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# Syntheses, crystal structures and Hirshfeld surface analyses of *N*-arylsulfonyl derivatives of cytisine

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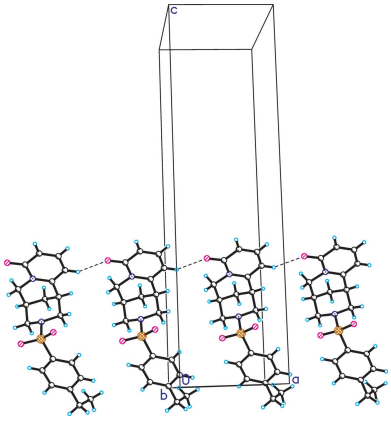
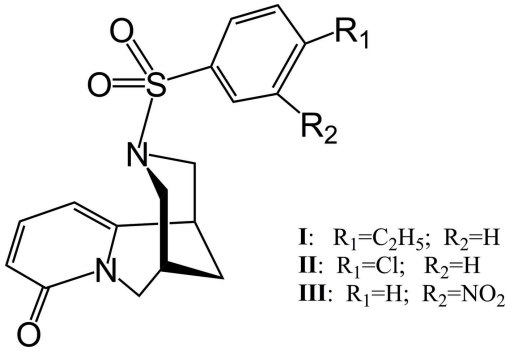
By arylsulfonylation of cytisine in the presence of triethylamine, three new compounds have been obtained in good yields: (7*R*,9*R*)-*N*-[(4-ethylphenyl)sulfonyl]cytisine, $C_{19}H_{22}N_2O_3S$ (**I**) {systematic name: (1*R*,5*R*)-3-[(4-ethylphenyl)sulfonyl]-1,2,3,4,5,6-hexahydro-8*H*-1,5-methanopyrido[1,2-*a*][1,5]diazocin-8-one}, (7*R*,9*R*)-*N*-[(4-chlorophenyl)sulfonyl]cytisine, $C_{17}H_{17}ClN_2O_3S$ (**II**) {systematic name: (1*R*,5*R*)-3-[(4-chlorophenyl)sulfonyl]-1,2,3,4,5,6-hexahydro-8*H*-1,5-methanopyrido[1,2-*a*][1,5]diazocin-8-one} and (7*R*,9*R*)-*N*-[(3-nitrophenyl)sulfonyl]cytisine, $C_{17}H_{17}N_3O_5S$ (**III**) {systematic name: (1*R*,5*R*)-3-[(3-nitrophenyl)sulfonyl]-1,2,3,4,5,6-hexahydro-8*H*-1,5-methanopyrido[1,2-*a*]-[1,5]diazocin-8-one}. The crystal structures of the compounds were determined on the basis of single-crystal X-ray diffraction data. The crystal structures of (**I**)–(**III**) are distinguished by the arrangement of two fragments of the molecule around the sulfonyl site. For all structures, weak C—H···O hydrogen bonds are developed. Hirshfeld surface analysis shows that H···H (for **I** and **II**) and H···O/O···H (for **III**) interactions make the most important contribution to the crystal packing.

## 1. Chemical context

Cytisine was first isolated by Huzeman and Marme from the seeds of *Cytisus Laburnum* Med. in 1865. To this day, other sources of cytisine have been found (Azimova & Yunusov, 2013), mainly isolated from plants of the legume family (especially the seeds of *Laburnum anagyroides*). Cytisine is a quinolizidine alkaloid, which is found in different sources by different names: 1,2,3,4,5,6-hexahydro-1,5-methano-8*H*-pyrido(1,2-*a*)(1,5)-diazocin-8-one (Freer *et al.*, 1987), 7,11-diazatricyclo[7.3.1.$0^{2,7}$]trideca-2,4-dien-6-one (Kulakov *et al.*, 2010), (1*R*,5*S*)-cytisine (Rouden *et al.*, 2014) or (7*R*,9*S*)-cytisine.

Various studies report modern methods for the synthesis of cytisine (Barát *et al.*, 2018; Hirschhäuser *et al.*, 2011; O'Neill *et al.*, 2000; Pérez *et al.*, 2012) or cytisine modification (Brel, 2016; Kulakov *et al.*, 2010; Kulakov & Nurkenov, 2012; Shishkin *et al.*, 2010; Marrière *et al.*, 2000; Frasinyuk *et al.*, 2007). From the large number of cytisine derivatives, substances with biological activity (Tutka *et al.*, 2019; Gotti & Clementi, 2021; Liu *et al.*, 2020) and agents used in medicine (Tabex) have been found. From a chemical point of view, derivation studies of cytisine as well as the development of new methods for the synthesis of various cytisine derivatives are of interest.






**I**: $R_1$=$C_2H_5$; $R_2$=H
**II**: $R_1$=Cl; $R_2$=H
**III**: $R_1$=H; $R_2$=$NO_2$

This communication describes the synthesis and crystal structures of three *N*-arylsulfonyl derivatives of cytisine. To obtain these *N*-arylsulfonyl derivatives, 4-ethylbenzenesulfonyl chloride, 4-chlorobenzenesulfonyl chloride and 3-nitrobenzenesulfonyl chloride were used, resulting in (7*R*,9*R*)-*N*-[(4-ethylphenyl)sulfonyl]cytisine (**I**), (7*R*,9*R*)-*N*-[(4-chlorophenyl)sulfonyl]cytisine (**II**) and (7*R*,9*R*)-*N*-[(3-nitrophenyl)sulfonyl]cytisine (**III**).

## 2. Structural commentary

The conformations of the cytisine cores in structures (**I**)–(**III**) are virtually identical and also do not differ from that of the cytisine molecule itself (Freer *et al.*, 1987), or its various *N*-derivatives. The configurations of the chiral C atoms in cytisine are 7*R*, 9*S*, whereas in the case of (**I**)–(**III**) obtained by arylsulfonation of cytisine, the configurations are 7*R*, 9*R* in each case.

The asymmetric unit of (**I**) consists of one molecule of (7*R*,9*R*)-*N*-[(4-ethylphenyl)sulfonyl]cytisine (Fig. 1). The methyl fragment (C8′*A*, C8′*B*) of the ethyl group bound to the phenyl ring is disordered over two sets of sites. In the crystal structures of (**II**) and (**III**), both asymmetric units likewise comprise one molecule of (7*R*,9*R*)-*N*-[(4-chlorophenyl)sulfonyl]cytisine and (7*R*,9*R*)-*N*-[(3-nitrophenyl)sulfonyl]cytisine, respectively (Figs. 2, 3). The cytisine moieties in (**I**)–(**III**) are almost superimposable in the three molecules (Fig. 4). Basically, the difference in the molecular structures pertains to the arrangement of two fragments around the sulfonyl site, *i.e.* the arrangement of fragments along the S1—N12 and S1—C1′ bonds). Corresponding torsion angles C1′—S1—N12—C11 and N12—S1—C1′—C2′ are listed in Tables 1, 2 and 3.

**Table 1**
Selected torsion angles (°) for (**I**).

| | | | |
|---|---|---|---|
| C1′—S1—N12—C11 | 76.6 (2) | N12—S1—C1′—C2′ | −79.4 (2) |

**Table 2**
Selected torsion angles (°) for (**II**).

| | | | |
|---|---|---|---|
| C1′—S1—N12—C11 | 72.1 (2) | N12—S1—C1′—C2′ | −79.4 (2) |

**Table 3**
Selected torsion angles (°) for (**III**).

| | | | |
|---|---|---|---|
| C1′—S1—N12—C11 | 57.3 (3) | N12—S1—C1′—C2′ | −87.9 (3) |

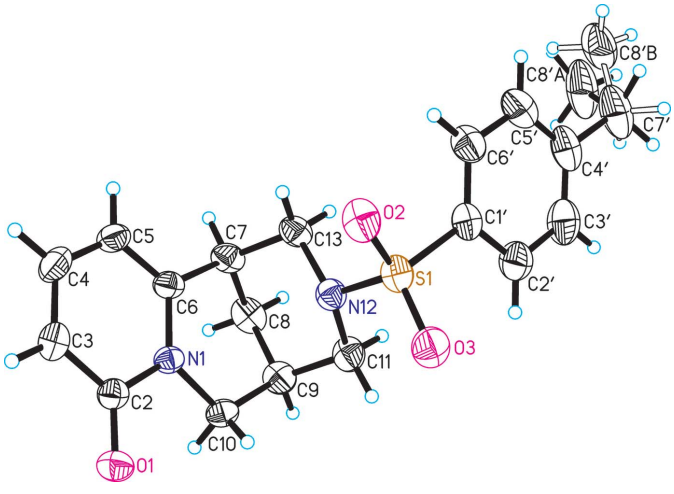



**Figure 1**
The asymmetric unit of (**I**) with atom labelling. Displacement ellipsoids represent 30% probability levels.

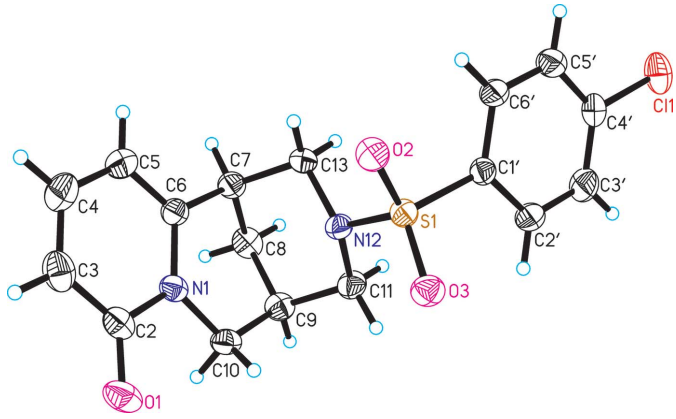



**Figure 2**
The asymmetric unit of (**II**) with atom labelling. Displacement ellipsoids represent 30% probability levels.

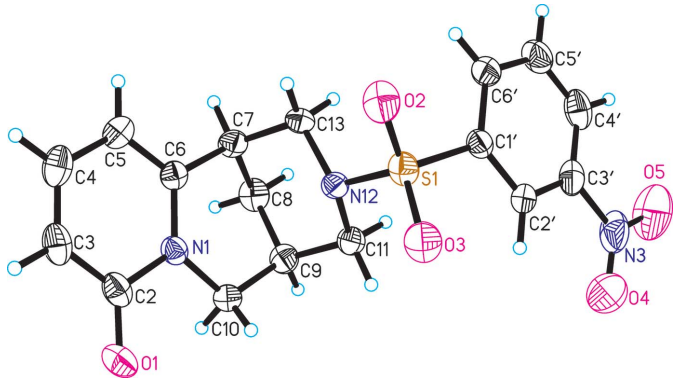



**Figure 3**
The asymmetric unit of (**III**) with atom labelling. Displacement ellipsoids represent 30% probability levels.

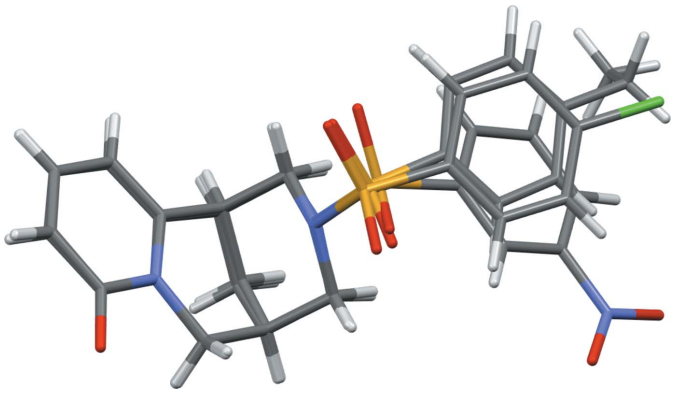


**Figure 4**
Overlay plot of the molecules in the crystal structures of (**I**)–(**III**).

## 3. Supramolecular features

In the crystal packing of (**I**)–(**III**), weak intermolecular hydrogen bonds of the type C—H···O(C) are developed. In the crystal structures of (**I**) and (**II**), C—H···O1 hydrogen bonds link molecules into chains directed parallel to [100] (Figs. 5, 6), besides other C—H···O or C—H···Cl (in the case of **II**) interactions (Tables 4, 5). In the crystal structure of (**III**), the C—H···O1 interactions link the molecules into a chain running along [1$\bar{1}$0] (Fig. 7, Table 6).

