

Antidepressant-like Activity of 2,5-Disubstituted- 3-selenophenes Obtained via Iron(III)/PhSeSePh- Mediated Cyclization of Z-Selenoenynes

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SUPPORTING INFORMATION

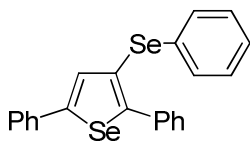
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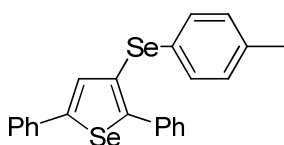
Materials and Methods

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 CDCl_3 solutions. Chemical shifts are reported in ppm, referenced to the solvent peak of CDCl_3 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 (^{13}C 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 CDCl_3 solutions. Chemical shifts are reported in ppm, referenced to the solvent peak of CDCl_3 . 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. 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.

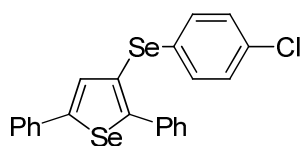
⁽¹⁾ Still, W.C., Kahn, M., Mitra, A.; *J. Org. Chem.* **1978**, *43*, 2923.



2,5-diphenyl-3-(phenylseleno)-selenophene 2a.² Yield Condition A: 0.081 g (74%). Yield Condition B: 0.074 g (67%). ¹H NMR (CDCl₃, 400 MHz): δ 7.56-7.54 (m, 2H), 7.49-7.47 (m, 2H), 7.40-7.19 (m, 12H). ¹³C NMR (CDCl₃, 100 MHz): δ 149.44, 135.90, 135.52, 132.43, 131.03, 129.32, 129.24, 128.95, 128.33, 128.24, 128.13, 127.94, 127.86, 126.67, 126.05, 122.16. MS (relative intensity) *m/z*: 439 (100), 284 (46), 207 (58), 156 (75), 129 (37), 77 (21). HRMS calcd for C₂₂H₁₆Se₂: 439.9582. Found: 439.9585.

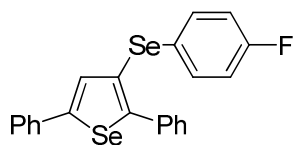


2,5-diphenyl-3-(4-methylphenylseleno)-selenophene 2b.² Yield Condition A: 0.092 g (81%). Yield Condition B: 0.081 g (71%). ¹H NMR (CDCl₃, 400 MHz): δ 7.55-7.46 (m, 4H), 7.38-7.22 (m, 9H), 7.15-7.09 (m, 2H), 2.33 (s, 3H). ¹³C NMR (CDCl₃, 100 MHz): δ 149.46, 138.07, 135.91, 135.50, 133.49, 132.49, 131.44, 130.98, 130.08, 129.21, 128.91, 128.31, 127.91, 126.74, 126.01, 121.73, 21.83. MS (relative intensity) *m/z*: 454 (56), 284 (79), 202 (100), 169 (6), 126 (7), 91 (18), 77 (7). HRMS calcd for C₂₃H₁₈Se₂: 453.9739. Found: 453.9743.



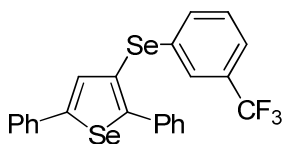
2,5-diphenyl-3-(4-chlorophenylseleno)-selenophene 2d.² Yield Condition A: 0.093 g (79%). Yield Condition B: 0.065 g (55%). ¹H NMR (CDCl₃, 400 MHz): δ 7.52-7.46 (m, 4H), 7.38-7.24 (m, 9H), 7.16 (d, *J* = 8.53 Hz, 2H). ¹³C NMR (CDCl₃, 100 MHz): δ 149.88, 149.78, 135.68, 135.34, 132.74, 132.17, 132.15, 130.86, 129.30, 129.26, 128.95, 128.34, 128.21, 128.03, 126.01, 121.65. MS (relative intensity) *m/z*: 474 (47), 394 (51), 358 (15), 282 (20), 202 (100), 77 (8). HRMS calcd for C₂₂H₁₅ClSe₂: 473.9193. Found: 473.9196

² Stein, A. L.; Alves, D.; Rocha, J. T.; Nogueira, C. W.; Zeni, G. *Org. Letters* **2008**, *10*, 4983.



