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N-Cycloamino substituent effects on the packing architecture of *ortho*-sulfanilamide molecular crystals and their *in silico* carbonic anhydrase II and IX inhibitory activities

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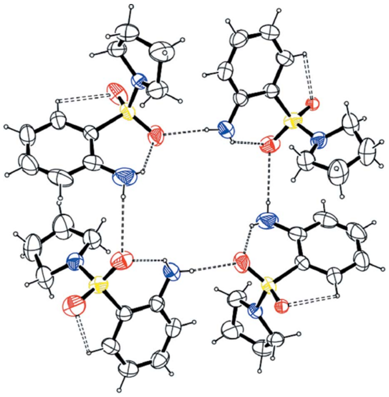
In the search for new 'sulfa drugs' with therapeutic properties, *o*-nitrosulfonamides and *N*-cycloamino-*o*-sulfanilamides were synthesized and characterized using techniques including ^{1}H NMR, ^{13}C NMR and FT–IR spectroscopy, and single-crystal X-ray diffraction (SC-XRD). The calculated density functional theory (DFT)-optimized geometry of the molecules showed similar conformations to those obtained by SC-XRD. Molecular docking of *N*-piperidinyl-*o*-sulfanilamide and *N*-indolinyl-*o*-sulfanilamide supports the notion that *o*-sulfanilamides are able to bind to human carbonic anhydrase II and IX inhibitors (hCA II and IX; PDB entries 4iwz and 5fl4). Hirshfeld surface analyses and DFT studies of three *o*-nitrosulfonamides {1-[(2-nitrophenyl)sulfonyl]pyrrolidine, $C_{10}H_{12}N_2O_4S$, **1**, 1-[(2-nitrophenyl)sulfonyl]piperidine, $C_{11}H_{14}N_2O_4S$, **2**, and 1-[(2-nitrophenyl)sulfonyl]-2,3-dihydro-1*H*-indole, $C_{14}H_{12}N_2O_4S$, **3**} and three *N*-cycloamino-*o*-sulfanilamides [2-(pyrrolidine-1-sulfonyl)aniline, $C_{10}H_{14}N_2O_2S$, **4**, 2-(piperidine-1-sulfonyl)aniline, $C_{11}H_{16}N_2O_2S$, **5**, and 2-(2,3-dihydro-1*H*-indole-1-sulfonyl)aniline, $C_{14}H_{14}N_2O_2S$, **6**] suggested that forces such as hydrogen bonding and π–π interactions hold molecules together and further showed that charge transfer could promote bioactivity and the ability to form biological interactions at the piperidinyl and phenyl moieties.



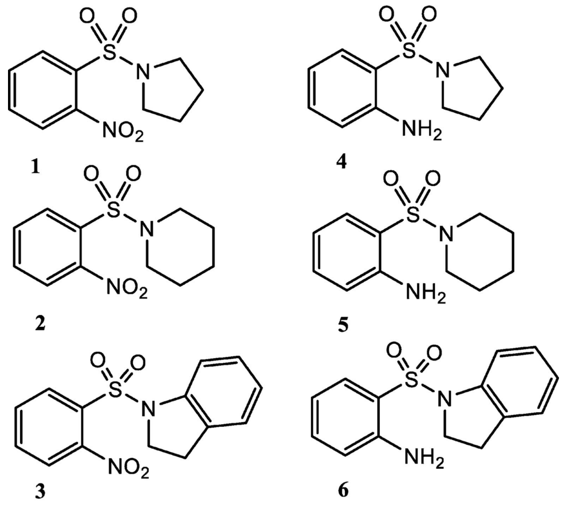
1. Introduction

Sulfanilamide (4-aminobenzenesulfonamide) is aptly described as the antecedent of the group of therapeutics known as 'sulfa drugs', which ushered in the modern era of antibacterial chemotherapy (Ajani *et al.*, 2012). Although it had been a component of a staple azo dye in the colour industry since the beginning of the 20th century, it did not gain prominence in medicine until the 1930s when Gerhard Domagk and co-workers patented Prontosil, **A** (Fig. 1), a sulfanilamide prodrug, which not only revolutionized the treatment of bacterial infections, but chemotherapy as a whole, and led to the development of other drugs for non-infectious diseases.

It has been established that the bacteriostatic properties of sulfanilamides (Fig. 1) are predicated based on two major motifs: the aryl amine (–NH_2) and sulfonamide (–SO_2NHR) groups (Lesch, 2007). A free or hydrolysable substituted amino (–NHR') moiety that is *para* to the sulfonamido group has been reported to be crucial for antibacterial activity, whereas modification of the position to the *ortho* and/or *meta* position results in non-antibacterial activities (Ajani *et al.*, 2012). The derivatization of the sulfonamido group with heterocycles has also produced more potent antibiotics (Ajani *et al.*, 2012; Lesch, 2007). In addition, it has been long reported that no correlations exist between the toxicities and therapeutic efficiencies, as well as toxicities and solubilities, of the three isomers of sulfanilamide, as evidenced by the finding that even though *meta*-sulfanilamide **C** was the least toxic of the three, only *para*-sulfanilamide **B** possessed bacteriostatic activity (Laug & Morris, 1939). Notably, the inhibitions of the *Helicobacter pylori* α-class carbonic anhydrase (hpCA) (Nishimori *et al.*, 2006) and tumour-associated transmembrane carbonic anhydrase IX (CA IX) (Vullo *et al.*, 2003) isozymes have been observed with *ortho*-sulfanilamide **D** (orthanilamide). Sulfonamides **E** are derivatives of sulfanilamide and remain an important class of drugs, with antibacterial and non-antibacterial potencies, such as diuretic, antimicrobial, antiepileptic, antileprotic, antimalarial, hypoglycemic, antiretroviral, antithyroid and anti-inflammatory activities (Gul *et al.*, 2016; Henry, 1943; Casini *et al.*, 2002; Mohan *et al.*, 2006; Alex & Storer, 2010).

They also inhibit carbonic anhydrase (Gul *et al.*, 2016; Ghorab *et al.*, 2014; Nocentini *et al.*, 2016) and have been reported to show *in vivo* and/or *in vitro* antitumour activities (Boyland, 1946). Many of these sulfonamide-based (sulfa) drugs, reported to be in clinical trials, are devoid of the side effects plaguing most of the current pharmacological agents (Casini *et al.*, 2002; Owa *et al.*, 2002; Lavanya, 2017; Andreucci *et al.*, 2019).

The identification of pharmacologically active moieties in model molecules and lead candidates of physiological significance from a vast array of substances, with the potential of further optimization, is a crucial facet of rational drug design and discovery (Voronin *et al.*, 2020). The process of optimization, it must be noted, typically involves structure–activity relationship studies that facilitate the selection of molecules with optimal receptor affinities (Bloom & Laubach, 1962; Kalgutkar *et al.*, 2010; Sly & Hu, 1995; Lehtonen *et al.*, 2004; Żołnowska *et al.*, 2014; Thiry *et al.*, 2008; Angeli *et al.*, 2020; Güzel-Akdemir *et al.*, 2015; Rutkauskas *et al.*, 2014; Congiu *et al.*, 2014; Temperini *et al.*, 2008*a*,*b*; Chiche *et al.*, 2010; Türeci *et al.*, 1998; PDB, http://www.rcsb.org/pdb; Berman *et al.*, 2000). In continuation of the design of potential 'sulfa drugs', we report the synthesis, structural and theoretical studies, and docking application of the *o*-nitrosulfonamides 1-[(2-nitrophenyl)sulfonyl]pyrrolidine, **1**, 1-[(2-nitrophenyl)sulfonyl]piperidine, **2**, and 1-[(2-nitrophenyl)sulfonyl]-2,3-dihydro-1*H*-indole, **3**, and the *N*-cycloamino-*o*-sulfanilamides 2-(pyrrolidine-1-sulfonyl)aniline, **4**, 2-(piperidine-1-sulfonyl)aniline, **5**, and 2-(2,3-dihydro-1*H*-indole-1-sulfonyl)aniline, **6**. The crystal structures, density functional theory (DFT) studies, Hirshfeld surface analysis, molecular electrostatic potential and electronic properties of the title sulfonamides and sulfanilamides (**1–6**) have been discussed. Molecular docking experiments with carbonic anhydrase II (PDB entry 4iwz) and IX (5fl4) active sites were conducted in order to predict their binding interactions with **1–6** (Scheme 1).



Scheme 1

2. Experimental

2.1. Instruments and measurements

All reagents were purchased from Millipore Sigma (Germany and South Africa) and were used without purification. The melting points were determined on an Electrothermal digital melting-point apparatus and are uncorrected. Reac-

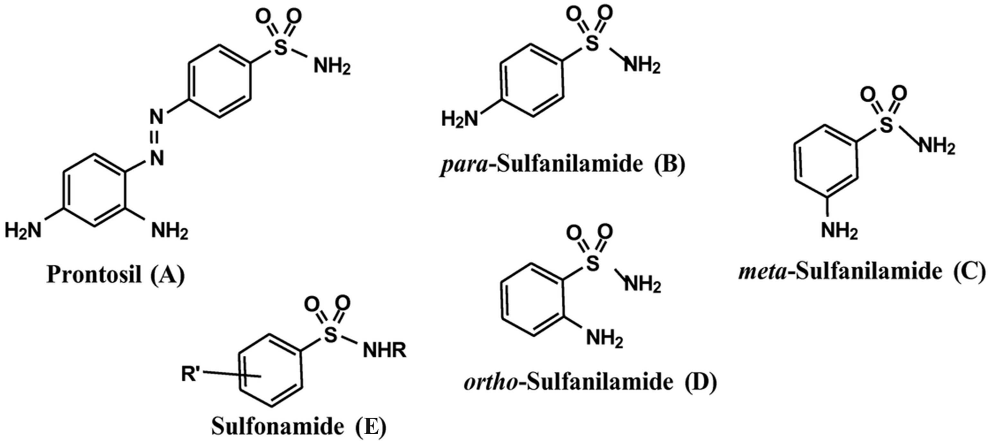


Figure 1
Sulfanilamides and some prodrugs.

tions were monitored by thin-layer chromatography (TLC) on Merck silica gel 60 F_{254} precoated plates using a dichloromethane/*n*-hexane (2 or 1.4:1 *v*/*v*) solvent system visualized under a UV lamp (254 nm). Column chromatography was performed with silica gel (70–230 mesh ASTM) and mobile phases were as indicated. Sample crystallization was achieved by the slow evaporation of the indicated solvent systems at ambient temperature. IR spectra were obtained using a Bruker Tensor 27 platinum ATR–FT–IR spectrometer. The ATR–FT–IR spectra were acquired in a single mode with a resolution of 4 cm^{-1} over 32 scans, in the region 4000–650 cm^{-1}. ^1H and ^{13}C NMR spectra were recorded, in $CDCl_3$, on a Bruker 400 MHz spectrometer. Chemical shift (δ) values were measured in parts per million (ppm) downfield from tetramethylsilane (TMS) and coupling constants (J) are reported in hertz (Hz). Theoretical studies were performed for the compounds and, in each case, their SC-XRD structures were used for optimization and global reactivity descriptor (GRD) calculations.

2.2. Synthesis and crystallization

2.2.1. Synthesis of *N*-cycloamino-*o*-nitrobenzenesulfonamides 1–3. *o*-Nitrobenzenesulfonyl chloride (1.00 mmol) was added slowly to a stirring dried toluene solution (30 ml) of the cycloamine (2.20 mmol) at ambient temperature and stirred for 12 h, monitored by TLC. The reaction mixture was then diluted with dichloromethane (30 ml) and washed with distilled water (3 × 10 ml). The organic layer was separated, dried over anhydrous sodium sulfate, filtered and concentrated to an oil, which was purified by column chromatography on silica gel (dichloromethane/*n*-hexane, 2:1 *v*/*v*). Crystals were obtained by the slow solvent evaporation of the requisite eluates at ambient temperature, except for **5**, which was recrystallized from dichloromethane, slowly evaporated and filtered to give single crystals.

*2.2.1.1. N-Pyrrolidinyl-o-nitrobenzenesulfonamide, **1**.* *o*-Nitrobenzenesulfonyl chloride (3.00 g, 13.54 mmol) and pyrrolidine (2.12 g, 2.45 ml, 29.81 mmol). Yellow crystals (2.95 g, 85%); R_F = 0.44 (CH_2Cl_2/*n*-hexane, 2:1 *v*/*v*); m.p. 81.7–81.9 °C. IR (Bruker, ATR, ν, cm^{-1}): 3080 (aryl C—H str.), 2968 (sp^3-C—H str.), 1597 (aryl C═C str.), 1543 (asym $C—NO_2$ str.), 1344 (sym $C—NO_2$ str.), 1342 (asym $SO_2—N$ str.), 1163 (sym $SO_2—N$ str.), 1078 (C—N str.). ^1H NMR (Bruker, 400 MHz, $CDCl_3$, δ_H, ppm): 7.94 (1H, *d*, *J* = 8 Hz, Ar**H**), 7.62 (2H, *t*, *J* = 4 Hz, Ar**H**), 7.54 (1H, *d*, *J* = 8 Hz, Ar**H**), 3.37–3.35 (4H, *m*, –**CH₂**N**CH₂**–), 1.85 (4H, *m*, –**CH₂CH₂**–). ^{13}C NMR (Bruker, 100 MHz, $CDCl_3$, δ_C, ppm): 148.4, 133.5, 132.1, 131.5, 130.6, 123.9 (Ar**H**), 48.2 (–**CH₂**N**CH₂**–), 25.9 (–**CH₂CH₂**–).

*2.2.1.2. N-Piperidinyl-o-nitrobenzenesulfonamide, **2**.* *o*-Nitrobenzenesulfonyl chloride (5.00 g, 22.57 mmol) and piperidine (3.84 g, 4.45 ml, 45.1 mmol). Yellow crystals (4.97 g, 81.5%); R_F = 0.56 (CH_2Cl_2/*n*-hexane, 2:1 *v*/*v*); m.p. 91.6–91.8 °C. IR (Bruker, ATR, ν, cm^{-1}): 3076 (aryl C—H str.), 2947 (sp^3-C—H str.), 1552 (aryl C═C str.), 1550 (asym $C—NO_2$ str.), 1354 (sym $C—NO_2$ str.), 1350 (asym $SO_2—N$ str.), 1166 (sym $SO_2—N$ str.), 1056 (C—N str.). ^1H NMR (Bruker, 400 MHz, $CDCl_3$, δ_H, ppm): 7.96 (1H, *d*, *J* = 4 Hz, Ar**H**), 7.70 (2H, *t*, *J* = 4 Hz, Ar**H**), 7.59 (1H, *d*, *J* = 8 Hz, Ar**H**), 3.26–3.24 (4H, *m*, –**CH₂**N**CH₂**–), 1.64–1.63 (4H, *m*, –**CH₂**CH₂**CH₂**–), 1.55–1.54 (2H, *m*, –CH₂**CH₂**CH₂–). ^{13}C NMR (Bruker, 100 MHz, $CDCl_3$, δ_C, ppm): 148.5, 133.6, 131.6, 131.5, 130.8, 123.5 (Ar**H**), 47.0 (–**CH₂**N**C**H₂–), 25.4 (–**C**H₂CH₂**C**H₂–), 23.5 (–CH₂**C**H₂CH₂–).

*2.2.1.3. N-Indolinyl-o-nitrobenzenesulfonamide, **3**.* *o*-Nitrobenzenesulfonyl chloride (3.00 g, 13.54 mmol) and indoline (3.55 g, 3.34 ml, 29.79 mmol). Yellow crystals (3.11 g, 75.5%); R_F = 0.79 (CH_2Cl_2/*n*-hexane, 2:1 *v*/*v*); m.p. 106.5–106.8 °C. IR (Bruker, ATR, ν, cm^{-1}): 3077 (aryl C—H str.), 2976 (sp^3-C—H str.), 1594 (aryl C═C str.), 1536 (asym $C—NO_2$ str.), 1356 (sym $C—NO_2$ str.), 1355 (asym $SO_2—N$ str.), 1161 (sym $SO_2—N$ str.), 1051 (C—N str.). ^1H NMR (Bruker, 400 MHz, $CDCl_3$, δ_H, ppm): 7.95 (1H, *d*, *J* = 8 Hz, Ar**H**), 7.71 (1H, *t*, *J* = 8 Hz, Ar**H**), 7.62 (2H, *t*, *J* = 8 Hz, Ar**H**), 7.48 (1H, *d*, *J* = 8 Hz, Ar**H**), 7.21 (2H, *t*, *J* = 8 Hz, Ar**H**), 7.05 (1H, *t*, *J* = 8 Hz, Ar**H**), 4.17 (2H, *t*, *J* = 8 Hz, –N**CH₂**–), 3.10 (2H, *t*, *J* = 8 Hz, –NCH₂**CH₂**–). ^{13}C NMR (Bruker, 100 MHz, $CDCl_3$, δ_C, ppm): 148.4, 141.1, 134.1, 131.8, 131.7, 131.6, 130.2, 127.8, 125.5, 124.3, 124.2, 114.5 (**Ar**H), 50.5 (–N**C**H₂–), 28.0 (–NCH₂**C**H₂–).

2.2.2. *N*-Cycloamino-*o*-sulfanilamides 4–6. An evacuated nitrogen-gas-filled round-bottomed flask was charged with *N*-cycloamino-*o*-nitrobenzenesulfonamides **1**–**3** (15.63 mmol) dissolved in ethanol (30 ml), at ambient temperature, and 10% palladium-on-charcoal catalyst (3.35 mol%) was added, with stirring. Hydrogen gas was then introduced *via* a balloon and stirring continued at ambient temperature for 12 h. The reaction mixture was filtered and the solvent was evaporated *in vacuo*. The resulting residue was extracted into dichloromethane (50 ml), dried over anhydrous sodium sulfate, filtered and concentrated under reduced pressure to afford an oil, which was purified on a silica-gel column using dichloromethane and *n*-hexane (2:1 *v*/*v*). Crystals were obtained *via* slow solvent evaporation of the eluates at ambient temperature.

*2.2.2.1. N-Pyrrolidinyl-o-sulfanilamide, **4**.* *N*-Pyrrolidinyl-*o*-nitrobenzenesulfonamide **1** (4.00 g, 15.63 mmol) with 10% palladium-on-charcoal catalyst (0.56 g, 5.26 mmol). Off-white crystals (2.90 g, 82%); R_F = 0.40 (CH_2Cl_2/*n*-hexane, 2:1 *v*/*v*); m.p. 75.2–75.4 °C. IR (Bruker, ATR, ν, cm^{-1}): 3464, 3363 (N—H str.), 3003 (aryl C—H str.), 2947 (sp^3-C—H str.), 1620 (aryl C═C str.), 1323 (asym $SO_2—N$ str.), 1132 (sym $SO_2—N$ str.), 1307 (C—N str.). ^1H NMR (Bruker, 400 MHz, $CDCl_3$, δ_H, ppm): 7.63 (1H, *d*, *J* = 8 Hz, Ar**H**), 7.28 (1H, *t*, *J* = 8 Hz, Ar**H**), 6.74 (2H, *d*, *J* = 8 Hz, Ar**H**), 5.13 (2H, *s*, NH), 3.31 (4H, *m*, –**CH₂**N**CH₂**–), 1.80 (4H, *m*, –**CH₂CH₂**–). ^{13}C NMR (Bruker, 100 MHz, $CDCl_3$, δ_C, ppm): 146.4, 134.0, 130.2, 119.1, 117.6, 117.1 (**Ar**H), 47.8 (–**C**H₂N**C**H₂–), 25.2 (–**C**H₂**C**H₂–).

*2.2.2.2. N-Piperidinyl-o-sulfanilamide, **5**.* *N*-Piperidinyl-*o*-nitrobenzenesulfonamide **2** (4.00 g, 14.81 mmol) with 10% palladium-on-charcoal catalyst (0.53 g, 4.98 mmol). Off-white crystals (3.06 g, 86%); R_F = 0.57 (CH_2Cl_2/*n*-hexane, 2:1 *v*/*v*); m.p. 76.6–76.8 °C. IR (Bruker, ATR, ν, cm^{-1}): 3487; 3383 (N—H str.), 3072 (aryl C—H str.), 2947 (sp^3-C—H str.), 1606 (aryl C═C str.), 1309 (asym $SO_2—N$ str.), 1136 (sym $SO_2—N$ str.), 1336 (C—N str.). ^1H NMR (Bruker, 400 MHz, $CDCl_3$, δ_H, ppm): 7.48 (1H, *d*, *J* = 8 Hz, Ar**H**), 7.21 (1H, *t*, *J* = 8 Hz, Ar**H**), 6.67 (1H, *d*, *J* = 8 Hz, Ar**H**), 6.64 (1H, *d*, *J* = 8 Hz, Ar**H**), 4.99 (2H, *s*, NH), 3.03–3.00 (4H, *m*, $–\mathbf{CH_2}\mathrm{N}\mathbf{CH_2}–$), 1.56–1.53 (4H, *m*, $–\mathbf{CH_2}\mathrm{CH_2}\mathbf{CH_2}–$), 1.39–1.37 (2H, *m*, $–\mathrm{CH_2}\mathbf{CH_2}\mathrm{CH_2}–$). ^{13}C NMR (Bruker, 100 MHz, $CDCl_3$, δ_C, ppm): 146.3, 134.0, 130.1, 118.0, 117.6, 117.0 (**Ar**H), 46.8 ($–\mathrm{CH_2N}\mathbf{C}\mathrm{H_2}–$), 25.2 ($–\mathbf{C}\mathrm{H_2CH_2}\mathbf{C}\mathrm{H_2}–$), 23.6 ($–\mathrm{CH_2}\mathbf{C}\mathrm{H_2CH_2}–$).

*2.2.2.3. N-Indolinyl-o-sulfanilamide, **6**.* *N*-Indolinyl-*o*-nitrobenzenesulfonamide **3** (2.50 g, 8.22 mmol) with 10% palladium-on-charcoal catalyst (0.29 g, 2.73 mmol). Off-white crystals (1.69 g, 75%); R_F = 0.80 (CH_2Cl_2/*n*-hexane, 2:1 *v*/*v*); m.p.: 111.9–112 °C. IR (Bruker, ATR, ν, cm^{-1}): 3448, 3367 (N—H str.), 3070 (aryl C—H str.), 2924 (sp^3-C—H str.), 1597 (aryl C═C str.), 1327 (asym SO_2—N str.), 1138 (sym SO_2—N str.), 1330 (C—N str.). ^{1}H NMR (Bruker, 400 MHz, $CDCl_3$, δ_H, ppm): 7.48 (2H, *d*, *J* = 8 Hz, Ar**H**), 7.16 (1H, *t*, *J* = 8 Hz, Ar**H**), 7.08 (1H, *t*, *J* = 6 Hz, Ar**H**), 7.03 (1H, *d*, *J* = 8 Hz, Ar**H**), 6.90 (1H, *t*, *J* = 8 Hz, Ar**H**), 6.58 (1H, *d*, *J* = 8 Hz, Ar**H**), 6.55 (1H, *d*, *J* = 8 Hz, Ar**H**), 5.00 (2H, *s*, NH), 3.96 (2H, *t*, *J* = 8 Hz, $–\mathrm{N}\mathbf{CH_2}–$), 2.86 (2H, *t*, *J* = 8 Hz, $–\mathrm{NCH_2}\mathbf{CH_2}–$). ^{13}C NMR (Bruker, 100 MHz, $CDCl_3$, δ_C, ppm): 146.4, 142.3, 134.4, 132.1, 129.8, 127.6, 125.1, 123.7, 119.4, 117.7, 117.3, 115.1 (**Ar**H), 50.0 ($–\mathrm{N}\mathbf{C}\mathrm{H_2}–$), 28.1 ($–\mathrm{NCH_2}\mathbf{C}\mathrm{H_2}$). Reaction synthesis of nitrosulfonamides **1**–**3** and aminosulfonamides **4**–**6** are presented in Scheme S1 in the supporting information. The FT–IR, MS and ^{1}H/^{13}C NMR spectra of compounds **1**–**6** are also presented in the supporting information.

2.3. Docking studies

2.3.1. Preparation of the ligands for docking. The X-ray crystal structures of synthesized compounds **1**–**6** (CIF files) were imported directly into the *Schrödinger Suite* (Schrödinger, 2022) for preparation. The reference drugs *N*-(5-sulfamoyl-1,3,4-thiadiazol-2-yl)-2-(thiophen-2-yl)acetamide (**A**) and 5-[1-(naphthalen-1-yl)-1,2,3-triazol-4-yl]thiophene-2-sulfonamide (**B**) were based on chemical structures downloaded from the *PubChem* (https://pubchem.ncbi.nlm.nih.gov/) website in SDF format. **A** and **B** were used as reference compounds because they are natural ligands in the crystalline state of 5fl4 and 4iwz. The *Ligprep* module of the molecular modelling platform of the *Schrödinger Suite* (Schrödinger, 2022) was then used to prepare the imported structures by assigning bond lengths, bond angles, generating possible ionization states at pH 7 using *Epik* and finally to optimize using the OPLS4 force field (Nainwal *et al.*, 2018).

2.3.2. Protein preparation. The protein structures of 4iwz and 5fl4, with resolutions of 1.60 and 1.82 Å, respectively, were downloaded from the Research Collaboratory for Structural Bioinformatics (RCSB) Protein Data Bank (PDB). Retrieved crystal coordinates were prepared in the 'Protein Preparation Wizard' of the *Schrödinger Suite* (Schrödinger, 2022), with default parameters of assigning bond orders, optimizing and minimization using OPLS4. A receptor grid generation module was applied to the prepared proteins by selecting the corresponding cocrystallized ligand to define the binding site. A default parameter for the radii of van der Waals having a scaling factor of 1 Å with a partial charge cut-off of 0.25 Å was used (Panwar & Singh, 2021; Yang *et al.*, 2022).

2.3.3. Molecular docking. Docking calculations were executed in the extra precision (XP) mode of the *Glide* module in the molecular modelling platform of the *Schrödinger Suite* (Schrödinger, 2022). The complexes with the highest negative docking scores have better binding towards the respective proteins 4iwz and 5fl4. Docking calculations of the synthesized *N*-cycloamino derivatives against the hCA II (PDB entry 5fl4) and XII (4iw7) isoforms will provide a selectivity profile that may be interesting for the development of novel anticancer agents with limited side effects. The hCA II (PDB entry 5fl4) and XII (4iw7) carbonic anhydrase isoforms have recently emerged as excellent targets for the design of novel therapeutic strategies for cancer, due to their involvement in the survival of tumour cells, as well as in the insurgence of resistance to classical anticancer protocols (Milite *et al.*, 2019).

2.4. DFT calculations

Theoretical studies were performed for compounds **1**–**6** whereupon the SC-XRD structures of the compounds were used for optimization and global reactivity descriptor (GRD) calculations. Computational studies and molecular electrostatic potential (MEP) for **1**–**6** were carried out using the *GAUSSIAN16* software package (Frisch *et al.*, 2016), whereas the calculations were performed using the standard hybrid density functional method (B3LYP) with a basis set of the 6-311++G**(p,d) level (Becke, 1993). Optimized molecules were obtained with the *Chemcraft* visualization program (https://www.chemcraftprog.com/).

2.5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. Carbon-bound H atoms were added in idealized geometrical positions in a riding model. Nitrogen-bound H atoms were located in a difference map and refined freely.

3. Results and discussion

3.1. Chemistry

The *N*-cycloamino-*o*-sulfanilamides **4**–**6** were prepared *via* a two-step reaction, starting from the condensation reaction of *o*-nitrobenzenesulfonyl chloride with alicyclic amines in toluene, at ambient temperature, to afford *N*-cycloamino-*o*-nitrobenzenesulfonamide adducts **1**–**3** (Scheme S1 in the supporting information). The use of toluene as a nonpolar reaction medium was, amongst other reasons, to drive the forward reaction. In the second step, adducts **1**–**3** were hydrogenated with hydrogen gas, in ethanol at ambient temperature, in the presence of 10% palladium-on-activated charcoal catalyst to give the target *N*-cycloamino-*o*-sulfanilamides **4**–**6** in 72–86% yield. The reactions were monitored by TLC.

Table 1
Experimental details.

Experiments were carried out with Mo *Kα* radiation using a Bruker APEXII CCD diffractometer. Absorption was corrected for by numerical methods (*SADABS*; Bruker, 2008).

	1	**2**	**3**
Crystal data			
Chemical formula	$C_{10}H_{12}N_2O_4S$	$C_{11}H_{14}N_2O_4S$	$C_{14}H_{12}N_2O_4S$
M_r	256.28	270.30	304.32
Crystal system, space group	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/n$
Temperature (K)	200	296	296
a, b, c (Å)	8.6173 (5), 14.6662 (9), 9.4885 (6)	8.6881 (9), 15.0266 (14), 9.8337 (10)	7.4701 (5), 23.6743 (12), 7.8614 (5)
α, β, γ (°)	90, 108.075 (3), 90	90, 106.526 (4), 90	90, 94.989 (3), 90
V (Å^3)	1140.01 (12)	1230.8 (2)	1385.02 (15)
Z	4	4	4
μ (mm^{-1})	0.29	0.27	0.25
Crystal size (mm)	0.67 × 0.67 × 0.12	0.48 × 0.47 × 0.45	0.62 × 0.51 × 0.43
Data collection			
$T_{\min}$, $T_{\max}$	0.934, 1.000	0.941, 1.000	0.913, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	24329, 2849, 2508	25328, 3059, 2662	28400, 3434, 2864
$R_{\rm int}$	0.018	0.017	0.024
$(\sin \theta/\lambda)_{\max}$ (Å^{-1})	0.669	0.668	0.669
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.041, 0.111, 1.05	0.036, 0.106, 1.06	0.040, 0.105, 1.05
No. of reflections	2849	3059	3434
No. of parameters	149	163	190
No. of restraints	20	0	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å^{-3})	0.43, −0.47	0.32, −0.31	0.26, −0.29

	4	**5**	**6**
Crystal data			
Chemical formula	$C_{10}H_{14}N_2O_2S$	$C_{11}H_{16}N_2O_2S$	$C_{14}H_{14}N_2O_2S$
M_r	226.29	240.32	274.33
Crystal system, space group	Monoclinic, $P2_1/c$	Orthorhombic, $Pbca$	Monoclinic, $P2_1/n$
Temperature (K)	296	200	296
a, b, c (Å)	16.399 (3), 7.9485 (12), 18.376 (3)	11.1747 (4), 10.4850 (4), 20.1368 (8)	9.7990 (11), 10.2612 (13), 13.2010 (16)
α, β, γ (°)	90, 113.907 (6), 90	90, 90, 90	90, 100.682 (5), 90
V (Å^3)	2189.7 (6)	2359.36 (15)	1304.4 (3)
Z	8	8	4
μ (mm^{-1})	0.28	0.26	0.25
Crystal size (mm)	0.47 × 0.32 × 0.20	0.49 × 0.26 × 0.25	0.54 × 0.34 × 0.34
Data collection			
$T_{\min}$, $T_{\max}$	0.927, 1.000	0.944, 1.000	0.925, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	55955, 5477, 4489	57075, 2935, 2583	28600, 3275, 2726
$R_{\rm int}$	0.032	0.020	0.031
$(\sin \theta/\lambda)_{\max}$ (Å^{-1})	0.670	0.668	0.675
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.035, 0.107, 1.04	0.029, 0.087, 1.05	0.039, 0.113, 1.05
No. of reflections	5477	2935	3275
No. of parameters	287	153	180
No. of restraints	0	0	0
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å^{-3})	0.33, −0.36	0.34, −0.36	0.27, −0.43

Computer programs: *APEX2* (Bruker, 2011), *SAINT* (Bruker, 2012), *SHELXT2018* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *ShelXle* (Hübschle *et al.*, 2011), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2020) and *Mercury* (Macrae *et al.*, 2020).

