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Hydrolysis of an Isolable Selenoseleninate under Acidic and Alkaline Conditions

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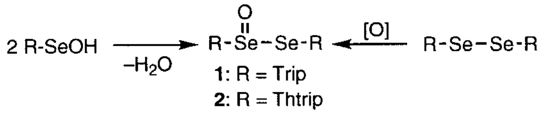
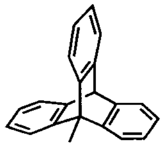
ABSTRACT: *Acid-catalyzed hydrolysis of a selenoseleninate [TripSe(O)SeTrip](Trip = 9-triptycyl) in 2 M $HClO_4$-1,4-dioxane at room temperature yielded the selenenic acid (TripSeOH). Alkaline hydrolysis of TripSe(O)SeTrip gave the diselenide (TripSeSeTrip) and the seleninic acid ($TripSeO_2H$), where air oxidations of the resulting selenol (TripSeH) and TripSeOH occur at a considerable rate.* © 2001 John Wiley & Sons, Inc. Heteroatom Chem 12:198–203, 2001

INTRODUCTION

Generally *Se*-substituted selenoseleninates [RSe(O)-SeR′] are unstable intermediates in condensation of alkaneselenenic acids [1,2] and in oxidation of diselenides [1–5]. Recently we obtained isolable, stable selenoseleninates **1** and **2** by condensation of the corresponding selenenic acids or by oxidation of the diselenides [6]. The selenoseleninate structure contrasts with the selenenic anhydride structure (ArSeOSeAr) of condensation products of areneselenenic acids [7–11].

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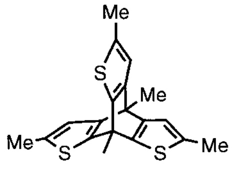
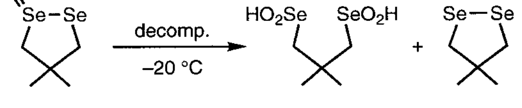


Thtrip-

Although a few selenoseleninates were detected by low-temperature NMR spectroscopy, they decomposed to seleninic acids and diselenides (Equation 1) [1–3,9]. On the other hand, comproportionation of diaryl diselenides and areneseleninic acids has been claimed to generate the areneselenenic acid (Equation 2) [5,12], where the corresponding areneselenenic anhydrides were proposed as the intermediate [13,14]. The low stability of selenoseleninates is in contrast with fairly high stability of the sulfur analogues, thiosulfinates [RS(O)SR′] [15,16]. Therefore it is worthy to clarify the chemical behaviors of selenoseleninates in relation with the chemistry of selenenic acids as well as in comparison with the chemistry of thiosulfinates [15–17]. Here we report hydrolysis of **1** both under acidic and alkaline conditions. (Throughout this article, the 9-triptycyl group is abbreviated to Trip for convenience.)



(1)

$$ArSeSeAr + ArSeO_2H + H_2O \rightleftharpoons 3\ ArSeOH \quad (2)$$

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Dedicated to Professor Naoki Inamoto on the occasion of his 72nd birthday.

RESULTS AND DISCUSSION

Under Acidic Conditions

Selenoseleninate **1** was stirred in 2 M $HClO_4$-1,4-dioxane at room temperature for 5.5 hours. The principal product was the selenenic acid **3** (74%) [6], with an 18% yield of the recovered **1** (Equation 3). The recovery of **1** would mainly result from its low solubility in the medium. A similar result was reported on areneselenenic anhydrides (ArSeOSeAr) being hydrolyzed under acidic conditions to give the areneselenenic acid (ArSeOH) [8,10].

$$\mathbf{1} \xrightarrow[\text{r.t., 5.5 h}]{\text{2M HClO}_4\text{-1,4-Dioxane}} \underset{\substack{\mathbf{3}\\ 74\%}}{\text{Trip-SeOH}} + \underset{18\%}{\mathbf{1}} \tag{3}$$

In the acidic hydrolysis, water attacks the selenenyl selenium of **1**, with selenenic acid **3** behaving as a good leaving group (Scheme 1).

