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Supplementary Information

Experimental and NMR Theoretical Methodology Applied to Geometric Analysis of the Bioactive Clerodane *trans*-Dehydrocrotonin

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Bond length and bond angles data for *t*-DCTN

Table S1. Selected bond length and bond angles of *t*- DCTN from X-ray diffraction and B3LYP method

Atoms	Bond length /Å		Atoms	Bond angles / °	
	X-ray	B3LYP		X-ray	B3LYP
C1–C2	1.506	1.519	C2–C1–C10	113.0	111.9
C1–C10	1.523	1.536	O1–C2–C3	122.5	122.3
C2–O1	1.215	1.220	O1–C2–C1	121.4	121.5
C2–C3	1.449	1.466	C3–C2–C1	116.0	116.1
C3–C4	1.325	1.347	C4–C3–C2	124.1	123.2
C4–C18	1.501	1.504	C3–C4–C18	120.0	120.0
C4–C5	1.506	1.522	C3–C4–C5	121.5	121.9
C5–C6	1.525	1.537	C18–C5–C4	118.5	118.0
C5–C10	1.539	1.548	C4–C5–C6	113.0	113.5
C6–C7	1.521	1.527	C4–C5–C10	111.1	111.3
C7–C8	1.508	1.533	C6–C5–C10	110.0	110.7
C8–C17	1.538	1.535	C7–C6–C5	110.4	110.7
C8–C9	1.545	1.578	C8–C7–C6	113.2	112.5
C9–C20	1.526	1.536	C7–C8–C17	109.7	110.3
C9–C11	1.544	1.547	C7–C8–C9	113.4	112.5
C9–C10	1.546	1.565	C17–C8–C9	114.1	114.0
C11–C12	1.523	1.544	C20–C9–C11	101.6	102.6
C12–C13	1.466	1.491	C20–C9–C8	111.6	110.5
C12–O3	1.470	1.459	C11–C9–C8	112.5	111.8
C13–C16	1.343	1.361	C20–C9–C10	110.3	110.8
C13–C14	1.424	1.440	C11–C9–C10	112.5	111.8
C15–O4	1.358	1.364	C8–C9–C10	110.5	110.0
C16–O4	1.347	1.358	C1–C10–C5	109.2	109.2
C20–O2	1.200	1.203	C1–C10–C9	113.0	113.5
C20–O3	1.346	1.353	C5–C10–C9	114.3	114.0
			C12–C11–C9	106.9	105.7
			C13–C12–O3	110.4	109.9
			O3–C12–C11	103.1	105.0
			C16–C13–C14	103.3	105.7
			C16–C13–C12	125.5	125.7
			C15–C14–C13	108.4	106.2
			C14–C15–O4	109.8	110.5
			C13–C16–O4	112.9	110.7
			O2–C20–O3	120.5	121.3
			O2–C20–C9	128.1	127.4
			O3–C20–C9	111.4	111.2
			C20–O3–C12	111.8	112.2
			C16–O4–C15	105.6	106.8

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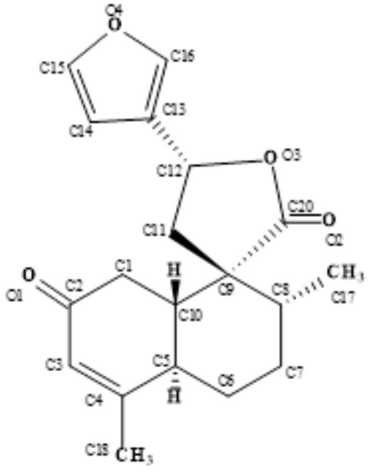
^{1}H and ^{13}C NMR chemical shift data, in ppm, for *t*-DCTN

Following the IUPAC diterpene numeration rule, both carbon (C) and hydrogen (H) atoms have the same numeration. The numbered skeleton structure of *t*-DCTN is indicated below Table S2.

Table S2. Experimental[59] and theoretical ^{1}H and ^{13}C NMR chemical shifts (ppm) for *t*-DCTN

Atoms	Experimental		Theoretical	
	δ C	δ H	δ C	δ H
1(p)a	39.76	2.19	48.71	2.38
1(p)e		2.54		2.70
2	197.50	–	206.96	–
3	126.73	5.89	138.58	6.19
4	165.70	–	180.28	–
5	39.59	3.18	49.84	3.42
6a	28.23	1.18	38.44	1.21
6e		2.27		2.32
7a	30.13	1.88	39.43	2.20
7e		1.66		1.56
8	41.74	1.69	54.05	1.59
9	51.40	–	63.10	–
10	46.11	1.81	57.73	1.67
11	40.53	2.42	51.67	2.49
11’		2.37		2.09
12	72.31	5.43	82.50	5.33
13	125.06	–	138.87	–
14	107.98	6.41	119.78	6.91
15	144.26	7.45	156.99	7.78
16	139.93	7.46	152.59	7.69
17	17.57	1.16	24.24	1.43
18	21.90	1.97	29.96	2.07
20	176.90	–	188.61	–

a: axial; e: equatorial; (p): pseudo (related to some hydrogens of decalin).



