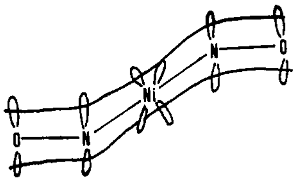
marily p_z type for N and O and d_{xz} for the nickel atom.



This results in pairing of the odd electrons in the radical ligands and a low-energy metal–ligand electron-transfer band in the visible spectra.

For comparison a recent study[15] on an oxidized octahedral cyclam complex of nickel is interesting. The Ni–N bond distances average 2.003 (3) Å, which is consistent with the average distances in the +2 and +4 complexes of the HP ligand, 2.058 (4) Å, but closer to the average distance with the +4 oxidation state, 1.986 (4) Å.

The discovery of this DMG-like complex opens several interesting new areas of study involving metal ion replacement. The observation that the complex color immediately disappears with even dilute acid and reappears (~70%) upon rapid neutralization suggests that the ligand has some independent stability and can re-form complexes. (There is still the possibility that protonation destroys the chromophore without dissociation of the ligand from the metal ion.) When dissociation and recombination is accomplished in the presence of an excess of another metal ion, i.e. Cu(II), new species are formed that contain the new metal ion. Purification and isolation of these complexes have not yet been accomplished.

Conclusions

A new planar nickel complex has been formed by the air oxidation of a tridentate amine oxime complex. It is formulated as a Ni(II) complex of an unstable radical ligand that achieves stability by π bonding utilizing an O=N=Ni=N=O delocalized bond system. This results in an extremely short Ni–N(HA) bond distance and a very planar complex.

Acknowledgment. Financial support from the National Science Foundation (Grant CHE81-06795) and from the University of Missouri Research Council is gratefully acknowledged.

Appendix

For those having a special interest in this type of structure, we have recently learned of an unpublished structure of bis(diiminosuccinonitrilo)nickel(II), which also apparently has delocalized π bonding including a nickel atom with an average Ni–N distance 1.829 (2) Å.[16]

Registry No. [Ni(HAO)$_2$], 84432-86-0.

Supplementary Material Available: Listings of observed and calculated structure factor amplitudes, anisotropic temperature factors, and least-squares planes and a stereoscopic view of the unit cell (8 pages). Ordering information is given on any current masthead page.

---

[15] Zeigerson, E.; Bar, I.; Bernstein, J.; Kirschenbaum, L. J.; Meyerstein, D. *Inorg. Chem.* **1980**, *21*, 73.

[16] Lauher, J. W., unpublished results. Correspondence should be addressed to Dr. Lauher at the Department of Chemistry, State University of New York.

Contribution from the Chemistry Division, Argonne National Laboratory, Argonne, Illinois 60439,
and Department of Chemistry, Northwestern University, Evanston, Illinois 60201

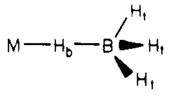
Metrical Characterization of Tridentate Tetrahydroborate Ligation to a Transition-Metal Ion. Structure and Bonding in Hf(BH$_4$)$_4$ by Single-Crystal Neutron Diffraction

ROBERT W. BROACH,[1a] I-SSUER CHUANG,[1b] TOBIN J. MARKS,*[1b] and JACK M. WILLIAMS*[1a]

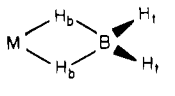
Received July 14, 1982

This paper reports the molecular structure of the transition-metal tetrahydroborate complex Hf(BH$_4$)$_4$ by single-crystal neutron diffraction at 110 K. The compound crystallizes in the cubic space group $P\bar{4}3m$ (T_d^1, No. 215) with a unit cell of dimensions a = 5.827 (4) Å. Full-matrix least-squares refinement gave a final value of $R(F_o^2)$ = 0.103 for 71 unique reflections with $F_o^2 > \sigma(F_o^2)$. The molecular structure consists of monomeric Hf(BH$_4$)$_4$ units having rigorously T_d symmetry and tridentate tetrahydroborate coordination. Important bond distances are Hf–B = 2.281 (8) Å, Hf–H(bridging) = 2.130 (9) Å, B–H(bridging) = 1.235 (10) Å, and B–H(terminal) = 1.150 (19) Å. Important bond angles are Hf–H(bridging)–B = 80.6 (6)°, H(bridging)–B–H(bridging) = 105.8 (6)°, and H(bridging)–B–H(terminal) = 112.9 (10)°. It is seen that the internal BH$_4^-$ metrical parameters do not differ significantly from those in Hf(CH$_3$C$_5$H$_4$)$_2$(BH$_4$)$_2$, in which the tetrahydroborate ligands are coordinated in an unsymmetrical bidentate fashion. However, Hf–B is 0.27 (1) Å longer and Hf–H(bridging, average) 0.035 (9) Å shorter in the latter molecule.

