Table 1. *Positional and equivalent isotropic thermal parameters for the non-H atoms and their e.s.d.'s*

$$B_{eq} = (8\pi^2/3)(U_{11} + U_{22} + U_{33}).$$

	x	y	z	$B_{eq}(\text{Å}^2)$
Os	0	0	$\frac{1}{4}$	1·34 (2)
Br	0	0	0·2704 (1)	2·56 (4)
S	−0·2536 (2)	0·0363 (2)	$\frac{1}{4}$	2·16 (8)
O	−0·3385 (6)	−0·1017 (7)	$\frac{1}{4}$	3·8 (3)
C	−0·3242 (8)	0·1395 (9)	0·3773 (5)	3·3 (3)

Table 2. *Selected bond distances (Å), bond angles (°) and their e.s.d.'s*

Os—Br	2·555 (1)	S—O	1·487 (6)
Os—S	2·351 (2)	S—C	1·784 (6)
O—S—C	105·2 (3)	C—S—C′	99·8 (5)
Os—S—O	113·5 (2)	Os—S—C	115·8 (2)

Fig. 2. Stereoscopic illustration of the molecular packing.

Fig. 1 illustrates the molecular configuration, thermal motion and atom-numbering scheme, while Fig. 2 displays the molecular packing. All computer programs from the *TEXSAN* crystal structure analysis package (Molecular Structure Corporation, 1985).

Related literature. Although Os(Me₂SO) compounds have been previously reported (Antonov, Kukushkin, Konnov & Ionin, 1978) the title compound is apparently the first to be structurally characterized. It is isomorphous with [RuBr₂(Me₂SO)₄] (Oliver & Riley, 1984).

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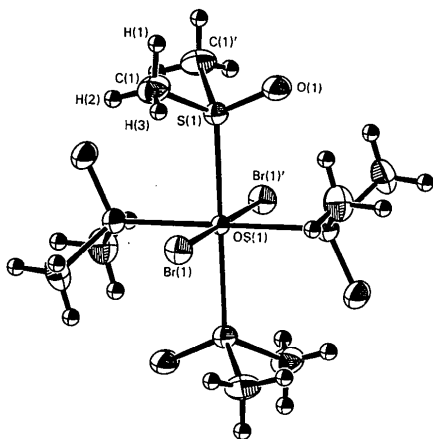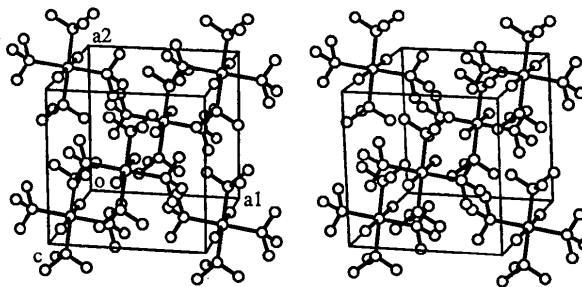OLIVER, J. D. & RILEY, D. P. (1984). *Inorg. Chem.* **23**, 156–158.

Fig. 1. Molecular structure and numbering scheme, thermal ellipsoids at the 50% probability level. H atoms shown with isotropic *B*'s of 1·0 Å². Primed and unprimed atoms are related by a plane of symmetry.

Bis[bis(trimethylsilyl)cyclopentadienyl](chloro)uranium(III) Bis(2,6-dimethylphenylisocyanide)

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Abstract. [U(C₁₁H₂₁Si₂)₂Cl](C₉H₉N)₂, $M_r = 954·77$, monoclinic, $P2_1/n$, $a = 16·000$ (4), $b = 22·806$ (4), $c = 13·093$ (3) Å, $\beta = 92·85$ (2)°, $V = 4771·7$ Å⁻³, $Z = 4$, $D_x = 1·33$ g cm⁻³, $\lambda(\text{Mo }K\alpha) = 0·71073$ Å, $\mu = 34·0$ cm⁻¹, $F(000) = 1916$, $T = 296$ K, $R = 0·031$ for 4116 [$F^2 > 2\sigma(F^2)$] of 8437 total unique data. The U atom is five coordinate with distances: U—Cl 2·6865 (20); U—Cp 2·505, 2·508; U—C(isocyanide) 2·654 (9), 2·681 (9); ⟨U—C(cyclopentadiene)⟩ 2·781 (11) Å.

