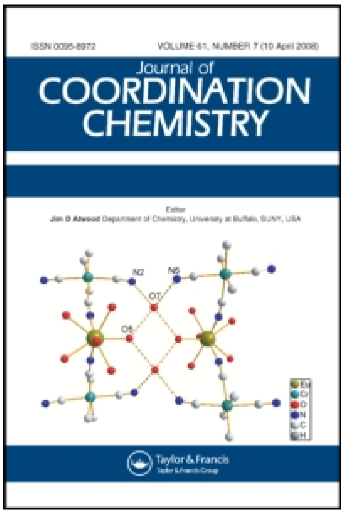

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# Solvothermal synthesis and crystal structure of the novel <i>closo</i> 12-vertex nickeladodecaborane cluster $\mathrm{H_2[Cl_2NiB_{11}H_6}$-2,3,4,5,6-(O<i>i</i>Pr)$_5$]

Libin Wu[a]; Jianmin Dou[a]; Qingliang Guo[a]; Dacheng Li[a]; Daqi Wang[a]
[a] Department of Chemistry, Liaocheng University, Liaocheng 252059, P.R. China

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# Solvothermal synthesis and crystal structure of the novel *closo* 12-vertex nickeladodecaborane cluster $H_2[Cl_2NiB_{11}H_6\text{-}2,3,4,5,6\text{-}(OiPr)_5]$

LIBIN WU, JIANMIN DOU*, QINGLIANG GUO,
DACHENG LI and DAQI WANG

Department of Chemistry, Liaocheng University, Liaocheng 252059, P.R. China

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The reaction of $[NiCl_2(PPh_3)_2]$ with $B_{11}H_{14}^-$ in *i*PrOH under solvothermal conditions gives a nickeladodecaborane cluster: $H_2[Cl_2NiB_{11}H_6\text{-}2,3,4,5,6\text{-}(OiPr)_5]$ which was characterized by FT-IR, NMR spectroscopy and elemental analysis. It was also characterized by an X-ray diffraction study of a single crystal. The title cluster belongs to monoclinic, space group $P2(1)/n$, with crystal cell parameters: $a = 12.355(13)$ Å, $b = 18.471(19)$ Å, $c = 12.956(14)$ Å, $\beta = 93.524(18)°$, $Z = 4$, $D_{\mathrm{Calcd}} = 1.242\,\mathrm{Mg\,m^{-3}}$, $F(000) = 1160$, $R_1 = 0.0746$, $wR_2 = 0.1829$. The cluster has a *closo* 12-vertex $\{NiB_{11}\}$ icosahedral skeleton, in which Ni connects with two Cl atoms and five B atoms. O*i*Pr groups substituted all five H atoms on the boron cage atoms adjacent to Ni.

*Keywords: Closo*; Nickeladodecaborane; Solvothermal synthesis; Crystal structure

## 1. Introduction

Since Greenwood discovered the first metallaborane $[Me_3NH][B_{10}H_{12}AlH_2]$ in 1965 [1], metallaboranes have been studied extensively. Several hundred polyhedral compounds have been synthesized and characterized, including about seventy types from 2-vertex to 26-vertex [2–4]. This field continues to maintain considerable momentum. There has been lively theoretical and experimental interest in 12-vertex metallaboranes and potential starting substrates such as *nido*-$B_{11}H_{14}^-$, which is easily made from $NaBH_4$. However, 12-vertex metallaboranes that incorporate an 11-borane configuration as a structural unit $\{MB_{11}\}$ are poorly represented. To our knowledge, *closo* 12-vertex monometallaboranes structurally characterized by X-ray diffraction include $[Br_2(PMe_2Ph)_3PdB_{11}H_8]$ [5], $[(C_5H_5)Ni(B_{11}H_{11})]^-$ [6], $[B_{11}H_{11}SnCH_3]PPh_3CH_3CH_2Cl_2$ [7], $[B_{11}H_{11}Sb]^-$, $[B_{11}H_{11}Bi]^-$ [8, 9], $[(PPh_3)_2RhB_{11}H_{11}]^{3-}$ [10], $[B_{11}H_{11}AlCH_3]^{2-}$ [11].

