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Crystal structures of 2-[(4,6-diaminopyrimidin-2-yl)sulfanyl]-*N*-(naphthalen-1-yl)acetamide and 2-[(4,6-diaminopyrimidin-2-yl)sulfanyl]-*N*-(4-fluorophenyl)acetamide

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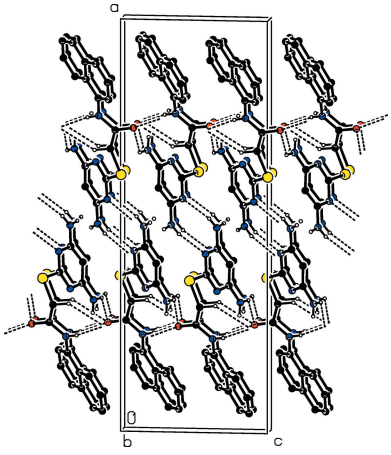
CCDC references: 1529608; 1529607

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

The title compounds, $C_{16}H_{15}N_5OS$, (I), and $C_{12}H_{12}FN_5OS$, (II), are [(diaminopyrimidine)sulfanyl]acetamide derivatives. In (I), the pyrimidine ring is inclined to the naphthalene ring system by 55.5 (1)°, while in (II), the pyrimidine ring is inclined to the benzene ring by 58.93 (8)°. In (II), there is an intramolecular N—H···N hydrogen bond and a short C—H···O contact. In the crystals of (I) and (II), molecules are linked by pairs of N—H···N hydrogen bonds, forming inversion dimers with $R_2^2(8)$ ring motifs. In the crystal of (I), the dimers are linked by bifurcated N—H···(O,O) and C—H···O hydrogen bonds, forming layers parallel to (100). In the crystal of (II), the dimers are linked by N—H···O hydrogen bonds, also forming layers parallel to (100). The layers are linked by C—H···F hydrogen bonds, forming a three-dimensional architecture.

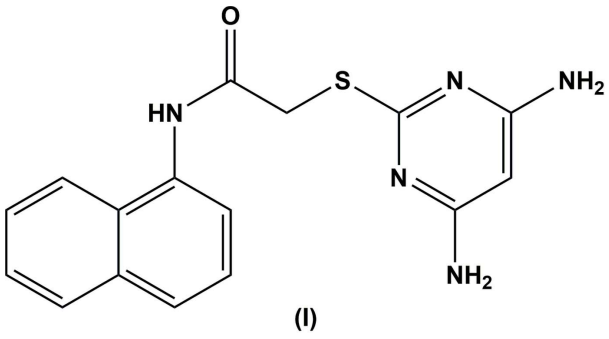
1. Chemical context

As a result of the innate ability of bacteria to develop resistance to available antibiotics, there is a critical need to develop new agents to treat more strains that are resilient. Several classes of diaminopyrimidines have been reported as new therapeutic agents. Derivatives of diaminopyrimidines also exhibit anti-cancer activity (Xu *et al.*, 2010), immune suppressant activity (Blumenkopf *et al.*, 2002), hair-growth-stimulant properties, anti-bacterial (Kandeel *et al.*, 1994) and potential anti-microbial properties (Holla *et al.*, 2006). They are also used as potential anti-AIDS agents (Nogueras *et al.*, 1993) and anti-viral agents (Hocková *et al.*, 2004). In this connection, the title 4,6-diaminopyrimidine-based analogues have been synthesized as potential antiviral agents against dengue for targeting NS2B/NS3 protease.

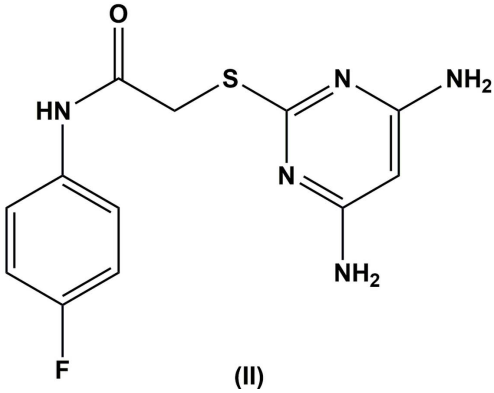


2. Structural commentary

The molecular structure of compound (I) is shown in Fig. 1. The pyrimidine ring is twisted with respect to the thioacetamide unit with the N1—C11—C12—S1 torsion angle being 140.88 (18)°. The pyrimidine ring (C13–C16/N2/N3) makes a dihedral angle of 55.5 (1)° with the naphthalene ring system (C1–C10). The amine nitrogen atoms, N4 and N5, deviate by 0.0235 and 0.0291 Å, respectively, from the plane of the pyrimidine ring.



(I)



(II)

The molecular structure of compound (II) is shown in Fig. 2. Here, the pyrimidine ring is twisted with respect to the thioacetamide unit with the N1—C7—C8—S1 torsion angle being −82.44 (14)°. The pyrimidine ring (C9–C12/N2/N3) makes a dihedral angle of 58.93 (8)° with the benzene ring (C1–C6). The amine nitrogen atoms, N4 and N5, deviate by 0.0247 and 0.0564 Å, respectively, from the pyrimidine ring. In compound (II), there is an intramolecular N—H···N hydrogen bond and a short C—H···O interaction present (Table 2 and Fig. 2).