Table 1. *Atomic parameters*

$$B_{eq} = \tfrac{4}{3}\Sigma_i\Sigma_j B_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	B_{eq} (Å²)
U	0·47976 (2)	0·21461 (1)	0·13690 (2)	3·428 (7)
Cl	0·56861 (15)	0·26416 (10)	0·29360 (16)	6·13 (7)
Si1	0·465200 (16)	0·12623 (12)	0·40786 (17)	5·39 (8)
Si2	0·22796 (14)	0·18402 (11)	0·05526 (17)	4·72 (7)
Si3	0·59890 (17)	0·36651 (12)	0·03497 (19)	5·77 (8)
Si4	0·54570 (20)	0·13484 (13)	−0·13756 (21)	7·1 (1)
N1	0·6855 (4)	0·1313 (3)	0·1778 (5)	5·1 (2)
N2	0·3692 (4)	0·3514 (4)	0·2263 (5)	5·1 (2)
C1	0·3437 (5)	0·1708 (4)	0·2369 (5)	4·2 (2)
C2	0·4118 (5)	0·1353 (4)	0·2728 (5)	4·3 (2)
C3	0·4351 (5)	0·1011 (3)	0·1862 (6)	4·2 (2)
C4	0·3807 (5)	0·1154 (3)	0·1016 (5)	4·0 (2)
C5	0·3238 (5)	0·1595 (3)	0·1306 (5)	3·9 (2)
C6	0·5818 (5)	0·2432 (4)	−0·0220 (6)	4·8 (3)
C7	0·5433 (5)	0·2959 (3)	0·0019 (5)	4·4 (3)
C8	0·4573 (4)	0·2892 (4)	−0·0292 (5)	4·1 (2)
C9	0·4458 (5)	0·2330 (4)	−0·0692 (5)	4·4 (2)
C10	0·5226 (5)	0·2030 (3)	−0·0652 (5)	4·4 (2)
C11	0·3966 (7)	0·0627 (5)	0·4661 (7)	7·8 (4)
C12	0·4305 (7)	0·1937 (4)	0·4819 (6)	7·6 (4)
C13	0·5654 (6)	0·1078 (4)	0·4167 (7)	7·2 (3)
C14	0·2267 (5)	0·2627 (4)	0·0230 (7)	6·1 (3)
C15	0·2182 (6)	0·1388 (4)	−0·0644 (7)	6·6 (3)
C16	0·1346 (6)	0·1712 (5)	0·1312 (8)	8·2 (4)
C17	0·6309 (9)	0·4001 (5)	−0·0858 (8)	11·7 (5)
C18	0·6949 (7)	0·3546 (5)	0·1165 (8)	9·2 (4)
C19	0·5317 (8)	0·4178 (5)	0·1007 (11)	12·1 (5)
C20	0·5762 (10)	0·0716 (5)	−0·0637 (9)	13·2 (6)
C21	0·6159 (11)	0·1524 (7)	−0·2363 (12)	16·9 (8)
C22	0·4474 (9)	0·1129 (6)	−0·2132 (10)	13·6 (6)
C23	0·6210 (6)	0·1518 (4)	0·1612 (6)	5·3 (3)
C24	0·3936 (5)	0·3073 (4)	0·1943 (6)	4·7 (3)
C25	0·7665 (6)	0·1083 (5)	0·1964 (6)	5·4 (3)
C26	0·7744 (7)	0·0481 (5)	0·2037 (8)	7·2 (4)
C27	0·8552 (9)	0·0274 (6)	0·2191 (9)	9·4 (5)
C28	0·9213 (8)	0·0640 (8)	0·2292 (11)	10·8 (6)
C29	0·9099 (7)	0·1237 (7)	0·2265 (8)	8·6 (5)
C30	0·8311 (6)	0·1489 (5)	0·2082 (6)	6·5 (4)
C31	0·3439 (6)	0·4049 (4)	0·2669 (8)	5·6 (3)
C32	0·2963 (6)	0·4432 (4)	0·2056 (8)	5·9 (3)
C33	0·2707 (7)	0·4938 (5)	0·2534 (10)	7·5 (4)
C34	0·2920 (9)	0·5064 (6)	0·3514 (11)	9·8 (5)
C35	0·3398 (9)	0·4681 (6)	0·4114 (10)	10·7 (6)
C36	0·3667 (8)	0·4158 (5)	0·3686 (8)	8·7 (4)
C37	0·6997 (7)	0·0078 (5)	0·1920 (10)	10·3 (5)
C38	0·8172 (7)	0·2135 (6)	0·2006 (8)	8·3 (4)
C39	0·2749 (6)	0·4299 (4)	0·0970 (7)	7·1 (3)
C40	0·4198 (10)	0·3723 (6)	0·4304 (8)	13·5 (6)

