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Pyridine and 3-methylpyridine solvates of the triple sulfa drug constitutent sulfamethazine

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Sulfonamides display a wide variety of pharmacological activities. Sulfamethazine [abbreviated as SMZ; systematic name 4-amino-*N*-(4,6-dimethylpyrimidin-2-yl)benzenesulfonamide], one of the constitutents of the triple sulfa drugs, has wide clinical use. Pharmaceutical solvates are crystalline solids of active pharmaceutical ingredients (APIs) incorporating one or more solvent molecules in the crystal lattice, and these have received special attention, as the solvent molecule can impart characteristic physicochemical properties to APIs and solvates, therefore playing a significant role in drug development. The ability of SMZ to form solvates has been investigated. Both pyridine and 3-methylpyridine form solvates with SMZ in 1:1 molar ratios. The pyridine monosolvate, $C_{12}H_{14}N_4O_2S\cdot C_5H_5N$, crystallizes in the orthorhombic space group $Pna2_1$, with Z = 8 and two molecules per assymetric unit, whereas the 3-methylpyridine monosolvate, $C_{12}H_{14}N_4O_2S\cdot C_6H_7N$, crystallizes in the orthorhombic space group $P2_12_12_1$, with Z = 4. Crystal structure analysis reveals intramolecular N—H$\cdots$N hydrogen bonds between the molecules of SMZ and the pyridine solvent molecules. The solvent molecules in both structures play an active part in strong intermolecular interactions, thereby contributing significantly to the stability of both structures. Three-dimensional hydrogen-bonding networks exist in both structures involving at least one sulfonyl O atom and the amine N atom. In the pyridine solvate, there is a short π–π interaction [centroid–centroid distance = 3.926 (3) Å] involving the centroids of the pyridine rings of two solvent molecules and a weak intermolecular C—H$\cdots\pi$ interaction also contributes to the stability of the crystal packing.

1. Introduction

Sulfonamides are recognized for their wide variety of pharmacological activities, including antibacterial, antitumor, anticarbonic anhydrase, hypoglycaemic, antithyroid and protease inhibitory activity. Clinically useful sulfonamides are derived from sulfanilamide, which is similar to 4-aminobenzoic acid, a factor required by bacteria for folic acid synthesis (Wolff, 1996). Sulfamethazine [abbreviated as SMZ; systematic name: 4-amino-*N*-(4,6-dimethylpyrimidin-2-yl)benzenesulfonamide], one of the constitutents of the triple sulfa drugs, has wide clinical use. The crystal structure of SMZ has been reported (Basak *et al.*, 1983; Tiwari *et al.*, 1984). Pharmaceutical solvates, crystalline solids of active pharmaceutical ingredients (APIs) which incorporate one or more solvent molecules in the crystal lattice, have received special attention as the presence of a particular solvent in the crystal lattice can impart characteristic physicochemical properties to the APIs. Therefore, solvates play a significant role in drug development (Byrn *et al.*, 1999; Lee *et al.*, 2011). As per our ongoing research program on crystallographic investigations of different derivatives of sulfonamides and their molecular

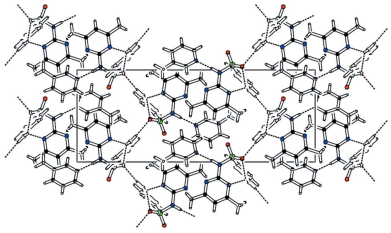
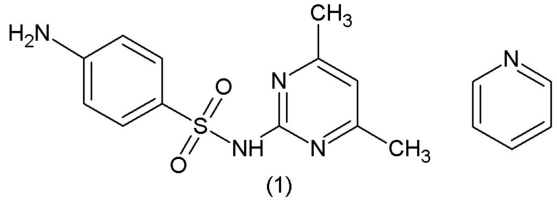


Table 1
Experimental details.

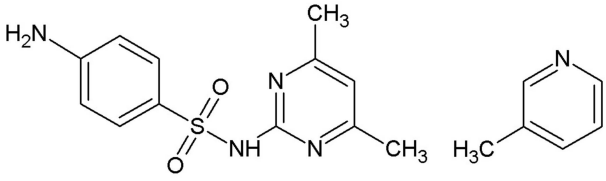
	(1)	(2)
Crystal data		
Chemical formula	$C_{12}H_{14}N_4O_2S\cdot C_5H_5N$	$C_{12}H_{14}N_4O_2S\cdot C_6H_7N$
M_r	357.43	371.46
Crystal system, space group	Orthorhombic, $Pna2_1$	Orthorhombic, $P2_12_12_1$
Temperature (K)	293	296
a, b, c (Å)	30.5388 (9), 8.0984 (2), 15.0810 (4)	9.6920 (2), 25.1673 (6), 7.9853 (2)
V (Å^3)	3729.76 (17)	1947.79 (8)
Z	8	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.19	0.19
Crystal size (mm)	0.65 × 0.30 × 0.25	0.65 × 0.60 × 0.35
Data collection		
Diffractometer	Bruker Kappa APEXII CCD	Bruker Kappa APEXII CCD
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	18564, 6825, 4494	10022, 4438, 4082
R_{int}	0.035	0.022
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.648	0.650
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.047, 0.105, 1.00	0.037, 0.102, 1.03
No. of reflections	6825	4438
No. of parameters	517	270
No. of restraints	1	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
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.19	0.23, −0.28
Absolute structure	Refined as an inversion twin (Flack, 1983)	Refined as an inversion twin (Flack, 1983)
Absolute structure parameter	0.04 (8)	−0.01 (9)

Computer programs: *APEX2* (Bruker, 2007), *SAINT* (Bruker, 2007), *SHELXS97* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 2015), *SHELXL2016* (Sheldrick, 2015), *PLATON* (Spek, 2009), *ORTEP-3* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

solvates (Tailor *et al.*, 2015), we report here the crystal structure of SMZ as the solvates of pyridine and 3-methylpyridine, *i.e.* (1) and (2), respectively.



(1)



(2)

2. Experimental

2.1. Synthesis and crystallization

Crystals of the title sulfamethazine (SMZ) solvates were grown by slow evaporation from a saturated solution of SMZ in the respective solvent [*i.e.* pyridine for (1) and 3-methylpyridine for (2)]. The solutions were allowed to stand at room temperature for a few days. Tiny transparent single crystals were collected and allowed to dry in the air.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The H atoms were positioned geometrically, with N—H = 0.90 Å for NH_2, C—H = 0.96 Å for CH_3 and C—H = 0.93 Å for aromatic H atoms. In addition, the H atoms are constrained to ride on their parent atoms, with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and $1.2U_{eq}(C,N)$ otherwise.

3. Results and discussion

The pyridine solvate of SMZ, (1), crystallizes in the orthorhombic space group $Pna2_1$, with $Z = 4$ and two molecules of SMZ and two molecules of pyridine in the asymmetric unit. The 3-methylpyridine solvate of SMZ, (2), crystallizes in the orthorhombic space group $P2_12_12_1$, also with $Z = 4$. In both solvates (Figs. 1 and 2), the bond lengths and angles of SMZ are comparable with those found in the unsolvated molecule (Basak *et al.*, 1983; Tiwari *et al.*, 1984). The endocyclic angles at atom C12 [127.3 (3)° for molecule *A* and 128.2 (4)° for molecule *B* in (1), and 127.8 (2)° in (2)] are similar to that observed in the unsolvated molecule [129.5 (1) (Tiwari *et al.*, 1984) and 129.1 (1)° (Basak *et al.*, 1983)]. The distorted tetrahedral geometries around the S atom for both the solvates are also analogous to that found in the unsolvated molecule (Basak *et al.*, 1983; Tiwari *et al.*, 1984). The angle of inclination between the planes of the two six-membered rings of SMZ is 89.03 (15)° in molecule *A* of (1), 89.40 (16)° in

