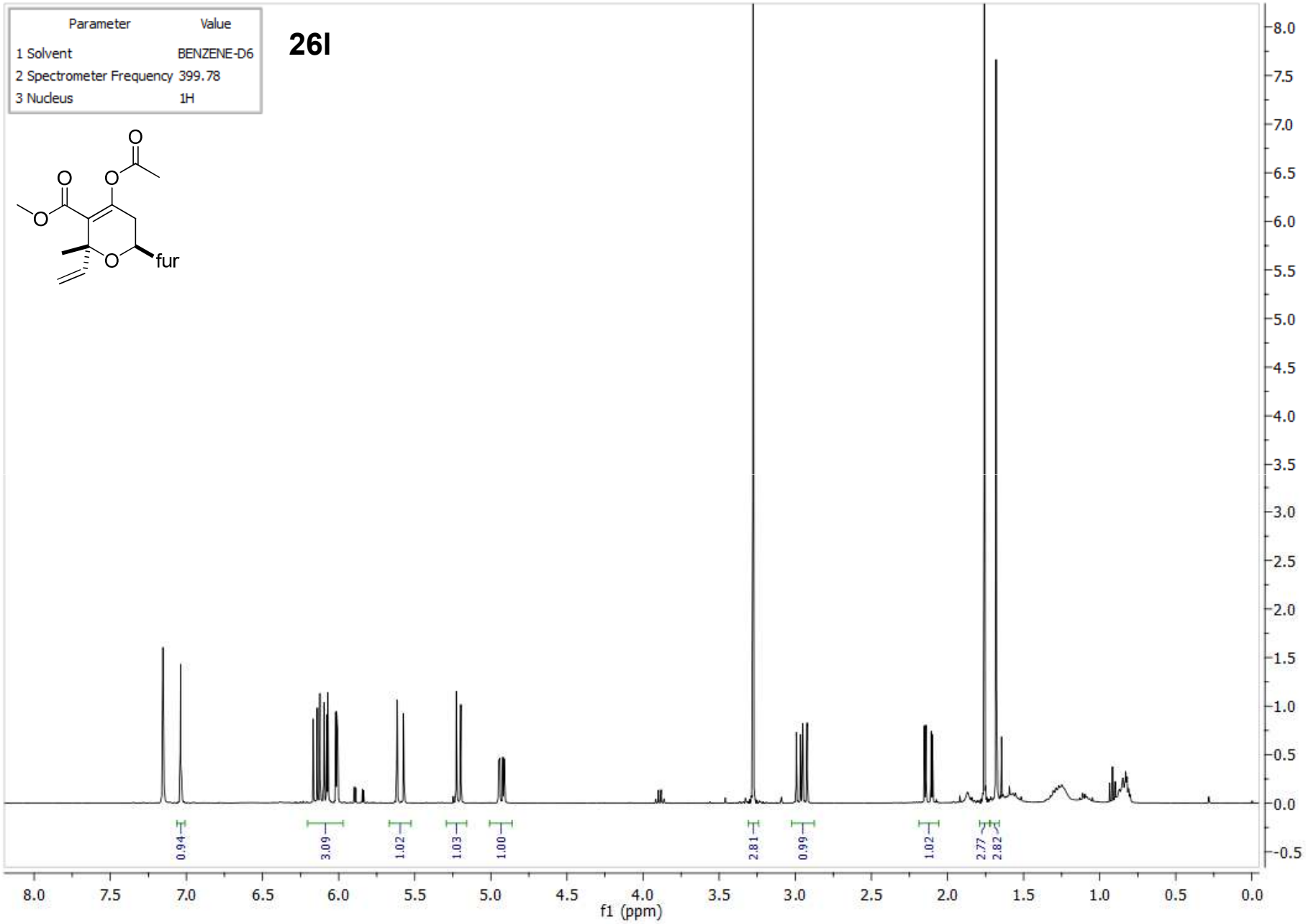

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
26i
O
O
O
O
Ph
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

