Electronic Supplementary Information

Diels-Alder trapping of in situ generated Dienes from 3,4-dihydro-2*H*-pyran with *p*-Quinone catalysed by *p*-Toluenesulfonic acid

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Experimental Section

General Experimental Information

All chemicals were purchased from Sigma-Aldrich and freshly distilled 3,4-dihydro-2*H*-pyran was used each on every time. All the solvents were used after distillation. Column chromatography was performed on Silica gel (Merck). ATR-IR spectra were recorded on a Jasco FT/IR-6400 spectrometer with an ATR accessory. NMR spectra were recorded on a Bruker NMR System spectrometer. Chemical shifts were referenced to the residual solvent signal ($CDCl_3$, 99.8 atom % D, δ_H = 7.26, δ_C = 77.00). Mass spectra were recorded on Electrospray Ionisation mass spectroscopy. Compounds 5a, 5b, 6a and 2,5-dichloro-1,4-Bis(tetrahydro-2*H*-pyran-2-yloxy)benzene were crystallized from an acetonitrile at room temperature with slow evaporation method. The colourless crystals were formed within a couple of days and were used for single crystal analysis. X-ray crystallographic data were collected on a Bruker SMART APEX II (Mo radiation) at 173 K in a nitrogen stream. The X-ray condition of was 50 kV 30 mA. Data collection and reduction were done by using the Bruker ApexII software package. The structures were solved by direct methods and refined by full-matrix least-squares on F2 with anisotropic displacement parameters for non-H atoms using SHELX-97.

Reaction procedure - 1: The reaction mixture consisting of 3,4-dihydro-2*H*-pyran (2, 4.5 mmol) and *p*TsOH (15 mol %) in 1,2-dichloroethane (2 ml) was stirred at 83^{o}C for 30 min. Under the oxygen atmosphere, the *p*-quinone (1, 1.0 mmol) in dry 1,2-dichloroethane (1 ml) was added drop wise to the above reaction mixture and the reflux was continued for appropriate time. The progress of the reaction was monitored by TLC (20% ethyl acetate/hexane). After completion, it was concentrated and the residue was subjected for separation of the respective products by column chromatography using silica gel (Elutant: hexane and ethyl acetate).

5,7-bis(3-((tetrahydro-2*H*-pyran-2-yl)oxy)propyl)naphthalene-1,4-dione (3a): Yield: 411 mg, 93%; pale yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ, 7.79 (s, 1H, Ar-H), 7.40 (s, 1H, Ar-H), 6.80 (d, J=3.2 Hz, 2H, HC=CH), 4.55 (t, J=2.8 Hz, 1H, -O/CH/O-), 4.51 (t, J=2.8 Hz, 1H, -O/CH/O-), 3.86-3.80 (m, 3H, -OCH_2-), 3.78-3.70 (m, 1H, -OCH_2-), 3.47-3.43 (m, 3H, -OCH_2-), 3.38-3.33 (m, 1H, -OCH_2-), 3.17-3.13 (m, 2H, Ar-CH_2-), 2.80-2.75 (m, 2H, Ar-CH_2-), 1.94-1.89 (m, 3H, -CH_2-), 1.78-1.76 (m, 2H, -CH_2-), 1.67-1.65 (m, 2H, -CH_2-), 1.52-1.47 (m, 7H, -CH_2-), 1.17 (m, 2H, -CH_2-); ^{13}C NMR (100 MHz, $CDCl_3$): δ, 185.5, 184.7, 146.9, 144.6, 139.6, 136.7,

135.6, 132.7, 126.2, 124.4, 98.06 (-O/CH/O-), 98.03 (-O/CH/O-), 66.3, 66.0, 61.5, 61.3, 32.3, 31.0, 30.4, 29.8, 29.6, 29.4, 28.6, 24.4, 24.3, 18.7; IR (neat): 2951, 1747, 1642, 1209, 1138, 1039 cm^{-1}; HR-MS (ESI, m/z): calcd. for $C_{26}H_{34}O_6$: 442.2355; found: 442.2348.

2-methyl-6,8-bis(3-(tetrahydro-2H-pyran-2-yloxy)propyl) naphthalene -1,4-dione (3b): Yield: 415 mg, 91%; pale yellow liquid; ^1H NMR (400 MHz, $CDCl_3$): δ, 7.75 (s, 1H, Ar-H), 7.30 (s, 1H, Ar-H), 6.67 (s, 1H, RC=CH), 4.53 (s, 1H, -O/CH/O-), 4.49 (s, 1H, -O/CH/O-), 3.80-3.75 (m, 3H, $-OCH_2-$), 3.70-3.68 (m, 1H, $-OCH_2-$), 3.43-3.41 (m, 3H, $-OCH_2-$), 3.33-3.31 (m, 1H, $-OCH_2-$), 3.12 (d, J=4 Hz, 2H, Ar-CH_2-), 2.72 (d, J=4 Hz, 2H, Ar-CH_2-), 2.07 (s, 3H, $-CH_3$), 1.89-1.88 (m, 1H, $-CH_2-$), 1.86-1.82 (m, 2H, $-CH_2-$), 1.79-1.75 (m, 2H, $-CH_2-$), 1.67-1.64 (m, 2H, $-CH_2-$), 1.53-1.46 (m, 9H, $-CH_2-$); ^{13}C NMR (100 MHz, $CDCl_3$): δ, 185.9, 184.5, 148.5, 146.8, 144.4, 136.3, 133.0, 132.9, 126.5, 124.1, 97.89 (-O/CH/O-), 97.86 (-O/CH/O-), 66.1, 65.3, 61.4, 61.3, 31.5, 30.7, 29.8, 29.7, 29.7, 29.5, 24.5, 24.4, 18.7, 18.6, 15.9; IR (neat): 2938, 1739, 1659, 1216, 1127, 1032 cm^{-1}; HR-MS (ESI, m/z): calcd. for $C_{27}H_{36}O_6$: 456.2512; found: 456.2508.

2-chloro-6,8-bis(3-(tetrahydro-2H-pyran-2-yloxy)propyl)naphthalene-1,4-dione (3c): Yield: 423 mg, 89%; pale yellow liquid; ^1H NMR (400 MHz, $CDCl_3$): δ, 7.77 (s, 1H, Ar-H), 7.34 (s, 1H, Ar-H), 6.79 (s, 1H, ClC=CH), 4.53 (s, 1H, -O/CH/O-), 4.49 (s, 1H, -O/CH/O-), 3.84-3.71 (m, 3H, $-OCH_2-$), 3.60-3.57 (m, 2H, $-OCH_2-$), 3.45-3.37 (m, 3H, $-OCH_2-$), 3.17-3.10 (m, 2H, Ar-CH_2-), 2.76-2.72 (m, J=8 Hz, 2H, Ar-CH_2-), 1.87-1.74 (m, 6H, $-CH_2-$), 1.69-1.63 (m, 2H, $-CH_2-$), 1.49-1.45 (m, 6H, $-CH_2-$), 1.17 (m, 2H, $-CH_2-$); ^{13}C NMR (100 MHz, $CDCl_3$): δ, 181.8, 178.7, 148.1, 146.6, 145.2, 136.9, 133.7, 132.4, 125.8, 124.7, 98.07 (-O/CH/O-), 98.03 (-O/CH/O-), 66.0, 65.3, 60.49, 60.45, 32.3, 31.0, 30.4, 29.7, 29.4, 28.6, 24.4, 18.7, 18.6; IR (neat): 2956, 1745, 1647, 1219, 1125, 1043 cm^{-1}; HR-MS (ESI, m/z): calcd. for $C_{26}H_{33}ClO_6$: 476.1966; found: 476.1870.

1,3-bis(3-(tetrahydro-2H-pyran-2-yloxy)propyl)anthracene-9,10-dione (3d): Yield: 418 mg, 85%; greenish yellow liquid; ^1H NMR (400 MHz, $CDCl_3$): δ, 8.15 (d, J=8 Hz, 2H, Ar-H), 8.02 (s, 1H, Ar-H), 7.69-7.65 (m, 2H, Ar-H), 7.38 (s, 1H, Ar-H), 4.55 (t, J=4 Hz, 1H, -O/CH/O-), 4.51 (t, J=4 Hz, 1H, -O/CH/O-), 3.86-3.77 (m, 3H, $-OCH_2-$), 3.75-3.70 (m, 1H, $-OCH_2-$), 3.47-3.40 (m, 3H, $-OCH_2-$), 3.38-3.32 (m, 1H, $-OCH_2-$), 3.26-3.21 (m, 2H, Ar-CH_2-), 2.79-2.74 (m, 2H, Ar-CH_2-), 1.95-1.87 (m, 4H, $-CH_2-$), 1.80-1.77 (m, 2H, $-CH_2-$), 1.69-1.63 (m, 2H, $-CH_2-$), 1.54-1.52 (m, 2H, $-CH_2-$), 1.47-1.46 (m, 4H, $-CH_2-$), 1.17 (s, 2H, $-CH_2-$); ^{13}C NMR (100 MHz, $CDCl_3$): δ, 183.4, 182.8, 147.1, 145.2, 136.9, 134.3, 133.9, 133.0, 132.2, 131.7, 127.9, 126.1, 125.5, 125.1, 97.9

(-O/CH/O-), 97.8 (-O/CH/O-), 66.1, 65.4, 61.4, 61.3, 31.5, 31.2, 29.8, 29.7, 28.6, 24.5, 24.4, 18.7, 18.6; IR (neat): 2947, 1667, 1590, 1279, 1126, 1027 cm^{-1}; HR-MS (ESI, m/z): calcd. for $C_{30}H_{36}O_6Na$ [M + Na]: 515.2410; found: 515.2381.

(6S,6aS,10aS,10bR)-8,10a-dimethyl-6-(3-(((S)-tetrahydro-2H-pyran-2-yl)oxy)propyl)-3,4,6,6a-tetrahydro-2H-benzo[h]chromene-7,10(10aH,10bH)-dione (5a): Yield: 368 mg, 95%; M.P: 82–84^oC; colourless solid; ^1H NMR (500 MHz, $CDCl_3$): δ, 6.43 (s, 1H, CH_3-C=CH-), 5.50 (s, 1H, HC=CR), 4.56 (t, 1H, J=2, -O/CH/O-), 3.88-3.84 (m, 1H, -OCH_2-), 3.80-3.74 (m, 2H, -OCH_2-), 3.51-3.46 (m, 1H, -OCH_2-), 3.44-3.40 (m, 2H, -OCH_2-), 3.38-3.33 (dt, J=3 Hz, J=12 Hz, 1H, -O/CH/C-), 2.87 (d, J=4 Hz, 1H, -C/CH/C-), 2.30-2.26 (m, 2H, -CH_2-), 2.15-2.10 (m, 1H, -C/CH/CH_2–), 2.04-1.97 (m, 1H, -CH_2-), 1.94 (s, 3H, -CH_3), 1.90-1.81 (m, 2H, -CH_2-), 1.73-1.69 (m, 2H, -CH_2-), 1.64-1.63 (m, 1H, -CH_2-), 1.59-1.50 (m, 6H, -CH_2-), 1.32 (s, 3H, -CH_3); ^{13}C NMR (125 MHz, $CDCl_3$): δ, 200.8, 198.5, 152.6, 136.4, 133.1, 123.4, 98.9 (-O/CH/O-), 80.5 (-O/CH/C-), 69.1, 67.5, 62.4, 53.1, 53.1, 33.4, 33.4, 30.7, 29.5, 28.4, 28.3, 25.4, 20.0, 19.7, 16.2; IR (neat): 2937, 2859, 1737, 1670, 1124, 1066, 1025 cm^{-1}; HR-MS (ESI, m/z): calcd. for $C_{23}H_{32}O_5Na$ [M + Na]: 411.2147; found: 411.2083.

(6S,6aS,12aS,12bR)-12a-methyl-6-(3-(((S)-tetrahydro-2H-pyran-2-yl)oxy)propyl)-3,4,6,6a-tetrahydro-2H-naphtho[2,3-h]chromene-7,12(12aH,12bH)-dione (5b): Yield: 386 mg, 91%; M.P: 142–146^oC; colourless solid; ^1H NMR (400 MHz, $CDCl_3$): δ, 8.05-8.03 (m, 1H, Ar-H), 7.79-7.76 (m, 1H, Ar-H), 7.64-7.61 (m, 2H, Ar-H), 5.59 (s, 1H, -HC=CR), 4.57 (s, 1H, -O/CH/O-), 3.88-3.76 (m, 2H, -OCH_2-), 3.55-3.47 (m, 2H, -OCH_2-), 3.45-3.42 (m, 2H, -OCH_2-), 3.33-3.25 (dt, J=4, J=16, 1H, -O/CH/C-), 3.11-3.10 (d, J=4 Hz, 1H, -C/CH/C-), 2.41 (m, 1H, -CH_2-), 2.33-2.29 (m, 1H, -C/CH/CH_2-), 2.14-2.09 (m, 2H, -CH_2-), 2.01-1.95 (m, 1H, -CH_2-), 1.80-1.70 (m, 4H, -CH_2-) 1.61-1.53 (m, 6H, -CH_2-) 1.46 (s, 3H, -CH_3); ^{13}C NMR (100 MHz, $CDCl_3$): δ, 199.3, 196.9, 138.7, 135.0, 133.3, 133.1, 132.5, 126.3, 125.0, 123.5, 98.9 (-O/CH/O-), 80.8 (-O/CH/C-), 69.0, 67.5, 62.4, 53.2, 53.0, 33.7, 33.3, 30.7, 29.3, 28.6, 28.3, 25.3, 20.0, 19.6; IR (neat): 2944, 2851, 1691, 1595, 1134, 1065, 1023 cm^{-1}; HR-MS (ESI, m/z): calcd. for $C_{26}H_{32}O_5Na$ [M + Na]: 447.2147; found: 447.2130.

(6S,6aR,12aR,12bR)-12a-bromo-6-(3-(((S)-tetrahydro-2H-pyran-2-yl)oxy)propyl)-3,4,6,6a-tetrahydro-2H-naphtho[2,3-h]chromene-7,12(12aH,12bH)-dione (5c): Yield: 434 mg, 89%; M.P: 118–122^oC; yellow solid; ^1H NMR (400 MHz, $CDCl_3$): δ, 8.12 (d, J=2 Hz, 1H, Ar-H), 7.82-7.80 (m, 1H, Ar-H), 7.69-7.67 (m, 2H, Ar-H), 5.69 (s, 1H, HC=CR), 4.57-4.56 (m, 1H, -O/CH/O-),

4.31 (s, 1H, -O/CH/C-), 3.86-3.80 (m, 1H, -OCH_2-), 3.79-3.68 (m, 1H, -OCH_2-), 3.63-3.60 (m, 1H, -OCH_2-), 3.55-3.39 (m, 3H, -OCH_2-), 2.95 (m, 1H, -C/CH/C-), 2.37-2.34 (m, 1H, -C/CH/CH_2-), 2.25-2.18 (m, 1H, -CH_2-), 2.09-2.06 (m, 1H, -CH_2-), 1.98-1.92 (m, 2H, -CH_2-), 1.77-1.72 (m, 4H, -CH_2-), 1.56-1.52 (m, 6H, -CH_2-); ^{13}C NMR (125 MHz, $CDCl_3$): δ, 192.8, 191.2, 137.9, 134.0, 133.8, 133.2, 131.3, 127.5, 125.6, 124.3, 98.9 (-O/CH/O-), 81.8 (-O/CH/C-), 70.4, 69.5, 67.3, 62.3, 56.7 (-C/CH/C-), 36.1 (-C/CH/CH_2-), 32.9, 30.7, 29.1, 28.5, 28.2, 25.4, 19.6; IR (neat): 2943, 2852, 1662, 1592, 1122, 1069, 1024 cm^{-1}; HR-MS (ESI, m/z): calcd. for $C_{25}H_{29}BrO_5Na$ [M + Na]: 511.1096; found: 511.1092.

Reaction procedure - 2: A solution of *p*-quinone (1, 1.0 mmol) and *p*TsOH (15 mol %) in dry acetonitrile (2 ml) was stirred at 81°C under oxygen atmosphere. To the above reaction mixture, 3,4-dihydro-2*H*-pyran (2, 4.5 mmol) in dry acetonitrile (1 ml) was added drop wise and the same was refluxed for appropriate time. The progress of the reaction was monitored by TLC (20% ethyl acetate/hexane). After completion, it was concentrated and the residue was subjected for separation of the respective products by column chromatography using silica gel (Elutant: hexane and ethyl acetate).

2-(tetrahydro-2H-pyran-2-yl)-5,7-bis(3-(tetrahydro-2H-pyran-2-yloxy)propyl)naphthalene-1,4-dione (4a): Yield: 494 mg, 94%; pale yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ, 7.77 (s, 1H, Ar-H), 7.30 (s, 1H, Ar-H), 6.89 (s, 1H, CH=CR), 4.53 (s, 1H, -O/CH/O-), 4.50 (s, 1H, -O/CH/O-), 4.47-4.44 (d, J=12 Hz, 1H, -O/CH/CH_2-), 4.06 (d, J=8 Hz, 1H, -OCH_2-), 3.81-3.76 (m, 3H, -OCH_2-), 3.71-3.69 (m, 1H, -OCH_2-), 3.53 (t, J=12 Hz, 1H, -OCH_2-), 3.43-3.41 (m, 3H, -OCH_2-), 3.33-3.31 (m, 1H, -OCH_2-), 3.11 (m, 2H, Ar-CH_2-), 2.73-2.72 (m, 2H, Ar-CH_2-), 1.95-1.92 (m, 1H, -CH_2-), 1.90-1.82 (m, 3H, -CH_2-), 1.68-1.59 (m, 4H, -CH_2-), 1.52-1.47 (m, 9H, -CH_2-), 1.21-1.18 (m, 5H, -CH_2-); ^{13}C NMR (100 MHz, $CDCl_3$): δ, 184.9, 184.8, 152.2, 146.9, 144.4, 136.4, 132.8, 130.5, 126.6, 124.2, 97.95 (-O/CH/O-), 97.90 (-O/CH/O-), 72.6 (-O/CH/CH_2-), 67.8, 66.1, 65.4, 61.4, 61.3, 32.1, 31.5, 30.7, 29.8, 29.7, 29.5, 28.6, 24.8, 24.5, 24.4, 22.7, 18.7, 18.6; IR (neat): 2893, 2849, 1652, 1621, 1122, 1073 cm^{-1}; HR-MS (ESI, m/z): calcd. for $C_{31}H_{42}O_7$: 526.2931; found: 526.2928.

3-methyl-2-(tetrahydro-2H-pyran-2-yl)-5,7-bis(3-((tetrahydro-2H-pyran-2-yl)oxy)propyl)naphthalene-1,4-dione (4b): Yield: 470 mg, 87%; pale yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ, 7.83 (s, 1H, Ar-H), 7.35 (s, 1H, Ar-H), 4.92 (d, J=8.4 Hz, 1H, -O/CH/CH_2-), 4.61 (t, J=2.4 Hz, 1H, -O/CH/O-), 4.58 (s, 1H, -O/CH/O-), 4.12-4.10 (m, 1H, -OCH_2-), 3.88-3.84

(m, 3H, -OCH_2-), 3.78-3.76 (m, 1H, -OCH_2-), 3.52-3.49 (m, 4H, -OCH_2-), 3.41-3.39 (m, 1H, -OCH_2-), 3.20-3.16 (m, 2H, Ar-CH_2-), 2.81-2.77 (m, 2H, Ar-CH_2-), 2.38 (s, 3H, -CH_3), 1.97-1.90 (m, 7H, -CH_2-), 1.73-1.70 (m, 6H, -CH_2-), 1.60-1.59 (m, 9H, -CH_2-); ^{13}C NMR (100 MHz, $CDCl_3$): δ, 187.4, 184.7, 147.5, 146.7, 144.9, 143.4, 136.9, 133.9, 127.5, 125.2, 98.94 (-O/CH/O-), 98.90 (-O/CH/O-), 74.1 (-O/CH/CH_2-), 69.0, 67.1, 66.4, 62.4, 62.3, 32.5, 31.6, 30.8, 30.7, 30.6, 29.8, 29.6, 25.8, 25.5, 25.4, 23.7, 19.7, 19.6, 13.4; IR (neat): 2874, 2834, 1659, 1631, 1137, 1067 cm^{-1}; HR-MS (ESI, m/z): calcd. for $C_{32}H_{44}O_7$: 540.3087; found: 540.3076.

3-chloro-2-(tetrahydro-2H-pyran-2-yl)-5,7-bis(3-((tetrahydro-2H-pyran-2-yl)oxy)propyl)naphthalene-1,4-dione (4c): Yield: 498 mg, 89%; pale yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ, 7.86 (s, 1H, Ar-H), 7.39 (s, 1H, Ar-H), 4.92 (dd, J=1.6 Hz, J=11.6 Hz, 1H, -O/CH/CH_2-), 4.59 (m, 1H, -O/CH/O-), 4.57 (m, 1H, -O/CH/O-), 4.15-4.11 (m, 1H, -OCH_2-), 3.88-3.81 (m, 3H, -OCH_2-), 3.78-3.75 (m, 1H, -OCH_2-), 3.55-3.49 (m, 4H, -OCH_2-), 3.41-3.38 (m, 1H, -OCH_2-), 3.20-3.17 (m, 2H, Ar-CH_2-), 2.81-2.80 (m, 2H, Ar-CH_2-),1.96-1.93 (m, 5H, -CH_2-), 1.91-1.71 (m, 7H, -CH_2-), 1.70-1.60 (m, 3H, -CH_2-), 1.59-1.57 (m, 4H, -CH_2-), 1.55-1.25 (m, 3H, -CH_2-); ^{13}C NMR (100 MHz, $CDCl_3$): δ, 182.3, 178.7, 148.7, 146.2, 144.8, 143.6, 137.4, 133.9, 126.3, 126.1, 98.98 (-O/CH/O-), 98.96 (-O/CH/O-), 75.4 (-O/CH/CH_2-), 69.1, 67.0, 66.3, 62.46, 62.41, 32.5, 31.8, 30.8, 30.7, 30.6, 30.4, 28.0, 25.49, 25.45, 24.6, 23.6, 19.7, 19.6; IR (neat): 2873, 2857, 1638, 1629, 1136, 1059 cm^{-1}; HR-MS (ESI, m/z): calcd. for $C_{31}H_{41}ClO_7$: 560.2541; found: 560.2493.

Reaction procedure - 3: A solution of *p*-quinone (1, 1.0 mmol) and *p*TsOH (15 mol %) in 1,2-dichloroethane (2 ml) was stirred at 83^{o}C under oxygen atmosphere. To the above reaction mixture, 3,4-dihydro-2*H*-pyran (2, 4.5 mmol) in dry 1,2-dichloroethane (1 ml) was added drop wise and the same was refluxed for appropriate time. The progress of the reaction was monitored by TLC (20% ethyl acetate/hexane). After completion, it was concentrated and the residue was subjected for separation of the respective products by column chromatography using silica gel (Elutant: hexane and ethyl acetate).

(3R,3aS,3a1R,6aS,7aS)-3a1,5-dichloro-3,3a,7a,9,10,11,11a,11b-octahydro-6a,3-(epoxymethano)benzo[de]pyrano[2,3-b]chromen-4(3a1H)-one (6a): Yield: 311 mg, 91%; M.P: 182-186^{o}C; colourless solid; ^{1}H NMR (500 MHz, $CDCl_3$): δ, 6.74 (s, 1H, ClC=CH), 5.86-5.82 (m, J=3 Hz, 1H, -HC=CH-), 5.79 (d, J=3 Hz, 1H, -HC=CH-), 5.76 (t, J=3.5 Hz, 1H, -O/CH/O-), 3.94-3.89 (dt, J_1=3 Hz, J_2=11 Hz, 1H, -OCH_2-), 3.79-3.73 (m, 2H, -OCH_2-), 3.64-3.61

(dt, J_1=1 Hz, J_2=12.5 Hz, 1H, -OCH_2-), 3.33-3.32 (dd, J_1=1 Hz, J_2=4 Hz, 1H, -CH-), 3.03 (d, J=2 Hz, 1H, -CH-), 2.70-2.67 (m, 1H, -CH_2-), 2.60-2.58 (m, 1H, -CH-), 2.08-2.04 (m, 1H, -CH-), 1.75-1.68 (m, 3H, -CH_2-); ^{13}C NMR (125 MHz, $CDCl_3$): δ, 190.3, 137.5, 133.7, 132.7, 125.5, 99.7 (-O/C/O-), 92.6 (-O/CH/O-), 66.5, 62.8, 62.3, 56.2, 47.9, 37.9, 37.6, 26.3, 25.3; IR (neat): 2964, 2881, 1739, 1220 cm^{-1}; HR-MS (ESI, m/z): calcd. for $C_{16}H_{16}Cl_2O_4$: 342.0426; found: 342.0416.

(3R,3aS,3a1R,6aS,7aS)-3a1,5-dibromo-3,3a,7a,9,10,11,11a,11b-octahydro-6a,3-(epoxymethano)benzo [de]pyrano[2,3-b]chromen-4(3a1H)-one (6b): Yield: 374 mg, 87%; brownish semi-solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ, 6.91 (s, 1H, BrC=CH), 5.93-5.90 (m, 1H, -HC=CH-), 5.76 (s, 1H, -HC=CH-), 5.73 (s, 1H, -O/CH/O-), 3.93-3.88 (m, J=4 Hz, 1H, -OCH_2-), 3.76-3.72 (m, 2H, -OCH_2-), 3.63-3.60 (m, 1H, -OCH_2-), 3.31-3.30 (d, J=4 Hz, 1H, -CH-), 3.01 (s, 1H, -CH-), 2.48-2.44 (m, 1H, -CH_2-), 2.38 (m, 1H, -CH-), 1.82-1.79 (d, J=12 Hz, 1H, -CH-), 1.52-1.49 (m, 3H, -CH_2-); ^{13}C NMR (100 MHz, $CDCl_3$): δ, 187.8, 141.1, 134.5, 132.8, 125.5, 97.8 (-O/C/O-), 91.7 (-O/CH/O-), 66.5, 62.8, 61.7, 54.2, 48.0, 37.9, 37.6, 25.5, 24.3; IR (neat): 2936, 2864, 1667, 1227 cm^{-1}; HR-MS (ESI, m/z): calcd. for $C_{16}H_{16}Br_2O_4$: 429.9415; found: 429.9371.

Reaction procedure – 4 (Stoichiometric condition): A solution of *p*-quinone (1, 2.0 mmol), 3,4-dihydro-2*H*-pyran (2, 5.0 mmol) and *p*TsOH (1.0 mmol) in 1,2-dichloroethane (5 ml) was stirred at 83^{o}C under oxygen atmosphere. The progress of the reaction was monitored by TLC (20% ethyl acetate/hexane). After completion, it was concentrated and the residue was subjected for separation of the respective products by column chromatography using silica gel (Elutant: hexane and ethyl acetate).

2,5-dichloro-1,4-Bis(tetrahydro-2H-pyran-2-yloxy)benzene: Yield: 128 mg, 37%; M.P: 126–128^{o}C; Colorless solid; ^{1}H NMR (500 MHz, $CDCl_3$): δ, 7.24 (s, 2H, Ar–H), 5.37 (t, 2H, –O–CH–O–), 3.96-3.91 (m, 2H, –OCH_2–), 3.67-3.63 (m, 2H, –OCH_2–), 2.08-2.03 (m, 2H, –CH_2–), 1.99-1.95 (m, 2H, –CH_2–), 1.91-1.85 (m, 2H, –CH_2–), 1.75–1.62 (m, 6H, –CH_2–); ^{13}C NMR (125 MHz, $CDCl_3$): δ, 147.5, 122.6, 119.0, 97.8 (–O–CH–O–), 61.9, 30.1, 25.1, 18.3; IR (neat): 2954, 1078, 1019 cm^{-1}; HR-MS (ESI, m/z): calculated for $C_{16}H_{20}Cl_2O_4H^+$ [M + H]: 347.0817; found: 347.0833.

1,4-Bis(tetrahydro-2H-pyran-2-yloxy)benzene: Yield: 78 mg, 28%; M.P: 120–122^{o}C; Colorless solid; ^{1}H NMR (300 MHz, $CDCl_3$): δ, 6.97 (s, 4H, Ar–H), 5.30 (t, J=3 Hz, 2H, –O–CH–O–), 3.97-3.89 (m, 2H, –OCH_2–), 3.62-3.55 (m, 2H, –OCH_2–), 2.04-1.92 (m, 2H, –CH_2–), 1.85-1.81 (m, 4H, –CH_2–), 1.69-1.56 (m, 6H, –CH_2–); ^{13}C NMR (75 MHz, $CDCl_3$): δ, 151.9, 117.5,

97.2 (–O–CH–O–), 62.0, 30.5, 25.2, 18.9; IR (neat): 2946, 1104, 1022 cm^{-1}; HR-MS (ESI, m/z): calcd. for $C_{16}H_{22}O_4Na$ [M + Na]: 301.1416; found: 301.1403.

2-(tetrahydro-2H-pyran-2-yl)naphthalene-1,4-dione: Yield: 61 mg, 25%; M.P: 70 – 72^oC; light brown solid; ^1H NMR (400 MHz, $CDCl_3$): δ, 8.07-8.04 (m, 2H, Ar–H), 7.72-7.71 (m, 2H, Ar–H), 7.05 (d, J=4 Hz, 1H, RC=CH), 4.54-4.51 (dt, J_1=1.6 Hz, J_2=8.8 Hz, 1H, –O/CH/CH_2–), 4.15-4.12 (m, 1H, –OCH_2–), 3.63-3.58 (td, J_1=2.4 Hz, J_2=9.2 Hz, 1H, –OCH_2–), 2.05-2.02 (m, 1H, –CH_2–), 1.92-1.89 (m, 1H, –CH_2–), 1.73–1.57 (m, 3H, –CH_2–), 1.31–1.23 (m, 1H, –CH_2–); ^{13}C NMR (100 MHz, $CDCl_3$): δ, 185.2, 184.2, 151.8, 133.7, 133.5, 133.2, 132.2, 131.9, 126.3, 126.0, 73.3 (–O/CH/CH_2–), 68.8 (–OCH_2–), 33.0, 25.7, 23.6; IR (neat): 2943, 1743, 1657, 1298, 1086, 1050 cm^{-1}; HR-MS (ESI, m/z): calcd. for $C_{15}H_{14}O_3$: 242.0943; found: 242.0881.

2,3-bis(tetrahydro-2H-pyran-2-yl)naphthalene-1,4-dione: Yield: 55 mg, 17%; M.P: 168 – 170^oC; pale yellow solid; ^1H NMR (400 MHz, $CDCl_3$): δ, 8.04-8.03 (m, 2H, Ar–H), 7.69-7.67 (m, 2H, Ar–H), 5.23-5.21 (dd, J_1=1.6 Hz, J_2=9.2 Hz, 2H, –O/CH/CH_2–), 4.18-4.15 (m, 2H, –OCH_2–), 3.59-3.53 (td, J=1.6 Hz, J=9.6 Hz, 2H, –OCH_2–), 2.03-1.96 (m, 4H, –CH_2–), 1.80–1.75 (m, 2H, –CH_2–), 1.69-1.62 (m, 6H, –CH_2–); ^{13}C NMR (100 MHz, $CDCl_3$): δ, 184.9, 145.9, 133.5, 132.0, 126.1, 74.8 (–O/CH/CH_2–), 69.1 (–OCH_2–), 30.9, 25.8, 24.1; IR (neat): 2929, 1750, 1663, 1285, 1082, 1038 cm^{-1}; HR-MS (ESI, m/z): calcd. for $C_{20}H_{22}O_4$: 326.1518; found: 326.1512.

Diene 1: A mixture of 3,4-dihydro-2*H*-pyran and *p*TsOH in Deuterochloroform ($CDCl_3$) was stirred for few minutes and the mixture was monitored by NMR analytical techniques. ^1H NMR (400 MHz, $CDCl_3$): δ, 6.20 (s, 1H, O–CH=C), 6.11-6.09 (d, 1H, CH=CH), 5.60-5.57 (m, 1H, CH=CH), 4.87-4.79 (m, 1H, –O/CH/O–), 3.78-3.31 (m, 6H, –OCH_2–), 1.75-1.45 (m, 14H, –CH_2–); ^{13}C NMR (100 MHz, $CDCl_3$): δ, 132.9, 131.6, 126.2, 98.9, 67.3, 66.8, 62.3, 32.1, 30.7, 29.6, 27.8, 27.6, 25.4, 23.4, 19.6.

