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Bridged Bis(dicarbadodecaborane)cobalt(III)

I. Císařová

Department of Inorganic Chemistry, Charles University, Albertov 2, 128 40 Praha 2, Czech Republic

K. Malý and L. Hummel

Institute of Physics, Czech Academy of Sciences, Na Slovance 2, 180 40 Praha 8, Czech Republic

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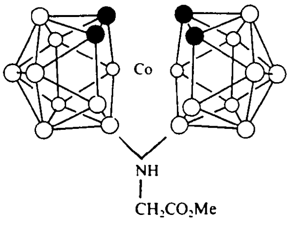
Abstract

The molecule of 8,8′-μ-(methoxycarbonylmethylamino)-3,3′-*commo*-bis[1,2-dicarba-3-cobalta-*closo*-dodecaborane(11)], $[Co(C_7H_{26}B_{18}NO_2)]$, comprises two dicarbaborane ligands coordinated to the Co atom to form a 'sandwich' compound linked by pentagonal faces. The ligands are also linked by a monoatomic nitrogen bridge to which a methoxycarbonylmethyl group is bound, B—N bond lengths being 1.577 (4) and 1.563 (4) Å.

Comment

The present study reports the results of the X-ray investigation of 8,8′-CH_3OCOCH_2NH-3-Co-(1,2-$C_2B_9H_{10})_2$ and forms part of our continuing study of the structure of metallocarbaboranes prepared at the Institute of Inorganic Chemistry of the Czech Academy of Sciences (Janoušek, Plešek, Heřmánek & Baše, 1981).

The significant feature of the title molecule (Fig. 1) is the angle between the open pentagonal faces of the dicarbaborane ligands, *i.e.* the angle, φ, between the best plane through the atoms C(1), C(2), B(4), B(7), B(8) and that through C(1′), C(2′), B(4′), B(7′), B(8′) is 26.0 (1)°. In Table 3, the title compound (III) is compared with analogous compounds $Co(C_2B_9H_{11})_2^-$ (I) (Borodinsky, Sinn & Grimes, 1982) and $Co(C_2B_9H_{10})_2SCH_2COOCH_3$ (II) (Císařová & Petříček, 1986). The geometric parameters derived from our molecule are in good agreement with the conclusion inferred from a previous paper by Císařová & Petříček (1986) that the increasing angle affects the position of the Co atom, which moves towards the bridge. Moreover, with the increase in Co—C distances the C—C distance tends to become shorter. The molecules are dimeric, connected by two symmetrically related hydrogen bonds (through inversion). The interatomic distances N···O(1^i) 2.885 (4) and H(N)···O(1^i) 2.05 (4) Å, and angle N—H···O(1^i) 160 (3)°, are close to the mean values for this type of hydrogen bond in organic compounds (Taylor, Kennard & Versichel, 1984).



(III)

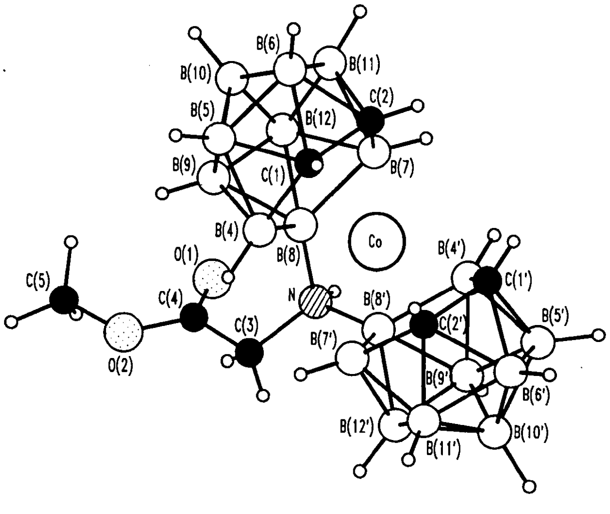


Fig. 1. View of the title molecule with the atom-numbering scheme.

