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Crystal structure and Hirshfeld surface analysis of *N*-(5-iodo-4-phenylthiazol-2-yl)acetamide

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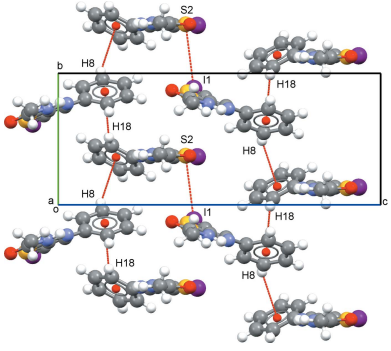
Two crystallographically independent molecules (*A* and *B*) are present in the asymmetric unit of the title compound, $C_{11}H_9IN_2OS$, which differ mainly in the dihedral angle between the phenyl and thiazole rings [38.94 (16) and 32.12 (15)°, respectively]. In the crystal, the molecules form ···*A*···*B*···*A*···*B*··· chains along the [001] and [010] directions through moderate N—H···O hydrogen bonds and C—H···π interactions, respectively. The overall three-dimensional network is formed by I···I and I···S interactions. Hirshfeld surface analysis indicates that the most important contributions to the crystal packing are from H···C/C···H (26.2%), H···H (20.9%), H···I/I···H (19.4%) and H···O/O···H (6.8%) interactions.

1. Chemical context

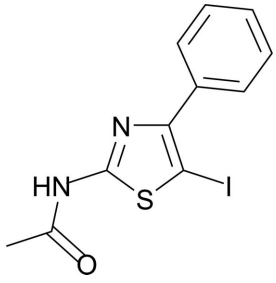
The 1,3-thiazole ring is a structural motif frequently found in the pharmaceutical field in antibacterial (Alam *et al.*, 2014), antifungal (Yu *et al.*, 2007) and antiviral (Liu *et al.*, 2011) agents among others. In the chemotherapy of protozoal diseases, 5-bromo-2-aminothiazole derivatives have been investigated as privileged structures in biological tests against intestinal parasites such as *Giardia* (Mocelo-Castell *et al.*, 2015). Halo-1,3-thiazole derivatives have proven to be suitable substrates in oxidative addition reactions in the presence of palladium (Wang *et al.*, 2015; Hämmerle *et al.*, 2010). The presence of halogens in the core of thiazole derivatives opens the door to using them as suitable substrates for coupling reactions and to expand the therapeutic potential of a compound by improving the pharmaceutical properties. Transition-metal-catalysed reactions constitute one of the most important and attractive research areas in academia, as well as in the pharmaceutical and fine chemical industries (Zhao *et al.*, 2017; Jana *et al.*, 2011). Cross-coupling reactions usually require, in addition to a transition metal, that the electrophilic coupling partner possesses leaving groups such as Br^- or I^- among others. The development of suitable halo-1,3-thiazole substrates for cross-coupling reactions allows us to report the crystal structure and the Hirshfeld surface analysis of *N*-(5-iodo-4-phenylthiazol-2-yl)acetamide.



2. Structural commentary

The title 2-acetoamidothiazole derivative crystallizes in the monoclinic space group $P2_1/c$ with two crystallographically

independent molecules in the asymmetric unit (Fig. 1). The principal difference between these molecules is the dihedral angle between the phenyl and thiazole rings. In molecule *A*, the thiazole ring (S1/N2/C3–C5) makes a dihedral angle of 38.94 (16)° with the adjacent phenyl ring (C6–C11) while for molecule *B* the dihedral angle between the S2/N4/C14–C16 and C17–C22 rings is 32.12 (15)°. Unlike the related compound 2-acetamido-4-*p*-tolyl-1,3-thiazole (Lynch *et al.*, 2004) in which the molecule is essentially flat, the presence of the iodine atom at C5 or C16 of the title compound induces rotation of the phenyl group attached to the thiazole ring, as also observed in some bromine-substituted phenylthiazole compounds (see the *Database survey*).



3. Supramolecular features

In the crystal, molecules are linked by N1—H1···O2 and N3—H3···O1 moderate hydrogen bonds *via* a *C*(4) synthon (Table 1, Fig. 2), forming chains along [001] in an ···*A*···*B*···*A*···*B*··· fashion. In the same way, the phenyl rings of molecules *A* and *B* interact through C—H···π contacts along [010] and the resulting chains are further connected through I1···S2($1-x$, $-y$, $1-z$) contacts [3.7758 (9) Å] (Fig. 3). Additionally, adjacent *B* molecules are linked by I2···I2($7-x$, $1-y$, $1-z$) contacts of type I [$\theta_1 = \theta_2$ = 146.91 (8)°] with a length of 3.8547 (5) Å.

