

Crystal structures of 3-halo-2-organochalcogenylbenzo[*b*]chalcogenophenes

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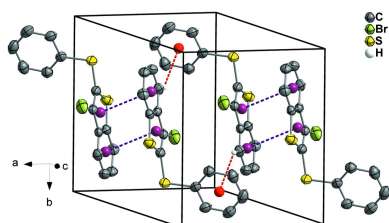
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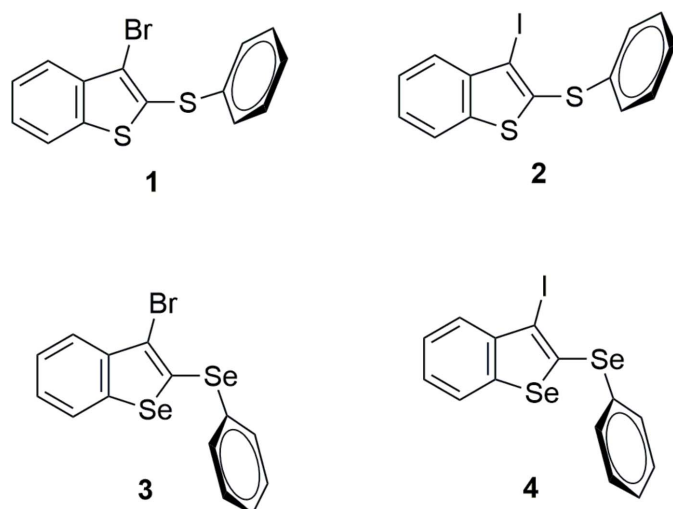
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The structure of the title compounds 3-bromo-2-(phenylsulfanyl)benzo[*b*]thiophene (C₁₄H₉BrS₂; **1**), 3-iodo-2-(phenylsulfanyl)benzo[*b*]thiophene (C₁₄H₉IS₂; **2**), 3-bromo-2-(phenylselanyl)benzo[*b*]selenophene (C₁₄H₉BrSe₂; **3**), and 3-iodo-2-(phenylselanyl)benzo[*b*]selenophene (C₁₄H₉ISE₂; **4**) were determined by single-crystal X-ray diffraction; all structures presented monoclinic (*P*2₁/*c*) symmetry. The phenyl group is distant from the halogen atom to minimize the steric hindrance repulsion for all structures. Moreover, the structures of **3** and **4** show an almost linear alignment of halogen–selenium–carbon atoms arising from the intramolecular orbital interaction between a lone pair of electrons on the halogen atom and the antibonding $\sigma^*_{\text{Se-C}}$ orbital ($n_{\text{halogen}} \rightarrow \sigma^*_{\text{Se-C}}$). This interaction leads to significant differences in the three-dimensional packing of the molecules, which are assembled through π – π and C–H... π interactions. These data provide a better comprehension of the intermolecular packing in benzo[*b*]chalcogenophenes, which is relevant for optoelectronic applications.

1. Chemical context

Chalcogenophenes derivatives are an attractive synthetic class of compounds with a wide range of relevant applications in medicinal chemistry (Keri *et al.*, 2017; Mahmoud *et al.*, 2017; Paegle *et al.*, 2016), electrochemistry (Wei *et al.*, 2017; Shahjad *et al.*, 2017), agrochemistry (Zani *et al.*, 2004) and as organic semiconductors (Yang *et al.*, 2018; Ostroverkhova, 2016). π -extended benzo[*b*]chalcogenophenes derivatives have been widely studied as improved materials for optoelectronic devices such as organic photovoltaic cells (OPVs) (Ashraf *et al.*, 2015; An *et al.*, 2018; Chen *et al.*, 2017), liquid-crystal displays (LCD) (Ghosh & Lehmann, 2017; Mei *et al.*, 2013), organic light-emitting diodes (OLEDs) (Grimsdale *et al.*, 2009; Zampetti *et al.*, 2017; Arsenyan *et al.*, 2019), and in organic field-effect transistors (OFETs) (Lee *et al.*, 2019; Tisovský *et al.*, 2019). Benzo[*b*]chalcogenophenes derivatives also show relevant biological activities as anti-tumor (Arsenyan *et al.*, 2011) and anti-inflammatory agents (Shah *et al.*, 2018). As part of our continuing work on benzo[*b*]chalcogenophenes (Luz *et al.*, 2021), we report herein the crystallographic structural comparison of four 3-halo-2-(organochalcogenyl)benzo[*b*]chalcogenophene derivatives.





2. Structural commentary

The four organic compounds crystallize in the monoclinic $P2_1/c$ space group, and all atoms occupy unique positions. Compounds **1** and **2** are isostructural containing an identical 3-halo-2-(phenylsulfanyl)benzo[*b*]thiophene unit with bro-

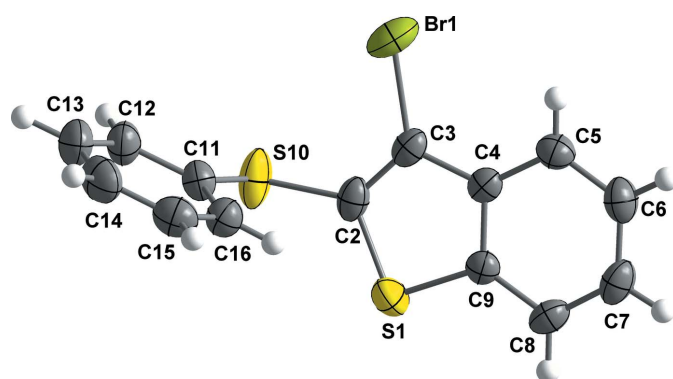


Figure 1
The molecular structure of 3-bromo-2-(phenylsulfanyl)benzo[*b*]thiophene (**1**), with displacement ellipsoids drawn at the 50% probability level.

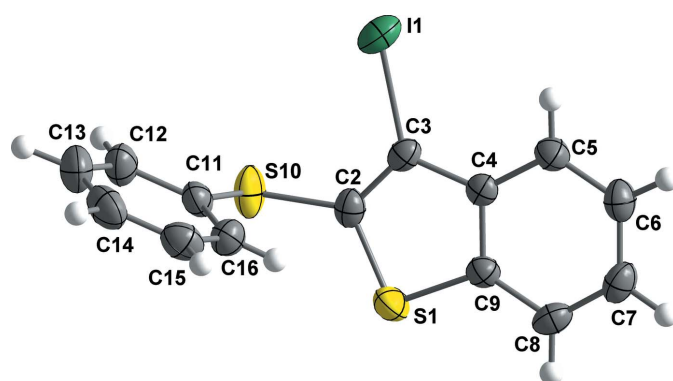


Figure 2
The molecular structure of 3-iodo-2-(phenylsulfanyl)benzo[*b*]thiophene (**2**), with displacement ellipsoids drawn at the 50% probability level.

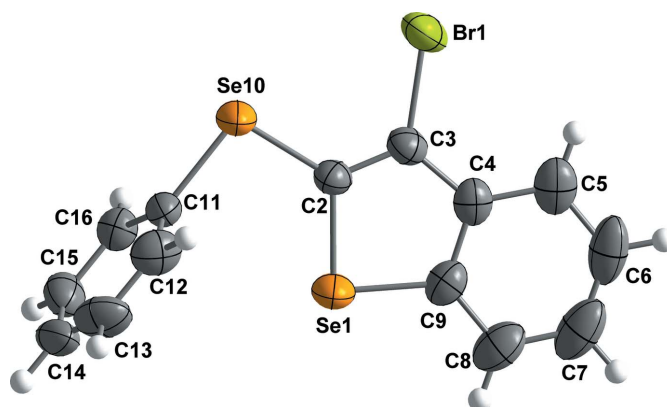


Figure 3
The molecular structure of 3-bromo-2-(phenylselanyl)benzo[*b*]selenophene (**3**), with displacement ellipsoids drawn at the 50% probability level.

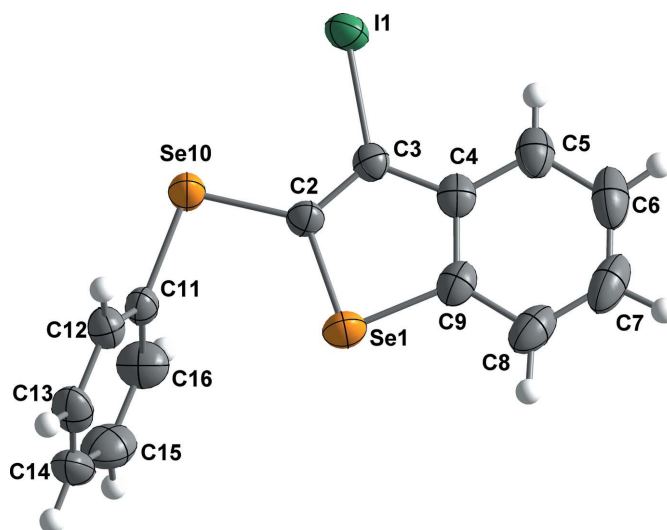


Figure 4
The molecular structure of 3-iodo-2-(phenylselanyl)benzo[*b*]selenophene (**4**), with displacement ellipsoids drawn at the 50% probability level.

mine (**1**) or iodine (**2**) at the C3 position of the benzo[*b*]thiophene ring (Figs. 1 and 2). The isostructural compounds **3** and **4** also contain identical 3-halo-2-(phenylselanyl)benzo[*b*]selenophene units with bromine (**3**) or iodine (**4**) at the C3 position of the benzo[*b*]selenophene ring (Figs. 3 and 4). The respective benzo[*b*]chalcogenophene rings and the phenylsulfanyl and phenylselanyl groups are planar. As expected, the carbon–selenium bonds in molecules **3** and **4** are longer than the respective carbon–sulfur bonds in molecules **1** and **2**.

