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# Crystal structure of *N*-[4-amino-5-cyano-6-(methylsulfanyl)pyridin-2-yl]acetamide hemihydrate

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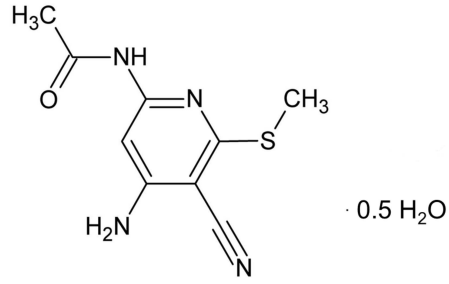
The title compound, $C_9H_{10}N_4OS\cdot0.5H_2O$, crystallizes with two independent molecules (*A* and *B*) in the asymmetric unit, together with a water molecule of crystallization. The acetamide moiety, which has an extended conformation, is inclined to the pyridine ring by 7.95 (16)° in molecule *A* and by 1.77 (16)° in molecule *B*. In the crystal, the *A* and *B* molecules are linked by two N—H···$O_{carbonyl}$ hydrogen bonds, forming a dimer. The dimers are linked *via* N—H···N hydrogen bonds, forming ribbons that are linked by N—H···$O_{water}$ hydrogen bonds to form sheets parallel to (110). The sheets are linked by O—H···N hydrogen bonds, forming slabs, and between the slabs there are weak slipped parallel $\pi$–$\pi$ interactions [inter-centroid distance = 3.734 (2) Å, interplanar distance = 3.3505 (11) Å and slippage = 1.648 Å], forming a three-dimensional structure.

**Keywords:** crystal structure; poly-functional pyridines; acetamide; disorder; hydrogen bonding.

**CCDC reference**: 1048267

## 1. Related literature

For various applications of polyfunctional pyridines, see: Knyazhanskii *et al.* (1996); Kurfurst *et al.* (1989); Enyedy *et al.* (2003); Arora & Knaus (1999); Kim *et al.* 2004); Pillai *et al.* (2003).




## 2. Experimental

### 2.1. Crystal data

$C_9H_{10}N_4OS\cdot0.5H_2O$
$M_r$ = 231.28
Triclinic, $P\overline{1}$
$a$ = 8.229 (3) Å
$b$ = 10.181 (4) Å
$c$ = 13.198 (5) Å
$\alpha$ = 84.221 (10)°
$\beta$ = 80.036 (10)°
$\gamma$ = 82.136 (11)°
$V$ = 1075.4 (7) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.29 mm$^{-1}$
$T$ = 200 K
0.40 × 0.40 × 0.40 mm

### 2.2. Data collection

Bruker SMART X2S benchtop diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.813, $T_{max}$ = 1.000
19554 measured reflections
3775 independent reflections
2517 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.066

### 2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.150
$S$ = 1.03
3775 reflections
305 parameters
9 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.63 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.31 e Å$^{-3}$

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N3—H3*AN*···O2 | 0.88 | 2.00 | 2.870 (3) | 169 |
| N7—H7*BN*···O1 | 0.88 | 1.99 | 2.860 (3) | 170 |
| N3—H3*BN*···N8$^{i}$ | 0.88 | 2.33 | 3.080 (4) | 143 |
| N7—H7*AN*···N4$^{ii}$ | 0.88 | 2.38 | 3.116 (4) | 142 |
| N2—H2*N*···O3*A* | 0.88 | 2.12 | 2.984 (10) | 166 |
| N2—H2*N*···O3*B* | 0.88 | 2.21 | 3.079 (9) | 172 |
| N6—H6*N*···O3*A*$^{iii}$ | 0.88 | 2.26 | 3.138 (10) | 173 |
| N6—H6*N*···O3*B*$^{iii}$ | 0.88 | 2.19 | 3.051 (9) | 166 |
| O3*A*—H3*A*2···N7$^{iv}$ | 0.85 (2) | 2.36 (6) | 3.083 (12) | 143 (9) |
| O3*B*—H3*B*1···N5$^{v}$ | 0.85 (2) | 2.57 (6) | 3.239 (8) | 137 (8) |

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS2014* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics:

*PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL2014* and *PLATON*.

## Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: SU5077).

