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Synthesis and crystal structure of *N*-(5-acetyl-4-methylpyrimidin-2-yl)benzenesulfonamide

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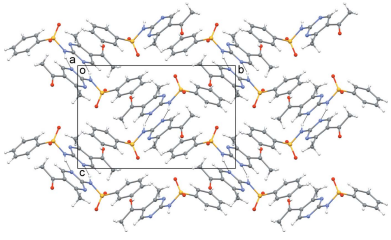
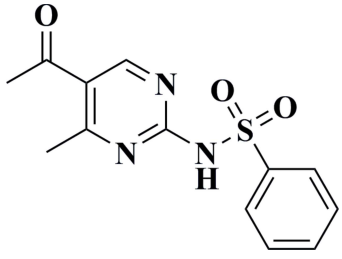
N-(5-Acetyl-4-methylpyrimidin-2-yl)benzenesulfonamide, $C_{13}H_{13}N_3O_3S$, was sythesized and characterized by single-crystal X-ray diffraction. In the crystal, π–π interactions between the phenyl and pyrimidine groups of neighbouring molecules form molecular chains parallel to [010]. Adjacent molecular chains are linked by N—H···N hydrogen-bonding interactions between the pyrimidine and amine groups of neighbouring molecules, resulting in a three-dimensional network.

1. Chemical context

Sulfonamide-bearing molecules with one or several pharmacological scaffolds constitute a class of drugs with antiviral, anticancer, anti-carbonic anhydrase (CA), diuretic, cyclooxigenase 2 (COX2) inhibitory, protease inhibitory, and/or antibacterial activities (Supuran, 2003; Scozzafava *et al.*, 2003; Casini & Scozzafava, 2002). It is noteworthy that the sulfonamide moiety is one of the significant, privileged building blocks that medicinal chemists frequently find in potent drugs (Elgemeie *et al.*, 2019). Thus, many widely marketed drugs incorporate this moiety. Several pyrimidine sulfonamides and other pyrimidine analogues that could be incorporated in new designs for bioactive molecules with medicinal applications have already been considered (Azzam, 2019; Azzam & Elgemeie, 2019; Azzam *et al.*, 2017, 2019; Mohamed-Ezzat *et al.*, 2021, 2022; Elgemeie *et al.*, 2015*a*,*b*, 2017). The synthesis of *N*-(5-acetyl-4-methylpyrimidin-2-yl)benzenesulfonamide (AMBS) was reported several decades ago (Gutsche *et al.*, 1964). In this article, we describe an alternative novel one-pot reaction methodology for the synthesis of this compound, which was also crystallized and crystallographically investigated.





2. Structural commentary

N-(5-Acetyl-4-methylpyrimidin-2-yl)benzenesulfonamide (AMBS) crystallizes in the monoclinic system, space group

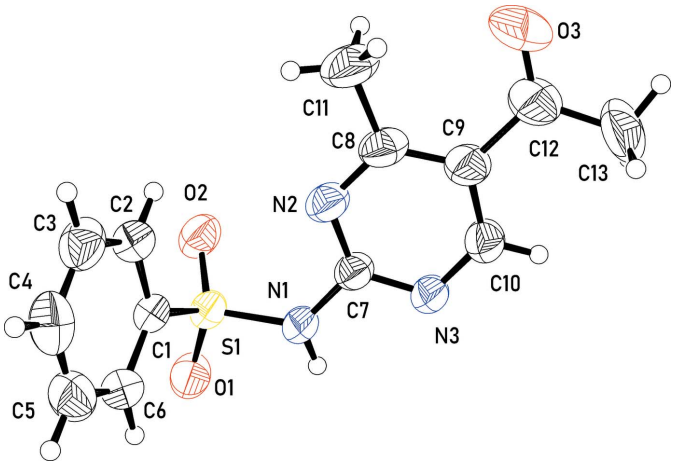


Figure 1
The molecular structure of the title compound with atom labels and 50% probability atomic displacement ellipsoids.

$P2_1/c$ and contains four molecules in the unit cell ($Z = 4$). The asymmetric unit is shown in Fig. 1. The acetaldehyde group of the molecule is disordered with two components related by a twist of 31.3 (1)° about the C_{ar}—C bond. Apart from a slight twist of the aldehyde group associated with the disorder, the 1-(2-amino-4-methylpyrimidin-5-yl)ethan-1-one segment of the molecule is essentially planar, the sulfonamide atom S1 being located only 0.423 (1) Å away from the plane of the pyrimidine group. The molecule exhibits a C7—N1—S1—C1 torsion angle of −79.0 (2)°, while the twist between the planes of the phenyl group and the pyrimidine ring comprises a dihedral angle of 63.07 (7)°.

