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Crystal structure of the uranium oxo borohydride complex $U_2(\mu\text{-}O)(BH_4)_6(dme)_2$ (dme = 1,2-dimethoxyethane) and reformulation of $U_2(\mu\text{-}H)_2(BH_4)_6(dme)_2$

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ABSTRACT

The reaction of uranium tetrachloride, UCl_4, with sodium *N*,*N*-dimethylaminodiboranate, $Na(H_3BNMe_2BH_3)$, in refluxing 1,2-dimethoxyethane (dme) yields small amounts of a new complex, which we formulate as (μ-oxo)hexakis(tetrahydroborato)bis(1,2-dimethoxyethane)diuranium(IV), toluene solvate, $U_2(\mu\text{-}O)(BH_4)_6(dme)_2{\cdot}C_7H_8$, **1**. Most likely, the formation of BH_4^- groups from $H_3BNMe_2BH_3^-$ occurs with the elimination of $[Me_2NBH_2]_2$, and the formation of the oxo group involves adventitious hydrolysis. Each uranium center in **1** adopts a *fac* octahedral geometry (counting the BH_4^- groups as occupying one coordination site); the bridging oxygen atom and the two coordinated oxygen atoms of the dme ligand occupy positions *trans* to the three BH_4^- groups. The hydrogen atom positions were located in the electron density difference maps and reveal that all three BH_4^- groups are bound in a κ^3H fashion. The U···B distances to the two BH_4^- groups that are *cis* to the bridging oxygen atom are 2.574(6) and 2.584(6) Å, whereas the U···B distance of 2.635(7) Å to the BH_4^- group that is *trans* to the bridging oxygen is distinctly longer. Thus, the bridging oxygen atom exerts a noticeable *trans* influence. The crystallographic and ^1H NMR data strongly suggest that the previously reported uranium hydride complex $U_2(\mu\text{-}H)_2(BH_4)_6(dme)_2$ should be reformulated as this oxo complex $U_2(\mu\text{-}O)(BH_4)_6(dme)_2$.

1. Introduction

The isolation of uranium(IV) hydride $U_2(\mu\text{-}H)_2(BH_4)_6(dme)_2$, where dme = 1,2-dimethoxyethane, in 1987 provided crystallographic evidence that uranium(IV) hydrides are intermediates in the reduction of U^{IV} borohydrides to U^{III} [1]. This complex is also notable as one of the few crystallographically characterized actinide hydrides [2–5]. We now present evidence that this $U_2(\mu\text{-}H)_2(BH_4)_6(dme)_2$ complex is actually the bridged oxo species $U_2(\mu\text{-}O)(BH_4)_6(dme)_2$.

2. Experimental

All operations were carried out in vacuum or under argon using standard Schlenk techniques. The solvents 1,2-dimethoxyethane and pentane were distilled under nitrogen from sodium/benzophenone and purged with argon immediately before use. Toluene was dried similarly over molten sodium. UCl_4 [6] and $Na(H_3BNMe_2BH_3)$ [7] were prepared by following the literature routes. The ^1H NMR data were obtained on a Varian Unity Inova 600 instrument at 600 MHz. Chemical shifts are reported in δ units (positive shifts to high frequency) relative to TMS.

2.1. (*μ*-*Oxo*)*hexakis*(*tetrahydroborato*)*bis*(*1,2-dimethoxyethane*)-*diuranium*(*IV*), **1**

To a suspension of UCl_4 (0.27 g, 0.71 mmol) in 1,2-dimethoxyethane (15 mL) was added a solution of sodium *N*,*N*-dimethylaminodiboranate (0.27 g, 2.8 mmol) in 1,2-dimethoxyethane (15 mL). The reaction mixture was heated to reflux for 12 h, over which time the solution color changed from green to light brown, and a dark precipitate formed. The solvent was removed under vacuum to afford a sticky, dark brown solid. The residue was extracted with toluene (20 mL), and the filtered extract was concentrated to ca. 10 mL and layered with pentane (10 mL). The mixture was kept at room temperature for several hours, and small green prisms formed. The crystals were collected, and the mother liquor was decanted and cooled to −20 °C overnight to yield a second crop of green prisms. Yield: 20 mg (7%). ^1H NMR (C_7D_8, −60 °C): δ −115 (s, fwhm = 290 Hz, BH_4, 4H), −62.0 (s, fwhm = 110 Hz, OMe, 6H), −52.9 (s, fwhm = 110 Hz, OCH_2,

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2H), −32.4 (s, OCH_2, fwhm = 80 Hz, 2H), 727 (br s, fwhm = 2700 Hz, BH_4).

