Halogen, chalcogen, and hydrogen bonding in organoiodine cocrystals of heterocyclic thiones: imidazolidine-2-thione, 2-mercaptopbenzimidazole, 2-mercaptop-5-methylbenzimidazole, 2-mercaptopbenzoxazole, and 2-mercaptopbenzothiazole

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Through the combination of heterocyclic thiones with variation in the identity of the heterocyclic elements, namely, imidazolidine-2-thione, 2-mercaptopbenzimidazole, 2-mercaptop-5-methylbenzimidazole, 2-mercaptopbenzoxazole, and 2-mercaptopbenzothiazole with the common halogen-bond donors 1,2-, 1,3-, and 1,4-diiodotetrafluorobenzene, 1,3,5-trifluorotriiodobenzene, and tetraiodoethylene, a series of 18 new crystalline structures were characterized. In most cases, N—H/C1/C1/S hydrogen bonding was observed, with these interactions in imidazole-containing structures typically resulting in two-dimensional motifs (i.e. ribbons). Lacking the second N—H group, the thiazole and oxazole hydrogen bonding resulted in only dimeric pairs. C—I/C1/C1/I and C—I/C1/C1/S chalcogen bonding, as well as C=S/C1/I chalcogen bonding, served to consolidate the packing by linking the hydrogen-bonding ribbons or dimeric pairs.

1. Introduction

Halogen and chalcogen bonding, defined by IUPAC as ‘a net attractive interaction between an electrophilic region associated with . . .’ a halogen or chalcogen atom, respectively, ‘ . . . in a molecular entity and a nucleophilic region in another, or the same, molecular entity (Desiraju et al., 2013; Aakeroy et al., 2019);’ has drawn increasing attention in recent years (Parisi et al., 2011; Zhou et al., 2010; Ajani et al., 2015; Arman et al., 2008; Aakeroy et al., 2015; Metrangolo & Resnati, 2012; Cavallo et al., 2016; Metrangolo et al., 2005; Legon, 1998). Similar to hydrogen bonding, halogen bonding is strong, selective, and directional. Organic iodines are among the most commonly utilized halogen-bond donors (Corradi et al., 2000), largely due to their greater polarizability. When paired with halogen-bond acceptor molecules with a diversity of heteroatoms, the combined effects of halogen, chalcogen, and hydrogen bonding can be revealed. Imidazoles, thiazoles, and oxazoles are ideal systems to study in this regard.

Benzimidazole, and its derivatives, have been investigated for a diverse range of biological applications, including in the treatment of tuberculosis (Foks et al., 2006), as antimicrobial agents (Alasmary et al., 2015), and also as analgesic and anti-inflammatory compounds (Achar et al., 2010; Fletcher et al., 2006). These mercaptopbenzimidazoles, thiazoles, and oxazoles have also seen significant utilization as ligands in transition-metal complexes. Providing some insight into the role of heteroatoms in differing positions, of the 31 crystal structures

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containing 2-mercaptopbenzothiazole (MBZTH) and a transition metal currently deposited with the Cambridge Structural Database (CSD; Groom et al., 2016), all demonstrate metal coordination through the thione S atom and not the thiazole S atom. They range from simple species, such as (2-mercaptopbenzothiazole)(bis(triphenylphosphine)silver(I) iodide (Banti et al., 2014), to more complex copper and ruthenium complexes (Zhou et al., 2013a; Zafar et al., 2019). Similarly, the mercaptopbenzimidazole (or benzimidazolethione) derivatives present an interesting field of study for their potential intermolecular interactions in halogen-bonding systems (Fig. 1). In these systems, hydrogen, halogen, and chalcogen bonding are all viable intermolecular interactions, and structural studies of the cocrystals can be useful in determining which interactions are preferred as the organoiodine and the heterocyclic systems are varied.

Our group has recently been interested in the role of the S atom in I···S halogen- and chalcogen-bonding interactions as a crystal design tool, as well as their roles in the formation of deep eutectic solvents derived from halogen bonding (Peloquin et al., 2021a,b,c,d, 2022). Herein, we report the solid-state structures of 18 new cocrystals derived from the combination of the heterocyclic molecules imidazolidine-2-thione (IT), 2-mercaptopbenzimidazole (MBZIM), 2-mercapt-o-5-methylbenzimidazole (MMBZIM), 2-mercaptopbenzoxazole (MBZOX), and 2-mercaptopbenzothiazole (MBZTH) with the organic halogen-bond donors 1,2-diododifluorobenzene (1,2-F4DIB), 1,3-diododifluorobenzene (1,3-F4DIB), 1,4-tetrafluorobenzene (1,4-F4DIB), 1,3,5-trifluoro-2,4,6-triiodobenzene (1,3,5-F3I3B), and tetraiodoethylene (TIE). This diverse pool of substrates yielded structures with the crystal packing dominated by N···H···S hydrogen bonding, leading to thiouamide dimers, with longer-range packing motifs created through C—I···S and C—I···I halogen bonding, as well as the occasional C=S···I chalcogen bond.

2. Experimental

2.1. Materials and instrumentation

For single-crystal X-ray analysis, crystals were mounted on low background cryogenic loops using paratone oil. Data were collected using Mo Kα radiation (λ = 0.71073 Å) on a Bruker D8 Venture diffractometer with an Incoatec Iμs microfocus source and a Photon 2 detector.

2.2. Preparation of cocrystals

Cocrystals were synthesized using imidazolidine-2-thione (TCI Americas, 98%), 2-mercaptopbenzimidazole (Acros, 98%), 2-mercapto-5-methylbenzimidazole (Acros, 99%), 2-mercaptopbenzoxazole (Acros, 99%), 2-mercaptopbenzothiazole (Acros, 98%), 1,2-diododifluorobenzene (Synquest Laboratories, 99%), 1,3-diododifluorobenzene (Synquest Laboratories, 99%), 1,4-tetrafluorobenzene (Synquest Laboratories, 97%), 1,3,5-trifluoro-2,4,6-triiodobenzene (Synquest Laboratories, 99%), and tetraiodoethylene (Santa Cruz Biotechnologies, 98%). Solvents were obtained from Fisher Scientific. All materials were used as received without further purification. Crystals were formed by slow evaporation under ambient conditions (20–23 °C). Methanol was utilized for the majority of cocrystal preparations; however, if this was not successful, acetone or ethyl acetate was utilized.

2.2.1. 2(IT)-(1,3-F4DIB). Imidazolidine-2-thione (50 mg, 0.489 mmol) and 1,3-diododifluorobenzene (196 mg, 0.489 mmol) were weighed into a 20 ml glass vial. Methanol (10 ml) was added and the mixture was stirred until a clear solution was obtained. The solvent was allowed to evaporate slowly and colorless needle-like crystals of 2(IT)-(1,3-F4DIB) were obtained after 3 d.

2.2.2. (IT)-(1,3,5-F3I3B). Imidazolidine-2-thione (50 mg, 0.489 mmol) and 1,3,5-trifluoro-2,4,6-triiodobenzene (249 mg, 0.489 mmol) were weighed into a 20 ml glass vial. Methanol (10 ml) was added and the mixture was stirred until a clear solution was obtained. The solvent was allowed to evaporate slowly and colorless needle-like crystals of (IT)-(1,3,5-F3I3B) were obtained after 4 d.

2.2.3. 4(MMBZIM)-3(1,3-F4DIB). 2-Mercaptopbenzimidazole (34 mg, 0.227 mmol) and 1,3-diododifluorobenzene (49 mg, 0.122 mmol) were weighed into a 20 ml glass vial. Methanol (10 ml) was added and the mixture was stirred until a clear solution was obtained. The solvent was allowed to evaporate slowly and colorless needle-like crystals of 4(MMBZIM)-3(1,3-F4DIB) were obtained after 4 d.

Figure 1

Organoiodines and mercaptopimidazoles utilized in this study.
Table 1
Experimental details.

Experiments were carried out at 100 K with Mo Ka radiation using a Bruker D8 Venture Photon 2 diffractometer. Absorption was corrected for by multi-scan methods (SADABS; Bruker, 2017). H atoms were treated by a mixture of independent and constrained refinement, except for 3(MBZTH)-4(1,2-F4DIB), for which H-atom parameters were constrained.

| Crystal data | 2(T1)+1,3-F4DIB | 4(MBZIM)-3(1,3-F4DIB) | (MBZIM)-(1,3-F4I,1B) |
|--------------|-----------------|-----------------------|----------------------|
| Chemical formula | C6F4I2·2C6H4N2S | C6F4I2·2C6H4N2S | C6F4I2·2C6H4N2S 2(H2O) | C6F4I2·2C6H4N2S |
| M | 681.82 | 566.08 | 766.34 | 673.98 |
| Crystal system, space group | Orthorhombic, Pnma | Triclinic, P1 | Triclinic, P1 | Monoclinic, P21/c |
| a, b, c (Å) | 11.7547 (10), 8.3525 (7), 15.1077 (13) | 4.5504 (5), 13.2872 (14), 13.8064 (14) | 4.9088 (3), 11.4670 (8), 11.9686 (8) | 15.191 (2), 5.0074 (7), 22.715 (3) |
| α, β, γ (°) | 90, 90, 90 | 94.766 (4), 98.124 (4), 99.588 (4) | 106.644 (2), 98.058 (2), 92.811 (2) | 90, 97.460 (6), 90 |
| V (Å³) | 1483.3 (2) | 809.97 (15) | 636.27 (7) | 1713.3 (4) |
| Z | 4 | 2 | 1 | 4 |
| μ (mm⁻¹) | 8.52 | 4.05 | 2.69 | 5.62 |
| Crystal size (mm) | 0.30 × 0.14 × 0.11 | 0.19 × 0.07 × 0.04 | 0.31 × 0.11 × 0.08 | 0.26 × 0.04 × 0.04 |

Data collection

Tmax, Tmin | 0.639, 0.746 | 0.563, 0.746 | 0.668, 0.746 | 0.501, 0.746 |
No. of measured, independent and observed | 5376 | 3615 | 12297 | 4579 |
No. of reflections | 234 | 172 | 717 | 207 |
No. of parameters | 0 | 2 | 8 | 0 |
Δρ max, Δρ min (e Å⁻³) | 0.42, −0.37 | 0.57, −0.77 | 0.52, −0.75 | 0.49, −0.68 |

Refinement

R[F²] > 2σ(F²), wR(F²), S | 0.015, 0.032, 1.25 | 0.019, 0.040, 1.11 | 0.021, 0.041, 1.06 | 0.020, 0.044, 1.12 |
No. of reflections | 5376 | 3615 | 12297 | 4579 |
No. of parameters | 234 | 172 | 717 | 207 |
No. of restraints | 0 | 2 | 8 | 0 |
Δρ max, Δρ min (e Å⁻³) | 0.42, −0.37 | 0.57, −0.77 | 0.52, −0.75 | 0.49, −0.68 |

Bonding in organoiodine cocrystals

sigma-hole interactions
### Table 1 (continued)

| Crystal data                                                                 | (MBZOX)-(1,2-F$_4$DIB) | (MBZOX)-(1,3-F$_4$DIB) | (MBZOX)-(1,4-F$_4$DIB) | (MBZOX)-(1,3,5-F$_3$I$_3$B) |
|------------------------------------------------------------------------------|------------------------|------------------------|------------------------|-----------------------------|
| Chemical formula                                                             | 4C$_2$F$_7$I$_2$·3C$_7$H$_5$NS$_2$ | C$_6$F$_5$I$_2$·C$_7$H$_5$NS$_2$ | 4C$_6$F$_4$I$_2$·2C$_7$H$_5$NS$_2$ | C$_6$F$_4$I$_2$·2C$_7$H$_5$NS$_2$ |
| $M_r$                                                                        | 2109.16                | 569.10                | 1941.92                | 736.34 |
| Crystal system, space group                                                 | Triclinic, $P\bar{T}$ | Triclinic, $P\bar{T}$ | Monoclinic, $P2_1$    | Monoclinic, $P2_1/n$        |
| $a$, $b$, $c$ ($\text{Å}$)                                                  | 7.9410 (8), 14.8483 (15), 24.641 (3) | 7.2175 (4), 8.2675 (5), 14.4948 (9) | 4.5581 (3), 34.358 (2), 15.6075 (10) | 5.5057 (2), 15.6087 (7), 13.5194 (6) |
| $\alpha$, $\beta$, $\gamma$ ($^\circ$)                                    | 79.264 (4), 87.104 (4), 97.936 (2), 91.297 (2), 109.178 (2) | 97.936 (2), 91.297 (2), 109.178 (2) | 90.9, 109.178 (2), 90.9, 94.707 (2), 90.9, 109.178 (2) | 90.9, 109.178 (2), 90.9, 109.178 (2), 90.9, 109.178 (2) |
| $V$ ($\text{Å}^3$)                                                           | 2830.9 (5)             | 804.44 (8)             | 2436.0 (3)             | 1158.61 (8) |
| $Z$                                                                          | 2                     | 2                     | 2                     | 2 |
| $\mu$ (mm$^{-1}$)                                                           | 4.69                  | 4.20                  | 5.36                  | 3.12 |
| Crystal size (mm)                                                           | 0.30 × 0.13 × 0.04    | 0.33 × 0.27 × 0.06    | 0.18 × 0.12 × 0.04    | 0.17 × 0.09 × 0.04 |
| Data collection                                                              |                        |                       |                       |                       |
| $T_{	ext{min}}$, $T_{	ext{max}}$                                           | 0.570, 0.746           | 0.496, 0.746           | 0.568, 0.746           | 0.559, 0.746 |
| No. of measured, independent and observed $| I > 2\sigma(I) |$ reflections               | 78566, 12466, 11325   | 27899, 4701, 4391     | 56285, 12660, 11766   | 22270, 3402, 2811 |
| $R_{	ext{int}}$ (sin $\theta$)$_{\text{max}}$ (Å$^{-1}$)                 | 0.067                 | 0.036                 | 0.050                 | 0.049 |
| Refinement                                                                  |                        |                       |                       |                       |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$                                    | 0.067, 0.220, 1.06    | 0.018, 0.044, 1.09    | 0.026, 0.046, 1.09    | 0.027, 0.057, 1.15 |
| No. of reflections                                                          | 12466                 | 4701                  | 12660                 | 3402 |
| No. of parameters                                                           | 704                   | 203                   | 622                   | 149 |
| No. of restraints                                                           | 66                    | 0                     | 2                     | 0 |
| $\Delta \rho_{\text{max}}$, $\Delta \rho_{\text{min}}$ (e Å$^{-3}$)       | 2.61, −1.48           | 1.08, −1.11           | 1.01, −0.71           | 0.90, −0.79 |
| Absolute structure (°)                                                     | –                     | –                     | –                     | – |
| Absolute structure parameter                                               | –                     | –                     | –                     | – |

### sigma-hole interactions

The sigma-hole interactions play a crucial role in the stabilization of organoiodine cocrystals. Understanding these interactions is essential for predicting the physical and chemical properties of these materials. The analysis of sigma-hole interactions can provide insights into the bonding nature and structural stability of organoiodine cocrystals.
evaporate slowly and colorless needle-like crystals of (MBZIM)-3(1,3-F4DIB) were obtained after 4 d.

2.2.4. (MBZIM)-(1,4-F4DIB). 2-Mercaptobenzimidazole (19 mg, 0.126 mmol) and 1,4-diodotetrafluorobenzene (50 mg, 0.124 mmol) were weighed into a 20 ml glass vial. Methanol (10 ml) was added and the mixture was stirred until a clear solution was obtained. The solvent was allowed to evaporate slowly and colorless plate-like crystals of (MBZIM)-(1,4-F4DIB) were obtained after 3 d.

2.2.5. (MBZIM)-(TIE). 2-Mercaptobenzimidazole (30 mg, 0.200 mmol) and tetraiodoethylene (15 mg, 0.099 mmol) and 1,3,5-trifluoro-2,4,6-triiodobenzene (50 mg, 0.098 mmol) were weighed into a 20 ml glass vial. Methanol (10 ml) was added and the mixture was stirred until a clear solution was obtained. The solvent was allowed to evaporate slowly and colorless tabular crystals of (MBZIM)-(TIE) were obtained after 7 d.

2.2.6. (MBBZIM)-(1,2-F4DIB). 2-Mercapto-5-methylbenzimidazole (20 mg, 0.122 mmol) and 1,2-diodotetrafluorobenzene (48 mg, 0.119 mmol) were weighed into a 20 ml glass vial. Ethyl acetate (15 ml) was added and the mixture was stirred until a clear solution was obtained. The solvent was allowed to evaporate slowly and colorless columnar crystals of (MBBZIM)-(1,2-F4DIB) were obtained after 3 d.

2.2.7. 2(MMBZIM)-(1,4-F4DIB)-2(H2O). 2-Mercapto-5-methylbenzimidazole (40 mg, 0.244 mmol) and 1,4-diodotetrafluorobenzene (51 mg, 0.127 mmol) were weighed into a 20 ml glass vial. Methanol (10 ml) was added and the mixture was stirred until a clear solution was obtained. The solvent was allowed to evaporate slowly and colorless plate-like crystals of 2(MMBZIM)-(1,4-F4DIB)-2(H2O) were obtained after 3 d.

2.2.8. (MBBZIM)-(1,3,5-F3I3B). 2-Mercapto-5-methylbenzimidazole (31 mg, 0.189 mmol) and 1,3,5-trifluoro-2,4,6-triiodobenzene (50 mg, 0.098 mmol) were weighed into a 20 ml glass vial. Methanol (10 ml) was added and the mixture was stirred until a clear solution was obtained. The solvent was allowed to evaporate slowly and colorless needle-like crystals of (MBBZIM)-(1,3,5-F3I3B) were obtained after 4 d.

2.2.9. (MBZOX)-(1,2-F4DIB). 2-Mercaptobenzoxazole (20 mg, 0.132 mmol) and 1,2-diodotetrafluorobenzene (102 mg, 0.254 mmol) were weighed into a 20 ml glass vial. Methanol (10 ml) was added and the mixture was stirred until a clear solution was obtained. The solvent was allowed to evaporate slowly and colorless needle-like crystals of (MBZOX)-(1,2-F4DIB) were obtained after 3 d.

2.2.10. (MBZOX)-(1,3,5-F3I3B). 2-Mercaptobenzoxazole (19 mg, 0.126 mmol) and 1,3-diiodotetrafluorobenzene (104 mg, 0.259 mmol) were weighed into a 20 ml glass vial. Acetone (10 ml) was added and the mixture was stirred until a clear solution was obtained. The solvent was allowed to evaporate slowly and colorless columnar crystals of (MBZOX)-(1,2-F4DIB) were obtained after 4 d.

2.2.11. (MBZOX)-(1,3-F4DIB). 2-Mercaptobenzoxazole (15 mg, 0.099 mmol) and 1,3,5-trifluoro-2,4,6-triiodobenzene (50 mg, 0.098 mmol) were weighed into a 20 ml glass vial. Acetone (10 ml) was added and the mixture was stirred until a clear solution was obtained. The solvent was allowed to evaporate slowly and colorless columnar crystals of (MBZOX)-(1,3,5-F3I3B) were obtained after 1 d.

2.2.12. (MBZOX)-(1,3,5-F3I3B). 2-Mercaptobenzothiazole (21 mg, 0.126 mmol) and 1,2-diodotetrafluorobenzene (103 mg, 0.256 mmol) were weighed into a 20 ml glass vial. Methanol (10 ml) was added and the mixture was stirred until a clear solution was obtained. The solvent was allowed to evaporate slowly and colorless columnar crystals of (MBZOX)-(1,3,5-F3I3B) were obtained after 1 d.

2.2.13. 3(MBZTH)-(4,1,2-F4DIB). 2-Mercaptobenzothiazole (24 mg, 0.143 mmol) and 1,3-diiodotetrafluorobenzene (50 mg, 0.124 mmol) were weighed into a 20 ml glass vial. Methanol (10 ml) was added and the mixture was stirred until a clear solution was obtained. The solvent was allowed to evaporate slowly and colorless plate-like crystals of 3(MBZTH)-(4,1,2-F4DIB) were obtained after 3 d.
slowly and colorless tabular crystals of (MBZTH)-2(1,3-F₄DIB) were obtained after 4 d.

2.2.16. 2-(MBZTH)-(1,4-F₄DIB). 2-Mercaptobenzothiazole (46 mg, 0.275 mmol) and 1,4-diodotetrafluorobenzene (50 mg, 0.124 mmol) were weighed into a 20 ml glass vial. Methanol (10 ml) was added and the mixture was stirred until a clear solution was obtained. The solvent was allowed to evaporate slowly and colorless block-like crystals of (MBZTH)-(1,3,5-F₃I₃B) were obtained after 2 d.

2.2.17. (MBZTH)-(1,3,5-F₃I₃B). 2-Mercaptobenzothiazole (32 mg, 0.191 mmol) and 1,3,5-trifluoro-2,4,6-triiodobenzene (50 mg, 0.098 mmol) were weighed into a 20 ml glass vial. Acetone (10 ml) was added and the mixture was stirred until a clear solution was obtained. The solvent was allowed to evaporate slowly and colorless tabular crystals of (MBZTH)-(1,3,5-F₃I₃B) were obtained after 5 d.

2.3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms on C atoms were calculated in idealized positions riding on their parent atoms, with C—H = 0.98 Å and Uiso(H) = 1.5Ueq(C) for methyl H atoms, and C—H = 0.95 Å and Uiso(H) = 1.2Ueq(C) for other H atoms. H atoms on heteroatoms were located in difference Fourier maps and refined isotropically, utilizing appropriate restraints [N—H = 0.86 (2) Å] where necessary to maintain chemically reasonable geometries. The H atoms of the water molecule in 2(MMBZIM)-(1,4-F₄DIB)-2(H₂O)were modeled in a disordered arrangement due to symmetry considerations.

3. Results and discussion

3.1. Cocrystals of imidazolidine-2-thione (IT)

The smallest of the sulfur-containing compounds within this study, imidazolidine-2-thione, contains a thiourea functionality within a five-membered saturated ring. The first cocrystal formed with this compound in the present study is 2(IT)-(1,3-F₄DIB), which was refined in the orthorhombic space group *Pbnm* with two unique molecules of IT and one molecule of 1,3-F₄DIB in the asymmetric unit (Fig. 2). As is common in thiourea-containing structures, a pair of N—H···S hydrogen bonds links thiourea molecules, in this case, into tetrameric units (Table 2) (Peloquin et al., 2021d, 2022). This is in contrast to the formation of hydrogen-bonded ribbons and discrete dimers, which are formed in the previously published 2(IT)-(1,2-F₂DIB) and (IT)-2(1,2-F₂DIB) cocrystals, respectively (Happonen et al., 2021). Tetrameric units align into staggered stacks in the *b* direction. These stacks are separated by additional tetrameric units, with the planes of the tetramers inclined by approximately 64°. This arrangement of inclined hydrogen-bonding units is also observed in the dimeric units of (IT)-(1,4-F₄DIB) (Happonen et al., 2021). At the end of each tetramer, the remaining N—H hydrogen serves to link to the next inclined tetramer via N—H···S hydrogen bonding. The S atom at this end, S₁, acts as a C—I···S halogen-bond acceptor to two different 1,3-F₄DIB molecules (Table 3). These halogen-bonding interactions link adjacent stacks of tetramers in the *c* direction. The second IT-containing cocrystal of this study, (IT)-(1,3,5-F₃I₃B), was refined in the orthorhombic space group *Pbca* with one molecule each of IT and 1,3,5-F₃I₃B in the asymmetric unit. This structure represents the only example within this study without N—H···S...
hydrogen bonding (Table 4). Instead, C—I···S halogen bonding occurs between alternating molecules of IT and 1,3,5-F3I3B to form chains propagating in the c direction. The third I atom of 1,3,5-F3I3B, which does not participate in significant interactions with sulfur, instead serves to link chains in the ac plane via C—I···I halogen bonding.

3.2. Cocystals of 2-mercaptobenzimidazole (MBZIM)

Moving to the larger thiourea-containing molecule 2-mercaptobenzimidazole (MBZIM) yielded three new structures dominated by co-operative hydrogen and halogen bonding (Fig. 3). With 1,3-F4DIB, the cocrystalline structure of 4(MBZIM)-3(1,3-F4DIB) was obtained in the triclinic space group P1, with four unique molecules of MBZIM and three molecules of 1,3-F4DIB in the asymmetric unit. In this structure, hydrogen bonding between thiourea molecules contributes to the formation of ribbons propagating along the a axis (Table 5). Two of the three 1,3-F4DIB molecules are pendants along these chains, linked via C—I···S. The second I atom of these particular 1,3-F4DIB molecules does not contribute to significant halogen- or chalcogen-bonding interactions. This

Table 3

Halogen- and chalcogen-bond geometries (Å, °).

| Compound            | d(D···A) | R_{DA}^0 | θ(C—D···A) | θ(D···A—C) | θ_D^0—θ_A^0 | γ^0 |
|---------------------|---------|---------|------------|------------|-------------|-----|
| 2(IT)(1,3,F4DIB)    |         |         |            |            |             |     |
| I1···S              | 3.2265  | 0.85    | 174.51     | 113.51     | 61.00       | 0.79 |
| I2···S              | 3.2806  | 0.87    | 176.10     | 99.67      | 76.43       | 0.03 |
| (IT)(1,3,5,F3I3B)   |         |         |            |            |             |     |
| I1···S              | 3.8376  | 0.97    | 162.95     | 106.24     | 56.71       | 0.64 |
| I2···S              | 3.1505  | 0.84    | 171.86     | 101.89     | 69.97       | 0.48 |
| 4(MBZIM)-3(1,3,F4DIB)|         |         |            |            |             |     |
| I1···S              | 3.1754  | 0.84    | 177.68     | 90.59      | 87.09       | 0.18 |
| I2···S              | 3.3361  | 0.88    | 172.18     | 136.86     | 35.22       | 0.83 |
| (MBZIM)(1,4,F4DIB)  |         |         |            |            |             |     |
| I1···S              | 3.2573  | 0.86    | 168.29     | 131.28     | 37.01       | 0.57 |

Notes: (i) R_{DA} = d(X—Y)/Σ(dvdW); the ratio of the distance between the donor atom (i.e. D) and the acceptor atom (i.e. S) to the sum of their van der Waals (vdW) radii (S = 1.80 Å and I = 1.98 Å) (Auffinger et al., 2004). (ii) θ_D^0—θ_A^0 = |[(θ(C—D···A) — θ(D···A—C))]. (iii) Linearity parameter (Setter et al., 2020).

Table 4

Hydrogen-bond geometry (Å, °) for (IT)(1,3,5-F3I3B).

| D—H···A | D—H | H—A | D···A | D—H···A |
|---------|-----|-----|-------|---------|
| N2—H2N2···I2 | 0.83 (2) | 3.10 (3) | 3.74 (3) | 137 (3) |
| C2—H2B···I1 | 0.99 | 3.31 | 3.92 (3) | 122 |
| C2—H2B···F3 | 0.99 | 2.47 | 3.14 (3) | 125 |

Symmetry codes: (i) x, y + 1, z; (ii) x + 1, y, z; (iii) x, y, z; (iv) x, y, z; (v) x, y, z; (vi) x, y, z.
hydrogen-bonding thiourea ribbon with halogen-bonding pendants is analogous to that observed in (MBZIM)-1,2-F4DIB (Arman et al., 2008, 2010). The final unique 1,3-F4DIB molecule lies between the ring planes of the pendant molecules of 1,3-F4DIB, contributing to only weak C—I···H, C—F···H, and C—F···F—C interactions. The combination of MBZIM and 1,4-F4DIB resulted in the (MBZIM)-1,4-F4DIB cocrystal, refined in the monoclinic space group P21/c, with one molecule each of both MBZIM and 1,4-F4DIB in the asymmetric unit. Just as in 4(MBZIM)-3(1,3-F4DIB), the structure of (MBZIM)-1,4-F4DIB consists of ribbons of MBZIM molecules propagating in the c direction, formed through thiourea hydrogen bonding (Table 6).

Table 5

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|-----|------|-------|---------|
| N1—HN1···S2' | 0.85 (2) | 2.52 (2) | 3.357 (2) | 172 (3) |
| N2—HN2···S2 | 0.85 (2) | 2.46 (2) | 3.297 (2) | 166 (2) |
| N3—HN3···S1 | 0.85 (2) | 2.51 (2) | 3.348 (2) | 173 (3) |
| N4—HN4···S1' | 0.85 (2) | 2.50 (2) | 3.326 (2) | 166 (2) |
| N5—HN5···S4' | 0.85 (2) | 2.49 (2) | 3.326 (2) | 169 (3) |
| N6—HN6···S4 | 0.85 (2) | 2.43 (2) | 3.270 (2) | 169 (3) |
| C17—H17···F36' | 0.95 | 2.61 | 3.385 (3) | 139 |
| C20—H20···F36'' | 0.95 | 2.51 | 3.235 (3) | 133 |
| N7—HN7···S3 | 0.84 (2) | 2.47 (2) | 3.300 (2) | 170 (3) |
| N8—HN8···S3'' | 0.85 (2) | 2.48 (2) | 3.302 (2) | 163 (3) |

Symmetry codes: (i) \( x+1, y, z \); (ii) \( x-1, y, z \); (iii) \( -x+1, -y+1, -z+1 \); (iv) \( -x, -y+1, -z+1 \).

Figure 3

Cocrystal structures containing MBZIM. Hydrogen and halogen bonding are indicated by black dotted lines. Displacement ellipsoids are drawn at the 50% probability level. H atoms, except those bound to N atoms, have been omitted for clarity.
**Table 6**
Hydrogen-bond geometry (Å, °) for (MBZIM)-(1,4-F4DIB).

| D—H···A     | D—H     | H···A   | D···A   | D—H···A  |
|------------|---------|---------|---------|----------|
| N1—H11···S1' | 0.84 (3) | 2.47 (3) | 3.3089 (18) | 172 (2)  |
| N2—H22···S1'' | 0.86 (3) | 2.50 (3) | 3.3527 (17) | 172 (2)  |

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

**Table 7**
Hydrogen-bond geometry (Å, °) for (MBZIM)-(TIE).

| D—H···A      | D—H     | H···A   | D···A   | D—H···A  |
|-------------|---------|---------|---------|----------|
| N1—H11···S1' | 0.87 (5) | 2.47 (5) | 3.335 (3) | 178 (5)  |
| C3—H3···I1'' | 0.95     | 3.28    | 3.881 (4) | 123      |

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

**Table 8**
Hydrogen-bond geometry (Å, °) for (MMBZIM)-(1,2-F4DIB).

| D—H···A       | D—H     | H···A   | D···A   | D—H···A  |
|---------------|---------|---------|---------|----------|
| N1—H11···S1'  | 0.88 (5) | 2.57 (5) | 3.444 (3) | 173 (4)  |
| N2—H22···I1   | 0.85 (2) | 3.07 (3) | 3.780 (3) | 142 (3)  |
| N2—H22···F4   | 0.85 (2) | 2.56 (3) | 3.122 (4) | 124 (3)  |
| C3—H3···I2''  | 0.95     | 3.06    | 3.966 (4) | 160      |
| C6—H6···F4    | 0.95     | 2.63    | 3.262 (4) | 125      |

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

1,4-F4DIB act as pendants along these ribbons, linked via C—I···S halogen bonding.

With four I atoms available, tetraiodoethylene (TIE) often enables structural motifs that are different from the typical aromatic halogen-bond donors. The cocrystal (MBZIM)-(TIE) was refined in the orthorhombic space group Pnma, with one molecule each of MBZIM and TIE in the asymmetric unit. As in the previous examples, molecules of MBZIM form infinite ribbons through thiourea hydrogen bonding (Table 7). Three of the four I atoms of TIE function as C—I···S halogen-bond donor atoms to link these ribbons, creating a three-dimensional framework through the combination of hydrogen and halogen bonding. The fourth I atom participates in a C—I···π interaction [I···π = 3.351 (3) Å] to reinforce the framework.

### 3.3. Cocrystals of 2-mercapto-5-methylbenzimidazole (MMBZIM)

Adding a methyl group to MBZIM, resulting in 2-mercapto-5-methylbenzimidazole (MMBZIM), induces significant changes to the overall hydrogen- and halogen-bonding motifs. The structural literature of this substrate is limited, having only been characterized by single-crystal X-ray diffraction when acting as a ligand for transition metals coordinating through its S atom (Lin et al., 2017; Ozturk et al., 2009; Mitra et al., 2012). The first halogen-bonded cocrystal of MMBZIM in this study, (MMBZIM)-(1,2-F4DIB), was refined in the triclinic space group P1̅, with one molecule each of MMBZIM and 1,2-F4DIB in the asymmetric unit (Fig. 4). A discrete hydrogen-bonded dimer of two MMBZIM molecules is observed, in contrast to the infinite ribbons in (MBZIM)-(1,2-F4DIB) and most of the cocrystals in the present study (Table 8). Two molecules of 1,2-F4DIB per MMBZIM molecule link the dimers via C—I···S halogen bonds, leading to the formation of chains along the c axis.

Isolated as a hydrated cocrystal from adventitious water, 2(MMBZIM)-(1,4-F4DIB)-2(H2O) crystallizes in the triclinic space group P1̅ with one molecule each of MMBZIM and H2O, as well as half a molecule of 1,4-F4DIB, in the asymmetric unit. All attempts to obtain an nonhydrated cocrystal...
with 1,4-F₄DIB were unsuccessful, suggesting the packing arrangement formed strictly by halogen bonding contains small but meaningful voids that must be occupied by the water molecule. Discrete hydrogen-bonded dimers are again observed by hydrogen bonding of the thioureas (Table 9). Differing from (MMBZIM)-(1,2-F₄DIB), with two halogen bonds to each S atom, 2(MMBZIM)-(1,4-F₄DIB)-2(H₂O) utilizes one C—I···S halogen bond and one O—H···S hydrogen bond at each S atom. It is the halogen bonding that contributes to the formation of infinite chains by linking the S atoms, contributing to chains propagating in the a direction. The third I atom is oriented as a potential acceptor for a C—F···I interaction, with the S halogen bond and one O—H···S hydrogen bond at each S atom, contributing to the formation of infinite hydrogen-bonding ribbon with pendant halogen-bonded dimers stacked along the b axis. Molecules of MMBZIM form dimeric pairs through hydrogen bonding of the thioureas (Table 10). The remaining N—H hydrogens are involved in weak N—H···I hydrogen bonds [H···I = 3.02 (8) Å]. A pair of C—I···S halogen bonds occurs at each S atom, contributing to chains propagating in the a direction. The third I atom is oriented as a potential acceptor for a C—F···I interaction, though the interaction distance is very near the sum of the van der Waals radii and it is unclear if there is a significant attraction to this interaction. Given the similar motifs of (MMBZIM)-(1,3,5-F₃I₃B) to (MMBZIM)-(1,2-F₄DIB), it may be that the C—F···I contact is merely coincident within the motif formed by the N—H···S and C—I···S interactions.

### 3.4. Cocrystals of 2-mercaptopbenzoxazole (MBZOX)

While infinite ribbons commonly formed through hydrogen bonding of the thioureas in MMBZIM, substituting one secondary N atom for an O atom in 2-mercaptopbenzoxazole (MBZOX) allows for the study of the structural motifs when only dimers can form through hydrogen bonding (Fig. 5). The structural literature surrounding MBZOX is sparse, limited to three reports of it acting as a ligand through the S atom in transition-metal complexes (McFarlane et al., 1998; Nakahodo et al., 2000; Mitra et al., 2012) and its reaction with diiodine (Cristiani et al., 1995). Combined with 1,2-F₄DIB, the cocrystal structure of (MBZOX)-(1,2-F₄DIB) was refined in the monoclinic space group P2₁/n, with one unique molecule each of MBZOX and 1,2-F₄DIB in the asymmetric unit. Here, a hydrogen-bonding thiouamide dimer is formed (Table 11), with each S atom acting as an acceptor to a single C—I···S halogen bond. The second I atom does not contribute to an additional halogen bond, instead being involved in a weak C—I···π interaction. This discrete four-molecule unit formed through hydrogen and halogen bonding stands in stark contrast to the infinite hydrogen-bonding ribbon with pendant halogen-bonded 1,2-F₄DIB molecules observed in (MMBZIM)-(1,2-F₄DIB). The pattern of interactions in (MBZOX)-(1,3-F₃DIB), which crystallizes in the monoclinic space group P2₁/c, with one molecule each of MBZOX and 1,3-F₃DIB in the asymmetric unit, is more complex. Thiouamide hydrogen-bonding dimers are once again observed (Table 12). These dimers stack along the b axis. Molecules of 1,3-F₃DIB link neighboring stacks of dimers in the a direction. One of the I atoms serves as both a C—I···S halogen-bond donor and a C=S···I chalcogen-bond acceptor. The combination of halogen, chalcogen, and hydrogen-bonding results in the formation of a two-dimensional motif of intermolecular

### Table 9

| D—H···A  | D—H | H···A | D—A | D—H···A |
|---------|-----|------|-----|--------|
| N—I—H11—S1 | 0.83 (2) | 2.06 (2) | 2.8763 (17) | 166 (2) |
| N—I—H12—S1 | 0.88 (2) | 2.57 (2) | 3.4211 (13) | 164 (2) |
| C—I—H4—S1 | 0.95 | 3.03 | 3.9505 (14) | 164 |
| O—I—H14—O1 | 0.88 (2) | 1.85 (2) | 2.708 (3) | 163 (4) |
| O—I—H13—O1 | 0.88 (2) | 1.89 (2) | 2.759 (3) | 167 (4) |
| O—I—H20—H—S1 | 0.87 (2) | 3.16 (3) | 3.7419 (12) | 126 (2) |
| O—I—H20—H—S1 | 0.87 (2) | 2.65 (2) | 3.4251 (13) | 149 (3) |

### Table 10

| D—H···A  | D—H | H···A | D—A | D—H···A |
|---------|-----|------|-----|--------|
| N—I—H11—S1 | 0.86 (2) | 2.57 (3) | 3.426 (7) | 173 (10) |
| N—I—H12—S1 | 0.85 (8) | 3.02 (8) | 3.657 (7) | 133 (7) |
| C—I—H3—S1 | 0.95 | 3.12 | 4.035 (9) | 163 |
| C—I—H6—S1 | 0.95 | 3.14 | 3.927 (8) | 142 |

### Table 11

| D—H···A  | D—H | H···A | D—A | D—H···A |
|---------|-----|------|-----|--------|
| N—I—H11—S1 | 0.85 (8) | 2.50 (8) | 3.335 (6) | 167 (8) |
| C—I—H3—S1 | 0.95 | 3.19 | 4.108 (7) | 162 |

### Table 12

| D—H···A  | D—H | H···A | D—A | D—H···A |
|---------|-----|------|-----|--------|
| N—I—H11—S1 | 0.88 (3) | 2.52 (3) | 3.3906 (19) | 172 (3) |
| C—I—H3—S1 | 0.95 | 3.10 | 4.030 (2) | 166 |

### Table 13

| D—H···A  | D—H | H···A | D—A | D—H···A |
|---------|-----|------|-----|--------|
| N—I—H11—S1 | 0.87 (4) | 2.45 (4) | 3.316 (3) | 178 (4) |
| C—I—H3—S1 | 0.95 | 3.16 | 4.066 (3) | 159 |
interactions. In 2(MBZOX)-(1,4-F₄DIB), which was refined in the monoclinic space group C2/c, with one molecule of MBZOX and one-half of a molecule of 1,4-F₄DIB, the packing motif is more reminiscent of its MMBZIM analogue. Thioamide hydrogen-bonding dimers are linked into chains through C—I···S halogen bonding (Table 13). The final example in the MBZOX series, (MBZOX)-(1,3,5-F₃I₃B), was refined in the monoclinic space group P2₁/c, with one molecule each of MBZOX and 1,3,5-F₃I₃B in the asymmetric unit. Much of the packing is similar to (MBZOX)-(1,3-F₄DIB), with thioamide hydrogen-bonding dimers stacking in the b direction (Table 14). Neighboring stacks are linked along the a axis by both C—I···S halogen bonding and a C—I···I chalcogen bond to again form a two-dimensional substructure. In this instance though, the third I atom of 1,3,5-F₃I₃B acts as a C—I···I halogen-bond donor, further consolidating the packing in the c direction to form a three-dimensional framework. In all cases of these MBZOX cocrystals, hydrogen- and halogen-bonding preference is given toward the thione S atom as the acceptor rather than the O atom of the heterocycle.

