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## 9-(2,2-Dimethylpropionyloxy)-9-(9-borabicyclo[3.3.1]nonanyl)-9-borabicyclo[3.3.1]nonane

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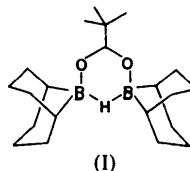
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**Abstract.** C<sub>21</sub>H<sub>38</sub>B<sub>2</sub>O<sub>7</sub>, *M<sub>r</sub>* = 344.16, monoclinic, space group *P*2<sub>1</sub>/*n*, *a* = 18.167 (1), *b* = 10.089 (1), *c* = 23.198 (1) Å, β = 97.64 (1)°, *V* = 4214.32 Å<sup>3</sup>, *Z* = 8, *D<sub>x</sub>* = 1.085 g cm<sup>-3</sup>, λ(Cu Kα) = 1.54178 Å (Ni filter), μ(Cu Kα) = 4.66 cm<sup>-1</sup>, *F*(000) = 1520, *T* = 294 K, *R* = 0.075 for 4050 unique observed reflections and 459 parameters. The title compound forms a cyclic carboxylic acid–diorganoborane adduct in which two 9-borabicyclo[3.3.1]nonanyl groups are connected by a bridging carboxylate and an additional three-centre–two-electron BHB bond. The six-membered rings OCOBHB are slightly puckered with a maximum deviation from their best plane of 0.14 Å for one ring atom. The average B–C bond length is 1.582 (9) Å and the average B–O bond length 1.52 (1) Å. The non-bonding B···B intramolecular distance is 2.496 (7) Å; the B–H bond is 1.33 (3) Å (average values). The B–H–B angle is 141 (3)° (average).

**Introduction.** In recent investigations of the dialkylhydroborane reduction of carboxylic acids, especially glucuronic acid (Köster, Idelmann & Dahlhoff, 1982), reaction intermediates with characteristic IR absorption of B–(μ-H)–B groups could be observed. Subsequent studies showed that these intermediates also occur in the reduction of simple monocarboxylic acids and established them as novel cyclic adducts of diorgano(acyloxy)boranes with diorgano(hydro)boranes. Upon substitution of the reactants with suitably bulky substituents some of these intermediates could be sufficiently stabilized to be isolable in the solid

state. Thus the reaction of pivalic acid with bis-9-borabicyclo[3.3.1]nonane (9-BBN) (Köster, 1960; Köster & Binger, 1974) in non-etheral solvents afforded 9-(2,2-dimethylpropionyloxy)-9-(9-borabicyclo[3.3.1]nonanyl)-9-borabicyclo[3.3.1]nonane (I) in quantitative yield (Idelmann, Müller, Scheidt, Schübler, Seevogel & Köster, 1984).



The novel character of this boron heterocycle as well as its alleged general role as intermediate in the reduction of carboxylic acids by boranes (Brown, 1972, 1980) prompted us to determine its crystal and molecular structure. Further impetus came from the observation that upon heating to 373 K the adduct disproportionates with elimination of bis(octane-1,5-diyl)diboroxane to give the unreduced acid and the corresponding alcohol, both as their 9-borabicyclo[3.3.1]nonanyl compounds. The disproportionation also occurs in the solid state, thereby suggesting an intermolecular reaction mechanism (Idelmann *et al.*, 1984). Thus, it was hoped that an investigation of the packing mode of these molecules in the crystal would give further insight into the mechanism leading to the observed products.

**Experimental.** Platelike crystals (0.8 × 0.2 × 0.4 mm), grown from heptane; Enraf–Nonius CAD-4 diffractometer; cell dimensions and their e.s.d.'s by least-

