Supporting Information

for

Thiophene/selenophene-based S-shaped double helicenes: regioselective synthesis and structures

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*Beilstein J. Org. Chem.* 2022, 18, 809–817. doi:10.3762/bjoc.18.81

Supporting crystallographic information for compounds DH-1 and DH-2
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X-ray crystallographic data

Complete crystal data for DH-1

Table S1. Crystal data and structure refinement for DH-1.

| Identification code | DH-1 |
|---------------------|------|
| Empirical formula   | C\(_{32}\)H\(_{26}\)S\(_6\)Si\(_2\) |
| Formula weight      | 659.07 |
| Temperature         | 150.0 K |
| Wavelength          | 0.71073 Å |
| Crystal system      | Triclinic |
| Space group         | P-1 |
| Unit cell dimensions| a = 11.1290(7) Å, b = 12.1240(7) Å, c = 13.7363(9) Å |
|                     | \(\alpha = 108.362(2)^\circ\), \(\beta = 111.318(2)^\circ\), \(\gamma = 100.961(2)^\circ\) |
| Volume              | 1538.32(17) Å\(^3\) |
| Z                   | 2 |
| Density (calculated)| 1.423 Mg/m\(^3\) |
| Absorption coefficient | 0.546 mm\(^{-1}\) |
| F(000)              | 684 |
| Crystal size        | 0.14 x 0.12 x 0.08 mm\(^3\) |
| Theta range for data collection | 2.284 to 25.499° |
| Index ranges        | -13 ≤ h ≤ 13, -14 ≤ k ≤ 13, -16 ≤ l ≤ 16 |
| Reflections collected | 19274 |
| Independent reflections | 5716 [R(int) = 0.1014] |
| Completeness to theta = 25.242° | 99.7% |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 0.7457 and 0.6783 |
| Refinement method   | Full-matrix least-squares on F\(^2\) |
| Data / restraints / parameters | 5716 / 0 / 367 |
| Goodness-of-fit on F\(^2\) | 1.047 |
| Final R indices [I>2sigma(I)] | R1 = 0.0562, wR2 = 0.0872 |
| R indices (all data) | R1 = 0.1093, wR2 = 0.1101 |
| Extinction coefficient | n / a |
| Largest diff. peak and hole | 0.476 and -0.451 e.Å\(^{-3}\) |
Figure S1. The crystal structures for compound DH-1. Carbon, sulfur, and silicon atoms are depicted with thermal ellipsoids set at 30% probability level, and all hydrogen atoms are omitted for clarity.

Figure S2. Molecular configuration of DH-1 in one unit cell.
**Figure S3.** Multiple interactions in the crystal packings of DH-1.

**Figure S4.** Molecular packing of DH-1.
Complete crystal data for DH-2

Table S2. Crystal data and structure refinement for DH-2.

| Property                              | Value                                      |
|---------------------------------------|--------------------------------------------|
| Identification code                   | DH-2                                       |
| Empirical formula                     | C$_{32}$H$_{26}$S$_2$Se$_4$Si$_2$          |
| Formula weight                        | 846.67                                     |
| Temperature                           | 149.98 K                                   |
| Wavelength                            | 0.71073 Å                                  |
| Crystal system                        | Triclinic                                  |
| Space group                           | P-1                                        |
| Unit cell dimensions                  | a = 11.0959(11) Å, $\alpha = 112.217(4)^\circ$. |
|                                      | b = 12.2773(12) Å, $\beta = 106.495(3)^\circ$. |
|                                      | c = 13.5535(13) Å, $\gamma = 99.898(3)^\circ$. |
| Volume                                | 1555.7(3) Å$^3$                            |
| Z                                      | 2                                          |
| Density (calculated)                  | 1.807 Mg/m$^3$                             |
| Absorption coefficient                | 4.951 mm$^{-1}$                            |
| F(000)                                | 828                                        |
| Crystal size                          | 0.21 x 0.17 x 0.12 mm$^3$                  |
| Theta range for data collection       | 2.288 to 28.326°                          |
| Index ranges                          | -14 ≤ h ≤ 14, -15 ≤ k ≤ 16, -18 ≤ l ≤ 18 |
| Reflections collected                 | 23440                                      |
| Independent reflections               | 7708 [R(int) = 0.0726]                     |
| Completeness to theta = 25.242°       | 99.7 %                                     |
| Absorption correction                 | Semi-empirical from equivalents            |
| Max. and min. transmission            | 0.7457 and 0.5103                          |
| Refinement method                     | Full-matrix least-squares on F$^2$         |
| Data / restraints / parameters        | 7708 / 0 / 367                             |
| Goodness-of-fit on F$^2$              | 1.027                                      |
| Final R indices [I>2sigma(I)]         | R1 = 0.0443, wR2 = 0.0999                  |
| R indices (all data)                  | R1 = 0.0690, wR2 = 0.1133                  |
| Extinction coefficient                | n/a                                        |
| Largest diff. peak and hole           | 1.175 and -0.880 e.Å$^{-3}$                |
Figure S5. The crystal structures for compound DH-2. Carbon, selenium, sulfur, and silicon atoms are depicted with thermal ellipsoids set at 30% probability level, and all hydrogen atoms are omitted for clarity.

Figure S6. Molecular configuration of DH-2 in one unit cell.
Figure S7. Multiple interactions in the crystal packings of DH-2

Figure S8. Molecular packing of DH-2.