Reductive Cleavage of the Se–Se Bond in Diselenides by the Sm/HgCl$_2$ System: Formation and Reactions of Samarium Selenolates

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ABSTRACT: *Treatment of diaryl diselenides and dialkyl diselenides with metallic samarium and mercury (II) chloride in tetrahydrofuran conveniently gave samarium arylselenolates and alkylselenolates. These "living" species reacted with acyl chlorides, acid anhydrides, methyl chloroformate, organic halides, epoxides, α,β-unsaturated esters, and an α,β-unsaturated nitrile to afford selenoesters, selenoformates, and unsymmetrical selenides in good yields under mild and neutral condition.* © 1999 John Wiley & Sons, Inc. Heteroatom Chem 10: 203–208, 1999

INTRODUCTION

Organoselenium compounds have received considerable attention as useful synthetic reagents and intermediates in organic synthesis [1]. While there are many methods for the introduction of a selenium moiety into organic molecules, the use of selenide anions is especially convenient and common. Several methods for the synthesis of selenide anions have been recommended, the more important of

which include the use of diphenyl diselenide with sodium borohydride in THF/EtOH [2] or by use of resin bound borohydride [3], reduction of the diselenide with sodium in ammonia [4] or in boiling THF followed by solvation in HMPA [5], and with lithium aluminum hydride [6]. Alternatively, sodium phenylselenide may be obtained from the selenol using sodium hydride in THF [5] or even by treatment with aqueous sodium hydroxide under certain conditions [7]. The use of diphenyl diselenide and samarium diiodide in THF/HMPA has also recently been reported to give a selenide anion [8,9]. Moreover, Grignard reagents also react with selenium to give selenide anions [10], although aggregates formed in these reactions can react with variable results.

As a powerful, versatile, and ether-soluble one-electron transfer reducing agent, SmI$_2$ has played an ever-increasing role in organic synthesis since its introduction by H. B. Kagan and his group [11]. Samarium diiodide promotes a number of important individual reactions found useful in organic synthesis [12]. Though SmI$_2$ is a useful reagent, storage is difficult because it is very sensitive to air oxidation. On the other hand, metallic samarium is stable in air, and its strong reducing power (Sm^{3+}/Sm = −2.41 V) is similar to that of magnesium (Mg^{2+}/Mg = −2.37 V), and superior to that of zinc (Zn^{2+}/Zn = −0.71 V). These properties influenced us to use the more convenient and cheaper metallic samarium directly instead of samarium (II) iodide. Recently,

there have been some reports on the direct use of Sm in organic synthesis [13] and on the use of the Sm/HgCl$_2$ system in the cyclopropanation of allylic and α-allenic alcohols [14]. Herein, we wish to report that reductive cleavage of the Se–Se bond in diselenides by the Sm/HgCl$_2$ system led to selenolate anion species, which reacted with acyl chlorides, acid anhydrides, methyl chloroformate, organic halides, epoxides, α,β-unsaturated esters, and an α,β-unsaturated nitrile to afford selenoesters, selenoformates, and unsymmetrical selenides in good yields under mild and neutral condition.

RESULTS AND DISCUSSION

To a solution of mercury (II) chloride in dry THF was added metallic samarium powder under a nitrogen atmosphere at 30–40°C. The mixture was stirred for 2 hours. Samarium was gradually dissolved in THF, and the color of the solution changed to greenish blue, which was similar to the THF solution of samarium (II) diiodide obtained by Kagan's method. Moreover, we found that metallic mercury is formed during the reaction. We assumed that a divalent samarium species is formed in situ by the reaction of metallic samarium powder with mercury (II) chloride in THF.

The formation and reactions of samarium arylselenolates and alkylselenolates, which were formed in situ by the cleavage of the corresponding diselenides with the Sm/HgCl$_2$ reduction system, are shown in Schemes 1 and 2. The formation of selenolate anion species with Sm/HgCl$_2$ is similar to that with SmI$_2$ [8,9]. The results of our experiments in this article are summarized in Tables 1 and 2.

