



# Crystal structure of 1-iodo-3-[[4-(*tert*-butylsulfanyl)phenyl]ethynyl]azulene

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Received 13 June 2015; accepted 30 June 2015

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

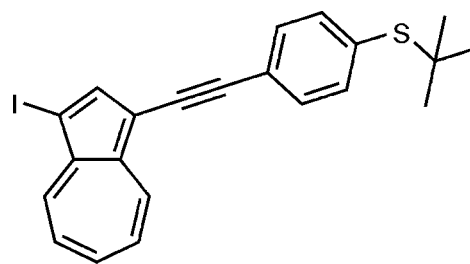
The title compound, C<sub>20</sub>H<sub>19</sub>IS, features a 1,3-disubstituted azulene involving an ethynylene elongated 4-(*tert*-butylsulfanyl)phenyl sidearm and an iodine atom as the substituents. The azulene ring system is almost planar (r.m.s. deviation = 0.012 Å) and subtends a dihedral angle of 35.7 (1)° with the benzene ring. As a result of the inherent dipole character of the azulene core, a supramolecular  $\pi$ - $\pi$  dimer [separation between the centroids of the five- and seven-membered rings = 3.7632 (10) Å] with antiparallel orientated molecules can be observed in the crystal. The packing is consolidated by an unusual I... $\pi$ (acetylene) contact [I...Cg = 3.34 Å, C—I...Cg = 173.3°], and a very weak C—H... $\pi$  interaction is also found in the structure, with the azulene five-membered ring as the acceptor.

**Keywords:** crystal structure; azulene; 1,3-disubstitution; C—H... $\pi$  interaction; I... $\pi$  contact.

**CCDC reference:** 1409619

## 1. Related literature

For background to this work, see: Strachota *et al.* (2008); Xia *et al.* (2014). For related structures, see: Förster *et al.* (2012, 2014). For the synthesis of the starting compounds 1,3-diiodoazulene and 1-(*tert*-butylsulfanyl)-4-ethynylbenzene, see: Wakabayashi *et al.* (1998); Mayor *et al.* (2003). For the Sonogashira–Hagihara cross-coupling reaction, see: Sonogashira *et al.* (1975). For I... $\pi$  contacts, see: Forni *et al.* (2012). For C—H... $\pi$  interactions, see: Nishio *et al.* (2009).



## 2. Experimental

### 2.1. Crystal data

C <sub>20</sub> H <sub>19</sub> IS	V = 1923.68 (6) Å <sup>3</sup>
M <sub>r</sub> = 442.33	Z = 4
Monoclinic, P2 <sub>1</sub> /n	Mo K $\alpha$ radiation
a = 12.7222 (2) Å	$\mu$ = 1.77 mm <sup>-1</sup>
b = 11.9892 (2) Å	T = 100 K
c = 13.7895 (3) Å	0.28 × 0.20 × 0.13 mm
$\beta$ = 113.851 (1)°	

### 2.2. Data collection

Bruker Kappa Apex CCD diffractometer	23474 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2008)	5163 independent reflections
T <sub>min</sub> = 0.637, T <sub>max</sub> = 0.802	4688 reflections with I > 2 $\sigma$ (I)
	R <sub>int</sub> = 0.024

### 2.3. Refinement

R[F <sup>2</sup> > 2 $\sigma$ (F <sup>2</sup> )] = 0.021	220 parameters
wR(F <sup>2</sup> ) = 0.059	H-atom parameters constrained
S = 1.04	$\Delta\rho_{\max}$ = 1.37 e Å <sup>-3</sup>
5163 reflections	$\Delta\rho_{\min}$ = -0.39 e Å <sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the C1–C4/C10 ring.

D—H...A	D—H	H...A	D...A	D—H...A
C17—H17...Cg1 <sup>i</sup>	0.95	2.95	3.471 (2)	116

Symmetry code: (i)  $-x + 1, -y, -z$ .

Data collection: APEX2 (Bruker, 2010); cell refinement: SAINT-NT (Sheldrick, 2008); data reduction: SAINT-NT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL2013 (Sheldrick, 2015); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012); software used to prepare material for publication: SHELXTL (Sheldrick, 2008).