In order to visualize and quantify intermolecular interactions in (**I**)–(**III**), a Hirshfeld surface analysis (Spackman & Jayatilaka, 2009) was performed with *Crystal Explorer 21* (Spackman *et al.*, 2021), and the associated two-dimensional fingerprint plots (McKinnon *et al.*, 2007) generated.

**Table 4**
Hydrogen-bond geometry (Å, °) for (**I**).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C5—H5*A*···O1$^{i}$ | 0.93 | 2.34 | 3.116 (3) | 141 |
| C7—H7*A*···O2$^{ii}$ | 0.98 | 2.44 | 3.254 (3) | 140 |

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

**Table 5**
Hydrogen-bond geometry (Å, °) for (**II**).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C5—H5*A*···O1$^{i}$ | 0.93 | 2.61 | 3.375 (4) | 140 |
| C13—H13*B*···O1$^{i}$ | 0.97 | 2.69 | 3.475 (3) | 139 |
| C5′—H5′*A*···O3$^{i}$ | 0.93 | 2.42 | 3.198 (3) | 142 |
| C8—H8*A*···O3$^{ii}$ | 0.97 | 2.56 | 3.424 (3) | 149 |
| C4—H4*A*···Cl1$^{iii}$ | 0.93 | 2.94 | 3.764 (3) | 148 |

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

**Table 6**
Hydrogen-bond geometry (Å, °) for (**III**).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C4′—H4′*A*···O1$^{i}$ | 0.93 | 2.61 | 3.257 (5) | 127 |
| C10—H10*A*···O5$^{ii}$ | 0.97 | 2.58 | 3.460 (6) | 151 |
| C11—H11*A*···O5$^{iii}$ | 0.97 | 2.55 | 3.428 (7) | 150 |
| C13—H13*A*···O3$^{iv}$ | 0.97 | 2.46 | 3.330 (4) | 150 |

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

The Hirshfeld surfaces for the molecules in (**I**)–(**III**) are shown in Figs. 8–10 in which the two-dimensional fingerprint plots of the most dominant contacts are also presented.

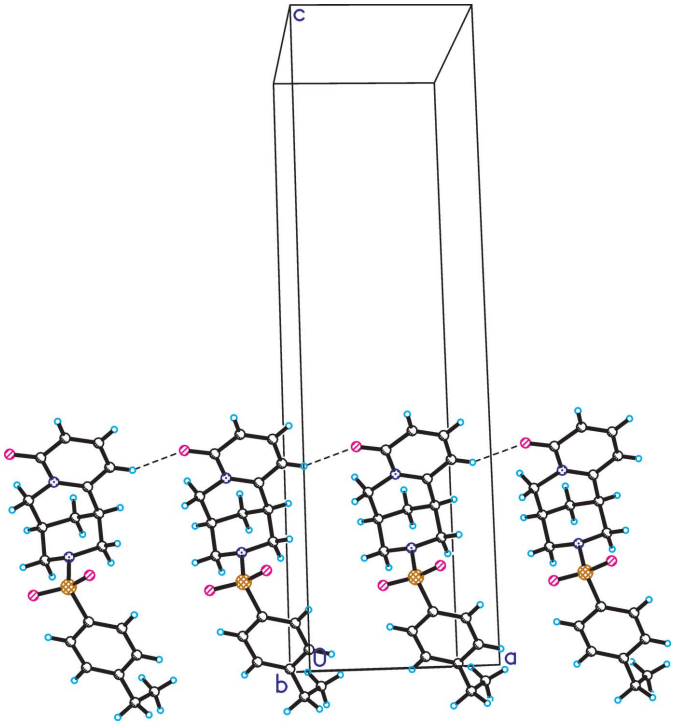



**Figure 5**
The observed C5—H···O1 hydrogen bond in the crystal structure of (**I**). For clarity, the disordered methyl fragment is not shown.

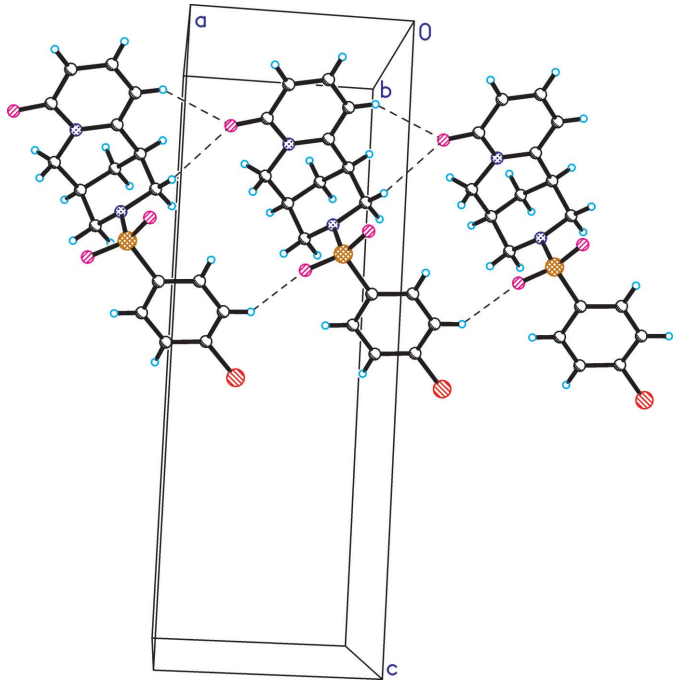



**Figure 6**
The observed hydrogen bonds (C5—H···O1, C13—H···O1, C5′—H···O3) in the crystal structure of (**II**).

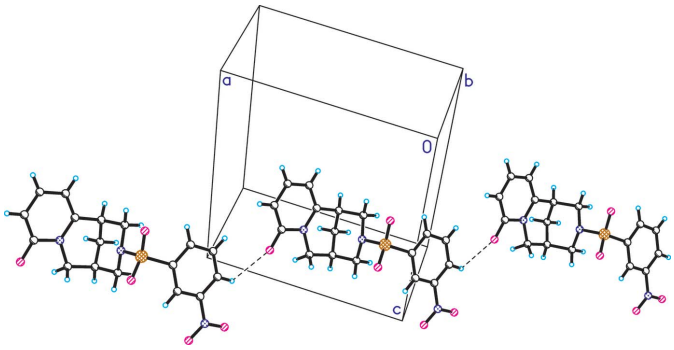



Figure 7
The observed hydrogen bonds (C4′—H···O1) in the crystal structure of (**III**).

For structure (**I**), H···H contacts are responsible for the largest contribution (54.9%) to the Hirshfeld surface. Besides these contacts, H···O/O···H (26.2%) and H···C/C···H (16.7%) interactions contribute significantly to the total Hirshfeld surface (Fig. 8). The contributions of further contacts are only minor and amount to H···N/N···H (1.8%), C···C (0.2%) and H···S/S···H (0.1%).

In structure (**II**), the contribution percentages of the most significant contacts change because of the presence of H···Cl/Cl···H interactions and amount to H···H (38.9%), H···O/O···H (25.4%), H···C/C···H (16.7%) and H···Cl/Cl···H (10.9%) (Fig. 9). The contributions of further contacts are only minor and are Cl···O/O···Cl (2.4%), Cl···C/C···Cl (1.8%), C···O/O···C (1.7%), H···N/N···H (1.6%), C···C (0.3%) and Cl···S/S···Cl (0.1%).

In structure (**III**), the existence of a nitro group likewise changes the contributions of the significant interactions: H···O/O···H (44.3%), H···H (33.3%) and H···C/C···H (10.2%) (Fig. 10). Other minor contributions amount to C···C (3.8%), C···O/O···C (3.2%), H···N/N···H (2.5%), O···N/N···O (1.3%), O···O (1.2%) and C···N/N···C (0.2%).

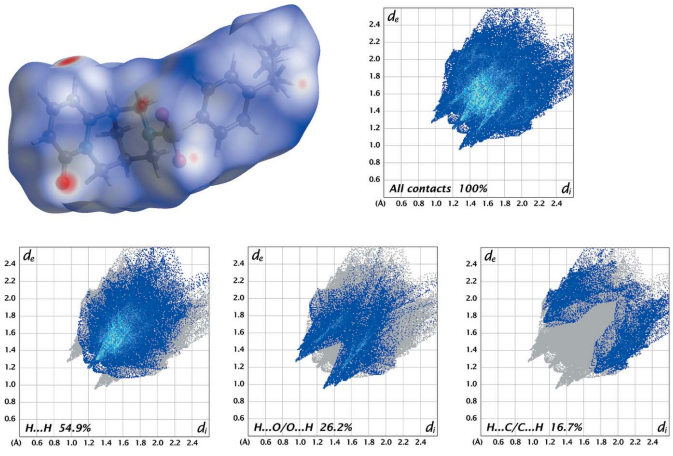



Figure 8
Three-dimensional Hirshfeld surfaces of the compound (**I**) plotted over $d_{norm}$ in the range −0.2931 to 1.5624 a.u., and Hirshfeld fingerprint plots for all contacts and decomposed into H···H, H···O/O···H and H···C/C···H contacts. $d_i$ and $d_e$ denote the closest internal and external distances (in Å) from a point on the surface.

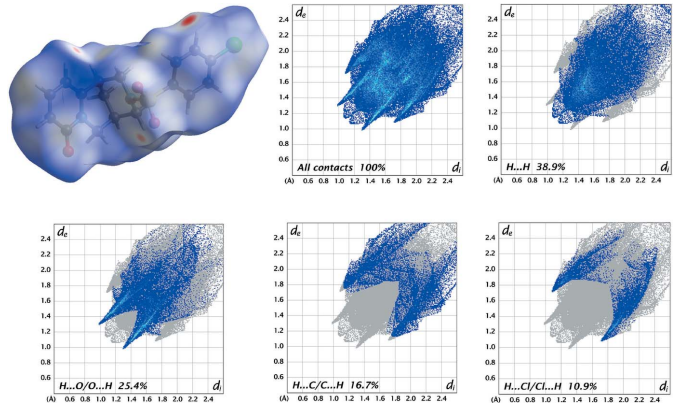



Figure 9
Three-dimensional Hirshfeld surfaces of the compound (**II**) plotted over $d_{norm}$ in the range −0.2332 to 1.6350 a.u., and Hirshfeld fingerprint plots for all contacts and decomposed into H···H, H···O/O···H, H···C/C···H and H···Cl/Cl···H contacts. $d_i$ and $d_e$ denote the closest internal and external distances (in Å) from a point on the surface.

## 4. Database survey

A Cambridge Structural Database search (version 2022.3.0; Groom *et al.*, 2016) revealed 99 *N*-derivatives of cytisine, of which twelve are *N*-benzyl derivatives of cytisine. *N*-arylsulfonylcytisine derivatives are not found. The most similar structure with respect to (**I**)–(**III**) is 3-[(4-bromophenyl)-methyl]-8-oxo-1,3,4,5,6,8-hexahydro-2*H*-1,5-methanopyrido-[1,2-*a*][1,5]diazocin-3-ium perchlorate (KINBOB; Przybył *et al.*, 2019).

