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Structure of (Tetrahydroborato)bis(tetrahydrofuran)uranium(IV): a Model for Hydrogen–Uranium Bonding

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Abstract. $[U(BH_4)_4(C_4H_8O)_2]$, $M_r = 441\cdot6$, orthorhombic, *Pnma*, $a = 10\cdot439\ (3)$, $b = 7\cdot120\ (2)$, $c = 11\cdot315\ (3)$ Å, $V = 841\cdot0\ (7)$ Å^3, $Z = 2$, $D_x = 1\cdot74$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71073$ Å, $\mu = 9\cdot13$ mm^{-1}, $F(000) = 416$, $T = 295$ K, $R = 0\cdot037$ for 216 unique observed data. The molecular geometry is in essential agreement with that reported by Rietz, Edelstein, Ruben, Templeton & Zalkin [*Inorg. Chem.* (1978), **17**, 658–660], the major difference being the relatively high accepted thermal parameters at room temperature for the C and B atoms instead of disorder in both the C atoms of tetrahydrofuran (THF) and the B atoms. The complex is confirmed as a monomer with an octahedral arrangement around the U atom of four BH_4^- and two *trans* THF molecules. The ligation of BH_4^- to the U atom is through bridging H atoms, the four equivalent BH_4^- acting as tridentate ligands [U—B 2·53 (6) Å]. However, the bending of the line U—B—HB(4) [164 (4)°] makes the three bridging H atoms unequivalent, one being as close as 2·16 Å to the U atom.

Experimental. Tetrahydrofuran added to a solution of $U(BH_4)_4$ in deuterated toluene; after recording the NMR spectrum of the adduct at 213 K the solution is stored at 243 K and emerald-green crystals obtained; crystal 0·40 × 0·30 × 0·25 mm; Enraf–Nonius CAD-4 diffractometer controlled by a PDP 11/23 Plus computer, graphite-monochromatized Mo $K\alpha$ radiation; $\omega/2\theta$ scan technique; cell parameters obtained from least-squares procedure on 25 reflections ($8 < \theta < 12°$); decay of 11·8% (in 10 h) in intensities of three standard reflections monitored every 100 measurements, linearly corrected; Lorentz and polarization corrections; max. and min. transmission factors for an empirical absorption correction 1·21 and 0·75 (Walker & Stuart, 1983); total of 921 reflections measured in range $1 < \theta < 20°$; $0 < h < 10$, $0 < k < 6$, $-10 < l < 10$; 355 unique reflections, 216 with $I > 2\sigma(I)$. Structure solved by Patterson method for U; remaining atoms located by difference Fourier techniques; full-matrix least-squares refinement based on F; anisotropic thermal parameters for U; scattering factors including anomalous-dispersion terms from *International Tables for X-ray Crystallography* (1974). H atoms of the THF

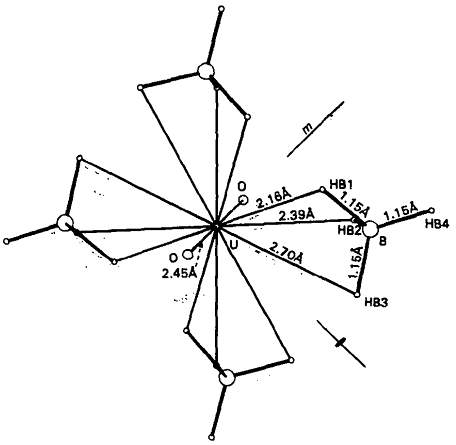

Fig. 1. *ORTEP* (Johnson, 1976) representation of the coordination sphere of the U atom, giving bond lengths (Å) and atomic numbering (symmetry of the U site 2/*m*).