Experimental

Crystal data

$[Co(C_7H_{26}B_{18}NO_2)]$
M_r = 409.80
Tetragonal
$I4_1/a$ (origin at −1 on glide plane *b*)
a = 23.971 (2) Å
c = 14.398 (2) Å

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 32 reflections
θ = 10.8–19.0°
μ = 0.8308 mm^{-1}
T = 293 K

$V = 8273$ (2) Å^3
$Z = 16$
$D_x = 1.316$ Mg m^{-3}
$D_m = 1.326$ Mg m^{-3}
D_m measured by flotation

Spherical
0.6 × 0.6 × 0.5 mm
Red

Data collection

Hilger & Watts diffractometer
θ-2θ scans
Absorption correction: none
5007 measured reflections
5007 independent reflections
3647 observed reflections [$I > 1.96\sigma(I)$]

$\theta_{\max} = 28°$
$h = 0 \rightarrow 29$
$k = 0 \rightarrow 29$
$l = 0 \rightarrow 19$
3 standard reflections monitored every 30 reflections intensity variation: 6%

Refinement

Refinement on F
$R = 0.051$
$wR = 0.064$
$S = 1.33$
3647 reflections
366 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F) + 0.0009F^2]$
$(\Delta/\sigma)_{\max} = 0.04$
$\Delta\rho_{\max} = 0.55$ e Å^{-3}
$\Delta\rho_{\min} = -0.36$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{\rm eq} = (4/3)\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
Co	0.04881 (2)	0.02594 (2)	0.81338 (3)	2.22 (1)
C(1)	−0.0019 (1)	0.0818 (1)	0.8857 (2)	2.99 (8)
C(2)	0.0432 (1)	0.1130 (1)	0.8251 (2)	3.10 (8)
B(4)	−0.0375 (1)	0.0332 (2)	0.8262 (2)	2.76 (8)
B(5)	−0.0680 (2)	0.0989 (2)	0.8509 (3)	3.4 (1)
B(6)	−0.0165 (2)	0.1501 (2)	0.8535 (3)	3.8 (1)
B(7)	0.0460 (2)	0.0904 (1)	0.7143 (3)	2.65 (8)
B(8)	−0.0073 (1)	0.0372 (1)	0.7103 (2)	2.39 (7)
B(9)	−0.0720 (2)	0.0707 (2)	0.7359 (3)	3.03 (9)
B(10)	−0.0592 (2)	0.1429 (2)	0.7534 (3)	3.6 (1)
B(11)	0.0130 (2)	0.1548 (2)	0.7416 (3)	3.7 (1)
B(12)	−0.0213 (2)	0.1058 (2)	0.6679 (3)	3.01 (9)
C(1′)	0.1296 (1)	−0.0015 (1)	0.8462 (2)	2.72 (7)
C(2′)	0.0838 (1)	−0.0336 (1)	0.9038 (2)	2.89 (8)
B(4′)	0.1156 (1)	0.0003 (1)	0.7308 (2)	2.45 (8)
B(5′)	0.1665 (2)	−0.0457 (2)	0.7766 (3)	2.95 (9)
B(6′)	0.1471 (2)	−0.0668 (2)	0.8885 (3)	3.21 (9)
B(7′)	0.0314 (2)	−0.0586 (2)	0.8379 (2)	2.84 (8)
B(8′)	0.0505 (1)	−0.0376 (1)	0.7218 (2)	2.40 (7)
B(9′)	0.1154 (2)	−0.0701 (1)	0.6958 (2)	2.60 (8)
B(10′)	0.1357 (2)	−0.1115 (2)	0.7923 (3)	3.09 (9)
B(11′)	0.0845 (2)	−0.1038 (2)	0.8792 (3)	3.18 (9)
B(12′)	0.0639 (2)	−0.1072 (2)	0.7610 (3)	3.97 (9)
N	0.0041 (1)	−0.0177 (1)	0.6531 (2)	2.48 (6)
O(1)	−0.0722 (1)	−0.0021 (1)	0.5089 (2)	4.14 (7)
O(2)	−0.1370 (1)	−0.0415 (1)	0.5982 (2)	3.59 (6)
C(3)	−0.0444 (1)	−0.0553 (2)	0.6408 (3)	3.21 (9)
C(4)	−0.0857 (1)	−0.0300 (1)	0.5742 (2)	2.92 (8)
C(5)	−0.1806 (2)	−0.0184 (2)	0.5400 (4)	4.8 (1)

Table 2. *Interatomic distances* (Å)