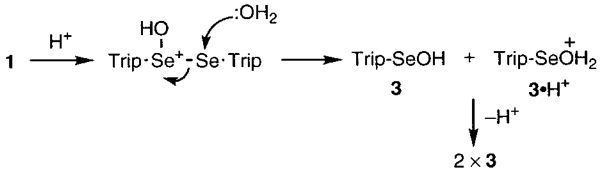
Under Alkaline Conditions

The aspect of hydrolysis of **1** under alkaline conditions was more complicated than that under acidic conditions. When a solution of selenoseleninate **1** (1 mol equiv.) in 2 M KOH-1,4-dioxane was stirred at room temperature for 1.5 hours, the diselenide **4** (0.38 mol equiv.) and the seleninic acid **5** (0.86 mol equiv.) were obtained (Equation 4).

$$\underset{(1\text{ mol eq})}{\mathbf{1}} \xrightarrow[\text{r.t., 1.5 h}]{\text{2M KOH-1,4-Dioxane}} \underset{\mathbf{4}\ (0.38\text{ mol eq})}{\text{Trip-SeSe-Trip}} + \underset{\mathbf{5}\ (0.86\text{ mol eq})}{\text{Trip-SeO}_2\text{H}} \tag{4}$$

Some control experiments were carried out to investigate the mechanism, in particular, for the formation of diselenide **4**. Supposed mechanisms are (i) air-oxidation of the selenol, Trip-SeH **6**; (ii) condensation between **6** and the selenenic acid **3**; and (iii) the reaction of **1** with the selenol **6**.

At the outset, we examined air oxidation of selenol **6** under alkaline conditions (mechanism [i]). When the selenol **6** (1 mol equiv.) was dissolved in 2 M KOH-1,4-dioxane, the color of the mixture changed soon from colorless to orange, indicating the formation of diselenide **4**. The mixture was stirred for 1.5 hours at room temperature to give the diselenide **4** (0.28 mol equiv., 56%), the selenenic acid **3** (0.36 mol equiv., 36%), and the seleninic acid **5** (0.075 mol equiv., 7.5%).



SCHEME 1

$$\underset{\substack{\mathbf{6}\\(1\text{ mol eq})}}{\text{Trip-SeH}} \xrightarrow[\substack{\text{1,4-Dioxane}\\ \text{r.t., 1.5 h}}]{\text{2M KOH}} \underset{\mathbf{4}\ (0.28\text{ mol eq})}{\text{Trip-SeSe-Trip}} + \underset{\mathbf{3}\ (0.36\text{ mol eq})}{\text{Trip-SeOH}} + \underset{\mathbf{5}\ (0.075\text{ mol eq})}{\text{Trip-SeO}_2\text{H}} \tag{5}$$

The isolation of the selenenic acid **3** in Equation 5 is an important observation because **3** was not detected in the reaction depicted in Equation 4. In addition, it implies that the condensation between selenol **6** and **3**, which gives the diselenide **4** (mechanism [ii]), is sluggish or does not take place. In fact, when a solution of equimolar amounts of **6** and **3** in 2 M KOH-1,4-dioxane was stirred for 1.5 hours at room temperature, the diselenide **4** was obtained only in a small amount (0.04 mol equiv.) along with selenenic acid **3** (1.10 mol equiv.) and seleninic acid **5** (0.81 mol equiv.) (Equation 6). The selenenic acid **3** itself was oxidized under similar conditions to give 0.29 mol equiv. of **5** (29%) with recovery of **3** (64%) (Equation 7).

