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Syntheses of Cyanoselenoamides and Diselenoamides: Conversion into Selenazoles and Selenazines

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ABSTRACT: *Reactions of dicyano compounds with a potassium selenocarboxylate afforded the corresponding cyanoselenoamides and diselenoamides in good yields of experimentally determined ratios. The obtained cyanoselenoamides were allowed to react with potassium selenocarboxylate, again to afford the corresponding diselenoamides in higher yields. The cyanoselenoamides and diselenoamides were investigated as substrates for preparations of selenazoles and selenazines.* © 2003 Wiley Periodicals, Inc. Heteroatom Chem 14:106–110, 2003; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.10111

INTRODUCTION

Many selenocarbonyl compounds have been prepared and recent reviews detail these studies [1]. Selenoamides have been used as precursors for the synthesis of both selenium- and nitrogen-containing heterocyclic compounds [2]. Generally, the preparation of primary selenoamides has been carried out by the reaction of nitriles with appropriate selenating reagents [3]. Diselenoamides are also important for the synthesis of both selenium- and nitrogen-containing heterocyclic compounds. However, there currently exists only one practical synthesis of diselenoamides [4]. In the present study, we investigated the preparation of cyanoselenoamides and diselenoamides and the syntheses of selenazoles and selenazines, using the cyanoselenoamides and diselenoamides.

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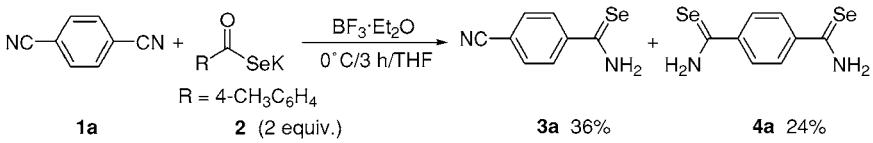
RESULTS AND DISCUSSION

Optimal conditions for the preparation of cyanoselenoamides **3** and diselenoamides **4** from the corresponding dicyano compounds **1** were determined by trial and error. The best conditions for the synthesis of **4** were found to be as shown in Scheme 1. The reactions at room temperature and −30°C gave **3** of higher yield and **4** of lower yield than under the conditions shown in Scheme 1. Results of reactions using various dicyano compounds **1** are summarized in Table 1. The reaction of **1c** gave the corresponding diselenoamide **4c** in very low yield because of steric hindrance. The reactions of **1d** and **1e** gave neither the cyanoselenoamides **3** nor the diselenoamides **4**.

Isolated **3a** was allowed to react with **2** again under similar conditions for the preparation of **4a** (Table 2), the best result being obtained by the use 2 equiv. of **2** and a reaction of 3 h. **4a–c** were synthesized using the best conditions (Table 2) (Scheme 2).

Reactions of **3b** and **4b** with four kinds of carbonyl compounds, i.e., an α-haloacyl halide, a bisacyl chloride, an α-halo ketone, and an α,β-unsaturated ketone, were attempted for syntheses of the selenazole and selenazine, respectively, according to the previously reported procedures with mono primary selenoamide (Scheme 3) [2a,c,d,4]. When **3b** reacted with chloroacetyl chloride, malonyl chloride, and phenacyl bromide, the corresponding



SCHEME 1

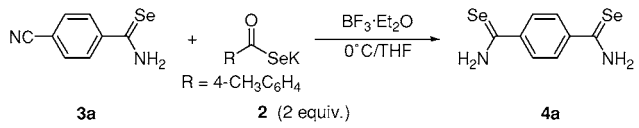
selenazol-4-one (**5**), selenazin-4-one (**6**), and selenazole (**7**), respectively, were formed. However, reaction with methyl vinyl ketone gave no identifiable products other than the starting material and complicated mixtures of the reactions of **4b** with the carbonyl compounds, only that with phenacyl bromide gave the corresponding diselenazole **8**. The other reactions of **4b** afforded no identifiable products owing to the instability and insolubility of **4b** in organic solvents. Methanol and ethanol, which dissolve **4b** could not be used in the reaction with an acyl chloride.

