[3,3]Sigmatropic Ring Expansion of Cyclic Thionocarbonates. VII.[1] On the Formation of 8-Membered Thionocarbonates as the Intermediates

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In order to clarify the reaction mechanism of [3,3]sigmatropic ring expansion of cyclic thionocarbonates, the 8-membered thionocarbonates (6 and 7) were synthesized by treatment of the corresponding diol monothionocarbonates (5 and 13) with lithium bis(trimethylsilyl)amide. However, reaction of 7 with *meta*-chloroperbenzoic acid afforded an 8-membered carbonate (14) in a quantitative yield.

Keywords [3,3]sigmatropic ring expansion; 8-membered thionocarbonate; diol monothionocarbonate; lithium bis(trimethylsilyl)amide; *meta*-chloroperbenzoic acid; 8-membered carbonate

The exclusive (*E*)-selectivity of the double bond created by [3,3]sigmatropic rearrangement has been extensively used in organic synthesis.[2] However, few studies on the opposite (*Z*)-selective [3,3]sigmatropic rearrangement have so far been reported.[3] We have recently reported that treatment of a diol monothionocarbonate (**1a**) with base in tetrahydrofuran (THF) instantly gives a 10-membered thiolcarbonate (**3a**) containing a (*Z*)-double bond *via* [3,3]sigmatropic rearrangement of the 8-membered thionocarbonate (**2a**) formed *in situ* (Chart 1).[4a,d] In order to demonstrate the synthetic utility of the present method, the sexual pheromone of yellow scale insect, (±)-(*E*)-6-isopropyl-3,9-dimethyl-5,8-decadienyl acetate, was stereoselectively synthesized.[4e] On the other hand, we recently reported that the highly stereoselective synthesis of a (*Z*)- or (*E*)-double bond in 10-membered thiolcarbonates (**3**) could be achieved by controlling the chair–boat-like transition state in the [3,3]sigmatropic rearrangement of 8-membered thionocarbonates (**2**) (Chart 2).[4b] Reaction of (*E*)-**1b** having an (*E*)-double bond gave a (*Z*)-10-membered thiolcarbonate (**3b**) in 88% yield and the same treatment of (*Z*)-**1c** having a (*Z*)-double bond correspondingly provided (*E*)-**3c** in 78% yield. The geometry of (*Z*)-**3b** can be explained by the chairlike transition state (T_C), whereas that of (*E*)-**3c** is due to the boatlike transition state (T_B).[4b] Further, the proposed transition states (T_C and T_B) were quantitatively evaluated by a combination of molecular mechanics and orbital computations.[4c] The 8-membered ring of the calculated T_C and T_B adopted a boat–chair conformation existing predominantly in cyclooctane and its simple derivatives. However, since the reactions go to completion instantly,

