Supplementary Data

Natural Product Based Inhibitors of the Thioredoxin – Thioredoxin Reductase System

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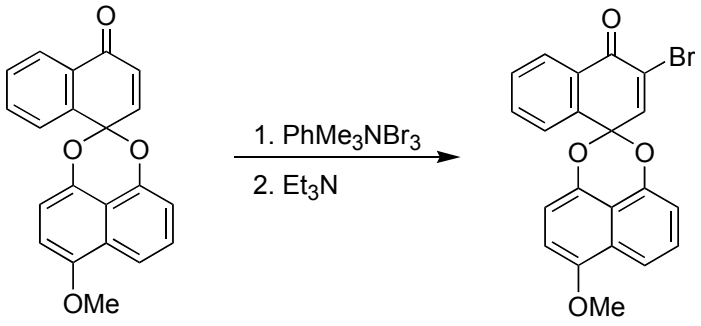
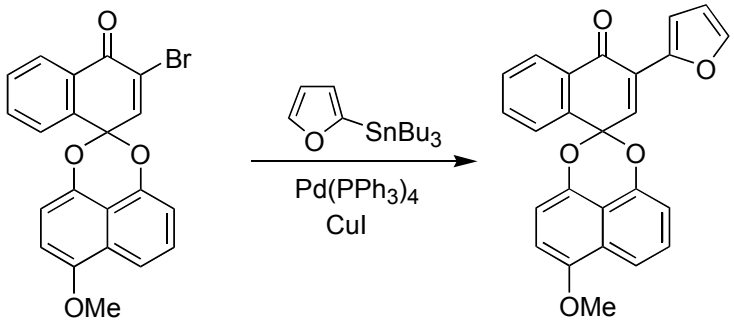
General: All reactions were performed in flame-dried or oven-dried glassware under a dry nitrogen atmosphere. THF and ether were distilled over Na/benzophenone, and CH_2Cl_2 was purified by passage through a column of activated alumina. All other reagents and solvents were used as received unless otherwise noted. NMR spectra were recorded in $CDCl_3$ (unless otherwise noted) at either 300 MHz (^{1}H NMR) or 75 MHz (^{13}C NMR) using a Bruker Avance 300 with XWIN-NMR software. Melting points were determined on a Mel-Temp II and are uncorrected.



SL-111 ***SL-114***

2-Bromo-1-oxo-1,4-dihydronaphthalene-4-spiro-2'-naphtho[4"-methoxy-1",8"-de][1',3']dioxin (S2). To a solution of **S1**[1] (100 mg, 0.303 mmol) in THF (3 mL) at 0 °C was added phenyltrimethylammonium tribromide (124 mg, 0.330 mmol). The reaction mixture was warmed to room temperature and stirred for 3 h. A white precipitate formed gradually, and the reaction mixture was quenched by addition of H_2O and extracted with EtOAc. The combined organic layers were washed with H_2O, dried ($MgSO_4$) and concentrated under reduced pressure. The foamy residue was dissolved in CH_2Cl_2 (3 mL), cooled to 0 °C, and Et_3N (63 μL, 0.45 mmol) was added. The reaction mixture was stirred at 0 °C for 1 h then quenched with H_2O and extracted with CH_2Cl_2. The combined organic layers were dried ($MgSO_4$) and concentrated under reduced pressure to afford 119 mg (97%) of **S2** as a yellow solid: mp 217 °C (CH_2Cl_2, dec); IR

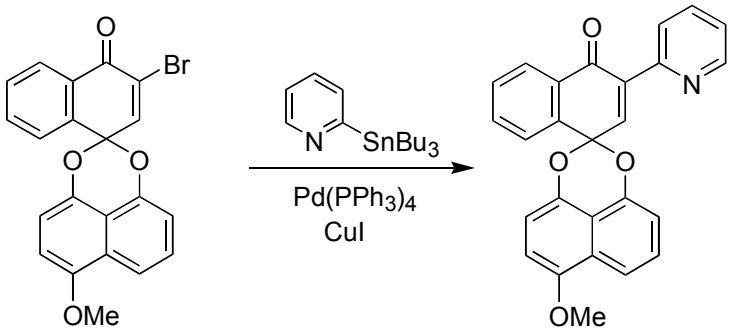
(neat) 1681, 1613, 1417, 1384, 1268, 1058, 815 cm^{-1}; ^1H NMR □ 8.23 (dd, 1H, *J* = 7.8, 0.9 Hz), 7.97-7.91 (m, 2H), 7.78 (td, 1H, *J* = 7.6, 1.3 Hz), 7.65 (td, 1H, *J* = 7.8, 1.2 Hz), 7.48 (t, 1H, *J* = 8.3 Hz), 7.44 (s, 1H), 7.03 (d, 1H, *J* = 7.6 Hz), 6.91 (d, 1H, *J* = 8.3 Hz), 6.80 (d, 1H, *J* = 8.3 Hz), 4.01 (s, 3H); ^{13}C NMR □ 177.1, 151.2, 147.1, 140.4, 139.5, 138.6, 134.5, 130.9, 128.8, 128.5, 128.2, 127.8, 127.2, 126.1, 116.8, 113.6, 111.2, 109.9, 105.2, 94.2, 56.1; MS (EI) *m/z* (rel intensity) 408 (M^+, 100), 395 (74), 305 (35), 276 (26), 129 (49), 101 (57); HRMS (EI) calcd for $C_{21}H_{13}O_4Br$ 407.9997, found 408.0011.



SL-114 ***SL-116***

2-(Furan-2-yl)-1-oxo-1,4-dihydronaphthalene-4-spiro-2'-naphtho[4"-methoxy-1",8"-de][1',3']dioxin (S3). To a solution of **S2** (35 mg, 0.085 mmol) in DMF (1 mL) was added 2-(tributylstannyl)furan (32 □L, 0.10 mmol), followed by $Pd(PPh_3)_4$ (5 mg, 0.003 mmol) and CuI (3 mg, 0.012 mmol). The reaction mixture was heated at 60 °C for 1.5 h, cooled to room temperature and diluted with EtOAc (10 mL). The precipitate was removed by filtration over Celite (EtOAc, 20 mL). The filtrate was washed with H_2O (3 x 10 mL), dried ($MgSO_4$), and concentrated under reduced pressure. Trituration of the oily residue with hexanes/EtOAc (19:1) afforded 30 mg (88%) of **S3** as a yellow-brown solid: mp 223 °C (hexanes/EtOAc, dec); IR (neat) 1677, 1632, 1600, 1417, 1383, 1259,

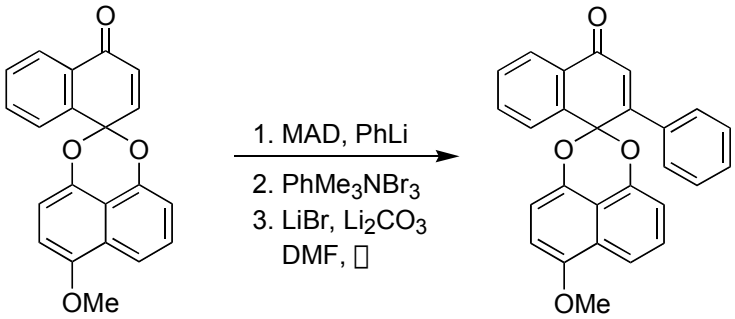
1060 cm^{-1}; ^1H NMR □ 8.24 (dd, 1H, *J* = 7.8, 1.1 Hz), 8.00 (dd, 1H, *J* = 7.8, 0.9 Hz), 7.93 (dd, 1H, *J* = 8.5, 0.6 Hz), 7.77 (td, 1H, *J* = 7.6, 1.3 Hz), 7.65 (td, 1H, *J* = 7.6, 1.2 Hz), 7.48 (dd, 1H, *J* = 8.4, 7.7 Hz), 7.34 (s, 1H), 7.33 (d, 1H, *J* = 3.5 Hz), 7.24 (d, 1H, *J* = 1.8 Hz), 7.03 (dd, 1H, *J* = 7.6, 0.5 Hz), 6.91 (d, 1H, *J* = 8.3 Hz), 6.80 (d, 1H, *J* = 8.3 Hz), 6.42 (dd, 1H, *J* = 3.5, 1.8 Hz), 4.02 (s, 3H); ^{13}C NMR □ 181.4, 151.0, 147.8, 147.3, 142.8, 141.1, 138.6, 134.0, 130.7, 130.6, 130.1, 129.2, 127.8, 127.1, 127.0, 126.2, 116.4, 114.1, 113.9, 112.3, 111.1, 109.7, 105.4, 93.5, 56.1; MS (EI) *m/z* (rel intensity) 396 (M^+, 85), 381 (100), 353 (12), 196 (5), 168 (6), 151 (6), 129 (12), 101 (16); HRMS (EI) calcd for $C_{25}H_{16}O_5$ 396.0998, found 396.1013.



SL-114 ***SL-128***

2-(Pyridin-2-yl)-1-oxo-1,4-dihydronaphthalene-4-spiro-2'-naphtho[4"-methoxy-1",8"-de][1',3']dioxin (S4). To a solution of **S2** (37 mg, 0.090 mmol) in DMF (1 mL) was added 2-(tributylstannyl)pyridine (50 mg, 0.14 mmol), followed by $Pd(PPh_3)_4$ (6 mg, 0.005 mmol) and CuI (7 mg, 0.04 mmol). The reaction mixture was heated at 80 °C for 2 h, cooled to room temperature and diluted with EtOAc (10 mL). The precipitate was removed by filtration over Celite (EtOAc, 20 mL). The filtrate was washed with H_2O (3 x 10 mL), dried ($MgSO_4$), and concentrated under reduced pressure. Chromatography of the oily residue on SiO_2 (hexanes/acetone, 7:3) gave 25 mg (70%) of **S4** as a bright

yellow solid: mp 220 °C (hexanes/acetone, dec); IR (neat) 1668, 1612, 1465, 1416, 1384, 1267, 1060 cm^{-1}; ^1H NMR □ 8.47 (dq, 1H, J = 4.8, 0.9 Hz), 8.27 (dd, 1H, *J* = 7.8, 1.4 Hz), 8.02 (dd, 1H, *J* = 7.8, 1.2 Hz), 7.90 (dd, 1H, *J* = 8.5, 0.8 Hz), 7.82-7.76 (m, 2H), 7.70-7.64 (m, 2H), 7.61 (s, 1H), 7.47 (dd, 1H, *J* = 8.5, 7.6 Hz), 7.19 (ddd, 1H, *J* = 7.5, 4.8, 1.2 Hz), 7.04 (dd, 1H, *J* = 7.6, 0.8 Hz), 6.92 (d, 1H, *J* = 8.3 Hz), 6.79 (d, 1H, *J* = 8.3 Hz), 4.00 (s, 3H); ^{13}C NMR □ 183.0, 152.3, 151.0, 149.6, 147.7, 141.0, 138.7, 138.6, 138.5, 136.1, 134.0, 131.0, 130.6, 127.9, 127.2, 127.1, 126.2, 125.2, 123.6, 116.4, 113.9, 111.2, 109.7, 105.4, 93.5, 56.1; MS (EI) *m/z* (rel intensity) 407 (M^+, 100), 392 (53), 364 (17), 336 (11), 219 (14), 189 (30), 129 (10), 101 (15); HRMS (EI) calcd for $C_{26}H_{17}NO_4$ 407.1158, found 407.1165.

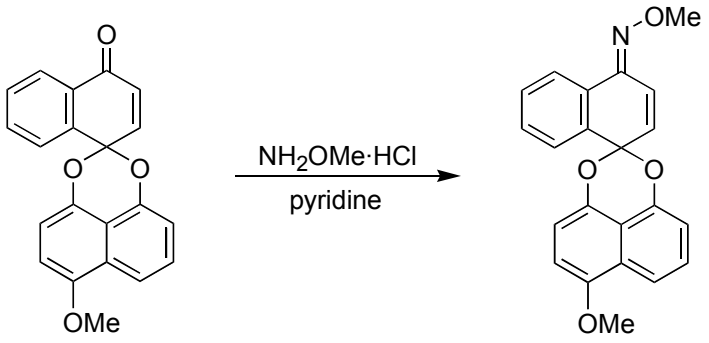


SL-111 ***SL-161***

1-Oxo-3-phenyl-1,4-dihydronaphthalene-4-spiro-2'-naphtho[4"-methoxy-1",8"-de][1',3']dioxin (S5). A solution of methylaluminum bis(2,6-di-*tert*-butyl-4-methylphenoxide) $(MAD)^2$ was prepared by dropwise addition of trimethylaluminum (2.0 M in toluene; 0.30 mL, 0.60 mmol) to a solution of 2,6-di-*tert*-butyl-4-methylphenol (264 mg, 1.20 mmol) in toluene (3 mL) at room temperature. When gas evolution had ceased (~15 min) the reaction mixture was cooled to -78 °C and a solution of **S1** (100 mg, 0.303 mmol) in CH_2Cl_2 (2 mL) was slowly added. To the resulting deep purple

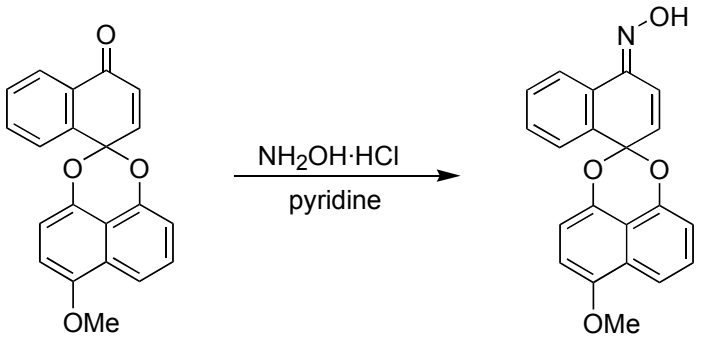
solution was added dropwise phenyllithium (1.8 M in cyclohexane/Et_2O (7:3); 0.18 mL, 0.33 mmol) which caused the solution to turn pale yellow. The reaction mixture was quenched with H_2O (0.5 mL), warmed to room temperature and stirred for 1 h. The dark colored precipitate was removed via filtration over Celite (CH_2Cl_2), and the filtrate was concentrated under reduced pressure. Chromatography of the residue on SiO_2 (hexanes/EtOAc, 4:1) gave 100 mg of a pale yellow foam which was dissolved in THF (2 mL) and treated with phenyltrimethylammonium tribromide (92 mg, 0.25 mmol). The reaction mixture was stirred at room temperature for 30 min as a thick white precipitate formed, quenched by addition of H_2O and extracted with EtOAc. The combined organic layers were washed with H_2O, dried ($MgSO_4$), and concentrated under reduced pressure. The orange residue was dissolved in DMF (2 mL) and LiBr (45 mg, 0.52 mmol) and Li_2CO_3 (58 mg, 0.78 mmol) were added. The suspension was heated at 130 °C for 2 h and cooled to room temperature. The solid precipitate was removed via filtration (EtOAc, 3 x 15 mL). The filtrate was washed with H_2O (3 x 10 mL), dried ($MgSO_4$), and concentrated under reduced pressure. Chromatography on SiO_2 (hexanes/EtOAc, 4:1) afforded 83 mg (68%) of **S5** as a yellow solid: mp 153-154 °C; IR (neat) 1667, 1609, 1510, 1419, 1384, 1269, 1143 cm^{-1}; ^1H NMR □ 8.16 (dd, 1H, *J* = 7.7, 1.1 Hz), 7.77 (dd, 1H, *J* = 8.5, 0.8 Hz), 7.50-7.43 (m, 3H), 7.40 (dd, 1H, *J* = 8.5, 7.7 Hz), 7.34 (td, 1H, *J* = 7.7, 1.5 Hz), 7.23-7.12 (m, 4H), 6.91 (dd, 1H, *J* = 7.6, 0.8 Hz), 6.78 (d, 1H, *J* = 8.3 Hz), 6.72 (d, 1H, *J* = 8.3 Hz), 6.63 (s, 1H), 3.96 (s, 3H); ^{13}C NMR □ 184.3, 155.8, 150.4, 146.9, 141.1, 140.0, 135.7, 133.4, 129.8, 129.7, 129.2, 128.6, 128.1, 127.1, 126.9, 125.6, 125.2, 115.7, 112.5, 110.2, 108.9, 105.4, 95.4, 56.0; MS (EI) *m/z*

(rel intensity) 406 (M^+, 100), 391 (58), 363 (10), 276 (12), 203 (18), 189 (25), 129 (24), 101 (25), 75 (13); HRMS (EI) calcd for $C_{27}H_{18}O_4$ 406.1205, found 406.1206.



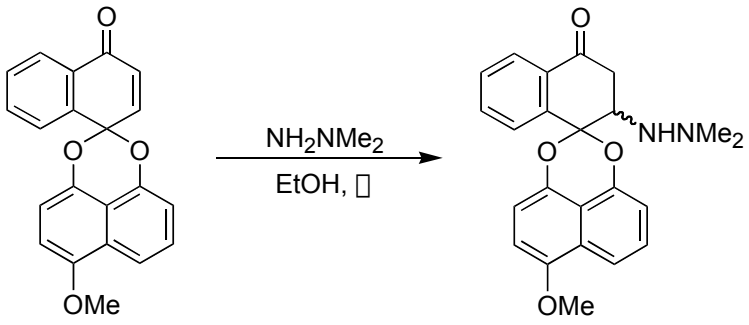
SL-111 ***SL-173***

1-Methoxyimino-1,4-dihydronaphthalene-4-spiro-2'-naphtho[4"-methoxy-1",8"-de][1',3']dioxin (S6). To a solution of **S1** (33 mg, 0.10 mmol) in pyridine (1 mL) was added methoxylamine hydrochloride (10 mg, 0.12 mmol). The reaction mixture was stirred at room temperature for 24 h, quenched by addition of H_2O and extracted with CH_2Cl_2. The organic layer was dried ($MgSO_4$) and concentrated under reduced pressure. Chromatography on SiO_2 (hexanes/EtOAc, 4:1) gave 28 mg (78%) of **S6** as a yellow solid: mp 157-158 °C; IR (neat) 1612, 1511, 1416, 1384, 1268, 1049 cm^{-1}; ^1H NMR □ 8.19 (dd, 1H, *J* = 7.6, 1.5 Hz), 7.94 (dd, 1H, *J* = 7.6, 1.4 Hz), 7.86 (dd, 1H, *J* = 8.5, 0.8 Hz), 7.56 (td, 1H, *J* = 7.4, 1.5 Hz), 7.49 (td, 1H, 7.6, 1.5 Hz), 7.45 (dd, 1H, *J* = 8.4, 7.6 Hz), 7.12 (d, 1H, *J* = 10.7 Hz), 6.98 (dd, 1H, *J* = 7.6, 0.8 Hz), 6.85 (d, 1H, *J* = 8.3 Hz), 6.77 (d, 1H, *J* = 8.3 Hz), 6.40 (d, 1H, *J* = 10.7 Hz), 4.08 (s, 3H), 3.99 (s, 3H); ^{13}C NMR □ 150.8, 148.2, 145.1, 141.5, 134.1, 130.0, 129.8, 129.3, 128.9, 127.9, 127.1, 126.2, 123.0, 118.2, 116.0, 114.2, 110.9, 109.4, 105.4, 95.0, 63.1, 56.1; MS (EI) *m/z* (rel intensity) 359 (M^+, 95), 344 (100), 330 (10), 233 (12), 183 (28), 130 (11), 101 (15), 75 (10); HRMS (EI) calcd for $C_{22}H_{17}NO_4$ 359.1158, found 359.1158.



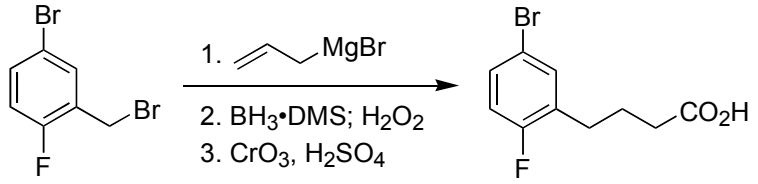
SL-111 ***SL-176***

1-Hydroxyimino-1,4-dihydronaphthalene-4-spiro-2'-naphtho[4"-methoxy-1",8"-de][1',3']dioxin (S7). To a solution of **S1** (33 mg, 0.10 mmol) in pyridine (1 mL) was added methoxylamine hydrochloride (9.0 mg, 0.12 mmol). The reaction mixture was stirred at room temperature for 24 h, quenched by addition of H_2O and extracted with CH_2Cl_2. The organic layer was dried ($MgSO_4$) and concentrated under reduced pressure. Chromatography on SiO_2 (hexanes/EtOAc, 4:1) gave 6 mg (18%) of **S7** as a yellow solid: mp 193 °C (hexanes/EtOAc, dec); ^{1}H NMR 8.11 (dd, 1H, *J* = 7.6, 1.4 Hz), 7.95 (dd, 1H, *J* = 7.6, 1.4 Hz), 7.94 (s, 1H), 7.87 (dd, 1H, *J* = 8.5, 0.8 Hz), 7.58 (td, 1H, *J* = 7.6, 1.5 Hz), 7.50 (td, 1H, *J* = 7.7, 1.4 Hz), 7.45 (dd, 1H, *J* = 8.4, 7.6 Hz), 7.25 (d, 1H, *J* = 10.7 Hz), 6.99 (dd, 1H, *J* = 7.6, 0.8 Hz), 6.86 (d, 1H, *J* = 8.3 Hz), 6.77 (d, 1H, *J* = 8.3 Hz), 6.46 (d, 1H, *J* = 10.7 Hz), 4.00 (s, 3H); MS (EI) *m/z* (rel intensity) 345 (M^+, 76), 331 (30), 189 (28), 143 (18), 111 (24), 97 (41), 83 (46), 69 (87), 57 (100); HRMS (EI) calcd for $C_{21}H_{15}NO_4$ 345.1001, found 345.1010.



SL-111 ***SL-171***

1-Oxo-3-(N',N'-dimethylhydrazino)-1,2,3,4-tetrahydronaphthalene-4-spiro-2'-naphtho[4''-methoxy-1'',8''-de][1',3']dioxin (S8). To a suspension of **S1** (33 mg, 0.10 mmol) in EtOH (2 mL) was added 1,1-dimethylhydrazine (40 μL, 0.50 mmol). After heating at reflux for 1 h all solids gradually dissolved. The reaction mixture was cooled to room temperature and concentrated under reduced pressure. Chromatography on SiO_2 (hexanes/EtOAc, 7:3) gave 31 mg (84%) of **S8** (1:1 mixture of diastereomers) as a beige solid: mp 182-192 °C; ^{1}H NMR δ 8.11 (dd, 1H, *J* = 7.7, 1.1 Hz), 7.93 (dt, 1H, *J* = 7.8, 1.2 Hz), 7.89 (dd, 0.5H, *J* = 8.5, 0.8 Hz), 7.85 (dd, 0.5H, *J* = 8.5, 0.8 Hz), 7.69 (td, 1H, *J* = 7.6, 1.4 Hz), 7.57 (td, 1H, *J* = 7.6, 1.2 Hz), 7.48 (dd, 0.5H, *J* = 8.4, 7.6 Hz), 7.43 (dd, 0.5H, *J* = 8.4, 7.6 Hz), 7.10 (d, 0.5H, *J* = 7.6 Hz), 6.96 (d, 1H, *J* = 8.1 Hz), 6.84 (d, 0.5H, *J* = 8.2 Hz), 6.78 (d, 0.5H, *J* = 8.3 Hz), 6.75 (d, 0.5H, *J* = 8.3 Hz), 4.00 (s, 1.5H), 3.99 (s, 1.5H), 3.78 (q, 1H, *J* = 3.2 Hz), 3.07 (dt, 1H, *J* = 16.6, 3.2 Hz), 2.95 (dt, 1H, *J* = 16.6, 3.0 Hz), 2.49 (brs, 1H), 2.07 (s, 3H), 2.04 (s, 3H); ^{13}C NMR δ 196.0, 151.1, 150.8, 147.8, 146.7, 141.1, 139.9, 138.2, 133.9, 132.8, 130.2, 127.3, 127.0, 126.9, 126.4, 126.2, 116.6, 116.1, 114.4, 110.7, 110.4, 109.3, 108.9, 105.4, 105.0, 99.2, 99.1, 56.0, 55.7, 55.6, 48.3, 48.2, 39.5, 39.4; MS (EI) *m/z* (rel intensity) 390 (M^+, 36), 332 (73), 330 (77), 315 (69), 276 (10), 201 (11), 129 (20), 101 (23), 75 (12), 59 (100); HRMS (EI) calcd for $C_{23}H_{22}N_2O_4$ 390.1580, found 390.1592.



1 **2**

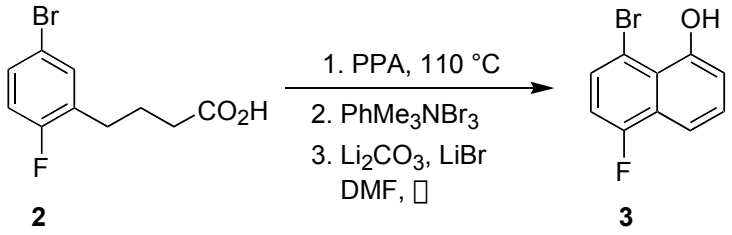
4-(5-Bromo-2-fluorophenyl)-butyric acid (2). To a solution of 4-bromo-2-bromomethyl-1-fluorobenzene[3] (**1**, 5.86 g, 21.8 mmol) in THF (25 mL) at 0 °C was slowly added allylmagnesium bromide (1.0 M in Et_2O; 33.0 mL, 33.0 mmol). During the addition a thick white precipitate formed. The reaction mixture was warmed to room temperature and stirred for 1 h, then cooled to 0 °C and quenched by slow dropwise addition of saturated aqueous NH_4Cl. The slurry was diluted with H_2O and extracted with Et_2O (3 x 100 mL). The combined organic layers were washed with H_2O, dried ($MgSO_4$) and concentrated under reduced pressure to afford 5.0 g of 4-bromo-2-but-3-enyl-1-fluorobenzene as a colorless oil which was used without further purification. An analytical sample was obtained by chromatography of the crude product on SiO_2 (hexanes/EtOAc, 19:1): IR (neat) 3078, 2930, 1641, 1485, 1233, 1173, 811 cm^{-1}; ^1H NMR 7.33-7.25 (m, 2H), 6.89 (t, 1H, *J* = 9.0 Hz), 5.90-5.76 (m, 1H), 5.08-4.98 (m, 2H), 2.70 (t, 2H, *J* = 7.7 Hz), 2.39-2.31 (m, 2H); ^{13}C NMR 160.4 (d, *J* = 243.6 Hz), 137.4 (s), 133.6 (d, *J* = 4.8 Hz), 131.2 (d, *J* = 17.5 Hz), 130.6 (d, *J* = 8.0 Hz), 117.2 (d, *J* = 23.9 Hz), 116.5 (d, *J* = 2.6 Hz), 115.8 (s), 34.1 (s), 28.6 (s); MS (EI) *m/z* (rel intensity) 228 (M^+, 25), 187 (100), 149 (24), 108 (44); HRMS (EI) calcd for $C_{10}H_{10}BrF$ 227.9950, found 227.9943.

To a solution of $BH_3•SMe_2$ (2.0 M in THF; 21.8 mL, 43.6 mmol) at 0 °C was slowly added a solution of this alkene (5.00 g, 21.8 mmol) in THF (20 mL). The reaction

mixture was warmed to room temperature and stirred for 2 h, then cooled to 0 °C and quenched ***slowly*** by dropwise addition of 3.0 M NaOH (24 mL, 72 mmol), which caused vigorous gas evolution. Then, 30% H_2O_2 (24.0 mL, 218 mmol) was added dropwise and the cloudy solution was warmed to room temperature and stirred overnight. The reaction mixture was diluted with H_2O and extracted with Et_2O (3 x 100 mL). The combined organic layers were washed with H_2O, 1.0 M HCl, and brine, dried ($MgSO_4$) and concentrated under reduced pressure to afford 5.5 g of 4-(5-bromo-2-fluorophenyl)-butan-1-ol as a colorless oil which was used without further purification. An analytical sample was obtained by chromatography of the crude product on SiO_2 (hexanes/EtOAc, 7:3): IR (neat) 3332 (br), 2937, 2865, 1462, 1232, 1177, 1060 cm^{-1}; ^1H NMR □ 7.32-7.23 (m, 2H), 6.88 (t, 1H, *J* = 9.0 Hz), 3.66 (t, 2H, *J* = 6.2 Hz), 2.63 (t, 2H, *J* = 7.1 Hz), 1.73-1.54 (m, 4H); ^{13}C NMR □ 160.4 (d, *J* = 243.6 Hz), 133.5 (d, *J* = 4.8 Hz), 131.7 (d, *J* = 17.5 Hz), 130.5 (d, *J* = 8.0 Hz), 117.2 (d, *J* = 23.9 Hz), 116.5 (d, *J* = 2.7 Hz), 62.8 (s), 32.4 (s), 28.7 (s), 26.3 (s); MS (EI) *m/z* (rel intensity) 246 (M^+, 41), 200 (100), 187 (53), 149 (65), 134 (52), 108 (53), 101 (23); HRMS (EI) calcd for $C_{10}H_{12}OBrF$ 246.0056, found 246.0067.

Jones reagent was prepared by slow addition of H_2SO_4 (4 mL) to a solution of CrO_3 (4.40 g, 44.0 mmol) in H_2O (16 mL) at 0 °C. This reagent was added dropwise at 0 °C via addition funnel to a solution of the above alcohol (5.38 g, 21.8 mmol) in acetone (100 mL). The reaction mixture was stirred at 0 °C for 1 h, warmed to room temperature and stirred for 1 h, then cooled back to 0 °C and quenched by dropwise addition of *i*-PrOH until precipitate formation had ceased. The precipitate was removed by filtration over Celite (acetone), and the filtrate was concentrated under reduced

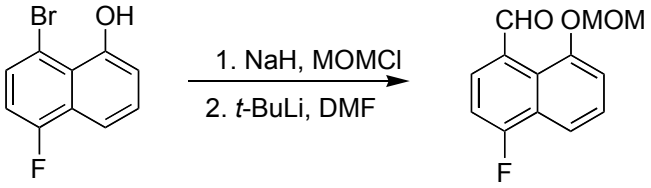
pressure. The residue was dissolved in Et_2O, washed with H_2O, and the organic layer was extracted with 10% NaOH. The basic aqueous layer was washed with Et_2O, then acidified with conc. HCl and extracted with Et_2O. The organic layer was dried ($MgSO_4$) and concentrated under reduced pressure. Recrystallization (hexanes) gave 3.15 g (55%) of **2** as a white solid: mp 91-92 °C (hexanes); IR (neat) 3052 (br), 2938, 1694, 1483, 1462, 1231, 1113, 816 cm^{-1}; ^1H NMR 11.43 (brs, 1H), 7.33-7.27 (m, 2H), 6.90 (t, 1H, *J* = 9.0 Hz), 2.68 (t, 2H, *J* = 7.6 Hz), 2.40 (t, 2H, *J* = 7.4 Hz), 1.95 (p, 2H); ^{13}C NMR 179.9 (s), 160.4 (d, *J* = 244.0 Hz), 133.6 (d, *J* = 4.6 Hz), 130.9 (d, *J* = 8.0 Hz), 130.6 (d, *J* = 17.5 Hz), 117.3 (d, *J* = 23.9 Hz), 116.7 (d, *J* = 2.4 Hz), 33.4 (s), 28.2 (s), 24.9 (s); MS (EI) *m/z* (rel intensity) 260 (M^+, 44), 200 (84), 187 (43), 134 (15), 122 (23), 108 (43), 101 (18); HRMS (EI) calcd for $C_{10}H_{10}O_2BrF$ 259.9848, found 259.9848.



8-Bromo-5-fluoronaphthalen-1-ol (3). To polyphosphoric acid prepared from 85% phosphoric acid (8 mL) and P_2O_5 (18 g) at 120 °C was added **2** (1.00 g, 3.83 mmol). The orange reaction mixture was heated at 120 °C for 1.5 h, then poured into a mixture of ice and water, shaken until PPA was fully decomposed, and extracted with Et_2O. The combined organic layers were washed with 5% NaOH solution and H_2O, dried ($MgSO_4$), and concentrated under reduced pressure to afford 0.81 g (87%) of 8-bromo-5-fluoro-3,4-dihydro-2*H*-naphthalen-1-one as an orange solid: mp 129-130 °C (Et_2O); IR (neat) 2955, 1688, 1593, 1449, 1281, 1247, 1185, 837 cm^{-1}; ^1H NMR 7.52 (dd, 1H, *J* = 8.7,

5.1 Hz), 7.03 (t, 1H, *J* = 8.7 Hz), 2.95 (t, 2H, *J* = 6.2 Hz), 2.70 (t, 2H, *J* = 6.2 Hz), 2.12 (p, 2H); ^{13}C NMR 195.9 (s), 159.4 (d, *J* = 245.2 Hz), 134.4 (d, *J* = 7.3 Hz), 134.1 (d, *J* = 18.0 Hz), 132.5 (s), 120.0 (d, *J* = 23.3 Hz), 116.3 (d, *J* = 3.4 Hz), 40.1 (s), 23.1 (d, *J* = 4.4 Hz), 22.1 (s); MS (EI) *m/z* (rel intensity) 242 (M^+, 83), 227 (13), 214 (100), 186 (50), 133 (40), 107 (94); HRMS (EI) calcd for $C_{10}H_8OBrF$ 241.9743, found 241.9752.

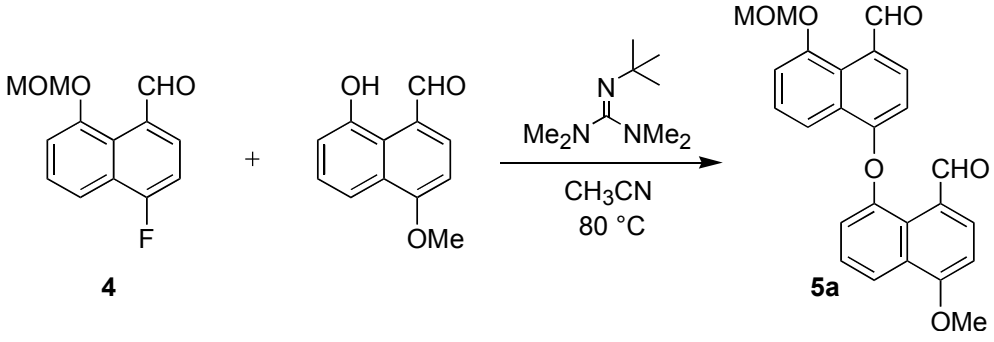
To a solution of this tetralone (1.08 g, 4.44 mmol) in THF (20 mL) was slowly added phenyltrimethylammonium tribromide (1.67 g, 4.44 mmol). The reaction mixture was stirred at room temperature for 30 min as a thick white precipitate formed, then quenched by addition of H_2O and extracted with EtOAc. The combined organic layers were washed with H_2O, dried ($MgSO_4$), and concentrated under reduced pressure. The orange residue was dissolved in DMF (20 mL) and LiBr (0.81 g, 9.32 mmol) and Li_2CO_3 (1.05 g, 14.20 mmol) were added. The suspension was heated at 130 °C for 1 h, cooled to room temperature, and the solids were removed by filtration (EtOAc (3 x 40 mL). The filtrate was washed with H_2O (3 x 40 mL), dried ($MgSO_4$), and concentrated under reduced pressure. Chromatography on SiO_2 (hexanes/EtOAc, 4:1) afforded 0.95 g (89%) of **3** as a beige solid: mp 84-86 °C (hexanes/EtOAc); IR (neat) 3473 (br), 1606, 1508, 1379, 1238, 922 cm^{-1}; ^{1}H NMR 8.07 (s, 1H), 7.69 (dt, 1H, *J* = 8.3, 0.9 Hz), 7.50 (dd, 1H, *J* = 8.3, 5.8 Hz), 7.44 (t, 1H, *J* = 8.1 Hz), 7.13 (d, 1H, *J* = 7.8 Hz), 6.93 (dd, 1H, *J* = 9.6, 8.3 Hz); ^{13}C NMR 159.0 (d, *J* = 251.3 Hz), 152.8 (d, *J* = 3.3 Hz), 130.7 (d, *J* = 8.8 Hz), 128.3 (s), 127.3 (d, *J* = 17.2 Hz), 121.7 (d, *J* = 4.2 Hz), 114.7 (s), 113.6 (d, *J* = 7.9 Hz), 110.1 (d, *J* = 22.0 Hz), 109.5 (d, *J* = 4.1 Hz); MS (EI) *m/z* (rel intensity) 240 (M^+, 100), 211 (9), 160 (15), 133 (87), 106 (21); HRMS (EI) calcd for $C_{10}H_6OBrF$ 239.9586, found 239.9575.



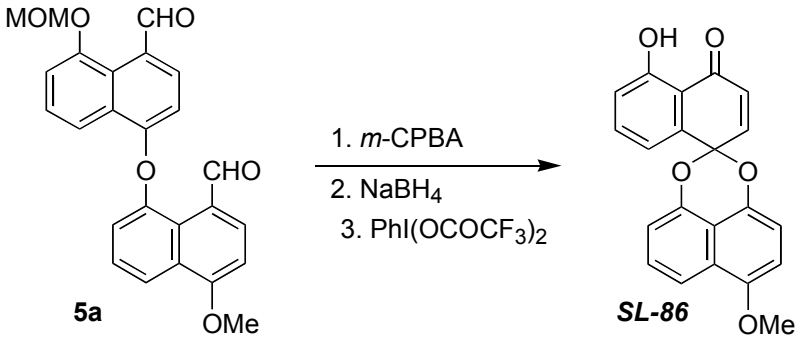
3 **4**

4-Fluoro-8-methoxymethoxynaphthalene-1-carbaldehyde (4). To a solution of **3** (950 mg, 3.94 mmol) in DMF (20 mL) at 0 °C was slowly added NaH (60% dispersion in mineral oil; 170 mg, 4.33 mmol). The reaction mixture was warmed to room temperature and stirred for 30 min, then cooled to 0 °C, and chloromethyl methyl ether (80%; 0.45 mL, 4.7 mmol) was added dropwise. The reaction mixture was again warmed to room temperature and stirred for 30 min, quenched at 0 °C by slow addition of H_2O and extracted with EtOAc (3 x 40 mL). The combined organic layers were washed with H_2O (3 x 40 mL), dried ($MgSO_4$), and concentrated under reduced pressure. Chromatography of the residue on SiO_2 (hexanes/EtOAc, 9:1) afforded 1.00 g (90%) of 4-bromo-1-fluoro-5-methoxymethoxynaphthalene as a yellow solid: mp 40-41 °C; IR (neat) 2955, 1604, 1568, 1507, 1416, 1258, 1153, 1002 cm^{-1}; ^1H NMR δ 7.78 (dt, 1H, *J* = 8.4, 1.0 Hz), 7.69 (dd, 1H, *J* = 8.3, 5.4 Hz), 7.46 (t, 1H, *J* = 8.1 Hz), 7.26 (dd, 1H, *J* = 7.7, 0.7 Hz), 6.96 (dd, 1H, *J* = 9.6, 8.3 Hz), 5.34 (s, 2H), 3.60 (s, 3H); ^{13}C NMR δ 158.4 (d, *J* = 250.1 Hz), 153.3 (d, *J* = 3.7 Hz), 132.4 (d, *J* = 8.4 Hz), 127.4 (s), 127.3 (s), 125.3 (d, *J* = 4.3 Hz), 115.0 (d, *J* = 7.9 Hz), 112.7 (s), 111.1 (d, *J* = 4.3 Hz), 110.6 (d, *J* = 21.3 Hz), 95.4 (s), 56.8 (s); MS (EI) *m/z* (rel intensity) 284 (M^+, 15), 254 (17), 211 (27), 174 (14), 144 (56), 132 (100), 106 (35); HRMS (EI) calcd for $C_{12}H_{10}O_2BrF$ 283.9848, found 283.9851.

To a solution of this bromide (1.00 g, 3.50 mmol) in Et_2O (20 mL) at -78 °C was added *t*-BuLi (1.7 M in pentane; 4.50 mL, 7.70 mmol) dropwise. The reaction mixture was stirred at -78 °C for 20 min, then DMF (0.800 mL, 10.5 mmol) was added dropwise. The chalky solution was stirred at -78 °C for 15 min, warmed to room temperature and stirred for 1 h, quenched with H_2O and extracted with EtOAc. The combined organic layers were washed with H_2O, dried ($MgSO_4$), and concentrated under reduced pressure. Chromatography on SiO_2 (hexanes/EtOAc, 4:1) provided 0.74 g (90%) of **4** as a pale yellow solid: mp 84-85 °C (hexanes/EtOAc); IR (neat) 2899, 2832, 1674, 1598, 1511, 1403, 1328, 1141, 977 cm^{-1}; ^1H NMR □ 11.06 (d, 1H, *J* = 0.6 Hz), 7.97 (dd, 1H, *J* = 8.1, 5.8 Hz), 7.83 (d, 1H, *J* = 8.4 Hz), 7.51 (t, 1H, *J* = 8.1 Hz), 7.36 (dd, 1H, *J* = 7.8, 0.7 Hz), 7.21 (dd, 1H, *J* = 9.3, 8.5 Hz), 5.40 (s, 2H), 3.53 (s, 3H); ^{13}C NMR □ 194.1 (s), 161.9 (d, *J* = 257.9 Hz), 154.0 (d, *J* = 3.3 Hz), 131.4 (d, *J* = 3.5 Hz), 128.8 (d, *J* = 9.9 Hz), 127.4 (s), 125.7 (d, *J* = 16.5 Hz), 125.5 (s), 115.1 (d, *J* = 7.1 Hz), 111.8 (s), 110.0 (d, *J* = 20.9 Hz), 95.2 (s), 56.8 (s); MS (EI) *m/z* (rel intensity) 234 (M^+, 61), 203 (14), 189 (100), 173 (20), 145 (17), 133 (83), 125 (15); HRMS (EI) calcd for $C_{13}H_{11}O_3F$ 234.0692, found 234.0696.