3. Supramolecular features

The packing of AMBS is shown in Fig. 2. In the crystal, partial π–π overlap is observed between the phenyl group of one molecule and the pyrimidine group of an adjacent one related by 2_1 symmetry ($1-x, -\frac{1}{2}+y, \frac{1}{2}-z$ or $1-x, \frac{1}{2}+y, \frac{1}{2}-z$). The dihedral angle between the planes of the rings is 9.04 (10)°

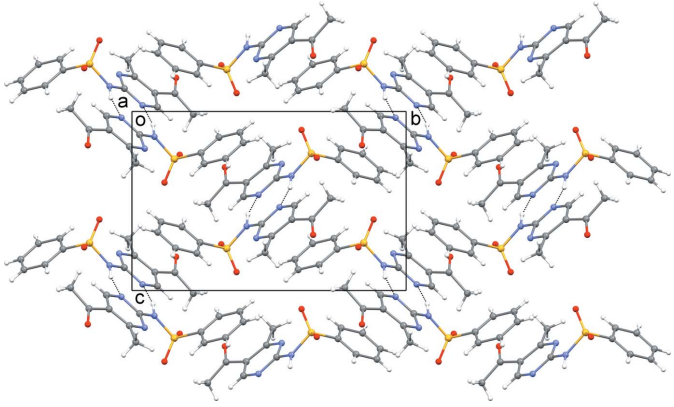


Figure 2
Crystal packing viewed down the *a* axis with N—H···N hydrogen bonds shown as dotted lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N3^{i}	0.83 (2)	2.06 (2)	2.891 (2)	179 (2)
C6—H6···O2^{ii}	0.93	2.62	3.338 (3)	135
C10—H10···O1^{i}	0.93	2.54	3.243 (3)	133
C11—H11*B*···O3*A*	0.96	2.26	2.769 (7)	113
C13*A*—H13*E*···O1^{iii}	0.96	2.50	3.40 (3)	156

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (iii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

with a ring centroid-to-centroid distance of 3.769 (1) Å (Fig. 3). The slippage distances between the overlapping rings are 1.44 Å ($1-x, -\frac{1}{2}+y, \frac{1}{2}-z$) and 1.58 Å ($1-x, \frac{1}{2}+y, \frac{1}{2}-z$). These π–π interactions form chains in the structure in which one AMBS molecule comprises the linker between two further molecules. The bent nature of the molecule results in a zigzag pattern of chains propagating parallel to [010].

The hydrogen-bonding interactions in the crystal are summarized in Table 1. Two linear N—H···N hydrogen bonds, with N···N distances of 2.891 (2) Å, occur between two neighbouring molecules related by inversion symmetry ($1-x, 1-y, 1-z$). A pair of hydrogen bonds is formed between the pyrimidine and amine groups of the two molecules, resulting in a $R_2^2(8)$ geometry (Fig. 2). The hydrogen bonds link the molecular chains formed by the π–π interactions and are perpendicular to the chains' protrusion. Additionally, non-classical hydrogen-bonding contacts of the C—H···O type with C···O distances in the range of *ca* 2.7–3.4 Å help to consolidate the structure.

4. Database survey

A survey of the Cambridge Structural Database (Groom *et al.*, 2016; accessed February 2023) using *CONQUEST* (Bruno *et al.*, 2002) for structures containing the *N*-(pyrimidin-2-yl)benzenesulfonamide group gave 164 hits, *i.e.* too many for them all to be analysed in detail.

An example of a closely related compound is 4,5,6-trimethyl-2-[(phenylsulfonyl)amino]pyrimidine (TPAP) (refcode VENKIJ; Li & Yang, 2006). In this structure, the dihedral angle between the planes through the phenyl and pyrimidine rings is 91.9°, larger than that observed for the title compound AMBS [63.07 (7)°]. In contrast to AMBS, π–π interactions are only observed between the pyrimidine rings in TPAP, resulting in stacking along the *a*-axis with interplanar distances of 3.81 Å.

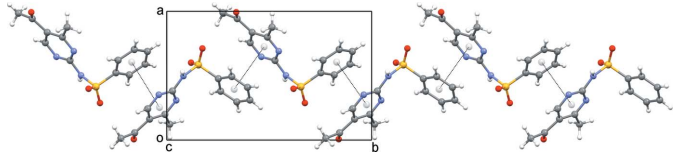


Figure 3
A segment of the crystal structure showing a chain of molecules linked by π–π interactions. The dotted lines connect the centroids of the molecules involved.