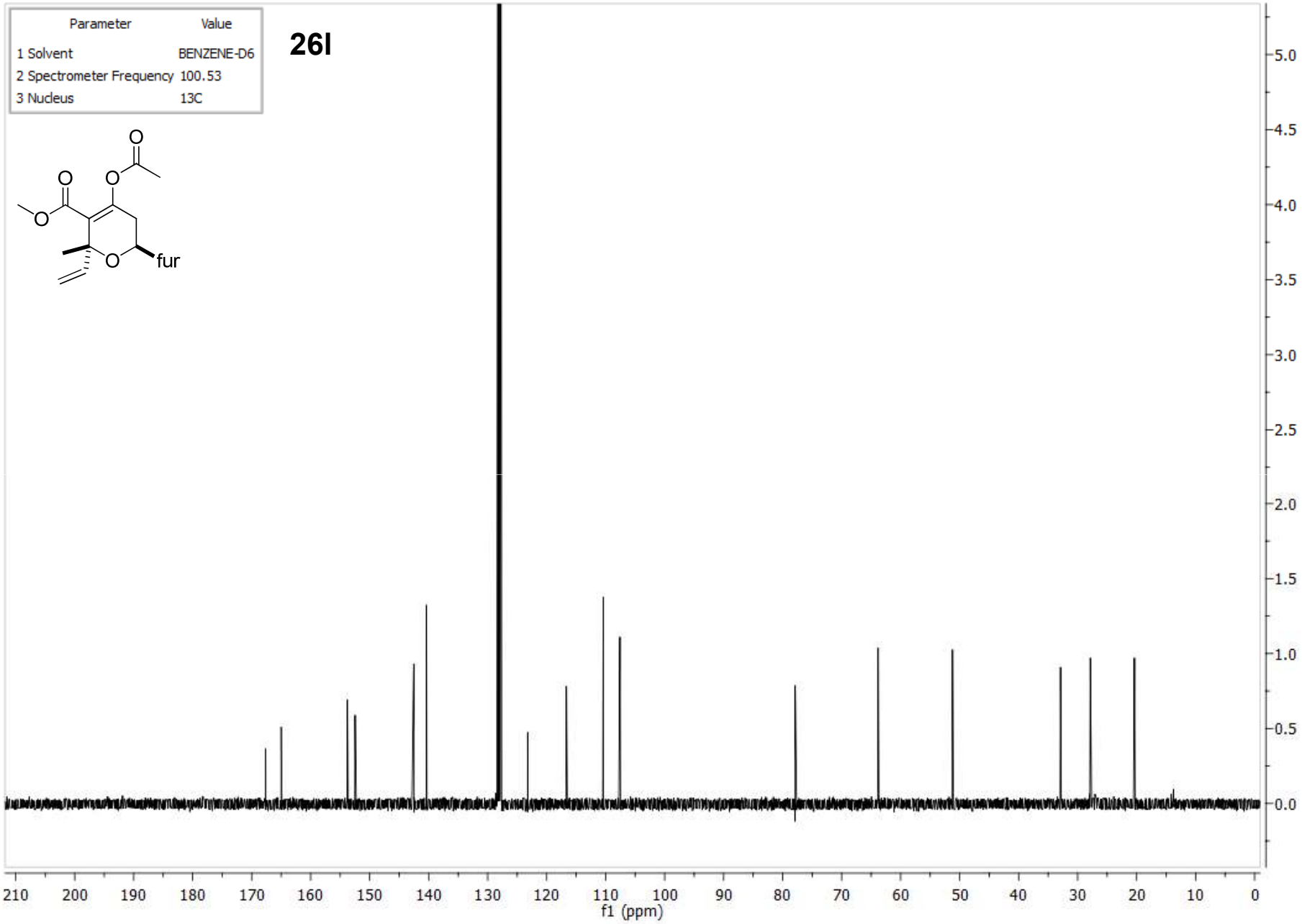

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
26j
Pr
1.00
1.04
1.04
1.03
3.03
1.03
1.00
2.87
2.76
2.24
2.12
2.75
f1 (ppm)

