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Urea–Hydrogen Peroxide Complex: A Selective Oxidant in the Synthesis of 2-Phenylselenyl-1,3-butadienes

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Abstract: 2-Phenylselenyl-1,3-butadienes were synthesized via the selective oxidation of 2,4-diphenylselenyl-1-butenes with urea–hydrogen peroxide followed by the selenoxide elimination.

Keywords: 1,3-Butadiene, methylenecyclopropane, oxidation, selenium, urea–hydrogen peroxide

For a long period, selenium compounds have attracted the attention of chemists because of their synthetic applications and biological activities including antitumor and antibacterial activities and other properties.[1] In the selenium methodology, the selenylated group could be easily introduced, transformed, and eliminated from the organic substrate to allow selective reactions under mild conditions. Conjugated dienes, which are also a kind of traditional building blocks in organic synthesis, have been widely investigated for decades.[2] The Diels–Alder reactions of conjugated dienes would help to build ring-contained organic molecules efficiently and conveniently. Therefore, 2-phenylselenyl-1,3-butadienes, containing both a selenium atom and a conjugated diene structure unit, may also play important roles in organic methodology research.[3]

In our previous investigations, we developed a method for the synthesis of 2-phenylselenyl-1,3-butadienes via the reaction of diaryl

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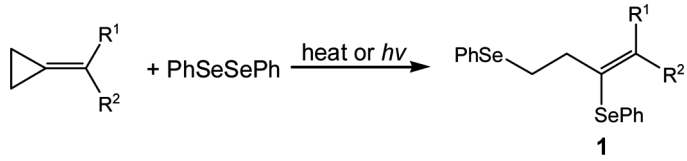
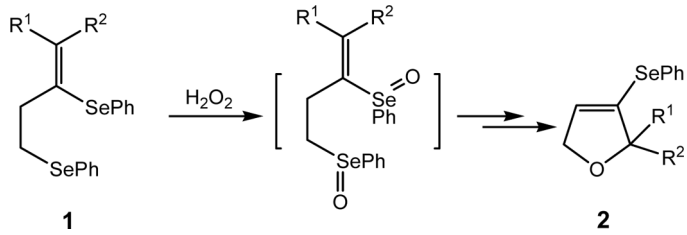


Scheme 1. Synthesis of acetic acid (4-bromo-phenyl)-(1-phenylselenyl-cyclopropyl)-methyl ester **5**.

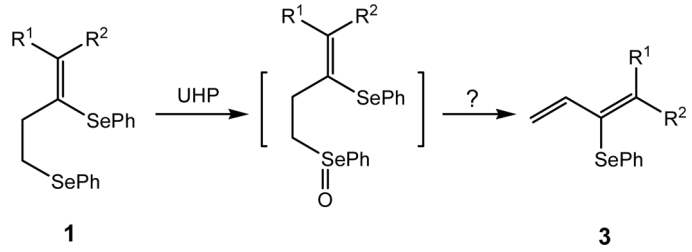
substituted methylenecyclopropanes (MCPs) with diphenyl diselenide in the presence of copper(II) acetate.[4] However, when alkyl substituted MCPs were employed, the corresponding 2-phenylselenyl-1,3-butadienes could not be obtained. This was probably because of the β-elimination of the organocopper intermediate with the adjacent hydrogen atom linked on the alkyl group. Thus, we tried to develop some other synthetic method that could be tolerant of the alkyl-substituted MCPs.

The selenoxide elimination has emerged as one of the synthetically most useful olefin-forming reactions.[1a] Hence, 2,4-diphenylselenyl-1-butenes **1**, which could be prepared via the free radical addition of MCPs with diphenyl diselenide[5] (Scheme 1), might be the possible material for the synthesis of 2-phenylselenyl-1,3-butadienes if the 4-position phenylselenyl group could be selectively oxidized. However, the literature has reported that when 2,4-diphenylselenyl-1-butenes were oxidized with 5 equivalents of hydrogen peroxide, both of their phenylselenyl groups were oxidized and 3-phenylselenyl-2,5-dihydrofurans **2** were obtained instead of the expected 2-phenylselenyl-1,3-butadienes[5a] (Scheme 2). This was probably due to the strong oxidation ability of hydrogen peroxide.