1-(5-Methoxy-8-formylnaphthalen-1-yloxy)-5-methoxymethoxynaphthalene-4-carbaldehyde (5a). To a solution of 8-hydroxy-4-methoxynaphthalene-1-carbaldehyde[4] (152 mg, 0.752 mmol) and fluoride **4** (117 mg, 0.500 mmol) in CH_3CN (3 mL) at room temperature was added 2-*tert*-butyl-1,1,3,3-tetramethylguanidine (0.15 mL, 0.75 mmol). The reaction mixture was heated at 80 °C for 5 h then cooled to room temperature, poured into 1.0 M HCl (10 mL), and extracted with CH_2Cl_2 (3 x 15 mL). The combined organic layers were washed with H_2O, dried ($MgSO_4$), and concentrated under reduced pressure. Chromatography on SiO_2 (hexanes/EtOAc, 3:2) followed by trituration with cold hexanes/EtOAc (4:1) afforded 148 mg (71%) of **5a** as a beige solid: mp 149-150 °C (hexanes/EtOAc); IR (neat) 2934, 1673, 1567, 1506, 1413, 1321, 1220, 1158, 1034 cm^{-1}; ^1H NMR □ 11.08 (s, 1H), 10.92 (s, 1H), 8.28 (dd, 1H, *J* = 8.4, 1.0 Hz), 8.20 (d, 1H, *J* = 8.3 Hz), 8.11 (dd, 1H, *J* = 8.3, 0.8 Hz), 7.87 (d, 1H, *J* = 8.1 Hz), 7.55-7.45 (m, 2H), 7.40 (d, 1H, *J* = 7.1 Hz), 7.21 (dd, 1H, *J* = 7.6, 1.0 Hz), 6.97 (d, 1H, *J* = 8.3 Hz), 6.78 (d, 1H, *J* = 8.1 Hz), 5.43 (s, 2H), 4.11 (s, 3H), 3.55 (s, 3H); ^{13}C NMR □ 194.4, 192.6, 160.3, 157.3, 154.2, 152.5, 131.2, 130.5, 128.8, 128.1, 127.9, 127.4, 126.6, 126.5, 126.1, 125.6, 120.2, 119.5, 116.3, 112.0, 111.6, 104.5, 95.2, 56.8, 56.3; MS (EI) *m/z* (rel intensity) 416 (M^+, 67), 371 (100), 343 (30), 201 (47), 185 (95), 171 (74), 155 (28), 114 (41); HRMS (EI) calcd for $C_{25}H_{20}O_6$ 416.1260, found 416.1256.

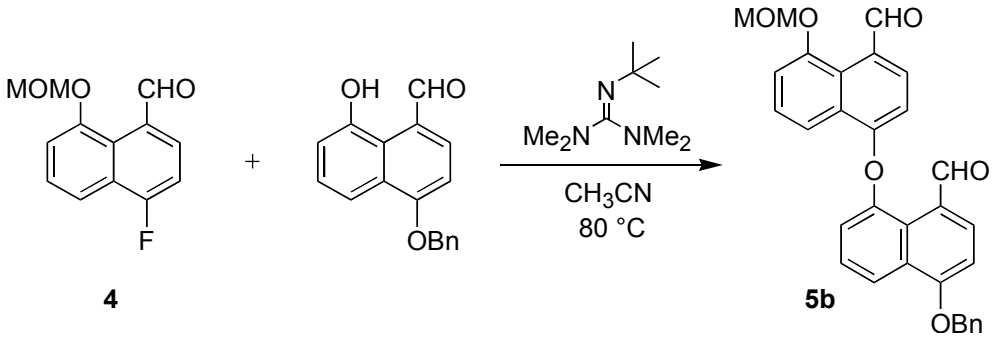


4'-Methoxypalmarumycin CP_1 (S9). To a solution of dialdehyde **5a** (611 mg, 1.46 mmol) in CH_2Cl_2 (30 mL) was added 70% *m*-CPBA (1.08 g, 4.38 mmol). The reaction mixture was stirred at room temperature for 15 h. A solution of 10% $Na_2S_2O_3$ (30 mL) was added and stirring was continued for 1 h. The organic layer was diluted with CH_2Cl_2, washed with saturated aqueous $NaHCO_3$, dried ($MgSO_4$), and concentrated under reduced pressure. Chromatography of the residue on SiO_2 (hexanes/EtOAc, 7:3) afforded 392 mg (60%) of 1-(5-methoxy-8-formyloxynaphthalen-1-yloxy)-5-methoxymethoxy-4-formyloxynaphthalene as a pale yellow foam: IR (neat) 2939, 1739, 1601, 1506, 1412, 1371, 1256, 1118 cm^{-1}; ^1H NMR □ 8.38 (s, 1H), 8.16 (dd, 1H, *J* = 8.5, 1.0 Hz), 8.08 (s, 1H), 7.99 (dd, 1H, *J* = 8.4, 0.8 Hz), 7.46 (t, 1H, *J* = 8.1 Hz), 7.41 (dd, 1H, *J* = 8.4, 7.8 Hz), 7.25 (dd, 1H, *J* = 7.8, 0.6 Hz), 7.08 (d, 1H, *J* = 8.3 Hz), 7.01-6.98 (m, 2H), 6.83 (d, 1H, *J* = 8.4 Hz), 6.67 (d, 1H, *J* = 8.3 Hz), 5.32 (s, 2H), 4.03 (s, 3H), 3.55 (s, 3H); ^{13}C NMR □ 160.6, 160.5, 154.2, 152.8, 152.4, 151.6, 141.1, 137.9, 129.3, 129.0, 127.4, 126.3, 121.1, 120.4, 120.0, 119.5, 119.1, 117.7, 116.3, 112.2, 111.7, 104.1, 95.4, 56.7, 56.1; MS (EI) *m/z* (rel intensity) 448 (M^+, 8), 420 (14), 392 (17), 347 (27), 189 (12), 145 (7), 115 (10), 84 (100); HRMS (EI) calcd for $C_{25}H_{20}O_8$ 448.1158, found 448.1145.

To a solution of this bisformate ester (105 mg, 0.234 mmol) in 1:1 MeOH/THF (5 mL) at 0 °C was slowly added $NaBH_4$ (18 mg, 0.46 mmol). The reaction mixture was stirred at 0 °C for 1 h, diluted with EtOAc (15 mL) and washed with H_2O. The organic layer was dried ($MgSO_4$) and concentrated under reduced pressure. The residue was *immediately* subjected to chromatography on SiO_2 (hexanes/EtOAc, 3:2) to afford 87 mg (97%) of 1-(5-methoxy-8-hydroxynaphthalen-1-yloxy)-5-methoxymethoxynaphth-4-ol as a white foam: IR (neat) 3438 (br), 1633, 1608, 1461, 1402, 1264, 1229, 1161, 1040 cm^{-1}; ^1H NMR 9.38 (s, 1H), 8.93 (s, 1H), 7.91 (dd, 1H, *J* = 8.5, 1.0 Hz), 7.64 (dd, 1H, *J* = 8.5, 0.8 Hz), 7.29 (dd, 1H, *J* = 8.3, 8.1 Hz), 7.26 (d, 1H, *J* = 8.4 Hz), 7.14 (t, 1H, *J* = 8.1 Hz), 7.12 (dd, 1H, *J* = 7.7, 0.7 Hz), 6.94 (d, 1H, *J* = 8.4 Hz), 6.92 (d, 1H, *J* = 8.4 Hz), 6.84 (d, 1H, *J* = 8.4 Hz), 6.49 (dd, 1H, *J* = 7.7, 0.9 Hz), 5.48 (s, 2H), 3.98 (s, 3H), 3.62 (s, 3H); ^{13}C NMR 156.4, 154.1, 152.6, 148.5, 147.7, 141.8, 130.0, 128.3, 127.2, 125.3, 120.5, 117.1, 116.7, 116.3, 115.8, 110.0, 109.7, 109.5, 108.9, 106.3, 96.0, 57.1, 56.1; MS (EI) *m/z* (rel intensity) 392 (M^+, 100), 347 (58), 319 (12), 287 (7), 189 (22), 130 (19); HRMS (EI) calcd for $C_{23}H_{20}O_6$ 392.1260, found 392.1262.

To a solution of this bisnaphthol (60 mg, 0.15 mmol) in dry CH_3CN (3 mL) at 0 °C was added $PhI(OCOCF_3)_2$ (72 mg, 0.17 mmol) in one portion. The reaction mixture was stirred at 0 °C for 30 min and concentrated under reduced pressure. The residue was dissolved in EtOAc (20 mL), washed with H_2O and saturated aqueous $NaHCO_3$, dried ($MgSO_4$) and concentrated under reduced pressure. Chromatography on SiO_2 (hexanes/EtOAc, 4:1) gave 39 mg (74%) of **S9** as a yellow-orange solid: mp 165-166 °C (hexanes/EtOAc, dec); IR (neat) 3417 (br), 1661, 1610, 1456, 1417, 1384, 1266, 1071 cm^{-1}; ^1H NMR 12.17 (s, 1H), 7.90 (dd, 1H, *J* = 8.5, 0.6 Hz), 7.65 (t, 1H, *J* = 8.0

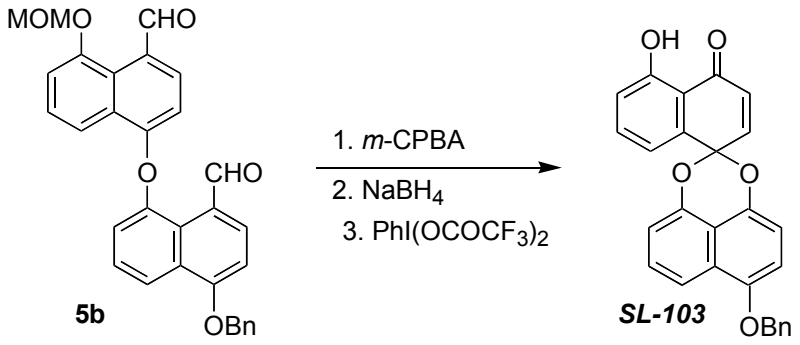
Hz), 7.46 (dd, 1H, *J* = 7.6, 1.0 Hz), 7.46 (dd, 1H, *J* = 8.4, 7.7 Hz), 7.13 (dd, 1H, *J* = 8.4, 1.0 Hz), 7.02 (dd, 1H, *J* = 7.6, 0.5 Hz), 7.01 (d, 1H, *J* = 10.5 Hz), 6.90 (d, 1H, *J* = 8.3 Hz), 6.78 (d, 1H, *J* = 8.3 Hz), 6.34 (d, 1H, *J* = 10.5 Hz), 4.00 (s, 3H); ^{13}C NMR 189.0, 162.1, 151.1, 147.5, 140.8, 140.2, 139.3, 136.8, 129.8, 127.1, 126.2, 119.8, 119.6, 116.6, 114.1, 113.8, 111.1, 109.7, 105.3, 93.0, 56.1; MS (EI) *m/z* (rel intensity) 346 (M^+, 31), 331 (16), 303 (8), 268 (11), 129 (12), 101 (12), 91 (100); HRMS (EI) calcd for $C_{21}H_{14}O_5$ 346.0841, found 346.0845.



1-(5-Benzyloxy-8-formylnaphthalen-1-yloxy)-5-methoxymethoxy-naphthalene-4-carbaldehyde (5b). To a solution of 4,8-dihydroxynaphthalene-1-carbaldehyde[5] (**6**, 474 mg, 2.52 mmol) in acetone (12 mL) was added K_2CO_3 (700 mg, 5.04 mmol) followed by benzyl bromide (0.20 mL, 1.7 mmol). The reaction mixture was heated at reflux for 2.5 h, cooled to room temperature, quenched with H_2O, and extracted with CH_2Cl_2 (3 x 30 mL). The combined organic layers were washed with H_2O, dried ($MgSO_4$), and concentrated under reduced pressure. Chromatography through a short pad of SiO_2 (CH_2Cl_2) followed by trituration with cold hexanes/EtOAc (4:1) gave 368 mg (79%) of 4-benzyloxy-8-hydroxynaphthalene-1-carbaldehyde as a yellow solid: mp 120-121 °C (hexanes/EtOAc); IR (neat) 3420 (br), 2806, 1646, 1518, 1277, 1222, 1096 cm$^-$

^{1}; ^{1}H NMR □ 12.15 (d, 1H, *J* = 1.0 Hz), 9.63 (d, 1H, *J* = 1.0 Hz), 7.97 (d, 1H, *J* = 8.1 Hz), 7.94 (dd, 1H, *J* = 8.2, 1.3 Hz), 7.52-7.39 (m, 6H), 7.20 (dd, 1H, *J* = 7.8, 1.3 Hz), 6.96 (d, 1H, *J* = 8.2 Hz), 5.37 (s, 2H); ^{13}C NMR □ 195.9, 162.3, 155.8, 145.7, 135.7, 129.1, 128.8, 128.7, 128.5, 127.7, 125.8, 122.7, 116.9, 114.0, 104.1, 71.1; MS (EI) *m/z* (rel intensity) 278 (M^+, 30), 187 (11), 170 (21), 131 (34), 114 (15), 91 (100); HRMS (EI) calcd for $C_{18}H_{14}O_3$ 278.0943, found 278.0953.

To a solution of this naphthol (250 mg, 0.898 mmol) and fluoride **4** (140 mg, 0.598 mmol) in CH_3CN (3 mL) at room temperature was added 2-*tert*-butyl-1,1,3,3-tetramethylguanidine (0.18 mL, 0.90 mmol). The reaction mixture was heated at 80 °C for 5 h, cooled to room temperature, poured into 1.0 M HCl (10 mL), and extracted with CH_2Cl_2 (3 x 15 mL). The combined organic layers were washed with H_2O, dried ($MgSO_4$), and concentrated under reduced pressure. Chromatography on SiO_2 (hexanes/EtOAc, 7:3) afforded 180 mg (61%) of **5b** as a yellow solid: mp 152-153 °C (hexanes/EtOAc); IR (neat) 2899, 1674, 1505, 1417, 1353, 1322, 1220, 1159, 1018 cm^{-1}; ^{1}H NMR □ 11.09 (s, 1H), 10.94 (s, 1H), 8.36 (dd, 1H, *J* = 8.4, 1.1 Hz), 8.18 (d, 1H, *J* = 8.3 Hz), 8.11 (dd, 1H, *J* = 8.3, 0.9 Hz), 7.88 (d, 1H, *J* = 8.1 Hz), 7.55-7.37 (m, 8H), 7.22 (dd, 1H, *J* = 7.6, 1.1 Hz), 7.06 (d, 1H, *J* = 8.3 Hz), 6.80 (d, 1H, *J* = 8.1 Hz), 5.43 (s, 2H), 5.36 (s, 2H), 3.56 (s, 3H); ^{13}C NMR □ 194.2, 192.5, 159.3, 157.3, 154.3, 152.7, 136.2, 131.1, 130.7, 129.0, 128.8, 128.7, 128.2, 127.7, 127.4, 127.0, 126.6, 126.2, 125.7, 120.3, 119.3, 116.4, 112.1, 111.8, 105.8, 95.4, 71.1, 56.8; MS (EI) *m/z* (rel intensity) 492 (M^+, 24), 447 (38), 277 (8), 171 (22), 114 (10), 91 (100); HRMS (EI) calcd for $C_{31}H_{24}O_6$ 492.1573, found 492.1570.



4'-Benzyloxypalmarumycin CP_1 (S10). To a solution of dialdehyde **5b** (55 mg, 0.11 mmol) in CH_2Cl_2 (3 mL) was added 70% *m*-CPBA (81 mg, 0.33 mmol). The reaction mixture was stirred at room temperature for 15 h. A solution of 10% $Na_2S_2O_3$ (5 mL) was added and stirring was continued for 1 h. The organic layer was separated and diluted with CH_2Cl_2, washed with saturated aqueous $NaHCO_3$, dried ($MgSO_4$), and concentrated under reduced pressure. Chromatography of the residue on SiO_2 (hexanes/EtOAc, 7:3) afforded 40 mg (69%) of 1-(5-benzyloxy-8-formyloxynaphthalen-1-yloxy)-5-methoxymethoxy-4-formyloxynaphthalene as a pale orange foam: IR (neat) 2934, 1739, 1601, 1506, 1415, 1370, 1256, 1118 cm^{-1}; ^1H NMR 8.39 (s, 1H), 8.25 (dd, 1H, *J* = 8.5, 1.0 Hz), 8.08 (s, 1H), 8.00 (dd, 1H, *J* = 8.5, 0.9 Hz), 7.56-7.36 (m, 7H), 7.25 (dd, 1H, *J* = 7.8, 0.7 Hz), 7.07 (d, 1H, *J* = 8.3 Hz), 7.02-6.99 (m, 2H), 6.92 (d, 1H, *J* = 8.4 Hz), 6.68 (d, 1H, *J* = 8.3 Hz), 5.33 (s, 2H), 5.28 (s, 2H), 3.56 (s, 3H); ^{13}C NMR 160.8, 160.6, 153.2, 152.7, 152.4, 151.5, 141.0, 138.0, 136.8, 129.3, 129.1, 128.9, 128.4, 127.7, 127.5, 126.4, 121.1, 120.2, 120.0, 119.5, 119.3, 117.8, 116.3, 112.1, 111.6, 105.4, 95.3, 70.8, 56.7; MS (EI) *m/z* (rel intensity) 524 (M^+, 47), 496 (22), 373 (33), 345 (61), 315 (19), 172 (23), 159 (30), 91 (100); HRMS (EI) calcd for $C_{31}H_{24}O_8$ 524.1471, found 524.1467.

To a solution of this bisformate ester (35 mg, 0.066 mmol) in 1:1 MeOH/THF (3 mL) at 0 °C was slowly added $NaBH_4$ (6.0 mg, 0.15 mmol). The reaction mixture was stirred at 0 °C for 1 h, diluted with EtOAc (10 mL) and washed with H_2O. The organic layer was dried ($MgSO_4$) and concentrated under reduced pressure. The residue was *immediately* subjected to chromatography on SiO_2 (hexanes/EtOAc, 3:2) to afford 28 mg (90%) of 1-(5-benzyloxy-8-hydroxynaphthalen-1-yloxy)-5-methoxymethoxynaphth-4-ol as a white foam: IR (neat) 3434 (br), 1608, 1460, 1409, 1263, 1226, 1031 cm^{-1}; ^1H NMR 9.37 (s, 1H), 8.94 (s, 1H), 8.00 (dd, 1H, *J* = 8.5, 1.0 Hz), 7.64 (dd, 1H, *J* = 8.5, 0.9 Hz), 7.55-7.52 (m, 2H), 7.45-7.35 (m, 3H), 7.30 (t, 1H, *J* = 8.1 Hz), 7.26 (d, 1H, *J* = 8.3 Hz), 7.16 (d, 1H, *J* = 8.4 Hz), 7.13 (d, 1H, *J* = 7.8 Hz), 6.92 (d, 1H, *J* = 8.3 Hz), 6.91 (s, 2H), 6.49 (dd, 1H, *J* = 7.7, 1.0 Hz), 5.48 (s, 2H), 5.22 (s, 2H), 3.62 (s, 3H); ^{13}C NMR 156.5, 154.2, 152.6, 148.1, 147.7, 141.8, 137.6, 130.1, 128.8, 128.7, 128.1, 127.6, 127.3, 125.4, 120.6, 117.4, 116.8, 116.4, 115.9, 110.1, 109.7, 109.6, 109.0, 108.2, 96.1, 71.0, 57.2; MS (EI) *m/z* (rel intensity) 468 (M^+, 34), 424 (9), 377 (10), 345 (39), 310 (8), 278 (14), 159 (11), 91 (100); HRMS (EI) calcd for $C_{29}H_{24}O_6$ 468.1573, found 468.1594.

To a solution of this bisnaphthol (46 mg, 0.10 mmol) in dry CH_3CN (3 mL) at 0 °C was added $PhI(OCOCF_3)_2$ (47 mg, 0.11 mmol) in one portion. The reaction mixture was stirred at 0 °C for 30 min and concentrated under reduced pressure. The residue was dissolved in EtOAc (20 mL), washed with H_2O and saturated aqueous $NaHCO_3$, dried ($MgSO_4$), and concentrated under reduced pressure. Chromatography on SiO_2 (hexanes/EtOAc, 7:3) gave 27 mg (66%) of **S10** as an orange-yellow solid: mp 174-176 °C (hexanes/EtOAc, dec); IR (neat) 3416 (br), 1661, 1610, 1455, 1422, 1373, 1267 cm^{-1}; ^1H NMR 12.17 (s, 1H), 7.99 (d, 1H, *J* = 8.5 Hz), 7.66 (t, 1H, *J* = 8.0 Hz), 7.55-7.35

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References:

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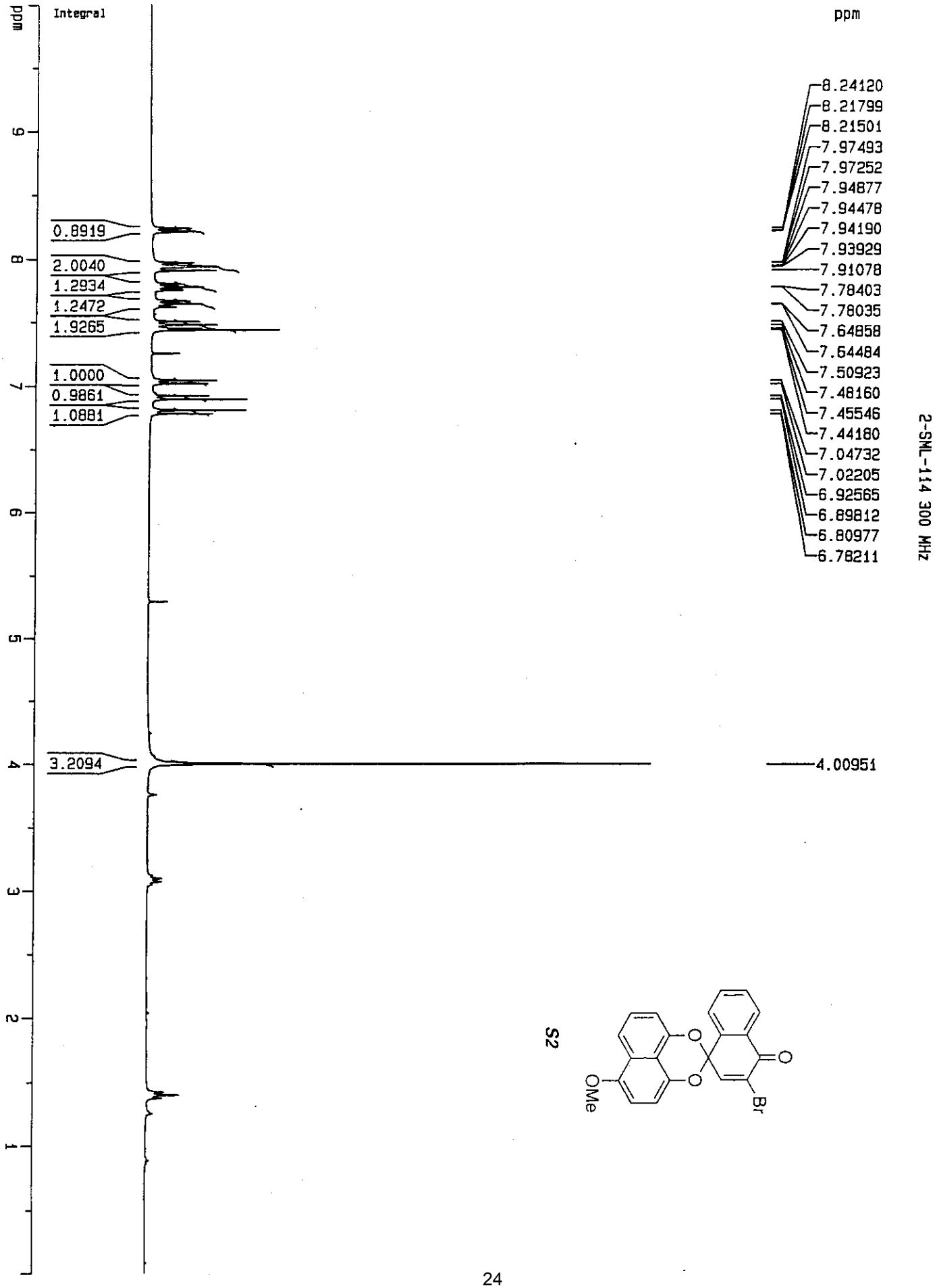
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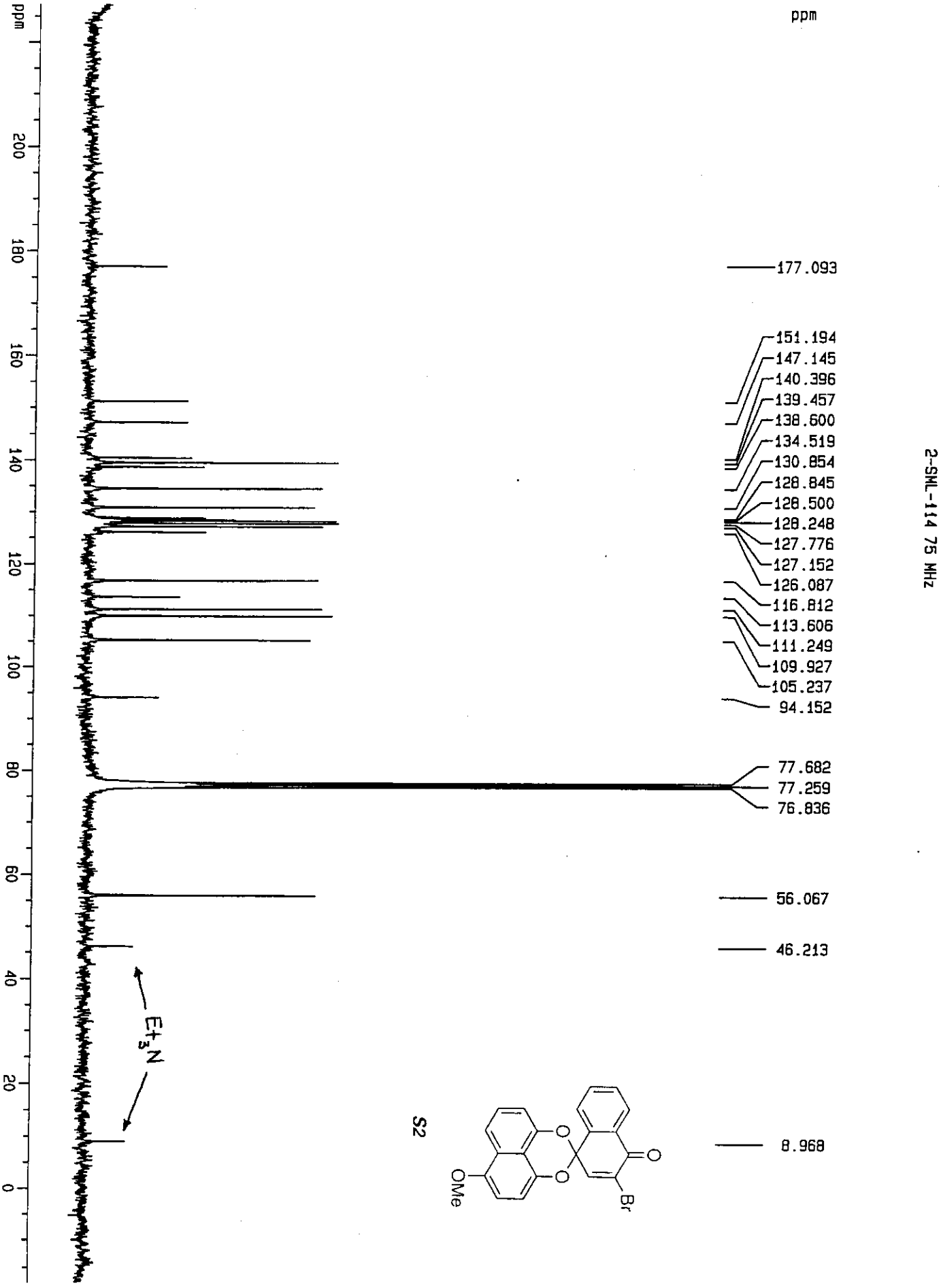
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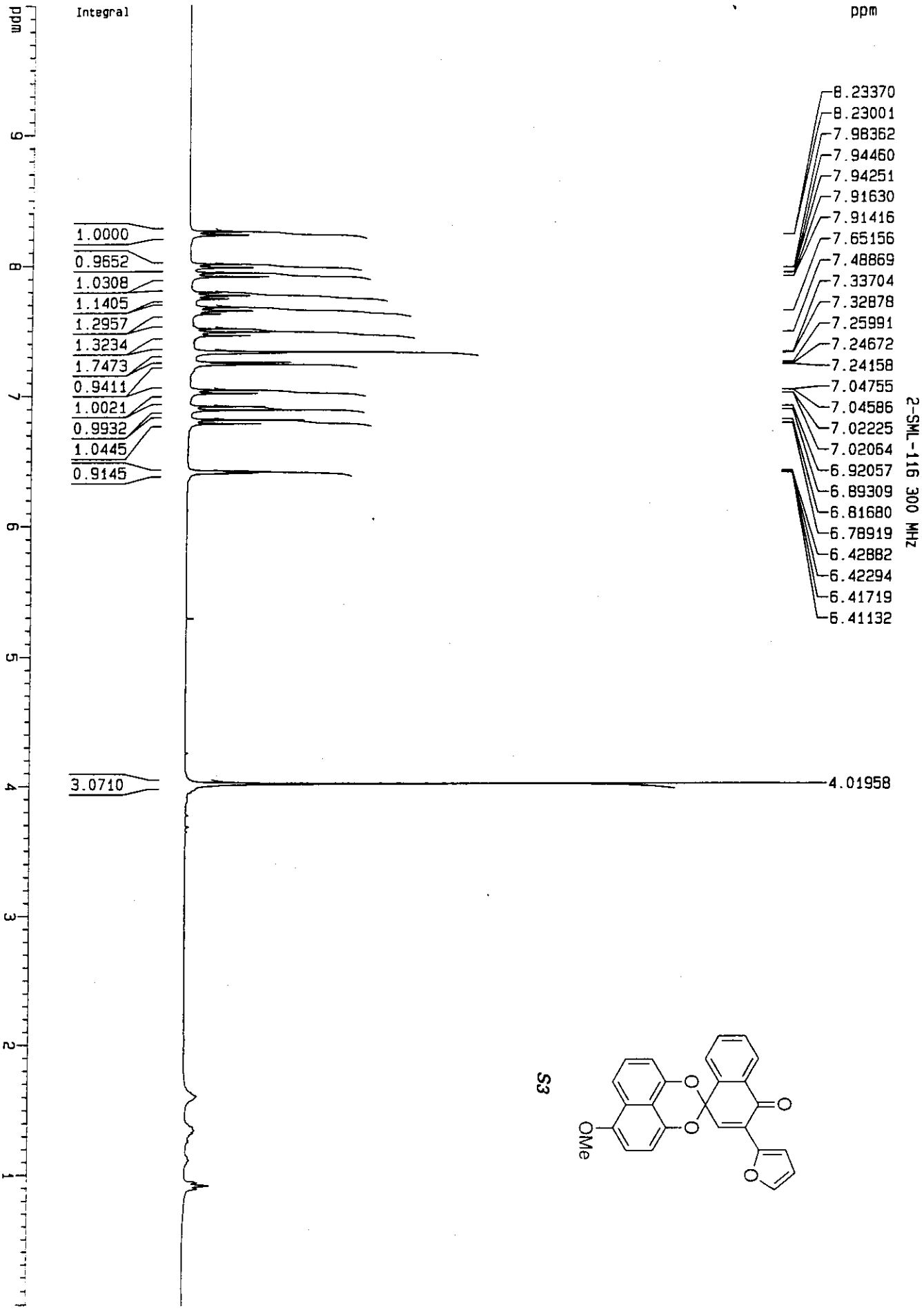
S2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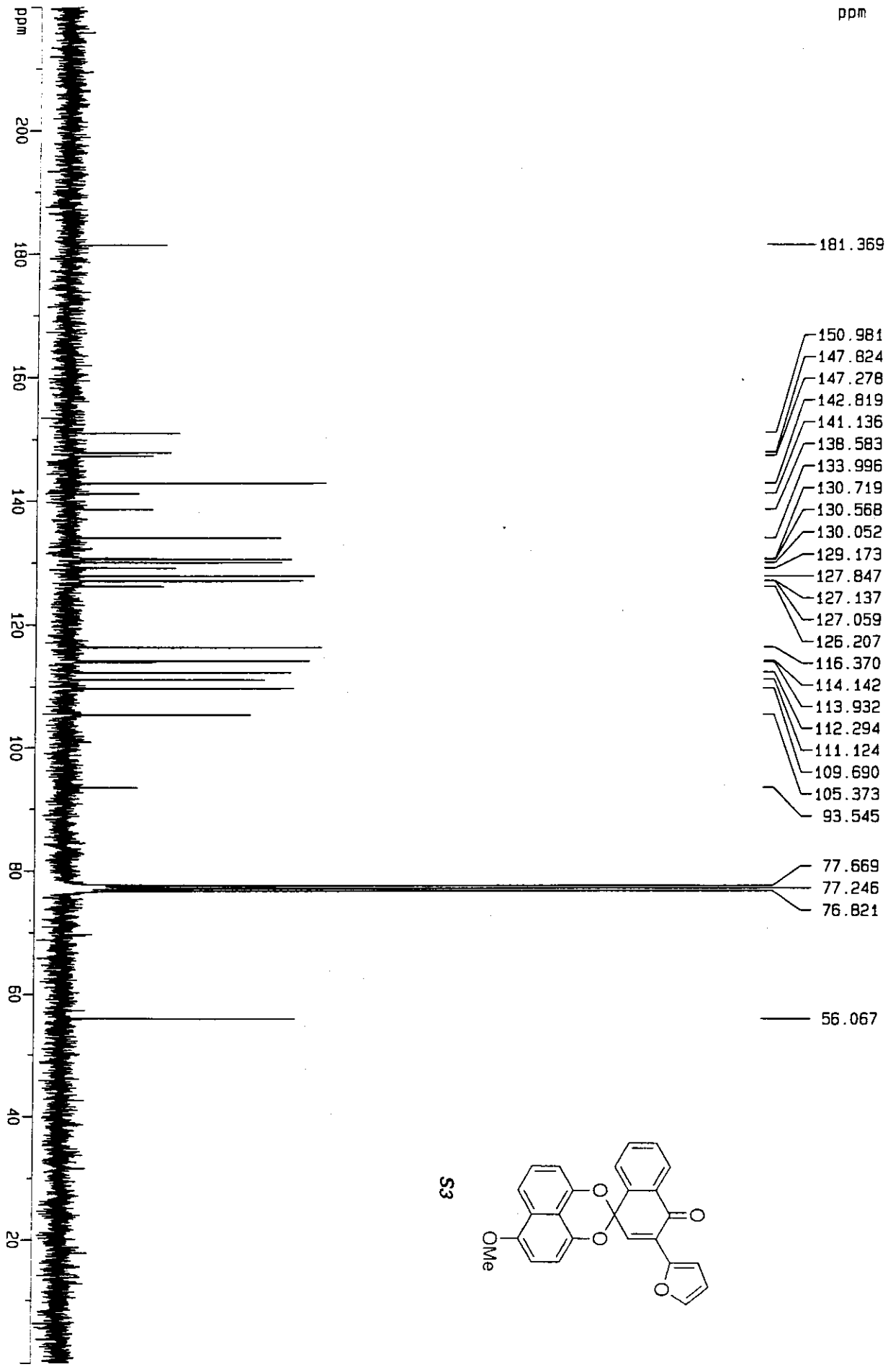
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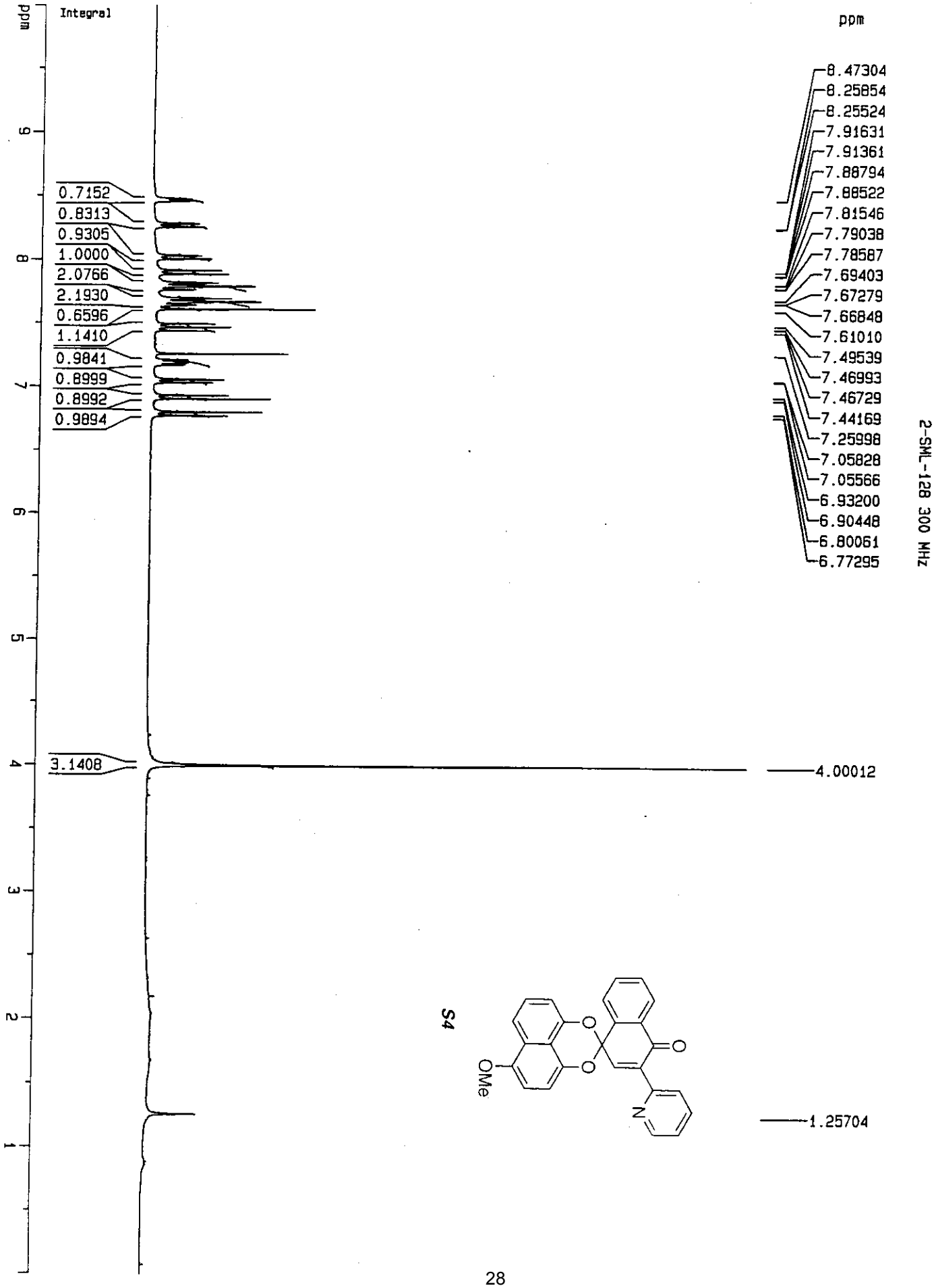
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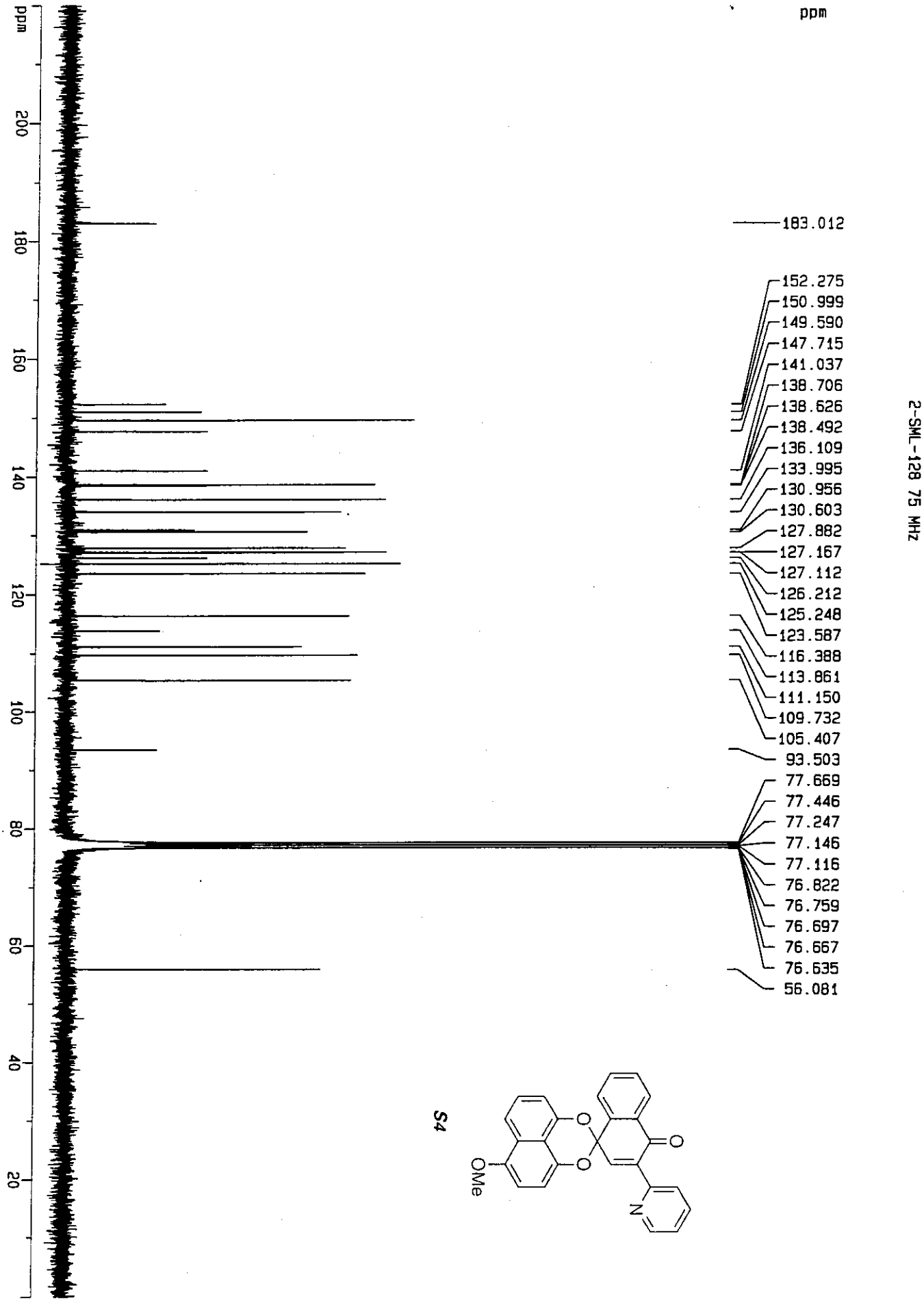
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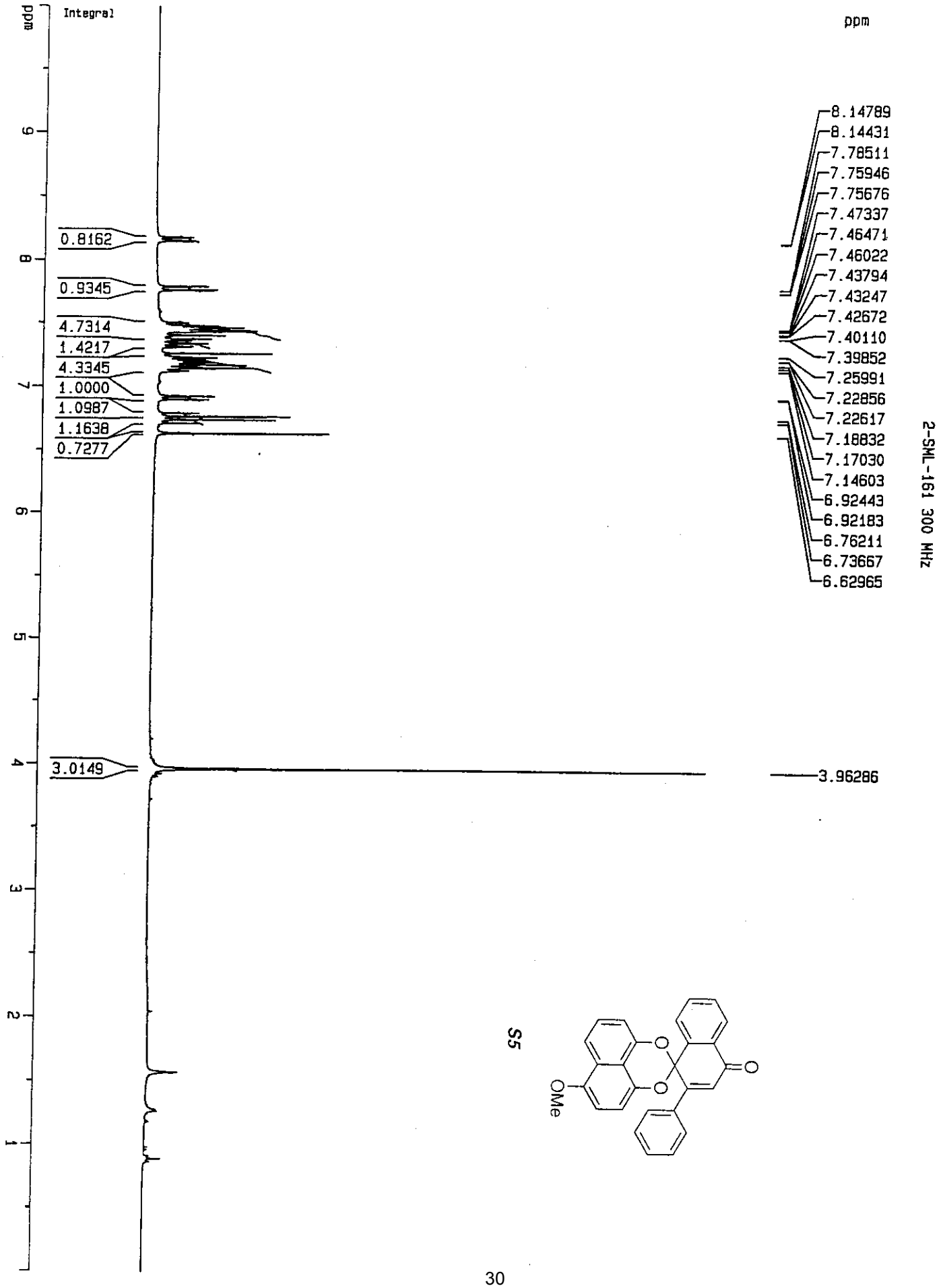
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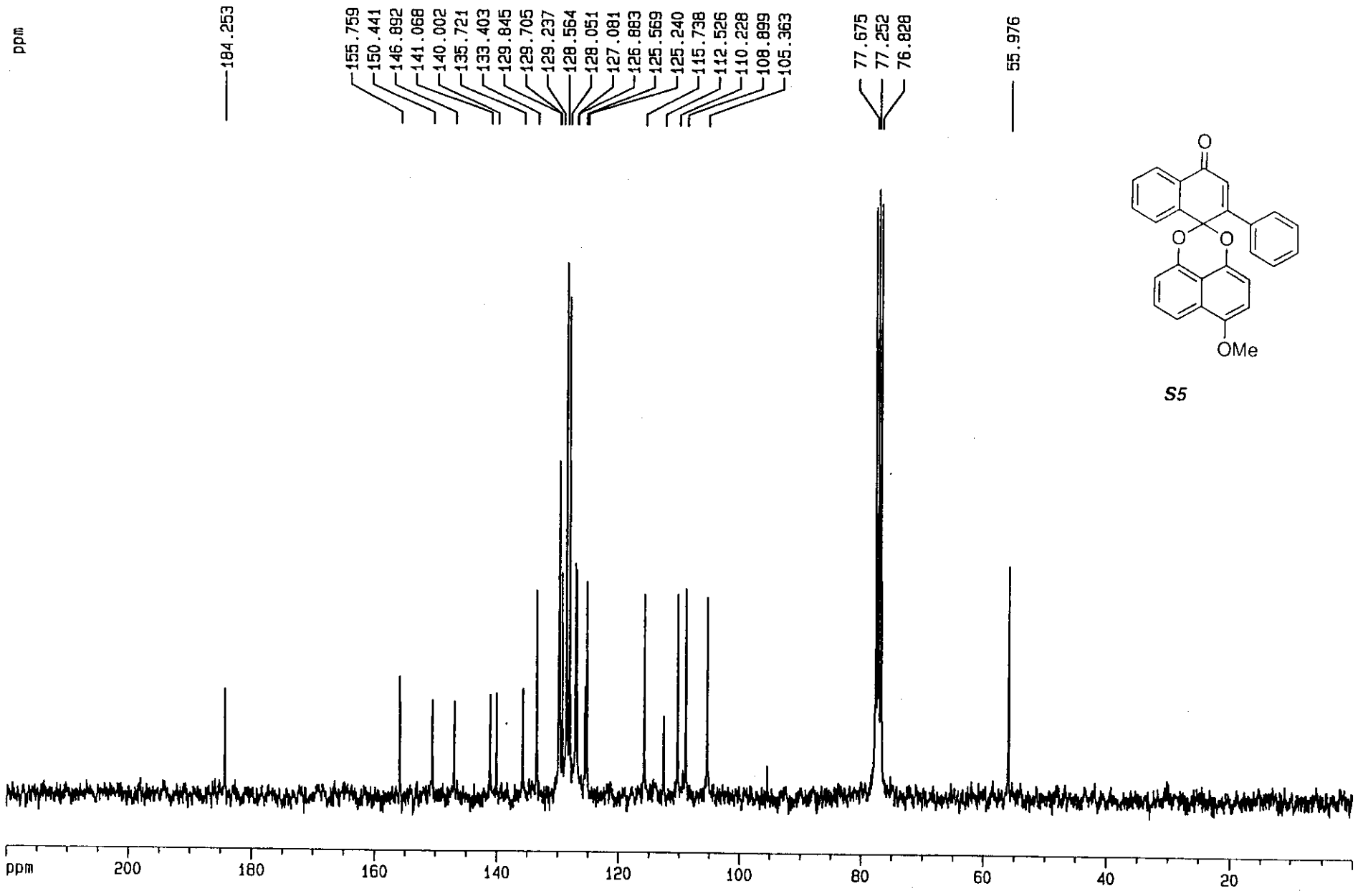