Another closely related compound is *N*-(pyrimidin-2-yl)benzenesulfonamide (PBS) (refcode XIFKAZ01; Coles *et al.*, 2000). In PBS, the dihedral angle between the planes through the phenyl and pyrimidine rings is 74.5°, again larger than for AMBS. Also unlike in AMBS, π–π interactions occur in PBS between pairs of molecules involving only the pyrimidine rings and with an interplanar distance of 3.5 Å. Similarly to AMBS, two linear N—H···N hydrogen bonds are observed in PBS between the pyrimidine and amine groups of neighbouring molecules, resulting in similar $R_2^2(8)$ motifs.

5. Synthesis and crystallization

Phenylsulfonyl guanidine **1** is a common starting material for the synthesis of several heterocyclic compounds and has been utilized effectively in the generation of a range of biologically active compounds. Our approach was based on synthesizing the substituted sulfonyl derivative **4** by reacting the sulfonyl guanidine **1** with triethylorthoformate **2** and acetyl acetone **3** (Fig. 4). The target product was identified by NMR spectroscopy and X-ray crystallography.

Synthesis of compound 4: Triethylorthoformate (5 ml) was added to a mixture of phenylsulfonyl guanidine (0.05 mol) and acetyl acetone (0.1 mol). The reaction mixture was then refluxed for 6 h. After cooling, the resulting precipitate was filtered and crystallized from ethanol.

Orange crystals; yield 45%; m.p. 469 K. ^{1}H NMR (400 MHz, DMSO-d_6): δ 2.49 (*s*, 3H, CH_3), 2.52 (*s*, 3H, CH_3), 7.57–7.66 (*m*, 3H, Ar-H), 8.00–8.02 (*m*, 2H, Ar-H), 8.93 (*s*, 1H, CH-pyrimidine), 12.34 (*s*, 1H, NH). Analysis calculated for $C_{13}H_{13}N_3O_3S$ (291.33): C, 53.60; H, 4.50; N, 14.42; S, 11.01. Found: C, 53.60; H, 4.49; N, 14.41; S, 11.00.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The N—H hydrogen was refined freely. The remaining hydrogen atoms were positioned geometrically and using a riding model [C—H = 0.93–0.96 Å with U_{iso}(H) = 1.2 or 1.5 U_{eq}(C). The acetaldehyde group of the molecule is disordered with two components related by a twist of 31.3 (1)° about the C_{ar}—C bond. In the refinement, the two components were restrained to have similar geometry (SAME in *SHELXL*) and atomic displacement parameters (SIMU and ISOR). The occupancies of the two components refined to 0.591 (11)/0.409 (11).

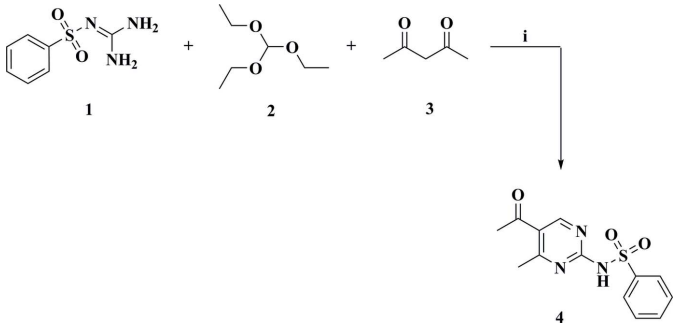


Figure 4
The synthesis of the title compound (**4**). Reagents & Conditions: (i) reflux; 6 h.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{13}N_3O_3S$
M_r	291.32
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, b, c (Å)	10.0699 (6), 14.7429 (6), 10.5212 (7)
β (°)	113.900 (7)
V (Å^3)	1428.04 (16)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.24
Crystal size (mm)	0.44 × 0.24 × 0.16
Data collection	
Diffractometer	Rigaku SuperNova, Dual, Cu at home/near, Atlas
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2022)
T_{min}, T_{max}	0.484, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12642, 3532, 2548
R_{int}	0.026
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.694
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.046, 0.125, 1.07
No. of reflections	3532
No. of parameters	216
No. of restraints	84
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.29

Computer programs: *CrysAlis PRO* (Rigaku OD, 2022), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018/1* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2020) and *ORTEP-3 for Windows* (Farrugia, 2012).

