**Highly Regio- and Chemoselective Palladium-Catalyzed
Three-Component Assembly of Arylethylidene Malononitriles,
Allylic Chlorides and Allenylstannanes: A Novel Route to 1,7-Enyne
Derivatives**

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Supporting Information

Experimental Section

General Procedure for the Three-Component Assembly of Arylethylidene Malononitriles, Allylic Chlorides, and Allenylstannanes. A 25-mL round-bottom side-arm flask containing Pd(PPh₃)₄ (0.050 mmol) and arylethylidene malononitrile (1.00 mmol) was evacuated and purged with nitrogen gas several times. To the flask were then added toluene (2.00 mL), allylic chloride (1.00 mmol), and allenylstannane (1.20 mmol) via syringe. The reaction mixture was stirred at ambient temperature for 8 h. The reaction mixture was then treated with 10 mL of saturated KF solution and stirred for 30 min. at ambient temperature. The solution was filtered through a Celite and silica gel pad and washed with 50 mL of dichloromethane. The organic layer was washed with water, dried (MgSO₄) and concentrated *in vacuo*. The residue was purified on a silica gel column (hexane/EtOAc = 20/1) to give pure product.

Compounds **4a-j**, **6a-c**, and **10** were prepared by following this procedure. Spectral data of **4a-j**, **6a-c** and **10** are listed below:

2-(1-Phenyl-3-butyryl)-2-(2-propenyl)propanedinitrile (4a): pale yellow oil; ¹H NMR (500 MHz, CDCl₃): δ 7.42 - 7.36 (m, 5H), 5.89 – 5.83 (m, 1H), 5.39 (d, *J* = 10.5 Hz, 1H), 5.31 (d, *J* = 17.0 Hz, 1H), 3.26 (dd, *J* = 10.5, 4.5 Hz, 1H), 3.05 - 3.01 (m, 2H), 2.53 (dd, *J* = 14.0, 7.5 Hz, 1H), 2.42 (dd, *J* = 14.0, 7.5 Hz, 1H), 1.90 (t, *J* = 3.0 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 134.55, 129.31, 129.12, 128.77, 128.38, 123.46, 114.74, 113.86, 79.27, 71.67, 50.00, 42.66, 40.59, 22.41; HRMS calcd for C₁₆H₁₄N₂ 234.1157, found 234.1156.

2-(2-Methyl-2-propenyl)-2-(1-phenyl-3-butyryl)propanedinitrile (4b): pale yellow oil; ¹H NMR (500 MHz, CDCl₃): δ 7.43 - 7.37 (m, 5H), 5.12 (s, 1H), 5.10 (s, 1H), 3.28 (dd, *J* = 10.5, 4.5 Hz, 1H), 3.10 - 3.01 (m, 2H), 2.50 (d, *J* = 14.0 Hz, 1H), 2.29 (d, *J* = 14.0 Hz, 1H), 1.90 (t, *J* = 2.5 Hz, 1H), 1.88 (s, 3H); ¹³C NMR (125 MHz,

CDC₃): δ 137.15, 134.62, 129.27, 129.08, 128.82, 118.63, 115.13, 114.24, 79.31, 71.57, 51.45, 44.18, 41.81, 23.02, 22.31; HRMS calcd for C₁₇H₁₆N₂ 248.1313, found 248.1303.

2-(1-Methyl-2-butenyl)-2-(1-phenyl-3-butyryl)propanedinitrile (4c): pale yellow oil; ¹H NMR (400 MHz, CDC₃): δ 7.44 – 7.38 (m, 5H), 5.74 – 5.69 (m, 1H), 5.47 – 5.41 (m, 1H), 3.29 (dd, J = 10.4, 4.4 Hz, 1H), 2.93 – 2.91 (m, 2H), 2.57 – 2.53 (m, 1H), 1.89 (t, J = 3.0 Hz, 1H), 1.75 (d, J = 6.8 Hz, 3H), 1.31 (d, J = 6.8 Hz, 3H); ¹³C NMR (100 MHz, CDC₃): δ 135.82, 132.55, 129.39, 129.08, 128.98, 127.94, 114.62, 113.71, 79.81, 71.81, 48.08, 42.37, 42.21, 22.23, 18.34, 17.16; HRMS calcd for C₁₈H₁₈N₂ 262.1470, found 262.1472.