4. Hirshfeld surface analysis and two-dimensional fingerprints plots

A Hirshfeld surface analysis was carried out using *Crystal Explorer17.5* (Turner *et al.*, 2017) in order to acquire a better understanding of the nature of the intermolecular interactions in the title compound. The Hirshfeld surface was generated using a standard (high) surface resolution with the three-

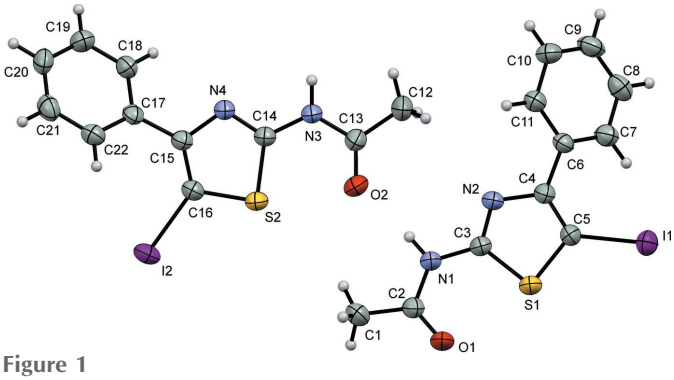


Figure 1
Molecular structure of the two crystallographically independent molecules in the asymmetric unit of the title compound, with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radius.

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*2 and *Cg*4 are the centroids of the C6–C11 and C17–C22 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.89 (3)	2.03 (3)	2.914 (3)	175 (3)
N3—H3···O1^{i}	0.89 (2)	2.03 (2)	2.902 (3)	167 (2)
C8—H8···*Cg*4^{ii}	0.93	2.94	3.655 (4)	134
C18—H18···*Cg*2^{ii}	0.93	2.82	3.594 (4)	141

Symmetry codes: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$.

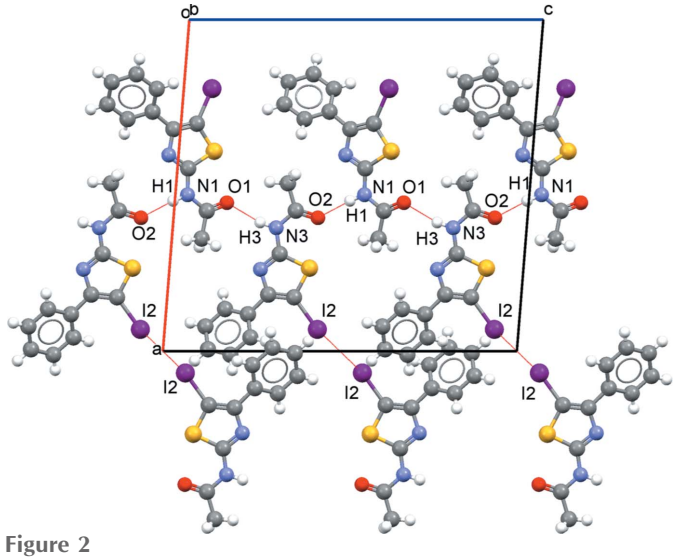


Figure 2
Part of the crystal structure of the title compound, showing the formation of hydrogen bonds and I···I contacts (red dashed lines) in the *ac* plane.

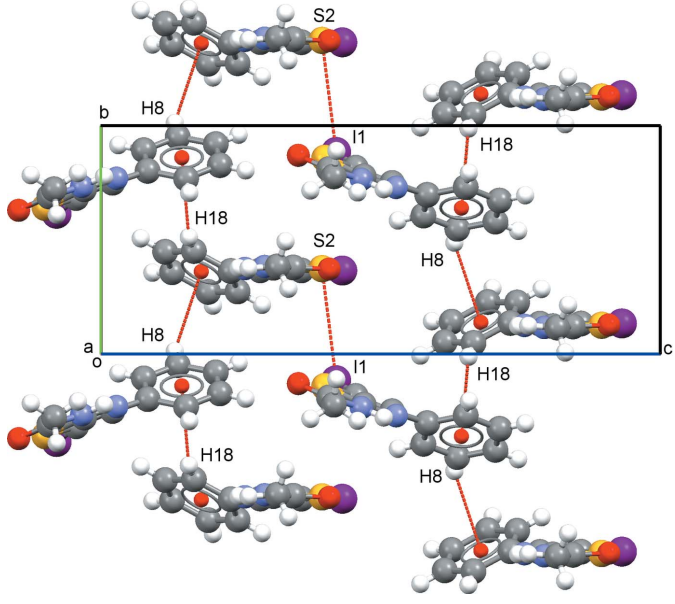


Figure 3
Packing viewed along the *a*-axis direction showing C–H···π and I···S interactions as red dashed lines.

