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Crystal structure of 4-allylsulfanyl-1*H*-pyrazolo[3,4-*d*]pyrimidine

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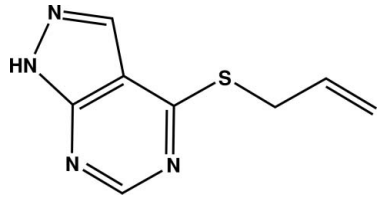
In the title compound, $C_8H_8N_4S$, the pyrazolo[3,4-*d*]pyrimidine ring system is essentially planar, with a maximum deviation from the mean plane of 0.025 (3) Å. The allyl group is disordered over two sites in a 0.512 (6):0.488 (6) ratio. In the crystal, molecules are linked by pairs of N—H···N hydrogen bonds, forming inversion dimers with an $R_2^2(8)$ graph-set motif.

Keywords: crystal structure; pyrazolopyrimidine; thiopyrazolopyrimidine; disorder.

CCDC reference: 1018090

1. Related literature

Antiviral, antimycobacterial and anticancer properties of pyrazolo[3,4-*d*]pyrimidine-4(5*H*)-thione derivatives are described, respectively, by Yuan *et al.* (2013), Ballell *et al.* (2007) and Rashad *et al.* (2011), and Alsubari *et al.* (2011). A similar structure, namely 4-benzylsulfanyl-1*H*-pyrazolo[3,4-*d*]pyrimidine, is reported by El Fal *et al.* (2013).



2. Experimental

2.1. Crystal data

$C_8H_8N_4S$ M_r = 192.24
Orthorhombic, *Pbcn*
a = 18.537 (6) Å
b = 5.1997 (17) Å
c = 19.059 (7) Å
V = 1837.0 (11) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 296 K
0.39 × 0.34 × 0.29 mm

2.2. Data collection

Bruker X8 APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.641, T_{max} = 0.746
20928 measured reflections
2189 independent reflections
1093 reflections with $I > 2\sigma(I)$
R_{int} = 0.068

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.181
S = 1.02
2189 reflections
128 parameters
6 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 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*N*···N2^{i}	0.86	2.09	2.940 (4)	172

Symmetry code: (i) $-x+1, -y+2, -z+2$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: IS5372).

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supporting information

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Crystal structure of 4-allylsulfanyl-1*H*-pyrazolo[3,4-*d*]pyrimidine

Mohammed El Fal, Youssef Ramli, El Mokhtar Essassi, Mohamed Saadi and Lahcen El Ammari

S1. Experimental

1*H*,5*H*-pyrazolo[3,4-*d*]pyrimidine-4-thione (0.5 g, 3.29 mmol), allyl bromide (0.5 ml, 5.70 mmol) and potassium carbonate (0.64 g, 4.8 mmol) with a catalytic amount of tetra-n-butylammonium bromide were stirred in DMF (15 ml) for 72 h. The solid obtained was removed by filtration and the solvent evaporated under vacuum. The solid product was purified by recrystallization from ethanol to afford yellow crystals in 55% yield.

S2. Refinement

The allyl group is disordered over two sites with refined occupancies of 0.512 (6) and 0.488 (6). For the disordered group, tight distance restraints of S—C = 1.753 (2) Å, C—C = 1.453 (2) Å and C═C = 1.287 (2) Å, and constraint of same displacement parameters were applied. The H atoms were located in a difference map and treated as riding with C—H = 0.93 Å (aromatic), C—H = 0.97 Å (methylene) and N—H = 0.86 Å (N–H), and with U_{iso}(H) = 1.2U_{eq}(C,N).