2-(1-Phenyl-3-butyryl)-2-((E)-3-phenyl-2-propenyl)propanedinitrile (4d): pale yellow oil; ¹H NMR (500 MHz, CDC₃): δ 7.45 - 7.28 (m, 10H), 6.57 (d, J = 16.0 Hz, 1H), 6.17 (m, 1H), 3.31 (dd, J = 10.5, 4.5 Hz, 1H), 3.11 - 2.99 (m, 2H), 2.69 (dd, J = 14.0, 7.5 Hz, 1H), 2.60 (dd, J = 14.0, 7.5 Hz, 1H), 1.92 (t, J = 3.0 Hz, 1H); ¹³C NMR (125 MHz, CDC₃): δ 138.02, 135.67, 134.58, 129.34, 129.16, 128.82, 128.67, 128.49, 126.69, 118.88, 114.88, 113.96, 79.28, 71.72, 50.04, 42.95, 40.07, 22.54; HRMS calcd for C₂₂H₁₈N₂ 310.1470, found 310.1460.

2-(1-(4-Chlorophenyl)-3-butyryl)-2-(2-methyl-2-propenyl)propanedinitrile (4e): pale yellow oil; ¹H NMR (500 MHz, CDC₃): δ 7.40 (d, J = 7.5 Hz, 2H), 7.32 (d, J = 7.5 Hz, 2H), 5.13 (s, 1H), 5.02 (s, 1H), 3.27 (dd, J = 11.0, 4.5 Hz, 1H), 3.09 - 2.93 (m, 2H), 2.50 (d, J = 14.5 Hz, 1H), 2.31 (d, J = 14.5 Hz, 1H), 1.92 (t, J = 2.5 Hz, 1H), 1.89 (s, 3H); ¹³C NMR (125 MHz, CDC₃): δ 136.91, 135.35, 133.11, 130.17, 129.34, 118.86, 114.80, 114.06, 78.92, 71.93, 50.73, 44.08, 41.72, 23.01, 22.12; HRMS calcd for C₁₇H₁₅ClN₂ 282.0924, found 282.0917.

2-(1-(4-(Methoxy)phenyl)-3-butyryl)-2-(2-methyl-2-propenyl)propanedinitrile

trile (4f): pale yellow oil; ^1H NMR (500 MHz, CDCl_3): δ 7.30 (d, $J = 7.5$ Hz, 2H), 6.92 (d, $J = 7.5$ Hz, 2H), 5.11 (s, 1H), 5.01 (s, 1H), 3.81 (s, 3H), 3.24 (dd, $J = 11.5$, 5.0 Hz, 1H), 3.09 – 3.00 (m, 2H), 2.50 (d, $J = 14.5$ Hz, 1H), 2.31 (d, $J = 14.5$ Hz, 1H), 1.86 (t, $J = 3.0$ Hz, 1H), 1.85 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 160.13, 137.24, 129.98, 126.39, 118.57, 115.24, 114.41, 114.34, 79.51, 71.44, 55.25, 50.88, 44.17, 42.05, 23.06, 22.36; HRMS calcd for $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}$ 278.1419, found 278.1420.

2-(2-Methyl-2-propenyl)-2-(1-(4-nitrophenyl)-3-butynyl)propanedinitrile

(4g): pale yellow oil; ^1H NMR (500 MHz, CDCl_3): δ 8.29 (d, $J = 7.5$ Hz, 2H), 7.60 (d, $J = 7.5$ Hz, 2H), 5.16 (s, 1H), 5.05 (s, 1H), 3.42 (dd, $J = 11.5$, 4.5 Hz, H), 3.13 - 2.98 (m, 2H), 2.59 (d, $J = 14.0$ Hz, 1H), 2.35 (d, $J = 14.5$ Hz, 1H), 1.93 (t, $J = 3.0$ Hz, 1H), 1.91 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 148.47, 141.85, 136.54, 130.04, 124.22, 119.33, 114.32, 113.78, 78.25, 72.55, 50.75, 44.12, 41.49, 23.01, 21.91; HRMS calcd for $\text{C}_{17}\text{H}_{15}\text{N}_3\text{O}_2$ 293.1164, found 293.1153.

