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2-[(2-Methoxyethyl)sulfanyl]-4-(2-methylpropyl)-6-oxo-1,6-dihydropyrimidine-5-carbonitrile

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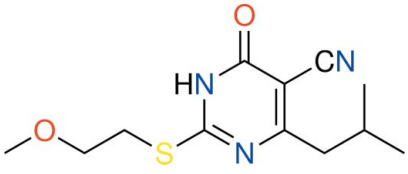
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.126; data-to-parameter ratio = 16.6.

In the title compound, $C_{12}H_{17}N_3O_2S$, the 4-methyl-2-methylsulfanyl-6-oxo-1,6-dihydropyrimidine-5-carbonitrile part of the molecule is almost planar (r.m.s deviation = 0.062 Å). In the crystal, molecules form centrosymmetric dimers *via* pairs of N—H···O hydrogen bonds.

Related literature

For related pyrimidine structures, see: Yan *et al.* (2011); El-Brollosy *et al.* (2011); Nasir *et al.* (2010); Tiekink (1989); Al-Deeb *et al.* (2012); Durkaya *et al.* (2011).



Experimental

Crystal data

$C_{12}H_{17}N_3O_2S$
M_r = 267.35
Triclinic, $P\overline{1}$
a = 5.0379 (5) Å
b = 10.5453 (10) Å
c = 13.3936 (13) Å
α = 85.274 (8)°
β = 82.170 (8)°
γ = 83.034 (8)°
V = 698.14 (12) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 296 K
0.68 × 0.47 × 0.15 mm

Data collection

Stoe IPDS 2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.859, T_{max} = 0.966
6663 measured reflections
2725 independent reflections
2090 reflections with $I > 2\sigma(I)$
R_{int} = 0.062

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.126
S = 1.02
2725 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	1.89	2.747 (2)	175

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *WinGX* (Farrugia, 1997) and *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5854).

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supporting information

Acta Cryst. (2012). E**68**, o1379 [doi:10.1107/S1600536812013372]

2-[(2-Methoxyethyl)sulfanyl]-4-(2-methylpropyl)-6-oxo-1,6-dihydropyrimidine-5-carbonitrile

Ali A. El-Emam, Güneş Demirtaş, Necmi Dege, Omar A. Al-Deeb and Nasser R. El-Brollosy

S1. Comment

In continuation to our work on the chemical and pharmacological properties of pyrimidine derivatives, we synthesized the title compound as a potential chemotherapeutic agent. The 4-methyl-2-(methylthio)-6-oxo-1,6-dihydropyrimidine-5-carbonitrile part of the molecule is almost planar. Some pyrimidine structures have been described in the literature (Yan *et al.*, 2011; El-Brollosy *et al.*, 2011; Nasir *et al.*, 2010; Tiekink 1989; Al-Deeb *et al.*, 2012; Durkaya *et al.*, 2011) and the bond distances of our crystal structure is comparable with these structures.

In the crystal, the molecules form centrosymmetric dimers via intermolecular N—H···O hydrogen bonds.

S2. Experimental

To a solution of 6-(2-methylpropyl)-4-oxo-2-sulfanylidene-1,2,3,4-tetrahydropyrimidine-5-carbonitrile (2.09 g, 0.01 mol) in DMF (10 ml), 1-bromo-2-methoxyethane (1.4 g, 0.01 mol) and anhydrous potassium carbonate (1.38 g, 0.01 mol) were added and the mixture was stirred at room temperature for 12 h. Water (15 ml) was then added and the mixture was stirred for further 30 min. The separated solid was filtered, washed with cold water, dried and crystallized from water to yield 1.15 g (43%) of the title compound ($C_{12}H_{17}N_3O_2S$) as colorless crystals. M.P.: 113–115 ^{o}C. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution of the title compound in EtOH at room temperature. ^{1}H NMR (DMSO-d_6, 500.13 MHz): δ 0.93 (d, 6H, CH_3, J = 7.0 Hz), 2.12–2.15 (m, 1H, CH), 2.53 (d, 2H, C***H***$_2$CH, J = 7.0 Hz), 3.27 (s, 3H, CH_3O), 3.53 (t, 2H, CH_2S, J = 6.5 Hz), 3.56 (t, 2H, OC***H***$_2$CH$_2$, J = 6.5 Hz), 13.55 (s, 1H, NH). ^{13}C NMR (DMSO-d_6, 125.76 MHz): δ 22.55 (CH_3), 27.94 (CH), 30.19 (CH_2S), 45.30 (***C***H$_2$CH), 58.36 (CH_3O), 70.25 (OCH_2), 95.97 (C-5), 115.55 (CN), 162.05 (C-6), 166.19 (C=O), 174.50 (C-2).