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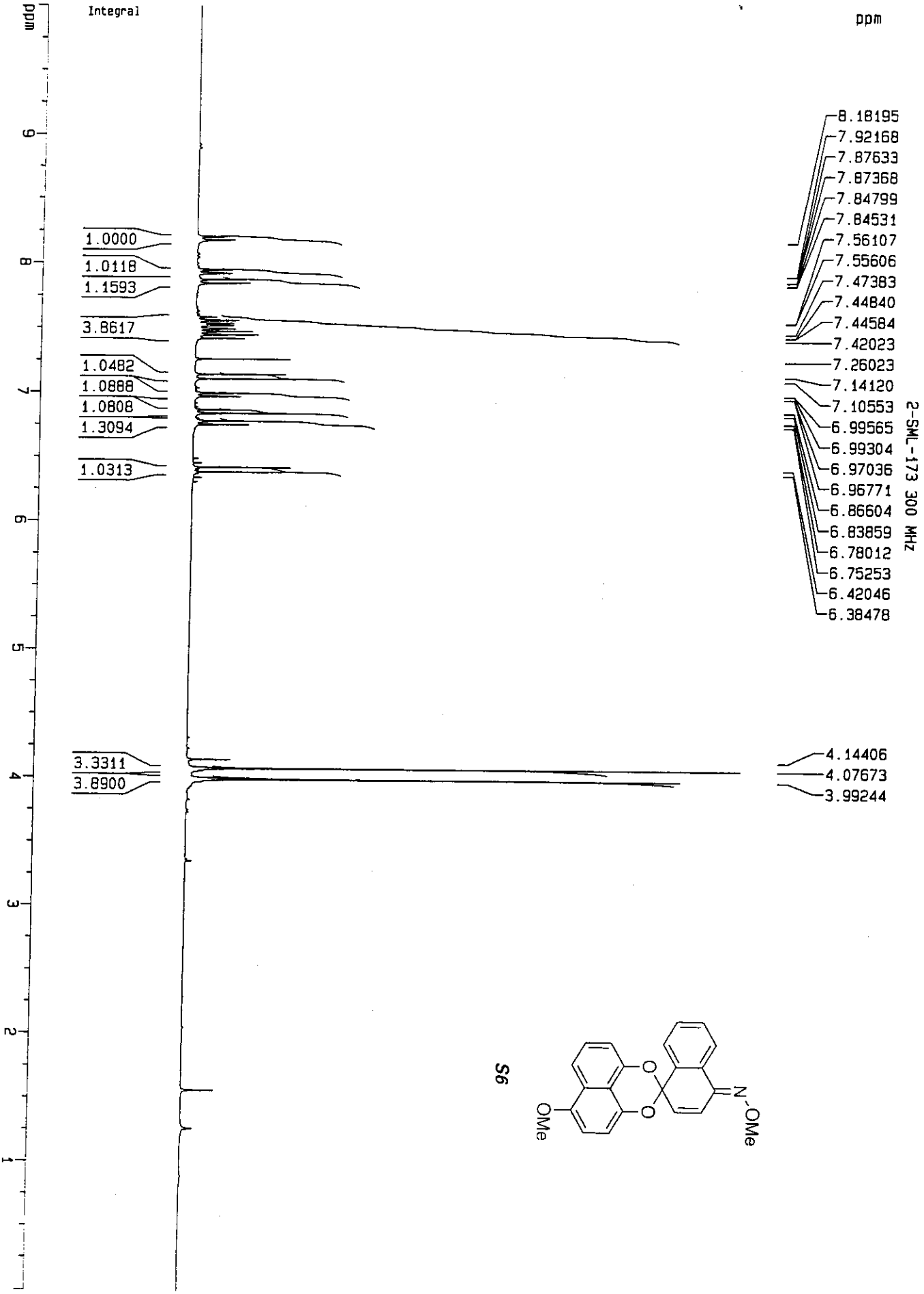
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S6

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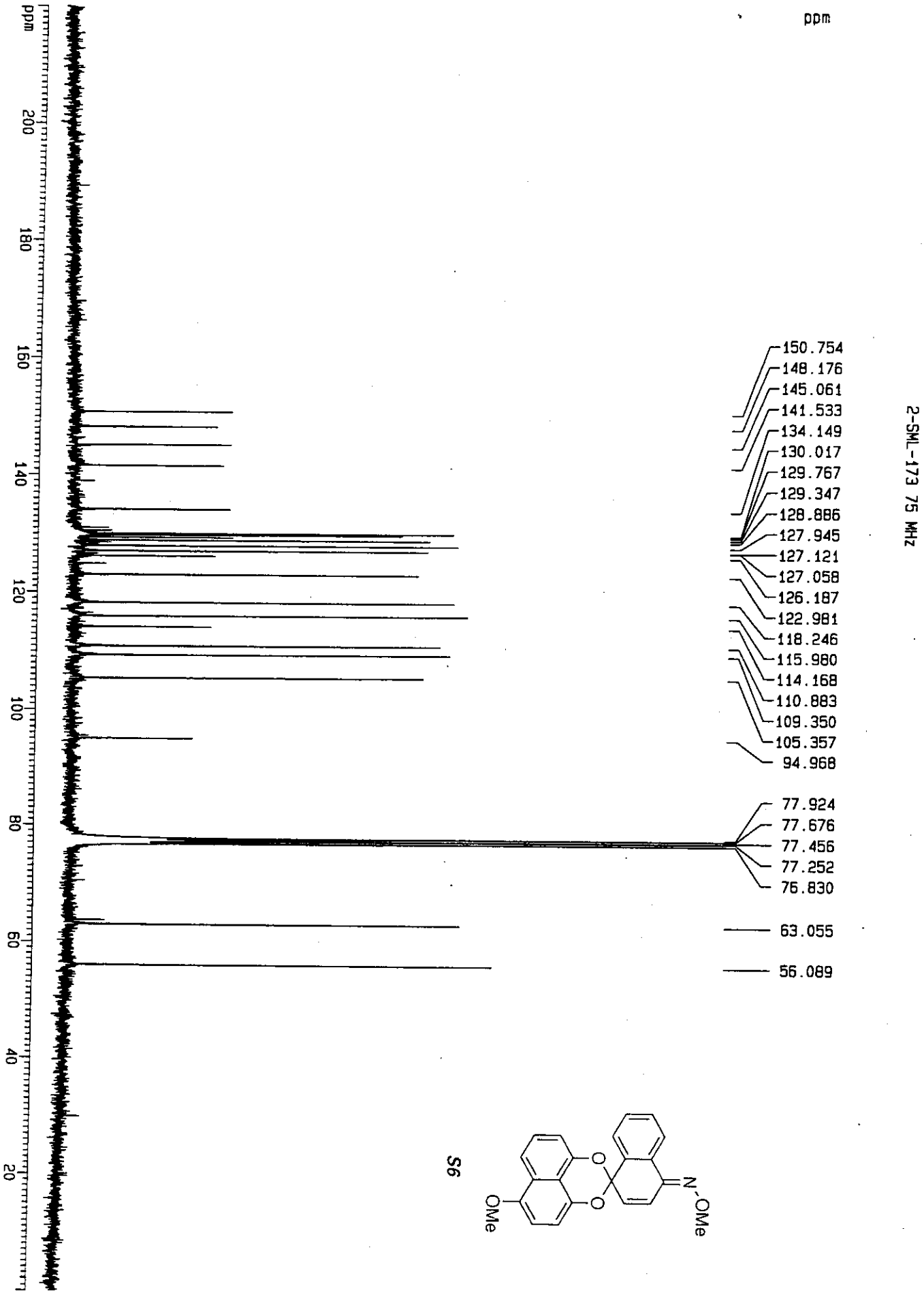
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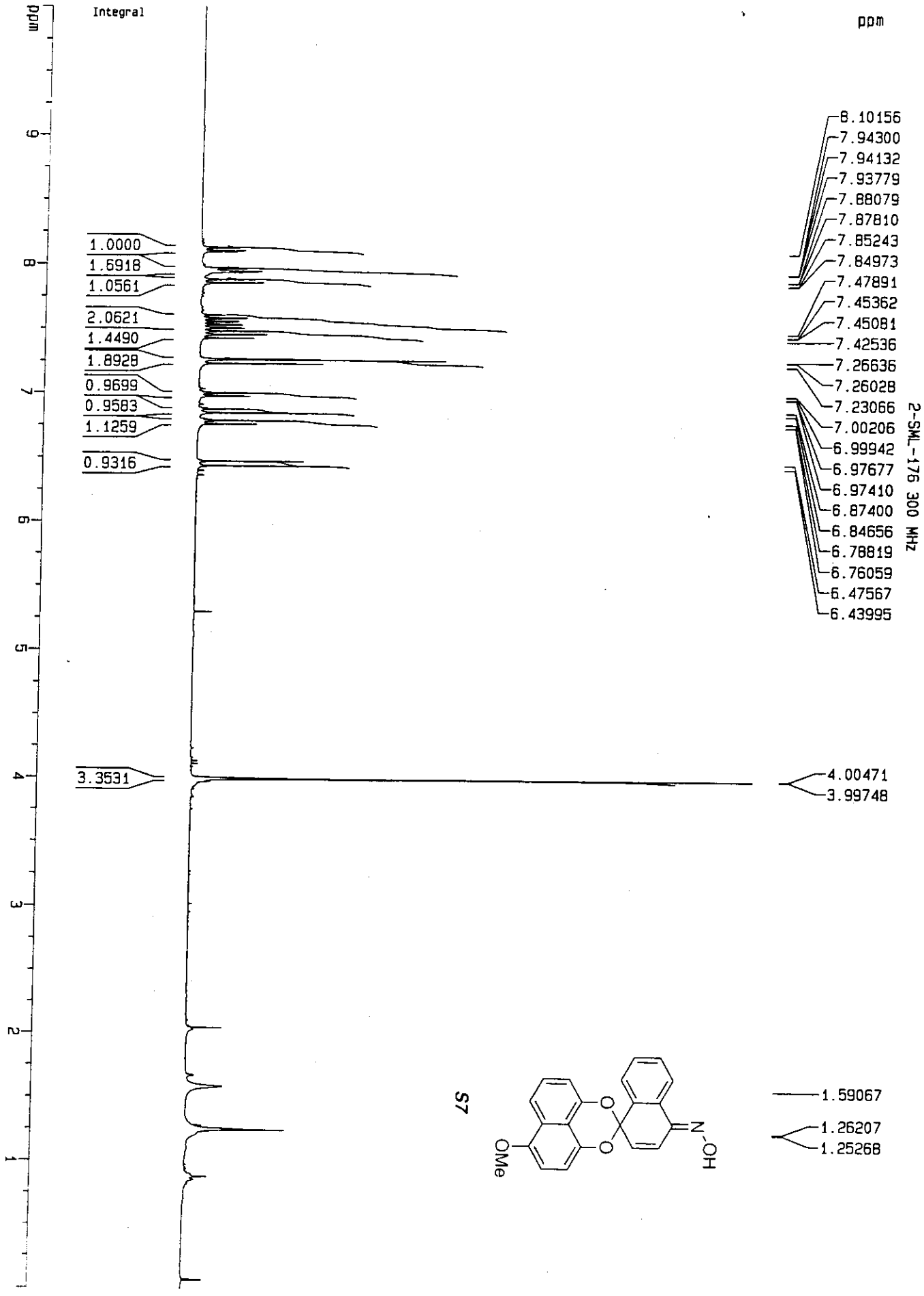
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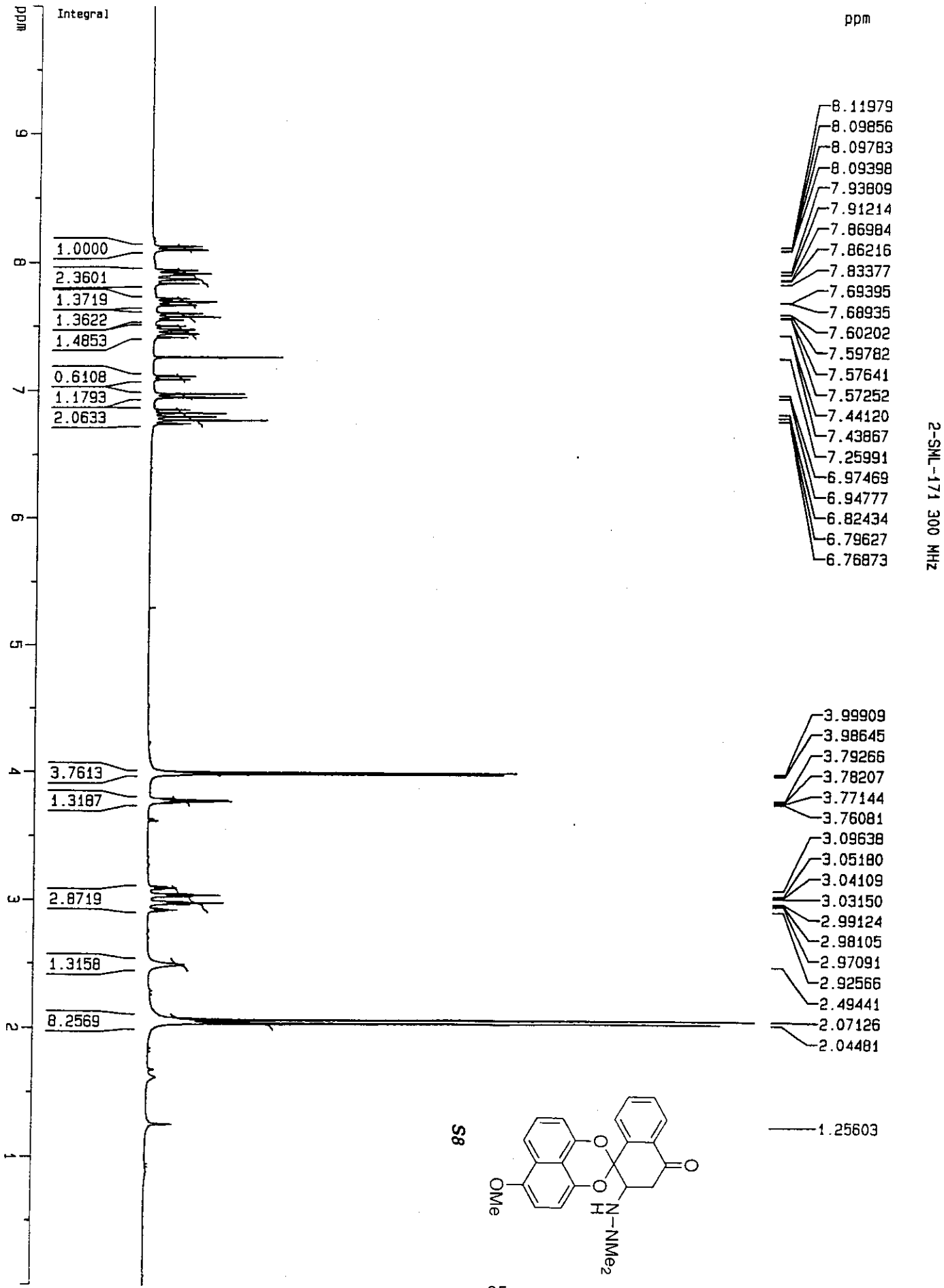
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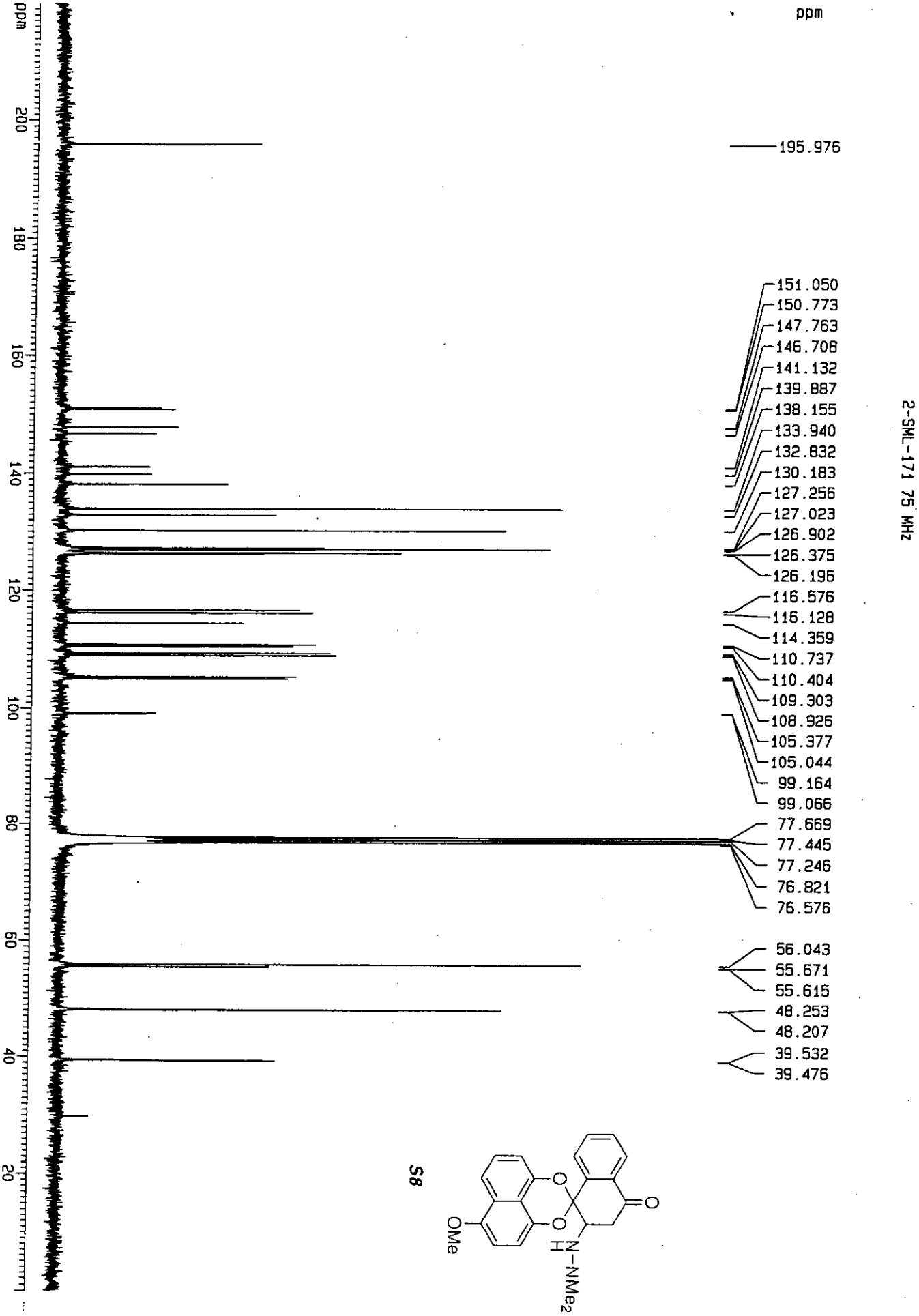
S8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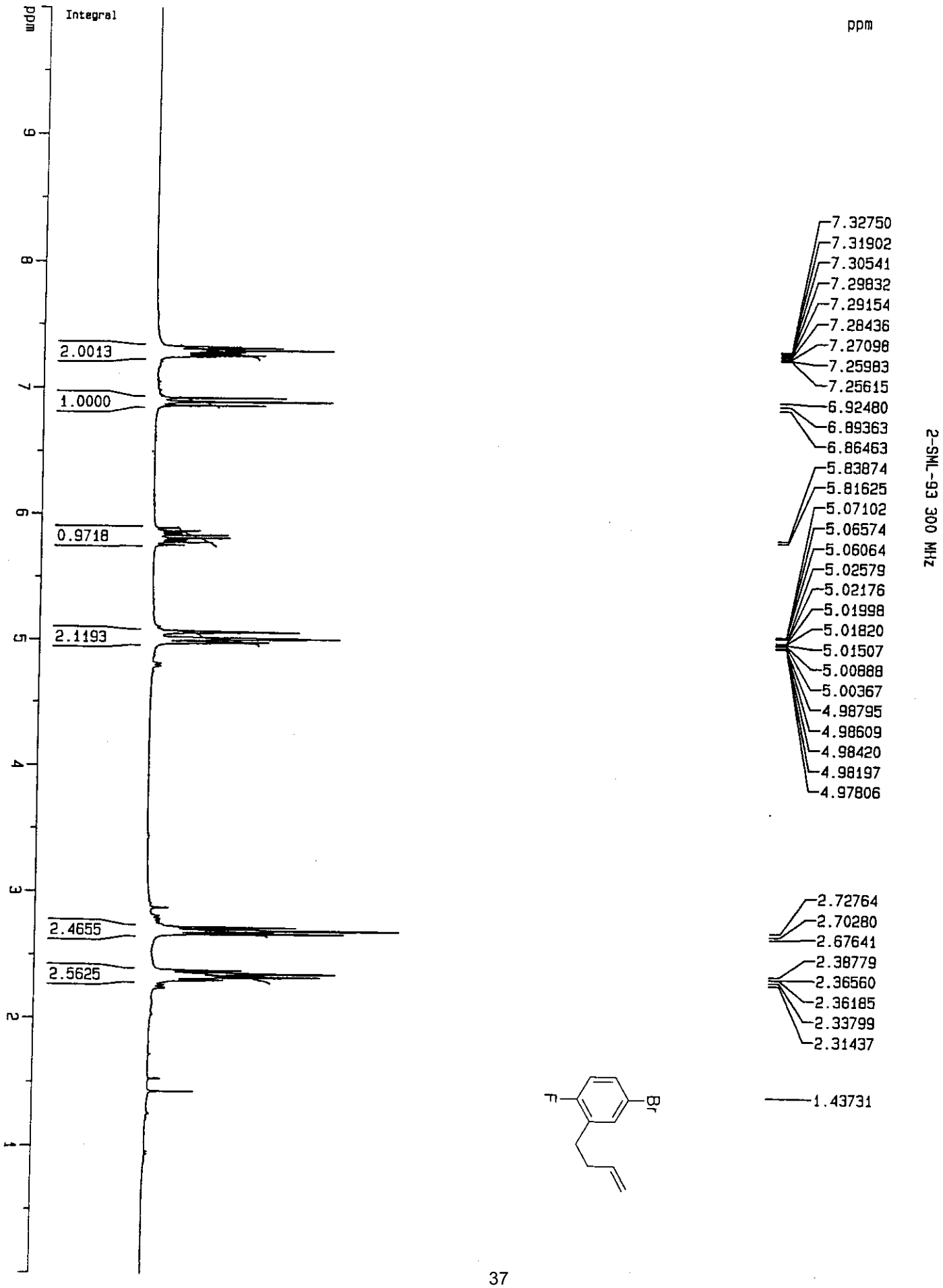
S8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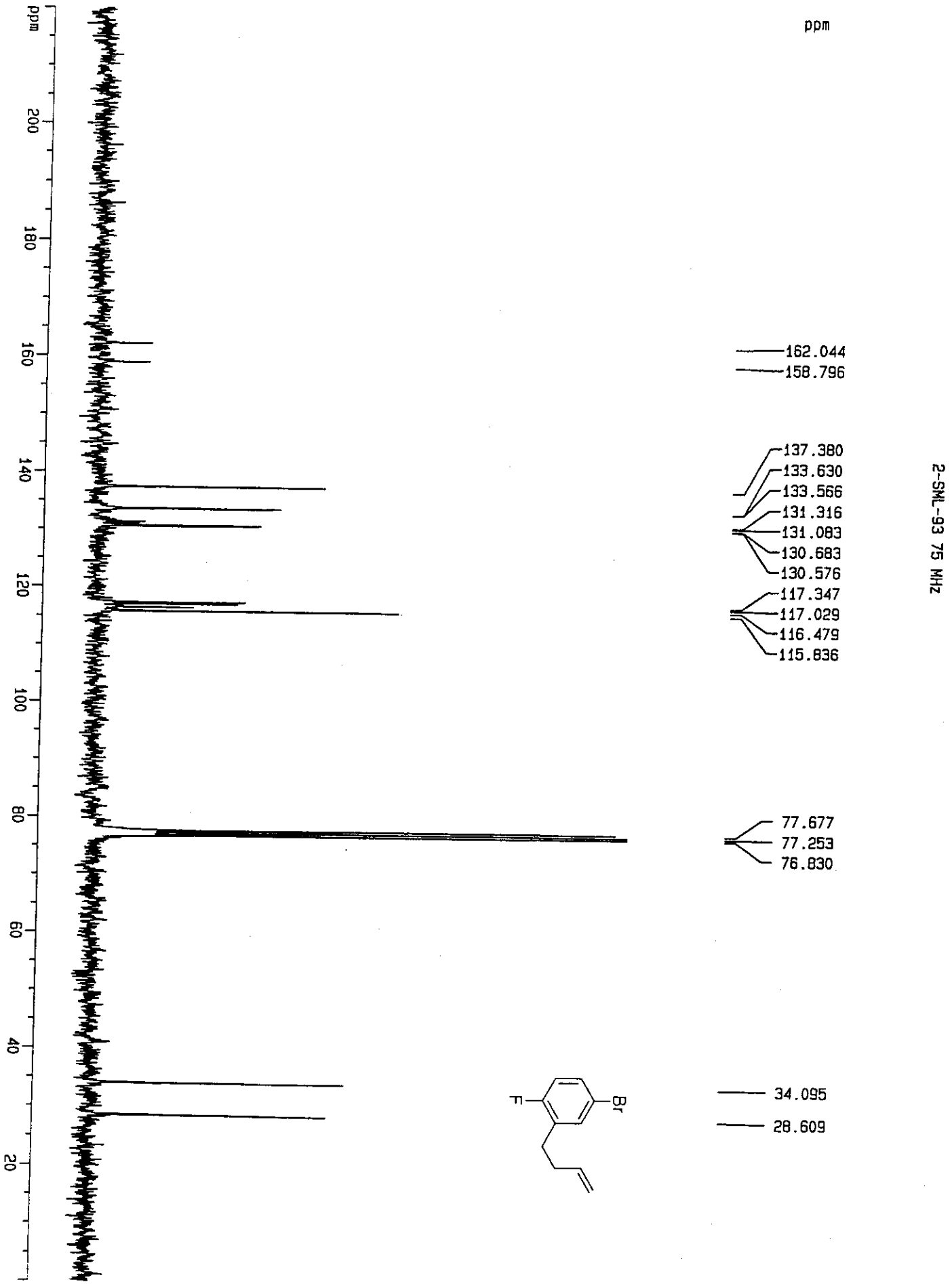
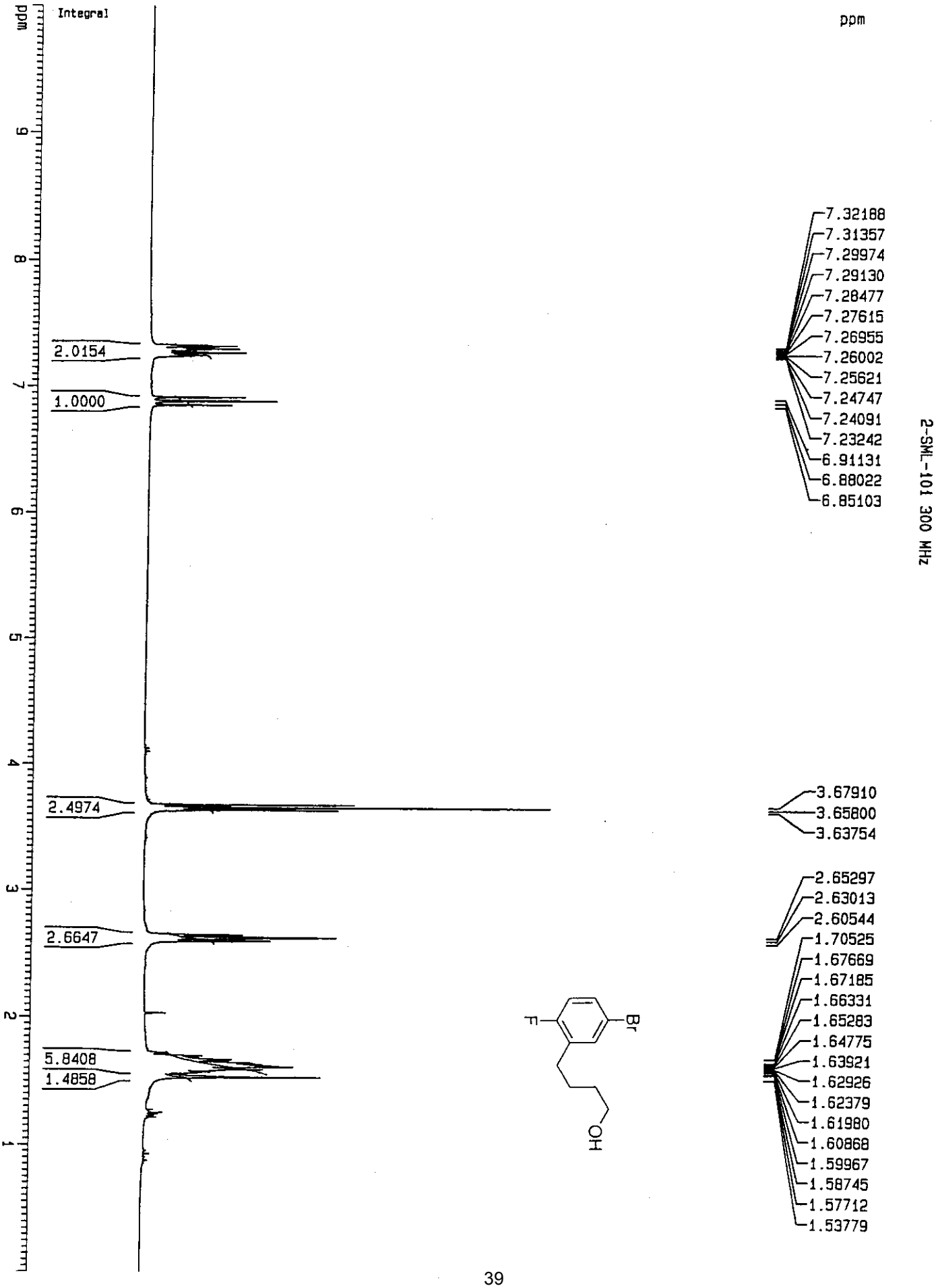
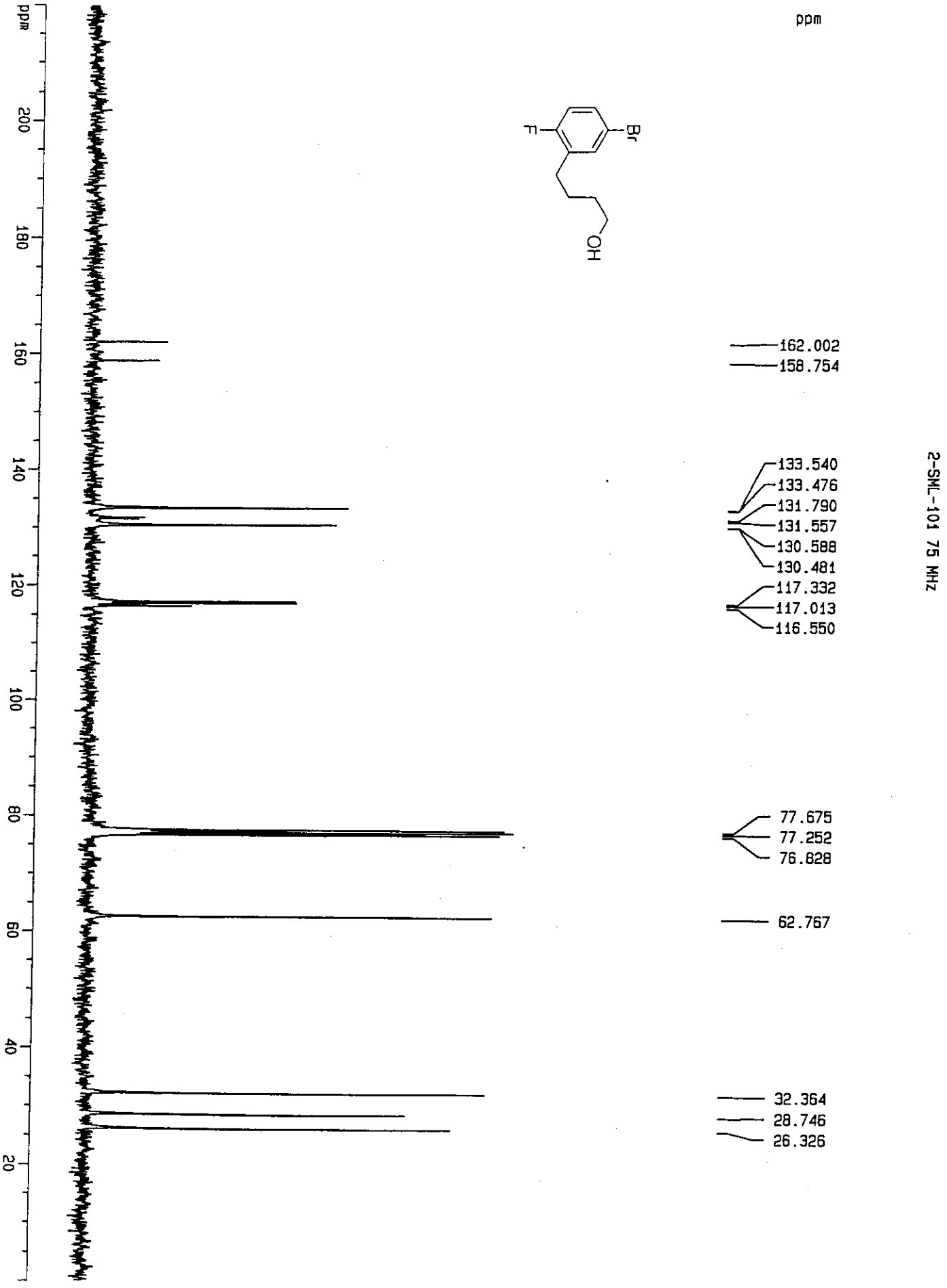
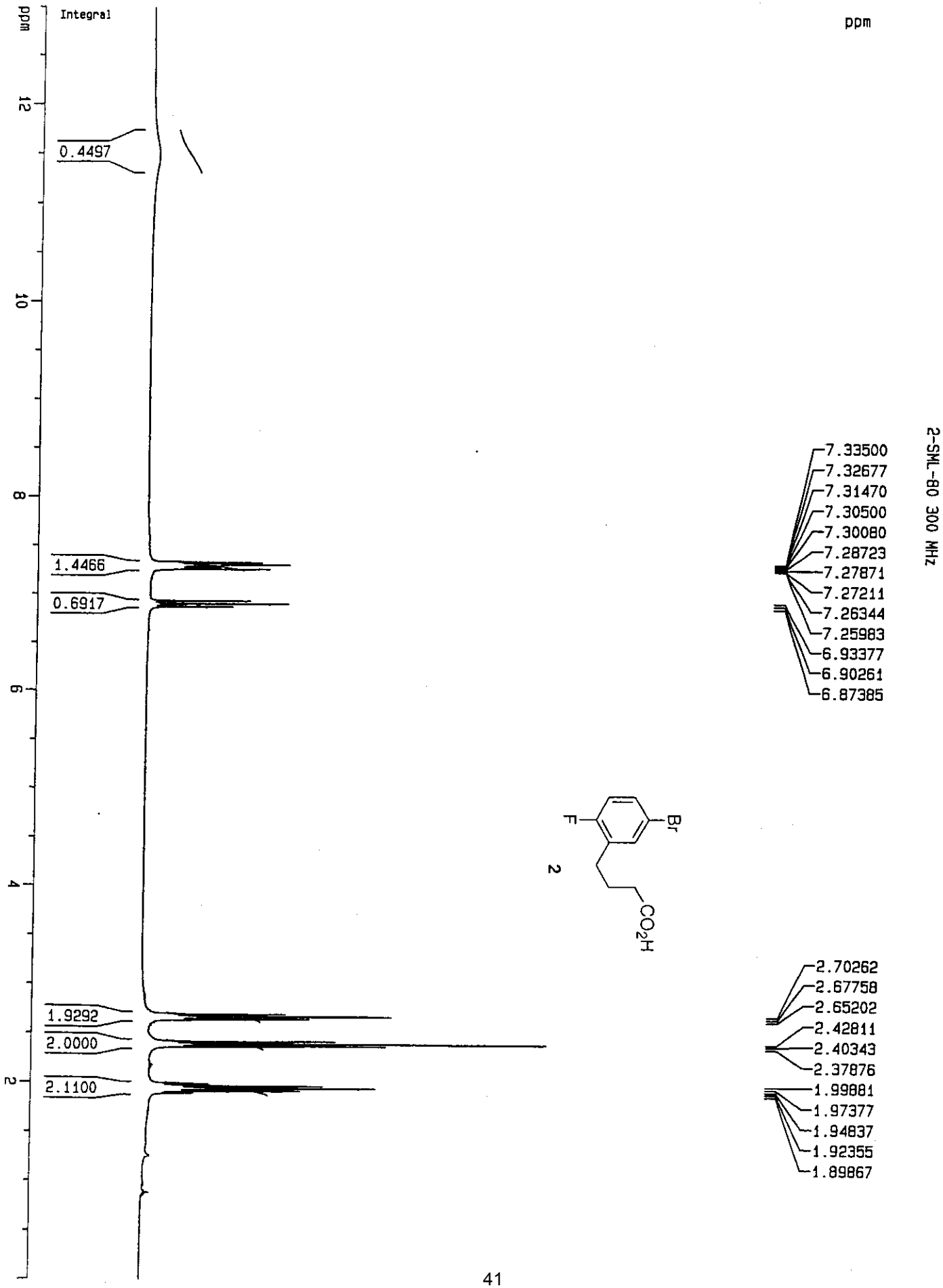
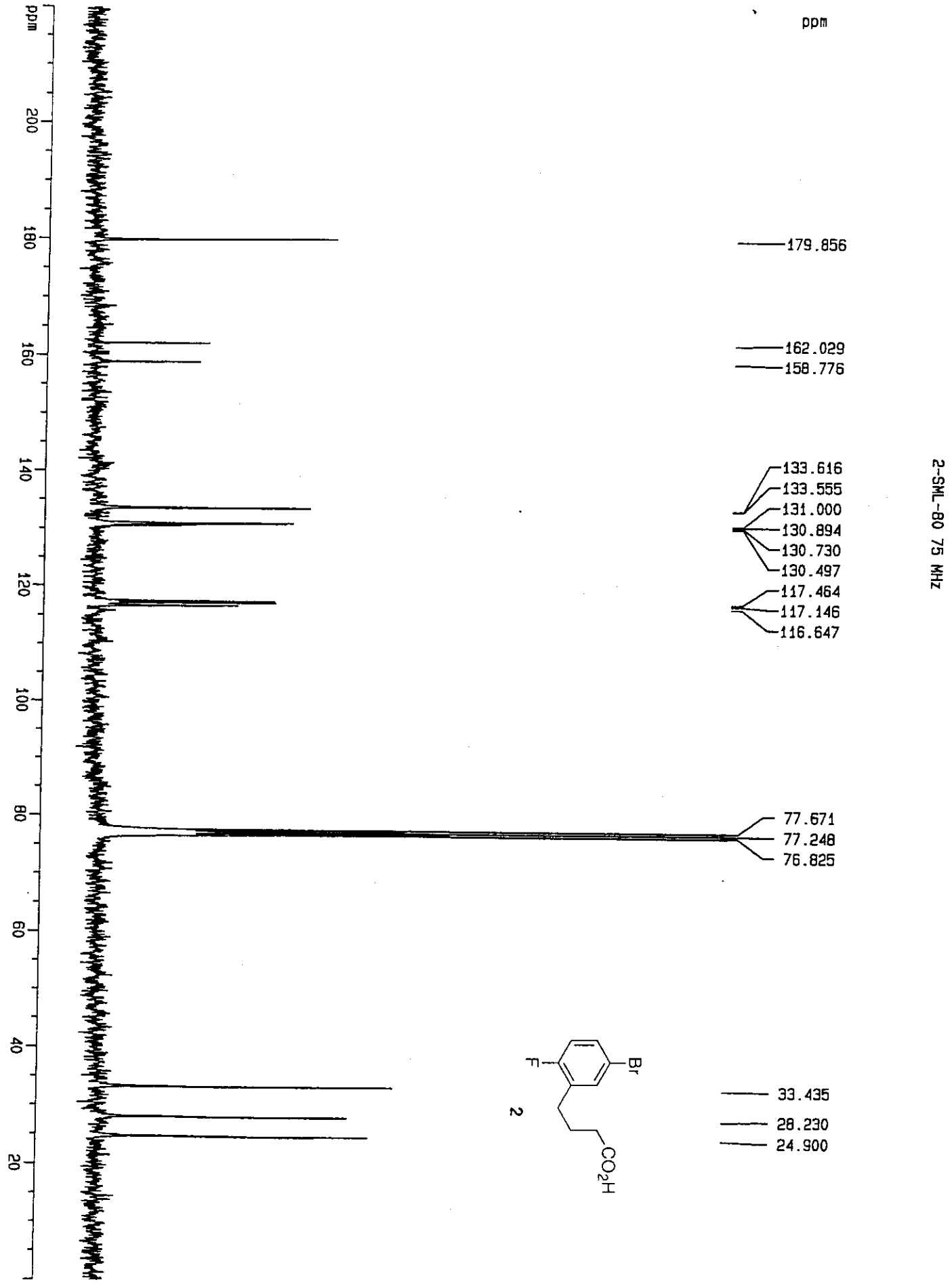
Br
2-SML-93 300 MHz