3.5. Cocrystals of 2-mercaptobenzothiazole (MBZTH)

As with MBZOX, 2-mercaptobenzothiazole lacks the thiourea functionality to allow for the formation of infinite ribbons through hydrogen bonding; however, the additional S atom can potentially act in either halogen- or chalcogen-bonding interactions (Fig. 6). Just as with MBZOX, the prior structural literature is dominated by examples of MBZTH acting as a ligand in transition-metal complexes (Aslanidis et al., 2002; Zhou et al., 2013b; Hadjikakou & Kubicki, 2000) or reactions with dihalides (Daga et al., 2002; Koskinen et al.,

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**Table 14**

Hydrogen-bond geometry (Å, °) for (MBZOX)-(1,3,5-F₃I₃B).

| D       | H       | A       | D—H     | H—A     | D—A     | D—H—A   |
|---------|---------|---------|---------|---------|---------|----------|
| N1—HN1 | 0.85    | S1      | 2.53    | 3.377   | 176     | 167      |
| C3—H3  | 0.95    | I1      | 3.04    | 3.969   | 167     | 140      |
| C6—H6  | 0.95    | I2      | 3.23    | 4.009   | 140     |          |

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

**Table 15**

Hydrogen-bond geometry (Å, °) for 3(MBZTH)-(4,1,2-F₄DIB).

| D       | H       | A       | D—H     | H—A     | D—A     | D—H—A   |
|---------|---------|---------|---------|---------|---------|----------|
| N2—HN2 | 0.88    | S1      | 2.45    | 3.266   | 149     | 174      |
| N1—HN1 | 0.88    | S3      | 2.40    | 3.266   | 169     |          |
| C6—H6  | 0.95    | F10     | 2.60    | 3.29    | 130     | 170      |
| N3—HN3 | 0.88    | S5      | 2.42    | 3.290   | 170     |          |
| C17—H17| 0.95    | F16     | 2.30    | 3.232   | 166     |          |
| C20—H20| 0.95    | F16     | 2.53    | 3.128   | 121     |          |
| C20—H20| 0.95    | F16     | 2.54    | 3.181   | 125     |          |

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

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Figure 5
Cocrystalline structures containing MBZOX. Hydrogen and halogen bonding are indicated by black dotted lines. Displacement ellipsoids are drawn at the 50% probability level. H atoms, except those bound to N atoms, have been omitted for clarity.
2015a,b). The first and most complex of the MBZTH structures obtained, 3(MBZTH)·4(1,2-F4DIB), crystallized in the triclinic space group $P\overline{1}$, with three molecules of MBZTH and four molecules of 1,2-F4DIB in the asymmetric unit. Thioamide dimers stack along the $a$ axis (Table 15), with one molecule of 1,2-F4DIB within alternating layers. The remaining molecules of 1,2-F4DIB are oriented approximately perpendicular to the thioamide dimers, linking layers of the stack through a series of C—I···S halogen bonds. The intra-stack molecule of 1,2-F4DIB is also linked to a molecule of 1,2-F4DIB on the edge of the stack through a C—I···I halogen bond. This complex series of interactions ultimately forms a three-dimensional framework.

The packing motif of (MBZTH)·(1,3-F4DIB), refined in the triclinic space group $P\overline{1}$, with one molecule each of MBZTH and 1,3-F4DIB within the asymmetric unit, is similar to that of (MMBZIM)·(1,2-F4DIB) and (MMBZIM)·(1,3,5-F3I3B). Thioamide hydrogen-bonding dimers (Table 16) are linked by a pair of unique C—I···S halogen bonds to the thione S atom, forming chains in the $c$ direction. Crystallizing in the monoclinic

Figure 6
Cocrystalline structures containing MBZTH. Hydrogen and halogen bonding are indicated by black dotted lines. Displacement ellipsoids are drawn at the 50% probability level. H atoms, except those bound to N atoms, have been omitted for clarity.
The third I atom serves to link neighboring chains through a weak C—I H...S S atoms. The geometry of this interaction (C—I H...S S = 149.3 (1) and 142.48 (13)°) is indicative of a dispersive Type I sigma-hole interaction.

Table 16
Hydrogen-bond geometry (Å, °) for (MBZTH)-(1,3-F₄DIB).

|         | D—H  | H···A | D—H  | H···A | D—D  | D—H  |
|---------|------|-------|------|-------|------|------|
| N1—HN1···S1' | 0.87 (3) | 2.45 (3) | 3.3120 (15) | 175 (2) |

Symmetry code: (i) −x + 2, −y + 2, −z + 2.

Table 17
Hydrogen-bond geometry (Å, °) for (MBZTH)-2(1,3-F₄DIB).

|         | D—H  | H···A | D—H  | H···A | D—D  | D—H  |
|---------|------|-------|------|-------|------|------|
| N1—HN1···S3 | 0.89 (6) | 2.51 (6) | 3.376 (6) | 165 (5) |
| C3—H3···I1 | 0.95 | 3.10 | 3.976 (7) | 154 |
| N2—HN2···S1 | 0.85 (3) | 2.52 (3) | 3.360 (6) | 161 (7) |
| C10—H10···F3' | 0.95 | 3.09 | 4.006 (6) | 161 |

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

Table 18
Hydrogen-bond geometry (Å, °) for 2(MBZTH)-(1,4-F₄DIB).

|         | D—H  | H···A | D—H  | H···A | D—D  | D—H  |
|---------|------|-------|------|-------|------|------|
| N1—HN1···S1' | 0.78 (3) | 2.60 (3) | 3.369 (2) | 170 (3) |
| C3—H3···F1' | 0.95 | 2.50 | 3.333 (3) | 146 |
| C6—H6···F2' | 0.95 | 2.44 | 3.357 (3) | 162 |

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

The thioamide hydrogen-bonding dimers (Table 17) are linked into chains by C—I H...S halogen bonding to the thione S atom. The hydrogen-bonded units were then aggregated into longer-range one- or two-dimensional motifs through C—I H...S halogen bonding. Additional C—I H...S halogen bonding, either through the stoichiometric excess of organoiodine or through the use of more iodine-rich organoiodine substrates (tetraiodoethylene, for example) extended some structures into three-dimensional frameworks. The Rₓ value for the majority of the halogen-bonding interactions lies within a typical range from 0.85 to 1.0. The interactions to a thione S atom generally occurred at shorter distances than the thiane S atom, as expected due to the hybridization state. The linearity parameter, ψ, ranges from 0.02 to 0.83. This wide range is supported by the distribution of electron density on S or I acceptor atoms. Occasional C=S...I chalcogen bonding was observed. Halogen-bond preference toward the thione S atom over the heterocyclic O or S atom was observed in both the benzoxazoles and benzothiazoles. However, there were at least some occasional occurrences of C—I H...S to the thiazole S atom.

Table 19
Hydrogen-bond geometry (Å, °) for (MBZTH)-(1,3,5-F₃I₃B).

|         | D—H  | H···A | D—H  | H···A | D—D  | D—H  |
|---------|------|-------|------|-------|------|------|
| N1—HN1···S1' | 0.86 (2) | 2.54 (2) | 3.389 (3) | 172 (4) |
| C3—H3···I1' | 0.95 | 3.03 | 3.928 (4) | 159 |

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

Table 20
Hydrogen-bond geometry (Å, °) for (MBZTH)-(TIE).

|         | D—H  | H···A | D—H  | H···A | D—D  | D—H  |
|---------|------|-------|------|-------|------|------|
| N1—HN1···S1' | 0.85 (2) | 2.43 (2) | 3.275 (5) | 170 (6) |

Symmetry code: (i) −x, −y + 1, −z + 2.

4. Conclusion
A rich structural chemistry of cocrystals was observed between organoiodine molecules and heterocyclic thiones in the present study of 18 crystal structures. The structures are primarily directed by the co-operative effects of hydrogen- and halogen-bonding interactions. Certain features of the long-range structures were controlled through the selection of the heterocyclic thione, where the formation of primarily hydrogen-bonded ribbons in benzimidazoles could be truncated to hydro-bonded dimers in benzoxazoles and benzothiazoles. The hydrogen-bonded units were then aggregated into longer-range one- or two-dimensional motifs through C—I H...S halogen bonding. Additional C—I H...S halogen bonding, either through the stoichiometric excess of organoiodine or through the use of more iodine-rich organoiodine substrates (tetraiodoethylene, for example) extended some structures into three-dimensional frameworks. The Rₓ value for the majority of the halogen-bonding interactions lies within a typical range from 0.85 to 1.0. The interactions to a thione S atom generally occurred at shorter distances than the thiane S atom, as expected due to the hybridization state. The linearity parameter, ψ, ranges from 0.02 to 0.83. This wide range is supported by the distribution of electron density on S or I acceptor atoms. Occasional C=S...I chalcogen bonding was observed. Halogen-bond preference toward the thione S atom over the heterocyclic O or S atom was observed in both the benzoxazoles and benzothiazoles. However, there were at least some occasional occurrences of C—I H...S to the thiazole S atom.

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Halogen, chalcogen, and hydrogen bonding in organoiodine cocrystals of heterocyclic thiones: imidazolidine-2-thione, 2-mercaptobenzimidazole, 2-mercapto-5-methylbenzimidazole, 2-mercaptobenzoxazole, and 2-mercaptobenzothiazole

Spencer Watts, Andrew J. Peloquin, Madhushi Bandara, Colin D. McMillen and William T. Pennington

Computing details
For all structures, data collection: APEX3 (Bruker, 2017); cell refinement: SAINT (Bruker, 2017); data reduction: SAINT (Bruker, 2017); program(s) used to solve structure: SHELXT2018 (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2018 (Sheldrick, 2015b). Molecular graphics: Mercury (Macrae et al., 2020) for 2IT_13F4DIB, 4MBZIM_313F4DIB, MBZIM_14F4DIB, MBZIM_TIE, MMBZIM_12F4DIB, 2MMBZIM_14F4DIB_2H2O, MMBZIM_135F3I3B, MBZOX_12F4DIB, MBZOX_13F4DIB, 2MBZOX_14F4DIB, MBZOX_135F3I3B, 3MBZTH_412F4DIB, MBZTH_13F4DIB, MBZTH_213F4DIB, MBZTH_14F4DIB, MBZTH_13F4DIB, MBZTH_135F3I3B, MBZTH_TIE; OLEX2 (Dolomanov et al., 2009) for IT_135F3I3B. For all structures, software used to prepare material for publication: OLEX2 (Dolomanov et al., 2009).

1,2,3,5-Tetrafluoro-4,6-diiodobenzene–imidazolidine-2-thione (1/2) (2IT_13F4DIB)

Crystal data
\[ C_6F_4I_2\cdot 2C_3H_6N_2S \]
\[ M_r = 606.18 \]
Orthorhombic, \( Pbcn \)
\[ a = 15.6704 (7)\, \text{Å} \]
\[ b = 8.9924 (4)\, \text{Å} \]
\[ c = 26.0573 (10)\, \text{Å} \]
\[ V = 3671.9 (3)\, \text{Å}^3 \]
\[ Z = 8 \]
\[ F(000) = 2288 \]
\[ D_c = 2.193\, \text{Mg}\, \text{m}^{-3} \]
Mo \( K\alpha \) radiation, \( \lambda = 0.71073\, \text{Å} \)
Cell parameters from 9343 reflections
\[ \theta = 2.6–30.1^\circ \]
\[ \mu = 3.69\, \text{mm}^{-1} \]
\[ T = 100\, \text{K} \]
Block, colourless
\[ 0.18 \times 0.17 \times 0.13\, \text{mm} \]

Data collection
Bruker D8 Venture Photon 2 diffractometer
Radiation source: Incoatec I\( \beta \)S
\( \varphi \) and \( \omega \) scans
Absorption correction: multi-scan
(SADABS; Bruker, 2017)
\[ T_{\text{min}} = 0.639, T_{\text{max}} = 0.746 \]
112821 measured reflections
5376 independent reflections
5198 reflections with \( I > 2\sigma(I) \)
\[ R_{\text{int}} = 0.035 \]
\[ \theta_{\text{max}} = 30.1^\circ, \theta_{\text{min}} = 2.0^\circ \]
\( h = -22\to 22 \)
\( k = -12\to 12 \)
\( l = -36\to 31 \)
Refinement

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.015$
$wR(F^2) = 0.032$
$S = 1.25$
5376 reflections
234 parameters
0 restraints
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma(F_o^2) + (0.0054P)^2 + 3.3158P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta \sigma)_{\text{max}} = 0.003$
$\Delta \rho_{\text{max}} = 0.42 \text{ e} \AA^{-3}$
$\Delta \rho_{\text{min}} = -0.36 \text{ e} \AA^{-3}$

Extinction correction: SHELXL2018
(Sheldrick, 2015b),
$Fc^* = kFc[1 + 0.001xFc^2\lambda^3/sin(2\theta)]^{1/4}$
Extinction coefficient: 0.00082 (4)

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   | $U_{\text{iso}}$/*$U_{\text{eq}}$ |
|---|-----|-----|-----|---------------------------------|
|I  | 0.71842 (2) | 0.27502 (2) | 0.20785 (2) | 0.01326 (3) |
|I2 | 0.98485 (2)  | 0.54110 (2)  | 0.35028 (2)  | 0.01761 (3)  |
|I1 | 0.89456 (6)  | 0.34626 (11) | 0.26335 (4)  | 0.01975 (19) |
|F2 | 0.81145 (7)  | 0.69420 (11) | 0.38910 (4)  | 0.0230 (2)   |
|F3 | 0.64754 (6)  | 0.66108 (12) | 0.36116 (4)  | 0.0232 (2)   |
|F4 | 0.60595 (6)  | 0.48196 (12) | 0.28175 (4)  | 0.0215 (2)   |
|C7 | 0.74984 (10) | 0.40891 (16) | 0.27097 (5)  | 0.0139 (3)   |
|C8 | 0.83358 (10) | 0.42536 (17) | 0.28725 (6)  | 0.0145 (3)   |
|C9 | 0.85758 (10) | 0.51990 (17) | 0.32680 (6)  | 0.0157 (3)   |
|C10| 0.79313 (10) | 0.59875 (17) | 0.35087 (6)  | 0.0165 (3)   |
|C11| 0.70856 (10) | 0.58350 (18) | 0.33669 (6)  | 0.0172 (3)   |
|C12| 0.68786 (10) | 0.49038 (18) | 0.29640 (6)  | 0.0155 (3)   |
|S1 | 0.68567 (2)  | 0.95036 (4)  | 0.61354 (2)  | 0.01476 (7)  |
|N1 | 0.69251 (9)  | 0.72075 (15) | 0.54616 (5)  | 0.0166 (3)   |
|HN1| 0.7189 (14)  | 0.672 (3)    | 0.5672 (9)   | 0.027 (6)*   |
|N2 | 0.64832 (10) | 0.92951 (15) | 0.51306 (5)  | 0.0182 (3)   |
|HN2| 0.6289 (14)  | 1.015 (2)    | 0.5136 (8)   | 0.022 (5)*   |
|C1 | 0.67475 (9)  | 0.86294 (17) | 0.55568 (6)  | 0.0134 (3)   |
|C2 | 0.68382 (11) | 0.68448 (18) | 0.49167 (6)  | 0.0186 (3)   |
|H2A| 0.644157     | 0.600134     | 0.486460     | 0.022*       |
|H2B| 0.739772     | 0.659716     | 0.476228     | 0.022*       |
|C3 | 0.64743 (11) | 0.82909 (18) | 0.46895 (6)  | 0.0199 (3)   |
|H3A| 0.683895     | 0.867131     | 0.440842     | 0.024*       |
|H3B| 0.588726     | 0.814231     | 0.455882     | 0.024*       |
|S2 | 0.42217 (3)  | 0.71986 (5)  | 0.49680 (2)  | 0.02184 (9)  |
|N3 | 0.47631 (10) | 0.51791 (16) | 0.42741 (5)  | 0.0199 (3)   |
|HN3| 0.4952 (13)  | 0.467 (2)    | 0.4491 (9)   | 0.020 (5)*   |
|N4 | 0.40214 (9)  | 0.69778 (16) | 0.39417 (5)  | 0.0173 (3)   |
HN4  0.3816 (15)  0.783 (3)  0.3921 (9)  0.028 (6)*
C4  0.43369 (10)  0.64323 (17)  0.43758 (6)  0.0150 (3)
C5  0.46475 (10)  0.47080 (17)  0.37446 (6)  0.0166 (3)
H5A  0.423029  0.388469  0.371861  0.020*
H5B  0.519445  0.439328  0.358855  0.020*
C6  0.43059 (11)  0.61337 (18)  0.34929 (6)  0.0180 (3)
H6A  0.476022  0.666661  0.330344  0.022*
H6B  0.382641  0.591836  0.325695  0.022*

Atomic displacement parameters (Å²)

|   | U¹¹   | U¹²   | U¹³   | U²²   | U²³   | U³³   |
|---|-------|-------|-------|-------|-------|-------|
| I1 | 0.01584 (5) | 0.01297 (5) | 0.01098 (4) | −0.00009 (3) | −0.00092 (3) | 0.00036 (3) |
| I2 | 0.01801 (5) | 0.01630 (5) | 0.01853 (5) | −0.00013 (4) | −0.00276 (4) | −0.00185 (4) |
| F1 | 0.0169 (4) | 0.0213 (5) | 0.0210 (5) | 0.0041 (4) | 0.0008 (4) | −0.0073 (4) |
| F2 | 0.0276 (5) | 0.0234 (5) | 0.0182 (5) | −0.0016 (4) | 0.0000 (4) | −0.0098 (4) |
| F3 | 0.0216 (5) | 0.0265 (5) | 0.0215 (5) | 0.0042 (4) | 0.0058 (4) | −0.0079 (4) |
| F4 | 0.0143 (4) | 0.0284 (5) | 0.0217 (5) | 0.0066 (4) | 0.0066 (4) | −0.0042 (4) |
| C7 | 0.0190 (7) | 0.0121 (6) | 0.0105 (6) | −0.0010 (5) | 0.0006 (5) | 0.0002 (5) |
| C8 | 0.0170 (7) | 0.0124 (6) | 0.0141 (6) | 0.0019 (5) | 0.0012 (5) | −0.0001 (5) |
| C9 | 0.0175 (7) | 0.0140 (7) | 0.0155 (7) | −0.0009 (5) | −0.0013 (5) | 0.0000 (5) |
| C10 | 0.0222 (8) | 0.0144 (7) | 0.0128 (6) | −0.0015 (6) | 0.0005 (6) | −0.0015 (5) |
| C11 | 0.0205 (7) | 0.0159 (7) | 0.0152 (7) | 0.0022 (6) | 0.0043 (6) | −0.0010 (6) |
| C12 | 0.0150 (7) | 0.0167 (7) | 0.0146 (6) | −0.0010 (6) | 0.0007 (5) | 0.0014 (5) |
| S1 | 0.01883 (17) | 0.01408 (16) | 0.01138 (15) | 0.00309 (13) | −0.00079 (13) | 0.00015 (13) |
| N1 | 0.0220 (7) | 0.0127 (6) | 0.0150 (6) | 0.0032 (5) | −0.0018 (5) | 0.0014 (5) |
| N2 | 0.0277 (7) | 0.0133 (6) | 0.0136 (6) | 0.0055 (5) | −0.0030 (5) | −0.0008 (5) |
| C1 | 0.0119 (6) | 0.0140 (6) | 0.0142 (6) | 0.0004 (5) | 0.0011 (5) | 0.0008 (5) |
| C2 | 0.0259 (8) | 0.0148 (7) | 0.0152 (7) | 0.0014 (6) | 0.0001 (6) | −0.0028 (6) |
| C3 | 0.0276 (8) | 0.0180 (7) | 0.0141 (7) | 0.0030 (6) | −0.0023 (6) | −0.0026 (6) |
| S2 | 0.0339 (2) | 0.01651 (18) | 0.01508 (17) | 0.01055 (16) | −0.00313 (16) | −0.00093 (14) |
| N3 | 0.0280 (7) | 0.0164 (6) | 0.0151 (6) | 0.0094 (6) | −0.0051 (5) | 0.0000 (5) |
| N4 | 0.0212 (7) | 0.0146 (6) | 0.0161 (6) | 0.0054 (5) | −0.0028 (5) | 0.0010 (5) |
| C4 | 0.0143 (7) | 0.0127 (6) | 0.0179 (7) | 0.0004 (5) | −0.0011 (5) | 0.0015 (5) |
| C5 | 0.0197 (7) | 0.0139 (7) | 0.0161 (7) | 0.0018 (6) | 0.0003 (6) | −0.0003 (5) |
| C6 | 0.0221 (8) | 0.0167 (7) | 0.0153 (7) | 0.0043 (6) | −0.0022 (6) | 0.0002 (6) |

Geometric parameters (Å, °)

|   |   |   |   |   |   |   |
|---|---|---|---|---|---|---|
| I1—I7 | 2.0969 (14) | N2—C3 | 1.462 (2) |
| I2—I9 | 2.0947 (16) | C2—H2A | 0.9900 |
| F1—C8 | 1.3442 (17) | C2—H2B | 0.9900 |
| F2—C10 | 1.3459 (17) | C2—C3 | 1.538 (2) |
| F3—C11 | 1.3445 (18) | C3—H3A | 0.9900 |
| F4—C12 | 1.3412 (18) | C3—H3B | 0.9900 |
| C7—C8 | 1.387 (2) | S2—C4 | 1.6995 (16) |
| C7—C12 | 1.385 (2) | N3—HN3 | 0.79 (2) |
| C8—C9 | 1.388 (2) | N3—C4 | 1.337 (2) |

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sup-3
| Bond                  | Length (Å) | Bond                  | Length (Å) |
|----------------------|------------|----------------------|------------|
| C9—C10               | 1.384 (2)  | N3—C5                | 1.455 (2)  |
| C10—C11              | 1.383 (2)  | N4—HN4               | 0.83 (2)   |
| C11—C12              | 1.382 (2)  | N4—C4                | 1.328 (2)  |
| S1—C1                | 1.7088 (15)| N4—C6                | 1.464 (2)  |
| N1—HN1               | 0.81 (2)   | C5—H5A               | 0.9900     |
| N1—C1                | 1.332 (2)  | C5—H5B               | 0.9900     |
| N1—C2                | 1.463 (2)  | C5—C6                | 1.536 (2)  |
| N2—HN2               | 0.83 (2)   | C6—H6A               | 0.9900     |
| N2—C1                | 1.3280 (19)| C6—H6B               | 0.9900     |
| C8—C7—I1             | 121.56 (11)| H2A—C2—H2B           | 109.1      |
| C12—C7—I1            | 120.94 (11)| C3—C2—H2A            | 111.2      |
| C12—C7—C8            | 117.43 (14)| C3—C2—H2B            | 111.2      |
| F1—C8—C7             | 118.31 (13)| N2—C3—C2             | 102.49 (12)|
| F1—C8—C9             | 118.39 (14)| N2—C3—H3A            | 111.3      |
| C7—C8—C9             | 123.29 (14)| N2—C3—H3B            | 111.3      |
| C8—C9—I2             | 122.05 (11)| C2—C3—H3A            | 111.3      |
| C10—C9—I2            | 121.04 (11)| C2—C3—H3B            | 111.3      |
| C10—C9—C8            | 116.90 (14)| H3A—C3—H3B           | 109.2      |
| F2—C10—C9            | 120.43 (14)| C4—N3—HN3            | 122.5 (16) |
| F2—C10—C11           | 117.74 (14)| C4—N3—C5             | 111.80 (13)|
| C11—C10—C9           | 121.83 (14)| C5—N3—HN3            | 124.0 (16) |
| F3—C11—C10           | 120.23 (14)| C4—N4—HN4            | 122.7 (16) |
| F3—C11—C12           | 120.52 (15)| C4—N4—C6             | 112.06 (13)|
| C12—C11—C10          | 119.23 (14)| C6—N4—HN4            | 122.8 (16) |
| F4—C12—C7            | 120.34 (14)| C3—C4—S2             | 125.10 (12)|
| F4—C12—C11           | 118.37 (14)| C4—C4—S2             | 125.73 (12)|
| C11—C12—C7           | 121.29 (15)| C4—C4—N3             | 109.17 (14)|
| C1—N1—HN1            | 119.7 (16) | N3—C5—H5A            | 111.4      |
| C1—N1—C2             | 112.04 (13)| N3—C5—H5B            | 111.4      |
| C2—N1—HN1            | 125.6 (16) | N3—C5—C6             | 101.85 (12)|
| C1—N2—HN2            | 121.3 (15) | H5A—C5—H5B           | 109.3      |
| C1—N2—C3             | 112.46 (13)| C6—C5—H5A            | 111.4      |
| C3—N2—HN2            | 125.8 (15) | C6—C5—H5B            | 111.4      |
| N1—C1—S1             | 125.81 (12)| N4—C6—C5             | 101.41 (12)|
| N2—C1—S1             | 124.18 (12)| N4—C6—H6A            | 111.5      |
| N2—C1—N1             | 110.01 (14)| N4—C6—H6B            | 111.5      |
| N1—C2—H2A            | 111.2      | C5—C6—H6A            | 111.5      |
| N1—C2—H2B            | 111.2      | C5—C6—H6B            | 111.5      |
| N1—C2—C3             | 102.68 (12)| H6A—C6—H6B           | 109.3      |

| Bond                  | Angle (°)  | Bond                  | Angle (°)  |
|----------------------|------------|----------------------|------------|
| I1—C7—C8—I1         | −3.75 (19) | C9—C10—C11—C12      | −1.9 (2)   |
| I1—C7—C8—C9         | 175.92 (12)| C10—C11—C12—F4      | −177.15 (14)|
| I1—C7—C12—F4        | 1.7 (2)    | C10—C11—C12—C7      | 1.9 (2)    |
| I1—C7—C12—C11       | −177.34 (12)| C12—C7—C8—F1       | 179.43 (13) |
| I2—C9—C10—F2        | 1.9 (2)    | C12—C7—C8—C9       | −0.9 (2)   |
| I2—C9—C10—C11       | −178.45 (12)| N1—C2—C3—N2      | 4.94 (17)   |
| F1—C8—C9—I2         | −0.4 (2)   | C1—N1—C2—C3        | −5.82 (18)  |
supporting information

F1—C8—C9—C10 −179.48 (14) C1—N2—C3—C2 −2.96 (19)
F2—C10—C11—F3 −0.8 (2) C2—N1—C1—S1 −174.62 (12)
F2—C10—C11—C12 177.76 (14) C2—N1—C1—N2 4.27 (19)
F3—C11—C12—F4 1.4 (2) C3—N2—C1—S1 178.31 (12)
F3—C11—C12—C7 −179.58 (14) C3—N2—C1—N1 −0.6 (2)
F3—C11—C12—C7 179.53 (14) C4—N3—C5—C6 16.94 (18)
F3—C11—C12—F4 178.51 (14) C4—N3—C5—C6 15.47 (18)
C7—C8—C9—I2 179.89 (11) C5—N3—C4—S2 172.13 (12)
C7—C8—C9—I2 −0.5 (2) C5—N3—C4—N4 −7.96 (19)
C8—C7—C12—F4 178.51 (14) C6—N4—C4—S2 174.32 (12)
C8—C7—C12—C11 −0.5 (2) C6—N4—C4—N3 −5.59 (19)
C8—C9—C10—F2 −179.09 (14) C6—N4—C4—N3 114.9 (18)
C8—C9—C10—C11 0.6 (2) C6—N4—C4—N3 −5.59 (19)
C9—C10—C11—F3 179.89 (14) C6—N4—C4—N3 114.9 (18)
C9—C10—C11—C12 177.76 (14) C6—N4—C4—N3 114.9 (18)
N3—C5—C6—N4 −18.18 (16)
C4—N3—C5—C6 16.94 (18)
C4—N3—C5—C6 15.47 (18)

Hydrogen-bond geometry (Å, º)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| N1—HN1···S1i | 0.81 (2) | 2.77 (2) | 3.5551 (14) | 163 (2) |
| N2—HN2···S2ii | 0.83 (2) | 2.53 (2) | 3.3507 (14) | 172 (2) |
| C2—H2···F2 | 0.99 | 2.55 | 3.3392 (19) | 136 |
| C3—H3···F2 | 0.99 | 2.94 | 3.7351 (19) | 138 |
| N3—H3···F2 | 0.79 (2) | 2.54 (2) | 3.3171 (15) | 167 (2) |
| N4—H4···F2 | 0.83 (2) | 3.31 (2) | 3.7383 (14) | 114.9 (18) |
| N4—H4···F2 | 0.83 (2) | 2.63 (2) | 3.4562 (14) | 179 (2) |
| C5—H5···F2 | 0.99 | 3.20 | 3.9922 (16) | 138 |
| C5—H5···F2 | 0.99 | 2.45 | 3.2774 (19) | 140 |
| C6—H6···F2 | 0.99 | 3.18 | 3.9223 (16) | 133 |

Symmetry codes: (i) −x+3/2, y−1/2, z; (ii) −x+1, −y+2, −z+1; (iii) −x+1, −y+1, −z+1; (iv) −x+3/2, y+1/2, z; (v) −x+1, y, −z+1/2; (vi) x−1/2, y+1/2, −z+1.

Imidazolidine-2-thione–1,3,5-trifluoro-2,4,6-triiodobenzene (1/1) (IT_135F3I3B)

Crystal data

| C₆F₃I₃·C₃H₆N₂S | D_s = 2.797 Mg m⁻³ |
|-----------------|-----------------|
| M_r = 611.92    | Mo Kα radiation, λ = 0.71073 Å |
| Orthorhombic, Pbca | Cell parameters from 9914 reflections |
| a = 18.0407 (14) Å | θ = 2.9–28.3° |
| b = 7.2816 (6) Å | µ = 6.61 mm⁻¹ |
| c = 22.1250 (19) Å | T = 100 K |
| V = 2906.5 (4) Å³ | Tabular, colourless |
| Z = 8 | 0.22 × 0.08 × 0.04 mm |
| F(000) = 2208 |

Data collection

Bruker D8 Venture Photon 2

3615 independent reflections
3220 reflections with I > 2σ(I)

Radiation source: Incoatec IμS
θ and ω scans
Absorption correction: multi-scan
(SADABS; Bruker, 2017)
T_min = 0.563, T_max = 0.746
49179 measured reflections
### Refinement

Refinement on $F^2$

Least-squares matrix: full

$R(F^2 > 2\sigma(F^2)) = 0.019$

$wR(F^2) = 0.040$

$S = 1.11$

3615 reflections

172 parameters

2 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma(F_c^2) + (0.0114P)^2 + 5.9439P]$

where $P = (F_c^2 + 2F_e^2)/3$

$(\Delta\sigma)_{\text{max}} = 0.002$

$\Delta\rho_{\text{max}} = 0.57$ e Å$^{-3}$

$\Delta\rho_{\text{min}} = -0.77$ e Å$^{-3}$

Extinction correction: SHELXL2018 (Sheldrick, 2015b),

$F_c^\ast=kF_c[1+0.001xF_c^2\lambda^3/sin(2\theta)]^{1/4}$

Extinction coefficient: 0.000108 (15)

### 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 (Å$^2$)

|   | x      | y      | z      | $U_{	ext{iso}}$/U$_{	ext{eq}}$ |
|---|--------|--------|--------|---------------------------------|
| I1| 0.53750 (2) | 0.23369 (3) | 0.54191 (2) | 0.01895 (5) |
| I2| 0.82382 (2) | 0.16514 (2) | 0.68496 (2) | 0.01507 (5) |
| I3| 0.83267 (2) | 0.32805 (2) | 0.41594 (2) | 0.01451 (5) |
| F1| 0.65074 (9)  | 0.1654 (2) | 0.64879 (8) | 0.0189 (4) |
| F2| 0.88045 (8)  | 0.2474 (2) | 0.55242 (8) | 0.0197 (4) |
| F3| 0.65981 (9)  | 0.3180 (2) | 0.44277 (8) | 0.0179 (3) |
| C4| 0.65271 (14) | 0.2418 (4) | 0.54544 (13) | 0.0139 (5) |
| C5| 0.69034 (15) | 0.2057 (4) | 0.59879 (13) | 0.0146 (5) |
| C6| 0.76726 (14) | 0.2079 (4) | 0.60287 (13) | 0.0135 (5) |
| C7| 0.80537 (14) | 0.2449 (4) | 0.54993 (13) | 0.0140 (5) |
| C8| 0.77196 (14) | 0.2814 (4) | 0.49545 (13) | 0.0133 (5) |
| C9| 0.69470 (14) | 0.2803 (4) | 0.49494 (13) | 0.0137 (5) |
| S1| 0.57097 (4)  | 0.61400 (10) | 0.79736 (3) | 0.01671 (14) |
| N1| 0.52694 (14) | 0.2877 (4) | 0.84174 (13) | 0.0228 (6) |
| HN1| 0.4953 (19)  | 0.345 (6) | 0.8617 (18) | 0.057 (14)* |
| N2| 0.62179 (16) | 0.2684 (4) | 0.78288 (13) | 0.0257 (6) |
| HN2| 0.6527 (16)  | 0.308 (5) | 0.7585 (14) | 0.029 (10)* |
| C1| 0.57324 (15) | 0.3832 (4) | 0.80740 (12) | 0.0161 (6) |
| C2| 0.54227 (16) | 0.0908 (4) | 0.84203 (14) | 0.0222 (6) |
| H2A| 0.498578  | 0.019283 | 0.828618 | 0.027* |
| H2B| 0.557562  | 0.048198 | 0.882651 | 0.027* |
| C3| 0.60613 (19) | 0.0758 (4) | 0.79654 (16) | 0.0277 (7) |
| H3A| 0.649751  | 0.014419 | 0.814660 | 0.033* |
| H3B| 0.590783  | 0.007958 | 0.759844 | 0.033* |
Atomic displacement parameters (Å²)

| Atom | U₁₁ | U₂₂ | U₃₃ | U₁₂ | U₁₃ | U₂₃ |
|------|-----|-----|-----|-----|-----|-----|
| I1   | 0.01058 (8) | 0.02130 (10) | 0.02496 (11) | 0.00013 (7) | 0.00038 (7) | −0.00336 (8) |
| I2   | 0.01700 (9) | 0.01397 (9) | 0.01424 (10) | −0.00039 (6) | −0.00190 (7) | 0.00115 (7) |
| I3   | 0.01480 (8) | 0.01468 (9) | 0.01405 (9) | 0.000001 (6) | 0.00204 (7) | 0.00044 (7) |
| F1   | 0.0176 (8) | 0.0235 (9) | 0.0157 (9) | −0.0029 (7) | 0.0045 (7) | 0.0016 (7) |
| F2   | 0.0101 (7) | 0.0284 (10) | 0.0205 (9) | −0.0005 (6) | −0.0003 (6) | 0.0023 (8) |
| F3   | 0.0170 (7) | 0.0204 (9) | 0.0163 (9) | 0.0018 (7) | −0.0034 (6) | 0.0024 (7) |
| C4   | 0.0098 (11) | 0.0127 (13) | 0.0193 (14) | −0.0006 (10) | −0.0004 (10) | −0.0018 (11) |
| C5   | 0.0172 (12) | 0.0111 (13) | 0.0155 (14) | −0.0027 (10) | 0.0038 (11) | −0.0007 (11) |
| C6   | 0.0161 (12) | 0.0118 (13) | 0.0124 (13) | 0.0014 (10) | −0.0043 (10) | −0.0012 (10) |
| C7   | 0.0118 (11) | 0.0110 (13) | 0.0191 (15) | 0.0014 (10) | −0.0010 (10) | 0.0000 (11) |
| C8   | 0.0141 (12) | 0.0111 (13) | 0.0148 (14) | 0.0003 (10) | 0.0033 (10) | −0.0016 (11) |
| C9   | 0.0137 (12) | 0.0120 (13) | 0.0153 (14) | 0.0024 (10) | −0.0036 (10) | −0.0001 (11) |
| S1   | 0.0211 (3) | 0.0144 (3) | 0.0146 (3) | 0.0003 (3) | 0.0002 (3) | 0.0002 (3) |
| N1   | 0.0236 (13) | 0.0181 (13) | 0.0267 (15) | −0.0027 (10) | 0.0051 (11) | 0.0020 (11) |
| N2   | 0.0321 (14) | 0.0158 (13) | 0.0292 (16) | 0.0020 (11) | 0.0127 (12) | 0.0046 (12) |
| C1   | 0.0168 (13) | 0.0223 (15) | 0.0092 (14) | −0.0006 (11) | −0.0037 (10) | −0.0001 (11) |
| C2   | 0.0244 (14) | 0.0209 (15) | 0.0213 (16) | −0.0062 (12) | −0.0049 (12) | 0.0046 (13) |
| C3   | 0.0397 (18) | 0.0155 (15) | 0.0278 (18) | 0.0006 (14) | 0.0044 (15) | 0.0037 (13) |

Geometric parameters (Å, °)

| Bond          | Length (Å) | Angle (°) |
|---------------|------------|-----------|
| I1—C4         | 2.081 (2)  | S1—C1     | 1.696 (3) |
| I2—C6         | 2.106 (3)  | N1—HN1    | 0.831 (19) |
| I3—C8         | 2.100 (3)  | N1—C1     | 1.326 (4) |
| F1—C5         | 1.349 (3)  | N1—C2     | 1.460 (4) |
| F2—C7         | 1.356 (3)  | N2—HN2    | 0.827 (18) |
| F3—C9         | 1.343 (3)  | N2—C1     | 1.327 (4) |
| C4—C5         | 1.387 (4)  | N2—C3     | 1.462 (4) |
| C4—C9         | 1.379 (4)  | C2—H2A    | 0.9900 |
| C5—C6         | 1.391 (4)  | C2—H2B    | 0.9900 |
| C6—C7         | 1.385 (4)  | C2—C3     | 1.534 (4) |
| C7—C8         | 1.374 (4)  | C3—H3A    | 0.9900 |
| C8—C9         | 1.394 (4)  | C3—H3B    | 0.9900 |
| C5—C4—I1      | 121.0 (2)  | C2—N1—HN1 | 128 (3) |
| C9—C4—I1      | 121.6 (2)  | C1—N2—HN2 | 119 (3) |
| C9—C4—C5      | 117.4 (2)  | C1—N2—C3 | 113.1 (3) |
| F1—C5—C4      | 118.7 (2)  | C3—N2—HN2 | 127 (3) |
| F1—C5—C6      | 118.5 (3)  | N1—C1—S1 | 125.4 (2) |
| C4—C5—C6      | 122.8 (3)  | N1—C1—N2 | 108.7 (3) |
| C5—C6—I2      | 122.5 (2)  | N2—C1—S1 | 125.9 (2) |
| C7—C6—I2      | 121.18 (19)| N1—C2—H2A | 111.4 |
| C7—C6—C5      | 116.3 (2)  | N1—C2—H2B | 111.4 |
| F2—C7—C6      | 117.7 (2)  | N1—C2—C3 | 102.1 (2) |
| F2—C7—C8      | 118.1 (2)  | H2A—C2—H2B | 109.2 |
C8—C7—C6 124.2 (2)  C3—C2—H2A 111.4
C7—C8—I3 122.51 (19)  C3—C2—H2B 111.4
C7—C8—C9 116.4 (3)  N2—C3—C2 102.3 (3)
C9—C8—I3 121.0 (2)  N2—C3—H3A 111.3
F3—C9—C4 118.7 (2)  N2—C3—H3B 111.3
F3—C9—C8 118.3 (2)  C2—C3—H3A 111.3
C4—C9—C8 122.9 (3)  C2—C3—H3B 111.3
C1—N1—HN1 118 (3)  H3A—C3—H3B 109.2
C1—N1—C2 113.5 (3)

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| N2···H2N2···I2’ | 0.83 (2) | 3.10 (3) | 3.742 (3) | 137 (3) |
| C2···H2B···I1” | 0.99 | 3.31 | 3.927 (3) | 122 |
| C2···H2B···F3”” | 0.99 | 2.47 | 3.147 (3) | 125 |

Symmetry codes: (i) −x+3/2, y+1/2, z; (ii) −x+1, y−1/2, −z+3/2; (iii) x, −y+1/2, z+1/2.