## 5. Synthesis and crystallization

**General method**

Arylsulfonyl chloride (0.01 mol) and 10 ml of acetone were placed in a two-necked flask with a volume of 50 ml. After cooling, a previously prepared solution (1.9 g (0.01 mol) of

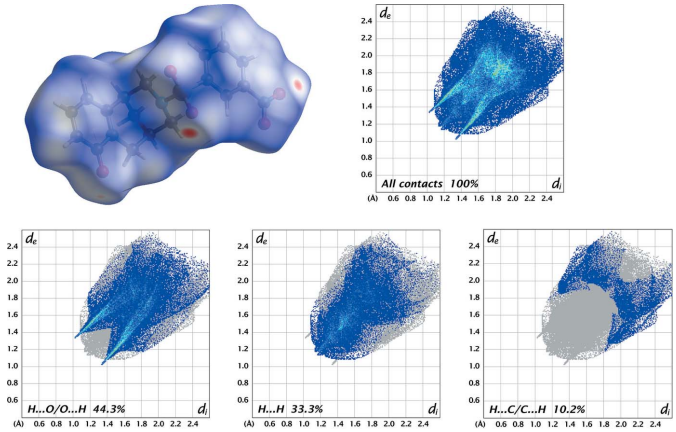



Figure 10
Three-dimensional Hirshfeld surfaces of the compound (**III**) plotted over $d_{norm}$ in the range −0.1815 to 1.3331 a.u., and Hirshfeld fingerprint plots for all contacts and decomposed into H···O/O···H, H···H and H···C/C···H contacts. $d_i$ and $d_e$ denote the closest internal and external distances (in Å) from a point on the surface.

**Table 7**
Experimental details.

| | (I) | (II) | (III) |
|---|---|---|---|
| Crystal data | | | |
| Chemical formula | $C_{19}H_{22}N_2O_3S$ | $C_{17}H_{17}ClN_2O_3S$ | $C_{17}H_{17}N_3O_5S$ |
| $M_r$ | 358.44 | 364.83 | 375.39 |
| Crystal system, space group | Orthorhombic, $P2_12_12_1$ | Orthorhombic, $P2_12_12_1$ | Monoclinic, $P2_1$ |
| Temperature (K) | 299 | 299 | 296 |
| $a, b, c$ (Å) | 6.9503 (14), 10.585 (2), 24.975 (5) | 7.1374 (14), 11.448 (2), 20.844 (4) | 11.040 (2), 6.2621 (13), 12.424 (3) |
| $\alpha, \beta, \gamma$ (°) | 90, 90, 90 | 90, 90, 90 | 90, 94.03 (3), 90 |
| $V$ (Å$^3$) | 1837.5 (6) | 1703.2 (6) | 856.8 (3) |
| $Z$ | 4 | 4 | 2 |
| Radiation type | Cu $K\alpha$ | Cu $K\alpha$ | Cu $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 1.73 | 3.29 | 2.00 |
| Crystal size (mm) | 0.25 × 0.20 × 0.10 | 0.30 × 0.20 × 0.15 | 0.20 × 0.15 × 0.10 |
| Data collection | | | |
| Diffractometer | XtaLAB Synergy, Single source at home/near, HyPix3000 | XtaLAB Synergy, Single source at home/near, HyPix3000 | XtaLAB Synergy, Single source at home/near, HyPix3000 |
| Absorption correction | Multi-scan (*SADABS*; Krause *et al.*, 2015) | Multi-scan (*SADABS*; Krause *et al.*, 2015) | Multi-scan (*SADABS*; Krause *et al.*, 2015) |
| $T_{min}$, $T_{max}$ | 0.032, 1.000 | 0.795, 1.000 | 0.639, 1.000 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 17786, 3553, 3348 | 16028, 3289, 3203 | 7776, 2446, 2363 |
| $R_{int}$ | 0.054 | 0.027 | 0.023 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.615 | 0.615 | 0.615 |
| Refinement | | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.038, 0.094, 1.11 | 0.030, 0.078, 1.05 | 0.039, 0.114, 1.04 |
| No. of reflections | 3553 | 3289 | 2446 |
| No. of parameters | 238 | 217 | 235 |
| No. of restraints | 0 | 0 | 1 |
| H-atom treatment | H-atom parameters constrained | H-atom parameters constrained | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.22, −0.44 | 0.42, −0.33 | 0.47, −0.22 |
| Absolute structure | Flack $x$ determined using 1319 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013) | Flack $x$ determined using 1318 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013) | Flack $x$ determined using 579 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013) |
| Absolute structure parameter | 0.012 (9) | −0.004 (5) | 0.017 (16) |

Computer programs: *CrysAlis PRO* (Rigaku OD, 2021), *SHELXS97* (Sheldrick, 2008), *SHELXL2018/3* (Sheldrick, 2015), *XP* in *SHELXTL* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2020), *PLATON* (Spek, 2020) and *publCIF* (Westrip, 2010).

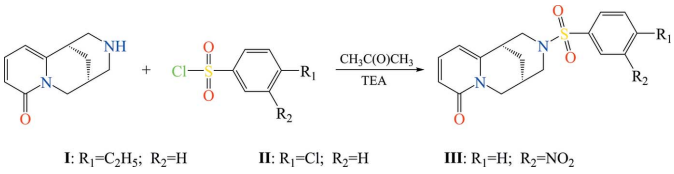
cytisine and 0.01 mol of triethylamine in 15 ml of acetone) was added under stirring through a separatory funnel. The reaction mixture was stirred at room temperature for 10 h. The reaction mixture was then left in the open air overnight to produce a dry mass. The mass was treated with 15 ml of distilled water and the remaining solid filtered off and dried in air. The reaction scheme is shown in Fig. 11.

**(7*R*,9*R*)-*N*-[(4-ethylphenyl)sulfonyl]cytisine (I)**

Yield 64% (2.29 g), m.p. 456–458 K, $R_f$ = 0.59 [5:1 (*v*/*v*) benzene–ethanol].

**(7*R*,9*R*)-*N*-[(4-chlorophenyl)sulfonyl]cytisine (II)**

Yield 76% (2.77 g), m.p. 488–490 K, $R_f$ = 0.71 [5:1 (*v*/*v*) benzene–ethanol].




**I**: $R_1$=$C_2H_5$; $R_2$=H **II**: $R_1$=Cl; $R_2$=H **III**: $R_1$=H; $R_2$=$NO_2$

**Figure 11**
General reaction scheme for the synthesis of *N*-arylsulfonyl derivatives of cytisine.

**(7*R*,9*R*)-*N*-[(3-nitrophenyl)sulfonyl]cytisine (III)**

Yield 72% (2.71 g), m.p. 524–526 K, $R_f$ = 0.50 [5:1 (*v*/*v*) benzene–ethanol].

Colourless crystals of (**I**)–(**III**) suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 7. In (**I**), the methyl C8′ atom is disordered over two positions (C8′*A*, C8′*B*). The site occupancy factors of the disordered fragment were refined with a free variable to a ratio of 0.55 (2):0.45 (2). Hydrogen atoms bonded to C atoms were placed geometrically (with C—H distances of 0.98 Å for CH, 0.97 Å for $CH_2$, 0.96 Å for $CH_3$ and 0.93 Å for $C_{ar}$) and included in the refinement in a riding motion approximation with $U_{iso}$(H) = 1.2$U_{eq}$(C) or $U_{iso}$ = 1.5$U_{eq}$(C) for methyl H atoms.

## Acknowledgements

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# supporting information

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## Syntheses, crystal structures and Hirshfeld surface analyses of *N*-arylsulfonyl derivatives of cytisine

**Rasul Ya. Okmanov, Manzura I. Olimova, Surayyo B. Karabaeva, Frunza A. Sapaev and Kudaybergen B. Abdireymov**

### Computing details

For all structures, data collection: *CrysAlis PRO* (Rigaku OD, 2021); cell refinement: *CrysAlis PRO* (Rigaku OD, 2021); data reduction: *CrysAlis PRO* (Rigaku OD, 2021); program(s) used to solve structure: *SHELXS7* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2020); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2015), *PLATON* (Spek, 2020) and *publCIF* (Westrip, 2010).

### (1*R*,5*R*)-3-[(4-Ethylphenyl)sulfonyl]-1,2,3,4,5,6-hexahydro-8*H*-1,5-methanopyrido[1,2-*a*][1,5]diazocin-8-one (I)

*Crystal data*

$C_{19}H_{22}N_2O_3S$
$M_r$ = 358.44
Orthorhombic, $P2_12_12_1$
$a$ = 6.9503 (14) Å
$b$ = 10.585 (2) Å
$c$ = 24.975 (5) Å
$V$ = 1837.5 (6) Å$^3$
$Z$ = 4
$F(000)$ = 760
$D_x$ = 1.296 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54184 Å
Cell parameters from 10591 reflections
$\theta$ = 3.5–70.9°
$\mu$ = 1.73 mm$^{-1}$
$T$ = 299 K
Prizmatic, colorless
0.25 × 0.20 × 0.10 mm

*Data collection*

XtaLAB Synergy, Single source at home/near, HyPix3000 diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Detector resolution: 10.0000 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
$T_{min}$ = 0.032, $T_{max}$ = 1.000
17786 measured reflections
3553 independent reflections
3348 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.054
$\theta_{max}$ = 71.4°, $\theta_{min}$ = 3.5°
$h$ = −8→8
$k$ = −12→13
$l$ = −23→30

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.094
$S$ = 1.11
3553 reflections
238 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2 + 0.0534P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å$^{-3}$
$\Delta\rho_{min} = -0.44$ e Å$^{-3}$

Absolute structure: Flack *x* determined using 1319 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.012 (9)

*Special details*

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

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| S1 | 0.72079 (9) | 0.74770 (5) | 0.14537 (2) | 0.05691 (18) | |
| O1 | 0.3969 (2) | 0.73366 (18) | 0.35879 (6) | 0.0609 (4) | |
| O2 | 0.8449 (3) | 0.64623 (16) | 0.16065 (7) | 0.0707 (5) | |
| O3 | 0.5240 (3) | 0.7222 (2) | 0.13301 (8) | 0.0766 (6) | |
| N1 | 0.6396 (3) | 0.83845 (17) | 0.31683 (7) | 0.0442 (4) | |
| N12 | 0.7204 (3) | 0.84820 (18) | 0.19511 (7) | 0.0525 (4) | |
| C2 | 0.5717 (3) | 0.7474 (2) | 0.35284 (8) | 0.0489 (5) | |
| C3 | 0.7162 (4) | 0.6760 (3) | 0.38004 (10) | 0.0620 (6) | |
| H3A | 0.679416 | 0.612285 | 0.403557 | 0.074* | |
| C4 | 0.9054 (4) | 0.6993 (3) | 0.37223 (11) | 0.0660 (7) | |
| H4A | 0.996719 | 0.652682 | 0.390946 | 0.079* | |
| C5 | 0.9646 (4) | 0.7917 (3) | 0.33673 (10) | 0.0605 (6) | |
| H5A | 1.095137 | 0.807418 | 0.331995 | 0.073* | |
| C6 | 0.8326 (3) | 0.8596 (2) | 0.30875 (9) | 0.0492 (5) | |
| C7 | 0.8915 (4) | 0.9562 (3) | 0.26785 (10) | 0.0585 (6) | |
| H7A | 1.017190 | 0.990569 | 0.278075 | 0.070* | |
| C8 | 0.7474 (5) | 1.0639 (2) | 0.26561 (11) | 0.0692 (7) | |
| H8A | 0.736511 | 1.104049 | 0.300400 | 0.083* | |
| H8B | 0.788228 | 1.126854 | 0.239766 | 0.083* | |
| C9 | 0.5559 (4) | 1.0075 (3) | 0.24915 (11) | 0.0612 (7) | |
| H9A | 0.461650 | 1.076197 | 0.247158 | 0.073* | |
| C10 | 0.4861 (3) | 0.9134 (2) | 0.29083 (10) | 0.0524 (5) | |
| H10A | 0.416201 | 0.959033 | 0.318316 | 0.063* | |
| H10B | 0.396456 | 0.855705 | 0.273849 | 0.063* | |
| C11 | 0.5739 (4) | 0.9493 (3) | 0.19342 (10) | 0.0641 (7) | |
| H11A | 0.451036 | 0.914628 | 0.182325 | 0.077* | |
| H11B | 0.611563 | 1.013563 | 0.167793 | 0.077* | |
| C13 | 0.9099 (4) | 0.8943 (3) | 0.21250 (10) | 0.0592 (6) | |
| H13A | 0.958528 | 0.955410 | 0.186892 | 0.071* | |
| H13B | 1.000046 | 0.824482 | 0.214244 | 0.071* | |
| C1′ | 0.8285 (4) | 0.8251 (2) | 0.09016 (9) | 0.0585 (6) | |
| C2′ | 0.7190 (5) | 0.9095 (3) | 0.06041 (11) | 0.0780 (8) | |
| H2′A | 0.590106 | 0.923114 | 0.068566 | 0.094* | |
| C3′ | 0.8057 (7) | 0.9728 (3) | 0.01842 (12) | 0.0894 (11) | |

