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Crystal structures of 2-[(4,6-diaminopyrimidin-2-yl)sulfanyl]-*N*-(3-nitrophenyl)acetamide monohydrate and *N*-(2-chlorophenyl)-2-[(4,6-diaminopyrimidin-2-yl)sulfanyl]acetamide

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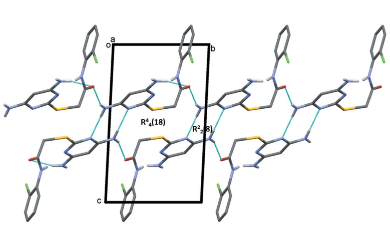
Supporting information: this article has supporting information at journals.iucr.org/e

The title compounds, $C_{12}H_{12}N_6O_3S{\cdot}H_2O$, (I), and $C_{12}H_{12}ClN_5OS$, (II), are 2-[(4,6-diaminopyrimidin-2-yl)sulfanyl]acetamides. Compound (I) crystallized as a monohydrate. In both compounds, the molecules have a folded conformation, with the pyrimidine ring being inclined to the benzene ring by 56.18 (6)° in (I) and by 67.84 (6)° in (II). In both molecules, there is an intramolecular N—H···N hydrogen bond stabilizing the folded conformation. In (I), there is also a C—H···O intramolecular short contact, and in (II) an intramolecular N—H···Cl hydrogen bond is present. In the crystal of (I), molecules are linked by a series of N—H···O, O—H···O and O—H···N hydrogen bonds, forming undulating sheets parallel to the (100). The sheets are linked *via* an N—H···O_{water} hydrogen bond, forming a three-dimensional network. In the crystal of (II), molecules are linked by a series of N—H···O, N—H···N and C—H···O hydrogen bonds, forming slabs parallel to (001).

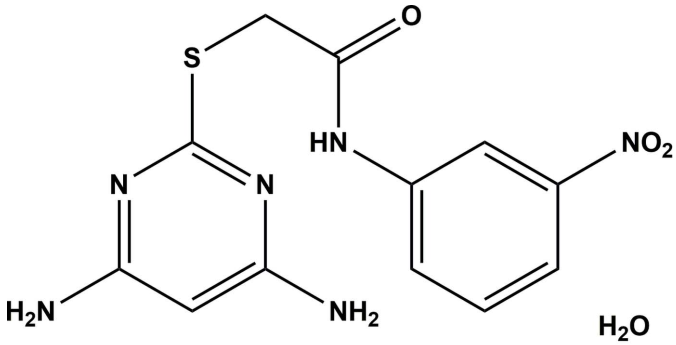
1. Chemical context

Recent studies have shown that diamino substituted pyrimidines are active inhibitors of human dihydrofolate reductase (hDHFR) and also possess inhibitory potency against tyrosine kinase (Gangjee *et al.*, 2006). 2,4-diamino pyrimidine derivatives have anti-retro viral activity (Hocková *et al.*, 2004) and also anti-trypanosoma brucei activity (Perales *et al.*, 2011). A series of 2,4-diaminopyrimidines have as also been prepared to study their immuno-suppressant activity (Blumenkopf *et al.*, 2003). Pyrimidines are also potent antiviral agents and a series of *N*-benzyl-2-(4,6-diaminopyrimidin-2-ylsulfanyl)acetamides have been designed to fight Dengue Virus Protease (Timiri *et al.*, 2016). A series 5-substituted benzyl-2,4-diamino pyrimidine derivatives have also been synthesized as c-Fms kinase inhibitors (Xu *et al.*, 2010). As part of our studies in this area, we now describe the syntheses and crystal structures of the title compounds.

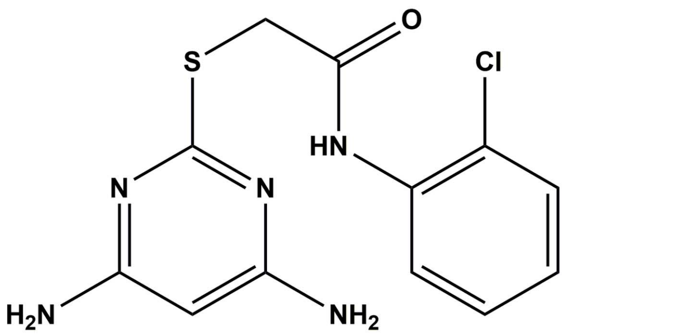


2. Structural commentary

The molecular structures of compounds (I) and (II) are illustrated in Figs. 1 and 2, respectively. In compound (I), the pyrimidine ring makes a dihedral angle of 56.18 (6)° with the benzene ring (C7–C12). The nitro group is inclined by 16.3 (3)° to the benzene ring to which it is attached. The amine nitrogen atoms, N1 and N2, are displaced from the pyrimidine ring by 0.028 (2) and 0.026 (2) Å, respectively.



(I)



(II)

In compound (II), the pyrimidine ring makes a dihedral angle of 67.84 (6)° with the chlorobenzene ring (C7–C12). The amine nitrogen atoms, N1 and N2, are displaced from the pyrimidine ring by 0.009 (2) and 0.030 (2) Å, respectively. The chlorine atom, Cl1, attached to the benzene ring deviates by 0.053 (1) Å from the ring plane.

In both the compounds, the folded conformation is reinforced by an intramolecular N—H···O hydrogen bond [Fig. 1, Table 1 for (I) and Fig. 2, Table 2 for (II)]. In (I) there is an intramolecular C—H···O contact (Table 1 and Fig. 1) and in (II) an intramolecular N—H···Cl hydrogen bond is also present (Table 2 and Fig. 2).