3. Supramolecular features

In the crystal of compound (I), molecules are linked by pairs of N5—H5*A*···N3^{i} hydrogen bonds, forming inversion dimers with an $R_2^2(8)$ ring motif (Table 1 and Fig. 3). The dimers are linked by bifurcated N—H···(O,O) and C—H···O hydrogen bonds, forming layers parallel to the *bc* plane (Table 1 and Fig. 3).

In the crystal of compound (II), inversion dimers, with an $R_2^2(8)$, ring motif, are also formed *via* pairs of N5—H5*A*···N2^{i} hydrogen bonds (Table 2 and Fig. 4). This time the dimers are linked by N—H···O hydrogen bonds and also form layers parallel to the *bc* plane (Table 2 and Fig. 4). The layers are linked by C—H···F hydrogen bonds, forming a three-dimensional architecture (Table 2 and Fig. 4).

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5*A*···N3^{i}	0.86	2.27	3.110 (4)	167
N1—H1*A*···O1^{ii}	0.86	2.05	2.890 (3)	165
N4—H4*B*···O1^{iii}	0.86	2.36	2.964 (3)	127
C12—H12*A*···O1^{ii}	0.97	2.58	3.408 (3)	143

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N3	0.86	2.25	2.990 (2)	145
C3—H3···O1	0.93	2.31	2.903 (2)	121
N5—H5*A*···N2^{i}	0.86	2.29	3.139 (2)	169
N4—H4*A*···O1^{ii}	0.86	2.23	2.9852 (18)	146
C2—H2···F1^{iii}	0.93	2.48	3.404 (3)	172

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

4. Database survey

A search of the Cambridge Structural Database (Version 5.37, update May 2016; Groom *et al.*, 2016) for 2-(pyrimidin-2-yl)-*N*-phenylacetamide yielded only five hits. They include two 4,6-dimethylpyrimidine analogues *viz.* 2-(4,6-dimethylpyrimidin-2-ylsulfanyl)-*N*-phenyl acetamide (DIWXAJ; Gao

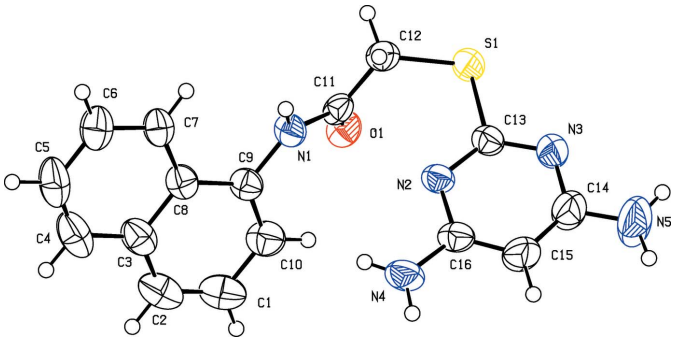


Figure 1
The molecular structure of compound (I), showing the atom labelling and displacement ellipsoids drawn at the 50% probability level.

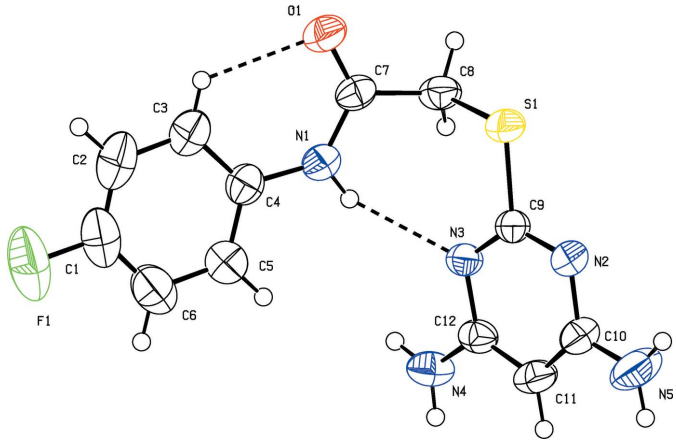


Figure 2
The molecular structure of compound (II), showing the atom labelling and displacement ellipsoids drawn at the 50% probability level.

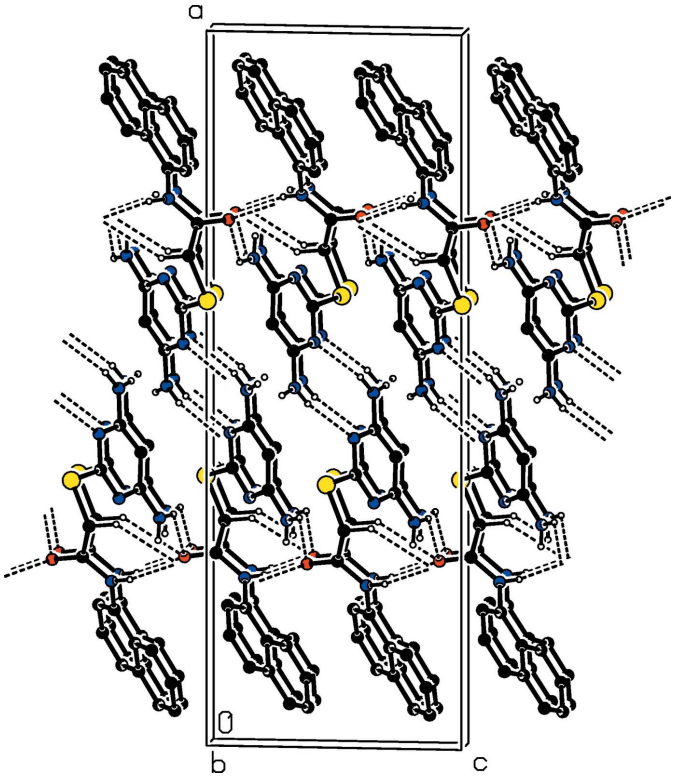


Figure 3
A view along the *b* axis, of the crystal packing of compound (I). Hydrogen bonds are shown as dashed lines (see Table 1). For clarity, only the NH and NH_2 hydrogens and the C-bound H atoms involved in hydrogen bonding have been included.