Conformational changes are observed when we compare molecules **1** and **2** containing sulfur atoms with molecules **3** and **4** containing selenium atoms, as described below. In molecules **1** and **2**, the benzo[*b*]thiophene ring is twisted away from the plane of phenylsulfanyl group showing interplanar angles of 88.9 (8) and 87.9 (6)°, respectively (Figs. 5 and 6). Additionally, for **1** and **2** the S1–C2–S10–C11 torsion angles are –97.56 (14) and 98.17 (15)°, respectively. Molecules **3** and **4** also show the benzo[*b*]selenophene ring twisted away

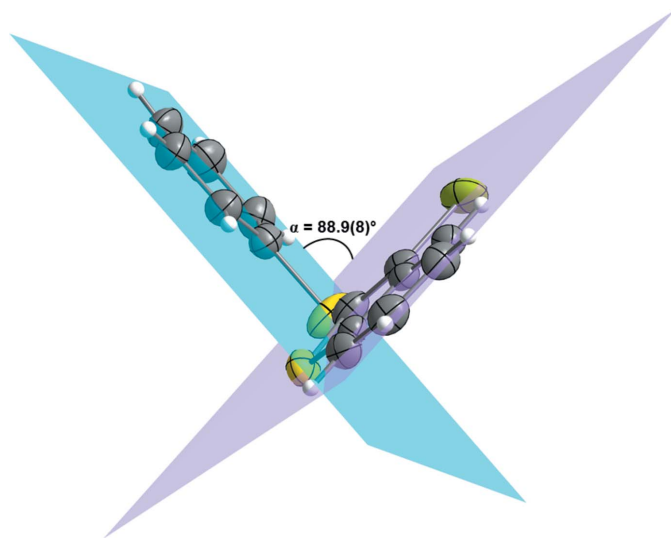


Figure 5
Representation of the interplanar angle (α) between the planes containing the phenylsulfanyl, blue plane, and the benzo[*b*]thiophene, purple plane, groups for 3-bromo-2-(phenylsulfanyl)benzo[*b*]thiophene (**1**). Displacement ellipsoids are drawn at the 50% probability level. Gray: carbon; yellow: sulfur; light green: bromine; white: hydrogen.

from the plane of the phenylselanyl group with interplanar angles of 80.4 (8) and 79.7 (7) $^\circ$, respectively (Figs. 7 and 8). Conversely, the torsion angles (Se1–C2–Se10–C11) in molecules **3** and **4** are 1.9 (3) and -4.0 (3) $^\circ$, respectively, quite different than the S1–C2–S10–C11 torsion angles in molecules **1** and **2**. It is clear that the coplanarity between the phenyl and benzo[*b*]chalcogenophene rings is avoided in both pairs of molecules to minimize steric hindrance. This structural arrangement is reinforced by the presence of the halogen atom at the C3 position of the benzo[*b*]chalcogenophene ring (Figs. 1, 2, 3 and 4). Nevertheless, there is an almost linear

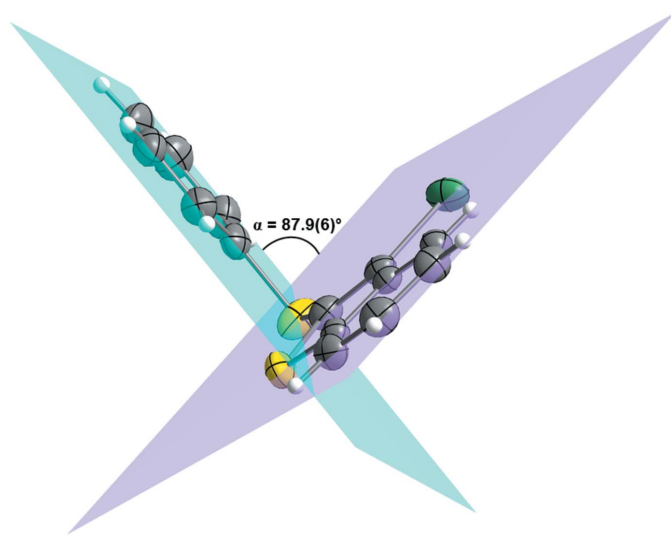


Figure 6
Representation of the interplanar angle (α) between the planes containing the phenylsulfanyl, blue plane, and the benzo[*b*]thiophene, purple plane, groups for 3-iodo-2-(phenylsulfanyl)benzo[*b*]thiophene (**2**). Displacement ellipsoids are drawn at the 50% probability level. Gray: carbon; yellow: sulfur; bluish green: iodine; white: hydrogen.

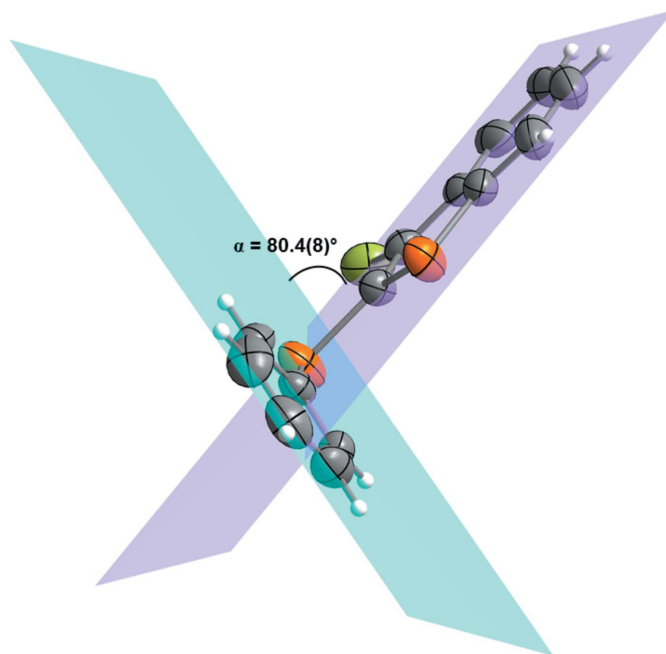


Figure 7
Representation of the interplanar angle (α) between the planes containing the phenylselanyl, blue plane, and the benzo[*b*]selenophene, purple plane, groups for 3-bromo-2-(phenylselanyl)benzo[*b*]selenophene (**3**). Displacement ellipsoids are drawn at the 50% probability level. Gray: carbon; orange: selenium; light green: bromine; white: hydrogen.

alignment between the atoms Br1–Se10–C11 (**3**) and I1–Se10–C11 (**4**), which cannot be explained by steric factors alone. For instance, if we consider merely the higher steric hindrance between the phenyl and benzo[*b*]selenophene rings arising from the lower intrinsic C11–Se10–C2 angle directing

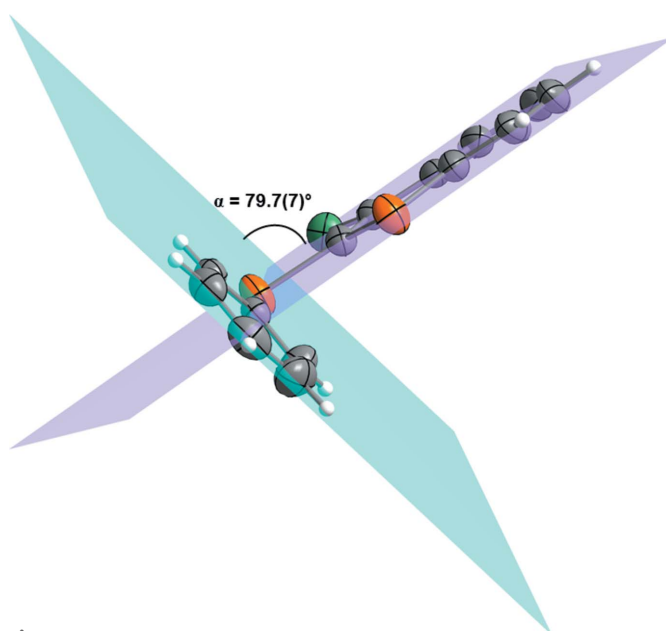


Figure 8
Representation of the interplanar angle (α) between the planes containing the phenylselanyl, blue plane, and the benzo[*b*]selenophene, purple plane, groups for 3-iodo-2-(phenylselanyl)benzo[*b*]selenophene (**4**). Displacement ellipsoids are drawn at the 50% probability level. Gray: carbon; orange: selenium; bluish green: iodine; white: hydrogen.

the conformation of molecules **3** and **4**, the almost linear alignment between the atoms Br1–Se10–C11 (**3**) and I1–Se10–C11 (**4**) is still not fully understood. We have observed that the interatomic distances between the chalcogen and the halogen atoms [S10···Br1 (**1**) = 3.5061 (8) Å, S10···I1 (**2**) = 3.6310 (7) Å, Se10···Br (**3**) = 3.4196 (7) Å, Se10···I (**4**) = 3.5260 (7) Å] are 0.14, 0.15, 0.33 and 0.35 Å shorter than the sum of the van der Waals radii of the respective two atoms in molecules **1**, **2**, **3**, and **4**, respectively (Bondi, 1964). The shorter interatomic distances Se10···Br and Se10···I and the remarkably almost linear alignment of the atoms in **3** [C11–Se10···Br1 = 152.95 (9)°] and in **4** [C11–Se10···I1 = 156.52 (1)°] when compared to molecules **1** [C11–S10···Br1 = 93.01 (7)°] and **2** [C11–S10···I1 = 91.35 (7)°] indicate a stabilizing intramolecular orbital interaction (3-center-4-electrons, 3c–4e) between a lone pair of electrons of the halogen atom and the antibonding $\sigma_{\text{Se-C11}}^*$ orbital ($n_{\text{halogen}} \rightarrow \sigma_{\text{Se-C11}}^*$) (Mukherjee, 2010). The lower energy of the antibonding $\sigma_{\text{Se-C11}}^*$ orbital makes it a better acceptor when compared to the higher energy antibonding $\sigma_{\text{S-C11}}^*$ orbital, therefore making the intramolecular $n_{\text{halogen}} \rightarrow \sigma_{\text{Se-C11}}^*$ orbital interaction in molecules **3** and **4** strong enough to change their molecular conformation.