S3. Refinement

All H atoms were positioned geometrically [N—H = 0.860 Å and C—H = 0.960 Å, 0.970 Å or 0.980 Å] and treated as riding with U_{iso}(H)=1.2U_{eq}(C,N).

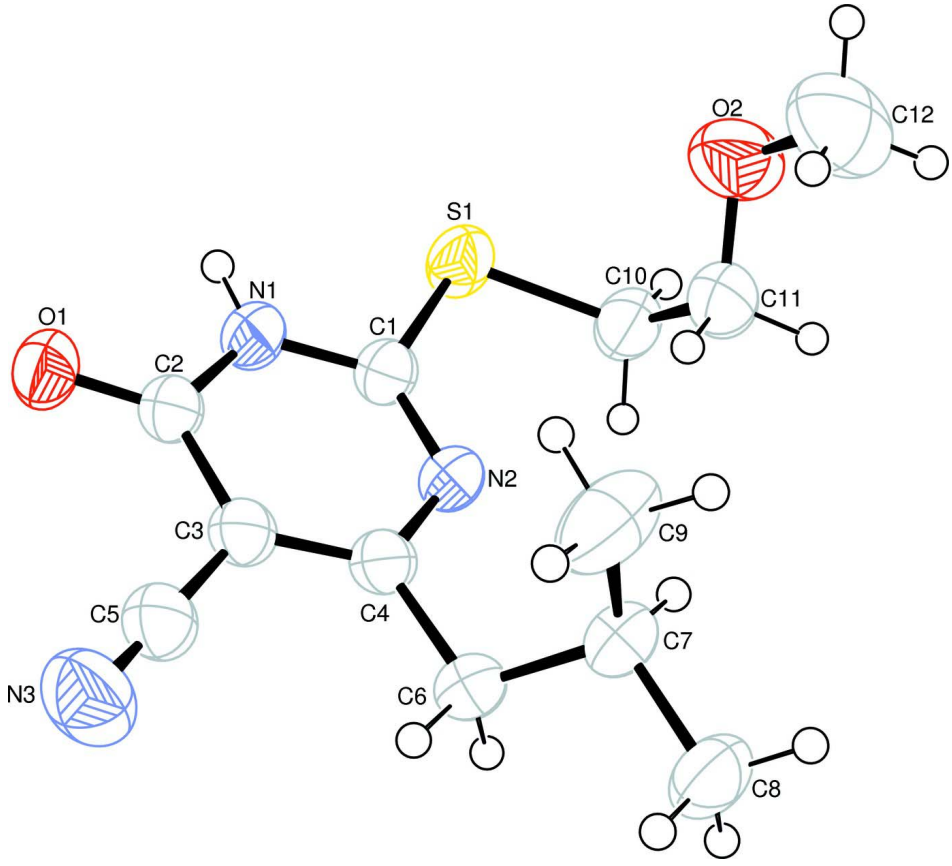

Figure 1

The asymmetric unit of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

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Crystal data

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$\beta = 82.170\ (8)°$
$\gamma = 83.034\ (8)°$
$V = 698.14\ (12)$ Å^3
$Z = 2$
$F(000) = 284$
$D_x = 1.272$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9417 reflections
$\theta = 3.1–27.9°$
$\mu = 0.23$ mm^{-1}
$T = 296$ K
Prism, colorless
0.68 × 0.47 × 0.15 mm

Data collection

Stoe IPDS 2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
rotation method scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min} = 0.859$, $T_{max} = 0.966$
6663 measured reflections
2725 independent reflections
2090 reflections with $I > 2\sigma(I)$

R_{int} = 0.062
θ_{max} = 26.0°, θ_{min} = 3.1°
h = −6→6
k = −12→12
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.126
S = 1.02
2725 reflections
164 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
$w = 1/[\sigma^2(F_o^2) + (0.0743P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.059 (9)

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.6121 (4)	0.32542 (17)	0.13412 (14)	0.0433 (4)
C2	0.9074 (4)	0.48214 (16)	0.15109 (15)	0.0450 (4)
C3	0.8266 (4)	0.46228 (16)	0.25758 (15)	0.0442 (4)
C4	0.6474 (4)	0.37554 (16)	0.29360 (14)	0.0437 (4)
C5	0.9463 (4)	0.53342 (18)	0.32209 (16)	0.0520 (5)
C6	0.5689 (4)	0.34539 (18)	0.40400 (14)	0.0495 (5)
H6A	0.3788	0.3735	0.4212	0.059*
H6B	0.6694	0.3929	0.4421	0.059*
C7	0.6216 (4)	0.20252 (19)	0.43492 (15)	0.0479 (4)
H7	0.5092	0.1569	0.3991	0.057*
C8	0.5377 (5)	0.1782 (2)	0.54676 (17)	0.0632 (6)
H8A	0.3491	0.2060	0.5625	0.076*
H8B	0.6398	0.2249	0.5837	0.076*
H8C	0.5710	0.0883	0.5652	0.076*
C9	0.9103 (5)	0.1502 (3)	0.4063 (2)	0.0851 (9)
H9A	1.0253	0.1946	0.4393	0.102*
H9B	0.9561	0.1620	0.3344	0.102*
H9C	0.9339	0.0606	0.4267	0.102*
C10	0.3129 (4)	0.1261 (2)	0.12329 (17)	0.0566 (5)
H10A	0.1835	0.1737	0.1707	0.068*