NMR Spectra

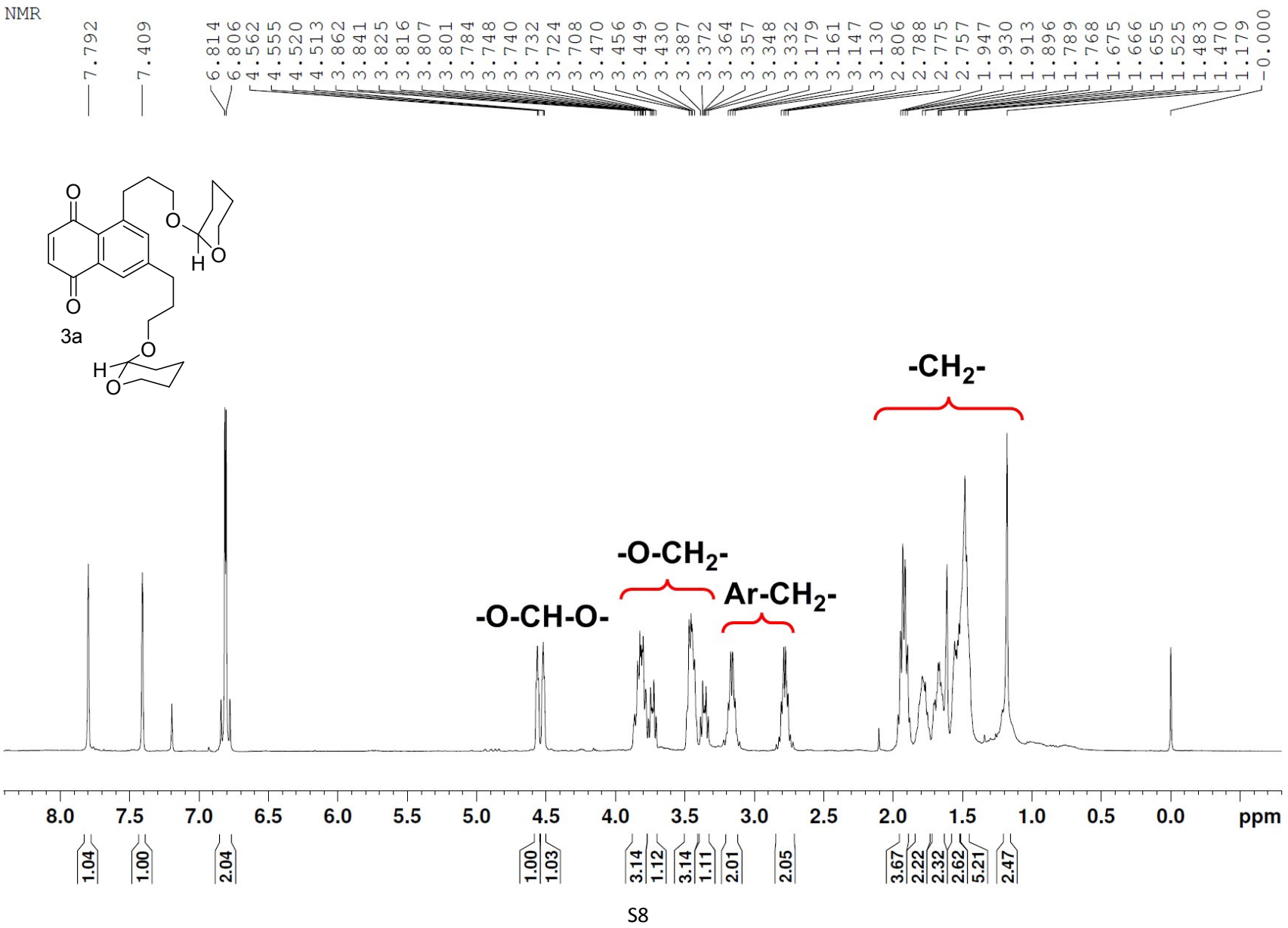


Figure 1. ^{1}H NMR (400 MHz, $CDCl_3$, 300K) of **3a** (TMS added as internal standard)

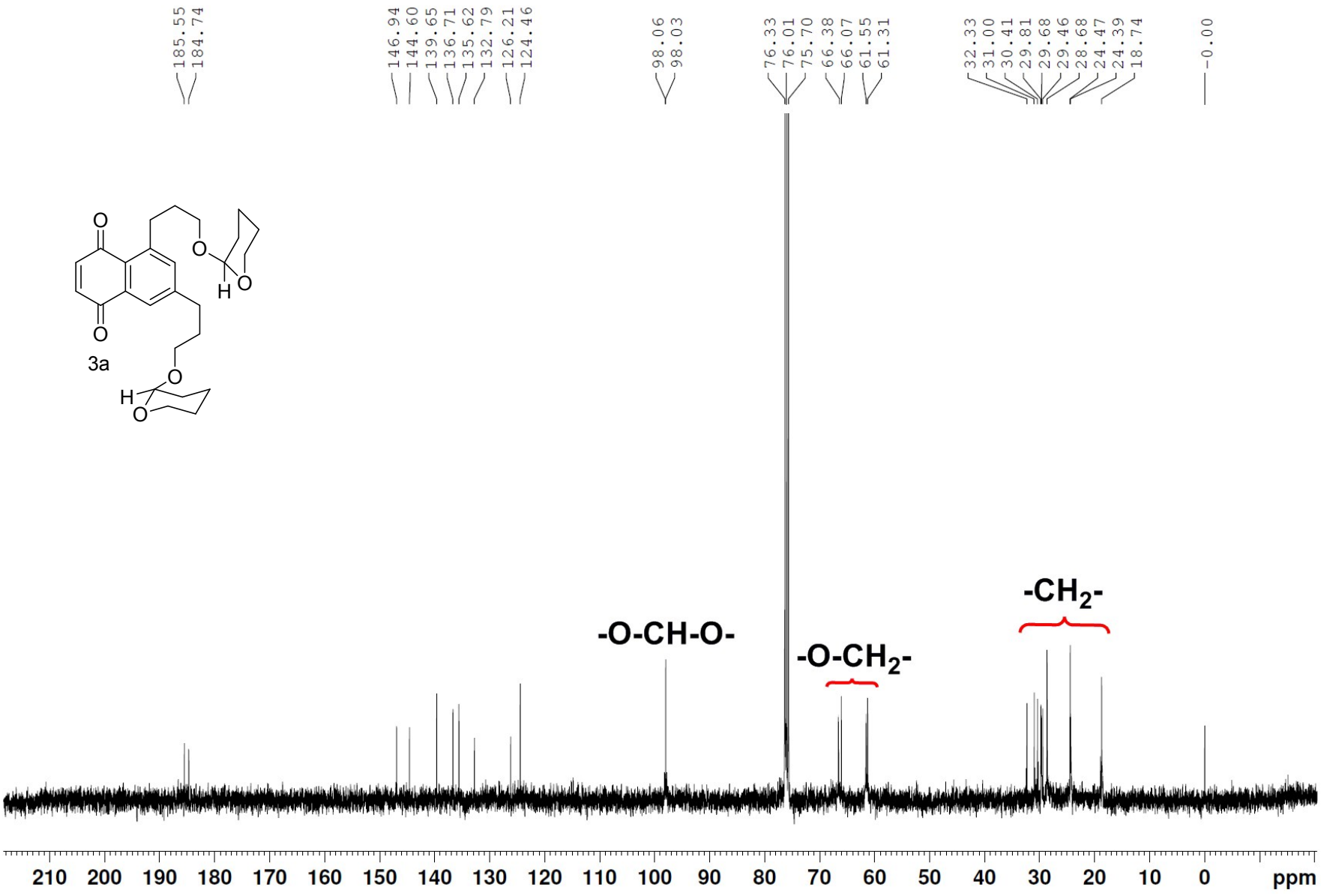


Figure 2. ^{13}C NMR (100 MHz, $CDCl_3$, 300K) of **3a**

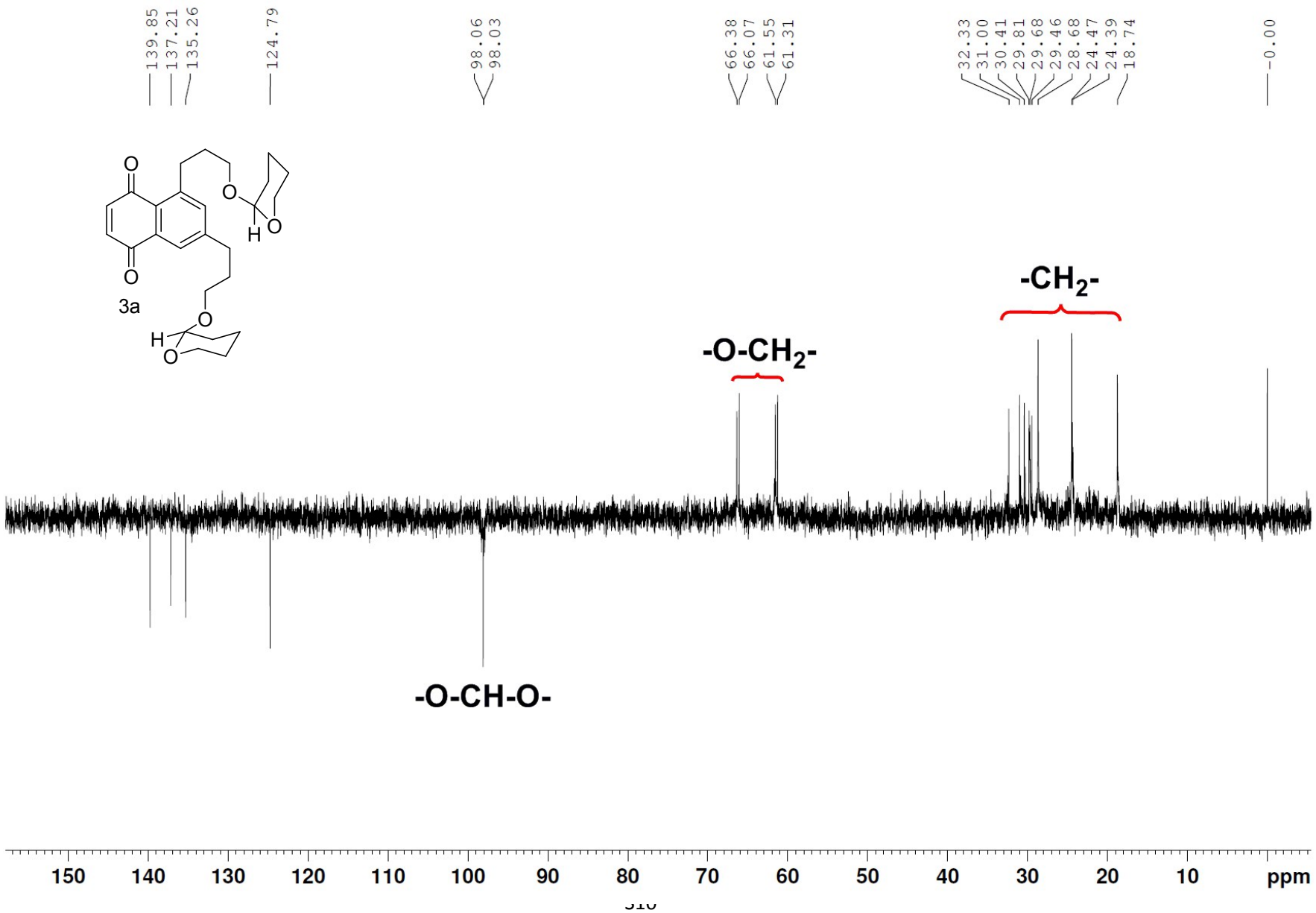


Figure 3. DEPT NMR (100 MHz, $CDCl_3$, 300K) of **3a**

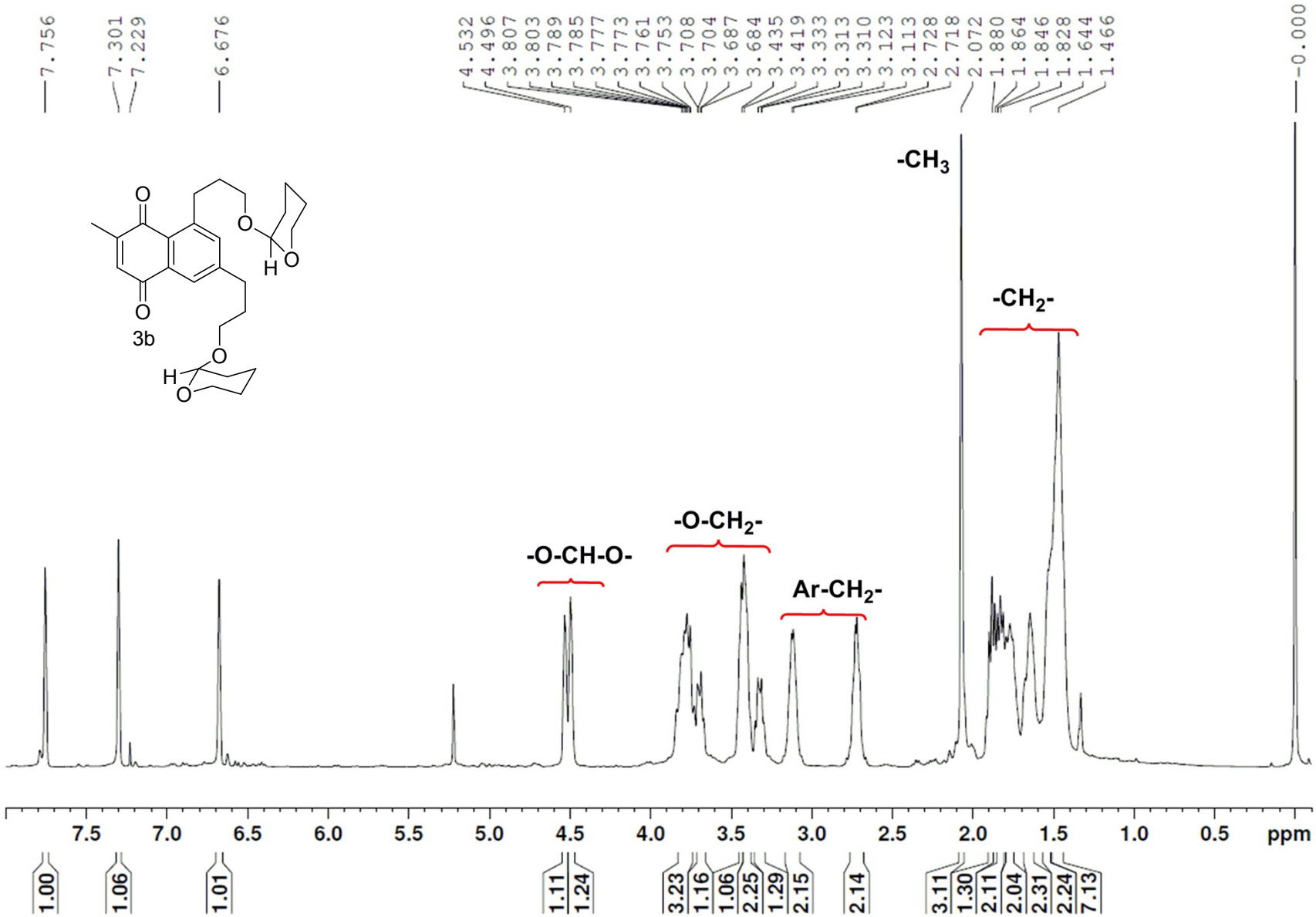


Figure 4. ^{1}H NMR (400 MHz, $CDCl_3$, 300K) of **3b** (TMS added as internal standard)

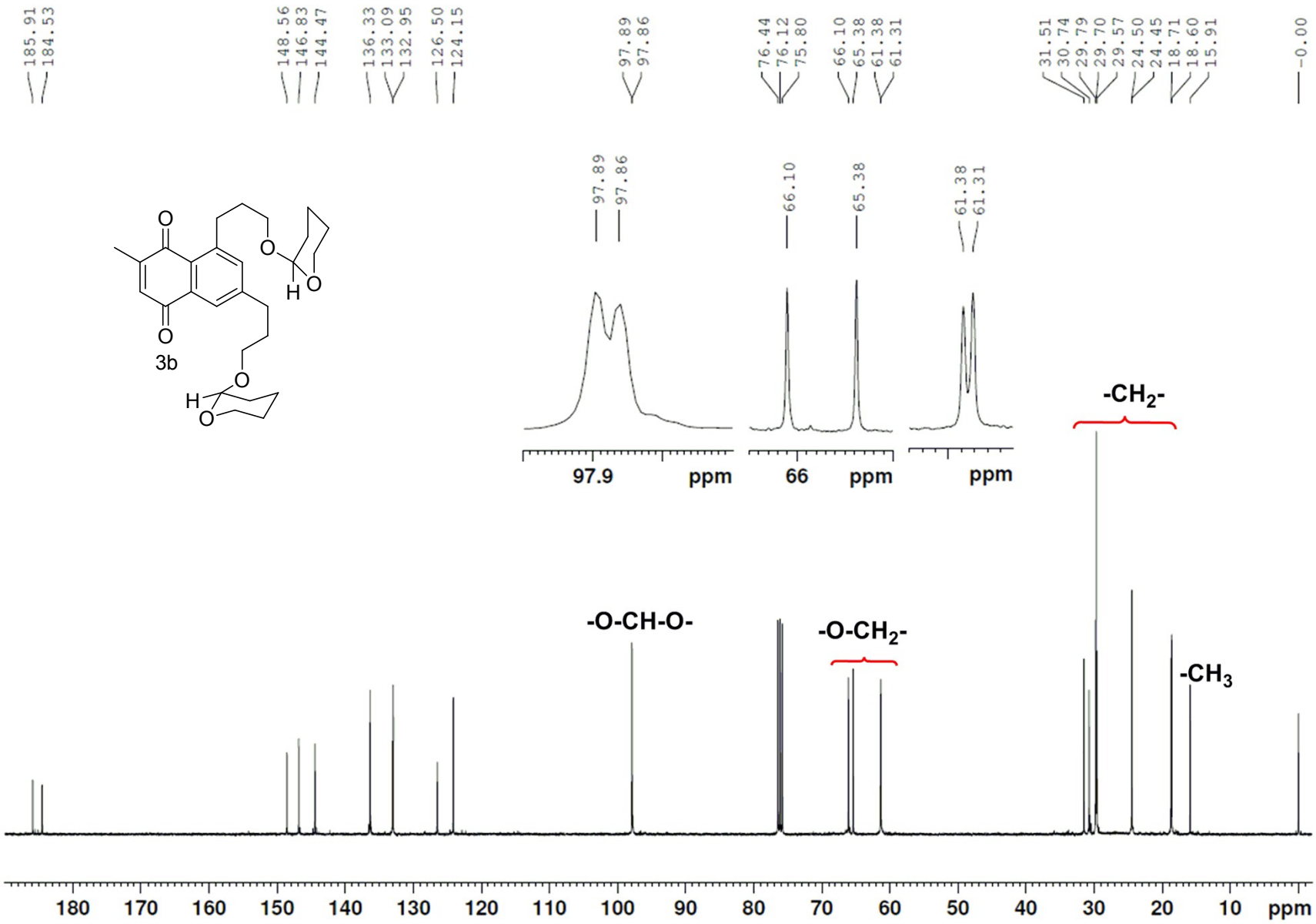


Figure 5. ^{13}C NMR (100 MHz, $CDCl_3$, 300K) of **3b**

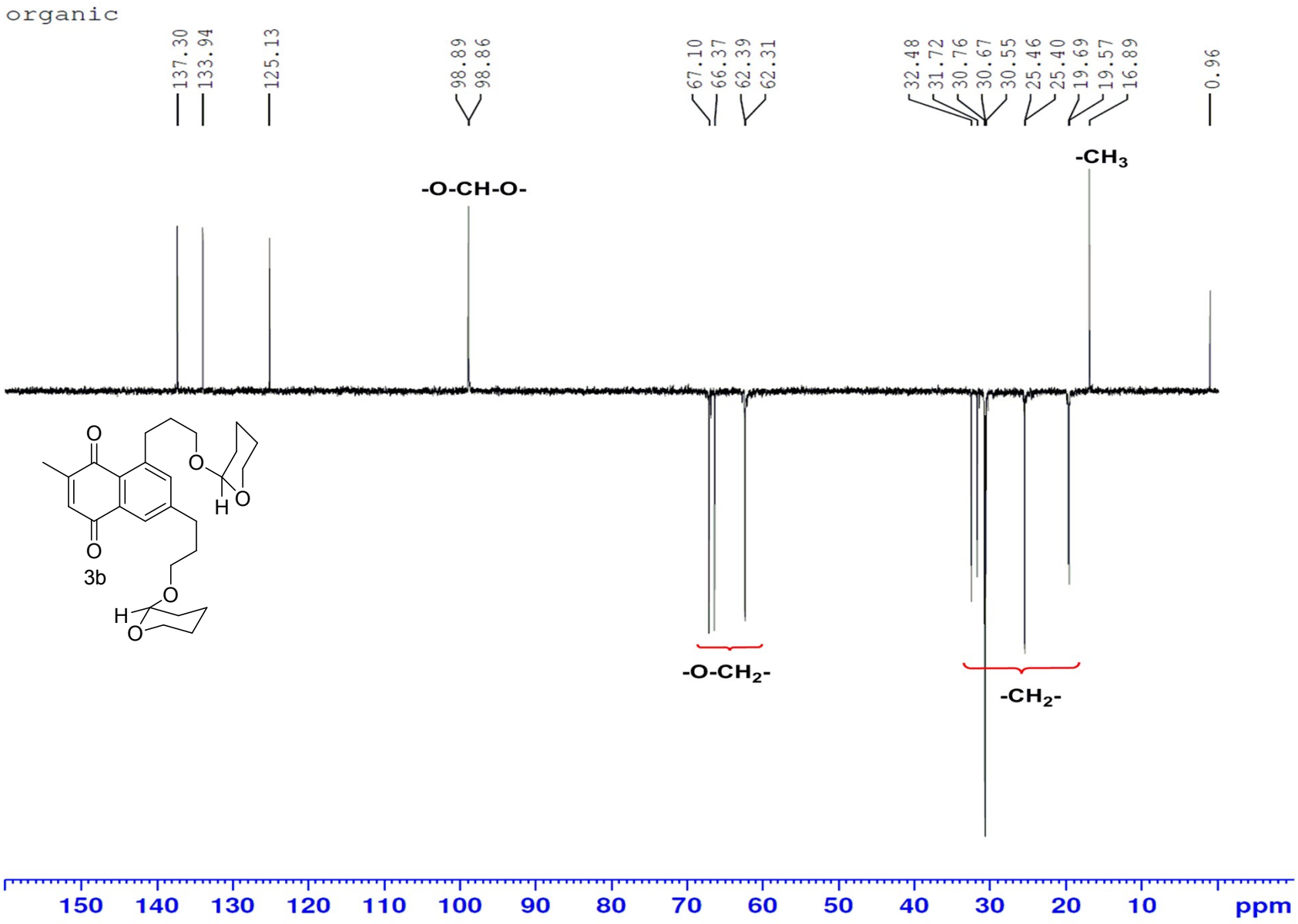


Figure 6. DEPT NMR (100 MHz, $CDCl_3$, 300K) of **3b**

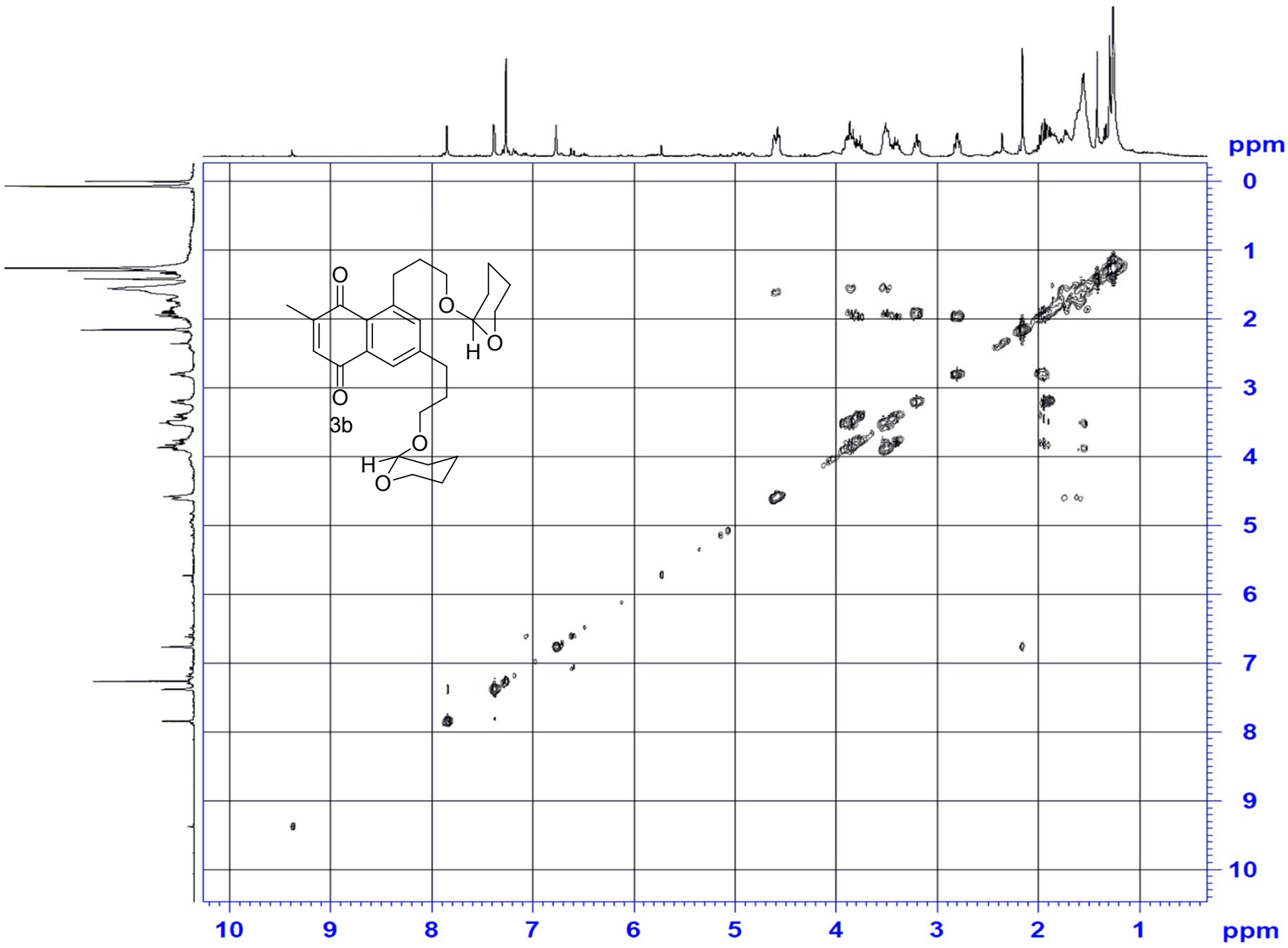


Figure 7. COSY NMR (400 MHz, $CDCl_3$, 300K) of **3b**

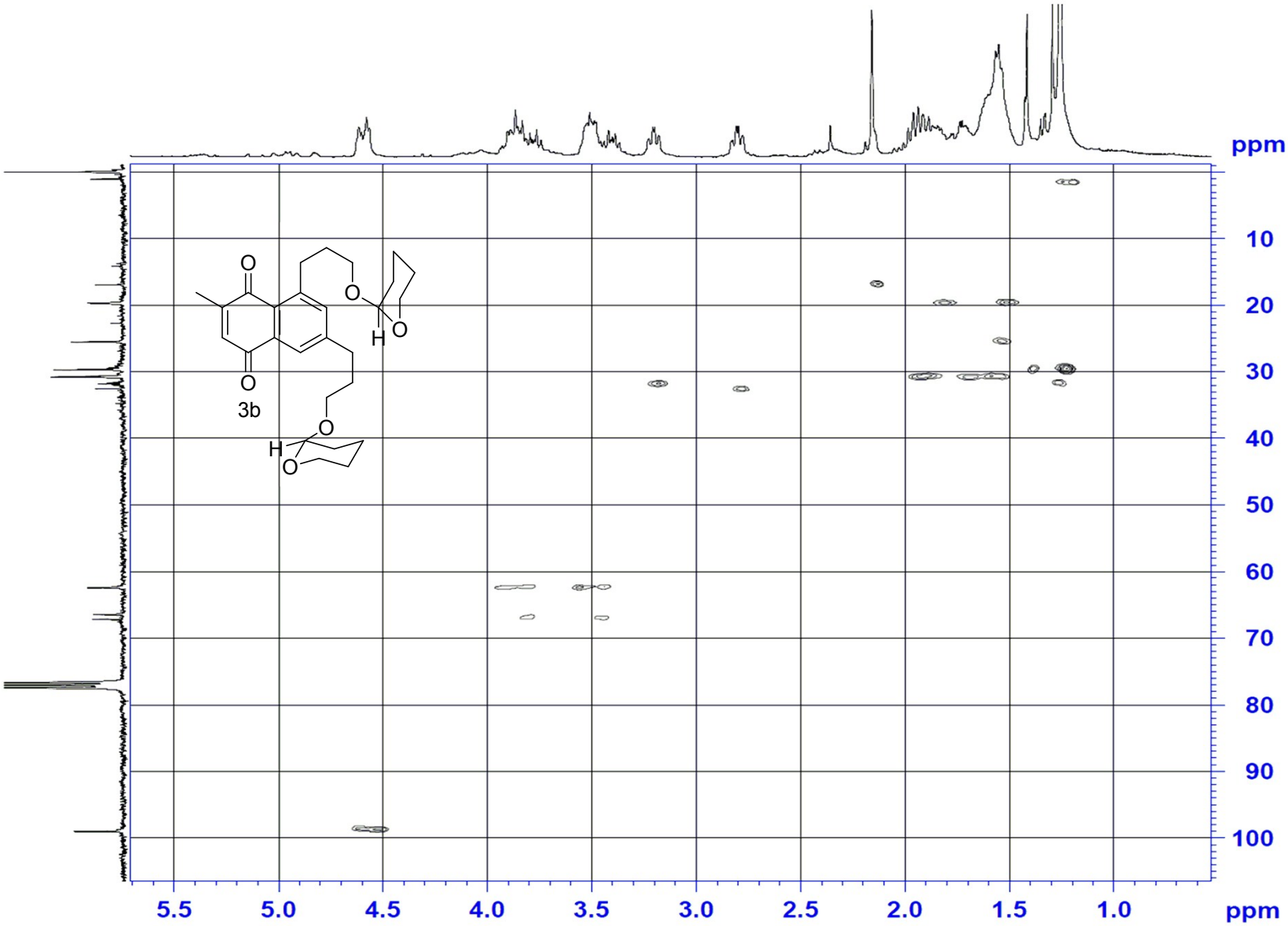


Figure 8. HSQC NMR (400 MHz, $CDCl_3$, 300K) of **3b**

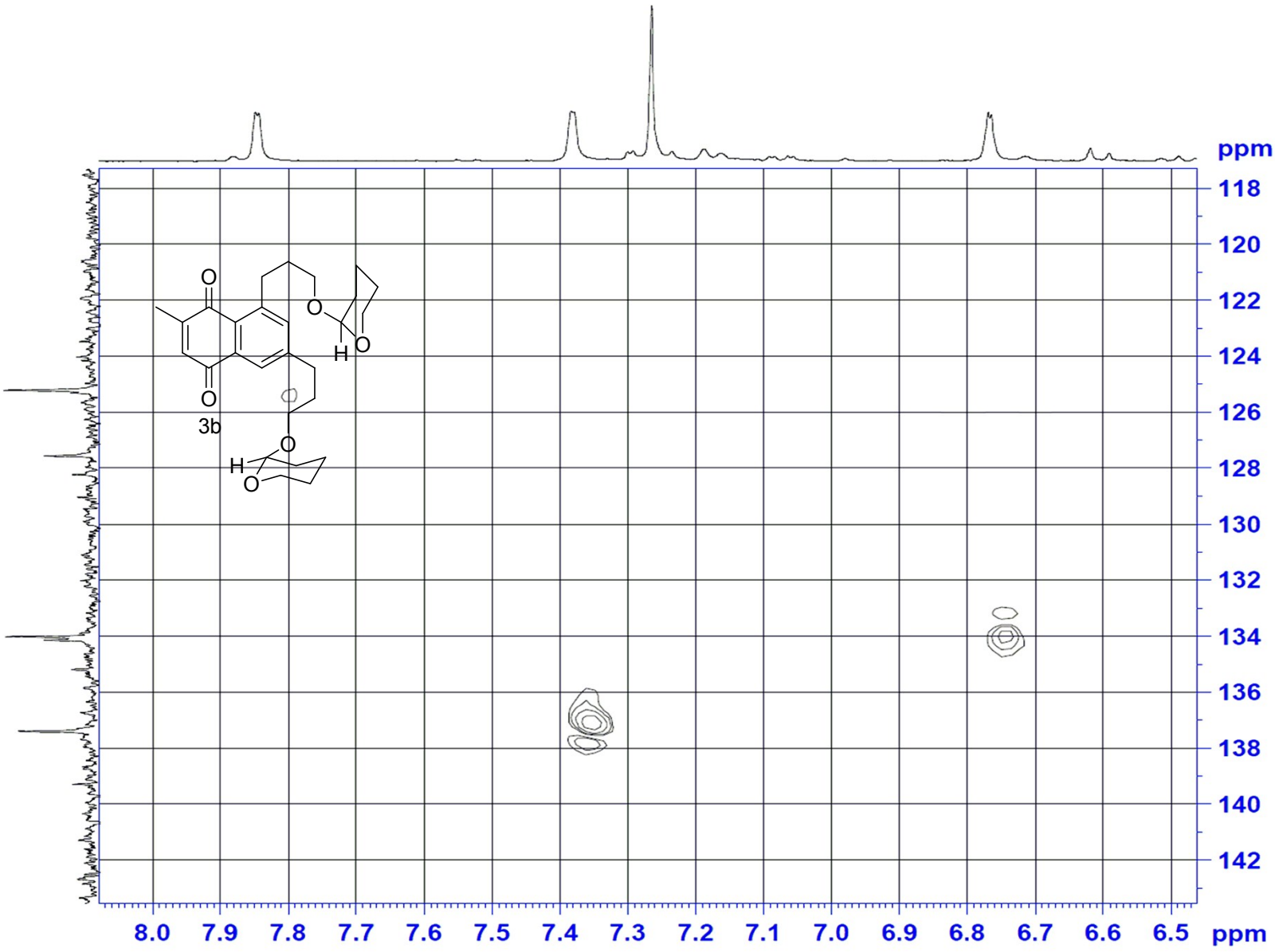


Figure 9. HSQC NMR (400 MHz, $CDCl_3$, 300K) of **3b**

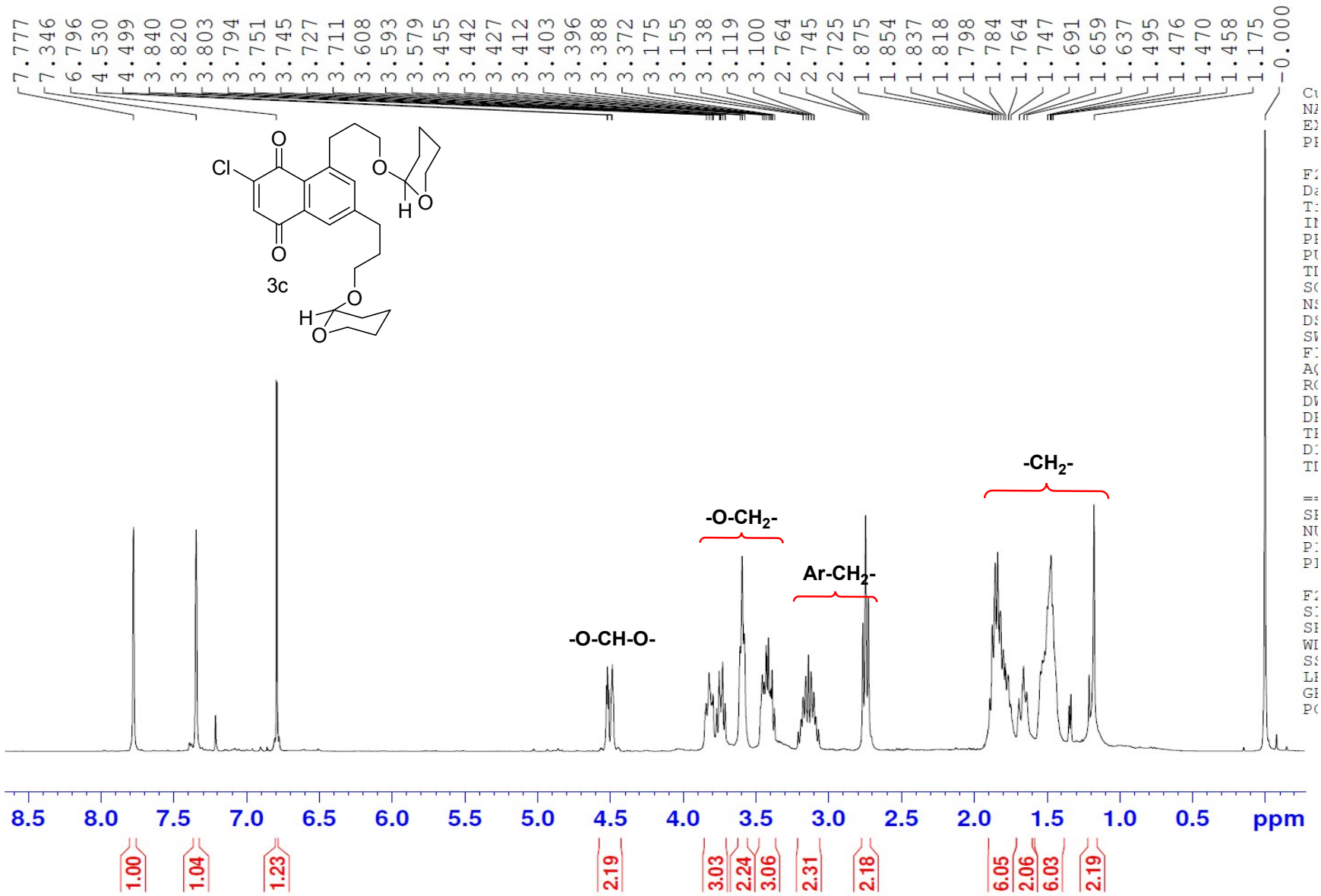


Figure 10. ^{1}H NMR (400 MHz, $CDCl_3$, 300K) of **3c** (TMS added as internal standard)

