Reductive coupling of disulfides and diselenides with alkyl halides catalysed by a silica-supported phosphine rhodium complex using hydrogen as a reducing agent

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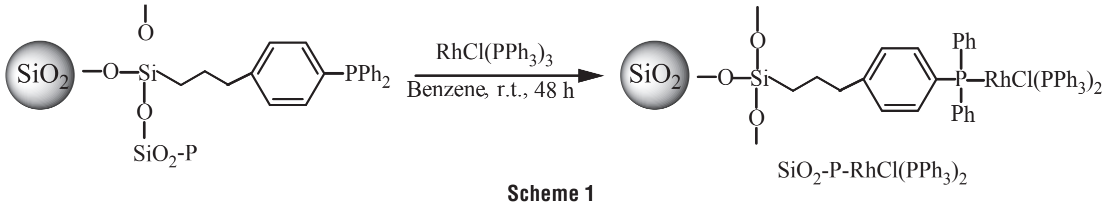
The reductive coupling of disulfides and diselenides with alkyl halides was achieved in THF at 65 °C in the presence of 3 mol% of a silica-supported phosphine rhodium complex and triethylamine using hydrogen as a reducing agent, affording a variety of unsymmetrical sulfides and selenides in high yields. The heterogeneous rhodium catalyst can be recovered by a simple filtration and reused several times without significant loss of activity. Reaction with an acyl halide was also observed.

Keywords: supported catalyst, phosphine rhodium complex, sulfide, selenide, heterogeneous catalysis

Organic sulfides and selenides have been used as versatile reagents in organic synthesis.[1–4] Recently, transition-metal-catalysed carbon–heteroatom cross-coupling reactions have provided convenient routes for the preparation of unsymmetrical sulfides and selenides. For instance, unsymmetrical diaryl sulfides could be obtained by copper-catalysed carbon-sulfur coupling of thiophenols with aryl iodides or aryl boronic acids;[5–7] unsymmetrical diorganyl selenides can be synthesised by ruthenium-catalysed reactions of dibenzyl or diphenyl diselenides with alkyl halides in the presence of zinc.[4] The reductive coupling of disulfides or diselenides with alkyl or aryl halides is an important reaction in the preparation of unsymmetrical sulfides and selenides, which can avoid the use of unstable and odoriferous thiols and selenols.[8–10] Although some efficient reductive coupling reactions using disulfides or diselenides have been reported, they require stoichiometric amount of metal reducing agents.[11–16] Tanaka *et al.*[17] described a $RhCl(PPh_3)_3$-catalysed reductive coupling of disulfides or diselenides with alkyl halides using hydrogen as a reducing agent, providing a convenient new route for the synthesis of unsymmetrical sulfides and selenides from disulfides or diselenides instead of thiols and selenols. However, industrial applications of homogeneous rhodium complexes remain a challenge because they are expensive, cannot be recycled and are difficult to separate from the product mixture, which is a particularly significant drawback for their application in the pharmaceutical industry. In contrast, heterogeneous catalysts can be easily separated from the reaction mixture by simple filtration and reused in successive reactions provided that the active sites have not become deactivated. The immobilisation of catalytically active species, *i.e.* organometallic complexes, onto solid supports to produce molecular heterogeneous catalysts has attracted much attention because they can combine the advantages of easy catalyst recovery, characteristic of a heterogeneous catalyst, with the high activity and selectivity of soluble complexes.[18] Heterogeneous catalysis also helps to minimise wastes derived from reaction workup, contributing to the development of green chemical processes.[19,20]

The immobilisation of rhodium complexes through covalent bond formation with functional groups on silica is the most commonly employed method to prepare surface organometallic catalysts. So far, silica-supported rhodium catalysts have successfully been used for hydrogenation reactions,[21–23] hydroformylation reactions,[24–26] and hydrosilylation reaction.[27,28] However, carbon–heteroatom bond formation reaction catalysed by heterogeneous rhodium complexes has received less attention.[29] In continuing our efforts to develop greener synthetic pathways for organic transformations, in this paper, we have designed and synthesised a new silica-supported phosphine rhodium(I) complex $[SiO_2\text{–P–RhCl}(PPh_3)_2]$, which is used as an effective rhodium catalyst for the reductive coupling of disulfides or diselenides with alkyl halides using hydrogen as a reducing agent.