All the compounds synthesized were characterized by their melting points and IR, ^{1}H/^{13}C NMR and MS spectra. In the IR spectra of *o*-nitrosulfonamide adducts **1**–**3**, the strong absorption bands observed at 1355–1342 and 1171–1161 cm^{-1} were ascribed to the asymmetric and symmetric stretching frequencies, respectively, of the SO_2—N moiety, thereby

Table 2
Hydrogen-bond, C—H···(π ring) and π–π stacking interaction geometry (Å, °) for the crystal structures of *p*-sulfanilamide (**B**), *o*-sulfanilamide (**D**), *o*-nitrosulfonamides **1–3** and *N*-cycloamino-*o*-sulfanilamides **4–6**.

Compound	Interaction	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*	π–π
o-Nitrosulfonamides						
1	C11—H11*A*···O1^{i}	0.99	2.56	3.398 (7)	143	
	C11—H11*B*···O4	0.99	2.48	3.093 (9)	120	
	C25—H25···O4^{ii}	0.95	2.54	3.240 (2)	131	
	C25—H25···O1^{iii}	0.95	2.47	3.229 (2)	137	
	C26—H26···O2	0.95	2.49	2.860 (2)	103	
	C26—H26···O3^{iv}	0.95	2.56	3.467 (2)	160	
2	C11—H11*A*···O2	0.97	2.48	2.907 (2)	107	
	C11—H11*A*···O2^{i}	0.97	2.59	3.476 (2)	152	
	C15—H15*B*···O1	0.97	2.51	2.943 (2)	107	
	C25—H25···O2^{v}	0.93	2.57	3.334 (2)	140	
	C26—H26···O1	0.93	2.53	2.877 (2)	103	
3	C16—H16···O2	0.93	2.41	2.975 (2)	119	
	C16—H16···O2^{vi}	0.93	2.55	3.195 (2)	127	
	C23—H23···O1^{vii}	0.93	2.30	3.086 (3)	142	
	C26—H26···N1	0.93	2.60	2.983 (2)	105	
	*Cg*1···*Cg*2^{i}					3.6967 (11)
	*Cg*2···*Cg*1^{i}					3.6968 (11)
N-Cycloamino-*o*-sulfanilamides						
4	N12—H12*C*···O21	0.78 (3)	2.40 (3)	3.121 (3)	155 (3)	
	N12—H12*D*···O11	0.84 (3)	2.10 (3)	2.776 (3)	138 (3)	
	N22—H21*C*···O11^{i}	0.82 (3)	2.30 (3)	3.106 (2)	168 (2)	
	N22—H21*D*···O21	0.87 (2)	2.07 (2)	2.783 (2)	139 (2)	
	C114—H114···O22^{viii}	0.93	2.59	3.391 (3)	144	
	C116—H116···O12	0.93	2.46	2.851 (2)	105	
	C216—H216···O22′	0.93	2.52	2.897 (2)	105	
5	N1—H1*A*···O1^{ix}	0.836 (19)	2.496 (19)	3.2999 (16)	161.8 (16)	
	N1—H1*B*···O2	0.852 (16)	2.156 (16)	2.8240 (17)	135.1 (14)	
	C16—H16···O1	0.95	2.48	2.8774 (15)	105	
	C16—H16···O2^{x}	0.95	2.48	3.2577 (15)	139	
	C21—H21*A*···O1	0.99	2.55	2.9635 (15)	105	
	C25—H25*B*···O2	0.99	2.44	2.8675 (16)	106	
6	N2—H2*C*···O2	0.86 (3)	2.19 (3)	2.857 (2)	134 (2)	
	N2—H2*D*···O1^{xi}	0.89 (2)	2.22 (2)	3.098 (2)	168.8 (19)	
	C15—H15···O2^{xii}	0.93	2.59	3.366 (2)	141	
	C16—H16···O1	0.93	2.58	3.105 (2)	117	
	C26—H26···O1	0.93	2.45	2.849 (2)	106	
	C1—H1*B*···*Cg*1^{xiii}	0.97	2.97	3.817 (2)	147	
	S1—O2···*Cg*2^{i}			3.5773 (15)		

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+\frac{3}{2}, y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (iv) $x+\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (v) $x-\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (vi) $-x+2, -y+1, -z+1$; (vii) $x+\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (viii) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (ix) $x-\frac{1}{2}, y, -z+\frac{1}{2}$; (x) $-x+\frac{3}{2}, y-\frac{1}{2}, z$; (xi) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (xii) $x-\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$. Centroids for **3**: *Cg*1 N1/C1/C2/C12/C11; *Cg*2 C11–C16; *Cg*3 C21–C26; for **4**: *Cg*1 C22–C27; for **5** *Cg*1 C11–C16; *Cg*2 N1/C2/C1/C12/C11; for **6** *Cg*1 C21–C26.

alluding to the formation of the sulfonamide bond. The disappearances of the SO_2—Cl (1420 and 1220 cm^{-1}) and N—H (3286–3265 cm^{-1}) stretching bands in the IR spectra of *o*-nitrobenzenesulfonyl chloride and cycloamines, respectively, were good indicators of a successful condensation reaction. This was corroborated by the shift of the sulfonyl (–SO_2–) absorption bands from 1420 and 1220 (in *o*-nitrobenzenesulfonyl chloride) to 1355–1342 and 1171–1161 cm^{-1} (in **1–3**). It is noteworthy that the lower wavenumbers observed in the IR spectra of *o*-nitrosulfonamides **1–3** for –SO_2– were not unusual as the Cl atom bonded to it had been replaced by a less electronegative N atom. In the IR spectra of *o*-sulfanilamides **4–6**, the appearance of two N—H stretching bands in the higher frequency region around 3467 ± 20 and 3383 ± 10 cm^{-1}, and the disappearance of the nitro (NO_2) absorption bands (observed at 1550–1536 and 1369–1342 cm^{-1}) in the spectra of **1–3** were attributed to the successful catalytic reduction of the nitro group to the amino group.

The ^{1}H NMR spectra of *o*-nitrosulfonamides **1–3** were additive of the individual spectra of the starting materials (*i.e.* *o*-nitrobenzenesulfonyl chloride and cycloamines), with the disappearance of the nitrogen proton peaks of cycloamines. The aromatic protons of *o*-sulfanilamides **4–6** resonated upfield in comparison to the same aromatic protons in precursors **1–3**. This general shift towards tetramethylsilane (TMS) was credited to the newly formed amino groups whose lone-pair electrons are suspected of having caused the increased mesomeric shielding of the aromatic protons. D_2O-exchangeable singlets were also observed in the ^{1}H NMR spectra of **4–6** between 5.13 and 4.99 ppm for the newly-formed amino protons. The success of the catalytic hydrogenation of nitro adducts **1–3** was corroborated by the ^{13}C NMR spectra of **4–6**, where the requisite C atoms ($C—NO_2 \rightarrow C—NH_2$) resonated upfield in the range 133.9–130.1 ppm. The spectroscopic data analyses of the synthesized compounds were consistent with the assigned structures of the compounds.

3.2. Crystal structure

The molecules of **1–3** and **4–6** crystallized in the monoclinic space group $P2_1/n$ or $P2_1/c$ (No. 14), except for **5**, which crystallized in the orthorhombic space group *Pbca* (No. 61). In addition, they all had one molecule in the asymmetric unit, with the exception of **4**, with two independent molecules per asymmetric unit cell. The two molecules per unit cell of compound **4** were identical but for the conformation of the pyrrolidine group (*cf.* Fig. S1 in the supporting information). It is noteworthy that the pyrrolidine ring in **1** is disordered. The molecular structures of **1–3** and **4–6** are shown in Fig. 2, while the crystal data collection parameters of *o*-nitrosulfonamides **1–3** and *N*-cycloamino-*o*-sulfanilamides **4–6** are presented in Table 1. They are compared with the crystal structure data of *para*-sulfanilamide and *ortho*-sulfanilamide, which crystallize in the orthorhombic *Pbca* (No. 61) and monoclinic $P2_1/c$ (No. 14) space groups, respectively (Gelbrich *et al.*, 2008; Shad *et al.*, 2008). Several sulfonamide derivatives have also been reported (El-Gaby *et al.*, 2020). Selected bond lengths and angles, as determined from the SC-XRD experiments, are collected in Table S1 (see supporting information).

It is instructive to note that the amino (NH_2) group in *N*-cycloamino-*o*-sulfanilamides **4–6** contributed significantly to their hydrogen-bond interactions (*cf.* Table 2). In all three structures, there were intramolecular N—H···O═S interactions resulting in ring closures that can be described with *S*(6) graph-set descriptors (Bernstein *et al.*, 1995; Etter *et al.*, 1990). Furthermore, compounds **5** and **6** exhibited infinite-chain intermolecular N—H···O═S interactions with *C*(6) descriptors. Interestingly, no infinite chain interaction was observed in **4**; instead, four molecules were linked into a ring structure with an $R_4^4(24)$ descriptor. The *p*-sulfanilamide (Gelbrich *et al.*, 2008) and *o*-sulfanilamide (Shad *et al.*, 2008) structures also each have a number of infinite-chain interactions and ring structures. Fig. 3 shows selected hydrogen-bond, C—H···(π ring) and π–π stacking interactions for sulfonamides **1–3** and sulfanilamides **4–6**. All the hydrogen bonds were of moderate (mostly electrostatic) strength (Jeffrey, 1997), with **4** giving the strongest hydrogen bonds (Table 2). Additionally, the compounds also exhibited both intra- and intermolecular C—H···O═S interactions, with the length of the shortest interactions varying in the range 2.30–2.48 Å.

Table 3
Percentage contributions of selected interatomic contacts to the Hirshfeld surface of compounds **1–6**.

	C···H	O···H	N···H
o-Nitrosulfonamides			
1	15.2	42.3	0.6
2	15.3	41.7	1.4
3	14.0	40.3	0.0
N-Cycloamino-*o*-sulfanilamides			
4	15.2	25.9	3.5
5	16.5	22.4	3.4
6	30.4	19.6	2.1

The only π–π stacking interaction of note occurred in **3**, where two centroid-to-centroid interactions with distances of 3.6967 (11) Å were observed between the centrosymmetric indoline moieties. An N═O···π ring interaction of 3.657 (2) Å was also evident in **3**, whereas intermolecular C—H···(π ring) interactions of 2.97 Å and S═O···(π ring) interactions of 3.5773 (15) Å were present in the structure of its hydrogenated analogue **6**. The packing diagrams of the crystal structures of compounds **1–6** are shown in Fig. S2 in the supporting information.

3.3. Hirshfeld surface analysis

The Hirshfeld surface analyses (Turner *et al.*, 2017) of compounds **1–6** showed intermolecular interactions such as O—H···O, O—H···N and C—H···π. Two sharp O—H spikes

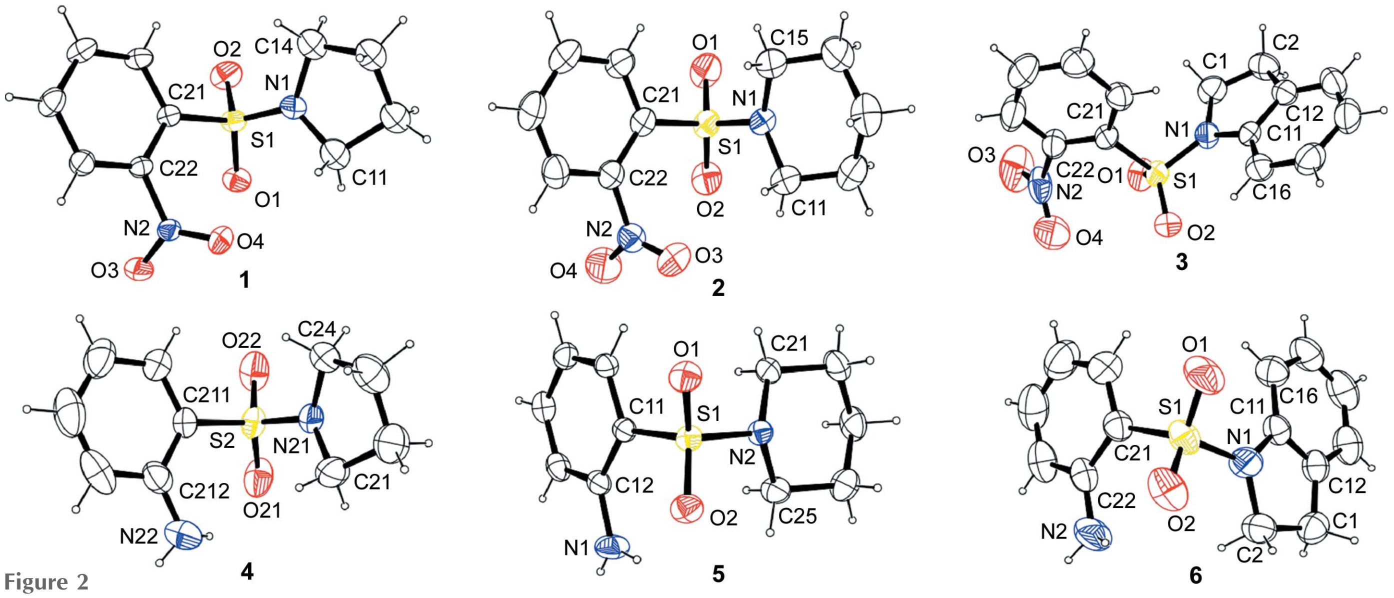


Figure 2
The molecular structures of *o*-nitrosulfonamides **1–3** and *N*-cycloamino-*o*-sulfanilamides **4–6** (molecule 2 of **4** shown). Displacement ellipsoids are drawn at the 50% probability level. Minor disorder components have been omitted.

Table 4
Surface interactions of *o*-nitrosulfonamides **1–3** and *N*-cycloamino-*o*-sulfanilamides **4–6**.

Compound	d_{norm}		d_{i}		d_{e}	
	Minimum value	Maximum value	Minimum value	Maximum value	Minimum value	Maximum value
1	−0.2565	0.9743	0.9092	2.3849	0.9083	2.4152
2	−0.1071	1.0914	1.0665	2.4390	1.0669	2.4804
3	−0.3223	1.7007	0.9319	2.6809	0.9322	2.6406
4	−0.3784	1.2864	0.8873	2.4989	0.9319	2.4948
5	−0.2060	1.3595	0.9944	2.6968	0.9957	2.5141
6	−0.3866	1.2989	0.8842	2.7791	0.8838	2.5779

typical of an O—H···O interaction from **1** contributed the highest O···H interaction of 42.3%. The fingerprint plots showed that C···H contacts were highest for **6** (30.4%), and this is closely related to C—H···π interactions (McKinnon *et al.*, 2007; Kolade *et al.*, 2020). The percentages of the major contributions, *e.g.* C···H, O···H and N···H interatomic contacts, for each molecule are compiled in Table 3.

The molecular Hirshfeld surfaces, mapped as d_{norm}, shape index and curvedness, confirmed interactions between neighbouring molecules of **1–6** and are presented in Fig. S3. The

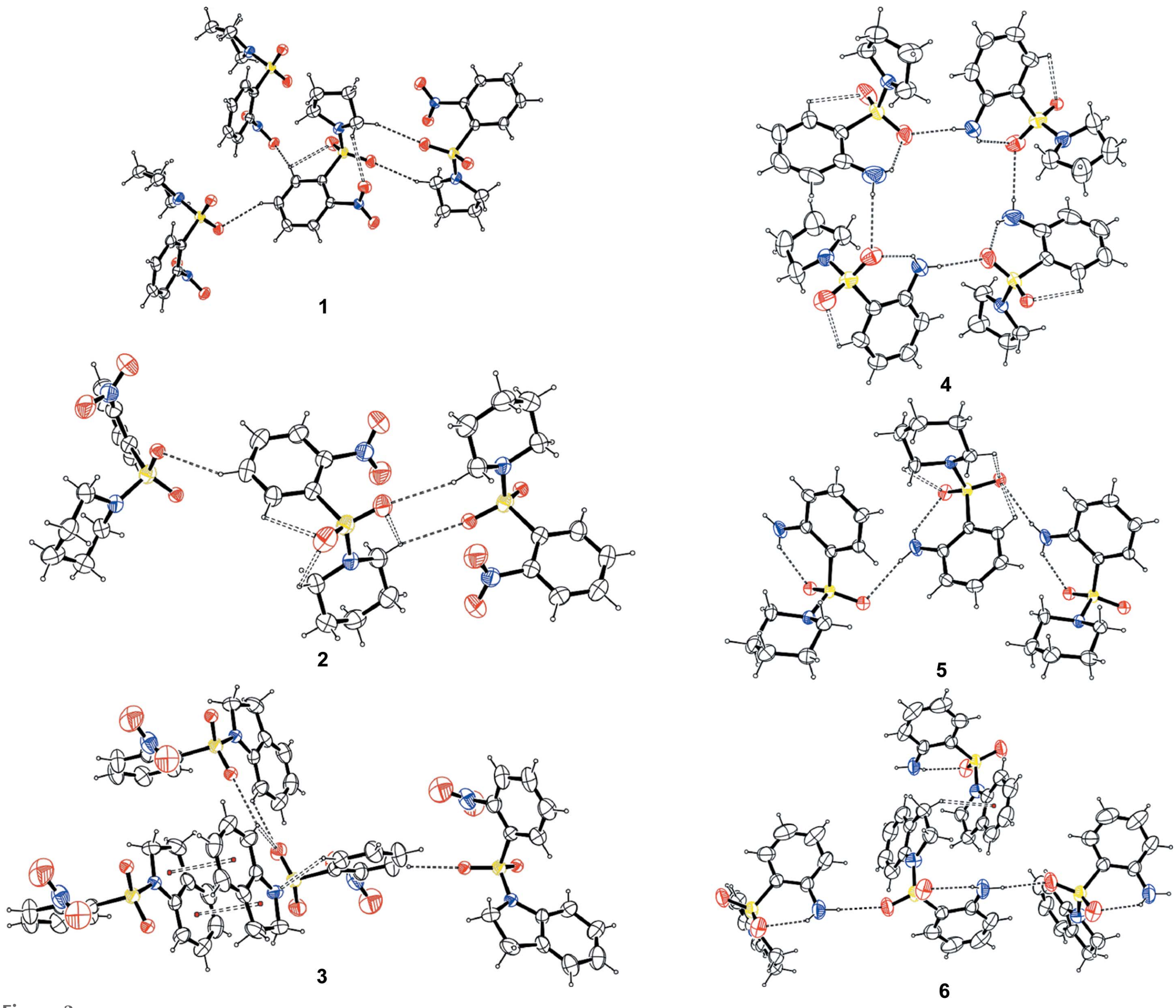


Figure 3
Selected hydrogen-bond, C—H···(π ring) and π–π stacking interactions for compounds **1–6**. Displacement ellipsoids are drawn at the 50% probability level. Minor disorder components have been omitted.

Table 5
Frontier molecular orbital (FMO) energies of synthesized compounds **1**–**6**.

Parameter (eV)	*o*-Nitrosulfonamides			*N*-Cycloamino-*o*-sulfanilamides		
	1	**2**	**3**	**4**	**5**	**6**
HOMO energy ($E_{\rm HOMO}$)	−7.228	−7.386	−6.415	−6.189	−6.211	−6.142
LUMO energy ($E_{\rm LUMO}$)	−2.982	−2.973	−3.175	−1.627	−1.287	−1.451
ΔE gap	4.246	4.413	3.24	4.562	4.924	4.691
Ionization potential (I)	7.228	7.386	6.415	6.189	6.211	6.142
Electron affinity (A)	2.982	2.973	3.175	1.627	1.287	1.451
Chemical potential (μ)	−5.105	−5.1795	−4.795	−3.908	−3.749	−3.7965
Electronegativity (χ)	5.105	5.1795	4.795	3.908	3.749	3.7965
Global hardness (η)	2.123	2.2065	1.62	2.281	2.462	2.3455
Global softness (S)	0.471	0.453	0.617	0.438	0.406	0.426
Global electrophilicity (ω)	27.664	29.597	18.624	17.418	17.301	16.903

large circular depressions (deep red) visible on the $d_{\rm norm}$ surfaces typically indicate that the molecule has a donor site(s) (*e.g.* amine and/or sulfone) or interactions with proteins.

Fingerprint plots of *o*-nitrosulfonamides **1**–**3** and *N*-cycloamino-*o*-sulfanilamides **4**–**6** in full and resolved into C···H, O···H and N···H are presented in Fig. S4 (supporting information). The intermolecular O···H and N···H interactions appear as two distinct spikes of almost equal length in the 2D (two-dimensional) fingerprint plots in the region $1.2 < (d_{\rm e} + d_{\rm i}) < 2.9$ Å as light-sky-blue patterns in full fingerprint 2D plots and characterized to be 2.56 ± 0.21 Å corresponds to O···H contacts which contributes the majority of the surface area. 2D fingerprint plots reveal the contributions of these interactions in the crystal structure quantitatively and are presented in Table 4 (with minimum and maximum values of $d_{\rm norm}$, $d_{\rm i}$ and $d_{\rm e}$ provided). Complementary regions are also visible in the fingerprint plots (Fig. S4), where one molecule acts as a donor ($d_{\rm e} > d_{\rm i}$) and the other acts as an acceptor ($d_{\rm e} < d_{\rm i}$). This finding was validated by the calculated molecular electrostatic potential of **1**–**6** (Fig. S5). The negative potential (acceptor) is indicated as a red surface around the O atoms attached to sulfur (–SO_2) and the N atoms attached to oxygen (–NO_2). The blue/purple surface area indicates that the positive potential (donor) is mapped in the proximity of the H atoms (Fig. S5).

3.4. Global reactivity descriptors (GRDs)

The full geometry of optimized molecules **1**–**6** presented bond lengths similar to those obtained from the crystal data. A comparison of selected torsion angles of the crystal structures of **1**–**6** and the DFT-optimized molecules showed that con-

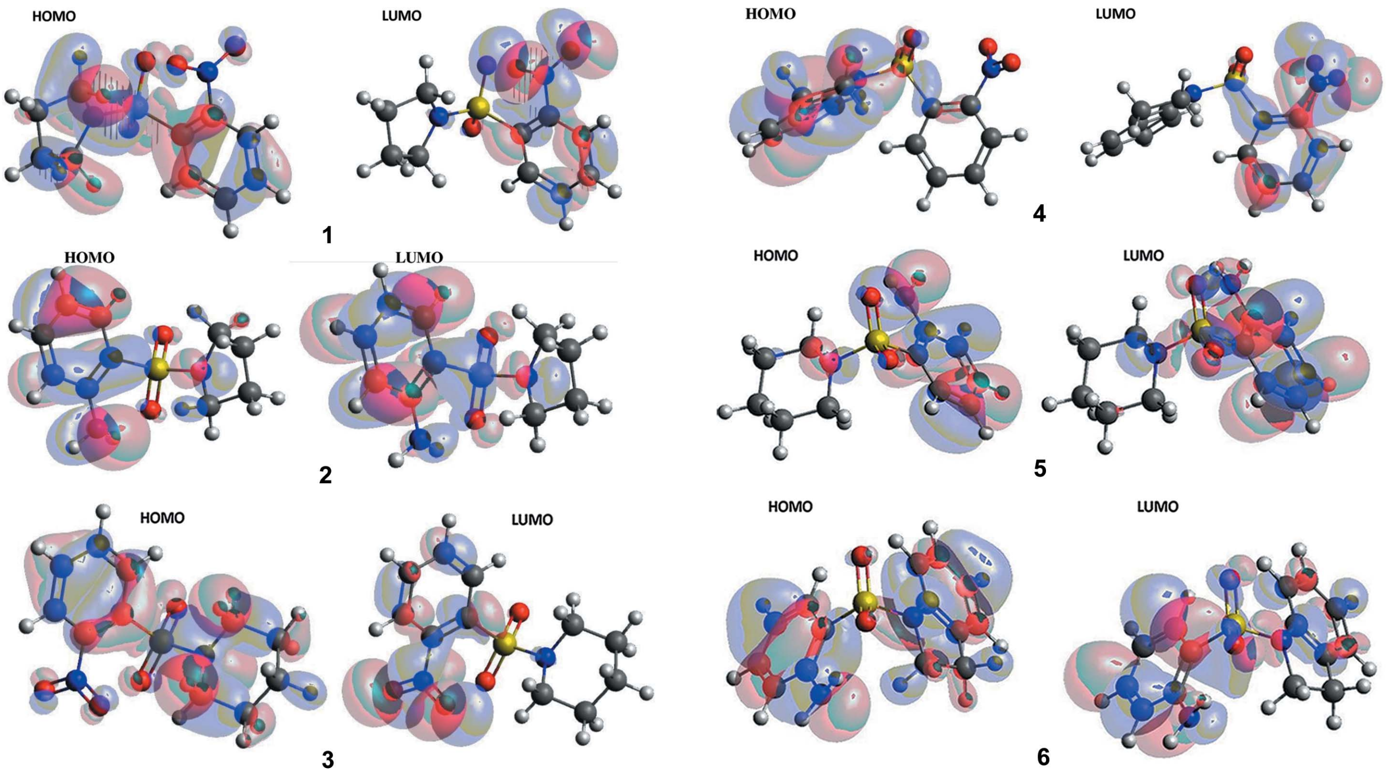


Figure 4
Frontier molecular orbitals for the optimized structures of **1**–**6**.

Table 6
Energies of *o*-nitrosulfonamides **1**–**3** and *N*-cycloamino-*o*-sulfanilamides **4**–**6** with the hCA II (PDB entry 4iwz) and hCA IX (5fl4) isoenzymes.

Entry	Docking score		E-model		Ligand efficiency	
	4iwz	5fl4	4iwz	5fl4	4iwz	5fl4
1	−1.351	−0.807	−38.102	−40.980	−0.079	−0.047
2	−2.223	−1.977	−41.329	−42.073	−0.124	−0.110
3	−1.288	−1.538	−40.486	−38.954	−0.061	−0.073
4	−1.645	−1.451	−37.113	−35.898	−0.110	−0.097
5	−1.636	−1.605	−41.034	−34.609	−0.102	−0.100
6	−0.784	−1.368	−47.945	−42.454	−0.041	0.072
A	−2.252	–	−55.202	–	−0.125	–
B	–	−1.969	–	−41.029	–	−0.082

Note: **A** is *N*-(5-sulfamoyl-1,3,4-thiadiazol-2-yl)-2-(thiophen-2-yl)acetamide and **B** is 5-[1-(naphthalen-1-yl)-1,2,3-triazol-4-yl]thiophene-2-sulfonamide.

formation of the molecules did not change significantly in the DFT-optimized state (Fig. S6). Generally, the observed, almost flat, O—S—N—C torsion angle of the DFT-optimized molecules suggest that the lone pairs on sulfur may have contributed to the π-electron delocalization that is observed in the DFT molecules.

The highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) electrons are distributed around various moieties within the various molecules (Fig. 4). Generally, electron distribution is mainly scattered in the HOMO over the phenyl, sulfur and indolinyl/pyrrolidinyl rings, with the exception of **3** and **5**. The LUMO is mainly spread over the phenyl moieties. This indicates that there is a transfer of charge between the indolinyl/pyrrolidinyl rings and the phenyl moieties within the molecule.

The HOMO–LUMO gap, which describes the stability of molecules and predicts reactivity between species by providing the electrical transport properties, as well as electron carrier and mobility in molecules (Rathi *et al.*, 2020), are provided in Table 5. *N*-Indolinyl-*o*-nitrobenzenesulfonamide **3** displayed the smallest energy gap (3.24 eV), indicating that it was the softest molecule with good polarizability and reactivity, whereas *N*-piperidinyl-*o*-sulfanilamide **5** presented the largest energy gap of 4.924 eV, thereby corroborating its high chemical hardness of 2.462 eV (*cf.* Table 5). The lowest LUMO energy was obtained from **3** ($E_{\rm LUMO}$ = −3.175 eV), indicating that it is the best electron acceptor of the molecules analyzed, whereas **6** was the best electron donor in the series, with the highest HOMO energy ($E_{\rm HOMO}$) of −6.142 eV (Table 5). The observed large energy gap (4.924 eV) in **5** suggests that charge transfer could promote its bioactivity and ability to form biological interactions at the piperidinyl and phenyl moiety (Al-Wahaibi *et al.*, 2019). Therefore, the predicted order of biological interactions are **5** > **6** > **4** > **2** > **1** > **3**.

The ionization potential (I), electron affinity (A), chemical potential (μ), electronegativity (χ), global hardness (η), global softness (S) and global electrophilicity (ω) values were calculated using the HOMO and LUMO energy values and are collated in Table 5. The lowest I value of 6.142 eV originated from sulfanilamide **6**, whereas sulfonamide **3** gave the largest A value of 3.175 eV. Amongst the compounds studied, **2** gave the highest χ value of 5.1795 eV. Interestingly, sulfanilamide **5** displayed the highest η value of 2.462 eV and the lowest chemical softness (S) of 0.406 eV, thus alluding to its having the most reactive nature of all the molecules investi-

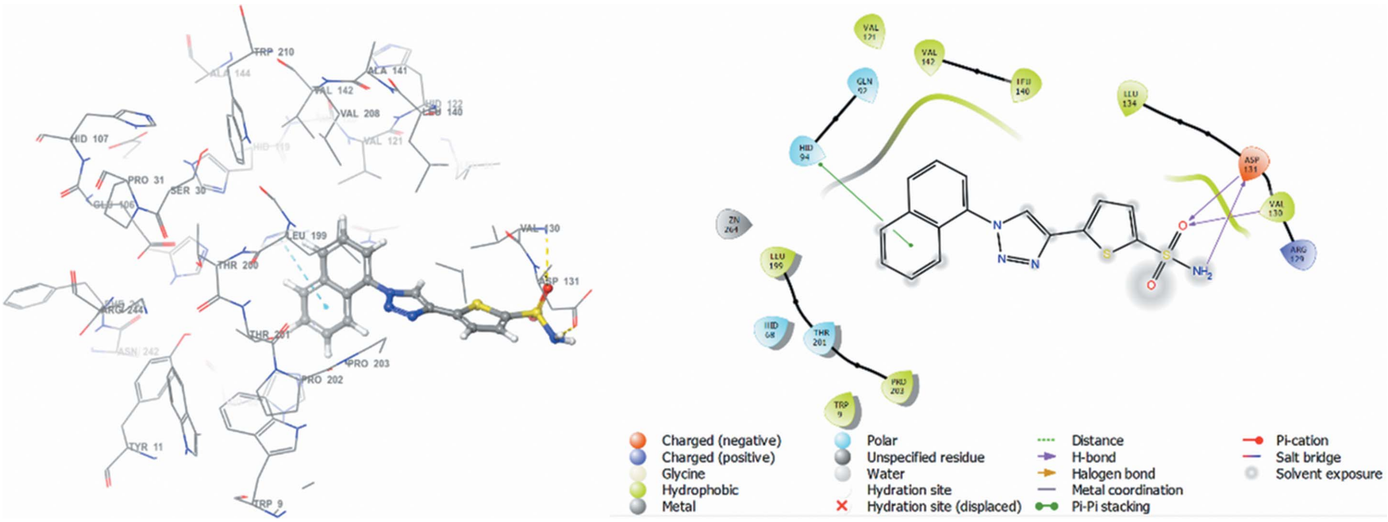


Figure 5
(*a*) 3D interaction diagram of *N*-(5-sulfamoyl-1,3,4-thiadiazol-2-yl)-2-(thiophen-2-yl)acetamide (**A**) and hCA II isoenzyme 4iwz (**A**), and (*b*) 2D interaction diagram depicting the binding residues of 4iwz.