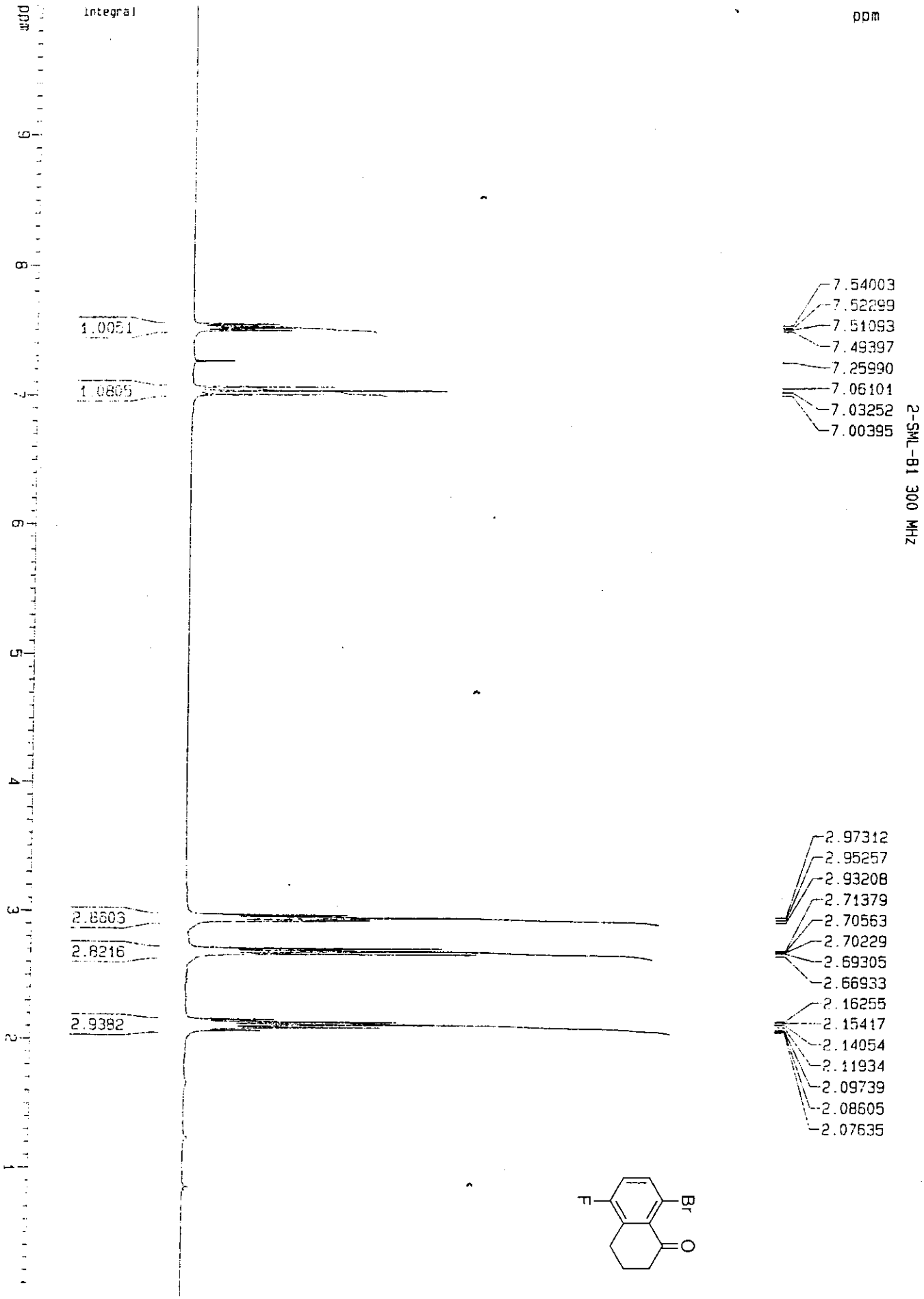

ppm
200
180
160
140
120
100
80
60
40
20
ppm
162.044
158.796
137.380
133.630
133.566
131.316
131.083
130.683
130.576
117.347
117.029
116.479
115.836
77.677
77.253
76.830
34.095
28.609
F
Br
2-SML-93 75 MHz

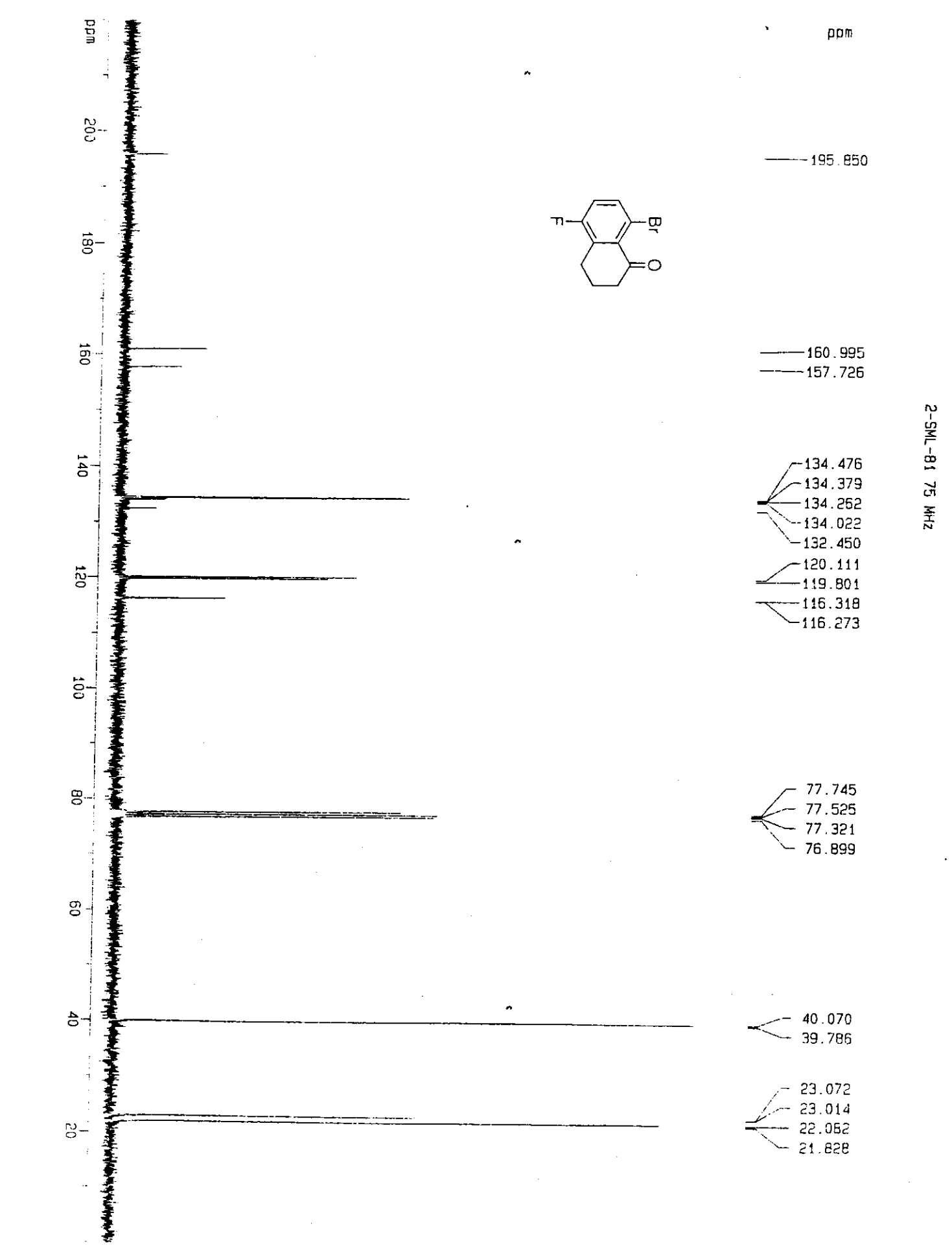

Integral
ppm
2.0154
1.0000
2.4974
2.6647
5.8408
1.4858
7.32188
7.31357
7.29974
7.29130
7.28477
7.27615
7.26955
7.26002
7.25621
7.24747
7.24091
7.23242
6.91131
6.88022
6.85103
3.67910
3.65800
3.63754
2.65297
2.63013
2.60544
1.70525
1.67669
1.67185
1.66331
1.65283
1.64775
1.63921
1.62926
1.62379
1.61980
1.60868
1.59967
1.58745
1.57712
1.53779
F
Br
OH
2-SML-101 300 MHz

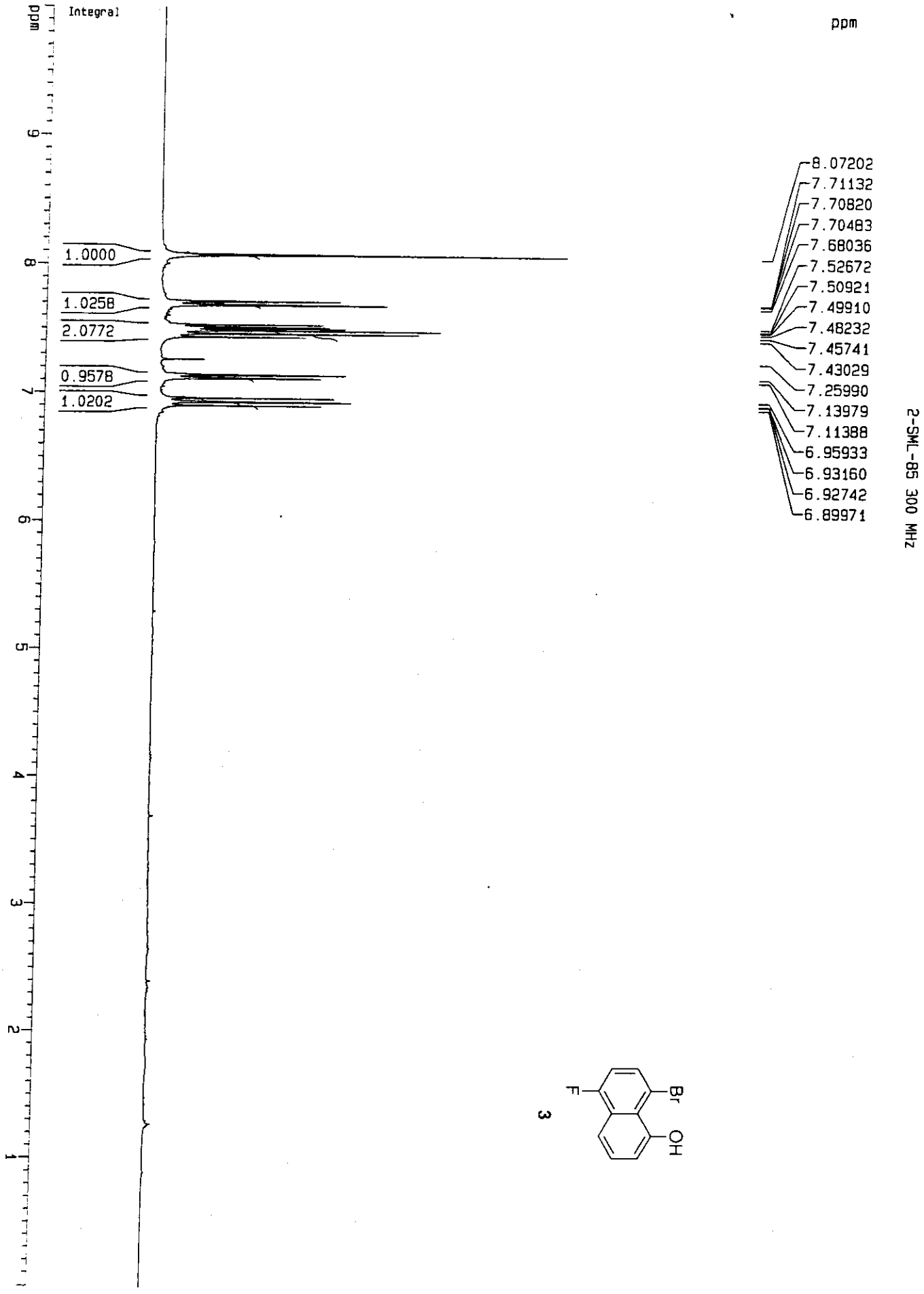

ppm
200
180
160
140
120
100
80
60
40
20
F
Br
OH
ppm
162.002
158.754
133.540
133.476
131.790
131.557
130.588
130.481
117.332
117.013
116.550
77.675
77.252
76.828
62.767
32.364
28.746
26.326
2-SML-101 75 MHz


Integral
ppm
12
10
8
6
4
2
0.4497
1.4466
0.6917
1.9292
2.0000
2.1100
ppm
7.33500
7.32677
7.31470
7.30500
7.30080
7.28723
7.27871
7.27211
7.26344
7.25983
6.93377
6.90261
6.87385
2-SML-80 300 MHz
F
Br
CO2H
2
2.70262
2.67758
2.65202
2.42811
2.40343
2.37876
1.99881
1.97377
1.94837
1.92355
1.89867


ppm
200
180
160
140
120
100
80
60
40
20
179.856
162.029
158.776
133.616
133.555
131.000
130.894
130.730
130.497
117.464
117.146
116.647
77.671
77.248
76.825
33.435
28.230
24.900
F
Br
CO2H
2
2-SML-80 75 MHz

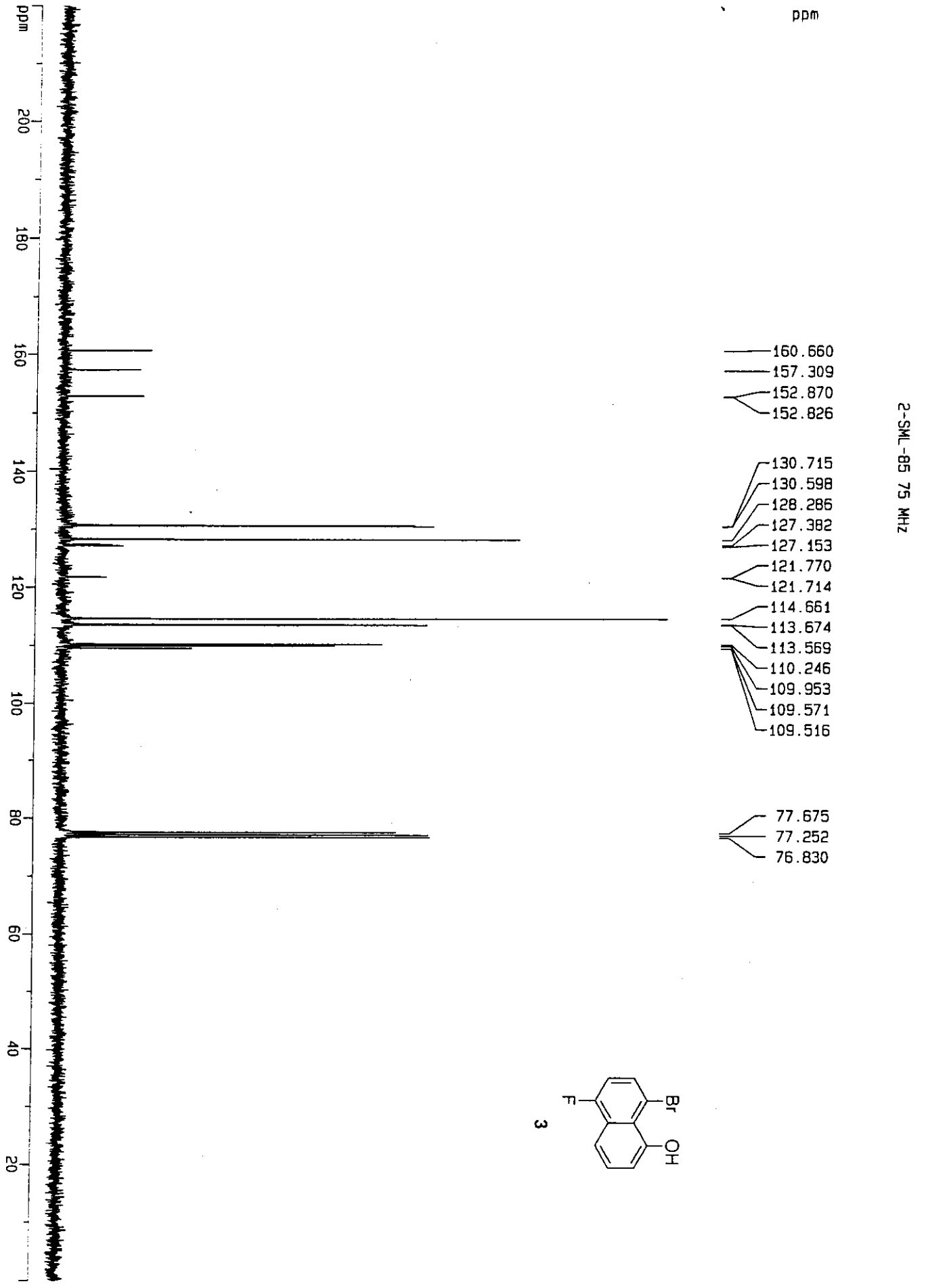

Integral
ppm
1.0051
1.0805
2.8603
2.8216
2.9382
7.54003
7.52299
7.51093
7.49397
7.25990
7.06101
7.03252
7.00395
2-SML-B1 300 MHz
2.97312
2.95257
2.93208
2.71379
2.70563
2.70229
2.69305
2.66933
2.16255
2.15417
2.14054
2.11934
2.09739
2.08605
2.07635
F
Br
O

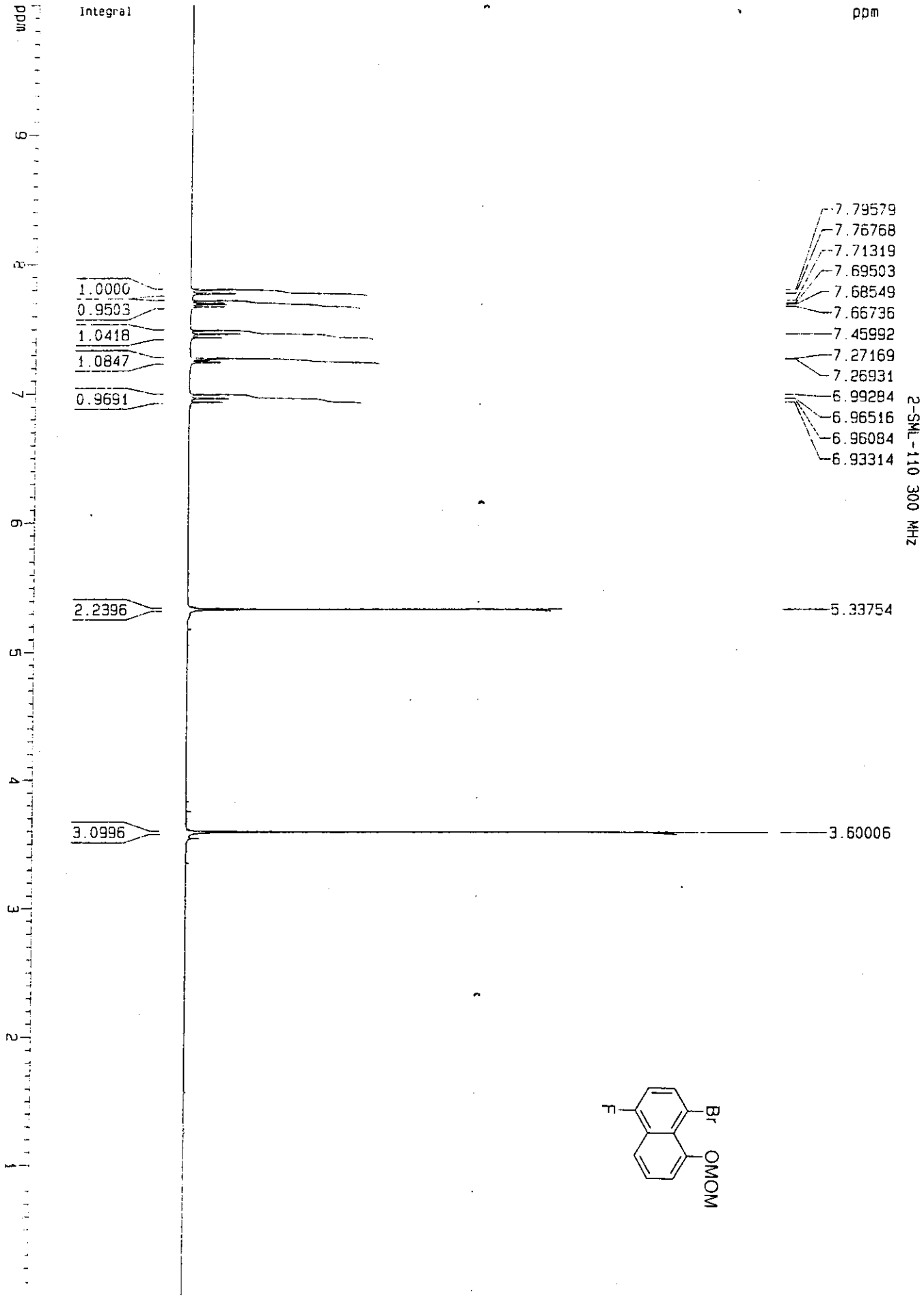

ppm
ppm
195.850
160.995
157.726
134.476
134.379
134.262
134.022
132.450
120.111
119.801
116.318
116.273
77.745
77.525
77.321
76.899
40.070
39.786
23.072
23.014
22.062
21.828
Br
F
O
2-SML-81 75 MHz

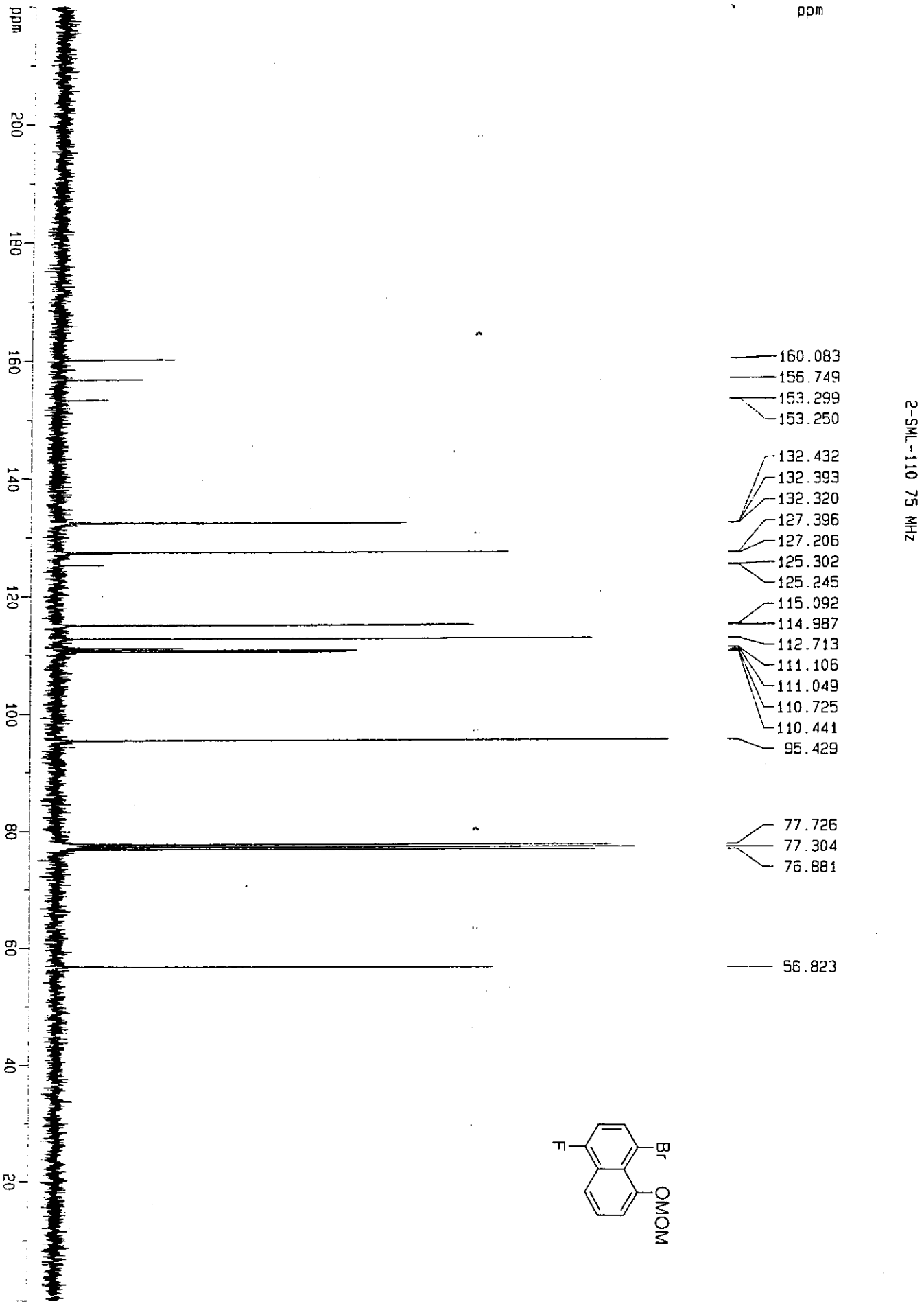

Integral
ppm
1.0000
1.0258
2.0772
0.9578
1.0202
8.07202
7.71132
7.70820
7.70483
7.68036
7.52672
7.50921
7.49910
7.48232
7.45741
7.43029
7.25990
7.13979
7.11388
6.95933
6.93160
6.92742
6.89971
2-SML-85 300 MHz

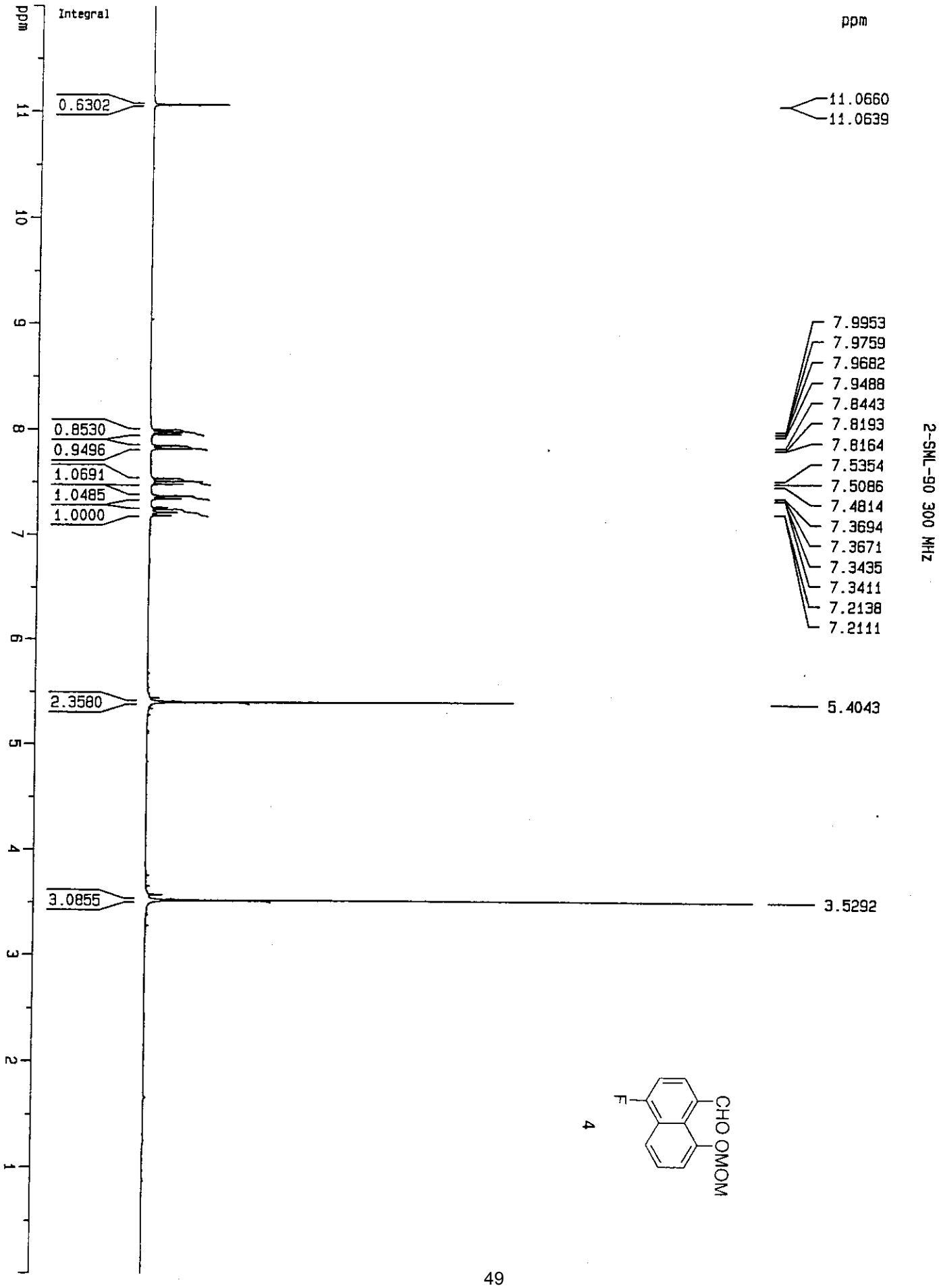
F
Br
OH

3


ppm
200
180
160
140
120
100
80
60
40
20
160.660
157.309
152.870
152.826
130.715
130.598
128.286
127.382
127.153
121.770
121.714
114.661
113.674
113.569
110.246
109.953
109.571
109.516
77.675
77.252
76.830
2-SML-85 75 MHz
F
Br
OH
3


Integral
ppm
1.0000
0.9503
1.0418
1.0847
0.9691
2.2396
3.0996
7.79579
7.76768
7.71319
7.69503
7.68549
7.66736
7.45992
7.27169
7.26931
6.99284
6.96516
6.96084
6.93314
5.33754
3.60006
2-SML-110 300 MHz
F
Br
OMOM


ppm
200
180
160
140
120
100
80
60
40
20
160.083
156.749
153.299
153.250
132.432
132.393
132.320
127.396
127.206
125.302
125.245
115.092
114.987
112.713
111.106
111.049
110.725
110.441
95.429
77.726
77.304
76.881
56.823
2-SML-110 75 MHz
F
Br
OMOM


ppm
Integral
0.6302
0.8530
0.9496
1.0691
1.0485
1.0000
2.3580
3.0855
ppm
11.0660
11.0639
7.9953
7.9759
7.9682
7.9488
7.8443
7.8193
7.8164
7.5354
7.5086
7.4814
7.3694
7.3671
7.3435
7.3411
7.2138
7.2111
5.4043
3.5292
2-SNL-90 300 MHZ
F
CHO
OMOM
4

2-SML-90 75 MHz

ppm

194.138

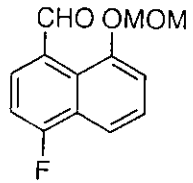
163.639
160.201
153.930

131.373
128.834
128.702
127.387
125.779
125.559
115.178
115.083
111.810
110.159
109.881

