

Formation of Carbocyclic Compounds from 1,5-Hexadiene by the Reaction with Benzeneselenenyl Chloride in the Presence of Acid

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Received July 3, 1992

Intramolecular carbon-carbon bond formation has proceeded by the reaction of 1,5-hexadiene with benzeneselenenyl chloride in the presence of metal chloride to afford carbocyclic compounds such as 4-(phenylseleno)cyclohexene, 4-chloro-1-(phenylseleno)cyclohexanes, and 3-chloro-1-(phenylseleno)cyclohexane.

KEY WORDS: Benzeneselenenyl chloride / Carbon-carbon bond formation / Lewis acid / Diolefin

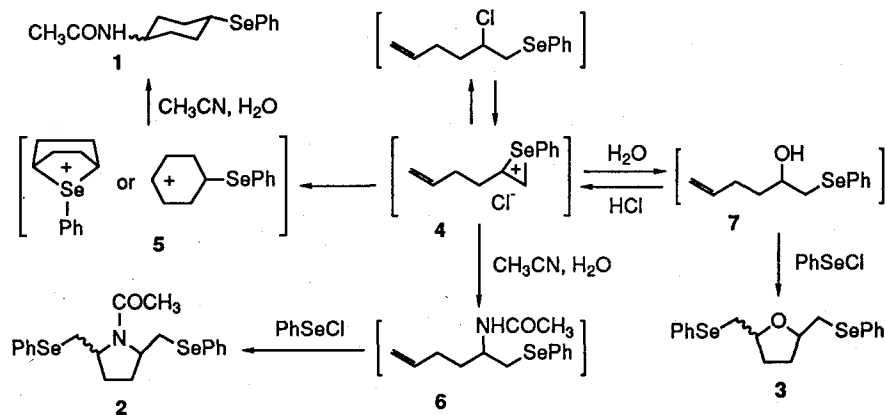
We have already reported two types of amidoselenation reactions. One is the reaction of olefins with benzeneselenenyl chloride in acetonitrile as solvent in the presence of trifluoromethanesulfonic acid and water (introduction of acylamino and phenylseleno groups into adjacent carbon atoms)¹⁾ and the other is the reaction of olefins, benzeneselenenyl chloride, and *p*-toluenesulfonamide in the presence of Lewis acid catalyst introducing tosylamino and phenylseleno groups into olefins.²⁾ We now applied these reactions to certain diolefins such as 1,5-hexadiene and observed the formation of nitrogen heterocycles presumably through the amidoselenation of one double bond and subsequent intramolecular amidoselenation³⁾ of another double bond. Interestingly, we found that varying amounts of cyclohexane derivatives were produced during these reactions. These results indicate that carbon-carbon bond formation proceeds by the reaction of diolefins with benzeneselenenyl chloride. We searched for the condition for selective carbon-carbon bond formation and found that carbocyclic compounds are produced in satisfactory yield by the reaction of benzeneselenenyl chloride with 1,5-hexadiene in the presence of aluminum(III) chloride in 1,2-dichloroethane-ether (5-10:1) as solvent. Our reaction represents the first example of the reaction of episelenonium ion bearing chloride ion as gegen anion⁴⁾ with carbon nucleophile and may add another good example to organoselenium induced carbon-carbon bond formations in diolefins.⁵⁾

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Results and Discussion

By the reaction of 1,5-hexadiene with benzeneselenenyl chloride (PhSeCl) in acetonitrile as solvent in the presence of trifluoromethanesulfonic acid and water, 1-(acylamino)-4-(phenylseleno)cyclohexanes (**1**) were produced accompanied by *N*-acetyl-2,5-bis[(phenylseleno)methyl]pyrrolidines (**2**) and 2,5-bis[(phenylseleno)methyl]tetrahydrofurans (**3**) (Scheme 1). Formation of these compounds can be interpreted by the competitive attack on the episelenonium ion (**4**) by carbon-carbon double bond (to afford **5** and ultimately **1**), by nitrogen atom of acetonitrile (to afford **2** via **6**), and by oxygen atom of water (to afford **3** via **7**) as shown in Scheme 1. We have already reported that PhSeCl is a good reagent for hydroxysele-
 nation of olefins⁶⁾ and also for the formation of cyclic ethers (**1**) from nonconjugated dienes.^{7, 6b)} We have also reported that PhSeCl can induce the intramolecular amidoselenation of *N*-alkenylamides (although less effective than PhSeBr).³⁾ Thus, the formation of **2** is also quite expected when combined with the fact that the present reaction conditions are similar to those reported for amidoselenation of monoolefins.¹⁾ This reaction, however, represents the first example of PhSeCl to induce the carbon-carbon bond formation in diolefins.^{4, 5)} Typical results are summarized in Table 1. Although we succeeded to suppress the formation of **1** or **3**