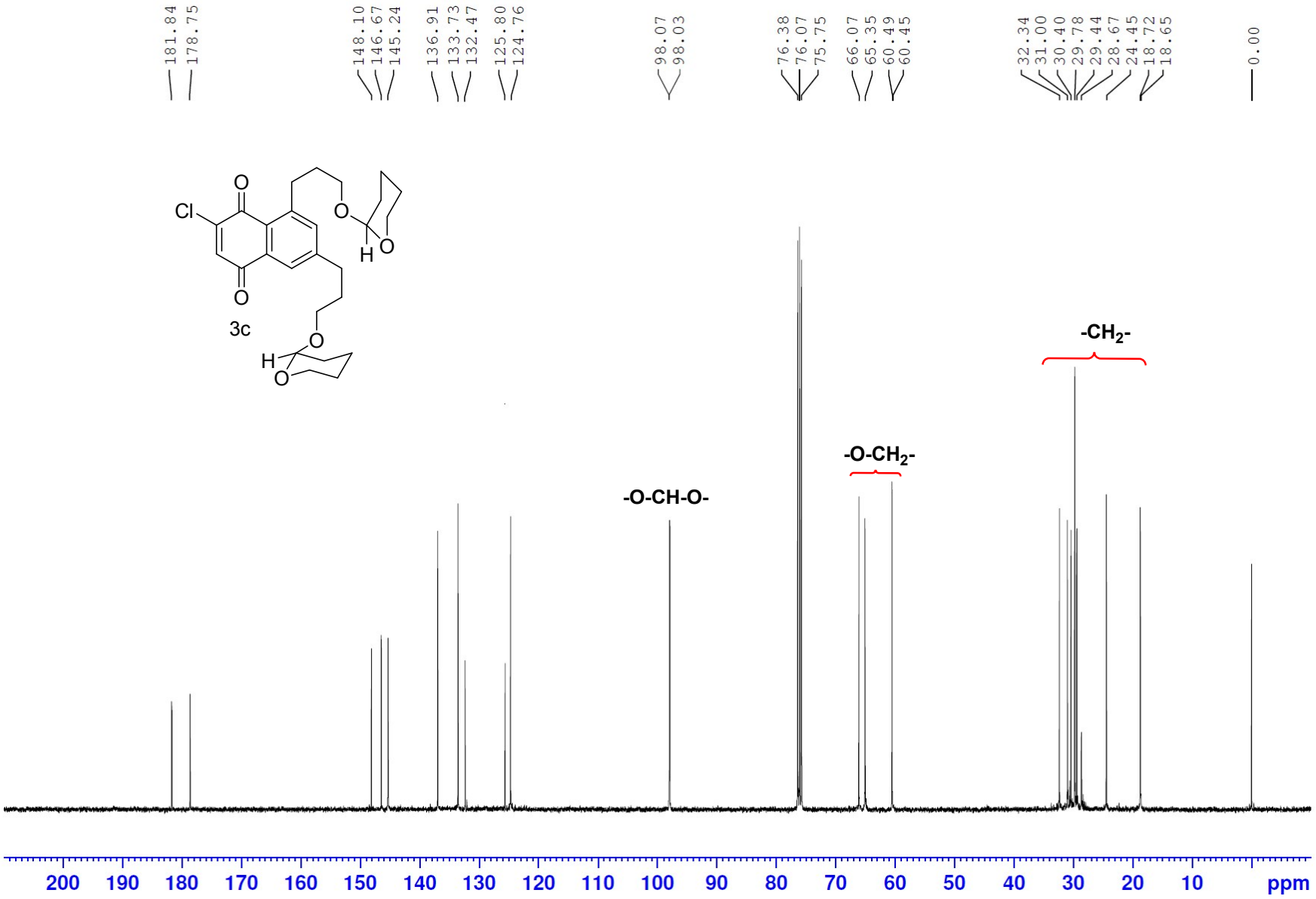


Figure 11. ^{13}C NMR (100 MHz, $CDCl_3$, 300K) of **3c**

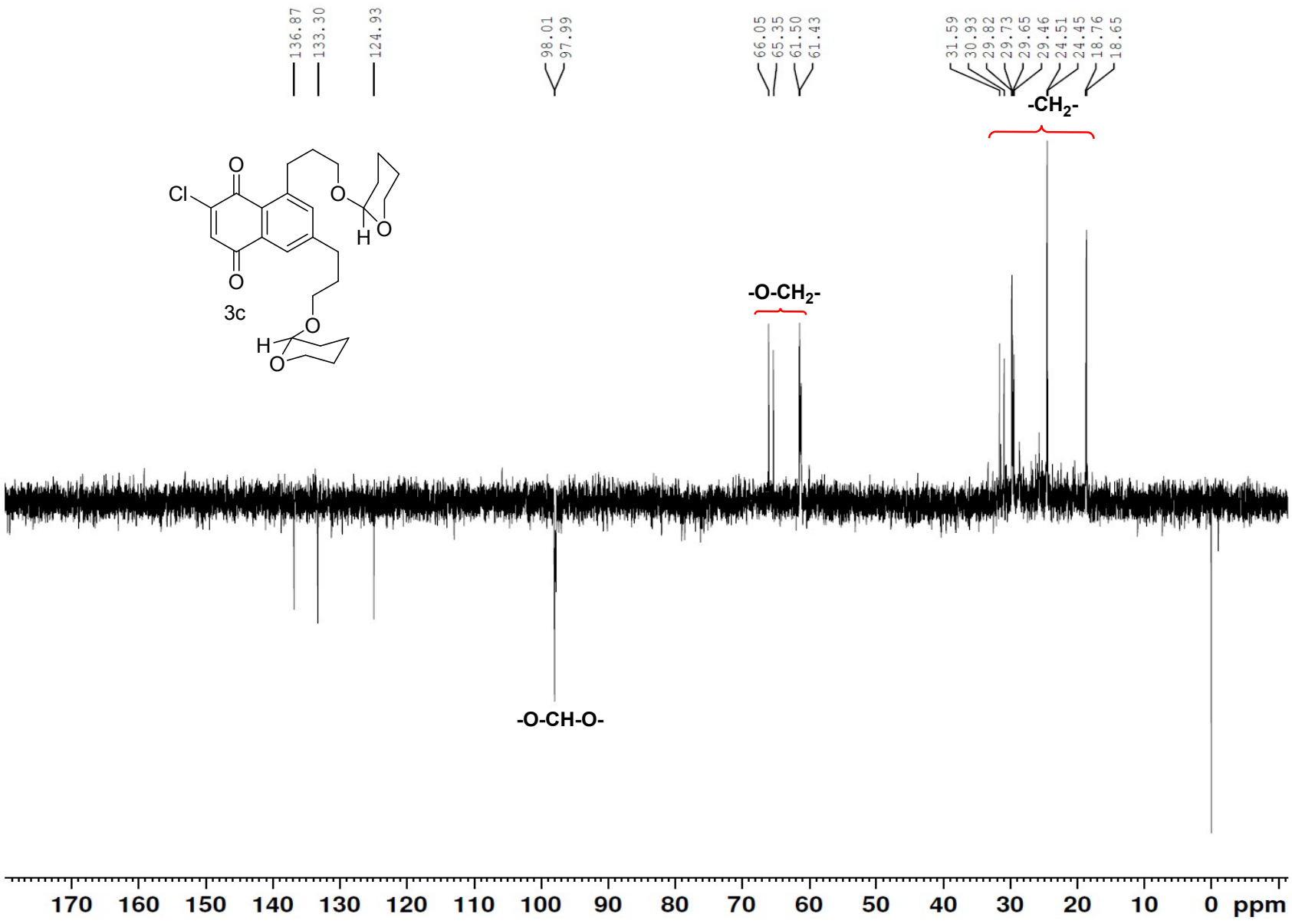


Figure 12. DEPT NMR (100 MHz, $CDCl_3$, 300K) of **3c**

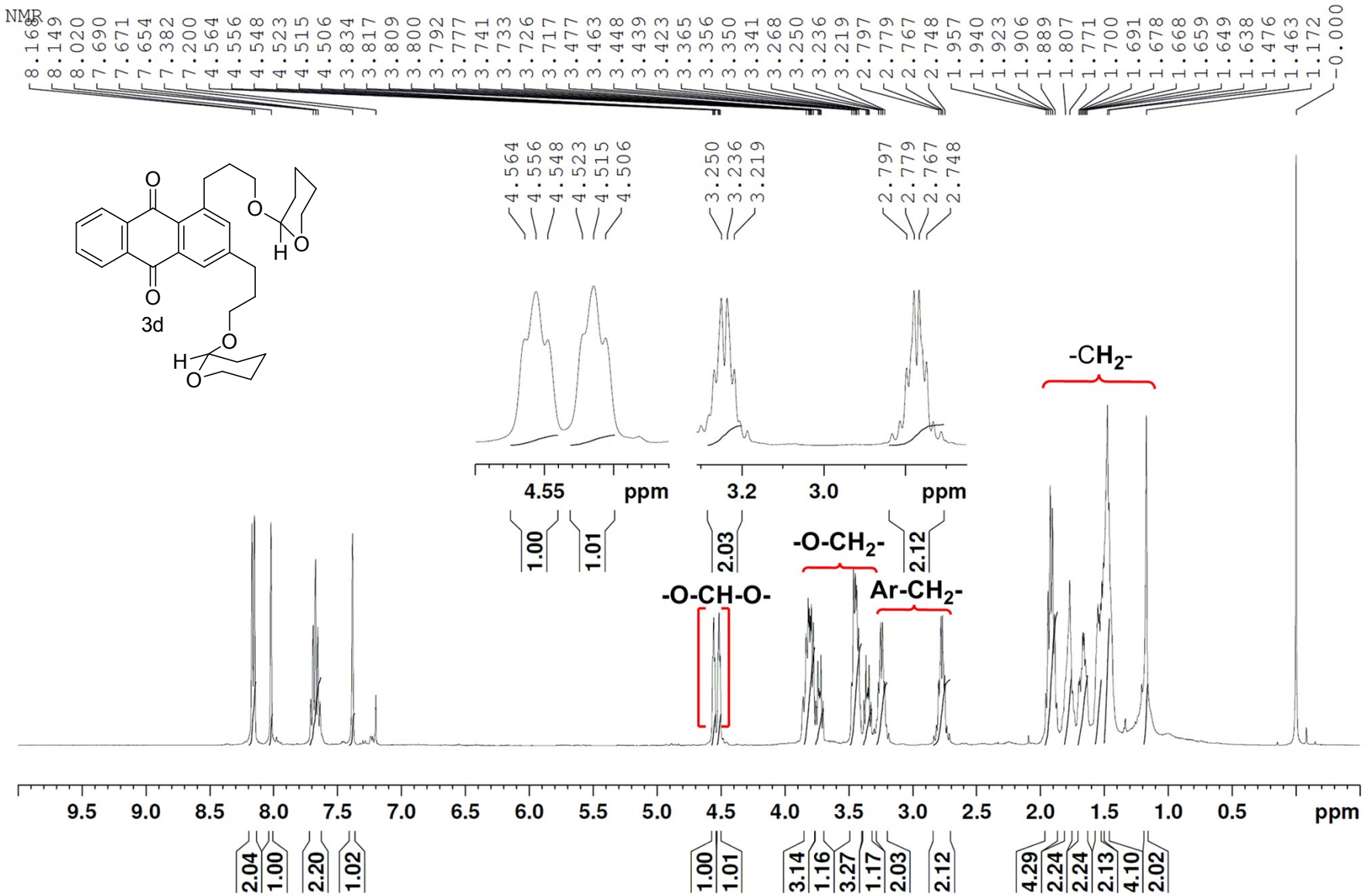


Figure 13. ^{1}H NMR (400 MHz, $CDCl_3$, 300K) of **3d** (TMS added as internal standard)

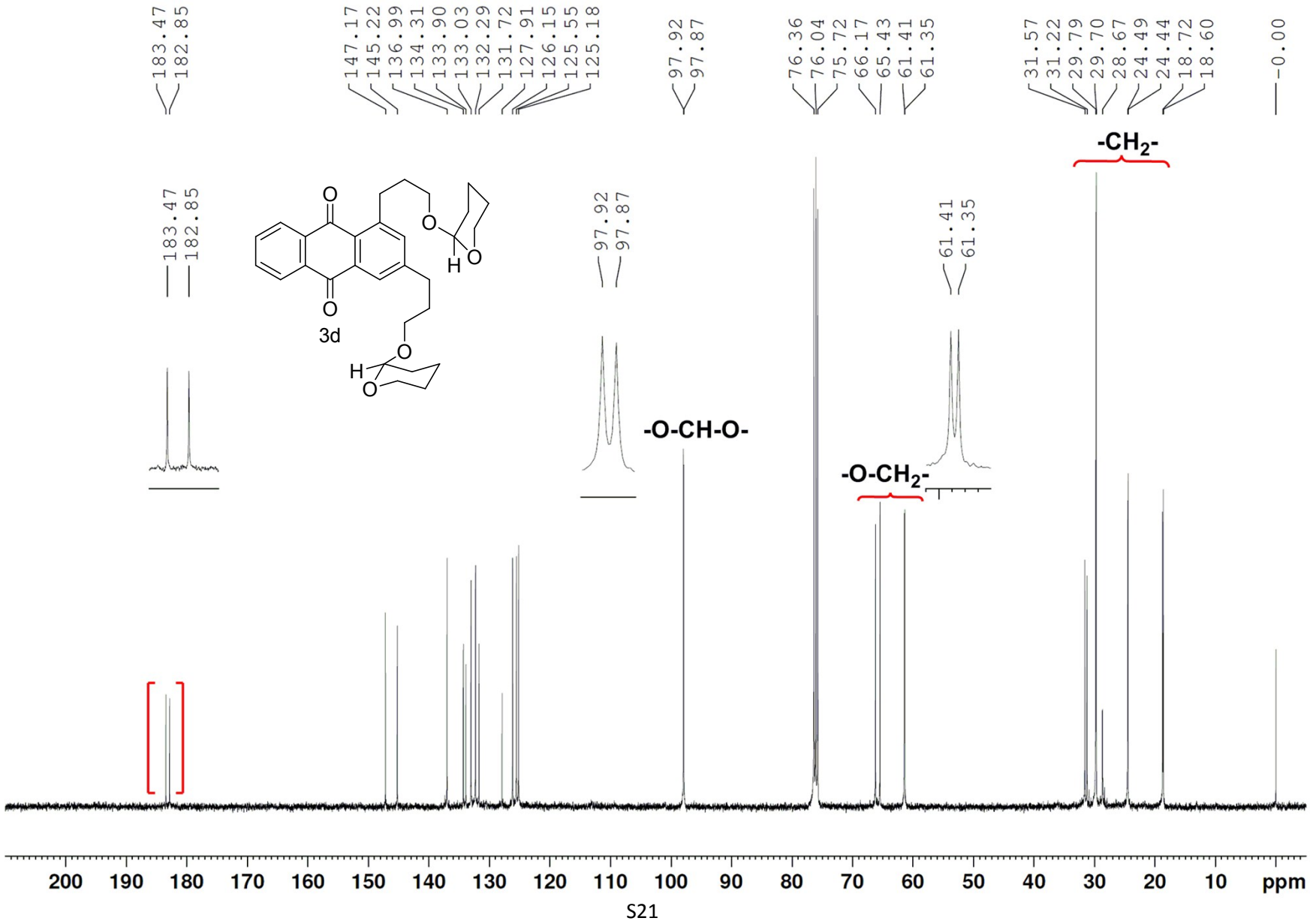


Figure 14. ^{13}C NMR (100 MHz, $CDCl_3$, 300K) of **3d**

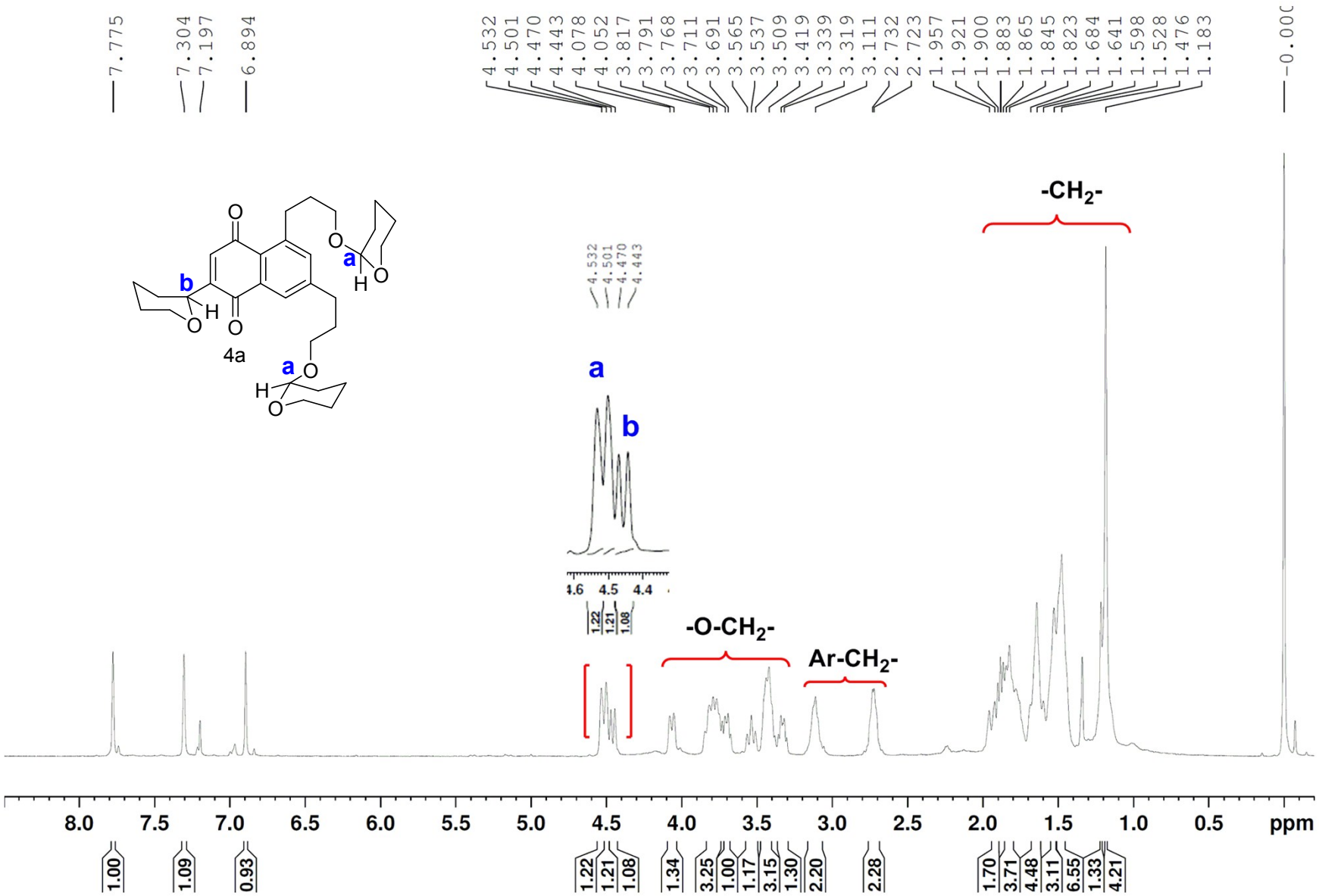


Figure 15. ^{1}H NMR (400 MHz, $CDCl_3$, 300K) of **4a** (TMS added as internal standard)

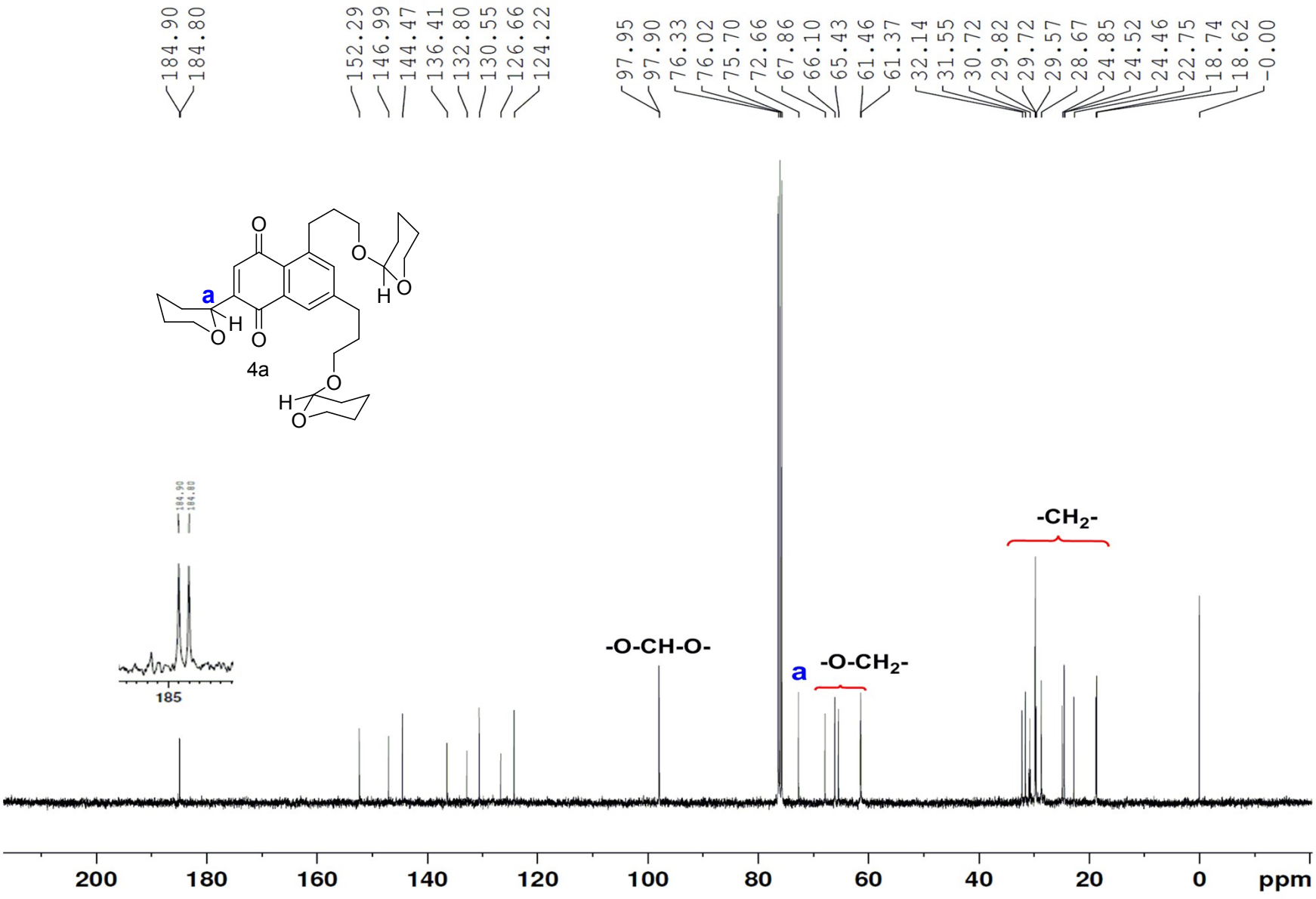


Figure 16. ^{13}C NMR (100 MHz, $CDCl_3$, 300K) of **4a**

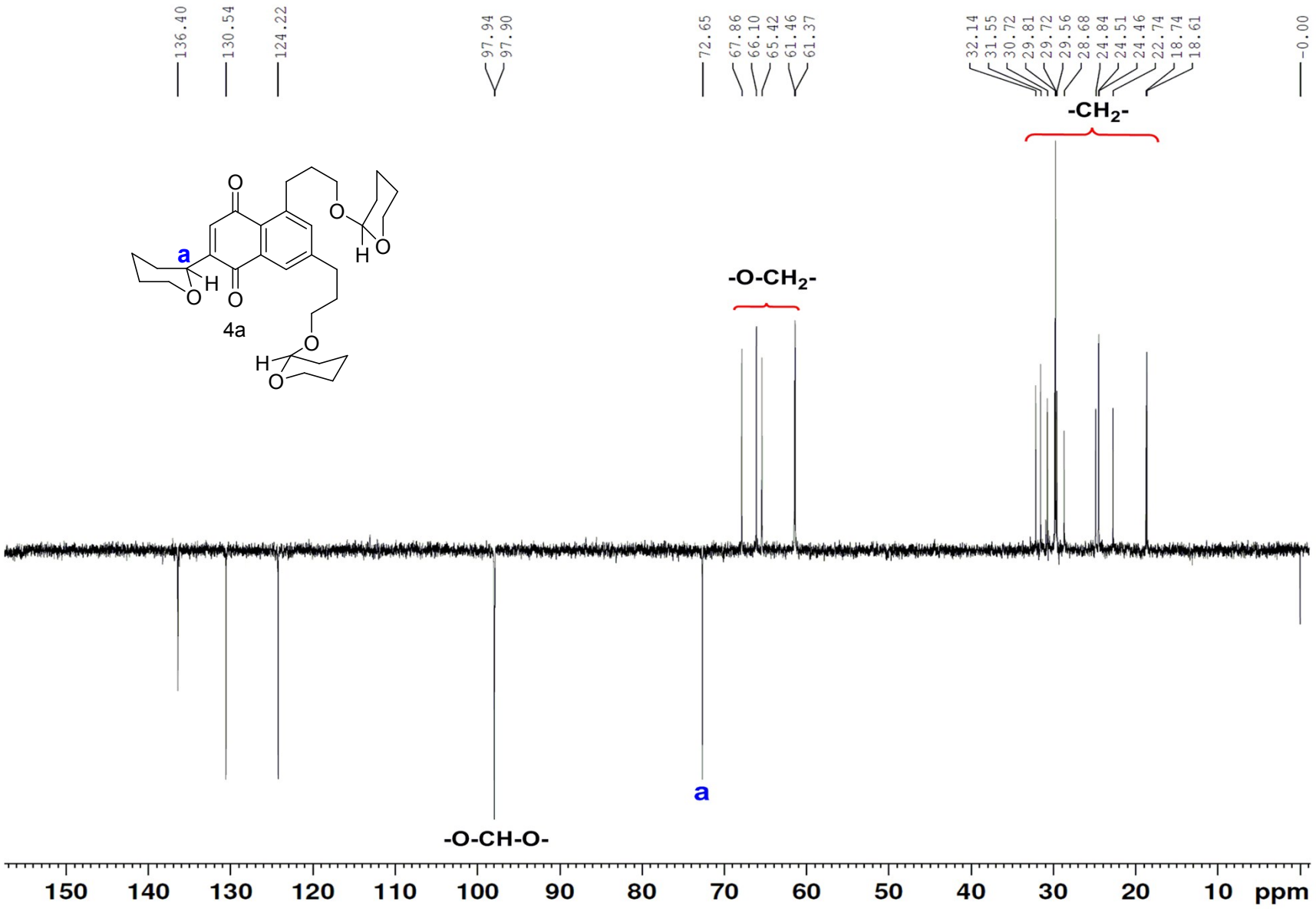


Figure 17. DEPT NMR (100 MHz, $CDCl_3$, 300K) of **4a**

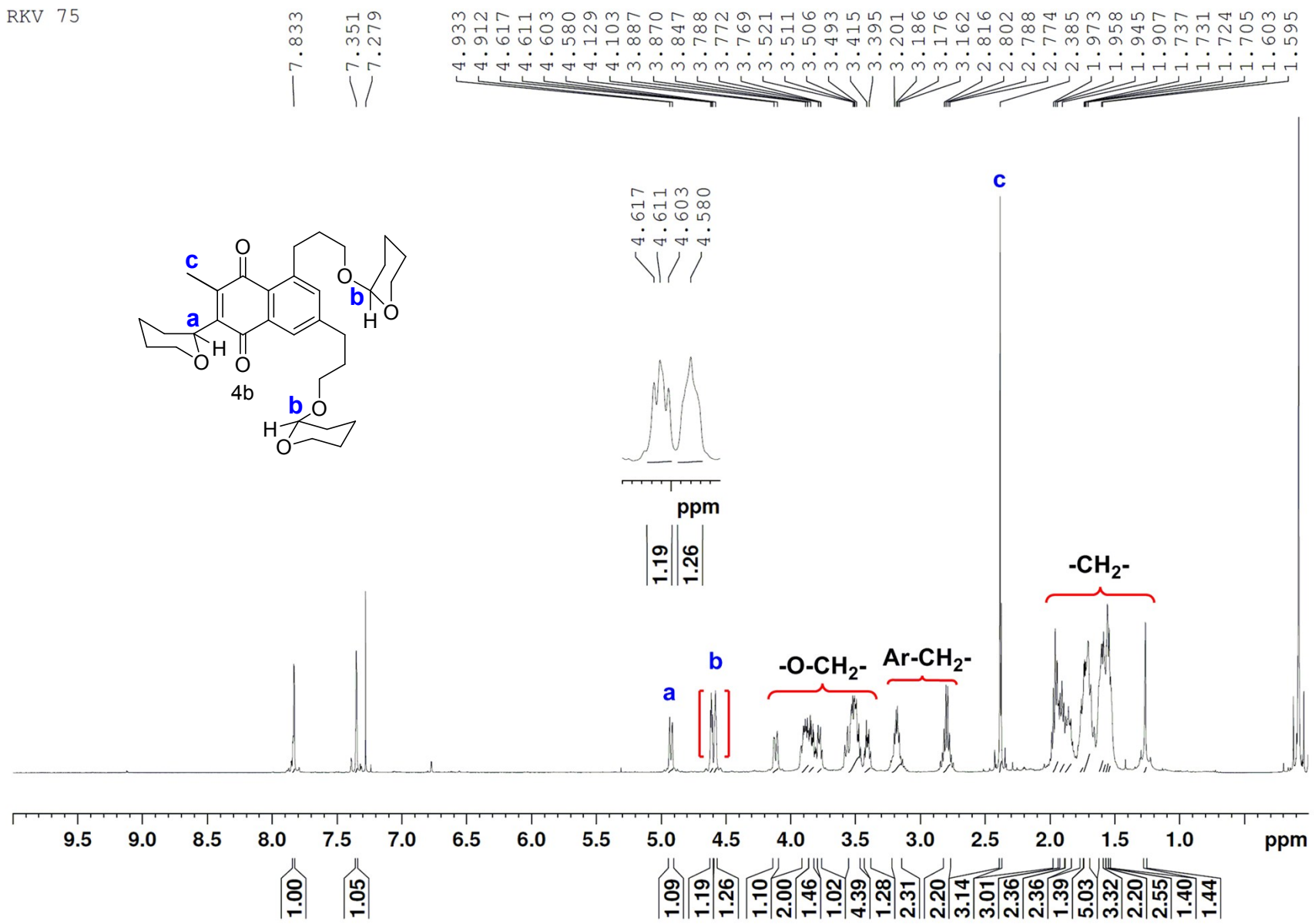


Figure 18. ^{1}H NMR (400 MHz, $CDCl_3$, 300K) of **4b**

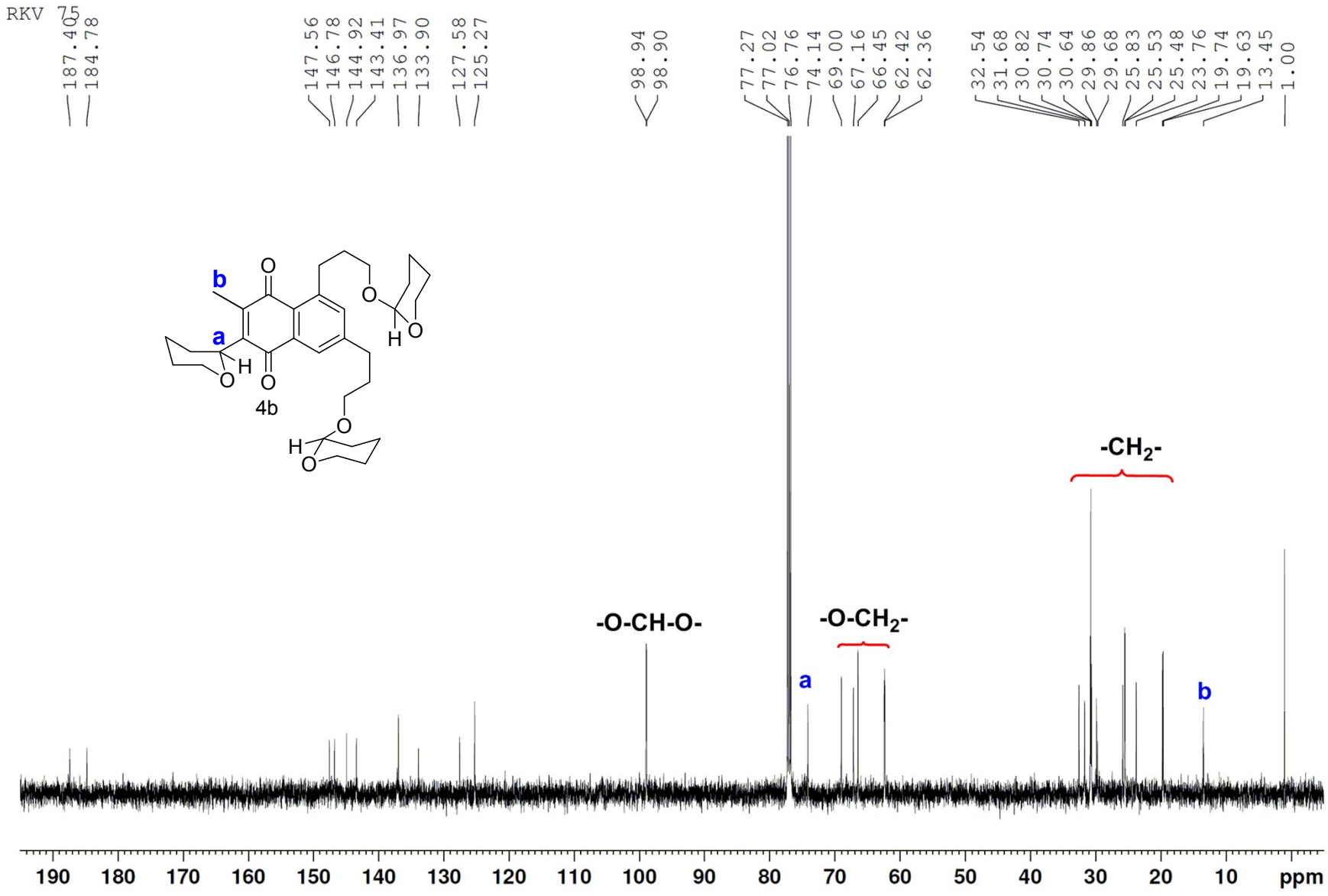


Figure 19. ^{13}C NMR (100 MHz, $CDCl_3$, 300K) of **4b**

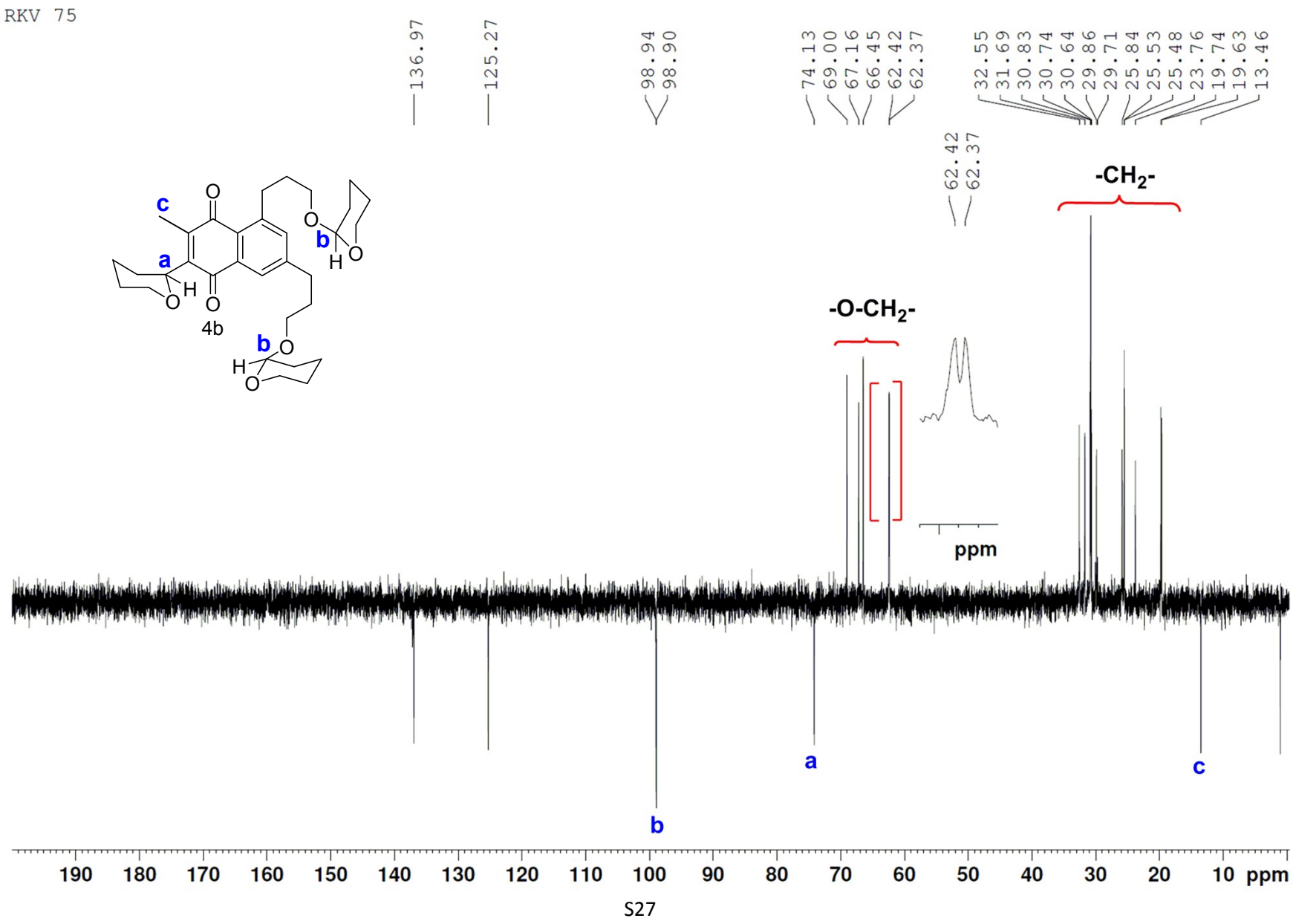


Figure 20. DEPT NMR (100 MHz, $CDCl_3$, 300K) of **4b**.