et al., 2008) and *N*-(2-chlorophenyl)-2-(4,6-dimethylpyrimidin-2-ylsulfanyl)acetamide (QOTQEW; Li *et al.*, 2009), and three 4,6-diaminopyrimidine compounds *viz.* 2-[(4,6-diaminopyrimidin-2-yl)sulfanyl]-*N*-2-methylphenyl)acetamide (GOKWIO; Subasri *et al.*, 2014), 2-[(4,6-diaminopyrimidin-2-yl)sulfanyl]-*N*-(3-nitrophenyl)acetamide (Subasri *et al.*, 2014) and 2-[(4,6-diaminopyrimidin-2-yl)sulfanyl]-*N*-(2-chlorophenyl)acetamide (Subasri *et al.*, 2014).

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, potassium hydroxide (0.2 g, 3.52 mmol) was added and the mixture refluxed for 30 min. Then 3.52 mmol of 2-chloro-*N*-(naphthalen-1-yl)acetamide was added and the mixture refluxed for 2.5 h. On completion of the reaction (monitored by TLC), the ethanol was evaporated *in vacuo* and cold water was added. The precipitate that formed was filtered and dried to give compound (I) as a crystalline powder (yield 92%).

Compound (II): To a solution of 4,6-diamino-pyrimidine-2-thiol (0.5 g, 3.52 mmol) in 25 ml of ethanol, potassium hydroxide (0.2 g, 3.52 mmol) was added and the mixture refluxed for 30 min. Then 3.52 mmol of 2-chloro-*N*-(4-fluorophenyl)acetamide was added and the mixture refluxed for 4 h. On completion of the reaction (monitored by TLC), ethanol was evaporated in vacuo and cold water was added and the precipitate formed was filtered and dried to give compound (II) as a crystalline powder (yield 88%).

Colourless block-like crystals were obtained by slow evaporation of a solution in CH_3OH for compound (I) and $C_4H_8O_2$ for compound (II).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. For both compounds the hydrogen atoms were placed in calculated positions and refined as

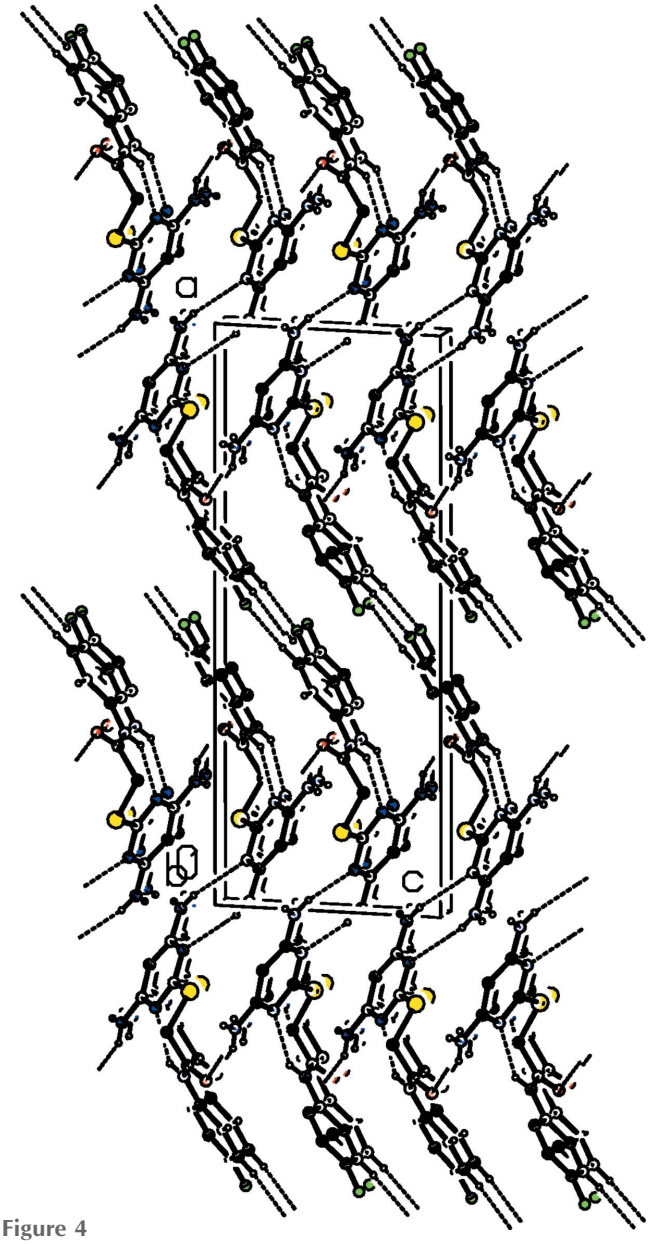


Figure 4
The crystal packing of compound (II) viewed along the *b* axis. Hydrogen bonds are shown as dashed lines (see Table 2). For clarity, only the NH and NH_2 hydrogens and the C-bound H atoms involved in hydrogen bonding have been included.

