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1-Oxa-*nido*-dodecaborate $[OB_{11}H_{12}]^-$ from the Controlled Oxidation of the *closo*-Borates $[B_{11}H_{11}]^{2-}$ and $[B_{12}H_{12}]^{2-}$

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Dedicated to Professor Hansgeorg Schnöckel on the Occasion of his 65th Birthday

Abstract. The *closo*-dodecaborate $[B_{12}H_{12}]^{2-}$ is degraded at room temperature by oxygen in an acidic aqueous solution in the course of several weeks to give $B(OH)_3$. The degradation is induced by Ag^{2+} ions, generated from Ag^+ by the action of $H_2S_2O_8$. Oxa-*nido*-dodecaborate(1−) is an intermediate anion, that can be separated from the reaction mixture as $[NBzlEt_3][OB_{11}H_{12}]$ after five days in a yield of 18 %. The action of $FeCl_3$ on the *closo*-undecaborate $[B_{11}H_{11}]^{2-}$ in an aqueous solution gives either $[B_{22}H_{22}]^{2-}$ (by fusion) or *nido*-$B_{11}H_{13}(OH)^-$ (by protonation and hydration), depending on the concentration of $FeCl_3$. In acetonitrile, however, $[B_{11}H_{11}]^{2-}$ is transformed into $[OB_{11}H_{12}]^-$ by Fe^{3+} and oxygen. The radical anions $[B_{12}H_{12}]^{\bullet -}$ and $[B_{11}H_{11}]^{\bullet -}$ are assumed to be the primary products of the oxidation with the one-electron oxidants Ag^{2+} and Fe^{3+}, respectively. These radical anions are subsequently transformed into $[OB_{11}H_{12}]^-$ by oxygen. The crystal structure analysis shows that the structure of $[OB_{11}H_{12}]^-$ is derived from the hypothetical *closo*-oxaborane $OB_{12}H_{12}$ by removal of the B3 vertex, leaving a non-planar pentagonal aperture with a three-coordinate O vertex, as predicted by NMR spectra and theory.

Keywords: Undecahydroundecaborate(2−); Dodecahydrododecaborate(2−); Dodecahydrooxadodecaborate(1−); Oxidative Cluster Degradation; Crystal Structure

Introduction

The chemistry of the *closo*-anions $[B_{11}H_{11}]^{2-}$ and $[B_{12}H_{12}]^{2-}$ has been summarized recently [1, 2]. The replacement of one or more H atoms by anionic ligands X^- ($\rightarrow[B_{11}H_{11-n}X_n]^{2-}$, $[B_{12}H_{12-n}X_n]^{2-}$) or by neutral ligands L ($\rightarrow[B_{11}H_{10}L]^-$, $[B_{12}H_{11}L]^-$) is a common reaction for both of the *closo*-anions. There is a distinct difference, however, in the skeletal reactivity. The $[B_{12}H_{12}]^{2-}$ skeleton is highly stable, and a controlled degradation at ambient temperature would not be expected. The $[B_{11}H_{11}]^{2-}$ skeleton, on the other hand, undergoes a rapid fluctuation in solution at room temperature, making the eleven B atoms equivalent with respect to the NMR time scale; in the ground state, the free $[B_{11}H_{11}]^{2-}$ anion was computed to have C_{2v} symmetry with five sets of equivalent B atoms. The *closo*-11-vertex skeleton can be easily extended to the *closo*-12-vertex skeleton by incorporating two-electron fragments like BH (from $[BH_3{\cdot}NEt_3]$; $\rightarrow[B_{12}H_{12}]^{2-}$) or $NiCp^+$ (from $[\{CpNi(CO)\}_2]$; $\rightarrow[B_{11}H_{11}(NiCp)]^-$). It can also be easily transformed into the *nido*-11-vertex skeleton by adding H^+ and HX ($\rightarrow[B_{11}H_{13}X]^-$) or H^+ and L ($\rightarrow[B_{11}H_{12}L]^-$). With one-electron acceptors like Fe^{3+} or Ce^{4+}, $[B_{11}H_{11}]^{2-}$ undergoes a fusion to give the $[B_{22}H_{22}]^{2-}$ anion; the radical anion $[B_{11}H_{11}]^{\bullet -}$ is presumably an intermediate, as was shown by an electrochemical study. The electrochemical oxidation of $[B_{12}H_{12}]^{2-}$ gives $[B_{24}H_{23}]^{3-}$ at 1.5 V [3] without altering the two icosahedral B_{12} skeletons, which are connected by a hydrogen bridge in $[B_{24}H_{23}]^{3-}$. The $[B_{12}H_{12}]^{2-}$ anion is not attacked by Fe^{3+} or Ce^{4+}, and a radical anion $[B_{12}H_{12}]^{\bullet -}$ could not be detected by electrochemical methods. Derivatives of $[B_{12}H_{12}]^{2-}$ like $[B_{12}Me_{12}]^{2-}$ or $[B_{12}(OR)_{12}]^{2-}$, however, can be oxidized to give the corresponding radical anions [4].