## Acknowledgements

This work has been performed within the Cluster of Excellence: 'Structure Design of Novel High-Performance Materials via Atomic Design and Defect Engineering' (ADDE), which was supported financially by the European Union (European Regional Development Fund) and by the Ministry of Science and Art of Saxony (SMWK).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7447).

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## References

- Bruker (2008). *SAINT-NT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2010). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
- Forni, A., Pieraccini, S., Rendine, S., Gabas, F. & Sironi, M. (2012). *ChemPhysChem*, **13**, 4224–4234.
- Förster, S., Hahn, T., Loose, C., Röder, C., Liebing, S., Seichter, W., Eissmann, F., Kortus, J. & Weber, E. (2012). *J. Phys. Org. Chem.* **25**, 856–863.
- Förster, S., Seichter, W., Kuhnert, R. & Weber, E. (2014). *J. Mol. Struct.* **1075**, 63–70.
- Mayor, M., Weber, H. B., Reichert, J., Elbing, M., von Hänisch, C., Beckmann, D. & Fischer, M. (2003). *Angew. Chem. Int. Ed.* **42**, 5834–5838.
- Nishio, M., Umezawa, Y., Honda, K., Tsuboyama, S. & Suezawa, H. (2009). *CrystEngComm*, **11**, 1757–1788.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.
- Sonogashira, K., Tohda, Y. & Hagihara, N. (1975). *Tetrahedron Lett.* **16**, 4467–4470.
- Strachota, A., Cimrová, V. & Thorn-Csányi, E. (2008). *Macromol. Symp.* **268**, 66–71.
- Wakabayashi, H., Kurihara, T., Shindo, K., Tsukada, M., Yang, P., Yasunami, M. & Nozoe, T. (1998). *J. Chin. Chem. Soc.* **45**, 391–400.
- Xia, J., Capozzi, B., Wei, S., Strange, M., Batra, A., Moreno, J. R., Amir, R. J., Amir, E., Solomon, G. C., Venkataraman, L. & Campos, L. M. (2014). *Nano Lett.* **14**, 2941–2945.

## supporting information

*Acta Cryst.* (2015). E71, o544–o545 [https://doi.org/10.1107/S2056989015012542]

Crystal structure of 1-iodo-3-{[4-(*tert*-butylsulfanyl)phenyl]ethynyl}azulene

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## S1. Comment

Owing to the remarkable electronic and optical properties of azulene and its derivatives, compounds of this type have recently received enormous interest for application in the field of molecular electronics (Strachota *et al.*, 2008; Xia *et al.*, 2014) Being connected to this, the title compound, C<sub>20</sub>H<sub>19</sub>I<sub>S</sub>, is a valuable intermediate in the preparation of corresponding azulenes having a characteristic 1,3-substitution pattern (Förster *et al.*, 2012).

The crystal structure of the compound contains one molecule in the asymmetric part of the unit cell. The molecule deviates from planarity, which can be reflected by a twist angle of 35.7° with respect to the aromatic ring systems of benzene and azulene. The ability of azulene to shift electronic density from the seven-membered ring element toward the five-membered part of the ring system has already been shown to exercise a controlling effect on the supramolecular interactions in the crystal especially regarding  $\pi$ -stacking behavior (Förster *et al.*, 2014). In the present case, supramolecular dimers with a head-to-tail fashioned orientation of the azulene cores are found (3.44 Å). On the side facing away from the azulene-contact, a benzene ring is located. However, considering the distance and geometry, the existence of a  $\pi\cdots\pi$ -interaction can be excluded. Instead, the presence of a weak C—H $\cdots\pi$ -interaction [H17 $\cdots$ Cg(C1, C10) 2.87 Å] is assumed (Nishio *et al.* 2009). In the crystal of the title compound, the packing is furthermore consolidated by a remarkable I $\cdots\pi$ -interaction (Forni *et al.* 2012) [I1 $\cdots$ Cg(C11, C12) 3.34 Å, 173.3°].

