

Supporting Information

Synthesis of 2-Substituted 7-Hydroxy-benzofuran-4-carboxylates via Addition of Silyl Enol

Ethers to *o*-Benzoquinone Esters

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Supporting Information

Synthesis of 2-Substituted 7-Hydroxy-benzofuran-4-carboxylates via Addition of Silyl Enol Ethers to *o*-Benzoquinone Esters

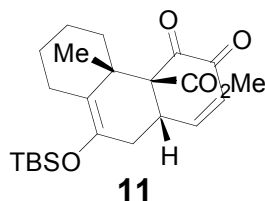
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General Experimental: All reactions were carried out under an atmosphere of argon unless otherwise specified. Anhydrous solvents were distilled immediately prior to use: tetrahydrofuran (THF) and methanol from magnesium turnings. All other reagents were reagent grade and used without purification unless otherwise stated. ¹H and ¹³C NMR spectra were recorded on Bruker or Varian spectrometers at 400 or 300 MHz for proton and at 100 or 75 MHz for carbon. ¹H and ¹³C NMR data are reported in parts per million (δ) downfield from tetramethylsilane. The following abbreviations are used: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), b (broad). Infrared spectra were recorded on a Thermo Nicolet Avatar 370 infrared spectrophotometer. Thin-layer chromatography (TLC) was performed using Merck silica gel 60 F254 0.2 mm aluminum-backed plates. Visualization was accomplished using ultraviolet light or one of the following stains: anisaldehyde or potassium permanganate. Flash column chromatography was carried out using ICN Biomedicals silica gel 60 (230-400 mesh).

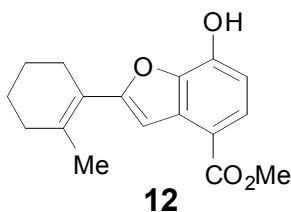
(±) (4aS, 4bS, 10aS) Methyl 9-(1,1-Dimethylethyl)dimethylsilyloxy-4b-methyl-3,4-dioxo-3,4,4a,4b,5,6,7,8,10,10a-decahydrophenanthrene-4a-carboxylate (11). To a solution of methyl 2,3-dihydroxybenzoate **7**¹ (84.1 mg, 0.5 mmol) and 1-(1,1-dimethylethyl)dimethylsilyloxyethenyl)-2-methylcyclohexene **10** (631.2 mg, 2.5 mmol), prepared from **9** as previously reported,² in 4 mL THF at 0 °C was added 1 mL (0.55 mmol) bis(trifluoroacetoxy) iodobenzene in (0.55 M solution in THF) dropwise over 10 min. A yellow solution formed and was allowed to warm and stir for 4 h at 21 °C, which resulted in a dark brown solution. The solution was

concentrated in vacuo and the crude residue was purified by flash column chromatography over silica gel (25% ethyl acetate in hexanes) to yield **11** as dark yellow crystals (129.7 mg, 0.31 mmol, 62%). Recrystallization from hexanes and ethyl acetate yielded light yellow crystals (106.7 mg, 0.23 mmol, 51%). mp 160-162 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.46 (dd, *J* = 9.8, 6.6 Hz, 1H), 6.28 (d, *J* = 9.8 Hz, 1H), 3.69 (s, 3H), 3.65 (ddd, *J* = 11.6, 6.6, 6.6 Hz, 1H), 2.85 (bd, *J* = 15.0 Hz, 1H), 2.53 (ddd, *J* = 17.6, 6.8, 2.9 Hz, 1H), 2.44 (ddd, *J* = 13.0, 13.0, 4.0 Hz, 1H), 1.84-1.77 (m, 1H), 1.71-1.66 (m, 2H), 1.69 (ddd, *J* = 17.5, 11.5, 3.3 Hz, 1H), 1.54-1.40 (m, 2H), 1.17 (s, 3H), 1.16 (bd, *J* = 15.0 Hz, 1H), 0.90 (s, 9H), 0.084 (s, 3H), 0.028 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 191.3, 185.3, 170.5, 158.5, 137.0, 129.8, 119.8, 70.9, 52.9, 39.3, 38.6, 36.2, 32.7, 26.0, 25.0, 23.9, 23.2, 22.3, 18.3, -3.44, -4.18. IR (neat, cm⁻¹) 2952, 2929, 2853, 1716, 1683.

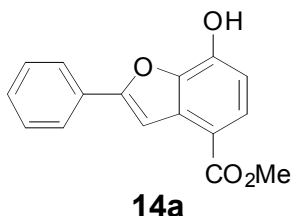


General Procedure For Benzofuran Synthesis. To a solution of methyl 3,4-dihydroxybenzoate **8**³ (1.0 mmol) and the silyl enol ether **13** (4 mmol) in 8 mL THF at 0 °C was added 2 mL (1.1 mmol) bis(trifluoroacetoxy) iodobenzene (0.55 M solution in THF) dropwise over 10 min, forming a green solution initially. The reaction was allowed to stir at 0 °C (unless specified otherwise) for 4 h, the solution becoming a dark yellow. To the solution was added 0.5 mL HCl, (4M solution in dioxane) and 1.0 mL methanol. The mixture was refluxed for 1 h and the solution lightened in color. The solution was extracted with 30 mL of diethyl ether, washed with 2 x 10 mL saturated NaHCO₃ and 1 x 10 mL brine, dried over MgSO₄, filtered and concentrated in vacuo to yield an oily residue. The residue was purified by flash column chromatography over silica gel (ether and hexanes) to yield the benzofuran product **14**. The products were often recrystallized to give purer material.

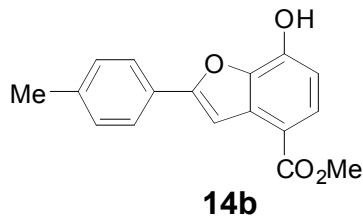
Methyl 7-hydroxy-2-(2-methylcyclohex-1-enyl)benzofuran-4-carboxylate (12). Recrystallization from hexanes and ethyl acetate gave a bright white solid in 25% yield. mp 170-172 °C. ¹H-NMR (400 MHz, DMSO) δ 10.92 (s, 1H), 7.72 (d, *J* = 8.4 Hz, 1H), 7.07 (s, 1H), 6.78 (d, *J* = 8.4 Hz, 1H), 3.84 (s, 3H), 2.44-2.37 (m, 2H), 2.23-2.15 (m, 2H), 2.00 (s, 3H), 1.72-1.57 (m, 4H). ¹³C-NMR (100 MHz, DMSO) δ 166.1, 158.8, 146.9, 141.9, 137.0, 130.9, 128.7, 127.3, 120.8, 112.0, 109.9, 51.5, 32.7, 27.2, 22.1, 22.1, 21.9. IR (neat, cm⁻¹) 3330, 2932, 2858, 1683.



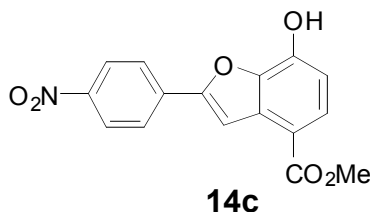
Methyl 7-hydroxy-2-phenylbenzofuran-4-carboxylate (14a). Tan crystals from toluene. 73% yield. mp 188-190 °C. ¹H NMR (400 MHz, DMSO) δ 11.12 (s, 1H), 7.99 (d, *J* = 7.5 Hz, 2H), 7.78 (d, *J* = 8.4, 1H), 7.73 (s, 1H), 7.53 (m, 2H), 7.46 (m, 1H), 6.86 (d, 1H), 3.89 (s, 3H). ¹³C NMR (100 MHz, DMSO) δ 166.0, 156.6, 147.1, 142.8, 131.2, 129.3, 129.1, 127.8, 125.0, 112.6, 110.6, 103.1, 51.6. IR (neat, cm⁻¹) 3264, 2956, 2924, 2850, 1675.



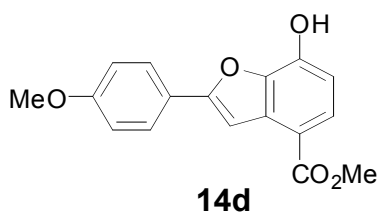
Methyl 7-hydroxy-2-(4-methylphenyl)benzofuran-4-carboxylate (14b). White solid. 74% yield. mp 188-189 °C. ¹H NMR (300 MHz, DMSO) δ 11.06 (s, 1H), 7.88 (d, *J* = 7.3 Hz, 2H), 7.76 (d, *J* = 8.1 Hz, 1H), 7.65 (s, 1H), 7.34 (d, *J* = 7.3 Hz, 2H), 6.83 (d, *J* = 8.1 Hz, 1H), 3.88 (s, 3H), 2.37 (s, 3H). ¹³C NMR (75 MHz, DMSO) δ 166.0, 156.8, 147.0, 142.6, 139.1, 131.4, 129.7, 127.6, 126.6, 125.0, 112.5, 110.4, 102.3, 51.5, 20.9. IR (neat, cm⁻¹) 3279, 3016, 2952, 1672.



Methyl 7-hydroxy-2-(4-nitrophenyl)benzofuran-4-carboxylate (14c). Bright yellow solid. 74% yield. mp 244-245 °C. ¹H NMR (400 MHz, DMSO) δ 11.30 (s, 1H), 8.36 (d, *J* = 8.9 Hz, 2H), 8.25 (d, *J* = 8.9, 2H), 8.03 (s, 1H), 7.81 (d, *J* = 8.4 Hz, 1H), 6.92 (d, *J* = 8.4 Hz, 1H), 3.90 (s, 3H). ¹³C NMR (100 MHz, DMSO) δ 165.8, 154.1, 147.4, 147.2, 143.5, 135.2, 130.6, 128.7, 128.2, 126.0, 124.4, 113.1, 106.9, 51.7. IR (neat, cm⁻¹) 3173, 2957, 1673.

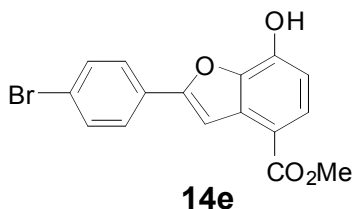


Methyl 7-hydroxy-2-(4-methoxyphenyl)benzofuran-4-carboxylate (14d). Tan solid. 29% yield. mp 180-182 °C. ¹H NMR (400 MHz, DMSO) δ 11.04 (s, 1H), 7.92 (d, *J* = 8.8 Hz, 2H), 7.75 (d, *J* = 8.4 Hz, 1H), 7.57 (s, 1H), 7.09 (d, *J* = 8.8 Hz, 2H), 6.81 (d, *J* = 8.4 Hz, 1H), 3.88 (s, 3H), 3.83 (s, 3H). ¹³C-NMR (100 MHz, DMSO) δ 166.1, 160.2, 156.9, 146.9, 142.5, 131.6, 128.7, 126.7, 122.0, 114.6, 112.3, 110.2, 101.3, 55.4, 51.5. IR (neat, cm⁻¹) 3273, 2951, 2838, 1675.

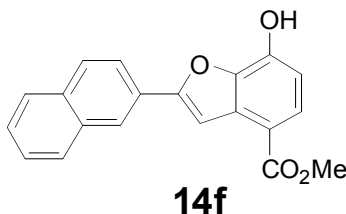


Methyl 2-(4-bromophenyl)-7-hydroxybenzofuran-4-carboxylate (14e). Tan solid. 54% yield. mp 245-247 °C. ¹H NMR (400 MHz, DMSO) δ 11.1 (s, 1H) 7.95 (d, *J* = 8.6 Hz, 2H), 7.80 (s, 1H), 7.78 (d, *J* = 8.4 Hz, 1H), 7.74 (d, *J* = 8.6 Hz, 2H), 6.86 (d, *J* = 8.4 Hz, 1H), 3.89 (s, 3H). ¹³C

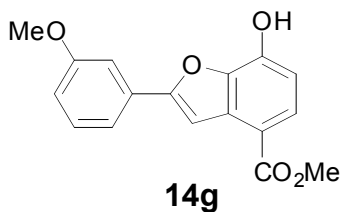
NMR (100 MHz, DMSO) δ 165.9, 155.4, 147.1, 142.9, 132.1, 131.1, 128.7, 128.5, 127.9, 127.0, 122.5, 112.7, 103.9, 51.6. IR (neat, cm^{-1}) 3284, 2952, 1677.



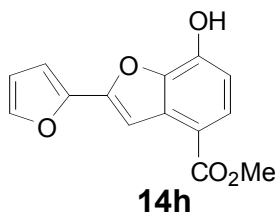
Methyl 7-hydroxy-2-(naphthalen-2-yl)benzofuran-4-carboxylate (14f). Grey solid. 79% yield. mp 217-219 °C. ^1H NMR (400 MHz, DMSO) δ 11.17 (s, 1H), 8.55 (s, 1H), 8.12 (dd, $J = 8.9, 1.6$ Hz, 1H), 8.07 (m, 2H), 7.97 (m, 1H), 7.88 (s, 1H), 7.80 (d, $J = 8.4$ Hz, 1H), 7.58 (m, 2H), 6.89 (d, $J = 8.4$ Hz, 1H), 3.91 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 166.0, 156.6, 147.1, 143.0, 133.1, 133.0, 131.3, 128.8, 128.5, 127.8, 127.8, 127.0, 127.0, 126.7, 124.0, 122.9, 112.6, 110.7, 103.8, 51.6. IR (neat, cm^{-1}) 3246, 2954, 1673.



