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Structural study of bioisosteric derivatives of 5-(1*H*-indol-3-yl)-benzotriazole and their ability to form chalcogen bonds

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Recently, interest in the isosteric replacement of a nitrogen atom to selenium, sulfur or oxygen atoms has been highlighted in the design of potential inhibitors for cancer research. In this context, the structures of 5-(1*H*-indol-3-yl)-2,1,3-benzotriazole derivatives [5-(1*H*-indol-3-yl)-2,1,3-benzothiadiazole (bS, C₁₄H₉N₃S) and 5-(1*H*-indol-3-yl)-2,1,3-benzoxadiazole (bO, C₁₄H₉N₃O)], as well as a synthesis intermediate of the selenated bioisostere [5-[1-(benzenesulfonyl)-1*H*-indol-3-yl]-2,1,3-benzoselenadiazole (p-bSe, C₂₀H₁₃N₃O₂SSe)] were determined using single-crystal X-ray diffraction (SCXRD) analyses. Despite being analogues, different crystal packing, torsion angles and supramolecular features were observed, depending on the substitution of the central atoms of the benzotriazole. In particular, chalcogen interactions were described in the case of p-bSe and not in the bS and bO derivatives. An investigation by *ab initio* computational methods was therefore conducted to understand the effect of the substitution on the ability to form chalcogen bonds and the flexibility of the compounds.

1. Chemical context

Isosteric replacement is a common strategy in drug design to modulate the physicochemical properties of potential inhibitors. In 2021, Kozlova and co-workers (Kozlova *et al.*, 2021a) highlighted a series of bioisosteric derivatives acting as potential new inhibitors of the protein hTDO2, a therapeutic target in cancer research. These new molecules differ in the replacement of the central atom of benzotriazole by an oxygen, sulfur or selenium atom (Fig. 1). At this time, these inhibitors have not yet been crystallized or structurally char-

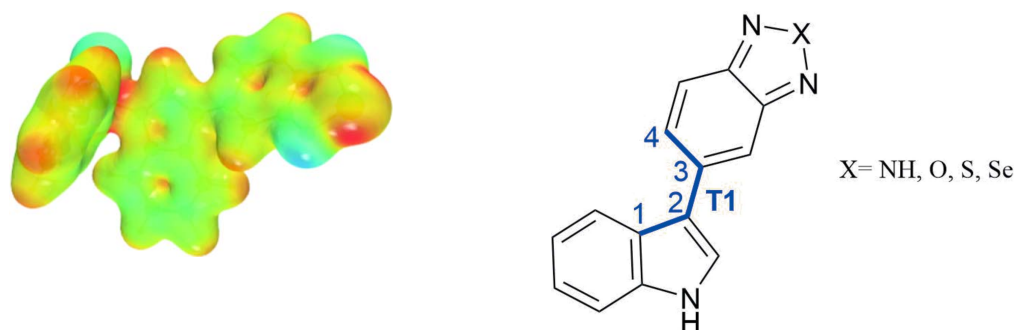
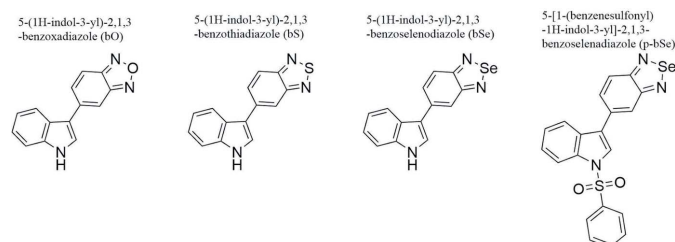


Figure 1
Structures of bioisosteres of 5-(1*H*-indol-3-yl)-benzotriazole with their torsion angle. X = NH, O, S, Se.

acterized. In this context, the present work provides a structural characterization of the inhibitors described by Kozlova *et al.* (2021*b*) completed by *ab initio* calculations for their conformational characterization.

The contribution of an oxygen, a sulfur or a selenium atom instead of a nitrogen affects the ability of these inhibitors to participate in the formation of chalcogen bonds (Vogel *et al.*, 2019). In particular, in these compounds, oxygen, sulfur and selenium atoms could act as chalcogen-bond donors. In recent years, the importance of chalcogen bonds in the stability and folding of proteins as well as their interaction with ligands has been highlighted by numerous investigations (Newberry & Raines, 2019; Kříž *et al.*, 2018; Iwaoka *et al.*, 2001; Iwaoka & Babe, 2015; Burling & Goldstein, 1992). In this article, the potential ability of the compounds to interact with aromatic groups, by chalcogen- π interactions (Aakeroy *et al.*, 2019), was revealed by the crystallization of 5-[1-(benzenesulfonyl)-1*H*-indol-3-yl] -2,1,3-benzoselenadiazole. Therefore, the effect of bioisosteric replacement on the ability to form chalcogen bonds has been studied by *ab initio* calculated electrostatic potential maps. This interesting series could be the starting point for the study of the effect of chalcogen interaction on protein stability and affinity.



2. Structural commentary

The compounds investigated in this study were kindly provided by the team of Raphaël Frédéric (UCLouvain, Belgium). Crystallization assays were performed, by slow evaporation at room temperature (293–298 K), in four different solvents [tetrahydrofuran (THF), chloroform, dichloromethane and *N,N*-dimethylformamide (DMF)]. Crystals of 5-(1*H*-indol-3-yl)-2,1,3-benzoxadiazole (bO) and of 5-(1*H*-indol-3-yl)-2,1,3-benzothiadiazole (bS) were obtained

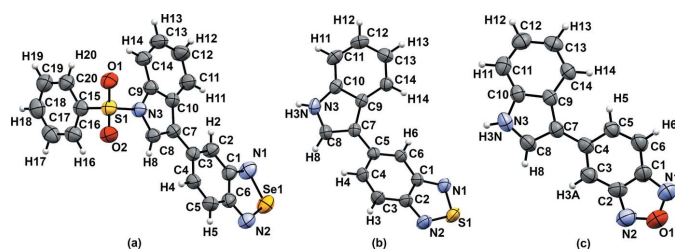


Figure 2

Ellipsoid plots with atom labeling for (a) p-bSe (b) bS and (c) bO. Displacement ellipsoids are drawn at the 50% probability level.

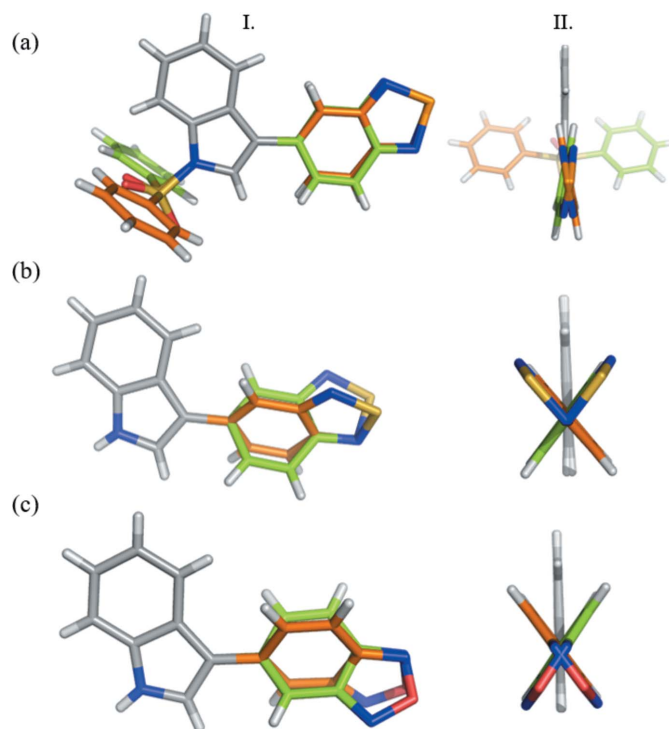


Figure 3

Two perpendicular views (I. and II.) of the two mirror images observed in the crystal packings for (a) p-bSe (b) bS and (c) bO. Superposition of the mirror images with respect to the indole group. Torsion angles are ± 168.3 (2) $^\circ$ for p-bSe, ± 36.9 (2) $^\circ$ for bS and ± 146.7 (2) $^\circ$ for bO.

from chloroform. Despite numerous attempts, we were not able to crystallize the compound 5-(1*H*-indol-3-yl)-2,1,3-benzoselenadiazole (bSe). However, crystals of a synthesis intermediate – 5-[1-(benzenesulfonyl)-1*H*-indol-3-yl]-2,1,3-benzoselenadiazole (p-bSe) – were obtained in THF.

5-[1-(Benzenesulfonyl)-1*H*-indol-3-yl]-2,1,3-benzoselenadiazole (p-bSe) crystallized in space group $P\bar{1}$ with one molecule of p-bSe in the asymmetric unit [Fig. 2(a)]. Interestingly, the molecule adopts an almost planar dihedral angle [-168.3 (2) $^\circ$] between the indole and benzoselenadiazole ring (Fig. 3). 5-(1*H*-indol-3-yl)-2,1,3-benzothiadiazole (bS) and 5-(1*H*-indol-3-yl)-2,1,3-benzoxadiazole (bO) crystallized in space group $Pbca$ [Fig. 2(b) and (c)]. The asymmetric units contain one molecule of bS or bO without disorder. In the three structures, two mirror images are observed in the crystal packing with a torsion angle of ± 168.3 (2) $^\circ$ for p-bSe, ± 36.9 (2) $^\circ$ for bS and ± 146.7 (2) $^\circ$ for bO between the two aromatic parts of the molecules (Fig. 3). The isosteric replacement does not change significantly the planarity of the benzotriazole ring (r.m.s. deviation from planarity: 0.013 Å for p-bSe, 0.006 Å for bS and bO) or the indole ring (0.010 Å for p-bSe, 0.025 Å for bS and 0.011 Å for bO).

3. Supramolecular features

In the structure of p-bSe, a synthesis intermediate of the selenated bioisostere of 5-(1*H*-indol-3-yl)benzotriazole, the benzenesulfonyl contributes to the stabilization of the crystal

Table 1

Hydrogen-bond geometry (Å, °) for p-bSe.