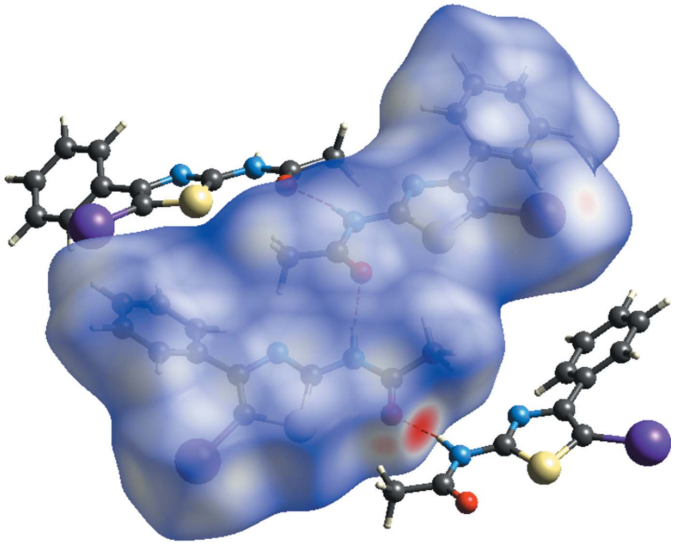


Figure 4
The three-dimensional Hirshfeld surface of the title compound mapped over d_{norm}, showing the N—H···O hydrogen bonds.

dimensional d_{norm} surface mapped over a fixed color scale of −0.5372 (red) to 1.3937 (blue) a.u. (Fig. 4). The intense red spots on the surface are due to the N—H···O hydrogen bonds, resulting from the interaction of the amide group of the 2-acetoamidothiazole derivative. The overall two-dimensional fingerprint plot for the title compound is shown in Fig. 5*a*, and those delineated into H···C/C···H, H···H, H···I/I···H, H···O/O···H and I···S/S···I contacts are shown in Fig. 5*b*–*f*. The major contribution to the crystal packing is from H···C/C···H interactions (26.2%). The pair of characteristic wings in this fingerprint plot corresponds to the C—H···π interactions between the phenyl groups (Fig. 5*b*). The H···H and H···I/I···H contacts (Fig. 5*c* and 5*d*) make similar contributions to the total Hirshfeld surface of 20.9 and 19.4%, respectively. The reciprocal H···O/O···H interactions (6.8%) are seen as sharp symmetrical spikes with tips at $d_e + d_i$ ∼1.9 Å and arising from the N—H···O hydrogen bond (Fig. 5*e*). Intermolecular I···S/S···I (Fig. 5*f*) and I···I interactions make smaller contributions to the Hirshfeld surface (2.2 and 1.1%, respectively).

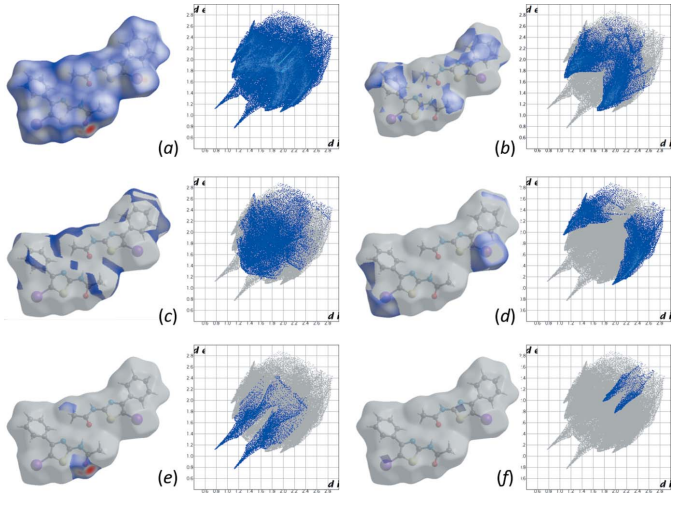


Figure 5
Two-dimensional fingerprint plots for the (*a*) all, (*b*) H···C/C···H (26.2%), (*c*) H···H (20.9%), (*d*) H···I/I···H (19.4%), (*e*) H···O/O···H (6.8%) and (*f*) I···S/S···I (2.2%) contacts in the title compound.

5. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.39, update of February 2018; Groom *et al.*, 2016) for a 1,3 thiazole ring with a benzene ring and a halogen as substituents in positions 4 and 5, respectively, gave four entries for the organobromine compounds *N*-[5-bromo-4-(4-methylphenyl)1,3-thiazol-2-yl]-4-chlorobutanamide (CCDC 1443533; Ghabbour *et al.*, 2016), 5,5′-dibromo-4,4′-bis(pentafluorophenyl)-2,2′-bi-1,3-thiazole (CCDC 889644; Siram *et al.*, 2013), 1-(5-bromo-4-phenyl-1,3-thiazol-2-yl)pyrrolidin-2-one (CCDC 886962; Ghabbour, Kadi, *et al.*, 2012) and 5-bromo-4-(3,4-dimethoxyphenyl)-1,3-thiazol-2-amine (CCDC 886876; Ghabbour, Chia, *et al.*, 2012). The dihedral angle between the thiazole and benzene rings in these compounds are in the range 36.69 (11) to 60.83 (3)°, with exception of *N*-(5-bromo-4-(4-methylphenyl)-1,3-thiazol-2-yl)-4-chlorobutanamide. In this compound the dihedral angle is smaller [8.8 (3)°] as a result of an intramolecular C—H···Br hydrogen bond. In the crystals of these compounds, only 5,5′-dibromo-4,4′-bis(pentafluorophenyl)-2,2′-bi-1,3-thiazole exhibits a type II halogen–halogen interaction with a Br···Br distance of 3.6777 (3) Å and angles of 68.88 (5) and 174.77 (5)°.