Table 1 indicates that, under mild conditions, selenolate anions species, easily prepared in situ from the cleavage of the Se–Se bond in diselenides by Sm/HgCl$_2$ reduction system, reacted smoothly with acyl chlorides, acid anhydrides, and methyl chloroformate, respectively, to afford the desired selenoesters
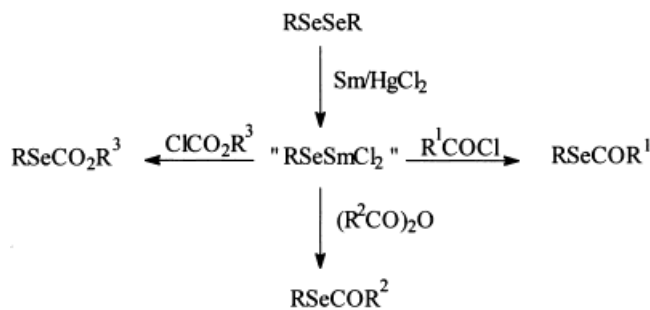
and selenoformates in good yields. We found that cleavage of the Se–Se bond in diaryl diselenide is easier than that in dialkyl diselenides (entries **g** and **h**).

We also found that the phenylselenolate anion species, formed in situ by the cleavage of the Se–Se bond in diphenyl diselenide with Sm/HgCl$_2$, reacted easily with epoxides, α,β-unsaturated esters, and an α,β-unsaturated nitrile, respectively, to give the unsymmetrical alkylphenylselenides in good yields (Table 2). In order to prevent polymerization during the coupling reaction, *iso*-propyl alcohol or *t*-butyl alcohol was added. From Table 2, we can see that the phenylselenolate anion, a "living" species, reacted smoothly with active organic halides, such as 2-bromoacetophenone and ethyl bromoacetate, to give the expected products in good yields at 40°C. When R^{4}X is a less active halide, such as an alkyl bromide or benzyl chloride, the coupling reaction should be carried out at a higher temperature (65°C). Unfortunately, alkyl chlorides are unreactive even with a longer reaction time (entry **r**).

EXPERIMENTAL

General

All melting points are uncorrected. ^{1}H NMR spectra were recorded on a Brucker AC-80 instrument. All NMR samples were measured in CCl$_4$ using TMS as an internal standard. IR spectra were obtained on a Perkin-Elmer 683 infrared spectrophotometer. Metallic samarium and other chemicals were purchased from commercial sources and used without purification. THF was freshly distilled from sodium/benzophenone ketyl prior to use.

Reaction of Diselenides with Acyl Chlorides, Acid Anhydrides, and Methyl Chloroformate Mediated by Sm/HgCl$_2$

General Procedure for the Synthesis of Selenoesters and Selenoformates. Under a nitrogen atmo-
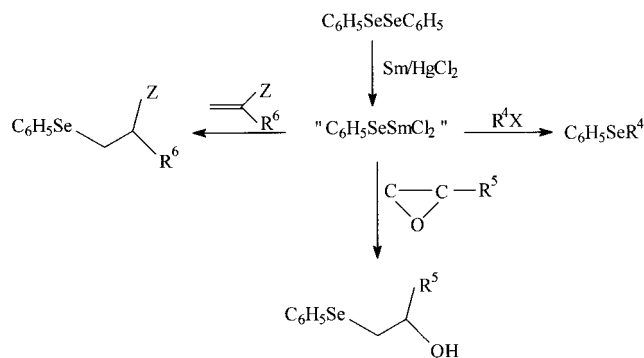
SCHEME 1

