Synthesis B. Mohan et al. Paper

Copper Nanoparticle Catalyzed Formation of C–S Bonds through Activation of S–S and C–H Bonds: An Easy Route to Alkynyl Sulfides

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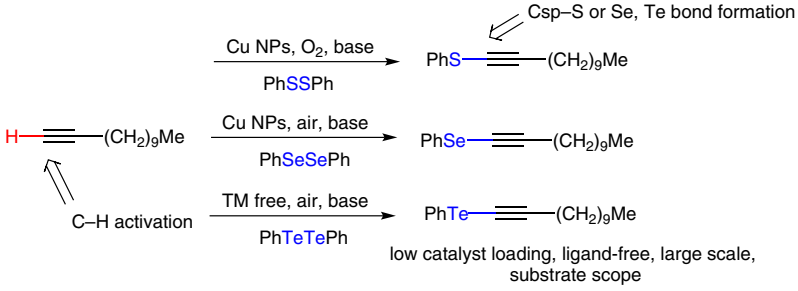

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Abstract An efficient method for copper nanoparticle catalyzed ligand-free S–C_{sp} bond formation was developed by using dimethyl sulfoxide as the solvent, a base, and molecular oxygen as a green oxidant. This study provides a new catalytic route for the production of alkynyl sulfides through dual activation of S–S and C–H bonds. A wide range of alkynyl sulfides were synthesized on a large scale with a low catalyst loading (0.5 mol% Cu). This synthetic methodology could also be extended to selenides, whereas tellurides underwent analogous reactions in the absence of a catalyst.

Key words alkynes, copper, cross-coupling, chalcogenides, nanocatalysis

Alkynyl sulfides are readily available precursors for the synthesis of a number of systems, including: vinylic sulfides by the hydroboration of thioacetylene,[1,2] substituted alkynes,[3,4] 2-chalcogene-1-halonaphthalenes by [4+2] cycloaddition annulation,[5] and zirconated vinyl chalcogenides.[6] Sulfur-substituted 1,2,3-triazoles[7] are herbicides with antifungal activity, and ThioClickFerrophos has been used as a ligand[8] in the preparation of *endo*-4-acyl pyrrolidines with high enantioselectivity (Figure 1).

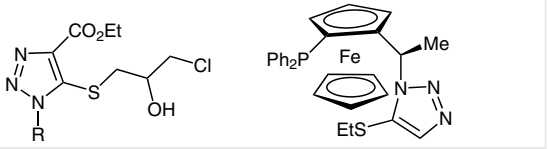


Figure 1 Antifungal drug (left) and ThioClickFerrophos (right)

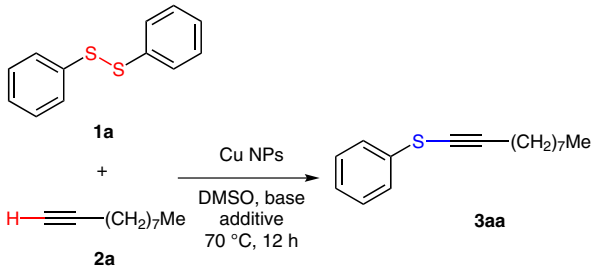
Recently, thioalkynes were effectively utilized as starting material for the synthesis of thio-substituted 1,2,3-triazoles.[9] With increasing demand for thioalkynes because of their vast range of applications, simple synthetic approaches that are compatible with various demanding substituents, operate under mild reaction conditions, and that are environmentally benign are continually being sought. Unfortunately, most of the existing synthetic protocols that are widely applicable require the use of selenides and tellurides.[10–20] Therefore, new synthetic strategies for the production of alkynyl sulfides from simple starting materials with a sustainable and atom-economical approach are being developed. Moreover, the present stringent environmental standards have forced researchers to develop alternative green protocols that involve the use of molecular oxygen as a primary oxidant in the presence of a catalyst.

