CONFORMATIONAL PREFERENCES OF ZAMPANOLIDE AND DACTYLOLIDE

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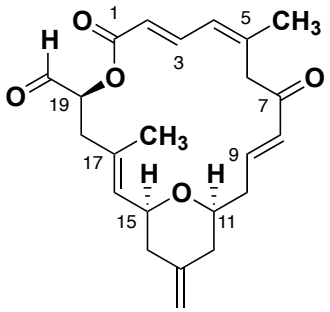
Supporting Information: Modeling and Spectral Data

NMR Data



(–)-dactylolide **3**

^{1}H and ^{13}C NMR Signals of Natural[1] and Synthetic (−)-dactylolide in $CDCl_3$

$^{1}H/^{13}C$ No.	**Natural* ^{13}C, 150 MHz, ppm**	**Synthetic ^{13}C, 150 MHz, ppm**	**Natural* ^{1}H, 600 MHz, (Hz)**	**Synthetic ^{1}H, 500 MHz, (Hz)**
1	166.0	166.5	-	-
2	120.0	119.9	5.96 d (15.1)	5.96 d (15.0)
3	140.7	140.6	7.62 dd (15.1, 11.6)	7.63 dd (15.0, 11.5)
4	125.7	125.7	6.15 d (11.6)	6.15 d (11.5)
5	144.4	144.2	-	-
5a	23.6	24.3	1.85 s	1.86 s
6	44.8	45.0	3.94 d (14.5) 3.23 d (14.5)	3.95 d (14.5) 3.23 d (14.5)
7	197.0	197.6	-	-
8	131.3	131.6	6.0 d (16.0)	6.0 d (16.5)
9	146.8	146.2	6.84 ddd (5.9, 8.5, 16.0)	6.84 ddd (5.5, 8.7, 16.5)
10	40.1	39.9	2.35 m 2.28 m	2.36 m 2.28 m
11	76.7	76.6	3.31 dddd (2.6, 2.6, 9.6, 12.3)	3.32 dddd (2.3, 2.3, 10.5, 11.5)
12	40.8	41.0	2.16 m 1.95 t (12.3)	2.17 dd (1.6, 13.0) 1.96 m
13	143.4	143.6	-	-
13a	109.0	109.5	4.73 bs	4.74 d (1.2) 4.75 d (1.2)
14	40.4	40.6	2.10 m 1.95 m	2.11 dd (2.1, 13.0) 1.96 m
15	76.1	75.9	3.96 ddd (2.6,	3.96 ddd (2.7, 8.0,

[1] Cutignano, A.; Bruno, I.; Bifulco, G.; Casapullo, A.; Cécile, D.; Gomez-Paloma, L.; Riccio, R. *Eur. J. Org. Chem*. **2001**, 775-778.

			8.3, 10.9)	11.5)
16	130.8	130.7	5.24 d (8.3)	5.24 d (8.0)
17	131.3	131.1	-	-
17a	16.2	16.2	1.70 s	1.71 s
18	39.6	39.9	2.53 brd (14.0) 2.31 brd (14.0)	2.54 brd (14.0) 2.31 dd (11.0, 14.0)
19	75.5	75.5	5.31 dd (11.2, 2.2)	5.31 dd (11.0, 2.5)
19a	199.7	199.3	9.66 s	9.66 s

* Natural dactylolide is reported to be (+)-dactylolide

^{1}H NMR signals and *J*-coupling constants of synthetic (−)-dactylolide and natural (−)-zampanolide in DMSO-$_{d6}$[2]

^{1}H/^{13}C No.	**Synthetic (−)-dactylolide ^{1}H (600 MHz), Hz**	**Natural (−)-zampanolide ^{1}H (500 MHz), Hz**
1	-	-
2	5.97 d (15.1)	5.93 d (15.0)
3	7.42 dd (15.1, 11.6)	7.52 dd (15.0, 11.6)
4	6.25 d (11.6)	6.20 d (11.6)
5	-	-
5a	1.82 s	1.75 s
6	3.30 d (15.5) 3.86 d (15.5)	3.01 d (14.4) 4.12 d (14.4)
7	-	-
8	6.02 d (16.1)	5.95 d (15.3)
9	6.79 dt (16.1, 7.2, 7.2)	6.74 ddd (15.3, 8.6, 5.5)
10	2.40 dddd (14.5, 7.5, 2.6, 1.2) 2.26 dddd (14.5, 10.0, 6.9, 1.5)	2.30 m
11	3.34 dddd (11.4, 10.6, 2.6, 2.0)	3.27 m
12	1.85 m 2.21 dd (13.0, 1.6)	1.84 m 2.17 brd (12.8)
13	-	-
13a	4.73 d (1.7) 4.74 d (1.7)	4.73 s
14	1.86 m 2.11 dd (13.1, 2.1)	1.88 m 2.07 m
15	3.93 ddd (10.6, 7.9, 2.4)	3.87 ddd (11.3, 7.6, 2.7)
16	5.20 d (7.9)	5.10 d (7.6)
17	-	-
17a	1.65 d (1.1)	1.61 s

[2] Tanaka, J.; Higa, T.; *Tetrahedron Lett.* **1996**, *37*, 5535-5538.

18	2.30 dd (14.1, 11.2) 2.53 d (14.5)	2.09 m 2.33 m
19	5.29 dd (11.2, 2.7)	4.96 ddd (10.0, 6.0, 1.8)
19a	9.56 s	-

ROESY-derived interproton distances used in DISCON calculations

H1	**H2**	**Peak Volume**	**Interproton distance (Å)**
10proR	10proS	1.00	1.78
16	14proR	0.11	2.65
18proR	16	0.27	2.32
10proR	8	0.06	2.94
3	9	0.07	2.81
9	11	0.09	2.79
2	4	0.14	2.65
3	6proS	0.27	2.30
3	6proR	0.13	2.56
6proS	9	0.12	2.69
17Me	15	0.29	2.22
17Me	19	0.18	2.42

^{1}H NMR coupling constants used in DISCON calculations

H1	**H2**	***J* (Hz)**
10proS	9	7.5
10proS	11	2.5
19	18proR	11.2
19	18proS	2.7
10proR	11	10.0
10proR	9	6.9

Conformational Searches

Conformer libraries were generated using the Batchmin package and the MM3, MMFF, and OPLS2005 force fields as implemented in MacroModel 9.0. Solvation was simulated using the GB/SA continuum model for water. Conformational searches were performed with the MCMM torsional sampling, typically using 50,000 Monte Carlo steps per run. Structures within a 20 kJ/mol window were saved and minimized for complete convergence using the Polak-Ribiere conjugate gradient method (5000 steps, convergence threshold of 0.05). The three libraries generated were combined and the redundant conformers were eliminated based on comparison of maximum atom deviation (threshold of 0.5Å) to yield a single library.