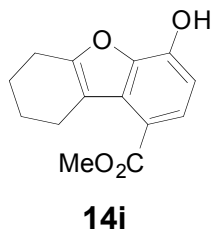
Methyl 7-hydroxy-2-(3-methoxyphenyl)benzofuran-4-carboxylate (14g). Tan solid. 33% yield. mp 175-177 °C. ^1H NMR (400 MHz, DMSO) δ 11.10 (s, 1H) 7.78 (d, $J = 8.4$ Hz, 1H), 7.76 (s, 1H), 7.57 (bd, $J = 8.0$ Hz, 1H), 7.52 (dd, $J = 2.5, 1.5$ Hz, 1H), 7.45 (dd, $J = 8.0, 8.0$ Hz, 1H), 7.03 (ddd, $J = 8.2, 2.5, 0.8$ Hz, 1H), 6.85 (d, $J = 8.4$ Hz, 1H), 3.89 (s, 3H), 3.87 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 166.0, 159.8, 156.4, 147.1, 142.8, 131.2, 130.7, 130.3, 128.7, 127.8, 115.3, 112.6, 110.6, 110.0, 103.4, 55.3, 51.6. IR (neat, cm^{-1}) 3219, 2959, 1672.

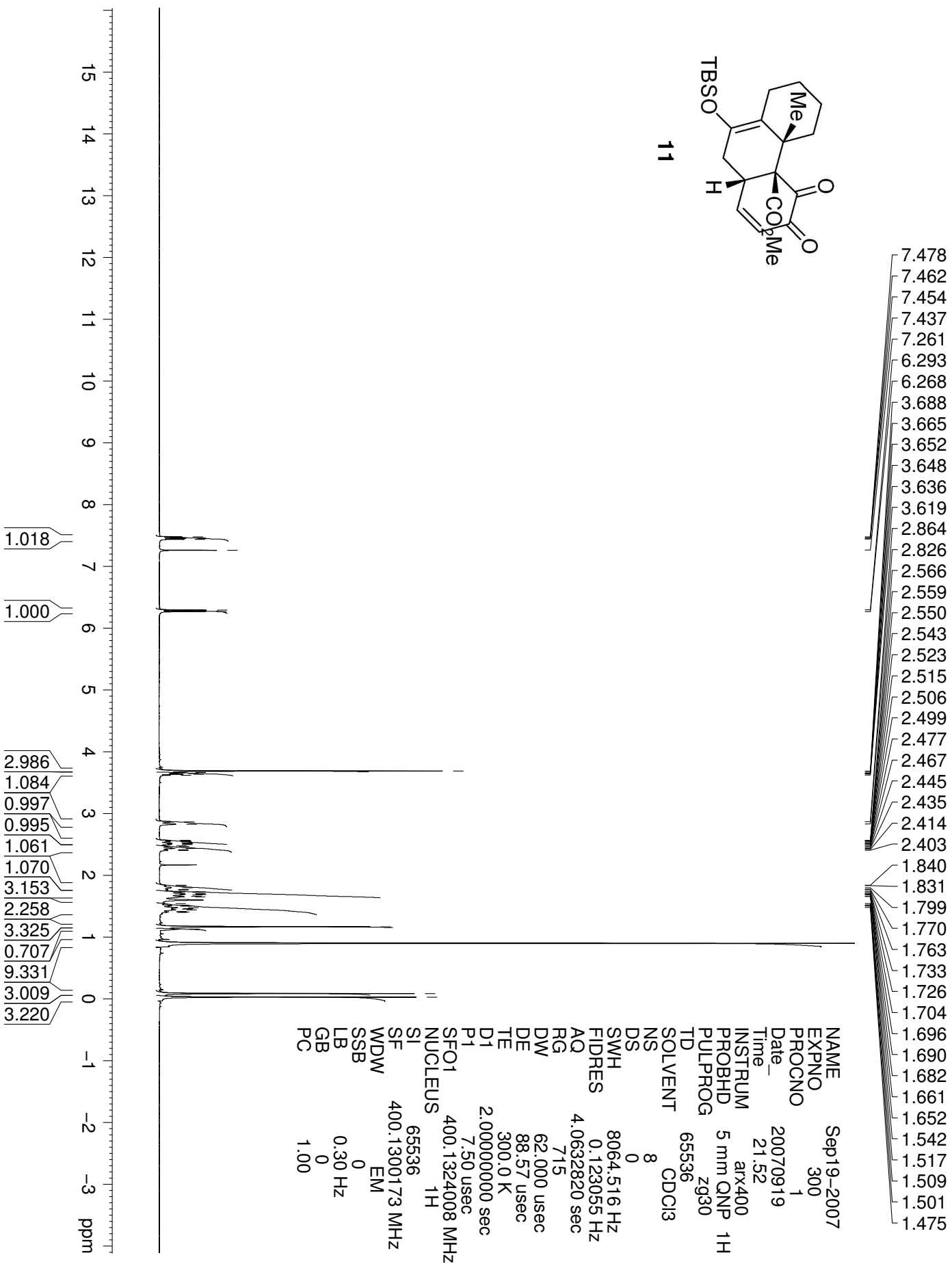
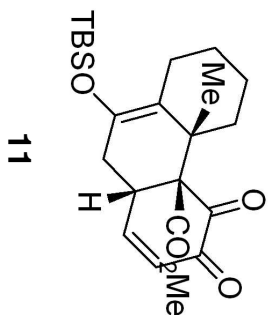


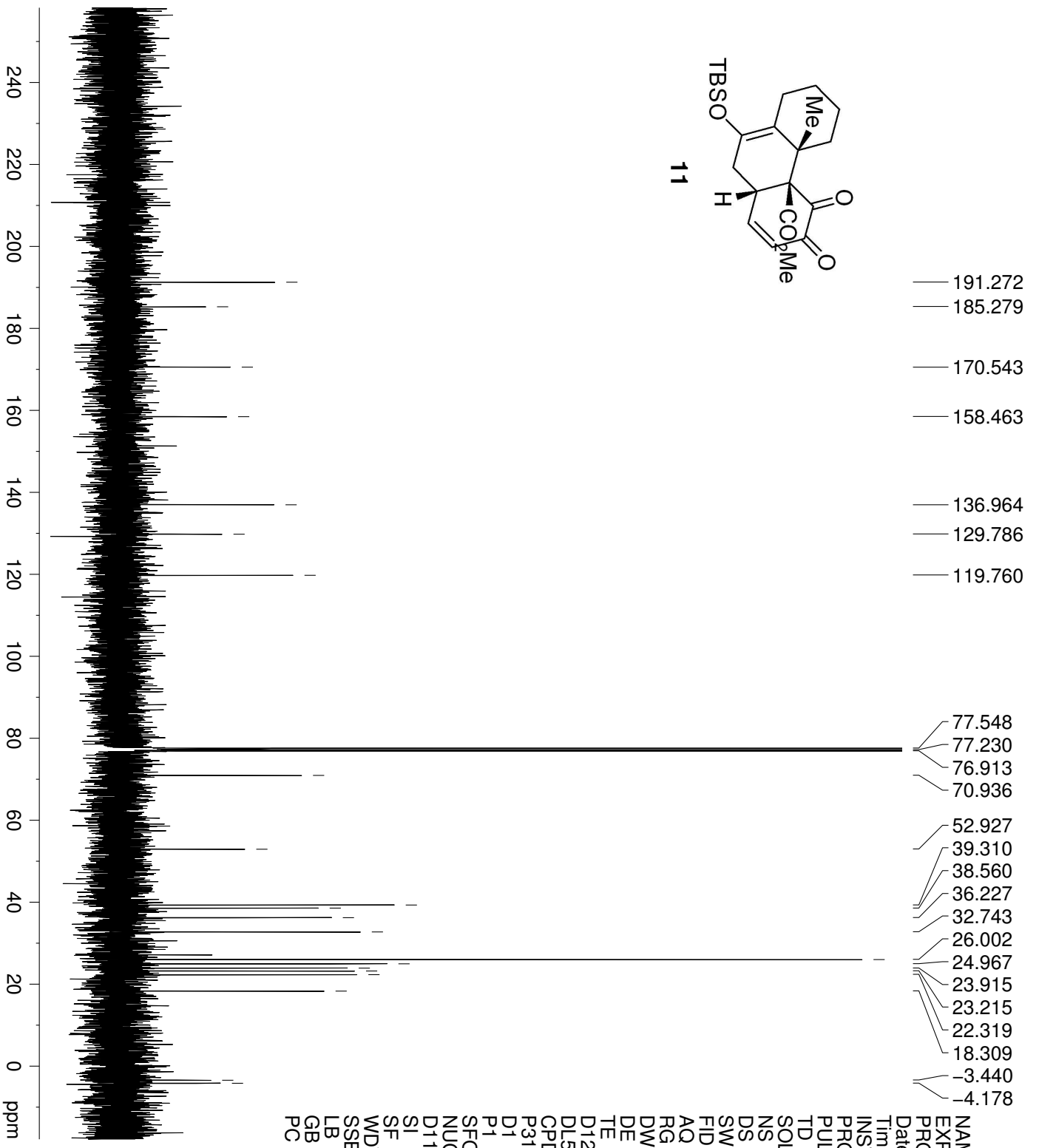
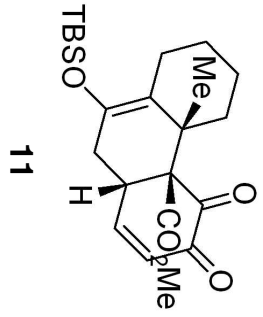
Methyl 2-(furan-2-yl)-7-hydroxybenzofuran-4-carboxylate (14h). White solid. 60% yield. mp 190-192 °C. ¹H NMR (400 MHz, DMSO) δ 11.17 (s, 1H), 7.92 (d, *J* = 1.4, 1H), 7.78 (d, *J* = 8.4 Hz, 1H), 7.43 (s, 1H), 7.08 (d, *J* = 3.2 Hz, 1H), 6.85 (d, *J* = 8.4 Hz, 1H), 6.72 (dd, *J* = 3.2, 1.8 Hz, 1H), 3.87 (s, 1H). ¹³C NMR (100 MHz, DMSO) δ 165.9, 148.7, 147.1, 144.8, 144.6, 142.5, 130.7, 128.7, 128.0, 112.4, 110.8, 109.4, 102.3, 51.6.



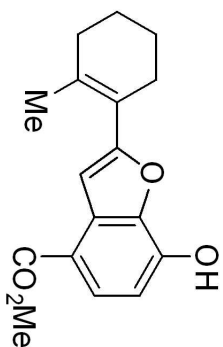
Methyl 4-hydroxy-6,7,8,9-tetrahydrodibenzo[*b,d*]furan-1-carboxylate (14i). Bright white solid. 34% yield. mp 138-140 °C. ¹H NMR (300 MHz, DMSO) δ 10.72 (s, 1H), 7.63 (d, *J* = 8.4 Hz, 1H), 6.72 (d, *J* = 8.4 Hz, 1H), 3.79 (s, 3H), 2.78-2.74 (m, 4H), 1.91-1.78 (m, 2H), 1.78-1.64 (m, 2H). ¹³C NMR (75 MHz, DMSO) δ 166.3, 155.8, 146.5, 142.5, 129.8, 127.2, 113.4, 113.3, 109.2, 51.3, 23.5, 23.4, 22.8, 21.8. IR (neat, cm⁻¹) 3273, 2951, 2838, 1675. IR (neat, cm⁻¹) 3274, 2951, 2921, 2854, 1678.



