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# 2,2′-(1,4-Butanediyldithio)-1,3-dithiazole

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**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.003 Å
*R* factor = 0.030
*wR* factor = 0.076
Data-to-parameter ratio = 18.1

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



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The title compound, $C_{10}H_{14}N_2S_4$, was synthesized by the reaction of the potassium salt of 2-thiothiazole and 1,4-dibromobutane. The two 2-thiazolethio groups are related by a center of symmetry. The plane defined by the butyl carbon chain is approximately orthogonal to the thiazole plane, with a dihedral angle of 84.7 (8)°. Intermolecular S···S interactions between adjacent molecules link them into infinite chains running along the *c* axis.

## Comment

It is well known that thiazole and its derivatives exhibit remarkable bioactivities, such as antibacterial activity (Chang *et al.*, 1982), antiphlogistic activity (Garbarczyk *et al.*, 1999) and antitumor activities El-Subbagh & Al-Obaid, 1996), as well as cytotoxic activities against a variety of human cancer cell lines *in vitro* (Gu *et al.*, 1999). This prompted us to explore new compounds containing two or more thiazole groups. We report here a new thiazole derivative, namely 2,2′-(1,4-Butanediyldithio)-1,3-dithiazole, (I).

(I)

The molecule of (I) is centrosymmetric (Fig. 1). The two thiazole rings, related by the centre of symmetry at the mid-point of the C5—C5*A* bond, lie on opposite sides of the zigzag 1,4-butanediyl chain. The C3—S1 bond length [1.752 (2) Å] is significantly shorter than C4—S1 [1.819 (2) Å] due to $p$-$\pi$ conjugation, similar to that observed in 2,2′-[1,4-phenylenebis(methylenethio)]dithiazole (Zhang *et al.*, 2003). The shortening of C3—S1 relative to C4—S1 is not unexpected, reflecting the difference between a C$sp^2$—S bond and a C$sp^3$—S bond. Exocyclic atom S1 is almost coplanar with the bonded thiazole ring, the deviation being 0.0499 (3) Å.

Intermolecular S···S interactions between S atoms belonging to two parallel molecules are observed, as can be seen in the packing diagram shown in Fig. 2. The S1···S1$^{i}$ and S1···S2$^{i}$ interactions [3.536 (3) and 3.542 (4) Å; symmetry code (i): $-x$, $-y$, $2-z$] link the molecules in the crystal structure into infinite chains running along the *c* axis.

## Experimental

The title compound was synthesized according to the literature method of Zhang *et al.* (2003). To a solution containing 0.5 g (4.3 mmol) of 2-thiothiazole, 0.24 g (4.3 mmol) of KOH and 3 ml of ethanol at 323–333K were added dropwise; 0.46 g (2.1 mmol) of 1,4-dibromobutane in 2 ml ethanol was added with stirring. The reaction mixture was then stirred at the same temperature for 24 h. The

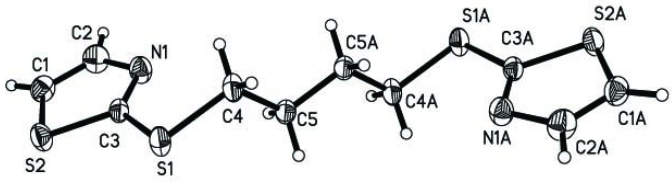


**Figure 1**
The molecular structure of (I), showing 30% probability displacement ellipsoids. H atoms are drawn as small spheres of arbitrary radii.

