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Bis[η^5-bis(trimethylsilyl)cyclopentadienyl]bromouranium(III) Bis(*tert*-butyl isocyanide)

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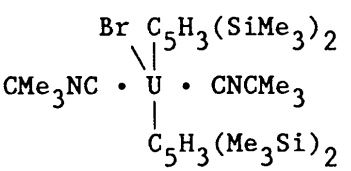
Abstract. $[UBr\{C_5H_3[Si(CH_3)_3]_2\}_2][CNC(CH_3)_3]_2$, $[UBr(C_{11}H_{21}Si_2)_2](C_5H_9N)_2$, $M_r = 903{\cdot}13$, monoclinic, $P2_1/n$, $a = 11{\cdot}765\ (3)$, $b = 12{\cdot}135\ (2)$, $c = 30{\cdot}125\ (5)$ Å, $\beta = 92{\cdot}29\ (2)°$, $V = 4297{\cdot}5$ Å^3, $Z = 4$, $D_x = 1{\cdot}396$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71073$ Å, $\mu = 46{\cdot}3$ cm^{-1}, $F(000) = 1796$, $T = 296$ K, $R = 0{\cdot}033$ for 4281 $F^2 > 2\sigma(F^2)$ of 7592 total unique data. The U atom is five coordinate with distances: U—Br 2·8761 (10); U—Cp 2·514, 2·520; U—C(isocyanide) 2·662 (8), 2·697 (7); ⟨U—C(cyclopentadiene)⟩ 2·791 (18) Å.

Experimental. The title compound, shown below, was



prepared by reaction of one equivalent of $[UBr\{C_5H_3[Si(CH_3)_3]_2\}_2]_2$ with four equivalents of $CN[C(CH_3)_3]$ in diethyl ether solution. The product was dissolved in hexane and dark crystals were isolated after cooling to 253 K. A brown-green air-sensitive crystal, 0·17 × 0·18 × 0·67 mm, was sealed inside a quartz capillary in an argon-filled drybox. X-ray diffraction intensities (θ–2θ scans) were obtained using a modified Picker FACS-I automatic diffractometer equipped with a graphite monochromator. Cell dimensions from 29 reflections, $20 < 2\theta < 30°$; analytical absorption correction, range 1·96–2·37; max. $(\sin\theta)/\lambda = 0{\cdot}60$ Å^{-1}, h −14 to 14, k 0 to 14, l −35 to 35; three standard reflections, 1·5%, 1·4%, 2·5% variation in standards' intensities from average, intensities adjusted isotropically; 14455 data, 7592 unique [including 3311, $F^2 < 3\sigma(F^2)$], R_{int}=0·048; structure solved by Patterson and Fourier methods; refined on F, 385 parameters; parameters of the six H atoms of the cyclopentadienyl rings included with isotropic thermal parameters; the methyl H atoms included with estimated positional and isotropic thermal parameters and were not refined; $R = 0{\cdot}094$ (all data), $R = 0{\cdot}033$ [$F^2 > 2\sigma(F^2)$ data], $wR = 0{\cdot}031$, $S = 1{\cdot}0$; $w = 4F^2[\sigma^2(F^2) + (0{\cdot}035F^2)^2]^{-1}$; max. $\Delta/\sigma < 0{\cdot}02$; max. empirical isotropic correction for extinction 16% of F; max., min. of ΔF synthesis 1·21, −1·42 e Å^{-3}; atomic f from *International Tables for X-ray Crystallography* (1974); local unpublished programs and *ORTEP* (Johnson, 1965).

Atomic parameters are listed in Table 1,* and distances and angles are listed in Table 2. Fig. 1 shows the molecule and the numbering scheme.

