Formal Synthesis of ($\pm$)-Guanacastepene A

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Supporting Material

Copies of ^1H and ^{13}C NMR Spectra Pages S2-S57

pl-pentenyl-OH

Pulse Sequence: zgpg30

Solvent: CDCl₃

Ambient temperature

File: pl-pentenyl-OH

INOVA-500 "gambler"

Pulse 47.6 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

100 repetitions

OBSERVE H1, 399.7532349 MHz

DATA PROCESSING

FT size 16384

Total time 2 min, 44 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	16006.590	159.241	21.9
2	13938.198	138.664	32.2
3	11516.554	114.572	55.2
4	10744.436	106.891	100.0
5	7770.407	77.304	39.5
6	7739.125	76.992	39.5
7	7707.081	76.674	40.5
8	7467.511	74.290	73.4
9	3575.638	35.572	68.2
10	3377.268	33.599	76.1
11	3038.512	30.229	39.2
12	2521.986	25.090	71.4
13	2331.246	23.192	60.8
14	2282.416	22.707	68.4

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

File: acetopentene2

INOVA-500 "gamma"

Pulse 47.6 degrees
Acq. time 1.638 sec
Width 5000.0 Hz
32 repetitions
OBSERVE H1, 399.7532349 MHz
DATA PROCESSING
F1 size 16384
Total time 2 min, 44 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

File: pentenketonocrd1

INOVA-500 "gamble"

Pulse 47.6 degrees
Acq. time 1.638 sec
Width 5000.0 Hz
100 repetitions
OBSERVE H1, 399.7532349 MHz
DATA PROCESSING
F1 size 16384
Total time 2 min, 44 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	20979.838	208.717	5.9
2	14380.005	143.059	37.3
3	13947.259	138.754	22.4
4	12368.865	123.051	3.9
5	12354.217	122.905	48.6
6	11491.777	114.326	49.5
7	7771.017	77.310	16.4
8	7739.278	76.994	16.9
9	7706.929	76.672	17.0
10	4367.035	43.445	4.0
11	4334.075	43.117	78.7
12	3450.273	34.325	2.8
13	3347.732	33.305	61.7
14	2996.164	29.807	17.4
15	2942.452	29.273	63.7
16	2930.245	29.151	3.5
17	2897.896	28.830	73.7
18	2711.125	26.972	3.9
19	2670.842	26.571	75.6
20	2460.267	24.476	47.4
21	2347.961	23.359	3.8
22	2215.512	22.041	6.9
23	2111.141	21.003	129.7

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

INOVA-400 "fid"

PULSE SEQUENCE

Pulse 47.6 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

32 repetitions

OBSERVE H1, 399.7532351 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 52 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	22555.186	224.389	14.7
2	13922.543	138.508	27.7
3	11517.418	114.581	38.4
4	7771.507	77.315	114.7
5	7739.889	77.000	115.8
6	7707.843	76.681	117.4
7	5227.518	52.006	29.4
8	4846.816	48.218	61.0
9	3791.022	37.715	63.0
10	3730.350	37.111	55.5
11	3433.394	34.157	57.0
12	2951.856	29.366	50.5
13	2436.137	24.236	49.4
14	2375.037	23.628	59.8
15	2221.218	22.098	45.3
16	2152.854	21.418	45.4
17	1839.662	18.302	39.1

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

INOVA-400 "fid"

PULSE SEQUENCE

Pulse 47.6 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

64 repetitions

OBSERVE H1, 399.7532376 MHz

DATA PROCESSING

FT size 16384

Total time 1 min, 45 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	15450.390	153.708	19.1
2	13916.071	138.444	45.4
3	12068.176	120.060	7.0
4	11747.731	116.872	6.7
5	11530.287	114.709	80.6
6	11246.465	111.885	76.7
7	7771.170	77.311	42.8
8	7739.125	76.992	43.6
9	7707.081	76.674	43.4
10	4882.593	48.574	41.0
11	4826.134	48.013	68.8
12	3672.534	36.536	79.0
13	3419.993	34.024	99.5
14	3111.756	30.957	100.0
15	2930.171	29.151	89.0
16	2402.964	23.906	98.8
17	2233.586	22.221	98.2
18	2149.660	21.386	91.4
19	1907.801	18.980	98.8