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# supporting information

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# Crystal structure of *N*-[4-amino-5-cyano-6-(methylsulfanyl)pyridin-2-yl]acetamide hemihydrate

**Mehmet Akkurt, Kyle S. Knight, Shaaban K. Mohamed, Bahgat R. M. Hussein and Mustafa R. Albayati**

## S1. Comment

Poly-functional pyridines are an interesting class of compounds due to their optical properties (Knyazhanskii *et al.*, 1996; Kurfurst *et al.*, 1989), and their biological activities (Enyedy *et al.*, 2003), such as anticonvulsants (Arora *et al.*, 1999), antihistaminic reagents (Kim *et al.*, 2004), and cardivascular disorder treatments (Pillai *et al.*, 2003). In view of such facts we herein report on the synthesis and crystal structure of the new title poly-functional pyridine compound.

The asymmetric unit of the title compound, Fig. 1, contains two independent title molecules (A and B) and one water molecule. In molecule A the dihedral angle between the pyridine ring (N1/C1–C5) and the acetamide moiety (O1/N2(C7/C8) is 7.95 (16)°, while in molecule B the corresponding angle, involving the pyridine ring (N5/C10-C14) and actemide moiety (O2/N2/C7/C8), is 1.77 (16)°.

In the crystal, the A and B molecules are linked by two N-H···$O_{carbonyl}$ hydrogen bonds forming a dimer (Table 1 and Fig. 1). The dimers are linked via N-H···N hydrogen bonds forming ribbons that are linked by N-H···$O_{water}$ hydrogen bonds to form sheets parallel to (110); see Table 1. The sheets are linked by O-H···N hydrogen bonds forming slabs (Table 1). Between the slabs there are weak slipped parallel $\pi$-$\pi$ interactions forming a three-dimensional structure, Fig. 2 [inter-centroid distance Cg1···$Cg1^{i}$ = 3.734 (2) Å, inter-planar distance = 3.351 (1) Å, slippage = 1.648 Å; Cg1 is the centroid of ring N5/C10-C14 (molecule B); symmetry code: (i) -x, -y+1, -z+1].

## S2. Experimental

A solution of 0.5 g (2.7 mmol) of 4,6-diamino-3-cyano-2-methylthiopyridine-2(1*H*)-thione in 30 ml glacial acetic acid was refluxed for 3 h. The reaction mixture was allowed to cool and it was then poured into 100 ml of ice cold water. The formed precipitate was collected and dried under vacuum. Yellow crystals, suitable for X-ray analysis, were obtained by recrystallization of the solid product from ethanol (yield: 92%; m.p.: 523–525 K).

## S3. Refinement

The water molecule O3 is disordered over two positions (O3A/O3B) and was refined with an occupancy ratio of 0.5:0.5. The H atoms were included in calculated positions (calc-OH in WinGX; Farrugia, 2012 and refined with distance restraints: O—H = 0.84 (2) Å and H···H = 1.35 (2) Å with $U_{iso}$(H) = 1.5$U_{eq}$(O). The C and N-bound H atoms were placed in calculated positions and treated as riding atoms: C—H = 0.95 - 0.98 Å and N—H = 0.88 Å, with $U_{iso}$(H) = 1.5$U_{eq}$(C) for methyl H atoms and = 1.2$U_{eq}$(N,C) for other H atoms.

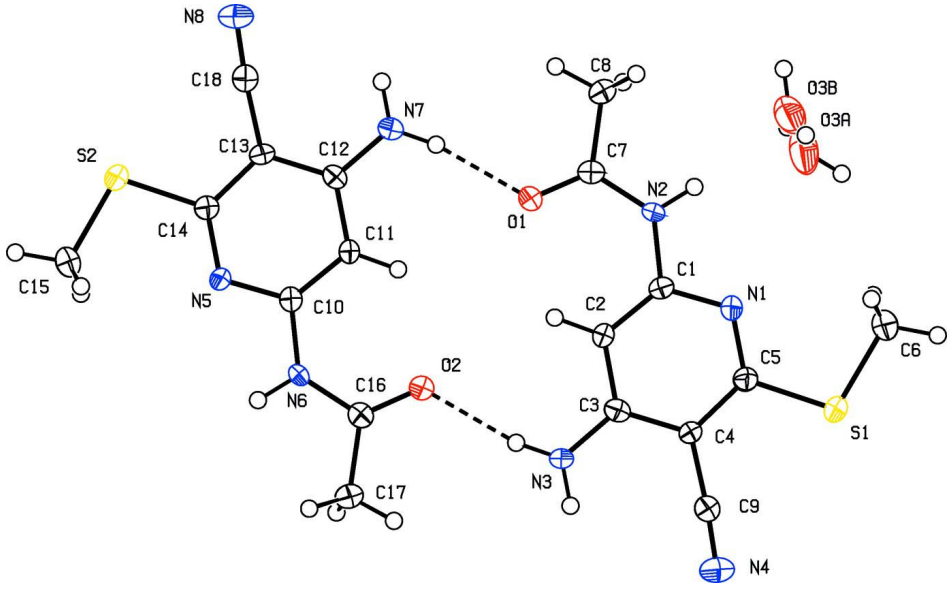


**Figure 1**

Molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level. The N—H···O hydrogen bonds are shown as dashed lines (see Table 1 for details).

