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4-Amino-6-(2,2-diethoxyethoxy)-2-(methylsulfanyl)pyrimidine

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The supramolecular structure of the title compound, $C_{11}H_{19}N_3O_3S$, consists of a ribbon of alternating centrosymmetric $R_2^2(8)$ and $R_2^2(18)$ rings.

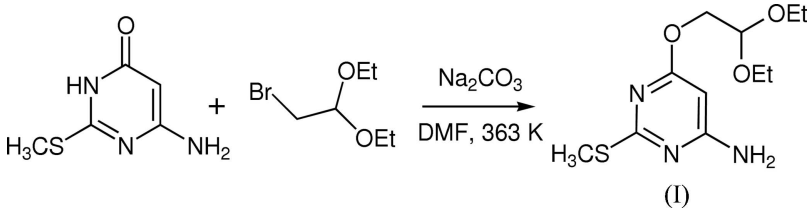
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.008 Å
R factor = 0.073
wR factor = 0.253
Data-to-parameter ratio = 18.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The title compound, (I), was prepared as an intermediate for the preparation of fused pyrimidine systems in our ongoing programme aimed at the development of novel syntheses of fused heterocyclic systems. The bond lengths and angles show no unusual features.



(I)

Atom N4 in the molecule at (x, y, z) acts *via* H4*A* as a hydrogen-bond donor to atom N3 in the molecule at ($1-x$, $1-y$, $-z$), so generating by inversion an $R_2^2(8)$ (Bernstein *et al.*, 1995) ring centred at ($\frac{1}{2}$, $\frac{1}{2}$, 0). Atom N4 at (x, y, z) acts as a hydrogen-bond donor *via* atom H4*B* to atom O62 in the molecule at ($-x$, $1-y$, $1-z$), so generating by inversion an $R_2^2(18)$ ring centred at (0, $\frac{1}{2}$, $\frac{1}{2}$). Propagation by inversion of these two hydrogen bonds then generates a chain of alternating $R_2^2(8)$ and $R_2^2(18)$ rings running parallel to the [$\bar{1}$01] direction with the $R_2^2(8)$ rings centred at ($\frac{1}{2}-n$, $\frac{1}{2}$, n) (n = zero or an integer) and the $R_2^2(18)$ rings centred at (n, $\frac{1}{2}$, $\frac{1}{2}-n$) (n = zero or an integer). There are no direction-specific interactions between adjacent chains.

Experimental

To a solution of 4-amino-2-(methylsulfanyl)pyrimidin-6(1*H*)-one (20 mmol) in dry dimethylformamide (40 ml) was added solid anhydrous sodium carbonate (30 mmol). The mixture was stirred at room temperature for 10 min, bromoacetaldehyde diethyl acetal (60 mmol) was added and the final mixture heated at 363 K for 15 h. Water (20 ml) was added and the mixture was then poured on to crushed ice. The resultant solid was collected by filtration and washed with cold water to give the title compound in 85% yield. This solid was recrystallized from methanol to give light-yellow crystalline micaceous blocks. HRMS found 273.1161, calculated for $C_{11}H_{19}N_3O_3S$ 273.1147.

Crystal data

$C_{11}H_{19}N_3O_3S$
M_r = 273.35
Triclinic, $P\overline{1}$
a = 7.2278 (8) Å
b = 9.6358 (9) Å
c = 10.0852 (10) Å
α = 81.197 (9)°
β = 82.390 (7)°
γ = 78.818 (8)°
V = 677.12 (12) Å^3
Z = 2
D_x = 1.341 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 120 (2) K
Block, yellow
0.52 × 0.29 × 0.14 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Thick–slice φ and ω scans
Absorption correction: multi-scan (*EVALCCD*; Duisenberg *et al.*, 2003)
T_{min} = 0.884, T_{max} = 0.967
10168 measured reflections
3000 independent reflections
1200 reflections with $I > 2\sigma(I)$
R_{int} = 0.118
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.253
S = 1.02
3000 reflections
166 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1062P)^2 + 1.0232P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.70 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4A···N3^{i}	1.00	2.07	3.052 (6)	167
N4—H4B···O62^{ii}	0.91	2.05	2.919 (6)	158