3. Supramolecular features

The crystals of organic compounds **1** and **2** are related by an inversion center and assembled through C–H··· π intermolecular interactions along the *b*-axis direction (Fig. 9). The weak C–H··· π interactions are between the H5 atom and the centroid formed by atoms C11–C16 of the phenylsulfanyl group. The distances and angles comprising these contacts are 2.97 (2) Å, 137.1 (2)° for **1** and 2.93 (3) Å, 138.4 (2)° for **2**. The structures **1** and **2** also show π – π stacking interactions between

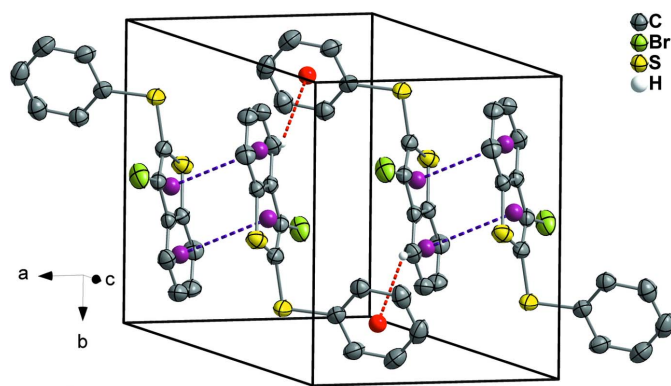


Figure 9
Representation of some molecules of 3-bromo-2-(phenylsulfanyl)benzo[*b*]selenophene (**1**) viewed approximately down the *c* axis of the unit cell. The red dashed lines represent C–H··· π interactions involving the H5 atom of the benzo[*b*]thiophene ring with an adjacent phenylsulfanyl group; the purple dashed lines represent π – π stacking interactions between adjacent benzo[*b*]thiophene rings. Displacement ellipsoids are drawn at the 50% probability level. The hydrogen atoms, except for H4, are omitted for clarity. Red and purple spheres represent the centroids of the respective organic groups.

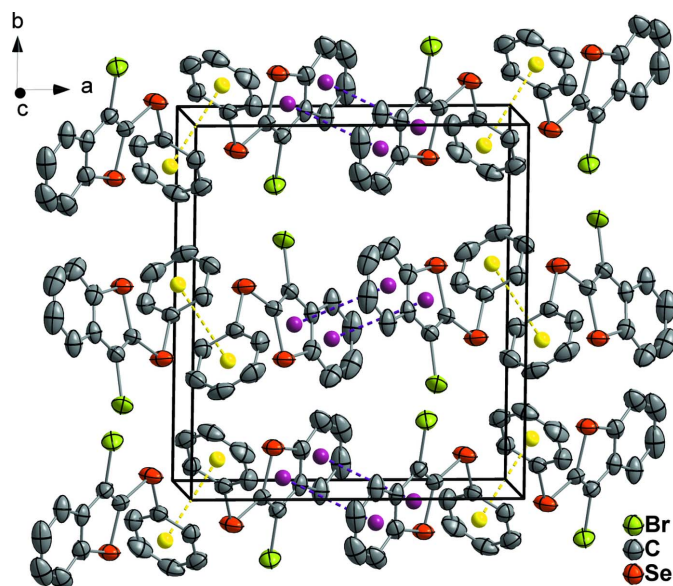


Figure 10
Representation of the molecules of 3-bromo-2-(phenylselenanyl)benzo[*b*]selenophene (**3**) viewed down the *c* axis of the unit cell. The purple and yellow dashed lines represent π – π stacking interactions between adjacent benzo[*b*]thiophene rings and between adjacent phenylsulfanyl groups, respectively. Displacement ellipsoids are drawn at the 50% probability level. The hydrogen atoms were omitted to clarity. Purple and yellow spheres represent the centroids of the respective organic groups.

adjacent benzo[*b*]thiophene rings along the *c*-axis direction with centroid–centroid distances of 3.7166 (2) and 3.7602 (4) Å for **1** and **2**, respectively (Fig. 9, for **1**). On the other hand, in compounds **3** and **4** C–H··· π interactions are not present. However, π – π stacking interactions involving adjacent benzo[*b*]thiophene rings are present along the *a*-axis direction, with centroid–centroid distances of 3.8139 (3) Å and 3.8772 (1) Å, respectively. Furthermore, π – π stacking interactions are observed along the *b*-axis direction between phenylsulfanyl groups related by an inversion center, with centroid–centroid distances of 3.6644 (2) and 3.7351 (1) Å for **3** and **4**, respectively (Fig. 10, for **3**).

4. Database survey

Several crystal structures of benzo[*b*]chalcogenophenes derivatives have been published. To the best of our knowledge, there are no studies about chalcogen atoms attached directly at position 2 of the benzo[*b*]chalcogenophene ring. With regard to benzo[*b*]thiophenes, Xu *et al.* (2017) described the structure of 3-(arylsulfonyl)benzo[*b*]thiophene obtained by single-crystal X-ray diffraction. Additionally, Ramesh *et al.* (2016) reported the structures of 6-fluoro-2,2-(diphenyl)benzo[*b*]thiophene and 6-isopropyl-2,2-(diphenyl)benzo[*b*]thiophene obtained by single-crystal X-ray diffraction studies.

5. Synthesis and crystallization

The structures reported here were obtained by the one-pot synthesis of 3-halo-2-organochalcogenylbenzo[*b*]chalcogeno-

phenes from 1-(2,2-dibromovinyl)-2-organochalcogenylbenzenes. By this method, a series of 2,3-disubstituted benzo[*b*]chalcogenophenes were prepared in yields of *ca* 80% (Luz *et al.*, 2021). The title compounds were prepared as follows:

3-Bromo-2-(phenylsulfanyl)benzo[*b*]thiophene (1)

To a Schlenk tube containing 1-(2,2-dibromovinyl)-2-butylsulfanylbenzene (0.25 mmol, 1.0 equiv.), diphenyl disulfide (0.125 mmol, 1.0 equiv.) was added in dry dimethyl sulfoxide (2.0 mL) followed by the addition of cesium carbonate (0.244 g, 0.75 mmol, 3.0 equiv.). The reaction system was heated to 383 K and stirred for 1.5 h. Then, the reaction mixture was cooled to room temperature and 2.5 equivalents of NBS (*N*-bromosuccinimide) in 2 mL of dichloromethane were slowly added (2.0 min) into the system. The reaction mixture was stirred at room temperature for 2 h. After this, the reaction solution was diluted in saturated thiosulfate solution (20 mL) and washed with ethyl acetate (3 × 10 mL). The organic phase was dried over magnesium sulfate and concentrated under reduced pressure. The product was further purified by flash chromatography using hexane as eluent. Colorless needle-shaped single crystals of **1** suitable for X-ray analysis were grown by slow evaporation of a concentrated ethyl acetate solution over several days at room temperature. Yield: 0.066 g (82%); white solid, m.p. 337–340 K. ¹H NMR (CDCl₃, 400 MHz) δ (ppm) = 7.77–7.75 (*m*, 1 H); 7.70–7.68 (*m*, 1H); 7.58–7.55 (*m*, 2H); 7.44–7.40 (*m*, 1H); 7.36–7.30 (*m*, 4H). ¹³C{¹H} NMR (CDCl₃, 100 MHz) δ (ppm) = 141.1, 138.5, 135.9, 133.1, 129.5, 128.3, 126.4, 125.4, 125.2, 123.3, 121.9, 114.4. MS (Rel. Int.) *m/z*: 321 (84.0), 241 (100), 210 (63.4), 77 (54.8). HRMS: Calculated mass for C₁₄H₁₀BrS₂ [*M*]⁺: 321.9302, found: 321.9310.

3-Iodo-2-(phenylsulfanyl)benzo[*b*]thiophene (2)

The first step for obtaining **2** was analogous to that described for **1**. The reaction mixture was cooled to room temperature and 1.5 equivalents of I₂ in 2 mL of dichloromethane were slowly added (2.0 min) into the system. The reaction mixture was stirred at room temperature for 3.5 h. After this, the reaction solution was diluted in saturated sodium thiosulfate solution (20 mL) and washed with ethyl acetate (3 × 10 mL). The organic phase was dried over magnesium sulfate and concentrated under reduced pressure. The product was further purified by flash chromatography using hexane as eluent. Colorless needle-shaped single crystals of **2** were obtained in the same way of **1**. Yield: 0.073 g (79%); yellow solid, m.p. 325–327 K. ¹H NMR (CDCl₃, 400 MHz) δ (ppm) = 7.72 (*d*, *J* = 8.0 Hz, 1 H); 7.66 (*d*, *J* = 7.6 Hz, 1 H); 7.44–7.40 (*m*, 2H); 7.34–7.21 (*m*, 5H). ¹³C{¹H} NMR (CDCl₃, 100 MHz) δ (ppm) = 141.5, 141.2, 136.5, 134.7, 130.3, 129.3, 127.7, 126.2, 126.0, 125.5, 122.1, 90.3. MS (Rel. Int.) *m/z*: 368 (94.3), 240 (100), 120 (50.3), 77 (10.5). HRMS: Calculated mass for C₁₄H₉IS₂ [*M*]⁺: 367.9185, found: 367.9188.

