APR 30 2002

Efficient Synthesis of 4-(2'-Alkenyl)-2,5-dihydrofurans and 5,6-Dihydro-2H-pyrans via the Pd-Catalyzed Cyclizative Coupling Reaction of 2,3- or 3,4-Allenols with Allylic Halides

Shengming Ma* and Wenzhong Gao

State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, 354 Fenglin Lu, Shanghai 200032, P. R. China

Supporting Information

¹H and ¹³C NMR spectra

Pulse Sequence: szpu1

HCHT2

```
Archive directory: /export/home/masm/vm1/sys/data
Sample directory:
Pulse Sequence: s2pul
```

HEFTZ

2237.70
2230.65
2203.70
2202.59
2194.88
2184.20
2177.90
2170.42
2163.12

1572.41.

1345.00
1346.00
1347.00
1348.00
1349.00
1350.00

513 74
537 45
534 11
530 14
526 77
520 59
514 30
509 47
504 88
501 86
494 43
490 84
485 57
484 24
483 74
483 65
478 37
471 50
467 43
427 69
420 47
411 62
409 59
405 27
390 26
387 64
381 16
330 34
275 84
264 47
163 74
68 84

HEHTL

$n\text{-C}_6\text{H}_{13}$

1m

HC

Hz

2150.83

1574.01
1567.58
1561.04
1556.12
1554.44
1552.98
1549.76
1548.36
1546.67
1543.39

583.44
578.99
576.22
461.09
450.31
442.14
417.57
412.24
408.47
393.18
389.88
386.41
382.73
379.75
375.00
373.64
248.94
241.85
234.71

$n\text{-C}_4\text{H}_9$

1p

HO

HERTZ	3234.35
3144.51	
3029.85	
2945.54	
2898.54	
2847.95	
2822.84	
2737.32	
2716.67	
2674.24	
2577.23	
2428.18	
2368.51	
2346.02	
2340.14	
2337.31	
2326.34	
2322.14	
2318.27	
2312.86	
2301.54	
2291.67	
2230.52	
2284.52	
2282.22	
2276.75	
2269.24	
2258.38	
2199.44	
2179.27	
2176.93	
2158.65	
2133.85	
2082.95	
2050.28	
2038.08	
2020.12	
2000.51	
1989.11	
1984.12	
1960.74	
1924.22	
1918.81	
1912.74	
1905.19	
1889.00	
1836.49	
1743.87	
1737.22	
1710.64	
1676.91	
1646.50	
1621.21	
1507.42	
1430.91	
1373.26	
1356.93	
1298.07	
1233.22	
1210.31	
1087.80	
1081.69	
1077.05	
1070.88	
1064.36	
1057.26	
1053.32	
1046.41	
1009.11	
971.64	
868.54	
665.43	
660.38	
653.83	
535.19	
592.13	
589.12	
580.95	
577.66	
483.87	
479.58	
469.84	
463.53	
457.32	
433.74	
430.62	
426.00	
419.00	
415.31	
412.30	
405.32	
398.88	
383.68	
364.51	
316.59	
312.74	
304.67	
307.43	
278.12	
271.07	
264.11	
239.05	
215.52	
208.72	
168.92

7.45194
7.34672
7.33274
7.25594
7.24792
7.25183
7.24673
7.23693
7.23150
7.22750
7.18022
7.17542
7.16261
7.14850
7.11446

5.12806
5.12075
5.09308
5.08210
5.07440
5.04958
5.03864

4.40701

3.67864
3.65416
3.64234
3.63246
3.62842
3.61377
3.51166
3.50311
3.49333
3.48564
3.48410

2.86314
2.84302
2.83217
2.82122
2.80001

1.63001

1.21044
1.10900
1.09136
1.08036
1.03865

Hz

Hz

2158.38

1541.41
1538.58
1535.66

1104.24
1103.58
1097.80
1091.80

645.87
642.82
639.78
564.08
471.29
392.88
391.51
389.42
387.78
284.61
284.00
282.92
281.67
277.86
277.26
276.16
274.89
255.76
248.71
241.45

6702-11-3

4a

5a

5a

HEATZ

4c

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
Mercury-300B8 "MERCURYvx300"

Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.000 sec
Width 25000.0 Hz
352 repetitions
OBSERVE C13, 75.4433982 MHz
DECOUPLE H1, 300.0346884 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 65536
Total time 78 hr, 29 min, 45 sec

nmr2

HERTZ

4g

18.79

HEHTZ

2208.03
2200.66
2199.59
2198.27
2197.71

1588.54
1543.64
1528.22
1488.41
1447.84
1442.14
1435.37
1430.12
1402.53
1390.25

944.82
909.33
899.67
872.45
867.38
856.99

477.33
473.12
465.06
439.78
432.89
426.05
413.22
398.90
398.77
393.73
385.26
377.07
370.82
363.85
357.52
253.06
235.95
239.78

HEX-72

9WZ-5-20C
13C OBSERVE

pad=2 run with findz0 before acquisition

Automation directory: /export/home/ASM/AUTO/auto_11.12.01
File : gwz-5-20c02

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Sample #3

File: gwz-5-20c01

Mercury-300BB "MERCURYvx300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.815 sec

Width 18867.9 Hz

600 repetitions

OBSERVE C13, 75.4433982 MHz

DECOUPLE H1, 300.0346884 MHz

Power 35 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 29 min, 38 sec

180.12
169.11
1606.21
1566.65
1522.77

1432.69
1433.59

1357.51
1357.76
1341.66
1347.88
1340.55
1339.82
1332.73
1331.73
1330.45
1319.11
1312.42
1301.81