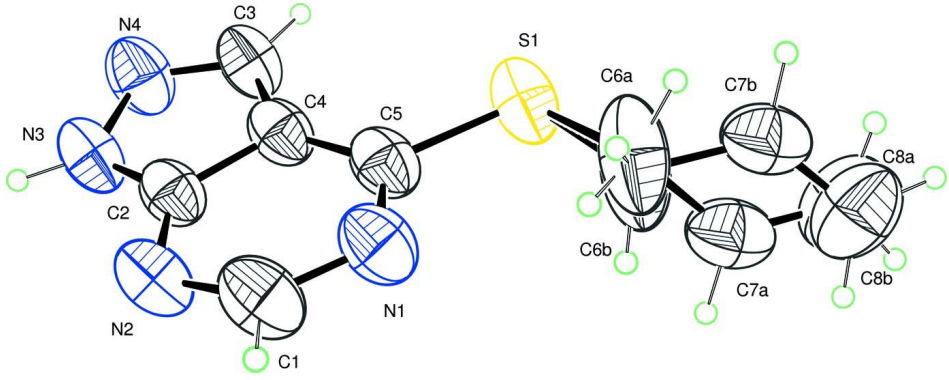

Figure 1

Molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles.

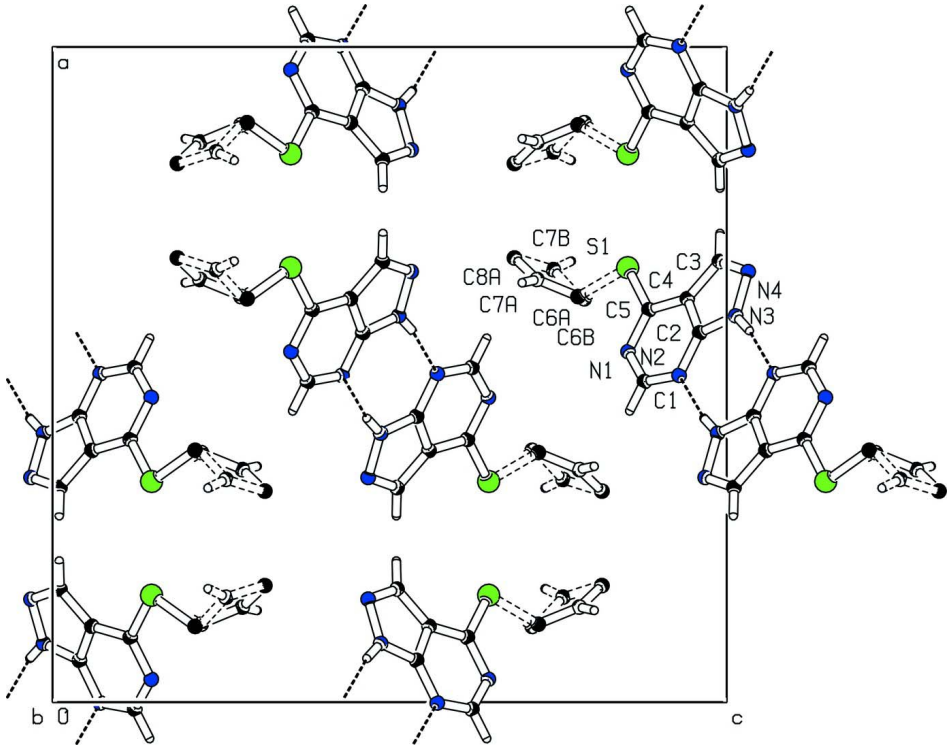

Figure 2

Packing diagram of the title compound viewed along the *b* axis, showing molecules linked through N3–H3N···N2 hydrogen bond (dashed lines).

4-Allylsulfanyl-1*H*-pyrazolo[3,4-*d*]pyrimidine

Crystal data

$C_8H_8N_4S$
M_r = 192.24
Orthorhombic, *Pbcn*
Hall symbol: -p 2n 2ab
a = 18.537 (6) Å
b = 5.1997 (17) Å
c = 19.059 (7) Å
V = 1837.0 (11) Å^3
Z = 8
F(000) = 800
D_x = 1.390 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 2189 reflections
θ = 2.4–27.9°
μ = 0.31 mm^{-1}
T = 296 K
Block, yellow
0.39 × 0.34 × 0.29 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.641, T_{max} = 0.746
20928 measured reflections
2189 independent reflections
1093 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
θ_{max} = 27.9°, θ_{min} = 2.4°
h = −15→24
k = −6→6
l = −24→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.181$
$S = 1.02$
2189 reflections
128 parameters
6 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
$w = 1/[\sigma^2(F_o^2) + (0.0784P)^2 + 0.5307P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}

