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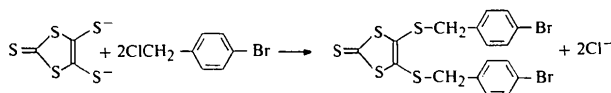
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tions, we have synthesized BBBT-DTT (Fang *et al.*, 1995) according to the reaction scheme below. The structure of BBBT-DTT is reported here.



BBBT-DTT contains three non-coplanar planes. The maximum deviation of the DMIT atoms from their least-squares plane is 0.036 Å (for atoms S2 and S3) and the atoms of each bromobenzyl moiety are coplanar, with maximum deviations of 0.008 Å (for atoms C8 and C11) and 0.027 Å (for atom C12). The terminal C=S double bond [1.62 (1) Å] is quite short compared to that of other

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4,5-Bis(4-bromobenzylthio)-1,3-dithiole-2-thione

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Abstract

The title compound, BBBT-DTT (C₁₇H₁₂Br₂S₅), consists of one DMIT (4,5-dimercapto-1,3-dithiole-2-thione) and two *p*-bromobenzyl moieties. The molecular packing is characterized by S···S (head-to-head) and Br···Br (tail-to-tail) intermolecular interactions, with intermolecular distances S2···S2(−*x*, −*y*, 1−*z*) 3.36 (1) and Br···Br(−2−*x*, 1−*y*, −*z*) 3.68 (1) Å.

Comment

The products of the nucleophilic reaction between 4,5-dimercapto-1,3-dithiole-2-thione (DMIT) and various halohydrocarbons are the key precursors for the synthesis of DMIT-based molecular electrical conductors, which form a large family of molecular conductors (Williams *et al.*, 1987; Bryce, 1991). Recently, it has been reported that the reaction between DMIT and 2,4-dinitrochlorobenzene led to the nonlinear optical material β-BNPT-DTO (Fang *et al.*, 1994). Both DMIT-based molecular conductors and DMIT-based nonlinear optical materials are characterized by relatively strong S···S intermolecular interactions. In the course of exploring new products of the above nucleophilic reac-

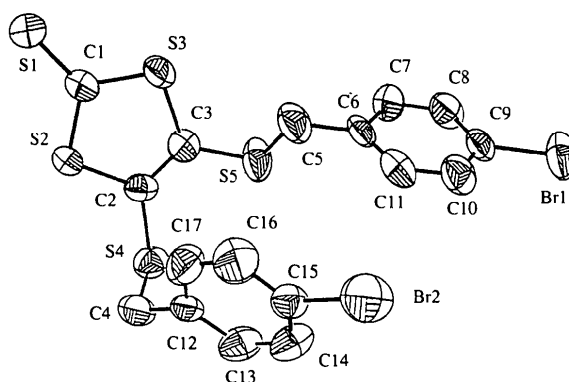


Fig. 1. Molecular structure showing 50% probability displacement ellipsoids. H atoms are omitted for clarity.

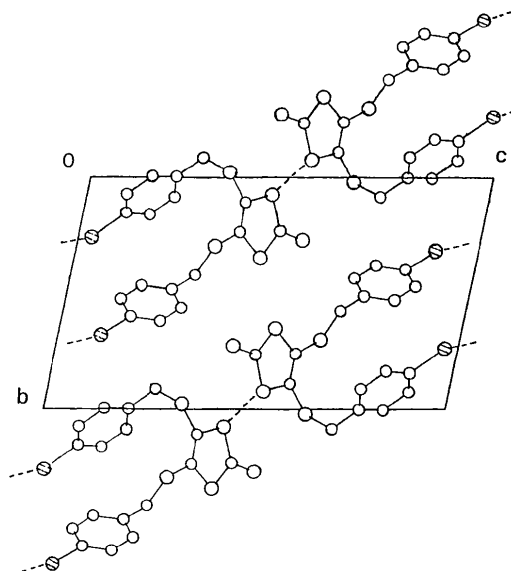


Fig. 2. Packing diagram viewed down the *a* axis. Br atoms are hatched and intermolecular interactions referred to in the text are shown as dashed lines.

DMIT derivatives (1.63–1.67 Å), but is still longer than the typical C=S bond length. The other C—S bonds are primarily single, with some double-bond character, so that the DMIT moiety has a relatively high degree of conjugation. The C=C bonds of the two phenyl rings are slightly shorter than the usual C=C benzene bond lengths, while S5—C5, C5—C6, S4—C4 and C4—C12 are all single bonds.

