Stereoselective Synthesis of Enynes by Nickel-Catalyzed Cross-Coupling of Divinylic Chalcogenides with Alkynes.

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Table of Contents Graphic

(E)-1,4-Diphenyl-1-buten-3-yne (11): mp 97-98°C (mp 96-97 °C)^{12h}; (KBr, cm⁻¹) 3033, 1956, 1593, 1487, 1440; ¹H NMR (CDCl₃) δ 7.47-7.28 (m, 10 H), 7.03 (d, *J* = 16.0 Hz, 1H), 6.37 (d, *J* = 16.0 Hz, 1H), ¹³C NMR (CDCl₃) δ 141.24, 136.32, 131.49, 128.71, 128.58, 128.31, 128.14, 126.28, 123.42, 108.13, 91.74, 88.89; EIMS *m/z* (rel int) 204 (base), 203 (90), 202 (91), 101 (42), 89 (17), 76 (14), 63 (7), 51 (6).

(Z)-Dec-2-en-4-yn-1-ol (14)^{3f} (oil); IR (neat, cm⁻¹) 3321, 2956, 2929, 2859, 2217, 1624, 1470, 1017, 722; ¹H NMR (CDCl₃) δ 5.99 (dt, *J* = 10.8, 6.3 Hz, 1H), 5.57 (d, *J* = 10.8 Hz, 1H), 4.37 (d, *J* = 6.2 Hz, 2H), 2.33 (td, *J* = 8.0, 1.9 Hz, 2H), 1.83 (s, 1H), 1.57-1.47 (m, 2H), 1.36-1.32 (m, 4H), 0.90 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (CDCl₃) δ 139.8, 111.2, 96.8, 76.2, 60.9, 31.0, 28.3, 22.1, 19.4, 13.9; EIMS *m/z* (rel int) 152, 123, 109, 95 (base), 81, 67.

Current Data Parameters
 NAME cBuc1791
 EXPNO 2
 PROCNO 2002

F2 - Acquisition Parameters
 Date_ 20020618
 Time 16.08
 INSTRUM spect
 PROBHD 5 mm Dual 13
 PULPROG dept135
 TD 65536
 SOLVENT CDCl3
 NS 27
 DS 1
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 16384
 DW 21.600 usec
 DE 4.50 usec
 TE 298.0 K
 P1 7.60 usec
 DELTA 0.0000097 sec
 D1 1.00000000 sec
 D12 0.00002000 sec
 PL2 -3.00 dB
 P3 11.80 usec
 SFO2 400.1316005 MHz
 NUC2 13
 D2 0.00357143 sec
 P4 23.60 usec
 SFO1 100.6237964 MHz
 NUC1 13C
 PL1 -6.00 dB
 P2 15.20 usec
 PL12 16.00 dB
 DE 4.50 usec
 CPDPRG2 waltz16
 PCPD2 106.00 usec

F2 - Processing parameters
 SI 32768
 SF 100.6127950 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 21.00 cm
 F1P 168.096 ppm
 F1 16912.80 Hz
 F2P -3.102 ppm
 F2 -312.08 Hz
 PPMCM 8.15237 ppm/cm
 HZCM 820.23224 Hz/cm

Current Data Parameters
 NAME cBuc2001
 EXPNO 1
 PROCNO 2001

F2 - Acquisition Parameters
 Date_ 20010632
 Time 10.27
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 2408.478 Hz
 FIDRES 0.073501 Hz
 AQ 6.8026967 sec
 RG 20.2
 DW 207.600 usec
 DE 7.07 usec
 TE 300.0 K
 D1 0.00100000 sec
 P1 7.00 usec
 DE 7.07 usec
 SFO1 200.1310007 MHz
 NUC1 1H
 PL1 -3.00 dB

F2 - Processing parameters
 SI 32768
 SF 200.1300367 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 22.00 cm
 F1P 9.399 ppm
 F1 1880.94 Hz
 F2P -0.345 ppm
 F2 -69.02 Hz
 PPMCM 0.44288 ppm
 HZCM 88.63455 Hz/

Current Data Parameters
 NAME cbu10701
 EXPNO 1
 PROCNO 2002

F2 - Acquisition Parameters
 Date_ 20030620
 Time 19.34
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 FIDRES 0.163111 Hz
 AQ 2.9884915 sec
 RG 16384
 DW 45.600 usec
 DE 5.00 usec
 TE 300.2 K
 P1 10.00 usec
 DELTA 0.000140 sec
 D1 0.000000 sec
 D12 0.000000 sec
 PL1 0.00 dB
 PL2 0.00 dB
 PL12 0.00 dB
 SF02 200.630005 MHz
 NUC2 13C
 SP 0.0057143 sec
 P4 14.00 usec
 SF01 50.326589 MHz
 NUC1 1H
 PL1 -3.00 dB
 PL2 22.00 usec
 PL12 30.00 dB
 DE 5.00 usec
 CPOPM2 waltz16
 POP2 110.00 usec