Recently, Robert and co-workers reported the copper salt catalyzed cross-dehydrogenative coupling of terminal alkynes with different functionalities in good yields.[21] However, they used a high loading of Cu and an environmentally unfriendly reagent, thiophenol. Schneider et al. developed an approach that involved the use of 10 mol% indium for the synthesis of chalcogenoacetylenes in anhydrous DMSO.[17] The Bieber research group used CuI as catalyst to couple diaryldichalcogens and terminal alkynes to afford organochalcogeno acetylenes.[20] Most of the aforementioned techniques suffer from a high loading of catalysts and limited substrate compatibility (aryl acetylenes), and they are not suitable for large-scale applications.

Nanoparticles (NPs) have been used to catalyze a wide range of reactions (nanocatalysis) in the past decade under both homogeneous and heterogeneous conditions.[22–25] Because NPs have a large surface-to-volume ratio compared with that of bulk materials, they are attractive candidates as catalysts. Thus, transition-metal NPs have been widely used as catalysts in various organic transformations,[26–28] as evident from the growing number of publications on this topic, indicating the importance of nanocatalysis. The preparation of efficient NPs without surfactants under mild reaction conditions is thus clearly important. In this context,

transition-metal NP-catalyzed cross-coupling reactions may also provide a good platform to synthesize more complex molecules.

In a continuation of our efforts to develop copper catalysts[29,30] and chalcogen chemistry,[31,32] we report here the efficient use of a copper nanoparticle catalyst in the cross-coupling reaction of an environmentally friendly reagent diphenyl disulfide with terminal alkynes. To investigate the copper nanoparticle-catalyzed alkynylation reaction, diphenyl disulfide (**1a**) and 1-decyne (**2a**) were selected as representative substrates for initial screening (Scheme 1). Dimethyl sulfoxide (DMSO) was used as solvent, and other parameters such as temperature, time, base, and additive were varied systematically (Table 1).



Scheme 1 Cross-coupling of diphenyl disulfide and 1-decyne

The reaction of **1a** with Cu NPs (0.5 mol%), base (2.2 equiv), and alkyne **2a** (2.2 equiv) in DMSO (4 mL) at 70 °C for 12 h afforded the corresponding product in 37% yield (Table 1, entry 1). Increasing the amount of catalyst from 0.5 to 2 mol% increased the yield only slightly (entry 2). Increasing the reaction time also produced similar results (entry 3). The reaction did not proceed at all in the absence of the catalyst (entry 4). The use of a diverse range of bases did not improve the yield (entries 5–9). Further investigations revealed that the use of zinc dust as an additive did not afford any cross-coupled product, whereas the addition of manganese powder minimized the yield (entries 10 and 11). The use of ligands such as bipyridyl, phenanthroline, and TMEDA failed to afford satisfactory results (entries 12–14). Running the reaction under oxygen (1 atm) afforded the cross-coupled product in 38% yield, whereas reaction under argon (1 atm) led to complete recovery of the starting materials (entries 15 and 16), which confirms the required use of oxidant to promote the cross-coupling. Surprisingly, the reaction occurred efficiently and afforded the desired cross-coupled product **3aa** in 77% yield under 2 atm oxygen. Thus, O_2 was essential as the oxidant for good conversion. An increase in the catalyst quantity provided almost the same results (entry 18). No significant improvement in yield was observed when 3 atm oxygen was used under identical conditions. Moreover, increasing the scale of the reaction (from 100 mg **1a** to 500 mg) did not reduce the yield (entry 20). Performing the reaction at room temperature for 24 hours resulted in a diminished yield (entry 21). Therefore, the optimized reaction conditions for the cross-coupling of diphenyl disulfide and terminal alkynes were established as: diphenyl disulfide (1 mmol), 0.5 mol% Cu NPs, Na_2CO_3 (2.2 equiv), terminal alkyne (2.2 equiv), and O_2 (2 atm) in DMSO at 70 °C for 12 h. The product **3aa** was characterized by GC-MS, HRMS, and by ^1H and ^{13}C NMR spectral analyses.