2-(2-Methyl-2-propenyl)-2-(1-(1-naphthyl)-3-butynyl)propanedinitrile (4h):

pale yellow oil; ^1H NMR (500 MHz, CDCl_3): δ 7.91 - 7.84 (m, 4H), 7.54 - 7.50 (m, 3H), 5.12 (s, 1H), 5.01 (s, 1H), 3.47 (dd, $J = 10.0$, 5.0 Hz, 1H), 3.18 - 3.14 (m, 2H), 2.54 (d, $J = 14.5$ Hz, 1H), 2.31 (d, $J = 14.5$ Hz, 1H), 1.87 (s, 3H), 1.86 (t, $J = 2.5$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 137.14, 133.49, 133.09, 131.94, 129.11, 128.88, 128.20, 127.77, 126.90, 126.75, 125.48, 118.71, 115.22, 114.32, 79.32, 71.70, 51.74, 44.31, 41.84, 23.06, 22.47; HRMS calcd for $\text{C}_{21}\text{H}_{18}\text{N}_2$ 298.1470, found 298.1473.

2-(1-(2-Furanyl)-3-butynyl)-2-(2-methyl-2-propenyl)propanedinitrile (4i):

pale yellow oil; ^1H NMR (500MHz, CDCl_3): δ 7.45 (d, $J = 2.0$ Hz, 1H), 6.50 (dd, $J = 5.5$, 3.5 Hz, 1H), 6.41 (d, $J = 3.0$ Hz, 1H), 5.13 (s, 1H), 5.04 (s, 1H), 3.50 (t, $J = 4.5$ Hz, 1H), 3.00 - 2.98 (m, 2H), 2.50 (d, $J = 14.5$ Hz, 1H), 2.36 (d, $J = 14.5$ Hz, 1H), 1.99 (t, $J = 3.0$ Hz, 1H), 1.90 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 147.90, 143.37,

136.96, 118.69, 114.59, 113.77, 110.78, 110.64, 78.68, 71.55, 45.26, 43.35, 41.09, 22.95, 21.00; HRMS calcd for C₁₅H₁₄N₂O 238.1106, found 238.1107.

2-(2-Methyl-2-propenyl)-2-(1-(2-thiophenyl)-3-butynyl)propanedinitrile (4j): pale yellow oil; ¹H NMR (500 MHz, CDCl₃): δ 7.35 (d, *J* = 4.5 Hz, 1H), 7.19 (d, *J* = 3.5 Hz, 1H), 7.06 (dd, *J* = 5.0, 3.5 Hz, 1H), 5.14 (s, 1H), 5.05 (s, 1H), 3.60 (dd, *J* = 11.0, 4.0 Hz, 1H), 3.14 - 2.94 (m, 2H), 2.52 (d, *J* = 14.0 Hz, 1H), 2.42 (d, *J* = 14.0 Hz, 1H), 1.97 (t, *J* = 3.0 Hz, 1H), 1.90 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 137.04, 136.46, 128.07, 127.37, 126.42, 118.78, 114.87, 114.07, 78.81, 71.93, 47.27, 43.99, 42.20, 24.24, 23.05; HRMS calcd for C₁₅H₁₄N₂S 254.0878, found 254.0886

2-(1-((*E*)-2-Phenyl-1-ethenyl)-3-butynyl)-2-(2-propenyl)propanedinitrile (6a): pale yellow oil; ¹H NMR (500 MHz, CDCl₃): δ 7.30 (d, *J* = 7.5 Hz, 2H), 7.28 – 7.25 (m, 3H), 6.67 (d, *J* = 15.5 Hz, 1H), 6.00 (dd, *J* = 15.5, 9.5 Hz, 1H), 5.88 (m, 1H), 5.43 (d, *J* = 10.5 Hz, 1H), 5.38 (d, *J* = 17.0 Hz, 1H), 2.84 – 2.63 (m, 5H), 2.07 (t, *J* = 3.0 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 137.95, 135.34, 128.78, 128.28, 126.83, 123.52, 121.95, 114.31, 113.70, 78.94, 72.29, 48.03, 41.48, 40.37, 22.27; HRMS calcd for C₁₈H₁₆N₂ 260.1313, found 260.1315.