## S2. Experimental

## S2.1. Synthesis and crystallization

The title compound was prepared using a Sonogashira–Hagihara cross-coupling reaction (Sonogashira *et al.*, 1975), starting with 1,3-diiodoazulene (Wakabayashi *et al.*, 1998) and 1-(*tert*-butylsulfanyl)-4-ethynylbenzene (Mayor *et al.*, 2003). The aryl iodide (8.0 g, 21.06 mmol) and the terminal alkyne (4.0 g, 21.06 mmol) were dissolved in 200 ml of diisopropylamine. After degassing of the solution and cooling to -30 °C, a mixture of bis(triphenylphosphane)palladium(II) chloride (110 mg, 0.16 mmol) and copper(I) iodide (60 mg, 0.32 mmol) was added. The mixture was stirred for 18 h at room temperature. After evaporation of the solvent, the residue was purified by column chromatography on SiO<sub>2</sub> [60 F254 Merck, eluent: hexane] to yield 4.11 g (44 %) of the title compound as a green solid. In addition to the product, 440 mg of 1,3-diiodoazulene and 80 mg of 1,3-bis{[4-(*tert*-butylsulfanyl)phenyl]-ethynyl}azulene were isolated. Analytical data: mp = 120 °C; <sup>1</sup>H-NMR: (CDCl<sub>3</sub>)  $\delta$ /ppm = 1.32 (s, 9H CH<sub>3</sub>), 7.35 (t, 2H, ArH, <sup>3</sup>J<sub>HH</sub>=9.8 Hz), 7.54 (m, 4H, ArH), 7.72 (d, 1H, ArH, <sup>3</sup>J<sub>HH</sub>=9.9 Hz), 8.10 (s, 1H, ArH), 8.25 (d, 1H, ArH, <sup>3</sup>J<sub>HH</sub>=9.7 Hz), 8.57 (d, 1H, ArH, <sup>3</sup>J<sub>HH</sub>=9.5 Hz); <sup>13</sup>C-NMR: (CDCl<sub>3</sub>)  $\delta$ /ppm = 31.01 (CH<sub>3</sub>), 46.50 (C-(CH<sub>3</sub>)<sub>3</sub>), 74.70 (ArC-I), 86.06 (C≡C), 94.34 (C≡C), 112.17 (ArC), 124.23 (ArC), 125.19 (ArC), 125.61 (ArC), 131.19 (ArC), 132.77 (ArC), 136.15 (ArC), 137.33 (ArC), 139.75 (ArC), 139.77 (ArC), 140.83 (ArC), 141.84 (ArC), 145.77 (ArC); GC/MS calc.: 442; found: 442 [M]<sup>+</sup>; EA calc.: C: 59.73 %, H: 4.33 %, S: 7.25 %; found C: 60.03 %, H: 4.35 %, S: 7.21 %; Crystallization

by slow solvent evaporation from acetone solution yielded suitable crystals.

## S2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The hydrogen atoms attached to C were fixed geometrically and treated as riding atoms, with  $d(\text{C—H}) = 0.93$  and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for aromatic and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$  for methyl groups.

