Lawrence Berkeley National Laboratory

Recent Work

Title

PREPARATION AND CRYSTAL STRUCTURES OF TETRAKIS (METHYLTRIHYDROBORATO)
URANIUM (IV) BIS (TETRAHYDROFURANATE) AND TETRAKIS (METHYLTRIHYDROBORATO)
URANIUM (IV) TETRAHYDROTHIOPHENATE

Permalink

<https://escholarship.org/uc/item/16d1h06m>

Authors

Shinomoto, R.
Zalkin, A.
Edelstein, N.M.

Publication Date

1987-04-01

Lawrence Berkeley Laboratory

UNIVERSITY OF CALIFORNIA

Materials & Chemical Sciences Division

RECEIVED
LAWRENCE
BERKELEY LABORATORY

JUN 9 1987

DOCUMENTS SECTION

Submitted to *Inorganica Chimica Acta*

**PREPARATION AND CRYSTAL STRUCTURES OF
TETRAKIS(METHYLTRIHYDROBORATO)URANIUM(IV)
BIS(TETRAHYDROFURANATE) AND
TETRAKIS(METHYLTRIHYDROBORATO)URANIUM(IV)
TETRAHYDROTHIOPHENATE**

R. Shinomoto, A. Zalkin, and N.M. Edelstein

April 1987

TWO-WEEK LOAN COPY

*This is a Library Circulating Copy
which may be borrowed for two weeks.*

LBL-23297
c.2

DISCLAIMER

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor the Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or the Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof or the Regents of the University of California.

Preparation and Crystal Structures of
Tetrakis(methyltrihydroborato)uranium(IV)bis(tetrahydrofuranate)
and
Tetrakis(methyltrihydroborato)uranium(IV)tetrahydrothiophenate

Ron Shinomoto, Allan Zalkin* and Norman M. Edelstein*

Materials and Chemical Sciences Division
Lawrence Berkeley Laboratory
University of California
Berkeley, California 94720 USA

Abstract. The U(IV) complexes, $U(BH_3CH_3)_4 \cdot 2OC_4H_8$ and $[U(BH_3CH_3)_4 \cdot SC_4H_8]_2$, have been prepared and their structures determined. Crystals of $(BH_3CH_3)_4U \cdot 2OC_4H_8$ are orthorhombic, $Aba2$, with $a = 12.911(3)$ Å, $b = 16.072(4)$ Å, $c = 10.116(4)$ Å, at 23 °C. For $Z = 2$ the calculated density is 1.574 g/cm³. Crystals of $[(CH_3BH_3)_4U \cdot SC_4H_8]_2$ are monoclinic, $P2_1/c$, with $a = 12.054(2)$, $b = 16.372(3)$, $c = 18.545(3)$ Å, $\beta = 108.82(2)^\circ$ at 23 °C. For $Z = 4$ the calculated density is 1.694 g/cm³. The structures were refined by full-matrix least-squares to a conventional R factor of 0.028, $[1378 \text{ data, } F^2 > 2\sigma(F^2)]$ for the tetrahydrofuran complex and 0.038, $[2732 \text{ data, } F^2 > 2\sigma(F^2)]$ for the tetrahydrothiophene complex. The tetrahydrofuran complex is monomolecular with the uranium atom on a two-fold axis and coordinated to four methyltrihydroborato and two tetrahydrofuran ligands; the U-B distances are 2.55(2) Å and 2.58(2) Å, and the U-O distance is 2.485(6) Å. The tetrahydrothiophene complex is a dimer in which two sulfur atoms bridge the uranium atoms of two tetra(methyltrihydroborato) uranium(IV) molecules; the U-B distances range from 2.468(2) to 2.591(2) Å, and the U-S distances are 3.120(4) and 3.239(5) Å; the S-S and U-U distances in the bridge are 3.477(5) and 5.374(1) Å, respectively.