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{16}H_{15}N_5OS$	$C_{12}H_{12}FN_5OS$
M_r	325.39	293.33
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$
Temperature (K)	293	293
a, b, c (Å)	25.1895 (16), 6.9411 (4), 8.9697 (6)	21.7358 (7), 7.3726 (3), 8.4487 (3)
β (°)	90.943 (4)	93.092 (1)
V (Å^3)	1568.08 (17)	1351.93 (9)
Z	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.22	0.25
Crystal size (mm)	0.30 × 0.25 × 0.20	0.31 × 0.25 × 0.20
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}	0.752, 0.831	0.652, 0.753
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	14522, 3849, 2095	12316, 3312, 2829
R_{int}	0.063	0.025
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.669	0.667
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.052, 0.153, 0.98	0.037, 0.109, 1.05
No. of reflections	3849	3312
No. of parameters	208	181
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.38, −0.23	0.22, −0.22

Computer programs: *APEX2* and *SAINT* (Bruker, 2008), *SHELXS97* (Sheldrick, 2008), *SHELXL2016* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

riding: C—H = 0.93–0.97 Å, N—H = 0.86 Å with U_{iso}(H) = 1.2U_{eq}(N,C).

Acknowledgements

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supporting information

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Crystal structures of 2-[(4,6-diaminopyrimidin-2-yl)sulfanyl]-*N*-(naphthalen-1-yl)acetamide and 2-[(4,6-diaminopyrimidin-2-yl)sulfanyl]-*N*-(4-fluorophenyl)-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: *SHELXL2016* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009). Software used to prepare material for publication: *SHELXL2016* (Sheldrick, 2015) and *PLATON* (Spek, 2009) for (I); *SHELXL2016*/4 (Sheldrick, 2015) and *PLATON* (Spek, 2009) for (II).

(I) 2-[(4,6-Diaminopyrimidin-2-yl)sulfanyl]-*N*-(naphthalen-1-yl)acetamide

Crystal data

$C_{16}H_{15}N_5OS$
M_r = 325.39
Monoclinic, $P2_1/c$
a = 25.1895 (16) Å
b = 6.9411 (4) Å
c = 8.9697 (6) Å
β = 90.943 (4)°
V = 1568.08 (17) Å^3
Z = 4
$F(000)$ = 680
D_x = 1.378 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3849 reflections
θ = 1.6–28.4°
μ = 0.22 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} = 0.752, T_{max} = 0.831
14522 measured reflections
3849 independent reflections
2095 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 28.4°, θ_{min} = 1.6°
h = −33→33
k = −7→9
l = −11→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.153
S = 0.98
3849 reflections
208 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0671P)^2 + 0.3358P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ 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}
N5	0.49526 (12)	−0.2169 (5)	0.6491 (4)	0.1242 (15)
H5A	0.517831	−0.151383	0.599131	0.149*
H5B	0.505205	−0.319772	0.695198	0.149*
C1	0.15258 (14)	−0.2816 (4)	0.5842 (3)	0.0706 (9)
H1	0.156295	−0.401205	0.538876	0.085*
C2	0.10801 (13)	−0.2407 (5)	0.6613 (3)	0.0696 (9)
H2	0.081326	−0.332820	0.667315	0.084*
C3	0.10145 (10)	−0.0625 (4)	0.7317 (3)	0.0562 (7)
C4	0.05553 (12)	−0.0167 (6)	0.8124 (3)	0.0725 (9)
H4	0.028614	−0.107749	0.819483	0.087*
C5	0.04960 (12)	0.1557 (6)	0.8794 (3)	0.0740 (9)
H5	0.019065	0.181697	0.932778	0.089*
C6	0.08929 (11)	0.2952 (5)	0.8688 (3)	0.0634 (8)
H6	0.085036	0.413896	0.915302	0.076*
C7	0.13418 (10)	0.2591 (4)	0.7911 (3)	0.0501 (6)
H7	0.160100	0.354018	0.784284	0.060*
C8	0.14195 (9)	0.0796 (4)	0.7205 (2)	0.0452 (6)
C9	0.18804 (10)	0.0334 (4)	0.6386 (2)	0.0432 (6)
C10	0.19288 (11)	−0.1441 (4)	0.5729 (3)	0.0576 (7)
H10	0.223334	−0.173379	0.520215	0.069*
C11	0.26297 (9)	0.1951 (3)	0.5182 (2)	0.0413 (6)
C12	0.30711 (10)	0.3383 (4)	0.5478 (3)	0.0483 (6)
H12A	0.310700	0.357511	0.654569	0.058*
H12B	0.297111	0.460871	0.503592	0.058*
C13	0.38351 (10)	0.0615 (3)	0.5853 (2)	0.0422 (6)
C14	0.44443 (12)	−0.1587 (5)	0.6552 (3)	0.0688 (8)
C15	0.40599 (12)	−0.2535 (4)	0.7352 (3)	0.0668 (8)
H15	0.414154	−0.364339	0.789169	0.080*
C16	0.35572 (11)	−0.1801 (4)	0.7331 (3)	0.0481 (6)
N1	0.22916 (8)	0.1726 (3)	0.6311 (2)	0.0438 (5)
H1A	0.232751	0.249634	0.705663	0.053*
N2	0.34334 (7)	−0.0183 (3)	0.6559 (2)	0.0427 (5)
N3	0.43337 (8)	0.0052 (3)	0.5783 (2)	0.0540 (6)
N4	0.31453 (10)	−0.2587 (3)	0.8082 (2)	0.0607 (6)