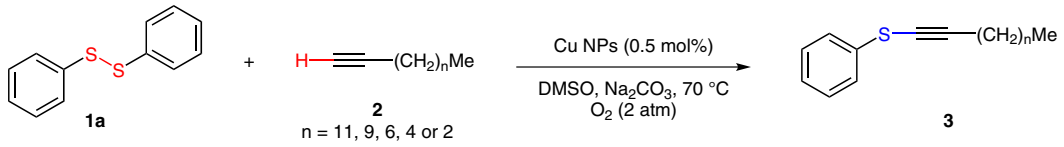
With the optimized reaction conditions established, the substrate scope and limitations of the coupling reaction were investigated; the results are summarized in Table 2. All the experiments were conducted with 500 mg diphenyl disulfide (**1a**). In general, the reaction proceeded well and afforded the desired alkynyl sulfides **3** in moderate yields. An assortment of long-chain terminal alkynes **2b–f** [1-tetradecyne (**2b**), 1-dodecyne (**2c**), 1-nonyne (**2d**), 1-heptyne (**2e**), and 1-pentyne (**2f**)] afforded the corresponding alkynylphenyl sulfides in good to moderate yields (entries 1–5). Unfortunately, in the case of 1-ethynylcyclohexene (**2g**), no cross-coupling product was observed under the standard conditions.

Table 1 Optimization of the Cu NP Catalyzed Cross-Coupling Reaction of **1a** with **2a**

Entry	Cat. (mol%)	Base	Additive	Yield (%)[b]
1	0.5	Na_2CO_3	–	37
2	2	Na_2CO_3	–	40
3	0.5	Na_2CO_3	–	39[c]
4	–	Na_2CO_3	–	ND
5	0.5	K_2CO_3	–	30
6	0.5	Cs_2CO_3	–	1
7	0.5	Ag_2CO_3	–	4
8	0.5	Li_2CO_3	–	5
9	0.5	K_3PO_4	–	trace
10	0.5	Na_2CO_3	zinc dust	ND
11	0.5	Na_2CO_3	manganese powder	30
12	0.5	Na_2CO_3	2,2′-bipyridyl	27
13	0.5	Na_2CO_3	phenanthroline	2
14	0.5	Na_2CO_3	TMEDA	27
15	0.5	Na_2CO_3	O_2 (1 atm)	38
16	0.5	Na_2CO_3	Ar (1 atm)	ND
17	0.5	Na_2CO_3	O_2 (2 atm)	77
18	1	Na_2CO_3	O_2 (2 atm)	80
19	0.5	Na_2CO_3	O_2 (3 atm)	79
20	0.5	Na_2CO_3	O_2 (2 atm)	76[d]
21	0.5	Na_2CO_3	O_2 (2 atm)	12[e]

[a] Reaction conditions: **1a** (100 mg, 1 equiv), **2a** (2.2 equiv), Cu NP, additive, base (2.2 equiv), DMSO (4 mL), 12 h, 70 °C.
[b] Determined by GC-MS analysis (referenced to diphenyl disulfide).
[c] Reaction for 24 h.
[d] Scale-up to ca. 500 mg diphenyl disulfide.
[e] Reaction conducted at r.t.

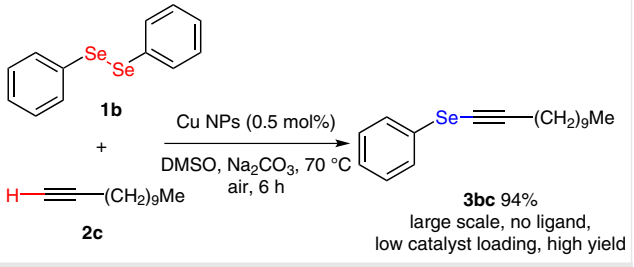
Table 2 Cross-Coupling of Diphenyl Disulfide (**1a**) and Alkynes