Polar Maps

Backbone dihedral angles were extracted from the conformers using Maestro 9.2 and converted to 0-360° scale before being plotted onto polar coordinate maps using Microsoft Excel 2007 with the Polar Plot2[3] add-in.

DISCON

DISCON is a Windows/Mac/Linux application developed by the Amos Smith laboratory for calculating the distribution of solution conformations of a flexible organic molecule by using NOE cross-peak volumes, coupling constants, and a pre-generated library of conformers. The program uses a similar approach to the NAMFIS methodology with the additional steps of using NMR variables to initially cluster compounds and the use of a genetic algorithm to assist with deconvolution, which are beneficial in identifying representative structures while avoiding overfitting. Additional information on DISCON, including software binaries, test files and program documentation, may be found at http://discon.sourceforge.net and in selected publications from the Smith lab[4].

Evaluation of the dactylolide conformer library with DISCON was initially undertaken with the aim of determining the proper clustering level. After examining a wide range, a level of 10 was found to provide an RMS deviation lower than any of the individual structures while still retaining them into distinctive conformational families. Additional testing with different genetic algorithm parameters and random selection of representative structures from individual clusters eventually led to the determination of the major conformers from their respective families.

Coordinates for Solution Conformations

Conformer A

56 57 0 0 1 0 999 V2000

[3] http://www.andypope.info/charts/polarplot3.htm

[4] Smith, A. B.; Atasoylu, O.; Risatti, C.; Bennett, C. S.; Liu, J.; Cheng, H.; TenDyke, K.; Xu, Q. *J. Am. Chem. Soc*. **2011**, *133*, 14042-14053.

1.6625	7.7373	15.8925	C	0	0	1	0	0	0
0.6574	8.7333	15.7041	O	0	0	0	0	0	0
1.1734	10.0584	15.8684	C	0	0	2	0	0	0
1.6173	10.2332	17.3270	C	0	0	0	0	0	0
2.1484	7.7878	17.3473	C	0	0	0	0	0	0
2.5092	7.9224	15.1982	H	0	0	0	0	0	0
2.0405	10.2199	15.1876	H	0	0	0	0	0	0
2.0380	11.2526	17.4788	H	0	0	0	0	0	0
0.7427	10.1375	18.0099	H	0	0	0	0	0	0
1.3169	7.5240	18.0402	H	0	0	0	0	0	0
2.9534	7.0352	17.5062	H	0	0	0	0	0	0
0.9947	6.4224	15.5719	C	0	0	0	0	0	0
1.4591	5.4347	14.7858	C	0	0	0	0	0	0
0.6179	4.2019	14.5147	C	0	0	0	0	0	0
1.2615	3.3040	14.6586	H	0	0	0	0	0	0
-0.1824	4.1134	15.2824	H	0	0	0	0	0	0
-0.7840	5.3390	12.8721	O	0	0	0	0	0	0
-2.7239	6.7907	13.3300	C	0	0	0	0	0	0
-2.2795	7.8030	12.5751	C	0	0	0	0	0	0
-3.0726	9.0272	12.3814	C	0	0	0	0	0	0
-2.6333	10.1533	11.7924	C	0	0	0	0	0	0
-1.2368	10.3846	11.2545	C	0	0	0	0	0	0
-0.5497	9.5505	11.5093	H	0	0	0	0	0	0
-1.2980	10.3822	10.1418	H	0	0	0	0	0	0
-0.1925	11.8445	13.1227	C	0	0	0	0	0	0
-0.3719	10.9063	14.0599	C	0	0	0	0	0	0
0.0622	11.0484	15.4975	C	0	0	0	0	0	0
-0.8217	10.8958	16.1582	H	0	0	0	0	0	0
0.4186	12.0872	15.6832	H	0	0	0	0	0	0
3.8571	9.4459	18.1578	C	0	0	0	0	0	0
2.6377	9.1777	17.6757	C	0	0	0	0	0	0
4.1794	10.4812	18.3579	H	0	0	0	0	0	0
4.5782	8.6400	18.3731	H	0	0	0	0	0	0
0.0042	6.2926	16.0444	H	0	0	0	0	0	0
-0.8489	9.9455	13.8085	H	0	0	0	0	0	0
0.2900	12.8015	13.3858	H	0	0	0	0	0	0
-3.6970	6.8510	13.8441	H	0	0	0	0	0	0
-1.2983	7.7061	12.0826	H	0	0	0	0	0	0
-4.1176	9.0113	12.7425	H	0	0	0	0	0	0
2.7854	5.4741	14.0618	C	0	0	0	0	0	0
3.4824	6.2327	14.4752	H	0	0	0	0	0	0
3.3086	4.4952	14.1335	H	0	0	0	0	0	0
2.6463	5.7027	12.9824	H	0	0	0	0	0	0
-3.5716	11.3223	11.6023	C	0	0	0	0	0	0
-3.6163	11.6279	10.5336	H	0	0	0	0	0	0
-4.6121	11.0895	11.9182	H	0	0	0	0	0	0