F2 - Processing parameters
 SI 32768
 SF 50.3267429 MHz
 MDW 0
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 22.00 cm
 F1P 154.453 ppm
 F1 7772.49 Hz
 F2P 90.407 ppm
 F2 4049.50 Hz
 PPMCM 2.91118 ppm/cm
 HZCM 146.49860 Hz/cm

Current Data Parameters
 NAME c8s56ah1
 EXPNO 1
 PROCNO 2001

F2 - Acquisition Parameters
 Date_ 20010912
 Time 16.19
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 2408.478 Hz
 FIDRES 0.073501 Hz
 AQ 6.8026867 sec
 RG 20.2
 DW 207.600 usec
 DE 7.07 usec
 TE 300.2 K
 D1 0.00100000 sec
 P1 7.00 usec
 DE 7.07 usec
 SF01 200.1310007 MHz
 NUC1 1H
 PL1 -3.00 dB

F2 - Processing parameters
 SI 32768
 SF 200.1300155 MHz
 MDW 0
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 22.00 cm
 F1P 9.339 ppm
 F1 1869.10 Hz
 F2P -0.710 ppm
 F2 -142.10 Hz
 PPMCM 0.45679 ppm
 HZCM 91.41832 Hz/cm

Current Data Parameters
NAME c8142b01
EXPNO 1
PROCNO 2001

F2 - Acquisition Parameters
Date_ 20010728
Time 15.28
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 2408.478 Hz
FIDRES 0.073501 Hz
AQ 6.8026867 sec
RG 18
DW 207.500 usec
DE 7.07 usec
TE 300.0 K
D1 0.00100000 sec
P1 7.00 usec
DE 7.07 usec
SF01 200.1310007 MHz
NUC1 1H
PL1 -3.00 dB

F2 - Processing parameters
SI 32768
SF 200.1300179 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 22.00 cm
F1P 8.429 ppm
F1 1686.81 Hz
F2P -0.457 ppm
F2 -91.41 Hz
PPMCM 0.40388 ppm
HZCM 80.82827 Hz/

Current Data Parameters
NAME c8142b03
EXPNO 1
PROCNO 2001

F2 - Acquisition Parameters
Date_ 20010730
Time 12.39
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2155
DS 0
SWH 11037.527 Hz
FIDRES 0.168419 Hz
AQ 2.9688308 sec
RG 32768
DW 45.300 usec
DE 5.00 usec
TE 300.0 K
D12 0.00002000 sec
PL13 20.00 dB
D1 2.00000000 sec
CPDPRG2 waltz16
PCPD2 110.00 usec
SF02 200.1308005 MHz
NUC2 13C
PL2 -3.00 dB
PL12 20.00 dB
P1 11.00 usec
DE 5.00 usec
SF01 50.3279929 MHz
NUC1 13C
PL1 -3.00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 50.3227336 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 22.00 cm
F1P 215.000 ppm
F1 10819.39 Hz
F2P -5.000 ppm
F2 -251.61 Hz
PPMCM 10.00000 ppm/cm
HZCM 503.22736 Hz/cm

Current Data Parameters
 NAME c8105n1
 EXPNO 1
 PROCNO 2002

F2 - Acquisition Parameters
 Date_ 20020700
 Time 16.44
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 2408.478 Hz
 FIDRES 0.073501 Hz
 AQ 6.8026867 sec
 RG 35.9
 DW 207.600 usec
 DE 7.07 usec
 TE 300.0 K
 D1 0.00100000 sec
 P1 7.00 usec
 DE 7.07 usec
 SFO1 200.1310007 MHz
 NUC1 ¹H
 PL1 -3.00 dB

F2 - Processing parameters
 SI 32768
 SF 200.1300106 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 22.00 cm
 F1P 8.979 ppm
 F1 1797.05 Hz
 F2P -6.623 ppm
 F2 -124.76 Hz
 PPMCM 0.43649 ppm
 HZCM 87.35528 Hz

Current Data Parameters
 NAME c8uc19cd
 EXPNO 1
 PROCNO 2002

F2 - Acquisition Parameters
 Date_ 20020618
 Time 15.50
 INSTRUM spect
 PROBHD 5 mm Dual 13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 54
 DS 0
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 32768
 DW 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 D12 0.00002000 sec
 PL13 16.00 dB
 D1 2.00000000 sec
 CPDPRG2 waltz16
 PCPD2 106.00 usec
 SFO2 400.1316005 MHz
 NUC2 ¹³C
 PL2 -3.00 dB
 PL12 16.00 dB
 P1 7.60 usec
 DE 4.50 usec
 SFO1 100.6237964 MHz
 NUC1 ¹H
 PL1 -6.00 dB
 D11 0.03000000 sec

F2 - Processing parameters
 SI 32768
 SF 100.6127771 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 21.00 cm
 F1P 179.524 ppm
 F1 18062.42 Hz
 F2P 1.358 ppm
 F2 136.72 Hz
 PPMCM 8.48406 ppm/cm
 HZCM 853.60437 Hz/cm

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