3-Bromo-2-(phenylselanyl)benzo[*b*]selenophene (3)

To a Schlenk tube containing 1-(2,2-dibromovinyl)-2-butylselanylbenzene (0.25 mmol, 1.0 equiv.), diphenyl diselenide (0.125 mmol, 1.0 equiv.) was added in dry dimethyl sulfoxide (2.0 mL) followed by the addition of cesium carbonate (0.244 g, 0.75 mmol, 3.0 equiv.). The reaction system was

heated to 384 K and stirred for 0.5 h. Then, the reaction mixture was cooled to room temperature and 2.5 equivalents of NBS (*N*-bromosuccinimide) in 2 mL of dichloromethane were slowly added (2.0 min) into the system. The reaction mixture was stirred at room temperature for 1 h. After this, the reaction solution was diluted in saturated sodium thiosulfate solution (20 mL) and washed with ethyl acetate (3 × 10 mL). The organic phase was dried over magnesium sulfate and concentrated under reduced pressure. The product was further purified by flash chromatography using hexane as eluent. Colorless needle-shaped single crystals of **3** were obtained in the same way as **1**. Yield: 0.081 g (79%); yellow oil. ¹H NMR (CDCl₃, 400 MHz) δ (ppm) = 7.76 (*dd*, *J* = 8.1 and 1.0 Hz, 1H); 7.69–7.66 (*m*, 3H); 7.41–7.32 (*m*, 4H); 7.24 (*ddd*, *J* = 8.2, 7.3 and 1.3 Hz, 1H). ¹³C{¹H} NMR (CDCl₃, 100 MHz) δ (ppm) = 141.1, 140.6, 134.4, 129.6, 129.5, 129.4, 129.0, 125.4, 125.1, 125.0, 124.9, 112.9. MS (Rel. Int.) *m/z*: 416 (96.8), 336 (100), 256 (42.4), 77 (62.0).

3-Iodo-2-(phenylselanyl)benzo[*b*]selenophene (4)

The first step for obtaining **4** was similar to that described for **3**. The reaction mixture was cooled to room temperature and 1.5 equivalents of I₂ in 2 mL of dichloromethane were slowly added (2.0 min) into the system. The reaction was stirred at room temperature for 1 h. After this, the reaction solution was diluted in saturated sodium thiosulfate solution (20 mL) and washed with ethyl acetate (3 × 10 mL). The organic phase was dried over magnesium sulfate and concentrated under reduced pressure. The product was further purified by flash chromatography using hexane as eluent. Colorless needle single crystals of **4** were obtained in the same way of (**1**). Yield: 0.090 g (78%); Orange solid, m.p. 329–331 K. ¹H NMR (CDCl₃, 400 MHz) δ (ppm) = 7.73–7.64 (*m*, 4H); 7.41–7.33 (*m*, 4H); 7.23–7.20 (*m*, 1H). ¹³C{¹H} NMR (CDCl₃, 100 MHz) δ (ppm) = 143.9, 142.2, 134.9, 134.7, 129.8, 129.7, 129.1, 127.6, 125.7, 125.1, 88.9. MS (Rel. Int.) *m/z*: 464 (48.2), 334 (47.0), 256 (51.4), 77 (53.2), 51 (100).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. Hydrogen atoms of **1**, **2** and **4** were located in difference-Fourier maps and were refined freely; the hydrogen atoms of **3** were included in idealized positions with aromatic C–H distances set to 0.93 Å and refined using a riding model $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

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Table 1
Experimental details.

	1	2	3	4
Crystal data				
Chemical formula	C ₁₄ H ₉ BrS ₂	C ₁₄ H ₉ IS ₂	C ₁₄ H ₉ BrSe ₂	C ₁₄ H ₉ ISe ₂
<i>M_r</i>	321.24	368.23	415.04	462.03
Crystal system, space group	Monoclinic, <i>P</i> ₂ ₁ / <i>c</i>	Monoclinic, <i>P</i> ₂ ₁ / <i>c</i>	Monoclinic, <i>P</i> ₂ ₁ / <i>c</i>	Monoclinic, <i>P</i> ₂ ₁ / <i>c</i>
Temperature (K)	296	294	297	292
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.2471 (8), 9.9562 (8), 15.7601 (14)	8.4872 (3), 9.9629 (4), 15.6485 (7)	12.3864 (11), 13.6816 (11), 8.0982 (6)	12.9606 (6), 13.5999 (7), 8.0448 (4)
β (°)	98.967 (3)	97.052 (1)	96.398 (3)	95.585 (2)
<i>V</i> (Å ³)	1278.2 (2)	1313.18 (9)	1363.82 (19)	1411.27 (12)
<i>Z</i>	4	4	4	4
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	3.51	2.73	8.33	7.40
Crystal size (mm)	0.28 × 0.21 × 0.14	0.16 × 0.13 × 0.05	0.17 × 0.17 × 0.12	0.51 × 0.47 × 0.24
Data collection				
Diffractionmeter	Bruker D8 Venture/Photon 100 CMOS	Bruker D8 Venture/Photon 100 CMOS	Bruker D8 Venture/Photon 100 CMOS	Bruker D8 Venture/Photon 100 CMOS
Absorption correction	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)
<i>T</i> _{min} , <i>T</i> _{max}	0.628, 0.746	0.690, 0.746	0.543, 0.746	0.390, 0.746
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>) reflections	55020, 3078, 2559	56046, 3001, 2585	43875, 2976, 2208	54241, 3391, 2702
<i>R</i> _{int}	0.041	0.045	0.060	0.054
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.660	0.650	0.639	0.660
Refinement				
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.029, 0.073, 1.04	0.022, 0.051, 1.08	0.035, 0.069, 1.03	0.037, 0.083, 1.04
No. of reflections	3078	3001	2976	3391
No. of parameters	190	190	154	190
H-atom treatment	All H-atom parameters refined	All H-atom parameters refined	H-atom parameters constrained	All H-atom parameters refined
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.41, -0.76	0.45, -0.86	0.93, -0.90	1.14, -1.82

Computer programs: *APEX3* (Bruker, 2015), *SAINT* (Bruker, 2002), *SHELXL97* (Sheldrick, 2008), *SHELXT* (Sheldrick, 2015a), *SHELXL2018/3* (Sheldrick, 2015b), *DIAMOND* (Brandenburg & Putz, 2006) and *WinGX* (Farrugia, 2012).

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supporting information

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Crystal structures of 3-halo-2-organochalcogenylbenzo[*b*]chalcogenophenes

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Computing details

For all structures, data collection: *APEX3* (Bruker, 2015); cell refinement: *SAINTE* (Bruker, 2002); data reduction: *SAINTE* (Bruker, 2002); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015b); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2006); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *WinGX* (Farrugia, 2012).

3-Bromo-2-(phenylsulfanyl)benzo[*b*]thiophene (1)

Crystal data

C₁₄H₉BrS₂

M_r = 321.24

Monoclinic, *P*2₁/*c*

a = 8.2471 (8) Å

b = 9.9562 (8) Å

c = 15.7601 (14) Å

β = 98.967 (3)°

V = 1278.2 (2) Å³

Z = 4

F(000) = 640

D_x = 1.669 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 9946 reflections

θ = 2.6–28.0°

μ = 3.51 mm⁻¹

T = 296 K

Parallelepiped, colourless

0.28 × 0.21 × 0.14 mm

Data collection

Bruker D8 Venture/Photon 100 CMOS diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 10.4167 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)

T_{min} = 0.628, *T_{max}* = 0.746

55020 measured reflections

3078 independent reflections

2559 reflections with *I* > 2σ(*I*)

R_{int} = 0.041

θ_{max} = 28.0°, θ_{min} = 3.2°

h = -10→10

k = -13→13

l = -20→20

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.029

wR(*F*²) = 0.073

S = 1.04

3078 reflections

190 parameters

0 restraints

Primary atom site location: dual

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

All H-atom parameters refined

w = 1/[σ²(*F_o*²) + (0.0347*P*)² + 0.7059*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.41 e Å⁻³

Δρ_{min} = -0.76 e Å⁻³

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C2	0.1651 (3)	0.3373 (2)	0.41693 (14)	0.0405 (5)
C3	0.2308 (2)	0.4295 (2)	0.47529 (13)	0.0361 (4)
C4	0.1731 (2)	0.56405 (19)	0.45645 (12)	0.0315 (4)
C5	0.2140 (3)	0.6832 (2)	0.50152 (14)	0.0399 (4)
C6	0.1421 (3)	0.8012 (2)	0.47034 (16)	0.0460 (5)
C7	0.0310 (3)	0.8041 (2)	0.39437 (16)	0.0468 (5)
C8	−0.0120 (3)	0.6888 (2)	0.34898 (14)	0.0426 (5)
C9	0.0599 (2)	0.56820 (19)	0.38079 (12)	0.0325 (4)
C11	0.3783 (3)	0.1481 (2)	0.36842 (14)	0.0392 (4)
C12	0.4614 (3)	0.0261 (2)	0.38205 (16)	0.0487 (5)
C13	0.6017 (3)	0.0046 (3)	0.34629 (17)	0.0562 (6)
C14	0.6591 (3)	0.1024 (3)	0.29691 (18)	0.0578 (6)
C15	0.5762 (3)	0.2225 (3)	0.28289 (16)	0.0512 (6)
C16	0.4356 (3)	0.2460 (2)	0.31852 (14)	0.0435 (5)
Br1	0.38366 (3)	0.38960 (3)	0.57285 (2)	0.05735 (10)
S1	0.02548 (7)	0.41026 (6)	0.33534 (4)	0.04409 (13)
S10	0.19752 (8)	0.16429 (6)	0.41634 (5)	0.05842 (19)
H5	0.289 (3)	0.680 (2)	0.5496 (15)	0.042 (6)*
H6	0.172 (3)	0.880 (3)	0.4980 (17)	0.053 (7)*
H7	−0.009 (4)	0.887 (3)	0.3766 (18)	0.060 (8)*
H8	−0.089 (3)	0.691 (3)	0.2997 (17)	0.055 (7)*
H12	0.429 (3)	−0.039 (3)	0.4186 (17)	0.056 (7)*
H13	0.660 (4)	−0.081 (3)	0.3556 (18)	0.065 (8)*
H14	0.751 (4)	0.092 (3)	0.278 (2)	0.073 (9)*
H15	0.613 (3)	0.286 (3)	0.2530 (18)	0.058 (8)*
H16	0.379 (3)	0.325 (3)	0.3084 (17)	0.058 (8)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.0426 (11)	0.0318 (9)	0.0515 (12)	0.0038 (8)	0.0207 (9)	0.0049 (9)
C3	0.0300 (9)	0.0371 (10)	0.0432 (10)	0.0039 (7)	0.0116 (8)	0.0087 (8)
C4	0.0281 (8)	0.0343 (9)	0.0337 (9)	0.0004 (7)	0.0100 (7)	0.0047 (7)
C5	0.0368 (10)	0.0437 (11)	0.0389 (11)	−0.0035 (8)	0.0049 (8)	−0.0012 (9)
C6	0.0514 (13)	0.0329 (10)	0.0562 (13)	−0.0040 (9)	0.0160 (10)	−0.0046 (10)
C7	0.0511 (13)	0.0332 (10)	0.0583 (14)	0.0064 (9)	0.0157 (10)	0.0122 (10)
C8	0.0433 (11)	0.0431 (11)	0.0405 (11)	0.0038 (9)	0.0038 (9)	0.0130 (9)
C9	0.0339 (9)	0.0331 (9)	0.0321 (9)	−0.0002 (7)	0.0097 (7)	0.0025 (7)
C11	0.0416 (11)	0.0342 (10)	0.0429 (11)	−0.0005 (8)	0.0103 (8)	−0.0054 (8)