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5···N4	0.86 (3)	2.05 (3)	2.832 (3)	151 (3)
C12—H12···O1	0.93	2.35	2.911 (3)	118
N1—H1*A*···O1*W*^{i}	0.83 (3)	2.16 (3)	2.979 (3)	170 (2)
N1—H1*B*···O2^{ii}	0.84 (3)	2.29 (3)	3.082 (3)	159 (3)
N2—H2*A*···O3^{iii}	0.80 (3)	2.58 (3)	3.255 (3)	143 (3)
N2—H2*B*···O1^{ii}	0.83 (3)	2.09 (3)	2.904 (3)	170 (3)
O1*W*—H1*WA*···N3^{iv}	0.86	2.09	2.919 (3)	162
O1*W*—H1*WB*···O3^{v}	0.90	2.64	3.294 (3)	130

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5···N4	0.85 (2)	2.12 (2)	2.898 (2)	152 (2)
N2—H2*A*···Cl1	0.81 (3)	2.81 (2)	3.493 (2)	143 (2)
N1—H1*A*···N3^{i}	0.85 (2)	2.21 (2)	3.058 (2)	174 (2)
N1—H1*B*···O1^{ii}	0.83 (2)	2.21 (2)	2.992 (2)	157 (2)
N2—H2*A*···O1^{iii}	0.81 (3)	2.56 (2)	3.095 (2)	124 (2)
C2—H2···O1^{ii}	0.93	2.64	3.353 (2)	134

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

3. Supramolecular features

In the crystal of compound (I), molecules are linked by a series of N—H···O, O—H···O and O—H···N hydrogen bonds, forming undulating sheets parallel to the *bc* plane (Table 1 and Fig. 3). The sheets are linked *via* an N—H···O$_{water}$ hydrogen bond, forming a three-dimensional network (Table 1 and Fig. 3). Through pairs of N—H···O

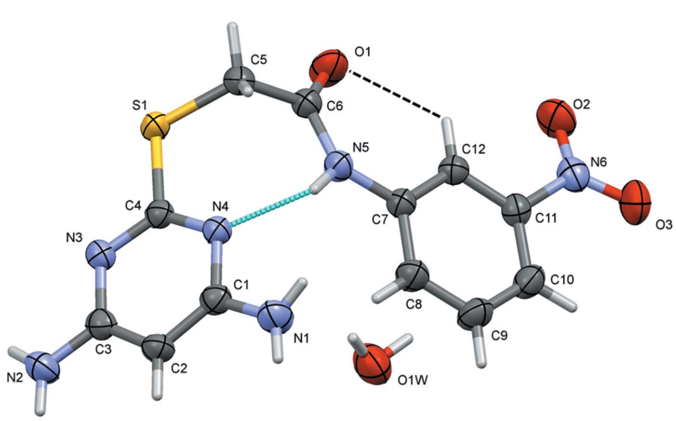


Figure 1
The molecular structure of compound (I), with atom labelling. Displacement ellipsoids are drawn at the 50% probability level. Intramolecular hydrogen bonds are shown as dashed lines (see Table 1).

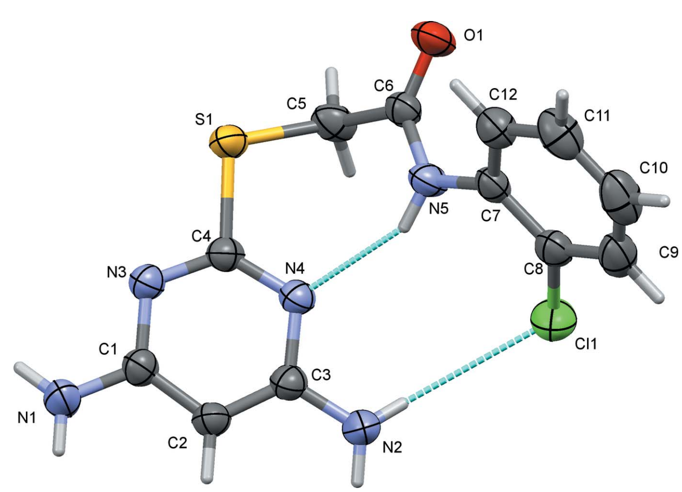


Figure 2
The molecular structure of compound (II), with atom labelling. Displacement ellipsoids are drawn at the 50% probability level. Intramolecular hydrogen bonds are shown as dashed lines (see Table 2).

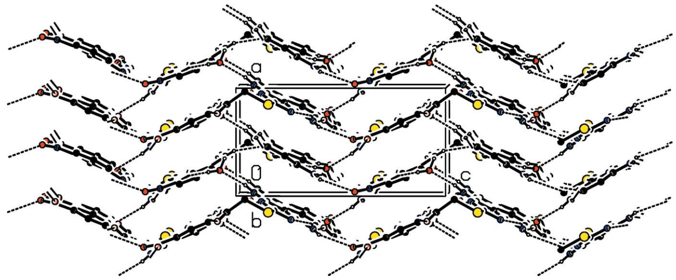


Figure 3
The crystal packing of compound (I), viewed along the *b* axis. Hydrogen bonds are shown as dashed lines (see Table 1). C-bound H atoms have been excluded for clarity.

hydrogen bonds, $R_2^2(15)$ and $R_4^4(29)$ ring motifs are generated (Table 1 and Fig. 4).

In the crystal of compound (II), molecules are linked by a series of N—H···O, N—H···N and C—H···O hydrogen bonds, forming slabs parallel to the *ab* plane (Table 2 and Fig. 5). Through pairs of N—H···N hydrogen bonds, $R^2{}_{2}(8)$ ring motifs are generated, and through further pairs of N—H···N and N—H···O hydrogen bonds $R_4^4(18)$ ring motifs are also formed (Table 2 and Fig. 6).

4. Database survey

A search of the Cambridge Structural Database (Version 5.37, update May 2016; Groom *et al.*, 2016) for 2-(pyrimidin-2-ylsulfanyl)-*N*-phenylacetamides yielded only three hits. There are two 4,6-dimethylpyrimidine analogues *viz*. 2-(4,6-dimethylpyrimidin-2-ylsulfanyl)-*N*-phenylacetamide (DIWXAJ; Gao *et al.*, 2008) and *N*-(2-chlorophenyl)-2-(4,6-dimethylpyrimidin-2-ylsulfanyl)acetamide QOTQEW; Li *et al.*, 2009), but only one 4,6-diaminopyrimidine compound *viz*. 2-[(4,6-diaminopyrimidin-2-yl)sulfanyl]-*N*-(2-methylphenyl)acetamide (GOKWIO; Subasri *et al.*, 2014). In the 4,6-dimethylpyrimidine analogues, DIWXAJ and QOTQEW, the pyrimidine ring is inclined to the benzene ring by 88.86 (15) and 79.60 (8)°, respectively. In the 4,6-diaminopyrimidine compound, GOKWIO, the two rings are inclined to one another by 54.73 (9)°. This last value is similar to that observed in the compound (I), *viz*. 56.18 (6)°.