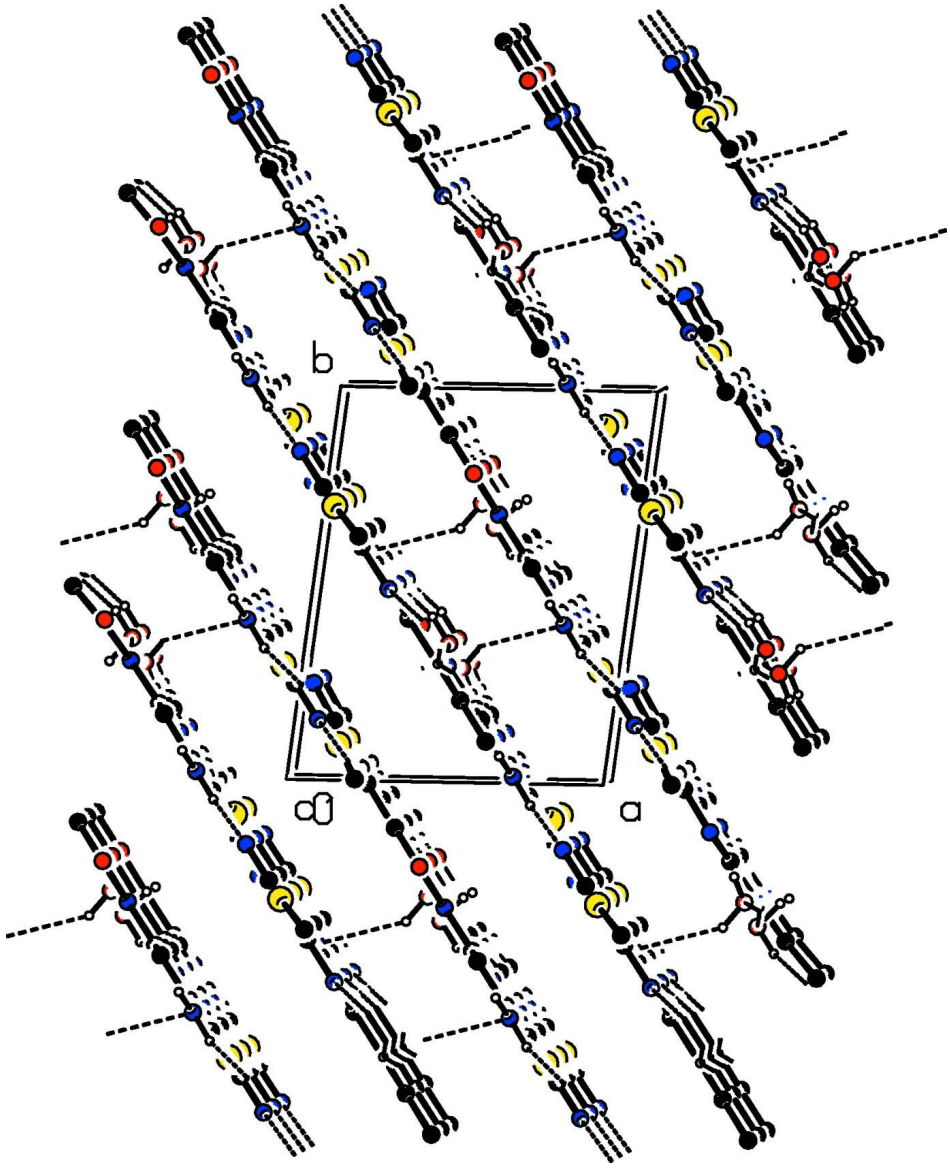


**Figure 2**

View along the *c* axis of the crystal packing of the title compound. The hydrogen bonds are shown as dashed lines (see Table 1 for details; H atoms not involved in hydrogen bonding have been omitted for clarity).

