Reusable Cu₂O/PPh₃/TBAB System for the Cross-Couplings of Aryl Halides and Heteroaryl Halides with Terminal Alkynes

Bo-Xiao Tang, Feng Wang, Jin-Heng Li,* Ye-Xiang Xie, and Man-Bo Zhang*

*Key Laboratory of Chemical Biology & Traditional Chinese Medicine Research
(Ministry of Education), College of Chemistry and Chemical Engineering, Hunan
Normal University, Changsha 410081, China*

jhli@hunnu.edu.cn

Supporting Information

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(A) Remarks

There shapes of Cu₂O nanoparticles including cubic Cu₂O nanoparticles, octahedral Cu₂O nanoparticles and spherical Cu₂O nanoparticles were prepared by known procedures.¹

(B) Typical experimental procedure

Typical experimental procedure for the Cu₂O/PPh₃/TBAB (*n*-Bu₄NBr) system for the cross-coupling reactions of aryl and heteroaryl halides with terminal alkynes. A mixture of aryl halide 1 (0.5 mmol), alkyne 2 (0.7 mmol), pyramid-like Cu₂O nanoparticles (10 mol %), PPh₃ (L2; 20 mol%), K₂CO₃ (2 equiv) and TBAB (1.5 g) was stirred at 135-140 °C for desired time (indicated in Table 2) until complete consumption of starting material as monitored by TLC. After the reaction was finished, diethyl ether was poured into the mixture, then washed with water, extracted with diethyl ether, dried by anhydrous Na₂SO₄ and evaporated under vacuum, the residue was purified by flash column chromatography (hexane or hexane/ethyl acetate) to afford the desired coupled product.

After initial experimentation, the residue (the Cu₂O nanoparticles/PPh₃/TBAB system) was then solidified (evaporated in vacuo and cooled) and subjected to a second run of the Sonogashira reaction by charging with the same substrates (aryl halide, alkyne and K₂CO₃).

Octahedral Cu₂O nanoparticles preparation. 2.4 mL of 6 M NaOH was dropped slowly to a mixture of PEG-20000 (400 mg), CuCl₂ (540 mg) and distilled H₂O. After

the mixture was stirred for 20 min, 2.5 mL of 13.54 M $\text{N}_2\text{H}_4\cdot\text{H}_2\text{O}$ was dropped slowly to give red precipitates Cu_2O . Then the Cu_2O precipitates were isolated by centrifuge, and washed with distilled water (5×5 mL), anhydrous ethanol (3×5 mL) and acetone (3×5 mL). Finally, the collected Cu_2O precipitates were dried in a vacuum oven at 60 °C to offer the Octahedral Cu_2O nanoparticles (210 mg).

Cubic Cu_2O nanoparticles preparation. A mixture of PEG-6000 (300 mg), $\text{Cu}(\text{OAc})_2\cdot\text{H}_2\text{O}$ (399 mg) and D-(+)-glucose (1800 mg) was dissolved in water (100 mL). The mixture was stirred at 50 °C to ensure them dissolved completely, followed by dropwise addition of NaOH (5 M, 2 mL) into the solution. After the solution was stirred at 50 °C for 40 min, the Cu_2O precipitate was isolated by centrifuge, then washed with distilled water, ethanol and acetone in turn, and dried in a vacuum oven at 40 °C to offer Cu_2O nanoparticles (110 mg).

Spherical Cu_2O nanoparticles preparation. $\text{Cu}(\text{OAc})_2\cdot\text{H}_2\text{O}$ (399 mg; 2 mmol) was dissolved in 25 mL of DMF, followed by the addition of poly(vinyl pyrrolidone) (PVP; 05-2-mmol; molecular weight = 30,000) and NaBH_4 (0.01-0.3 g). After stirring for several minutes, the mixture was heated at 90 °C, and in 2-6 min, the color of the mixture was an orange color. The mixture was cooled to room temperature at once and washed by alcohol several times to give Cu_2O nanoparticles (93 mg).