| | | | | | |
|---|---|---|---|---|---|
| H3′A | 0.732521 | 1.029034 | −0.001706 | 0.107* | |
| C4′ | 0.9946 (6) | 0.9558 (3) | 0.00544 (11) | 0.0819 (10) | |
| C5′ | 1.1005 (6) | 0.8709 (3) | 0.03537 (12) | 0.0849 (9) | |
| H5′A | 1.229082 | 0.857186 | 0.026912 | 0.102* | |
| C6′ | 1.0180 (5) | 0.8052 (3) | 0.07808 (11) | 0.0712 (7) | |
| H6′A | 1.091003 | 0.748680 | 0.098065 | 0.085* | |
| C7′ | 1.0838 (8) | 1.0285 (4) | −0.04059 (14) | 0.1161 (17) | |
| H7′A | 1.164041 | 0.971279 | −0.061123 | 0.139* | 0.55 (2) |
| H7′B | 0.981596 | 1.057716 | −0.063967 | 0.139* | 0.55 (2) |
| H7′C | 1.053155 | 0.983559 | −0.073354 | 0.139* | 0.45 (2) |
| H7′D | 1.019922 | 1.109830 | −0.042531 | 0.139* | 0.45 (2) |
| C8′A | 1.199 (3) | 1.1360 (16) | −0.0246 (4) | 0.136 (7) | 0.55 (2) |
| H8′A | 1.233872 | 1.184056 | −0.055681 | 0.205* | 0.55 (2) |
| H8′B | 1.312957 | 1.107082 | −0.006780 | 0.205* | 0.55 (2) |
| H8′C | 1.125652 | 1.188309 | −0.000638 | 0.205* | 0.55 (2) |
| C8′B | 1.280 (2) | 1.050 (2) | −0.0404 (4) | 0.111 (5) | 0.45 (2) |
| H8′D | 1.312947 | 1.105567 | −0.069535 | 0.166* | 0.45 (2) |
| H8′E | 1.347397 | 0.971814 | −0.044349 | 0.166* | 0.45 (2) |
| H8′F | 1.316353 | 1.089349 | −0.007150 | 0.166* | 0.45 (2) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0714 (4) | 0.0452 (3) | 0.0542 (3) | −0.0046 (3) | 0.0012 (2) | 0.0040 (2) |
| O1 | 0.0488 (9) | 0.0641 (10) | 0.0699 (10) | −0.0067 (8) | 0.0057 (7) | 0.0023 (9) |
| O2 | 0.0982 (14) | 0.0453 (9) | 0.0687 (10) | 0.0091 (9) | 0.0036 (10) | 0.0092 (8) |
| O3 | 0.0790 (13) | 0.0775 (13) | 0.0733 (11) | −0.0218 (11) | −0.0037 (9) | −0.0046 (10) |
| N1 | 0.0400 (9) | 0.0428 (9) | 0.0498 (9) | 0.0003 (7) | −0.0009 (7) | −0.0053 (7) |
| N12 | 0.0558 (11) | 0.0508 (10) | 0.0510 (9) | 0.0010 (9) | 0.0009 (9) | 0.0024 (8) |
| C2 | 0.0483 (11) | 0.0486 (11) | 0.0499 (10) | −0.0027 (10) | −0.0015 (8) | −0.0064 (11) |
| C3 | 0.0698 (16) | 0.0617 (14) | 0.0546 (12) | 0.0022 (13) | −0.0104 (12) | 0.0050 (11) |
| C4 | 0.0597 (15) | 0.0767 (17) | 0.0614 (13) | 0.0145 (13) | −0.0193 (11) | −0.0041 (13) |
| C5 | 0.0406 (11) | 0.0813 (18) | 0.0595 (12) | 0.0023 (11) | −0.0057 (10) | −0.0113 (12) |
| C6 | 0.0419 (11) | 0.0543 (12) | 0.0514 (11) | −0.0040 (10) | 0.0013 (9) | −0.0133 (10) |
| C7 | 0.0522 (13) | 0.0583 (14) | 0.0649 (14) | −0.0145 (11) | 0.0065 (11) | −0.0104 (11) |
| C8 | 0.088 (2) | 0.0459 (12) | 0.0741 (15) | −0.0106 (14) | 0.0117 (15) | −0.0046 (11) |
| C9 | 0.0679 (17) | 0.0469 (13) | 0.0689 (15) | 0.0121 (12) | 0.0039 (14) | 0.0033 (11) |
| C10 | 0.0443 (11) | 0.0503 (12) | 0.0625 (13) | 0.0082 (10) | −0.0002 (10) | −0.0019 (10) |
| C11 | 0.0740 (17) | 0.0561 (14) | 0.0623 (14) | 0.0117 (13) | −0.0004 (13) | 0.0086 (12) |
| C13 | 0.0566 (13) | 0.0607 (14) | 0.0603 (13) | −0.0092 (11) | 0.0106 (11) | 0.0014 (11) |
| C1′ | 0.0768 (17) | 0.0495 (12) | 0.0492 (11) | −0.0015 (12) | 0.0001 (11) | 0.0000 (10) |
| C2′ | 0.087 (2) | 0.0819 (19) | 0.0648 (15) | 0.0054 (18) | −0.0035 (15) | 0.0168 (14) |
| C3′ | 0.126 (3) | 0.079 (2) | 0.0635 (17) | 0.003 (2) | −0.0097 (19) | 0.0196 (15) |
| C4′ | 0.125 (3) | 0.0704 (19) | 0.0503 (14) | −0.0161 (19) | 0.0094 (17) | 0.0020 (13) |
| C5′ | 0.094 (2) | 0.093 (2) | 0.0684 (17) | −0.0076 (19) | 0.0210 (16) | −0.0031 (16) |
| C6′ | 0.0838 (19) | 0.0683 (16) | 0.0615 (14) | 0.0077 (15) | 0.0101 (14) | 0.0040 (13) |
| C7′ | 0.182 (5) | 0.106 (3) | 0.0603 (18) | −0.035 (3) | 0.018 (2) | 0.0090 (19) |
| C8′A | 0.219 (15) | 0.128 (10) | 0.062 (4) | −0.069 (11) | 0.000 (6) | 0.020 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C8′B | 0.130 (9) | 0.134 (12) | 0.069 (5) | −0.043 (8) | 0.023 (5) | 0.010 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—O3 | 1.428 (2) | C10—H10B | 0.9700 |
| S1—O2 | 1.429 (2) | C11—H11A | 0.9700 |
| S1—N12 | 1.635 (2) | C11—H11B | 0.9700 |
| S1—C1′ | 1.770 (3) | C13—H13A | 0.9700 |
| O1—C2 | 1.233 (3) | C13—H13B | 0.9700 |
| N1—C6 | 1.375 (3) | C1′—C6′ | 1.368 (4) |
| N1—C2 | 1.400 (3) | C1′—C2′ | 1.389 (4) |
| N1—C10 | 1.479 (3) | C2′—C3′ | 1.383 (5) |
| N12—C13 | 1.471 (3) | C2′—H2′A | 0.9300 |
| N12—C11 | 1.478 (3) | C3′—C4′ | 1.365 (6) |
| C2—C3 | 1.429 (3) | C3′—H3′A | 0.9300 |
| C3—C4 | 1.352 (4) | C4′—C5′ | 1.381 (5) |
| C3—H3A | 0.9300 | C4′—C7′ | 1.515 (4) |
| C4—C5 | 1.383 (4) | C5′—C6′ | 1.396 (4) |
| C4—H4A | 0.9300 | C5′—H5′A | 0.9300 |
| C5—C6 | 1.359 (3) | C6′—H6′A | 0.9300 |
| C5—H5A | 0.9300 | C7′—C8′B | 1.385 (12) |
| C6—C7 | 1.502 (4) | C7′—C8′A | 1.447 (11) |
| C7—C8 | 1.519 (4) | C7′—H7′A | 0.9700 |
| C7—C13 | 1.535 (3) | C7′—H7′B | 0.9700 |
| C7—H7A | 0.9800 | C7′—H7′C | 0.9700 |
| C8—C9 | 1.516 (4) | C7′—H7′D | 0.9700 |
| C8—H8A | 0.9700 | C8′A—H8′A | 0.9600 |
| C8—H8B | 0.9700 | C8′A—H8′B | 0.9600 |
| C9—C10 | 1.520 (4) | C8′A—H8′C | 0.9600 |
| C9—C11 | 1.527 (4) | C8′B—H8′D | 0.9600 |
| C9—H9A | 0.9800 | C8′B—H8′E | 0.9600 |
| C10—H10A | 0.9700 | C8′B—H8′F | 0.9600 |
| | | | |
| O3—S1—O2 | 119.57 (13) | C9—C11—H11A | 109.9 |
| O3—S1—N12 | 106.61 (11) | N12—C11—H11B | 109.9 |
| O2—S1—N12 | 106.68 (10) | C9—C11—H11B | 109.9 |
| O3—S1—C1′ | 108.91 (13) | H11A—C11—H11B | 108.3 |
| O2—S1—C1′ | 107.49 (13) | N12—C13—C7 | 109.45 (19) |
| N12—S1—C1′ | 106.94 (11) | N12—C13—H13A | 109.8 |
| C6—N1—C2 | 122.35 (18) | C7—C13—H13A | 109.8 |
| C6—N1—C10 | 123.50 (19) | N12—C13—H13B | 109.8 |
| C2—N1—C10 | 114.07 (17) | C7—C13—H13B | 109.8 |
| C13—N12—C11 | 112.7 (2) | H13A—C13—H13B | 108.2 |
| C13—N12—S1 | 116.04 (16) | C6′—C1′—C2′ | 120.6 (3) |
| C11—N12—S1 | 116.78 (16) | C6′—C1′—S1 | 120.5 (2) |
| O1—C2—N1 | 119.4 (2) | C2′—C1′—S1 | 118.9 (2) |
| O1—C2—C3 | 125.0 (2) | C3′—C2′—C1′ | 118.6 (3) |
| N1—C2—C3 | 115.6 (2) | C3′—C2′—H2′A | 120.7 |

