

Bis-vinyl Selenides Obtained via Iron(III) Catalyzed Addition of PhSeSePh to Alkynes: Synthesis and Antinociceptive Activity

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SUPPORTING INFORMATION

Materials and Methods

Proton nuclear magnetic resonance spectra (¹H NMR) were obtained at 200 MHz on a DPX-200 NMR spectrometer or at 400 MHz on a DPX-400 NMR spectrometer. Spectra were recorded in CDCl₃ solutions. Chemical shifts are reported in ppm, referenced to the solvent peak of CDCl₃ or tetramethylsilane (TMS) as the external reference. Data are reported as follows: chemical shift (δ), multiplicity, coupling constant (*J*) in Hertz and integrated intensity. Carbon-13 nuclear magnetic resonance spectra (¹³C NMR) were obtained either at 50 MHz on a DPX-200 NMR spectrometer or at 100 MHz on a DPX-400 NMR spectrometer. Spectra were recorded in CDCl₃ solutions. Chemical shifts are reported in ppm, referenced to the solvent peak of CDCl₃. Abbreviations to denote the multiplicity of a particular signal are s (singlet), d (doublet), t (triplet), q (quartet), quint (quintet), sex (sextet), dt (double triplet), td (triple doublet) and m (multiplet). High resolution mass spectra were recorded on a Kratos MS50TC double focusing magnetic sector mass spectrometer using EI at 70 eV. Column chromatography was performed using Merck Silica Gel (230-400 mesh) following the methods described by Still.¹ Thin layer chromatography (TLC) was performed using Merck Silica Gel GF₂₅₄, 0.25 mm thickness. For visualization, TLC plates were either placed under ultraviolet light, or stained with iodine vapor, or acidic vanillin. Most reactions were monitored by TLC for disappearance of starting material. The following solvents were dried and purified by distillation from the reagents indicated: tetrahydrofuran from sodium with a benzophenone ketyl indicator. All other solvents were ACS or HPLC grade unless otherwise noted. Air- and moisture-sensitive reactions were conducted in flame-dried or oven dried glassware equipped with tightly fitted rubber septa and under a positive atmosphere of dry nitrogen or argon.

⁽¹⁾ Still, W.C., Kahn, M., Mitra, A.; *J. Org. Chem.* **1978**, *43*, 2923.

All synthesized and tested compounds were obtained in purity superior to 98% determined by combustion analysis, HPLC and gas chromatography. Reagents and solvents were handled using standard syringe techniques. Temperatures above room temperature were maintained by use of a mineral oil bath with an electrically heated coil connected to a Variac controller.

Table 1. Effect of different pretreatment times with bis-vinyl selenides administered to mice in the formalin test.

Groups	Time (min)	Formalin		Edema(mm)
		First (sec)	Second (sec)	
Control		70.5 (± 3.19)	152.3 (± 0.63)	0.06 (± 0.003)
3t				
	30	48.3* (± 6.08)	95.5 (± 29.06)	0.05 (± 0.009)
	60	31.2* (± 3.96)	37.25* (± 22.4)	0.05 (± 0.005)
3f				
	30	77 (± 12.7)	133 (± 12.7)	0.06 (± 0.006)
	60	73.4 (± 9.64)	183 (± 9.64)	0.05 (± 0.005)
3h				
	30	48* (± 2.8)	76* (± 9.01)	0.06 (± 0.005)
	60	57.3 (± 6.94)	90.7* (± 9.21)	0.05 (± 0.007)
3a				
	30	34.5* (± 5.13)	145 (± 5.13)	0.05 (± 0.004)
	60	36* (± 9.73)	131.6 (± 9.73)	0.06 (± 0.008)
3d				
	30	58.8 (± 10.64)	106* (± 9.07)	0.05 (± 0.004)
	60	44.1 (± 7.04)	96.7* (± 18.1)	0.06 (± 0.006)

Bis-vinyl selenides were administered at a dose of 50 mg/kg (i.g.) at different pre-treatment times before the formalin test. Data are reported as the mean ± S.E.M. of 5-7 animals. Asterisks denote the significance levels, when compared to the control group (One-way ANOVA followed by the Duncan's test) (*) $p < 0.05$.

Table 2. Effect of a single acute dose of bis-vinyl selenides administered to mice on parameters of toxicity.