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 -3.2425   12.2049   12.1934 H   0  0  0  0  0  0
 -1.9507    5.5471   13.5235 C   0  0  0  0  0  0
 -2.3810    4.6991   14.2724 O   0  0  0  0  0  0
 -0.6010   11.6963   11.7080 C   0  0  0  0  0  0
 -0.4106   12.6082   10.9305 O   0  0  0  0  0  0
  0.0025    4.1715   13.1062 C   0  0  2  0  0  0
  0.8390    4.1845   12.3698 H   0  0  0  0  0  0
 -1.6011    2.9621   12.0679 H   0  0  0  0  0  0
 -0.7729    2.8913   12.8187 C   0  0  0  0  0  0
 -0.5265    1.8116   13.3109 O   0  0  0  0  0  0
 1  2  1  0  0  0
 1  5  1  0  0  0
 1  6  1  0  0  0
 1 12  1  0  0  0
 2  3  1  0  0  0
 3  4  1  0  0  0
 3  7  1  0  0  0
 3 27  1  0  0  0
 4  8  1  0  0  0
 4  9  1  0  0  0
 4 31  1  0  0  0
 5 10  1  0  0  0
 5 11  1  0  0  0
 5 31  1  0  0  0
12 13  2  0  0  0
12 34  1  0  0  0
13 14  1  0  0  0
13 40  1  0  0  0
14 15  1  0  0  0
14 16  1  0  0  0
14 52  1  0  0  0
17 48  1  0  0  0
17 52  1  0  0  0
18 19  2  0  0  0
18 37  1  0  0  0
18 48  1  0  0  0
19 20  1  0  0  0
19 38  1  0  0  0
20 21  2  0  0  0
20 39  1  0  0  0
21 22  1  0  0  0
21 44  1  0  0  0
22 23  1  0  0  0
22 24  1  0  0  0
22 50  1  0  0  0
25 26  2  0  0  0
```

```
25 36  1  0  0  0
25 50  1  0  0  0
26 27  1  0  0  0
26 35  1  0  0  0
27 28  1  0  0  0
27 29  1  0  0  0
30 31  2  0  0  0
30 32  1  0  0  0
30 33  1  0  0  0
40 41  1  0  0  0
40 42  1  0  0  0
40 43  1  0  0  0
44 45  1  0  0  0
44 46  1  0  0  0
44 47  1  0  0  0
48 49  2  0  0  0
50 51  2  0  0  0
52 53  1  0  0  0
52 55  1  0  0  0
54 55  1  0  0  0
55 56  2  0  0  0
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Conformer B

```
 56 57  0  0  1  0            999 V2000
    1.6624    7.7374   15.8614 C   0  0  1  0  0  0
    0.6645    8.7528   15.7717 O   0  0  0  0  0  0
    1.1910   10.0617   16.0083 C   0  0  2  0  0  0
    1.6803   10.1407   17.4597 C   0  0  0  0  0  0
    2.1888    7.6933   17.3025 C   0  0  0  0  0  0
    2.4874    7.9507   15.1479 H   0  0  0  0  0  0
    2.0380   10.2639   15.3131 H   0  0  0  0  0  0
    2.1144   11.1455   17.6623 H   0  0  0  0  0  0
    0.8269   10.0069   18.1626 H   0  0  0  0  0  0
    1.3732    7.3928   17.9994 H   0  0  0  0  0  0
    2.9900    6.9256   17.3932 H   0  0  0  0  0  0
    0.9592    6.4561   15.4773 C   0  0  0  0  0  0
    1.4817    5.3959   14.8361 C   0  0  0  0  0  0
    0.6238    4.1873   14.5224 C   0  0  0  0  0  0
    1.0550    3.3128   15.0623 H   0  0  0  0  0  0
   -0.3949    4.3323   14.9503 H   0  0  0  0  0  0
   -0.0028    4.9973   12.3426 O   0  0  0  0  0  0
   -0.4571    6.1107   10.1796 C   0  0  0  0  0  0
   -0.8533    7.2809   10.6944 C   0  0  0  0  0  0
   -1.4182    8.3380    9.8397 C   0  0  0  0  0  0
   -1.5601    9.6232   10.2038 C   0  0  0  0  0  0
```

```
-1.0896   10.1984   11.5247 C   0  0  0  0  0  0
-0.1706    9.6564   11.8415 H   0  0  0  0  0  0
-0.7796   11.2555   11.3621 H   0  0  0  0  0  0
-1.6530   10.3962   14.0223 C   0  0  0  0  0  0
-0.4505   10.9078   14.3156 C   0  0  0  0  0  0
 0.0640   11.0635   15.7258 C   0  0  0  0  0  0
-0.7640   10.9150   16.4556 H   0  0  0  0  0  0
 0.4329   12.1045   15.8696 H   0  0  0  0  0  0
 3.9374    9.2825   18.1636 C   0  0  0  0  0  0
 2.7006    9.0559   17.7051 C   0  0  0  0  0  0
 4.2760   10.2999   18.4206 H   0  0  0  0  0  0
 4.6573    8.4588   18.3020 H   0  0  0  0  0  0
-0.1029    6.4145   15.7796 H   0  0  0  0  0  0
 0.2559   11.1952   13.5192 H   0  0  0  0  0  0
-2.3503   10.1169   14.8307 H   0  0  0  0  0  0
-0.5213    5.9276    9.0942 H   0  0  0  0  0  0
-0.7626    7.4509   11.7808 H   0  0  0  0  0  0
-1.7649    8.0452    8.8314 H   0  0  0  0  0  0
 2.9237    5.2979   14.3974 C   0  0  0  0  0  0
 3.0112    5.3666   13.2909 H   0  0  0  0  0  0
 3.5618    6.0976   14.8286 H   0  0  0  0  0  0
 3.3702    4.3291   14.7122 H   0  0  0  0  0  0
-2.2101   10.6260    9.2815 C   0  0  0  0  0  0
-1.4920   11.4237    8.9889 H   0  0  0  0  0  0
-2.5841   10.1606    8.3433 H   0  0  0  0  0  0
-3.0788   11.1173    9.7727 H   0  0  0  0  0  0
 0.0910    5.0063   10.9924 C   0  0  0  0  0  0
 0.6172    4.0725   10.4281 O   0  0  0  0  0  0
-2.1260   10.1451   12.6435 C   0  0  0  0  0  0
-3.2935    9.8837   12.4444 O   0  0  0  0  0  0
 0.4809    3.8436   13.0331 C   0  0  2  0  0  0
 1.4838    3.5546   12.6440 H   0  0  0  0  0  0
-1.2184    2.7299   12.0202 H   0  0  0  0  0  0
-0.4624    2.6571   12.8428 C   0  0  0  0  0  0
-0.4299    1.6453   13.5091 O   0  0  0  0  0  0
1  2  1  0  0  0
1  5  1  0  0  0
1  6  1  0  0  0
1 12  1  0  0  0
2  3  1  0  0  0
3  4  1  0  0  0
3  7  1  0  0  0
3 27  1  0  0  0
4  8  1  0  0  0
4  9  1  0  0  0
4 31  1  0  0  0
```

```
 5 10  1  0  0  0
 5 11  1  0  0  0
 5 31  1  0  0  0
12 13  2  0  0  0
12 34  1  0  0  0
13 14  1  0  0  0
13 40  1  0  0  0
14 15  1  0  0  0
14 16  1  0  0  0
14 52  1  0  0  0
17 48  1  0  0  0
17 52  1  0  0  0
18 19  2  0  0  0
18 37  1  0  0  0
18 48  1  0  0  0
19 20  1  0  0  0
19 38  1  0  0  0
20 21  2  0  0  0
20 39  1  0  0  0
21 22  1  0  0  0
21 44  1  0  0  0
22 23  1  0  0  0
22 24  1  0  0  0
22 50  1  0  0  0
25 26  2  0  0  0
25 36  1  0  0  0
25 50  1  0  0  0
26 27  1  0  0  0
26 35  1  0  0  0
27 28  1  0  0  0
27 29  1  0  0  0
30 31  2  0  0  0
30 32  1  0  0  0
30 33  1  0  0  0
40 41  1  0  0  0
40 42  1  0  0  0
40 43  1  0  0  0
44 45  1  0  0  0
44 46  1  0  0  0
44 47  1  0  0  0
48 49  2  0  0  0
50 51  2  0  0  0
52 53  1  0  0  0
52 55  1  0  0  0
54 55  1  0  0  0
55 56  2  0  0  0
```