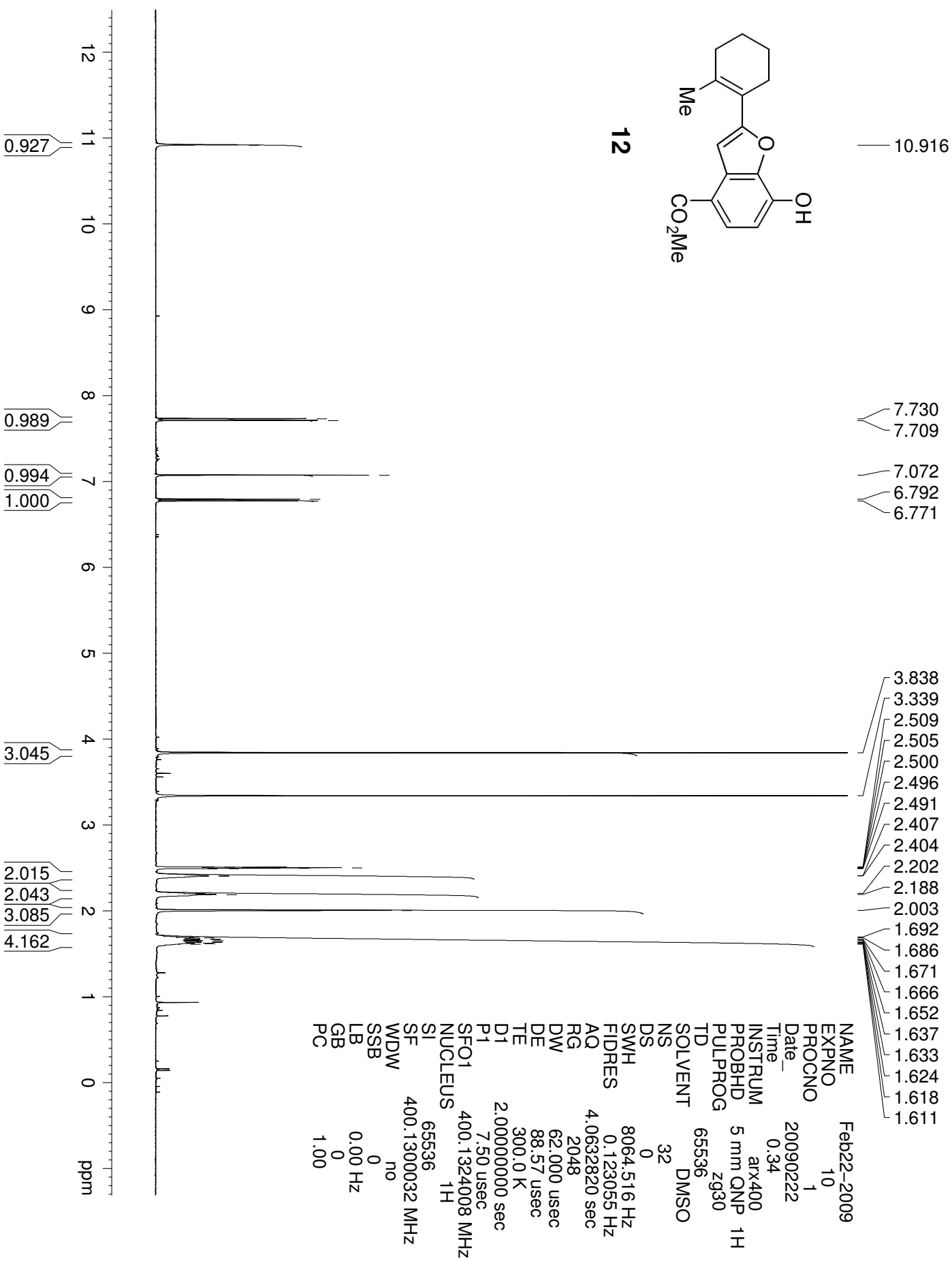


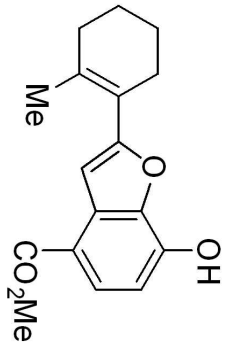


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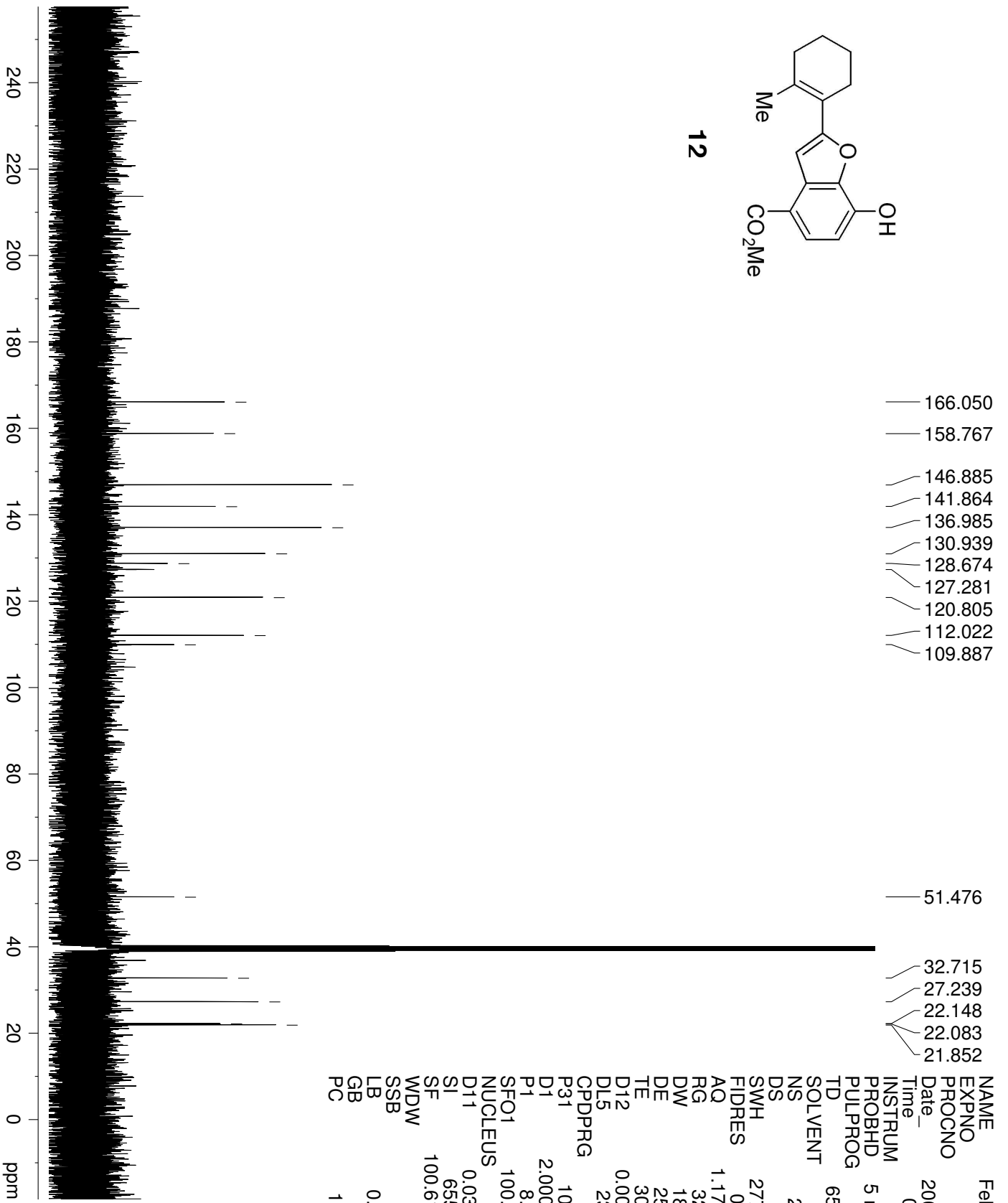


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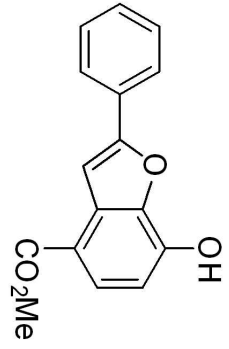


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14a



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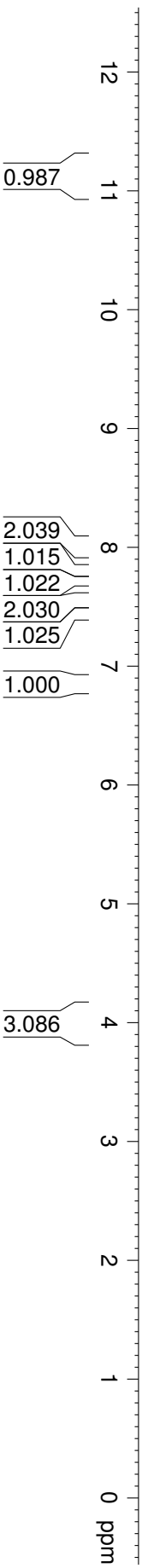
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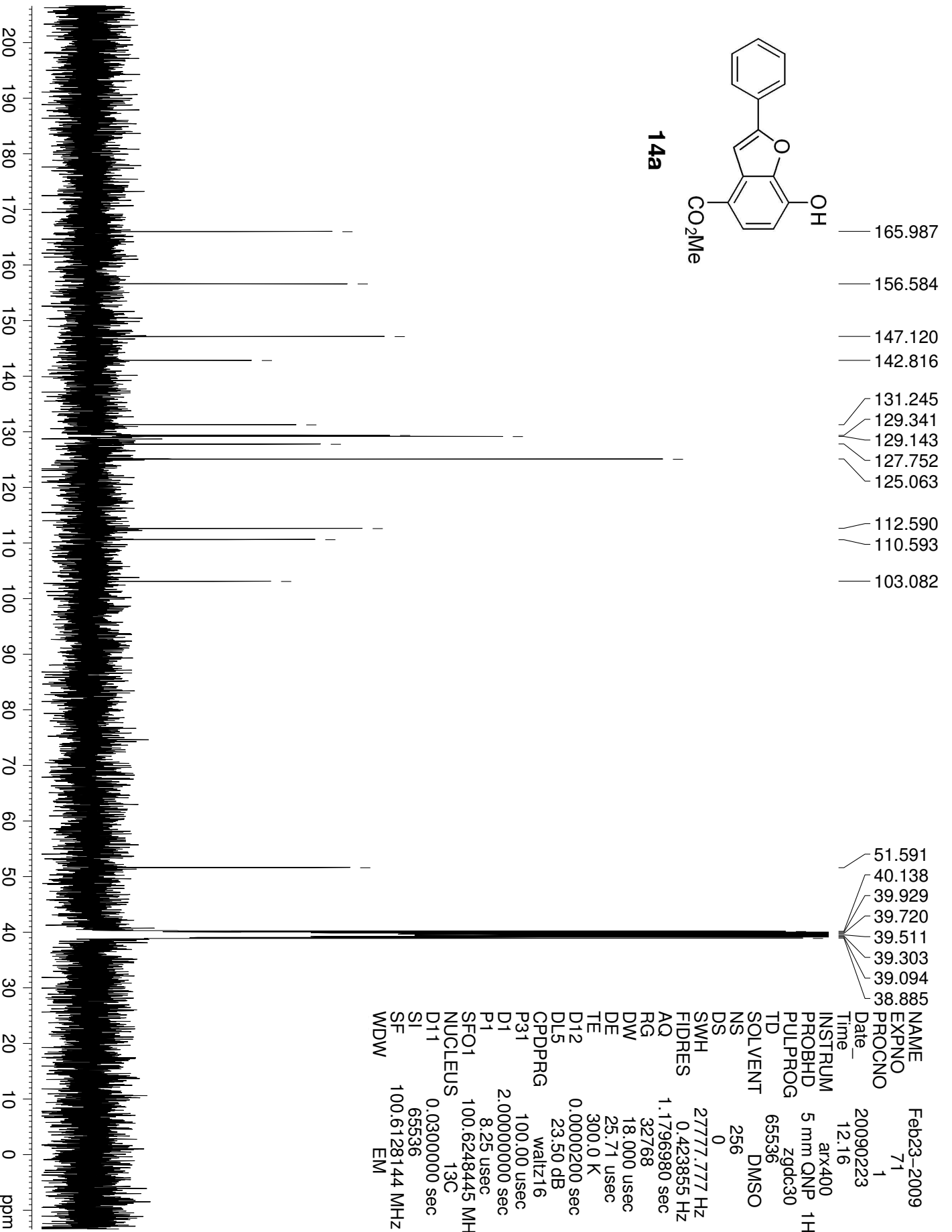
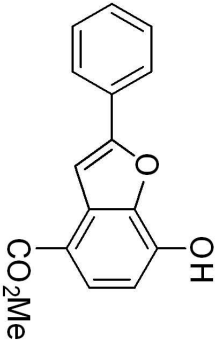
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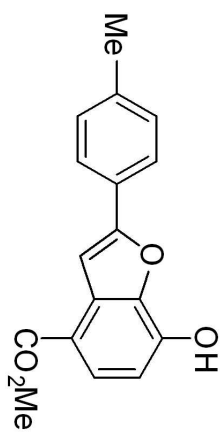
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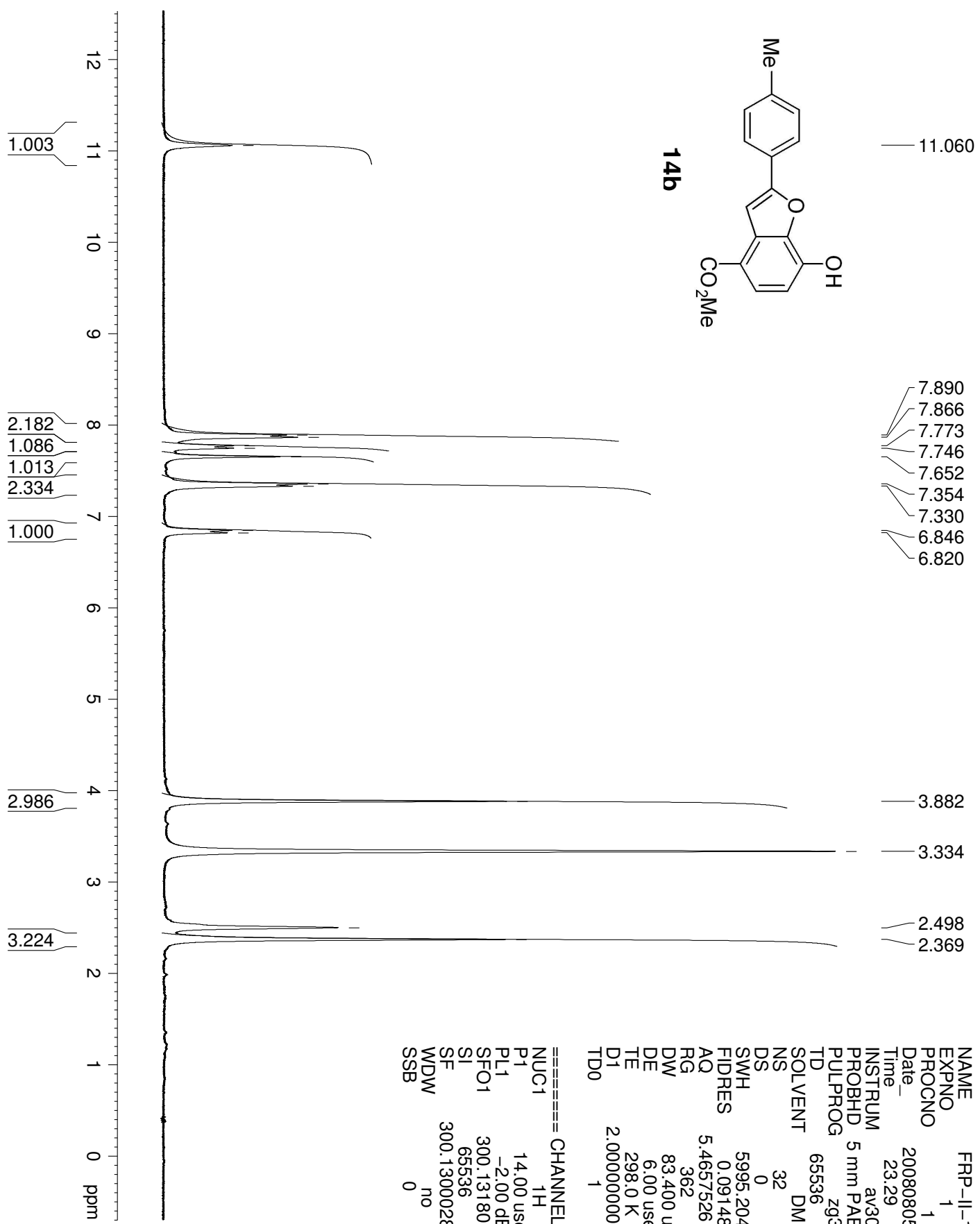
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 TE 300.0 K
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14b

