HETEROCYCLES, Vol. 68, No. 10, 2006, pp. 2145 - 2152. © The Japan Institute of Heterocyclic Chemistry
Received, 15th June, 2006, Accepted, 28th July, 2006, Published online, 1st August, 2006. COM-06-10813

PREPARATION OF 2-AMINO-1,3-SELENAZOLES BY REACTION OF *N*,*N*-UNSUBSTITUTED SELENOUREAS WITH α,β-UNSATURATED KETONES IN ALCOHOL

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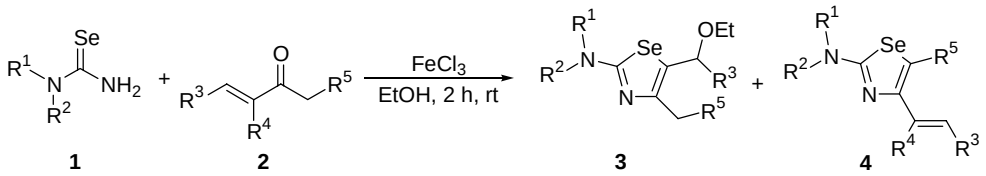
Abstract – 2-Dialkylamino-1,3-selenazoles were yielded by the reaction of *N*,*N*-unsubstituted selenoureas with α,β-unsaturated ketones in alcohol in the presence of ferric chloride at room temperature.

INTRODUCTION

There are many 1,3-selenazoles found in the literatures.[1] Many of them are studied as potential pharmaceutical and dye agents.[2] The use of selenoureas as the most efficient starting materials for the synthesis of 1,3-selenazoles containing selenium-nitrogen has been reported.[3] For the synthesis of 1,3-selenazole derivatives using selenoureas, several methods have been developed. For example, reactions of selenourea with α-haloketones,[4] chloroacetonitrile[5] and α-haloacyl halides[6] afforded 1,3-selenazole derivatives. Most of methods include the use of lachrymatory halo carbonyl compounds. Recently we have reported a new route to 1,3-selenazoles by reactions of *N*,*N*-unsubstituted selenoureas with ketones in the presence of ferric chloride without use of lachrymatory halo carbonyl compounds.[7] In continuation of our studies of the reactions, we have confirmed that *N*,*N*-unsubstituted selenoureas reacted with α,β-unsaturated ketones in alcohol to give 2-amino-1,3-selenazole derivatives. We describe here the syntheses of 2-dialkylamino-1,3-selenazole derivatives by the reaction of *N*,*N*-unsubstituted selenoureas with α,β-unsaturated ketones in alcohol.

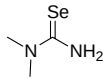
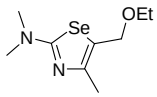
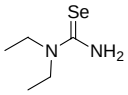
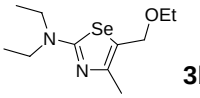
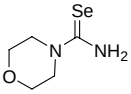
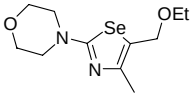
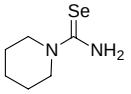
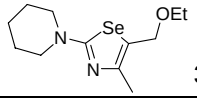
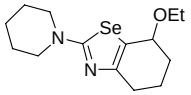
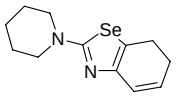
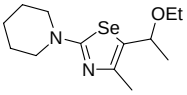
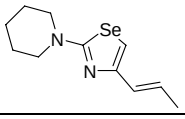
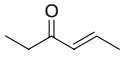
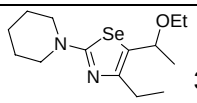
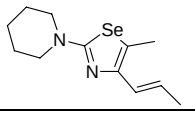
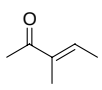
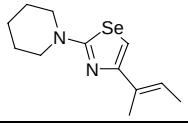
RESULTS AND DISCUSSION



1 2 3 4

Scheme 1

Table 1. Synthesis of 2-Amino-1,3-selenazoles (**3**)