TABLE 1 Syntheses of Cyanoselenoamides and Diselenoamides

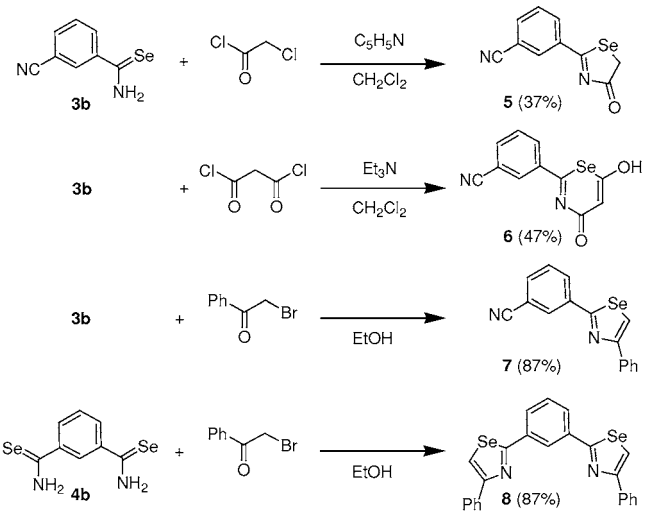
1 *or* **3**	**3**		**4**	
1a	**3a**	36%	**4a**	24%
1b	**3b**	49%	**4b**	24%
1c	**3c**	49%	**4c**	5%
1d		0%		0%
1e		0%		0%
1f		0%	**4f**	45%
1g	**3g**	27%	**4g**	23%
1h	**3h**	37%	**4h**	8%
1i	**3i**	41%	**4i**	7%
1j	**3j**	33%	**4j**	10%
3a	**3a**	17%	**4a**	79%
3b	**3b**	29%	**4b**	68%
3c	**3c**	74%	**4c**	23%

TABLE 2 Investigations of Optimal Conditions for Synthesis of Diselenoamide **4a** from Cyanoselenoamide **3a**

2 *(equiv.)*	*Time (h)*	*Recovery of* **3a** *(%)*	*Yield of* **4a** *(%)*
1	3	66	27
1.5	3	42	55
2	3	17	79
3	3	19	78
2	1	21	74
2	5	24	72



SCHEME 2



SCHEME 3

EXPERIMENTAL

General

Melting points were determined by use of a Yanagimoto micromelting point apparatus and are

uncorrected. IR spectra were measured on a Perkin-Elmer 1600 spectrometer. ^{1}H, ^{13}C, and ^{77}Se NMR spectra were recorded on a JEOL-JNM-α400 spectrometer. Mass spectra were obtained on a Shimadzu 9020-DF mass spectrometer. Tetrahydrofuran was distilled from sodium-benzophenone and immediately used. The ^{77}Se chemical shifts are expressed in ppm, deshielded, with respect to Me_2Se in $CDCl_3$.