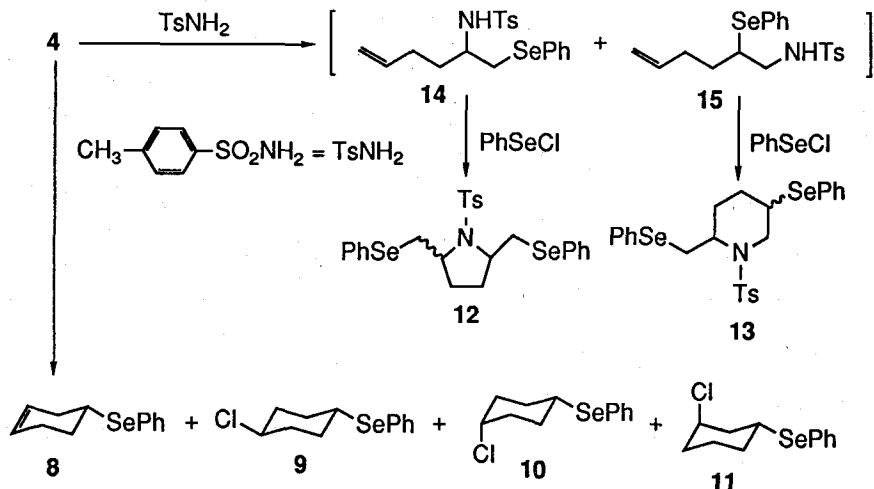
Scheme 1

 Table 1. Yields of **1**, **2**, and **3** from 1,5-Hexadiene^{a)}

Entry	PhSeCl (mmol)	CF ₃ SO ₃ H (mmol)	H ₂ O (mmol)	Time (h)	Yield (%) ^{b)}		
					1	2	3
1	5	5	25	2	trace	53	10
2	5	5	5	1	31	39	0
3	10	5	5	2	trace	40	19
4	10	10	50	3	0	27	36
5	10	10	10	1	9	43	4

^{a)} Carried out using 1,5-hexadiene (5 mmol) in acetonitrile (30ml) at reflux temperature.

^{b)} Isolated yields by column chromatography. Based on the amount of PhSeCl.



Scheme 2

(Entries 1, 3, and 4 and Entry 2 respectively) by the change of reaction conditions, we could not find the conditions for selective formation of 1 or 2. While episelenonium ion (4) can react with any kind of nucleophile, carbenium ion (5), produced as a result of carbon-carbon bond formation, reacts selectively with acetonitrile to afford 1. We could not detect cyclohexanol or chlorocyclohexane derivatives which would be produced by the trap of 5 with oxygen or chlorine nucleophile.

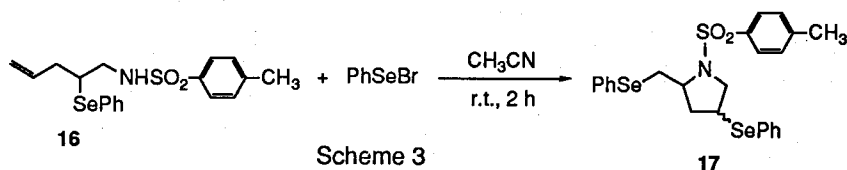
When 1,5-hexadiene was allowed to react with PhSeCl in the presence of *p*-toluenesulfonamide and zinc(II) chloride, only two kinds of cyclization have proceeded; namely, formation of nitrogen heterocycles (12, 13) (43%) and of carbocyclic compounds (8, 9, 10, and 11) (46%) (Scheme 2). Although nitrogen heterocycles consisted of five- and six-membered compounds, carbocyclic compounds were found to consist only of cyclohexane derivatives. The identification of the products (8-11) were carried out as follows. We succeeded to separate 8 from other carbocyclic compounds by column chromatography and isolated as a mixture with diphenyl diselenide. In the ^{13}C NMR spectrum of this mixture, the resonance of the carbon bearing phenylseleno group appeared at 39.2 ppm as doublet, indicating that this carbon is contained in the ring (*endo*-cyclization). It is quite reasonable that olefinic product consists of one regioisomer, namely 4-(phenylseleno)cyclohexene (8), as *endo*-cyclization of 4 produces symmetrical carbenium ion (5) which affords the same olefin by the loss of proton from either carbon. A mixture of 9 and 10 (41:59) was isolated from their mixture with 11. Quite simple ^{13}C NMR spectrum of them strongly suggested that 9 and 10 are 1,4-substituted cyclohexanes. Their structure was further confirmed by reductive replacement of phenylseleno group by hydrogen atom (nickel chloride-sodium borohydride)⁸⁾ to afford chlorocyclohexane as a sole product. Assignment of *trans* (9) and *cis* (10) structure was based on the half-band-width in their ^1H NMR spectrum of the protons attached to the carbon bearing chlorine atom. The structure of 11 was confirmed by the comparison of their