The novel silica-supported phosphine rhodium complex $[SiO_2\text{–P–RhCl}(PPh_3)_2]$ was conveniently synthesised by the reaction of triphenylphosphine-functionalised silica (SiO_2–P)[30] with $RhCl(PPh_3)_3$ (Scheme 1). In our initial screening experiments, the reductive coupling reaction of di-*p*-tolyl disulfide with 1-bromododecane was investigated to optimise the reaction conditions, and the results are summarised in Table 1. At first, the temperature effect was examined and a significant effect was observed. For the temperatures studied [25, 50, 65, and 80 °C], 65 °C gave the best result and the reaction proceeded slowly at 25 °C. Other reaction temperatures such as 50 and 80 °C also gave good results. Our next studies focused on the effect of solvent on the model reaction. Among the solvents used [toluene, benzene, THF, and dioxane], THF was the best choice. Increasing the amount of rhodium catalyst could shorten the reaction time, but did not increase the yield of *p*-tolyl dodecyl sulfide (entry 9). Low rhodium concentration usually led to a long period of reaction, as indicated by our experimental result (entry 10). No reaction was observed in the absence of rhodium catalyst or hydrogen. An excellent result was obtained when the reductive coupling reaction was carried out with 3 mol% of the rhodium catalyst in a mixture of THF and Et_3N (5:1, v/v) at 65 °C (entry 6).



Scheme 1

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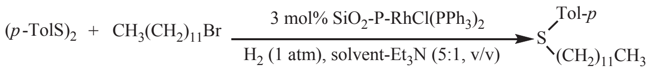
Table 1 Screening of reaction conditions for the coupling reaction of di-*p*-tolyl disulfide with 1-bromododecane catalysed by SiO_2–P–$RhCl(PPh_3)_2$[a]



$$(p\text{-TolS})_2 + CH_3(CH_2)_{11}Br \xrightarrow[H_2\text{ (1 atm), solvent-}Et_3N\text{ (5:1, v/v)}]{3\text{ mol\% }SiO_2\text{-P-}RhCl(PPh_3)_2} S(\text{Tol-}p)(CH_2)_{11}CH_3$$

Entry	Solvent	Temp./°C	Time/h	Yield/%[b]
1	Toluene	25	36	53
2	Toluene	50	30	81
3	Toluene	65	24	89
4	Toluene	80	16	86
5	THF	50	30	87
6	THF	65	24	93
7	Benzene	65	24	80
8	Dioxane	65	24	77
9[c]	THF	65	18	92
10[d]	THF	65	48	90

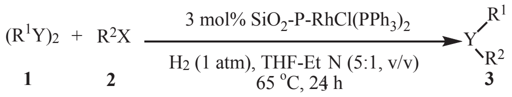
[a] Reaction was carried out with di-*p*-tolyl disulfide (0.2 mmol), 1-bromododecane (0.44 mmol) in THF (1.0 mL) and Et_3N (0.2 mL).

[b] Isolated yield based on disulfide.

[c] 5 mol% Rh catalyst was used.

[d] 2 mol% Rh catalyst was used.

Table 2 Reductive coupling of disulfides and diselenides with alkyl halides catalysed by SiO_2–P–$RhCl(PPh_3)_2$[a]



$$\underset{\mathbf{1}}{(R^1Y)_2} + \underset{\mathbf{2}}{R^2X} \xrightarrow[H_2\text{ (1 atm), THF-Et N (5:1, v/v)}\ 65\,^{o}C,\ 24\text{ h}]{3\text{ mol\% }SiO_2\text{-P-}RhCl(PPh_3)_2} \underset{\mathbf{3}}{Y(R^1)(R^2)}$$

