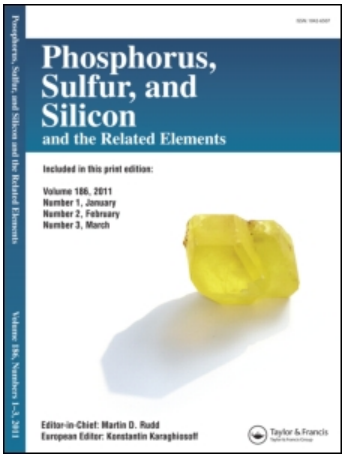

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The Preparation of Acylselenourea and Selenocarbamate Using Isoselenocyanate

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The Preparation of Acylselenourea and Selenocarbamate Using Isoselenocyanate

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Acyl isoselenocyanates were prepared by a reaction of acyl chloride with KSeCN. The acyl isoselenocyanates formed in situ were ready for further reaction without concentration. N-*Acyl selenoureas were obtained by a reaction of acyl isoselenocyanates with amines. The reaction of acyl isoselenocyanates with nucleophiles gave the corresponding selenocarbamate. All the compounds were well characterized by using spectral data, such as* ^{13}C *and* ^{77}Se *NMR and X-ray diffraction.*

Keywords Isoselenocyanate; selenourea; selenocarbamate

Acyl isoselenocyanates are useful intermediates for the preparation of selenium-containing heterocycles.[1] Acyl isoselenocyanates were prepared by a reaction of acyl chloride **1** with potassium selenocyanate, a method first investigated by Douglas.[2] The acyl isoselenocyanates were never isolated. It was assumed that a polymeric form was present in equilibrium with the monomer that underwent the observed reactions. A generation of the isoselenocyanates was confirmed by a reaction with nucleophiles. Douglas reported a reaction of the isoselenocyanates with amines in 1937. However, it included a limited number of products and lacked modern technology data, such as ^{13}C and ^{77}Se NMR and X-ray diffraction.[3] Although many studies of isothiocyanates

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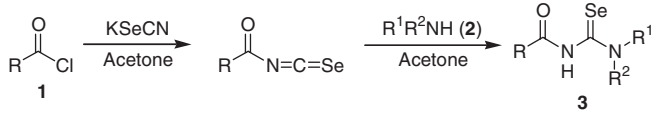
have been reported,[4] studies on isoselenocyanates have been limited.[5] In the present study, we describe reactions of acyl isoselenocyanates with amines or nucleophiles. The crystal structure of acylselenourea was investigated.

RESULTS AND DISCUSSION

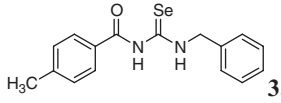
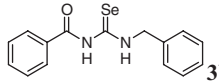
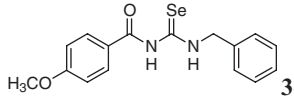
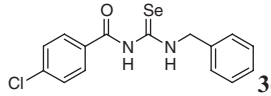
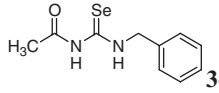
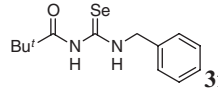
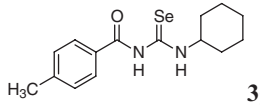
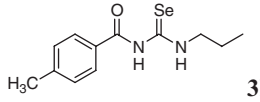
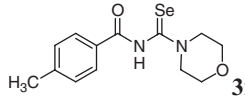
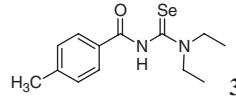
p-Toluoylisoselenocyanate was prepared by the reaction of *p*-toluoyl chloride **1a** with potassium selenocyanide in acetone in situ, to which benzylamine **2a** was added and stirred for 15 min. After the usual workup, *N*-benzyl-*N′*-*p*-toluoylselenourea **3a** was obtained quantitatively (Scheme 1). Various kinds of *N*-acylselenoureas were obtained in moderate to high yields (Table I). Reactions with primary amines (Table I, entries 1–8) and reactions with secondary amines (Table I, entries 9–10) gave **3** in nearly the same yields. In the case of reactions using aliphatic acyl chloride, i.e., methyl and butyl groups (Table I, entries 5–6), though the product **3e** and **3f** was apparently confirmed to be quantitatively formed in the reaction mixture from the result of TLC monitoring, **3e** and **3f** were obtained in low yields due to decomposition in the purification process. Typical spectroscopic properties of compounds **3** are summarized in Table I. The chemical shifts of amide carbonyl carbon (C=O) of compounds **3** in the ^{13}C NMR spectra are δ 165.3 ± 2.14 (aroyl acyl group; Table I, entries 1–4 and 7–10) and δ 175.2 ± 5.87 (acetyl and pivaloyl groups; Table I, entries 5–6). We can not distinguish between *N*-acylselenoureas **3a–3h** obtained by the reactions with primary amines (δ180.5 ± 0.99) and *N*-acylselenoureas **3i** and **3j** obtained by the reactions with secondary amines (δ180.7 ± 0.21) from the differences of the chemical shifts of C=Se in ^{13}C NMR spectra. ^{77}Se NMR was used to find the difference between the previously discussed compounds. Significant differences of the chemical shifts of selenium between **3a–3h** (δ337.9 ± 12.2) and **3i** and **3j** (δ469.8 ± 9.26) were clear in the ^{77}Se NMR spectra (Table I). ^{77}Se NMR spectra are one of the useful methods for structural determination.

