Supporting Information
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Supporting Information

for

Recyclable Polystyrene-Supported Copper Catalysts for the Aerobic Oxidative Homocoupling of Terminal Alkynes

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Experimental Section

General. $^1$H NMR spectra were recorded on a JEOL JNM-AL400 (400 MHz) spectrometer. The chemical shifts were reported in parts per million ($\delta$) relative to internal standard TMS (0 ppm) for CDCl$_3$. The peak patterns are indicated as follows: s, singlet; d, doublet; dd, doublet of doublet; t, triplet; m, multiplet; q, quartet. The coupling constants, $J$, are reported in Hertz (Hz). $^{13}$C NMR spectra were obtained by a JEOL JNM-AL400 (100 MHz) spectrometers and referenced to the internal solvent signals (central peak is 77.0 ppm in CDCl$_3$). CDCl$_3$ was used as a NMR solvent. GC analysis was carried out on a Hewlett Packard 4890 system. High-resolution mass spectra (HRMS) were measured on a JEOL AccuTOF JMS-T100LC with ESI method, and IR spectra were recorded on a JASCO FT/IR 460 Plus spectrometer in ATR mode. ICP analysis was performed on a LEEMAN LABS Profile plus plasma spectrometer. All reagents were purchased from Wako, Kanto, Aldrich and TCI and used without further purification. Chloromethylated polystyrene (2.4 mmol/g Cl loading, crosslinked with 1.0% DVB (divinylbenzene), particle size 100-200 mesh) was purchased from TCI. Water was deionized with a Millipore system as a Milli-Q grade.

Preparation of polystyrene-supported $N,N,N',N'$-tetraethyldiethylenetriamine (PS-TEDETA):
A mixture of chloromethylated polystyrene (1.0 g, 2.4 mmol/Cl), $N,N,N',N'$-tetraethyldiethylenetriamine (2.47 mL, 9.6 mmol), NEt$_3$ (1.33 mL, 9.6 mmol), and NaI (14.9 mg, 0.1 mmol) in CH$_3$CN (100 mL) was refluxed for 72 h. After cooling, the mixture was filtered and the resulting resin beads were washed sequentially with CH$_3$CN (10 mL x3), 1:1 CH$_3$OH-H$_2$O (10 mL x3), CH$_3$OH (10 mL x3), dichloromethane (10 mL x3), Et$_2$O (10 mL x3), and then dried in vacuo.
Fig. S1. SR-MAS NMR $^{13}$C spectra of (a) chloromethylated polystyrene, (b) polystyrene-supported TEDETA, and (c) TEDETA.
Preparation of polystyrene-supported CuSO₄-TEDETA complex (A): A mixture of PS-TEDETA (200 mg) and CuSO₄·5H₂O (0.504 mmol) in EtOH (3 mL) was stirred at 50 °C for 6 h. After cooling, the mixture was filtered and the resulting resin beads were washed with EtOH (4 mL x 10), dried in vacuo overnight to provide the corresponding polystyrene-supported Cu(II)-TEDETA complex (A). The loading value of copper was determined by ICP analysis: 1.16 mmol/g.

Other polystyrene-supported Cu(II)-TEDETA complexes (B and C) were prepared following the similar procedure.

**Fig. S2.** Optical microscopic images of (a) chloromethylated polystyrene, (b) polystyrene-supported TEDETA, (c) polystyrene-supported TEDETA-CuSO₄, and (d) polystyrene-supported TEDETA-CuSO₄ after the reaction.
Fig. S3. FT-IR spectra of (a) chloromethylated polystyrene (PS-Cl), (b) polystyrene-supported TEDETA (PS-TEDETA), and (c) polystyrene-supported TEDETA-CuSO₄ (A, PS-TEDETA-CuSO₄).
Fig. S4. FT-IR spectra of polystyrene-supported TEDETA-CuSO₄ (black color: fresh; red color: recovered).