Special details

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

Refinement. Refinement of F^2 against all reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on all data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1	0.66313 (5)	0.15662 (17)	0.85306 (5)	0.0890 (4)	
N1	0.53488 (15)	0.3882 (5)	0.85359 (15)	0.0819 (8)	
N2	0.49883 (12)	0.7352 (5)	0.92947 (15)	0.0759 (8)	
N3	0.58954 (12)	0.8446 (5)	1.01342 (15)	0.0760 (8)	
H3N	0.5667	0.9680	1.0337	0.091*	
N4	0.65714 (13)	0.7630 (6)	1.03189 (17)	0.0867 (9)	
C1	0.48931 (17)	0.5665 (7)	0.8781 (2)	0.0887 (10)	
H1	0.4446	0.5730	0.8561	0.106*	
C2	0.56370 (15)	0.7078 (5)	0.96002 (17)	0.0639 (8)	
C3	0.67252 (15)	0.5738 (7)	0.98923 (19)	0.0787 (9)	
H3	0.7154	0.4812	0.9902	0.094*	
C4	0.61592 (14)	0.5282 (5)	0.94180 (17)	0.0654 (8)	
C5	0.59890 (15)	0.3712 (5)	0.88464 (18)	0.0710 (9)	
C6A	0.6154 (5)	−0.011 (2)	0.7877 (4)	0.1188 (19)	0.512 (6)
H6A1	0.6140	−0.1915	0.7999	0.143*	0.512 (6)
H6A2	0.5660	0.0515	0.7869	0.143*	0.512 (6)
C7A	0.6458 (4)	0.0154 (16)	0.7178 (5)	0.0919 (15)	0.512 (6)
H7A	0.6385	0.1722	0.6954	0.110*	0.512 (6)
C8A	0.682 (3)	−0.155 (6)	0.6835 (9)	0.121 (3)	0.512 (6)
H8A1	0.6906	−0.3151	0.7035	0.145*	0.512 (6)
H8A2	0.6989	−0.1179	0.6388	0.145*	0.512 (6)
C6B	0.6188 (5)	0.019 (2)	0.7806 (4)	0.1188 (19)	0.488 (6)
H6B1	0.5749	−0.0628	0.7973	0.143*	0.488 (6)
H6B2	0.6046	0.1563	0.7491	0.143*	0.488 (6)
C7B	0.6598 (4)	−0.1692 (16)	0.7409 (5)	0.0919 (15)	0.488 (6)

H7B	0.6757	−0.3111	0.7663	0.110*	0.488 (6)
C8B	0.677 (3)	−0.168 (7)	0.6756 (10)	0.121 (3)	0.488 (6)
H8B1	0.6634	−0.0313	0.6470	0.145*	0.488 (6)
H8B2	0.7042	−0.3024	0.6569	0.145*	0.488 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0830 (6)	0.0671 (6)	0.1169 (9)	0.0018 (4)	0.0180 (5)	−0.0160 (5)
N1	0.0756 (16)	0.0624 (16)	0.108 (2)	−0.0024 (13)	−0.0034 (15)	−0.0048 (15)
N2	0.0599 (14)	0.0576 (15)	0.110 (2)	0.0017 (11)	−0.0054 (14)	−0.0014 (16)
N3	0.0589 (13)	0.0613 (15)	0.108 (2)	0.0089 (11)	0.0049 (14)	−0.0078 (15)
N4	0.0621 (15)	0.083 (2)	0.115 (2)	0.0106 (13)	−0.0057 (14)	−0.0153 (18)
C1	0.0683 (18)	0.073 (2)	0.124 (3)	−0.0002 (17)	−0.0086 (19)	0.000 (2)
C2	0.0604 (15)	0.0472 (15)	0.084 (2)	−0.0016 (12)	0.0065 (15)	0.0029 (15)
C3	0.0630 (17)	0.069 (2)	0.104 (3)	0.0116 (14)	0.0028 (17)	−0.009 (2)
C4	0.0584 (15)	0.0511 (16)	0.087 (2)	−0.0005 (12)	0.0069 (15)	0.0023 (16)
C5	0.0704 (18)	0.0502 (17)	0.092 (2)	−0.0041 (14)	0.0122 (17)	0.0058 (17)
C6A	0.103 (3)	0.098 (4)	0.155 (4)	−0.023 (3)	0.038 (3)	−0.054 (3)
C7A	0.094 (3)	0.076 (4)	0.106 (4)	−0.009 (3)	−0.019 (3)	0.018 (3)
C8A	0.102 (6)	0.167 (5)	0.093 (5)	−0.012 (4)	−0.007 (6)	−0.010 (5)
C6B	0.103 (3)	0.098 (4)	0.155 (4)	−0.023 (3)	0.038 (3)	−0.054 (3)
C7B	0.094 (3)	0.076 (4)	0.106 (4)	−0.009 (3)	−0.019 (3)	0.018 (3)
C8B	0.102 (6)	0.167 (5)	0.093 (5)	−0.012 (4)	−0.007 (6)	−0.010 (5)