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spectral data with authentic sample of *trans*-1-chloro-3-(phenylseleno)cyclohexane prepared from 2-cyclohexen-1-one through the 1,4-addition of lithium benzeneselenolate, reduction of the carbonyl group, and replacement of the hydroxyl group by chlorine atom by the reaction with thionyl chloride in the presence of zinc(II) chloride.⁹ In the reduction of this transformation, axial attack of hydride ion on the carbonyl group is expected to produce 3-(phenylseleno)cyclohexanol with both substituents (OH and PhSe) on equatorial positions. As the replacement of the hydroxy group by chlorine atom is expected to proceed with inversion of configuration,⁹ it is reasonable to assume that configuration of carbon-chlorine bond in this product is axial (*vide infra*).

We confirmed that intramolecular amidoselenation of *N*-4-pentenyl-*p*-toluenesulfonamide (16) proceeds via "exo-cyclization" to afford pyrrolidine derivatives (17) (Scheme 3). Thus, 12 and 13 in Scheme 2 are expected to be formed via "exo-cyclization" of Markownikof- and anti-Markownikof-type amidoselenation intermediates (14 and 15), respectively, in contrast to the "endo-cyclization" in carbon-carbon bond forming reactions to afford cyclohexane derivatives (8, 9, 10, and 11).

When the Lewis acid was changed from zinc(II) chloride to aluminum(III) chloride, formation of nitrogen heterocycles were completely suppressed and carbocyclic compounds (8-11) were produced in 70% total yield (Entry 2 in Table 2). We were surprised to find that the yield of carbocyclic compounds were considerably lowered (37%) in the reaction without *p*-toluenesulfonamide (Entry 4 in Table 2) in spite of the fact that the amide was not incorporated in the carbon-carbon bond formation products. We attributed the role of *p*-toluenesulfonamide to the


 Table 2. Yields of Nitrogen Heterocycles and Carbocyclic Compounds^{a)}

Entry	Lewis Acid (1 mmol)	<i>p</i> -Toluenesulfonamide (mmol)	Solvent (ml)	Yields (%) ^{b)}	
				12+13	8+9+10+11
1	ZnCl ₂ /Et ₂ O	1	ClCH ₂ CH ₂ Cl (3)	43 ^{c)}	46
2	AlCl ₃	1	ClCH ₂ CH ₂ Cl (3)	0	70
3	—	1	ClCH ₂ CH ₂ Cl (3)	0	0
4	AlCl ₃	0	ClCH ₂ CH ₂ Cl (3)	—	37
5	AlCl ₃	0	ClCH ₂ CH ₂ Cl (3) Et ₂ O (0.3)	—	71 ^{d)}
6	AlCl ₃	0	ClCH ₂ CH ₂ Cl (3) Et ₂ O (0.6)	—	70

^{a)} Carried out using 1,5-hexadiene (1 mmol) and PhSeCl (1 mmol) at room temperature for 24 h.

^{b)} Isolated yields by column chromatography. Based on the amount of PhSeCl.

^{c)} Isomer ratio; 12 : 13 = 72 : 28. ^{d)} Isomer ratio; 8 : 9 : 10 : 11 = 7 : 37 : 40 : 16.

coordination with aluminum(III) chloride and tried the use of other weak donors. As a result, we found that diethyl ether shows the same effect when used as co-solvent in the ratio of 1/10–1/5 to 1,2-dichloroethane (Entries 5 and 6 in Table 2). The isomer ratio of **8**, **9**, **10**, and **11** was estimated to be 7:37:40:16 by the integrals in ^1H NMR spectra of protons attached to olefinic carbons or carbons bearing chlorine atom. In the 1-chloro-4-(phenylseleno)cyclohexanes, the *cis* isomer (**10**) is expected to be less stable than the *trans* isomer (**9**). Thus, the trap of the open chain carbenium ion (**5**) by chloride anion would afford **10** as minor product. The formation of **10** as the major product and **11** (*trans* isomer) may be explained as the addition products of hydrogen chloride to the double bond of **8**. As the phenylseleno group plays the role as an anchor, the entering hydrogen and chlorine atoms are left on the axial positions as the result of *trans* addition to the double bond.

