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4,4′,6,6′-Tetramethyl-2,2′-(butane-1,4-diyldithio)dipyrimidine

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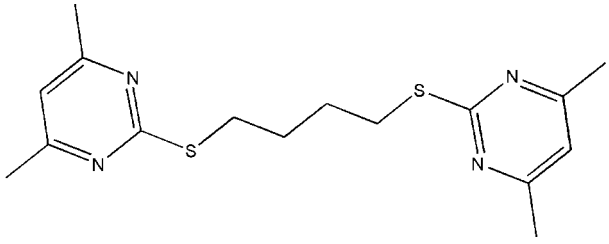
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.109; data-to-parameter ratio = 20.3.

The molecule of the title compound, $C_{16}H_{22}N_4S_2$, has a center of symmetry. The crystal packing is mainly stabilized by van der Waals forces.

Related literature

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



Experimental

Crystal data

$C_{16}H_{22}N_4S_2$
M_r = 334.50
Monoclinic, $P2_1/c$
a = 5.3851 (2) Å
b = 12.6233 (6) Å
c = 13.3282 (6) Å
β = 97.048 (3)°
V = 899.17 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 296 (2) K
0.24 × 0.17 × 0.08 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*APEX2*; Bruker, 2005)
T_{min} = 0.93, T_{max} = 0.98
4768 measured reflections
2067 independent reflections
1398 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.109
S = 1.03
2067 reflections
102 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2134).

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supplementary materials

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4,4',6,6'-Tetramethyl-2,2'-(butane-1,4-diyldithio)dipyrimidine

M. Wang, L.-H. Cheng and A.-M. Wang

Comment

Flexible thioethers are well established ligands in coordination and metallosupramolecular chemistry (Bu *et al.*, 2002), but less attention has been paid to pyrimidine dithioethers in contrast to the extensive studies on the coordination chemistry of the heterocyclic thiolates (Raper, 1997). As part of our ongoing investigtion on pyrimidine derivatives, the title compound has been prepared, and its crystal structure is presented here.

The molecule structure of the title compound is shown in Fig. 1. Bond lengths and angles are in agreement with the values reported in the literature (Allen *et al.*, 1987). The molecule has a center of symmetry. The two pyrimidine rings are planar and parallel. The crystal packing (Fig. 2) is mainly stabilized by van der Waals forces.

Experimental

For the preparation of 2,2'-(methylenedithio)bis(4,6-dimethylpyrimidine), a solution of 1,4-dibromobutane (1.08 g, 5 mmol) in ethanol (10 ml) was slowly dripped into a refluxing solution of 4,6-dimethylpyrimidine-2-thiol (1.40 g, 10 mmol) and powdered NaOH (0.4 g, 10 mmol) in ethanol. The reaction mixture was refluxed for 3 h with stirring and cooled to room temperature. The white powder of title compound was filtered and washed thoroughly with water and then air dried (yield 75%). Single crystals suitable for X-ray analysis were obtained by slow evaporation from a dichloromethane/2-propanol (5:1) 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

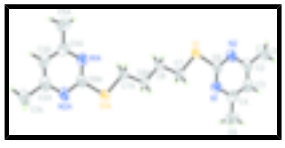

Fig. 1. The molecular structure of the title compound showing 50% probability displacement ellipsoids. Symmetry operation for C8A: $1-x,1-y,1-z$.

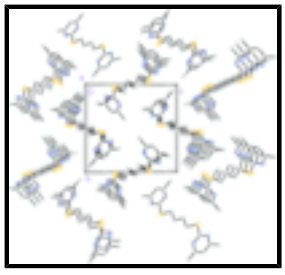

Fig. 2. Packing diagram of the title compound viewed along the *a* axis. Hydrogen atoms are omitted for clarity.

4,4',6,6'-Tetramethyl-2,2'-(butane-1,4-diyldithio)dipyrimidine

Crystal data

$C_{16}H_{22}N_4S_2$	F_{000} = 356
M_r = 334.50	D_x = 1.235 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1360 reflections
a = 5.3851 (2) Å	θ = 2.2–24.2º
b = 12.6233 (6) Å	μ = 0.30 mm^{-1}
c = 13.3282 (6) Å	T = 296 (2) K
β = 97.048 (3)º	Prism, colourless
V = 899.17 (7) Å^3	0.24 × 0.17 × 0.08 mm
Z = 2	