1H-1,3-Benzodiazole-2-thiol-1,2,3,5-tetrafluoro-4,6-diiodobenzene (4/3) (4MBZIM_313F4DIB)

Crystal data

3C6F4I2·4C7H6N2S  
Z = 2  
F(000) = 1692  
D(0) = 2.238 Mg m−3  
Mo Kα radiation, λ = 0.71073 Å

Cell parameters from 9879 reflections

θ = 2.4–27.5°  
µ = 3.72 mm−1  
T = 100 K  
Needle, colourless

V = 2680.9 (9) Å³

Acta Cryst. (2022), C78, 702-715
**Data collection**

Bruker D8 Venture Photon 2
diffractometer
Radiation source: Incoatec IμS
φ and ω scans
Absorption correction: multi-scan
(SADABS; Bruker, 2017)

118524 measured reflections
12297 independent reflections
10558 reflections with I > 2σ(I)

θ max = 27.6°, θ min = 2.2°

Refinement

Refinement on F^2
Least-squares matrix: full

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

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

| x       | y       | z       | Uiso*/Ueq |
|---------|---------|---------|-----------|
| I1      | 0.23723 (2) | 0.28696 (2) | 0.64287 (2) | 0.01892 (4) |
| I2      | 0.11203 (2) | 0.62311 (2) | 0.81696 (2) | 0.02185 (4) |
| F29     | 0.12514 (17) | 0.43584 (9)  | 0.76730 (8)  | 0.0228 (3)  |
| F30     | 0.28968 (19) | 0.64658 (9)  | 0.67800 (9)  | 0.0259 (3)  |
| F31     | 0.41295 (19) | 0.54429 (9)  | 0.56177 (9)  | 0.0271 (4)  |
| F32     | 0.38986 (17) | 0.38762 (9)  | 0.54469 (8)  | 0.0234 (3)  |
| F33     | 0.2585 (3)  | 0.40830 (15) | 0.65651 (14) | 0.0161 (5)  |
| C30     | 0.1965 (3)  | 0.46280 (16) | 0.71636 (14) | 0.0182 (5)  |
| C31     | 0.2045 (3)  | 0.54314 (15) | 0.72577 (14) | 0.0166 (5)  |
| C32     | 0.2787 (3)  | 0.56938 (15) | 0.67263 (14) | 0.0186 (5)  |
| C33     | 0.3417 (3)  | 0.51713 (16) | 0.61237 (14) | 0.0193 (5)  |
| C34     | 0.3307 (3)  | 0.43733 (16) | 0.60458 (14) | 0.0183 (5)  |
| I3      | 0.39366 (2) | 0.29864 (2)  | 0.15711 (2)  | 0.02594 (4) |
| I4      | 0.19057 (2) | 0.58106 (2)  | 0.39910 (2)  | 0.02628 (4) |
| F33     | 0.31982 (19) | 0.47479 (9)  | 0.24734 (9)  | 0.0278 (4)  |
| F34     | 0.11956 (18) | 0.43808 (10) | 0.46524 (9)  | 0.0290 (4)  |
| F35     | 0.17740 (18) | 0.28360 (10) | 0.41706 (9)  | 0.0295 (4)  |
| F36     | 0.30202 (17) | 0.22313 (9)  | 0.28451 (9)  | 0.0256 (3)  |
| C35     | 0.2209 (3)  | 0.46038 (15) | 0.35750 (15) | 0.0190 (5)  |
| C36     | 0.2842 (3)  | 0.42746 (16) | 0.28917 (14) | 0.0191 (5)  |
| C37     | 0.3114 (3)  | 0.34781 (15) | 0.26241 (14) | 0.0178 (5)  |
| Atom | U1   | U2   | U3   | U12  |
|------|------|------|------|------|
| C38  | 0.2759 (3) | 0.30052 (15) | 0.30711 (15) | 0.0192 (5) |
| C39  | 0.2115 (3) | 0.33102 (16) | 0.37517 (15) | 0.0210 (6) |
| C40  | 0.1836 (3) | 0.40999 (16) | 0.39927 (14) | 0.0200 (5) |
| I5   | 0.82526 (2) | 0.39902 (2) | −0.11345 (2) | 0.02532 (4) |
| I6   | 0.75419 (2) | 0.73973 (2) | 0.07191 (2) | 0.01718 (4) |
| F37  | 0.85766 (18) | 0.58847 (9) | −0.05559 (8) | 0.0244 (3) |
| F38  | 0.59562 (17) | 0.64129 (9) | 0.16783 (8) | 0.0218 (3) |
| F39  | 0.54190 (18) | 0.48581 (9) | 0.14581 (9) | 0.0262 (4) |
| F40  | 0.63996 (18) | 0.38040 (9) | 0.02360 (9) | 0.0255 (3) |
| C41  | 0.7536 (3) | 0.48240 (15) | −0.01841 (14) | 0.0169 (5) |
| C42  | 0.7782 (3) | 0.56261 (15) | −0.00623 (14) | 0.0164 (5) |
| C43  | 0.7249 (3) | 0.61822 (14) | 0.05532 (14) | 0.0149 (5) |
| C44  | 0.6468 (3) | 0.59054 (15) | 0.10618 (14) | 0.0168 (5) |
| C45  | 0.6198 (3) | 0.51085 (15) | 0.09575 (14) | 0.0180 (5) |
| C46  | 0.6721 (3) | 0.45764 (15) | 0.03328 (15) | 0.0182 (5) |
| S1   | 1.26980 (7) | 0.02971 (4) | 0.13364 (4) | 0.01658 (13) |
| N1   | 1.3937 (2) | −0.08314 (13) | 0.19002 (11) | 0.0155 (4) |
| HN1  | 1.487 (2) | −0.0747 (18) | 0.1788 (17) | 0.031 (9)* |
| N2   | 1.1363 (2) | −0.08180 (12) | 0.18912 (11) | 0.0153 (4) |
| HN2  | 1.041 (2) | −0.0708 (15) | 0.1806 (14) | 0.015 (7)* |
| C1   | 1.2668 (3) | −0.04645 (15) | 0.17100 (13) | 0.0158 (5) |
| C2   | 1.3451 (3) | −0.14290 (15) | 0.21949 (13) | 0.0154 (5) |
| C3   | 1.4285 (3) | −0.19430 (15) | 0.24861 (14) | 0.0188 (5) |
| H3   | 1.540814 | −0.194912 | 0.248974 | 0.023* |
| C4   | 1.3406 (3) | −0.24474 (16) | 0.27716 (14) | 0.0208 (6) |
| H4   | 1.393923 | −0.280671 | 0.297743 | 0.025* |
| C5   | 1.1748 (3) | −0.24408 (16) | 0.27645 (14) | 0.0197 (5) |
| H5   | 1.118578 | −0.279849 | 0.296263 | 0.024* |
| C6   | 1.0905 (3) | −0.19247 (15) | 0.24751 (14) | 0.0182 (5) |
| H6   | 0.978144 | −0.191915 | 0.247143 | 0.022* |
| C7   | 1.1792 (3) | −0.14174 (15) | 0.21913 (13) | 0.0159 (5) |
| S2   | 0.76230 (7) | −0.07080 (4) | 0.13517 (3) | 0.01460 (12) |
| N3   | 0.8902 (2) | 0.04164 (12) | 0.07893 (12) | 0.0142 (4) |
| HN3  | 0.985 (2) | 0.0339 (17) | 0.0913 (16) | 0.026 (8)* |
| N4   | 0.6315 (2) | 0.03610 (12) | 0.07390 (11) | 0.0134 (4) |
| HN4  | 0.539 (2) | 0.0257 (15) | 0.0851 (15) | 0.016 (7)* |
| C8   | 0.7619 (3) | 0.00336 (14) | 0.09496 (13) | 0.0135 (5) |
| C9   | 0.8425 (3) | 0.10176 (14) | 0.04983 (13) | 0.0149 (5) |
| C10  | 0.9275 (3) | 0.15703 (15) | 0.02577 (14) | 0.0186 (5) |
| H10  | 1.040174 | 0.160378 | 0.028830 | 0.022* |
| C11  | 0.8386 (3) | 0.20721 (15) | −0.00307 (15) | 0.0199 (5) |
| H11  | 0.892259 | 0.246317 | −0.019649 | 0.024* |
| C12  | 0.6733 (3) | 0.20197 (15) | −0.00842 (14) | 0.0185 (5) |
| H12  | 0.617475 | 0.236637 | −0.029681 | 0.022* |
| C13  | 0.5882 (3) | 0.14726 (15) | 0.01657 (13) | 0.0160 (5) |
| H13  | 0.475508 | 0.143780 | 0.013282 | 0.019* |
| C14  | 0.6767 (3) | 0.09798 (14) | 0.04661 (13) | 0.0137 (5) |
| S3   | 0.21077 (7) | 1.09011 (4) | 0.59658 (4) | 0.01674 (13) |
N5  0.3335 (2)  0.98621 (12)  0.66496 (12)  0.0153 (4)
HN5  0.427 (2)  0.9957 (18)  0.6541 (17)  0.035 (9)^
N6  0.0755 (2)  0.98679 (12)  0.66091 (12)  0.0149 (4)
HN6 −0.020 (2)  0.9958 (16)  0.6501 (15)  0.021 (8)^
C15  0.2063 (3)  1.02032 (14)  0.64225 (13)  0.0147 (5)
C16  0.2825 (3)  0.92908 (14)  0.69697 (13)  0.0149 (5)
C17  0.3645 (3)  0.87884 (15)  0.72898 (14)  0.0196 (5)
H17  0.477019  0.878891  0.731657  0.024*
C18  0.2730 (3)  0.82880 (15)  0.75670 (14)  0.0211 (6)
H18  0.324318  0.793463  0.772293  0.026*
C19  0.1078 (3)  0.82884 (15)  0.75312 (14)  0.0213 (6)
H19  0.049812  0.793080  0.772293  0.026*
C20  0.0256 (3)  0.87954 (15)  0.72420 (14)  0.0186 (5)
C22 −0.29930 (7)  0.99445 (4)  0.61172 (3)  0.01608 (12)
N7 −0.1672 (2)  1.09905 (13)  0.54729 (12)  0.0157 (4)
HN7 −0.073 (2)  1.0901 (18)  0.5580 (17)  0.033 (9)^
N8 −0.4249 (2)  1.09692 (13)  0.54319 (12)  0.0156 (4)
HN8 −0.519 (2)  1.0848 (17)  0.5511 (17)  0.029 (8)^
C22 −0.2972 (3)  1.06481 (14)  0.56708 (13)  0.0146 (5)
C23 −0.2121 (3)  1.15416 (14)  0.51139 (13)  0.0145 (5)
C24 −0.1251 (3)  1.20294 (15)  0.48049 (14)  0.0186 (5)
H24 −0.012656  1.203249  0.481615  0.022*
C25 −0.2099 (3)  1.25129 (15)  0.44782 (14)  0.0189 (5)
H25 −0.154262  1.285731  0.426292  0.023*
C26 −0.3758 (3)  1.25043 (15)  0.44591 (14)  0.0189 (5)
H26 −0.429872  1.284470  0.423194  0.023*
C27 −0.4633 (3)  1.20121 (15)  0.47629 (14)  0.0185 (5)
H27 −0.575825  1.200557  0.474834  0.022*
C28 −0.3778 (3)  1.15290 (14)  0.50896 (13)  0.0147 (5)

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### Atomic displacement parameters (Å²)

|     | U₁₁  | U₂₂  | U₃₃  | U₁₂  | U₁₃  | U₂₃  |
|-----|------|------|------|------|------|------|
| I1  | 0.01909 (8) | 0.01702 (9) | 0.02029 (9) | 0.00047 (6) | −0.00052 (6) | 0.00538 (7) |
| I2  | 0.02488 (9) | 0.02320 (9) | 0.01763 (9) | 0.00734 (7) | 0.00435 (7) | 0.00505 (7) |
| F29 | 0.0267 (8) | 0.0243 (9) | 0.0207 (8) | 0.0015 (6) | 0.0077 (6) | 0.0105 (7) |
| F30 | 0.0389 (9) | 0.0165 (8) | 0.0242 (9) | 0.0008 (7) | 0.0055 (7) | 0.0085 (7) |
| F31 | 0.0365 (9) | 0.0267 (9) | 0.0217 (8) | −0.0001 (7) | 0.0121 (7) | 0.0112 (7) |
| F32 | 0.0259 (8) | 0.0241 (9) | 0.0192 (8) | 0.0051 (6) | 0.0067 (6) | 0.0034 (7) |
| C29 | 0.0157 (12) | 0.0150 (13) | 0.0176 (13) | 0.0003 (9) | −0.0012 (9) | 0.0052 (11) |
| C30 | 0.0148 (12) | 0.0233 (14) | 0.0179 (13) | 0.0010 (10) | 0.0011 (10) | 0.0080 (11) |
| C31 | 0.0165 (12) | 0.0192 (14) | 0.0134 (12) | 0.0051 (10) | 0.0015 (9) | 0.0035 (10) |
| C32 | 0.0210 (13) | 0.0174 (14) | 0.0185 (13) | 0.0012 (10) | −0.0005 (10) | 0.0074 (11) |
| C33 | 0.0193 (13) | 0.0244 (15) | 0.0167 (13) | 0.0000 (10) | 0.0034 (10) | 0.0098 (11) |
| C34 | 0.0151 (12) | 0.0228 (14) | 0.0154 (13) | 0.0019 (10) | 0.0009 (9) | 0.0030 (11) |
| I3  | 0.03176 (10) | 0.02680 (10) | 0.01832 (9) | 0.00506 (7) | 0.00720 (7) | 0.00377 (8) |
|    | I4      | F33     | F34     | F35     | F36     | C35     | C36     | C37     | C38     | C39     | I5      | I6      | F37     | F38     | F39     | F40     | C41     | C42     | C43     | C44     | C45     | C46     | S1      | N1      | N2      | C1      | C2      | C3      | C4      | C5      | C6      | C7      | S2      | N3      | N4      | C8      | C9      | C10     | C11     | C12     | C13     | C14     | S3      | N5      | N6      | C15     | C16     |
|----|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
|    | 0.02547 (9) | 0.01905 (9) | 0.02967 (10) | 0.00413 (7) | −0.00068 (7) | 0.00011 (8) | 0.0086 (7) | 0.00127 (7) | 0.0040 (7) | 0.0040 (7) | 0.0029 (11) | 0.0090 (11) | 0.0048 (11) | 0.0092 (7) | 0.0039 (7) | 0.0158 (7) | 0.0066 (7) | 0.0011 (10) | 0.0064 (11) | 0.0049 (10) | 0.0041 (11) | 0.0090 (11) | 0.0050 (11) | 0.0057 (3) | 0.0037 (9) | 0.0057 (9) | 0.0000 (9) | 0.0004 (10) | 0.0029 (11) | 0.0052 (11) | 0.0026 (11) | 0.0019 (11) | 0.0029 (10) | 0.0030 (10) | 0.0054 (3) | 0.0061 (9) | 0.0036 (9) | 0.0004 (10) | 0.0022 (10) | 0.0000 (10) | 0.0024 (2) | 0.0003 (8) | 0.0039 (9) | 0.0043 (9) | 0.0015 (10) | 0.0005 (10) |
|    | C17  | C18  | C19  | C20  | C21  | S4   | N7   | N8   | C22  | C23  | C24  | C25  | C26  | C27  | C28  |
|----|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|
|    | 0.0189 (13) | 0.0306 (14) | 0.0400 (14) | 0.0206 (13) | 0.0158 (12) | 0.0130 (3) | 0.0114 (10) | 0.0111 (10) | 0.0146 (12) | 0.0166 (12) | 0.0182 (12) | 0.0254 (13) | 0.0242 (13) | 0.0164 (12) | 0.0162 (12) |
|    | 0.0204 (14) | 0.0167 (14) | 0.0166 (14) | 0.0167 (13) | 0.0137 (12) | 0.0184 (3) | 0.0195 (12) | 0.0199 (12) | 0.0152 (13) | 0.0127 (12) | 0.0192 (14) | 0.0141 (13) | 0.0166 (13) | 0.0191 (14) | 0.0134 (12) |
|    | 0.0163 (13) | 0.0141 (13) | 0.0179 (14) | 0.0165 (13) | 0.0108 (12) | 0.0182 (3) | 0.0159 (11) | 0.0168 (11) | 0.0119 (12) | 0.0110 (12) | 0.0164 (13) | 0.0169 (13) | 0.0153 (13) | 0.0187 (13) | 0.0143 (12) |
|    | 0.0065 (10) | 0.0056 (11) | -0.0005 (11) | -0.0009 (10) | 0.0019 (9)  | -0.0002 (2) | -0.0007 (8) | -0.0001 (8) | 0.0007 (9)  | 0.0000 (9)  | -0.0050 (10) | -0.0022 (10) | 0.0017 (10) | 0.0018 (10) | -0.0012 (9) |
|    | -0.0022 (10) | -0.0041 (10) | 0.0020 (11)  | 0.0015 (10)  | -0.0008 (9) | 0.0001 (2)  | -0.0008 (8) | 0.0016 (8)  | 0.0003 (11) | 0.0001 (9)  | 0.0018 (10)  | 0.0030 (10)  | 0.0009 (10) | 0.0005 (10) | 0.0016 (9)  |
|    | 0.0003 (11) | 0.0022 (11)  | 0.0063 (11)  | 0.0020 (11)  | 0.0007 (10) | 0.0078 (3)  | 0.0054 (9)  | 0.0070 (9)  | 0.0037 (10) | 0.0011 (10) | 0.0028 (11)  | 0.0044 (11)  | 0.0037 (11) | 0.0035 (11) | 0.0037 (10) |

Geometric parameters (Å, º)

|    | I1—C29 | C6—C7 | C29—C30 | C29—C31 | C29—C32 | C29—C33 | C29—C34 | C29—C35 | C29—C36 | C29—C37 | C29—C38 | C29—C39 | C29—C40 | C29—C41 | C29—C42 |
|----|--------|-------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
|    | 2.090 (3) | 1.390 (3) | 1.348 (3) | 1.344 (3) | 1.343 (3) | 1.382 (4) | 1.378 (4) | 1.344 (3) | 1.343 (3) | 1.342 (3) | 1.342 (3) | 1.390 (4) | 1.389 (4) | 1.386 (4) | 1.385 (4) |
|    | 2.088 (2) | 1.696 (2) | 0.846 (17) | 1.354 (3) | 0.849 (16) | 1.356 (3) | 1.390 (3) | 1.391 (4) | 0.9500  | 1.394 (3) | 1.388 (3) | 1.699 (2) | 0.850 (17) | 1.360 (3) | 1.388 (3) |
|    | 1.348 (3) | 1.344 (3) | 1.343 (3) | 1.344 (3) | 1.342 (3) | 1.390 (4) | 1.389 (4) | 1.386 (4) | 1.385 (4) | 1.383 (4) | 1.373 (4) | 1.347 (3) | 1.345 (3) | 1.344 (3) | 1.343 (3) |
|    | 2.090 (3) | 2.088 (2) | 1.342 (3) | 1.342 (3) | 1.342 (3) | 1.390 (4) | 1.389 (4) | 1.386 (4) | 1.385 (4) | 1.383 (4) | 1.373 (4) | 1.347 (3) | 1.345 (3) | 1.344 (3) | 1.343 (3) |
| Bond | Distance (Å) | Bond | Distance (Å) | Bond | Distance (Å) |
|------|-------------|------|-------------|------|-------------|
| C41—C42 | 1.379 (4) | C18—C19 | 1.396 (4) | C41—C46 | 1.382 (4) | C19—H19 | 0.9500 |
| C42—C43 | 1.390 (3) | C19—C20 | 1.387 (3) | C43—C44 | 1.382 (3) | C20—H20 | 0.9500 |
| C44—C45 | 1.377 (4) | C20—C21 | 1.386 (3) | C45—C46 | 1.378 (4) | S4—C22 | 1.693 (2) |
| S1—C1 | 1.693 (3) | N7—HN7 | 0.844 (17) | N1—HN1 | 0.847 (17) | N7—C22 | 1.358 (3) |
| C41—C46 | 1.382 (4) | C19—C20 | 1.387 (3) | C18—C19 | 1.396 (4) | C19—C20 | 1.387 (3) |
| C42—C43 | 1.390 (3) | C19—C20 | 1.387 (3) | C41—C46 | 1.378 (4) | S4—C22 | 1.693 (2) |
| S1—C1 | 1.693 (3) | N7—HN7 | 0.844 (17) | N1—HN1 | 0.847 (17) | N7—C22 | 1.358 (3) |
| C41—C42 | 1.379 (4) | C18—C19 | 1.396 (4) | C41—C46 | 1.382 (4) | C19—H19 | 0.9500 |
| C42—C43 | 1.390 (3) | C19—C20 | 1.387 (3) | C43—C44 | 1.382 (3) | C20—H20 | 0.9500 |
| C44—C45 | 1.377 (4) | C20—C21 | 1.386 (3) | C45—C46 | 1.378 (4) | S4—C22 | 1.693 (2) |
| S1—C1 | 1.693 (3) | N7—HN7 | 0.844 (17) | N1—HN1 | 0.847 (17) | N7—C22 | 1.358 (3) |

*Acta Cryst. (2022), C78, 702-715*
| Bond          | Angle (°) | Bond          | Angle (°) |
|---------------|-----------|---------------|-----------|
| C38—C37—I3    | 120.42 (19)| C13—C14—N4   | 131.5 (2) |
| C38—C37—C36  | 117.6 (2)  | C13—C14—C9   | 121.9 (2) |
| F36—C38—C37  | 120.2 (2)  | C15—N5—HN5   | 122 (2)   |
| F36—C38—C39  | 118.4 (2)  | C15—N5—C16   | 109.8 (2) |
| C39—C38—C37  | 121.4 (2)  | C16—N5—HN5   | 128 (2)   |
| F35—C39—C38  | 120.0 (2)  | C15—N6—HN6   | 125.4 (19)|
| F35—C39—C40  | 120.6 (2)  | C15—N6—C21   | 110.28 (19)|
| C40—C39—C38  | 119.4 (2)  | C21—N6—HN6   | 124.0 (19)|
| F34—C40—C35  | 120.2 (2)  | N5—C15—S3    | 126.48 (18)|
| F34—C40—C39  | 118.3 (2)  | N6—C15—S3    | 126.32 (18)|
| C39—C40—C35  | 121.5 (2)  | N6—C15—N5    | 107.2 (2) |
| C42—C41—I5   | 122.49 (18)| N5—C16—C17   | 132.2 (2) |
| C42—C41—C46  | 117.7 (2)  | N5—C16—C21   | 106.5 (2) |
| C46—C41—I5   | 119.79 (19)| C17—C16—C21  | 121.2 (2) |
| F37—C42—C41  | 119.0 (2)  | C16—C17—H17  | 121.8     |
| F37—C42—C43  | 118.4 (2)  | C18—C17—C16  | 116.4 (2) |
| C41—C42—C43  | 122.6 (2)  | C18—C17—H17  | 121.8     |
| C42—C43—C16  | 127.8 (18)| C17—C18—H18  | 119.1     |
| C44—C43—C16  | 119.64 (18)| C17—C18—C19  | 121.9 (2) |
| C44—C43—C42  | 117.6 (2)  | C19—C18—H18  | 119.1     |
| C46—C41—I5   | 119.79 (19)| C17—C16—C21  | 121.2 (2) |
| F39—C45—C44  | 120.0 (2)  | C19—C20—H20  | 122.0     |
| F39—C45—C46  | 120.8 (2)  | C21—C20—C19  | 116.1 (2) |
| C44—C45—C46  | 119.3 (2)  | C21—C20—H20  | 122.0     |
| F40—C46—C41  | 120.7 (2)  | N6—C21—C16   | 106.2 (2) |
| F40—C46—C45  | 117.8 (2)  | C20—C21—N6   | 131.4 (2) |
| C45—C46—C41  | 121.5 (2)  | C20—C21—C16  | 122.4 (2) |
| C1—N1—HN1    | 123 (2)    | C22—N7—H7    | 124 (2)   |
| C1—N1—C2     | 110.7 (2)  | C22—N7—C23   | 110.29 (19)|
| C2—N1—HN1    | 126 (2)    | C23—N7—H7    | 125 (2)   |
| C1—N2—HN2    | 124.7 (18)| C22—N8—H8    | 122 (2)   |
| C1—N2—C7     | 110.48 (19)| C22—N8—C28   | 110.6 (2) |
| C7—N2—HN2    | 124.7 (18)| C28—N8—H8    | 127 (2)   |
| N1—C1—S1     | 127.03 (19)| N7—C22—S4    | 126.72 (18)|
| N1—C1—N2     | 106.5 (2)  | N8—C22—S4    | 126.58 (18)|
| N2—C1—S1     | 126.41 (18)| N8—C22—N7    | 106.7 (2) |
| N1—C2—C7     | 106.0 (2)  | N7—C23—C28   | 106.2 (2) |
| C3—C2—N1     | 132.4 (2)  | C24—C23—N7   | 132.1 (2) |
| C3—C2—C7     | 121.6 (2)  | C24—C23—C28  | 121.6 (2) |
| C2—C3—H3     | 121.5      | C23—C24—H24  | 121.5     |
| C4—C3—C2     | 116.9 (2)  | C23—C24—C25  | 116.9 (2) |
| C4—C3—H3     | 121.5      | C25—C24—H24  | 121.5     |
| C3—C4—H4     | 119.2      | C24—C25—H25  | 119.3     |
| C3—C4—C5     | 121.6 (2)  | C24—C25—C26  | 121.4 (2) |
| C5—C4—H4     | 119.2      | C26—C25—H25  | 119.3     |
| C4—C5—H5     | 119.1      | C25—C26—H26  | 119.1     |
| Bond                  | Angle (°) ± Standard Error (°) | Bond                  | Angle (°) ± Standard Error (°) | Bond                  | Angle (°) ± Standard Error (°) |
|-----------------------|--------------------------------|-----------------------|--------------------------------|-----------------------|--------------------------------|
| C6—C5—C4             | 121.8 (2)                      | C27—C26—C25          | 121.8 (2)                      |
| C6—C5—H5             | 119.1                          | C27—C26—H26          | 119.1                          |
| C5—C6—H6             | 121.8                          | C26—C27—H27          | 121.7                          |
| C7—C6—C5             | 116.5 (2)                      | C26—C27—C28          | 116.5 (2)                      |
| C7—C6—H6             | 121.8                          | C28—C27—H27          | 121.7                          |
| N2—C7—C2             | 106.3 (2)                      | N8—C28—C23           | 106.2 (2)                      |
| N2—C7—C6             | 132.0 (2)                      | N8—C28—C27           | 132.1 (2)                      |
| C6—C7—C2             | 121.6 (2)                      | C27—C28—C23          | 121.7                          |
| I1—C29—C30—F29       | 1.8 (3)                        | N1—C2—C7—C6         | −177.7 (2)                     |
| I1—C29—C30—C31       | −178.02 (18)                   | C1—N1—C2—C3         | −177.6 (3)                     |
| I1—C29—C34—F32       | −0.7 (3)                       | C1—N1—C2—C7         | −0.7 (3)                       |
| I1—C29—C34—C33       | 178.45 (19)                    | C1—N2—C7—C2         | 0.1 (3)                        |
| I2—C31—C32—F30       | −1.4 (3)                       | C1—N2—C7—C6         | 177.9 (3)                      |
| I2—C31—C32—C33       | 179.38 (19)                    | C2—N1—C1—S1         | 179.67 (19)                    |
| F29—C30—C31—I2       | 0.8 (3)                        | C2—N1—C1—N2         | 0.8 (3)                        |
| F29—C30—C31—C32      | 179.9 (2)                      | C2—C3—C4—C5         | 0.2 (4)                        |
| F30—C32—C33—F31      | 0.7 (4)                        | C3—C2—C7—N2         | 177.6 (2)                      |
| F30—C32—C33—C34      | −179.2 (2)                     | C3—C2—C7—C6         | −0.5 (4)                       |
| F31—C33—C34—F32      | −1.3 (4)                       | C3—C4—C5—C6         | −0.4 (4)                       |
| F31—C33—C34—C29      | 179.6 (2)                      | C4—C5—C6—C7         | 0.2 (4)                        |
| C29—C30—C31—I2       | −179.37 (18)                   | C5—C6—C7—N2         | −177.3 (3)                     |
| C29—C30—C31—C32      | −0.3 (4)                       | C5—C6—C7—C2         | 0.2 (4)                        |
| C30—C29—C30—C32      | −178.6 (2)                     | C7—N2—C1—S1         | −179.44 (19)                   |
| C30—C29—C30—C33      | 0.5 (4)                        | C7—N2—C1—N1         | −0.6 (3)                       |
| C30—C31—C32—F30      | 179.5 (2)                      | C7—C2—C3—C4         | 0.2 (4)                        |
| C30—C31—C32—C33      | 0.3 (4)                        | N3—C9—C10—C11       | 177.7 (3)                      |
| C31—C32—C33—F31      | 180.0 (2)                      | N3—C9—C14—N4        | 0.1 (3)                        |
| C31—C32—C33—C34      | 0.1 (4)                        | N3—C9—C14—C13       | −176.7 (2)                     |
| C32—C33—C34—F32      | 178.6 (2)                      | C8—N3—C9—C10        | 179.5 (3)                      |
| C32—C33—C34—C29      | −0.5 (4)                       | C8—N3—C9—C14        | −1.5 (3)                       |
| C34—C29—C30—F29      | 179.7 (2)                      | C8—N4—C14—C9        | 1.3 (3)                        |
| C34—C29—C30—C31      | −0.1 (4)                       | C8—N4—C14—C13       | 177.6 (3)                      |
| I3—C37—C38—F36       | 3.5 (3)                        | C9—N3—C8—S2         | −176.81 (19)                   |
| I3—C37—C38—C39       | −175.80 (19)                   | C9—N3—C8—N4         | 2.3 (3)                        |
| I4—C35—C36—F33       | −3.1 (3)                       | C9—C10—C11—C12      | −0.7 (4)                       |
| I4—C35—C36—C37       | 177.10 (19)                    | C10—C9—C14—N4       | 179.3 (2)                      |
| I4—C35—C40—F34       | 3.5 (3)                        | C10—C9—C14—C13      | 2.5 (4)                        |
| I4—C35—C40—C39       | −175.96 (19)                   | C10—C11—C12—C13     | 1.6 (4)                        |
| F33—C36—C37—I3       | −3.5 (3)                       | C11—C12—C13—C14     | −0.4 (4)                       |
| F33—C36—C37—C38      | 179.1 (2)                      | C12—C13—C14—N4      | −177.5 (2)                     |
| F35—C39—C40—F34      | −0.8 (4)                       | C12—C13—C14—C9      | −1.6 (4)                       |
| F35—C39—C40—C35      | 178.7 (2)                      | C14—N4—C8—S2        | 176.89 (18)                     |
| F36—C38—C39—F35      | 0.3 (4)                        | C14—N4—C8—N3        | −2.2 (3)                       |
| F36—C38—C39—C40      | −179.9 (2)                     | C14—C9—C10—C11      | −1.3 (4)                       |
| C35—C36—C37—I3       | 176.28 (19)                    | N5—C16—C17—C18      | 179.3 (3)                      |
| C35—C36—C37—C38      | −1.2 (4)                       | N5—C16—C21—N6       | −0.4 (3)                       |
| C36—C35—C40—F34      | −178.9 (2)                     | N5—C16—C21—C20      | −179.2 (2)                     |
supporting information

C36—C35—C40—C39 1.6 (4) C15—N5—C16—C17 179.3 (3)
C36—C37—C38—F36 179.0 (2) C15—N6—C21—C20 179.8 (3)
C36—C37—C38—C39 1.7 (4) C15—N6—C16—C21 0.6 (3)
C37—C38—C39—C40 0.6 (4) C16—N5—C15—S3 177.01 (19)
C37—C38—C39—C40 179.4 (2) C16—N5—C15—N6 1.3 (3)
C37—C38—C39—C40 179.3 (2) C16—N6—C21—C16 179.5 (2)
C38—C39—C40—F36 179.0 (2) C15—N5—C16—C21 0.3 (4)
C38—C39—C40—C35 −1.1 (4) C16—N6—C21—C20 −0.3 (4)
C40—C35—C36—F33 179.3 (2) C17—C16—C21—N6 178.5 (2)
C40—C35—C36—C37 0.4 (4) C17—C16—C21—C20 0.3 (4)
I5—C41—C42—F37 3.0 (3) C17—C16—C21—C20 −0.7 (4)
I5—C41—C42—C43 177.25 (18) C21—N6—C15—S3 176.77 (19)
I5—C41—C46—F40 0.8 (3) C21—N6—C15—N5 1.6 (3)
I5—C41—C46—C45 −178.55 (18) C21—N6—C15—N5 −1.6 (3)
I6—C43—C44—F38 2.9 (3) C21—N6—C15—S3 176.77 (19)
I6—C43—C44—C45 177.54 (18) C21—N6—C15—N5 1.6 (3)
F37—C42—C43—C44 2.7 (3) C21—C16—C17—C18 0.8 (4)
F37—C42—C43—C44 178.6 (2) C21—C16—C17—C18 179.5 (2)
F38—C44—C45—F39 1.2 (3) C21—C16—C17—C18 0.8 (4)
F38—C44—C45—C46 −179.6 (2) C21—C16—C17—C18 179.5 (2)
F39—C45—C46—F40 1.2 (3) C21—C16—C17—C18 179.5 (2)
F39—C45—C46—C41 −179.4 (2) C22—N7—C23—C24 179.8 (3)
C41—C42—C43—C44 −177.57 (18) C22—N7—C23—C24 −179.8 (3)
C41—C42—C43—C44 1.1 (4) C22—N7—C23—C24 179.8 (3)
C42—C41—C46—F40 178.0 (2) C22—N7—C23—C24 −179.8 (3)
C42—C41—C46—C45 −1.3 (4) C22—N7—C23—C24 179.8 (3)
C42—C43—C44—F38 178.4 (2) C22—N7—C23—C24 179.8 (3)
C42—C43—C44—F38 −1.2 (4) C22—N7—C23—C24 179.8 (3)
C43—C44—C45—F39 −179.2 (2) C22—N7—C23—C24 −179.8 (3)
C43—C44—C45—C46 0.0 (4) C22—N7—C23—C24 179.8 (3)
C44—C45—C46—F40 −178.1 (2) C22—N7—C23—C24 179.8 (3)
C44—C45—C46—C41 1.3 (4) C22—N7—C23—C24 179.8 (3)
C46—C41—C42—F37 179.8 (2) C22—N7—C23—C24 179.8 (3)
C46—C41—C42—C43 0.1 (4) C22—N7—C23—C24 179.8 (3)
N1—C2—C3—C4 176.7 (3) C28—N8—C22—S4 179.97 (19)
N1—C2—C7—N2 0.4 (3) C28—N8—C22—S4 179.97 (19)

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|-----|------|-------|---------|
| N1—H1N1···S2i | 0.85 (2) | 2.52 (2) | 3.357 (2) | 172 (3) |
| N2—H2N2···S2 | 0.85 (2) | 2.46 (2) | 3.297 (2) | 166 (2) |
| N3—H3N3···S1 | 0.85 (2) | 2.51 (2) | 3.348 (2) | 173 (3) |
| N4—H4N4···S1ii | 0.85 (2) | 2.50 (2) | 3.326 (2) | 166 (2) |
| N5—H5N5···S4i | 0.85 (2) | 2.49 (2) | 3.326 (2) | 169 (3) |
| N6—H6N6···S4 | 0.85 (2) | 2.43 (2) | 3.270 (2) | 169 (3) |
| C17—H17···F36iii | 0.95 | 2.61 | 3.385 (3) | 139 |
| C20—H20···F36iv | 0.95 | 2.51 | 3.235 (3) | 133 |
N7—HN7···S3 0.84 (2) 2.47 (2) 3.300 (2) 170 (3)
N8—HN8···S3ii 0.85 (2) 2.48 (2) 3.302 (2) 163 (3)

Symmetry codes: (i) x+1, y, z; (ii) x−1, y, z; (iii) −x+1, −y+1, −z+1; (iv) −x, −y+1, −z+1.

