

(vi), Fig. 3],* le changement de chiralité de l'une des deux molécules ne modifie pas sensiblement les orientations relatives des dipôles.

* Déposée.

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Acta Cryst. (1987). **C43**, 1420–1422

Structure of Trimethylammonium 2-Mercaptononahydrodecaborate

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(Received 23 January 1987; accepted 9 March 1987)

Abstract. [(CH₃)₃NH]⁺₂.B₁₀H₁₀S²⁻, *M_r* = 270.48, orthorhombic, *Pbca*, *a* = 11.719 (1), *b* = 21.167 (2), *c* = 14.485 (1) Å, *V* = 3593.2 (5) Å³, *Z* = 8, *D_x* = 1.000 Mg m⁻³, λ(Cu Kα) = 1.5418 Å, μ(Cu Kα) = 1.36 mm⁻¹, *F*(000) = 1168, *T* = 295 K, *R* = 0.098 for 2071 reflections. The anion, a bicapped square antiprismatic cage, is substituted by an SH group at the equatorial position.

Introduction. The sodium salt of ¹⁰B-enriched (B₁₂H₁₁SH)²⁻ is now being clinically tested for use in boron-neutron capture therapy of malignant brain tumors. With the expectation of achieving similar clinical effects, two positional isomers of (B₁₀H₉SH)²⁻ were regio-selectively prepared (Komura, Nakai & Shiro, 1987), and one of them was confirmed in this work to be the 2-substituted anion.

Experimental. Prismatic colourless crystals grown from aqueous solution. Crystal of dimensions 0.3 × 0.2 × 0.2 mm used. Hilger-Watts Y-290 diffractometer, Ni-filtered Cu Kα. Cell dimensions from 2θ angles for 12 reflections (30 < 2θ < 40°). Intensities measured up to 2θ = 114° in *h* 0/12, *k* 0/22 and *l* 0/15 by ω-2θ step-scanning mode, 80 steps with 0.01° interval of ω and for 1 s at each step. One standard reflection monitored every ten measurements (±1% variation). 2419 unique reflections measured, 2359 observed [|*F_o*| > σ(*F_o*)], no absorption corrections. Structure solved by *MULTAN74* (Main, Germain & Woolfson,

1974). H atoms of the anion located on a difference electron density map but not those of the cation. The latter were placed at their calculated positions and included in calculation of structure factors. Positional parameters of the atoms excluding H of the cation and thermal parameters of non-H atoms refined by block-diagonal least squares. Temperature factor of each H atom equal to *B_{eq}* of the bonded atom. *R* = 0.098, *wR* = 0.147, *S* = 1.2496 for 2071 observed reflections (*w* ≠ 0). Large *R* value due to poor quality of crystal and large motion of C atoms. No significant peaks in final difference map, highest peak 0.5 e Å⁻³. Σ(*w*|Δ*F*|²) minimized, *w* = [σ²(*F_o*) + 0.0106|*F_o*|²]⁻¹ for *w*^{1/2}|*F_c*| ≥ 1 and *w*^{1/2}|Δ*F*| < 3, *w* = 0 otherwise. Ratio of final shift to e.s.d. < 0.1. Atomic scattering factors calculated by *f* = Σ[*a_i*exp(-*b_i*sin²θ/λ²)] + *c* (*i* = 1, ..., 4) (*International Tables for X-ray Crystallography*, 1974). Calculations performed by FACOM 270-30 computer at our laboratories.

Discussion. Atomic coordinates and equivalent isotropic temperature factors are listed in Table 1.* Bond lengths and angles are listed in Table 2. A perspective view of the anionic boron cage with the atom-labelling

* Lists of anisotropic temperature factors of the non-H atoms, atomic coordinates of the H atoms and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43860 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

system and a stereoscopic view of the crystal structure drawn using the program *PLUTO* (Motherwell & Clegg, 1978) are shown in Figs. 1 and 2, respectively. The anion has the bicapped square antiprismatic structure in which B(1) and B(10) occupy the apical positions and B(2)–B(9) occupy the equatorial positions, respectively. The SH group is bonded to the atom at the equatorial position.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10$) with *e.s.d.*'s in parentheses