C12	0.0587 (14)	0.0356 (11)	0.0536 (13)	0.0060 (10)	0.0147 (11)	-0.0019 (10)
C13	0.0590 (15)	0.0477 (13)	0.0632 (15)	0.0141 (12)	0.0134 (12)	-0.0077 (12)
C14	0.0489 (14)	0.0706 (17)	0.0573 (15)	0.0079 (12)	0.0186 (11)	-0.0125 (13)
C15	0.0492 (13)	0.0611 (15)	0.0452 (12)	-0.0055 (11)	0.0135 (10)	0.0010 (11)
C16	0.0439 (11)	0.0415 (11)	0.0455 (12)	0.0020 (9)	0.0083 (9)	0.0024 (9)
Br1	0.03881 (13)	0.06510 (17)	0.06559 (17)	0.00695 (10)	0.00017 (10)	0.02733 (12)
S1	0.0519 (3)	0.0402 (3)	0.0404 (3)	-0.0055 (2)	0.0080 (2)	-0.0061 (2)
S10	0.0637 (4)	0.0289 (3)	0.0928 (5)	0.0022 (2)	0.0440 (4)	0.0058 (3)

Geometric parameters (Å, °)

C2—C3	1.352 (3)	C8—H8	0.92 (3)
C2—S10	1.744 (2)	C9—S1	1.733 (2)
C2—S1	1.745 (2)	C11—C16	1.381 (3)
C3—C4	1.437 (3)	C11—C12	1.395 (3)
C3—Br1	1.873 (2)	C11—S10	1.781 (2)
C4—C9	1.396 (3)	C12—C13	1.380 (3)
C4—C5	1.397 (3)	C12—H12	0.93 (3)
C5—C6	1.373 (3)	C13—C14	1.376 (4)
C5—H5	0.90 (2)	C13—H13	0.98 (3)
C6—C7	1.390 (4)	C14—C15	1.377 (4)
C6—H6	0.91 (3)	C14—H14	0.87 (3)
C7—C8	1.369 (3)	C15—C16	1.385 (3)
C7—H7	0.92 (3)	C15—H15	0.87 (3)
C8—C9	1.397 (3)	C16—H16	0.91 (3)
C3—C2—S10	128.98 (18)	C4—C9—S1	111.78 (14)
C3—C2—S1	111.54 (15)	C8—C9—S1	126.76 (16)
S10—C2—S1	119.44 (14)	C16—C11—C12	119.9 (2)
C2—C3—C4	114.06 (18)	C16—C11—S10	124.14 (17)
C2—C3—Br1	124.17 (16)	C12—C11—S10	115.92 (17)
C4—C3—Br1	121.76 (15)	C13—C12—C11	119.7 (2)
C9—C4—C5	119.07 (18)	C13—C12—H12	118.7 (16)
C9—C4—C3	111.10 (17)	C11—C12—H12	121.4 (17)
C5—C4—C3	129.82 (18)	C14—C13—C12	120.3 (2)
C6—C5—C4	119.2 (2)	C14—C13—H13	120.0 (17)
C6—C5—H5	122.1 (16)	C12—C13—H13	119.7 (17)
C4—C5—H5	118.6 (16)	C13—C14—C15	119.9 (2)
C5—C6—C7	121.1 (2)	C13—C14—H14	121 (2)
C5—C6—H6	119.9 (17)	C15—C14—H14	119 (2)
C7—C6—H6	118.9 (17)	C14—C15—C16	120.5 (2)
C8—C7—C6	121.0 (2)	C14—C15—H15	120.8 (18)
C8—C7—H7	123.0 (18)	C16—C15—H15	118.7 (18)
C6—C7—H7	116.0 (18)	C11—C16—C15	119.6 (2)
C7—C8—C9	118.2 (2)	C11—C16—H16	119.7 (18)
C7—C8—H8	120.6 (18)	C15—C16—H16	120.7 (18)
C9—C8—H8	121.2 (18)	C9—S1—C2	91.51 (10)
C4—C9—C8	121.46 (19)	C2—S10—C11	103.35 (10)

S10—C2—C3—C4	-178.52 (15)	C7—C8—C9—S1	179.94 (17)
S1—C2—C3—C4	-0.7 (2)	C16—C11—C12—C13	0.7 (4)
S10—C2—C3—Br1	1.5 (3)	S10—C11—C12—C13	179.2 (2)
S1—C2—C3—Br1	179.28 (10)	C11—C12—C13—C14	-0.4 (4)
C2—C3—C4—C9	0.2 (2)	C12—C13—C14—C15	-0.1 (4)
Br1—C3—C4—C9	-179.83 (13)	C13—C14—C15—C16	0.3 (4)
C2—C3—C4—C5	-179.69 (19)	C12—C11—C16—C15	-0.5 (3)
Br1—C3—C4—C5	0.3 (3)	S10—C11—C16—C15	-178.85 (18)
C9—C4—C5—C6	0.0 (3)	C14—C15—C16—C11	0.0 (4)
C3—C4—C5—C6	179.8 (2)	C4—C9—S1—C2	-0.72 (15)
C4—C5—C6—C7	-0.7 (3)	C8—C9—S1—C2	179.07 (19)
C5—C6—C7—C8	1.0 (4)	C3—C2—S1—C9	0.82 (16)
C6—C7—C8—C9	-0.5 (3)	S10—C2—S1—C9	178.86 (13)
C5—C4—C9—C8	0.5 (3)	C3—C2—S10—C11	-84.8 (2)
C3—C4—C9—C8	-179.35 (18)	S1—C2—S10—C11	97.56 (14)
C5—C4—C9—S1	-179.67 (14)	C16—C11—S10—C2	-19.9 (2)
C3—C4—C9—S1	0.5 (2)	C12—C11—S10—C2	161.70 (18)
C7—C8—C9—C4	-0.3 (3)		

3-Iodo-2-(phenylsulfanyl)benzo[*b*]thiophene (2)

Crystal data

C₁₄H₉IS₂
M_r = 368.23
 Monoclinic, *P*2₁/*c*
a = 8.4872 (3) Å
b = 9.9629 (4) Å
c = 15.6485 (7) Å
 β = 97.052 (1)°
V = 1313.18 (9) Å³
Z = 4

F(000) = 712
D_x = 1.863 Mg m⁻³
 Mo *K*α radiation, λ = 0.71073 Å
 Cell parameters from 9841 reflections
 θ = 2.6–27.5°
 μ = 2.73 mm⁻¹
T = 294 K
 Parallelepiped, colourless
 0.16 × 0.13 × 0.05 mm

Data collection

Bruker D8 Venture/Photon 100 CMOS
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 Detector resolution: 10.4167 pixels mm⁻¹
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Krause *et al.*, 2015)
T_{min} = 0.690, *T_{max}* = 0.746

56046 measured reflections
 3001 independent reflections
 2585 reflections with *I* > 2σ(*I*)
R_{int} = 0.045
 θ_{\max} = 27.5°, θ_{\min} = 2.6°
h = -11→11
k = -12→12
l = -20→20

Refinement

Refinement on *F*²
 Least-squares matrix: full
R[*F*² > 2σ(*F*²)] = 0.022
wR(*F*²) = 0.051
S = 1.08
 3001 reflections
 190 parameters
 0 restraints