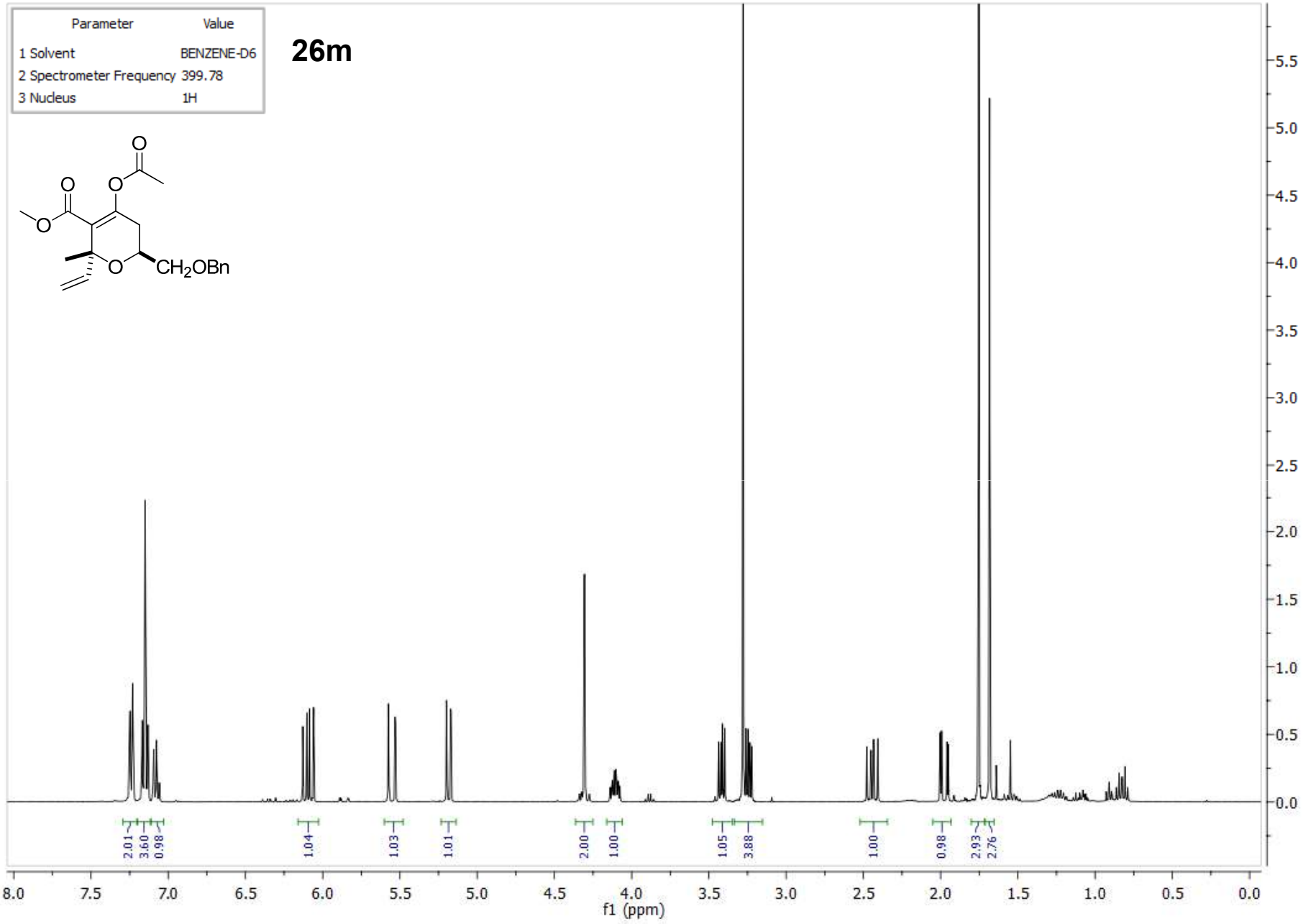

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
26j
O
O
O
O
Pr
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