$$B_{\text{eq}} = \frac{1}{3} \sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq}
B(1)	988 (4)	1670 (2)	5252 (3)	45 (1)
B(2)	2373 (4)	1807 (2)	5477 (3)	33 (1)
B(3)	1586 (4)	1168 (2)	6037 (3)	36 (1)
B(4)	1097 (4)	903 (2)	4903 (3)	43 (1)
B(5)	1883 (4)	1540 (2)	4343 (3)	35 (1)
B(6)	3137 (4)	1127 (2)	5893 (3)	38 (1)
B(7)	2225 (4)	483 (2)	5502 (3)	40 (1)
B(8)	2414 (4)	750 (2)	4302 (3)	38 (1)
B(9)	3318 (4)	1396 (2)	4711 (3)	36 (1)
B(10)	3497 (4)	640 (2)	5035 (3)	41 (1)
S	2784 (1)	2618.1 (4)	5913 (1)	44.1 (3)
N(1)	2509 (3)	3390 (2)	3993 (2)	50 (1)
C(11)	1278 (6)	3337 (3)	3881 (5)	83 (2)
C(12)	2977 (8)	4043 (3)	3989 (7)	108 (3)
C(13)	3132 (5)	3044 (2)	3298 (3)	58 (1)
N(2)	4807 (3)	759 (2)	2980 (3)	50 (1)
C(21)	4827 (5)	110 (2)	2607 (4)	73 (2)
C(22)	4288 (4)	1186 (2)	2284 (3)	56 (1)
C(23)	5932 (4)	963 (3)	3244 (4)	69 (2)

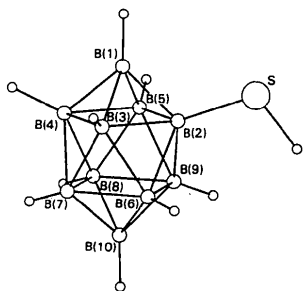


Fig. 1. Perspective view of the 2-mercaptanonahydrodecaborate anion with atom-labelling system.

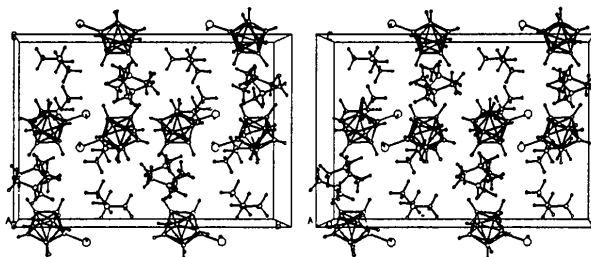


Fig. 2. Stereoscopic projection of the crystal packing viewed down the *a* axis.

The boron cage adopts approximately D_{4d} symmetry like that of cuprous decahydrodecaborate (Dobrott & Lipscomb, 1962). The mean B–B bond distances of the apex–equatorial, equatorial–equatorial, and equatorial–equatorial' edges are respectively 1.686 (3), 1.827 (3) and 1.803 (3) Å, compared with 1.73, 1.86 and 1.82 Å in the copper salt. The B–S bond length is similar to that [1.90 (2) Å] in caesium monomercaptoundeca-