| | | | |
|---|---|---|---|
| C4—C3—C2 | 121.2 (3) | C1′—C2′—H2′A | 120.7 |
| C4—C3—H3A | 119.4 | C4′—C3′—C2′ | 122.4 (3) |
| C2—C3—H3A | 119.4 | C4′—C3′—H3′A | 118.8 |
| C3—C4—C5 | 120.7 (2) | C2′—C3′—H3′A | 118.8 |
| C3—C4—H4A | 119.6 | C3′—C4′—C5′ | 118.0 (3) |
| C5—C4—H4A | 119.6 | C3′—C4′—C7′ | 120.4 (4) |
| C6—C5—C4 | 120.2 (2) | C5′—C4′—C7′ | 121.6 (4) |
| C6—C5—H5A | 119.9 | C4′—C5′—C6′ | 121.3 (3) |
| C4—C5—H5A | 119.9 | C4′—C5′—H5′A | 119.4 |
| C5—C6—N1 | 119.8 (2) | C6′—C5′—H5′A | 119.4 |
| C5—C6—C7 | 121.7 (2) | C1′—C6′—C5′ | 119.2 (3) |
| N1—C6—C7 | 118.5 (2) | C1′—C6′—H6′A | 120.4 |
| C6—C7—C8 | 110.9 (2) | C5′—C6′—H6′A | 120.4 |
| C6—C7—C13 | 110.2 (2) | C8′B—C7′—C4′ | 119.0 (6) |
| C8—C7—C13 | 110.0 (2) | C8′A—C7′—C4′ | 114.5 (5) |
| C6—C7—H7A | 108.6 | C8′A—C7′—H7′A | 108.6 |
| C8—C7—H7A | 108.6 | C4′—C7′—H7′A | 108.6 |
| C13—C7—H7A | 108.6 | C8′A—C7′—H7′B | 108.6 |
| C9—C8—C7 | 107.0 (2) | C4′—C7′—H7′B | 108.6 |
| C9—C8—H8A | 110.3 | H7′A—C7′—H7′B | 107.6 |
| C7—C8—H8A | 110.3 | C8′B—C7′—H7′C | 107.6 |
| C9—C8—H8B | 110.3 | C4′—C7′—H7′C | 107.6 |
| C7—C8—H8B | 110.3 | C8′B—C7′—H7′D | 107.6 |
| H8A—C8—H8B | 108.6 | C4′—C7′—H7′D | 107.6 |
| C8—C9—C10 | 110.6 (2) | H7′C—C7′—H7′D | 107.0 |
| C8—C9—C11 | 109.5 (2) | C7′—C8′A—H8′A | 109.5 |
| C10—C9—C11 | 112.7 (2) | C7′—C8′A—H8′B | 109.5 |
| C8—C9—H9A | 107.9 | H8′A—C8′A—H8′B | 109.5 |
| C10—C9—H9A | 107.9 | C7′—C8′A—H8′C | 109.5 |
| C11—C9—H9A | 107.9 | H8′A—C8′A—H8′C | 109.5 |
| N1—C10—C9 | 115.0 (2) | H8′B—C8′A—H8′C | 109.5 |
| N1—C10—H10A | 108.5 | C7′—C8′B—H8′D | 109.5 |
| C9—C10—H10A | 108.5 | C7′—C8′B—H8′E | 109.5 |
| N1—C10—H10B | 108.5 | H8′D—C8′B—H8′E | 109.5 |
| C9—C10—H10B | 108.5 | C7′—C8′B—H8′F | 109.5 |
| H10A—C10—H10B | 107.5 | H8′D—C8′B—H8′F | 109.5 |
| N12—C11—C9 | 108.8 (2) | H8′E—C8′B—H8′F | 109.5 |
| N12—C11—H11A | 109.9 | | |

| | | | |
|---|---|---|---|
| O3—S1—N12—C13 | −176.35 (18) | C8—C9—C10—N1 | −35.6 (3) |
| O2—S1—N12—C13 | 54.8 (2) | C11—C9—C10—N1 | 87.4 (3) |
| C1′—S1—N12—C13 | −60.0 (2) | C13—N12—C11—C9 | −58.7 (3) |
| O3—S1—N12—C11 | −39.7 (2) | S1—N12—C11—C9 | 163.28 (18) |
| O2—S1—N12—C11 | −168.57 (19) | C8—C9—C11—N12 | 60.8 (3) |
| C1′—S1—N12—C11 | 76.6 (2) | C10—C9—C11—N12 | −62.8 (3) |
| C6—N1—C2—O1 | 179.2 (2) | C11—N12—C13—C7 | 57.4 (3) |
| C10—N1—C2—O1 | 2.3 (3) | S1—N12—C13—C7 | −164.26 (18) |
| C6—N1—C2—C3 | −1.1 (3) | C6—C7—C13—N12 | 64.3 (3) |

| | | | |
|---|---|---|---|
| C10—N1—C2—C3 | −178.03 (19) | C8—C7—C13—N12 | −58.3 (3) |
| O1—C2—C3—C4 | −178.3 (2) | O3—S1—C1′—C6′ | −146.7 (2) |
| N1—C2—C3—C4 | 2.1 (3) | O2—S1—C1′—C6′ | −15.8 (3) |
| C2—C3—C4—C5 | −1.3 (4) | N12—S1—C1′—C6′ | 98.4 (2) |
| C3—C4—C5—C6 | −0.7 (4) | O3—S1—C1′—C2′ | 35.4 (3) |
| C4—C5—C6—N1 | 1.7 (4) | O2—S1—C1′—C2′ | 166.3 (2) |
| C4—C5—C6—C7 | −176.9 (2) | N12—S1—C1′—C2′ | −79.4 (2) |
| C2—N1—C6—C5 | −0.7 (3) | C6′—C1′—C2′—C3′ | 0.0 (5) |
| C10—N1—C6—C5 | 175.9 (2) | S1—C1′—C2′—C3′ | 177.9 (3) |
| C2—N1—C6—C7 | 177.89 (18) | C1′—C2′—C3′—C4′ | −0.4 (5) |
| C10—N1—C6—C7 | −5.5 (3) | C2′—C3′—C4′—C5′ | 0.7 (6) |
| C5—C6—C7—C8 | −148.0 (2) | C2′—C3′—C4′—C7′ | −179.3 (3) |
| N1—C6—C7—C8 | 33.4 (3) | C3′—C4′—C5′—C6′ | −0.7 (5) |
| C5—C6—C7—C13 | 90.0 (3) | C7′—C4′—C5′—C6′ | 179.3 (3) |
| N1—C6—C7—C13 | −88.6 (3) | C2′—C1′—C6′—C5′ | 0.0 (4) |
| C6—C7—C8—C9 | −61.4 (3) | S1—C1′—C6′—C5′ | −177.9 (2) |
| C13—C7—C8—C9 | 60.7 (3) | C4′—C5′—C6′—C1′ | 0.4 (5) |
| C7—C8—C9—C10 | 62.7 (3) | C3′—C4′—C7′—C8′B | 154.4 (12) |
| C7—C8—C9—C11 | −62.1 (3) | C5′—C4′—C7′—C8′B | −25.7 (12) |
| C6—N1—C10—C9 | 6.6 (3) | C3′—C4′—C7′—C8′A | 101.1 (12) |
| C2—N1—C10—C9 | −176.56 (19) | C5′—C4′—C7′—C8′A | −78.9 (12) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C5—H5*A*···O1$^{i}$ | 0.93 | 2.34 | 3.116 (3) | 141 |
| C7—H7*A*···O2$^{ii}$ | 0.98 | 2.44 | 3.254 (3) | 140 |

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

**(1*R*,5*R*)-3-[(4-chlorophenyl)sulfonyl]-1,2,3,4,5,6-hexahydro-8*H*-1,5-methanopyrido[1,2-*a*][1,5]diazocin-8-one (II)**

*Crystal data*

$C_{17}H_{17}ClN_2O_3S$
$M_r$ = 364.83
Orthorhombic, $P2_12_12_1$
*a* = 7.1374 (14) Å
*b* = 11.448 (2) Å
*c* = 20.844 (4) Å
*V* = 1703.2 (6) Å$^3$
*Z* = 4
*F*(000) = 760
$D_x$ = 1.423 Mg m$^{-3}$
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 12750 reflections
*θ* = 3.9–71.2°
*μ* = 3.29 mm$^{-1}$
*T* = 299 K
Prizmatic, colorless
0.30 × 0.20 × 0.15 mm

*Data collection*

XtaLAB Synergy, Single source at home/near, HyPix3000 diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Detector resolution: 10.0000 pixels mm$^{-1}$
*ω* scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
$T_{min}$ = 0.795, $T_{max}$ = 1.000
16028 measured reflections
3289 independent reflections

3203 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 71.4°$, $\theta_{min} = 4.2°$

$h = -8 \rightarrow 8$
$k = -14 \rightarrow 13$
$l = -25 \rightarrow 25$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.078$
$S = 1.05$
3289 reflections
217 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 0.1799P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.42$ e Å$^{-3}$
$\Delta\rho_{min} = -0.33$ e Å$^{-3}$
Absolute structure: Flack *x* determined using 1318 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −0.004 (5)

*Special details*

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

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.22478 (8) | 0.27207 (5) | 0.34739 (3) | 0.03902 (15) |
| Cl1 | −0.32153 (13) | 0.49086 (7) | 0.54785 (3) | 0.0699 (2) |
| O1 | 0.7771 (3) | 0.28000 (19) | 0.15317 (11) | 0.0647 (5) |
| O2 | 0.1269 (3) | 0.18068 (15) | 0.31475 (8) | 0.0502 (4) |
| O3 | 0.3999 (3) | 0.24823 (16) | 0.37824 (9) | 0.0524 (4) |
| N1 | 0.4944 (3) | 0.36786 (17) | 0.16841 (9) | 0.0409 (4) |
| N12 | 0.2648 (3) | 0.37419 (16) | 0.29491 (8) | 0.0401 (4) |
| C2 | 0.6102 (4) | 0.2880 (2) | 0.13668 (12) | 0.0488 (6) |
| C3 | 0.5244 (5) | 0.2229 (3) | 0.08706 (15) | 0.0627 (7) |
| H3A | 0.596685 | 0.171265 | 0.063104 | 0.075* |
| C4 | 0.3400 (5) | 0.2340 (3) | 0.07356 (14) | 0.0655 (8) |
| H4A | 0.287153 | 0.189194 | 0.041014 | 0.079* |
| C5 | 0.2283 (4) | 0.3117 (3) | 0.10791 (13) | 0.0562 (6) |
| H5A | 0.100971 | 0.317377 | 0.098975 | 0.067* |
| C6 | 0.3060 (3) | 0.3793 (2) | 0.15466 (11) | 0.0423 (5) |
| C7 | 0.1922 (3) | 0.4659 (2) | 0.19194 (12) | 0.0451 (5) |
| H7A | 0.088762 | 0.492844 | 0.164794 | 0.054* |
| C8 | 0.3121 (4) | 0.5714 (2) | 0.21021 (14) | 0.0529 (6) |
| H8A | 0.363572 | 0.607885 | 0.172078 | 0.064* |
| H8B | 0.237218 | 0.628713 | 0.232979 | 0.064* |
| C9 | 0.4687 (4) | 0.5263 (2) | 0.25296 (13) | 0.0476 (5) |
| H9A | 0.546300 | 0.592999 | 0.265695 | 0.057* |
| C10 | 0.5921 (3) | 0.4407 (2) | 0.21691 (13) | 0.0471 (6) |
| H10A | 0.690567 | 0.484286 | 0.195534 | 0.057* |
| H10B | 0.651477 | 0.389329 | 0.247811 | 0.057* |