6. Synthesis and crystallization

A mixture of *N*-(4-phenylthiazol-2-yl) acetamide (0.5 mmol, 109 mg, 1 eq) and iodine (1 mmol, 127 mg, 2 eq) was placed in an open vessel containing a Teflon-coated stir bar. The mixture was dissolved in 3 mL of ethanol and the vessel was placed in the microwave cavity (CEM, Discover) and subjected to MW irradiation (150 W) for 60 min, at 363 K and a pressure of 2 psi. The reaction mixture was then cooled at room temperature and 5 mL of NH_4OH were added. The obtained mixture was dissolved in ethyl acetate (50 mL) and washed with brine (3×). The organic layer was separated, dehydrated with Na_2SO_4, and evaporated in vacuo until dryness. The product was purified by flash column chromatography (silica gel, 2–25 µm) with a mixture of petrol–dichloromethane–acetone (5:3:2). The title compound was obtained as pale-yellow needles in 30% yield (52.2 mg, 0.15 mmol). A diluted solution of the compound was prepared in hexane and kept on a dry and dark place at room temperature. Crystals were obtained after one week of slow evaporation. Spectroscopic data: ^{1}H NMR (400 MHz, $CDCl_3$): 11.37 (*s*, 1H), 7.80 (*m*, 2H), 7.43 (*m*, 3H), 1.62 (*s*, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): 168.8 (*s*), 163.6 (*s*), 151.4 (*s*), 134.5 (*s*), 129.0 (*d*), 128.9 (*d*), 128.7 (*d*), 62.4 (*s*) 21.9 (*c*).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Hydrogen atoms bonded to C atoms were positioned geometrically and refined using a riding model: C—H = 0.95–1.00 Å with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C-methyl).

Acknowledgements

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Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_9IN_2OS$
M_r	344.16
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	298
a, b, c (Å)	17.4130 (6), 7.5325 (3), 18.5443 (6)
β (°)	94.567 (1)
V (Å^3)	2424.61 (15)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	2.79
Crystal size (mm)	0.30 × 0.27 × 0.09
Data collection	
Diffractometer	Bruker D8 Venture κ-geometry diffractometer 208039-01
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.595, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	21512, 4448, 3987
R_{int}	0.020
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.604
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.029, 0.073, 1.12
No. of reflections	4448
No. of parameters	297
No. of restraints	2
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.97, −0.49

Computer programs: *APEX3* and *SAINT* (Bruker, 2014), *SHELXS2014* (Bruker, 2014), *SHELXL2014* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2006), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

References

Alam, M. S., Ahmed, J. U. & Lee, D. (2014). *Chem. Pharm. Bull.* **62**, 1259–1268.
Bruker (2014). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Ghabbour, H. A. & Al-Omar, M. A. (2016). *Z. Kristallogr. New Cryst. Struct.* **231**, 859–860.
Ghabbour, H. A., Chia, T. S. & Fun, H.-K. (2012). *Acta Cryst.* E**68**, o1631–o1632.
Ghabbour, H. A., Kadi, A. A., El-Subbagh, H. I., Chia, T. S. & Fun, H.-K. (2012). *Acta Cryst.* E**68**, o1738–o1739.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Hämmerle, J., Schnürch, M., Iqbal, N., Mihovilovic, M. D. & Stanetty, P. (2010). *Tetrahedron*, **66**, 8051–8059.
Jana, R., Pathak, T. P. & Sigman, M. S. (2011). *Chem. Rev.* **111**, 1417–1492.
Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.
Liu, Y., Zhang, L., Gong, J., Fang, H., Liu, A., Du, G. & Xu, W. (2011). *J. Enzyme Inhib. Med. Chem.* **26**, 506–513.
Lynch, D. E. & McClenaghan, I. (2004). *Acta Cryst.* C**60**, o815–o817.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Mocelo-Castell, R., Villanueva-Novelo, C., Cáceres-Castillo, D., Carballo, R. M., Quijano-Quiñones, R. F., Quesadas-Rojas, M., Cantillo-Ciau, Z., Cedillo-Rivera, R., Moo-Puc, R. E., Moujir, L. M. & Mena-Rejón, G. J. (2015). *Open Chem.* **13**, 1127–1136.
Sheldrick, G. M. (2015). *Acta Cryst.* A**71**, 3–8.
Siram, R. B. K., Karothu, D. P., Guru Row, T. N. & Patil, S. (2013). *Cryst. Growth Des.* **13**, 1045–1049.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Spackman, P. R., Jayatilaka, D. & Spackman, M. A. (2017). *Crystal. Explorer17*. The University of Western Australia.
Wang, F., Yang, Z., Liu, Y., Ma, L., Wu, Y., He, L., Shao, M., Yu, K., Wu, W., Pu, Y., Nie, C. & Chen, L. (2015). *Bioorg. Med. Chem.* **23**, 3337–3350.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Yu, H., Shao, L. & Fang, J. (2007). *J. Organomet. Chem.* **692**, 991–996.
Zhao, K., Shen, L., Shen, Z. L. & Loh, T. P. (2017). *Chem. Soc. Rev.* **46**, 586–602.