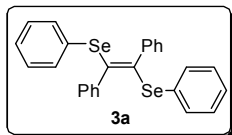
Groups	AST ^a	ALT ^a	Urea ^b	δ- ALA-D ^c		Na,K ATPase ^d
				Hepatic	Cerebral	
Control	170.73(±0.38)	50.54(±1.57)	25.5(±1.004)	24.0 (±0.30)	5.17(±0.43)	27.32(±0.50)
3t	144.68(±16.81)	51.15(±2.10)	25.0(±1.21)	22.75 (±0.73)	4.01(±0.27)	32.47(±3.75)
3f	256.43*(±29.71)	43.15(±8.14)	20.1(±2.08)	20.7*(±1.34)	5.75(±0.48)	26.8(±5.79)
3h	189.7(±13.11)	61.5(±7.52)	29.4(±1.60)	24.16 (±0.87)	5.5(±0.81)	29.7(±5.9)
3a	188.32(±20.55)	45.43(±2.80)	22.9(±2.10)	24.83(±1.625)	5.51(±0.38)	31.92(±5.52)
3d	188.27(±36.87)	47.0(±2.21)	23.9(±1.95)	24.78 (±2.25)	5.41(±0.21)	26.57(±2.24)

Bis-vinyl selenides were administered at a dose of 50 mg/kg (i.g.), 72h after administration the parameters of toxicity were determined in mice. Data are reported as the mean ± S.E.M. of 5-7 animals. Asterisks denote the significance levels, when compared to the control group (Student t test) (*) $p < 0.05$ ^a expressed as UI/l, ^b expressed as mg/dl, ^c expressed as nmol PBG/mg protein/h, ^d expressed as nmol de Pi/mg protein/min.

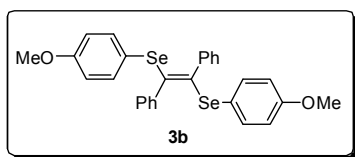
General Procedure for the Preparation of the bis-vinyl-chalcogenides.

To a Schlenk tube, under ambient atmosphere, containing a mixture of the appropriate diorganoyl dichalcogenide (0.55 equiv) in CH₃NO₂ (0.5 mL) was added, at room temperature, the FeCl₃ (0.1 equiv, 10

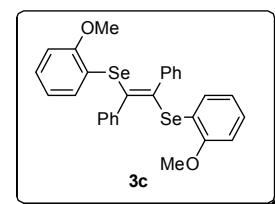
mol%). The resulting solution was stirred for 15 min at this temperature. After that the corresponding alkyne (0.5 mmol) in CH_3NO_2 (0.5 mL) was added and resulting solution was stirred under reflux for 12 hours. The reaction mixture was diluted with ethyl acetate (20 mL) and washed with a saturated solution of NH_4Cl (3 x 10 mL). The organic phase was separated, dried over MgSO_4 , and concentrated under vacuum. The residue was purified by flash chromatography eluted with hexane/acetate (95:5).



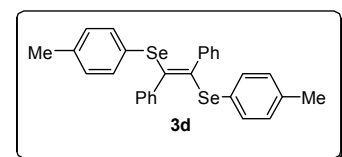
(E)-1,2-diphenyl-1,2-bis(phenylselenenyl)ethene (3a): Yield: 0.226g (92%). White solid. mp = 155-158 °C. ^1H RMN (CDCl_3 , 400 MHz), δ (ppm): 7.24-7.21 (m, 4H), 7.19-7.03 (m, 12H), 7.01-6.96 (m, 4H). RMN ^{13}C (CDCl_3 , 100 MHz), δ (ppm): 140.5, 135.0, 133.1, 130.0, 129.5, 128.3, 127.6, 127.4, 102.3. MS (relative intensity) m/z : 492 (13), 335 (25), 254 (27), 178 (100), 152 (19), 77 (10). Anal. (%) calcd. for $\text{C}_{26}\text{H}_{20}\text{Se}_2$: C 63.68, H 4.110. Found: C 63.74, H 4.18.