2.2. Crystallographic studies [8]

Single crystals of $U_2(\mu\text{-}O)(BH_4)_6(dme)_2{\cdot}C_7H_8$, grown from a 1:1 mixture of toluene and pentane, were mounted on glass fibers with Paratone–N oil (Exxon) and immediately cooled to −75 °C in a cold nitrogen gas stream on the diffractometer. Standard peak search and indexing procedures gave rough cell dimensions, and least squares refinement using 975 reflections yielded the cell dimensions given in Table 1.

Data were collected with an area detector by using the measurement parameters listed in Table 1. The triclinic lattice and the average values of the normalized structure factors suggested the space group $P\bar{1}$, which was confirmed by the success of the subsequent refinement. The measured intensities were reduced to structure factor amplitudes and their estimated standard deviations (esd's) by correction for background, scan speed, and Lorentz and polarization effects. No corrections for crystal decay were necessary, but a face-indexed absorption correction was applied, the minimum and maximum transmission factors being 0.251 and 0.610. Systematically absent reflections were deleted and symmetry equivalent reflections were averaged to yield a set of unique data. The reflections 0 0 1 and $0\,\bar{1}\,1$ were obscured by the beam stop and were deleted; the remaining 5603 unique data were used in the least squares refinement.

The structure was solved using direct methods (SHELXTL). Correct positions for the uranium atoms were deduced from an E-map. Subsequent least-squares refinement and difference Fourier calculations revealed the positions of the remaining non-hydrogen atoms. The toluene molecule that co-crystallized with the compound was disordered over two positions. The quantity minimized by the least-squares program was $\sum w(F_o^2 - F_c^2)^2$, where $w = \{[\sigma(F_o)]^2 + (0.0106P)^2\}^{-1}$ and $P = (F_o^2 + 2F_c^2)/3$. The analytical approximations to the scattering factors were used, and all structure factors were corrected for both real and imaginary components of anomalous dispersion. In the final cycle of least squares, independent anisotropic displacement factors were refined for the non-hydrogen atoms. The C–Me distances in the disordered toluene molecule were constrained to be equal within an esd of 0.01 Å, and the aromatic cores were constrained to hexagonal geometries. The boranyl hydrogen atoms were located in the difference maps, and their positions were refined with independent isotropic displacement parameters. The chemically equivalent B–H and H···H distances within the BH_4^- groups were constrained to be equal within an esd of 0.01 Å. Hydrogen atoms on methyl, methylene, and aromatic carbons were placed in idealized positions with C–H = 0.98, 0.99, and 0.95 Å, respectively; the methyl groups were allowed to rotate about the C–C or C–O axis to find the best least-squares positions. The displacement parameters for methylene and aromatic hydrogens were set equal to 1.2 times U_{eq} for the attached carbon; those for methyl hydrogens were set to 1.5 times U_{eq} for the attached carbon. No correction for isotropic extinction was necessary. Successful convergence was indicated by the maximum shift/error of 0.000 for the last cycle. Final refinement parameters are given in Table 1. The largest peak in the final Fourier difference map (1.63 e $Å^{-3}$) was located 1.07 Å from U2. A final analysis of variance between observed and calculated structure factors showed no apparent errors.

Table 1
Crystallographic data for $U_2(\mu\text{-}O)(BH_4)_6(dme)_2{\cdot}C_7H_8$, **1**.

Formula	$C_{15}H_{52}B_6O_5U_2$
Formula weight (g mol^{-1})	853.49
λ (Å)	0.71073
Crystal system	triclinic
Space group	$P\bar{1}$
Unit cell dimensions	
a (Å)	9.595(3)
b (Å)	11.500(4)
c (Å)	14.135(4)
α (°)	85.383(4)
β (°)	83.555(4)
γ (°)	85.021(4)
V ($Å^3$)	1540.0(8)
Z	2
ρ_{calc} (g cm^{-3})	1.841
μ (mm^{-1})	10.520
Absorption correction	face-indexed
Maximum, minimum transmission factors	0.610, 0.251
Data/restraints/parameters	5603/475/395
Goodness-of-fit (GOF) on F^2	0.845
R_1 [$I > 2\sigma(I)$][a]	0.0236
wR_2 (all data)[b]	0.0505
Largest difference peak and hole (e $Å^{-3}$)	1.632, −1.219

[a] $R_1 = \sum||F_o| - |F_c||/\sum|F_o|$ for reflections with $F_o^2 > 2\sigma(F_o^2)$.