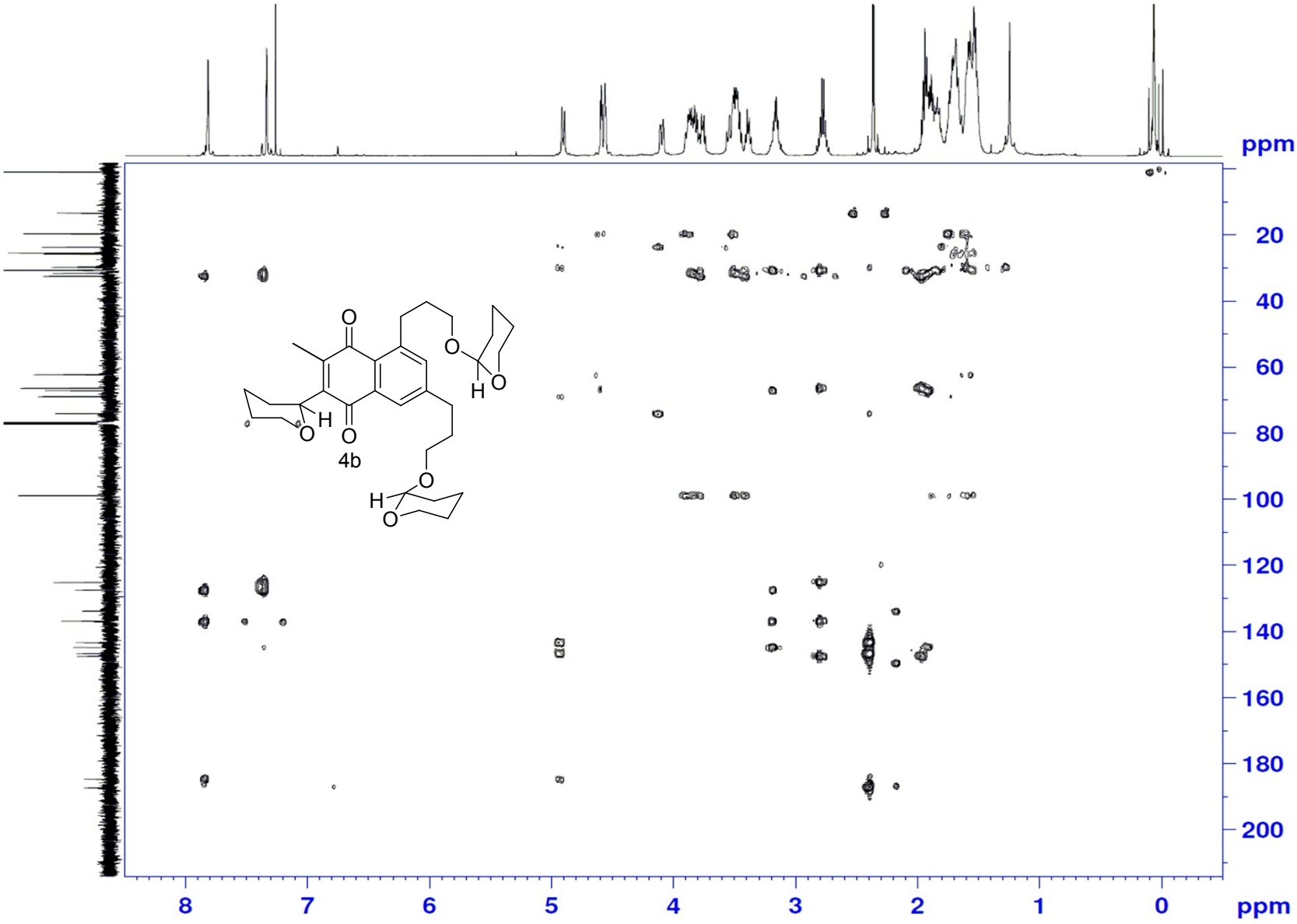


Figure 21. HMBC NMR (400 MHz, $CDCl_3$, 300K) of **4b**

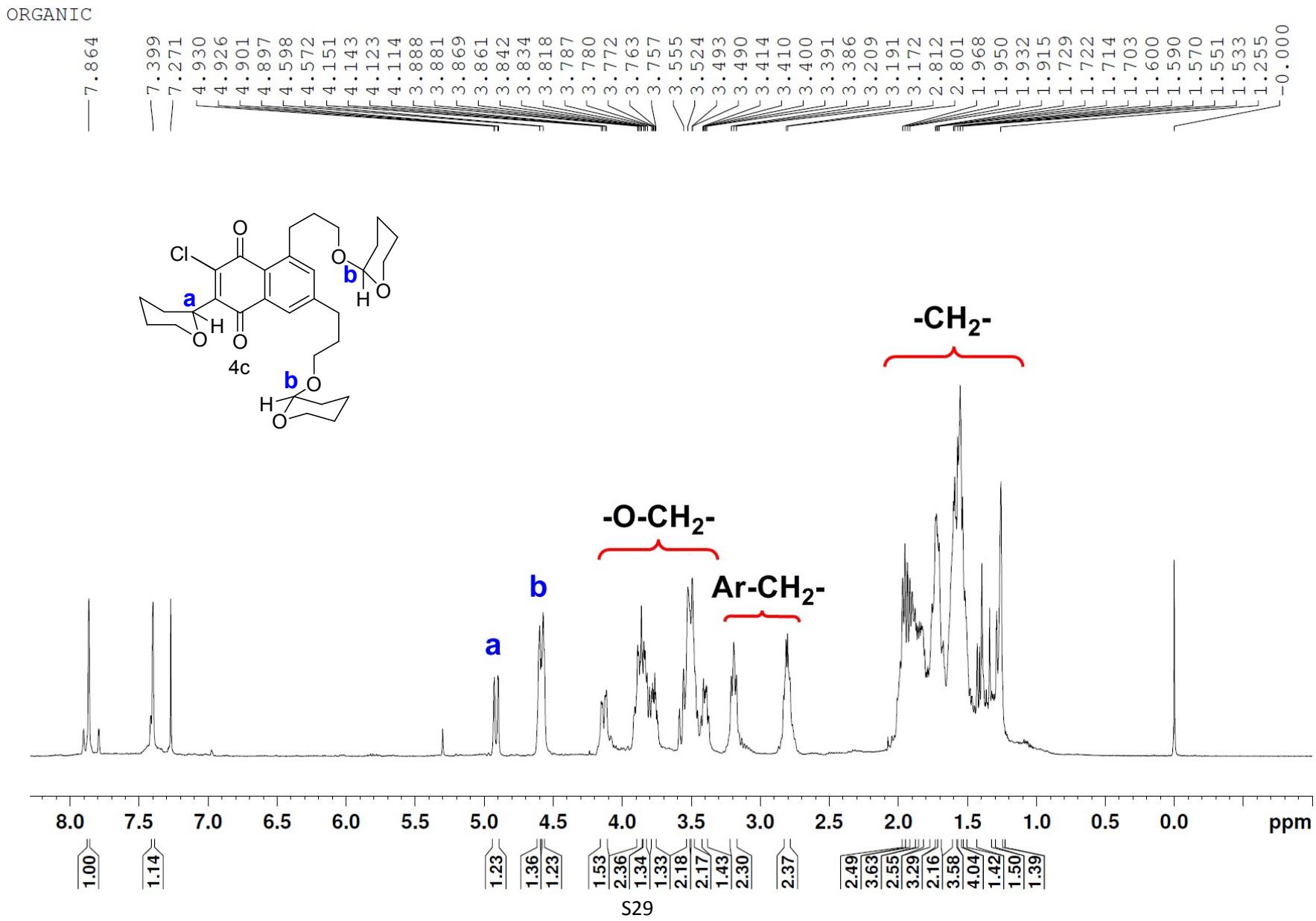


Figure 22. ^{1}H NMR (400 MHz, $CDCl_3$, 300K) of **4c** (TMS added as internal standard)

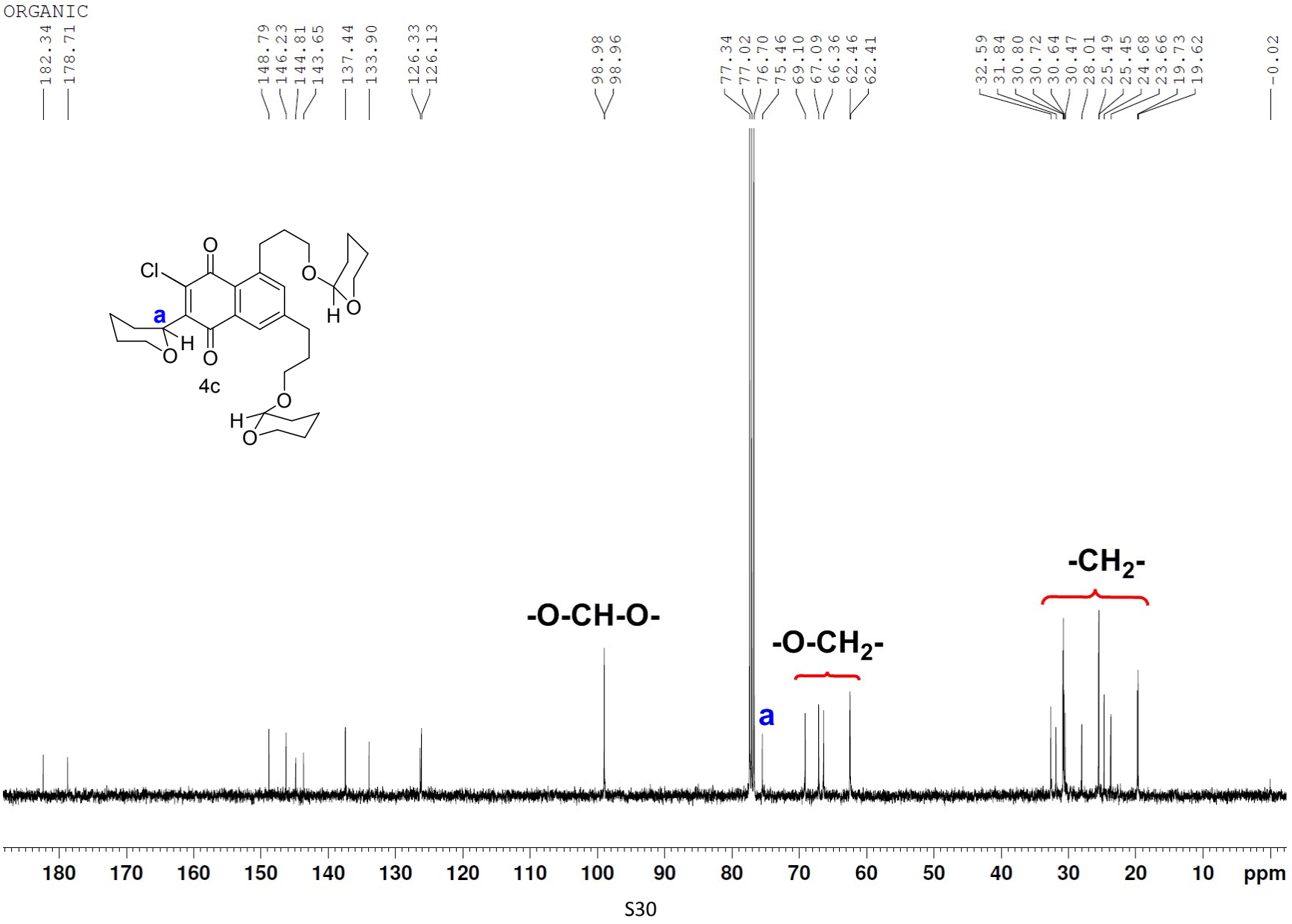


Figure 23. ^{13}C NMR (100 MHz, $CDCl_3$, 300K) of **4c**

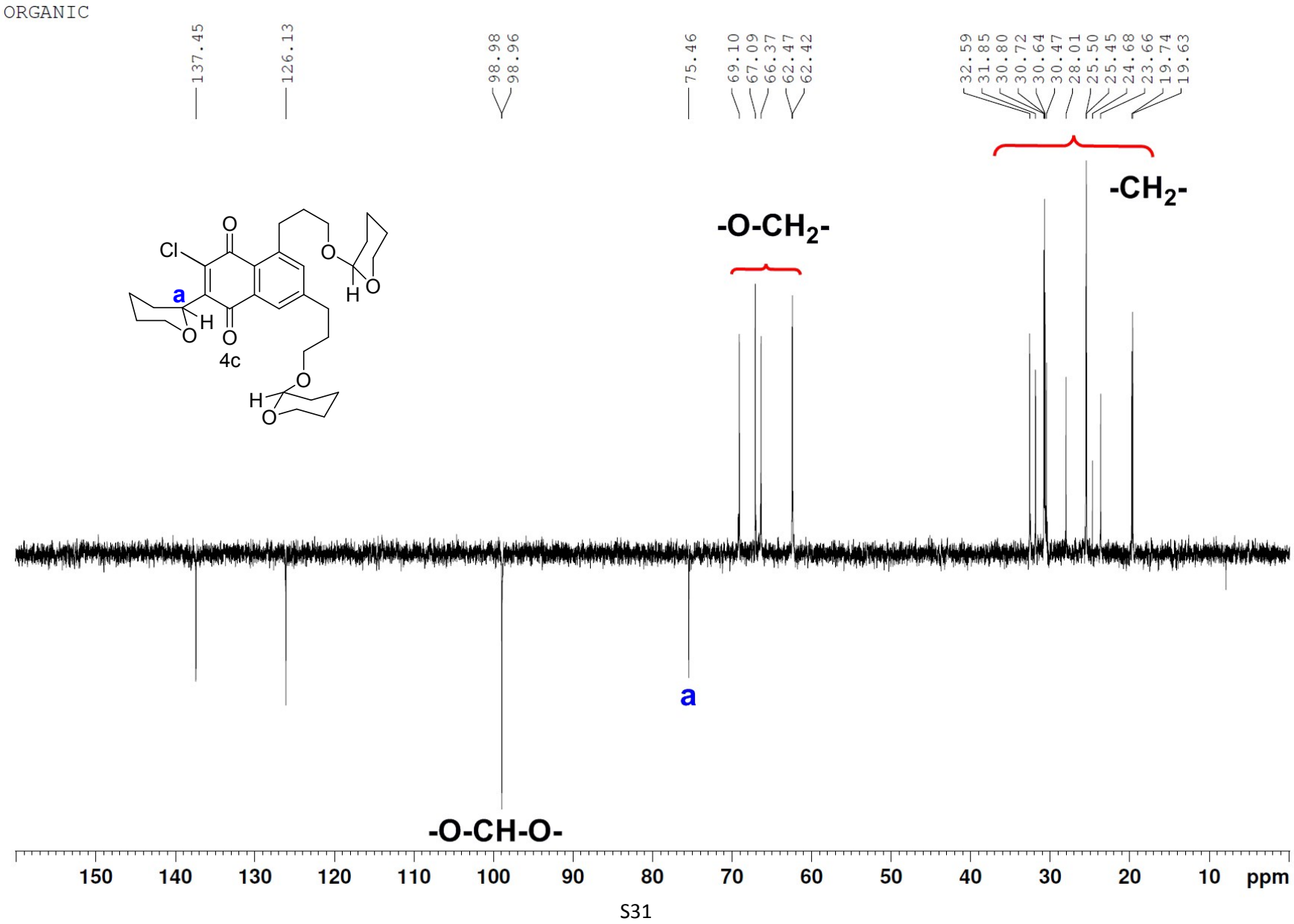


Figure 24. DEPT NMR (100 MHz, $CDCl_3$, 300K) of **4c**

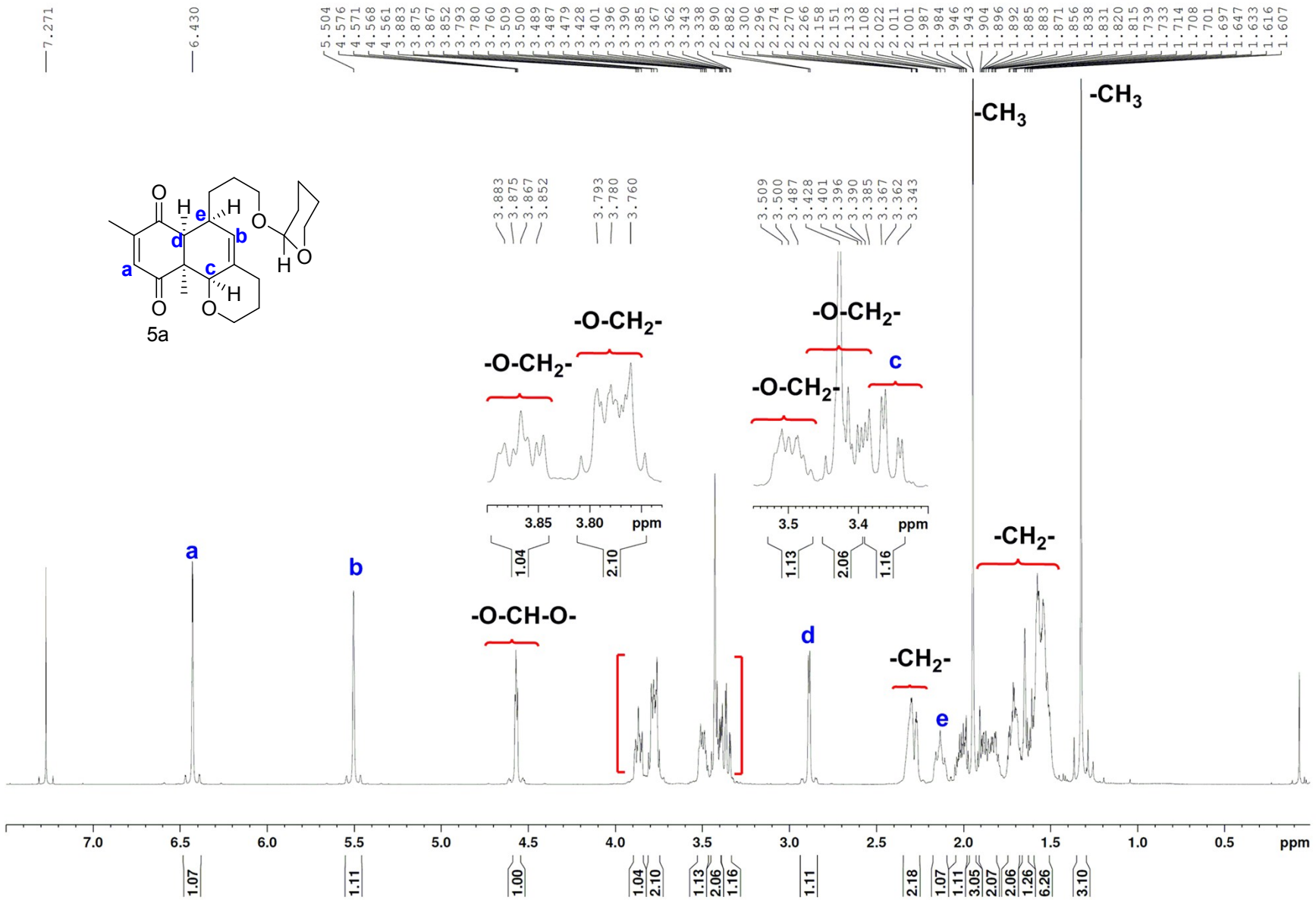


Figure 25. ^{1}H NMR (500 MHz, $CDCl_3$, 300K) of **5a** (TMS added as internal standard)