2-(2-Methyl-2-propenyl)-2-(1-((*E*)-2-phenyl-1-ethenyl)-3-butynyl)propanedinitrile (6b): pale yellow oil; ¹H NMR (500 MHz, CDCl₃): δ 7.42 (d, *J* = 7.5 Hz, 2H), 7.36 – 7.30 (m, 3H), 6.69 (d, *J* = 15.5 Hz, 1H), 6.04 (dd, *J* = 15.5, 9.5 Hz, 1H), 5.15 (s, 1H), 5.08 (s, 1H), 2.93 - 2.84 (m, 2H), 2.73 - 2.65 (m, 2H), 2.55 (d, *J* = 14.5 Hz, 1H), 2.07 (t, *J* = 3.0 Hz, 1H), 1.94 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 138.04, 137.14, 135.37, 128.77, 128.72, 126.84, 122.10, 118.77, 114.74, 114.06, 79.04, 72.20, 49.54, 43.98, 40.81, 23.10, 22.20; HRMS calcd for C₁₉H₁₈N₂ 274.1470, found 274.1477.

2-(2-Methyl-2-propenyl)-2-(1-((*E*)-2-phenyl-1-ethenyl)-3-butynyl)propanedinitrile (6c): pale yellow oil; ¹H NMR (500 MHz, CDCl₃): δ 7.38 – 7.26 (m, 10H),

6.68 (t, $J = 15.5$ Hz, 2H), 6.20 (m, 1H), 6.07 (dd, $J = 16.0, 9.5$ Hz, 1H), 2.97 – 2.70 (m, 5H), 2.07 (t, $J = 3.0$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 138.05, 138.01, 135.63, 135.35, 128.81, 128.73, 128.55, 127.39, 126.84, 126.70, 121.94, 118.73, 114.44, 113.78, 78.94, 72.33, 48.05, 41.73, 39.88, 22.34; HRMS calcd for $\text{C}_{24}\text{H}_{20}\text{N}_2$ 336.1626, found 336.1626

2-(3-Deuterio-1-phenylbut-3-ynyl)-2-(2-propenyl)propanedinitrile (10): pale yellow oil; ^1H NMR (500 MHz, CDCl_3): δ 7.42 – 7.36 (m, 5H), 5.89 – 5.83 (m, 1H), 5.39 (d, $J = 10.0$ Hz, 1H), 5.31 (d, $J = 17.0$ Hz, 1H), 3.26 (dd, $J = 10.5, 4.5$ Hz, 1H), 3.08 – 2.96 (m, 2H), 2.52 (dd, $J = 14.0, 7.5$ Hz, 1H), 2.43 (dd, $J = 14.0, 7.5$ Hz, 1H); ^{13}C NMR (100.4 MHz, CDCl_3): δ 134.81, 129.55, 129.36, 129.01, 128.65, 123.68, 115.00, 114.11, 79.07 (t), 71.92, 50.23, 42.91, 40.82, 22.62; HRMS calcd for $\text{C}_{16}\text{H}_{13}\text{DN}_2$ 235.1219, found 235.1221.

The regiochemistry of **4a,b** was established by ^1H - ^1H decoupling experiments. For example, on irradiating the H_a proton at δ 3.26 in **4a**, the multiplet at δ 3.03 (H_b and H_c protons) collapses partially. Irradiation of the two protons (H_b and H_c) multiplet at δ 3.03 causes the signal at δ 3.26 (doublet of a doublet) to a sharp singlet and also converts the triplet at δ 1.90 (H_d proton) to a sharp singlet. Irradiation of the H_d proton at δ 1.90 leads to partial collapse of the multiplet at δ 3.03 (H_b and H_c protons).

Similarly, irradiation of the H_a proton at δ 3.28 in **4b** led to partial collapse of the multiplet at δ 3.05 (H_b and H_c protons). Irradiation of the two protons (H_b and H_c) multiplet at δ 3.05 converts the doublet of a doublet at δ 3.28 to a sharp singlet and also the triplet at δ 1.90 (H_d proton) to a sharp singlet. Irradiation of the H_d proton at δ 1.90 results in partial collapse of the multiplet at δ 3.05 (H_b and H_c protons). The above data strongly confirm the regiochemistry of **4a,b**.

The 1,2-addition product **6a,b** was established by ^1H NMR NOE experiments. For example, selective irradiation of the H_a proton at δ 6.67 in **6a** caused 2.32% enhancement of the ortho protons of the phenyl ring at δ 7.30. Selective irradiation of the H_b proton at δ 6.00 caused 3.30% enhancement of the ortho protons at δ 7.30. Similar result is obtained for **6b** and is shown below. These NOE results strongly confirm the structures of **6a,b**.