Experimental

The IR spectra were taken with a JASCO IR-810 spectrometer. ^1H and ^{13}C NMR spectra were recorded with JEOLCO JNM-FX-100 (100 MHz), JNM-GX-400 (400 MHz), and Varian VXR-200 (200 MHz) instruments in CDCl_3 using TMS as internal standard. Mass spectra were measured on a JEOL JMS-DX-300 mass spectrometer. Melting points were determined with a Shimadzu MM-2 micro melting points determination apparatus and were uncorrected. Liquid chromatographic analyses were carried out with a Waters HPLC system equipped with a 6000A solvent delivery system, a Model 440 absorbance detector (at 254 nm), and a μ -Porasil (3.9 mm \times 0.3 m) column.

Materials. Diethyl ether was dried over benzophenone ketyl and 1,2-dichloroethane was dried over calcium hydride and they were distilled just before use. *N*-[2-(phenylseleno)-4-pentenyl]-*p*-toluenesulfonamide (**16**) was prepared according to the reported procedure.²⁾ Aluminum(III) chloride was purified by sublimation. Zinc (II) chloride was also purified by sublimation and was stored and used as saturated solution in diethyl ether (0.69 M). All other organic and inorganic materials were commercial products and were used without further purification.

Reaction of 1,5-Hexadiene with Benzeneselenenyl Chloride in the Presence of Trifluoromethanesulfonic Acid in Acetonitrile as Solvent. To a solution of benzeneselenenyl chloride (0.99 g, 5 mmol) in acetonitrile (20 ml) was added a solution of 1,5-hexadiene (0.41 g, 5 mmol) in acetonitrile (5 ml) to give a pale yellow solution. A mixture of trifluoromethanesulfonic acid and water (molar ratio; 1/1; 5 mmol, 0.84 g) and acetonitrile (5 ml) were added to the solution and the resulting mixture was heated under reflux for 1 h. After cooling down to room temperature, the reaction mixture was poured into aq. NaHCO_3 (75 ml) and the products were extracted with CHCl_3 (40 ml \times 3). The organic layer was washed with brine (80 ml), dried (MgSO_4), and evaporated in vacuo to leave a yellow oil. Column chromatography [silica gel (200 mesh), hexane-ethyl acetate (5:1-2:1) as eluant] of this oil

afforded diphenyl diselenide (0.24 g, 0.77 mmol; 30%), *cis*- and *trans*-2,5-bis [(phenylseleno)methyl]pyrrolidine (**2**) (0.44 g, 0.98 mmol; 39%), and *cis*- and *trans*-1-(acetylamino)-4-(phenylseleno)cyclohexane (**1**) (0.46 g, 1.54 mmol; 31%). Liquid chromatographic analysis [hexane-chloroform-tetrahydrofuran (4:4:2) as eluant] of the final fraction revealed that the isomer ratio in **1** is *cis*:*trans* = 16:84. Repeated column chromatography allowed the separation of *cis*-**1** and *trans*-**1**. *cis*-**1**: Colorless oil. IR (liq. film) 3350 and 1645 cm^{-1} . ^1H NMR (100 MHz) δ 1.5–2.0 (m, 8 H), 1.98 (s, 3H), 3.4–3.5 (m, 1H), 3.7–4.0 (m, 1H), 5.5 (br. s, 1H), 7.1–7.3 (m, 3H), and 7.4–7.6 (m, 2H). ^{13}C NMR (25 MHz) δ 23.3 (q), 29.4* (t), 30.1* (t), 42.3 (d), 46.8 (d), 127.0 (d), 128.7* (d), 129.4 (s), 133.9* (d), and 169.2 (s) (* two signals overlapping). Found: C, 56.48; H, 6.52; N, 4.98%. Calcd for $\text{C}_{14}\text{H}_{19}\text{NOSe}$: C, 56.76; H, 6.46; N, 4.73%. *trans*-**1**: White needles, mp 126.5–127 °C [from hexane-chloroform (2:1)]. IR (KBr disc) 3360 and 1637 cm^{-1} . ^1H NMR (100 MHz) δ 1.0–2.2 (m, 8H), 1.97 (s, 3H), 3.05 (tt, 1H, $J=11.5$ and 3.5 Hz), 3.73 (dtt, 1H, $J=8, 11.5,$ and 3.5 Hz), 5.35 (br. d, 1H, $J=8$ Hz), 7.1–7.3 (m, 3H), and 7.4–7.6 (m, 2H). ^{13}C NMR (25 MHz) δ 23.1 (q), 32.9* (t), 33.3* (t), 40.8 (d), 47.7 (d), 127.4 (d), 128.4 (s), 128.7* (d), 134.8* (d), and 170.7 (d) (* two signals overlapping). Found: C, 57.04; H, 6.47; N, 4.77%.