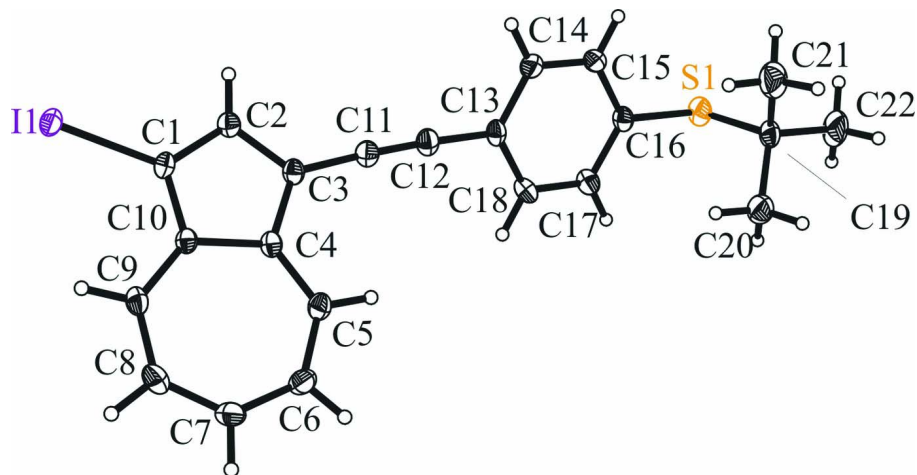


Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

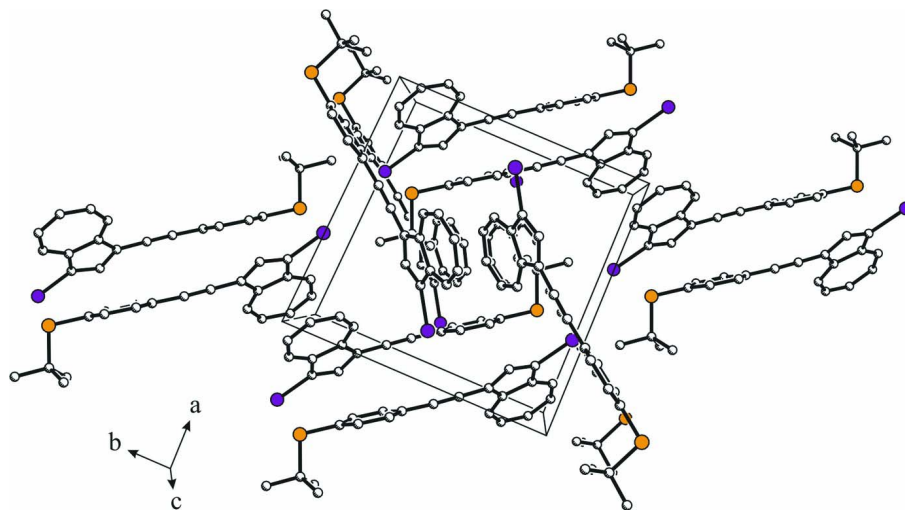


Figure 2

A view along the  $c$  axis of the crystal packing of the title compound.

### 3-[[4-(*tert*-Butylsulfanyl)phenyl]ethynyl]-1-iodo-azulene

#### Crystal data

$\text{C}_{22}\text{H}_{19}\text{IS}$

$M_r = 442.33$

Monoclinic,  $P2_1/n$

$a = 12.7222(2) \text{ \AA}$

$b = 11.9892(2) \text{ \AA}$

$c = 13.7895(3) \text{ \AA}$

$\beta = 113.851(1)^\circ$

$V = 1923.68(6) \text{ \AA}^3$

$Z = 4$

$F(000) = 880$

$D_x = 1.527 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$   
 Cell parameters from 9945 reflections  
 $\theta = 2.5\text{--}33.1^\circ$

$\mu = 1.77 \text{ mm}^{-1}$   
 $T = 100 \text{ K}$   
 Irregular, violet  
 $0.28 \times 0.20 \times 0.13 \text{ mm}$

#### Data collection

Bruker Kappa Apex CCD  
 diffractometer  
 phi and  $\omega$  scans  
 Absorption correction: multi-scan  
 (SADABS; Bruker, 2008)  
 $T_{\min} = 0.637$ ,  $T_{\max} = 0.802$   
 23474 measured reflections

5163 independent reflections  
 4688 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.024$   
 $\theta_{\max} = 29.1^\circ$ ,  $\theta_{\min} = 1.8^\circ$   
 $h = -17 \rightarrow 17$   
 $k = -16 \rightarrow 16$   
 $l = -18 \rightarrow 18$

#### Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.021$   
 $wR(F^2) = 0.059$   
 $S = 1.04$   
 5163 reflections  
 220 parameters  
 0 restraints

Hydrogen site location: inferred from  
 neighbouring sites  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0333P)^2 + 0.9341P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 1.37 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.39 \text{ e \AA}^{-3}$

#### Special details

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

#### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
I1	0.84013 (2)	0.46782 (2)	0.23899 (2)	0.02206 (5)
S2	0.11506 (4)	-0.30969 (4)	0.07255 (4)	0.02563 (10)
C1	0.69313 (13)	0.37992 (13)	0.14949 (13)	0.0173 (3)
C2	0.64454 (13)	0.29623 (13)	0.18826 (13)	0.0179 (3)
H2	0.6733	0.2710	0.2596	0.022*
C3	0.54571 (13)	0.25519 (13)	0.10422 (13)	0.0173 (3)
C4	0.53172 (13)	0.31415 (13)	0.01096 (13)	0.0163 (3)
C5	0.44468 (14)	0.29644 (14)	-0.08807 (14)	0.0204 (3)
H5	0.3916	0.2391	-0.0919	0.024*
C6	0.42455 (15)	0.35115 (15)	-0.18259 (14)	0.0236 (3)
H6	0.3600	0.3256	-0.2426	0.028*
C7	0.48585 (16)	0.43818 (16)	-0.20148 (14)	0.0238 (3)
H7	0.4551	0.4657	-0.2721	0.029*
C8	0.58518 (16)	0.49110 (16)	-0.13288 (16)	0.0249 (4)
H8	0.6140	0.5480	-0.1634	0.030*
C9	0.64791 (15)	0.47161 (13)	-0.02540 (15)	0.0207 (3)
H9	0.7142	0.5168	0.0076	0.025*
C10	0.62711 (13)	0.39541 (13)	0.04023 (13)	0.0160 (3)