Entry	Selenourea (**1**)	α,β-Unsaturated Ketone (**2**)	2-Amino-1,3-selenazole (**3** or **4**)	
			Product	Yield (%)[a]
1	 **1a**	 **2a**	 **3a**	83
2	 **1b**	**2a**	 **3b**	77
3	 **1c**	**2a**	 **3c**	80
4	 **1d**	**2a**	 **3d**	89
5	**1d**	 **2b**	 **3e**	34
			 **4e**	30
6	**1d**	 **2c**	 **3f**	53
			 **4f**	34
7	**1d**	 **2d**	 **3g**	26
			 **4g**	50
8	**1d**	 **2e**	 **4h**	24

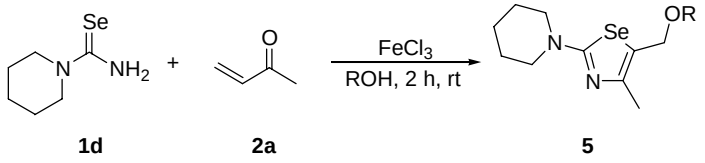
[a] Isolated yield.

Optimal conditions for the reaction of 1-selenocarbamoylpiperidine (**1d**) with methyl vinyl ketone (**2a**) were studied. The previously reported reactions of *N*,*N*-unsubstituted selenoureas with ketones in the

presence of ferric chloride were carried out under reflux conditions affording 2-amino-1,3-selnazoles in high yields.[7] The reactions of **1d** with **2a** in the presence of ferric chloride under reflux conditions in ethanol gave unidentifiable mixtures, whilst the reaction at room temperature afforded 5-(1-ethoxymethyl)-4-methyl-2-piperidino-1,3-selenazole (**3d**) in 89% yield (Scheme 1). As reaction solvent, ethanol, dichloromethane and THF were used. The reaction using ethanol gave exclusively **3d** in the highest yields. The structure of **3d** was elucidated by studies of IR, ^{1}H-, ^{13}C-, ^{77}Se-NMR, HMQC, HMBC and elemental analysis. In the HMBC spectra of **3d**, H4 methyl signals have cross peaks with C4 and C5 quaternary carbons. Signal of CH_2 at C5 has cross peaks with CH_2 of ethoxy, C4 and C5 quaternary carbons. These spectra indicates that the structure of **3d** is 5-(1-ethoxymethyl)-4-methyl-2-piperidino-1,3-selenazole. The structures of products (**3a**-**3g**) were determined by comparing the spectral data with those of **3d**.

Reactions of *N*,*N*-unsubstituted selenoureas (**1a**-**1d**) with methyl vinyl ketone (**2a**) gave the corresponding 2-amino-5-(1-ethoxymethyl)-1,3-selenazole (**3a**-**3d**) in 77-89% yields (Table 1, Entries 1-4). Using the optimal reaction conditions, several 2-amino-1,3-selenazole derivatives (**3a**-**3g** and **4e**-**4h**) were prepared from the reactions of corresponding *N*,*N*-unsubstituted selenoureas (**1a**-**1d**) with α,β-unsaturated ketones (**2a**-**2e**) in the presence of ferric chloride at room temperature in ethanol solvent. The reaction gave two kinds of selenazole derivatives, 2-amino-5-(1-ethoxyalkyl)-1,3-selenazoles (**3**) and 2-amino-1,3-selenazoles (**4**), in moderate to high yields in the present study. Selenoureas (**1**) reacted with α,β-unsaturated ketones (**2b**-**2d**) at both α position of carbonyl carbon to give two kinds of products in a certain ratio (Entries 5-7).

Reactions in other alcohols were carried out. Reactions of **1d** with **2a** in methanol, *n*-propanol, isopropyl alcohol and *tert*-butyl alcohol also gave corresponding 2-amino-5-(1-alkoxymethyl)-1,3-selenazole derivatives (**3**) in moderate to high yields. The yields of the products were higher, when the reaction was carried out in primary alcohol and decreased when secondary and tertiary alcohols were used as solvent. Reaction in phenol gave only unidentifiable mixtures instead of **5**.



