

Fig. 2. Stereoscopic view of the unit-cell contents; H atoms omitted for clarity.

Related literature. Crystal structures of dibenzo-24-crown-8 (Hanson, Hughes & Truter, 1976) and dibenzo-30-crown-10 (Bush & Truter, 1972) and the complexation of guanidinium perchlorate with benzo

crown ethers (de Boer, Uiterwijk, Geevers, Harkema & Reinhoudt, 1983) have been reported.

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Structure of Bruceine B Hydrate

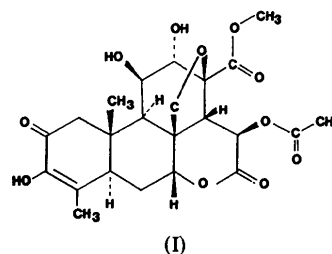
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Abstract. $C_{23}H_{28}O_{11} \cdot H_2O$, $M_r = 498.48$, monoclinic, $P2_1$, $a = 8.696$ (3), $b = 11.493$ (5), $c = 11.601$ (3) Å, $\beta = 100.7$ (1)°, $V = 1139.3$ (3) Å³, $Z = 2$, $D_x = 1.45$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.13$ mm⁻¹, $F(000) = 528$, $T = 295$ K, final $R = 0.037$, $wR = 0.033$, for 1423 independent reflections. Overall the molecule has an extended conformation. The only evidence of strain is in the five-membered ring where the average intra-ring bond angle for junction atoms is only 99.7°. Packing is dominated by hydrogen bonding involving all possible H atoms. The ether O atom in the five-membered ring has two intermolecular O...O approaches slightly less than van der Waals separations.

Experimental. The title compound (I), purified and crystallized from medicinal plant, *Brucea javanica*, has



been found to be an active anti-malarial agent (Pavanand *et al.*, 1986). Crystal dimensions 0.15 × 0.10 × 0.25 mm, automated Nicolet R3m diffractometer with incident-beam monochromator. 25 centered reflections within $15 \leq 2\theta \leq 23^\circ$ used for determining lattice parameters. $(\sin\theta/\lambda)_{\max} = 0.538$ Å⁻¹, range of hkl : $-9 \leq h \leq 4$, $-13 \leq k \leq 0$, $-12 \leq l \leq 12$. Standards

200, 020, 006, monitored every 60 reflections with random variation of 2.5% over data collection, $\theta/2\theta$ mode, scan width $(1.8 + \Delta_{\alpha 1\alpha 2})^\circ$, scan rate a function of count rate (4° min^{-1} minimum, $30^\circ \text{ min}^{-1}$ maximum), 2702 reflections measured, 1589 unique, $R_{\text{int}} = 0.034$, 1423 observed with $F_o > 3\sigma(F_o)$. Data corrected for Lorentz and polarization, but not absorption effects; isotropic secondary-extinction value $p = 0.0014(5)$ in $F_c^* = F_c/[1.0 + 0.002pF_o^2/\sin 2\theta]^{0.25}$. Structure solved by direct methods. The least-squares refinement used program *SHELXTL* (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0.00012$. 381 parameters refined: non-H atoms anisotropic, H-atom coordinates only with isotropic thermal parameters riding on covalently bonded atoms. $(\Delta/\sigma)_{\text{max}} = 0.059$, $R = 0.037$, $wR = 0.033$, $S = 1.32$. Final difference Fourier excursions 0.169 and $-0.154 \text{ e } \text{\AA}^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).† Atom numbering for Tables 1 and 2, atom coordinates, bond distances and angles, follows that shown in Fig. 1.

† Lists of structure factors, anisotropic thermal parameters, hydrogen coordinates and hydrogen-bond parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43584 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates and equivalent isotropic temperature coefficients* ($\text{\AA}^2 \times 10^2$)

Equivalent isotropic U defined as one-third of the trace of the orthogonalized U_{ij} tensor.