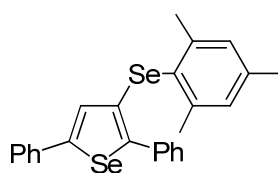
2,5-diphenyl-3-(4-fluorophenylseleno)-selenophene 2c.² Yield

Condition A: 0.089 g (78%). Yield Condition B: 0.084 g (73%). ¹H NMR (CDCl₃, 200 MHz): δ 7.55-7.27 (m, 13H), 6.93 (t, *J* = 8.80 Hz, 2H). ¹³C NMR (CDCl₃, 100 MHz): δ 162.16 (d, *J* = 246.63 Hz), 149.56, 148.47, 135.62 (d, *J* = 38.05 Hz), 133.70 (d, *J* = 7.32 Hz), 131.75, 131.39, 129.26, 128.95, 128.36, 128.14, 127.99, 126.57, 126.02, 122.72, 116.39 (d, *J* = 21.95). MS (relative intensity) *m/z*: 458 (48), 378 (50), 282 (26), 220 (15), 202 (100), 176 (5), 96 (6), 77 (7). HRMS calcd for C₂₂H₁₅FSe₂: 457.9488. Found: 457.9490.



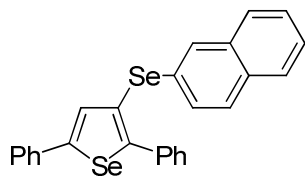
2,5-diphenyl-3-(3-trifluoromethylphenylseleno)-selenophene 2e.²

Yield Condition A: 0.084 g (66%). Yield Condition B: 0.072 g (57%). ¹H NMR (CDCl₃, 400 MHz): δ 7.57 (s, 1H), 7.52-7.25 (m, 14H). ¹³C NMR (CDCl₃, 100 MHz): δ 151.06, 150.11, 135.58, 135.32, 134.10, 133.60, 132.27, 131.33 (q, *J* = 32.20 Hz), 129.45, 129.30, 128.99, 128.35, 128.32, 128.11, 126.97 (q, *J* = 3.66 Hz), 126.05, 123.66 (q, *J* = 272.97 Hz), 123.20 (q, *J* = 3.66 Hz), 120.76. MS (relative intensity) *m/z*: 508 (51), 428 (43), 347 (8), 282 (23), 202 (100), 126 (7), 89 (11), 77 (6). HRMS calcd for C₂₃H₁₅F₃Se₂: 507.9456. Found: 507.9459.



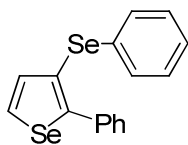
2,5-diphenyl-3-(2,4,6-trimethylphenylseleno)-selenophene 2f.²

Yield Condition B: 0.060 g (50%). ¹H NMR (CDCl₃, 400 MHz): δ 7.42-7.19 (m, 11H), 6.91 (s, 2H), 2.40 (s, 6H), 2.56 (s, 3H). ¹³C NMR (CDCl₃, 100 MHz): δ 148.69, 142.78, 141.85, 138.70, 136.37, 135.72, 129.38, 128.83, 128.81, 128.78, 128.43, 128.28, 128.26, 127.69, 126.03, 125.26, 24.31, 20.98. MS (relative intensity) *m/z*: 482 (45), 401 (6), 284 (100), 202 (71), 119 (19), 105 (10), 91 (21), 77 (12). HRMS calcd for C₂₅H₂₂Se₂: 482.0052. Found: 482.0057.



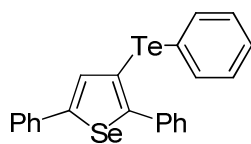
2,5-diphenyl-3-(2-naphthylseleno)-selenophene 2g.² Yield

Condition B: 0.072 g (59%). ¹H NMR (CDCl₃, 400 MHz): δ 7.85 (s, 1H), 7.68-7.66 (m, 2H), 7.58-7.56 (m, 2H), 7.46-7.23 (m, 13H). ¹³C NMR (CDCl₃, 100 MHz): δ 149.53, 149.27, 135.87, 135.44, 133.96, 132.32, 132.08, 130.02, 129.60, 129.29, 128.90, 128.82, 128.63, 128.34, 128.12, 127.91, 127.72, 127.25, 126.43, 126.01, 125.87, 122.18. MS (relative intensity) *m/z*: 488 (49), 410 (55), 329 (36), 282 (18), 202 (100), 126 (20), 77 (9), 51 (9). HRMS calcd for C₂₆H₁₈Se₂: 489.9739. Found: 489.9743.