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

INOVA-400 "fid"

PULSE SEQUENCE

Pulse 51.3 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

96 repetitions

OBSERVE H1, 399.7532388 MHz

DATA PROCESSING

FT size 16384

Total time 3 min, 30 sec

16

INDEX	FREQUENCY	PPM	HEIGHT
1	15000.242	149.229	19.3
2	13987.790	139.157	31.5
3	13361.450	131.931	40.2
4	12686.176	126.208	67.8
5	11482.984	114.238	65.2
6	11408.976	113.502	60.7
7	7771.933	77.319	40.1
8	7739.888	77.000	41.4
9	7707.844	76.681	39.8
10	5118.348	50.920	70.1
11	5040.526	50.145	28.6
12	3805.290	37.857	70.5
13	3517.653	34.995	66.1
14	3463.482	34.456	77.1
15	2943.905	29.287	58.0
16	2412.120	23.997	74.0
17	2289.283	22.775	84.4
18	2264.105	22.524	54.8
19	2066.498	20.558	80.9

16

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

INOVA-400 "fid"

PULSE SEQUENCE

Pulse 47.6 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

48 repetitions

OBSERVE H1, 399.7532382 MHz

DATA PROCESSING

FT size 16384

Total time 1 min, 45 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	15277.960	151.992	12.7
2	13008.909	129.419	25.1
3	12822.746	127.567	21.1
4	12612.169	125.472	24.7
5	7771.170	77.311	35.0
6	7739.125	76.992	35.9
7	7707.081	76.674	34.8
8	5886.652	58.563	23.1
9	5073.333	50.472	7.1
10	4146.334	41.250	31.6
11	3589.371	35.709	24.5
12	3207.127	31.906	35.2
13	2948.482	29.333	18.3
14	2357.949	23.458	34.1
15	2306.831	22.949	22.4
16	2209.935	21.985	27.9
17	1874.231	18.646	31.8

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

INOVA-400 "fid"

PULSE SEQUENCE

Pulse 47.6 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

64 repetitions

OBSERVE H1, 399.7532376 MHz

DATA PROCESSING

FT size 16384

Total time 1 min, 45 sec

18

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

INOVA-400 "fid"

PULSE SEQUENCE

Pulse 59.7 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

48 repetitions

OBSERVE H1, 399.7532345 MHz

DATA PROCESSING

FT size 16384

Total time 1 min, 45 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	17118.990	170.308	38.1
2	15811.272	157.298	25.9
3	12990.598	129.236	63.2
4	7771.933	77.319	90.9
5	7739.889	77.000	95.3
6	7707.844	76.681	95.9
7	7464.459	74.260	49.7
8	7346.200	73.083	73.5
9	5531.112	55.026	66.6
10	4768.149	47.436	46.2
11	3967.038	39.466	41.9
12	3639.727	36.210	54.1
13	3418.468	34.009	50.0
14	2776.816	27.625	70.5
15	2409.068	23.967	59.9
16	2241.979	22.304	61.9
17	2213.750	22.023	53.3
18	2139.742	21.287	65.1
19	1671.283	16.627	44.7

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

INOVA-400 "fid"

PULSE SEQUENCE

Pulse 59.7 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

64 repetitions

OBSERVE H1, 399.7532376 MHz

DATA PROCESSING

FT size 16384

Total time 7 min, 1 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	17141.115	170.528	21.0
2	15693.012	156.121	23.0
3	12849.450	127.832	42.1
4	7771.933	77.319	125.5
5	7739.889	77.000	126.5
6	7707.844	76.681	123.0
7	7477.429	74.389	41.7
8	7359.170	73.212	43.5
9	5503.645	54.753	42.7
10	4703.297	46.791	30.2
11	3929.653	39.094	25.8
12	3768.668	37.492	28.4
13	3403.971	33.864	25.0
14	2782.920	27.686	32.5
15	2592.942	25.796	136.4
16	2412.120	23.997	34.6
17	2251.135	22.395	35.2
18	2235.875	22.244	27.1
19	2153.475	21.424	38.3
20	1806.327	17.970	17.1
21	1684.253	16.756	30.1
22	-413.894	-4.118	29.6
23	-448.227	-4.459	34.8