*Typical Procedure for 4-Cyanobenzoselenoamide (***3a***) and 1,4-Benzenediselenocarboxamide (***4a***).* Terephtalonitrile (0.64 g, 5.0 mmol) and boron trifluoride ether complex (3.62 g, 12 mmol) were added to the THF solution (30 ml) of potassium 4-methylselenobenzoate (2.37 g, 10 mmol) under an argon atmosphere at 0°C. The reaction mixture was stirred at 0°C for 3 h. The mixture was extracted with ethyl acetate and washed with water. The organic layer was dried over sodium sulfate and evaporated to dryness. The residue was purified by flash chromatography on silica gel with dichloromethane/diethyl ether (10:1) as eluent to give **3a** (0.38 g; yield 36%) as a white powder and **4a** (0.35 g; yield 24%) of 4-Cyanobenzoselenoamide (**3a**), an orange solid; mp 151.2–152.6°C; IR (KBr) 3348, 3181, 2240, 1624 cm^{-1}; ^{1}H NMR (400 MHz, acetone-d_6): δ 7.79 (d, $J = 8.8$ Hz, 2H, Ar), 8.07 (d, $J = 8.8$ Hz, 2H, Ar), 9.78 (br s, 1H, NH), 10.26 (br s, 1H, NH); ^{13}C NMR (100 MHz, acetone-d_6): δ 114.7, 118.9, 128.4, 132.7, 147.4, 205.6; ^{77}Se NMR (76 MHz, acetone-d_6): δ 707.1; MS (CI) $m/z = 211$ [M$^+$ + 1]; Anal calcd for $C_8H_6N_2Se$: C, 45.95; H, 2.89; N, 13.40. Found: C, 45.98; H, 2.99; N, 13.34. 1,4-Benzenediselenocarboxamide (**4a**) Brown solid; mp 185.0–187.0°C; IR (KBr) 3266, 3111, 1630 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.84 (s, 4H, Ar), 10.32 (br s, 2H, NH), 10.94 (br s, 2H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 126.8, 144.0, 203.0; ^{77}Se NMR (76 MHz, DMSO-d_6): δ 611.8; MS (CI) $m/z = 293$ [M$^+$ + 1].

*3-Cyanobenzeselenocarboxamide (***3b***).* A red solid; mp 132.4–133.6°C; IR (KBr) 3352, 3123, 2234, 1649 cm^{-1}; ^{1}H NMR (400 MHz, acetone-d_6): δ 7.65 (t, $J = 7.8$ Hz, 1H, Ar), 7.94 (d, $J = 7.8$ Hz, 1H, Ar), 8.26–8.31 (m, 2H, Ar), 9.79 (br s, 1H, NH), 10.25 (br s, 1H, NH); ^{13}C NMR (100 MHz, acetone-d_6): δ 112.9, 118.6, 130.2, 131.2, 132.4, 134.9, 144.8, 204.9; ^{77}Se NMR (76 MHz, acetone-d_6): δ 683.2; MS (EI) $m/z = 210$ [M$^+$]; Anal calcd for $C_8H_6N_2Se$: C, 45.95; H, 2.89; N, 13.40. Found: C, 46.11; H, 3.04; N, 13.25.

*1,3-Benzenediselenocarboxamide (***4b***).* Orange solid; mp 155.6–156.8°C; IR (KBr) 3267, 3134, 1633 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.43 (t, $J = 7.8$ Hz, 1H, Ar), 7.96 (dd, $J = 2.0$, 7.8 Hz, 2H, Ar), 8.27 (s, 1H, Ar), 10.29 (br s, 2H, NH), 10.90 (br s, 2H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 125.8, 127.7, 129.6, 142.4, 203.4; ^{77}Se NMR (76 MHz, DMSO-d_6): δ 599.7; MS (CI) $m/z = 293$ [M$^+$ + 1].

*3-Cyano-2-methylbenzoselenoamide (***3c***).* Yellow solid; mp 145.0–146.0°C; IR (KBr) 3319, 3128, 2226, 1638 cm^{-1}; ^{1}H NMR (400 MHz, acetone-d_6): δ 2.56 (s, 3H, CH_3), 7.39 (t, $J = 7.8$ Hz, 1H, Ar), 7.59 (d, $J = 7.8$ Hz, 1H, Ar), 7.68 (d, $J = 7.8$ Hz, 1H, Ar), 9.70 (br s, 1H, NH), 10.33 (br s, 1H, NH); ^{13}C NMR (100 MHz, acetone-d_6): δ 17.9, 114.2, 118.0, 127.1, 130.6, 133.2, 135.4, 148.6, 208.0; ^{77}Se NMR (76 MHz, acetone-d_6): δ 720.6; MS (CI) $m/z = 225$ [M$^+$ + 1]; Anal calcd for $C_9H_8N_2Se$: C, 48.44; H, 3.61; N, 12.55. Found: C, 48.78; H, 3.88; N, 12.51.

