

Table 2. Bond lengths (Å), angles (°) and torsion angles (°) with *e.s.d.*'s

Pt—P	2.212 (3)	P—C(1B)	1.784 (12)
Pt—Cl(1)	2.318 (3)	P—C(1C)	1.806 (12)
Pt—Cl(2)	2.282 (3)	C(1A)—C(2A)	1.534 (15)
Pt—Cl(1')	2.431 (3)	C(1B)—C(2B)	1.530 (17)
P—C(1A)	1.813 (9)	C(1C)—C(2C)	1.467 (17)
P—Pt—Cl(1)	95.93 (9)	Pt—P—C(1C)	112.5 (4)
P—Pt—Cl(2)	89.43 (10)	C(1A)—P—C(1B)	108.0 (5)
P—Pt—Cl(1')	179.07 (9)	C(1A)—P—C(1C)	105.7 (5)
Cl(1)—Pt—Cl(2)	174.59 (9)	C(1B)—P—C(1C)	105.5 (5)
Cl(1)—Pt—Cl(1')	83.52 (9)	Pt—Cl(1)—Pt'	96.48 (10)
Cl(2)—Pt—Cl(1')	91.12 (9)	P—C(1A)—C(2A)	115.7 (7)
Pt—P—C(1A)	112.5 (3)	P—C(1B)—C(2B)	115.5 (8)
Pt—P—C(1B)	112.2 (4)	P—C(1C)—C(2C)	113.9 (9)
Cl(1)—Pt—P—C(1A)	-1.7 (3)	Pt—P—C(1A)—C(2A)	176.6 (6)
Cl(1)—Pt—P—C(1B)	120.2 (4)	C(1B)—P—C(1A)—C(2A)	52.2 (8)
Cl(1)—Pt—P—C(1C)	-121.0 (4)	C(1C)—P—C(1A)—C(2A)	-60.3 (8)
Cl(2)—Pt—P—C(1A)	179.0 (3)	Pt—P—C(1B)—C(2B)	-58.6 (9)
Cl(2)—Pt—P—C(1B)	-59.1 (4)	C(1A)—P—C(1B)—C(2B)	66.0 (9)
Cl(2)—Pt—P—C(1C)	59.8 (4)	C(1C)—P—C(1B)—C(2B)	178.6 (8)
P—Pt—Cl(1)—Pt'	179.24 (9)	Pt—P—C(1C)—C(2C)	47.6 (10)
Cl(2)—Pt—Cl(1)—Pt'	-8.5 (10)	C(1A)—P—C(1C)—C(2C)	-75.5 (9)
Cl(2)—Pt—Cl(1')—Pt'	179.21 (10)	C(1B)—P—C(1C)—C(2C)	170.3 (9)

Related literature. The heavy-atom (Pt,Cl,P) skeletons of the corresponding complexes with tri-*n*-propylphosphine (Black, Mais & Owston, 1969) and di-*tert*-butylcyclopropylphosphine (Simms, Shang, Jiayi Lu, Youngs & Ibers, 1987) exhibit similar geometries.

We thank the University of Edinburgh for a Studentship (to AMM) and the SERC for funding towards the purchase of a four-circle diffractometer.

Acta Cryst. (1989). **C45**, 1219–1221

Bis[η^5 -bis(trimethylsilyl)cyclopentadienyl]chlorouranium(III) Bis(trimethylsilyl cyanide)

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(Received 9 November 1988; accepted 31 January 1989)

Abstract. [UCl{C₅H₃[Si(CH₃)₃]₂}]₂[NCSi(CH₃)₃]₂, [UCl(C₁₁H₂₁Si₂)(C₄H₉NSi)₂], *M_r* = 890.83, monoclinic, *P*2₁/*n*, *a* = 11.602 (2), *b* = 36.947 (5), *c* = 10.999 (2) Å, β = 99.31 (2)°, *V* = 4652.7 Å³, *Z* = 4, *D_x* = 1.272 g cm⁻³, λ (Cu *K*α) = 1.54180 Å, μ = 127.6 cm⁻¹, *F*(000) = 1788, *T* = 296 K, *R* = 0.041 for 4138 *F*² > 2σ(*F*²) of 6929 total unique data. The U atom is coordinated to five ligands with distances: U—Cl 2.698 (3); U—Cp 2.510, 2.520; U—N(cyanide) 2.592 (11), 2.619 (10); ⟨U—C(cyclopentadiene)⟩ 2.789 (22) Å.