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squares refinement from setting angles of 75 reflections ( $12.3 \leq \theta \leq 46.9^\circ$ ); 9503 measured reflections ( $\theta$ - $2\theta$  scans,  $1.9 \leq \theta \leq 75.6^\circ$ ,  $hkl$  range:  $\pm 22, + 12, + 29$ ) merged to give 8759 unique reflections ( $R_{\text{int}} = 0.025$ ) of which 4709 were unobserved [ $I < 2.0\sigma(I)$ ]; orientation (every 250 reflections) and intensity control (every 4000 s of X-ray exposure) by means of three standard reflections (040, 600, 0,0,10); corrections for decay (ca 22%), for Lp and for absorption effects (Walker & Stuart, 1983); solution by direct methods using *SHELX76* (Sheldrick, 1976); after several rescaling attempts of the  $E^2$ 's a fragmentary solution could be obtained only by omission of the 20 strongest reflections which had most phase relations; completion of the structure first by tangent refinement of the partial structure then by Fourier methods, most H atoms from difference Fourier syntheses except nine H atoms at C(3), C(17), C(34), C(36) which were calculated at idealized positions [ $d(\text{C}-\text{H}) = 1.0 \text{ \AA}$ ]; anisotropic refinement of all non-H atoms, H atoms at B isotropic, all others included as fixed atom contributions, parameters of each independent molecule in one block;  $R = 0.075$ ,  $wR = 0.087$  for 4050 observed reflections and 459 parameters,  $R = 0.095$  for all reflections,  $S = 3.5$ ; the function minimized was  $\sum w(|F_o| - |F_c|)^2$ ,  $w = 1/\sigma^2(F_o)$ ; unobserved reflections not included in refinement, omission of 004 in final cycles due to large  $F_o/F_c$  difference; average shift/e.s.d. in last cycle 0.051; final difference Fourier synthesis qualitatively featureless, max. residual electron density:  $0.17 \text{ e \AA}^{-3}$ . The rather poor agreement between observed and calculated structure factors is due to poor crystal quality. Further details of the computing procedures and programs used as described elsewhere (Krüger, Müller, Erker, Dorf & Engel, 1985), scattering factors for neutral, spherical atoms (except for H) from Cromer & Waber (1965), for the H atoms, based on a bonded spherical-atom model (Stewart, Davidson & Simpson, 1965).\*

**Discussion.** Table 1 contains the final positional parameters of the non-H atoms and the bridging-H atoms. Selected interatomic distances and angles are summarized in Table 2. Fig. 1 gives perspective views of the two crystallographically independent molecules. Fig. 2 shows a projection of the unit-cell contents onto the  $ac$  plane.

Both crystallographically independent molecules consist of two 9-borabicyclo[3.3.1]nonanyl units which

are symmetrically connected by a pivaloate bridge and a three-center-two-electron ( $3c-2e$ ) BHB bond. The so-formed six-membered OCOBHB rings deviate slightly from planarity. The largest deviation of a ring atom from the best ring plane is only  $0.14 \text{ \AA}$  [for B(4)]. The resulting slight puckering of the rings (twist conformation, Fig. 2) is the most significant difference between the two crystallographically independent molecules in the asymmetric unit. Owing to the conformation of the rings and of the *tert*-butyl groups, the molecules are enantiomers. The coordination of the pivaloate bridge to the two B atoms results in an average\* (nonbonding)  $\text{B} \cdots \text{B}$  distance of  $2.496 (7) \text{ \AA}$ .

\* The standard deviations of the average values are calculated either by  $[\sum_{i=1}^n (p_i - \bar{p})^2 / n(n-1)]^{1/2}$  or  $(\sum_{i=1}^n \sigma_i^2)^{1/2} / n$ , the larger value being reported.  $\sigma_i$  is derived as usual from the errors quoted in Table 1.

Table 1. Atomic coordinates and equivalent isotropic temperature factors ( $\text{\AA}^2$ )