Hydrogen-bonded urea–hydrogen peroxide complex [$CO(NH_2)_2 \cdot H_2O_2$, UHP], which is obtained by recrystallization of urea from commercially available 33% aqueous hydrogen peroxide, is a cheap and commercially available white crystalline solid (mp 84–86 °C, dec.) and can be accurately measured.[6] Compared with hydrogen peroxide, it is a much milder oxidant. Hence, we presumed whether the 4-position



Scheme 2. Oxidation of 2,4-diphenylselenyl-1-butenes by hydrogen peroxide.

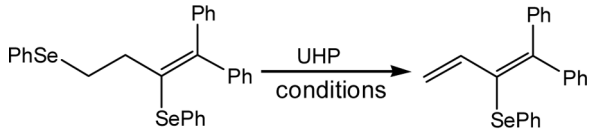


Scheme 3. Selective oxidation of 2,4-diphenylselenyl-1-butenes by UHP.

phenylselenyl group of 2,4-diphenylselenyl-1-butenes could be selectively oxidized with 1 equivalent of UHP followed by the selenoxide elimination with the adjacent hydrogen atom to provide the corresponding 2-phenylselenyl-1,3-butadienes (Scheme 3).

Initially, we stirred 0.1 mmol of 1,1-diphenyl-2,4-diphenylselenyl-1-butene (**1a**) and 0.1 mmol of UHP in acetone at room temperature. After 24 h, the expected product 1,1-diphenyl-2-phenylselenyl-1,3-butadiene (**3a**) was obtained in 43% yield, and 12% of **1a** was recovered (Table 1, entry 1). Further screening demonstrated that the reaction finished completely if 10% excess of UHP was employed, and the yield of **3a**

Table 1. Reactions of 1,1-diphenyl-2,4-diphenylselenyl-1-butene (**2a**) and UHP under different conditions[a]



1a **3a**

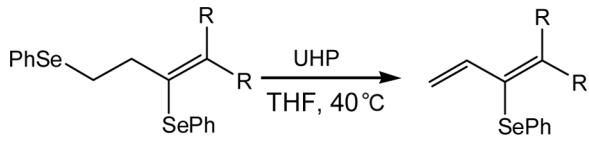
Entry	Solvent	T (°C)	Time (h)[b]	Yield of **3a** (%)[c]
1	Acetone	20	24	43
2	Acetone	20	16	51
3	C_2H_5OH	20	24	14
4	CH_2Cl_2	20	9	56
5	THF	20	5	62
6	THF	40	5	73
7	THF	60	4	51

[a]**1a** (0.1 mmol) and solvent (2 mL) were employed. In entry 1, 0.1 mmol of UHP was employed whereas in entries 2–7, 0.11 mmol of UHP was employed.

[b]The reaction was monitored by TLC (eluent: petroleum ether).

[c]Isolated yields.

Table 2. Synthesis of 2-phenylselenyl-1,3-butadienes[a]



1 **3**

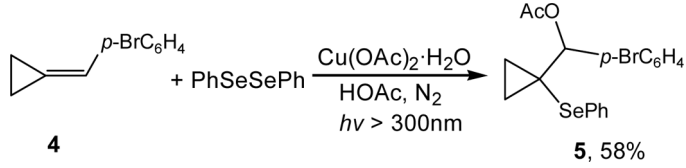
Entry	R, R	Yield of **3** (%)[b]
1	C_6H_5, C_6H_5 (**1a**)	73 (**3a**)
2	*p*-$CH_3C_6H_4$, *p*-$CH_3C_6H_4$ (**1b**)	60 (**3b**)
3	*p*-$CH_3OC_6H_4$, *p*-$CH_3OC_6H_4$ (**1c**)	43 (**3c**)
4	*p*-FC_6H_4, *p*-FC_6H_4 (**1d**)	62 (**3d**)
5	*p*-ClC_6H_4, *p*-ClC_6H_4 (**1e**)	55 (**3e**)
6	$-CH_2CH_2CH(C_6H_5)CH_2CH_2-$ (**1f**)	52 (**3f**)
7	$-(CH_2)_5-$ (**1g**)	34 (**3g**)
8	$-(CH_2)_7-$ (**1h**)	50 (3**h**)

[a]**1** (0.1 mmol), UHP (0.11 mmol), and THF (2 mL) were employed.
[b]Isolated yields.

was enhanced to 51% (Table 1, entry 2). It is obviously that THF was a better solvent (Table 1, entries 1–5). This was probably because of the better solubility of UHP in THF. The yield of **3a** was enhanced when the reaction was carried out at 40 °C (Table 1, entry 6).

