and before the next portion was added. After ca. half of the quinone had been added, a white precipitate appeared. The reaction mixture was stirred for an hour after the addition of quinone was completed, and the white precipitate was filtered off. Recrystallization from MeCN gave white prisms (2.5 g, 68% yield): mp 234–235 °C; IR (Nujol) 3300 (br), 1600 (br), 1170, 910, 865 cm^{-1}; ^{13}C NMR (Me_2SO-d_6) δ 190.9, 145.3, 124.4, 122.0, 107.9, 22.8. Anal. Calcd for $C_{16}H_{16}O_6Cl_2$: 374.0322. Found: 374.0308 (by MS). Calcd: C, 51.22; H, 4.30. Found: C, 50.81; H, 4.31.

A diacetate derivative (**8**) was prepared (Ac_2O–py) and recrystallized from EtOH: mp 222–228 °C; IR (Nujol) 1770, 1590 (br), 1175, 1005, 920 cm^{-1}; ^1H NMR ($CDCl_3$) δ 16.8 (s, 1 H), 2.26 (s, 3 H), 1.90 (s, 6 H); ^{13}C NMR ($CDCl_3$) δ 191.4, 167.4, 145.5, 132.1, 129.2, 106.5, 23.4, 20.1. Anal. Calcd for $C_{20}H_{20}O_8Cl_2$: C, 52.30; H, 4.39. Found: C, 52.14; H, 5.01. Calcd: 458.0532. Found: 458.0520 (by MS).

1,4-Diacetoxy-2,5-dichloro-3-(diacetylmethyl)benzene. The filtrate from the isolation of **7** was evaporated and treated with acetic anhydride and pyridine catalyst. Pouring into cold water and stirring gave a white solid which was recrystallized repeatedly from EtOH giving 1,4-diacetoxy-2,5-dichlorobenzene mp 139–141 °C (lit.[12] 141 °C), and the diacetate derivative of **2d**: mp 126–128 °C; IR (Nujol) 1780, 1770, 1580, 1165 cm^{-1}; ^1H NMR ($CDCl_3$) δ 16.8 (s, 1 H), 7.28 (s, 1 H), 2.35 (s, 3 H), 2.24 (s, 3 H), 1.87 (s, 6 H); ^{13}C NMR ($DCCl_3$) δ 191.5, 167.7, 167.5, 145.4, 145.0, 132.4, 128.3, 126.7, 124.6, 106.5, 23.4, 20.5, 20.1. Anal. Calcd for $C_{15}H_{14}O_6Cl_2$: C, 49.88; H, 3.91. Found: C, 49.89; H, 4.17.

2,5-Dichloro-3,6-bis(diacetylmethyl)-1,4-benzoquinone (9). The hydroquinone **7** was smoothly oxidized by Ag_2O (CH_2Cl_2, Na_2SO_4) or by $FeCl_3$ (diethyl ether, MeOH, water) to the bright orange quinone: mp 205–206 °C; IR (Nujol) 3240 (br), 1730, 1680 (sh), 1580 (br), 1180, 1020 cm^{-1}; ^1H NMR ($CDCl_3$) δ 16.9 (s, 1 H), 1.95 (s, 6 H); ^{13}C NMR ($CDCl_3$) 190.3, 177.0, 144.9, 141.7, 105.4, 23.8. Anal. Calcd for $C_{16}H_{14}O_6Cl_2$: 372.0166. Found: 372.0164 (by MS). Calcd: C, 51.49; H, 3.79. Found: C, 51.24; H, 3.65.

1,1′-(2,5-Diacetoxy-3,6-dichloro-1,4-phenylene)bis(2-propanone) (4d). When 1.7 g of **7** was suspended in 35 mL of tetrahydrofuran and treated with ca. 0.5 mL of Triton B, the reaction mixture became homogeneous immediately and turned successively orange and then brown. After 1 h of stirring at room temperature the reaction mixture was concentrated to a brown oil and triturated with MeOH. Filtration gave 0.25 g of a white solid with a melting point of 230–232 °C. The filtrate was poured into H_2O, and the tan solid thus obtained was recrystallized twice from EtOH. The white needles (1.4 g, 80% combined yield) had a melting point of 233–235 °C. IR (Nujol) 1770, 1715, 1175; ^1H NMR ($CDCl_3$) 3.77 (s, 2 H), 2.33 (s, 3 H), 2.13 (s, 3 H); ^{13}C NMR ($CDCl_3$) δ 202.7, 167.4, 144.8, 128.5, 127.7, 43.9, 29.1, 20.2. Anal. Calcd for $C_{16}H_{16}O_6Cl_2$: C, 51.22; H, 4.30. Found: C, 50.92; H, 4.29. Calcd: 374.0322. Found: 374.0315 (by MS).