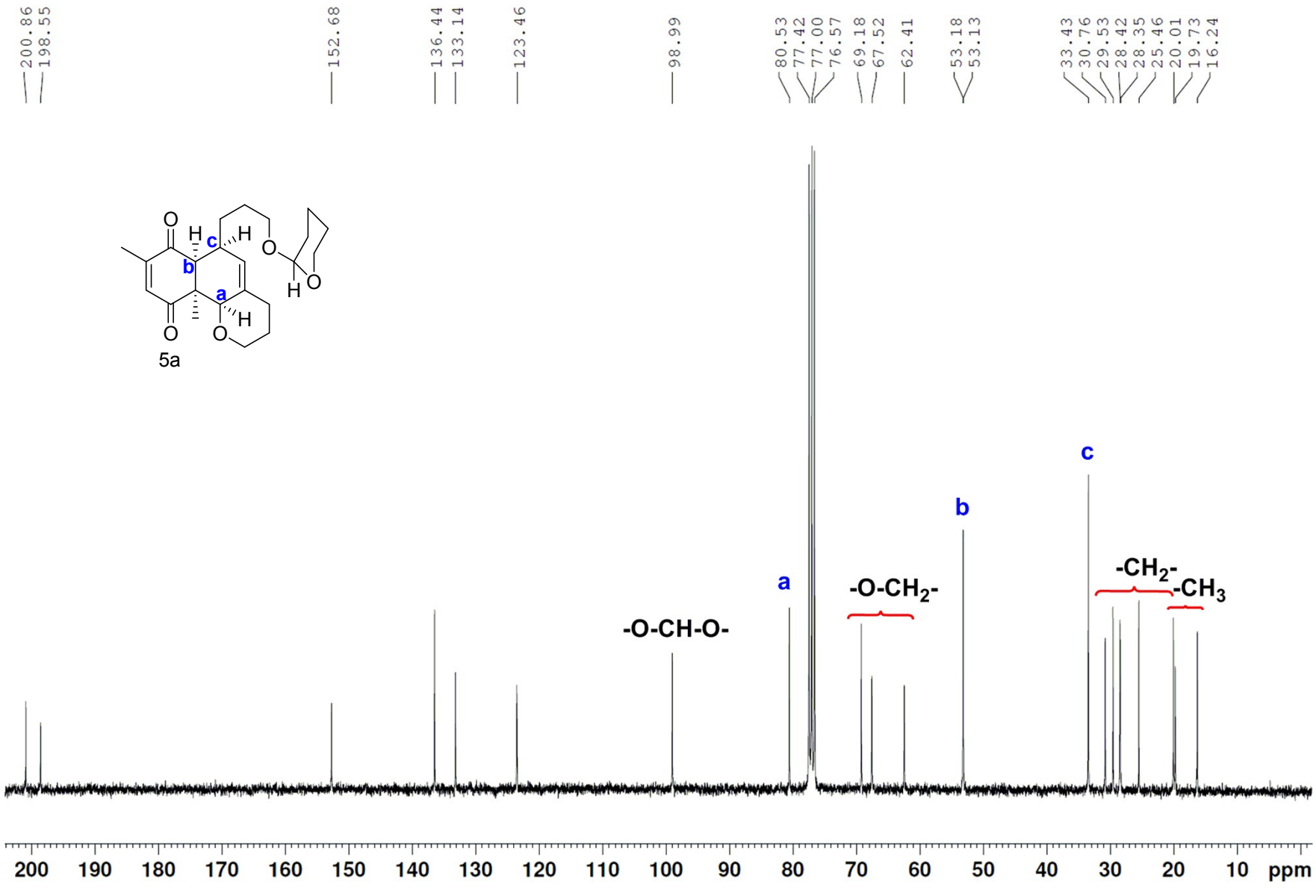


Figure 26. ^{13}C NMR (125 MHz, $CDCl_3$, 300K) of **5a**

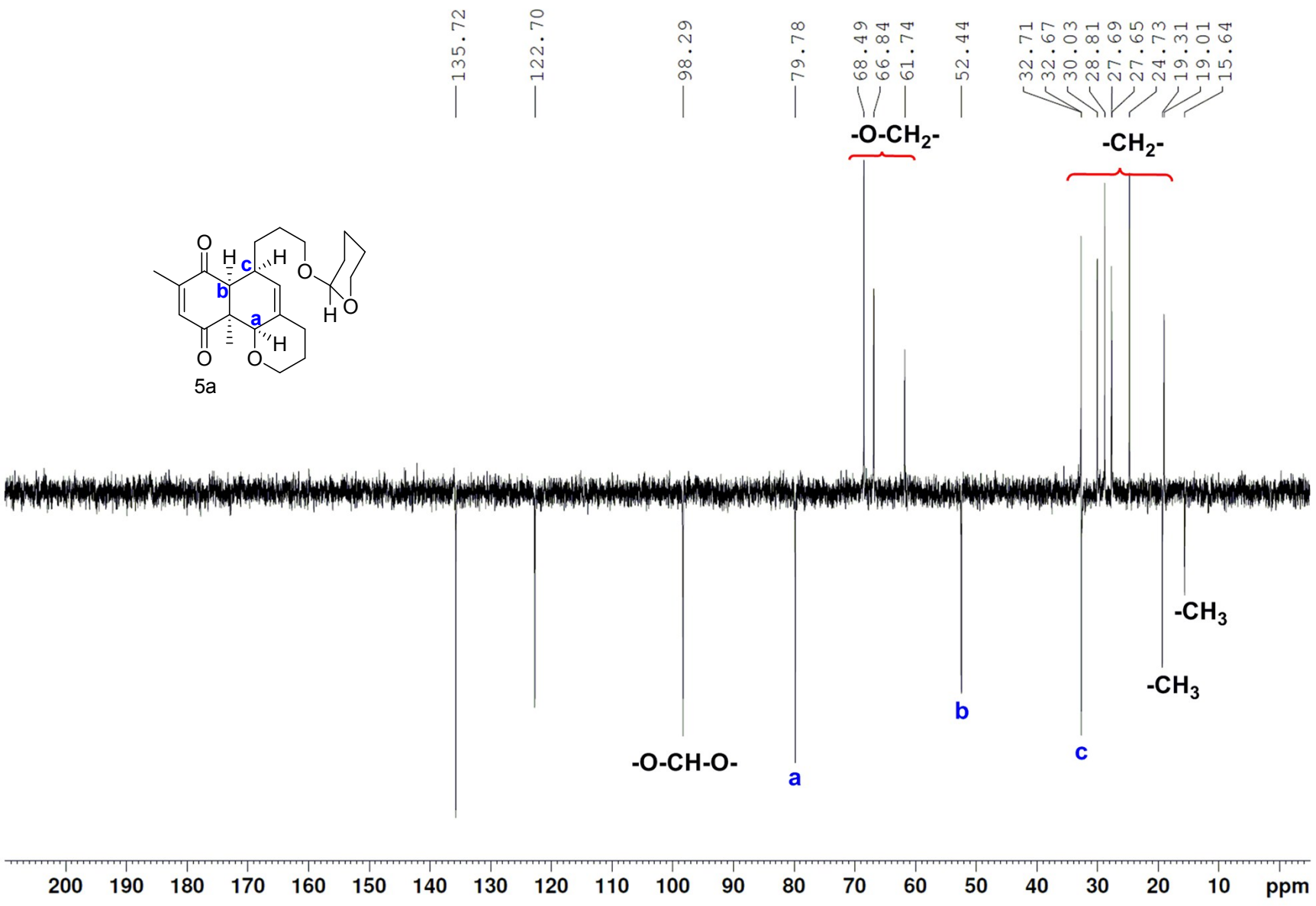


Figure 27. DEPT NMR (125 MHz, $CDCl_3$, 300K) of **5a**

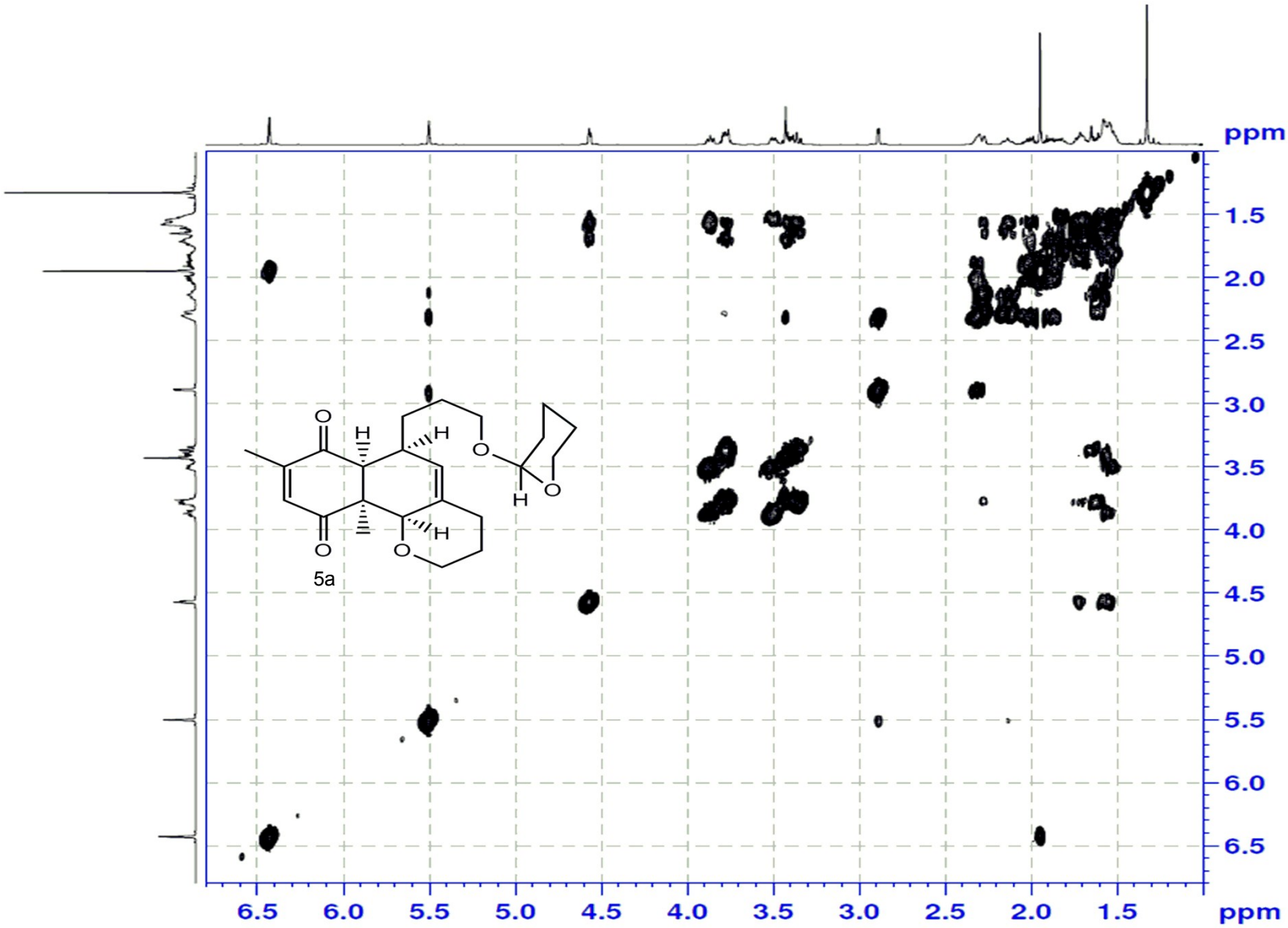


Figure 28. COSY NMR (500 MHz, $CDCl_3$, 300K) of **5a**

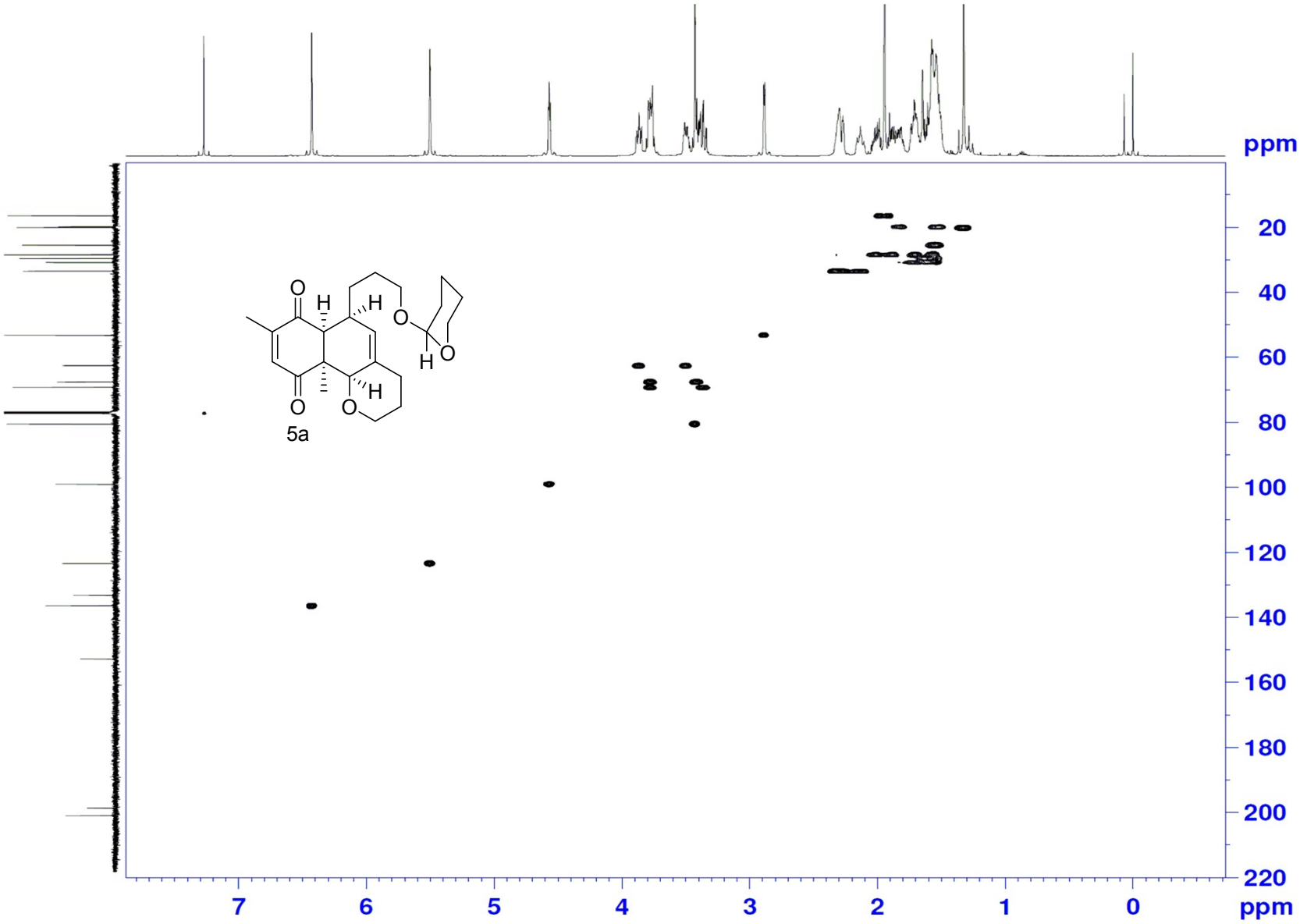


Figure 29. HSQC NMR (500 MHz, $CDCl_3$, 300K) of **5a**

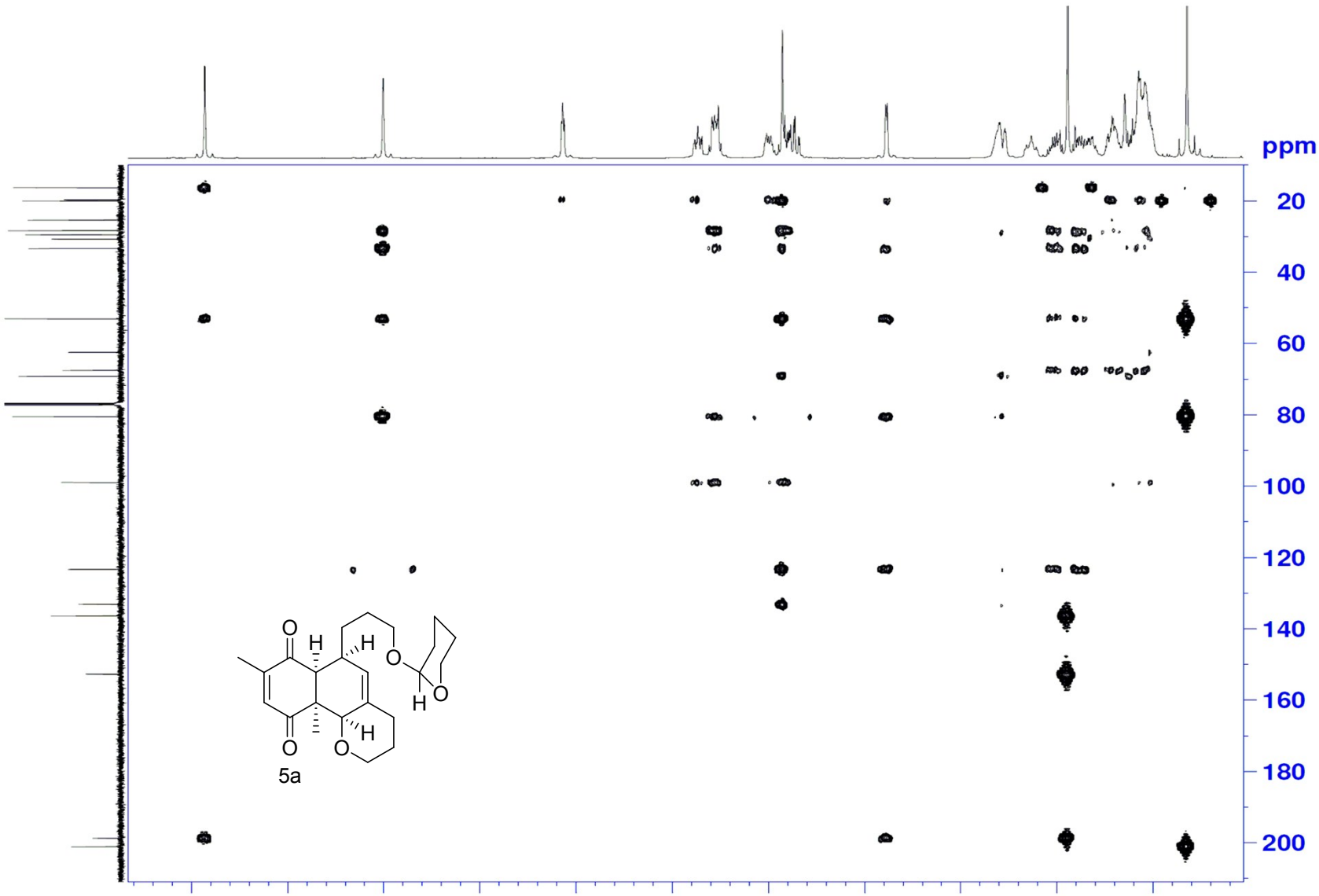


Figure 30. HMBC NMR (500 MHz, $CDCl_3$, 300K) of **5a**

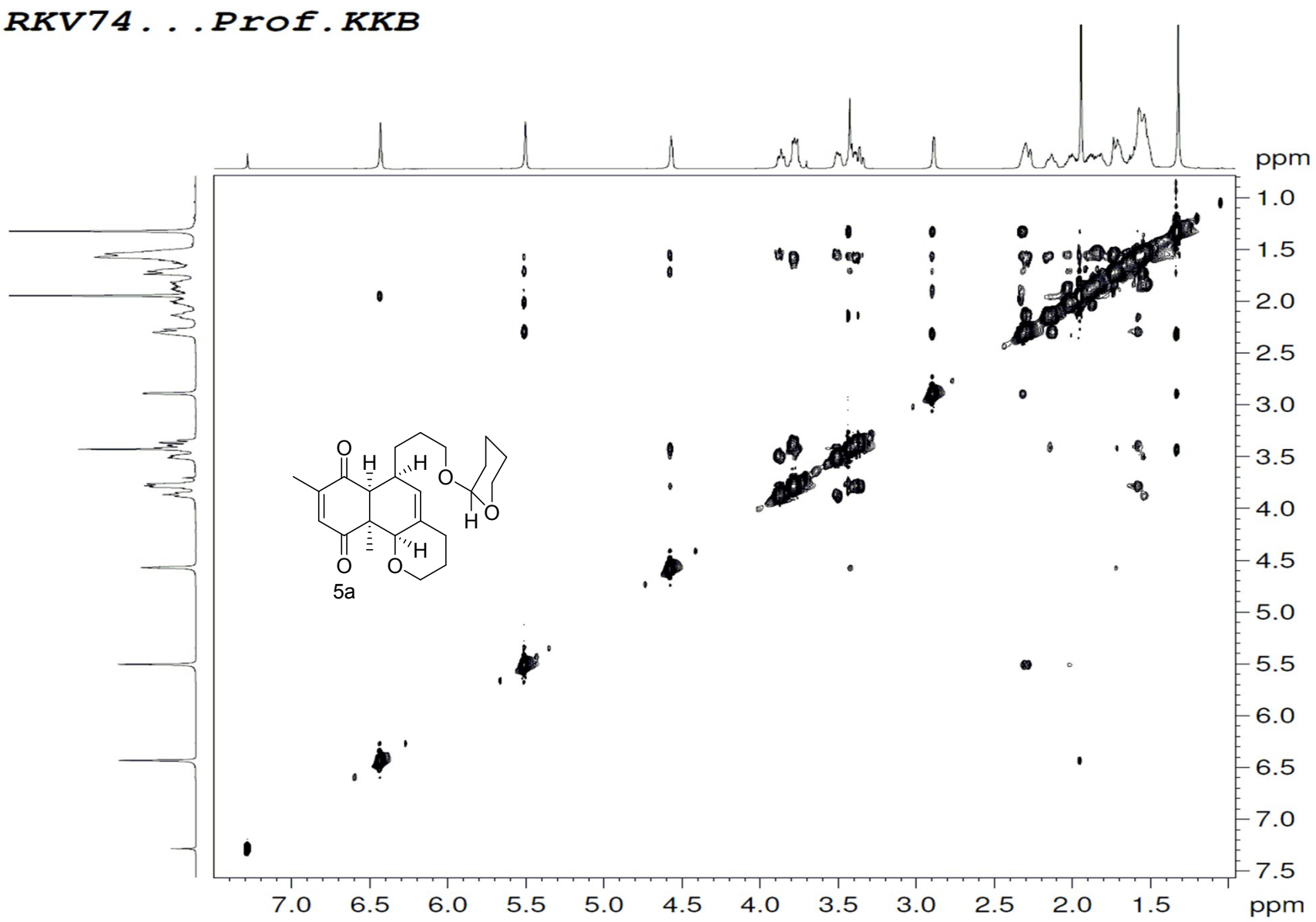
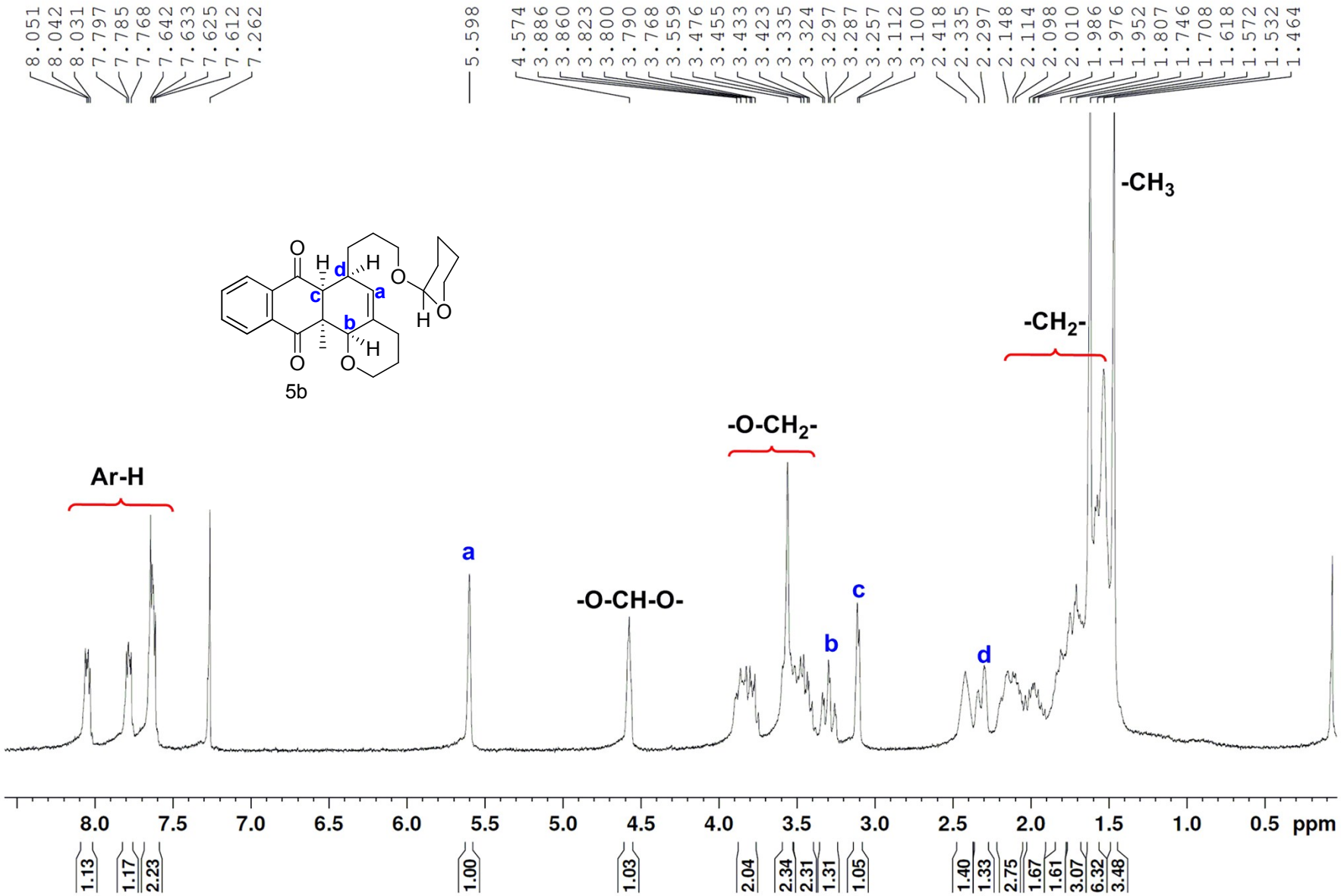


Figure 31. NOSY NMR (500 MHz, $CDCl_3$, 300K) of **5a**



5b

Figure 32. ^{1}H NMR (400 MHz, $CDCl_3$, 300K) of **5b** (TMS added as internal standard)

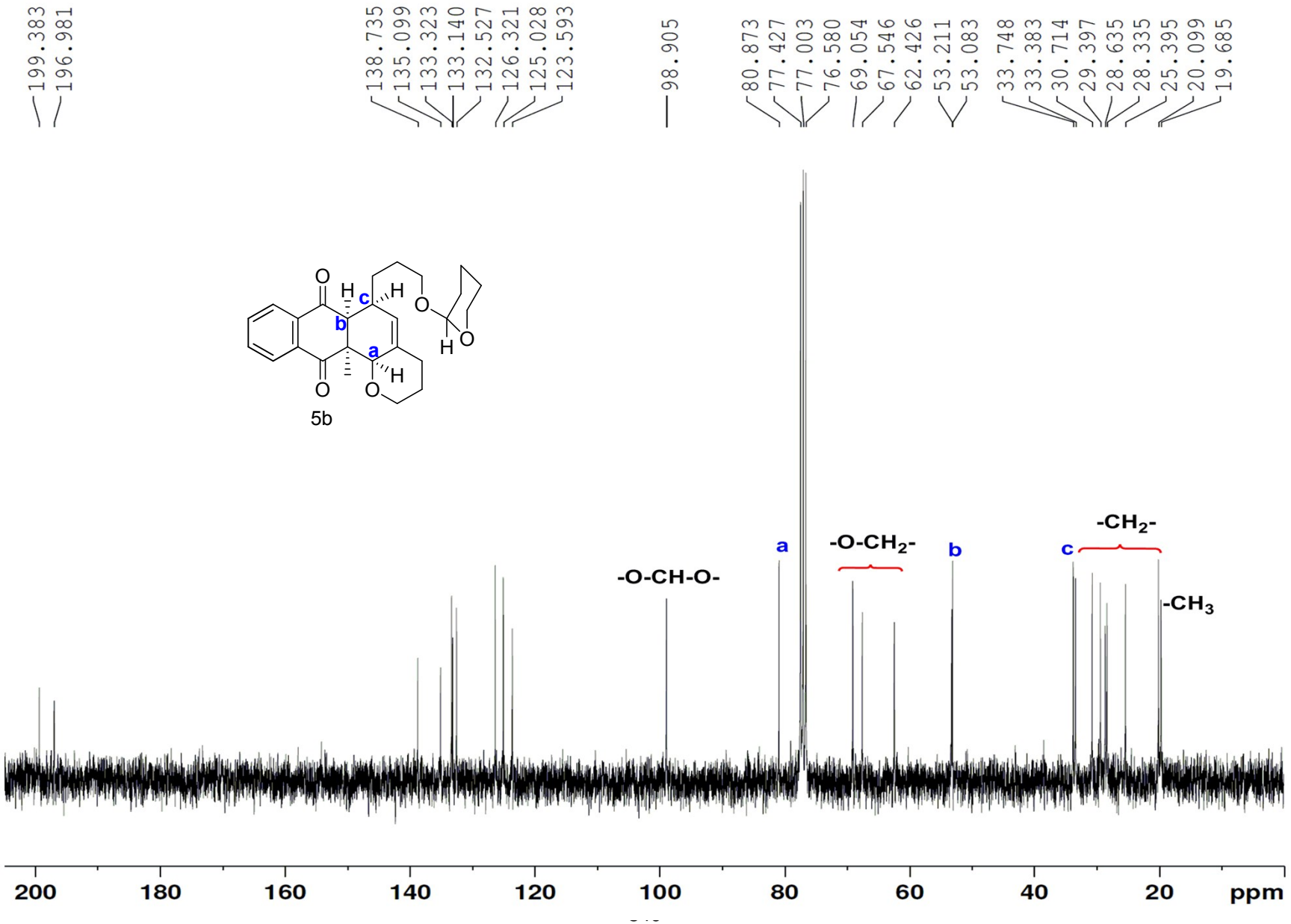


Figure 33. ^{13}C NMR (100 MHz, $CDCl_3$, 300K) of **5b**

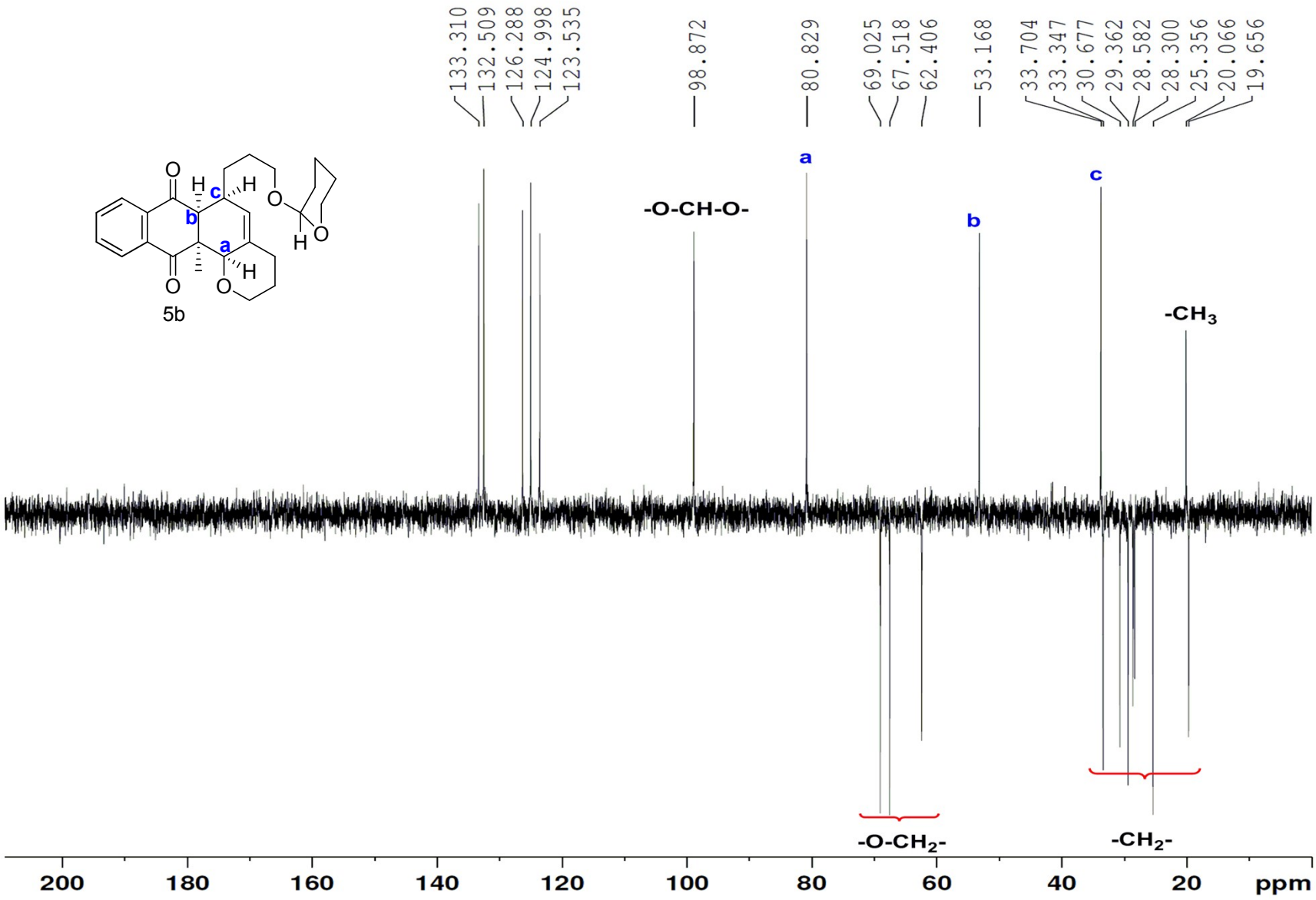


Figure 34. DEPT NMR (100 MHz, $CDCl_3$, 300K) of **5b**

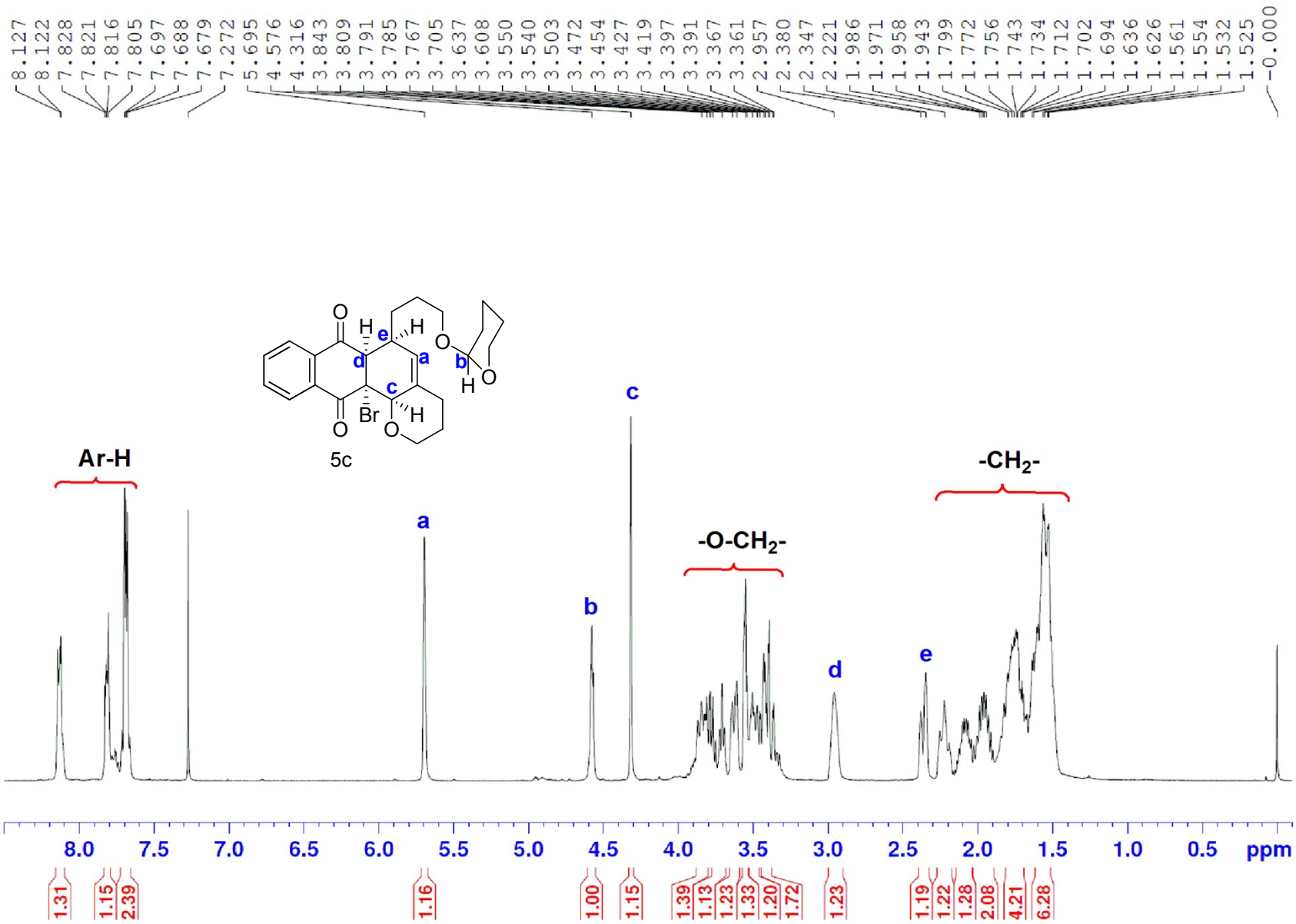


Figure 35. ^{1}H NMR (400 MHz, $CDCl_3$, 300K) of **5c** (TMS added as internal standard)

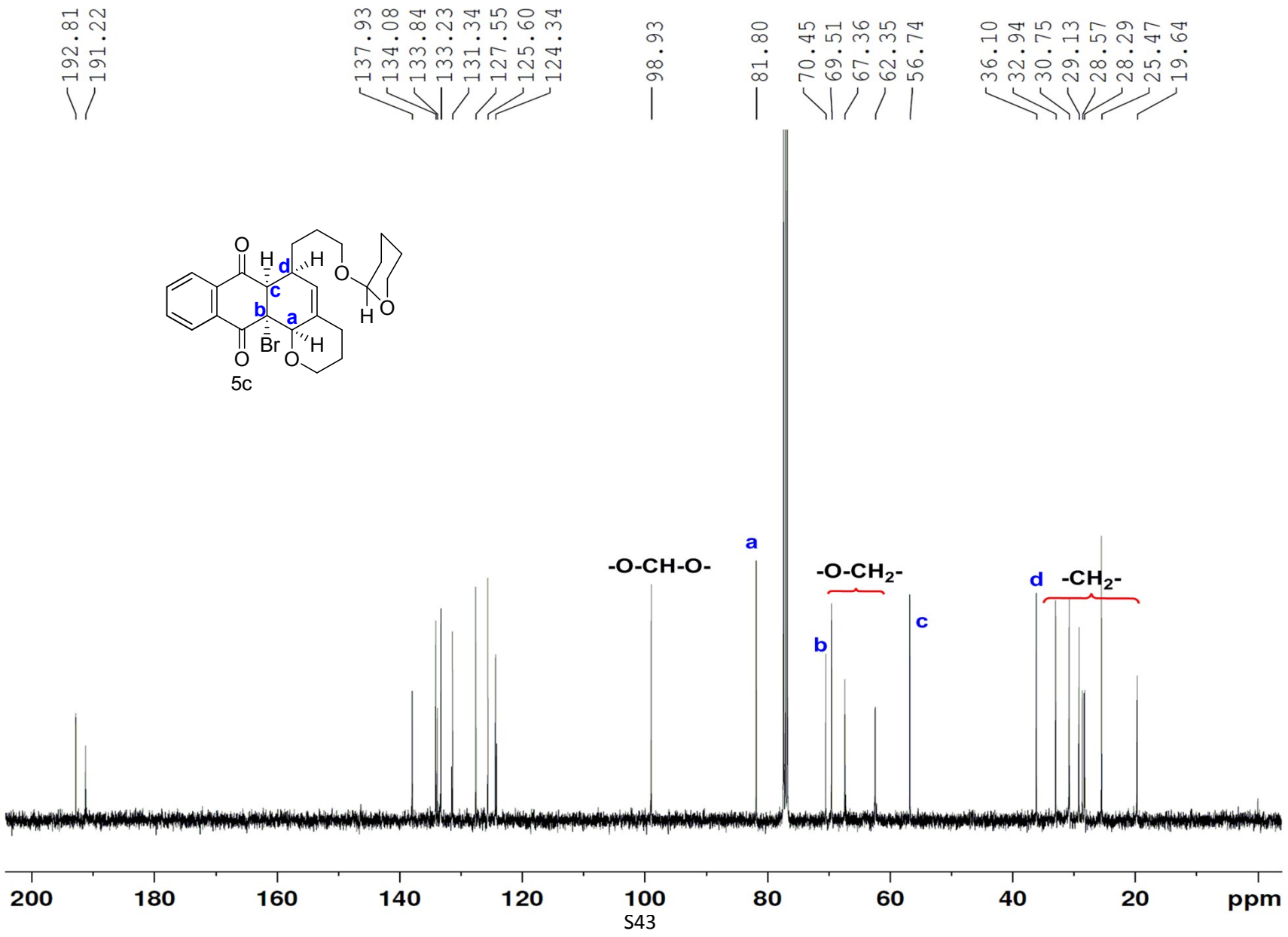


Figure 36. ^{13}C NMR (100 MHz, $CDCl_3$, 300K) of **5c**

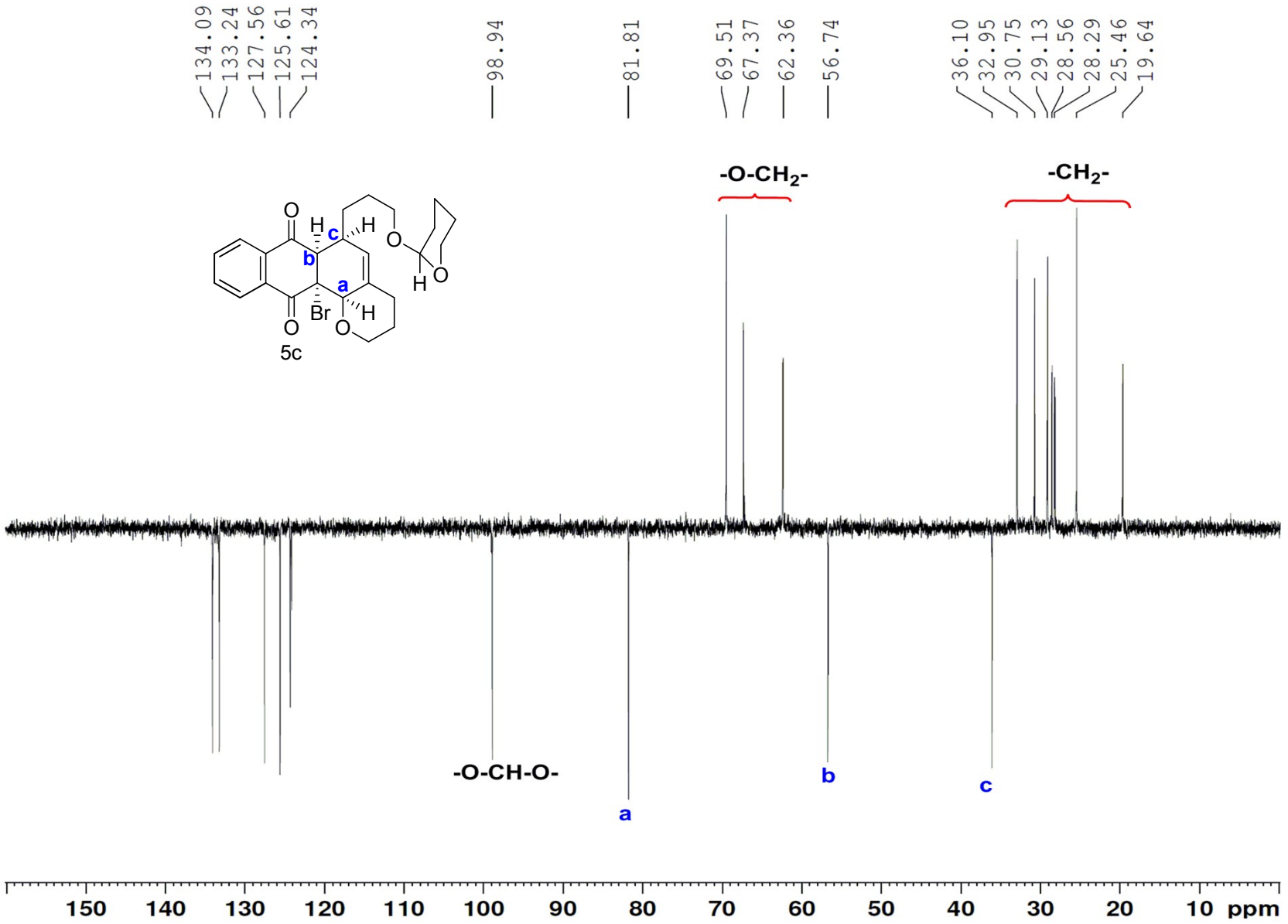
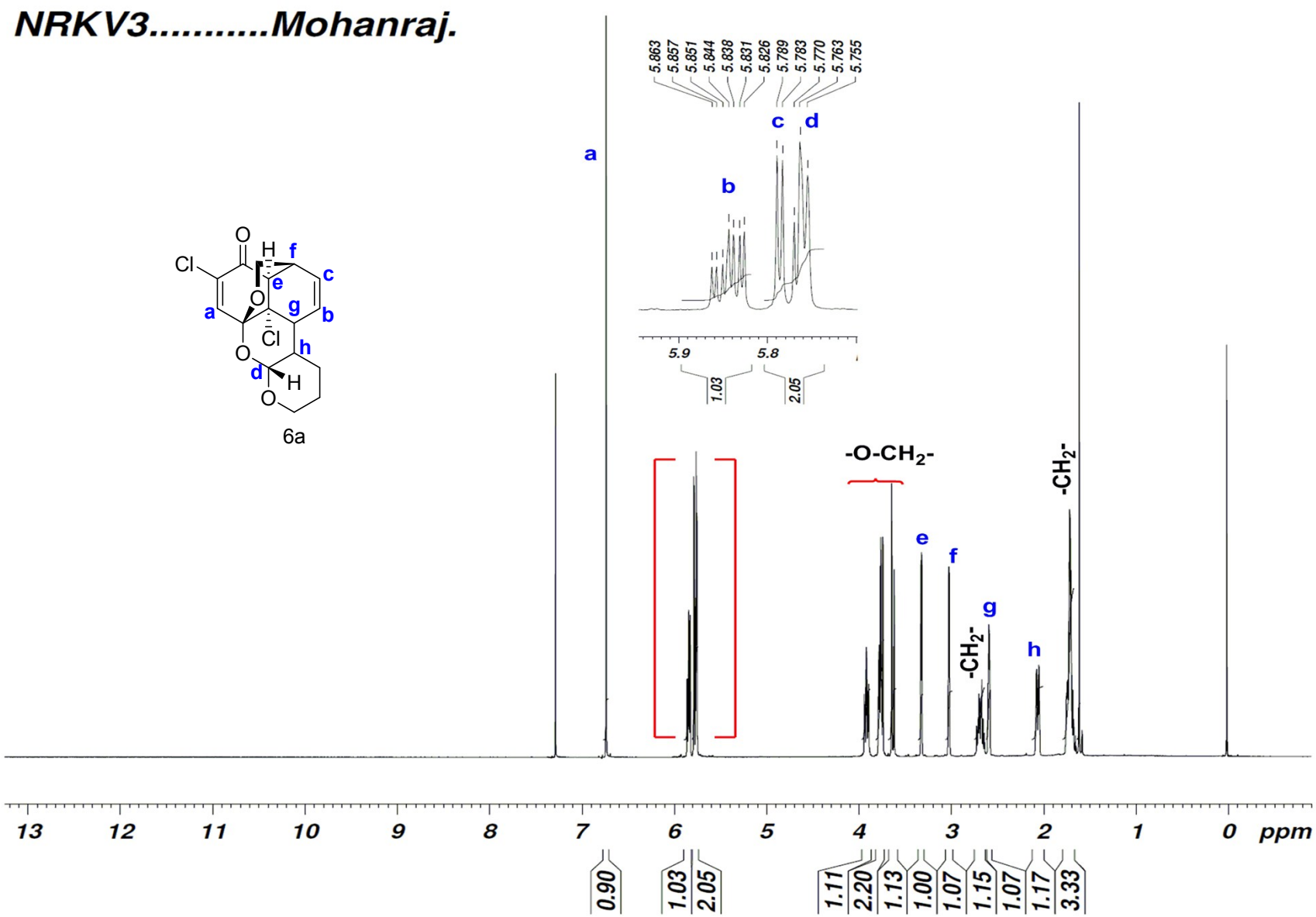


Figure 37. DEPT NMR (100 MHz, $CDCl_3$, 300K) of **5c**



6a

Figure 38. ^{1}H NMR (500 MHz, $CDCl_3$, 300K) of **6a** (TMS added as internal standard)

