Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

5-Benzyl-1,3,5-trimethylpyrimidine-2,4,6(1*H*,3*H*,5*H*)-trithione

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Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.002 Å
R factor = 0.028
wR factor = 0.089
Data-to-parameter ratio = 17.1

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

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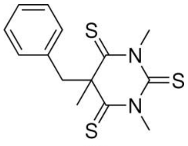
Received 27 December 2006
Accepted 9 January 2007

The rings in the title compound, $C_{14}H_{16}N_2S_3$, adopt a *syn* conformation with respect to each other. The pyrimidinetrithione ring system exhibits conjugation involving the lone pairs on the N atoms and the three C=S bonds. Intermolecular C—H···S interactions and S···S contacts help stabilize the crystal packing.

Comment

Barbiturates act as central nervous system depressants and produce a wide spectrum of effects, from mild sedation to anesthesia (Bideau *et al.*, 1976; Gotthardt *et al.*, 1980). Some are also used as anticonvulsants. Although a number of barbiturates have been studied, there have been only a few studies of their sulfur analogs (trithiobarbiturate) (Greenberg *et al.*, 2000) and while most crystals of barbiturates are colorless those of thiobarbiturates, such as (I), are usually colored.



(I)

In (I), shown in Fig. 1, the C—N bond lengths (Table 1) are shorter than the value of 1.416 Å (Allen *et al.*, 1987) usually observed for Csp^2—Nsp^3 bonds, indicating that the lone pairs on N1 and N2 are conjugated with the three C=S bonds. The heterocyclic six-membered ring has a flattened boat conformation. The pyrimidinetrithione group and the phenyl ring are in a *syn* conformation with respect to each other. There are some close intermolecular S···S contacts in (I); S1···S2^{i} and S2···S1^{ii} at 3.5076 (4) Å [symmetry codes: (i) x, $1 + y$, z; (ii) x, $y - 1$, z] lie within the range of S···S contacts (3.35–3.65 Å) found in similar compounds (Dai *et al.*, 1997; Zhou *et al.*, 2001). Some intermolecular C—H···S interactions are also observed in (I) (Table 2). These S···S contacts and C—H···S interactions help stabilize the crystal packing (Fig. 2).

Experimental

Compound (I) was synthesized by using 1,3-dimethyl-2-thiourea and benzylmethylmalonyl chloride in triethylamine (Takechi *et al.*, 1999) to produce dihydro-1,3,5-trimethyl-5-(phenylmethyl)-2-thioxo-4,6(1*H*,5*H*)-pyrimidinedione, (II). Reflux of (II) (15 mmol) and Lawesson's reagent (9.7 g, 24 mmol) in xylene (40 ml) for 24 h afforded compound (I) in 24% yield. Crystals of (I) were grown from a chloroform solution by slow evaporation.

doi:10.1107/S1600536807000888

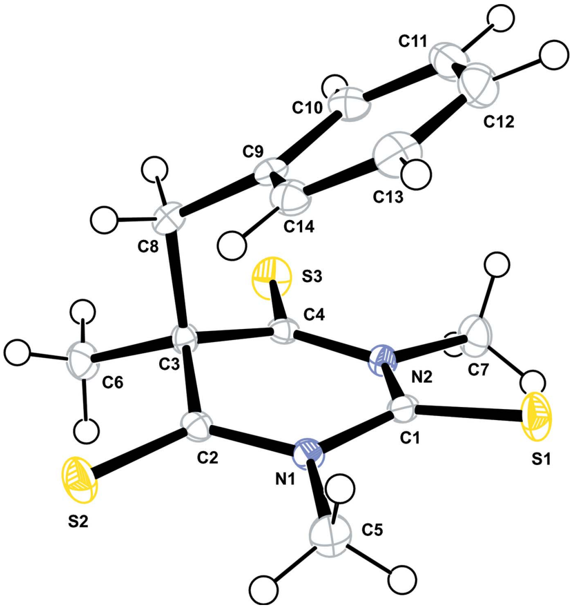


Figure 1
Molecular structure of (I), showing 50% probability displacement ellipsoids.

