

Monoclinic polymorph of 2-(pyrimidin-2-ylsulfanyl)acetic acid

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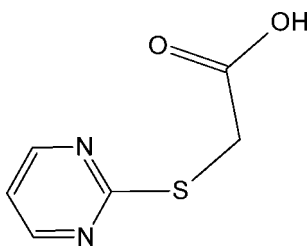
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.054; wR factor = 0.133; data-to-parameter ratio = 16.9.

The title compound, $\text{C}_6\text{H}_6\text{N}_2\text{O}_2\text{S}$, is a new polymorphic form of 2-(pyrimidin-2-ylsulfanyl)acetic acid. Unlike the previous orthorhombic polymorph [Pan & Chen (2009) *Acta Cryst. E* **65**, o652], the molecules are not planar: the aromatic ring makes an angle of 80.67 (17)° with the carboxyl plane. In the crystal, molecules are linked by $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds into chains along $[\bar{1}02]$.

Related literature

For the previously reported orthorhombic polymorph, see: Pan & Chen (2009).



Experimental

Crystal data

$\text{C}_6\text{H}_6\text{N}_2\text{O}_2\text{S}$

$M_r = 170.19$

Monoclinic, $P2_1/c$
 $a = 8.2617$ (3) Å
 $b = 10.3028$ (4) Å
 $c = 9.9289$ (3) Å
 $\beta = 119.845$ (2)°
 $V = 733.05$ (4) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.39$ mm⁻¹
 $T = 293$ K
 $0.40 \times 0.23 \times 0.20$ mm

Data collection

Bruker APEX CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 2000)
 $T_{\min} = 0.891$, $T_{\max} = 0.999$

14257 measured reflections
 1688 independent reflections
 1532 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.054$
 $wR(F^2) = 0.133$
 $S = 1.17$
 1688 reflections
 100 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.31$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.18$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O2}-\text{H2}\cdots\text{N2}^i$	0.74	1.97	2.700 (3)	166

Symmetry code: (i) $x - 1, -y + \frac{3}{2}, z - \frac{1}{2}$.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINTE* (Bruker, 2003); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5454).

References

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 Pan, J. X. & Chen, Q. W. (2009). *Acta Cryst. E* **65**, o652.
 Sheldrick, G. M. (2000). *SADABS*. University of Göttingen, Germany.
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supplementary materials

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Comment

In an attempt to synthesize low dimensional compounds with transition metal elements, crystals of a new monoclinic phase of the title compound (Fig. 1) were obtained. In the previously reported orthorhombic phase the molecules are planar and pack in layers, joined by a O—H...N intermolecular bond (Pan & Chen, 2009). In the new monoclinic phase the molecules are not planar, the C3—S1—C2—C1 torsion angle is 77.84 (18)°. There is a similar strong hydrogen bond (Table 1) as in the orthorhombic phase, but in the title compound this intermolecular bond groups the molecules in chains, that run along the [-102] direction.

Experimental

0.2 mmol of 2-(Pyrimidin-2-ylsulfanyl)acetic acid (98%) and 0.2 mmol of CuCl₂·2H₂O (99.0%) were dissolved in 20 ml of water plus 20 ml of ethanol. The solution was slightly warmed and left to evaporate for a few weeks. After that time, small yellowish single crystals were obtained.

Refinement

Hydrogen atoms bound to C atoms were positioned geometrically with C—H = 0.93–0.97 Å, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The coordinates of the H atom bound to O were freely refined with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

Figures

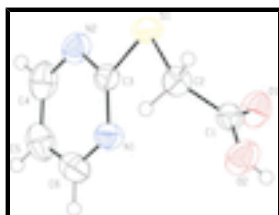


Fig. 1. ORTEP (Johnson, 1976) plot of the title compound. Displacement ellipsoids are drawn at the 50% level.

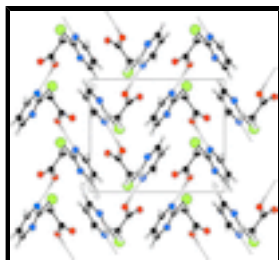


Fig. 2. Packing of the molecules in the unit cell showing the H-bonds as dashed lines.

