Electronic Supplementary Material (ESI) for Green Chemistry

Solar Photochemistry: Optimisation of the Photo Friedel-Crafts Acylation of Naphthoquinones

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Supplementary Information

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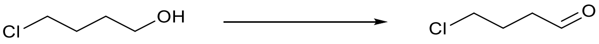
Experimental Details for the Preparation of Starting Materials

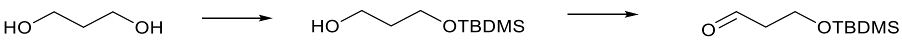
Preparation of Functionalised Aldehydes

4-Chlorobutanal



The reaction was carried out according to the procedure in Snyder *et al.*[1] with minor modifications. To 4-chloro-1-butanol (2.80 mL, 28.2 mmol) in dichloromethane (100 mL) was added PCC (7.30 g, 33.9 mmol) and silica gel (7.30 g) and the reaction mixture was stirred at ambient temperature for 16 h. The resulting black slurry was filtered through a short column of florasil, washed with ether (3 × 100 mL) and concentrated *in vacuo*. The *title compound* (2.17 g, 73%) was isolated as a colourless oil which was used in the next step without further purification; δ_H (400 MHz; $CDCl_3$) 9.79 (1 H, s, CH), 3.57 (2 H, t, *J* 6.50, CH_2Cl), 2.65 (2 H, td, *J* 7.0, 0.8, CH_2CO), 2.08 (2 H, pent, *J* 6.5, CH_2); δ_C (100 MHz; $CDCl_3$) 200.9 (CHO), 44.0 (CH_2), 40.8 (CH_2), 24.8 (CH_2). Data are consistent with those reported in the literature.[2]

3-(*t*-Butyldimethylsiloxy)propanal

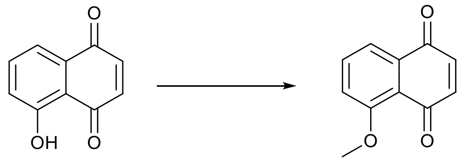


The reaction was carried out according to the procedure in McDougal *et al.*[3] with minor modifications. Sodium hydride (2.63 g, 65.7 mmol) was washed with pentane (3 × 10 mL) and dried under argon. THF (125 mL) was added followed by subsequent slow addition of the propane-1,3-diol (5.00 g, 65.7 mmol) and the reaction mixture was stirred at ambient temperature. After 45 min, TBDMS-Cl (9.90 g, 65.7 mmol) was added and the reaction mixture was stirred for a further 45 min. The reaction was diluted with ether (200 mL) and washed with sodium carbonate solution (200 mL), brine (200 mL), dried ($MgSO_4$), filtered and concentrated *in vacuo*. The residue was purified by flash chromatography on silica gel (light petroleum → 70:30 light petroleum/ethyl acetate) to afford product (10.6 g, 85%) as a colourless oil; δ_H (400 MHz; $CDCl_3$) 3.73 (1 H, td, *J* 6.1, 1.5, CH_2), 3.68 (1 H, td, *J* 5.8, 1.8, CH_2), 3.14 (1 H, s, OH), 1.69 (1 H, quint., *J* 5.6, CH_2), 0.83 (9 H, d, *J* 0.6, 3 × CH_3), 0.00 (6 H, d, *J* 1.6, 2 × CH_3); δ_C (100 MHz; $CDCl_3$) 62.0 (CH_2), 61.2 (CH_2), 34.4 (CH_2), 25.7 (CH_3), 18.0 (C), -5.67 (CH_3). Data are consistent with those reported in the literature.[3]

The next step was carried out according to the procedure by Grünanger *et al*.[4] Oxalyl chloride (1.04 mL, 12.2 mmol) in dichloromethane (27 mL) was cooled to -60 °C and DMSO (1.8 mL, 25.2 mmol) in dichloromethane (5 mL) was added and the reaction mixture stirred. After 5 min, 3-(*t*-butyldimethylsiloxy)propan-1-ol (2.00 g, 10.5 mmol) was added dropwise, followed by further dropwise addition of triethylamine (7.38 mL, 53.2 mmol). The reaction mixture was stirred whilst being gradually warmed to ambient temperature. After 1 h, water (50 mL) was added and the phases separated. The aqueous phase was back-extracted with further dichloromethane (50 mL) and the combined organic layers were washed with brine (100 mL), water (50 mL) and further brine (50 mL), dried ($MgSO_4$), filtered and concentrated *in vacuo*. The residue was dissolved in ether, filtered through Celite and concentrated *in vacuo* to afford the *title compound* (1.69 g, 86%) as a colourless oil; δ_H (400 MHz; $CDCl_3$) 9.79 (1 H, t, *J* 2.3, CH), 3.98 (2 H, t, *J* 6.0, CH_2), 2.59 (2 H, td, *J* 6.0, 2.2, CH_2), 0.87 (9 H, s, 3 × CH_3), 0.05 (6 H, s, 2 × CH_3); δ_C (100 MHz; $CDCl_3$) 201.9 (CH), 57.4 (CH_2), 46.5 (CH_2), 25.8 (CH_3), 18.2 (C), -5.5 (CH_3). Data are consistent with those previously reported.[5]

Preparation of Functionalised Naphthoquinones

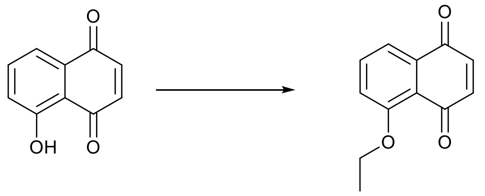
5-Methoxy-1,4-naphthoquinone 8b



8a **8b**

The reaction was carried out according to the procedure in Tietze *et al.*,[6] with minor modifications. To 5-hydroxy-1,4-naphthoquinone **8a** (100 mg, 0.57 mmol) and silver(I) oxide (100 mg, 0.46 mmol) in dichloromethane (2 mL) was added iodomethane (0.07 mL, 1.12 mmol) and the mixture stirred at ambient temperature. After 20 h further iodomethane (0.03 mL, 0.46 mmol) and silver(I) oxide (100 mg, 0.46 mmol) were added and the reaction mixture stirred for a further 3 h. The reaction mixture was filtered through Celite, washed (dichloromethane) and concentrated *in vacuo*. The residue was purified by flash chromatography on silica gel (light petroleum → 8:2 light petroleum: ethyl acetate) to give the *title compound* **8b** (96 mg, 90%) as fine orange crystals; mp 180-183 °C (lit.,[7] mp 180-185 °C); δ_H (400 MHz; $CDCl_3$) 7.72 (2 H, m, ArH), 7.32 (1 H, dd, *J* 8.2, 1.0, ArH), 6.87 (2 H, app. br s, CH), 4.02 (3 H, s, OMe); δ_C (100 MHz; $CDCl_3$) 185.1 (C), 184.3 (C), 159.6 (C), 140.8 (CH), 136.1 (CH), 134.9 (CH), 134.0 (C), 119.6 (C), 119.1 (CH), 117.9 (CH), 56.4 (CH_3). Data are consistent with those reported in the literature.[6,7]

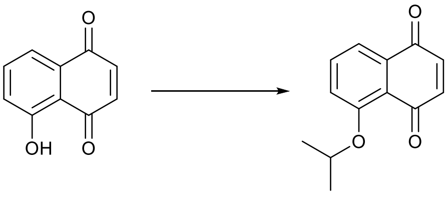
5-Ethoxy-1,4-naphthoquinone 8c



8a **8c**

To 5-hydroxy-1,4-naphthoquinone **8a** (100 mg, 0.57 mmol) and silver(I) oxide (100 mg, 0.46 mmol) in dichloromethane (2 mL) was added iodoethane (0.09 mL, 1.12 mmol) and the reaction mixture was stirred at ambient temperature. After 16 h further silver(I) oxide (100 mg, 0.46 mmol) and iodoethane (0.04 mL, 0.46 mmol) were added and the reaction mixture was stirred for a further 4 h. The reaction mixture was concentrated *in vacuo* and the product was crystallised from the minimum volume of hot toluene/light petroleum to yield the *title compound* **8c** (114 mg, 99%) as a brown solid; mp 78-79 °C; (Found: C, 71.28; H, 4.98; $C_{12}H_{10}O_3$ requires C, 71.32; H, 5.18 %); (Found: M+Na$^+$, 225.0521. $C_{12}H_{10}O_3Na^+$ requires 225.0522); λ_{max} (CH_2Cl_2)/nm 274 (log ε 5.71), 400 (4.37); ν_{max} ($CHCl_3$)/cm^{-1} 3686, 3012, 2415, 1521, 1239; δ_H (400 MHz; $CDCl_3$) 7.69 (1 H, dd, *J* 7.6, 1.4, ArH), 7.63 (1 H, app. br t, *J* 8.3, ArH), 7.27 (1 H, dd, *J* 8.3, 1.4, ArH), 6.83 (2 H, app. br s, CH), 4.20 (2 H, q, *J* 7.0, CH_2), 1.53 (3 H, t, *J* 7.0, CH_3); δ_C (100 MHz; $CDCl_3$) 185.3 (C), 184.2 (C), 159.0 (C), 140.9 (CH), 136.1 (CH), 134.8 (CH), 134.0 (C), 119.8 (C), 119.1 (CH), 119.0 (CH), 65.1 (CH_2), 14.6 (CH_3); *m/z* (ESI) 225 (M+Na$^+$, 100%), 103 (M+H$^+$, 5).

5-Isopropoxy-1,4-Naphthoquinone 8d

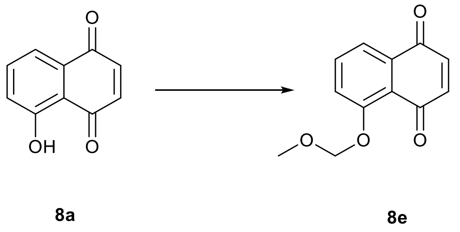


8a 8d

To 5-hydroxy-1,4-naphthoquinone **8a** (200 mg, 1.15 mmol) and silver(I) oxide (220 mg, 0.96 mmol) in dichloromethane (2 mL) was added 2-iodopropane (0.12 mL, 2.29 mmol) and the reaction mixture was stirred at ambient temperature. After 16 h further silver(I) oxide (440 mg, 1.92 mmol) and 2-iodopropane (0.4 mL, 9.20 mmol) were added and the reaction mixture was stirred for a further 4 h. The reaction mixture was concentrated *in vacuo* to yield the *title compound* **8d** (248 mg, 100%) as a dark green solid; mp 89-90 °C; (Found: M+Na$^+$, 239.0669. $C_{13}H_{12}O_3Na^+$ requires 239.0679);

λ_{max} (CH_2Cl_2)/nm 275 (log ε 4.40), 322 (2.91), 403 (3.46); ν_{max} (CH_2Cl_2)/cm^{-1} 2983, 1662, 1585, 1464, 1298, 1251; δ_H (400 MHz; $CDCl_3$) 7.70 (1 H, dd, *J* 7.6, 1.2, ArH), 7.63 (1 H, app. br t, *J* 8.3, ArH), 7.30 (1 H, app. br d, *J* 8.3, ArH), 6.85 (2 H, app. br s, CH), 4.69 (1 H, sept, *J* 6.2, CH), 1.45 (6 H, d, *J* 6.2, CH_3); δ_C (100 MHz; $CDCl_3$) 185.3 (C), 184.0 (C), 158.3 (C), 141.0 (CH), 136.0 (CH), 134.5 (CH), 134.2 (C), 121.3 (CH), 120.8 (C), 119.1 (CH), 72.3 (CH), 22.0 (CH_3); *m/z* (ESI) 239 (M+Na$^+$, 100%).

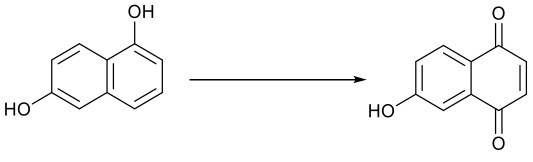
5-Methoxymethoxy-1,4-naphthoquinone 8e



The reaction was carried out according to the procedure in Nandaluru *et al.*[8] with minor modifications. To 5-hydroxy-1,4-naphthoquinone **8a** (200 mg, 1.15 mmol) and MOM-Cl (0.23 mL, 2.87 mmol) in dichloromethane (3 mL) at 0 °C was added DIPEA (0.40 mL, 2.30 mmol) dropwise over 5 min and the reaction mixture was stirred at ambient temperature for 16 h. To the reaction mixture was added saturated ammonium chloride solution (50 mL) and the phases separated. The aqueous layer was washed with dichloromethane (50 mL), and the organic layers combined, dried ($MgSO_4$), filtered and concentrated *in vacuo*. The residue was purified by flash chromatography on silica gel (4:1 light petroleum: ethyl acetate) to afford the *title compound* **8e** (179 mg, 79%) as an orange solid; mp 98-100 °C (lit.,[8] mp 98-101 °C); (Found: C, 66.00; H, 4.67; $C_{12}H_{10}O_4$ requires C, 66.05; H, 4.62 %); (Found: M+Na$^+$, 241.0467. $C_{12}H_{10}O_4Na^+$ requires 241.0471); λ_{max} (CH_2Cl_2)/nm 246 (log ε 5.16), 380 (4.28); ν_{max} ($CHCl_3$)/cm^{-1} 3012, 1663, 1587, 1335, 994; δ_H (400 MHz; $CDCl_3$) 7.75 (1 H, dd, *J* 7.5, 1.0, ArH), 7.63

(1 H, app. b. t, *J* 8.3, ArH), 7.51 (1 H, dd, *J* 8.3, 1.0, ArH), 6.85 (2 H, app. br. s, CH), 5.33 (2 H, s, CH_2), 3.52 (3 H, s, OCH_3); δ_C (100 MHz; $CDCl_3$) 184.8 (C), 184.0 (C), 157.0 (C), 140.6 (CH), 136.2 (CH), 134.6 (CH), 133.8 (C), 122.2 (CH), 120.5 (C), 120.3 (CH), 95.0 (CH_2), 56.5 (CH_3); *m/z* (ESI) 241 ($M+Na^+$, 100%).

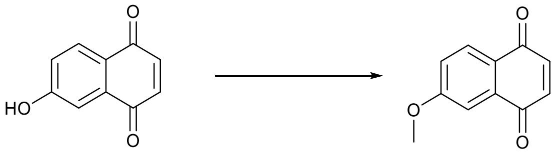
6-Hydroxy-1,4-naphthoquinone 12a



11 **12a**

A stirred solution of 1,6-dihydroxynapthalene **11** (5.00 g, 31.2 mmol) and salcomine (507 mg, 1.56 mmol) in DMF (33 mL) was bubbled rapidly with oxygen at ambient temperature. After 16 h, ether (200 mL) and brine (200 mL) were added and phases separated. The aqueous layer was back-extracted with further ether (10 × 200 mL) and the organic layers combined, dried (Na_2SO_4), filtered and concentrated *in vacuo*. The crude product was purified by flash chromatography on silica gel (7:3 light petroleum: ethyl acetate) to give the *title compound* **12a** (2.50 g, 46%) as a bright orange solid; mp 170-171 °C (decomp) (lit.,[9] mp 170 °C); (Found: C, 68.67; H, 3.42; $C_{10}H_6O_3$ requires C, 68.97; H, 3.47 %); (Found: $M-H^+$, 173.0238. $C_{10}H_5O_3^-$ requires 173.0244); λ_{max} (CH_2Cl_2)/nm 274 (log ε 5.26), 385 (3.26); ν_{max} ($CHCl_3$)/cm^{-1} 3689, 3607, 3045, 1602, 1523, 1240; δ_H (400 MHz; MeOD) 7.92 (1 H, d, *J* 8.5, ArH), 7.34 (1 H, d, *J* 2.6, ArH), 7.13 (1 H, dd, *J* 8.5, 2.6, ArH), 6.90 (2 H, d, *J* 2.1, CH); δ_C (100 MHz; MeOD) 186.7 (C), 185.6 (C), 164.6 (C), 140.3 (CH), 139.5 (CH), 135.6 (C), 130.2 (CH), 125.8 (C), 121.9 (CH), 113.2 (CH); *m/z* (ESI) 173 ($M-H^+$, 100%).

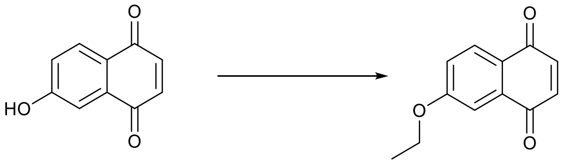
6-Methoxy-1,4-naphthoquinone 12b



12a **12b**

To 6-hydroxy-1,4-naphthoquinone **12a** (100 mg, 0.57 mmol) and silver(I) oxide (200 mg, 0.92 mmol) in CH_2Cl_2 (2 mL) was added iodomethane (0.1 mL, 1.60 mmol) and the mixture stirred at ambient temperature for 3 d. The reaction mixture was filtered through Celite, washed with dichloromethane (50 mL) and concentrated *in vacuo* to afford the *title compound* **12b** (107 mg, 100%) as a fine yellow solid; mp 132–133 °C (lit.,[10] mp 133-135 °C); (Found: C, 69.82; H, 4.22; $C_{11}H_8O_3$ requires C, 70.21; H, 4.29 %); (Found: M+H$^+$, 189.0546. $C_{11}H_9O_3^+$ requires 189.0546); λ_{max} (CH_2Cl_2)/nm 261 (log ε 4.91), 382 (3.89); ν_{max} ($CHCl_3$)/cm^{-1}; 3012, 1667, 1593, 131, 1292; δ_H (400 MHz; $CDCl_3$) 8.02 (1 H, d, *J* 8.6, ArH), 7.50 (1 H, d, *J* 2.7, ArH), 7.21 (1 H, dd, *J* 8.6, 2.7, ArH), 6.92 (2 H, app br. s, CH), 3.95 (3 H, s, OCH_3); δ_C (100 MHz; $CDCl_3$) 185.2 (C), 184.1 (C), 164.1 (C), 139.0 (CH), 138.2 (CH), 133.9 (C), 128.9 (CH), 125.5 (C), 120.5 (CH), 109.6 (CH), 55.9 (CH_3); *m/z* (ESI) 399 (2M+Na$^+$, 100%), 211 (M+Na$^+$, 98), 189 (M+H$^+$, 47).

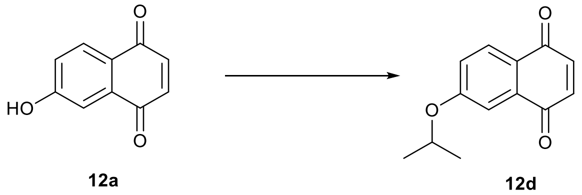
6-Ethoxy-1,4-naphthoquinone 12c



12a **12c**

To 6-hydroxy-1,4-naphthoquinone **12a** (50 mg, 0.29 mmol) and silver(I) oxide (106 mg, 0.46 mmol) in CH_2Cl_2 (1 mL) was added iodoethane (0.06 mL, 0.81 mmol) and the mixture stirred at ambient temperature for 16 h. The reaction mixture was filtered through Celite, washed with dichloromethane (50 mL) and concentrated *in vacuo* to afford the *title compound* **12c** (44 mg, 76%) as a fine yellow solid; mp 116-117 °C; (Found: C, 71.07; H, 4.99; $C_{13}H_{12}O_3$ requires C, 71.28; H, 4.98%); (Found: M+Na$^+$, 225.0522. $C_{12}H_{10}O_3Na^+$ requires 225.0522); λ_{max} (CH_2Cl_2)/nm 255 (log ε 4.37), 263 (4.39), 383 (3.27), 386 (3.20); ν_{max} ($CHCl_3$)/cm^{-1}; 3012, 2415, 1666, 1595, 1333, 1239, 965; δ_H (400 MHz; $CDCl_3$) 8.02 (1 H, d, *J* 8.6, ArH), 7.50 (1 H, d, *J* 2.7, ArH), 7.20 (1 H, dd, *J* 8.6, 2.7, ArH), 6.92 (2 H, app br. s, CH), 4.19 (2 H, q, *J* 6.9, CH_2), 1.48 (3 H, t, *J* 7.0, CH_3); δ_C (100 MHz; $CDCl_3$) 185.2 (C), 184.1 (C), 163.6 (C), 139.0 (CH), 138.2 (CH), 133.9 (C), 128.9 (CH), 125.3 (C), 120.8 (CH), 110.1 (CH), 64.4 (CH_2), 14.6 (CH_3); *m/z* (ESI) 225 (M+Na$^+$, 100%), 203 (M+H$^+$, 35).

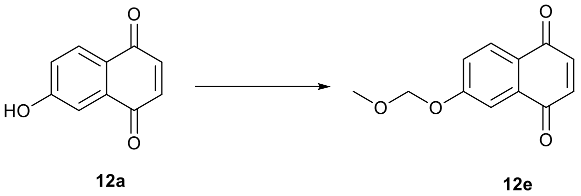
6-Isopropoxy-1,4-naphthoquinone 12d



To 6-hydroxy-1,4-naphthoquinone **12a** (100 mg, 0.57 mmol) and silver(I) oxide (110 mg, 0.48 mmol) in dichloromethane (1 mL) was added 2-iodopropane (0.1 mL, 1.12 mmol) and the mixture stirred at ambient temperature. After 16 h further silver(I) oxide (220 mg, 0.96 mmol) and 2-iodopropane (0.2 mL, 2.2 mmol) was added and the reaction mixture was stirred for a further 4 h. The reaction mixture was concentrated *in vacuo* to yield the *title compound* **12d** (123 mg, 100%) as a pale green solid; mp 111-113 °C; (Found: M+Na$^+$, 239.0671. $C_{13}H_{12}O_3Na^+$ requires 239.0679); λ_{max} (CH_2Cl_2)/nm 257 (log ε 4.54), 264 (4.56), 330 (2.96), 390 (3.54); ν_{max} ($CHCl_3$)/cm^{-1}; 2983, 1665, 1592, 1491, 1316, 1110, 1045, 961; δ_H (400 MHz; $CDCl_3$) 8.02 (1 H, d, *J* 8.6, ArH), 7.48

(1 H, d, *J* 2.6, ArH), 7.17 (1 H, dd, *J* 8.6, 2.7, ArH), 6.92 (2 H, app. br. s, CH), 4.76 (1 H, sept, *J* 6.0, CH), 1.40 (6 H, d, *J* 6.0, CH_3); δ_C (100 MHz; $CDCl_3$) 185.3 (C), 184.1 (C), 162.7 (C), 139.0 (CH), 138.2 (CH), 133.9 (C), 129.0 (CH), 125.0 (C), 121.7 (CH), 111.0 (CH), 70.8 (CH), 21.8 (CH_3); *m/z* (ESI) 239 (M+Na$^+$, 100%), 217 (M+H$^+$, 11).

6-Methoxymethoxy-1,4-naphthoquinone 12e



To 6-hydroxy-1,4-naphthoquinone **12a** (100 mg, 0.57 mmol) and MOM-Cl (0.12 mL, 1.44 mmol) in dichloromethane (2 mL) at 0 °C was added DIPEA (0.2 mL, 1.15 mmol) dropwise over 5 min and the reaction mixture was stirred at ambient temperature for 16 h. To the reaction mixture was added saturated ammonium chloride solution (50 mL) and the phases separated. The aqueous layer was washed with dichloromethane (50 mL) and the organic layers combined, dried ($MgSO_4$), filtered and concentrated *in vacuo*. The residue was purified by flash chromatography on silica gel (4:1 light petroleum: ethyl acetate) to afford the *title compound* **12e** (82 mg, 66%) as an orange solid; mp 110-112 °C; (Found: M+Na$^+$, 241.0468. $C_{12}H_{10}O_4Na^+$ requires 241.0471); λ_{max} (CH_2Cl_2)/nm 254 (log ε 4.69), 260 (4.69), 372 (3.69); ν_{max} ($CHCl_3$)/cm^{-1}; 3012, 1668, 1595, 1312, 988; δ_H (400 MHz; $CDCl_3$) 8.00 (1 H, d, *J* 8.6, ArH), 7.62 (1 H, d, *J* 2.6, ArH), 7.32 (1 H, dd, *J* 8.6, 2.6, ArH), 6.91 (2 H, app br. s, CH), 5.29 (2 H, s, CH_2), 3.48 (3 H, s, CH_3); δ_C (100 MHz; $CDCl_3$) 184.8 (C), 184.0 (C), 161.6 (C), 138.8 (CH), 138.3 (CH), 133.8 (C), 128.8 (CH), 126.1 (C), 121.5 (CH), 112.5 (CH), 94.1 (CH_2), 56.4 (CH_3); *m/z* (ESI) 241 (M+Na$^+$, 100%), 219 (M+H$^+$, 17).

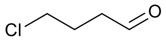
X-Ray Crystallographic Studies

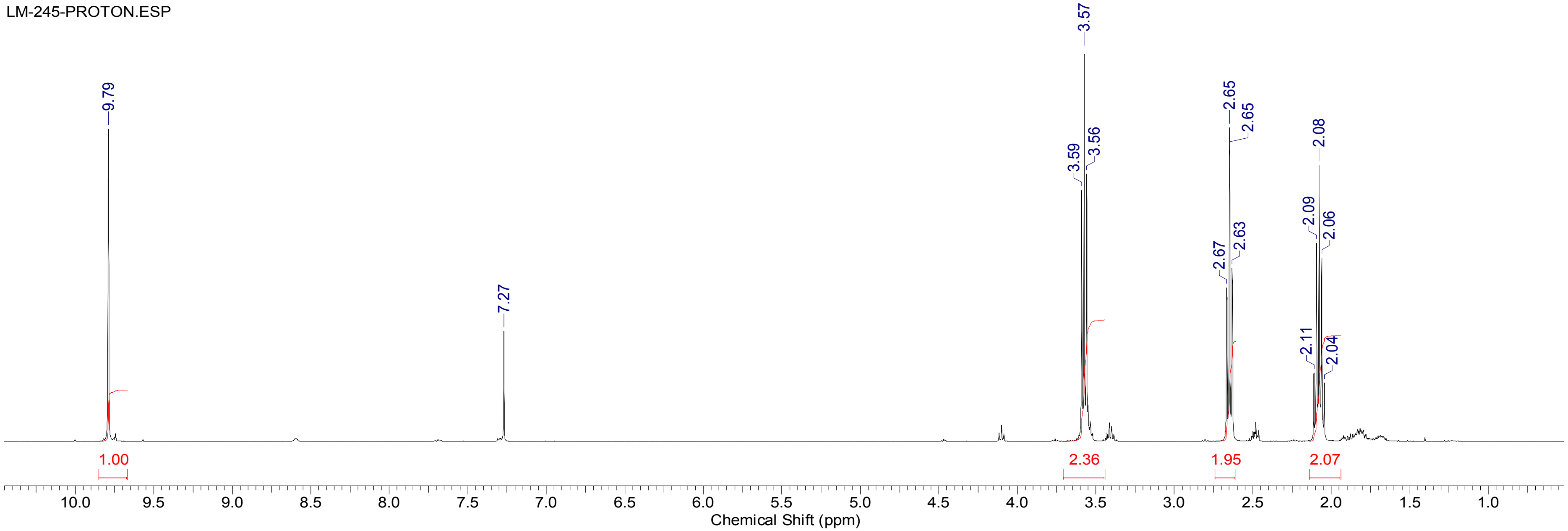
All data sets were collected either with a SuperNova Cu diffractometer or a GV1000 diffractometer. The crystal was kept at 120(2)K during data collection. Using Olex2, the structure was solved with the olex2.solve structure solution program using Charge Flipping and refined with the ShelXL refinement package using Least Squares minimisation. Crystal structure data and details are listed in Table 1.

Table 1. Crystal structure data

Compound		**9e**	**10c**	**13b**
CCDC		952123	952121	952122
Empirical formula		$C_{16}H_{18}O_5$	$C_{16}H_{18}O_4$	$C_{15}H_{16}O_4$
Formula weight		290.30	274.30	260.28
Crystal size/mm^3		0.51 × 0.0864 × 0.0426	0.126 × 0.1016 × 0.0629	0.1243 × 0.0791 × 0.0316
Crystal system		monoclinic	monoclinic	orthorhombic
Space group		$P2_1/c$	$P2_1/n$	Pbca
Unit cell dimensions	a/Å	15.7243(11)	7.7968(2)	12.8287(3)
	b/Å	5.0123(3)	21.1512(5)	7.9789(3)
	c/Å	18.5815(12)	9.1046(3)	24.2643(9)
	α/°	90	90	90
	β/°	107.285	114.189	90
	γ/°	90	90	90
Volume/Å^3		1398.35(17)	1369.64(7)	2483.67(15)
Z		4	4	8
Reflections collected		5181	12604	13106
Independent reflections		2731 [R(int) = 0.0207]	2766 [R(int) = 0.0207]	2494 [R(int) = 0.0371]
Final R indexes R [I>=2σ (I)]		R_1 = 0.0362, wR_2 = 0.0987	R_1 = 0.0329, wR_2 = 0.0900	R_1 = 0.0438, wR_2 = 0.1128
Final R indexes (all data)		R_1 = 0.0362, wR_2 = 0.0987	R_1 = 0.0378, wR_2 = 0.0941	R_1 = 0.0525 wR_2 = 0.1191

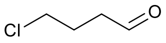
4-Chlorobutanal

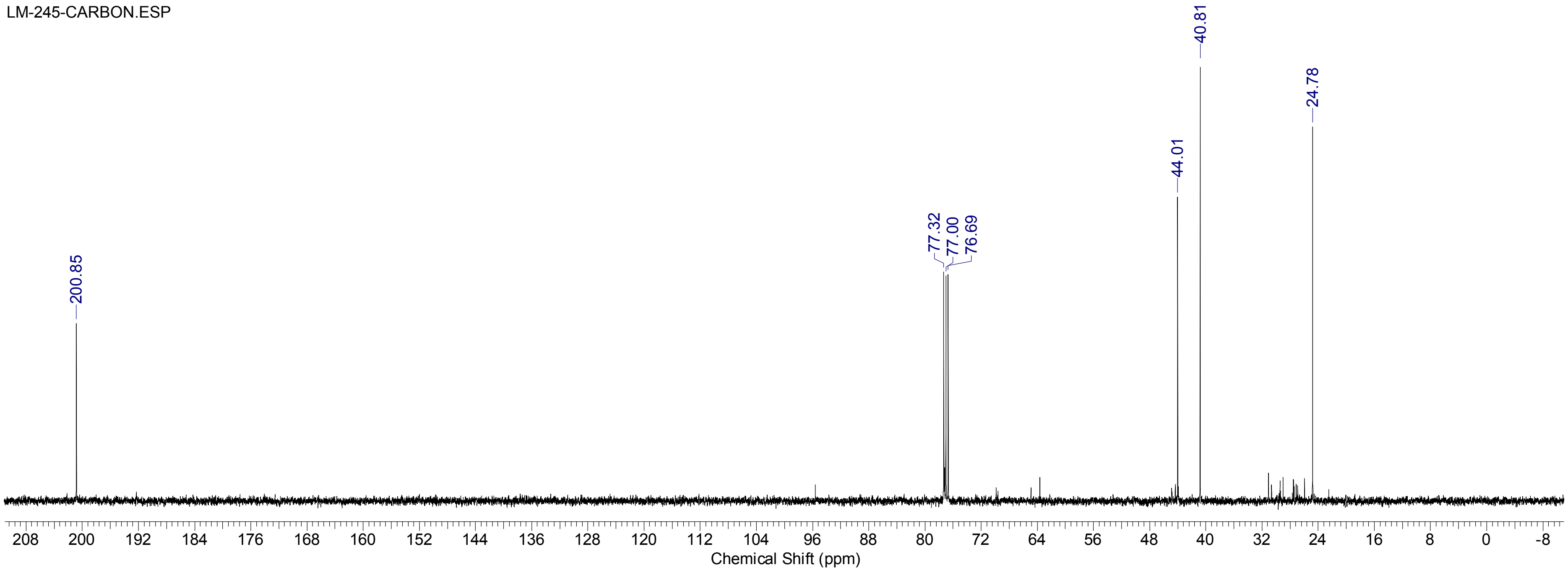




^{1}H NMR at 400 MHz in $CDCl_3$

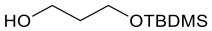
4-Chlorobutanal

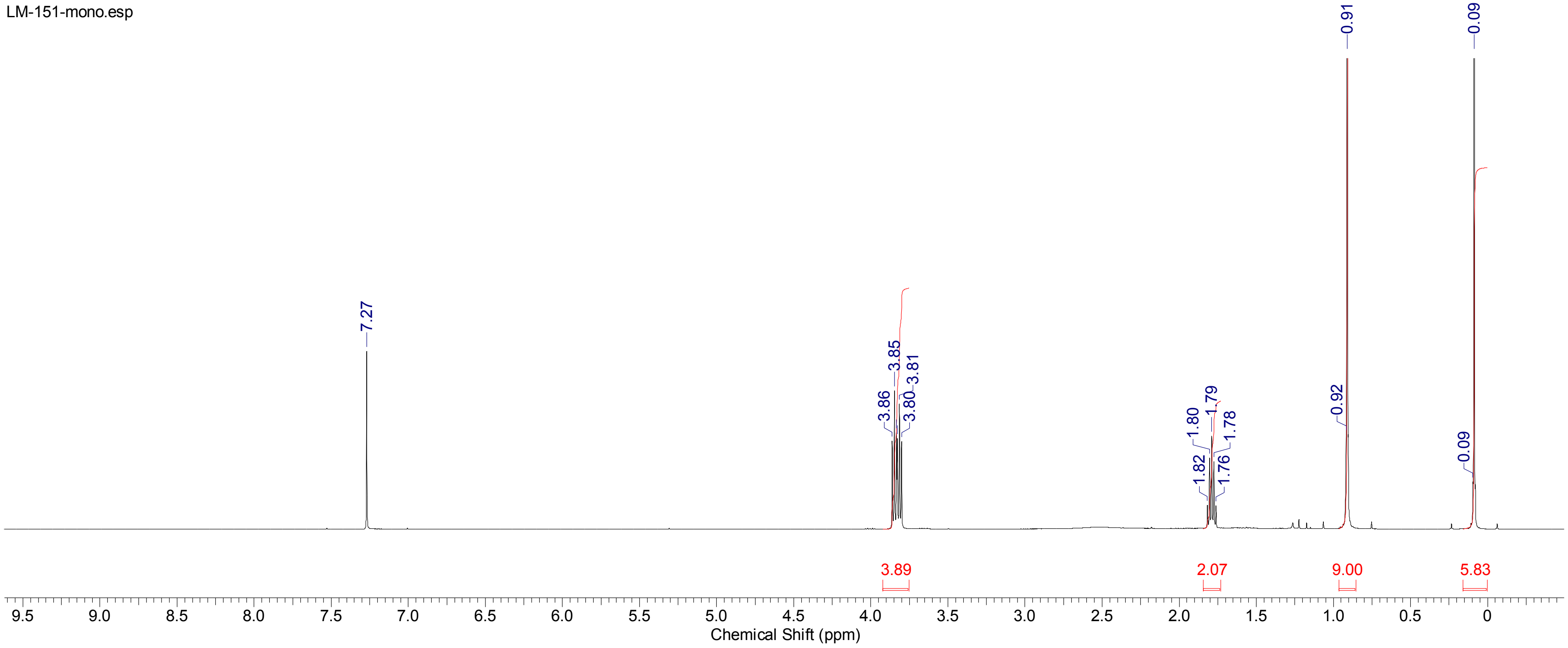




^{13}C NMR at 100 MHz in $CDCl_3$

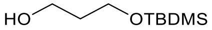
3-((*t*-butyldimethylsilyl)oxy)propan-1-ol

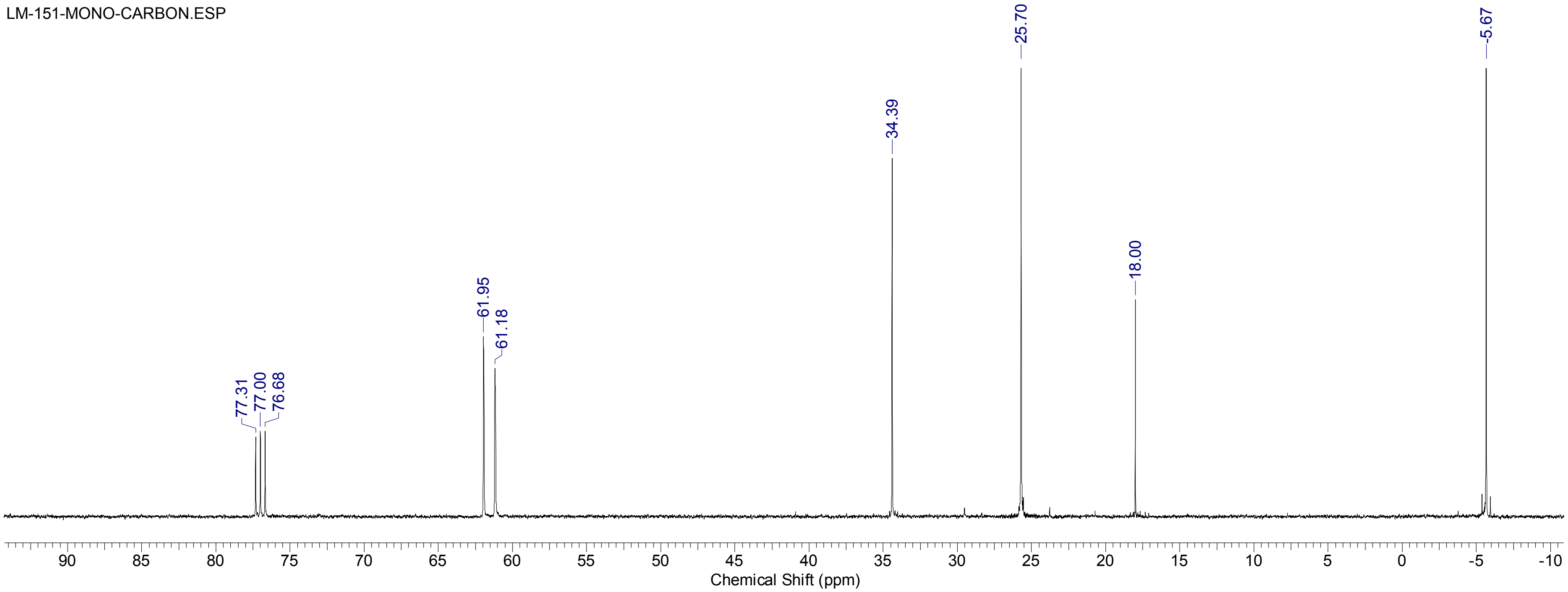




^{1}H NMR at 400 MHz in $CDCl_3$

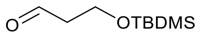
3-((*t*-butyldimethylsilyl)oxy)propan-1-ol