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

Cp1 and Cp2 are the centroids of atoms C1–C5 and C6–C10 respectively.

Cp1–U	2·508	C5–U	2·792 (7)
Cp2–U	2·505	C6–U	2·785 (8)
C23–U	2·681 (9)	C7–U	2·788 (7)
C24–U	2·654 (9)	C8–U	2·771 (7)
Cl–U	2·6865 (20)	C9–U	2·759 (7)
C1–U	2·781 (7)	C10–U	2·779 (7)
C2–U	2·795 (7)	C23–N1	1·144 (9)
C3–U	2·771 (7)	C24–N2	1·164 (10)
C4–U	2·788 (7)		

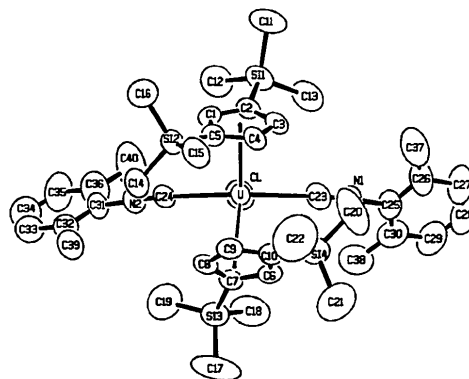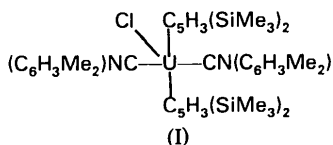|			
Cl–U–Cp1	115·30	Cp1–U–Cp2	129·40
Cl–U–Cp2	115·30	Cl–U–C23	73·95 (19)
C23–U–Cp1	97·96	Cl–U–C24	73·22 (18)
C23–U–Cp2	95·35	C23–N1–C25	177·4 (10)
C24–U–Cp1	98·33	C24–N2–C31	177·1 (9)
C24–U–Cp2	96·12		

Fig. 1. *ORTEP* (Johnson, 1965) drawing showing thermal ellipsoids at the 50% probability level.

Experimental. The title compound (I) was prepared by reaction of one equivalent of $[U\{C_5H_3(SiMe_3)_2\}_2Cl]_2$ with four equivalents of $CN(C_6H_3Me_2)$ in diethyl ether solution. The product was dissolved in hexane, and dark crystals were isolated after cooling at 253 K.