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Computing details

Data collection: *APEX3* (Bruker, 2014); cell refinement: *APEX3* (Bruker, 2014); data reduction: *SAINT* (Bruker, 2014); program(s) used to solve structure: *SHELXS2014* (Bruker, 2014); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

N-(5-Iodo-4-phenylthiazol-2-yl)acetamide

Crystal data

$C_{11}H_9IN_2OS$
M_r = 344.16
Monoclinic, $P2_1/c$
a = 17.4130 (6) Å
b = 7.5325 (3) Å
c = 18.5443 (6) Å
β = 94.567 (1)°
V = 2424.61 (15) Å^3
Z = 8
$F(000)$ = 1328
D_x = 1.886 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9666 reflections
θ = 2.5–25.4°
μ = 2.79 mm^{-1}
T = 298 K
Prism, colourless
0.30 × 0.27 × 0.09 mm

Data collection

Bruker D8 Venture κ-geometry diffractometer 208039-01
Radiation source: micro-focus X-ray source
Detector resolution: 52.0833 pixels mm^{-1}
ω–scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.595, T_{max} = 0.745
21512 measured reflections
4448 independent reflections
3987 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 25.4°, θ_{min} = 2.2°
h = −15→21
k = −9→9
l = −21→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.073
S = 1.12
4448 reflections
297 parameters
2 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0357P)^2 + 2.3197P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.97 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.20804 (2)	0.11466 (3)	0.57958 (2)	0.05010 (9)
I2	0.93426 (2)	0.36580 (3)	0.43149 (2)	0.05145 (9)
S1	0.39704 (5)	0.14260 (11)	0.59979 (4)	0.04052 (18)
S2	0.74959 (5)	0.37404 (11)	0.39534 (4)	0.03938 (18)
O1	0.55222 (13)	0.1295 (3)	0.64721 (12)	0.0505 (6)
O2	0.59773 (14)	0.3722 (3)	0.40790 (12)	0.0505 (6)
N1	0.52822 (14)	0.2346 (4)	0.53438 (13)	0.0413 (6)
H1	0.5467 (19)	0.278 (4)	0.4945 (12)	0.050*
N2	0.40840 (14)	0.2698 (4)	0.47229 (13)	0.0388 (6)
N3	0.62765 (15)	0.3834 (4)	0.29243 (13)	0.0408 (6)
H3	0.612 (2)	0.377 (4)	0.2456 (7)	0.049*
N4	0.75315 (14)	0.3745 (3)	0.25708 (13)	0.0372 (6)
C1	0.66028 (18)	0.2219 (5)	0.58392 (19)	0.0510 (8)
H1A	0.6829	0.2837	0.6256	0.076*
H1B	0.6656	0.2923	0.5414	0.076*
H1C	0.6860	0.1103	0.5792	0.076*
C2	0.57687 (17)	0.1901 (4)	0.59246 (16)	0.0393 (7)
C3	0.44875 (17)	0.2214 (4)	0.53026 (15)	0.0369 (6)
C4	0.33023 (16)	0.2505 (4)	0.47953 (15)	0.0350 (6)
C5	0.31384 (16)	0.1834 (4)	0.54453 (16)	0.0371 (6)
C6	0.27744 (17)	0.3026 (4)	0.41671 (15)	0.0365 (6)
C7	0.20880 (19)	0.3934 (4)	0.42351 (18)	0.0433 (7)
H7	0.1925	0.4152	0.4692	0.052*
C8	0.16480 (19)	0.4514 (5)	0.3630 (2)	0.0509 (8)
H8	0.1190	0.5121	0.3681	0.061*
C9	0.1884 (2)	0.4198 (5)	0.29482 (19)	0.0522 (9)
H9	0.1591	0.4607	0.2541	0.063*
C10	0.2554 (2)	0.3275 (5)	0.28749 (18)	0.0501 (8)
H10	0.2710	0.3046	0.2416	0.060*
C11	0.29986 (18)	0.2687 (4)	0.34786 (16)	0.0417 (7)
H11	0.3450	0.2060	0.3423	0.050*
C12	0.4936 (2)	0.3841 (7)	0.3156 (2)	0.0688 (12)
H12A	0.4668	0.2859	0.3350	0.103*
H12B	0.4900	0.3761	0.2638	0.103*
H12C	0.4707	0.4934	0.3298	0.103*
C13	0.57608 (18)	0.3794 (4)	0.34378 (17)	0.0413 (7)
C14	0.70669 (17)	0.3778 (4)	0.30805 (15)	0.0354 (6)
C15	0.82943 (17)	0.3668 (4)	0.28480 (15)	0.0338 (6)
C16	0.83746 (17)	0.3663 (4)	0.35831 (16)	0.0363 (6)