Table 7
Hydrogen-bond and mixed π-interactions (Å) of *o*-nitrosulfonamides **1–3** and *N*-cycloamino-*o*-sulfanilamides **4–6** with the hCA II (PDB entry 4iwz) and hCA IX (5fl4) isoenzymes.

Isoenzyme/Entry	Carbonic anhydrase II (PDB entry 4iwz)		Carbonic anhydrase IX (PDB entry 5fl4)	
	Hydrogen bond	π–π or π–cation	Hydrogen bond	π–π or π–cation
o-Nitrosulfonamides				
1	GLN$_{92}$ (2.13)		GLN$_{92}$ (2.14)	HID$_{94}$ (4.08) π–cation
2	ASN$_{62}$ (2.33), ASN$_{67}$ (2.55)		GLN$_{71}$ (2.28), THR$_{201}$ (2.13)	HID$_{94}$ (4.07) π–cation
3	GLN$_{92}$ (2.25), ASN$_{62}$ (2.74)	TRP$_5$ (5.36) π–π stacking	GLN$_{71}$ (2.26)	HID$_{94}$ (4.05) π–cation
N-Cycloamino-*o*-sulfanilamides				
4	GLN$_{92}$ (2.04), THR$_{199}$ (2.29)		THR$_{201}$ (2.03)	
5	GLN$_{92}$ (1.78), THR$_{199}$ (2.26)			
6	GLN$_{92}$ (1.88), TRP$_5$ (2.11)	THR$_{199}$ (1.81) π–π stacking	GLU92 (2.47)	
A	GLN$_{92}$ (2.39), HIE$_{64}$ (2.24)	HIS$_{94}$ (4.75) π–π stacking		
B			ASP$_{13}$ (1.59–2.73), VAL$_{130}$ (2.53)	HID$_{94}$ (5.49) π–cation

Note: **A** is *N*-(5-sulfamoyl-1,3,4-thiadiazol-2-yl)-2-(thiophen-2-yl)acetamide and **B** is 5-[1-(naphthalen-1-yl)-1,2,3-triazol-4-yl]thiophene-2-sulfonamide.

gated. The highest global electrophilicity of 29.597 eV was also recorded for sulfonamide **2**, indicating that it is a strong electrophile. In general, the chemical reactivities of compounds **1–6** have been shown to vary with the groups attached to the compounds (Abbaz *et al.*, 2018).

3.5. Docking studies

Docking studies of synthesized **1–6** with human carbonic anhydrase II and IX inhibitors (hCA II and IX; PDB entries: 4iwz and 5fl4) (Biswas *et al.*, 2013; Leitans *et al.*, 2015), downloaded from the Research Collaboratory for Structural Bioinformatics (RCSB) Protein Data Bank (PDB) was carried out in *Maestro* (Version 13.1.137, MMshare Version 5.7.137, Release 2022-1, Platform Windows-x64) (Schrödinger, 2022). The binding strengths of the docked complexes were analysed through docking score, glide E-model and ligand efficiency (*cf.* Table 6). These energies define the degree of stability of binding between the respective isoenzymes and target compounds **1–6**. *N*-(5-Sulfamoyl-1,3,4-thiadiazol-2-yl)-2-(thiophen-2-yl)acetamide (**A**) and 5-[1-(naphthalen-1-yl)-1,2,3-triazol-4-yl]thiophene-2-sulfonamide (**B**) were also docked with respective proteins 4iwz and 5fl4, and taken as reference or standard drugs. Docking poses for the synthesized compounds are displayed in Figs. S7–S18, while those for the reference drugs are shown in Figs. 5 and 6.

Docking calculations between 4iwz and **A** (reference drug) displayed a docking score of −2.252 kcal mol^{-1}, which is higher than for all synthesized compounds **1–6** (*cf.* Table 6).

Also, **A** interacted with amino acid residues GLN$_{92}$ (2.39 Å) and HIE$_{64}$ (2.24 Å) *via* hydrogen-bonding interactions and with amino acid residue HIS$_{94}$ (4.75 Å) *via* π–π stacking interactions (*cf.* Table 7). Some bad interactions/contacts were observed between the amino acid residue GLU$_{106}$ and **A** (Fig. 5). Compound **2** displayed the best binding affinity among the synthesized compounds, with a docking score of −2.223 kcal mol^{-1}, slightly lower than that of the reference drug. Sulfanilamides **4** and **5** also displayed significantly good binding affinities, with docking scores of −1.645 and −1.636 kcal mol^{-1}, respectively. Sulfanilamide **6** was char-

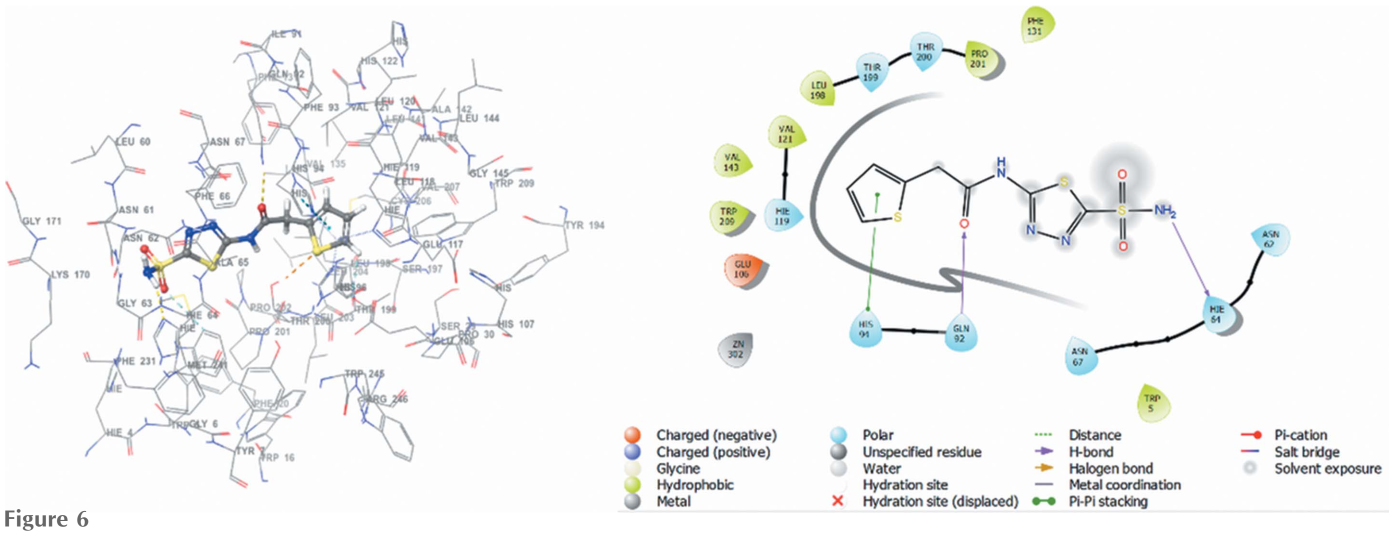


Figure 6
(*a*) 3D interaction diagram of 5-[1-(naphthalen-1-yl)-1,2,3-triazol-4-yl]thiophene-2-sulfonamide (**B**) and hCA IX isoenzyme 5fl4, and (*b*) 2D interaction diagram depicting the binding residues of 5fl4.

acterized by the lowest binding affinity, evidenced by its docking score of −0.784 kcal mol^{-1}. Compound **6** displayed glide a E-model energy of −47.945 kcal mol^{-1} and a ligand efficiency of −0.041 kcal mol^{-1}. Structurally, sulfanilamide **6** interacted with the protein 4iwz through hydrogen bonding with GLN_{92} (1.88 Å) and TRP_5 (2.11 Å), and through π–π stacking with THR_{199} (1.81 Å) (Fig. S12).

To determine the mode of interaction of the synthesized compounds with human carbonic anhydrase IX inhibitor (hCA IX), the synthesized compounds were docked into the active site of 5fl4, and the results obtained were compared with the docked results of the reference drug **B**. We observed that the reference drug interacts with amino acid residues ASP_{13} (1.59–2.73 Å) and VAL_{130} (2.53 Å) *via* hydrogen bonding, and with HID_{94} (5.49 Å) *via* π–cation interactions (*cf*. Table 7). Furthermore, **B** exhibited a docking score of −1.969 kcal mol^{-1}, a glide E-model energy of −41.029 kcal mol^{-1} and a ligand efficiency of −0.082 kcal mol^{-1}, and is surrounded by several amino acid residues. Some of the residues are TRP_9, PRO_{203}, THR_{201}, HID_{68}, LEU_{199}, HID_{94}, GLN_{92}, VAL_{171} and ZN_{264}, with bad contacts or interactions observed on residue ASP_{131} (Fig. 6). Benzenesulfonamide **2** presented the highest binding affinity, with a docking score of −1.977 kcal mol^{-1}, higher than the reference drug. All other synthesized compounds, except for *N*-cycloamino-*o*-nitrobenzenesulfonamide **1** (docking score = −0.807), displayed significantly good docking scores; however, they were lower than the reference drug (*cf*. Table 6). Compound **3** displayed hydrogen-bond interactions with amino acid residue GLN_{71}, with a bond length of 2.26 Å, and a π–cation interaction with amino acid residue HID_{94}, with a bond length of 4.05 Å (Fig. S15).

We observed that the docking scores of **2** with 4iwz and 5fl4 are close to those obtained for **A** with 4iwz and **B** with 5fl4. Docking scores of molecules with ring structures **1** and **3–6** (in the range > −1.67 kcal mol^{-1}) also correlated with the electronegativity and electrophilicity values presented in Table 5. This is informed by the HOMO and LUMO properties (Kumar *et al.*, 2018).

4. Conclusion

o-Nitrosulfonamides **1–3** and *N*-cycloamino-*o*-sulfanilamides **4–6** have been successfully synthesized, characterized and the intermolecular interactions analysed, as well as being tested *in silico* for carbonic anhydrase II (4iwz) and IX (5fl4) inhibitory activities. The results obtained from crystal packing and DFT analysis suggests that the molecules are held together by forces such as hydrogen bonding and π–π interactions. The results of the DFT study of compounds **1–6** were correlated with the molecular docking data and indicate that electronegativity and electrophilicity of the title compounds play an important role in their interaction with carbonic anhydrase II (4iwz) and IX (5fl4).

O-Nitrosulfonamide **2** displayed a good docking score against 4iwz (lower than the reference drug) and the best against 5fl4 (higher than the reference drug). These results provided a valuable synthesis approach and structural and docking information for compounds **1–6** that may be used for the development of potent antibacterial drugs.

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N-Cycloamino substituent effects on the packing architecture of *ortho*-sulfanilamide molecular crystals and their *in silico* carbonic anhydrase II and IX inhibitory activities

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Computing details

For all structures, data collection: *APEX2* (Bruker, 2011); cell refinement: *APEX2* (Bruker, 2011); data reduction: *SAINT* (Bruker, 2012); program(s) used to solve structure: SHELXT2018 (Sheldrick, 2015*a*); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015*b*) and ShelXle (Hübschle *et al.*, 2011); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2020) and *Mercury* (Macrae *et al.*, 2020).

1-[(2-Nitrophenyl)sulfonyl]pyrrolidine (ka097)

Crystal data

$C_{10}H_{12}N_2O_4S$
$M_r = 256.28$
Monoclinic, $P2_1/n$
$a = 8.6173$ (5) Å
$b = 14.6662$ (9) Å
$c = 9.4885$ (6) Å
$\beta = 108.075$ (3)°
$V = 1140.01$ (12) Å^3
$Z = 4$
$F(000) = 536$
$D_x = 1.493$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9888 reflections
$\theta = 2.7$–28.3°
$\mu = 0.29$ mm^{-1}
$T = 200$ K
Block, yellow
0.67 × 0.67 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: numerical (SADABS; Bruker, 2008)
$T_{\min} = 0.934$, $T_{\max} = 1.000$
24329 measured reflections
2849 independent reflections
2508 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.018$
$\theta_{\max} = 28.4°$, $\theta_{\min} = 2.7°$
$h = -11 \rightarrow 11$
$k = -17 \rightarrow 19$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.111$
$S = 1.05$
2849 reflections
149 parameters
20 restraints

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

$w = 1/[\sigma^2(F_o^2) + (0.0517P)^2 + 0.7926P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ e Å^{-3}

Special details

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

Refinement. Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2 U_{eq}(C).
The pyrrolidine group is disordered necessitating the use of the restraints SADI, EADP and SAME.
Single-crystal X-ray diffraction (SC-XRD) data were collected at 200 or 296 K on a Bruker APEXII CCD diffractometer with graphite-monochromated Mo *Kα* radiation using the *APEX2* data collection software and *SAINT* (Bruker 2012) for cell refinement and data reduction. The structures were solved by dual-space methods applying *SHELXT2018* (Sheldrick, 2015*a*) and refined by least-squares procedures using *SHELXL2018* (Sheldrick, 2015*b*). Data were corrected for absorption effects using the numerical method implemented in *SADABS* (Bruker 2012). All non-H atoms were refined anisotropically. The crystal structure diagrams were drawn with *ORTEP-3 for Windows* (Farrugia 2012).

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1	0.49089 (5)	0.66797 (3)	0.32216 (4)	0.02954 (13)	
O1	0.52737 (15)	0.62543 (9)	0.46416 (13)	0.0360 (3)	
O2	0.41164 (17)	0.75483 (10)	0.29726 (18)	0.0481 (4)	
O3	0.91161 (16)	0.56570 (9)	0.57998 (13)	0.0374 (3)	
O4	0.74333 (16)	0.49165 (8)	0.40041 (15)	0.0387 (3)	
N1	0.3849 (2)	0.59886 (12)	0.20100 (17)	0.0424 (4)	
N2	0.82322 (16)	0.55892 (9)	0.45200 (15)	0.0261 (3)	
C21	0.67960 (18)	0.68563 (10)	0.28603 (17)	0.0243 (3)	
C22	0.82125 (18)	0.63575 (9)	0.35184 (16)	0.0233 (3)	
C23	0.9678 (2)	0.65569 (11)	0.32834 (19)	0.0300 (3)	
H23	1.063007	0.621352	0.376473	0.036*	
C24	0.9742 (2)	0.72656 (12)	0.2335 (2)	0.0347 (4)	
H24	1.073735	0.740356	0.214723	0.042*	
C25	0.8355 (2)	0.77707 (12)	0.1663 (2)	0.0348 (4)	
H25	0.839913	0.825347	0.100842	0.042*	
C26	0.6899 (2)	0.75768 (11)	0.19374 (18)	0.0304 (3)	
H26	0.596179	0.793966	0.149089	0.036*	
C11	0.3771 (10)	0.4994 (5)	0.2187 (8)	0.0525 (5)	0.579 (7)
H11A	0.365697	0.483433	0.316363	0.063*	0.579 (7)
H11B	0.475419	0.469108	0.207784	0.063*	0.579 (7)
C12	0.2269 (6)	0.4739 (3)	0.0945 (5)	0.0525 (5)	0.579 (7)
H12A	0.126167	0.490743	0.117592	0.063*	0.579 (7)
H12B	0.224619	0.407828	0.072438	0.063*	0.579 (7)
C13	0.2474 (6)	0.5302 (4)	−0.0321 (5)	0.0525 (5)	0.579 (7)
H13A	0.142720	0.536536	−0.112750	0.063*	0.579 (7)

H13B	0.329694	0.502920	−0.072615	0.063*	0.579 (7)
C14	0.3052 (11)	0.6216 (4)	0.0416 (7)	0.0525 (5)	0.579 (7)
H14A	0.384050	0.650475	−0.001515	0.063*	0.579 (7)
H14B	0.211994	0.663509	0.029701	0.063*	0.579 (7)
C15	0.3620 (14)	0.5024 (7)	0.2240 (11)	0.0525 (5)	0.421 (7)
H15A	0.289652	0.492782	0.285820	0.063*	0.421 (7)
H15B	0.467484	0.471263	0.270293	0.063*	0.421 (7)
C16	0.2804 (9)	0.4696 (4)	0.0620 (7)	0.0525 (5)	0.421 (7)
H16A	0.362038	0.441474	0.021428	0.063*	0.421 (7)
H16B	0.193459	0.424491	0.057238	0.063*	0.421 (7)
C17	0.2083 (8)	0.5567 (5)	−0.0247 (7)	0.0525 (5)	0.421 (7)
H17A	0.207484	0.551559	−0.128998	0.063*	0.421 (7)
H17B	0.094759	0.566045	−0.023970	0.063*	0.421 (7)
C18	0.3140 (15)	0.6354 (6)	0.0501 (10)	0.0525 (5)	0.421 (7)
H18A	0.399357	0.649069	0.003320	0.063*	0.421 (7)
H18B	0.248645	0.691003	0.049588	0.063*	0.421 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0263 (2)	0.0333 (2)	0.0318 (2)	0.00668 (14)	0.01314 (16)	0.00676 (15)
O1	0.0363 (6)	0.0479 (7)	0.0283 (6)	0.0048 (5)	0.0166 (5)	0.0052 (5)
O2	0.0392 (7)	0.0428 (7)	0.0700 (10)	0.0185 (6)	0.0280 (7)	0.0142 (7)
O3	0.0405 (7)	0.0391 (7)	0.0282 (6)	0.0012 (5)	0.0045 (5)	0.0077 (5)
O4	0.0373 (7)	0.0259 (6)	0.0495 (8)	−0.0061 (5)	0.0087 (6)	0.0034 (5)
N1	0.0400 (8)	0.0479 (9)	0.0327 (8)	−0.0083 (7)	0.0017 (6)	0.0123 (7)
N2	0.0242 (6)	0.0237 (6)	0.0306 (7)	0.0030 (5)	0.0090 (5)	0.0045 (5)
C21	0.0261 (7)	0.0239 (7)	0.0246 (7)	0.0026 (5)	0.0103 (6)	0.0011 (5)
C22	0.0276 (7)	0.0195 (6)	0.0231 (7)	0.0016 (5)	0.0081 (6)	0.0009 (5)
C23	0.0270 (8)	0.0281 (7)	0.0363 (9)	0.0030 (6)	0.0116 (7)	0.0023 (6)
C24	0.0350 (8)	0.0329 (8)	0.0428 (9)	−0.0009 (7)	0.0218 (7)	0.0038 (7)
C25	0.0446 (10)	0.0287 (8)	0.0375 (9)	0.0036 (7)	0.0222 (8)	0.0087 (7)
C26	0.0351 (8)	0.0272 (7)	0.0305 (8)	0.0080 (6)	0.0125 (7)	0.0075 (6)
C11	0.0476 (9)	0.0604 (10)	0.0421 (7)	−0.0101 (7)	0.0034 (6)	0.0040 (7)
C12	0.0476 (9)	0.0604 (10)	0.0421 (7)	−0.0101 (7)	0.0034 (6)	0.0040 (7)
C13	0.0476 (9)	0.0604 (10)	0.0421 (7)	−0.0101 (7)	0.0034 (6)	0.0040 (7)
C14	0.0476 (9)	0.0604 (10)	0.0421 (7)	−0.0101 (7)	0.0034 (6)	0.0040 (7)
C15	0.0476 (9)	0.0604 (10)	0.0421 (7)	−0.0101 (7)	0.0034 (6)	0.0040 (7)
C16	0.0476 (9)	0.0604 (10)	0.0421 (7)	−0.0101 (7)	0.0034 (6)	0.0040 (7)
C17	0.0476 (9)	0.0604 (10)	0.0421 (7)	−0.0101 (7)	0.0034 (6)	0.0040 (7)
C18	0.0476 (9)	0.0604 (10)	0.0421 (7)	−0.0101 (7)	0.0034 (6)	0.0040 (7)

Geometric parameters (Å, º)

S1—O1	1.4285 (12)	C11—H11A	0.9900
S1—O2	1.4300 (13)	C11—H11B	0.9900
S1—N1	1.5913 (17)	C12—C13	1.513 (6)
S1—C21	1.7818 (15)	C12—H12A	0.9900

O3—N2	1.2209 (18)	C12—H12B	0.9900
O4—N2	1.2157 (18)	C13—C14	1.522 (6)
N1—C15	1.454 (10)	C13—H13A	0.9900
N1—C11	1.472 (8)	C13—H13B	0.9900
N1—C18	1.472 (8)	C14—H14A	0.9900
N1—C14	1.492 (6)	C14—H14B	0.9900
N2—C22	1.4706 (18)	C15—C16	1.555 (10)
C21—C26	1.393 (2)	C15—H15A	0.9900
C21—C22	1.394 (2)	C15—H15B	0.9900
C22—C23	1.380 (2)	C16—C17	1.543 (8)
C23—C24	1.387 (2)	C16—H16A	0.9900
C23—H23	0.9500	C16—H16B	0.9900
C24—C25	1.382 (2)	C17—C18	1.505 (8)
C24—H24	0.9500	C17—H17A	0.9900
C25—C26	1.388 (2)	C17—H17B	0.9900
C25—H25	0.9500	C18—H18A	0.9900
C26—H26	0.9500	C18—H18B	0.9900
C11—C12	1.503 (8)		

O1—S1—O2	119.93 (9)	C13—C12—H12A	111.4
O1—S1—N1	108.19 (8)	C11—C12—H12B	111.4
O2—S1—N1	108.08 (9)	C13—C12—H12B	111.4
O1—S1—C21	107.34 (7)	H12A—C12—H12B	109.3
O2—S1—C21	104.85 (8)	C12—C13—C14	102.6 (4)
N1—S1—C21	107.90 (8)	C12—C13—H13A	111.2
C15—N1—C18	117.8 (5)	C14—C13—H13A	111.2
C11—N1—C14	108.3 (4)	C12—C13—H13B	111.2
C15—N1—S1	125.8 (4)	C14—C13—H13B	111.2
C11—N1—S1	125.8 (3)	H13A—C13—H13B	109.2
C18—N1—S1	116.2 (3)	N1—C14—C13	104.5 (4)
C14—N1—S1	124.8 (2)	N1—C14—H14A	110.8
O4—N2—O3	124.55 (14)	C13—C14—H14A	110.8
O4—N2—C22	118.04 (13)	N1—C14—H14B	110.8
O3—N2—C22	117.32 (13)	C13—C14—H14B	110.8
C26—C21—C22	117.54 (14)	H14A—C14—H14B	108.9
C26—C21—S1	118.12 (12)	N1—C15—C16	101.2 (6)
C22—C21—S1	124.21 (11)	N1—C15—H15A	111.5
C23—C22—C21	122.31 (14)	C16—C15—H15A	111.5
C23—C22—N2	116.04 (13)	N1—C15—H15B	111.5
C21—C22—N2	121.64 (13)	C16—C15—H15B	111.5
C22—C23—C24	119.09 (15)	H15A—C15—H15B	109.4
C22—C23—H23	120.5	C17—C16—C15	104.7 (6)
C24—C23—H23	120.5	C17—C16—H16A	110.8
C25—C24—C23	119.87 (15)	C15—C16—H16A	110.8
C25—C24—H24	120.1	C17—C16—H16B	110.8
C23—C24—H24	120.1	C15—C16—H16B	110.8
C24—C25—C26	120.41 (15)	H16A—C16—H16B	108.9
C24—C25—H25	119.8	C18—C17—C16	107.4 (6)

C26—C25—H25	119.8	C18—C17—H17A	110.2
C25—C26—C21	120.75 (15)	C16—C17—H17A	110.2
C25—C26—H26	119.6	C18—C17—H17B	110.2
C21—C26—H26	119.6	C16—C17—H17B	110.2
N1—C11—C12	102.5 (5)	H17A—C17—H17B	108.5
N1—C11—H11A	111.3	N1—C18—C17	100.8 (5)
C12—C11—H11A	111.3	N1—C18—H18A	111.6
N1—C11—H11B	111.3	C17—C18—H18A	111.6
C12—C11—H11B	111.3	N1—C18—H18B	111.6
H11A—C11—H11B	109.2	C17—C18—H18B	111.6
C11—C12—C13	101.6 (5)	H18A—C18—H18B	109.4
C11—C12—H12A	111.4		

O1—S1—N1—C15	−13.5 (6)	O4—N2—C22—C21	65.74 (19)
O2—S1—N1—C15	−144.8 (6)	O3—N2—C22—C21	−117.70 (16)
C21—S1—N1—C15	102.3 (6)	C21—C22—C23—C24	−1.4 (2)
O1—S1—N1—C11	−21.2 (5)	N2—C22—C23—C24	179.13 (15)
O2—S1—N1—C11	−152.5 (5)	C22—C23—C24—C25	1.2 (3)
C21—S1—N1—C11	94.6 (5)	C23—C24—C25—C26	0.4 (3)
O1—S1—N1—C18	171.1 (7)	C24—C25—C26—C21	−1.8 (3)
O2—S1—N1—C18	39.8 (7)	C22—C21—C26—C25	1.5 (2)
C21—S1—N1—C18	−73.1 (7)	S1—C21—C26—C25	177.41 (13)
O1—S1—N1—C14	172.3 (5)	C14—N1—C11—C12	−27.8 (8)
O2—S1—N1—C14	41.0 (5)	S1—N1—C11—C12	163.9 (4)
C21—S1—N1—C14	−71.9 (5)	N1—C11—C12—C13	43.3 (8)
O1—S1—C21—C26	−152.34 (13)	C11—C12—C13—C14	−42.6 (7)
O2—S1—C21—C26	−23.76 (15)	C11—N1—C14—C13	1.2 (8)
N1—S1—C21—C26	91.26 (14)	S1—N1—C14—C13	169.7 (3)
O1—S1—C21—C22	23.24 (15)	C12—C13—C14—N1	25.6 (7)
O2—S1—C21—C22	151.82 (14)	C18—N1—C15—C16	6.6 (11)
N1—S1—C21—C22	−93.15 (15)	S1—N1—C15—C16	−168.8 (4)
C26—C21—C22—C23	0.1 (2)	N1—C15—C16—C17	−21.2 (10)
S1—C21—C22—C23	−175.54 (12)	C15—C16—C17—C18	29.7 (11)
C26—C21—C22—N2	179.49 (14)	C15—N1—C18—C17	11.2 (12)
S1—C21—C22—N2	3.9 (2)	S1—N1—C18—C17	−173.0 (5)
O4—N2—C22—C23	−114.81 (16)	C16—C17—C18—N1	−24.5 (11)
O3—N2—C22—C23	61.75 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*A*···O1^{i}	0.99	2.56	3.398 (7)	143
C11—H11*B*···O4	0.99	2.48	3.093 (8)	120
C25—H25···O4^{ii}	0.95	2.54	3.240 (2)	131
C25—H25···O1^{iii}	0.95	2.47	3.228 (2)	137

C26—H26···O2	0.95	2.49	2.860 (2)	103
C26—H26···O3^{iv}	0.95	2.56	3.467 (2)	160

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

1-[(2-Nitrophenyl)sulfonyl]piperidine (ja198)

Crystal data

$C_{11}H_{14}N_2O_4S$
M_r = 270.30
Monoclinic, $P2_1/n$
a = 8.6881 (9) Å
b = 15.0266 (14) Å
c = 9.8337 (10) Å
β = 106.526 (4)°
V = 1230.8 (2) Å^3
Z = 4
$F(000)$ = 568
D_x = 1.459 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9983 reflections
θ = 2.6–28.3°
μ = 0.27 mm^{-1}
T = 296 K
Block, colourless
0.48 × 0.47 × 0.45 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: numerical (SADABS; Bruker, 2008)
T_{min} = 0.941, T_{max} = 1.000
25328 measured reflections
3059 independent reflections
2662 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 28.3°, θ_{min} = 2.6°
h = −11→11
k = −20→19
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.106
S = 1.06
3059 reflections
163 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0546P)^2 + 0.3577P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Special details

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

Refinement. Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2 U_{eq}(C).
Single-crystal X-ray diffraction (SC-XRD) data were collected at 200 or 296 K on a Bruker APEXII CCD diffractometer with graphite-monochromated Mo $K\alpha$ radiation using the *APEX2* data collection software and *SAINT* (Bruker 2012) for cell refinement and data reduction. The structures were solved by dual-space methods applying *SHELXT2018* (Sheldrick, 2015*a*) and refined by least-squares procedures using *SHELXL2018* (Sheldrick, 2015*b*). Data were corrected for absorption effects using the numerical method implemented in *SADABS* (Bruker 2012). All non-H atoms were refined anisotropically. The crystal structure diagrams were drawn with *ORTEP-3 for Windows* (Farrugia 2012).