H10B	0.2118	0.0853	0.0808	0.068*
C11	0.4729 (5)	0.0231 (2)	0.18197 (18)	0.0646 (6)
H11A	0.5673	0.0621	0.2278	0.078*
H11B	0.3517	−0.0319	0.2218	0.078*
C12	0.7991 (8)	−0.1519 (3)	0.1697 (3)	0.1020 (10)
H12A	0.6742	−0.2072	0.2050	0.122*
H12B	0.8951	−0.1190	0.2173	0.122*
H12C	0.9246	−0.1993	0.1222	0.122*
N1	0.7874 (3)	0.40967 (14)	0.09326 (12)	0.0468 (4)
H1	0.8246	0.4179	0.0286	0.056*
N2	0.5378 (3)	0.30759 (14)	0.23047 (12)	0.0452 (4)
N3	1.0420 (5)	0.5910 (2)	0.37314 (18)	0.0761 (6)
O1	1.0717 (3)	0.55469 (13)	0.11167 (11)	0.0575 (4)
O2	0.6562 (4)	−0.04885 (17)	0.11760 (15)	0.0855 (6)
S1	0.50676 (11)	0.23771 (5)	0.04486 (4)	0.0547 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0492 (9)	0.0408 (9)	0.0385 (10)	−0.0040 (7)	−0.0042 (8)	0.0019 (7)
C2	0.0564 (10)	0.0373 (9)	0.0396 (10)	−0.0048 (7)	−0.0018 (8)	0.0007 (7)
C3	0.0553 (10)	0.0365 (9)	0.0393 (10)	−0.0011 (7)	−0.0048 (8)	−0.0003 (7)
C4	0.0518 (10)	0.0375 (8)	0.0388 (10)	0.0004 (7)	−0.0015 (8)	−0.0005 (7)
C5	0.0637 (12)	0.0444 (10)	0.0473 (11)	−0.0056 (8)	−0.0067 (9)	−0.0014 (9)
C6	0.0616 (11)	0.0483 (10)	0.0365 (10)	−0.0073 (8)	0.0017 (8)	−0.0027 (8)
C7	0.0479 (10)	0.0544 (10)	0.0402 (10)	−0.0077 (8)	−0.0033 (8)	0.0033 (8)
C8	0.0734 (14)	0.0715 (14)	0.0423 (12)	−0.0143 (11)	0.0004 (10)	0.0074 (10)
C9	0.0608 (14)	0.103 (2)	0.0744 (18)	0.0151 (13)	0.0095 (12)	0.0300 (16)
C10	0.0606 (12)	0.0580 (12)	0.0512 (12)	−0.0167 (9)	0.0002 (9)	−0.0007 (9)
C11	0.0896 (16)	0.0523 (11)	0.0507 (13)	−0.0130 (11)	−0.0013 (11)	−0.0013 (10)
C12	0.127 (3)	0.0788 (18)	0.096 (3)	0.0192 (17)	−0.029 (2)	−0.0031 (17)
N1	0.0598 (9)	0.0449 (8)	0.0347 (8)	−0.0099 (7)	−0.0015 (7)	0.0027 (6)
N2	0.0537 (8)	0.0448 (8)	0.0357 (8)	−0.0073 (6)	−0.0008 (7)	0.0007 (6)
N3	0.0948 (15)	0.0671 (12)	0.0729 (15)	−0.0169 (11)	−0.0209 (12)	−0.0147 (11)
O1	0.0751 (9)	0.0526 (8)	0.0456 (8)	−0.0238 (7)	0.0009 (7)	0.0019 (6)
O2	0.1202 (15)	0.0712 (11)	0.0588 (11)	0.0131 (10)	−0.0080 (10)	−0.0074 (9)
S1	0.0701 (4)	0.0594 (3)	0.0368 (3)	−0.0201 (2)	−0.0053 (2)	−0.0007 (2)

Geometric parameters (Å, º)