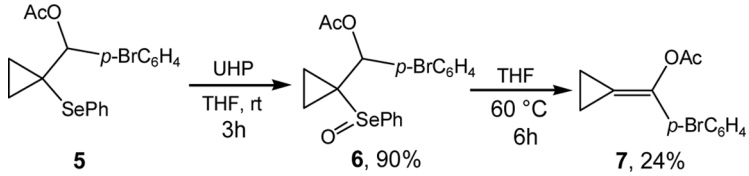
With these results in hand, a series of 2,4-diphenylselenyl-1-butenes were employed, and the corresponding 2-phenylselenyl-1,3-butadienes were synthesized under these conditions (Table 2). In this method, both aryl (Table 2, entries 1–5) and alkyl (Table 2, entries 6–8) substituted MCPs could be transformed to the corresponding 2-phenylselenyl-1,3-butadienes smoothly.

Previously, we disclosed that when mono aryl substituted MCP **4** and diphenyl dieslenide were irradiated under visible light in the presence of copper(II) acetate with acetate acid as solvent, acetic acid (4-bromo-phenyl)-(1-phenylselanyl-cyclopropyl)-methyl ester **5** was obtained[4] (Scheme 4).



4 **5**, 58%

Scheme 4. Synthesis of acetic acid (4-bromo-phenyl)-(1-phenylselenyl-cyclopropyl)-methyl ester **5**.



Scheme 5. Oxidation of **5** by UHP.

In the molecule of **5**, there is a hydrogen atom adjacent to the phenylselenyl group. Hence, we wondered whether **5** could be oxidized by UHP, followed by selenoxide elimination to produce the C=C double-bond-functionalized MCP **7** . The experimental results showed that when **5** was oxidized with UHP at room temperature, **6** could be separated in 90% yield instead of the expected product **7** . When **6** was heated in THF at 60 °C for 6 h, **7** could be obtained in 24% yield while more than 60% of **6** was recovered (Scheme 5). The selenoxide elimination of **6** was not easy.

In conclusion, we disclosed that UHP could oxidize the 4-position phenylselenyl group of 2,4-diphenylselenyl-1-butenes selectively and further selenoxide elimination would give 2-phenyselenyl-1,3-butadienes. Compared with our previous work, this methodology is much more tolerant of the substituted group on the starting material of MCPs, and alkyl substituted 2-phenylselenyl-1,3-butadienes could also be prepared in this method. The synthetic applications of this methodology are under further investigations in our laboratory.

EXPERIMENTAL

^{1}H NMR spectra were recorded on a Bruker Avance 400-MHz spectrometer in $CDCl_3$ with TMS as the internal standard.^{13}C NMR spectra were recorded on a Bruker AC-400 (100-MHz) spectrometer in $CDCl_3$. IR spectra were recorded on a Shimadzu IR-408 spectrometer. EIMS were run on a HP 5989B mass spectrometer.

Typical Procedure for Preparation of 3

A solution of 1,1-diphenyl-2,4-diphenylselenyl-1-butene (**1a**, 0.052 g, 0.1 mmol) and UHP (0.010 g, 0.11 mmol) was stirred in 2 mL of THF at room temperature for 1 h. Then the mixture was stirred at 40 °C for an extra 4 h. When the reaction was completed, the solvent was evaporated under vacuum, and the residue was subjected to preparative TLC

(eluent: petroleum ether) to afford **3a** in 73% yield. The spectrum data were consistent with literature.[4]

Typical Procedure for Preparation of 6

Under a nitrogen atmosphere, UHP (0.030 g, 0.33 mmol) was added to the solution of **5** (0.127g, 0.3 mmol) in THF (2 mL). The mixture was stirred at room temperature for 3 h. Then, the solvent was evaporated under vacuum, and the residue was separated by preparative TLC (eluent: petroleum ether–EtOAc 1:1) to give **6** in 90% yield.

