Structure of 1-Trimethylamine-1-carba-*closo*-dodecaborane(11)

By K. Malý, V. Šubrtová and V. Petříček

Institute of Physics, Czechoslovak Academy of Sciences, 180 40 Praha 8, Czechoslovakia

Abstract. $C_4H_{20}B_{11}N$, $M_r = 201 \cdot 1$, monoclinic, $P2_1/m$, $a = 6 \cdot 982$ (1), $b = 10 \cdot 689$ (3), $c = 8 \cdot 673$ (1) Å, $\beta = 110 \cdot 35$ (1)°, $V = 606 \cdot 9$ (2) Å^3, $Z = 2$, $D_m = 1 \cdot 096$, $D_x = 1 \cdot 101$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0 \cdot 71069$ Å, $\mu = 0 \cdot 47$ cm^{-1}, $F(000) = 212$, room temperature, $R = 0 \cdot 053$ for 1450 observed reflections. The $N(CH_3)_3$ group, which has normal bond lengths and angles, is attached to the C atom of the icosahedral *closo*-carbaborane cage with a C—N bond length of $1 \cdot 547$ (1) Å. The molecule has crystallographic m symmetry. C(1), B(2), B(9), B(12), N and one methyl group are situated on the mirror plane ($y = 0 \cdot 25$).

Experimental. Colourless transparent crystals prepared in the Institute of Inorganic Chemistry, Czechoslovak Academy of Sciences (Plešek, Jelínek, Drdátová,

Heřmánek & Štíbr, 1984). D_m by flotation. Crystal size $0 \cdot 3 \times 0 \cdot 3 \times 0 \cdot 4$ mm, Hilger & Watts diffractometer, Mo $K\alpha$ radiation, Zr filter. Monoclinic cell parameters and standard deviations by least squares from 33 high-order reflections. 1858 independent reflections measured by $\omega/2\theta$ scan, $[(\sin\theta/\lambda)_{max} = 0 \cdot 704$ Å$^{-1}]$ for $h = -9 \rightarrow 9$, $k = 0 \rightarrow 12$, $l = 0 \rightarrow 14$. 1450 reflections observed $[I > 1 \cdot 96\sigma(I)]$. Three standard reflections, insignificant changes. Data scaled according to standard reflections and corrected for Lorentz and polarization effects, but not for absorption or extinction. Structure solved by direct methods with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), H atoms from difference Fourier synthesis. Refinement with local version of full-matrix least-squares program *ORFLS* (Busing, Martin & Levy, 1962) with anisotropic temperature factors for non-H atoms and individual isotropic factors for H atoms. Function minimized $\sum w(|F_o| - |F_c|)^2$ with $w^{-1} = \sigma^2(F_o) + (0 \cdot 03F_o)^2$. In final cycle $R = 0 \cdot 053$, $wR = 0 \cdot 070$, average $\Delta/\sigma < 0 \cdot 01$, $(\Delta/\sigma)_{max} = 0 \cdot 03$, $\Delta\rho_{max} = 0 \cdot 21$, $\Delta\rho_{min} = -0 \cdot 33$ e Å^{-3} in final difference Fourier map. Scattering factors from *International Tables for X-ray Crystallography* (1974) except for H (Stewart, Davidson & Simpson, 1965). Calculations performed on Siemens 7536 computer. Table 1* lists

Table 1. *Fractional coordinates and B_{eq} values (Hamilton, 1959) with e.s.d.'s in parentheses for non-hydrogen atoms*

$$B_{eq} = \tfrac{4}{3}\sum_i\sum_j \beta_{ij}\mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
C(1)	0·2675 (2)	0·2500	0·4775 (1)	1·88 (3)
B(2)	0·0067 (2)	0·2500	0·3836 (2)	2·46 (4)
B(3)	0·1533 (2)	0·3851 (1)	0·3770 (1)	2·46 (3)
B(4)	0·3930 (2)	0·3333 (1)	0·3715 (1)	2·40 (3)
B(7)	−0·0402 (2)	0·3329 (1)	0·1972 (1)	2·68 (3)
B(8)	0·1991 (2)	0·3842 (1)	0·1895 (1)	2·85 (3)
B(9)	0·3475 (2)	0·2500	0·1870 (2)	2·85 (4)
B(12)	0·0792 (3)	0·2500	0·0778 (2)	2·84 (4)
N	0·3562 (2)	0·2500	0·6676 (1)	2·42 (3)
C(2)	0·5880 (3)	0·2500	0·7332 (2)	4·35 (5)
C(3)	0·2901 (2)	0·3639 (1)	0·7384 (1)	3·80 (4)