* Lists of structure factors, anisotropic thermal parameters, calculated H positions, additional distances and angles and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51772 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

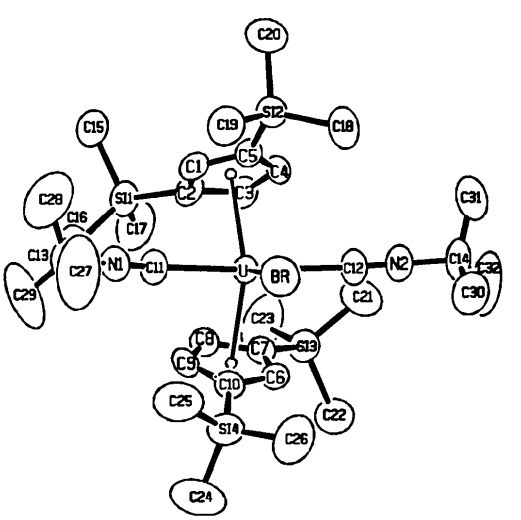


Fig. 1. *ORTEP* drawing showing atomic numbering; 50% probability ellipsoids.

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Table 1. *Atomic parameters*

$B_{eq} = \frac{1}{3}\sum_i\sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$

	x	y	z	B_{eq}(Å^2)
U	0·18863 (2)	0·11824 (2)	0·35897 (1)	3·339 (6)
Br	0·20423 (9)	0·32988 (8)	0·31666 (3)	6·40 (3)
Si1	−0·04814 (17)	−0·12204 (21)	0·39988 (7)	4·88 (6)
Si2	0·02958 (18)	0·10755 (20)	0·23520 (7)	4·59 (6)
Si3	0·44003 (18)	−0·08801 (18)	0·41748 (7)	4·79 (7)
Si4	0·30450 (20)	0·36631 (21)	0·44719 (8)	5·68 (7)
N1	−0·0751 (6)	0·2705 (6)	0·39312 (20)	5·1 (2)
N2	0·4408 (5)	0·1163 (6)	0·28308 (21)	5·5 (2)
C1	−0·0078 (6)	0·0171 (6)	0·32377 (25)	3·9 (2)
C2	0·0352 (6)	−0·0609 (6)	0·35467 (23)	3·9 (2)
C3	0·1379 (6)	−0·0997 (6)	0·33657 (25)	4·1 (2)
C4	0·1548 (6)	−0·0474 (6)	0·29623 (24)	3·7 (2)
C5	0·0640 (6)	0·0292 (6)	0·28696 (22)	3·7 (2)
C6	0·3734 (6)	0·1462 (7)	0·41853 (24)	4·2 (2)
C7	0·3434 (6)	0·0323 (6)	0·42288 (22)	3·9 (2)
C8	0·2362 (7)	0·0345 (7)	0·44315 (24)	4·0 (2)
C9	0·2049 (6)	0·1444 (6)	0·45058 (22)	4·2 (2)
C10	0·2899 (6)	0·2171 (6)	0·43543 (22)	3·9 (2)
C11	0·0051 (7)	0·2264 (6)	0·38401 (25)	4·7 (2)
C12	0·3708 (6)	0·1122 (8)	0·30743 (25)	5·4 (2)
C13	−0·1774 (8)	0·3305 (9)	0·4045 (3)	6·8 (3)
C14	0·5317 (7)	0·1299 (7)	0·25126 (29)	5·7 (3)
C15	−0·1787 (7)	−0·1862 (8)	0·37371 (28)	6·8 (3)
C16	−0·0956 (8)	−0·0163 (8)	0·44006 (28)	7·0 (3)
C17	0·0324 (8)	−0·2327 (8)	0·4300 (3)	8·1 (3)
C18	0·1632 (7)	0·1462 (7)	0·20699 (26)	6·3 (3)
C19	−0·0592 (7)	0·2296 (7)	0·24854 (27)	6·5 (3)
C20	−0·0574 (8)	0·0197 (8)	0·19556 (27)	7·3 (3)
C21	0·4795 (8)	−0·1160 (8)	0·35979 (28)	7·6 (3)
C22	0·5758 (7)	−0·0600 (9)	0·4497 (3)	7·8 (3)
C23	0·3701 (9)	−0·2108 (8)	0·4393 (4)	10·1 (4)
C24	0·3462 (10)	0·3811 (9)	0·5074 (3)	10·0 (4)
C25	0·1702 (9)	0·4444 (8)	0·4381 (4)	8·9 (4)
C26	0·4186 (9)	0·4271 (8)	0·4141 (4)	10·1 (4)
C27	−0·1685 (12)	0·4458 (12)	0·3910 (5)	14·2 (7)
C28	−0·2720 (11)	0·2769 (14)	0·3779 (5)	15·3 (7)
C29	−0·1970 (12)	0·3198 (13)	0·4513 (4)	14·2 (6)
C30	0·5678 (8)	0·2492 (8)	0·2525 (3)	7·7 (4)
C31	0·4829 (9)	0·0971 (10)	0·2059 (3)	10·2 (4)
C32	0·6292 (9)	0·0546 (9)	0·2667 (4)	10·4 (5)
H1	−0·079 (5)	0·055 (5)	0·3259 (20)	4·2 (16)†
H2	0·183 (5)	−0·157 (5)	0·3478 (19)	3·9 (16)†
H3	0·208 (5)	−0·059 (5)	0·2780 (18)	2·5 (13)†
H4	0·440 (5)	0·171 (5)	0·4059 (19)	3·6 (15)†
H5	0·202 (5)	−0·029 (5)	0·4498 (19)	2·8 (15)†
H6	0·134 (5)	0·166 (5)	0·4625 (18)	3·0 (13)†