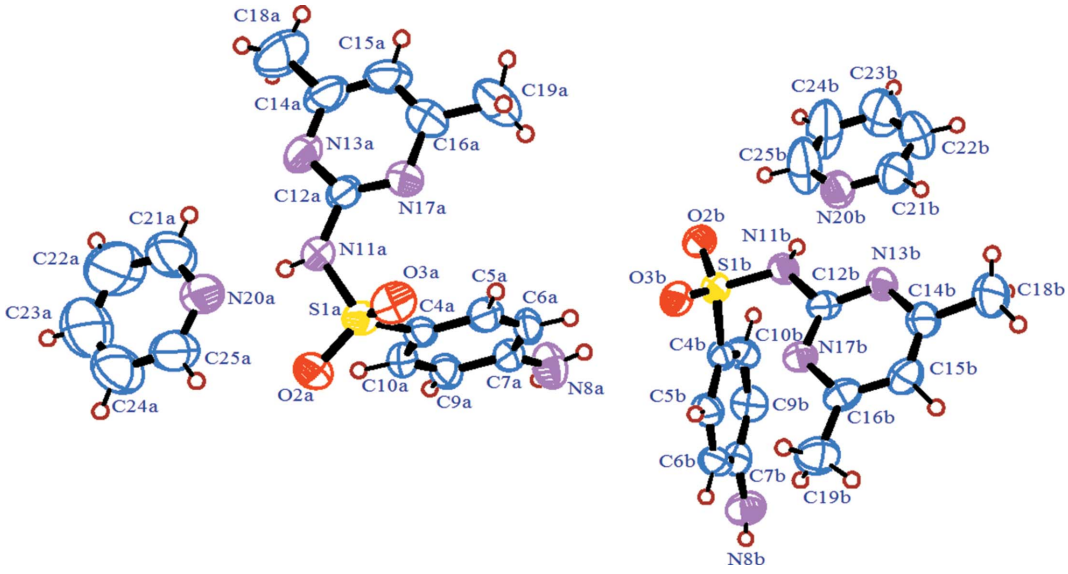


Figure 1
The molecular structure of sulfamethazine pyridine monosolvate, (1). Displacement ellipsoids are drawn at the 50% probability level.

molecule *B* of (1) and 82.81 (10)° for (2), which are different than those of the unsolvated molecule [78.1 (Tiwari *et al.*, 1984) and 75.5° (Basak *et al.*, 1983)]. The planes of the arene ring of SMZ and the pyridine ring of the solvent molecule are nearly perpendicular to each other in both solvates. The solvated structures are generally similar to each other; the orientation of the arene ring described by the C10—C4—S1—N11 torsion angle [55.1 (3)° in the unsolvated molecule; Basak *et al.*, 1983] is 75.9 (3)° for molecule *A* of (1), 78.1 (4)° for molecule *B* of (1) and 79.7 (2)° for (2). The orientation of the pyridine ring described by the N17—C12—N11—S1 torsion angle is −12.0 (6)° for molecule *A* of (1), −10.8 (5)° for molecule *B* of (1) and 18.1 (3)° for (2). The orientation of the molecule about the S1—N11 bond [83.0 (3)° in the unsolvated molecule; Basak *et al.*, 1983] is 58.9 (4)° for molecule *A* of (1), 56.1 (4)° for molecule *B* of (1) and 49.5 (2)° for (2).

In (1), the SMZ and pyridine molecules are linked *via* a hydrogen-bond interaction involving the sulfonyl N—H group of SMZ and the pyridine N atom (Fig. 3 and Table 2). In addition, the amino N atom of molecule *B* (N8*B*) hydrogen bonds *via* H81*B* to both sulfonyl atom O3*B*^{i} and pyrimidine atom N17*B*^{i} of an adjacent SMZ molecule, while the amino N atom of molecule *A* (N8*A*) hydrogen bonds *via* atom H82*A* to sulfonyl atom O3*A* of the same SMZ molecule and *via* H81*A* to sulfonyl atom O3*B* of an adjacent SMZ molecule. These hydrogen bonds link the molecules along the *b* axis. The two pyridine solvent molecules associated with molecules *A* and *B*

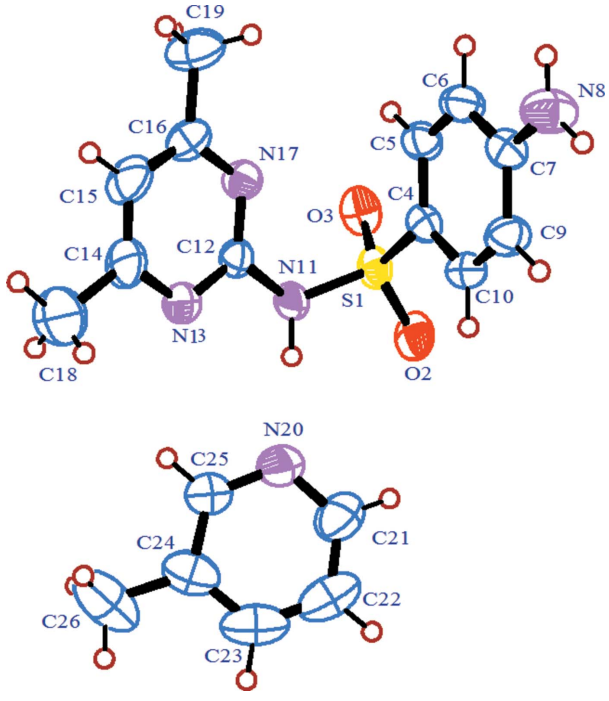


Figure 2
The molecular structure of sulfamethazine 3-methylpyridine monosolvate, (2). Displacement ellipsoids are drawn at the 50% probability level.

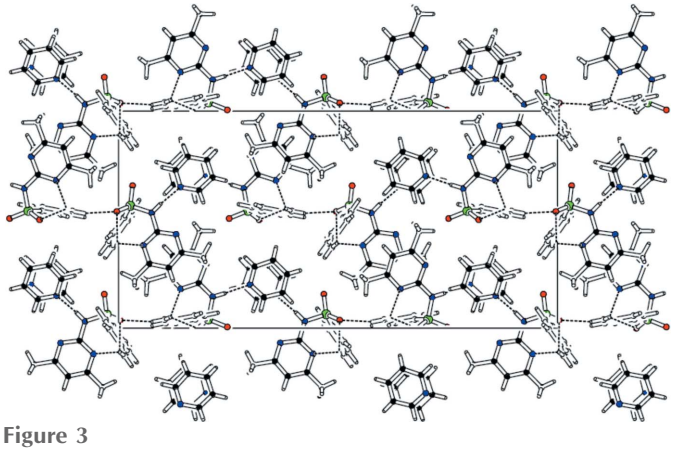


Figure 3
Packing diagram showing the hydrogen-bonding interactions in (1). The symmetry codes are as in Table 2.

Table 2
Hydrogen-bond geometry (Å, °) for (1).

*Cg*3 is the centroid of the C4–C7/C9/C10 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N8*A*—H81*A*···O3*B*	0.81 (5)	2.34 (5)	3.176 (6)	172 (5)
N8*A*—H82*A*···O3*A*	0.78 (4)	2.49 (4)	3.168 (6)	139 (4)
N8*B*—H81*B*···O3*B*^{i}	0.82 (5)	2.59 (5)	3.275 (6)	142 (4)
N8*B*—H81*B*···N17*B*^{i}	0.82 (5)	2.55 (5)	3.261 (6)	145 (4)
N11*A*—H11*A*···N20*A*	0.81 (4)	2.09 (5)	2.894 (6)	171 (5)
N11*B*—H11*B*···N20*B*	0.80 (4)	2.04 (4)	2.834 (5)	174 (3)
C15*A*—H15*A*···*Cg*3^{ii}	0.93 (4)	2.85	3.765 (5)	170

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

Table 3
Hydrogen-bond geometry (Å, °) for (2).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N8—H8*A*···O3^{i}	0.87	2.31	3.1792 (3)	173
N8—H8*B*···O3^{ii}	0.88	2.57	3.2821 (3)	139
N8—H8*B*···N17^{ii}	0.88	2.46	3.1984 (3)	141
N11—H11···N20	0.96	1.90	2.8550 (3)	174

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

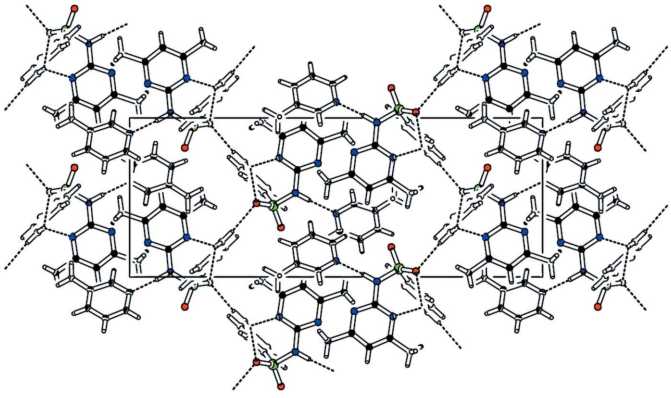


Figure 4
Packing diagram showing the hydrogen-bonding interactions (dashed lines) in (2).

are linked by a weak but significant π–π interaction [$Cg1\cdots Cg2(-x+\frac{1}{2}, y-\frac{1}{2}, z-\frac{1}{2})$ = 3.926 (3) Å; *Cg*1 and *Cg*2 are the centroids of the pyridine rings N20*A*/C21*A*–C25*A* and N20*B*/C21*B*–C25*B*, respectively, of the solvent molecules]; there is also a C—H···π interaction involving atom C15*A*, *via* H15*A*, to the centroid (*Cg*3^{ii}; Table 2) of an arene ring (atoms C4–C7/C9/C10) of an adjacent SMZ molecule.