1H-1,3-Benzodiazole-2-thiol–1,2,4,5-tetrafluoro-3,6-diiodobenzene (1/1) (MBZIM_14F4DIB)

Crystal data

\[ \text{C}_6\text{F}_4\text{I}_2\cdot\text{C}_7\text{H}_6\text{N}_2\text{S} \]

\[ M_r = 552.06 \]

Monoclinic, \( P2_1/c \)

\[ a = 5.5641 (2) \text{ Å} \]

\[ b = 33.1320 (11) \text{ Å} \]

\[ c = 8.4710 (3) \text{ Å} \]

\[ \beta = 92.754 (1)^\circ \]

\[ V = 1559.82 (9) \text{ Å}^3 \]

\[ Z = 4 \]

\[ F(000) = 1024 \]

\[ D_x = 2.351 \text{ Mg m}^{-3} \]

Mo \( K\alpha \) radiation, \( \lambda = 0.71073 \text{ Å} \)

Cell parameters from 9812 reflections

\[ \theta = 2.5–30.2^\circ \]

\[ \mu = 4.20 \text{ mm}^{-1} \]

\[ T = 100 \text{ K} \]

Plate, colourless

0.22 \times 0.18 \times 0.06 \text{ mm}

Data collection

Bruker D8 Venture Photon 2
diffractometer

4579 independent reflections

4211 reflections with \( I > 2\sigma(I) \)

Radiation source: Incoatec I\( \mu \)S

\( \varphi \) and \( \omega \) scans

Absorption correction: multi-scan

(SADABS; Bruker, 2017)

\( k = -46 \rightarrow 46 \)

\( l = -11 \rightarrow 11 \)

45583 measured reflections

Refinement

Refinement on \( F^2 \)

Least-squares matrix: full

\( R[F^2 > 2\sigma(F^2)] = 0.020 \)

\( wR(F^2) = 0.044 \)

\[ S = 1.12 \]

4579 reflections

207 parameters

0 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

\( w = 1/[\sigma^2(F_o^2) + (0.0059P)^2 + 1.8833P] \)

where \( P = (F_o^2 + 2F_c^2)/3 \)

\( (\Delta\sigma)_{\text{max}} = 0.002 \)

\( \Delta\rho_{\text{max}} = 0.49 \text{ e Å}^{-3} \)

\( \Delta\rho_{\text{min}} = -0.67 \text{ e Å}^{-3} \)

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     | \( U_{	ext{iso}}^*/U_{	ext{eq}} \) |
|-----|-------|-------|-------|-------------------------------|
| I1  | 0.61301 (2) | 0.38195 (2) | 0.76376 (2) | 0.01796 (4) |
| I2  | 0.89550 (3) | 0.17772 (2) | 0.69482 (2) | 0.02372 (4) |
| F1  | 1.0451 (2)  | 0.33570 (4) | 0.60867 (16) | 0.0251 (3)  |
| F2  | 1.1463 (3)  | 0.25762 (4) | 0.57675 (17) | 0.0283 (3)  |
| F3  | 0.4636 (3)  | 0.22323 (4) | 0.85802 (18) | 0.0292 (3)  |
| F4  | 0.3576 (2)  | 0.30171 (4) | 0.88704 (18) | 0.0285 (3)  |
### Atomic displacement parameters (Å²)

|     | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|-----|----------|----------|----------|----------|----------|----------|
| I1  | 0.02099 (7) | 0.01539 (7) | 0.01731 (7) | 0.00072 (5) | -0.00093 (5) | -0.00014 (5) |
| I2  | 0.03021 (8) | 0.01541 (7) | 0.02536 (8) | 0.00224 (6) | -0.00059 (6) | -0.00099 (5) |
| F1  | 0.0259 (7) | 0.0215 (6) | 0.0286 (7) | -0.0048 (6) | 0.0099 (6) | 0.0039 (5) |
| F2  | 0.0264 (7) | 0.0260 (7) | 0.0339 (8) | 0.0022 (6) | 0.0152 (6) | -0.0012 (6) |
| F3  | 0.0255 (7) | 0.0191 (7) | 0.0440 (9) | -0.0047 (6) | 0.0132 (6) | 0.0058 (6) |
| F4  | 0.0222 (7) | 0.0248 (7) | 0.0400 (8) | 0.0019 (6) | 0.0160 (6) | 0.0021 (6) |
| C8  | 0.0175 (9) | 0.0145 (9) | 0.0159 (9) | 0.0007 (8) | -0.0011 (7) | -0.0006 (7) |
| C9  | 0.0195 (10) | 0.0183 (10) | 0.0151 (9) | -0.0039 (8) | 0.0025 (8) | 0.0026 (7) |
| C10 | 0.0171 (10) | 0.0210 (10) | 0.0169 (9) | 0.0011 (8) | 0.0036 (8) | -0.0004 (8) |
| C11 | 0.0199 (10) | 0.0146 (9) | 0.0174 (10) | -0.0004 (8) | -0.0018 (8) | -0.0009 (7) |
| C12 | 0.0187 (10) | 0.0178 (10) | 0.0200 (10) | -0.0040 (8) | 0.0030 (8) | 0.0013 (8) |
| C13 | 0.0172 (10) | 0.0202 (10) | 0.0207 (10) | 0.0021 (8) | 0.0041 (8) | -0.0001 (8) |
| S1  | 0.0208 (2) | 0.0150 (2) | 0.0116 (2) | 0.00308 (19) | 0.00018 (18) | -0.00025 (16) |
| N1  | 0.0177 (8) | 0.0184 (8) | 0.0096 (8) | 0.0020 (7) | -0.0017 (6) | -0.0003 (6) |
| N2  | 0.0175 (8) | 0.0172 (8) | 0.0101 (8) | 0.0005 (7) | 0.0010 (6) | -0.0007 (6) |
| C1  | 0.0180 (9) | 0.0129 (9) | 0.0119 (8) | -0.0026 (8) | -0.0003 (7) | -0.0004 (7) |
| C2  | 0.0150 (9) | 0.0152 (9) | 0.0139 (9) | -0.0016 (8) | -0.0011 (7) | 0.0003 (7) |
| C3  | 0.0198 (10) | 0.0191 (10) | 0.0151 (9) | -0.0017 (8) | 0.0010 (8) | -0.0029 (7) |
| C4  | 0.0182 (10) | 0.0201 (10) | 0.0206 (10) | 0.0009 (8) | 0.0021 (8) | -0.0022 (8) |
| C5  | 0.0191 (10) | 0.0186 (10) | 0.0229 (11) | 0.0009 (8) | -0.0017 (8) | 0.0021 (8) |
| C6  | 0.0203 (10) | 0.0212 (10) | 0.0139 (9) | 0.0000 (8) | -0.0021 (8) | 0.0021 (7) |
| C7  | 0.0158 (9) | 0.0158 (9) | 0.0125 (9) | -0.0017 (8) | -0.0006 (7) | -0.0007 (7) |
Geometric parameters (Å, °)

| Bond                  | Distance (Å) | Angle (°) |
|-----------------------|--------------|-----------|
| I1—C8                 | 2.084 (2)    |           |
| I2—C11                | 2.078 (2)    |           |
| F1—C9                 | 1.342 (2)    |           |
| F2—C10                | 1.338 (2)    |           |
| F3—C12                | 1.338 (2)    |           |
| F4—C13                | 1.346 (2)    |           |
| C8—C9                 | 1.388 (3)    |           |
| C8—C13                | 1.387 (3)    |           |
| C9—C10                | 1.379 (3)    |           |
| C10—C11               | 1.386 (3)    |           |
| C11—C12               | 1.384 (3)    |           |
| C12—C13               | 1.382 (3)    |           |
| S1—C1                 | 1.700 (2)    |           |
| N1—HN1                | 0.84 (3)     |           |
| N1—C2                 | 1.352 (2)    |           |
| C9—C8—I1              | 120.68 (15)  | 128.7 (18) |
| C13—C8—I1             | 121.91 (15)  | 126.31 (16)|
| C13—C8—C9             | 117.41 (19)  | 126.27 (15)|
| F1—C9—C8              | 120.03 (19)  | 107.42 (18)|
| F1—C9—C10             | 118.85 (18)  | 106.11 (17)|
| C10—C9—C8             | 121.11 (19)  | 131.90 (19)|
| F2—C10—C9             | 118.48 (19)  | 121.97 (19)|
| C9—C8—I1              | 120.25 (19)  | 121.6     |
| C13—C8—I1             | 121.27 (19)  | 116.70 (19)|
| C10—C11—I2            | 120.42 (15)  | 112.6     |
| C12—C11—I2            | 121.65 (16)  | 119.4     |
| C12—C11—C10           | 117.92 (19)  | 121.2 (2) |
| F3—C12—C11            | 120.16 (19)  | 119.4     |
| F3—C12—C13            | 119.09 (19)  | 119.0     |
| C13—C12—C11           | 120.75 (19)  | 121.9 (2) |
| F4—C13—C8             | 119.71 (19)  | 119.0     |
| F4—C13—C12            | 118.75 (19)  | 121.7     |
| C12—C13—C8            | 121.54 (19)  | 116.65 (19)|
| C1—N1—HN1             | 125.0 (18)   | 121.7     |
| C1—N1—C2              | 110.19 (17)  | 106.19 (17)|
| C2—N1—HN1             | 124.6 (18)   | 132.27 (18)|
| C1—N2—HN2             | 121.0 (19)   | 121.53 (19)|
| C1—N2—C7              | 110.07 (17)  |           |
| I1—C8—I2              | 2.5 (3)      |           |
| I1—C8—C9—I1           | −178.66 (16) |           |
| I1—C8—C9—C10          | −1.1 (3)     |           |
| I1—C8—C13—C12         | 178.80 (17)  |           |
| I2—C11—C12—F3         | −0.8 (3)     |           |
| I2—C11—C12—C13        | 178.65 (17)  |           |
Hydrogen-bond geometry (Å, °)

| D—H···A       | D—H | H···A | D···A  | D—H···A |
|---------------|-----|-------|--------|---------|
| N1—H1N1···S1i | 0.84 (3) | 2.47 (3) | 3.3089 (18) | 172 (2) |
| N2—H2N2···S1ii | 0.86 (3) | 2.50 (3) | 3.3527 (17) | 172 (2) |

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

1H-1,3-Benzodiazole-2-thiol–1,1,2,2-tetraiodoethene (1/1) (MBZIM_TIE)

Crystal data

C_{12}C_{13}H_{13}N_{2}S  
Mr = 681.82  
Orthorhombic, Pnma  
a = 11.7547 (10) Å  
b = 8.3525 (7) Å  
c = 15.1077 (13) Å  
V = 1483.3 (2) Å³  
Z = 4  
F(000) = 1208  

Data collection

Bruker D8 Venture Photon 2 diffractometer  
Radiation source: Incoatec IµS  
φ and ω scans  
Absorption correction: multi-scan (SADABS; Bruker, 2017)  
T_{min} = 0.256, T_{max} = 0.746  
32859 measured reflections  

Refinement

Refinement on F²  
Least-squares matrix: full  
R[F² > 2σ(F²)] = 0.026  
wR(F²) = 0.062  
S = 1.25  
89 parameters  
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

\[ w = 1/\left[ \sigma^2(F_o^2) + (0.0213P)^2 + 6.7951P \right] \]

where \( P = (F_o^2 + 2F_c^2)/3 \)

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

\( \Delta \rho_{\text{max}} = 1.25 \text{ e Å}^{-3} \)

\( \Delta \rho_{\text{min}} = -1.48 \text{ e Å}^{-3} \)

**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   | 0.71885 (3) | 0.250000 | 0.41396 (2) | 0.01158 (9) |
| I2   | 0.48169 (3) | 0.250000 | 0.27066 (2) | 0.01399 (9) |
| I3   | 0.29263 (3) | 0.250000 | 0.46197 (2) | 0.01401 (9) |
| I4   | 0.52953 (3) | 0.250000 | 0.60280 (2) | 0.02001 (10) |
| C5   | 0.5414 (5) | 0.250000 | 0.4030 (4) | 0.0165 (11) |
| C6   | 0.4724 (5) | 0.250000 | 0.4721 (4) | 0.0174 (11) |
| S1   | 1.01449 (11) | 0.250000 | 0.45747 (8) | 0.0100 (2) |
| N1   | 0.9064 (3) | 0.3808 (4) | 0.5997 (2) | 0.0103 (6) |
| HN1  | 0.925 (4) | 0.478 (6) | 0.586 (3) | 0.016 (12)* |
| C1   | 0.9436 (4) | 0.250000 | 0.5556 (3) | 0.0113 (10) |
| C2   | 0.8394 (3) | 0.3332 (4) | 0.6716 (2) | 0.0101 (7) |
| C3   | 0.7765 (3) | 0.4217 (5) | 0.7322 (2) | 0.0124 (7) |
| H3   | 0.776830 | 0.535466 | 0.732234 | 0.015* |
| C4   | 0.7128 (3) | 0.3335 (5) | 0.7928 (2) | 0.0134 (7) |
| H4   | 0.668132 | 0.388696 | 0.835332 | 0.016* |

**Atomic displacement parameters (Å²)**

|      | \( U^1 \)  | \( U^2 \)  | \( U^3 \)  | \( U^{12} \) | \( U^{13} \) | \( U^{23} \) |
|------|-------------|-------------|-------------|-------------|-------------|-------------|
| I1   | 0.00779 (16) | 0.01634 (17) | 0.01059 (16) | 0.000 | -0.00164 (11) | 0.000 |
| I2   | 0.01027 (17) | 0.02331 (19) | 0.00839 (16) | 0.000 | -0.00137 (11) | 0.000 |
| I3   | 0.00843 (16) | 0.01822 (18) | 0.01538 (17) | 0.000 | 0.00119 (12) | 0.000 |
| I4   | 0.01263 (18) | 0.0392 (2) | 0.00821 (17) | 0.000 | -0.00150 (12) | 0.000 |
| C5   | 0.012 (3)  | 0.023 (3)   | 0.014 (3)   | 0.000 | -0.002 (2)  | 0.000 |
| C6   | 0.019 (3)  | 0.025 (3)   | 0.008 (2)   | 0.000 | -0.004 (2)  | 0.000 |
| S1   | 0.0102 (5) | 0.0095 (5) | 0.0102 (5) | 0.000 | 0.0029 (4) | 0.000 |
| N1   | 0.0103 (14) | 0.0088 (14) | 0.0117 (14) | -0.0008 (11) | 0.0015 (11) | -0.0004 (11) |
| C1   | 0.008 (2)  | 0.014 (2)   | 0.012 (2)   | 0.000 | -0.0048 (18) | 0.000 |
| C2   | 0.0090 (15) | 0.0123 (18) | 0.0090 (15) | 0.000 (13) | -0.0024 (12) | -0.0001 (13) |
| C3   | 0.0132 (17) | 0.0116 (16) | 0.0125 (16) | 0.0013 (13) | 0.0002 (13) | -0.0011 (13) |
| C4   | 0.0109 (16) | 0.0172 (19) | 0.0119 (16) | 0.0023 (14) | 0.0030 (13) | -0.0017 (14) |
Geometric parameters (Å, °)

| Bond          | Length  | Angle     |
|---------------|---------|-----------|
| I1—C5         | 2.093 (6) | N1—C2 1.400 (4) |
| I2—C5         | 2.119 (6) | C2—C2i 1.390 (7) |
| I3—C6         | 2.118 (6) | C2—C3 1.389 (5) |
| I4—C6         | 2.086 (5) | C3—H3 0.9500 |
| C5—C6         | 1.321 (8) | C3—C4 1.393 (5) |
| S1—C1         | 1.701 (6) | C4—C4i 1.395 (8) |
| N1—HN1        | 0.87 (5) | C4—H4 0.9500 |
| N1—C1         | 1.352 (4) |                  |
| I1—C5—I2      | 113.9 (3) | N1i—C1—N1 107.8 (5) |
| C6—C5—I1      | 123.3 (4) | C2i—C2—N1 106.5 (2) |
| C6—C5—I2      | 122.8 (4) | C3—C2—N1 131.3 (3) |
| C6—C5—I4      | 123.7 (4) | C2—C3—H3 122.1 |
| C5—C6—I3      | 123.3 (5) | C2—C3—C4 115.9 (3) |
| C5—C6—I4      | 123.3 (5) | C3—C4—C4i 121.4 (2) |
| C1—N1—HN1     | 124 (3)  | C4i—C4—H4 119.0 |
| C1—N1—C2      | 109.6 (3) |                  |
| C2—N1—HN1     | 127 (3)  |                  |
| N1—C1—S1      | 126.0 (2) |                  |
| N1i—C1—S1     | 126.0 (2) |                  |
| I1—C5—C6—I3   | 180.000 (1) | C1—N1—C2—C3 -174.9 (4) |
| I1—C5—C6—I4   | 0.000 (1)  | C2—N1—C1—S1 172.5 (3) |
| I2—C5—C6—I3   | 0.000 (1)  | C2—N1—C1—N1i -2.6 (5) |
| I2—C5—C6—I4   | 180.000 (1) | C2i—C2—C3—C4 -0.3 (4) |
| N1—C2—C3—C4   | 175.8 (4)  | C2—C3—C4—C4i 0.3 (4) |
| C1—N1—C2—C2i  | 1.6 (3)    |                  |

Symmetry code: (i) x, −y+1/2, z.

Hydrogen-bond geometry (Å, °)

| Bond          | D—H—A   |
|---------------|---------|
| N1—HN1···S1ii | 0.87 (5) |
| C3—H3···I1iii| 0.95     |

Symmetry codes: (ii) −x+2, −y+1, −z+1; (iii) −x+3/2, −y+1, z+1/2.

5-Methyl-1H-1,3-benzodiazole-2-thiol–1,2,3,4-tetrafluoro-5,6-diiodobenzene (1/1) (MMBZIM_12F4DIB)

Crystal data

$C_6F_4I_2·C_8H_8N_2S$

$M_r = 566.08$

Triclinic, $P\bar{1}$

$\alpha = 4.5504$ Å

$\beta = 13.2872$ (14) Å

$\gamma = 13.8064$ (14) Å

$a = 94.766$ (4)$^\circ$

$\beta = 98.124$ (4)$^\circ$

$\gamma = 99.588$ (4)$^\circ$

$V = 809.97$ (15) Å$^3$

$D_x = 2.321$ Mg m$^{-3}$

$F(000) = 528$

$D_{\rho} = 2.321$ Mg m$^{-3}$

$\theta = 2.3–27.5^\circ$

Cell parameters from 9940 reflections

$\theta = 2.3–27.5^\circ$

$\alpha = 94.766$ (4)$^\circ$

$\beta = 98.124$ (4)$^\circ$

$\gamma = 99.588$ (4)$^\circ$
$\mu = 4.05$ mm$^{-1}$

$T = 100$ K

Needle, colourless

0.19 $\times$ 0.07 $\times$ 0.04 mm

**Data collection**

Bruker D8 Venture Photon 2
diffractometer

Radiation source: Incoatec I\(\mu\)S

$\varphi$ and $\omega$ scans

Absorption correction: multi-scan
(SADABS; Bruker, 2017)

$T_{\text{min}} = 0.636$, $T_{\text{max}} = 0.746$

21426 measured reflections

3704 independent reflections

3174 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.042$

$\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 2.3^\circ$

$h = -5 \rightarrow 5$

$k = -17 \rightarrow 17$

$l = -17 \rightarrow 17$

**Refinement**

Refinement on $F^2$

Least-squares matrix: full

$R(F^2 > 2\sigma(F^2)) = 0.026$

$wR(F^2) = 0.055$

$S = 1.24$

3704 reflections

217 parameters

1 restraint

Primary atom site location: dual

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma(F^2)' + 2.2494P]$

where $P = (F^2 + 2F^2)/3$

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

$\Delta\rho_{\text{max}} = 1.33$ e Å$^{-3}$

$\Delta\rho_{\text{min}} = -1.06$ 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 ($\text{Å}^2$)**

|    | $x$   | $y$   | $z$  | $U_{\text{iso}}$/\(U_{\text{eq}}\) |
|----|-------|-------|------|---------------------------------|
| I1 | 0.65636 (6) | 0.37716 (2) | 0.58352 (2) | 0.02050 (7) |
| I2 | 0.65659 (5) | 0.36653 (2) | 0.30961 (2) | 0.01661 (7) |
| F1 | 0.1607 (5) | 0.17992 (18) | 0.21872 (16) | 0.0254 (5) |
| F2 | -0.2407 (6) | 0.04739 (19) | 0.29154 (19) | 0.0334 (6) |
| F3 | -0.2418 (6) | 0.05622 (19) | 0.4886 (2) | 0.0326 (6) |
| F4 | 0.1622 (6) | 0.1944 (2) | 0.61205 (17) | 0.0280 (6) |
| C9 | 0.3758 (8) | 0.2652 (3) | 0.4798 (3) | 0.0154 (7) |
| C10 | 0.3778 (8) | 0.2613 (3) | 0.3784 (3) | 0.0151 (7) |
| C11 | 0.1718 (9) | 0.1857 (3) | 0.3168 (3) | 0.0174 (8) |
| C12 | -0.0361 (9) | 0.1169 (3) | 0.3527 (3) | 0.0206 (8) |
| C13 | -0.0373 (9) | 0.1222 (3) | 0.4535 (3) | 0.0206 (8) |
| C14 | 0.1684 (9) | 0.1943 (3) | 0.5145 (3) | 0.0182 (8) |
| S1 | -0.0441 (2) | 0.50172 (8) | 0.83086 (7) | 0.0204 (2) |
| N1 | 0.2256 (7) | 0.3842 (3) | 0.9578 (2) | 0.0184 (7) |
| HN1 | 0.165 (10) | 0.414 (4) | 1.008 (4) | 0.029 (13)* |
| N2 | 0.2606 (7) | 0.3443 (2) | 0.8049 (2) | 0.0165 (7) |
| HN2 | 0.254 (9) | 0.349 (3) | 0.7436 (15) | 0.009 (10)* |
| C1 | 0.1504 (9) | 0.4093 (3) | 0.8658 (3) | 0.0186 (8) |
| C2 | 0.3863 (8) | 0.3038 (3) | 0.9558 (3) | 0.0172 (8) |
| Atomic displacement parameters (Å²) | \(U_{11}^{11}\) | \(U_{22}^{12}\) | \(U_{33}^{13}\) | \(U_{12}^{12}\) | \(U_{13}^{13}\) | \(U_{23}^{13}\) |
|-----------------------------------|----------|----------|----------|----------|----------|----------|
| I1 0.02224 (14)                   | 0.02260 (14) | 0.01557 (12) | 0.00692 (10) | −0.00119 (9) | −0.00285 (10) |
| I2 0.01925 (13)                   | 0.01578 (12) | 0.01588 (12) | 0.00435 (9) | 0.00466 (9) | 0.00245 (9) |
| F1 0.0295 (13)                    | 0.0253 (13) | 0.0173 (11) | −0.0002 (10) | −0.0002 (10) | −0.0036 (10) |
| F2 0.0269 (14)                    | 0.0274 (14) | 0.0386 (15) | −0.0070 (11) | −0.0003 (11) | −0.0060 (11) |
| F3 0.0246 (13)                    | 0.0283 (14) | 0.0489 (17) | 0.0016 (11) | 0.0171 (12) | 0.0143 (12) |
| F4 0.0320 (14)                    | 0.0389 (15) | 0.0181 (12) | 0.0110 (11) | 0.0106 (10) | 0.0116 (11) |
| C9 0.0157 (18)                    | 0.0167 (18) | 0.0136 (17) | 0.0051 (15) | 0.0014 (14) | −0.0022 (14) |
| C10 0.0173 (18)                   | 0.0148 (18) | 0.0135 (17) | 0.0033 (14) | 0.0024 (14) | 0.0016 (14) |
| C11 0.0205 (19)                   | 0.0172 (19) | 0.0151 (18) | 0.0071 (15) | 0.0012 (14) | 0.0000 (15) |
| C12 0.0161 (19)                   | 0.0167 (19) | 0.027 (2) | 0.0010 (15) | 0.0000 (15) | −0.0015 (16) |
| C13 0.0132 (18)                   | 0.0170 (19) | 0.035 (2) | 0.0052 (15) | 0.0101 (16) | 0.0061 (17) |
| C14 0.021 (2)                     | 0.021 (2) | 0.0155 (18) | 0.0099 (16) | 0.0041 (15) | 0.0056 (15) |
| S1 0.0227 (5)                     | 0.0197 (5) | 0.0214 (5) | 0.0062 (4) | 0.0081 (4) | 0.0052 (4) |
| N1 0.0228 (18)                    | 0.0185 (17) | 0.0150 (16) | 0.0047 (14) | 0.0065 (13) | 0.0005 (13) |
| N2 0.0214 (17)                    | 0.0202 (17) | 0.0102 (15) | 0.0073 (13) | 0.0045 (12) | 0.0037 (13) |
| C1 0.021 (2)                      | 0.0172 (19) | 0.0179 (19) | −0.0010 (15) | 0.0066 (15) | 0.0044 (15) |
| C2 0.0175 (19)                    | 0.0193 (19) | 0.0152 (18) | 0.0014 (15) | 0.0063 (14) | 0.0018 (15) |
| C3 0.024 (2)                      | 0.029 (2) | 0.0122 (18) | 0.0037 (17) | 0.0042 (15) | 0.0039 (16) |
| C4 0.020 (2)                      | 0.026 (2) | 0.019 (2) | 0.0011 (17) | −0.0010 (15) | 0.0054 (16) |
| C5 0.0158 (19)                    | 0.025 (2) | 0.026 (2) | 0.0015 (16) | 0.0018 (16) | 0.0020 (17) |
| C6 0.0193 (19)                    | 0.0207 (19) | 0.0145 (18) | 0.0041 (16) | 0.0039 (15) | 0.0014 (15) |
| C7 0.0192 (19)                    | 0.0186 (19) | 0.0146 (18) | 0.0027 (15) | 0.0023 (14) | 0.0018 (15) |
| C8 0.020 (2)                      | 0.026 (2) | 0.029 (2) | 0.0066 (17) | 0.0019 (17) | 0.0049 (18) |

Geometric parameters (Å, °)

| I1—C9 | 2.095 (4) | N2—HN2 | 0.850 (18) |
|-------|----------|--------|------------|
| I2—C10 | 2.106 (4) | N2—C1 | 1.358 (5) |
| F1—C11 | 1.344 (4) | N2—C7 | 1.393 (5) |
| F2—C12 | 1.340 (4) | C2—C3 | 1.385 (5) |
| F3—C13 | 1.340 (4) | C2—C7 | 1.391 (5) |
| F4—C14 | 1.352 (4) | C3—H3 | 0.9500 |
| Bond                  | Distance (Å) | Bond                  | Distance (Å) |
|----------------------|--------------|----------------------|--------------|
| C9—C10               | 1.399 (5)    | C3—C4                | 1.392 (6)    |
| C9—C14               | 1.383 (5)    | C4—H4                | 0.9500       |
| C10—C11              | 1.389 (5)    | C4—C5                | 1.406 (6)    |
| C11—C12              | 1.377 (6)    | C5—C6                | 1.390 (5)    |
| C12—C13              | 1.389 (6)    | C5—C8                | 1.511 (6)    |
| C13—C14              | 1.358 (6)    | C6—H6                | 0.9500       |
| S1—C1                | 1.693 (4)    | C6—C7                | 1.391 (5)    |
| N1—HN1               | 0.88 (5)     | C8—H8A               | 0.9800       |
| N1—C1                | 1.351 (5)    | C8—H8B               | 0.9800       |
| N1—C2                | 1.392 (5)    | C8—H8C               | 0.9800       |
| C10—C9—I1            | 123.8 (3)    | N2—C1—S1             | 125.9 (3)    |
| C14—C9—I1            | 117.3 (3)    | C3—C2—N1             | 132.5 (4)    |
| C14—C9—C10           | 118.8 (3)    | C3—C2—C7             | 120.7 (4)    |
| C9—C10—I2            | 125.0 (3)    | C7—C2—N1             | 106.9 (3)    |
| C11—C10—I2           | 116.6 (3)    | C2—C3—H3             | 121.3        |
| C11—C10—C9           | 118.3 (4)    | C2—C3—C4             | 117.4 (4)    |
| F1—C11—C10           | 120.6 (3)    | C4—C3—H3             | 121.3        |
| F1—C11—C12           | 117.3 (3)    | C3—C4—H4             | 119.0        |
| C12—C11—C10          | 122.0 (4)    | C3—C4—C5             | 122.1 (4)    |
| F2—C12—C11           | 120.9 (4)    | C5—C4—H4             | 119.0        |
| F2—C12—C13           | 120.0 (4)    | C4—C5—C8             | 119.4 (4)    |
| C11—C12—C13          | 119.1 (4)    | C6—C5—C4             | 120.0 (4)    |
| F3—C13—C12           | 119.2 (4)    | C6—C5—C8             | 120.6 (4)    |
| F3—C13—C14           | 121.4 (4)    | C5—C6—H6             | 121.2        |
| C14—C13—C12          | 119.3 (4)    | C5—C6—C7             | 117.5 (4)    |
| F4—C14—C9            | 120.6 (3)    | C7—C6—H6             | 121.2        |
| F4—C14—C13           | 116.9 (4)    | C2—C7—N2             | 105.5 (3)    |
| C13—C14—C9           | 122.5 (4)    | C6—C7—N2             | 132.2 (3)    |
| C1—N1—HN1            | 121 (3)      | C6—C7—C2             | 122.3 (3)    |
| C1—N1—C2             | 110.4 (3)    | C5—C8—H8A            | 109.5        |
| C2—N1—HN1            | 128 (3)      | C5—C8—H8B            | 109.5        |
| C1—N2—HN2            | 124 (3)      | C5—C8—H8C            | 109.5        |
| C1—N2—C7             | 111.0 (3)    | H8A—C8—H8B           | 109.5        |
| C7—N2—HN2            | 125 (3)      | H8A—C8—H8C           | 109.5        |
| N1—C1—S1             | 127.9 (3)    | H8B—C8—H8C           | 109.5        |
| N1—C1—N2             | 106.2 (3)    |                       |              |
| I1—C9—C10—I2         | −0.5 (5)     | C14—C9—C10—C11      | −0.4 (5)     |
| I1—C9—C10—C11        | −177.4 (3)   | N1—C2—C3—C4         | 179.6 (4)    |
| I1—C9—C14—F4         | −3.9 (5)     | N1—C2—C7—N2         | −0.5 (4)     |
| I1—C9—C14—C13        | 175.8 (3)    | N1—C2—C7—C6         | 179.7 (4)    |
| I2—C10—C11—F1        | 1.7 (5)      | C1—N1—C2—C3         | −179.6 (4)   |
| I2—C10—C11—C12       | −175.5 (3)   | C1—N1—C2—C7         | 0.0 (4)      |
| F1—C11—C12—F2        | −0.1 (5)     | C1—N2—C7—C2         | 0.8 (4)      |
| F1—C11—C12—C13       | −178.3 (3)   | C1—N2—C7—C6         | −179.4 (4)   |
| F2—C12—C13—F3        | 1.0 (5)      | C2—N1—C1—S1         | −180.0 (3)   |
| F2—C12—C13—C14       | −178.9 (3)   | C2—N1—C1—N2         | 0.5 (4)      |
F3—C13—C14—F4  1.7 (5)  C2—C3—C4—C5  −0.2 (6)
F3—C13—C14—C9  −178.0 (3)  C3—C2—C7—N2  179.2 (4)
C9—C10—C11—F1  178.8 (3)  C3—C2—C7—C6  −0.7 (6)
C9—C10—C11—C12  1.5 (6)  C3—C4—C5—C8  −178.8 (4)
C10—C9—C14—F4  179.0 (3)  C3—C4—C5—C8  −178.8 (4)
C10—C9—C14—C13  −1.3 (6)  C4—C5—C6—C7  −1.3 (6)
C10—C11—C12—F2  177.2 (3)  C5—C6—C7—N2  −178.5 (4)
C10—C11—C12—C13  −1.0 (6)  C5—C6—C7—C2  1.3 (6)
C11—C12—C13—F3  179.2 (3)  C7—N2—C1—S1  179.6 (3)
C11—C12—C13—C14  −0.7 (6)  C7—N2—C1—N1  −0.8 (4)
C12—C13—C14—F4  −178.4 (3)  C7—C2—C3—C4  0.1 (6)
C12—C13—C14—C9  1.9 (6)  C8—C5—C6—C7  178.3 (4)
C14—C9—C10—I2  176.4 (3)

Hydrogen-bond geometry (Å, °)

| D—H···A     | D—H   | H···A  | D···A   | D—H···A |
|-------------|--------|--------|---------|---------|
| N1—H1N1···S1i | 0.88 (5) | 2.57 (5) | 3.444 (3) | 173 (4) |
| N2—H2N2···I1  | 0.85 (2) | 3.07 (3) | 3.780 (3) | 142 (3) |
| N2—H2N2···F4  | 0.85 (2) | 2.56 (3) | 3.122 (4) | 124 (3) |
| C3—H3···I2ii  | 0.95    | 3.06   | 3.966 (4) | 160     |
| C6—H6···F4    | 0.95    | 2.63   | 3.262 (4) | 125     |

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

5-Methyl-1H-1,3-benzodiazole-2-thiol–1,2,4,5-tetrafluoro-3,6-diiodobenzene–water (2/1/2)
(2MMBZIM_14F4DIB_2H2O)

Crystal data

C₆F₄I₂·2C₈H₈N₂S·2(H₂O)
Mᵣ = 766.34
Triclinic, Pائر
a = 4.9088 (3) Å
b = 11.4670 (8) Å
c = 11.9686 (8) Å
α = 106.644 (2)°
β = 11.9686 (8) Å
γ = 92.811 (2)°
V = 636.27 (7) Å³
Z = 1
F(000) = 370
Dₐ = 2.000 Mg m⁻³
Mo Ka radiation, λ = 0.71073 Å
Cell parameters from 9704 reflections
θ = 3.0–29.6°
μ = 2.69 mm⁻¹
T = 100 K
Plate, colourless
0.31 × 0.11 × 0.08 mm

Data collection

Bruker D8 Venture Photon 2
diffractometer
Radiation source: Incoatec IµS
φ and ω scans
Absorption correction: multi-scan
(SADABS; Bruker, 2017)
T_min = 0.536, T_max = 0.746
31584 measured reflections
3558 independent reflections
3500 reflections with I > 2σ(I)
R(int) = 0.036
θ_max = 29.7°, θ_min = 3.0°
h = −6→6
k = −15→15
l = −16→16

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**Refinement**

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.014$

$wR(F^2) = 0.034$

$S = 1.18$

3558 reflections

184 parameters

7 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma(F^2) + 0.4884P]$

where $P = (F^2 + 2F^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.002$

$\Delta \rho_{\text{max}} = 0.44$ e Å$^{-3}$

$\Delta \rho_{\text{min}} = -0.42$ 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 (Å$^2$)**

| Atom | x     | y     | z     | $U_{eq}$ | Occ. (<1) |
|------|-------|-------|-------|----------|-----------|
| I1   | 0.4470 (2) | 0.37987 (2) | 0.76749 (2) | 0.01303 (3) |           |
| F1   | 0.8875 (2) | 0.62085 (8)  | 0.83664 (8)  | 0.02138 (19) |           |
| F2   | 1.31349 (19) | 0.70566 (8)  | 1.01073 (9)  | 0.02069 (19) |           |
| C9   | 0.7732 (3)  | 0.45489 (12) | 0.90761 (12) | 0.0132 (2)   |           |
| C10  | 0.9374 (3)  | 0.56045 (13) | 0.91719 (13) | 0.0143 (3)   |           |
| C11  | 1.1591 (3)  | 0.60420 (12) | 1.00759 (13) | 0.0145 (3)   |           |
| S1   | 1.03801 (7) | 0.78475 (3)  | 0.43889 (3)  | 0.01464 (7)  |           |
| N1   | 0.7347 (3)  | 0.75457 (11) | 0.60568 (11) | 0.0147 (2)   |           |
| HN1  | 0.770 (4)   | 0.6828 (15)  | 0.5977 (19)  | 0.023 (5)*   |           |
| N2   | 0.7391 (3)  | 0.93710 (11) | 0.58449 (11) | 0.0138 (2)   |           |
| HN2  | 0.788 (4)   | 1.0008 (16)  | 0.5627 (18)  | 0.025 (5)*   |           |
| C1   | 0.8342 (3)  | 0.82603 (13) | 0.54519 (12) | 0.0136 (2)   |           |
| C2   | 0.5751 (3)  | 0.82008 (13) | 0.68510 (13) | 0.0136 (2)   |           |
| C3   | 0.5789 (3)  | 0.93718 (12) | 0.67155 (12) | 0.0129 (2)   |           |
| C4   | 0.4354 (3)  | 1.02719 (13) | 0.73639 (13) | 0.0154 (3)   |           |
| H4   | 0.437594    | 1.106246     | 0.725878     | 0.018*       |           |
| C5   | 0.2880 (3)  | 0.99734 (13) | 0.81756 (13) | 0.0157 (3)   |           |
| C6   | 0.2857 (3)  | 0.87925 (14) | 0.83004 (13) | 0.0173 (3)   |           |
| H6   | 0.183714    | 0.860585     | 0.885580     | 0.021*       |           |
| C7   | 0.4267 (3)  | 0.78842 (14) | 0.76427 (14) | 0.0176 (3)   |           |
| H7   | 0.421629    | 0.708673     | 0.773170     | 0.021*       |           |
| C8   | 0.1331 (3)  | 1.09189 (15) | 0.89265 (14) | 0.0207 (3)   |           |
| H8A  | -0.041876   | 1.052972     | 0.901230     | 0.031*       |           |
| H8B  | 0.095344    | 1.155912     | 0.854696     | 0.031*       |           |
| H8C  | 0.245015    | 1.128188     | 0.970784     | 0.031*       |           |
| O1   | 0.7691 (3)  | 0.49501 (11) | 0.54916 (12) | 0.0247 (2)   |           |
| H1AO | 0.936 (5)   | 0.503 (4)    | 0.532 (4)    | 0.023 (10)*  | 0.5       |
| H1BO | 0.594 (5)   | 0.486 (4)    | 0.516 (4)    | 0.034 (13)*  | 0.5       |
| H2O1 | 0.778 (6)   | 0.432 (2)    | 0.575 (3)    | 0.063 (9)*   |           |
### Atomic displacement parameters (Å²)

|        | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$    | $U^{13}$    | $U^{23}$    |
|--------|-------------|-------------|-------------|-------------|-------------|-------------|
| I1     | 0.01281 (5) | 0.01303 (5) | 0.01267 (5) | 0.00156 (3) | 0.00261 (3) | 0.00256 (3) |
| F1     | 0.0247 (5)  | 0.0200 (4)  | 0.0214 (4)  | −0.0027 (4) | −0.0034 (4) | 0.0133 (4)  |
| F2     | 0.0216 (4)  | 0.0163 (4)  | 0.0242 (5)  | −0.0061 (3) | −0.0011 (4) | 0.0096 (4)  |
| C9     | 0.0124 (6)  | 0.0130 (6)  | 0.0129 (6)  | 0.0018 (5)  | 0.0027 (5)  | 0.0016 (5)  |
| C10    | 0.0160 (6)  | 0.0138 (6)  | 0.0145 (6)  | 0.0027 (5)  | 0.0034 (5)  | 0.0058 (5)  |
| C11    | 0.0152 (6)  | 0.0114 (6)  | 0.0171 (6)  | 0.0000 (5)  | 0.0044 (5)  | 0.0037 (5)  |
| S1     | 0.01389 (15)| 0.01446 (15)| 0.01405 (15)| 0.00125 (12)| 0.00305 (12)| 0.00144 (12)|
| N1     | 0.0158 (6)  | 0.0114 (5)  | 0.0165 (6)  | 0.0028 (4)  | 0.0036 (5)  | 0.0031 (4)  |
| N2     | 0.0140 (5)  | 0.0117 (5)  | 0.0161 (6)  | 0.0008 (4)  | 0.0037 (4)  | 0.0039 (4)  |
| C1     | 0.0117 (6)  | 0.0133 (6)  | 0.0136 (6)  | 0.0005 (5)  | −0.0011 (5) | 0.0021 (5)  |
| C2     | 0.0126 (6)  | 0.0123 (6)  | 0.0156 (6)  | 0.0012 (5)  | 0.0011 (5)  | 0.0042 (5)  |
| C3     | 0.0112 (6)  | 0.0125 (6)  | 0.0138 (6)  | −0.0012 (5) | 0.0014 (5)  | 0.0027 (5)  |
| C4     | 0.0149 (6)  | 0.0121 (6)  | 0.0180 (7)  | 0.0014 (5)  | 0.0020 (5)  | 0.0030 (5)  |
| C5     | 0.0125 (6)  | 0.0172 (6)  | 0.0152 (6)  | 0.0013 (5)  | 0.0012 (5)  | 0.0017 (5)  |
| C6     | 0.0168 (6)  | 0.0203 (7)  | 0.0164 (6)  | 0.0014 (5)  | 0.0044 (5)  | 0.0072 (5)  |
| C7     | 0.0188 (7)  | 0.0165 (6)  | 0.0195 (7)  | 0.0031 (5)  | 0.0035 (6)  | 0.0082 (5)  |
| C8     | 0.0188 (7)  | 0.0211 (7)  | 0.0209 (7)  | 0.0039 (6)  | 0.0074 (6)  | 0.0019 (6)  |
| O1     | 0.0289 (6)  | 0.0185 (5)  | 0.0315 (6)  | 0.0046 (5)  | 0.0105 (5)  | 0.0118 (5)  |

### Geometric parameters (Å, °)

|        | C1—C9 | C3—C4 | C7—C8 | O1—H1AO | O1—H1BO | O1—H2O1 | C2—C7 |
|--------|-------|-------|-------|---------|---------|---------|--------|
| I1—C9  | 2.0981 (14) | C3—C4 | 1.3888 (19) |       |         |         | C10—C9—I1 |
| F1—C10 | 1.3424 (16) | C4—H4 | 0.9500 |         |         |         |        |
| F2—C11 | 1.3452 (16) | C4—C5 | 1.396 (2) |         |         |         |        |
| C9—C10 | 1.3882 (19) | C5—C6 | 1.404 (2) |         |         |         |        |
| C9—C11 | 1.386 (2) | C5—C8 | 1.509 (2) |         |         |         |        |
| C10—C11 | 1.383 (2) | C6—H6 | 0.9500 |         |         |         |        |
| S1—C1  | 1.7035 (15) | C6—C7 | 1.392 (2) |         |         |         |        |
| N1—HN1 | 0.830 (15) | C7—H7 | 0.9500 |         |         |         |        |
| N1—C1  | 1.3542 (19) | C8—H8A | 0.9800 |         |         |         |        |
| N1—C2  | 1.3926 (18) | C8—H8B | 0.9800 |         |         |         |        |
| N2—HN2 | 0.876 (15) | C8—H8C | 0.9800 |         |         |         |        |
| N2—C1  | 1.3557 (18) | O1—H1AO | 0.880 (18) |         |         |         |        |
| N2—C3  | 1.3905 (18) | O1—H1BO | 0.883 (18) |         |         |         |        |
| C2—C3  | 1.3975 (19) | O1—H2O1 | 0.872 (17) |         |         |         |        |
| C2—C7  | 1.387 (2) |        |         |         |         |         |        |
C10—C11—C9$^i$  
121.68 (13)  
C5—C6—H6  
118.7  
C1—N1—HN1  
124.2 (15)  
C7—C6—C5  
122.63 (14)  
C1—N1—C2  
110.28 (12)  
C7—C6—H6  
118.7  
C2—N1—HN1  
125.5 (15)  
C2—C7—C6  
121.6  
C1—N2—C3  
110.09 (12)  
C6—C7—C5  
122.63 (14)  
C3—N2—C2  
106.11 (12)  
C5—C8—H8A  
109.5  
C1—N1—C2  
110.28 (12)  
C5—C8—H8B  
109.5  
N1—C1—S1  
126.80 (11)  
C5—C8—H8C  
109.5  
N1—C1—N2  
107.02 (12)  
C6—C7—H7  
121.6  
N2—C1—S1  
126.17 (11)  
H8A—C8—H8B  
109.5  
N1—C2—C3  
106.11 (12)  
H8A—C8—H8C  
109.5  
C7—C2—N1  
132.60 (13)  
C2—C7—H7  
121.6  
C7—C2—C3  
121.28 (13)  
H1AO—O1—H2O1  
101 (3)  
N2—C3—C2  
106.49 (12)  
H1BO—O1—H2O1  
101 (3)

$^{i}$Symmetry code: (i) $-x+2, -y+1, -z+2$.