Table 2. Bond lengths (Å) and angles ($^\circ$) with *e.s.d.*'s in parentheses

B(1)–B(2)	1.681 (7)	B(5)–B(9)	1.790 (7)
B(1)–B(3)	1.707 (7)	B(6)–B(7)	1.822 (7)
B(1)–B(4)	1.705 (7)	B(6)–B(9)	1.817 (7)
B(1)–B(5)	1.706 (7)	B(6)–B(10)	1.669 (7)
B(2)–B(3)	1.827 (7)	B(7)–B(8)	1.841 (7)
B(2)–B(5)	1.830 (7)	B(7)–B(10)	1.670 (7)
B(2)–B(6)	1.799 (7)	B(8)–B(9)	1.828 (7)
B(2)–B(9)	1.793 (7)	B(8)–B(10)	1.671 (7)
B(2)–S	1.892 (5)	B(9)–B(10)	1.681 (7)
B(3)–B(4)	1.828 (7)	N(1)–C(11)	1.456 (8)
B(3)–B(6)	1.832 (7)	N(1)–C(12)	1.487 (11)
B(3)–B(7)	1.807 (7)	N(1)–C(13)	1.443 (7)
B(4)–B(5)	1.823 (7)	N(2)–C(21)	1.476 (7)
B(4)–B(7)	1.814 (7)	N(2)–C(22)	1.484 (6)
B(4)–B(8)	1.801 (7)	N(2)–C(23)	1.439 (8)
B(5)–B(8)	1.785 (7)		
B(2)–B(1)–B(3)	65.3 (3)	B(2)–B(6)–B(7)	101.7 (3)
B(2)–B(1)–B(4)	98.6 (3)	B(2)–B(6)–B(9)	59.4 (3)
B(2)–B(1)–B(5)	65.4 (3)	B(2)–B(6)–B(10)	111.8 (3)
B(3)–B(1)–B(4)	64.8 (3)	B(3)–B(6)–B(7)	59.3 (3)
B(3)–B(1)–B(5)	99.3 (3)	B(3)–B(6)–B(9)	102.0 (3)
B(4)–B(1)–B(5)	64.6 (3)	B(3)–B(6)–B(10)	111.4 (3)
B(1)–B(2)–B(3)	58.1 (3)	B(7)–B(6)–B(9)	90.6 (3)
B(1)–B(2)–B(5)	58.0 (3)	B(7)–B(6)–B(10)	57.0 (3)
B(1)–B(2)–B(6)	114.0 (3)	B(9)–B(6)–B(10)	57.5 (3)
B(1)–B(2)–B(9)	113.1 (3)	B(3)–B(7)–B(4)	60.6 (3)
B(1)–B(2)–S	117.9 (3)	B(3)–B(7)–B(6)	60.6 (3)
B(3)–B(2)–B(5)	90.7 (3)	B(3)–B(7)–B(8)	102.0 (3)
B(3)–B(2)–B(6)	60.7 (3)	B(3)–B(7)–B(10)	112.6 (3)
B(3)–B(2)–B(9)	103.1 (3)	B(4)–B(7)–B(6)	102.1 (3)
B(3)–B(2)–S	130.7 (3)	B(4)–B(7)–B(8)	59.0 (3)
B(5)–B(2)–B(6)	102.1 (3)	B(4)–B(7)–B(10)	111.0 (3)
B(5)–B(2)–B(9)	59.2 (3)	B(6)–B(7)–B(8)	89.6 (3)
B(5)–B(2)–S	131.3 (3)	B(6)–B(7)–B(10)	56.9 (3)
B(6)–B(2)–B(9)	60.8 (3)	B(8)–B(7)–B(10)	56.6 (3)
B(6)–B(2)–S	119.2 (3)	B(4)–B(8)–B(5)	61.1 (3)
B(9)–B(2)–S	119.3 (3)	B(4)–B(8)–B(7)	59.7 (3)
B(1)–B(3)–B(2)	56.7 (3)	B(4)–B(8)–B(9)	101.9 (3)
B(1)–B(3)–B(4)	57.6 (3)	B(4)–B(8)–B(10)	111.6 (3)
B(1)–B(3)–B(6)	111.2 (3)	B(5)–B(8)–B(7)	102.4 (3)
B(1)–B(3)–B(7)	112.6 (3)	B(5)–B(8)–B(9)	59.4 (3)
B(2)–B(3)–B(4)	89.2 (3)	B(5)–B(8)–B(10)	112.0 (3)
B(2)–B(3)–B(6)	58.9 (3)	B(7)–B(8)–B(9)	89.6 (3)
B(2)–B(3)–B(7)	101.2 (3)	B(7)–B(8)–B(10)	56.5 (3)
B(4)–B(3)–B(6)	101.2 (3)	B(9)–B(8)–B(10)	57.2 (3)
B(4)–B(3)–B(7)	59.9 (3)	B(2)–B(9)–B(5)	61.4 (3)
B(6)–B(3)–B(7)	60.1 (3)	B(2)–B(9)–B(6)	59.8 (3)
B(1)–B(4)–B(3)	57.6 (3)	B(2)–B(9)–B(8)	101.9 (3)
B(1)–B(4)–B(5)	57.7 (3)	B(2)–B(9)–B(10)	111.5 (3)
B(1)–B(4)–B(7)	112.3 (3)	B(5)–B(9)–B(6)	103.0 (3)
B(1)–B(4)–B(8)	112.2 (3)	B(5)–B(9)–B(8)	59.1 (3)
B(3)–B(4)–B(5)	90.8 (3)	B(5)–B(9)–B(10)	111.3 (3)
B(3)–B(4)–B(7)	59.5 (3)	B(6)–B(9)–B(8)	90.2 (3)
B(3)–B(4)–B(8)	102.8 (3)	B(6)–B(9)–B(10)	56.8 (3)
B(5)–B(4)–B(7)	102.0 (3)	B(8)–B(9)–B(10)	56.7 (3)
B(5)–B(4)–B(8)	59.0 (3)	B(6)–B(10)–B(7)	66.2 (3)
B(7)–B(4)–B(8)	61.2 (3)	B(6)–B(10)–B(8)	101.3 (3)
B(1)–B(5)–B(2)	56.6 (3)	B(6)–B(10)–B(9)	65.7 (3)
B(1)–B(5)–B(4)	57.7 (3)	B(7)–B(10)–B(8)	66.9 (3)
B(1)–B(5)–B(8)	113.0 (3)	B(7)–B(10)–B(9)	101.0 (3)
B(1)–B(5)–B(9)	112.0 (3)	B(8)–B(10)–B(9)	66.1 (3)
B(2)–B(5)–B(4)	89.3 (3)	C(11)–N(1)–C(12)	115.9 (6)
B(2)–B(5)–B(8)	102.1 (3)	C(11)–N(1)–C(13)	112.6 (5)
B(2)–B(5)–B(9)	59.4 (3)	C(12)–N(1)–C(13)	106.4 (5)
B(4)–B(5)–B(8)	59.9 (3)	C(21)–N(2)–C(22)	108.9 (4)
B(4)–B(5)–B(9)	102.5 (3)	C(21)–N(2)–C(23)	111.2 (4)
B(8)–B(5)–B(9)	61.5 (3)	C(22)–N(2)–C(23)	111.9 (4)
B(2)–B(6)–B(3)	60.4 (3)		