$$\underset{\substack{\mathbf{6}\\(1\text{ mol eq})}}{\text{Trip-SeH}} + \underset{\substack{\mathbf{3}\\(1\text{ mol eq})}}{\text{Trip-SeOH}} \xrightarrow[\substack{\text{1,4-Dioxane}\\ \text{r.t., 1.5 h}}]{\text{2M KOH}} \begin{array}{c}\mathbf{4}\ (0.04\text{ mol eq})\\ +\\ \mathbf{3}\ (1.10\text{ mol eq})\\ +\\ \mathbf{5}\ (0.81\text{ mol eq})\end{array} \tag{6}$$

$$\underset{\substack{\mathbf{3}\\(1\text{ mol eq})}}{\text{Trip-SeOH}} \xrightarrow[\substack{\text{1,4-Dioxane}\\ \text{r.t., 1.5 h}}]{\text{2M KOH}} \underset{(0.29\text{ mol eq})}{\mathbf{5}} + \underset{(0.64\text{ mol eq})}{\mathbf{3}} \tag{7}$$

The result of the chemistry depicted in Equation 6 indicates that the presence of selenenic acid **3** substantially suppressed air oxidation of **6**, giving the diselenide **4**. As the air oxidation of thiols to disulfides [18] did, the air-oxidation of selenol **6** to the diselenide **4** under alkaline conditions could conceivably also be proceeded by a radical mechanism involving dimerization of Trip-Se·. It is likely that selenenic acid **3** acts as a radical scavenger to keep the concentration of the putative selenenyl radical low. Some sulfenic acids have been proved to have high antioxidant activity [19]. Thus, condensation between selenol **6** and selenenic acid **3** (mechanism [ii]) is not important under alkaline conditions.

It was reported that both areneselenenic anhydrides and areneselenenic acids react with thiols to give the selenenyl sulfides (Equations 8 and 9) [8,9,11,20,21], where the anhydrides are more reactive than the corresponding acids by 4.5 to 15 times depending upon the pH of the media [8,11]. It has also been reported that thioseleninates [ArSe(=O)SR] [22] and thiosulfinates [RS(=O)SR′] [15–17] react with thiols to yield disulfides.

$$\text{ArSeOSeAr} + \text{RSH} \longrightarrow \text{ArSeSR} + \text{ArSeOH} \qquad (8)$$

$$\text{AeSeOH} + \text{RSH} \longrightarrow \text{ArSeSR} + \text{H}_2\text{O} \qquad (9)$$

Selenoseleninate **1** (1 mol equiv.) was allowed to react with selenol **6** (1 mol equiv.) (mechanism [iii]). Surprisingly, the selenol **6** was completely consumed within 1 minute to give the diselenide **4** (0.45 mol equiv.) and the seleninic acid **5** (0.70 mol equiv.) in high yields along with selenenic acid **3** (0.18 mol equiv.) and **1** (0.39 mol equiv.) (Equation 10). This result proved that selenol **6** reacts with **1** to give diselenide **4** and selenenic acid **3**. A similar result was also obtained for the reaction conducted in an organic solvent.

$$\underset{(1\ \text{mol eq})}{\mathbf{1}} + \underset{\substack{\mathbf{6}\\(1\ \text{mol eq})}}{\text{Trip-SeH}} \xrightarrow[\substack{\text{1,4-Dioxane}\\ \text{r.t., 1 min}}]{\text{2M KOH}} \underset{\mathbf{4}\ (0.45\ \text{mol eq})}{\text{Trip-SeSe-Trip}} + \underset{\mathbf{3}\ (0.18\ \text{mol eq})}{\text{Trip-SeOH}} + \underset{\mathbf{5}\ (0.70\ \text{mol eq})}{\text{Trip-SeO}_2\text{H}} + \underset{(0.39\ \text{mol eq})}{\mathbf{1}} \qquad (10)$$

When selenoseleninate **1** (1 mol equiv.) was allowed to react with selenol **6** (2 mol equiv.) in dichloromethane at room temperature, the diselenide **4** (0.87 mol equiv., 87%) and the selenenic acid **3** (0.83 mol equiv., 83%) were obtained in high yields along with **6** (0.97 mol equiv., 49% recovery) (Equation 11). Selenenic acid **3** and selenol **6** did not react in this case, either. This low reactivity of **3** toward **6** under neutral conditions is comparable with that of 2-methyl-2-propanesulfenic acid (*t*-BuSOH) toward 2-methyl-2-propanethiol (*t*-BuSH) without acid [23].