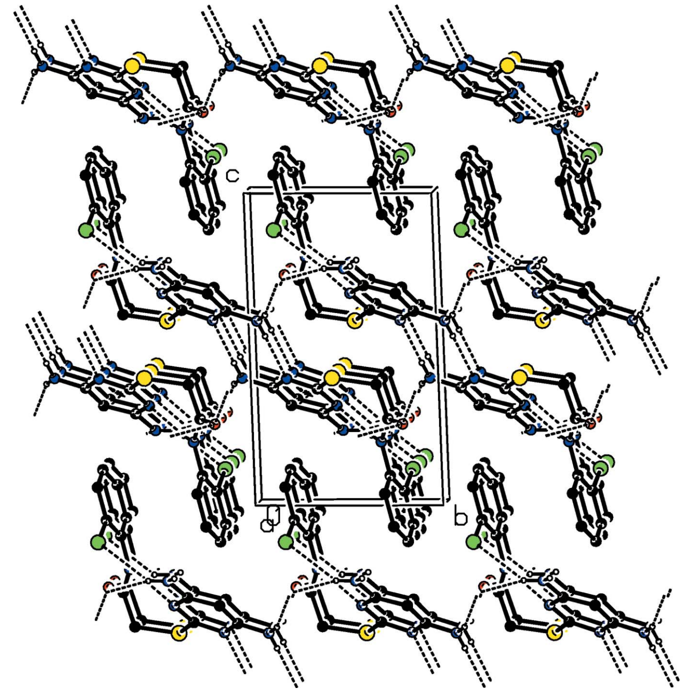


Figure 5
The crystal packing of compound (II), viewed along the *a* axis. Hydrogen bonds are shown as dashed lines (see Table 2)·C-bound H atoms have been excluded for clarity.

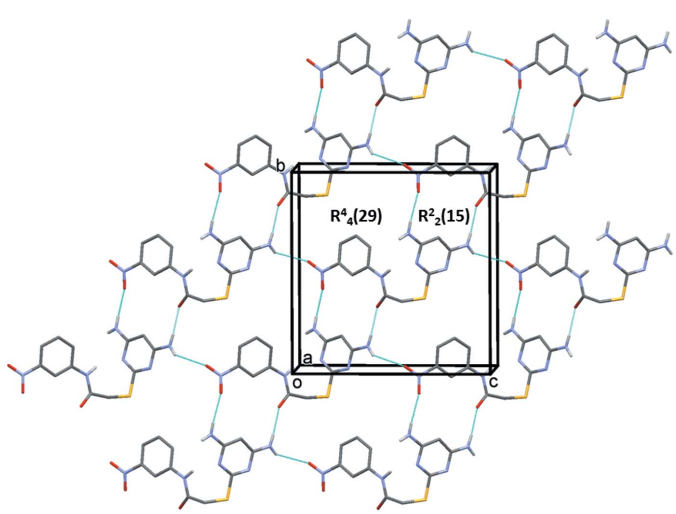


Figure 4
A view of the hydrogen-bonded ring motifs in the crystal of compound (I). Details of the hydrogen bonding are given in Table 1.

5. Synthesis and crystallization

Compound (I): To a solution of 4,6-diamino-pyrimidine-2-thiol (0.5 g; 3.52 mmol) in 25 ml of ethanol in a round-bottom flask, potassium hydroxide (0.2 g; 3.52 mmol) was added and the mixture was refluxed for half an hour and to it 3.52 mmol

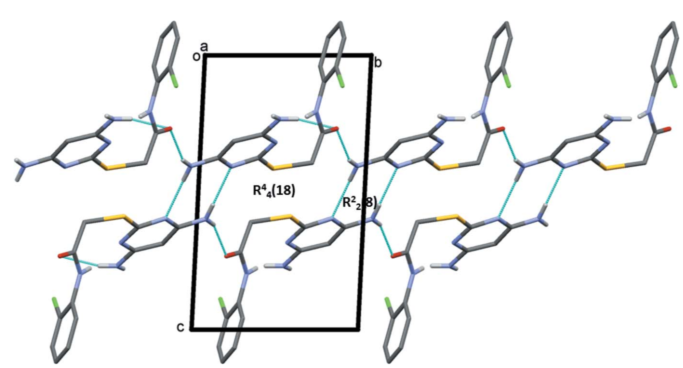


Figure 6
A view of the hydrogen-bonded ring motifs in the crystal of compound (II). Details of the hydrogen bonding are given in Table 2.

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{12}H_{12}N_6O_3S{\cdot}H_2O$	$C_{12}H_{12}ClN_5OS$
M_r	338.35	309.78
Crystal system, space group	Orthorhombic, $Pna2_1$	Triclinic, $P\overline{1}$
Temperature (K)	293	293
a, b, c (Å)	7.2326 (1), 14.3442 (2), 14.0940 (3)	7.2528 (2), 7.6249 (3), 13.0649 (4)
α, β, γ (°)	90, 90, 90	91.410 (2), 105.924 (2), 94.647 (2)
V (Å^3)	1462.19 (4)	691.68 (4)
Z	4	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.25	0.43
Crystal size (mm)	0.30 × 0.25 × 0.20	0.30 × 0.20 × 0.15
Data collection		
Diffractometer	Bruker SMART APEXII area-detector	Bruker SMART APEXII area-detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2008)	Multi-scan (*SADABS*; Bruker, 2008)
T_{min}, T_{max}	0.785, 0.845	0.785, 0.845
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7912, 3265, 3034	10154, 2822, 2519
R_{int}	0.021	0.022
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.667	0.626
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.027, 0.069, 1.04	0.035, 0.099, 1.04
No. of reflections	3265	2822
No. of parameters	229	201
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})	0.16, −0.17	0.50, −0.50
Absolute structure	Flack x determined using 1217 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)	–
Absolute structure parameter	0.07 (3)	–

Computer programs: *APEX2* (Bruker, 2008), *SAINT* (Bruker, 2008), *SHELXS97* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2008) and *PLATON* (Spek, 2009), *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

of 3-nitro phenylacetamide was added and refluxed for 4 h. At the end of the reaction (observed by TLC), ethanol was evaporated under vacuum and cold water was added and the precipitate filtered and dried to give compound (I) as a crystalline powder (yield 88–96%). After purification, the compound was recrystallized from ethyl acetate solution by slow evaporation of the solvent.

