Transition Metal-Catalyzed Synthesis of (*E*)-2-(Alkylthio)alka-1,3-dienes from Allenes and Dialkyl Disulfides with Concomitant Hydride Transfer

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Abstract: Reaction of dialkyl disulfides or diselenides with allenes is catalyzed by a rhodium-phosphine complex and trifluoromethanesulfonic acid giving (*E*)-2-alkylthio(seleno)-1,3-dienes and (*E*)-2-alkylthio(seleno)-2-alkenes. Unlike the reaction of alkynes, the reaction of allene is accompanied by hydride transfer.

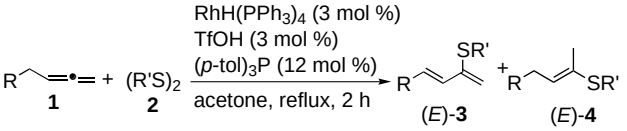
Keywords: (*E*)-2-(alkylthio)alka-1,3-diene; allene; diselenide; disulfide; hydride transfer; rhodium

Reaction of organic disulfides or diselenides with unsaturated compounds catalyzed by a transition metal complex is becoming one of the important methodologies for the synthesis of organosulfur and organoselenium compounds.[1] The *cis*-addition of disulfides and diselenides to alkynes and alkenes with S-S and Se-Se bond cleavage has been studied by others[2] and by us.[3] We have shown that a catalyst system of a rhodium-phosphine complex and trifluoromethanesulfonic acid is effective not only for the addition of aromatic disulfides to alkynes but also of aliphatic disulfides.[3] Notably, when allene is employed as the substrate, concomitant hydride transfer takes place giving (*E*)-2-alkylthio(seleno)-1,3-dienes and (*E*)-2-alkylthio(seleno)-2-alkenes. The results are contrasted to the fact that organic disulfides and diselenides undergo 1,2-addition to allenes under radical conditions.[4] Previous syntheses of (*E*)-2-(alkylthio)alka-1,3-dienes employed stepwise methods,[5,6] and the present reaction provides a convenient access to such organosulfur and organoselenium compounds.

When undeaca-1,2-diene **1a** was treated with dibutyl disulfide **2a** (0.5 equiv.) in the presence of $RhH(PPh_3)_4$ (3 mol %), tris(*p*-tolyl)phosphine (12 mol %), and trifluoromethanesulfonic acid (3 mol %) in refluxing acetone for 2 h, (*E*)-2-(butylthio)undeca-1,3-diene (*E*)-**3a** and (*E*)-2-butylthio-2-undecene (*E*)-**4a** were obtained in 47% and 47% yields, respectively (Table 1, entry 1). The stereochemistry of (*E*)-**3a** was determined by the ^{1}H-NMR coupling constant ($J = 15.6$ Hz) and that of (*E*)-**4a** by the NOE between 1- and 4-protons. Both the rhodium complex and trifluoromethanesulfonic acid are essential, and no reaction occurs in the absence of either of the reagents. Addition of tris(*p*-tolyl)phosphine increases the yields (*vide infra*). Since allene **1a** is used for the acceptor of thiol giving (*E*)-**4a**, the yield of (*E*)-**3a** does not exceed 50%. The mass balance, however, is almost quantitative. The reaction can be applied to various combinations of monosubstituted allenes and aliphatic disulfides (Table 1). A 1,3-diene derived from diphenyl disulfide is relatively unstable, and decomposes during silica gel chromatography (entry 8). A cystine derivative (*R*,*R*)-**2b** gives the adducts (*R*,*E*)-**3b** and (*R*,*E*)-**4b** without racemization (Scheme 1): Reaction of (*R*,*R*)-**2b** containing 3% of *meso*-**2b** gives (*R*,*E*)-**3b** and (*R*,*E*)-**4b** in more the 97% ee.

Alkyl diselenides react similarly. When **1a** was treated with dioctyl diselenide **5a** (0.5 equiv.) in the presence of $RhH(PPh_3)_4$ (5 mol %), tris(*p*-chlorophenyl)phosphine (15 mol %), and trifluoromethanesulfonic acid (5 mol %) in refluxing acetone for 30 min, (*E*)-2-(octylseleno)un-

Table 1. Rh-catalyzed addition of disulfides to allenes.



entry	R	R'	Yield [%][a] (*E*)-**3**	(*E*)-**4**
1	n-C_7H_{15} (**1a**)	n-C_4H_9 (**2a**)	47 (**3a**)	47 (**4a**)
2		C_2H_5	46	41
3		n-C_3H_7	43	42
4	n-C_5H_{11}	n-C_4H_9	43	43
5	$HO(CH_2)_2$		42	42
6	t-$BuMe_2SiOCH_2$		40	40
7	$PhCOOCH_2$	$HO(CH_2)_6$	38	38
8	n-C_7H_{15}	Ph	23[b]	23[b]

[a] Isolated yields.
[b] ^{1}H-NMR yields.