SCHEME 2

TABLE 1 Synthesis of Selenoesters and Selenoformates *via* the Selenolate Anion Intermediates

| Entry | R | R^1 | R^2 | R^3 | Cleavage Reaction Conditions | | Yield (%)[a] |
					Temp. (°C)	Time (h)	
a	C$_6$H$_5$	CH$_3$			40	2	75
b	C$_6$H$_5$	n-C$_3$H$_7$			40	2	73
c	C$_6$H$_5$	C$_6$H$_5$			40	2	74
d	C$_6$H$_5$	4-CH$_3$OC$_6$H$_4$			40	2	78
e	C$_6$H$_5$	2-BrC$_6$H$_4$			40	2	76
f	C$_6$H$_5$	4-ClC$_6$H$_4$			40	2	79
g	n-C$_4$H$_9$	C$_6$H$_5$			55	4	65
h	n-C$_4$H$_9$	2-BrC$_6$H$_4$			55	4	68
i	4-ClC$_6$H$_4$	C$_6$H$_5$			40	2	77
j	C$_6$H$_5$		CH$_3$		40	2	83
k	C$_6$H$_5$		n-C$_3$H$_7$		40	2	76
l	C$_6$H$_5$			CH$_3$	40	2	85
m	4-ClC$_6$H$_4$			CH$_3$	40	2	80

Reaction conditions: diselenide 0.5 mmol, Sm 1.2 mmol, HgCl$_2$ 1.0 mmol, THF 7 mL, nucleophilic substitution reaction temperature 40°C for 1 hour.
[a]Isolated yields.

TABLE 2 Preparation of Unsymmetrical Phenylselenides *via* the Phenylselenolate Anion Intermediate

| Entry | R^{4}X | R^5 | R^6 | Z | Coupling Reaction Conditions | | Yield (%)[a] |
					Temp. (°C)	Time (h)	
n	C$_6$H$_5$CH$_2$Cl				65	4	72
o	C$_6$H$_5$COCH$_2$Br				40	3	69
p	BrCH$_2$CO$_2$C$_2$H$_5$				40	3	68
q	n-C$_4$H$_9$Br				65	4	70
r	n-C$_4$H$_9$Cl				65	8	—
s	n-C$_6$H$_{13}$Br				65	4	68
t	n-C$_8$H$_{17}$Br				65	4	72
u		H			40	2	80
v		CH$_2$Cl			40	2	81
w			H	CN	40	2	76
x			H	CO$_2$CH$_3$	40	2	85
y			CH$_3$	CO$_2$CH$_3$	40	2	83

[a]Isolated yields.
Reaction conditions: diphenyl diselenide 0.5 mmol, Sm 1.2 mmol, HgCl$_2$ 1.0 mmol, THF 7 mL, cleavage reaction temperature 40°C for 2 hours.

sphere, metallic samarium powder (0.18 g, 1.2 mmol), mercury (II) chloride (0.272 g, 1.0 mmol), and the appropriate diselenide (0.5 mmol) were placed in a three-necked reaction flask and THF (7 mL) was added in one portion. The resulting mixture was magnetically stirred at 40°C for the time indicated in Table 1 until the powdered samarium was almost consumed. To the mixture was added successively the acyl chloride (1.2 mmol) or the acid anhydride (1.2 mmol) or methyl chloroformate (1.2 mmol) in THF (5 mL), respectively. When the reaction appeared to be finished, water (4 mL) was added, and the mixture was extracted with ether (20 mL × 2). (Note: Because the metallic mercury formed in the reaction is poisonous, the water phase containing the mercury species cannot be poured out as trash, and the recommendable method in our laboratory is to decant the water and recover the mercury at the bottom of the beaker.) After the extracts had been washed with brine, dried over anhydrous Na$_2$SO$_4$, and the solvent removed under reduced pressure, the residue was then purified by preparative TLC on silica gel with cyclohexane and ethyl acetate (19:1) as eluent.

Se-phenyl Ethaneselenoate [15]. Light yellow

oil. ^{1}H NMR δ 2.26 (3H, s, -CH$_3$), 6.90–7.45 (5H, m, ArH). IR (film) v_{max} (cm^{-1}) 3090, 2950, 1745, 1580, 1485, 1445, 1440, 1355, 1100, 940, 780, 750, 685.