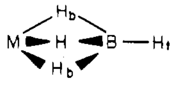
Transition-metal, lanthanide, and actinide complexes of the tetrahydroborate ligand, BH$_4^-$, exhibit a number of interesting and unusual properties.[2] These include coordination through metal–hydrogen–boron multicenter bonds (A, B, or C), very low barriers to intramolecular bridge–terminal (H$_b$–H$_t$) hydrogen interchange, covalent properties reminiscent of organometallic compounds such as η^3-allyls, and, in some instances, catalytic activity. That BH$_4^-$ and CH$_4$ are isoelec-



A



B



C

---

[1] (a) Argonne National Laboratory. (b) Northwestern University.

[2] (a) Marks, T. J.; Kolb, J. R. *Chem. Rev.* **1977**, *77*, 263–293. (b) Wegner, P. A. In "Boron Hydride Chemistry"; Muetterties, E. L., Ed.; Academic Press: New York, 1973; Chapter 12. (c) James, B. D.; Wallbridge, M. G. H. *Prog. Inorg. Chem.* **1970**, *11*, 99–231.

0020-1669/83/1322-1081$01.50/0

Table I. Final Positional[a] and Thermal[b] Parameters for $Hf(BH_4)_4$

atom	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Hf	0.0	0.0	0.0	0.017 (4)	0.017	0.017	0.0	0.0	0.0
B	0.226 (2)	0.226	0.226	0.022 (4)	0.022	0.022	−0.002 (4)	−0.002	−0.002
H_t	0.340 (5)	0.340	0.340	0.040 (10)	0.040	0.040	−0.010 (8)	−0.010	−0.010
H_b	0.258 (2)	0.258	0.019 (2)	0.050 (6)	0.050	0.032 (3)	−0.018 (3)	−0.001 (4)	−0.001

[a] x, y, and z are fractional coordinates. [b] Anisotropic temperature factors are of the form $\exp[-2\pi^2(a^{*}U_{11}h^2 + \ldots + 2a^{*}b^{*}U_{12}hk + \ldots)]$.

tronic also suggests the possibility of structurally modeling some aspects of saturated hydrocarbon C–H activation processes. Although impressive progress has been made in elaborating the variety of known metal tetrahydroborates and in elucidating chemical and structural/dynamic properties, areas of deficiency still exist in our understanding. In particular, there is still a paucity of accurate, neutron-diffraction-derived structural data that would allow more precise correlation of spectral and metrical parameters with bonding models and with chemical reactivity.

The homoleptic tetrahydroborates, $M(BH_4)_4$, where M = Zr, Hf, are perhaps the simplest and have been by far the most extensively studied transition-metal tetrahydroborates. Areas of investigation have included vibrational[3] and photoelectron[4] spectroscopy, LCAO–HFS (Xα) electronic structure calculations[4b], molecular structure determination by X-ray[5] and electron[6] diffraction (M = Zr), and NMR studies of molecular dynamics and boron quadrupolar relaxation in solution[7] and in the solid state,[8] as well as catalysis and surface chemistry.[2,9] However, structural data of neutron precision are still lacking for these or indeed any tridentate (C) transition-metal tetrahydroborate complex. In the present contribution, we report a study of the molecular structure of $Hf(BH_4)_4$ (T = 110 K) by single-crystal neutron diffraction.[10] Our results provide further metrical information on the ligation of BH_4^- by a triple hydrogen bridge[11] and complement our recent spectroscopic and neutron diffraction study of $Hf(\eta^5\text{-}CH_3C_5H_4)_2(BH_4)_2$,[12] in which tetrahydroborate is bound to the *same* metal ion, but via an unsymmetrical, double hydrogen bridge.