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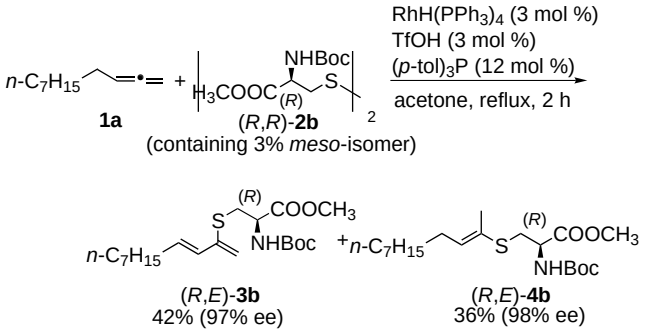
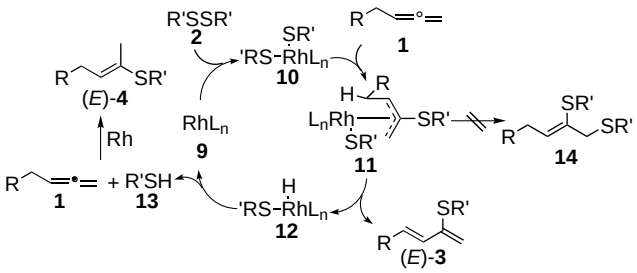
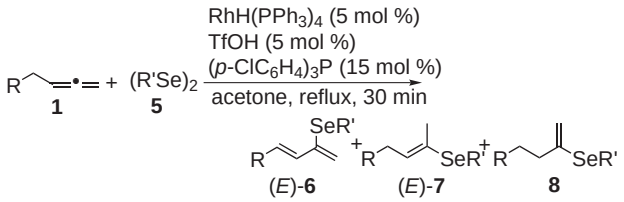


Scheme 1.



Scheme 2.

Table 2. Rh-catalyzed addition of diselenides to allenes

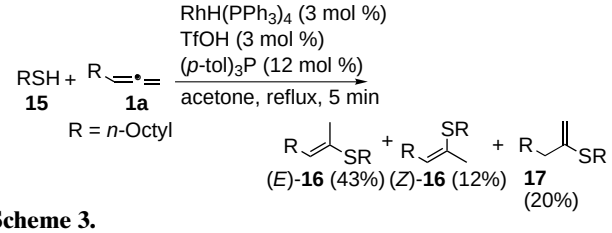


entry	R	R'	Yield (%)[a] (*E*)-**6**	(*E*)-**7**	**8**
1	*n*-C_7H_{15} (**1a**)	*n*-C_4H_9	37	28	4
2		*n*-C_8H_{17} (**5a**)	40 (**6a**)	28 (**7a**)	5 (**8a**)
3		$PhCH_2CH_2$	40	27	5
4		$CH_3(CH_2)CH(CH_2CH_3)CH_2$	41	29	9
5		$(CH_3)_3CCH_2$	47	23	17
6	*n*-C_5H_{11}	*n*-C_8H_{17}	37	26	-
7	$BzO(CH_2)_2$	*n*-C_4H_9	33	26	-
8	*n*-C_7H_{15}	Ph	9[b]	9[b]	-

[a] Isolated yields.
[b] H-NMR yields.

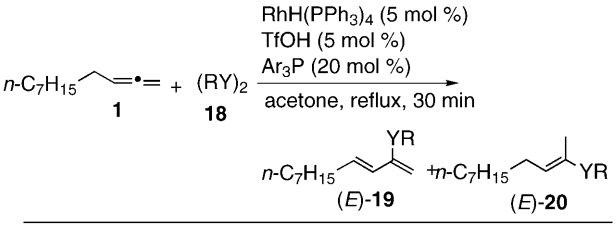
deca-1,3-diene (*E*)-**6a** and (*E*)-2-octylseleno-2-undecene (*E*)-**7a** were obtained in 40% and 28% yields, respectively (Table 2, entry 2). A small amount of an *exo*-olefin **8a** (5%) was also formed. The stereochemistry of (*E*)-**6a** was determined by the ^{1}H NMR coupling constant ($J = 15.6$ Hz), and that of (*E*)-**7a** by the NOE between the vinyl proton and α-CH_2 protons of the selenium atom. Diphenyl diselenide gives the adducts in low yields with the recovered starting materials (Table 2, entry 8), which may be due to deactivation of the catalyst.