Compound (II): To a solution of 4,6-diamino-pyrimidine-2-thiol (0.5 g; 3.52 mmol) in 25 ml of ethanol in a round-bottom flask potassium hydroxide (0.2 g; 3.52 mmol) was added and refluxed for half an hour and to it 3.52 mmol of 2-chlorophenylacetamide was added and the mixture was refluxed for 3 h. At the end of the reaction (observed by TLC), ethanol was evaporated under vacuum and cold water was added, and the precipitate was filtered and dried to give compound (II) as a crystalline powder (yield 88–96%). After purification, the compound was recrystallized from ethanol solution by slow evaporation of the solvent.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. For both compounds, the NH_2 and NH H atoms, and the water H atoms for (I), were located in difference Fourier maps. The N-bound H atoms were freely refined, while the water H atoms were initially freely refined and in the final cycles of refinement as riding atoms. The C-bound H atoms were placed in calculated positions and refined as riding: C—H = 0.93–0.97 Å with $U_{iso}(H) = 1.2U_{eq}(C)$.

Acknowledgements

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References

Blumenkopf, T. A., Mueller, E. E. & Roskamp, E. J. (2003). Patent US20030191307A1.

Bruker (2008). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Gangjee, A., Yang, J., McGuire, J. J. & Kisliuk, R. L. (2006). *Bioorg. Med. Chem.* **14**, 8590–8598.

Gao, L.-X., Fang, G.-J., Feng, J.-G., Liang, D. & Wang, W. (2008). *Acta Cryst.* E**64**, o760.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Hocková, D., Holý, A. N., Masojídková, M., Andrei, G., Snoeck, R., De Clercq, E. & Balzarini, J. (2004). *Bioorg. Med. Chem.* **12**, 3197–3202.
Li, Q., Wang, W., Wang, H., Gao, Y. & Qiu, H. (2009). *Acta Cryst.* E**65**, o959.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.
Perales, J. B., Freeman, J., Bacchi, C. J., Bowling, T., Don, R., Gaukel, E., Mercer, L., Moore, J. A. III, Nare, B., Nguyen, T. M., Noe, R. A., Randolph, R., Rewerts, C., Wring, S. A., Yarlett, N. & Jacobs, R. T. (2011). *Bioorg. Med. Chem. Lett.* **21**, 2816–2819.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Subasri, S., Kumar, T. A., Sinha, B. N., Jayaprakash, V. & Velmurugan, D. (2014). *Acta Cryst.* E**70**, o850.
Timiri, A. K., Sinha, B. N. & Jayaprakash, V. (2016). *Eur. J. Med. Chem.* **117**, 125–143.
Xu, L.-B., Sun, W., Liu, H.-Y., Wang, L.-L., Xiao, J.-H., Yang, X.-H. & Li, S. (2010). *Chin. Chem. Lett.* **21**, 1318–1321.

supporting information

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Crystal structures of 2-[(4,6-diaminopyrimidin-2-yl)sulfanyl]-*N*-(3-nitrophenyl)-acetamide monohydrate and *N*-(2-chlorophenyl)-2-[(4,6-diaminopyrimidin-2-yl)sulfanyl]acetamide

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Computing details

For both compounds, data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *Mercury* (Macrae *et al.*, 2008) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

(I) 2-[(4,6-Diaminopyrimidin-2-yl)sulfanyl]-*N*-(3-nitrophenyl)acetamide monohydrate

Crystal data

$C_{12}H_{12}N_6O_3S{\cdot}H_2O$
M_r = 338.35
Orthorhombic, $Pna2_1$
a = 7.2326 (1) Å
b = 14.3442 (2) Å
c = 14.0940 (3) Å
V = 1462.19 (4) Å^3
Z = 4
F(000) = 704
D_x = 1.537 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3265 reflections
θ = 2.0–28.3°
μ = 0.25 mm^{-1}
T = 293 K
Block, colourless
0.30 × 0.25 × 0.20 mm

Data collection

Bruker SMART APEXII area-detector diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.785, T_{max} = 0.845
7912 measured reflections
3265 independent reflections
3034 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 28.3°, θ_{min} = 2.0°
$h = -5 \rightarrow 9$
$k = -18 \rightarrow 19$
$l = -15 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.069
S = 1.04
3265 reflections
229 parameters
1 restraint
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