ring introduced at calculated positions, not refined but constrained to ride their C atoms (C—H 0·95 Å; $B = 5$ Å^2). The last ΔF Fourier map exhibits poorly resolved peaks between the U and B atoms but a better peak for the terminal hydrogen HB(4) of the BH_4 group (height 0·8 e Å^{-3}), remaining H atoms located by imposing tetrahedral hydroborate geometry (B—H 1·15 Å and H—B—H 109°): H atoms constrained to ride their B atom. $R = 0{\cdot}037$, $wR = 0{\cdot}047$ ($w = 1$), $(\Delta/\sigma)_{\max} = 0{\cdot}06$, $S = 4{\cdot}15$, $(\Delta\rho)_{\max} = 0{\cdot}6$ e Å^{-3}; no extinction correction; *SDP* Enraf–Nonius programs (Frenz, 1983). Atomic parameters are given in Table 1, the coordination sphere of the U atom is in Fig. 1.*

Related literature. The bending of the line U—B—HB (terminal) of tridentate bonding was found in some other uranium hydroborates: $U(BH_4)_4$ form II (Charpin, Nierlich, Vigner, Lance & Baudry, 1987) and $U(BH_4)[OP(C_6H_5)_3]_2$ (Charpin, Nierlich, Chevrier, Vigner, Lance & Baudry, 1987).

Table 1. *Positional and equivalent isotropic thermal parameters*

	x	y	z	B(Å^2)
U	0·000	0·000	0·000	3·46 (4)*
B	0·174 (5)	0·217 (8)	−0·077 (5)	10 (2)
O	0·000	0·159 (4)	0·192 (3)	4·7 (7)
C(1)	0·000	0·08 (1)	0·307 (7)	12 (3)
C(2)	0·000	0·22 (1)	0·396 (8)	14 (3)
C(3)	0·000	0·386 (9)	0·331 (6)	10 (2)
C(4)	0·000	0·36 (1)	0·210 (8)	14 (3)
HB(1)	0·080	0·193	−0·127	
HB(2)	0·174	0·250	0·021	
HB(3)	0·236	0·083	−0·082	
HB(4)	0·229	0·340	−0·118	

* $B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43859 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of a Benzoyloxytetraphenylazadiene

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Abstract. 2-Diphenylmethyleneamino-1-phenylstyryl benzoate, $C_{34}H_{25}NO_2$, $M_r = 479{\cdot}579$, monoclinic, $P2_1/n$, $a = 19{\cdot}364$ (1), $b = 9{\cdot}775$ (2), $c = 14{\cdot}683$ (1) Å, $\beta = 108{\cdot}38$ (1)°, $V = 2632{\cdot}1$ (6) Å^3, $Z = 4$, $D_x = 1{\cdot}2102$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 5{\cdot}515$ cm^{-1}, $F(000) = 1008$, room temperature, final $R = 0{\cdot}060$, $wR = 0{\cdot}054$ for 3714 reflections. The azadiene chain is not planar. The benzoate group is very nearly planar with the carboxyl group rotated by 178·3 (4)° with respect to the benzene ring. The two phenyl rings attached to the C=C double bond in the azadiene chain are *trans*.

Experimental. Crystal 0·10 × 0·10 × 0·30 mm, Philips 1100 automatic four-circle diffractometer, graphite monochromator, Cu $K\alpha$ radiation. Lattice parameters determined by least-squares fit from 50 reflections with $\theta_{\max} = 24°$. Intensities measured with the same diffractometer, ω–2θ scan, $2 < \theta < 67°$; two reflections used as standard and remeasured after every 90 min, no decomposition observed. 4464 independent reflections ($-20 < h < 20$, $0 < k < 11$, $0 < l < 16$), 3714 observed with $I > 2\sigma(I)$, where $\sigma(I)$ is the standard deviation based on counting statistics. Lorentz and polarization corrections, no correction for absorption.

Structure solved by direct methods with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), refined by full-matrix least squares, $\sum w(|F_o| - |F_c|)^2$ minimized. After isotropic and anisotropic refinement, the resulting difference-density map revealed the positions of the H atoms. Further refinement with anisotropic tempera-

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