A proposed mechanism is shown in Scheme 2. Oxidative addition of rhodium complex **9** and dialkyl disulfides gives **10**, to which allene **1** inserts forming a π-allyl complex **11**. β-Hydride elimination leading to **12** is more rapid than the reductive elimination giving **14**, and alkylthiol **13** is formed by the reductive elimination of **12**. 2-Alkylthioalkene (*E*)-**4** is obtained by the addition of **13** to **1**, which is also catalyzed by rhodium. Treatment of 1-octanethiol **15** and **1a** with the same catalyst system for 5 min gave adducts (*E*)-**16**, (*Z*)-**16**,



Scheme 3.

Table 3. Effect of phosphine.



$(RY)_2$	Ar	Yield (%) (*E*)-**19**	(*E*)-**20**	**18** (recovery)
$(n\text{-}C_4H_9S)_2$	none	28	28	16
	$p\text{-}CH_3C_6H_4$	40	38	-
	Ph	34	32	13
	$p\text{-}ClC_6H_4$	15	14	48
$(n\text{-}C_8H_{17}Se)_2$	none	31	28	18
	$p\text{-}CH_3C_6H_4$	28	20	50
	Ph	32	33	10
	$p\text{-}ClC_6H_4$	40	28	-

and **17** in 43%, 12%, and 20% yields, respectively (Scheme 3). Such adducts were not obtained in the absence of the rhodium complex. Formation of (*Z*)-**16** and **17** in the model reaction, however, suggests involvement of slightly different catalyst species in the actual reaction of **1** and **13** giving (*E*)-**4** (Scheme 2). Phosphines exhibit different reactivities for disulfide and diselenide (Table 3). Reaction of disulfide is promoted by tri(*p*-tolyl)phosphine, and that of diselenide by tris(*p*-chlorophenyl)phosphine. Some part of the mechanism differs depending to the heteroatoms.

Experimental Section

(*E*)-2-Butylthioundeca-1,3-diene [(*E*)-3a] and (*E*)-2-Butylthioundec-2-ene [(*E*)-4a]

In a two-necked flask equipped with a reflux condenser were placed tetrakis(triphenylphosphine)hydriderhodium (3 mol %, 17.1 mg), tris(*p*-tolyl)phosphine (12 mol %, 21 mg), trifluoromethanesulfonic acid (3 mol %, 1.4 μL), undeca-1,2-diene **1a** (0.5 mmol, 76.0 mg), and dibutyl disulfide **2a** (0.25 mmol, 47.5 μL) in acetone (2.0 mL) under an argon atmosphere, and the solution was heated at reflux for 2 h. Then, the solvent was removed under reduced pressure, and the residue was purified by flash chromatography on silica gel giving (*E*)-**3a** (56 mg, 47%) and (*E*)-**4a** (56 mg, 47%).

(*E*)-**3a**: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.88 (3H, t, *J* = 7.2 Hz), 0.93 (3H, t, *J* = 7.2 Hz), 1.21 – 1.32 (8H, m), 1.39 – 1.47 (4H, m), 1.64 (2H, quintet, *J* = 7.2 Hz), 2.11 (2H, q, *J* = 7.2 Hz), 2.74 (2H, t, *J* = 7.2 Hz), 4.89 (1H, s), 5.19 (1H, s), 6.02 (1H, dt, *J* = 15.6, 6.4 Hz), 6.13 (1H, d, *J* = 15.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.8, 14.2, 22.3, 22.8, 29.2, 29.3, 29.3, 30.7, 31.0, 31.9, 32.7, 109.5, 129.8, 133.0, 142.1. IR (neat): $\tilde{\nu}$ = 2957, 2925, 2855, 1568, 1465 cm^{-1}; MS (EI): *m/z* = 240 (M^+, 36%), 183 (M^+ – 57, 100%); HRMS: calcd. for $C_{15}H_{28}S$: 240.1912; found: 240.1911.