$w = 1/[\sigma^2(F_o^2) + (0.0396P)^2 + 0.1263P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0039 (10)
Absolute structure: Flack x determined using 1217 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.07 (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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.12955 (8)	0.37215 (3)	0.66439 (4)	0.03810 (14)
O1	0.2112 (3)	0.32497 (11)	0.42274 (13)	0.0555 (5)
O2	0.4515 (3)	0.40381 (12)	0.13791 (14)	0.0570 (5)
O3	0.4586 (3)	0.53670 (14)	0.06709 (14)	0.0602 (5)
N1	−0.0426 (3)	0.69929 (14)	0.58998 (15)	0.0483 (5)
H1A	−0.112 (3)	0.6759 (19)	0.550 (2)	0.039 (7)*
H1B	−0.049 (4)	0.7564 (18)	0.601 (2)	0.048 (7)*
N2	0.2912 (3)	0.62828 (15)	0.87403 (16)	0.0473 (5)
H2A	0.343 (4)	0.587 (2)	0.902 (2)	0.052 (9)*
H2B	0.303 (4)	0.683 (2)	0.891 (2)	0.058 (8)*
N3	0.2016 (2)	0.51355 (12)	0.77054 (12)	0.0346 (4)
N4	0.0368 (2)	0.54948 (11)	0.62840 (12)	0.0327 (4)
N5	0.1324 (3)	0.47612 (12)	0.44843 (13)	0.0361 (4)
H5	0.081 (4)	0.5115 (19)	0.490 (2)	0.050 (7)*
N6	0.4308 (3)	0.48792 (13)	0.13678 (13)	0.0404 (4)
C1	0.0378 (3)	0.64158 (12)	0.65303 (16)	0.0351 (4)
C2	0.1209 (3)	0.67214 (15)	0.73550 (15)	0.0386 (5)
H2	0.1206	0.7349	0.7522	0.046*
C3	0.2048 (3)	0.60567 (14)	0.79264 (17)	0.0359 (4)
C4	0.1191 (3)	0.49297 (13)	0.68900 (14)	0.0318 (4)
C5	0.0093 (3)	0.36034 (14)	0.55277 (16)	0.0369 (5)
H5A	−0.0345	0.2967	0.5466	0.044*
H5B	−0.0979	0.4010	0.5532	0.044*
C6	0.1278 (3)	0.38380 (14)	0.46763 (15)	0.0350 (4)
C7	0.2255 (3)	0.52300 (13)	0.37571 (15)	0.0327 (4)
C8	0.2440 (4)	0.61934 (14)	0.38488 (18)	0.0418 (5)
H8	0.2021	0.6488	0.4396	0.050*
C9	0.3239 (4)	0.67138 (15)	0.31366 (19)	0.0471 (6)
H9	0.3368	0.7355	0.3211	0.057*
C10	0.3850 (3)	0.62925 (15)	0.23135 (18)	0.0418 (5)
H10	0.4364	0.6640	0.1823	0.050*
C11	0.3670 (3)	0.53390 (15)	0.22449 (15)	0.0339 (4)

C12	0.2907 (3)	0.47907 (13)	0.29473 (15)	0.0331 (4)
H12	0.2832	0.4147	0.2880	0.040*
O1W	0.7494 (3)	0.61566 (15)	0.42859 (15)	0.0615 (5)
H1WA	0.7637	0.5885	0.3748	0.092*
H1WB	0.6377	0.6003	0.4521	0.092*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0581 (3)	0.0259 (2)	0.0302 (2)	−0.00098 (19)	−0.0040 (2)	0.0025 (2)
O1	0.0851 (13)	0.0319 (8)	0.0494 (11)	0.0075 (8)	0.0205 (9)	−0.0010 (7)
O2	0.0763 (12)	0.0476 (9)	0.0470 (11)	0.0082 (9)	0.0072 (9)	−0.0122 (8)
O3	0.0765 (12)	0.0711 (12)	0.0328 (9)	−0.0071 (10)	0.0107 (9)	0.0022 (9)
N1	0.0692 (15)	0.0309 (9)	0.0448 (12)	0.0072 (9)	−0.0120 (11)	−0.0024 (8)
N2	0.0644 (14)	0.0385 (11)	0.0390 (12)	−0.0024 (10)	−0.0091 (10)	−0.0079 (9)
N3	0.0424 (9)	0.0331 (8)	0.0283 (9)	−0.0021 (7)	0.0003 (7)	−0.0017 (7)
N4	0.0427 (9)	0.0278 (7)	0.0278 (8)	−0.0008 (7)	0.0018 (7)	−0.0007 (6)
N5	0.0507 (11)	0.0288 (8)	0.0287 (9)	0.0018 (7)	0.0073 (8)	−0.0020 (7)
N6	0.0394 (9)	0.0484 (10)	0.0336 (10)	−0.0015 (8)	−0.0007 (8)	−0.0030 (8)
C1	0.0408 (10)	0.0297 (8)	0.0349 (11)	0.0008 (7)	0.0066 (9)	−0.0008 (8)
C2	0.0517 (13)	0.0281 (9)	0.0360 (11)	−0.0036 (9)	0.0036 (10)	−0.0058 (8)
C3	0.0396 (10)	0.0362 (9)	0.0320 (11)	−0.0049 (9)	0.0062 (8)	−0.0038 (8)
C4	0.0378 (10)	0.0285 (9)	0.0290 (11)	−0.0033 (8)	0.0059 (8)	−0.0007 (7)
C5	0.0467 (11)	0.0319 (10)	0.0322 (11)	−0.0077 (8)	−0.0008 (9)	−0.0009 (8)
C6	0.0446 (12)	0.0303 (9)	0.0302 (11)	−0.0013 (8)	−0.0024 (9)	−0.0007 (7)
C7	0.0395 (11)	0.0295 (9)	0.0292 (10)	−0.0005 (8)	−0.0003 (8)	0.0008 (8)
C8	0.0561 (13)	0.0317 (10)	0.0376 (12)	0.0005 (9)	0.0074 (10)	−0.0035 (9)
C9	0.0664 (15)	0.0280 (9)	0.0469 (13)	−0.0015 (10)	0.0084 (12)	0.0025 (9)
C10	0.0500 (13)	0.0350 (11)	0.0404 (13)	−0.0008 (9)	0.0078 (11)	0.0076 (9)
C11	0.0360 (10)	0.0369 (10)	0.0288 (10)	0.0026 (9)	−0.0001 (8)	−0.0002 (8)
C12	0.0380 (10)	0.0301 (8)	0.0311 (10)	−0.0006 (8)	−0.0011 (8)	−0.0003 (8)
O1W	0.0706 (12)	0.0695 (12)	0.0444 (11)	−0.0050 (9)	−0.0003 (10)	−0.0118 (9)

Geometric parameters (Å, º)