STANDARD 1H OBSERVE

Pulse Sequence: zgpg30

Solvent: CDCl3

Ambient temperature

INOVA-400 "fid"

PULSE SEQUENCE

Pulse 59.7 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

48 repetitions

OBSERVE H1, 399.7532370 MHz

DATA PROCESSING

FT size 16384

Total time 3 min, 30 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	15590.655	155.103	16.8
2	13260.984	131.926	39.4
3	7771.933	77.319	78.4
4	7739.889	77.000	81.3
5	7707.845	76.681	81.8
6	7482.393	74.438	37.6
7	7110.073	70.734	37.9
8	5504.060	54.757	39.3
9	4704.869	46.806	22.4
10	3940.011	39.197	32.5
11	3825.950	38.062	34.6
12	3770.636	37.512	33.4
13	2779.944	27.656	36.1
14	2595.309	25.819	115.8
15	2415.253	24.028	35.5
16	2250.074	22.385	56.3
17	1808.325	17.990	15.4
18	1676.335	16.677	30.3
19	-408.429	-4.063	28.0
20	-448.103	-4.458	29.3

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

INOVA-400 "fid"

PULSE SEQUENCE

Pulse 59.7 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

69 repetitions

OBSERVE H1, 399.753233 MHz

DATA PROCESSING

FT size 16384

Total time 3 min, 30 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	20602.678	204.965	20.3
2	17151.033	170.626	23.3
3	12466.443	124.022	39.9
4	7771.933	77.319	67.2
5	7739.889	77.000	69.7
6	7707.844	76.681	68.9
7	7372.141	73.341	45.4
8	5469.312	54.411	51.0
9	4910.823	48.855	29.6
10	4467.542	44.445	43.2
11	3970.853	39.504	33.7
12	3677.875	36.589	40.2
13	2853.875	28.392	40.5
14	2592.942	25.796	124.4
15	2377.787	23.655	41.9
16	2202.305	21.910	38.4
17	2169.498	21.583	33.5
18	2029.876	20.194	38.0
19	1820.061	18.107	18.4
20	-464.250	-4.619	38.9
21	-470.353	-4.679	38.4

23b

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

INOVA-400 "fid"

PULSE SEQUENCE

Pulse 47.6 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

16 repetitions

OBSERVE H1, 399.7532400 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 26 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	14315.101	142.413	42.7
2	14062.561	139.901	14.5
3	13875.635	138.041	13.1
4	13460.583	133.912	93.5
5	12973.050	129.062	52.3
6	12843.346	127.772	93.3
7	9930.355	98.792	45.0
8	7771.933	77.319	73.8
9	7739.889	77.000	75.3
10	7707.844	76.681	75.8
11	6638.933	66.047	40.0
12	6229.985	61.979	40.3
13	5862.237	58.320	32.0
14	3028.594	30.130	39.8
15	3000.364	29.849	36.0
16	2536.483	25.234	41.6
17	1936.794	19.268	39.0
18	-281.139	-2.797	26.7
19	-309.368	-3.078	26.0

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

INOVA-400 "fid"

PULSE SEQUENCE

Pulse 47.6 degrees

Acq. time 1.638 sec

Width 5000.0 Hz

4 repetitions

OBSERVE H1, 399.7532412 MHz

DATA PROCESSING

FT size 16384

Total time 0 min, 6 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	14115.205	140.425	13.1
2	14025.939	139.537	31.2
3	13838.250	137.669	13.4
4	13467.450	133.980	88.1
5	12979.917	129.130	45.9
6	12846.398	127.802	74.4
7	9920.436	98.693	42.9
8	7771.933	77.319	73.6
9	7739.889	77.000	74.3
10	7707.844	76.681	73.0
11	6580.185	65.463	33.9
12	6241.430	62.093	36.6
13	3069.031	30.532	38.3
14	2979.001	29.636	28.0
15	2550.216	25.371	38.2
16	1955.105	19.450	37.0
17	120.943	1.203	23.2
18	-313.946	-3.123	25.3
19	-320.050	-3.184	23.9