The X-ray crystal structure of *N*-benzyl-*N′*-*p*-toluoylselenourea **3a** was studied. An ORTEP drawing, depicted in Figure 1, shows the molecular structure of **3a**. The crystal was built up of cetrosymmetric Se and



SCHEME 1

TABLE I The Preparation of *N*-Acylselenourea 3

Entry	Product (**3**)	Yield (%) (**3**)	C═O ^{13}C NMR	C═Se ^{13}C NMR	C═Se ^{77}Se NMR
1	 **3a**	quant.	166.7	181.0	344.1
2	 **3b**	78	166.8	181.0	351.0
3	 **3c**	71	166.2	181.0	338.2
4	 **3d**	63	165.7	180.9	356.9
5	 **3e**	trace[a]	171.0	180.6	326.4
6	 **3f**	19[a]	179.3	180.9	333.7
7	 **3g**	quant.	166.5	178.1	320.9
8	 **3h**	71	166.8	180.4	331.6
9	 **3i**	64	161.7	180.8	463.2
10	 **3j**	quant.	162.1	180.5	476.3

[a]A product was generated in a reaction mixture in situ quantitatively. However, it decomposed in the process of purification.

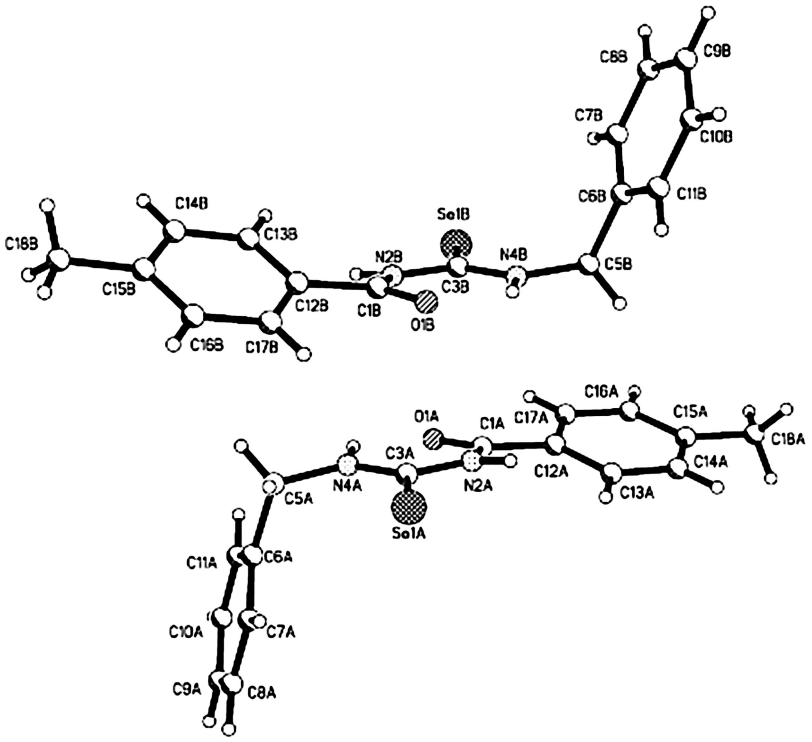
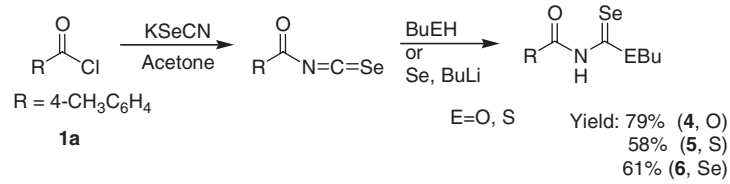


