with the values observed in three related structures: Ishida, Yamashita, Takai & Inoue (1983), Bechtel, Bideau & Cotrait (1979) and Lovell & Perkinson (1978).

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# Sequential Reaction of 2-Lithio-2-trimethylsilyl-1,3-dithiane with Dimethylformamide and Ammonia: Structure of Bis[(1,3-dithian-2-ylidene)methyl]amine


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**Abstract.** $C_{10}H_{15}NS_4$, $M_r = 277{\cdot}48$, monoclinic, $P2_1/c$, $a = 12{\cdot}522\ (3)$, $b = 17{\cdot}81\ (1)$, $c = 6{\cdot}140\ (1)$ Å, $\beta = 100{\cdot}41\ (1)°$, $V = 1346{\cdot}7$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}37$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71069 Å, $\mu = 6{\cdot}48$ cm$^{-1}$, $F(000) = 584$, room temperature, $R = 0{\cdot}038$ for 1492 observed reflections. The bridging C=C—N—C=C group atoms are planar to within 0·014 Å; the C—N—C bond angle is 125°. The S—C distances range from 1·759 to 1·824 Å.


**Experimental.** Compound isolated unexpectedly (in 50% yield) from reaction between 2-lithio-2-trimethylsilyl-1,3-dithiane (generated from 2-trimethylsilyl-1,3-dithiane and *n*-butyllithium) and dimethylformamide following work-up with aqueous ammonium chloride solution. Crystals obtained by recrystallization from ethanol. Large block cut to *ca* 0·35 × 0·3 × 0·3 mm and mounted on a glass fibre; Stoe Stadi-2 2-circle diffractometer, 18 centred reflections ($3{\cdot}3 < \theta < 17{\cdot}3°$), graphite-monochromated Mo $K\alpha$; for data collection: $\theta_{max} = 25°$, $\omega$ scans in 0·01° steps, $\omega$-scan width $(2{\cdot}00 + 0{\cdot}6 \sin\mu/\tan\theta)°$, $h$ 0 to 15, $k$ 0 to 21, $l$ −7 to 7 (collected in layers of $k$), intensity-control reflections (one per layer) varied ±3%, yielding 2383 unique reflections of which 1492 with $F > 6\sigma(F)$ were retained. Space group $P2_1/c$ from systematic absences ($h0l$: $l = 2n$; $0k0$: $k = 2n$).

Atoms located by automatic direct methods (*SHELXS*86; Sheldrick, 1986) followed by difference-Fourier syntheses with full-matrix, least-squares refinement (*SHELX*76; Sheldrick, 1976), $w = [\sigma^2(F) + 0{\cdot}000164\ F^2]^{-1}$, anisotropic thermal parameters for all non-H atoms. H atoms were located from difference Fourier synthesis but in the later stages of refinement were constrained at fixed bond distances (C—H, 1·00 Å; N—H, 0·95 Å) and were refined with a common isotropic thermal parameter (0·0791 Å$^2$ at convergence). $R = 0{\cdot}038$, $wR = 0{\cdot}035$, $S = 1{\cdot}192$, data:variable = 7·3:1, $(\Delta\rho)_{max} = 0{\cdot}22$, $(\Delta\rho)_{min} = -0{\cdot}20$ e Å$^{-3}$. Max $\Delta/\sigma$ in final cycle was 0·018. Scattering factors were from *SHELX*76. Other programs used were *CHEM*3*D* and *CALC* (Gould, 1984).

Atomic parameters are listed in Table 1 and bond lengths and angles in Table 2.* The molecule and numbering scheme are shown in Fig. 1. Atoms C(4), C(8), C(9), C(10), and N(1) are planar to within 0·014 Å; deviations from the best plane are as follows: C(4), −0·012 (3); C(8), 0·002 (4); C(9), 0·014 (3); C(10), −0·014 (4); N(1), 0·009 (3) Å.

**Related literature.** The compound $C_{10}H_{15}NS_4$ is a dienamine in which the $\beta$-carbon atoms of the vinyl


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* Lists of structure factors, anisotropic thermal parameters, full bond lengths, angles, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53178 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional atomic coordinates (ring H atoms not included) for* $\{(C_4H_6S_2)CH\}_2NH$