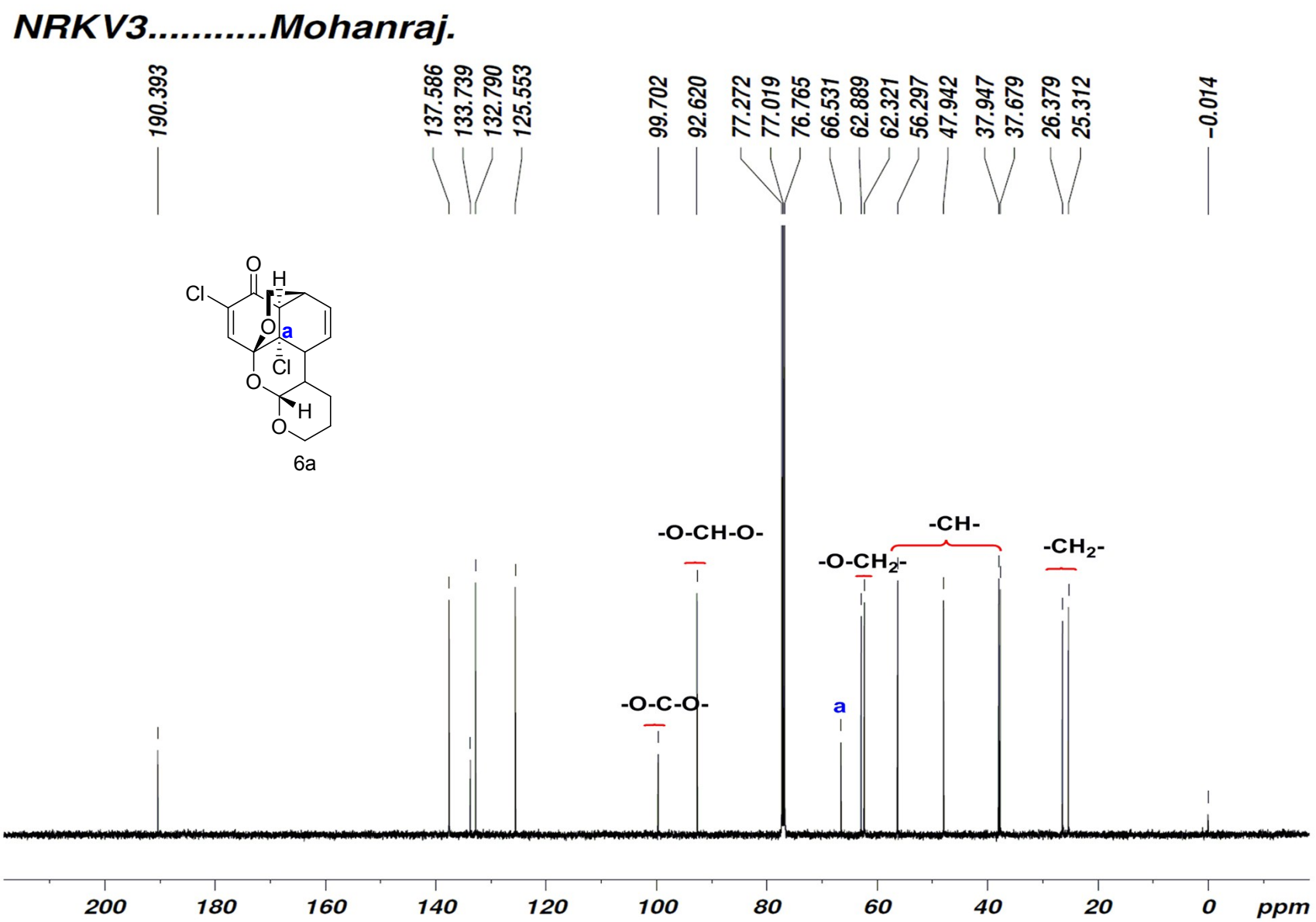


Figure 39. ^{13}C NMR (125 MHz, $CDCl_3$, 300K) of **6a**

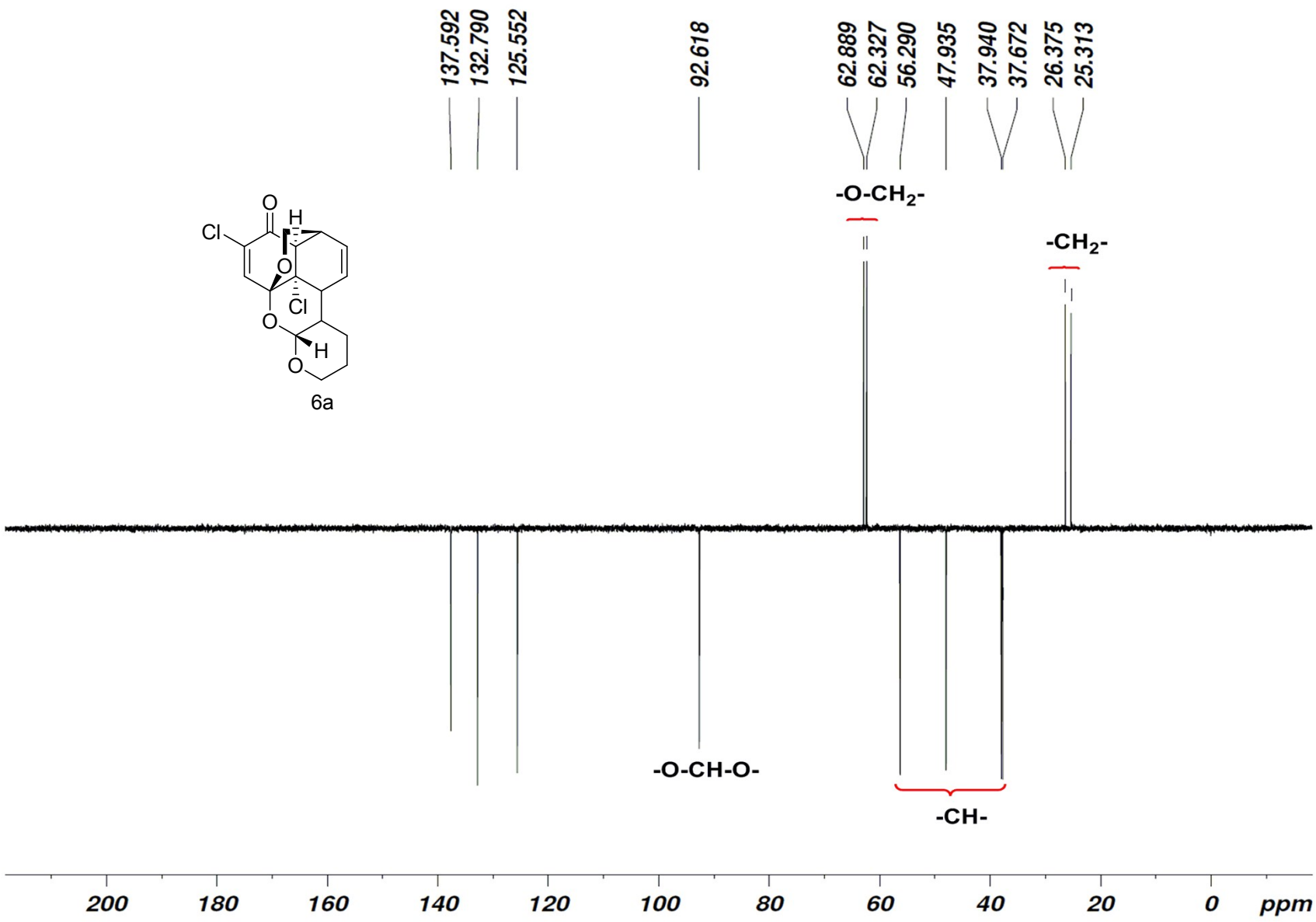


Figure 40. DEPT NMR (125 MHz, $CDCl_3$, 300K) of **6a**

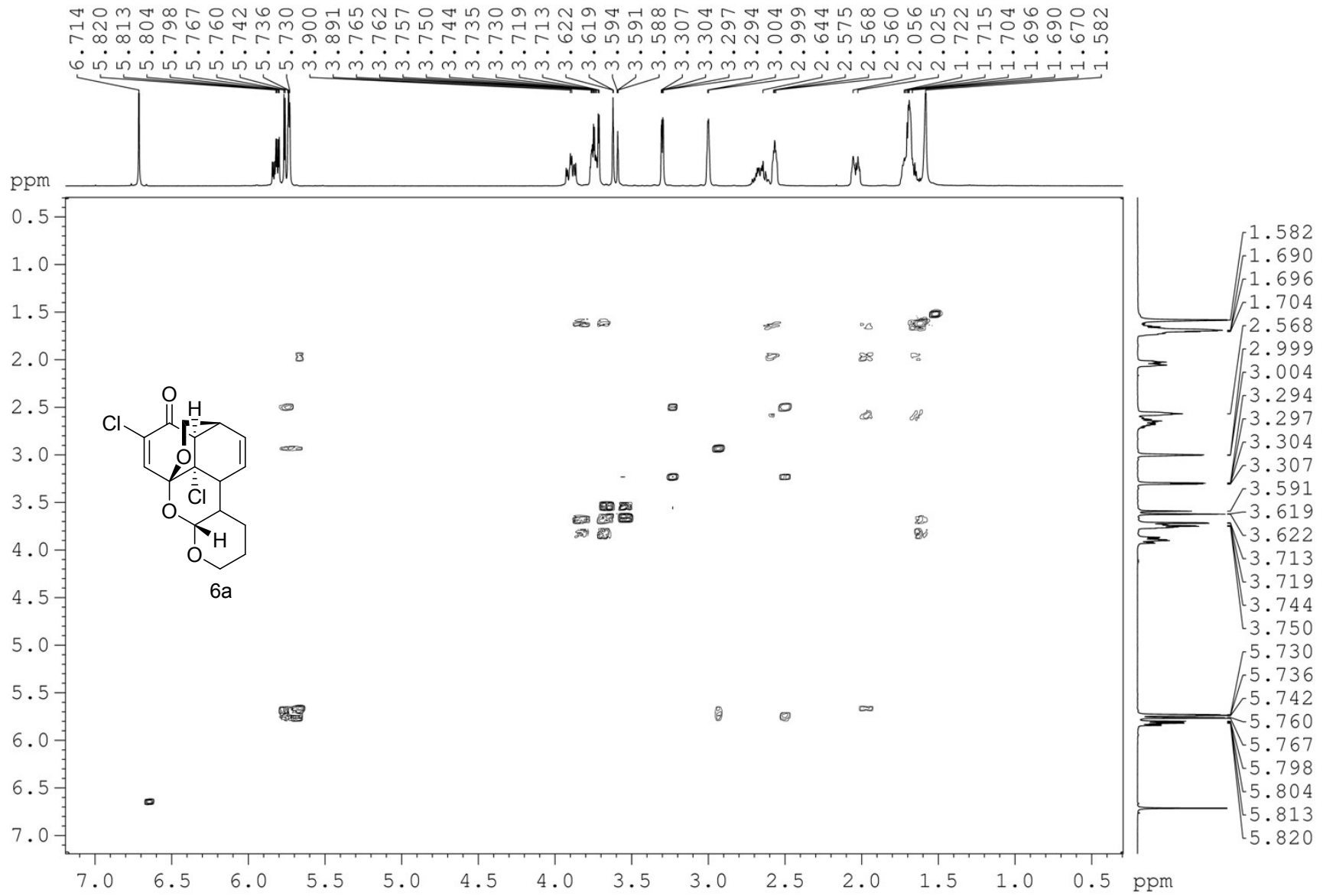


Figure 41. COSY NMR (500 MHz, $CDCl_3$, 300K) of **6a**

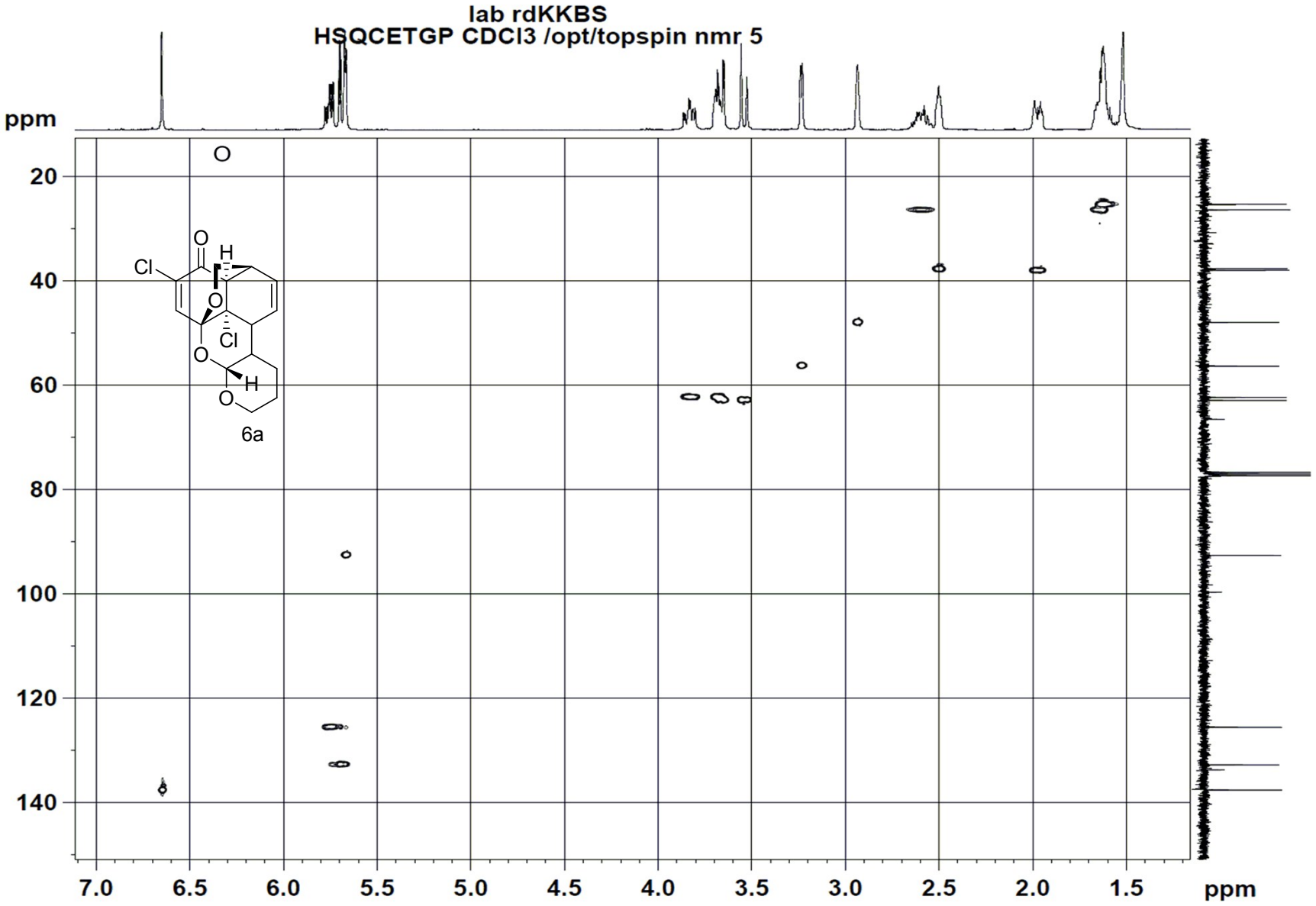


Figure 42. HSQC NMR (500 MHz, $CDCl_3$, 300K) of **6a**

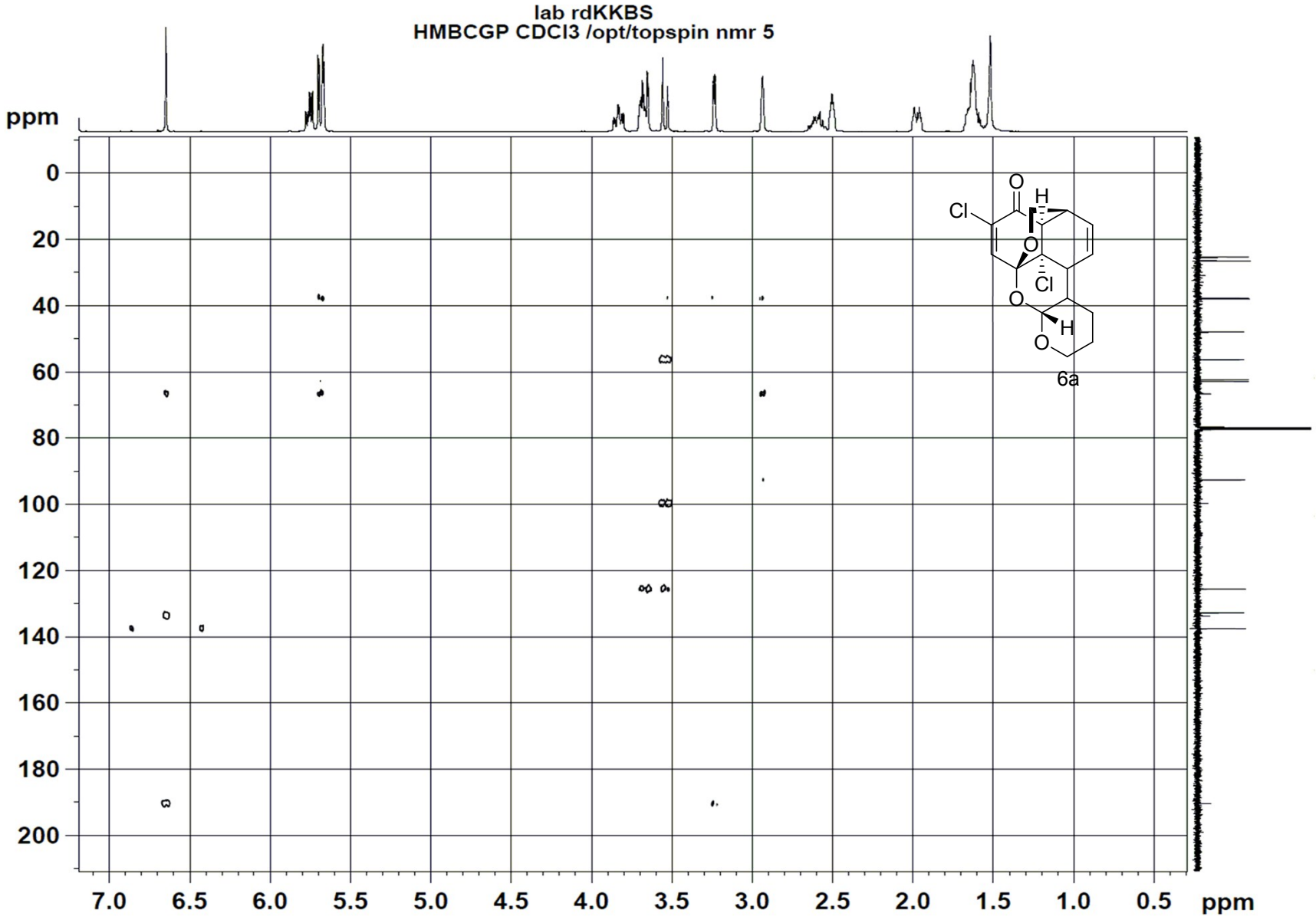


Figure 43. HMBC NMR (500 MHz, $CDCl_3$, 300K) of **6a**

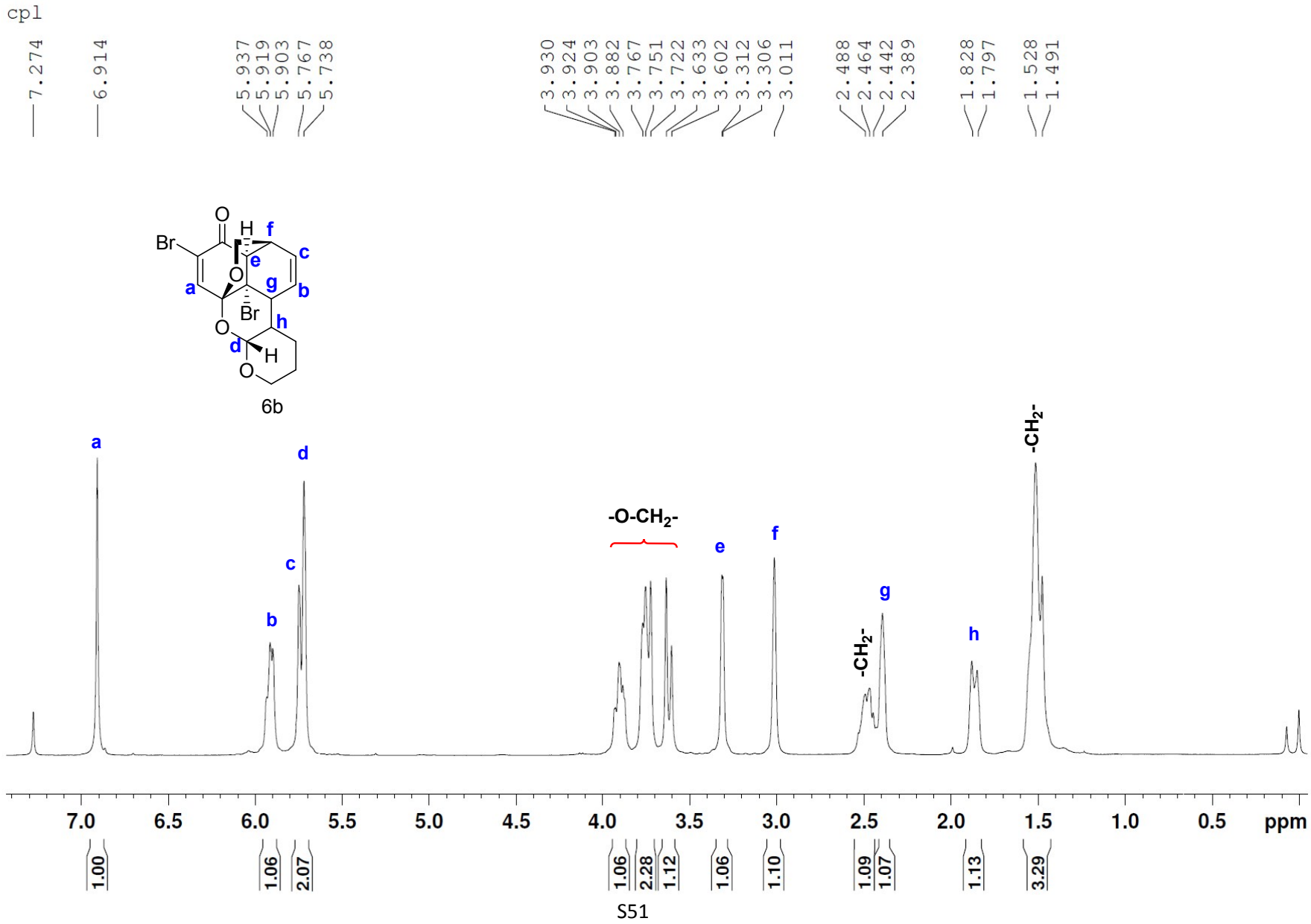


Figure 44. ^{1}H NMR (400 MHz, $CDCl_3$, 300K) of **6b** (TMS added as internal standard)

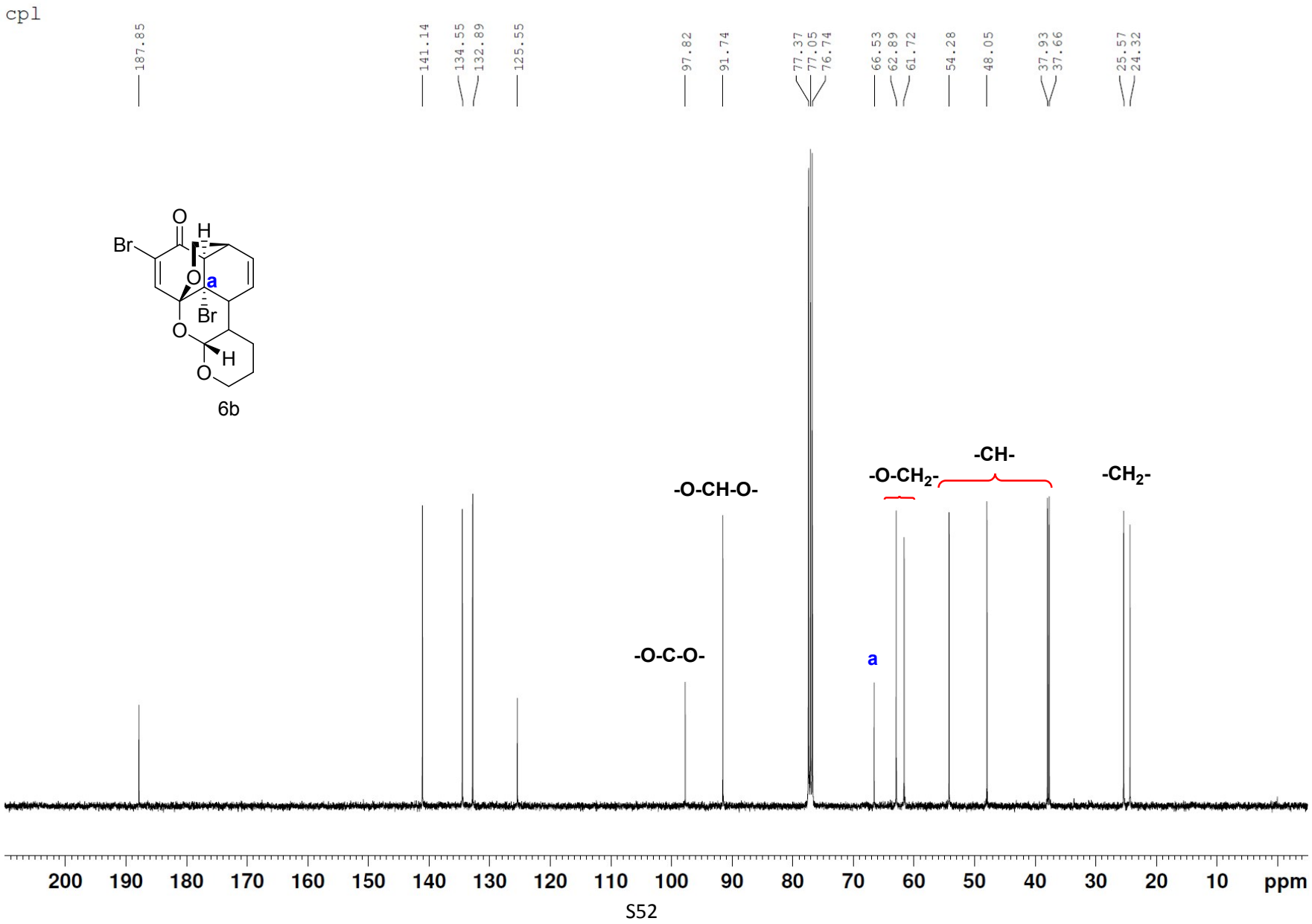


Figure 45. ^{13}C NMR (100 MHz, $CDCl_3$, 300K) of **6b**

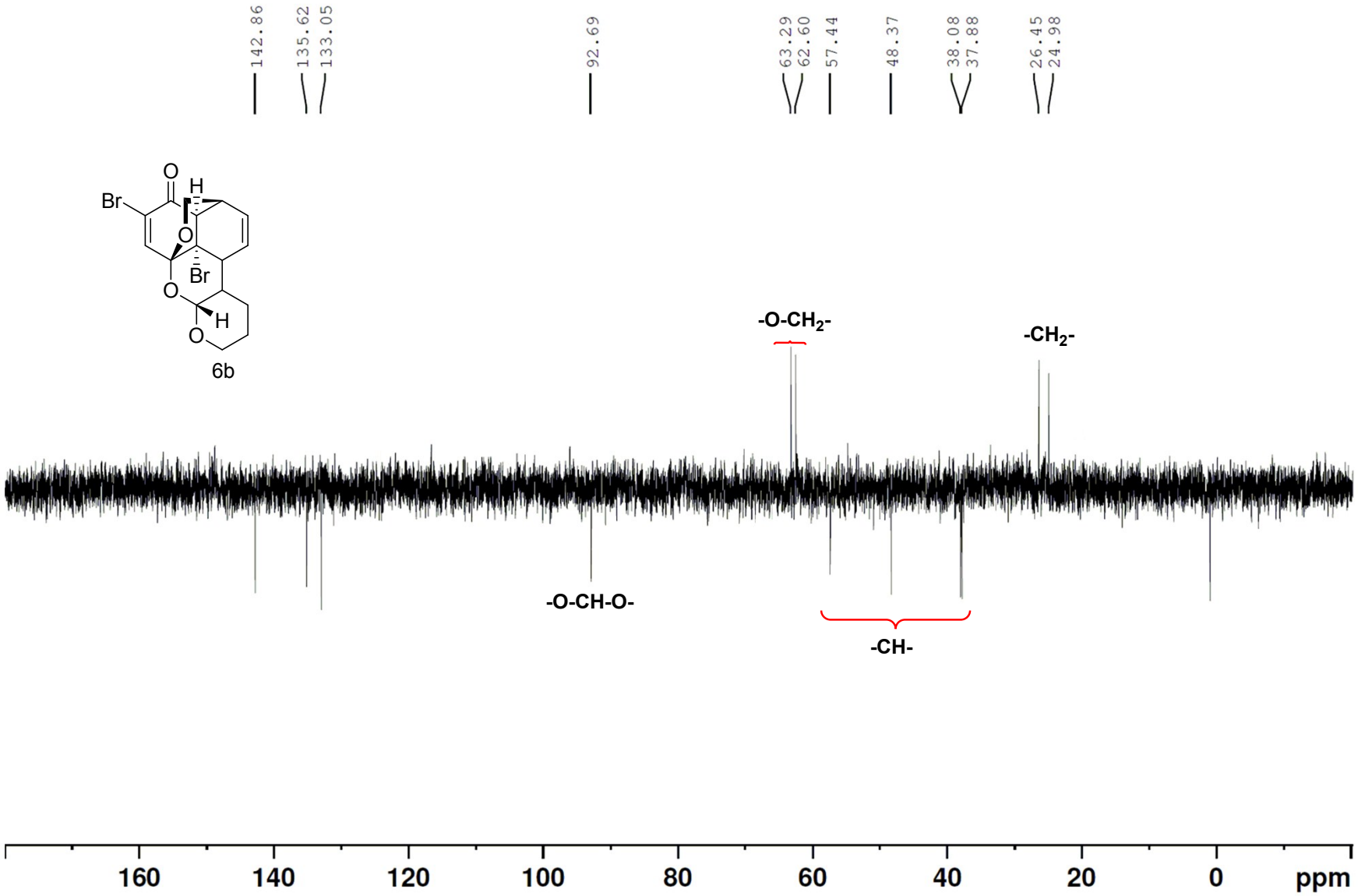


Figure 46. DEPT NMR (100 MHz, $CDCl_3$, 300K) of **6b**

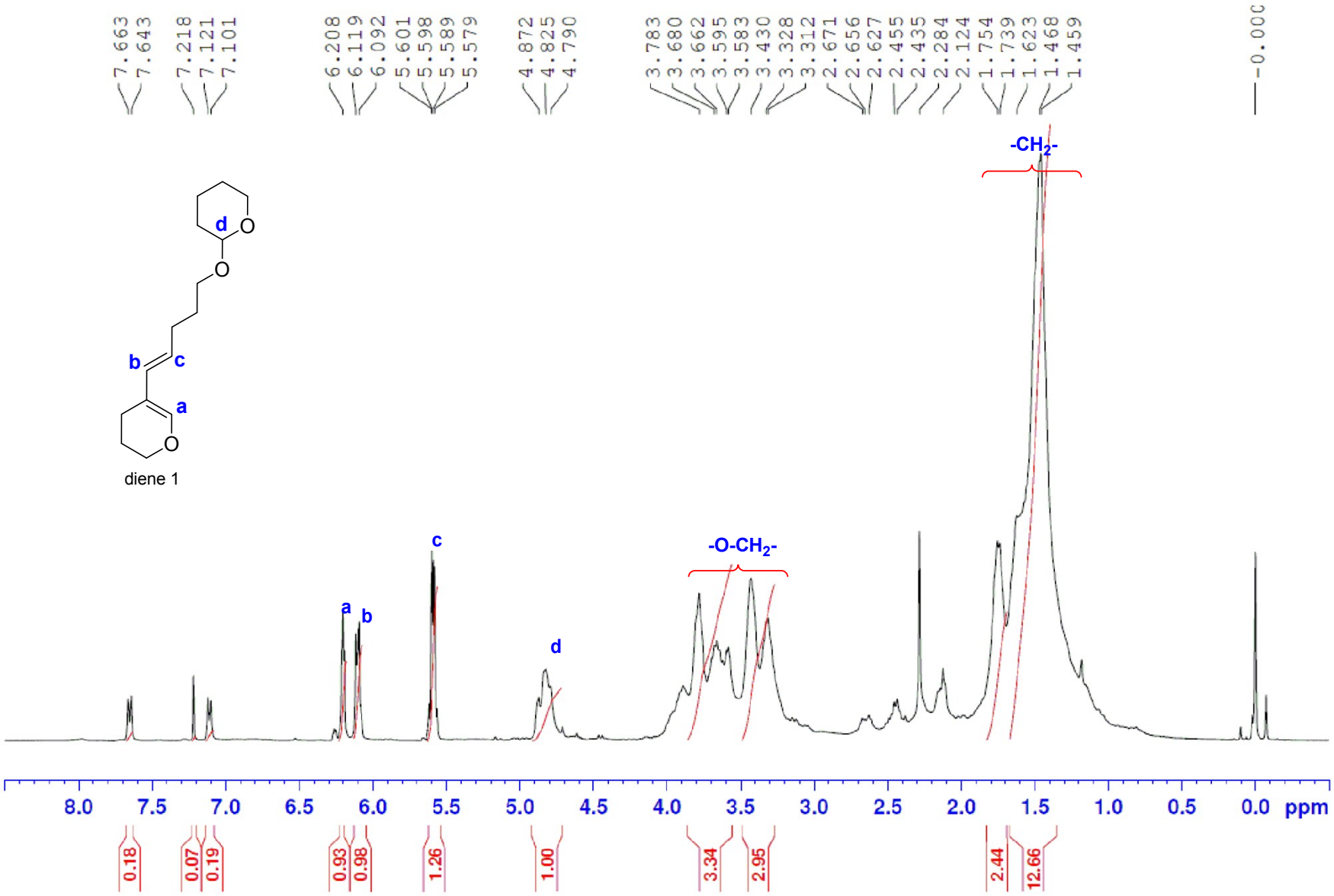


Figure 47. Crude - ^{1}H NMR (400 MHz, $CDCl_3$, 300K) of **Diene 1 contaminated with *p*TsOH** (TMS added as internal standard)

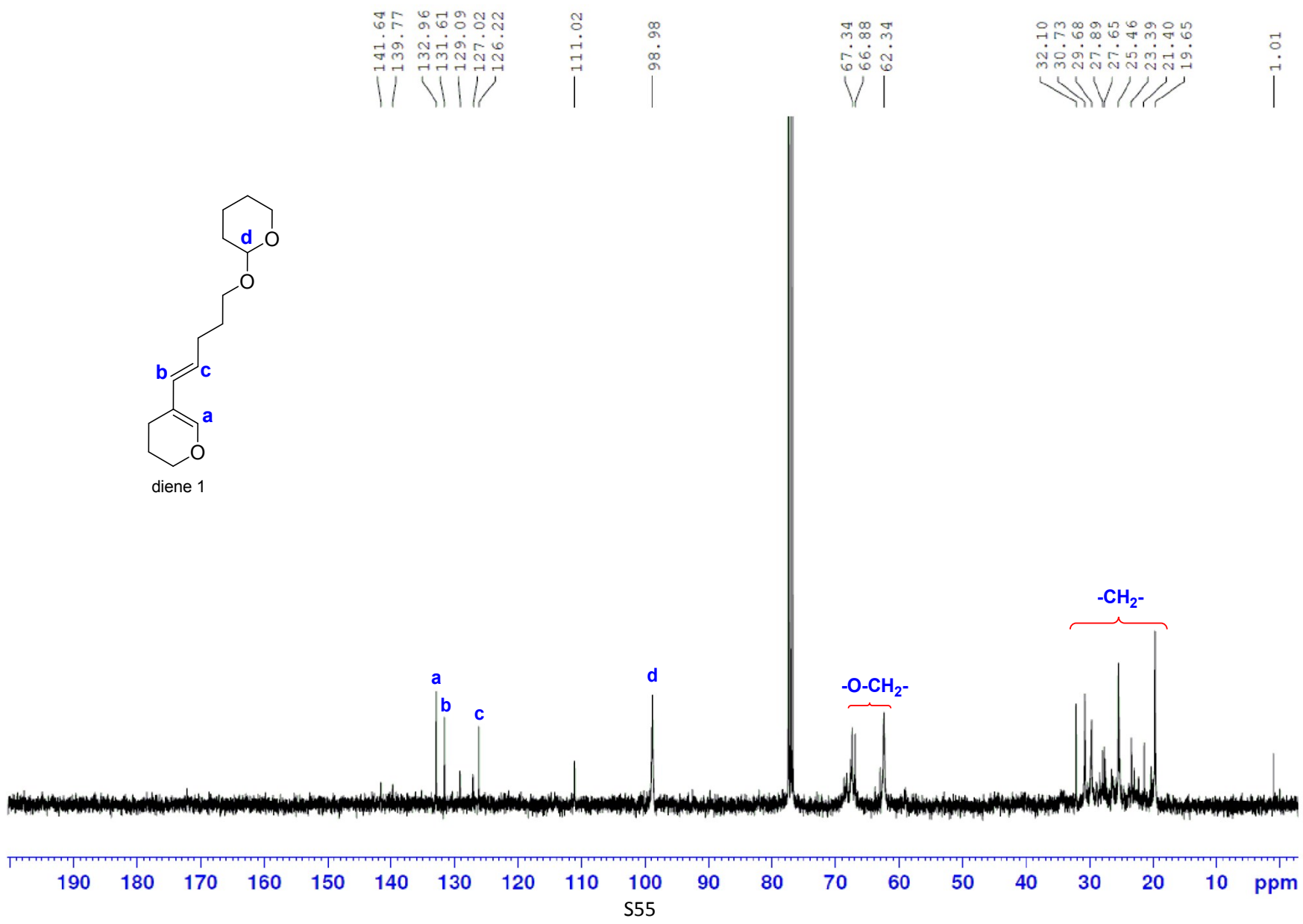


Figure 48. Crude - ^{13}C NMR (100 MHz, $CDCl_3$, 300K) of **Diene 1 contaminated with *p*TsOH**