### *N*-[4-Amino-5-cyano-6-(methylsulfanyl)pyridin-2-yl]acetamide hemihydrate

*Crystal data*

$C_9H_{10}N_4OS·0.5H_2O$
$M_r = 231.28$
Triclinic, $P\overline{1}$
$a = 8.229$ (3) Å
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$c = 13.198$ (5) Å
$\alpha = 84.221$ (10)°
$\beta = 80.036$ (10)°
$\gamma = 82.136$ (11)°
$V = 1075.4$ (7) Å$^3$
$Z = 4$
$F(000) = 484$
$D_x = 1.428$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4579 reflections
$\theta = 2.5$–25.1°
$\mu = 0.29$ mm$^{-1}$
$T = 200$ K
Block, yellow
0.40 × 0.40 × 0.40 mm

*Data collection*

Bruker SMART X2S benchtop diffractometer
Radiation source: XOS X-beam microfocus source
Doubly curved silicon crystal monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.813, $T_{max}$ = 1.000
19554 measured reflections
3775 independent reflections
2517 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.066
$\theta_{max}$ = 25.2°, $\theta_{min}$ = 2.5°
$h$ = −9→9
$k$ = −12→12
$l$ = −15→15

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.150
$S$ = 1.03
3775 reflections
305 parameters
9 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0859P)^2 + 0.1759P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.63 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.31 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) |
|---|---|---|---|---|---|
| S1 | 0.84898 (9) | −0.09268 (8) | −0.11814 (5) | 0.0324 (2) | |
| O1 | 0.3621 (3) | 0.3985 (2) | 0.14350 (16) | 0.0444 (6) | |
| N1 | 0.6422 (3) | 0.1137 (2) | −0.04595 (17) | 0.0243 (5) | |
| N2 | 0.4586 (3) | 0.2943 (2) | −0.00302 (17) | 0.0263 (6) | |
| H2N | 0.4539 | 0.3015 | −0.0695 | 0.032* | |
| N3 | 0.7191 (3) | 0.0201 (3) | 0.25778 (18) | 0.0369 (7) | |
| H3AN | 0.6676 | 0.0723 | 0.3056 | 0.044* | |
| H3BN | 0.7858 | −0.0510 | 0.2744 | 0.044* | |
| N4 | 0.9584 (4) | −0.2438 (3) | 0.1290 (2) | 0.0453 (7) | |
| C1 | 0.5671 (3) | 0.1876 (3) | 0.0309 (2) | 0.0234 (6) | |
| C2 | 0.5899 (3) | 0.1621 (3) | 0.1320 (2) | 0.0267 (7) | |
| H2 | 0.5348 | 0.2200 | 0.1827 | 0.032* | |
| C3 | 0.6957 (3) | 0.0492 (3) | 0.1588 (2) | 0.0263 (7) | |
| C4 | 0.7727 (3) | −0.0312 (3) | 0.0799 (2) | 0.0224 (6) | |
| C5 | 0.7434 (3) | 0.0062 (3) | −0.0213 (2) | 0.0229 (6) | |
| C6 | 0.7793 (4) | −0.0068 (3) | −0.2309 (2) | 0.0440 (9) | |
| H6A | 0.8123 | 0.0831 | −0.2403 | 0.066* | |
| H6B | 0.6579 | −0.0013 | −0.2227 | 0.066* | |
| H6C | 0.8295 | −0.0553 | −0.2914 | 0.066* | |