Conformer C

```
 56 57  0  0  1  0            999 V2000
    1.6590    7.7329   15.9303 C   0  0  1  0  0  0
    0.6632    8.7224   15.6727 O   0  0  0  0  0  0
    1.1287   10.0558   15.9087 C   0  0  2  0  0  0
    1.4716   10.2120   17.3960 C   0  0  0  0  0  0
    2.0605    7.7850   17.4117 C   0  0  0  0  0  0
    2.5457    7.9166   15.2887 H   0  0  0  0  0  0
    2.0390   10.2521   15.2961 H   0  0  0  0  0  0
    1.8489   11.2401   17.5955 H   0  0  0  0  0  0
    0.5633   10.0710   18.0240 H   0  0  0  0  0  0
    1.1965    7.5006   18.0552 H   0  0  0  0  0  0
    2.8691    7.0463   17.6124 H   0  0  0  0  0  0
    1.0073    6.4145   15.5904 C   0  0  0  0  0  0
    1.4371    5.4621   14.7433 C   0  0  0  0  0  0
    0.6250    4.1977   14.5323 C   0  0  0  0  0  0
    1.3177    3.3272   14.5902 H   0  0  0  0  0  0
   -0.0936    4.0647   15.3735 H   0  0  0  0  0  0
   -1.0990    5.2225   13.2277 O   0  0  0  0  0  0
   -2.6335    6.7397   12.0362 C   0  0  0  0  0  0
   -3.1548    7.2526   13.1579 C   0  0  0  0  0  0
   -4.2033    8.2852   13.1248 C   0  0  0  0  0  0
   -4.7440    8.8761   14.2049 C   0  0  0  0  0  0
   -4.3780    8.5926   15.6463 C   0  0  0  0  0  0
   -5.3158    8.4639   16.2330 H   0  0  0  0  0  0
   -3.8395    7.6264   15.7471 H   0  0  0  0  0  0
   -2.2330    9.9563   15.6153 C   0  0  0  0  0  0
   -1.3141   10.7825   16.1291 C   0  0  0  0  0  0
    0.0239   11.0239   15.4646 C   0  0  0  0  0  0
    0.3416   12.0744   15.6546 H   0  0  0  0  0  0
   -0.1010   10.9402   14.3599 H   0  0  0  0  0  0
    3.6878    9.4708   18.3285 C   0  0  0  0  0  0
    2.5014    9.1799   17.7820 C   0  0  0  0  0  0
    3.9735   10.5108   18.5579 H   0  0  0  0  0  0
    4.4172    8.6797   18.5696 H   0  0  0  0  0  0
    0.0577    6.2375   16.1275 H   0  0  0  0  0  0
   -1.4965   11.2961   17.0888 H   0  0  0  0  0  0
   -2.0288    9.4243   14.6721 H   0  0  0  0  0  0
   -2.9752    7.0757   11.0436 H   0  0  0  0  0  0
   -2.7817    6.8866   14.1281 H   0  0  0  0  0  0
   -4.5640    8.6058   12.1299 H   0  0  0  0  0  0
    2.6941    5.5534   13.9105 C   0  0  0  0  0  0
    2.4520    5.6903   12.8339 H   0  0  0  0  0  0
    3.3560    6.3931   14.2061 H   0  0  0  0  0  0
```