NMR spin-spin coupling constant data for *t*-DCTN

Table S3. Experimental[59] and theoretical (B3LYP/6-311++(d,p)) NMR spin-spin coupling constants and delocalization indexes (DI) for hydrogen pairs in *t*-DCTN.

H pairs	(*J*2-*J*4) / Hz		DI × 10^{3} / a.u.
	Experimental	B3LYP	
H1(p)a–H10	13.8	12.13	12.096
H1(p)e–H10	2.8	2.71	2.662
H3–H18	1.27	1.96	1.245
H3–H5	1.19	2.99	1.253
H5–H6a	12.5	10.51	11.098
H5–H10	10.7	8.42	10.546
H5–H6e	3.35	3.58	3.328
H5–H18	1.2	2.07	2.303
H6a–H7a	12.7	11.41	11.808
H6a–H7e	3.61	3.51	4.134
H6e–H7a	3.6	3.601	3.882
H6e–H7e	3.3	3.13	3.765
H7a–H8	12.31	10.5	11.877
H7e–H8	3.58	3.38	3.991
H8–H17	6.8	3.61	3.531
H11- H12	8.62	7.82	6.998
H11’- H12	8.65	8.1	7.309
H14–H15	1.83	1.72	2.101
H14–H16	0.89	0.34	1.651
H15–H16	1.66	0.87	1.919

a: axial; e: equatorial; (p): pseudo (related to some hydrogens of decalin).

Z-Matrix structure for *t*-DCTN

0 1
C
C 1 B1
C 2 B2 1 A1
C 3 B3 2 A2 1 D1
C 4 B4 3 A3 2 D2
C 1 B5 2 A4 3 D3
H 4 B6 3 A5 2 D4
H 4 B7 3 A6 2 D5
H 6 B8 1 A7 2 D6
C 3 B9 2 A8 1 D7
C 2 B10 1 A9 6 D8
H 11 B11 2 A10 1 D9
H 11 B12 2 A11 1 D10
C 11 B13 2 A12 1 D11
H 14 B14 11 A13 2 D12
H 14 B15 11 A14 2 D13
C 14 B16 11 A15 2 D14
H 17 B17 14 A16 11 D15

C	1	B18	6	A17	5	D16	B24	1.09333367
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H	19	B20	1	A19	6	D18	B26	1.54470272
H	19	B21	1	A20	6	D19	B27	1.09139224
C	17	B22	14	A21	11	D20	B28	1.08851816
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H	23	B25	17	A24	14	D23	B31	1.35438936
C	10	B26	3	A25	2	D24	B32	1.20106107
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H	27	B28	10	A27	3	D26	B34	1.49165820
C	10	B29	3	A28	2	D27	B35	1.36102398
C	27	B30	10	A29	3	D28	B36	2.18706738
O	30	B31	10	A30	3	D29	B37	1.07745495
O	30	B32	10	A31	3	D30	B38	1.07667935
H	31	B33	27	A32	10	D31	B39	1.21874690
C	31	B34	27	A33	10	D32	B40	1.35577876
C	35	B35	31	A34	27	D33	B41	1.07837670
C	36	B36	35	A35	31	D34	B42	1.35920779
H	36	B37	35	A36	31	D35	B43	1.07000000
H	37	B38	36	A37	35	D36	B44	1.07000000
O	5	B39	4	A38	3	D37	A1	113.69588071
C	37	B40	36	A39	35	D38	A2	112.64210726
H	41	B41	37	A40	36	D39	A3	111.84531507
O	36	B42	35	A41	31	D40	A4	122.44597069
H	3	B43	2	A42	1	D41	A5	111.31552864
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							A7	120.97398975
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B13	1.54115916						A20	111.30366213
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B16	1.53322782						A23	109.58903837
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CIF data for *t*-DCTN

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C20 C 0.0852(6) 0.5476(4) 0.1903(3) 0.0646(11) Uani 1 1 d . . .
O1 O 0.2325(5) 0.6745(2) -0.11208(19) 0.0905(10) Uani 1 1 d . . .
O2 O -0.0543(4) 0.5905(2) 0.19977(19) 0.0798(9) Uani 1 1 d . . .
O3 O 0.0855(4) 0.4511(2) 0.1829(2) 0.0809(10) Uani 1 1 d . . .
O4 O 0.2726(6) 0.1733(3) 0.0894(3) 0.1154(13) Uani 1 1 d . . .

