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4,4′,6,6′-Tetramethyl-2,2′-(propyldithio)-dipyrimidine

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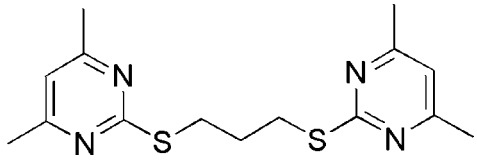
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.123; data-to-parameter ratio = 14.9.

The crystal structure of the title compound, $C_{15}H_{20}N_4S_2$, is mainly stabilized by van der Waals forces. The molecule resides on a twofold axis.

Related literature

For related literature, see: Allen *et al.* (1987); Bu *et al.* (2002); Raper (1997).



Experimental

Crystal data

$C_{15}H_{20}N_4S_2$
M_r = 320.47
Monoclinic, $C2/c$
a = 13.4654 (14) Å
b = 8.8808 (9) Å
c = 14.2371 (16) Å
β = 103.340 (2)°
V = 1656.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 298 (2) K
0.52 × 0.50 × 0.45 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.851, T_{max} = 0.869
4000 measured reflections
1461 independent reflections
1174 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.123
S = 1.01
1461 reflections
98 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2044).

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supplementary materials

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4,4',6,6'-Tetramethyl-2,2'-(propyldithio)dipyrimidine

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Comment

Previous studies have shown that flexible thioethers are well -established ligands in coordination and metallosupramolecular chemistry (Bu *et al.*, 2002; Raper, 1997). Therefore we pay our attention to the pyrimidine dithioethers, which has well known reactivity in the pyrimidine ring (positions 2, 4 and 6). As part of our ongoing investigtion on pyrimidine derivatives, the title compound, has been prepared and its crystal structure is presented here.

The molecular structure shown in Fig. 1. The bond lengths and angles are generally within normal ranges (Allen *et al.*, 1987). A view of the packing diagram is given in Fig. 2. The dihedral angle between two pyrimidine rings is 72.9 (1) °. The crystal packing (Fig. 2) is mainly stabilized by van der Waals forces.

Experimental

A solution of 1,3-dibromopropane (1.01 g, 5 mmol) in ethanol(10 ml) was slowly dripped into a refluxing solution of 2-thiol-4,6-dimethylpyrimidine (1.40 g, 10 mmol) and powdered NaOH (0.4 g, 10 mmol) in ethanol. The reaction mixture was refluxed for 5 h with stirring and cool to room temperature. The white powder of title compound was filtered and washed thoroughly with water and then air dried (yield 65%). Single crystals suitable for X-ray analysis were obtained by slow evaporation from a dichloromethane/2-propanol (3:1) solution.

Refinement

The H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93 − 0.97 Å and U_{iso} = 1.2 or 1.5 U_{eq}(parent atom).

Figures

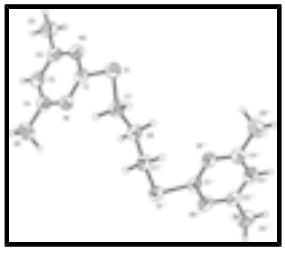

Fig. 1. The molecular structure of the title compound showing 50% probability displacement ellipsoids. Symmetry operation: $1 - x, y, 1/2 - z$.

Fig. 2. Packing diagram of the title compound viewed along the *b* axis. Hydrogen atoms are omitted for clarity.

4,4',6,6'-Tetramethyl-2,2'-(propyldithio)dipyrimidine

Crystal data

$C_{15}H_{20}N_4S_2$	$F_{000} = 680$
$M_r = 320.47$	$D_x = 1.285$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 2114 reflections
$a = 13.4654$ (14) Å	$\theta = 2.8$–27.6º
$b = 8.8808$ (9) Å	$\mu = 0.32$ mm^{-1}
$c = 14.2371$ (16) Å	$T = 298$ (2) K
$\beta = 103.340$ (2)º	Block, pale-yellow
$V = 1656.6$ (3) Å^3	0.52 × 0.50 × 0.45 mm
$Z = 4$	