S1—C4	1.769 (2)	C1—C2	1.380 (3)
S1—C5	1.805 (2)	C2—C3	1.388 (3)
O1—C6	1.215 (3)	C2—H2	0.9300
O2—N6	1.216 (2)	C5—C6	1.512 (3)
O3—N6	1.223 (3)	C5—H5A	0.9700
N1—C1	1.347 (3)	C5—H5B	0.9700
N1—H1A	0.83 (3)	C7—C12	1.386 (3)
N1—H1B	0.84 (3)	C7—C8	1.394 (3)
N2—C3	1.346 (3)	C8—C9	1.378 (3)
N2—H2A	0.80 (3)	C8—H8	0.9300
N2—H2B	0.83 (3)	C9—C10	1.381 (3)
N3—C4	1.328 (3)	C9—H9	0.9300
N3—C3	1.358 (3)	C10—C11	1.377 (3)

N4—C4	1.319 (3)	C10—H10	0.9300
N4—C1	1.366 (2)	C11—C12	1.380 (3)
N5—C6	1.352 (3)	C12—H12	0.9300
N5—C7	1.398 (3)	O1W—H1WA	0.8582
N5—H5	0.86 (3)	O1W—H1WB	0.9004
N6—C11	1.475 (3)		

C4—S1—C5	104.01 (9)	C6—C5—H5A	108.9
C1—N1—H1A	117.8 (18)	S1—C5—H5A	108.9
C1—N1—H1B	120 (2)	C6—C5—H5B	108.9
H1A—N1—H1B	120 (3)	S1—C5—H5B	108.9
C3—N2—H2A	117 (2)	H5A—C5—H5B	107.7
C3—N2—H2B	121 (2)	O1—C6—N5	124.3 (2)
H2A—N2—H2B	121 (3)	O1—C6—C5	122.68 (19)
C4—N3—C3	114.97 (18)	N5—C6—C5	113.01 (18)
C4—N4—C1	115.30 (18)	C12—C7—C8	119.6 (2)
C6—N5—C7	129.02 (18)	C12—C7—N5	123.27 (17)
C6—N5—H5	115.5 (19)	C8—C7—N5	117.05 (19)
C7—N5—H5	115.1 (19)	C9—C8—C7	120.6 (2)
O2—N6—O3	123.9 (2)	C9—C8—H8	119.7
O2—N6—C11	118.11 (18)	C7—C8—H8	119.7
O3—N6—C11	117.96 (18)	C8—C9—C10	120.6 (2)
N1—C1—N4	115.1 (2)	C8—C9—H9	119.7
N1—C1—C2	123.28 (19)	C10—C9—H9	119.7
N4—C1—C2	121.57 (19)	C11—C10—C9	117.6 (2)
C1—C2—C3	117.44 (19)	C11—C10—H10	121.2
C1—C2—H2	121.3	C9—C10—H10	121.2
C3—C2—H2	121.3	C10—C11—C12	123.6 (2)
N2—C3—N3	116.0 (2)	C10—C11—N6	118.26 (19)
N2—C3—C2	122.1 (2)	C12—C11—N6	118.14 (18)
N3—C3—C2	121.9 (2)	C11—C12—C7	117.89 (17)
N4—C4—N3	128.79 (18)	C11—C12—H12	121.1
N4—C4—S1	119.62 (15)	C7—C12—H12	121.1
N3—C4—S1	111.59 (15)	H1WA—O1W—H1WB	108.8
C6—C5—S1	113.43 (15)		

C4—N4—C1—N1	178.57 (19)	S1—C5—C6—N5	−84.2 (2)
C4—N4—C1—C2	0.5 (3)	C6—N5—C7—C12	18.1 (3)
N1—C1—C2—C3	−177.6 (2)	C6—N5—C7—C8	−164.9 (2)
N4—C1—C2—C3	0.3 (3)	C12—C7—C8—C9	1.0 (4)
C4—N3—C3—N2	−178.7 (2)	N5—C7—C8—C9	−176.0 (2)
C4—N3—C3—C2	2.5 (3)	C7—C8—C9—C10	0.8 (4)
C1—C2—C3—N2	179.3 (2)	C8—C9—C10—C11	−1.5 (4)
C1—C2—C3—N3	−1.9 (3)	C9—C10—C11—C12	0.5 (3)
C1—N4—C4—N3	0.3 (3)	C9—C10—C11—N6	179.7 (2)
C1—N4—C4—S1	−179.23 (14)	O2—N6—C11—C10	164.8 (2)
C3—N3—C4—N4	−1.7 (3)	O3—N6—C11—C10	−15.6 (3)
C3—N3—C4—S1	177.82 (16)	O2—N6—C11—C12	−15.9 (3)

C5—S1—C4—N4	−0.85 (19)	O3—N6—C11—C12	163.6 (2)
C5—S1—C4—N3	179.56 (14)	C10—C11—C12—C7	1.3 (3)
C4—S1—C5—C6	81.31 (16)	N6—C11—C12—C7	−177.94 (17)
C7—N5—C6—O1	1.4 (4)	C8—C7—C12—C11	−2.0 (3)
C7—N5—C6—C5	−179.6 (2)	N5—C7—C12—C11	174.86 (19)
S1—C5—C6—O1	94.8 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5···N4	0.86 (3)	2.05 (3)	2.832 (3)	151 (3)
C12—H12···O1	0.93	2.35	2.911 (3)	118
N1—H1*A*···O1*W*^{i}	0.83 (3)	2.16 (3)	2.979 (3)	170 (2)
N1—H1*B*···O2^{ii}	0.84 (3)	2.29 (3)	3.082 (3)	159 (3)
N2—H2*A*···O3^{iii}	0.80 (3)	2.58 (3)	3.255 (3)	143 (3)
N2—H2*B*···O1^{ii}	0.83 (3)	2.09 (3)	2.904 (3)	170 (3)
O1*W*—H1*WA*···N3^{iv}	0.86	2.09	2.919 (3)	162
O1*W*—H1*WB*···O3^{v}	0.90	2.64	3.294 (3)	130