C11	0.47274 (14)	0.16724 (14)	0.10849 (14)	0.0197 (3)
C12	0.41110 (14)	0.09113 (14)	0.10767 (14)	0.0214 (3)
C13	0.34086 (13)	-0.00436 (14)	0.10156 (14)	0.0183 (3)
C14	0.31147 (16)	-0.03408 (14)	0.18573 (15)	0.0230 (4)
H14	0.3377	0.0096	0.2486	0.028*
C15	0.24401 (15)	-0.12745 (15)	0.17689 (14)	0.0219 (3)
H15	0.2249	-0.1478	0.2343	0.026*
C16	0.20377 (14)	-0.19206 (13)	0.08477 (14)	0.0188 (3)
C17	0.23511 (15)	-0.16333 (14)	0.00239 (15)	0.0217 (3)
H17	0.2099	-0.2079	-0.0599	0.026*
C18	0.30281 (15)	-0.07036 (15)	0.01021 (14)	0.0218 (3)
H18	0.3234	-0.0514	-0.0467	0.026*
C19	-0.03091 (15)	-0.24833 (16)	0.01235 (15)	0.0258 (4)
C20	-0.05345 (17)	-0.19664 (19)	-0.09463 (17)	0.0337 (4)
H20A	-0.1330	-0.1700	-0.1272	0.051*
H20B	-0.0411	-0.2527	-0.1407	0.051*
H20C	-0.0008	-0.1339	-0.0851	0.051*
C21	-0.04654 (19)	-0.1617 (2)	0.0863 (2)	0.0435 (6)
H21A	0.0031	-0.0974	0.0917	0.065*
H21B	-0.0259	-0.1946	0.1567	0.065*
H21C	-0.1269	-0.1374	0.0579	0.065*
C22	-0.1104 (2)	-0.3474 (2)	-0.0007 (2)	0.0454 (6)
H22A	-0.1904	-0.3218	-0.0299	0.068*
H22B	-0.0920	-0.3824	0.0684	0.068*
H22C	-0.0999	-0.4018	-0.0491	0.068*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Il	0.01979 (7)	0.02162 (7)	0.02101 (7)	-0.00596 (4)	0.00437 (5)	-0.00162 (4)
S2	0.0270 (2)	0.01496 (19)	0.0329 (2)	-0.00596 (16)	0.01011 (19)	0.00387 (16)
C1	0.0149 (7)	0.0151 (7)	0.0203 (8)	-0.0020 (6)	0.0054 (6)	-0.0022 (6)
C2	0.0170 (7)	0.0161 (7)	0.0202 (8)	0.0007 (6)	0.0070 (6)	0.0020 (6)
C3	0.0165 (7)	0.0136 (7)	0.0214 (8)	-0.0004 (6)	0.0071 (6)	0.0008 (6)
C4	0.0162 (7)	0.0121 (7)	0.0212 (8)	0.0000 (5)	0.0081 (6)	-0.0007 (6)
C5	0.0202 (7)	0.0167 (7)	0.0229 (8)	-0.0031 (6)	0.0073 (7)	-0.0020 (6)
C6	0.0238 (8)	0.0243 (9)	0.0183 (8)	-0.0017 (7)	0.0040 (7)	-0.0010 (6)
C7	0.0294 (9)	0.0243 (8)	0.0176 (8)	0.0005 (7)	0.0094 (7)	0.0021 (6)
C8	0.0269 (9)	0.0213 (8)	0.0295 (10)	-0.0012 (7)	0.0146 (8)	0.0059 (7)
C9	0.0208 (8)	0.0174 (8)	0.0251 (9)	-0.0047 (6)	0.0106 (7)	-0.0014 (6)
C10	0.0157 (7)	0.0135 (7)	0.0193 (7)	-0.0006 (5)	0.0076 (6)	-0.0016 (6)
C11	0.0178 (7)	0.0171 (7)	0.0225 (8)	0.0009 (6)	0.0064 (6)	0.0035 (6)
C12	0.0168 (7)	0.0203 (8)	0.0244 (8)	0.0003 (6)	0.0057 (6)	0.0055 (6)
C13	0.0141 (7)	0.0153 (7)	0.0239 (8)	-0.0005 (6)	0.0060 (6)	0.0037 (6)
C14	0.0229 (8)	0.0243 (9)	0.0195 (8)	-0.0065 (6)	0.0063 (7)	-0.0008 (6)
C15	0.0231 (8)	0.0238 (8)	0.0190 (8)	-0.0051 (6)	0.0087 (7)	0.0018 (6)
C16	0.0181 (7)	0.0140 (7)	0.0240 (8)	-0.0013 (6)	0.0080 (6)	0.0026 (6)
C17	0.0248 (8)	0.0175 (8)	0.0269 (9)	-0.0028 (6)	0.0147 (7)	-0.0041 (6)