In order to oxidize $[B_{12}H_{12}]^{2-}$, we applied the strong one-electron acceptor Ag^{2+} [$\varepsilon_0(Ag^{2+}/Ag^+) = 1.98$ V]. This cation is formed in an aqueous solution from Ag^+ and peroxodisulfuric acid; it slowly decomposes water into oxygen. We report here on the results of this oxidation and also of a similar oxidation of $[B_{11}H_{11}]^{2-}$.

Oxidation of $[B_{12}H_{12}]^{2-}$

An aqueous solution of $K_2B_{12}H_{12}$, $K_2S_2O_8$, $AgNO_3$, and $HClO_4$ in the ratio 2:6:1:3 is stirred at the open air for five days. According to the ^{11}B NMR data, three boron containing compounds are found in a comparable amount: unchanged $[B_{12}H_{12}]^{2-}$, the known oxa-*nido*-dodecaborate

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$[OB_{11}H_{12}]^-$ [5], and boric acid $B(OH)_3$. Silver oxides would precipitate, if perchloric acid was not present. More $B(OH)_3$ and less $[OB_{11}H_{12}]^-$ are found, when the reaction is conducted under pure oxygen instead of air. No decomposition of $[B_{12}H_{12}]^{2-}$, on the other hand, is observed, when the reaction is conducted under pure nitrogen; oxygen seems to be a necessary reactant for the decomposition of $[B_{12}H_{12}]^{2-}$. A prolongation of the reaction time from five days to five weeks resulted in the formation of $B(OH)_3$ as the only product. In a separate experiment, the oxadodecaborate $[OB_{11}H_{12}]^-$ was shown to be slowly decomposed by O_2 to give $B(OH)_3$ in an acidic aqueous solution. No decomposition of $[B_{12}H_{12}]^{2-}$ is observed in the above described reaction, when no silver salt was added. All of the mentioned components, including oxygen, are necessary in order to decompose $[B_{12}H_{12}]^{2-}$.

The oxadodecaborate could be separated from the reaction mixture after neutralization and precipitation as the ammonium salt $[NBzlEt_3][OB_{11}H_{12}]$. After dissolving this salt in acetonitrile, removal of solid impurities, replacing the solvent MeCN by CH_2Cl_2, purification on silica gel, removal of CH_2Cl_2 and recrystallization from CH_2Cl_2/EtOH, the yield of $[NBzlEt_3][OB_{11}H_{12}]$ was 18 %. Single crystals for X-ray analysis could be obtained by replacing the cation $[NBzlEt_3]^+$ by $[CpFe\{C_5H_4(CH_2NMe_3)\}]^+$.

The above mentioned ratio of the components is optimal. Major changes of the amount of $K_2S_2O_8$ or $AgNO_3$ reduce the yield of $[OB_{11}H_{12}]^-$.