| | | | | |
|---|---|---|---|---|
| C11 | 0.3847 (4) | 0.4731 (2) | 0.31352 (12) | 0.0480 (6) |
| H11A | 0.483789 | 0.446415 | 0.341787 | 0.058* |
| H11B | 0.311175 | 0.531235 | 0.336171 | 0.058* |
| C13 | 0.1095 (3) | 0.4089 (2) | 0.25235 (12) | 0.0451 (5) |
| H13A | 0.027946 | 0.463659 | 0.274269 | 0.054* |
| H13B | 0.036134 | 0.340816 | 0.240581 | 0.054* |
| C1′ | 0.0721 (3) | 0.3307 (2) | 0.40561 (11) | 0.0395 (5) |
| C2′ | 0.1424 (4) | 0.4029 (3) | 0.45401 (13) | 0.0536 (6) |
| H2′A | 0.270287 | 0.417502 | 0.456776 | 0.064* |
| C3′ | 0.0214 (5) | 0.4522 (3) | 0.49743 (13) | 0.0607 (7) |
| H3′A | 0.066574 | 0.501348 | 0.529405 | 0.073* |
| C4′ | −0.1678 (4) | 0.4284 (2) | 0.49336 (11) | 0.0472 (6) |
| C5′ | −0.2378 (4) | 0.3554 (3) | 0.44671 (12) | 0.0549 (6) |
| H5′A | −0.365241 | 0.338706 | 0.445122 | 0.066* |
| C6′ | −0.1173 (4) | 0.3073 (2) | 0.40242 (12) | 0.0496 (6) |
| H6′A | −0.163668 | 0.259030 | 0.370264 | 0.060* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0399 (3) | 0.0367 (3) | 0.0404 (3) | −0.0018 (2) | 0.0007 (2) | −0.0021 (2) |
| Cl1 | 0.0922 (6) | 0.0660 (4) | 0.0514 (4) | 0.0183 (4) | 0.0234 (4) | −0.0014 (3) |
| O1 | 0.0432 (9) | 0.0705 (12) | 0.0805 (13) | 0.0155 (9) | 0.0077 (9) | 0.0098 (11) |
| O2 | 0.0568 (10) | 0.0399 (8) | 0.0539 (9) | −0.0081 (7) | 0.0038 (8) | −0.0110 (8) |
| O3 | 0.0469 (9) | 0.0530 (10) | 0.0572 (10) | 0.0070 (8) | −0.0037 (8) | 0.0050 (8) |
| N1 | 0.0363 (9) | 0.0433 (10) | 0.0432 (10) | 0.0015 (8) | 0.0017 (7) | 0.0079 (8) |
| N12 | 0.0380 (9) | 0.0428 (9) | 0.0395 (9) | −0.0068 (8) | −0.0036 (8) | 0.0006 (8) |
| C2 | 0.0471 (13) | 0.0464 (13) | 0.0530 (14) | 0.0065 (10) | 0.0087 (11) | 0.0103 (10) |
| C3 | 0.0742 (19) | 0.0554 (15) | 0.0586 (15) | 0.0143 (15) | 0.0122 (14) | −0.0012 (14) |
| C4 | 0.077 (2) | 0.0630 (17) | 0.0562 (15) | 0.0023 (16) | −0.0086 (14) | −0.0106 (14) |
| C5 | 0.0486 (14) | 0.0666 (16) | 0.0533 (14) | 0.0015 (12) | −0.0082 (12) | −0.0014 (12) |
| C6 | 0.0393 (11) | 0.0476 (12) | 0.0398 (11) | 0.0024 (9) | −0.0019 (9) | 0.0099 (10) |
| C7 | 0.0398 (12) | 0.0509 (13) | 0.0446 (12) | 0.0077 (10) | −0.0028 (9) | 0.0080 (10) |
| C8 | 0.0580 (15) | 0.0406 (12) | 0.0602 (15) | 0.0069 (11) | 0.0042 (12) | 0.0103 (11) |
| C9 | 0.0495 (13) | 0.0387 (12) | 0.0547 (13) | −0.0094 (10) | −0.0014 (12) | 0.0027 (10) |
| C10 | 0.0369 (12) | 0.0502 (13) | 0.0543 (14) | −0.0047 (10) | −0.0028 (10) | 0.0054 (11) |
| C11 | 0.0506 (13) | 0.0461 (12) | 0.0472 (12) | −0.0113 (11) | −0.0041 (11) | −0.0037 (10) |
| C13 | 0.0335 (10) | 0.0559 (14) | 0.0458 (12) | 0.0017 (10) | −0.0003 (9) | 0.0004 (10) |
| C1′ | 0.0424 (11) | 0.0396 (11) | 0.0365 (10) | −0.0049 (9) | 0.0009 (9) | −0.0017 (9) |
| C2′ | 0.0494 (14) | 0.0638 (15) | 0.0477 (13) | −0.0126 (12) | −0.0036 (12) | −0.0117 (12) |
| C3′ | 0.0720 (18) | 0.0653 (17) | 0.0447 (14) | −0.0100 (15) | −0.0002 (13) | −0.0196 (13) |
| C4′ | 0.0609 (15) | 0.0447 (12) | 0.0360 (11) | 0.0056 (11) | 0.0060 (10) | 0.0023 (10) |
| C5′ | 0.0441 (13) | 0.0681 (16) | 0.0527 (13) | −0.0021 (12) | 0.0034 (11) | −0.0091 (12) |
| C6′ | 0.0454 (13) | 0.0590 (15) | 0.0444 (12) | −0.0114 (11) | −0.0008 (11) | −0.0125 (11) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—O2 | 1.4304 (17) | C8—C9 | 1.520 (4) |
| S1—O3 | 1.4319 (18) | C8—H8A | 0.9700 |
| S1—N12 | 1.6262 (19) | C8—H8B | 0.9700 |
| S1—C1′ | 1.764 (2) | C9—C10 | 1.517 (4) |
| Cl1—C4′ | 1.733 (3) | C9—C11 | 1.524 (4) |
| O1—C2 | 1.243 (3) | C9—H9A | 0.9800 |
| N1—C6 | 1.381 (3) | C10—H10A | 0.9700 |
| N1—C2 | 1.399 (3) | C10—H10B | 0.9700 |
| N1—C10 | 1.485 (3) | C11—H11A | 0.9700 |
| N12—C11 | 1.471 (3) | C11—H11B | 0.9700 |
| N12—C13 | 1.474 (3) | C13—H13A | 0.9700 |
| C2—C3 | 1.414 (4) | C13—H13B | 0.9700 |
| C3—C4 | 1.352 (5) | C1′—C6′ | 1.379 (3) |
| C3—H3A | 0.9300 | C1′—C2′ | 1.398 (3) |
| C4—C5 | 1.393 (4) | C2′—C3′ | 1.372 (4) |
| C4—H4A | 0.9300 | C2′—H2′A | 0.9300 |
| C5—C6 | 1.363 (4) | C3′—C4′ | 1.380 (4) |
| C5—H5A | 0.9300 | C3′—H3′A | 0.9300 |
| C6—C7 | 1.499 (3) | C4′—C5′ | 1.376 (4) |
| C7—C8 | 1.528 (4) | C5′—C6′ | 1.377 (4) |
| C7—C13 | 1.537 (3) | C5′—H5′A | 0.9300 |
| C7—H7A | 0.9800 | C6′—H6′A | 0.9300 |
| | | | |
| O2—S1—O3 | 120.05 (11) | C8—C9—C11 | 109.4 (2) |
| O2—S1—N12 | 106.96 (10) | C10—C9—H9A | 108.0 |
| O3—S1—N12 | 106.62 (11) | C8—C9—H9A | 108.0 |
| O2—S1—C1′ | 107.67 (11) | C11—C9—H9A | 108.0 |
| O3—S1—C1′ | 107.63 (11) | N1—C10—C9 | 115.3 (2) |
| N12—S1—C1′ | 107.33 (11) | N1—C10—H10A | 108.4 |
| C6—N1—C2 | 122.6 (2) | C9—C10—H10A | 108.4 |
| C6—N1—C10 | 123.0 (2) | N1—C10—H10B | 108.4 |
| C2—N1—C10 | 114.32 (19) | C9—C10—H10B | 108.4 |
| C11—N12—C13 | 112.9 (2) | H10A—C10—H10B | 107.5 |
| C11—N12—S1 | 118.55 (15) | N12—C11—C9 | 108.5 (2) |
| C13—N12—S1 | 117.80 (16) | N12—C11—H11A | 110.0 |
| O1—C2—N1 | 118.9 (3) | C9—C11—H11A | 110.0 |
| O1—C2—C3 | 125.4 (3) | N12—C11—H11B | 110.0 |
| N1—C2—C3 | 115.7 (2) | C9—C11—H11B | 110.0 |
| C4—C3—C2 | 121.6 (3) | H11A—C11—H11B | 108.4 |
| C4—C3—H3A | 119.2 | N12—C13—C7 | 108.57 (19) |
| C2—C3—H3A | 119.2 | N12—C13—H13A | 110.0 |
| C3—C4—C5 | 120.7 (3) | C7—C13—H13A | 110.0 |
| C3—C4—H4A | 119.7 | N12—C13—H13B | 110.0 |
| C5—C4—H4A | 119.7 | C7—C13—H13B | 110.0 |
| C6—C5—C4 | 119.9 (3) | H13A—C13—H13B | 108.4 |
| C6—C5—H5A | 120.1 | C6′—C1′—C2′ | 120.1 (2) |

| | | | |
|---|---|---|---|
| C4—C5—H5A | 120.1 | C6′—C1′—S1 | 119.92 (18) |
| C5—C6—N1 | 119.4 (2) | C2′—C1′—S1 | 120.00 (19) |
| C5—C6—C7 | 121.7 (2) | C3′—C2′—C1′ | 119.6 (2) |
| N1—C6—C7 | 118.9 (2) | C3′—C2′—H2′A | 120.2 |
| C6—C7—C8 | 110.4 (2) | C1′—C2′—H2′A | 120.2 |
| C6—C7—C13 | 110.61 (19) | C2′—C3′—C4′ | 119.6 (2) |
| C8—C7—C13 | 110.3 (2) | C2′—C3′—H3′A | 120.2 |
| C6—C7—H7A | 108.5 | C4′—C3′—H3′A | 120.2 |
| C8—C7—H7A | 108.5 | C5′—C4′—C3′ | 121.2 (2) |
| C13—C7—H7A | 108.5 | C5′—C4′—Cl1 | 118.9 (2) |
| C9—C8—C7 | 106.80 (19) | C3′—C4′—Cl1 | 119.8 (2) |
| C9—C8—H8A | 110.4 | C4′—C5′—C6′ | 119.3 (2) |
| C7—C8—H8A | 110.4 | C4′—C5′—H5′A | 120.3 |
| C9—C8—H8B | 110.4 | C6′—C5′—H5′A | 120.3 |
| C7—C8—H8B | 110.4 | C5′—C6′—C1′ | 120.2 (2) |
| H8A—C8—H8B | 108.6 | C5′—C6′—H6′A | 119.9 |
| C10—C9—C8 | 110.9 (2) | C1′—C6′—H6′A | 119.9 |
| C10—C9—C11 | 112.4 (2) | | |