Introduction

Structural studies of actinide(IV) tetrahydroborate and methyltrihydroborate complexes have been reported [1-5]. Typically the tetrahydroborate or methyltrihydroborate group is coordinated to the metal through bidentate or tridentate ligation:

In the case of the actinide(IV) tetrahydroborate complexes, the bidentate bridge often links two adjacent metal atoms. Examples include $\text{U}(\text{BH}_4)_4$ [1], $\text{U}(\text{BH}_4)_4 \cdot \text{OMe}_2$ [2a], $\text{U}(\text{BH}_4)_4 \cdot \text{OEt}_2$ [2a], and $\text{U}(\text{BH}_4)_4 \cdot \text{O}(\text{n-Pr})_2$ [2c]. In the dimeric complexes, $[\text{Th}(\text{BH}_3\text{CH}_3)_4]_2 \cdot \text{OEt}_2$ and $[\text{Th}(\text{BH}_3\text{CH}_3)_4 \cdot \text{THF}]_2$ [5c], the methyltrihydroborate ligand serves in a unique bridging capacity since it cannot form bidentate hydrogen bridges between adjacent metal atoms as in the actinide(IV) tetrahydroborate complexes.* The Th atoms are linked by a $\text{Th-H-BH}(\text{CH}_3)\text{-H-Th}$ bridge, with the unique hydrogen atom directly bridging the two Th atoms:

* $\text{Me} = \text{CH}_3$, $\text{Et} = \text{C}_2\text{H}_5$, $\text{Pr} = \text{C}_3\text{H}_7$, $\text{THF} = \text{OC}_4\text{H}_8$, $\text{THT} = \text{SC}_4\text{H}_8$.

This type of bridging is similar to that found in the complex, $\text{Co}_6(\text{BH}_4)_2[\text{Ph}_2\text{P}(\text{CH}_2)_5\text{PPh}_2]_2 \cdot 0.5 \text{C}_6\text{H}_6$ [6] and occurs when the coordination sphere about the metal ion is not saturated.

In order to explore further the coordination geometry of the BH_3CH_3^- ligand, we have prepared several other mono-base adducts of $\text{Th}(\text{BH}_3\text{CH}_3)_4$ and $\text{U}(\text{BH}_3\text{CH}_3)_4$ as well as some bis-base adducts [5d]. In this paper we report the synthesis and crystal structures of two uranium(IV) methyltrihydroborate complexes, $\text{U}(\text{BH}_3\text{CH}_3)_4 \cdot 2\text{THF}$ and $[\text{U}(\text{BH}_3\text{CH}_3)_4 \cdot \text{THT}]_2$.

Experimental

All preparations were carried out under an atmosphere of argon or under high vacuum. Toluene was dried over Na and distilled under argon before use. Diethyl ether and THF were distilled from sodium benzophenone ketyl under argon. Tetrahydrothiophene was dried over CaH_2 and distilled under argon before use. Benzene- d_6 and toluene- d_8 were degassed and dried over Na. Melting points were determined in sealed argon-filled capillaries and are uncorrected. LiBH_3CH_3 was prepared according to the procedure of Wartik and Schlesinger [7]. $\text{U}(\text{BH}_3\text{CH}_3)_4$ was prepared as described earlier [5a].

Preparation of $\text{U}(\text{BH}_3\text{CH}_3)_4 \cdot 2\text{THF}$. To 0.75 g (2.0 mmol) UCl_4 and 0.30 g (8.4 mmol) LiBH_3CH_3 , about 5 mL of THF was added and stirred for 12 hours. The THF was pumped off and the yellow brown residue sublimed at 75°C . A yield of 0.17 g (17%) of emerald green crystals was obtained. Its elemental analysis and ^1H NMR were consistent with the formula $\text{U}(\text{BH}_3\text{CH}_3)_4 \cdot 2\text{THF}$. Crystals suitable for X-ray crystallographic analysis were grown from a toluene solution at -20°C . M. p. = $95-8^\circ\text{C}$. Calculated for $\text{C}_{12}\text{H}_{10}\text{B}_4\text{O}_2\text{U}$: C, 28.96; H, 8.10. Found: C, 28.92; H, 7.87.