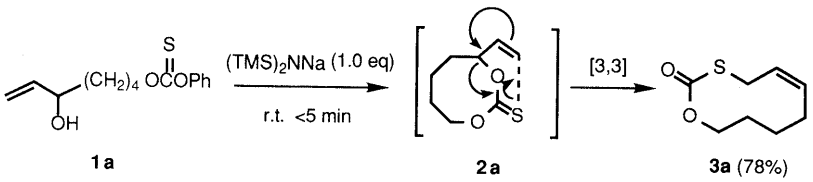


Chart 1

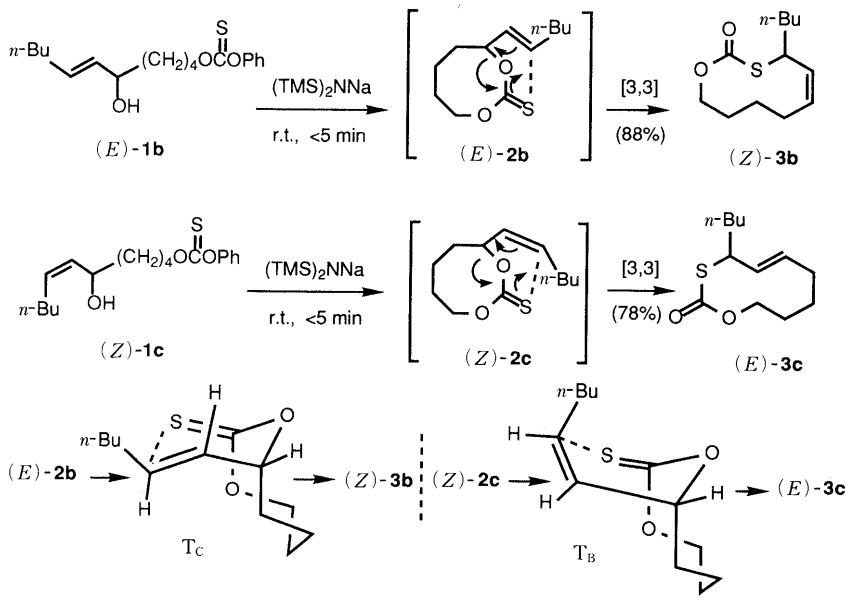
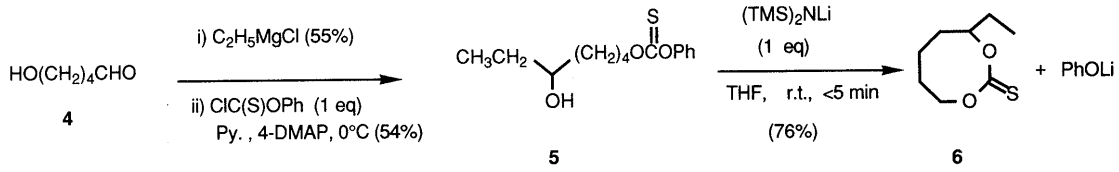


Chart 2

the 8-membered thionocarbonates intermediates (**2**) have not been isolated. Thus, we have studied the formation of 8-membered thionocarbonates (**2**) to clarify the above reaction mechanism.

First, we examined whether a diol monothionocarbonate (**5**), which contains an ethyl group instead of the ethenyl group of **1a**, can form an 8-membered thiolcarbonates (**6**) on treatment with base. Compound **5** was easily synthesized from 5-hydroxypentanal (**4**) (Chart 3).[5] Reaction of **4** with ethylmagnesium chloride followed by treatment with phenyl chlorothionoformate (1 eq) in the presence of pyridine (1 eq) and 4-dimethylaminopyridine (4-DMAP) (0.1 eq) in acetonitrile gave the monothionocarbonate (**5**). Although sodium bis(trimethylsilyl)amide [$(TMS)_2NNa$] has been used as a base for the present reaction,[4d] we have newly found that its lithium salt [$(TMS)_2NLi$] is more effective through our study of the yellow scale pheromone synthesis.[4e] A hexane solution of $(TMS)_2NLi$ (1 eq) was injected rapidly into a dry THF solution of **5** at room temperature under N_2. The reaction went to completion immediately and the desired 8-membered heterocyclic thionocarbonate (**6**) could be isolated by chromatography even in the presence of the counter ion (PhO^-) generated *in situ*. The structure of **6** was indicated to be 4-ethyl-1,3-dioxocan-2-thione by the proton nuclear magnetic resonance (^{1}H-NMR) and carbon 13 nuclear magnetic resonance (^{13}C-NMR) spectra. Each proton at C_8 was separately observed at δ4.21 (1H, dt, J=11.6, 5.0 Hz) and δ4.80 (1H, dt, J=11.6, 5.0 Hz), suggesting that this 8-membered ring is conformationally fixed at room temperature. Although Copeland *et al.*[6] reported that the chemical shift of carbon [OC(S)O] in a variety of thionocarbonates was centered at about 193 ppm, it was observed that the carbon resonance [OC(S)O: 200.5 ppm] of **6** showed a downfield shift by about 7 ppm.