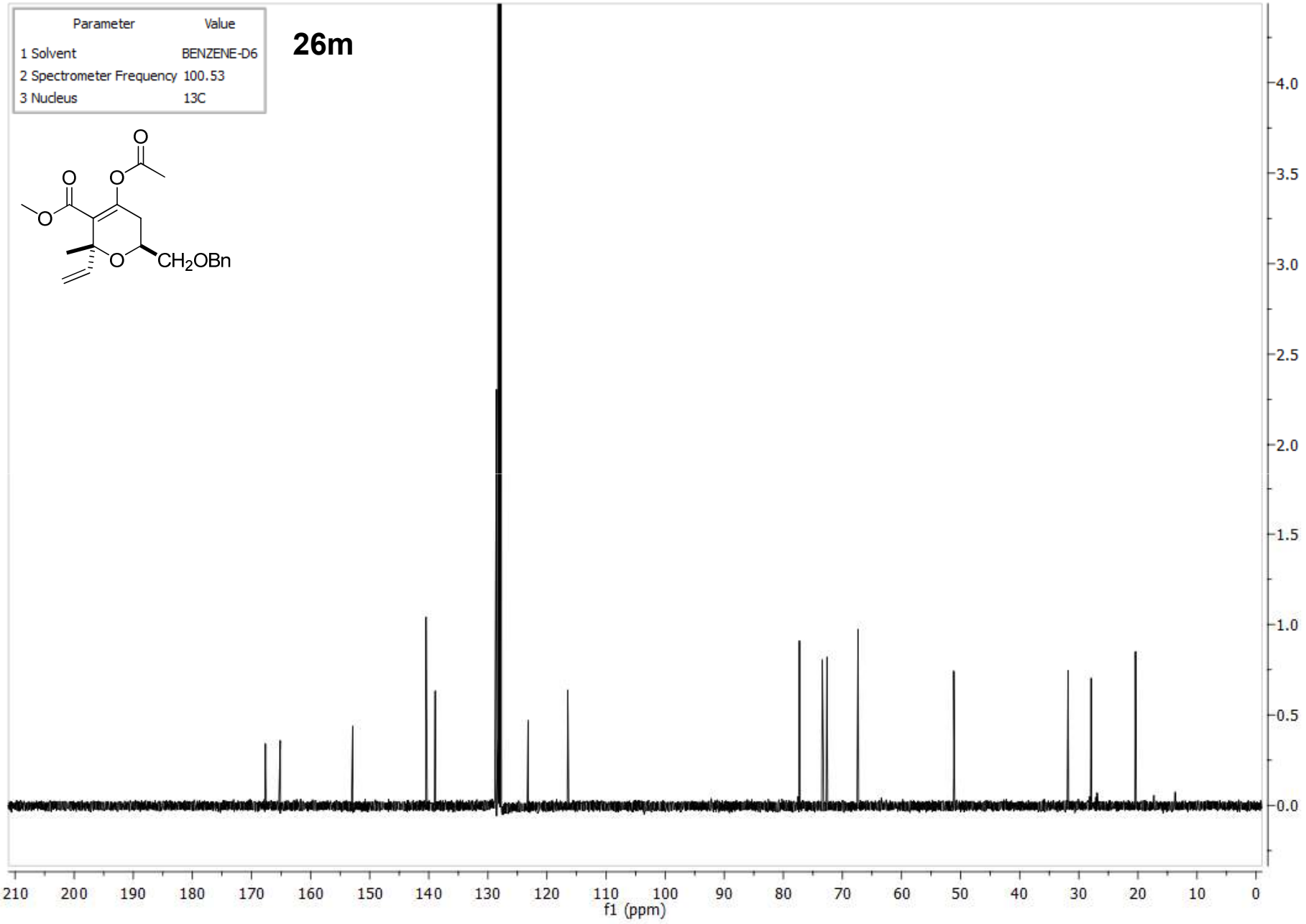

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
26k
O
O
O
O
O
iPr
1.00
1.04
1.04
1.04
2.98
1.02
0.99
2.85
2.73
1.05
2.82
2.69
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