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
O1	0.6974 (3)	0.2500	0.3171 (2)	4.0 (1)
O2	0.2849 (3)	0.4335 (2)	0.4535 (2)	3.8 (1)
O3	0.2698 (3)	0.0720 (3)	-0.1753 (3)	6.1 (1)
O4	0.0554 (4)	0.0135 (3)	-0.0451 (3)	7.9 (1)
O5	0.0641 (3)	0.2646 (2)	0.3206 (2)	4.7 (1)
O6	0.3247 (3)	0.1376 (2)	0.5707 (2)	4.4 (1)
O7	0.8547 (3)	0.1525 (2)	0.4524 (3)	5.2 (1)
O8	0.2560 (3)	0.3793 (3)	0.6937 (2)	5.5 (1)
O9	0.5024 (3)	0.4318 (3)	0.6952 (3)	7.0 (1)
O10	0.6908 (3)	0.2112 (2)	0.6265 (2)	3.8 (1)
O11	0.8434 (3)	0.3635 (2)	0.5995 (3)	6.2 (1)
C1a	0.5721 (4)	0.3353 (3)	0.2755 (3)	3.4 (1)
C1b	0.4378 (4)	0.3254 (3)	0.3419 (3)	3.1 (1)
C2	0.5331 (4)	0.3146 (3)	0.1453 (3)	3.8 (1)
C2a	0.4413 (4)	0.2004 (3)	0.1173 (3)	3.6 (1)
C3	0.4188 (4)	0.1629 (3)	-0.0092 (3)	4.0 (1)
C4	0.2932 (5)	0.1029 (3)	-0.0592 (4)	4.5 (1)
C5	0.1716 (5)	0.0641 (3)	0.0058 (4)	4.8 (2)
C6	0.1994 (5)	0.0899 (3)	0.1352 (4)	3.9 (1)
C6a	0.2852 (4)	0.2068 (3)	0.1655 (3)	3.4 (1)
C6b	0.3334 (4)	0.2175 (3)	0.3015 (3)	3.1 (1)
C7	0.2022 (4)	0.2006 (3)	0.3732 (3)	3.6 (1)
C8	0.2472 (4)	0.2321 (3)	0.5037 (3)	3.5 (1)
C8a	0.3584 (4)	0.3376 (3)	0.5238 (3)	3.2 (1)
C8b	0.5079 (4)	0.3208 (3)	0.4742 (3)	3.1 (1)
C9	0.6127 (4)	0.2157 (3)	0.5049 (3)	3.1 (1)
C10	0.7339 (4)	0.2063 (3)	0.4261 (3)	3.8 (1)
C11	0.3421 (4)	0.4373 (3)	0.3421 (3)	3.6 (1)
C12	0.5468 (5)	0.1883 (4)	-0.0784 (4)	6.0 (2)
C13	0.3845 (4)	0.3860 (3)	0.6486 (3)	4.0 (1)
C14	0.2581 (6)	0.4389 (4)	0.8033 (4)	7.0 (2)
C15	0.8093 (4)	0.2892 (3)	0.6617 (4)	4.6 (1)
C16	0.8851 (5)	0.2647 (5)	0.7845 (4)	6.6 (2)
C17	0.1787 (4)	0.3026 (3)	0.1035 (3)	4.2 (1)
W	0.0453 (4)	0.0084 (3)	-0.3635 (4)	7.7 (1)

Related literature. For compounds with similar structures and biological activity see papers on isobruceine B (Moretti, Polonsky, Vuilhorgne & Prange, 1982), bruceine C (Polonsky, Varenne, Prange & Pascard, 1980).

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Table 2. *Bond lengths* (\AA) *and angles* ($^\circ$)