C17	0.88862 (17)	0.3640 (4)	0.23203 (16)	0.0340 (6)
C18	0.87296 (17)	0.4513 (5)	0.16611 (16)	0.0418 (7)
H18	0.8267	0.5121	0.1570	0.050*
C19	0.92545 (19)	0.4480 (5)	0.11460 (18)	0.0494 (8)
H19	0.9145	0.5069	0.0709	0.059*
C20	0.99420 (19)	0.3583 (5)	0.1270 (2)	0.0495 (8)
H20	1.0293	0.3562	0.0918	0.059*
C21	1.01068 (18)	0.2721 (5)	0.19149 (19)	0.0477 (8)
H21	1.0572	0.2119	0.2000	0.057*
C22	0.95846 (18)	0.2741 (4)	0.24411 (17)	0.0404 (7)
H22	0.9701	0.2152	0.2877	0.049*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.03894 (13)	0.06520 (16)	0.04648 (14)	−0.01106 (9)	0.00551 (10)	0.00738 (10)
I2	0.04289 (14)	0.07225 (18)	0.03680 (13)	0.00079 (10)	−0.01192 (9)	−0.00143 (10)
S1	0.0370 (4)	0.0575 (5)	0.0268 (4)	−0.0013 (3)	0.0002 (3)	0.0065 (3)
S2	0.0389 (4)	0.0537 (5)	0.0252 (4)	−0.0033 (3)	0.0004 (3)	−0.0013 (3)
O1	0.0390 (12)	0.0830 (18)	0.0287 (11)	−0.0006 (11)	−0.0023 (9)	0.0113 (10)
O2	0.0479 (13)	0.0750 (17)	0.0293 (12)	−0.0054 (11)	0.0081 (10)	−0.0006 (10)
N1	0.0318 (13)	0.0654 (18)	0.0266 (13)	−0.0003 (12)	0.0010 (10)	0.0076 (12)
N2	0.0324 (13)	0.0572 (16)	0.0261 (12)	0.0005 (11)	−0.0020 (10)	0.0031 (11)
N3	0.0316 (13)	0.0656 (18)	0.0249 (12)	−0.0035 (11)	0.0015 (10)	−0.0014 (11)
N4	0.0309 (13)	0.0530 (16)	0.0275 (12)	−0.0010 (10)	0.0014 (10)	−0.0009 (10)
C1	0.0359 (17)	0.071 (2)	0.0444 (18)	0.0009 (16)	−0.0046 (14)	0.0087 (17)
C2	0.0363 (16)	0.0507 (18)	0.0307 (15)	0.0033 (14)	0.0006 (12)	−0.0016 (13)
C3	0.0341 (15)	0.0496 (18)	0.0269 (14)	−0.0006 (13)	0.0024 (12)	0.0015 (12)
C4	0.0315 (15)	0.0432 (16)	0.0300 (15)	0.0004 (12)	0.0004 (11)	−0.0019 (12)
C5	0.0308 (15)	0.0481 (17)	0.0323 (15)	−0.0034 (13)	0.0013 (12)	0.0022 (13)
C6	0.0325 (15)	0.0445 (17)	0.0313 (15)	−0.0051 (13)	−0.0044 (12)	0.0016 (13)
C7	0.0426 (18)	0.0506 (19)	0.0360 (16)	0.0042 (14)	−0.0015 (14)	0.0006 (13)
C8	0.0389 (18)	0.054 (2)	0.058 (2)	0.0072 (15)	−0.0064 (15)	0.0037 (17)
C9	0.050 (2)	0.063 (2)	0.0411 (19)	−0.0014 (17)	−0.0146 (15)	0.0079 (16)
C10	0.052 (2)	0.067 (2)	0.0300 (16)	−0.0039 (17)	−0.0016 (14)	0.0049 (15)
C11	0.0345 (16)	0.0541 (19)	0.0358 (16)	−0.0004 (14)	−0.0005 (13)	0.0053 (14)
C12	0.0346 (18)	0.129 (4)	0.044 (2)	−0.005 (2)	0.0073 (16)	−0.002 (2)
C13	0.0355 (16)	0.0538 (19)	0.0353 (17)	−0.0041 (13)	0.0059 (13)	−0.0032 (13)
C14	0.0340 (15)	0.0439 (17)	0.0279 (14)	−0.0019 (12)	0.0003 (12)	−0.0013 (12)
C15	0.0326 (15)	0.0380 (16)	0.0302 (15)	−0.0005 (12)	−0.0012 (12)	−0.0020 (11)
C16	0.0328 (15)	0.0438 (17)	0.0314 (15)	−0.0010 (12)	−0.0028 (12)	0.0004 (12)
C17	0.0306 (15)	0.0390 (16)	0.0316 (15)	−0.0027 (12)	−0.0017 (12)	−0.0064 (12)
C18	0.0328 (15)	0.0544 (19)	0.0376 (16)	0.0050 (14)	0.0001 (13)	0.0007 (14)
C19	0.0456 (19)	0.069 (2)	0.0339 (16)	0.0018 (17)	0.0043 (14)	0.0064 (16)
C20	0.0331 (17)	0.068 (2)	0.048 (2)	−0.0023 (15)	0.0115 (15)	−0.0061 (16)
C21	0.0327 (16)	0.056 (2)	0.055 (2)	0.0065 (14)	0.0008 (14)	−0.0093 (16)
C22	0.0375 (16)	0.0443 (18)	0.0382 (16)	0.0021 (13)	−0.0050 (13)	0.0007 (13)