We next considered that oxidation of the 8-membered thionocarbonate (**7**) having a phenylselenoethyl group might lead to **3a** *via* the spontaneous [3,3]sigmatropic ring expansion of the intermediate (**2a**) which would be formed by an elimination reaction[7] of the resulting selenoxide (**8**) (Chart 4). Thus, the selenide (**7**) was prepared from acrolein *via* the sequence outlined in Chart 5. Conjugate addition of sodium benzeneselenoate to acrolein according to Yoshikoshi's procedure[8] gave the aldehyde (**9**). Reaction of **9** with the lithium salt (**11**)[9] derived from 1,4-butanediol gave an alcohol (**12**) in 66% yield. Deprotection



Py.=pyridine

Chart 3

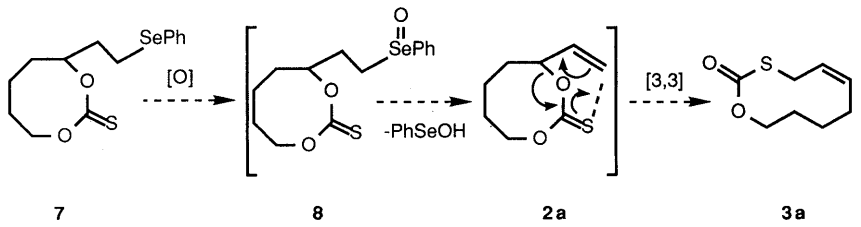


Chart 4

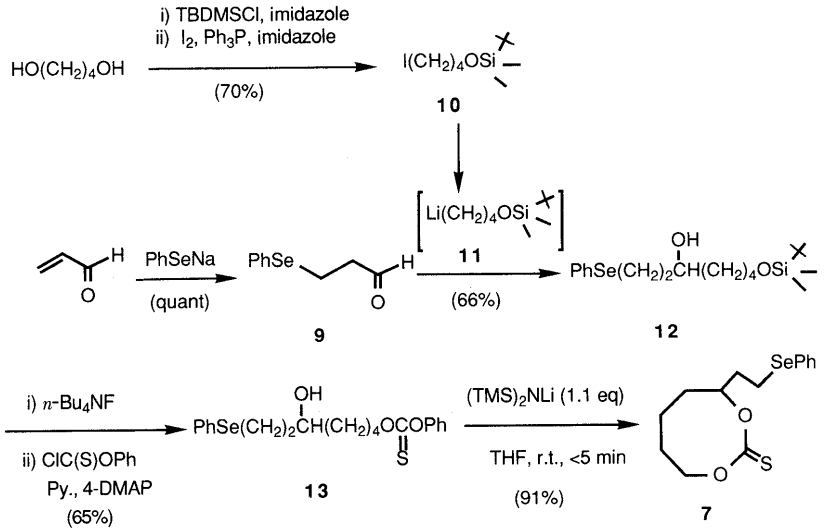


Chart 5

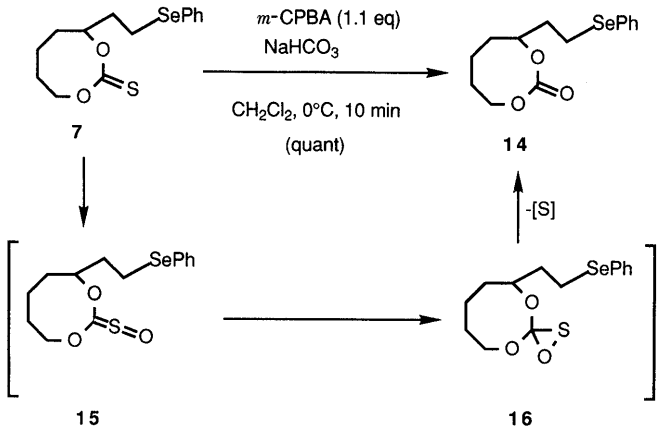


Chart 6

of **12** with tetrabutylammonium fluoride followed by treatment with phenyl chlorothionoformate gave a diol monothionocarbonate (**13**) in 65% yield. Reaction of **13** with $(TMS)_2NLi$ by the procedure described above smoothly afforded the 8-membered thionocarbonate (**7**) in 91% yield. The C_8 proton signals [$-CH_aH_bO-$: δ 4.23 (dt, $J=11.2$, 5.3 Hz), 4.78 (dt, $J=11.2$, 5.3 Hz)] and the thiocarbonyl carbon signal (δ 200.0) of **7** were similar to those of **6**. The oxidation of **7** thus obtained with *meta*-chloroperbenzoic acid (*m*-CPBA) at 0 °C in dichloromethane (CH_2Cl_2) was completed immediately to give unexpectedly a cyclic carbonate (**14**) containing an O atom instead of the S atom of **7**. Although the ^{1}H-NMR spectrum of **14** closely resembled that of **7**, the infrared (IR) (1750 cm^{-1}, C=O) and mass spectra (MS) (M^+ 314) indicated the structure of **14**. Yamamoto *et al.*[10] reported that thionolactones are easily oxidized to the corresponding lactones in high yield with *m*-CPBA in CH_2Cl_2 at room temperature. Metzner *et al.*[11] also showed that reaction of thioketones[11a] or thionoesters[11b] with *m*-CPBA easily affords the sulfines,[12] which are converted into the corresponding ketones or esters with loss of sulfur. By analogy with these recent reports, our observation can be explained by the formation of an oxathiirane (**16**) arising from thermally allowed electrocyclization of the sulfine (**15**) followed by sulfur extrusion.

In conclusion, although the formation of **3a** *via* [3,3]-sigmatropic ring expansion from the selenoxide (**8**) as outlined in Chart 4 has not been accomplished, easy production of the 8-membered thionocarbonates (**6** and **7**) supports the hypothesis that the reaction of diol monothionocarbonates (**1**) having a double bond with base proceeds *via* the [3,3]sigmatropic ring expansion of the postulated 8-membered thionocarbonates (**2**) to give the 10-membered thiolcarbonates (**3**).