The observed reaction can be interpreted by assuming the formation of Ag^{2+} as the first reaction step [Eq. (1)], followed by the formation of the $[B_{12}H_{12}]^{\bullet-}$ radical anion [Eq. (2)].

$$2\ Ag^+ + H_2S_2O_8 \rightarrow 2\ Ag^{2+} + 2\ HSO_4^- \qquad (1)$$

$$[B_{12}H_{12}]^{2-} + Ag^{2+} \rightleftarrows [B_{12}H_{12}]^{\bullet-} + Ag^+ \qquad (2)$$

Reaction (2) is assumed to proceed as an equilibrium, in which the $[B_{12}H_{12}]^{2-}$ anion predominates. Otherwise, we should have found some radical anion as a product in the absence of oxygen. Obviously, the radical anion is rapidly attacked by oxygen, and the products $[OB_{11}H_{12}]^-$ and $B(OH)_3$ are formed during a sequence of unknown reaction steps [Eq. (3)].

$$4\ [B_{12}H_{12}]^{\bullet-} + 5\ O_2 + 6\ H_2O \rightarrow 4\ [OB_{11}H_{12}]^- + 4\ B(OH)_3 \qquad (3)$$

The oxadodecaborate is slowly decomposed by oxygen [Eq. (4)]. The peroxodisulfuric acid cannot play a major role as the oxidant in the formation of $B(OH)_3$, because 22 mol of $H_2S_2O_8$ would be needed for the transformation of 1 mol of $[OB_{11}H_{12}]^-$ into 11 mol of $B(OH)_3$, much more than available.

$$[OB_{11}H_{12}]^- + 11\ O_2 + 10\ H_2O + H^+ \rightarrow 11\ B(OH)_3 \qquad (4)$$

The Ag^{2+} cations act as a catalyst. Most of them attack $[B_{12}H_{12}]^{2-}$, a few of them may also transform water into O_2, but all of the oxidation equivalents are finally provided by $H_2S_2O_8$. An excess of $K_2S_2O_8$, with respect to $[B_{12}H_{12}]^{2-}$, is necessary. We suppose the formation of O_2 from water to be less important, because no decomposition of $[B_{12}H_{12}]^{2-}$ is observed on working under an atmosphere of nitrogen. The major part of the needed oxygen seems to be transported from the gas phase into the stirred solution.

Oxidation of $[B_{11}H_{11}]^{2-}$

When Fe^{3+} ions attack the *closo*-$[B_{11}H_{11}]^{2-}$ anion in an aqueous solution, the $[B_{22}H_{22}]^{2-}$ anion is formed [6] [Eq. (5)].

$$2\ [B_{11}H_{11}]^{2-} + 2\ Fe^{3+} \rightarrow [B_{22}H_{22}]^{2-} + 2\ Fe^{2+} \qquad (5)$$

A rapid addition of the aqueous iron(III) solution to the aqueous solution of $K_2B_{12}H_{12}$ is necessary in order to observe the fusion reaction (5). Meanwhile, we realized that a dropwise slow addition strongly reduces the yield of $[B_{22}H_{22}]^{2-}$ and makes the hydroxo-*nido*-undecaborate $[B_{11}H_{13}(OH)]^-$ the main product. We had found this product earlier from the reaction of $[B_{11}H_{11}]^{2-}$ with hydrochloric acid: protonation and subsequent addition of H_2O [7] [Eq. (6)]. In the case of an $FeCl_3$ solution, the hydrated Fe^{3+} ions act as Brønsted acids. Obviously, a high stationary concentration of $[B_{11}H_{11}]^{\bullet-}$ radical anion is formed upon the rapid addition of the iron(III) solution and makes the fusion reaction (5) possible.

$$[B_{11}H_{11}]^{2-} + H^+ + H_2O \rightarrow [B_{11}H_{13}(OH)]^- \qquad (6)$$

We now changed the solvent. Upon addition of a solution of $FeCl_3{\cdot}6H_2O$ in acetonitrile to a solution of $[NBzlEt_3]_2[B_{11}H_{11}]$ in acetonitrile and stirring at the open air, the crystalline material $[NBzlEt_3][OB_{11}H_{12}]$ could be isolated in a 30 % yield. The small amount of water from the starting iron component is necessary for this reaction as well as the availability of oxygen from the gas phase. We suppose that the radical anion $[B_{11}H_{11}]^{\bullet-}$ is formed in a first step [Eq. (7)], followed by the attack of oxygen to give $[OB_{11}H_{12}]^-$, certainly in a sequence of unknown steps [Eq. (8)].