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--------------------|-------|-------------|-------------|---------------|
| $C8-H8\cdots O2^i$ | 0.93 | 2.46 | 3.380 (3) | 168 |
| $C14-H14\cdots O1$ | 0.93 | 2.54 | 3.099 (4) | 119 |

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

packing through weak hydrogen bonds [Table 1, Fig. 4(a)] and chalcogen- π interactions. π -stacking interactions are observed between the selenadiazole and indole groups [centroid (Se/N1/C1/C6/N2) \cdots centroid (N3/C7-C10) distance of 3.732 (2) Å, perpendicular distance of 3.587 (1) Å and horizontal displacement of 1.506 Å, Fig. 4(b)]. A second π -stacking interaction is observed between the selenadiazole group (Se/N1/C1/C6/N2) and the indole group (C9-C14) [centroid \cdots centroid distance of 3.915 (2) Å, perpendicular distance of 3.646 (1) Å and horizontal displacement of 1.331 Å, Fig. 4(b)]. A chalcogen- π interaction between Se and the benzensulfonyl group (C15-C20) is also involved in crystal-packing stabilization [Se \cdots centroid distance of 3.388 (1) Å and N2-Se \cdots centroid angle of 159.83 (8)°, Fig. 4(b)]. The presence of the protecting group (benzenesulfonyl) could explain the crystallization of the p-bSe compound with respect to the bSe compound. Indeed, in

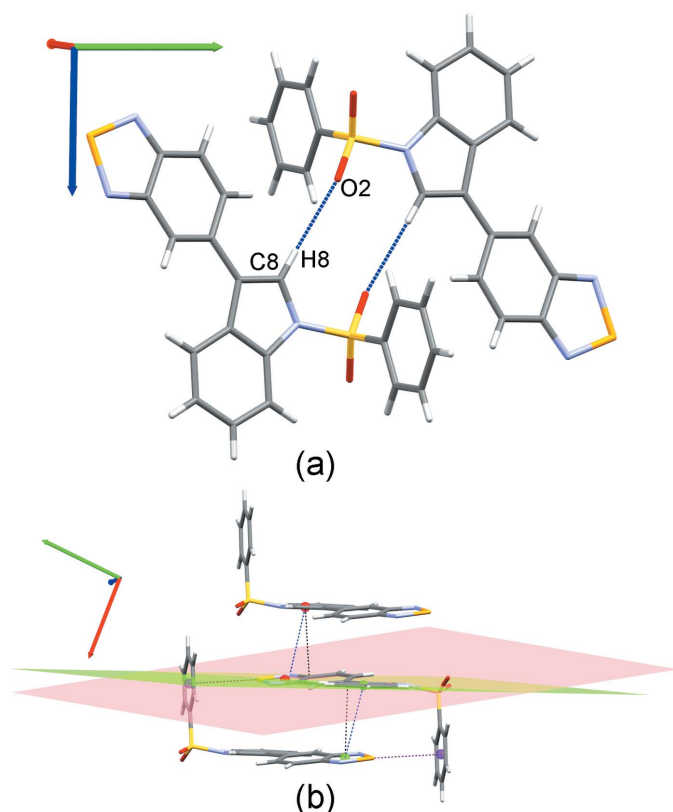


Figure 4
Supramolecular organization of p-bSe: (a) hydrogen-bond interactions; (b) π -stacking interaction between the benzoselenadiazole group and the indole groups (centroids in red: Se/N1/C1/C6/N2 and N3/C7-C10 and centroids in green: Se/N1/C1/C6/N2 and C9-C14) as well as chalcogen- π interactions (in purple).

p-bSe, the orientation of the protecting group is ideal for allowing a chalcogen- π interaction whereas this type of interactions would be more difficult to set up in bSe.

In the structure of compound bS, π -interactions stabilize the crystal packing. π -stacking is observed between benzothiadiazole groups [centroid (C1-C6) \cdots centroid (S1/N1/C1/C2/N2) distance of 3.689 (1) Å, perpendicular distance of 3.4989 (7) Å and horizontal displacement of 1.326 Å, Fig. 5(a)]. An N-H $\cdots \pi$ interaction is also observed between indole groups [N3 \cdots centroid (C9-C14) distance of 3.345 (2) Å, H3N \cdots centroid distance of 2.57 (2) Å and N3-H3N \cdots centroid angle of 169 (2)°, Fig. 5(b)]. In this structure, no chalcogen interaction involving the sulfur atom is observed.

The crystal packing of bO is stabilized through π -interactions. π -stacking is observed between benzoxadiazole groups [centroid (O1/N1/C1-C6/N2) \cdots centroid (O1/N1/C1-C6/N2) distance of 3.893 (1) Å, perpendicular distance of 3.5469 (8) Å and horizontal displacement of 1.570 Å, Fig. 6(a)]. An N-H $\cdots \pi$ interaction is also observed between indole groups [N3 \cdots centroid (C9-C14) distance of 3.226 (2) Å, H3N \cdots centroid distance of 2.57 (2) Å and N3-H3N \cdots centroid angle of 138 (2)°, Fig. 6(b)]. No chalcogen interaction is observed.

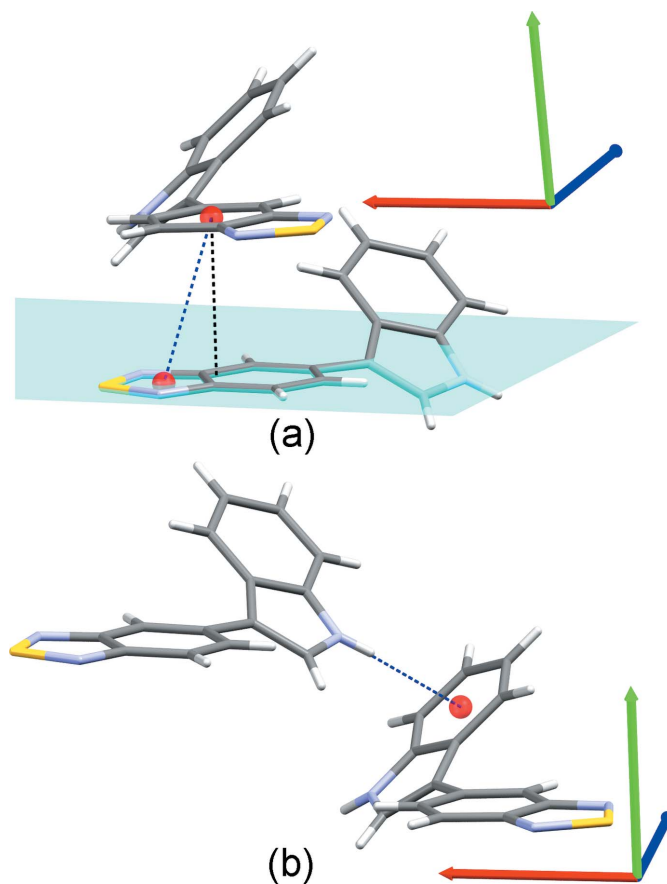


Figure 5
Supramolecular organization of bS: (a) π -stacking interaction between the benzothiadiazole groups and (b) N-H $\cdots \pi$ interaction between two indole groups.

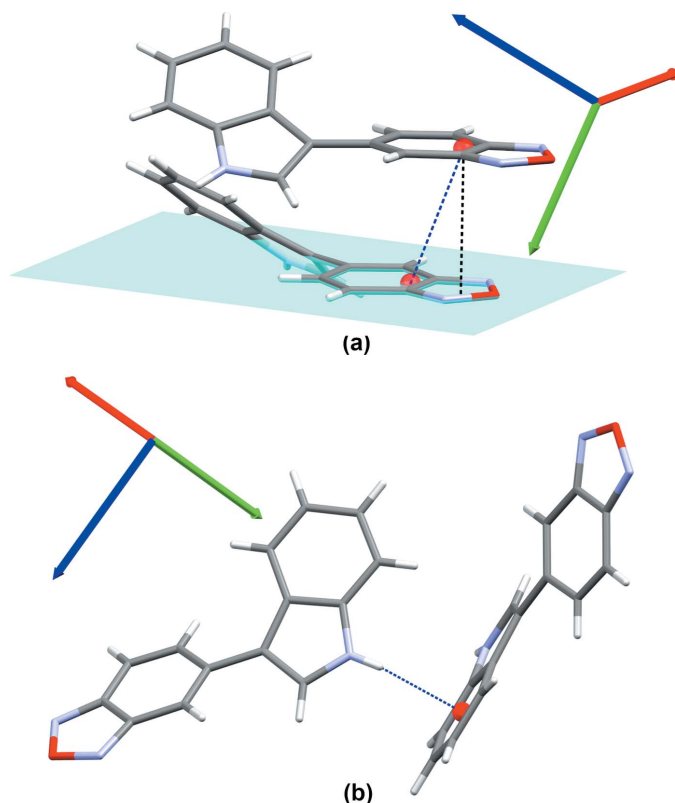


Figure 6
Supramolecular organization of bO. (a) π -stacking interaction between the benzoxadiazole groups and (b) $N-H\cdots\pi$ interaction between two indole groups

4. Quantum *ab initio* studies of the bioisosteric substitution effect

As mentioned previously, the different derivatives vary mainly in their ability to interact through chalcogen bonds. In order to

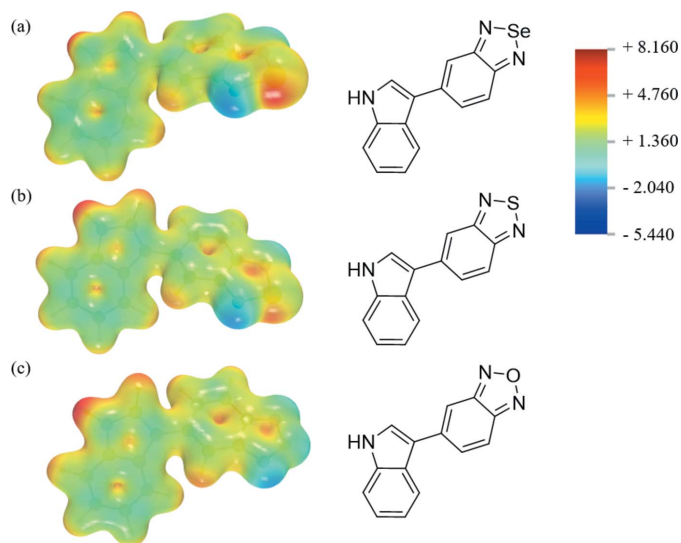


Figure 7
Computed electrostatic potential (EPS) surfaces and associated molecular structures (a) bSe (b) bS (c) bO. The EPS color scale ranges from +8.160 volt to -5.440 volt.

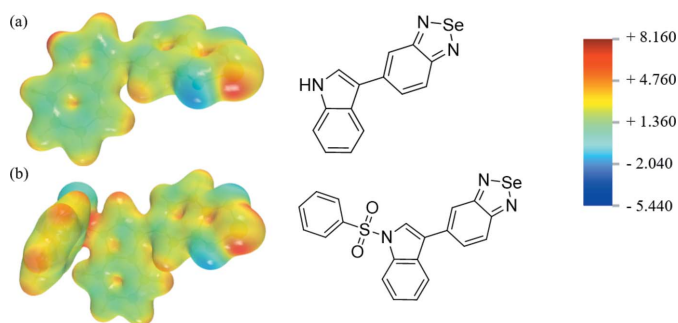


Figure 8
Computed electrostatic potential (EPS) surfaces and associated molecular structures (a) bSe (b) p-bSe. The EPS color scale ranges from +8.160 volts to -5.440 volts.

characterize these differences in depth, quantum mechanics studies have been conducted. First, the presence of a σ -hole in the electron density was studied by means of electrostatic maps. Analysis indicates that the oxygen in benzoxadiazole [Fig. 7(c)] has a weakly positive environment. The σ -hole formation is enhanced by substitution of the central atom with sulfur [Fig. 7(b)] and selenium [Fig. 7(a)], with selenium having the most positive environment. The bioisosteric series thus has different characteristics in terms of the ability to form chalcogen bonds, with the selenium compound being the best chalcogen-bond donor in this bioisosteric series of molecules. These results may explain why chalcogen bonds are observed only in the supramolecular organization of the p-bSe molecule. The donor character of the selenium atom is not affected by the protected group [Fig. 8(a) and (b)]. The difficulty in crystallizing bSe (while p-bSe crystallized readily in THF) could be explained by the absence of the protecting group (benzenesulfonyl) in bSe. Indeed, the benzenesulfonyl group in p-bSe is electron-rich and acts as a well-oriented chalcogen-bond acceptor in p-bSe.