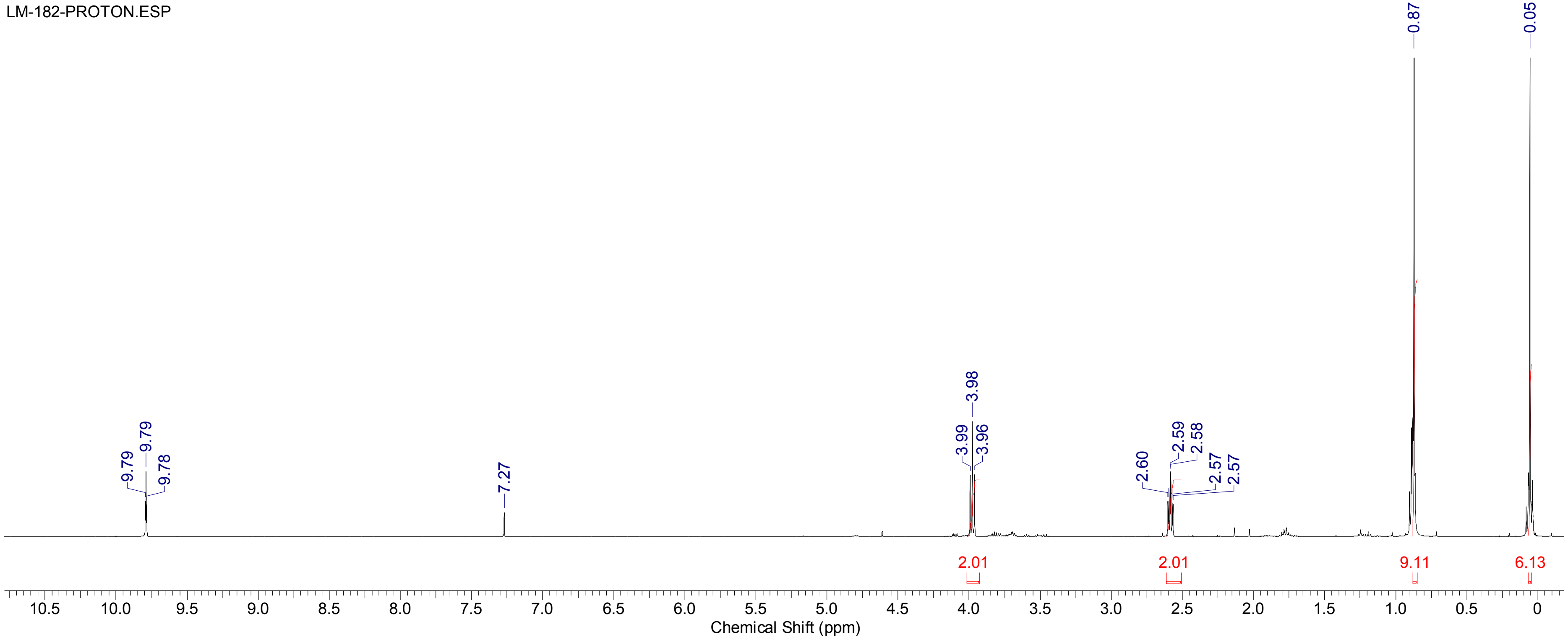




^{13}C NMR at 100 MHz in $CDCl_3$

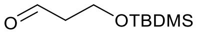
3-(*t*-Butyldimethylsiloxy)propanal

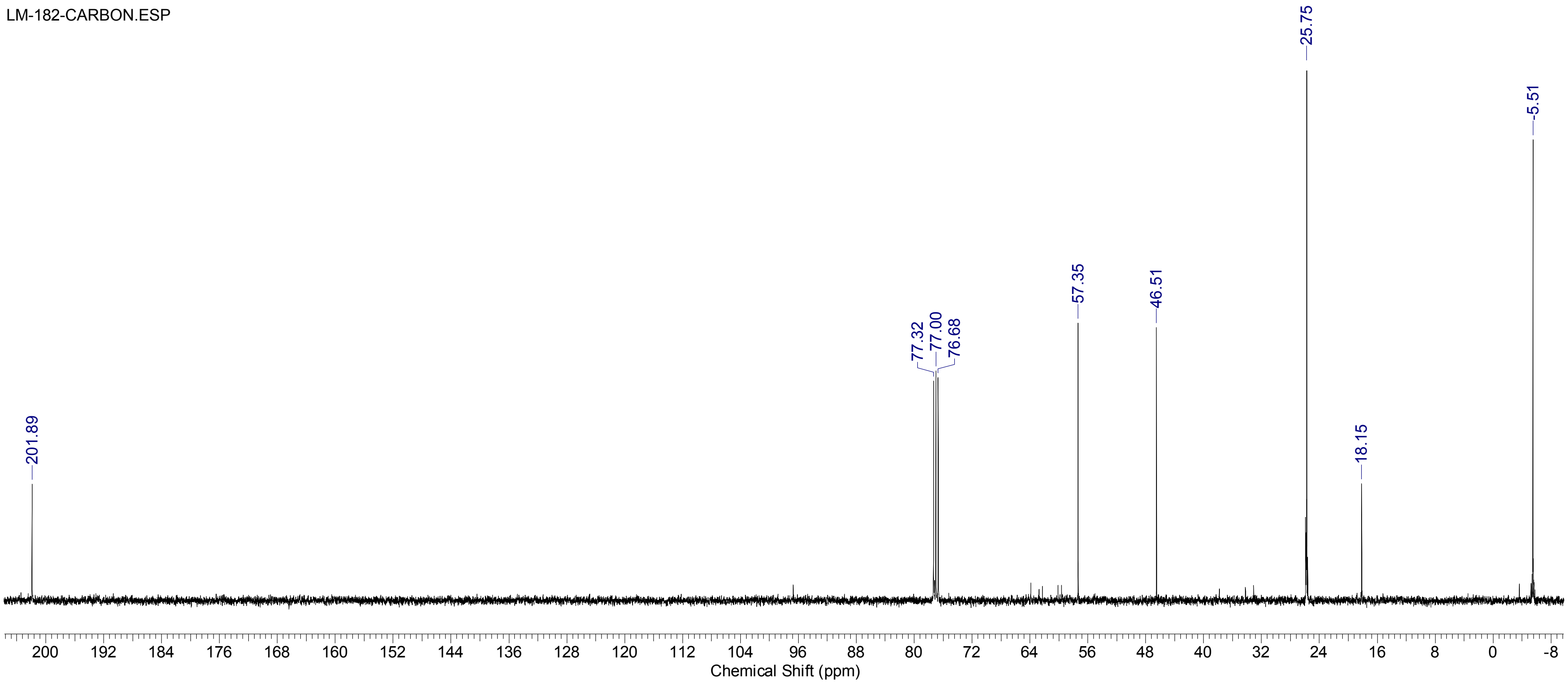




^{1}H NMR at 400 MHz in $CDCl_3$

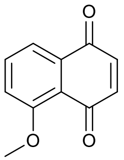
3-(t-Butyldimethylsiloxy)propanal

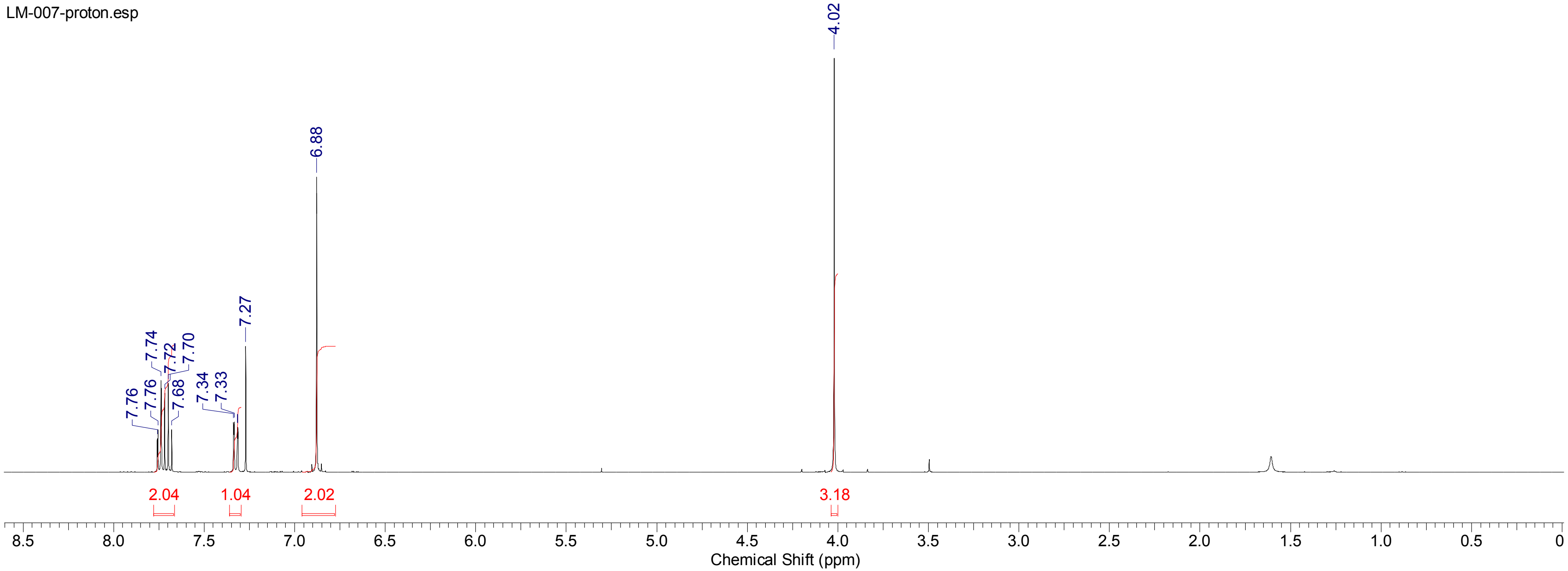




^{13}C NMR at 100 MHz in $CDCl_3$

5-Methoxy-1,4-naphthoquinone 8b





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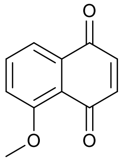
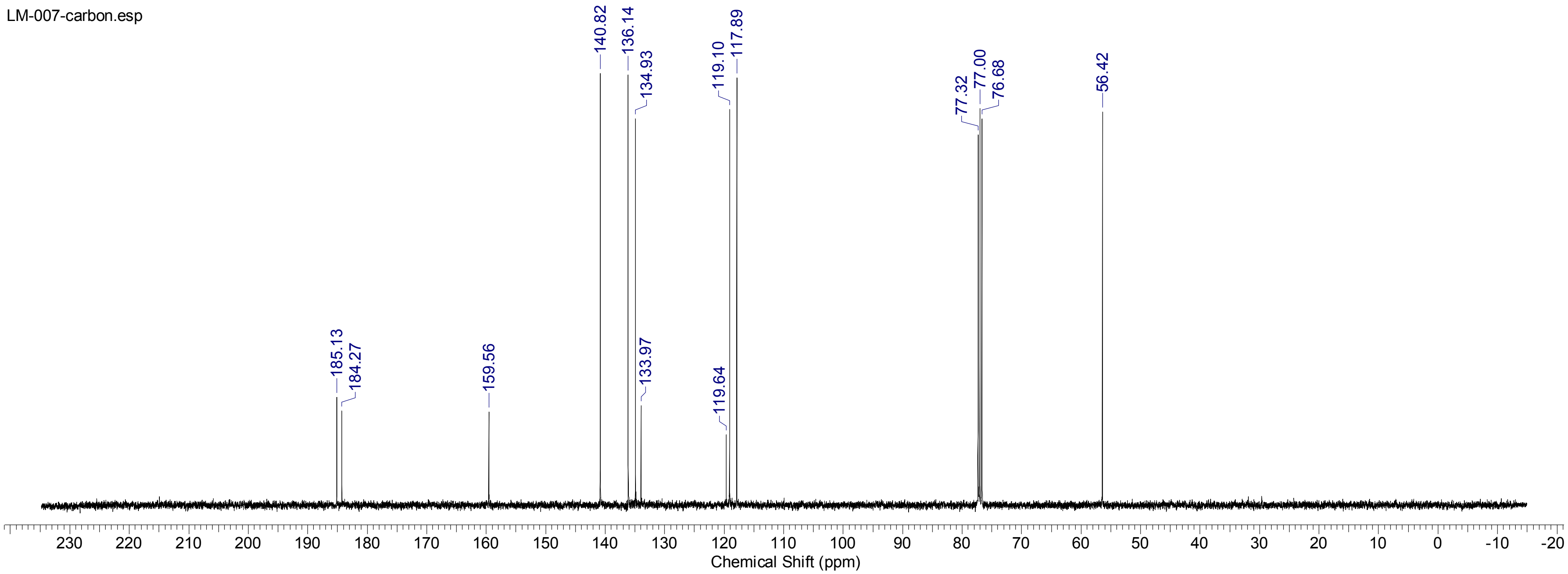
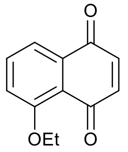
5-Methoxy-1,4-naphthoquinone 8b

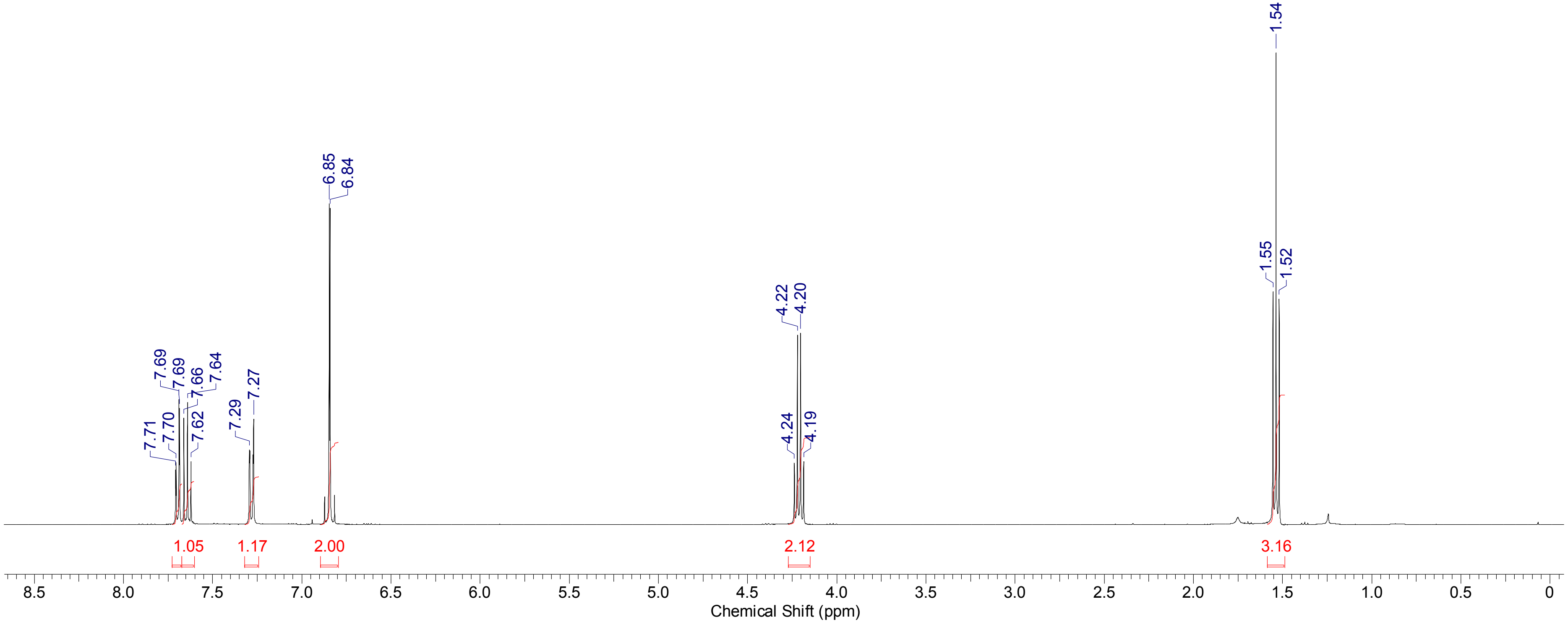




^{13}C NMR at 100 MHz in $CDCl_3$

5-Ethoxy-1,4-naphthoquinone 8c





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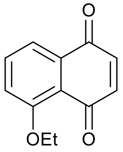
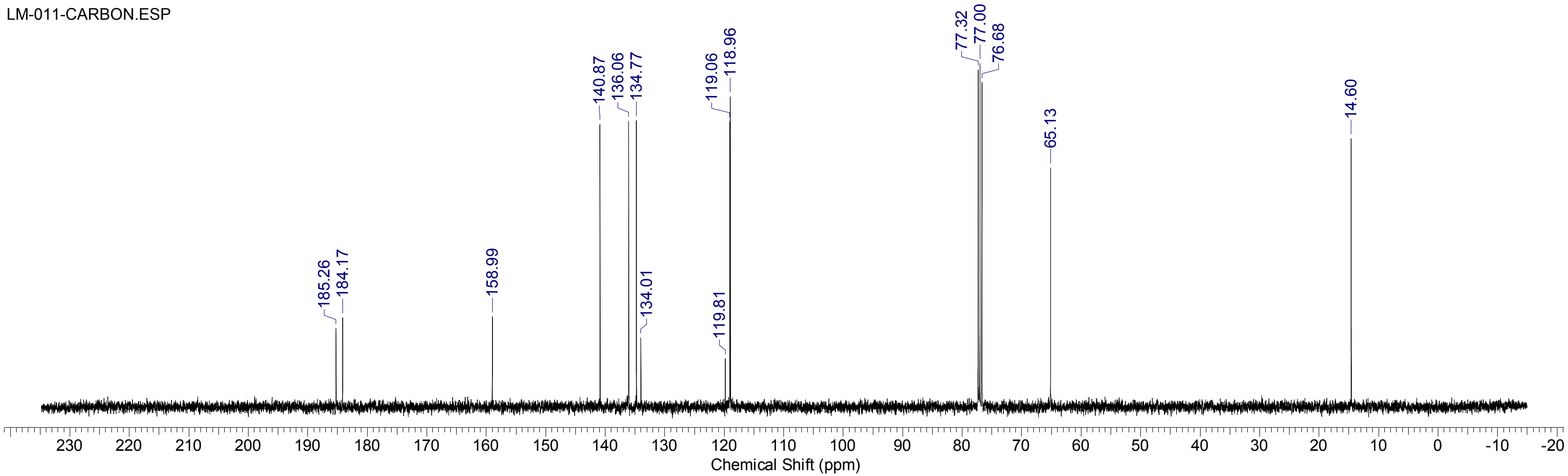
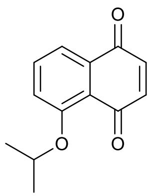
5-Ethoxy-1,4-naphthoquinone 8c