C18	0.0240 (8)	0.0207 (8)	0.0261 (9)	-0.0010 (6)	0.0157 (7)	0.0022 (7)
C19	0.0210 (8)	0.0274 (9)	0.0279 (9)	-0.0097 (7)	0.0087 (7)	0.0018 (7)
C20	0.0284 (9)	0.0367 (11)	0.0314 (10)	-0.0069 (8)	0.0072 (8)	0.0088 (8)
C21	0.0270 (10)	0.0586 (15)	0.0486 (14)	-0.0018 (10)	0.0191 (10)	-0.0132 (11)
C22	0.0338 (11)	0.0454 (13)	0.0482 (14)	-0.0219 (10)	0.0073 (10)	0.0110 (11)

*Geometric parameters (Å, °)*

I1—C1	2.0651 (15)	C13—C18	1.398 (3)
S2—C16	1.7712 (16)	C13—C14	1.402 (2)
S2—C19	1.8526 (19)	C14—C15	1.386 (2)
C1—C2	1.394 (2)	C14—H14	0.9500
C1—C10	1.409 (2)	C15—C16	1.396 (2)
C2—C3	1.410 (2)	C15—H15	0.9500
C2—H2	0.9500	C16—C17	1.391 (2)
C3—C4	1.414 (2)	C17—C18	1.386 (2)
C3—C11	1.422 (2)	C17—H17	0.9500
C4—C5	1.383 (2)	C18—H18	0.9500
C4—C10	1.479 (2)	C19—C20	1.517 (3)
C5—C6	1.387 (2)	C19—C22	1.523 (3)
C5—H5	0.9500	C19—C21	1.523 (3)
C6—C7	1.389 (3)	C20—H20A	0.9800
C6—H6	0.9500	C20—H20B	0.9800
C7—C8	1.388 (3)	C20—H20C	0.9800
C7—H7	0.9500	C21—H21A	0.9800
C8—C9	1.390 (3)	C21—H21B	0.9800
C8—H8	0.9500	C21—H21C	0.9800
C9—C10	1.385 (2)	C22—H22A	0.9800
C9—H9	0.9500	C22—H22B	0.9800
C11—C12	1.200 (2)	C22—H22C	0.9800
C12—C13	1.434 (2)		
C16—S2—C19	102.19 (8)	C13—C14—H14	120.1
C2—C1—C10	109.87 (14)	C14—C15—C16	120.96 (16)
C2—C1—H1	124.97 (12)	C14—C15—H15	119.5
C10—C1—H1	125.15 (12)	C16—C15—H15	119.5
C1—C2—C3	108.81 (14)	C17—C16—C15	119.00 (15)
C1—C2—H2	125.6	C17—C16—S2	120.11 (13)
C3—C2—H2	125.6	C15—C16—S2	120.89 (13)
C2—C3—C4	108.39 (14)	C18—C17—C16	120.67 (16)
C2—C3—C11	127.23 (15)	C18—C17—H17	119.7
C4—C3—C11	124.35 (15)	C16—C17—H17	119.7
C5—C4—C3	125.25 (15)	C17—C18—C13	120.22 (16)
C5—C4—C10	127.69 (15)	C17—C18—H18	119.9
C3—C4—C10	107.06 (14)	C13—C18—H18	119.9
C4—C5—C6	128.67 (16)	C20—C19—C22	110.38 (17)
C4—C5—H5	115.7	C20—C19—C21	110.34 (19)
C6—C5—H5	115.7	C22—C19—C21	110.54 (18)