Secondly, in order to determine the effect of the substitution on the flexibility of the derivatives, conformational scans were performed around the torsion angle formed between the indole ring and the benzodiazole part ($T1$). As presented in Fig. 9, bO and bS are characterized by a very similar ΔE energy profile associated with the rotation around $T1$. For all three molecules (bO, bS and bSe), four minima are observed for each molecule, with symmetry on each side of the planar molecule. The energy transitions are low (maximum 15 kJ mol^{-1}) and the molecules are flexible. Moreover, the $T1$ torsion angles observed in the crystal structures of bS

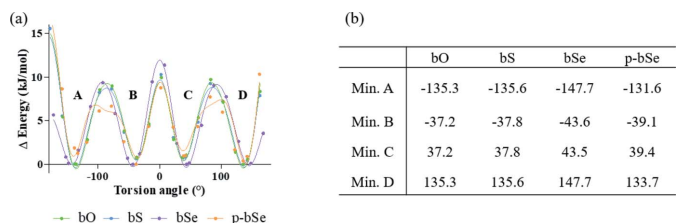


Figure 9
Conformational scans and associated torsion angles calculated with *Gaussian16a* (ω B97XD, 6-31+G*).

Table 2
Chalcogen bonds (Å, °) observed in benzoselenadiazole fragments in the CSD.

| CCDC refcode | Se label | Atoms of the π system | Se...centroid distance | N—Se...centroid angle |
|---------------------|----------|---------------------------|------------------------|-----------------------|
| QIBQUQ ^a | Se1 | C18–C23 | 3.3676 (7) | 147.08 (4) |
| QIBQUQ ^a | Se1 | C12–C17 | 3.0597 (8) | 172.30 (5) |
| VOPMEV ^b | Se1 | C5/C7/C10/C5B/C7B/C10B | 3.8142 (3) | 163.56 (4) |
| VOPNAS ^c | Se1 | C1–C5/C10 | 3.802 (2) | 162.5 (1) |
| VOPNAS ^c | Se2 | C1–C5/C10 | 3.654 (2) | 166.5 (1) |
| YIWLOG ^d | Se5 | C10–C15 | 4.032 (3) | 144.6 (2) |
| YIWLOG ^d | Se6 | C34–C39 | 4.232 (4) | 164.3 (4) |

Notes: (a) Lee *et al.* (2018); (b) Lee *et al.*, 2019); (c) Lee *et al.*, 2019); (d) Tan *et al.* (2008).

[± 36.9 (2)°] and bO [± 146.7 (2)°] are consistent with the energy minima determined by *ab initio* calculations with a relative deviation lower than 10%. Although the bioisosteric character of the flexibility is retained, two differences are observed between the bSe molecule and the bS and bO molecules. The first one is the energy at a torsion angle of 180°, which is lower for the molecule of bSe while it is slightly higher at 0°. The second one is a small shift observed between the angle associated with the energy minima of the bSe molecule and the bS and bO molecules.

The same calculations were performed for the protected molecule (p-bSe). The energy profile associated with the rotation around T1 in p-bSe is similar to those determined for bS and bO. There is a small shift of the values of the angle corresponding to the energy minimum with respect to bSe. This shift may be due to the protecting group that causes steric hindrance in p-bSe. The energies corresponding to minima A and C are higher than the energies for minima B and D, involving two local minima and a preference in the conformers. The maxima of the energies between A/B and C/D are also lower than in the case of the other compounds, supporting this hypothesis. In the case of p-bSe, the torsion angle observed in the crystallographic structure [± 168.3 (2)°] corresponds to an energy maximum on the energy profile associated with the rotation around T1 determined by *ab initio* calculations. The quasi-planarity of the molecule observed in the crystallographic structure could be encouraged by the formation of chalcogen bonds stabilizing this conformation of p-bSe.

5. Database survey

The Cambridge Structural Database (CSD version 5.42; Groom *et al.*, 2016) was searched with *ConQuest* (version

(a) bSe fragment (b) bS fragment (c) bO fragment

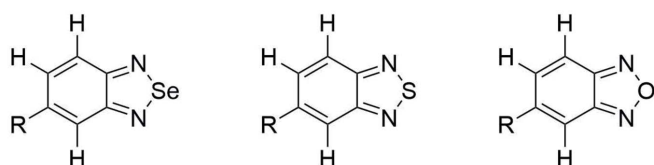


Figure 10
Fragments searched for in the CSD during the database survey analysis: (a) bSe fragment (b) bS fragment (c) bO fragment.

2021.2.0; Bruno *et al.*, 2002) for benzoselenadiazole, benzothiadiazole and benzoxadiazole fragments (Fig. 10). The search for the benzoselenadiazole fragment resulted in 57 hits. Chalcogen bonds are observed in all of these hits. In particular, chalcogen– π interactions are observed in four structures [CSD refcodes: QIBQUQ (Lee *et al.*, 2018), VOPMEV (Lee *et al.*, 2019), VOPNAS (Lee *et al.*, 2019), and YIWLOG (Tan *et al.*, 2008)], listed in Table 2. The search for a benzothiadiazole fragment resulted in 34 hits. Chalcogen bonds but no chalcogen– π interactions are observed in these hits. The search for a benzoxadiazole fragment resulted in 24 hits but no chalcogen interactions are observed with the oxygen atom of benzoxadiazole as a chalcogen donor.

6. Synthesis and crystallization

The synthesis of the various compounds was reported by Kozlova *et al.* (2021a). Crystallization of the 5-(1*H*-indol-3-yl)-benzotriazole derivatives were carried out by the solvent evaporation method. The compounds were dissolved in THF, chloroform, dichloromethane or DMF until complete dissolution. Slow evaporation of the solvent at room temperature (293–297 K) yielded colorless crystals that were then picked for XRD analysis. Crystals of 5-(1*H*-indol-3-yl)-2,1,3-benzothiadiazole and 5-(1*H*-indol-3-yl)-2,1,3-benzoxadiazole were obtained from chloroform while the protected benzoselenadiazole (5-[1-(benzenesulfonyl)-1*H*-indol-3-yl]-2,1,3-benzoselenadiazole) was crystallized in THF.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. In all of the structures, hydrogen atoms were placed in calculated positions and refined using a riding model [C–H bond length of 0.93 Å, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$]. In the structure of 5-(1*H*-indol-3-yl)-2,1,3-benzothiadiazole, bS, the hydrogen on the nitrogen atom in the indole group was refined without constraint and the refined N–H distance is 0.78 (2) Å.

8. Quantum *ab initio* methodology

All the molecules investigated in the study (bS, bO, bSe, p-bSe) were optimized starting from the crystal coordinates using the density functional method (DFT) with the exchange-

Table 3
Experimental details.

| | p-bSe | bS | bO |
|---|---|--|--|
| Crystal data | | | |
| Chemical formula | C ₂₀ H ₁₃ N ₃ O ₂ SSe | C ₁₄ H ₉ N ₃ S | C ₁₄ H ₉ N ₃ O |
| <i>M_r</i> | 438.35 | 251.30 | 235.24 |
| Crystal system, space group | Triclinic, <i>P</i> $\bar{1}$ | Orthorhombic, <i>Pbca</i> | Orthorhombic, <i>Pbca</i> |
| Temperature (K) | 295 | 295 | 295 |
| <i>a</i> , <i>b</i> , <i>c</i> (Å) | 7.7760 (3), 9.9573 (4), 11.4124 (6) | 7.5884 (1), 7.1060 (1), 43.2464 (7) | 12.0256 (7), 7.7396 (5), 23.8551 (16) |
| α , β , γ (°) | 90.970 (4), 92.771 (4), 94.283 (3) | 90, 90, 90 | 90, 90, 90 |
| <i>V</i> (Å ³) | 879.95 (7) | 2331.98 (6) | 2220.3 (2) |
| <i>Z</i> | 2 | 8 | 8 |
| Radiation type | Cu <i>K</i> α | Cu <i>K</i> α | Cu <i>K</i> α |
| μ (mm ⁻¹) | 4.18 | 2.32 | 0.75 |
| Crystal size (mm) | 0.19 × 0.10 × 0.01 | 0.29 × 0.18 × 0.04 | 0.12 × 0.09 × 0.03 |
| Data collection | | | |
| Diffractometer | Xcalibur, Ruby, Gemini ultra | Xcalibur, Ruby, Gemini ultra R | Xcalibur, Ruby, Gemini ultra |
| Absorption correction | Analytical (<i>CrysAlis PRO</i> ; Rigaku OD, 2020) | Analytical (<i>CrysAlis PRO</i> ; Rigaku OD, 2020) | Analytical (<i>CrysAlis PRO</i> ; Rigaku OD, 2020) |
| <i>T</i> _{min} – <i>T</i> _{max} | 0.663, 0.958 | 0.663, 0.920 | 0.941, 0.981 |
| No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections | 9941, 3113, 2571 | 10906, 2072, 1825 | 6883, 1972, 1275 |
| <i>R</i> _{int} | 0.030 | 0.025 | 0.047 |
| (<i>sin</i> θ / λ) _{max} (Å ⁻¹) | 0.598 | 0.598 | 0.597 |
| Refinement | | | |
| <i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i> | 0.037, 0.101, 1.04 | 0.035, 0.098, 1.07 | 0.050, 0.134, 1.06 |
| No. of reflections | 3113 | 2072 | 1972 |
| No. of parameters | 244 | 167 | 166 |
| H-atom treatment | H-atom parameters constrained | H atoms treated by a mixture of independent and constrained refinement | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³) | 0.46, -0.61 | 0.20, -0.30 | 0.14, -0.16 |

Computer programs: *CrysAlis PRO* (Rigaku OD, 2020), *SHELXT2014/5* (Sheldrick, 2015a), *SHELXL2016/6* (Sheldrick, 2015b), *ShelXle* (Hübschle *et al.*, 2011), *OLEX2* (Dolomanov *et al.*, 2009), and *Mercury* (Macrae *et al.*, 2020).

correlation functional ω B97XD and the 6-31+G* basis set. Because we were not able to crystallize the bSe compound, this molecule was created by substitution of the sulfur atom for a selenium atom from the coordinates of the bS molecule. The optimizations were performed with *Gaussian16a* (Frisch *et al.*, 2016) in the gas phase. The electrostatic potential was calculated from the SCF-type density and was sliced by making 80 cubic points evenly distributed on a rectangular grid automatically generated by *Gaussian16a*. The resulting maps were visualized using *DrawMol* (Liegeois, 2021). For the conformational scans, the optimized structures were analyzed using relaxed scans around the torsion angle (*T1*) formed between the indole ring and the benzodiazole part from 0 to 360° by steps of 20°. The resulting conformations close to an energy minimum were extracted and refined by a new optimization at the same level of approximation. The preparation of the input files, as well as the visualization of the results was performed with the *DrawMol* and *DrawSpectrum* suite of programs (Liegeois, 2021). The graphs were drawn with the program *Prism* from *GraphPad* (one-way ANOVA followed by Dunnett's multiple comparisons test, *Prism* version 8.0.0 for Windows; GraphPad, 2021).

