

Towards Structural Correctness: Aquatolide and the Importance of 1D Proton NMR FID Archiving

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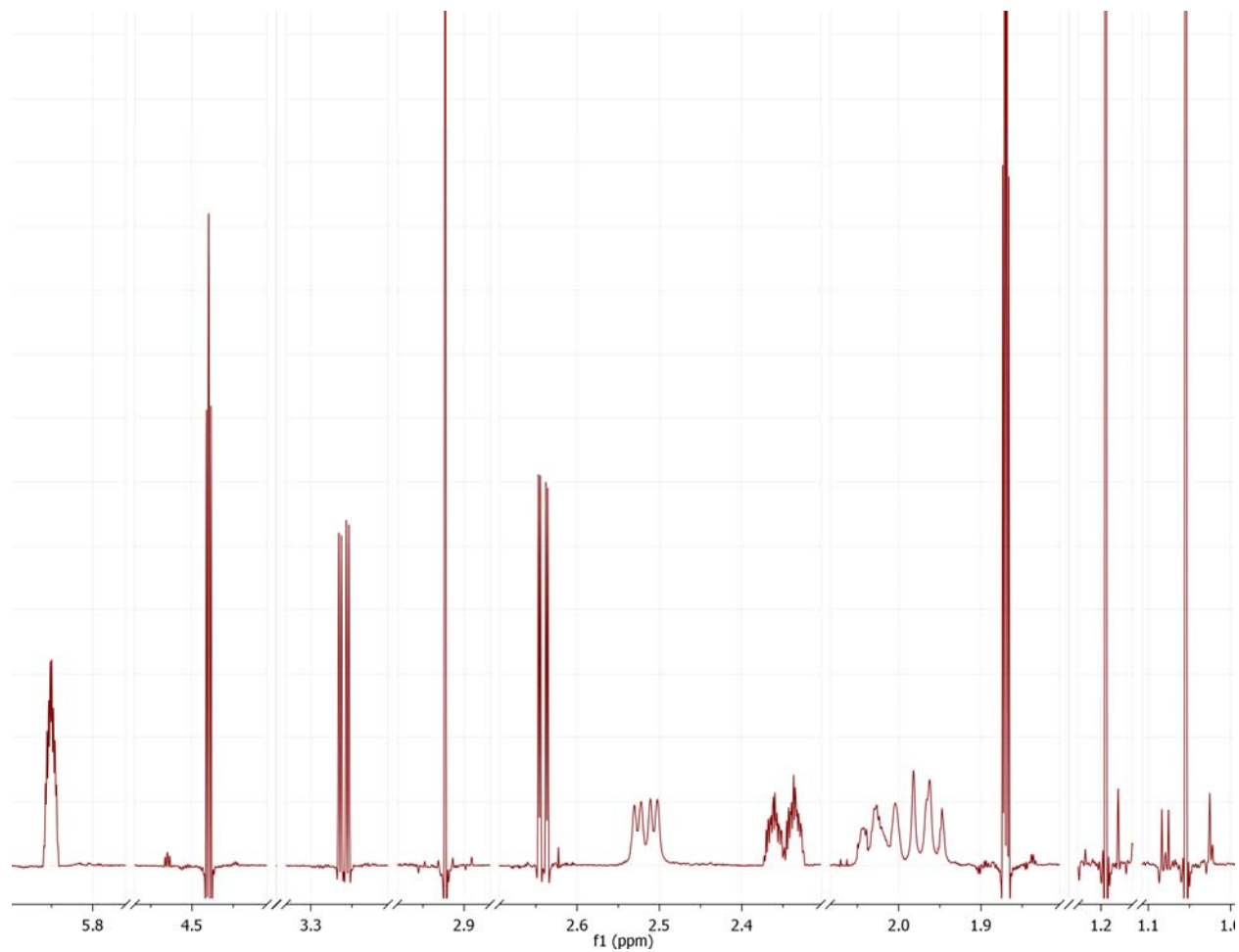
S1. Predicted coupling constants for the revised structure of aquatolide, 1b, displayed on a *J*-correlation map.

This figure was adapted from the data from page S29 of Lodewyk, M. W.; Soldi, C.; Jones, P. B.; Olmstead, M. M.; Rita, J.; Shaw, J. T.; Tantillo, D. J. *J. Amer. Chem. Soc.* **2012**, *134*, 18550-18553.