FIGURE 1 An ORTEP diagram (50% thermal ellipsoids) of compound **3a**.

H—N bonded dimmers.[6] The bond lengths of C1A—N2A (1.393(4) Å), N2A—C3A (1.381(4) Å), C3A—N4A (1.317(4) Å), C1B—N2B (1.377(4) Å), N2B—C3B (1.386(4) Å), and C3B—N4B (1.328(4) Å) in **3a** also were shorter than the usual value of 1.47 Å.[7] The sums of the three angles around the C1A, C3A, C1B, and C3B were 360.0°. The arrangement of O1, C1, N2, C3, Se1, and N4 atoms was almost planarowing to the result of the double bond character of the C1—N2, N2—C3, and C3—N4 bonds. These results can be attributed to the delocalization of the lone-pair electrons on N2 and N4 to the C1(=O1)—N2—C3(=Se1)—N4. Delocalization of the electron is consistent with an observation of an NH proton in a low field (δ11.5 for **3a**) in ^{1}H NMR.

Next, we investigated the reaction of *p*-toluoyl isoselenocyanates with nucleophiles. For example, an anhydrous acetone solution of in situ generated *p*-toluoyl isoselenocyanate was added to a solution of BuSe (5.0 equiv), which was freshly prepared by a reaction of BuSeLi with an ether solution of hydrochloric acid in



SCHEME 2

dry THF under an argon atmosphere. After workup, *Se*-butyl *N*-*p*-toluoyldiselenocarbamate **6** was obtained as orange needle crystals in a 61% yield (Scheme 2). Reactions with butanol or butanethiol also gave the corresponding *O*-butyl *N*-*p*-toluoylselenocarbamate **4** or *S*-butyl *N*-*p*-toluoylselenothiocarbamate **5** in 79% or 58% yields, respectively. Previously, the synthesis of *Te*-alkyl selenotellurocabamate was reported.[8] Present reactions with nucleophiles also proceeded similarly.

Acylselenoureas are used as useful precursors for the synthesis of heterocycles and *N*-selenocarbamoyl benzimidoyl chloride.[9] In this study, various kinds of acylselenoureas were prepared by reactions of acyl isoselenocyanates with amines. Also acyl isoselenocyanates were reacted with nucleophiles to give the corresponding acyl selenocabamate ester. The compounds were characterized by spectral data, such as ^{13}C and ^{77}Se NMR and X-ray diffraction.

EXPERIMENTAL

General

^{77}Se chemical shifts were expressed in ppm deshielded with respect to neat Me_2Se in $CDCl_3$.

The Synthesis of *N*-benzyl-*N′*-p-toluselenourea 3a

To a solution of KSeCN (0.28 g, 2 mmol) in acetone (5 mL) was added *p*-toluoyl chloride **1a** (0.13 mL, 1 mmol) in acetone (5 mL). The reaction mixture was stirred at 25°C for 10 min under an argon atmosphere. Benzylamine **2a** (0.22 mL, 2 mmol) in acetone (5 mL) was added to the mixture, and the mixture was stirred for 15 min. The reaction mixture was extracted with diethyl ether and washed with water. The organic layer was dried over sodium sulfate and evaporated to dryness. The residue was purified by flash column chromatography on silica gel using *n*-hexane:diethyl ether (5:1) as an eluent to give **3a** (0.35 g, quant). M.p.: 111.2–111.5°C, IR (KBr): 1669, 1503 cm^{-1}, ^1H NMR (500 MHz, $CDCl_3$): δ 2.43 (3H, s, CH_3), 4.96 (2H, d, $J = 5.2$ Hz,CH_2), 7.30–7.41 (4H, m, Ar),