Funding information

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supporting information

Acta Cryst. (2023). E**79**, 331-334 [https://doi.org/10.1107/S2056989023001871]

Synthesis and crystal structure of *N*-(5-acetyl-4-methylpyrimidin-2-yl)benzenesulfonamide

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Computing details

Data collection: *CrysAlis PRO* 1.171.42.54a (Rigaku OD, 2022); cell refinement: *CrysAlis PRO* 1.171.42.54a (Rigaku OD, 2022); data reduction: *CrysAlis PRO* 1.171.42.54a (Rigaku OD, 2022); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018/1* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2020), *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *Mercury* (Macrae *et al.*, 2020).

N-(5-Acetyl-4-methylpyrimidin-2-yl)benzenesulfonamide

Crystal data

$C_{13}H_{13}N_3O_3S$
M_r = 291.32
Monoclinic, $P2_1/c$
a = 10.0699 (6) Å
b = 14.7429 (6) Å
c = 10.5212 (7) Å
β = 113.900 (7)°
V = 1428.04 (16) Å^3
Z = 4
$F(000)$ = 608
D_x = 1.355 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5035 reflections
θ = 3.9–27.7°
μ = 0.24 mm^{-1}
T = 293 K
Block, orange
0.44 × 0.24 × 0.16 mm

Data collection

Rigaku SuperNova, Dual, Cu at home/near, Atlas diffractometer
ω scans
Absorption correction: gaussian (CrysAlisPro; Rigaku OD, 2022)
T_{min} = 0.484, T_{max} = 1.000
12642 measured reflections
3532 independent reflections
2548 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 29.5°, θ_{min} = 3.5°
h = −13→10
k = −18→18
l = −13→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.125
S = 1.07
3532 reflections
216 parameters
84 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2 + 0.420P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.5229 (2)	0.75192 (12)	0.26887 (19)	0.0461 (4)	
C2	0.6205 (2)	0.76291 (14)	0.2086 (2)	0.0552 (5)	
H2	0.630570	0.718727	0.150041	0.066*	
C3	0.7033 (2)	0.84155 (15)	0.2374 (3)	0.0651 (6)	
H3	0.768869	0.850929	0.196825	0.078*	
C4	0.6886 (3)	0.90550 (15)	0.3257 (3)	0.0712 (7)	
H4	0.746248	0.957279	0.346473	0.085*	
C5	0.5901 (3)	0.89393 (15)	0.3833 (2)	0.0696 (6)	
H5	0.579890	0.938259	0.441639	0.083*	
C6	0.5065 (3)	0.81732 (14)	0.3555 (2)	0.0580 (5)	
H6	0.439261	0.809307	0.394462	0.070*	
C7	0.6186 (2)	0.52796 (12)	0.37144 (19)	0.0463 (4)	
C8	0.8002 (2)	0.49089 (16)	0.3045 (2)	0.0579 (5)	
C10	0.7564 (2)	0.40722 (14)	0.4752 (2)	0.0568 (5)	
H10	0.780068	0.359996	0.539203	0.068*	
C11	0.8767 (3)	0.5141 (2)	0.2123 (3)	0.0835 (8)	
H11A	0.853870	0.469409	0.140165	0.125*	
H11B	0.979663	0.514976	0.266457	0.125*	
H11C	0.845364	0.572710	0.171410	0.125*	
N1	0.50291 (19)	0.58192 (11)	0.35938 (18)	0.0508 (4)	
N2	0.69070 (18)	0.54638 (11)	0.29291 (17)	0.0537 (4)	
N3	0.64728 (17)	0.46152 (10)	0.46533 (16)	0.0496 (4)	
O1	0.28821 (16)	0.67414 (10)	0.25840 (16)	0.0653 (4)	
O2	0.39885 (17)	0.61859 (10)	0.10316 (14)	0.0621 (4)	
S1	0.41467 (5)	0.65419 (3)	0.23421 (5)	0.04929 (17)	
C9	0.8367 (2)	0.41647 (16)	0.3968 (2)	0.0600 (5)	0.591 (11)
C12	0.9420 (7)	0.3432 (4)	0.3990 (8)	0.0750 (19)	0.591 (11)
C13	0.9860 (17)	0.2756 (10)	0.5150 (18)	0.098 (4)	0.591 (11)
H13A	1.053800	0.233375	0.505298	0.148*	0.591 (11)
H13B	0.901665	0.243536	0.511702	0.148*	0.591 (11)
H13C	1.030623	0.306600	0.602434	0.148*	0.591 (11)
O3	0.9931 (8)	0.3397 (4)	0.3128 (8)	0.110 (2)	0.591 (11)
C9A	0.8367 (2)	0.41647 (16)	0.3968 (2)	0.0600 (5)	0.409 (11)
C12A	0.9727 (9)	0.3620 (7)	0.4306 (12)	0.079 (3)	0.409 (11)
C13A	0.990 (2)	0.2750 (12)	0.509 (3)	0.089 (4)	0.409 (11)
H13D	1.082486	0.248722	0.525215	0.133*	0.409 (11)
H13E	0.913988	0.233796	0.454891	0.133*	0.409 (11)
H13F	0.983594	0.286757	0.595831	0.133*	0.409 (11)
O3A	1.0616 (8)	0.3857 (7)	0.3876 (10)	0.106 (3)	0.409 (11)