A number of intermolecular distances shorter than the sum of van der Waals radii are observed: S2···S2(−x, −y, 1−z) 3.36(1), S2···S1(−x, −y, 1−z) 3.65(1) and Br2···Br1(−2−x, 1−y, −z) 3.68(1) Å. BBT-DTT consists, therefore, of one-dimensional chains formed by (BrPhCH₂)₂DMIT···DMIT(CH₂PhBr)₂ and DMIT(CH₂PhBr)₂···(BrPhCH₂)₂DMIT (head-to-head through S···S and tail-to-tail through Br···Br) intermolecular interactions.

Experimental

The title compound was synthesized by Fang *et al.* (1995). The crystal used for the structure determination was cut from a long needle-shaped crystal which was obtained by evaporating slowly a BBT-DTT/CH₃CN solution containing equimolar tetracyanoquinodimethane (TCNQ) at room temperature.

Crystal data

C₁₇H₁₂Br₂S₅
M_r = 536.42
 Triclinic
*P*1̄
a = 5.487(3) Å
b = 10.510(4) Å
c = 17.796(9) Å
 α = 100.61(4)°
 β = 96.03(4)°
 γ = 97.23(4)°
V = 992(1) Å³
Z = 2
D_x = 1.80 Mg m^{−3}

Data collection

Nicolet R3m/E diffractometer
 $\omega/2\theta$ scans
 Absorption correction:
 ψ scans (North, Phillips & Mathews, 1968)
T_{min} = 0.288, *T_{max}* = 0.418
 4023 measured reflections
 3940 independent reflections

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 25 reflections
 θ = 3.0–12.0°
 μ = 4.83 mm^{−1}
T = 293 K
 Parallelepiped
 0.3 × 0.2 × 0.2 mm
 Orange

1798 observed reflections
 $[F > 4\sigma(F)]$
 $R_{\text{int}} = 0.0212$
 $\theta_{\text{max}} = 25^\circ$
 $h = 0 \rightarrow 7$
 $k = -13 \rightarrow 13$
 $l = -22 \rightarrow 22$
 2 standard reflections monitored every 100 reflections
 intensity decay: <5%

Refinement

Refinement on *F*
R = 0.076

$w = 1/[\sigma^2(F) + 0.002F^2]$
 $(\Delta/\sigma)_{\text{max}} = 0.04$

$wR = 0.074$
 $S = 1.358$
 1798 reflections
 217 parameters
 H-atom parameters not refined

$\Delta\rho_{\text{max}} = 0.87 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.99 \text{ e \AA}^{-3}$
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Br1	−1.1777(3)	0.6822(2)	0.1061(1)	0.087(1)
Br2	−0.3199(4)	0.2617(2)	0.0327(1)	0.103(1)
S1	0.1817(6)	0.2727(3)	0.5588(2)	0.068(1)
S2	−0.2157(6)	0.0761(3)	0.4601(2)	0.062(1)
S3	−0.2308(5)	0.3471(3)	0.4632(2)	0.062(1)
S4	−0.6638(5)	−0.0216(3)	0.3440(2)	0.061(1)
S5	−0.6868(6)	0.2984(3)	0.3454(2)	0.080(2)
C1	−0.071(2)	0.2345(10)	0.4981(6)	0.054(4)
C2	−0.453(2)	0.1099(10)	0.3981(6)	0.049(4)
C3	−0.461(2)	0.2387(10)	0.3995(6)	0.050(4)
C4	−0.480(2)	−0.0853(11)	0.2711(6)	0.066(5)
C5	−0.509(2)	0.4228(12)	0.3104(8)	0.076(6)
C6	−0.670(2)	0.4861(9)	0.2599(7)	0.053(4)
C7	−0.833(2)	0.5646(11)	0.2924(7)	0.069(5)
C8	−0.984(2)	0.6214(11)	0.2469(7)	0.069(5)
C9	−0.973(2)	0.6024(10)	0.1708(7)	0.054(5)
C10	−0.816(2)	0.5272(11)	0.1378(7)	0.068(5)
C11	−0.670(2)	0.4688(11)	0.1817(7)	0.064(5)
C12	−0.441(2)	−0.0024(10)	0.2132(6)	0.048(4)
C13	−0.624(2)	−0.0048(12)	0.1532(7)	0.070(5)
C14	−0.590(2)	0.0742(13)	0.0991(7)	0.074(5)
C15	−0.371(2)	0.1558(11)	0.1067(6)	0.054(5)
C16	−0.191(2)	0.1638(13)	0.1662(8)	0.079(6)
C17	−0.225(2)	0.0870(12)	0.2190(7)	0.070(5)

Table 2. Selected geometric parameters (Å, °)