7.72 (2H, d, $J = 8.6$ Hz, Ar), 9.37 (1H, br, NH), 11.5 (1H, br, NH), ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.6, 53.0, 127.5, 128.0, 128.1, 128.3, 128.9, 129.8, 135.5, 144.9, 166.7, 181.0,^{77}Se NMR (75 MHz, $CDCl_3$): δ 344.1, MS (CI): $m/z = 333$ [$M^+ + H$]. Anal. calcd. for $C_{16}H_{16}N_2OSe$: C, 58.01; H, 4.87; N, 8.46. Found: C, 58.14; H, 5.02; N, 8.09.

X-ray Crystallographic Data

Single crystals were grown from a diethyl ether-hexane Crystal system Orthorhombic; space group $Pna2_1$; T = 190(2) K; a = 19.0835(19) Å, b = 6.2335(6) Å, c = 24.888(2) Å, V = 2960.6(5) Å^3, $Z = 8$; $D_{calc} = 1.486$ g cm^{-3}; crystal size $0.24 \times 0.11 \times 0.11$ mm; θ range for data collection 3.3 to 27.5°, Limiting indices $-24 \leq h \leq 24$, $-6 \leq k \leq 8$, $-32 \leq l \leq 32$; reflections collected: 35037, independent reflections: 6764 refinement method: full-matrix least-squares on F^2, goodness of fit on F^2: 1.072, final R indices [$I > 2\sigma(I)$]$R1 = 0.0384$, wR2 = 0.0785, R indices (all data) $R1 = 0.0505$, $wR2 = 0.0835$, largest diff. peak and hole 1.777 and −0.415 e. Å^{-3}; selected bond lengths (Å) and angles (°), Se(1A)—C(3A): 1.851(3), C(1A)—O(1A): 1.217(4), C(1A)—N(2A): 1.393(4), C(1A)—C(12A): 1.490(5), N(2A)—C(3A): 1.381(4), C(3A)—N(4A): 1.317(4), N(4A)—C(5A): 1.460(4), C(5A)—C(6A): 1.525(5), Se(1B)—C(3B): 1.833(3), O(1B)—C(1B): 1.220(4), C(1B)—N(2B): 1.377(4), C(1B)—C(12B): 1.493(5), N(2B)—C(3B): 1.386(4), C(3B)—N(4B): 1.328(4), N(4B)—C(5B): 1.469(5), C(5B)—C(6B): 1.516(5), O(1A)—C(1A)—N(2A): 121.7(3), O(1A)—C(1A)—C(12A): 122.6(3), N(2A)—C(1A)—C(12A): 115.7(3), C(3A)—N(2A)—C(1A): 127.4(3), N(4A)—C(3A)—N(2A): 117.8(3), N(4A)—C(3A)—Se(1A): 124.9(2), N(2A)—C(3A)—Se(1A): 117.3(2), C(3A)—N(4A)—C(5A): 124.8(3), O(1B)—C(1B)—N(2B): 122.9(3), O(1B)—C(1B)—C(12B): 121.3(3), N(2B)—C(1B)—C(12B): 115.8(3), C(1B)—N(2B)—C(3B): 127.9(3), N(4B)—C(3B)—N(2B): 117.1(3), N(4B)—C(3B)—Se(1B): 125.5(3), N(2B)—C(3B)—Se(1B): 117.4(3), C(3B)—N(4B)—C(5B): 123.5(3) for all data.[6]

N-Benzoyl-*N*′-benzylselenourea 3b

M.p.: 114.8–115.2°C, IR (KBr): 1675, 1528 cm^{-1}, ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.95 (2H, s, CH_2), 7.30–7.43 (5H, m, Ar), 7.49 (2H, t, $J = 7.75$ Hz, Ar), 7.61 (1H, t, $J = 7.45$ Hz, Ar), 7.82 (2H, d, $J = 7.45$ Hz, Ar), 9.43 (1H, br, NH), 11.5 (1H, br, NH), ^{13}C NMR (125 MHz, $CDCl_3$): δ 52.9, 127.4, 127.9, 128.0, 128.8, 129.1, 133.7,166.8, 181.0, ^{77}Se NMR (75 MHz, $CDCl_3$): δ 351.0, MS (CI): $m/z = 319$ [$M^+ + H$]. Anal. calcd. for $C_{15}H_{14}N_2OSe$: C, 56.79; H, 4.45; N, 8.83. Found: C, 57.22; H, 4.62; N, 8.47.