Se-phenyl Butaneselenoate [16].   Pale yellow oil. ^{1}H NMR δ 0.93 (3H, t, -CH$_3$), 1.10–2.01 (2H, m, -CH$_2$-), 2.55 (2H, t, -COCH$_2$-), 6.90–7.50 (5H, m, ArH). IR(film) v_{max} (cm^{-1}) 3070, 2940, 1730, 1590, 1490, 1440, 1400, 1350, 1115, 940, 740, 690.

Se-phenyl Benzeneselenoate [15].   Light yellow solid, mp 39–40°C (Ref. [15] 40°C): ^{1}H NMR δ 7.00–8.04 (10H, m, ArH). IR (KBr) v_{max} (cm^{-1}) 3080, 3060, 2970, 1710, 1590, 1460, 1440, 1280, 1180, 1110, 935, 780, 755, 705, 680.

Se-phenyl 4-Methoxybenzeneselenoate [17].   Pale light crystals, mp 64–65°C (Ref. [17] 62–63°C); ^{1}H NMR δ 3.61 (3H, s, -OCH$_3$), 6.50–7.95 (9H, m, ArH). IR (KBr) v_{max} (cm^{-1}) 3080, 3040, 2990, 2860, 1700, 1610, 1585, 1513, 1485, 1445, 1445, 1320, 1165, 880, 830, 780, 760, 686.

Se-phenyl 2-Bromobenzeneselenoate [15].   Pale light crystals, mp 66–67°C (Ref. [15] 65°C); ^{1}H NMR δ 7.05–7.75 (9H, m, ArH). IR (KBr) v_{max} (cm^{-1}) 3040, 2960, 1650, 1590, 1540, 1430, 1420, 1213, 1140, 980, 950, 830, 810, 715, 690.

Se-phenyl 4-Chlorobenzeneselenoate [17].   Pale light crystals, mp 84–85°C (Ref. [17] 83–84°C); ^{1}H NMR δ 7.00–7.70 (9H, m, ArH). IR (KBr) v_{max} (cm^{-1}) 3070, 2980, 1670, 1580, 1560, 1482, 1460, 1445, 1250, 1230, 1150, 950, 940, 880, 805, 760, 728, 680.

Se-butyl Benzeneselenoate [15].   Pale yellow oil. ^{1}H NMR δ 0.90 (3H, t, -CH$_3$), 1.15–1.96 [4H, m, -(CH$_2$)$_2$-], 3.01 (2H, t, -SeCH$_2$-), 7.20–7.95 (5H, m, ArH). IR (film) v_{max} (cm^{-1}) 3078, 2986, 2940, 2890, 1670, 1580, 1480, 1467, 1450, 1260, 1200, 1173, 930, 880, 760, 682.

Se-butyl 2-Bromobenzeneselenoate [15].   Light yellow oil. ^{1}H NMR δ 0.90 (3H, t, -CH$_3$), 1.16–1.98 [4H, m, -(CH$_2$)$_2$-], 2.95 (2H, t, -SeCH$_2$-), 6.95–7.70 (4H, m, ArH). IR (film) v_{max} (cm^{-1}) 3080, 2980, 1700, 1600, 1580, 1480, 1452, 1200, 1175, 925, 870, 760, 725, 680.

Se-4-chlorophenyl Benzeneselenoate [18].   Light yellow crystals, mp 72–73°C (Ref. [18] 74–75°C); ^{1}H NMR δ 7.03–8.08 (9H, m, ArH). IR (KBr) v_{max} (cm^{-1}) 3072, 2980, 1700, 1600, 1580, 1480, 1452, 1200, 1175, 1110, 925, 870, 810, 760, 725, 680.

Phenylselenoformate [19].   Pale yellow oil. ^{1}H NMR δ 3.65 (3H, s, -OCH$_3$), 6.95–7.67 (5H, m, ArH). IR (film) v_{max} (cm^{-1}) 3030, 2970, 1735, 1580, 1485, 1440, 1420, 1275, 1190, 1120, 800, 740, 720, 685.