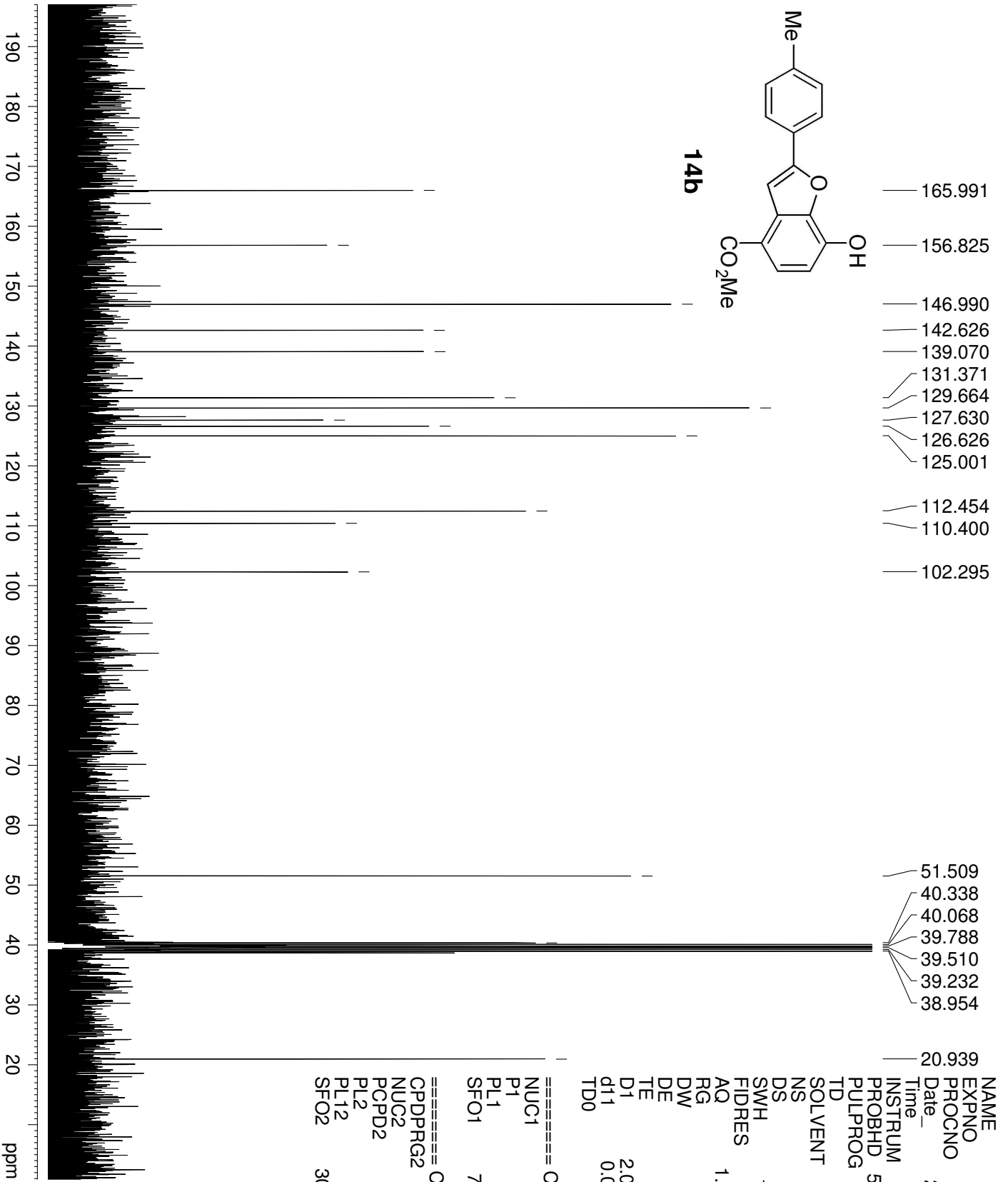


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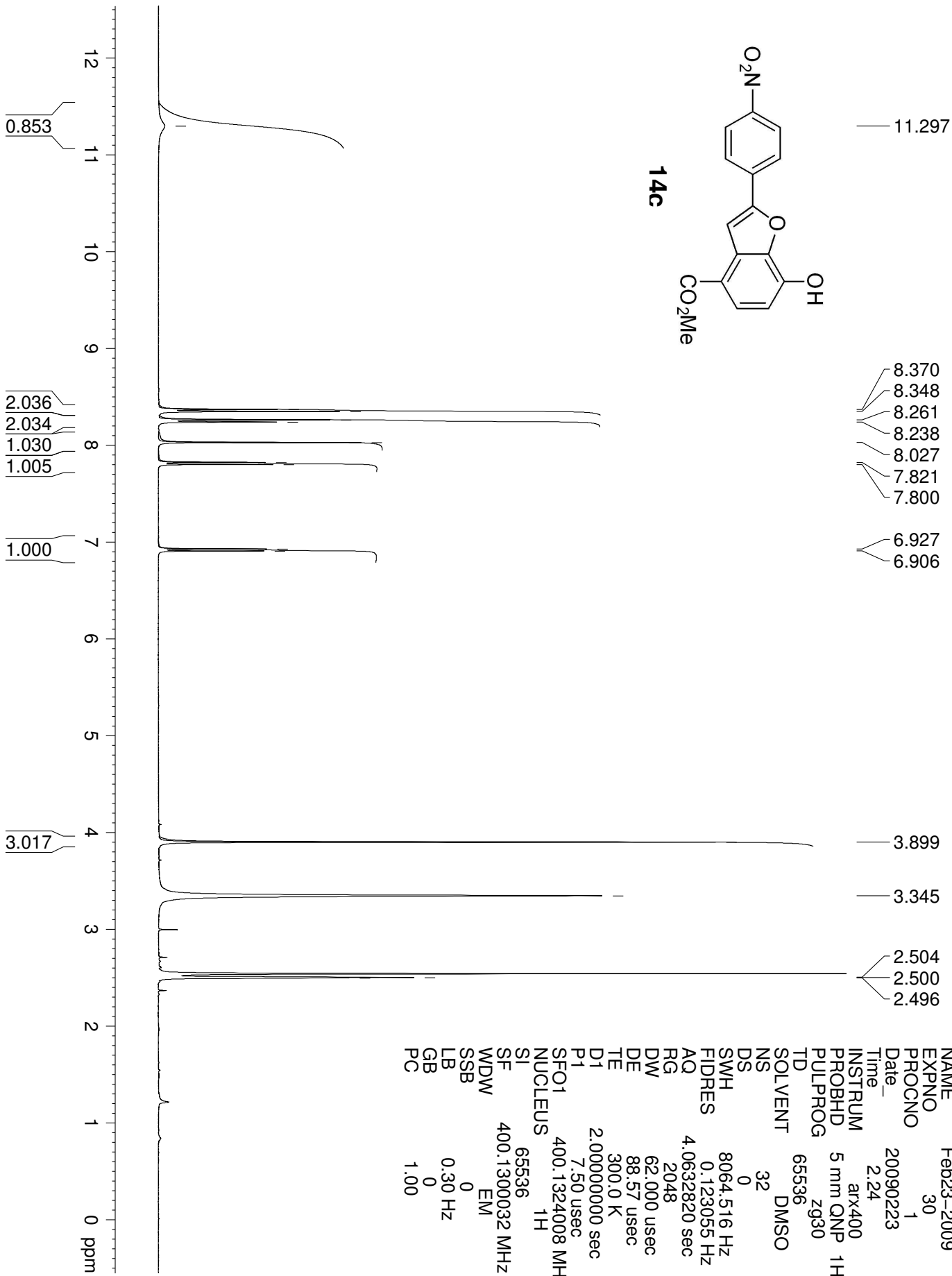
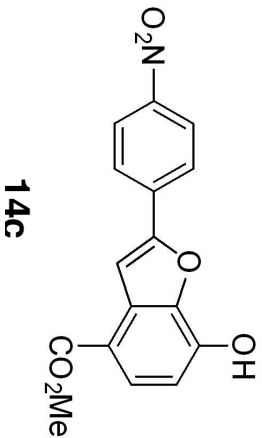
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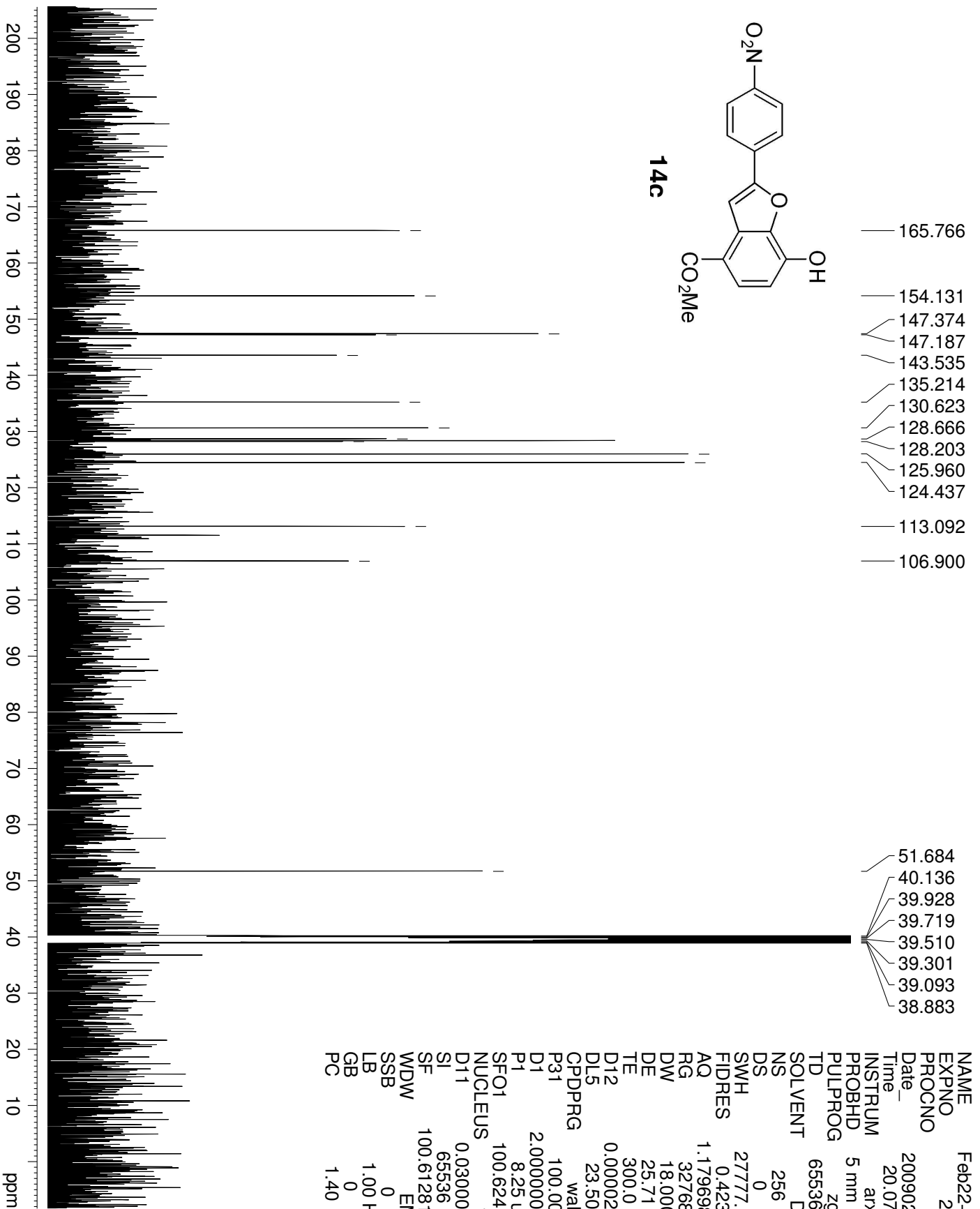
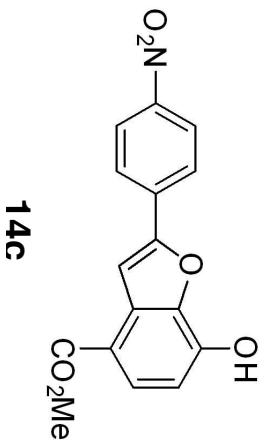
NAME FRP-11-12
 EXPNO 3
 PROCNO 1
 Date_ 20080806
 Time 0.11
 INSTRUM av300
 PROBHD 5 mm PABBO BB
 PULPROG zgdc30
 TD 65536
 SOLVENT CDCl3
 NS 256
 DS 0
 SWH 18832.393 Hz
 FIDRES 0.287360 Hz
 AQ 1.7400308 sec
 RG 9195.2
 DW 26.550 usec
 DE 6.00 usec
 TE 298.8 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 TD0 1