Experimental Section

Preparation of $Hf(BH_4)_4$. The extremely air-sensitive compound $Hf(BH_4)_4$ was prepared by the procedure of James and Smith[13] and was handled under purified nitrogen or in vacuo at all times. Large single crystals suitable for neutron data collection were obtained by slow (several months) sublimation in a closed container at −10 °C under nitrogen.

Table II. Selected Interatomic Distances and Intramolecular Angles in $Hf(BH_4)_4$

Distances, Å

Hf-B	2.281 (8)	Hf-H_b	2.130 (9)	B-H_b	1.235 (10)
B-H_t	1.150 (9)	H_b-H_t	1.988 (10)	H_b-H_b	1.970 (16)

Angles, deg

Hf-B-H_b	67.1 (5)	Hf-H_b-B	80.6 (6)
B-Hf-H_b	32.3 (3)	H_b-B-H_b	105.8 (6)
H_b-B-H_t	112.9 (10)		

Single-Crystal Neutron Data Collection. A well-formed, cube-shaped crystal, having equal dimensions of 1.5 mm on each edge and weighing approximately 7.3 mg, was selected for collection of intensity data. After carefully wedging the crystal with quartz glass wool into the end of a lead glass capillary,[14] the capillary was sealed under nitrogen and mounted in an arbitrary orientation on the fully automated[15] Electronics and Alloys four-circle diffractometer at the CP-5 research reactor at Argonne National Laboratory. The crystal was cooled and maintained at 110 K throughout the alignment and data collection.[16] Least-squares refinement of the setting angles for 19 carefully centered reflections gave a lattice constant of a = 5.827 (4) Å and a cell volume of 197.8 Å^3 for the cubic space group $P\bar{4}3m$ (T_d^1, No. 215). The calculated density is 1.997 g cm^{-3} for one molecule per cell.

An orientation matrix for data collection was generated from the centered reflections. Intensity data were measured by using coupled θ–2θ step-scans in one independent unit ($^1/_{24}$th sphere; $h \geq k \geq l \geq 0$) up to a maximum 2θ value of 120° (λ = 1.142 (1) Å). The step intervals were set at 0.1°, and scan ranges at 54 steps when $2\theta \leq 40°$, 58 steps when $40° < 2\theta \leq 70°$, 62 steps when $70° < 2\theta \leq 100°$, and 66 steps when $2\theta > 100°$. The diffracted reflection intensities, I, and estimated errors, $\sigma(I)$, were obtained from the step-scan data by optimizing the positions of the peak boundary points with a modification of the Lehmann and Larsen algorithm.[17] Intensities were corrected for absorption and the minimum and maximum transmission coefficients were 0.155 and 0.269, respectively (μ_c = 12.99 cm^{-1}).

The observed squared structure factors, F_o^2, were obtained from the intensities by application of eq 1,[18] where ω is the angular velocity

$$F_o^2 = (\omega I \sin (2\theta))/(I_0\lambda^3N^2V) \qquad (1)$$

of rotation of the crystal, θ the Bragg angle, I_0 the incident intensity, λ the wavelength, N the number of unit cells per unit volume, and V the specimen volume. A well-characterized NaCl crystal, for which precise absorption and secondary-extinction corrections had been made for all sampled reflections, was used to obtain I_0 in order to place the F_o^2 on an approximate absolute scale. The variances of F_o^2 were calculated from $\sigma^2(F_o^2) = \sigma_c^2(F_o^2) + (0.03F_o^2)^2$, where $\sigma_c^2(F_o^2)$ is determined from the counting statistics. The estimate of standard error of 0.03 was based on the maximum 3% variation in intensities of two check reflections that were measured at the beginning and end of data collection. The total of 162 data were then sorted and merged under $\bar{4}3m$ symmetry to give 99 unique data, all of which were used

---

(3) (a) Cyvin, B. N.; Cyvin, S. J.; Whitmer, J. C. *J. Mol. Struct.* **1979**, *53*, 281–286. (b) Smith, B. E.; Shurvell, H. F.; James, B. D. *J. Chem. Soc., Dalton Trans.* **1978**, 710–722. (c) Keiderling, T. A.; Wozniak, W. T.; Gay, R. S.; Jurkowitz, D.; Bernstein, E. R.; Lippard, S. J.; Spiro, T. J. *Inorg. Chem.* **1975**, *14*, 576–579. (d) Marks, T. J.; Kennelly, W. J.; Kolb, J. R.; Shimp, L. A. *Ibid.* **1972**, *11*, 2540–2546.