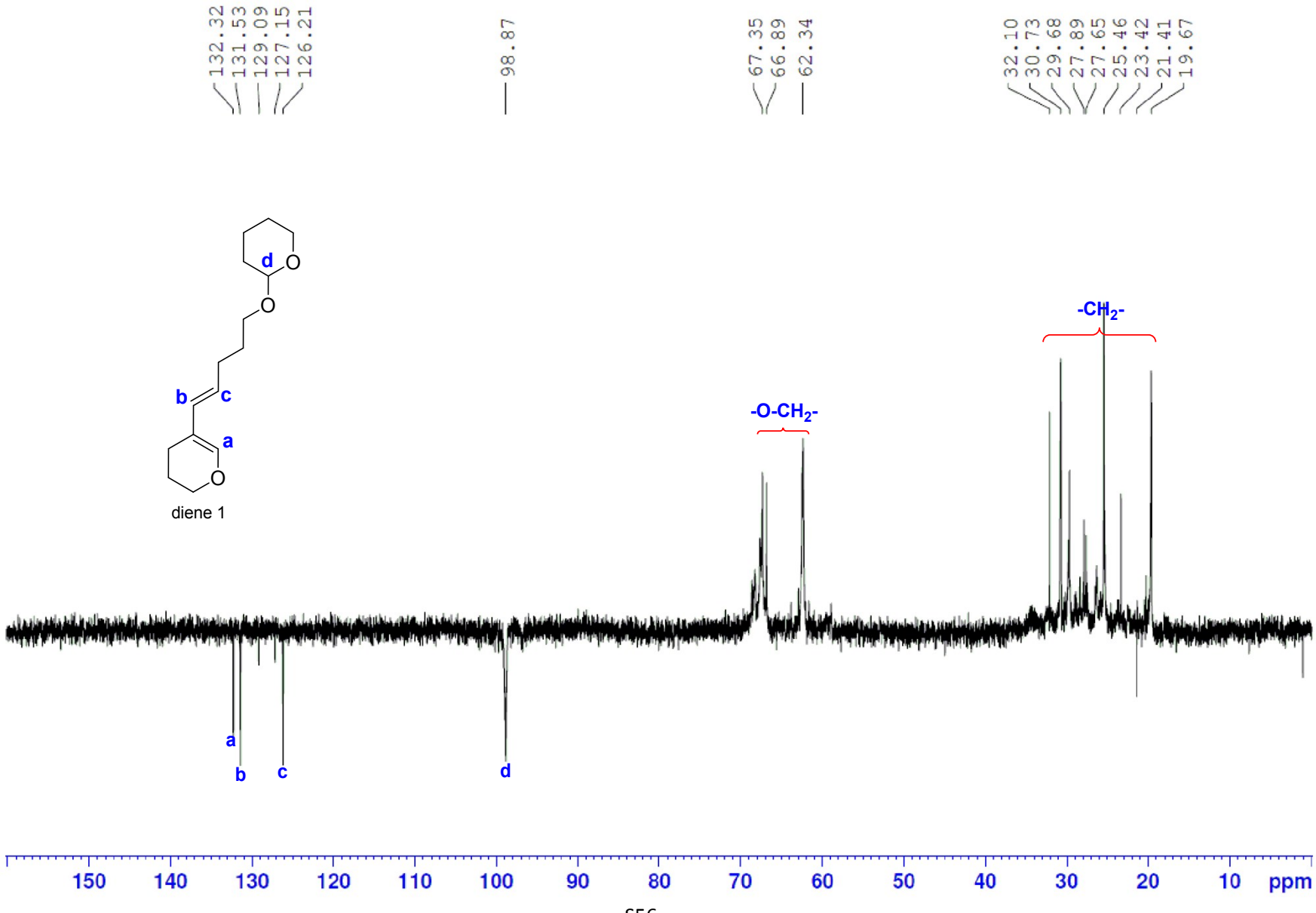


Figure 49. Crude - DEPT NMR (100 MHz, $CDCl_3$, 300K) of **Diene 1 contaminated with *p*TsOH**

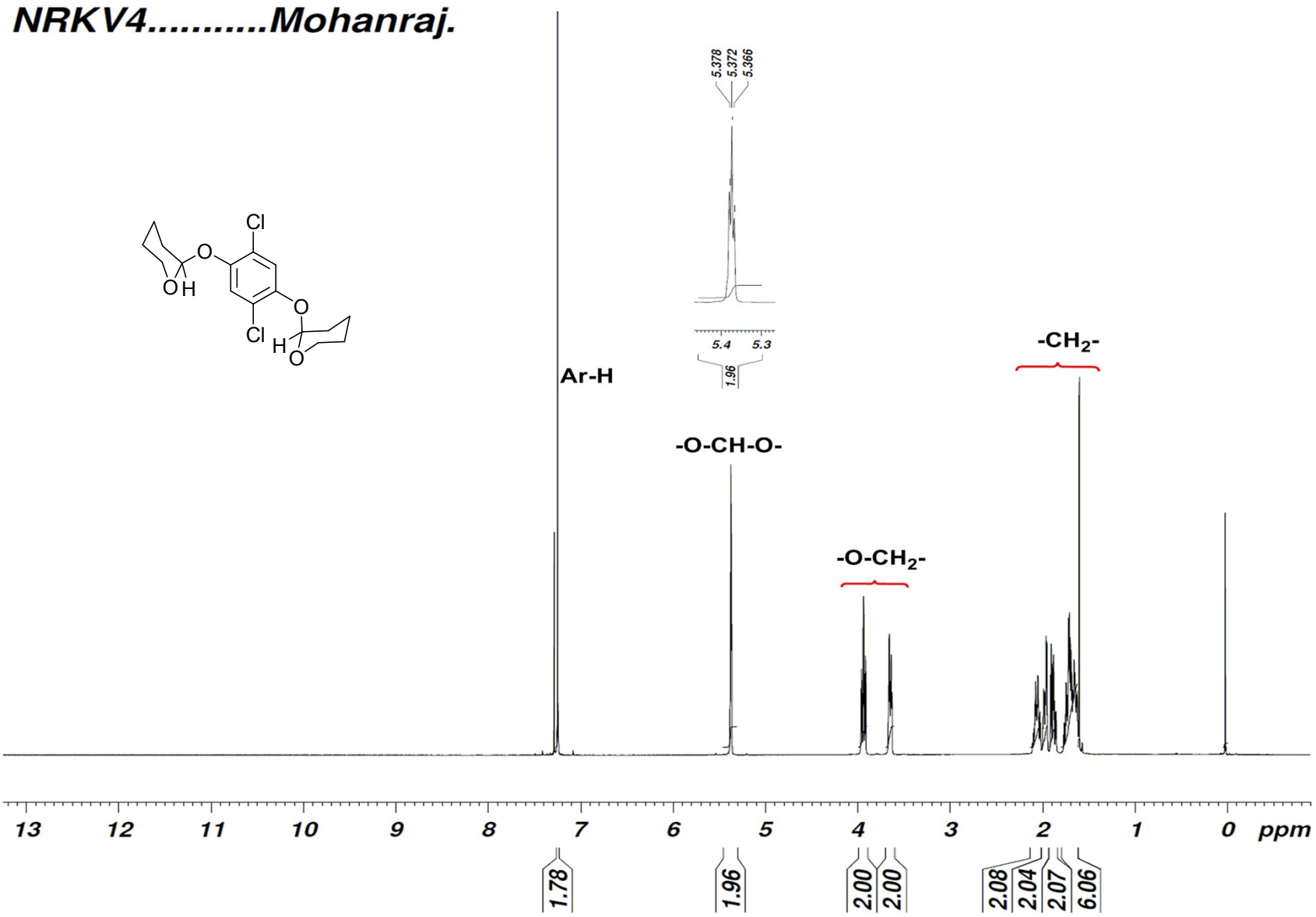


Figure 50. ^{1}H NMR (500 MHz, $CDCl_3$, 300K) of **2,5-dichloro-1,4-Bis(tetrahydro-2*H*-pyran-2-yloxy)benzene** (TMS added as internal standard)

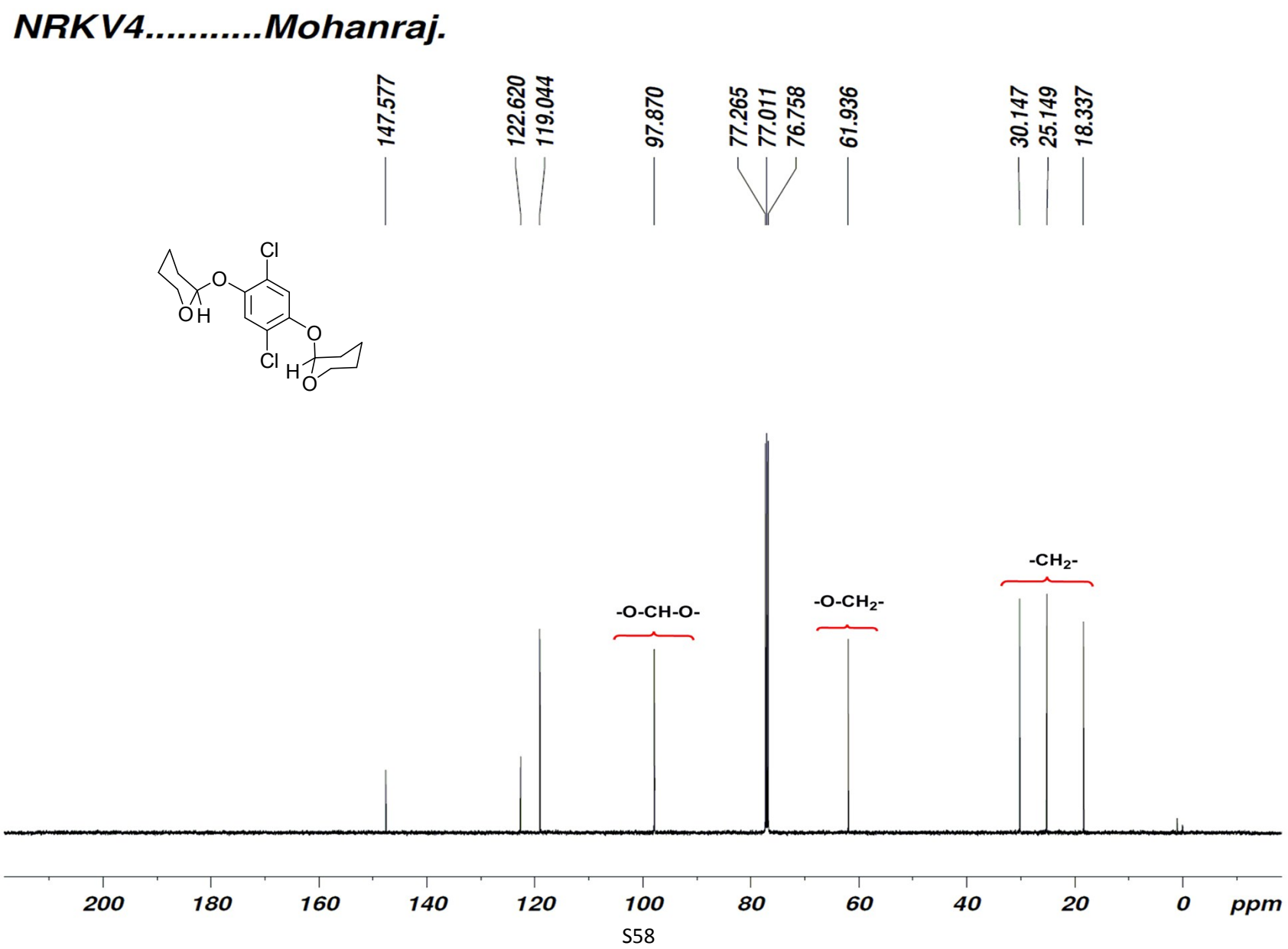


Figure 51. ^{13}C NMR (125 MHz, $CDCl_3$, 300K) of **2,5-dichloro-1,4-Bis(tetrahydro-2*H*-pyran-2-yloxy)benzene**

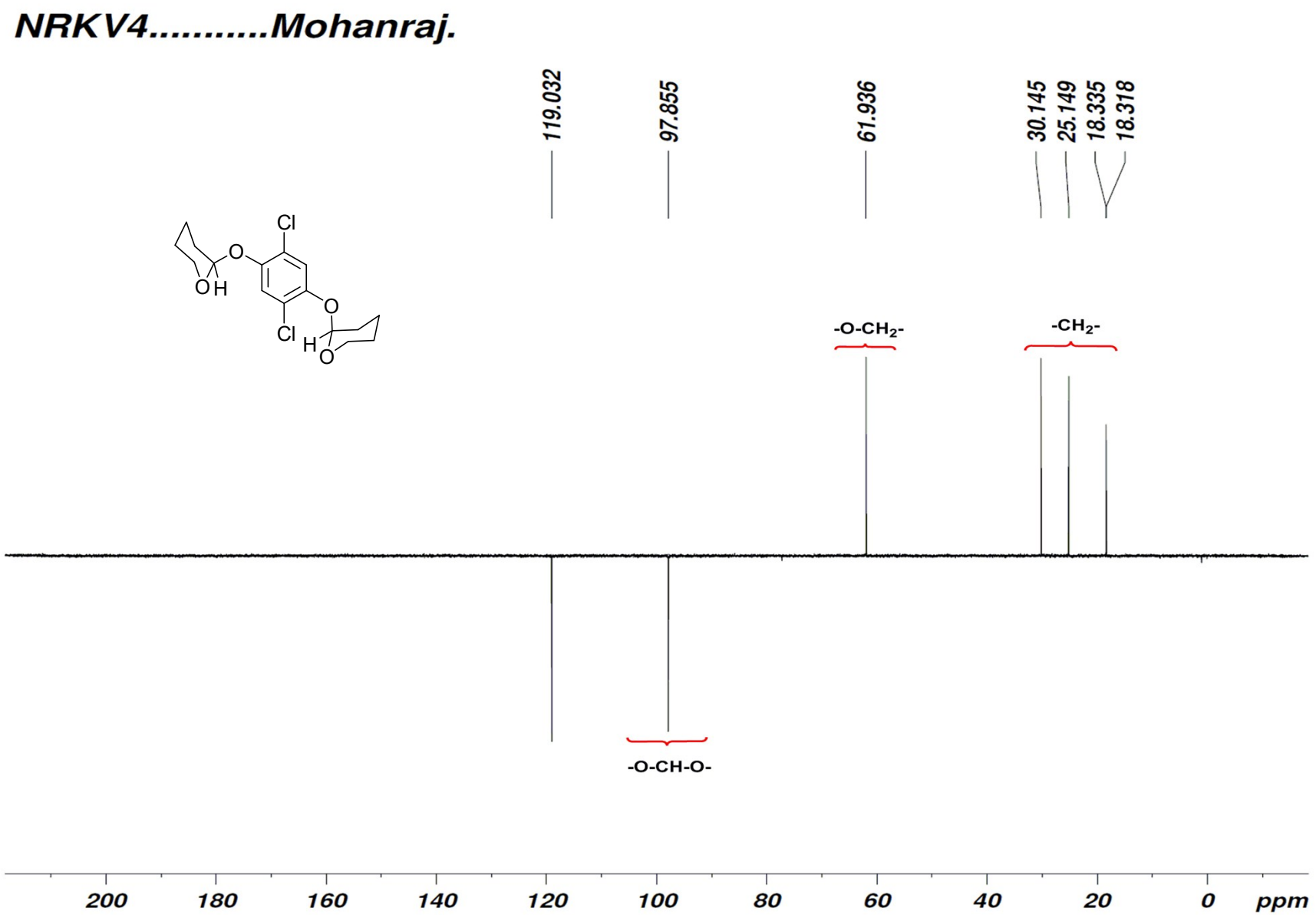


Figure 52. DEPT NMR (125 MHz, $CDCl_3$, 300K) of **2,5-dichloro-1,4-Bis(tetrahydro-2*H*-pyran-2-yloxy)benzene**

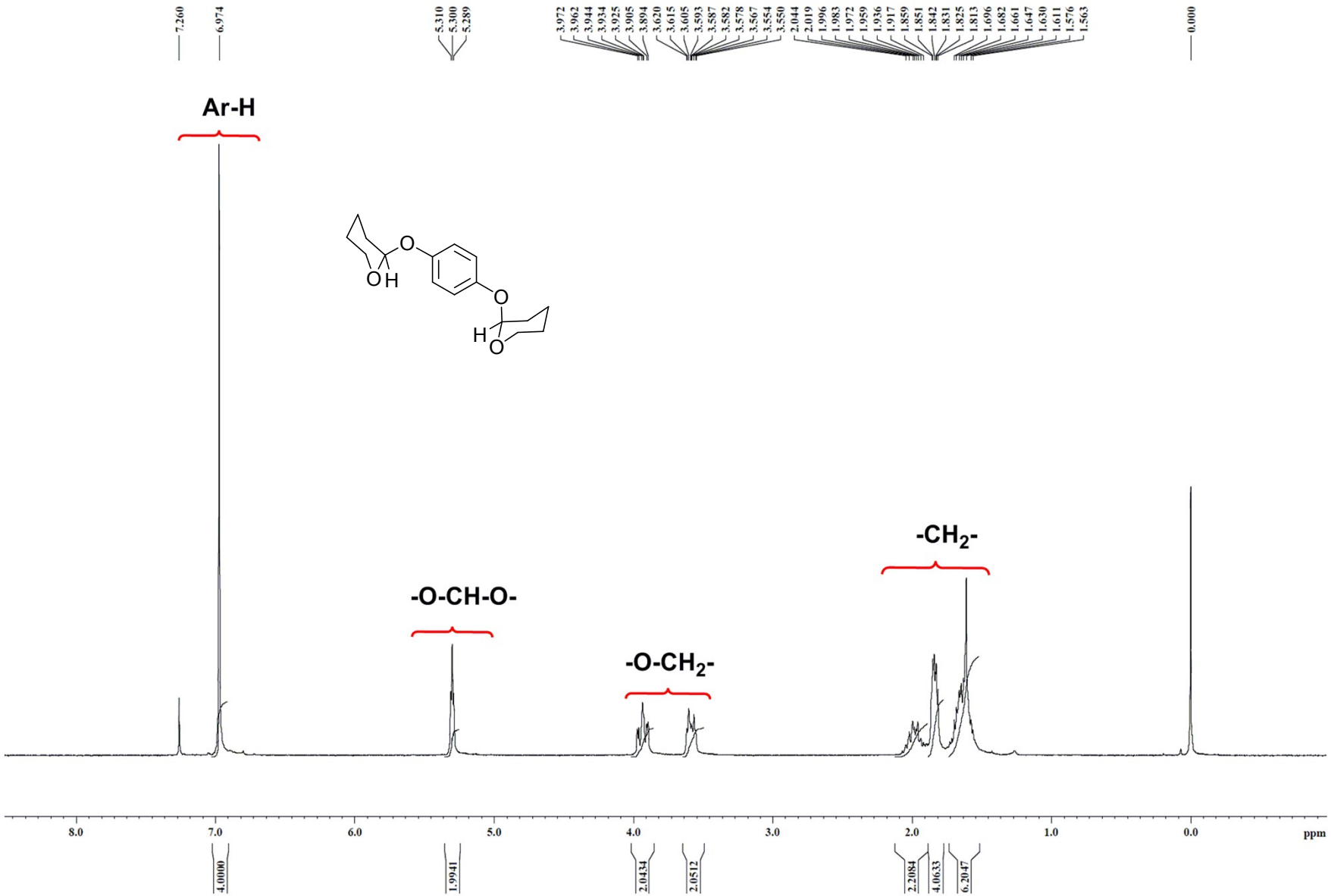


Figure 53. ^{1}H NMR (300 MHz, $CDCl_3$, 300K) of **1,4-Bis(tetrahydro-2*H*-pyran-2-yloxy)benzene** (TMS added as internal standard)[1]

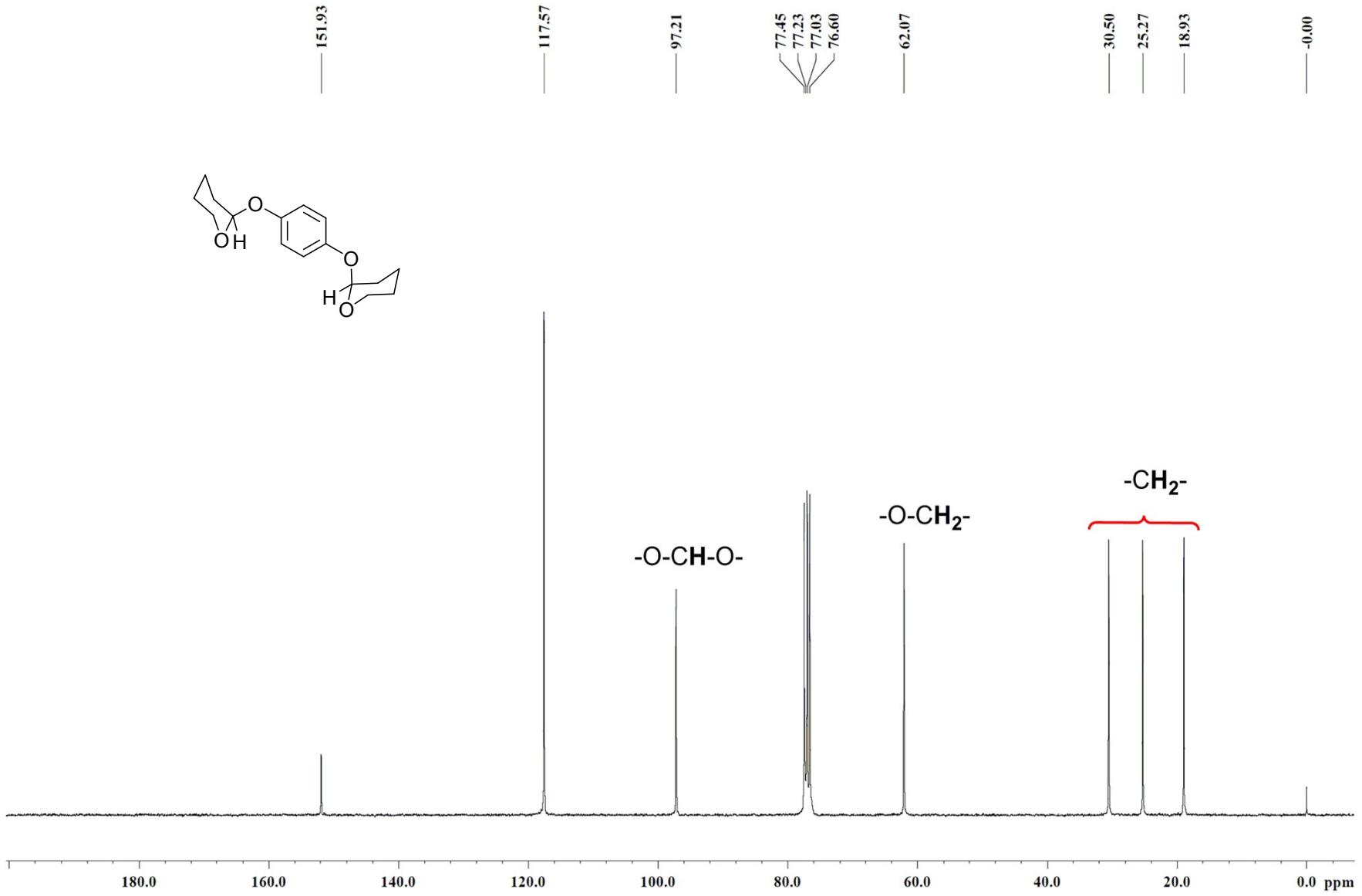


Figure 54. ^{13}C NMR (75 MHz, $CDCl_3$, 300K) of **1,4-Bis(tetrahydro-2*H*-pyran-2-yloxy)benzene**[1]

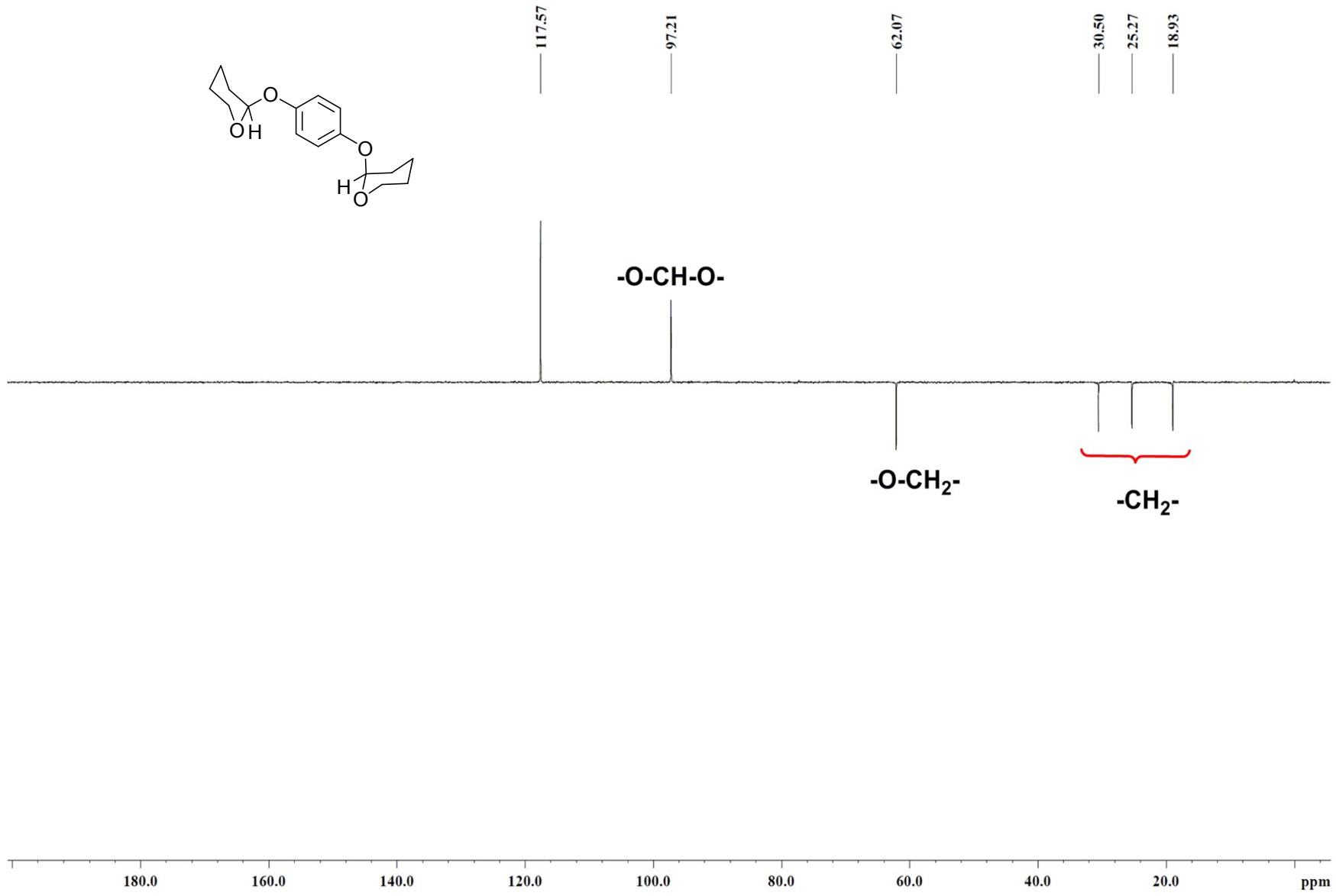


Figure 55. DEPT NMR (75 MHz, $CDCl_3$, 300K) of **1,4-Bis(tetrahydro-2*H*-pyran-2-yloxy)benzene**[1]

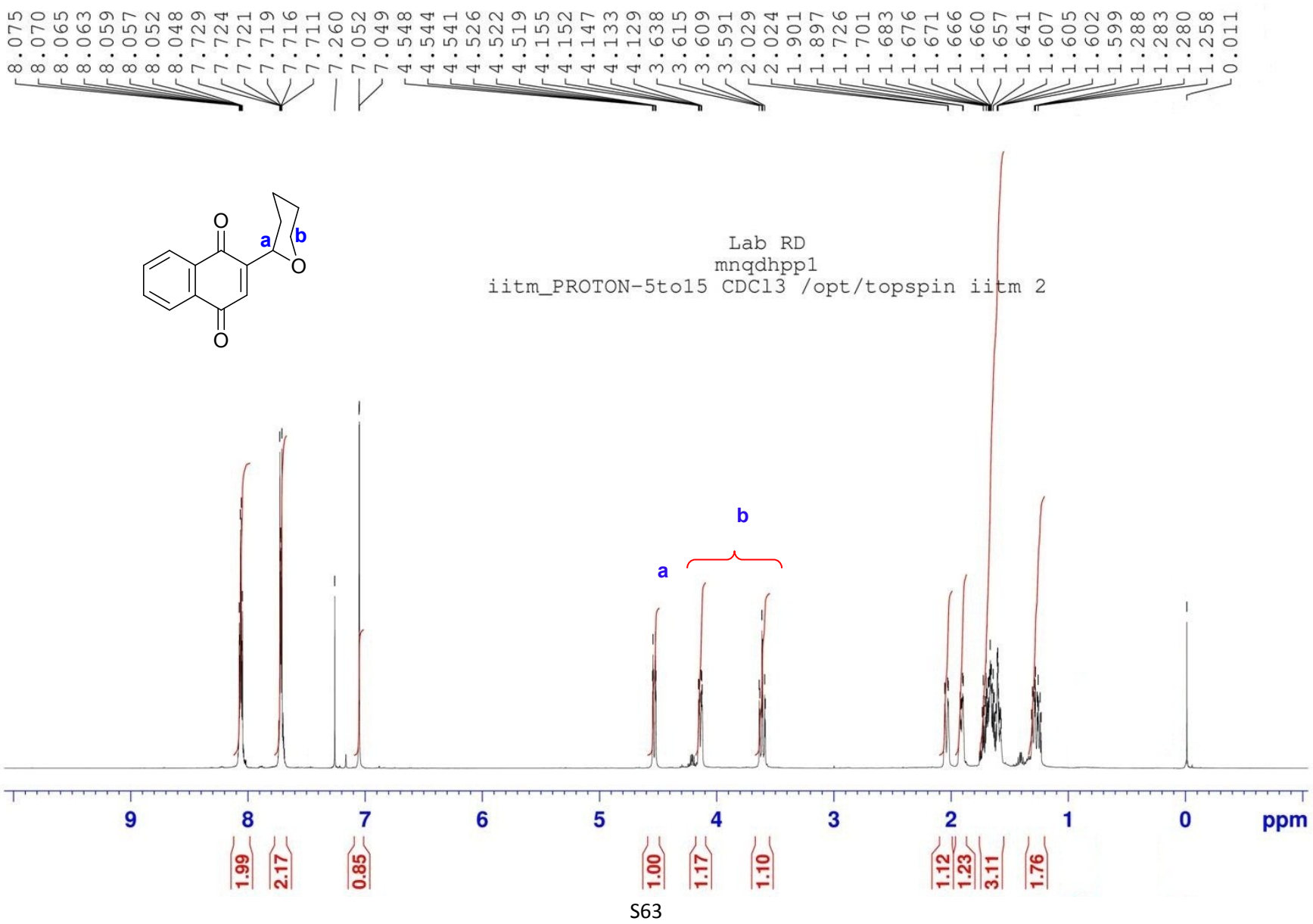


Figure 56. ^{1}H NMR (400 MHz, $CDCl_3$, 300K) of **2-(tetrahydro-2*H*-pyran-2-yl)naphthalene-1,4-dione** (TMS added as internal standard).

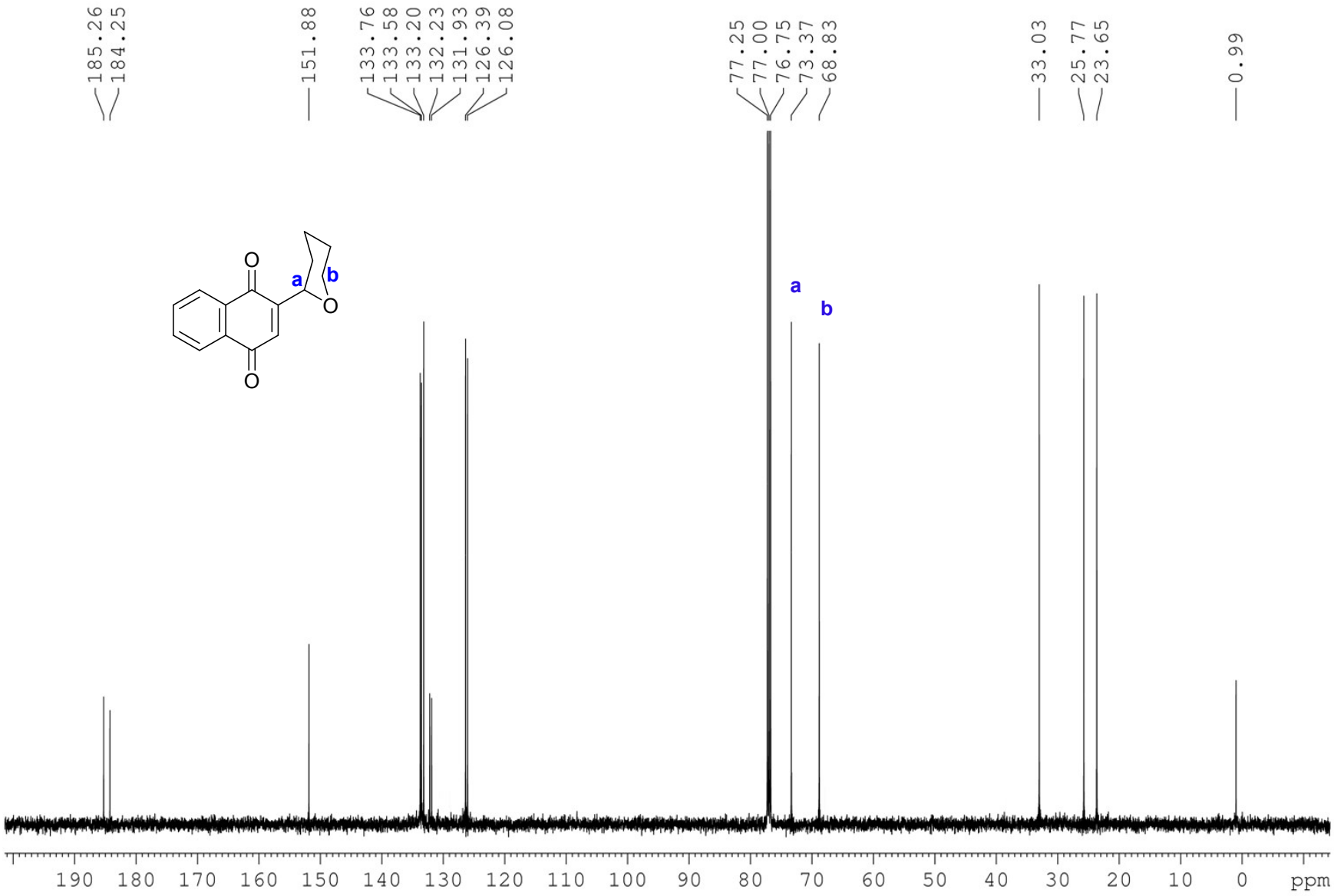


Figure 57. ^{13}C NMR (100 MHz, $CDCl_3$, 300K) of **2-(tetrahydro-2*H*-pyran-2-yl)naphthalene-1,4-dione**.

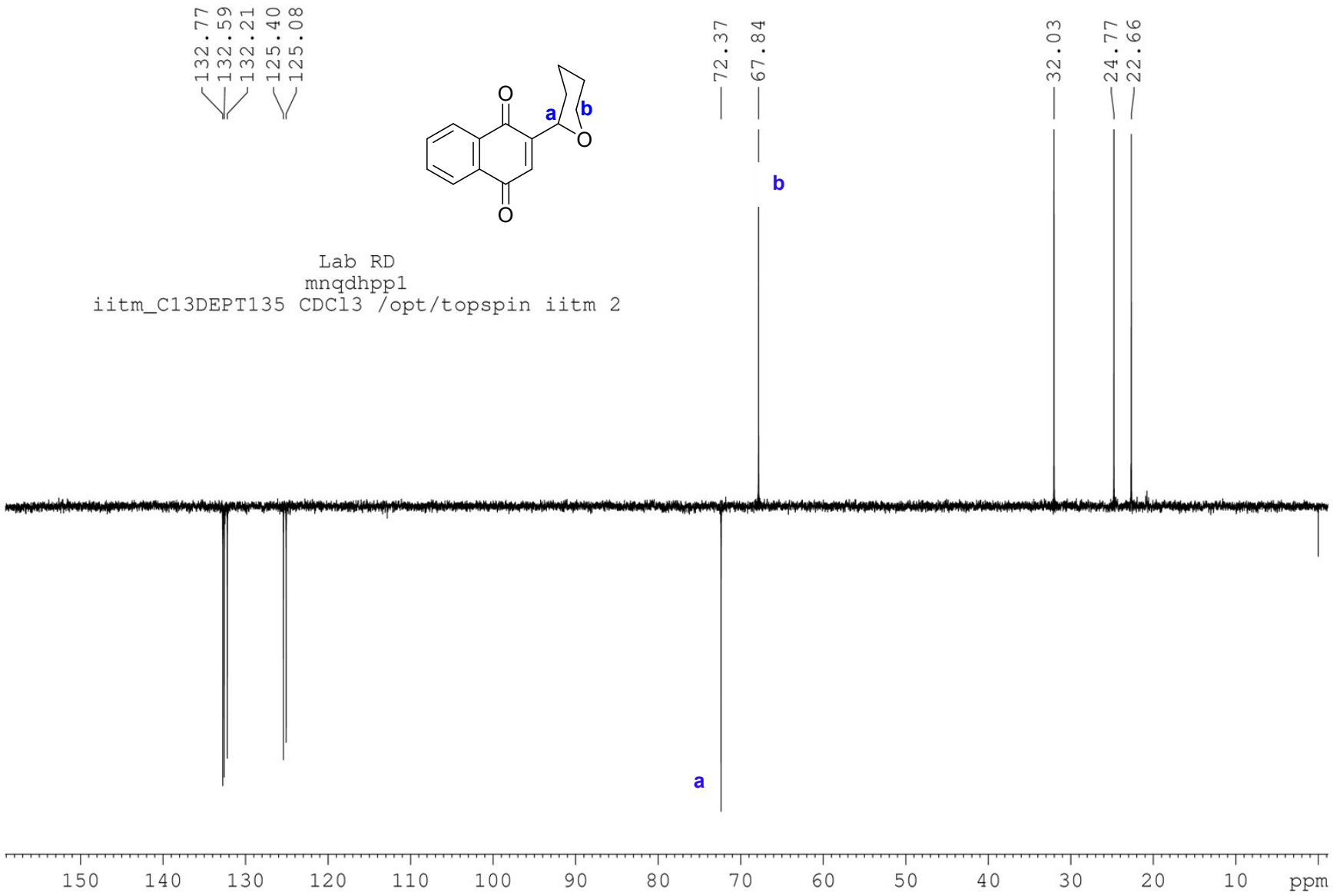


Figure 58. DEPT NMR (100 MHz, $CDCl_3$, 300K) of **2-(tetrahydro-2*H*-pyran-2-yl)naphthalene-1,4-dione**.