Experimental

IR spectra were recorded on a Shimadzu IR-435 spectrophotometer. ^{1}H- and ^{13}C-NMR spectra were taken with tetramethylsilane as an internal standard on a Varian Germini-200 spectrometer in $CDCl_3$. Low-resolution and high-resolution mass spectra (HRMS) were obtained on a Hitachi M-80 instrument. All reactions were carried out under a nitrogen atmosphere. For column chromatography, SiO_2 (Merck 9385) was used.

***o*-(5-Hydroxyheptanyl) *o*-Phenyl Thionocarbonate (5)** A 2 M solution of ethylmagnesium chloride (60 mM) in THF (20 ml) was stirred efficiently at 0 °C while a solution of 5-hydroxypentanal (**4**)[5] (2.04 g, 20 mM) in THF (20 ml) was added dropwise over a 40-min period. The mixture was stirred at room temperature for 1 h, then again cooled in an ice bath, and cautiously hydrolyzed with saturated aqueous NH_4Cl solution. When the hydrolysis was complete, the solvent was evaporated off to give a white residue. The residue was diluted with saturated NH_4Cl solution and EtOAc–hexane (1 : 1). The organic layer was washed with brine and dried over anhydrous Na_2SO_4. Evaporation of the solvent left 1,5-heptanediol (1.45 g, 55%) [^{1}H-NMR δ: 0.90 (3H, t, $J=7.5$ Hz, CH_3), 1.30—1.65 (8H, br, $CH_2\times4$), 3.44—3.67 (3H, br, CHOH, CH_2OH)] as a pale yellow oil. This oil was subjected to the following reaction without further purification. A solution of phenyl chlorothionoformate[13] (1.8 ml, 12.72 mM) in acetonitrile (10 ml) was added dropwise to a solution of 1,5-heptanediol (1.4 g, 10.6 mM) in acetonitrile (20 ml) in the presence of pyridine (1.0 g, 12.72 mM) and 4-DMAP (129 mg, 1.06 mM) over 2.5 h at 0 °C. After the reaction temperature had risen to room temperature, the solvent was evaporated off under reduced pressure. The oily residue was dissolved in EtOAc–hexane (1 : 1). The organic layer was washed with H_2O and brine, dried over anhydrous Na_2SO_4, and then evaporated off under reduced pressure. The residue was purified by column chromatography (10% EtOAc in hexane) to give **5** (1.52 g, 54%). IR (film) cm^{-1}: 3380—3390 (OH). ^{1}H-NMR δ: 0.93 (3H, t, $J=7.5$ Hz, CH_3), 1.35—1.68 (6H, br, $CH_2\times3$), 1.75—1.92 (2H, br, CH_2CH_3), 3.45—3.60 (1H, br, CHOH), 4.51 (2H, t, $J=6.5$ Hz, OCH_2), 7.05—7.46 (5H, m, ArH). This compound did not give the expected MS peaks because of thermal instability.