*2-Methyl-1,3-benzenediselenocarboxamide (***4c***).* Yellow solid; mp 164.2–165.6°C; IR (KBr) 3195, 3018, 1648 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ 2.27 (s, 3H, CH_3), 7.12–7.20 (m, 3H, Ar), 10.36 (br s, 2H, NH), 10.99 (br s, 2H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 15.6, 124.0, 124.5, 125.0, 146.7, 206.6; ^{77}Se NMR (76 MHz, DMSO-d_6): δ 642.1; MS (CI) $m/z = 307$ [M$^+$ + 1].

*1,4-Butanediselenoamide (***4f***).* Brown solid; mp 96.6–98.0°C; IR (KBr) 3262, 3110, 1635 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ 2.87 (s, 4H, CH_2), 10.02 (br s, 2H, NH), 10.36 (br s, 2H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 46.6, 210.1; ^{77}Se NMR (76 MHz, DMSO-d_6): δ 493.6; MS (CI) $m/z = 245$ [M$^+$ + 1].

*4-Cyanobutaneselenoamide (***3g***).* Red liquid; IR (neat) 3299, 3178, 2249, 1635 cm^{-1}; ^{1}H NMR (400 MHz, acetone-d_6): δ 2.16 (quint, $J = 7.2$ Hz, 2H, CH_2), 2.59 (t, $J = 7.2$ Hz, 2H, CH_2), 2.76 (t, $J = 7.2$ Hz, 2H, CH_2), 9.48 (br s, 1H, NH), 9.65 (br s, 1H, NH); ^{13}C NMR (100 MHz, acetone-d_6): δ 16.2, 26.1, 46.9, 120.2, 213.6; ^{77}Se NMR (76 MHz, acetone-d_6): δ 550.4; MS (CI) $m/z = 177$ [M$^+$ + 1]; Anal calcd for $C_5H_8N_2Se$: C, 34.30; H, 4.61; N, 16.00. Found: C, 34.67; H, 4.85; N, 15.91.

*1,5-Pentanediselenoamide (***4g***).* Yellow solid; mp 94.2–95.8°C; IR (KBr) 3272, 3108, 1636 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ 2.04 (quint, $J = 7.6$ Hz, 2H, CH_2), 2.53 (t, $J = 7.6$ Hz, 4H, CH_2), 9.95 (br s, 2H, NH), 10.29 (br s, 2H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 28.7, 46.8, 211.3; ^{77}Se NMR (76 MHz, DMSO-d_6): δ 505.5; MS (CI) $m/z = 259$ [M$^+$ + 1].

*5-Cyanopentaneselenoamide (**3h**)*. Yellow liquid; IR (neat) 3299, 3172, 2248, 1635 cm^{-1}; ^1H NMR (400 MHz, acetone-d_6): δ 1.72 (quint, $J = 7.8$ Hz, 2H, CH_2), 1.94 (quint, $J = 7.8$ Hz, 2H, CH_2), 2.52 (t, $J = 7.8$ Hz, 2H, CH_2), 2.74 (t, $J = 7.8$ Hz, 2H, CH_2), 9.40 (br s, 1H, NH), 9.59 (br s, 1H, NH); ^{13}C NMR (100 MHz, acetone-d_6): δ 17.0, 25.2, 29.3, 48.4, 120.6, 214.9; ^{77}Se NMR (76 MHz, acetone-d_6): δ 549.6; MS (CI) $m/z = 191$ [$M^+ + 1$]; Anal calcd for $C_6H_{10}N_2Se$: C, 38.11; H, 5.33; N, 14.81. Found: C, 38.24; H, 5.38; N, 14.81.

*1,6-Hexanediselenoamide (**4h**)*. Yellow solid; mp 145.2–146.4°C; IR (KBr) 3285, 3109, 1643 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 1.62 (m, 4H, CH_2), 2.49 (m, 4H, CH_2), 9.90 (br s, 2H, NH), 10.22 (br s, 2H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 28.2, 47.8, 211.9; ^{77}Se NMR (76 MHz, DMSO-d_6): δ 497.8; MS (CI) $m/z = 273$ [$M^+ + 1$].