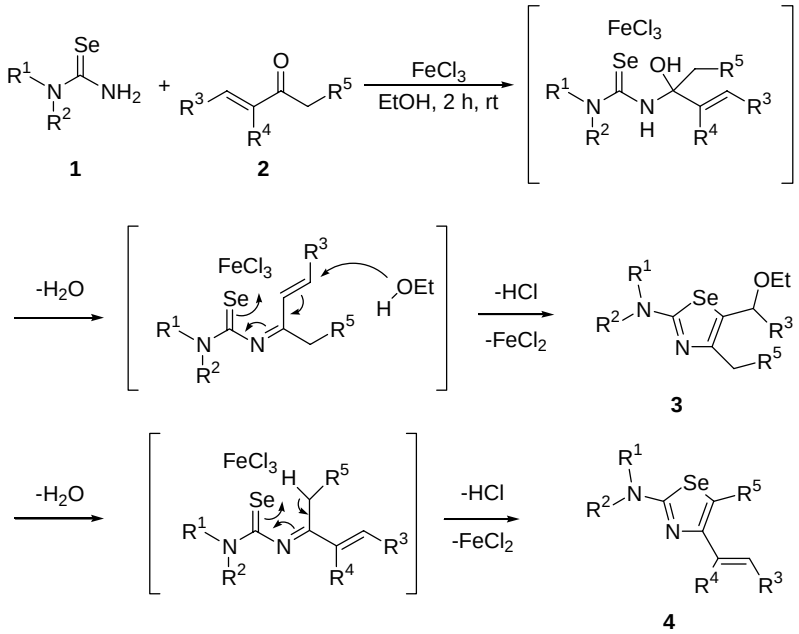
1d **2a** **5**

R	Yield (%)
CH_3	80 (**5a**)
$CH_3(CH_2)_2$	83 (**5b**)
$(CH_3)_2CH$	72 (**5c**)
$(CH_3)_3C$	41 (**5d**)

Scheme 2

The formations of **3** and **4** could be explained by the following mechanism; the reaction of *N*,*N*-unsubstituted selenourea **1** with α,β-unsaturated ketone (**2**) is initiated by the nucleophilic addition of

the nitrogen of the selenourea to the carbonyl carbon, affording 2-amino-1,3-selenazoles **3** and **4** (Scheme 3).



Scheme 3

In the present study, it was confirmed that the reactions of *N*,*N*-unsubstituted selenoureas (**1**) with α,β-unsaturated ketones (**2**) in alcohol in the presence of ferric chloride give various type of 2-dialkylamino-1,3-selenazole derivatives (**3**) at room temperature.

EXPERIMENTAL

General

Selenoureas were synthesized according to previously described procedures.[8] The ^{77}Se chemical shifts are expressed in ppm deshielded with respect to Me_2Se in $CDCl_3$. *J* (^{77}Se-^{1}H) values and ^{1}J (^{77}Se-^{13}C) values are the ^{77}Se satellites of the ^{1}H NMR spectra and proton-decoupled ^{13}C NMR spectra, respectively.

General procedure for synthesis of 2-dimethylamino-5-ethoxymethyl-4-methyl-1,3-selenazole (3a). Methyl vinyl ketone (**2a**) (0.13 ml, 1.5 mmol) was added to stirred solution of *N*,*N*-dimethylselenourea (**1a**) (75 mg, 0.5 mmol) in dry ethanol (5 mL) under an argon atmosphere. Ferric chloride (0.29 g, 1.8 mmol) was added into the reaction mixture. The reaction mixture was stirred for 2 h under room temperature. The mixture was diluted with ethyl acetate and saturated Na_2CO_3 aq. The organic layer was separated, dried over sodium sulfate and evaporated to dryness. The residue was purified by flash chromatography on silica gel with diethyl ether:hexane (1:3) to give **3a** (102 mg, 83 %) as green liquid.