Geometric parameters (Å, º)

S1—C5	1.739 (3)	C4—C5	1.397 (4)
S1—C6A	1.759 (2)	C6A—C7A	1.452 (2)
S1—C6B	1.759 (2)	C6A—H6A1	0.9700
N1—C5	1.329 (4)	C6A—H6A2	0.9700
N1—C1	1.339 (4)	C7A—C8A	1.287 (2)
N2—C1	1.326 (4)	C7A—H7A	0.9300
N2—C2	1.344 (4)	C8A—H8A1	0.9300
N3—C2	1.331 (4)	C8A—H8A2	0.9300
N3—N4	1.369 (3)	C6B—C7B	1.453 (2)
N3—H3N	0.8600	C6B—H6B1	0.9700
N4—C3	1.308 (4)	C6B—H6B2	0.9700
C1—H1	0.9300	C7B—C8B	1.287 (2)
C2—C4	1.389 (4)	C7B—H7B	0.9300
C3—C4	1.405 (4)	C8B—H8B1	0.9300
C3—H3	0.9300	C8B—H8B2	0.9300
C5—S1—C6A	102.6 (4)	C7A—C6A—H6A1	108.7
C5—S1—C6B	102.3 (4)	S1—C6A—H6A1	108.7
C5—N1—C1	117.0 (3)	C7A—C6A—H6A2	108.7
C1—N2—C2	111.6 (3)	S1—C6A—H6A2	108.7
C2—N3—N4	111.1 (3)	H6A1—C6A—H6A2	107.6

C2—N3—H3N	124.4	C8A—C7A—C6A	127.1 (11)
N4—N3—H3N	124.4	C8A—C7A—H7A	116.5
C3—N4—N3	105.8 (3)	C6A—C7A—H7A	116.5
N2—C1—N1	129.2 (3)	C7A—C8A—H8A1	120.0
N2—C1—H1	115.4	C7A—C8A—H8A2	120.0
N1—C1—H1	115.4	H8A1—C8A—H8A2	120.0
N3—C2—N2	126.7 (3)	C7B—C6B—S1	116.0 (7)
N3—C2—C4	107.4 (3)	C7B—C6B—H6B1	108.3
N2—C2—C4	125.9 (3)	S1—C6B—H6B1	108.3
N4—C3—C4	111.4 (3)	C7B—C6B—H6B2	108.3
N4—C3—H3	124.3	S1—C6B—H6B2	108.3
C4—C3—H3	124.3	H6B1—C6B—H6B2	107.4
C2—C4—C5	115.5 (3)	C8B—C7B—C6B	129.2 (10)
C2—C4—C3	104.2 (3)	C8B—C7B—H7B	115.4
C5—C4—C3	140.2 (3)	C6B—C7B—H7B	115.4
N1—C5—C4	120.7 (3)	C7B—C8B—H8B1	120.0
N1—C5—S1	120.0 (3)	C7B—C8B—H8B2	120.0
C4—C5—S1	119.3 (2)	H8B1—C8B—H8B2	120.0
C7A—C6A—S1	114.1 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*N*···N2^{i}	0.86	2.09	2.940 (4)	172

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+2.