Primary atom site location: structure-invariant
 direct methods
 Secondary atom site location: difference Fourier
 map
 Hydrogen site location: difference Fourier map
 All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.0218P)^2 + 0.8146P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.45 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.85 \text{ e } \text{\AA}^{-3}$$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C2	0.6667 (3)	0.8425 (2)	-0.08322 (15)	0.0350 (5)
C3	0.7271 (2)	0.9337 (2)	-0.02410 (14)	0.0308 (4)
C4	0.6724 (2)	1.0681 (2)	-0.04289 (13)	0.0279 (4)
C5	0.7097 (3)	1.1876 (2)	0.00262 (15)	0.0351 (5)
C6	0.6420 (3)	1.3057 (2)	-0.02866 (17)	0.0415 (5)
C7	0.5388 (3)	1.3091 (2)	-0.10526 (17)	0.0434 (6)
C8	0.5000 (3)	1.1942 (2)	-0.15099 (15)	0.0397 (5)
C9	0.5672 (2)	1.0732 (2)	-0.11926 (13)	0.0306 (4)
C11	0.8796 (3)	0.6562 (2)	-0.13223 (14)	0.0359 (5)
C12	0.9622 (3)	0.5360 (2)	-0.11839 (17)	0.0459 (6)
C13	1.1021 (3)	0.5172 (3)	-0.15346 (19)	0.0547 (7)
C14	1.1602 (3)	0.6167 (3)	-0.20217 (19)	0.0539 (7)
C15	1.0784 (3)	0.7353 (3)	-0.21568 (17)	0.0466 (6)
C16	0.9382 (3)	0.7558 (2)	-0.18094 (16)	0.0408 (5)
S1	0.53633 (8)	0.91592 (6)	-0.16492 (4)	0.04052 (14)
S10	0.69891 (8)	0.66946 (6)	-0.08560 (5)	0.04924 (16)
I1	0.88517 (2)	0.88685 (2)	0.08375 (2)	0.04613 (7)
H5	0.779 (3)	1.183 (3)	0.0538 (17)	0.047 (7)*
H6	0.667 (3)	1.387 (3)	-0.0011 (18)	0.048 (8)*
H7	0.502 (3)	1.394 (3)	-0.1290 (19)	0.059 (9)*
H8	0.434 (3)	1.196 (3)	-0.2021 (17)	0.049 (7)*
H12	0.924 (3)	0.471 (3)	-0.0845 (17)	0.049 (7)*
H13	1.162 (3)	0.435 (3)	-0.1465 (19)	0.061 (8)*
H14	1.251 (4)	0.604 (3)	-0.229 (2)	0.071 (10)*
H15	1.116 (3)	0.797 (3)	-0.2450 (17)	0.043 (7)*
H16	0.882 (3)	0.829 (3)	-0.1901 (18)	0.053 (8)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.0372 (11)	0.0290 (11)	0.0406 (12)	0.0014 (9)	0.0124 (9)	0.0018 (9)
C3	0.0288 (10)	0.0313 (10)	0.0331 (11)	0.0020 (8)	0.0070 (8)	0.0060 (8)
C4	0.0265 (9)	0.0296 (10)	0.0286 (10)	-0.0001 (8)	0.0083 (8)	0.0024 (8)
C5	0.0355 (11)	0.0351 (12)	0.0347 (12)	-0.0020 (9)	0.0036 (9)	-0.0015 (9)
C6	0.0468 (13)	0.0287 (12)	0.0501 (14)	-0.0016 (10)	0.0104 (11)	-0.0024 (10)
C7	0.0490 (14)	0.0316 (12)	0.0501 (14)	0.0057 (10)	0.0089 (11)	0.0110 (10)
C8	0.0425 (12)	0.0415 (13)	0.0345 (12)	0.0017 (10)	0.0020 (10)	0.0118 (10)

C9	0.0330 (10)	0.0313 (10)	0.0281 (10)	-0.0021 (8)	0.0066 (8)	0.0019 (8)
C11	0.0447 (12)	0.0290 (10)	0.0344 (11)	-0.0015 (9)	0.0058 (9)	-0.0068 (9)
C12	0.0600 (16)	0.0310 (13)	0.0482 (15)	0.0038 (11)	0.0125 (12)	-0.0003 (11)
C13	0.0622 (17)	0.0407 (14)	0.0622 (18)	0.0121 (13)	0.0111 (14)	-0.0098 (13)
C14	0.0503 (15)	0.0624 (18)	0.0507 (16)	0.0050 (13)	0.0131 (12)	-0.0176 (14)
C15	0.0530 (15)	0.0495 (15)	0.0386 (13)	-0.0092 (12)	0.0100 (11)	-0.0004 (12)
C16	0.0479 (13)	0.0340 (13)	0.0406 (13)	0.0002 (11)	0.0052 (10)	0.0022 (10)
S1	0.0494 (3)	0.0374 (3)	0.0340 (3)	-0.0051 (2)	0.0024 (2)	-0.0054 (2)
S10	0.0561 (4)	0.0257 (3)	0.0708 (4)	-0.0024 (3)	0.0276 (3)	0.0005 (3)
I1	0.03649 (9)	0.04740 (10)	0.05267 (11)	0.00385 (7)	-0.00193 (6)	0.01649 (7)

Geometric parameters (Å, °)

C2—C3	1.353 (3)	C8—H8	0.92 (3)
C2—S1	1.745 (2)	C9—S1	1.728 (2)
C2—S10	1.746 (2)	C11—C16	1.381 (3)
C3—C4	1.436 (3)	C11—C12	1.391 (3)
C3—I1	2.076 (2)	C11—S10	1.782 (2)
C4—C9	1.402 (3)	C12—C13	1.380 (4)
C4—C5	1.403 (3)	C12—H12	0.92 (3)
C5—C6	1.374 (3)	C13—C14	1.378 (4)
C5—H5	0.93 (3)	C13—H13	0.96 (3)
C6—C7	1.395 (4)	C14—C15	1.374 (4)
C6—H6	0.93 (3)	C14—H14	0.93 (3)
C7—C8	1.369 (4)	C15—C16	1.383 (4)
C7—H7	0.96 (3)	C15—H15	0.85 (3)
C8—C9	1.399 (3)	C16—H16	0.87 (3)
C3—C2—S1	111.84 (17)	C8—C9—S1	126.88 (17)
C3—C2—S10	129.12 (18)	C4—C9—S1	111.62 (15)
S1—C2—S10	119.02 (14)	C16—C11—C12	119.7 (2)
C2—C3—C4	113.64 (19)	C16—C11—S10	123.96 (19)
C2—C3—I1	123.89 (16)	C12—C11—S10	116.29 (19)
C4—C3—I1	122.46 (16)	C13—C12—C11	119.8 (3)
C9—C4—C5	118.87 (19)	C13—C12—H12	120.7 (17)
C9—C4—C3	111.34 (19)	C11—C12—H12	119.5 (17)
C5—C4—C3	129.8 (2)	C14—C13—C12	120.4 (3)
C6—C5—C4	119.2 (2)	C14—C13—H13	117.1 (18)
C6—C5—H5	122.5 (17)	C12—C13—H13	122.5 (18)
C4—C5—H5	118.3 (17)	C15—C14—C13	119.7 (3)
C5—C6—C7	121.2 (2)	C15—C14—H14	118 (2)
C5—C6—H6	121.4 (17)	C13—C14—H14	122 (2)
C7—C6—H6	117.3 (17)	C14—C15—C16	120.7 (3)
C8—C7—C6	120.9 (2)	C14—C15—H15	119.1 (18)
C8—C7—H7	119.3 (18)	C16—C15—H15	120.2 (18)
C6—C7—H7	119.5 (18)	C11—C16—C15	119.7 (2)
C7—C8—C9	118.3 (2)	C11—C16—H16	117.6 (19)
C7—C8—H8	121.6 (18)	C15—C16—H16	122.6 (19)

C9—C8—H8	120.0 (18)	C9—S1—C2	91.54 (11)
C8—C9—C4	121.5 (2)	C2—S10—C11	103.12 (11)
S1—C2—C3—C4	-0.9 (2)	C3—C4—C9—S1	0.7 (2)
S10—C2—C3—C4	-179.35 (17)	C16—C11—C12—C13	0.0 (4)
S1—C2—C3—I1	179.47 (11)	S10—C11—C12—C13	179.1 (2)
S10—C2—C3—I1	1.0 (3)	C11—C12—C13—C14	-0.1 (4)
C2—C3—C4—C9	0.1 (3)	C12—C13—C14—C15	0.1 (4)
I1—C3—C4—C9	179.77 (14)	C13—C14—C15—C16	0.0 (4)
C2—C3—C4—C5	-179.4 (2)	C12—C11—C16—C15	0.1 (4)
I1—C3—C4—C5	0.2 (3)	S10—C11—C16—C15	-178.93 (19)
C9—C4—C5—C6	0.2 (3)	C14—C15—C16—C11	-0.1 (4)
C3—C4—C5—C6	179.7 (2)	C8—C9—S1—C2	178.9 (2)
C4—C5—C6—C7	-0.9 (4)	C4—C9—S1—C2	-1.01 (17)
C5—C6—C7—C8	0.9 (4)	C3—C2—S1—C9	1.09 (17)
C6—C7—C8—C9	-0.3 (4)	S10—C2—S1—C9	179.73 (14)
C7—C8—C9—C4	-0.3 (3)	C3—C2—S10—C11	-83.5 (2)
C7—C8—C9—S1	179.78 (19)	S1—C2—S10—C11	98.17 (15)
C5—C4—C9—C8	0.4 (3)	C16—C11—S10—C2	-20.5 (2)
C3—C4—C9—C8	-179.2 (2)	C12—C11—S10—C2	160.48 (19)
C5—C4—C9—S1	-179.70 (16)		

3-Bromo-2-(phenylselanyl)benzo[*b*]selenophene (3)

Crystal data

C₁₄H₉BrSe₂
M_r = 415.04
 Monoclinic, *P*2₁/*c*
a = 12.3864 (11) Å
b = 13.6816 (11) Å
c = 8.0982 (6) Å
 β = 96.398 (3)°
V = 1363.82 (19) Å³
Z = 4

F(000) = 784
D_x = 2.021 Mg m⁻³
 Mo *K*α radiation, λ = 0.71073 Å
 Cell parameters from 9947 reflections
 θ = 2.9–27.1°
 μ = 8.33 mm⁻¹
T = 297 K
 Parallelepiped, colourless
 0.17 × 0.17 × 0.12 mm

Data collection

Bruker D8 Venture/Photon 100 CMOS
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 Detector resolution: 10.4167 pixels mm⁻¹
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Krause *et al.*, 2015)
T_{min} = 0.543, *T_{max}* = 0.746

43875 measured reflections
 2976 independent reflections
 2208 reflections with *I* > 2σ(*I*)
R_{int} = 0.060
 θ_{\max} = 27.0°, θ_{\min} = 3.0°
h = -15→15
k = -17→17
l = -10→10

Refinement

Refinement on *F*²
 Least-squares matrix: full
R [*F*² > 2σ(*F*²)] = 0.035
wR(*F*²) = 0.069
S = 1.03