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  3.3005    4.6255   14.0024 H   0  0  0  0  0  0
 -5.7937    9.9501   14.0436 C   0  0  0  0  0  0
 -5.4506   10.9138   14.4803 H   0  0  0  0  0  0
 -6.0450   10.1448   12.9780 H   0  0  0  0  0  0
 -6.7391    9.6650   14.5559 H   0  0  0  0  0  0
 -1.5892    5.6949   12.0604 C   0  0  0  0  0  0
 -1.1779    5.2461   11.0136 O   0  0  0  0  0  0
 -3.5325    9.6961   16.2715 C   0  0  0  0  0  0
 -3.8875   10.3159   17.2517 O   0  0  0  0  0  0
 -0.1485    4.1571   13.2073 C   0  0  2  0  0  0
  0.5729    4.2887   12.3700 H   0  0  0  0  0  0
 -1.8781    2.8494   12.5509 H   0  0  0  0  0  0
 -0.8581    2.8207   13.0123 C   0  0  0  0  0  0
 -0.3813    1.7432   13.2949 O   0  0  0  0  0  0
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 1  6  1  0  0  0
 1 12  1  0  0  0
 2  3  1  0  0  0
 3  4  1  0  0  0
 3  7  1  0  0  0
 3 27  1  0  0  0
 4  8  1  0  0  0
 4  9  1  0  0  0
 4 31  1  0  0  0
 5 10  1  0  0  0
 5 11  1  0  0  0
 5 31  1  0  0  0
12 13  2  0  0  0
12 34  1  0  0  0
13 14  1  0  0  0
13 40  1  0  0  0
14 15  1  0  0  0