4-Chlorophenylselenoformate [20].   Pale yellow oil. ^{1}H NMR δ 3.65 (3H, s, -OCH$_3$), 7.00–7.65 (4H, m, ArH). IR (film) v_{max} (cm^{-1}) 3030, 2970, 1740, 1580, 1480, 1430, 1380, 1265, 1190, 1125, 1090, 1010, 935, 810, 725, 625.

Reaction of Diphenyl Diselenide with Organic Halides Mediated by Sm/HgCl$_2$

General Procedure for the Preparation of Unsymmetrical Phenylselenides.   Under a nitrogen atmosphere, metallic samarium powder (0.18 g, 1.2 mmol), mercury (II) chloride (0.272 g, 1.0 mmol), and diphenyl diselenide (0.16 g, 0.5 mmol) were placed in a three-necked reaction flask, and THF (7 mL) was added in one portion. The resulting mixture was magnetically stirred for 2 hours at 40°C until the powdered samarium was almost consumed. To the mixture was added successively the organic halide (1.2 mmol) in THF (5 mL). When the reaction appeared to be finished, water (4 mL) was added, and the mixture was extracted with ether (20 mL × 2). (Note: Because the metallic mercury formed in the reaction is poisonous, the water phase containing the mercury species cannot be poured out as trash, and the recommendable method in our laboratory is to decant the water and recover the mercury at the bottom of the beaker.) After the extracts had been washed with brine, dried over anhydrous Na$_2$SO$_4$, and the solvent removed under reduced pressure, the residue was then purified by preparative TLC on silica gel to give pure product.

Benzyl Phenylselenide [21].   Light yellow oil. ^{1}H NMR δ 3.95 (2H, s, -CH$_2$Se-), 7.05–7.45 (10H, m, ArH). IR (film) v_{max} (cm^{-1}) 3090, 2980, 1580, 1490, 1440, 1150, 940, 740, 690.

2-(Phenylseleno)acetophenone [9].   Light yellow oil. ^{1}H NMR δ 4.05 (2H, s, -CH$_2$Se-), 7.00–7.95 (10H, m, ArH). IR (film) v_{max} (cm^{-1}) 3085, 2960, 1682, 1600, 1590, 1480, 1410, 1275, 1180, 925, 735, 700, 680.

Ethyl 2-(Phenylseleno)acetate [22].   Pale yellow oil. ^{1}H NMR δ 1.12 (3H, t, -CH$_3$), 3.29 (2H, s, -CH$_2$Se-), 3.96 (2H, q, -OCH$_2$-), 7.01–7.65 (5H, m, ArH). IR (film) v_{max} (cm^{-1}) 3070, 2960, 1750, 1590, 1485, 1460, 1350, 1220, 1170, 935, 740, 690.

1-Butyl Phenylselenide [23].   Light yellow oil. ^{1}H NMR δ 0.80 (3H, t, -CH$_3$), 1.00–1.85 [4H, m, -(CH$_2$)$_2$-], 2.70 (2H, t, -CH$_2$Se-), 6.95–7.55 (5H, m, ArH). IR (film) v_{max} (cm^{-1}) 3050, 2950, 1590, 1470, 1420, 1370, 1220, 1115, 920, 735, 700.

1-Hexyl Phenylselenide [24].   Pale yellow oil. ¹H NMR δ 0.80 (3H, t, -CH₃), 1.00–1.95 [8H, m, -(CH₂)₄-], 2.95 (2H, t, -CH₂Se-), 7.00–7.60 (5H, m, ArH). IR (film) v_{max} (cm⁻¹) 3040, 2940, 1580, 1480, 1420, 1380, 1230, 1100, 930, 740, 690.