Data collection

Bruker APEXII CCD area-detector diffractometer	1461 independent reflections
Radiation source: fine-focus sealed tube	1174 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.029$
$T = 298$(2) K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 2.8$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -16 \rightarrow 9$
$T_{min} = 0.851$, $T_{max} = 0.869$	$k = -10 \rightarrow 10$
4000 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.040$	H-atom parameters constrained
$wR(F^2) = 0.123$	$w = 1/[\sigma^2(F_o^2) + (0.0756P)^2 + 0.7397P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
1461 reflections	$\Delta\rho_{max} = 0.16$ e Å^{-3}
98 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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}	Occ. (<1)
N1	0.41047 (13)	0.20385 (19)	0.45126 (11)	0.0418 (4)	
N2	0.24595 (13)	0.0889 (2)	0.40016 (12)	0.0471 (5)	
S1	0.29908 (4)	0.25812 (7)	0.27214 (4)	0.0571 (3)	
C1	0.32337 (15)	0.1745 (2)	0.38755 (13)	0.0409 (5)	
C2	0.25732 (15)	0.0265 (2)	0.48815 (15)	0.0441 (5)	
C3	0.34420 (16)	0.0511 (2)	0.55924 (14)	0.0455 (5)	
H3	0.3514	0.0080	0.6200	0.055*	
C4	0.42034 (15)	0.1404 (2)	0.53904 (14)	0.0427 (5)	
C5	0.17108 (18)	−0.0686 (3)	0.50475 (18)	0.0618 (6)	
H5A	0.1094	−0.0104	0.4918	0.093*	
H5B	0.1861	−0.1023	0.5706	0.093*	
H5C	0.1625	−0.1543	0.4625	0.093*	
C6	0.51623 (18)	0.1744 (3)	0.61268 (15)	0.0604 (6)	
H6A	0.5737	0.1645	0.5839	0.091*	
H6B	0.5232	0.1050	0.6655	0.091*	
H6C	0.5133	0.2753	0.6359	0.091*	
C7	0.41447 (15)	0.3608 (2)	0.27135 (14)	0.0463 (5)	
H7A	0.3985	0.4400	0.2233	0.056*	
H7B	0.4385	0.4084	0.3338	0.056*	
C8	0.5000	0.2651 (3)	0.2500	0.0448 (7)	
H8A	0.4729	0.2008	0.1950	0.054*	0.50
H8B	0.5271	0.2008	0.3050	0.054*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0414 (9)	0.0490 (9)	0.0372 (8)	−0.0040 (8)	0.0140 (7)	0.0000 (7)
N2	0.0423 (10)	0.0542 (10)	0.0480 (10)	−0.0044 (8)	0.0170 (8)	−0.0052 (8)
S1	0.0414 (4)	0.0905 (5)	0.0399 (4)	−0.0004 (3)	0.0102 (3)	0.0100 (3)
C1	0.0399 (11)	0.0473 (11)	0.0389 (10)	0.0022 (9)	0.0161 (9)	−0.0032 (8)
C2	0.0415 (11)	0.0435 (11)	0.0522 (11)	−0.0022 (9)	0.0207 (10)	−0.0030 (9)
C3	0.0520 (12)	0.0457 (11)	0.0430 (11)	−0.0003 (9)	0.0194 (10)	0.0047 (9)

C4	0.0457 (11)	0.0442 (11)	0.0407 (10)	−0.0012 (9)	0.0150 (9)	−0.0014 (8)
C5	0.0560 (14)	0.0636 (14)	0.0709 (15)	−0.0157 (12)	0.0251 (12)	−0.0003 (12)
C6	0.0615 (15)	0.0731 (16)	0.0444 (12)	−0.0164 (13)	0.0074 (11)	0.0045 (11)
C7	0.0484 (12)	0.0551 (13)	0.0386 (10)	0.0044 (10)	0.0167 (9)	0.0070 (9)
C8	0.0460 (17)	0.0470 (16)	0.0443 (16)	0.000	0.0162 (13)	0.000

Geometric parameters (Å, °)