14 16  1  0  0  0
14 52  1  0  0  0
17 48  1  0  0  0
17 52  1  0  0  0
18 19  2  0  0  0
18 37  1  0  0  0
18 48  1  0  0  0
19 20  1  0  0  0
19 38  1  0  0  0
20 21  2  0  0  0
20 39  1  0  0  0
21 22  1  0  0  0
21 44  1  0  0  0
```

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22 24 1 0 0 0
22 50 1 0 0 0
25 26 2 0 0 0
25 36 1 0 0 0
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26 27 1 0 0 0
26 35 1 0 0 0
27 28 1 0 0 0
27 29 1 0 0 0
30 31 2 0 0 0
30 32 1 0 0 0
30 33 1 0 0 0
40 41 1 0 0 0
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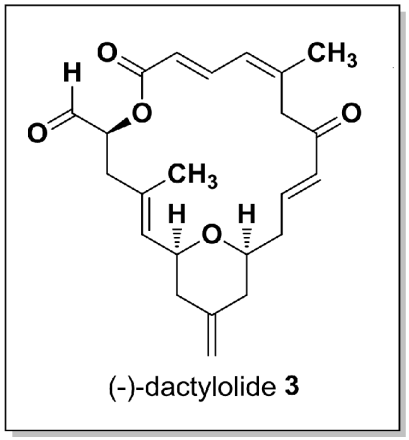
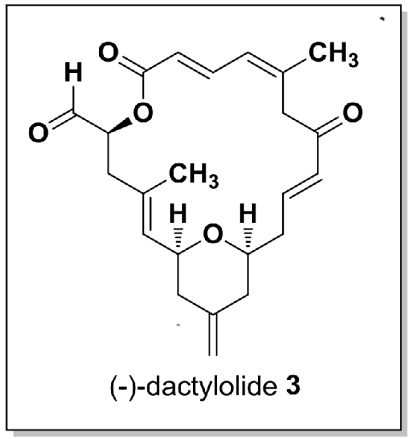


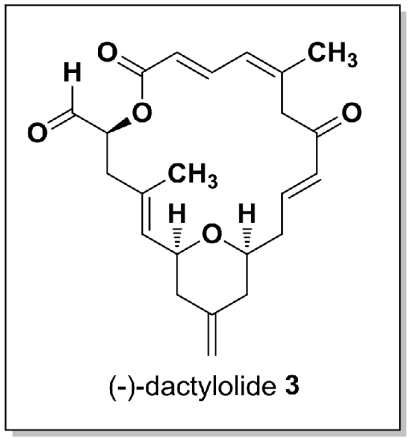
(-)-dactylolide **3**

^{1}H NMR spectrum of (-)-dactylolide in $CDCl_3$

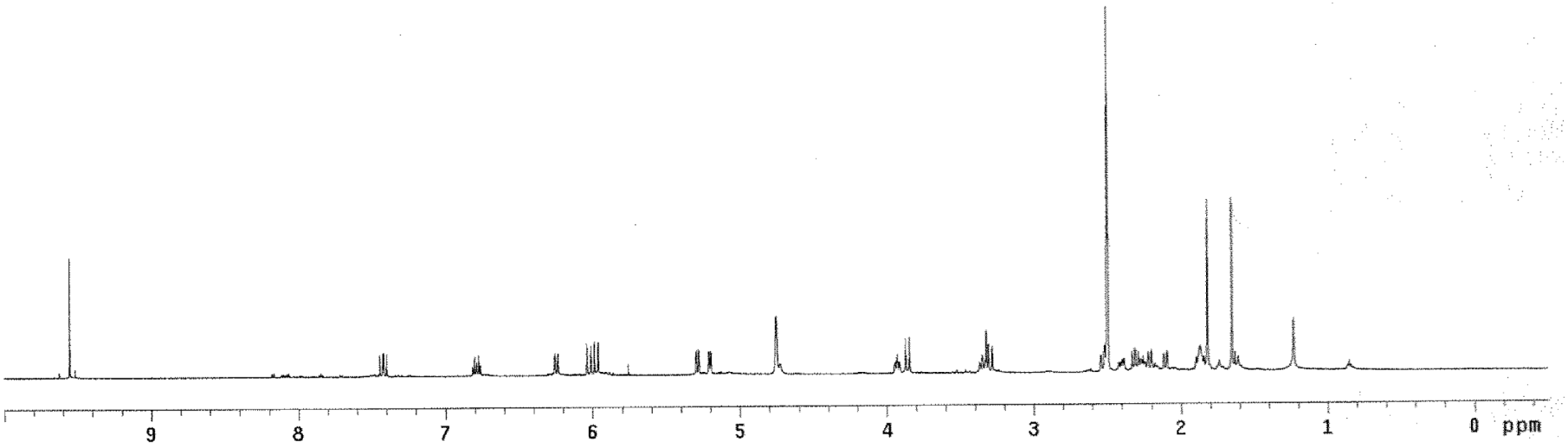


(-)-dactylolide **3**

^{13}C NMR spectrum of (-)-dactylolide in $CDCl_3$



(-)-dactylolide **3**



^{1}H NMR spectrum of (-)-dactylolide in DMSO-d_6

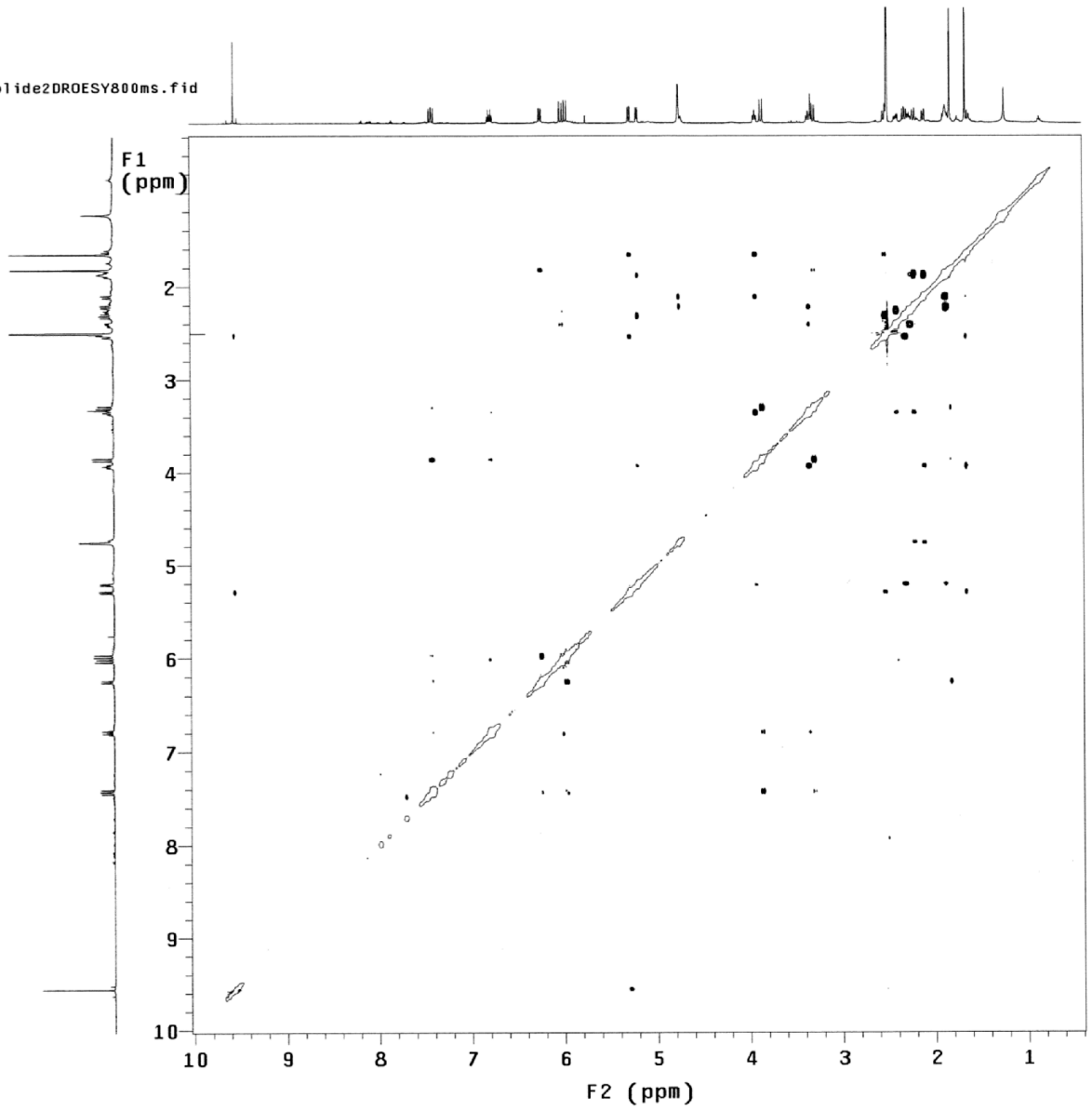
Std proton

File: afs/nd.edu/user23/mwilso16/Private/NMR/Dactylolide2DROESY800ms.fid

Pulse Sequence: ROESY