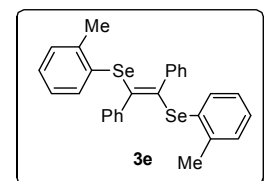
(E)-1,2-bis(4-methoxyphenylselenenyl)-1,2-diphenylethene(3b): Yield: 0.165g (60%). Yellow solid. mp = 164-167 °C. ^1H RMN (CDCl_3 , 400 MHz), δ (ppm): 7.20-7.11 (m, 10H), 7.08-7.05 (m, 4H), 6.54-6.49 (m, 4H), 3.68 (s, 6H). ^{13}C RMN (CDCl_3 , 50 MHz), δ (ppm): 159.9, 140.5, 137.2, 132.5, 129.4, 127.7, 127.2, 120.3, 113.9, 55.1. MS (relative intensity) m/z : 552 (9), 373 (7), 365 (25), 281 (30), 253 (25), 207 (100), 186 (58), 178 (75), 133 (17), 77 (99). Anal. HRMS calcd. for $\text{C}_{28}\text{H}_{24}\text{O}_2\text{Se}_2$ ($\text{M}+\text{Na}^+$): 552.0107. Found: 552.0113.



(E)-1,2-bis(2-methoxyphenylselenenyl)-1,2-diphenylethene (3c): Yield: 0.196g (71%). White solid. mp = 156-159 °C. ^1H RMN (CDCl_3 , 200 MHz), δ (ppm): 7.37-7.25 (m, 6H), 7.16-7.07 (m, 8H), 6.71-6.63 (m, 2H), 6.58-6.53 (m, 2H), 3.69 (s, 6H). ^{13}C RMN (CDCl_3 , 50 MHz), δ (ppm): 158.2, 141.2, 135.5, 133.5, 129.4, 129.0, 128.9, 127.3, 127.2, 120.7, 119.9, 110.2, 55.5. MS (relative intensity) m/z : 551 (17), 365 (29), 284 (14), 207 (21), 178 (100), 159 (17), 107 (36), 77 (33). Anal. HRMS calcd. for $\text{C}_{28}\text{H}_{24}\text{O}_2\text{Se}_2$ ($\text{M}+\text{Na}^+$): 551.0107. Found: 552.0117.

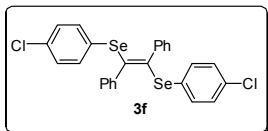


(E)-1,2-diphenyl-1,2-bis(p-tolylselenenyl)ethene (3d): Yield: 0.195g (75%). Yellow solid. mp = 133-136 °C. ^1H RMN (CDCl_3 , 200 MHz), δ (ppm): 7.32-7.08 (m, 10H), 7.05 (d, $J = 8.1$ Hz, 4H), 6.79 (d, $J = 7.8$ Hz, 4H), 2.17 (s, 6H). ^{13}C RMN (CDCl_3 , 100 MHz), δ (ppm): 140.8, 137.3, 135.1, 133.0, 125.5, 129.1, 129.6, 127.3, 126.4, 21.0. MS (relative intensity) m/z : 518 (16), 353 (7), 372 (16), 353 (12), 207 (28), 178 (100), 152 (22), 133 (5), 76 (4). Anal. (%) calcd. for $\text{C}_{28}\text{H}_{24}\text{Se}_2$: C 64.87, H 4.67. Found: C 64.96, H 4.72.

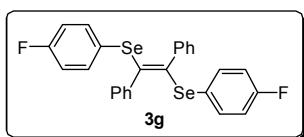


(E)-1,2-diphenyl-1,2-bis(o-tolylselenenyl)ethene (3e): Yield: 0.182g (70%). White solid. mp = 190-193 °C. ^1H RMN (CDCl_3 , 400 MHz), δ (ppm): 7.25-7.20 (m, 5H), 7.16-7.09 (m, 5H), 6.97-6.95 (m, 2H), 6.84-6.80 (m, 2H), 2.23 (s, 6H). ^{13}C RMN (CDCl_3 , 50 MHz), δ (ppm): 141.3, 140.9, 136.6, 133.5, 131.0, 129.5, 129.0,

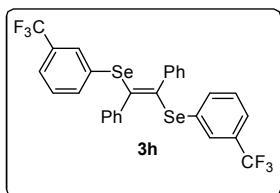
128.1, 127.48, 127.3, 125.8, 22.8. MS (relative intensity) m/z : 520 (7), 349 (16), 281 (27), 253 (28), 107 (100), 178 (80), 152 (19), 133 (17), 91 (66), 73 (40). Anal. (%) calcd. for $C_{28}H_{24}Se_2$: C 64.87, H 4.67. Found: C 64.93, H 4.71.