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References

- Aakeroy, C. B., Bryce, D. L., Desiraju, G. R., Frontera, A., Legon, A. C., Nicotra, F., Rissanen, K., Scheiner, S., Terraneo, G., Metrangolo, P. & Resnati, G. (2019). *Pure Appl. Chem.* **91**, 1889–1892.
- Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, M., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* **B58**, 389–397.
- Burling, F. T. & Goldstein, B. M. (1992). *J. Am. Chem. Soc.* **114**, 2313–2320.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Frisch, M. J., Trucks, G. W., Schlegel, H. B., Scuseria, G. E., Robb, M. A., Cheeseman, J. R., Scalmani, G., Barone, V., Petersson, G. A.,

- Nakatsuji, H., Li, X., Caricato, M., Marenich, A. V., Bloino, J., Janesko, B. G., Gomperts, R., Mennucci, B., Hratchian, H. P., Ortiz, J. V., Izmaylov, A. F., Sonnenberg, J. L., Williams-Young, D., Ding, F., Lipparini, F., Egidi, F., Goings, J., Peng, B., Petrone, A., Henderson, T., Ranasinghe, D., Zakrzewski, V. G., Gao, J., Rega, N., Zheng, G., Liang, W., Hada, M., Ehara, M., Toyota, K., Fukuda, R., Hasegawa, J., Ishida, M., Nakajima, T., Honda, Y., Kitao, O., Nakai, H., Vreven, T., Throssell, K., Montgomery, J. A. Jr, Peralta, J. E., Ogliaro, F., Bearpark, M. J., Heyd, J. J., Brothers, E. N., Kudin, K. N., Staroverov, V. N., Keith, T. A., Kobayashi, R., Normand, J., Raghavachari, K., Rendell, A. P., Burant, J. C., Iyengar, S. S., Tomasi, J., Cossi, M., Millam, J. M., Klene, M., Adamo, C., Cammi, R., Ochterski, J. W., Martin, R. L., Morokuma, K., Farkas, O., Foresman, J. B. & Fox, D. J. (2016). *Gaussian16a*. Gaussian, Inc., Wallingford, CT, USA.
- GraphPad (2021). *GraphPad*. GraphPad Software, San Diego, California USA, www.graphpad.com
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Hübschle, C. B., Sheldrick, G. M. & Dittrich, B. (2011). *J. Appl. Cryst.* **44**, 1281–1284.
- Iwaoka, M. & Babe, N. (2015). *Phosphorus Sulfur Silicon*, **190**, 1257–1264.
- Iwaoka, M., Takemoto, S., Okada, M. & Tomoda, S. (2001). *Chem. Lett.* **30**, 132–133.
- Kozlova, A., Thabault, L., Dauguet, N., Deskeuvre, M., Stroobant, V., Pilotte, L. & Frédérick, R. (2021a). *Eur. J. Med. Chem.* **227**, 113892. <https://doi.org/10.1016/j.ejmech.2021.113892>
- Kozlova, A., Thabault, L., Liberelle, M., Klaessens, S., Prévost, J. R., Mathieu, C., Pilotte, L., Stroobant, V., Van den Eynde, B. & Frédérick, R. (2021b). *J. Med. Chem.* **64**, 10967–10980.
- Kříž, K., Fanfrlík, J. & Lepšík, M. (2018). *ChemPhysChem*, **19**, 2540–2548.
- Lee, J., Lee, L. M., Arnott, Z., Jenkins, H., Britten, J. F. & Vargas-Baca, I. (2018). *New J. Chem.* **42**, 10555–10562.
- Lee, L. M., Corless, V., Luu, H., He, A., Jenkins, H., Britten, J. F., Adam Pani, F. & Vargas-Baca, I. (2019). *Dalton Trans.* **48**, 12541–12548.
- Liegeois, V. (2021). *DrawMol*. University of Namur, Belgium. www.unamur.be/drawmol
- Macrae, C. F., Sovago, I., Cottrell, S. J., Galek, P. T. A., McCabe, P., Pidcock, E., Platings, M., Shields, G. P., Stevens, J. S., Towler, M. & Wood, P. A. (2020). *J. Appl. Cryst.* **53**, 226–235.
- Newberry, R. W. & Raines, R. T. (2019). *Chem. Biol.* **14**, 1677–1686.
- Rigaku OD (2020). *CrysAlis PRO*. Rigaku Oxford Diffraction Ltd, Yarnton, England.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Tan, C. K., Wang, J., Leng, J. D., Zheng, L. L. & Tong, M. L. (2008). *Eur. J. Inorg. Chem.* pp. 771–778.
- Vogel, L., Wonner, P. & Huber, S. M. (2019). *Angew. Chem. Int. Ed.* **58**, 1880–1891.

supporting information

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Structural study of bioisosteric derivatives of 5-(1*H*-indol-3-yl)-benzotriazole and their ability to form chalcogen bonds

Manon Mirgaux, Tanguy Scaillet, Arina Kozlova, Nikolay Tumanov, Raphaël Frederick, Laurie Bodart and Johan Wouters

Computing details

For all structures, data collection: *CrysAlis PRO* (Rigaku OD, 2020); cell refinement: *CrysAlis PRO* (Rigaku OD, 2020); data reduction: *CrysAlis PRO* (Rigaku OD, 2020); program(s) used to solve structure: *SHELXT2014/5* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2016/6* (Sheldrick, 2015b), *ShelXle* (Hübschle *et al.*, 2011), *OLEX2* (Dolomanov *et al.*, 2009); molecular graphics: *Mercury* (Macrae *et al.*, 2020).

5-[1-(Benzenesulfonyl)-1*H*-indol-3-yl]-2,1,3-benzoselenadiazole (p-bSe)

Crystal data

C₂₀H₁₃N₃O₂SSe

M_r = 438.35

Triclinic, *P*1̄

a = 7.7760 (3) Å

b = 9.9573 (4) Å

c = 11.4124 (6) Å

α = 90.970 (4)°

β = 92.771 (4)°

γ = 94.283 (3)°

V = 879.95 (7) Å³

Z = 2

F(000) = 440

D_x = 1.654 Mg m⁻³

Cu *Kα* radiation, *λ* = 1.54184 Å

Cell parameters from 4183 reflections

θ = 3.9–66.7°

μ = 4.18 mm⁻¹

T = 295 K

Plate, colourless

0.19 × 0.10 × 0.01 mm

Data collection

Xcalibur, Ruby, Gemini ultra
diffractometer

Radiation source: fine-focus sealed X-ray tube,
Enhance Ultra (Cu) X-ray Source

Detector resolution: 5.1856 pixels mm⁻¹

ω scans

Absorption correction: analytical
(*CrysAlisPro*; Rigaku OD, 2020)

T_{min} = 0.663, *T_{max}* = 0.958

9941 measured reflections

3113 independent reflections

2571 reflections with *I* > 2σ(*I*)

R_{int} = 0.030

θ_{max} = 67.2°, *θ_{min}* = 3.9°

h = −9→9

k = −11→9

l = −13→13

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.037

wR(*F*²) = 0.101

S = 1.04

3113 reflections

244 parameters

0 restraints

Primary atom site location: dual

Secondary atom site location: dual

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0588P)^2 + 0.2071P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.46 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.61 \text{ e } \text{\AA}^{-3}$

Special details

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

Refinement. Structures were solved by the dual-space method of ShelXT (Sheldrick, 2015a) within Olex² (Dolomanov *et al.*, 2009). Structures were refined by the least-squares method implemented in SHELXL (Sheldrick, 2015b) within ShelXle (Hübschle *et al.*, 2011). Structures and crystal packings were visualized using Mercury (Macrae *et al.*, 2020).

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

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|-------------|--------------|----------------------------------|
| Se1 | 0.90079 (4) | 0.81216 (3) | 0.75959 (3) | 0.07181 (16) |
| S1 | 0.52981 (9) | 0.00138 (6) | 0.25117 (6) | 0.05354 (19) |
| O1 | 0.4385 (3) | -0.0082 (2) | 0.13980 (18) | 0.0639 (5) |
| O2 | 0.4503 (3) | -0.0486 (2) | 0.35371 (18) | 0.0650 (5) |
| N1 | 0.9080 (3) | 0.7558 (3) | 0.6120 (2) | 0.0674 (7) |
| N2 | 0.7790 (4) | 0.6657 (3) | 0.8062 (2) | 0.0685 (7) |
| N3 | 0.5808 (3) | 0.1632 (2) | 0.2811 (2) | 0.0539 (5) |
| C1 | 0.8223 (3) | 0.6341 (3) | 0.6050 (2) | 0.0529 (6) |
| C2 | 0.7984 (4) | 0.5541 (3) | 0.5005 (2) | 0.0558 (6) |
| H2 | 0.846444 | 0.585254 | 0.432073 | 0.067* |
| C3 | 0.7060 (3) | 0.4321 (2) | 0.4993 (2) | 0.0481 (6) |
| C4 | 0.6317 (4) | 0.3868 (3) | 0.6070 (2) | 0.0570 (7) |
| H4 | 0.566610 | 0.304496 | 0.606199 | 0.068* |
| C5 | 0.6530 (4) | 0.4592 (3) | 0.7089 (3) | 0.0620 (7) |
| H5 | 0.604349 | 0.426314 | 0.776555 | 0.074* |
| C6 | 0.7506 (4) | 0.5862 (3) | 0.7115 (2) | 0.0553 (6) |
| C7 | 0.6741 (3) | 0.3458 (2) | 0.3939 (2) | 0.0477 (6) |
| C8 | 0.6014 (3) | 0.2176 (3) | 0.3945 (2) | 0.0519 (6) |
| H8 | 0.569709 | 0.172160 | 0.461603 | 0.062* |
| C9 | 0.6494 (3) | 0.2603 (3) | 0.2037 (2) | 0.0517 (6) |
| C10 | 0.7064 (3) | 0.3750 (3) | 0.2714 (2) | 0.0503 (6) |
| C11 | 0.7763 (5) | 0.4873 (3) | 0.2134 (3) | 0.0675 (8) |
| H11 | 0.814269 | 0.565849 | 0.255173 | 0.081* |
| C12 | 0.7881 (5) | 0.4801 (3) | 0.0936 (3) | 0.0802 (10) |
| H12 | 0.834370 | 0.554638 | 0.054712 | 0.096* |
| C13 | 0.7326 (5) | 0.3643 (3) | 0.0297 (3) | 0.0730 (9) |
| H13 | 0.744430 | 0.362173 | -0.050976 | 0.088* |
| C14 | 0.6602 (4) | 0.2520 (3) | 0.0831 (2) | 0.0616 (7) |
| H14 | 0.620702 | 0.174545 | 0.040267 | 0.074* |
| C15 | 0.7292 (4) | -0.0687 (2) | 0.2387 (2) | 0.0545 (6) |
| C16 | 0.8268 (4) | -0.0925 (3) | 0.3403 (3) | 0.0637 (7) |
| H16 | 0.787011 | -0.072995 | 0.413787 | 0.076* |
| C17 | 0.9851 (4) | -0.1459 (3) | 0.3296 (3) | 0.0732 (9) |