H4A	0.283743	−0.205232	0.803960	0.073*
H4B	0.319286	−0.361782	0.859841	0.073*
O1	0.25760 (7)	0.1132 (3)	0.39688 (16)	0.0502 (5)
S1	0.37019 (3)	0.26816 (10)	0.47685 (8)	0.0554 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N5	0.0625 (18)	0.132 (3)	0.179 (3)	0.0403 (19)	0.033 (2)	0.099 (3)
C1	0.092 (2)	0.0530 (19)	0.0668 (19)	−0.0148 (18)	0.0000 (17)	−0.0022 (14)
C2	0.077 (2)	0.064 (2)	0.0671 (19)	−0.0257 (17)	−0.0050 (16)	0.0057 (15)
C3	0.0522 (17)	0.065 (2)	0.0511 (14)	−0.0107 (14)	0.0013 (12)	0.0105 (13)
C4	0.0508 (18)	0.102 (3)	0.0654 (18)	−0.0192 (18)	0.0071 (14)	0.0168 (18)
C5	0.0437 (17)	0.113 (3)	0.0657 (18)	0.0019 (19)	0.0160 (13)	0.008 (2)
C6	0.0519 (17)	0.082 (2)	0.0570 (16)	0.0086 (16)	0.0112 (13)	−0.0039 (15)
C7	0.0420 (14)	0.0617 (18)	0.0467 (14)	0.0001 (13)	0.0078 (11)	−0.0006 (12)
C8	0.0440 (14)	0.0538 (16)	0.0380 (12)	−0.0021 (12)	0.0010 (10)	0.0044 (11)
C9	0.0475 (14)	0.0455 (15)	0.0370 (11)	−0.0025 (12)	0.0051 (10)	0.0015 (10)
C10	0.0686 (19)	0.0518 (17)	0.0526 (15)	−0.0014 (15)	0.0095 (13)	−0.0034 (13)
C11	0.0460 (14)	0.0401 (14)	0.0383 (12)	0.0119 (11)	0.0109 (10)	0.0039 (10)
C12	0.0532 (16)	0.0384 (14)	0.0538 (14)	0.0032 (12)	0.0137 (12)	0.0049 (11)
C13	0.0452 (14)	0.0398 (14)	0.0417 (12)	−0.0040 (12)	0.0053 (10)	0.0008 (10)
C14	0.0571 (19)	0.070 (2)	0.080 (2)	0.0153 (16)	0.0118 (15)	0.0257 (17)
C15	0.069 (2)	0.0556 (19)	0.0763 (19)	0.0123 (16)	0.0136 (16)	0.0266 (15)
C16	0.0615 (17)	0.0387 (15)	0.0444 (13)	−0.0024 (13)	0.0094 (12)	0.0017 (11)
N1	0.0465 (12)	0.0458 (12)	0.0395 (10)	−0.0020 (10)	0.0129 (8)	−0.0056 (9)
N2	0.0477 (12)	0.0369 (12)	0.0437 (10)	−0.0037 (9)	0.0069 (9)	0.0039 (9)
N3	0.0437 (13)	0.0538 (14)	0.0646 (13)	0.0023 (11)	0.0074 (10)	0.0137 (11)
N4	0.0713 (16)	0.0460 (14)	0.0656 (14)	−0.0062 (12)	0.0217 (12)	0.0134 (11)
O1	0.0575 (11)	0.0562 (11)	0.0373 (9)	0.0086 (9)	0.0110 (7)	−0.0004 (8)
S1	0.0494 (4)	0.0524 (5)	0.0651 (4)	0.0038 (3)	0.0200 (3)	0.0208 (3)

Geometric parameters (Å, º)

N5—C14	1.345 (4)	C9—N1	1.419 (3)
N5—H5A	0.8600	C10—H10	0.9300
N5—H5B	0.8600	C11—O1	1.234 (3)
C1—C2	1.358 (4)	C11—N1	1.343 (3)
C1—C10	1.398 (4)	C11—C12	1.512 (4)
C1—H1	0.9300	C12—S1	1.789 (2)
C2—C3	1.400 (4)	C12—H12A	0.9700
C2—H2	0.9300	C12—H12B	0.9700
C3—C4	1.411 (4)	C13—N3	1.318 (3)
C3—C8	1.424 (4)	C13—N2	1.324 (3)
C4—C5	1.349 (5)	C13—S1	1.762 (2)
C4—H4	0.9300	C14—N3	1.357 (3)
C5—C6	1.396 (4)	C14—C15	1.382 (4)
C5—H5	0.9300	C15—C16	1.365 (4)

C6—C7	1.362 (3)	C15—H15	0.9300
C6—H6	0.9300	C16—N2	1.353 (3)
C7—C8	1.413 (3)	C16—N4	1.360 (3)
C7—H7	0.9300	N1—H1A	0.8600
C8—C9	1.421 (3)	N4—H4A	0.8600
C9—C10	1.372 (3)	N4—H4B	0.8600