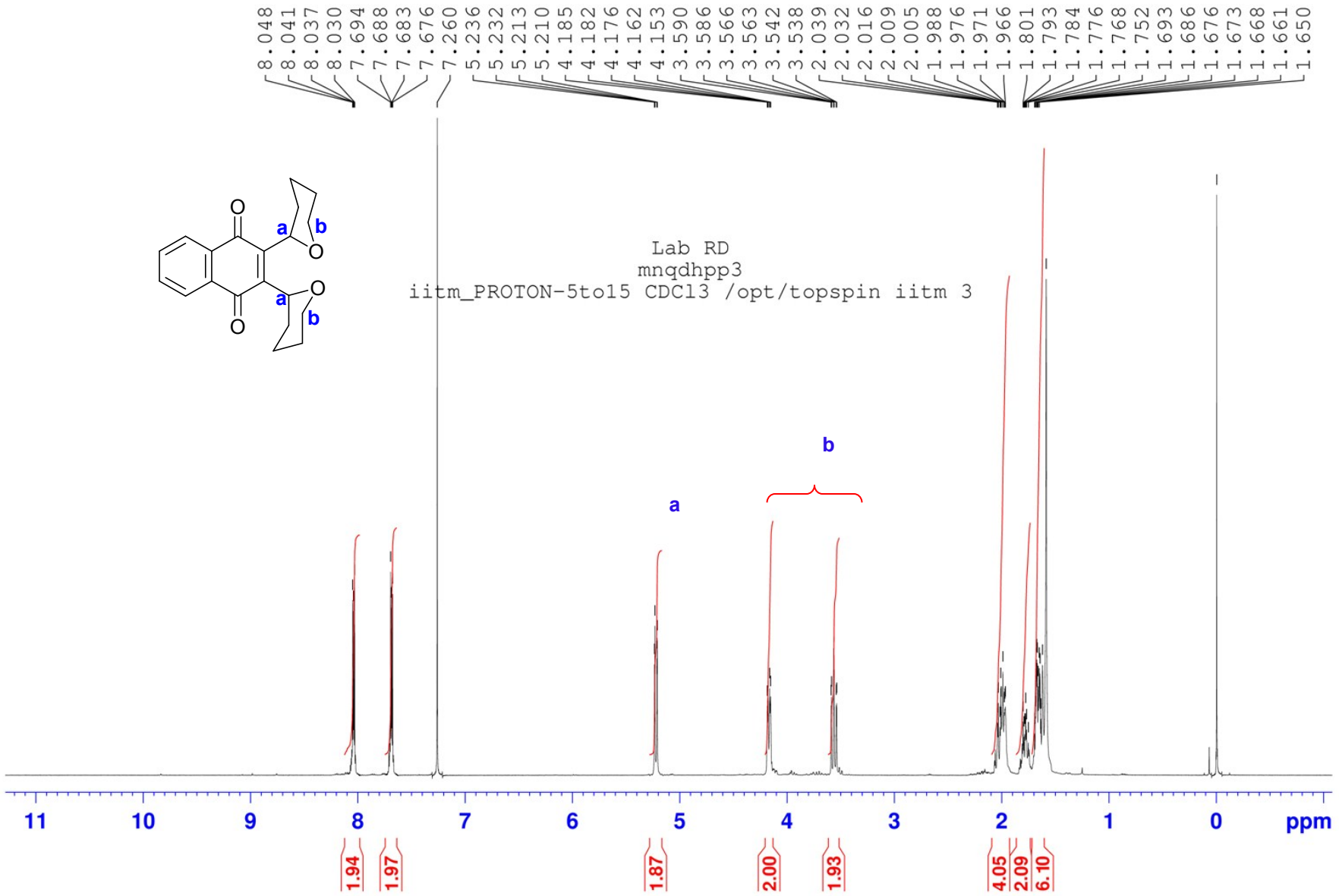


Figure 59. ^{1}H NMR (400 MHz, $CDCl_3$, 300K) of **2,3-bis(tetrahydro-2*H*-pyran-2-yl)naphthalene-1,4-dione** (TMS added as internal standard).

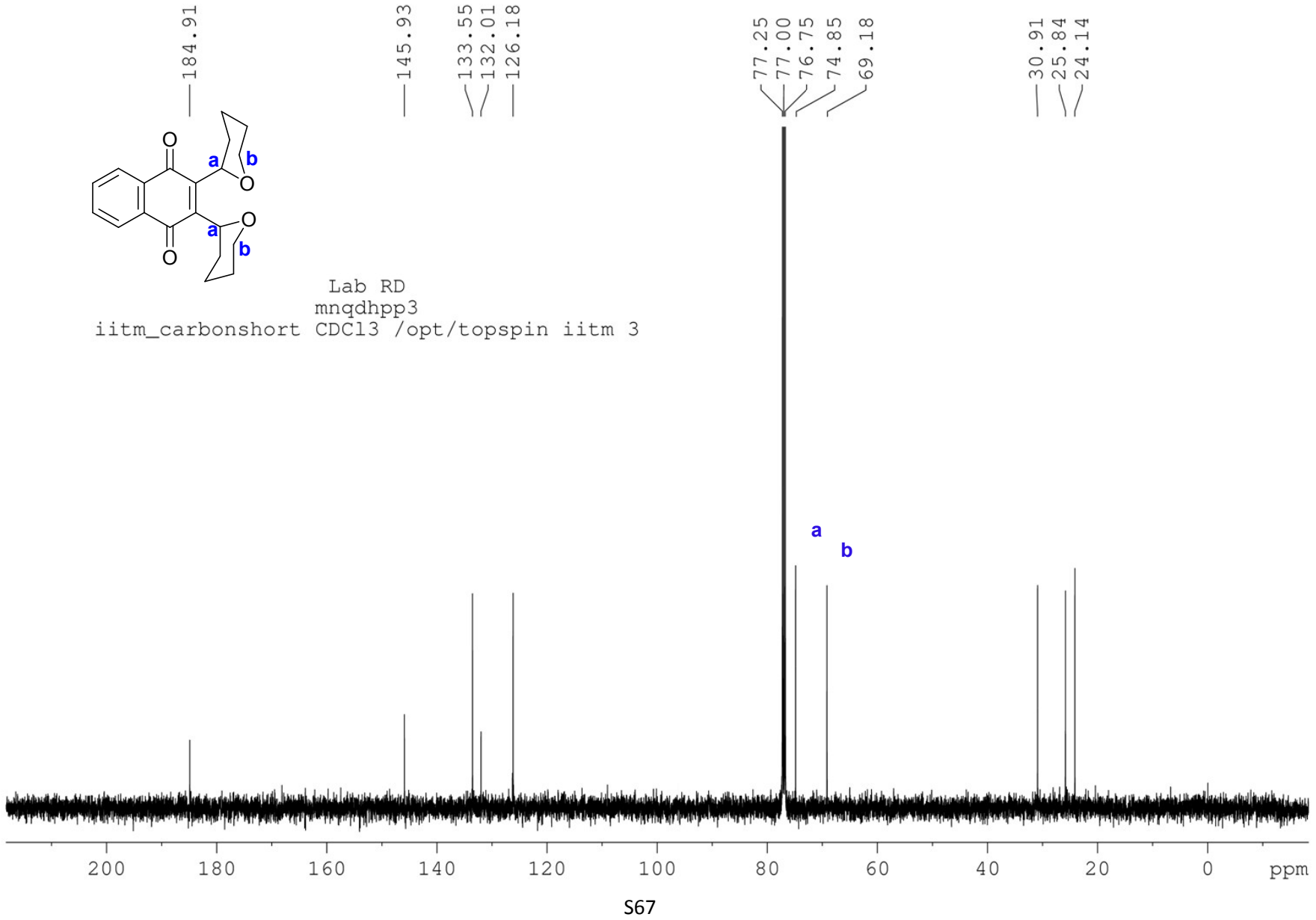


Figure 60. ^{13}C NMR (100 MHz, $CDCl_3$, 300K) of **2,3-bis(tetrahydro-2*H*-pyran-2-yl)naphthalene-1,4-dione**.

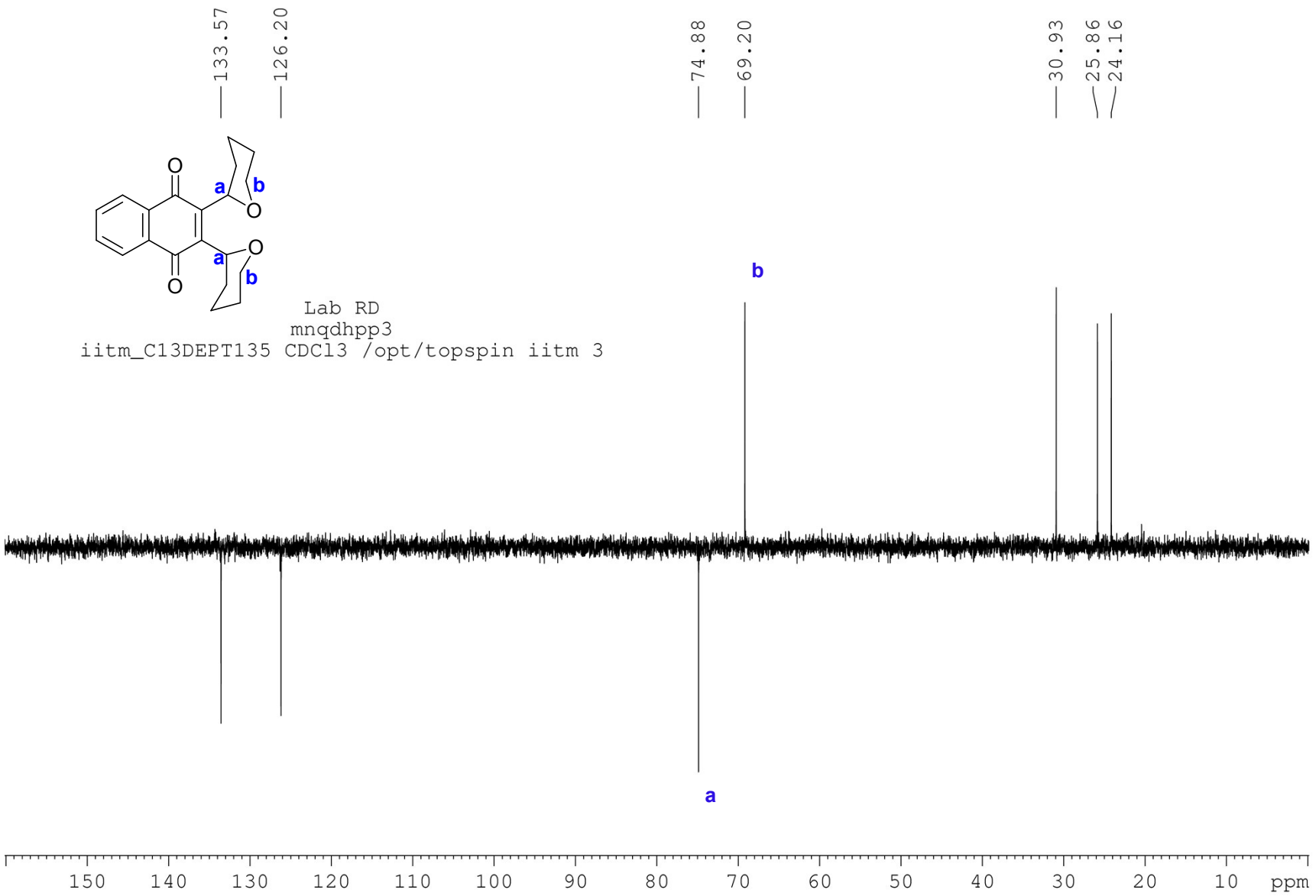


Figure 61. DEPT NMR (100 MHz, $CDCl_3$, 300K) of **2,3-bis(tetrahydro-2*H*-pyran-2-yl)naphthalene-1,4-dione**.

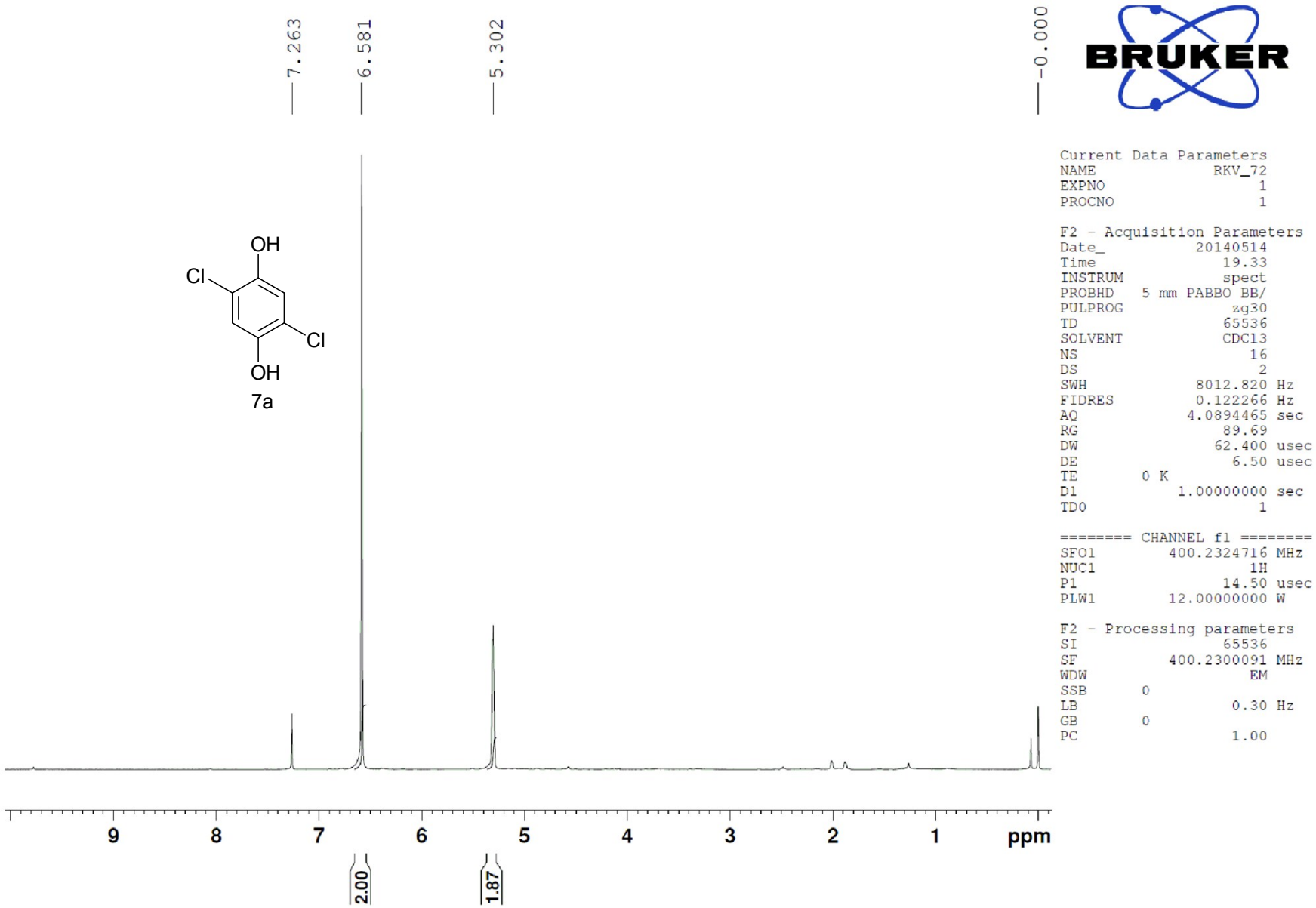


Figure 62. ^{1}H NMR (400 MHz, $CDCl_3$, 300K) of **7**a (TMS added as internal standard)

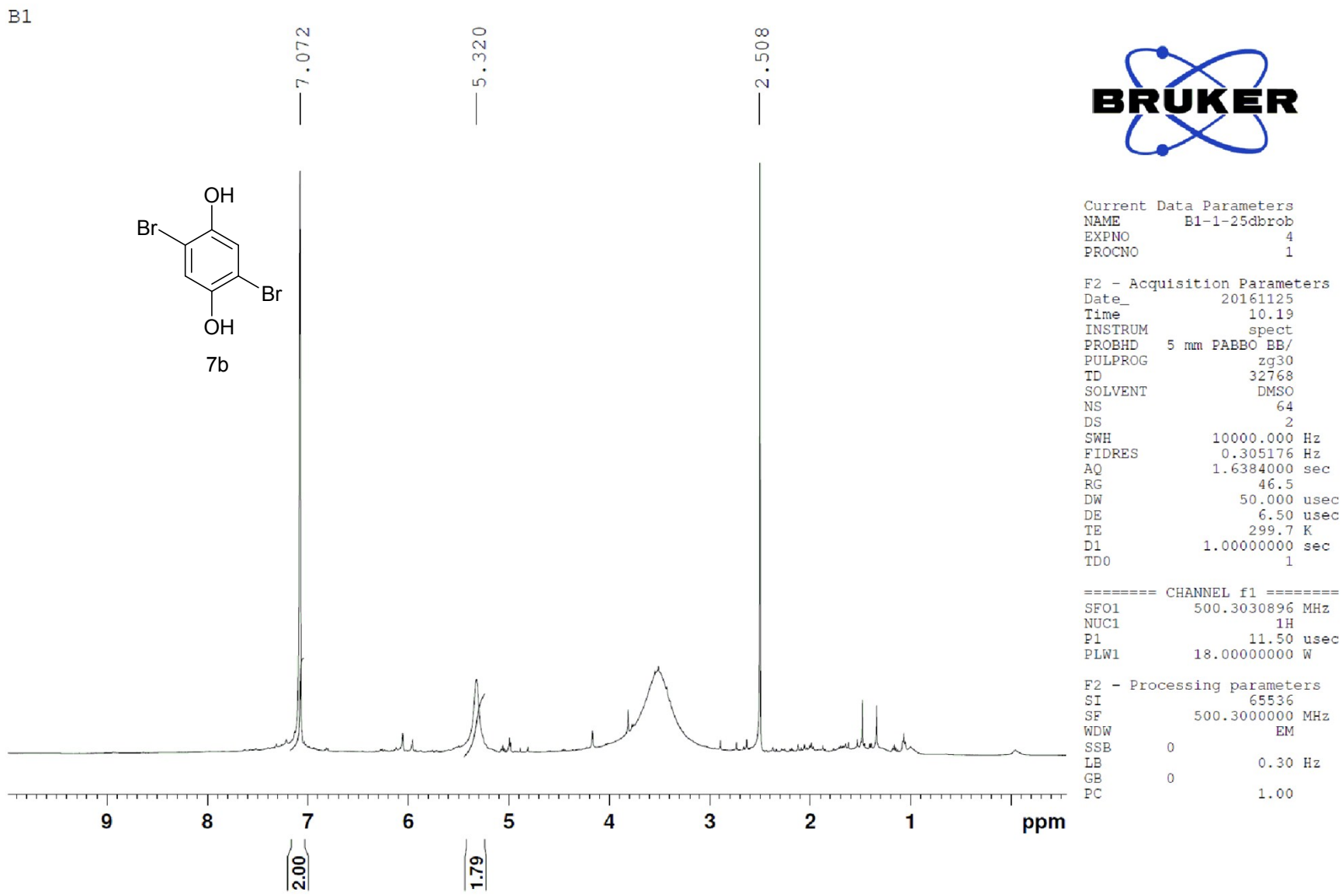


Figure 63. ^{1}H NMR (400 MHz, DMSO, 300K) of **7b**.

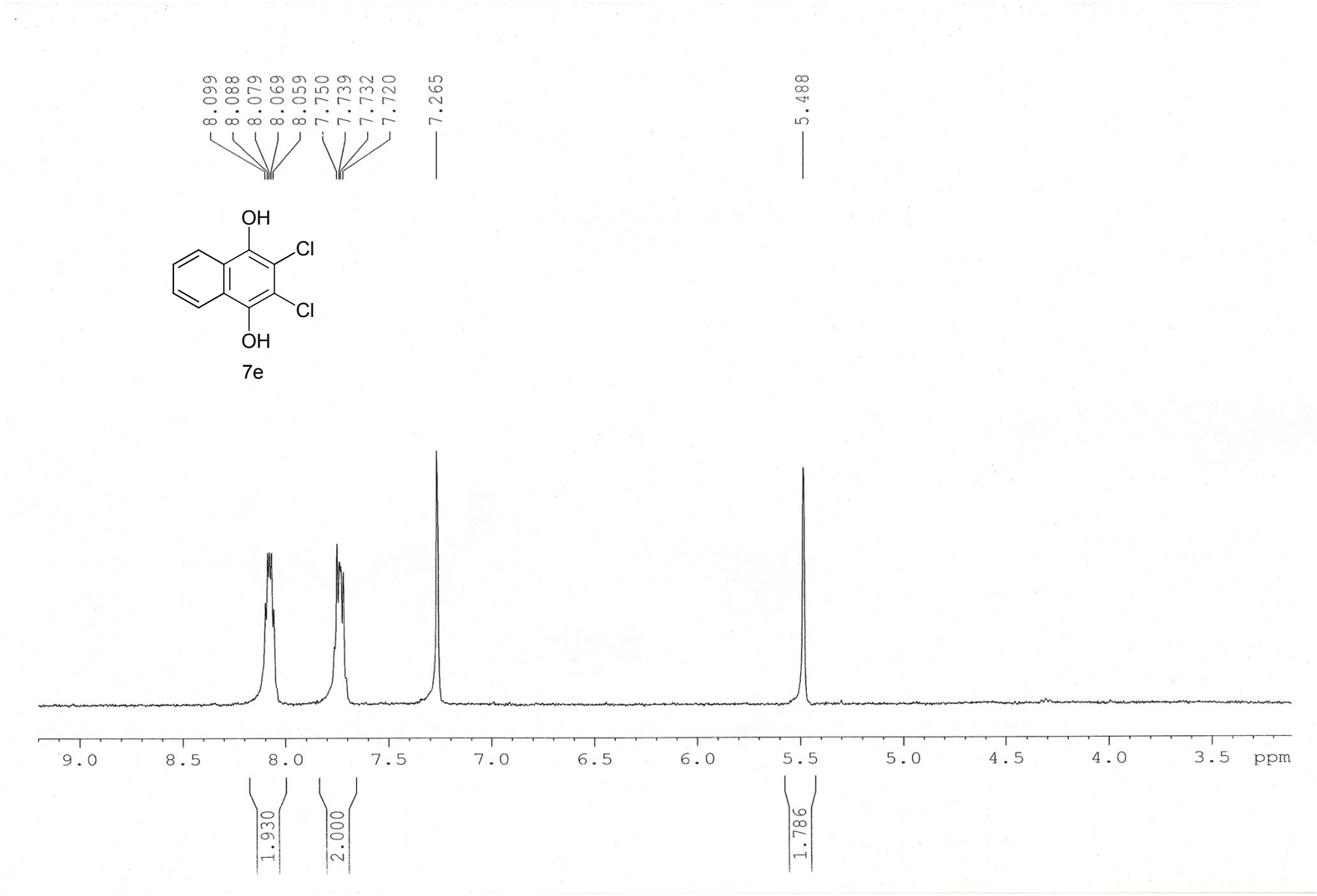


Figure 64. ^{1}H NMR (400 MHz, $CDCl_3$, 300K) of **7e**

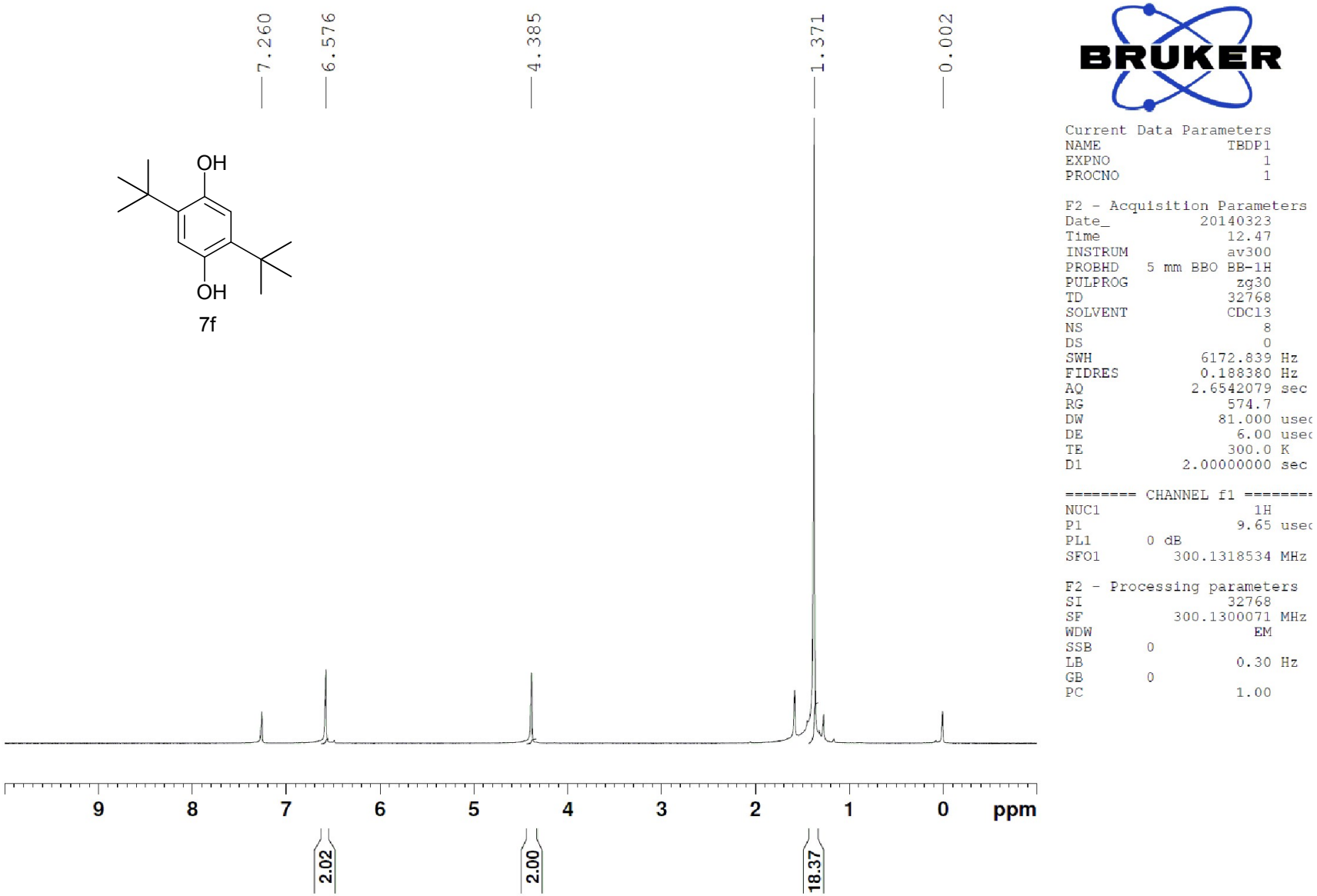


Figure 65. ^{1}H NMR (400 MHz, $CDCl_3$, 300K) of **7f** (TMS added as internal standard)

Crystal structure and Refinement data for 5a. The molecular structure is shown in Figure 1, while the refinement data are summarized in Table 1. The crystallographic data have been deposited at the Cambridge Crystallographic Data Centre (CCDC No. 1023672).

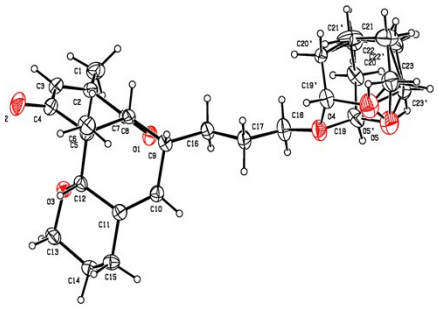


Figure 1. Single crystal X-ray structure of **5a**

Table 1. Refinement data for **5a**

Identification code	Shelxl
Empirical formula	$C_{23}H_{32}O_5$
Formula weight	388.49
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)
Unit cell dimensions	a = 11.734(5) A alpha = 90.000(5) deg.
	b = 7.397(5) A beta = 107.317(5) deg.
	c = 12.645(5) A gamma = 90.000(5) deg.
Volume	1047.8(9) A^3
Z, Calculated density	2, 1.231 Mg/m^3
Absorption coefficient	0.085 mm^-1
F(000)	420
Crystal size	0.35 x 0.30 x 0.30 mm
Theta range for data collection	1.69 to 27.99 deg.
Limiting indices	-15<=h<=14, -9<=k<=9, -16<=l<=16
Reflections collected / unique	9980 / 4622 [R(int) = 0.0230]
Completeness to theta = 27.99	100.0 %
Max. and min. transmission	0.9749 and 0.9708
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4622 / 104 / 309
Goodness-of-fit on F^2	1.050
Final R indices [I>2sigma(I)]	R1 = 0.0592, wR2 = 0.1734
R indices (all data)	R1 = 0.0810, wR2 = 0.1932
Absolute structure parameter	1.4(16)
Largest diff. peak and hole	0.277 and -0.204 e.A^-3

Crystal structure and Refinement data for 5b. The molecular structure is shown in Figure 2, while the refinement data are summarized in Table 2. The crystallographic data have been deposited at the Cambridge Crystallographic Data Centre (CCDC No. 1438171).

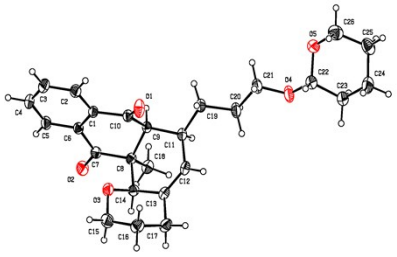


Figure 2. Single crystal X-ray structure of **5b**

Table 2. Refinement data for **5b**.

Identification code	shelx
Empirical formula	$C_{26}H_{32}O_5$
Formula weight	424.52
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Triclinic, P -1
Unit cell dimensions	a = 8.7034(4) A alpha = 92.963(3) deg. b = 9.1817(4) A beta = 104.082(3) deg. c = 16.3996(9) A gamma = 117.611(2) deg
Volume	1105.56(9) A^3
Z, Calculated density	2, 1.275 Mg/m^3
Absorption coefficient	0.087 mm^-1
F(000)	456
Crystal size	0.24 x 0.18 x 0.12 mm
Theta range for data collection	1.30 to 26.39 deg.
Limiting indices	-10<=h<=10, -10<=k<=11, -20<=l<=20
Reflections collected / unique	15987 / 4476 [R(int) = 0.0277]
Completeness to theta = 25.00	99.6 %
Max. and min. transmission	0.9896 and 0.9794
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4476 / 0 / 282
Goodness-of-fit on F^2	1.124
Final R indices [I>2sigma(I)]	R1 = 0.0720, wR2 = 0.1832
R indices (all data)	R1 = 0.0778, wR2 = 0.1857
Extinction coefficient	0.0095(18)
Largest diff. peak and hole	0.485 and -0.236 e.A^-3

Crystal structure and Refinement data for 6a. The molecular structure is shown in Figure 3, while the refinement data are summarized in Table 3. The crystallographic data have been deposited at the Cambridge Crystallographic Data Centre (CCDC No. 976243).

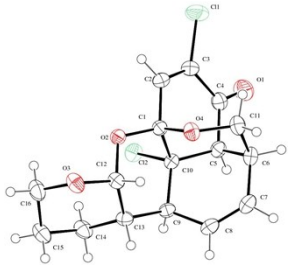


Figure 3. Single crystal X-ray structure of **6a**

Table 3. Refinement data for **6a**.

Identification code	shelxl	
Empirical formula	$C_{16}H_{16}Cl_2O_4$	
Formula weight	343.19	
Temperature	293(2) K	
Wavelength	0.71073 A	
Crystal system, space group	Monoclinic, P21/c	
Unit cell dimensions	a = 6.8501(5) A	alpha = 90 deg.
	b = 22.1100(15) A	beta = 99.315(2) deg.
	c = 9.8260(7) A	gamma = 90 deg.
Volume	1468.58(18) A^3	
Z, Calculated density	4, 1.552 Mg/m^3	
Absorption coefficient	0.458 mm^-1	
F(000)	712	
Crystal size	0.35 x 0.30 x 0.25 mm	
Theta range for data collection	2.29 to 27.50 deg.	
Limiting indices	-8<=h<=8, -27<=k<=28, -12<=l<=12	
Reflections collected / unique	15996 / 3369 [R(int) = 0.0277]	
Completeness to theta = 27.50	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8942 and 0.8263	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3369 / 0 / 199	
Goodness-of-fit on F^2	1.028	
Final R indices [I>2sigma(I)]	R1 = 0.0336, wR2 = 0.0854	
R indices (all data)	R1 = 0.0399, wR2 = 0.0899	
Largest diff. peak and hole	0.255 and -0.348 e.A^-3	

Crystal structure and Refinement data (2,5-dichloro-1,4-Bis(tetrahydro-2*H*-pyran-2-yloxy)benzene). The molecular structure is shown in Figure 4. The crystallographic data have been deposited at the Cambridge Crystallographic Data Centre (CCDC No. 976244).

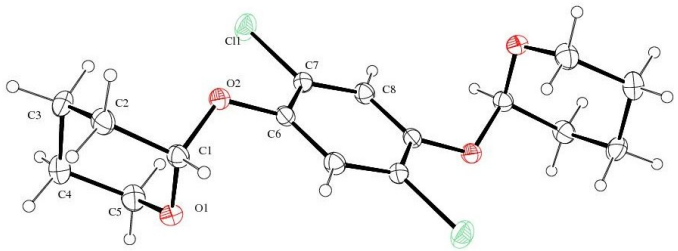


Figure 4. Single crystal X-ray structure

Table 3. Refinement data (**2,5-dichloro-1,4-Bis(tetrahydro-2*H*-pyran-2-yloxy)benzene**).

Identification code	shelxl
Empirical formula	$C_{16}H_{20}Cl_2O_4$
Formula weight	347.22
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P21/c
Unit cell dimensions	a = 5.945(5) A alpha = 90.000(5) deg. b = 18.659(5) A beta = 103.111(5) deg. c = 7.352(5) A gamma = 90.000(5) deg.
Volume	794.3(9) A^3
Z, Calculated density	2, 1.452 Mg/m^3
Absorption coefficient	0.424 mm^-1
F(000)	364
Crystal size	0.20 x 0.10 x 0.10 mm
Theta range for data collection	2.18 to 25.00 deg.
Limiting indices	-7<=h<=7, -22<=k<=22, -8<=l<=8
Reflections collected / unique	6895 / 1396 [R(int) = 0.0193]
Completeness to theta = 25.00	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9589 and 0.9001
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1396 / 0 / 104
Goodness-of-fit on F^2	1.058
Final R indices [I>2sigma(I)]	R1 = 0.0282, wR2 = 0.0675
R indices (all data)	R1 = 0.0313, wR2 = 0.0698
Largest diff. peak and hole	0.170 and -0.228 e.A^-3

Reference

[1] a) R. Stern, J. English and H. G. Cassidy, *J. Am. Chem. Soc*., **1957**, *79*, 5797; D. J. Brondani, C. R. Nascimento, M. Moreira , A. C. Lima Leite, I.A. Sou8a and L.W. Bieber, *Med. Chem*., **2007**, *3*, 369; D. Dutta, A. Pulsipher and M. N. Yousaf, *Langmuir*, **2010**, *26*, 9835.