The solvothermal technique involves heating reaction mixtures in a sealed vessel such that reaction temperature greater than the boiling point of the solvents can be reached,

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*Corresponding author. Tel.: 86 635 8239069. Fax: 86 635 8239121. Email: jmdou@lctu.edu.cn

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typically under autogenous pressure [12]. Superheated solvents exhibit reduced viscosity and very different solubilising properties compared to refluxing conditions, leading to very different chemistry [13]. We have introduced solvothermal techniques into the synthesis of metallaborane and obtained two novel clusters $[(PPh_3)_2(\mu\text{-}PPh_2)Pt_2B_9H_6\text{-}3,10,11\text{-}(O\textit{i}Pr)_3]$ and $[(PPh_3)_2(\mu\text{-}PPh_2)Pt_2B_9H_6\text{-}3,10\text{-}(O\textit{i}Pr)_2\text{-}11\text{-}Cl]$ [14]. When reaction of $[NiCl_2(PPh_3)_2]$ with $B_{11}H_{14}^-$ in *i*PrOH was carried out under solvothermal condition, a novel *closo* 12-vertex nickelaborane $H_2[Cl_2NiB_{11}H_6\text{-}2,3,4,5,6\text{-}(O\textit{i}Pr)_5]$ containing the $\{NiB_{11}\}$ skeleton was obtained. It was characterized by FT-IR spectroscopy, elemental analysis and single-crystal X-ray diffraction.

## 2. Experimental

### 2.1. *Preparation*

The reaction was carried out under an atmosphere of dry, oxygen-free dinitrogen with exclusion of air and moisture. Some subsequent manipulations were performed in air. *i*PrOH and $CH_2Cl_2$ was distilled from $CaH_2$ prior to use. $[Et_3NH][B_{11}H_{14}]$ was prepared by the method of Dunks *et al.* [15] and $[NiCl_2(PPh_3)_2]$ [16] were prepared according to literature methods. $[Et_3NH][B_{11}H_{14}]$ (0.188 g, 0.8 mmol), $[NiCl_2(PPh_3)_2]$ (0.260 g, 0.4 mmol) and 25 mL distilled *i*PrOH were mixed in a Teflon-lined autoclave in dry $N_2$ atmosphere, and then maintained at 170°C for 96 h, followed by slow cooling to room temperature. The solvent was removed under reduced pressure, and the residue extracted with a small amount of dichloromethane. The dichloromethane extract was chromatographed on a silica gel column. Elution with 4 : 1 dichloromethane–light petroleum gave a brown material as the major fraction. The yield after crystallization from *n*-pentane/dichloromethane was 82.8 mg (18.4%) with Rf = 0.38, FT-IR (KBr, $cm^{-1}$): 2919($\nu$(C–H), s), 2505($\nu$(B–H), vs), 1618($\delta(CH_3)$, w), 1434(s), 1228($\nu$(B–O), s), 1095($\delta$(C–O), s), 686(s), 519(s). $^1H$ NMR (400.15 MHz, $CDCl_3$): $\delta$ 0.88 ppm (1H, $CH_3$), 1.25, 1.28, 1.36, 1.37 ppm (15H, $CH_3$), 4.69, 4.70 ppm (2H, CH). $^{13}C$ NMR (100.63 MHz, $CDCl_3$): $\delta$ 24.7 ppm (10C, $CH_3$), 72.2, 76.7, 77.0, 77.2, 77.3 ppm (5C, CH). Anal. Calcd for $C_{15}H_{41}B_{11}Cl_2NiO_5$: C, 32.73; H, 7.45. Found: C, 32.58; H, 7.36.

### 2.2. *Crystal structure determination*

A suitable single crystal of the title cluster was glued to a fine glass fiber and then mounted on a Bruker Smart-1000 CCD diffractometer with Mo-K$\alpha$ radiation, $\lambda = 0.71073$ Å. The intensity data were collected at 298 ± 2 K. The structure was solved by direct methods and expanded using Fourier difference techniques with the SHELXTL-97 program package [17]. The non-hydrogen atoms were refined anisotropically by full-matrix least-squares calculations on $F^2$. The hydrogen atoms were added theoretically, riding on the concerned atoms and not refined. Detailed information about crystal data and structure determination is summarized in table 1. The selected bond lengths and angles are listed in table 2.