==== CHANNEL f1 ====
 NUC1 13C
 P1 6.50 usec
 PL1 -4.00 dB
 SFO1 75.4768051 MHz

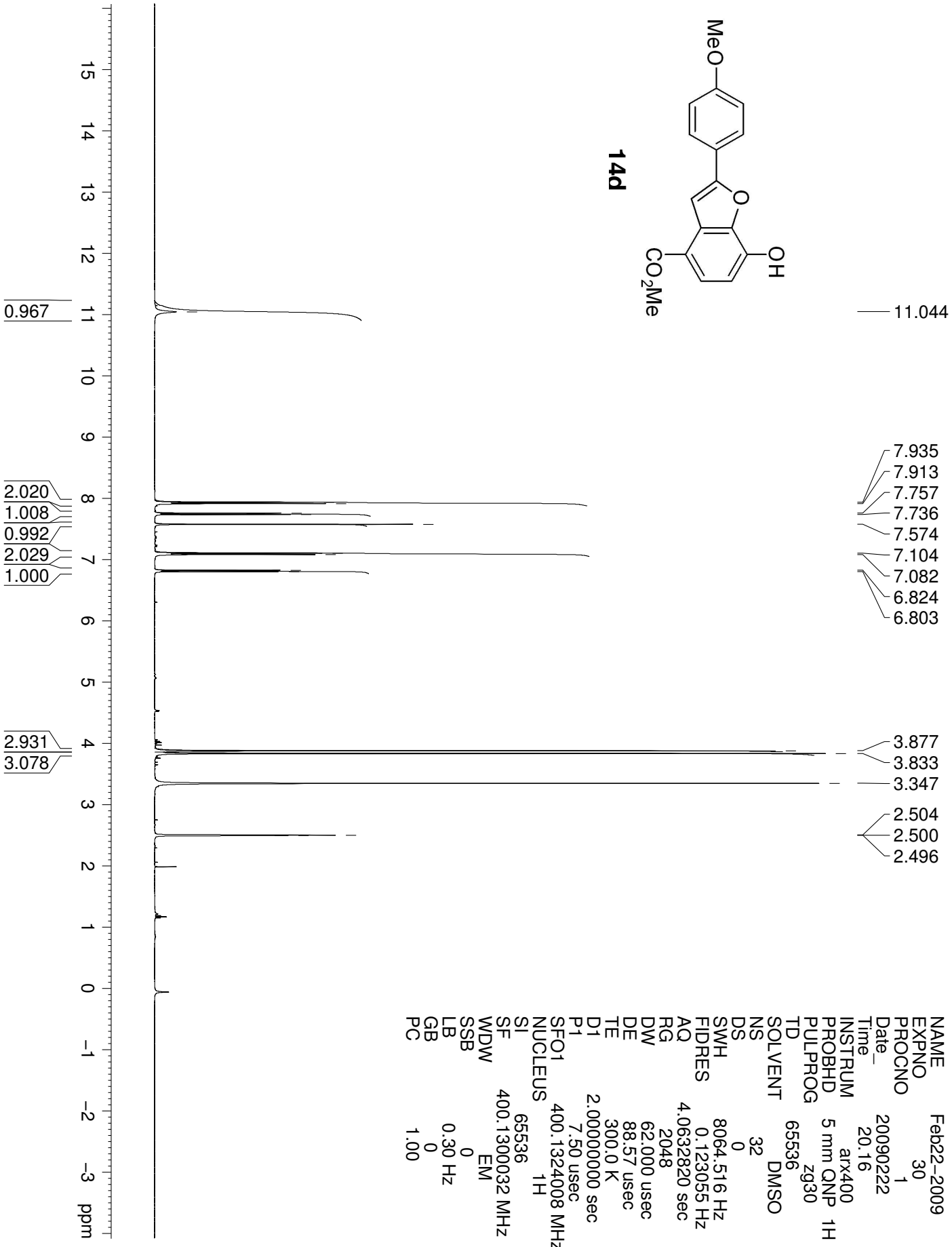
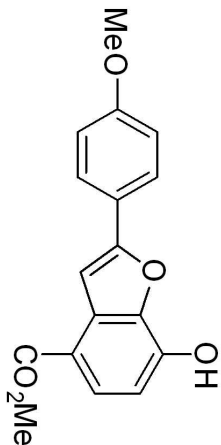
==== CHANNEL f2 ====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 13.75 dB
 SFO2 300.1318008 MHz



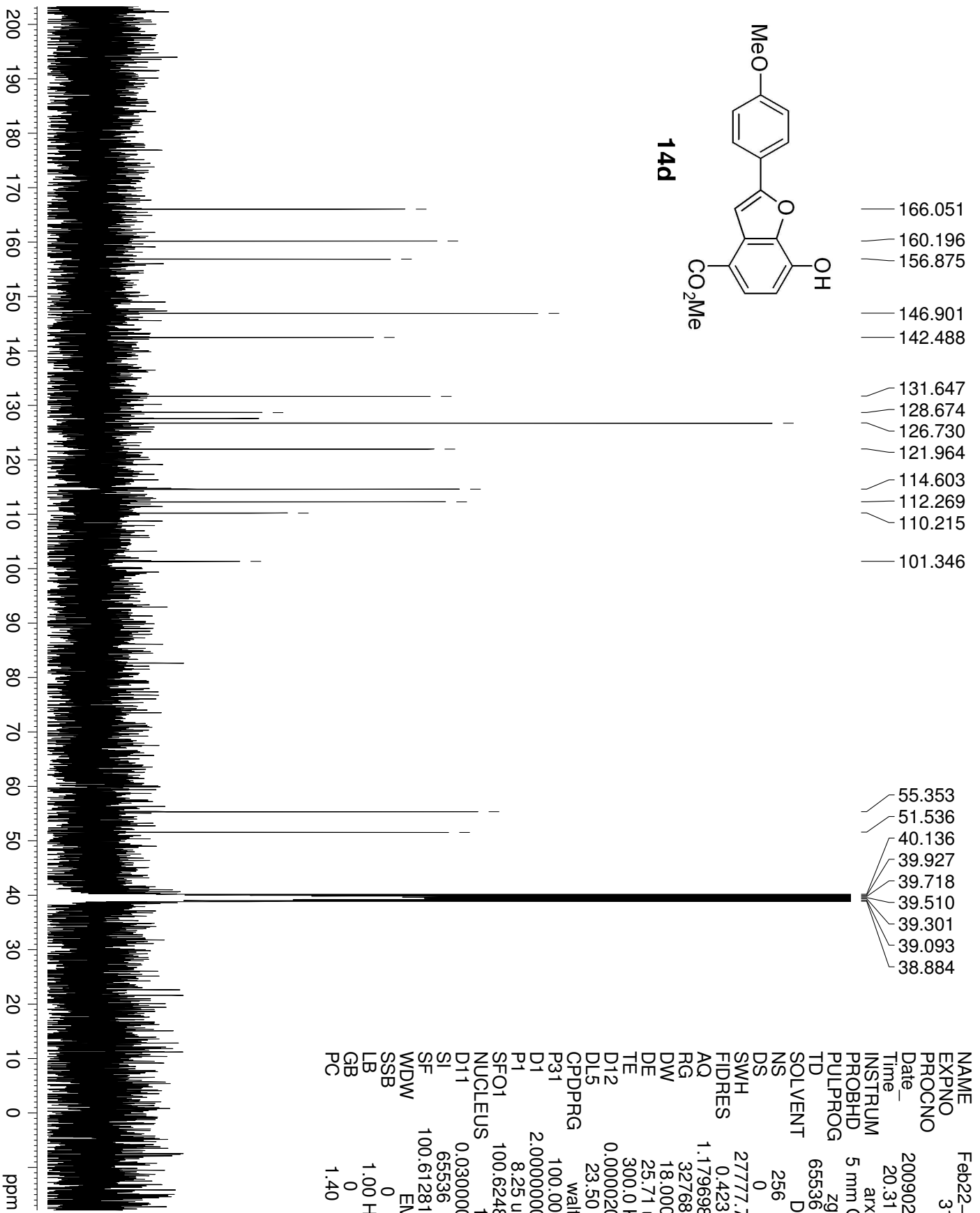
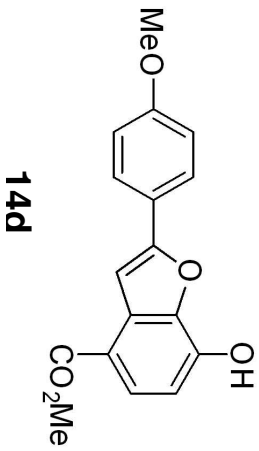
NAME Feb23-2009
 EXPNO 30
 PROCNO 1
 Date_ 20090223
 Time 2.24
 INSTRUM arx400
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 32
 DS 0
 SWH 8064.516 Hz
 FIDRES 0.123055 Hz
 AQ 4.0632820 sec
 RG 2048
 DW 62.000 usec
 DE 88.57 usec
 TE 300.0 K
 D1 2.00000000 sec
 P1 7.50 usec
 SFO1 400.1324008 MHz
 NUCLEUS 1H
 SI 65536
 SF 400.1300032 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



NAME Feb22-2009
 EXPNO 21
 PROCNO 1
 Date_ 20090222
 Time 20.07
 INSTRUM arx400
 PROBHD 5 mm QNP 1H
 PULPROG zgdc30
 TD 65536
 SOLVENT DMSO
 NS 256
 DS 0
 SWH 27777.777 Hz
 FIDRES 0.423855 Hz
 AQ 1.1796980 sec
 RG 32768
 DW 18.000 usec
 DE 25.71 usec
 TE 300.0 K
 D12 0.0000200 sec
 DL5 23.50 dB
 CPDPRG waltz16
 P31 100.00 usec
 D1 2.0000000 sec
 P1 8.25 usec
 SFO1 100.6248445 MHz
 NUCLEUS 13C
 D11 0.0300000 sec
 SI 65536
 SF 100.6128151 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

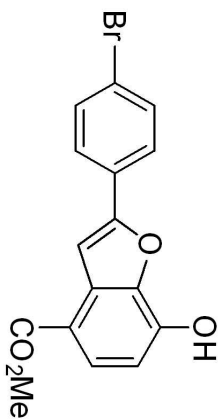


NAME Feb22-2009
 EXPNO 30
 PROCNO 1
 Date_ 20090222
 Time 20.16
 INSTRUM arx400
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 32
 DS 0
 SWH 8064.516 Hz
 FIDRES 0.123055 Hz
 AQC 4.0632820 sec
 RG 2048
 DW 62.000 usec
 DE 88.57 usec
 TE 300.0 K
 D1 2.00000000 sec
 P1 7.50 usec
 SFO1 400.1324008 MHz
 NUCLEUS 1H
 SI 65536
 SF 400.1300032 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

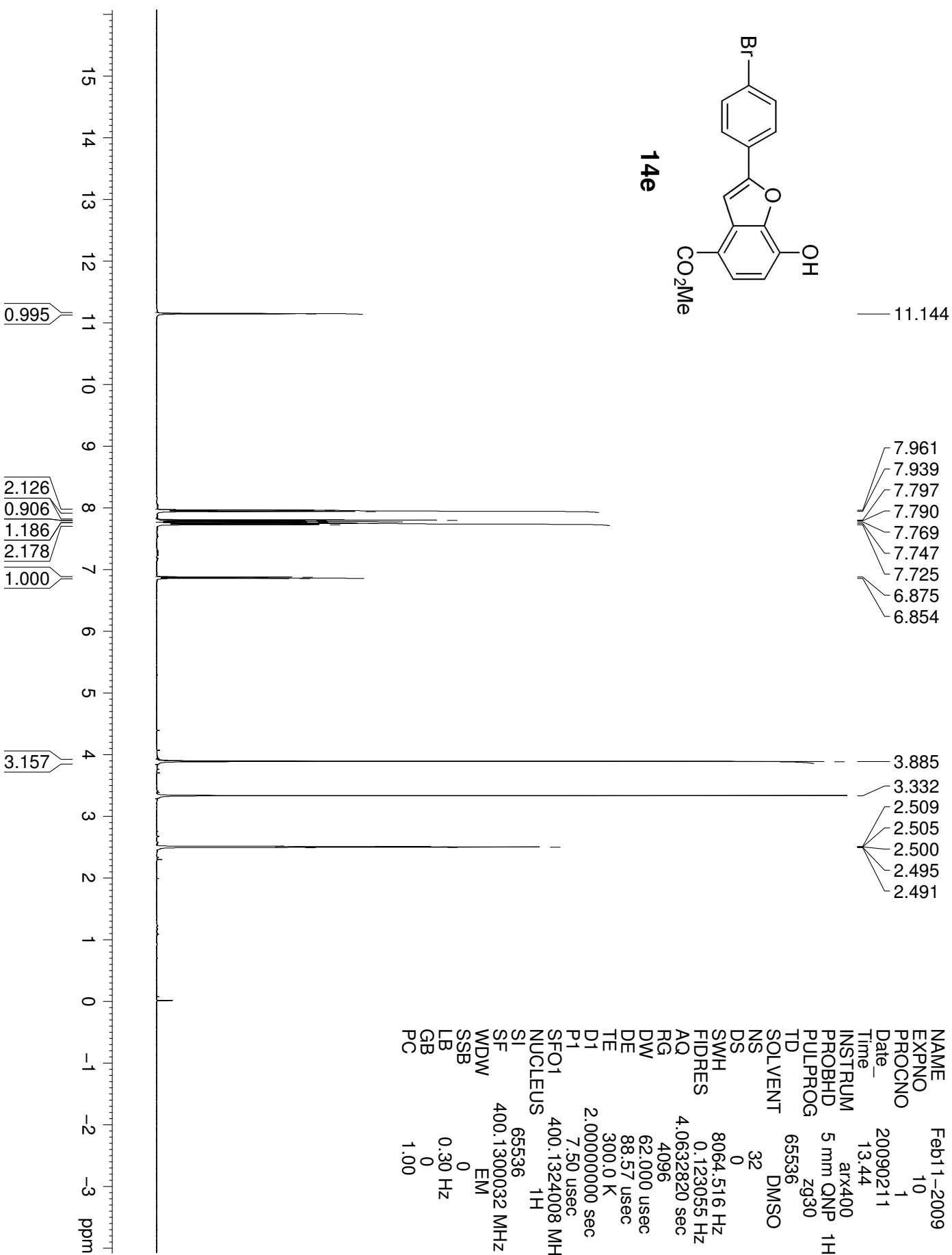


- 166.051
- 160.196
- 156.875
- 146.901
- 142.488
- 131.647
- 128.674
- 126.730
- 121.964
- 114.603
- 112.269
- 110.215
- 101.346
- 55.353
- 51.536
- 40.136
- 39.927
- 39.718
- 39.510
- 39.301
- 39.093
- 38.884