2-(pyrimidin-2-ylsulfanyl)acetic acid

Crystal data

$C_6H_6N_2O_2S$	$F(000) = 352$
$M_r = 170.19$	$D_x = 1.542 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 8.2617 (3) \text{ \AA}$	Cell parameters from 6019 reflections
$b = 10.3028 (4) \text{ \AA}$	$\theta = 2.8\text{--}27.2^\circ$
$c = 9.9289 (3) \text{ \AA}$	$\mu = 0.39 \text{ mm}^{-1}$
$\beta = 119.845 (2)^\circ$	$T = 293 \text{ K}$
$V = 733.05 (4) \text{ \AA}^3$	Prism, yellow
$Z = 4$	$0.40 \times 0.23 \times 0.20 \text{ mm}$

Data collection

Bruker APEX CCD area-detector diffractometer	1688 independent reflections
Radiation source: fine-focus sealed tube graphite	1532 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.027$
Absorption correction: multi-scan (SADABS; Sheldrick, 2000)	$\theta_{\text{max}} = 27.6^\circ$, $\theta_{\text{min}} = 2.8^\circ$
$T_{\text{min}} = 0.891$, $T_{\text{max}} = 0.999$	$h = -10 \rightarrow 10$
14257 measured reflections	$k = -13 \rightarrow 13$
	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.054$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.133$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.17$	$w = 1/[\sigma^2(F_o^2) + (0.0619P)^2 + 0.3572P]$
1688 reflections	where $P = (F_o^2 + 2F_c^2)/3$
100 parameters	$(\Delta/\sigma)_{\text{max}} < 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.18 \text{ e \AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.79236 (8)	0.78200 (6)	0.43768 (7)	0.0451 (2)
O2	0.2618 (3)	0.7405 (2)	0.1323 (3)	0.0642 (6)
H2	0.2106	0.7051	0.0572	0.096*
O1	0.5054 (3)	0.63141 (18)	0.1564 (2)	0.0544 (5)
C1	0.4398 (3)	0.7107 (2)	0.2002 (3)	0.0383 (5)
C2	0.5444 (3)	0.7939 (2)	0.3434 (3)	0.0411 (5)
H2A	0.5048	0.7701	0.4171	0.049*
H2B	0.5094	0.8838	0.3146	0.049*
C3	0.8442 (3)	0.8782 (2)	0.3184 (2)	0.0358 (5)
N1	0.7074 (2)	0.92846 (19)	0.1883 (2)	0.0407 (4)
C6	0.7635 (4)	1.0039 (2)	0.1100 (3)	0.0495 (6)
H6	0.6732	1.0425	0.0185	0.059*
C5	0.9468 (4)	1.0271 (3)	0.1575 (3)	0.0544 (7)
H5	0.9820	1.0810	0.1013	0.065*
C4	1.0766 (4)	0.9677 (3)	0.2916 (3)	0.0536 (7)
H4	1.2028	0.9803	0.3261	0.064*
N2	1.0268 (3)	0.8926 (2)	0.3736 (2)	0.0459 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0382 (3)	0.0520 (4)	0.0360 (3)	-0.0017 (2)	0.0116 (2)	0.0089 (2)
O2	0.0394 (10)	0.0662 (13)	0.0664 (13)	0.0044 (8)	0.0108 (9)	-0.0106 (10)
O1	0.0514 (10)	0.0554 (11)	0.0595 (11)	-0.0035 (8)	0.0299 (9)	-0.0133 (9)
C1	0.0362 (11)	0.0383 (12)	0.0411 (12)	-0.0037 (8)	0.0197 (9)	0.0045 (9)
C2	0.0398 (12)	0.0454 (13)	0.0418 (12)	-0.0021 (9)	0.0233 (10)	-0.0007 (10)
C3	0.0341 (10)	0.0336 (11)	0.0360 (11)	0.0000 (8)	0.0147 (9)	-0.0030 (8)
N1	0.0371 (9)	0.0426 (11)	0.0396 (10)	0.0039 (8)	0.0169 (8)	0.0058 (8)
C6	0.0604 (15)	0.0433 (14)	0.0478 (13)	0.0062 (11)	0.0291 (12)	0.0072 (11)
C5	0.0705 (17)	0.0440 (14)	0.0667 (17)	-0.0068 (12)	0.0477 (15)	-0.0022 (12)
C4	0.0437 (13)	0.0522 (15)	0.0729 (18)	-0.0101 (11)	0.0350 (13)	-0.0138 (13)
N2	0.0325 (9)	0.0479 (12)	0.0511 (12)	-0.0016 (8)	0.0161 (9)	-0.0032 (9)

Geometric parameters (\AA , $^\circ$)

S1—C3	1.753 (2)	C3—N2	1.333 (3)
S1—C2	1.783 (2)	N1—C6	1.335 (3)
O2—C1	1.313 (3)	C6—C5	1.365 (4)
O2—H2	0.7449	C6—H6	0.9300
O1—C1	1.177 (3)	C5—C4	1.370 (4)