(A) Analytical data for 3-16, 18-22 and 24

1-(2-(4-Nitrophenyl)ethynyl)benzene (3):² ¹H NMR (400 MHz, CDCl₃) δ: 8.23 (d, *J* = 8.8 Hz, 2H), 7.67 (d, *J* = 8.8 Hz, 2H), 7.57 (d, *J* = 9.2 Hz, 2H), 7.39 (t, *J* = 7.6 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ: 141.3, 132.6, 132.2, 130.6, 129.6, 128.8, 124.0, 122.5, 95.0, 87.9; LRMS (EI, 70 eV) *m/z* (%): 223 (M⁺, 100).

1,2-Diphenylethyne (4):² ¹H NMR (300 MHz, CDCl₃) δ: 7.60–7.51 (m, 4H), 7.39–7.26 (m, 6H); ¹³C NMR (75 MHz, CDCl₃) δ: 132.0, 128.7, 128.6, 123.7, 89.7; LRMS (EI, 70 eV) *m/z* (%): 178 (M⁺, 100).

1-(Hept-1-ynyl)benzene (5):² ¹H NMR (400 MHz, CDCl₃) δ: 7.41 (d, *J* = 8.0 Hz, 2H), 7.28 (t, *J* = 5.2 Hz, 3H), 2.42 (t, *J* = 7.2 Hz, 2H), 1.66-1.57 (m, 2H), 1.49-1.35 (m, 4H), 0.94 (t, *J* = 7.2 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ: 131.6, 128.2, 127.5, 124.1, 90.5, 80.6, 31.2, 28.5, 22.3, 19.4, 14.0; LRMS (EI, 70 eV) *m/z* (%): 172 (M⁺, 13), 143 (28), 129 (30), 128 (33), 115 (100).

4-Phenylbut-3-yn-1-ol (6):³ ¹H NMR (400 MHz, CDCl₃) δ: 7.41 (d, *J* = 8.0 Hz, 2H), 7.29 (t, *J* = 6.2 Hz, 3H), 3.81 (t, *J* = 6.4 Hz, 2H), 2.68 (t, *J* = 6.6 Hz, 2H), 2.33 (brs, 1H); ¹³C NMR (100 MHz, CDCl₃) δ: 131.6, 18.3, 128.0, 123.4, 86.5, 82.4, 61.2, 23.8; LRMS (EI, 70 eV) *m/z* (%): 146 (M⁺, 11), 128 (-OH, 5), 116 (37), 115 (100).

1-(2-*o*-Tolyethynyl)benzene (7):⁴ ¹H NMR (400 MHz, CDCl₃) δ: 7.53–7.52 (m, 3H), 7.35–7.33 (m, 4H), 7.23–7.22 (m, 2H) 2.52 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 140.2, 132.6, 131.9, 131.6, 129.5, 129.2, 128.4, 128.3, 128.2, 125.6, 88.4, 81.7, 20.7; LRMS (EI, 70 eV) *m/z* (%): 192 (M⁺, 100).

1-Methoxy-4-(2-phenylethynyl)benzene (8):² ¹H NMR (400 MHz, CDCl₃) δ: 7.53–7.47 (m, 4H), 7.34–7.32 (m, 3H), 6.88 (d, *J* = 8.8 Hz, 2H), 3.83 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 159.6, 133.0, 131.4, 128.3, 127.9, 123.6, 115.4, 114.0, 89.4, 88.1, 55.3; LRMS (EI, 70 eV) *m/z* (%): 208 (M⁺, 100).

1-(Hept-1-ynyl)-4-methoxybenzene (9):² ¹H NMR (400 MHz, CDCl₃) δ: 7.32 (d, *J* = 8.4 Hz, 2H), 6.80 (d, *J* = 8.4 Hz, 2H), 3.78 (s, 3H), 2.37 (t, *J* = 7.0 Hz, 2H), 1.63–1.56 (m, 2H), 1.46–1.25 (m, 4H), 0.92 (t, *J* = 7.0 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ: 159.0, 132.6, 116.3, 113.8, 88.8, 80.2, 55.2, 31.2, 28.6, 23.5, 19.4, 14.0; LRMS (EI, 70 eV) *m/z* (%): 202 (M⁺, 23), 173 (18), 159 (23), 145 (67), 115 (63), 102 (100).