$$[B_{11}H_{11}]^{2-} + Fe^{3+} \rightarrow [B_{11}H_{11}]^{\bullet-} + Fe^{2+} \qquad (7)$$

$$4\ [B_{11}H_{11}]^{\bullet-} + O_2 + 2\ H_2O \rightarrow 4\ [OB_{11}H_{12}]^- \qquad (8)$$

It remains an unanswered question, why the medium, water or acetonitrile, so distinctly influences the product formation. It was unexpected that the radical anions $[B_{11}H_{11}]^{\bullet-}$ and $[B_{12}H_{12}]^{\bullet-}$ give the same product, $[OB_{11}H_{12}]^-$, upon oxidation with oxygen. Obviously, the formation of the $[OB_{11}H_{12}]^-$ anion is a thermodynamically favoured product during the oxidative degradation on the way to the final product $B(OH)_3$.

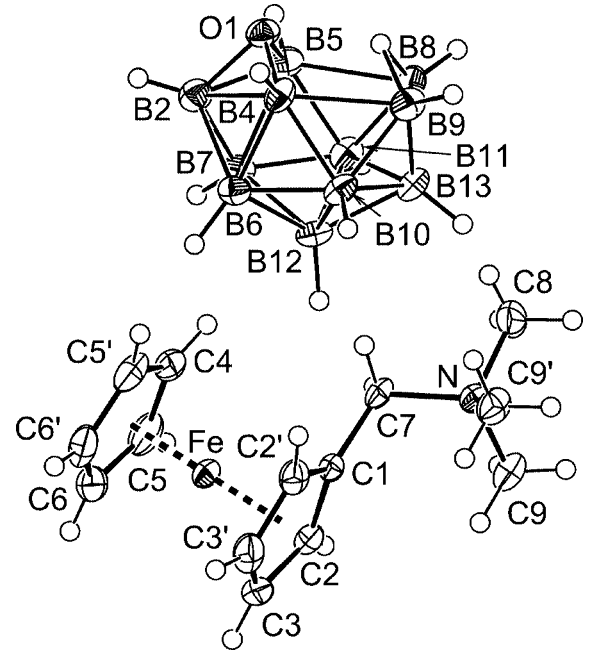

Fig. 1 Structure of $[CpFe\{C_5H_4(CH_2NMe_3)\}][OB_{11}H_{12}]$ (PLATON [9], 50 % displacement ellipsoids); primed atoms are generated by the symmetry operation x, $-y+{}^1/_2$, z.

Structure of $[CpFe\{C_5H_4(CH_2NMe_3)\}][OB_{11}H_{12}]$

This compound crystallizes in the monoclinic space group $P2_1/m$. The structures of cation and anion are depicted in Fig. 1. A mirror plane goes through the atoms Fe, C1, C4, C7, C8, and N of the cation. The 28 different lengths of the cluster edges are collected in Table 1. The atoms B4, B5, B9, B8, and O form a pentagonal aperture, the four B vertices of which are planarly arranged, with the O vertex above this plane. There are six long edges in the range 182.9−207.4 pm; four of them form the pentagonal plane B8−B5−B2−B4−B9. Four short edges are found in the range 172.7−173.5 pm [Fig. 2(a)]. The observed bond lengths of the anion in the crystal are in good accord with those of the free anion, computed at the B3LYP/6-31G* level [8] (Table 1). In the localized bond picture, the OB_{11} skeleton can be built up from five (2c2e) and nine (3c2e) bonds in accord with the octet rule [Fig. 2(b); a total of 60 atomic orbitals and 52 valence electrons of $[OB_{11}H_{12}]^-$ correspond in a unique way to nine (3c2e) bonds (8 BBB + 1 BHB), to sixteen (2c2e) bonds (11 BH + 3 BO + 2 BB), and one lone pair at oxygen]. On attributing three of the (2c2e) bonds to oxygen, this strongly electronegative atom will be embedded in a "classical" oxonium structure.

The bond lengths of the terminal BH bonds range from 97(7) to 110(5) pm with an average value over 11 bonds of 105.4 pm. The BHB bridge is not symmetrical in the crystal with BH distances of 127(7) and 132(7) pm, respectively.