[b] $wR_2 = [\sum w(F_o^2 - F_c^2)^2/\sum(F_o^2)^2]^{1/2}$ for all reflections.

3. Results and discussion

Recently, several papers have described the synthesis of actinide complexes of the chelating borohydride ligand $H_3BNMe_2BH_3^-$, the *N*,*N*-dimethylaminodiboranate anion [9–11]. For example, at room temperature, UCl_4 reacts with 4 equiv of $Na(H_3BNMe_2BH_3)$ [7,12,13] in diethyl ether to yield $U(H_3BNMe_2BH_3)_3$, whereas reactions carried out in the presence of tetrahydrofuran afford the adduct $U(H_3BNMe_2BH_3)_3(thf)$ [9,10]. In both cases, the reaction is accompanied by reduction to uranium(III) and no uranium(IV) products could be isolated.

In further studying this system, we carried out the reaction of UCl_4 and 4 equiv of $Na(H_3BNMe_2BH_3)$ in refluxing 1,2-dimethoxyethane (dme). This reaction yields small amounts of a new complex, which we formulate as $U_2(\mu\text{-}O)(BH_4)_6(dme)_2{\cdot}C_7H_8$, **1**, as emerald green prisms by crystallization from a 1:1 toluene/pentane mixture. The formation of BH_4^- groups from $H_3BNMe_2BH_3^-$ at elevated temperatures has a precedent: we have shown elsewhere that an identical conversion takes place in the coordination sphere of thorium at elevated temperatures, and that this reaction occurs with the elimination of 1 equiv of the aminoborane $[Me_2NBH_2]_2$ [11]. The bridging oxo ligand in **1** almost certainly arises from adventitious water [14,15].

Single-crystal X-ray diffraction studies of **1** support the assigned stoichiometry (Fig. 1). Each uranium center adopts a *fac* octahedral geometry (counting the BH_4^- groups as occupying one coordination site); the bridging oxygen atom and the two coordinated oxygen atoms of the dme ligand occupy positions *trans* to the three BH_4^- groups. The hydride positions were located in the difference maps and reveal that all three BH_4^- groups are bound in a κ^3H (tridentate) fashion. The U···B distance to the BH_4^- group that is *trans* to the bridging oxygen, 2.635(7) Å, is about 0.06 Å longer that the U···B distances to the two groups that are *cis* to the bridging oxygen atom, 2.574(6) and 2.584(6) Å. Thus, the bridging oxygen atom exerts a noticeable *trans* influence, as is usually seen for oxo groups. In general, the U···B distances are similar to those observed in other U^{IV} complexes with κ^3-BH_4^- groups [3,16].

The U–O bond distances to the dme molecule are 2.498(3)–2.544(4) Å. The distances are slightly longer than those observed for the adducts of $U(BH_4)_4$ with dimethyl ether (2.44 Å) [17], diethyl ether (2.49 Å) [17], di-*n*-propyl ether (2.48 Å) [18], and tetrahydrofuran (2.47 Å) [19]. The bridging oxygen atom in **1** rests between the uranium atoms, and the U–O–U angle is nearly linear at 172.9(2)°. The U–O distances of 2.074(3) and 2.080(3) Å are