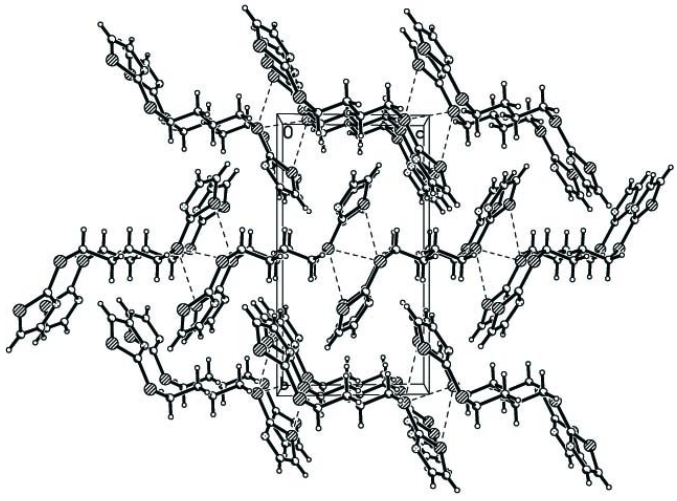

**Figure 2**
The packing diagram for (I), viewed along the *a* axis. The dashed lines show the short contacts between neighboring molecules.

precipitate was filtered off and washed with ethanol. The filtrate was concentrated and left in the ambient atmosphere to give colorless crystals of (I) suitable for X-ray analysis, in a yield of 64.6% (m.p. 333–334 K). IR (KBr) 3116 (*w*), 3086 (*w*), 2943 (*w*), 2867 (*w*), 1477 (*ms*), 1386 (*s*), 1294 (*w*), 1042 (*s*), 1019 (*s*), 726 (*ms*) $cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta$ 1.95–1.92 (4H, *m*), 3.26 (4H, *s*), 7.22 (4H, *d*, *J* = 3.2 Hz), 7.67–7.66 (2H, *d*, *J* = 2.8 Hz) p.p.m.

*Crystal data*

| | |
|---|---|
| $C_{10}H_{12}N_2S_4$ | $D_x$ = 1.459 Mg $m^{-3}$ |
| $M_r$ = 288.46 | Mo $K\alpha$ radiation |
| Monoclinic, $P2_1/n$ | Cell parameters from 754 reflections |
| $a$ = 6.067 (2) Å | $\theta$ = 3.0–25.9° |
| $b$ = 14.099 (5) Å | $\mu$ = 0.70 $mm^{-1}$ |
| $c$ = 7.733 (3) Å | $T$ = 293 (2) K |
| $\beta$ = 97.101 (6)° | Needle, colorless |
| $V$ = 656.4 (4) $Å^3$ | 0.56 × 0.32 × 0.20 mm |
| $Z$ = 2 | |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 1336 independent reflections |
| $\varphi$ and $\omega$ scans | 1021 reflections with $I > 2\sigma(I)$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 1997) | $R_{int}$ = 0.027 |
| $T_{min}$ = 0.772, $T_{max}$ = 0.870 | $\theta_{max}$ = 26.4° |
| 3664 measured reflections | $h = -4 \rightarrow 7$ |
| | $k = -17 \rightarrow 17$ |
| | $l = -9 \rightarrow 9$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0344P)^2 + 0.1369P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)]$ = 0.030 | $(\Delta/\sigma)_{max} < 0.001$ |
| $wR(F^2)$ = 0.076 | $\Delta\rho_{max}$ = 0.20 e $Å^{-3}$ |
| $S$ = 1.04 | $\Delta\rho_{min}$ = −0.18 e $Å^{-3}$ |
| 1336 reflections | |
| 74 parameters | |
| H-atom parameters constrained | |

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| S1—C3 | 1.752 (2) | S2—C3 | 1.7413 (18) |
| S1—C4 | 1.819 (2) | N1—C3 | 1.297 (3) |
| S2—C1 | 1.717 (2) | N1—C2 | 1.394 (3) |
| | | | |
| C3—S1—C4 | 101.28 (10) | C3—N1—C2 | 109.12 (17) |
| C1—S2—C3 | 88.91 (10) | C2—C1—S2 | 109.93 (17) |

The H atoms were included in calculated positions nand refined with riding-model constraints.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997) and *SHELXTL* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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