Table S1. Optimization of reaction conditions

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General procedure for the aerobic oxidative homocoupling of terminal alkynes: A mixture of catalyst (PS-TEDETA-CuSO₄, 0.05 mmol of Cu), terminal alkyne (0.5 mmol) and piperidine (0.5 mmol) in toluene (1.0 mL) was stirred at 60 °C for 24-48 h under air. After cooling, the mixture was filtered, and the residue was washed by dichloromethane (2 mL x3). The combined organic phases were concentrated in vacuo and the crude products were purified by column chromatography (hexane/AcOEt) to give the corresponding 1,3-diynes.

\[
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\text{C} \\
\text{C} \\
\text{C}
\end{array} \begin{array}{c}
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\text{C} \\
\text{C} \\
\text{C}
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\text{Me}
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1,4-Diphenylbuta-1,3-dyne (2a) [CAS: 886-66-8]. Isolated by column chromatography (hexane/AcOEt = 99: 1, Rₕ = 0.7). The title compound was obtained as a white solid (96%). \(^1\)H NMR (ppm) δ 7.54-7.52 (m, 4H), 7.39-7.31 (m, 6H); \(^{13}\)C NMR (ppm) δ 132.5, 129.2, 128.4, 121.7, 81.5, 73.9.

\[
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\text{C} \\
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\text{C} \\
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\text{Me}
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1,4-Bis(p-methoxyphenyl)buta-1,3-dyne (2b) [CAS: 22779-05-1]. Isolated by column chromatography (hexane/AcOEt = 99: 1, Rₕ = 0.6). The title compound was obtained as a white solid (96%). \(^1\)H NMR (ppm) δ 7.46 (d, J = 9.0 Hz, 4H), 6.85 (d, J = 9.0 Hz, 4H), 3.82 (s, 6H); \(^{13}\)C NMR (ppm) δ 160.2, 134.0, 114.1, 113.9, 81.2, 72.9, 55.3.

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\text{C} \\
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\text{Me}
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1,4-Di([1,1'-biphenyl]-4-yl)buta-1,3-dyne (2c) [CAS: 29079-15-0]. Isolated by column chromatography (hexane/AcOEt = 9: 1, Rₕ = 0.6). The title compound was obtained as a white solid (98%). \(^1\)H NMR (ppm) δ 7.63-7.54 (m, 12H), 7.48-7.44 (m, 4H), 7.40-7.36 (m, 2H); \(^{13}\)C NMR (ppm) δ 141.9, 140.1, 132.9, 128.9, 127.9, 127.1, 127.0, 120.6, 81.8, 74.6.
1,4-Bis(p-pentylphenyl)buta-1,3-diyne (2d) [CAS: 121477-67-6]. Isolated by column chromatography (hexane/AcOEt = 9: 1, Rf = 0.6). The title compound was obtained as a white solid (96%). $^1$H NMR (ppm) δ 7.43 (d, J = 8.2 Hz, 4H), 7.14 (d, J = 8.2 Hz, 4H), 2.60 (t, J = 8.2 Hz, 4H), 1.62-1.55 (m, 4H), 1.32-1.28 (m, 8H), 0.89 (t, J = 6.6 Hz, 6H); $^{13}$C NMR (ppm) δ 144.5, 132.4, 128.5, 118.9, 81.6, 73.5, 35.9, 31.4, 30.8, 22.5, 14.0.

1,4-Di(p-tolyl)buta-1,3-diyne (2e) [CAS: 22666-07-5]. Isolated by column chromatography (hexane/AcOEt = 99: 1, Rf = 0.7). The title compound was obtained as a white solid (95%). $^1$H NMR (ppm) δ 7.41 (d, J = 8.6 Hz, 4H), 7.14 (d, J = 8.6 Hz, 4H), 2.36 (s, 6H); $^{13}$C NMR (ppm) δ 139.5, 132.4, 129.2, 118.7, 81.5, 73.4, 21.6.

1,4-Bis(4-(trifluoromethyl)phenyl)buta-1,3-diyne (2f) [CAS: 151362-06-0]. Isolated by column chromatography (hexane/AcOEt = 99: 1, Rf = 0.6). The title compound was obtained as a white solid (99%). $^1$H NMR (ppm) δ 7.63 (bs, 8H); $^{13}$C NMR (ppm) δ 132.8, 131.1 (d, J = 32.6 Hz, 1C), 126.9 (q, J = 3.6 Hz, 1C), 125.2 (d, J = 2.0 Hz, 1C), 123.7 (d, J = 274.0 Hz, 1C), 80.9, 75.6.