IR (neat): 2866, 1557 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.21 (3H, t, *J* =6.9 Hz, CH_3), 2.18 (3H, s, CH_3), 3.05 (6H, s, CH_3), 3.51 (2H, q, *J* =6.9 Hz, CH_2), 4.51 (2H, s, CH_2) [^{3}J (^{77}Se-^{1}H) = 10.9 Hz]; ^{13}C NMR (125 MHz, $CDCl_3$): δ 15.1, 15.6, 40.8, 64.4, 66.0, 121.2, 147.4, 171.3; ^{77}Se NMR (95 MHz, $CDCl_3$): δ 562.8.

2-Diethylamino-5-ethoxymethyl-4-methyl-1,3-selenazole (3b) Yellow liquid. IR (neat): 2972, 1541 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.21 (9H, t, *J* =6.9 Hz, CH_3), 2.16 (3H, s, CH_3), 3.42 (4H, q, *J* =6.9 Hz, CH_2), 3.51 (2H, q, *J* =6.9 Hz, CH_2), 4.51 (2H, s, CH_2) [^{3}J (^{77}Se-^{1}H) = 10.9 Hz]; ^{13}C NMR (125 MHz, $CDCl_3$): δ 12.5, 15.1, 15.7, 46.0, 64.5, 66.1, 119.7, 147.4, 169.5; ^{77}Se NMR (95 MHz, $CDCl_3$): δ 559.7; Anal. Calcd for $C_{11}H_{20}N_2OSe$: C, 48.00; H, 7.32; N, 10.18. Found: C, 48.01; H, 7.20; N, 9.85.

5-Ethoxymethyl-4-methyl-2-morpholino-1,3-selenazole (3c) Yellow solid. mp 34–35 °C; IR (KBr): 2856, 1530 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.21 (3H, t, *J* =6.9 Hz, CH_3), 2.17 (3H, s, CH_3), 3.40 (4H, t, *J* =5.1 Hz, CH_2), 3.51 (2H, q, *J* =6.9 Hz, CH_2), 3.77 (4H, t, *J* =5.1 Hz, CH_2), 4.51 (2H, s, CH_2) [^{3}J (^{77}Se-^{1}H) = 10.3 Hz]; ^{13}C NMR (125 MHz, $CDCl_3$): δ 15.0, 15.6, 49.2, 64.6, 65.9, 66.0, 122.5, 146.9, 172.1; ^{77}Se NMR (95 MHz, $CDCl_3$): δ 572.8; Anal. Calcd for $C_{11}H_{18}N_2O_2Se$: C, 45.68; H, 6.27; N, 9.69. Found: C, 45.89; H, 6.27; N, 9.29.

5-Ethoxymethyl-4-methyl-2-piperidino-1,3-selenazole (3d) Orange liquid. IR (neat): 2934, 1532 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.21 (3H, t, *J* =6.9 Hz, CH_3), 1.60-1.67 (6H, m, CH_2), 2.16 (3H, s, CH_3), 3.38-3.42 (4H, m, CH_2) 3.50 (2H, q, *J* =6.9 Hz, CH_2), 4.51 (2H, s, CH_2) [^{3}J (^{77}Se-^{1}H) = 10.3 Hz]; ^{13}C NMR (125 MHz, $CDCl_3$): δ 15.1, 15.7, 24.1, 25.1, 50.4, 64.5, 66.0, 120.9 [^{1}J (^{77}Se-^{13}C) = 90.3 Hz], 147.2, 172.0; ^{77}Se NMR (95 MHz, $CDCl_3$): δ 565.8; Anal. Calcd for $C_{12}H_{20}N_2OSe$: C, 50.17; H, 7.02; N, 9.75. Found: C, 50.34; H, 6.78; N, 9.51.