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

H atoms were treated as riding atoms, with aromatic C—H = 0.95 Å, aliphatic C—H = 1.00 Å and CH_2 C—H = 0.99 Å, with U_{iso}(H) = 1.2U_{eq}(C), methyl C—H = 0.98 Å, with U_{iso}(H) = 1.5U_{eq}(C), and N—H = 0.92 and 1.00 Å, with U_{iso} = 1.2U_{eq}(N). In the case of atom N4, atoms H4*A* and H4*B* were located in a difference map, refined and then treated as riding atoms in the latter stages of refinement. The difference map showed that the peak related to H4*A* was extended from N4 lying along the N4···N3^{i} [symmetry code: (i) $1-x, 1-y, -z$] vector. The methyl H atoms of the methylsulfanyl group were modelled as six equally spaced half-H atoms. The crystal quality was generally poor due to the micaceous habit of the crystals; this has resulted in a high R_{int} value and a low ratio of observed/unique reflections.

Data collection: *COLLECT* (Bruker–Nonius, 2004); cell refinement: *DIRAX/LSQ* (Duisenberg *et al.*, 2000); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *OSCAIL* (McArdle, 2003) and *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

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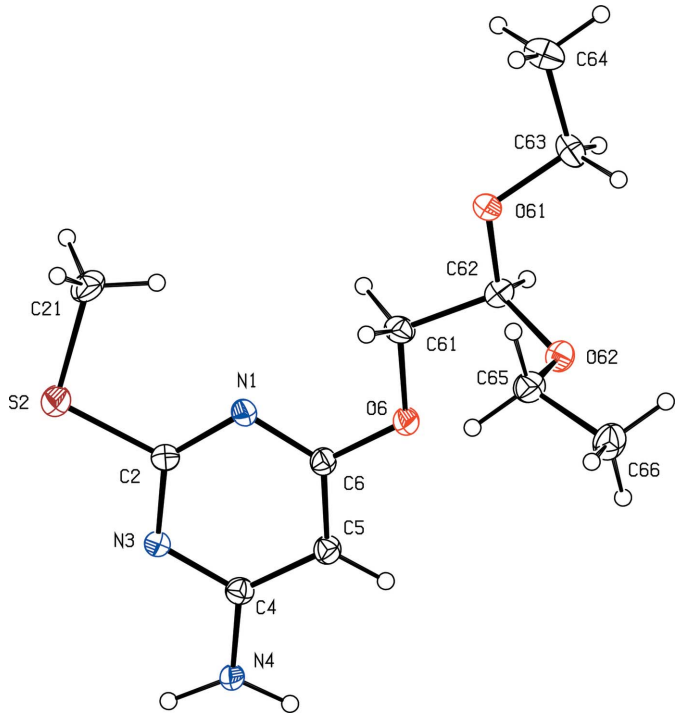

Figure 1
A view of the molecular structure of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms as spheres of arbitrary radii.

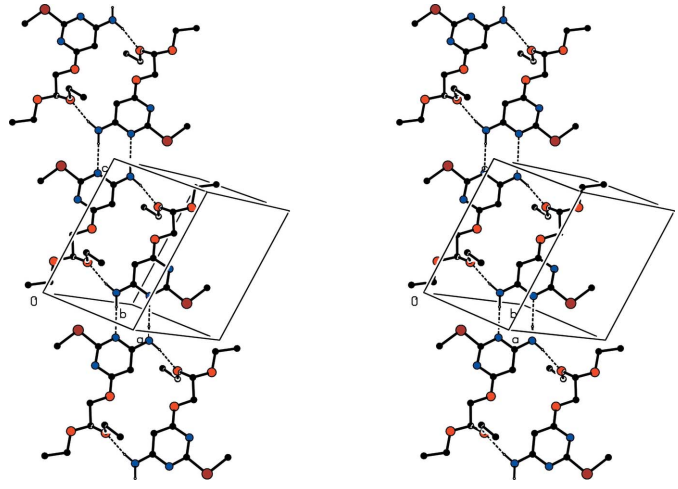

Figure 2
A stereoscopic view of the ribbon formed by alternating $R_2^2(8)$ and $R_2^2(18)$ centrosymmetric dimers. H atoms bonded to C atoms have been omitted.