N1—C1	1.332 (2)	C5—H5B	0.9600
N1—C4	1.349 (2)	C5—H5C	0.9600
N2—C1	1.335 (3)	C6—H6A	0.9600
N2—C2	1.346 (3)	C6—H6B	0.9600
S1—C1	1.7634 (19)	C6—H6C	0.9600
S1—C7	1.804 (2)	C7—C8	1.518 (2)
C2—C3	1.376 (3)	C7—H7A	0.9700
C2—C5	1.499 (3)	C7—H7B	0.9700
C3—C4	1.378 (3)	C8—C7^{i}	1.518 (2)
C3—H3	0.9300	C8—H8A	0.9700
C4—C6	1.494 (3)	C8—H8B	0.9700
C5—H5A	0.9600		
C1—N1—C4	115.61 (16)	H5B—C5—H5C	109.5
C1—N2—C2	115.78 (17)	C4—C6—H6A	109.5
C1—S1—C7	104.11 (10)	C4—C6—H6B	109.5
N1—C1—N2	127.70 (18)	H6A—C6—H6B	109.5
N1—C1—S1	119.80 (15)	C4—C6—H6C	109.5
N2—C1—S1	112.47 (14)	H6A—C6—H6C	109.5
N2—C2—C3	120.95 (18)	H6B—C6—H6C	109.5
N2—C2—C5	116.95 (18)	C8—C7—S1	114.38 (15)
C3—C2—C5	122.10 (19)	C8—C7—H7A	108.7
C2—C3—C4	118.99 (18)	S1—C7—H7A	108.7
C2—C3—H3	120.5	C8—C7—H7B	108.7
C4—C3—H3	120.5	S1—C7—H7B	108.7
N1—C4—C3	120.97 (18)	H7A—C7—H7B	107.6
N1—C4—C6	116.77 (17)	C7—C8—C7^{i}	111.9 (2)
C3—C4—C6	122.25 (18)	C7—C8—H8A	109.2
C2—C5—H5A	109.5	C7^{i}—C8—H8A	109.2
C2—C5—H5B	109.5	C7—C8—H8B	109.2
H5A—C5—H5B	109.5	C7^{i}—C8—H8B	109.2
C2—C5—H5C	109.5	H8A—C8—H8B	107.9
H5A—C5—H5C	109.5		
C4—N1—C1—N2	−0.8 (3)	N2—C2—C3—C4	−0.3 (3)
C4—N1—C1—S1	177.08 (14)	C5—C2—C3—C4	−179.7 (2)
C2—N2—C1—N1	0.8 (3)	C1—N1—C4—C3	0.3 (3)
C2—N2—C1—S1	−177.29 (14)	C1—N1—C4—C6	−178.60 (19)
C7—S1—C1—N1	3.68 (19)	C2—C3—C4—N1	0.2 (3)
C7—S1—C1—N2	−178.10 (15)	C2—C3—C4—C6	179.1 (2)
C1—N2—C2—C3	−0.1 (3)	C1—S1—C7—C8	81.15 (13)
C1—N2—C2—C5	179.30 (18)	S1—C7—C8—C7^{i}	167.50 (15)

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

Fig. 1

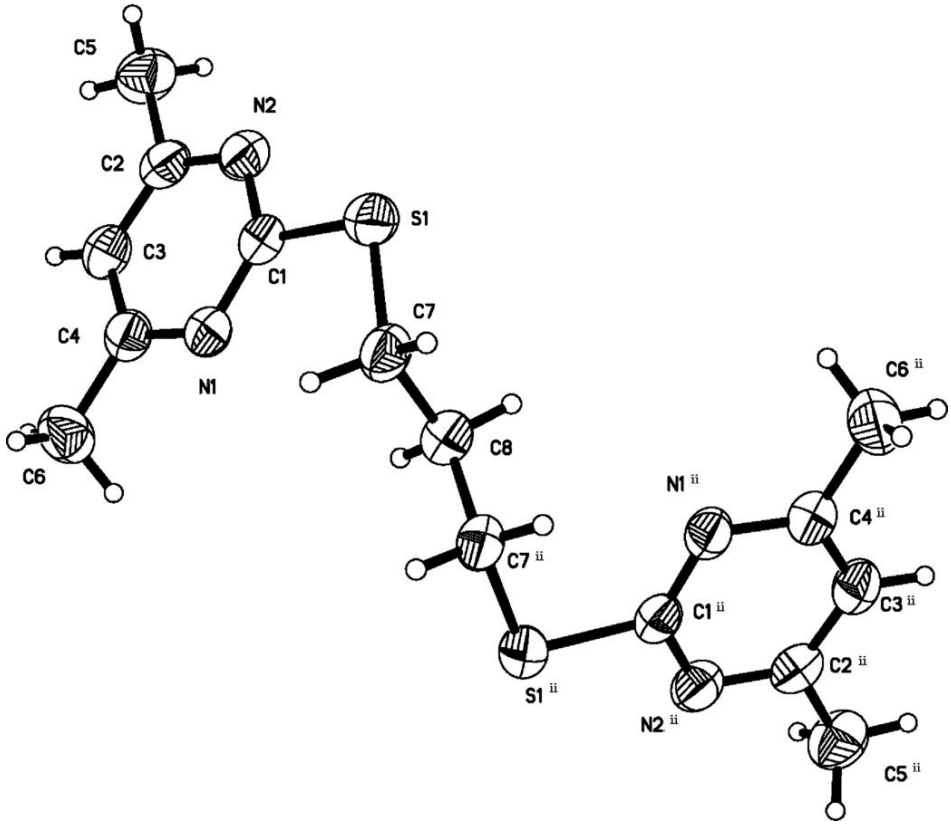

Fig. 2