Entry	$(R^1Y)_2$	R^2–X	Product	Yield/%[b]
1	$(p\text{-TolS})_2$	$CH_3(CH_2)_{11}Br$	**3a**	93
2	$(4\text{-ClC}_6H_4S)_2$	$CH_3(CH_2)_{11}Br$	**3b**	90
3	$(4\text{-MeOC}_6H_4S)_2$	$CH_3(CH_2)_{11}Br$	**3c**	91
4	$(PhCH_2S)_2$	$CH_3(CH_2)_{11}Br$	**3d**	83
5	$(n\text{-}C_{12}H_{25}S)_2$	$CH_3(CH_2)_{11}Br$	**3e**	86
6	$(p\text{-TolS})_2$	$PhCH_2Cl$	**3f**	87
7[c]	$(p\text{-TolS})_2$	PhCOCl	**3g**	89
8[c]	$(p\text{-TolS})_2$	EtO_2CCH_2Br	**3h**	56
9	$(PhSe)_2$	$CH_3(CH_2)_{11}Br$	**3i**	91
10	$(PhSe)_2$	$PhCH_2Cl$	**3j**	90
11	$(PhSe)_2$	PhCOCl	**3k**	82

[a] Reaction was carried out with disulfide or diselenide (0.2 mmol), alkyl halide or acyl halide (0.44 mmol) in THF (1.0 mL) and Et_3N (0.2 mL).

[b] Isolated yield based on disulfide or diselenide.

[c] Reaction was conducted with 5 mol% catalyst in toluene at 100 °C for 36 h.

With this promising result in hand, we started to investigate the scope of this reaction under the optimised conditions. A series of disulfides and alkyl halides (and one acyl halide) were subjected to the above optimal reaction conditions and the results are summarised in Table 2. As shown in Table 2, the reductive coupling reactions of aryl, benzyl, and alkyl disulfides with 1-bromododecane proceeded smoothly under mild conditions in good to excellent yields (entries 1–5). The reaction of di-*p*-tolyl disulfide with benzyl chloride also proceeded effectively to give the corresponding benzyl *p*-tolyl sulfide in 87% yield (entry 6). However, when ethyl bromoacetate and benzoyl chloride were used as the substrates, the reactions required a high catalyst loading (5 mol%) and high reaction temperature (100 °C) and good to high yields were obtained on a prolonged reaction time (36 h) (entries 7 and 8). We were pleased to find that the heterogeneous rhodium-catalysed reductive coupling protocol can be applicable to the preparation of various selenides. The reactions of diphenyl diselenide with 1-bromododecane, benzyl chloride, and benzoyl chloride proceeded smoothly under the conditions optimised for disulfides to afford the corresponding selenides in good to excellent yields (entries 9–11).

To verify whether the observed catalysis was due to the heterogeneous catalyst SiO_2–P–$RhCl(PPh_3)_2$ or a leached rhodium species in solution, we performed a hot filtration test.[31] We focused on the reductive coupling reaction of di-*p*-tolyl disulfide with 1-bromododecane. We filtered off the SiO_2–P–$RhCl(PPh_3)_2$ after 12 h of reaction time and allowed the filtrate to react further. The catalyst filtration was performed at the reaction temperature (65 °C) in order to avoid possible recoordination or precipitation of soluble rhodium upon cooling. We found that, after this hot filtration, no further reaction was observed and no rhodium could be detected in the hot filtered solution by ICP-AES. This result suggests that the rhodium catalyst remains on the support at elevated temperatures during the reaction and points to a process of heterogeneous nature. The reusability of the SiO_2–P–$RhCl(PPh_3)_2$ complex was tested through the repeated reaction of di-*p*-tolyl disulfide with 1-bromododecane. The SiO_2–P–$RhCl(PPh_3)_2$ complex can be easily recovered by a simple filtration of the reaction solution and washed with THF and diethyl ether. After being air-dried, it can be reused directly in the next run without further purification. The recovered catalyst was used in the next run and almost consistent activity was observed for five consecutive cycles (Table 3). This result is important from a practical point of view.

In summary, we have successfully developed a novel, practical and environmentally friendly method for the synthesis of a variety of unsymmetrical sulfides and selenides through the reductive coupling of disulfides and diselenides with alkyl halides by using a silica-supported phosphine rhodium complex [SiO_2–P–$RhCl(PPh_3)_2$] as catalyst and hydrogen as a reducing agent under mild reaction conditions. The reactions generated the corresponding unsymmetrical sulfides and selenides in good to excellent yields. This heterogeneous rhodium catalyst could be easily recovered and recycled by a simple filtration of the reaction solution and reused for five cycles without significant loss of activity, thus making this procedure environmentally more acceptable.