Reaction of 1,5-Hexadiene with Benzeneselenenyl Chloride in the Presence of Aluminum(III) Chloride in 1,2-Dichloroethane and Diethyl Ether as Solvents. To a solution of benzeneselenenyl chloride (0.20 g, 1 mmol) in 1,2-dichloroethane (1.5 ml) was added 1,5-hexadiene (0.13 ml, 1.1 mmol) and the resulting solution was stirred at ambient temperature for 15 min. Then, diethyl ether (0.3 ml), aluminum (III) chloride (0.13 g, 1 mmol), and 1,2-dichloroethane (1.5 ml) were added successively and the resulting mixture was stirred at ambient temperature for 24 h. After the usual work up as described above, column chromatography [silica gel (200 mesh), hexane-benzene (3:1) as eluant] afforded 4-(phenylseleno)cyclohexene (**8**) (0.013 g, 0.05 mmol; 5%) as a mixture with diphenyl diselenide and a mixture of *trans*-1-chloro-4-(phenylseleno)-cyclohexane (**9**), *cis*-1-chloro-4-(phenylseleno)-cyclohexane (**10**), and *trans*-1-chloro-3-(phenylseleno)cyclohexane (**11**) (0.18 g, 0.66 mmol; 66%). **8**: Yellow liquid. IR (liq. film) 1650 cm^{-1} . ^1H NMR (200 MHz) δ 1.6–2.6 (m, 6H), 3.4–3.6 (m, 1H), 5.5–5.8 (m, 2H), 7.2–7.4 (m, 3H), and 7.5–7.7 (m, 2H). ^{13}C NMR (25 MHz) δ 25.5 (t), 29.8 (t), 32.7 (t), 39.2 (d), 125.8 (d), 126.7 (d), and phenyl signals. High resolution mass spectrum, M^+ . Found: m/z 238.02612. Calcd for $\text{C}_{12}\text{H}_{14}\text{Se}$: M , 238.02611. **9**, **10**, and **11**: Pale yellow oil. IR (liq. film) 1440 cm^{-1} . ^1H NMR (400 MHz) δ 1.5–2.3 (m, 8H), 3.2–3.3 (m, 1H; **9**), 3.3–3.4 (m, 1H; **10**), 3.70 (tt, 1H, $J = 9.5$ and 3.7 Hz; **11**), 3.9–4.0 (m, 1H; **9**), 4.2–4.3 (m, 1H; **10**), 4.4–4.5 (m, 1H; **11**), 7.2–7.3 (m, 3H), and 7.5–7.6 (m, 2H). ^{13}C NMR (25 MHz) δ (**9**) 32.5* (t), 36.5* (t), 40.4 (d), 58.4 (d), and phenyl signals; (**10**) 29.1* (t), 34.2* (t), 41.3 (d), 58.3 (d), and phenyl signals; (**11**) 21.6 (t), 32.7 (t), 34.3 (t), 38.1 (d), 41.7 (t), 58.9 (d), and phenyl signals (* two signals overlapping). Mass spectrum, M^+ 272, 274, and 276 (ca. 1:2:1). Found: C, 52.97; H, 5.53%. Calcd for $\text{C}_{12}\text{H}_{15}\text{ClSe}$: C, 52.66; H, 5.54%.