| | | | | |
|-----|------------|-------------|------------|------------|
| H17 | 1.052470 | -0.162969 | 0.396443 | 0.088* |
| C18 | 1.0426 (4) | -0.1736 (3) | 0.2205 (3) | 0.0733 (9) |
| H18 | 1.149596 | -0.208273 | 0.214087 | 0.088* |
| C19 | 0.9442 (5) | -0.1509 (3) | 0.1210 (3) | 0.0723 (8) |
| H19 | 0.984606 | -0.170801 | 0.047749 | 0.087* |
| C20 | 0.7853 (4) | -0.0986 (3) | 0.1287 (3) | 0.0636 (7) |
| H20 | 0.717547 | -0.083800 | 0.061463 | 0.076* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|---------------|---------------|---------------|
| Se1 | 0.0753 (2) | 0.0682 (2) | 0.0690 (3) | -0.00422 (16) | -0.00371 (17) | -0.02183 (16) |
| S1 | 0.0587 (4) | 0.0467 (4) | 0.0525 (4) | -0.0097 (3) | -0.0021 (3) | -0.0040 (3) |
| O1 | 0.0655 (12) | 0.0654 (12) | 0.0575 (12) | -0.0055 (9) | -0.0107 (9) | -0.0115 (9) |
| O2 | 0.0722 (12) | 0.0598 (11) | 0.0604 (12) | -0.0146 (9) | 0.0078 (10) | 0.0011 (9) |
| N1 | 0.0683 (15) | 0.0635 (15) | 0.0673 (16) | -0.0116 (11) | 0.0024 (12) | -0.0126 (12) |
| N2 | 0.0805 (17) | 0.0695 (16) | 0.0551 (15) | 0.0090 (13) | -0.0007 (13) | -0.0112 (12) |
| N3 | 0.0681 (14) | 0.0440 (11) | 0.0473 (12) | -0.0086 (9) | 0.0006 (10) | -0.0024 (9) |
| C1 | 0.0520 (14) | 0.0522 (14) | 0.0534 (16) | 0.0035 (11) | -0.0050 (12) | -0.0072 (11) |
| C2 | 0.0604 (16) | 0.0571 (15) | 0.0486 (15) | -0.0046 (12) | 0.0043 (12) | -0.0038 (11) |
| C3 | 0.0519 (14) | 0.0454 (13) | 0.0471 (14) | 0.0061 (10) | -0.0019 (11) | -0.0009 (10) |
| C4 | 0.0734 (18) | 0.0461 (14) | 0.0511 (16) | 0.0018 (12) | 0.0011 (13) | 0.0019 (11) |
| C5 | 0.081 (2) | 0.0578 (16) | 0.0477 (16) | 0.0068 (14) | 0.0042 (14) | 0.0048 (12) |
| C6 | 0.0607 (16) | 0.0553 (15) | 0.0501 (15) | 0.0136 (12) | -0.0058 (13) | -0.0059 (12) |
| C7 | 0.0507 (14) | 0.0481 (13) | 0.0440 (14) | 0.0034 (10) | 0.0012 (11) | -0.0017 (10) |
| C8 | 0.0604 (15) | 0.0502 (14) | 0.0443 (14) | -0.0010 (11) | 0.0010 (12) | 0.0005 (11) |
| C9 | 0.0562 (15) | 0.0506 (14) | 0.0477 (15) | 0.0020 (11) | -0.0008 (12) | 0.0019 (11) |
| C10 | 0.0567 (15) | 0.0463 (14) | 0.0483 (15) | 0.0048 (11) | 0.0041 (12) | 0.0004 (11) |
| C11 | 0.096 (2) | 0.0478 (15) | 0.0566 (18) | -0.0091 (14) | 0.0058 (16) | -0.0007 (12) |
| C12 | 0.120 (3) | 0.0613 (18) | 0.0573 (19) | -0.0120 (18) | 0.0133 (18) | 0.0073 (14) |
| C13 | 0.103 (2) | 0.0705 (19) | 0.0446 (16) | -0.0002 (16) | 0.0070 (16) | 0.0032 (13) |
| C14 | 0.0788 (19) | 0.0597 (16) | 0.0450 (15) | 0.0000 (14) | -0.0025 (14) | -0.0026 (12) |
| C15 | 0.0639 (16) | 0.0401 (13) | 0.0568 (16) | -0.0098 (11) | -0.0022 (13) | -0.0009 (11) |
| C16 | 0.0712 (19) | 0.0583 (16) | 0.0591 (17) | -0.0064 (13) | -0.0054 (14) | 0.0028 (13) |
| C17 | 0.072 (2) | 0.0647 (19) | 0.080 (2) | -0.0023 (15) | -0.0186 (17) | 0.0078 (16) |
| C18 | 0.0690 (19) | 0.0574 (18) | 0.093 (3) | 0.0020 (14) | 0.0006 (18) | 0.0007 (16) |
| C19 | 0.082 (2) | 0.0645 (19) | 0.071 (2) | 0.0070 (16) | 0.0059 (17) | -0.0070 (15) |
| C20 | 0.0748 (19) | 0.0565 (16) | 0.0581 (18) | 0.0004 (13) | -0.0013 (15) | -0.0041 (13) |

Geometric parameters (Å, °)

| | | | |
|--------|-----------|---------|-----------|
| Se1—N1 | 1.772 (3) | C8—H8 | 0.9300 |
| Se1—N2 | 1.782 (3) | C9—C14 | 1.384 (4) |
| S1—O1 | 1.424 (2) | C9—C10 | 1.399 (4) |
| S1—O2 | 1.430 (2) | C10—C11 | 1.398 (4) |
| S1—N3 | 1.657 (2) | C11—C12 | 1.376 (5) |
| S1—C15 | 1.758 (3) | C11—H11 | 0.9300 |
| N1—C1 | 1.337 (4) | C12—C13 | 1.383 (5) |

| | | | |
|-----------|-------------|-------------|-----------|
| N2—C6 | 1.329 (4) | C12—H12 | 0.9300 |
| N3—C8 | 1.391 (3) | C13—C14 | 1.379 (4) |
| N3—C9 | 1.414 (4) | C13—H13 | 0.9300 |
| C1—C2 | 1.420 (4) | C14—H14 | 0.9300 |
| C1—C6 | 1.436 (4) | C15—C20 | 1.383 (4) |
| C2—C3 | 1.365 (4) | C15—C16 | 1.388 (4) |
| C2—H2 | 0.9300 | C16—C17 | 1.386 (5) |
| C3—C4 | 1.448 (4) | C16—H16 | 0.9300 |
| C3—C7 | 1.466 (4) | C17—C18 | 1.374 (5) |
| C4—C5 | 1.355 (4) | C17—H17 | 0.9300 |
| C4—H4 | 0.9300 | C18—C19 | 1.370 (5) |
| C5—C6 | 1.424 (4) | C18—H18 | 0.9300 |
| C5—H5 | 0.9300 | C19—C20 | 1.383 (5) |
| C7—C8 | 1.357 (4) | C19—H19 | 0.9300 |
| C7—C10 | 1.463 (4) | C20—H20 | 0.9300 |
| | | | |
| N1—Se1—N2 | 95.07 (11) | C14—C9—C10 | 123.6 (3) |
| O1—S1—O2 | 120.66 (12) | C14—C9—N3 | 129.3 (3) |
| O1—S1—N3 | 107.53 (12) | C10—C9—N3 | 107.1 (2) |
| O2—S1—N3 | 104.65 (12) | C11—C10—C9 | 117.9 (3) |
| O1—S1—C15 | 108.75 (13) | C11—C10—C7 | 134.3 (3) |
| O2—S1—C15 | 109.34 (13) | C9—C10—C7 | 107.8 (2) |
| N3—S1—C15 | 104.72 (12) | C12—C11—C10 | 119.0 (3) |
| C1—N1—Se1 | 106.3 (2) | C12—C11—H11 | 120.5 |
| C6—N2—Se1 | 105.8 (2) | C10—C11—H11 | 120.5 |
| C8—N3—C9 | 108.0 (2) | C11—C12—C13 | 121.5 (3) |
| C8—N3—S1 | 123.64 (18) | C11—C12—H12 | 119.3 |
| C9—N3—S1 | 126.7 (2) | C13—C12—H12 | 119.3 |
| N1—C1—C2 | 124.0 (3) | C14—C13—C12 | 121.4 (3) |
| N1—C1—C6 | 116.0 (3) | C14—C13—H13 | 119.3 |
| C2—C1—C6 | 120.0 (2) | C12—C13—H13 | 119.3 |
| C3—C2—C1 | 120.8 (3) | C13—C14—C9 | 116.6 (3) |
| C3—C2—H2 | 119.6 | C13—C14—H14 | 121.7 |
| C1—C2—H2 | 119.6 | C9—C14—H14 | 121.7 |
| C2—C3—C4 | 118.3 (2) | C20—C15—C16 | 121.6 (3) |
| C2—C3—C7 | 123.5 (2) | C20—C15—S1 | 119.6 (2) |
| C4—C3—C7 | 118.2 (2) | C16—C15—S1 | 118.8 (2) |
| C5—C4—C3 | 122.8 (3) | C17—C16—C15 | 118.4 (3) |
| C5—C4—H4 | 118.6 | C17—C16—H16 | 120.8 |
| C3—C4—H4 | 118.6 | C15—C16—H16 | 120.8 |
| C4—C5—C6 | 119.4 (3) | C18—C17—C16 | 120.2 (3) |
| C4—C5—H5 | 120.3 | C18—C17—H17 | 119.9 |
| C6—C5—H5 | 120.3 | C16—C17—H17 | 119.9 |
| N2—C6—C5 | 124.5 (3) | C19—C18—C17 | 120.8 (3) |
| N2—C6—C1 | 116.8 (3) | C19—C18—H18 | 119.6 |
| C5—C6—C1 | 118.7 (2) | C17—C18—H18 | 119.6 |
| C8—C7—C10 | 106.2 (2) | C18—C19—C20 | 120.4 (3) |
| C8—C7—C3 | 123.9 (2) | C18—C19—H19 | 119.8 |