Preparation of $[\text{U}(\text{BH}_3\text{CH}_3)_4 \cdot \text{THT}]_2$. $\text{U}(\text{BH}_3\text{CH}_3)_4$ (0.32 g, 0.91 mmol) was dissolved in toluene; 0.16 mL (1.8 mmol) THT was added and stirred for a

couple of hours; the solution was filtered, concentrated to 3 mL and cooled to -20°C . Dark green crystals were obtained, which were washed with cold (-78°C) hexane. A yield of 0.08 g (20%) was obtained. Its elemental analysis and ^1H NMR indicate it has the empirical formula, $\text{U}(\text{BH}_3\text{CH}_3)_4 \cdot \text{THT}$. M. p. = $92-4^{\circ}\text{C}$. Calculated for $\text{C}_{16}\text{H}_{24}\text{B}_4\text{S}_2\text{U}_2$: C, 21.76; H, 7.30; S, 7.26. Found: C, 21.76; H, 7.17; S, 6.89.

Infra-red and ^1H NMR data are given in Tables I-III.

X-ray Diffraction. The air-sensitive crystals were sealed in quartz capillaries inside a dry box. A modified FACS-I Picker diffractometer was used to collect θ - 2θ scanned single crystal X-ray diffraction data using graphite monochromated $\text{MoK}\alpha$ radiation. For the orthorhombic tetrahydrofuran structure a quadrant of data was collected to a 2θ angle of 60° ; for the monoclinic thiophenate half a sphere of data was collected to a 2θ value of 50° . The data were corrected for absorption effects. The Friedel pairs in the non-centric orthorhombic structure were treated as independent reflections; the centric monoclinic equivalent reflections were averaged. Experimental details of the data collection and the results of the least-squares refinements are tabulated in the supplementary material. The structures were solved by Patterson and Fourier methods. The positional and anisotropic thermal parameters of the non-hydrogen atoms were refined by full-matrix least-squares. Hydrogen atoms were not included. Some of the difference Fourier peaks were identified as possible hydrogen atoms, but attempts to process them as hydrogen atoms in the least-squares refinements failed. The absolute configuration of the tetrahydrofuran complex was determined by comparing the results of the refinements of the enantiomorphic structure using hkl and $h\bar{k}l$ data (Friedel pairs); the structure reported

here resulted in an R factor of 0.028 vs 0.032 for the inverted structure. Neutral atomic scattering factors were used [8] with the corrections for anomalous scattering applied.

Discussion.

The crystal structure of $U(BH_3CH_3)_4 \cdot 2THF$ is very similar to that of $U(BH_4)_4 \cdot 2THF$ [2b]. The four BH_3CH_3 groups and 2 THF groups form a distorted octahedron about the uranium (Fig. 1). The THF groups are trans to each other. The U-B-C angles are close to 180° , indicative of tridentate ligation to the metal. The average U-B distance (2.57 ± 0.02 Å) compares favorably with the tridentate U-B distance found in $U(BH_4)_4 \cdot THF$ ($2.56(4)$ Å) [2b]. It is larger than that found for $U(BH_3CH_3)_4$ (2.48 ± 0.03 Å) [5a], presumably due to its larger coordination number (14 vs. 12). The U-O bond distance ($2.485(6)$ Å) is also close to that of $U(BH_4)_4 \cdot 2THF$ ($2.47(1)$ Å) [2b]. Selected bond distances and angles are given in Tables V and VI, respectively.

The crystal structure shows that $[U(BH_3CH_3)_4 \cdot THT]_2$ is dimeric, but with a different structure than $[Th(BH_3CH_3)_4 \cdot THF]_2$ [5c]. The two U atoms are bridged by the sulfur atoms of the THT groups (see Figure 2). The plane of the two bridging THT groups is perpendicular to the plane containing the U(1), U(2), S(1), S(2) atoms. Selected bond distances and angles are given in Tables V and VI. All of the BH_3CH_3 groups are tridentate. The average U-B distance is 2.54 ± 0.04 Å, which is comparable with the average $U-B_{\text{tridentate}}$ bond length of 2.57 ± 0.04 Å found in $U(BH_3CH_3)_4 \cdot 2THF$. The two average U-S bond lengths are 3.14 ± 0.03 and 3.26 ± 0.03 Å. There are two different BH_3CH_3 sites of equal population, those that are pointed towards the THT groups and those that are pointed away. These two different sites are observed in the 1H NMR spectrum. IR and 1H NMR spectral data are

presented in Tables I-III. In the ^1H NMR spectrum of $[\text{U}(\text{BH}_3\text{CH}_3)_4 \cdot \text{THT}]_2$, we see one BH_3CH_3 site, which coalesces and then splits into two different BH_3CH_3 sites of equal population at -90°C (see Table III). The mono-THT adduct of $\text{U}(\text{BH}_3\text{CH}_3)_4$ is probably not a monomer in solution like $\text{U}(\text{BH}_3\text{CH}_3)_4 \cdot \text{THF}$ since equivalent BH_3CH_3 sites were observed in the NMR spectrum at low temperatures for the latter [5c].