H1	0.458 (2)	0.5700 (14)	0.409 (2)	0.054 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0557 (11)	0.0456 (9)	0.0403 (10)	0.0056 (8)	0.0228 (9)	0.0082 (7)
C2	0.0619 (13)	0.0559 (11)	0.0551 (12)	0.0062 (9)	0.0311 (10)	0.0070 (9)
C3	0.0567 (13)	0.0676 (14)	0.0760 (16)	0.0015 (10)	0.0320 (12)	0.0183 (11)
C4	0.0695 (15)	0.0511 (12)	0.0789 (17)	−0.0026 (10)	0.0157 (13)	0.0080 (11)
C5	0.0897 (18)	0.0535 (12)	0.0634 (14)	0.0051 (11)	0.0287 (13)	−0.0043 (10)
C6	0.0754 (15)	0.0552 (11)	0.0510 (12)	0.0089 (10)	0.0335 (11)	0.0040 (9)
C7	0.0469 (11)	0.0518 (10)	0.0433 (10)	0.0003 (8)	0.0215 (9)	0.0043 (7)
C8	0.0491 (12)	0.0804 (14)	0.0498 (12)	−0.0014 (10)	0.0258 (10)	0.0012 (10)
C10	0.0515 (12)	0.0632 (12)	0.0570 (12)	0.0109 (9)	0.0233 (10)	0.0133 (9)
C11	0.0691 (16)	0.125 (2)	0.0738 (17)	0.0090 (15)	0.0472 (14)	0.0168 (15)
N1	0.0595 (10)	0.0514 (9)	0.0521 (10)	0.0093 (7)	0.0336 (8)	0.0141 (7)
N2	0.0550 (10)	0.0635 (10)	0.0499 (10)	−0.0001 (8)	0.0289 (8)	0.0072 (7)
N3	0.0506 (9)	0.0558 (9)	0.0481 (9)	0.0081 (7)	0.0259 (8)	0.0112 (7)
O1	0.0542 (9)	0.0753 (9)	0.0733 (10)	0.0134 (7)	0.0331 (8)	0.0233 (8)
O2	0.0792 (11)	0.0595 (8)	0.0457 (8)	−0.0060 (7)	0.0234 (7)	0.0020 (6)
S1	0.0546 (3)	0.0506 (3)	0.0465 (3)	0.0042 (2)	0.0244 (2)	0.00978 (19)
C9	0.0466 (11)	0.0798 (14)	0.0577 (13)	0.0102 (10)	0.0254 (10)	0.0069 (10)
C12	0.045 (2)	0.101 (3)	0.084 (4)	0.011 (2)	0.030 (3)	0.006 (3)
C13	0.081 (6)	0.098 (5)	0.094 (6)	0.048 (5)	0.012 (5)	0.016 (5)
O3	0.096 (4)	0.128 (4)	0.144 (5)	0.037 (3)	0.087 (4)	0.017 (3)
C9A	0.0466 (11)	0.0798 (14)	0.0577 (13)	0.0102 (10)	0.0254 (10)	0.0069 (10)
C12A	0.054 (4)	0.105 (4)	0.077 (4)	0.025 (4)	0.025 (4)	0.001 (4)
C13A	0.072 (7)	0.107 (8)	0.086 (7)	0.025 (7)	0.032 (6)	−0.007 (7)
O3A	0.070 (4)	0.144 (6)	0.127 (6)	0.026 (4)	0.062 (4)	0.009 (4)

Geometric parameters (Å, º)