2976 reflections
 154 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier map
 Hydrogen site location: inferred from neighbouring sites
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0201P)^2 + 2.3843P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.93 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.90 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.27259 (4)	0.68913 (3)	-0.08792 (6)	0.06404 (14)
C2	0.2358 (3)	0.5151 (2)	0.0834 (5)	0.0433 (8)
C3	0.2903 (3)	0.5551 (3)	-0.0323 (5)	0.0451 (8)
C4	0.3618 (3)	0.4929 (3)	-0.1127 (4)	0.0465 (9)
C5	0.4294 (3)	0.5160 (4)	-0.2330 (5)	0.0632 (12)
H5	0.431618	0.579335	-0.274038	0.076*
C6	0.4933 (4)	0.4437 (5)	-0.2911 (5)	0.0795 (16)
H6	0.540102	0.459228	-0.369395	0.095*
C7	0.4887 (4)	0.3486 (5)	-0.2344 (6)	0.0819 (16)
H7	0.531156	0.300749	-0.277171	0.098*
C8	0.4225 (4)	0.3237 (4)	-0.1160 (6)	0.0700 (13)
H8	0.419621	0.259777	-0.077799	0.084*
C9	0.3605 (3)	0.3961 (3)	-0.0552 (5)	0.0496 (9)
Se1	0.26699 (4)	0.38117 (3)	0.11070 (6)	0.05761 (13)
Se10	0.13853 (4)	0.58065 (3)	0.21047 (6)	0.06282 (15)
C11	0.1055 (3)	0.4715 (2)	0.3458 (4)	0.0422 (8)
C12	0.1734 (4)	0.4475 (3)	0.4854 (5)	0.0616 (11)
H12	0.236377	0.483315	0.515093	0.074*
C13	0.1472 (5)	0.3698 (4)	0.5807 (6)	0.0723 (14)
H13	0.193489	0.352550	0.674522	0.087*
C14	0.0552 (4)	0.3181 (3)	0.5402 (6)	0.0653 (13)
H14	0.038533	0.265737	0.606089	0.078*
C15	-0.0132 (4)	0.3424 (3)	0.4031 (6)	0.0611 (11)
H15	-0.076485	0.306615	0.375672	0.073*
C16	0.0109 (3)	0.4202 (3)	0.3041 (5)	0.0475 (9)
H16	-0.036003	0.437373	0.210910	0.057*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0617 (3)	0.0504 (2)	0.0805 (3)	-0.0051 (2)	0.0103 (2)	0.0238 (2)
C2	0.043 (2)	0.0332 (17)	0.054 (2)	-0.0027 (15)	0.0087 (17)	-0.0002 (15)
C3	0.043 (2)	0.0412 (19)	0.051 (2)	-0.0057 (16)	0.0034 (17)	0.0038 (16)
C4	0.036 (2)	0.063 (2)	0.0385 (19)	-0.0047 (18)	-0.0016 (16)	-0.0023 (17)

C5	0.045 (2)	0.098 (4)	0.045 (2)	-0.002 (2)	-0.0015 (19)	0.001 (2)
C6	0.052 (3)	0.145 (5)	0.042 (2)	0.007 (3)	0.008 (2)	-0.008 (3)
C7	0.070 (3)	0.117 (5)	0.057 (3)	0.027 (3)	0.001 (3)	-0.026 (3)
C8	0.070 (3)	0.075 (3)	0.063 (3)	0.016 (2)	-0.001 (2)	-0.021 (2)
C9	0.045 (2)	0.057 (2)	0.046 (2)	0.0023 (18)	0.0009 (17)	-0.0117 (18)
Se1	0.0678 (3)	0.0342 (2)	0.0741 (3)	0.00042 (18)	0.0227 (2)	0.00167 (18)
Se10	0.0762 (3)	0.0350 (2)	0.0840 (3)	0.00590 (19)	0.0385 (3)	0.00663 (19)
C11	0.046 (2)	0.0352 (18)	0.048 (2)	0.0015 (16)	0.0148 (17)	-0.0002 (15)
C12	0.051 (3)	0.061 (3)	0.070 (3)	0.000 (2)	-0.008 (2)	-0.002 (2)
C13	0.089 (4)	0.066 (3)	0.058 (3)	0.023 (3)	-0.009 (3)	0.012 (2)
C14	0.095 (4)	0.045 (2)	0.060 (3)	0.017 (2)	0.030 (3)	0.015 (2)
C15	0.063 (3)	0.049 (2)	0.075 (3)	-0.012 (2)	0.024 (2)	-0.006 (2)
C16	0.046 (2)	0.051 (2)	0.045 (2)	-0.0014 (18)	0.0037 (17)	-0.0043 (17)

Geometric parameters (Å, °)

Br1—C3	1.895 (4)	C8—H8	0.9300
C2—C3	1.333 (5)	C9—Se1	1.881 (4)
C2—Se1	1.881 (3)	Se10—C11	1.923 (3)
C2—Se10	1.894 (3)	C11—C12	1.372 (5)
C3—C4	1.435 (5)	C11—C16	1.375 (5)
C4—C5	1.391 (5)	C12—C13	1.374 (6)
C4—C9	1.404 (6)	C12—H12	0.9300
C5—C6	1.381 (7)	C13—C14	1.350 (7)
C5—H5	0.9300	C13—H13	0.9300
C6—C7	1.383 (8)	C14—C15	1.361 (6)
C6—H6	0.9300	C14—H14	0.9300
C7—C8	1.372 (7)	C15—C16	1.385 (5)
C7—H7	0.9300	C15—H15	0.9300
C8—C9	1.378 (6)	C16—H16	0.9300
C3—C2—Se1	111.6 (3)	C8—C9—Se1	126.1 (4)
C3—C2—Se10	126.2 (3)	C4—C9—Se1	111.7 (3)
Se1—C2—Se10	122.15 (18)	C9—Se1—C2	86.85 (16)
C2—C3—C4	117.5 (3)	C2—Se10—C11	97.51 (14)
C2—C3—Br1	120.7 (3)	C12—C11—C16	120.5 (4)
C4—C3—Br1	121.8 (3)	C12—C11—Se10	120.4 (3)
C5—C4—C9	118.4 (4)	C16—C11—Se10	119.1 (3)
C5—C4—C3	129.3 (4)	C11—C12—C13	119.2 (4)
C9—C4—C3	112.3 (3)	C11—C12—H12	120.4
C6—C5—C4	119.3 (5)	C13—C12—H12	120.4
C6—C5—H5	120.3	C14—C13—C12	121.0 (4)
C4—C5—H5	120.3	C14—C13—H13	119.5
C5—C6—C7	120.9 (5)	C12—C13—H13	119.5
C5—C6—H6	119.5	C13—C14—C15	120.1 (4)
C7—C6—H6	119.5	C13—C14—H14	119.9
C8—C7—C6	121.0 (5)	C15—C14—H14	119.9
C8—C7—H7	119.5	C14—C15—C16	120.4 (4)

C6—C7—H7	119.5	C14—C15—H15	119.8
C7—C8—C9	118.1 (5)	C16—C15—H15	119.8
C7—C8—H8	120.9	C11—C16—C15	118.9 (4)
C9—C8—H8	120.9	C11—C16—H16	120.5
C8—C9—C4	122.2 (4)	C15—C16—H16	120.5
Se1—C2—C3—C4	-0.7 (5)	C5—C4—C9—Se1	178.4 (3)
Se10—C2—C3—C4	178.9 (3)	C3—C4—C9—Se1	-0.9 (4)
Se1—C2—C3—Br1	179.03 (19)	C8—C9—Se1—C2	179.9 (4)
Se10—C2—C3—Br1	-1.4 (5)	C4—C9—Se1—C2	0.5 (3)
C2—C3—C4—C5	-178.2 (4)	C3—C2—Se1—C9	0.1 (3)
Br1—C3—C4—C5	2.1 (6)	Se10—C2—Se1—C9	-179.5 (2)
C2—C3—C4—C9	1.1 (5)	C3—C2—Se10—C11	-177.6 (4)
Br1—C3—C4—C9	-178.6 (3)	Se1—C2—Se10—C11	1.9 (3)
C9—C4—C5—C6	-0.4 (6)	C16—C11—C12—C13	1.7 (6)
C3—C4—C5—C6	178.8 (4)	Se10—C11—C12—C13	179.3 (3)
C4—C5—C6—C7	1.7 (7)	C11—C12—C13—C14	-1.0 (7)
C5—C6—C7—C8	-1.6 (8)	C12—C13—C14—C15	0.1 (7)
C6—C7—C8—C9	0.1 (7)	C13—C14—C15—C16	0.0 (7)
C7—C8—C9—C4	1.2 (6)	C12—C11—C16—C15	-1.5 (6)
C7—C8—C9—Se1	-178.2 (3)	Se10—C11—C16—C15	-179.2 (3)
C5—C4—C9—C8	-1.0 (6)	C14—C15—C16—C11	0.7 (6)
C3—C4—C9—C8	179.6 (4)		

3-Iodo-2-(phenylselanyl)benzo[*b*]selenophene (4)

Crystal data

C₁₄H₉ISE₂
M_r = 462.03
 Monoclinic, *P*2₁/*c*
a = 12.9606 (6) Å
b = 13.5999 (7) Å
c = 8.0448 (4) Å
 β = 95.585 (2)°
V = 1411.27 (12) Å³
Z = 4

F(000) = 856
D_x = 2.175 Mg m⁻³
 Mo *K*α radiation, λ = 0.71073 Å
 Cell parameters from 9830 reflections
 θ = 3.0–27.9°
 μ = 7.40 mm⁻¹
T = 292 K
 Parallelepiped, colourless
 0.51 × 0.47 × 0.24 mm

Data collection

Bruker D8 Venture/Photon 100 CMOS
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 Detector resolution: 10.4167 pixels mm⁻¹
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Krause *et al.*, 2015)
T_{min} = 0.390, *T_{max}* = 0.746