Geometric calculations using the cone angle model of Bagnall and Li [9] do not indicate any major steric factors in the coordination geometry of the $[\text{U}(\text{BH}_3\text{CH}_3)_4 \cdot \text{THT}]_2$ with bridging THT groups and an assumed structure based on the geometry of $[\text{Th}(\text{BH}_3\text{CH}_3)_4 \cdot \text{THF}]_2$ with bridging methyltrihydroborate groups. Thus electronic effects may be the reason for the preferred dimeric geometry in $[\text{U}(\text{BH}_3\text{CH}_3)_4 \cdot \text{THT}]_2$.

Comparison of the Lewis base adducts $\text{Th}(\text{BH}_3\text{CH}_3)_4$ and $\text{U}(\text{BH}_3\text{CH}_3)_4$ is instructive. The Th(IV) ion appears to require a larger coordination number which results in the formation of the unusual dimers $[\text{Th}(\text{BH}_3\text{CH}_3)_4]_2 \cdot \text{OEt}_2$ and $[\text{Th}(\text{BH}_3\text{CH}_3)_4]_2 \cdot 2\text{THF}$. The analagous $\text{U}(\text{BH}_3\text{CH}_3)_4$ compounds have not been isolated. $\text{Th}(\text{BH}_3\text{CH}_3)_4$ forms a bis THF complex, a bis THT complex and a mono THT complex. On the other hand we have only been able to isolate for $\text{U}(\text{BH}_3\text{CH}_3)_4$ the bis THF complex and the dimeric THT complex, $[\text{U}(\text{BH}_3\text{CH}_3)_4 \cdot \text{THT}]_2$. Clearly, the larger ionic radius of Th(IV) plays a major role in the unusual coordination chemistry of ether adducts of $\text{Th}(\text{BH}_3\text{CH}_3)_4$.

Supplementary Material Available

Listings of thermal parameters, additional distances and angles, and observed structure factors have been deposited with the Editor-in-Chief.

Acknowledgment

This work is supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Chemical Sciences Division of the U. S. Department of Energy under contract No. DE-AC03-76SF- 00098.

References

1. a) E. R. Bernstein, T. A. Keiderling, S. J. Lippard, J. J. Mayerle, J. Am. Chem. Soc., 94, 2522 (1972).
b) E. R. Bernstein, W. E. Hamilton, T. A. Keiderling, S. J. LaPlaca, S. J. Lippard, J. J. Mayerle, Inorg. Chem., 11, 3009 (1972).
2. a) R. R. Rietz, A. Zalkin, D. H. Templeton, N. M. Edelstein, L. K. Templeton, Inorg. Chem., 17, 653 (1978).
b) R. R. Rietz, N. M. Edelstein, H. W. Ruben, D. H. Templeton, A. Zalkin, Inorg. Chem., 17, 658 (1978).
c) A. Zalkin, R. R. Rietz, D. H. Templeton, N. M. Edelstein, Inorg. Chem., 17, 661 (1978).
3. a) R. H. Banks, N. M. Edelstein, R. R. Rietz, D. H. Templeton, A. Zalkin, J. Am. Chem. Soc., 100, 1957 (1978).
b) R. H. Banks, N. M. Edelstein, B. Spencer, D. H. Templeton, A. Zalkin, J. Am. Chem. Soc., 102, 620 (1980).
4. a) H. J. Wasserman, D. C. Moody, R. R. Ryan, J. Chem. Soc., Chem. Commun., 532 (1984).
b) H. J. Wasserman, D. C. Moody, R. T. Paine, R. R. Ryan, K. V. Salazar, J. Chem. Soc., Chem. Commun., 533 (1984).
5. a) R. Shinomoto, E. Gamp, N. M. Edelstein, D. H. Templeton, A. Zalkin, Inorg. Chem., 22, 2351 (1983).
b) J. Brennan, R. Shinomoto, A. Zalkin, N. M. Edelstein, Inorg. Chem., 23, 4143 (1984).
c) R. Shinomoto, J. G. Brennan, N. M. Edelstein, A. Zalkin, Inorg. Chem., 24, 2896 (1985).