Hydrogen-bond geometry ($\AA$, °)

\begin{tabular}{lcccr}
$D_{\cdot\cdot\cdot}A$ & $D$ & $H$ & $A$ & $D_{\cdot\cdot\cdot}A$ & $D$ & $H_{\cdot\cdot\cdot}A$ \\
\hline
N1—HN1···O1 & 0.83 (2) & 2.06 (2) & 2.8763 (17) & 166 (2) \\
N2—HN2···S1$^{ii}$ & 0.88 (2) & 2.57 (2) & 3.4211 (13) & 164 (2) \\
C4—H4···I1$^{iii}$ & 0.95 & 3.03 & 3.9505 (14) & 164 \\
O1—H1AO···O1$^{iv}$ & 0.88 (2) & 1.85 (2) & 2.708 (3) & 163 (4) \\
O1—H1BO···O1$^{v}$ & 0.88 (2) & 1.89 (2) & 2.759 (3) & 167 (4) \\
O1—H2O1···I1 & 0.87 (2) & 3.16 (3) & 3.7419 (12) & 126 (2) \\
O1—H2O1···S1$^{iv}$ & 0.87 (2) & 2.65 (2) & 3.4251 (13) & 149 (3) \\
\end{tabular}

Symmetry codes: (ii) $-x+2, -y+2, -z+1$; (iii) $x, y+1, z$; (iv) $-x+2, -y+1, -z+1$; (v) $-x+1, -y+1, -z+1$.
5-Methyl-1H-1,3-benzodiazole-2-thiol–1,3,5-trifluoro-2,4,6-triiodobenzene (1/1) (MMBZIM_135F3I3B)

Crystal data

C6F3I3·C8H8N2S  
Mr = 673.98

Monoclinic, P21/c  
a = 15.191 (2) Å  
b = 5.0074 (7) Å  
c = 22.715 (3) Å  
β = 97.460 (6)°  
V = 1713.3 (4) Å³  
Z = 4

F(000) = 1232  
Dx = 2.613 Mg m⁻³

Mo Kα radiation, λ = 0.71073 Å

Cell parameters from 9964 reflections

θ = 2.4–27.5°  
µ = 5.62 mm⁻¹

T = 100 K

Needle, colourless

0.26 × 0.04 × 0.04 mm

Data collection

Bruker D8 Venture Photon 2 diffractometer  
3971 independent reflections

Radiation source: Incoatec IμS

φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2017)

θmin = 0.582, Tmax = 0.746

23258 measured reflections

Refinement

Refinement on F²  
Hydrogen site location: mixed

Least-squares matrix: full  
H atoms treated by a mixture of independent and constrained refinement

R[F² > 2σ(F²)] = 0.047  
where P = (F² + 2Fc²)/3

wR(F²) = 0.105  
S = 1.22

3971 reflections

215 parameters

1 restraint

Primary atom site location: dual

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    | Uiso* Ueq |
|----|------|------|------|-----------|
| I1 | 0.83468 (4) | 0.73539 (12) | 0.22592 (3) | 0.02269 (15) |
| I2 | 1.12973 (3) | 0.55191 (11) | 0.42927 (2) | 0.01871 (14) |
| I3 | 0.78829 (3) | −0.06392 (11) | 0.42073 (2) | 0.01766 (13) |
| F1 | 1.0160 (3) | 0.7879 (10) | 0.3105 (2) | 0.0238 (11) |
| F2 | 0.9829 (3) | 0.1538 (10) | 0.4578 (2) | 0.0217 (11) |
| F3 | 0.7534 (3) | 0.2862 (11) | 0.3013 (2) | 0.0226 (11) |
| C9 | 0.8839 (5) | 0.5360 (16) | 0.3041 (4) | 0.0161 (16) |
| C10 | 0.9672 (5) | 0.5984 (17) | 0.3334 (4) | 0.0166 (17) |
| C11 | 1.0030 (5) | 0.4712 (18) | 0.3850 (4) | 0.0190 (17) |
| C12 | 0.9504 (5) | 0.2824 (17) | 0.4079 (3) | 0.0146 (16) |
### Atomic displacement parameters (Å²)

|     | U¹¹   | U²²   | U³³   | U¹²   | U¹³   | U²³   |
|-----|-------|-------|-------|-------|-------|-------|
| I1  | 0.0241 (3) | 0.0236 (3) | 0.0196 (3) | 0.0058 (2) | 0.0002 (2) | 0.0034 (2) |
| I2  | 0.0134 (2) | 0.0228 (3) | 0.0198 (3) | -0.0017 (2) | 0.0018 (2) | -0.0022 (2) |
| I3  | 0.0150 (2) | 0.0183 (3) | 0.0204 (3) | -0.0014 (2) | 0.0050 (2) | -0.0012 (2) |
| F1  | 0.023 (3) | 0.026 (3) | 0.024 (3) | -0.008 (2) | 0.005 (2) | 0.007 (2) |
| F2  | 0.017 (2) | 0.027 (3) | 0.019 (3) | 0.000 (2) | -0.001 (2) | 0.005 (2) |
| F3  | 0.012 (2) | 0.031 (3) | 0.023 (3) | -0.002 (2) | -0.0043 (19) | 0.000 (2) |
| C9  | 0.017 (4) | 0.014 (4) | 0.017 (4) | 0.003 (3) | 0.002 (3) | -0.003 (3) |
| C10 | 0.011 (4) | 0.022 (4) | 0.018 (4) | 0.000 (3) | 0.006 (3) | 0.000 (3) |
| C11 | 0.015 (4) | 0.024 (5) | 0.017 (4) | 0.002 (3) | -0.001 (3) | -0.003 (3) |
| C12 | 0.014 (4) | 0.020 (4) | 0.009 (4) | 0.001 (3) | 0.001 (3) | -0.005 (3) |
| C13 | 0.015 (4) | 0.021 (4) | 0.017 (4) | 0.001 (3) | 0.003 (3) | 0.003 (3) |
| C14 | 0.012 (4) | 0.018 (4) | 0.017 (4) | 0.000 (3) | 0.001 (3) | -0.002 (3) |
| S1  | 0.0153 (9) | 0.0217 (11) | 0.0185 (10) | -0.0007 (8) | 0.0010 (8) | -0.0038 (8) |
| N1  | 0.017 (3) | 0.018 (4) | 0.024 (4) | -0.002 (3) | 0.002 (3) | -0.002 (3) |
| N2  | 0.010 (3) | 0.021 (4) | 0.022 (4) | 0.001 (3) | 0.001 (3) | -0.003 (3) |
| C1  | 0.019 (4) | 0.010 (4) | 0.019 (4) | 0.002 (3) | 0.002 (3) | 0.003 (3) |
| C2  | 0.017 (4) | 0.018 (4) | 0.017 (4) | -0.003 (3) | 0.005 (3) | 0.010 (3) |
| C3  | 0.016 (4) | 0.021 (4) | 0.026 (5) | -0.003 (3) | 0.008 (3) | 0.003 (4) |
| C4  | 0.016 (4) | 0.018 (4) | 0.028 (5) | -0.002 (3) | 0.003 (3) | -0.010 (4) |
| C5  | 0.020 (4) | 0.021 (4) | 0.023 (5) | -0.006 (3) | 0.005 (3) | 0.002 (4) |
| C6  | 0.017 (4) | 0.019 (4) | 0.019 (4) | -0.002 (3) | 0.001 (3) | 0.000 (3) |
| C7  | 0.009 (3) | 0.026 (5) | 0.020 (4) | 0.004 (3) | 0.000 (3) | -0.001 (4) |
| C8  | 0.021 (4) | 0.032 (5) | 0.030 (5) | 0.005 (4) | 0.010 (4) | -0.003 (4) |
| Bond  | Length (Å) | Bond  | Length (Å) | Angle (°)  |
|-------|------------|-------|------------|------------|
| I1—C9 | 2.089 (8)  | N2—HN2 | 0.84 (10)  |
| I2—C11 | 2.093 (8) | N2—C1 | 1.363 (11) |
| I3—C13 | 2.094 (8) | N2—C7 | 1.378 (11) |
| F1—C10 | 1.349 (9) | C2—C3 | 1.382 (12) |
| F2—C12 | 1.342 (9) | C2—C7 | 1.405 (11) |
| F3—C14 | 1.340 (9) | C3—H3 | 0.9500 |
| C9—C10 | 1.388 (11) | C3—C4 | 1.390 (12) |
| C9—C14 | 1.372 (12) | C4—C5 | 1.402 (11) |
| C10—C11 | 1.381 (12) | C4—C8 | 1.500 (12) |
| C11—C12 | 1.382 (12) | C5—H5 | 0.9500 |
| C12—C13 | 1.399 (11) | C5—C6 | 1.384 (12) |
| C13—C14 | 1.391 (11) | C6—H6 | 0.9500 |
| S1—C1 | 1.688 (8)  | C6—C7 | 1.396 (12) |
| N1—HN1 | 0.86 (2)   | C8—H8A | 0.9800 |
| N1—C1 | 1.365 (11) | C8—H8B | 0.9800 |
| N1—C2 | 1.391 (11) | C8—H8C | 0.9800 |

Geometric parameters (Å, °)
Hydrogen-bond geometry (Å, °)

| D—H···A | D—H  | H···A | D···A | D—H···A |
|---------|-------|-------|-------|---------|
| N1—H1···S1i | 0.86 (2) | 2.57 (2) | 3.427 (7) | 174 (9) |
| N2—H2···I2ii | 0.84 (10) | 3.03 (10) | 3.657 (7) | 133 (8) |
| C3—H3···I3iii | 0.95 | 3.12 | 4.035 (9) | 163 |
| C6—H6···I1iv | 0.95 | 3.14 | 3.927 (8) | 142 |

Symmetry codes: (i) −x+1, −y+3, −z+1; (ii) x−1, y+1, z; (iii) x, y+1, z; (iv) −x+1, y−1/2, −z+1/2.

1,3-Benzoxazole-2-thiol–1,2,3,4-tetrafluoro-5,6-diiodobenzene (1/1) (MBZOX_12F4DIB)

Crystal data

C₆F₄I₂·C₇H₅NOS

$M_r = 553.04$

Monoclinic, $P2_1/n$

$a = 13.7789$ (12) Å

$b = 4.4129$ (4) Å

$c = 25.252$ (2) Å

$\beta = 96.337$ (3)°

$V = 1526.0$ (2) Å³

$Z = 4$

$D(000) = 1024$

$D_x = 2.407$ Mg m⁻³

$\lambda (Mo K\alpha) = 0.71073$ Å

Cell parameters from 5954 reflections

$\theta = 3.0–26.8$°

$\mu = 4.30$ mm⁻¹

$T = 100$ K

Needle, colourless

0.46 × 0.06 × 0.02 mm

Data collection

Bruker D8 Venture Photon 2

diffractometer

Radiation source: Incoatec IµS

$\phi$ and $\omega$ scans

Absorption correction: multi-scan

(SADABS; Bruker, 2017)

$T_{min} = 0.578$, $T_{max} = 0.745$

12498 measured reflections

3210 independent reflections

Acta Cryst. (2022), C78, 702-715
2510 reflections with $I > 2\sigma(I)$  
$R_{int} = 0.066$  
$\theta_{\text{max}} = 26.8^\circ$, $\theta_{\text{min}} = 3.0^\circ$

**Refinement**

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.047$

$wR(F^2) = 0.087$

$S = 1.11$

3210 reflections

203 parameters

0 restraints

Primary atom site location: dual

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/\left[\sigma^2(F_o^2) + (0.0191P)^2 + 7.3427P\right]$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\text{max}} = 1.58 \text{ e Å}^{-3}$

$\Delta\rho_{\text{min}} = -1.52 \text{ e Å}^{-3}$

**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 ($\AA^2$)**

| x   | y   | z   | U_{iso}/U_{eq} |
|-----|-----|-----|----------------|
| 1   | 0.30796 (3) | −0.08467 (10) | 0.67864 (2) | 0.02151 (14) |
| 2   | 0.44314 (3) | −0.05194 (10) | 0.81380 (2) | 0.01770 (13) |
| F1  | 0.6245 (3)  | 0.3640 (9)    | 0.80387 (17) | 0.0263 (10) |
| F2  | 0.6876 (3)  | 0.6765 (10)   | 0.7230 (2)   | 0.0339 (11)  |
| F3  | 0.5903 (3)  | 0.6411 (10)   | 0.62370 (19) | 0.0350 (11)  |
| F4  | 0.4320 (3)  | 0.2897 (10)   | 0.60463 (17) | 0.0298 (10)  |
| C8  | 0.4399 (5)  | 0.1468 (15)   | 0.6957 (3)   | 0.0158 (15)  |
| C9  | 0.4896 (5)  | 0.1682 (14)   | 0.7472 (3)   | 0.0158 (15)  |
| C10 | 0.5749 (5)  | 0.3417 (15)   | 0.7561 (3)   | 0.0180 (16)  |
| C11 | 0.6078 (5)  | 0.4963 (15)   | 0.7141 (3)   | 0.0250 (17)  |
| C12 | 0.5583 (6)  | 0.4836 (16)   | 0.6629 (3)   | 0.0273 (18)  |
| C13 | 0.4767 (5)  | 0.3023 (16)   | 0.6542 (3)   | 0.0209 (16)  |
| S1  | 0.07783 (13)| 0.0388 (4)    | 0.57938 (7)  | 0.0193 (4)   |
| O1  | 0.2157 (3)  | 0.4196 (10)   | 0.55679 (19) | 0.0193 (11)  |
| N1  | 0.1100 (4)  | 0.3227 (13)   | 0.4874 (2)   | 0.0152 (13)  |
| HN1 | 0.068 (6)   | 0.228 (18)    | 0.466 (3)    | 0.03 (2)*    |
| C1  | 0.1325 (5)  | 0.2629 (14)   | 0.5389 (3)   | 0.0147 (14)  |
| C2  | 0.1772 (5)  | 0.5252 (15)   | 0.4695 (3)   | 0.0158 (14)  |
| C3  | 0.1868 (5)  | 0.6569 (16)   | 0.4216 (3)   | 0.0219 (16)  |
| H3  | 0.141810    | 0.617405      | 0.391053     | 0.026*       |
| C4  | 0.2659 (5)  | 0.8527 (15)   | 0.4193 (3)   | 0.0212 (16)  |
| H4  | 0.274897    | 0.948201      | 0.386523     | 0.025*       |
| C5  | 0.3316 (6)  | 0.9111 (16)   | 0.4637 (3)   | 0.0229 (16)  |
| H5  | 0.384448    | 1.045851      | 0.460649     | 0.028*       |
| C6  | 0.3218 (5)  | 0.7761 (15)   | 0.5130 (3)   | 0.0193 (16)  |
| H6  | 0.366596    | 0.813408      | 0.543631     | 0.023*       |
| C7  | 0.2429 (5)  | 0.5852 (14)   | 0.5140 (3)   | 0.0142 (14)  |
### Atomic displacement parameters (Å²)

|  | U₁₁  | U₂₂  | U₃₃  | U₁₂  | U₁₃  | U₂₃  |
|---|------|------|------|------|------|------|
| I1 | 0.0210 (3) | 0.0213 (2) | 0.0210 (3) | −0.0009 (2) | −0.0034 (2) | −0.0037 (2) |
| I2 | 0.0223 (2) | 0.0177 (2) | 0.0130 (2) | 0.0024 (2) | 0.00165 (18) | −0.00026 (18) |
| F1 | 0.018 (2) | 0.031 (2) | 0.027 (3) | 0.0013 (18) | −0.0068 (18) | −0.0058 (19) |
| F2 | 0.021 (2) | 0.029 (2) | 0.052 (3) | −0.0102 (19) | 0.010 (2) | −0.003 (2) |
| F3 | 0.043 (3) | 0.027 (2) | 0.039 (3) | −0.001 (2) | 0.020 (2) | 0.012 (2) |
| F4 | 0.041 (3) | 0.032 (2) | 0.015 (2) | 0.005 (2) | −0.001 (2) | 0.0042 (19) |
| C8 | 0.011 (3) | 0.021 (3) | 0.016 (4) | 0.005 (3) | 0.004 (3) | −0.003 (3) |
| C9 | 0.016 (3) | 0.013 (3) | 0.020 (4) | 0.005 (3) | 0.007 (3) | 0.002 (3) |
| C10 | 0.016 (4) | 0.015 (3) | 0.023 (4) | 0.004 (3) | −0.001 (3) | −0.006 (3) |
| C11 | 0.024 (4) | 0.014 (4) | 0.038 (5) | 0.002 (3) | 0.010 (4) | −0.002 (3) |
| C12 | 0.037 (5) | 0.021 (4) | 0.027 (4) | 0.005 (3) | 0.019 (4) | 0.013 (3) |
| C13 | 0.027 (4) | 0.020 (3) | 0.016 (4) | 0.010 (3) | 0.001 (3) | 0.006 (3) |
| S1 | 0.0249 (9) | 0.0194 (9) | 0.0132 (9) | −0.0053 (7) | 0.0007 (7) | 0.0000 (7) |
| O1 | 0.023 (3) | 0.017 (2) | 0.017 (3) | 0.000 (2) | −0.003 (2) | 0.003 (2) |
| N1 | 0.021 (3) | 0.013 (3) | 0.010 (3) | 0.000 (2) | −0.002 (3) | −0.003 (2) |
| C1 | 0.013 (3) | 0.012 (3) | 0.019 (4) | 0.004 (3) | 0.003 (3) | −0.002 (3) |
| C2 | 0.021 (4) | 0.016 (3) | 0.011 (4) | 0.000 (3) | 0.004 (3) | −0.002 (3) |
| C3 | 0.026 (4) | 0.021 (4) | 0.018 (4) | 0.000 (3) | 0.000 (3) | −0.009 (3) |
| C4 | 0.029 (4) | 0.016 (3) | 0.019 (4) | 0.002 (3) | 0.005 (3) | 0.004 (3) |
| C5 | 0.029 (4) | 0.018 (4) | 0.022 (4) | 0.000 (3) | 0.008 (3) | 0.008 (3) |
| C6 | 0.017 (4) | 0.018 (4) | 0.021 (4) | −0.002 (3) | −0.003 (3) | −0.003 (3) |
| C7 | 0.022 (4) | 0.012 (3) | 0.010 (3) | −0.003 (3) | 0.005 (3) | −0.002 (3) |

### Geometric parameters (Å, °)

|   | I1—C8  | O1—C7  | O1—C1  |
|---|--------|--------|--------|
| I2 | 2.088 (7) | 1.390 (8) |
| F1 | 2.101 (7) | N1—HN1 0.85 (8) |
| F2 | 1.322 (8) | N1—C1 1.330 (9) |
| F3 | 1.355 (8) | N1—C2 1.396 (9) |
| F4 | 1.323 (8) | C2—C3 1.364 (10) |
| C8 | 1.333 (8) | C2—C7 1.387 (9) |
| C9 | 1.406 (10) | C3—H3 0.9500 |
| C10 | 1.394 (10) | C3—C4 1.397 (10) |
| C11 | 1.400 (9) | C4—H4 0.9500 |
| C12 | 1.380 (11) | C4—C5 1.385 (10) |
| C13 | 1.396 (11) | C5—H5 0.9500 |
| S1 | 1.378 (11) | C5—C6 1.399 (10) |
| O1 | 1.662 (7) | C6—H6 0.9500 |
| C9 | 1.372 (8) | C6—C7 1.378 (9) |
| C8 | 123.2 (5) | O1—C1—S1 121.1 (5) |
| C10 | 117.8 (5) | N1—C1—S1 130.3 (5) |
| C12 | 118.9 (6) | N1—C1—O1 108.6 (6) |
| C13 | 123.3 (5) | C3—C2—N1 133.8 (6) |
| C10 | 116.7 (5) | C3—C2—C7 121.2 (6) |
C10—C9—C8 120.0 (6)  C7—C2—N1 104.9 (6)
F1—C10—C9  121.8 (7)  C2—C3—H3  121.6
F1—C10—C11 118.9 (6)  C2—C3—C4  116.9 (6)
C11—C10—C9 119.3 (7)  C3—C4—H4  119.1
C11—C10—C12 118.9 (7)  C5—C4—H4  119.1
C10—C11—C12 121.6 (7)  C3—C4—C5  119.3
F3—C12—C11 120.2 (7)  C4—C5—H5  121.4 (7)
F3—C12—C13 121.2 (7)  C4—C5—C6  119.3
C13—C12—C11 118.6 (7)  C6—C5—H5  122.2
F4—C13—C8 121.1 (6)  C5—C6—H6  115.5 (6)
F4—C13—C12 117.3 (7)  C7—C6—C5  115.5 (6)
C12—C13—C8 121.5 (7)  C7—C6—H6  122.2
C1—O1—C7 107.2 (5)  C2—C7—O1  108.7 (5)
C1—N1—HN1 126 (6)  C6—C7—O1  128.1 (6)
C1—N1—C2 110.5 (6)  C6—C7—C2  123.2 (6)
C2—N1—HN1 123 (6)
I1—C8—C9—I2  4.0 (8)  C13—C8—C9—I2 179.9 (5)
I1—C8—C9—C10 −176.3 (5)  C13—C8—C9—C10 −0.5 (10)
I1—C8—C13—F4 −3.7 (9)  N1—C2—C3—C4 −179.9 (7)
I1—C8—C13—C12 173.9 (5)  N1—C2—C3—C4 −179.7 (7)
I2—C9—C10—F1 0.7 (8)  N1—C2—C3—C4 −179.8 (5)
I2—C9—C10—C11 −178.7 (5)  N1—C2—C3—C4 −0.8 (7)
F1—C10—C11—F2 −2.7 (10)  C2—C3—C4—C5  0.4 (10)
F1—C10—C11—C12 −179.5 (6)  C2—C3—C4—C5  0.4 (10)
F2—C11—C12—F3 1.3 (10)  C2—C3—C4—C5  0.4 (10)
F2—C11—C12—C13 −179.3 (6)  C2—C3—C4—C5  0.4 (10)
F3—C12—C13—F4 0.7 (10)  C2—C3—C4—C5  0.4 (10)
F3—C12—C13—C8 −177.0 (6)  C2—C3—C4—C5  0.4 (10)
C8—C9—C10—F1 −179.0 (6)  C3—C2—C3—C4  179.6 (6)
C8—C9—C10—C11 1.6 (10)  C3—C2—C3—C4  179.6 (6)
C9—C8—C13—F4 −179.8 (6)  C3—C2—C3—C4  179.6 (6)
C9—C8—C13—C12 −2.2 (10)  C3—C2—C3—C4  179.6 (6)
C9—C10—C11—F2 176.7 (6)  C5—C6—C7—O1  179.3 (6)
C9—C10—C11—C12 −0.1 (10)  C5—C6—C7—C2  179.3 (6)
C10—C11—C12—C13 −2.5 (11)  C7—O1—C1—S1  −179.7 (5)
C11—C12—C13—F4 −178.7 (6)  C7—O1—C1—N1  1.2 (7)
C11—C12—C13—C8  3.7 (11)  C7—C2—C3—C4  0.3 (10)

Hydrogen-bond geometry (Å, °)

|  | D—H···A |  | D—H···A |  | D···A |  | D—H···A |
|---|---|---|---|---|---|---|---|
| N1—HN1···S1i | 0.85 (8) | 2.50 (8) | 3.335 (6) | 167 (8) |
| C3—H3···I2ii | 0.95 | 3.19 | 4.108 (7) | 162 |

Symmetry codes: (i) −x, −y, −z+1; (ii) x−1/2, −y+1/2, z−1/2.
1,3-Benzoxazole-2-thiol–1,2,3,5-tetrafluoro-4,6-diiodobenzene (1/1) (MBZOX_13F4DIB)

Crystal data

C₆F₄I₂·C₇H₅NOS  

F(000) = 1024  

Dₓ = 2.437 Mg m⁻³  

Monoclinic, P2₁/c  

Mo Kα radiation, λ = 0.71073 Å  

Cell parameters from 9330 reflections  

θ = 2.7–30.6°  

µ = 4.35 mm⁻¹  

T = 100 K  

Needle, colourless  

0.23 × 0.12 × 0.09 mm

Data collection

Bruker D8 Venture Photon 2 diffractometer  

4625 independent reflections  

4199 reflections with I > 2σ(I)

Absorption correction: multi-scan  

Tmin = 0.541, Tmax = 0.746  

39610 measured reflections

Refinement

Refinement on F²  

Hydrogen site location: mixed  

H atoms treated by a mixture of independent and constrained refinement

Least-squares matrix: full  

wR(F²) = 0.048  

S = 1.16

4625 reflections  

4625 independent reflections  

4199 reflections with I > 2σ(I)

203 parameters  

0 restraints  

Primary atom site location: dual  

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      | Ueq   |
|----|--------|--------|--------|-------|
| I1 | 0.71955 (2) | -0.00881 (3) | 0.06483 (2) | 0.01983 (4) |
| I2 | 0.36910 (2) | 0.55990 (4) | 0.09449 (2) | 0.02789 (5) |
| F1 | 0.51550 (9) | 0.1796 (3) | 0.04571 (6) | 0.0245 (3) |
| F2 | 0.50261 (12) | 0.8071 (4) | 0.20979 (7) | 0.0336 (4) |
| F3 | 0.67787 (12) | 0.7060 (4) | 0.24932 (7) | 0.0422 (4) |
| F4 | 0.77333 (10) | 0.3490 (4) | 0.18773 (7) | 0.0352 (4) |
| C8 | 0.64548 (14) | 0.2498 (5) | 0.11554 (10) | 0.0173 (4) |
| C9 | 0.55516 (14) | 0.3056 (5) | 0.09648 (10) | 0.0171 (4) |
| C10 | 0.50419 (15) | 0.4888 (5) | 0.12703 (10) | 0.0187 (4) |
| C11 | 0.54714 (17) | 0.6213 (5) | 0.17885 (11) | 0.0223 (5) |
C12 0.63676 (18) 0.5704 (6) 0.19943 (10) 0.0250 (5)
C13 0.68526 (16) 0.3866 (6) 0.16770 (11) 0.0226 (5)
S1 0.85453 (4) 0.94524 (12) 0.49409 (2) 0.01660 (10)
O1 0.82568 (10) 0.5576 (3) 0.40484 (7) 0.0155 (3)
N1 0.96873 (12) 0.6582 (4) 0.43253 (8) 0.0150 (3)
HN1 1.0171 (19) 0.742 (7) 0.4529 (13) 0.024 (7)*
C1 0.88629 (14) 0.7182 (5) 0.44345 (9) 0.0151 (4)
C2 0.96367 (14) 0.4504 (5) 0.38611 (9) 0.0140 (4)
C3 1.02660 (14) 0.3138 (5) 0.35766 (10) 0.0173 (4)
H3 1.08876 0.35487 0.368581 0.021*
C4 0.99394 (15) 0.1129 (5) 0.3126 (10) 0.0176 (4)
H4 1.035003 0.013545 0.291629 0.021*
C5 0.90240 (15) 0.0533 (5) 0.29583 (10) 0.0182 (4)
H5 0.882849 −0.085313 0.264531 0.022*
C6 0.83913 (14) 0.1930 (5) 0.32450 (10) 0.0177 (4)
H6 0.776786 0.154860 0.313733 0.021*
C7 0.87317 (14) 0.3897 (5) 0.36943 (9) 0.0141 (4)

Atomic displacement parameters (Å²)

Geometric parameters (Å, °)

I1—C8 2.089 (2) O1—C7 1.389 (3)
I2—C10 2.080 (2) N1—HN1 0.88 (3)
| Bond          | Distance (Å) | Bond          | Distance (Å) | Bond          | Distance (Å) |
|--------------|--------------|--------------|--------------|--------------|--------------|
| F1—C9        | 1.339 (3)    | N1—C1        | 1.343 (3)    | C1—O1        | 1.224 (2)    |
| F2—C11       | 1.339 (3)    | N1—C2        | 1.396 (3)    | C1—N1—C2     | 115.6 (3)    |
| F3—C12       | 1.347 (3)    | C2—C3        | 1.383 (3)    | C2—C3—N1     | 127.4 (3)    |
| F4—C13       | 1.346 (3)    | C2—C7        | 1.386 (3)    | C2—C7—O1     | 110.2 (3)    |
| C8—C9        | 1.386 (3)    | C3—H3        | 0.9500       | C3—H3—C2     | 125.5 (3)    |
| C8—C13       | 1.384 (3)    | C3—C4        | 1.393 (3)    | C3—C4—C6     | 117.1 (3)    |
| C9—C10       | 1.387 (3)    | C4—H4        | 0.9500       | C4—H4—C2     | 123.5 (3)    |
| C10—C11      | 1.385 (3)    | C4—C5        | 1.399 (3)    | C4—C5—C6     | 120.1 (3)    |
| C11—C12      | 1.378 (4)    | C5—H5        | 0.9500       | C5—H5—C4     | 123.1 (3)    |
| C12—C13      | 1.382 (4)    | C5—C6        | 1.396 (3)    | C5—C6—C7     | 120.0 (3)    |
| S1—C1        | 1.666 (2)    | C6—H6        | 0.9500       | C6—H6—C5     | 124.5 (3)    |
| O1—C1        | 1.360 (2)    | C6—C7        | 1.377 (3)    | C6—C7—C8     | 121.4 (3)    |

| Angle          | Value (°) | Angle          | Value (°) | Angle          | Value (°) |
|----------------|-----------|----------------|-----------|----------------|-----------|
| C9—C8—I1      | 121.04 (17)| O1—C1—S1      | 121.49 (15)|         |           |
| C13—C8—I1     | 121.61 (17)| N1—C1—O1      | 108.82 (18)|         |           |
| C13—C8—C9     | 117.2 (2)  | N1—C1—S1      | 129.70 (17)|         |           |
| F1—C9—C8      | 118.4 (2)  | C3—C2—N1      | 133.9 (2) |         |           |
| F1—C9—C10     | 118.4 (2)  | C3—C2—C7      | 121.1 (2) |         |           |
| C8—C9—C10     | 123.2 (2)  | C7—C2—N1      | 104.99 (18)|         |           |
| C9—C10—I2     | 120.27 (17)| C2—C3—H3      | 121.8     |         |           |
| C11—C10—I2    | 122.39 (17)| C2—C3—C4      | 116.4 (2) |         |           |
| C11—C10—C9    | 117.3 (2)  | C4—C3—H3      | 121.8     |         |           |
| F2—C11—C10    | 120.5 (2)  | C3—C4—H4      | 119.1     |         |           |
| F2—C11—C12    | 118.2 (2)  | C3—C4—C5      | 121.9 (2) |         |           |
| C12—C11—C10   | 121.2 (2)  | C5—C4—H4      | 119.1     |         |           |
| F3—C12—C11    | 120.4 (2)  | C4—C5—H5      | 119.3     |         |           |
| F3—C12—C13    | 119.9 (2)  | C6—C5—C4      | 121.5 (2) |         |           |
| C11—C12—C13   | 119.7 (2)  | C6—C5—H5      | 119.3     |         |           |
| F4—C13—C8     | 120.2 (2)  | C5—C6—H6      | 122.2     |         |           |
| F4—C13—C12    | 118.5 (2)  | C7—C6—C5      | 115.5 (2) |         |           |
| C12—C13—C8    | 121.3 (2)  | C7—C6—H6      | 122.2     |         |           |
| C1—O1—C7      | 107.32 (16)| C2—C7—O1      | 108.94 (18)|         |           |
| C1—N1—HN1     | 122.5 (19) | C6—C7—O1      | 127.43 (19)|         |           |
| C1—N1—C2      | 109.92 (18)| C6—C7—C2      | 123.6 (2) |         |           |
| C2—N1—HN1     | 127.6 (19) |               |           |         |           |

| Bond          | Torsion (°) | Bond          | Torsion (°) | Bond          | Torsion (°) |
|--------------|-------------|--------------|-------------|--------------|-------------|
| I1—C8—C9—F1 | 2.6 (3)     | C13—C8—C9—F1| 179.0 (2)  |           |             |
| I1—C8—C9—C10| −176.56 (17)| C13—C8—C9—C10| −0.1 (3) |         |             |
| I1—C8—C13—F4| −1.6 (3)    | N1—C2—C3—C4 | 179.9 (2)  |         |             |
| I1—C8—C13—C12| 176.58 (18)| N1—C2—C7—O1 | 0.2 (2)    |         |             |
| I2—C10—C11—F2| −1.1 (3)    | N1—C2—C7—C6 | −180.0 (2) |         |             |
| I2—C10—C11—C12| 180.00 (18)| C1—O1—C7—C2 | −0.6 (2)  |         |             |
| F1—C9—C10—I2 | 0.6 (3)     | C1—O1—C7—C6 | 179.6 (2)  |         |             |
| F1—C9—C10—C11| −178.8 (2)  | C1—N1—C2—C3 | −179.9 (2) |         |             |
| F2—C11—C12—F3| −0.2 (3)    | C1—N1—C2—C7 | 0.3 (2)    |         |             |
| F2—C11—C12—C13| −178.2 (2)  | C2—N1—C1—S1 | 179.30 (17)|         |             |
| F3—C12—C13—F4| −0.3 (4)    | C2—N1—C1—O1 | −0.7 (2)   |         |             |
| F3—C12—C13—C8| −178.4 (2)  | C2—C3—C4—C5 | 0.3 (3)    |         |             |
C8—C9—C10—I2 179.72 (17) C3—C2—C7—O1 79.62 (19)
C8—C9—C10—C11 0.3 (3) C3—C2—C7—C6 0.2 (3)
C9—C8—C13—F4 178.0 (2) C4—C5—C6—C7 0.0 (3)
C9—C8—C13—C12 0.1 (3) C5—C6—C7—O1 179.9 (2)
C9—C10—C11—F2 178.3 (2) C5—C6—C7—C2 0.1 (3)
C9—C10—C11—C12 −0.6 (3) C7—O1—C1—S1 79.20 (15)
C10—C11—C12—F3 178.7 (2) C7—O1—C1—N1 0.8 (2)
C10—C11—C12—C13 0.7 (4) C7—C2—C3—C4 −0.4 (3)
C11—C12—C13—F4 177.8 (2) C7—C2—C3—C4 −0.4 (3)
C11—C12—C13—C8 0.9 (4)

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| N1—H1N1···S1i | 0.88 (3) | 2.52 (3) | 3.3906 (19) | 172 (3) |
| C3—H3···I1ii | 0.95 | 3.10 | 4.030 (2) | 166 |

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

1,3-Benzoxazole-2-thiol–1,2,4,5-tetrafluoro-3,6-diiodobenzene (2/1) (2MBZOX_14F4DIB)

Crystal data

C6F4I2·2C7H5NOS

F(000) = 1336

D, = 2.013 Mg m⁻³

Mo Kα radiation, λ = 0.71073 Å

Cell parameters from 9971 reflections

θ = 2.4–28.7°

µ = 2.94 mm⁻¹

T = 100 K

Tabular, colourless

0.29 × 0.12 × 0.03 mm

Data collection

Bruker D8 Venture Photon 2 diffractometer

Radiation source: Incoatec IμS

φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2017)