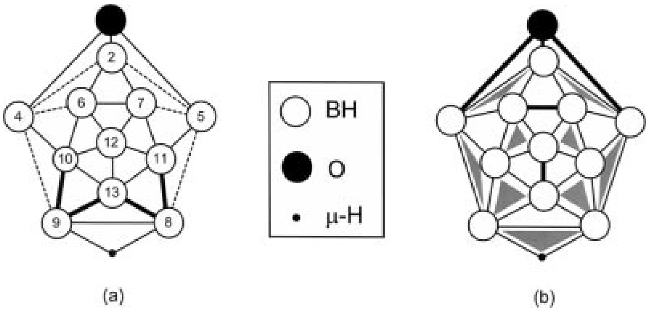

Fig. 2 Planarized structure of the $[OB_{11}H_{12}]^-$ anion: (a) 6 long edges (182.9−207.4 pm; dashed lines), 4 short edges (172.1−173.5 pm; bold lines), and 18 edges of medium lengths (175.2−179.6 pm); (b) one out of eight resonance formulae, including five (2c2e) bonds (bold lines) and nine (3c2e) bonds (greyish triangles).

Table 1 O−B, B−B, and B−μH bond lengths (pm) in crystalline $[CpFe\{C_5H_4(CH_2NMe_3)\}][OB_{11}H_{12}]$ (in brackets: computed values [8] of the free anion in C_s symmetry).

O−B2	147.9(9)	[149.5]	B6−B7	178.3(7)	[180.0]
O−B4	152.6(8)	[152.3]	B6−B12	176.4(9)	[176.2]
O−B5	150.5(12)	″	B7−B12	174.4(10)	″
B2−B4	191.3(10)	[192.9]	B8−B9	177.9(10)	[177.7]
B2−B5	191.5(16)	″	B8−B11	173.5(11)	[173.5]
B2−B6	175.2(8)	[176.8]	B9−B10	173.4(11)	″
B2−B7	175.9(9)	″	B8−B13	173.5(11)	[174.3]
B4−B6	182.9(9)	[184.8]	B9−B13	172.7(9)	″
B5−B7	185.9(12)	″	B10−B12	176.9(10)	[180.2]
B4−B9	207.4(11)	[210.6]	B11−B12	178.0(11)	″
B5−B8	207.3(15)	″	B10−B13	176.9(10)	[180.6]
B4−B10	179.6(10)	[178.3]	B11−B13	179.2(10)	″
B5−B11	179.2(13)	″	B12−B13	177.0(11)	[179.2]
B6−B10	178.0(9)	[178.5]	B8−H89	132(7)	[131.4]
B7−B11	177.9(10)	″	B9−H89	127(7)	″

The $[OB_{11}H_{12}]^-$ anion is isoelectronic with the azadodecaborate anion $[(MeN)B_{11}H_{12}]^-$ and derivatives thereof [10]. These *nido*-anions are derived from the hypothetical *closo*-13-vertex cluster $YB_{12}H_{12}$ (Y = O, RN) by removing the connectivity-5 vertex B3, formally as B^+. The connectivity of B4 and B5 is thereby reduced from the unpleasant value of six to the more convenient value of five; the thereby formed connectivity-4 vertices B8 and B9 become bridged by hydrogen, and the electronegative vertices Y can get rid of their electron-deficiency.

Experimental Section

Oxa-nido-dodecaborate(1−) $[OB_{11}H_{12}]^-$ *from* $[B_{12}H_{12}]^{2-}$

A suspension of $K_2B_{12}H_{12}$ (0.454 mmol), $K_2S_2O_8$ (1.36 mmol), $AgNO_3$ (0.227 mmol), $HClO_4$ (0.7 mmol) in water (14 ml) was stirred for 5 d at the open air. After neutralization with soda, a solution of benzyltriethylammonium chloride (1 mmol) in water (5 ml) was added to the reaction mixture. The precipitate of $[NBzlEt_3][OB_{11}H_{12}]$ was filtrated and dissolved in CH_2Cl_2 (10 ml). The solution was passed through a column for purification at silica gel, applying another portion of CH_2Cl_2 (20 ml). After removal of the solvent, $[NBzlEt_3][OB_{11}H_{12}]$ could be recrystallized from CH_2Cl_2/EtOH (0.082 mmol, 18 %). The product was pure, according to the ^1H and ^{11}B NMR spectra [5]. − After addition of MI

($\mathrm{M} = [\eta^5\text{-}(C_5H_5)Fe\{\eta^5\text{-}C_5H_4(CH_2NMe_3)\}]$) to a warm solution of $[NBzlEt_3][OB_{11}H_{22}]$ in CH_2Cl_2, a precipitate of $MOB_{11}H_{12}$ was obtained upon addition of EtOH, which was recrystallized from CH_2Cl_2/EtOH. Upon slow evaporation of the solvent mixture, single crystals of $MOB_{11}H_{12}$ could be grown.