$$\underset{(1\ \text{mol eq})}{\mathbf{1}} + \underset{\substack{\mathbf{6}\\(2\ \text{mol eq})}}{\text{Trip-SeH}} \xrightarrow[\text{r.t., 1 h}]{\text{CH}_2\text{Cl}_2} \underset{\mathbf{4}\ (0.87\ \text{mol eq})}{\text{Trip-SeSe-Trip}} + \underset{\mathbf{3}\ (0.83\ \text{mol eq})}{\text{Trip-SeOH}} + \mathbf{6}\ (0.97\ \text{mol eq}) \qquad (11)$$

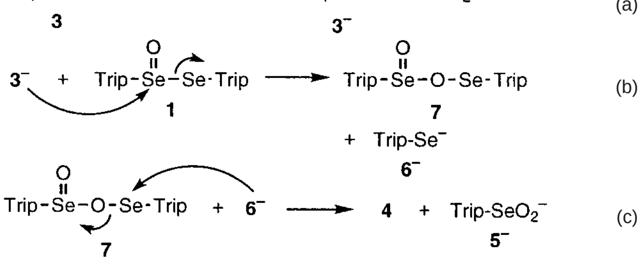
The reaction of Equation 10 showed that the air oxidation of selenol **6** to **3** and then **5** took place quickly, as did the reaction of **6** with **1** to give **4**. The yield of the selenenic acid **3** (0.18 mol equiv.) was lower, whereas that of seleninic acid **5** (0.70 mol equiv.) was higher than expected: the reaction of Equation 11 shows that the reaction of selenol **6** with **1** yields equimolar amounts of the diselenide **4** and the selenenic acid **3**. This also implies that selenenic acid **3** reacts with **1** at an appropriate rate. Hydrolysis of selenoseleninate **1** (1 mol equiv.) in the presence of selenenic acid **3** (1 mol equiv.) gave diselenide **4** (0.50 mol equiv.) and seleninic acid **5** (1.58 mol equiv.) (Equation 12). Although the selenenic acid **3** was not recovered at all (see Equation 7), the yield of **4** increased by 20% in comparison with that of **4** in Equation 4, suggesting that selenenic acid **3** participates in the formation of **4** by reacting with **1**.

$$\underset{(1\ \text{mol eq})}{\mathbf{1}} + \underset{\substack{\mathbf{3}\\(1\ \text{mol eq})}}{\text{Trip-SeOH}} \xrightarrow[\substack{\text{1,4-Dioxane}\\ \text{r.t., 1.5 h}}]{\text{2M KOH}} \underset{\mathbf{4}\ (0.50\ \text{mol eq})}{\text{Trip-SeSe-Trip}} + \underset{\mathbf{5}\ (1.58\ \text{mol eq})}{\text{Trip-SeO}_2\text{H}} \qquad (12)$$