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

(II) *N*-(2-Chlorophenyl)-2-[(4,6-diaminopyrimidin-2-yl)sulfanyl]acetamide

Crystal data

$C_{12}H_{12}ClN_5OS$
M_r = 309.78
Triclinic, $P\overline{1}$
a = 7.2528 (2) Å
b = 7.6249 (3) Å
c = 13.0649 (4) Å
α = 91.410 (2)°
β = 105.924 (2)°
γ = 94.647 (2)°
V = 691.68 (4) Å^3
Z = 2
F(000) = 320
D_x = 1.487 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 2822 reflections
θ = 1.6–26.4°
µ = 0.43 mm^{-1}
T = 293 K
Block, colourless
0.30 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEXII area-detector diffractometer
ω and *φ* scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.785, T_{max} = 0.845
10154 measured reflections
2822 independent reflections
2519 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 26.4°, θ_{min} = 1.6°
h = −9→8
k = −9→9
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.099
S = 1.04
2822 reflections
201 parameters
0 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

$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 0.2692P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.50$ e Å^{-3}
$\Delta\rho_{min} = -0.50$ 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}
Cl1	−0.32045 (7)	0.83502 (8)	0.11080 (5)	0.06488 (18)
S1	0.22688 (6)	0.44103 (6)	0.41282 (3)	0.04714 (15)
O1	0.35760 (17)	0.81194 (17)	0.26781 (10)	0.0503 (3)
N1	−0.2505 (3)	−0.0531 (2)	0.40354 (14)	0.0541 (4)
H1A	−0.163 (3)	−0.091 (3)	0.4537 (18)	0.051 (6)*
H1B	−0.361 (3)	−0.101 (3)	0.3829 (18)	0.058 (6)*
N2	−0.4671 (2)	0.4665 (2)	0.23211 (16)	0.0576 (5)
H2A	−0.437 (3)	0.572 (3)	0.2321 (18)	0.062 (7)*
H2B	−0.584 (4)	0.428 (3)	0.2221 (19)	0.070 (7)*
N3	−0.03937 (19)	0.19020 (19)	0.40540 (10)	0.0396 (3)
N4	−0.14840 (18)	0.44884 (18)	0.31711 (10)	0.0371 (3)
N5	0.0527 (2)	0.6847 (2)	0.20471 (11)	0.0416 (3)
H5	−0.040 (3)	0.631 (3)	0.2233 (15)	0.045 (5)*
C1	−0.2221 (2)	0.1113 (2)	0.37325 (12)	0.0383 (3)
C2	−0.3723 (2)	0.1992 (2)	0.31353 (13)	0.0403 (4)
H2	−0.4970	0.1447	0.2910	0.048*
C3	−0.3310 (2)	0.3694 (2)	0.28860 (13)	0.0378 (3)
C4	−0.0176 (2)	0.3525 (2)	0.37342 (11)	0.0353 (3)
C5	0.2110 (3)	0.6742 (2)	0.39173 (13)	0.0453 (4)
H5A	0.0924	0.7068	0.4045	0.054*
H5B	0.3169	0.7396	0.4442	0.054*
C6	0.2155 (2)	0.7302 (2)	0.28193 (12)	0.0359 (3)
C7	0.0187 (2)	0.7245 (2)	0.09613 (12)	0.0375 (3)
C8	−0.1521 (2)	0.7917 (2)	0.04306 (14)	0.0428 (4)
C9	−0.1897 (3)	0.8314 (3)	−0.06301 (15)	0.0545 (5)
H9	−0.3048	0.8763	−0.0976	0.065*
C10	−0.0557 (4)	0.8039 (3)	−0.11667 (15)	0.0603 (6)
H10	−0.0795	0.8312	−0.1879	0.072*
C11	0.1148 (3)	0.7360 (3)	−0.06546 (15)	0.0556 (5)
H11	0.2048	0.7168	−0.1024	0.067*
C12	0.1516 (3)	0.6965 (2)	0.04065 (14)	0.0454 (4)
H12	0.2665	0.6508	0.0748	0.055*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0473 (3)	0.0730 (4)	0.0803 (4)	0.0106 (2)	0.0244 (2)	0.0255 (3)
S1	0.0309 (2)	0.0609 (3)	0.0435 (2)	−0.00578 (18)	0.00128 (17)	0.0193 (2)
O1	0.0404 (6)	0.0548 (7)	0.0492 (7)	−0.0158 (5)	0.0072 (5)	0.0071 (6)
N1	0.0440 (9)	0.0474 (9)	0.0579 (10)	−0.0079 (7)	−0.0061 (8)	0.0187 (7)
N2	0.0319 (8)	0.0501 (10)	0.0858 (13)	0.0021 (7)	0.0066 (8)	0.0271 (9)
N3	0.0339 (7)	0.0466 (8)	0.0349 (7)	−0.0005 (6)	0.0043 (5)	0.0105 (6)
N4	0.0318 (6)	0.0431 (7)	0.0360 (7)	−0.0010 (5)	0.0094 (5)	0.0082 (5)
N5	0.0339 (7)	0.0515 (8)	0.0360 (7)	−0.0097 (6)	0.0072 (6)	0.0099 (6)
C1	0.0388 (8)	0.0418 (8)	0.0307 (7)	−0.0015 (7)	0.0052 (6)	0.0049 (6)
C2	0.0314 (8)	0.0458 (9)	0.0390 (8)	−0.0034 (6)	0.0037 (6)	0.0068 (7)
C3	0.0322 (8)	0.0447 (9)	0.0364 (8)	0.0017 (6)	0.0097 (6)	0.0070 (6)
C4	0.0315 (7)	0.0469 (9)	0.0262 (7)	−0.0018 (6)	0.0073 (6)	0.0049 (6)
C5	0.0447 (9)	0.0511 (10)	0.0343 (8)	−0.0120 (7)	0.0062 (7)	−0.0025 (7)
C6	0.0355 (8)	0.0324 (7)	0.0376 (8)	−0.0031 (6)	0.0082 (6)	0.0013 (6)
C7	0.0398 (8)	0.0337 (8)	0.0350 (8)	−0.0089 (6)	0.0069 (6)	0.0043 (6)
C8	0.0405 (9)	0.0384 (8)	0.0446 (9)	−0.0071 (7)	0.0061 (7)	0.0053 (7)
C9	0.0582 (11)	0.0472 (10)	0.0447 (10)	−0.0083 (8)	−0.0049 (8)	0.0103 (8)
C10	0.0873 (15)	0.0526 (11)	0.0325 (9)	−0.0121 (10)	0.0073 (9)	0.0031 (8)
C11	0.0761 (14)	0.0496 (10)	0.0447 (10)	−0.0064 (9)	0.0269 (10)	−0.0024 (8)
C12	0.0486 (10)	0.0421 (9)	0.0456 (9)	−0.0015 (7)	0.0145 (8)	0.0029 (7)