1-Octyl Phenylselenide [25].   Pale yellow oil. ¹H NMR δ 0.80 (3H, t, -CH₃), 1.00–2.10 [12H, m, -(CH₂)₆-], 3.20 (2H, t, -CH₂Se-), 7.02–7.70 (5H, m, ArH). IR (film) v_{max} (cm⁻¹) 3020, 2945, 2860, 1589, 1460, 1400, 1220, 1110, 1041, 928, 790, 740, 690.

Reaction of Diphenyl Diselenide with Epoxides, α,β-Unsaturated Esters and an α,β-Unsaturated Nitrile Mediated by Sm/HgCl₂

General Procedure.   Under a nitrogen atmosphere, metallic samarium powder (0.18 g, 1.2 mmol), mercury (II) chloride (0.272 g, 1.0 mmol), and diphenyl diselenide (0.16 g, 0.5 mmol) were placed in a three-necked reaction flask, and THF (7 mL) was added in one portion. The resulting mixture was magnetically stirred for 2 hours at 40°C until the powdered samarium was almost consumed. To the mixture was added successively the epoxide (1.2 mmol) or the α,β-unsaturated ester (1.2 mmol) or an α,β-unsaturated nitrile (1.2 mmol) in THF (5 mL) and *iso*-propyl alcohol (0.2 mL, in order to prevent the polymerization), respectively, and the same workup procedure as described previously provided the desired unsymmetrical selenides.

2-(Phenylseleno)ethanol [26].   Light yellow oil. ¹H NMR δ 2.34 (1H, s, OH, disappeared by adding D₂O), 2.90 (2H, t, -CH₂Se-), 3.59 (2H, t, -CH₂O-), 7.00–7.54 (5H, m, ArH). IR (film) v_{max} (cm⁻¹) 3530, 3040, 2955, 2900, 1600, 1490, 1462, 925, 740, 700.

1-Chloro-3-(phenylseleno)-2-propanol   [27].   Light yellow oil. ¹H NMR δ 2.73 (1H, s, OH, disappeared by adding D₂O), 3.00 (2H, d, -CH₂Se-), 3.52 (2H, d, -CH₂Cl), 3.64–4.00 (1H, m, CH), 7.03–7.49 (5H, m, ArH). IR (film) v_{max} (cm⁻¹) 3450, 3089, 3050, 2945, 2900, 1590, 1485, 1460, 1050, 1020, 940, 735, 700.

β-(Phenylseleno)propionitrile [28].   Light yellow oil. ¹H NMR δ 2.30–2.70 (2H, m, -CH₂CN), 2.74–3.13 (2H, m, -CH₂Se-), 7.00–7.52 (5H, m, ArH). IR (film) v_{max} (cm⁻¹) 3100, 3080, 2950, 2254, 1588, 1485, 1445, 1420, 1070, 1020, 932, 735, 685.

Methyl-3-(phenoylseleono)propanate [25].   Pale light oil. ¹H NMR δ 2.33–2.77 (2H, m, -CH₂CO₂R),

2.80–3.18 (2H, m, -CH₂Se-), 3.50 (3H, s, -OCH₃), 6.97–7.53 (5H, m, ArH). IR (film) v_{max} (cm⁻¹) 3060, 2965, 1750, 1590, 1485, 1460, 1355, 1225, 1165, 1021, 935, 735, 690.

Methyl 2-Methyl-3-(phenylseleno)propanate [29].   Pale yellow oil. ¹H NMR δ 1.10–1.20 (3H, d, -CH₃), 2.40–2.60 (2H, m, CH), 2.65–3.20 (2H, m, -CH₂Se-), 3.54 (3H, s, -OCH₃), 7.00–7.65 (5H, m, ArH). IR (film) v_{max} (cm⁻¹) 3065, 2950, 1745, 1590, 1480, 1450, 1360, 1230, 1170, 930, 730, 690.

CONCLUSION

We have demonstrated that the Sm/HgCl₂ system can be used for the reductive cleavage of the Se–Se bond in diselenides. This method for the synthesis of selenoesters, selenoformates, and unsymmetrical selenides appears to be a very convenient and practical method in terms of mild reaction conditions, simple operation, and high yields.

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