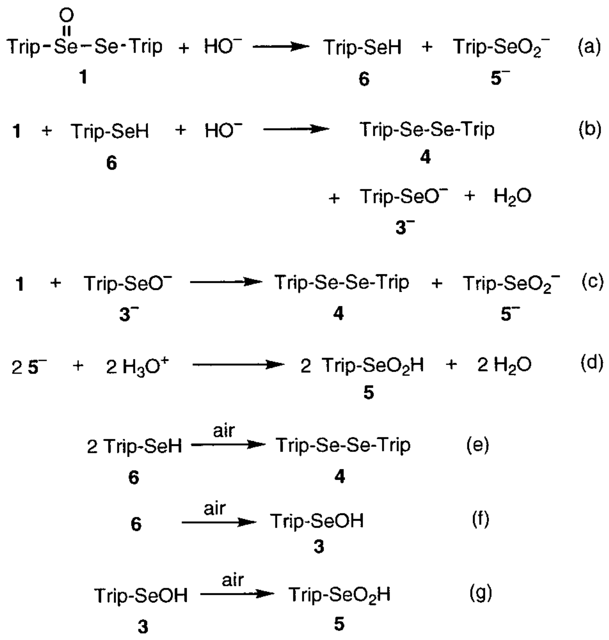
Scheme 2 shows a plausible mechanism for the reaction of **3** with **1**. The anion $\mathbf{3}^-$ attacks the seleninyl selenium of **1** to give *O*-selenenyl selenoseleninate **7** and selenolate $\mathbf{6}^-$ (Reaction b). The intermediate **7** further reacts with $\mathbf{6}^-$ to yield the diselenide **4** and seleninic acid **5** (Reaction c). *O*-Selenenyl selenoseleninates are a second oxidation product of diselenides [4,9] and have never been isolated nor observed spectroscopically.

Thus, three pathways for the formation of diselenide **4** were verified: (i) air oxidation of selenol **6** (Equation 5), (ii) the reaction of **6** and selenoseleninate **1** (Equation 10), and (iii) the reaction of selenenic acid **3** with **1** (Equation 12). Considering that the presence of selenenic acid **3** pushed up the yield of **4** by only 20% (Equation 12), the pathway (iii) is a minor contributor.

Scheme 3 depicts the mechanism for the alkaline hydrolysis of **1**. At first, hydroxide ion, a hard nucleophile, attacks the seleninyl selenium of **1** to give selenol **6** and seleninate $\mathbf{5}^-$ (Reaction a) [17]. The selenol **6** reacts with **1** to give diselenide **4** and selenenate $\mathbf{3}^-$ (Reaction b), and the latter further reacts with **1** to yield **4** and $\mathbf{5}^-$ (Reaction c). Finally, acidi-

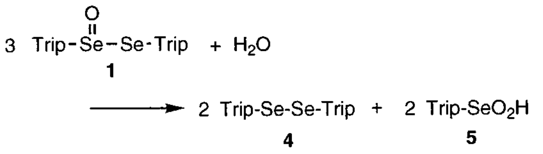


SCHEME 2



SCHEME 3

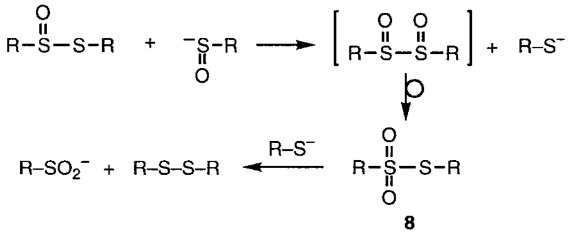
fication of 5^- gives seleninic acid **5** (Reaction d). Summation of the Reactions (a–d) gives Equation 13, meaning that the maximum amounts of **4** and **5** from 1 mol equiv. of **1** are both 0.67 mol equiv. The much lower yield of diselenide **4** in Equation 4 (0.38 mol equiv.) indicates that selenol **6** and selenenic acid **3** are susceptible to air oxidations (e–g), where the Reactions f and g are more important than the Reaction e.



$$3\ \underset{\mathbf{1}}{\text{Trip-}\overset{\text{O}}{\overset{\|}{\text{Se}}}\text{-Se-Trip}} + H_2O \longrightarrow 2\ \underset{\mathbf{4}}{\text{Trip-Se-Se-Trip}} + 2\ \underset{\mathbf{5}}{\text{Trip-SeO}_2\text{H}} \qquad (13)$$

The Reactions a–d in Scheme 3 are quite similar to the mechanism for alkaline hydrolysis of thiosulfinates [RS(=O)SR], which was investigated by Oae et al. by use of ^{18}O-tracer experiments [17]. In the Oae mechanism, the reaction of RS$^-$(=O) with RS(=O)SR, corresponding to Reaction c in Scheme 3, gives RS$^-$ and thiosulfonate **8** by rearrangement of intermediary formed RS(=O)S(=O)R (Scheme 4). Although they confirmed the formation of **8** by liquid chromatography, we could not detect the corresponding selenoselenonate [TripSe(O$_2$)SeTrip].