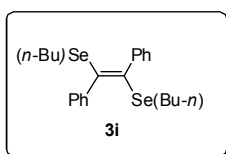
(E)-1,2-bis(4-chlorophenylselenenyl)-1,2-diphenylethene (3f): Yield: 0.179g (65%). Yellow solid. mp = 178-181 °C. 1H RMN ($CDCl_3$, 200 MHz), δ (ppm): 7.23-7.13 (m, 10H), 7.08 (d, $J = 8.3$ Hz, 4H), 6.96 (d, $J = 8.6$ Hz, 4 H). ^{13}C RMN ($CDCl_3$, 100 MHz), δ (ppm): 140.2, 136.3, 133.9, 133.1, 129.4, 129.1, 128.1, 129.9, 127.7. MS (relative intensity) m/z : 553 (5), 348 (15), 281 (927), 253 (21), 214 (12), 207 (100), 176 (28), 151 (10), 96 (3), 91 (54), 73 (40). Anal. HRMS calcd. for $C_{26}H_{18}Cl_2Se_2$ ($M+Na^+$): 559.9113. Found: 559.9141.



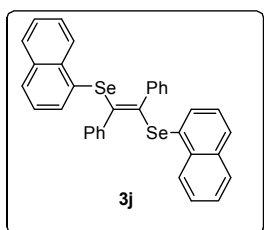
(E)-1,2-bis(4-fluorophenylselenenyl)-1,2-diphenylethene (3g): Yield: 0.198g (75%). White solid. mp = 134-137 °C. 1H RMN ($CDCl_3$, 400 MHz), δ (ppm): 7.22-7.01 (m, 14H), 6.71-6.65 (m, 4H), ^{13}C RMN ($CDCl_3$, 100 MHz), δ (ppm): 162.5 (d, $^1J = 248$ Hz), 140.2, 135.5 (d, $^3J = 8.7$ Hz), 132.7, 128.4, 127.8, 127.6, 124.5 (d, $^4J = 3.6$ Hz), 115.5 (d, $^2J = 21.9$ Hz). MS (relative intensity) m/z : 528 (8), 353 (20), 281 (4), 253 (28), 207 (100), 178 (80), 152 (19), 133 (917), 91 (66), 73 (40). Anal. (%) calcd. for $C_{26}H_{18}F_2Se_2$: C 59.33, H 3.45. Found: C 59.48, H 3.51.



(E)-1,2-diphenyl-1,2-bis(3-(trifluoromethyl)phenylselenenyl)ethene (3h): Yield: 0.255g (81%). White solid. mp = 128-131 °C. 1H RMN ($CDCl_3$, 400 MHz), δ (ppm): 7.39-7.35 (m, 4H), 7.32-7.29 (m, 2H), 7.25-7.08 (m, 12H). ^{13}C RMN ($CDCl_3$, 100 MHz), δ (ppm): 139.8, 138.1, 133.0, 131.8 (q, $J = 4.4$ Hz), 131.0, 130.7 (q, $J = 32.2$ Hz), 129.4, 128.6, 128.0, 127.9, 124.2 (q, $J = 3.6$ Hz), 123.5 (q, $J = 272.2$ Hz). MS (relative intensity) m/z : 628 (3), 625 (7), 403 (16), 363 (9), 322 (8), 178 (100), 152 (13), 126 (34). Anal. HRMS calcd. for $C_{28}H_{18}F_6Se_2$ ($M+Na^+$): 697.9643. Found: 697.9758.

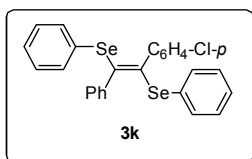


(E)-1,2-bis(butylselenenyl)-1,2-diphenylethene (3i): Yield: 0.132g (58%). Yellow solid. mp = 80-83 °C. 1H RMN ($CDCl_3$, 400 MHz), δ (ppm): 7.41-7.35 (m, 8H), 7.32-7.28 (m, 2H), 2.14 (qui, $J = 7.3$ Hz, 4H), 1.33 (qui, $J = 7.6$ Hz, 4H), 1.12 (sex, $J = 7.6$ Hz, 4H), 0.71 (t, $J = 7.6$ Hz, 6H). ^{13}C RMN ($CDCl_3$, 100 MHz), δ (ppm): 141.1, 130.6, 129.5, 128.1, 127.5, 32.3, 27.0, 22.7, 13.4. MS (relative intensity) m/z : 352 (10), 304 (23), 228 (21), 207 (53), 178 (84), 152 (18), 91 (5), 55 (14). Anal. HRMS calcd. for $C_{22}H_{28}Se_2$ ($M+Na^+$): 452.0521. Found: 452.0530.