2,6-Dichloro-3-(diacetylmethyl)-1,4-hydroquinone (10). A mixture of 1.8 g (10 mmol) of 2,6-dichloro-1,4-benzoquinone, 2.0 g (20 mmol) of 2,4-pentadione, and 0.1 g of $Ni(acac)_2$ in 30 mL of MeOH was stirred overnight. The reaction mixture was poured into ca. 250 mL of water, and 2.3 g of a tan powder was filtered off. Recrystallization from ($EtOH/H_2O$) gave **2e** (2.1 g, 76% yield): mp 190–192 °C; IR (Nujol) 3420, 1580 (br), 1185 cm^{-1}; ^1H NMR ($CDCl_3$) δ 16.9 (s, 1 H), 6.95 (s, 1 H), 4.7–5.7 (br, 1 H), 1.89 (s, 6 H).

A diacetate derivative (**11**) was prepared (Ac_2O–py): mp 132–133 °C; IR (Nujol) 1770, 1590 (br), 1200, 1165 cm^{-1}; ^1H NMR ($CDCl_3$) δ 16.9 (s, 1 H), 7.22 (s, 1 H), 2.42 (s, 3 H), 2.20 (s, 3 H), 1.87 (s, 6 H); ^{13}C NMR δ 191.6, 168.3, 166.9, 147.9, 142.7, 131.5, 129.6, 128.8, 122.9, 101.1, 23.3, 20.4, 20.1. Anal. Calcd for $C_{15}H_{14}Cl_2O_6$: 360.0167. Found: 360.0156 (by MS). Calcd: C, 49.88; H, 3.91. Found: C, 49.59; H, 3.84.

2,6-Dichloro-3-(diacetylmethyl)-1,4-benzoquinone (12). A suspension of 0.50 g (1.8 mmol) of **2e** in 50 mL of CH_2Cl_2 was treated with 0.9 g (3.9 mmol) of Ag_2O and 1 g of Na_2SO_4 and stirred overnight at room temperature. The reaction mixture was filtered, and the solvent was evaporated. Recrystallization of the orange residue from EtOH gave 0.42 g (84%) of the bright orange-red quinone: mp 149–150 °C; IR (Nujol) 1685, 1645, 1585 (br), 1195, 1040 cm^{-1}; ^1H NMR ($CDCl_3$) δ 16.9 (s, 1 H), 7.21 (s, 1 H), 1.94 (s, 6 H); ^{13}C NMR ($CDCl_3$) δ 190.2, 182.0, 172.6, 143.9, 143.7, 141.6, 133.9, 105.1, 23.8. Anal. Calcd for $C_{11}H_8Cl_2O_4$: C, 48.03; H, 2.93. Found: C, 48.29; H, 3.01. calcd: 273.9800. Found: 273.9801 (by MS).

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Registry No. **1a**, 935-92-2; **1b**, 137-18-8; **1c**, 844-51-9; **1d**, 615-93-0; **1e**, 697-91-6; **2d**, 93645-28-4; **2d** diacetate, 93645-29-5; **2e**, 93645-30-8; **2e** diacetate, 93645-31-9; **3a**, 68591-15-1; **3a** acetate, 93645-32-0; **3c**, 93645-34-2; **3c** acetate, 93645-35-3; **4b**, 93645-36-4; **4c**, 93645-37-5; **4d**, 93645-38-6; **7**, 93645-39-7; **8**, 93645-40-0; **9**, 93645-41-1; **12**, 93645-42-2; acetylacetone, 123-54-6.