| | | | |
|---|---|---|---|
| O2—S1—N12—C11 | −172.55 (18) | C6—N1—C10—C9 | 2.5 (3) |
| O3—S1—N12—C11 | −43.0 (2) | C2—N1—C10—C9 | −178.8 (2) |
| C1′—S1—N12—C11 | 72.1 (2) | C8—C9—C10—N1 | −33.3 (3) |
| O2—S1—N12—C13 | 45.5 (2) | C11—C9—C10—N1 | 89.4 (3) |
| O3—S1—N12—C13 | 175.12 (17) | C13—N12—C11—C9 | −60.2 (3) |
| C1′—S1—N12—C13 | −69.78 (19) | S1—N12—C11—C9 | 156.07 (18) |
| C6—N1—C2—O1 | −177.7 (2) | C10—C9—C11—N12 | −62.0 (3) |
| C10—N1—C2—O1 | 3.6 (3) | C8—C9—C11—N12 | 61.6 (3) |
| C6—N1—C2—C3 | 3.0 (3) | C11—N12—C13—C7 | 58.3 (3) |
| C10—N1—C2—C3 | −175.7 (2) | S1—N12—C13—C7 | −157.68 (17) |
| O1—C2—C3—C4 | 177.7 (3) | C6—C7—C13—N12 | 64.2 (3) |
| N1—C2—C3—C4 | −3.0 (4) | C8—C7—C13—N12 | −58.2 (3) |
| C2—C3—C4—C5 | 0.9 (5) | O2—S1—C1′—C6′ | −15.8 (3) |
| C3—C4—C5—C6 | 1.5 (5) | O3—S1—C1′—C6′ | −146.5 (2) |
| C4—C5—C6—N1 | −1.5 (4) | N12—S1—C1′—C6′ | 99.0 (2) |
| C4—C5—C6—C7 | 178.7 (3) | O2—S1—C1′—C2′ | 165.7 (2) |
| C2—N1—C6—C5 | −0.8 (3) | O3—S1—C1′—C2′ | 35.0 (2) |
| C10—N1—C6—C5 | 177.8 (2) | N12—S1—C1′—C2′ | −79.4 (2) |
| C2—N1—C6—C7 | 179.0 (2) | C6′—C1′—C2′—C3′ | −1.3 (4) |
| C10—N1—C6—C7 | −2.4 (3) | S1—C1′—C2′—C3′ | 177.2 (2) |
| C5—C6—C7—C8 | −147.4 (2) | C1′—C2′—C3′—C4′ | 1.0 (4) |
| N1—C6—C7—C8 | 32.8 (3) | C2′—C3′—C4′—C5′ | 0.4 (5) |
| C5—C6—C7—C13 | 90.2 (3) | C2′—C3′—C4′—Cl1 | −179.7 (2) |
| N1—C6—C7—C13 | −89.6 (3) | C3′—C4′—C5′—C6′ | −1.5 (4) |
| C6—C7—C8—C9 | −62.1 (3) | Cl1—C4′—C5′—C6′ | 178.7 (2) |
| C13—C7—C8—C9 | 60.5 (3) | C4′—C5′—C6′—C1′ | 1.2 (4) |
| C7—C8—C9—C10 | 62.6 (3) | C2′—C1′—C6′—C5′ | 0.2 (4) |
| C7—C8—C9—C11 | −61.9 (3) | S1—C1′—C6′—C5′ | −178.2 (2) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C5—H5*A*···O1$^{i}$ | 0.93 | 2.61 | 3.375 (4) | 140 |
| C13—H13*B*···O1$^{i}$ | 0.97 | 2.69 | 3.475 (3) | 139 |
| C5′—H5′*A*···O3$^{i}$ | 0.93 | 2.42 | 3.198 (3) | 142 |
| C8—H8*A*···O3$^{ii}$ | 0.97 | 2.56 | 3.424 (3) | 149 |
| C4—H4*A*···Cl1$^{iii}$ | 0.93 | 2.94 | 3.764 (3) | 148 |

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

## (1*R*,5*R*)-3-((3-nitrophenyl)sulfonyl)-1,2,3,4,5,6-hexahydro-8*H*-1,5-methanopyrido[1,2-*a*][1,5]diazocin-8-one (III)

*Crystal data*

$C_{17}H_{17}N_3O_5S$
$M_r$ = 375.39
Monoclinic, $P2_1$
$a$ = 11.040 (2) Å
$b$ = 6.2621 (13) Å
$c$ = 12.424 (3) Å
$\beta$ = 94.03 (3)°
$V$ = 856.8 (3) Å$^3$
$Z$ = 2
$F(000)$ = 392
$D_x$ = 1.455 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54184 Å
Cell parameters from 5501 reflections
$\theta$ = 4.0–71.4°
$\mu$ = 2.00 mm$^{-1}$
$T$ = 296 K
Prizmatic, colorless
0.20 × 0.15 × 0.10 mm

*Data collection*

XtaLAB Synergy, Single source at home/near, HyPix3000 diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Detector resolution: 10.0000 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
$T_{min}$ = 0.639, $T_{max}$ = 1.000
7776 measured reflections
2446 independent reflections
2363 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.023
$\theta_{max}$ = 71.5°, $\theta_{min}$ = 3.6°
$h$ = −13→13
$k$ = −6→7
$l$ = −15→15

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.114
$S$ = 1.04
2446 reflections
235 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0754P)^2 + 0.1248P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.47 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.22 e Å$^{-3}$
Absolute structure: Flack $x$ determined using 579 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.017 (16)

*Special details*

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

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.15419 (6) | 0.10657 (14) | 0.65911 (6) | 0.0503 (2) |
| O1 | 0.6728 (3) | −0.1378 (5) | 0.7756 (3) | 0.0777 (8) |
| O2 | 0.1535 (2) | 0.1620 (5) | 0.54790 (18) | 0.0696 (9) |
| O3 | 0.1536 (3) | −0.1126 (4) | 0.6914 (3) | 0.0761 (8) |
| O4 | −0.1062 (4) | 0.0114 (12) | 0.9888 (4) | 0.143 (2) |
| O5 | −0.2278 (4) | 0.2805 (12) | 0.9936 (3) | 0.140 (2) |
| N1 | 0.5589 (2) | 0.1629 (4) | 0.7462 (2) | 0.0446 (6) |
| N12 | 0.2750 (2) | 0.2153 (4) | 0.71985 (18) | 0.0421 (5) |
| N3 | −0.1493 (3) | 0.1778 (10) | 0.9537 (3) | 0.0916 (16) |
| C2 | 0.6474 (3) | 0.0176 (6) | 0.7157 (3) | 0.0557 (8) |
| C3 | 0.7019 (3) | 0.0648 (7) | 0.6191 (3) | 0.0639 (11) |
| H3A | 0.759057 | −0.028879 | 0.594281 | 0.077* |
| C4 | 0.6726 (3) | 0.2440 (8) | 0.5615 (3) | 0.0638 (10) |
| H4A | 0.710971 | 0.272896 | 0.498892 | 0.077* |
| C5 | 0.5843 (3) | 0.3867 (6) | 0.5960 (3) | 0.0553 (8) |
| H5A | 0.564335 | 0.509117 | 0.556234 | 0.066* |
| C6 | 0.5290 (3) | 0.3440 (5) | 0.6875 (2) | 0.0436 (6) |
| C7 | 0.4346 (3) | 0.4905 (5) | 0.7275 (3) | 0.0492 (7) |
| H7A | 0.451784 | 0.635774 | 0.703421 | 0.059* |
| C8 | 0.4401 (3) | 0.4899 (7) | 0.8499 (3) | 0.0580 (9) |
| H8A | 0.520190 | 0.532007 | 0.879470 | 0.070* |
| H8B | 0.380876 | 0.588526 | 0.875750 | 0.070* |
| C9 | 0.4120 (3) | 0.2638 (7) | 0.8834 (2) | 0.0526 (8) |
| H9A | 0.413307 | 0.261073 | 0.962380 | 0.063* |
| C10 | 0.5094 (3) | 0.1127 (8) | 0.8501 (2) | 0.0539 (7) |
| H10A | 0.575597 | 0.113061 | 0.905801 | 0.065* |
| H10B | 0.476096 | −0.030658 | 0.846320 | 0.065* |
| C11 | 0.2850 (3) | 0.1977 (6) | 0.8394 (2) | 0.0514 (8) |
| H11A | 0.269259 | 0.051691 | 0.860401 | 0.062* |
| H11B | 0.225028 | 0.289081 | 0.869484 | 0.062* |
| C13 | 0.3064 (3) | 0.4300 (6) | 0.6827 (3) | 0.0495 (7) |
| H13A | 0.248524 | 0.533033 | 0.706739 | 0.059* |
| H13B | 0.302369 | 0.432878 | 0.604459 | 0.059* |
| C1′ | 0.0293 (3) | 0.2314 (5) | 0.7149 (2) | 0.0449 (6) |
| C2′ | −0.0143 (3) | 0.1467 (6) | 0.8071 (2) | 0.0517 (8) |
| H2′A | 0.014481 | 0.017681 | 0.835735 | 0.062* |
| C3′ | −0.1031 (3) | 0.2624 (8) | 0.8550 (3) | 0.0606 (10) |
| C4′ | −0.1479 (3) | 0.4520 (8) | 0.8136 (4) | 0.0693 (11) |
| H4′A | −0.207272 | 0.525883 | 0.847895 | 0.083* |
| C5′ | −0.1040 (3) | 0.5310 (7) | 0.7212 (4) | 0.0696 (10) |
| H5′A | −0.135141 | 0.657808 | 0.691690 | 0.084* |
| C6′ | −0.0138 (3) | 0.4237 (6) | 0.6715 (3) | 0.0558 (8) |
| H6′A | 0.017549 | 0.479393 | 0.609888 | 0.067* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0495 (4) | 0.0415 (4) | 0.0608 (4) | 0.0046 (3) | 0.0094 (3) | −0.0112 (4) |
| O1 | 0.0648 (16) | 0.0635 (19) | 0.105 (2) | 0.0234 (14) | 0.0084 (14) | 0.0117 (17) |
| O2 | 0.0688 (14) | 0.090 (3) | 0.0508 (11) | −0.0015 (14) | 0.0079 (10) | −0.0178 (13) |
| O3 | 0.0725 (17) | 0.0361 (14) | 0.121 (2) | 0.0036 (12) | 0.0151 (16) | −0.0143 (15) |
| O4 | 0.109 (3) | 0.205 (6) | 0.120 (3) | 0.006 (4) | 0.038 (2) | 0.085 (4) |
| O5 | 0.123 (3) | 0.186 (6) | 0.120 (3) | −0.016 (4) | 0.071 (3) | −0.029 (4) |
| N1 | 0.0405 (11) | 0.0402 (15) | 0.0533 (12) | 0.0025 (10) | 0.0038 (9) | −0.0029 (11) |
| N12 | 0.0437 (12) | 0.0376 (14) | 0.0458 (11) | 0.0058 (11) | 0.0079 (9) | 0.0014 (10) |
| N3 | 0.060 (2) | 0.148 (5) | 0.069 (2) | −0.013 (2) | 0.0244 (16) | 0.009 (3) |
| C2 | 0.0444 (15) | 0.0499 (19) | 0.072 (2) | 0.0058 (15) | 0.0022 (14) | −0.0110 (18) |
| C3 | 0.0494 (16) | 0.069 (3) | 0.074 (2) | 0.0040 (17) | 0.0143 (15) | −0.022 (2) |
| C4 | 0.060 (2) | 0.072 (3) | 0.0609 (18) | −0.0118 (19) | 0.0194 (16) | −0.0169 (19) |
| C5 | 0.0612 (19) | 0.056 (2) | 0.0495 (16) | −0.0082 (16) | 0.0095 (13) | −0.0003 (15) |
| C6 | 0.0436 (14) | 0.0395 (16) | 0.0477 (14) | −0.0042 (12) | 0.0023 (11) | −0.0048 (13) |
| C7 | 0.0570 (16) | 0.0318 (16) | 0.0595 (17) | 0.0019 (13) | 0.0089 (13) | −0.0002 (13) |
| C8 | 0.0631 (18) | 0.054 (2) | 0.0570 (17) | 0.0011 (17) | 0.0084 (14) | −0.0178 (16) |
| C9 | 0.0546 (17) | 0.062 (2) | 0.0415 (13) | 0.0025 (16) | 0.0069 (12) | −0.0016 (15) |
| C10 | 0.0491 (14) | 0.0588 (19) | 0.0539 (15) | 0.0031 (18) | 0.0039 (12) | 0.0138 (18) |
| C11 | 0.0500 (15) | 0.058 (2) | 0.0474 (14) | 0.0080 (15) | 0.0120 (12) | 0.0053 (15) |
| C13 | 0.0505 (16) | 0.0400 (17) | 0.0578 (16) | 0.0086 (14) | 0.0039 (13) | 0.0096 (15) |
| C1′ | 0.0380 (13) | 0.0422 (17) | 0.0542 (15) | 0.0021 (12) | 0.0018 (11) | −0.0016 (14) |
| C2′ | 0.0394 (13) | 0.055 (2) | 0.0613 (16) | −0.0045 (14) | 0.0056 (12) | 0.0088 (16) |
| C3′ | 0.0426 (15) | 0.083 (3) | 0.0572 (17) | −0.0108 (17) | 0.0072 (13) | −0.0053 (19) |
| C4′ | 0.0446 (16) | 0.076 (3) | 0.089 (3) | 0.0053 (18) | 0.0116 (16) | −0.024 (2) |
| C5′ | 0.0525 (17) | 0.056 (2) | 0.100 (3) | 0.0152 (17) | 0.0041 (18) | 0.004 (2) |
| C6′ | 0.0490 (16) | 0.051 (2) | 0.0682 (19) | 0.0059 (15) | 0.0058 (14) | 0.0066 (17) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—O2 | 1.424 (3) | C7—H7A | 0.9800 |
| S1—O3 | 1.430 (3) | C8—C9 | 1.514 (6) |
| S1—N12 | 1.634 (3) | C8—H8A | 0.9700 |
| S1—C1′ | 1.768 (3) | C8—H8B | 0.9700 |
| O1—C2 | 1.245 (5) | C9—C10 | 1.512 (5) |
| O4—N3 | 1.214 (8) | C9—C11 | 1.526 (5) |
| O5—N3 | 1.212 (7) | C9—H9A | 0.9800 |
| N1—C6 | 1.376 (4) | C10—H10A | 0.9700 |
| N1—C2 | 1.407 (4) | C10—H10B | 0.9700 |
| N1—C10 | 1.471 (4) | C11—H11A | 0.9700 |
| N12—C13 | 1.471 (4) | C11—H11B | 0.9700 |
| N12—C11 | 1.485 (4) | C13—H13A | 0.9700 |
| N3—C3′ | 1.460 (5) | C13—H13B | 0.9700 |
| C2—C3 | 1.411 (5) | C1′—C2′ | 1.379 (4) |
| C3—C4 | 1.357 (6) | C1′—C6′ | 1.390 (5) |
| C3—H3A | 0.9300 | C2′—C3′ | 1.386 (5) |