Data collection

Bruker APEXII CCD area-detector diffractometer	2067 independent reflections
Radiation source: fine-focus sealed tube	1398 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.022
T = 296(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (APEX2; Bruker, 2005)	h = −3→7
T_{min} = 0.93, T_{max} = 0.98	k = −16→13
4768 measured reflections	l = −17→17

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.041	H-atom parameters constrained
$wR(F^2)$ = 0.109	$w = 1/[\sigma^2(F_o^2) + (0.0474P)^2 + 0.1008P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max} < 0.001$
2067 reflections	$\Delta\rho_{max}$ = 0.17 e Å^{-3}
102 parameters	$\Delta\rho_{min}$ = −0.16 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}
C1	−0.7375 (4)	0.7125 (2)	0.12509 (18)	0.0826 (8)
H1A	−0.8199	0.6453	0.1274	0.124*
H1B	−0.6684	0.7189	0.0623	0.124*
H1C	−0.8561	0.7684	0.1303	0.124*
C2	−0.5309 (4)	0.72010 (18)	0.21157 (16)	0.0593 (6)
C3	−0.5043 (4)	0.80616 (17)	0.27491 (17)	0.0644 (6)
H3A	−0.6156	0.8626	0.2653	0.077*
C4	−0.3120 (4)	0.80799 (16)	0.35237 (16)	0.0597 (5)
C5	−0.2650 (5)	0.89945 (18)	0.4234 (2)	0.0888 (8)
H5A	−0.0991	0.9262	0.4207	0.133*
H5B	−0.2810	0.8765	0.4910	0.133*
H5C	−0.3847	0.9544	0.4043	0.133*
C6	−0.1932 (3)	0.64617 (15)	0.30250 (14)	0.0474 (5)
C7	0.2211 (3)	0.57281 (15)	0.42539 (14)	0.0468 (5)
H7A	0.3101	0.6365	0.4101	0.056*
H7B	0.1287	0.5882	0.4817	0.056*
C8	0.4073 (3)	0.48445 (14)	0.45449 (14)	0.0463 (5)
H8A	0.4976	0.4685	0.3977	0.056*
H8B	0.3180	0.4211	0.4704	0.056*
N1	−0.1516 (3)	0.72556 (12)	0.36750 (12)	0.0534 (4)
N2	−0.3725 (3)	0.63767 (13)	0.22425 (12)	0.0567 (5)
S1	0.00656 (9)	0.53510 (4)	0.31687 (4)	0.0528 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0707 (16)	0.0858 (18)	0.0830 (17)	0.0020 (13)	−0.0240 (13)	0.0300 (14)
C2	0.0504 (12)	0.0619 (14)	0.0631 (13)	0.0006 (10)	−0.0028 (10)	0.0260 (11)
C3	0.0632 (14)	0.0549 (13)	0.0734 (15)	0.0158 (11)	0.0012 (12)	0.0223 (12)
C4	0.0660 (14)	0.0503 (12)	0.0620 (13)	0.0082 (10)	0.0038 (11)	0.0121 (10)
C5	0.114 (2)	0.0588 (15)	0.0897 (19)	0.0248 (14)	−0.0033 (16)	−0.0057 (13)
C6	0.0422 (11)	0.0478 (11)	0.0512 (11)	−0.0011 (8)	0.0013 (9)	0.0101 (9)

C7	0.0406 (10)	0.0460 (10)	0.0516 (11)	0.0035 (8)	−0.0026 (8)	0.0025 (9)
C8	0.0374 (10)	0.0492 (11)	0.0513 (11)	0.0042 (8)	0.0009 (8)	−0.0004 (9)
N1	0.0521 (10)	0.0470 (9)	0.0589 (10)	0.0050 (8)	−0.0017 (8)	0.0052 (8)
N2	0.0507 (10)	0.0589 (10)	0.0566 (11)	−0.0007 (8)	−0.0085 (8)	0.0122 (8)
S1	0.0477 (3)	0.0492 (3)	0.0580 (3)	0.0055 (2)	−0.0083 (2)	−0.0025 (2)

Geometric parameters (Å, °)