C14—N5—H5A	120.0	C1—C10—H10	119.6
C14—N5—H5B	120.0	O1—C11—N1	123.4 (2)
H5A—N5—H5B	120.0	O1—C11—C12	121.8 (2)
C2—C1—C10	120.1 (3)	N1—C11—C12	114.7 (2)
C2—C1—H1	119.9	C11—C12—S1	114.45 (17)
C10—C1—H1	119.9	C11—C12—H12A	108.6
C1—C2—C3	121.3 (3)	S1—C12—H12A	108.6
C1—C2—H2	119.4	C11—C12—H12B	108.6
C3—C2—H2	119.4	S1—C12—H12B	108.6
C2—C3—C4	122.3 (3)	H12A—C12—H12B	107.6
C2—C3—C8	119.4 (3)	N3—C13—N2	129.4 (2)
C4—C3—C8	118.3 (3)	N3—C13—S1	112.83 (17)
C5—C4—C3	121.8 (3)	N2—C13—S1	117.67 (18)
C5—C4—H4	119.1	N5—C14—N3	114.8 (3)
C3—C4—H4	119.1	N5—C14—C15	123.6 (3)
C4—C5—C6	120.0 (3)	N3—C14—C15	121.6 (3)
C4—C5—H5	120.0	C16—C15—C14	118.2 (3)
C6—C5—H5	120.0	C16—C15—H15	120.9
C7—C6—C5	120.6 (3)	C14—C15—H15	120.9
C7—C6—H6	119.7	N2—C16—N4	114.5 (2)
C5—C6—H6	119.7	N2—C16—C15	121.5 (2)
C6—C7—C8	120.9 (3)	N4—C16—C15	123.9 (2)
C6—C7—H7	119.5	C11—N1—C9	126.0 (2)
C8—C7—H7	119.5	C11—N1—H1A	117.0
C7—C8—C9	123.4 (2)	C9—N1—H1A	117.0
C7—C8—C3	118.4 (2)	C13—N2—C16	114.9 (2)
C9—C8—C3	118.2 (2)	C13—N3—C14	114.3 (2)
C10—C9—N1	121.4 (2)	C16—N4—H4A	120.0
C10—C9—C8	120.2 (2)	C16—N4—H4B	120.0
N1—C9—C8	118.3 (2)	H4A—N4—H4B	120.0
C9—C10—C1	120.8 (3)	C13—S1—C12	100.80 (11)
C9—C10—H10	119.6		

C10—C1—C2—C3	−0.5 (5)	O1—C11—C12—S1	−42.3 (3)
C1—C2—C3—C4	180.0 (3)	N1—C11—C12—S1	140.88 (18)
C1—C2—C3—C8	1.0 (4)	N5—C14—C15—C16	179.9 (3)
C2—C3—C4—C5	−179.8 (3)	N3—C14—C15—C16	1.3 (5)
C8—C3—C4—C5	−0.8 (4)	C14—C15—C16—N2	−0.4 (4)
C3—C4—C5—C6	0.7 (5)	C14—C15—C16—N4	−179.3 (3)
C4—C5—C6—C7	0.0 (5)	O1—C11—N1—C9	10.4 (4)
C5—C6—C7—C8	−0.6 (4)	C12—C11—N1—C9	−172.8 (2)

C6—C7—C8—C9	−179.7 (2)	C10—C9—N1—C11	31.7 (4)
C6—C7—C8—C3	0.5 (4)	C8—C9—N1—C11	−150.3 (2)
C2—C3—C8—C7	179.3 (2)	N3—C13—N2—C16	0.8 (4)
C4—C3—C8—C7	0.2 (4)	S1—C13—N2—C16	177.84 (17)
C2—C3—C8—C9	−0.6 (4)	N4—C16—N2—C13	178.5 (2)
C4—C3—C8—C9	−179.7 (2)	C15—C16—N2—C13	−0.5 (3)
C7—C8—C9—C10	−180.0 (2)	N2—C13—N3—C14	0.0 (4)
C3—C8—C9—C10	−0.1 (3)	S1—C13—N3—C14	−177.2 (2)
C7—C8—C9—N1	2.1 (3)	N5—C14—N3—C13	−179.8 (3)
C3—C8—C9—N1	−178.1 (2)	C15—C14—N3—C13	−1.1 (4)
N1—C9—C10—C1	178.4 (2)	N3—C13—S1—C12	−165.84 (19)
C8—C9—C10—C1	0.6 (4)	N2—C13—S1—C12	16.6 (2)
C2—C1—C10—C9	−0.2 (4)	C11—C12—S1—C13	−64.63 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5*A*···N3^{i}	0.86	2.27	3.110 (4)	167
N1—H1*A*···O1^{ii}	0.86	2.05	2.890 (3)	165
N4—H4*B*···O1^{iii}	0.86	2.36	2.964 (3)	127
C12—H12*A*···O1^{ii}	0.97	2.58	3.408 (3)	143