54241 measured reflections
 3391 independent reflections
 2702 reflections with *I* > 2σ(*I*)
R_{int} = 0.054
 θ_{max} = 28.0°, θ_{min} = 3.0°
h = -17→17
k = -17→17
l = -10→10

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.037$ $wR(F^2) = 0.083$ $S = 1.04$

3391 reflections

190 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
map

Hydrogen site location: difference Fourier map

All H-atom parameters refined

 $w = 1/[\sigma^2(F_o^2) + (0.030P)^2 + 4.781P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 1.14 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -1.82 \text{ e } \text{\AA}^{-3}$ *Special details*

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C2	0.7614 (3)	0.4907 (3)	0.4123 (5)	0.0348 (9)
C3	0.7104 (3)	0.4513 (3)	0.5349 (5)	0.0353 (9)
C4	0.6366 (3)	0.5137 (4)	0.6061 (5)	0.0395 (10)
C5	0.5726 (4)	0.4915 (5)	0.7306 (6)	0.0511 (13)
C6	0.5065 (4)	0.5634 (6)	0.7810 (7)	0.0655 (18)
C7	0.5049 (5)	0.6563 (6)	0.7130 (8)	0.0693 (19)
C8	0.5669 (5)	0.6814 (5)	0.5885 (7)	0.0587 (15)
C9	0.6321 (4)	0.6082 (4)	0.5366 (6)	0.0447 (11)
C11	0.8883 (3)	0.5295 (3)	0.1453 (5)	0.0354 (9)
C12	0.9754 (4)	0.5864 (4)	0.1749 (6)	0.0407 (10)
C13	0.9950 (4)	0.6605 (4)	0.0638 (7)	0.0512 (13)
C14	0.9290 (5)	0.6751 (4)	-0.0757 (7)	0.0569 (15)
C15	0.8411 (6)	0.6181 (5)	-0.1068 (8)	0.0673 (17)
C16	0.8210 (5)	0.5442 (5)	0.0037 (8)	0.0566 (14)
I1	0.74197 (2)	0.30752 (2)	0.61759 (4)	0.04627 (11)
Se1	0.72215 (4)	0.62182 (4)	0.36770 (7)	0.05061 (15)
Se10	0.86334 (4)	0.42591 (4)	0.29872 (7)	0.04875 (15)
H5	0.575 (4)	0.430 (4)	0.777 (6)	0.033 (12)*
H6	0.465 (5)	0.541 (5)	0.859 (8)	0.08 (2)*
H8	0.571 (5)	0.750 (5)	0.540 (8)	0.069 (19)*
H7	0.459 (5)	0.703 (5)	0.757 (8)	0.08 (2)*
H12	1.022 (4)	0.576 (4)	0.263 (7)	0.052 (16)*
H13	1.057 (5)	0.701 (5)	0.085 (8)	0.068 (18)*
H14	0.941 (6)	0.724 (6)	-0.147 (9)	0.09 (2)*
H15	0.793 (6)	0.631 (5)	-0.196 (9)	0.09 (2)*
H16	0.768 (5)	0.507 (5)	-0.015 (8)	0.08 (2)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.035 (2)	0.028 (2)	0.041 (2)	-0.0025 (17)	0.0056 (18)	-0.0003 (17)
C3	0.033 (2)	0.036 (2)	0.037 (2)	-0.0010 (17)	0.0029 (17)	-0.0001 (18)
C4	0.034 (2)	0.050 (3)	0.034 (2)	-0.0012 (19)	-0.0009 (17)	-0.0065 (19)
C5	0.041 (3)	0.072 (4)	0.039 (3)	0.004 (2)	0.003 (2)	-0.007 (3)
C6	0.044 (3)	0.111 (6)	0.042 (3)	0.007 (3)	0.008 (2)	-0.015 (3)
C7	0.062 (4)	0.090 (5)	0.054 (3)	0.033 (3)	0.000 (3)	-0.024 (3)
C8	0.064 (3)	0.059 (4)	0.051 (3)	0.023 (3)	-0.006 (3)	-0.014 (3)
C9	0.040 (2)	0.050 (3)	0.043 (3)	0.007 (2)	0.000 (2)	-0.009 (2)
C11	0.037 (2)	0.032 (2)	0.039 (2)	0.0015 (17)	0.0112 (18)	-0.0031 (17)
C12	0.040 (2)	0.042 (3)	0.040 (2)	-0.004 (2)	0.008 (2)	-0.003 (2)
C13	0.054 (3)	0.039 (3)	0.064 (3)	-0.012 (2)	0.021 (3)	-0.004 (2)
C14	0.079 (4)	0.038 (3)	0.057 (3)	0.008 (3)	0.025 (3)	0.014 (2)
C15	0.076 (4)	0.070 (4)	0.054 (3)	0.013 (3)	-0.008 (3)	0.007 (3)
C16	0.052 (3)	0.055 (3)	0.061 (3)	-0.009 (3)	-0.006 (3)	0.001 (3)
I1	0.04859 (18)	0.04025 (18)	0.05068 (19)	-0.00477 (13)	0.00843 (13)	0.01099 (13)
Se1	0.0609 (3)	0.0314 (3)	0.0616 (3)	0.0050 (2)	0.0163 (2)	0.0066 (2)
Se10	0.0558 (3)	0.0320 (3)	0.0628 (3)	0.0051 (2)	0.0282 (2)	0.0073 (2)

Geometric parameters (\AA , $^\circ$)

C2—C3	1.351 (6)	C8—H8	1.01 (7)
C2—Se1	1.880 (4)	C9—Se1	1.885 (5)
C2—Se10	1.895 (4)	C11—C12	1.370 (6)
C3—C4	1.438 (6)	C11—C16	1.380 (7)
C3—I1	2.093 (4)	C11—Se10	1.921 (4)
C4—C5	1.393 (7)	C12—C13	1.387 (7)
C4—C9	1.402 (7)	C12—H12	0.90 (6)
C5—C6	1.386 (8)	C13—C14	1.358 (9)
C5—H5	0.92 (5)	C13—H13	0.97 (6)
C6—C7	1.377 (10)	C14—C15	1.381 (9)
C6—H6	0.92 (7)	C14—H14	0.90 (7)
C7—C8	1.386 (10)	C15—C16	1.383 (9)
C7—H7	0.96 (7)	C15—H15	0.92 (7)
C8—C9	1.396 (7)	C16—H16	0.86 (7)
C3—C2—Se1	111.9 (3)	C8—C9—Se1	125.5 (5)
C3—C2—Se10	125.6 (3)	C4—C9—Se1	111.9 (3)
Se1—C2—Se10	122.5 (2)	C12—C11—C16	120.4 (5)
C2—C3—C4	116.7 (4)	C12—C11—Se10	119.3 (4)
C2—C3—I1	120.5 (3)	C16—C11—Se10	120.3 (4)
C4—C3—I1	122.8 (3)	C11—C12—C13	119.8 (5)
C5—C4—C9	118.7 (5)	C11—C12—H12	122 (4)
C5—C4—C3	128.6 (5)	C13—C12—H12	118 (4)
C9—C4—C3	112.7 (4)	C14—C13—C12	119.9 (5)
C6—C5—C4	119.1 (6)	C14—C13—H13	120 (4)

C6—C5—H5	122 (3)	C12—C13—H13	120 (4)
C4—C5—H5	119 (3)	C13—C14—C15	120.7 (5)
C7—C6—C5	121.1 (6)	C13—C14—H14	120 (5)
C7—C6—H6	127 (4)	C15—C14—H14	119 (5)
C5—C6—H6	112 (5)	C14—C15—C16	119.7 (6)
C6—C7—C8	121.8 (5)	C14—C15—H15	121 (5)
C6—C7—H7	117 (4)	C16—C15—H15	119 (5)
C8—C7—H7	122 (4)	C11—C16—C15	119.5 (5)
C7—C8—C9	116.8 (6)	C11—C16—H16	119 (5)
C7—C8—H8	124 (4)	C15—C16—H16	121 (5)
C9—C8—H8	119 (4)	C2—Se1—C9	86.8 (2)
C8—C9—C4	122.5 (5)	C2—Se10—C11	97.92 (18)
Se1—C2—C3—C4	0.8 (5)	C5—C4—C9—Se1	-178.7 (4)
Se10—C2—C3—C4	179.3 (3)	C3—C4—C9—Se1	1.6 (5)
Se1—C2—C3—I1	-177.8 (2)	C16—C11—C12—C13	1.4 (7)
Se10—C2—C3—I1	0.7 (6)	Se10—C11—C12—C13	179.1 (4)
C2—C3—C4—C5	178.7 (5)	C11—C12—C13—C14	-1.3 (8)
I1—C3—C4—C5	-2.7 (7)	C12—C13—C14—C15	1.2 (9)
C2—C3—C4—C9	-1.6 (6)	C13—C14—C15—C16	-1.1 (10)
I1—C3—C4—C9	177.0 (3)	C12—C11—C16—C15	-1.3 (8)
C9—C4—C5—C6	0.3 (7)	Se10—C11—C16—C15	-178.9 (5)
C3—C4—C5—C6	180.0 (5)	C14—C15—C16—C11	1.1 (10)
C4—C5—C6—C7	-1.5 (8)	C3—C2—Se1—C9	0.1 (3)
C5—C6—C7—C8	1.8 (9)	Se10—C2—Se1—C9	-178.5 (3)
C6—C7—C8—C9	-0.8 (9)	C8—C9—Se1—C2	179.7 (5)
C7—C8—C9—C4	-0.5 (8)	C4—C9—Se1—C2	-1.0 (4)
C7—C8—C9—Se1	178.8 (4)	C3—C2—Se10—C11	177.6 (4)
C5—C4—C9—C8	0.7 (7)	Se1—C2—Se10—C11	-4.0 (3)
C3—C4—C9—C8	-179.1 (5)		