T = 0.637, T = 0.746

25197 measured reflections

Refinement

Refinement on F²

Least-squares matrix: full

R[F² > 2σ(F²)] = 0.028

wR(F²) = 0.060

S = 1.32

2950 reflections

149 parameters

0 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

Δρ max = 1.54 e Å⁻³

Δρ min = −1.15 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    | Uiso*/Ueq |
|---|------|------|------|-----------|
| I1| 0.64136 (2) | 0.39079 (5) | 0.46092 (2) | 0.01937 (7) |
| F1| 0.71102 (7) | 0.7954 (5) | 0.60381 (11) | 0.0276 (5) |
| F2| 0.70741 (7) | 0.4164 (5) | 0.36823 (11) | 0.0273 (5) |
| C8| 0.70659 (11) | 0.6029 (8) | 0.48500 (19) | 0.0189 (6) |
| C9| 0.72975 (11) | 0.7707 (8) | 0.55172 (19) | 0.0194 (7) |
| C10| 0.72770 (12) | 0.5825 (8) | 0.43364 (19) | 0.0201 (7) |
| S1| 0.54594 (3) | 0.0194 (2) | 0.43700 (5) | 0.01964 (17) |
| O1| 0.49241 (8) | 0.4059 (5) | 0.32770 (12) | 0.0181 (5) |
| N1| 0.46292 (10) | 0.3062 (7) | 0.41195 (16) | 0.0180 (6) |
| HN1| 0.4609 (14) | 0.224 (10) | 0.452 (2) | 0.028 (11)* |
| C1| 0.49912 (12) | 0.2491 (8) | 0.39283 (18) | 0.0182 (6) |
| C2| 0.43102 (12) | 0.5092 (8) | 0.35848 (18) | 0.0187 (6) |
| C3| 0.38827 (12) | 0.6362 (8) | 0.35038 (19) | 0.0227 (7) |
| H3| 0.374567 | 0.592471 | 0.385578 | 0.027* |
| C4| 0.36660 (12) | 0.8320 (8) | 0.2875 (2) | 0.0253 (7) |
| H4| 0.337264 | 0.925746 | 0.279739 | 0.030* |
| C5| 0.38649 (13) | 0.8950 (8) | 0.2355 (2) | 0.0251 (7) |
| H5| 0.370406 | 1.030939 | 0.193581 | 0.030* |
| C6| 0.42930 (12) | 0.7643 (8) | 0.24340 (19) | 0.0224 (7) |
| H6| 0.443055 | 0.805267 | 0.208135 | 0.027* |
| C7| 0.45026 (11) | 0.5714 (7) | 0.30592 (18) | 0.0181 (6) |

Atomic displacement parameters (Å²)

|   | U¹¹ | U²² | U³³ | U¹² | U¹³ | U²³ |
|---|-----|-----|-----|-----|-----|-----|
| I1| 0.01700 (11) | 0.01887 (11) | 0.02288 (11) | −0.00041 (8) | 0.00889 (8) | 0.00070 (8) |
| F1| 0.0248 (10) | 0.0379 (12) | 0.0253 (10) | −0.0054 (9) | 0.0154 (9) | −0.0045 (9) |
| F2| 0.0246 (10) | 0.0342 (12) | 0.0238 (10) | −0.0064 (9) | 0.0107 (9) | −0.0095 (9) |
| C8| 0.0180 (15) | 0.0161 (15) | 0.0231 (16) | −0.0009 (12) | 0.0092 (13) | 0.0014 (13) |
| C9| 0.0178 (15) | 0.0229 (17) | 0.0198 (16) | 0.0016 (13) | 0.0101 (13) | 0.0002 (13) |
| C10| 0.0196 (16) | 0.0197 (16) | 0.0190 (15) | 0.0010 (13) | 0.0061 (13) | −0.0027 (13) |
| S1| 0.0185 (4) | 0.0201 (4) | 0.0209 (4) | −0.0009 (3) | 0.0089 (3) | 0.0015 (3) |
| O1| 0.0198 (11) | 0.0198 (12) | 0.0159 (10) | −0.0014 (9) | 0.0087 (9) | 0.0002 (9) |
| N1| 0.0202 (14) | 0.0190 (14) | 0.0166 (13) | 0.0002 (11) | 0.0092 (11) | 0.0030 (11) |
| C1| 0.0217 (16) | 0.0163 (16) | 0.0172 (15) | −0.0055 (13) | 0.0085 (13) | −0.0038 (12) |
| C2| 0.0239 (17) | 0.0140 (15) | 0.0164 (15) | −0.0035 (13) | 0.0064 (13) | −0.0009 (12) |
| C3| 0.0219 (16) | 0.0246 (18) | 0.0220 (16) | 0.0004 (14) | 0.0094 (14) | −0.0018 (14) |
| C4| 0.0212 (17) | 0.0241 (19) | 0.0289 (18) | −0.0004 (14) | 0.0088 (15) | −0.0016 (15) |
| C5| 0.0274 (18) | 0.0215 (17) | 0.0206 (16) | −0.0003 (15) | 0.0041 (14) | 0.0016 (14) |
### Geometric parameters (Å, °)

| Bond/Cond | Length/Distance | Angle |  
|-----------|-----------------|-------|
| I1—C8     | 2.092 (3)       |       |
| F1—C9     | 1.346 (4)       |       |
| F2—C10    | 1.347 (4)       |       |
| C8—C9     | 1.379 (5)       |       |
| C8—C10    | 1.388 (4)       |       |
| C9—C10    | 1.385 (5)       |       |
| S1—C1     | 1.670 (3)       |       |
| O1—C1     | 1.352 (4)       |       |
| O1—C7     | 1.394 (4)       |       |
| N1—HN1    | 0.87 (4)        |       |
| N1—C1     | 1.339 (4)       |       |
| C9—C8—I1  | 121.3 (2)       |       |
| C9—C8—C10 | 117.8 (3)       |       |
| C10—C8—I1 | 120.9 (2)       |       |
| F1—C9—C8  | 120.2 (3)       |       |
| F1—C9—C10 | 118.8 (3)       |       |
| C8—C9—I1  | 121.1 (3)       |       |
| C8—C9—C10 | 120.7 (3)       |       |
| F2—C10—C8 | 120.7 (3)       |       |
| F2—C10—C9 | 118.1 (3)       |       |
| C9—C10—C8 | 121.2 (3)       |       |
| C1—O1—C7  | 107.4 (2)       |       |
| C1—N1—HN1 | 123 (3)         |       |
| C1—N1—C2  | 109.7 (3)       |       |
| C2—N1—HN1 | 128 (3)         |       |
| O1—C1—S1  | 122.1 (2)       |       |
| N1—C1—S1  | 128.6 (3)       |       |
| N1—C1—O1  | 109.2 (3)       |       |
| C3—C2—N1  | 133.4 (3)       |       |

### Bond/Cond Length/Distances

| Bond/Cond | Distance |  
|-----------|----------|
| C6        | 0.0274 (18) |
| C7        | 0.0185 (15) |
| C6        | 0.0226 (18) |
| C7        | 0.0158 (16) |
| C6        | 0.0174 (16) |
| C7        | 0.0194 (15) |
| C6        | 0.0048 (14) |
| C7        | 0.0034 (12) |
| C6        | 0.0097 (14) |
| C7        | 0.0073 (13) |
| C6        | −0.0026 (12) |
| C7        | 0.0001 (13) |
C1—O1—C7—C6 179.8 (3) C7—C2—C3—C4 −1.0 (5)

C1—N1—C2—C3 178.6 (4)

Symmetry code: (i) −x+3/2, −y+3/2, −z+1.

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|-----|-------|-------|---------|
| N1—H1N1···S1ii | 0.87 (4) | 2.45 (4) | 3.316 (3) | 178 (4) |
| C3—H3···I1iii | 0.95 | 3.16 | 4.066 (3) | 159 |

Symmetry codes: (ii) −x+1, −y, −z+1; (iii) −x+1, −y+1, −z+1.

1,3-Benzoxazole-2-thiol–1,3,5-trifluoro-2,4,6-triiodobenzene (1/1) (MBZOX_135F3I3B)

Crystal data

C6F3I3·C7H5NOS
Mr = 660.94
Monoclinic, P21/c
a = 14.9295 (7) Å
b = 4.6119 (2) Å
c = 23.5065 (12) Å
β = 92.548 (2)°
V = 1616.90 (13) Å3
Z = 4

F(000) = 1200
Dx = 2.715 Mg m−3
Mo Kα radiation, λ = 0.71073 Å

Cell parameters from 9982 reflections
θ = 2.7–26.5°
µ = 5.96 mm−1
T = 100 K

Data collection

Bruker D8 Venture Photon 2 diffractometer
Radiation source: Incoatec IμS
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2017)
Tmin = 0.551, Tmax = 0.745
19413 measured reflections
3348 independent reflections
2845 reflections with I > 2σ(I)
Rint = 0.050
θmax = 26.6°, θmin = 2.2°

Refinement

Refinement on F2
Least-squares matrix: full
R[F2 > 2σ(F2)] = 0.029
wr(F2) = 0.061
S = 1.22
3348 reflections
203 parameters
0 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

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\textsubscript{eq} |
|-----------|-----------|-----------|---------------------|
| I1        | 0.70807 (2) | 0.45232 (7) | 0.55419 (2) | 0.01748 (9) |
| I2        | 0.67869 (2) | 1.23515 (8) | 0.76030 (2) | 0.02173 (10) |
| I3        | 0.36351 (2) | 1.08539 (8) | 0.59175 (2) | 0.02080 (10) |
| F1        | 0.75694 (18) | 0.8001 (7) | 0.66897 (14) | 0.0224 (7) |
| F2        | 0.4884 (2) | 1.2983 (7) | 0.69902 (14) | 0.0233 (7) |
| F3        | 0.50893 (19) | 0.6840 (7) | 0.54104 (13) | 0.0216 (7) |
| C8        | 0.6336 (3) | 0.7273 (11) | 0.6051 (2) | 0.0148 (10) |
| C9        | 0.6700 (3) | 0.8566 (12) | 0.6536 (2) | 0.0171 (11) |
| C10       | 0.6230 (3) | 1.0473 (12) | 0.6861 (2) | 0.0172 (11) |
| C11       | 0.5357 (3) | 1.1100 (11) | 0.6684 (2) | 0.0182 (11) |
| C12       | 0.4950 (3) | 0.9888 (12) | 0.6197 (2) | 0.0161 (11) |
| C13       | 0.5456 (3) | 0.8001 (11) | 0.5888 (2) | 0.0150 (11) |
| S1        | 0.85264 (8) | 0.0073 (3) | 0.48739 (6) | 0.0189 (3) |
| O1        | 0.8417 (2) | 0.3886 (8) | 0.40283 (15) | 0.0173 (8) |
| N1        | 0.9806 (3) | 0.3173 (10) | 0.43450 (19) | 0.0160 (9) |
| HN1       | 1.021 (4) | 0.235 (15) | 0.456 (3) | 0.04 (2)* |
| C1        | 0.8953 (3) | 0.2405 (12) | 0.4417 (2) | 0.0179 (11) |
| C2        | 0.9850 (3) | 0.5192 (11) | 0.3901 (2) | 0.0161 (11) |
| C3        | 1.0549 (4) | 0.6679 (12) | 0.3661 (2) | 0.0214 (12) |
| H3        | 1.115554 | 0.640591 | 0.378875 | 0.026* |
| C4        | 1.0309 (4) | 0.8596 (12) | 0.3222 (2) | 0.0209 (12) |
| H4        | 1.076369 | 0.965301 | 0.304220 | 0.025* |
| C5        | 0.9402 (4) | 0.9002 (12) | 0.3038 (2) | 0.0215 (12) |
| C6        | 0.926165 | 1.034052 | 0.274018 | 0.026* |
| C7        | 0.8718 (3) | 0.7504 (12) | 0.3281 (2) | 0.0194 (11) |
| C8        | 0.8969 (3) | 0.5632 (11) | 0.3715 (2) | 0.0175 (11) |

### Atomic displacement parameters (Å²)

| \( U^{11} \) | \( U^{22} \) | \( U^{33} \) | \( U^{12} \) | \( U^{13} \) | \( U^{23} \) |
|--------------|--------------|--------------|--------------|--------------|--------------|
| I1           | 0.01571 (16) | 0.01825 (18) | 0.01870 (18) | 0.00147 (13) | 0.00332 (13) | 0.00178 (14) |
| I2           | 0.02249 (18) | 0.0226 (2)   | 0.01963 (19) | −0.00506 (14) | −0.00417 (14) | −0.00151 (15) |
| I3           | 0.01328 (16) | 0.0296 (2)   | 0.01939 (19) | 0.00433 (14) | −0.00095 (13) | 0.00351 (16) |
| F1           | 0.0106 (14) | 0.0263 (18) | 0.0298 (18) | 0.0009 (12) | −0.0040 (13) | 0.0000 (15) |
| F2           | 0.0196 (15) | 0.0230 (18) | 0.0274 (18) | 0.0043 (13) | 0.0018 (13) | −0.0061 (15) |
| F3           | 0.0164 (15) | 0.0279 (18) | 0.0201 (16) | 0.0002 (13) | −0.0031 (12) | −0.0072 (14) |
| C8           | 0.019 (3)   | 0.006 (2)   | 0.018 (3)   | 0.0031 (19) | −0.004 (2)   | −0.002 (2)   |
| C9           | 0.010 (2)   | 0.021 (3)   | 0.020 (3)   | −0.001 (2)   | 0.001 (2)    | 0.007 (2)    |
| C10          | 0.015 (2)   | 0.024 (3)   | 0.012 (3)   | −0.003 (2)   | 0.000 (2)    | 0.001 (2)    |
| C11          | 0.018 (3)   | 0.011 (3)   | 0.026 (3)   | 0.001 (2)    | 0.007 (2)    | 0.003 (2)    |
| C12          | 0.011 (2)   | 0.020 (3)   | 0.017 (3)   | −0.001 (2)   | −0.001 (2)   | 0.002 (2)    |
| C13          | 0.015 (2)   | 0.015 (3)   | 0.014 (3)   | −0.0009 (19) | −0.002 (2)   | 0.001 (2)    |
| S1           | 0.0167 (6)  | 0.0202 (7)  | 0.0200 (7)  | 0.0025 (5)   | 0.0036 (5)   | 0.0035 (6)   |
| O1           | 0.0153 (17) | 0.019 (2)   | 0.0177 (19) | 0.0016 (14)  | 0.0007 (14)  | 0.0059 (16)  |

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### Supporting Information

| Atom | U(1)  | U(2)  | U(3)  | U(4)  | U(5)  | U(6)  |
|------|-------|-------|-------|-------|-------|-------|
| N1   | 0.012 (2) | 0.023 (3) | 0.012 (2) | 0.0051 (18) | −0.0012 (17) | 0.0016 (19) |
| C1   | 0.016 (2) | 0.022 (3) | 0.016 (3) | 0.006 (2) | −0.002 (2) | −0.005 (2) |
| C2   | 0.014 (2) | 0.015 (3) | 0.018 (3) | 0.004 (2) | −0.001 (2) | −0.004 (2) |
| C3   | 0.018 (3) | 0.025 (3) | 0.021 (3) | 0.002 (2) | 0.002 (2) | −0.001 (2) |
| C4   | 0.023 (3) | 0.020 (3) | 0.020 (3) | −0.002 (2) | 0.011 (2) | −0.002 (2) |
| C5   | 0.037 (3) | 0.017 (3) | 0.011 (3) | 0.004 (2) | 0.003 (2) | 0.002 (2) |
| C6   | 0.016 (3) | 0.021 (3) | 0.018 (3) | 0.005 (2) | −0.002 (2) | −0.002 (2) |
| C7   | 0.018 (3) | 0.014 (3) | 0.020 (3) | −0.001 (2) | 0.002 (2) | −0.004 (2) |

**Geometric parameters (Å, °)**

| Bond/Angle | Distance/Angle |
|------------|---------------|
| I1—C8      | 2.096 (5)     |
| I2—C10     | 2.086 (5)     |
| I3—C12     | 2.091 (5)     |
| F1—C9      | 1.356 (5)     |
| F2—C11     | 1.347 (6)     |
| F3—C13     | 1.340 (6)     |
| C8—C9      | 1.378 (7)     |
| C8—C13     | 1.392 (7)     |
| C9—C10     | 1.378 (8)     |
| C10—C11    | 1.381 (7)     |
| C11—C12    | 1.389 (7)     |
| C12—C13    | 1.379 (7)     |
| C9—C8—I1   | 122.3 (4)     |
| C9—C8—I13  | 120.5 (4)     |
| F1—C9—C8   | 118.4 (5)     |
| F1—C9—I1   | 118.7 (5)     |
| C10—C9—I2  | 122.3 (4)     |
| C11—C10—I1 | 120.0 (4)     |
| F2—C11—I2  | 119.0 (5)     |
| F2—C11—I13 | 118.7 (5)     |
| C10—C11—I2 | 122.4 (5)     |
| C11—C12—I3 | 119.8 (4)     |
| C13—C12—I3 | 119.8 (4)     |
| F3—C13—I13 | 117.3 (5)     |
| F3—C13—I12 | 118.6 (4)     |
| F3—C13—I13 | 118.7 (4)     |
| C1—O1—I1   | 107.6 (4)     |
| C1—N1—I13  | 117.5 (5)     |
| C1—N1—C2   | 110.2 (4)     |
| C2—N1—I13  | 132 (5)       |

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Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| N1—HN1···S1<sup>i</sup> | 0.85 (7) | 2.53 (7) | 3.377 (4) | 176 (6) |
| C3—H3···I1<sup>ii</sup> | 0.95 | 3.04 | 3.969 (5) | 179.1 (6) |
| C6—H6···I2<sup>iii</sup> | 0.95 | 3.23 | 4.009 (5) | 179.1 (6) |

Symmetry codes: (i) −x+2, −y, −z+1; (ii) −x+2, −y+1, −z+1; (iii) x, −y+5/2, z−1/2.

1,3-Benzothiazole-2-thiol)–1,2,3,4-tetrafluoro-5,6-diiodobenzene (3/4) (3MBZTH_412F4DIB)

Crystal data

| Parameter | Value |
|-----------|-------|
| \(V\) | 2830.9 (5) Å³ |
| \(Z\) | 2 |
| \(F(000)\) | 1940 |
| \(D_x\) | 2.474 Mg m⁻³ |
| Mo \(K\alpha\) radiation, \(\lambda\) | 0.71073 Å |
| \(\mu\) | 4.69 mm⁻¹ |
| \(T\) | 100 K |
| Crystal habit | Plate, colourless |
| Dimensions | \(0.30 \times 0.13 \times 0.04\) mm |

Data collection

| Parameter | Value |
|-----------|-------|
| Bruker D8 Venture Photon 2 diffractometer | |
| Radiation source: Incoatec IμS | |
| \(\phi\) and \(\omega\) scans | |
| Absorption correction: multi-scan (SADABS; Bruker, 2017) | |
| \(T_{	ext{min}} = 0.570, T_{	ext{max}} = 0.746\) | |
| 78566 measured reflections | |
12466 independent reflections
11325 reflections with $|I| > 2\sigma(I)$
$R_{int} = 0.067$
$\theta_{max} = 27.2^\circ$, $\theta_{min} = 2.5^\circ$

Refinement

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.067$
$wR(F^2) = 0.220$
$S = 1.06$
12466 reflections
704 parameters
66 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/\sigma^2(Fo^2) + (0.1285P)^2 + 109.2112P$
where $P = (Fo^2 + 2Fc^2)/3$
$\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 2.61 \text{ e } \AA^{-3}$
$\Delta\rho_{min} = -1.48 \text{ e } \AA^{-3}$

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.

Refinement. Refined as a 2-component twin.

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

|     | x        | y        | z        | $U_{eq}$/$U_{eq}$ |
|-----|----------|----------|----------|-------------------|
|I1   | 0.36226  | 1.16753  | 0.05962  | 0.0199 (2)        |
|I2   | -0.11193 | 1.17703  | 0.07045  | 0.0192 (2)        |
|S3   | 0.8569   | 0.2851   | 0.27854  | 0.0222 (8)        |
|S4   | 0.9358   | 0.3501   | 0.15807  | 0.0219 (8)        |
|F1   | -0.1901  | 0.9764   | 0.0748   | 0.023 (2)         |
|F2   | 0.0046   | 0.8143   | 0.0705   | 0.0210 (18)       |
|F3   | 0.3454   | 0.8083   | 0.0614   | 0.0218 (19)       |
|F4   | 0.4959   | 0.9599   | 0.0585   | 0.023 (2)         |
|N2   | 0.7575   | 0.4523   | 0.2157   | 0.022 (3)         |
|HN2  | 0.702118 | 0.472763 | 0.243675 | 0.026*            |
|C8   | 0.8414   | 0.3657   | 0.2214   | 0.022 (3)         |
|C9   | 0.8613   | 0.4590   | 0.1256   | 0.022 (3)         |
|C10  | 0.877    | 0.5034   | 0.0712   | 0.025 (3)         |
|H10  | 0.946102 | 0.474610 | 0.045108 | 0.030*            |
|C11  | 0.792    | 0.5893   | 0.0558   | 0.033 (3)         |
|H11  | 0.792908 | 0.617175 | 0.017859 | 0.040*            |
|C12  | 0.704    | 0.6375   | 0.0933   | 0.029 (3)         |
|H12  | 0.656903 | 0.699744 | 0.081256 | 0.035*            |
|C13  | 0.683    | 0.5975   | 0.1470   | 0.024 (3)         |
|H13  | 0.616999 | 0.629053 | 0.172475 | 0.029*            |
|C14  | 0.764    | 0.5071   | 0.1632   | 0.021 (3)         |
|C22  | 0.2314   | 1.0522   | 0.0647   | 0.014 (3)         |
|C23  | 0.0557   | 1.0544   | 0.0689   | 0.016 (3)         |
|C24  | -0.0213  | 0.9775   | 0.0714   | 0.014 (2)         |
|C25  | 0.0758   | 0.8923   | 0.0684   | 0.015 (2)         |
| Atom | x     | y     | z     | U11   | U22   | U33   |
|------|-------|-------|-------|-------|-------|-------|
| C26  | 0.2483 (19) | 0.8892 (10) | 0.0642 (6) | 0.016 (3) |
| C27  | 0.3254 (18) | 0.9675 (10) | 0.0619 (6) | 0.015 (3) |
| I3   | 0.64678 (12) | 1.13261 (7) | 0.23166 (4) | 0.0194 (2) |
| I4   | 0.18439 (13) | 1.10869 (8) | 0.23135 (5) | 0.0227 (2) |
| F5   | 0.1660 (12) | 0.9119 (8) | 0.2046 (5) | 0.028 (2) |
| F6   | 0.4004 (14) | 0.7773 (7) | 0.1803 (5) | 0.032 (2) |
| F7   | 0.7341 (13) | 0.8013 (7) | 0.1742 (5) | 0.028 (2) |
| F8   | 0.8342 (12) | 0.9554 (7) | 0.1967 (5) | 0.027 (2) |
| C28  | 0.548 (2) | 1.0190 (11) | 0.2120 (6) | 0.019 (3) |
| C29  | 0.3765 (17) | 1.0068 (10) | 0.2139 (6) | 0.014 (3) |
| C30  | 0.3304 (19) | 0.9261 (12) | 0.2037 (6) | 0.019 (3) |
| C31  | 0.447 (2) | 0.8555 (11) | 0.1910 (7) | 0.020 (3) |
| C32  | 0.618 (2) | 0.8672 (12) | 0.1869 (7) | 0.022 (3) |
| C33  | 0.6663 (18) | 0.9461 (11) | 0.1986 (7) | 0.018 (3) |
| C34  | 0.23233 (12) | 0.71859 (7) | 0.32850 (4) | 0.0183 (2) |
| C35  | −0.23322 (12) | 0.70593 (7) | 0.32425 (4) | 0.0200 (2) |
| C36  | −0.4093 (13) | 0.8951 (8) | 0.3451 (4) | 0.028 (2) |
| C37  | −0.2991 (13) | 1.0414 (7) | 0.3748 (5) | 0.028 (2) |
| C38  | 0.0385 (13) | 1.0534 (7) | 0.3782 (5) | 0.027 (2) |
| C39  | 0.2640 (12) | 0.9152 (7) | 0.3547 (5) | 0.026 (2) |
| C40  | 0.0485 (19) | 0.8268 (10) | 0.3394 (6) | 0.017 (3) |
| C41  | −0.1293 (18) | 0.8223 (10) | 0.3377 (6) | 0.015 (2) |
| C42  | −0.2433 (18) | 0.8975 (10) | 0.3469 (6) | 0.016 (2) |
| C43  | −0.1897 (18) | 0.9730 (10) | 0.3622 (6) | 0.015 (2) |
| C44  | −0.016 (2) | 0.9776 (11) | 0.3644 (7) | 0.020 (3) |
| C45  | 0.097 (2) | 0.9065 (10) | 0.3535 (7) | 0.019 (3) |
| C46  | 0.2233 (13) | 0.46575 (7) | 0.37019 (4) | 0.0196 (2) |
| C47  | −0.02757 (15) | 0.35978 (8) | 0.51719 (5) | 0.0269 (3) |
| C48  | 0.1440 (16) | 0.1570 (8) | 0.5289 (4) | 0.036 (3) |
| C49  | 0.3307 (17) | 0.0627 (7) | 0.4593 (5) | 0.037 (3) |
| C50  | 0.3867 (14) | 0.1426 (7) | 0.3536 (4) | 0.030 (2) |
| C51  | 0.2600 (13) | 0.3180 (7) | 0.3159 (4) | 0.026 (2) |
| C52  | 0.1256 (19) | 0.3310 (9) | 0.4037 (6) | 0.015 (3) |
| C53  | 0.100 (2) | 0.2902 (12) | 0.4582 (7) | 0.020 (3) |
| C54  | 0.171 (2) | 0.2029 (13) | 0.4767 (7) | 0.025 (3) |
| C55  | 0.263 (2) | 0.1486 (12) | 0.4419 (7) | 0.023 (3) |
| C56  | 0.292 (2) | 0.1890 (11) | 0.3876 (7) | 0.022 (3) |
| C57  | 0.223 (2) | 0.2791 (10) | 0.3680 (6) | 0.017 (3) |
| S1   | 0.5243 (5) | 0.5375 (3) | 0.31454 (16) | 0.0193 (7) |
| S2   | 0.4491 (5) | 0.4693 (3) | 0.43557 (17) | 0.0217 (8) |
| N1   | 0.6185 (17) | 0.3686 (9) | 0.3737 (6) | 0.020 (2) |
| HN1  | 0.671175 | 0.349037 | 0.345043 | 0.025* |
| C1   | 0.5386 (19) | 0.4543 (10) | 0.3712 (6) | 0.0156 (18) |
| C2   | 0.518 (2) | 0.3547 (12) | 0.4654 (7) | 0.022 (3) |
| C3   | 0.498 (2) | 0.3107 (13) | 0.5198 (6) | 0.024 (3) |
| H3   | 0.442344 | 0.342280 | 0.547045 | 0.029* |
| C4   | 0.566 (3) | 0.2171 (16) | 0.5328 (9) | 0.042 (5) |
| H4   | 0.548501 | 0.183201 | 0.568902 | 0.050* |
| Atomic displacement parameters (Å²) | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|-----------------------------------|---------|---------|---------|---------|---------|---------|
| I1                                | 0.0179 (5) | 0.0178 (5) | 0.0249 (5) | −0.0061 (4) | 0.0017 (4) | −0.0046 (4) |
| I2                                | 0.0168 (5) | 0.0176 (4) | 0.0238 (5) | 0.0017 (3) | 0.0003 (4) | −0.0076 (4) |
| S3                                | 0.026 (2) | 0.0144 (17) | 0.026 (2) | 0.0011 (14) | 0.0023 (16) | −0.0064 (15) |
| S4                                | 0.0223 (19) | 0.0171 (18) | 0.027 (2) | −0.0012 (14) | 0.0041 (16) | −0.0092 (15) |
| F1                                | 0.014 (4) | 0.025 (5) | 0.030 (5) | −0.001 (4) | 0.001 (4) | −0.004 (4) |
| F2                                | 0.021 (2) | 0.022 (2) | 0.022 (2) | −0.0089 (18) | −0.0027 (18) | −0.0044 (18) |
| F3                                | 0.016 (4) | 0.016 (4) | 0.032 (5) | 0.004 (3) | 0.000 (4) | −0.006 (4) |
| F4                                | 0.007 (4) | 0.024 (5) | 0.038 (6) | −0.004 (3) | 0.001 (4) | −0.006 (4) |
| N2                                | 0.020 (7) | 0.020 (7) | 0.026 (7) | 0.002 (5) | 0.001 (5) | −0.009 (6) |
| C8                                | 0.011 (7) | 0.025 (8) | 0.032 (9) | −0.002 (6) | −0.011 (6) | −0.011 (7) |
| C9                                | 0.004 (6) | 0.021 (8) | 0.043 (10) | 0.000 (5) | −0.001 (6) | −0.011 (7) |
| C10                               | 0.030 (7) | 0.021 (7) | 0.025 (7) | 0.001 (6) | −0.012 (6) | −0.009 (6) |
| C11                               | 0.042 (7) | 0.028 (7) | 0.031 (7) | 0.011 (6) | −0.019 (6) | −0.013 (6) |
| C12                               | 0.028 (7) | 0.025 (7) | 0.034 (7) | 0.002 (6) | −0.010 (6) | −0.008 (6) |
| C13                               | 0.012 (7) | 0.019 (8) | 0.041 (10) | −0.001 (6) | −0.005 (7) | −0.007 (7) |
| C14                               | 0.023 (8) | 0.024 (8) | 0.019 (7) | −0.009 (6) | 0.015 (6) | −0.010 (6) |
| C22                               | 0.018 (7) | 0.014 (6) | 0.006 (6) | 0.004 (5) | 0.001 (5) | 0.001 (5) |
| C23                               | 0.009 (5) | 0.018 (6) | 0.018 (6) | 0.001 (5) | 0.002 (5) | −0.001 (5) |
| C24                               | 0.005 (5) | 0.016 (5) | 0.021 (5) | 0.002 (4) | 0.004 (4) | −0.004 (4) |
| C25                               | 0.016 (6) | 0.013 (6) | 0.016 (6) | 0.000 (5) | 0.002 (5) | −0.004 (5) |
| C26                               | 0.016 (7) | 0.015 (7) | 0.017 (7) | 0.006 (5) | −0.008 (6) | −0.008 (6) |
| C27                               | 0.012 (6) | 0.012 (6) | 0.020 (7) | 0.001 (5) | 0.004 (5) | −0.005 (6) |
| I3                                | 0.0164 (5) | 0.0180 (5) | 0.0230 (5) | −0.0020 (3) | 0.0015 (4) | −0.0025 (4) |
| I4                                | 0.0135 (4) | 0.0257 (5) | 0.0256 (5) | 0.0039 (4) | 0.0016 (4) | −0.0007 (4) |
|     |      |      |      |      |      |      |
|-----|------|------|------|------|------|------|
| F5  | 0.010 | 0.041 | 0.036 | -0.007 | 0.001 | -0.012 |
| F6  | 0.026 | 0.025 | 0.049 | -0.004 | -0.003 | -0.013 |
| F7  | 0.022 | 0.028 | 0.036 | 0.000 | 0.004 | -0.015 |
| F8  | 0.009 | 0.028 | 0.047 | -0.003 | 0.001 | -0.013 |
| C28 | 0.019 | 0.017 | 0.016 | 0.001 | 0.008 | 0.001 |
| C29 | 0.006 | 0.018 | 0.022 | -0.003 | -0.001 | -0.010 |
| C30 | 0.013 | 0.034 | 0.008 | -0.003 | -0.005 | -0.001 |
| C31 | 0.016 | 0.020 | 0.025 | 0.000 | -0.005 | -0.004 |
| C32 | 0.020 | 0.027 | 0.021 | -0.001 | -0.004 | -0.007 |
| C33 | 0.004 | 0.022 | 0.028 | -0.002 | 0.003 | -0.007 |
| I5  | 0.012 | 0.018 | 0.023 | 0.001 | 0.002 | -0.004 |
| I6  | 0.016 | 0.019 | 0.024 | -0.002 | -0.003 | -0.009 |
| F9  | 0.016 | 0.031 | 0.032 | 0.002 | -0.001 | 0.004 |
| F10 | 0.022 | 0.020 | 0.042 | 0.008 | 0.002 | -0.010 |
| F11 | 0.025 | 0.022 | 0.038 | -0.004 | -0.001 | -0.013 |
| F12 | 0.012 | 0.020 | 0.043 | -0.002 | -0.001 | 0.003 |
| C34 | 0.014 | 0.008 | 0.023 | 0.003 | 0.006 | 0.007 |
| C35 | 0.008 | 0.016 | 0.020 | -0.001 | -0.003 | 0.004 |
| C36 | 0.006 | 0.017 | 0.020 | 0.001 | -0.001 | 0.004 |
| C37 | 0.006 | 0.017 | 0.021 | 0.001 | 0.001 | 0.001 |
| C38 | 0.021 | 0.017 | 0.021 | -0.006 | -0.004 | 0.003 |
| C39 | 0.027 | 0.010 | 0.019 | -0.006 | 0.001 | 0.000 |
| I7  | 0.018 | 0.016 | 0.023 | 0.003 | -0.002 | -0.016 |
| I8  | 0.028 | 0.031 | 0.021 | -0.007 | 0.003 | -0.009 |
| F13 | 0.048 | 0.029 | 0.022 | -0.004 | 0.001 | 0.012 |
| F14 | 0.056 | 0.018 | 0.033 | 0.009 | -0.011 | 0.000 |
| F15 | 0.031 | 0.030 | 0.030 | 0.010 | 0.007 | -0.016 |
| F16 | 0.026 | 0.030 | 0.018 | 0.003 | 0.006 | 0.001 |
| C40 | 0.019 | 0.007 | 0.017 | 0.005 | 0.002 | -0.002 |
| C41 | 0.015 | 0.027 | 0.017 | -0.002 | -0.006 | 0.003 |
| C42 | 0.019 | 0.035 | 0.013 | 0.009 | 0.001 | 0.007 |
| C43 | 0.022 | 0.022 | 0.020 | 0.004 | -0.008 | 0.004 |
| C44 | 0.024 | 0.020 | 0.024 | -0.002 | 0.002 | -0.009 |
| C45 | 0.018 | 0.014 | 0.016 | 0.003 | 0.002 | 0.000 |
| S1  | 0.018 | 0.016 | 0.022 | 0.004 | 0.001 | -0.003 |
| S2  | 0.019 | 0.026 | 0.019 | 0.001 | 0.006 | -0.007 |
| N1  | 0.020 | 0.021 | 0.023 | -0.004 | -0.001 | -0.007 |
| C1  | 0.015 | 0.019 | 0.017 | -0.006 | -0.002 | -0.010 |
| C2  | 0.015 | 0.030 | 0.021 | -0.008 | 0.003 | -0.003 |
| C3  | 0.019 | 0.042 | 0.010 | -0.005 | 0.001 | -0.001 |
| C4  | 0.041 | 0.051 | 0.032 | -0.030 | -0.007 | 0.011 |
| C5  | 0.058 | 0.026 | 0.022 | -0.009 | -0.008 | 0.010 |
| C6  | 0.031 | 0.018 | 0.033 | -0.004 | -0.012 | 0.001 |
| C7  | 0.026 | 0.014 | 0.031 | -0.002 | -0.009 | 0.002 |
| S5  | 0.019 | 0.011 | 0.022 | 0.003 | 0.002 | -0.002 |
| S6  | 0.021 | 0.015 | 0.020 | -0.005 | -0.005 | -0.000 |
| N3  | 0.016 | 0.014 | 0.017 | 0.004 | 0.000 | -0.003 |
| C15 | 0.018 | 0.011 | 0.020 | -0.002 | -0.007 | -0.001 |

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### Geometric parameters (Å, °)

|         |    |    |    |    |    |    |
|---------|----|----|----|----|----|----|
| I1—C22 | 2.094 (15) | C35—C36 | 1.39 (2) |
| I2—C23 | 2.121 (15) | C36—C37 | 1.37 (2) |
| S3—C8  | 1.669 (18) | C37—C38 | 1.40 (2) |
| S4—C8  | 1.738 (18) | C38—C39 | 1.35 (2) |
| S4—C9  | 1.711 (17) | I7—C40  | 2.097 (14) |
| F1—C24 | 1.341 (16) | I8—C41  | 2.086 (17) |
| F2—C25 | 1.344 (17) | F13—C42 | 1.358 (19) |
| F3—C26 | 1.355 (16) | F14—C43 | 1.320 (19) |
| F4—C27 | 1.344 (17) | F15—C44 | 1.328 (18) |
| N2—HN2 | 0.8800 | C40—C41 | 1.38 (2) |
| N2—C8  | 1.36 (2)  | C40—C45 | 1.41 (2) |
| C9—C10 | 1.39 (3)  | C41—C42 | 1.35 (2) |
| C9—C14 | 1.42 (2)  | C42—C43 | 1.40 (2) |
| C10—H10| 0.9500   | C43—C44 | 1.38 (2) |
| C10—C11| 1.36 (2)  | C44—C45 | 1.39 (2) |
| C11—H11| 0.9500   | S1—C1  | 1.680 (16) |
| C11—C12| 1.38 (3)  | S2—C1  | 1.747 (15) |
| C12—H12| 0.9500   | S2—C2  | 1.755 (19) |
| C12—C13| 1.36 (3)  | N1—HN1 | 0.8800 |
| C13—H13| 0.9500   | N1—C1  | 1.34 (2) |
| C13—C14| 1.41 (2)  | N1—C7  | 1.39 (2) |
| C22—C23| 1.39 (2)  | C2—C3  | 1.39 (2) |
| C22—C27| 1.391 (19) | C2—C7  | 1.38 (2) |
| C23—C24| 1.35 (2)  | C3—H3  | 0.9500 |
| C24—C25| 1.408 (19) | C3—C4  | 1.41 (3) |
| C25—C26| 1.36 (2)  | C4—H4  | 0.9500 |
| C26—C27| 1.37 (2)  | C4—C5  | 1.41 (3) |
| I3—C28 | 2.089 (16) | C5—H5  | 0.9500 |
| I4—C29 | 2.097 (14) | C5—C6  | 1.42 (3) |
| F5—C30 | 1.347 (18) | C6—H6  | 0.9500 |
| F6—C31 | 1.336 (19) | C6—C7  | 1.43 (2) |
| F7—C32 | 1.330 (19) | S5—C15 | 1.671 (16) |
| F8—C33 | 1.355 (17) | S6—C15 | 1.749 (16) |
| C28—C29| 1.40 (2)  | S6—C16 | 1.740 (15) |
| C28—C33| 1.42 (2)  | N3—HN3 | 0.8800 |
| C29—C30| 1.37 (2)  | N3—C15 | 1.331 (19) |
| C30—C31| 1.38 (2)  | N3—C21 | 1.416 (19) |
| C31—C32| 1.38 (2)  | C16—C17 | 1.41 (2) |
F3—C26—C27 119.2 (13)  C5—C4—H4 119.6
C25—C26—C27 120.8 (13)  C4—C5—H5 119.0
F4—C27—C22 120.4 (13)  C4—C5—C6 122.0 (19)
F4—C27—C26 118.2 (13)  C6—C5—H6 119.0
C26—C27—C22 121.4 (14)  C5—C6—H6 122.2
C29—C28—I3 125.4 (11)  C5—C6—C7 115.5 (18)
C29—C28—C33 117.2 (14)  C7—C6—H6 122.2
C33—C28—I3 117.3 (11)  N1—C7—C6 123.2 (17)
C29—C28—I4 122.4 (11)  C2—C7—N1 115.2 (15)
C30—C29—I4 118.3 (10)  C2—C7—C6 121.5 (17)
C30—C29—C28 119.3 (14)  C16—S6—C15 91.6 (7)
F5—C30—C29 121.1 (14)  C15—N3—HN3 122.0
F5—C30—C31 116.1 (15)  C15—N3—C21 116.0 (13)
C29—C30—C31 127.7 (14)  C21—N3—HN3 122.0
F6—C31—C29 122.3 (14)  S5—C15—S6 123.6 (9)
F6—C31—C32 118.6 (15)  N3—C15—S5 125.8 (12)
C30—C31—C32 119.1 (15)  N3—C15—S6 110.6 (12)
C33—C32—I5 121.1 (15)  C34—C35—C36—F9 179.6 (14)
C33—C32—C31 119.0 (15)  C34—C35—C36—C37 −6 (2)
C33—C32—C34 119.8 (14)  C35—C36—C37—F12 178.7 (13)
C32—C33—C31 118.6 (14)  C35—C36—C37—C38 5 (2)
C35—C34—I5 123.6 (11)  C36—C37—C38—F11 178.3 (14)
C39—C34—C35 119.6 (11)  C36—C37—C38—C39 −3 (2)
C39—C34—I5 119.6 (11)  C37—C38—C39—F12 178.2 (14)
C39—C34—C35 116.6 (14)  C37—C38—C39—C34 179.7 (14)
C34—C35—I6 123.6 (11)  C34—C35—C36—F9 179.6 (14)
C34—C35—I6 123.6 (11)  C34—C35—C36—C37 −6 (2)
C36—C35—C34 119.6 (14)  C35—C36—C37—F11 178.3 (14)
F9—C36—C35 120.7 (14)  C35—C36—C37—C38 −3 (2)
F9—C36—C37 117.6 (13)  C36—C37—C38—F11 178.2 (14)
C37—C36—C35 121.5 (13)  C36—C37—C38—C39 −1 (2)
F10—C37—C36 121.4 (13)  C37—C38—C39—F12 −3 (2)
F10—C37—C38 119.6 (14)  C37—C38—C39—C34 179.7 (14)
C36—C37—C38 119.0 (14)  C37—C38—C39—C34 179.7 (14)
F11—C38—C37 119.3 (14)  C38—C39—F12 178.2 (14)