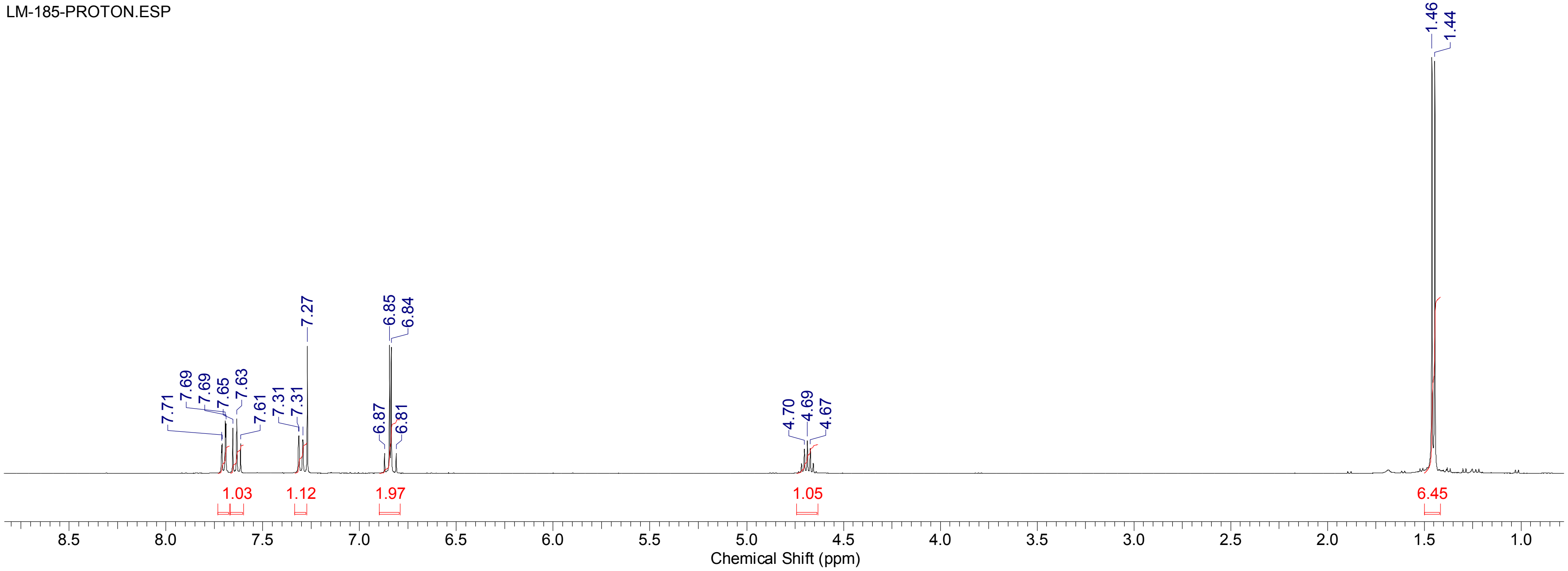




^{13}C NMR at 100 MHz in $CDCl_3$

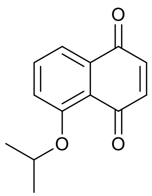
5-Isopropoxy-1,4-Naphthoquinone 8d

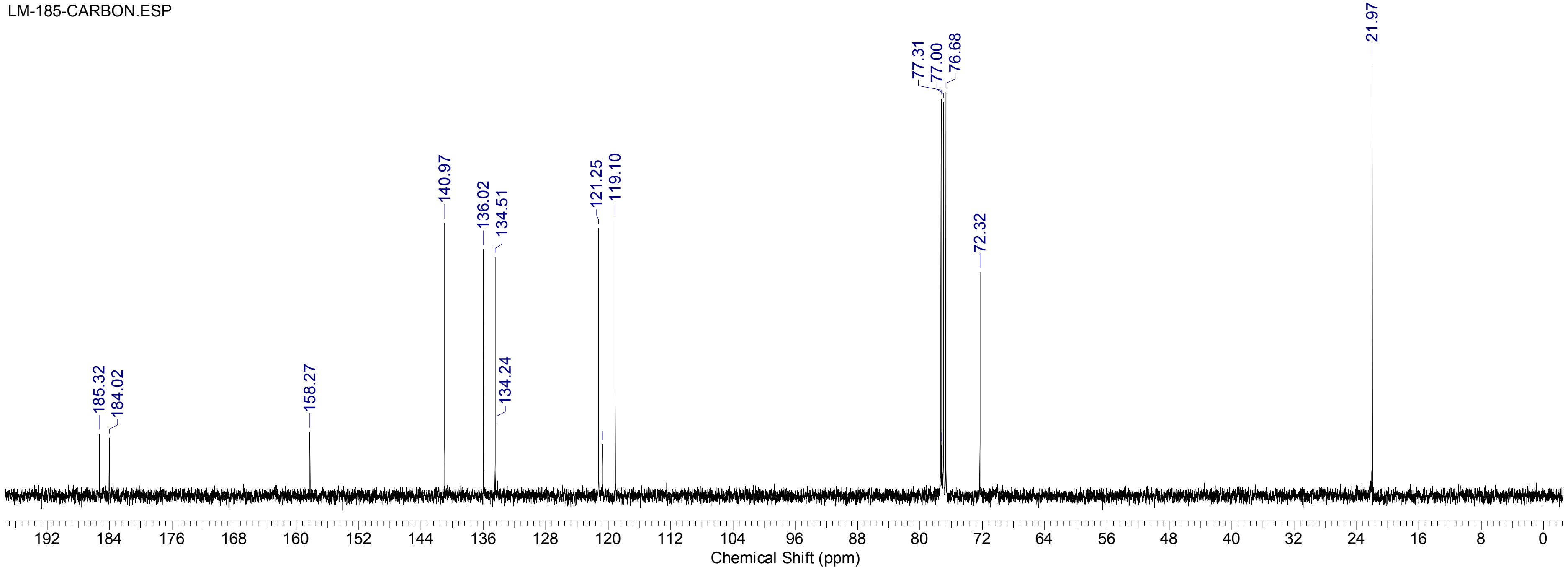




^{1}H NMR at 400 MHz in $CDCl_3$

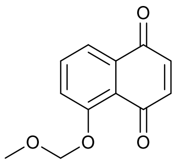
5-Isopropoxy-1,4-Naphthoquinone 8d

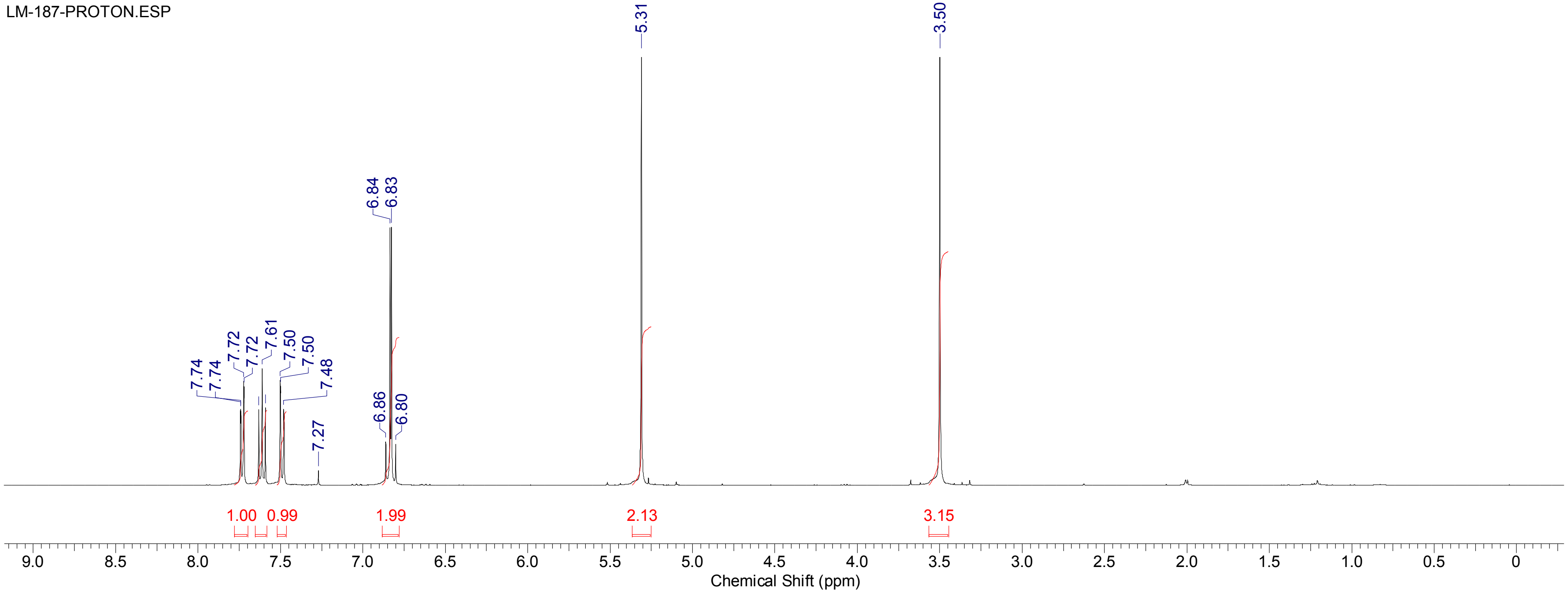




^{13}C NMR at 100 MHz in CDCl$_3$

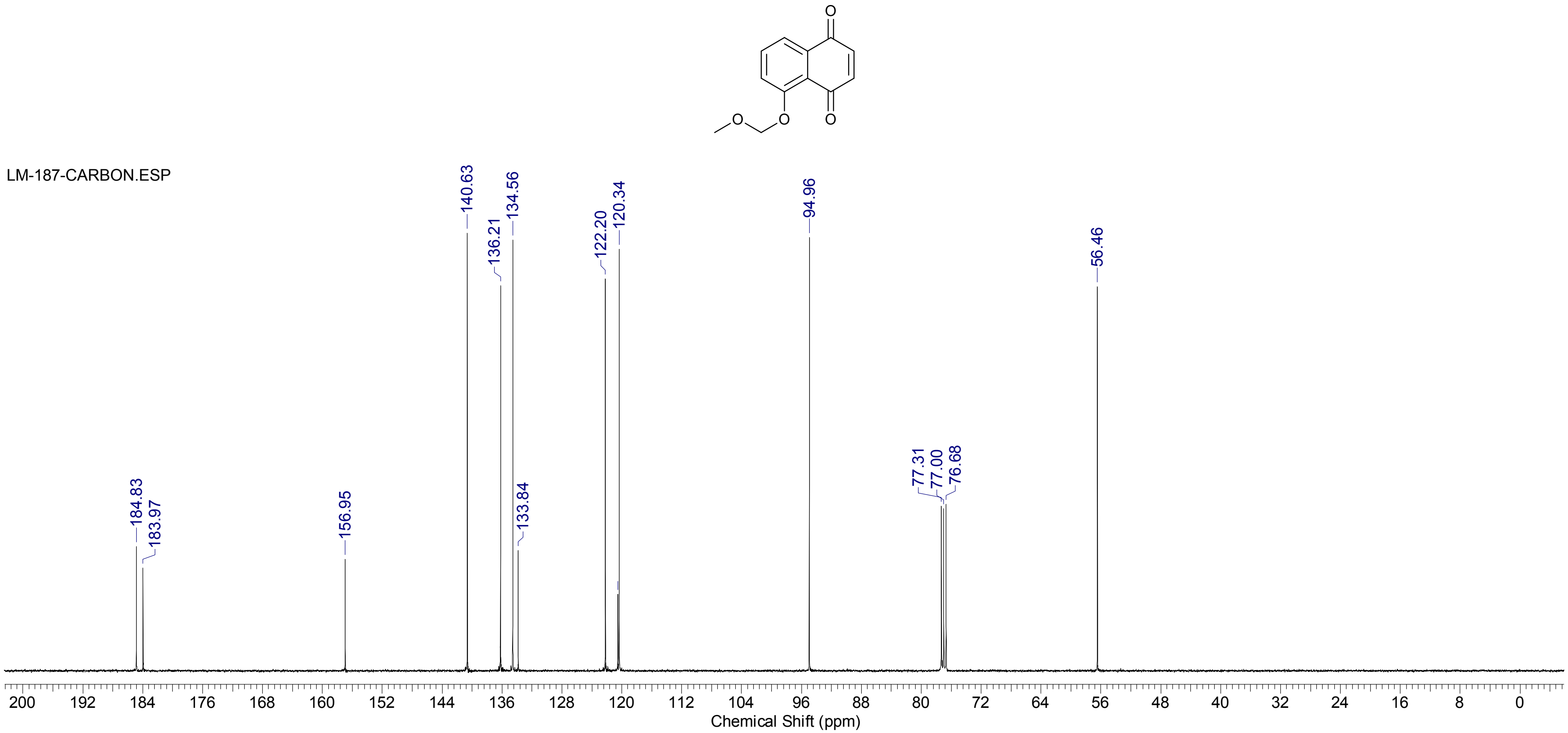
5-Methoxymethoxy-1,4-naphthoquinone 8e





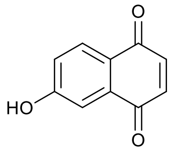
^{1}H NMR at 400 MHz in $CDCl_3$

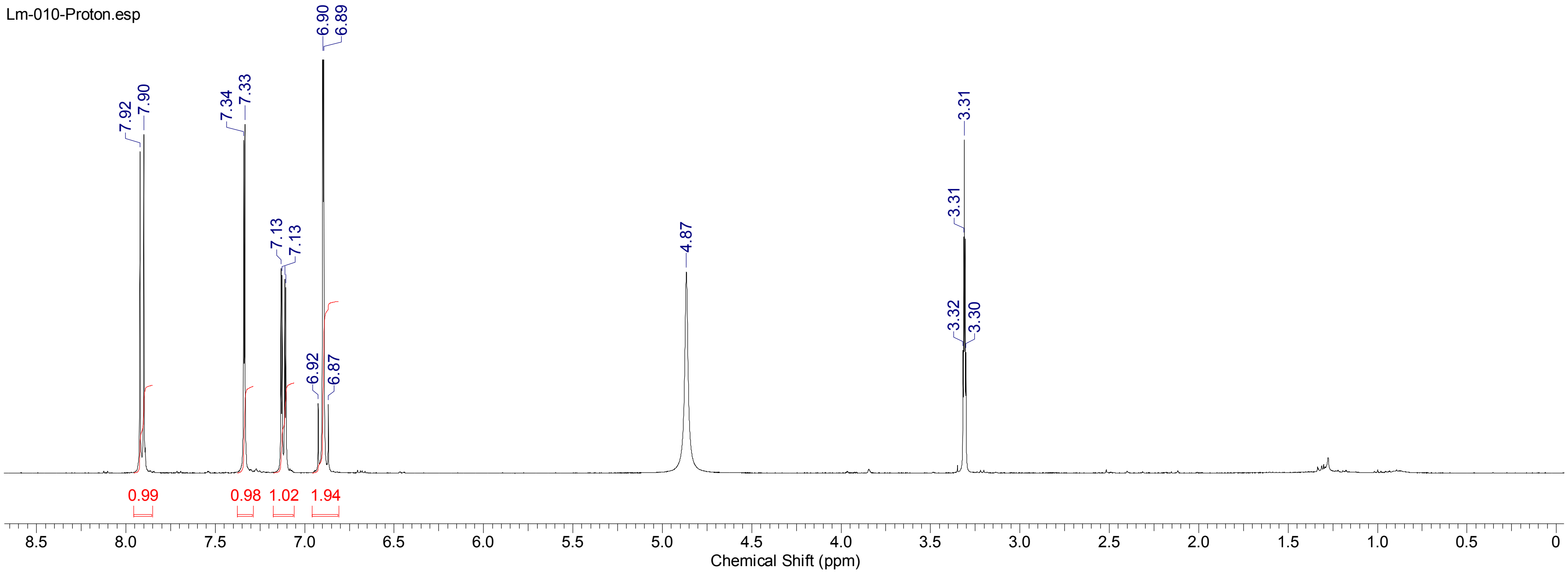
5-Methoxymethoxy-1,4-naphthoquinone 8e



^{13}C NMR at 100 MHz in $CDCl_3$

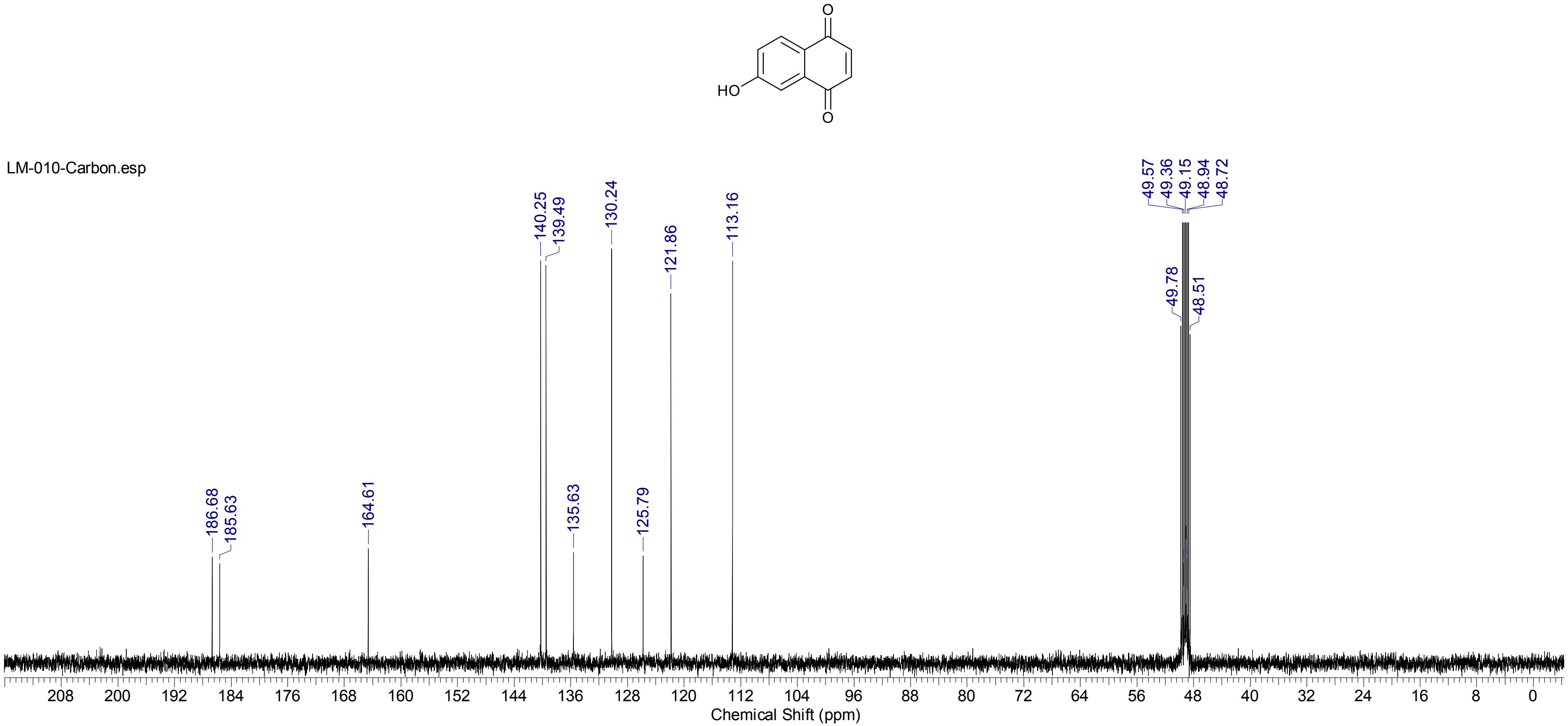
6-Hydroxy-1,4-naphthoquinone 12a





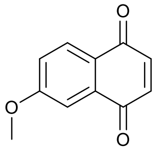
^{1}H NMR at 400 MHz in MeOD

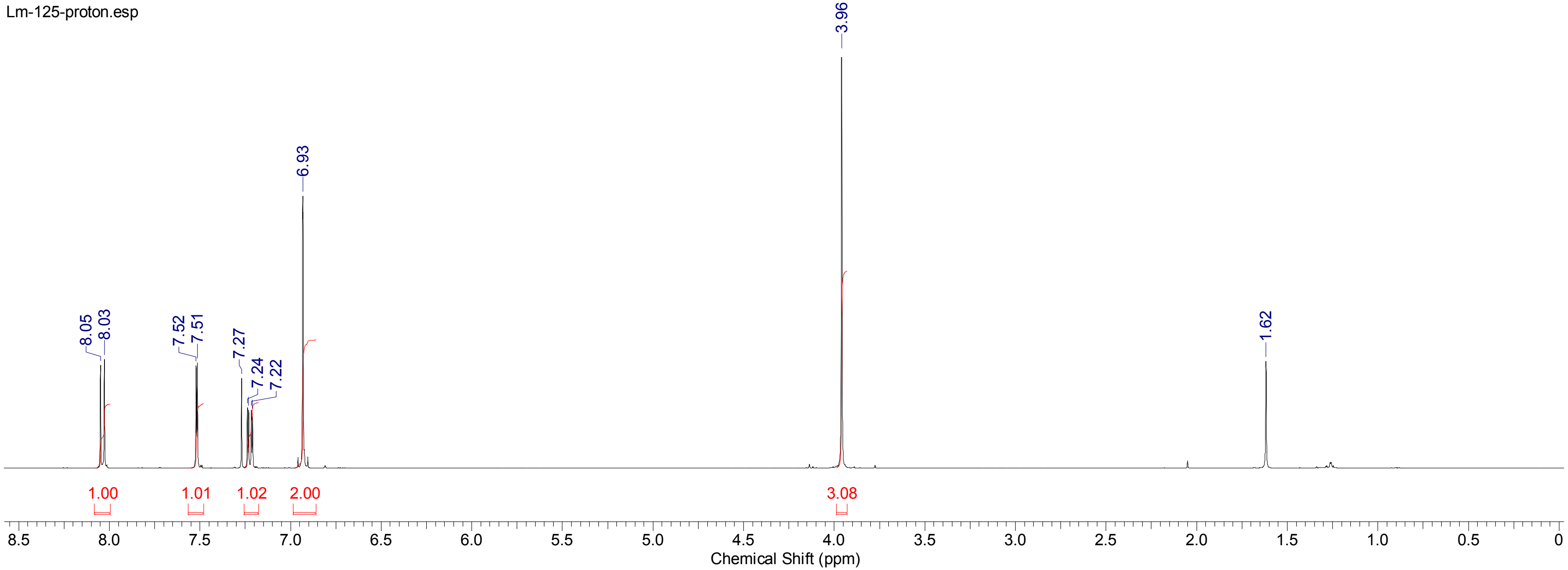
6-Hydroxy-1,4-naphthoquinone 12a



^{13}C NMR at 100 MHz in MeOD

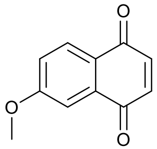
6-Methoxy-1,4-naphthoquinone 12b

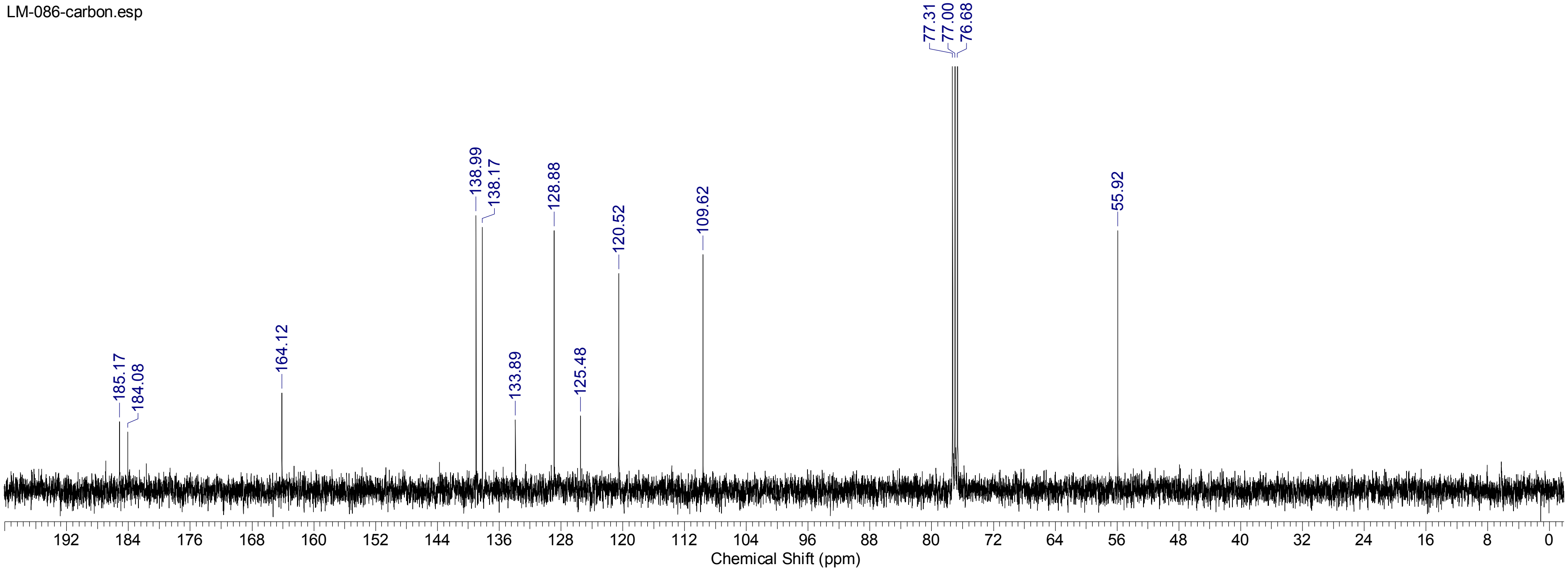




^{1}H NMR at 400 MHz in CDCl$_3$

6-Methoxy-1,4-naphthoquinone 12b





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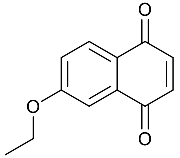
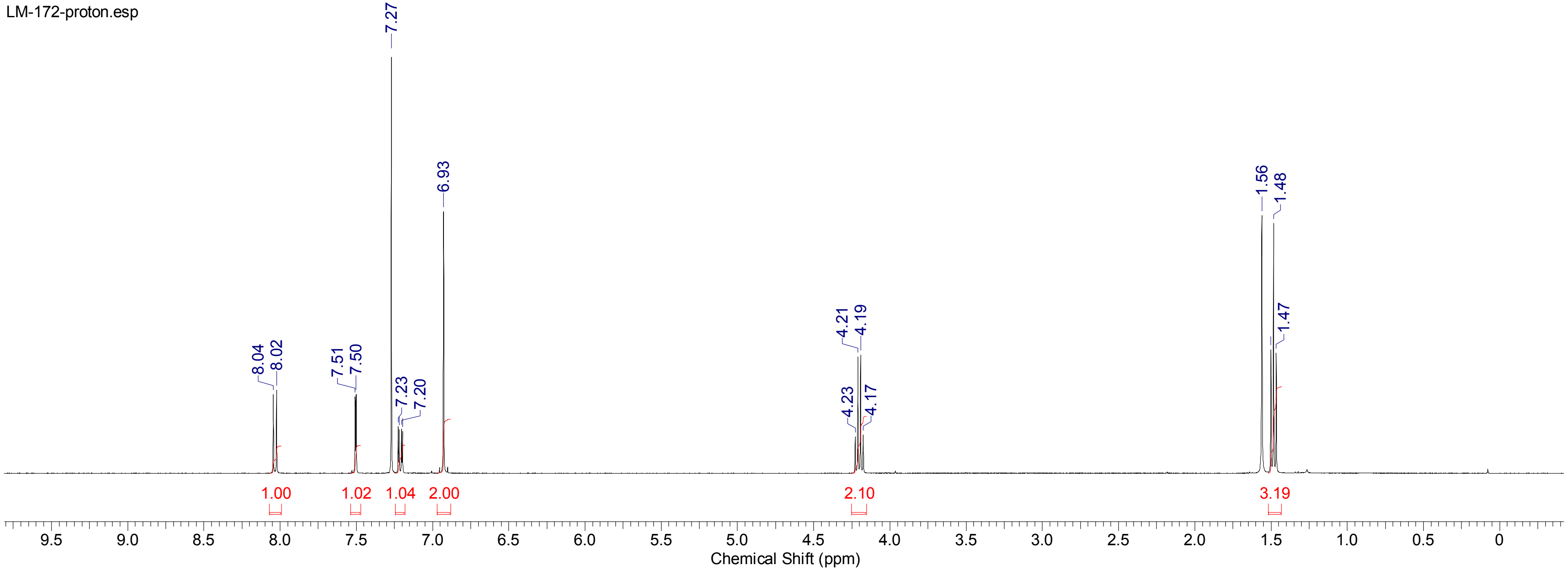
6-Ethoxy-1,4-naphthoquinone 12c





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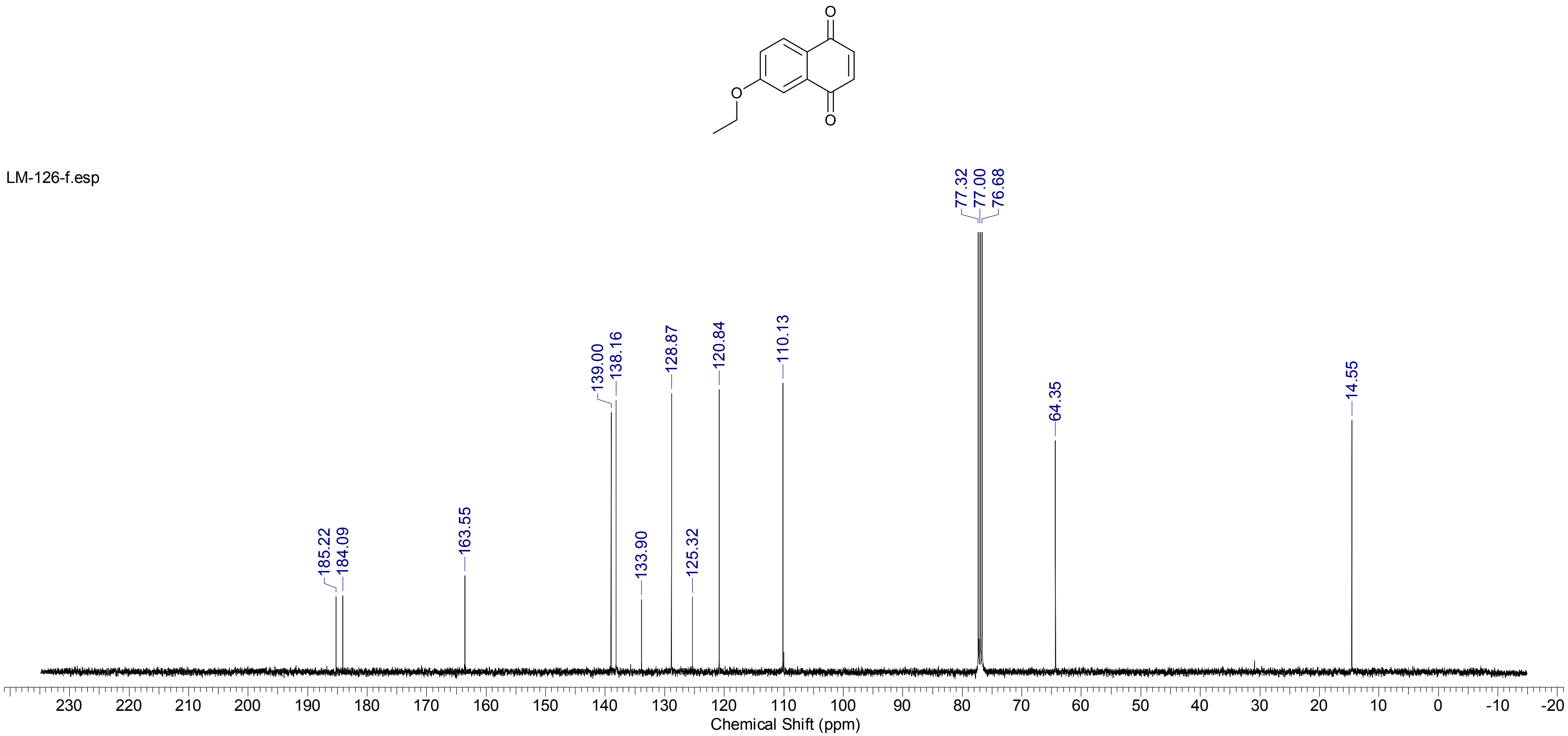
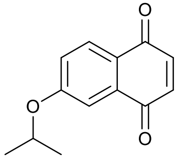
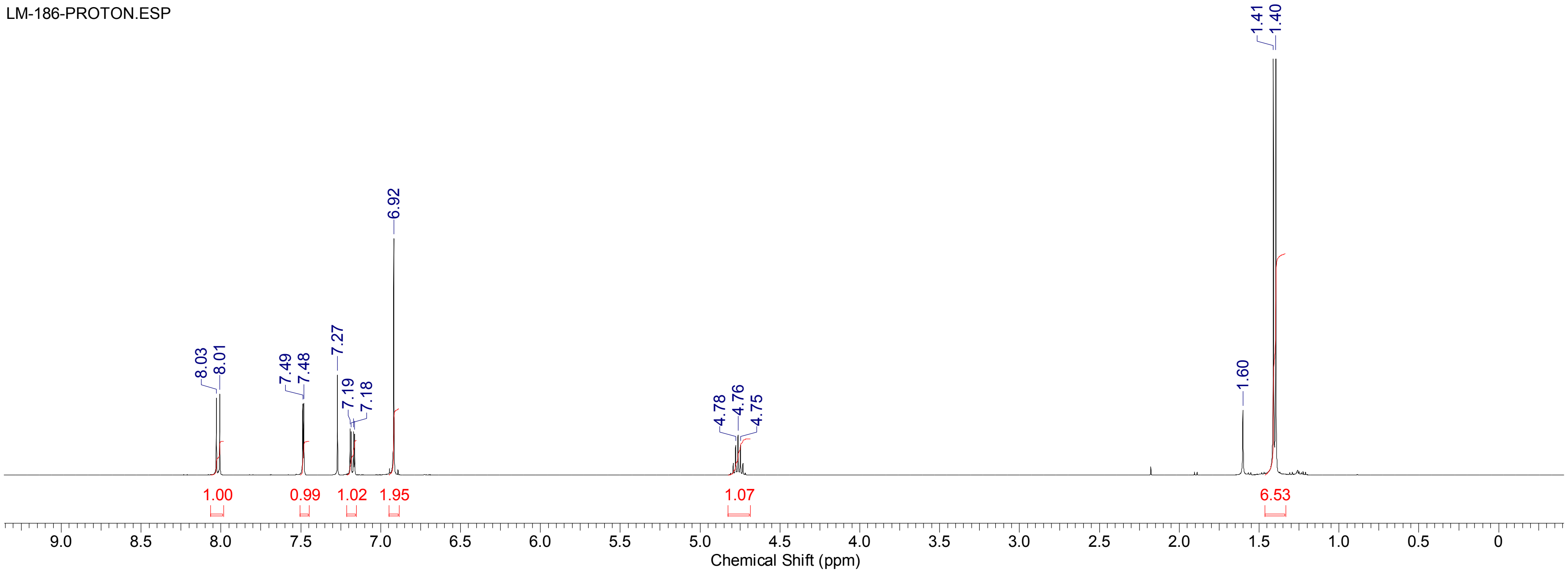
6-Ethoxy-1,4-naphthoquinone 12c



^{13}C NMR at 100 MHz in $CDCl_3$

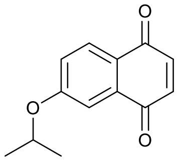
6-Isopropoxy-1,4-naphthoquinone 12d

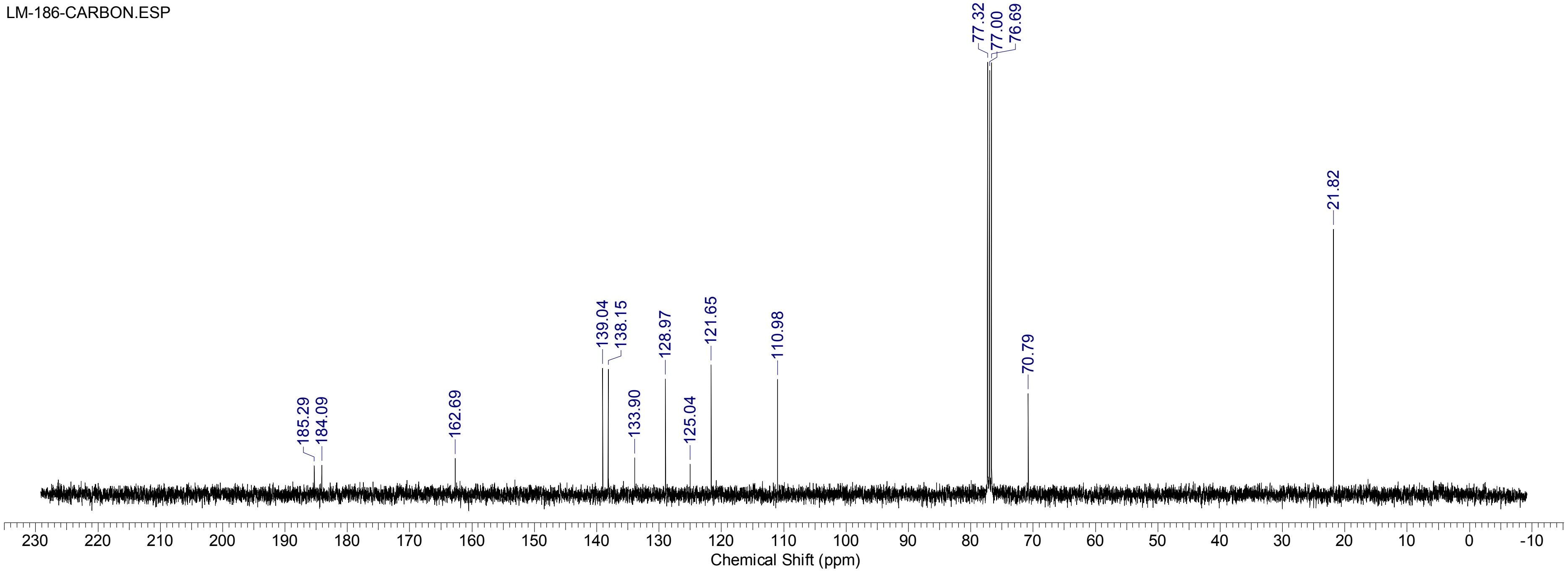




^{1}H NMR at 400 MHz in $CDCl_3$

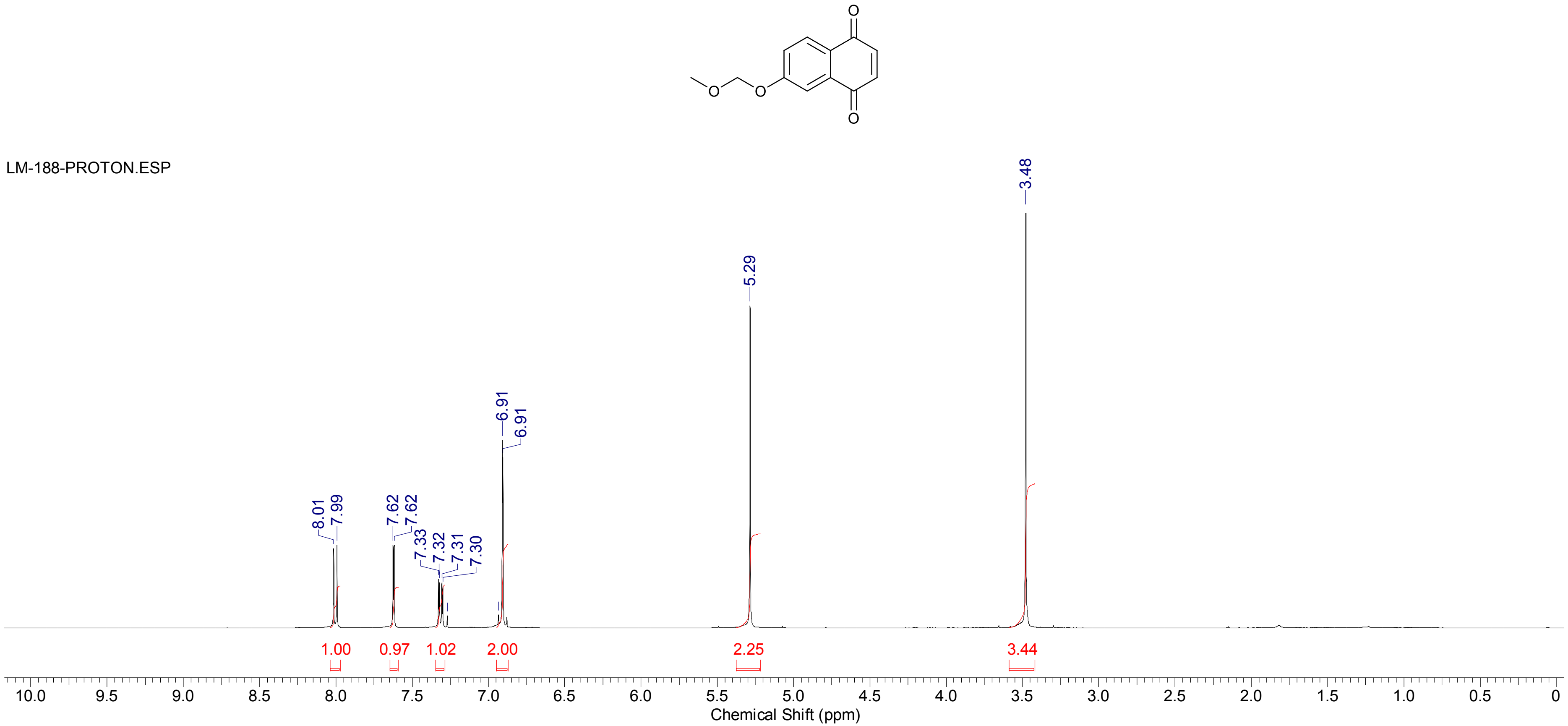
6-Isopropoxy-1,4-naphthoquinone 12d





^{13}C NMR at 100 MHz in $CDCl_3$

6-(Methoxymethoxy)-1,4-naphthoquinone 12e



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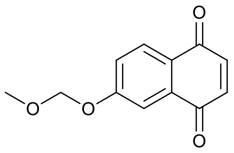
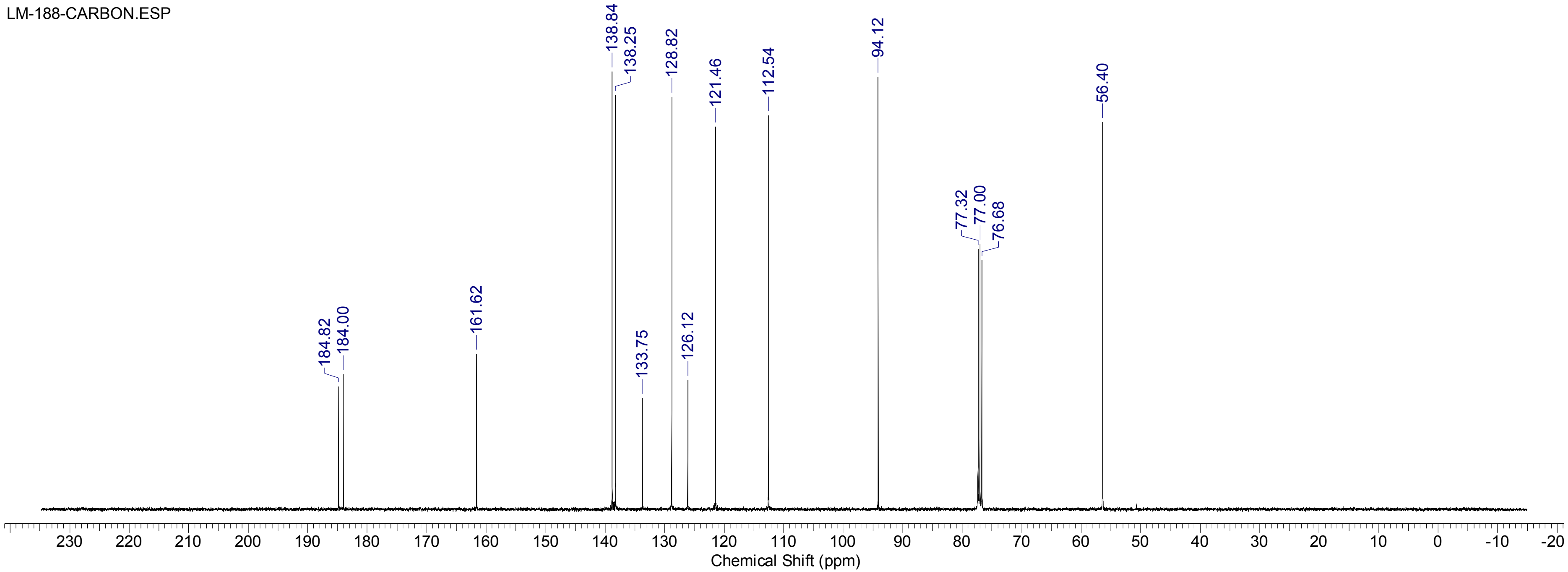
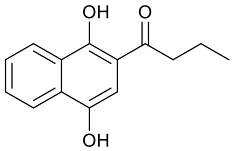
6-(Methoxymethoxy)-1,4-naphthoquinone 12e

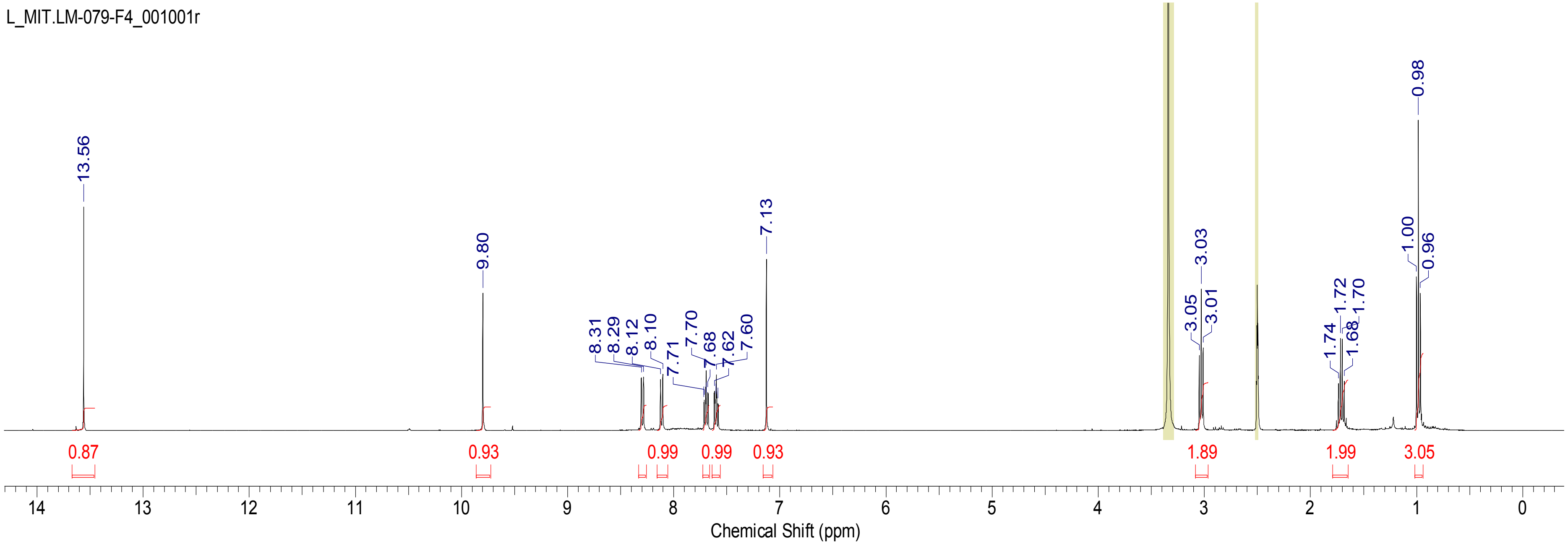




^{13}C NMR at 100 MHz in $CDCl_3$

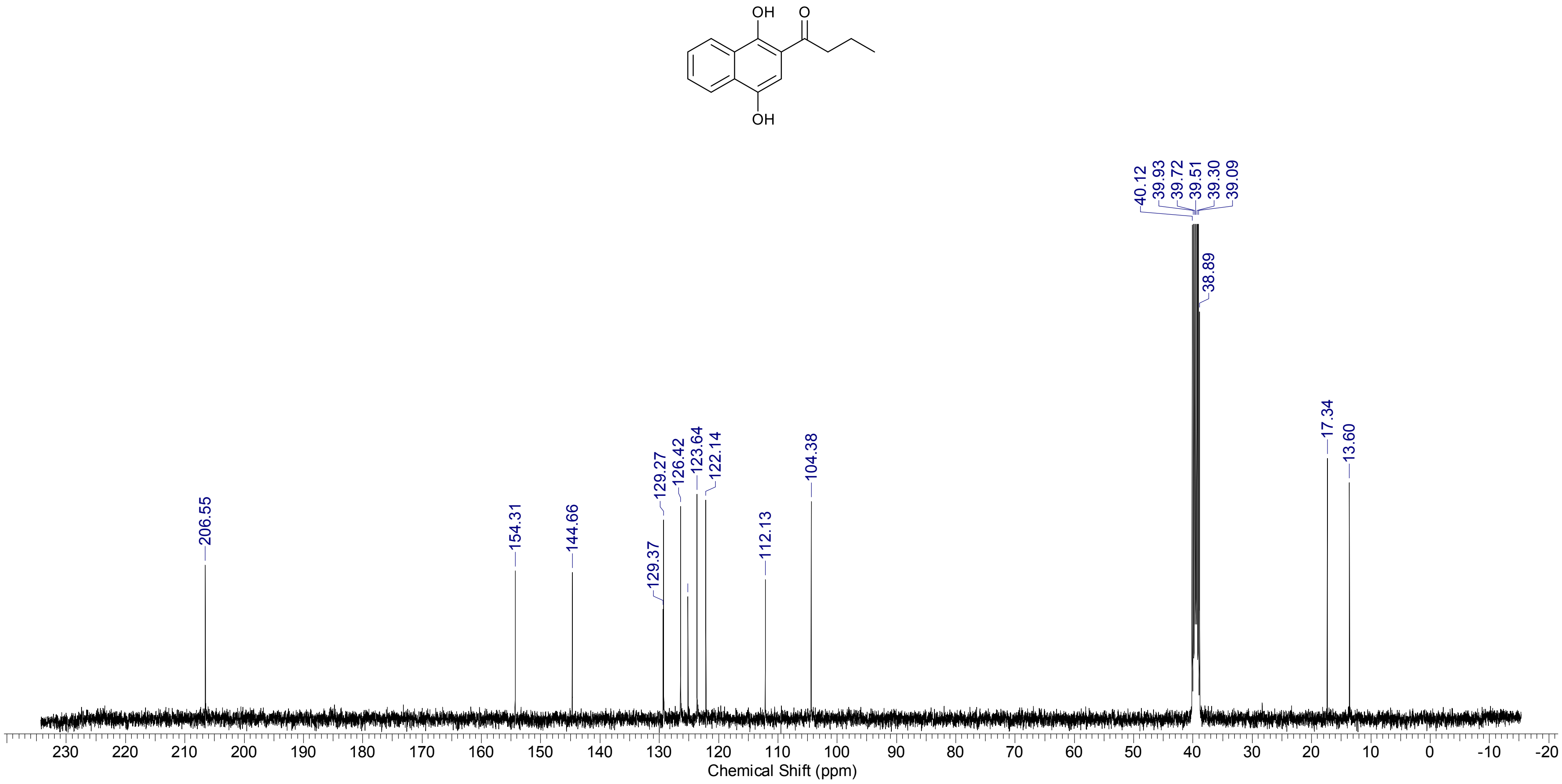
1-(1,4-Dihydroxy-2-naphthyl)-1-butanone 6a





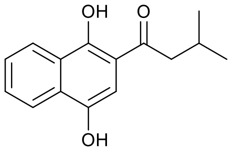
^{1}H NMR at 400 MHz in d_6-DMSO

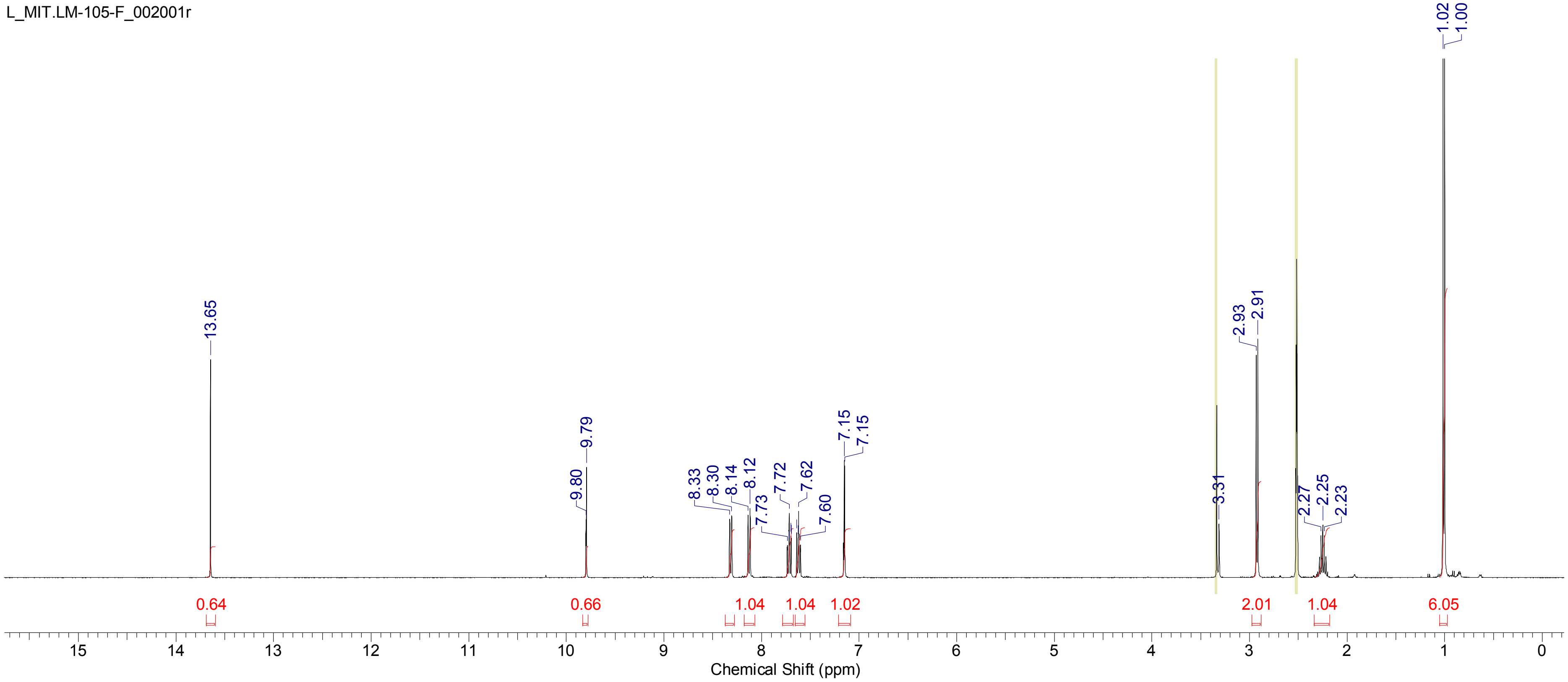
1-(1,4-Dihydroxy-2-naphthyl)-1-butanone 6a



^{13}C NMR at 100 MHz in d_6-DMSO

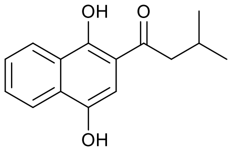
1-(1,4-Dihydroxy-2-naphthyl)-2-methyl-1-propanone 6b