4-Ethyl-1,3-dioxocan-2-thione (6) A 1 M solution of $(TMS)_2NLi$[13] in THF (0.5 ml, 0.5 mM) was added rapidly to a solution of **5** (134 mg, 0.5 mM) in THF (50 ml) at room temperature, and the mixture was stirred for 5 min. The reaction was quenched by the addition of H_2O and the solvent was evaporated off under reduced pressure. The residue was extracted with EtOAc–hexane (1 : 1) and the extract was washed with brine, dried over anhydrous Na_2SO_4, and then evaporated under reduced pressure. The residual oil was purified by column chromatography (4% EtOAc in hexane) to give **6** (66 mg, 76%) as an oil. IR (film) cm^{-1}: 2940, 1440, 1350, 1180, 1070, 1030, 930, 910. ^{1}H-NMR δ: 0.98 (3H, t, $J=8.0$ Hz, CH_3), 1.50—2.0 (8H, br, $CH_2\times4$), 4.21 [1H, dt, $J=11.6$, 5.0 Hz, $CH_aH_bOC(S)$], 4.80 [1H, dt, $J=11.6$, 5.0 Hz, $CH_aH_bOC(S)$], 4.72—4.92 (1H, br, CHO). ^{13}C-NMR δ: 10.5, 23.8, 27.5, 31.5, 35.5, 75.0, 90.4, 200.5. MS *m/z*: 174 (M^+). HRMS Calcd for $C_8H_{14}O_2S$: 174.0714. Found: 174.0716.

3-Phenylselenopropanal (9) This compound was quantitatively prepared from acrolein on a 2 mM scale according to Yoshikoshi's procedure.[8] IR (film) cm^{-1}: 1715 (CO). ^{1}H-NMR δ: 2.83 (2H, t, $J=7.5$ Hz, CH_2Se), 3.08 (2H, t, $J=7.5$ Hz, CH_2CHO), 7.20—7.65 (5H, br, ArH), 9.75 (1H, s, CHO). MS *m/z*: 214 (M^+). HRMS Calcd for $C_9H_{10}OSe$: 213.9896. Found: 213.9894.

4-Iodobutyl *tert*-Butyldimethylsilyl Ether (10) A solution of *tert*-butyldimethylsilyl chloride (TBDMSCl) (6.8 g, 45 mM) in dimethylformamide (DMF) (20 ml) was added dropwise to a mixture of 1,4-butanediol (4.04 g, 45 mM) and imidazole (7.65 g, 112 mM) in DMF (20 ml) over 4 h at 0 °C. After additional stirring for 40 min at 0 °C, the mixture was dissolved in EtOAc–hexane (3 : 1) (100 ml). The organic layer was washed with H_2O (×3) and brine, dried over anhydrous Na_2SO_4, and then evaporated under reduced pressure. The oily residue was purified by column chromatography (10% EtOAc in hexane) to give 4-*tert*-butyldimethylsilyloxybutanol (6.83 g, 74%) as an oil. IR (film) cm^{-1}: 3700—3050 (OH), 1280—1200, 1140—920. ^{1}H-NMR δ: 0.05 [6H, s, $(CH_3)_2Si$], 0.87 [9H, s, $(CH_3)_3CSi$], 1.57—1.67 (4H, m, $CH_2\times4$), 2.63 (1H, br, OH), 3.65 (4H, br t, $J=5.5$ Hz, CH_2OH, CH_2OSi). Imidazole (544 mg, 8 mM), triphenylphosphine (1.42 g, 8 mM) and iodine (1.63 g, 6.4 mM) were added in turn to a solution of 4-*tert*-butyldimethylsilyloxybutanol (653 mg, 3.2 mM) in benzene (30 ml). After being stirred at room temperature for 0.5 h, the reaction mixture was washed with saturated sodium sulfite solution and brine and then dried over anhydrous $MgSO_4$. The solvent was evaporated off *in vacuo* to give an oily residue, which was purified by column chromatography (5% EtOAc in hexane) to give **10** (940 mg, 94%) as a colorless liquid. IR (film) cm^{-1}: 1280—1190, 1140—1040. ^{1}H-NMR δ: 0.02 [6H, s, $(CH_3)_2Si$], 0.87 [9H, s, $(CH_3)_3CSi$], 1.58 (2H, dt, $J=14.3$, 6.0 Hz, CH_2), 1.88 (2H, dt, $J=14.3$, 7.0 Hz, CH_2), 3.20 (2H, t, $J=7.0$ Hz, CH_2I), 3.60 (2H, t, $J=6.0$ Hz, CH_2O). MS *m/z*: 313 (M^+-1), 299 (M^+-CH_3). HRMS Calcd for $C_9H_{20}IOSi$ (M^+-CH_3): 299.0328. Found: 299.0327.