| | | | | | |
|---|---|---|---|---|---|
| C7 | 0.3596 (3) | 0.3882 (3) | 0.0525 (2) | 0.0286 (7) | |
| C8 | 0.2446 (4) | 0.4802 (3) | −0.0064 (2) | 0.0359 (8) | |
| H8A | 0.1873 | 0.5507 | 0.0368 | 0.054* | |
| H8B | 0.1626 | 0.4302 | −0.0258 | 0.054* | |
| H8C | 0.3088 | 0.5199 | −0.0688 | 0.054* | |
| C9 | 0.8785 (4) | −0.1495 (3) | 0.1042 (2) | 0.0290 (7) | |
| S2 | 0.05057 (10) | 0.70420 (8) | 0.65341 (6) | 0.0356 (3) | |
| O2 | 0.5272 (3) | 0.2080 (2) | 0.39329 (15) | 0.0378 (5) | |
| N5 | 0.2537 (3) | 0.4955 (2) | 0.58226 (16) | 0.0215 (5) | |
| N6 | 0.4339 (3) | 0.3125 (2) | 0.53944 (17) | 0.0255 (5) | |
| H6N | 0.4420 | 0.3077 | 0.6053 | 0.031* | |
| N7 | 0.1738 (3) | 0.5867 (3) | 0.27873 (18) | 0.0362 (7) | |
| H7AN | 0.1079 | 0.6583 | 0.2620 | 0.043* | |
| H7BN | 0.2260 | 0.5349 | 0.2308 | 0.043* | |
| N8 | −0.0881 (3) | 0.8386 (3) | 0.4093 (2) | 0.0424 (7) | |
| C10 | 0.3278 (3) | 0.4203 (3) | 0.5052 (2) | 0.0236 (6) | |
| C11 | 0.3036 (3) | 0.4451 (3) | 0.4037 (2) | 0.0256 (6) | |
| H11 | 0.3597 | 0.3881 | 0.3526 | 0.031* | |
| C12 | 0.1947 (3) | 0.5562 (3) | 0.3782 (2) | 0.0240 (6) | |
| C13 | 0.1145 (3) | 0.6354 (3) | 0.4567 (2) | 0.0251 (7) | |
| C14 | 0.1495 (3) | 0.6013 (3) | 0.5577 (2) | 0.0251 (6) | |
| C15 | 0.1416 (4) | 0.6296 (3) | 0.7623 (2) | 0.0427 (9) | |
| H15C | 0.0914 | 0.6770 | 0.8233 | 0.064* | |
| H15B | 0.2615 | 0.6348 | 0.7486 | 0.064* | |
| H15A | 0.1216 | 0.5362 | 0.7747 | 0.064* | |
| C16 | 0.5260 (3) | 0.2144 (3) | 0.4854 (2) | 0.0262 (7) | |
| C17 | 0.6252 (4) | 0.1123 (3) | 0.5476 (2) | 0.0337 (7) | |
| H17A | 0.5529 | 0.0487 | 0.5854 | 0.051* | |
| H17B | 0.6719 | 0.1564 | 0.5967 | 0.051* | |
| H17C | 0.7157 | 0.0652 | 0.5014 | 0.051* | |
| C18 | 0.0001 (4) | 0.7485 (3) | 0.4338 (2) | 0.0298 (7) | |
| O3A | 0.4983 (13) | 0.2878 (10) | −0.2316 (7) | 0.066 (3) | 0.5 |
| H3A1 | 0.541 (6) | 0.223 (3) | −0.2637 (16) | 0.099* | 0.5 |
| H3A2 | 0.555 (10) | 0.352 (5) | −0.254 (6) | 0.099* | 0.5 |
| O3B | 0.4470 (11) | 0.3490 (9) | −0.2356 (7) | 0.052 (2) | 0.5 |
| H3B1 | 0.383 (8) | 0.416 (5) | −0.255 (7) | 0.077* | 0.5 |
| H3B2 | 0.386 (8) | 0.289 (6) | −0.214 (2) | 0.077* | 0.5 |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0361 (5) | 0.0342 (5) | 0.0234 (4) | 0.0139 (4) | −0.0053 (3) | −0.0092 (3) |
| O1 | 0.0625 (15) | 0.0445 (15) | 0.0215 (12) | 0.0279 (12) | −0.0163 (10) | −0.0105 (10) |
| N1 | 0.0268 (12) | 0.0269 (14) | 0.0176 (12) | 0.0047 (11) | −0.0036 (10) | −0.0051 (10) |
| N2 | 0.0342 (13) | 0.0255 (14) | 0.0165 (12) | 0.0124 (11) | −0.0094 (10) | −0.0014 (10) |
| N3 | 0.0503 (16) | 0.0343 (16) | 0.0206 (13) | 0.0244 (13) | −0.0121 (11) | −0.0046 (11) |
| N4 | 0.0523 (17) | 0.0385 (18) | 0.0426 (17) | 0.0174 (15) | −0.0188 (14) | −0.0031 (14) |
| C1 | 0.0246 (14) | 0.0214 (15) | 0.0222 (14) | 0.0048 (12) | −0.0043 (11) | −0.0014 (12) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C2 | 0.0325 (15) | 0.0264 (17) | 0.0194 (14) | 0.0093 (13) | −0.0059 (12) | −0.0069 (12) |
| C3 | 0.0306 (15) | 0.0257 (17) | 0.0226 (15) | 0.0035 (13) | −0.0098 (12) | −0.0014 (12) |