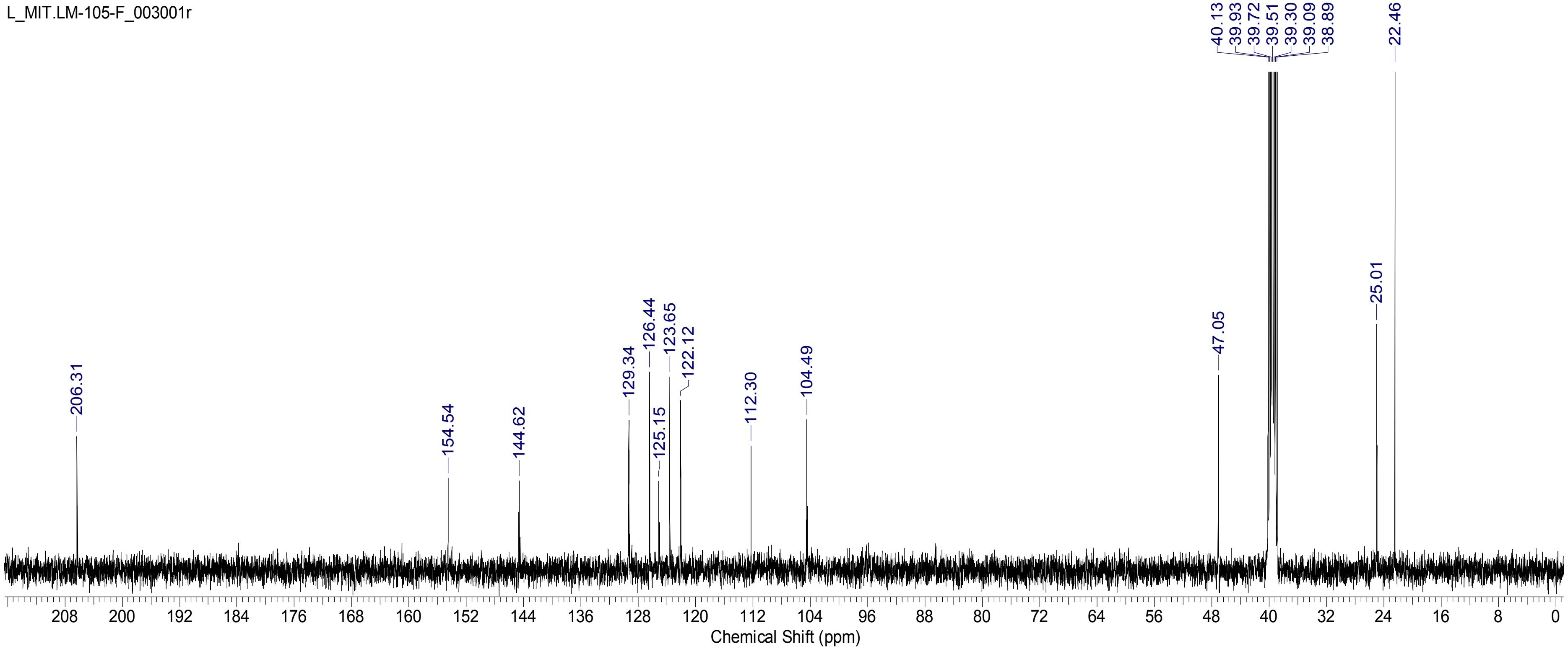




^{1}H NMR at 400 MHz in d$_6$-DMSO

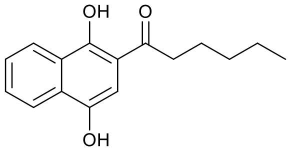
1-(1,4-Dihydroxy-2-naphthyl)-2-methyl-1-propanone 6b

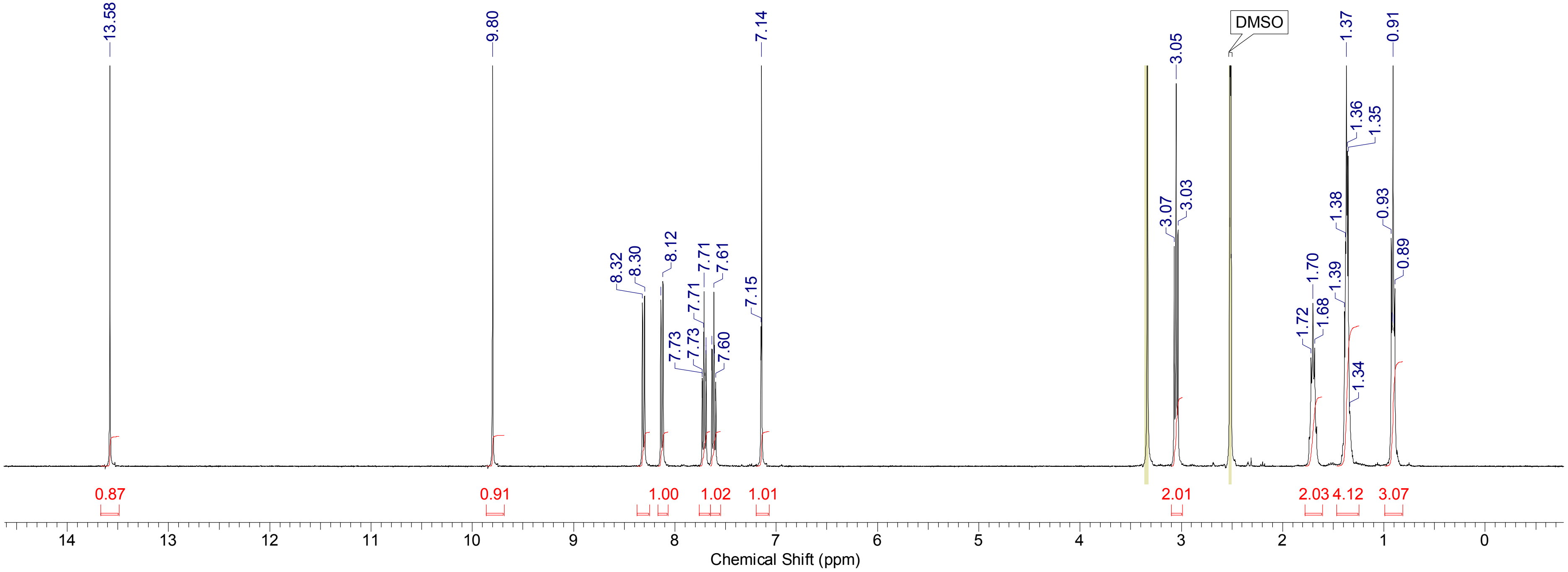




^{13}C NMR at 100 MHz in d$_6$-DMSO

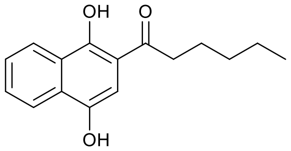
1-(1,4-Dihydroxy-2-naphthyl)-1-hexanone 6c

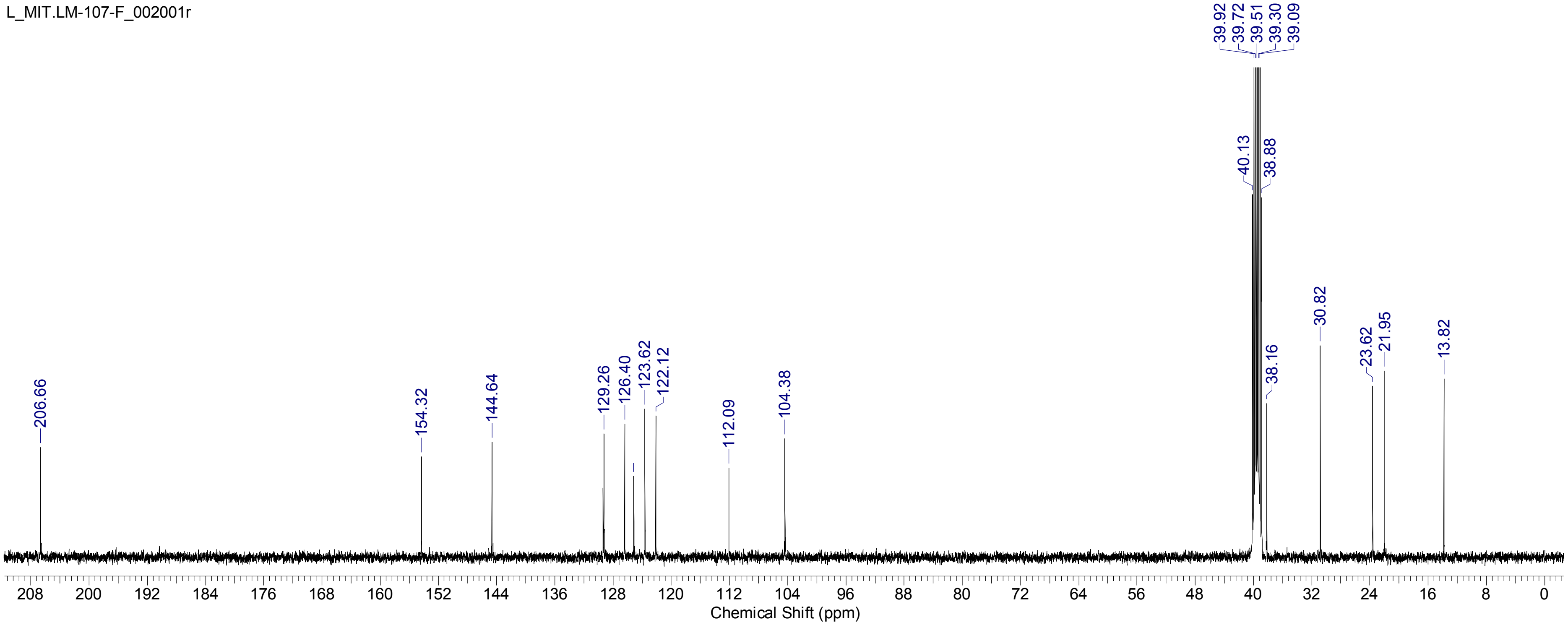




^{1}H NMR at 400 MHz in d$_6$-DMSO

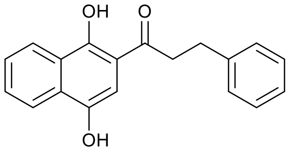
1-(1,4-Dihydroxy-2-naphthyl)-1-hexanone 6c

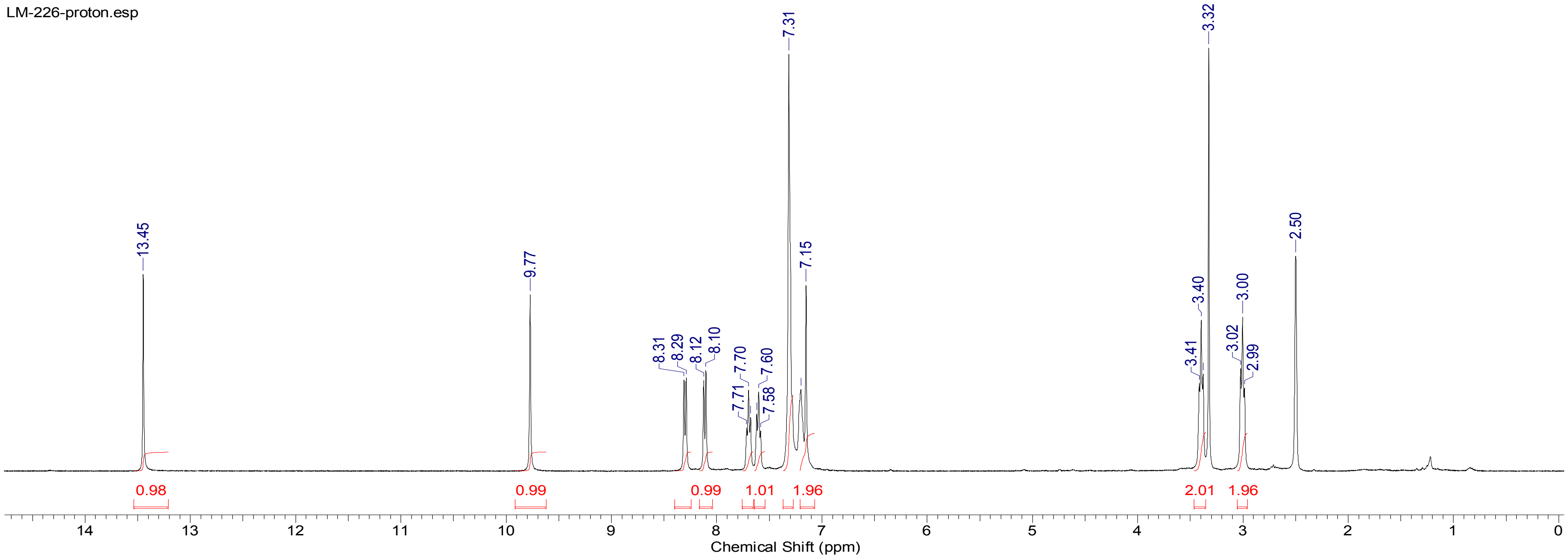




^{13}C NMR at 100 MHz in d_6-DMSO

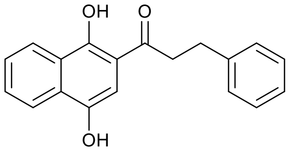
1-(1,4-dihydroxynaphthalen-2-yl)-3-phenylpropan-1-one 6d

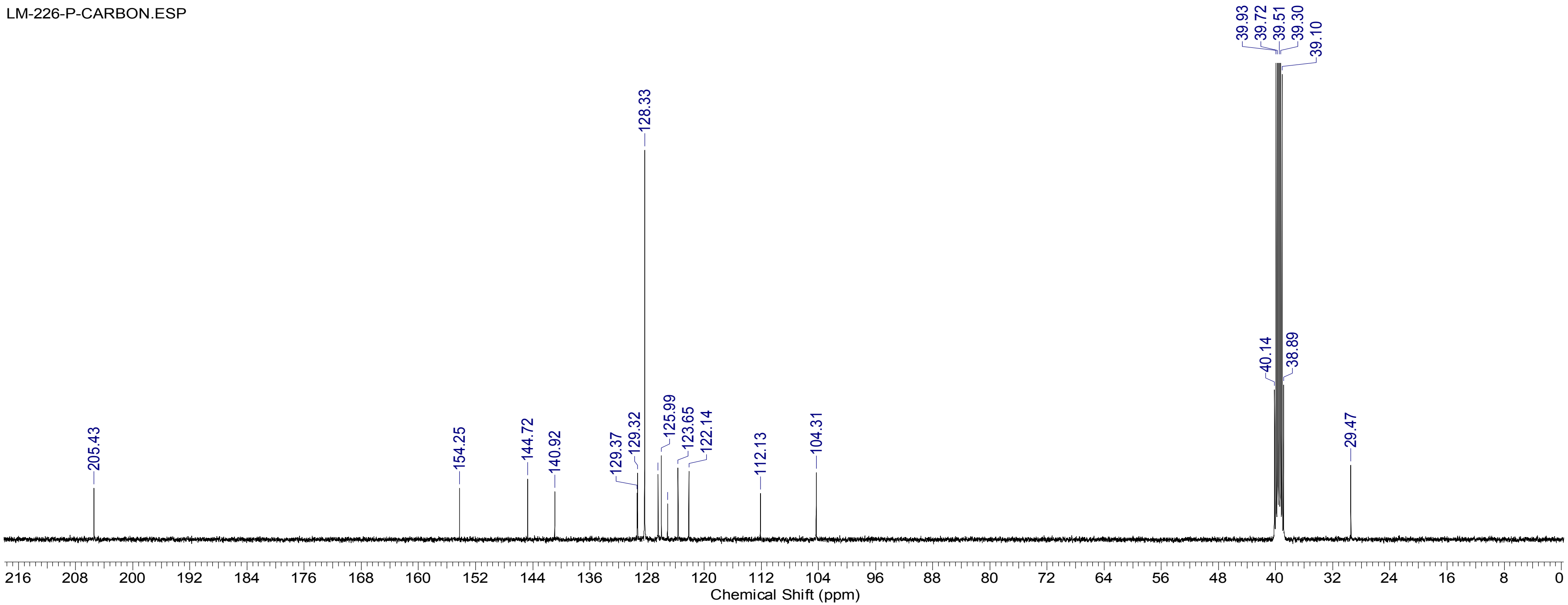




^{1}H NMR at 400 MHz in d$_6$-DMSO

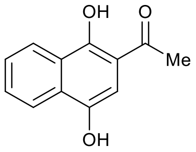
1-(1,4-dihydroxynaphthalen-2-yl)-3-phenylpropan-1-one 6d

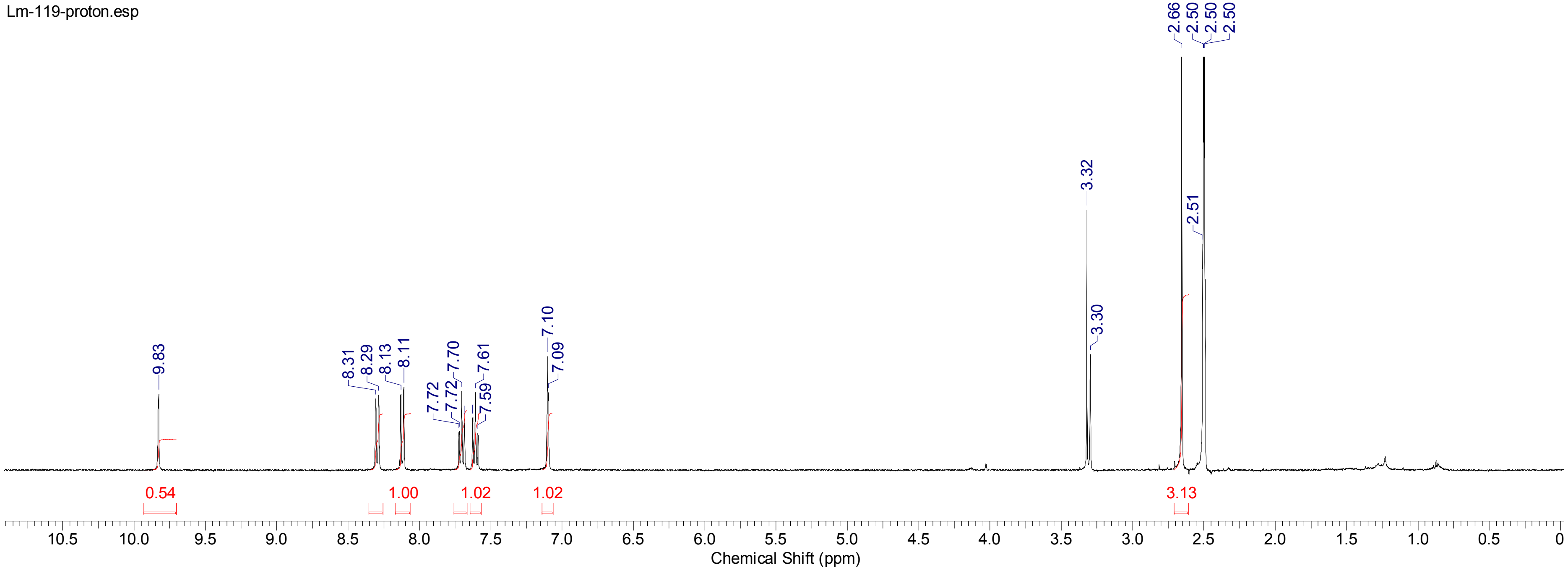




^{13}C NMR at 100 MHz in d_6-DMSO

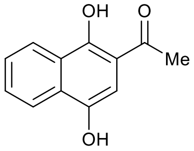
1-(1,4-Dihydroxy-2-naphthyl)-1-ethanone 6e

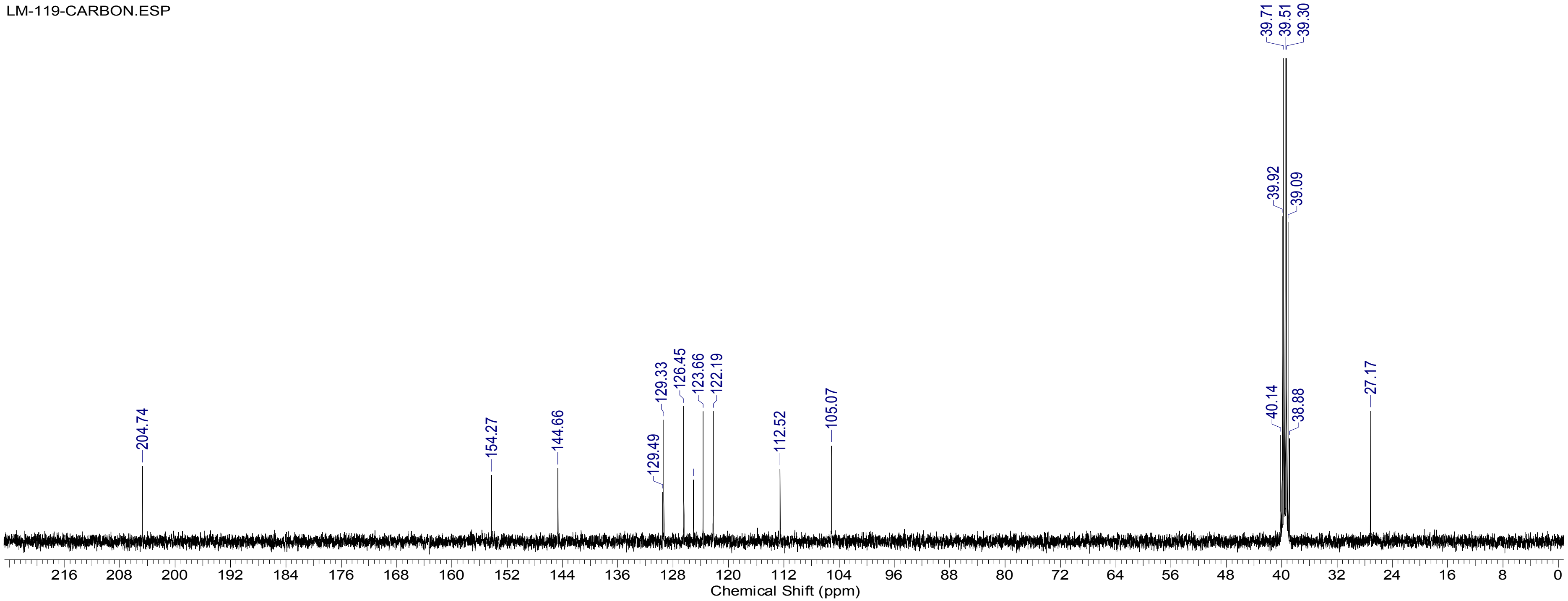




^{1}H NMR at 400 MHz in d$_6$-DMSO

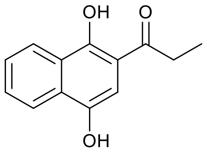
1-(1,4-Dihydroxy-2-naphthyl)-1-ethanone 6e

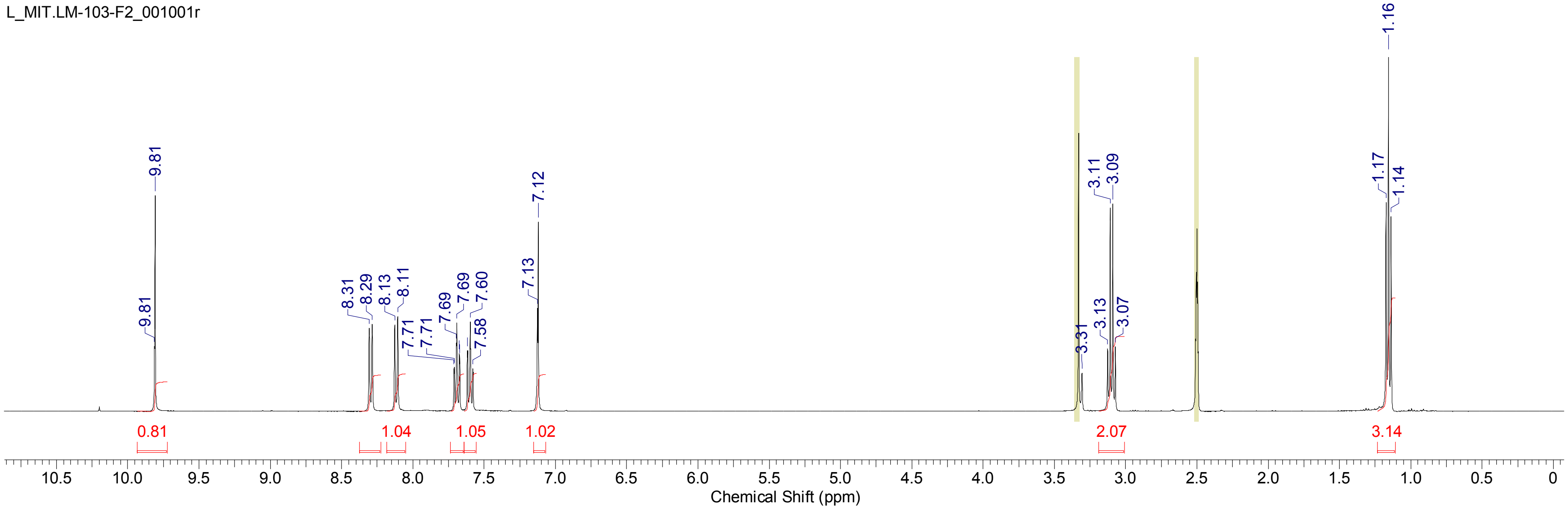




^{13}C NMR at 100 MHz in d_6-DMSO

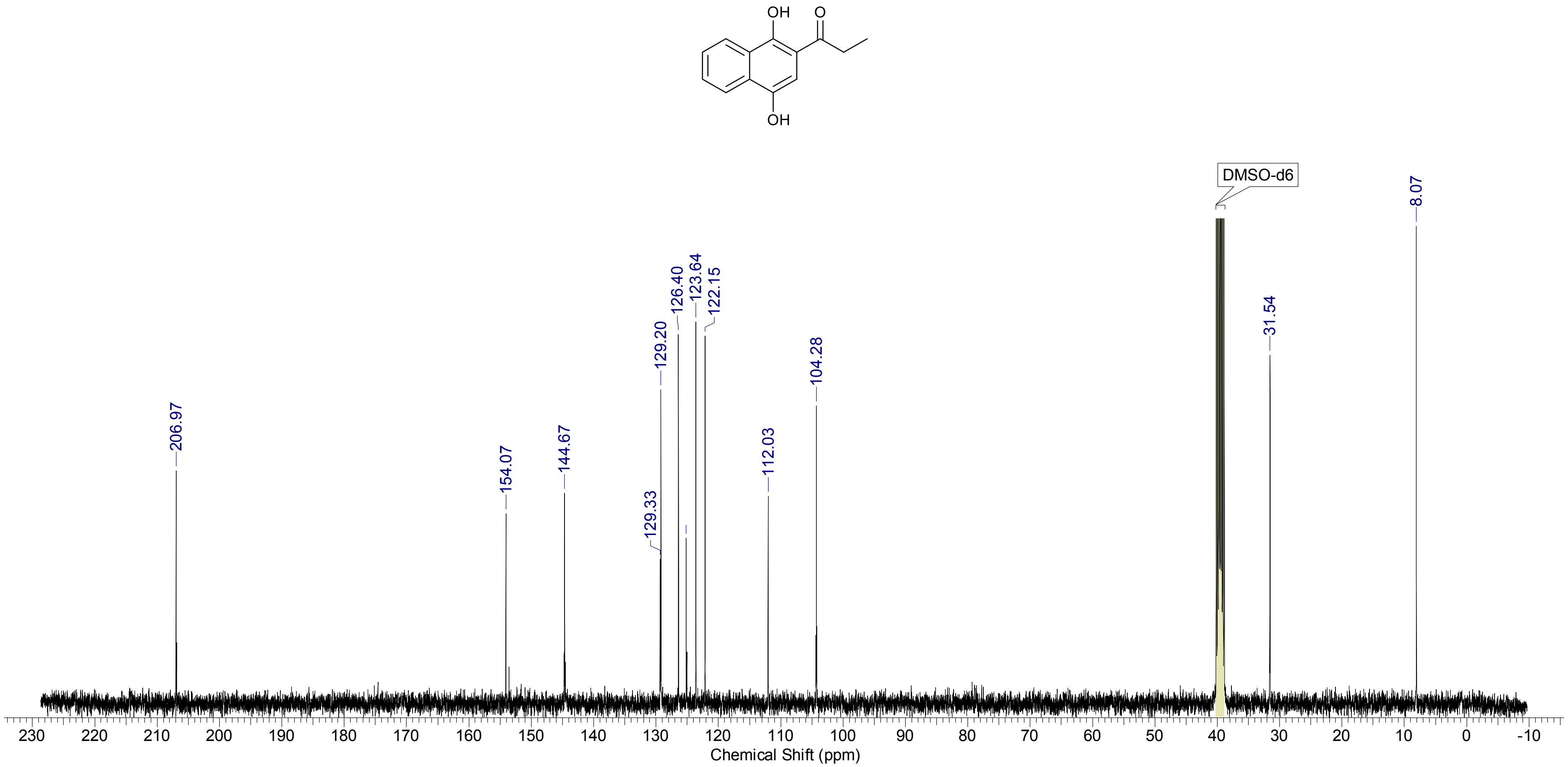
1-(1,4-Dihydroxy-2-naphthyl)-1-propanone 6f





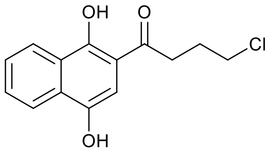
^{1}H NMR at 400 MHz in d$_6$-DMSO

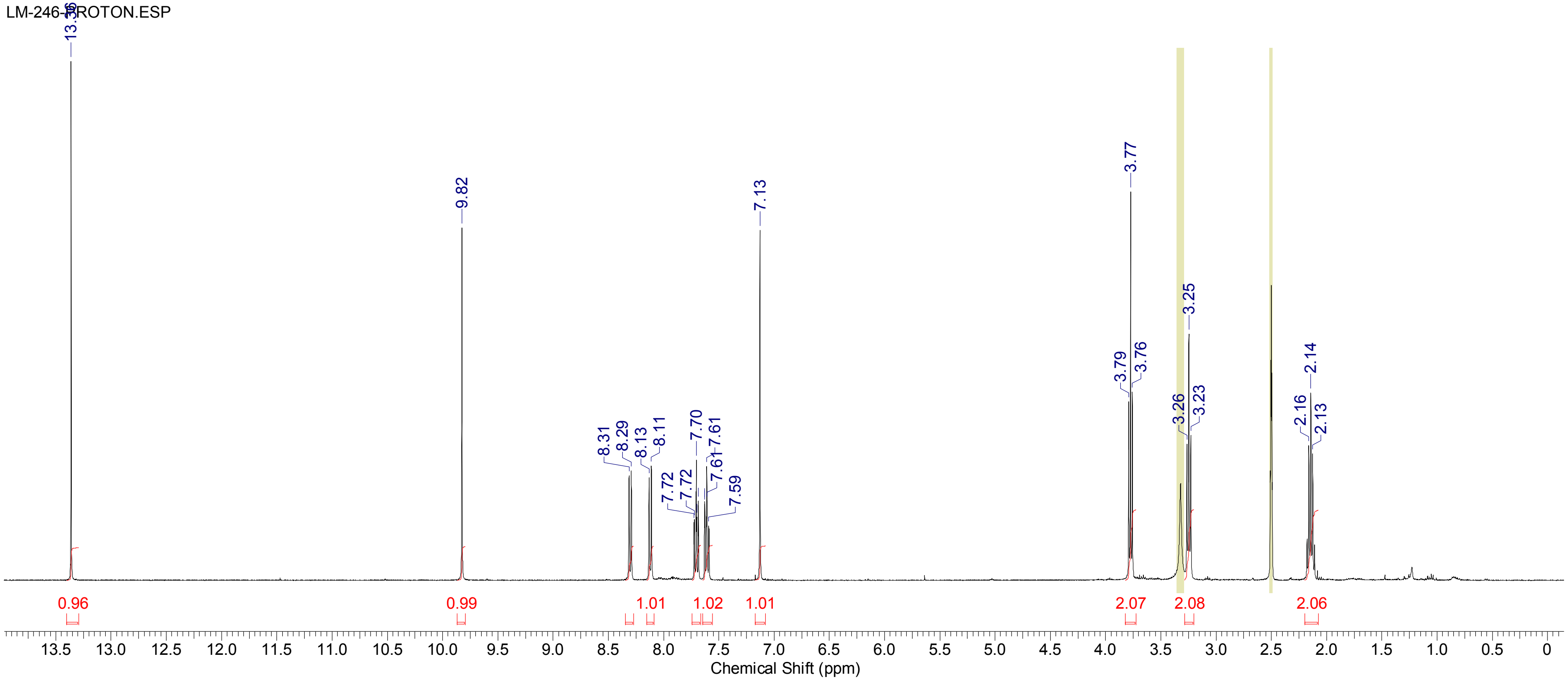
1-(1,4-Dihydroxy-2-naphthyl)-1-propanone 6f



^{13}C NMR at 100 MHz in d$_6$-DMSO

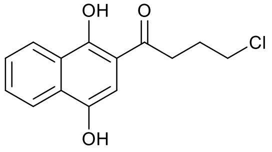
4-Chloro-1-(1,4-dihydroxynaphthalen-2-yl)butan-1-one 6g

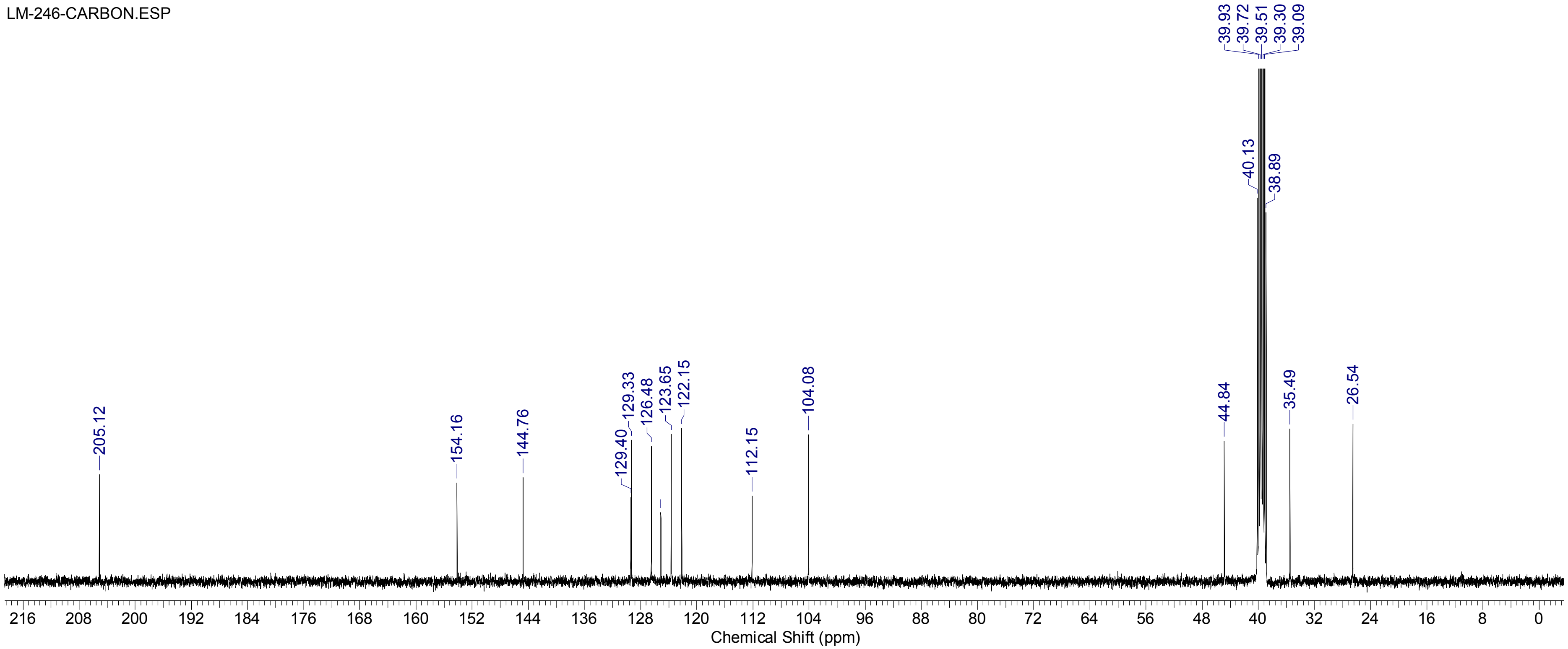




^{1}H NMR at 400 MHz in d$_6$-DMSO

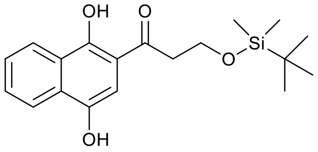
4-Chloro-1-(1,4-dihydroxynaphthalen-2-yl)butan-1-one 6g

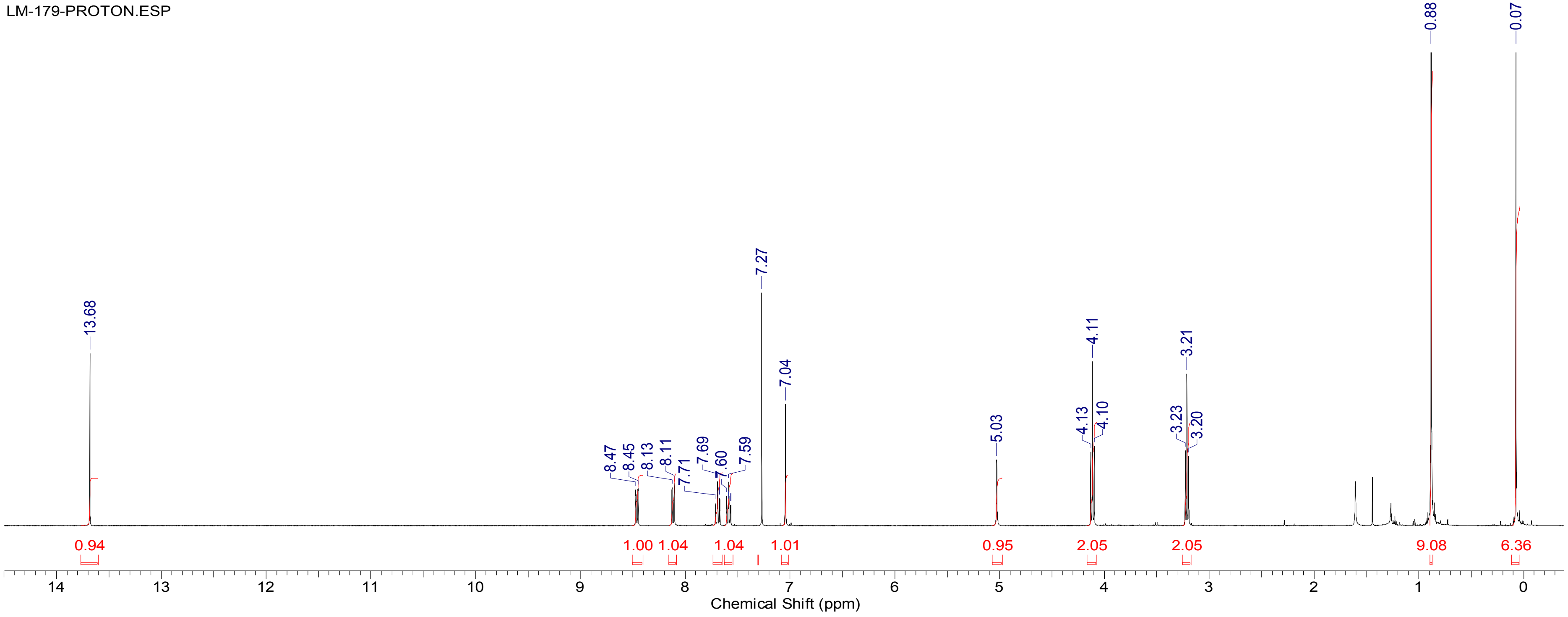




^{13}C NMR at 100 MHz in d$_6$-DMSO

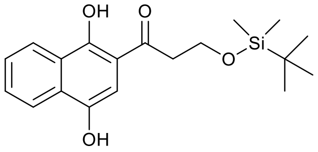
3-((*t*-Butyldimethylsilyl)oxy)-1-(1,4-dihydroxynaphthalen-2-yl)1-propanone 6h