(4) (a) Downs, A. J.; Egdell, R. G.; Orchard, A. F.; Thomas, P. D. P. *J. Chem. Soc., Dalton Trans.* **1978**, 1755–1761. (b) Hitchcock, A. P.; Hao, N.; Werstiuk, N. H.; McGlinchey, M. J.; Ziegler, T. *Inorg. Chem.* **1982**, *21*, 793–798.

(5) Bird, P. H.; Churchill, M. R. *Chem. Commun.* **1967**, 403.

(6) Plato, V.; Hedberg, K. *Inorg. Chem.* **1971**, *10*, 590–594.

(7) (a) Marks, T. J.; Shimp, L. A. *J. Am. Chem. Soc.* **1972**, *94*, 1542–1550. (b) Sayer, B. G.; Thompson, J. I. A.; Hao, N.; Birchall, T.; Eaton, D.; McGlinchey, M. J. *Inorg. Chem.* **1981**, *20*, 3748–3750.

(8) Chuang, I-S.; Marks, T. J.; Kennelly, W. J.; Kolb, J. R. *J. Am. Chem. Soc.* **1977**, *99*, 7539–7545.

(9) (a) Evans, H. E.; Weinberg, W. H. *J. Am. Chem. Soc.* **1980**, *102*, 2548–2553. (b) Evans, H. E.; Weinberg, W. H. *Ibid.* **1980**, *102*, 2554–2558.

(10) For preliminary results from an earlier, incomplete structure determination, see ref 2a.

(11) The only other neutron diffraction study of a structure with tridentate BH_4^- coordination involves polymeric $U(BH_4)_4$. Here bidentate BH_4^- units, which bridge two uranium ions, are also present: Bernstein, E. R.; Hamilton, W. C.; Keiderling, T. A.; LaPlaca, S. J.; Lippard, S. J.; Mayerle, J. J. *Inorg. Chem.* **1972**, *11*, 3009–3016.

(12) Johnson, P. L.; Cohen, S. A.; Marks, T. J.; Williams, J. M. *J. Am. Chem. Soc.* **1978**, *100*, 2709–2716.

(13) James, B. D.; Smith, B. E. *Synth. Rect. Inorg. Met.-Org. Chem.* **1974**, *4*, 461–465.

(14) Mounting operations were carried out in a gloveblox. The laboratory temperature was maintained at ca. 5 °C to prevent melting of the crystals.

(15) Day, P.; Hines, J. *Oper. Syst. Rev.* **1973**, *7*, 28–38.

(16) Strouse, C. E. *Rev. Sci. Instrum.* **1976**, *47*, 871–876.

(17) Broach, R. W. Ph.D. Thesis, University of Wisconsin, Madison; Appendix I.

(18) Peterson S. W.; Levy, H. A. *J. Chem. Phys.* **1952**, *20*, 704–707.

Table III. Metrical Data from Selected Metal Tetrahydroborate Structure Determinations

compd	structural technique[d]	ligation mode	B-H_t, Å	B-H_b, Å	M-H_b, Å	M-B, Å	M-H-B, deg	ref
$Hf(BH_4)_4$	neutron	tridentate	1.150 (19)	1.235 (10)	2.130 (9)	2.281 (8)	80.6 (6)	this work
$Zr(BH_4)_4$	electron	tridentate	1.176 (40)	1.272 (16)	2.211 (19)	2.308 (3)	*a*	6
$U(BH_4)_4$	neutron	tridentate	1.24 (3)	1.23 (3)	2.34 (2)	2.52 (1)	83 (1)	11
		bidentate		1.25 (4)	2.41 (2)	2.86 (2)	98 (1)	
$Hf(CH_3C_5H_4)_2(BH_4)_2$	neutron	bidentate	1.186 (16)	1.255 (9)	2.069 (7)	2.553 (6)	98.1 (7)	12
		(unsymmetrical)	1.198 (15)	1.208 (13)	2.120 (8)		96.5 (7)	
$Co(terpy)BH_4$	neutron	bidentate	1.214 (10)	1.290 (9)	1.740 (12)	2.15 (1)	90.8 (6)	*b*
		(unsymmetrical)	1.217 (11)	1.287 (10)	1.707 (12)		89.2 (6)	
$Cu[P(CH_3)(C_6H_5)_2]_3BH_4$	neutron	monodentate	1.23 (5)	1.170 (5)	1.697 (5)	2.518 (3)	121.7 (4)	22
KBH_4	neutron	ionic	1.260 (2)	1.260 (2)				*c*
B_2H_6	electron	bidentate	1.196 (8)	1.339 (6)			83.0 (3)	23
$Al(BH_4)_3$	electron	bidentate	1.196 (12)	1.283 (12)	1.801 (6)	2.143 (3)	*a*	24