I1—C22—C23—I2 0.4 (18)  F11—C38—C39—F12 −3 (2)
I1—C22—C23—C24 179.1 (11)  F11—C38—C39—C34 179.7 (14)
I1—C22—C27—F4 2.9 (19)  C34—C35—C36—F9 179.6 (14)
I1—C22—C27—C26 −179.1 (11)  C34—C35—C36—C37 −6 (2)
I2—C23—C24—F1 0 (2)  C35—C34—C39—F12 −178.7 (13)
I2—C23—C24—C25 177.4 (11)  C35—C34—C39—C38 −1 (2)
S4—C9—C10—C11 174.9 (14)  C35—C36—C37—F10 −175.0 (14)
S4—C9—C14—N2 0.4 (17)  C35—C36—C37—C38 5 (2)
S4—C9—C14—C13 −178.2 (13)  C36—C37—C38—F11 178.3 (14)
F1—C24—C25—F2 −2 (2)  C36—C37—C38—C39 −3 (2)
F1—C24—C25—C26 179.3 (13)  C37—C38—C39—F12 178.2 (14)
F2—C25—C26—F3 1 (2) C37—C38—C39—C34 1 (2)
F2—C25—C26—C27 179.9 (13) C39—C34—C35—I6 -174.6 (11)
F3—C26—C27—F4 -2 (2) C39—C34—C35—C36 3 (2)
F3—C26—C27—C22 -179.9 (13) I7—C40—C41—I8 -5.3 (19)
C8—S4—C9—C10 -178.3 (16) I7—C40—C41—I42 179.9 (13)
C8—S4—C9—C14 -0.2 (12) I7—C40—C45—F16 6 (2)
C8—N2—C14—C9 -1 (2) I7—C40—C45—C44 -178.8 (12)
C8—N2—C14—C13 178.0 (16) I8—C41—C42—F13 8 (2)
C9—S4—C8—S3 -179.6 (10) I8—C41—C42—C43 -178.7 (14)
C9—S4—C8—N2 -0.1 (12) F13—C42—C43—F14 -5 (3)
C9—C10—C11—C12 6 (3) F13—C42—C43—C44 178.0 (15)
C10—C9—C14—N2 178.8 (14) F14—C43—C44—F15 -1 (3)
C10—C9—C14—C13 0 (2) F14—C43—C44—C45 -179.9 (15)
C10—C11—C12—C13 -7 (3) F15—C44—C45—F16 -3 (2)
C11—C12—C13—C14 4 (3) F15—C44—C45—C40 -178.3 (15)
C12—C13—C14—N2 -178.8 (16) C40—C41—C42—F13 -176.5 (16)
C12—C13—C14—C9 0 (2) C40—C41—C42—C43 -176.6 (16)
C14—N2—C8—S3 179.9 (12) C41—C40—C45—F16 -175.7 (14)
C14—N2—C8—S4 0.4 (17) C41—C40—C45—C44 0 (2)
C14—C9—C10—C11 -3 (2) C41—C42—C43—F14 -178.9 (17)
C22—C23—C24—F1 179.2 (14) C41—C42—C43—C44 4 (3)
C22—C23—C24—C25 -179.2 (14) C42—C43—C44—F15 176.2 (16)
C23—C22—C27—F4 -179.1 (13) C42—C43—C44—C45 -3 (3)
C23—C22—C27—C26 -179.1 (13) C43—C44—C45—F16 176.8 (15)
C23—C24—C25—F2 -179.8 (14) C43—C44—C45—C40 1 (2)
C23—C24—C25—C26 1 (2) C45—C40—C41—I8 176.1 (11)
C24—C25—C26—F3 179.9 (13) C45—C40—C41—I42 1 (2)
C24—C25—C26—C27 -1 (2) S2—C2—C3—C4 179.7 (14)
C25—C26—C27—F4 179.1 (14) S2—C2—C3—C4 179.7 (14)
C25—C26—C27—C22 1 (2) C2—S2—C1—S1 178.3 (15)
C27—C22—C23—J2 -177.4 (11) C2—S2—C1—N1 -1.8 (12)
C27—C22—C23—C24 1 (2) C2—S2—C1—N1 179.0 (15)
I3—C28—C33—F8 -175.4 (11) C2—S2—C1—N1 179.0 (15)
I3—C28—C33—C32 177.7 (13) C2—S2—C1—N1 187.3 (10)
I4—C29—C30—F5 0 (2) C2—S2—C1—N1 -1.8 (12)
I4—C29—C30—C31 179.0 (12) C2—S2—C1—C6 4 (3)
F5—C30—C31—F6 0 (2) C3—C2—C7—N1 -178.9 (15)
F5—C30—C31—C32 177.2 (14) C3—C2—C7—N1 178.9 (15)
F6—C31—C32—F7 -2 (2) C3—C2—C7—N1 178.9 (15)
F6—C31—C32—C33 -179.0 (15) C3—C2—C7—N1 178.9 (15)
F7—C32—C33—F8 0 (2) C5—C6—C7—N1 -177.9 (17)
F7—C32—C33—C28 179.6 (15) C5—C6—C7—N1 -177.9 (17)
C28—C29—C30—F5 -179.4 (14) C7—N1—C1—S1 -179.9 (12)
C28—C29—C30—C31 0 (2) C7—N1—C1—S1 179.9 (12)
C29—C28—C33—C8 179.5 (14) C7—N1—C1—S1 179.9 (12)
C29—C28—C33—C31 1 (2) S6—C16—C17—C18 -178.4 (13)
C29—C28—C33—C32 1 (2) S6—C16—C21—N3 -4.0 (16)
Hydrogen-bond geometry (Å, °)

| D—H···A | D—H  | H···A  | D···A  | D—H···A |
|---------|-------|--------|--------|---------|
| N2—H2···S1 | 0.88  | 2.45   | 3.326 (14) | 174    |
| N1—H1···S3  | 0.88  | 2.40   | 3.266 (14) | 169    |
| C6—H6···F10i | 0.95  | 2.60   | 3.29 (2)   | 130    |
| N3—H3···S5ii | 0.88  | 2.42   | 3.290 (14) | 170    |
| C17—H17···F16 | 0.95  | 2.30   | 3.232 (18) | 166    |
| C20—H20···F2  | 0.95  | 2.53   | 3.128 (18) | 121    |
| C20—H20···F3  | 0.95  | 2.54   | 3.181 (17) | 125    |

Symmetry codes: (i) x+1, y-1, z; (ii) -x+1, -y+1, -z.

1,3-Benzothiazole-2-thiol–1,2,3,5-tetrafluoro-4,6-diiodobenzene (1/1) (MBZTH_13F4DIB)

Crystal data

C6F4I2·C7H5NS2  
Z = 2  
F(000) = 528  
Dc = 2.349 Mg m⁻³

Triclinic, P̅1

a = 7.2175 (4) Å  
b = 8.2675 (5) Å  
c = 14.4498 (9) Å

Mo Ka radiation, λ = 0.71073 Å

Cell parameters from 9969 reflections  
θ = 2.6–30.1°

µ = 4.20 mm⁻¹  
T = 100 K

Plate, colourless

0.33 × 0.27 × 0.06 mm

Data collection

Bruker D8 Venture Photon 2 diffractometer

Absorption correction: multi-scan  
(SADABS; Bruker, 2017)

Radiation source: Incoatec IμS  
Tmin = 0.496, Tmax = 0.746

φ and ω scans  
27899 measured reflections
4701 independent reflections  
4391 reflections with $I > 2\sigma(I)$  
$R_{int} = 0.036$  
$\theta_{max} = 30.1^\circ$, $\theta_{min} = 2.6^\circ$

**Refinement**

Refinement on $F^2$  
Least-squares matrix: full  
$R[F^2 > 2\sigma(F^2)] = 0.018$  
$wR(F^2) = 0.044$  
$S = 1.09$

4701 reflections  
203 parameters  
0 restraints  
Primary atom site location: dual  
Hydrogen site location: mixed  
H atoms treated by a mixture of independent and constrained refinement  
$w = 1/[\sigma^2(F_o^2) + (0.0115P)^2 + 0.801P]$

$S = 1.09$

4701 reflections  
203 parameters  
0 restraints  
Primary atom site location: dual  
Hydrogen site location: mixed  
H atoms treated by a mixture of independent and constrained refinement  
$w = 1/[\sigma^2(F_o^2) + (0.0115P)^2 + 0.801P]$

$S = 1.09$

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203 parameters  
0 restraints  
Primary atom site location: dual  
Hydrogen site location: mixed  
H atoms treated by a mixture of independent and constrained refinement  
$w = 1/[\sigma^2(F_o^2) + (0.0115P)^2 + 0.801P]$

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203 parameters  
0 restraints  
Primary atom site location: dual  
Hydrogen site location: mixed  
H atoms treated by a mixture of independent and constrained refinement  
$w = 1/[\sigma^2(F_o^2) + (0.0115P)^2 + 0.801P]$

$S = 1.09$

4701 reflections  
203 parameters  
0 restraints  
Primary atom site location: dual  
Hydrogen site location: mixed  
H atoms treated by a mixture of independent and constrained refinement  
$w = 1/[\sigma^2(F_o^2) + (0.0115P)^2 + 0.801P]$

$S = 1.09$

4701 reflections  
203 parameters  
0 restraints  
Primary atom site location: dual  
Hydrogen site location: mixed  
H atoms treated by a mixture of independent and constrained refinement  
$w = 1/[\sigma^2(F_o^2) + (0.0115P)^2 + 0.801P]$

$S = 1.09$

4701 reflections  
203 parameters  
0 restraints  
Primary atom site location: dual  
Hydrogen site location: mixed  
H atoms treated by a mixture of independent and constrained refinement  
$w = 1/[\sigma^2(F_o^2) + (0.0115P)^2 + 0.801P]$

$S = 1.09$
### Atomic Displacement Parameters (Å²)

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$    | $U^{13}$    | $U^{23}$    |
|-----|-------------|-------------|-------------|-------------|-------------|-------------|
| I1  | 0.02600 (7) | 0.02230 (6) | 0.01536 (6) | 0.00997 (5) | 0.00446 (4) | 0.00537 (4) |
| I2  | 0.03060 (7) | 0.02527 (7) | 0.01257 (6) | 0.01186 (5) | −0.00081 (5) | 0.00492 (4) |
| F1  | 0.0198 (6)  | 0.0426 (7)  | 0.0197 (6)  | 0.0086 (5)  | −0.0015 (4)  | 0.0078 (5)  |
| F2  | 0.0252 (6)  | 0.0391 (7)  | 0.0221 (6)  | −0.0032 (5) | 0.0029 (5)   | 0.0105 (5)  |
| F3  | 0.0199 (6)  | 0.0372 (7)  | 0.0270 (7)  | 0.0003 (5)  | −0.0064 (5)  | 0.0059 (5)  |
| F4  | 0.0294 (6)  | 0.0222 (6)  | 0.0134 (5)  | 0.0085 (5)  | −0.0059 (4)  | 0.0017 (4)  |
| C8  | 0.0223 (9)  | 0.0171 (8)  | 0.0132 (8)  | 0.0072 (7)  | 0.0010 (6)   | 0.0024 (6)  |
| C9  | 0.0202 (9)  | 0.0214 (9)  | 0.0149 (8)  | 0.0064 (7)  | −0.0019 (6)  | 0.0009 (7)  |
| C10 | 0.0242 (9)  | 0.0171 (8)  | 0.0126 (8)  | 0.0055 (7)  | −0.0027 (7)  | 0.0023 (6)  |
| C11 | 0.0232 (9)  | 0.0190 (9)  | 0.0163 (9)  | 0.0019 (7)  | 0.0018 (7)   | 0.0037 (7)  |
| C12 | 0.0196 (9)  | 0.0196 (9)  | 0.0194 (9)  | 0.0020 (7)  | −0.0037 (7)  | 0.0020 (7)  |
| C13 | 0.0275 (9)  | 0.0147 (8)  | 0.0107 (8)  | 0.0066 (7)  | −0.0029 (7)  | −0.0005 (6) |
| S1  | 0.0184 (2)  | 0.01360 (18)| 0.01265 (19)| 0.00228 (15)| −0.00070 (15)| 0.00408 (15)|
| S2  | 0.01404 (19)| 0.01277 (18)| 0.01164 (18)| 0.00286 (14)| −0.00167 (14)| 0.00058 (14)|
| N1  | 0.0128 (7)  | 0.0128 (6)  | 0.0110 (6)  | 0.0022 (5)  | −0.0004 (5)  | 0.0018 (5)  |
| C1  | 0.0124 (7)  | 0.0134 (7)  | 0.0117 (7)  | 0.0041 (6)  | 0.0017 (6)   | 0.0017 (6)  |
| C2  | 0.0105 (7)  | 0.0143 (7)  | 0.0137 (7)  | 0.0044 (6)  | 0.0014 (6)   | 0.0029 (6)  |
| C3  | 0.0145 (8)  | 0.0126 (7)  | 0.0209 (9)  | 0.0030 (6)  | 0.0001 (6)   | 0.0021 (6)  |
| C4  | 0.0183 (8)  | 0.0150 (8)  | 0.0232 (9)  | 0.0052 (7)  | 0.0026 (7)   | 0.0073 (7)  |
| C5  | 0.0158 (8)  | 0.0205 (8)  | 0.0184 (8)  | 0.0078 (7)  | 0.0020 (6)   | 0.0082 (7)  |
| C6  | 0.0116 (7)  | 0.0184 (8)  | 0.0158 (8)  | 0.0055 (6)  | 0.0003 (6)   | 0.0039 (6)  |
| C7  | 0.0115 (7)  | 0.0128 (7)  | 0.0142 (8)  | 0.0038 (6)  | 0.0018 (6)   | 0.0026 (6)  |

### Geometric Parameters (Å, °)

|     |   |   |   |   |   |   |
|-----|---|---|---|---|---|---|
| I1  | C8 |  2.0910 (19) | S2—C2 | 1.7452 (18) |
| I2  | C10|  2.0875 (18) | N1—HN1| 0.87 (3)   |
| F1  | C9 |  1.343 (2)  | N1—C1 | 1.342 (2)  |
| F2  | C11|  1.337 (2)  | C2—C3 | 1.393 (2)  |
| F3  | C12|  1.341 (2)  | C2—C3 | 1.393 (2)  |
| F4  | C13|  1.346 (2)  | C2—C7 | 1.401 (2)  |
| C8  | C9 |  1.390 (3)  | C3—H3 | 0.9500     |
| C8  | C13|  1.387 (3)  | C3—C4 | 1.392 (3)  |
| C9  | C10|  1.385 (3)  | C4—H4 | 0.9500     |
| C10 | C11|  1.388 (3)  | C4—C5 | 1.401 (3)  |
| C11 | C12|  1.388 (3)  | C5—H5 | 0.9500     |
| C12 | C13|  1.383 (3)  | C5—C6 | 1.387 (3)  |
| S1  | C1 |  1.6799 (18)| C6—H6 | 0.9500     |
| S2  | C1 |  1.7355 (18)| C6—C7 | 1.393 (2)  |
| C9  | C8 |  120.65 (14)| S1—C1 | 122.60 (10) |
| C13 | C8 |  122.00 (14)| N1—C1 | 127.05 (13) |
| C13 | C8 |  117.34 (18)| N1—C2 | 110.35 (13) |

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### Hydrogen-bond geometry (Å, °)

| D—H···A       | D—H | H···A  | D···A  | D—H···A |
|---------------|------|--------|--------|---------|
| N1—HN1···S1i  | 0.87 (3) | 2.45 (3) | 3.3120 (15) | 175 (2) |
Symmetry code: (i) $-x+2, -y+2, -z+2$.

1,3-Benzothiazole-2-thiol–1,2,3,5-tetrafluoro-4,6-diiodobenzene (1/2) (MBZTH_213F4DIB)

Crystal data

$4C_6F_4I_2·2C_7H_5NS_2$

$M_r = 1941.92$

Monoclinic, $P2_1$

$\alpha = 4.5581 (3) \text{\AA}$

$\beta = 34.358 (2) \text{\AA}$

$\gamma = 15.6075 (10) \text{\AA}$

$\beta = 94.707 (2) \degree$

$V = 2436.0 (3) \text{\AA}^3$

$Z = 2$

$F(000) = 1768$

$D_x = 2.647 \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{\AA}$

Cell parameters from 9841 reflections

$\theta = 2.4–28.8\degree$

$\mu = 5.36 \text{mm}^{-1}$

$T = 100 \text{K}$

Prism, colourless

0.18 $\times$ 0.12 $\times$ 0.04 mm

Data collection

Bruker D8 Venture Photon 2 diffractometer

Radiation source: Incoatec $\mu S$

$\varphi$ and $\omega$ scans

Absorption correction: multi-scan

(SADABS; Bruker, 2017)

$T_{\text{min}} = 0.568, T_{\text{max}} = 0.746$

56285 measured reflections

12660 independent reflections

11766 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.050$

$\theta_{\text{max}} = 28.8\degree, \theta_{\text{min}} = 2.2\degree$

$h = -6 \rightarrow 6$

$k = -46 \rightarrow 46$

$l = -21 \rightarrow 21$

Refinement

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.026$

$wR(F^2) = 0.046$

$S = 1.09$

12660 reflections

622 parameters

2 restraints

Primary atom site location: dual

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/\sigma^2(F^2) + (0.0071P)^2 + 0.5877P$ where $P = (F^2 + 2F_C^2)/3$

$\Delta \rho_{\text{max}} = 1.01 \text{e \AA}^{-3}$

$\Delta \rho_{\text{min}} = -0.71 \text{e \AA}^{-3}$

Absolute structure: Refined as an inversion twin

Absolute structure parameter: 0.454 (15)

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.

Refinement. Refined as a 2-component inversion twin.

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

|            | $x$      | $y$      | $z$      | $U_{11}^*$/U_eq |
|------------|----------|----------|----------|-----------------|
| I1         | 1.31057 (9) | 0.37856 (2) | 0.44709 (3) | 0.01694 (9) |
| I2         | 0.58291 (10) | 0.34353 (2) | 0.11336 (3) | 0.02224 (10) |
| I3         | 0.13207 (9) | 0.44552 (2) | 0.04395 (3) | 0.01659 (9) |
| I4         | 0.84342 (11) | 0.47934 (2) | 0.38106 (3) | 0.02660 (11) |
| F1         | 1.0269 (9) | 0.38363 (11) | 0.2525 (2) | 0.0233 (9) |
| F2         | 0.4263 (9) | 0.27169 (12) | 0.2331 (3) | 0.0284 (10) |

*sup-60*
| Atom | X    | Y    | Z    | U1  | U2  | U3  | U12 | U13 | U23 |
|------|------|------|------|-----|-----|-----|-----|-----|-----|
| F3   | 0.6051 (10) | 0.25383 (12) | 0.3971 (3) | 0.0324 (10) |
| F4   | 0.9918 (10) | 0.29933 (12) | 0.4890 (2)  | 0.0276 (10) |
| F5   | 0.3945 (8)  | 0.44111 (11) | 0.2407 (2)  | 0.0205 (8)  |
| F6   | 1.0552 (9)  | 0.54664 (12) | 0.2573 (3)  | 0.0301 (10) |
| F7   | 0.9034 (9)  | 0.56331 (12) | 0.0915 (3)  | 0.0331 (10) |
| F8   | 0.4927 (9)  | 0.52114 (12) | 0.0003 (2)  | 0.0247 (9)  |
| C15  | 1.0262 (13) | 0.34210 (19) | 0.3717 (4)  | 0.0147 (13) |
| C16  | 0.9275 (14) | 0.35093 (18) | 0.2876 (4)  | 0.0163 (13) |
| C17  | 0.7260 (13) | 0.32806 (19) | 0.2383 (4)  | 0.0150 (13) |
| C18  | 0.6246 (14) | 0.2950 (2)   | 0.2766 (4)  | 0.0181 (14) |
| C19  | 0.7144 (15) | 0.2853 (2)   | 0.3607 (4)  | 0.0197 (14) |
| C20  | 0.9139 (15) | 0.3087 (2)   | 0.4070 (4)  | 0.0187 (14) |
| C21  | 0.4273 (13) | 0.48043 (17) | 0.1192 (4)  | 0.0118 (12) |
| C22  | 0.5141 (14) | 0.47193 (19) | 0.2048 (4)  | 0.0160 (13) |
| C23  | 0.7212 (14) | 0.49397 (19) | 0.2538 (4)  | 0.0170 (13) |
| C24  | 0.8475 (14) | 0.5248 (2)   | 0.2137 (5)  | 0.0204 (15) |
| C25  | 0.7690 (15) | 0.53442 (19) | 0.1293 (5)  | 0.0207 (15) |
| C26  | 0.5582 (14) | 0.51219 (19) | 0.0829 (4)  | 0.0164 (13) |
| I5   | 0.60231 (10) | 0.58298 (2)  | 0.42231 (3) | 0.02666 (11) |
| I6   | −0.04120 (12) | 0.67426 (2)  | 0.67979 (3) | 0.03213 (12) |
| F13  | 0.3626 (9)  | 0.61104 (13) | 0.5966 (2)  | 0.0298 (9)  |
| F14  | −0.1966 (10) | 0.72120 (12) | 0.5061 (3)  | 0.0340 (11) |
| F15  | −0.0233 (11) | 0.71370 (13) | 0.3475 (3)  | 0.0379 (11) |
| F16  | 0.3387 (10) | 0.65460 (13) | 0.3104 (3)  | 0.0340 (10) |
| C33  | 0.3592 (15) | 0.63148 (19) | 0.4533 (4)  | 0.0215 (14) |
| C34  | 0.2700 (15) | 0.63678 (19) | 0.5348 (4)  | 0.0212 (14) |
| C35  | 0.0859 (15) | 0.6674 (2)   | 0.5562 (4)  | 0.0215 (15) |
| C36  | −0.0098 (16) | 0.6922 (2)   | 0.4907 (5)  | 0.0241 (15) |
| C37  | 0.0756 (16) | 0.6885 (2)   | 0.4086 (5)  | 0.0267 (16) |
| C38  | 0.2587 (16) | 0.6584 (2)   | 0.3901 (4)  | 0.0241 (15) |
| I7   | 0.27675 (10) | 0.60812 (2)  | −0.10563 (3)| 0.02281 (10) |
| I8   | 1.08854 (10) | 0.74677 (2)  | −0.05353 (3)| 0.02152 (10) |
| F9   | 0.6582 (9)  | 0.68287 (11) | −0.1413 (2) | 0.0239 (9)  |
| F10  | 1.0852 (9)  | 0.71219 (12) | 0.1364 (2)  | 0.0286 (10) |
| F11  | 0.7847 (10) | 0.65341 (14) | 0.2015 (2)  | 0.0365 (11) |
| F12  | 0.4105 (9)  | 0.61029 (12) | 0.0985 (3)  | 0.0293 (9)  |
| C27  | 0.5318 (13) | 0.64475 (18) | −0.0249 (4) | 0.0164 (13) |
| C28  | 0.6884 (14) | 0.67579 (19) | −0.0565 (4) | 0.0166 (13) |
| C29  | 0.8730 (13) | 0.69920 (18) | −0.0044 (4) | 0.0165 (13) |
| C30  | 0.9054 (15) | 0.6911 (2)   | 0.0830 (4)  | 0.0219 (15) |
| C31  | 0.7507 (16) | 0.6609 (2)   | 0.1168 (4)  | 0.0233 (15) |
| C32  | 0.5630 (14) | 0.63862 (19) | 0.0628 (4)  | 0.0202 (14) |
| S1   | 0.6754 (4)  | 0.40568 (5)  | 0.88863 (11)| 0.0189 (3)  |
| S2   | 1.0703 (4)  | 0.33606 (5)  | 0.91405 (10)| 0.0186 (3)  |
| N1   | 0.9694 (12) | 0.36855 (16) | 0.7688 (3)  | 0.0152 (11) |
| HN1  | 0.905 (14)  | 0.3871 (19)  | 0.732 (4)   | 0.007 (16)* |
| C1   | 0.8985 (13) | 0.37204 (18) | 0.8506 (4)  | 0.0151 (13) |
| C2   | 1.1602 (14) | 0.33784 (19) | 0.7534 (4)  | 0.0162 (13) |
Atomic displacement parameters (Å²)

|   | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{12}$  | $U^{13}$  | $U^{23}$  |
|---|-----------|-----------|-----------|-----------|-----------|-----------|
| I1| 0.0177 (2)| 0.0159 (2)| 0.01700 (19)| 0.00011 (16)| 0.00007 (16)| −0.00234 (17)|
| I2| 0.0263 (2)| 0.0245 (2)| 0.0153 (2)| 0.00547 (19)| −0.00254 (18)| −0.00115 (18)|
| I3| 0.0171 (2)| 0.0170 (2)| 0.01558 (19)| 0.00146 (16)| 0.00089 (16)| −0.00224 (17)|
| I4| 0.0313 (3)| 0.0303 (3)| 0.0169 (2)| 0.0123 (2)| −0.00624 (19)| −0.00536 (19)|
| F1| 0.032 (2)| 0.019 (2)| 0.0194 (19)| −0.0084 (17)| 0.0019 (17)| 0.0046 (17)|
| F2| 0.028 (2)| 0.029 (2)| 0.027 (2)| −0.0075 (18)| −0.0056 (18)| −0.0054 (19)|
| F3| 0.041 (3)| 0.023 (2)| 0.033 (2)| −0.0147 (19)| −0.001 (2)| 0.0100 (19)|
| F4| 0.040 (3)| 0.025 (2)| 0.0168 (19)| −0.0053 (19)| −0.0029 (18)| 0.0056 (17)|
| F5| 0.029 (2)| 0.016 (2)| 0.0167 (18)| −0.0053 (17)| 0.0061 (16)| 0.0037 (16)|
| F6| 0.019 (2)| 0.029 (2)| 0.042 (3)| −0.0079 (18)| −0.0024 (19)| −0.010 (2)|
| F7| 0.032 (2)| 0.024 (2)| 0.044 (3)| −0.0084 (19)| 0.011 (2)| 0.010 (2)|
| F8| 0.029 (2)| 0.028 (2)| 0.0170 (19)| −0.0002 (18)| 0.0002 (17)| 0.0100 (17)|
| C15| 0.010 (3)| 0.016 (3)| 0.019 (3)| 0.000 (2)| 0.002 (2)| −0.001 (3)|
| C16| 0.017 (3)| 0.013 (3)| 0.020 (3)| 0.001 (2)| 0.005 (3)| 0.003 (3)|
| C17| 0.013 (3)| 0.021 (4)| 0.011 (3)| 0.002 (3)| 0.000 (2)| −0.001 (3)|
| C18| 0.016 (3)| 0.019 (4)| 0.018 (3)| −0.002 (3)| −0.001 (3)| −0.002 (3)|
| C19| 0.021 (4)| 0.017 (4)| 0.021 (3)| −0.004 (3)| 0.004 (3)| −0.001 (3)|
| C20| 0.024 (4)| 0.017 (4)| 0.014 (3)| 0.004 (3)| 0.001 (3)| 0.001 (3)|
| C21| 0.017 (3)| 0.009 (3)| 0.010 (3)| 0.001 (2)| 0.003 (2)| −0.001 (2)|
| C22| 0.018 (3)| 0.015 (3)| 0.016 (3)| 0.001 (3)| 0.007 (3)| 0.001 (3)|
C23 0.017 (3) 0.016 (3) 0.016 (3) 0.004 (3) −0.003 (3) −0.002 (3)
C24 0.012 (3) 0.021 (4) 0.028 (4) 0.000 (3) 0.000 (3) −0.008 (3)
C25 0.018 (3) 0.009 (3) 0.035 (4) −0.002 (3) 0.007 (3) 0.006 (3)
C26 0.014 (3) 0.017 (3) 0.018 (3) −0.001 (3) 0.004 (3) 0.002 (3)
I5 0.0251 (2) 0.0252 (3) 0.0291 (2) 0.00486 (19) −0.00180 (19) −0.0083 (2)
I6 0.0376 (3) 0.0383 (3) 0.0218 (2) 0.0001 (2) 0.0099 (2) −0.0023 (2)
F13 0.037 (2) 0.029 (2) 0.024 (2) 0.002 (2) 0.0010 (19) 0.0053 (19)
F14 0.034 (3) 0.031 (3) 0.038 (2) 0.014 (2) 0.012 (2) 0.000 (2)
F15 0.047 (3) 0.033 (3) 0.032 (2) 0.005 (2) −0.002 (2) 0.019 (2)
F16 0.036 (3) 0.045 (3) 0.021 (2) 0.009 (2) 0.0073 (19) 0.0024 (19)
I7 0.0204 (2) 0.0188 (2) 0.0290 (2) −0.00085 (18) −0.00007 (19) −0.00546 (19)
I8 0.0220 (2) 0.0161 (2) 0.0263 (2) −0.00127 (17) 0.00103 (18) 0.00192 (18)
F9 0.029 (2) 0.025 (2) 0.0171 (19) −0.0009 (17) −0.0005 (17) −0.0004 (16)
F10 0.028 (2) 0.036 (3) 0.021 (2) −0.0123 (19) 0.0001 (18) −0.0051 (18)
F11 0.039 (3) 0.052 (3) 0.018 (2) −0.013 (2) 0.0041 (19) 0.004 (2)
F12 0.026 (2) 0.030 (2) 0.034 (2) −0.0084 (19) 0.0099 (19) 0.006 (2)
C27 0.015 (3) 0.009 (3) 0.025 (3) −0.001 (2) −0.002 (2) −0.005 (3)
C28 0.018 (3) 0.016 (3) 0.015 (3) −0.003 (3) 0.001 (3) 0.002 (3)
C29 0.010 (3) 0.012 (3) 0.027 (3) −0.001 (2) 0.004 (3) −0.003 (3)
C30 0.018 (3) 0.027 (4) 0.021 (3) −0.002 (3) 0.001 (3) −0.005 (3)
C31 0.025 (4) 0.030 (4) 0.015 (3) 0.002 (3) 0.002 (3) 0.000 (3)
C32 0.017 (3) 0.016 (3) 0.030 (4) 0.000 (3) 0.010 (3) 0.000 (3)
S1 0.0189 (8) 0.0203 (9) 0.0175 (8) 0.0005 (7) 0.0009 (7) −0.0035 (7)
S2 0.0224 (8) 0.0197 (9) 0.0134 (7) −0.0011 (7) −0.0004 (6) 0.0024 (6)
N1 0.022 (3) 0.014 (3) 0.010 (2) 0.000 (2) 0.000 (2) 0.001 (2)
C1 0.013 (3) 0.016 (3) 0.016 (3) −0.004 (2) 0.002 (2) −0.001 (3)
C2 0.015 (3) 0.017 (3) 0.016 (3) −0.004 (3) −0.002 (3) −0.003 (3)
C3 0.024 (4) 0.030 (4) 0.016 (3) 0.001 (3) 0.002 (3) 0.001 (3)
C4 0.019 (4) 0.035 (4) 0.023 (4) 0.003 (3) −0.003 (3) −0.008 (3)
C5 0.017 (4) 0.027 (4) 0.033 (4) 0.004 (3) −0.004 (3) −0.008 (3)
C6 0.021 (4) 0.019 (4) 0.020 (3) 0.000 (3) −0.003 (3) −0.002 (3)
C7 0.016 (3) 0.018 (3) 0.015 (3) −0.002 (3) −0.004 (3) −0.004 (3)
S3 0.0190 (8) 0.0206 (9) 0.0172 (8) −0.0007 (7) 0.0001 (7) −0.0018 (7)
S4 0.0191 (8) 0.0217 (9) 0.0129 (7) −0.0017 (7) 0.0002 (6) 0.0016 (6)
N2 0.013 (3) 0.020 (3) 0.015 (3) −0.001 (2) −0.002 (2) 0.003 (2)
C8 0.017 (3) 0.018 (3) 0.013 (3) −0.007 (3) −0.001 (3) −0.001 (2)
C9 0.017 (3) 0.019 (3) 0.015 (3) −0.005 (3) −0.004 (3) −0.002 (3)
C10 0.021 (3) 0.022 (4) 0.014 (3) −0.006 (3) 0.003 (3) 0.001 (3)
C11 0.026 (4) 0.025 (4) 0.016 (3) −0.006 (3) 0.007 (3) −0.008 (3)
C12 0.018 (3) 0.019 (4) 0.025 (4) 0.000 (3) 0.003 (3) −0.006 (3)
C13 0.017 (3) 0.022 (4) 0.026 (4) −0.003 (3) −0.004 (3) 0.003 (3)
C14 0.011 (3) 0.020 (3) 0.015 (3) −0.004 (3) −0.002 (3) −0.003 (3)
Geometric parameters (Å, °)