- d) A. Zalkin, R. Shinomoto, N. M. Edelstein, D. Zhang, Submitted for publication.
6. D. G. Holah, A. N. Hughes, S. Maciaszek, V. R. Magnuson, J. Chem Soc., Chem. Commun., 1308 (1983).
 7. T. Wartik, H. I. Schlesinger, J. Am. Chem. Soc., 75, 835 (1953).
 8. International Tables for X-ray Crystallography Vol. IV: Birmingham: Kynoch Press, 1974, Table 2.2, pp 71-102.
 9. K. W. Bagnall, X.-f. Li, J. Chem. Soc., Dalton Trans., 1365 (1982).
 10. C. K. Johnson, ORTEP. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee (1965).

Table I. Infrared Spectroscopic Data

Compound	
$\text{U}(\text{BH}_3\text{CH}_3)_4 \cdot 2\text{THF}$	2950m, 2230m, 2140s, 2080s, 1340m, 1300s, 1225s, 1100m, 1065m, 1040m, 1010s, 950m, 925m, 850s, 835m, 735m, 670m
$[\text{U}(\text{BH}_3\text{CH}_3)_4 \cdot \text{THT}]_2$	2950m, 2160s, 2080s, 1300s, 1240s, 1070m, 1030w, 960w, 890w, 800m, 725m, 665w

Table II. Proton Magnetic Resonance Data^a

Compound		
$\text{U}(\text{BH}_3\text{CH}_3)_4 \cdot 2\text{THF}^{\text{b}}$	+5.51 (8H, s),	+10.63 (12H, s)
	+11.08 (8H, s),	+127.34 (12H, doub)
$[\text{U}(\text{BH}_3\text{CH}_3)_4 \cdot \text{THT}]_2^{\text{c}}$	+3.45 (8H, s),	+8.77 (8H, s)
	+13.81 (24H, s),	+145.17 (24H, doub)

a) Shifts in ppm from tetramethylsilane. Positive sign indicates downfield shift. All values at 30°C.

b) Concentration = 1.2×10^{-3} g/mL benzene- d_6 .

c) Concentration = 1.2×10^{-2} g/mL toluene- d_8 .

Table III. ^1H NMR Data for $[\text{U}(\text{BH}_3\text{CH}_3)_4 \cdot \text{THT}]_2$ ^{a,b,c}

T (°C)	BH ₃	CH ₃	THT
-90	-105.68 (12H, s)	-43.23 (12H, s)	-47.23 (8H, s)
	d	104.82 (12H, s)	-23.52 (8H, s)
-72	e	f	-30.75 (8H, s)
			-11.94 (8H, s)
-54	e	20.41 (24H, s) ^g	-10.79 (8H, s)
			-3.25 (8H, s)
30	145.17 (24H, s) ^h	13.81 (24H, s) ^g	3.45 (8H, s)
			8.77 (8H, s)

a) In toluene-d₈.

b) Shifts in ppm from Me₄Si. Positive sign indicates downfield shift.

c) Concentration = 0.012 g/mL.

d) Other BH₃ peak too far downfield to be observed.

e) BH₃ peak broadened into baseline due to rapid exchange of BH₃CH₃ sites.

f) CH₃ peak broadened into baseline due to rapid exchange of BH₃CH₃ sites.

g) Averaged CH₃ peak.

h) Averaged BH₃ peak.