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

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$
S1	0.50241 (4)	0.66804 (2)	0.66863 (4)	0.04006 (12)
O1	0.57946 (15)	0.75271 (8)	0.69071 (16)	0.0609 (3)
O2	0.46987 (14)	0.62642 (8)	0.53305 (11)	0.0521 (3)
O3	0.23990 (17)	0.49620 (9)	0.58739 (17)	0.0705 (4)
O4	0.08960 (18)	0.57419 (11)	0.41946 (13)	0.0734 (4)
N1	0.61016 (14)	0.60117 (8)	0.78644 (13)	0.0416 (3)
N2	0.17049 (15)	0.56417 (9)	0.54124 (14)	0.0463 (3)
C11	0.5872 (2)	0.50422 (11)	0.76898 (19)	0.0517 (4)
H11A	0.564441	0.488569	0.669528	0.062*
H11B	0.496745	0.485803	0.801683	0.062*
C12	0.7374 (2)	0.45729 (13)	0.8536 (2)	0.0627 (5)
H12A	0.720629	0.393457	0.845762	0.075*
H12B	0.825213	0.471853	0.814835	0.075*
C13	0.7814 (2)	0.48376 (14)	1.0071 (2)	0.0606 (5)
H13A	0.881221	0.455002	1.057712	0.073*
H13B	0.698481	0.463866	1.048463	0.073*
C14	0.8004 (2)	0.58345 (15)	1.0233 (2)	0.0662 (5)
H14A	0.819586	0.599222	1.122396	0.079*
H14B	0.892831	0.602183	0.993857	0.079*
C15	0.6533 (2)	0.63160 (13)	0.93604 (18)	0.0582 (4)
H15A	0.564368	0.620532	0.975063	0.070*
H15B	0.673748	0.695143	0.939930	0.070*
C21	0.31422 (16)	0.68463 (9)	0.70518 (14)	0.0357 (3)
C22	0.17424 (16)	0.63776 (9)	0.64082 (14)	0.0372 (3)
C23	0.02972 (18)	0.65753 (11)	0.66598 (17)	0.0468 (3)
H23	−0.062624	0.626220	0.619609	0.056*
C24	0.0237 (2)	0.72436 (12)	0.76084 (19)	0.0530 (4)
H24	−0.072622	0.737406	0.780184	0.064*
C25	0.1604 (2)	0.77165 (12)	0.82675 (18)	0.0529 (4)
H25	0.156178	0.816624	0.890548	0.063*
C26	0.30433 (19)	0.75250 (10)	0.79841 (16)	0.0449 (3)
H26	0.395471	0.785498	0.842346	0.054*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.03940 (19)	0.0373 (2)	0.0487 (2)	−0.00205 (13)	0.02106 (15)	0.00020 (13)
O1	0.0540 (7)	0.0420 (6)	0.0951 (10)	−0.0107 (5)	0.0347 (6)	0.0018 (6)
O2	0.0559 (6)	0.0647 (7)	0.0425 (6)	0.0024 (5)	0.0248 (5)	−0.0001 (5)
O3	0.0696 (8)	0.0434 (7)	0.0929 (10)	0.0075 (6)	0.0143 (7)	−0.0118 (7)
O4	0.0767 (9)	0.0890 (10)	0.0474 (7)	0.0056 (8)	0.0066 (6)	−0.0153 (7)
N1	0.0405 (6)	0.0379 (6)	0.0457 (6)	0.0027 (5)	0.0110 (5)	−0.0090 (5)
N2	0.0405 (6)	0.0466 (7)	0.0525 (7)	−0.0037 (5)	0.0141 (5)	−0.0078 (6)
C11	0.0540 (9)	0.0402 (8)	0.0565 (9)	0.0011 (7)	0.0089 (7)	−0.0080 (7)
C12	0.0674 (11)	0.0466 (9)	0.0712 (11)	0.0155 (8)	0.0150 (9)	−0.0021 (8)

C13	0.0515 (9)	0.0706 (12)	0.0588 (10)	0.0106 (8)	0.0142 (8)	0.0126 (9)
C14	0.0570 (10)	0.0777 (13)	0.0555 (10)	0.0069 (9)	0.0024 (8)	−0.0120 (9)
C15	0.0583 (9)	0.0592 (10)	0.0521 (9)	0.0086 (8)	0.0074 (7)	−0.0185 (8)
C21	0.0383 (6)	0.0327 (6)	0.0390 (6)	0.0012 (5)	0.0157 (5)	0.0032 (5)
C22	0.0403 (7)	0.0344 (6)	0.0381 (6)	0.0015 (5)	0.0128 (5)	0.0032 (5)
C23	0.0386 (7)	0.0481 (8)	0.0551 (9)	0.0006 (6)	0.0155 (6)	0.0046 (7)
C24	0.0493 (8)	0.0539 (9)	0.0645 (10)	0.0104 (7)	0.0302 (7)	0.0049 (8)
C25	0.0634 (10)	0.0457 (8)	0.0580 (9)	0.0072 (7)	0.0307 (8)	−0.0055 (7)
C26	0.0491 (8)	0.0386 (7)	0.0496 (8)	−0.0008 (6)	0.0184 (6)	−0.0045 (6)

Geometric parameters (Å, º)

S1—O1	1.4252 (12)	C13—H13B	0.9700
S1—O2	1.4263 (12)	C14—C15	1.506 (3)
S1—N1	1.6160 (13)	C14—H14A	0.9700
S1—C21	1.7885 (14)	C14—H14B	0.9700
O3—N2	1.2070 (19)	C15—H15A	0.9700
O4—N2	1.2132 (18)	C15—H15B	0.9700
N1—C11	1.474 (2)	C21—C26	1.390 (2)
N1—C15	1.4833 (19)	C21—C22	1.3921 (19)
N2—C22	1.4713 (19)	C22—C23	1.379 (2)
C11—C12	1.508 (2)	C23—C24	1.382 (2)
C11—H11A	0.9700	C23—H23	0.9300
C11—H11B	0.9700	C24—C25	1.378 (3)
C12—C13	1.501 (3)	C24—H24	0.9300
C12—H12A	0.9700	C25—C26	1.386 (2)
C12—H12B	0.9700	C25—H25	0.9300
C13—C14	1.511 (3)	C26—H26	0.9300
C13—H13A	0.9700		

O1—S1—O2	119.57 (8)	C15—C14—C13	111.53 (16)
O1—S1—N1	107.14 (7)	C15—C14—H14A	109.3
O2—S1—N1	108.53 (7)	C13—C14—H14A	109.3
O1—S1—C21	105.16 (7)	C15—C14—H14B	109.3
O2—S1—C21	107.43 (7)	C13—C14—H14B	109.3
N1—S1—C21	108.59 (6)	H14A—C14—H14B	108.0
C11—N1—C15	114.07 (14)	N1—C15—C14	110.56 (14)
C11—N1—S1	119.97 (10)	N1—C15—H15A	109.5
C15—N1—S1	116.79 (10)	C14—C15—H15A	109.5
O3—N2—O4	124.29 (15)	N1—C15—H15B	109.5
O3—N2—C22	118.14 (13)	C14—C15—H15B	109.5
O4—N2—C22	117.42 (14)	H15A—C15—H15B	108.1
N1—C11—C12	109.30 (14)	C26—C21—C22	117.46 (13)
N1—C11—H11A	109.8	C26—C21—S1	117.79 (11)
C12—C11—H11A	109.8	C22—C21—S1	124.65 (11)
N1—C11—H11B	109.8	C23—C22—C21	122.11 (13)
C12—C11—H11B	109.8	C23—C22—N2	115.62 (13)
H11A—C11—H11B	108.3	C21—C22—N2	122.27 (12)

C13—C12—C11	111.34 (15)	C22—C23—C24	119.28 (15)
C13—C12—H12A	109.4	C22—C23—H23	120.4
C11—C12—H12A	109.4	C24—C23—H23	120.4
C13—C12—H12B	109.4	C25—C24—C23	119.93 (14)
C11—C12—H12B	109.4	C25—C24—H24	120.0
H12A—C12—H12B	108.0	C23—C24—H24	120.0
C12—C13—C14	110.97 (16)	C24—C25—C26	120.31 (15)
C12—C13—H13A	109.4	C24—C25—H25	119.8
C14—C13—H13A	109.4	C26—C25—H25	119.8
C12—C13—H13B	109.4	C25—C26—C21	120.88 (15)
C14—C13—H13B	109.4	C25—C26—H26	119.6
H13A—C13—H13B	108.0	C21—C26—H26	119.6

O1—S1—N1—C11	−163.84 (12)	O2—S1—C21—C22	21.08 (14)
O2—S1—N1—C11	−33.45 (14)	N1—S1—C21—C22	−96.11 (13)
C21—S1—N1—C11	83.04 (13)	C26—C21—C22—C23	0.7 (2)
O1—S1—N1—C15	51.20 (14)	S1—C21—C22—C23	−175.47 (11)
O2—S1—N1—C15	−178.40 (12)	C26—C21—C22—N2	−179.50 (13)
C21—S1—N1—C15	−61.91 (13)	S1—C21—C22—N2	4.32 (19)
C15—N1—C11—C12	−56.72 (19)	O3—N2—C22—C23	−111.68 (16)
S1—N1—C11—C12	157.43 (13)	O4—N2—C22—C23	64.06 (19)
N1—C11—C12—C13	56.4 (2)	O3—N2—C22—C21	68.51 (19)
C11—C12—C13—C14	−56.2 (2)	O4—N2—C22—C21	−115.74 (16)
C12—C13—C14—C15	54.1 (2)	C21—C22—C23—C24	−1.7 (2)
C11—N1—C15—C14	55.3 (2)	N2—C22—C23—C24	178.45 (14)
S1—N1—C15—C14	−157.73 (14)	C22—C23—C24—C25	1.4 (2)
C13—C14—C15—N1	−52.6 (2)	C23—C24—C25—C26	0.0 (3)
O1—S1—C21—C26	−26.70 (14)	C24—C25—C26—C21	−1.0 (3)
O2—S1—C21—C26	−155.08 (12)	C22—C21—C26—C25	0.7 (2)
N1—S1—C21—C26	87.72 (12)	S1—C21—C26—C25	177.14 (12)
O1—S1—C21—C22	149.47 (12)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*A*···O2	0.97	2.48	2.907 (2)	107
C11—H11*A*···O2$^{\rm i}$	0.97	2.59	3.476 (2)	152
C15—H15*B*···O1	0.97	2.51	2.943 (2)	107
C25—H25···O2$^{\rm ii}$	0.93	2.57	3.3337 (19)	140
C26—H26···O1	0.93	2.53	2.877 (2)	103

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

1-[(2-Nitrophenyl)sulfonyl]-2,3-dihydro-1*H*-indole (ja250)

Crystal data

$C_{14}H_{12}N_2O_4S$
M_r = 304.32
Monoclinic, $P2_1/n$

a = 7.4701 (5) Å
b = 23.6743 (12) Å
c = 7.8614 (5) Å

β = 94.989 (3)°
V = 1385.02 (15) Å^3
Z = 4
F(000) = 632
D_x = 1.459 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9952 reflections
θ = 2.7–27.1°
μ = 0.25 mm^{-1}
T = 296 K
Block, yellow
0.62 × 0.51 × 0.43 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: numerical (SADABS; Bruker, 2008)
T_{min} = 0.913, T_{max} = 1.000
28400 measured reflections
3434 independent reflections
2864 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 28.4°, θ_{min} = 2.7°
$h = -9 \rightarrow 9$
$k = -31 \rightarrow 28$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.105
S = 1.05
3434 reflections
190 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 0.5135P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Special details

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

Refinement. Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2 U_{eq}(C).
Single-crystal X-ray diffraction (SC-XRD) data were collected at 200 or 296 K on a Bruker APEXII CCD diffractometer with graphite-monochromated Mo $K\alpha$ radiation using the *APEX2* data collection software and *SAINT* (Bruker 2012) for cell refinement and data reduction. The structures were solved by dual-space methods applying *SHELXT2018* (Sheldrick, 2015*a*) and refined by least-squares procedures using *SHELXL2018* (Sheldrick, 2015*b*). Data were corrected for absorption effects using the numerical method implemented in *SADABS* (Bruker 2012). All non-H atoms were refined anisotropically. The crystal structure diagrams were drawn with *ORTEP-3 for Windows* (Farrugia 2012).

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

	x	y	z	U_{iso}*/U_{eq}
S1	0.79243 (5)	0.61702 (2)	0.46891 (5)	0.03709 (12)
O1	0.73327 (18)	0.65999 (5)	0.57767 (16)	0.0546 (3)
O2	0.91672 (16)	0.57553 (5)	0.53509 (15)	0.0487 (3)
O3	0.9987 (3)	0.77311 (7)	0.5254 (3)	0.1022 (6)
O4	1.1144 (3)	0.69290 (8)	0.6043 (2)	0.0839 (5)
N1	0.61681 (17)	0.58580 (6)	0.38325 (18)	0.0418 (3)

N2	1.0368 (2)	0.72407 (8)	0.5003 (2)	0.0611 (5)
C1	0.4442 (2)	0.61631 (8)	0.3457 (3)	0.0549 (5)
H1A	0.456425	0.646065	0.262694	0.066*
H1B	0.404075	0.632810	0.448776	0.066*
C2	0.3136 (2)	0.57142 (9)	0.2744 (3)	0.0576 (5)
H2A	0.224774	0.563294	0.354023	0.069*
H2B	0.252109	0.583591	0.166870	0.069*
C11	0.6039 (2)	0.53032 (6)	0.31204 (19)	0.0368 (3)
C12	0.4274 (2)	0.52060 (8)	0.2492 (2)	0.0436 (4)
C13	0.3805 (3)	0.46882 (9)	0.1789 (2)	0.0572 (5)
H13	0.262278	0.461664	0.137548	0.069*
C14	0.5100 (3)	0.42764 (9)	0.1703 (2)	0.0611 (5)
H14	0.478867	0.392525	0.123633	0.073*
C15	0.6852 (3)	0.43842 (8)	0.2306 (2)	0.0567 (5)
H15	0.771627	0.410511	0.222516	0.068*
C16	0.7352 (2)	0.48989 (7)	0.3029 (2)	0.0478 (4)
H16	0.853542	0.496970	0.344103	0.057*
C21	0.8884 (2)	0.65319 (6)	0.30000 (19)	0.0365 (3)
C22	0.9890 (2)	0.70184 (7)	0.3266 (2)	0.0466 (4)
C23	1.0551 (3)	0.73080 (9)	0.1950 (3)	0.0681 (6)
H23	1.120293	0.763931	0.215631	0.082*
C24	1.0234 (3)	0.70999 (11)	0.0308 (3)	0.0726 (6)
H24	1.068889	0.728941	−0.059696	0.087*
C25	0.9256 (3)	0.66178 (10)	0.0010 (2)	0.0609 (5)
H25	0.905119	0.647920	−0.109653	0.073*
C26	0.8568 (2)	0.63337 (8)	0.1351 (2)	0.0460 (4)
H26	0.788883	0.600801	0.113758	0.055*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0407 (2)	0.0361 (2)	0.0349 (2)	0.00043 (15)	0.00584 (15)	−0.00023 (14)
O1	0.0670 (8)	0.0504 (7)	0.0492 (7)	−0.0017 (6)	0.0214 (6)	−0.0118 (5)
O2	0.0468 (7)	0.0481 (7)	0.0493 (7)	0.0008 (5)	−0.0064 (5)	0.0089 (5)
O3	0.1429 (18)	0.0630 (10)	0.1030 (14)	−0.0190 (11)	0.0239 (12)	−0.0363 (10)
O4	0.0995 (13)	0.0977 (13)	0.0515 (9)	−0.0136 (10)	−0.0109 (8)	−0.0084 (9)
N1	0.0335 (7)	0.0364 (7)	0.0555 (8)	0.0049 (5)	0.0039 (6)	0.0012 (6)
N2	0.0699 (11)	0.0578 (10)	0.0574 (10)	−0.0260 (9)	0.0151 (8)	−0.0171 (8)
C1	0.0417 (9)	0.0549 (11)	0.0684 (12)	0.0155 (8)	0.0075 (8)	0.0090 (9)
C2	0.0349 (9)	0.0831 (14)	0.0548 (11)	0.0060 (9)	0.0043 (8)	0.0050 (10)
C11	0.0387 (8)	0.0370 (8)	0.0348 (8)	−0.0030 (6)	0.0032 (6)	0.0070 (6)
C12	0.0381 (8)	0.0584 (10)	0.0347 (8)	−0.0066 (7)	0.0054 (6)	0.0078 (7)
C13	0.0530 (11)	0.0703 (13)	0.0478 (10)	−0.0229 (10)	0.0017 (8)	0.0001 (9)
C14	0.0835 (15)	0.0499 (11)	0.0495 (11)	−0.0217 (10)	0.0023 (10)	−0.0015 (8)
C15	0.0749 (13)	0.0394 (9)	0.0548 (11)	0.0063 (9)	−0.0001 (9)	0.0024 (8)
C16	0.0457 (9)	0.0415 (9)	0.0547 (10)	0.0042 (7)	−0.0045 (8)	0.0018 (7)
C21	0.0382 (8)	0.0369 (7)	0.0349 (7)	0.0017 (6)	0.0052 (6)	0.0008 (6)
C22	0.0528 (10)	0.0438 (9)	0.0437 (9)	−0.0075 (7)	0.0071 (7)	−0.0018 (7)

C23	0.0842 (15)	0.0601 (12)	0.0616 (13)	−0.0261 (11)	0.0150 (11)	0.0068 (10)
C24	0.0845 (16)	0.0843 (15)	0.0509 (12)	−0.0173 (13)	0.0167 (11)	0.0202 (11)
C25	0.0674 (13)	0.0810 (14)	0.0344 (9)	−0.0051 (11)	0.0056 (8)	0.0028 (9)
C26	0.0474 (9)	0.0527 (10)	0.0376 (8)	−0.0043 (7)	0.0022 (7)	−0.0025 (7)

Geometric parameters (Å, º)

S1—O2	1.4190 (12)	C13—C14	1.379 (3)
S1—O1	1.4236 (12)	C13—H13	0.9300
S1—N1	1.6022 (14)	C14—C15	1.376 (3)
S1—C21	1.7822 (15)	C14—H14	0.9300
O3—N2	1.216 (2)	C15—C16	1.382 (3)
O4—N2	1.211 (2)	C15—H15	0.9300
N1—C11	1.428 (2)	C16—H16	0.9300
N1—C1	1.485 (2)	C21—C26	1.380 (2)
N2—C22	1.478 (2)	C21—C22	1.381 (2)
C1—C2	1.517 (3)	C22—C23	1.369 (3)
C1—H1A	0.9700	C23—C24	1.382 (3)
C1—H1B	0.9700	C23—H23	0.9300
C2—C12	1.497 (3)	C24—C25	1.364 (3)
C2—H2A	0.9700	C24—H24	0.9300
C2—H2B	0.9700	C25—C26	1.387 (2)
C11—C16	1.377 (2)	C25—H25	0.9300
C11—C12	1.386 (2)	C26—H26	0.9300
C12—C13	1.377 (3)		

O2—S1—O1	120.12 (8)	C12—C13—C14	119.61 (18)
O2—S1—N1	108.69 (7)	C12—C13—H13	120.2
O1—S1—N1	107.25 (8)	C14—C13—H13	120.2
O2—S1—C21	108.23 (7)	C15—C14—C13	120.12 (18)
O1—S1—C21	105.68 (7)	C15—C14—H14	119.9
N1—S1—C21	106.04 (7)	C13—C14—H14	119.9
C11—N1—C1	109.96 (13)	C14—C15—C16	121.33 (19)
C11—N1—S1	127.90 (11)	C14—C15—H15	119.3
C1—N1—S1	121.62 (12)	C16—C15—H15	119.3
O4—N2—O3	125.30 (19)	C11—C16—C15	117.77 (17)
O4—N2—C22	118.03 (17)	C11—C16—H16	121.1
O3—N2—C22	116.6 (2)	C15—C16—H16	121.1
N1—C1—C2	104.61 (14)	C26—C21—C22	118.20 (15)
N1—C1—H1A	110.8	C26—C21—S1	119.31 (12)
C2—C1—H1A	110.8	C22—C21—S1	122.45 (12)
N1—C1—H1B	110.8	C23—C22—C21	122.03 (17)
C2—C1—H1B	110.8	C23—C22—N2	116.49 (16)
H1A—C1—H1B	108.9	C21—C22—N2	121.44 (15)
C12—C2—C1	104.93 (14)	C22—C23—C24	118.92 (19)
C12—C2—H2A	110.8	C22—C23—H23	120.5
C1—C2—H2A	110.8	C24—C23—H23	120.5
C12—C2—H2B	110.8	C25—C24—C23	120.27 (19)

C1—C2—H2B	110.8	C25—C24—H24	119.9
H2A—C2—H2B	108.8	C23—C24—H24	119.9
C16—C11—C12	121.72 (16)	C24—C25—C26	120.26 (18)
C16—C11—N1	129.46 (15)	C24—C25—H25	119.9
C12—C11—N1	108.81 (14)	C26—C25—H25	119.9
C13—C12—C11	119.44 (17)	C21—C26—C25	120.31 (17)
C13—C12—C2	129.88 (17)	C21—C26—H26	119.8
C11—C12—C2	110.67 (15)	C25—C26—H26	119.8

O2—S1—N1—C11	−26.31 (16)	N1—C11—C16—C15	178.24 (16)
O1—S1—N1—C11	−157.59 (13)	C14—C15—C16—C11	−0.3 (3)
C21—S1—N1—C11	89.85 (14)	O2—S1—C21—C26	90.65 (14)
O2—S1—N1—C1	162.82 (14)	O1—S1—C21—C26	−139.48 (14)
O1—S1—N1—C1	31.54 (16)	N1—S1—C21—C26	−25.83 (15)
C21—S1—N1—C1	−81.01 (15)	O2—S1—C21—C22	−91.72 (15)
C11—N1—C1—C2	10.22 (19)	O1—S1—C21—C22	38.16 (16)
S1—N1—C1—C2	−177.44 (12)	N1—S1—C21—C22	151.80 (14)
N1—C1—C2—C12	−9.09 (19)	C26—C21—C22—C23	0.8 (3)
C1—N1—C11—C16	173.54 (17)	S1—C21—C22—C23	−176.87 (17)
S1—N1—C11—C16	1.8 (3)	C26—C21—C22—N2	−176.72 (17)
C1—N1—C11—C12	−7.25 (18)	S1—C21—C22—N2	5.6 (2)
S1—N1—C11—C12	−178.98 (12)	O4—N2—C22—C23	−122.4 (2)
C16—C11—C12—C13	1.4 (2)	O3—N2—C22—C23	55.7 (3)
N1—C11—C12—C13	−177.85 (15)	O4—N2—C22—C21	55.3 (3)
C16—C11—C12—C2	−179.67 (16)	O3—N2—C22—C21	−126.6 (2)
N1—C11—C12—C2	1.05 (18)	C21—C22—C23—C24	−1.4 (3)
C1—C2—C12—C13	−175.99 (18)	N2—C22—C23—C24	176.2 (2)
C1—C2—C12—C11	5.3 (2)	C22—C23—C24—C25	0.9 (4)
C11—C12—C13—C14	−0.8 (3)	C23—C24—C25—C26	0.2 (4)
C2—C12—C13—C14	−179.42 (18)	C22—C21—C26—C25	0.3 (3)
C12—C13—C14—C15	−0.4 (3)	S1—C21—C26—C25	178.08 (14)
C13—C14—C15—C16	1.0 (3)	C24—C25—C26—C21	−0.8 (3)
C12—C11—C16—C15	−0.9 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16···O2	0.93	2.41	2.975 (2)	119
C16—H16···O2^{i}	0.93	2.55	3.195 (2)	127
C23—H23···O1^{ii}	0.93	2.30	3.085 (2)	142
C26—H26···N1	0.93	2.60	2.982 (2)	106

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

2-(Pyrrolidine-1-sulfonyl)aniline (ja192)

Crystal data

$C_{10}H_{14}N_2O_2S$
$M_r = 226.29$
Monoclinic, $P2_1/c$
$a = 16.399\ (3)$ Å
$b = 7.9485\ (12)$ Å
$c = 18.376\ (3)$ Å
$\beta = 113.907\ (6)°$
$V = 2189.7\ (6)$ Å^3
$Z = 8$

$F(000) = 960$
$D_x = 1.373$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9858 reflections
$\theta = 2.3–28.3°$
$\mu = 0.28$ mm^{-1}
$T = 296$ K
Block, colourless
0.47 × 0.32 × 0.20 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: numerical (SADABS; Bruker, 2008)
$T_{\min} = 0.927$, $T_{\max} = 1.000$

55955 measured reflections
5477 independent reflections
4489 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.032$
$\theta_{\max} = 28.4°$, $\theta_{\min} = 2.3°$
$h = -21 \rightarrow 21$
$k = -10 \rightarrow 10$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.107$
$S = 1.04$
5477 reflections
287 parameters
0 restraints
Primary atom site location: dual

Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 0.5724P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.33$ e Å^{-3}
$\Delta\rho_{\min} = -0.36$ e Å^{-3}

Special details

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

Refinement. Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with $U_{\text{iso}}(\text{H})$ set to 1.2 $U_{\text{eq}}(\text{C})$.
The nitrogen-bound H atoms were located on a difference map and refined freely.
A reflection with large difference between its observed and calculated intensities was omitted. This is due to obstruction by the beam stop.
Single-crystal X-ray diffraction (SC-XRD) data were collected at 200 or 296 K on a Bruker APEXII CCD diffractometer with graphite-monochromated Mo $K\alpha$ radiation using the *APEX2* data collection software and *SAINT* (Bruker 2012) for cell refinement and data reduction. The structures were solved by dual-space methods applying *SHELXT2018* (Sheldrick, 2015*a*) and refined by least-squares procedures using *SHELXL2018* (Sheldrick, 2015*b*). Data were corrected for absorption effects using the numerical method implemented in *SADABS* (Bruker 2012). All non-H atoms were refined anisotropically. The crystal structure diagrams were drawn with *ORTEP-3 for Windows* (Farrugia 2012).

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

	x	y	z	U_{iso}*/U_{eq}
S1	0.60593 (2)	0.11220 (4)	0.38373 (2)	0.03995 (10)
S2	0.83675 (3)	0.54094 (5)	0.72672 (2)	0.04473 (11)
O11	0.55812 (7)	0.21253 (15)	0.41849 (9)	0.0639 (3)
O12	0.59914 (8)	0.15372 (16)	0.30611 (7)	0.0604 (3)
O21	0.76909 (9)	0.41413 (14)	0.70735 (7)	0.0567 (3)
O22	0.91886 (9)	0.51408 (18)	0.79373 (7)	0.0691 (4)
N11	0.56877 (8)	−0.07718 (15)	0.37882 (7)	0.0403 (3)
N12	0.69275 (18)	0.1559 (3)	0.56795 (13)	0.0824 (7)
N21	0.86112 (9)	0.55871 (17)	0.65022 (8)	0.0461 (3)
N22	0.63790 (10)	0.6609 (2)	0.65060 (10)	0.0564 (4)
C11	0.55387 (14)	−0.1535 (2)	0.44477 (11)	0.0597 (5)
H11A	0.508003	−0.094373	0.455235	0.072*
H11B	0.608273	−0.156605	0.492981	0.072*
C12	0.52380 (17)	−0.3286 (2)	0.41255 (14)	0.0740 (6)
H12A	0.533347	−0.407503	0.455504	0.089*
H12B	0.461089	−0.329150	0.376874	0.089*
C13	0.58061 (14)	−0.3722 (2)	0.36888 (11)	0.0592 (4)
H13A	0.549989	−0.450820	0.326115	0.071*
H13B	0.636476	−0.422062	0.404671	0.071*
C14	0.59696 (11)	−0.2074 (2)	0.33613 (9)	0.0490 (4)
H14A	0.659546	−0.194754	0.346526	0.059*
H14B	0.561903	−0.200442	0.279189	0.059*
C21	0.79052 (12)	0.5923 (3)	0.57112 (10)	0.0560 (4)
H21A	0.756395	0.691183	0.572202	0.067*
H21B	0.750349	0.497049	0.552570	0.067*
C22	0.83964 (15)	0.6204 (4)	0.51900 (13)	0.0842 (7)
H22A	0.830797	0.525926	0.483167	0.101*
H22B	0.818267	0.721612	0.487506	0.101*
C23	0.93579 (15)	0.6377 (4)	0.57256 (15)	0.0835 (7)
H23A	0.960942	0.736580	0.558485	0.100*
H23B	0.968833	0.539558	0.568498	0.100*
C24	0.94016 (12)	0.6543 (3)	0.65616 (13)	0.0707 (5)
H24A	0.994454	0.605166	0.695106	0.085*
H24B	0.936292	0.770978	0.669874	0.085*
C111	0.71909 (9)	0.11558 (16)	0.44789 (8)	0.0362 (3)
C112	0.74894 (11)	0.1352 (2)	0.53050 (9)	0.0492 (4)
C113	0.84137 (14)	0.1302 (3)	0.57508 (11)	0.0701 (6)
H113	0.863875	0.141455	0.630123	0.084*
C114	0.89883 (13)	0.1091 (3)	0.53914 (14)	0.0747 (6)
H114	0.959847	0.107237	0.570304	0.090*
C115	0.86913 (11)	0.0906 (3)	0.45820 (13)	0.0648 (5)
H115	0.909391	0.076405	0.434745	0.078*
C116	0.77956 (10)	0.0933 (2)	0.41274 (10)	0.0456 (3)
H116	0.758579	0.080195	0.357861	0.055*
C211	0.79105 (9)	0.73166 (17)	0.74078 (8)	0.0391 (3)

C212	0.69962 (9)	0.76850 (18)	0.70242 (8)	0.0395 (3)
C213	0.67185 (11)	0.9265 (2)	0.71751 (10)	0.0475 (3)
H213	0.611709	0.954422	0.692911	0.057*
C214	0.73005 (12)	1.0408 (2)	0.76707 (11)	0.0556 (4)
H214	0.709137	1.144299	0.775829	0.067*
C215	0.81985 (13)	1.0037 (2)	0.80430 (12)	0.0638 (5)
H215	0.859590	1.081726	0.837922	0.077*
C216	0.84970 (11)	0.8504 (2)	0.79108 (10)	0.0549 (4)
H216	0.910132	0.825037	0.816091	0.066*
H12C	0.7160 (18)	0.193 (4)	0.6109 (17)	0.095 (9)*
H12D	0.641 (2)	0.190 (4)	0.5399 (19)	0.113 (12)*
H21C	0.5862 (16)	0.690 (3)	0.6389 (13)	0.073 (7)*
H21D	0.6538 (13)	0.556 (3)	0.6567 (12)	0.060 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.02972 (16)	0.03424 (17)	0.0509 (2)	−0.00093 (12)	0.01122 (14)	0.00394 (13)
S2	0.0512 (2)	0.03890 (19)	0.03977 (19)	0.00835 (15)	0.01394 (16)	0.00206 (14)
O11	0.0418 (6)	0.0440 (6)	0.1067 (10)	0.0032 (5)	0.0311 (6)	−0.0126 (6)
O12	0.0500 (6)	0.0649 (7)	0.0500 (6)	−0.0067 (5)	0.0034 (5)	0.0230 (5)
O21	0.0805 (8)	0.0364 (5)	0.0607 (7)	−0.0048 (5)	0.0364 (6)	0.0000 (5)
O22	0.0678 (8)	0.0739 (8)	0.0485 (7)	0.0292 (7)	0.0060 (6)	0.0050 (6)
N11	0.0384 (6)	0.0377 (6)	0.0470 (6)	−0.0090 (5)	0.0196 (5)	−0.0053 (5)
N12	0.0955 (16)	0.1087 (16)	0.0550 (10)	−0.0348 (14)	0.0430 (11)	−0.0311 (11)
N21	0.0429 (6)	0.0480 (7)	0.0467 (7)	0.0047 (5)	0.0174 (5)	−0.0002 (5)
N22	0.0381 (7)	0.0600 (9)	0.0651 (9)	−0.0058 (7)	0.0148 (7)	−0.0136 (7)
C11	0.0825 (12)	0.0509 (9)	0.0633 (10)	−0.0215 (9)	0.0475 (10)	−0.0106 (8)
C12	0.1049 (16)	0.0496 (10)	0.0838 (14)	−0.0309 (10)	0.0550 (13)	−0.0085 (9)
C13	0.0738 (11)	0.0421 (8)	0.0551 (10)	−0.0033 (8)	0.0192 (9)	−0.0041 (7)
C14	0.0550 (9)	0.0456 (8)	0.0495 (8)	−0.0087 (7)	0.0242 (7)	−0.0122 (6)
C21	0.0502 (9)	0.0693 (11)	0.0465 (9)	0.0003 (8)	0.0175 (7)	0.0075 (8)
C22	0.0775 (14)	0.121 (2)	0.0628 (12)	0.0000 (13)	0.0370 (11)	0.0190 (12)
C23	0.0653 (12)	0.1083 (19)	0.0937 (16)	0.0056 (12)	0.0495 (12)	0.0194 (14)
C24	0.0447 (9)	0.0847 (14)	0.0824 (13)	−0.0046 (9)	0.0255 (9)	−0.0003 (11)
C111	0.0322 (6)	0.0331 (6)	0.0394 (7)	−0.0036 (5)	0.0105 (5)	0.0005 (5)
C112	0.0577 (9)	0.0442 (8)	0.0423 (8)	−0.0116 (7)	0.0168 (7)	−0.0070 (6)
C113	0.0710 (12)	0.0640 (11)	0.0459 (9)	−0.0063 (9)	−0.0066 (9)	−0.0071 (8)
C114	0.0401 (9)	0.0711 (13)	0.0851 (15)	0.0055 (8)	−0.0035 (9)	−0.0100 (11)
C115	0.0383 (8)	0.0682 (11)	0.0867 (14)	0.0021 (8)	0.0240 (9)	−0.0051 (10)
C116	0.0390 (7)	0.0473 (8)	0.0508 (8)	−0.0013 (6)	0.0185 (6)	0.0010 (6)
C211	0.0400 (7)	0.0362 (7)	0.0388 (7)	0.0002 (5)	0.0138 (6)	−0.0023 (5)
C212	0.0389 (7)	0.0420 (7)	0.0405 (7)	−0.0038 (6)	0.0190 (6)	0.0014 (6)
C213	0.0458 (8)	0.0462 (8)	0.0578 (9)	0.0050 (6)	0.0286 (7)	0.0064 (7)
C214	0.0698 (11)	0.0390 (8)	0.0714 (11)	−0.0006 (7)	0.0425 (9)	−0.0054 (7)
C215	0.0622 (11)	0.0522 (9)	0.0772 (12)	−0.0157 (8)	0.0285 (10)	−0.0267 (9)
C216	0.0437 (8)	0.0553 (9)	0.0582 (10)	−0.0054 (7)	0.0129 (7)	−0.0162 (8)