Table 1. X-ray diffraction data for the title cluster.

| | |
|---|---|
| Empirical formula | $C_{15}H_{43}B_{11}Cl_2NiO_5$ |
| Formula weight | 552.01 |
| Crystal size ($mm^3$) | $0.38 \times 0.31 \times 0.15$ |
| Temperature (K) | 298(2) |
| Wavelength (Å) | 0.71073 |
| Crystal system | Monoclinic |
| Space group | $P2(1)/n$ |
| $a$ (Å) | 12.355(13) |
| $b$ (Å) | 18.471(19) |
| $c$ (Å) | 12.956(14) |
| $\alpha$ (°) | 90 |
| $\beta$ (°) | 93.524(18) |
| $\gamma$ (°) | 90 |
| $V$ ($Å^{3)}$ | 2951(5) |
| $Z$ | 4 |
| $D$ ($Mg\,m^{-3}$) | 1.242 |
| Limiting indices | $-13 < h < 14$, $-18 < k < 21$, $-15 < l < 15$ |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 0.8816 and 0.7354 |
| $F(000)$ | 1160 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data/restraints/parameters | 5046/24/307 |
| $\theta$ range (°) | 2.35–25.03 |
| Reflection collected | 14825 |
| Independent reflection | 5046 |
| Goodness-of-fit on $F^2$ | 1.026 |
| $R[I > 2\sigma(I)]$ | $R_1 = 0.0746$, $wR_2 = 0.1829$ |
| $R$ (all date) | $R_1 = 0.1348$, $wR_2 = 0.2362$ |
| Largest diff. peak and hole ($\times 10^2\,e\,Å^{-3}$) | 0.740 and −0.609 |

## 3. Results and discussion

### 3.1. *Synthesis*

The *nido*-anion $B_{11}H_{14}^-$ is presumed to have a geometry based on a 12-vertex regular icosahedron with one vertex missing. There are three nonadjacent bridging hydrogen atoms on the five-boron open face. It is believed that the three non-*exo*-terminal hydrogen atom sites interchange rapidly with very low activation energy. So in the reaction of $B_{11}H_{14}^-$ with metallic compounds, the nonadjacent bridging hydrogen atoms should be deprotonized using alkali such as sodium hydroxide [18], methyllithium [5], *n*-butyllithium [7], triethylamine [8] and potassium hydroxide [10] to give the ollide anion $B_{11}H_{14}^-$. During our reaction which utilizing solvothermal techniques, the three nonadjacent bridging hydrogen atoms were removed. One reason is the high temperature and pressure removing the bridging hydrogen atoms, another reason is that $PPh_3$ acts as a strong base to accept the proton [10].

There are many of poly-substituent compounds in borane and carborane chemistry, such as perhalogenated clusters: $[closo\text{-}B_{10}Cl_{10}]^{2-}$ [19] and $[closo\text{-}B_{12}Cl_{12}]^{2-}$ [20]; fully methylated icosahedra: $[closo\text{-}B_{12}(CH_3)_{12}]^{2-}$ [21], $[closo\text{-}CB_{11}(CH_3)_{12}]^{2-}$ [22] and $[closo\text{-}C_2B_{10}(CH_3)_{12}]^{2-}$ [23]; perhydroxylated icosahedra: $[closo\text{-}B_{12}(OH)_{12}]^{2-}$ [24]. Similar highly substituted polyhedra were seldom seen in metallaborane chemistry and there were no reports about substitution on all of the B atoms connected to the

Table 2. Selected bond lengths (Å) and angles (°) of the title cluster.

| | | | | | |
|---|---|---|---|---|---|
| Ni(1)–Cl(1) | 1.922(4) | B(2)–B(6) | 1.892(11) | B(5)–B(11) | 1.747(10) |
| Ni(1)–Cl(2) | 1.946(4) | B(3)–O(2) | 1.408(8) | B(5)–B(6) | 1.880(10) |
| Ni(1)–B(6) | 2.105(7) | B(3)–B(9) | 1.767(10) | B(6)–O(5) | 1.354(9) |
| Ni(1)–B(3) | 2.107(7) | B(3)–B(8) | 1.770(10) | B(6)–B(11) | 1.757(11) |
| Ni(1)–B(4) | 2.109(7) | B(3)–B(4) | 1.866(10) | B(6)–B(7) | 1.758(11) |
| Ni(1)–B(2) | 2.138(7) | B(4)–O(3) | 1.389(8) | B(7)–B(8) | 1.752(13) |
| Ni(1)–B(5) | 2.156(7) | B(4)–B(10) | 1.774(10) | B(7)–B(12) | 1.759(14) |
| B(2)–O(1) | 1.407(9) | B(4)–B(9) | 1.782(10) | B(7)–B(11) | 1.809(12) |
| B(2)–B(8) | 1.720(12) | B(4)–B(5) | 1.807(10) | B(8)–B(12) | 1.765(12) |
| B(2)–B(7) | 1.754(11) | B(5)–O(4) | 1.406(8) | B(8)–B(9) | 1.813(12) |
| B(2)–B(3) | 1.823(11) | B(5)–B(10) | 1.727(11) | B(9)–B(10) | 1.776(12) |