Entry	Alkyne	Product	Yield (%)[a]
1	H—(CH2)11Me **2b**	S—(CH2)11Me **3ab**	77
2	H—(CH2)9Me **2c**	S—(CH2)9Me **3ac**	61
3	H—(CH2)6Me **2d**	S—(CH2)6Me **3ad**	72
4	H—(CH2)4Me **2e**	S—(CH2)4Me **3ae**	63
5	H—(CH2)2Me **2f**	S—(CH2)2Me **3af**	54
6	H— **2g**	S— **3ag**	ND

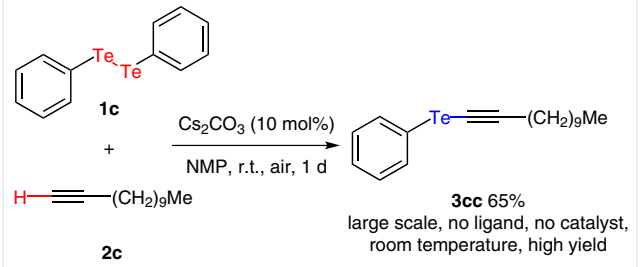
[a] Determined by GC-MS analysis.

The protocol could also be applied to the reaction of diphenyl diselenide with long chain alkyne 1-dodecyne under air (atmospheric air as oxidant) to afford the corresponding cross-coupled product alkynylphenyl selenide **3bc** in excellent yield under mild conditions (Scheme 2).



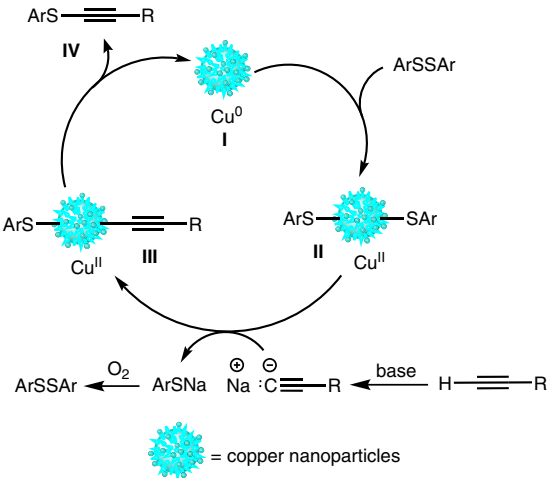
Scheme 2 Cross-coupling of diphenyl diselenide and 1-dodecyne under air

Applying the reaction conditions to diphenyl ditellurides led to the formation of **3cc**. In this case, the reaction proceeded smoothly even in the absence of copper nanoparticles at room temperature and rendered the product alkynylphenyl telluride in 65% yield by utilizing only *N*-methyl-2-pyrrolidone (NMP) as solvent and just 10 mol% Cs_2CO_3 as base (Scheme 3).



Scheme 3 Cross-coupling of diphenyl ditelluride and 1-dodecyne under air

Based on existing reports,[14,35,36] we hypothesize that the oxidative addition of Cu(0) to diphenyl disulfide affords intermediate **II** followed by the insertion of **II** to alkyne in the presence of base and oxygen to generate intermediate **III**. Finally, reductive elimination of **III** furnishes the corresponding cross-coupled product **IV**, as illustrated in Scheme 4.



Scheme 4 Plausible catalytic cycle for Cu NP catalyzed alkynylation of diphenyl disulfide

In summary, we have disclosed an efficient catalytic system that activates S–S and C–H bonds efficiently and that leads to the formation of S–C_{sp} bonds. This methodology permits the preparation of challenging compounds and proceeds in the absence of ligands under mild conditions with inexpensive and green oxidant with low catalyst loading. Remarkably, the present technique is also compatible for use with diphenyl diselenides, and analogous tellurides proceed through a cesium effect.