Solvent: dmso
Temp. 22.0 C / 295.1 K
Operator: mwilso16
File: Dactylolide2DROESY800ms
VNMRS-600 "nmr600"

Relax. delay 4.000 sec
Mixing 0.800 sec
Acq. time 0.177 sec
Width 5787.0 Hz
2D Width 5787.0 Hz
32 repetitions
2 x 128 increments
OBSERVE H1, 599.8756950 MHz
DATA PROCESSING
Gauss apodization 0.082 sec
F1 DATA PROCESSING
Gauss apodization 0.041 sec
FT size 2048 x 2048
Total time 11 hr, 25 min, 53 sec



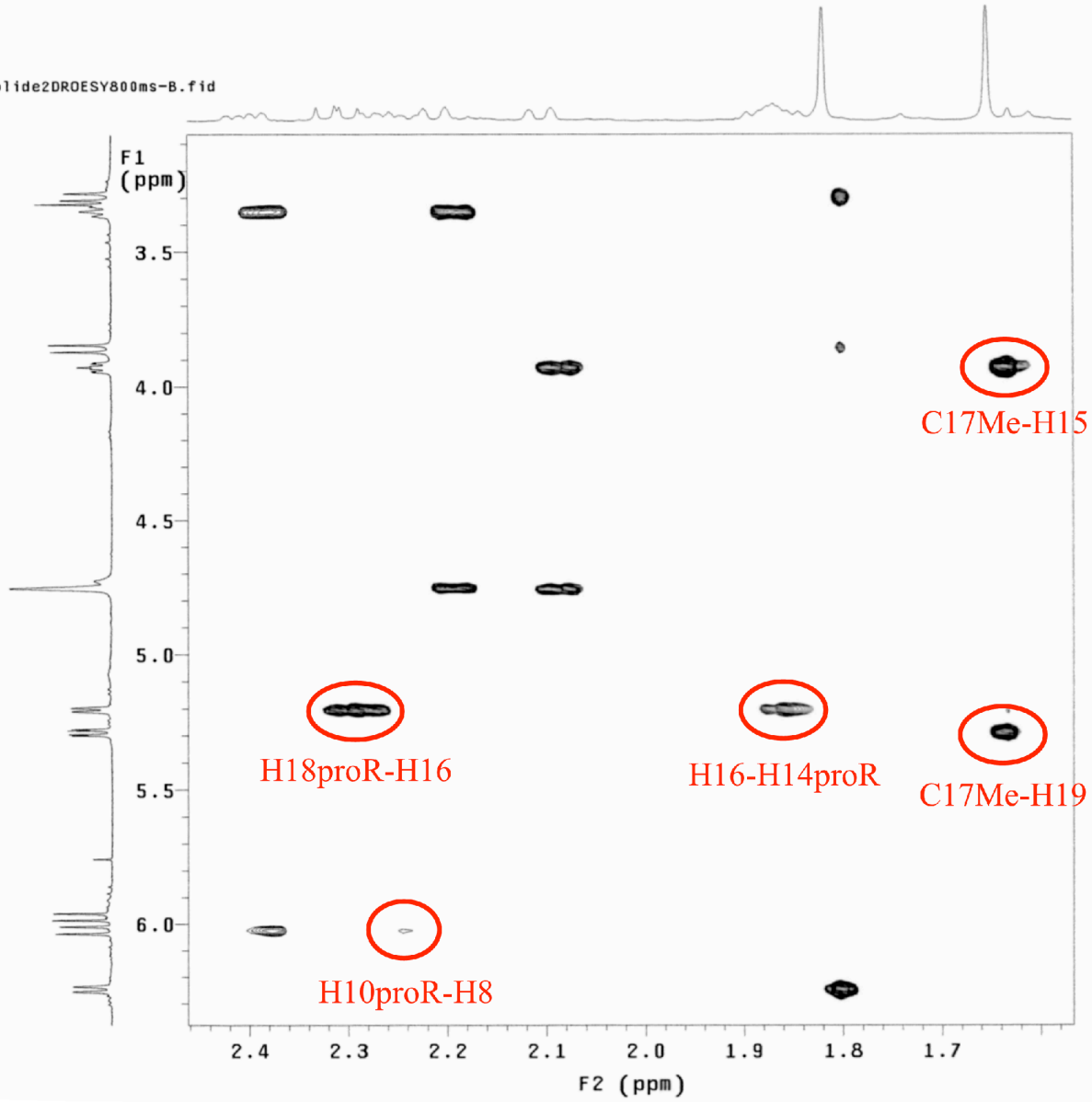
Std proton

File: afs/nd.edu/user23/mwilso16/Private/NMR/Dactylolide2DROESY800ms-B.fid

Pulse Sequence: ROESY

Solvent: dmso
Temp. 22.0 C / 295.1 K
Operator: mwilso16
File: Dactylolide2DROESY800ms-B
VNMRS-600 "nmr600"

Relax. delay 4.000 sec
Mixing 0.800 sec
Acq. time 0.177 sec
Width 5787.0 Hz
2D Width 5787.0 Hz
32 repetitions
2 x 128 increments
OBSERVE H1, 599.8757069 MHz
DATA PROCESSING
Gauss apodization 0.082 sec
F1 DATA PROCESSING
Gauss apodization 0.041 sec
FT size 2048 x 2048
Total time 11 hr, 25 min, 53 sec



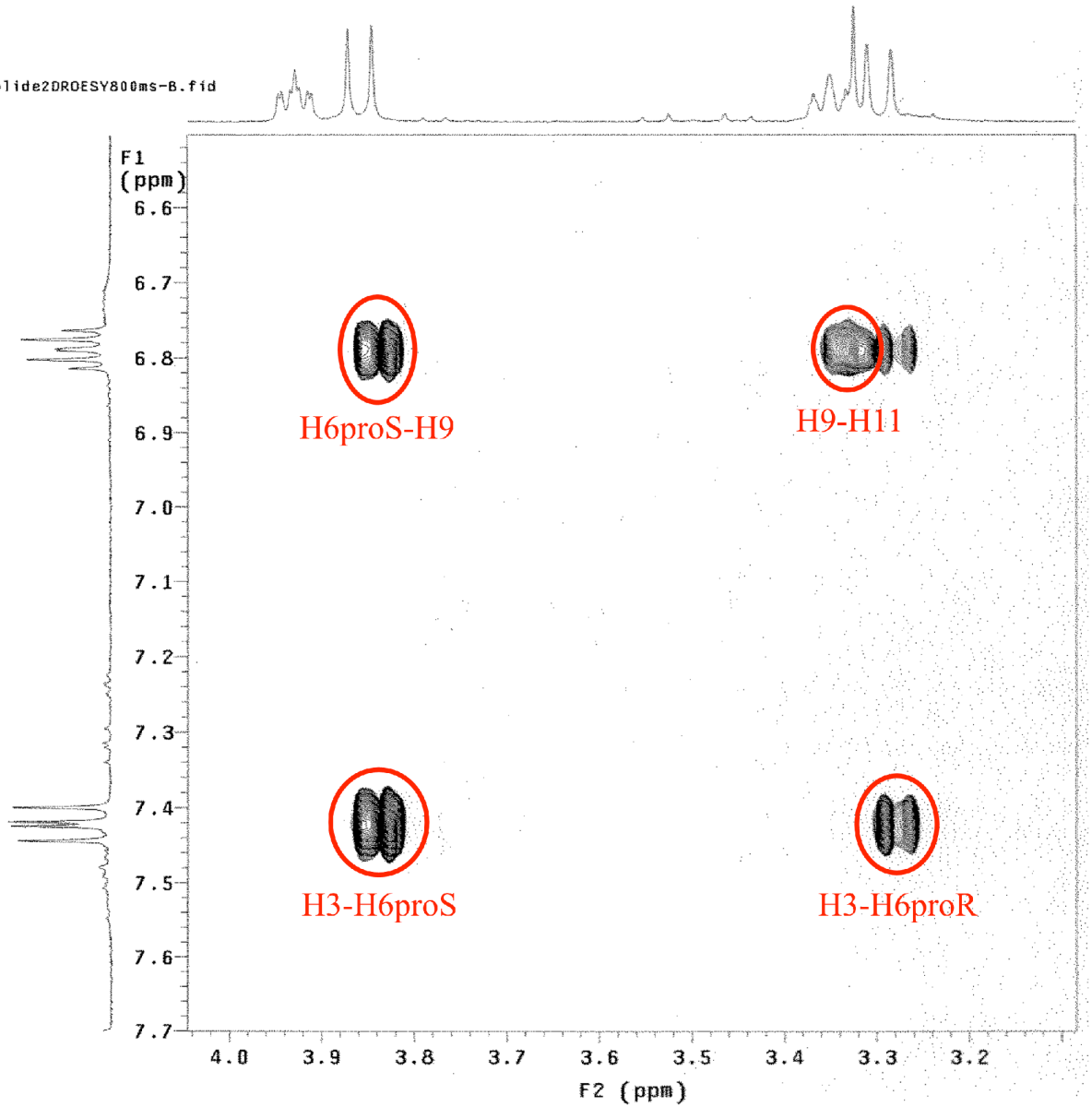
Std proton

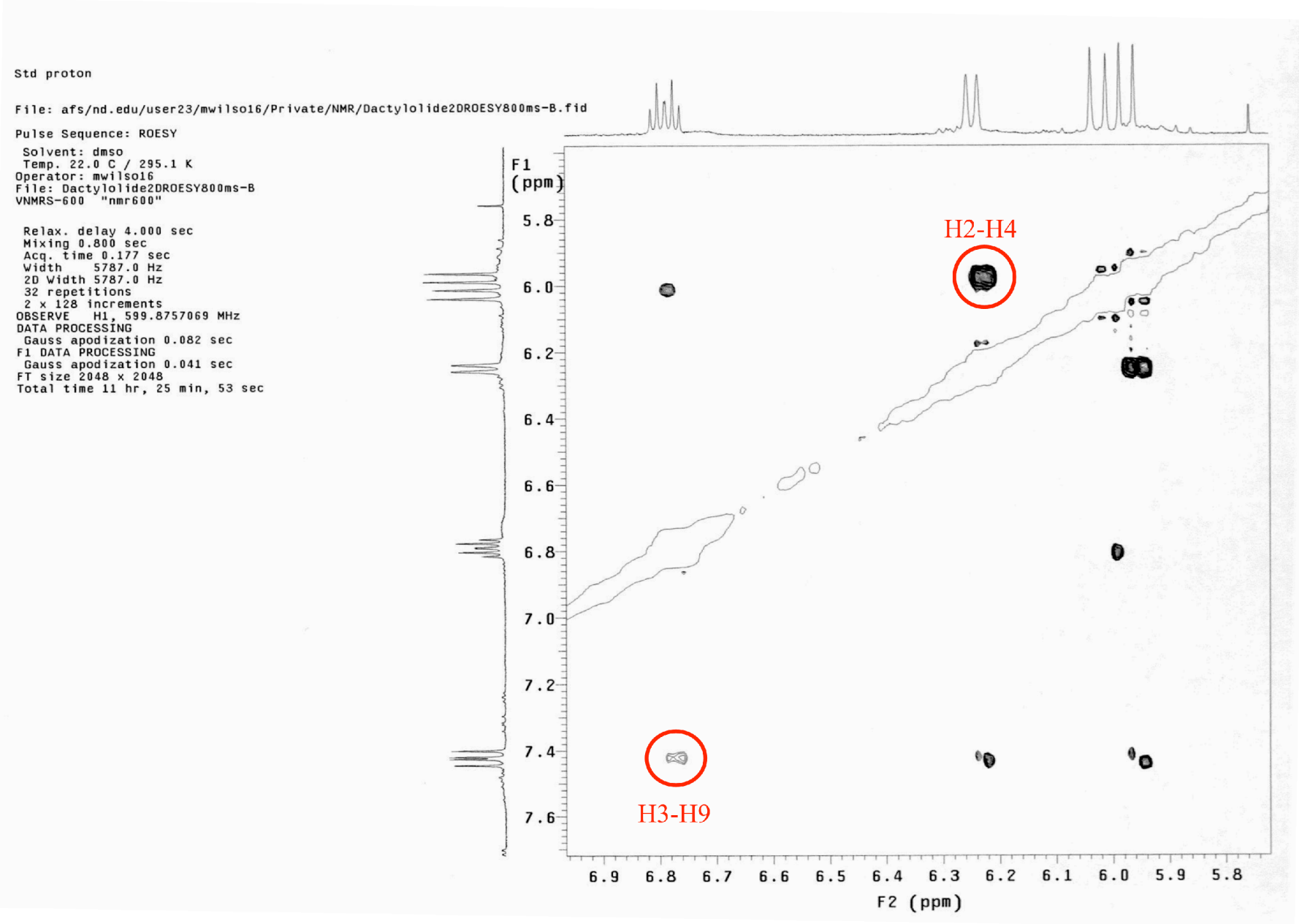