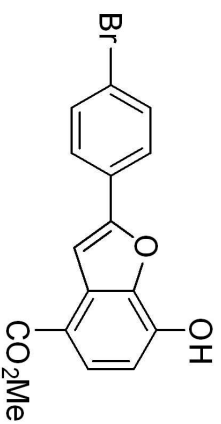
NAME Feb22-2009
 EXPNO 31
 PROCNO 1
 Date_ 20090222
 Time 20.31
 INSTRUM arx400
 PROBHD 5 mm QNP 1H
 PULPROG zgdc30
 TD 65536
 SOLVENT DMSO
 NS 256
 DS 0
 SWH 27777.777 Hz
 FIDRES 0.423855 Hz
 AQ 1.1796980 sec
 RG 32768
 DW 18.000 usec
 DE 25.71 usec
 TE 300.0 K
 D12 0.0000200 sec
 DL5 23.50 dB
 CPDPRG waltz16
 P31 100.00 usec
 D1 2.0000000 sec
 P1 8.25 usec
 SFO1 100.6248445 MHz
 NUCLEUS 13C
 D11 0.0300000 sec
 SI 65536
 SF 100.6128146 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



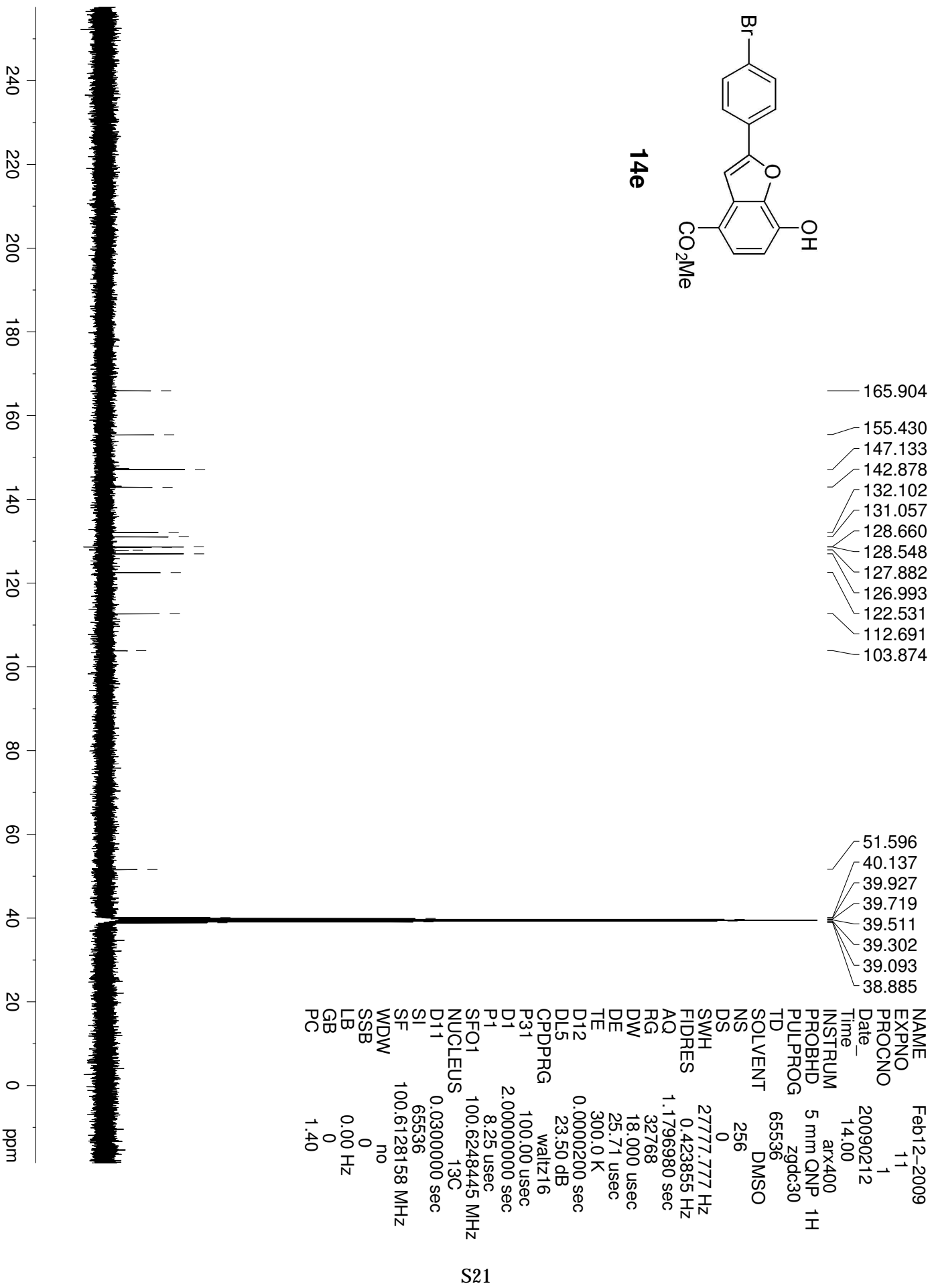
14e

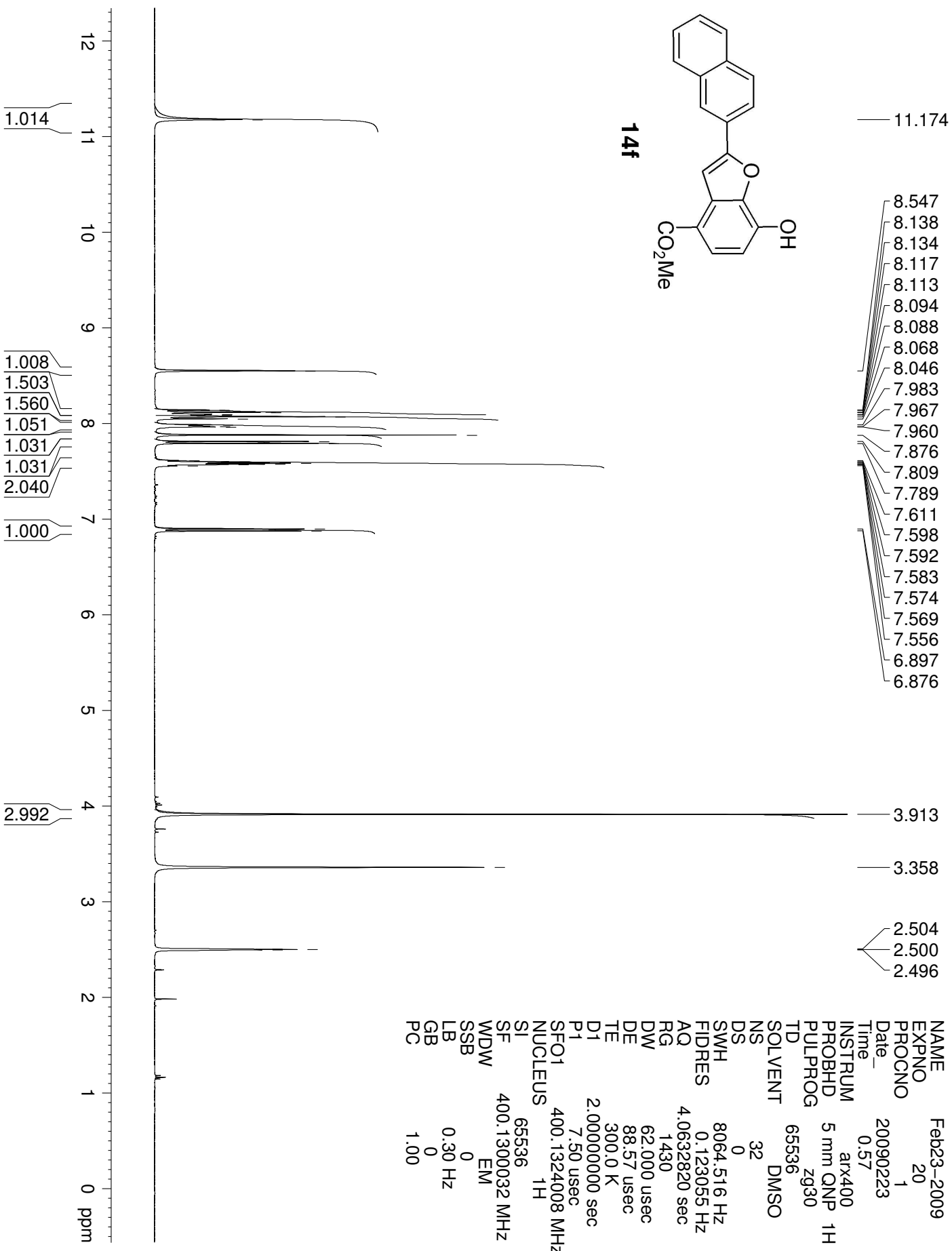
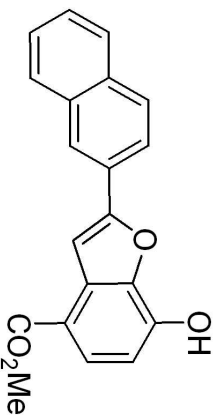


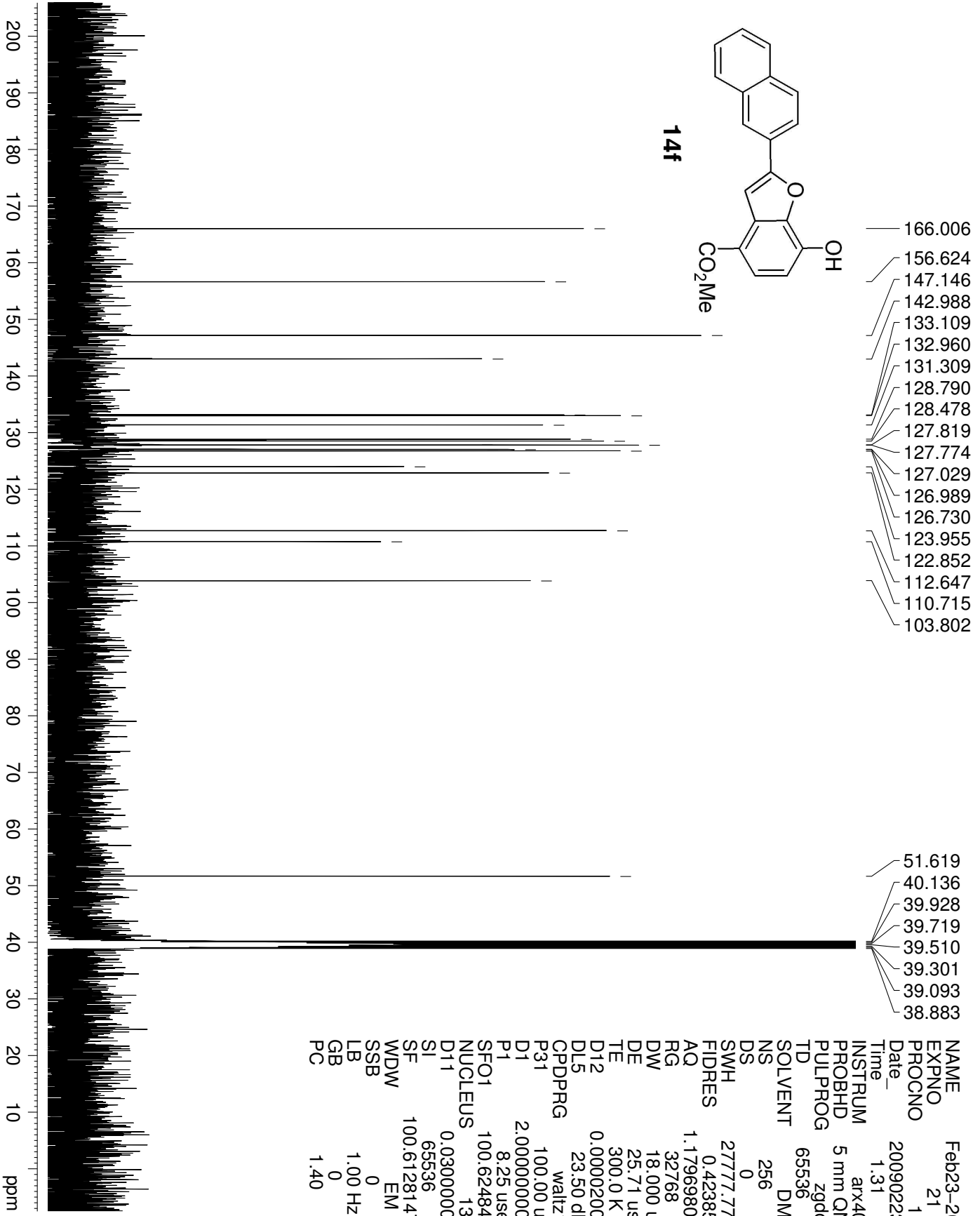
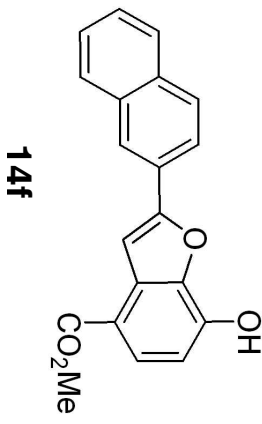
NAME Feb11-2009
 EXPNO 10
 PROCNO 1
 Date_ 20090211
 Time 13.44
 INSTRUM arx400
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 32
 DS 0
 SWH 8064.516 Hz
 FIDRES 0.123055 Hz
 AQ 4.0632820 sec
 RG 4096
 DW 62.000 usec
 DE 88.57 usec
 TE 300.0 K
 D1 2.00000000 sec
 P1 7.50 usec
 SFO1 400.1324008 MHz
 NUCLEUS 1H
 SI 65536
 SF 400.1300032 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