Table IV. Positional Parameters with Estimated Standard Deviations^a

$$(\text{BH}_3\text{CH}_3)_4\text{U} \cdot 2(\text{OC}_4\text{H}_8)$$

Atom	x	y	z
U	0	0	0
O	-0.1732(5)	0.0674(4)	-0.0070(22)
B(1)	0.0384(13)	0.1097(10)	-0.1800(17)
B(2)	0.0849(11)	0.0903(10)	0.1773(15)
C(1)	0.0739(10)	0.1738(9)	-0.2940(14)
C(2)	0.1287(10)	0.1504(9)	0.2945(15)
C(3)	-0.2699(10)	0.0277(9)	0.026(6)
C(4)	-0.3554(9)	0.0915(11)	0.018(4)
C(5)	-0.3068(11)	0.1704(10)	0.015(5)
C(6)	-0.1922(11)	0.1544(8)	-0.023(5)

$$[(\text{CH}_3\text{BH}_3)_4\text{U} \cdot \text{SC}_4\text{H}_8]_2$$

Atom	x	y	z
U(1)	0.21704(6)	0.20201(4)	0.30664(4)
U(2)	0.27555(6)	-0.09460(4)	0.20013(4)
S(1)	0.3126(3)	0.10326(25)	0.19534(22)
S(2)	0.1840(3)	0.00630(28)	0.31517(23)
B(1)	0.1187(18)	0.1958(15)	0.4083(12)
B(2)	0.4320(15)	0.1723(11)	0.3856(11)
B(3)	0.2561(19)	0.3498(12)	0.2890(15)
B(4)	0.0398(17)	0.1933(13)	0.1876(12)
B(5)	0.0593(19)	-0.0632(14)	0.1264(13)
B(6)	0.4585(18)	-0.0821(13)	0.3205(12)

B(7)	0.2412(18)	-0.2406(13)	0.2185(14)
B(8)	0.3633(19)	-0.0935(13)	0.0944(14)
C(1)	0.0576(19)	0.1946(14)	0.4732(12)
C(2)	0.5670(15)	0.1593(12)	0.4342(11)
C(3)	0.2853(16)	0.4445(11)	0.2798(12)
C(4)	-0.0740(15)	0.1844(13)	0.1078(12)
C(5)	-0.0755(14)	-0.0427(12)	0.0826(11)
C(6)	0.5686(18)	-0.0786(12)	0.3957(12)
C(7)	0.2177(15)	-0.3373(12)	0.2252(13)
C(8)	0.4115(18)	-0.0980(12)	0.0230(12)
C(9)	0.4687(13)	0.1227(10)	0.2057(11)
C(10)	0.4670(19)	0.1660(17)	0.1324(13)
C(11)	0.3568(23)	0.1623(19)	0.0731(13)
C(12)	0.2513(15)	0.1381(12)	0.0956(9)
C(13)	0.0291(13)	-0.0181(11)	0.3090(10)
C(14)	0.0345(19)	-0.0569(18)	0.3823(14)
C(15)	0.1433(23)	-0.0566(20)	0.4386(15)
C(16)	0.2479(16)	-0.0280(12)	0.4165(9)

^a Estimated standard deviations in this and subsequent tables are indicated in parentheses.

Table V. Selected Distances(A) and Angles(°)

$(\text{BH}_3\text{CH}_3)_4\text{U} \cdot 2(\text{OC}_4\text{H}_8)^{\text{a}}$			$[(\text{CH}_3\text{BH}_3)_4\text{U} \cdot \text{SC}_4\text{H}_8]_2$			
U	-2 B(1)	2.583(17)	U(1) -B(1)	2.533(17)	U(2) -B(5)	2.574(22)
			U(1) -B(2)	2.578(18)	U(2) -B(6)	2.591(22)
U	-2 B(2)	2.554(15)	U(1) -B(3)	2.506(20)	U(2) -B(7)	2.468(21)
			U(1) -B(4)	2.532(22)	U(2) -B(8)	2.511(20)
U	-2 O	2.485(6)	U(1) -S(1)	3.120(4)	U(2) -S(1)	3.275(4)
			U(1) -S(2)	3.239(5)	U(2) -S(2)	3.165(4)
B(1)-	C(1)	1.613(21)	B(1) -C(1)	1.600(22)	B(5) -C(5)	1.601(26)
B(2)-	C(2)	1.630(19)	B(2) -C(2)	1.600(23)	B(6) -C(6)	1.585(27)
			B(3) -C(3)	1.611(24)	B(7) -C(7)	1.621(27)
			B(4) -C(4)	1.669(28)	B(8) -C(8)	1.610(25)
			S(1) -S(2)	3.477(5)	U(1) -U(2)	5.374(1)