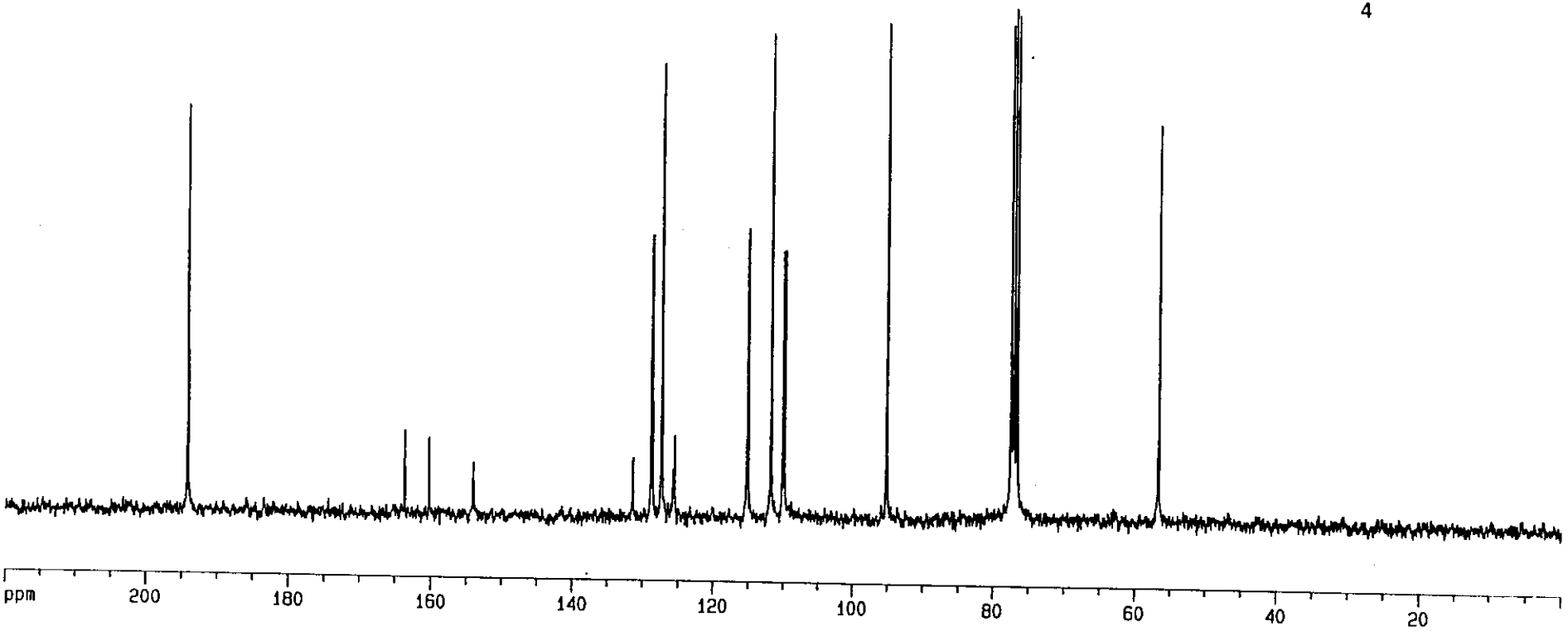
95.214

77.676
77.252
76.829

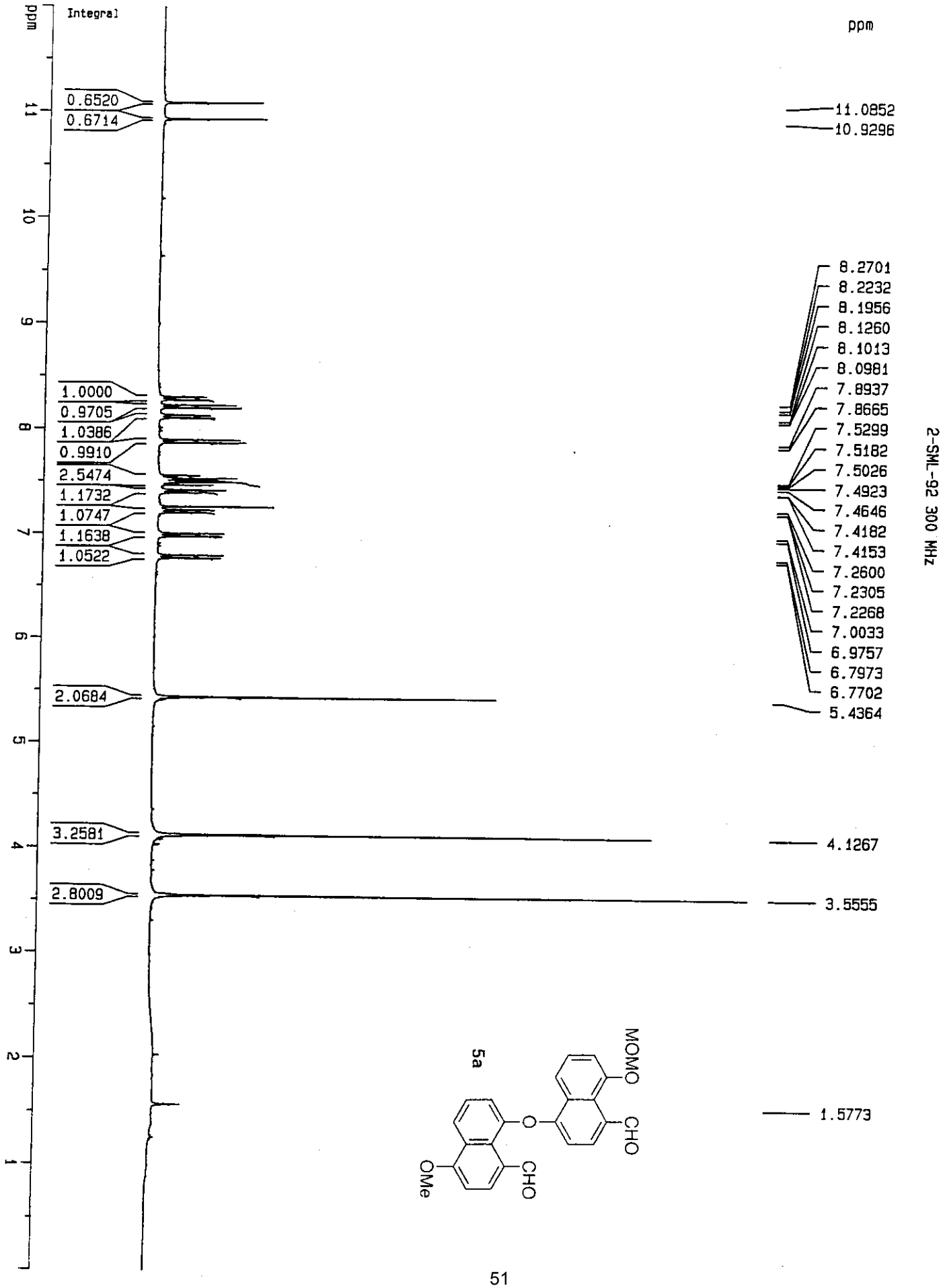
56.840

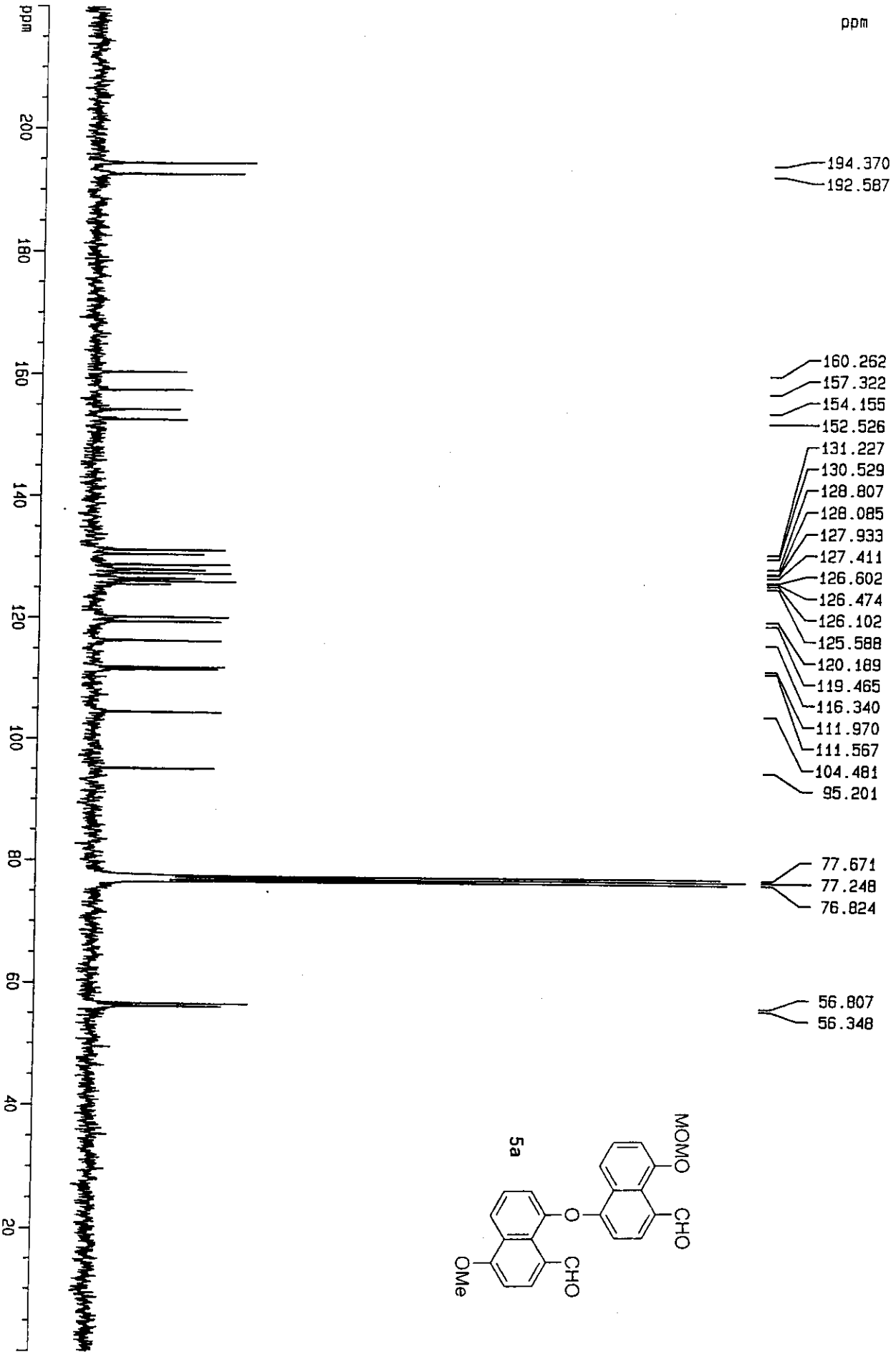


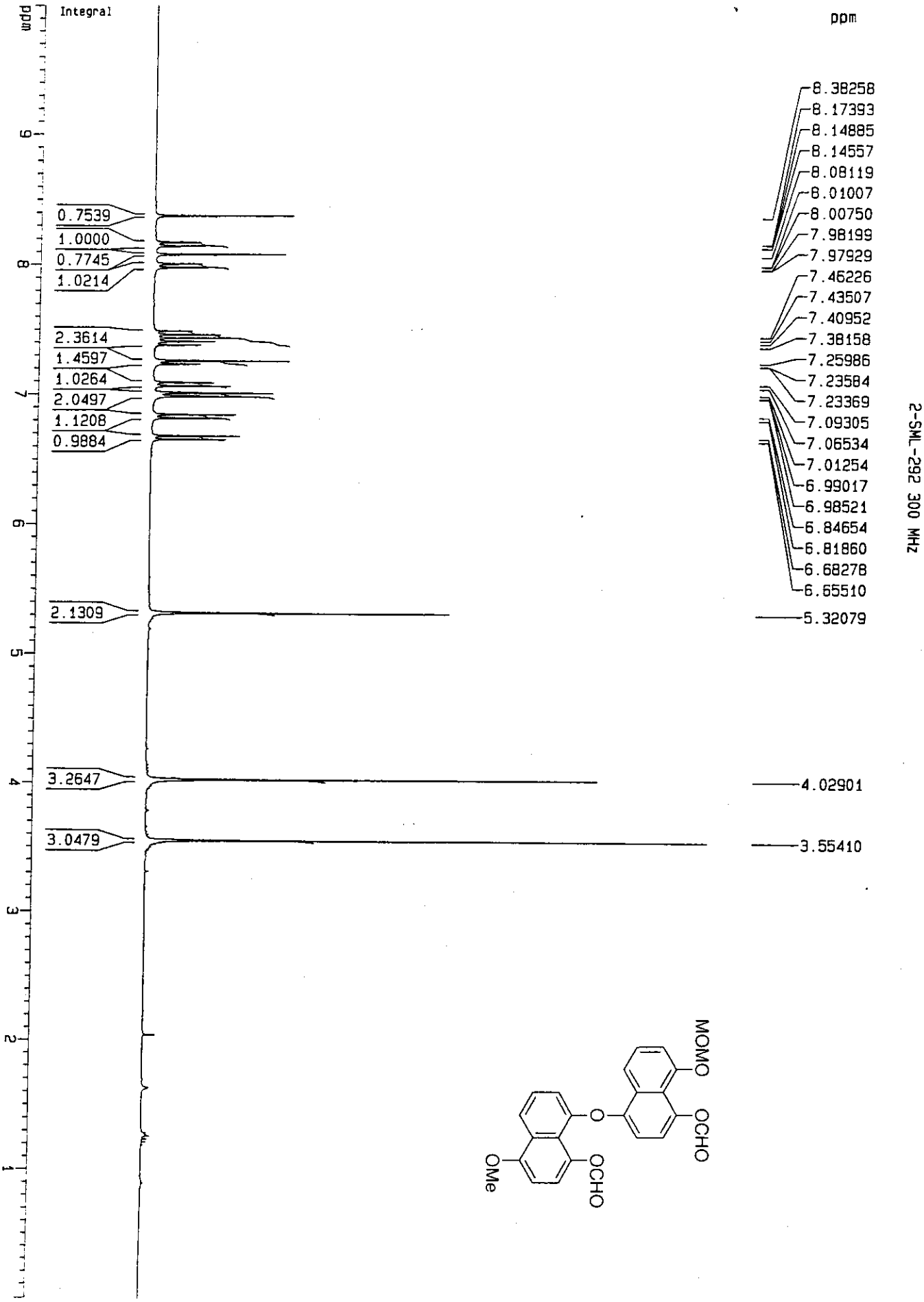
4

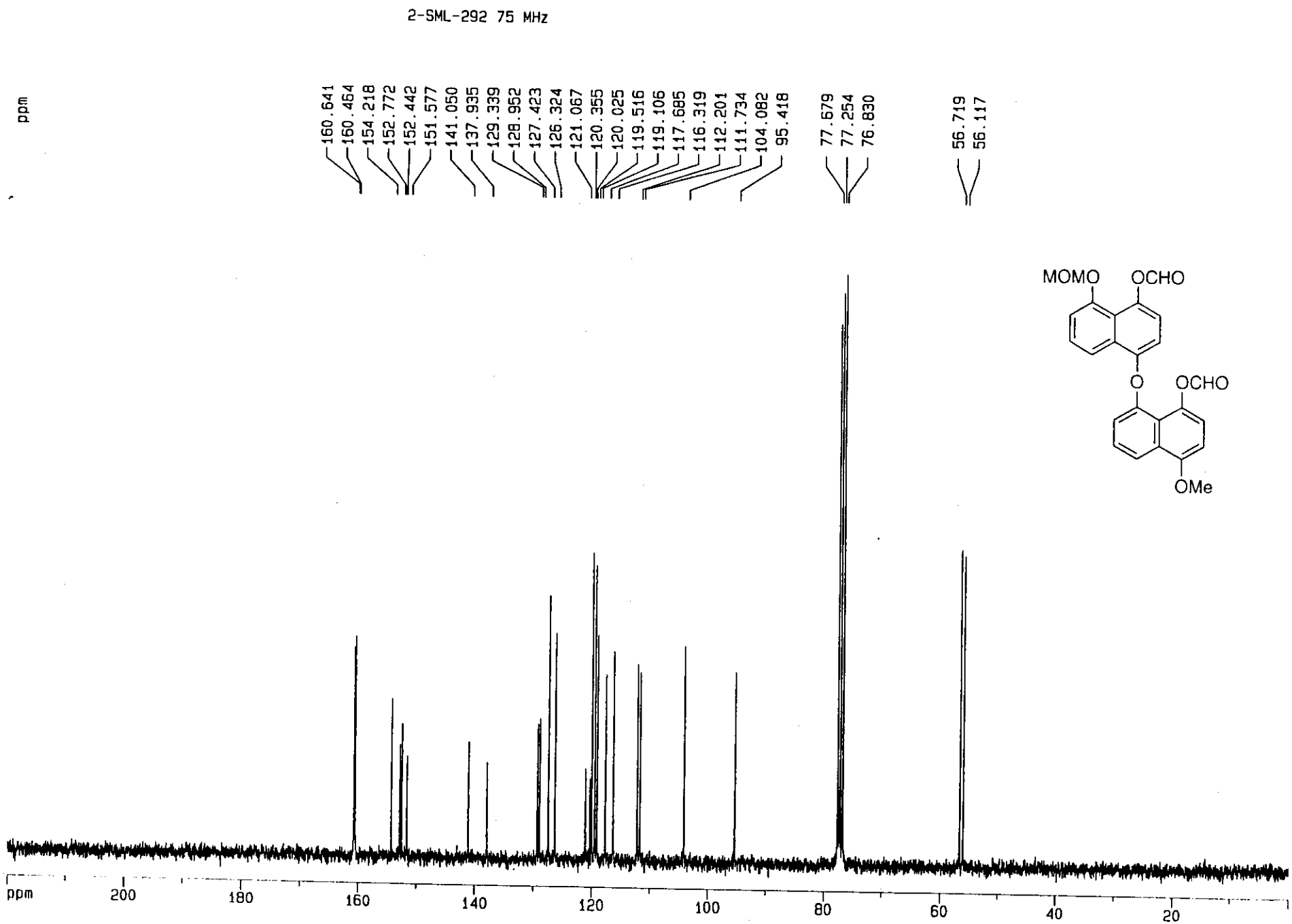


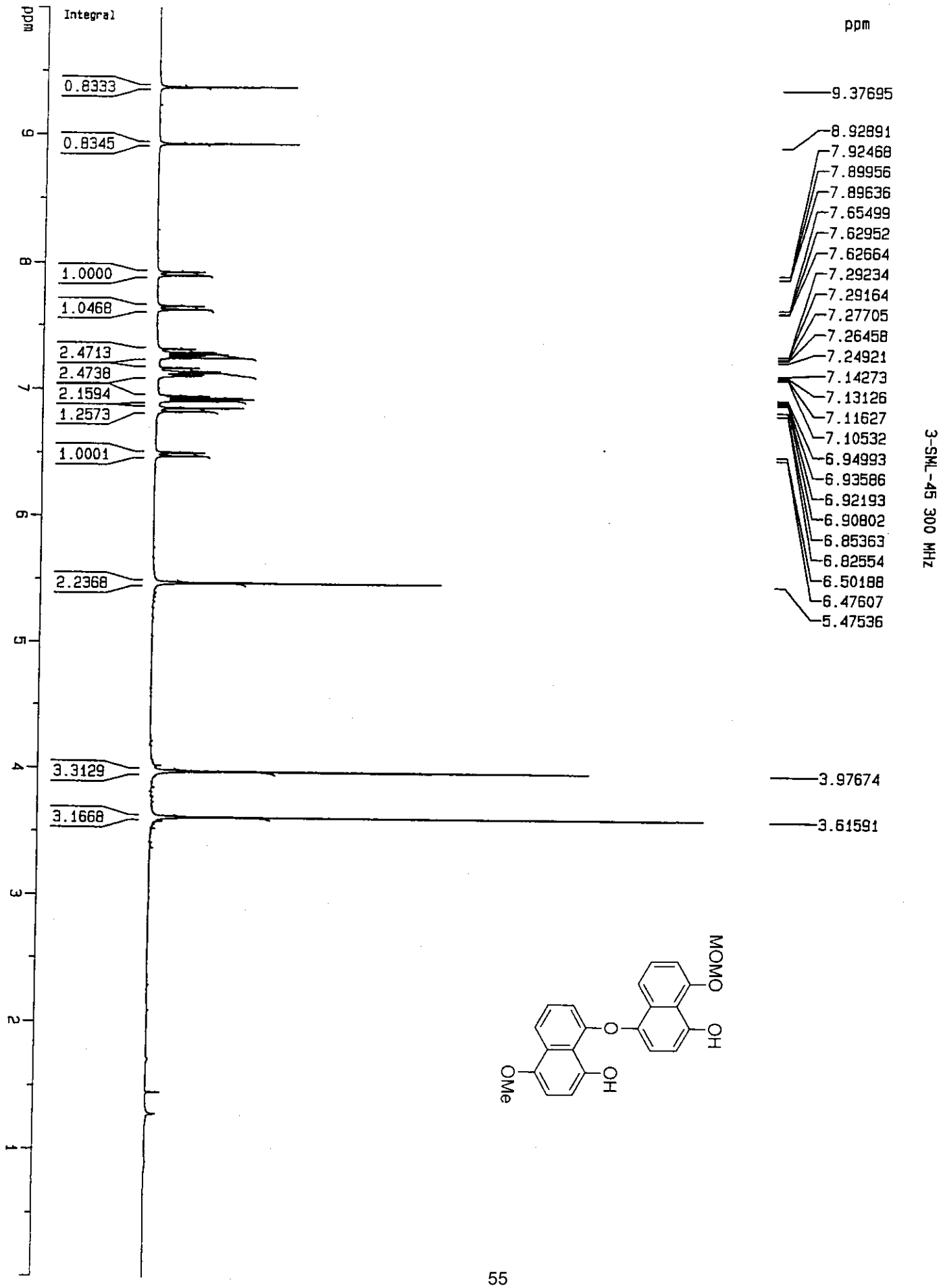
50

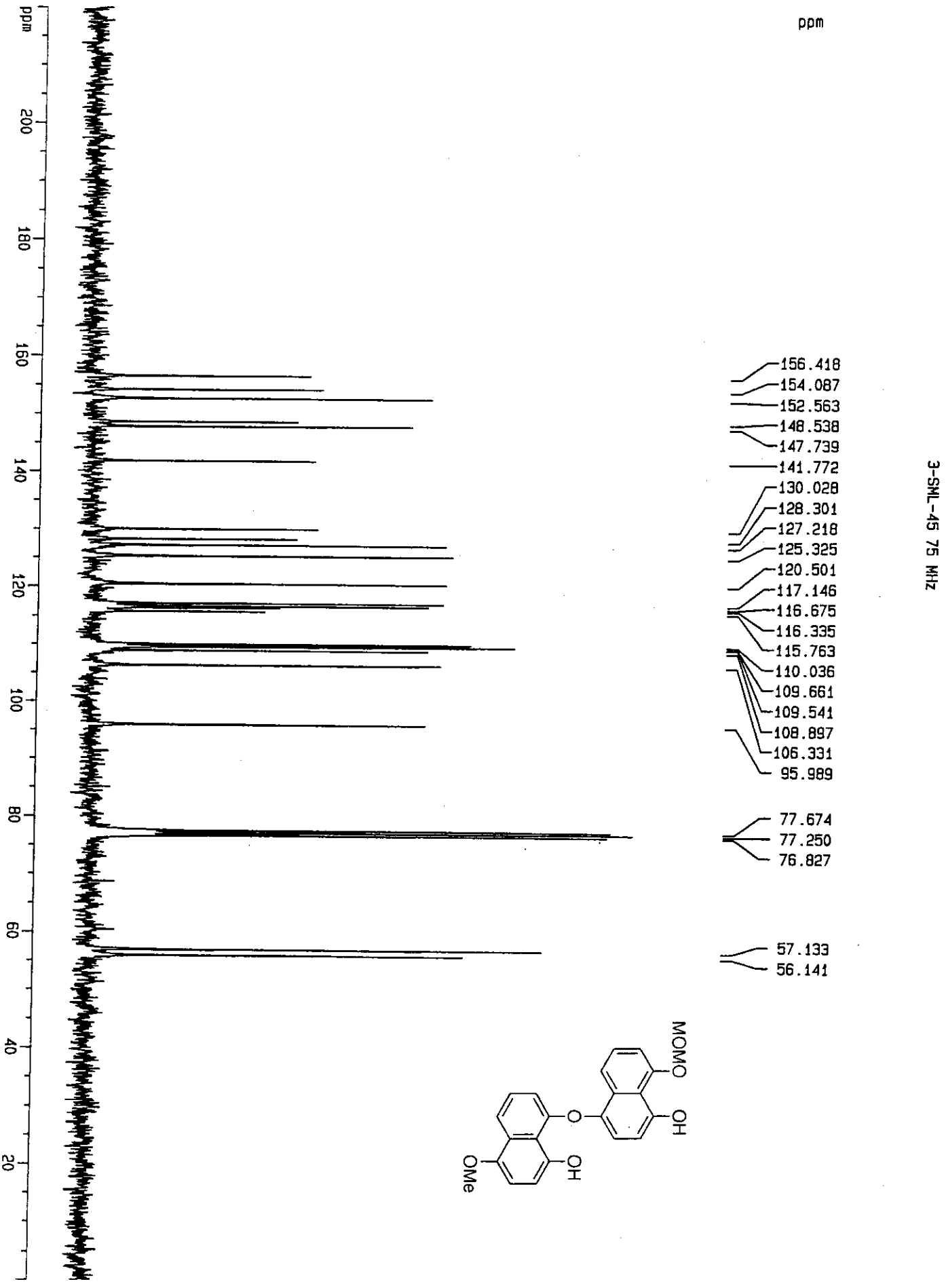

ppm
Integral
0.6520
0.6714
1.0000
0.9705
1.0386
0.9910
2.5474
1.1732
1.0747
1.1638
1.0522
2.0684
3.2581
2.8009
ppm
11.0852
10.9296
8.2701
8.2232
8.1956
8.1260
8.1013
8.0981
7.8937
7.8665
7.5299
7.5182
7.5026
7.4923
7.4646
7.4182
7.4153
7.2600
7.2305
7.2268
7.0033
6.9757
6.7973
6.7702
5.4364
4.1267
3.5555
1.5773
2-SML-92 300 MHZ
5a
MOMO
CHO
O
OMe
CHO

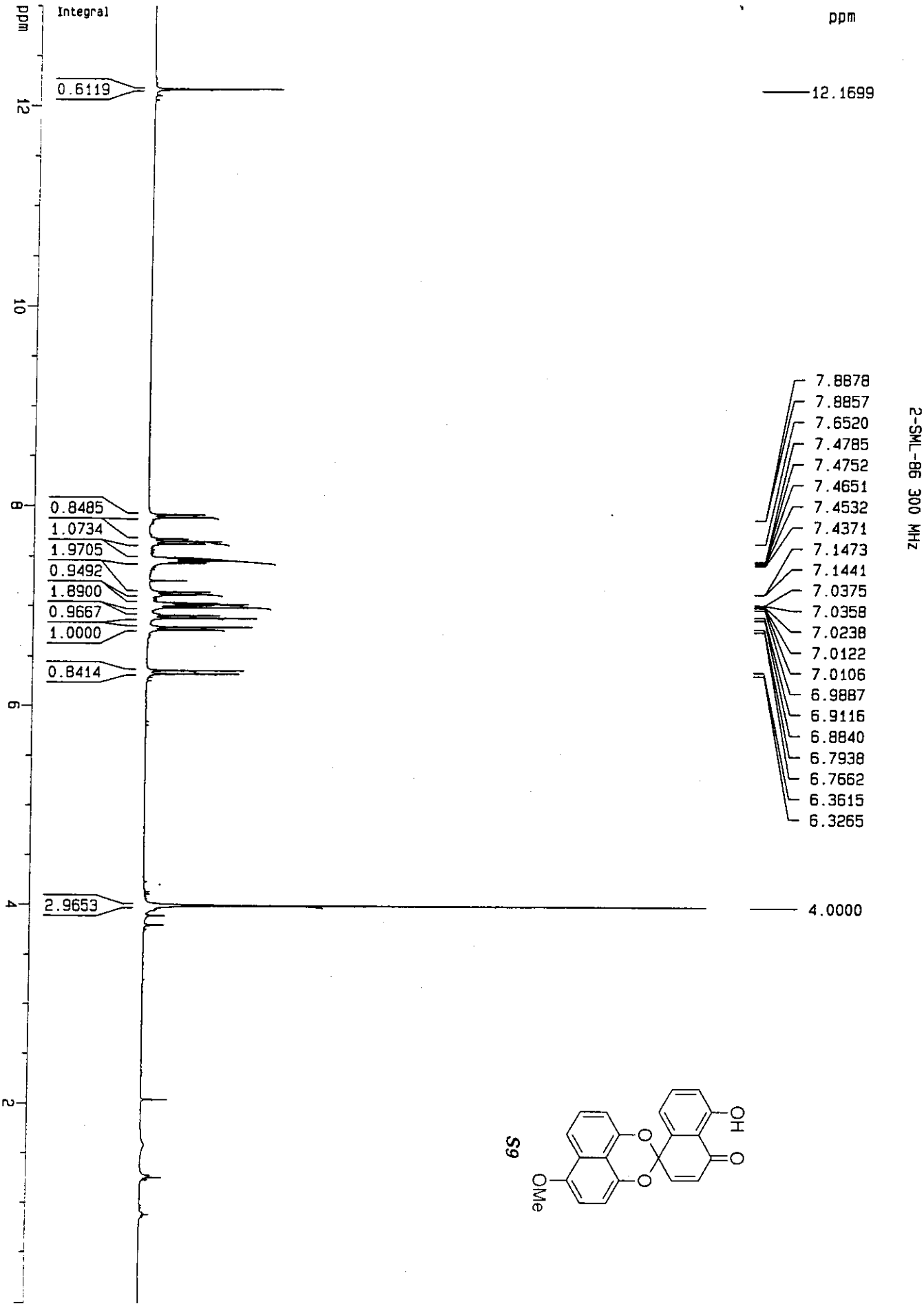

ppm
200
180
160
140
120
100
80
60
40
20
ppm
194.370
192.587
160.262
157.322
154.155
152.526
131.227
130.529
128.807
128.085
127.933
127.411
126.602
126.474
126.102
125.588
120.189
119.465
116.340
111.970
111.567
104.481
95.201
77.671
77.248
76.824
56.807
56.348
2-SML-92 75 MHz
5a
MOMO
CHO
O
CHO
OMe


ppm
Integral
0.7539
1.0000
0.7745
1.0214
2.3614
1.4597
1.0264
2.0497
1.1208
0.9884
2.1309
3.2647
3.0479
ppm
8.38258
8.17393
8.14885
8.14557
8.08119
8.01007
8.00750
7.98199
7.97929
7.46226
7.43507
7.40952
7.38158
7.25986
7.23584
7.23369
7.09305
7.06534
7.01254
6.99017
6.98521
6.84654
6.81860
6.68278
6.65510
5.32079
4.02901
3.55410
2-SML-292 300 MHz
MOMO
OCHO
OMe
OCHO

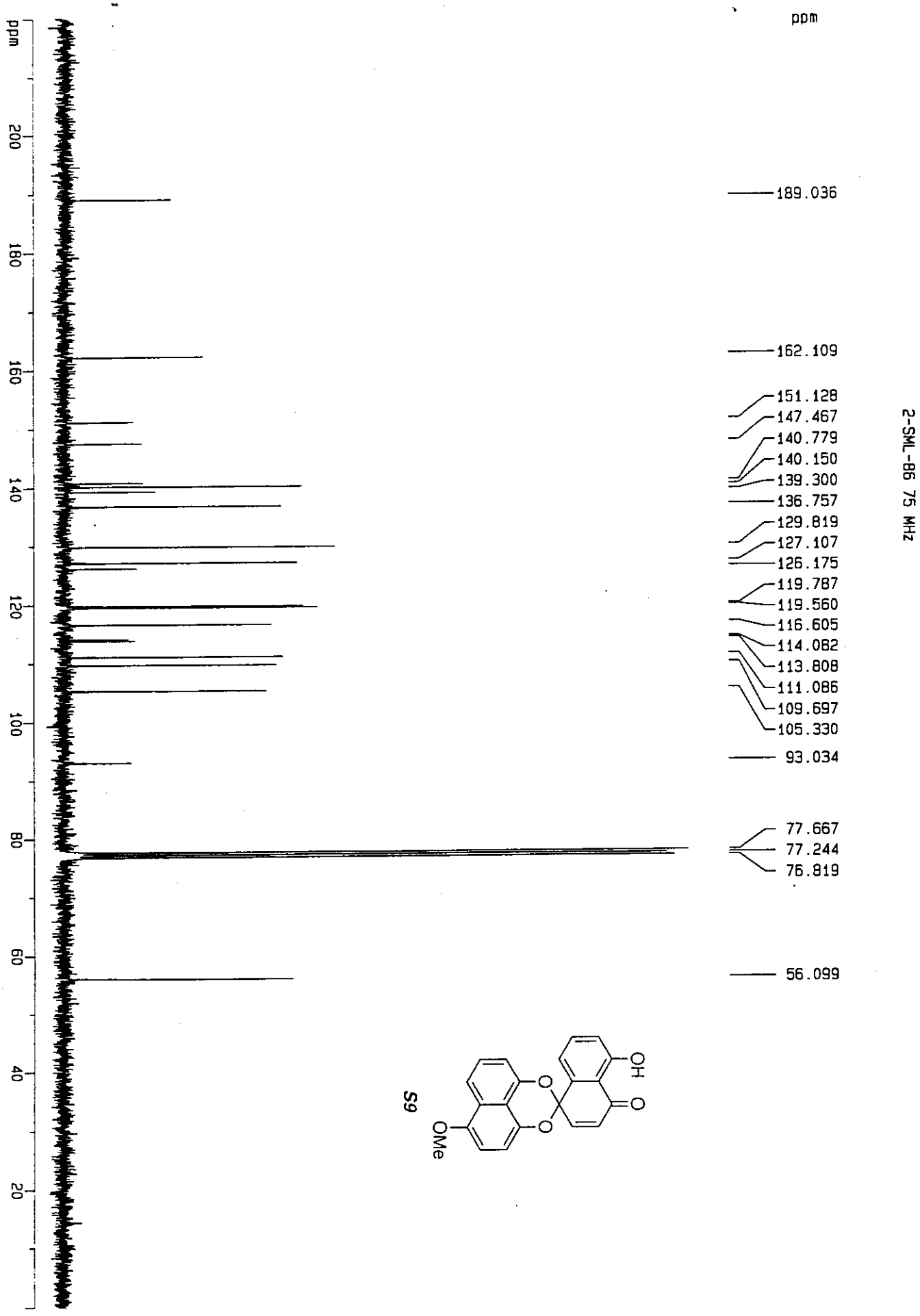

2-SML-292 75 MHz
ppm
160.641
160.464
154.218
152.772
152.442
151.577
141.050
137.935
129.339
128.952
127.423
126.324
121.067
120.355
120.025
119.516
119.106
117.685
116.319
112.201
111.734
104.082
95.418
77.679
77.254
76.830
56.719
56.117
MOMO
OCHO
O
OCHO
OMe
ppm
200
180
160
140
120
100
80
60
40
20


ppm
Integral
0.8333
0.8345
1.0000
1.0468
2.4713
2.4738
2.1594
1.2573
1.0001
2.2368
3.3129
3.1668
ppm
9.37695
8.92891
7.92468
7.89956
7.89636
7.65499
7.62952
7.62664
7.29234
7.29164
7.27705
7.26458
7.24921
7.14273
7.13126
7.11627
7.10532
6.94993
6.93586
6.92193
6.90802
6.85363
6.82554
6.50188
6.47607
5.47536
3.97674
3.61591
3-SML-45 300 MHz
MOMO
O
HO
OMe
OH

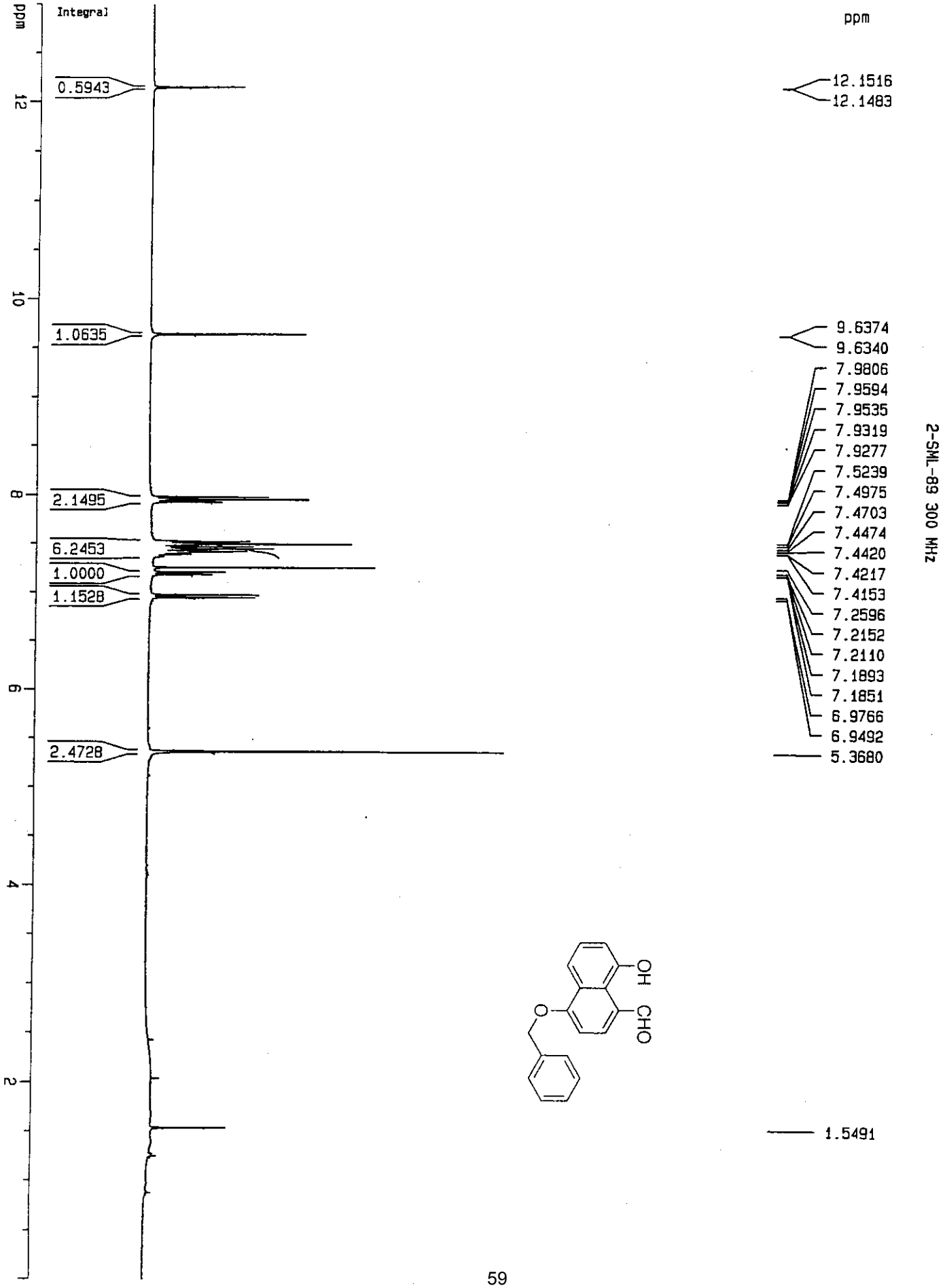

ppm
200
180
160
140
120
100
80
60
40
20
ppm
156.418
154.087
152.563
148.538
147.739
141.772
130.028
128.301
127.218
125.325
120.501
117.146
116.675
116.335
115.763
110.036
109.661
109.541
108.897
106.331
95.989
77.674
77.250
76.827
57.133
56.141
MOMO
OH
O
OH
OMe
3-SML-45 75 MHz

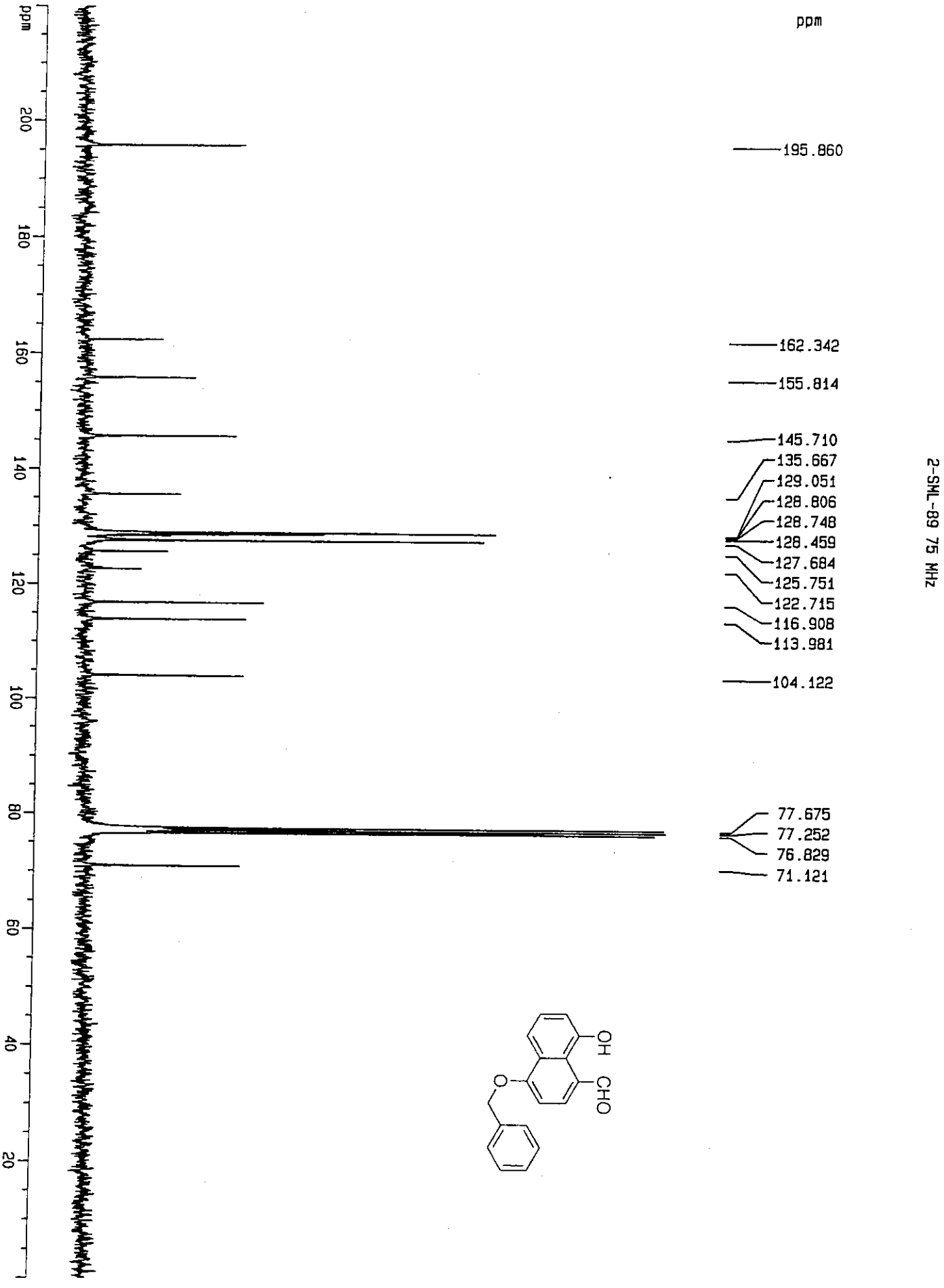

ppm
Integral
0.6119
0.8485
1.0734
1.9705
0.9492
1.8900
0.9667
1.0000
0.8414
2.9653
ppm
12.1699
7.8878
7.8857
7.6520
7.4785
7.4752
7.4651
7.4532
7.4371
7.1473
7.1441
7.0375
7.0358
7.0238
7.0122
7.0106
6.9887
6.9116
6.8840
6.7938
6.7662
6.3615
6.3265
4.0000
2-SML-86 300 MHz
OH
O
O
O
OMe