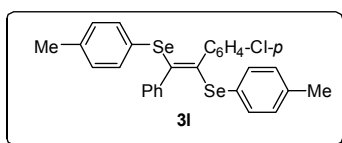


(E)-1,2-bis(naphthalen-1-ylselenenyl)-1,2-diphenylethene (3j): Yield: 0.151g (51%). White solid. mp = 167-170 °C. 1H RMN ($CDCl_3$, 200 MHz), δ (ppm):

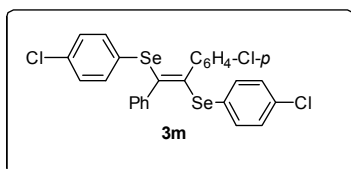
8.19 -8.12 (m, 2H), 7.71-7.54 (m, 4H), 7.51-7.38 (m, 6H), 7.12-6.95 (m, 12H). ^{13}C RMN (CDCl_3 , 100 MHz), δ (ppm): 150.5, 135.7, 134.9, 133.7, 133.5, 129.5, 129.1, 129.0, 128.4, 128.2, 127.2, 126.3, 125.8, 125.1. MS (relative intensity) m/z : 392 (5), 304 (23), 228 (21), 207 (53), 178 (84), 152 (18), 128 (22), 126 (19) 115 (100), 77 (7). Anal. (%) calcd. for $\text{C}_{34}\text{H}_{24}\text{Se}_2$: C 69.16, H 4.10. Found: C 69.28, H 4.17.



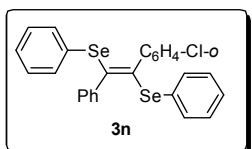
(E)-(1-(4-chlorophenyl)-2-phenylethene-1,2-diyl)bis(phenylselene) (3k): Yield: 0.255g (91%). White solid. mp = 80-83 °C. ^1H RMN (CDCl_3 , 400 MHz), δ (ppm): 7.22-7.10 (m, 14H), 6.72-6.65 (m, 4H). ^{13}C RMN (CDCl_3 , 50 MHz), δ (ppm): 140.5, 139.2, 135.1, 135.6, 134.7, 134.0, 133.2, 131.4, 130.9, 129.9, 129.4, 128.8, 128.6, 128.4, 127.9, 127.7, 127.6. MS (relative intensity) m/z : 525 (9), 368 (14), 333 (27), 331 (15), 252 (22), 211 (100), 176 (80), 156 (22), 150 (26), 77 (14). Anal. (%) calcd. for $\text{C}_{26}\text{H}_{19}\text{ClSe}_2$: C 59.60, H 3.65. Found: C 59.62, H 3.69.



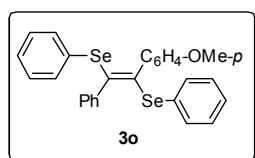
(E)-(1-(4-chlorophenyl)-2-phenylethene-1,2-diyl)bis(p-tolylselene) (3l): Yield: 0.177g (64%). White solid. mp = 135-138 °C. ^1H RMN (CDCl_3 , 200 MHz), δ (ppm): 7.23-6.99 (m, 13H), 6.88-6.77 (m, 4H), 2.22-1.14 (m, 6H). ^{13}C RMN (CDCl_3 , 100 MHz), δ (ppm): 140.7, 139.4, 137.7, 137.6, 135.2, 134.1, 133.0, 131.8, 130.9, 130.8, 129.5, 129.4, 129.2, 128.8, 127.8, 127.7, 127.4, 126.6, 126.2, 21.0 (2C). MS (relative intensity) m/z : 556 (5), 553 (10), 383 (19), 383 (19), 348 (31), 281 (16), 268 (10), 212 (86), 176 (59), 170 (20), 151 (21), 83 (10), 91 (100), 73 (25). Anal. HRMS calcd. for $\text{C}_{29}\text{H}_{26}\text{ClSe}_2$ ($\text{M}+\text{Na}^+$): 553.9819. Found: 553.9829.