Crystal data

$C_{14}H_{16}N_2S_3$
$M_r = 308.47$
Monoclinic, $P2_1/a$
$a = 13.112$ (13) Å
$b = 8.350$ (8) Å
$c = 13.299$ (13) Å
$\beta = 90.721$ (7)°
$V = 1456$ (2) Å^3
$Z = 4$
$D_x = 1.407$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.50$ mm^{-1}
$T = 123.1$ K
Prism, red
$0.15 \times 0.13 \times 0.10$ mm

Data collection

Rigaku Saturn diffractometer
ω scans
Absorption correction: multi-scan (*REQAB*; Jacobson, 1998)
$T_{\min} = 0.891$, $T_{\max} = 0.952$
16694 measured reflections
3216 independent reflections
2731 reflections with $F^2 > 2\sigma(F^2)$
$R_{\rm int} = 0.035$
$\theta_{\max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.089$
$S = 1.06$
3216 reflections
188 parameters
H-atom parameters constrained
$w = 1/[0.0007F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.40$ e Å^{-3}
$\Delta\rho_{\min} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.6447 (13)	N2—C1	1.3941 (17)
S2—C2	1.6436 (13)	N2—C4	1.3658 (16)
S3—C4	1.6455 (13)	N2—C7	1.4745 (17)
N1—C1	1.3897 (16)	C2—C3	1.5162 (18)
N1—C2	1.3757 (16)	C3—C4	1.5182 (18)
N1—C5	1.4736 (17)		
C5—N1—C1—S1	−11.48 (16)	S2—C2—C3—C8	80.73 (12)
C5—N1—C2—S2	−2.78 (16)	C2—C3—C8—C9	64.59 (13)
C7—N2—C1—S1	10.58 (16)	C4—C3—C8—C9	−57.31 (14)
C7—N2—C4—S3	3.69 (16)	C6—C3—C8—C9	−176.19 (11)
S2—C2—C3—C6	−35.98 (15)		

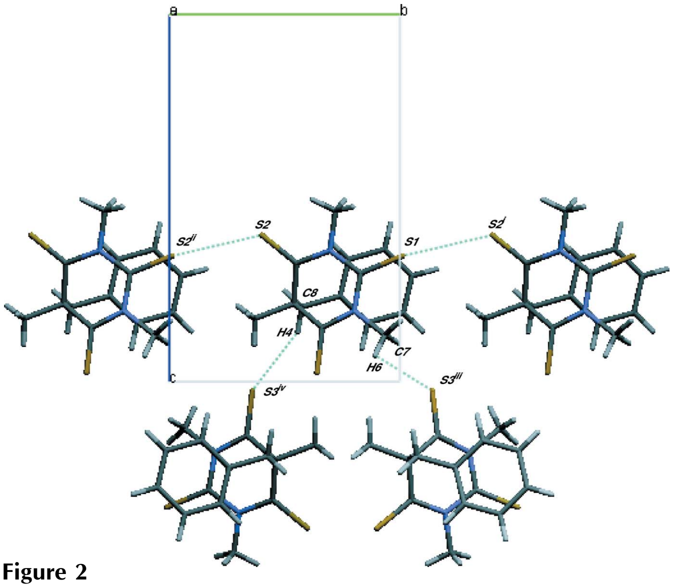


Figure 2
Intermolecular C—H···S interactions and S···S contacts of (I). Broken lines indicate these interactions. [Symmetry codes: (i) x, $1 + y$, z; (ii) x, $y - 1$, z; (iii) $\frac{3}{2} - x$, $\frac{1}{2} + y$, $2 - z$; $2 - x$, $1 - y$, $2 - z$.]

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C7—H6···S3$^{\rm iii}$	0.95	2.95	3.415 (2)	111
C8—H4···S3$^{\rm iv}$	0.95	2.88	3.749 (4)	153

Symmetry codes: (iii) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + 2$; (iv) $-x + 2, -y + 1, -z + 2$.

H atoms bonded to C atoms were included in the refinement at calculated positions and refined with a riding model, with C—H = 0.95 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *Mercury* (Version 1.3; Bruno *et al.*, 2002); software used to prepare material for publication: *CrystalStructure*.

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