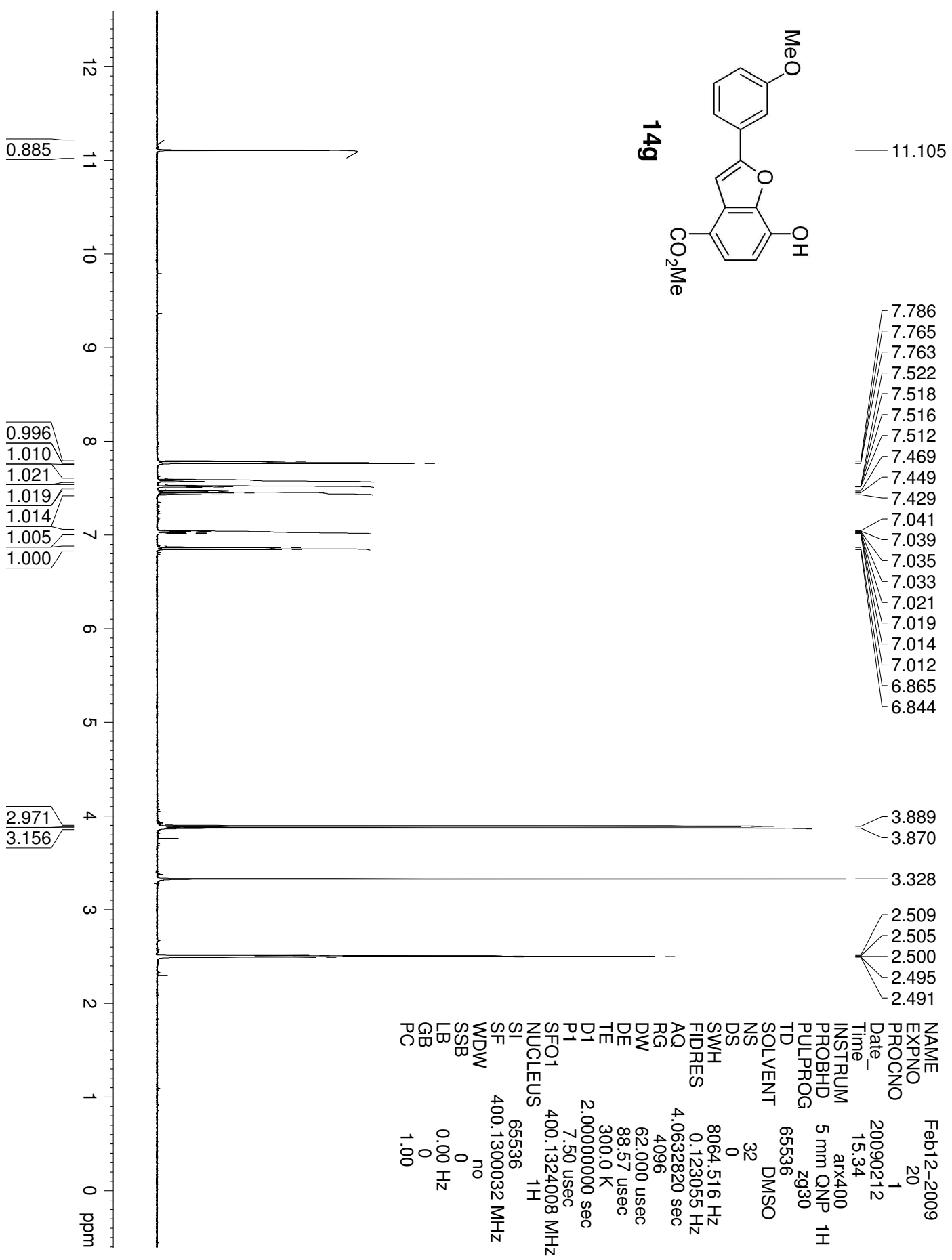
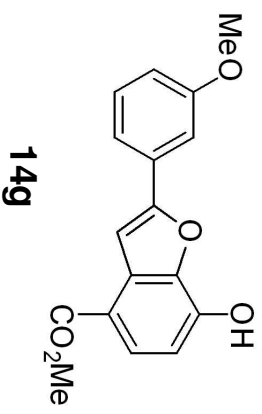
14e



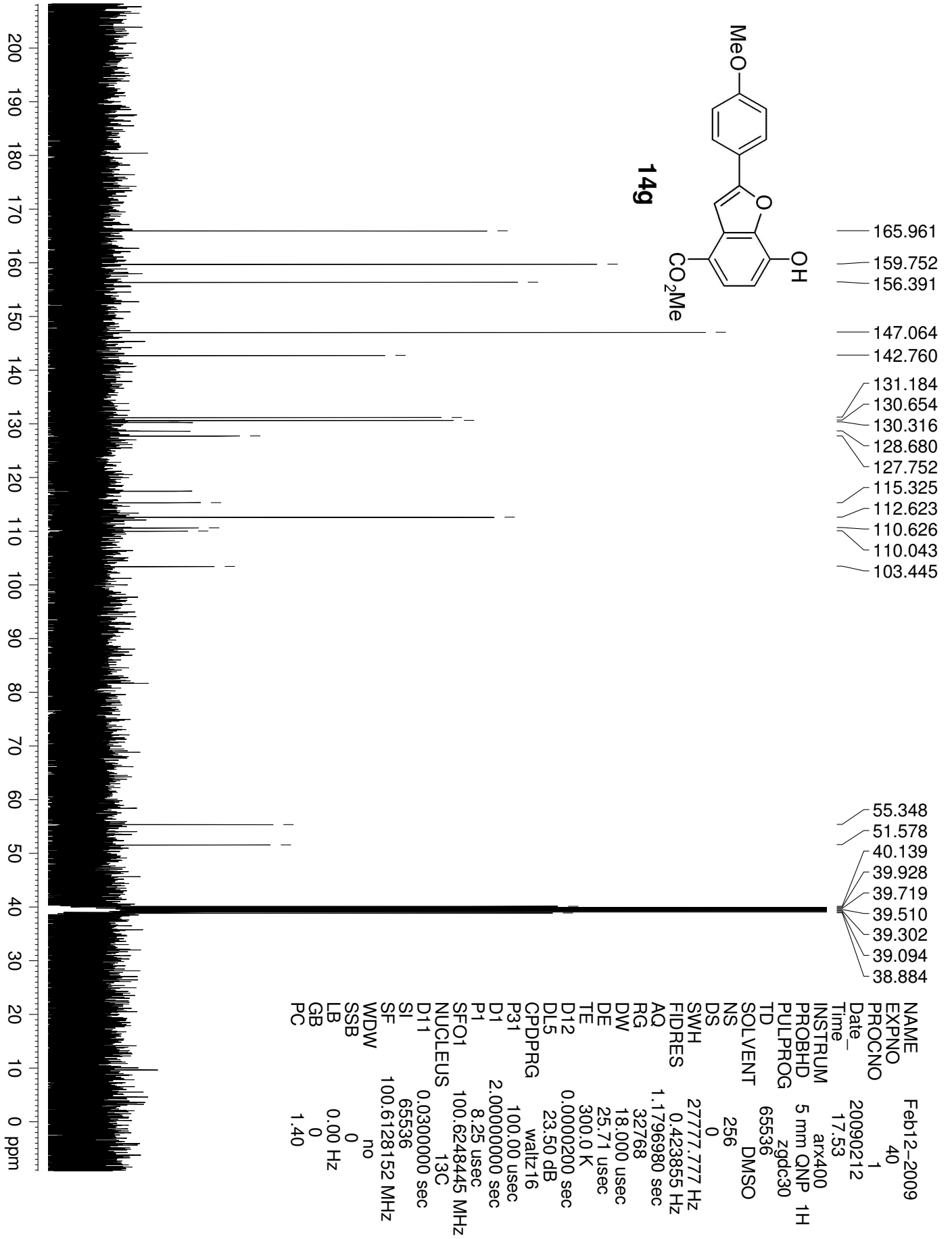


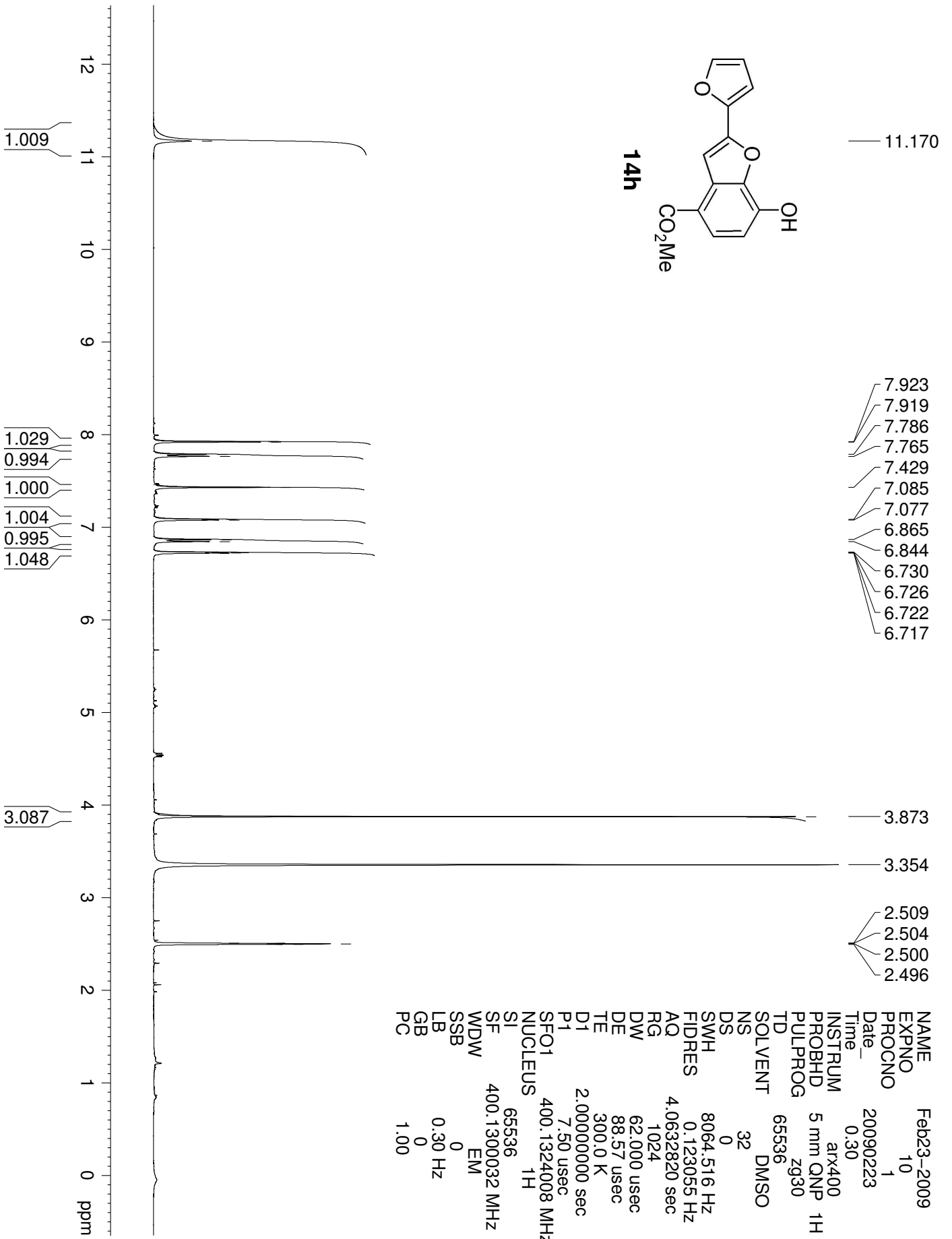
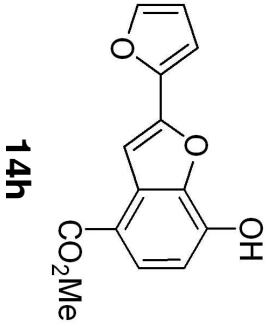