(*E*)-**4a**: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.88 (3H, t, *J* = 7.2 Hz), 0.92 (3H, t, *J* = 7.2 Hz), 1.23 – 1.35 (12H, m), 1.42 (2H, sextet, *J* = 7.2 Hz), 1.57 (2H, quintet, *J* = 7.2 Hz), 1.87 (3H, s), 2.06 (2H, q, *J* = 7.2 Hz), 2.65 (2H, t, *J* = 7.2 Hz), 5.39 (1H, dt, *J* = 7.2, 1.6 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 13.9, 14.3, 18.0, 22.2, 22.8, 29.0, 29.3, 29.4, 29.6, 29.7, 31.0, 31.2, 32.0, 127.1, 128.7; IR (neat): $\tilde{\nu}$ = 2956, 2924, 2854, 1465 cm^{-1}. MS (EI): *m/z* = 242 (M^+, 44%), 185 (M^+ – 57, 100%); HRMS: calcd. for $C_{15}H_{30}S$: 242.2068; found: 242.2055.

Methyl (*R*,*E*)-2-(*t*-butoxycarbonylamino)-3-(undeca-1,3-dienyl-2-thio)propanoate [(*R*,*E*)-3b]

The purity of the starting material (*R*,*R*)-**2b** was determined by HPLC using Daicel Chiralcel OD-H (0.25 mL/min, hexane/2-PrOH = 5): (*R*,*S*)-**2b**: t_1 = 16.86 min, (*S*,*S*)-**2b**: t_2 = 21.92 min, (*R*,*R*)-**2b**: t_3 = 24.27 min. HPLC conditions for the separation of (*R*,*E*)-**3b** and (*S*,*E*)-**3b** are as follows: 0.5 mL/min, hexane/2-PrOH = 10. (*S*,*E*)-**3b**: t_1 = 8.10 min, (*R*,*E*)-**3b**: t_2 = 9.22 min.

(*R*,*E*)-**3b**: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.88 (3H, t, *J* = 7.6 Hz), 1.23 – 1.31 (8H, m), 1.38 – 1.45 (2H, m), 1.45 (9H, s), 2.12 (2H, q, *J* = 6.8 Hz), 3.13 (1H, dd, *J* = 13.2, 6.0 Hz), 3.20 (1H, dd, *J* = 13.2, 5.2 Hz), 3.75 (3H, s), 4.59 (1H, q, *J* = 7.2 Hz), 5.11 (1H, s), 5.28 (1H, s), 5.30 (1H, d, *J* = 8.0 Hz), 6.03 (1H, dt, *J* = 16.0, 6.8 Hz), 6.10 (1H, d, *J* = 16.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.2, 22.8, 28.4, 29.1, 29.2, 29.3, 31.9, 32.7, 34.1, 52.5, 52.9, 80.1, 113.5, 129.0, 134.3, 140.4, 154.8, 171.0; IR (neat): $\tilde{\nu}$ = 3372, 2926, 2855, 1749, 1717, 1503, 1366, 1168, 1015 cm^{-1}; MS (EI): *m/z* = 385 (M^+, 0.2%), 284 (M^+ – 101, 39%), 57 (M^+ – 328, 100%); HRMS: calcd. for $C_{20}H_{35}NSO_4$: 385.2286; found: 385.2278; $[\alpha]_D^{23}$: 13.60 (*c* 0.6, $CHCl_3$).

Methyl (*R*,*E*)-2-(*t*-butoxycarbonylamino)-3-(undecenyl-2-thio)propanoate [(*R*,*E*)-4b]

HPLC conditions for the separation of (*R*,*E*)-**4b** and (*S*,*E*)-**4b** are as follows: 0.25 mL/min, hexane/2-PrOH = 20: (*S*,*E*)-**4b**: t_1 = 18.24 min, (*R*,*E*)-**4b**: t_2 = 19.58 min.

(*R*,*E*)-**4b**: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.88 (3H, t, *J* = 7.2 Hz), 1.24 – 1.36 (12H, m), 1.45 (9H, s), 1.90 (3H, s), 2.04 (2H, q, *J* = 7.6 Hz), 3.04 (1H, dd, *J* = 15.2, 5.2 Hz), 3.09 (1H, dd, *J* = 15.2, 5.2 Hz), 3.73 (3H, s), 4.53 – 4.57 (1H, m), 5.32 (1H, d, *J* = 7.2 Hz), 5.66 (1H, t, *J* = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.2, 17.7, 22.8, 28.4, 29.2, 29.3, 29.3, 29.3, 29.5, 32.0, 33.3, 52.4, 53.5, 80.0, 127.1, 132.5, 154.9, 171.1; IR (neat): $\tilde{\nu}$ = 3378, 2926, 2855, 1750, 1718, 1503, 1366, 1168, 1053, 1016 cm^{-1}; MS (EI): *m/z* = 387 (M^+, 14%), 152 (M^+ – 235, 69%), 57 (M^+ – 330, 100%); HRMS: calcd. for $C_{20}H_{37}NSO_4$: 387.2443; found: 387.2423; $[\alpha]_D^{23}$: 27.20 (*c* 1.0, $CHCl_3$).