| C4 | 0.0235 (14) | 0.0223 (15) | 0.0199 (14) | 0.0059 (12) | −0.0051 (11) | −0.0040 (11) |
| C5 | 0.0217 (14) | 0.0245 (16) | 0.0226 (15) | 0.0017 (12) | −0.0060 (11) | −0.0042 (12) |
| C6 | 0.054 (2) | 0.049 (2) | 0.0224 (16) | 0.0151 (17) | −0.0047 (15) | −0.0050 (15) |
| C7 | 0.0321 (16) | 0.0274 (17) | 0.0244 (16) | 0.0072 (13) | −0.0079 (13) | −0.0014 (13) |
| C8 | 0.0411 (18) | 0.0340 (19) | 0.0298 (17) | 0.0164 (15) | −0.0111 (14) | −0.0085 (14) |
| C9 | 0.0347 (16) | 0.0286 (18) | 0.0233 (15) | 0.0044 (15) | −0.0085 (13) | −0.0046 (13) |
| S2 | 0.0450 (5) | 0.0339 (5) | 0.0249 (4) | 0.0171 (4) | −0.0098 (3) | −0.0110 (3) |
| O2 | 0.0507 (13) | 0.0362 (13) | 0.0234 (11) | 0.0194 (10) | −0.0134 (10) | −0.0081 (9) |
| N5 | 0.0245 (12) | 0.0211 (13) | 0.0185 (12) | 0.0055 (10) | −0.0059 (9) | −0.0061 (10) |
| N6 | 0.0319 (13) | 0.0292 (14) | 0.0139 (11) | 0.0102 (11) | −0.0090 (10) | −0.0042 (10) |
| N7 | 0.0516 (16) | 0.0314 (15) | 0.0213 (13) | 0.0178 (13) | −0.0114 (12) | −0.0028 (11) |
| N8 | 0.0465 (16) | 0.0372 (17) | 0.0392 (16) | 0.0147 (14) | −0.0137 (13) | 0.0030 (13) |
| C10 | 0.0263 (15) | 0.0249 (16) | 0.0189 (14) | 0.0021 (13) | −0.0042 (11) | −0.0042 (12) |
| C11 | 0.0324 (15) | 0.0262 (16) | 0.0174 (14) | 0.0078 (13) | −0.0071 (12) | −0.0072 (12) |
| C12 | 0.0286 (15) | 0.0252 (16) | 0.0184 (14) | 0.0013 (13) | −0.0078 (11) | −0.0016 (12) |
| C13 | 0.0284 (15) | 0.0213 (16) | 0.0241 (15) | 0.0080 (13) | −0.0084 (12) | −0.0030 (12) |
| C14 | 0.0279 (15) | 0.0246 (16) | 0.0222 (15) | 0.0042 (13) | −0.0072 (12) | −0.0041 (12) |
| C15 | 0.056 (2) | 0.046 (2) | 0.0249 (16) | 0.0141 (17) | −0.0152 (15) | −0.0120 (15) |
| C16 | 0.0281 (15) | 0.0284 (17) | 0.0219 (15) | 0.0018 (13) | −0.0066 (12) | −0.0028 (13) |
| C17 | 0.0413 (18) | 0.0279 (17) | 0.0300 (17) | 0.0108 (14) | −0.0107 (14) | −0.0044 (13) |
| C18 | 0.0358 (17) | 0.0283 (18) | 0.0238 (15) | 0.0029 (15) | −0.0052 (13) | −0.0039 (13) |
| O3A | 0.077 (7) | 0.086 (8) | 0.031 (3) | −0.003 (5) | 0.002 (4) | −0.017 (5) |
| O3B | 0.050 (5) | 0.072 (6) | 0.029 (3) | 0.004 (4) | −0.006 (3) | −0.005 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—C5 | 1.743 (3) | O2—C16 | 1.222 (3) |
| S1—C6 | 1.790 (3) | N5—C14 | 1.332 (3) |
| O1—C7 | 1.219 (3) | N5—C10 | 1.345 (3) |
| N1—C5 | 1.330 (4) | N6—C16 | 1.353 (3) |
| N1—C1 | 1.336 (4) | N6—C10 | 1.394 (3) |
| N2—C7 | 1.356 (4) | N6—H6N | 0.8800 |
| N2—C1 | 1.398 (3) | N7—C12 | 1.353 (3) |
| N2—H2N | 0.8800 | N7—H7AN | 0.8800 |
| N3—C3 | 1.350 (3) | N7—H7BN | 0.8800 |
| N3—H3AN | 0.8800 | N8—C18 | 1.145 (4) |
| N3—H3BN | 0.8800 | C10—C11 | 1.381 (4) |
| N4—C9 | 1.140 (4) | C11—C12 | 1.395 (4) |
| C1—C2 | 1.374 (4) | C11—H11 | 0.9500 |
| C2—C3 | 1.399 (4) | C12—C13 | 1.395 (4) |
| C2—H2 | 0.9500 | C13—C14 | 1.411 (4) |
| C3—C4 | 1.399 (4) | C13—C18 | 1.427 (4) |
| C4—C5 | 1.403 (4) | C15—H15C | 0.9800 |
| C4—C9 | 1.430 (4) | C15—H15B | 0.9800 |
| C6—H6A | 0.9800 | C15—H15A | 0.9800 |
| C6—H6B | 0.9800 | C16—C17 | 1.500 (4) |