| | | | |
|---|---|---|---|
| Cl(1)–Ni(1)–Cl(2) | 88.70(18) | B(8)–B(3)–Ni(1) | 114.3(5) |
| Cl(1)–Ni(1)–B(6) | 118.4(3) | B(2)–B(3)–Ni(1) | 65.4(3) |
| Cl(2)–Ni(1)–B(6) | 112.8(2) | B(4)–B(3)–Ni(1) | 63.8(3) |
| Cl(1)–Ni(1)–B(3) | 141.5(3) | O(3)–B(4)–Ni(1) | 107.8(4) |
| Cl(2)–Ni(1)–B(3) | 104.8(2) | B(10)–B(4)–Ni(1) | 114.9(5) |
| B(6)–Ni(1)–B(3) | 89.8(3) | B(9)–B(4)–Ni(1) | 114.2(4) |
| Cl(1)–Ni(1)–B(4) | 98.9(2) | B(5)–B(4)–Ni(1) | 66.3(3) |
| Cl(2)–Ni(1)–B(4) | 149.4(2) | B(3)–B(4)–Ni(1) | 63.7(3) |
| B(6)–Ni(1)–B(4) | 89.7(3) | O(4)–B(5)–Ni(1) | 107.3(4) |
| B(3)–Ni(1)–B(4) | 52.5(3) | B(10)–B(5)–Ni(1) | 114.8(4) |
| Cl(1)–Ni(1)–B(2) | 167.6(3) | B(11)–B(5)–Ni(1) | 113.2(4) |
| Cl(2)–Ni(1)–B(2) | 87.6(2) | B(4)–B(5)–Ni(1) | 63.6(3) |
| B(6)–Ni(1)–B(2) | 53.0(3) | B(6)–B(5)–Ni(1) | 62.4(3) |
| B(3)–Ni(1)–B(2) | 50.9(3) | O(5)–B(6)–Ni(1) | 100.0(4) |
| B(4)–Ni(1)–B(2) | 90.2(3) | B(11)–B(6)–Ni(1) | 115.2(5) |
| Cl(1)–Ni(1)–B(5) | 88.9(2) | B(7)–B(6)–Ni(1) | 114.4(5) |
| Cl(2)–Ni(1)–B(5) | 160.3(2) | B(5)–B(6)–Ni(1) | 65.2(3) |
| B(6)–Ni(1)–B(5) | 52.3(3) | B(2)–B(6)–Ni(1) | 64.4(3) |
| B(3)–Ni(1)–B(5) | 89.0(3) | O(1)–B(2)–B(8) | 126.6(6) |
| B(4)–Ni(1)–B(5) | 50.1(3) | O(1)–B(2)–B(7) | 127.3(6) |
| B(2)–Ni(1)–B(5) | 90.6(3) | O(2)–B(3)–B(9) | 124.9(6) |
| O(1)–B(2)–Ni(1) | 107.7(4) | O(2)–B(3)–B(8) | 126.4(5) |
| B(8)–B(2)–Ni(1) | 115.1(5) | O(3)–B(4)–B(10) | 128.5(6) |
| B(7)–B(2)–Ni(1) | 113.1(5) | O(3)–B(4)–B(9) | 124.7(6) |
| B(3)–B(2)–Ni(1) | 63.7(3) | O(4)–B(5)–B(11) | 127.1(5) |
| B(6)–B(2)–Ni(1) | 62.6(3) | O(4)–B(5)–B(10) | 126.9(6) |
| O(2)–B(3)–Ni(1) | 108.2(4) | O(5)–B(6)–B(11) | 126.4(6) |
| B(9)–B(3)–Ni(1) | 115.0(4) | O(5)–B(6)–B(7) | 136.2(6) |

metal. In the title cluster, the O*i*Pr from the solvent, substituted all five H atoms on boron cage atoms adjacent to Ni.