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Synthesis Utilizing Reducing Ability of Carbon Monoxide. A New Method for Synthesis of Selenocarboxamides: Reaction of Nitriles with Selenium, Carbon Monoxide, and Water

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Recently a variety of metal carbonyl complexes[1] were reported to catalyze the water–gas shift reaction under basic conditions. Furthermore, metal hydrides, which are intermediates in the water–gas shift reaction, have found use in several organic reactions such as reduction[2] or reductive carbonylation[3] of nitrobenzenes and carbonylation of olefins.[4]

During our studies on the selenium-catalyzed carbonylation of various organic compounds with carbon mon-

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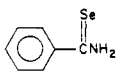
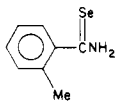
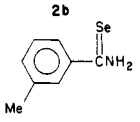
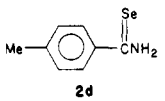
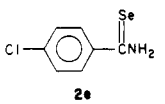
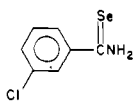
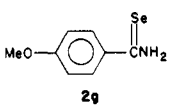
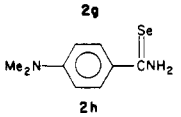
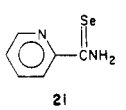
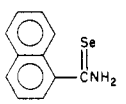
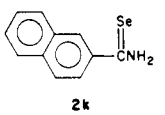
Table I. Synthesis of Aromatic Selenocarboxamides[a]

entry	formula	yield,[e] %	mp, °C (lit.)	IR, cm^{-1} (KBr)	^1H NMR (Me_2SO-d_6)	MS (m/e M^+)[e]
1	 2a	76	126.0-126.5 (125[9g])	3325, 3265, 1622	7.20-7.70 (m, 3 H), 7.70-8.10 (m, 2 H), 10.17 (br s, 1 H), 10.72 (br s, 1 H)	185
2[b]	 2b	22	104.0-106.0	3390, 3270, 1615	2.26 (s, 3 H), 7.70 (s, 4 H), 9.97-10.64 (br s, 2 H)	199
3	 2c	78	72.0-73.0 (74[9g])	3280, 1610,	2.27 (s, 3 H), 7.15-7.47 (m, 4 H), 9.83 (br s, 1 H), 10.40 (br s, 1 H)	199
4[d]	 2d	100	165.0-167.0 (186,[9g] 161[9b])	3370, 3270, 1620	2.30 (s, 3 H), 7.02-7.15 (d, 2 H), 7.69-7.82 (d, 2 H), 9.85 (br s, 1 H), 10.35 (br s, 1 H)	199
5	 2e	99	127.5-128.5 (129[9g])	3320, 3280, 1630	7.33-7.47 (d, 2 H), 7.79-7.93 (d, 2 H), 10.15 (br s, 1 H), 10.75 (br s, 1 H)	219
6[b]	 2f	61	125.5-126.5	3330, 3285, 1620	7.21-7.81 (m, 4 H), 10.20 (br s, 1 H), 10.80 (br s, 1 H)	219
7	 2g	91	155.0-156.5 (157[9g])	3360, 3270, 1598	3.80 (s, 3 H), 6.81-6.95 (d, 2 H), 7.85-7.99 (d, 2 H), 9.90 (br s, 1 H), 10.35 (br s, 1 H)	215
8[b]	 2h	82	221.0-222.5	3370, 3270, 1605	2.94 (s, 6 H), 6.46-6.62 (d, 2 H), 7.78-7.94 (d, 2 H), 9.53 (br s, 1 H), 9.84 (br s, 1 H)	228
9	 2i	74	142.0-143.5 (142[9g])	3320, 3250, 1580	7.36-8.57 (m, 4 H), 10.70 (br s, 2 H)	186
10[b]	 2j	21	131.0-132.0	3370, 3260, 1615	7.30-8.20 (m, 7 H), 10.30 (br s, 1 H), 10.90 (br s, 1 H)	235
11[b]	 2k	82	138.0-140.0	3355, 3280, 1620	7.33-8.33 (m, 7 H), 10.17 (br s, 1 H), 10.67 (br s, 1 H)	235

[a] All reactions were performed on 5-mmol scale according to the procedures described in the text. [b] New compound. [c] Isolated yield. [d] The melting point is different from the values reported in some literatures;[9b,g] however, both elemental analyses for C, H, N and spectral analyses (IR, ^1H NMR, and mass spectrum) satisfactorily support compound **2d**. [e] The molecular weight (M^+) is based on the ^{80}Se isotope.