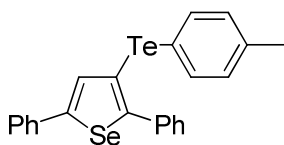
2-phenyl-3-(phenylseleno)-selenophene 2n.² Yield Condition A: 0.066 g

(73%). ¹H NMR (CDCl₃, 400 MHz): δ 7.88 (d, *J* = 5.87 Hz, 1H), 7.51-7.49 (m, 2H), 7.36-7.16 (m, 9H). ¹³C NMR (CDCl₃, 100 MHz): δ 149.85, 136.89, 135.94, 132.52, 131.19, 130.02, 129.38, 129.13, 128.25, 127.98, 126.63, 121.40. MS (relative intensity) *m/z*: 364 (65), 284 (62), 202 (47), 126 (59), 115 (100), 77 (33), 51 (18). HRMS calcd for C₁₆H₁₂Se₂: 363.9269. Found: 363.9271.



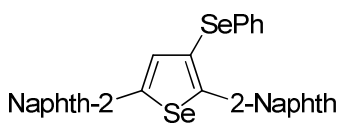
2,5-diphenyl-3-(phenyltelluro)-selenophene 2h.² Yield Condition A:

0.044 g (36%). Yield Condition B: 0.028 g (23%). ¹H NMR (CDCl₃, 400 MHz): δ 7.65-7.63 (m, 2H), 7.48-7.17 (m, 14H). ¹³C NMR (CDCl₃, 50 MHz): δ 151.22, 150.87, 137.48, 135.56, 135.34, 129.43, 129.12, 129.01, 128.89, 128.36, 128.16, 127.79, 127.74, 126.15, 115.44, 107.58. MS (relative intensity) *m/z*: 488 (29), 360 (31), 282 (15), 202 (100), 77 (22), 51 (9). HRMS calcd for C₂₂H₁₆SeTe: 489.9479. Found: 489.9481.



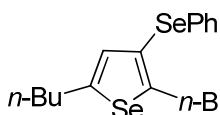
2,5-diphenyl-3-(4-methylphenyltelluro)-selenophene 2i.² Yield

Condition A: 0.054 g (43%). ¹H NMR (CDCl₃, 200 MHz): δ 7.58 (d, *J* = 7.94 Hz, 2H), 7.50-7.24 (m, 11H), 7.02 (d, *J* = 7.94 Hz, 2H), 2.32 (s, 3H). ¹³C NMR (CDCl₃, 100 MHz): δ 151.02, 149.88, 138.22, 137.95, 137.61, 135.68, 134.97, 130.38, 129.09, 128.86, 128.38, 128.10, 127.74, 126.20, 110.98, 107.98, 21.20. MS (relative intensity) *m/z*: 502 (30), 374 (40), 282 (14), 202 (100), 91 (24), 77 (5). HRMS calcd for C₂₃H₁₈SeTe: 503.9636. Found: 503.9639.



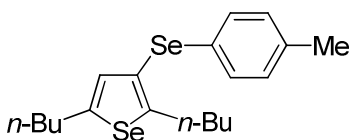
2,5-di(naphthalen-2-yl)-3-(phenylseleno)-selenophene 2l.

Yield Condition A: 0.11 g (82%). ¹H NMR (CDCl₃, 200 MHz): δ 8.37-8.32 (m, 1H), 8.08-8.03 (m, 1H), 7.89-7.78 (m, 4H), 7.61-7.36 (m, 11H), 7.19-7.11 (m, 3H). ¹³C NMR (CDCl₃, 50 MHz): δ 148.14, 147.03, 134.78, 133.85, 133.55, 133.52, 133.33, 132.27, 132.12, 131.15, 128.99, 128.96, 128.74, 128.56, 128.31, 128.22, 127.95, 126.85, 126.50, 126.38, 16.35, 126.14, 126.06, 126.03, 125.62, 125.55, 125.20, 124.96. MS (EI, 70 eV) *m/z* (relative intensity): 539 (32), 459 (11), 379 (18), 301 (100), 236 (5), 188 (9), 150 (18), 77 (10), 55 (6). HRMS calcd for C₃₀H₂₀Se₂: 539.9895. Found: 539.9907.