8-Ethoxy-2-piperidino-4,7,8,9-tetrahydrobenzo-1,3-selenazole (3e) Yellow solid. mp 45-46 ºC IR (neat): 2928, 1527 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.15 (3H, t, *J* =6.9 Hz, CH_3), 1.53-1.60 (6H, m, CH_2), 1.60-1.68 (1H, m, CH_2), 1.75-1.83 (1H, m, CH_2), 1.83-1.94 (2H, m, CH_2), 2.38-2.44 (1H, m, CH_2), 2.49-2.55 (1H, m, CH_2), 3.29-3.38 (4H, m, CH_2) 3.38-3.57 (2H, m, CH_2) 4.42 (1H, s, CH) [^{3}J (^{77}Se-^{1}H) = 9.7 Hz]; ^{13}C NMR (125 MHz, $CDCl_3$): δ 15.7, 19.6, 24.2, 25.2, 28.0, 29.5, 50.7, 63.4, 73.7, 122.3, 150.1, 172.9; ^{77}Se NMR (95 MHz, $CDCl_3$): δ 548.7; Anal. Calcd for $C_{14}H_{22}N_2OSe$: C, 53.67; H, 7.08; N, 8.94. Found; C, 53.57; H, 6.97; N, 8.70.

2-Piperidino-6,7-dihydrobenzo-1,3-selenazole (4e) Yellow liquid. IR (neat): 2933, 1522 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.59-1.68(6H, m, CH_2), 2.33-2.39 (2H, m, CH_2), 2.82 (2H, t, *J* =9.7 Hz, CH_2), 3.36-3.42 (4H, m, CH_2), 5.80-5.84 (1H, m, CH), 6.36-6.40 (1H, m, CH); ^{13}C NMR (125 MHz, $CDCl_3$): δ 23.4, 23.9, 24.3, 25.2, 50.7, 120.6, 123.8, 125.1, 146.5, 171.8; ^{77}Se NMR (95 MHz, $CDCl_3$): δ 561.4; Anal. Calcd for $C_{12}H_{16}N_2Se$: C, 53.93; H, 6.03; N, 10.48. Found: C, 54.03; H, 6.20; N, 10.11.

5-(1-Ethoxyethyl)-4-methyl-2-piperidino-1,3-selenazole (3f) Orange solid. mp 39-40 ºC IR (KBr): 2934, 1534 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 1.17 (3H, t, *J* =6.9 Hz, CH_3), 1.43 (3H, d, *J* =6.3 Hz, CH_3), 1.60-1.68 (6H, m, CH_2), 2.14 (3H, s, CH_3), 3.31-3.43 (5H, m, CH_2) 3.48-3.53 (1H, m, CH_2), 4.54 (1H, q, *J* =6.3 Hz, CH); ^{13}C NMR (125 MHz, $CDCl_3$): δ 15.3, 16.0, 24.2, 25.1, 25.2, 50.3, 63.2, 72.1, 128.9, 145.2, 171.4; ^{77}Se NMR (95 MHz, $CDCl_3$): δ 538.0; Anal. Calcd for $C_{13}H_{22}N_2OSe$: C, 51.82; H, 7.36; N, 9.30. Found: C, 52.15; H, 7.24; N, 9.06.

2-Piperidino-4-(1-propenyl)-1,3-selenazole (4f) Orange liquid. IR (neat): 2934, 1543 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 1.59-1.70 (6H, m, CH_2), 1.82 (3H, dd, *J* =1.7, 6.9 Hz, CH_3), 3.40-3.48 (4H, m, CH_2), 6.15 (1H, dd, *J* =1.7, 15.5 Hz, CH), 6.40-6.47 (1H, m, CH), 6.72 (1H, s, CH) [^2J (^{77}Se-^1H) = 50.4 Hz]; ^{13}C NMR (125 MHz, $CDCl_3$): δ 18.0, 24.3, 25.3, 50.6, 106.2, 126.1, 127.6, 152.2, 172.7; ^{77}Se NMR (95 MHz, $CDCl_3$): δ 554.9; Anal. Calcd for $C_{11}H_{16}N_2Se$: C, 51.77; H, 6.32; N, 10.98. Found: C, 52.17; H, 6.47; N, 10.60.