7-*tert*-Butyldimethylsilyloxy-1-phenylselenoheptan-3-ol (12) The dry flask was charged with **10** (597 mg, 1.9 mM) and dry *n*-pentane–diethyl ether (3 : 2 by volume, 19 ml).[9] The solution was cooled to −78 °C and

1.7 M *tert*-BuLi in *n*-pentane (2.3 ml) was then added. Stirring was continued at −78 °C for 5 min, then at room temperature for 1 h. The mixture was again cooled to −78 °C, the aldehyde (**9**) was added and the resulting mixture was stirred for 10 min. The reaction was quenched by the addition of H_2O, and then the mixture was dissolved in EtOAc–hexane (1 : 1). The organic layer was washed with H_2O (×2) and brine, dried over anhydrous Na_2SO_4, and then evaporated under reduced pressure. The residue was purified by column chromatography (5% EtOAc in hexane, then 10% EtOAc in hexane) to give **12** (764 mg, 66%) as an oil. IR (film) cm^{-1}: 3620—3100 (OH), 1250, 1150—920. ^{1}H-NMR δ: 0.02 [6H, s, $(CH_3)_2Si$], 0.87 [(9H, s, $(CH_3)_2CSi$], 1.30—1.55 (4H, br m, $CH_2 \times 2$), 1.59—1.67 (2H, br, CH_2), 1.72—1.88 (2H, m, CH_2), 3.0 (2H, m, CH_2Se), 3.57 (2H, t, J=6.0 Hz, CH_2O), 3.64—3.79 (1H, br, CHOH), 7.18—7.31 (3H, m, ArH), 7.42—7.53 (2H, m, ArH). MS *m/z*: 402 (M^+). HRMS Calcd for $C_{19}H_{34}O_2SeSi$: 402.1491. Found: 402.1494.

***o*-(5-Hydroxy-7-phenylselenoheptanyl) *o*-Phenyl Thionocarbonate (13)** A 1 M solution of tetrabutylammonium fluoride[13] (4.1 ml, 4.1 mM) in THF was added to a solution of **12** (1.37 g, 3.43 mM) in THF (50 ml) and the mixture was stirred for 4 h. The reaction was quenched by the addition of H_2O and the solvent was evaporated off under reduced pressure. The residue was dissolved in EtOAc–hexane (1 : 1) and the organic layer was washed with H_2O, brine, dried over anhydrous Na_2SO_4, and then evaporated under reduced pressure. The residue was purified by column chromatography (80% EtOAc in hexane) to give 7-phenylseleno-1,5-heptanediol (876 mg, 88%). IR (film) cm^{-1}: 3700—3000 (OH), 1220—940. ^{1}H-NMR δ: 1.28 (8H, m, $CH_2 \times 4$), 1.72—1.88 (2H, m, CH_2), 3.0 (2H, m, CH_2Se), 3.61 (2H, br t, J=6.0 Hz, CH_2OH), 3.72 (1H, br, CHOH), 7.18—7.31 (3H, m, ArH), 7.41—7.55 (2H, m, ArH). MS *m/z*: 288 (M^+). HRMS Calcd for $C_{13}H_{20}O_2Se$: 288.0627. Found: 288.0625. A solution of phenyl chlorothionoformate (48 mg, 0.28 mM) in acetonitrile (5 ml) was added dropwise to a solution of 7-phenylseleno-1,5-pentanediol (79 mg, 0.28 mM) in acetonitrile (6 ml) in the presence of pyridine (22 mg, 0.28 mM) and 4-DMAP (3.4 mg, 0.028 mM) over 6 h at 0 °C. The mixture was evaporated under reduced pressure and the oily residue was dissolved in EtOAc–hexane (1 : 1). The organic layer was washed with H_2O and brine, dried over anhydrous Na_2SO_4, and then evaporated under reduced pressure. The residue was purified by column chromatography (13% EtOAc in hexane) to give **13** (87 mg, 74%) as an oil. IR (film) cm^{-1}: 3400, 2920, 1725, 1585. ^{1}H-NMR δ: 1.35—1.63 (4H, m, $CH_2 \times 2$), 1.68—1.90 (4H, m, $CH_2 \times 2$), 2.88—3.09 (2H, m, CH_2Se), 3.63—3.83 (1H, br, CHOH), 4.50 (2H, t, J=6.5 Hz, CH_2O), 6.90—7.53 (10H, m, ArH). MS *m/z*: 424 (M^+). HRMS Calcd for $C_{20}H_{24}O_3SSe$: 424.0610. Found: 424.0617.