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588.8 wkp
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4b

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gain not used

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SAMPLE DEC. 8 VT
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rent CDC13 dn H1
1 /export/home/~dfrq 3B
a/vnarsys/data- dot
ME/gane1864.81- dm nmh
RM-718 dm
ACQUISITION 439.845 dfrq 11111
H1 dres 1.0
2.182 homo
32768
7589.6
not used PROC
4 fn ft
57 math 32768
5.5
8.881 wfrt
588.8 wexp
32 wds
32 wnt
ck not used wft
1 FLAGS
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DISPLAY -258.3
5248.8
33
258
18.58
176.09
765.1
8
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100.000
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7.455
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2.3491.985
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SAMPLE DEC. 8 VT
 date Aug 7 2082 dfrq 499.846
 solvent CDCl3 dn
 file /export/home/~dpr
 oper/vmrsvs/data-
 1/QMNE/gene808.81-
 6H.fid dm
 C
 11111
 ACQUISITION
 sfrq 499.847 dseq
 tn H1 dres 1.8
 at 2.162 homo
 np 32768
 sw 7589.6 wfil18
 fb not used proc
 bs 4 fn
 tprf 5.7 math
 pu 5.5
 di 8.801 werr
 tot 588.8 wexp
 nt 32 wds
 ct 32 wt
 alock y
 gain not used
 FLAOS
 11 n
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 DISPLAY
 sp -258.3
 wp 5248.0
 ve 5248.78
 sc 8
 uc 258
 hznm 5.17
 18 237.58
 f1 767.4
 f7p 8
 th 14
 ins 180.000
 ph

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SAMPLE DEC. 8 VT
 date Aug 7 2002 dfrq 499.846
 solvent CDCl3 dn H1
 file /export/home/~ dpr 38
 opera/vnarsys/data-dot 8
 1/NAME/gene888-81- da nnn
 3H-fid daa
 ACQUISITION daf 11111
 sfrq 499.847 d8eq
 tn H1 dres 1.8
 at 2.162 homo
 np 32766 PROCESSING
 sv 7589.6 wfile
 fb not used proc ft
 bs 4 tn 32768 f
 tprf 57 math
 pr 5.5
 dl 8.881 wrr
 tot 588.8 wexp
 nt 32 wbs
 ct 32 wnt
 alock Y
 gain not used
 FLAS not used
 f1 n
 tn n
 dp y
 hs nn
 DISPLAY
 sp -250.3
 wd 5248.0
 vs 95
 sc 8
 wc 250
 hzmm 5.76
 ls 227.43
 rfi 767.4
 rtd 0
 th 4
 ins 188.800
 na ph

C#CCC(C#N)(C#N)C(=C/c1ccccc1)CC=C

CC(C)=CC(C#N)(C#N)C#CC#CC=Cc1ccccc1

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expt 12pul1

SAMPLE DEC. 8 VT
 date NOV 6 2002 dfrq 439.845
 solvent CDCl3 dn H1
 file /export/home/-dpwr 0
 opara/vnervs/date-0
 1/NAME/gemall08-09-08
 1H, F10 dm
 ACQUISITION
 srfq 439.845 dseq 200
 tn H1 dres 1.0
 at 2.182 homo n
 np 32768 wtf10
 sv 7589.6 wtf10
 fb not used proc
 bs 4 fn 32768 f
 tprf 5.5 math
 pl 8.001 werr
 ol 580.0 wexp
 nt 32 wbs
 ct 32 wnt
 alock y
 gain not used
 f1a05
 11 n
 1a n
 dp y
 hs mn
 DISPLAY
 sp -258.3
 up 5248.8
 vs 101
 sc 0
 vc 250
 hzcm 1.10
 18 480.32
 rf1 4363.5
 rf2 3618.9
 10 100.000
 ph

7.362
 7.364
 7.349
 7.334
 7.325
 7.320
 7.312
 7.306
 7.296
 7.287
 7.282
 7.276
 7.269
 7.261
 7.240
 7.219
 7.215
 7.202
 7.168
 7.154
 6.712
 6.681
 6.649
 6.237
 6.222
 6.206
 6.191
 6.175
 6.085
 6.066
 6.053
 6.035

2.976
 2.964
 2.962
 2.950
 2.936
 2.921
 2.918
 2.915
 2.905
 2.901
 2.897
 2.882
 2.879
 2.873
 2.866
 2.848
 2.824
 2.812
 2.796
 2.081
 2.075
 1.556

—0.047

C#CC(C#N)(C#N)C(C#N)Cc1ccccc1

10