C1—C2	1.378 (3)	C10—C9	1.376 (3)
C1—C6	1.382 (3)	C10—H10	0.9300
C1—S1	1.7538 (19)	C11—H11A	0.9600
C2—C3	1.388 (3)	C11—H11B	0.9600
C2—H2	0.9300	C11—H11C	0.9600
C3—C4	1.373 (3)	N1—S1	1.6465 (16)
C3—H3	0.9300	N1—H1	0.83 (2)
C4—C5	1.367 (3)	O1—S1	1.4267 (15)
C4—H4	0.9300	O2—S1	1.4226 (15)
C5—C6	1.368 (3)	C9—C12	1.507 (4)
C5—H5	0.9300	C12—O3	1.211 (5)
C6—H6	0.9300	C12—C13	1.497 (6)
C7—N2	1.330 (2)	C13—H13A	0.9600
C7—N3	1.337 (2)	C13—H13B	0.9600
C7—N1	1.374 (2)	C13—H13C	0.9600
C8—N2	1.338 (3)	C9A—C12A	1.501 (5)

C8—C9A	1.412 (3)	C12A—O3A	1.207 (5)
C8—C9	1.412 (3)	C12A—C13A	1.495 (6)
C8—C11	1.502 (3)	C13A—H13D	0.9600
C10—N3	1.329 (2)	C13A—H13E	0.9600
C10—C9A	1.376 (3)	C13A—H13F	0.9600
C2—C1—C6	121.33 (19)	C7—N1—S1	127.69 (14)
C2—C1—S1	120.01 (15)	C7—N1—H1	118.2 (14)
C6—C1—S1	118.66 (15)	S1—N1—H1	112.6 (15)
C1—C2—C3	118.4 (2)	C7—N2—C8	117.10 (17)
C1—C2—H2	120.8	C10—N3—C7	115.02 (17)
C3—C2—H2	120.8	O2—S1—O1	119.43 (10)
C4—C3—C2	120.1 (2)	O2—S1—N1	110.41 (9)
C4—C3—H3	120.0	O1—S1—N1	102.84 (9)
C2—C3—H3	120.0	O2—S1—C1	108.71 (9)
C5—C4—C3	120.8 (2)	O1—S1—C1	108.55 (9)
C5—C4—H4	119.6	N1—S1—C1	106.06 (9)
C3—C4—H4	119.6	C10—C9—C8	115.92 (19)
C4—C5—C6	120.2 (2)	C10—C9—C12	120.0 (3)
C4—C5—H5	119.9	C8—C9—C12	123.6 (3)
C6—C5—H5	119.9	O3—C12—C13	120.4 (5)
C5—C6—C1	119.3 (2)	O3—C12—C9	121.9 (4)
C5—C6—H6	120.3	C13—C12—C9	117.6 (5)
C1—C6—H6	120.3	C12—C13—H13A	109.5
N2—C7—N3	126.86 (17)	C12—C13—H13B	109.5
N2—C7—N1	118.71 (17)	H13A—C13—H13B	109.5
N3—C7—N1	114.43 (16)	C12—C13—H13C	109.5
N2—C8—C9A	120.86 (18)	H13A—C13—H13C	109.5
N2—C8—C9	120.86 (18)	H13B—C13—H13C	109.5
N2—C8—C11	114.9 (2)	C10—C9A—C8	115.92 (19)
C9A—C8—C11	124.2 (2)	C10—C9A—C12A	120.4 (3)
C9—C8—C11	124.2 (2)	C8—C9A—C12A	122.5 (3)
N3—C10—C9A	124.15 (19)	O3A—C12A—C13A	121.1 (5)
N3—C10—C9	124.15 (19)	O3A—C12A—C9A	120.1 (5)
N3—C10—H10	117.9	C13A—C12A—C9A	118.6 (6)
C9—C10—H10	117.9	C12A—C13A—H13D	109.5
C8—C11—H11A	109.5	C12A—C13A—H13E	109.5
C8—C11—H11B	109.5	H13D—C13A—H13E	109.5
H11A—C11—H11B	109.5	C12A—C13A—H13F	109.5
C8—C11—H11C	109.5	H13D—C13A—H13F	109.5
H11A—C11—H11C	109.5	H13E—C13A—H13F	109.5
H11B—C11—H11C	109.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N3^{i}	0.83 (2)	2.06 (2)	2.891 (2)	179 (2)
C6—H6···O2^{ii}	0.93	2.62	3.338 (3)	135

C10—H10···O1^{i}	0.93	2.54	3.243 (3)	133
C11—H11*B*···O3*A*	0.96	2.26	2.769 (7)	113
C13*A*—H13*E*···O1^{iii}	0.96	2.50	3.40 (3)	156

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