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supporting information

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Crystal data

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$M_r = 273.35$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.2280$ (8) Å
$b = 9.6360$ (9) Å
$c = 10.085$ (1) Å
$\alpha = 81.197$ (9)°
$\beta = 82.390$ (7)°
$\gamma = 78.817$ (8)°
$V = 677.12$ (12) Å^3
$Z = 2$
$F(000) = 292$
$D_x = 1.341$ Mg m^{-3}
Melting point: 438 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3000 reflections
$\theta = 5.1$–27.5°
$\mu = 0.24$ mm^{-1}
$T = 120$ K
Block, yellow
0.52 × 0.29 × 0.14 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Thick–slice π and ω scans
Absorption correction: multi-scan (EvalCCD; Duisenberg *et al.*, 2003)
$T_{min} = 0.884$, $T_{max} = 0.967$
10168 measured reflections
3000 independent reflections
1200 reflections with $I > 2\sigma(I)$
$R_{int} = 0.118$
$\theta_{max} = 27.5°$, $\theta_{min} = 5.1°$
$h = -8 \rightarrow 9$
$k = -12 \rightarrow 12$
$l = -13 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.073$
$wR(F^2) = 0.253$
$S = 1.02$
3000 reflections
166 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
$w = 1/[\sigma^2(F_o^2) + (0.1062P)^2 + 1.0232P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.70$ e Å^{-3}
$\Delta\rho_{min} = -0.53$ e Å^{-3}

Special details

Experimental. The scale factors in the experimental table are calculated from the 'size' command in the *SHELXL97* input file.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
N1	0.4599 (6)	0.6665 (4)	0.3719 (4)	0.0270 (10)	
C2	0.5473 (7)	0.6411 (5)	0.2507 (5)	0.0281 (12)	
S2	0.7579 (2)	0.70417 (16)	0.19170 (14)	0.0373 (5)	
C21	0.7992 (8)	0.7871 (6)	0.3302 (6)	0.0401 (15)	
N3	0.4967 (6)	0.5670 (4)	0.1636 (4)	0.0246 (10)	
C4	0.3395 (7)	0.5080 (5)	0.2048 (5)	0.0266 (12)	
N4	0.2892 (6)	0.4329 (5)	0.1184 (4)	0.0317 (11)	
C5	0.2369 (7)	0.5264 (5)	0.3319 (5)	0.0278 (12)	
C6	0.3043 (7)	0.6092 (5)	0.4078 (5)	0.0252 (12)	
O6	0.2063 (5)	0.6314 (4)	0.5289 (3)	0.0318 (9)	
C61	0.2784 (8)	0.7194 (6)	0.6052 (5)	0.0317 (13)	
C62	0.1616 (7)	0.7193 (6)	0.7414 (5)	0.0307 (13)	
O61	0.2315 (5)	0.8144 (4)	0.8062 (3)	0.0325 (9)	
C63	0.1706 (9)	0.8063 (6)	0.9472 (5)	0.0377 (14)	
C64	0.2834 (9)	0.8905 (6)	1.0088 (6)	0.0455 (16)	
O62	−0.0357 (5)	0.7540 (4)	0.7356 (3)	0.0307 (9)	
C65	−0.1042 (8)	0.8906 (6)	0.6607 (5)	0.0359 (14)	
C66	−0.3171 (8)	0.9162 (6)	0.6835 (6)	0.0428 (15)	
H21A	0.9162	0.8266	0.3072	0.060*	0.50
H21B	0.8115	0.7160	0.4106	0.060*	0.50
H21C	0.6924	0.8640	0.3484	0.060*	0.50
H21D	0.6972	0.7778	0.4036	0.060*	0.50
H21E	0.8019	0.8884	0.3002	0.060*	0.50
H21F	0.9210	0.7404	0.3624	0.060*	0.50
H4A	0.3713	0.4190	0.0323	0.038*	
H4B	0.1858	0.3925	0.1537	0.038*	
H5	0.1280	0.4843	0.3632	0.033*	
H61A	0.2684	0.8179	0.5574	0.038*	
H61B	0.4135	0.6812	0.6168	0.038*	
H62	0.1910	0.6218	0.7929	0.037*	
H63A	0.1914	0.7056	0.9896	0.045*	
H63B	0.0337	0.8460	0.9619	0.045*	
H64A	0.2631	0.9896	0.9654	0.068*	
H64B	0.4184	0.8491	0.9955	0.068*	
H64C	0.2421	0.8878	1.1056	0.068*	
H65A	−0.0615	0.8898	0.5633	0.043*	
H65B	−0.0547	0.9672	0.6925	0.043*	
H66A	−0.3576	0.9137	0.7804	0.064*	
H66B	−0.3647	0.8419	0.6486	0.064*	
H66C	−0.3680	1.0098	0.6365	0.064*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.030 (3)	0.031 (2)	0.021 (2)	−0.011 (2)	−0.0009 (19)	−0.0021 (18)

