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Key indicators

Single-crystal X-ray study
T = 120 K
Mean $\sigma(\text{C}-\text{C}) = 0.008 \text{ \AA}$
R factor = 0.073
wR factor = 0.253
Data-to-parameter ratio = 18.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

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

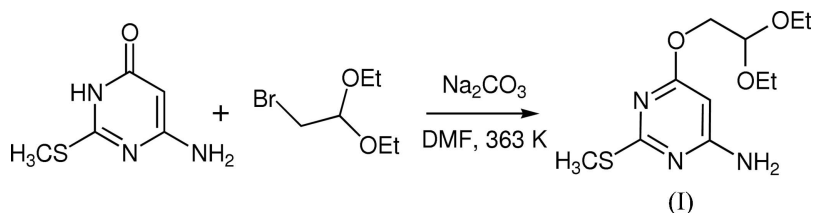
The supramolecular structure of the title compound, $\text{C}_{11}\text{H}_{19}\text{N}_3\text{O}_3\text{S}$, consists of a ribbon of alternating centrosymmetric $R_2^2(8)$ and $R_2^2(18)$ rings.

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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.



Atom N4 in the molecule at (x, y, z) acts *via* H4A 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 H4B 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(1H)-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 $\text{C}_{11}\text{H}_{19}\text{N}_3\text{O}_3\text{S}$ 273.1147.

Crystal data

$C_{11}H_{19}N_3O_3S$
 $M_r = 273.35$
 Triclinic, $P\bar{1}$
 $a = 7.2278$ (8) Å
 $b = 9.6358$ (9) Å
 $c = 10.0852$ (10) Å
 $\alpha = 81.197$ (9)°
 $\beta = 82.390$ (7)°
 $\gamma = 78.818$ (8)°

$V = 677.12$ (12) Å³
 $Z = 2$
 $D_x = 1.341$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.24$ mm⁻¹
 $T = 120$ (2) K
 Block, yellow
 $0.52 \times 0.29 \times 0.14$ 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 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.118$
 $\theta_{\text{max}} = 27.5^\circ$

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 $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.70$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.53$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N4-H4A\cdots N3^i$	1.00	2.07	3.052 (6)	167
$N4-H4B\cdots O62^{ii}$	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 CH₂ C–H = 0.99 Å, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, methyl C–H = 0.98 Å, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$, and N–H = 0.92 and 1.00 Å, with $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{N})$. In the case of atom N4, atoms H4A and H4B 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 H4A was extended from N4 lying along the $N4\cdots N3^i$ [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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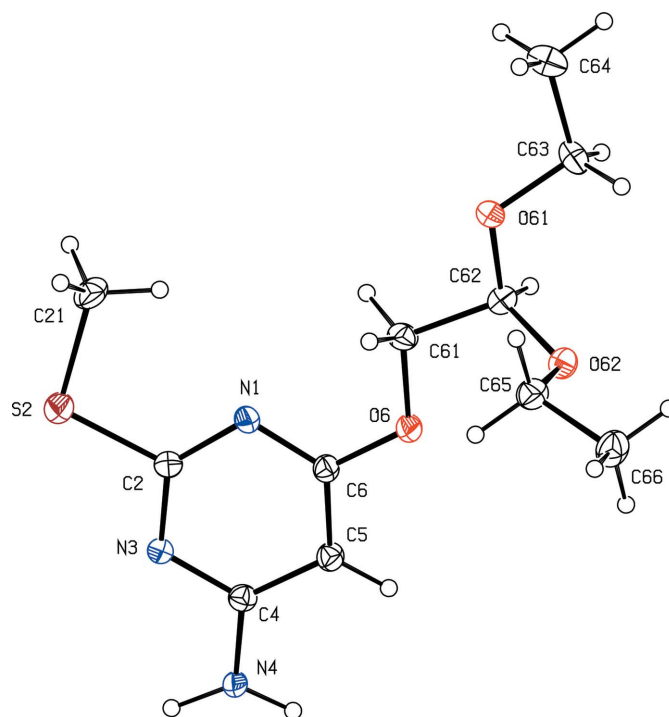


Figure 1

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.

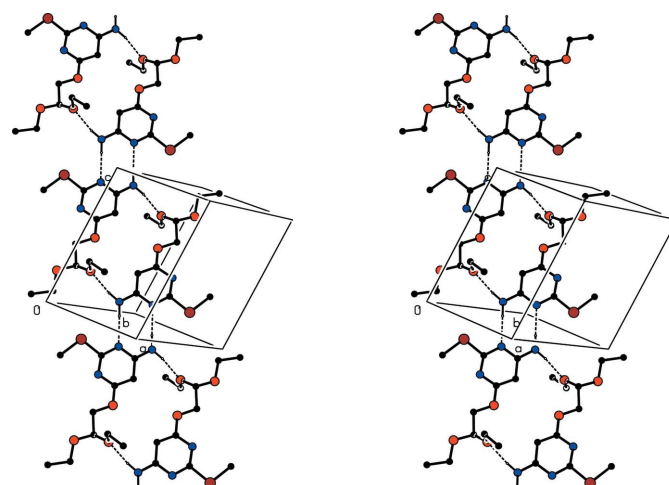


Figure 2

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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