δ	H-#	1	2	4a	4b	5a	5b	6	9	10	13	14	15
4.48	1		3	5	5	6	6	7	4	4	7	4	4
3.25	2	2.3254		4	4	5	5	6	3	4	6	5	5
2.50	4a	0.4581	-0.3956		2	3	3	4	5	4	6	6	6
2.02	4b	-0.0273	-0.0673	-15.7261		3	3	4	5	4	6	6	6
2.38	5a	0.0223	0.0021	1.8121	11.6851		2	3	6	5	5a	7	7
2.07	5b	0.0044	-0.0280	6.7326	13.0129	-20.9269		3	6	5	5a	7	7
5.84	6	-0.0077	0.1760	0.9081	-0.2494	3.4453	5.3638		5	6	4a	8	8
2.92	9	0.0670	-0.1190	-0.5521	-0.3150	-0.4070	-0.2731	-0.0002		3	5	5	5
2.64	10	1.8327	6.7667	-0.0503	-0.2801	-0.0022	-0.0575	0.0056	0.0735		6	4	4
1.86	13	-0.0021	0.0277	0.0115	-0.0185	2.8324	2.5084	1.8150	0.1372	0.0116		8	8
1.04	14	-0.0677	0.0898	0.0217	0.0477	-0.0051	0.0042	0.0021	-0.3778	-0.1962	0.0000		4
1.18	15	-0.1491	0.3876	0.0050	0.0016	0.0059	-0.0051	0.0047	0.0533	-0.1232	0.0000	0.5341	

S2. NMR Data (800 MHz) Isolated Aquatolide (1b) from Lodewyk et al. JACS 2012. ^1H NMR spectrum (Lorentzian-Gaussian window function with LB=(-)1.0 Hz and GF=0.10 Hz).

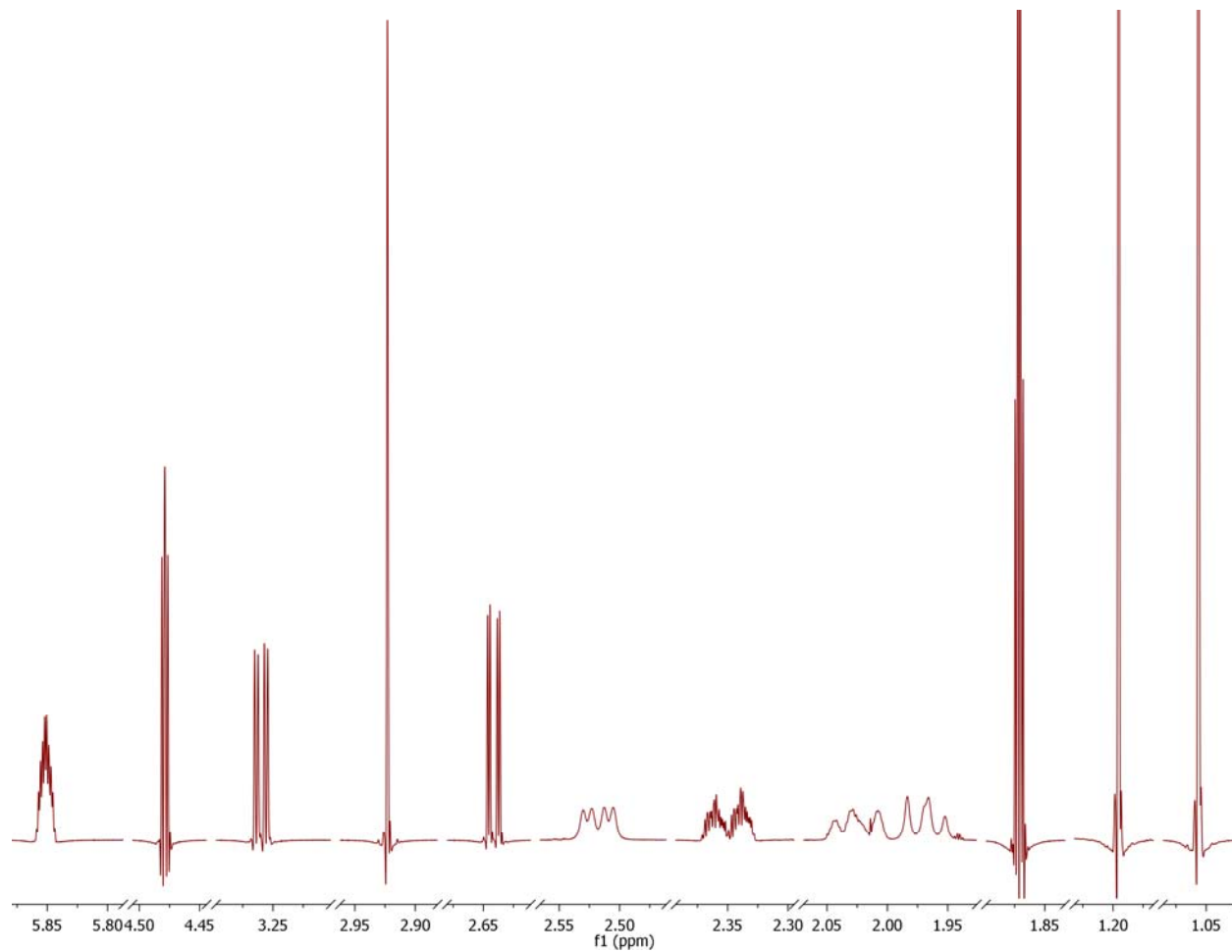
Raw NMR data: ^1H NMR FID (800 MHz), HiFSA PMS file: included in separate ZIP file.