4-(4-Methoxyphenyl)but-3-yn-1-ol (10):³ ¹H NMR (400 MHz, CDCl₃) δ: 7.35 (d, *J* = 8.8 Hz, 2H), 6.83 (d, *J* = 8.8 Hz, 2H), 3.83 (s, 3H), 3.80 (t, *J* = 6.4 Hz, 2H), 2.68 (t, *J* = 6.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ: 159.3, 133.0, 115.4, 113.9, 84.7, 82.3, 61.2, 55.3, 23.9; LRMS (EI, 70 eV) *m/z* (%): 176 (M⁺, 33), 158 (-OH, 4), 116 (37), 115 (100).

3-(4-Methoxyphenyl)prop-2-yn-1-ol (11)³

¹H NMR (300 MHz, CDCl₃) δ: 7.38 (d, *J* = 8.8 Hz, 2H), 6.84 (d, *J* = 8.8 Hz, 2H), 4.49 (s, 2H), 3.82 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ: 159.7, 133.2, 114.5, 113.9, 85.8, 85.6, 55.3, 51.7; LRMS (EI, 70 eV) *m/z* (%): 162 (M⁺, 21), 144 (-OH, 3), 115 (100).

3-(2-(4-Methoxyphenyl)ethynyl)pyridine (12)⁵

¹H NMR (400 MHz, CDCl₃) δ: 8.78 (s, 1H), 8.56 (s, 1H), 7.78 (d, *J* = 8.0 Hz, 1H), 7.48 (d, *J* = 8.8 Hz, 2H), 7.32-7.27 (m, 1H), 6.89 (d, *J* = 8.8 Hz, 2H), 3.82 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 160.1, 152.2, 148.2, 138.3, 133.6, 133.2, 114.5, 114.2, 92.8, 84.7, 55.3; LRMS (EI, 70 eV) *m/z* (%): 209 (M⁺, 100).

2-(2-Phenylethynyl)benzenamine (13)³

¹H NMR (400 MHz, CDCl₃) δ: 7.53-7.51 (m, 2H), 7.37-7.32 (m, 4H), 7.13 (t, *J* = 8.8 Hz, 1H), 6.72-6.69 (m, 2H), 4.26 (brs, 2H); ¹³C NMR (100 MHz, CDCl₃) δ: 148.6, 132.2, 131.5, 129.8, 128.6, 128.3, 123.4, 118.0, 114.0, 108.0, 94.6, 85.0; LRMS (EI, 70 eV) *m/z* (%): 193 (M⁺, 100).

1-(4-(2-Phenylethynyl)phenyl)ethanone (14)⁴

¹H NMR (400 MHz, CDCl₃) δ: 7.95 (d, *J* = 8.8 Hz, 2H), 7.61 (d, *J* = 8.8 Hz, 2H), 7.57-7.55 (m, 2H), 7.38 (t, *J* = 3.2 Hz, 3H), 2.63 (s, 3H); ¹³C NMR (100 MHz, CDCl₃)

δ : 197.4, 136.1, 131.8, 131.7, 128.8, 128.4, 128.3, 122.6, 92.7, 88.6, 26.7; LRMS (EI, 70 eV) m/z (%): 220 (M^+ , 77), 205 (100).

3-(2-Phenylethynyl)pyridine (15)⁵

^1H NMR (400 MHz, CDCl_3) δ : 8.63 (s, 1H), 7.68 (t, $J = 7.6$ Hz, 1H), 7.62–7.60 (m, 2H), 7.54 (d, $J = 7.6$ Hz, 1H), 7.38–7.36 (m, 3H), 7.25 (t, $J = 8.0$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ : 150.0, 143.5, 136.2, 132.1, 129.0, 128.4, 127.3, 122.9, 122.3, 89.0, 88.7; LRMS (EI, 70 eV) m/z (%): 179 (M^+ , 100).

3-(Hept-1-ynyl)pyridine (16)⁶

^1H NMR (400 MHz, CDCl_3) δ : 8.63 (s, 1H), 8.48 (d, $J = 8.0$ Hz, 1H), 7.67 (d, $J = 8.0$ Hz, 1H), 7.21 (t, $J = 7.6$ Hz, 1H), 2.42 (t, $J = 7.2$ Hz, 2H), 1.66–1.59 (m, 2H), 1.47–1.25 (m, 4H), 0.93 (t, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 152.4, 147.9, 138.4, 122.9, 121.2, 94.2, 31.1, 28.3, 22.2, 19.4, 14.0; LRMS (EI, 70 eV) m/z (%): 173 (M^+ , 12), 158 (6), 144 (44), 130 (35), 118 (55), 103 (45), 89 (87), 63 (100).