2,5-dibutyl-3-(phenylseleno)-selenophene 2w. Yield Condition A:

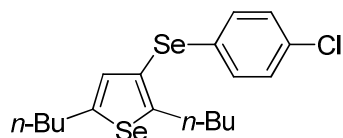
0.067 g (67%). ¹H NMR (CDCl₃, 400 MHz): δ 7.25-7.13 (m, 5H), 6.80 (s, 1H), 2.93 (t, *J* = 7.7 Hz, 2H), 2.79 (t, *J* = 7.7 Hz, 2H), 1.62-1.55 (m, 4H), 1.47-1.28 (m, 4H), 0.95-0.85 (m, 6H). ¹³C NMR (CDCl₃, 50 MHz): δ 153.63, 159.88, 132.54, 129.02, 125.85, 119.52, 34.99, 34.30, 32.37, 32.27, 22.24, 22.11, 13.82, 13.78. MS (EI, 70 eV) *m/z* (relative intensity): 399 (100), 356 (74), 276 (39), 242 (62), 200 (36), 153 (30), 77 (46). Anal. (%) Calcd for C₁₈H₂₄Se₂: C 54.28; H 6.07. Found: C 54.40, H 6.10.



2,5-dibutyl-3-(4-tolylphenylseleno)-selenophene 2q. Yield

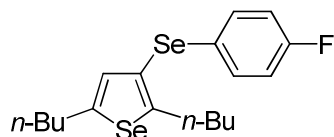
Condition A: 0.060 g (58%). ¹H NMR (CDCl₃, 200 MHz): δ 7.12 (d, *J* = 7.9 Hz, 2H), 7.01

(d, $J = 7.9$ Hz, 2H), 6.77 (s, 1H), 2.92 (t, $J = 7.7$ Hz, 2H), 2.76 (t, $J = 7.7$ Hz, 2H), 2.28 (s, 3H), 1.64-1.55 (m, 4H), 1.42-1.32 (m, 4H), 0.93-0.88 (m, 6H). C^{13} NMR ($CDCl_3$, 50 MHz): δ 152.72, 149.69, 153.85, 132.37, 130.19, 129.84, 129.35, 120.08, 34.99, 34.31, 32.34, 32.27, 22.25, 22.11, 20.95, 13.84, 13.78. MS (EI, 70 eV) m/z (relative intensity): 413 (100), 370 (52), 290 (47), 242 (61), 200 (32), 91 (89), 77 (30). Anal. (%) Calcd for $C_{19}H_{26}Se_2$: C 55.34; H 6.36. Found: C 55.41, H 6.4.



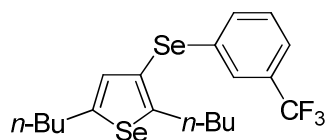
2,5-dibutyl-3-(4-chlorophenylseleno)-selenophene 2r. Yield

Condition A: 0.067 g (62%). H^1 NMR ($CDCl_3$, 200 MHz): δ 7.25-7.10 (m, 4H), 6.78 (s, 1H), 2.91 (t, $J = 7.7$ Hz, 2H), 2.79 (t, $J = 7.7$ Hz, 2H), 1.70-1.51 (m, 4H), 1.47-1.25 (m, 4H), 0.96-0.85 (m, 6H). C^{13} NMR ($CDCl_3$, 50 MHz): δ 154.08, 150.28, 134.27, 132.26, 131.79, 130.77, 129.36, 129.12, 119.11, 35.01, 34.30, 32.35, 22.23, 22.12, 13.83, 13.80. MS (EI, 70 eV) m/z (relative intensity): 434 (40), 391 (71), 311 (33), 247 (24), 243 (71), 201 (55), 153 (34), 91 (100), 77 (80). HRMS calcd for $C_{18}H_{23}ClSe_2$: 433.9819. Found: 433.9831.