### 3.2. *Description of the crystal structure*

The structure of the dianion $[\mathrm{Cl_2NiB_{11}H_6\text{-}2,3,4,5,6\text{-}(O\mathit{i}Pr)_5}]^{2-}$, as determined by X-ray diffraction, is shown in figure 1. It consists of one Ni atom and eleven B atoms to form a *closo* 12-vertex $\{\mathrm{NiB_{11}}\}$ icosahedral skeleton in which Ni atom connects to five B atoms on the same pentagonal plane. The $\mathrm{B_{11}H_{14}^{-}}$ ligand has the ability to coordinate $\eta^5$ to a transition metal, apparently in the same manner in which the well-known and iso-electronic dicarbollide ligand $\mathrm{C_2B_9H_{11}^{2-}}$ does. The skeleton of the title cluster was similar to the reported nickeladodecaborane $[(\mathrm{C_5H_5})\mathrm{Ni}(\mathrm{B_{11}H_{11}})]^{-}$ [6], but there were two Cl atoms bonded to Ni in the title cluster but a cyclopentadienyl group connected

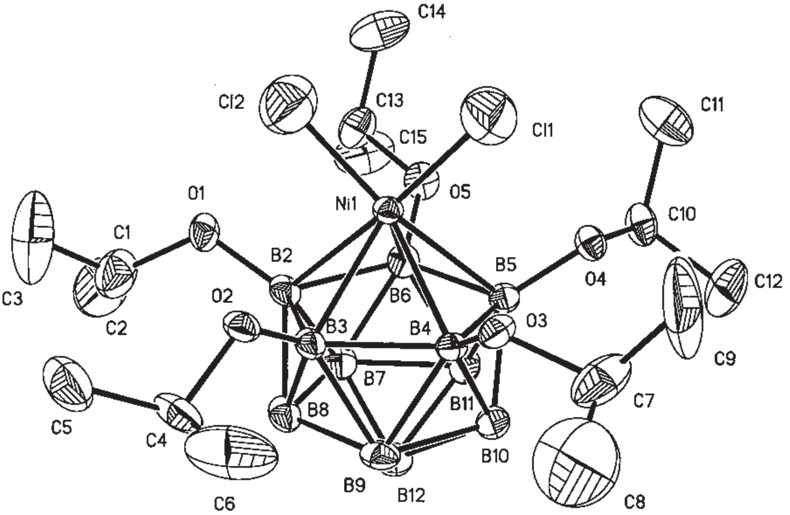

Figure 1. ORTEP drawing of the title cluster with 30% probability thermal ellipsoids. Hydrogen atoms were omitted for clarity.

to Ni in $[(C_5H_5)Ni(B_{11}H_{11})]^-$ and all five H atoms on boron cage atoms adjacent to the Ni were substituted by O*i*Pr groups in the title cluster.

The average bond length of Ni–B is 2.123 Å, which was consistent with the corresponding Ni–B distance in [1,1-$(PhMe_2P)_2$-2,4-$Cl_2$-*closo*-1-$NiB_9H_7$] [average 2.093(3) Å] [25]. The average bond length of B–O is 1.393(8) Å, which is similar to that of reported B–OR groups in $[Pt(SePh)(PEt_3)\{\eta^5\text{-8-O}(CH_2)_4Cl\text{-7-}CB_{10}H_{10}\}]$ [1.369(7) Å] [26], shorter than that in $(PPh_3)_2Pt[SB_8H_9(OEt)]$ [1.476(24) Å] [27]. The average bond length of B–B on the pentagonal face, which was substituted by O*i*Pr groups, is 1.854(10) Å, longer than the average bond length [1.766(10) Å] of other B–B bonds in the title cluster. This may be due to the effect of the five O*i*Pr groups. The bond lengths of Ni(1)–Cl(1) and Ni(1)–Cl(2) [1.922(4) and 1.946(4) Å respectively] were shorter than those in $[Ni_4(Pym)_4Cl_4(CH_3OH)_4]$ [2.369(3) Å] [28] and *trans*-$[NiCl(C\equiv CCl)(PPh_3)_2]$ [2.197(4) Å] [29].

## Supplementary data

Crystallographic data for the structural analysis (excluding structure factors) have been deposited with the Cambridge Crystallographic Data Center as supplementary publication CCDC No. 253617. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: (+44)1223 336033; E-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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