5-(1-Ethoxyethyl)-4-ethyl-2-piperidino-1,3-selenazole (3g) Yellow solid. mp 36-37 ºC IR (KBr): 2933, 1532 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 1.17 (3H, q, *J* =6.9 Hz, CH_3), 1.18 (3H, q, *J* =6.9 Hz, CH_3), 1.44 (3H, d, *J* =6.3 Hz, CH_3), 1.60-1.68 (6H, m, CH_2), 2.42-2.53 (2H, m, CH_2), 3.30-3.42 (5H, m, CH_2), 3.48-3.56 (1H, m, CH_2), 4.56 (1H, q, *J* =6.3 Hz, CH); ^{13}C NMR (125 MHz, $CDCl_3$): δ 14.3, 15.3, 23.7, 24.2, 25.3, 25.6, 50.4, 63.2, 71.9, 128.8, 151.5, 171.6; ^{77}Se NMR (95 MHz, $CDCl_3$): δ 538.3; Anal. Calcd for $C_{14}H_{24}N_2OSe$: C, 53.33; H, 7.67; N, 8.88. Found: C, 53.69; H, 7.63; N, 8.80.

5-Methyl-2-piperidino-4-(1-propenyl)-1,3-selenazole (4g) Yellow solid. mp 36-37 ºC IR (KBr): 2933, 1552 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 1.60-1.66 (6H, m, CH_2), 1.84 (3H, dd, *J* =1.2, 6.9 Hz, CH_3), 2.35 (3H, s, CH_3) [^3J (^{77}Se-^1H) = 10.3 Hz], 3.37-3.40 (4H, m, CH_2), 6.23 (1H, dd, *J* =1.2, 15.2 Hz, CH), 6.42 (1H, sixtet, *J* =6.9 Hz, CH); ^{13}C NMR (125 MHz, $CDCl_3$): δ 12.7, 18.3, 24.3, 25.3, 25.6, 50.4, 121.3, 122.7, 127.2, 146.3, 169.2; ^{77}Se NMR (95 MHz, $CDCl_3$): δ 595.7; Anal. Calcd for $C_{12}H_{18}N_2Se$: C, 53.53; H, 6.74; N, 10.40. Found: C, 53.93; H, 6.76; N, 10.26.

2-Piperidino-4-(1-methyl-1-propenyl)-1,3-selenazole (4h) Pink solid. mp 94-95 ºC IR (KBr): 2931, 1542 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 1.61-1.68 (6H, m, CH_2), 1.77 (3H, d, *J* =6.9 Hz, CH_3), 1.92 (3H, s, CH_3), 3.40-3.46 (4H, m, CH_2), 6.58 (1H, qd, *J* =1.2, 6.9 Hz, CH), 6.80 (1H, s, CH) [^2J (^{77}Se-^1H) = 51.6 Hz]; ^{13}C NMR (125 MHz, $CDCl_3$): δ 13.3, 13.8, 24.3, 25.3, 50.6, 103.7, 124.0, 130.5, 155.4, 171.6; ^{77}Se NMR (95 MHz, $CDCl_3$): δ 564.4; Anal. Calcd for $C_{12}H_{18}N_2Se$: C, 53.53; H, 6.74; N, 10.40. Found: C, 53.64; H, 6.80; N, 10.08