All starting materials were purchased from Aldrich Chemical Co., TCI, or Strem Chemical Co. and used as received. Cesium carbonate was purchased from Acros Organics (Cat. No. 192041000). Reaction products were analyzed by GC-MS (Shimadzu-QP2010 SE), and by ^{1}H and ^{13}C NMR spectroscopy (Varian Mercury Plus, 300 MHz). Chemical shift values are recorded as parts per million (ppm) relative to tetramethylsilane as internal standard, unless otherwise indicated; coupling constant values are in Hz. HRMS analysis was carried out at the Korea Basic Science Institute. The copper nanoparticle catalysts were synthesized as described in our previous publication.[33]

Alkynylation of Diphenyl Sisulfides; General Procedure

An autoclave was charged with diphenyl disulfide (1 equiv), alkyne (2.2 equiv), base (2.2 equiv), catalyst (0.5 mol%), DMSO (4 mL), and a magnetic stirring bar. The autoclave was purged and filled with O_2 to the stated gauge pressure (2 atm), and the reaction mixture was stirred at 70 °C for 12 h. After the reaction, the reaction mixture was diluted with Et_2O, passed through Celite, washed with H_2O, and the organic extract was dried with anhydrous $MgSO_4$, filtered, and the solvent was removed under rotary evaporation. The crude product was analyzed by GC-MS. After analysis, the pure product was isolated by column chromatography using hexane as eluent.

Synthesis of Alkynyl Selenides

To a Schlenk tube, diphenyl diselenide (1.60 mmol), 1-dodecyne (3.52 mmol), Na_2CO_3 (3.52 mmol), and DMSO (2 mL) were added, followed by Cu NPs (0.5 mol%) dispersed after sonication in DMSO (2 mL). The mixture was stirred at 70 °C for 6 h, then worked up as described for the sulfides.

Synthesis of Alkynyl Tellurides

To a Schlenk tube, diphenyl ditelluride (1.22 mmol), 1-dodecyne (2.68 mmol), Cs_2CO_3 (10 mol%), and NMP (4 mL) were added and the mixture was stirred at r.t. for 24 h. After the reaction, the mixture was diluted with Et_2O and washed with H_2O. The combined organic extracts were dried with anhydrous $MgSO_4$, filtered, and the solvent was removed under rotary evaporation. After GC-MS analysis, the crude product was purified by chromatography on silica gel using hexane as an eluent to afford the desired product.

Dec-1-yn-1-yl(phenyl)sulfane (3aa)

Yield: 434 mg (77%); colorless oil.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 6.3 Hz, 3 H), 1.27–1.46 (m, 10 H), 1.55–1.65 (m, 2 H), 2.45 (t, *J* = 6.3 Hz, 2 H), 7.16–7.21 (m, 1 H), 7.29–7.34 (m, 2 H), 7.39–7.42 (m, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.1, 20.3, 22.7, 28.6, 28.9, 29.1, 29.3, 29.5, 29.6, 31.9, 64.4, 100.1, 125.7, 126.0, 129.0, 133.8.

HRMS (EI): *m*/*z* calcd for $C_{16}H_{22}S$: 246.1442; found: 246.1442.

Tetradec-1-yn-1-yl(phenyl)sulfane (3ab)

Yield: 533 mg (77%); colorless oil.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.91 (t, *J* = 6.3 Hz, 3 H), 1.29–1.67 (m, 20 H), 2.47 (t, *J* = 7.2 Hz, 2 H), 7.18–7.23 (m, 1 H), 7.31–7.44 (m, 4 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.2, 20.4, 22.8, 28.8, 29.0, 29.2, 29.5, 29.7, 29.8, 32.0, 64.7, 100.2, 125.8, 126.2, 129.2, 133.9.

HRMS (EI): *m*/*z* calcd for $C_{20}H_{30}S$: 302.2068; found: 302.2068.