Table V (continued)

$(\text{CH}_3\text{BH}_3)_4\text{U} \cdot 2(\text{OC}_4\text{H}_8)^{\text{a}}$		$[(\text{CH}_3\text{BH}_3)_4\text{U} \cdot \text{SC}_4\text{H}_8]_2$			
O-U-O	176.7(10)	S(1)-U(1)-S(2)	66.3(1)	S(1)-U(2)-S(2)	65.3(1)
O-U-B(1)	81.6(5)	S(1)-U(1)-B(1)	146.4(6)	S(1)-U(2)-B(5)	84.9(5)
O-U-B(1)	96.0(5)	S(1)-U(1)-B(2)	76.1(4)	S(1)-U(2)-B(6)	81.9(5)
O-U-B(2)	99.1(5)	S(1)-U(1)-B(3)	106.8(5)	S(1)-U(2)-B(7)	172.7(6)
O-U-B(2)	83.2(5)	S(1)-U(1)-B(4)	77.4(4)	S(1)-U(2)-B(8)	82.9(5)
B(1)-U-B(1)'	90.3(8)	S(2)-U(1)-B(1)	80.2(6)	S(2)-U(2)-B(5)	74.9(5)
B(1)-U-B(2)	91.4(4)	S(2)-U(1)-B(2)	84.4(4)	S(2)-U(2)-B(6)	76.3(5)
B(1)-U-B(2)'	164.9(4)	S(2)-U(1)-B(3)	173.0(5)	S(2)-U(2)-B(7)	107.6(6)
B(2)-U-B(2)'	90.8(7)	S(2)-U(1)-B(4)	84.5(5)	S(2)-U(2)-B(8)	148.1(5)
U-B(1)-C(1)	174.0(11)	B(1)-U(1)-B(2)	101.3(7)	B(5)-U(2)-B(6)	151.2(6)
O-B(1)-C(1)	138.0(10)	B(1)-U(1)-B(3)	106.8(8)	B(5)-U(2)-B(7)	94.6(7)
U-B(2)-C(2)	174.9(11)	B(1)-U(1)-B(4)	100.4(7)	B(5)-U(2)-B(8)	101.2(7)
O-B(2)-C(2)	137.4(11)	B(2)-U(1)-B(3)	93.4(7)	B(6)-U(2)-B(7)	95.3(7)
		B(2)-U(1)-B(4)	153.5(6)	B(6)-U(2)-B(8)	102.5(7)
		B(3)-U(1)-B(4)	94.7(8)	B(7)-U(2)-B(8)	104.3(7)
		U(1)-S(1)-U(2)	114.3(1)	U(1)-S(2)-U(2)	114.09(1)
		U(1)-B(1)-C(1)	178.3(16)	U(2)-B(5)-C(5)	178.5(14)
		U(1)-B(2)-C(2)	176.7(13)	U(2)-B(6)-C(6)	177.1(14)
		U(1)-B(3)-C(3)	178.1(17)	U(2)-B(7)-C(7)	176.5(18)
		U(1)-B(4)-C(4)	177.5(14)	U(2)-B(8)-C(8)	175.4(15)

^a Primed atoms are in position -x,-y,z.

Figures

Figure 1. ORTEP [10] drawing of $\text{U}(\text{BH}_3\text{CH}_3)_4 \cdot 2(\text{OC}_4\text{H}_8)$; thermal ellipsoids at 50% probability level.

Figure 2. ORTEP [10] drawing of $[(\text{CH}_3\text{BH}_3)_4\text{U} \cdot \text{S}_4\text{H}_8]$; thermal ellipsoids at 50% probability level.

XBL 8410-4299

Figure 1

XBL 8410-4301

Figure 2

This report was done with support from the Department of Energy. Any conclusions or opinions expressed in this report represent solely those of the author(s) and not necessarily those of The Regents of the University of California, the Lawrence Berkeley Laboratory or the Department of Energy.

Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Department of Energy to the exclusion of others that may be suitable.

*LAWRENCE BERKELEY LABORATORY
TECHNICAL INFORMATION DEPARTMENT
UNIVERSITY OF CALIFORNIA
BERKELEY, CALIFORNIA 94720*