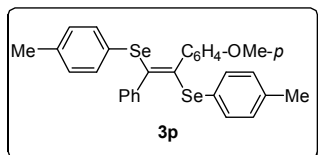
(E)-(1-(4-chlorophenyl)-2-phenylethene-1,2-diyl)bis(4-chlorophenylselene) (3m): Yield: 0.232g (78%). White solid. mp = 131-139 °C. RMN ^1H (CDCl_3 , 400 MHz), δ (ppm): 7.23-7.14 (m, 9H), 7.01-7.05 (m, 4H), 7.02-6.95 (m, 4H). RMN ^{13}C (CDCl_3 , 100 MHz), δ (ppm): 140.2, 138.8, 136.5, 136.27, 134.6, 134.3, 134.3, 133.7, 130.8, 129.4, 129.9, 128.7, 128.2, 127.9, 127.8. MS (relative intensity) m/z : 596 (4), 593 (5), 403 (12), 370 (10), 366 (11), 281 (17), 153 (16), 212 (100), 207 (62), 190 (21), 176 (70), 156 (14), 133 (11), 77 (7). Anal. HRMS calcd. for $\text{C}_{26}\text{H}_{17}\text{Cl}_3\text{Se}_2$ ($\text{M}+\text{Na}^+$): 593.8726. Found: 593.8738.



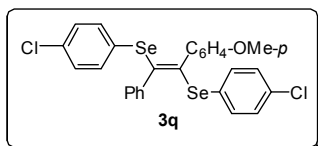
(E)-(1-(4-chlorophenyl)-2-phenylethene-1,2-diyl)bis(phenylselene) (3n): Yield: 0.205g (78%). White solid. mp = 115-118 °C. ^1H RMN (CDCl_3 , 400 MHz), δ (ppm): 7.36-7.15 (m, 10H), 7.12-6.96 (m, 9H). ^{13}C RMN (CDCl_3 , 100 MHz), δ (ppm): 139.9, 138.8, 136.6, 134.98, 132.8, 132.7, 131.2, 131.1, 129.7, 129.5, 129.3, 128.7, 128.6, 128.4, 128.3, 128.1, 127.8, 127.7, 127.4, 125.9. MS (relative intensity) m/z : 525 (5), 341 (12), 334 (16), 332 (10), 282 (10), 212 (63), 207 (100), 176 (49), 116 (16), 77 (19). Anal. HRMS calcd. for $\text{C}_{26}\text{H}_{19}\text{ClSe}_2$ ($\text{M}+\text{Na}^+$): 525.9506. Found: 525.9533.



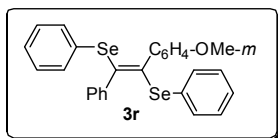
(E)-(1-(4-methoxyphenyl)-2-phenylethene-1,2-diyl)bis(phenylselenene) (3o): Yield: 0.188g (72%). Yellow solid. mp = 52-55 °C. ¹H RMN (CDCl₃, 400 MHz), δ (ppm): 7.28-6.94 (m, 16 H), 6.9-6.84 (m, 1H), 6.76-6.63 (m, 2H). ¹³C RMN (CDCl₃, 100 MHz), δ (ppm): 158.9, 140.8, 135.1, 134.2, 134.0, 133.2, 131.4, 130.9, 130.5, 130.3, 130.17, 128.5, 128.6, 128.3, 127.6, 127.2, 126.2, 113.1, 55.1. MS (relative intensity) *m/z*: 522 (11), 365 (42), 284 (12), 208 (100), 193 (62), 165 (66), 139 (9), 77 (9). Anal. HRMS calcd. for C₂₇H₂₂OSe₂ (M+Na⁺): 522.0001. Found: 522.0012.