[a] Data not available. [b] Corey, E. J.; Cooper, N. J.; Canning, W. M.; Lipscomb, W. N.; Koetzle, T. F. *Inorg. Chem.* **1982**, *21*, 192-199. [c] Peterson, E. R. *Diss. Abstr.* **1965**, *25*, 5588. [d] Neutron or electron diffraction.

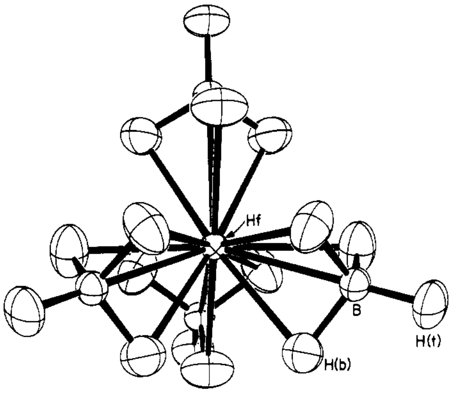


Figure 1. Static molecular structure of $Hf(BH_4)_4$ at 110 K as determined by neutron diffraction. Atoms are drawn to enclose 50% probability ellipsoids.

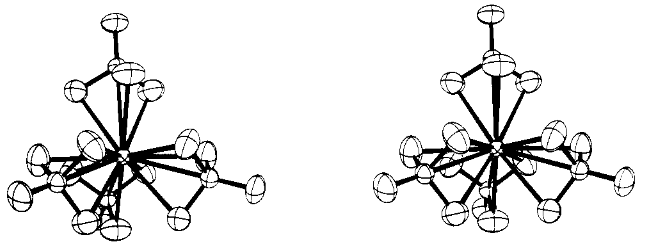


Figure 2. Stereoscopic view of the $Hf(BH_4)_4$ molecule, which possesses crystallographic $\bar{4}3m$ (T_d) symmetry.

in subsequent calculations. For reflections with negative intensities, F_o^2 was set to zero.

Structural Refinement. Since the unit cell contains only one molecule, the $Hf(BH_4)_4$ is required to have tetrahedral $\bar{4}3m$ (T_d) site symmetry. There are only four independent atoms in the structure: one hafnium, one boron, one bridging hydrogen, and one terminal hydrogen atom. Initial positional parameters for these atoms were taken from the previous neutron diffraction refinment.[10] Full-matrix least-squares refinement of the independent positional parameters and anisotropic temperature factors for each atom converged, with final discrepancy indices of $R(F_o) = 0.103$, $R(F_o^2) = 0.103$, and $R_w(F_o^2) = 0.172$ for data with $F_o^2 > \sigma(F_o^2)$ and $R(F_o) = 0.178$, $R(F_o^2) = 0.126$, $R_w(F_o^2) = 0.185$ for all data. These relatively high values are most likely indicative of the low intensities for the crystal examined. The correctness of the structure is indicated by the fact that a final difference Fourier map was actually featureless. The final data to parameter ratio was 7:1, and the standard deviation in an observation of unit weight was 1.62. There was no evidence for extinction. All least-squares refinements were based on the minimization of $\sum_i w_i(F_o^2 - F_c^2)^2$, with the weights w_i set equal to $1/\sigma^2(F_o^2)$. The coherent neutron scattering amplitudes used for Hf, B, and H were 0.78, 0.54, 0.021, and −0.374, respectively, all in units of 10^{-12} cm.