In conclusion, acid-catalyzed hydrolysis of selenoseleninate **1** yielded the selenenic acid **3**, while alkaline hydrolysis of **1** gave diselenide **4** and seleninic acid **5**. Whereas the alkaline hydrolysis of **1**, in principle, takes place by a series of reactions similar to that for thiosulfinates [17], air oxidations of the resulting selenol **6** and selenenic acid **3** to seleninic acid **5** also occur at a considerable rate to decrease the yield of diselenide **4**.



SCHEME 4

EXPERIMENTAL

Melting points were determined on a capillary tube apparatus and are uncorrected. ^{1}H and ^{13}C NMR spectra were obtained in CDCl$_3$ as the solvent unless otherwise noted. Elemental analyses were performed by the Chemical Analysis Center of Saitama University. In workup of the reactions, the extracts were dried over anhydrous MgSO$_4$ after washing with water.

Hydrolysis of TripSe(O)SeTrip 1 *under Acidic Conditions*

To selenoseleninate **1** (42.7 mg, 0.0627 mmol), placed in a 50 mL round-bottom flask, was added 1,4-dioxane (3 mL) to give a slightly suspended solution. 2 M HClO$_4$ (1.0 mL) was added to it, and the mixture was stirred for 5.5 hours at room temperature. The mixture was extracted with dichloromethane, and the extract was washed with water, dried, and evaporated to dryness to provide 40.3 mg of a mixture of selenenic acid **3** (74%) and **1** (18%). The yields were calculated on the basis of the integral ratio of the ^{1}H NMR spectrum.

Hydrolysis of TripSe(O)SeTrip 1 *under Alkaline Conditions*

To selenoseleninate **1** (57.7 mg, 0.0848 mmol), placed in a 30 mL round-bottom flask, was added 1,4-dioxane (6 mL). 2 M KOH (2 mL) was added to the slightly suspended solution, and the mixture was stirred for 1.5 hours at room temperature. The mixture was extracted with dichloromethane. The organic layer was washed with 0.2 M KOH several times and then with water, dried, and evaporated to dryness to give diselenide **4** (21.4 mg, 0.0322 mmol). On the other hand, the aqueous layer and the washings of the organic layer were combined, acidified to pH 5 by dilute hydrochloric acid, and extracted with dichloromethane. The extract was dried and evaporated to dryness to give seleninic acid **5** (26.5 mg, 0.0725 mmol).

Preparation of the Authentic Sample of TripSeO$_2$H **5** *by Oxidation of Trip-SeOH* **3**

To a solution of selenenic acid **3** (64.9 mg, 0.186 mmol) in dichloromethane (15 mL) was added dimethyldioxirane in acetone [24] (0.069 M, 2.7 mL, 0.186 mmol) at −78°C. After having been stirred for 1 hour at −78°C, the mixture was warmed to room temperature, dried, and evaporated to dryness to give **5** (64.7 mg, 95%): colorless crystals, m.p. 170°C decomp. ^{1}H NMR (400 MHz): in $CDCl_3$ δ 5.40 (s, 1H), 7.08–7.09 (m, 6H), 7.45–7.47 (m, 3H), 8.06 (br s, 3H); in DMSO-d$_6$ δ 5.66 (s, 1H), 7.04–7.10 (m, 6H), 7.52–7.54 (m, 3H), 7.94 (br s, 3H); IR (KBr) 3416, 2476, 847 cm^{-1}. Anal. calcd. for $C_{20}H_{14}O_2Se$; C, 65.76; H, 3.86. Found: C, 65.72; H, 3.84.