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{\text{eq}}/U_{\text{iso}}$
O(1)	0.7949 (1)	-0.0751 (3)	0.1062 (1)	0.056
O(2)	0.6759 (1)	-0.0763 (3)	0.1152 (1)	0.058
O(3)	0.3195 (1)	0.3119 (3)	0.3782 (1)	0.054
O(4)	0.2024 (2)	0.3002 (3)	0.3920 (1)	0.059
C(1)	0.7337 (2)	-0.1361 (4)	0.1050 (2)	0.041
C(2)	0.7306 (2)	-0.2816 (4)	0.0930 (2)	0.046
C(3)	0.7895 (3)	-0.3245 (4)	0.0562 (2)	0.069
C(4)	0.7413 (4)	-0.3503 (5)	0.1523 (2)	0.100
C(5)	0.6537 (3)	-0.3199 (4)	0.0620 (2)	0.071
C(6)	0.8498 (2)	0.1514 (4)	0.0850 (2)	0.049
C(7)	0.9261 (2)	0.0844 (5)	0.0840 (2)	0.065
C(8)	0.9712 (2)	0.0597 (5)	0.1436 (2)	0.062
C(9)	0.9262 (2)	0.0028 (4)	0.1889 (2)	0.060
C(10)	0.8505 (2)	0.0722 (4)	0.1920 (2)	0.050
C(11)	0.8577 (2)	0.2171 (5)	0.2125 (2)	0.059
C(12)	0.8870 (3)	0.3144 (4)	0.1708 (2)	0.066
C(13)	0.8545 (2)	0.2960 (4)	0.1066 (2)	0.061
C(14)	0.6166 (3)	0.1420 (5)	0.0760 (2)	0.066
C(15)	0.5398 (3)	0.0757 (5)	0.0747 (3)	0.091
C(16)	0.5087 (3)	0.0717 (5)	0.1335 (3)	0.090
C(17)	0.5664 (3)	0.0318 (5)	0.1853 (2)	0.086
C(18)	0.6409 (2)	0.0995 (5)	0.1868 (2)	0.057
C(19)	0.6388 (3)	0.2496 (5)	0.1975 (2)	0.076
C(20)	0.5994 (3)	0.3314 (5)	0.1471 (3)	0.087
C(21)	0.6185 (3)	0.2906 (5)	0.0874 (3)	0.083
C(31)	0.2607 (2)	0.3657 (4)	0.3901 (2)	0.043
C(32)	0.2597 (2)	0.5123 (4)	0.4008 (2)	0.048
C(33)	0.3366 (3)	0.5606 (4)	0.4266 (2)	0.079
C(34)	0.2362 (4)	0.5762 (5)	0.3420 (2)	0.099
C(35)	0.2043 (3)	0.5468 (4)	0.4422 (2)	0.073
C(36)	0.3753 (3)	0.1045 (5)	0.4304 (2)	0.067
C(37)	0.4524 (3)	0.1737 (6)	0.4358 (3)	0.094
C(38)	0.4917 (3)	0.1668 (5)	0.3814 (3)	0.095
C(39)	0.4416 (3)	0.1935 (5)	0.3233 (2)	0.079
C(40)	0.3653 (2)	0.1238 (4)	0.3178 (2)	0.057
C(41)	0.3708 (3)	-0.0267 (5)	0.3143 (2)	0.079
C(42)	0.4061 (3)	-0.0978 (5)	0.3699 (3)	0.096
C(43)	0.3785 (3)	-0.0453 (6)	0.4241 (3)	0.093
C(44)	0.1502 (2)	0.0704 (4)	0.4111 (2)	0.057
C(45)	0.0734 (3)	0.1369 (5)	0.4129 (2)	0.074
C(46)	0.0282 (2)	0.1646 (5)	0.3528 (2)	0.070
C(47)	0.0726 (2)	0.2216 (4)	0.3067 (2)	0.062
C(48)	0.1499 (2)	0.1550 (4)	0.3054 (2)	0.052
C(49)	0.1447 (2)	0.0124 (5)	0.2837 (2)	0.062
C(50)	0.1130 (2)	-0.0881 (5)	0.3238 (2)	0.065
C(51)	0.1445 (3)	-0.0725 (4)	0.3880 (2)	0.065
B(1)	0.8074 (3)	0.0664 (5)	0.1279 (2)	0.046
B(2)	0.6698 (3)	0.0695 (5)	0.1262 (2)	0.046
B(3)	0.3297 (2)	0.1625 (5)	0.3737 (2)	0.044
B(4)	0.1917 (2)	0.1586 (5)	0.3692 (2)	0.047
H(1)	0.739 (2)	0.111 (3)	0.128 (1)	0.036
H(2)	0.264 (2)	0.115 (3)	0.372 (1)	0.044

\* Lists of crystal structure data, observed structure-factor amplitudes and their standard deviations, bond lengths and angles, calculated structure factors, anisotropic thermal parameters for the non-H atoms, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43243 (59 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Selected interatomic distances (Å) and angles (°) with *e.s.d.*'s in parentheses