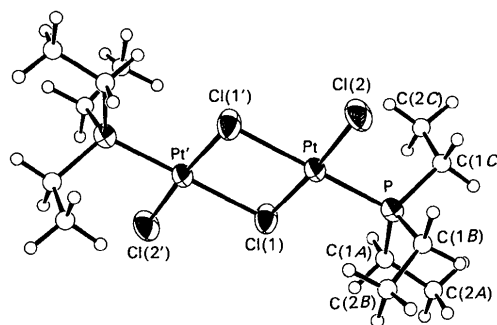
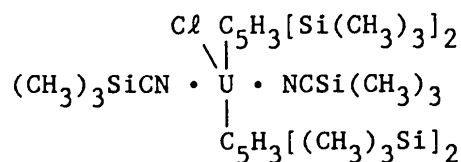


Fig. 1. View of the molecule showing atom numbering scheme. Thermal ellipsoids are drawn at the 30% probability level, excepting those of C and H which have artificial radii of 0.15 and 0.10 Å, respectively, for clarity.

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Experimental. The title compound, shown below, was



prepared by reaction of one equivalent of [UCl{C₅H₃[Si(CH₃)₃]₂}]₂ with four equivalents of NCSi(CH₃)₃ in diethyl ether solution. The product was

Table 1. Atomic parameters

$$B_{eq} = \frac{1}{3} \sum_i \sum_j B_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	B _{eq} (Å ²)
U	0.48183 (3)	0.12269 (1)	0.13262 (3)	4.979 (9)
Cl	0.4154 (3)	0.18629 (10)	0.0225 (4)	9.9 (1)
Si1	0.3471 (3)	0.02559 (9)	0.2582 (4)	7.8 (1)
Si2	0.2625 (3)	0.17889 (11)	0.3217 (4)	8.4 (1)
Si3	0.81667 (27)	0.07811 (11)	0.2593 (3)	7.6 (1)
Si4	0.5918 (3)	0.13708 (14)	-0.2026 (4)	9.3 (1)
Si5	0.0850 (4)	0.09335 (13)	-0.2196 (5)	11.0 (2)
Si6	0.7633 (3)	0.23424 (10)	0.3583 (4)	8.2 (1)
N1	0.2985 (9)	0.1051 (4)	-0.0281 (10)	8.6 (4)
N2	0.6267 (9)	0.1699 (3)	0.2447 (11)	8.5 (4)
C1	0.3008 (9)	0.1033 (4)	0.2558 (11)	6.4 (3)
C2	0.3783 (9)	0.0740 (3)	0.2820 (10)	6.0 (3)
C3	0.4764 (10)	0.0899 (3)	0.3607 (10)	6.3 (3)
C4	0.4566 (10)	0.1261 (3)	0.3785 (10)	6.8 (3)
C5	0.3439 (10)	0.1354 (3)	0.3131 (11)	6.6 (3)
C6	0.6954 (8)	0.1160 (3)	0.0485 (12)	6.6 (3)
C7	0.6978 (9)	0.0876 (3)	0.1328 (10)	5.8 (3)
C8	0.6073 (10)	0.0628 (3)	0.0778 (13)	6.9 (4)
C9	0.5537 (10)	0.0765 (4)	-0.0392 (12)	6.9 (4)
C10	0.6062 (10)	0.1098 (4)	-0.0597 (11)	6.6 (4)
C11	0.2173 (12)	0.1004 (4)	-0.0999 (13)	8.3 (4)
C12	0.6798 (11)	0.1948 (4)	0.2875 (14)	8.0 (4)
C13	0.4795 (14)	-0.0020 (4)	0.3021 (17)	10.0 (5)
C14	0.2417 (17)	0.0136 (5)	0.3646 (20)	12.5 (7)
C15	0.2826 (16)	0.0153 (5)	0.0954 (15)	11.1 (6)
C16	0.1574 (15)	0.1849 (5)	0.1804 (18)	11.9 (7)
C17	0.1795 (16)	0.1764 (7)	0.4547 (16)	13.7 (8)
C18	0.3632 (17)	0.2171 (4)	0.3490 (20)	11.6 (7)
C19	0.9473 (27)	0.0814 (9)	0.205 (3)	17.8 (8)†
C20	0.806 (4)	0.0310 (14)	0.318 (5)	24.5 (15)†
C21	0.820 (3)	0.1082 (11)	0.389 (4)	21.1 (11)†
C22	0.6716 (15)	0.1130 (5)	-0.3137 (17)	10.6 (4)†
C23	0.6614 (18)	0.1832 (6)	-0.1672 (20)	12.6 (5)†
C24	0.4395 (17)	0.1438 (6)	-0.2800 (20)	12.4 (5)†
C25	-0.0048 (22)	0.0616 (7)	-0.1593 (25)	15.2 (7)†
C26	0.0264 (20)	0.1384 (7)	-0.2597 (23)	14.1 (6)†
C27	0.1542 (22)	0.0749 (7)	-0.3622 (27)	15.8 (7)†
C28	0.8845 (20)	0.2404 (7)	0.2755 (22)	14.3 (6)†
C29	0.8130 (22)	0.2199 (7)	0.5120 (26)	15.6 (7)†
C30	0.6635 (17)	0.2720 (5)	0.3288 (19)	12.0 (5)†