| | | | |
|---|---|---|---|
| C4—C5 | 1.411 (6) | C2′—H2′A | 0.9300 |
| C4—H4A | 0.9300 | C3′—C4′ | 1.372 (7) |
| C5—C6 | 1.354 (4) | C4′—C5′ | 1.370 (6) |
| C5—H5A | 0.9300 | C4′—H4′A | 0.9300 |
| C6—C7 | 1.499 (4) | C5′—C6′ | 1.383 (5) |
| C7—C8 | 1.519 (4) | C5′—H5′A | 0.9300 |
| C7—C13 | 1.531 (5) | C6′—H6′A | 0.9300 |

| | | | |
|---|---|---|---|
| O2—S1—O3 | 120.4 (2) | C10—C9—C8 | 110.3 (3) |
| O2—S1—N12 | 107.14 (14) | C10—C9—C11 | 112.7 (3) |
| O3—S1—N12 | 106.87 (16) | C8—C9—C11 | 110.8 (3) |
| O2—S1—C1′ | 108.78 (16) | C10—C9—H9A | 107.6 |
| O3—S1—C1′ | 107.17 (17) | C8—C9—H9A | 107.6 |
| N12—S1—C1′ | 105.56 (13) | C11—C9—H9A | 107.6 |
| C6—N1—C2 | 122.4 (3) | N1—C10—C9 | 114.9 (3) |
| C6—N1—C10 | 123.5 (3) | N1—C10—H10A | 108.5 |
| C2—N1—C10 | 114.0 (3) | C9—C10—H10A | 108.5 |
| C13—N12—C11 | 112.2 (3) | N1—C10—H10B | 108.5 |
| C13—N12—S1 | 116.0 (2) | C9—C10—H10B | 108.5 |
| C11—N12—S1 | 115.6 (2) | H10A—C10—H10B | 107.5 |
| O5—N3—O4 | 125.6 (5) | N12—C11—C9 | 109.9 (2) |
| O5—N3—C3′ | 116.9 (6) | N12—C11—H11A | 109.7 |
| O4—N3—C3′ | 117.5 (4) | C9—C11—H11A | 109.7 |
| O1—C2—N1 | 118.4 (3) | N12—C11—H11B | 109.7 |
| O1—C2—C3 | 125.5 (3) | C9—C11—H11B | 109.7 |
| N1—C2—C3 | 116.1 (3) | H11A—C11—H11B | 108.2 |
| C4—C3—C2 | 121.4 (3) | N12—C13—C7 | 110.2 (2) |
| C4—C3—H3A | 119.3 | N12—C13—H13A | 109.6 |
| C2—C3—H3A | 119.3 | C7—C13—H13A | 109.6 |
| C3—C4—C5 | 120.5 (3) | N12—C13—H13B | 109.6 |
| C3—C4—H4A | 119.7 | C7—C13—H13B | 109.6 |
| C5—C4—H4A | 119.7 | H13A—C13—H13B | 108.1 |
| C6—C5—C4 | 119.4 (4) | C2′—C1′—C6′ | 121.7 (3) |
| C6—C5—H5A | 120.3 | C2′—C1′—S1 | 118.9 (3) |
| C4—C5—H5A | 120.3 | C6′—C1′—S1 | 119.1 (2) |
| C5—C6—N1 | 120.1 (3) | C1′—C2′—C3′ | 117.0 (3) |
| C5—C6—C7 | 121.5 (3) | C1′—C2′—H2′A | 121.5 |
| N1—C6—C7 | 118.4 (3) | C3′—C2′—H2′A | 121.5 |
| C6—C7—C8 | 110.5 (3) | C4′—C3′—C2′ | 122.5 (3) |
| C6—C7—C13 | 112.0 (3) | C4′—C3′—N3 | 119.4 (4) |
| C8—C7—C13 | 109.5 (3) | C2′—C3′—N3 | 118.1 (4) |
| C6—C7—H7A | 108.2 | C5′—C4′—C3′ | 119.2 (3) |
| C8—C7—H7A | 108.2 | C5′—C4′—H4′A | 120.4 |
| C13—C7—H7A | 108.2 | C3′—C4′—H4′A | 120.4 |
| C9—C8—C7 | 106.5 (3) | C4′—C5′—C6′ | 120.5 (4) |
| C9—C8—H8A | 110.4 | C4′—C5′—H5′A | 119.8 |
| C7—C8—H8A | 110.4 | C6′—C5′—H5′A | 119.8 |
| C9—C8—H8B | 110.4 | C5′—C6′—C1′ | 119.1 (3) |

| | | | |
|---|---|---|---|
| C7—C8—H8B | 110.4 | C5′—C6′—H6′A | 120.5 |
| H8A—C8—H8B | 108.6 | C1′—C6′—H6′A | 120.5 |

| | | | |
|---|---|---|---|
| O2—S1—N12—C13 | 38.6 (2) | C8—C9—C10—N1 | −36.5 (4) |
| O3—S1—N12—C13 | 168.9 (2) | C11—C9—C10—N1 | 87.9 (4) |
| C1′—S1—N12—C13 | −77.3 (2) | C13—N12—C11—C9 | −55.3 (4) |
| O2—S1—N12—C11 | 173.1 (2) | S1—N12—C11—C9 | 168.5 (2) |
| O3—S1—N12—C11 | −56.6 (3) | C10—C9—C11—N12 | −65.9 (4) |
| C1′—S1—N12—C11 | 57.3 (3) | C8—C9—C11—N12 | 58.2 (4) |
| C6—N1—C2—O1 | 177.1 (3) | C11—N12—C13—C7 | 56.7 (3) |
| C10—N1—C2—O1 | 1.8 (5) | S1—N12—C13—C7 | −167.3 (2) |
| C6—N1—C2—C3 | −1.8 (4) | C6—C7—C13—N12 | 62.7 (3) |
| C10—N1—C2—C3 | −177.0 (3) | C8—C7—C13—N12 | −60.3 (4) |
| O1—C2—C3—C4 | −176.7 (4) | O2—S1—C1′—C2′ | 157.4 (3) |
| N1—C2—C3—C4 | 2.0 (5) | O3—S1—C1′—C2′ | 25.8 (3) |
| C2—C3—C4—C5 | −1.3 (6) | N12—S1—C1′—C2′ | −87.9 (3) |
| C3—C4—C5—C6 | 0.2 (5) | O2—S1—C1′—C6′ | −29.2 (3) |
| C4—C5—C6—N1 | 0.1 (5) | O3—S1—C1′—C6′ | −160.8 (3) |
| C4—C5—C6—C7 | −180.0 (3) | N12—S1—C1′—C6′ | 85.5 (3) |
| C2—N1—C6—C5 | 0.8 (4) | C6′—C1′—C2′—C3′ | −0.3 (5) |
| C10—N1—C6—C5 | 175.6 (3) | S1—C1′—C2′—C3′ | 173.0 (2) |
| C2—N1—C6—C7 | −179.2 (3) | C1′—C2′—C3′—C4′ | 0.7 (5) |
| C10—N1—C6—C7 | −4.4 (4) | C1′—C2′—C3′—N3 | −179.0 (3) |
| C5—C6—C7—C8 | −147.0 (3) | O5—N3—C3′—C4′ | 1.1 (6) |
| N1—C6—C7—C8 | 33.0 (4) | O4—N3—C3′—C4′ | −178.9 (5) |
| C5—C6—C7—C13 | 90.6 (4) | O5—N3—C3′—C2′ | −179.3 (4) |
| N1—C6—C7—C13 | −89.4 (3) | O4—N3—C3′—C2′ | 0.8 (6) |
| C6—C7—C8—C9 | −62.1 (3) | C2′—C3′—C4′—C5′ | 0.2 (6) |
| C13—C7—C8—C9 | 61.7 (4) | N3—C3′—C4′—C5′ | 179.8 (4) |
| C7—C8—C9—C10 | 64.2 (3) | C3′—C4′—C5′—C6′ | −1.4 (6) |
| C7—C8—C9—C11 | −61.3 (3) | C4′—C5′—C6′—C1′ | 1.8 (6) |
| C6—N1—C10—C9 | 6.1 (5) | C2′—C1′—C6′—C5′ | −0.9 (5) |
| C2—N1—C10—C9 | −178.7 (3) | S1—C1′—C6′—C5′ | −174.1 (3) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C4′—H4′*A*···O1$^{i}$ | 0.93 | 2.61 | 3.257 (5) | 127 |
| C10—H10*A*···O5$^{ii}$ | 0.97 | 2.58 | 3.460 (6) | 151 |
| C11—H11*A*···O5$^{iii}$ | 0.97 | 2.55 | 3.428 (7) | 150 |
| C13—H13*A*···O3$^{iv}$ | 0.97 | 2.46 | 3.330 (4) | 150 |

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