| | | | |
|--------------|------------|-----------------|------------|
| C10—C7—C3 | 129.8 (2) | C20—C19—H19 | 119.8 |
| C7—C8—N3 | 110.8 (2) | C19—C20—C15 | 118.6 (3) |
| C7—C8—H8 | 124.6 | C19—C20—H20 | 120.7 |
| N3—C8—H8 | 124.6 | C15—C20—H20 | 120.7 |
| | | | |
| N2—Se1—N1—C1 | -0.2 (2) | C8—N3—C9—C14 | 179.1 (3) |
| N1—Se1—N2—C6 | -0.4 (2) | S1—N3—C9—C14 | 13.3 (4) |
| O1—S1—N3—C8 | 151.5 (2) | C8—N3—C9—C10 | -1.8 (3) |
| O2—S1—N3—C8 | 22.0 (3) | S1—N3—C9—C10 | -167.7 (2) |
| C15—S1—N3—C8 | -93.0 (2) | C14—C9—C10—C11 | 0.7 (4) |
| O1—S1—N3—C9 | -44.8 (3) | N3—C9—C10—C11 | -178.4 (3) |
| O2—S1—N3—C9 | -174.2 (2) | C14—C9—C10—C7 | -179.9 (3) |
| C15—S1—N3—C9 | 70.8 (3) | N3—C9—C10—C7 | 1.0 (3) |
| Se1—N1—C1—C2 | -180.0 (2) | C8—C7—C10—C11 | 179.5 (3) |
| Se1—N1—C1—C6 | 0.7 (3) | C3—C7—C10—C11 | 0.8 (5) |
| N1—C1—C2—C3 | -178.5 (3) | C8—C7—C10—C9 | 0.3 (3) |
| C6—C1—C2—C3 | 0.8 (4) | C3—C7—C10—C9 | -178.5 (3) |
| C1—C2—C3—C4 | 0.5 (4) | C9—C10—C11—C12 | -0.8 (5) |
| C1—C2—C3—C7 | 178.9 (2) | C7—C10—C11—C12 | 180.0 (3) |
| C2—C3—C4—C5 | -1.3 (4) | C10—C11—C12—C13 | -0.1 (6) |
| C7—C3—C4—C5 | -179.8 (3) | C11—C12—C13—C14 | 1.2 (6) |
| C3—C4—C5—C6 | 0.7 (4) | C12—C13—C14—C9 | -1.3 (5) |
| Se1—N2—C6—C5 | -178.2 (2) | C10—C9—C14—C13 | 0.3 (5) |
| Se1—N2—C6—C1 | 0.9 (3) | N3—C9—C14—C13 | 179.3 (3) |
| C4—C5—C6—N2 | 179.7 (3) | O1—S1—C15—C20 | 11.5 (3) |
| C4—C5—C6—C1 | 0.6 (4) | O2—S1—C15—C20 | 145.2 (2) |
| N1—C1—C6—N2 | -1.2 (4) | N3—S1—C15—C20 | -103.2 (2) |
| C2—C1—C6—N2 | 179.5 (3) | O1—S1—C15—C16 | -168.6 (2) |
| N1—C1—C6—C5 | 178.0 (3) | O2—S1—C15—C16 | -35.0 (2) |
| C2—C1—C6—C5 | -1.4 (4) | N3—S1—C15—C16 | 76.7 (2) |
| C2—C3—C7—C8 | 171.4 (3) | C20—C15—C16—C17 | 0.8 (4) |
| C4—C3—C7—C8 | -10.2 (4) | S1—C15—C16—C17 | -179.0 (2) |
| C2—C3—C7—C10 | -10.1 (4) | C15—C16—C17—C18 | 0.3 (4) |
| C4—C3—C7—C10 | 168.3 (3) | C16—C17—C18—C19 | -0.9 (5) |
| C10—C7—C8—N3 | -1.4 (3) | C17—C18—C19—C20 | 0.4 (5) |
| C3—C7—C8—N3 | 177.4 (2) | C18—C19—C20—C15 | 0.6 (5) |
| C9—N3—C8—C7 | 2.1 (3) | C16—C15—C20—C19 | -1.3 (4) |
| S1—N3—C8—C7 | 168.4 (2) | S1—C15—C20—C19 | 178.6 (2) |

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--------------------------------|-------|-------------|-------------|---------------|
| C8—H8 \cdots O2 ⁱ | 0.93 | 2.46 | 3.380 (3) | 168 |
| C14—H14 \cdots O1 | 0.93 | 2.54 | 3.099 (4) | 119 |

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

5-[1-(Benzenesulfonyl)-1*H*-indol-3-yl]-2,1,3-benzothiadiazole (bS)*Crystal data*

| | |
|---------------------------------|---|
| $C_{14}H_9N_3S$ | $D_x = 1.432 \text{ Mg m}^{-3}$ |
| $M_r = 251.30$ | Cu $K\alpha$ radiation, $\lambda = 1.54184 \text{ \AA}$ |
| Orthorhombic, <i>Pbca</i> | Cell parameters from 4620 reflections |
| $a = 7.5884 (1) \text{ \AA}$ | $\theta = 4.1\text{--}67.1^\circ$ |
| $b = 7.1060 (1) \text{ \AA}$ | $\mu = 2.32 \text{ mm}^{-1}$ |
| $c = 43.2464 (7) \text{ \AA}$ | $T = 295 \text{ K}$ |
| $V = 2331.98 (6) \text{ \AA}^3$ | Plate, colourless |
| $Z = 8$ | $0.29 \times 0.18 \times 0.04 \text{ mm}$ |
| $F(000) = 1040$ | |

Data collection

| | |
|---|--|
| Xcalibur, Ruby, Gemini ultra R diffractometer | 10906 measured reflections |
| Radiation source: fine-focus sealed tube | 2072 independent reflections |
| Detector resolution: $5.1856 \text{ pixels mm}^{-1}$ | 1825 reflections with $I > 2\sigma(I)$ |
| ω scans | $R_{\text{int}} = 0.025$ |
| Absorption correction: analytical (CrysAlisPro; Rigaku OD, 2020) | $\theta_{\text{max}} = 67.2^\circ$, $\theta_{\text{min}} = 4.1^\circ$ |
| $T_{\text{min}} = 0.663$, $T_{\text{max}} = 0.920$ | $h = -9 \rightarrow 8$ |
| | $k = -8 \rightarrow 7$ |
| | $l = -51 \rightarrow 49$ |

Refinement

| | |
|----------------------------------|---|
| Refinement on F^2 | Secondary atom site location: dual |
| Least-squares matrix: full | Hydrogen site location: mixed |
| $R[F^2 > 2\sigma(F^2)] = 0.035$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.098$ | $w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 0.7413P]$ |
| $S = 1.07$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 2072 reflections | $(\Delta/\sigma)_{\text{max}} = 0.001$ |
| 167 parameters | $\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$ |
| 0 restraints | $\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$ |
| 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.

Refinement. Structures were solved by the dual-space method of ShelXT (Sheldrick, 2015a) within Olex² (Dolomanov *et al.*, 2009). Structures were refined by the least-squares method implemented in SHELXL (Sheldrick, 2015b) within ShelXle (Hübschle *et al.*, 2011). Structures and crystal packings were visualized using Mercury (Macrae *et al.*, 2020).

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

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|-------------|-------------|----------------------------------|
| S1 | 1.08880 (6) | 0.65005 (9) | 0.46249 (2) | 0.0684 (2) |
| N1 | 1.05463 (19) | 0.6759 (2) | 0.42591 (3) | 0.0539 (4) |
| N3 | 0.2880 (2) | 0.8454 (2) | 0.33487 (4) | 0.0567 (4) |
| H3N | 0.206 (3) | 0.904 (3) | 0.3295 (5) | 0.065 (6)* |
| C9 | 0.5346 (2) | 0.6723 (2) | 0.33531 (4) | 0.0369 (3) |
| C5 | 0.6078 (2) | 0.7131 (2) | 0.39422 (3) | 0.0384 (4) |
| C6 | 0.7877 (2) | 0.7053 (2) | 0.39392 (3) | 0.0396 (4) |

| | | | | |
|-----|------------|------------|-------------|------------|
| H6 | 0.849325 | 0.714886 | 0.375400 | 0.048* |
| C7 | 0.5027 (2) | 0.7429 (2) | 0.36611 (4) | 0.0395 (4) |
| N2 | 0.8896 (2) | 0.6472 (3) | 0.47547 (4) | 0.0649 (5) |
| C14 | 0.6590 (2) | 0.5510 (2) | 0.32215 (4) | 0.0453 (4) |
| H14 | 0.750683 | 0.502873 | 0.334031 | 0.054* |
| C10 | 0.3973 (2) | 0.7415 (2) | 0.31639 (4) | 0.0441 (4) |
| C1 | 0.8784 (2) | 0.6826 (2) | 0.42215 (4) | 0.0415 (4) |
| C4 | 0.5157 (2) | 0.6949 (3) | 0.42314 (4) | 0.0480 (4) |
| H4 | 0.393193 | 0.698955 | 0.422964 | 0.058* |
| C2 | 0.7846 (2) | 0.6662 (3) | 0.45063 (4) | 0.0476 (4) |
| C3 | 0.5985 (2) | 0.6722 (3) | 0.45054 (4) | 0.0543 (5) |
| H3 | 0.534867 | 0.660972 | 0.468820 | 0.065* |
| C13 | 0.6439 (3) | 0.5037 (3) | 0.29140 (4) | 0.0567 (5) |
| H13 | 0.725749 | 0.422248 | 0.282623 | 0.068* |
| C11 | 0.3836 (3) | 0.6946 (3) | 0.28513 (4) | 0.0547 (5) |
| H11 | 0.293096 | 0.742288 | 0.272936 | 0.066* |
| C8 | 0.3506 (2) | 0.8457 (3) | 0.36445 (4) | 0.0518 (4) |
| H8 | 0.297698 | 0.906861 | 0.381037 | 0.062* |
| C12 | 0.5077 (3) | 0.5761 (3) | 0.27307 (4) | 0.0594 (5) |
| H12 | 0.501492 | 0.542837 | 0.252302 | 0.071* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|---------------|-------------|
| S1 | 0.0416 (3) | 0.1158 (5) | 0.0479 (3) | −0.0007 (3) | −0.00962 (19) | −0.0036 (3) |
| N1 | 0.0354 (7) | 0.0777 (11) | 0.0486 (8) | −0.0024 (7) | −0.0023 (6) | −0.0024 (7) |
| N3 | 0.0500 (9) | 0.0557 (9) | 0.0645 (10) | 0.0187 (8) | −0.0130 (7) | 0.0043 (7) |
| C9 | 0.0349 (8) | 0.0340 (7) | 0.0417 (8) | −0.0034 (6) | −0.0024 (6) | 0.0053 (6) |
| C5 | 0.0371 (8) | 0.0380 (8) | 0.0399 (8) | −0.0005 (6) | 0.0003 (6) | −0.0026 (6) |
| C6 | 0.0363 (8) | 0.0460 (9) | 0.0365 (8) | 0.0000 (7) | 0.0031 (6) | −0.0003 (6) |
| C7 | 0.0363 (8) | 0.0375 (8) | 0.0448 (8) | 0.0013 (7) | −0.0009 (6) | 0.0014 (7) |
| N2 | 0.0484 (9) | 0.1062 (14) | 0.0401 (8) | 0.0008 (9) | −0.0042 (7) | −0.0029 (8) |
| C14 | 0.0431 (9) | 0.0462 (9) | 0.0466 (9) | 0.0039 (7) | −0.0008 (7) | 0.0019 (7) |
| C10 | 0.0456 (9) | 0.0373 (8) | 0.0494 (9) | −0.0009 (7) | −0.0065 (7) | 0.0082 (7) |
| C1 | 0.0336 (8) | 0.0490 (9) | 0.0421 (8) | −0.0013 (7) | 0.0012 (6) | −0.0035 (7) |
| C4 | 0.0323 (8) | 0.0651 (11) | 0.0465 (9) | 0.0003 (8) | 0.0048 (7) | −0.0020 (8) |
| C2 | 0.0423 (9) | 0.0634 (11) | 0.0370 (8) | 0.0001 (8) | −0.0005 (7) | −0.0032 (7) |
| C3 | 0.0424 (10) | 0.0812 (13) | 0.0393 (9) | −0.0010 (9) | 0.0090 (7) | −0.0006 (8) |
| C13 | 0.0638 (12) | 0.0558 (11) | 0.0504 (10) | 0.0032 (9) | 0.0050 (9) | −0.0068 (8) |
| C11 | 0.0603 (11) | 0.0553 (10) | 0.0486 (10) | −0.0060 (9) | −0.0165 (8) | 0.0119 (8) |
| C8 | 0.0473 (10) | 0.0537 (10) | 0.0543 (10) | 0.0137 (8) | −0.0026 (8) | −0.0043 (8) |
| C12 | 0.0739 (13) | 0.0635 (12) | 0.0409 (9) | −0.0103 (11) | −0.0051 (9) | −0.0008 (8) |