Geometric parameters (Å, º)

S1—O12	1.4236 (13)	C21—H21A	0.9700
S1—O11	1.4369 (12)	C21—H21B	0.9700
S1—N11	1.6127 (12)	C22—C23	1.489 (3)
S1—C111	1.7496 (14)	C22—H22A	0.9700
S2—O22	1.4252 (12)	C22—H22B	0.9700
S2—O21	1.4330 (13)	C23—C24	1.514 (3)
S2—N21	1.6161 (14)	C23—H23A	0.9700
S2—C211	1.7558 (14)	C23—H23B	0.9700
N11—C11	1.462 (2)	C24—H24A	0.9700
N11—C14	1.4813 (19)	C24—H24B	0.9700
N12—C112	1.365 (3)	C111—C116	1.397 (2)
N12—H12C	0.78 (3)	C111—C112	1.402 (2)
N12—H12D	0.84 (3)	C112—C113	1.401 (3)
N21—C24	1.468 (2)	C113—C114	1.363 (3)
N21—C21	1.470 (2)	C113—H113	0.9300
N22—C212	1.372 (2)	C114—C115	1.373 (3)
N22—H21C	0.82 (2)	C114—H114	0.9300
N22—H21D	0.87 (2)	C115—C116	1.364 (2)
C11—C12	1.514 (2)	C115—H115	0.9300
C11—H11A	0.9700	C116—H116	0.9300
C11—H11B	0.9700	C211—C216	1.396 (2)
C12—C13	1.496 (3)	C211—C212	1.405 (2)
C12—H12A	0.9700	C212—C213	1.402 (2)
C12—H12B	0.9700	C213—C214	1.364 (2)
C13—C14	1.510 (2)	C213—H213	0.9300
C13—H13A	0.9700	C214—C215	1.381 (3)
C13—H13B	0.9700	C214—H214	0.9300
C14—H14A	0.9700	C215—C216	1.371 (2)
C14—H14B	0.9700	C215—H215	0.9300
C21—C22	1.497 (3)	C216—H216	0.9300

O12—S1—O11	118.78 (8)	C23—C22—C21	106.93 (17)
O12—S1—N11	106.62 (7)	C23—C22—H22A	110.3
O11—S1—N11	106.32 (7)	C21—C22—H22A	110.3
O12—S1—C111	107.62 (7)	C23—C22—H22B	110.3
O11—S1—C111	108.23 (7)	C21—C22—H22B	110.3
N11—S1—C111	109.00 (6)	H22A—C22—H22B	108.6
O22—S2—O21	118.33 (8)	C22—C23—C24	106.31 (16)
O22—S2—N21	106.53 (8)	C22—C23—H23A	110.5
O21—S2—N21	106.55 (7)	C24—C23—H23A	110.5
O22—S2—C211	108.20 (7)	C22—C23—H23B	110.5
O21—S2—C211	108.07 (7)	C24—C23—H23B	110.5
N21—S2—C211	108.86 (7)	H23A—C23—H23B	108.7
C11—N11—C14	110.00 (13)	N21—C24—C23	101.67 (16)
C11—N11—S1	121.83 (10)	N21—C24—H24A	111.4
C14—N11—S1	119.32 (10)	C23—C24—H24A	111.4

C112—N12—H12C	114 (2)	N21—C24—H24B	111.4
C112—N12—H12D	117 (2)	C23—C24—H24B	111.4
H12C—N12—H12D	117 (3)	H24A—C24—H24B	109.3
C24—N21—C21	107.31 (14)	C116—C111—C112	120.86 (14)
C24—N21—S2	120.18 (12)	C116—C111—S1	116.38 (11)
C21—N21—S2	120.19 (11)	C112—C111—S1	122.75 (11)
C212—N22—H21C	113.7 (16)	N12—C112—C113	119.99 (18)
C212—N22—H21D	114.3 (13)	N12—C112—C111	123.25 (17)
H21C—N22—H21D	122 (2)	C113—C112—C111	116.75 (16)
N11—C11—C12	101.23 (14)	C114—C113—C112	121.12 (17)
N11—C11—H11A	111.5	C114—C113—H113	119.4
C12—C11—H11A	111.5	C112—C113—H113	119.4
N11—C11—H11B	111.5	C113—C114—C115	121.80 (17)
C12—C11—H11B	111.5	C113—C114—H114	119.1
H11A—C11—H11B	109.3	C115—C114—H114	119.1
C13—C12—C11	104.33 (14)	C116—C115—C114	118.88 (18)
C13—C12—H12A	110.9	C116—C115—H115	120.6
C11—C12—H12A	110.9	C114—C115—H115	120.6
C13—C12—H12B	110.9	C115—C116—C111	120.58 (16)
C11—C12—H12B	110.9	C115—C116—H116	119.7
H12A—C12—H12B	108.9	C111—C116—H116	119.7
C12—C13—C14	105.24 (14)	C216—C211—C212	119.99 (14)
C12—C13—H13A	110.7	C216—C211—S2	117.42 (11)
C14—C13—H13A	110.7	C212—C211—S2	122.57 (11)
C12—C13—H13B	110.7	N22—C212—C213	119.25 (14)
C14—C13—H13B	110.7	N22—C212—C211	123.75 (14)
H13A—C13—H13B	108.8	C213—C212—C211	116.98 (13)
N11—C14—C13	104.53 (13)	C214—C213—C212	122.24 (15)
N11—C14—H14A	110.8	C214—C213—H213	118.9
C13—C14—H14A	110.8	C212—C213—H213	118.9
N11—C14—H14B	110.8	C213—C214—C215	120.37 (15)
C13—C14—H14B	110.8	C213—C214—H214	119.8
H14A—C14—H14B	108.9	C215—C214—H214	119.8
N21—C21—C22	104.32 (15)	C216—C215—C214	119.19 (16)
N21—C21—H21A	110.9	C216—C215—H215	120.4
C22—C21—H21A	110.9	C214—C215—H215	120.4
N21—C21—H21B	110.9	C215—C216—C211	121.23 (16)
C22—C21—H21B	110.9	C215—C216—H216	119.4
H21A—C21—H21B	108.9	C211—C216—H216	119.4

O12—S1—N11—C11	−171.19 (13)	O11—S1—C111—C112	29.11 (14)
O11—S1—N11—C11	−43.54 (15)	N11—S1—C111—C112	−86.12 (13)
C111—S1—N11—C11	72.91 (14)	C116—C111—C112—N12	−179.45 (18)
O12—S1—N11—C14	44.67 (13)	S1—C111—C112—N12	−1.1 (2)
O11—S1—N11—C14	172.32 (12)	C116—C111—C112—C113	−0.4 (2)
C111—S1—N11—C14	−71.23 (13)	S1—C111—C112—C113	178.01 (13)
O22—S2—N21—C24	−40.89 (16)	N12—C112—C113—C114	179.8 (2)
O21—S2—N21—C24	−168.09 (14)	C111—C112—C113—C114	0.7 (3)

C211—S2—N21—C24	75.59 (15)	C112—C113—C114—C115	−0.5 (3)
O22—S2—N21—C21	−178.33 (13)	C113—C114—C115—C116	0.0 (3)
O21—S2—N21—C21	54.47 (14)	C114—C115—C116—C111	0.4 (3)
C211—S2—N21—C21	−61.86 (14)	C112—C111—C116—C115	−0.2 (2)
C14—N11—C11—C12	−30.58 (19)	S1—C111—C116—C115	−178.64 (14)
S1—N11—C11—C12	−177.66 (14)	O22—S2—C211—C216	23.95 (15)
N11—C11—C12—C13	38.2 (2)	O21—S2—C211—C216	153.20 (13)
C11—C12—C13—C14	−32.5 (2)	N21—S2—C211—C216	−91.45 (14)
C11—N11—C14—C13	11.09 (18)	O22—S2—C211—C212	−157.40 (13)
S1—N11—C14—C13	159.11 (12)	O21—S2—C211—C212	−28.14 (14)
C12—C13—C14—N11	13.63 (19)	N21—S2—C211—C212	87.21 (13)
C24—N21—C21—C22	31.2 (2)	C216—C211—C212—N22	178.30 (16)
S2—N21—C21—C22	173.45 (15)	S2—C211—C212—N22	−0.3 (2)
N21—C21—C22—C23	−11.7 (3)	C216—C211—C212—C213	−0.2 (2)
C21—C22—C23—C24	−11.0 (3)	S2—C211—C212—C213	−178.81 (11)
C21—N21—C24—C23	−37.5 (2)	N22—C212—C213—C214	−178.60 (15)
S2—N21—C24—C23	−179.70 (14)	C211—C212—C213—C214	0.0 (2)
C22—C23—C24—N21	29.2 (2)	C212—C213—C214—C215	0.3 (3)
O12—S1—C111—C116	−22.94 (13)	C213—C214—C215—C216	−0.3 (3)
O11—S1—C111—C116	−152.46 (11)	C214—C215—C216—C211	0.0 (3)
N11—S1—C111—C116	92.31 (12)	C212—C211—C216—C215	0.2 (3)
O12—S1—C111—C112	158.62 (12)	S2—C211—C216—C215	178.89 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N12—H12*C*···O21	0.78 (3)	2.39 (3)	3.121 (2)	155 (3)
N12—H12*D*···O11	0.84 (3)	2.10 (3)	2.776 (3)	138 (3)
N22—H21*C*···O11^{i}	0.82 (2)	2.30 (2)	3.106 (2)	168 (2)
N22—H21*D*···O21	0.87 (2)	2.07 (2)	2.783 (2)	138.8 (17)
C114—H114···O22^{ii}	0.93	2.59	3.391 (2)	144
C116—H116···O12	0.93	2.46	2.8505 (19)	105
C216—H216···O22	0.93	2.52	2.897 (2)	105

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

2-(Piperidine-1-sulfonyl)aniline (ka115)

Crystal data

$C_{11}H_{16}N_2O_2S$
M_r = 240.32
Orthorhombic, *Pbca*
a = 11.1747 (4) Å
b = 10.4850 (4) Å
c = 20.1368 (8) Å
V = 2359.36 (15) Å^3
Z = 8
$F(000)$ = 1024

D_x = 1.353 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9577 reflections
θ = 2.9–28.3°
μ = 0.26 mm^{-1}
T = 200 K
Block, colourless
0.49 × 0.26 × 0.25 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: numerical (SADABS; Bruker, 2008)
T_{min} = 0.944, T_{max} = 1.000

57075 measured reflections
2935 independent reflections
2583 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 28.3°, θ_{min} = 2.0°
$h = -14 \rightarrow 14$
$k = -13 \rightarrow 13$
$l = -26 \rightarrow 26$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.087$
$S = 1.05$
2935 reflections
153 parameters
0 restraints
Primary atom site location: dual

Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0456P)^2 + 0.8126P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Special details

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

Refinement. Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2 U_{eq}(C).
The nitrogen-bound H atoms were located on a difference map and refined freely.
Single-crystal X-ray diffraction (SC-XRD) data were collected at 200 or 296 K on a Bruker APEXII CCD diffractometer with graphite-monochromated Mo *Kα* radiation using the *APEX2* data collection software and *SAINT* (Bruker 2012) for cell refinement and data reduction. The structures were solved by dual-space methods applying *SHELXT2018* (Sheldrick, 2015*a*) and refined by least-squares procedures using *SHELXL2018* (Sheldrick, 2015*b*). Data were corrected for absorption effects using the numerical method implemented in *SADABS* (Bruker 2012). All non-H atoms were refined anisotropically. The crystal structure diagrams were drawn with *ORTEP-3 for Windows* (Farrugia 2012).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.66247 (2)	0.46255 (3)	0.35628 (2)	0.02489 (10)
O1	0.78413 (7)	0.45299 (8)	0.37905 (5)	0.0331 (2)
O2	0.60807 (8)	0.58657 (8)	0.35302 (5)	0.0344 (2)
N1	0.48648 (11)	0.51544 (13)	0.23579 (7)	0.0417 (3)
N2	0.58073 (8)	0.37768 (9)	0.40733 (5)	0.0257 (2)
C11	0.65595 (9)	0.38973 (11)	0.27755 (6)	0.0245 (2)
C12	0.56770 (10)	0.41892 (11)	0.22957 (6)	0.0281 (2)
C13	0.56702 (11)	0.34467 (13)	0.17139 (6)	0.0340 (3)
H13	0.509126	0.361787	0.138016	0.041*
C14	0.64803 (12)	0.24808 (13)	0.16172 (6)	0.0357 (3)
H14	0.644395	0.199006	0.122112	0.043*

C15	0.73532 (11)	0.22069 (12)	0.20879 (6)	0.0336 (3)
H15	0.791553	0.154219	0.201449	0.040*
C16	0.73871 (10)	0.29170 (11)	0.26622 (6)	0.0280 (2)
H16	0.798089	0.273989	0.298703	0.034*
C21	0.61856 (11)	0.24349 (12)	0.41533 (6)	0.0320 (3)
H21A	0.706433	0.239499	0.421235	0.038*
H21B	0.597532	0.194456	0.374968	0.038*
C22	0.55700 (14)	0.18533 (15)	0.47531 (7)	0.0429 (3)
H22A	0.584669	0.229165	0.516052	0.051*
H22B	0.579347	0.094219	0.478891	0.051*
C23	0.42189 (13)	0.19665 (15)	0.47012 (8)	0.0460 (3)
H23A	0.392700	0.144432	0.432495	0.055*
H23B	0.384336	0.164250	0.511330	0.055*
C24	0.38708 (12)	0.33508 (15)	0.45955 (8)	0.0448 (3)
H24A	0.408917	0.385359	0.499357	0.054*
H24B	0.299337	0.341143	0.453508	0.054*
C25	0.44964 (11)	0.39042 (13)	0.39903 (7)	0.0358 (3)
H25A	0.423672	0.344456	0.358573	0.043*
H25B	0.428110	0.481486	0.393957	0.043*
H1A	0.4305 (18)	0.5177 (17)	0.2083 (9)	0.050 (5)*
H1B	0.4888 (14)	0.5639 (15)	0.2697 (8)	0.037 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.02117 (15)	0.02347 (15)	0.03003 (16)	−0.00296 (9)	−0.00057 (10)	−0.00017 (10)
O1	0.0226 (4)	0.0393 (5)	0.0373 (5)	−0.0060 (3)	−0.0045 (3)	−0.0021 (4)
O2	0.0350 (5)	0.0231 (4)	0.0451 (5)	−0.0009 (3)	0.0032 (4)	0.0006 (4)
N1	0.0315 (6)	0.0460 (7)	0.0476 (7)	0.0119 (5)	−0.0111 (5)	0.0009 (6)
N2	0.0240 (4)	0.0249 (5)	0.0280 (5)	−0.0019 (4)	0.0011 (4)	0.0007 (4)
C11	0.0214 (5)	0.0253 (5)	0.0267 (5)	−0.0027 (4)	0.0000 (4)	0.0037 (4)
C12	0.0223 (5)	0.0299 (6)	0.0319 (6)	−0.0030 (4)	−0.0010 (4)	0.0078 (5)
C13	0.0318 (6)	0.0419 (7)	0.0284 (6)	−0.0068 (5)	−0.0052 (5)	0.0056 (5)
C14	0.0425 (7)	0.0369 (7)	0.0277 (6)	−0.0089 (5)	0.0021 (5)	−0.0001 (5)
C15	0.0360 (6)	0.0300 (6)	0.0349 (6)	0.0010 (5)	0.0073 (5)	0.0021 (5)
C16	0.0251 (5)	0.0287 (6)	0.0303 (5)	0.0002 (4)	0.0008 (4)	0.0056 (4)
C21	0.0366 (6)	0.0278 (6)	0.0315 (6)	0.0029 (5)	0.0030 (5)	0.0057 (5)
C22	0.0485 (8)	0.0458 (8)	0.0344 (7)	0.0000 (6)	0.0024 (6)	0.0149 (6)
C23	0.0457 (8)	0.0512 (8)	0.0409 (7)	−0.0114 (7)	0.0090 (6)	0.0132 (6)
C24	0.0321 (6)	0.0538 (9)	0.0486 (8)	−0.0040 (6)	0.0118 (6)	0.0041 (7)
C25	0.0234 (5)	0.0384 (7)	0.0455 (7)	−0.0019 (5)	0.0014 (5)	0.0089 (6)

Geometric parameters (Å, º)

S1—O2	1.4369 (9)	C15—H15	0.9500
S1—O1	1.4382 (8)	C16—H16	0.9500
S1—N2	1.6381 (10)	C21—C22	1.5178 (17)
S1—C11	1.7611 (12)	C21—H21A	0.9900

N1—C12	1.3652 (17)	C21—H21B	0.9900
N1—H1A	0.836 (19)	C22—C23	1.518 (2)
N1—H1B	0.852 (17)	C22—H22A	0.9900
N2—C21	1.4780 (15)	C22—H22B	0.9900
N2—C25	1.4804 (15)	C23—C24	1.518 (2)
C11—C16	1.4014 (16)	C23—H23A	0.9900
C11—C12	1.4141 (15)	C23—H23B	0.9900
C12—C13	1.4066 (18)	C24—C25	1.5200 (18)
C13—C14	1.3723 (19)	C24—H24A	0.9900
C13—H13	0.9500	C24—H24B	0.9900
C14—C15	1.3901 (18)	C25—H25A	0.9900
C14—H14	0.9500	C25—H25B	0.9900
C15—C16	1.3759 (17)		

O2—S1—O1	118.52 (5)	N2—C21—C22	109.85 (11)
O2—S1—N2	106.51 (5)	N2—C21—H21A	109.7
O1—S1—N2	106.81 (5)	C22—C21—H21A	109.7
O2—S1—C11	109.50 (5)	N2—C21—H21B	109.7
O1—S1—C11	107.21 (5)	C22—C21—H21B	109.7
N2—S1—C11	107.84 (5)	H21A—C21—H21B	108.2
C12—N1—H1A	117.2 (12)	C21—C22—C23	111.39 (11)
C12—N1—H1B	119.7 (10)	C21—C22—H22A	109.4
H1A—N1—H1B	122.5 (16)	C23—C22—H22A	109.4
C21—N2—C25	112.41 (10)	C21—C22—H22B	109.4
C21—N2—S1	115.21 (8)	C23—C22—H22B	109.4
C25—N2—S1	115.59 (8)	H22A—C22—H22B	108.0
C16—C11—C12	120.52 (11)	C24—C23—C22	109.84 (12)
C16—C11—S1	115.92 (8)	C24—C23—H23A	109.7
C12—C11—S1	123.37 (9)	C22—C23—H23A	109.7
N1—C12—C13	118.89 (11)	C24—C23—H23B	109.7
N1—C12—C11	124.15 (12)	C22—C23—H23B	109.7
C13—C12—C11	116.94 (11)	H23A—C23—H23B	108.2
C14—C13—C12	121.54 (11)	C23—C24—C25	111.08 (12)
C14—C13—H13	119.2	C23—C24—H24A	109.4
C12—C13—H13	119.2	C25—C24—H24A	109.4
C13—C14—C15	121.24 (12)	C23—C24—H24B	109.4
C13—C14—H14	119.4	C25—C24—H24B	109.4
C15—C14—H14	119.4	H24A—C24—H24B	108.0
C16—C15—C14	118.73 (12)	N2—C25—C24	109.28 (11)
C16—C15—H15	120.6	N2—C25—H25A	109.8
C14—C15—H15	120.6	C24—C25—H25A	109.8
C15—C16—C11	121.03 (11)	N2—C25—H25B	109.8
C15—C16—H16	119.5	C24—C25—H25B	109.8
C11—C16—H16	119.5	H25A—C25—H25B	108.3

O2—S1—N2—C21	177.25 (8)	N1—C12—C13—C14	178.65 (12)
O1—S1—N2—C21	−55.17 (9)	C11—C12—C13—C14	0.20 (17)
C11—S1—N2—C21	59.79 (9)	C12—C13—C14—C15	−0.85 (19)

O2—S1—N2—C25	43.41 (10)	C13—C14—C15—C16	0.67 (19)
O1—S1—N2—C25	170.99 (9)	C14—C15—C16—C11	0.15 (18)
C11—S1—N2—C25	−74.05 (10)	C12—C11—C16—C15	−0.80 (17)
O2—S1—C11—C16	158.65 (8)	S1—C11—C16—C15	174.39 (9)
O1—S1—C11—C16	28.86 (10)	C25—N2—C21—C22	−58.70 (14)
N2—S1—C11—C16	−85.83 (9)	S1—N2—C21—C22	166.02 (9)
O2—S1—C11—C12	−26.31 (11)	N2—C21—C22—C23	55.96 (15)
O1—S1—C11—C12	−156.11 (9)	C21—C22—C23—C24	−54.92 (17)
N2—S1—C11—C12	89.20 (10)	C22—C23—C24—C25	55.71 (17)
C16—C11—C12—N1	−177.75 (12)	C21—N2—C25—C24	59.34 (14)
S1—C11—C12—N1	7.43 (17)	S1—N2—C25—C24	−165.56 (9)
C16—C11—C12—C13	0.61 (16)	C23—C24—C25—N2	−57.41 (16)
S1—C11—C12—C13	−174.21 (8)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.836 (19)	2.496 (19)	3.3000 (16)	161.8 (16)
N1—H1*B*···O2	0.852 (17)	2.156 (17)	2.8240 (17)	135.1 (14)
C16—H16···O1	0.95	2.48	2.8774 (15)	105
C16—H16···O2^{ii}	0.95	2.48	3.2576 (14)	139
C21—H21*A*···O1	0.99	2.55	2.9635 (15)	105
C25—H25*B*···O2	0.99	2.44	2.8675 (15)	106

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

2-(2,3-Dihydro-1*H*-indole-1-sulfonyl)aniline (ja189)

Crystal data

$C_{14}H_{14}N_2O_2S$
$M_r = 274.33$
Monoclinic, $P2_1/n$
$a = 9.7990$ (11) Å
$b = 10.2612$ (13) Å
$c = 13.2010$ (16) Å
$\beta = 100.682$ (5)°
$V = 1304.4$ (3) Å^3
$Z = 4$

$F(000) = 576$
$D_x = 1.397$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9932 reflections
$\theta = 2.4$–28.4°
$\mu = 0.25$ mm^{-1}
$T = 296$ K
Block, colourless
0.54 × 0.34 × 0.34 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: numerical (SADABS; Bruker, 2008)
$T_{min} = 0.925$, $T_{max} = 1.000$

28600 measured reflections
3275 independent reflections
2726 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 28.7°$, $\theta_{min} = 2.4°$
$h = -13 \rightarrow 13$
$k = -13 \rightarrow 12$
$l = -16 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.113$
$S = 1.05$
3275 reflections
180 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.053P)^2 + 0.4178P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}

Special details

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

Refinement. Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2 U_{eq}(C).
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Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.38977 (4)	0.26951 (4)	0.46693 (3)	0.04455 (13)
O1	0.46972 (13)	0.21097 (14)	0.55683 (9)	0.0646 (4)
O2	0.45941 (11)	0.31734 (14)	0.38820 (9)	0.0593 (3)
N1	0.31329 (14)	0.39561 (13)	0.50488 (10)	0.0476 (3)
N2	0.1992 (2)	0.27928 (18)	0.25271 (12)	0.0630 (4)
C1	0.20394 (18)	0.60239 (16)	0.50168 (14)	0.0536 (4)
H1A	0.262936	0.675102	0.528584	0.064*
H1B	0.119055	0.635928	0.460230	0.064*
C2	0.27807 (18)	0.51224 (17)	0.43895 (13)	0.0532 (4)
H2A	0.218044	0.489119	0.374487	0.064*
H2B	0.361389	0.553051	0.423985	0.064*
C11	0.23714 (14)	0.39828 (15)	0.58651 (11)	0.0418 (3)
C12	0.17276 (15)	0.51848 (15)	0.58744 (12)	0.0457 (3)
C13	0.09543 (18)	0.5451 (2)	0.66236 (15)	0.0621 (5)
H13	0.052108	0.625529	0.663818	0.074*
C14	0.08286 (19)	0.4512 (2)	0.73524 (14)	0.0681 (5)
H14	0.030634	0.468525	0.785769	0.082*
C15	0.1472 (2)	0.3326 (2)	0.73330 (13)	0.0626 (5)
H15	0.137895	0.270445	0.782820	0.075*
C16	0.22563 (19)	0.30382 (18)	0.65894 (13)	0.0542 (4)
H16	0.269140	0.223417	0.657880	0.065*

C21	0.26219 (15)	0.15726 (15)	0.41371 (11)	0.0437 (3)
C22	0.18015 (15)	0.17834 (16)	0.31512 (11)	0.0460 (3)
C23	0.07113 (18)	0.0912 (2)	0.28389 (15)	0.0631 (5)
H23	0.012937	0.103508	0.220484	0.076*
C24	0.0478 (3)	−0.0110 (2)	0.3436 (2)	0.0826 (7)
H24	−0.026770	−0.066288	0.320608	0.099*
C25	0.1320 (3)	−0.0348 (2)	0.4375 (2)	0.0866 (7)
H25	0.116577	−0.106941	0.476522	0.104*
C26	0.2397 (2)	0.04998 (18)	0.47264 (15)	0.0658 (5)
H26	0.297316	0.035268	0.535935	0.079*
H2C	0.283 (3)	0.308 (3)	0.259 (2)	0.093 (8)*
H2D	0.143 (2)	0.282 (2)	0.1916 (19)	0.079 (7)*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.03429 (19)	0.0609 (3)	0.0368 (2)	0.00384 (14)	0.00238 (14)	−0.00607 (15)
O1	0.0543 (7)	0.0854 (9)	0.0466 (6)	0.0190 (6)	−0.0106 (5)	−0.0049 (6)
O2	0.0413 (6)	0.0893 (9)	0.0505 (6)	−0.0069 (6)	0.0168 (5)	−0.0118 (6)
N1	0.0533 (7)	0.0477 (7)	0.0460 (7)	−0.0005 (5)	0.0201 (6)	0.0009 (5)
N2	0.0658 (10)	0.0767 (11)	0.0394 (7)	−0.0037 (8)	−0.0085 (7)	0.0020 (7)
C1	0.0514 (9)	0.0459 (8)	0.0602 (10)	−0.0043 (7)	0.0018 (7)	−0.0025 (7)
C2	0.0580 (9)	0.0544 (9)	0.0467 (8)	−0.0048 (7)	0.0082 (7)	0.0049 (7)
C11	0.0386 (7)	0.0491 (8)	0.0382 (7)	−0.0074 (6)	0.0089 (5)	−0.0069 (6)
C12	0.0367 (7)	0.0504 (8)	0.0478 (8)	−0.0058 (6)	0.0021 (6)	−0.0117 (6)
C13	0.0494 (9)	0.0730 (12)	0.0643 (11)	0.0018 (8)	0.0120 (8)	−0.0241 (9)
C14	0.0543 (10)	0.1020 (16)	0.0522 (10)	−0.0112 (10)	0.0210 (8)	−0.0224 (10)
C15	0.0631 (10)	0.0831 (13)	0.0444 (8)	−0.0183 (10)	0.0172 (8)	−0.0028 (9)
C16	0.0612 (10)	0.0570 (9)	0.0472 (8)	−0.0061 (8)	0.0174 (7)	0.0004 (7)
C21	0.0431 (7)	0.0464 (8)	0.0408 (7)	0.0043 (6)	0.0059 (6)	−0.0059 (6)
C22	0.0404 (7)	0.0561 (9)	0.0410 (7)	0.0047 (6)	0.0063 (6)	−0.0137 (7)
C23	0.0492 (9)	0.0793 (13)	0.0604 (10)	−0.0090 (8)	0.0090 (8)	−0.0296 (9)
C24	0.0846 (15)	0.0786 (14)	0.0913 (16)	−0.0327 (12)	0.0334 (13)	−0.0391 (13)
C25	0.126 (2)	0.0500 (11)	0.0938 (17)	−0.0192 (12)	0.0453 (16)	−0.0082 (11)
C26	0.0888 (14)	0.0504 (10)	0.0582 (10)	0.0058 (9)	0.0139 (9)	0.0003 (8)

Geometric parameters (Å, º)

S1—O1	1.4277 (12)	C13—C14	1.383 (3)
S1—O2	1.4317 (12)	C13—H13	0.9300
S1—N1	1.6205 (14)	C14—C15	1.373 (3)
S1—C21	1.7486 (16)	C14—H14	0.9300
N1—C11	1.4203 (18)	C15—C16	1.387 (2)
N1—C2	1.482 (2)	C15—H15	0.9300
N2—C22	1.358 (2)	C16—H16	0.9300
N2—H2C	0.86 (3)	C21—C26	1.389 (2)
N2—H2D	0.89 (2)	C21—C22	1.414 (2)
C1—C12	1.498 (2)	C22—C23	1.396 (2)

C1—C2	1.514 (2)	C23—C24	1.357 (3)
C1—H1A	0.9700	C23—H23	0.9300
C1—H1B	0.9700	C24—C25	1.378 (4)
C2—H2A	0.9700	C24—H24	0.9300
C2—H2B	0.9700	C25—C26	1.380 (3)
C11—C16	1.381 (2)	C25—H25	0.9300
C11—C12	1.386 (2)	C26—H26	0.9300
C12—C13	1.380 (2)		