File: afs/nd.edu/user23/mwilso16/Private/NMR/Dactylolide2DROESY800ms-B.fid

Pulse Sequence: ROESY

Solvent: dmso
Temp. 22.0 C / 295.1 K
Operator: mwilso16
File: Dactylolide2DROESY800ms-B
VNMRS-600 "nmr600"

Relax. delay 4.000 sec
Mixing 0.800 sec
Acq. time 0.177 sec
Width 5787.0 Hz
2D Width 5787.0 Hz
32 repetitions
2 x 128 increments
OBSERVE H1, 599.8757069 MHz
DATA PROCESSING
Gauss apodization 0.082 sec
F1 DATA PROCESSING
Gauss apodization 0.041 sec
FT size 2048 x 2048
Total time 11 hr, 25 min, 53 sec




Std proton
File: afs/nd.edu/user23/mwilso16/Private/NMR/Dactylolide2DROESY800ms-B.fid
Pulse Sequence: ROESY
Solvent: dmso
Temp. 22.0 C / 295.1 K
Operator: mwilso16
File: Dactylolide2DROESY800ms-B
VNMRS-600 "nmr600"
Relax. delay 4.000 sec
Mixing 0.800 sec
Acq. time 0.177 sec
Width 5787.0 Hz
2D Width 5787.0 Hz
32 repetitions
2 x 128 increments
OBSERVE H1, 599.8757069 MHz
DATA PROCESSING
Gauss apodization 0.082 sec
F1 DATA PROCESSING
Gauss apodization 0.041 sec
FT size 2048 x 2048
Total time 11 hr, 25 min, 53 sec
F1 (ppm)
5.8
6.0
6.2
6.4
6.6
6.8
7.0
7.2
7.4
7.6
H2-H4
H3-H9
6.9
6.8
6.7
6.6
6.5
6.4
6.3
6.2
6.1
6.0
5.9
5.8
F2 (ppm)