C5—C6—C7	128.83 (17)	C20—C19—S2	111.05 (13)
C5—C6—H6	115.6	C22—C19—S2	103.93 (14)
C7—C6—H6	115.6	C21—C19—S2	110.44 (14)
C8—C7—C6	129.86 (17)	C19—C20—H20A	109.5
C8—C7—H7	115.1	C19—C20—H20B	109.5
C6—C7—H7	115.1	H20A—C20—H20B	109.5
C7—C8—C9	128.44 (17)	C19—C20—H20C	109.5
C7—C8—H8	115.8	H20A—C20—H20C	109.5
C9—C8—H8	115.8	H20B—C20—H20C	109.5
C10—C9—C8	128.97 (16)	C19—C21—H21A	109.5
C10—C9—H9	115.5	C19—C21—H21B	109.5
C8—C9—H9	115.5	H21A—C21—H21B	109.5
C9—C10—C1	126.65 (15)	C19—C21—H21C	109.5
C9—C10—C4	127.49 (15)	H21A—C21—H21C	109.5
C1—C10—C4	105.87 (13)	H21B—C21—H21C	109.5
C12—C11—C3	176.91 (19)	C19—C22—H22A	109.5
C11—C12—C13	175.55 (19)	C19—C22—H22B	109.5
C18—C13—C14	119.41 (15)	H22A—C22—H22B	109.5
C18—C13—C12	119.05 (16)	C19—C22—H22C	109.5
C14—C13—C12	121.53 (16)	H22A—C22—H22C	109.5
C15—C14—C13	119.71 (16)	H22B—C22—H22C	109.5
C15—C14—H14	120.1		
C10—C1—C2—C3	0.28 (19)	C5—C4—C10—C9	1.0 (3)
I1—C1—C2—C3	-179.64 (11)	C3—C4—C10—C9	-179.53 (16)
C1—C2—C3—C4	-0.09 (18)	C5—C4—C10—C1	-179.21 (16)
C1—C2—C3—C11	177.91 (16)	C3—C4—C10—C1	0.28 (17)
C2—C3—C4—C5	179.39 (15)	C18—C13—C14—C15	-0.8 (3)
C11—C3—C4—C5	1.3 (3)	C12—C13—C14—C15	-179.58 (16)
C2—C3—C4—C10	-0.12 (17)	C13—C14—C15—C16	-0.6 (3)
C11—C3—C4—C10	-178.19 (15)	C14—C15—C16—C17	1.8 (3)
C3—C4—C5—C6	179.33 (17)	C14—C15—C16—S2	-178.67 (14)
C10—C4—C5—C6	-1.3 (3)	C19—S2—C16—C17	-89.74 (15)
C4—C5—C6—C7	-0.8 (3)	C19—S2—C16—C15	90.77 (15)
C5—C6—C7—C8	2.6 (3)	C15—C16—C17—C18	-1.6 (3)
C6—C7—C8—C9	-1.7 (3)	S2—C16—C17—C18	178.85 (14)
C7—C8—C9—C10	-0.4 (3)	C16—C17—C18—C13	0.2 (3)
C8—C9—C10—C1	-179.23 (18)	C14—C13—C18—C17	1.0 (3)
C8—C9—C10—C4	0.5 (3)	C12—C13—C18—C17	179.80 (16)
C2—C1—C10—C9	179.47 (16)	C16—S2—C19—C20	60.73 (15)
I1—C1—C10—C9	-0.6 (2)	C16—S2—C19—C22	179.40 (14)
C2—C1—C10—C4	-0.35 (18)	C16—S2—C19—C21	-62.02 (16)
I1—C1—C10—C4	179.57 (11)		



*Hydrogen-bond geometry (Å, °)*

Cg1 is the centroid of the C1–C4/C10 ring.

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C17—H17 $\cdots$ Cg1 <sup>i</sup>	0.95	2.95	3.471 (2)	116

Symmetry code: (i)  $-x+1, -y, -z$ .