oxide,[5] we found that elemental selenium[6] was readily reduced to hydrogen selenide (**1'**) by carbon monoxide and water in the presence of triethylamine under mild conditions (eq 1).[7] This method would be exceedingly convenient, in terms of manipulation without isolation of air-sensitive and highly toxic hydrogen selenide, for the preparation of various selenium-containing organic compounds. On the basis of this view, we examined the reaction of in situ formed hydrogen selenide with nitriles to synthesize selenocarboxamides **2** (eq 2), which are useful reagents for the synthesis of selenium–nitrogen heterocycles such as 1,3-selenazoles.[8]

$$Se + CO + H_2O \xrightarrow{\text{base}} H_2Se + CO_2 \qquad (1)$$

$$\underset{\mathbf{1}}{RCN} + Se + CO + H_2O \xrightarrow{\text{base}} \underset{\mathbf{2}}{R\overset{\overset{Se}{\|}}{C}NH_2} + CO_2 \qquad (2)$$

In general, selenocarboxamides have been prepared by the addition of hydrogen selenide (**1'**) to the corresponding nitriles.[9] In earlier investigations,[9a-f] poisonous hydrogen

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(6) Both metallic and amorphous selenium can be used.

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Table II. Synthesis of Aliphatic Selenocarboxamides[a]

entry	formula		yield,[b] %
1	$CH_3CH_2CH_2C(=Se)NH_2$	**2l**	35
2	$PhCH_2C(=Se)NH_2$	**2m**	38
3	$CH_3OCH_2C(=Se)NH_2$	**2n**	35

[a] All reactions were performed on 5-mmol scale according to the procedures described in the text. [b] Isolated yield.

selenide was directly bubbled into the reaction vessel; however, the yields were generally very low. Better yields have been reported by Cohen[9g] using excess amounts of aluminum selenide (Al_2Se_3) and water as the source of hydrogen selenide.

Treatment of benzonitrile with selenium, carbon monoxide, and water in the presence of triethylamine at 100 °C for 5 h gave rise to the corresponding selenocarboxamide **2a** (76% yield).

In a similar manner a number of aromatic and heterocyclic selenocarboxamides were successfully synthesized from the corresponding nitriles as shown in Table I. Structural assignments of selenocarboxamides were based on spectral analyses (IR, ^1H NMR, and mass spectrum) after isolation of the products (see Table I). The elemental analyses of the new amides (**2b**, **2f**, **2h**, **2j**, and **2k**) were also in good agreement with the calculated values. The yields of selenocarboxamides were high except for some cases of sterically hindered amides such as **2b** and **2j**.

The aromatic selenoamides obtained are all yellow solids and are stable enough under nitrogen at ordinary temperature to be kept for several weeks in high purity. On exposure to air, they gradually decomposed into the starting nitriles, elemental selenium, and water at room temperature or below. These observations suggest that the selenoamides are in equilibrium with the corresponding nitriles and hydrogen selenide (eq 3) and that the equi-

$$RC(=Se)NH_2 \rightleftharpoons RCN + H_2Se \qquad (3)$$

librium lies to the selenoamides at room temperature under nitrogen. However, in air, hydrogen selenide is gradually consumed by oxidation to water and selenium, so that the equilibrium may be shifted to the right of eq 3.

In contrast to aromatic selenoamides, little is known about the isolation of N-unsubstituted aliphatic selenoamides. Phenylselenoacetamide has been prepared by Kindler[9c] and shown to be thermally unstable and susceptible to air. Another example is a carbohydrate possessing the selenoamide group.[10] Aliphatic selenoamides are assumed to be less stable owing to the lack of conjugation between the aromatic ring and the selenocarbonyl group observed in aromatic ones. The synthesis of aliphatic selenoamides was attempted by using aliphatic nitriles in a similar fashion to that described above, and the corresponding selenoamides were successfully isolated as shown in Table II. These selenoamides isolated were thermally unstable and highly sensitive to air and, even under nitrogen, gradually dissociated at room temperature[11] into the starting nitriles and hydrogen selenide, which deposited elemental selenium on exposure to air: The position of the equilibrium of aliphatic selenoamides was slighlty shifted to left compared with that of aromatic ones. This is the cause of the lower yields of the aliphatic selenoamides. Owing to such instablity of aliphatic selenoamides, confirmation of their structures by mass spectrum and elemental analyses was unsuccessful and therefore the assignments of selenoamides **21**, **2m**, and **2n** were performed only on the basis of spectral analyses (IR and ^1H NMR spectrum).[12]

The present method for selenoamide synthesis described permitted high-yield synthesis of aromatic and heterocyclic selenoamides with simple operations and made it possible to prepare unstable aliphatic selenoamides.

