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Structure of Tris(1–3-η-indenyl)uranium Bromide

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Abstract. $[U(C_9H_7)_3]Br$, $M_r = 663{\cdot}41$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}528$ (2), $b = 14{\cdot}609$ (4), $c = 16{\cdot}650$ (4) Å, $V = 2074$ (2) Å^3, $Z = 4$, $D_x = 2{\cdot}124$ g cm^{-3}, $\lambda(\text{Mo }K\bar{\alpha}) = 0{\cdot}71073$ Å, $\mu = 93{\cdot}390$ cm^{-1}, $F(000) = 1240$, $T = 295$ (1) K, $R = 0{\cdot}033$ for 1055 observed reflections. The title compound is isostructural with its chlorine analogue, the Br and the five-membered rings of the three indenyl moieties being arranged tetrahedrally about the U atom. The U atom is bonded to the C atoms of the five-membered rings of the three indenides at distances ranging from 2·68 (2) to 2·91 (3) Å. The closest U—C approach is observed, in each case, to the three non-bridging atoms of the five-membered rings. This suggests trihapto U—C covalent bonding. Bond lengths and angles in the indenyl rings are all normal and within the range expected for five- and six-membered conjugated ring systems.

Experimental. Compound synthesized as described elsewhere (Goffart, Fuger, Gilbert, Hocks & Duyckaerts, 1975). Light-brown prismatic single crystals obtained by recrystallization from pentane solution at low temperature (243 K). Selected specimen (0·35 × 0·22 × 0·25 mm) sealed in a thin-walled glass capillary under an inert atmosphere. Enraf–Nonius CAD-4 X-ray diffractometer, graphite-monochromated radiation using θ–2θ scans. Unit cell determined from 25 reflections, $2 < 2\theta < 30°$. Space group established from systematic absences. Data collected in range $4 \le 2\theta \le 46°$, h 0→9, k −16→16, l −18→0, 6219 reflections collected, 4127 considered observed $[I \ge 2\sigma(I)]$, 1696 unique ($R_{\text{int}} = 0{\cdot}05$), 1055 reflections with $I \ge 3\sigma(I)$ used in refinement. Intensities of three standard reflections measured at 30 min intervals, anisotropic decay correction applied (total decay 6·1%). Empirical absorption corrections (transmission factors range from 33·25 to 99·65%). Structure solved by direct methods and refined by full-matrix least-squares techniques which minimized $\sum w(\Delta F)^2$. Anisotropic thermal parameters for U and Br atoms. H atoms included in final least-squares cycles in idealized positions with $B_{\text{iso}} = 5{\cdot}0$ Å^2. A secondary-extinction coefficient refined to $g = 1{\cdot}6 \times 10^{-7}$ $\{F_c = F_c/[1 + g(F_c)^2\text{Lp}]\}$. $R = 0{\cdot}033$, $wR = 0{\cdot}038$, $S = 1{\cdot}03$. Final $(\Delta/\sigma)_{\text{max}} = 0{\cdot}01$. Maximum and minimum heights in final difference Fourier map $+1{\cdot}56$ and $-1{\cdot}32$ e Å^{-3} around U atom (less than 1·1 Å away). Weighting scheme based on counting statistics: $w = 1/[\sigma(F_o)]^2$, $\sigma(F_o) = \sigma(F_o^2)/2F_o$, $\sigma(F_o^2) = [\sigma^2(I) + (PI)^2]^{1/2}/\text{Lp}$, P (ignorance factor used to downweight intense reflections) = 0·06. Atomic factors and anomalous-dispersion terms from *International Tables for X-ray Crystallography* (1974). Enraf–Nonius (1981) *SDP* programs. Atomic coordinates and isotropic temperature factors are given in Table 1.†

Selected bond distances are presented in Table 2. An *ORTEP* illustration (Johnson, 1976) of the molecular structure is given in Fig. 1.

Related literature. Structure data of the following organoactinide(IV) complexes of the indenide ligand have been reported in the literature: $[U(C_9H_7)_3]Cl$ (Burns & Laubereau, 1971), $[Th(C_{12}H_{13})_3]Cl$ (Spirlet, Rebizant & Goffart, 1982), $[U(C_9H_7)]Cl_3.2C_4H_8O$ (Rebizant, Spirlet & Goffart, 1983), $[U(C_9H_7)]Br_3.2C_4H_8O$ (Rebizant, Spirlet & Goffart, 1985), $[U(C_9H_7)]Br_3.C_4H_8O.OP(C_6H_5)_3$ (Meunier-Piret, Germain, Declercq & Van Meerssche, 1980).

