Program(s) used to refine structure: *SHELXL*93. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

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A *closo*-12-Vertex Dinickelaborane: $[(\eta^5\text{-}C_5H_5)_2Ni_2B_{10}H_{10}]$

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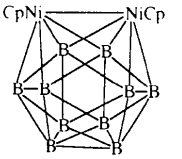
Abstract

In the title compound, 1,2-di-η^5-cyclopentadienyl-1,2-dinickela-*closo*-dodecaborane, $[Ni_2(B_{10}H_{10})(C_5H_5)_2]$, the cluster has a *closo*-twelve-vertex icosahedral $\{Ni_2B_{10}\}$ framework. The Ni atoms occupy adjacent vertices of the polyhedron and each Ni atom is coordinated by a η^5-cyclopentadienyl ring.

Comment

Di- or polymetallaborane clusters are well known in metallaborane chemistry (Crook *et al.*, 1982; Pipal & Grimes, 1977, 1979*a*,*b*; Bowser *et al.*, 1979), but twelve-vertex metallaborane species are rare. There are two methods for the synthesis of these species. One is *nido*-cage closure, as used in the preparation of dicobalta- (Schubert *et al.*, 1988) and diplatinaboranes (Cheek *et al.*, 1985). The other, developed by Hawthorne's group, is *closo*-polyhedral cage expansion; Hawthorne and co-workers reported the synthesis of *closo*-$[(C_5H_5)_2Ni_2B_{10}H_{10}]$ and *closo*-$[(C_5H_5)_2NiCoB_{10}H_{10}]^-$ (Leyden *et al.*, 1978), which were characterized by NMR spectroscopy. We have previously isolated and characterized the novel *closo*-twelve-vertex dinickelaborane $[(\mu\text{-}1,2\text{-}Cl)\text{-}3\text{-}Cl\text{-}6\text{-}(PPh_3)\text{-}(\mu\text{-}1,4\text{-}Ph_2PC_6H_4)\text{-}(\mu\text{-}2,8\text{-}Ph_2PC_6H_4)\text{-}1,2\text{-}Ni_2B_{10}H_6]$ (Dou *et al.*, 1997) by X-ray diffraction analysis. We now report the crystal and molecular structure of *closo*-$[(\eta^5\text{-}C_5H_5)_2Ni_2B_{10}H_{10}]$, (I).



(I)

The structure of the title compound consists of a *closo*-twelve-vertex icosahedral $\{Ni_2B_{10}\}$ framework. The Ni atoms occupy adjacent vertices of the polyhedron. Although the Ni—Ni distance of 2.4233 (6) Å is similar to that found in another *closo*-twelve-vertex

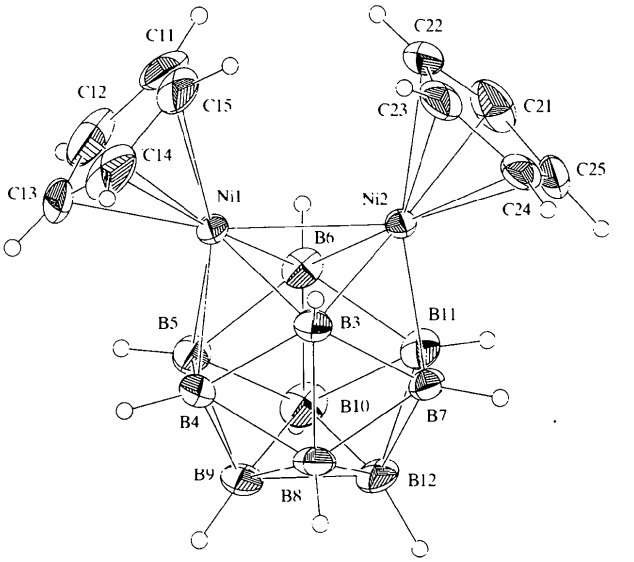


Fig. 1. View of the title compound with 30% probability displacement ellipsoids. H atoms are shown as spheres of arbitrary radii.

dinickelaborane [2.4008 (12) Å; Dou *et al.*, 1997] and those in other nickelaborane clusters [2.354 (1) (Bowser *et al.*, 1979) and 2.404 (1) Å (Salentine *et al.*, 1976)], it is the longest yet reported. The average Ni—B bond length [2.106 (18) Å] is also slightly longer than the average Ni—B distances in certain other clusters: 2.037 (14) Å in $[(C_5H_5)_4Ni_4B_4H_4]$ (Bowser *et al.*, 1979), 2.03 (2) Å in $[(C_5H_5)_3Ni_3B_6H_6]$ (Lagun *et al.*, 1994) and 2.060 (14) Å in $[(C_5H_5)_3Ni_3CB_5H_5]$ (Salentine *et al.*, 1976). The variation of the bond lengths is probably related to the differing metal-atom coordination numbers in the metallaborane systems. Each Ni atom coordinates to a η^5-cyclopentadienyl (Cp) ring and the mean Ni—C_{Cp} distance [2.09 (2) Å] is slightly shorter than those (2.13–2.15 Å) in the clusters mentioned above which contain Cp ligands.

Experimental

The title compound was prepared according to the literature method of Leyden *et al.* (1978) and recrystallized from an *n*-pentane/dichloromethane (3/1) solution.

Crystal data

$[Ni_2(B_{10}H_{10})(C_5H_5)_2]$
M_r = 365.78
Monoclinic
$P2_1/c$
a = 12.544 (1) Å
b = 9.017 (2) Å
c = 15.120 (1) Å
β = 110.54 (1)°
V = 1601.5 (4) Å^3
Z = 4
D_x = 1.517 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 9.31–14.31°
μ = 2.331 mm^{-1}
T = 293 (2) K
Square prism
0.54 × 0.20 × 0.13 mm
Dark green

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (North *et al.*, 1968)
T_{min} = 0.366, T_{max} = 0.752
3083 measured reflections
2961 independent reflections

2343 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 25.47°
$h = -14 \rightarrow 14$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 18$
3 standard reflections every 200 reflections
intensity variation: 1.6%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.099
S = 1.100
2961 reflections
279 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.061P)^2 + 0.57P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.631 e Å^{-3}
$\Delta\rho_{min}$ = −0.696 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Ni1—C13	2.041 (4)	Ni2—C25	2.062 (4)
Ni1—C14	2.078 (4)	Ni2—C21	2.082 (4)
Ni1—B5	2.079 (4)	Ni2—C24	2.087 (3)
Ni1—B4	2.090 (4)	Ni2—B7	2.090 (4)
Ni1—C12	2.091 (5)	Ni2—C23	2.097 (4)
Ni1—C15	2.112 (4)	Ni2—B11	2.099 (4)
Ni1—C11	2.118 (4)	Ni2—B3	2.108 (3)
Ni1—B3	2.124 (4)	Ni2—C22	2.114 (4)
Ni1—B6	2.127 (4)	Ni2—B6	2.129 (4)
Ni1—Ni2	2.4233 (6)		
B5—Ni1—Ni2	97.09 (12)	B7—Ni2—Ni1	97.53 (11)
B4—Ni1—Ni2	97.21 (11)	B11—Ni2—Ni1	97.21 (11)
B3—Ni1—Ni2	54.76 (10)	B3—Ni2—Ni1	55.38 (10)
B6—Ni1—Ni2	55.33 (11)	B6—Ni2—Ni1	55.25 (10)

H atoms were located using Fourier methods and refined isotropically.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *SDP-Plus* (Frenz, 1985). Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ZORTEP* (Zsolnai & Huttner, 1994). Software used to prepare material for publication: *SHELXL97*.

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