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

(II) 2-[(4,6-Diaminopyrimidin-2-yl)sulfanyl]-*N*-(4-fluorophenyl)acetamide

Crystal data

$C_{12}H_{12}FN_5OS$
M_r = 293.33
Monoclinic, $P2_1/c$
a = 21.7358 (7) Å
b = 7.3726 (3) Å
c = 8.4487 (3) Å
β = 93.092 (1)°
V = 1351.93 (9) Å^3
Z = 4
$F(000)$ = 608
D_x = 1.441 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3312 reflections
θ = 1.9–28.3°
μ = 0.25 mm^{-1}
T = 293 K
Block, colourless
0.31 × 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} = 0.652, T_{max} = 0.753
12316 measured reflections
3312 independent reflections
2829 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 28.3°, θ_{min} = 1.9°
h = −28→28
k = −5→9
l = −7→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.109
S = 1.05
3312 reflections
181 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 0.3099P]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} = 0.001$
H-atom parameters constrained	$\Delta\rho_{max} = 0.22$ e Å^{-3}
	$\Delta\rho_{min} = -0.22$ 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}
C1	0.43036 (9)	−0.1374 (4)	0.4152 (3)	0.0777 (6)
C2	0.42379 (9)	0.0399 (4)	0.3738 (3)	0.0824 (6)
H2	0.450358	0.092466	0.304272	0.099*
C3	0.37714 (8)	0.1422 (3)	0.4359 (2)	0.0658 (4)
H3	0.372002	0.263568	0.408377	0.079*
C4	0.33831 (6)	0.0600 (2)	0.53966 (17)	0.0480 (3)
C5	0.34659 (8)	−0.1211 (2)	0.5791 (2)	0.0605 (4)
H5	0.320667	−0.175666	0.649109	0.073*
C6	0.39282 (10)	−0.2211 (3)	0.5158 (3)	0.0746 (5)
H6	0.398185	−0.342957	0.541273	0.089*
C7	0.26854 (6)	0.32063 (19)	0.58470 (16)	0.0432 (3)
C8	0.21206 (7)	0.36589 (19)	0.67409 (17)	0.0482 (3)
H8A	0.210250	0.496199	0.688963	0.058*
H8B	0.216105	0.310216	0.778152	0.058*
C9	0.13013 (6)	0.07360 (17)	0.65615 (14)	0.0378 (3)
C10	0.06207 (7)	−0.1564 (2)	0.67975 (18)	0.0501 (3)
C11	0.10494 (7)	−0.2466 (2)	0.77884 (18)	0.0505 (3)
H11	0.095821	−0.358065	0.823502	0.061*
C12	0.16170 (7)	−0.16497 (18)	0.80890 (15)	0.0415 (3)
F1	0.47632 (7)	−0.2347 (3)	0.3527 (2)	0.1187 (6)
N1	0.28896 (5)	0.15094 (17)	0.60807 (15)	0.0480 (3)
H1	0.269193	0.087989	0.674351	0.058*
N2	0.07407 (5)	0.00964 (16)	0.61864 (14)	0.0452 (3)
N3	0.17559 (5)	−0.00336 (14)	0.74173 (12)	0.0390 (2)
N4	0.20765 (7)	−0.23810 (17)	0.90067 (16)	0.0546 (3)
H4A	0.242409	−0.182765	0.912926	0.066*
H4B	0.202291	−0.340228	0.947136	0.066*
N5	0.00588 (8)	−0.2236 (2)	0.6394 (2)	0.0815 (5)
H5A	−0.019470	−0.162080	0.579047	0.098*
H5B	−0.004701	−0.328149	0.674068	0.098*
O1	0.29125 (5)	0.43187 (15)	0.49776 (14)	0.0597 (3)
S1	0.14082 (2)	0.29101 (5)	0.57535 (5)	0.05097 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0526 (10)	0.0988 (16)	0.0819 (13)	0.0139 (10)	0.0035 (9)	−0.0235 (12)
C2	0.0581 (11)	0.1083 (18)	0.0830 (13)	−0.0068 (11)	0.0248 (10)	−0.0037 (13)
C3	0.0548 (9)	0.0710 (11)	0.0728 (11)	−0.0055 (8)	0.0142 (8)	0.0061 (9)
C4	0.0429 (7)	0.0532 (8)	0.0478 (7)	−0.0038 (6)	−0.0002 (6)	−0.0007 (6)
C5	0.0577 (9)	0.0563 (10)	0.0677 (10)	0.0023 (7)	0.0049 (7)	0.0015 (8)
C6	0.0682 (11)	0.0665 (12)	0.0885 (14)	0.0138 (9)	−0.0011 (10)	−0.0103 (10)
C7	0.0453 (7)	0.0420 (7)	0.0418 (6)	−0.0102 (5)	−0.0029 (5)	0.0014 (5)
C8	0.0597 (8)	0.0347 (7)	0.0508 (7)	−0.0064 (6)	0.0081 (6)	−0.0023 (6)
C9	0.0447 (6)	0.0333 (6)	0.0366 (6)	0.0004 (5)	0.0123 (5)	−0.0009 (5)
C10	0.0498 (8)	0.0469 (8)	0.0543 (8)	−0.0080 (6)	0.0102 (6)	0.0061 (6)
C11	0.0604 (9)	0.0395 (7)	0.0521 (8)	−0.0086 (6)	0.0088 (7)	0.0089 (6)
C12	0.0551 (7)	0.0340 (6)	0.0361 (6)	0.0012 (5)	0.0082 (5)	−0.0014 (5)
F1	0.0774 (9)	0.1484 (14)	0.1323 (13)	0.0357 (9)	0.0230 (8)	−0.0415 (11)
N1	0.0499 (6)	0.0442 (6)	0.0508 (6)	−0.0030 (5)	0.0106 (5)	0.0070 (5)
N2	0.0442 (6)	0.0422 (6)	0.0499 (6)	−0.0029 (5)	0.0079 (5)	0.0056 (5)
N3	0.0467 (6)	0.0320 (5)	0.0390 (5)	−0.0001 (4)	0.0081 (4)	0.0003 (4)
N4	0.0677 (8)	0.0397 (6)	0.0557 (7)	−0.0015 (6)	−0.0045 (6)	0.0089 (5)
N5	0.0586 (9)	0.0747 (11)	0.1098 (14)	−0.0267 (8)	−0.0091 (8)	0.0363 (10)
O1	0.0564 (6)	0.0514 (6)	0.0719 (7)	−0.0071 (5)	0.0085 (5)	0.0188 (5)
S1	0.0472 (2)	0.0404 (2)	0.0655 (2)	−0.00120 (14)	0.00437 (16)	0.01690 (16)