Typical procedure for Preparation of 7

Under a nitrogen atmosphere, **6** (0.044 g, 0.1 mmol) was heated in THF (2 mL) at 60 °C. After 6 h, the solvent was evaporated under vacuum, and the residue was separated by TLC (eluent: petroleum ether–EtOAc 8:1), and **7** was obtained in 24% yield. Compound **6** could be recovered in 60% yield.

Data

Compound **3a**: Oil. ^{1}H NMR (400 Hz, $CDCl_3$): δ 7.14–7.34 (m, 15H), 6.56–6.63 (m, 1H), 5.81–5.85 (m, 1H), 5.22–5.25 (m, 1H). ^{13}C NMR (100 Hz, $CDCl_3$): δ 120.9, 125.8, 127.3, 127.7, 128.1, 128.9, 129.4, 130.0, 130.1, 130.2, 133.0, 135.4, 141.6, 144.1, 151.6. IR (film, cm^{-1}): 3054, 1576, 1477, 1440, 737, 697. MS (70 eV, EI) m/z: 362 (M^+, 30), 281 (15), 205 (100). HRMS (EI) calcd. for $C_{22}H_{18}Se$ (M^+): 362.0574; found: 362.0567.

Compound **3b**: Oil. ^{1}H NMR (400 Hz, $CDCl_3$, δ, ppm): 7.08–7.35 (m, 13H), 6.59–6.66 (m, 1H), 5.77–5.81 (m, 1H), 5.19–5.22 (m, 1H), 2.39 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (100 Hz, $CDCl_3$, δ, ppm): 21.2 (d), 120.3, 125.6, 128.3, 128.6, 128.7, 128.8, 129.3, 129.9, 130.1, 133.2, 135.5, 137.1, 137.5, 138.8, 141.4, 151.9; IR (film, ν, cm^{-1}): 2921, 1507, 1022, 735; MS (70 eV, EI) m/z: 390 (M^+, 47), 233 (77), 218 (100).

Compound **3c**: Oil. ^{1}H NMR (400 Hz, $CDCl_3$, δ, ppm): 6.75–7.32 (m, 13H), 6.58–6.62 (m, 1H), 5.75–5.79 (m, 1H), 5.18–5.21 (m, 1H), 3.83 (s, 3H), 3.78 (s, 3H); ^{13}C NMR (100 Hz, $CDCl_3$, δ, ppm): 55.1, 55.2, 112.9, 113.3, 119.9, 125.6, 128.1, 128.8, 129.9, 131.0, 131.8, 133.3, 134.2, 135.8, 136.9, 151.2, 158.9, 159.2; IR (film, ν, cm^{-1}): 2924, 1603, 1507, 1248, 1029; MS (70 eV, EI) m/z: 422 (M^+, 80), 341 (34), 265 (100).

Compound **3d**: Oil. ^{1}H NMR (400 Hz, $CDCl_3$, δ, ppm): 6.87–7.26 (m, 13H), 6.49–6.52 (m, 1H), 5.83–5.88 (m, 1H), 5.26–5.29 (m, 1H); ^{13}C NMR (100 Hz, $CDCl_3$, δ, ppm): 114.6, 114.8, 115.0, 115.3, 121.4, 126.1, 128.9, 129.0, 130.5, 131.2, 131.3, 131.9, 132.0, 132.6, 135.4, 148.6; IR (film, ν, cm^{-1}): 2924, 1504, 1227, 835; MS (70 eV, EI) m/z: 398 (M^+, 48), 241 (76), 220 (100).

Compound **3e**: Oil. ^{1}H NMR (400 Hz, $CDCl_3$, δ, ppm): 7.03–7.60 (m, 13H), 6.47–6.50 (m, 1H), 5.85–5.89 (m, 1H), 5.28–5.31 (m, 1H); ^{13}C NMR (100 Hz, $CDCl_3$, δ, ppm): 121.9, 126.2, 128.0, 128.4, 128.8, 129.0, 129.2, 129.3, 129.6, 130.1, 130.6, 130.9, 131.5, 132.4, 135.3, 139.6; IR (film, ν, cm^{-1}): 2921, 1738, 1244, 735; MS (70 eV, EI) m/z: 430 (M^+, 37), 238 (100).