In (2), the 3-methylpyridine solvent molecule and the pyrimidine residue of SMZ lie in the *ab* plane and are arranged in a row along the *a* axis, as highlighted in Fig. 4. The SMZ molecule and the 3-methylpyridine solvent molecule are linked *via* a hydrogen bond between the sulfonyl N—H group of SMZ and the pyridine N atom (Table 3), while the amine N—H group of SMZ is hydrogen bonded to one of the sulfonyl O atoms of two adjacent SMZ molecules.

Acknowledgements

We are thankful to DST, New Delhi, for providing the single-crystal diffractometer (Kappa APEXII) at the Department of Physics, Sardar Patel University, Vallabh Vidyanagar, Gujarat, under the DST–FIST facility. KPP is also thankful to UGC for financial support (RFSMS) to carry out research work.

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supporting information

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Pyridine and 3-methylpyridine solvates of the triple sulfa drug constitutent sulfamethazine

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Computing details

For both compounds, data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008). Program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015) for (1); *SHELXL2016* (Sheldrick, 2015) for (2). For both compounds, molecular graphics: *PLATON* (Spek, 2009) and *ORTEP-3* (Farrugia, 2012); software used to prepare material for publication: *publCIF* (Westrip, 2010).

(1) 4-Amino-*N*-(4,6-dimethylpyrimidin-2-yl)benzenesulfonamide pyridine monosolvate

Crystal data

$C_{12}H_{14}N_4O_2S·C_5H_5N$
M_r = 357.43
Orthorhombic, $Pna2_1$
a = 30.5388 (9) Å
b = 8.0984 (2) Å
c = 15.0810 (4) Å
V = 3729.76 (17) Å^3
Z = 8
$F(000)$ = 1504
D_x = 1.273 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4378 reflections
θ = 2.7–21.2°
μ = 0.19 mm^{-1}
T = 293 K
Needle, colourless
0.65 × 0.30 × 0.25 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
φ and ω scans
18564 measured reflections
6825 independent reflections
4494 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 27.4°, θ_{min} = 1.3°
h = −26→39
k = −10→8
l = −19→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.105
S = 1.00
6825 reflections
517 parameters
1 restraint
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0506P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.005
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: SHELXL2013 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0016 (4)

Absolute structure: Refined as an inversion twin (Flack, 1983)
Absolute structure parameter: 0.04 (8)

Special details

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

Refinement. Refined as a 2-component inversion twin.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N20A	−0.30004 (15)	0.8913 (5)	0.2996 (3)	0.0820 (13)
C25A	−0.3316 (2)	0.8679 (7)	0.3576 (4)	0.0906 (17)
H25A	−0.324254	0.873117	0.417375	0.109*
C24A	−0.3736 (2)	0.8368 (9)	0.3368 (6)	0.115 (2)
H24A	−0.394463	0.819105	0.380762	0.138*
C21A	−0.3108 (2)	0.8853 (10)	0.2166 (5)	0.123 (2)
H21A	−0.289126	0.904116	0.174472	0.147*
C22A	−0.3520 (3)	0.8528 (13)	0.1880 (6)	0.167 (4)
H22A	−0.358288	0.844580	0.127862	0.200*
C23A	−0.3845 (3)	0.8322 (11)	0.2516 (9)	0.158 (3)
H23A	−0.413451	0.815444	0.234656	0.189*
S1B	0.02690 (3)	0.27065 (11)	0.43631 (7)	0.0441 (3)
S1A	−0.20516 (3)	0.95563 (13)	0.46009 (7)	0.0536 (3)
O3B	−0.00381 (8)	0.3942 (3)	0.46281 (19)	0.0526 (7)
N17B	0.05700 (11)	0.3537 (4)	0.6183 (2)	0.0497 (8)
O2B	0.03220 (9)	0.2389 (3)	0.34374 (18)	0.0573 (8)
C10B	0.02973 (14)	−0.0611 (5)	0.4606 (3)	0.0533 (11)
C7A	−0.12822 (12)	0.4776 (5)	0.4602 (3)	0.0489 (9)
C4A	−0.17513 (12)	0.7738 (4)	0.4598 (3)	0.0442 (9)
C12B	0.08850 (13)	0.3473 (4)	0.5573 (3)	0.0436 (9)
N11B	0.07562 (11)	0.3225 (4)	0.4690 (2)	0.0481 (8)
O3A	−0.17959 (11)	1.0823 (3)	0.5007 (2)	0.0653 (8)
C10A	−0.19598 (14)	0.6254 (5)	0.4419 (3)	0.0532 (11)
N17A	−0.14181 (12)	1.0502 (4)	0.3192 (2)	0.0578 (9)
C6A	−0.10755 (14)	0.6291 (5)	0.4763 (3)	0.0512 (11)
N8A	−0.10533 (17)	0.3354 (6)	0.4608 (4)	0.0746 (13)
N13B	0.13131 (11)	0.3637 (4)	0.5687 (2)	0.0541 (9)
C5A	−0.13036 (14)	0.7736 (5)	0.4765 (3)	0.0513 (11)
N11A	−0.21463 (13)	1.0049 (5)	0.3560 (3)	0.0583 (10)
C4B	0.01261 (12)	0.0878 (4)	0.4906 (3)	0.0400 (9)
C6B	−0.02321 (14)	−0.0545 (5)	0.6085 (3)	0.0530 (11)
O2A	−0.24790 (9)	0.9224 (3)	0.4943 (2)	0.0667 (9)
N13A	−0.19812 (13)	1.0397 (5)	0.2096 (3)	0.0705 (11)
C16B	0.07060 (15)	0.3713 (5)	0.7036 (3)	0.0518 (11)
C15B	0.11458 (17)	0.3878 (6)	0.7215 (3)	0.0639 (12)