5-(1-Methoxyethyl)-4-methyl-2-piperidino-1,3-selenazole (5a) Yellow liquid. IR (neat): 2932, 1532 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 1.61-1.68 (6H, m, CH_2), 2.17 (3H, s, CH_3), 3.33 (3H, s, CH_3), 3.38-3.43 (4H, m, CH_2), 4.46 (2H, s, CH_2) [^3J (^{77}Se-^1H) = 10.3 Hz]; ^{13}C NMR (125 MHz, $CDCl_3$): δ 15.7,

24.1, 25.1, 50.4, 56.9, 67.9, 120.2 [^{1}J (^{77}Se-^{13}C) = 91.2 Hz], 147.6, 172.0; ^{77}Se NMR (95 MHz, $CDCl_3$): δ 564.5; Anal. Calcd for $C_{11}H_{18}N_2OSe$: C, 48.35; H, 6.64; N, 10.25. Found: C, 47.96; H, 6.52; N, 10.00.

4-Methyl-2-piperidino-5-propoxymethyl-1,3-selenazole (5b) Yellow liquid. IR (neat): 2935, 1540 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 0.92 (3H, t, J =7.4 Hz, CH_3), 1.56-1.67 (8H, m, J =7.4 Hz, CH_2), 2.16 (3H, s, CH_3), 3.36-3.42 (4H, m, CH_2), 4.51 (2H, s, CH_2) [^{3}J (^{77}Se-^{1}H) = 10.9 Hz]; ^{13}C NMR (125 MHz, $CDCl_3$): δ 10.5, 15.7, 22.8, 24.1, 25.1, 50.3, 66.1, 70.8, 121.0 [^{1}J (^{77}Se-^{13}C) = 91.2 Hz], 147.0, 171.9; ^{77}Se NMR (95 MHz, $CDCl_3$): δ 566.0; Anal. Calcd for $C_{13}H_{22}N_2OSe$: C, 51.82; H, 7.36; N, 9.30. Found: C, 51.87; H, 7.41; N, 9.37.

4-Isopropoxymethyl-4-methyl-2-piperidino-1,3-selenazole (5c) Orange liquid. IR (neat): 2933, 1532 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.17 (6H, d, J = 6.3 Hz, CH_3), 1.60-1.67 (6H, m, CH_2), 2.15 (3H, s, CH_3), 3.36-3.42 (4H, m, CH_2), 3.69 (1H, seventet, J = 6.3 Hz, CH), 4.51 (2H, s, CH_2) [^{3}J (^{77}Se-^{1}H) = 10.3 Hz]; ^{13}C NMR (125 MHz, $CDCl_3$): δ 15.7, 21.9, 24.1, 25.1, 50.3, 63.5, 69.4, 121.6, 146.5, 171.8; ^{77}Se NMR (95 MHz, $CDCl_3$): δ 566.9; Anal. Calcd for $C_{13}H_{22}N_2OSe$: C, 51.82; H, 7.36; N, 9.30. Found: C, 51.75; H, 7.25; N, 9.10.

5-*tert*-Butoxymethyl-4-methyl-2-piperidino-1,3-selenazole (5d) Yellow liquid. IR (neat): 2935, 1532 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.26 (9H, s, CH_3), 1.60-1.66 (6H, m, CH_2), 2.14 (3H, s, CH_3), 3.34-3.42 (4H, m, CH_2), 4.43 (2H, s, CH_2) [^{3}J (^{77}Se-^{1}H) = 9.7 Hz]; ^{13}C NMR (125 MHz, $CDCl_3$): δ 15.8, 24.2, 25.1, 27.6, 50.4, 58.4, 73.3, 122.5, 145.4, 171.9; ^{77}Se NMR (95 MHz, $CDCl_3$): δ 567.5; Anal. Calcd for $C_{14}H_{24}N_2OSe$: C, 53.33; H, 7.67; N, 8.88. Found: C, 53.68; H, 7.78; N, 8.78.

ACKNOWLEDGMENTS

This work was supported by a Grant-in-Aid for Science Research from the Ministry of Education, Culture, Sports, Science and Technology of Japan (No. 15550030 and 17550099) to which we are grateful.

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