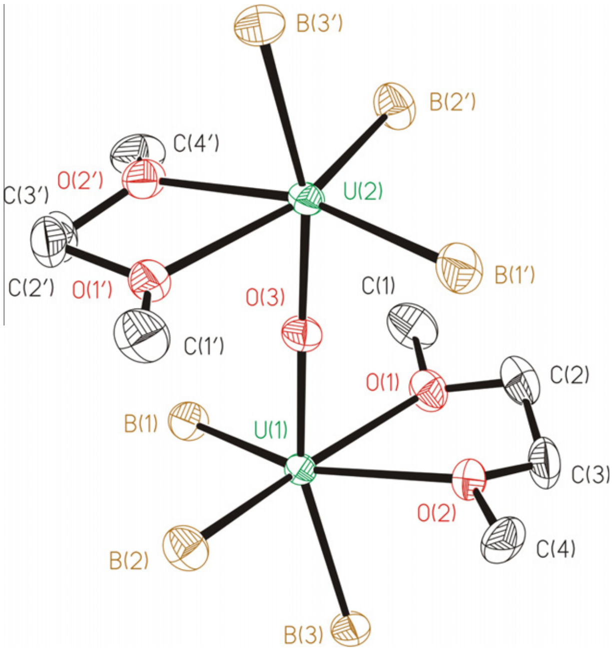

Fig. 1. Molecular structure of $\mathrm{U_2(\mu\text{-}O)(BH_4)_6(dme)_2}$, **1**. Ellipsoids are drawn at the 35% probability level. Hydrogen atoms and the disordered toluene solvate molecule have been omitted. The primed and unprimed atoms are not related by symmetry, but are related by the inversion center in the smaller cell chosen for $\mathrm{U_2(\mu\text{-}H)_2(BH_4)_6(dme)_2}$.

comparable to those observed in other $\mathrm{U^{IV}}$ μ-oxo compounds, such as $\mathrm{U_2(\mu\text{-}O)_2[C_5H_3(SiMe_3)_2]_4}$ (2.10 and 2.13 Å) [20], $\mathrm{U_2(\mu\text{-}O)(C_5H_4SiMe_3)_6}$ (2.11 Å) [21], and $\mathrm{U_3(\mu\text{-}O)_3(C_5H_4SiMe_3)_6}$ (2.05–2.12 Å) [22]. Only one other uranium oxo/borohydride complex has been isolated and crystallographically characterized, the uranyl complex $\mathrm{UO_2(\kappa^2\text{-}BH_4)_2(hmpa)_2}$, where hmpa = hexamethylphosphoramide [23].

Aside from the identity of the bridging ligands, the structure of $\mathrm{U_2(\mu\text{-}O)(BH_4)_6(dme)_2}$ is remarkably similar to that of a previously reported complex, the hydride $\mathrm{U_2(\mu\text{-}H)_2(BH_4)_6(dme)_2}$ (Table 2). Both compounds crystallize from 1:1 pentane:toluene in the triclinic space group $P\bar{1}$, with one molecule of toluene per dinuclear unit. The U···U distance is 4.146(3) Å in **1** versus 4.12 Å in the hydride, and all the ligands are disposed in exactly the same fashion. Significantly, the structure report for $\mathrm{U_2(\mu\text{-}H)_2(BH_4)_6(dme)_2}$ noted that there was a spurious peak of electron density midway between the two uranium atoms that could not be explained. We believe that this peak was due to an oxygen atom and that the hydride complex should be reformulated as $\mathrm{U_2(\mu\text{-}O)(BH_4)_6(dme)_2}$.

In the study of $\mathrm{U_2(\mu\text{-}H)_2(BH_4)_6(dme)_2}$, direct evidence in support of the presence of two bridging hydrides was unavailable: no hydride resonance could be located in the ^{1}H NMR spectrum, and no electron density corresponding to a pair of bridging hydrogen atoms could be found in the electron density difference map. It is well known that it is difficult to obtain direct evidence of bridging hydrogen atoms in uranium complexes owing to the paramagnetism and the low X-ray scattering power of hydrogen atoms relative to uranium, but a re-evaluation of the evidence suggests that the two uranium atoms are bridged by an oxygen atom and not by two hydrogen atoms. The paper describing the uranium hydride reported that the microanalytical data were satisfactory, and this finding is consistent with the proposed reformulation: the calculated C, H, B, and U weight percentages of 12.6%, 5.83%, 8.52%, and 62.5% for the oxo formula and 12.9%, 6.20%, 8.68%, and 63.7% for the hydride formula are almost identical.

Interestingly, the unit cell for **1** is different from that reported for the uranium hydride, the current cell having approximately twice the volume and different cell parameters. But we believe that both crystal structures are of the same crystalline substance. If the cell parameters for the hydride crystal structure are designated with primes, then the two unit cells are related by the following transformation: $\vec{a}' = ½\,(-\vec{a}+\vec{c})$, $\vec{b}' = ½\,(\vec{a}+\vec{c})$, and $\vec{c}' = \vec{b}$. If we apply this transformation to the cell parameters measured for **1**, the result is as follows (with the cell parameters reported for the hydride given in parentheses): $a' = 8.084$ (8.126), $b' = 8.976$ (8.950), $c' = 11.500$ (11.638) Å, $\alpha' = 83.70$ (83.50)°, $\beta' = 88.92$ (89.44)°, and $\gamma' = 68.21$ (69.76)°. The exact values are slightly different, probably because the crystal temperature was different for the two data sets.