O1-C1a	1.479 (4)	C6a-C6b	1.560 (5)
O1-C10	1.342 (4)	C6a-C17	1.546 (5)
O2-C8a	1.448 (4)	C6b-C7	1.545 (5)
O2-C11	1.469 (5)	C7-C8	1.535 (5)
C1a-C1b	1.519 (5)	C7-O5	1.442 (4)
C1a-C2	1.504 (5)	C8-C8a	1.541 (5)
C1b-C6b	1.556 (4)	C8-O6	1.429 (4)
C1b-C8b	1.543 (5)	C8a-C8b	1.528 (5)
C1b-C11	1.533 (5)	C8a-C13	1.528 (5)
C2-C2a	1.539 (5)	C8b-C9	1.516 (5)
C2a-C3	1.507 (5)	C9-C10	1.522 (5)
C2a-C6a	1.563 (5)	C9-O10	1.449 (4)
C3-C4	1.330 (5)	C10-O7	1.208 (4)
C3-C12	1.516 (6)	C13-O8	1.321 (5)
C4-C5	1.477 (6)	C13-O9	1.190 (4)
C4-O3	1.372 (5)	C14-O8	1.442 (6)
C5-C6	1.505 (6)	C15-C16	1.482 (6)
C5-O4	1.219 (5)	C15-O10	1.370 (4)
C6-C6a	1.546 (5)	C15-O11	1.190 (5)
C1a-O1-C10	125.9	C6a-C6b-C7	116.6
C8a-O2c-C11	109.4	C1b-C6b-C7	112.7
O1-C1a-C1b	112.0	C6b-C7-O5	110.4
O1-C1a-C2	103.8	C6b-C7-C8	115.0
C1b-C1a-C2	116.5	C8-C7-O5	109.7
C1a-C1b-C6b	111.5	C7-C8-C8a	111.9
C1a-C1b-C8b	108.0	C7-C8-O6	111.4
C1a-C1b-C11	114.0	C8a-C8-O6	107.2
C6b-C1b-C8b	111.9	C8-C8a-C8b	113.5
C6b-C1b-C11	112.5	C8-C8a-C13	114.1
C8b-C1b-C11	98.1	C8-C8a-O2	108.3
C1a-C2-C2a	111.0	C8b-C8a-C13	114.6
C2-C2a-C3	114.7	C8b-C8a-O2	101.6
C2-C2a-C6a	109.6	C13-C8a-O2	103.2
C3-C2a-C6a	113.4	C8a-C8b-C9	121.9
C2a-C3-C4	121.3	C8a-C8b-C1b	99.4
C2a-C3-C12	119.0	C1b-C8b-C9	111.7
C4-C3-C12	119.6	C8b-C9-O10	114.2
C3-C4-C5	122.9	C8b-C9-C10	111.6
C3-C4-O3	121.5	C10-C9-O10	109.4
C5-C4-O3	115.7	C9-C10-O7	123.4
C4-C5-C6	116.8	C9-C10-O1	118.1
C4-C5-O4	120.3	O1-C10-O7	118.1
C6-C5-O4	122.8	C1b-C11-O2	104.4
C5-C6-C6a	112.3	C8a-C13-O8	111.5
C6-C6a-C2a	107.0	C8a-C13-O9	123.8
C6-C6a-C6b	109.0	O8-C13-O9	124.5
C6-C6a-C17	107.1	O10-C15-C16	109.7
C2a-C6a-C6b	106.1	O11-C15-C16	127.1
C2a-C6a-C17	110.9	C13-O8-C14	116.7
C6b-C6a-C17	116.4	C9 O10 C15	117.2
C6a-C6b-C1b	113.6		

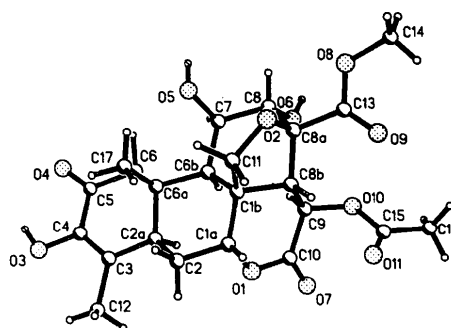


Fig. 1. Bruceine B illustrating the results of the X-ray study and the numbering scheme.

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Reaction Between 2-Lithio-1,3-dithiane and Tetrahydrofuran: Structure of Bis[2-(1,3-dithianyl)]methanol

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Abstract. $C_9H_{16}OS_4$, $M_r = 268.47$, monoclinic, $P2_1/c$, $a = 6.673$ (4), $b = 9.806$ (3), $c = 19.084$ (5) Å, $\beta = 97.12$ (3)°, $V = 1239.14$ Å³, $Z = 4$, $D_m = 1.44$, $D_x = 1.44$ g cm⁻³, $\lambda(Cu K\alpha) = 1.5418$ Å, $\mu = 65.58$ cm⁻¹, $F(000) = 568$, $T = 293$ K, $R = 0.041$ for 1637 unique observed reflections. The two dithiane rings are in chair conformations having similar geometries: the angle between mean (least-squares) planes through the rings is 57.9°. The S–C distances range from 1.798 (5) to 1.817 (3) Å and the C–C distances from 1.516 (6) to 1.542 (4) Å; the O–C distance is 1.420 (4) Å.