N-Anisoyl-*N′*-benzylselenourea 3c

M.p.: 136.5–137.1°C, IR (KBr): 1600, 1500 cm^{-1}, ^1H NMR (500 MHz, $CDCl_3$): δ 3.88 (3H, s, CH_3O—Ph), 4.96 (2H, d, $J = 5.2$ Hz, CH_2), 6.97 (2H, d, $J = 8.6$ Hz, Ar), 7.30–7.42 (5H, m, Ar), 7.81 (2H, d, $J = 8.6$ Hz, Ar), 9.32 (1H, br, NH), 11.6 (1H, br, NH), ^{13}C NMR (100 MHz, $CDCl_3$): δ 53.0, 55.6, 114.4, 123.1, 127.9, 128.1, 128.8, 129.7, 135.5, 164.0, 166.2, 181.0, ^{77}Se NMR (75 MHz, $CDCl_3$): δ 338.2, MS (CI): $m/z = 349$ [$M^+ + H$].

N-Benzyl-*N′*-(4-chlorobenzoyl)selenourea 3d

M.p.: 112.0–112.9°C, IR (KBr):1674, 1560 cm^{-1}, ^1H NMR (500 MHz, $CDCl_3$): δ 4.95 (2H, d, $J = 5.8$ Hz, CH_2), 7.32–7.40 (5H, m, Ar), 7.49 (2H, d, $J = 8.6$ Hz, Ar), 7.78 (2H, d, $J = 8.6$ Hz, Ar), 9.43 (1H, br, NH), 11.6 (1H, br, NH), ^{13}C NMR (100 MHz, $CDCl_3$): δ 53.0, 128.0, 128.2, 128.9, 129.5, 135.4, 140.4, 165.7, 180.9, ^{77}Se NMR (75 MHz, $CDCl_3$): δ 356.9, MS (CI): $m/z = 351$ [$M^+ + H$].

N-Acetyl-*N′*-benzylselenourea 3e

IR (KBr): 1699, 1541 cm^{-1}, ^1H NMR (500 MHz, $CDCl_3$): δ 2.15 (3H, s, CH_3), 4.91 (2H, d, $J = 5.2$ Hz, CH_2), 7.32–7.39 (5H, m, Ar), 9.27 (1H, br, NH), 11.3 (1H, br, NH), ^{13}C NMR (125 MHz, $CDCl_3$): δ 24.3, 52.6, 127.9, 128.1, 128.9, 135.4, 171.0, 180.6, ^{77}Se NMR (95 MHz, $CDCl_3$): δ 326.4, MS (CI): $m/z = 257$ [$M^+ + H$].

N-Benzyl-*N′*-pivaloylselenourea 3f

M.p.: 111.2–112.0°C, IR (KBr): 1678, 1548 cm^{-1}, ^1H NMR (500 MHz, $CDCl_3$): δ 1.26 (9H, s, t-Bu), 4.88 (2H, d, $J = 5.2$ Hz, CH_2), 7.30–7.37 (5H, m, Ar), 8.81 (1H, br, NH), 11.4 (1H, br, NH), ^{13}C NMR (125 MHz, $CDCl_3$): δ 26.8, 39.7, 53.0, 128.0, 128.1, 128.9, 135.3, 179.3, 180.9, ^{77}Se NMR (95 MHz, $CDCl_3$): δ 333.7, MS (CI): $m/z = 298$ [$M^+ + H$]. Anal. calcd. for $C_{13}H_{18}N_2OSe$: C, 52.53; H, 6.10; N, 9.42. Found: C, 52.36; H, 6.10; N, 8.92.