Experimental Section

General Methods. The instruments used were as follows: melting points, Yanagimoto micro melting point apparatus; ^1H NMR, Hitachi R-24B; IR, Shimadzu IR-400; MS, Hitachi RMU-6A.

Metallic selenium (99.99%) of Nakarai Chem. Co. and carbon monoxide (99.999%) of Seitetsu Chem. Co. were used. Nitriles, triethylamine, and tetrahydrofuran (THF) were all purchased from commercial sources and purified by distillation or recrystallization.

General Procedures for Synthesis of Selenocarboxamide. In a typical reaction, a stirred mixture of benzonitrile (0.52 g, 5 mmol),[13] selenium (0.43 g, 5.5 mmol), water (1 mL, 56 mmol), triethylamine (1 mL, 7 mmol), and tetrahydrofuran (5 mL) in a 50-mL stainless steel autoclave was heated under the pressure of carbon monoxide (5 atm: initial pressure at 25 °C) at 100 °C for 5 h. After the reaction, carbon monoxide was purged in the well-ventilated hood, and the reaction mixture was slightly acidified with aqueous hydrochloric acid (2 N)[14] and extracted with diethyl ether (50 mL × 3). The combined extracts were dried ($MgSO_4$) under nitrogen, filtered, and evaporated. The crude material was chromatographed on silica gel, affording 0.69 g (76%) of benzeneselenocarboxamide (**2a**). Anal. Calcd for C_7H_7NSe: C, 45.67; H, 3.83; N, 7.61. Found: C, 45.78; H, 3.91; N, 7.48.

4-(Dimethylamino)benzeneselenocarboxamide (2h). The reaction was carried out in the same manner described above for the general procedure of selenoamide synthesis. After the reaction, the reaction mixture was neutralized. A yellow precipitate deposited, was filtered off, and washed with water, *n*-hexane, and benzene successively. The remaining solid, containing metallic selenium, was dissolved in acetone, and metallic selenium was removed by filtration. The filtrate was dried and evaporated to give 0.92 g of selenoamide **2h** (82% yield).

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Registry No. 1′, 7783-07-5; **1a**, 100-47-0; **1b**, 529-19-1; **1c**, 620-22-4; **1d**, 104-85-8; **1e**, 623-03-0; **1f**, 766-84-7; **1g**, 874-90-8; **1h**, 1197-19-9; **1i**, 100-70-9; **1j**, 86-53-3; **1k**, 613-46-7; **1l**, 109-74-0; **1m**, 140-29-4; **1n**, 1738-36-9; **2a**, 5977-82-2; **2b**, 93756-97-9; **2c**, 68090-01-7; **2d**, 67213-27-8; **2e**, 67213-28-9; **2f**, 93756-98-0; **2g**, 68090-03-9; **2h**, 93756-99-1; **2i**, 68090-07-3; **2j**, 93757-00-7; **2k**, 93757-01-8; **2l**, 93757-02-9; **2m**, 93757-03-0; **2n**, 93757-04-1; Se, 7782-49-2; H_2O, 7732-18-5; CO, 630-08-0.

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(11) Aliphatic selenoamides obtained decomposed even at −20 °C for several days under nitrogen.

(12) **21**: IR (Nujol) 3280, 1630 cm^{-1}; ^1H NMR (Me_2SO-d_6) δ 0.87 (t, 3 H), 1.62 (t-q, 2 H), 2.52 (t, 2 H), 9.80 (br s, 1 H), 10.00 (br s, 1 H). **2m**: IR (KBr) 3280, 1635 cm^{-1}; ^1H NMR (Me_2SO-d_6) δ 3.09 (s, 2 H), 7.31 (m, 5 H), 9.90 (br s, 2 H). **2n**: IR (KBr) 3260, 1635 cm^{-1}; ^1H NMR (Me_2SO-d_6) δ 3.41 (s, 3 H), 4.04 (s, 2 H), 9.40 (br s, 1 H), 10.60 (br s, 1 H).

(13) A larger scale reaction is also possible: benzonitrile (5.1 mL, 50 mmol), Se (3.95 g, 50 mmol), water (2 mL, 112 mmol), carbon monoxide (40 atm), Et_3N (5 mmol, 35 mmol), THF (7 mL).

(14) The remaining hydrogen selenide, which was generated upon the acidification of the reaction system, could be captured entirely by introducing into aqueous KOH solution. Elemental selenium was easily recovered on exposure of the solution to air.