2,5-dibutyl-3-(4-fluorophenylseleno)-selenophene 2s. Yield

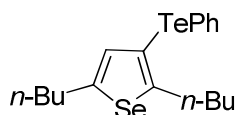
Condition A: 0.056 g (54%). H^1 NMR ($CDCl_3$, 200 MHz): δ 7.26-7.19 (m, 2H), 7.02-6.87 (m, 2H), 6.76 (s, 1H), 2.92 (t, $J = 7.7$ Hz, 4H), 2.77 (t, $J = 7.7$ Hz, 4H), 1.68-1.50 (m, 4H), 1.46-1.25 (m, 4H), 0.95-0.86 (m, 6H). C^{13} NMR ($CDCl_3$, 50 MHz): δ 161.90 (d, $J = 245.90$ Hz), 153.05, 150.06, 132.21 (d, $J = 8.78$ Hz), 132.09, 127.65 (d, $J = 3.66$ Hz), 120.17, 116.17 (d, $J = 21.22$ Hz), 34.99, 34.30, 32.38, 22.25, 22.12, 13.76, 13.71. MS (EI, 70 eV) m/z (relative intensity): 418 (100), 375 (88), 321 (12), 295 (43), 243 (59), 201 (40), 171 (43), 91 (47), 77 (35). Anal. (%) Calcd for $C_{18}H_{23}FSe_2$: C 51.93; H 5.57. Found: C 52.02, H 5.60.



2,5-dibutyl-3-(3-(trifluoromethyl)phenylseleno)-selenophene

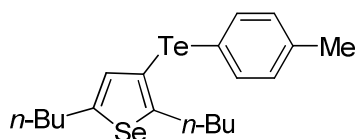
2t. Yield Condition A: 0.061 g (53%). H^1 NMR ($CDCl_3$, 200 MHz): δ 7.44-7.26 (m, 4H),

6.81 (s, 1H), 2.93 (t, $J = 7.7$ Hz, 2H), 2.81 (t, $J = 7.7$ Hz, 2H), 1.67-1.51 (m, 4H), 1.47-1.26 (m, 4H), 0.96-0.84 (m, 4H). C^{13} NMR ($CDCl_3$, 100 MHz): δ 155.05, 150.64, 135.16, 132.28, 132.22, 131.27 (q, $J = 32.2$ Hz), 129.24, 125.64 (q, $J = 3.66$ Hz), 122.45 (q, $J = 3.66$ Hz), 118.28, 34.99, 34.28, 32.24, 32.19, 22.19, 22.02, 13.78. MS (EI, 70 eV) m/z (relative intensity): 465 (90), 425 (100), 344 (32), 300 (34), 242 (83), 200 (47), 144 (27), 91 (66), 77 (47). Anal. (%) Calcd for $C_{19}H_{23}F_3Se_2$: C 48.94; H 4.97. Found: C 49.08, H 5.02.



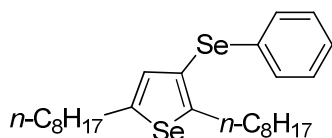
2,5-dibutyl-3-(phenyltelluro)-selenophene 2u. Yield Condition A:

0.060 g (54%). H^1 NMR ($CDCl_3$, 200 MHz): δ 7.49-7.44 (m, 2H), 7.25-7.10 (m, 3H), 6.90 (s, 1H), 2.96 (t, $J = 7.7$ Hz, 2H), 2.79 (t, $J = 7.7$ Hz, 2H), 1.69-1.51 (m, 4H), 1.47-1.25 (m, 4H), 0.95-0.85 (m, 6H). C^{13} NMR ($CDCl_3$, 50 MHz): δ 156.05, 151.66, 137.93, 136.43, 135.52, 129.26, 126.90, 116.21, 105.40, 35.82, 35.33, 34.39, 32.19, 22.24, 22.13, 13.89, 13.81. MS (EI, 70 eV) m/z (relative intensity): 447 (66), 404 (13), 276 (100), 200 (31), 153 (18), 91 (37), 77 (51). HRMS calcd for $C_{18}H_{24}SeTe$: 450.0105. Found: 450.0118.