C1—N2	1.299 (2)	C8—H8B	0.9600
C1—N1	1.359 (2)	C8—H8C	0.9600
C1—S1	1.744 (2)	C9—H9A	0.9600
C2—O1	1.232 (2)	C9—H9B	0.9600
C2—N1	1.374 (2)	C9—H9C	0.9600
C2—C3	1.434 (3)	C10—C11	1.505 (3)
C3—C4	1.376 (3)	C10—S1	1.800 (2)
C3—C5	1.425 (3)	C10—H10A	0.9700

C4—N2	1.363 (2)	C10—H10B	0.9700
C4—C6	1.496 (3)	C11—O2	1.376 (3)
C5—N3	1.139 (3)	C11—H11A	0.9700
C6—C7	1.530 (3)	C11—H11B	0.9700
C6—H6A	0.9700	C12—O2	1.417 (3)
C6—H6B	0.9700	C12—H12A	0.9600
C7—C9	1.502 (3)	C12—H12B	0.9600
C7—C8	1.510 (3)	C12—H12C	0.9600
C7—H7	0.9800	N1—H1	0.8600
C8—H8A	0.9600		

N2—C1—N1	123.67 (18)	C7—C9—H9A	109.5
N2—C1—S1	122.84 (14)	C7—C9—H9B	109.5
N1—C1—S1	113.43 (14)	H9A—C9—H9B	109.5
O1—C2—N1	120.88 (18)	C7—C9—H9C	109.5
O1—C2—C3	125.26 (18)	H9A—C9—H9C	109.5
N1—C2—C3	113.85 (16)	H9B—C9—H9C	109.5
C4—C3—C5	122.88 (18)	C11—C10—S1	115.59 (16)
C4—C3—C2	120.35 (18)	C11—C10—H10A	108.4
C5—C3—C2	116.75 (17)	S1—C10—H10A	108.4
N2—C4—C3	121.83 (17)	C11—C10—H10B	108.4
N2—C4—C6	115.56 (15)	S1—C10—H10B	108.4
C3—C4—C6	122.56 (18)	H10A—C10—H10B	107.4
N3—C5—C3	179.5 (2)	O2—C11—C10	110.6 (2)
C4—C6—C7	112.75 (16)	O2—C11—H11A	109.5
C4—C6—H6A	109.0	C10—C11—H11A	109.5
C7—C6—H6A	109.0	O2—C11—H11B	109.5
C4—C6—H6B	109.0	C10—C11—H11B	109.5
C7—C6—H6B	109.0	H11A—C11—H11B	108.1
H6A—C6—H6B	107.8	O2—C12—H12A	109.5
C9—C7—C8	110.84 (19)	O2—C12—H12B	109.5
C9—C7—C6	112.29 (18)	H12A—C12—H12B	109.5
C8—C7—C6	110.23 (18)	O2—C12—H12C	109.5
C9—C7—H7	107.8	H12A—C12—H12C	109.5
C8—C7—H7	107.8	H12B—C12—H12C	109.5
C6—C7—H7	107.8	C1—N1—C2	122.62 (16)
C7—C8—H8A	109.5	C1—N1—H1	118.7
C7—C8—H8B	109.5	C2—N1—H1	118.7
H8A—C8—H8B	109.5	C1—N2—C4	117.66 (16)
C7—C8—H8C	109.5	C11—O2—C12	112.2 (2)
H8A—C8—H8C	109.5	C1—S1—C10	102.06 (10)
H8B—C8—H8C	109.5		

O1—C2—C3—C4	−177.90 (18)	N2—C1—N1—C2	1.5 (3)
N1—C2—C3—C4	0.9 (3)	S1—C1—N1—C2	−175.81 (14)
O1—C2—C3—C5	0.4 (3)	O1—C2—N1—C1	177.75 (17)
N1—C2—C3—C5	179.25 (15)	C3—C2—N1—C1	−1.1 (3)
C5—C3—C4—N2	−179.28 (17)	N1—C1—N2—C4	−1.5 (3)

C2—C3—C4—N2	−1.1 (3)	S1—C1—N2—C4	175.58 (13)
C5—C3—C4—C6	−1.9 (3)	C3—C4—N2—C1	1.3 (3)
C2—C3—C4—C6	176.28 (17)	C6—C4—N2—C1	−176.22 (16)
N2—C4—C6—C7	53.7 (2)	C10—C11—O2—C12	−176.3 (2)
C3—C4—C6—C7	−123.80 (19)	N2—C1—S1—C10	−3.49 (19)
C4—C6—C7—C9	56.1 (3)	N1—C1—S1—C10	173.88 (14)
C4—C6—C7—C8	−179.82 (18)	C11—C10—S1—C1	−70.78 (19)
S1—C10—C11—O2	−59.3 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	1.89	2.747 (2)	175

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