Hydroxo-nido-undecaborate $[B_{11}H_{13}(OH)]^-$ *from* $[B_{11}H_{11}]^{2-}$

An aqueous solution (10 ml) of $FeCl_3 \cdot 6H_2O$ (0.72 mmol) was added dropwise to an aqueous solution (10 ml) of $K_2B_{11}H_{11}$ (0.36 mmol) at the open air under ice-cooling. After stirring (10 min), a saturated solution of $[NBzlEt_3]Cl$ in water was added to the reaction mixture. A precipitate was formed, filtrated, dried in vacuo, and dissolved in CH_2Cl_2 (10 ml). The solution was purified at silica gel, applying an additional amount of CH_2Cl_2 (40 ml). After reducing the volume of the solution in vacuo and adding ethanol, a colorless solid precipitated, which was recrystallized from CH_2Cl_2/hexane and identified by NMR [7] as $[NBzlEt_3][B_{11}H_{13}(OH)]$ (46 %).

Oxa-nido-dodecaborate(1−) $[OB_{11}H_{12}]^-$ *from* $[B_{11}H_{11}]^{2-}$

A solution of $FeCl_3 \cdot 6H_2O$ (0.33 mmol) in MeCN (10 ml) was added dropwise to a solution of $[NBzlEt_3]_2[B_{11}H_{11}]$ (0.29 mmol) in MeCN (10 ml) at the open air under ice-cooling. After stirring (20 min), MeCN was evaporated in vacuo and CH_2Cl_2 (40 ml) was added to the solid reaction product. A suspension was formed after stirring (40 min), which was transported through silica gel, together with a further amount of CH_2Cl_2 (30 ml). The volume of the solution was reduced in vacuo. Addition of ethanol to the solution resulted in the formation of a colorless product, which was recrystallized from MeCN/EtOH, dried in vacuo and identified by NMR as $[NBzlEt_3][OB_{11}H_{12}]$ (31 %). This product was not formed, when the reaction was performed under an N_2 atmosphere.

Crystal structure analysis

$C_{14}H_{32}B_{11}FeNO$, M = 405.18 g mol^{-1}. Bruker SMART Apex CCD area detector on a D8 goniometer; MoK_α radiation (71.073 pm); graphite monochromator; 293(2) K; range of Θ for data collection: 1.34−26.10°; ω-scan method [11], processed with the SAINT program [12]; empirical absorption correction with the SADABS program for area detection [13]; μ = 7.17 cm^{-1}. Crystal size: 0.04·0.12·0.33 mm^3; space group $P2_1/m$ (No. 11); a = 721.18(7), b = 969.29(9), c = 1526.95(14) pm, β = 97.000(5)°; V = 1.05943(17) nm^3; Z = 2; d_{calc} = 1.270 g cm^{-3}. The structure was solved by direct methods using SHELXTL for refinement [14]; non-hydrogen atoms were refined with anisotropic replacement parameters; carbon-bound hydrogen atoms were placed in idealized positions (C−H: 98 pm) and included as riding with $U_{iso}(H) = 1.3\ U_{eq}$(non-H); boron-bound hydrogen atoms were located from difference Fourier maps and refined isotropically. Collected reflections: 11241; independent reflections: 2217; no restraints; refined parameters: 280. $R1 = 0.0420$ [$I > 2\sigma(I)$], 0.0649 (all data), $wR2$ = 0.0954 (all data); GOF = 1.005; maximal residue electron density: 0.694/−0.356 e Å^{-3}. The crystallographic data have been deposited at the Cambridge Crystallographic Data Centre as supplementary publication No. CCDC 289223. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1 EZ, UK (e-mail: **deposit@ccdc.com.ac.de**).

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