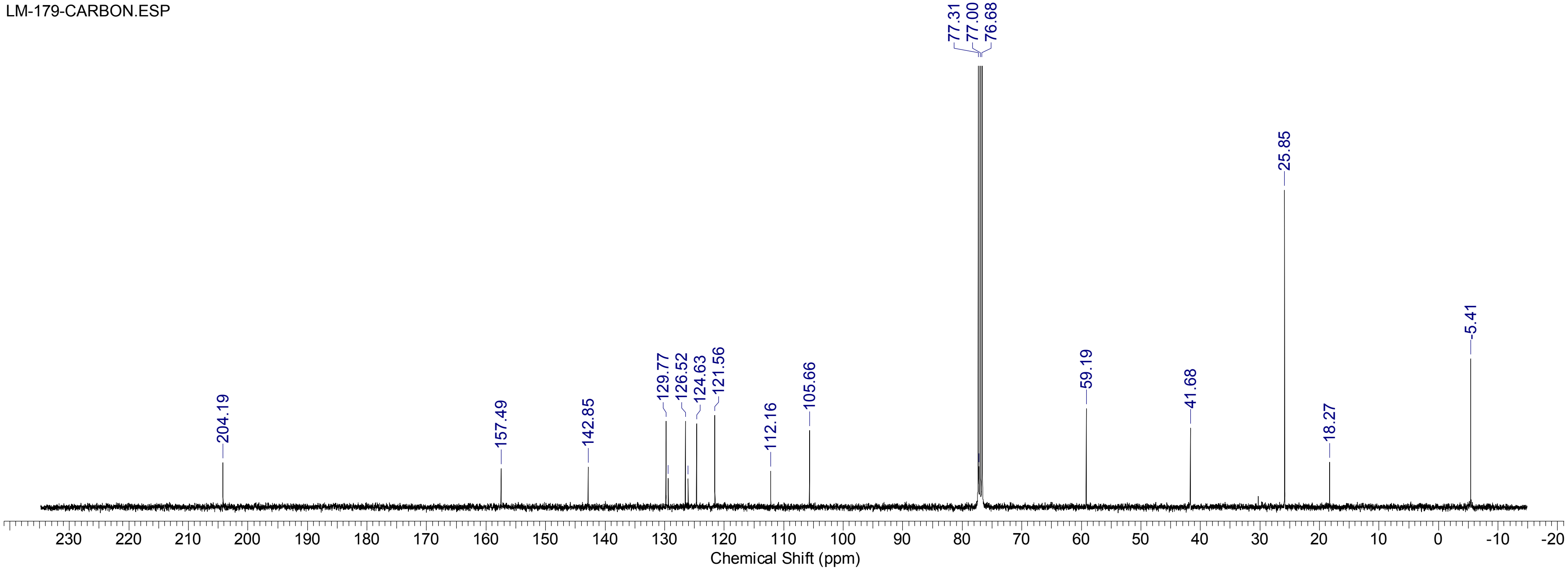




^{1}H NMR at 400 MHz in $CDCl_3$

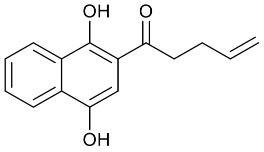
3-((*t*-Butyldimethylsilyl)oxy)-1-(1,4-dihydroxynaphthalen-2-yl)1-propanone 6h

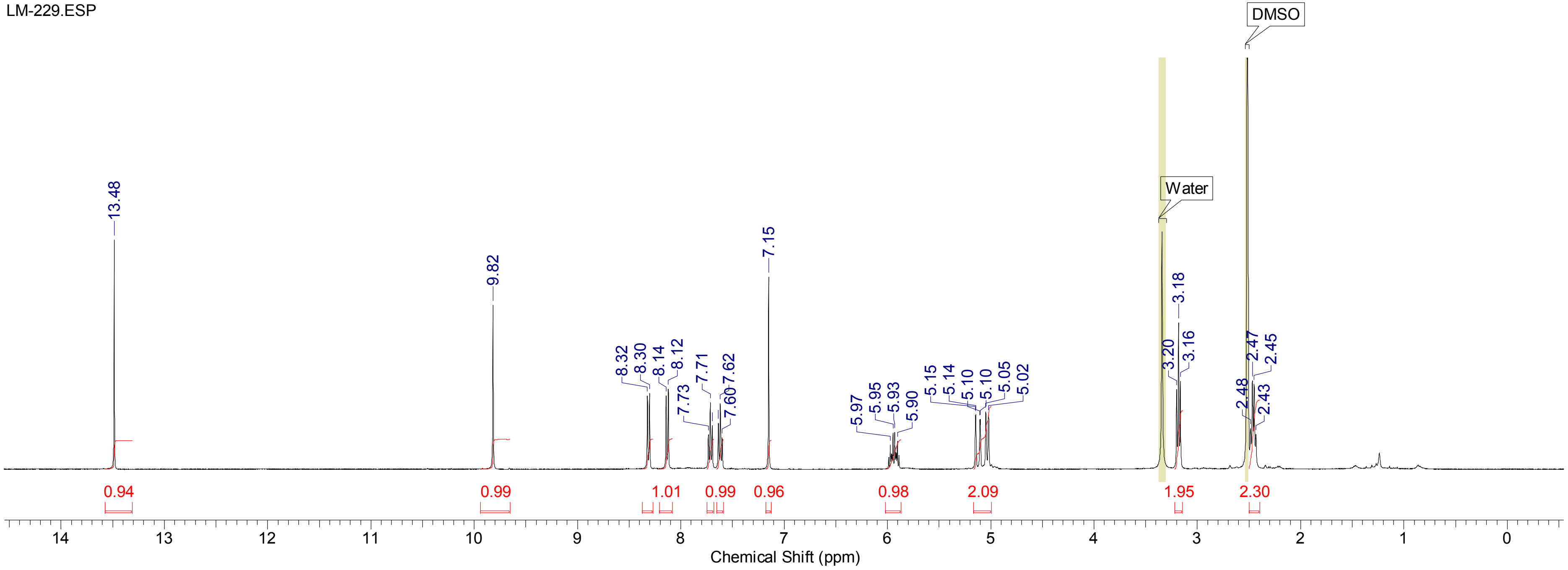




^{13}C NMR at 100 MHz in $CDCl_3$

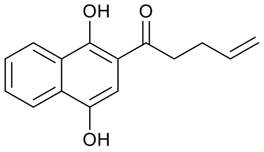
1-(1,4-Dihydroxynaphthalen-2-yl)pent-4-en-1-one 6i

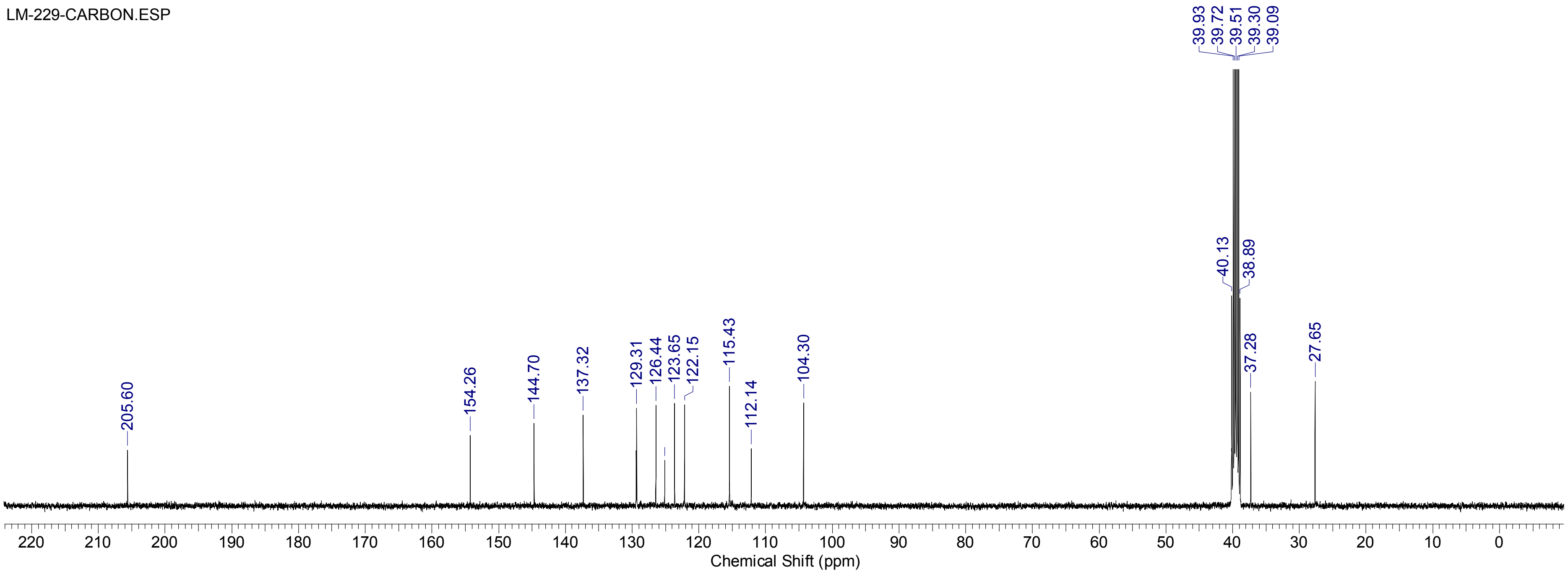




^{1}H NMR at 400 MHz in d$_6$-DMSO

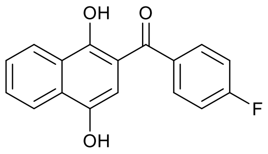
1-(1,4-Dihydroxynaphthalen-2-yl)pent-4-en-1-one 6i

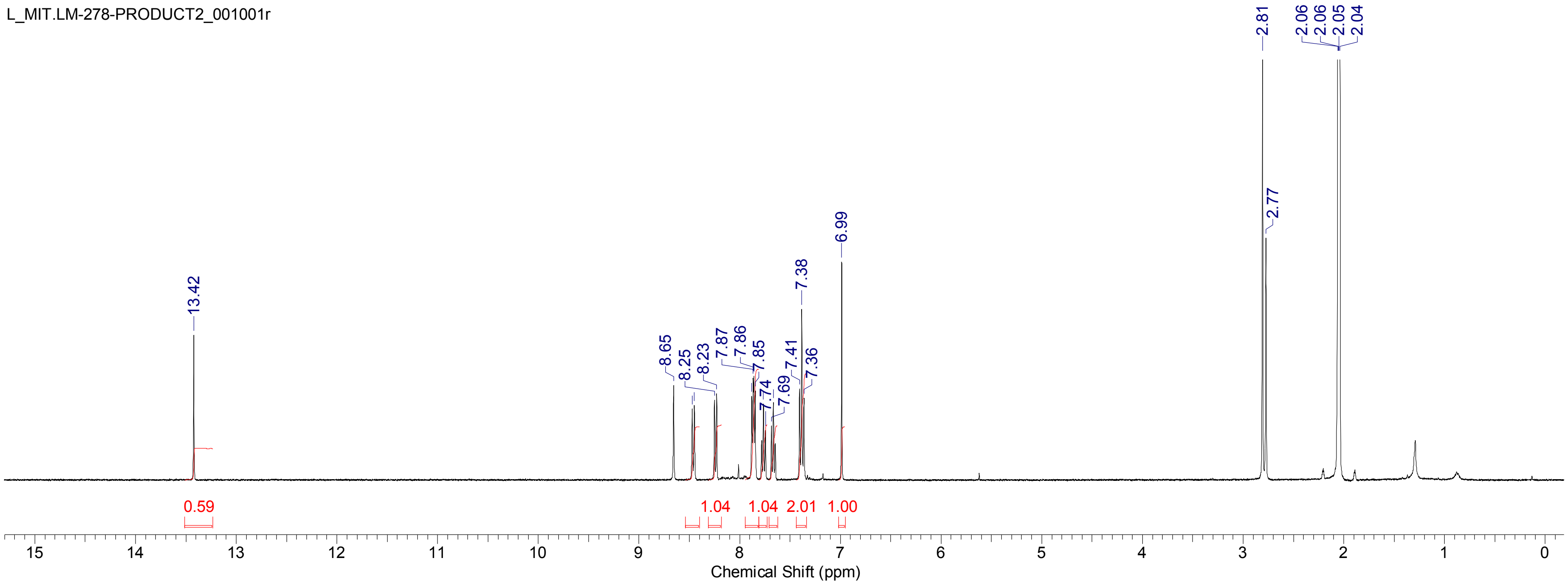




^{13}C NMR at 100 MHz in d$_6$-DMSO

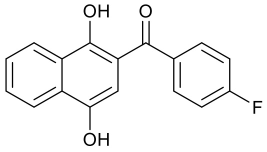
(1,4-dihydroxynaphthalen-2-yl)(4-fluorophenyl)methanone 6j

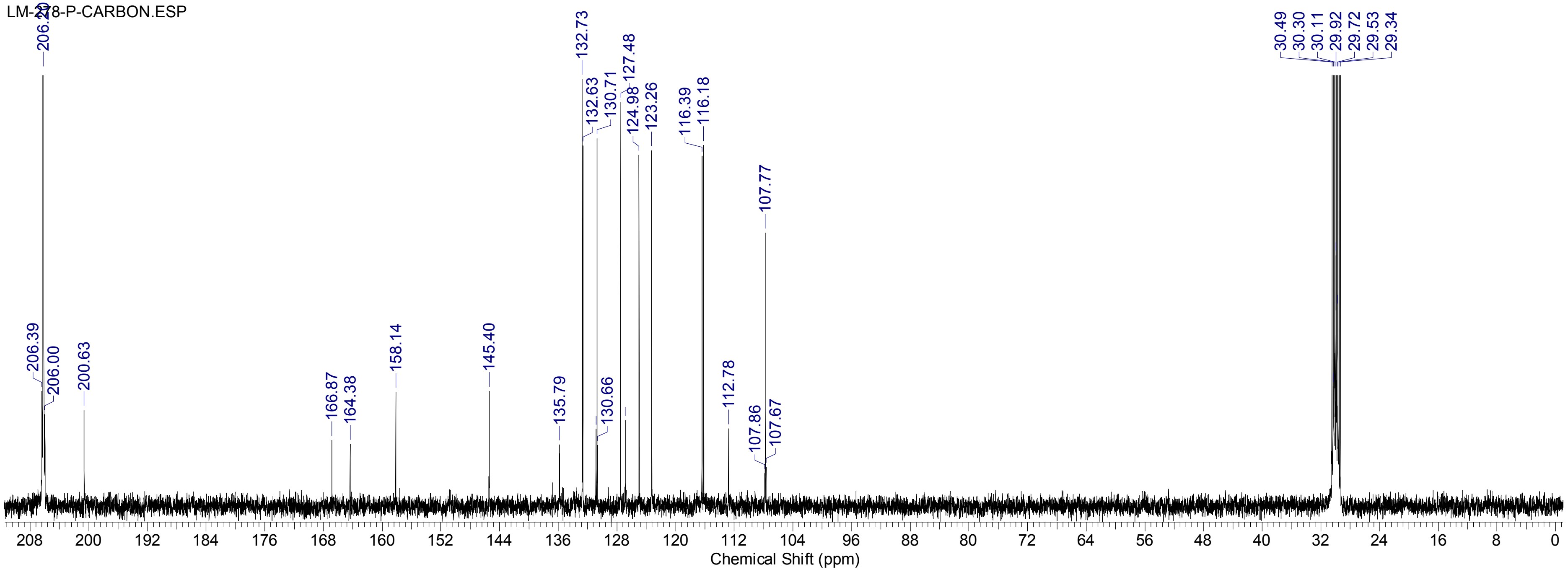




^{1}H NMR at 400 MHz in d$_6$-acetone

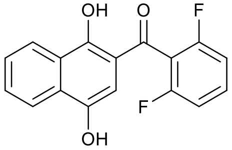
(1,4-dihydroxynaphthalen-2-yl)(4-fluorophenyl)methanone 6j

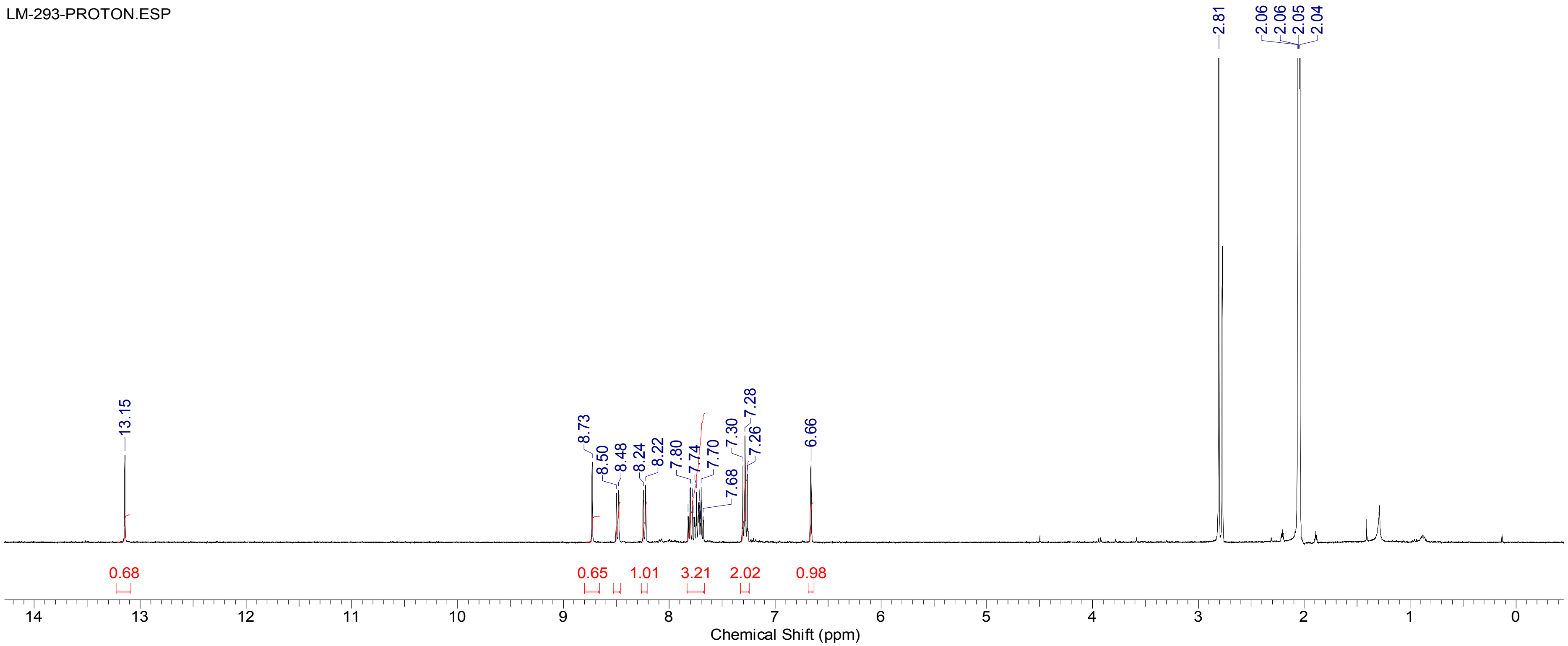




^{13}C NMR at 100 MHz in d_6-acetone

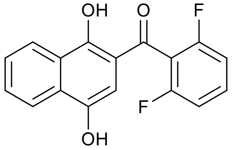
(2,6-difluorophenyl)(1,4-dihydroxynaphthalen-2-yl)methanone 6k

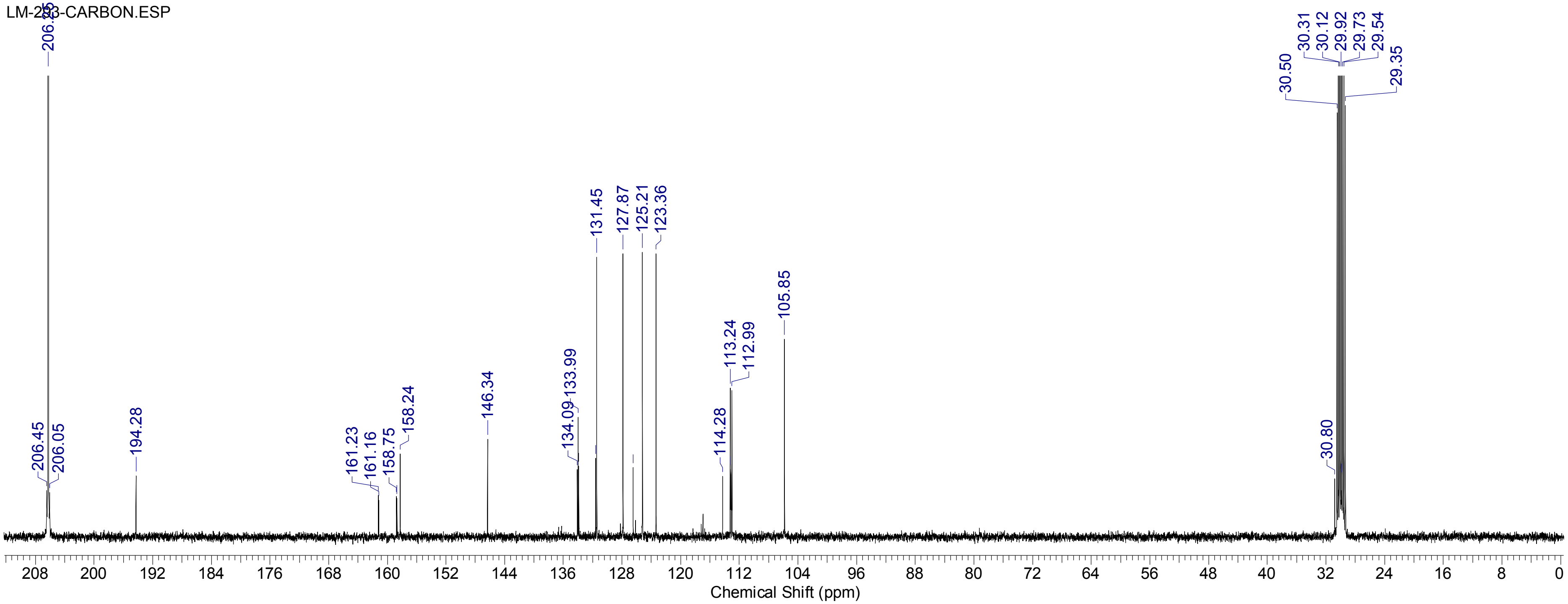




^{1}H NMR at 400 MHz in d$_6$-acetone

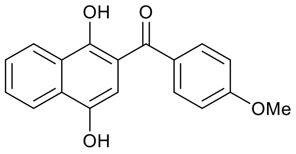
(2,6-difluorophenyl)(1,4-dihydroxynaphthalen-2-yl)methanone 6k

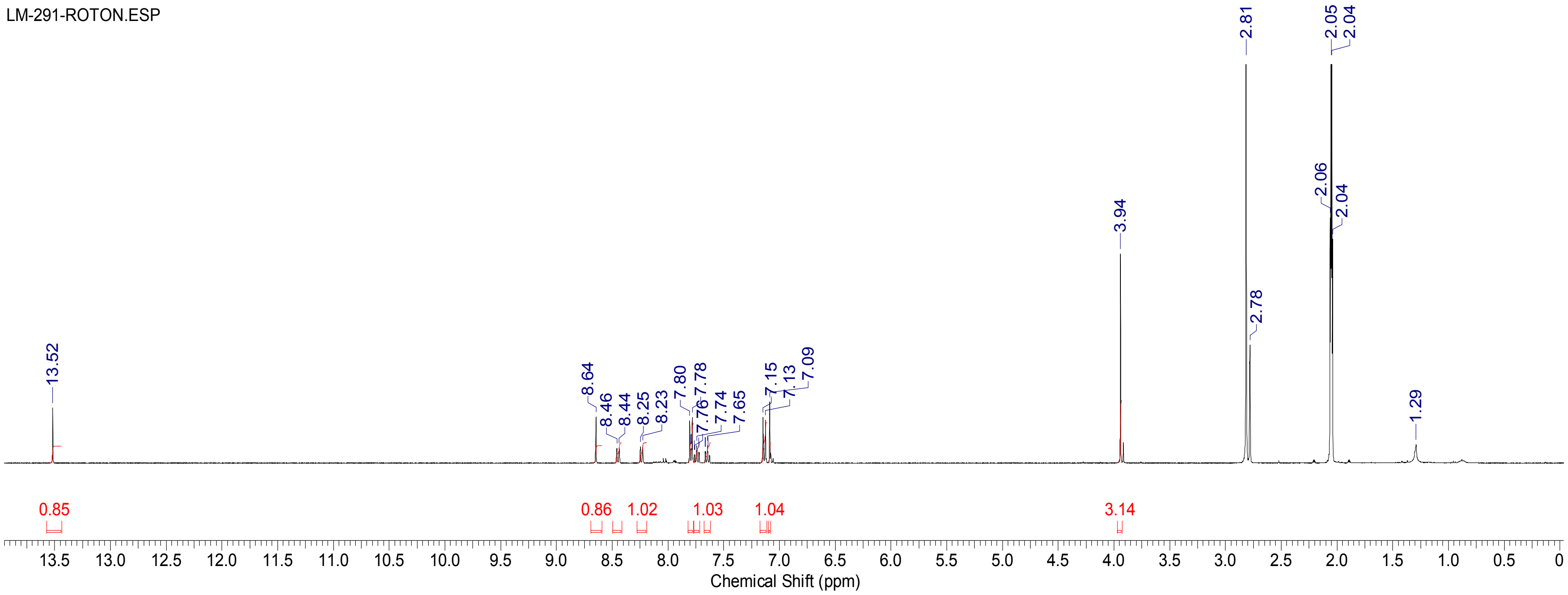




^{13}C NMR at 100 MHz in d$_6$-acetone

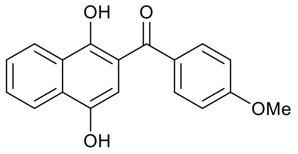
(1,4-dihydroxynaphthalen-2-yl)(4-methoxyphenyl)methanone 6l

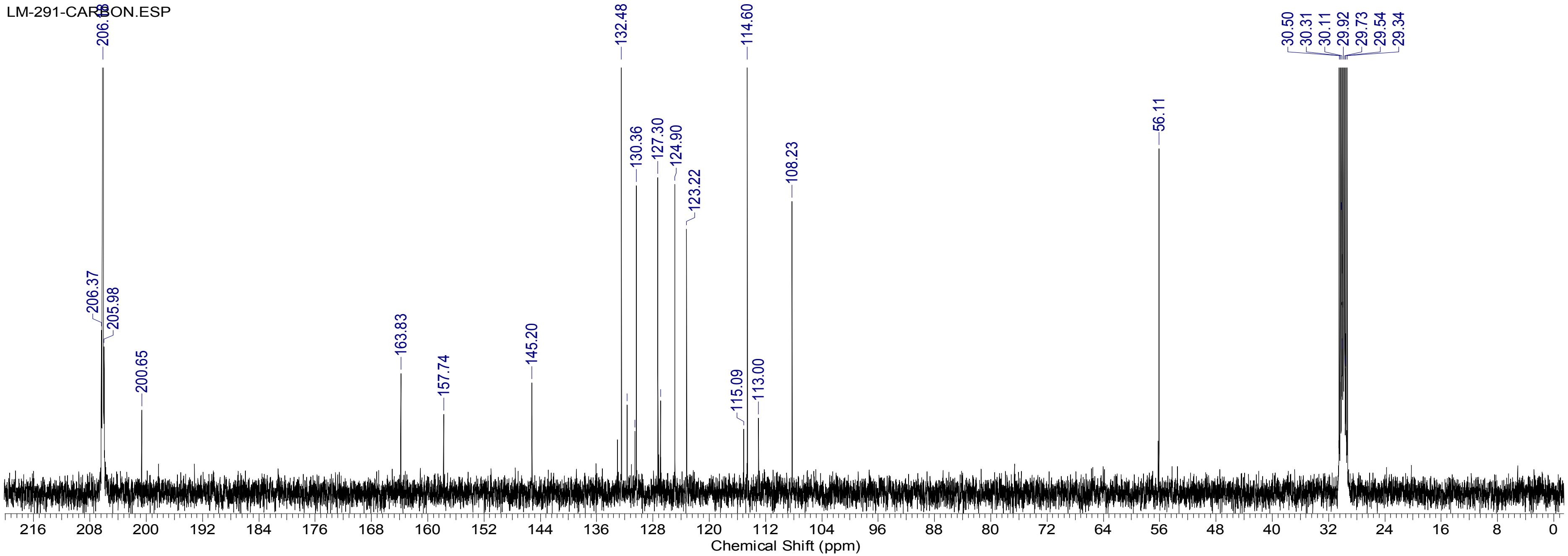




^{1}H NMR at 400 MHz in d$_6$-acetone

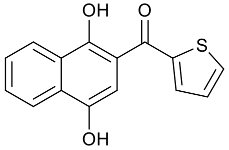
(1,4-dihydroxynaphthalen-2-yl)(4-methoxyphenyl)methanone 6l

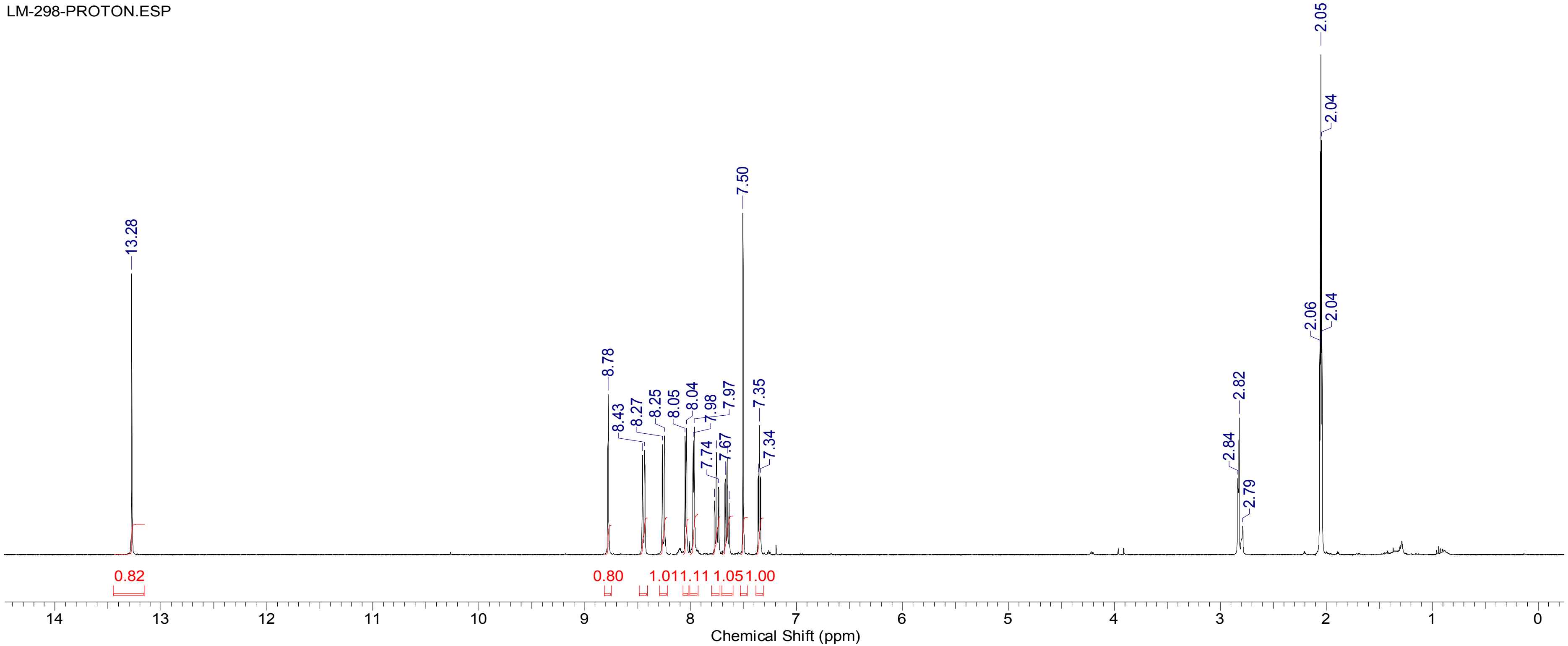




^{13}C NMR at 100 MHz in d$_6$-acetone

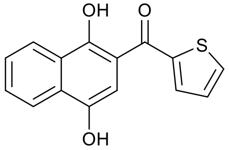
(1,4-dihydroxynaphthalen-2-yl)(thiophen-2-yl)methanone 6m

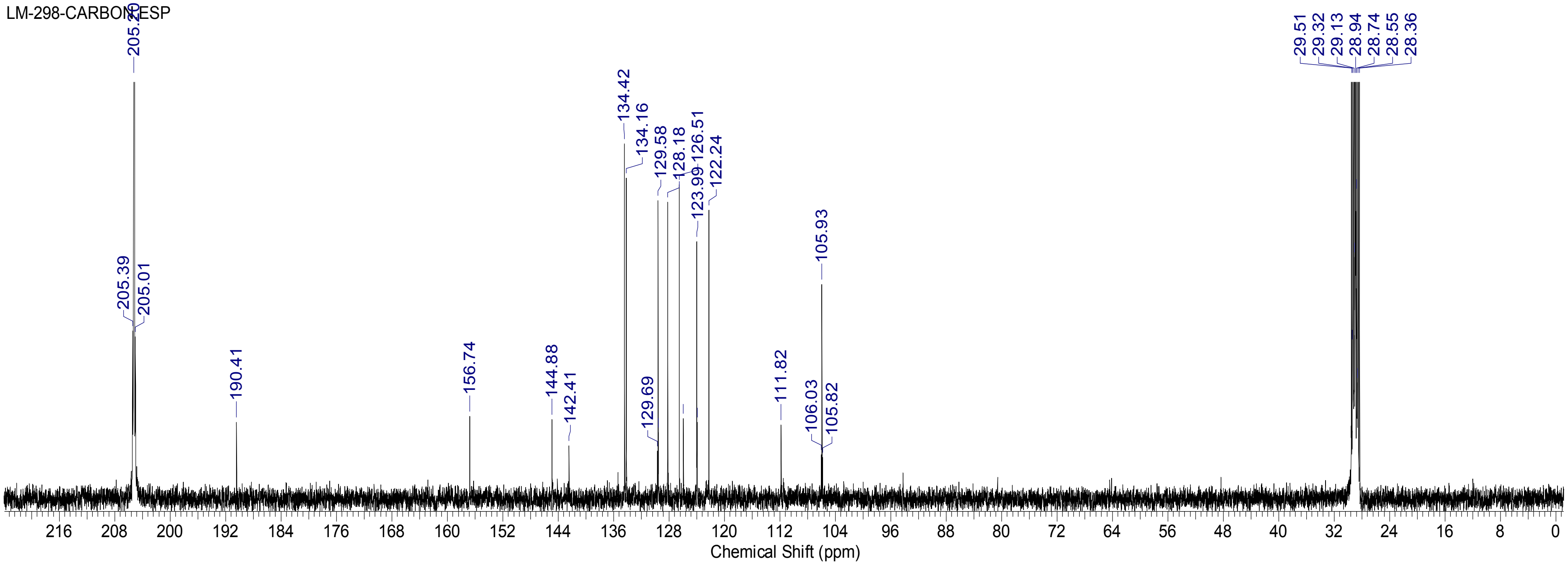




^{1}H NMR at 400 MHz in d$_6$-acetone

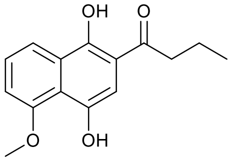
(1,4-dihydroxynaphthalen-2-yl)(thiophen-2-yl)methanone 6m