Cl   C₅H₃(SiMe₃)₂
(C₆H₃Me₂)NC—U—CN(C₆H₃Me₂)
C₅H₃(SiMe₃)₂

(I)

A black, air-sensitive crystal, $0.18 \times 0.36 \times 0.72$ 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 28 reflections, $20 < 2\theta < 27°$; analytical absorption correction, range 1.78–3.09; max. $(\sin\theta)/\lambda = 0.60$ Å^{-1}, h −19 to 19, k 0

to 27, l −15 to 15; three standard reflections, 1·1, 1·6, 0·8% variation in standards intensities from average, intensities adjusted isotropically; 12 995 data, 8441 unique [including 4116 with $F^2 < 3\sigma(F^2)$], $R_{int} = 0.075$; structure solved by Patterson and Fourier methods; refined on F, 433 parameters; H atoms included with estimated positional and isotropic thermal parameters; $R = 0.12$ (all data), $R = 0.031$ [$F^2 > 3\sigma(F^2)$ data], $wR = 0.031$, $S = 1.0$; $w = 4F^2[\sigma^2(F^2) + (0.035F^2)^2]^{-1}$; max. (shift/$\sigma$) < 0.001; no extinction correction indicated; max., min. of ΔF synthesis 1·0, −1·8 e Å^{-3}; atomic f values 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.

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* Lists of structure factors, anisotropic thermal parameters, calculated hydrogen positions, distances and angles, and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51740 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The U^{III} dimer, $[U\{C_5H_3(SiMe_3)_2\}_2$-$Cl]_2$, has previously been prepared and structurally characterized (Blake, Lappert, Taylor, Atwood, Hunter & Zhang, 1986). 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 (4R,5R)-4-(D-arabino-1,2,3,4-Tetraacetoxybutyl)-1,2-dimethyl-5-nitro-1-cyclohexene

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Abstract. $C_{20}H_{29}NO_{10}$, $M_r = 443 \cdot 45$, triclinic, $P1$, $a = 7 \cdot 646$ (2), $b = 10 \cdot 085$ (3), $c = 15 \cdot 490$ (4) Å, $\alpha = 78 \cdot 73$ (1), $\beta = 88 \cdot 31$ (1), $\gamma = 80 \cdot 70$ (1)°, $V = 1156$ (1) Å^3, $Z = 2$, $D_m = 1 \cdot 242$ (6), $D_x = 1 \cdot 274$ (1) Mg m^{-3}, λ(Mo $K\alpha$) $= 0 \cdot 71069$ Å, $\mu = 0 \cdot 11$ mm^{-1}, $F(000) = 472$, $T = 297$ K, $R = 0 \cdot 056$ for 3306 unique observed reflections. The structure consists of two $(CH_3)_2C_6H_6(NO_2)(CHOCOCH_3)_3CH_2$-$OCOCH_3$ molecules, which are almost identical in their bond distances and angles but different in the conformation of their cyclohexene rings: $\Delta C_2(C1A,C2A) = 7 \cdot 9$ and $\Delta C_2(C1B,C2B) = 2 \cdot 4°$ [Duax, Weeks & Rohrer (1976). *Top. Stereochem.* **9**, 271–273].

Experimental. The compound was prepared by Diels–Alder addition of 2,3-dimethyl-1,3-butadiene and *trans*-3,4,5,6-tetra-*O*-acetyl-1-nitro-D-*arabino*-1-hexene-3,4,-5,6-tetrol. The crystals were obtained by recrystallization from ethanol [m.p. 360·8 (3) K] and are optically active ($[\alpha]_D^{20 \cdot 0°C} = 0$ (1·6)° [chloroform, 16·7 g dm^{-3}]) (Pacák, 1987). Colourless crystals were stable in air and to X-rays. Density was determined pycnometrically. A prismatic crystal of dimensions $0 \cdot 2 \times 0 \cdot 3 \times 0 \cdot 5$ mm was used for the measurements with an automatic Stoe four-circle diffractometer with graphite monochromator. The lattice parameters were obtained by least-squares fit of 44 reflections with $10 \leq 2\theta \leq 20°$. Intensity data were collected using ω–2θ scan; absorption ignored; max. $(\sin\theta)/\lambda = 0 \cdot 60$ Å^{-1}; $h - 8 \rightarrow 0$, $k - 11 \rightarrow 11$, $l - 18 \rightarrow 18$; three standard reflections measured after 50 reflections with no significant variation; 4247 measured reflections, 3950 unique reflections, 3306 observed reflections with $I \geq 3\sigma(I)$. The structure was solved by direct methods;