Br1—C9	1.902(12)	S5—C5	1.778(14)
S2—C1	1.732(10)	C5—C6	1.495(18)
S3—C3	1.743(9)	C14—C15	1.363(17)
S5—C3	1.740(12)	S1—C1	1.623(11)
C4—C12	1.483(16)	S3—C1	1.725(12)
C9—C10	1.346(17)	S4—C4	1.808(13)
Br2—C15	1.895(12)	C2—C3	1.355(15)
S2—C2	1.733(11)	C8—C9	1.341(18)
S4—C2	1.746(9)	C15—C16	1.354(17)
C1—S2—C2	99.1(5)	C1—S3—C3	98.7(5)
C2—S4—C4	100.6(5)	C3—S5—C5	102.2(6)
S1—C1—S2	124.7(7)	S1—C1—S3	124.2(6)
S2—C1—S3	111.1(6)	S2—C2—S4	118.0(6)
S2—C2—C3	115.3(7)	S4—C2—C3	126.7(8)
S3—C3—S5	120.0(6)	S3—C3—C2	115.7(8)
S5—C3—C2	124.3(7)	S4—C4—C12	114.1(8)
S5—C5—C6	111.0(8)		

All H atoms were fixed at ideal positions with common isotropic displacement parameters ($U_{\text{iso}} = 0.08 \text{ \AA}^2$). The relatively high *R* value is believed to be a result of the poor quality of the crystal.

Data collection and cell refinement used Nicolet R3m/E software (Nicolet Instrument Corporation, 1985). Structure solution and refinement were performed using *SHELXTL* (Sheldrick, 1985), which was also used to produce the molecular graphics.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data, and packing diagrams viewed down the *b* and *c* axes have been deposited with the IUCr (Reference: TA1027). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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7-[3-(Dibenzylamino)-2-hydroxypropyl]-8-(furfurylamino)theophylline

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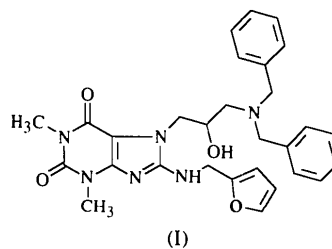
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Abstract

The theophylline moiety of the title compound, 7-[3-(dibenzylamino)-2-hydroxypropyl]-8-(furfurylamino)-3,7-dihydro-1,3-dimethyl-1*H*-purine-2,6-dione, C₂₉H₃₂N₆O₄, is planar. The conformation of the 7-aminohydroxyalkyl substituent may be influenced by an O—H···N intramolecular hydrogen bond and the structure is stabilized by an N—H···O intermolecular bond.

Comment

As part of our studies of the structures and pharmacological properties of 7,8-disubstituted derivatives of theophylline (Karolak-Wojciechowska & Pawłowski, 1990; Karczmarzyk, Karolak-Wojciechowska & Pawłowski, 1991, 1995), we report here the results of an X-ray structure determination of the title compound, (I), which shows potent antihypertensive and vasodilating activity. A tertiary N atom with two benzyl groups present in the substituent at the 7 position may be responsible for this pharmacological effect (Olejnik *et al.*, 1989).



Both rings of the theophylline moiety are planar: to within 0.028 (2) Å for the six-membered ring and 0.010 (2) Å for the five-membered ring. They are inclined at 1.3 (1)° with respect to each other. The amino group of the 8-furfurylamino substituent is conjugated with the π -electron system of the imidazole ring [N8—C8 = 1.347 (3) Å] and the sum of valence angles around C8 is 356 (3)°.

One particular reason for the present study was the determination of the conformation of the amino-hydroxyalkyl substituent at the 7 position of the molecule. The conformation of the 2-hydroxyamino-propyl side chain is *gauche-gauche-trans-gauche*. The orientation of the hydroxy group with respect to the N7—C7 and C12—N13 bonds is *trans-gauche*. The conformation of the substituent is stabilized by an O11—H11O···N13 intramolecular hydrogen bond [O11···N13 2.822 (3), H11O···N13 2.29 (4) Å and O11—H11O···N13 129 (5)°]; this type of hydrogen bond has also been observed in a similar 7,8-disubstituted derivative of theophylline (Karczmarzyk, Karolak-Wojciechowska & Pawłowski, 1995). The phenyl rings are planar; their least-squares planes are inclined by 53.4 (1)° with respect to each other and by 78.7 (1) and 28.0 (1)° with respect to the plane of the theophylline moiety. The position of the benzyl substituents with respect to the C12—N13 bond is given by the torsion angles C12—N13—C21—C22 = 166.2 (2) and C12—N13—C31—C32 = -74.7 (3)°.

The furfurylamino group in position 8 possesses a *trans-gauche-gauche* conformation. The geometry of the furan ring does not show any unusual features (Galešić, Vlahov & Galešić, 1987).