O1—S1—O2	119.06 (8)	C12—C13—C14	119.49 (18)
O1—S1—N1	107.04 (7)	C12—C13—H13	120.3
O2—S1—N1	105.10 (8)	C14—C13—H13	120.3
O1—S1—C21	107.35 (8)	C15—C14—C13	120.27 (16)
O2—S1—C21	109.61 (7)	C15—C14—H14	119.9
N1—S1—C21	108.24 (7)	C13—C14—H14	119.9
C11—N1—C2	109.39 (13)	C14—C15—C16	121.27 (18)
C11—N1—S1	126.02 (11)	C14—C15—H15	119.4
C2—N1—S1	122.27 (11)	C16—C15—H15	119.4
C22—N2—H2C	115.9 (18)	C11—C16—C15	117.89 (18)
C22—N2—H2D	116.2 (15)	C11—C16—H16	121.1
H2C—N2—H2D	120 (2)	C15—C16—H16	121.1
C12—C1—C2	104.35 (13)	C26—C21—C22	120.97 (16)
C12—C1—H1A	110.9	C26—C21—S1	118.22 (13)
C2—C1—H1A	110.9	C22—C21—S1	120.75 (12)
C12—C1—H1B	110.9	N2—C22—C23	119.58 (16)
C2—C1—H1B	110.9	N2—C22—C21	123.81 (15)
H1A—C1—H1B	108.9	C23—C22—C21	116.57 (17)
N1—C2—C1	104.80 (13)	C24—C23—C22	121.75 (19)
N1—C2—H2A	110.8	C24—C23—H23	119.1
C1—C2—H2A	110.8	C22—C23—H23	119.1
N1—C2—H2B	110.8	C23—C24—C25	121.5 (2)
C1—C2—H2B	110.8	C23—C24—H24	119.3
H2A—C2—H2B	108.9	C25—C24—H24	119.3
C16—C11—C12	121.52 (14)	C24—C25—C26	118.9 (2)
C16—C11—N1	129.33 (15)	C24—C25—H25	120.5
C12—C11—N1	109.13 (13)	C26—C25—H25	120.5
C13—C12—C11	119.57 (16)	C25—C26—C21	120.1 (2)
C13—C12—C1	130.07 (16)	C25—C26—H26	119.9
C11—C12—C1	110.35 (14)	C21—C26—H26	119.9

O1—S1—N1—C11	−48.70 (15)	C13—C14—C15—C16	−0.1 (3)
O2—S1—N1—C11	−176.21 (12)	C12—C11—C16—C15	0.0 (2)
C21—S1—N1—C11	66.72 (14)	N1—C11—C16—C15	178.04 (15)
O1—S1—N1—C2	150.47 (13)	C14—C15—C16—C11	−0.1 (3)
O2—S1—N1—C2	22.96 (14)	O1—S1—C21—C26	12.71 (16)
C21—S1—N1—C2	−94.10 (13)	O2—S1—C21—C26	143.37 (14)
C11—N1—C2—C1	14.13 (17)	N1—S1—C21—C26	−102.51 (14)
S1—N1—C2—C1	177.78 (11)	O1—S1—C21—C22	−169.99 (12)

C12—C1—C2—N1	−12.92 (16)	O2—S1—C21—C22	−39.34 (14)
C2—N1—C11—C16	172.19 (16)	N1—S1—C21—C22	74.79 (13)
S1—N1—C11—C16	9.3 (2)	C26—C21—C22—N2	−178.22 (17)
C2—N1—C11—C12	−9.61 (16)	S1—C21—C22—N2	4.6 (2)
S1—N1—C11—C12	−172.49 (11)	C26—C21—C22—C23	4.0 (2)
C16—C11—C12—C13	0.1 (2)	S1—C21—C22—C23	−173.18 (12)
N1—C11—C12—C13	−178.25 (13)	N2—C22—C23—C24	−179.93 (19)
C16—C11—C12—C1	179.21 (14)	C21—C22—C23—C24	−2.1 (3)
N1—C11—C12—C1	0.84 (16)	C22—C23—C24—C25	−1.0 (3)
C2—C1—C12—C13	−173.18 (16)	C23—C24—C25—C26	2.1 (4)
C2—C1—C12—C11	7.85 (17)	C24—C25—C26—C21	−0.1 (3)
C11—C12—C13—C14	−0.2 (2)	C22—C21—C26—C25	−3.0 (3)
C1—C12—C13—C14	−179.12 (16)	S1—C21—C26—C25	174.28 (16)
C12—C13—C14—C15	0.2 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*C*···O2	0.86 (3)	2.19 (3)	2.857 (2)	134 (2)
N2—H2*D*···O1^{i}	0.89 (2)	2.22 (3)	3.098 (2)	169 (2)
C15—H15···O2^{ii}	0.93	2.59	3.366 (2)	141
C16—H16···O1	0.93	2.58	3.105 (2)	117
C26—H26···O1	0.93	2.45	2.849 (3)	106

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

supporting information

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N-Cycloamino substituent effects on the packing architecture of *ortho*-sulfanilamide molecular crystals and their *in silico* carbonic anhydrase II and IX inhibitory activities

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Computing details

For all structures, data collection: *APEX2* (Bruker, 2011); cell refinement: *APEX2* (Bruker, 2011); data reduction: *SAINT* (Bruker, 2012); program(s) used to solve structure: SHELXT2018 (Sheldrick, 2015*a*); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015*b*) and ShelXle (Hübschle *et al.*, 2011); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2020) and *Mercury* (Macrae *et al.*, 2020).

1-[(2-Nitrophenyl)sulfonyl]pyrrolidine (ka097)

Crystal data

$C_{10}H_{12}N_2O_4S$	$F(000) = 536$
$M_r = 256.28$	$D_x = 1.493$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 8.6173\ (5)$ Å	Cell parameters from 9888 reflections
$b = 14.6662\ (9)$ Å	$\theta = 2.7$–$28.3°$
$c = 9.4885\ (6)$ Å	$\mu = 0.29$ mm^{-1}
$\beta = 108.075\ (3)°$	$T = 200$ K
$V = 1140.01\ (12)$ Å^3	Block, yellow
$Z = 4$	0.67 × 0.67 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer	24329 measured reflections
Radiation source: sealed tube	2849 independent reflections
Graphite monochromator	2508 reflections with $I > 2\sigma(I)$
Detector resolution: 8.3333 pixels mm^{-1}	$R_{int} = 0.018$
φ and ω scans	$\theta_{max} = 28.4°$, $\theta_{min} = 2.7°$
Absorption correction: numerical (SADABS; Bruker, 2008)	$h = -11 \rightarrow 11$
$T_{min} = 0.934$, $T_{max} = 1.000$	$k = -17 \rightarrow 19$
	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	$S = 1.05$
Least-squares matrix: full	2849 reflections
$R[F^2 > 2\sigma(F^2)] = 0.041$	149 parameters
$wR(F^2) = 0.111$	20 restraints

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

$w = 1/[\sigma^2(F_o^2) + (0.0517P)^2 + 0.7926P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ e Å^{-3}

Special details

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

Refinement. Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2 U_{eq}(C).
The pyrrolidine group is disordered necessitating the use of the restraints SADI, EADP and SAME.
Single-crystal X-ray diffraction (SC-XRD) data were collected at 200 or 296 K on a Bruker APEXII CCD diffractometer with graphite-monochromated Mo *Kα* radiation using the *APEX2* data collection software and *SAINT* (Bruker 2012) for cell refinement and data reduction. The structures were solved by dual-space methods applying *SHELXT2018* (Sheldrick, 2015*a*) and refined by least-squares procedures using *SHELXL2018* (Sheldrick, 2015*b*). Data were corrected for absorption effects using the numerical method implemented in *SADABS* (Bruker 2012). All non-H atoms were refined anisotropically. The crystal structure diagrams were drawn with *ORTEP-3 for Windows* (Farrugia 2012).

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1	0.49089 (5)	0.66797 (3)	0.32216 (4)	0.02954 (13)	
O1	0.52737 (15)	0.62543 (9)	0.46416 (13)	0.0360 (3)	
O2	0.41164 (17)	0.75483 (10)	0.29726 (18)	0.0481 (4)	
O3	0.91161 (16)	0.56570 (9)	0.57998 (13)	0.0374 (3)	
O4	0.74333 (16)	0.49165 (8)	0.40041 (15)	0.0387 (3)	
N1	0.3849 (2)	0.59886 (12)	0.20100 (17)	0.0424 (4)	
N2	0.82322 (16)	0.55892 (9)	0.45200 (15)	0.0261 (3)	
C21	0.67960 (18)	0.68563 (10)	0.28603 (17)	0.0243 (3)	
C22	0.82125 (18)	0.63575 (9)	0.35184 (16)	0.0233 (3)	
C23	0.9678 (2)	0.65569 (11)	0.32834 (19)	0.0300 (3)	
H23	1.063007	0.621352	0.376473	0.036*	
C24	0.9742 (2)	0.72656 (12)	0.2335 (2)	0.0347 (4)	
H24	1.073735	0.740356	0.214723	0.042*	
C25	0.8355 (2)	0.77707 (12)	0.1663 (2)	0.0348 (4)	
H25	0.839913	0.825347	0.100842	0.042*	
C26	0.6899 (2)	0.75768 (11)	0.19374 (18)	0.0304 (3)	
H26	0.596179	0.793966	0.149089	0.036*	
C11	0.3771 (10)	0.4994 (5)	0.2187 (8)	0.0525 (5)	0.579 (7)
H11A	0.365697	0.483433	0.316363	0.063*	0.579 (7)
H11B	0.475419	0.469108	0.207784	0.063*	0.579 (7)
C12	0.2269 (6)	0.4739 (3)	0.0945 (5)	0.0525 (5)	0.579 (7)
H12A	0.126167	0.490743	0.117592	0.063*	0.579 (7)
H12B	0.224619	0.407828	0.072438	0.063*	0.579 (7)
C13	0.2474 (6)	0.5302 (4)	−0.0321 (5)	0.0525 (5)	0.579 (7)
H13A	0.142720	0.536536	−0.112750	0.063*	0.579 (7)

H13B	0.329694	0.502920	−0.072615	0.063*	0.579 (7)
C14	0.3052 (11)	0.6216 (4)	0.0416 (7)	0.0525 (5)	0.579 (7)
H14A	0.384050	0.650475	−0.001515	0.063*	0.579 (7)
H14B	0.211994	0.663509	0.029701	0.063*	0.579 (7)
C15	0.3620 (14)	0.5024 (7)	0.2240 (11)	0.0525 (5)	0.421 (7)
H15A	0.289652	0.492782	0.285820	0.063*	0.421 (7)
H15B	0.467484	0.471263	0.270293	0.063*	0.421 (7)
C16	0.2804 (9)	0.4696 (4)	0.0620 (7)	0.0525 (5)	0.421 (7)
H16A	0.362038	0.441474	0.021428	0.063*	0.421 (7)
H16B	0.193459	0.424491	0.057238	0.063*	0.421 (7)
C17	0.2083 (8)	0.5567 (5)	−0.0247 (7)	0.0525 (5)	0.421 (7)
H17A	0.207484	0.551559	−0.128998	0.063*	0.421 (7)
H17B	0.094759	0.566045	−0.023970	0.063*	0.421 (7)
C18	0.3140 (15)	0.6354 (6)	0.0501 (10)	0.0525 (5)	0.421 (7)
H18A	0.399357	0.649069	0.003320	0.063*	0.421 (7)
H18B	0.248645	0.691003	0.049588	0.063*	0.421 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0263 (2)	0.0333 (2)	0.0318 (2)	0.00668 (14)	0.01314 (16)	0.00676 (15)
O1	0.0363 (6)	0.0479 (7)	0.0283 (6)	0.0048 (5)	0.0166 (5)	0.0052 (5)
O2	0.0392 (7)	0.0428 (7)	0.0700 (10)	0.0185 (6)	0.0280 (7)	0.0142 (7)
O3	0.0405 (7)	0.0391 (7)	0.0282 (6)	0.0012 (5)	0.0045 (5)	0.0077 (5)
O4	0.0373 (7)	0.0259 (6)	0.0495 (8)	−0.0061 (5)	0.0087 (6)	0.0034 (5)
N1	0.0400 (8)	0.0479 (9)	0.0327 (8)	−0.0083 (7)	0.0017 (6)	0.0123 (7)
N2	0.0242 (6)	0.0237 (6)	0.0306 (7)	0.0030 (5)	0.0090 (5)	0.0045 (5)
C21	0.0261 (7)	0.0239 (7)	0.0246 (7)	0.0026 (5)	0.0103 (6)	0.0011 (5)
C22	0.0276 (7)	0.0195 (6)	0.0231 (7)	0.0016 (5)	0.0081 (6)	0.0009 (5)
C23	0.0270 (8)	0.0281 (7)	0.0363 (9)	0.0030 (6)	0.0116 (7)	0.0023 (6)
C24	0.0350 (8)	0.0329 (8)	0.0428 (9)	−0.0009 (7)	0.0218 (7)	0.0038 (7)
C25	0.0446 (10)	0.0287 (8)	0.0375 (9)	0.0036 (7)	0.0222 (8)	0.0087 (7)
C26	0.0351 (8)	0.0272 (7)	0.0305 (8)	0.0080 (6)	0.0125 (7)	0.0075 (6)
C11	0.0476 (9)	0.0604 (10)	0.0421 (7)	−0.0101 (7)	0.0034 (6)	0.0040 (7)
C12	0.0476 (9)	0.0604 (10)	0.0421 (7)	−0.0101 (7)	0.0034 (6)	0.0040 (7)
C13	0.0476 (9)	0.0604 (10)	0.0421 (7)	−0.0101 (7)	0.0034 (6)	0.0040 (7)
C14	0.0476 (9)	0.0604 (10)	0.0421 (7)	−0.0101 (7)	0.0034 (6)	0.0040 (7)
C15	0.0476 (9)	0.0604 (10)	0.0421 (7)	−0.0101 (7)	0.0034 (6)	0.0040 (7)
C16	0.0476 (9)	0.0604 (10)	0.0421 (7)	−0.0101 (7)	0.0034 (6)	0.0040 (7)
C17	0.0476 (9)	0.0604 (10)	0.0421 (7)	−0.0101 (7)	0.0034 (6)	0.0040 (7)
C18	0.0476 (9)	0.0604 (10)	0.0421 (7)	−0.0101 (7)	0.0034 (6)	0.0040 (7)

Geometric parameters (Å, º)

S1—O1	1.4285 (12)	C11—H11A	0.9900
S1—O2	1.4300 (13)	C11—H11B	0.9900
S1—N1	1.5913 (17)	C12—C13	1.513 (6)
S1—C21	1.7818 (15)	C12—H12A	0.9900

O3—N2	1.2209 (18)	C12—H12B	0.9900
O4—N2	1.2157 (18)	C13—C14	1.522 (6)
N1—C15	1.454 (10)	C13—H13A	0.9900
N1—C11	1.472 (8)	C13—H13B	0.9900
N1—C18	1.472 (8)	C14—H14A	0.9900
N1—C14	1.492 (6)	C14—H14B	0.9900
N2—C22	1.4706 (18)	C15—C16	1.555 (10)
C21—C26	1.393 (2)	C15—H15A	0.9900
C21—C22	1.394 (2)	C15—H15B	0.9900
C22—C23	1.380 (2)	C16—C17	1.543 (8)
C23—C24	1.387 (2)	C16—H16A	0.9900
C23—H23	0.9500	C16—H16B	0.9900
C24—C25	1.382 (2)	C17—C18	1.505 (8)
C24—H24	0.9500	C17—H17A	0.9900
C25—C26	1.388 (2)	C17—H17B	0.9900
C25—H25	0.9500	C18—H18A	0.9900
C26—H26	0.9500	C18—H18B	0.9900
C11—C12	1.503 (8)		

O1—S1—O2	119.93 (9)	C13—C12—H12A	111.4
O1—S1—N1	108.19 (8)	C11—C12—H12B	111.4
O2—S1—N1	108.08 (9)	C13—C12—H12B	111.4
O1—S1—C21	107.34 (7)	H12A—C12—H12B	109.3
O2—S1—C21	104.85 (8)	C12—C13—C14	102.6 (4)
N1—S1—C21	107.90 (8)	C12—C13—H13A	111.2
C15—N1—C18	117.8 (5)	C14—C13—H13A	111.2
C11—N1—C14	108.3 (4)	C12—C13—H13B	111.2
C15—N1—S1	125.8 (4)	C14—C13—H13B	111.2
C11—N1—S1	125.8 (3)	H13A—C13—H13B	109.2
C18—N1—S1	116.2 (3)	N1—C14—C13	104.5 (4)
C14—N1—S1	124.8 (2)	N1—C14—H14A	110.8
O4—N2—O3	124.55 (14)	C13—C14—H14A	110.8
O4—N2—C22	118.04 (13)	N1—C14—H14B	110.8
O3—N2—C22	117.32 (13)	C13—C14—H14B	110.8
C26—C21—C22	117.54 (14)	H14A—C14—H14B	108.9
C26—C21—S1	118.12 (12)	N1—C15—C16	101.2 (6)
C22—C21—S1	124.21 (11)	N1—C15—H15A	111.5
C23—C22—C21	122.31 (14)	C16—C15—H15A	111.5
C23—C22—N2	116.04 (13)	N1—C15—H15B	111.5
C21—C22—N2	121.64 (13)	C16—C15—H15B	111.5
C22—C23—C24	119.09 (15)	H15A—C15—H15B	109.4
C22—C23—H23	120.5	C17—C16—C15	104.7 (6)
C24—C23—H23	120.5	C17—C16—H16A	110.8
C25—C24—C23	119.87 (15)	C15—C16—H16A	110.8
C25—C24—H24	120.1	C17—C16—H16B	110.8
C23—C24—H24	120.1	C15—C16—H16B	110.8
C24—C25—C26	120.41 (15)	H16A—C16—H16B	108.9
C24—C25—H25	119.8	C18—C17—C16	107.4 (6)

C26—C25—H25	119.8	C18—C17—H17A	110.2
C25—C26—C21	120.75 (15)	C16—C17—H17A	110.2
C25—C26—H26	119.6	C18—C17—H17B	110.2
C21—C26—H26	119.6	C16—C17—H17B	110.2
N1—C11—C12	102.5 (5)	H17A—C17—H17B	108.5
N1—C11—H11A	111.3	N1—C18—C17	100.8 (5)
C12—C11—H11A	111.3	N1—C18—H18A	111.6
N1—C11—H11B	111.3	C17—C18—H18A	111.6
C12—C11—H11B	111.3	N1—C18—H18B	111.6
H11A—C11—H11B	109.2	C17—C18—H18B	111.6
C11—C12—C13	101.6 (5)	H18A—C18—H18B	109.4
C11—C12—H12A	111.4		

O1—S1—N1—C15	−13.5 (6)	O4—N2—C22—C21	65.74 (19)
O2—S1—N1—C15	−144.8 (6)	O3—N2—C22—C21	−117.70 (16)
C21—S1—N1—C15	102.3 (6)	C21—C22—C23—C24	−1.4 (2)
O1—S1—N1—C11	−21.2 (5)	N2—C22—C23—C24	179.13 (15)
O2—S1—N1—C11	−152.5 (5)	C22—C23—C24—C25	1.2 (3)
C21—S1—N1—C11	94.6 (5)	C23—C24—C25—C26	0.4 (3)
O1—S1—N1—C18	171.1 (7)	C24—C25—C26—C21	−1.8 (3)
O2—S1—N1—C18	39.8 (7)	C22—C21—C26—C25	1.5 (2)
C21—S1—N1—C18	−73.1 (7)	S1—C21—C26—C25	177.41 (13)
O1—S1—N1—C14	172.3 (5)	C14—N1—C11—C12	−27.8 (8)
O2—S1—N1—C14	41.0 (5)	S1—N1—C11—C12	163.9 (4)
C21—S1—N1—C14	−71.9 (5)	N1—C11—C12—C13	43.3 (8)
O1—S1—C21—C26	−152.34 (13)	C11—C12—C13—C14	−42.6 (7)
O2—S1—C21—C26	−23.76 (15)	C11—N1—C14—C13	1.2 (8)
N1—S1—C21—C26	91.26 (14)	S1—N1—C14—C13	169.7 (3)
O1—S1—C21—C22	23.24 (15)	C12—C13—C14—N1	25.6 (7)
O2—S1—C21—C22	151.82 (14)	C18—N1—C15—C16	6.6 (11)
N1—S1—C21—C22	−93.15 (15)	S1—N1—C15—C16	−168.8 (4)
C26—C21—C22—C23	0.1 (2)	N1—C15—C16—C17	−21.2 (10)
S1—C21—C22—C23	−175.54 (12)	C15—C16—C17—C18	29.7 (11)
C26—C21—C22—N2	179.49 (14)	C15—N1—C18—C17	11.2 (12)
S1—C21—C22—N2	3.9 (2)	S1—N1—C18—C17	−173.0 (5)
O4—N2—C22—C23	−114.81 (16)	C16—C17—C18—N1	−24.5 (11)
O3—N2—C22—C23	61.75 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*A*···O1^{i}	0.99	2.56	3.398 (7)	143
C11—H11*B*···O4	0.99	2.48	3.093 (8)	120
C25—H25···O4^{ii}	0.95	2.54	3.240 (2)	131
C25—H25···O1^{iii}	0.95	2.47	3.228 (2)	137

C26—H26···O2	0.95	2.49	2.860 (2)	103
C26—H26···O3^{iv}	0.95	2.56	3.467 (2)	160

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

1-[(2-Nitrophenyl)sulfonyl]piperidine (ja198)

Crystal data

$C_{11}H_{14}N_2O_4S$	$F(000) = 568$
$M_r = 270.30$	$D_x = 1.459$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 8.6881$ (9) Å	Cell parameters from 9983 reflections
$b = 15.0266$ (14) Å	$\theta = 2.6$–$28.3°$
$c = 9.8337$ (10) Å	$\mu = 0.27$ mm^{-1}
$\beta = 106.526$ (4)°	$T = 296$ K
$V = 1230.8$ (2) Å^3	Block, colourless
$Z = 4$	0.48 × 0.47 × 0.45 mm

Data collection

Bruker APEXII CCD diffractometer	25328 measured reflections
Radiation source: sealed tube	3059 independent reflections
Graphite monochromator	2662 reflections with $I > 2\sigma(I)$
Detector resolution: 8.3333 pixels mm^{-1}	$R_{\text{int}} = 0.017$
φ and ω scans	$\theta_{\max} = 28.3°$, $\theta_{\min} = 2.6°$
Absorption correction: numerical (SADABS; Bruker, 2008)	$h = -11 \rightarrow 11$
$T_{\min} = 0.941$, $T_{\max} = 1.000$	$k = -20 \rightarrow 19$
	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.036$	H-atom parameters constrained
$wR(F^2) = 0.106$	$w = 1/[\sigma^2(F_o^2) + (0.0546P)^2 + 0.3577P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{\max} = 0.001$
3059 reflections	$\Delta\rho_{\max} = 0.32$ e Å^{-3}
163 parameters	$\Delta\rho_{\min} = -0.31$ e Å^{-3}
0 restraints	
Primary atom site location: dual	

Special details

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

Refinement. Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2 U_{eq}(C).

Single-crystal X-ray diffraction (SC-XRD) data were collected at 200 or 296 K on a Bruker APEXII CCD diffractometer with graphite-monochromated Mo $K\alpha$ radiation using the *APEX2* data collection software and *SAINT* (Bruker 2012) for cell refinement and data reduction. The structures were solved by dual-space methods applying *SHELXT2018* (Sheldrick, 2015*a*) and refined by least-squares procedures using *SHELXL2018* (Sheldrick, 2015*b*). Data were corrected for absorption effects using the numerical method implemented in *SADABS* (Bruker 2012). All non-H atoms were refined anisotropically. The crystal structure diagrams were drawn with *ORTEP-3 for Windows* (Farrugia 2012).

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

	x	y	z	U_{iso}*/U_{eq}
S1	0.50241 (4)	0.66804 (2)	0.66863 (4)	0.04006 (12)
O1	0.57946 (15)	0.75271 (8)	0.69071 (16)	0.0609 (3)
O2	0.46987 (14)	0.62642 (8)	0.53305 (11)	0.0521 (3)
O3	0.23990 (17)	0.49620 (9)	0.58739 (17)	0.0705 (4)
O4	0.08960 (18)	0.57419 (11)	0.41946 (13)	0.0734 (4)
N1	0.61016 (14)	0.60117 (8)	0.78644 (13)	0.0416 (3)
N2	0.17049 (15)	0.56417 (9)	0.54124 (14)	0.0463 (3)
C11	0.5872 (2)	0.50422 (11)	0.76898 (19)	0.0517 (4)
H11A	0.564441	0.488569	0.669528	0.062*
H11B	0.496745	0.485803	0.801683	0.062*
C12	0.7374 (2)	0.45729 (13)	0.8536 (2)	0.0627 (5)
H12A	0.720629	0.393457	0.845762	0.075*
H12B	0.825213	0.471853	0.814835	0.075*
C13	0.7814 (2)	0.48376 (14)	1.0071 (2)	0.0606 (5)
H13A	0.881221	0.455002	1.057712	0.073*
H13B	0.698481	0.463866	1.048463	0.073*
C14	0.8004 (2)	0.58345 (15)	1.0233 (2)	0.0662 (5)
H14A	0.819586	0.599222	1.122396	0.079*
H14B	0.892831	0.602183	0.993857	0.079*
C15	0.6533 (2)	0.63160 (13)	0.93604 (18)	0.0582 (4)
H15A	0.564368	0.620532	0.975063	0.070*
H15B	0.673748	0.695143	0.939930	0.070*
C21	0.31422 (16)	0.68463 (9)	0.70518 (14)	0.0357 (3)
C22	0.17424 (16)	0.63776 (9)	0.64082 (14)	0.0372 (3)
C23	0.02972 (18)	0.65753 (11)	0.66598 (17)	0.0468 (3)
H23	−0.062624	0.626220	0.619609	0.056*
C24	0.0237 (2)	0.72436 (12)	0.76084 (19)	0.0530 (4)
H24	−0.072622	0.737406	0.780184	0.064*
C25	0.1604 (2)	0.77165 (12)	0.82675 (18)	0.0529 (4)
H25	0.156178	0.816624	0.890548	0.063*
C26	0.30433 (19)	0.75250 (10)	0.79841 (16)	0.0449 (3)
H26	0.395471	0.785498	0.842346	0.054*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.03940 (19)	0.0373 (2)	0.0487 (2)	−0.00205 (13)	0.02106 (15)	0.00020 (13)
O1	0.0540 (7)	0.0420 (6)	0.0951 (10)	−0.0107 (5)	0.0347 (6)	0.0018 (6)
O2	0.0559 (6)	0.0647 (7)	0.0425 (6)	0.0024 (5)	0.0248 (5)	−0.0001 (5)
O3	0.0696 (8)	0.0434 (7)	0.0929 (10)	0.0075 (6)	0.0143 (7)	−0.0118 (7)
O4	0.0767 (9)	0.0890 (10)	0.0474 (7)	0.0056 (8)	0.0066 (6)	−0.0153 (7)
N1	0.0405 (6)	0.0379 (6)	0.0457 (6)	0.0027 (5)	0.0110 (5)	−0.0090 (5)
N2	0.0405 (6)	0.0466 (7)	0.0525 (7)	−0.0037 (5)	0.0141 (5)	−0.0078 (6)
C11	0.0540 (9)	0.0402 (8)	0.0565 (9)	0.0011 (7)	0.0089 (7)	−0.0080 (7)
C12	0.0674 (11)	0.0466 (9)	0.0712 (11)	0.0155 (8)	0.0150 (9)	−0.0021 (8)

C13	0.0515 (9)	0.0706 (12)	0.0588 (10)	0.0106 (8)	0.0142 (8)	0.0126 (9)
C14	0.0570 (10)	0.0777 (13)	0.0555 (10)	0.0069 (9)	0.0024 (8)	−0.0120 (9)
C15	0.0583 (9)	0.0592 (10)	0.0521 (9)	0.0086 (8)	0.0074 (7)	−0.0185 (8)
C21	0.0383 (6)	0.0327 (6)	0.0390 (6)	0.0012 (5)	0.0157 (5)	0.0032 (5)
C22	0.0403 (7)	0.0344 (6)	0.0381 (6)	0.0015 (5)	0.0128 (5)	0.0032 (5)
C23	0.0386 (7)	0.0481 (8)	0.0551 (9)	0.0006 (6)	0.0155 (6)	0.0046 (7)
C24	0.0493 (8)	0.0539 (9)	0.0645 (10)	0.0104 (7)	0.0302 (7)	0.0049 (8)
C25	0.0634 (10)	0.0457 (8)	0.0580 (9)	0.0072 (7)	0.0307 (8)	−0.0055 (7)
C26	0.0491 (8)	0.0386 (7)	0.0496 (8)	−0.0008 (6)	0.0184 (6)	−0.0045 (6)

Geometric parameters (Å, º)

S1—O1	1.4252 (12)	C13—H13B	0.9700
S1—O2	1.4263 (12)	C14—C15	1.506 (3)
S1—N1	1.6160 (13)	C14—H14A	0.9700
S1—C21	1.7885 (14)	C14—H14B	0.9700
O3—N2	1.2070 (19)	C15—H15A	0.9700
O4—N2	1.2132 (18)	C15—H15B	0.9700
N1—C11	1.474 (2)	C21—C26	1.390 (2)
N1—C15	1.4833 (19)	C21—C22	1.3921 (19)
N2—C22	1.4713 (19)	C22—C23	1.379 (2)
C11—C12	1.508 (2)	C23—C24	1.382 (2)
C11—H11A	0.9700	C23—H23	0.9300
C11—H11B	0.9700	C24—C25	1.378 (3)
C12—C13	1.501 (3)	C24—H24	0.9300
C12—H12A	0.9700	C25—C26	1.386 (2)
C12—H12B	0.9700	C25—H25	0.9300
C13—C14	1.511 (3)	C26—H26	0.9300
C13—H13A	0.9700		

O1—S1—O2	119.57 (8)	C15—C14—C13	111.53 (16)
O1—S1—N1	107.14 (7)	C15—C14—H14A	109.3
O2—S1—N1	108.53 (7)	C13—C14—H14A	109.3
O1—S1—C21	105.16 (7)	C15—C14—H14B	109.3
O2—S1—C21	107.43 (7)	C13—C14—H14B	109.3
N1—S1—C21	108.59 (6)	H14A—C14—H14B	108.0
C11—N1—C15	114.07 (14)	N1—C15—C14	110.56 (14)
C11—N1—S1	119.97 (10)	N1—C15—H15A	109.5
C15—N1—S1	116.79 (10)	C14—C15—H15A	109.5
O3—N2—O4	124.29 (15)	N1—C15—H15B	109.5
O3—N2—C22	118.14 (13)	C14—C15—H15B	109.5
O4—N2—C22	117.42 (14)	H15A—C15—H15B	108.1
N1—C11—C12	109.30 (14)	C26—C21—C22	117.46 (13)
N1—C11—H11A	109.8	C26—C21—S1	117.79 (11)
C12—C11—H11A	109.8	C22—C21—S1	124.65 (11)
N1—C11—H11B	109.8	C23—C22—C21	122.11 (13)
C12—C11—H11B	109.8	C23—C22—N2	115.62 (13)
H11A—C11—H11B	108.3	C21—C22—N2	122.27 (12)

C13—C12—C11	111.34 (15)	C22—C23—C24	119.28 (15)
C13—C12—H12A	109.4	C22—C23—H23	120.4
C11—C12—H12A	109.4	C24—C23—H23	120.4
C13—C12—H12B	109.4	C25—C24—C23	119.93 (14)
C11—C12—H12B	109.4	C25—C24—H24	120.0
H12A—C12—H12B	108.0	C23—C24—H24	120.0
C12—C13—C14	110.97 (16)	C24—C25—C26	120.31 (15)
C12—C13—H13A	109.4	C24—C25—H25	119.8
C14—C13—H13A	109.4	C26—C25—H25	119.8
C12—C13—H13B	109.4	C25—C26—C21	120.88 (15)
C14—C13—H13B	109.4	C25—C26—H26	119.6
H13A—C13—H13B	108.0	C21—C26—H26	119.6

O1—S1—N1—C11	−163.84 (12)	O2—S1—C21—C22	21.08 (14)
O2—S1—N1—C11	−33.45 (14)	N1—S1—C21—C22	−96.11 (13)
C21—S1—N1—C11	83.04 (13)	C26—C21—C22—C23	0.7 (2)
O1—S1—N1—C15	51.20 (14)	S1—C21—C22—C23	−175.47 (11)
O2—S1—N1—C15	−178.40 (12)	C26—C21—C22—N2	−179.50 (13)
C21—S1—N1—C15	−61.91 (13)	S1—C21—C22—N2	4.32 (19)
C15—N1—C11—C12	−56.72 (19)	O3—N2—C22—C23	−111.68 (16)
S1—N1—C11—C12	157.43 (13)	O4—N2—C22—C23	64.06 (19)
N1—C11—C12—C13	56.4 (2)	O3—N2—C22—C21	68.51 (19)
C11—C12—C13—C14	−56.2 (2)	O4—N2—C22—C21	−115.74 (16)
C12—C13—C14—C15	54.1 (2)	C21—C22—C23—C24	−1.7 (2)
C11—N1—C15—C14	55.3 (2)	N2—C22—C23—C24	178.45 (14)
S1—N1—C15—C14	−157.73 (14)	C22—C23—C24—C25	1.4 (2)
C13—C14—C15—N1	−52.6 (2)	C23—C24—C25—C26	0.0 (3)
O1—S1—C21—C26	−26.70 (14)	C24—C25—C26—C21	−1.0 (3)
O2—S1—C21—C26	−155.08 (12)	C22—C21—C26—C25	0.7 (2)
N1—S1—C21—C26	87.72 (12)	S1—C21—C26—C25	177.14 (12)
O1—S1—C21—C22	149.47 (12)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*A*···O2	0.97	2.48	2.907 (2)	107
C11—H11*A*···O2^{i}	0.97	2.59	3.476 (2)	152
C15—H15*B*···O1	0.97	2.51	2.943 (2)	107
C25—H25···O2^{ii}	0.93	2.57	3.3337 (19)	140
C26—H26···O1	0.93	2.53	2.877 (2)	103

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

1-[(2-Nitrophenyl)sulfonyl]-2,3-dihydro-1*H*-indole (ja250)

Crystal data

$C_{14}H_{12}N_2O_4S$
M_r = 304.32
Monoclinic, $P2_1/n$

a = 7.4701 (5) Å
b = 23.6743 (12) Å
c = 7.8614 (5) Å

β = 94.989 (3)°
V = 1385.02 (15) Å^3
Z = 4
$F(000)$ = 632
D_x = 1.459 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 9952 reflections
θ = 2.7–27.1°
μ = 0.25 mm^{-1}
T = 296 K
Block, yellow
0.62 × 0.51 × 0.43 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: numerical (SADABS; Bruker, 2008)
$T_{\min}$ = 0.913, $T_{\max}$ = 1.000

28400 measured reflections
3434 independent reflections
2864 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.024
$\theta_{\max}$ = 28.4°, $\theta_{\min}$ = 2.7°
$h = -9\rightarrow9$
$k = -31\rightarrow28$
$l = -10\rightarrow10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.105
S = 1.05
3434 reflections
190 parameters
0 restraints
Primary atom site location: dual

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 0.5135P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.002
$\Delta\rho_{\max}$ = 0.26 e Å^{-3}
$\Delta\rho_{\min}$ = −0.29 e Å^{-3}

Special details

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

Refinement. Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with $U_{\rm iso}$(H) set to 1.2 $U_{\rm eq}$(C).
Single-crystal X-ray diffraction (SC-XRD) data were collected at 200 or 296 K on a Bruker APEXII CCD diffractometer with graphite-monochromated Mo $K\alpha$ radiation using the *APEX2* data collection software and *SAINT* (Bruker 2012) for cell refinement and data reduction. The structures were solved by dual-space methods applying *SHELXT2018* (Sheldrick, 2015*a*) and refined by least-squares procedures using *SHELXL2018* (Sheldrick, 2015*b*). Data were corrected for absorption effects using the numerical method implemented in *SADABS* (Bruker 2012). All non-H atoms were refined anisotropically. The crystal structure diagrams were drawn with *ORTEP-3 for Windows* (Farrugia 2012).