† Isotropically refined thermal parameters.

dissolved in hexane and dark-green crystals were isolated after cooling at 253 K. A green, air-sensitive crystal, 0.10 × 0.25 × 0.31 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 18 reflections, $39 < 2\theta < 72^\circ$; analytical absorption correction, range 3.0–14.6; min., max. $\sin\theta/\lambda = 0.20, 0.56 \text{ \AA}^{-1}$ (all 288 data with $\sin\theta/\lambda < 0.2 \text{ \AA}^{-1}$ given zero weight because of large residuals due to an inadequate absorption correction), $h-13$ to 13, $k 0$ to 41, $l-10$ to 12; three standard reflections, 4.3%, 2.9%, 2.0% variation in standards' intensities from average, intensities adjusted isotropically; 13 527 data, 6929 unique [including 2503, $F^2 < 2\sigma(F^2)$], $R_{int} = 0.050$; structure solved by Patterson and Fourier methods; refined on F , 301 parameters; anisotropic thermal parameters on U, Cl, Si, N and on 18 of the 30 C atoms; parameters of H atoms included with estimated positional and isotropic thermal parameters and were not refined; $R = 0.078$ (all data), $R = 0.041$ [$F^2 > 2\sigma(F^2)$ and $\sin\theta/\lambda < 0.2 \text{ \AA}^{-1}$ data], $wR = 0.048$, $S = 1.0$; $w = 4F^2[\sigma^2(F^2) + (0.06F^2)^2]^{-1}$; max. $\Delta/\sigma < 0.02$; max. empirical isotropic correction for extinction

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

Cp1-U	2.520	C4-U	2.770 (11)
Cp2-U	2.510	C5-U	2.783 (11)
Cl-U	2.698 (3)	C6-U	2.792 (10)
N1-U	2.618 (11)	C7-U	2.821 (10)
N2-U	2.592 (12)	C8-U	2.769 (10)
C1-U	2.771 (11)	C9-U	2.772 (11)
C2-U	2.832 (11)	C10-U	2.788 (12)
C3-U	2.795 (11)		
Cl-U-Cp1	116.98	Cp1-U-Cp2	130.48
Cl-U-Cp2	112.52	Cl-U-N1	76.79 (29)
N1-U-Cp1	94.87	Cl-U-N2	75.15 (28)
N1-U-Cp2	97.52	N1-U-N2	151.9 (4)
N2-U-Cp1	96.08	U-N1-C11	174.4 (4)
N2-U-Cp2	94.88	U-N2-C12	170.0 (4)