4-(2-Phenylselenoethyl)-1,3-dioxocan-2-thione (7) A 1 M solution of $(TMS)_2NLi$ in THF (0.44 ml, 0.44 mM) was added rapidly to a solution of **13** (188 mg, 0.44 mM) in THF (44 ml). The reaction mixture was stirred and $(TMS)_2NLi$ (0.05 ml, 0.05 mM) in THF was further added after 5 min. The reaction was quenched by the addition of H_2O and the solvent was evaporated off under reduced pressure. The residue was extracted with EtOAc–hexane (1 : 1) and the extract was washed with H_2O and brine and dried over anhydrous Na_2SO_4, and then evaporated under reduced pressure. The residual oil was purified by column chromatography (6% EtOAc in hexane) to give **7** (132 mg, 91%) as an oil. IR (film) cm^{-1}: 2910, 1575. ^{1}H-NMR δ: 1.50—2.33 (8H, br, $CH_2 \times 4$), 2.87—3.14 (2H, m, CH_2Se), 4.23 (1H, dt, J=11.2, 5.3 Hz, OCH_aH_b), 4.78 (1H, dt, J=11.2, 5.3 Hz, OCH_aH_b), 4.97—5.12 (1H, br m, CH–O), 7.15—7.33 (3H, m, ArH), 7.41—7.57 (2H, m, ArH). ^{13}C-NMR δ: 23.5, 31.1, 35.2, 35.8, 74.0, 88.0, 121.0, 127.6, 128.0, 130.1, 133.2, 200.0. MS *m/z*: 330 (M^+). HRMS Calcd for $C_{14}H_{18}O_2SSe$: 330.0192. Found: 330.0195.

4-(2-Phenylselenoethyl)-1,3-dioxocan-2-one (14) *m*-CPBA (80%, 7.6 mg, 0.33 mM) was added to a suspension of **7** (10.5 mg, 0.03 mM) and $NaHCO_3$ (3 mg, 0.33 mM) in CH_2Cl_2 (3 ml) at 0 °C. After being stirred for 1 h, the mixture was washed with H_2O, saturated aqueous $NaHCO_3$ solution and saturated aqueous Na_2SO_3 solution, and dried over anhydrous $MgSO_4$. Evaporation of the solvent left a crude oil, which was purified by column chromatography (10% EtOAc in hexane) to give **14** (9.3 mg, quant.) as an oil. IR (film) cm^{-1}: 1750 (CO). ^{1}H-NMR δ: 1.44—2.23 (8H, br m, $CH_2 \times 4$), 2.84—3.13 (2H, m, CH_2Se), 4.04 (1H, dt, J=11.0, 5.5 Hz, OCH_aH_b), 4.49 (1H, dt, J=11.0, 5.5 Hz, OCH_aH_b), 4.63—4.80 (1H, br m, CH–O), 7.19—7.32 (3H, m, ArH), 7.42—7.53 (2H, m, ArH). MS *m/z*: 314 (M^+). HRMS Calcd for $C_{14}H_{18}O_3Se$: 314.0420. Found: 314.0427.

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References and Notes

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