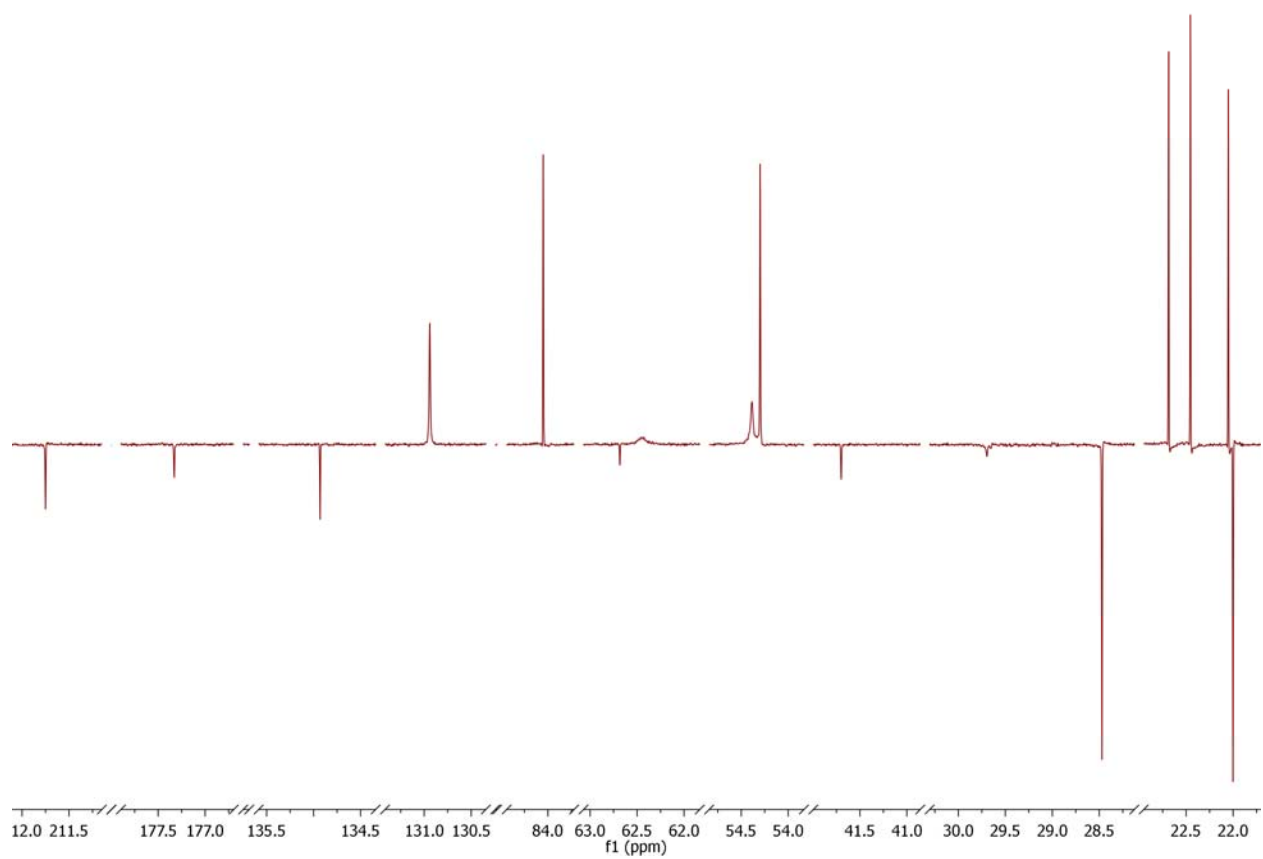
S3. NMR Data Synthetic Aquatolide Using the Sample from Saya et al. Orgl. Lett. 2015. 900 MHz spectra: ^1H NMR (A), ^{13}C NMR DEPT-135 (B), COSY (C), HSQC (D), and HMBC (E)