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

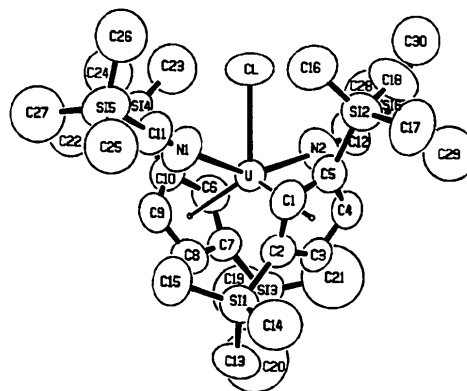


Fig. 1. ORTEP drawing showing atomic numbering; 50% probability ellipsoids.

9% of F ; max., min. of ΔF synthesis 1.5, $-1.0 e \text{ \AA}^{-3}$; 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.

Related literature. The U^{III} dimer, [UCl{C₅H₃[Si(CH₃)₃]₂}]₂, has been synthesized and characterized (Blake, Lappert, Taylor, Atwood, Hunter & Zhang, 1986).

Helpful discussions with Professor R. A. Andersen are appreciated. This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Chemical Sciences Division of the US Department of Energy, under contract No. DE-AC03-76SF-F00098.

*Lists of structure factors, anisotropic thermal parameters, calculated H positions, additional distances and angles, and least-squares planes and an additional view of the molecule have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51771 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

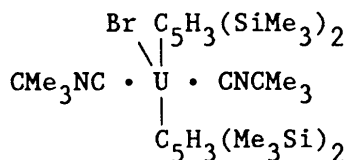
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(Received 3 November 1988; accepted 3 February 1989)

Abstract. [UBr{C₅H₃[Si(CH₃)₃]₂}]₂[CNC(CH₃)₃]₂, [UBr(C₁₁H₂₁Si₂)](C₅H₉N)₂, *M_r* = 903.13, monoclinic, *P*2₁/*n*, *a* = 11.765 (3), *b* = 12.135 (2), *c* = 30.125 (5) Å, β = 92.29 (2)°, *V* = 4297.5 Å³, *Z* = 4, *D_x* = 1.396 g cm⁻³, λ (Mo *K*α) = 0.71073 Å, μ = 46.3 cm⁻¹, *F*(000) = 1796, *T* = 296 K, *R* = 0.033 for 4281 *F*² > 2σ(*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{C₅H₃-[Si(CH₃)₃]₂}]₂ with four equivalents of CN[C(CH₃)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 -2 θ scans) were obtained using a modified Picker FACS-I automatic diffractometer equipped with a graphite monochromator. Cell dimensions from 29 reflections, 20 < 2 θ < 30°; analytical absorption correction, range 1.96–2.37; max. (sin θ)/ λ = 0.60 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 < 3σ(*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* = 0.094 (all data), *R* = 0.033 [*F*² > 2σ(*F*²) data], *wR* = 0.031, *S* = 1.0; *w* = 4*F*²[σ²(*F*²) + (0.035*F*²)²]⁻¹; max. Δ/σ < 0.02; 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.

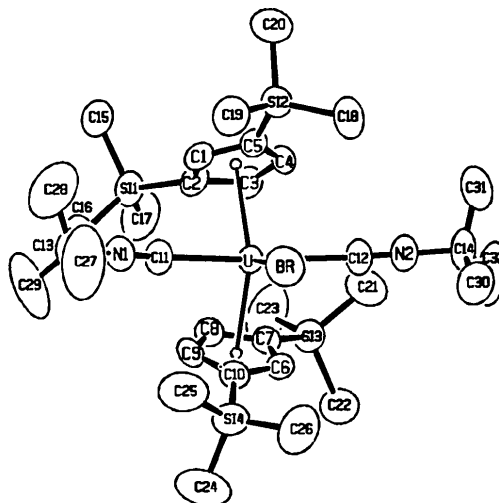


Fig. 1. ORTEP drawing showing atomic numbering; 50% probability ellipsoids.