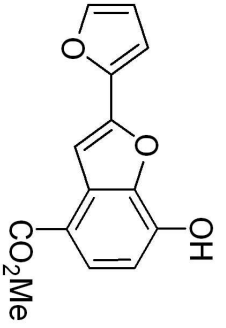
NAME Feb23-2009
 EXPNO 21
 PROCNO 1
 Date_ 20090223
 Time 1.31
 INSTRUM arx400
 PROBHD 5 mm QNP 1H
 PULPROG zgpgc30
 TD 65536
 SOLVENT DMSO
 NS 256
 DS 0
 SWH 27777.777 Hz
 FIDRES 0.423855 Hz
 AQ 1.1796980 sec
 RG 32768
 DW 18.000 usec
 DE 25.71 usec
 TE 300.0 K
 D12 0.0000200 sec
 DL5 23.50 dB
 CPDPRG waltz16
 P31 100.00 usec
 D1 2.00000000 sec
 P1 8.25 usec
 SFO1 100.6248445 MHz
 NUCLEUS 13C
 D11 0.03000000 sec
 SI 65536
 SF 100.6128147 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



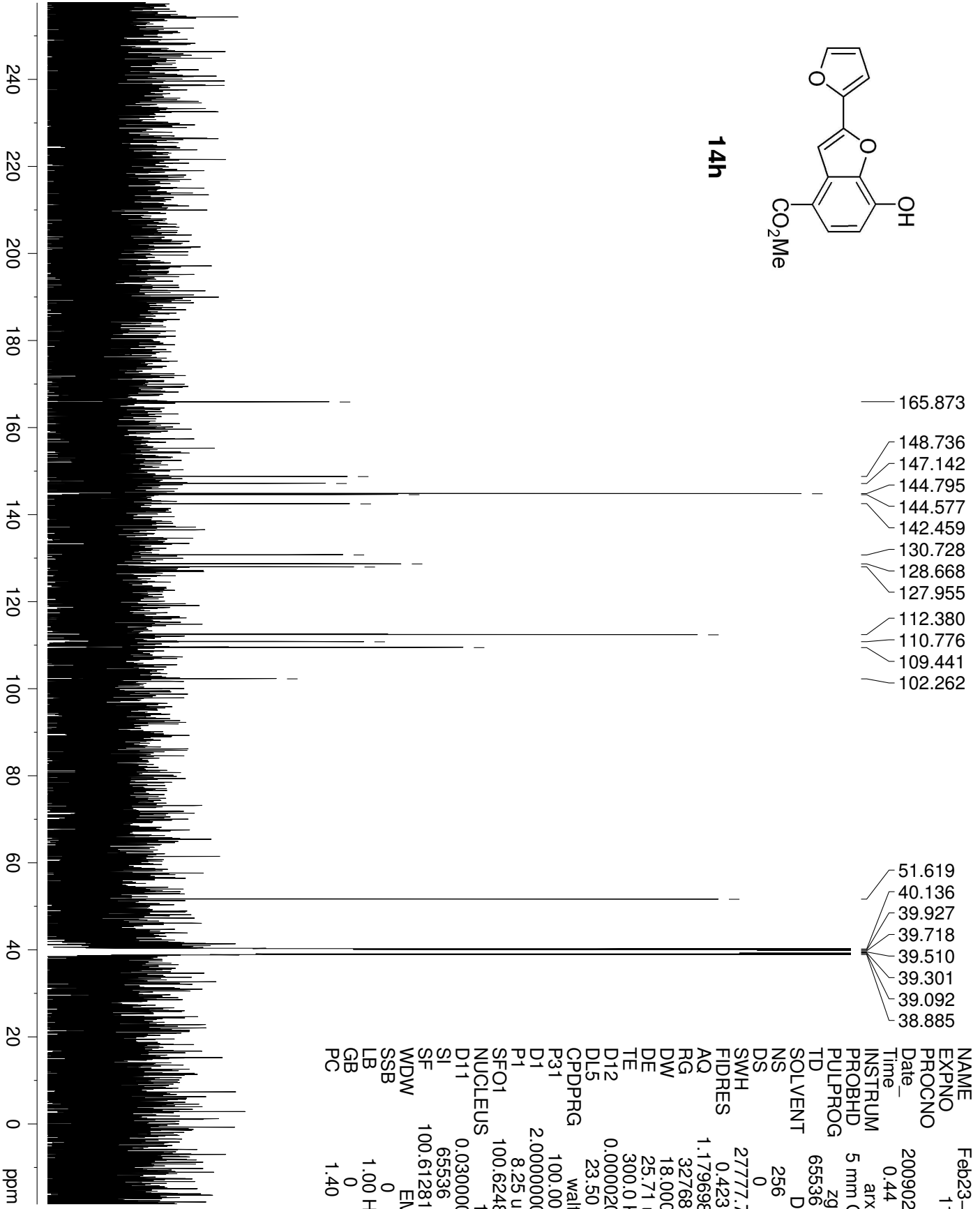
NAME Feb12-2009
 EXPNO 20
 PROCNO 1
 Date_ 20090212
 Time 15.34
 INSTRUM arx400
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 32
 DS 0
 SWH 8064.516 Hz
 FIDRES 0.123055 Hz
 AQC 4.0632820 sec
 RG 4096
 DW 62.000 usec
 DE 88.57 usec
 TE 300.0 K
 D1 2.00000000 sec
 P1 7.50 usec
 SFO1 400.1324008 MHz
 NUCLEUS 1H
 SI 65536
 SF 400.1300032 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00







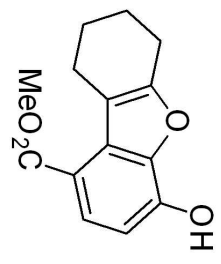
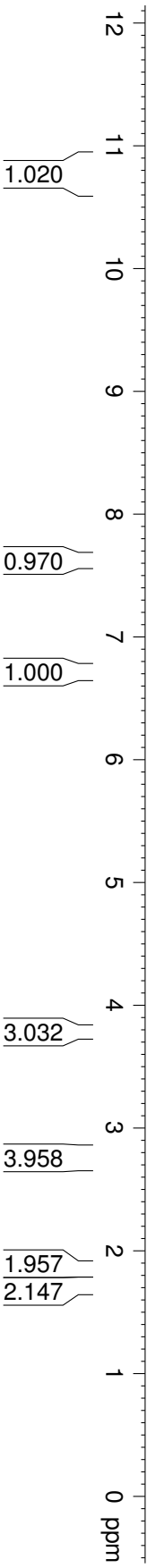
14h



- 165.873
- 148.736
- 147.142
- 144.795
- 144.577
- 142.459
- 130.728
- 128.668
- 127.955
- 112.380
- 110.776
- 109.441
- 102.262

- 51.619
- 40.136
- 39.927
- 39.718
- 39.510
- 39.301
- 39.092
- 38.885

NAME Feb23-2009
 EXPNO 11
 PROCNO 1
 Date_ 20090223
 Time 0.44
 INSTRUM arx400
 PROBHD 5 mm QNP 1H
 PULPROG zgdc30
 TD 65536
 SOLVENT DMSO
 NS 256
 DS 0
 SWH 27777.777 Hz
 FIDRES 0.423855 Hz
 AQ 1.1796980 sec
 RG 32768
 DW 18.000 usec
 DE 25.71 usec
 TE 300.0 K
 D12 0.0000200 sec
 DL5 23.50 dB
 CPDPRG waltz16
 P31 100.00 usec
 D1 2.0000000 sec
 P1 8.25 usec
 SFO1 100.6248445 MHz
 NUCLEUS 13C
 D11 0.0300000 sec
 SI 65536
 SF 100.6128151 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



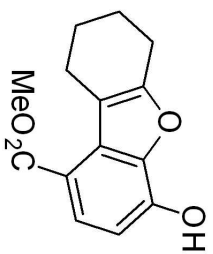
- 10.718
- 7.641
- 7.613
- 6.729
- 6.702
- 3.793
- 3.334
- 2.782
- 2.766
- 2.739
- 2.506
- 2.500
- 2.494
- 1.826
- 1.729

```

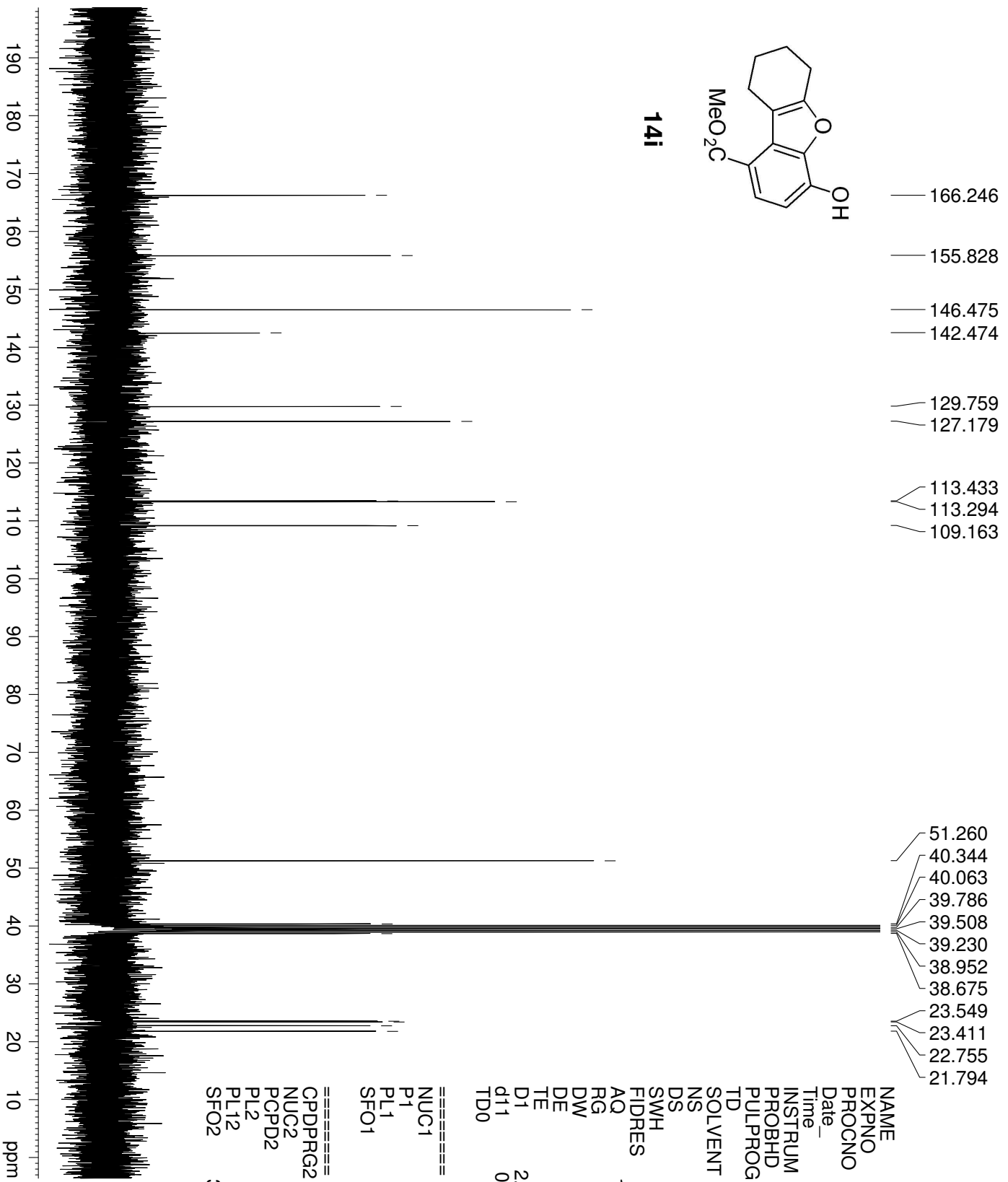
NAME FRP-II-11
EXPNO 1
PROCNO 1
Date_ 20080804
Time 21.44
INSTRUM av300
PROBHD 5 mm PABBO BB
PULPROG zg30
TD 65536
SOLVENT CDCI3
NS 32
DS 0
SWH 5995.204 Hz
FIDRES 0.091480 Hz
AQ 5.4657526 sec
RG 256
DW 83.400 usec
DE 6.00 usec
TE 297.9 K
D1 2.00000000 sec
TD0 1
  
```

```

===== CHANNEL f1 =====
NUC1 1H
P1 14.00 usec
PL1 -2.00 dB
SFO1 300.1318008 MHz
SI 65536
SF 300.1300030 MHz
WDW no
SSB 0
  
```



14i



- 166.246
- 155.828
- 146.475
- 142.474
- 129.759
- 127.179
- 113.433
- 113.294
- 109.163
- 51.260
- 40.344
- 40.063
- 39.786
- 39.508
- 39.230
- 38.952
- 38.675
- 23.549
- 23.411
- 22.755
- 21.794

```

NAME FRP-II-11
EXPNO 3
PROCNO 1
Date_ 20080805
Time 14.07
INSTRUM av300
PROBHD 5 mm PABBO BB
PULPROG zgdc30
TD 65536
SOLVENT DMSO
NS 256
DS 0
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 6502
DW 26.550 usec
DE 6.00 usec
TE 298.9 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 6.50 usec
PL1 -4.00 dB
SFO1 75.4768051 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 13.75 dB
SFO2 300.1318008 MHz
  
```

References:

- 1) Dallacker, F.; Thiemann, E.; Uddrich, P. *Chem. Ber.* **1971**, *104*, 2347.
- 2) Jung, M. E.; Ho, D.; Chu, H. V. *Org. Lett.* **2005**, *7*, 1649.
- 3) Rama Rao, A. V.; Deshmukh, M. N.; Sivadasan, L. *Chem. & Ind.* **1981**, 164.