N-Cyclohexyl-*N′*-*p*-toluoylselenourea 3g

M.p.: 112.9–113.4°C, IR (KBr):1666, 1521 cm^{-1}, ^1H NMR (500 MHz, $CDCl_3$): δ 1.23–1.80 (8H, m, CH), 2.05–2.19 (2H, m, CH), 2.43 (3H, s, CH_3), 4.34 (1H, m, CH), 7.29 (2H, d, $J = 8.1$ Hz, Ar), 7.73 (2H, d, $J = 8.1$ Hz, Ar), 9.33 (1H, br, NH), 11.3 (1H, br, NH), ^{13}C NMR (125

MHz, $CDCl_3$): δ 21.4, 24.0, 25.0, 57.0, 127.3, 128.2, 129.5, 144.3, 166.5, 178.1, ^{77}Se NMR (95 MHz, $CDCl_3$): δ 320.9, MS (CI): $m/z = 325$ [$M^+ + H$].

N-Propyl-*N'*-*p*-toluselenourea 3h

M.p.: 88.5–90.0°C, IR (KBr):1601, 1521 cm^{-1}, ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.04 (3H, t, $J = 7.5$ Hz, CH_3), 1.78 (2H, six, $J = 7.5$ Hz, CH_2), 2.43 (3H, s, CH_3), 3.71 (2H, q, $J = 6.9$ Hz, CH_3), 7.31 (2H, d, $J = 8.6$ Hz, Ar), 7.74 (2H, d, $J = 8.1$ Hz, Ar), 9.27 (1H, br, NH), 11.3 (1H, br, NH), ^{13}C NMR (125 MHz, $CDCl_3$): δ 11.4, 21.5, 21.6, 50.6, 127.5, 128.4, 129.8, 144.7, 166.8, 180.4, ^{77}Se NMR (95 MHz, $CDCl_3$): δ 331.6, MS (CI): $m/z = 284$ [$M^+ + H$].

N-(Morpholinoselenocarbonyl)-*p*-toluamide 3i

M.p.: 155.3–155.9°C, IR (KBr): 1666, 1525 cm^{-1}, ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.43 (3H, s, CH_3), 3.64 (2H, d, CH_2), 3.80 (2H, t, $J = 4.6$ Hz, CH_2), 3.90 (2H, t, $J = 4.9$ Hz, CH_2), 4.34 (2H, t, $J = 4.6$ Hz, CH_2), 7.29 (2H, d, $J = 8.0$ Hz, Ar), 7.74 (2H, d, $J = 8.6$ Hz, Ar), 8.60 (1H, br, NH), ^{13}C NMR (125 MHz, $CDCl_3$): δ 21.6, 53.6, 55.3, 66.0, 66.2, 127.9, 129.2, 129.7, 144.2, 161.7, 180.8, ^{77}Se NMR (95 MHz, $CDCl_3$): δ 463.2, MS (CI): $m/z = 313$ [$M^+ + H$].

N,N-Diethyl-*N'*-*p*-toluselenourea 3j

M.p.: 126.0–127.6°C, IR (KBr): 1641, 1527 cm^{-1}, ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.31 (3H, t, $J = 7.2$ Hz, CH_3), 1.42 (3H, t, $J = 6.9$ Hz, CH_3), 2.43 (3H, s, CH_3), 3.60 (2H, q, $J = 6.9$ Hz, CH_2), 4.15 (2H, q, $J = 6.9$ Hz, CH_2), 7.29 (2H, d, $J = 8.6$ Hz, Ar), 7.74 (2H, d, $J = 8.1$ Hz, Ar), 8.50 (1H, br, NH), ^{13}C NMR (125 MHz, $CDCl_3$): δ 11.7, 12.8, 21.5, 48.4, 51.2, 127.9, 129.5, 129.6, 143.8, 162.1, 180.5, ^{77}Se NMR (95 MHz, $CDCl_3$): δ 476.3, MS (CI): $m/z = 298$ [$M^+ + H$].

O-Butyl-N-p-toluselenocarbamate 4

To a solution of KSeCN (0.22 g, 1.3 mmol) in acetone (5 mL) was added *p*-toluoyl chloride (0.13 mL, 1 mmol) in acetone (5 mL), and the reaction mixture was stirred at 25°C for 90 min under an argon atmosphere. *n*-Butyl alcohol (0.46 mL, 5 mmol) was added to the mixture, and the mixture was refluxed under for 24 h. The reaction mixture was extracted with diethyl ether and washed with water. The organic layer was dried over sodium sulfate and evaporated to dryness. The residue was purified by flash column chromatography on silica gel using *n*-hexane:dichloromethane (1:1) as an eluent to give **4** (0.24 g, 79%).