The atomic coordinates and thermal parameters are given in Table I. Interatomic distances and intramolecular angles are given in Table II. Estimated standard deviations given in the tables were calculated from the full variance–covariance matrix obtained from the final least-squares cycle.

Results and Discussion

The molecular structure of $Hf(BH_4)_4$ and the atom-labeling scheme are shown in Figure 1, a stereoview of the molecule is shown in Figure 2, and a stereoview of the unit cell contents is presented in Figure 3. Relevant metrical parameters are set out in Table II. The $Hf(BH_4)_4$ molecule possesses crystallographic $\bar{4}3m$ (T_d) symmetry. Each of the four tetrahydroborate anions functions as a tridentate ligand, bound to the hafnium ion through three hydrogen bridges. The hafnium, boron, and terminal hydrogen (H_t) atoms lie along the four crystallographic threefold axes: thus, the boron and terminal hydrogen atoms surround the metal in a tetrahedral arrangement, as required by the site symmetry. This is also

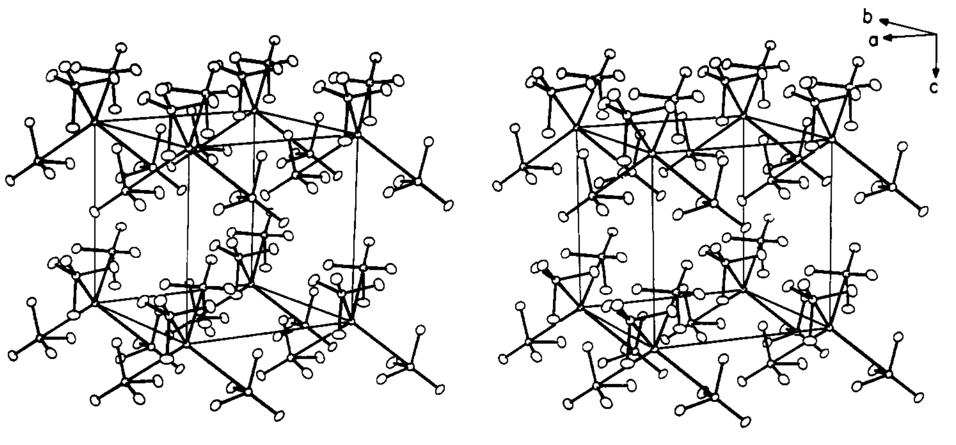


Figure 3. Stereoscopic projection of the cubic ($P\bar{4}3m$) unit cell contents. Atomic ellipsoids are drawn at the 50% probability level.

the arrangement found[5] by X-ray diffraction for $Zr(BH_4)_4$, which is isostructural.

The bond distances and angles in $Hf(BH_4)_4$ are most meaningful when compared to data for other metal tetrahydroborates. Examples where precise data are available are compiled in Table III,[19,20] which also indicates the structure determination method and the mode of BH_4^- coordination. It should be noted that $U(BH_4)_4$[11] is the only other tridentate tetrahydroborate for which neutron diffraction data are available. It can be seen that $Hf(BH_4)_4$ conforms to the general trend that $B-H_b$ distances are almost invariably longer than $B-H_t$ distances. This disparity is no doubt a consequence of the three-center two-electron bonding and is also evident in metal tetrahydroborate vibrational spectra ($B-H_b$ vs. $B-H_t$ stretching force constants) as well as in the structure of main-group borohydrides (cf. B_2H_6,[23] $Al(BH_4)_3$[24]). Unfortunately, the available $B-H_b$ vs. $B-H_t$ bond distance data are not in general precise enough to draw comparative conclusions about relative covalency and other factors that might depopulate the $B-H_b$ bonding molecular orbitals. The vibrational spectra appear to be somewhat more sensitive to such effects.[2a,12,25]