† B_{iso}.

Table 2. *Selected distances* (Å) *and angles* (°)

Cp1–U	2·520	C5–U	2·787 (7)
Cp2–U	2·514	C6–U	2·784 (7)
Br–U	2·8761 (10)	C7–U	2·798 (7)
C11–U	2·662 (8)	C8–U	2·769 (7)
C12–U	1·697 (7)	C9–U	2·777 (7)
C1–U	2·788 (7)	C10–U	2·819 (7)
C2–U	2·825 (7)	C11–N1	1·128 (8)
C3–U	2·789 (7)	C12–N2	1·126 (8)
C4–U	2·777 (7)		

Br–U–C11	75·65 (17)	C12–U–Cp1	86·97
Br–U–C12	72·73 (20)	C12–U–Cp2	99·22
C11–U–C12	148·09 (25)	Cp1–U–Cp2	129·08
Br–U–Cp1	118·73	C11–N1–C13	178·4 (9)
Br–U–Cp2	112·16	C12–N2–C14	176·2 (10)
C11–U–Cp1	94·41	U–C11–N1	177·1 (7)
C11–U–Cp2	96·44	U–C12–N2	173·1 (7)

Cp1 and Cp2 represent the centers of cyclopentadienyl rings C1–C5 and C6–C10 respectively.

Related literature. The U^{III} dimer, $[UBr\{C_5H_3[Si(CH_3)_3]_2\}_2]_2$, has been synthesized and structurally characterized (Blake, Lappert, Taylor, Atwood & Zhang, 1987). The ability of isocyanides to act as good donor ligands toward uranium metal centers has been previously observed (Kanellakopulos, Fischer, Dornberger & Baumgartner, 1970).

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Structure of $[Ag(PPh_3)_4]PF_6$

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Abstract. Tetrakis(triphenylphosphine)silver hexafluorophosphate, $[Ag\{P(C_6H_5)_3\}_4]PF_6$, $M_r = 1302{\cdot}01$, trigonal, $R\bar{3}$, $a = 14{\cdot}330\ (6)$, $b = 14{\cdot}330\ (6)$, $c = 51{\cdot}57\ (1)$ Å, $V = 9171\ (5)$ Å^3, $Z = 6$, $D_x = 1{\cdot}414$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 5{\cdot}139$ cm^{-1}, $F(000) = 4008$, $T = 292$ K, $R = 0{\cdot}0401$

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