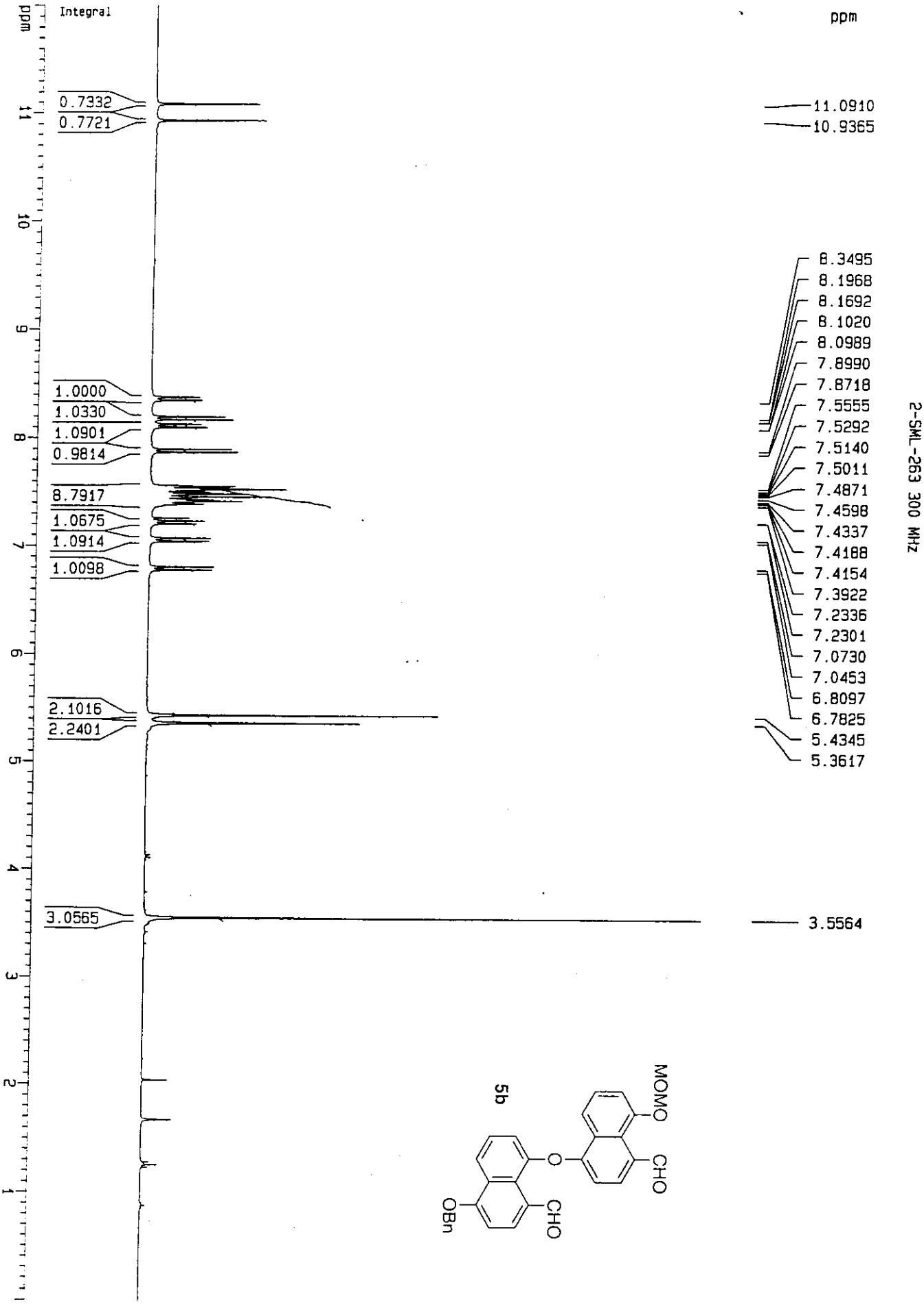
S9

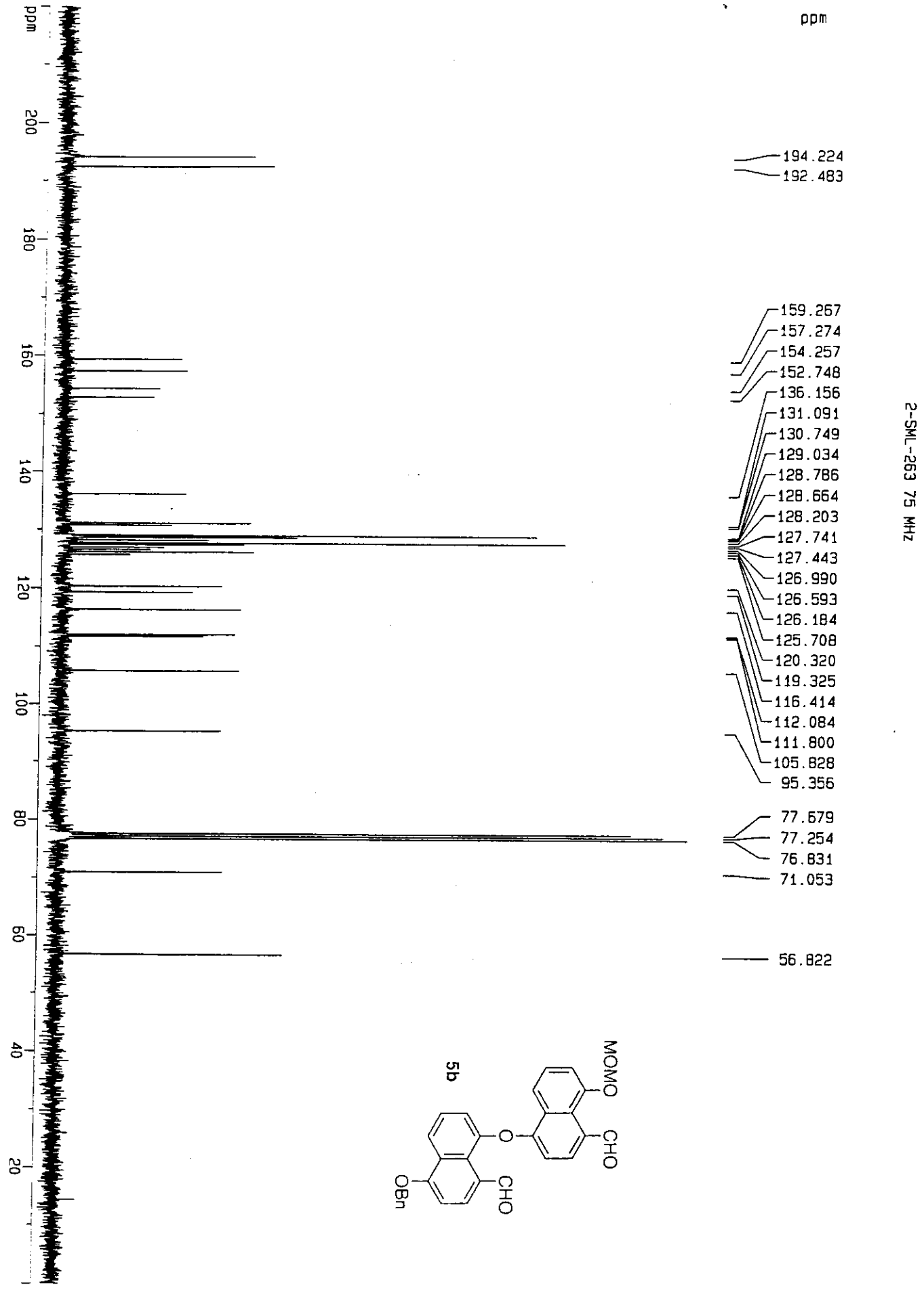

ppm
189.036
162.109
151.128
147.467
140.779
140.150
139.300
136.757
129.819
127.107
126.175
119.787
119.560
116.605
114.082
113.808
111.086
109.697
105.330
93.034
77.667
77.244
76.819
56.099
2-SML-86 75 MHz
OH
O
O
O
OMe

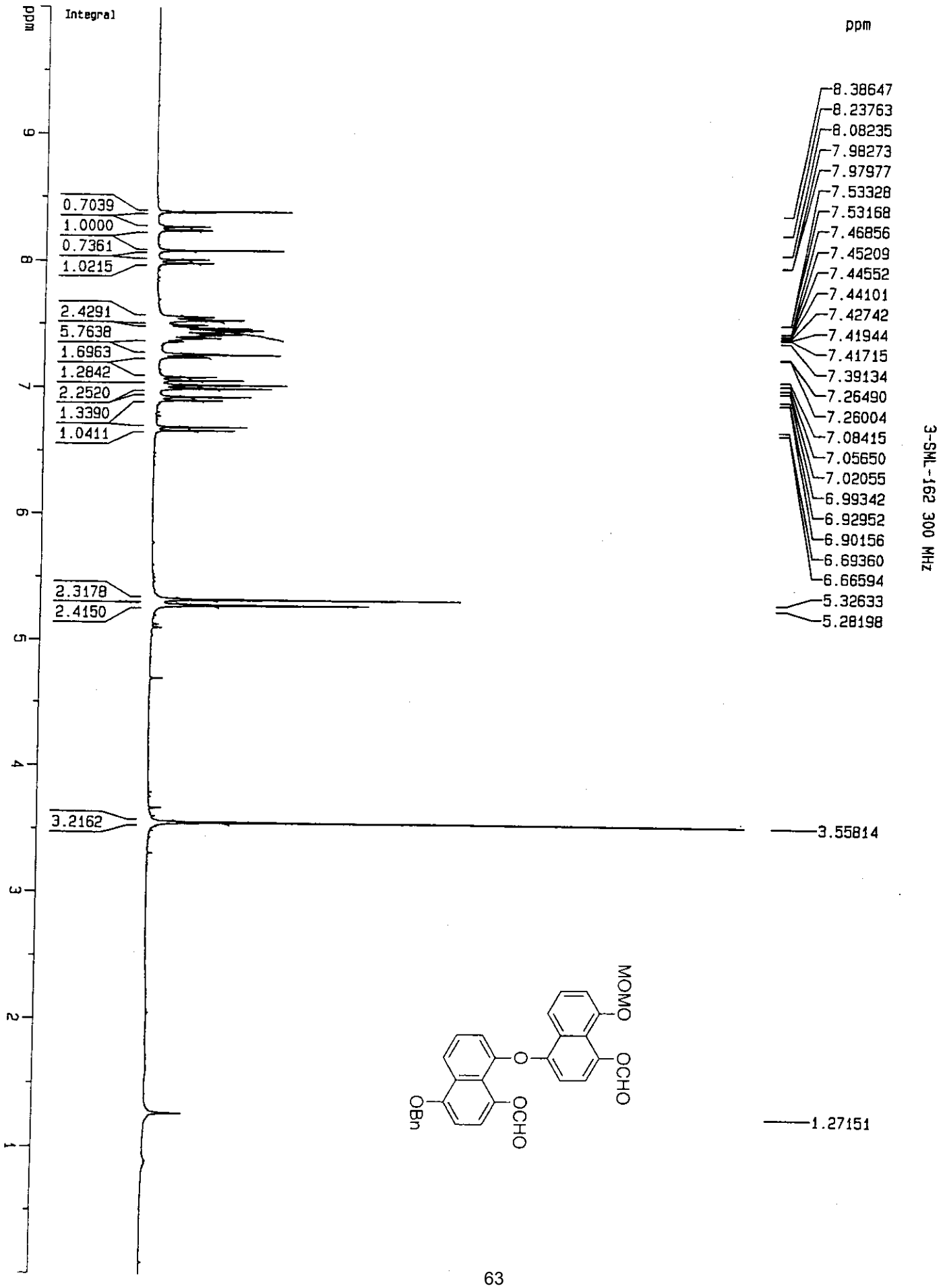
S9

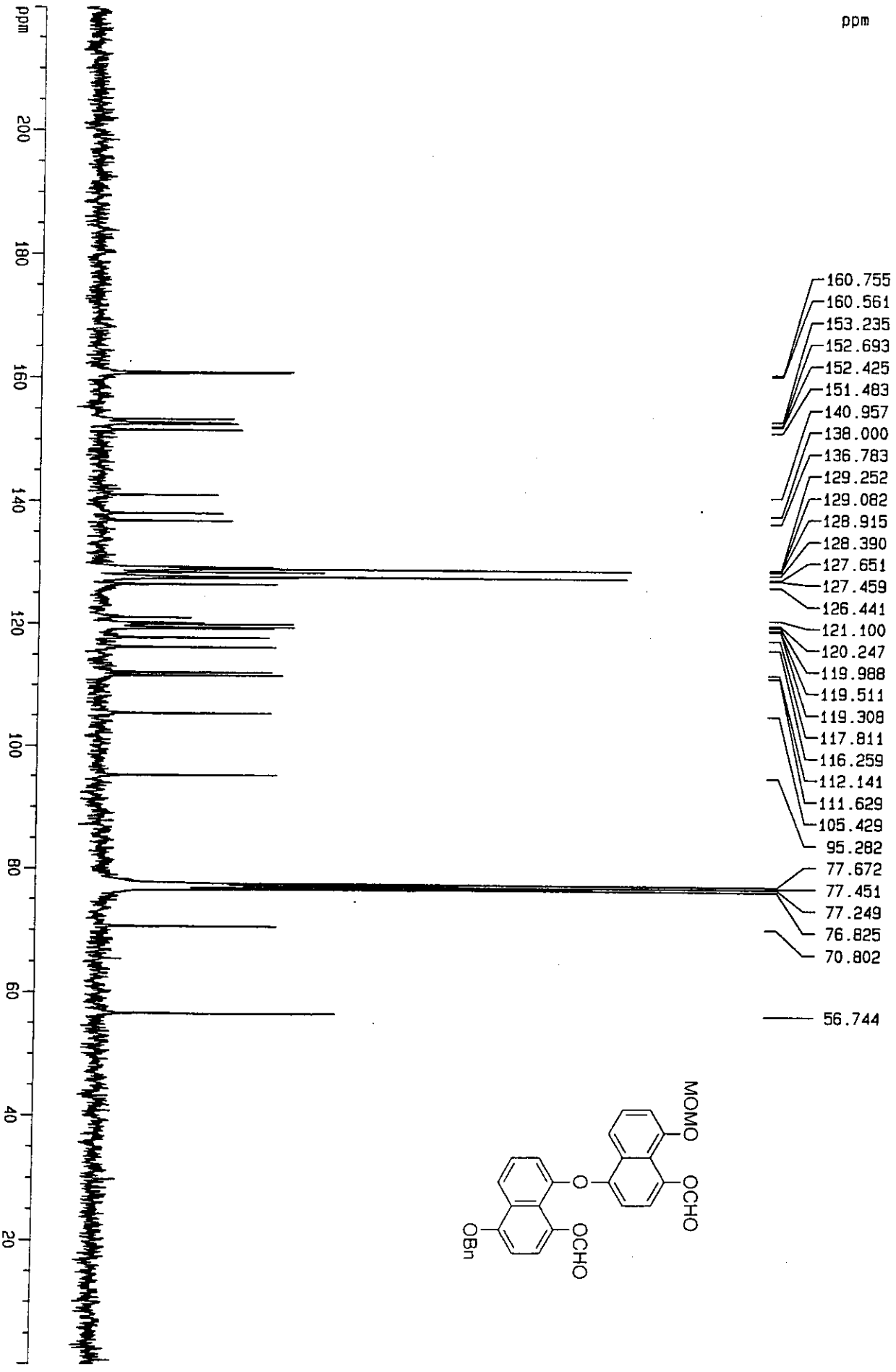

Integral
ppm
0.5943
1.0635
2.1495
6.2453
1.0000
1.1528
2.4728
12.1516
12.1483
9.6374
9.6340
7.9806
7.9594
7.9535
7.9319
7.9277
7.5239
7.4975
7.4703
7.4474
7.4420
7.4217
7.4153
7.2596
7.2152
7.2110
7.1893
7.1851
6.9766
6.9492
5.3680
1.5491
OH
CHO
O
2-SML-89 300 MHz


ppm
195.860
162.342
155.814
145.710
135.667
129.051
128.806
128.748
128.459
127.684
125.751
122.715
116.908
113.981
104.122
77.675
77.252
76.829
71.121
2-SWL-89 75 MHz
OH
CHO
O

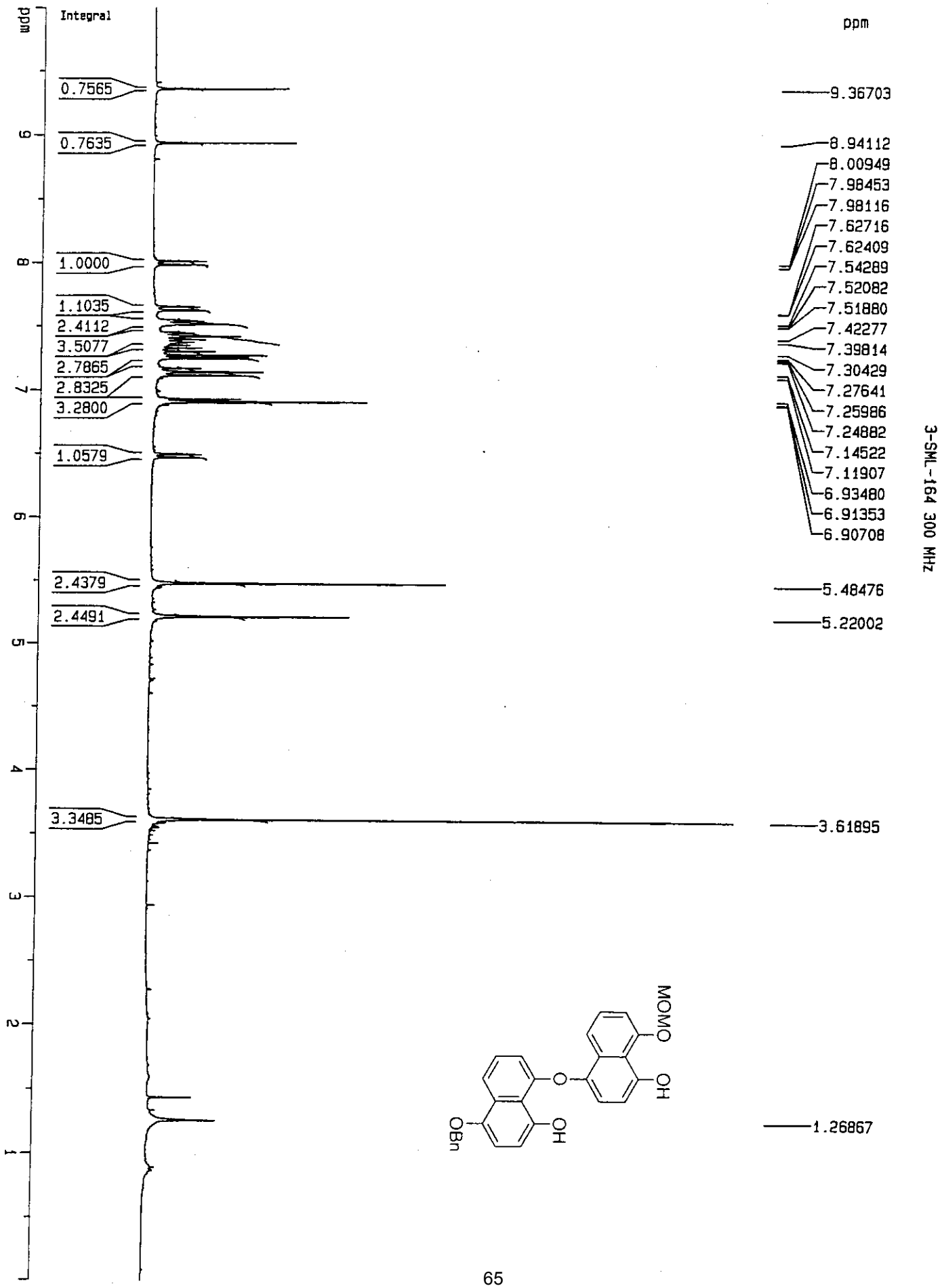

Integral
ppm
0.7332
0.7721
1.0000
1.0330
1.0901
0.9814
8.7917
1.0675
1.0914
1.0098
2.1016
2.2401
3.0565
11.0910
10.9365
8.3495
8.1968
8.1692
8.1020
8.0989
7.8990
7.8718
7.5555
7.5292
7.5140
7.5011
7.4871
7.4598
7.4337
7.4188
7.4154
7.3922
7.2336
7.2301
7.0730
7.0453
6.8097
6.7825
5.4345
5.3617
3.5564
2-SML-263 300 MHz
5b
MOMO
CHO
OBn
CHO

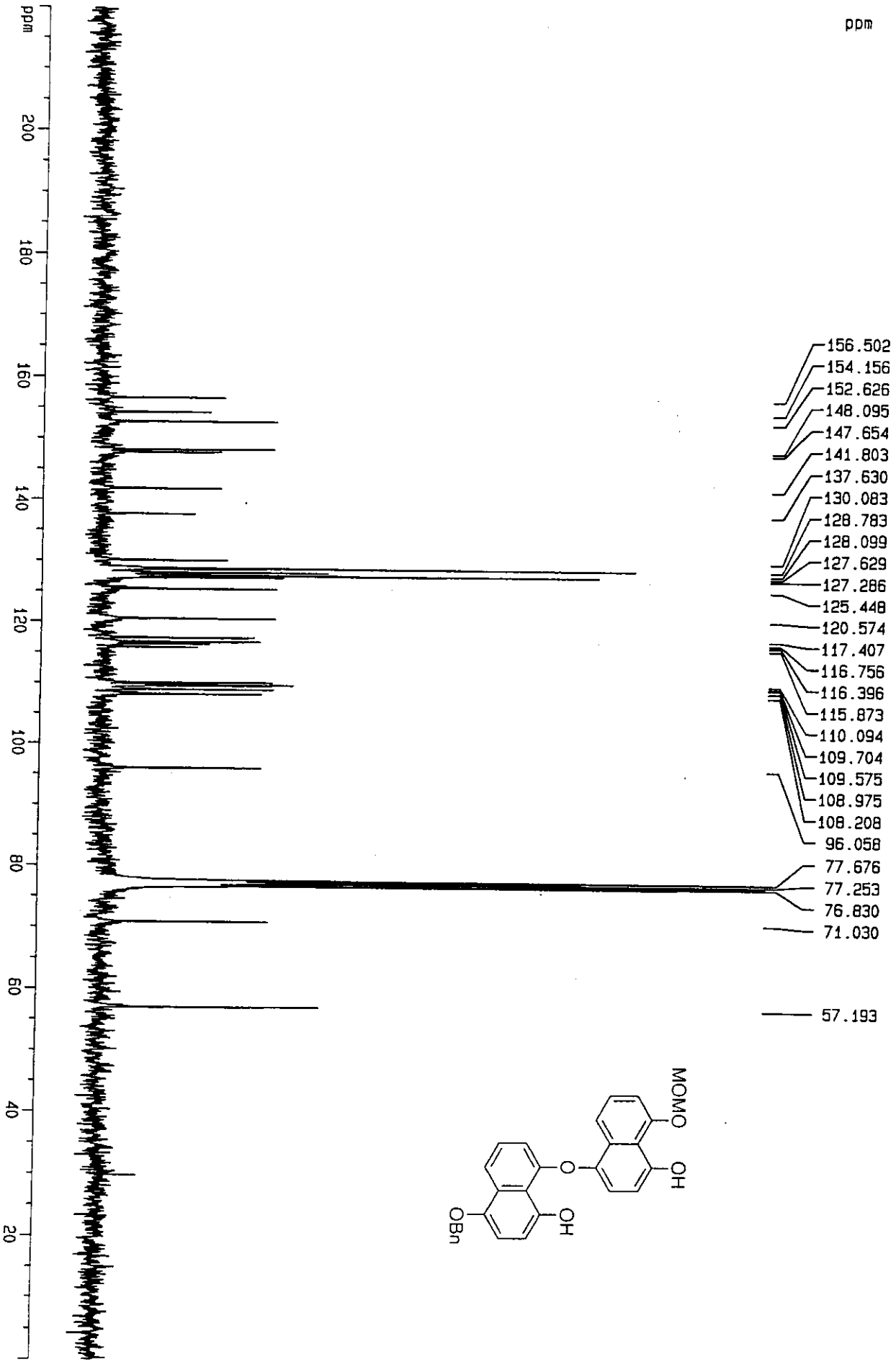

ppm
200
180
160
140
120
100
80
60
40
20
194.224
192.483
159.267
157.274
154.257
152.748
136.156
131.091
130.749
129.034
128.786
128.664
128.203
127.741
127.443
126.990
126.593
126.184
125.708
120.320
119.325
116.414
112.084
111.800
105.828
95.356
77.679
77.254
76.831
71.053
56.822
2-SML-263 75 MHz
5b
MOMO
CHO
O
OBn
CHO


Integral
ppm
0.7039
1.0000
0.7361
1.0215
2.4291
5.7638
1.6963
1.2842
2.2520
1.3390
1.0411
2.3178
2.4150
3.2162
8.38647
8.23763
8.08235
7.98273
7.97977
7.53328
7.53168
7.46856
7.45209
7.44552
7.44101
7.42742
7.41944
7.41715
7.39134
7.26490
7.26004
7.08415
7.05650
7.02055
6.99342
6.92952
6.90156
6.69360
6.66594
5.32633
5.28198
3.55814
1.27151
3-SWL-162 300 MHz
MOMO
OCHO
OBn
OCHO

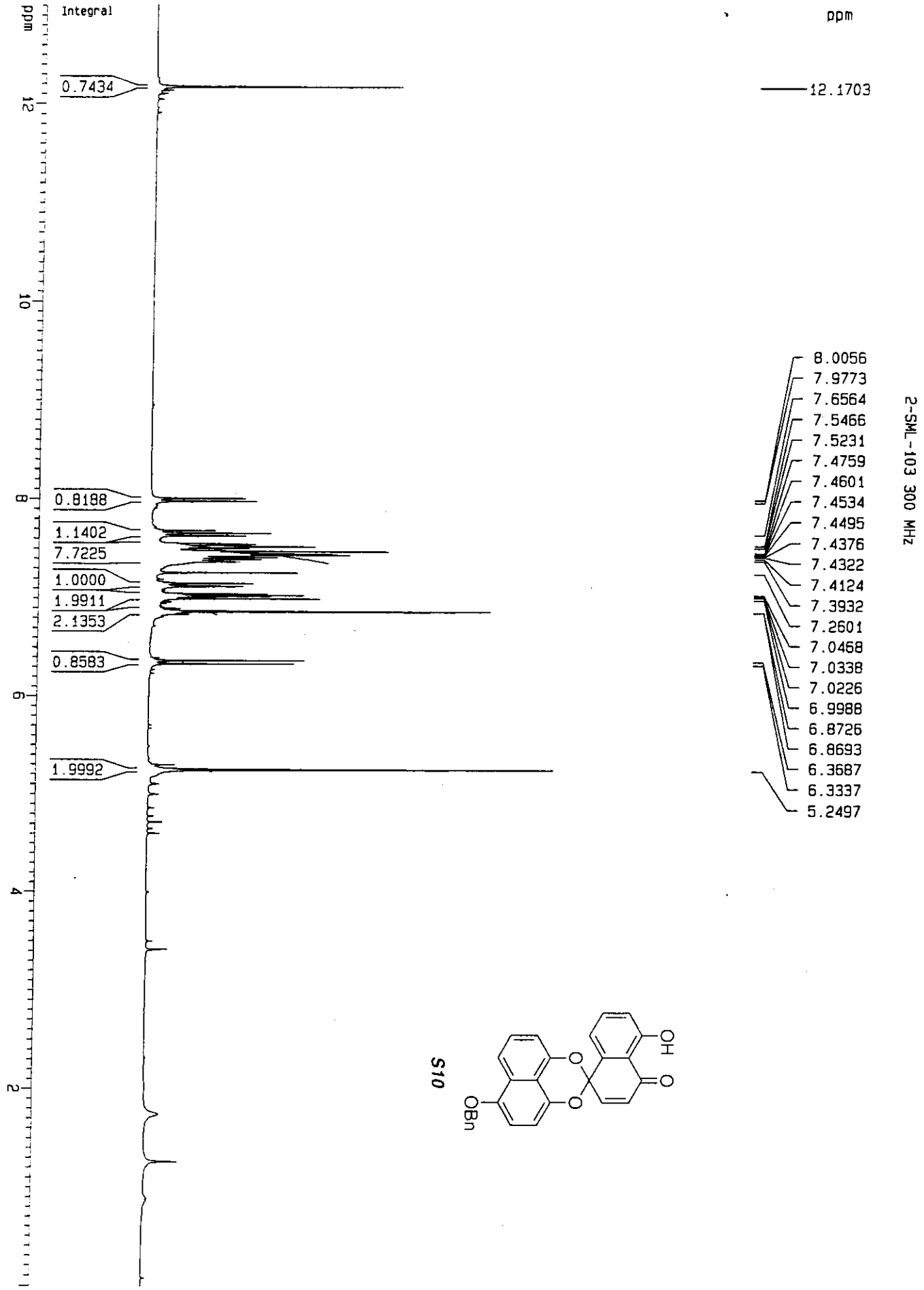


3-SML-162 75 MHz

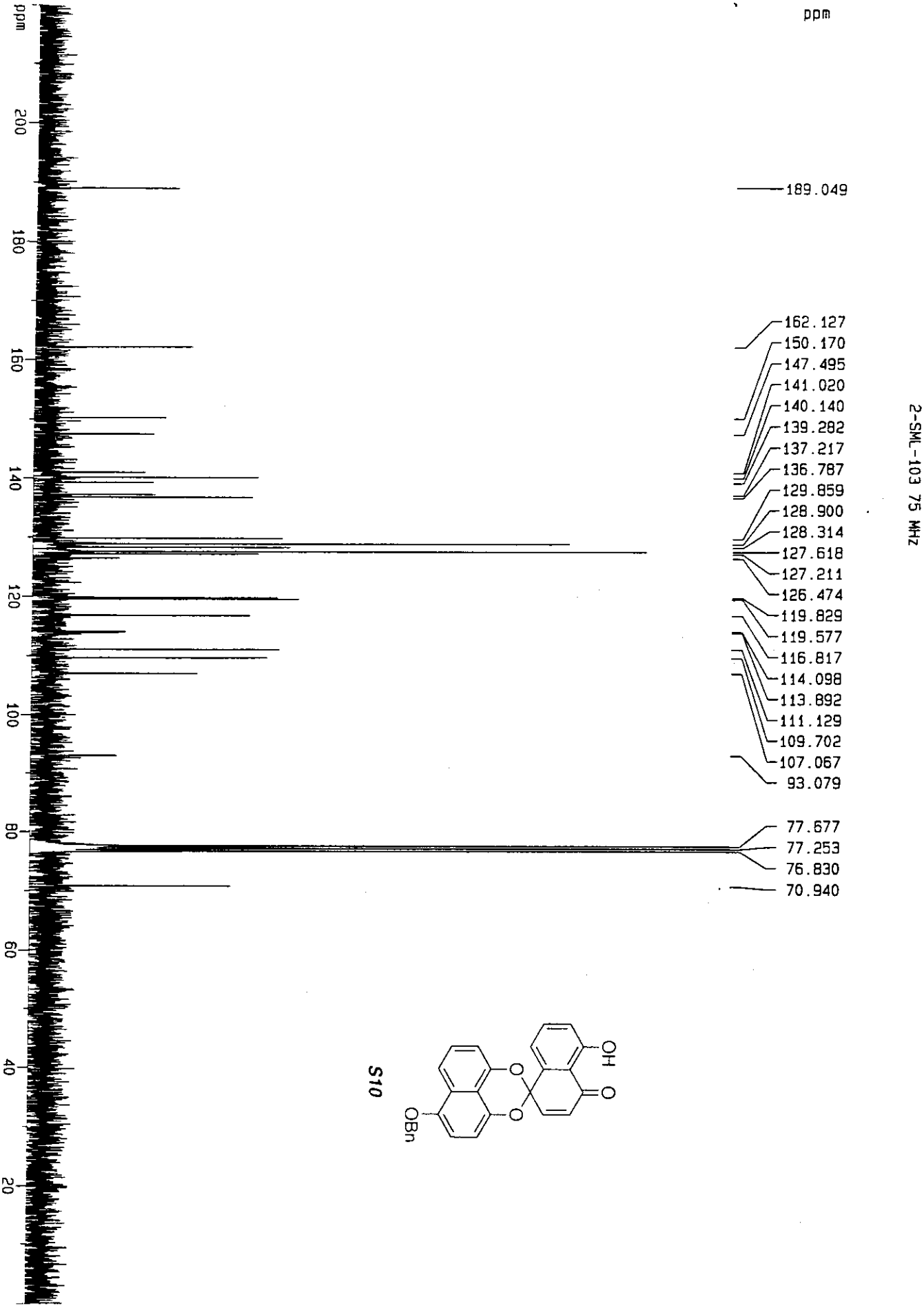

ppm
Integral
0.7565
0.7635
1.0000
1.1035
2.4112
3.5077
2.7865
2.8325
3.2800
1.0579
2.4379
2.4491
3.3485
9.36703
8.94112
8.00949
7.98453
7.98116
7.62716
7.62409
7.54289
7.52082
7.51880
7.42277
7.39814
7.30429
7.27641
7.25986
7.24882
7.14522
7.11907
6.93480
6.91353
6.90708
5.48476
5.22002
3.61895
1.26867
MOMO
OH
O
OBn
OH
3-SML-164 300 MHz



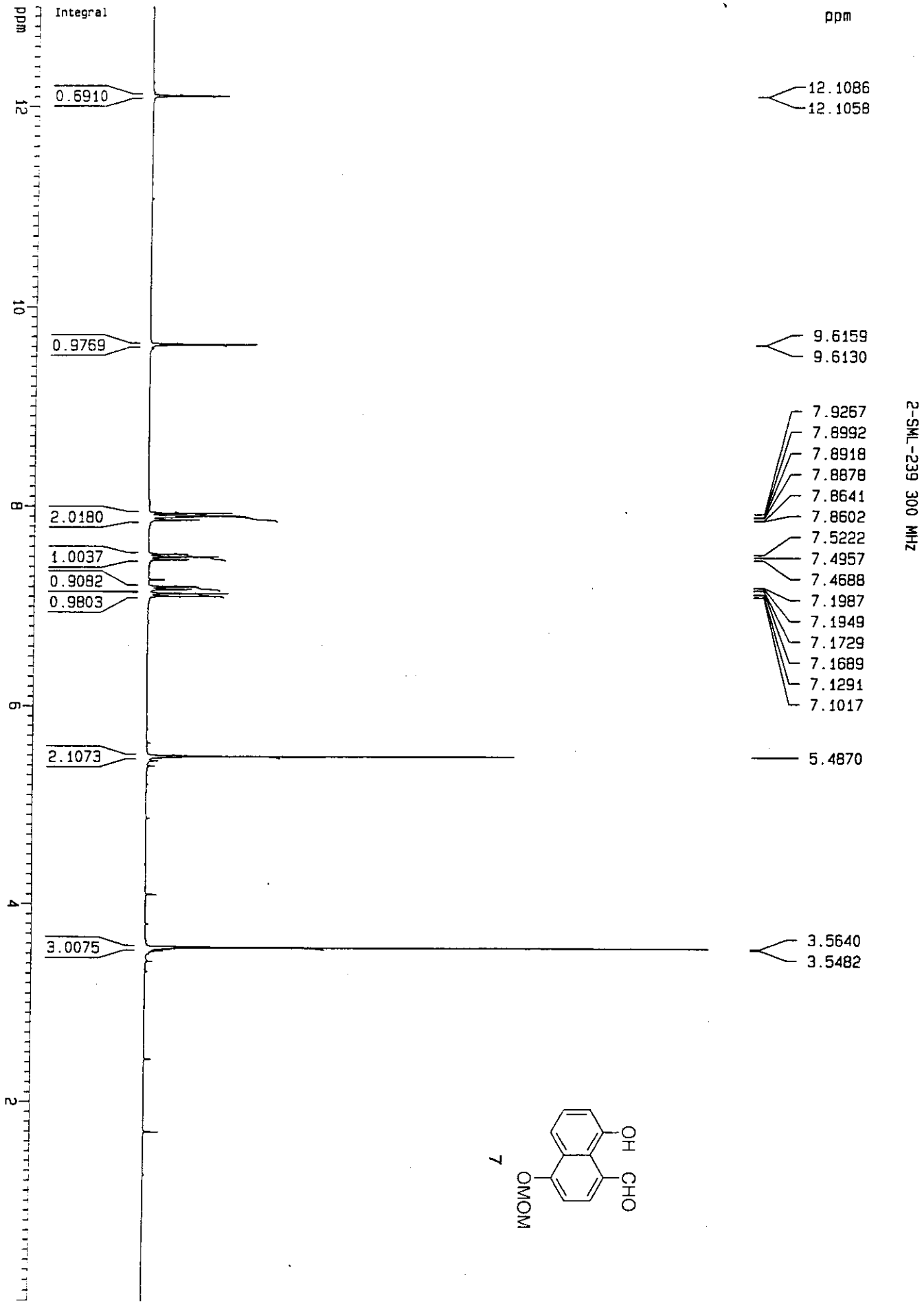
3-SWL-164 75 MHz

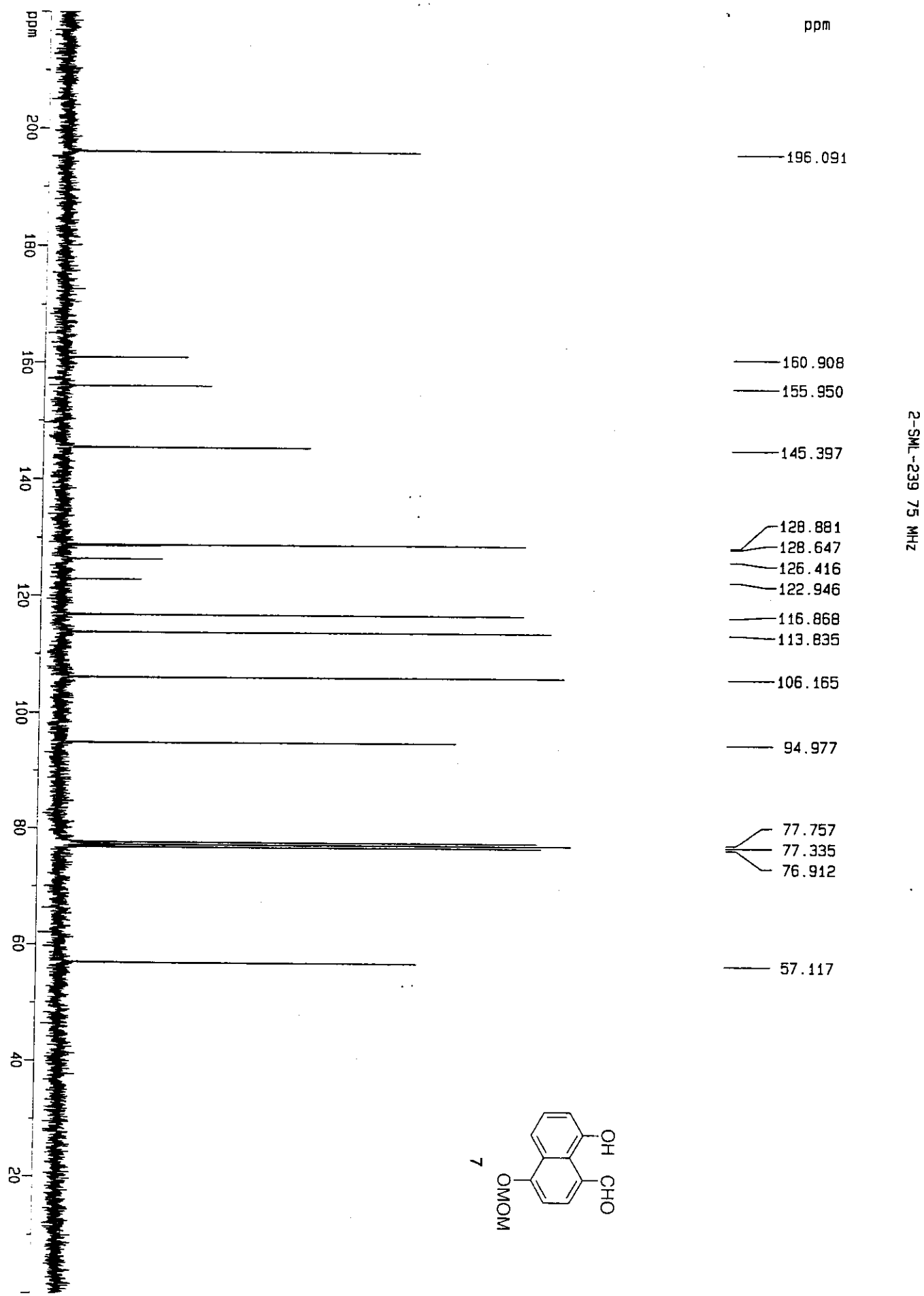

ppm
Integral
0.7434
0.8188
1.1402
7.7225
1.0000
1.9911
2.1353
0.8583
1.9992
ppm
12.1703
8.0056
7.9773
7.6564
7.5466
7.5231
7.4759
7.4601
7.4534
7.4495
7.4376
7.4322
7.4124
7.3932
7.2601
7.0468
7.0338
7.0226
6.9988
6.8726
6.8693
6.3687
6.3337
5.2497
2-SML-103 300 MHz
OH
O
O
O
OBn