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† Lists of structure factors, anisotropic temperature factors, H coordinates and a complete list of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43441 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Atomic positional and isotropic thermal parameters with e.s.d.'s in parentheses*

	x	y	z	B(Å^2)
U	0·03719 (7)	0·27246 (4)	0·24365 (4)	2·45 (1)*
Br	0·0247 (2)	0·4603 (1)	0·2391 (1)	4·09 (4)*
C(1)	0·323 (2)	0·223 (1)	0·184 (1)	4·5 (4)
C(2)	0·293 (2)	0·163 (1)	0·247 (1)	3·9 (4)
C(3)	0·307 (2)	0·214 (1)	0·319 (1)	3·4 (4)
C(4)	0·389 (2)	0·384 (1)	0·343 (1)	5·1 (5)
C(5)	0·439 (2)	0·463 (1)	0·305 (1)	3·8 (4)
C(6)	0·445 (2)	0·465 (1)	0·222 (1)	4·9 (4)
C(7)	0·408 (2)	0·394 (1)	0·174 (1)	3·0 (4)
C(8)	0·366 (2)	0·312 (1)	0·212 (1)	3·9 (4)
C(9)	0·342 (2)	0·306 (1)	0·294 (1)	2·9 (4)
C(1')	−0·232 (2)	0·278 (1)	0·331 (1)	3·6 (4)
C(2')	−0·182 (2)	0·184 (1)	0·329 (1)	4·0 (4)
C(3')	−0·052 (2)	0·175 (1)	0·374 (1)	4·4 (4)
C(4')	0·090 (2)	0·289 (1)	0·473 (1)	4·7 (5)
C(5')	0·079 (2)	0·376 (1)	0·503 (1)	5·4 (5)
C(6')	−0·028 (2)	0·439 (2)	0·475 (1)	5·2 (5)
C(7')	−0·138 (2)	0·414 (1)	0·419 (1)	5·1 (5)
C(8')	−0·139 (2)	0·323 (1)	0·386 (1)	3·8 (4)
C(9')	−0·020 (2)	0·261 (1)	0·415 (1)	3·5 (4)
C(1'')	0·012 (2)	0·180 (1)	0·104 (1)	3·2 (4)
C(2'')	−0·127 (2)	0·163 (1)	0·143 (1)	3·4 (4)
C(3'')	−0·217 (2)	0·243 (1)	0·146 (1)	3·0 (4)
C(4'')	−0·170 (2)	0·401 (1)	0·076 (1)	4·0 (4)
C(5'')	−0·073 (2)	0·444 (1)	0·028 (1)	4·6 (5)
C(6'')	0·081 (2)	0·411 (2)	0·008 (1)	5·7 (5)
C(7'')	0·121 (2)	0·327 (1)	0·028 (1)	4·1 (4)
C(8'')	0·015 (2)	0·269 (1)	0·077 (1)	4·5 (4)
C(9'')	−0·127 (2)	0·314 (1)	0·100 (1)	3·5 (4)

* $B_{eq} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab\cos\gamma B(1,2) + ac\cos\beta B(1,3) + bc\cos\alpha B(2,3)]$.

Table 2. *Selected bond distances* (Å)

E.s.d.'s are given in parentheses.

U–Br	2·747 (2)	U–C(3')	2·71 (2)
U–C(1)	2·73 (3)	U–C(8')	2·91 (3)
U–C(2)	2·70 (2)	U–C(9')	2·90 (2)
U–C(3)	2·76 (2)	U–C(1'')	2·69 (2)
U–C(8)	2·91 (3)	U–C(2'')	2·70 (2)
U–C(9)	2·78 (2)	U–C(3'')	2·74 (2)
U–C(1')	2·72 (2)	U–C(8'')	2·78 (2)
U–C(2')	2·68 (2)	U–C(9'')	2·84 (2)

The present structure analysis shows that the title compound is isostructural with the corresponding chloride derivative [U(C_9H_7)$_3$]Cl.

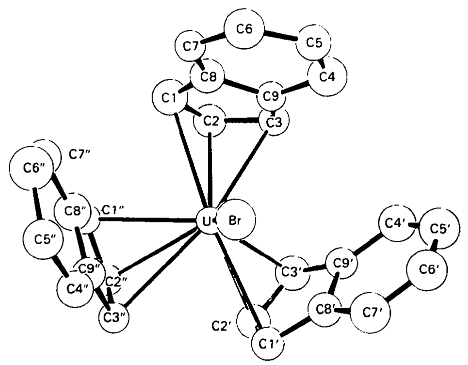

Fig. 1. The molecular structure of [U(C_9H_7)$_3$]Br.

The U–Br bond length of 2·747 (2) Å is comparable to those in [U(C_9H_7)]Br_3.2C_4H_8O and to the U–Br (non-bridging apical Br atom) distance of 2·78 (3) Å in uranium tetrabromide (Taylor & Wilson, 1974).

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Bis(triphenylphosphoranediyl)ammonium Tetrachloronitridomolybdate(IV)

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Abstract. $C_{36}H_{30}NP_2^+.MoNCl_4^-$, $M_r = 790·3$, orthorhombic, $Pna2_1$, $a = 21·635$ (10), $b = 16·922$ (6), $c = 9·709$ (3) Å, $U = 3555$ Å^3, $Z = 4$, $D_x = 1·48$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 8·0$ cm^{-1}, $F(000) = 1600$, $T = 293$ K, $R = 0·050$ for 3643 reflections. The anion is approximately square pyramidal, with Mo 0·6 Å out

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