Metal-cage distances			
Co—C(1)	2.086 (3)	Co—C(1′)	2.099 (3)
Co—C(2)	2.097 (3)	Co—C(2′)	2.106 (3)
Co—B(4)	2.085 (3)	Co—B(4′)	2.087 (3)
Co—B(7)	2.103 (4)	Co—B(7′)	2.098 (4)
Co—B(8)	2.022 (3)	Co—B(8′)	2.014 (3)
Cage distances			
C(1)—C(2)	1.578 (5)	C(1′)—C(2′)	1.576 (5)
C(1)—B(4)	1.678 (5)	C(1′)—B(4′)	1.695 (5)
C(1)—B(5)	1.711 (5)	C(1′)—B(5′)	1.705 (5)
C(1)—B(6)	1.736 (6)	C(1′)—B(6′)	1.732 (5)
C(2)—B(6)	1.735 (6)	C(2′)—B(6′)	1.729 (5)
C(2)—B(7)	1.685 (5)	C(2′)—B(7′)	1.683 (5)
C(2)—B(11)	1.724 (6)	C(2′)—B(11′)	1.720 (5)
B(4)—B(5)	1.770 (6)	B(4′)—B(5′)	1.771 (5)
B(4)—B(8)	1.823 (5)	B(4′)—B(8′)	1.812 (5)
B(4)—B(9)	1.784 (5)	B(4′)—B(9′)	1.761 (5)
B(5)—B(6)	1.740 (6)	B(5′)—B(6′)	1.751 (5)
B(5)—B(9)	1.791 (6)	B(5′)—B(9′)	1.789 (5)
B(5)—B(10)	1.768 (6)	B(5′)—B(10′)	1.756 (5)
B(6)—B(10)	1.776 (6)	B(6′)—B(10′)	1.772 (6)
B(6)—B(11)	1.763 (6)	B(6′)—B(11′)	1.748 (6)
B(7)—B(8)	1.805 (5)	B(7′)—B(8′)	1.804 (5)
B(7)—B(11)	1.778 (5)	B(7′)—B(11′)	1.773 (6)
B(7)—B(12)	1.785 (5)	B(7′)—B(12′)	1.786 (5)
B(8)—B(9)	1.785 (5)	B(8′)—B(9′)	1.779 (5)
B(8)—B(12)	1.786 (5)	B(8′)—B(12′)	1.790 (5)
B(8)—N	1.577 (4)	B(8′)—N	1.563 (4)
B(9)—B(10)	1.776 (6)	B(9′)—B(10′)	1.776 (5)
B(9)—B(12)	1.773 (5)	B(9′)—B(12′)	1.789 (5)
B(10)—B(11)	1.762 (6)	B(10′)—B(11′)	1.762 (6)
B(10)—B(12)	1.770 (6)	B(10′)—B(12′)	1.783 (6)
B(11)—B(12)	1.785 (6)	B(11′)—B(12′)	1.773 (5)
Other distances			
N—C(3)	1.482 (4)	O(2)—C(5)	1.450 (6)
O(1)—C(4)	1.198 (4)	C(3)—C(4)	1.507 (5)
O(2)—C(4)	1.307 (4)		

Table 3. *Comparison of structural parameters* (Å, °) *for* $Co(C_2B_9H_{11})_2^-$ (I), $Co(C_2B_9H_{10})_2SCH_2COOCH_3$ (II) *and* $Co(C_2B_9H_{10})_2NHCH_2COOCH_3$ (III)

	(I)	(II)	(III)
Co—C	2.045 (9)	2.079 (4)	2.097 (4)
Co—B(8)	2.105 (4)	2.043 (2)	2.022 (3)
C—C	1.624 (4)	1.593 (10)	1.577 (2)
φ*	3.8 (2)	16.6 (1)	26.0 (1)

* φ is the angle between the best planes through C(1), C(2), B(4), B(7), B(8) and C(1′), C(2′), B(4′), B(7′), B(8′).

The title compound was obtained on alkylation of $Co(C_2B_9H_{10})_2NH$ (Plešek, Heřmánek, Baše, Todd & Wright, 1976) with chloroacetic acid in methanolic KOH solution. The resulting acid, $Co(C_2B_9H_{10})_2NHCH_2COOH$, was esterified with methanol and sulfuric acid. The crystals were grown from benzenic solution by slow diffusion of hexene.