Table 3 Reusability study of the SiO_2–P–$RhCl(PPh_3)_2$ in the reductive coupling reaction of di-*p*-tolyl disulfide with 1-bromododecane[a]

Cycle	1	2	3	4	5
Yield/%[b]	93	92	91	91	90

[a] Reaction conditions: di-*p*-tolyl disulfide (0.2 mmol), 1-bromododecane (0.44 mmol), SiO_2–P–$RhCl(PPh_3)_2$ (3 mol%) in THF (1.0 mL) and Et_3N (0.2 mL) at 65 °C for 24 h under 1 atm of H_2.

[b] Isolated yield.

Experimental

THF was distilled from sodium prior to use, Et_3N was dried over KOH and distilled before use. All other reagents were used as received without further purification. All reaction were carried out under an atmosphere of Ar in oven-dried glassware with magnetic stirring. ^1H NMR spectra were recorded on a Bruker Avance 400 (400 MHz) spectrometer with TMS as an internal standard using $CDCl_3$ as the solvent. ^{13}C NMR spectra were recorded on a Bruker Avance 400 (100 MHz) spectrometer using $CDCl_3$ as the solvent. The triphenylphosphine-functionalised silica (SiO_2–P) was prepared according to a literature method,[30] the phosphine content was determined to be 0.71 mmol g^{-1} by elemental analysis.

Synthesis of the SiO_2–P–$RhCl(PPh_3)_2$ complex

SiO_2–P (2.0 g) was added to a solution of $RhCl(PPh_3)_3$ (1.109 g, 1.2 mmol) in benzene (50 mL). The mixture was stirred under an argon atmosphere at 25 °C for 48 h. The solid product was filtered by suction, washed with benzene (5 × 10 mL), and dried at 70 °C / 26.7 Pa under an argon atmosphere for 3 h to give the light yellow rhodium complex [SiO_2–P–RhCl(PPh_3)$_2$] (2.68 g). The phosphine and rhodium content was 1.34 mmol g^{-1} and 0.38 mmol g^{-1}, respectively.

Reactions of disulfides and diselenides with alkyl halides; general procedure

A 25 mL, three-necked round-bottom flask equipped with a reflux condenser and a magnetic stir bar was charged sequentially with SiO_2–P–RhCl(PPh_3)$_2$ (16 mg, 0.006 mmol), disulfide or diselenide (0.2 mmol), alkyl halide (0.44 mmol), THF (1.0 mL), and Et_3N (0.2 mL) under argon. Then hydrogen was introduced to the resulting suspension. The mixture was stirred at 65 °C for 24 h. After being cooled to room temperature, the mixture was diluted with Et_2O (10 mL) and filtered. The SiO_2–P–RhCl(PPh_3)$_2$ complex was washed with THF (2 × 5 mL) and Et_2O (2 × 5 mL) and reused in the next run. The ether solution was concentrated under a reduced pressure, and the residue was purified by preparative TLC (hexane) to afford the desired product.

Dodecyl p-tolyl sulfide (**3a**): Oil.[32] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26–7.19 (m, 2H), 7.07 (d, J = 8.0 Hz, 2H), 2.87 (t, J = 7.4 Hz, 2H), 2.32 (s, 3H), 1.60 (quint, J = 7.4 Hz, 2H), 1.43–1.20 (m, 18H), 0.89 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 135.8, 133.2, 129.7, 129.6, 34.4, 31.9, 29.6, 29.5, 29.3, 29.2, 28.8, 22.8, 21.1, 14.0.

Dodecyl p-chlorophenyl sulfide (**3b**): White solid, m.p. 35–36 °C (lit.[33] 36–37 °C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26–7.21 (m, 4H), 2.89 (t, J = 7.4 Hz, 2H), 1.61 (quint, J = 7.4 Hz, 2H), 1.45–1.20 (m, 18H), 0.89 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 135.7, 131.6, 130.2, 129.0, 33.9, 31.9, 29.6, 29.5, 29.3, 29.2, 29.1, 28.8, 22.7, 14.1.