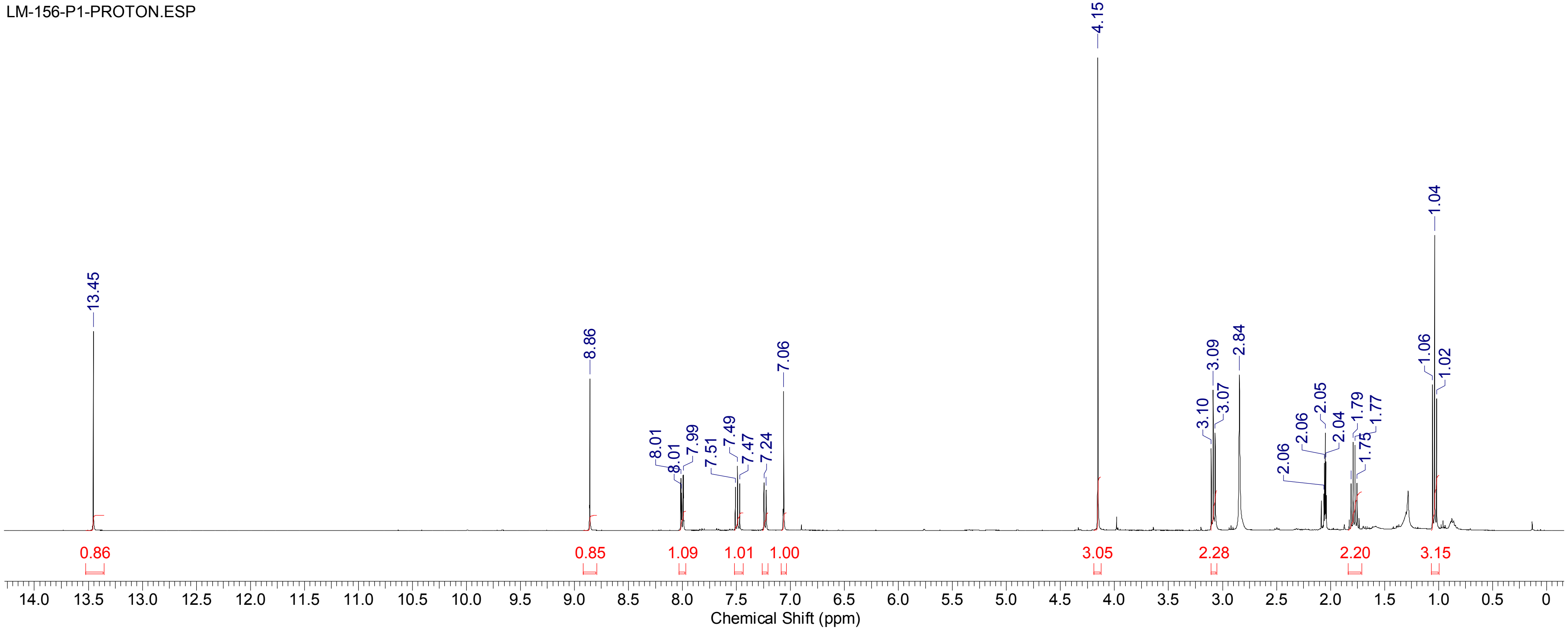




^{13}C NMR at 100 MHz in d_6-acetone

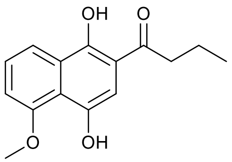
1-(1,4-Dihydroxy-5-methoxynaphthalen-2-yl)butan-1-one 9b

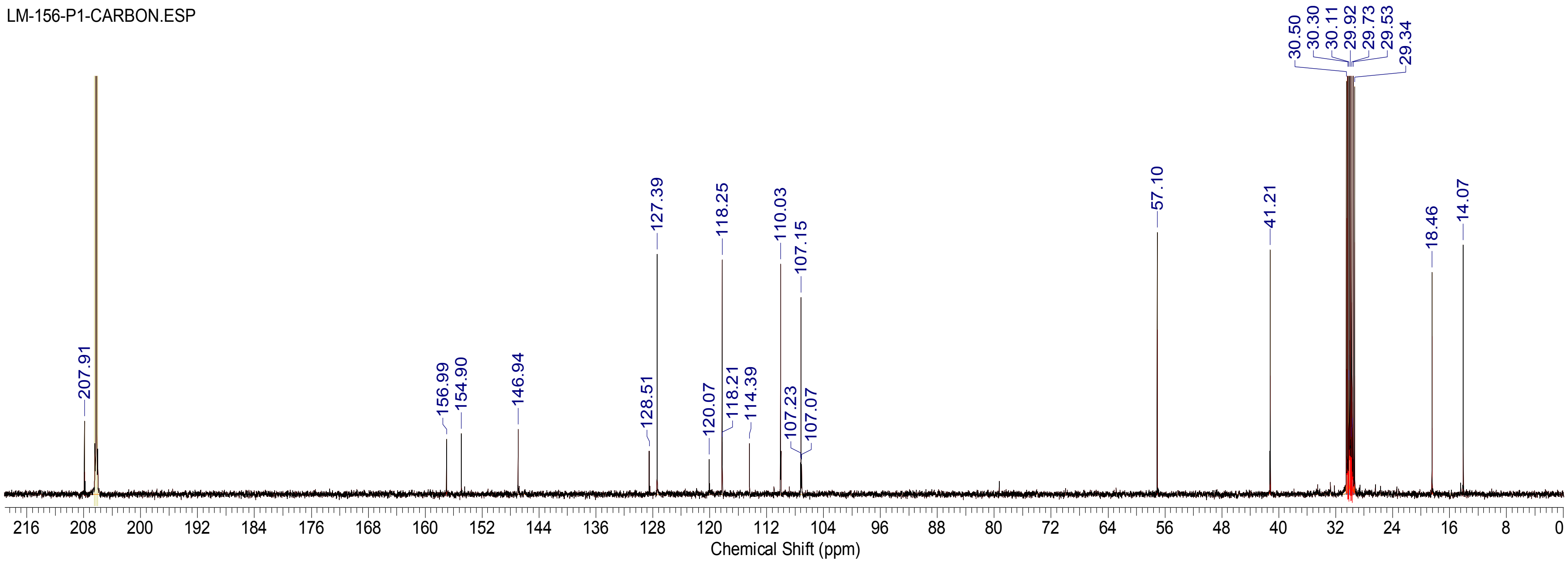




^{1}H NMR at 400 MHz in d$_6$-acetone

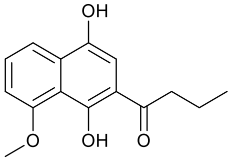
1-(1,4-Dihydroxy-5-methoxynaphthalen-2-yl)butan-1-one 9b

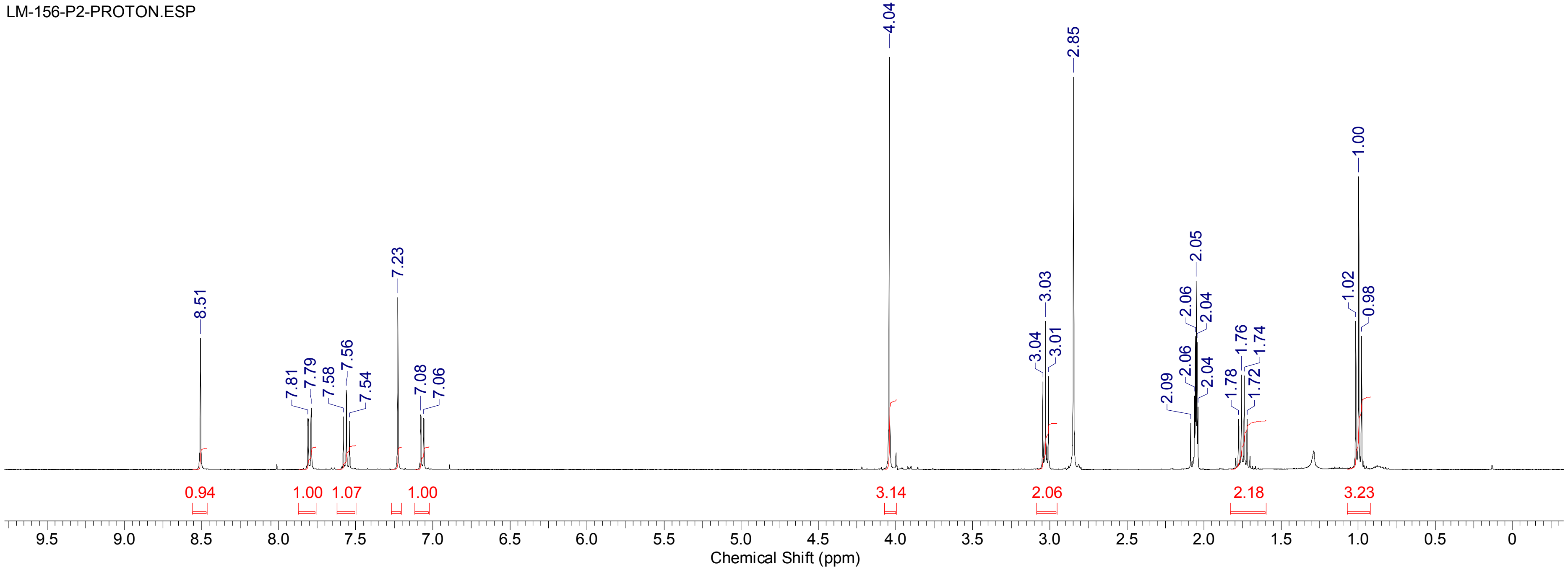




^{13}C NMR at 100 MHz in d$_6$-acetone

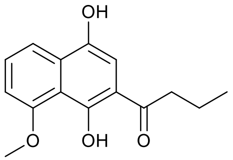
1-(1,4-Dihydroxy-8-methoxynaphthalen-2-yl)butan-1-one 10b

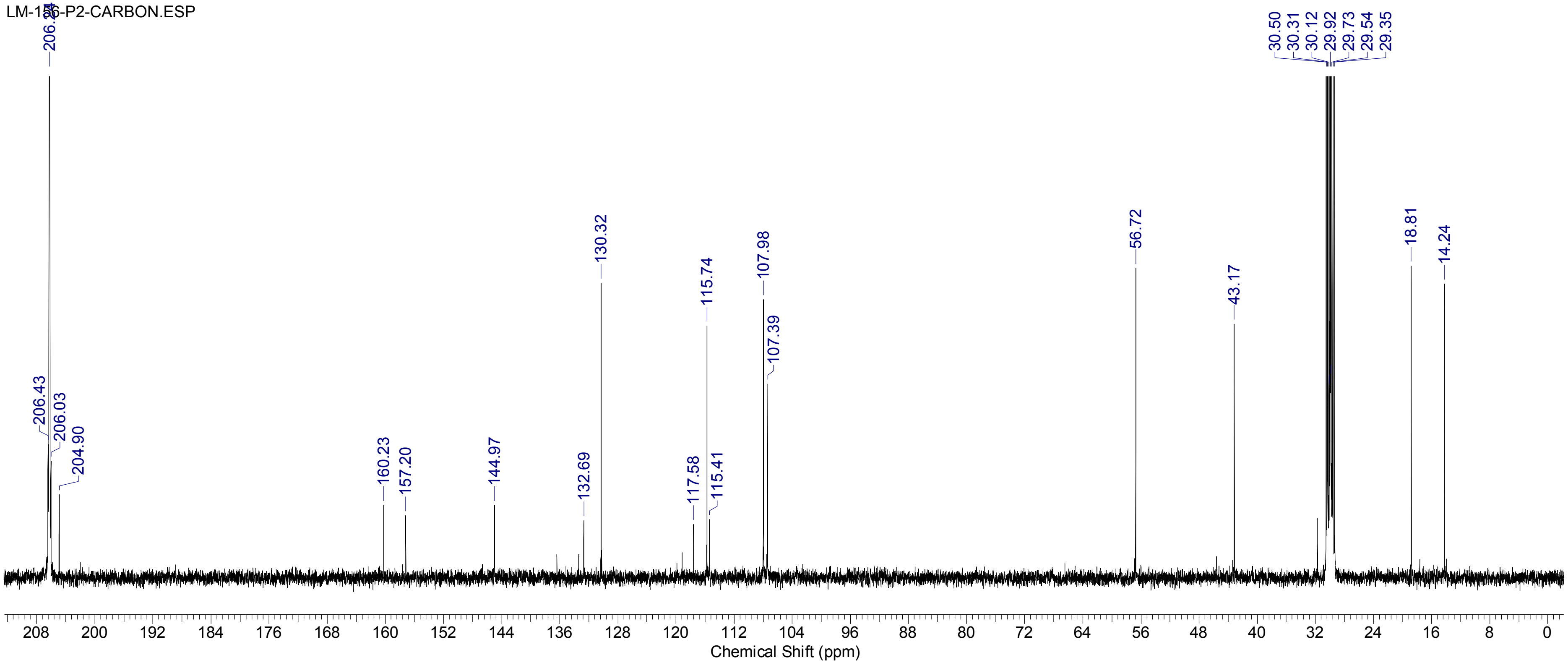




^{1}H NMR at 400 MHz in d$_6$-acetone

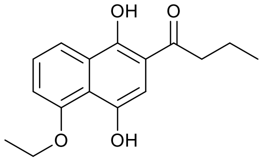
1-(1,4-Dihydroxy-8-methoxynaphthalen-2-yl)butan-1-one 10b

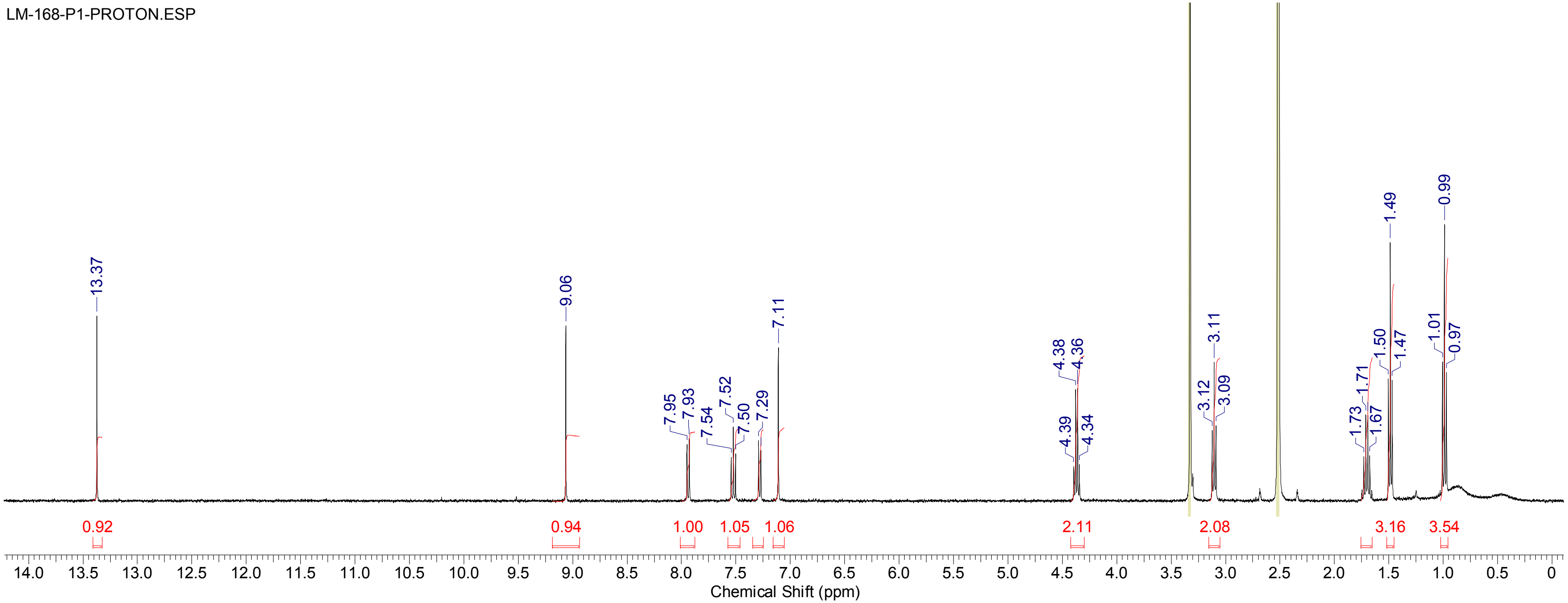




^{13}C NMR at 100 MHz in d$_6$-acetone

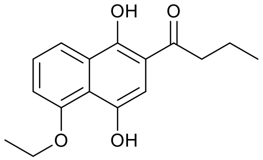
1-(5-Ethoxy-1,4-dihydroxynaphthalen-2-yl)butan-1-one 9c

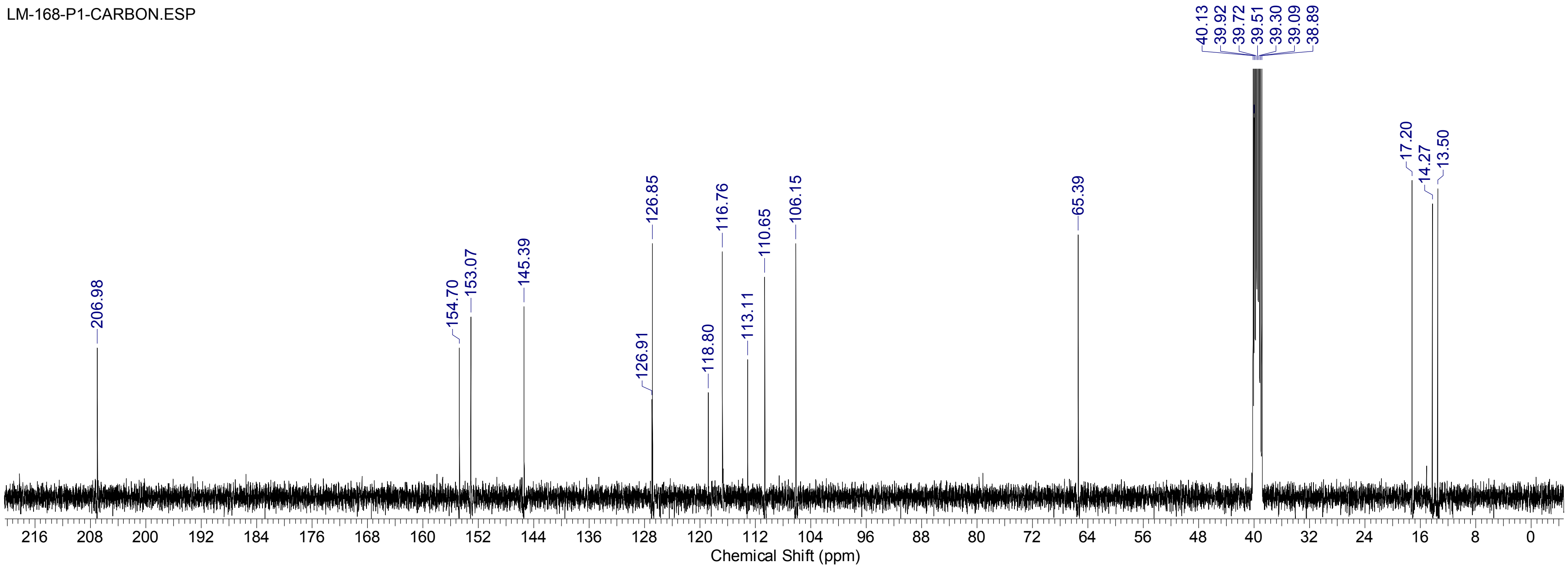




^{1}H NMR at 400 MHz in d$_6$-DMSO

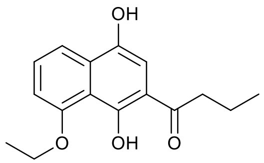
1-(5-Ethoxy-1,4-dihydroxynaphthalen-2-yl)butan-1-one 9c

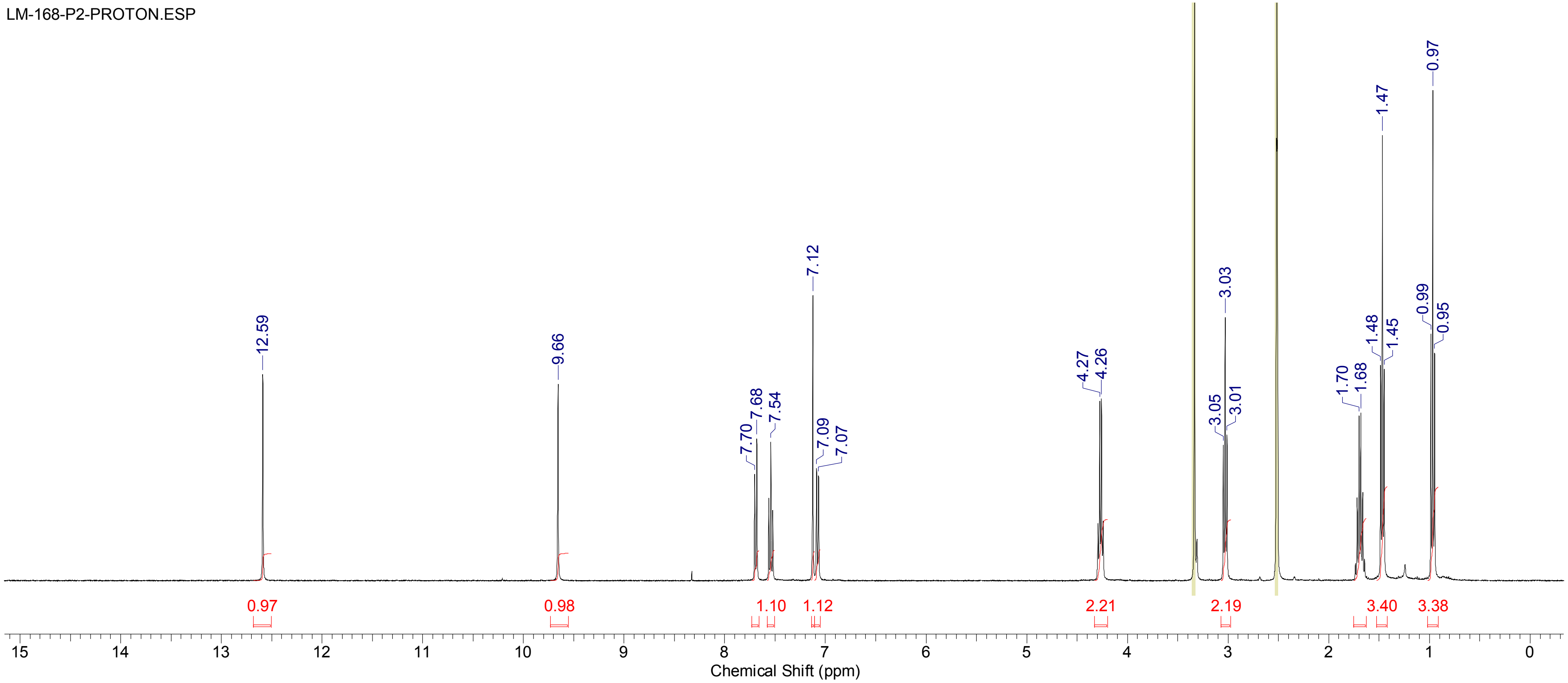




^{13}C NMR at 100 MHz in d_6-DMSO

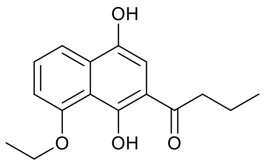
1-(8-Ethoxy-1,4-dihydroxynaphthalen-2-yl)butan-1-one 10c

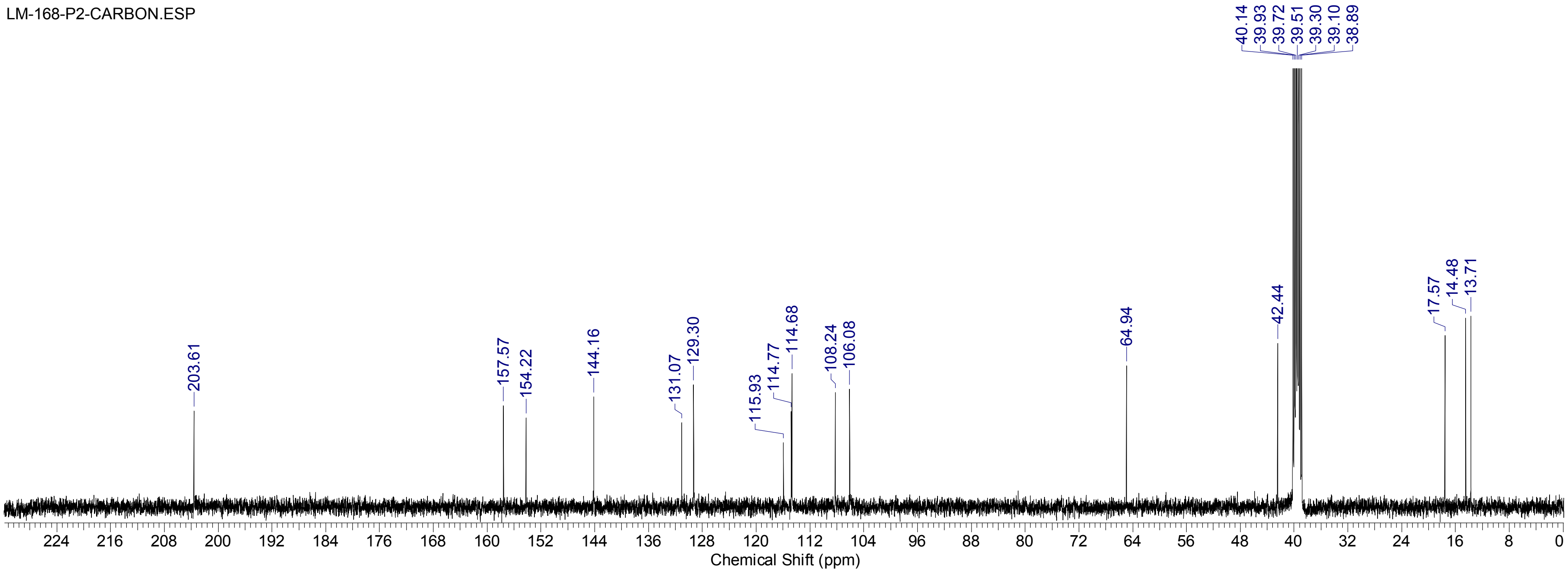




^{1}H NMR at 400 MHz in d$_6$-DMSO

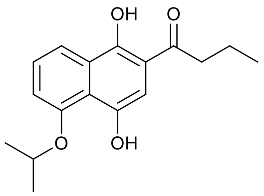
1-(8-Ethoxy-1,4-dihydroxynaphthalen-2-yl)butan-1-one 10c

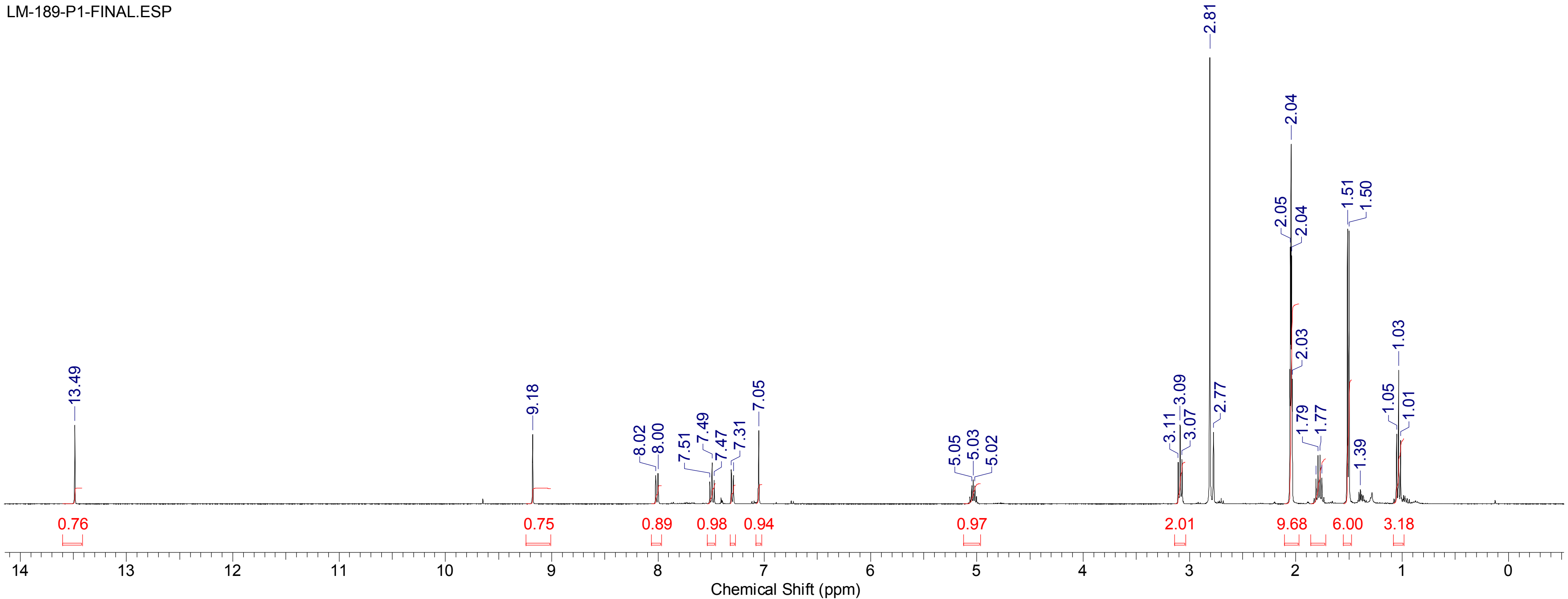




^{13}C NMR at 100 MHz in d_6-DMSO

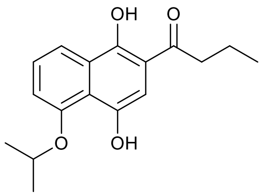
1-(1,4-Dihydroxy-5-isopropoxynaphthalen-2-yl)butan-1-one 9d

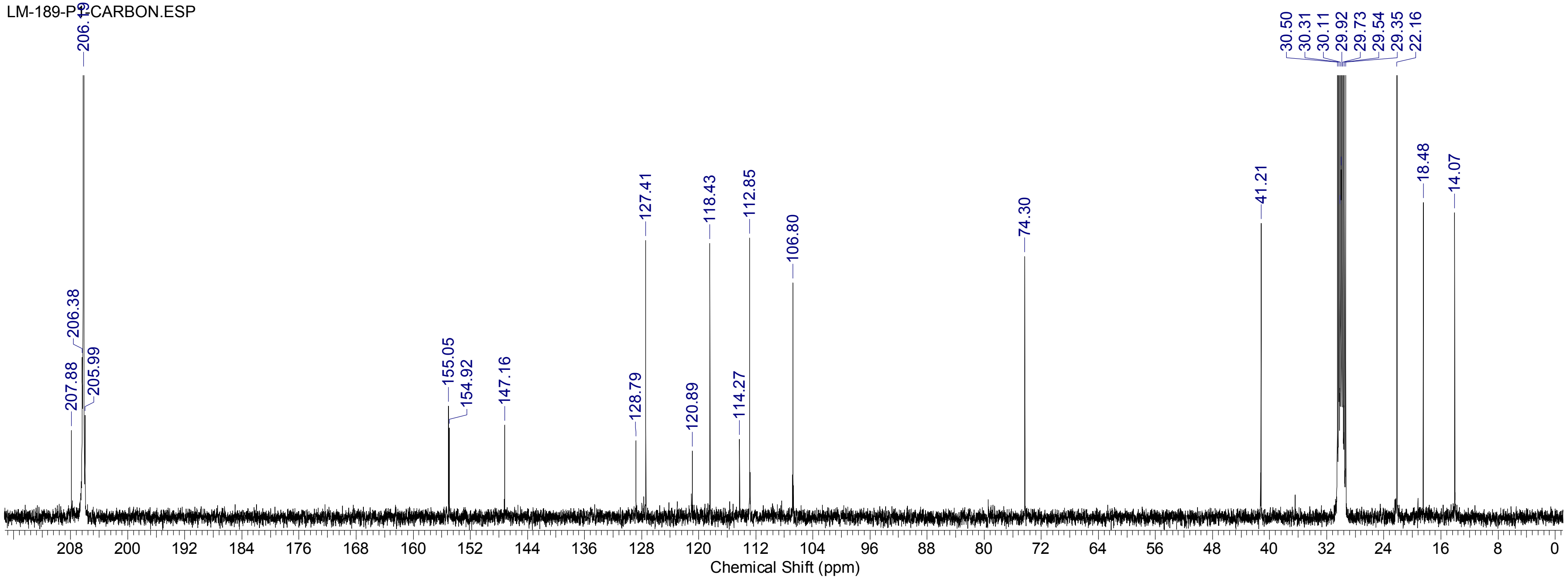




^{1}H NMR at 400 MHz in d$_6$-acetone

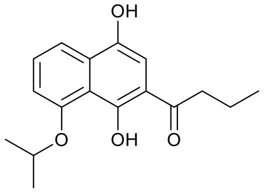
1-(1,4-Dihydroxy-5-isopropoxynaphthalen-2-yl)butan-1-one 9d

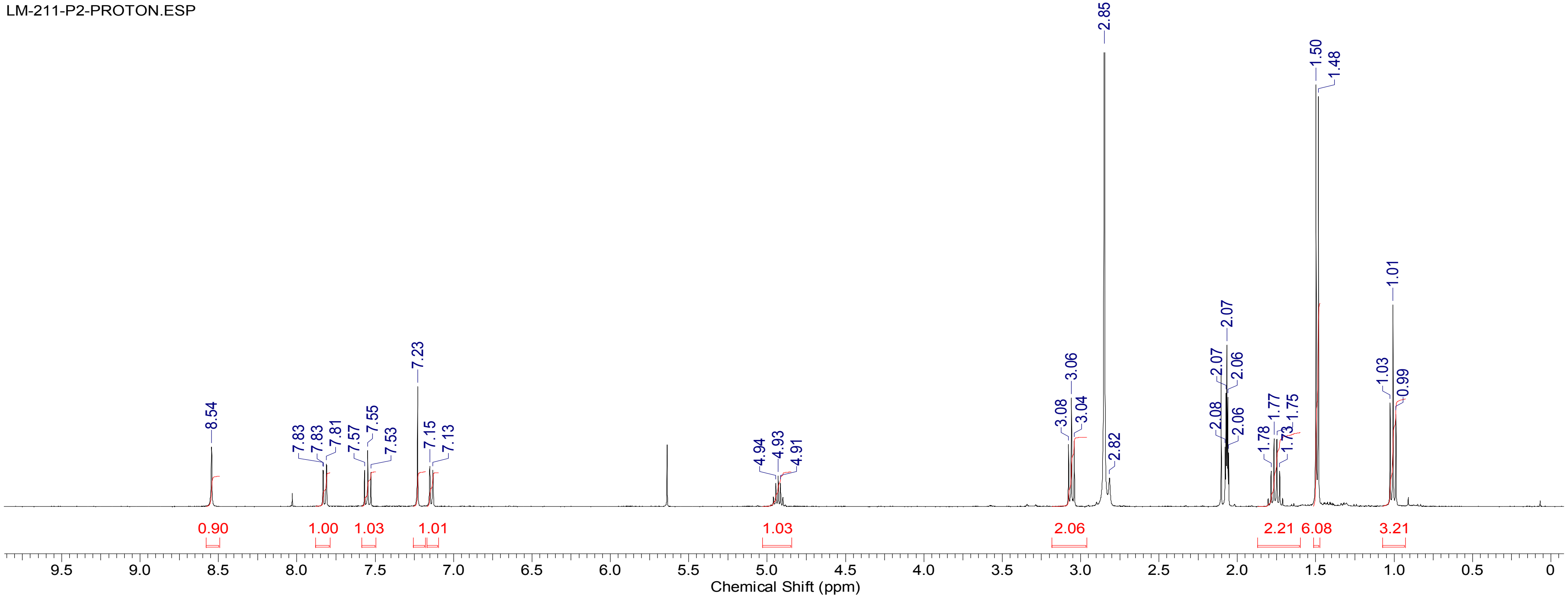




^{13}C NMR at 100 MHz in d$_6$-acetone

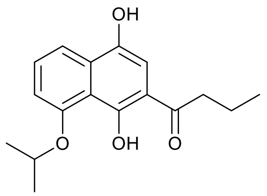
1-(1,4-Dihydroxy-8-isopropoxynaphthalen-2-yl)butan-1-one 10d

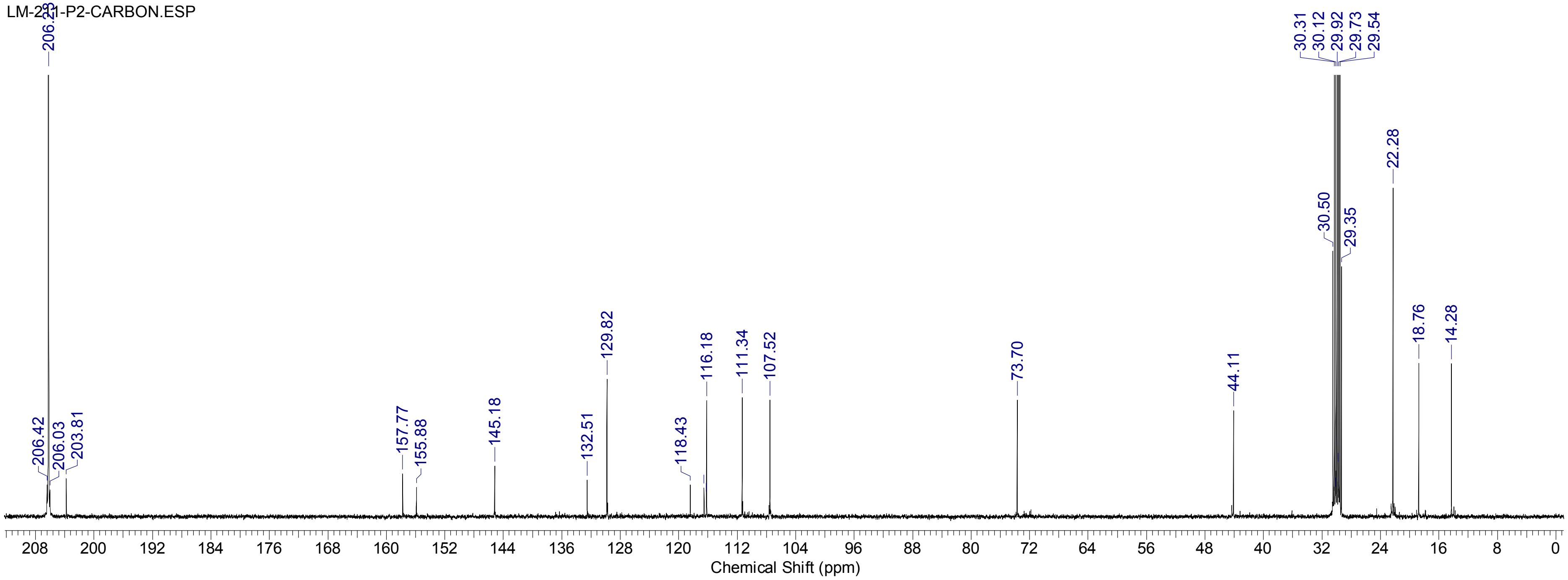




^{1}H NMR at 400 MHz in d_6-acetone

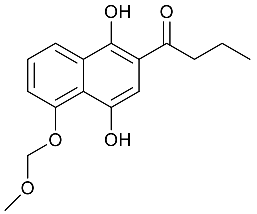
1-(1,4-Dihydroxy-8-isopropoxynaphthalen-2-yl)butan-1-one 10d