M.p.: 62–63°C, IR (KBr): 3167, 1686 cm^{-1}, ^1H NMR (270 MHz, $CDCl_3$): δ 0.98 (3H, t, $J = 7.4$ Hz, CH_3), 1.52 (2H, six, $J = 7.4$ Hz, CH_2), 1.85 (2H, quintet, $J = 7.4$ Hz, CH_2), 2.42 (3H, s, CH_3), 4.72 (2H, q, $J = 7.4$ Hz, CH_2), 7.30 (2H, d, $J = 8.2$ Hz, Ar), 7.75 (2H, d, $J = 8.2$ Hz, Ar), 9.69 (1H, br, NH), ^{13}C NMR (68 MHz, $CDCl_3$): δ 13.6, 19.0, 21.6, 30.2, 78.0, 127.9, 129.7, 144.3, 161.4, 196.3.

S-Butyl-*N*-*p*-toluselenothiocarbamate 5

M.p.: 62–63°C, IR (KBr): 3182, 1696 cm^{-1}, ^1H NMR (270 MHz, $CDCl_3$): δ 0.98 (3H, t, $J = 7.3$ Hz, CH_3), 1.52 (2H, six, $J = 7.4$ Hz, CH_2), 1.82 (2H, quintet, $J = 7.4$ Hz, CH_2), 2.43 (3H, s, CH_3), 3.34 (2H, q, $J = 7.4$ Hz, CH_2), 7.32 (2H, d, $J = 8.4$ Hz, Ar), 7.81 (2H, d, $J = 8.4$ Hz, Ar), 10.6 (1H, br, NH), ^{13}C NMR (68 MHz, $CDCl_3$): δ 13.7, 21.7, 22.3, 29.1, 41.7, 127.8–144.6, 163.1, 207.7, Anal. calcd. for $C_{13}H_{17}NOSSe$: C, 49.67; H, 5.45. Found: C, 49.37; H, 5.58.

Se-Butyl-*N*-*p*-toludiselenocarbamate 6

To a solution of selenium (0.39 g, 5 mmol) in dry THF (5 mL) was added 1.67 M solution of *n*-BuLi in *n*-hexane (3 mL, 5 mmol) at 0°C under an argon atmosphere, and the mixture stirred for 15 min. After the mixture was cooled to −78°C, 1M of HCl solution in diethyl ether (6.5 mL, 6.5 mmol) was added, and the reaction mixture was stirred at −78°C for 15 min. To the resulting solution of *n*-BuSe the *p*-toluoyl isoselenocyanate suspension (1 mmol) was added at −78°C, and it was stirred for 1 h. After the reaction mixture was warmed to 25°C and stirred at the same temperature for 1.5 h. The reaction mixture was extracted with diethyl ether and washed with water. The organic layer was dried over sodium sulfate and evaporated to dryness. The residue was purified by flash column chromatography on silica gel using *n*-hexane:dichloromethane (1:1) as an eluent to give **6** (0.22 g, 61%). M.p.: 73–75°C, IR (KBr): 3134, 1693 cm^{-1}, ^1H NMR (270 MHz, $CDCl_3$): δ 0.98 (3H, t, $J = 7.4$ Hz, CH_3), 1.51 (2H, six, $J = 7.4$ Hz, CH_2), 1.85 (2H, quintet, $J = 7.4$ Hz, CH_2), 2.44 (3H, s, CH_3), 3.32 (2H, q, $J = 7.4$ Hz, CH_2), 7.33 (2H, d, $J = 8.3$ Hz, Ar), 7.83 (2H, d, $J = 8.3$ Hz, Ar), 10.96 (1H, br, NH), ^{13}C NMR (68 MHz, $CDCl_3$): δ 13.7, 21.7, 23.4, 29.7, 37.0, 127.8, 128.4, 130.0, 144.7, 163.8, 206.1, Anal. Calcd for $C_{13}H_{17}NOSe_2$: C, 43.21; H, 4.71. Found: C, 43.23; H, 4.67.

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