Geometric parameters (Å, º)

I1—C5	2.068 (3)	C7—C8	1.378 (5)
I2—C16	2.078 (3)	C7—H7	0.9300
S1—C5	1.734 (3)	C8—C9	1.381 (5)
S1—C3	1.735 (3)	C8—H8	0.9300
S2—C16	1.727 (3)	C9—C10	1.374 (5)
S2—C14	1.728 (3)	C9—H9	0.9300
O1—C2	1.222 (4)	C10—C11	1.382 (4)
O2—C13	1.220 (4)	C10—H10	0.9300
N1—C2	1.358 (4)	C11—H11	0.9300
N1—C3	1.383 (4)	C12—C13	1.490 (5)
N1—H1	0.890 (10)	C12—H12A	0.9600
N2—C3	1.289 (4)	C12—H12B	0.9600
N2—C4	1.386 (4)	C12—H12C	0.9600
N3—C13	1.360 (4)	C15—C16	1.359 (4)
N3—C14	1.384 (4)	C15—C17	1.477 (4)
N3—H3	0.891 (10)	C17—C22	1.394 (4)
N4—C14	1.292 (4)	C17—C18	1.396 (4)
N4—C15	1.387 (4)	C18—C19	1.374 (4)
C1—C2	1.493 (4)	C18—H18	0.9300
C1—H1A	0.9600	C19—C20	1.378 (5)
C1—H1B	0.9600	C19—H19	0.9300
C1—H1C	0.9600	C20—C21	1.371 (5)
C4—C5	1.358 (4)	C20—H20	0.9300
C4—C6	1.478 (4)	C21—C22	1.386 (4)
C6—C11	1.388 (4)	C21—H21	0.9300
C6—C7	1.392 (4)	C22—H22	0.9300
C5—S1—C3	87.64 (14)	C9—C10—H10	119.8
C16—S2—C14	87.64 (14)	C11—C10—H10	119.8
C2—N1—C3	125.7 (3)	C10—C11—C6	120.4 (3)
C2—N1—H1	120 (2)	C10—C11—H11	119.8
C3—N1—H1	114 (2)	C6—C11—H11	119.8
C3—N2—C4	111.3 (2)	C13—C12—H12A	109.5
C13—N3—C14	123.6 (3)	C13—C12—H12B	109.5
C13—N3—H3	121 (2)	H12A—C12—H12B	109.5
C14—N3—H3	115 (2)	C13—C12—H12C	109.5
C14—N4—C15	111.5 (2)	H12A—C12—H12C	109.5
C2—C1—H1A	109.5	H12B—C12—H12C	109.5
C2—C1—H1B	109.5	O2—C13—N3	120.9 (3)
H1A—C1—H1B	109.5	O2—C13—C12	123.9 (3)
C2—C1—H1C	109.5	N3—C13—C12	115.2 (3)
H1A—C1—H1C	109.5	N4—C14—N3	121.2 (3)
H1B—C1—H1C	109.5	N4—C14—S2	115.8 (2)
O1—C2—N1	120.9 (3)	N3—C14—S2	123.0 (2)
O1—C2—C1	123.9 (3)	C16—C15—N4	113.0 (3)
N1—C2—C1	115.3 (3)	C16—C15—C17	130.0 (3)