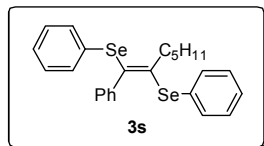
(Z)-(1-(4-methoxyphenyl)-2-phenylethene-1,2-diyl)bis(p-tolylselenene) (3p): Yield: 0.173g (63%). Yellow solid. mp = 121-123 °C. ¹H RMN (CDCl₃, 400 MHz), δ (ppm): 7.24-7.09 (m, 7H), 7.06-7.03 (m, 4H), 6.82-7.78 (m, 4H), 6.719-6.69 (m, 2H), 3.74 (s, 3H), 2.19 (s, 6H). ¹³C RMN (CDCl₃, 50 MHz), δ (ppm): 158.6, 140.8, 137.3, 137.1, 135.2, 137.7, 133.2, 132.5, 130.8, 129.5, 129.2, 129.1, 127.5, 127.2, 126.7, 126.4, 113.0, 55.1, 21.1. MS (relative intensity) *m/z*: 550 (14), 348 (13), 379 (52), 364 (9), 298 (17), 208 (100), 193 (78), 165 (79), 139 (10), 91 (35). Anal. (%) calcd. for C₂₉H₂₆OSe₂: C 63.51, H 4.78. Found: C 63.74, H 4.82.



(E)-(1-(4-methoxyphenyl)-2-phenylethene-1,2-diyl)bis(4-chlorophenylselenene) (3q): Yield: 0.224g (76%). Yellow solid. mp = 80-86 °C. ¹H RMN (CDCl₃, 400 MHz), δ (ppm): 7.25-7.05 (m, 10H), 7.03-6.92 (m, 4H), 6.83-6.81 (m, 1H), 6.76-6.68 (m, 2H), 3.73 (s, 3H). ¹³C RMN (CDCl₃, 50 MHz), δ (ppm): 159.0, 141.3, 140.0, 136.3, 136.2, 133.9, 133.8, 129.3, 128.9, 128.5(2C), 128.1, 128.0, 127.8, 127.7, 121.9, 114.8, 113.5, 55.2. MS (relative intensity) *m/z*: 591 (4), 401 (13), 399 (29), 364 (19), 208 (100), 193 (60), 165 (64), 139 (9), 73 (11). Anal. HRMS calcd. for C₂₇H₂₀Cl₂OSe₂ (M+Na⁺): 589.9222. Found: 589.9230.

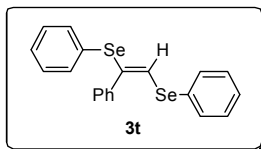


(E)-(1-(3-methoxyphenyl)-2-phenylethene-1,2-diyl)bis(phenylselenene) (3r): Yield: 0.162g (62%). White solid. mp = 112-115 °C. RMN ¹H (CDCl₃, 400 MHz), δ (ppm): 7.24- 6.97 (m, 16H), 6.87-6.85 (m, 1H), 6.72 (s, 1H), 6.68-6.65 (m, 1H), 3.69 (s, 3H). RMN ¹³C (CDCl₃, 100 MHz), δ (ppm): 158.9, 141.8, 140.5, 135.1, 134.3, 134.0, 128.7, 127.5, 127.4 (2C), 122.0, 114.7, 113.6, 55.2. MS (relative intensity) *m/z*: 522 (23), 520 (21), 350 (13), 313 (10), 285 (19), 253 (17), 208 (100), 178 (73), 165 (65), 152 (14), 77 (23). Anal. HRMS calcd. for C₂₇H₂₂OSe₂: C 62.32, H 4.26. Found: C 62.45, H 4.30.

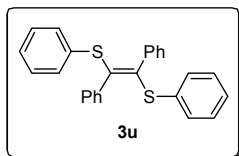


(E)-(1-phenylhept-1-ene-1,2-diyl)bis(phenylselenene) (3s): Yield: 0.206g (85%). Yellow oil. ¹H RMN (CDCl₃, 400 MHz), δ (ppm): 7.48-7.45 (m, 2H), 7.29-7.23 (m, 5H), 7.13-7.05 (m, 8H), 2.64 (t, *J* = 7.8 Hz, 2H), 1.57 (qui, *J* = 7.6 Hz, 2H), 1.27-1.19 (m, 4H), 0.85 (t, *J* = 6.6 Hz, 3H). ¹³C RMN (CDCl₃, 100 MHz), δ (ppm): 141.8, 137.3, 134.6, 134.2, 131.3, 130.5, 130.3, 129.5, 129.9, 128.6, 127.6, 127.5, 127.2, 127.1, 37.7, 31.3, 29.0, 22.4, 13.9. MS (relative intensity) *m/z*: 488 (4), 486 (13), 258 (35), 207(17), 178 (100), 156 (17), 143 (40), 129 (48), 115

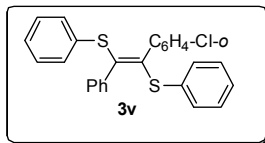
(78), 91 (64), 71 (22), 65 (6), 55 (7), 51 (6). Anal. HRMS calcd. for $C_{25}H_{26}Se_2$: C 486.0365. Found: 486.0378.