Geometric parameters (Å, °)

| | | | |
|-------|-------------|---------|-----------|
| S1—N2 | 1.6128 (17) | N2—C2 | 1.344 (2) |
| S1—N1 | 1.6136 (16) | C14—C13 | 1.376 (2) |
| N1—C1 | 1.348 (2) | C14—H14 | 0.9300 |

| | | | |
|--------------|--------------|-------------|--------------|
| N3—C8 | 1.365 (2) | C10—C11 | 1.396 (2) |
| N3—C10 | 1.368 (2) | C1—C2 | 1.428 (2) |
| N3—H3N | 0.79 (2) | C4—C3 | 1.351 (2) |
| C9—C14 | 1.399 (2) | C4—H4 | 0.9300 |
| C9—C10 | 1.413 (2) | C2—C3 | 1.413 (3) |
| C9—C7 | 1.444 (2) | C3—H3 | 0.9300 |
| C5—C6 | 1.367 (2) | C13—C12 | 1.400 (3) |
| C5—C4 | 1.438 (2) | C13—H13 | 0.9300 |
| C5—C7 | 1.469 (2) | C11—C12 | 1.366 (3) |
| C6—C1 | 1.411 (2) | C11—H11 | 0.9300 |
| C6—H6 | 0.9300 | C8—H8 | 0.9300 |
| C7—C8 | 1.368 (2) | C12—H12 | 0.9300 |
| | | | |
| N2—S1—N1 | 101.07 (8) | N1—C1—C6 | 126.36 (15) |
| C1—N1—S1 | 106.37 (12) | N1—C1—C2 | 112.81 (15) |
| C8—N3—C10 | 109.73 (15) | C6—C1—C2 | 120.82 (15) |
| C8—N3—H3N | 123.5 (16) | C3—C4—C5 | 123.18 (16) |
| C10—N3—H3N | 126.6 (16) | C3—C4—H4 | 118.4 |
| C14—C9—C10 | 118.42 (15) | C5—C4—H4 | 118.4 |
| C14—C9—C7 | 134.62 (15) | N2—C2—C3 | 126.70 (17) |
| C10—C9—C7 | 106.86 (14) | N2—C2—C1 | 113.72 (16) |
| C6—C5—C4 | 119.35 (15) | C3—C2—C1 | 119.58 (15) |
| C6—C5—C7 | 122.68 (14) | C4—C3—C2 | 118.11 (16) |
| C4—C5—C7 | 117.97 (14) | C4—C3—H3 | 120.9 |
| C5—C6—C1 | 118.95 (14) | C2—C3—H3 | 120.9 |
| C5—C6—H6 | 120.5 | C14—C13—C12 | 121.24 (18) |
| C1—C6—H6 | 120.5 | C14—C13—H13 | 119.4 |
| C8—C7—C9 | 106.16 (14) | C12—C13—H13 | 119.4 |
| C8—C7—C5 | 125.33 (15) | C12—C11—C10 | 117.74 (17) |
| C9—C7—C5 | 128.51 (14) | C12—C11—H11 | 121.1 |
| C2—N2—S1 | 106.03 (13) | C10—C11—H11 | 121.1 |
| C13—C14—C9 | 119.16 (16) | N3—C8—C7 | 110.01 (16) |
| C13—C14—H14 | 120.4 | N3—C8—H8 | 125.0 |
| C9—C14—H14 | 120.4 | C7—C8—H8 | 125.0 |
| N3—C10—C11 | 130.51 (16) | C11—C12—C13 | 121.26 (17) |
| N3—C10—C9 | 107.23 (15) | C11—C12—H12 | 119.4 |
| C11—C10—C9 | 122.17 (16) | C13—C12—H12 | 119.4 |
| | | | |
| N2—S1—N1—C1 | -0.35 (15) | C5—C6—C1—N1 | -178.66 (17) |
| C4—C5—C6—C1 | -1.0 (2) | C5—C6—C1—C2 | 0.5 (2) |
| C7—C5—C6—C1 | 177.87 (14) | C6—C5—C4—C3 | 0.8 (3) |
| C14—C9—C7—C8 | -175.18 (18) | C7—C5—C4—C3 | -178.16 (17) |
| C10—C9—C7—C8 | 0.92 (18) | S1—N2—C2—C3 | -179.70 (18) |
| C14—C9—C7—C5 | 4.5 (3) | S1—N2—C2—C1 | -0.2 (2) |
| C10—C9—C7—C5 | -179.43 (15) | N1—C1—C2—N2 | -0.1 (2) |
| C6—C5—C7—C8 | -143.47 (18) | C6—C1—C2—N2 | -179.37 (16) |
| C4—C5—C7—C8 | 35.4 (2) | N1—C1—C2—C3 | 179.49 (18) |
| C6—C5—C7—C9 | 36.9 (3) | C6—C1—C2—C3 | 0.2 (3) |

| | | | |
|----------------|--------------|-----------------|--------------|
| C4—C5—C7—C9 | -144.19 (17) | C5—C4—C3—C2 | 0.0 (3) |
| N1—S1—N2—C2 | 0.31 (16) | N2—C2—C3—C4 | 179.05 (19) |
| C10—C9—C14—C13 | 0.2 (2) | C1—C2—C3—C4 | -0.4 (3) |
| C7—C9—C14—C13 | 175.93 (18) | C9—C14—C13—C12 | 0.6 (3) |
| C8—N3—C10—C11 | 176.87 (18) | N3—C10—C11—C12 | -175.41 (19) |
| C8—N3—C10—C9 | 0.3 (2) | C9—C10—C11—C12 | 0.7 (3) |
| C14—C9—C10—N3 | 176.06 (14) | C10—N3—C8—C7 | 0.3 (2) |
| C7—C9—C10—N3 | -0.78 (18) | C9—C7—C8—N3 | -0.7 (2) |
| C14—C9—C10—C11 | -0.8 (2) | C5—C7—C8—N3 | 179.60 (15) |
| C7—C9—C10—C11 | -177.65 (15) | C10—C11—C12—C13 | 0.1 (3) |
| S1—N1—C1—C6 | 179.53 (14) | C14—C13—C12—C11 | -0.7 (3) |
| S1—N1—C1—C2 | 0.27 (19) | | |

5-(1*H*-Indol-3-yl)-2,1,3-benzoxadiazole (bO)

Crystal data

C₁₄H₉N₃O

M_r = 235.24

Orthorhombic, *Pbca*

a = 12.0256 (7) Å

b = 7.7396 (5) Å

c = 23.8551 (16) Å

V = 2220.3 (2) Å³

Z = 8

F(000) = 976

D_x = 1.407 Mg m⁻³

Cu *Kα* radiation, λ = 1.54184 Å

Cell parameters from 1395 reflections

θ = 7.4–66.3°

μ = 0.75 mm⁻¹

T = 295 K

Plate, clear yellow

0.12 × 0.09 × 0.03 mm

Data collection

Xcalibur, Ruby, Gemini ultra
diffractometer

Radiation source: fine-focus sealed X-ray tube,
Enhance Ultra (Cu) X-ray Source

Detector resolution: 5.1856 pixels mm⁻¹

ω scans

Absorption correction: analytical
(CrysAlisPro; Rigaku OD, 2020)

T_{min} = 0.941, *T_{max}* = 0.981

6883 measured reflections

1972 independent reflections

1275 reflections with *I* > 2σ(*I*)

R_{int} = 0.047

θ_{max} = 67.1°, θ_{min} = 3.7°

h = -14→12

k = -6→9

l = -28→19

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.050

wR(*F*²) = 0.134

S = 1.06

1972 reflections

166 parameters

0 restraints

Primary atom site location: dual

Secondary atom site location: dual

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

w = 1/[σ²(*F_o*²) + (0.0633*P*)² + 0.0873*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.14 e Å⁻³

Δρ_{min} = -0.16 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.