C5B	−0.01422 (13)	0.0901 (5)	0.5644 (3)	0.0512 (10)
C12A	−0.18292 (14)	1.0333 (5)	0.2928 (3)	0.0503 (10)
C9B	0.02067 (15)	−0.2048 (6)	0.5052 (3)	0.0600 (12)
N8B	−0.01412 (18)	−0.3465 (6)	0.6281 (4)	0.0688 (13)
C15A	−0.1248 (2)	1.0851 (7)	0.1674 (4)	0.0804 (17)
C16A	−0.11190 (16)	1.0772 (5)	0.2545 (4)	0.0656 (13)
C7B	−0.00593 (13)	−0.2046 (5)	0.5817 (3)	0.0525 (11)
C19B	0.03641 (17)	0.3704 (7)	0.7725 (3)	0.0792 (15)
H191	0.049730	0.383666	0.829738	0.119*
H192	0.016349	0.459635	0.762120	0.119*
H193	0.020867	0.267458	0.770616	0.119*
C9A	−0.17331 (13)	0.4795 (5)	0.4425 (3)	0.0549 (10)
C14B	0.14401 (15)	0.3858 (6)	0.6528 (3)	0.0596 (12)
C18B	0.19249 (16)	0.4075 (8)	0.6669 (4)	0.0966 (19)
H183	0.198278	0.422347	0.728971	0.145*
H182	0.207671	0.311243	0.646000	0.145*
H181	0.202439	0.502674	0.634770	0.145*
C14A	−0.1681 (2)	1.0682 (6)	0.1464 (3)	0.0781 (16)
C19A	−0.06528 (16)	1.0934 (7)	0.2830 (4)	0.0945 (18)
H19X	−0.047145	1.112204	0.231963	0.142*
H19Z	−0.062509	1.184781	0.323170	0.142*
H19Y	−0.056193	0.993779	0.312124	0.142*
C18A	−0.1855 (2)	1.0738 (10)	0.0532 (4)	0.135 (3)
H18Z	−0.161873	1.094631	0.012719	0.202*
H18X	−0.198941	0.969936	0.038944	0.202*
H18Y	−0.206839	1.160412	0.048313	0.202*
N20B	0.14115 (12)	0.2099 (5)	0.3505 (3)	0.0682 (10)
C21B	0.18363 (18)	0.2129 (6)	0.3658 (4)	0.0786 (15)
H21B	0.193231	0.251730	0.420524	0.094*
C25B	0.1298 (2)	0.1555 (10)	0.2723 (4)	0.125 (3)
H25B	0.100148	0.155104	0.258292	0.150*
C23B	0.2015 (2)	0.1057 (9)	0.2257 (5)	0.110 (2)
H23B	0.221605	0.072602	0.183023	0.132*
C24B	0.1583 (2)	0.0993 (11)	0.2099 (4)	0.140 (3)
H24B	0.147917	0.056723	0.156685	0.168*
C22B	0.21464 (17)	0.1617 (7)	0.3053 (5)	0.0904 (17)
H22B	0.244274	0.165852	0.319309	0.108*
H9A	−0.1899 (12)	0.378 (5)	0.427 (3)	0.059 (12)*
H82A	−0.1168 (14)	0.252 (5)	0.449 (3)	0.058 (16)*
H5A	−0.1172 (12)	0.881 (5)	0.484 (2)	0.049 (11)*
H10B	0.0500 (11)	−0.054 (4)	0.411 (3)	0.046 (10)*
H5B	−0.0245 (12)	0.199 (5)	0.591 (3)	0.061 (12)*
H10A	−0.2265 (13)	0.628 (4)	0.429 (3)	0.055 (11)*
H15B	0.1231 (12)	0.389 (4)	0.784 (3)	0.049 (11)*
H9B	0.0287 (13)	−0.307 (5)	0.484 (3)	0.066 (14)*
H81A	−0.0789 (16)	0.342 (5)	0.465 (4)	0.071 (16)*
H6A	−0.0789 (13)	0.625 (4)	0.490 (3)	0.056 (12)*
H6B	−0.0404 (14)	−0.059 (5)	0.654 (3)	0.068 (15)*

H11B	0.0926 (12)	0.286 (4)	0.434 (3)	0.047 (12)*
H11A	−0.2393 (15)	0.974 (5)	0.346 (3)	0.067 (16)*
H81B	−0.0035 (14)	−0.430 (6)	0.606 (3)	0.064 (16)*
H15A	−0.1058 (19)	1.108 (7)	0.127 (5)	0.11 (2)*
H82B	−0.025 (2)	−0.347 (8)	0.674 (5)	0.10 (3)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N20A	0.078 (3)	0.100 (3)	0.068 (3)	−0.010 (2)	−0.003 (3)	0.015 (2)
C25A	0.093 (4)	0.107 (4)	0.072 (4)	−0.027 (3)	−0.004 (4)	0.012 (3)
C24A	0.086 (5)	0.145 (6)	0.113 (6)	−0.036 (4)	0.002 (5)	0.000 (5)
C21A	0.091 (5)	0.204 (8)	0.073 (5)	−0.020 (5)	0.002 (4)	0.009 (5)
C22A	0.143 (8)	0.276 (12)	0.082 (6)	−0.057 (8)	−0.039 (6)	0.017 (6)
C23A	0.092 (6)	0.215 (9)	0.165 (10)	−0.041 (6)	−0.032 (7)	0.015 (8)
S1B	0.0428 (5)	0.0524 (6)	0.0373 (5)	−0.0013 (4)	−0.0031 (5)	0.0002 (5)
S1A	0.0545 (6)	0.0593 (6)	0.0471 (7)	0.0040 (5)	0.0064 (6)	0.0056 (5)
O3B	0.0523 (15)	0.0511 (14)	0.0544 (18)	0.0066 (12)	−0.0028 (15)	0.0054 (13)
N17B	0.051 (2)	0.060 (2)	0.038 (2)	−0.0011 (17)	0.0039 (18)	0.0018 (16)
O2B	0.0572 (17)	0.0802 (19)	0.0344 (16)	−0.0083 (14)	−0.0048 (14)	−0.0008 (14)
C10B	0.056 (2)	0.057 (3)	0.048 (3)	−0.002 (2)	0.010 (3)	−0.009 (2)
C7A	0.045 (2)	0.055 (2)	0.047 (2)	0.004 (2)	0.002 (2)	0.004 (2)
C4A	0.044 (2)	0.055 (2)	0.034 (2)	−0.0043 (18)	0.0018 (19)	0.002 (2)
C12B	0.053 (3)	0.041 (2)	0.037 (2)	−0.0071 (18)	0.002 (2)	0.0013 (17)
N11B	0.0394 (19)	0.072 (2)	0.0333 (19)	−0.0053 (16)	0.0031 (18)	−0.0053 (17)
O3A	0.081 (2)	0.0565 (17)	0.058 (2)	0.0004 (16)	0.0001 (17)	−0.0046 (13)
C10A	0.041 (2)	0.063 (3)	0.056 (3)	−0.002 (2)	−0.006 (2)	0.008 (2)
N17A	0.052 (2)	0.069 (2)	0.052 (2)	−0.0009 (18)	0.005 (2)	0.0056 (17)
C6A	0.034 (2)	0.072 (3)	0.048 (3)	0.000 (2)	−0.005 (2)	−0.001 (2)
N8A	0.055 (3)	0.060 (3)	0.109 (4)	0.005 (2)	−0.004 (3)	−0.005 (3)
N13B	0.043 (2)	0.079 (2)	0.040 (2)	−0.0167 (17)	−0.0072 (17)	0.0001 (17)
C5A	0.055 (3)	0.051 (3)	0.048 (3)	−0.010 (2)	−0.005 (2)	0.0015 (19)
N11A	0.046 (2)	0.076 (3)	0.053 (3)	0.003 (2)	0.000 (2)	0.0191 (19)
C4B	0.0341 (18)	0.044 (2)	0.042 (2)	0.0022 (17)	0.0003 (18)	−0.0006 (16)
C6B	0.048 (3)	0.061 (3)	0.051 (3)	0.004 (2)	0.012 (2)	0.006 (2)
O2A	0.0549 (17)	0.0802 (19)	0.065 (2)	0.0100 (15)	0.0162 (16)	0.0102 (15)
N13A	0.074 (3)	0.083 (3)	0.055 (3)	0.015 (2)	−0.004 (2)	0.017 (2)
C16B	0.066 (3)	0.055 (2)	0.034 (2)	0.002 (2)	0.001 (2)	−0.0030 (18)
C15B	0.068 (3)	0.085 (3)	0.039 (3)	−0.010 (2)	−0.011 (3)	0.000 (2)
C5B	0.047 (2)	0.052 (3)	0.055 (3)	0.007 (2)	0.007 (2)	0.000 (2)
C12A	0.056 (3)	0.051 (2)	0.044 (3)	0.007 (2)	0.000 (2)	0.009 (2)
C9B	0.070 (3)	0.046 (3)	0.064 (3)	0.005 (2)	0.005 (3)	−0.011 (2)
N8B	0.086 (3)	0.049 (3)	0.071 (3)	0.002 (2)	0.005 (3)	0.012 (3)
C15A	0.089 (5)	0.097 (4)	0.055 (4)	−0.001 (3)	0.024 (4)	0.017 (3)
C16A	0.061 (3)	0.067 (3)	0.069 (4)	−0.004 (2)	0.014 (3)	−0.003 (2)
C7B	0.049 (2)	0.055 (3)	0.053 (3)	0.000 (2)	−0.010 (2)	−0.001 (2)
C19B	0.084 (4)	0.105 (4)	0.049 (3)	0.009 (3)	0.012 (3)	−0.002 (3)
C9A	0.046 (2)	0.053 (3)	0.065 (3)	−0.005 (2)	−0.001 (2)	0.004 (2)