3-(2-(Pyridin-3-yl)ethynyl)pyridine (18)^{5,8}

^1H NMR (400 MHz, CDCl_3) δ : 8.80 (s, 2H), 8.60 (s, 2H), 7.84 (d, $J = 6.8$ Hz, 2H), 7.34–7.31 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 152.3, 149.1, 138.5, 123.1, 119.8, 89.2; LRMS (EI, 70 eV) m/z (%): 180 (M^+ , 100).

2-(2-Phenylethynyl)pyrazine (19)⁷

^1H NMR (400 MHz, CDCl_3) δ : 8.77 (s, 1H), 8.59 (s, 1H), 8.49 (s, 1H), 7.63 (d, J = 7.6 Hz, 2H), 7.39 (t, J = 8.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 147.8, 144.5, 142.8, 140.4, 132.2, 132.1, 129.6, 128.5, 121.5, 93.3, 85.8; LRMS (EI, 70 eV) m/z (%): 180 (M^+ , 69), 127 (100).

5-(2-Phenylethynyl)pyrimidine (20)⁸

^1H NMR (400 MHz, CDCl_3) δ : 9.15 (s, 1H), 8.87 (s, 1H), 7.56 (d, J = 7.6 Hz, 2H), 7.41–7.39 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 158.6, 156.6, 131.7, 129.3, 128.5, 121.7, 119.9, 96.3, 82.2; LRMS (EI, 70 eV) m/z (%): 180 (M^+ , 100).

3-(2-Phenylethynyl)quinoline (21)⁸

^1H NMR (400 MHz, CDCl_3) δ : 9.02 (s, 1H), 8.34 (s, 1H), 8.12 (d, J = 8.0 Hz, 2H), 7.82–7.73 (m, 2H), 7.60–7.57 (m, 2H), 7.41–7.39 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 152.2, 146.9, 138.3, 132.1, 131.8, 130.1, 129.5, 128.9, 128.5, 127.6, 127.3, 122.6, 117.5, 92.6, 86.7; LRMS (EI, 70 eV) m/z (%): 229 (M^+ , 84), 200 (14), 126 (13), 114 (55), 101 (66), 88 (100).

2-(2-Phenylethynyl)thiazole (22)⁹

^1H NMR (400 MHz, CDCl_3) δ : 7.87 (s, 1H), 7.60 (d, J = 9.6 Hz, 2H), 7.42–7.37 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ : 154.6, 143.6, 131.9, 129.5, 128.5, 121.4, 120.8, 93.9, 82.2; LRMS (EI, 70 eV) m/z (%): 185 (M^+ , 100)

2-(2-Phenylethynyl)pyrimidine (**24**)⁸

¹H NMR (400 MHz, CDCl₃) δ : 8.77 (s, 1H), 7.68 (d, J = 7.6 Hz, 2H), 7.40 (t, J = 8.0 Hz, 3H), 7.26 (t, J = 5.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ : 157.3, 153.4, 132.6, 129.7, 128.7, 121.3, 119.7, 88.1, 87.9; LRMS (EI, 70 eV) m/z (%): 180 (M⁺, 100)

(B) References

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(C) Spectra

Fig. 1 TEM picture showing octahedral Cu_2O nanoparticles.

Fig. 2 Histogram of the Cu_2O nanoparticles' size distribution and XRD pattern of the octahedral Cu_2O nanoparticles.

Fig. 3 TEM picture showing spherical Cu_2O nanoparticles.

Fig. 4 XRD pattern of the spherical Cu_2O nanoparticles.