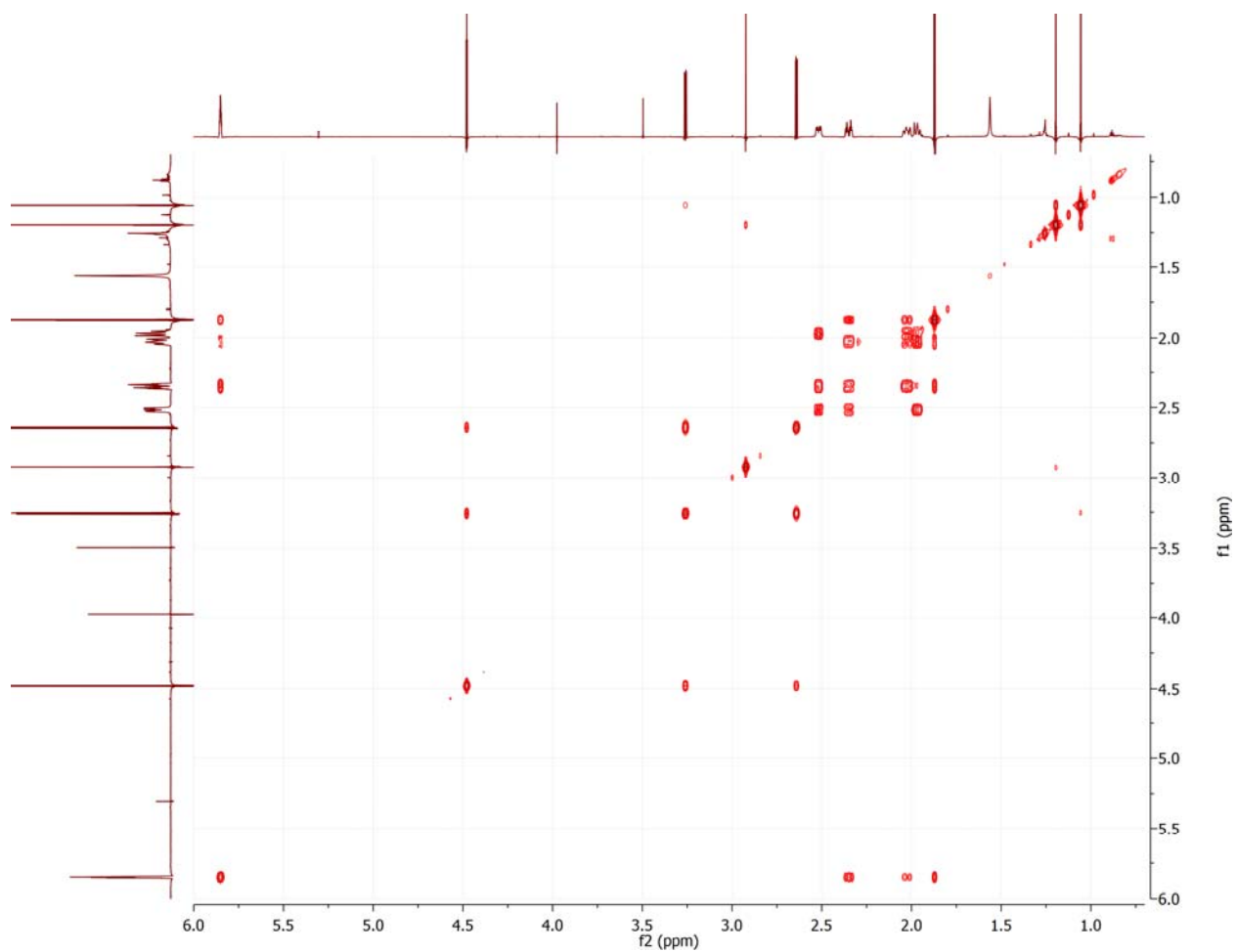
Raw NMR data: ^1H NMR FID (400 MHz), ^1H NMR FID (900 MHz), HiFSA PMS file: included in separate ZIP file.