It can also be seen from the data in Tables II and III that the $M-H_b-B$ angle in $Hf(BH_4)_4$ conforms to the general trend that this angle decreases in the order monodentate > bidentate > tridentate. The angle in the present case agrees closely with that reported for the tridentate units in $U(BH_4)_4$,[11] i.e., 80.6 (6)° vs. 83 (1)°. It has already been noted that the metal–boron distances in metal tetrahydroborate complexes conform closely to the sum of the Shannon[26] effective ionic radii for the metal and the ionic radius for a bidentate (ca. 1.6 (1) Å) or tridentate (ca. 1.36 (6) Å) BH_4^- ligand.[2a,20a] In the present case, the observed Hf–B distance of 2.281 (8) Å is in good agreement with the calculated value of 2.31 Å and is substantially shorter than that in $Hf(CH_3C_5H_4)_2(BH_4)_2$, 2.553 (6) Å.[12] Other comparisons with this bidentate tetrahydroborate are also of interest. It can be seen in Table III that the $Hf-H_b$ (tridentate) distance of 2.130 (9) Å appears to be significantly longer than the average $Hf-H_b$ (bidentate) distance of 2.095 (8) Å. This result contrasts with that for $U(BH_4)_4$, where the $U-H_b$ (tridentate) distance of 2.34 (2) Å is probably shorter than the $U-H_b$ (bidentate, but bridging) distance of 2.41 (2) Å.

In regard to the relative internal dimensions of BH_4^- bound to Hf^{+4} via tridentate and bidentate ligation, it is not possible to conclude that there are significant differences. That is, a $B-H_t$(tridentate) contact of 1.150 (19) Å compares favorably to an average $B-H_t$(bidentate) contact of 1.192 (16) Å, and a $B-H_b$(tridentate) distance of 1.235 (10) Å is not significantly different from an average $B-H_b$(bidentate) distance of 1.232 (13) Å. Likewise, only marginally significant disparities are evident in the coordinated BH_4^- valence angles, where H_b-B-H_b(tridentate) = 105.8 (6)° vs. H_b-B-H_b(bidentate) = 108.4 (6)° and where H_b-B-H_t(tridentate) = 112.9 (1)° vs. an average H_b-B-H_t(bidentate) angle of 108.2 (9)°. Clearly, the perturbations of the BH_4^- framework are rather similar in the two hafnium complexes.

Acknowledgment. This work was performed under the auspices of the U.S. Department of Energy, Office of Basic Energy Sciences, Division of Materials Sciences, under Contract W-31-109-Eng.38. It was also supported by the National Science Foundation through support to T.J.M. (Grant CHE8009060) and through partial support of collaborative research between J.M.W. and T.J.M. (Grant CHE7820698).

Registry No. $Hf(BH_4)_4$, 53608-70-1.

Supplementary Material Available: Structure factor table (1 page). Ordering information is given on any current masthead page.

---

(19) For a discussion of the limitations of the various structural methods see ref 2a and 12 (footnote 26) and: Teller, R. G.; Bau, R. *Struct. Bonding (Berlin)* **1981**, *44*, 1-82.

(20) For compilations of all available structural data, see ref 2a and: (a) Edelstein, N. *Inorg. Chem.* **1981**, *20*, 297-299. (b) Segal, B.; Lippard, S. J. *Ibid.* **1978**, *17*, 844-850.

(21) Monodentate $Cu[P(CH_3)(C_6H_5)_2]_3BH_4$[22] and $U(BH_4)_4$[11] are cases where the magnitudes of the standard deviations do not permit a conclusion.

(22) Takusagawa, F.; Fumagalli, A.; Koetzle, T. F.; Shore, S. G.; Schmitkons, T.; Fratini, A. V.; Morse, K. W.; Wei, C.-Y.; Bau, R. *J. Am. Chem. Soc.* **1981**, *103*, 5165-5171.

(23) Bartell, L. S.; Carroll, B. L. *J. Chem. Phys.* **1965**, *42*, 1135-1139.

(24) Almenningen, A.; Gundersen, G.; Haaland, A. *Acta Chem. Scand.* **1968**, *22*, 328-334.

(25) (a) Kirtley, S. W.; Andrews, M. A.; Bau, R.; Grynkewich, G. W.; Marks, T. J.; Tipton, D. L.; Whittlesey, B. R. *J. Am. Chem. Soc.* **1977**, *99*, 7154-7162. (b) Marks, T. J.; Kennelly, W. J. *Ibid.* **1975**, *97*, 1439-1443.

(26) Shannon, R. D. *Acta Crystallogr., Sect. A* **1976**, *A32*, 751-757.