Dodecyl p-methoxyphenyl sulfide (**3c**): White solid, m.p. 45–46 °C (lit.[17] 44–45 °C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36–7.30 (m, 2H), 6.87–6.80 (m, 2H), 3.77 (s, 3H), 2.81 (t, J = 7.4 Hz, 2H), 1.58 (quint, J = 7.4 Hz, 2H), 1.45–1.19 (m, 18H), 0.88 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.7, 132.9, 127.1, 114.5, 55.3, 35.9, 31.9, 29.6, 29.5, 29.3, 29.1, 28.8, 22.7, 14.1.

Benzyl dodecyl sulfide (**3d**): Oil.[34] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36–7.20 (m, 5H), 3.71 (s, 2H), 2.42 (t, J = 7.4 Hz, 2H), 1.56 (quint, J = 7.4 Hz, 2H), 1.40–1.19 (m, 18H), 0.88 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.7, 128.9, 128.5, 126.9, 36.2, 31.9, 31.5, 29.6, 29.5, 29.3, 29.2, 28.9, 22.8, 14.0.

Didodecyl sulfide (**3e**): White solid, m.p. 36–37 °C (lit.[17] 37–38 °C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.51 (t, J = 7.4 Hz, 4H), 1.58 (quint, J = 7.4 Hz, 4H), 1.43–1.20 (m, 36H), 0.89 (t, J = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 32.2, 31.9, 29.8, 29.7, 29.6, 29.5, 29.3, 29.2, 29.1, 22.7, 14.1.

Benzyl p-tolyl sulfide (**3f**): Oil.[35] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.27–7.16 (m, 7H), 7.06 (d, J = 8.0 Hz, 2H), 4.06 (s, 2H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.9, 136.5, 132.4, 130.8, 129.6, 128.7, 128.4, 127.1, 39.9, 21.1.

Benzenecarbothioic acid S-(4-methylphenyl)ester (**3g**): White solid, m.p. 72–73 °C (lit.[17] 72–74 °C). IR (KBr): ν (cm^{-1}) 2918, 1668, 1594, 1490, 1447; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10–7.99 (m, 2H), 7.66–7.55 (m, 1H), 7.54–7.37 (m, 4H), 7.32–7.22 (m, 2H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.6, 139.8, 136.7, 135.1, 133.5, 130.2, 128.7, 127.4, 123.7, 21.3.

4-Methyl(phenylthio)acetic acid ethylester (**3h**): Oil.[17] IR (film): ν (cm^{-1}) 3065, 2923, 1740, 1597, 1489, 1108; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37–7.30 (m, 2H), 7.16–7.09 (m, 2H), 4.16 (q, J = 7.4 Hz, 2H), 3.59 (s, 2H), 2.33 (s, 3H), 1.23 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.8, 137.4, 131.2, 130.9, 129.8, 61.5, 37.4, 21.1, 14.1.

Dodecyl phenyl selenide (**3i**): Oil.[36] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52–7.46 (m, 2H), 7.28–7.17 (m, 3H), 2.91 (t, J = 7.4 Hz, 2H), 1.70 (quint, J = 7.4 Hz, 2H), 1.45–1.21 (m, 18H), 0.88 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 132.3, 130.8, 128.9, 126.6, 31.9, 30.2, 29.8, 29.6, 29.5, 29.3, 29.1, 27.9, 22.7, 14.2.

Benzyl phenyl selenide (**3j**): Oil.[36] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46–7.40 (m, 2H), 7.31–7.19 (m, 8H), 4.11 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.6, 133.6, 130.5, 128.9, 128.8, 128.5, 127.4, 126.8, 32.3.

Benzoyl phenyl selenide (**3k**): Oil.[36] IR (film): ν (cm^{-1}) 3062, 1692, 1578, 1474, 1446; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96–7.90 (m, 2H), 7.64–7.55 (m, 3H), 7.52–7.37 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.4, 138.6, 136.4, 133.8, 129.4, 129.0, 128.9, 127.4, 125.8.

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