(*E*)-2-(Octylseleno)undeca-1,3-diene [(*E*)-6a], (*E*)-2-Octylselenoundec-2-ene [(*E*)-7a], and 2-Octylselenoundec-1-ene (8a)

In a two-necked flask equipped with a reflux condenser were placed tetrakis(triphenylphosphine)hydriderhodium (5 mol %, 28.8 mg), tris(*p*-chlorophenyl)phosphine (15 mol %, 27.4 mg), trifluoromethanesulfonic acid (5 mol %, 2.2 μL), undeca-1,2-diene **1a** (0.5 mmol, 76.1 mg), and dioctyl diselenide **5a** (0.25 mmol, 96.0 mg) in acetone (2.0 mL) under an argon atmosphere, and the solution was heated at reflux for 30 min. Then, the solvent was removed under reduced pressure, and the residue was purified by flash chromatography on silica gel giving (*E*)-**6a** (69 mg, 40%), (*E*)-**7a** (48 mg, 28%), and **8a** (9 mg, 5%).

(*E*)-**6a**: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.87 (6H, t, *J* = 7.2 Hz), 1.22 – 1.34 (16H, m), 1.35 – 1.42 (4H, m), 1.68 (2H, quintet, *J* = 7.2 Hz), 2.12 (2H, q, *J* = 7.6 Hz), 2.72 (2H, t, *J* = 7.2 Hz), 5.18 (1H, s), 5.57 (1H, s), 5.97 (1H, dt, *J* = 15.2, 6.4 Hz), 6.11 (1H, d, *J* = 15.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.2, 22.7, 25.6, 29.2, 29.2, 29.2, 29.8, 30.1, 31.9, 32.6, 115.6, 130.9, 135.0, 138.0. IR (neat): $\tilde{\nu}$ = 2956, 2924, 2853, 1573, 1464, 954 cm^{-1}. MS (EI): *m/z* = 344 (M^+, 46), 232 (M^+ – 112, 77), 231 (M^+ – 113, 66), 81 (M^+ – 263, 82), 69 (M^+ – 275, 88), 67 (M^+ – 277, 61), 57 (M^+ – 287, 64), 55 (M^+ – 289, 93), 43 (M^+ – 301, 100), 41 (M^+ – 303, 82); HRMS: calcd. for $C_{19}H_{36}Se$: 344.1982; found: 344.1974.

(*E*)-**7a**: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.88 (6H, t, *J* = 7.2 Hz), 1.24 – 1.31 (20H, m), 1.33 – 1.41 (4H, m), 1.65 (2H, quintet, *J* = 7.2 Hz), 1.99 (3H, s), 2.05 (2H, q, *J* = 7.6 Hz), 2.67 (2H, t, *J* = 7.2 Hz), 5.66 (1H, t, *J* = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.2, 19.9, 22.7, 22.8, 25.0, 29.2, 29.3, 29.3, 29.3, 29.4, 29.5, 29.5, 30.0, 30.2, 31.9, 31.9, 123.9, 132.5; IR (neat): $\tilde{\nu}$ = 2956, 2924, 2853, 1456 cm^{-1}; MS (EI): *m/z* = 346 (M^+, 14), 55 (M^+ – 291, 77), 43 (M^+ – 303, 99), 41 (M^+ – 305, 100); HRMS: calcd. for $C_{19}H_{38}Se$: 346.2138; found: 346.2164.

8a: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.87 (6H, t, *J* = 7.2 Hz), 1.22 – 1.34 (20H, m), 1.38 (2H, quintet, *J* = 6.4 Hz), 1.51 (2H, quintet, *J* = 7.2 Hz), 1.71 (2H, quintet, *J* = 7.6 Hz), 2.27 (2H, t, *J* = 6.8 Hz), 2.70 (2H, t, *J* = 7.6 Hz), 4.97 (1H, s), 5.39 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.2, 22.7, 22.8, 25.3, 29.0, 29.2, 29.2, 29.4, 29.5, 29.6, 29.7, 30.2, 30.4, 31.0, 31.9, 31.9, 39.2, 111.3, 142.4; IR (neat): $\tilde{\nu}$ = 2956, 2925, 2853, 1464 cm^{-1}; MS

(EI): $m/z = 346$ (M^+, 89), 233 ($M^+ - 113$, 100), 97 ($M^+ - 249$, 88); HRMS: calcd. for $C_{19}H_{38}Se$: 346.2138; found: 346.2123.

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