A



B

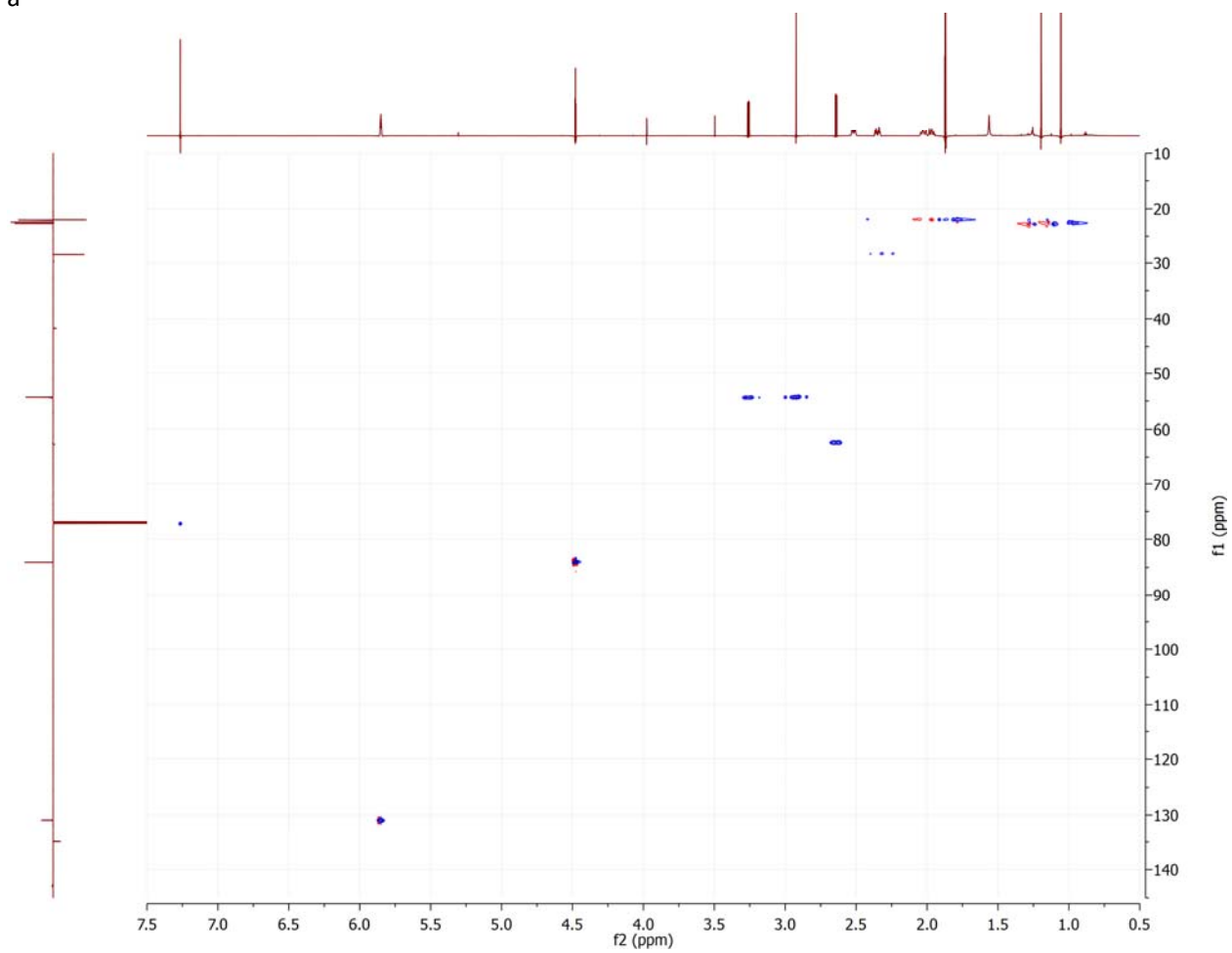




C

D

a



E