Table 2. *Bond distances (Å) and selected angles (°)*

C(1)—B(2)	1·717 (2)	B(4)—B(8)	1·770 (1)
C(1)—B(3)	1·731 (1)	B(4)—B(9)	1·761 (2)
C(1)—B(4)	1·722 (2)	B(7)—B(8)	1·782 (2)
C(1)—N	1·547 (1)	B(7)—B(7^i)†	1·772 (2)
B(2)—B(3)	1·782 (2)	B(7)—B(12)	1·776 (2)
B(2)—B(7)	1·771 (2)	B(8)—B(9)	1·774 (2)
B(3)—B(4)	1·779 (2)	B(8)—B(12)	1·770 (1)
B(3)—B(7)	1·762 (1)	B(9)—B(12)	1·781 (2)
B(3)—B(8)	1·763 (1)	N—C(2)	1·517 (2)
B(4)—B(4^i)*	1·781 (2)	N—C(3)	1·506 (1)

C(1)—N—C(2)	112·3 (1)	C(2)—N—C(3)	106·6 (1)
C(1)—N—C(3)	111·6 (1)		

Symmetry code: (i) $x, \tfrac{1}{2}-y, z$.

* B(5) in Fig. 1.   † B(11) in Fig. 1.

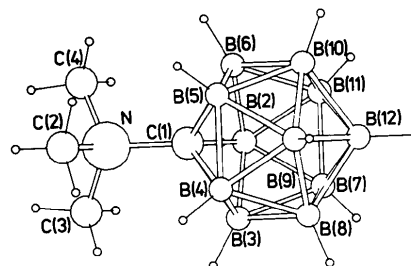

Fig. 1. Structure and numbering (Adams, 1972) of $(CH_3)_3NCB_{11}H_{11}$ [atoms labelled B(5), B(6), B(10), B(11) and C(4) are related by the mirror plane ($y = 0 \cdot 25$) to atoms B(4), B(3), B(8), B(7) and C(3), respectively].

fractional coordinates and equivalent isotropic thermal parameters, Table 2 bond distances and selected angles. The molecular structure is shown in Fig. 1.

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Structures of Carbocyclic Analogues of Penicillin. 2. *N*-(3,4-Dihydroxy-7-oxobicyclo[3.2.0]hept-6-yl)succinimide

BY B. SHELDRICK AND D. AKRIGG

Astbury Department of Biophysics, University of Leeds, Leeds LS2 9JT, England

AND M. I. PAGE AND G. COX

Department of Chemical Sciences, The Polytechnic, Queensgate, Huddersfield HD1 3DH, England

Abstract. $C_{11}H_{13}NO_5$, $M_r = 239.23$, triclinic, $P1$, $a = 5.470$ (2), $b = 6.066$ (1), $c = 8.953$ (2) Å, $\alpha = 98.55$ (2), $\beta = 97.31$ (2), $\gamma = 113.96$ (3)°, $V = 262.6$ Å^3, $Z = 1$, $D_x = 1.51$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 0.92$ mm^{-1}, $F(000) = 126$, $T = 293$ K, $R = 0.046$ for 978 unique observed reflections. The determination confirms the chemical structure. The four-membered ring is twisted by 13.4 (4)° and the least-squares best plane through it makes an angle of 108.3 (13)° with the pentane ring. Bond lengths and angles are normal.

Experimental. Material prepared by M. I. Page and G. Cox and crystallized from ethanol. Tabular crystal 0.20 × 0.15 × 0.40 mm. Enraf–Nonius CAD-4F diffractometer. No correction for absorption. $2\theta_{max} = 140°$, $+/-h,k,l$; 1917 reflections measured and 41 classed as unobserved ($I < 0$). Check reflection $10\bar{3}$: average count = 9897, calculated σ (of the distribution) = 206 (2.1%). Cell dimensions from θ measurements of 24 reflections (16 < θ < 50°). Data merged using *SHELX*76 (Sheldrick, 1976) giving 991 unique reflections, index range $h +/-6$, $k +/-7$, l 0/10; merging $R_{int} = 0.026$. *SHELX*76 used to solve structure, by direct methods. Least-squares refinement; 13 reflec-

tions, thought to show the effects of extinction, were omitted from the refinement and an anisotropic scale factor (Shakked & Rabinovich, 1977) was used; positional parameters of all atoms and anisotropic thermal vibration parameters for non-H atoms refined; U_{iso} for H fixed at value of U_{eq} of bonded atom; $\sum w(\Delta F)^2$ minimized with $w = 1/\sigma^2(F)$. H atoms from difference Fourier syntheses. In final cycle max. $\Delta/\sigma = 0.051$, average = 0.007. $\Delta\rho$ in final difference Fourier map within +0.15 and −0.20 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). $R = 0.046$, $wR = 0.058$ for 978 observed reflections [$F_o > 3\sigma(F_o)$].

Atom parameters are given in Table 1,* with bond distances in Table 2. Fig. 1 drawn with *PLUTO*78 (Motherwell & Clegg, 1978) shows the molecule numbering scheme and is a projection along **a**.