1,4-Bis(p-fluorophenyl)buta-1,3-diyne (2g) [CAS: 55606-94-5]. Isolated by column chromatography (hexane/AcOEt = 99: 1, Rf = 0.7). The title compound was obtained as a white solid (95%). $^1$H NMR (ppm) δ 7.51 (dd, J = 8.8, 5.6 Hz, 4H), 7.05 (dd, J = 8.8, 5.6 Hz, 4H); $^{13}$C NMR (ppm) δ 163.0 (d, J = 252.9 Hz, 1C), 134.5 (d, J = 8.6 Hz, 1C), 117.8 (d, J = 3.9 Hz, 1C), 115.9 (d, J
= 22.1 Hz, 1C), 80.4, 73.5.

\[
\text{MeO}_2\text{C} \equiv \equiv \equiv \text{CO}_2\text{Me}
\]

1,4-Bis(p-carbomethoxyphenyl)buta-1,3-diyne (2h) [CAS: 120617-78-9]. Isolated by column chromatography (hexane/AcOEt = 20: 1, Rf = 0.7). The title compound was obtained as a white solid (95%). \(^1\)H NMR (ppm) δ 8.02 (d, \(J = 8.8\) Hz, 4H), 7.60 (d, \(J = 8.8\) Hz, 4H), 3.93 (s, 6H); \(^{13}\)C NMR (ppm) δ 166.3, 132.5, 130.5, 129.6, 126.1, 81.8, 76.2, 52.4.

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1,4-Di(m-tolyl)buta-1,3-diyne (2i) [CAS: 53903-61-0]. Isolated by column chromatography (hexane/AcOEt = 99: 1, Rf = 0.7). The title compound was obtained as a white solid (97%). \(^1\)H NMR (ppm) δ 7.33-7.32 (m, 4H), 7.23-7.16 (m, 4H), 2.33 (s, 6H); \(^{13}\)C NMR (ppm) δ 138.1, 132.9, 130.1, 129.6, 128.3, 121.6, 81.6, 73.6, 21.2.

\[
\begin{array}{c}
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\equiv \\
\text{Me}
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1,4-Di(o-tolyl)buta-1,3-diyne (2j) [CAS: 136053-56-0]. Isolated by column chromatography (hexane/AcOEt = 99: 1, Rf = 0.7). The title compound was obtained as a white solid (96%). \(^1\)H NMR (ppm) δ 7.50 (d, \(J = 7.2\) Hz, 2H), 7.27-7.20 (m, 4H), 7.17-7.13 (m, 2H), 2.49 (s, 6H); \(^{13}\)C NMR (ppm) δ 141.6, 132.9, 129.5, 129.1, 125.6, 121.7, 81.1, 77.5, 20.7.

\[
\begin{array}{c}
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\text{F}_3\text{C}
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1,4-Bis(2-(trifluoromethyl)phenyl)buta-1,3-diyne (2k) [CAS: 90735-59-4]. Isolated by column
chromatography (hexane/AcOEt = 20: 1, R$_f$ = 0.6). The title compound was obtained as a white solid (83%). $^1$H NMR (ppm) $\delta$ 7.69 (dd, $J$ = 8.0, 7.6 Hz, 4H), 7.50 (dd, $J$ = 7.2, 6.4 Hz, 2H), 7.47 (dd, $J$ = 7.6, 7.2 Hz, 2H); $^{13}$C NMR (ppm) $\delta$ 135.1, 132.5 (q, $J$ = 0.6 Hz, 1C), 132.1, 129.1, 126.0 (q, $J$ = 4.8 Hz, 1C), 123.2 (d, $J$ = 275 Hz, 1C), 119.7 (q, $J$ = 1.9 Hz, 1C), 78.7, 78.6.

1,4-Bis(2-fluorophenyl)buta-1,3-diyne (2l) [CAS: 332919-95-6]. Isolated by column chromatography (hexane/AcOEt = 99: 1, R$_f$ = 0.6). The title compound was obtained as a white solid (90%). $^1$H NMR (ppm) $\delta$ 7.54-7.50 (m, 2H), 7.39-7.34 (m, 2H), 7.15-7.08 (m, 4H); $^{13}$C NMR (ppm) $\delta$ 163.7 (d, $J$ = 254.9 Hz, 1C), 134.3, 131.1 (d, $J$ = 7.7 Hz, 1C), 124.1 (d, $J$ = 3.8 Hz, 1C), 115.7 (d, $J$ = 20.2 Hz, 1C), 110.4 (d, $J$ = 15.3 Hz, 1C), 78.3, 75.8.