1246.35

1167.14
1154.58
1151.49
1148.51
1145.91
1141.82
1135.10
1131.56
1128.96
1125.04
1118.50
1101.97
1098.19
1089.31
1082.83
1041.23
1034.87
1025.60
1021.41
1014.62
1013.19
1003.93
990.17
988.73
954.71

1292.57

1227.65

1120.55
1121.71
1114.54
1097.81
1090.51

1036.65

1008.91

972.57

948.41

844.78

887.74

830.73

811.57

792.08

590.21

134.48

39.11

106.74

HERTZ

2126.52

16.91

4m

gwz-7-62c
13C OBSERVE

pad-2 run with findz0 before acquisition

Automation directory: /export/home/ASH/AUTO/auto_11.12.01
File : gwz-7-62c02

Pulse Sequence: s2pu1

Solvent: cdcl3

Ambient temperature

Sample #2

File: gwz-7-62c01

Mercury-300BB "MERCURYvx300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.815 sec

Width 18867.9 Hz

320 repetitions

OBSERVE C13, 75.4433982 MHz

DECOUPLE H1, 300.0346884 MHz

Power 35 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 29 min, 38 sec

200
150
100
50
0 PPM

- 141.250
- 134.965
- 120.647
- 116.803
- 86.881
- 74.772
- 34.267
- 32.100
- 31.870
- 29.682
- 24.864
- 22.864
- 14.323

4p

200

150

100

50

0PPM

140.095

135.030

127.830

116.311

88.117

85.002

77.423

77.000

76.577

38.855

37.963

34.427

31.702

26.982

25.555

23.697

23.622

22.878

14.082

4q

4q

4q

145.972
135.859
123.323
116.465
88.795
83.228
77.683
77.251
76.832
37.423
36.692
34.866
31.326
27.829
25.826
23.110
22.825
22.348
14.380

4r

QW2-7-81

180
160
140
120
100
80
60
40
20
PPM

- 138.591
- 134.635
- 123.812
- 116.382
- 86.671
- 77.347
- 76.930
- 76.506
- 76.298
- 35.951
- 31.597
- 27.368
- 22.726
- 13.986

GWZ-8.1

5s

HERTZ

4t

HERTZ

HERTZ

1422.00

1375.93

797.53

4v

5.716
5.693
5.660

5.037
5.032
4.980
4.974
4.968
4.946
4.940

3.938
3.695
3.682
3.660
3.647
3.525
3.512
3.488
3.475

2.741
2.720
2.688
2.668
2.632
2.610
2.580
2.559
2.172
2.158
2.145
2.124
1.870

1.357
1.335
1.321
1.311
1.302
1.289
1.278
1.270
1.263
1.257
1.249
1.051
1.028
0.914
0.892
0.869

ppm

ppm

135.825
134.860

126.384

115.060

77.450
77.028
76.604
71.103
68.143

33.050
31.437
30.768
29.268
26.867
22.877
16.850
14.021
13.945

6b

7.44378
7.44358
7.39580
7.39586
7.31548
7.31558
7.31540
7.31544
7.27775
7.27279
7.26434
7.25403
7.24938
7.24006
7.22406
7.22523
7.22052
7.14123
7.13127
7.11313
7.10979
6.27885
6.06354
5.75305
5.73170
5.72164
5.71056
5.70312
5.69505
5.68348
5.68418
5.67853
5.67330
5.67351
5.65244
5.64187
5.63036
5.44321
5.33484
5.24427
5.01372
5.00436
5.00235
4.99930
4.99431
4.96111
4.95601
4.95073
4.92752
4.44585
4.34397
4.22104
4.18321
4.07993
4.03811
4.03665
4.03060
3.91302
3.90516
3.88354
3.86440
3.74412
3.67466
3.66513
3.64203
3.62870
2.95479
2.64285
2.62214
2.59273
2.57128
2.54835
2.43586
2.44135
1.69288
1.63015
1.42404
1.26442
1.10447
.98347
.97548
.91863
.83552
.83093
.70560
.64282

Pulse Sequence: s2pu1
Solvent: CDC13
Ambient temperature
Mercury-30088 "MERCURYVX300"

Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.000 sec
Width 18797.0 Hz
204 repetitions
OBSERVE C13, 75.443382 MHz
DECUPLE H1, 300.0346884 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 65536
Total time 73 hr, 29 min, 45 sec

140.732
137.394
136.421
129.767
128.748
128.330
126.901
— 116.004

— 71.431
— 68.230

34.750
34.507

— 17.315

142.489

136.712

128.570

116.096

71.306

68.864

35.576

35.326

31.337

31.299

20.187

Hz

Integral

ppm

9

8

7

6

5

4

3

2

1

0.961

0.178

0.168

0.545

1.529

1.232

2.628

1.080

0.158

0.199

1.507

1.380

1.449

10.413

3.529

4.340

4aa : 4ab = 14:86

4aa

4ab

Hz

2879.2

2295.2

2288.7

2282.6

2282.1

2278.0

1986.9

1985.1

1982.9

1969.2

1967.6

1965.9

1964.3

1958.7

1957.3

1909.4

1905.7

1785.4

1781.8

1779.8

1768.9

1768.2

1767.4

1766.5

1275.3

860.0

845.0

643.2

631.6

497.1

493.9

490.1

471.3

470.4

465.0

464.1

424.9

424.2

417.8

417.1

341.3

336.2

334.5

329.3

Integral
ppm

4ja : 4jb = 15:85

H₂

0.0