C2	0.026 (3)	0.033 (3)	0.026 (3)	−0.006 (2)	−0.005 (2)	−0.003 (2)
S2	0.0361 (9)	0.0440 (9)	0.0357 (8)	−0.0165 (7)	0.0007 (6)	−0.0091 (6)
C21	0.034 (4)	0.041 (3)	0.051 (3)	−0.016 (3)	−0.002 (3)	−0.017 (3)
N3	0.025 (3)	0.028 (2)	0.022 (2)	−0.007 (2)	−0.0029 (18)	−0.0031 (18)
C4	0.026 (3)	0.027 (3)	0.027 (3)	−0.003 (2)	−0.004 (2)	−0.005 (2)
N4	0.029 (3)	0.047 (3)	0.025 (2)	−0.019 (2)	0.0042 (19)	−0.014 (2)
C5	0.026 (3)	0.028 (3)	0.029 (3)	−0.005 (2)	0.000 (2)	−0.007 (2)
C6	0.021 (3)	0.027 (3)	0.025 (3)	−0.001 (2)	−0.002 (2)	−0.002 (2)
O6	0.030 (2)	0.042 (2)	0.0268 (18)	−0.0138 (18)	0.0040 (16)	−0.0128 (16)
C61	0.032 (3)	0.038 (3)	0.028 (3)	−0.006 (3)	−0.003 (2)	−0.012 (2)
C62	0.027 (3)	0.035 (3)	0.033 (3)	−0.008 (3)	−0.004 (2)	−0.005 (2)
O61	0.032 (2)	0.044 (2)	0.0250 (17)	−0.0116 (18)	−0.0007 (15)	−0.0112 (16)
C63	0.045 (4)	0.043 (3)	0.024 (3)	−0.008 (3)	0.002 (2)	−0.005 (2)
C64	0.057 (4)	0.046 (4)	0.036 (3)	−0.006 (3)	−0.013 (3)	−0.010 (3)
O62	0.027 (2)	0.030 (2)	0.0335 (19)	−0.0045 (17)	−0.0013 (16)	−0.0045 (16)
C65	0.036 (4)	0.037 (3)	0.037 (3)	−0.011 (3)	−0.005 (3)	−0.004 (3)
C66	0.028 (3)	0.048 (4)	0.048 (3)	−0.001 (3)	−0.003 (3)	−0.003 (3)

Geometric parameters (Å, º)

N1—C6	1.331 (6)	C61—C62	1.514 (7)
N1—C2	1.335 (6)	C61—H61A	0.99
C2—N3	1.339 (6)	C61—H61B	0.99
C2—S2	1.746 (5)	C62—O61	1.404 (6)
S2—C21	1.796 (5)	C62—O62	1.407 (6)
C21—H21A	0.98	C62—H62	1.00
C21—H21B	0.98	O61—C63	1.427 (6)
C21—H21C	0.98	C63—C64	1.501 (7)
C21—H21D	0.98	C63—H63A	0.99
C21—H21E	0.98	C63—H63B	0.99
C21—H21F	0.98	C64—H64A	0.98
N3—C4	1.357 (6)	C64—H64B	0.98
C4—N4	1.340 (6)	C64—H64C	0.98
C4—C5	1.411 (7)	O62—C65	1.446 (6)
N4—H4A	0.9959	C65—C66	1.503 (8)
N4—H4B	0.9151	C65—H65A	0.99
C5—C6	1.377 (7)	C65—H65B	0.99
C5—H5	0.95	C66—H66A	0.98
C6—O6	1.354 (6)	C66—H66B	0.98
O6—C61	1.435 (6)	C66—H66C	0.98
C6—N1—C2	114.3 (4)	C6—O6—C61	116.0 (4)
N1—C2—N3	128.0 (5)	O6—C61—C62	107.8 (4)
N1—C2—S2	119.7 (4)	O6—C61—H61A	110.1
N3—C2—S2	112.3 (4)	C62—C61—H61A	110.1
C2—S2—C21	102.8 (3)	O6—C61—H61B	110.1
S2—C21—H21A	109.5	C62—C61—H61B	110.1
S2—C21—H21B	109.5	H61A—C61—H61B	108.5