| Bond          | Length  | Bond          | Length  |
|---------------|---------|---------------|---------|
| I1—C15        | 2.094 (6)| F11—C31       | 1.344 (7)|
| I2—C17        | 2.075 (6)| F12—C32       | 1.343 (7)|
| I3—C21        | 2.090 (6)| C27—C28       | 1.396 (9)|
| I4—C23        | 2.080 (6)| C27—C32       | 1.381 (9)|
| F1—C16        | 1.345 (7)| C28—C29       | 1.379 (9)|
| F2—C18        | 1.349 (8)| C29—C30       | 1.387 (9)|
| F3—C19        | 1.336 (7)| C30—C31       | 1.383 (9)|
| F4—C20        | 1.338 (7)| C31—C32       | 1.382 (10)|
| F5—C22        | 1.335 (7)| S1—C1         | 1.679 (6)|
| F6—C24        | 1.348 (8)| S2—C1         | 1.730 (7)|
| F7—C25        | 1.330 (7)| S2—C7         | 1.748 (7)|
| F8—C26        | 1.336 (7)| N1—HN1        | 0.89 (6)|
| C15—C16       | 1.386 (9)| N1—C1         | 1.347 (7)|
| C15—C20       | 1.389 (9)| N1—C2         | 1.401 (8)|
| C16—C17       | 1.391 (9)| C2—C3         | 1.389 (9)|
| C17—C18       | 1.381 (9)| C2—C7         | 1.385 (9)|
| C18—C19       | 1.383 (9)| C3—H3         | 0.9500|
| C19—C20       | 1.373 (10)| C3—C4         | 1.382 (10)|
| C21—C22       | 1.393 (8)| C4—H4         | 0.9500|
| C21—C26       | 1.387 (8)| C4—C5         | 1.398 (10)|
| C22—C23       | 1.389 (9)| C5—H5         | 0.9500|
| C23—C24       | 1.380 (10)| C5—C6         | 1.387 (9)|
| C24—C25       | 1.377 (10)| C6—H6         | 0.9500|
| C25—C26       | 1.384 (9)| C6—C7         | 1.390 (9)|
| I5—C33        | 2.080 (6)| S3—C8         | 1.679 (7)|
| I6—C35        | 2.073 (6)| S4—C8         | 1.741 (7)|
| F13—C34       | 1.351 (8)| S4—C14        | 1.748 (6)|
| F14—C36       | 1.346 (8)| N2—HN2        | 0.85 (3)|
| F15—C37       | 1.339 (8)| N2—C8         | 1.343 (8)|
| F16—C38       | 1.331 (7)| N2—C9         | 1.398 (8)|
| C33—C34       | 1.380 (9)| C9—C10        | 1.396 (9)|
| C33—C38       | 1.400 (10)| C9—C14        | 1.403 (9)|
| C34—C35       | 1.402 (9)| C10—H10       | 0.9500|
| C35—C36       | 1.373 (10)| C10—C11       | 1.369 (10)|
| C36—C37       | 1.375 (10)| C11—H11       | 0.9500|
| C37—C38       | 1.375 (10)| C11—C12       | 1.405 (9)|
| I7—C27        | 2.069 (6)| C12—H12       | 0.9500|
| I8—C29        | 2.085 (6)| C12—C13       | 1.384 (9)|
| F9—C28        | 1.342 (7)| C13—H13       | 0.9500|
| F10—C30       | 1.334 (8)| C13—C14       | 1.394 (9)|
| C16—C15—I1    | 122.6 (5)| C28—C29—C30   | 118.2 (6)|
| C16—C15—C20   | 117.1 (6)| C30—C29—I8    | 120.1 (5)|
| C20—C15—I1    | 120.2 (5)| F10—C30—C29   | 121.1 (6)|
| F1—C16—C15    | 118.3 (6)| F10—C30—C31   | 118.4 (6)|
| F1—C16—C17    | 118.4 (6)| C31—C30—C29   | 120.6 (6)|
| Bond          | Angle (°) (esd) | Bond          | Angle (°) (esd) | Bond          | Angle (°) (esd) |
|---------------|----------------|---------------|----------------|---------------|----------------|
| C15—C16—C17  | 123.2 (6)      | F11—C31—C30  | 119.9 (6)      |                |                |
| C16—C17—I2   | 121.1 (5)      | F11—C31—C32  | 120.5 (6)      |                |                |
| C18—C17—I2   | 121.8 (5)      | C32—C31—C30  | 119.6 (6)      |                |                |
| C18—C17—C16  | 117.1 (6)      | F12—C32—C27  | 120.5 (6)      |                |                |
| F2—C18—C17   | 120.3 (6)      | F12—C32—C31  | 117.7 (6)      |                |                |
| F2—C18—C19   | 118.0 (6)      | C27—C32—C31  | 121.8 (6)      |                |                |
| C17—C18—C19  | 121.7 (6)      | C1—S2—C7     | 92.2 (3)       |                |                |
| F3—C19—I2    | 120.4 (6)      | C1—N1—HN1    | 117 (4)        |                |                |
| F3—C19—C20   | 120.2 (6)      | C1—N1—C2     | 115.5 (5)      |                |                |
| C20—C19—I2   | 120.0 (6)      | C2—N1—HN1    | 127 (4)        |                |                |
| F4—C20—C15   | 119.3 (6)      | S1—C1—S2     | 123.3 (4)      |                |                |
| F4—C20—C19   | 118.3 (6)      | N1—C1—S1     | 126.5 (5)      |                |                |
| C19—C20—I2   | 121.6 (6)      | N1—C1—S2     | 110.2 (5)      |                |                |
| C22—C21—I3   | 122.5 (5)      | C3—C2—N1     | 126.3 (6)      |                |                |
| C26—C21—I3   | 120.1 (4)      | C7—C2—N1     | 112.2 (6)      |                |                |
| C26—C21—C22  | 117.3 (6)      | C7—C2—C3     | 121.5 (6)      |                |                |
| F5—C22—C21   | 118.4 (6)      | C2—C3—H3     | 121.1          |                |                |
| F5—C22—C23   | 118.9 (6)      | C4—C3—C2     | 117.9 (6)      |                |                |
| C23—C22—C21  | 122.7 (6)      | C4—C3—H3     | 121.1          |                |                |
| C22—C23—I4   | 120.7 (5)      | C3—C4—H4     | 119.4          |                |                |
| C24—C23—I4   | 122.0 (5)      | C3—C4—C5     | 121.1 (7)      |                |                |
| C24—C23—C22  | 117.3 (6)      | C5—C4—H4     | 119.4          |                |                |
| F6—C24—C23   | 120.0 (6)      | C4—C5—H5     | 119.8          |                |                |
| F6—C24—C25   | 117.8 (6)      | C6—C5—C4     | 120.5 (7)      |                |                |
| C25—C24—C23  | 122.2 (6)      | C6—C5—H5     | 119.8          |                |                |
| F7—C25—C24   | 120.7 (6)      | C5—C6—H6     | 120.8          |                |                |
| F7—C25—C26   | 120.3 (6)      | C5—C6—C7     | 118.5 (6)      |                |                |
| C24—C25—C26  | 118.9 (6)      | C7—C6—H6     | 120.8          |                |                |
| F8—C26—C21   | 120.4 (6)      | C2—C7—S2     | 109.9 (5)      |                |                |
| F8—C26—C25   | 117.9 (6)      | C2—C7—C6     | 120.5 (6)      |                |                |
| C25—C26—C21  | 121.6 (6)      | C6—C7—S2     | 129.6 (5)      |                |                |
| C34—C33—I5   | 121.5 (5)      | C8—S4—C14    | 92.1 (3)       |                |                |
| C34—C33—C38  | 117.3 (6)      | C8—N2—HN2    | 129 (5)        |                |                |
| C38—C33—I5   | 121.0 (5)      | C8—N2—C9     | 116.3 (6)      |                |                |
| F13—C34—C33  | 118.3 (6)      | C9—N2—HN2    | 113 (5)        |                |                |
| F13—C34—C35  | 118.6 (6)      | S3—C8—S4     | 123.3 (4)      |                |                |
| C33—C34—C35  | 123.1 (6)      | N2—C8—S3     | 126.7 (5)      |                |                |
| C34—C35—I6   | 121.7 (5)      | N2—C8—S4     | 110.0 (5)      |                |                |
| C36—C35—I6   | 121.7 (5)      | N2—C9—C14    | 111.7 (6)      |                |                |
| C36—C35—C34  | 116.6 (6)      | C10—C9—N2    | 127.3 (6)      |                |                |
| F14—C36—C35  | 119.6 (6)      | C10—C9—C14   | 121.0 (6)      |                |                |
| F14—C36—C37  | 117.8 (6)      | C9—C10—H10   | 121.0          |                |                |
| C35—C36—C37  | 122.6 (7)      | C11—C10—C9   | 117.9 (6)      |                |                |
| F15—C37—C36  | 120.0 (6)      | C11—C10—H10  | 121.0          |                |                |
| F15—C37—C38  | 120.8 (6)      | C10—C11—H11  | 119.3          |                |                |
| C38—C37—C36  | 119.2 (7)      | C10—C11—C12  | 121.4 (6)      |                |                |
| F16—C38—C33  | 119.7 (6)      | C12—C11—H11  | 119.3          |                |                |
| F16—C38—C37  | 119.1 (6)      | C11—C12—H12  | 119.4          |                |                |
C37—C38—C33 121.2 (6)  C13—C12—C11 121.2 (6)
C28—C27—I7 121.9 (5)  C13—C12—H12 119.4
C32—C27—I7 121.2 (5)  C12—C13—C14 117.7 (6)
C9—C28—C27 118.2 (6)  C14—C13—H13 121.1
F9—C28—C29 118.9 (6)  C9—C14—S4 109.9 (5)
C29—C28—C27 122.9 (6)  C13—C14—S4 129.3 (5)
C28—C29—I8 121.6 (5)  C13—C14—C9 120.7 (6)
I1—C15—C16—F1 2.2 (8)  C35—C36—C37—C38 1.5 (11)
I1—C15—C16—C17 −176.3 (5)  C36—C37—C38—F16 179.3 (6)
I1—C15—C20—F4 −1.3 (8)  C36—C37—C38—C33 0.0 (11)
I1—C15—C20—C19 176.4 (5)  C38—C33—C34—F13 179.5 (6)
I2—C17—C18—F2 −0.2 (9)  C38—C33—C34—C35 0.1 (10)
I2—C17—C18—C19 −178.3 (5)  I7—C27—C28—F9 3.5 (8)
I3—C21—C22—F5 −2.7 (8)  I7—C27—C28—C29 −176.2 (5)
I3—C21—C22—C23 176.7 (5)  I7—C27—C32—F12 −4.9 (9)
I3—C21—C26—F8 1.3 (8)  I7—C27—C32—C31 174.8 (5)
I3—C21—C26—C25 −175.7 (5)  I8—C29—C30—F10 −3.5 (9)
I4—C23—C24—F6 −0.5 (9)  I8—C29—C30—C31 176.4 (5)
I4—C23—C24—C25 179.5 (5)  F9—C28—C29—I8 3.1 (8)
F1—C16—C17—I2 0.4 (8)  F9—C28—C29—C30 −179.0 (6)
F1—C16—C17—C18 −179.0 (6)  F10—C30—C31—F11 −0.2 (10)
F2—C18—C19—F3 −0.4 (10)  F10—C30—C31—C32 −179.9 (6)
F2—C18—C19—C20 −179.4 (6)  F11—C31—C32—F12 2.2 (10)
F3—C19—C20—F4 −0.8 (10)  F11—C31—C32—C27 −177.6 (6)
F3—C19—C20—C15 −178.5 (6)  C27—C28—C29—I8 −177.3 (5)
F5—C22—C23—I4 −0.1 (8)  C27—C28—C29—C30 0.6 (10)
F5—C22—C23—C24 177.8 (6)  C28—C27—C32—F12 177.4 (6)
F6—C24—C25—F7 2.8 (10)  C28—C27—C32—C31 −2.9 (10)
F6—C24—C25—C26 179.4 (6)  C28—C29—C30—F10 178.6 (6)
F7—C25—C26—F8 −1.0 (10)  C28—C29—C30—C31 −1.5 (10)
F7—C25—C26—C21 176.0 (6)  C29—C30—C31—F11 179.9 (6)
C15—C16—C17—I2 179.0 (5)  C29—C30—C31—C32 0.2 (10)
C15—C16—C17—C18 −0.4 (10)  C30—C31—C32—F12 −178.1 (6)
C16—C15—C20—F4 −177.5 (6)  C30—C31—C32—C27 2.1 (10)
C16—C15—C20—C19 0.2 (10)  C32—C27—C28—F9 −178.8 (6)
C16—C17—C18—I2 179.2 (6)  C32—C27—C28—C29 1.5 (10)
C16—C17—C18—C19 1.2 (10)  N1—C2—C3—C4 −179.8 (6)
C17—C18—C19—I3 177.7 (6)  N1—C2—C7—S2 −0.7 (7)
C17—C18—C19—C20 −1.3 (11)  N1—C2—C7—C6 −179.7 (6)
C18—C19—C20—F4 178.3 (6)  C1—S2—C7—C2 0.5 (5)
C18—C19—C20—C15 0.6 (10)  C1—S2—C7—C6 179.4 (7)
C20—C15—C16—I1 178.3 (5)  C1—N1—C2—C3 179.5 (6)
C20—C15—C16—C17 −0.2 (10)  C1—N1—C2—C7 0.6 (8)
C21—C22—C23—I4 −179.5 (5)  C2—N1—C1—S1 −179.8 (5)
C21—C22—C23—C24 −1.5 (10)  C2—N1—C1—S2 −0.2 (7)
C22—C21—C26—F8 177.6 (6)  C2—C3—C4—C5 0.8 (11)
Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| N1—H1···S3 | 0.89 (6) | 2.51 (6) | 3.376 (6) | 165 (5) |
| C3—H3···I1 | 0.95 | 3.10 | 3.976 (7) | 154 |
| N2—H2···S1 | 0.85 (3) | 2.52 (3) | 3.360 (6) | 169 (7) |
| C10—H10···I3i | 0.95 | 3.09 | 4.006 (6) | 161 |

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

1,3-Benzothiazole-2-thiol–1,2,4,5-tetrafluoro-3,6-diiodobenzene (2/1) (2MBZTH_14F4DIB)

Crystal data

C_{6}F_{4}I_{2}·2C_{7}H_{5}NS_{2} \quad V = 1158.61 (8) Å³

M_r = 736.34 \quad Z = 2

Monoclinic, P2_1/n \quad F(000) = 700

a = 5.5057 (2) Å \quad D_x = 2.111 Mg m⁻³

b = 15.6087 (7) Å \quad Mo Kα radiation, λ = 0.71073 Å

C = 13.5194 (6) Å \quad Cell parameters from 9937 reflections

β = 94.259 (2)° \quad θ = 2.6–30.1°
µ = 3.12 mm⁻¹  
T = 100 K  
Needle, colourless  
0.17 × 0.09 × 0.04 mm

Data collection
Buer D8 Venture Photon 2 diffractometer  
Radiation source: Incoatec ImS  
φ and ω scans  
Absorption correction: multi-scan  
(SADABS; Bruker, 2017)  
Tmin = 0.559, Tmax = 0.746  
22270 measured reflections

Refinement
Refinement on F²  
Least-squares matrix: full  
R[F² > 2σ(F²)] = 0.027  
wR(F²) = 0.057  
S = 1.15  
3402 reflections  
149 parameters  
0 restraints  
Hydrogen site location: mixed  
H atoms treated by a mixture of independent and constrained refinement

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   | Ueq | Uiso* |
|-----|-----|-----|-----|-------|
| I1  | 0.09620 (3) | 0.50667 (2) | 0.18841 (2) | 0.01843 (6) |
| F1  | 0.4987 (3) | 0.36936 (10) | 0.13226 (14) | 0.0285 (4) |
| F2  | 0.8063 (3) | 0.36478 (10) | −0.00971 (13) | 0.0267 (4) |
| C8  | 0.3371 (5) | 0.50280 (17) | 0.0761 (2) | 0.0179 (5) |
| C9  | 0.4956 (5) | 0.43488 (17) | 0.0673 (2) | 0.0210 (6) |
| C10 | 0.6546 (5) | 0.43202 (17) | −0.0070 (2) | 0.0190 (5) |
| S1  | −0.29400 (12) | 0.50758 (4) | 0.36239 (5) | 0.01927 (14) |
| S2  | 0.07419 (13) | 0.64355 (4) | 0.42496 (5) | 0.02067 (15) |
| N1  | −0.2698 (4) | 0.60239 (14) | 0.52996 (18) | 0.0175 (5) |
| SN1 | −0.379 (6) | 0.577 (2) | 0.548 (3) | 0.024 (9)* |
| C1  | −0.1812 (5) | 0.58112 (16) | 0.4433 (2) | 0.0166 (5) |
| C2  | −0.1450 (5) | 0.66593 (16) | 0.5855 (2) | 0.0173 (5) |
| C3  | −0.2004 (5) | 0.69814 (17) | 0.6767 (2) | 0.0225 (6) |
| H3  | −0.337581 | 0.678027 | 0.708502 | 0.027* |
| C4  | −0.0491 (6) | 0.76050 (19) | 0.7197 (2) | 0.0271 (7) |
| H4  | −0.083090 | 0.783531 | 0.782261 | 0.032* |
| C5  | 0.1516 (6) | 0.79028 (19) | 0.6737 (2) | 0.0269 (7) |
| H5  | 0.253410 | 0.832708 | 0.705534 | 0.032* |
| C6  | 0.2060 (5) | 0.75900 (17) | 0.5816 (2) | 0.0237 (6) |
### Atomic displacement parameters (Å²)

|   | U₁¹  | U₂²  | U₃³  | U₁²  | U₁³  | U₂³  |
|---|------|------|------|------|------|------|
| I1 | 0.01632 (9) | 0.02275 (9) | 0.01671 (10) | 0.00025 (7) | 0.00446 (6) | −0.00296 (6) |
| F1 | 0.0349 (10) | 0.0250 (8) | 0.0273 (10) | 0.0077 (7) | 0.0135 (8) | 0.0069 (7) |
| F2 | 0.0284 (9) | 0.0247 (8) | 0.0283 (10) | 0.0099 (7) | 0.0107 (8) | 0.0003 (7) |
| C8 | 0.0152 (12) | 0.0226 (13) | 0.0162 (14) | −0.0007 (10) | 0.0035 (10) | −0.0063 (10) |
| C9 | 0.0216 (14) | 0.0217 (13) | 0.0200 (15) | −0.0003 (11) | 0.0031 (12) | −0.0003 (10) |
| C10 | 0.0169 (12) | 0.0196 (12) | 0.0207 (15) | 0.0022 (10) | 0.0019 (11) | −0.0047 (10) |
| S1 | 0.0194 (3) | 0.0228 (3) | 0.0161 (3) | −0.0054 (3) | 0.0047 (3) | −0.0013 (2) |
| S2 | 0.0195 (3) | 0.0236 (3) | 0.0198 (4) | −0.0062 (3) | 0.0071 (3) | −0.0005 (3) |
| N1 | 0.0170 (11) | 0.0165 (10) | 0.0196 (13) | −0.0033 (9) | 0.0057 (9) | 0.0011 (9) |
| C1 | 0.0154 (12) | 0.0176 (12) | 0.0170 (14) | −0.0002 (10) | 0.0030 (10) | 0.0036 (9) |
| C2 | 0.0189 (13) | 0.0148 (11) | 0.0184 (14) | −0.0022 (10) | 0.0016 (11) | 0.0020 (9) |
| C3 | 0.0227 (14) | 0.0226 (13) | 0.0230 (16) | 0.0016 (11) | 0.0061 (12) | 0.0009 (11) |
| C4 | 0.0322 (16) | 0.0254 (14) | 0.0238 (17) | 0.0003 (12) | 0.0036 (13) | −0.0066 (12) |
| C5 | 0.0273 (15) | 0.0221 (14) | 0.0307 (18) | −0.0041 (12) | −0.0022 (13) | −0.0029 (12) |
| C6 | 0.0232 (14) | 0.0194 (13) | 0.0286 (17) | −0.0051 (11) | 0.0023 (12) | 0.0013 (11) |
| C7 | 0.0173 (12) | 0.0170 (12) | 0.0193 (15) | −0.0007 (10) | 0.0027 (11) | 0.0026 (10) |

### Geometric parameters (Å, °)

|   | I1—C8 | F1—C9 | F2—C10 | C8—C9 | C8—C10\(i\) | C9—C10 | S1—C1 | S2—C7 | N1—HN1 | N1—C1 | C9—C8—I1 | C9—C8—C10\(i\) | C10\(i\)—C8—I1 | F1—C9—C8 | F1—C9—C10 | C10—C9—C8 | F2—C10—C8 | C9—C10—C8 | C1—S2—C7 | C1—N1—HN1 | C1—N1—C1 |
|---|-------|-------|--------|-------|-------------|--------|-------|-------|-------|-------|---------|----------------|---------------|-------------|---------|---------|---------|---------|---------|--------|---------|-------|
|   | 2.090 (3) | 1.348 (3) | 1.344 (3) | 1.384 (4) | 1.385 (4) | 1.381 (4) | 1.673 (3) | 1.743 (3) | 0.78 (3) | 1.344 (3) | 121.6 (2) | 116.8 (2) | 121.59 (19) | 120.2 (2) | 118.1 (2) | 121.7 (3) | 120.2 (2) | 118.3 (2) | 121.4 (2) | 92.03 (13) | 119 (3) | 0.779762 | 0.549629 | 0.028* | 0.0542 (5) | 0.69629 (16) | 0.5379 (2) | 0.0178 (5) | 0.342177 | 0.579629 | 0.549629 | 0.028* |

### Geometric parameters (Å, °)
Supporting Information

C1—N1—C2 116.7 (2) C5—C6—H6 121.1
C2—N1—HN1 124 (3) C7—C6—C5 117.7 (3)
S1—C1—S2 123.72 (16) C7—C6—H6 121.1
N1—C1—S1 126.7 (2) C2—C7—S2 110.2 (2)
N1—C1—C2 109.5 (2) C6—C7—S2 128.9 (2)
N1—C2—C7 111.5 (2) C6—C7—C2 120.8 (3)

Symmetry code: (i) −x+1, −y+1, −z.

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|----------|------|-------|-------|--------|
| N1—HN1···S1ii | 0.78 (3) | 2.60 (3) | 3.369 (2) | 170 (3) |
| C3—H3···F1iii | 0.95 | 2.50 | 3.333 (3) | 146 |
| C6—H6···F2iv | 0.95 | 2.44 | 3.357 (3) | 162 |

Symmetry codes: (ii) −x−1, −y+1, −z+1; (iii) −x, −y+1, −z+1; (iv) −x+3/2, y+1/2, −z+1/2.

1,3-Benzothiazole-2-thiol–1,3,5-trifluoro-2,4,6-triiodobenzene (1/1) (MBZTH_135F3I3B)

Crystal data

C6F3I3·C7H5NS2

F(000) = 1232

M_r = 677.00

Monoclinic, P2_1/c

a = 15.2665 (6) Å

b = 4.7380 (2) Å

θ = 3.1–28.4°

Cell parameters from 9894 reflections

µ = 5.86 mm⁻¹

T = 100 K

V = 1677.15 (12) Å³

Plate, colourless

Z = 4

0.16 × 0.08 × 0.05 mm

Data collection

Bruker D8 Venture Photon 2 diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2017)

T = 0.610, T_max = 0.746

35222 measured reflections

4212 independent reflections

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sup-70
supporting information

3611 reflections with $I > 2\sigma(I)$
\[ R_{int} = 0.057 \]
\[ \theta_{\text{max}} = 28.4^\circ, \theta_{\text{min}} = 2.2^\circ \]
\[ h = -19\rightarrow 20 \]
\[ k = -6\rightarrow 6 \]
\[ l = -30\rightarrow 31 \]

Refinement

Refinement on $F^2$
Least-squares matrix: full
\[ R[F^2 > 2\sigma(F^2)] = 0.024 \]
\[ wR(F^2) = 0.052 \]
\[ S = 1.18 \]
4212 reflections
203 parameters
1 restraint

Primary atom site location: dual
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
\[ w = 1/[\sigma^2(F_o^2) + 4.0997P] \]
where $P = (F_o^2 + 2F_c^2)/3$

\[ (\Delta/\sigma)_{\text{max}} = 0.003 \]
\[ \Delta\rho_{\text{max}} = 0.67 \text{ e Å}^{-3} \]
\[ \Delta\rho_{\text{min}} = -0.73 \text{ e Å}^{-3} \]

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     | $U_{iso}/U_{eq}$ |
|----|-------|-------|-------|------------------|
| I1 | 0.7016 (2) | 0.58105 (5) | 0.55847 (2) | 0.01897 (6) |
| I2 | 0.68104 (2) | -0.18871 (6) | 0.76567 (2) | 0.02456 (7) |
| I3 | 0.37648 (2) | -0.11384 (6) | 0.58903 (2) | 0.02243 (6) |
| F1 | 0.75133 (13) | 0.2505 (5) | 0.67513 (9) | 0.0224 (5) |
| F2 | 0.49679 (14) | -0.2816 (5) | 0.70076 (9) | 0.0247 (5) |
| F3 | 0.51145 (13) | 0.3138 (5) | 0.54048 (9) | 0.0224 (5) |
| C8 | 0.6317 (2) | 0.2972 (7) | 0.60769 (15) | 0.0161 (7) |
| C9 | 0.6683 (2) | 0.1805 (8) | 0.65793 (15) | 0.0168 (7) |
| C10 | 0.6242 (2) | -0.0129 (8) | 0.69039 (15) | 0.0170 (7) |
| C11 | 0.5404 (2) | -0.0910 (8) | 0.67047 (15) | 0.0195 (7) |
| C12 | 0.5008 (2) | 0.0172 (8) | 0.62016 (15) | 0.0170 (7) |
| C13 | 0.5477 (2) | 0.2102 (8) | 0.58980 (15) | 0.0177 (7) |
| S1 | 0.85079 (6) | 1.0132 (2) | 0.49475 (4) | 0.02217 (19) |
| S2 | 0.81182 (6) | 0.6044 (2) | 0.39819 (4) | 0.02095 (19) |
| N1 | 0.9677 (2) | 0.7071 (7) | 0.43672 (13) | 0.0205 (6) |
| HN1 | 1.011 (2) | 0.777 (10) | 0.4571 (18) | 0.040 (14)* |
| C1 | 0.8842 (2) | 0.7835 (8) | 0.44595 (15) | 0.0185 (7) |
| C2 | 0.9777 (2) | 0.5076 (8) | 0.39362 (16) | 0.0203 (7) |
| C3 | 1.0556 (3) | 0.3935 (9) | 0.37636 (18) | 0.0278 (9) |
| H3 | 1.110354 | 0.453291 | 0.393589 | 0.033* |
| C4 | 1.0518 (3) | 0.1913 (9) | 0.33364 (18) | 0.0303 (9) |
| H4 | 1.104619 | 0.107792 | 0.322010 | 0.036* |
| C5 | 0.9720 (3) | 0.1072 (9) | 0.30721 (18) | 0.0304 (9) |
| H5 | 0.971216 | -0.031349 | 0.277614 | 0.036* |
| C6 | 0.8934 (3) | 0.2228 (8) | 0.32348 (17) | 0.0260 (8) |
| H6 | 0.838874 | 0.165478 | 0.305463 | 0.031* |
| C7 | 0.8970 (2) | 0.4256 (8) | 0.36705 (15) | 0.0194 (7) |
**Atomic displacement parameters (Å²)**

|   | U¹¹ | U¹² | U¹³ | U²² | U²³ | U³³ |
|---|-----|-----|-----|-----|-----|-----|
| I1 | 0.01882 (12) | 0.01952 (12) | 0.01885 (12) | −0.00162 (9) | 0.00346 (9) | −0.00136 (9) |
| I2 | 0.02741 (13) | 0.02835 (14) | 0.01735 (12) | 0.00846 (10) | −0.00403 (9) | 0.00139 (10) |
| I3 | 0.01542 (11) | 0.03132 (14) | 0.02042 (12) | −0.00464 (9) | −0.00022 (9) | −0.00501 (10) |
| F1 | 0.0151 (10) | 0.0285 (12) | 0.0230 (11) | −0.00088 (9) | −0.00397 (8) | −0.00378 (9) |
| F2 | 0.0261 (11) | 0.02539 (12) | 0.0221 (11) | −0.00066 (9) | 0.00140 (9) | 0.00767 (9) |
| F3 | 0.0198 (10) | 0.0286 (12) | 0.0184 (10) | 0.0021 (9) | −0.0027 (8) | 0.0066 (9) |
| C8 | 0.0174 (16) | 0.0146 (17) | 0.0163 (16) | −0.0009 (13) | 0.0025 (13) | −0.0019 (13) |
| C9 | 0.0137 (16) | 0.0191 (17) | 0.0175 (17) | 0.0018 (13) | −0.0010 (13) | −0.0058 (14) |
| C10 | 0.0180 (17) | 0.0184 (17) | 0.0142 (16) | 0.0044 (13) | −0.0036 (13) | −0.0014 (14) |
| C11 | 0.0206 (18) | 0.0202 (18) | 0.0180 (17) | −0.0001 (14) | 0.0032 (14) | −0.0004 (14) |
| C12 | 0.0130 (16) | 0.0198 (18) | 0.0180 (17) | −0.0001 (13) | 0.0000 (13) | −0.0020 (14) |
| C13 | 0.0159 (16) | 0.0208 (18) | 0.0163 (16) | 0.0037 (14) | 0.0007 (13) | −0.0005 (14) |
| S1 | 0.0217 (5) | 0.0233 (5) | 0.0217 (5) | −0.0019 (4) | 0.0019 (4) | −0.0025 (4) |
| S2 | 0.0158 (4) | 0.0237 (5) | 0.0229 (5) | −0.0027 (3) | −0.0021 (3) | −0.0020 (4) |
| N1 | 0.0183 (15) | 0.0251 (17) | 0.0178 (15) | −0.0035 (13) | −0.0019 (12) | −0.0011 (13) |
| C1 | 0.0178 (17) | 0.0183 (18) | 0.0191 (17) | −0.0026 (14) | −0.0004 (14) | 0.0025 (14) |
| C2 | 0.0218 (18) | 0.0196 (18) | 0.0194 (18) | 0.0014 (14) | −0.0019 (14) | 0.0022 (15) |
| C3 | 0.0201 (19) | 0.0333 (2) | 0.0302 (2) | 0.0003 (16) | −0.0023 (16) | 0.0032 (18) |
| C4 | 0.033 (2) | 0.031 (2) | 0.028 (2) | 0.0097 (18) | 0.0072 (17) | 0.0002 (18) |
| C5 | 0.043 (3) | 0.022 (2) | 0.026 (2) | 0.0013 (18) | 0.0067 (18) | −0.0007 (17) |
| C6 | 0.033 (2) | 0.022 (2) | 0.0223 (19) | −0.0059 (16) | −0.0037 (16) | 0.0010 (16) |
| C7 | 0.0198 (18) | 0.0199 (18) | 0.0183 (17) | −0.0008 (14) | 0.0004 (14) | 0.0026 (14) |

**Geometric parameters (Å, °)**

|   |   |   |   |   |   |   |
|---|---|---|---|---|---|---|
| I1—C8 | 2.095 (3) | S2—C7 | 1.742 (4) |
| I2—C10 | 2.082 (3) | N1—HN1 | 0.857 (19) |
| I3—C12 | 2.088 (3) | N1—C1 | 1.353 (5) |
| F1—C9 | 1.349 (4) | N1—C2 | 1.391 (5) |
| F2—C11 | 1.343 (4) | C2—C3 | 1.386 (5) |
| F3—C13 | 1.338 (4) | C2—C7 | 1.402 (5) |
| C8—C9 | 1.381 (5) | C3—H3 | 0.9500 |
| C8—C13 | 1.389 (5) | C3—C4 | 1.378 (6) |
| C9—C10 | 1.384 (5) | C4—H4 | 0.9500 |
| C10—C11 | 1.388 (5) | C4—C5 | 1.393 (6) |
| C11—C12 | 1.384 (5) | C5—H5 | 0.9500 |
| C12—C13 | 1.379 (5) | C5—C6 | 1.390 (6) |
| S1—C1 | 1.671 (4) | C6—H6 | 0.9500 |
| S2—C1 | 1.743 (4) | C6—C7 | 1.394 (5) |
| C9—C8—I1 | 121.6 (3) | S1—C1—S2 | 122.8 (2) |
| C9—C8—C13 | 117.3 (3) | N1—C1—S1 | 127.4 (3) |
| C13—C8—I1 | 121.1 (3) | N1—C1—S2 | 109.8 (3) |
| F1—C9—C8 | 118.8 (3) | N1—C2—C7 | 112.1 (3) |
| F1—C9—C10 | 118.6 (3) | C3—C2—N1 | 127.1 (4) |
C8—C9—C10 122.6 (3) C3—C2—C7 120.8 (4)  
C9—C10—I2 122.0 (3) C2—C3—H3 120.8  
C9—C10—C11 117.5 (3) C4—C3—C2 118.5 (4)  
C11—C10—I2 120.5 (3) C4—C3—H3 120.8  
C2—C11—C12 119.0 (3) C5—C4—H4 119.4  
C12—C11—C10 122.3 (3) C5—C4—C3 121.1 (4)  
C11—C12—I3 121.9 (3) C4—C5—H5 119.5  
C9—C10—I2 120.5 (3) C6—C5—C4 121.0 (4)  
C3—C12—C11 117.6 (3) C6—C5—H5 119.5  
F3—C13—C8 118.7 (3) C5—C6—C7 121.0  
F3—C13—C12 118.6 (3) C5—C6—H6 121.0  
C12—C13—C8 122.7 (3) C7—C6—H6 121.0  
C7—S2—C1 92.24 (18) C2—C7—S2 117.9 (4)  
C1—N1—C2 115.9 (3) C3—C2—C7—S2 120.6 (3)  
C2—N1—HN1 123 (3) C3—C2—C7—C6 129.5 (3)  
Hydrogen-bond geometry (Å, °)  

\[
\begin{array}{cccc}
D & H \cdots A & D & H \cdots A & D \cdots A & D \cdots H \cdots A \\
N1 \cdots HN1 & 0.86 (2) & 2.54 (2) & 3.389 (3) & 172 (4)  \\
C3 \cdots H3 \cdots H1 & 0.95 & 3.03 & 3.928 (4) & 159  \\
\end{array}
\]

Symmetry codes: (i) \(-x+2, -y+2, -z+1\); (ii) \(-x, -y+1, -z+1\).
Crystal data

C$_4$I$_4$C$_2$H$_4$NS$_2$  
$M_r = 698.86$  
Triclinic, $P \bar{1}$  
$a = 7.4085$ (6) Å  
b = 10.8180 (9) Å  
c = 11.1989 (10) Å  
$a = 66.616^\circ$  
$\beta = 70.765^\circ$  
$\gamma = 70.792^\circ$  
$V = 757.20$ (11) Å$^3$  
$Z = 2$  
$F(000) = 620$  
$D_r = 3.065$ Mg m$^{-3}$  
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å  
Cell parameters from 9908 reflections  
$\theta = 2.4$–27.6$^\circ$  
$\mu = 8.48$ mm$^{-1}$  
$T = 100$ K  
Block, yellow  
$0.08 \times 0.07 \times 0.07$ mm

Data collection

Bruker D8 Venture Photon 2 diffractometer  
Radiation source: Incoatec I$_\alpha$S  
$\varphi$ and $\omega$ scans  
Absorption correction: multi-scan  
(SADABS; Bruker, 2017)  
$\theta_{min} = 0.589$, $\theta_{max} = 0.746$  
$T_{min} = 0.589$, $T_{max} = 0.746$  
3484 independent reflections  
3037 reflections with $I > 2\sigma(I)$  
22463 measured reflections

Refinement

Refinement on $F^2$  
Least-squares matrix: full  
$R[F^2 > 2\sigma(F^2)] = 0.029$  
wR($F^2$) = 0.072  
$S = 1.13$  
3484 reflections  
159 parameters  
7 restraints  
Primary atom site location: dual  
Hydrogen site location: mixed  
H atoms treated by a mixture of independent and constrained refinement  
$w = 1/[\sigma^2(F^2) + (0.0264P)^2 + 3.2353P]$  
$\Delta(\sigma)_{max} = 0.002$  
$\Delta\rho_{max} = 1.43$ e Å$^{-3}$  
$\Delta\rho_{min} = -1.76$ 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 (Å$^2$)

|     | x    | y    | z    | $U_{eq}$/$U_{eq}$ | Occ. (<1) |
|-----|------|------|------|-----------------|-----------|
| I1  | 0.18703 (5) | 0.90012 (3) | 0.68231 (3) | 0.02172 (9) |          |
| I2  | 0.32280 (5) | 0.95731 (4) | 0.33480 (3) | 0.02290 (9) |          |
| C8  | 0.0936 (8)  | 0.9743 (5)  | 0.5033 (6)  | 0.0239 (11) |          |
| I3  | 0.72172 (6) | 0.21253 (4) | 0.95745 (4) | 0.03285 (11) |          |
| I4  | 0.76779 (5) | -0.15110 (4) | 1.05340 (4) | 0.02545 (10) |          |
| C9A | 0.9080 (15) | 0.0083 (10) | 1.0028 (9)  | 0.016 (3) | 0.529 (19) |
| C9B | 0.9990 (16) | -0.0669 (12) | 1.0232 (11) | 0.016 (3) | 0.471 (19) |
| S1  | 0.33205 (18) | 0.46883 (15) | 0.92558 (14) | 0.0257 (3) |          |
| S2  | 0.34982 (18) | 0.53599 (14) | 0.63493 (13) | 0.0224 (3) |          |
### Atomic displacement parameters (Å²)

|     | $U^{11}$   | $U^{22}$   | $U^{33}$   | $U^{12}$   | $U^{13}$   | $U^{23}$   |
|-----|------------|------------|------------|------------|------------|------------|
| I1  | 0.01984 (17) | 0.02418 (18) | 0.02146 (18) | −0.00095 (13) | −0.00713 (13) | −0.00908 (13) |
| I2  | 0.01628 (16) | 0.02862 (19) | 0.02166 (18) | −0.00312 (13) | −0.00118 (13) | −0.01016 (14) |
| C8  | 0.004 (3)   | 0.020 (2)   | 0.025 (3)   | 0.002 (2)   | −0.006 (2)   | −0.009 (2)   |
| I3  | 0.0351 (2)  | 0.0308 (2)  | 0.0316 (2)  | 0.01363 (16) | −0.01664 (17) | −0.01885 (16) |
| I4  | 0.02582 (18) | 0.02565 (18) | 0.02593 (19) | −0.01160 (14) | −0.00886 (14) | −0.00245 (14) |
| C9A | 0.020 (5)   | 0.005 (4)   | 0.016 (4)   | 0.003 (4)   | −0.004 (3)   | −0.001 (3)   |
| C9B | 0.009 (5)   | 0.012 (6)   | 0.019 (5)   | 0.003 (4)   | −0.004 (4)   | −0.003 (4)   |
| S1  | 0.0144 (6)  | 0.0329 (7)  | 0.0213 (6)  | 0.0021 (5)  | −0.0034 (5)  | −0.0067 (6)  |
| S2  | 0.0158 (6)  | 0.0261 (6)  | 0.0215 (6)  | −0.0002 (5) | −0.0008 (5)  | −0.0105 (5)  |
| N1  | 0.017 (2)   | 0.021 (2)   | 0.019 (2)   | −0.0026 (17) | −0.0031 (17) | −0.0042 (18) |
| C1  | 0.016 (2)   | 0.015 (2)   | 0.024 (3)   | −0.0034 (18) | −0.004 (2)   | −0.005 (2)   |
| C2  | 0.017 (2)   | 0.018 (2)   | 0.022 (3)   | −0.0040 (19) | −0.0020 (19) | −0.008 (2)   |
| C3  | 0.026 (3)   | 0.026 (3)   | 0.022 (3)   | −0.005 (2)  | −0.002 (2)   | −0.012 (2)   |
| C4  | 0.031 (3)   | 0.024 (3)   | 0.025 (3)   | −0.009 (2)  | −0.005 (2)   | −0.005 (2)   |
| C5  | 0.025 (3)   | 0.021 (3)   | 0.034 (3)   | 0.000 (2)   | −0.012 (2)   | −0.006 (2)   |
| C6  | 0.019 (2)   | 0.020 (2)   | 0.029 (3)   | −0.002 (2)  | −0.004 (2)   | −0.008 (2)   |
| C7  | 0.020 (2)   | 0.015 (2)   | 0.018 (2)   | −0.0022 (18) | −0.002 (2)   | −0.0051 (19) |

### Geometric parameters (Å, °)

|     |          |          |          |          |          |          |
|-----|----------|----------|----------|----------|----------|----------|
| I1—C8 | 2.101 (6) | N1—C1   | 1.343 (6) |
| I2—C8 | 2.106 (6) | N1—C7   | 1.396 (7) |
| C8—C8 | 1.329 (11) | C2—C3   | 1.400 (8) |
| I3—C9A | 2.146 (10) | C2—C7   | 1.386 (7) |
| I3—C9B | 2.166 (11) | C3—H3   | 0.9500 |
| I4—C9A | 2.095 (10) | C3—C4   | 1.373 (8) |
| I4—C9B | 2.069 (11) | C4—H4   | 0.9500 |
| C9A—C9A | 1.30 (2) | C4—C5   | 1.392 (8) |
| C9B—C9B | 1.33 (2) | C5—H5   | 0.9500 |
| S1—C1 | 1.682 (5) | C5—C6   | 1.388 (8) |
| S2—C1 | 1.738 (5) | C6—H6   | 0.9500 |
S2—C2 1.750 (5) C6—C7 1.392 (7)  
N1—HN1 0.85 (2)

I1—C8—I2 114.1 (2) C7—C2—S2 109.9 (4)
C8—C8—I1 123.2 (6) C7—C2—C3 121.0 (5)
C8—C8—I2 122.8 (6) C2—C3—H3 121.2
C9A—I3—C9Bii 26.4 (4) C4—C3—C2 117.6 (5)
I4—C9A—I3 114.6 (5) C4—C3—H3 121.2
C9Aii—C9A—I3 120.0 (10) C3—C4—H4 119.4
C9Aii—C9A—I4 125.3 (10) C3—C4—C5 121.3 (6)
I4—C9B—I3ii 115.8 (5) C5—C4—H4 119.4
C9Bii—C9B—I4 127.1 (11) C4—C5—H5 119.2

C1—S2—C2 91.8 (2) C6—C5—C4 121.7 (5)
C1—N1—HN1 122 (5) C6—C5—H5 119.2
C1—N1—C7 115.7 (4) C5—C6—H6 121.5
C7—N1—HN1 122 (5) C5—C6—C7 116.9 (5)
S1—C1—S2 123.1 (3) C7—C6—H6 121.5
N1—C1—S1 126.6 (4) C2—C7—N1 112.4 (4)
N1—C1—S2 110.2 (4) C2—C7—C6 121.5 (5)
C3—C2—S2 129.1 (4) C6—C7—N1 126.1 (5)

S2—C2—C3—C4 −178.0 (4) C3—C2—C7—N1 −178.9 (5)
S2—C2—C7—N1 0.4 (5) C3—C2—C7—C6 −0.3 (8)
S2—C2—C7—C6 179.0 (4) C3—C4—C5—C6 0.2 (9)
C1—S2—C2—C3 177.8 (5) C4—C5—C6—C7 0.7 (8)
C1—S2—C2—C7 −1.4 (4) C5—C6—C7—N1 177.7 (5)
C1—N1—C7—C2 1.3 (6) C5—C6—C7—C2 −0.7 (8)
C1—N1—C7—C6 −177.3 (5) C7—N1—C1—S1 176.2 (4)
C2—S2—C1—S1 −176.4 (3) C7—N1—C1—S2 −2.3 (6)
C2—S2—C1—N1 2.1 (4) C7—C2—C3—C4 1.2 (8)
C2—C3—C4—C5 −1.1 (8)

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

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|------|-------|--------|
| N1—HN1···S1iii | 0.85 (2) | 2.43 (2) | 3.275 (5) | 170 (6) |

Symmetry code: (iii) −x, −y+1, −z+2.