2,5-dibutyl-3-(p-tolyltelluro)-selenophene 2v. Yield

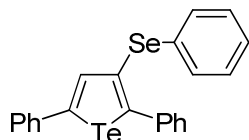
Condition A: 0.050 g (43%). H^1 NMR ($CDCl_3$, 200 MHz): δ 7.41 (d, $J = 7.9$ Hz, 2H), 6.99 (d, $J = 7.9$ Hz, 2H), 6.86 (s, 1H), 2.95 (t, $J = 7.7$ Hz, 2H), 2.77 (t, $J = 7.7$ Hz, 2H), 2.31 (s, 3H), 1.68-1.51 (m, 4H), 1.46-1.28 (m, 4H), 0.94-0.86 (m, 6H). C^{13} NMR ($CDCl_3$, 50 MHz): δ 155.20, 151.47, 137.02, 136.24, 136.19, 130.19, 111.57, 105.60, 35.76, 35.32, 34.42, 32.21, 22.27, 22.15, 21.09, 13.90, 13.82. MS (EI, 70 eV) m/z (relative intensity): 461 (57), 333 (8), 290 (100), 200 (22), 91 (74), 77 (17). HRMS calcd for $C_{19}H_{26}SeTe$: 464.0262. Found: 464.0274.



2,5-dioctyl-3-(phenylseleno)-selenophene 2x. Yield Condition

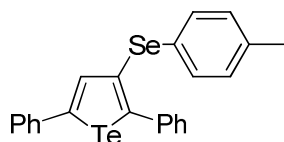
A: 0.057 g (45%). H^1 NMR ($CDCl_3$, 200 MHz): δ 7.25-7.12 (m, 5H), 6.80 (s, 1H), 2.92 (t, $J = 7.7$ Hz, 2H), 2.77 (t, $J = 7.7$ Hz, 2H), 1.70-1.52 (m, 4H), 1.39-1.30 (m, 20H), 0.92-0.83 (m, 6H). C^{13} NMR ($CDCl_3$, 50 MHz): δ 153.74, 149.94, 133.54, 132.57, 129.60, 129.02, 125.84, 119.52, 32.87, 32.67, 32.59, 32.19, 31.82, 29.30, 29.19, 29.15, 29.12, 29.02, 22.63.

MS (EI, 70 eV) m/z (relative intensity): 512 (48), 413 (40), 355 (100), 257 (52), 159 (20), 91 (33), 57 (39). HRMS calcd for $C_{26}H_{40}Se_2$: 512. 1460. Found: 512.1474.



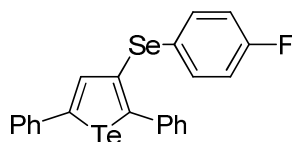
2,5-diphenyl-3-(phenylseleno)-tellurophene 2y.² Yield Condition A:

0.056 g (46%). H^1 NMR ($CDCl_3$, 200 MHz): δ 7.82 (s, 1H), 7.49-7.18 (m, 15H). C^{13} NMR ($CDCl_3$, 50 MHz): δ 147.86, 146.22, 139.82, 139.69, 139.11, 132.91, 131.26, 129.19, 129.16, 128.92, 128.26, 127.88, 127.74, 126.72, 126.64, 126.02. MS (relative intensity) m/z : 488 (24), 360 (13), 279 (10), 202 (100), 77 (10), 51 (6). Anal. calcd for $C_{22}H_{16}SeTe$: C, 54.27; H, 3.88. Found: C, 54.58; H, 3.72.



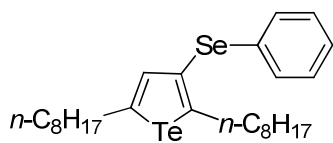
2,5-diphenyl-3-(p-tolylseleno)-tellurophene 2aa.² Yield Condition

A: 0.064 g (51%). H^1 NMR ($CDCl_3$, 200 MHz): δ 7.65-7.57 (m, 3H), 7.42-7.16 (m, 10H), 7.00 (d, $J = 7.82$ Hz, 2H), 2.30 (s, 3H). C^{13} NMR ($CDCl_3$, 100 MHz): δ 148.36, 146.99, 140.36, 140.23, 139.62, 138.81, 134.28, 131.92, 130.56, 129.61, 129.43, 129.09, 128.76, 128.23, 127.32, 127.22, 22.45. MS (relative intensity) m/z : 502 (23), 293 (17), 202 (100), 91 (16), 77 (6). HRMS calcd for $C_{23}H_{18}SeTe$: 503.9636. Found: 503.9651.