Experimental. Compound isolated unexpectedly (in 25% yield) from reaction between 2-lithio-1,3-dithiane (generated from 1,3-dithiane and *n*-butyllithium) and tetrahydrofuran which occurs on warming of the mixture from 195 to 298 K during 12 h. Crystals obtained by slow diffusion of hexane into a solution in ethyl ethanoate. Cuboid cut to ca 0.30 × 0.35 × 0.30 mm and mounted on glass fibre. Density measured by flotation in aqueous sodium bromide solution. Intensities measured by SERC service with an Enraf–Nonius CAD-4 diffractometer and ω – 2θ scans. Unit cell determined from least-squares analysis of angle data for 25 reflections with $16 < \theta < 28^\circ$. Data collected to $\sin\theta/\lambda$ of 0.59 Å⁻¹, $0 < h < 7$, $0 < k < 11$, $-22 < l < 22$; empirical absorption correction applied, transmission factors 1.000–0.663. Three standard reflections (018̄, 045, 227̄) varied $\pm 2\%$; linear drift correction applied, 2299 reflections measured, 2103 unique ($R_{int} = 0.0186$), 466 reflections with $I < 2\sigma(I)$ considered unobserved. Solved by direct methods with *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain,

Table 1. Fractional atomic coordinates, mean isotropic thermal parameters, and their e.s.d.'s

$$\bar{U} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	<i>x</i>	<i>y</i>	<i>z</i>	\bar{U} (Å ²)
S(1)	0.1998 (2)	0.3182 (1)	0.59865 (5)	0.0531 (6)
S(3)	0.4288 (2)	0.0630 (1)	0.57485 (4)	0.0499 (6)
S(7)	0.0105 (2)	0.0727 (1)	0.76365 (5)	0.0481 (6)
S(9)	0.3424 (1)	0.2400 (1)	0.84315 (4)	0.0390 (5)
O(14)	0.4172 (4)	0.0122 (3)	0.7306 (1)	0.0474 (15)
C(2)	0.2688 (6)	0.1497 (3)	0.6309 (2)	0.0358 (17)
C(4)	0.6377 (7)	0.1793 (5)	0.5768 (2)	0.0610 (26)
C(5)	0.5793 (7)	0.3221 (5)	0.5495 (2)	0.0629 (27)
C(6)	0.4451 (8)	0.3959 (4)	0.5957 (2)	0.0638 (27)
C(8)	0.2079 (5)	0.1968 (3)	0.7573 (1)	0.0296 (16)
C(10)	0.1367 (6)	0.3103 (4)	0.8850 (2)	0.0413 (19)
C(11)	−0.0397 (6)	0.2116 (4)	0.8878 (2)	0.0458 (20)
C(12)	−0.1451 (6)	0.1734 (4)	0.8148 (2)	0.0483 (21)
C(13)	0.3589 (5)	0.1466 (3)	0.7093 (1)	0.0335 (17)

Table 2. Bond distances (Å), bond angles (°), and their e.s.d.'s (not including H atoms)

S(1)–C(2)	1.804 (3)	S(1)–C(6)	1.813 (5)
S(3)–C(2)	1.813 (3)	S(3)–C(4)	1.798 (5)
S(7)–C(8)	1.808 (3)	S(7)–C(12)	1.805 (4)
S(9)–C(8)	1.817 (3)	S(9)–C(10)	1.808 (4)
O(14)–C(13)	1.420 (4)	C(2)–C(13)	1.542 (4)
C(4)–C(5)	1.527 (6)	C(5)–C(6)	1.516 (6)
C(8)–C(13)	1.526 (4)	C(10)–C(11)	1.530 (5)
C(11)–C(12)	1.528 (5)		
C(2)–S(1)–C(6)	101.7 (2)	C(2)–S(3)–C(4)	101.7 (2)
C(8)–S(7)–C(12)	98.1 (2)	C(8)–S(9)–C(10)	99.8 (2)
S(1)–C(2)–S(3)	111.9 (2)	S(1)–C(2)–C(13)	113.6 (2)
S(3)–C(2)–C(13)	112.1 (2)	S(3)–C(4)–C(5)	114.2 (3)
C(4)–C(5)–C(6)	112.6 (3)	S(1)–C(6)–C(5)	114.7 (3)
S(7)–C(8)–S(9)	112.3 (2)	S(7)–C(8)–C(13)	111.3 (2)
S(9)–C(8)–C(13)	109.1 (2)	S(9)–C(10)–C(11)	113.8 (2)
C(10)–C(11)–C(12)	113.1 (3)	S(7)–C(12)–C(11)	113.4 (3)
O(14)–C(13)–C(2)	110.8 (3)	O(14)–C(13)–C(8)	107.8 (2)
C(2)–C(13)–C(8)	111.7 (3)		