The integrated intensities were measured by the learnt-profile method (Clegg, 1981). Corrections were applied for Lorentz and polarization effects but not for absorption since $\mu R = 0.26$ and $A^*(\theta = 0°) = 1.56, A^*(\theta = 30°) = 1.55$. The structure was solved using the Patterson method to locate the Co atom, followed by successive Fourier syntheses, which provided the positions of all the remaining non-H atoms. The non-H atoms were refined with anisotropic temperature factors by the full-matrix least-squares method. H atoms were determined from a difference Fourier map and refined isotropically. All calculations were performed on a Siemens 7536 computer with local programs (Petříček & Malý, 1981).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71474 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1022]

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Tribenzylammonium Dibenzylammonium Tetrachlorocuprate

GUANG-FU ZENG,* MEI QIN, YONG-HUA LIN AND SHI-QUAN XI

Changchun Institute of Applied Chemistry,
Academia Sinica, 130022 Changchun,
People's Republic of China

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Abstract

In the $CuCl_4^{2-}$ anion of the title compound, $(C_{21}H_{22}N)(C_{14}H_{16}N)[CuCl_4]$, the copper coordination is intermediate between tetrahedral and square planar.

Comment

Two types of metal coordination occur in compounds of the general formula $(C_nH_{2n+1}NH_3)_2MCl_4$: octahedral (M = Cu, Cd, Mn, Fe, Hg) or tetrahedral (M = Zn, Co) (Ciajolo, Corradini & Pavone, 1977). However, square-planar copper coordination is found in anilinium tetrachlorocuprate (Larsen, 1974). The distorted-tetrahedral copper coordination in the title compound, (I), contrasts with the structures of $(C_3H_7NH_3)_2CuCl_4$ (Barendregt & Schenk, 1970) and $(C_2H_5NH_3)_2CuCl_4$ (Steadman & Willet, 1970), which contain octahedrally coordinated metal atoms, and also differs from the structure of anilinium tetrachlorocuprate (Larsen, 1970). In addition, the average Cu—Cl distance in (I) is shorter than the corresponding values in the octahedral complexes. In (I) the shortest N···Cl distances involving the $(C_6H_5CH_2)_2NH_2^+$ and $(C_6H_5CH_2)_3NH^+$ cations are 3.234 and 3.251 Å, respectively.

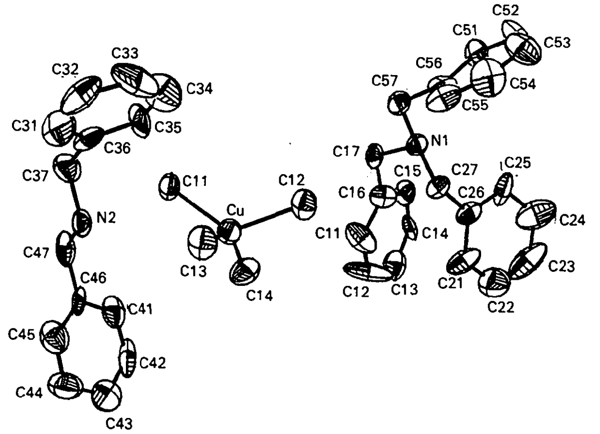

Fig. 1. Atomic numbering scheme of the non-H atoms of the title compound with 50% probability displacement ellipsoids.

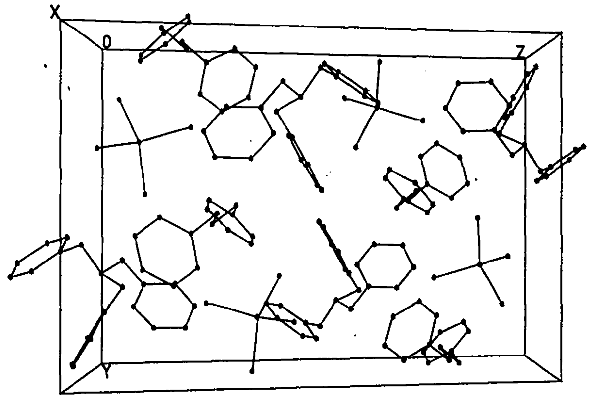

Fig. 2. Molecular packing in the unit cell of the title compound.

Experimental

Crystal data

$(C_{21}H_{22}N)(C_{14}H_{16}N)[CuCl_4]$
M_r = 692.05
Monoclinic
$P2_1/n$
a = 11.823 (5) Å
b = 14.433 (5) Å
c = 20.857 (5) Å
β = 102.65 (3)°
V = 3472.7 Å^3
Z = 4
D_x = 1.32 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 2.3–12.4°
μ = 0.956 mm^{-1}
T = 298 K
Lamina
0.62 × 0.46 × 0.12 mm
Brownish yellow

Data collection

Nicolet $R3m/E$ diffractometer
$R_{\rm int}$ = 0.07
$\theta_{\rm max}$ = 48°

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