C14B	0.055 (3)	0.074 (3)	0.050 (3)	−0.018 (2)	−0.008 (3)	0.002 (2)
C18B	0.071 (4)	0.149 (5)	0.070 (4)	−0.033 (3)	−0.021 (3)	0.002 (3)
C14A	0.099 (4)	0.090 (4)	0.045 (3)	0.019 (3)	0.007 (3)	0.016 (3)
C19A	0.063 (4)	0.126 (5)	0.094 (4)	−0.023 (3)	0.019 (3)	−0.017 (3)
C18A	0.152 (6)	0.196 (7)	0.056 (4)	0.017 (5)	−0.008 (5)	0.035 (4)
N20B	0.054 (2)	0.105 (3)	0.046 (2)	−0.003 (2)	−0.001 (2)	−0.009 (2)
C21B	0.073 (4)	0.095 (4)	0.068 (4)	−0.008 (3)	−0.012 (3)	−0.005 (3)
C25B	0.063 (4)	0.250 (8)	0.062 (4)	0.030 (5)	−0.007 (3)	−0.053 (5)
C23B	0.076 (5)	0.167 (6)	0.086 (5)	0.034 (4)	0.021 (4)	−0.017 (4)
C24B	0.078 (5)	0.284 (10)	0.057 (4)	0.055 (5)	−0.006 (4)	−0.054 (5)
C22B	0.050 (3)	0.109 (4)	0.113 (6)	0.007 (3)	0.002 (4)	−0.015 (4)

Geometric parameters (Å, º)

N20A—C21A	1.294 (8)	C6B—C5B	1.374 (6)
N20A—C25A	1.314 (7)	C6B—C7B	1.385 (6)
C25A—C24A	1.347 (8)	C6B—H6B	0.87 (4)
C25A—H25A	0.9300	N13A—C12A	1.339 (5)
C24A—C23A	1.327 (11)	N13A—C14A	1.342 (6)
C24A—H24A	0.9300	C16B—C15B	1.377 (6)
C21A—C22A	1.356 (10)	C16B—C19B	1.473 (6)
C21A—H21A	0.9300	C15B—C14B	1.372 (6)
C22A—C23A	1.390 (12)	C15B—H15B	0.98 (4)
C22A—H22A	0.9300	C5B—H5B	1.02 (4)
C23A—H23A	0.9300	C9B—C7B	1.412 (6)
S1B—O3B	1.428 (3)	C9B—H9B	0.92 (4)
S1B—O2B	1.429 (3)	N8B—C7B	1.368 (6)
S1B—N11B	1.623 (3)	N8B—H81B	0.82 (4)
S1B—C4B	1.747 (4)	N8B—H82B	0.76 (6)
S1A—O3A	1.427 (3)	C15A—C14A	1.367 (8)
S1A—O2A	1.429 (3)	C15A—C16A	1.373 (8)
S1A—N11A	1.645 (4)	C15A—H15A	0.86 (6)
S1A—C4A	1.735 (4)	C16A—C19A	1.493 (7)
N17B—C12B	1.332 (5)	C19B—H191	0.9600
N17B—C16B	1.359 (5)	C19B—H192	0.9600
C10B—C9B	1.372 (6)	C19B—H193	0.9600
C10B—C4B	1.389 (5)	C9A—H9A	1.00 (4)
C10B—H10B	0.97 (4)	C14B—C18B	1.506 (6)
C7A—N8A	1.347 (5)	C18B—H183	0.9600
C7A—C6A	1.401 (5)	C18B—H182	0.9600
C7A—C9A	1.403 (5)	C18B—H181	0.9600
C4A—C10A	1.387 (5)	C14A—C18A	1.505 (8)
C4A—C5A	1.390 (5)	C19A—H19X	0.9600
C12B—N13B	1.325 (5)	C19A—H19Z	0.9600
C12B—N11B	1.403 (5)	C19A—H19Y	0.9600
N11B—H11B	0.80 (4)	C18A—H18Z	0.9600
C10A—C9A	1.369 (6)	C18A—H18X	0.9600
C10A—H10A	0.95 (4)	C18A—H18Y	0.9600

N17A—C12A	1.324 (5)	N20B—C25B	1.306 (7)
N17A—C16A	1.354 (6)	N20B—C21B	1.318 (6)
C6A—C5A	1.362 (6)	C21B—C22B	1.380 (7)
C6A—H6A	0.90 (4)	C21B—H21B	0.9300
N8A—H82A	0.78 (4)	C25B—C24B	1.360 (8)
N8A—H81A	0.81 (5)	C25B—H25B	0.9300
N13B—C14B	1.338 (5)	C23B—C24B	1.340 (8)
C5A—H5A	0.96 (4)	C23B—C22B	1.345 (8)
N11A—C12A	1.379 (5)	C23B—H23B	0.9300
N11A—H11A	0.81 (4)	C24B—H24B	0.9300
C4B—C5B	1.383 (5)	C22B—H22B	0.9300

C21A—N20A—C25A	116.9 (5)	C14B—C15B—H15B	124 (2)
N20A—C25A—C24A	124.8 (6)	C16B—C15B—H15B	117 (2)
N20A—C25A—H25A	117.6	C6B—C5B—C4B	119.8 (4)
C24A—C25A—H25A	117.6	C6B—C5B—H5B	119 (2)
C23A—C24A—C25A	118.0 (8)	C4B—C5B—H5B	121 (2)
C23A—C24A—H24A	121.0	N17A—C12A—N13A	127.3 (4)
C25A—C24A—H24A	121.0	N17A—C12A—N11A	118.4 (4)
N20A—C21A—C22A	123.4 (7)	N13A—C12A—N11A	114.3 (4)
N20A—C21A—H21A	118.3	C10B—C9B—C7B	121.0 (4)
C22A—C21A—H21A	118.3	C10B—C9B—H9B	123 (3)
C21A—C22A—C23A	117.8 (8)	C7B—C9B—H9B	116 (3)
C21A—C22A—H22A	121.1	C7B—N8B—H81B	114 (3)
C23A—C22A—H22A	121.1	C7B—N8B—H82B	123 (5)
C24A—C23A—C22A	119.0 (7)	H81B—N8B—H82B	122 (6)
C24A—C23A—H23A	120.5	C14A—C15A—C16A	119.6 (5)
C22A—C23A—H23A	120.5	C14A—C15A—H15A	121 (4)
O3B—S1B—O2B	118.29 (17)	C16A—C15A—H15A	120 (4)
O3B—S1B—N11B	109.62 (18)	N17A—C16A—C15A	120.3 (5)
O2B—S1B—N11B	103.88 (18)	N17A—C16A—C19A	116.7 (5)
O3B—S1B—C4B	107.37 (17)	C15A—C16A—C19A	123.0 (5)
O2B—S1B—C4B	109.47 (17)	N8B—C7B—C6B	121.2 (5)
N11B—S1B—C4B	107.80 (18)	N8B—C7B—C9B	121.5 (4)
O3A—S1A—O2A	118.69 (19)	C6B—C7B—C9B	117.3 (4)
O3A—S1A—N11A	109.4 (2)	C16B—C19B—H191	109.5
O2A—S1A—N11A	103.3 (2)	C16B—C19B—H192	109.5
O3A—S1A—C4A	108.77 (18)	H191—C19B—H192	109.5
O2A—S1A—C4A	108.91 (16)	C16B—C19B—H193	109.5
N11A—S1A—C4A	107.25 (19)	H191—C19B—H193	109.5
C12B—N17B—C16B	115.9 (3)	H192—C19B—H193	109.5
C9B—C10B—C4B	120.1 (4)	C10A—C9A—C7A	120.5 (4)
C9B—C10B—H10B	124 (2)	C10A—C9A—H9A	117 (2)
C4B—C10B—H10B	116 (2)	C7A—C9A—H9A	122 (2)
N8A—C7A—C6A	120.9 (4)	N13B—C14B—C15B	121.8 (4)
N8A—C7A—C9A	121.3 (4)	N13B—C14B—C18B	115.8 (4)
C6A—C7A—C9A	117.8 (4)	C15B—C14B—C18B	122.4 (4)
C10A—C4A—C5A	119.1 (4)	C14B—C18B—H183	109.5