Molecule 1		Molecule 2	
H(1)–B(1)	1.33 (3)	H(2)–B(3)	1.29 (3)
H(1)–B(2)	1.32 (3)	H(2)–B(4)	1.37 (3)
O(1)–C(1)	1.268 (5)	O(3)–C(31)	1.263 (5)
O(1)–B(1)	1.521 (5)	O(3)–B(3)	1.524 (5)
O(2)–C(1)	1.262 (5)	O(4)–C(31)	1.255 (5)
O(2)–B(2)	1.500 (5)	O(4)–B(4)	1.528 (6)
C(1)–C(2)	1.494 (6)	C(31)–C(32)	1.501 (6)
C(6)–B(1)	1.589 (6)	C(36)–B(3)	1.571 (7)
C(10)–B(1)	1.586 (7)	C(40)–B(3)	1.574 (6)
C(14)–B(2)	1.590 (7)	C(44)–B(4)	1.581 (6)
C(18)–B(2)	1.594 (7)	C(48)–B(4)	1.572 (7)
B(1)···B(2)	2.495 (7)	B(3)···B(4)	2.497 (6)
O(1)···O(2)	2.200 (4)	O(3)···O(4)	2.197 (4)
B(1)–H(1)–B(2)	141 (3)	B(3)–H(2)–B(4)	140 (3)
C(1)–O(1)–B(1)	123.7 (3)	C(31)–O(3)–B(3)	123.7 (3)
C(1)–O(2)–B(2)	125.7 (3)	C(31)–O(4)–B(4)	123.4 (3)
O(1)–C(1)–O(2)	120.9 (4)	O(3)–C(31)–O(4)	121.6 (4)
O(1)–C(1)–C(2)	119.5 (4)	O(3)–C(31)–C(32)	119.3 (4)
O(2)–C(1)–C(2)	119.5 (4)	O(4)–C(31)–C(32)	119.0 (4)
O(1)–B(1)–H(1)	103 (1)	O(3)–B(3)–H(2)	104 (2)
O(1)–B(1)–C(6)	111.3 (4)	O(3)–B(3)–C(36)	111.5 (4)
O(1)–B(1)–C(10)	112.3 (3)	O(3)–B(3)–C(40)	111.6 (4)
C(6)–B(1)–C(10)	110.3 (3)	C(36)–B(3)–C(40)	111.7 (4)
O(2)–B(2)–H(1)	103 (1)	O(4)–B(4)–H(2)	102 (1)
O(2)–B(2)–C(14)	112.3 (4)	O(4)–B(4)–C(44)	111.3 (4)
O(2)–B(2)–C(18)	112.1 (4)	O(4)–B(4)–C(48)	111.9 (4)
C(14)–B(2)–C(18)	108.6 (4)	C(44)–B(4)–C(48)	110.8 (4)

This value signals a significant increase in B···B distance over the parent 9-BBN whose dimeric nature leads to a B···B distance of 1.818 (3) Å (Brauer & Krüger, 1973). It is still longer than the value observed when two 9-borabicyclo[3.3.1]nonanyl groups are doubly bridged by NH<sub>2</sub> groups: 2.294 (6) Å (Tsay & Krüger, 1970). The overall geometry of the novel six-membered B heterocycle shows some resemblance to the adducts of acyloxy(difluoro)boranes with alkoxy(difluoro)boranes, where perfectly planar OCOBOB heterocycles have been observed in one of two crystallographically independent molecules (Binder, Matheis, Deiseroth & Fu-Son, 1983).

The formation of a six-membered ring has drastic geometrical consequences for the 3*c*–2*e* BHB bond. Although being obscured by relatively high errors the observed B–H distances of 1.33 (3), 1.32 (3) Å [molecule (1)] and 1.29 (3), 1.37 (3) Å [molecule (2)] are equal within standard deviations. Their mean value of 1.33 (3) Å is in accord both with commonly observed B–H(bridging) distances and with the average B–H value of 1.3 Å for unsymmetric 3*c*–2*e* BHB bonds in binary boranes (Lipscomb, 1963; Hawthorne, 1967; Shore, 1975). The B–H–B angles of 141 (3) and 140 (3)° are significantly larger than the 94 (2)° observed in the parent bis(9-BBN) (Brauer & Krüger, 1973). Together with the average B–H distance of 1.33 (3) Å and the long B···B distances, a picture of a BHB 3*c*–2*e* bond can be drawn which is unparalleled in simple binary boranes where  $d(\text{B}\cdots\text{B}) = 1.70\text{--}1.86$ ,  $d(\text{B}\text{--}\text{H}(b)) = 1.3$  Å and  $\angle(\text{B}\text{--}\text{H}\text{--}\text{B}) = 100^\circ$  are commonly observed (Lipscomb, 1963, Hawthorne, 1967; Shore, 1975).

The formation of the central six-membered ring geometry forces the 9-BBN groups to bend 'backwards' from the bridging pivaloate as manifested, for example, by an angle of 111.1° between the normals to the planes through B(1), C(6), C(10) and B(2), C(14), C(18) (Fig. 1). However, this value includes contributions from a twisting of the 9-BBN groups against each other (Fig. 2) although this effect should be quite small [average value of the OBC angles: 111.8 (4)°].