Geometric parameters (Å, º)

C1—C6	1.358 (3)	C8—H8B	0.9700
C1—C2	1.359 (3)	C9—N3	1.3207 (17)
C1—F1	1.359 (2)	C9—N2	1.3288 (17)
C2—C3	1.389 (3)	C9—S1	1.7625 (13)
C2—H2	0.9300	C10—N5	1.345 (2)
C3—C4	1.388 (2)	C10—N2	1.3594 (18)
C3—H3	0.9300	C10—C11	1.388 (2)
C4—C5	1.386 (2)	C11—C12	1.384 (2)
C4—N1	1.4144 (19)	C11—H11	0.9300
C5—C6	1.378 (3)	C12—N4	1.3438 (19)
C5—H5	0.9300	C12—N3	1.3606 (17)
C6—H6	0.9300	N1—H1	0.8600
C7—O1	1.2227 (16)	N4—H4A	0.8600
C7—N1	1.3386 (19)	N4—H4B	0.8600
C7—C8	1.513 (2)	N5—H5A	0.8600
C8—S1	1.8054 (15)	N5—H5B	0.8600
C8—H8A	0.9700		
C6—C1—C2	122.69 (18)	H8A—C8—H8B	107.7
C6—C1—F1	118.8 (2)	N3—C9—N2	128.87 (12)
C2—C1—F1	118.5 (2)	N3—C9—S1	119.50 (10)
C1—C2—C3	119.57 (19)	N2—C9—S1	111.64 (10)

C1—C2—H2	120.2	N5—C10—N2	115.21 (15)
C3—C2—H2	120.2	N5—C10—C11	123.17 (14)
C4—C3—C2	118.9 (2)	N2—C10—C11	121.61 (14)
C4—C3—H3	120.6	C12—C11—C10	117.71 (13)
C2—C3—H3	120.6	C12—C11—H11	121.1
C5—C4—C3	119.78 (16)	C10—C11—H11	121.1
C5—C4—N1	116.72 (14)	N4—C12—N3	114.66 (13)
C3—C4—N1	123.49 (16)	N4—C12—C11	123.98 (13)
C6—C5—C4	120.67 (18)	N3—C12—C11	121.34 (13)
C6—C5—H5	119.7	C7—N1—C4	129.45 (13)
C4—C5—H5	119.7	C7—N1—H1	115.3
C1—C6—C5	118.4 (2)	C4—N1—H1	115.3
C1—C6—H6	120.8	C9—N2—C10	114.92 (12)
C5—C6—H6	120.8	C9—N3—C12	115.35 (11)
O1—C7—N1	125.03 (14)	C12—N4—H4A	120.0
O1—C7—C8	121.11 (13)	C12—N4—H4B	120.0
N1—C7—C8	113.84 (12)	H4A—N4—H4B	120.0
C7—C8—S1	113.63 (10)	C10—N5—H5A	120.0
C7—C8—H8A	108.8	C10—N5—H5B	120.0
S1—C8—H8A	108.8	H5A—N5—H5B	120.0
C7—C8—H8B	108.8	C9—S1—C8	103.11 (7)
S1—C8—H8B	108.8		

C6—C1—C2—C3	0.2 (3)	O1—C7—N1—C4	−1.5 (2)
F1—C1—C2—C3	179.92 (18)	C8—C7—N1—C4	177.03 (13)
C1—C2—C3—C4	0.2 (3)	C5—C4—N1—C7	−176.10 (15)
C2—C3—C4—C5	−0.1 (3)	C3—C4—N1—C7	3.0 (2)
C2—C3—C4—N1	−179.19 (16)	N3—C9—N2—C10	1.3 (2)
C3—C4—C5—C6	−0.3 (3)	S1—C9—N2—C10	−178.73 (10)
N1—C4—C5—C6	178.83 (15)	N5—C10—N2—C9	−178.64 (15)
C2—C1—C6—C5	−0.6 (3)	C11—C10—N2—C9	2.5 (2)
F1—C1—C6—C5	179.66 (18)	N2—C9—N3—C12	−4.68 (19)
C4—C5—C6—C1	0.7 (3)	S1—C9—N3—C12	175.34 (9)
O1—C7—C8—S1	96.18 (14)	N4—C12—N3—C9	−177.50 (12)
N1—C7—C8—S1	−82.44 (14)	C11—C12—N3—C9	4.35 (18)
N5—C10—C11—C12	178.71 (16)	N3—C9—S1—C8	−11.34 (11)
N2—C10—C11—C12	−2.5 (2)	N2—C9—S1—C8	168.68 (10)
C10—C11—C12—N4	−179.08 (14)	C7—C8—S1—C9	91.88 (11)
C10—C11—C12—N3	−1.1 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N3	0.86	2.25	2.990 (2)	145
C3—H3···O1	0.93	2.31	2.903 (2)	121
N5—H5*A*···N2^{i}	0.86	2.29	3.139 (2)	169

N4—H4*A*···O1^{ii}	0.86	2.23	2.9852 (18)	146
C2—H2···F1^{iii}	0.93	2.48	3.404 (3)	172

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