C1—C2	1.503 (3)	C5—H5C	0.9600
C1—H1A	0.9600	C6—N1	1.326 (2)
C1—H1B	0.9600	C6—N2	1.336 (2)
C1—H1C	0.9600	C6—S1	1.7631 (19)
C2—N2	1.343 (2)	C7—C8	1.518 (2)
C2—C3	1.372 (3)	C7—S1	1.8009 (18)
C3—C4	1.370 (3)	C7—H7A	0.9700
C3—H3A	0.9300	C7—H7B	0.9700
C4—N1	1.352 (2)	C8—C8^{i}	1.525 (3)
C4—C5	1.495 (3)	C8—H8A	0.9700
C5—H5A	0.9600	C8—H8B	0.9700
C5—H5B	0.9600		
C2—C1—H1A	109.5	H5B—C5—H5C	109.5
C2—C1—H1B	109.5	N1—C6—N2	128.23 (17)
H1A—C1—H1B	109.5	N1—C6—S1	118.57 (14)
C2—C1—H1C	109.5	N2—C6—S1	113.20 (15)
H1A—C1—H1C	109.5	C8—C7—S1	110.46 (13)
H1B—C1—H1C	109.5	C8—C7—H7A	109.6
N2—C2—C3	121.27 (19)	S1—C7—H7A	109.6
N2—C2—C1	116.5 (2)	C8—C7—H7B	109.6
C3—C2—C1	122.3 (2)	S1—C7—H7B	109.6
C4—C3—C2	119.29 (19)	H7A—C7—H7B	108.1
C4—C3—H3A	120.4	C7—C8—C8^{i}	111.19 (19)
C2—C3—H3A	120.4	C7—C8—H8A	109.4
N1—C4—C3	120.6 (2)	C8^{i}—C8—H8A	109.4
N1—C4—C5	116.38 (19)	C7—C8—H8B	109.4
C3—C4—C5	123.0 (2)	C8^{i}—C8—H8B	109.4
C4—C5—H5A	109.5	H8A—C8—H8B	108.0
C4—C5—H5B	109.5	C6—N1—C4	115.51 (17)
H5A—C5—H5B	109.5	C6—N2—C2	115.04 (18)
C4—C5—H5C	109.5	C6—S1—C7	101.52 (9)
H5A—C5—H5C	109.5		
N2—C2—C3—C4	0.1 (3)	C5—C4—N1—C6	−178.67 (19)
C1—C2—C3—C4	179.9 (2)	N1—C6—N2—C2	−0.9 (3)
C2—C3—C4—N1	−0.9 (3)	S1—C6—N2—C2	179.09 (14)
C2—C3—C4—C5	178.5 (2)	C3—C2—N2—C6	0.7 (3)
S1—C7—C8—C8^{i}	179.27 (17)	C1—C2—N2—C6	−179.14 (17)
N2—C6—N1—C4	0.2 (3)	N1—C6—S1—C7	−0.55 (17)
S1—C6—N1—C4	−179.78 (15)	N2—C6—S1—C7	179.49 (13)
C3—C4—N1—C6	0.7 (3)	C8—C7—S1—C6	177.54 (13)

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

Fig. 1

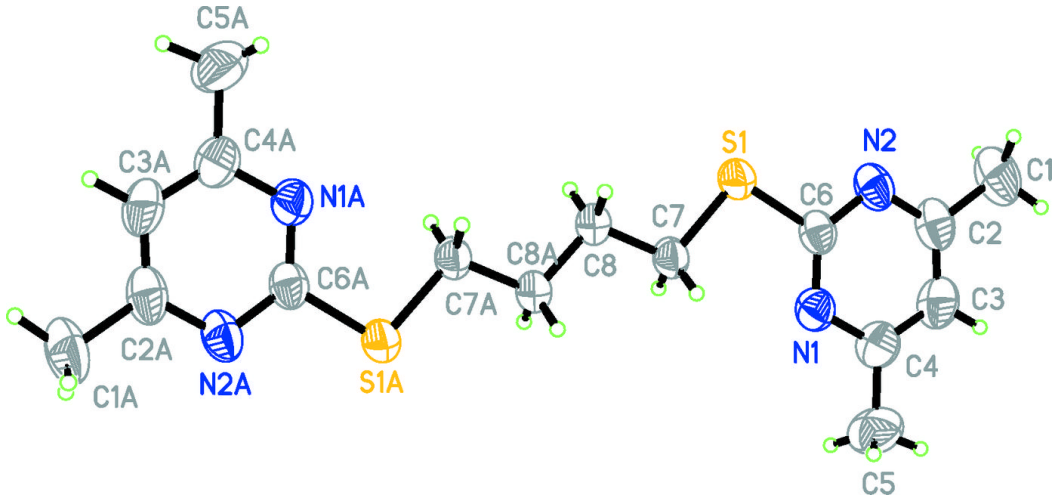

Fig. 2