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

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
S1	0.79243 (5)	0.61702 (2)	0.46891 (5)	0.03709 (12)
O1	0.73327 (18)	0.65999 (5)	0.57767 (16)	0.0546 (3)
O2	0.91672 (16)	0.57553 (5)	0.53509 (15)	0.0487 (3)
O3	0.9987 (3)	0.77311 (7)	0.5254 (3)	0.1022 (6)
O4	1.1144 (3)	0.69290 (8)	0.6043 (2)	0.0839 (5)
N1	0.61681 (17)	0.58580 (6)	0.38325 (18)	0.0418 (3)

N2	1.0368 (2)	0.72407 (8)	0.5003 (2)	0.0611 (5)
C1	0.4442 (2)	0.61631 (8)	0.3457 (3)	0.0549 (5)
H1A	0.456425	0.646065	0.262694	0.066*
H1B	0.404075	0.632810	0.448776	0.066*
C2	0.3136 (2)	0.57142 (9)	0.2744 (3)	0.0576 (5)
H2A	0.224774	0.563294	0.354023	0.069*
H2B	0.252109	0.583591	0.166870	0.069*
C11	0.6039 (2)	0.53032 (6)	0.31204 (19)	0.0368 (3)
C12	0.4274 (2)	0.52060 (8)	0.2492 (2)	0.0436 (4)
C13	0.3805 (3)	0.46882 (9)	0.1789 (2)	0.0572 (5)
H13	0.262278	0.461664	0.137548	0.069*
C14	0.5100 (3)	0.42764 (9)	0.1703 (2)	0.0611 (5)
H14	0.478867	0.392525	0.123633	0.073*
C15	0.6852 (3)	0.43842 (8)	0.2306 (2)	0.0567 (5)
H15	0.771627	0.410511	0.222516	0.068*
C16	0.7352 (2)	0.48989 (7)	0.3029 (2)	0.0478 (4)
H16	0.853542	0.496970	0.344103	0.057*
C21	0.8884 (2)	0.65319 (6)	0.30000 (19)	0.0365 (3)
C22	0.9890 (2)	0.70184 (7)	0.3266 (2)	0.0466 (4)
C23	1.0551 (3)	0.73080 (9)	0.1950 (3)	0.0681 (6)
H23	1.120293	0.763931	0.215631	0.082*
C24	1.0234 (3)	0.70999 (11)	0.0308 (3)	0.0726 (6)
H24	1.068889	0.728941	−0.059696	0.087*
C25	0.9256 (3)	0.66178 (10)	0.0010 (2)	0.0609 (5)
H25	0.905119	0.647920	−0.109653	0.073*
C26	0.8568 (2)	0.63337 (8)	0.1351 (2)	0.0460 (4)
H26	0.788883	0.600801	0.113758	0.055*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0407 (2)	0.0361 (2)	0.0349 (2)	0.00043 (15)	0.00584 (15)	−0.00023 (14)
O1	0.0670 (8)	0.0504 (7)	0.0492 (7)	−0.0017 (6)	0.0214 (6)	−0.0118 (5)
O2	0.0468 (7)	0.0481 (7)	0.0493 (7)	0.0008 (5)	−0.0064 (5)	0.0089 (5)
O3	0.1429 (18)	0.0630 (10)	0.1030 (14)	−0.0190 (11)	0.0239 (12)	−0.0363 (10)
O4	0.0995 (13)	0.0977 (13)	0.0515 (9)	−0.0136 (10)	−0.0109 (8)	−0.0084 (9)
N1	0.0335 (7)	0.0364 (7)	0.0555 (8)	0.0049 (5)	0.0039 (6)	0.0012 (6)
N2	0.0699 (11)	0.0578 (10)	0.0574 (10)	−0.0260 (9)	0.0151 (8)	−0.0171 (8)
C1	0.0417 (9)	0.0549 (11)	0.0684 (12)	0.0155 (8)	0.0075 (8)	0.0090 (9)
C2	0.0349 (9)	0.0831 (14)	0.0548 (11)	0.0060 (9)	0.0043 (8)	0.0050 (10)
C11	0.0387 (8)	0.0370 (8)	0.0348 (8)	−0.0030 (6)	0.0032 (6)	0.0070 (6)
C12	0.0381 (8)	0.0584 (10)	0.0347 (8)	−0.0066 (7)	0.0054 (6)	0.0078 (7)
C13	0.0530 (11)	0.0703 (13)	0.0478 (10)	−0.0229 (10)	0.0017 (8)	0.0001 (9)
C14	0.0835 (15)	0.0499 (11)	0.0495 (11)	−0.0217 (10)	0.0023 (10)	−0.0015 (8)
C15	0.0749 (13)	0.0394 (9)	0.0548 (11)	0.0063 (9)	−0.0001 (9)	0.0024 (8)
C16	0.0457 (9)	0.0415 (9)	0.0547 (10)	0.0042 (7)	−0.0045 (8)	0.0018 (7)
C21	0.0382 (8)	0.0369 (7)	0.0349 (7)	0.0017 (6)	0.0052 (6)	0.0008 (6)
C22	0.0528 (10)	0.0438 (9)	0.0437 (9)	−0.0075 (7)	0.0071 (7)	−0.0018 (7)

C23	0.0842 (15)	0.0601 (12)	0.0616 (13)	−0.0261 (11)	0.0150 (11)	0.0068 (10)
C24	0.0845 (16)	0.0843 (15)	0.0509 (12)	−0.0173 (13)	0.0167 (11)	0.0202 (11)
C25	0.0674 (13)	0.0810 (14)	0.0344 (9)	−0.0051 (11)	0.0056 (8)	0.0028 (9)
C26	0.0474 (9)	0.0527 (10)	0.0376 (8)	−0.0043 (7)	0.0022 (7)	−0.0025 (7)

Geometric parameters (Å, º)

S1—O2	1.4190 (12)	C13—C14	1.379 (3)
S1—O1	1.4236 (12)	C13—H13	0.9300
S1—N1	1.6022 (14)	C14—C15	1.376 (3)
S1—C21	1.7822 (15)	C14—H14	0.9300
O3—N2	1.216 (2)	C15—C16	1.382 (3)
O4—N2	1.211 (2)	C15—H15	0.9300
N1—C11	1.428 (2)	C16—H16	0.9300
N1—C1	1.485 (2)	C21—C26	1.380 (2)
N2—C22	1.478 (2)	C21—C22	1.381 (2)
C1—C2	1.517 (3)	C22—C23	1.369 (3)
C1—H1A	0.9700	C23—C24	1.382 (3)
C1—H1B	0.9700	C23—H23	0.9300
C2—C12	1.497 (3)	C24—C25	1.364 (3)
C2—H2A	0.9700	C24—H24	0.9300
C2—H2B	0.9700	C25—C26	1.387 (2)
C11—C16	1.377 (2)	C25—H25	0.9300
C11—C12	1.386 (2)	C26—H26	0.9300
C12—C13	1.377 (3)		
O2—S1—O1	120.12 (8)	C12—C13—C14	119.61 (18)
O2—S1—N1	108.69 (7)	C12—C13—H13	120.2
O1—S1—N1	107.25 (8)	C14—C13—H13	120.2
O2—S1—C21	108.23 (7)	C15—C14—C13	120.12 (18)
O1—S1—C21	105.68 (7)	C15—C14—H14	119.9
N1—S1—C21	106.04 (7)	C13—C14—H14	119.9
C11—N1—C1	109.96 (13)	C14—C15—C16	121.33 (19)
C11—N1—S1	127.90 (11)	C14—C15—H15	119.3
C1—N1—S1	121.62 (12)	C16—C15—H15	119.3
O4—N2—O3	125.30 (19)	C11—C16—C15	117.77 (17)
O4—N2—C22	118.03 (17)	C11—C16—H16	121.1
O3—N2—C22	116.6 (2)	C15—C16—H16	121.1
N1—C1—C2	104.61 (14)	C26—C21—C22	118.20 (15)
N1—C1—H1A	110.8	C26—C21—S1	119.31 (12)
C2—C1—H1A	110.8	C22—C21—S1	122.45 (12)
N1—C1—H1B	110.8	C23—C22—C21	122.03 (17)
C2—C1—H1B	110.8	C23—C22—N2	116.49 (16)
H1A—C1—H1B	108.9	C21—C22—N2	121.44 (15)
C12—C2—C1	104.93 (14)	C22—C23—C24	118.92 (19)
C12—C2—H2A	110.8	C22—C23—H23	120.5
C1—C2—H2A	110.8	C24—C23—H23	120.5
C12—C2—H2B	110.8	C25—C24—C23	120.27 (19)

C1—C2—H2B	110.8	C25—C24—H24	119.9
H2A—C2—H2B	108.8	C23—C24—H24	119.9
C16—C11—C12	121.72 (16)	C24—C25—C26	120.26 (18)
C16—C11—N1	129.46 (15)	C24—C25—H25	119.9
C12—C11—N1	108.81 (14)	C26—C25—H25	119.9
C13—C12—C11	119.44 (17)	C21—C26—C25	120.31 (17)
C13—C12—C2	129.88 (17)	C21—C26—H26	119.8
C11—C12—C2	110.67 (15)	C25—C26—H26	119.8

O2—S1—N1—C11	−26.31 (16)	N1—C11—C16—C15	178.24 (16)
O1—S1—N1—C11	−157.59 (13)	C14—C15—C16—C11	−0.3 (3)
C21—S1—N1—C11	89.85 (14)	O2—S1—C21—C26	90.65 (14)
O2—S1—N1—C1	162.82 (14)	O1—S1—C21—C26	−139.48 (14)
O1—S1—N1—C1	31.54 (16)	N1—S1—C21—C26	−25.83 (15)
C21—S1—N1—C1	−81.01 (15)	O2—S1—C21—C22	−91.72 (15)
C11—N1—C1—C2	10.22 (19)	O1—S1—C21—C22	38.16 (16)
S1—N1—C1—C2	−177.44 (12)	N1—S1—C21—C22	151.80 (14)
N1—C1—C2—C12	−9.09 (19)	C26—C21—C22—C23	0.8 (3)
C1—N1—C11—C16	173.54 (17)	S1—C21—C22—C23	−176.87 (17)
S1—N1—C11—C16	1.8 (3)	C26—C21—C22—N2	−176.72 (17)
C1—N1—C11—C12	−7.25 (18)	S1—C21—C22—N2	5.6 (2)
S1—N1—C11—C12	−178.98 (12)	O4—N2—C22—C23	−122.4 (2)
C16—C11—C12—C13	1.4 (2)	O3—N2—C22—C23	55.7 (3)
N1—C11—C12—C13	−177.85 (15)	O4—N2—C22—C21	55.3 (3)
C16—C11—C12—C2	−179.67 (16)	O3—N2—C22—C21	−126.6 (2)
N1—C11—C12—C2	1.05 (18)	C21—C22—C23—C24	−1.4 (3)
C1—C2—C12—C13	−175.99 (18)	N2—C22—C23—C24	176.2 (2)
C1—C2—C12—C11	5.3 (2)	C22—C23—C24—C25	0.9 (4)
C11—C12—C13—C14	−0.8 (3)	C23—C24—C25—C26	0.2 (4)
C2—C12—C13—C14	−179.42 (18)	C22—C21—C26—C25	0.3 (3)
C12—C13—C14—C15	−0.4 (3)	S1—C21—C26—C25	178.08 (14)
C13—C14—C15—C16	1.0 (3)	C24—C25—C26—C21	−0.8 (3)
C12—C11—C16—C15	−0.9 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16···O2	0.93	2.41	2.975 (2)	119
C16—H16···O2^{i}	0.93	2.55	3.195 (2)	127
C23—H23···O1^{ii}	0.93	2.30	3.085 (2)	142
C26—H26···N1	0.93	2.60	2.982 (2)	106

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

2-(Pyrrolidine-1-sulfonyl)aniline (ja192)

Crystal data

$C_{10}H_{14}N_2O_2S$
$M_r = 226.29$
Monoclinic, $P2_1/c$
$a = 16.399\ (3)$ Å
$b = 7.9485\ (12)$ Å
$c = 18.376\ (3)$ Å
$\beta = 113.907\ (6)°$
$V = 2189.7\ (6)$ Å^3
$Z = 8$

$F(000) = 960$
$D_x = 1.373$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9858 reflections
$\theta = 2.3–28.3°$
$\mu = 0.28$ mm^{-1}
$T = 296$ K
Block, colourless
0.47 × 0.32 × 0.20 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: numerical (SADABS; Bruker, 2008)
$T_{\min} = 0.927$, $T_{\max} = 1.000$

55955 measured reflections
5477 independent reflections
4489 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.032$
$\theta_{\max} = 28.4°$, $\theta_{\min} = 2.3°$
$h = -21 \rightarrow 21$
$k = -10 \rightarrow 10$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.107$
$S = 1.04$
5477 reflections
287 parameters
0 restraints
Primary atom site location: dual

Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 0.5724P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.33$ e Å^{-3}
$\Delta\rho_{\min} = -0.36$ e Å^{-3}

Special details

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

Refinement. Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with $U_{\text{iso}}(\text{H})$ set to 1.2 $U_{\text{eq}}(\text{C})$.
The nitrogen-bound H atoms were located on a difference map and refined freely.
A reflection with large difference between its observed and calculated intensities was omitted. This is due to obstruction by the beam stop.
Single-crystal X-ray diffraction (SC-XRD) data were collected at 200 or 296 K on a Bruker APEXII CCD diffractometer with graphite-monochromated Mo $K\alpha$ radiation using the *APEX2* data collection software and *SAINT* (Bruker 2012) for cell refinement and data reduction. The structures were solved by dual-space methods applying *SHELXT2018* (Sheldrick, 2015*a*) and refined by least-squares procedures using *SHELXL2018* (Sheldrick, 2015*b*). Data were corrected for absorption effects using the numerical method implemented in *SADABS* (Bruker 2012). All non-H atoms were refined anisotropically. The crystal structure diagrams were drawn with *ORTEP-3 for Windows* (Farrugia 2012).

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

	x	y	z	U_{iso}*/U_{eq}
S1	0.60593 (2)	0.11220 (4)	0.38373 (2)	0.03995 (10)
S2	0.83675 (3)	0.54094 (5)	0.72672 (2)	0.04473 (11)
O11	0.55812 (7)	0.21253 (15)	0.41849 (9)	0.0639 (3)
O12	0.59914 (8)	0.15372 (16)	0.30611 (7)	0.0604 (3)
O21	0.76909 (9)	0.41413 (14)	0.70735 (7)	0.0567 (3)
O22	0.91886 (9)	0.51408 (18)	0.79373 (7)	0.0691 (4)
N11	0.56877 (8)	−0.07718 (15)	0.37882 (7)	0.0403 (3)
N12	0.69275 (18)	0.1559 (3)	0.56795 (13)	0.0824 (7)
N21	0.86112 (9)	0.55871 (17)	0.65022 (8)	0.0461 (3)
N22	0.63790 (10)	0.6609 (2)	0.65060 (10)	0.0564 (4)
C11	0.55387 (14)	−0.1535 (2)	0.44477 (11)	0.0597 (5)
H11A	0.508003	−0.094373	0.455235	0.072*
H11B	0.608273	−0.156605	0.492981	0.072*
C12	0.52380 (17)	−0.3286 (2)	0.41255 (14)	0.0740 (6)
H12A	0.533347	−0.407503	0.455504	0.089*
H12B	0.461089	−0.329150	0.376874	0.089*
C13	0.58061 (14)	−0.3722 (2)	0.36888 (11)	0.0592 (4)
H13A	0.549989	−0.450820	0.326115	0.071*
H13B	0.636476	−0.422062	0.404671	0.071*
C14	0.59696 (11)	−0.2074 (2)	0.33613 (9)	0.0490 (4)
H14A	0.659546	−0.194754	0.346526	0.059*
H14B	0.561903	−0.200442	0.279189	0.059*
C21	0.79052 (12)	0.5923 (3)	0.57112 (10)	0.0560 (4)
H21A	0.756395	0.691183	0.572202	0.067*
H21B	0.750349	0.497049	0.552570	0.067*
C22	0.83964 (15)	0.6204 (4)	0.51900 (13)	0.0842 (7)
H22A	0.830797	0.525926	0.483167	0.101*
H22B	0.818267	0.721612	0.487506	0.101*
C23	0.93579 (15)	0.6377 (4)	0.57256 (15)	0.0835 (7)
H23A	0.960942	0.736580	0.558485	0.100*
H23B	0.968833	0.539558	0.568498	0.100*
C24	0.94016 (12)	0.6543 (3)	0.65616 (13)	0.0707 (5)
H24A	0.994454	0.605166	0.695106	0.085*
H24B	0.936292	0.770978	0.669874	0.085*
C111	0.71909 (9)	0.11558 (16)	0.44789 (8)	0.0362 (3)
C112	0.74894 (11)	0.1352 (2)	0.53050 (9)	0.0492 (4)
C113	0.84137 (14)	0.1302 (3)	0.57508 (11)	0.0701 (6)
H113	0.863875	0.141455	0.630123	0.084*
C114	0.89883 (13)	0.1091 (3)	0.53914 (14)	0.0747 (6)
H114	0.959847	0.107237	0.570304	0.090*
C115	0.86913 (11)	0.0906 (3)	0.45820 (13)	0.0648 (5)
H115	0.909391	0.076405	0.434745	0.078*
C116	0.77956 (10)	0.0933 (2)	0.41274 (10)	0.0456 (3)
H116	0.758579	0.080195	0.357861	0.055*
C211	0.79105 (9)	0.73166 (17)	0.74078 (8)	0.0391 (3)

C212	0.69962 (9)	0.76850 (18)	0.70242 (8)	0.0395 (3)
C213	0.67185 (11)	0.9265 (2)	0.71751 (10)	0.0475 (3)
H213	0.611709	0.954422	0.692911	0.057*
C214	0.73005 (12)	1.0408 (2)	0.76707 (11)	0.0556 (4)
H214	0.709137	1.144299	0.775829	0.067*
C215	0.81985 (13)	1.0037 (2)	0.80430 (12)	0.0638 (5)
H215	0.859590	1.081726	0.837922	0.077*
C216	0.84970 (11)	0.8504 (2)	0.79108 (10)	0.0549 (4)
H216	0.910132	0.825037	0.816091	0.066*
H12C	0.7160 (18)	0.193 (4)	0.6109 (17)	0.095 (9)*
H12D	0.641 (2)	0.190 (4)	0.5399 (19)	0.113 (12)*
H21C	0.5862 (16)	0.690 (3)	0.6389 (13)	0.073 (7)*
H21D	0.6538 (13)	0.556 (3)	0.6567 (12)	0.060 (6)*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.02972 (16)	0.03424 (17)	0.0509 (2)	−0.00093 (12)	0.01122 (14)	0.00394 (13)
S2	0.0512 (2)	0.03890 (19)	0.03977 (19)	0.00835 (15)	0.01394 (16)	0.00206 (14)
O11	0.0418 (6)	0.0440 (6)	0.1067 (10)	0.0032 (5)	0.0311 (6)	−0.0126 (6)
O12	0.0500 (6)	0.0649 (7)	0.0500 (6)	−0.0067 (5)	0.0034 (5)	0.0230 (5)
O21	0.0805 (8)	0.0364 (5)	0.0607 (7)	−0.0048 (5)	0.0364 (6)	0.0000 (5)
O22	0.0678 (8)	0.0739 (8)	0.0485 (7)	0.0292 (7)	0.0060 (6)	0.0050 (6)
N11	0.0384 (6)	0.0377 (6)	0.0470 (6)	−0.0090 (5)	0.0196 (5)	−0.0053 (5)
N12	0.0955 (16)	0.1087 (16)	0.0550 (10)	−0.0348 (14)	0.0430 (11)	−0.0311 (11)
N21	0.0429 (6)	0.0480 (7)	0.0467 (7)	0.0047 (5)	0.0174 (5)	−0.0002 (5)
N22	0.0381 (7)	0.0600 (9)	0.0651 (9)	−0.0058 (7)	0.0148 (7)	−0.0136 (7)
C11	0.0825 (12)	0.0509 (9)	0.0633 (10)	−0.0215 (9)	0.0475 (10)	−0.0106 (8)
C12	0.1049 (16)	0.0496 (10)	0.0838 (14)	−0.0309 (10)	0.0550 (13)	−0.0085 (9)
C13	0.0738 (11)	0.0421 (8)	0.0551 (10)	−0.0033 (8)	0.0192 (9)	−0.0041 (7)
C14	0.0550 (9)	0.0456 (8)	0.0495 (8)	−0.0087 (7)	0.0242 (7)	−0.0122 (6)
C21	0.0502 (9)	0.0693 (11)	0.0465 (9)	0.0003 (8)	0.0175 (7)	0.0075 (8)
C22	0.0775 (14)	0.121 (2)	0.0628 (12)	0.0000 (13)	0.0370 (11)	0.0190 (12)
C23	0.0653 (12)	0.1083 (19)	0.0937 (16)	0.0056 (12)	0.0495 (12)	0.0194 (14)
C24	0.0447 (9)	0.0847 (14)	0.0824 (13)	−0.0046 (9)	0.0255 (9)	−0.0003 (11)
C111	0.0322 (6)	0.0331 (6)	0.0394 (7)	−0.0036 (5)	0.0105 (5)	0.0005 (5)
C112	0.0577 (9)	0.0442 (8)	0.0423 (8)	−0.0116 (7)	0.0168 (7)	−0.0070 (6)
C113	0.0710 (12)	0.0640 (11)	0.0459 (9)	−0.0063 (9)	−0.0066 (9)	−0.0071 (8)
C114	0.0401 (9)	0.0711 (13)	0.0851 (15)	0.0055 (8)	−0.0035 (9)	−0.0100 (11)
C115	0.0383 (8)	0.0682 (11)	0.0867 (14)	0.0021 (8)	0.0240 (9)	−0.0051 (10)
C116	0.0390 (7)	0.0473 (8)	0.0508 (8)	−0.0013 (6)	0.0185 (6)	0.0010 (6)
C211	0.0400 (7)	0.0362 (7)	0.0388 (7)	0.0002 (5)	0.0138 (6)	−0.0023 (5)
C212	0.0389 (7)	0.0420 (7)	0.0405 (7)	−0.0038 (6)	0.0190 (6)	0.0014 (6)
C213	0.0458 (8)	0.0462 (8)	0.0578 (9)	0.0050 (6)	0.0286 (7)	0.0064 (7)
C214	0.0698 (11)	0.0390 (8)	0.0714 (11)	−0.0006 (7)	0.0425 (9)	−0.0054 (7)
C215	0.0622 (11)	0.0522 (9)	0.0772 (12)	−0.0157 (8)	0.0285 (10)	−0.0267 (9)
C216	0.0437 (8)	0.0553 (9)	0.0582 (10)	−0.0054 (7)	0.0129 (7)	−0.0162 (8)

Geometric parameters (Å, º)

S1—O12	1.4236 (13)	C21—H21A	0.9700
S1—O11	1.4369 (12)	C21—H21B	0.9700
S1—N11	1.6127 (12)	C22—C23	1.489 (3)
S1—C111	1.7496 (14)	C22—H22A	0.9700
S2—O22	1.4252 (12)	C22—H22B	0.9700
S2—O21	1.4330 (13)	C23—C24	1.514 (3)
S2—N21	1.6161 (14)	C23—H23A	0.9700
S2—C211	1.7558 (14)	C23—H23B	0.9700
N11—C11	1.462 (2)	C24—H24A	0.9700
N11—C14	1.4813 (19)	C24—H24B	0.9700
N12—C112	1.365 (3)	C111—C116	1.397 (2)
N12—H12C	0.78 (3)	C111—C112	1.402 (2)
N12—H12D	0.84 (3)	C112—C113	1.401 (3)
N21—C24	1.468 (2)	C113—C114	1.363 (3)
N21—C21	1.470 (2)	C113—H113	0.9300
N22—C212	1.372 (2)	C114—C115	1.373 (3)
N22—H21C	0.82 (2)	C114—H114	0.9300
N22—H21D	0.87 (2)	C115—C116	1.364 (2)
C11—C12	1.514 (2)	C115—H115	0.9300
C11—H11A	0.9700	C116—H116	0.9300
C11—H11B	0.9700	C211—C216	1.396 (2)
C12—C13	1.496 (3)	C211—C212	1.405 (2)
C12—H12A	0.9700	C212—C213	1.402 (2)
C12—H12B	0.9700	C213—C214	1.364 (2)
C13—C14	1.510 (2)	C213—H213	0.9300
C13—H13A	0.9700	C214—C215	1.381 (3)
C13—H13B	0.9700	C214—H214	0.9300
C14—H14A	0.9700	C215—C216	1.371 (2)
C14—H14B	0.9700	C215—H215	0.9300
C21—C22	1.497 (3)	C216—H216	0.9300

O12—S1—O11	118.78 (8)	C23—C22—C21	106.93 (17)
O12—S1—N11	106.62 (7)	C23—C22—H22A	110.3
O11—S1—N11	106.32 (7)	C21—C22—H22A	110.3
O12—S1—C111	107.62 (7)	C23—C22—H22B	110.3
O11—S1—C111	108.23 (7)	C21—C22—H22B	110.3
N11—S1—C111	109.00 (6)	H22A—C22—H22B	108.6
O22—S2—O21	118.33 (8)	C22—C23—C24	106.31 (16)
O22—S2—N21	106.53 (8)	C22—C23—H23A	110.5
O21—S2—N21	106.55 (7)	C24—C23—H23A	110.5
O22—S2—C211	108.20 (7)	C22—C23—H23B	110.5
O21—S2—C211	108.07 (7)	C24—C23—H23B	110.5
N21—S2—C211	108.86 (7)	H23A—C23—H23B	108.7
C11—N11—C14	110.00 (13)	N21—C24—C23	101.67 (16)
C11—N11—S1	121.83 (10)	N21—C24—H24A	111.4
C14—N11—S1	119.32 (10)	C23—C24—H24A	111.4