Geometric parameters (Å, º)

Cl1—C8	1.7397 (19)	C1—C2	1.386 (2)
S1—C4	1.7753 (15)	C2—C3	1.375 (2)
S1—C5	1.8135 (19)	C2—H2	0.9300
O1—C6	1.2207 (19)	C5—C6	1.515 (2)
N1—C1	1.340 (2)	C5—H5A	0.9700
N1—H1A	0.85 (2)	C5—H5B	0.9700
N1—H1B	0.83 (2)	C7—C12	1.382 (2)
N2—C3	1.346 (2)	C7—C8	1.388 (2)
N2—H2A	0.81 (3)	C8—C9	1.383 (3)
N2—H2B	0.85 (3)	C9—C10	1.371 (3)
N3—C4	1.327 (2)	C9—H9	0.9300
N3—C1	1.359 (2)	C10—C11	1.382 (3)
N4—C4	1.318 (2)	C10—H10	0.9300
N4—C3	1.360 (2)	C11—C12	1.384 (3)
N5—C6	1.340 (2)	C11—H11	0.9300
N5—C7	1.417 (2)	C12—H12	0.9300
N5—H5	0.85 (2)		
C4—S1—C5	103.22 (8)	S1—C5—H5A	108.5
C1—N1—H1A	118.4 (15)	C6—C5—H5B	108.5
C1—N1—H1B	116.7 (16)	S1—C5—H5B	108.5
H1A—N1—H1B	123 (2)	H5A—C5—H5B	107.5

C3—N2—H2A	116.6 (17)	O1—C6—N5	124.20 (15)
C3—N2—H2B	118.0 (17)	O1—C6—C5	121.24 (15)
H2A—N2—H2B	120 (2)	N5—C6—C5	114.56 (14)
C4—N3—C1	115.11 (13)	C12—C7—C8	118.68 (15)
C4—N4—C3	114.72 (13)	C12—C7—N5	121.45 (15)
C6—N5—C7	125.63 (14)	C8—C7—N5	119.87 (15)
C6—N5—H5	116.8 (13)	C9—C8—C7	121.22 (18)
C7—N5—H5	117.5 (13)	C9—C8—Cl1	118.54 (15)
N1—C1—N3	117.08 (15)	C7—C8—Cl1	120.20 (13)
N1—C1—C2	121.83 (15)	C10—C9—C8	119.36 (19)
N3—C1—C2	121.08 (14)	C10—C9—H9	120.3
C3—C2—C1	117.94 (14)	C8—C9—H9	120.3
C3—C2—H2	121.0	C9—C10—C11	120.31 (17)
C1—C2—H2	121.0	C9—C10—H10	119.8
N2—C3—N4	115.59 (15)	C11—C10—H10	119.8
N2—C3—C2	122.48 (15)	C10—C11—C12	120.09 (19)
N4—C3—C2	121.90 (15)	C10—C11—H11	120.0
N4—C4—N3	129.18 (14)	C12—C11—H11	120.0
N4—C4—S1	118.71 (12)	C7—C12—C11	120.34 (18)
N3—C4—S1	112.10 (11)	C7—C12—H12	119.8
C6—C5—S1	115.29 (12)	C11—C12—H12	119.8
C6—C5—H5A	108.5		

C4—N3—C1—N1	179.80 (16)	C7—N5—C6—C5	178.60 (16)
C4—N3—C1—C2	−1.4 (2)	S1—C5—C6—O1	−106.66 (16)
N1—C1—C2—C3	178.13 (17)	S1—C5—C6—N5	74.10 (18)
N3—C1—C2—C3	−0.6 (3)	C6—N5—C7—C12	47.4 (2)
C4—N4—C3—N2	179.24 (16)	C6—N5—C7—C8	−133.27 (18)
C4—N4—C3—C2	−2.5 (2)	C12—C7—C8—C9	−0.5 (2)
C1—C2—C3—N2	−179.19 (18)	N5—C7—C8—C9	−179.80 (15)
C1—C2—C3—N4	2.7 (3)	C12—C7—C8—Cl1	−178.14 (12)
C3—N4—C4—N3	0.3 (2)	N5—C7—C8—Cl1	2.5 (2)
C3—N4—C4—S1	178.72 (11)	C7—C8—C9—C10	0.0 (3)
C1—N3—C4—N4	1.7 (2)	Cl1—C8—C9—C10	177.71 (14)
C1—N3—C4—S1	−176.86 (11)	C8—C9—C10—C11	0.5 (3)
C5—S1—C4—N4	15.56 (15)	C9—C10—C11—C12	−0.5 (3)
C5—S1—C4—N3	−165.73 (12)	C8—C7—C12—C11	0.5 (2)
C4—S1—C5—C6	−89.86 (13)	N5—C7—C12—C11	179.79 (15)
C7—N5—C6—O1	−0.6 (3)	C10—C11—C12—C7	0.0 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5···N4	0.85 (2)	2.12 (2)	2.898 (2)	152 (2)
N2—H2*A*···Cl1	0.81 (3)	2.81 (2)	3.493 (2)	143 (2)
N1—H1*A*···N3^{i}	0.85 (2)	2.21 (2)	3.058 (2)	174 (2)
N1—H1*B*···O1^{ii}	0.83 (2)	2.21 (2)	2.992 (2)	157 (2)

N2—H2*A*···O1^{iii}	0.81 (3)	2.56 (2)	3.095 (2)	124 (2)
C2—H2···O1^{ii}	0.93	2.64	3.353 (2)	134

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