Compound **3f**: Oil. ^{1}H NMR (400 Hz, $CDCl_3$, δ, ppm): 7.15–7.34 (m, 10H), 6.75–6.82 (m, 1H), 5.68–5.72 (m, 1H), 5.18–5.21 (m, 1H), 3.58–3.62 (m, 1H), 3.24–3.29 (m, 1H), 2.77–2.83 (m, 1H), 2.19–2.27 (m, 2H), 1.98–2.18 (m, 2H), 1.49–1.63 (m, 2H); ^{13}C NMR (100 Hz, $CDCl_3$, δ, ppm): 31.5, 35.3, 35.4, 37.1, 44.5, 119.3, 123.3, 125.6, 126.1, 126.8, 128.4, 128.9, 129.6, 132.9, 133.4, 146.1, 151.2; IR (film, ν, cm^{-1}): 2924, 2853, 1606, 1474, 735, 696; MS (70 eV, EI) m/z: 354 (M^+, 77), 197 (20), 91 (100).

Compound **3g**: Oil. ^{1}H NMR (400 Hz, $CDCl_3$, δ, ppm): 7.12–7.29 (m, 5H), 6.75–6.82 (m, 1H), 5.62–5.66 (m, 1H), 5.12–5.15 (m, 1H), 2.71–2.74 (m, 2H), 2.55–2.58 (m, 2H), 1.55–1.62 (m, 4H), 0.86–0.88 (m, 2H); ^{13}C NMR (100 Hz, $CDCl_3$, δ, ppm): 26.7, 28.4, 29.7, 31.9, 37.5, 118.7, 122.4, 125.4, 128.9, 129.5, 133.0, 133.4, 152.9; IR (film, ν, cm^{-1}): 2925, 2853, 1608, 1473, 1095, 733; MS (70 eV, EI) m/z: 278 (M^+, 5), 157 (33), 55 (100).

Compound **3h** : Oil. ^{1}H NMR (400 Hz, $CDCl_3$, δ, ppm): 7.14–7.30 (m, 5H), 6.78–6.82 (m, 1H), 5.64–5.68 (m, 1H), 5.12–5.15 (m, 1H), 2.74–2.77 (m, 2H), 2.61–2.64 (m, 2H), 1.72–1.84 (m, 4H), 1.46–1.56 (m, 6H); ^{13}C NMR (100 Hz, $CDCl_3$, δ, ppm): 25.9, 26.3, 26.7, 27.5, 27.7, 32.0, 37.9, 118.0, 124.6, 125.4, 128.8, 129.5, 133.0, 133.7, 155.1; IR (film, ν, cm^{-1}): 2923, 2853, 1474, 1096, 734; MS (70 eV, EI) m/z: 306 (M^+, 100), 155 (20).

Compound **6** : Oil. ^{1}H NMR (400 Hz, $CDCl_3$): δ 6.99–7.26 (m, 9H), 5.96 (s, 1H), 2.04 (s, 3H), 1.20–1.24 (m, 2H), 0.90–0.94 (m, 2H). ^{13}C NMR (100 Hz, $CDCl_3$): δ 169.7, 139.7, 136.0, 134.9, 131.7, 129.4, 128.2, 126.6, 122.6, 74.8, 20.7, 6.3, 5.6. IR (film, cm^{-1}): 1720, 1242, 1003, 740. MS (70 eV, EI) m/z: 441 ($M^+ + 1$, 21), 440 (M^+, 4), 145 (100). HRMS (EI) calcd. for $C_{18}H_{17}BrO_3Se$ (M^+): 439.9526; found: 439.9520.

Compound **7** : Oil. ^{1}H NMR (400 Hz, $CDCl_3$): δ 7.44–7.46 (m, 4H), 2.31 (s, 3H), 1.65 (t, $J = 7.2$ Hz, 2H), 1.29 (t, $J = 7.2$ Hz, 2H). ^{13}C NMR (100 Hz, $CDCl_3$): δ 168.9, 135.9, 133.6, 131.4, 125.9, 122.0, 112.0, 20.8,

5.5, 2.6. IR (film, cm^{-1}): 3743, 1747, 1210, 1137, 1008, 827. MS (70 eV, EI) m/z: 266 (M^+, 9), 145 (100). HRMS (EI) calcd. for $C_{12}H_{11}BrO_2$ (M^+): 265.9942; found: 265.9954.

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