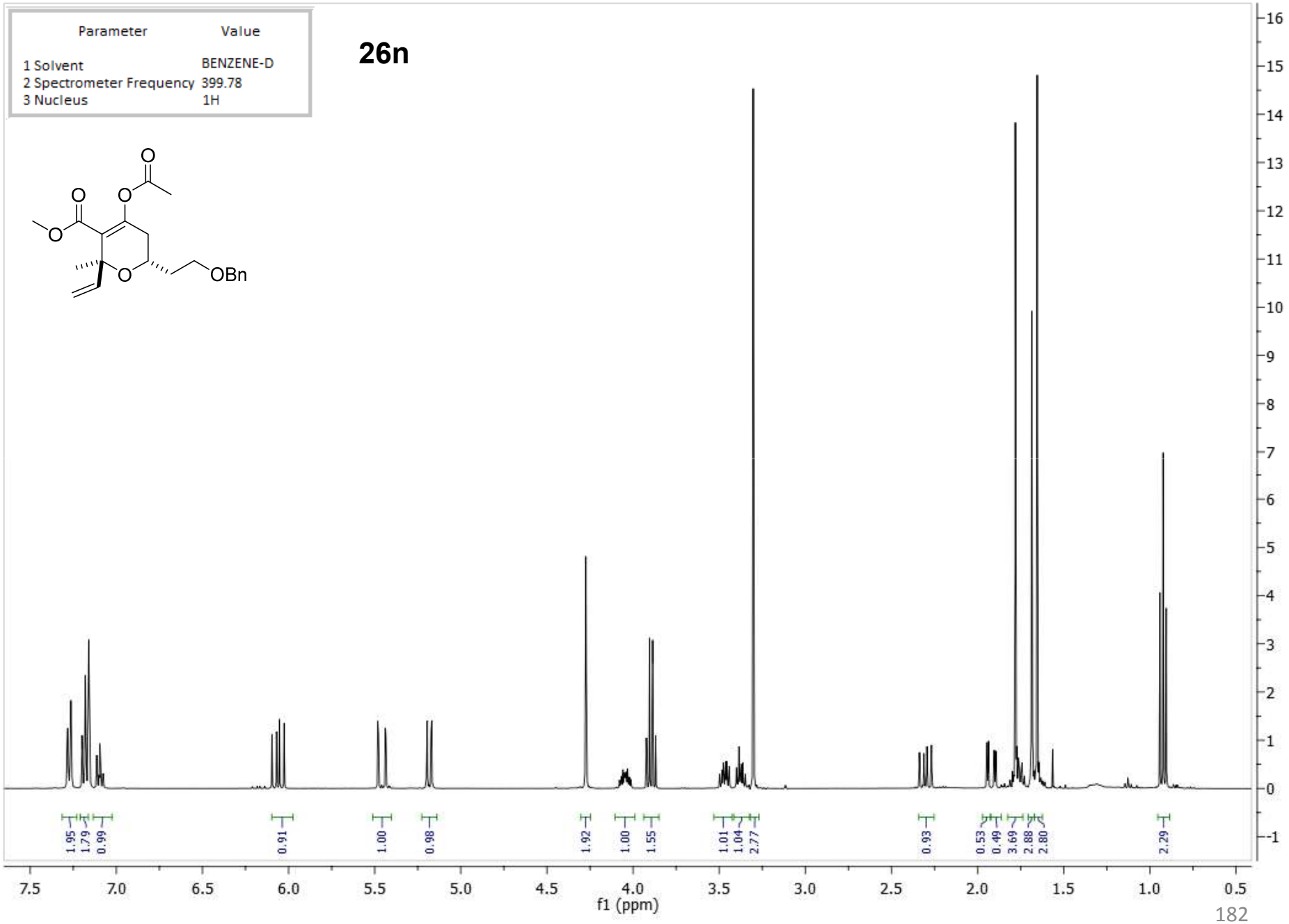

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
26k
O
O
O
O
iPr
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