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

| | $x$ | $y$ | $z$ | $U_{eq}/U_{iso}$(Å$^2$) |
|---|---|---|---|---|
| S(1) | 0·1394 (1) | 0·4918 (1) | 0·0505 (2) | 0·0659 (6) |
| S(2) | 0·3781 (1) | 0·4726 (1) | 0·0390 (2) | 0·0637 (6) |
| S(3) | 0·0920 (1) | 0·1627 (1) | 0·6286 (2) | 0·0770 (7) |
| S(4) | −0·0350 (1) | 0·3046 (1) | 0·4821 (2) | 0·0662 (6) |
| C(1) | 0·1312 (4) | 0·5009 (2) | −0·2469 (7) | 0·0727 (29) |
| C(2) | 0·2322 (4) | 0·5358 (2) | −0·3110 (7) | 0·0729 (27) |
| C(3) | 0·3321 (4) | 0·4861 (2) | −0·2576 (7) | 0·0730 (27) |
| C(4) | 0·2588 (3) | 0·4370 (2) | 0·1140 (6) | 0·0532 (21) |
| C(5) | 0·0611 (4) | 0·1896 (2) | 0·8951 (7) | 0·0766 (29) |
| C(6) | −0·0451 (4) | 0·2301 (2) | 0·8795 (7) | 0·0786 (30) |
| C(7) | −0·0485 (4) | 0·3073 (2) | 0·7705 (7) | 0·0763 (29) |
| C(8) | 0·0869 (3) | 0·2528 (2) | 0·5056 (6) | 0·0602 (22) |
| C(9) | 0·2598 (3) | 0·3735 (2) | 0·2292 (6) | 0·0539 (22) |
| H(13) | 0·3275 (16) | 0·3446 (16) | 0·2771 (52) | 0·0791 (30) |
| C(10) | 0·1714 (3) | 0·2788 (2) | 0·4237 (6) | 0·0581 (23) |
| H(14) | 0·2391 (16) | 0·2484 (16) | 0·4461 (54) | 0·0791 (30) |
| N(1) | 0·1722 (2) | 0·3457 (2) | 0·3114 (5) | 0·0605 (19) |
| H(15) | 0·1104 (17) | 0·3765 (15) | 0·2759 (56) | 0·0791 (30) |

Table 2. *Bond lengths* (Å) *and angles* (°) *for* $\{(C_4H_6S_2)CH\}_2NH$

| | | | | | |
|---|---|---|---|---|---|
| S(1) | C(1) | 1·817 (5) | C(1) | C(2) | 1·522 (6) |
| S(1) | C(4) | 1·769 (4) | C(2) | C(3) | 1·519 (6) |
| S(2) | C(3) | 1·824 (5) | C(4) | C(9) | 1·332 (5) |
| S(2) | C(4) | 1·760 (4) | C(5) | C(6) | 1·501 (6) |
| S(3) | C(5) | 1·813 (5) | C(6) | C(7) | 1·526 (6) |
| S(3) | C(8) | 1·769 (4) | C(8) | C(10) | 1·333 (5) |
| S(4) | C(7) | 1·810 (5) | C(9) | N(1) | 1·379 (5) |
| S(4) | C(8) | 1·767 (4) | C(10) | N(1) | 1·378 (5) |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| C(1) | S(1) | C(4) | 99·4 (2) | S(3) | C(5) | C(6) | 113·6 (3) |
| C(3) | S(2) | C(4) | 100·3 (2) | C(5) | C(6) | C(7) | 114·5 (4) |
| C(5) | C(3) | C(8) | 98·6 (2) | S(4) | C(7) | C(6) | 114·0 (3) |
| C(7) | S(4) | C(8) | 99·5 (2) | S(3) | C(8) | S(4) | 118·2 (2) |
| S(1) | C(1) | C(2) | 113·4 (3) | S(3) | C(8) | C(10) | 120·4 (3) |
| C(1) | C(2) | C(3) | 113·4 (4) | S(4) | C(8) | C(10) | 121·3 (3) |
| S(2) | C(3) | C(2) | 113·1 (3) | C(4) | C(9) | N(1) | 124·6 (3) |
| S(1) | C(4) | S(2) | 117·8 (2) | C(8) | C(10) | N(1) | 124·6 (3) |
| S(1) | C(4) | C(9) | 120·7 (3) | C(9) | N(1) | C(10) | 125·0 (3) |
| S(2) | C(4) | C(9) | 121·2 (3) | | | | |

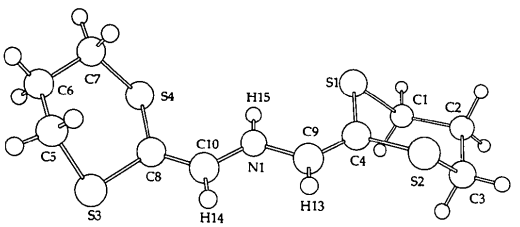


Fig. 1. A view of the molecule drawn with *CHEM3D*.

groups occupy the 2-position of 1,3-dithianyl rings. Although 56 compounds containing the enamine moiety are listed in the Cambridge Crystallographic Database, only one (Neunhoffer & Metz, 1983) contains the dienamine moiety.

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# Redetermination of the Structure of Bi(9,10-dihydro-9,10-anthracenediyl)* at 198 K

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**Abstract.** $C_{28}H_{20}$, $M_r = 356·4$, orthorhombic, *Pbca*, $a = 8·101$ (1), $b = 12·034$ (2), $c = 18·753$ (6) Å, $V = 1828·1$ (7) Å$^3$, $Z = 4$ (the asymmetric unit is half a molecule), $D_x = 1·295$ g cm$^{-3}$, Mo $K\alpha$ ($\lambda = 0·71073$ Å), $\mu = 0·68$ cm$^{-1}$, $F(000) = 752$, $T = 198$ K, $R = 0·0416$ and $wR = 0·0442$ for 1162 reflections ($F_o \geq 6\sigma|F_o|$). The molecule consists of two anthracene units linked together through two bonds. The bond angles at C9 and C10 appear to be normal. The two symmetrically equivalent bonds, C9—C10′ and C10—C9′, are unusually long, 1·618 (3) Å, com-

* Several other names have been used for the title compound. One of the most common is the obvious one, 'Dianthracene'. The similarity of the model to the space ships of Darth Vader in the movie *The Empire Strikes Back* led one of the authors (RMR) to refer to it as 'Darthvaderene'.

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