hydrododecaborate monohydrate (Shiro, Aono & Watanabe, 1970). The B—H distances range from 0.89 (7) to 1.26 (7) Å. The C—N bonds in the trimethylammonium ions average 1.464 (4) Å, but range in the broad region of 1.439–1.487 Å owing to the inaccurate positions of the C atoms subject to the large thermal motion.

The N—H bond of a cation points to the S atom and forms the hydrogen bond N—H...S, the distance between N and S being 3.242 (4) Å. There are no other contacts shorter than the sum of van der Waals radii.

We thank Dr T. Nakagawa for his continuing interest throughout this investigation.

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Acta Cryst. (1987). **C43**, 1422–1424

Conformation of a Cycloundeca-1,5-diene Ring: Structure of (6*R*,12*R*)-6-Acetoxy-12,16-dihydroxydolabella-3,7-diene

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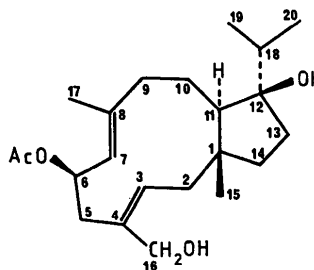
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(Received 23 January 1987; accepted 18 March 1987)

Abstract. $C_{22}H_{36}O_4$, $M_r = 364.55$, monoclinic, $P2_1$, $a = 9.304$ (3), $b = 9.606$ (9), $c = 12.371$ (13) Å, $\beta = 100.69$ (4)°, $V = 1086$ (1) Å³, $Z = 2$, $D_x = 1.11$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 0.08$ mm⁻¹, $F(000) = 400$, $T = 293$ K, $R = 0.031$ for 1667 reflections [$I/\sigma(I) \geq 2.5$]. The 11-membered ring exhibits a conformation that is one of the low-energy forms found for cycloundeca-1,5-diene by molecular-mechanics calculations. The C—C(*sp*³)—C angles in the 11-membered ring are 110.3–119.5°, mean 114.7°. The fusion distance, 1.578 (4) Å, is long due to torsional strain.

Introduction. The conformational properties of odd-numbered organic rings of medium size ($n = 9, 11, 13$) are not as well characterized as those of even-numbered rings and only limited information is available about the conformations of 11-membered rings (Anet & Rawdah, 1978). Such rings occur in various natural products, e.g. cytochalasins, dolabellane diterpenoids, and pyrrolizidine alkaloids, and studies of these compounds provide conformational details of substituted 11-membered rings. An X-ray study of the title

compound was undertaken to determine the conformation of its cycloundeca-1,5-diene ring.



Experimental. Colourless crystal, dimensions 0.15 × 0.20 × 0.40 mm. Enraf–Nonius CAD-4 diffractometer, Mo *K*α radiation. Cell dimensions from setting angles of 25 independent reflections with θ ca 12°; 2136 intensities surveyed in range 1.5–25.0°; h $\bar{1}$ –11, k 0–11, l 0–14; 2034 independent reflections after deletion of systematic absences and averaging of equivalent reflections, 1667 reflections with $I > 2.5\sigma(I)$, $R_{\text{int}} = 0.022$. Two reference reflections monitored periodically showed no significant variation in intensity.

0108-2701/87/071422-03\$01.50

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