Dodec-1-yn-1-yl(phenyl)sulfane (3ac)

Yield: 383 mg (61%); colorless oil.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.89 (t, *J* = 6.3 Hz, 3 H), 1.29–1.46 (m, 14 H), 1.56–1.65 (m, 2 H), 2.45 (t, *J* = 6.9 Hz, 2 H), 7.16–7.21 (m, 1 H), 7.29–7.34 (m, 2 H), 7.39–7.42 (m, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.1, 20.3, 22.6, 28.6, 28.9, 29.1, 29.2, 31.8, 64.4, 100.1, 125.7, 126.0, 129.0, 133.7.

HRMS (EI): *m*/*z* calcd for $C_{18}H_{26}S$: 274.1755; found: 274.1756.

Non-1-yn-1-yl(phenyl)sulfane (3ad)

Yield: 383 mg (72%); yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.89 (t, *J* = 6.6 Hz, 3 H), 1.29–1.46 (m, 8 H), 1.55–1.65 (m, 2 H), 2.44 (t, *J* = 6.9 Hz, 2 H), 7.16–7.21 (m, 1 H), 7.29–7.34 (m, 2 H), 7.38–7.42 (m, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.1, 20.3, 22.6, 28.7, 28.8, 28.9, 31.8, 64.5, 100.1, 125.7, 126.0, 133.8.

HRMS (EI): *m*/*z* calcd for $C_{15}H_{20}S$: 232.1286; found: 232.1286.

Hept-1-yn-1-yl(phenyl)sulfane (3ae)

Yield: 294 mg (63%); colorless oil.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.92 (t, *J* = 7.2 Hz, 3 H), 1.31–1.47 (m, 4 H), 1.56–1.66 (m, 2 H), 2.45 (t, *J* = 7.2 Hz, 2 H), 7.17–7.22 (m, 1 H), 7.29–7.35 (m, 2 H), 7.38–7.43 (m, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.0, 20.3, 22.2, 28.3, 31.0, 64.5, 100.2, 125.7, 126.1, 129.0, 133.8.

HRMS (EI): *m*/*z* calcd for $C_{13}H_{16}S$: 204.0973; found: 204.0973.

Pent-1-yn-1-yl(phenyl)sulfane (3af)

Yield: 218 mg (54%); yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.04 (t, *J* = 6.9 Hz, 3 H), 1.57–1.68 (m, 2 H), 2.44 (t, *J* = 7.2 Hz, 2 H), 7.17–7.22 (m, 1 H), 7.29–7.34 (m, 2 H), 7.39–7.43 (m, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.6, 22.1, 22.3, 64.6, 99.9, 125.7, 126.0, 129.0, 133.7.

HRMS (EI): *m*/*z* calcd for $C_{11}H_{12}S$: 176.0660; found: 176.0660.

Dodec-1-yn-1-yl(phenyl)selane (3bc)

Yield: 485 mg (94%); yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 6.3 Hz, 3 H), 1.26–1.45 (m, 14 H), 1.54–1.64 (m, 2 H), 2.45 (t, *J* = 14.1 Hz, 2 H), 7.20–7.33 (m, 3 H), 7.49–7.53 (m, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.1, 20.6, 22.7, 28.7, 28.9, 29.1, 29.3, 29.5, 29.6, 31.9, 57.3, 104.8, 126.7, 128.4, 128.5, 129.3.

HRMS (EI): *m*/*z* calcd for $C_{18}H_{26}Se$: 322.1200; found: 322.1200.

Dodec-1-yn-1-yl(phenyl)tellane (3cc)

Yield: 294 mg (65%); yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 6.3 Hz, 3 H), 1.27–1.43 (m, 14 H), 1.52–1.61 (m, 2 H), 2.57 (t, *J* = 6.9 Hz, 2 H), 7.22–7.27 (m, 3 H), 7.65–7.68 (m, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.1, 21.1, 22.7, 28.8, 28.9, 29.1, 29.3, 29.5, 29.6, 31.9, 34.6, 113.1, 116.2, 127.6, 129.2, 129.6, 134.7, 137.6.

HRMS (EI): *m*/*z* calcd for $C_{18}H_{26}Te$: 372.1097; found: 372.1098.

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Supporting Information

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