Preparation of the Authentic Sample of *trans*-1-Chloro-3-(phenylseleno)cyclohexane (11). Ethanol (20 ml) was added to a mixture of diphenyl diselenide (5 mmol) and sodium borohydride (12.7 mmol) under ice bath cooling under nitrogen atmosphere, and the mixture was stirred until homogeneous solution. Then, 2-cyclohexen-1-one (10 mmol) in ethanol (5 ml) was added and the resulting mixture was heated under reflux for 20 h. After the usual work up, the solvents were removed under reduced pressure and the residual oil was dissolved in ether (30 ml). Lithium aluminum hydride (5 mmol) was added to this solution and the resulting mixture was stirred at ambient temperature for 1 h. 3-(Phenylseleno)cyclohexanol (0.66 g, 2.6 mmol; 25%) was isolated after the work up and column chromatography.

To a solution of zinc(II) chloride (0.05 g, 0.4 mmol) in benzene, were added 3-(phenylseleno)cyclohexanol (0.23 g, 1 mmol) in benzene and thionyl chloride (0.19 g, 1.6 mmol) in benzene, and the resulting solution (10 ml) was stirred at ambient temperature for 1 h. After the usual work up, column chromatography afford the authentic sample of 11 (0.03 g, 0.11 mmol; 11%).

Spectral and analytical data of other cyclic compounds are as follows.

***N*-Tosyl-2,5-bis [(phenylseleno) methyl] pyrrolidine (12) and *N*-Tosyl-2-[(phenylseleno) methyl]-5-(phenylseleno)piperidine (13).** (identified as a mixture, 12:13=72:28 by ^{13}C NMR): White crystals. IR (KBr disc) 1330 and 1160 cm^{-1} . ^1H NMR (400 MHz) δ 1.4-2.0 (m, 4H), 2.34 (s, 3H; 12), 2.38 (s, 3H; 13), 2.91 (dd, 2H, $J=12.5$ and 10.5 Hz; 12), 2.9-3.1 (m, 4H; 13), 3.57 (dd, 2H, $J=12.5$ and 3.2 Hz; 12), 3.6-3.7 (m, 2H; 12), 3.89 (dd, 1H, $J=14.0$ and 3.7 Hz; 13), 4.2-4.3 (m, 1H; 13), and 7.1-7.6 (m, 14H). ^{13}C NMR (25 MHz) δ (12) 21.2 (q), 29.1* (t), 33.1* (t), 61.8* (d), and phenyl signals (* two signals overlapping); (13) 21.2 (q), 25.6 (t), 27.3 (t), 27.6 (t), 37.9 (d), 46.2 (t), 51.7 (d), and phenyl signals. Found: C, 53.26; H, 4.79; N, 2.44%. Calcd for $\text{C}_{25}\text{H}_{27}\text{NO}_2\text{SSe}_2$: C, 53.28; H, 4.84; N, 2.49%.

***N*-Tosyl-2-[(phenylseleno) methyl]-4-(phenylseleno)pyrrolidine (17).** (identified as a mixture of *cis*- and *trans*-isomer, 66:34 by ^{13}C NMR): White crystals. IR (KBr disc) 1350 and 1160 cm^{-1} . ^1H NMR (400 MHz) δ (major isomer) 1.6-1.8 (m, 1H), 2.3-2.3 (m, 1H), 2.40 (s, 3H), 2.76 (tt, 1H, $J=9.9$ and 6.7 Hz), 2.99 (dd, 1H, $J=12.9$ and 11.0 Hz), 3.36 (dd, 1H, $J=12.2$ and 9.8 Hz), 3.5-3.8 (m, 3H), and 7.1-7.6 (m, 14H): (minor isomer) 1.6-1.8 (m, 1H), 2.27 (ddd, 1H, 13.4, 6.4, and 2.7 Hz), 2.40 (s, 3H), 2.86 (dd, 1H, $J=12.5$ and 10.5 Hz), 3.03 (dd, 1H, $J=9.8$ and 8.8 Hz), 3.5-3.8 (m, 3H), 3.86 (dd, 1H, $J=10.0$ and 6.6 Hz), and 7.1-7.6 (m, 14H). ^{13}C NMR (25 MHz) δ (major isomer) 21.4 (q), 33.6 (t), 36.7 (d), 39.1 (t), 56.2 (t), 60.5 (d), and phenyl signals; (minor isomer) 21.4 (q), 32.9 (t), 35.8 (d), 38.0 (t), 56.0 (t), 59.7 (d), and phenyl signals. Found: C, 52.32; H, 4.60; N, 2.52%. Calcd for $\text{C}_{24}\text{H}_{25}\text{NO}_2\text{SSe}_2$: C, 52.46; H, 4.60; N, 2.55%.

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