1-(2-(4-nitrophenyl)ethynyl)benzene (3)

Current Data Parameters
NAME n12007-01-04
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070104
Time 13.34
INSTRUM av400
PROBHD 5 mm PABBO B9-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 812.7
DM 62.400 usec
DE 6.00 usec
TE 290.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCORR 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 4.00 usec
PL1 -3.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 16384
SF 400.1300079 MHz
MOM EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10 NMR plot parameters
CX 22.00 cm
CY 6.00 cm
FIR 9.919 ppm
F1 3969.05 Hz
F2 -1.139 ppm
F3 -455.94 Hz
PPMCM 0.50268 ppm/cm
HZCM 201.13594 Hz/cm

1-(2-(4-nitrophenyl)ethynyl)benzene (3)

1,2-diphenylethyn (4)

1,2-diphenylethyn (4)

1-(Hept-1-ynyl)benzene (5)

1-(Hept-1-ynyl)benzene (5)

4-Phenylbut-3-yn-1-ol (6)

OCC#Cc1ccccc1

Current Data Parameters	
NAME	C13-2007-01-12
EXPNO	11
PROCNO	1

F2 - Acquisition Parameters	
Date_	20070114
Time	17.16
INSTRUM	av400
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1280
DS	0
SWH	25125.629 Hz
FIDRES	0.38397 Hz
AQ	1.3042614 sec
RG	16384
DM	19.900 usec
DE	6.00 usec
TE	293.6 K
D1	2.00000000 sec
d11	0.03000000 sec
MCNST	0.00000000 sec
MCNKR	0.01500000 sec

CHANNEL f1	
MUCL1	13C
P1	6.40 usec
PL1	-1.00 dB
SFO1	100.6258903 MHz

CHANNEL f2	
CPDPRG2	waltz16
MUCL2	1H
PCPD2	80.00 usec
PL2	-3.00 dB
PL12	16.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters	
SF	32768
SF2	100.6127687 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters	
CK	22.00 cm
CV	12.00 cm
F1P	208.218 ppm
F2P	20949.37 Hz
F2	-9.951 ppm
F2	-1001.18 Hz
SPINDR	9.91675000/cm
QZCH	997.75214 Hz/cm

Cc1ccccc1C#Cc2ccccc2

1-(2-*o*-tolylethynyl)benzene (7)

1-Methoxy-4-(2-phenylethynyl)benzene (8)

1-methoxy-4-(2-phenylethynyl)benzene (8)

1-(Hept-1-ynyl)-4-methoxybenzene (9)

1-(Hept-1-ynyl)-4-methoxybenzene (9)

4-(4-Methoxyphenyl)but-3-yn-1-ol (10)

4-(4-Methoxyphenyl)but-3-yn-1-ol (10)

3-(4-Methoxyphenyl)prop-2-yn-1-ol (11)

3-(4-Methoxyphenyl)prop-2-yn-1-ol (11)

3-(2-(4-Methoxyphenyl)ethynyl)pyridine (12)

3-(2-(4-Methoxyphenyl)ethynyl)pyridine (12)

2-(2-Phenylethynyl)benzenamine (13)

2-(2-Phenylethynyl)benzenamine (13)

Chemical structure: O=C1C=CC(=C1)/C=C/C(=O)C2=CC=CC=C2

¹H NMR spectrum (ppm):

- 7.963, 7.941, 7.630, 7.608, 7.571, 7.564, 7.558, 7.547, 7.555, 7.379, 7.372, 7.264
- Integration: 2.04, 2.01, 1.98, 2.01, 2.98, 3.00, 2.04

1-(4-(2-Phenylethynyl)phenyl)ethanone (14)

3-(2-Phenylethynyl)pyridine (15)

3-(2-Phenylethynyl)pyridine (15)

3-(Hept-1-ynyl)pyridine (16)

3-(Hept-1-ynyl)pyridine (16)

3-(2-(Pyridin-3-yl)ethynyl)pyridine (18)

3-(2-(Pyridin-3-yl)ethynyl)pyridine (18)

2-(2-Phenylethynyl)pyrazine (19)

2-(2-Phenylethynyl)pyrazine (19)

c1ccc(cc1)/C=C/c2ccncc2

5-(2-Phenylethynyl)pyrimidine (20)

3-(2-Phenylethynyl)quinoline (21)

3-(2-Phenylethynyl)quinoline (21)

c1ccccc1C#N2C=CCSC2

2-(2-Phenylethynyl)thiazole (22)

2-(2-Phenylethynyl)pyrimidine (24)

2-(2-Phenylethynyl)pyrimidine (24)