C10A—C4A—S1A	119.6 (3)	C14B—C18B—H182	109.5
C5A—C4A—S1A	121.4 (3)	H183—C18B—H182	109.5
N13B—C12B—N17B	128.2 (4)	C14B—C18B—H181	109.5
N13B—C12B—N11B	114.5 (4)	H183—C18B—H181	109.5
N17B—C12B—N11B	117.3 (3)	H182—C18B—H181	109.5
C12B—N11B—S1B	125.6 (3)	N13A—C14A—C15A	121.0 (5)
C12B—N11B—H11B	120 (3)	N13A—C14A—C18A	115.3 (6)
S1B—N11B—H11B	107 (3)	C15A—C14A—C18A	123.7 (6)
C9A—C10A—C4A	121.0 (4)	C16A—C19A—H19X	109.5
C9A—C10A—H10A	121 (2)	C16A—C19A—H19Z	109.5
C4A—C10A—H10A	118 (2)	H19X—C19A—H19Z	109.5
C12A—N17A—C16A	116.1 (4)	C16A—C19A—H19Y	109.5
C5A—C6A—C7A	121.5 (4)	H19X—C19A—H19Y	109.5
C5A—C6A—H6A	122 (2)	H19Z—C19A—H19Y	109.5
C7A—C6A—H6A	117 (2)	C14A—C18A—H18Z	109.5
C7A—N8A—H82A	120 (3)	C14A—C18A—H18X	109.5
C7A—N8A—H81A	117 (3)	H18Z—C18A—H18X	109.5
H82A—N8A—H81A	121 (5)	C14A—C18A—H18Y	109.5
C12B—N13B—C14B	115.0 (4)	H18Z—C18A—H18Y	109.5
C6A—C5A—C4A	120.2 (4)	H18X—C18A—H18Y	109.5
C6A—C5A—H5A	124 (2)	C25B—N20B—C21B	115.2 (5)
C4A—C5A—H5A	116 (2)	N20B—C21B—C22B	123.6 (5)
C12A—N11A—S1A	125.2 (3)	N20B—C21B—H21B	118.2
C12A—N11A—H11A	125 (4)	C22B—C21B—H21B	118.2
S1A—N11A—H11A	106 (4)	N20B—C25B—C24B	124.6 (6)
C5B—C4B—C10B	119.8 (4)	N20B—C25B—H25B	117.7
C5B—C4B—S1B	120.9 (3)	C24B—C25B—H25B	117.7
C10B—C4B—S1B	119.3 (3)	C24B—C23B—C22B	117.8 (6)
C5B—C6B—C7B	122.0 (5)	C24B—C23B—H23B	121.1
C5B—C6B—H6B	123 (3)	C22B—C23B—H23B	121.1
C7B—C6B—H6B	115 (3)	C23B—C24B—C25B	119.6 (6)
C12A—N13A—C14A	115.8 (4)	C23B—C24B—H24B	120.2
N17B—C16B—C15B	119.6 (4)	C25B—C24B—H24B	120.2
N17B—C16B—C19B	116.8 (4)	C23B—C22B—C21B	119.1 (5)
C15B—C16B—C19B	123.6 (4)	C23B—C22B—H22B	120.4
C14B—C15B—C16B	119.4 (4)	C21B—C22B—H22B	120.4

C21A—N20A—C25A—C24A	0.7 (9)	C12B—N17B—C16B—C15B	2.5 (5)
N20A—C25A—C24A—C23A	−1.2 (11)	C12B—N17B—C16B—C19B	−177.1 (4)
C25A—N20A—C21A—C22A	−1.5 (11)	N17B—C16B—C15B—C14B	−0.2 (6)
N20A—C21A—C22A—C23A	2.9 (15)	C19B—C16B—C15B—C14B	179.4 (4)
C25A—C24A—C23A—C22A	2.6 (13)	C7B—C6B—C5B—C4B	0.4 (6)
C21A—C22A—C23A—C24A	−3.3 (15)	C10B—C4B—C5B—C6B	1.0 (6)
O3A—S1A—C4A—C10A	166.0 (3)	S1B—C4B—C5B—C6B	−176.8 (3)
O2A—S1A—C4A—C10A	35.2 (4)	C16A—N17A—C12A—N13A	0.1 (6)
N11A—S1A—C4A—C10A	−75.9 (4)	C16A—N17A—C12A—N11A	179.7 (4)
O3A—S1A—C4A—C5A	−14.6 (4)	C14A—N13A—C12A—N17A	−0.8 (7)
O2A—S1A—C4A—C5A	−145.3 (3)	C14A—N13A—C12A—N11A	179.6 (4)

N11A—S1A—C4A—C5A	103.6 (4)	S1A—N11A—C12A—N17A	−12.0 (6)
C16B—N17B—C12B—N13B	−3.5 (5)	S1A—N11A—C12A—N13A	167.6 (3)
C16B—N17B—C12B—N11B	177.5 (3)	C4B—C10B—C9B—C7B	−0.1 (6)
N13B—C12B—N11B—S1B	170.0 (3)	C12A—N17A—C16A—C15A	−0.3 (6)
N17B—C12B—N11B—S1B	−10.8 (5)	C12A—N17A—C16A—C19A	−178.9 (4)
O3B—S1B—N11B—C12B	60.5 (4)	C14A—C15A—C16A—N17A	1.1 (8)
O2B—S1B—N11B—C12B	−172.2 (3)	C14A—C15A—C16A—C19A	179.6 (5)
C4B—S1B—N11B—C12B	−56.1 (4)	C5B—C6B—C7B—N8B	177.9 (4)
C5A—C4A—C10A—C9A	1.6 (7)	C5B—C6B—C7B—C9B	−1.6 (6)
S1A—C4A—C10A—C9A	−179.0 (4)	C10B—C9B—C7B—N8B	−178.1 (4)
N8A—C7A—C6A—C5A	−179.5 (4)	C10B—C9B—C7B—C6B	1.5 (6)
C9A—C7A—C6A—C5A	1.2 (6)	C4A—C10A—C9A—C7A	−0.9 (7)
N17B—C12B—N13B—C14B	1.6 (6)	N8A—C7A—C9A—C10A	−179.7 (5)
N11B—C12B—N13B—C14B	−179.4 (3)	C6A—C7A—C9A—C10A	−0.5 (7)
C7A—C6A—C5A—C4A	−0.6 (6)	C12B—N13B—C14B—C15B	1.2 (6)
C10A—C4A—C5A—C6A	−0.8 (6)	C12B—N13B—C14B—C18B	−179.0 (4)
S1A—C4A—C5A—C6A	179.7 (3)	C16B—C15B—C14B—N13B	−1.8 (7)
O3A—S1A—N11A—C12A	58.8 (4)	C16B—C15B—C14B—C18B	178.4 (5)
O2A—S1A—N11A—C12A	−173.9 (4)	C12A—N13A—C14A—C15A	1.5 (7)
C4A—S1A—N11A—C12A	−58.9 (4)	C12A—N13A—C14A—C18A	179.6 (5)
C9B—C10B—C4B—C5B	−1.2 (6)	C16A—C15A—C14A—N13A	−1.7 (8)
C9B—C10B—C4B—S1B	176.7 (3)	C16A—C15A—C14A—C18A	−179.6 (5)
O3B—S1B—C4B—C5B	−18.3 (4)	C25B—N20B—C21B—C22B	−0.6 (8)
O2B—S1B—C4B—C5B	−147.9 (3)	C21B—N20B—C25B—C24B	2.2 (11)
N11B—S1B—C4B—C5B	99.7 (3)	C22B—C23B—C24B—C25B	2.9 (13)
O3B—S1B—C4B—C10B	163.9 (3)	N20B—C25B—C24B—C23B	−3.4 (14)
O2B—S1B—C4B—C10B	34.3 (4)	C24B—C23B—C22B—C21B	−1.4 (11)
N11B—S1B—C4B—C10B	−78.1 (4)	N20B—C21B—C22B—C23B	0.2 (9)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N8*A*—H81*A*···O3*B*	0.81 (5)	2.34 (5)	3.176 (6)	172 (5)
N8*A*—H82*A*···O3*A*	0.78 (4)	2.49 (4)	3.168 (6)	139 (4)
N8*B*—H81*B*···O3*B*^{i}	0.82 (5)	2.59 (5)	3.275 (6)	142 (4)
N8*B*—H81*B*···N17*B*^{i}	0.82 (5)	2.55 (5)	3.261 (6)	145 (4)
N11*A*—H11*A*···N20*A*	0.81 (4)	2.09 (5)	2.894 (6)	171 (5)
N11*B*—H11*B*···N20*B*	0.80 (4)	2.04 (4)	2.834 (5)	174 (3)

Symmetry code: (i) *x*, *y*−1, *z*.