Air Oxidation of TripSeH **6** *under Alkaline Conditions*

To a solution of selenol **6** (24.2 mg, 0.726 mmol) in 1,4-dioxane (3 mL) was added 1 M KOH (1 mL). The color of the mixture soon turned orange, indicating the formation of diselenide **4**. After having been stirred for 1.5 hours at room temperature, the mixture was extracted with dichloromethane. The organic layer was washed with 0.2 M KOH and water, dried, and evaporated to dryness. The residue was subjected to column chromatography (CCl_4) to give diselenide **4** (13.5 mg, 0.0203 mmol, 56%) and selenenic acid **3** (9.1 mg, 0.026 mmol, 36%). The aqueous layer and the washings of the dichloromethane layer were combined, acidified by dilute hydrochloric acid, and extracted with dichloromethane. The extract was dried and evaporated to dryness to give seleninic acid **5** (2.0 mg, 0.0055 mmol, 7.5%).

Air Oxidation of TripSeOH **3** *under Alkaline Conditions*

Selenenic acid **3** (20.3 mg, 0.0581 mmol) in 1,4-dioxane (2.4 mL) was treated with 2 M KOH (0.8 mL). A workup similar to the one previously described gave **3** (5.9 mg, 0.017 mmol, 29%) from the organic layer and seleninic acid **5** (13.5 mg, 0.370 mmol, 64%) from the aqueous layer.

Reaction of TripSeH **6** *and TripSeOH* **3** *under Alkaline Conditions*

A mixture of selenol **6** (14.5 mg, 0.0435 mmol) and selenenic acid **3** (15.2 mg, 0.0435 mmol) in 1,4-dioxane (3.6 mL) was treated with 2 M KOH (1.2 mL). A workup similar to the one previously described gave diselenide **4** (1.3 mg, 0.0019 mmol) and selenenic acid **3** (16.7 mg, 0.0478 mmol) from the organic layer and seleninic acid **5** (12.8 mg, 0.0350 mmol) from the aqueous layer.

Reaction of TripSe(O)SeTrip **1** *and TripSeH* **6** *under Alkaline Conditions*

A mixture of selenoseleninate **1** (22.4 mg, 0.033 mmol) and selenol **6** (11.0 mg, 0.033 mmol) in 1,4-dioxane (3.6 mL) was treated with 2 M KOH (1.2 mL). The color of the mixture immediately turned from yellow to orange, and after 1 minute the mixture was extracted with dichloromethane three times. A workup similar to the one previously described gave diselenide **4** (10.3 mg, 0.0155 mmol) and selenenic acid **3** (2.1 mg, 0.06 mmol) from the organic layer, and seleninic acid **5** (8.4 mg, 0.023 mmol) from the aqueous layer.

Reaction of TripSe(O)SeTrip **1** *and TripSeH* **6** *in Dichloromethane*

Selenoseleninate **1** (15.1 mg, 0.0222 mmol) and selenol **6** (14.9 mg, 0.0447 mmol) were placed in a round-bottom flask and dissolved in dichloromethane (5 mL). The color of the solution gradually turned from colorless to orange. The solution was stirred for 1 hour at room temperature, and then the solvent was removed under reduced pressure. The residue was subjected to column chromatography (CH_2Cl_2) to give selenenic acid **3** (6.4 mg, 0.018 mmol) and a mixture of selenol **6** and diselenide **4**. The mixture was separated by column chromatography (silica gel, hexane-CH_2Cl_2 4:1) to provide selenol **6** (7.2 mg, 0.022 mmol) and diselenide **4** (12.9 mg, 0.0194 mmol).

Hydrolysis of TripSe(O)SeTrip **1** *under Alkaline Conditions in the Presence of TripSeOH* **3**

To selenoseleninate **1** (46.2 mg, 0.0679 mmol) and selenenic acid **3** (23.7 mg, 0.0679 mmol), placed in a bottom-round flask under argon, was added 1,4-dioxane (6 mL) and 2 M KOH (2 mL). The mixture was stirred for 1.5 hours at room temperature and diluted with dichloromethane. The organic layer was separated, washed with 0.2 M KOH, dried, and evaporated to dryness to give diselenide **4** (22.5 mg, 0.0339 mmol). The aqueous layer and the washing of the organic layer were combined, acidified by dilute hydrochloric acid, and extracted with dichloromethane. The extract was dried and evaporated to dryness to give seleninic acids **5** (39.2 mg, 0.107 mmol).