*6-Cyanohexaneselenoamide (**3i**)*. Yellow solid; mp 44.0–45.2°C; IR (KBr) 3298, 3104, 2248, 1637 cm^{-1}; ^1H NMR (400 MHz, acetone-d_6): δ 1.48 (quint, $J = 7.6$ Hz, 2H, CH_2), 1.65 (quint, $J = 7.6$ Hz, 2H, CH_2), 1.82 (quint, $J = 7.6$ Hz, 2H, CH_2), 2.45 (t, $J = 7.6$ Hz, 2H, CH_2), 2.68 (t, $J = 7.6$ Hz, 2H, CH_2), 9.35 (br s, 1H, NH), 9.52 (br s, 1H, NH); ^{13}C NMR (100 MHz, acetone-d_6): δ 17.1, 25.9, 28.4, 29.5, 49.1, 120.7, 215.5; ^{77}Se NMR (76 MHz, acetone-d_6): δ 544.1; MS (CI) m/z 205 [$M^+ + 1$]; Anal calcd for $C_7H_{12}N_2Se$: C, 41.39; H, 5.95; N, 13.79. Found: C, 41.41; H, 6.02; N, 13.82.

*1,7-Heptanediselenoamide (**4i**)*. Yellow solid; mp 129.0–130.6°C; IR (KBr) 3280, 3116, 1631 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 1.27 (quint, $J = 7.6$ Hz, 2H, CH_2), 1.67 (quint, $J = 7.6$ Hz, 4H, CH_2), 2.53 (t, $J = 7.6$ Hz, 4H, CH_2), 9.94 (br s, 2H, NH), 10.27 (br s, 2H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 27.2, 28.7, 47.9, 212.1; ^{77}Se NMR (76 MHz, DMSO-d_6): δ 497.3; MS (CI) $m/z = 287$ [$M^+ + 1$].

*6-Cyano-4-oxaheptaneselenoamide (**3j**)*. Yellow liquid; IR (neat) 3303, 3188, 2253, 1631 cm^{-1}; ^1H NMR (400 MHz, acetone-d_6): δ 2.70 (t, $J = 6.1$ Hz, 2H, CH_2), 2.89 (t, $J = 6.1$ Hz, 2H, CH_2), 3.69 (t, $J = 6.1$ Hz, 2H, CH_2), 3.88 (t, $J = 6.1$ Hz, 2H, CH_2), 9.36 (br s, 1H, NH), 9.60 (br s, 1H, NH); ^{13}C NMR (100 MHz, acetone-d_6): δ 19.1, 49.3, 66.5, 70.4, 119.2, 212.1; ^{77}Se NMR (76 MHz, acetone-d_6): δ 549.4; MS (CI) $m/z = 207$ [$M^+ + 1$]; Anal calcd for $C_6H_{10}N_2OSe$: C, 35.13; H, 4.91; N, 13.66. Found: C, 35.41; H, 4.99; N, 13.64.

*4-Oxa-1,7-heptanediselenoamide (**4j**)*. Yellow solid; mp 116.0–117.4°C; IR (KBr) 3330, 3090, 1648 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 2.75 (t, $J = 6.5$ Hz, 4H, CH_2), 3.73 (t, $J = 6.5$ Hz, 4H, CH_2), 10.00 (br s, 2H, NH), 10.37 (br s, 2H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 47.8, 69.1, 208.7; ^{77}Se NMR (76 MHz, DMSO-d_6): δ 500.9; MS (CI) $m/z = 289$ [$M^+ + 1$].