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C1 0.059(3) 0.061(2) 0.070(2) -0.003(2) 0.001(2) 0.005(2)
C2 0.055(3) 0.077(3) 0.065(3) 0.000(2) -0.001(3) -0.002(2)
C3 0.059(3) 0.066(3) 0.089(3) 0.020(3) -0.009(3) -0.010(2)
C4 0.049(3) 0.059(3) 0.100(3) -0.006(3) -0.004(3) -0.009(2)
C5 0.047(2) 0.062(3) 0.080(3) -0.012(2) 0.005(2) -0.004(2)
C6 0.073(3) 0.086(3) 0.093(3) -0.027(3) -0.006(3) -0.006(3)
C7 0.079(3) 0.121(4) 0.069(3) -0.023(3) 0.000(3) -0.018(4)
C8 0.065(3) 0.108(4) 0.066(3) -0.001(3) -0.005(2) -0.010(3)
C9 0.045(2) 0.079(3) 0.061(2) 0.003(2) -0.001(2) 0.000(2)
C10 0.045(2) 0.064(2) 0.060(2) -0.003(2) 0.006(2) -0.006(2)
C11 0.053(2) 0.085(3) 0.080(3) 0.012(3) -0.007(2) 0.002(3)
C12 0.060(3) 0.078(3) 0.082(3) 0.022(3) -0.011(3) 0.007(3)
C13 0.068(3) 0.069(3) 0.092(4) 0.019(3) -0.012(3) -0.002(3)
C14 0.136(5) 0.086(4) 0.088(4) 0.009(3) -0.010(4) 0.002(4)
C15 0.147(6) 0.102(5) 0.113(5) 0.001(5) -0.026(4) 0.016(5)
C16 0.069(3) 0.093(4) 0.123(5) 0.029(4) -0.007(3) 0.009(3)
```

C17 0.119(5) 0.158(5) 0.067(3) 0.013(4) -0.014(3) -0.038(5)
C18 0.112(4) 0.059(3) 0.145(5) -0.013(3) -0.017(4) 0.003(3)
C20 0.054(3) 0.077(3) 0.063(3) 0.006(3) 0.005(3) -0.006(3)
O1 0.109(3) 0.099(2) 0.0640(18) -0.0010(18) -0.006(2) -0.004(2)
O2 0.057(2) 0.090(2) 0.093(2) -0.0023(19) 0.0137(17) 0.0001(18)
O3 0.063(2) 0.077(2) 0.103(3) 0.018(2) 0.009(2) -0.0064(19)
O4 0.102(3) 0.079(2) 0.165(4) 0.009(3) -0.025(3) 0.005(3)

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C3 C4 1.324(6) . ?
C4 C18 1.501(6) . ?
C4 C5 1.506(6) . ?
C5 C6 1.526(5) . ?
C5 C10 1.538(5) . ?
C6 C7 1.521(6) . ?
C7 C8 1.508(6) . ?
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C9 C10 1.547(5) . ?
C11 C12 1.522(6) . ?
C12 C13 1.467(6) . ?
C12 O3 1.470(5) . ?
C13 C16 1.343(6) . ?
C13 C14 1.423(7) . ?
C14 C15 1.331(7) . ?
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C20 O2 1.200(5) . ?
C20 O3 1.346(5) . ?

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C4 C3 C2 124.1(4) . . ?
C3 C4 C18 120.0(4) . . ?
C3 C4 C5 121.5(4) . . ?
C18 C4 C5 118.5(4) . . ?
C4 C5 C6 113.0(4) . . ?
C4 C5 C10 111.1(3) . . ?
C6 C5 C10 110.0(3) . . ?
C7 C6 C5 110.4(3) . . ?
C8 C7 C6 113.2(4) . . ?
C7 C8 C17 109.7(4) . . ?
C7 C8 C9 113.4(4) . . ?
C17 C8 C9 114.1(4) . . ?
C20 C9 C11 101.6(3) . . ?
C20 C9 C8 111.6(3) . . ?
C11 C9 C8 110.2(4) . . ?
C20 C9 C10 110.3(3) . . ?
C11 C9 C10 112.4(3) . . ?
C8 C9 C10 110.5(3) . . ?
C1 C10 C5 109.1(3) . . ?
C1 C10 C9 113.0(3) . . ?
C5 C10 C9 114.3(3) . . ?
C12 C11 C9 106.9(3) . . ?
C13 C12 O3 110.4(4) . . ?
C13 C12 C11 116.3(4) . . ?
O3 C12 C11 103.1(3) . . ?
C16 C13 C14 103.3(5) . . ?
C16 C13 C12 125.5(5) . . ?
C14 C13 C12 131.1(5) . . ?
C15 C14 C13 108.4(5) . . ?
C14 C15 O4 109.8(6) . . ?
C13 C16 O4 112.8(5) . . ?
O2 C20 O3 120.5(4) . . ?
O2 C20 C9 128.0(4) . . ?
O3 C20 C9 111.4(4) . . ?
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C16 O4 C15 105.6(5) . . ?

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