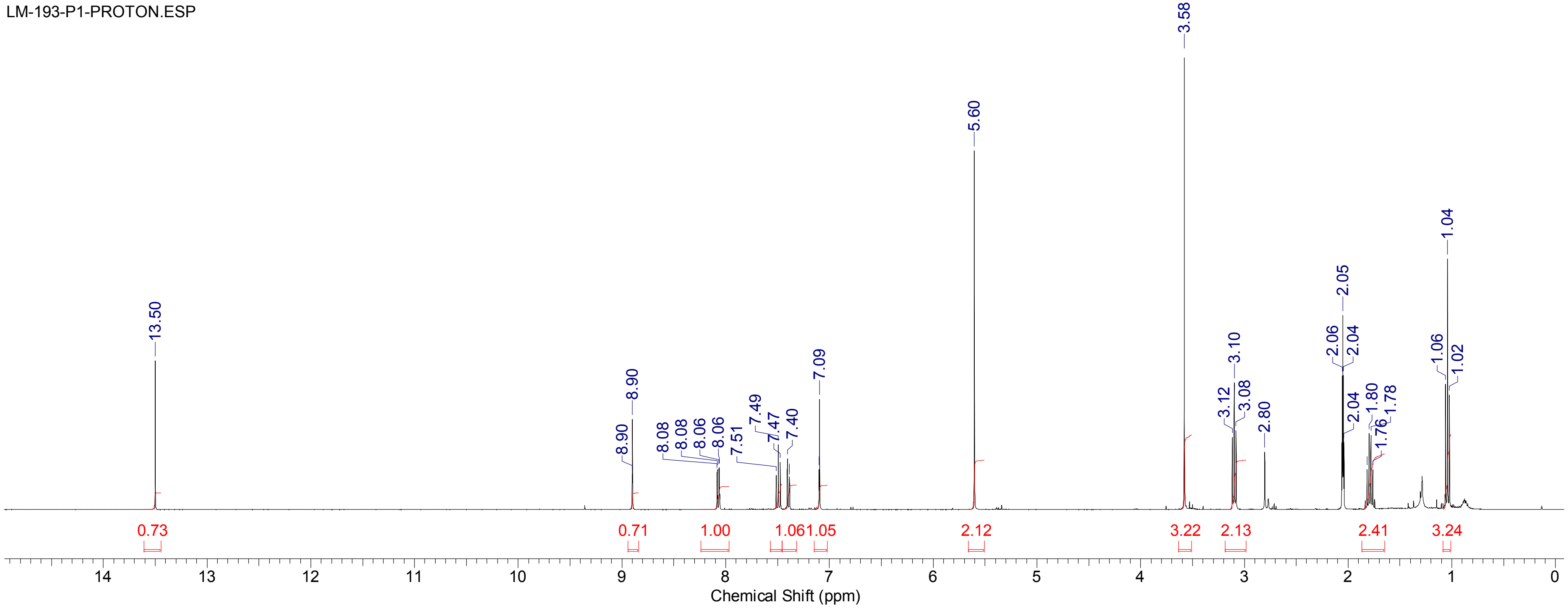




^{13}C NMR at 100 MHz in d$_6$-acetone

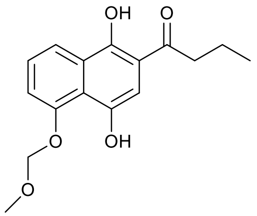
1-(1,4-Dihydroxy-5-(methoxymethoxy)naphthalen-2-yl)butan-1-one 9e

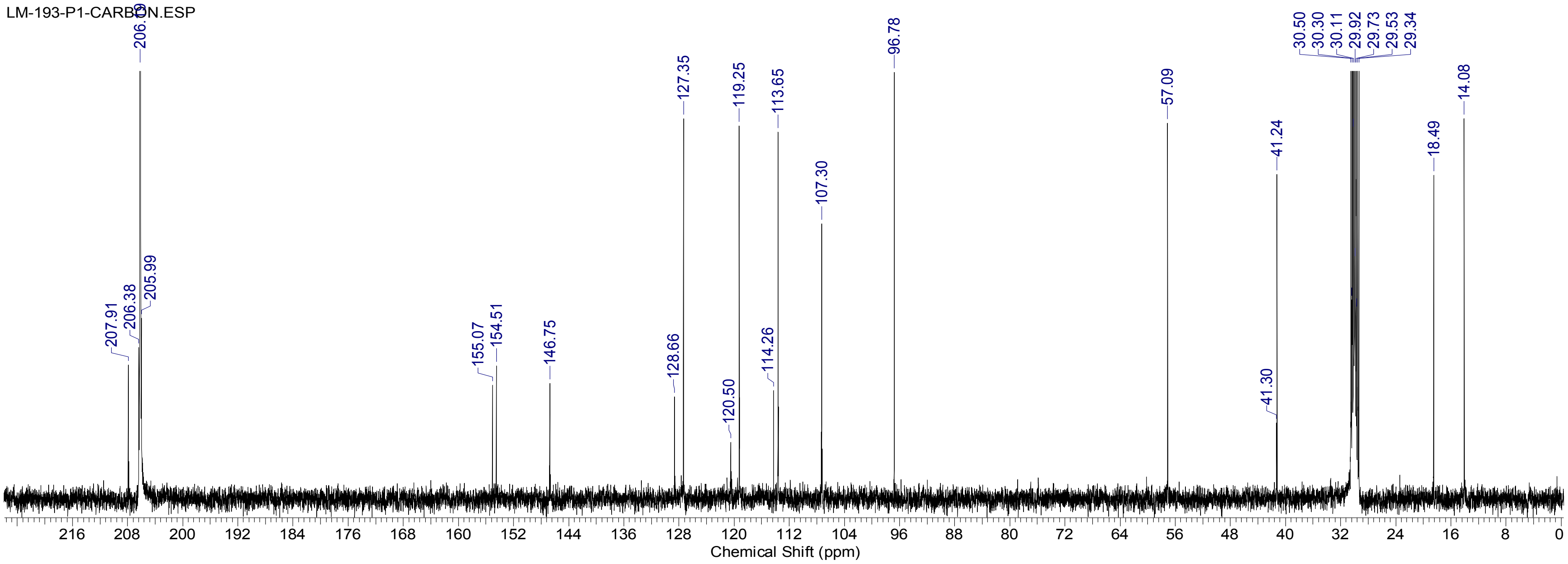




^{1}H NMR at 400 MHz in d_6-acetone

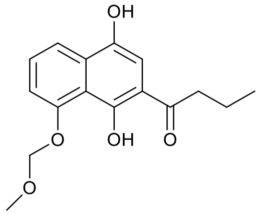
1-(1,4-Dihydroxy-5-(methoxymethoxy)naphthalen-2-yl)butan-1-one 9e

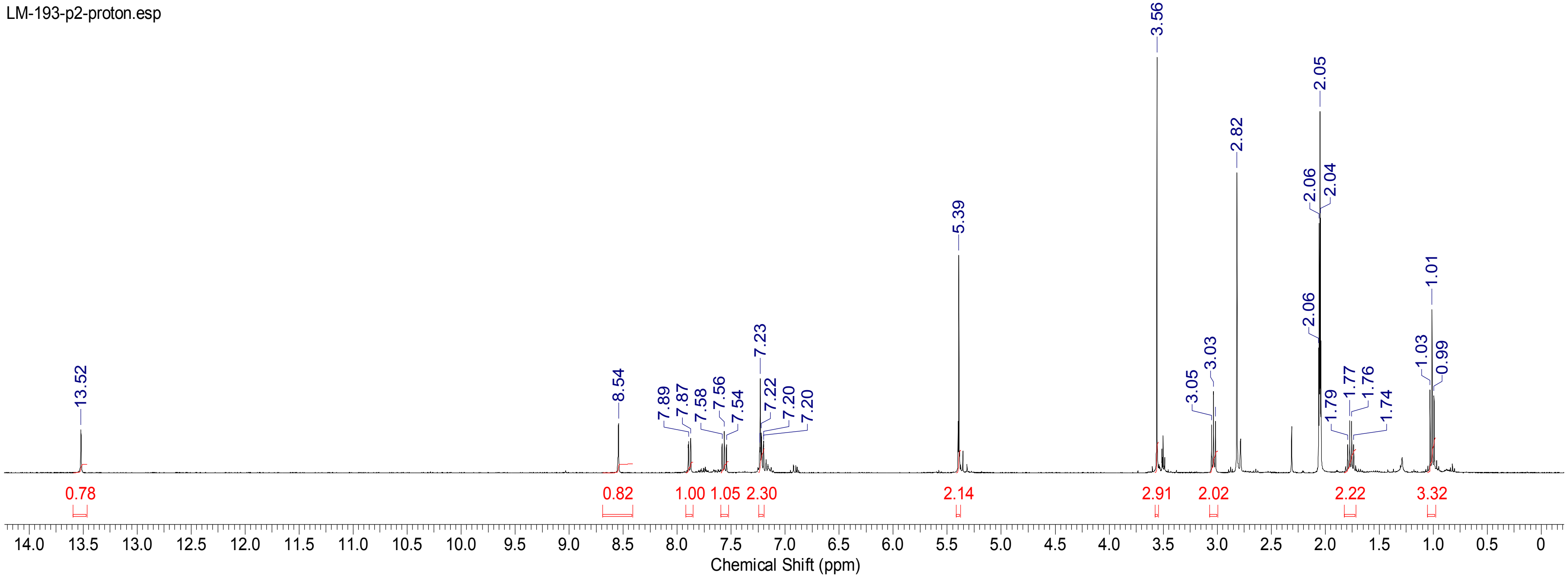




^{13}C NMR at 100 MHz in d$_6$-acetone

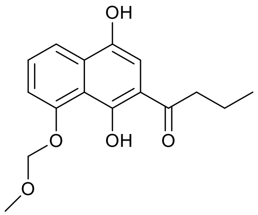
1-(1,4-Dihydroxy-8-(methoxymethoxy)naphthalen-2-yl)butan-1-one 10e

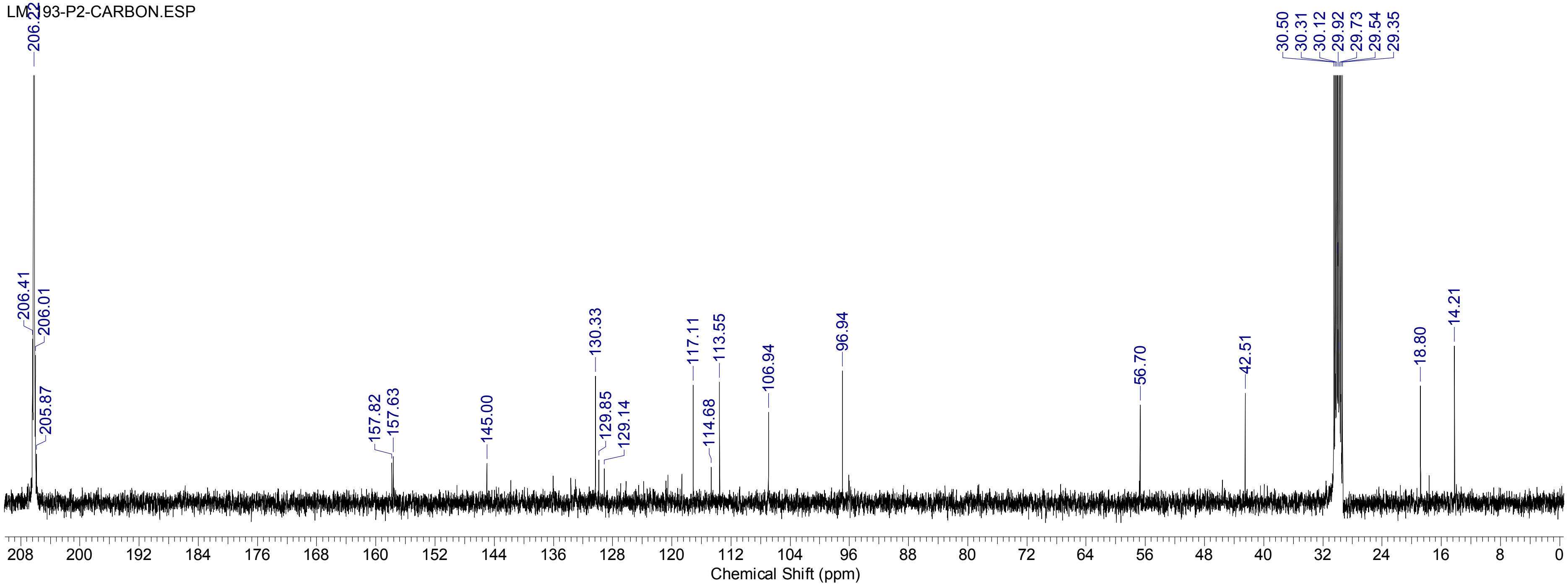




^{1}H NMR at 400 MHz in d$_6$-acetone

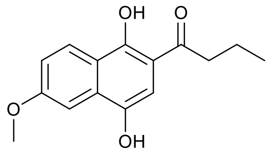
1-(1,4-Dihydroxy-8-(methoxymethoxy)naphthalen-2-yl)butan-1-one 10e

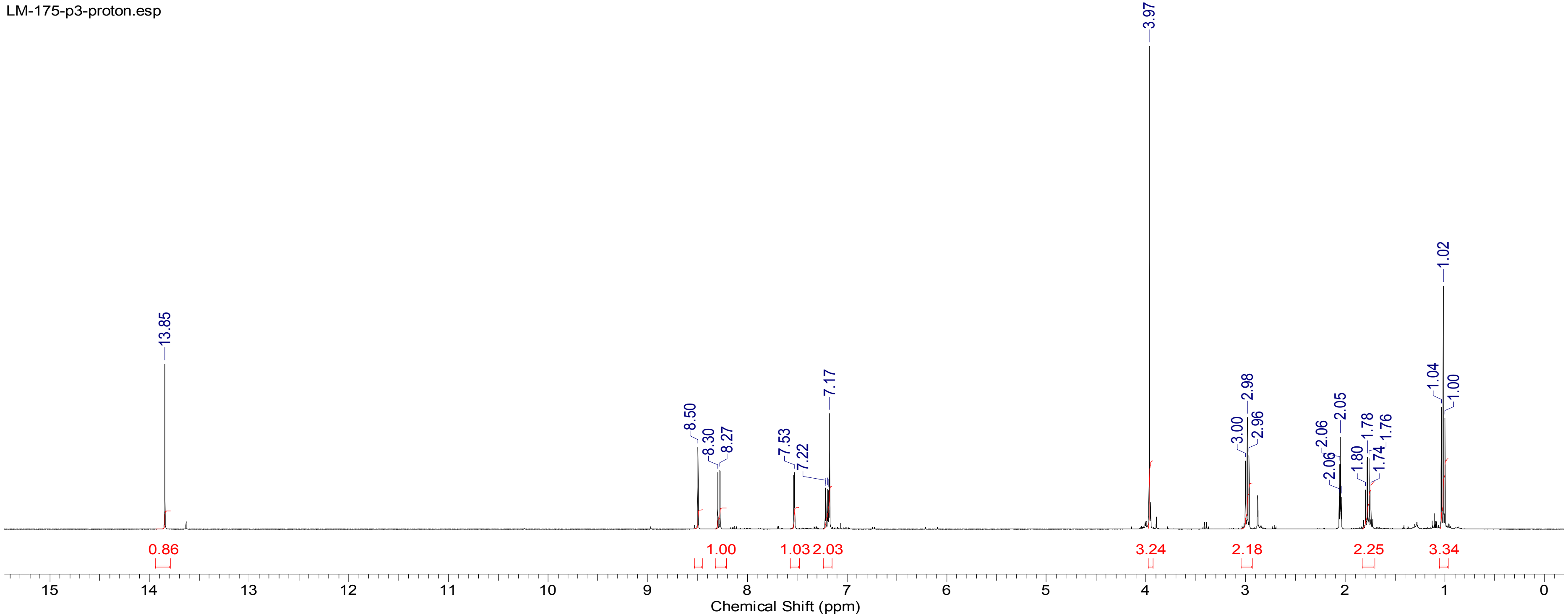




^{13}C NMR at 100 MHz in d_6-acetone

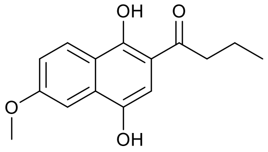
1-(1,4-Dihydroxy-6-methoxynaphthalen-2-yl)butan-1-one 13b

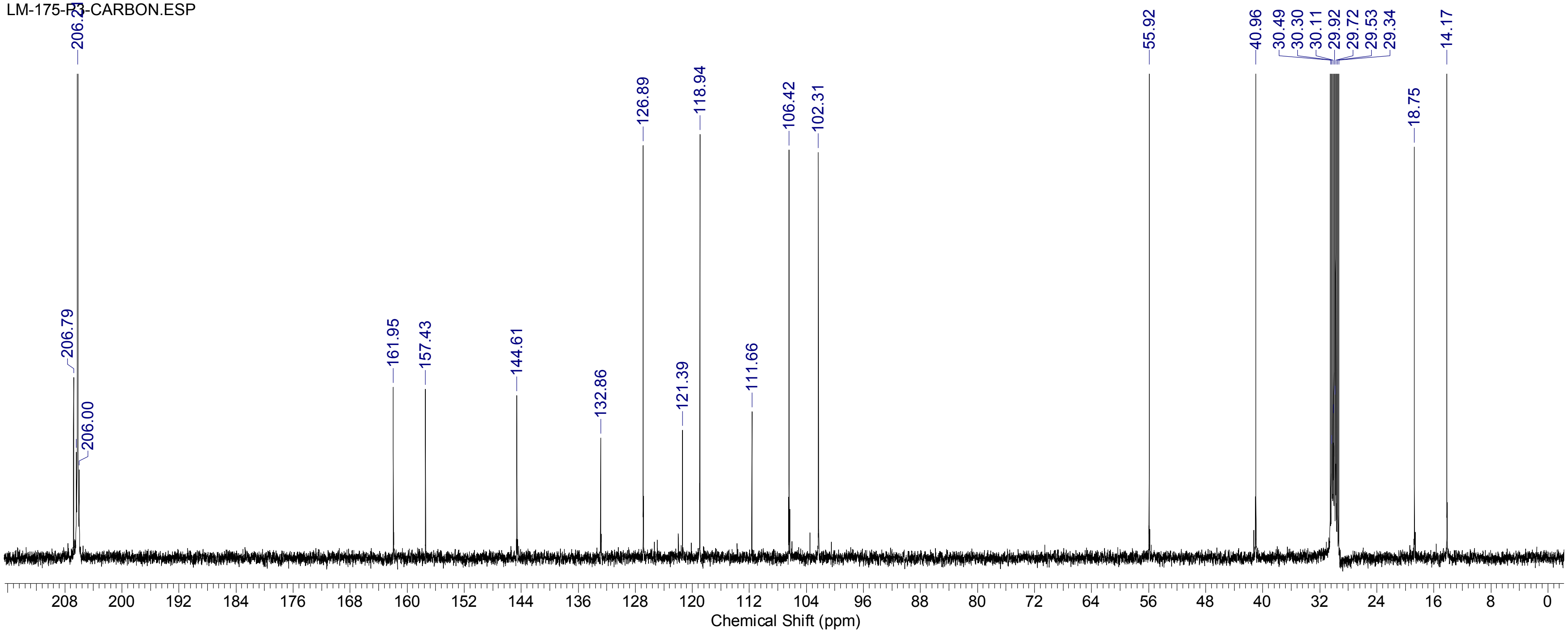




^{1}H NMR at 400 MHz in d_6-acetone

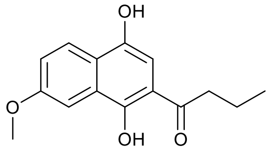
1-(1,4-Dihydroxy-6-methoxynaphthalen-2-yl)butan-1-one 13b

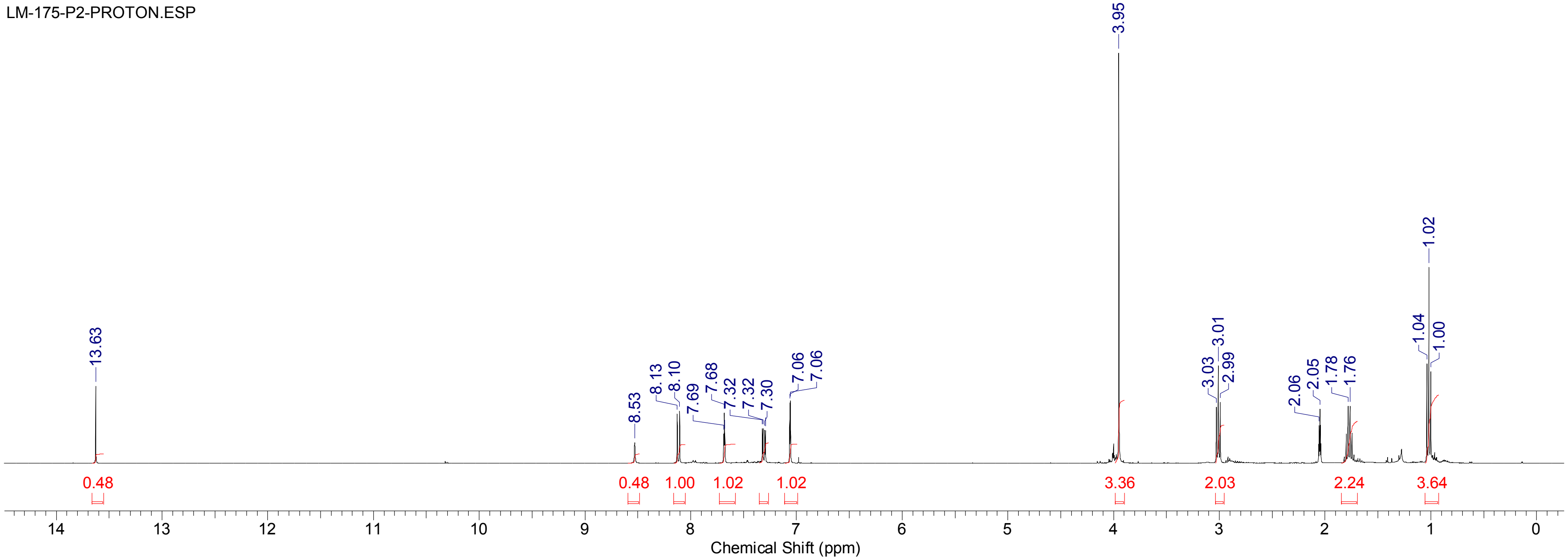




^{13}C NMR at 100 MHz in d$_6$-acetone

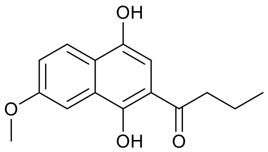
1-(1,4-Dihydroxy-7-methoxynaphthalen-2-yl)butan-1-one 14b

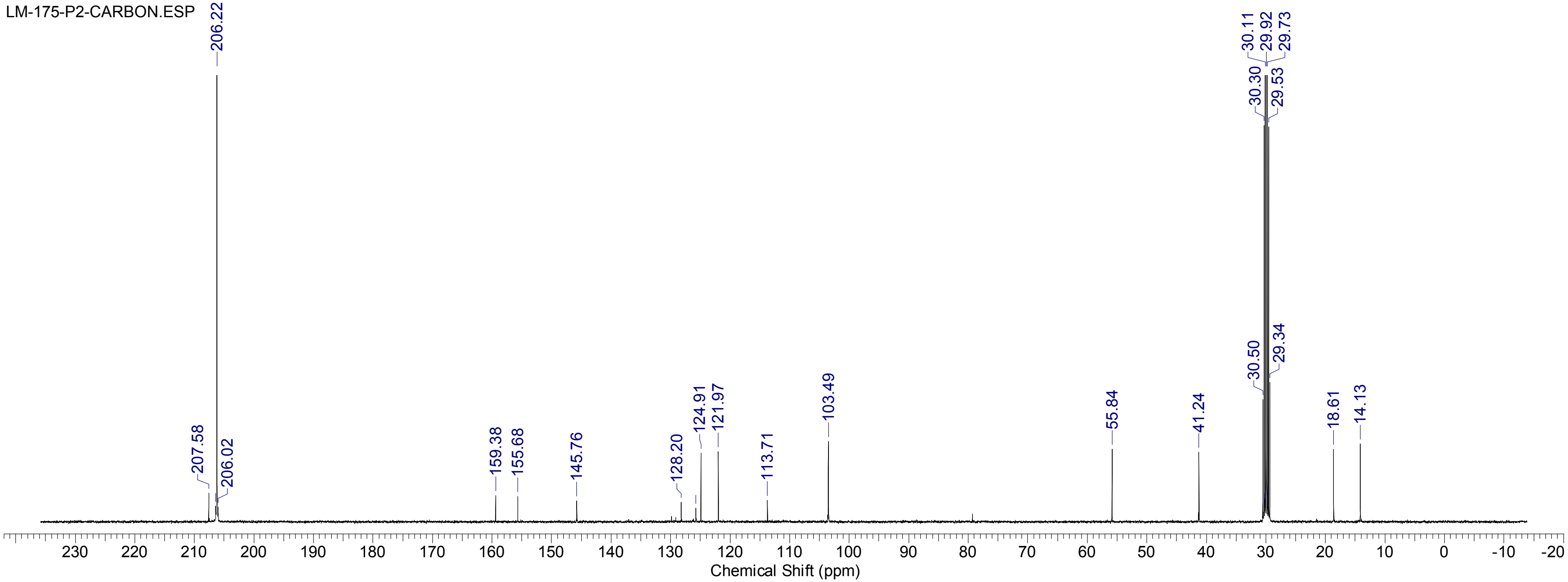




^{1}H NMR at 400 MHz in d$_6$-acetone

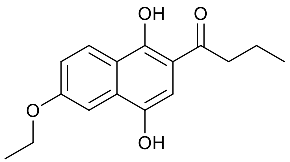
1-(1,4-Dihydroxy-7-methoxynaphthalen-2-yl)butan-1-one 14b

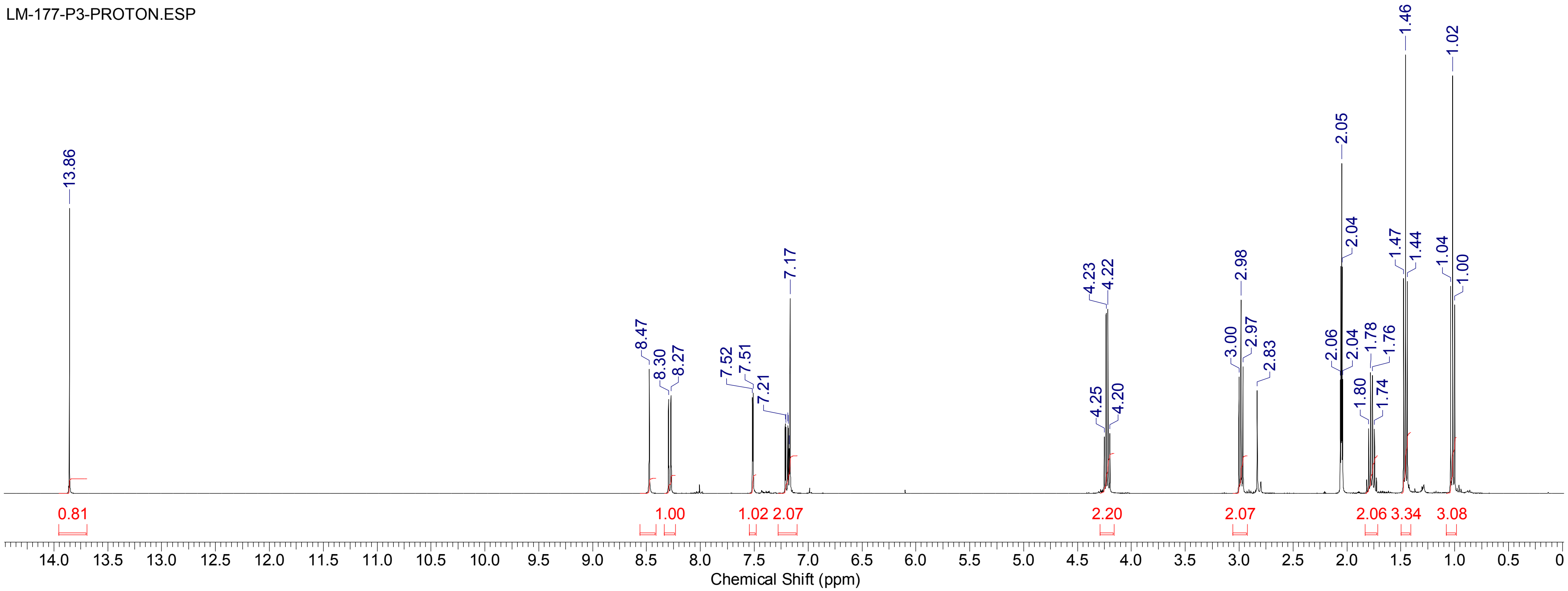




^{13}C NMR at 100 MHz in d$_6$-acetone

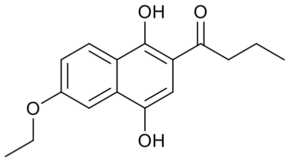
1-(6-Ethoxy-1,4-dihydroxynaphthalen-2-yl)butan-1-one 13c

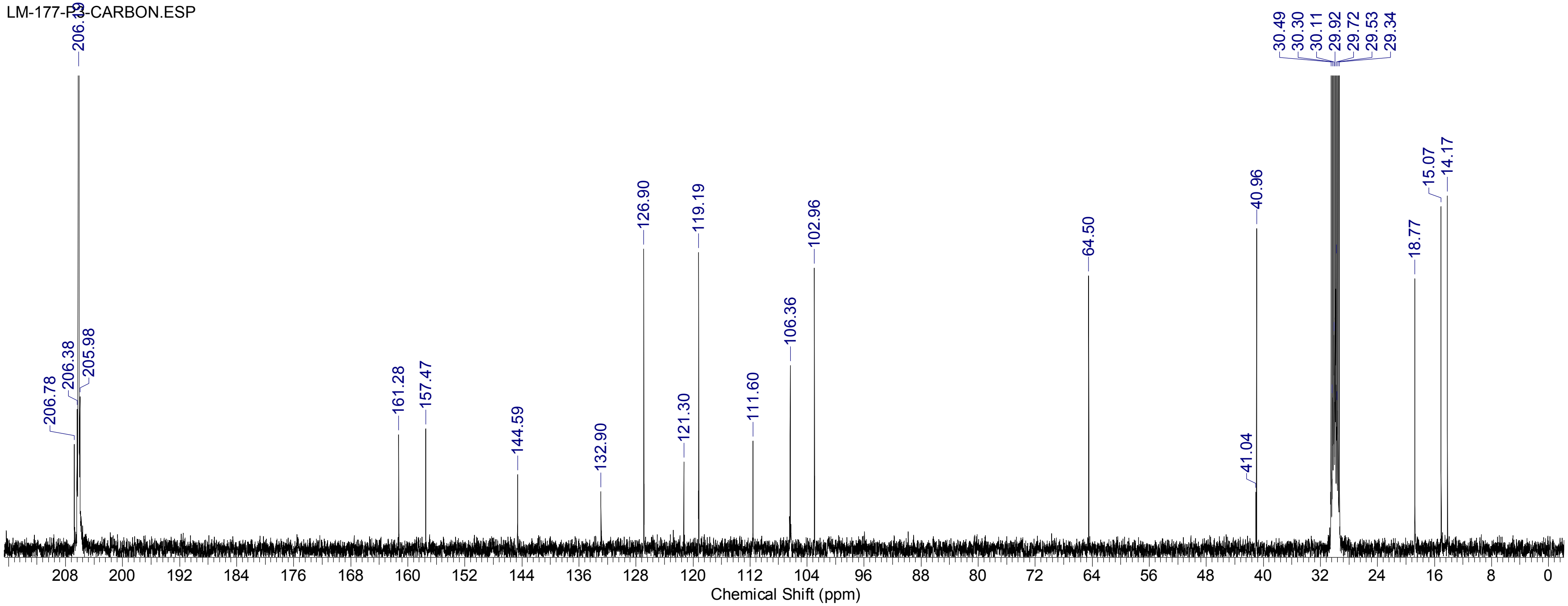




^{1}H NMR at 400 MHz in d$_6$-acetone

1-(6-Ethoxy-1,4-dihydroxynaphthalen-2-yl)butan-1-one 13c





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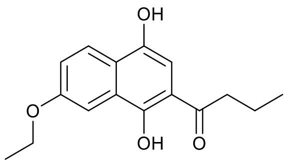

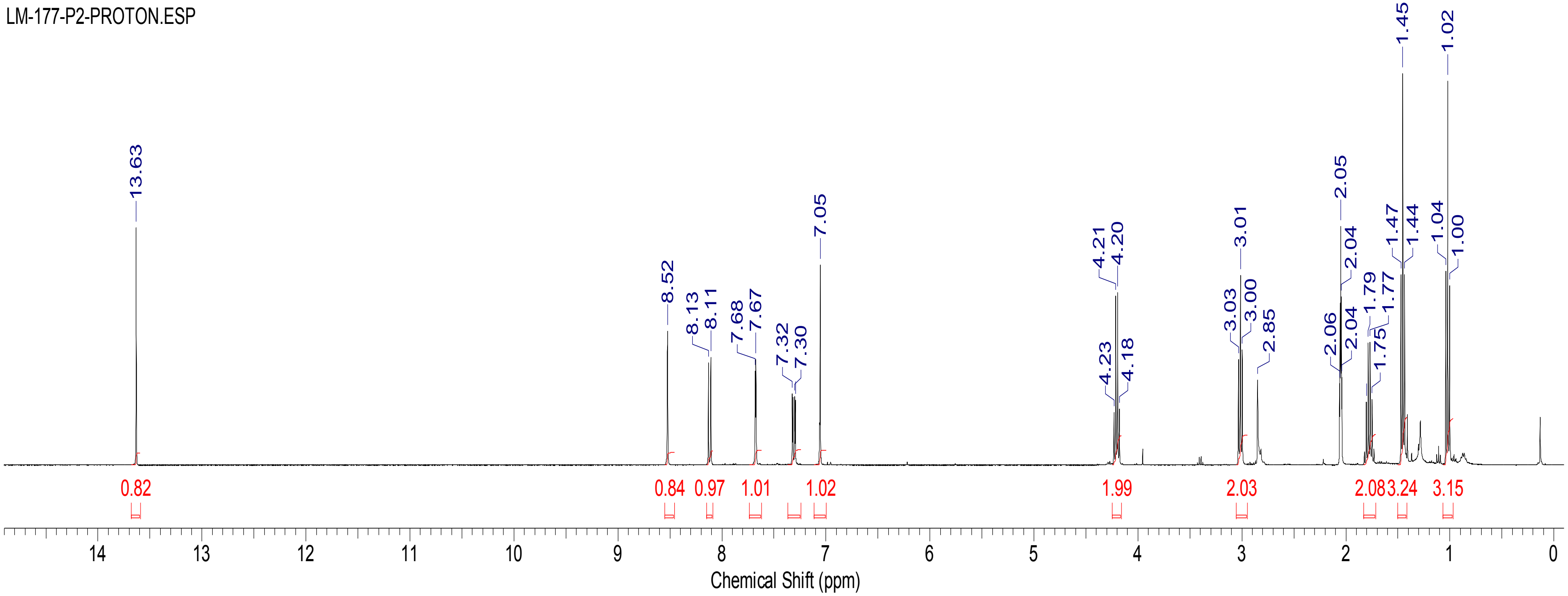

^{1}H NMR at 400 MHz in d$_6$-acetone

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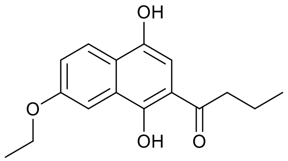
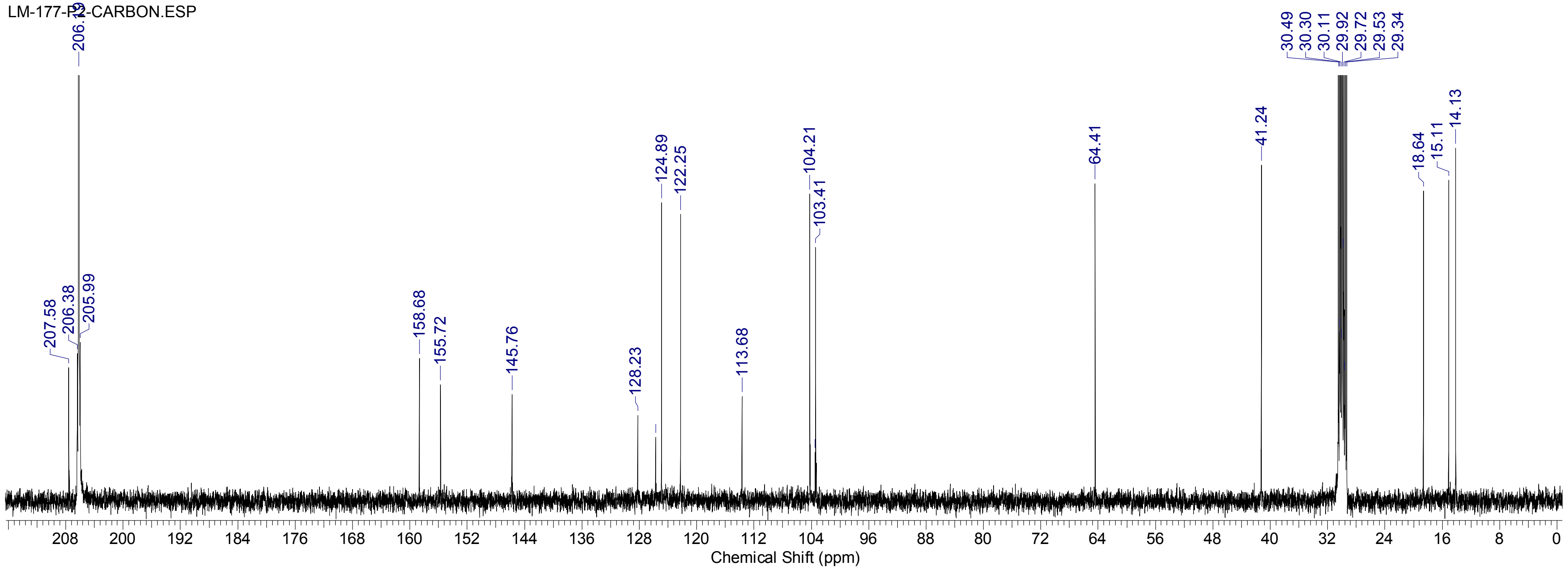
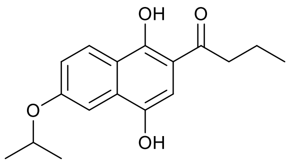
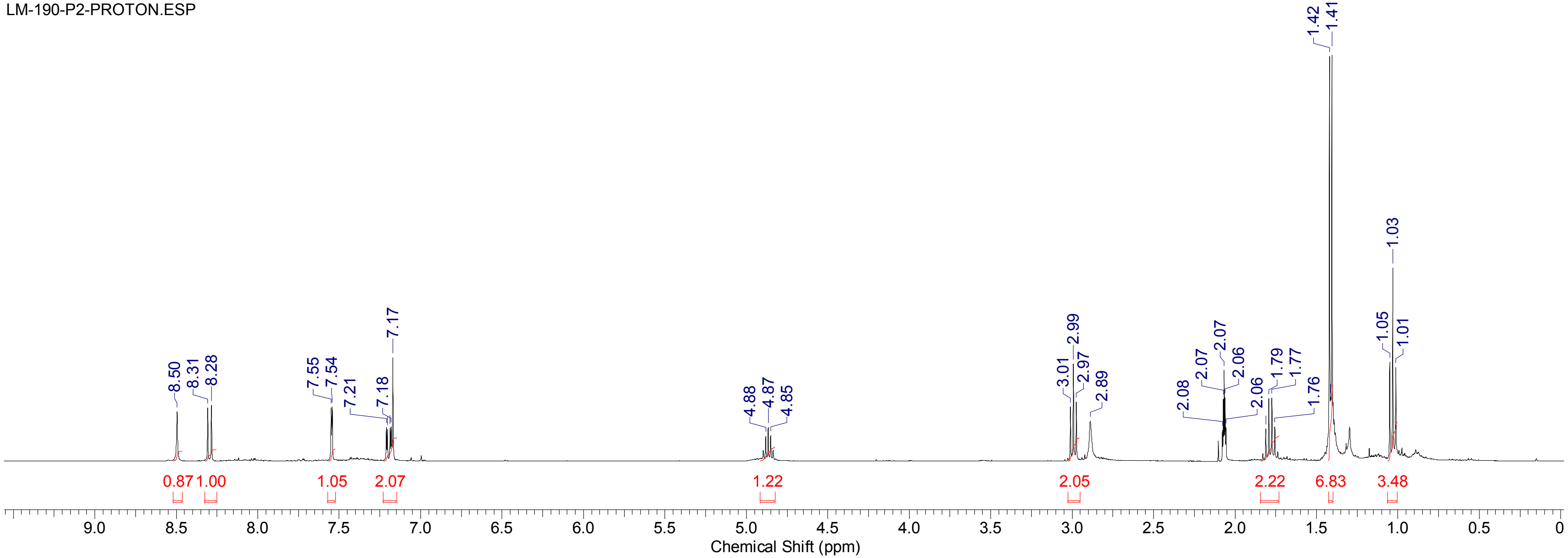