Refinement. Structures were solved by the dual-space method of ShelXT (Sheldrick, 2015a) within Olex² (Dolomanov *et al.*, 2009). Structures were refined by the least-squares method implemented in SHELXL (Sheldrick, 2015b) within ShelXle (Hübschle *et al.*, 2011). Structures and crystal packings were visualized using Mercury (Macrae *et al.*, 2020).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

| | <i>x</i> | <i>y</i> | <i>z</i> | <i>U</i> _{iso} [*] / <i>U</i> _{eq} |
|-----|--------------|------------|--------------|---|
| O1 | 0.26211 (19) | 0.2142 (3) | 0.43917 (8) | 0.0909 (6) |
| N1 | 0.1692 (2) | 0.2567 (3) | 0.46968 (9) | 0.0801 (7) |
| N2 | 0.3589 (2) | 0.2507 (3) | 0.46824 (9) | 0.0822 (7) |
| N3 | 0.52478 (16) | 0.6406 (3) | 0.70573 (9) | 0.0640 (6) |
| H3N | 0.581 (2) | 0.696 (3) | 0.7137 (11) | 0.077* |
| C1 | 0.2075 (2) | 0.3199 (3) | 0.51701 (10) | 0.0629 (6) |
| C2 | 0.3259 (2) | 0.3161 (3) | 0.51611 (10) | 0.0616 (6) |
| C3 | 0.3883 (2) | 0.3735 (3) | 0.56268 (10) | 0.0610 (6) |
| H3A | 0.465597 | 0.368956 | 0.562224 | 0.073* |
| C4 | 0.33330 (18) | 0.4356 (3) | 0.60815 (9) | 0.0520 (6) |
| C5 | 0.21290 (18) | 0.4396 (3) | 0.60788 (10) | 0.0566 (6) |
| H5 | 0.176549 | 0.482253 | 0.639392 | 0.068* |
| C6 | 0.1509 (2) | 0.3849 (3) | 0.56443 (10) | 0.0658 (7) |
| H6 | 0.073675 | 0.389643 | 0.565567 | 0.079* |
| C7 | 0.39274 (17) | 0.5038 (3) | 0.65679 (9) | 0.0506 (5) |
| C8 | 0.49217 (18) | 0.5924 (3) | 0.65382 (10) | 0.0610 (6) |
| H8 | 0.530990 | 0.615615 | 0.620952 | 0.073* |
| C9 | 0.36413 (16) | 0.4987 (2) | 0.71516 (9) | 0.0473 (5) |
| C10 | 0.44927 (17) | 0.5855 (3) | 0.74484 (10) | 0.0513 (6) |
| C11 | 0.4497 (2) | 0.6039 (3) | 0.80242 (11) | 0.0624 (6) |
| H11 | 0.506433 | 0.663393 | 0.820582 | 0.075* |
| C12 | 0.3635 (2) | 0.5313 (3) | 0.83195 (11) | 0.0654 (7) |
| H12 | 0.361933 | 0.541208 | 0.870791 | 0.078* |
| C13 | 0.27815 (19) | 0.4427 (3) | 0.80449 (11) | 0.0606 (6) |
| H13 | 0.220822 | 0.394500 | 0.825468 | 0.073* |
| C14 | 0.27709 (17) | 0.4252 (3) | 0.74691 (10) | 0.0527 (5) |
| H14 | 0.219735 | 0.365798 | 0.729246 | 0.063* |

Atomic displacement parameters (Å²)

| | <i>U</i> ¹¹ | <i>U</i> ²² | <i>U</i> ³³ | <i>U</i> ¹² | <i>U</i> ¹³ | <i>U</i> ²³ |
|----|------------------------|------------------------|------------------------|------------------------|------------------------|------------------------|
| O1 | 0.1045 (15) | 0.1109 (16) | 0.0571 (10) | −0.0033 (12) | −0.0023 (11) | −0.0055 (10) |
| N1 | 0.0843 (15) | 0.0991 (17) | 0.0569 (14) | −0.0107 (13) | −0.0056 (13) | 0.0038 (12) |
| N2 | 0.0843 (16) | 0.1040 (17) | 0.0582 (14) | 0.0035 (13) | 0.0010 (12) | −0.0021 (12) |
| N3 | 0.0455 (10) | 0.0674 (13) | 0.0791 (15) | −0.0127 (9) | −0.0027 (10) | 0.0060 (11) |
| C1 | 0.0670 (15) | 0.0709 (15) | 0.0507 (15) | −0.0074 (12) | −0.0041 (12) | 0.0099 (12) |
| C2 | 0.0682 (15) | 0.0693 (15) | 0.0473 (14) | 0.0029 (12) | 0.0075 (12) | 0.0066 (12) |
| C3 | 0.0510 (13) | 0.0737 (15) | 0.0585 (15) | 0.0027 (12) | 0.0037 (12) | 0.0057 (12) |
| C4 | 0.0474 (11) | 0.0533 (12) | 0.0552 (13) | −0.0006 (10) | 0.0027 (11) | 0.0089 (11) |
| C5 | 0.0466 (12) | 0.0683 (14) | 0.0547 (14) | 0.0018 (11) | 0.0013 (11) | 0.0035 (11) |
| C6 | 0.0506 (13) | 0.0811 (17) | 0.0655 (16) | −0.0038 (12) | −0.0020 (12) | 0.0079 (13) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C7 | 0.0437 (11) | 0.0512 (11) | 0.0570 (14) | 0.0000 (10) | -0.0030 (10) | 0.0048 (11) |
| C8 | 0.0485 (12) | 0.0708 (14) | 0.0636 (15) | -0.0056 (11) | 0.0031 (11) | 0.0118 (12) |
| C9 | 0.0414 (10) | 0.0439 (10) | 0.0567 (13) | 0.0029 (9) | 0.0002 (10) | 0.0028 (10) |
| C10 | 0.0440 (11) | 0.0474 (12) | 0.0626 (15) | 0.0012 (10) | -0.0038 (11) | 0.0010 (11) |
| C11 | 0.0602 (14) | 0.0566 (13) | 0.0704 (16) | 0.0025 (12) | -0.0105 (13) | -0.0099 (12) |
| C12 | 0.0720 (16) | 0.0630 (15) | 0.0613 (15) | 0.0119 (13) | 0.0023 (13) | -0.0066 (12) |
| C13 | 0.0551 (13) | 0.0618 (14) | 0.0648 (15) | 0.0076 (11) | 0.0122 (12) | 0.0040 (12) |
| C14 | 0.0424 (11) | 0.0514 (12) | 0.0642 (14) | 0.0018 (10) | 0.0023 (11) | 0.0031 (11) |

Geometric parameters (Å, °)

| | | | |
|------------|-------------|-------------|-------------|
| O1—N1 | 1.373 (3) | C5—H5 | 0.9300 |
| O1—N2 | 1.384 (3) | C6—H6 | 0.9300 |
| N1—C1 | 1.314 (3) | C7—C8 | 1.380 (3) |
| N2—C2 | 1.310 (3) | C7—C9 | 1.435 (3) |
| N3—C8 | 1.351 (3) | C8—H8 | 0.9300 |
| N3—C10 | 1.370 (3) | C9—C14 | 1.411 (3) |
| N3—H3N | 0.83 (3) | C9—C10 | 1.415 (3) |
| C1—C6 | 1.413 (3) | C10—C11 | 1.381 (3) |
| C1—C2 | 1.424 (4) | C11—C12 | 1.374 (3) |
| C2—C3 | 1.413 (3) | C11—H11 | 0.9300 |
| C3—C4 | 1.358 (3) | C12—C13 | 1.397 (4) |
| C3—H3A | 0.9300 | C12—H12 | 0.9300 |
| C4—C5 | 1.448 (3) | C13—C14 | 1.380 (3) |
| C4—C7 | 1.461 (3) | C13—H13 | 0.9300 |
| C5—C6 | 1.345 (3) | C14—H14 | 0.9300 |
| N1—O1—N2 | 111.69 (18) | C8—C7—C9 | 105.7 (2) |
| C1—N1—O1 | 105.1 (2) | C8—C7—C4 | 124.2 (2) |
| C2—N2—O1 | 105.1 (2) | C9—C7—C4 | 130.03 (19) |
| C8—N3—C10 | 110.23 (19) | N3—C8—C7 | 110.0 (2) |
| C8—N3—H3N | 126.4 (19) | N3—C8—H8 | 125.0 |
| C10—N3—H3N | 123.3 (19) | C7—C8—H8 | 125.0 |
| N1—C1—C6 | 130.7 (2) | C14—C9—C10 | 117.4 (2) |
| N1—C1—C2 | 109.2 (2) | C14—C9—C7 | 135.2 (2) |
| C6—C1—C2 | 120.1 (2) | C10—C9—C7 | 107.39 (18) |
| N2—C2—C3 | 130.2 (2) | N3—C10—C11 | 130.0 (2) |
| N2—C2—C1 | 108.9 (2) | N3—C10—C9 | 106.7 (2) |
| C3—C2—C1 | 120.8 (2) | C11—C10—C9 | 123.3 (2) |
| C4—C3—C2 | 118.7 (2) | C12—C11—C10 | 117.7 (2) |
| C4—C3—H3A | 120.6 | C12—C11—H11 | 121.1 |
| C2—C3—H3A | 120.6 | C10—C11—H11 | 121.1 |
| C3—C4—C5 | 119.4 (2) | C11—C12—C13 | 121.0 (2) |
| C3—C4—C7 | 121.6 (2) | C11—C12—H12 | 119.5 |
| C5—C4—C7 | 119.0 (2) | C13—C12—H12 | 119.5 |
| C6—C5—C4 | 123.4 (2) | C14—C13—C12 | 121.4 (2) |
| C6—C5—H5 | 118.3 | C14—C13—H13 | 119.3 |
| C4—C5—H5 | 118.3 | C12—C13—H13 | 119.3 |

| | | | |
|-------------|------------|-----------------|-------------|
| C5—C6—C1 | 117.5 (2) | C13—C14—C9 | 119.2 (2) |
| C5—C6—H6 | 121.2 | C13—C14—H14 | 120.4 |
| C1—C6—H6 | 121.2 | C9—C14—H14 | 120.4 |
| | | | |
| N2—O1—N1—C1 | 0.4 (3) | C5—C4—C7—C9 | 35.3 (3) |
| N1—O1—N2—C2 | -0.4 (3) | C10—N3—C8—C7 | 0.3 (3) |
| O1—N1—C1—C6 | 179.9 (3) | C9—C7—C8—N3 | -0.1 (2) |
| O1—N1—C1—C2 | -0.3 (3) | C4—C7—C8—N3 | 179.06 (19) |
| O1—N2—C2—C3 | 178.7 (2) | C8—C7—C9—C14 | -177.6 (2) |
| O1—N2—C2—C1 | 0.2 (3) | C4—C7—C9—C14 | 3.3 (4) |
| N1—C1—C2—N2 | 0.0 (3) | C8—C7—C9—C10 | -0.1 (2) |
| C6—C1—C2—N2 | 179.9 (2) | C4—C7—C9—C10 | -179.2 (2) |
| N1—C1—C2—C3 | -178.6 (2) | C8—N3—C10—C11 | 179.1 (2) |
| C6—C1—C2—C3 | 1.2 (3) | C8—N3—C10—C9 | -0.4 (2) |
| N2—C2—C3—C4 | -179.5 (3) | C14—C9—C10—N3 | 178.34 (18) |
| C1—C2—C3—C4 | -1.1 (3) | C7—C9—C10—N3 | 0.3 (2) |
| C2—C3—C4—C5 | 0.6 (3) | C14—C9—C10—C11 | -1.2 (3) |
| C2—C3—C4—C7 | -177.4 (2) | C7—C9—C10—C11 | -179.3 (2) |
| C3—C4—C5—C6 | -0.1 (3) | N3—C10—C11—C12 | -178.4 (2) |
| C7—C4—C5—C6 | 177.9 (2) | C9—C10—C11—C12 | 1.0 (3) |
| C4—C5—C6—C1 | 0.2 (3) | C10—C11—C12—C13 | -0.3 (3) |
| N1—C1—C6—C5 | 179.0 (3) | C11—C12—C13—C14 | -0.1 (3) |
| C2—C1—C6—C5 | -0.8 (3) | C12—C13—C14—C9 | -0.1 (3) |
| C3—C4—C7—C8 | 34.3 (3) | C10—C9—C14—C13 | 0.7 (3) |
| C5—C4—C7—C8 | -143.6 (2) | C7—C9—C14—C13 | 178.0 (2) |
| C3—C4—C7—C9 | -146.7 (2) | | |