(2) 4-Amino-*N*-(4,6-dimethylpyrimidin-2-yl)benzenesulfonamide pyridine monosolvate

Crystal data

$C_{12}H_{14}N_4O_2S·C_6H_7N$
M_r = 371.46
Orthorhombic, $P2_12_12_1$
a = 9.6920 (2) Å
b = 25.1673 (6) Å
c = 7.9853 (2) Å
V = 1947.79 (8) Å^3
Z = 4
F(000) = 784
D_x = 1.267 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6058 reflections
θ = 2.3–27.5°
μ = 0.19 mm^{-1}
T = 296 K
Needle, colorless
0.65 × 0.60 × 0.35 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
φ and ω scans
10022 measured reflections
4438 independent reflections
4082 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 27.5°, θ_{min} = 1.6°
h = −7→12
k = −32→28
l = −9→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.102
S = 1.03
4438 reflections
270 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0685P)^2 + 0.1007P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Absolute structure: Refined as an inversion twin.
Absolute structure parameter: −0.01 (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.

Refinement. Refined as a 2-component inversion twin

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

	x	y	z	U_{iso}*/U_{eq}
S1	0.44885 (5)	0.65278 (2)	0.74692 (7)	0.04252 (15)
O2	0.31271 (17)	0.63361 (8)	0.7099 (2)	0.0599 (5)
O3	0.4646 (2)	0.69307 (7)	0.8725 (2)	0.0568 (5)
N13	0.7158 (2)	0.54388 (8)	0.8259 (3)	0.0539 (5)
C6	0.6878 (3)	0.73158 (9)	0.4223 (3)	0.0495 (5)
N11	0.5299 (2)	0.59874 (7)	0.8069 (3)	0.0440 (4)
N8	0.7199 (3)	0.72280 (10)	0.1257 (3)	0.0609 (6)
C4	0.5271 (2)	0.67515 (8)	0.5639 (3)	0.0388 (4)
C5	0.6251 (3)	0.71551 (9)	0.5680 (3)	0.0460 (5)
N17	0.74845 (19)	0.63772 (7)	0.8302 (2)	0.0436 (4)
C7	0.6565 (2)	0.70779 (8)	0.2691 (3)	0.0442 (5)
C12	0.6727 (2)	0.59385 (8)	0.8216 (3)	0.0401 (4)
C9	0.5570 (3)	0.66678 (9)	0.2682 (3)	0.0496 (5)
C10	0.4941 (3)	0.65099 (9)	0.4133 (3)	0.0451 (5)
C16	0.8857 (2)	0.63022 (11)	0.8340 (3)	0.0511 (5)
C15	0.9413 (3)	0.58014 (12)	0.8330 (4)	0.0642 (7)
C24	0.4181 (4)	0.40510 (12)	0.7755 (4)	0.0715 (8)

N20	0.4000 (2)	0.49939 (9)	0.7369 (4)	0.0648 (6)
C21	0.2893 (3)	0.49161 (15)	0.6425 (5)	0.0752 (9)
H21	0.244679	0.520952	0.596926	0.090*
C25	0.4616 (3)	0.45649 (11)	0.8002 (4)	0.0652 (7)
H25	0.539742	0.461726	0.865845	0.078*
C19	0.9738 (3)	0.67906 (13)	0.8382 (5)	0.0723 (8)
H19A	0.975191	0.693219	0.949842	0.108*
H19B	0.936751	0.705135	0.762881	0.108*
H19C	1.066083	0.670151	0.804634	0.108*
C14	0.8535 (3)	0.53738 (11)	0.8339 (5)	0.0657 (8)
C22	0.2380 (4)	0.44171 (19)	0.6095 (5)	0.0879 (11)
H22	0.160184	0.437432	0.542666	0.105*
C26	0.4937 (6)	0.35923 (15)	0.8538 (8)	0.125 (2)
H26A	0.449231	0.326556	0.823323	0.187*
H26B	0.587379	0.358838	0.814647	0.187*
H26C	0.492866	0.363021	0.973420	0.187*
C18	0.9020 (4)	0.48042 (14)	0.8406 (9)	0.1108 (17)
H18A	0.877941	0.462790	0.738079	0.166*
H18B	0.858499	0.462644	0.932975	0.166*
H18C	1.000285	0.479600	0.855061	0.166*
C23	0.3029 (4)	0.39881 (15)	0.6761 (5)	0.0830 (11)
H23	0.269328	0.364876	0.654559	0.100*
H5	0.648 (3)	0.7310 (10)	0.679 (3)	0.041 (6)*
H9	0.531 (3)	0.6502 (13)	0.167 (4)	0.066 (8)*
H6	0.748 (3)	0.7616 (13)	0.425 (4)	0.065 (9)*
H8B	0.692 (3)	0.7089 (13)	0.031 (4)	0.062 (9)*
H10	0.424 (4)	0.6244 (14)	0.414 (4)	0.068 (8)*
H8A	0.782 (3)	0.7477 (12)	0.132 (4)	0.054 (8)*
H11	0.481 (3)	0.5667 (14)	0.780 (4)	0.074 (9)*
H15	1.031 (4)	0.5753 (13)	0.844 (4)	0.074 (10)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0365 (3)	0.0450 (3)	0.0461 (3)	0.0105 (2)	0.0038 (2)	0.0067 (2)
O2	0.0322 (8)	0.0758 (12)	0.0717 (11)	0.0089 (8)	0.0035 (7)	0.0193 (9)
O3	0.0677 (12)	0.0560 (10)	0.0466 (8)	0.0166 (9)	0.0066 (9)	−0.0011 (7)
N13	0.0430 (11)	0.0420 (9)	0.0767 (13)	0.0055 (8)	−0.0122 (10)	0.0026 (10)
C6	0.0558 (15)	0.0391 (11)	0.0535 (12)	−0.0099 (11)	−0.0007 (11)	−0.0010 (10)
N11	0.0339 (9)	0.0384 (8)	0.0596 (10)	0.0031 (7)	−0.0027 (8)	0.0098 (8)
N8	0.0699 (16)	0.0616 (14)	0.0511 (12)	−0.0179 (12)	0.0136 (11)	−0.0072 (10)
C4	0.0402 (11)	0.0355 (9)	0.0406 (9)	0.0042 (8)	0.0025 (9)	0.0038 (8)
C5	0.0596 (15)	0.0351 (10)	0.0434 (10)	−0.0022 (9)	−0.0046 (11)	−0.0038 (9)
N17	0.0376 (9)	0.0435 (9)	0.0496 (10)	0.0009 (7)	0.0028 (8)	−0.0002 (8)
C7	0.0475 (11)	0.0371 (9)	0.0481 (11)	0.0008 (8)	0.0022 (10)	0.0020 (9)
C12	0.0346 (10)	0.0416 (10)	0.0440 (10)	0.0036 (8)	−0.0032 (9)	0.0048 (9)
C9	0.0587 (13)	0.0483 (11)	0.0417 (11)	−0.0094 (10)	−0.0021 (11)	−0.0067 (9)
C10	0.0470 (12)	0.0403 (10)	0.0480 (10)	−0.0077 (9)	−0.0042 (9)	0.0004 (10)