As a consequence of the rehybridization of the B atoms towards *sp*<sup>3</sup> the B–C bond lengths [average: 1.582 (9) Å] are slightly elongated in comparison with those observed in bis(9-BBN) [1.567 (2) Å] (Brauer & Krüger, 1973). All other geometrical parameters of the 9-BBN groups are comparable with those in bis(9-BBN).

The unit-cell packing diagram (Fig. 2) shows the molecules to be arranged in layers along *c*. As indicated

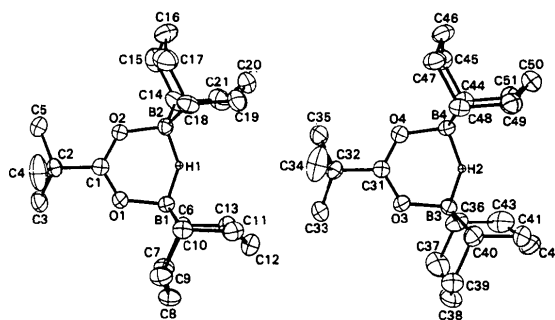


Fig. 1. ORTEP (Johnson, 1976) views of the two crystallographically independent molecules in the asymmetric unit, together with the numbering scheme (thermal ellipsoids at the 33% probability level).

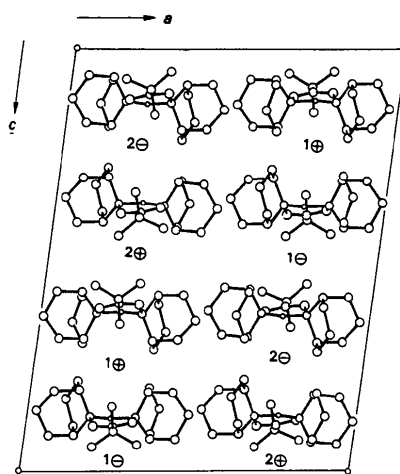


Fig. 2. Projection of the unit-cell contents onto the *ac* plane. The numbering denotes the crystallographically independent molecules as well as their relative position with respect to the projection plane.

in Fig. 2, molecules forming the respective layers have not the same height above the projection plane *ac* but rather are displaced against each other relative to the *b* axis. The relatively loose packing of the molecules in the layer structure is probably the origin of the slightly different conformations of the two crystallographically independent molecules and also for the high proportion of unobserved data. Apart from the layer structure no particular orientation phenomena or exceptionally short intermolecular distances are observed. On the basis of the room-temperature crystal structure analysis alone no definite conclusions can be drawn regarding a mechanism for the solid-state disproportionation reaction observed upon heating of the title compound.

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## Triterpenoids. I. Structure of 3 $\beta$ -Acetoxy-11-oxo-18 $\beta$ -olean-12-en-28-oic Acid Methyl Ester

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**Abstract.** C<sub>33</sub>H<sub>50</sub>O<sub>5</sub>, *M<sub>r</sub>* = 526.40, monoclinic, *P*2<sub>1</sub>, *a* = 6.824 (1), *b* = 12.658 (2), *c* = 17.848 (3) Å,  $\beta$  = 97.80 (1)°, *V* = 1527.4 (4) Å<sup>3</sup>, *D<sub>m</sub>* = 1.15 (1), *D<sub>x</sub>* = 1.14 Mg m<sup>-3</sup>, *Z* = 2,  $\lambda$ (Cu *K*α) = 1.54178 Å,  $\mu$ (Cu *K*α) = 0.519 mm<sup>-1</sup>, *F*(000) = 576, room temperature, final *R* = 0.070 for 1762 observed reflections. The molecule consists of five six-membered rings, *D* and *E* rings *cis* fused. Ring *C* has a slightly distorted sofa conformation. The H atom at the C(18)

asymmetric centre takes an equatorial position with respect to the *D* ring.

**Introduction.** The structure investigation of the title compound has been undertaken to facilitate the interpretation of <sup>1</sup>H NMR, <sup>13</sup>C NMR and MS data of pentacyclic triterpenoids with the 11-oxo-18 $\beta$ -olean-12-ene skeleton.

**Experimental.** The title compound, m.p. 522–524 K,  $[\alpha]_D^{22} = +79^\circ$  (CHCl<sub>3</sub>) was prepared by the method

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