2,5-diphenyl-3-(p-fluorophenylseleno)-tellurophene 2z.² Yield

Condition A: 0.054 g (43%). H^1 NMR ($CDCl_3$, 200 MHz): δ 7.75 (s, 1H), 7.47-7.21 (m, 12H), 6.96-6.88 (d, $J = 8.80$ Hz, 2H). C^{13} NMR ($CDCl_3$, 50 MHz): δ 150.85, 147.90, 142.66, 141.60, 139.09, 134.20, 129.57, 128.97, 128.86, 128.76, 128.40, 127.93, 127.88, 126.89, 115.29, 113.48. MS (relative intensity) m/z : 506 (23), 378 (12), 297 (7), 202 (100), 77 (6). Anal. calcd for $C_{22}H_{15}FSeTe$: C, 52.33; H, 2.99. Found: C, 52.55; H, 3.21.

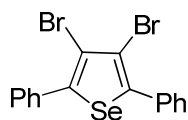


2,5-dioctyl-3-(phenylseleno)-tellutophene 2ab. Yield

Condition A: 0.052 g (37%). ^1H NMR (CDCl_3 , 200 MHz): δ 7.25-7.14 (m, 6H), 2.91 (t, $J = 7.7$ Hz, 2H), 2.76 (t, $J = 7.7$ Hz, 2H), 1.65-1.51 (m, 4H), 1.38-1.17 (m, 20H), 0.90-0.84 (m, 6H). ^{13}C NMR (CDCl_3 , 50 MHz): δ 151.88, 148.17, 140.42, 133.64, 129.81, 129.02, 125.85, 99.97, 36.73, 36.54, 34.83, 34.03, 31.82, 29.35, 29.32, 29.19, 29.16, 28.96, 22.63, 14.08. MS (EI, 70 eV) m/z (relative intensity): 560 (73), 483 (22), 461 (19), 403 (50), 275 (100), 177 (37), 147 (20), 91 (88), 77 (62), 55 (61). HRMS calcd for $\text{C}_{26}\text{H}_{40}\text{SeTe}$: 562.1357. Found: 562.1341.

General procedure for the preparation of the 3,4-dibromo-2,5-diphenylselenophene 3.

A solution of bromine (0.12 g, 0.75 mmol) in CHCl_3 (1 mL) was added dropwise to a solution of 2,5-diphenyl-3-(butylseleno)-selenophene (0.105 g, 0.25 mmol) in CHCl_3 (5 mL) at room temperature. After the addition was complete, the reaction was stirred under reflux for 3 h. After this the mixture was diluted with CH_2Cl_2 (10 mL), and washed with brine (3 x 10 mL). The organic phase was separated, dried over MgSO_4 , and concentrated under vacuum. The residue was purified by flash chromatography on silica gel using hexane as the eluent.



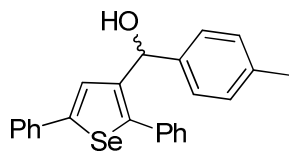
3,4-dibromo-2,5-diphenylselenophene. Yield : 0.096 g (87%).

^1H NMR (CDCl_3 , 200 MHz): δ 7.63-7.59 (m, 4H), 7.50-7.38 (m, 4H), 7.27-7.23 (m, 2H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 143.07, 134.87, 131.46, 129.23, 129.17, 128.60, 112.47. MS (EI, 70 eV) m/z (relative intensity): 442 (47), 361 (5), 182 (30), 202 (100), 141 (13), 101 (43), 88 (4). HRMS calcd for $\text{C}_{16}\text{H}_{10}\text{Br}_2\text{Se}$: 439.8314. Found: 439.8322.