*2-(3-Cyanophenyl)-1,3-selenazol-4-one (**5**)*. Yellow solid; mp 167.2–169.0°C; IR (KBr) 2230, 1714, 1603 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 4.55 (s, 2H, CH_2), 7.82 (t, $J = 7.8$ Hz, 1H, Ar), 8.23 (d, $J = 7.8$ Hz, 1H, Ar), 8.35 (d, $J = 7.8$ Hz, 1H, Ar), 8.45 (s, 1H, Ar); ^{13}C NMR (100 MHz, DMSO-d_6): δ 36.5, 112.8, 117.6, 130.8, 131.9, 132.8, 135.0, 138.0, 192.7, 194.5; ^{77}Se NMR (76 MHz, DMSO-d_6): δ 355.5; MS (EI) $m/z = 250$ [M^+]; Anal calcd for $C_{10}H_6N_2OSe$: C, 48.21; H, 2.43; N, 11.24. Found: C, 48.25; H, 2.44; N, 11.31.

*2-(3-Cyanophenyl)-6-hydroxy-1,3-selenazin-4-one (**6**)*. Yellow solid; mp 173.0–174.2°C; IR (KBr) 3423, 2242, 1655 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 5.24 (s, 1H, CH), 7.80 (t, $J = 7.8$ Hz, 1H, Ar), 8.16 (d, $J = 7.8$ Hz, 1H, Ar), 8.28 (d, $J = 7.8$ Hz, 1H, Ar), 8.40 (s, 1H, Ar), 12.60 (br s, 1H, OH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 89.1, 112.6, 117.8, 130.7, 131.9, 136.4, 139.1, 171.2, 178.5, 181.8; ^{77}Se NMR (76 MHz, DMSO-d_6): δ 692.6; MS (CI) m/z 279 [$M^+ + 1$]; Anal calcd for $C_{11}H_6N_2O_2Se$: C, 47.67; H, 2.18; N, 10.11. Found: C, 47.74; H, 2.23; N, 10.22.

*2-(3-Cyanophenyl)-4-phenyl-1,3-selenazole (**7**)*. White solid; mp 104.4–106.0°C; IR (KBr) 2229, 1601 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.37 (tt, $J = 1.6$, 7.6 Hz, 1H, Ar), 7.45 (t, $J = 7.6$ Hz, 2H, Ar), 7.54 (t, $J = 7.6$ Hz, 1H, Ar), 7.70 (dt, $J = 1.6$, 7.6 Hz, 1H, Ar), 7.98 (d, $J = 7.6$ Hz, 2H, Ar), 8.15 (dt, $J = 1.6$, 7.6 Hz, 1H, Ar), 8.16 (s, 1H, CH), 8.29 (t, $J = 1.6$ Hz, 1H, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ 113.3, 118.2, 119.6, 126.7, 128.2, 128.8, 129.8, 130.2, 131.0, 133.1, 134.8, 137.4, 157.4, 170.8; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 724.1; MS (CI) m/z 311 = [$M^+ + 1$]; Anal calcd for $C_{16}H_{10}N_2Se$: C, 62.15; H, 3.26; N, 9.06. Found: C, 64.23; H, 3.31; N, 9.00.

*1,3-Bis(4-phenyl-1,3-selenazol-2-yl)benzene (**8**)*. White solid; mp 181.2–183.2°C; IR (KBr) 1599 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.36 (t, $J = 7.8$ Hz, 2H, Ar), 7.46 (t, $J = 7.8$ Hz, 4H, Ar), 7.52 (t, $J = 7.8$ Hz, 1H, Ar), 8.03 (dd, $J = 1.6$, 7.8 Hz, 4H, Ar), 8.07 (dd, $J = 1.6$, 7.8 Hz, 2H, Ar), 8.14 (s, 2H, CH), 8.53 (t, $J = 1.6$ Hz, 1H, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ 118.9, 125.5, 126.8, 128.0, 128.6, 128.7, 129.6, 135.3,

137.2, 157.1, 172.9; ^{77}Se NMR (76 MHz, $CDCl_3$): δ 719.1; MS (CI) $m/z = 493$ [$M^+ + 1$]; Anal calcd for $C_{24}H_{16}N_2Se_2$: C, 58.79; H, 3.29; N, 5.71. Found: C, 58.75; H, 3.41; N, 5.74.

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