H21A—C21—H21B	109.5	O61—C62—O62	113.1 (4)
S2—C21—H21C	109.5	O61—C62—C61	104.5 (4)
H21A—C21—H21C	109.5	O62—C62—C61	114.6 (4)
H21B—C21—H21C	109.5	O61—C62—H62	108.1
S2—C21—H21D	109.5	O62—C62—H62	108.1
H21A—C21—H21D	141.1	C61—C62—H62	108.1
H21B—C21—H21D	56.3	C62—O61—C63	113.5 (4)
H21C—C21—H21D	56.3	O61—C63—C64	108.1 (5)
S2—C21—H21E	109.5	O61—C63—H63A	110.1
H21A—C21—H21E	56.3	C64—C63—H63A	110.1
H21B—C21—H21E	141.1	O61—C63—H63B	110.1
H21C—C21—H21E	56.3	C64—C63—H63B	110.1
H21D—C21—H21E	109.5	H63A—C63—H63B	108.4
S2—C21—H21F	109.5	C63—C64—H64A	109.5
H21A—C21—H21F	56.3	C63—C64—H64B	109.5
H21B—C21—H21F	56.3	H64A—C64—H64B	109.5
H21C—C21—H21F	141.1	C63—C64—H64C	109.5
H21D—C21—H21F	109.5	H64A—C64—H64C	109.5
H21E—C21—H21F	109.5	H64B—C64—H64C	109.5
C2—N3—C4	116.0 (4)	C62—O62—C65	115.9 (4)
N4—C4—N3	116.2 (4)	O62—C65—C66	107.5 (4)
N4—C4—C5	122.9 (5)	O62—C65—H65A	110.2
N3—C4—C5	120.9 (4)	C66—C65—H65A	110.2
C4—N4—H4A	119.3	O62—C65—H65B	110.2
C4—N4—H4B	111.8	C66—C65—H65B	110.2
H4A—N4—H4B	128.5	H65A—C65—H65B	108.5
C6—C5—C4	115.9 (5)	C65—C66—H66A	109.5
C6—C5—H5	122.1	C65—C66—H66B	109.5
C4—C5—H5	122.1	H66A—C66—H66B	109.5
N1—C6—O6	117.9 (4)	C65—C66—H66C	109.5
N1—C6—C5	124.9 (4)	H66A—C66—H66C	109.5
O6—C6—C5	117.2 (4)	H66B—C66—H66C	109.5

C6—N1—C2—N3	0.3 (7)	C4—C5—C6—O6	178.6 (4)
C6—N1—C2—S2	−177.9 (3)	N1—C6—O6—C61	1.9 (6)
N1—C2—S2—C21	2.1 (5)	C5—C6—O6—C61	−179.3 (4)
N3—C2—S2—C21	−176.3 (4)	C6—O6—C61—C62	−174.7 (4)
N1—C2—N3—C4	−1.5 (7)	O6—C61—C62—O61	−176.2 (4)
S2—C2—N3—C4	176.8 (3)	O6—C61—C62—O62	−51.8 (6)
C2—N3—C4—N4	−179.6 (4)	O62—C62—O61—C63	68.0 (5)
C2—N3—C4—C5	0.6 (7)	C61—C62—O61—C63	−166.7 (4)
N4—C4—C5—C6	−178.5 (5)	C62—O61—C63—C64	169.2 (4)
N3—C4—C5—C6	1.3 (7)	O61—C62—O62—C65	62.1 (5)
C2—N1—C6—O6	−179.3 (4)	C61—C62—O62—C65	−57.5 (6)
C2—N1—C6—C5	2.0 (7)	C62—O62—C65—C66	−171.9 (4)
C4—C5—C6—N1	−2.7 (7)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*A*···N3^{i}	1.00	2.07	3.052 (6)	167
N4—H4*B*···O62^{ii}	0.91	2.05	2.919 (6)	158

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