C16	0.0377 (12)	0.0607 (14)	0.0548 (12)	−0.0026 (11)	−0.0001 (10)	−0.0054 (11)
C15	0.0355 (12)	0.0691 (16)	0.0880 (19)	0.0097 (13)	−0.0086 (14)	−0.0101 (15)
C24	0.081 (2)	0.0565 (14)	0.0769 (19)	−0.0125 (14)	0.0183 (17)	−0.0003 (14)
N20	0.0543 (12)	0.0563 (12)	0.0838 (16)	−0.0073 (10)	−0.0075 (12)	0.0001 (13)
C21	0.0542 (17)	0.085 (2)	0.087 (2)	−0.0046 (16)	−0.0100 (15)	−0.0019 (18)
C25	0.0632 (17)	0.0558 (14)	0.0767 (17)	−0.0085 (13)	−0.0096 (14)	0.0002 (13)
C19	0.0449 (15)	0.0752 (18)	0.097 (2)	−0.0154 (13)	0.0101 (15)	−0.0133 (18)
C14	0.0503 (15)	0.0541 (14)	0.093 (2)	0.0195 (12)	−0.0156 (15)	−0.0038 (15)
C22	0.060 (2)	0.116 (3)	0.088 (2)	−0.026 (2)	−0.0038 (17)	−0.024 (2)
C26	0.174 (6)	0.060 (2)	0.140 (4)	0.007 (3)	0.011 (4)	0.019 (3)
C18	0.072 (2)	0.0599 (18)	0.201 (5)	0.0315 (17)	−0.028 (3)	−0.009 (3)
C23	0.083 (2)	0.076 (2)	0.089 (2)	−0.035 (2)	0.025 (2)	−0.0230 (19)

Geometric parameters (Å, º)

S1—O3	1.4342 (18)	C16—C19	1.497 (4)
S1—O2	1.4357 (19)	C15—C14	1.372 (4)
S1—N11	1.6421 (18)	C15—H15	0.88 (4)
S1—C4	1.740 (2)	C24—C25	1.375 (4)
N13—C12	1.326 (3)	C24—C23	1.379 (6)
N13—C14	1.346 (3)	C24—C26	1.504 (6)
C6—C5	1.373 (4)	N20—C21	1.325 (4)
C6—C7	1.396 (3)	N20—C25	1.334 (4)
C6—H6	0.96 (3)	C21—C22	1.376 (5)
N11—C12	1.394 (3)	C21—H21	0.9300
N11—H11	0.96 (4)	C25—H25	0.9300
N8—C7	1.354 (3)	C19—H19A	0.9600
N8—H8B	0.88 (3)	C19—H19B	0.9600
N8—H8A	0.87 (3)	C19—H19C	0.9600
C4—C10	1.385 (3)	C14—C18	1.509 (4)
C4—C5	1.392 (3)	C22—C23	1.358 (6)
C5—H5	0.99 (3)	C22—H22	0.9300
N17—C12	1.328 (3)	C26—H26A	0.9600
N17—C16	1.344 (3)	C26—H26B	0.9600
C7—C9	1.412 (3)	C26—H26C	0.9600
C9—C10	1.369 (3)	C18—H18A	0.9600
C9—H9	0.94 (3)	C18—H18B	0.9600
C10—H10	0.95 (3)	C18—H18C	0.9600
C16—C15	1.371 (4)	C23—H23	0.9300
O3—S1—O2	118.66 (12)	C16—C15—H15	121 (2)
O3—S1—N11	109.31 (11)	C14—C15—H15	120 (2)
O2—S1—N11	102.80 (11)	C25—C24—C23	116.1 (3)
O3—S1—C4	108.19 (11)	C25—C24—C26	120.9 (4)
O2—S1—C4	109.64 (11)	C23—C24—C26	123.1 (3)
N11—S1—C4	107.72 (10)	C21—N20—C25	117.3 (3)
C12—N13—C14	115.4 (2)	N20—C21—C22	122.4 (3)
C5—C6—C7	121.3 (2)	N20—C21—H21	118.8

C5—C6—H6	119 (2)	C22—C21—H21	118.8
C7—C6—H6	119 (2)	N20—C25—C24	124.7 (3)
C12—N11—S1	124.94 (16)	N20—C25—H25	117.6
C12—N11—H11	116 (2)	C24—C25—H25	117.6
S1—N11—H11	113 (2)	C16—C19—H19A	109.5
C7—N8—H8B	119 (2)	C16—C19—H19B	109.5
C7—N8—H8A	118 (2)	H19A—C19—H19B	109.5
H8B—N8—H8A	123 (3)	C16—C19—H19C	109.5
C10—C4—C5	119.9 (2)	H19A—C19—H19C	109.5
C10—C4—S1	119.13 (17)	H19B—C19—H19C	109.5
C5—C4—S1	120.96 (17)	N13—C14—C15	121.3 (2)
C6—C5—C4	119.8 (2)	N13—C14—C18	115.2 (3)
C6—C5—H5	122.9 (15)	C15—C14—C18	123.5 (3)
C4—C5—H5	117.2 (15)	C23—C22—C21	118.9 (3)
C12—N17—C16	115.6 (2)	C23—C22—H22	120.6
N8—C7—C6	121.5 (2)	C21—C22—H22	120.6
N8—C7—C9	120.6 (2)	C24—C26—H26A	109.5
C6—C7—C9	117.8 (2)	C24—C26—H26B	109.5
N13—C12—N17	127.8 (2)	H26A—C26—H26B	109.5
N13—C12—N11	113.5 (2)	C24—C26—H26C	109.5
N17—C12—N11	118.68 (19)	H26A—C26—H26C	109.5
C10—C9—C7	120.8 (2)	H26B—C26—H26C	109.5
C10—C9—H9	118 (2)	C14—C18—H18A	109.5
C7—C9—H9	121 (2)	C14—C18—H18B	109.5
C9—C10—C4	120.3 (2)	H18A—C18—H18B	109.5
C9—C10—H10	122 (2)	C14—C18—H18C	109.5
C4—C10—H10	118 (2)	H18A—C18—H18C	109.5
N17—C16—C15	121.2 (2)	H18B—C18—H18C	109.5
N17—C16—C19	116.7 (2)	C22—C23—C24	120.6 (3)
C15—C16—C19	122.0 (2)	C22—C23—H23	119.7
C16—C15—C14	118.5 (2)	C24—C23—H23	119.7

O3—S1—N11—C12	−67.8 (2)	C6—C7—C9—C10	−0.1 (4)
O2—S1—N11—C12	165.2 (2)	C7—C9—C10—C4	0.0 (4)
C4—S1—N11—C12	49.5 (2)	C5—C4—C10—C9	−0.3 (4)
O3—S1—C4—C10	−162.20 (18)	S1—C4—C10—C9	−178.1 (2)
O2—S1—C4—C10	−31.4 (2)	C12—N17—C16—C15	−1.5 (4)
N11—S1—C4—C10	79.7 (2)	C12—N17—C16—C19	178.1 (2)
O3—S1—C4—C5	20.0 (2)	N17—C16—C15—C14	−2.2 (4)
O2—S1—C4—C5	150.80 (19)	C19—C16—C15—C14	178.2 (3)
N11—S1—C4—C5	−98.0 (2)	C25—N20—C21—C22	0.1 (5)
C7—C6—C5—C4	−0.7 (4)	C21—N20—C25—C24	−0.5 (5)
C10—C4—C5—C6	0.6 (4)	C23—C24—C25—N20	0.7 (5)
S1—C4—C5—C6	178.42 (19)	C26—C24—C25—N20	−179.3 (4)
C5—C6—C7—N8	−178.7 (3)	C12—N13—C14—C15	−1.7 (5)
C5—C6—C7—C9	0.5 (4)	C12—N13—C14—C18	179.4 (4)
C14—N13—C12—N17	−2.6 (4)	C16—C15—C14—N13	3.9 (5)
C14—N13—C12—N11	177.7 (3)	C16—C15—C14—C18	−177.2 (4)

C16—N17—C12—N13	4.2 (4)	N20—C21—C22—C23	0.1 (6)
C16—N17—C12—N11	−176.1 (2)	C21—C22—C23—C24	0.1 (6)
S1—N11—C12—N13	−162.20 (19)	C25—C24—C23—C22	−0.5 (5)
S1—N11—C12—N17	18.1 (3)	C26—C24—C23—C22	179.5 (4)
N8—C7—C9—C10	179.1 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N8—H8*A*···O3^{i}	0.87	2.31	3.1792 (3)	173
N8—H8*B*···O3^{ii}	0.88	2.57	3.2821 (3)	139
N8—H8*B*···N17^{ii}	0.88	2.46	3.1984 (3)	141
N11—H11···N20	0.96	1.90	2.8550 (3)	174

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