N2—C3—N1	120.1 (3)	N4—C15—C17	117.0 (2)
N2—C3—S1	115.8 (2)	C15—C16—S2	112.0 (2)
N1—C3—S1	124.0 (2)	C15—C16—I2	131.9 (2)
C5—C4—N2	113.7 (3)	S2—C16—I2	116.02 (16)
C5—C4—C6	129.6 (3)	C22—C17—C18	118.5 (3)
N2—C4—C6	116.7 (2)	C22—C17—C15	123.1 (3)
C4—C5—S1	111.5 (2)	C18—C17—C15	118.4 (3)
C4—C5—I1	128.9 (2)	C19—C18—C17	120.4 (3)
S1—C5—I1	119.55 (15)	C19—C18—H18	119.8
C11—C6—C7	118.7 (3)	C17—C18—H18	119.8
C11—C6—C4	118.2 (3)	C18—C19—C20	120.7 (3)
C7—C6—C4	122.9 (3)	C18—C19—H19	119.7
C8—C7—C6	120.5 (3)	C20—C19—H19	119.7
C8—C7—H7	119.7	C21—C20—C19	119.8 (3)
C6—C7—H7	119.7	C21—C20—H20	120.1
C7—C8—C9	120.2 (3)	C19—C20—H20	120.1
C7—C8—H8	119.9	C20—C21—C22	120.4 (3)
C9—C8—H8	119.9	C20—C21—H21	119.8
C10—C9—C8	119.7 (3)	C22—C21—H21	119.8
C10—C9—H9	120.2	C21—C22—C17	120.3 (3)
C8—C9—H9	120.2	C21—C22—H22	119.8
C9—C10—C11	120.5 (3)	C17—C22—H22	119.8

C3—N1—C2—O1	1.3 (5)	C14—N3—C13—O2	0.7 (5)
C3—N1—C2—C1	−178.3 (3)	C14—N3—C13—C12	−179.2 (3)
C4—N2—C3—N1	−178.5 (3)	C15—N4—C14—N3	−179.3 (3)
C4—N2—C3—S1	1.2 (4)	C15—N4—C14—S2	0.6 (3)
C2—N1—C3—N2	179.2 (3)	C13—N3—C14—N4	177.1 (3)
C2—N1—C3—S1	−0.5 (5)	C13—N3—C14—S2	−2.7 (4)
C5—S1—C3—N2	−0.9 (3)	C16—S2—C14—N4	−0.3 (2)
C5—S1—C3—N1	178.8 (3)	C16—S2—C14—N3	179.5 (3)
C3—N2—C4—C5	−0.9 (4)	C14—N4—C15—C16	−0.6 (4)
C3—N2—C4—C6	179.6 (3)	C14—N4—C15—C17	−179.5 (3)
N2—C4—C5—S1	0.3 (4)	N4—C15—C16—S2	0.4 (3)
C6—C4—C5—S1	179.6 (3)	C17—C15—C16—S2	179.1 (2)
N2—C4—C5—I1	−175.6 (2)	N4—C15—C16—I2	−177.5 (2)
C6—C4—C5—I1	3.7 (5)	C17—C15—C16—I2	1.3 (5)
C3—S1—C5—C4	0.3 (3)	C14—S2—C16—C15	0.0 (2)
C3—S1—C5—I1	176.6 (2)	C14—S2—C16—I2	178.15 (17)
C5—C4—C6—C11	−142.8 (3)	C16—C15—C17—C22	33.8 (5)
N2—C4—C6—C11	36.5 (4)	N4—C15—C17—C22	−147.5 (3)
C5—C4—C6—C7	41.2 (5)	C16—C15—C17—C18	−148.0 (3)
N2—C4—C6—C7	−139.5 (3)	N4—C15—C17—C18	30.7 (4)
C11—C6—C7—C8	−1.3 (5)	C22—C17—C18—C19	0.0 (5)
C4—C6—C7—C8	174.7 (3)	C15—C17—C18—C19	−178.2 (3)
C6—C7—C8—C9	0.1 (5)	C17—C18—C19—C20	0.2 (5)
C7—C8—C9—C10	1.0 (6)	C18—C19—C20—C21	−0.3 (6)
C8—C9—C10—C11	−1.0 (6)	C19—C20—C21—C22	0.3 (5)

C9—C10—C11—C6	−0.2 (5)	C20—C21—C22—C17	0.0 (5)
C7—C6—C11—C10	1.3 (5)	C18—C17—C22—C21	−0.1 (4)
C4—C6—C11—C10	−174.8 (3)	C15—C17—C22—C21	178.1 (3)

Hydrogen-bond geometry (Å, º)

*Cg*2 and *Cg*4 are the centroids of the C6–C11 and C17–C22 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.89 (3)	2.03 (3)	2.914 (3)	175 (3)
N3—H3···O1^{i}	0.89 (2)	2.03 (2)	2.902 (3)	167 (2)
C8—H8···*Cg*4^{ii}	0.93	2.94	3.655 (4)	134
C18—H18···*Cg*2^{ii}	0.93	2.82	3.594 (4)	141

Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2; (ii) −*x*+1, *y*+1/2, −*z*+1/2.