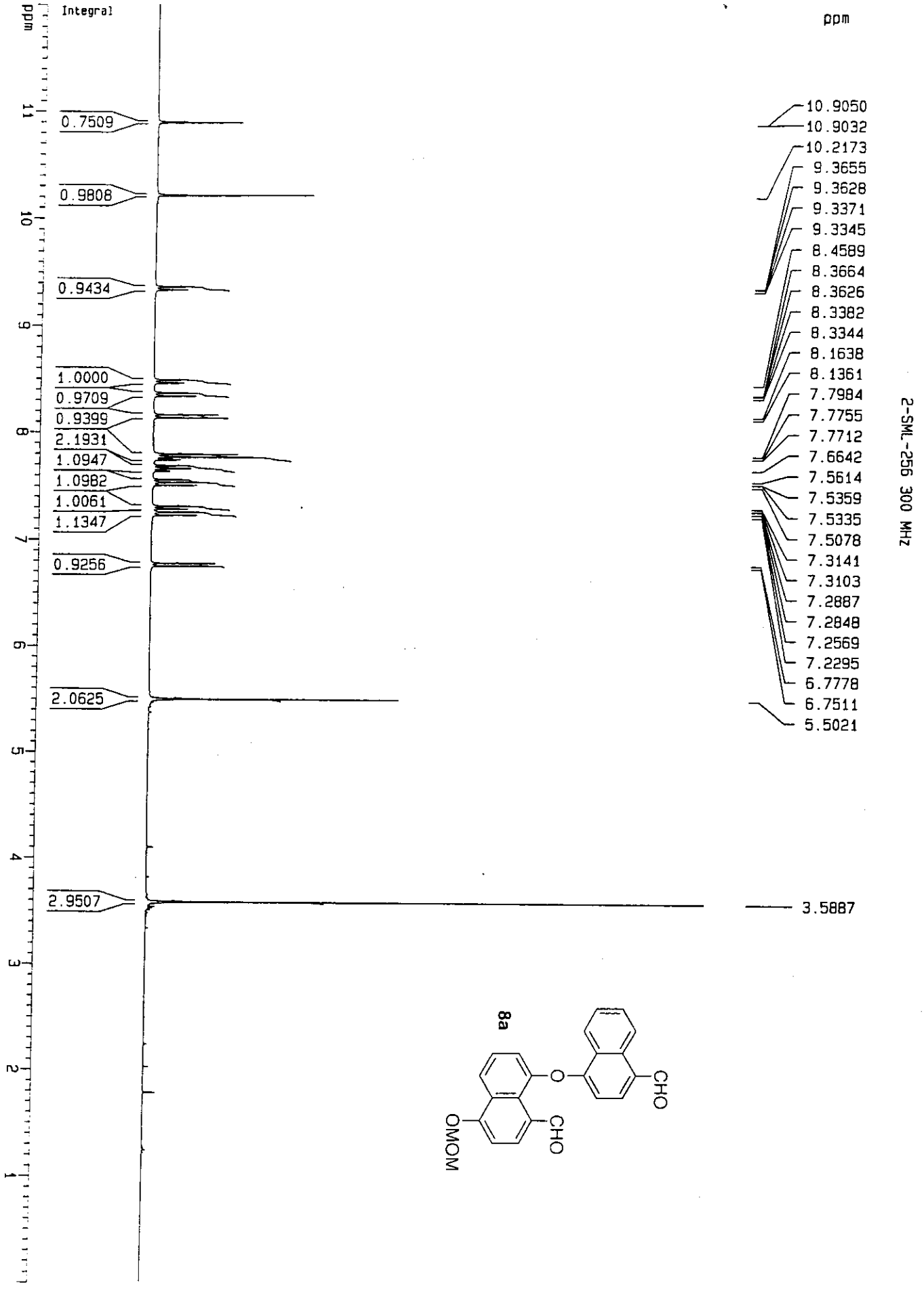
S10

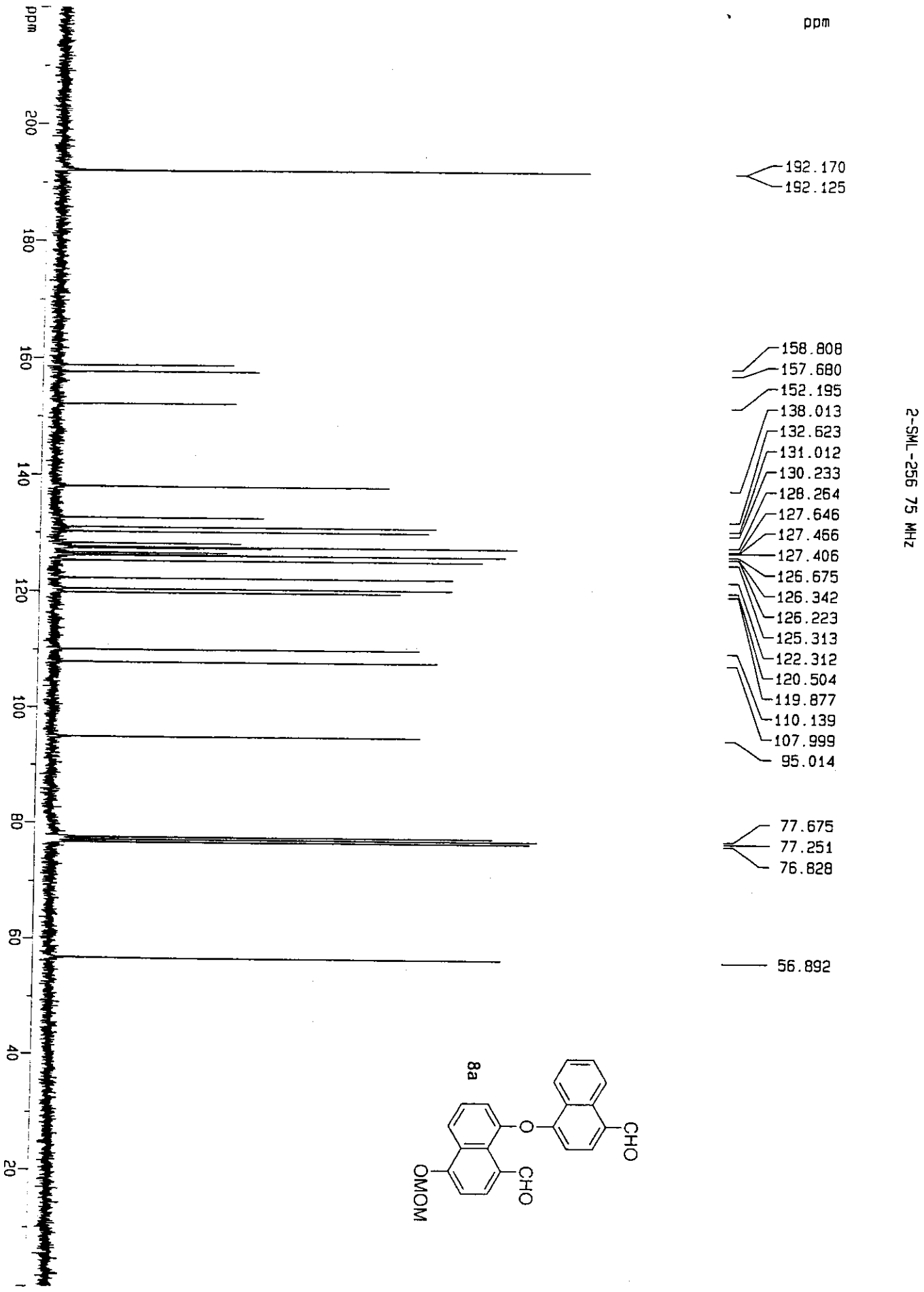

ppm
ppm
189.049
162.127
150.170
147.495
141.020
140.140
139.282
137.217
136.787
129.859
128.900
128.314
127.618
127.211
126.474
119.829
119.577
116.817
114.098
113.892
111.129
109.702
107.067
93.079
77.677
77.253
76.830
70.940
2-SML-103 75 MHz
OH
O
O
O
OBn

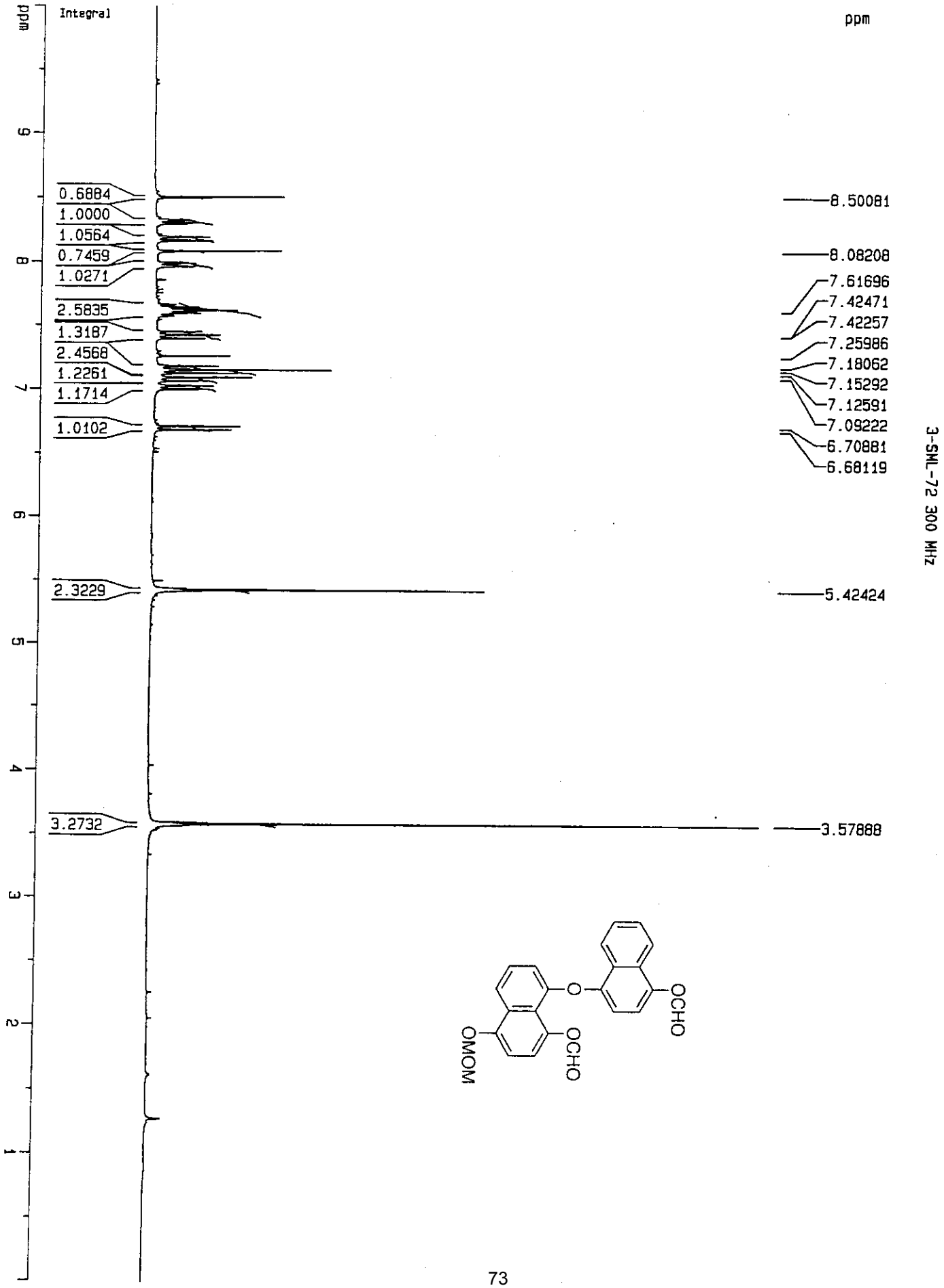
S10

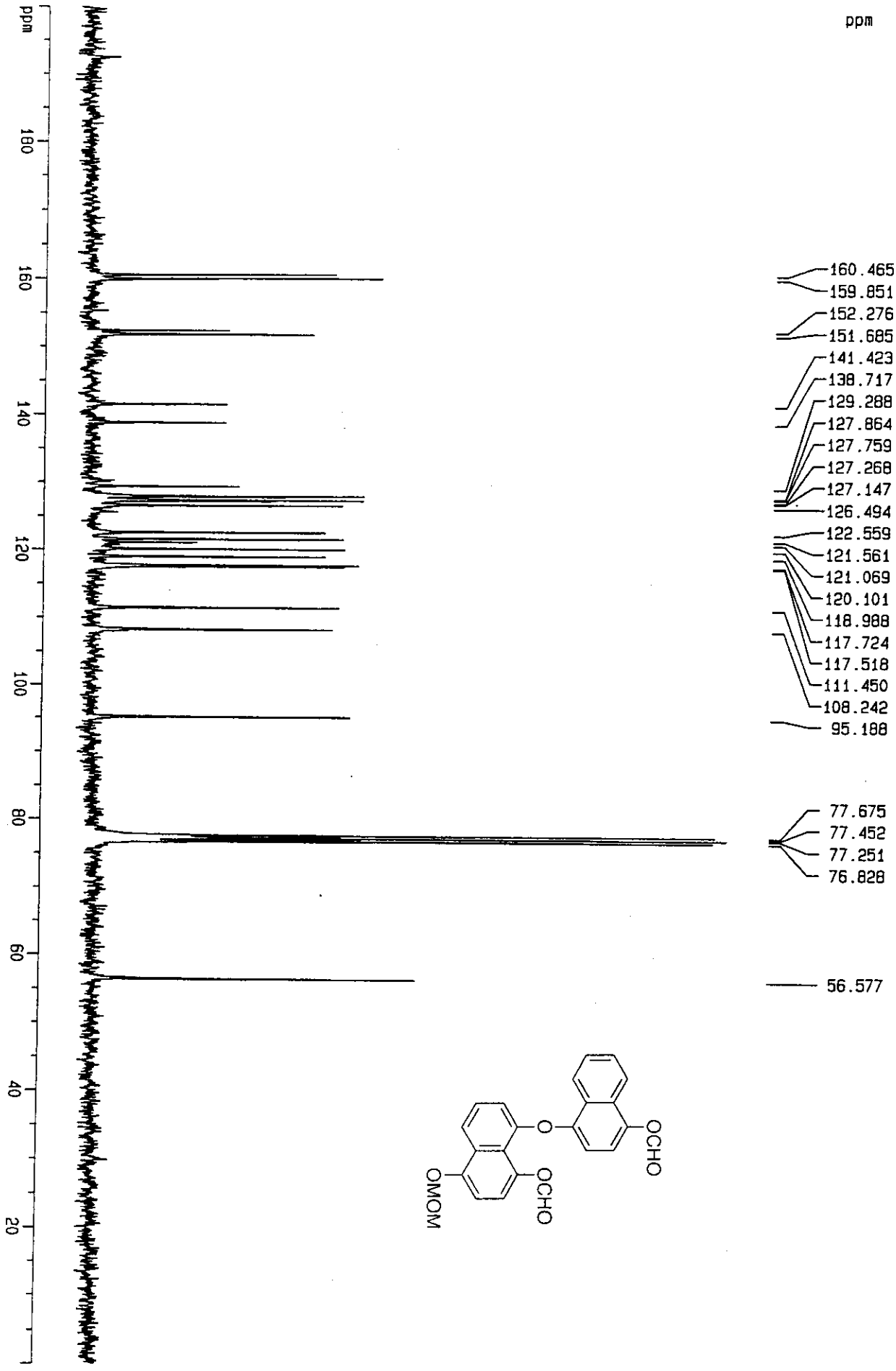

ppm
Integral
0.6910
0.9769
2.0180
1.0037
0.9082
0.9803
2.1073
3.0075
12.1086
12.1058
9.6159
9.6130
7.9267
7.8992
7.8918
7.8878
7.8641
7.8602
7.5222
7.4957
7.4688
7.1987
7.1949
7.1729
7.1689
7.1291
7.1017
5.4870
3.5640
3.5482
2-SML-239 300 MHz
OMOM
OH
CHO
7

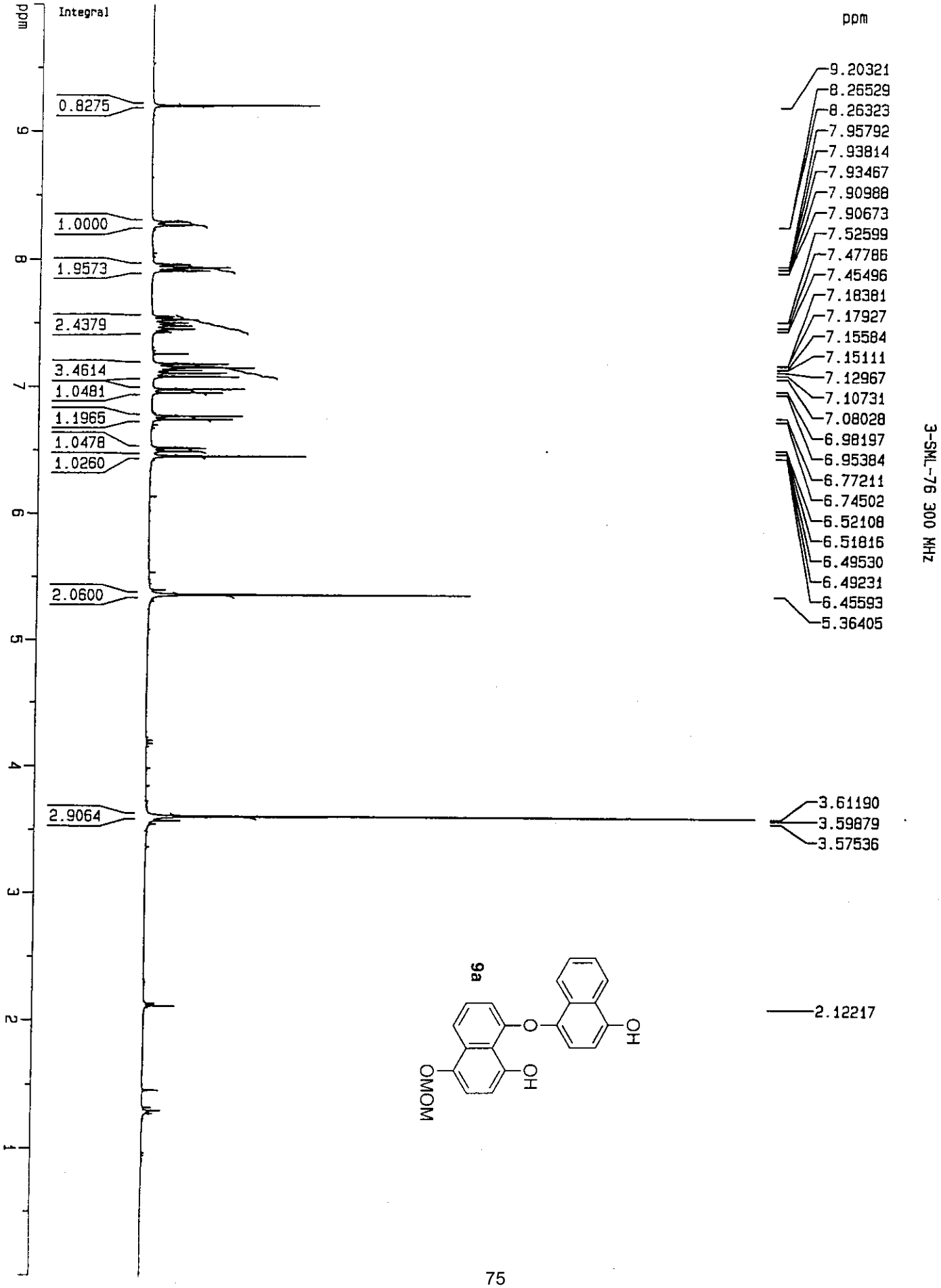

ppm
200
180
160
140
120
100
80
60
40
20
ppm
196.091
160.908
155.950
145.397
128.881
128.647
126.416
122.946
116.868
113.835
106.165
94.977
77.757
77.335
76.912
57.117
2-SML-239 75 MHz
OH
CHO
OMOM
7

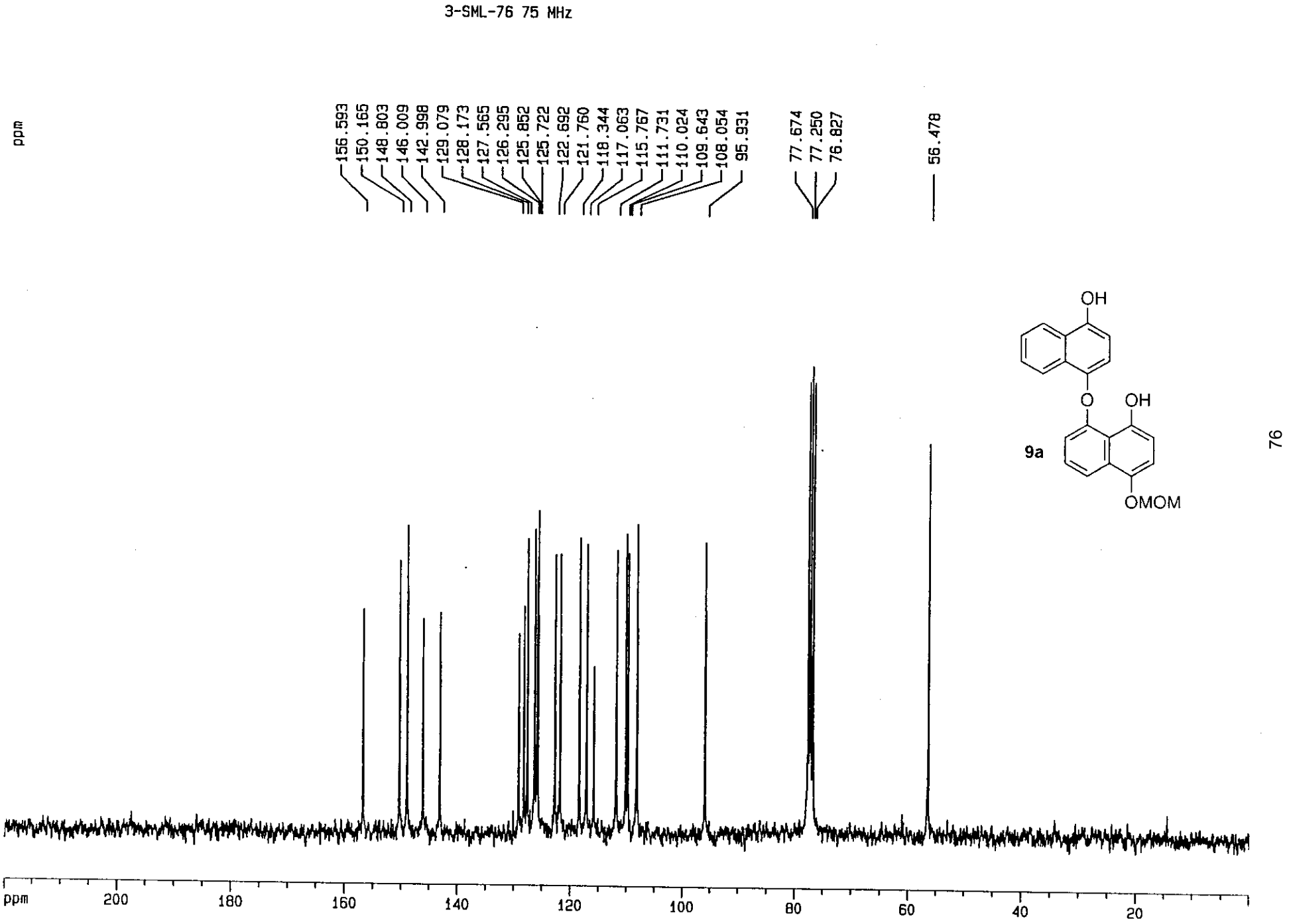

ppm
Integral
0.7509
0.9808
0.9434
1.0000
0.9709
0.9399
2.1931
1.0947
1.0982
1.0061
1.1347
0.9256
2.0625
2.9507
ppm
10.9050
10.9032
10.2173
9.3655
9.3628
9.3371
9.3345
8.4589
8.3664
8.3626
8.3382
8.3344
8.1638
8.1361
7.7984
7.7755
7.7712
7.6642
7.5614
7.5359
7.5335
7.5078
7.3141
7.3103
7.2887
7.2848
7.2569
7.2295
6.7778
6.7511
5.5021
3.5887
2-SML-256 300 MHz
8a
OMOM
CHO
CHO
O

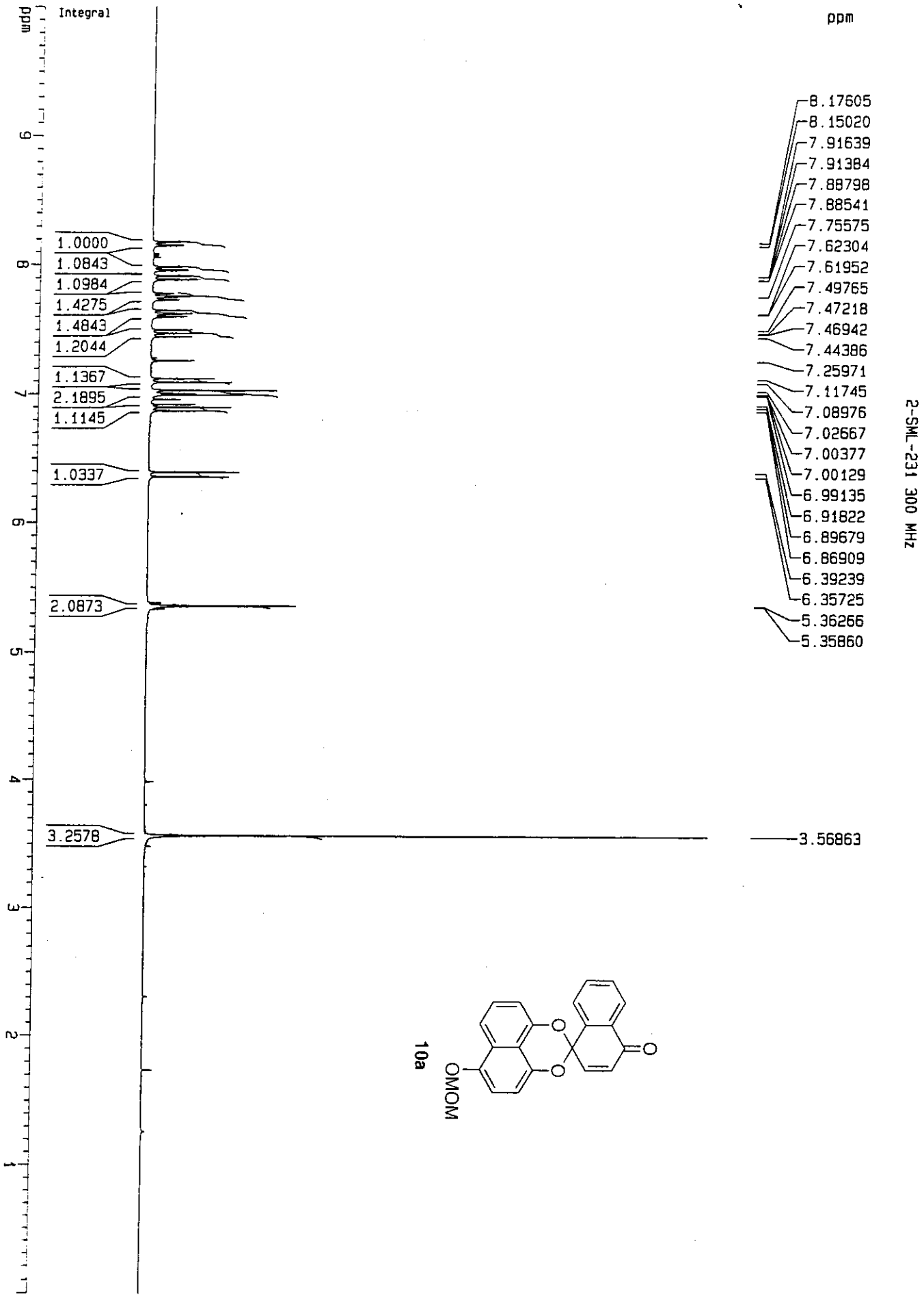

ppm
200
180
160
140
120
100
80
60
40
20
ppm
192.170
192.125
158.808
157.680
152.195
138.013
132.623
131.012
130.233
128.264
127.646
127.466
127.406
126.675
126.342
126.223
125.313
122.312
120.504
119.877
110.139
107.999
95.014
77.675
77.251
76.828
56.892
2-SML-256 75 MHz
8a
O
CHO
CHO
OMOM

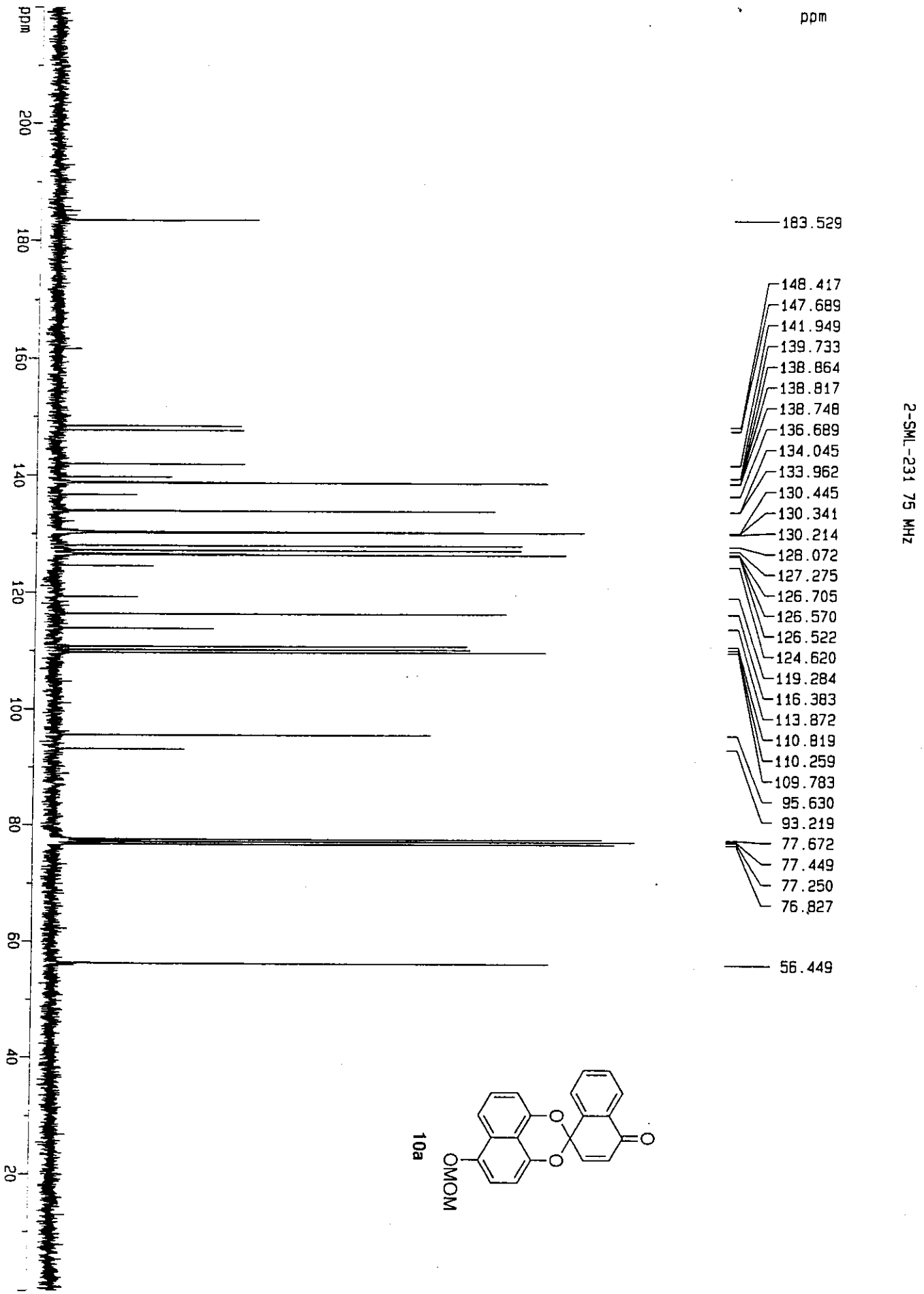

Integral
ppm
0.6884
1.0000
1.0564
0.7459
1.0271
2.5835
1.3187
2.4568
1.2261
1.1714
1.0102
2.3229
3.2732
ppm
8.50081
8.08208
7.61696
7.42471
7.42257
7.25986
7.18062
7.15292
7.12591
7.09222
6.70881
6.68119
5.42424
3.57888
3-SML-72 300 MHz
OMOM
OCHO
O
OCHO

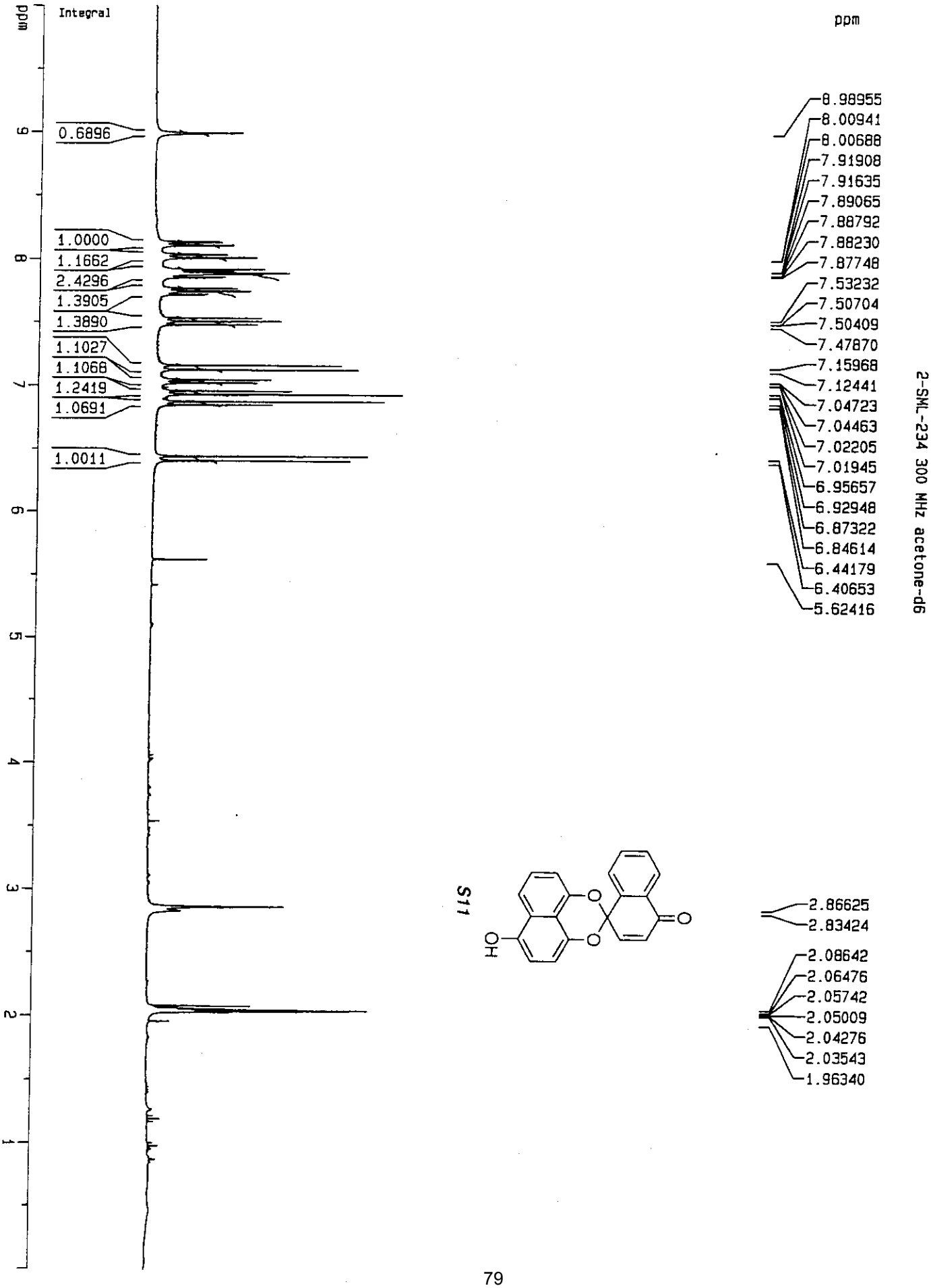

ppm
160.465
159.851
152.276
151.685
141.423
138.717
129.288
127.864
127.759
127.268
127.147
126.494
122.559
121.561
121.069
120.101
118.988
117.724
117.518
111.450
108.242
95.188
77.675
77.452
77.251
76.828
56.577
OMOM
OCHO
OCHO
3-SML-72 75 MHz

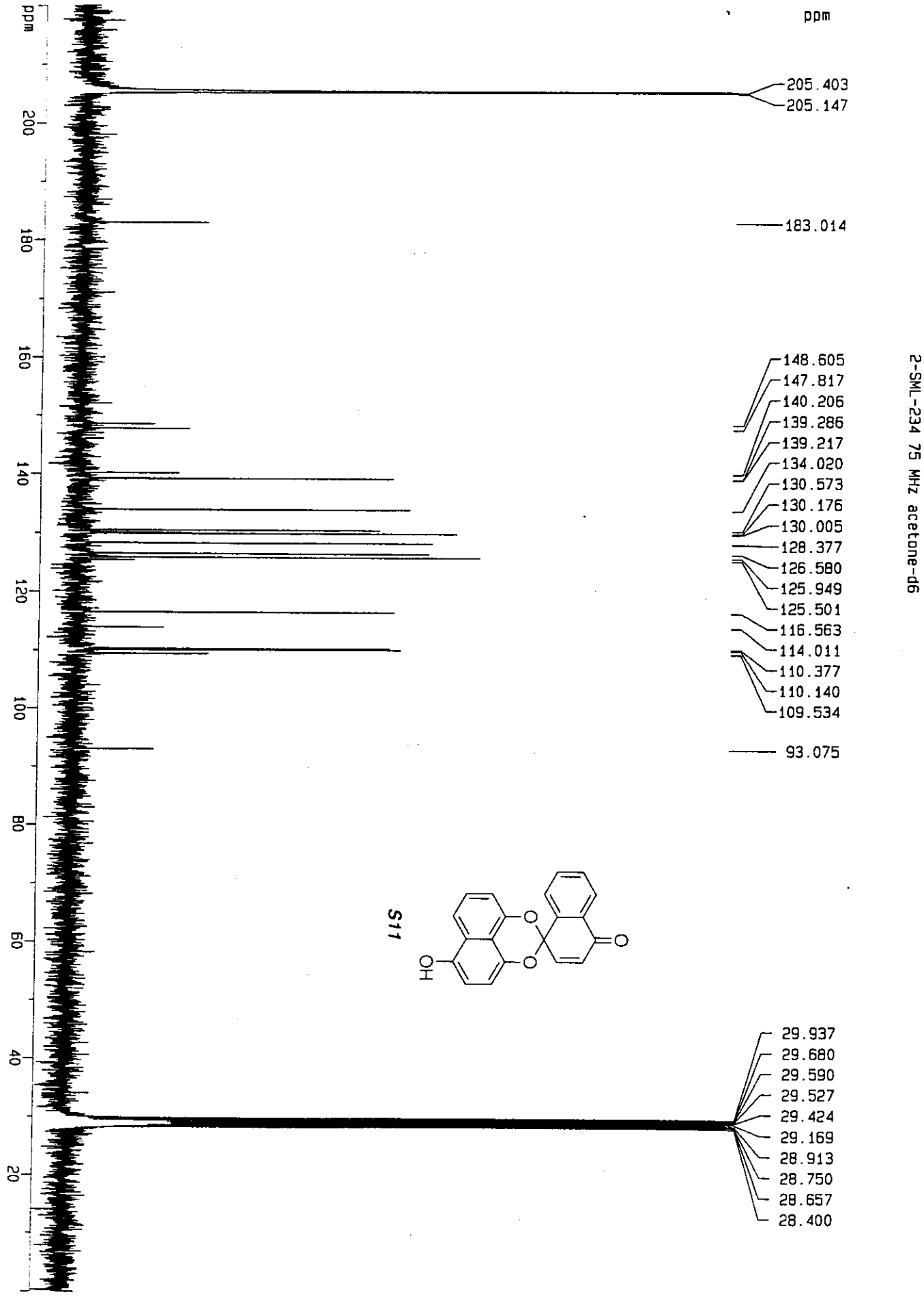

ppm
Integral
0.8275
1.0000
1.9573
2.4379
3.4614
1.0481
1.1965
1.0478
1.0260
2.0600
2.9064
ppm
9.20321
8.26529
8.26323
7.95792
7.93814
7.93467
7.90988
7.90673
7.52599
7.47786
7.45496
7.18381
7.17927
7.15584
7.15111
7.12967
7.10731
7.08028
6.98197
6.95384
6.77211
6.74502
6.52108
6.51816
6.49530
6.49231
6.45593
5.36405
3.61190
3.59879
3.57536
2.12217
3-SWL-76 300 MHz
9a
OMOM
OH
OH
O

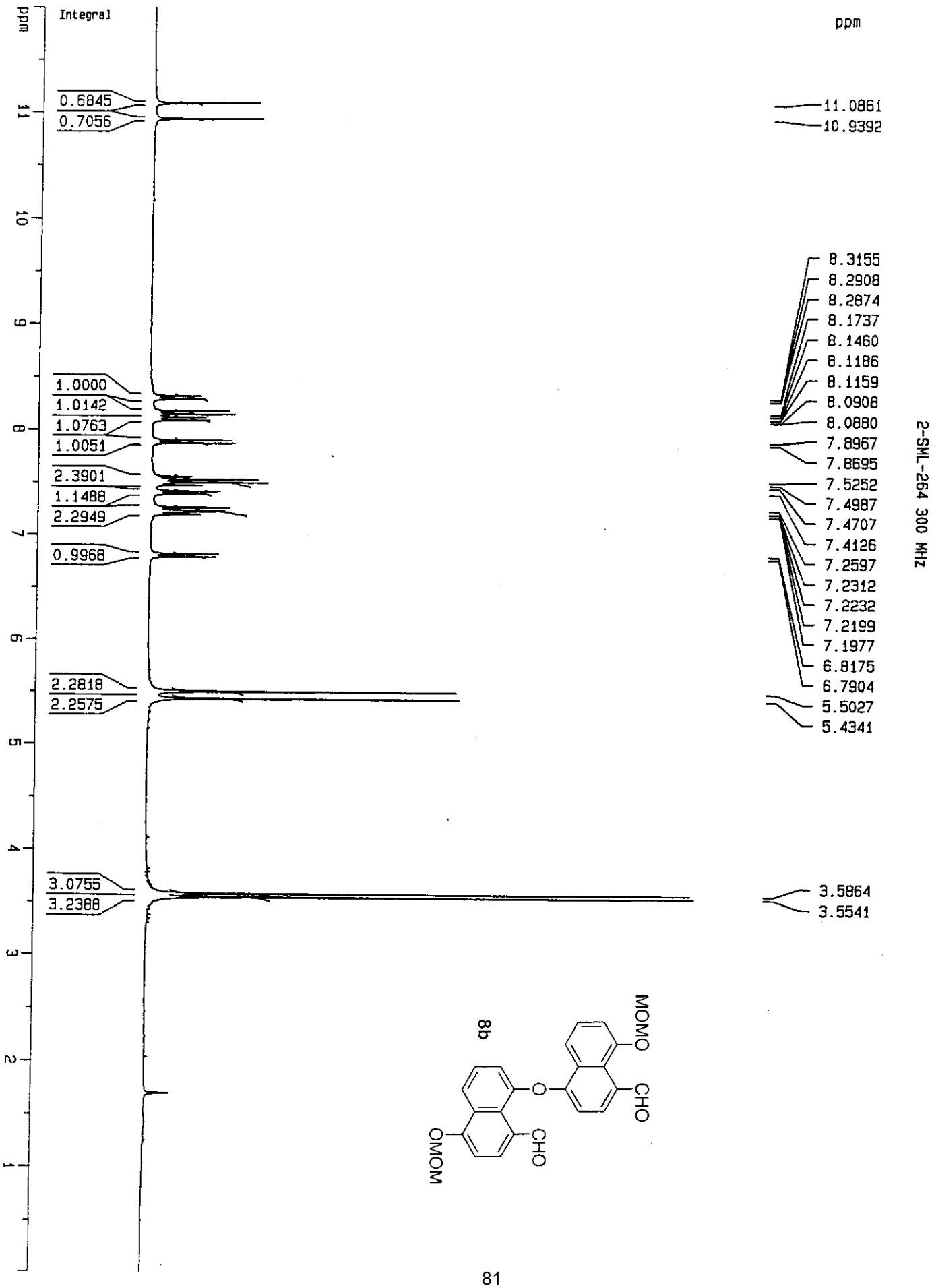

3-SML-76 75 MHz
ppm
156.593
150.165
148.803
146.009
142.998
129.079
128.173
127.565
126.295
125.852
125.722
122.692
121.760
118.344
117.063
115.767
111.731
110.024
109.643
108.054
95.931
77.674
77.250
76.827
56.478
OH
O
OH
OMOM
9a
ppm
200
180
160
140
120
100
80
60
40
20