| | | | |
|---|---|---|---|
| C6—H6C | 0.9800 | C17—H17A | 0.9800 |
| C7—C8 | 1.497 (4) | C17—H17B | 0.9800 |
| C8—H8A | 0.9800 | C17—H17C | 0.9800 |
| C8—H8B | 0.9800 | O3A—H3A1 | 0.823 (18) |
| C8—H8C | 0.9800 | O3A—H3A2 | 0.85 (2) |
| S2—C14 | 1.742 (3) | O3B—H3B1 | 0.85 (2) |
| S2—C15 | 1.789 (3) | O3B—H3B2 | 0.84 (2) |

| | | | |
|---|---|---|---|
| C5—S1—C6 | 102.00 (14) | C14—N5—C10 | 116.8 (2) |
| C5—N1—C1 | 116.9 (2) | C16—N6—C10 | 129.0 (2) |
| C7—N2—C1 | 128.8 (2) | C16—N6—H6N | 115.5 |
| C7—N2—H2N | 115.6 | C10—N6—H6N | 115.5 |
| C1—N2—H2N | 115.6 | C12—N7—H7AN | 120.0 |
| C3—N3—H3AN | 120.0 | C12—N7—H7BN | 120.0 |
| C3—N3—H3BN | 120.0 | H7AN—N7—H7BN | 120.0 |
| H3AN—N3—H3BN | 120.0 | N5—C10—C11 | 124.8 (3) |
| N1—C1—C2 | 124.8 (3) | N5—C10—N6 | 111.9 (2) |
| N1—C1—N2 | 112.0 (2) | C11—C10—N6 | 123.2 (2) |
| C2—C1—N2 | 123.2 (3) | C10—C11—C12 | 118.2 (2) |
| C1—C2—C3 | 118.7 (3) | C10—C11—H11 | 120.9 |
| C1—C2—H2 | 120.6 | C12—C11—H11 | 120.9 |
| C3—C2—H2 | 120.6 | N7—C12—C13 | 122.0 (3) |
| N3—C3—C2 | 120.2 (3) | N7—C12—C11 | 119.7 (3) |
| N3—C3—C4 | 122.4 (3) | C13—C12—C11 | 118.3 (2) |
| C2—C3—C4 | 117.3 (2) | C12—C13—C14 | 118.7 (2) |
| C3—C4—C5 | 119.1 (2) | C12—C13—C18 | 119.8 (2) |
| C3—C4—C9 | 119.3 (2) | C14—C13—C18 | 121.5 (2) |
| C5—C4—C9 | 121.6 (2) | N5—C14—C13 | 123.2 (2) |
| N1—C5—C4 | 123.1 (2) | N5—C14—S2 | 119.3 (2) |
| N1—C5—S1 | 119.3 (2) | C13—C14—S2 | 117.6 (2) |
| C4—C5—S1 | 117.6 (2) | S2—C15—H15C | 109.5 |
| S1—C6—H6A | 109.5 | S2—C15—H15B | 109.5 |
| S1—C6—H6B | 109.5 | H15C—C15—H15B | 109.5 |
| H6A—C6—H6B | 109.5 | S2—C15—H15A | 109.5 |
| S1—C6—H6C | 109.5 | H15C—C15—H15A | 109.5 |
| H6A—C6—H6C | 109.5 | H15B—C15—H15A | 109.5 |
| H6B—C6—H6C | 109.5 | O2—C16—N6 | 122.9 (2) |
| O1—C7—N2 | 123.4 (2) | O2—C16—C17 | 122.4 (3) |
| O1—C7—C8 | 121.8 (3) | N6—C16—C17 | 114.8 (2) |
| N2—C7—C8 | 114.8 (2) | C16—C17—H17A | 109.5 |
| C7—C8—H8A | 109.5 | C16—C17—H17B | 109.5 |
| C7—C8—H8B | 109.5 | H17A—C17—H17B | 109.5 |
| H8A—C8—H8B | 109.5 | C16—C17—H17C | 109.5 |
| C7—C8—H8C | 109.5 | H17A—C17—H17C | 109.5 |
| H8A—C8—H8C | 109.5 | H17B—C17—H17C | 109.5 |
| H8B—C8—H8C | 109.5 | N8—C18—C13 | 176.0 (3) |
| N4—C9—C4 | 176.3 (3) | H3A1—O3A—H3A2 | 108 (3) |
| C14—S2—C15 | 101.47 (14) | H3B1—O3B—H3B2 | 106 (3) |