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C1—C2	1.510 (3)	C5—H5	0.9300
C2—H2A	0.9700	C4—N2	1.328 (3)
C2—H2B	0.9700	C4—H4	0.9300
C3—N1	1.327 (3)		
C3—S1—C2	102.08 (10)	N2—C3—S1	113.08 (16)
C1—O2—H2	109.4	C3—N1—C6	114.8 (2)
O1—C1—O2	125.2 (2)	N1—C6—C5	123.2 (2)
O1—C1—C2	126.1 (2)	N1—C6—H6	118.4
O2—C1—C2	108.7 (2)	C5—C6—H6	118.4
C1—C2—S1	115.15 (16)	C6—C5—C4	117.1 (2)
C1—C2—H2A	108.5	C6—C5—H5	121.5
S1—C2—H2A	108.5	C4—C5—H5	121.5
C1—C2—H2B	108.5	N2—C4—C5	121.6 (2)
S1—C2—H2B	108.5	N2—C4—H4	119.2
H2A—C2—H2B	107.5	C5—C4—H4	119.2
N1—C3—N2	126.8 (2)	C4—N2—C3	116.5 (2)
N1—C3—S1	120.15 (16)		
O1—C1—C2—S1	8.2 (3)	C3—N1—C6—C5	0.8 (4)
O2—C1—C2—S1	-172.38 (16)	N1—C6—C5—C4	0.8 (4)
C3—S1—C2—C1	77.84 (18)	C6—C5—C4—N2	-1.2 (4)
C2—S1—C3—N1	-4.9 (2)	C5—C4—N2—C3	0.0 (4)
C2—S1—C3—N2	174.94 (16)	N1—C3—N2—C4	1.8 (4)
N2—C3—N1—C6	-2.2 (3)	S1—C3—N2—C4	-177.99 (18)
S1—C3—N1—C6	177.60 (17)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H2 \cdots N2 ⁱ	0.74	1.97	2.700 (3)	166

Symmetry codes: (i) $x-1, -y+3/2, z-1/2$.

Fig. 1

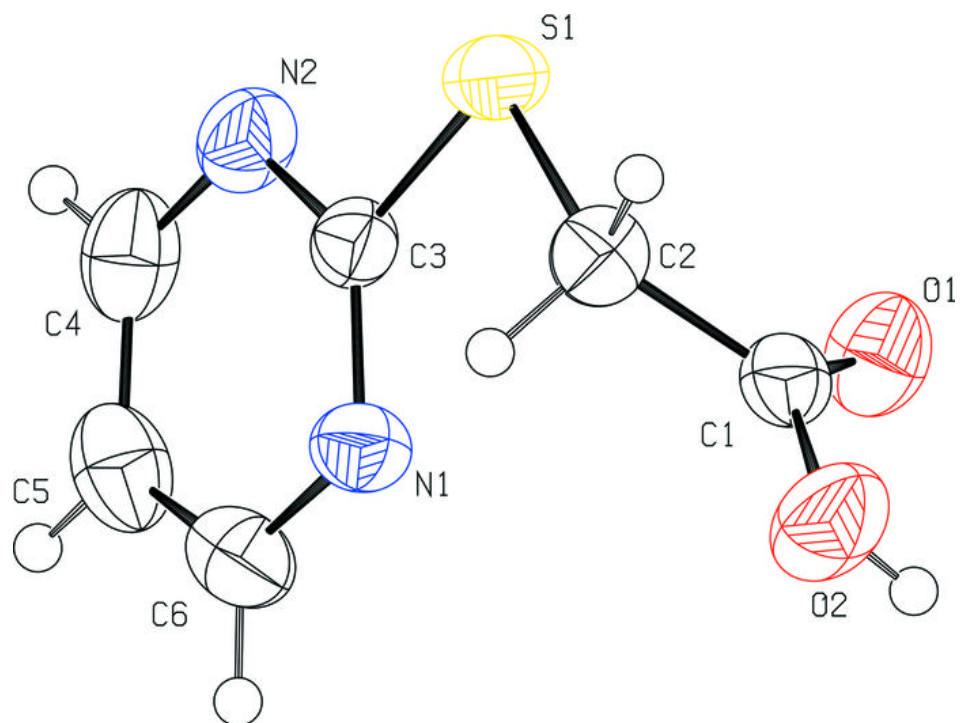


Fig. 2