1,4-Di(naphthalen-1-yl)buta-1,3-diyne (2m) [CAS: 20199-30-8]. Isolated by column chromatography (hexane/AcOEt = 99: 1, R$_f$ = 0.6). The title compound was obtained as a white solid (98%). $^1$H NMR (ppm) $\delta$ 8.43 (d, $J$ = 8.0 Hz, 2H), 7.88 (dd, $J$ = 7.8, 2.6 Hz, 4H), 7.83 (dd, $J$ = 7.0, 1.0 Hz, 2H), 7.65-7.61 (m, 2H), 7.57-7.53 (m, 2H), 7.48-7.44 (m, 2H); $^{13}$C NMR (ppm) $\delta$ 133.9, 133.1, 132.1, 129.8, 128.5, 127.2, 126.7, 126.1, 125.2, 119.5, 80.9, 78.7.

1,4-Di(cyclohexenyl)buta-1,3-diyne (2n) [CAS: 2979-05-7]. Isolated by column chromatography (hexane/AcOEt = 9: 1, R$_f$ = 0.6). The title compound was obtained as colorless oil (86%). $^1$H NMR
(ppm) δ 6.26-6.24 (m, 2H), 2.14-2.09 (m, 8H), 1.66-1.54 (m, 8H); 13C NMR (ppm) δ 138.1, 119.9, 82.7, 71.5, 28.7, 25.9, 22.1, 21.3.

1,6-Diphenoxyhexa-2,4-diyne (2o) [CAS: 30980-37-1]. Isolated by column chromatography (hexane/AcOEt = 50: 1, Rf = 0.6). The title compound was obtained as a white solid (78%). 1H NMR (ppm) δ 7.30 (dd, J = 8.4, 7.6 Hz, 4H), 7.00 (t, J = 7.6 Hz, 2H), 6.94 (d, J = 8.4 Hz, 4H), 4.74 (s, 4H); 13C NMR (ppm) δ 157.3, 129.5, 121.7, 114.8, 74.6, 71.0, 56.1.

1,8-Bis(benzoxy)octa-3,5-diyne (2p) [CAS: none]. Isolated by column chromatography (hexane/AcOEt = 99: 1, Rf = 0.6). The title compound was obtained as a white solid (80%). Mp: 88-89 °C; 1H NMR (ppm) δ 8.06-8.04 (m, 4H), 7.59-7.54 (m, 2H), 7.46-7.42 (m, 4H), 4.41 (t, J = 6.8 Hz, 4H), 2.74 (t, J = 6.8 Hz, 4H); 13C NMR (ppm) δ 166.2, 133.1, 129.7, 129.6, 128.4, 73.5, 66.6, 62.2, 19.8; IR (ATR, neat): 3063, 2920, 1715, 1450, 1386, 1265, 1116, 1021, 985, 704, 684, 677 cm⁻¹; HRMS(ESI) calcd for C22H19O4 (M⁺+H): 347.1283; found: 347.1284.
**Dibenzyl dodeca-5,7-diynedioate (2q)** [CAS: *none*]. Isolated by column chromatography (hexane/AcOEt = 9: 1, Rf = 0.5). The title compound was obtained as colorless oil (67%). $^1$H NMR (ppm) $\delta$ 7.39-7.29 (m, 10H), 5.11 (s, 4H), 2.48 (t, $J = 7.2$ Hz, 4H), 2.32 (t, $J = 6.8$ Hz, 4H), 1.89-1.82 (m, 4H); $^{13}$C NMR (ppm) $\delta$ 172.7, 135.8, 128.5, 128.2, 128.1, 76.3, 66.2, 66.0, 32.9, 23.4, 18.6; IR (ATR, neat): 3033, 2942, 1731, 1455, 1212, 1148, 968, 736, 696 cm$^{-1}$; HRMS(ESI) calcd for C$_{26}$H$_{27}$O$_4$ (M$^{+}$+H): 403.1909; found: 403.1906.

**Deca-4,6-diyn-1,10-diol (2r)** [CAS: 70283-74-8]. Isolated by column chromatography (hexane/AcOEt = 9: 1, Rf = 0.6). The title compound was obtained as colorless oil (58%). $^1$H NMR (ppm) $\delta$ 3.74 (t, $J = 6.2$ Hz, 4H), 2.39 (t, $J = 7.0$ Hz, 4H), 1.89 (bs, 2H), 1.81-1.75 (m, 4H); $^{13}$C NMR (ppm) $\delta$ 76.8, 65.6, 61.3, 30.9, 15.7.