| | | | |
|---|---|---|---|
| C5—N1—C1—C2 | 1.5 (4) | C14—N5—C10—C11 | 0.1 (4) |
| C5—N1—C1—N2 | −177.4 (2) | C14—N5—C10—N6 | −180.0 (2) |
| C7—N2—C1—N1 | 178.7 (3) | C16—N6—C10—N5 | 178.3 (3) |
| C7—N2—C1—C2 | −0.2 (5) | C16—N6—C10—C11 | −1.7 (4) |
| N1—C1—C2—C3 | −1.7 (4) | N5—C10—C11—C12 | 0.0 (4) |
| N2—C1—C2—C3 | 177.1 (3) | N6—C10—C11—C12 | −179.9 (2) |
| C1—C2—C3—N3 | −179.1 (3) | C10—C11—C12—N7 | 177.1 (3) |
| C1—C2—C3—C4 | 0.1 (4) | C10—C11—C12—C13 | −0.8 (4) |
| N3—C3—C4—C5 | −179.5 (3) | N7—C12—C13—C14 | −176.5 (3) |
| C2—C3—C4—C5 | 1.4 (4) | C11—C12—C13—C14 | 1.3 (4) |
| N3—C3—C4—C9 | 0.8 (4) | N7—C12—C13—C18 | 3.2 (4) |
| C2—C3—C4—C9 | −178.4 (2) | C11—C12—C13—C18 | −179.0 (3) |
| C1—N1—C5—C4 | 0.1 (4) | C10—N5—C14—C13 | 0.5 (4) |
| C1—N1—C5—S1 | −178.62 (19) | C10—N5—C14—S2 | −179.4 (2) |
| C3—C4—C5—N1 | −1.6 (4) | C12—C13—C14—N5 | −1.2 (4) |
| C9—C4—C5—N1 | 178.2 (3) | C18—C13—C14—N5 | 179.1 (3) |
| C3—C4—C5—S1 | 177.2 (2) | C12—C13—C14—S2 | 178.7 (2) |
| C9—C4—C5—S1 | −3.1 (4) | C18—C13—C14—S2 | −1.1 (4) |
| C6—S1—C5—N1 | 0.3 (3) | C15—S2—C14—N5 | 3.4 (3) |
| C6—S1—C5—C4 | −178.5 (2) | C15—S2—C14—C13 | −176.4 (2) |
| C1—N2—C7—O1 | 6.3 (5) | C10—N6—C16—O2 | 0.0 (5) |
| C1—N2—C7—C8 | −173.6 (3) | C10—N6—C16—C17 | −179.2 (3) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N3—H3*AN*···O2 | 0.88 | 2.00 | 2.870 (3) | 169 |
| N7—H7*BN*···O1 | 0.88 | 1.99 | 2.860 (3) | 170 |
| N3—H3*BN*···N8$^{i}$ | 0.88 | 2.33 | 3.080 (4) | 143 |
| N7—H7*AN*···N4$^{ii}$ | 0.88 | 2.38 | 3.116 (4) | 142 |
| N2—H2*N*···O3*A* | 0.88 | 2.12 | 2.984 (10) | 166 |
| N2—H2*N*···O3*B* | 0.88 | 2.21 | 3.079 (9) | 172 |
| N6—H6*N*···O3*A*$^{iii}$ | 0.88 | 2.26 | 3.138 (10) | 173 |
| N6—H6*N*···O3*B*$^{iii}$ | 0.88 | 2.19 | 3.051 (9) | 166 |
| O3*A*—H3*A*2···N7$^{iv}$ | 0.85 (2) | 2.36 (6) | 3.083 (12) | 143 (9) |
| O3*B*—H3*B*1···N5$^{v}$ | 0.85 (2) | 2.57 (6) | 3.239 (8) | 137 (8) |

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