In the larger (correct) unit cell, the bridging oxygen atom lies at a general position, but its coordinates are very near (¼,0,¾). As a result, this atom lies almost exactly halfway between inversion centers in the *ac* plane of the larger $P\bar{1}$ unit cell (Fig. 2). Because the individual molecules of **1** have very nearly ideal (but noncrystallographic) inversion symmetry, there is a large degree of pseudosymmetry: the crystal coordinates almost (but not exactly) correspond to a smaller unit cell, also of $P\bar{1}$ symmetry, in which an additional inversion center is present on the bridging oxygen atom. Several lines of evidence speak in support of the larger unit cell being the correct one: (1) additional (albeit weak) reflections appear in the diffraction record that correspond only to this larger cell, (2) the two ends of each molecule in the larger unit cell are not related by symmetry (as assessed by Platon) [24], (3) there are no large correlation coefficients between any of the parameters for the non-hydrogen atoms in the least squares matrix, and (4) hydrogen atoms could be located and refined (which was not possible for the crystal of the supposed uranium hydride, although this result could also have been a consequence of larger errors in the intensity measurements). In the previous refinement, in contrast, the incorrect choice of unit cell and consequent averaging of the atomic coordinates related by the pseudosymmetry caused some significant errors. For example, the boron atom *trans* to the bridging oxo group had unusually large displacement parameters, and, in contrast to the expected *trans* influence, its U···B distance was 0.09 Å shorter (instead of 0.06 Å longer) than the two U···B distances to the other $\mathrm{BH_4^-}$ ligands.

Table 2
Selected bond lengths and angles for $\mathrm{U_2(\mu\text{-}O)(BH_4)_6(dme)_2{\cdot}C_7H_8}$, **1**, with those reported for $\mathrm{U_2(\mu\text{-}H)_2(BH_4)_6(dme)_2{\cdot}C_7H_8}$ in square brackets.

Bond lengths (Å)				
U(1)–O(1)	2.544(4)	[2.53]	U(2)–O(1′)	2.503(4)
U(1)–O(2)	2.498(3)	[2.47]	U(2)–O(2′)	2.518(4)
U(1)–B(1)	2.574(6)	[2.64]	U(2)–B(1′)	2.595(7)
U(1)–B(2)	2.584(8)	[2.64]	U(2)–B(2′)	2.577(7)
U(1)–B(3)	2.635(7)	[2.53]	U(2)–B(3′)	2.631(7)
U(1)–O(3)	2.080(3)	[N.A.]	U(2)–O(3)	2.074(3)
U(1)–U(2)	4.146(3)	[4.12]		
Bond angles (°)				
O(1)–U(1)–O(2)	65.12(12)	[65.1]	O(1′)–U(2)–O(2′)	65.39(13)
B(1)–U(1)–B(2)	104.2(2)	[105]	B(1′)–U(2)–B(2′)	104.8(2)
B(1)–U(1)–B(3)	96.8(2)	[103]	B(1′)–U(2)–B(3′)	95.6(2)
B(2)–U(1)–B(3)	96.0(2)	[97]	B(2′)–U(2)–B(3′)	95.6(2)
O(1)–U(1)–B(1)	88.68(19)	[93.4]	O(1′)–U(2)–B(1′)	98.8(2)
O(1)–U(1)–B(3)	88.82(18)	[84]	O(1′)–U(2)–B(3′)	84.63(18)
O(2)–U(1)–B(2)–	101.83(18)	[96]	O(2′)–U(2)–B(2′)	90.79(19)
O(2)–U(1)–B(3)	83.00(17)	[78]	O(2′)–U(2)–B(3′)	89.14(18)
U(1)–O(3)–U(2)	172.94(18)	[180]		