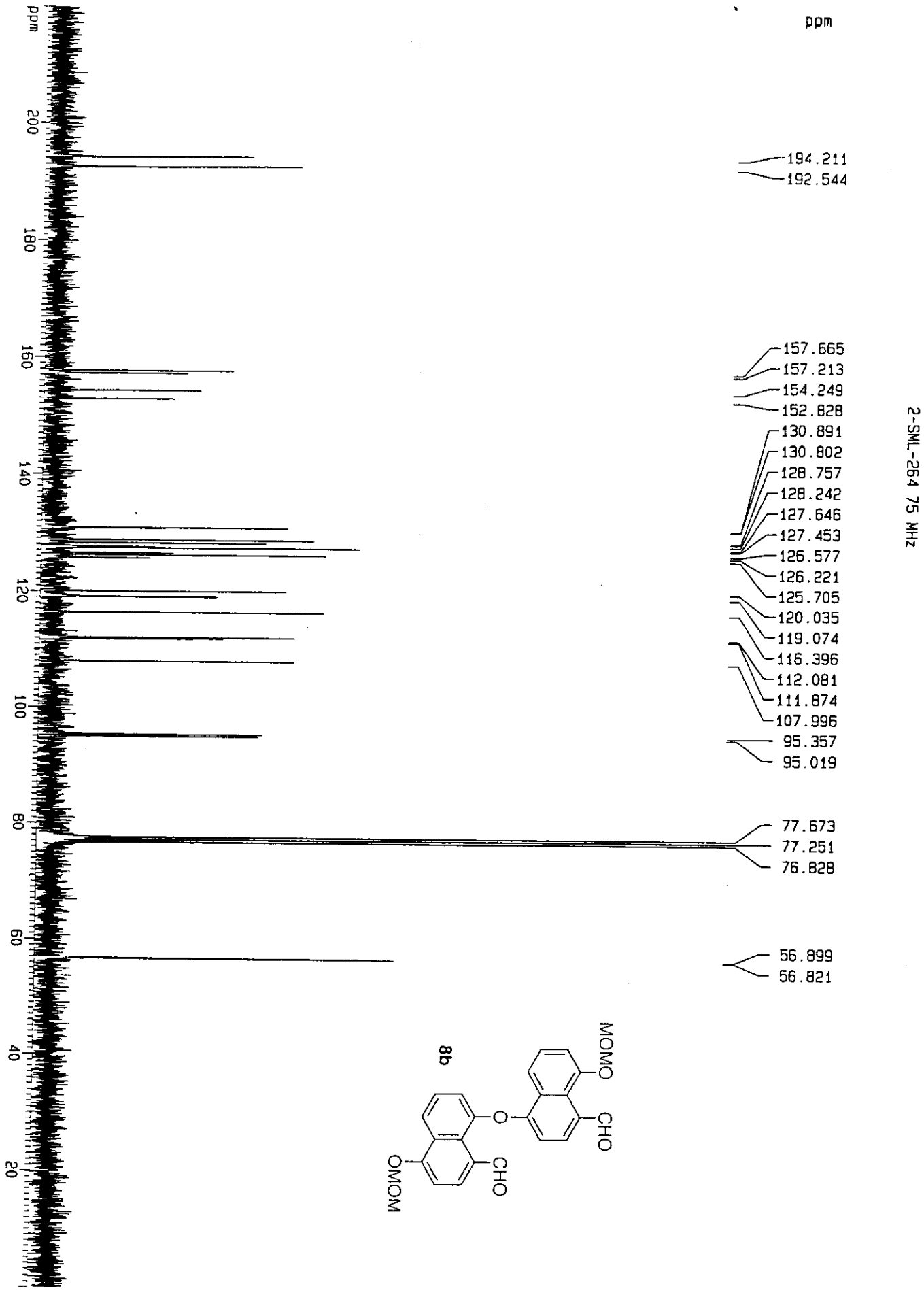

Integral
ppm
1.0000
1.0843
1.0984
1.4275
1.4843
1.2044
1.1367
2.1895
1.1145
1.0337
2.0873
3.2578
8.17605
8.15020
7.91639
7.91384
7.88798
7.88541
7.75575
7.62304
7.61952
7.49765
7.47218
7.46942
7.44386
7.25971
7.11745
7.08976
7.02667
7.00377
7.00129
6.99135
6.91822
6.89679
6.86909
6.39239
6.35725
5.36266
5.35860
3.56863
2-SML-231 300 MHz
OMOM
10a

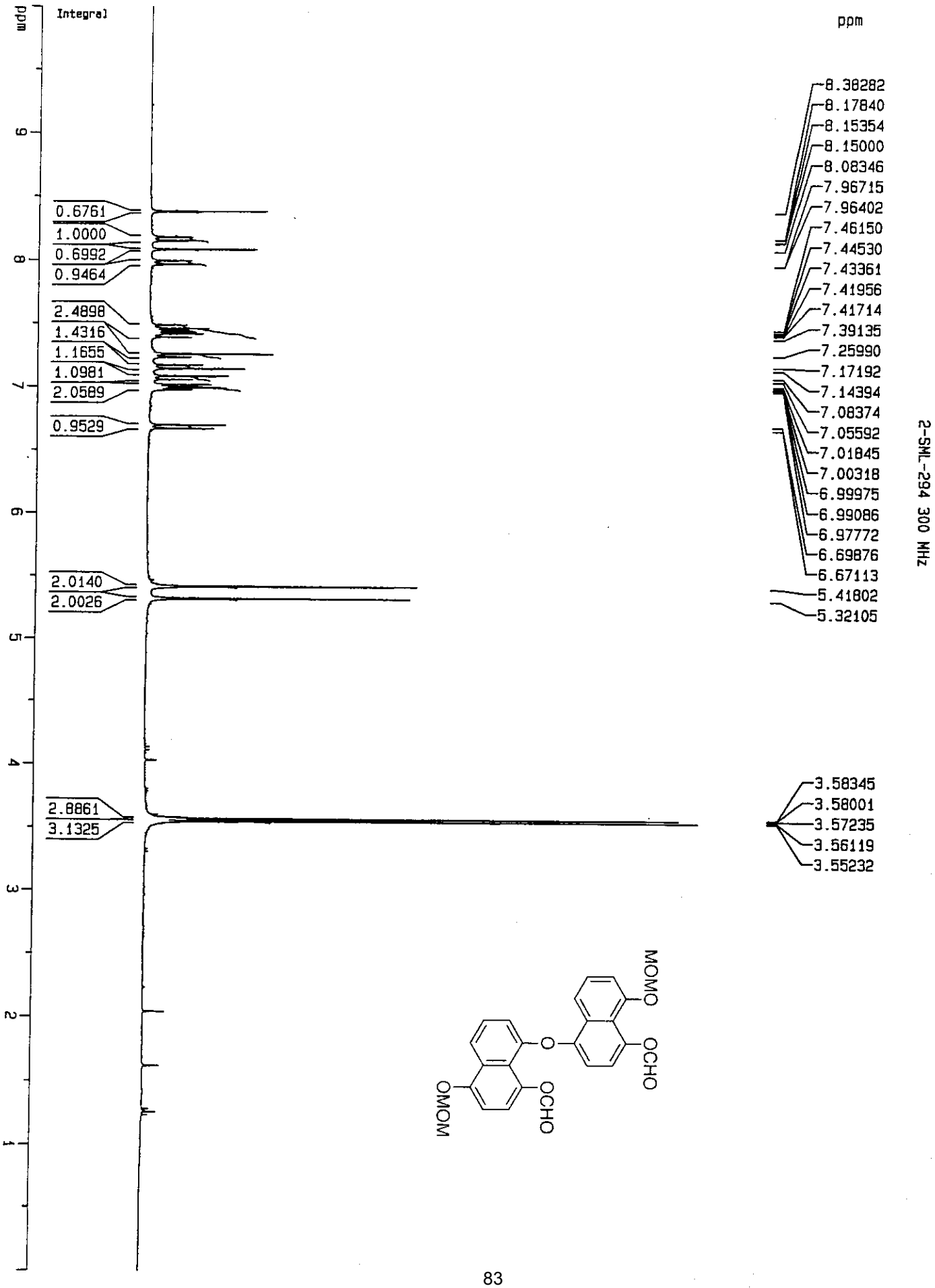

ppm
200
180
160
140
120
100
80
60
40
20
ppm
183.529
148.417
147.689
141.949
139.733
138.864
138.817
138.748
136.689
134.045
133.962
130.445
130.341
130.214
128.072
127.275
126.705
126.570
126.522
124.620
119.284
116.383
113.872
110.819
110.259
109.783
95.630
93.219
77.672
77.449
77.250
76.827
56.449
2-SML-231 75 MHz
10a
OMOM
O
O
O

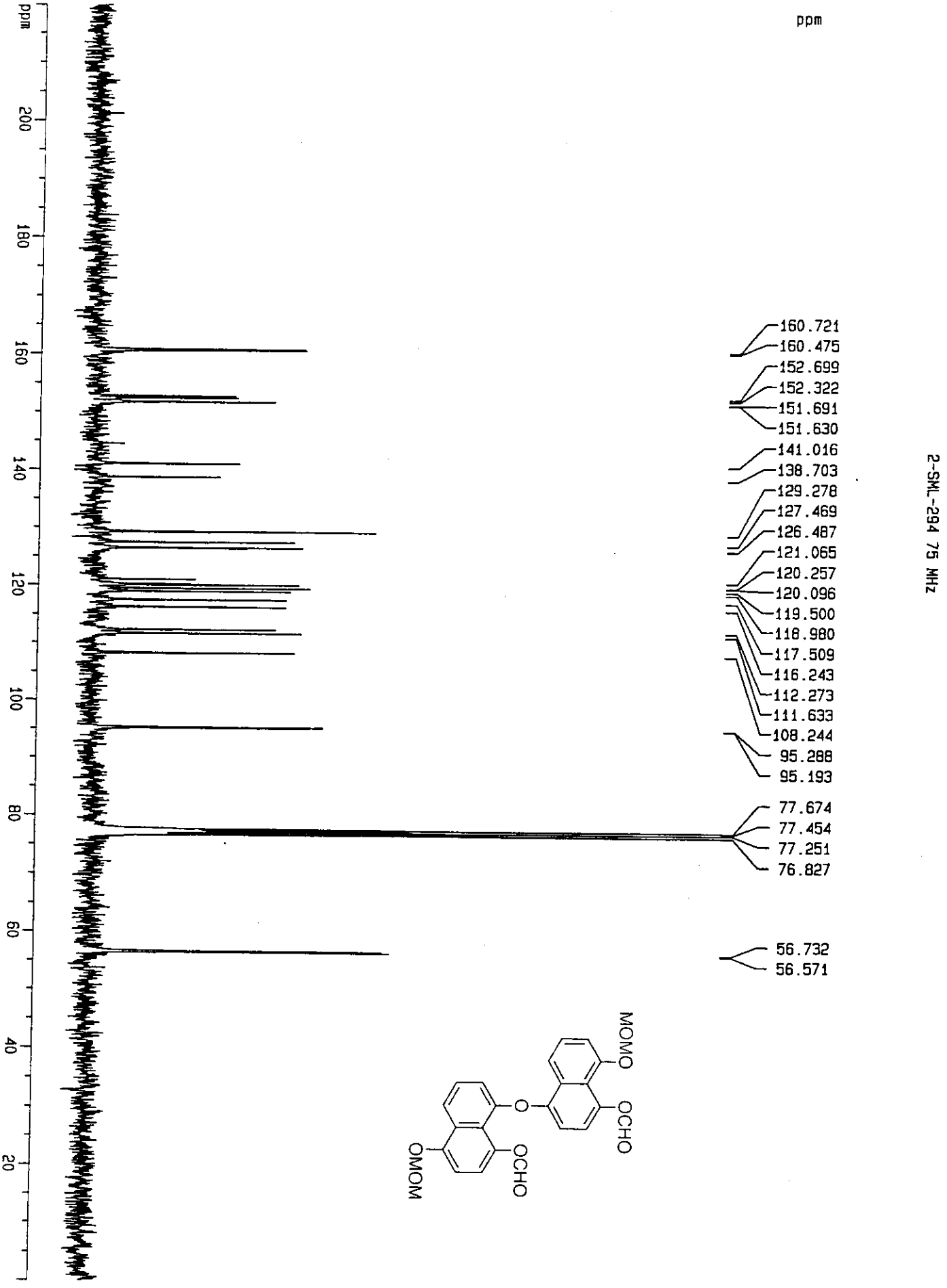

ppm
Integral
0.6896
1.0000
1.1662
2.4296
1.3905
1.3890
1.1027
1.1068
1.2419
1.0691
1.0011
ppm
8.98955
8.00941
8.00688
7.91908
7.91635
7.89065
7.88792
7.88230
7.87748
7.53232
7.50704
7.50409
7.47870
7.15968
7.12441
7.04723
7.04463
7.02205
7.01945
6.95657
6.92948
6.87322
6.84614
6.44179
6.40653
5.62416
2-SML-234 300 MHz acetone-d6
S11
OH
O
O
O
2.86625
2.83424
2.08642
2.06476
2.05742
2.05009
2.04276
2.03543
1.96340

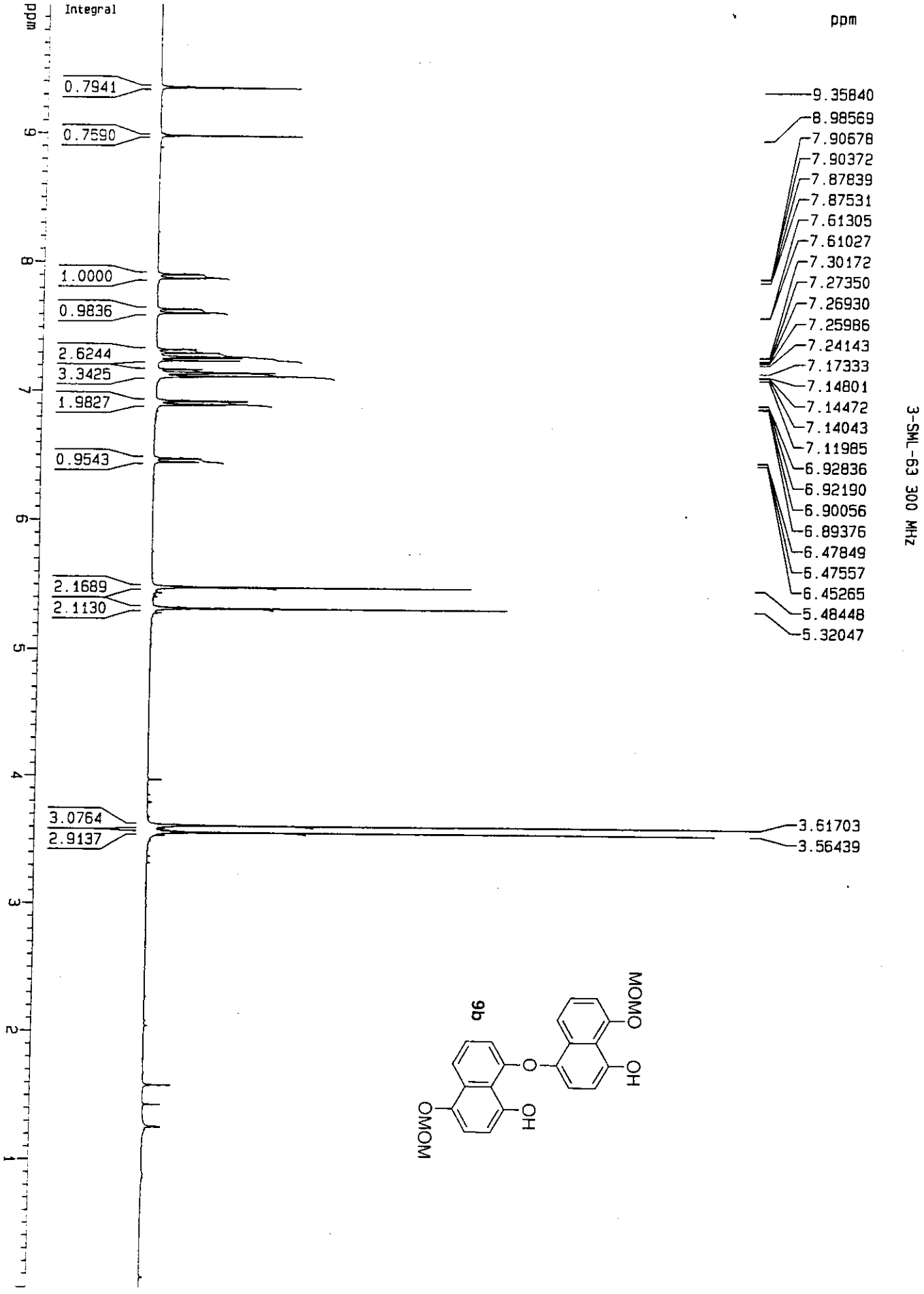

ppm
205.403
205.147
183.014
148.605
147.817
140.206
139.286
139.217
134.020
130.573
130.176
130.005
128.377
126.580
125.949
125.501
116.563
114.011
110.377
110.140
109.534
93.075
29.937
29.680
29.590
29.527
29.424
29.169
28.913
28.750
28.657
28.400
2-SML-234 75 MHz acetone-d6
S11
OH
O
O
O


ppm
Integral
0.6845
0.7056
1.0000
1.0142
1.0763
1.0051
2.3901
1.1488
2.2949
0.9968
2.2818
2.2575
3.0755
3.2388
ppm
11.0861
10.9392
8.3155
8.2908
8.2874
8.1737
8.1460
8.1186
8.1159
8.0908
8.0880
7.8967
7.8695
7.5252
7.4987
7.4707
7.4126
7.2597
7.2312
7.2232
7.2199
7.1977
6.8175
6.7904
5.5027
5.4341
3.5864
3.5541
2-SML-264 300 MHz
8b
MOMO
CHO
O
CHO
OMOM


ppm
194.211
192.544
157.665
157.213
154.249
152.828
130.891
130.802
128.757
128.242
127.646
127.453
126.577
126.221
125.705
120.035
119.074
116.396
112.081
111.874
107.996
95.357
95.019
77.673
77.251
76.828
56.899
56.821
2-SML-264 75 MHz
8b
MOMO
CHO
O
OMOM
CHO

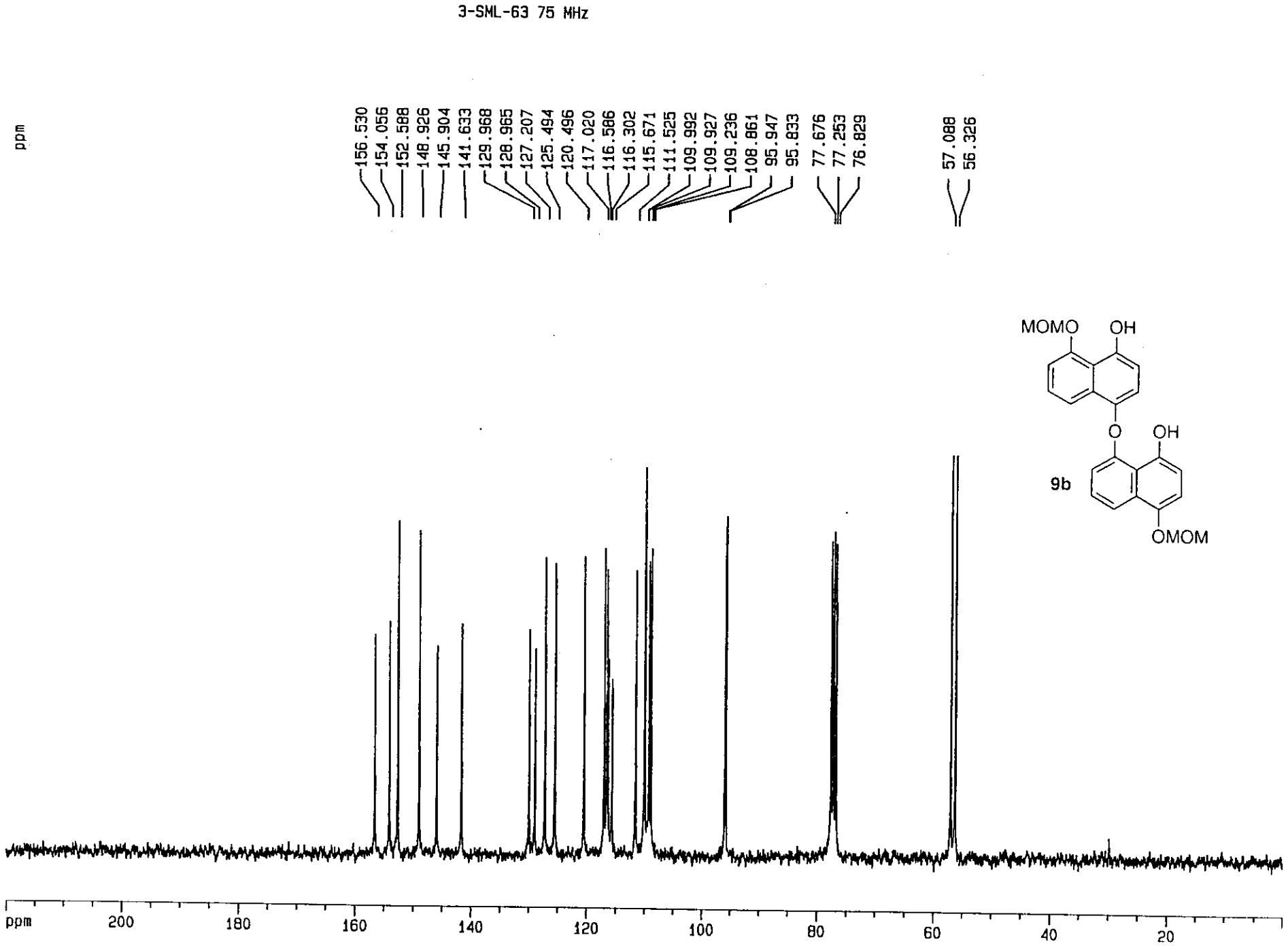

Integral
ppm
0.6761
1.0000
0.6992
0.9464
2.4898
1.4316
1.1655
1.0981
2.0589
0.9529
2.0140
2.0026
2.8861
3.1325
ppm
8.38282
8.17840
8.15354
8.15000
8.08346
7.96715
7.96402
7.46150
7.44530
7.43361
7.41956
7.41714
7.39135
7.25990
7.17192
7.14394
7.08374
7.05592
7.01845
7.00318
6.99975
6.99086
6.97772
6.69876
6.67113
5.41802
5.32105
3.58345
3.58001
3.57235
3.56119
3.55232
2-SML-294 300 MHz
MOMO
OCHO
O
OMOM
OCHO

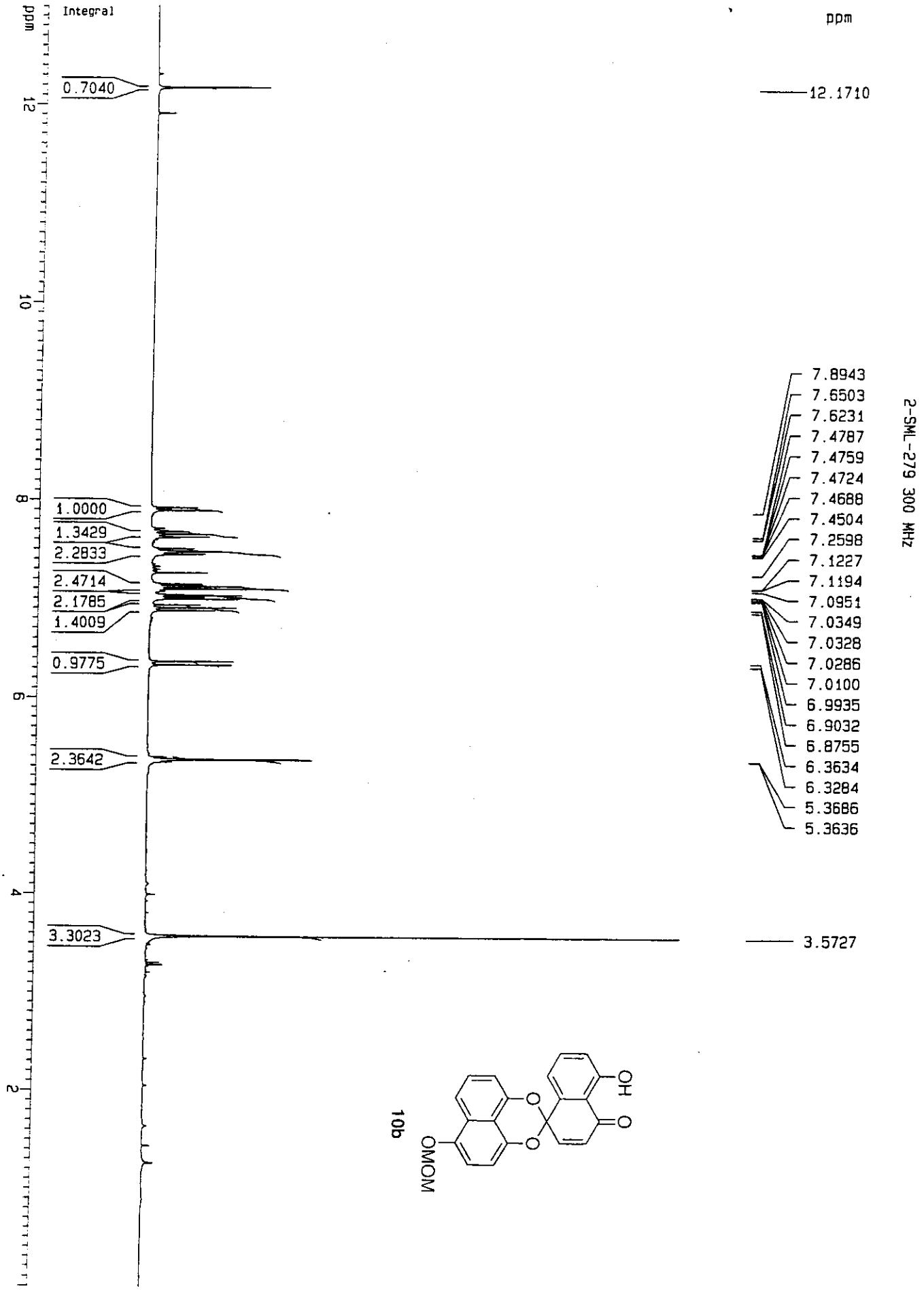

ppm
ppm
2-SML-294 75 MHz
160.721
160.475
152.699
152.322
151.691
151.630
141.016
138.703
129.278
127.469
126.487
121.065
120.257
120.096
119.500
118.980
117.509
116.243
112.273
111.633
108.244
95.288
95.193
77.674
77.454
77.251
76.827
56.732
56.571
MOMO
OCHO
OMOM
OCHO

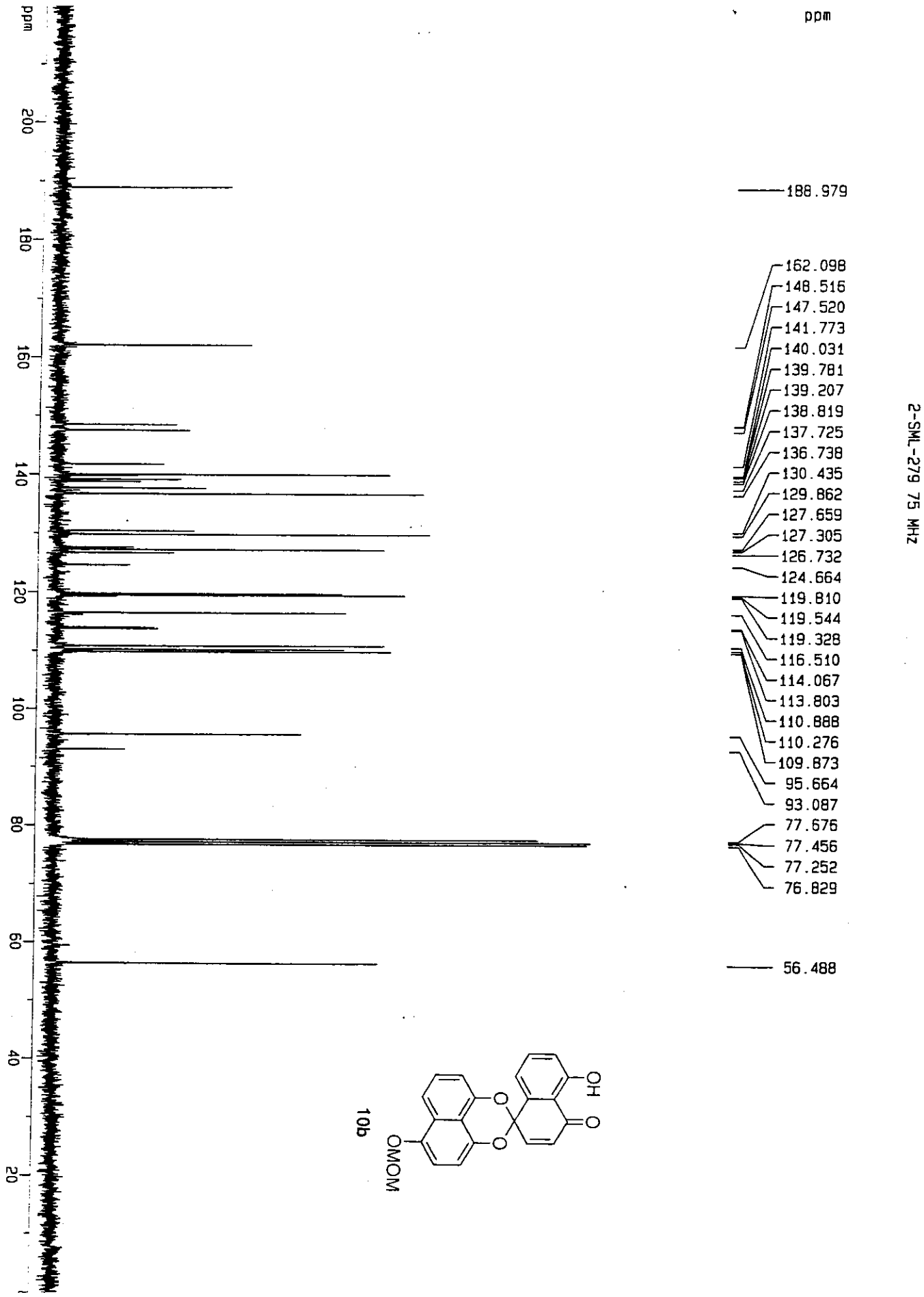

Integral
ppm
0.7941
0.7590
1.0000
0.9836
2.6244
3.3425
1.9827
0.9543
2.1689
2.1130
3.0764
2.9137
9.35840
8.98569
7.90678
7.90372
7.87839
7.87531
7.61305
7.61027
7.30172
7.27350
7.26930
7.25986
7.24143
7.17333
7.14801
7.14472
7.14043
7.11985
6.92836
6.92190
6.90056
6.89376
6.47849
6.47557
6.45265
5.48448
5.32047
3.61703
3.56439
3-SWL-63 300 MHz

9b

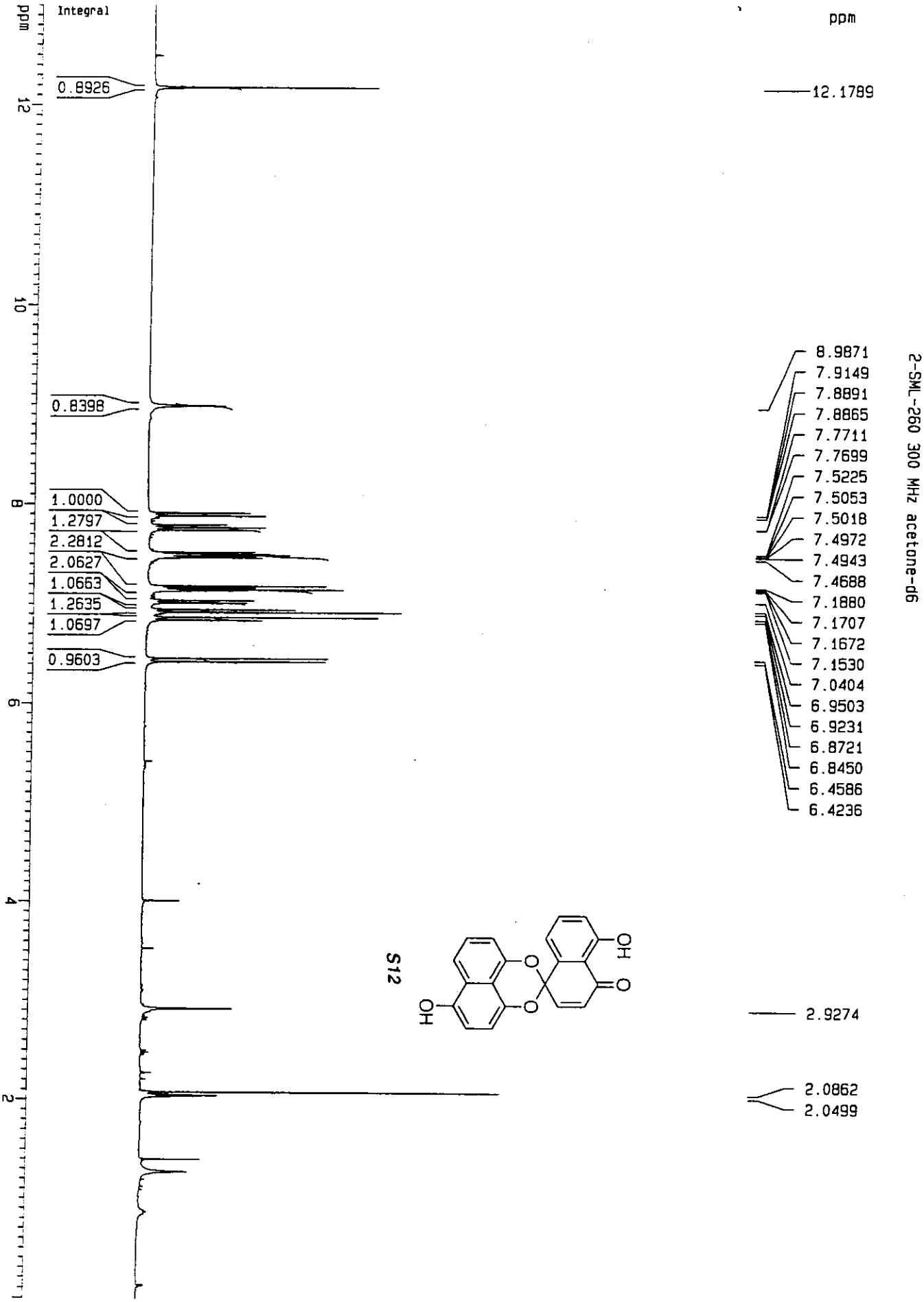
MOMO
OH
O
OH
OMOM

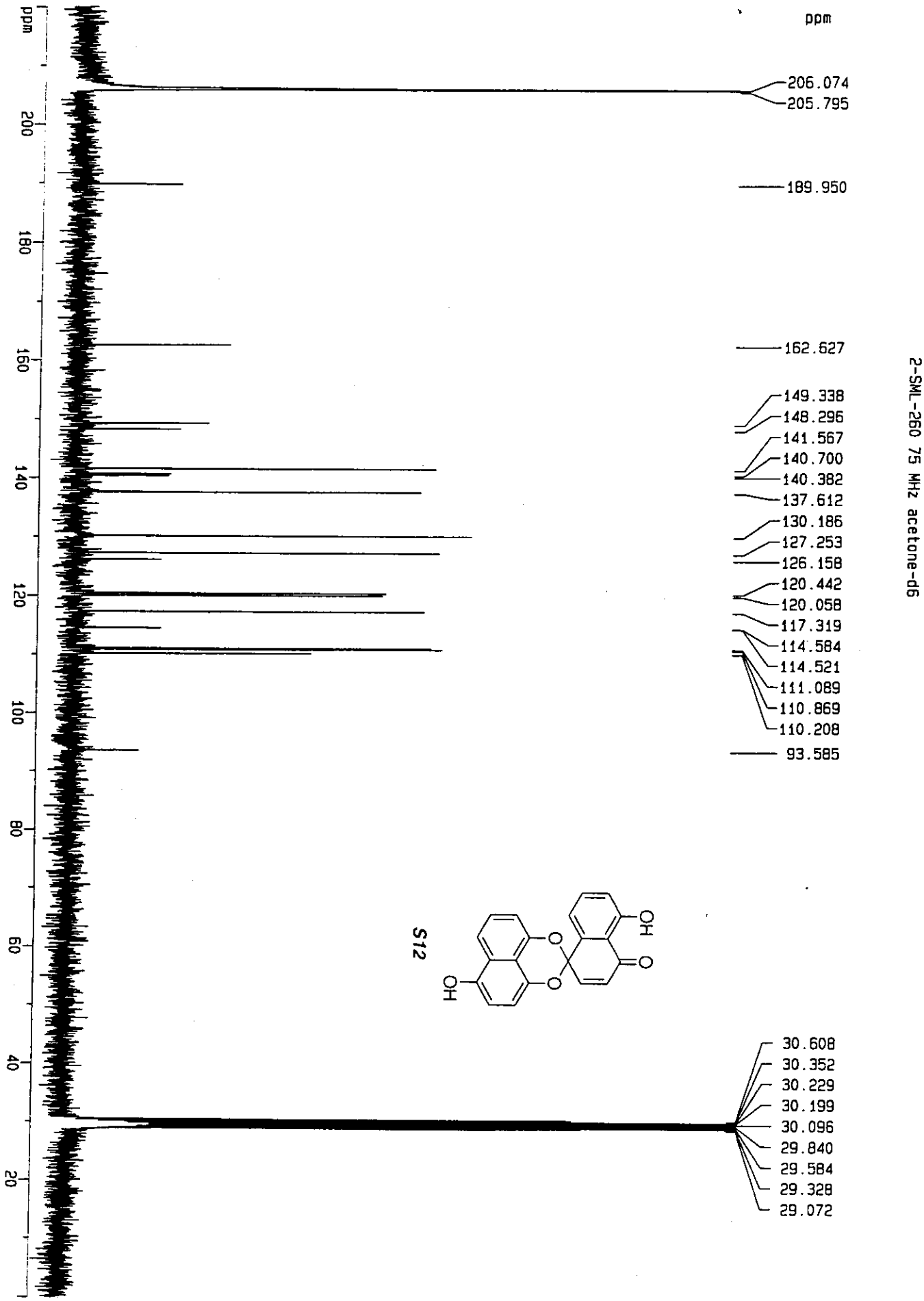

3-SML-63 75 MHz
ppm
156.530
154.056
152.588
148.926
145.904
141.633
129.968
128.965
127.207
125.494
120.496
117.020
116.586
116.302
115.671
111.525
109.992
109.927
109.236
108.861
95.947
95.833
77.676
77.253
76.829
57.088
56.326
MOMO
OH
O
OH
9b
OMOM
ppm
200
180
160
140
120
100
80
60
40
20


ppm
Integral
0.7040
1.0000
1.3429
2.2833
2.4714
2.1785
1.4009
0.9775
2.3642
3.3023
ppm
12.1710
7.8943
7.6503
7.6231
7.4787
7.4759
7.4724
7.4688
7.4504
7.2598
7.1227
7.1194
7.0951
7.0349
7.0328
7.0286
7.0100
6.9935
6.9032
6.8755
6.3634
6.3284
5.3686
5.3636
3.5727
2-SML-279 300 MHZ
10b
OMOM
HO
O
O
O


ppm
188.979
162.098
148.516
147.520
141.773
140.031
139.781
139.207
138.819
137.725
136.738
130.435
129.862
127.659
127.305
126.732
124.664
119.810
119.544
119.328
116.510
114.067
113.803
110.888
110.276
109.873
95.664
93.087
77.676
77.456
77.252
76.829
56.488
2-SWL-279 75 MHz
OH
O
O
O
OMOM

10b


Integral
ppm
0.8926
0.8398
1.0000
1.2797
2.2812
2.0627
1.0663
1.2635
1.0697
0.9603
ppm
12.1789
8.9871
7.9149
7.8891
7.8865
7.7711
7.7699
7.5225
7.5053
7.5018
7.4972
7.4943
7.4688
7.1880
7.1707
7.1672
7.1530
7.0404
6.9503
6.9231
6.8721
6.8450
6.4586
6.4236
2-SML-260 300 MHz acetone-d6
S12
OH
O
O
O
OH
2.9274
2.0862
2.0499


ppm
206.074
205.795
189.950
162.627
149.338
148.296
141.567
140.700
140.382
137.612
130.186
127.253
126.158
120.442
120.058
117.319
114.584
114.521
111.089
110.869
110.208
93.585
30.608
30.352
30.229
30.199
30.096
29.840
29.584
29.328
29.072
2-SML-260 75 MHz acetone-d6
OH
O
O
O
OH

S12