REFERENCES

[1] Reich, H. J.; Hoeger, C. A.; Willis, W. W. Jr. J Am Chem Soc 1982, 104, 2936–2937.

[2] Reich, H. J.; Hoeger, C. A.; Willis, W. W. Jr. Tetrahedron 1985, 41, 4771–4779.

[3] Bergson, G. Acta Chem Scand 1961, 15, 1611–1613.

[4] Ayrey, G.; Barnard, D.; Woodbridge, D. T. J Chem Soc 1962, 2089–2099.

[5] Reich, H. J.; Jasperse, C. P. J Org Chem 1988, 53, 2390–2392.

[6] Ishii, A.; Matsubayashi, S.; Takahashi, T.; Nakayama, J. J Org Chem 1999, 64, 1084–1085.

[7] Reich, H. J.; Willis, W. W. Jr.; Wollowitz, S. Tetrahedron Lett 1982, 23, 3319–3322.

[8] Kice, J. L.; McAfee, F.; Slebocka-Tilk, H. Tetrahedron Lett 1982, 23, 3323–3326.

[9] Kice, J. L.; Kang, Y.-H.; Manek, M. B. J Org Chem 1988, 53, 2435–2439.

[10] Kice, J. L.; McAfee, F.; Slebocka-Tilk, H. J Org Chem 1984, 49, 3100–3106.

[11] Kice, J. L.; McAfee, F.; Slebocka-Tilk, H. J Org Chem 1984, 49, 3106–3114.

[12] Hori, T.; Sharpless, K. B. J Org Chem 1978, 43, 1689–1697.

[13] Reich, H. J.; Wollowitz, S.; Trend, J. E.; Chow, F.; Wendelborn, D. F. J Org Chem 1978, 43, 1697–1705.

[14] Gancarz, R. A.; Kice, J. L. Tetrahedron Lett 1981, 22, 1661–1662.

[15] Lacombe, S. M. In Reviews on Heteroatom Chemistry; Oae, S., Ed.; MYU: Tokyo, 1999; Vol. 21, pp 1–41.

[16] (a) Isenberg, N.; Grdinic, M. Int J Sulfur Chem 1973, 8, 307–320; (b) Kice, J. L. Adv Phys Org Chem 1980, 17, 65–181; (c) Hogg, D. R. In The Chemistry of Sulfenic Acids and Their Derivatives; Patai, S., Ed.; Wiley: New York, 1990; pp 361–402.

[17] Oae, S.; Takata, T.; Kim, Y. H. Tetrahedron Lett 1977, 4219–4222, and references cited therein.

[18] Uemura, S. Comprehensive Organic Synthesis; Trost, B. M., Ed.; Pergamon: Oxford, 1991; Vol. 7, pp 757–787.

[19] Koelewijn, P.; Berger, H. Recueil 1972, 91, 1275–1286.

[20] Goto, K.; Okazaki, R. Libigs Ann/Recueil 1997, 2393–2407.

[21] Saiki, T.; Goto, K.; Okazaki, R. Angew Chem Int Ed Engl 1997, 36, 2223–2224.

[22] Kice, J. L.; Lee, T. W. S. J Am Chem Soc 1978, 100, 5094–5102.

[23] Davis, F. A.; Billmers, R. L. J Am Chem Soc 1981, 103, 7016–7018.

[24] (a) Adam, W.; Bialas, J.; Hadjiarapoglou, L. Chem Ber 1991, 124, 2377; (b) Adam, W.; Hadjiarapoglou, L.; Smerz, A. Chem Ber 1991, 124, 227–232.