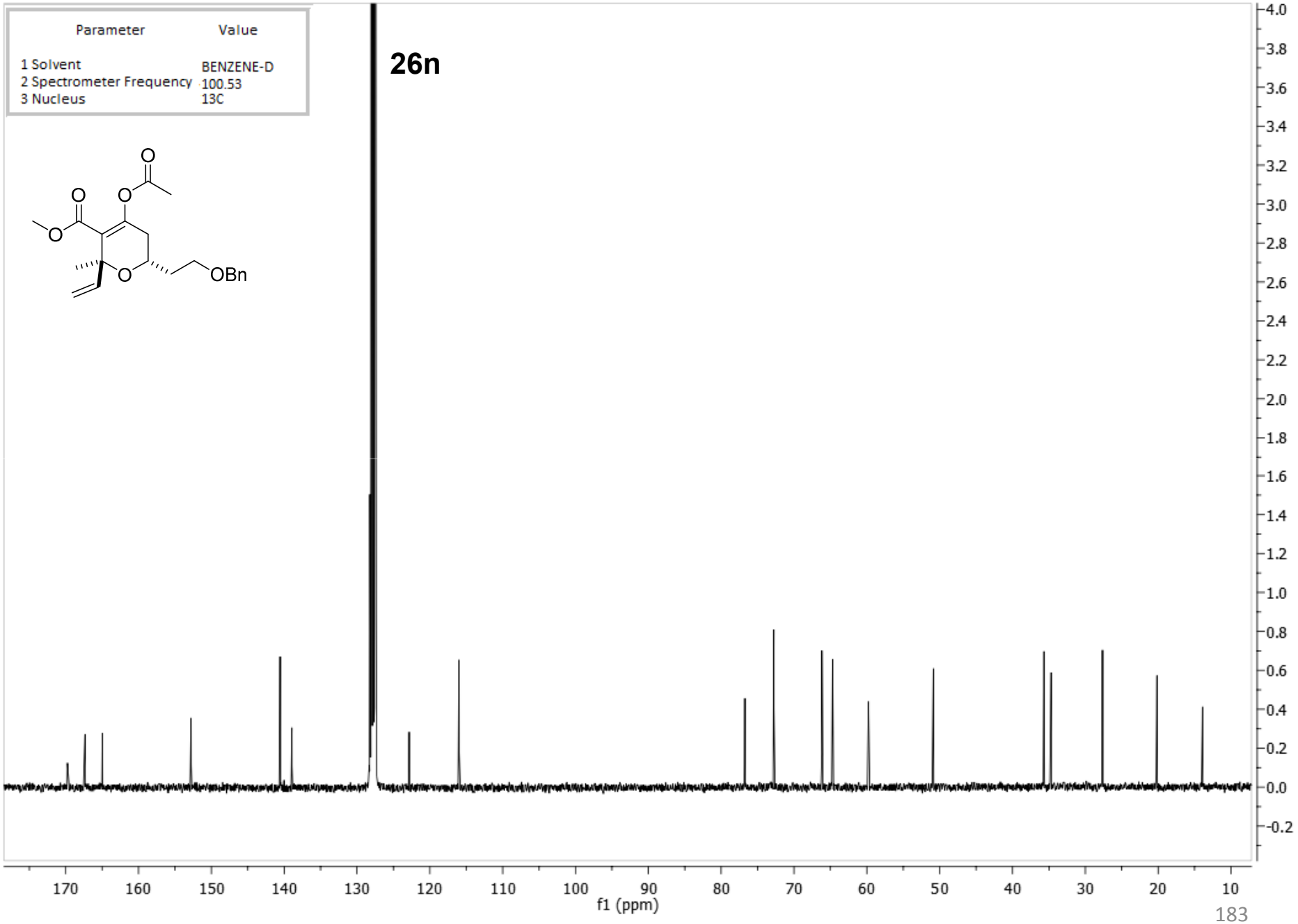

Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
26l
O
O
O
O
fur
0.94
3.09
1.02
1.03
1.00
2.81
0.99
1.02
2.77
2.82
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
26l
O
O
O
O
fur
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 399.78
3 Nucleus 1H
26m
O
O
O
O
CH2OBn
2.01
3.60
0.98
1.04
1.03
1.01
2.00
1.00
1.05
3.88
1.00
0.98
2.93
2.76
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


Parameter Value
1 Solvent BENZENE-D6
2 Spectrometer Frequency 100.53
3 Nucleus 13C
26m
O
O
O
O
CH2OBn
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Parameter Value
1 Solvent BENZENE-D
2 Spectrometer Frequency 399.78
3 Nucleus 1H
26n
OBn
1.95
1.79
0.99
0.91
1.00
0.98
1.92
1.00
1.55
1.01
1.04
2.77
0.93
0.53
0.49
3.69
2.88
2.80
2.29
f1 (ppm)


Parameter
Value
1 Solvent
BENZENE-D
2 Spectrometer Frequency
100.53
3 Nucleus
13C
26n
OBn
f1 (ppm)