^{13}C NMR at 100 MHz in d_6-acetone

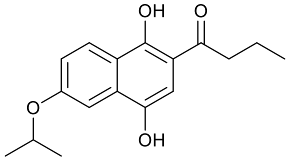
1-(1,4-Dihydroxy-6-isopropoxynaphthalen-2-yl)butan-1-one 13d

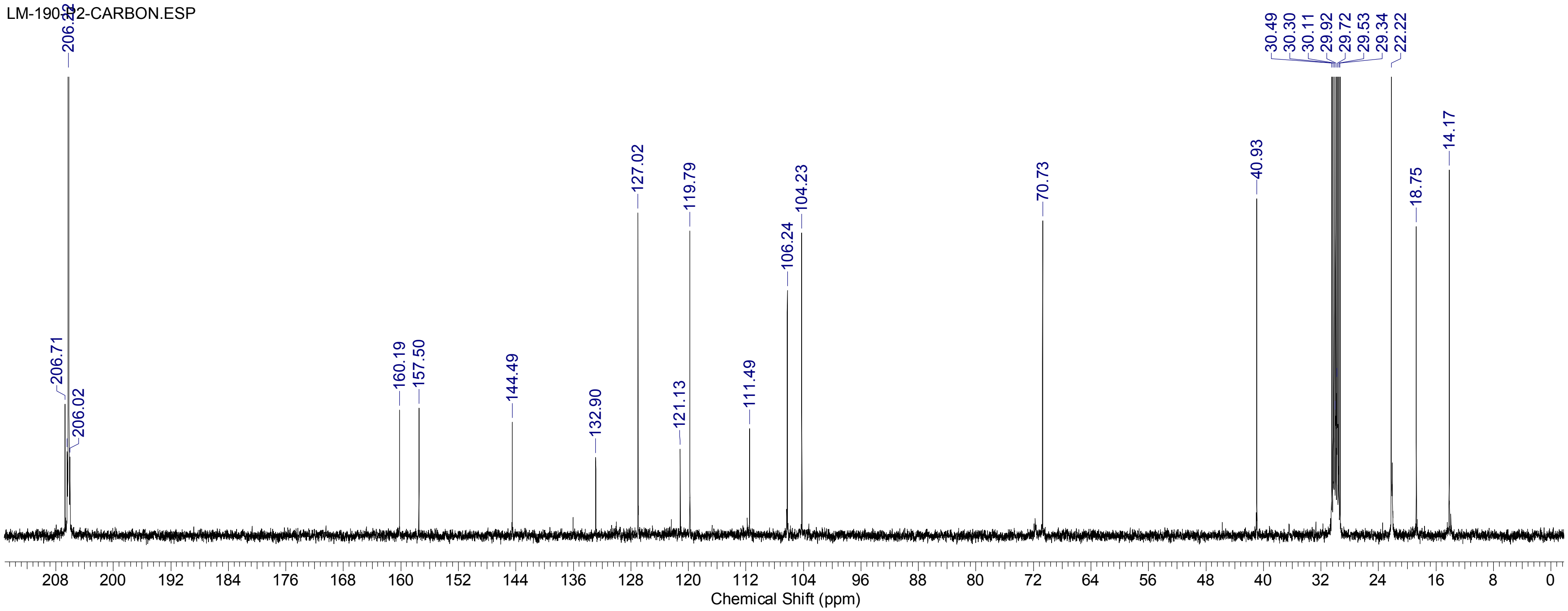




^{1}H NMR at 400 MHz in d$_6$-acetone

1-(1,4-Dihydroxy-6-isopropoxynaphthalen-2-yl)butan-1-one 13d





^{13}C NMR at 100 MHz in d_6-acetone

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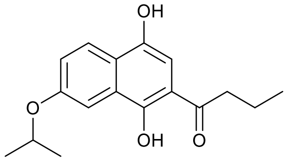

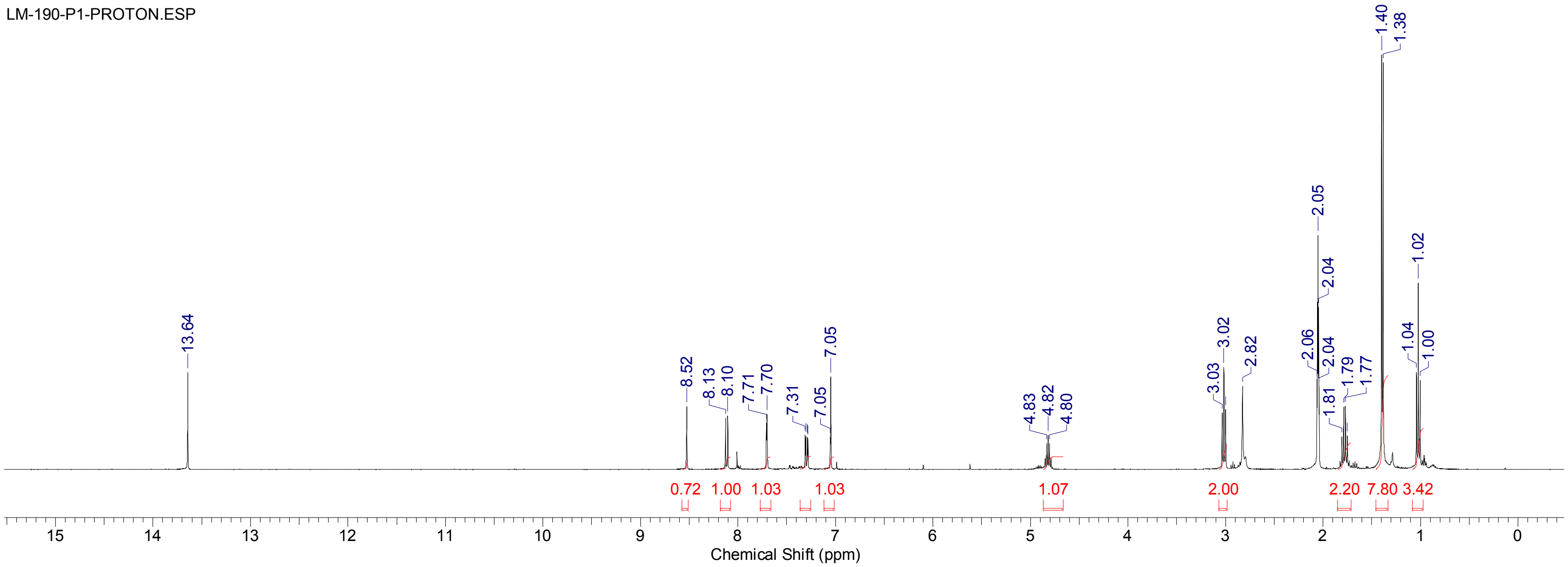

^{1}H NMR at 400 MHz in d$_6$-acetone

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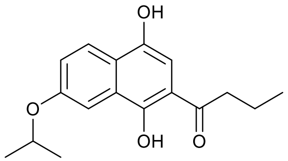
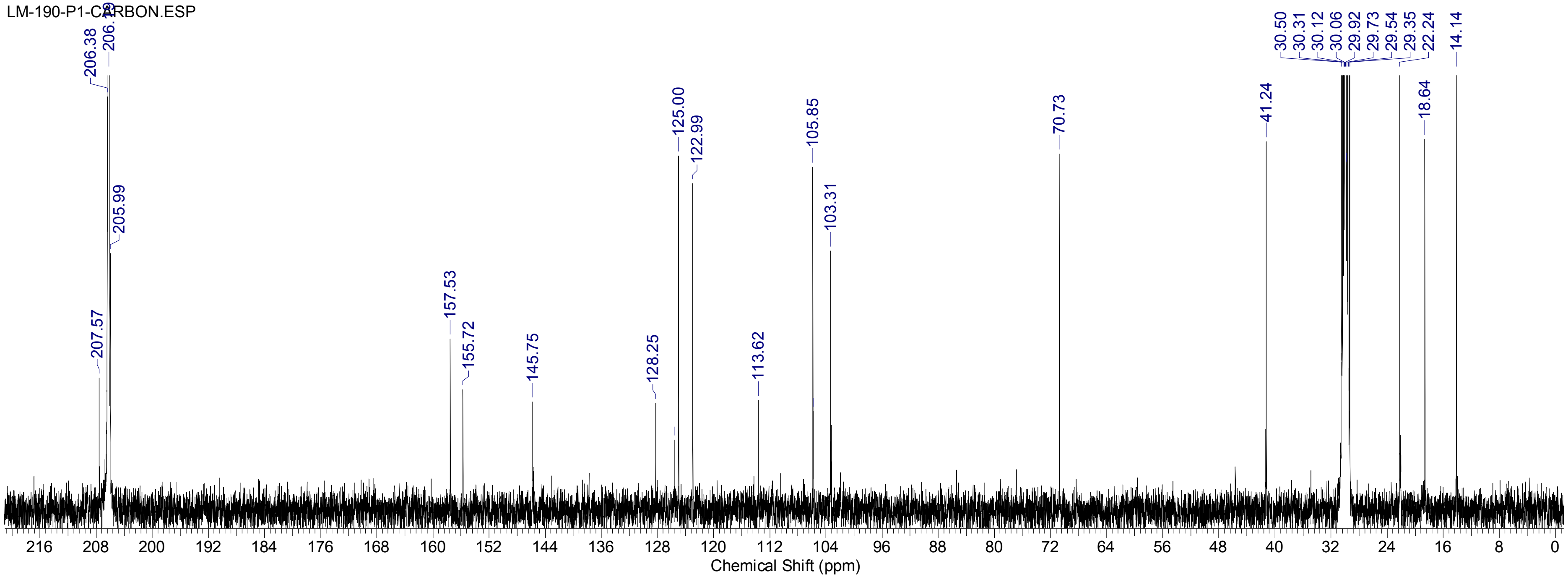
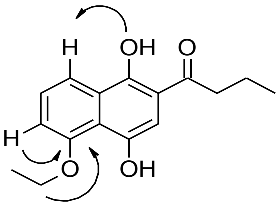
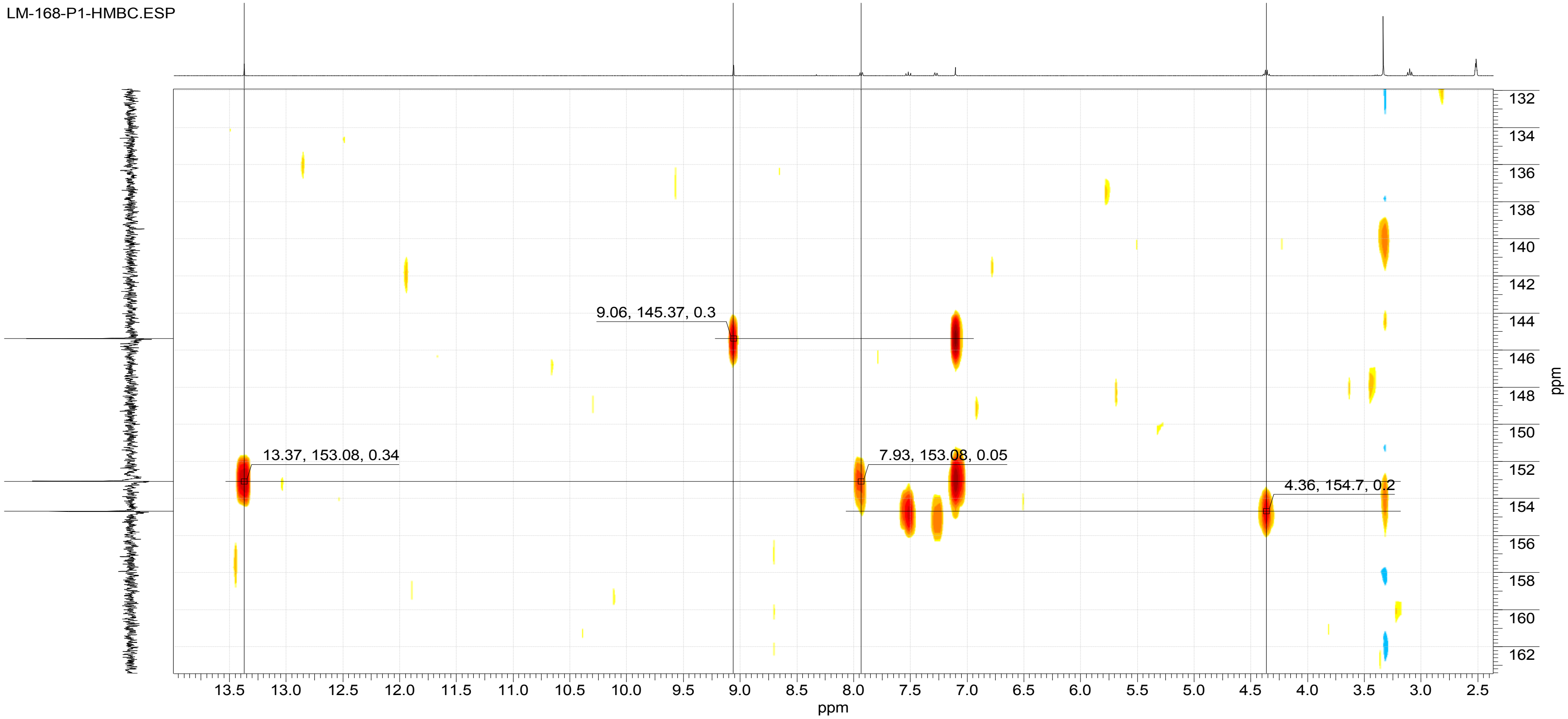




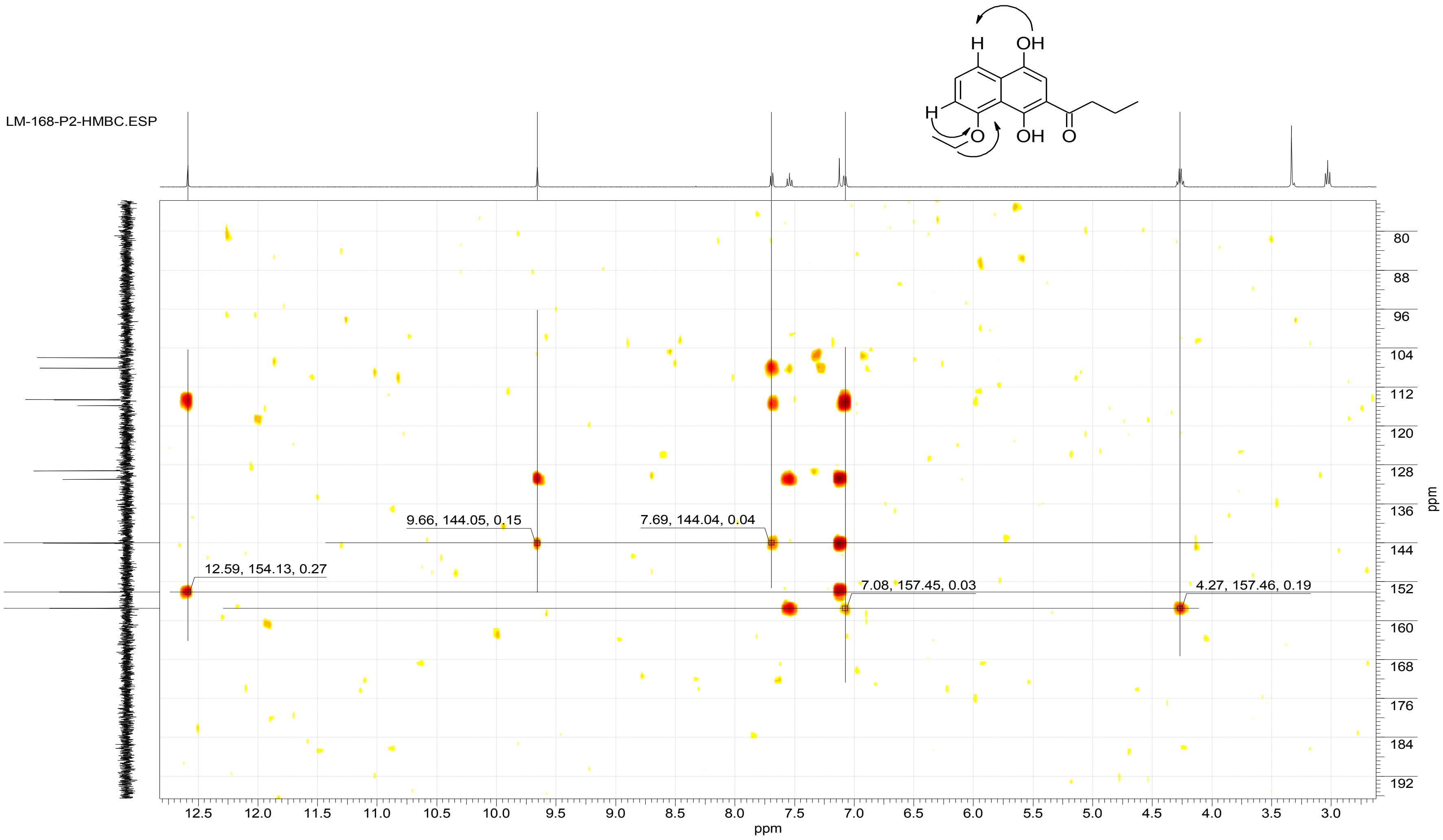
^{13}C NMR at 100 MHz in d_6-acetone

Key HMBC Interactions: 1-(5-Ethoxy-1,4-dihydroxynaphthalen-2-yl)butan-1-one 9c





Key HMBC Interactions: 1-(8-Ethoxy-1,4-dihydroxynaphthalen-2-yl)butan-1-one 10c



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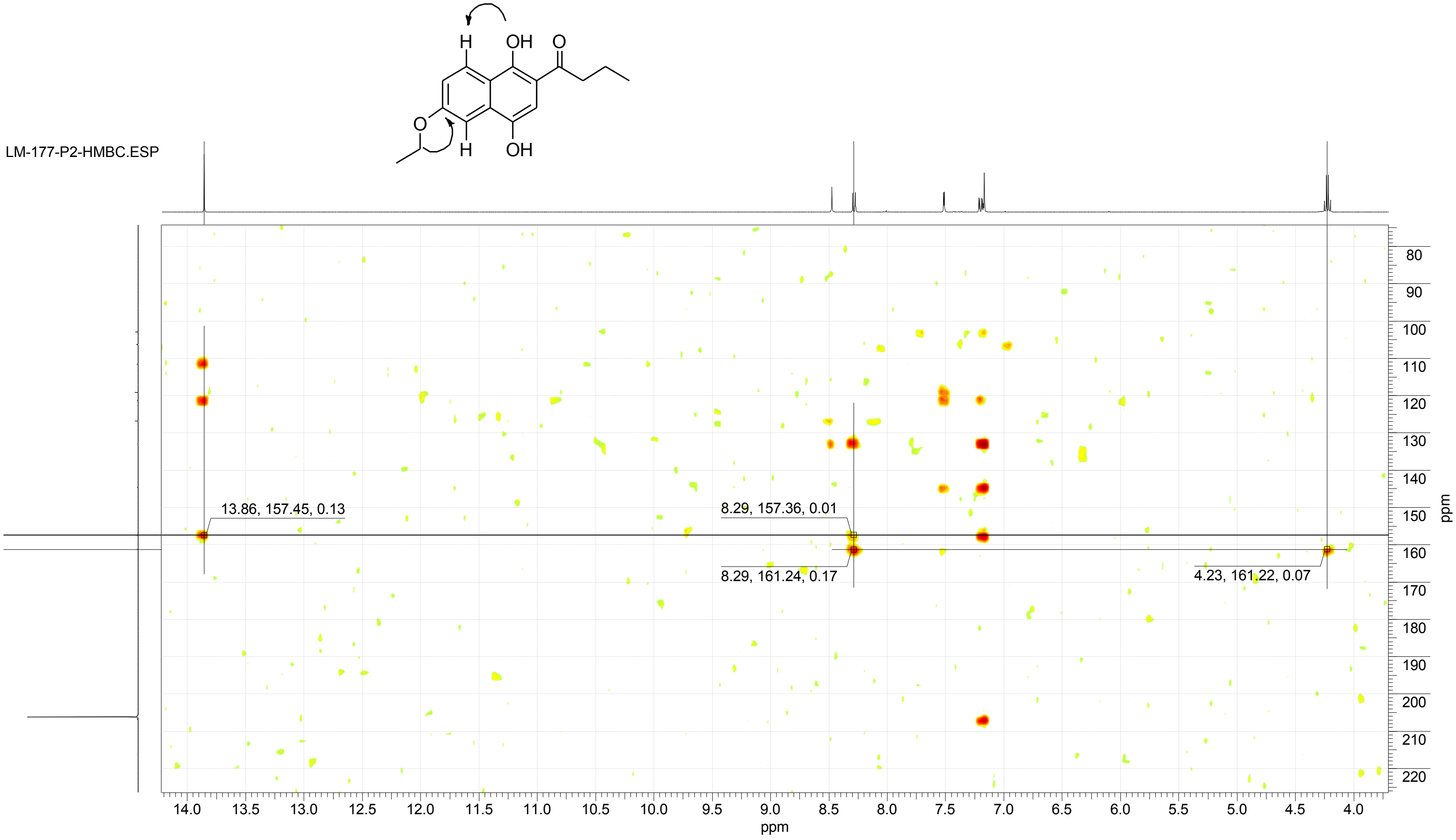

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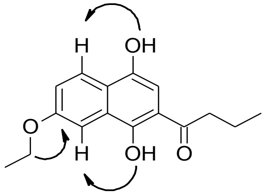
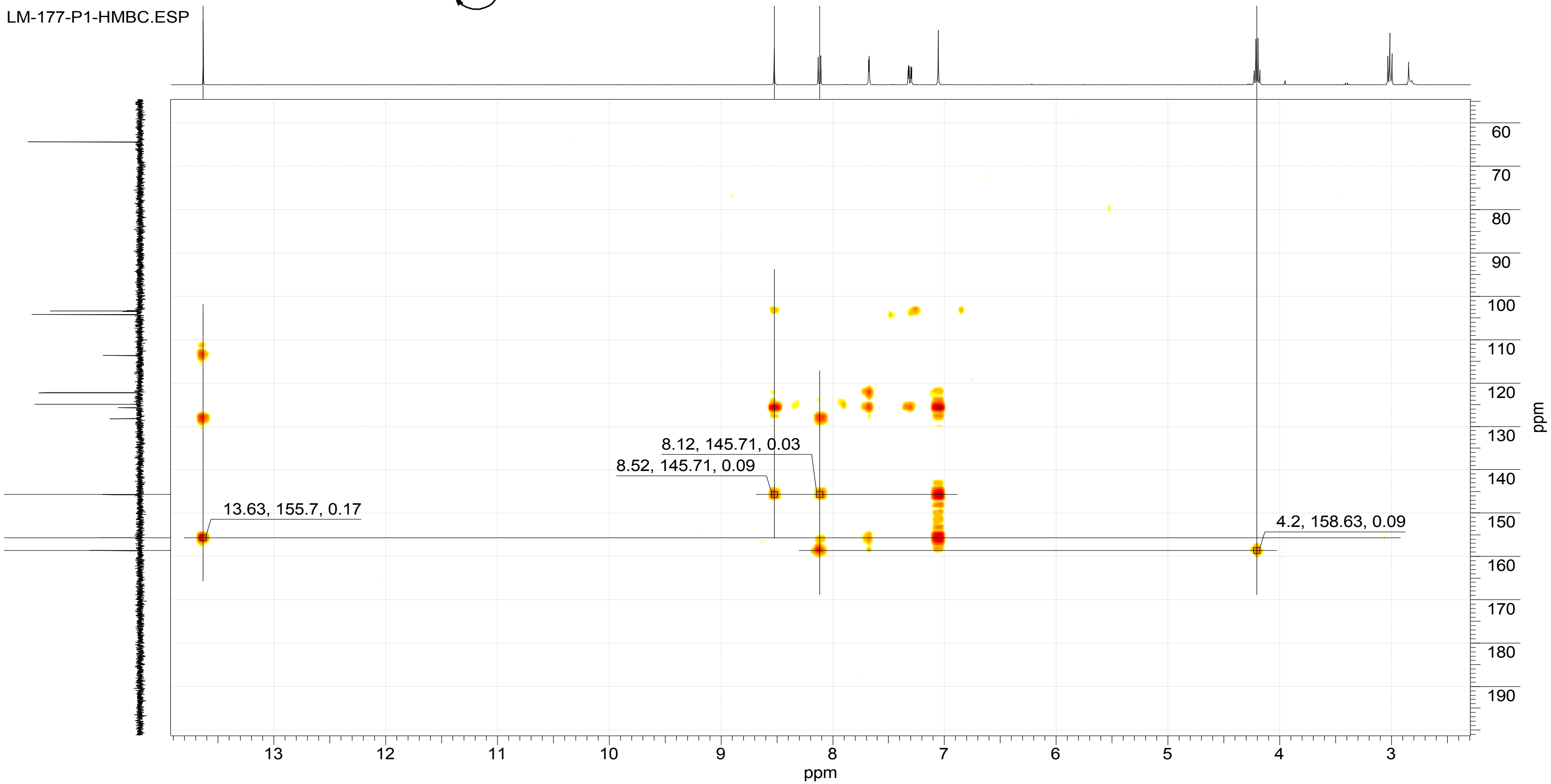
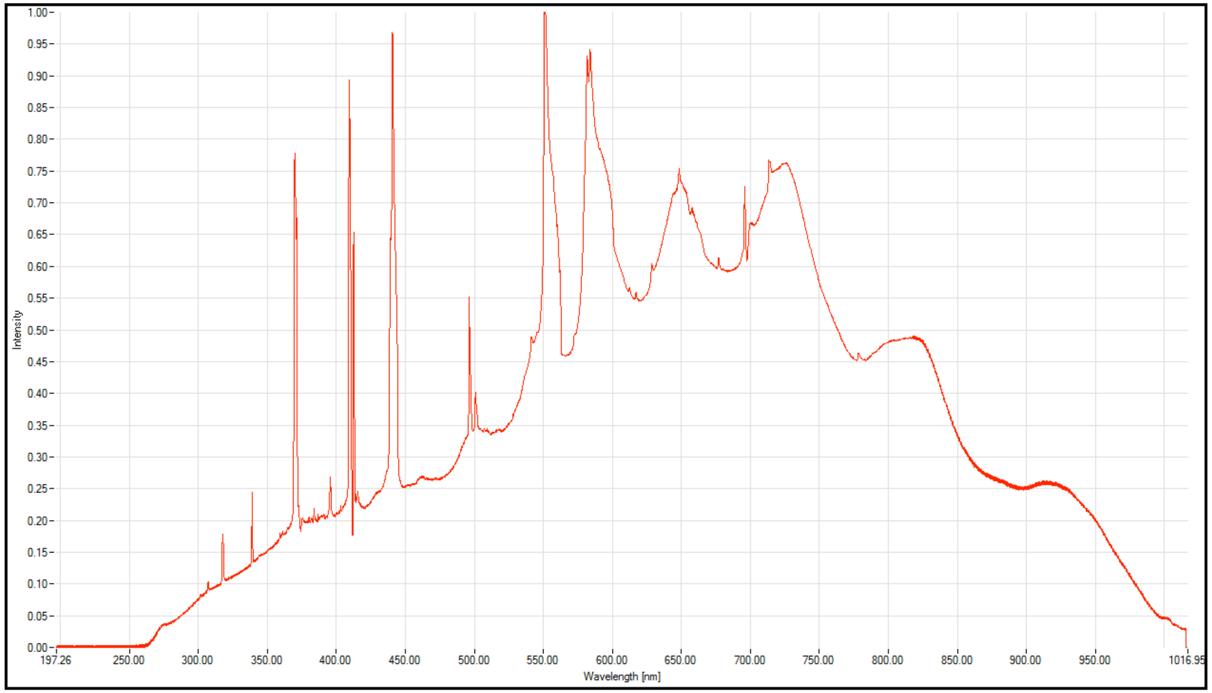




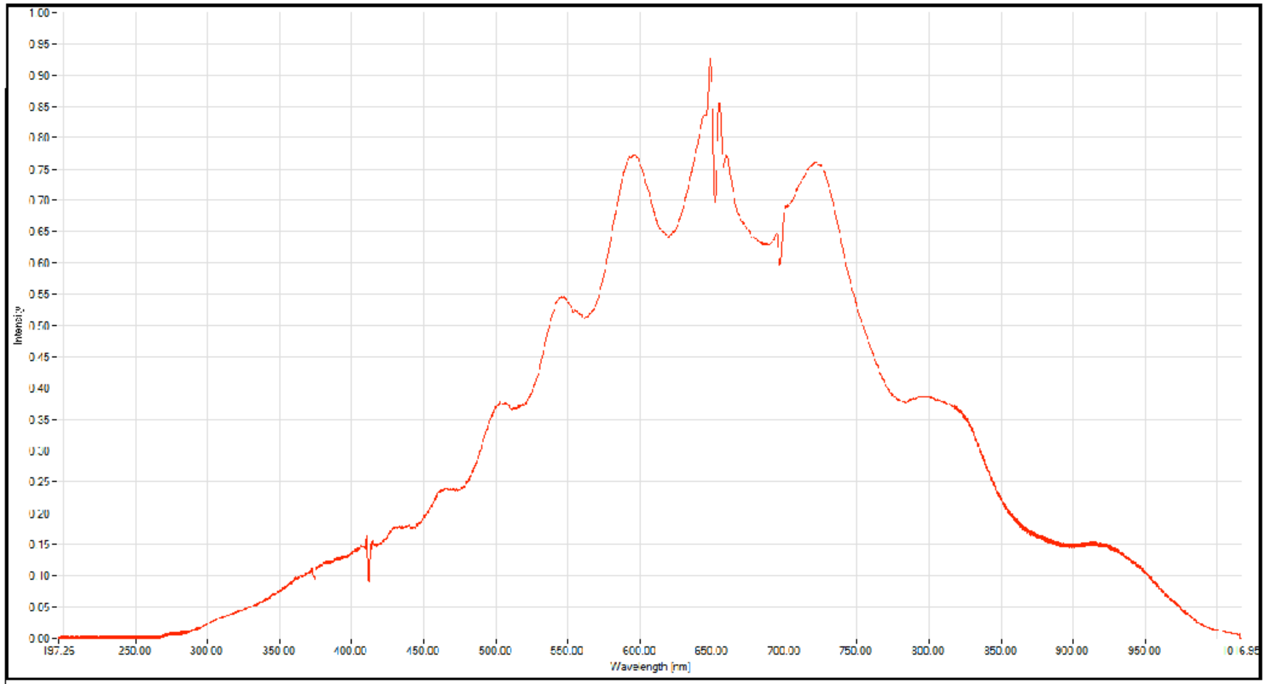
Wavelength Emission Spectra

Wavelength Emission Spectra of the UV Sunlamp (Table 2, Entry 3)



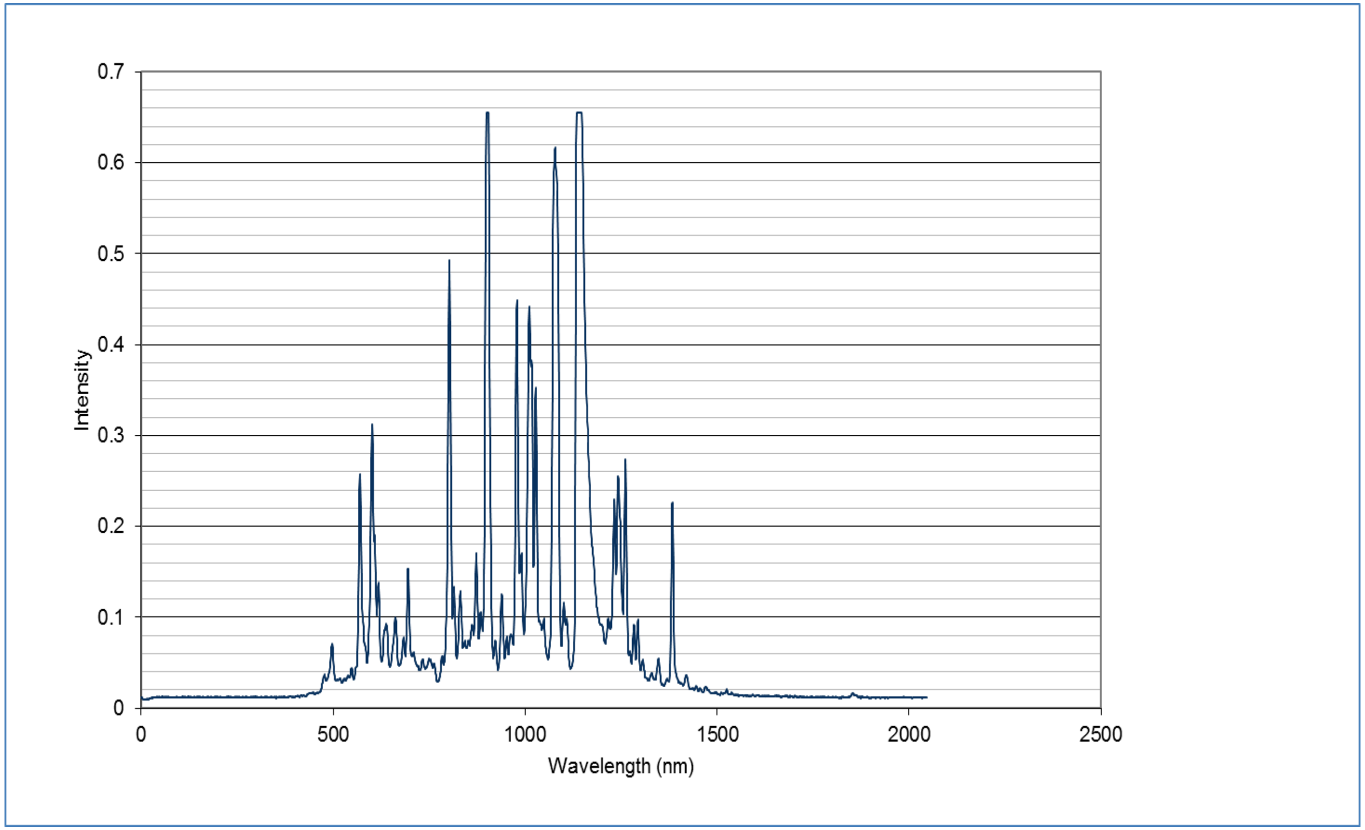
Measured using a compact CCD (Model: CCS150) spectrometer, produced by Thorlabs

Wavelength Emission Spectra of the Halogen Floodlight (Table 2, Entry 4)



Measured using a compact CCD (Model: CCS150) spectrometer, produced by Thorlabs

Wavelength Emission Spectra of the HQI-T HID Light (Table 2, Entry 5)



Measured using a USB Miniature Fiber Optic Spectrometer, produced by Ocean Optics.

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