The ^{1}H NMR spectrum of $\mathrm{U_2(\mu\text{-}O)(BH_4)_6(dme)_2}$ at −60 °C in $\mathrm{C_7D_8}$ closely resembles that reported for $\mathrm{U_2(\mu\text{-}H)_2(BH_4)_6(dme)_2}$ under similar conditions (Table 3). The most striking similarity is the chemical shift of one of the $\mathrm{BH_4}$ resonances, which is shifted

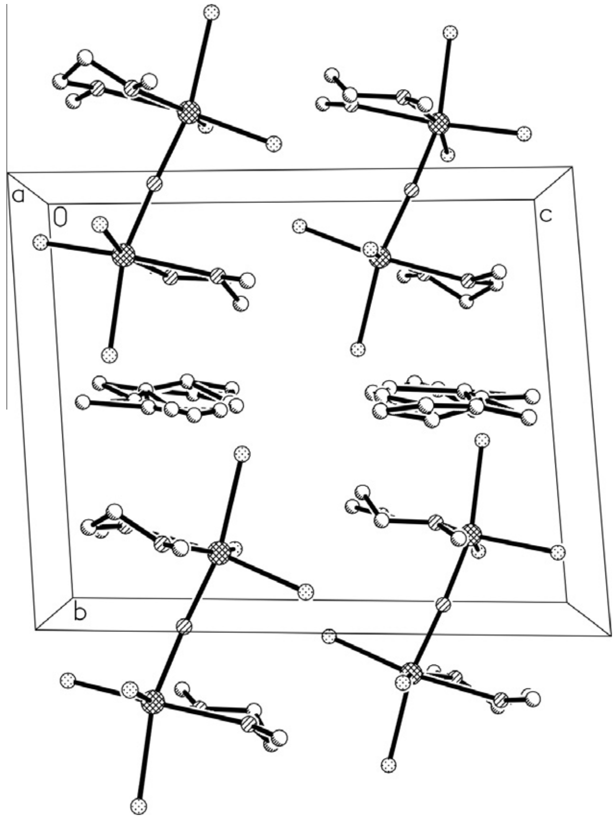

Fig. 2. Unit cell of $U_2(\mu\text{-}O)(BH_4)_6(dme)_2{\cdot}C_7H_8$, viewed down the *a*-axis. Ellipsoids are drawn at the 50% probability level. Both disordered components of the toluene molecule are shown. Hydrogen atoms have been deleted for clarity.

Table 3
^{1}H NMR shifts for $U_2(\mu\text{-}O)(BH_4)_6(dme)_2$, **1**, at −60 °C in toluene (600 MHz) and comparison to those reported for $U_2(\mu\text{-}H)_2(BH_4)_6(dme)_2$ (60 MHz) [1].

	$U_2(\mu\text{-}O)(BH_4)_6(dme)_2$	$U_2(\mu\text{-}H)_2(BH_4)_6(dme)_2$
BH_4	727 (br s, fwhm = 2700 Hz)	752.6 (br s, fwhm = 530 Hz, 4H)
BH_4	−115 (s, fwhm = 290 Hz, 4H)	−121.3 (br s, fwhm = 140 Hz, 8H)
OCH_2	−32.4 (s, fwhm = 80 Hz, 2H)	−31.6 (s, 2H)
OCH_2	−52.9 (s, fwhm = 110 Hz, 2H)	−52.3 (s, 2H)
OMe	−62.0 (s, fwhm = 110 Hz, 6H)	−62.8 (s, 6H)

dramatically to a lower field: δ 727, compared to the reported value of δ 753 for $U_2(\mu\text{-}H)_2(BH_4)_6(dme)_2$. We do suggest that the assignments of the two BH_4 resonances should be reversed, however: the resonance at δ −115 integrates to four protons in our spectrum (versus eight reported previously), and thus this resonance is best assigned to the BH_4^- group that is *trans* to the bridging oxo ligand. We could not obtain an accurate integral for the resonance at δ 727 owing to its large shift and line width. Integrations and chemical shifts for the dme resonances match those reported previously.

In summary, the crystallographic and ^{1}H NMR data presented here strongly suggest that the uranium hydride complex $U_2(\mu\text{-}H)_2(BH_4)_6(dme)_2$ should be reformulated as the oxo complex $U_2(\mu\text{-}O)(BH_4)_6(dme)_2$.

Acknowledgments

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Appendix A. Supplementary data

CCDC 838088 contains the supplementary crystallographic data for compound **1**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223 336 033; or e-mail: deposit@ccdc.cam.ac.uk.

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