C112—N12—H12C	114 (2)	N21—C24—H24B	111.4
C112—N12—H12D	117 (2)	C23—C24—H24B	111.4
H12C—N12—H12D	117 (3)	H24A—C24—H24B	109.3
C24—N21—C21	107.31 (14)	C116—C111—C112	120.86 (14)
C24—N21—S2	120.18 (12)	C116—C111—S1	116.38 (11)
C21—N21—S2	120.19 (11)	C112—C111—S1	122.75 (11)
C212—N22—H21C	113.7 (16)	N12—C112—C113	119.99 (18)
C212—N22—H21D	114.3 (13)	N12—C112—C111	123.25 (17)
H21C—N22—H21D	122 (2)	C113—C112—C111	116.75 (16)
N11—C11—C12	101.23 (14)	C114—C113—C112	121.12 (17)
N11—C11—H11A	111.5	C114—C113—H113	119.4
C12—C11—H11A	111.5	C112—C113—H113	119.4
N11—C11—H11B	111.5	C113—C114—C115	121.80 (17)
C12—C11—H11B	111.5	C113—C114—H114	119.1
H11A—C11—H11B	109.3	C115—C114—H114	119.1
C13—C12—C11	104.33 (14)	C116—C115—C114	118.88 (18)
C13—C12—H12A	110.9	C116—C115—H115	120.6
C11—C12—H12A	110.9	C114—C115—H115	120.6
C13—C12—H12B	110.9	C115—C116—C111	120.58 (16)
C11—C12—H12B	110.9	C115—C116—H116	119.7
H12A—C12—H12B	108.9	C111—C116—H116	119.7
C12—C13—C14	105.24 (14)	C216—C211—C212	119.99 (14)
C12—C13—H13A	110.7	C216—C211—S2	117.42 (11)
C14—C13—H13A	110.7	C212—C211—S2	122.57 (11)
C12—C13—H13B	110.7	N22—C212—C213	119.25 (14)
C14—C13—H13B	110.7	N22—C212—C211	123.75 (14)
H13A—C13—H13B	108.8	C213—C212—C211	116.98 (13)
N11—C14—C13	104.53 (13)	C214—C213—C212	122.24 (15)
N11—C14—H14A	110.8	C214—C213—H213	118.9
C13—C14—H14A	110.8	C212—C213—H213	118.9
N11—C14—H14B	110.8	C213—C214—C215	120.37 (15)
C13—C14—H14B	110.8	C213—C214—H214	119.8
H14A—C14—H14B	108.9	C215—C214—H214	119.8
N21—C21—C22	104.32 (15)	C216—C215—C214	119.19 (16)
N21—C21—H21A	110.9	C216—C215—H215	120.4
C22—C21—H21A	110.9	C214—C215—H215	120.4
N21—C21—H21B	110.9	C215—C216—C211	121.23 (16)
C22—C21—H21B	110.9	C215—C216—H216	119.4
H21A—C21—H21B	108.9	C211—C216—H216	119.4

O12—S1—N11—C11	−171.19 (13)	O11—S1—C111—C112	29.11 (14)
O11—S1—N11—C11	−43.54 (15)	N11—S1—C111—C112	−86.12 (13)
C111—S1—N11—C11	72.91 (14)	C116—C111—C112—N12	−179.45 (18)
O12—S1—N11—C14	44.67 (13)	S1—C111—C112—N12	−1.1 (2)
O11—S1—N11—C14	172.32 (12)	C116—C111—C112—C113	−0.4 (2)
C111—S1—N11—C14	−71.23 (13)	S1—C111—C112—C113	178.01 (13)
O22—S2—N21—C24	−40.89 (16)	N12—C112—C113—C114	179.8 (2)
O21—S2—N21—C24	−168.09 (14)	C111—C112—C113—C114	0.7 (3)

C211—S2—N21—C24	75.59 (15)	C112—C113—C114—C115	−0.5 (3)
O22—S2—N21—C21	−178.33 (13)	C113—C114—C115—C116	0.0 (3)
O21—S2—N21—C21	54.47 (14)	C114—C115—C116—C111	0.4 (3)
C211—S2—N21—C21	−61.86 (14)	C112—C111—C116—C115	−0.2 (2)
C14—N11—C11—C12	−30.58 (19)	S1—C111—C116—C115	−178.64 (14)
S1—N11—C11—C12	−177.66 (14)	O22—S2—C211—C216	23.95 (15)
N11—C11—C12—C13	38.2 (2)	O21—S2—C211—C216	153.20 (13)
C11—C12—C13—C14	−32.5 (2)	N21—S2—C211—C216	−91.45 (14)
C11—N11—C14—C13	11.09 (18)	O22—S2—C211—C212	−157.40 (13)
S1—N11—C14—C13	159.11 (12)	O21—S2—C211—C212	−28.14 (14)
C12—C13—C14—N11	13.63 (19)	N21—S2—C211—C212	87.21 (13)
C24—N21—C21—C22	31.2 (2)	C216—C211—C212—N22	178.30 (16)
S2—N21—C21—C22	173.45 (15)	S2—C211—C212—N22	−0.3 (2)
N21—C21—C22—C23	−11.7 (3)	C216—C211—C212—C213	−0.2 (2)
C21—C22—C23—C24	−11.0 (3)	S2—C211—C212—C213	−178.81 (11)
C21—N21—C24—C23	−37.5 (2)	N22—C212—C213—C214	−178.60 (15)
S2—N21—C24—C23	−179.70 (14)	C211—C212—C213—C214	0.0 (2)
C22—C23—C24—N21	29.2 (2)	C212—C213—C214—C215	0.3 (3)
O12—S1—C111—C116	−22.94 (13)	C213—C214—C215—C216	−0.3 (3)
O11—S1—C111—C116	−152.46 (11)	C214—C215—C216—C211	0.0 (3)
N11—S1—C111—C116	92.31 (12)	C212—C211—C216—C215	0.2 (3)
O12—S1—C111—C112	158.62 (12)	S2—C211—C216—C215	178.89 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N12—H12*C*···O21	0.78 (3)	2.39 (3)	3.121 (2)	155 (3)
N12—H12*D*···O11	0.84 (3)	2.10 (3)	2.776 (3)	138 (3)
N22—H21*C*···O11^{i}	0.82 (2)	2.30 (2)	3.106 (2)	168 (2)
N22—H21*D*···O21	0.87 (2)	2.07 (2)	2.783 (2)	138.8 (17)
C114—H114···O22^{ii}	0.93	2.59	3.391 (2)	144
C116—H116···O12	0.93	2.46	2.8505 (19)	105
C216—H216···O22	0.93	2.52	2.897 (2)	105

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

2-(Piperidine-1-sulfonyl)aniline (ka115)

Crystal data

$C_{11}H_{16}N_2O_2S$
M_r = 240.32
Orthorhombic, *Pbca*
a = 11.1747 (4) Å
b = 10.4850 (4) Å
c = 20.1368 (8) Å
V = 2359.36 (15) Å^3
Z = 8
$F(000)$ = 1024

D_x = 1.353 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 9577 reflections
θ = 2.9–28.3°
μ = 0.26 mm^{-1}
T = 200 K
Block, colourless
0.49 × 0.26 × 0.25 mm

Data collection

Bruker APEXII CCD diffractometer	57075 measured reflections
Radiation source: sealed tube	2935 independent reflections
Graphite monochromator	2583 reflections with $I > 2\sigma(I)$
Detector resolution: 8.3333 pixels mm^{-1}	$R_{\rm int} = 0.020$
φ and ω scans	$\theta_{\rm max} = 28.3°$, $\theta_{\rm min} = 2.0°$
Absorption correction: numerical (SADABS; Bruker, 2008)	$h = -14\rightarrow14$
$T_{\rm min} = 0.944$, $T_{\rm max} = 1.000$	$k = -13\rightarrow13$
	$l = -26\rightarrow26$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: mixed
$R[F^2 > 2\sigma(F^2)] = 0.029$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.087$	$w = 1/[\sigma^2(F_o^2) + (0.0456P)^2 + 0.8126P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\rm max} = 0.001$
2935 reflections	$\Delta\rho_{\rm max} = 0.34$ e Å^{-3}
153 parameters	$\Delta\rho_{\rm min} = -0.36$ e Å^{-3}
0 restraints	
Primary atom site location: dual	

Special details

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

Refinement. Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with $U_{\rm iso}$(H) set to 1.2 $U_{\rm eq}$(C).

The nitrogen-bound H atoms were located on a difference map and refined freely.

Single-crystal X-ray diffraction (SC-XRD) data were collected at 200 or 296 K on a Bruker APEXII CCD diffractometer with graphite-monochromated Mo *Kα* radiation using the *APEX2* data collection software and *SAINT* (Bruker 2012) for cell refinement and data reduction. The structures were solved by dual-space methods applying *SHELXT2018* (Sheldrick, 2015*a*) and refined by least-squares procedures using *SHELXL2018* (Sheldrick, 2015*b*). Data were corrected for absorption effects using the numerical method implemented in *SADABS* (Bruker 2012). All non-H atoms were refined anisotropically. The crystal structure diagrams were drawn with *ORTEP-3 for Windows* (Farrugia 2012).

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

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$
S1	0.66247 (2)	0.46255 (3)	0.35628 (2)	0.02489 (10)
O1	0.78413 (7)	0.45299 (8)	0.37905 (5)	0.0331 (2)
O2	0.60807 (8)	0.58657 (8)	0.35302 (5)	0.0344 (2)
N1	0.48648 (11)	0.51544 (13)	0.23579 (7)	0.0417 (3)
N2	0.58073 (8)	0.37768 (9)	0.40733 (5)	0.0257 (2)
C11	0.65595 (9)	0.38973 (11)	0.27755 (6)	0.0245 (2)
C12	0.56770 (10)	0.41892 (11)	0.22957 (6)	0.0281 (2)
C13	0.56702 (11)	0.34467 (13)	0.17139 (6)	0.0340 (3)
H13	0.509126	0.361787	0.138016	0.041*
C14	0.64803 (12)	0.24808 (13)	0.16172 (6)	0.0357 (3)
H14	0.644395	0.199006	0.122112	0.043*

C15	0.73532 (11)	0.22069 (12)	0.20879 (6)	0.0336 (3)
H15	0.791553	0.154219	0.201449	0.040*
C16	0.73871 (10)	0.29170 (11)	0.26622 (6)	0.0280 (2)
H16	0.798089	0.273989	0.298703	0.034*
C21	0.61856 (11)	0.24349 (12)	0.41533 (6)	0.0320 (3)
H21A	0.706433	0.239499	0.421235	0.038*
H21B	0.597532	0.194456	0.374968	0.038*
C22	0.55700 (14)	0.18533 (15)	0.47531 (7)	0.0429 (3)
H22A	0.584669	0.229165	0.516052	0.051*
H22B	0.579347	0.094219	0.478891	0.051*
C23	0.42189 (13)	0.19665 (15)	0.47012 (8)	0.0460 (3)
H23A	0.392700	0.144432	0.432495	0.055*
H23B	0.384336	0.164250	0.511330	0.055*
C24	0.38708 (12)	0.33508 (15)	0.45955 (8)	0.0448 (3)
H24A	0.408917	0.385359	0.499357	0.054*
H24B	0.299337	0.341143	0.453508	0.054*
C25	0.44964 (11)	0.39042 (13)	0.39903 (7)	0.0358 (3)
H25A	0.423672	0.344456	0.358573	0.043*
H25B	0.428110	0.481486	0.393957	0.043*
H1A	0.4305 (18)	0.5177 (17)	0.2083 (9)	0.050 (5)*
H1B	0.4888 (14)	0.5639 (15)	0.2697 (8)	0.037 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.02117 (15)	0.02347 (15)	0.03003 (16)	−0.00296 (9)	−0.00057 (10)	−0.00017 (10)
O1	0.0226 (4)	0.0393 (5)	0.0373 (5)	−0.0060 (3)	−0.0045 (3)	−0.0021 (4)
O2	0.0350 (5)	0.0231 (4)	0.0451 (5)	−0.0009 (3)	0.0032 (4)	0.0006 (4)
N1	0.0315 (6)	0.0460 (7)	0.0476 (7)	0.0119 (5)	−0.0111 (5)	0.0009 (6)
N2	0.0240 (4)	0.0249 (5)	0.0280 (5)	−0.0019 (4)	0.0011 (4)	0.0007 (4)
C11	0.0214 (5)	0.0253 (5)	0.0267 (5)	−0.0027 (4)	0.0000 (4)	0.0037 (4)
C12	0.0223 (5)	0.0299 (6)	0.0319 (6)	−0.0030 (4)	−0.0010 (4)	0.0078 (5)
C13	0.0318 (6)	0.0419 (7)	0.0284 (6)	−0.0068 (5)	−0.0052 (5)	0.0056 (5)
C14	0.0425 (7)	0.0369 (7)	0.0277 (6)	−0.0089 (5)	0.0021 (5)	−0.0001 (5)
C15	0.0360 (6)	0.0300 (6)	0.0349 (6)	0.0010 (5)	0.0073 (5)	0.0021 (5)
C16	0.0251 (5)	0.0287 (6)	0.0303 (5)	0.0002 (4)	0.0008 (4)	0.0056 (4)
C21	0.0366 (6)	0.0278 (6)	0.0315 (6)	0.0029 (5)	0.0030 (5)	0.0057 (5)
C22	0.0485 (8)	0.0458 (8)	0.0344 (7)	0.0000 (6)	0.0024 (6)	0.0149 (6)
C23	0.0457 (8)	0.0512 (8)	0.0409 (7)	−0.0114 (7)	0.0090 (6)	0.0132 (6)
C24	0.0321 (6)	0.0538 (9)	0.0486 (8)	−0.0040 (6)	0.0118 (6)	0.0041 (7)
C25	0.0234 (5)	0.0384 (7)	0.0455 (7)	−0.0019 (5)	0.0014 (5)	0.0089 (6)

Geometric parameters (Å, º)

S1—O2	1.4369 (9)	C15—H15	0.9500
S1—O1	1.4382 (8)	C16—H16	0.9500
S1—N2	1.6381 (10)	C21—C22	1.5178 (17)
S1—C11	1.7611 (12)	C21—H21A	0.9900

N1—C12	1.3652 (17)	C21—H21B	0.9900
N1—H1A	0.836 (19)	C22—C23	1.518 (2)
N1—H1B	0.852 (17)	C22—H22A	0.9900
N2—C21	1.4780 (15)	C22—H22B	0.9900
N2—C25	1.4804 (15)	C23—C24	1.518 (2)
C11—C16	1.4014 (16)	C23—H23A	0.9900
C11—C12	1.4141 (15)	C23—H23B	0.9900
C12—C13	1.4066 (18)	C24—C25	1.5200 (18)
C13—C14	1.3723 (19)	C24—H24A	0.9900
C13—H13	0.9500	C24—H24B	0.9900
C14—C15	1.3901 (18)	C25—H25A	0.9900
C14—H14	0.9500	C25—H25B	0.9900
C15—C16	1.3759 (17)		

O2—S1—O1	118.52 (5)	N2—C21—C22	109.85 (11)
O2—S1—N2	106.51 (5)	N2—C21—H21A	109.7
O1—S1—N2	106.81 (5)	C22—C21—H21A	109.7
O2—S1—C11	109.50 (5)	N2—C21—H21B	109.7
O1—S1—C11	107.21 (5)	C22—C21—H21B	109.7
N2—S1—C11	107.84 (5)	H21A—C21—H21B	108.2
C12—N1—H1A	117.2 (12)	C21—C22—C23	111.39 (11)
C12—N1—H1B	119.7 (10)	C21—C22—H22A	109.4
H1A—N1—H1B	122.5 (16)	C23—C22—H22A	109.4
C21—N2—C25	112.41 (10)	C21—C22—H22B	109.4
C21—N2—S1	115.21 (8)	C23—C22—H22B	109.4
C25—N2—S1	115.59 (8)	H22A—C22—H22B	108.0
C16—C11—C12	120.52 (11)	C24—C23—C22	109.84 (12)
C16—C11—S1	115.92 (8)	C24—C23—H23A	109.7
C12—C11—S1	123.37 (9)	C22—C23—H23A	109.7
N1—C12—C13	118.89 (11)	C24—C23—H23B	109.7
N1—C12—C11	124.15 (12)	C22—C23—H23B	109.7
C13—C12—C11	116.94 (11)	H23A—C23—H23B	108.2
C14—C13—C12	121.54 (11)	C23—C24—C25	111.08 (12)
C14—C13—H13	119.2	C23—C24—H24A	109.4
C12—C13—H13	119.2	C25—C24—H24A	109.4
C13—C14—C15	121.24 (12)	C23—C24—H24B	109.4
C13—C14—H14	119.4	C25—C24—H24B	109.4
C15—C14—H14	119.4	H24A—C24—H24B	108.0
C16—C15—C14	118.73 (12)	N2—C25—C24	109.28 (11)
C16—C15—H15	120.6	N2—C25—H25A	109.8
C14—C15—H15	120.6	C24—C25—H25A	109.8
C15—C16—C11	121.03 (11)	N2—C25—H25B	109.8
C15—C16—H16	119.5	C24—C25—H25B	109.8
C11—C16—H16	119.5	H25A—C25—H25B	108.3

O2—S1—N2—C21	177.25 (8)	N1—C12—C13—C14	178.65 (12)
O1—S1—N2—C21	−55.17 (9)	C11—C12—C13—C14	0.20 (17)
C11—S1—N2—C21	59.79 (9)	C12—C13—C14—C15	−0.85 (19)

O2—S1—N2—C25	43.41 (10)	C13—C14—C15—C16	0.67 (19)
O1—S1—N2—C25	170.99 (9)	C14—C15—C16—C11	0.15 (18)
C11—S1—N2—C25	−74.05 (10)	C12—C11—C16—C15	−0.80 (17)
O2—S1—C11—C16	158.65 (8)	S1—C11—C16—C15	174.39 (9)
O1—S1—C11—C16	28.86 (10)	C25—N2—C21—C22	−58.70 (14)
N2—S1—C11—C16	−85.83 (9)	S1—N2—C21—C22	166.02 (9)
O2—S1—C11—C12	−26.31 (11)	N2—C21—C22—C23	55.96 (15)
O1—S1—C11—C12	−156.11 (9)	C21—C22—C23—C24	−54.92 (17)
N2—S1—C11—C12	89.20 (10)	C22—C23—C24—C25	55.71 (17)
C16—C11—C12—N1	−177.75 (12)	C21—N2—C25—C24	59.34 (14)
S1—C11—C12—N1	7.43 (17)	S1—N2—C25—C24	−165.56 (9)
C16—C11—C12—C13	0.61 (16)	C23—C24—C25—N2	−57.41 (16)
S1—C11—C12—C13	−174.21 (8)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.836 (19)	2.496 (19)	3.3000 (16)	161.8 (16)
N1—H1*B*···O2	0.852 (17)	2.156 (17)	2.8240 (17)	135.1 (14)
C16—H16···O1	0.95	2.48	2.8774 (15)	105
C16—H16···O2^{ii}	0.95	2.48	3.2576 (14)	139
C21—H21*A*···O1	0.99	2.55	2.9635 (15)	105
C25—H25*B*···O2	0.99	2.44	2.8675 (15)	106

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

2-(2,3-Dihydro-1*H*-indole-1-sulfonyl)aniline (ja189)

Crystal data

$C_{14}H_{14}N_2O_2S$
M_r = 274.33
Monoclinic, $P2_1/n$
a = 9.7990 (11) Å
b = 10.2612 (13) Å
c = 13.2010 (16) Å
β = 100.682 (5)°
V = 1304.4 (3) Å^3
Z = 4

$F(000)$ = 576
D_x = 1.397 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9932 reflections
θ = 2.4–28.4°
μ = 0.25 mm^{-1}
T = 296 K
Block, colourless
0.54 × 0.34 × 0.34 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: numerical (SADABS; Bruker, 2008)
T_{min} = 0.925, T_{max} = 1.000

28600 measured reflections
3275 independent reflections
2726 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 28.7°, θ_{min} = 2.4°
h = −13→13
k = −13→12
l = −16→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.113$
$S = 1.05$
3275 reflections
180 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.053P)^2 + 0.4178P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}

Special details

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

Refinement. Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2 U_{eq}(C).
The nitrogen-bound H atoms were located on a difference map and refined freely.
Single-crystal X-ray diffraction (SC-XRD) data were collected at 200 or 296 K on a Bruker APEXII CCD diffractometer with graphite-monochromated Mo *Kα* radiation using the *APEX2* data collection software and *SAINT* (Bruker 2012) for cell refinement and data reduction. The structures were solved by dual-space methods applying *SHELXT2018* (Sheldrick, 2015*a*) and refined by least-squares procedures using *SHELXL2018* (Sheldrick, 2015*b*). Data were corrected for absorption effects using the numerical method implemented in *SADABS* (Bruker 2012). All non-H atoms were refined anisotropically. The crystal structure diagrams were drawn with *ORTEP-3 for Windows* (Farrugia 2012).

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

	x	y	z	U_{iso}*/U_{eq}
S1	0.38977 (4)	0.26951 (4)	0.46693 (3)	0.04455 (13)
O1	0.46972 (13)	0.21097 (14)	0.55683 (9)	0.0646 (4)
O2	0.45941 (11)	0.31734 (14)	0.38820 (9)	0.0593 (3)
N1	0.31329 (14)	0.39561 (13)	0.50488 (10)	0.0476 (3)
N2	0.1992 (2)	0.27928 (18)	0.25271 (12)	0.0630 (4)
C1	0.20394 (18)	0.60239 (16)	0.50168 (14)	0.0536 (4)
H1A	0.262936	0.675102	0.528584	0.064*
H1B	0.119055	0.635928	0.460230	0.064*
C2	0.27807 (18)	0.51224 (17)	0.43895 (13)	0.0532 (4)
H2A	0.218044	0.489119	0.374487	0.064*
H2B	0.361389	0.553051	0.423985	0.064*
C11	0.23714 (14)	0.39828 (15)	0.58651 (11)	0.0418 (3)
C12	0.17276 (15)	0.51848 (15)	0.58744 (12)	0.0457 (3)
C13	0.09543 (18)	0.5451 (2)	0.66236 (15)	0.0621 (5)
H13	0.052108	0.625529	0.663818	0.074*
C14	0.08286 (19)	0.4512 (2)	0.73524 (14)	0.0681 (5)
H14	0.030634	0.468525	0.785769	0.082*
C15	0.1472 (2)	0.3326 (2)	0.73330 (13)	0.0626 (5)
H15	0.137895	0.270445	0.782820	0.075*
C16	0.22563 (19)	0.30382 (18)	0.65894 (13)	0.0542 (4)
H16	0.269140	0.223417	0.657880	0.065*

C21	0.26219 (15)	0.15726 (15)	0.41371 (11)	0.0437 (3)
C22	0.18015 (15)	0.17834 (16)	0.31512 (11)	0.0460 (3)
C23	0.07113 (18)	0.0912 (2)	0.28389 (15)	0.0631 (5)
H23	0.012937	0.103508	0.220484	0.076*
C24	0.0478 (3)	−0.0110 (2)	0.3436 (2)	0.0826 (7)
H24	−0.026770	−0.066288	0.320608	0.099*
C25	0.1320 (3)	−0.0348 (2)	0.4375 (2)	0.0866 (7)
H25	0.116577	−0.106941	0.476522	0.104*
C26	0.2397 (2)	0.04998 (18)	0.47264 (15)	0.0658 (5)
H26	0.297316	0.035268	0.535935	0.079*
H2C	0.283 (3)	0.308 (3)	0.259 (2)	0.093 (8)*
H2D	0.143 (2)	0.282 (2)	0.1916 (19)	0.079 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.03429 (19)	0.0609 (3)	0.0368 (2)	0.00384 (14)	0.00238 (14)	−0.00607 (15)
O1	0.0543 (7)	0.0854 (9)	0.0466 (6)	0.0190 (6)	−0.0106 (5)	−0.0049 (6)
O2	0.0413 (6)	0.0893 (9)	0.0505 (6)	−0.0069 (6)	0.0168 (5)	−0.0118 (6)
N1	0.0533 (7)	0.0477 (7)	0.0460 (7)	−0.0005 (5)	0.0201 (6)	0.0009 (5)
N2	0.0658 (10)	0.0767 (11)	0.0394 (7)	−0.0037 (8)	−0.0085 (7)	0.0020 (7)
C1	0.0514 (9)	0.0459 (8)	0.0602 (10)	−0.0043 (7)	0.0018 (7)	−0.0025 (7)
C2	0.0580 (9)	0.0544 (9)	0.0467 (8)	−0.0048 (7)	0.0082 (7)	0.0049 (7)
C11	0.0386 (7)	0.0491 (8)	0.0382 (7)	−0.0074 (6)	0.0089 (5)	−0.0069 (6)
C12	0.0367 (7)	0.0504 (8)	0.0478 (8)	−0.0058 (6)	0.0021 (6)	−0.0117 (6)
C13	0.0494 (9)	0.0730 (12)	0.0643 (11)	0.0018 (8)	0.0120 (8)	−0.0241 (9)
C14	0.0543 (10)	0.1020 (16)	0.0522 (10)	−0.0112 (10)	0.0210 (8)	−0.0224 (10)
C15	0.0631 (10)	0.0831 (13)	0.0444 (8)	−0.0183 (10)	0.0172 (8)	−0.0028 (9)
C16	0.0612 (10)	0.0570 (9)	0.0472 (8)	−0.0061 (8)	0.0174 (7)	0.0004 (7)
C21	0.0431 (7)	0.0464 (8)	0.0408 (7)	0.0043 (6)	0.0059 (6)	−0.0059 (6)
C22	0.0404 (7)	0.0561 (9)	0.0410 (7)	0.0047 (6)	0.0063 (6)	−0.0137 (7)
C23	0.0492 (9)	0.0793 (13)	0.0604 (10)	−0.0090 (8)	0.0090 (8)	−0.0296 (9)
C24	0.0846 (15)	0.0786 (14)	0.0913 (16)	−0.0327 (12)	0.0334 (13)	−0.0391 (13)
C25	0.126 (2)	0.0500 (11)	0.0938 (17)	−0.0192 (12)	0.0453 (16)	−0.0082 (11)
C26	0.0888 (14)	0.0504 (10)	0.0582 (10)	0.0058 (9)	0.0139 (9)	0.0003 (8)

Geometric parameters (Å, º)

S1—O1	1.4277 (12)	C13—C14	1.383 (3)
S1—O2	1.4317 (12)	C13—H13	0.9300
S1—N1	1.6205 (14)	C14—C15	1.373 (3)
S1—C21	1.7486 (16)	C14—H14	0.9300
N1—C11	1.4203 (18)	C15—C16	1.387 (2)
N1—C2	1.482 (2)	C15—H15	0.9300
N2—C22	1.358 (2)	C16—H16	0.9300
N2—H2C	0.86 (3)	C21—C26	1.389 (2)
N2—H2D	0.89 (2)	C21—C22	1.414 (2)
C1—C12	1.498 (2)	C22—C23	1.396 (2)

C1—C2	1.514 (2)	C23—C24	1.357 (3)
C1—H1A	0.9700	C23—H23	0.9300
C1—H1B	0.9700	C24—C25	1.378 (4)
C2—H2A	0.9700	C24—H24	0.9300
C2—H2B	0.9700	C25—C26	1.380 (3)
C11—C16	1.381 (2)	C25—H25	0.9300
C11—C12	1.386 (2)	C26—H26	0.9300
C12—C13	1.380 (2)		

O1—S1—O2	119.06 (8)	C12—C13—C14	119.49 (18)
O1—S1—N1	107.04 (7)	C12—C13—H13	120.3
O2—S1—N1	105.10 (8)	C14—C13—H13	120.3
O1—S1—C21	107.35 (8)	C15—C14—C13	120.27 (16)
O2—S1—C21	109.61 (7)	C15—C14—H14	119.9
N1—S1—C21	108.24 (7)	C13—C14—H14	119.9
C11—N1—C2	109.39 (13)	C14—C15—C16	121.27 (18)
C11—N1—S1	126.02 (11)	C14—C15—H15	119.4
C2—N1—S1	122.27 (11)	C16—C15—H15	119.4
C22—N2—H2C	115.9 (18)	C11—C16—C15	117.89 (18)
C22—N2—H2D	116.2 (15)	C11—C16—H16	121.1
H2C—N2—H2D	120 (2)	C15—C16—H16	121.1
C12—C1—C2	104.35 (13)	C26—C21—C22	120.97 (16)
C12—C1—H1A	110.9	C26—C21—S1	118.22 (13)
C2—C1—H1A	110.9	C22—C21—S1	120.75 (12)
C12—C1—H1B	110.9	N2—C22—C23	119.58 (16)
C2—C1—H1B	110.9	N2—C22—C21	123.81 (15)
H1A—C1—H1B	108.9	C23—C22—C21	116.57 (17)
N1—C2—C1	104.80 (13)	C24—C23—C22	121.75 (19)
N1—C2—H2A	110.8	C24—C23—H23	119.1
C1—C2—H2A	110.8	C22—C23—H23	119.1
N1—C2—H2B	110.8	C23—C24—C25	121.5 (2)
C1—C2—H2B	110.8	C23—C24—H24	119.3
H2A—C2—H2B	108.9	C25—C24—H24	119.3
C16—C11—C12	121.52 (14)	C24—C25—C26	118.9 (2)
C16—C11—N1	129.33 (15)	C24—C25—H25	120.5
C12—C11—N1	109.13 (13)	C26—C25—H25	120.5
C13—C12—C11	119.57 (16)	C25—C26—C21	120.1 (2)
C13—C12—C1	130.07 (16)	C25—C26—H26	119.9
C11—C12—C1	110.35 (14)	C21—C26—H26	119.9

O1—S1—N1—C11	−48.70 (15)	C13—C14—C15—C16	−0.1 (3)
O2—S1—N1—C11	−176.21 (12)	C12—C11—C16—C15	0.0 (2)
C21—S1—N1—C11	66.72 (14)	N1—C11—C16—C15	178.04 (15)
O1—S1—N1—C2	150.47 (13)	C14—C15—C16—C11	−0.1 (3)
O2—S1—N1—C2	22.96 (14)	O1—S1—C21—C26	12.71 (16)
C21—S1—N1—C2	−94.10 (13)	O2—S1—C21—C26	143.37 (14)
C11—N1—C2—C1	14.13 (17)	N1—S1—C21—C26	−102.51 (14)
S1—N1—C2—C1	177.78 (11)	O1—S1—C21—C22	−169.99 (12)

C12—C1—C2—N1	−12.92 (16)	O2—S1—C21—C22	−39.34 (14)
C2—N1—C11—C16	172.19 (16)	N1—S1—C21—C22	74.79 (13)
S1—N1—C11—C16	9.3 (2)	C26—C21—C22—N2	−178.22 (17)
C2—N1—C11—C12	−9.61 (16)	S1—C21—C22—N2	4.6 (2)
S1—N1—C11—C12	−172.49 (11)	C26—C21—C22—C23	4.0 (2)
C16—C11—C12—C13	0.1 (2)	S1—C21—C22—C23	−173.18 (12)
N1—C11—C12—C13	−178.25 (13)	N2—C22—C23—C24	−179.93 (19)
C16—C11—C12—C1	179.21 (14)	C21—C22—C23—C24	−2.1 (3)
N1—C11—C12—C1	0.84 (16)	C22—C23—C24—C25	−1.0 (3)
C2—C1—C12—C13	−173.18 (16)	C23—C24—C25—C26	2.1 (4)
C2—C1—C12—C11	7.85 (17)	C24—C25—C26—C21	−0.1 (3)
C11—C12—C13—C14	−0.2 (2)	C22—C21—C26—C25	−3.0 (3)
C1—C12—C13—C14	−179.12 (16)	S1—C21—C26—C25	174.28 (16)
C12—C13—C14—C15	0.2 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*C*···O2	0.86 (3)	2.19 (3)	2.857 (2)	134 (2)
N2—H2*D*···O1^{i}	0.89 (2)	2.22 (3)	3.098 (2)	169 (2)
C15—H15···O2^{ii}	0.93	2.59	3.366 (2)	141
C16—H16···O1	0.93	2.58	3.105 (2)	117
C26—H26···O1	0.93	2.45	2.849 (3)	106

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