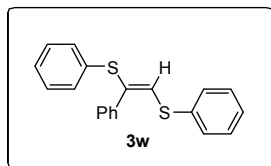
(E)-(1-phenylethene-1,2-diyl)bis(phenylselenene) (3t): Yield: 0.124g (60%). Yellow oil. 1H RMN ($CDCl_3$, 400 MHz), δ (ppm): 7.55-7.14 (m, 15H), 7.07 (s, 1H). ^{13}C RMN ($CDCl_3$, 50 MHz), δ (ppm): 139.5, 133.0, 132.1, 131.1, 130.6, 130.4, 129.3, 129.2, 128.6, 128.3, 128.4, 127.5, 127.4, 126.0. MS (relative intensity) m/z : 415 (9), 280 (10), 258 (19), 252 (9), 206 (25), 178 (100), 156 (48), 151 (10), 77 (66). Anal. HRMS calcd. for $C_{20}H_{16}Se_2$: C 415.9582. Found: 415.9602.



(E)-1,2-diphenyl-1,2-bis(phenylthio)ethene (3u): Yield: 0.123g (62%). White solid. mp = 106-106 °C. 1H RMN ($CDCl_3$, 400 MHz), δ (ppm): 7.49-7.37 (m, 3H), 7.19-6.99 (m, 16H), 7.00-6.89 (m, 1H). ^{13}C RMN ($CDCl_3$, 100 MHz), δ (ppm): 138.9, 134.4, 131.7, 130.6, 129.9, 128.1, 128.3, 127.3, 126.8. MS (relative intensity) m/z : 396 (49), 287 (30), 271 (13), 253 (41), 209 (51), 206 (56), 178 (100), 165 (43), 152 (42), 108 (29), 77 (18). Anal. HRMS calcd. for $C_{26}H_{20}S_2$ ($M+Na^+$): 396.1006. Found: 396.1019.



(E)-(1-(2-chlorophenyl)-2-phenylethene-1,2-diyl)bis(phenylsulfide) (3v): Yield: 0.091g (42%). White solid. mp = 43-46 °C. 1H RMN ($CDCl_3$, 400 MHz), δ (ppm): 7.54-6.99 (m, 19H). ^{13}C RMN ($CDCl_3$, 100 MHz), δ (ppm): 137.6, 137.0, 134.7, 134.2, 133.3, 132.5, 132.0, 131.5, 130.1, 129.6, 129.3, 129.0, 128.8, 128.3 (2C), 127.8, 127.7, 127.1, 126.0. MS (intensidade relativa) m/z : 430 (3), 341 (5), 281 (21), 253 (17), 207 (100), 190 (16), 133 (17), 96 (15), 73 (26). Anal. HRMS calcd. for $C_{26}H_{19}ClS_2$: 430.0617. Found: 430.0629.



(E)-(1-phenylethene-1,2-diyl)bis(phenylsulfide) (3w): Yield: 0.064g (40%). colorless oil. 1H RMN ($CDCl_3$, 400 MHz), δ (ppm): 7.60-7.56 (m, 2 H), 7.34-7.17 (m, 15 H), 7.14-7.09 (m, 1H), 6.89 (s, 1H). ^{13}C RMN ($CDCl_3$, 50 MHz), δ (ppm): 138.9, 137.4, 134.4, 134.2, 131.7, 131.5, 130.6, 129.9, 128.5, 128.3, 127.6 (2C), 127.3, 126.7. MS (relative intensity) m/z : 331 (4), 281 (12), 253 (15), 209 (13), 207 (100), 191 (13), 123 (14), 96 (14), 73 (7). Anal.

SELECTED SPETRA