**Hexadeca-7,9-diyne (2s)** [CAS: 18277-20-8]. Isolated by column chromatography (hexane/AcOEt = 9: 1, Rf = 0.6). The title compound was obtained as colorless oil (70%). $^1$H NMR (ppm) $\delta$ 2.24 (t, $J = 7.2$ Hz, 4H), 1.55-1.48 (m, 4H), 1.42-1.22 (m, 12H), 0.89 (t, $J = 7.2$ Hz, 6H); $^{13}$C NMR (ppm) $\delta$ 77.5, 65.2, 31.3, 28.5, 28.3, 22.5, 19.2, 14.0.
Icosa-9,11-diyne (2t) [CAS: 28393-07-9]. Isolated by column chromatography (hexane/AcOEt = 99:1, R<sub>f</sub> = 0.6). The title compound was obtained as colorless oil (65%). <sup>1</sup>H NMR (ppm) δ 2.24 (t, J = 7.0 Hz, 4H), 1.56-1.48 (m, 4H), 1.40-1.27 (m, 20H), 0.88 (t, J = 6.8 Hz, 6H); <sup>13</sup>C NMR (ppm) δ 77.5, 65.2, 31.8, 29.1, 29.0, 28.8, 28.3, 22.6, 19.2, 14.1.
Copies of $^1$H NMR and $^{13}$C NMR spectra for all compounds
## single pulse

**Formula:** MeO₂C – CHO₂C Me

**2h**

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## single pulse decoupled gated NOE

**Formula:**

**DIFL:** Y130_carbon-1-1 abs
**COMNT:** single pulse decoupled gated NOE
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**OBNUC:** 13C
**EXMOD:** carbon, ppp
**OBRES:** 99.55 MHz
**OBSET:** 5.13 KHz
**OBFIN:** 0.98 Hz
**POWLT:** 3270 Hz
**FREQU:** 31250 Hz
**SCANS:** 512
**ACQTM:** 10886 sec
**FTN:** 20009 sec
**PT1:** 3.42 sec
**REDUC:** H2
**CTEMP:** 25.2 x
**SLYNT:** CDCl₃
**EXREF:** 77.00 ppm
**RF:** 0.00 Hz
**ROAINE:** 60

---

**Diagram:**

1. **Single Pulse Spectroscopy:**

   - **Sample:** MeO₂C – CHO₂C Me
   - **Conditions:** Single pulse, ppp, proton
   - **Parameters:**
     - DFLF: Y130_proton-1-1 abs
     - COMNT: single pulse
     - DATIM: 2015-08-18 15:51:11
     - OBNUC: H2
     - EXMOD: proton, ppp
     - OBRES: 39.88 MHz
     - OBSET: 6.28 Hz
     - OBFIN: 0.47 Hz
     - POINT: 1040 Hz
     - FREQU: 7422 Hz
     - SCANS: 8
     - ACQTM: 2.2073 sec
     - FTN: 5.0009 sec
     - PT1: 3.12 sec
     - REDUC: H2
     - CTEMP: 20.3 x
     - SLYNT: CDCl₃
     - EXREF: 0.00 ppm
     - RF: 0.00 Hz
     - ROAINE: 48

2. **Single Pulse Decoupled Gated NOE Spectroscopy:**

   - **Sample:** MeO₂C – CHO₂C Me
   - **Conditions:** Single pulse decoupled gated NOE
   - **Parameters:**
     - DFLF: Y130_carbon-1-1 abs
     - COMNT: single pulse decoupled gated NOE
     - DATIM: 2015-08-18 16:01:48
     - OBNUC: 13C
     - EXMOD: carbon, ppp
     - OBRES: 99.55 MHz
     - OBSET: 5.13 KHz
     - OBFIN: 0.98 Hz
     - POINT: 3270 Hz
     - FREQU: 31250 Hz
     - SCANS: 512
     - ACQTM: 10886 sec
     - FTN: 20009 sec
     - PT1: 3.42 sec
     - REDUC: H2
     - CTEMP: 25.2 x
     - SLYNT: CDCl₃
     - EXREF: 77.00 ppm
     - RF: 0.00 Hz
     - ROAINE: 60

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**Graphs:**

1. **Graph 1:**
   - **Y-axis:** ppm
   - **X-axis:** ppm
   - **Peaks:** 4.15, 4.08, and 6.90

2. **Graph 2:**
   - **Y-axis:** ppm
   - **X-axis:** ppm
   - **Peaks:** 106.399 to 126.990 ppm
single pulse
decoupled gated NOE