General procedure for the reactions of intermediate 2,5-diphenyl-3-lithioselenophene

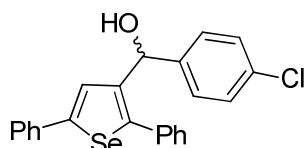
with aldehydes. To a two-neck round-bottomed flask, under argon, containing a solution of 2,5-diphenyl-3-(butylseleno)-selenophene (0.1045g; 0.25 mmol) in THF (2 mL) at -78°C was added $n\text{-BuLi}$ (1 mL of a 2.5 M solution in hexane, 0.25 mmol) in one portion. The reaction mixture was stirred for 10 minutes, and then a solution of appropriated aldehyde (0.3 mmol) in THF (1 mL) at -78°C was added. The reaction mixture was allowed to stir at room temperature for 3 hours. After this time, the mixture was diluted with ethyl acetate

(20 mL) and washed with saturated aq NH_4Cl (20 mL) and water (3x20 mL). The organic phase was separated, dried over MgSO_4 , and concentrated under vacuum. The residue was purified by flash chromatography on silica gel using ethyl acetate/hexane as the eluent.



(2,5-Diphenyl-selenophen-3-yl)-*p*-tolyl-methanol.³ Yield: 0.073g

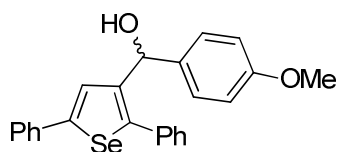
(72%). ^1H NMR (CDCl_3 , 400 MHz), δ (ppm): 7.51-7.46 (m, 5H), 7.41-7.21 (m, 8H), 7.14 (d, $J = 7.9$ Hz, 2H), 5.84 (s, 1H), 2.33 (s, 3H), 2.31 (sl, 1H). C^{13} NMR (CDCl_3 , 50 MHz) δ (ppm): 149.35, 145.68, 142.56, 140.49, 136.99, 136.07, 135.36, 129.47, 129.10, 128.80, 128.60, 127.95, 127.63, 126.12, 126.05, 125.82, 70.55, 21.06. MS (intensidade relativa) m/z : 385 (100), 281 (32), 205 (44), 128 (62), 115 (17), 104 (23), 91 (29), 77 (21). HRMS calculado para $\text{C}_{24}\text{H}_{20}\text{OSe}$: 404.0679. Encontrado: 404.0682.



(2,5-Diphenyl-selenophen-3-yl)-(*p*-chloro-phenyl)-methanol.³

Yield: 0.072g (68%). ^1H NMR(CDCl_3 , 200 MHz), δ (ppm): 7.51-7.22 (m, 15H), 5.83 (s, 1H), 2.42 (sl, 1H). C^{13} NMR (CDCl_3 , 100 MHz) δ (ppm): 149.79, 146.16, 143.27, 141.95, 135.83, 135.11, 133.06, 129.40, 128.87, 128.72, 128.49, 128.14, 127.54, 127.24, 126.02, 125.55, 73.94. MS (intensidade relativa) m/z : 369 (100), 356 (21), 281 (35), 205 (49), 128 (72), 124 (38), 115 (23), 77 (39). HRMS calculado para $\text{C}_{23}\text{H}_{17}\text{ClOSe}$: 424.0133.

Encontrado: 424.0140.



(2,5-Diphenyl-selenophen-3-yl)-(*p*-methoxy-phenyl)-

methanol.³ Yield: 0.076g (73%). RMN ^1H (CDCl_3 , 200 MHz), δ (ppm): 7.54-7.25 (m, 13H), 6.91-6.85 (m, 2H), 5.83 (s, 1H), 3.79 (s, 3H), 2.27 (sl, 1H). RMN C^{13} (CDCl_3 , 100 MHz) δ (ppm): 158.85, 149.37, 145.56, 142.61, 136.05, 135.54, 135.33, 129.45, 128.84, 128.62, 127.96, 127.67, 127.50, 126.05, 125.96, 113.80, 70.38, 55.24. MS (intensidade

³ Alves, D.; Luchese, C.; Nogueira, C. W.; Zeni, G. *J. Org. Chem.* **2007**, *72*, 6726.

relativa) m/z : 401 (100), 370 (19), 281 (45), 205 (32), 128 (82), 120 (54), 77 (28). HRMS
calculado para $C_{24}H_{20}O_2Se$: 420.0628. Encontrado: 420.0633.

SELECTED SPECTRA

