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Acta Cryst. (1988). C**44**, 1826–1827

Di-μ-oxo-bis{bis[bis(trimethylsilyl)cyclopentadienyl]uranium(IV)}

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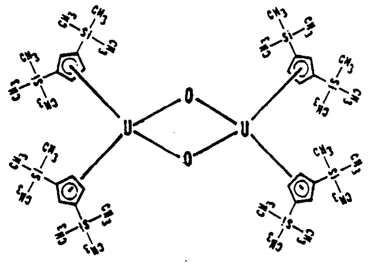
Abstract. $[\{[((CH_3)_3Si)_2C_5H_3]_2UO\}_2]$, $M_r = 1345\cdot91$, monoclinic, $P2_1/n$, $a = 15\cdot648\,(3)$, $b = 15\cdot951\,(3)$, $c = 12\cdot195\,(3)$ Å, $\beta = 99\cdot99\,(2)°$, $V = 2997\cdot7$ Å^3, $Z = 2$, $D_x = 1\cdot49$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0\cdot71073$ Å, $\mu = 53\cdot0$ cm^{-1}, $F(000) = 1320$, $T = 296$ K, $R = 0\cdot036$ for 2997 $[F^2 > 2\sigma(F^2)]$. The dimeric complex, in which two $(Me_3Si)_2C_5H_3U$ moieties are bridge-bonded by two O atoms, lies on a center of symmetry. Distances are: U—O 2·096 (6), 2·129 (6); ⟨U—C⟩ 2·77 (3); ⟨U—Cp⟩ 2·497 (4) Å.

Experimental. The title compound was generated as a side-production reaction of [bis(trimethylsilyl)cyclopentadienyl]uranium(III) complexes with unsaturated molecules, and it is believed to be formed as a result of oxygen contamination.



A green air-sensitive crystal, 0·13 × 0·16 × 0·29 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 23 reflections, $20 < 2\theta < 33°$; analytical absorption correction, range 1·65–2·43; max.$(\sin\theta)/\lambda = 0\cdot60$ Å^{-1}, h −18 to 18, k 0 to 18, l −14 to 14; three standard reflections, 2·4%, 1·2%, 1·0% variation in standards intensities from average, intensities adjusted isotropically; 10 592 data, 5304 unique [including 2307, $F^2 < 2\sigma(F^2)$], $R_{\text{int}} = 0\cdot062$; structure solved by Patterson and Fourier methods; refined on F, 389 parameters; H atoms included with isotropic thermal parameters, methyl H atoms with distance restraints [C—H 1·00 (2), H—H 1·64 (3) Å] and global thermal parameters; $R = 0\cdot093$ (all data), $R = 0\cdot036$ $[F^2 > 2\sigma(F^2)$ data], $wR = 0\cdot034$, $S = 1\cdot03$; $w = 4F^2[\sigma^2(F^2) + (0\cdot035F^2)^2]^{-1}$; max. (shift/$\sigma$) = 0·02; no extinction correction indicated; max., min. of ΔF synthesis 1·6 and −1·1 e Å^{-3}; atomic f for neutral U, O, C and spherical-bonded H from

Table 1. *Atomic parameters*

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

	x	y	z	B_{eq}(Å^2)
U	0·06058 (2)	0·07242 (2)	0·08068 (3)	3·178 (8)
O	0·0534 (3)	−0·0568 (3)	0·0363 (5)	3·7 (2)
Si1	0·30464 (18)	0·00222 (18)	0·02560 (26)	4·59 (9)
Si2	0·05474 (19)	0·26406 (17)	−0·15177 (25)	4·50 (9)
Si3	−0·06890 (25)	0·26147 (21)	0·2376 (3)	6·6 (1)
Si4	0·18098 (20)	0·00519 (19)	0·40943 (24)	4·72 (9)
C1	0·1614 (6)	0·1175 (7)	−0·0718 (9)	4·0 (3)
C2	0·2233 (6)	0·0895 (5)	0·0215 (8)	3·8 (3)
C3	0·2216 (6)	0·1512 (6)	0·1050 (8)	3·7 (3)
C4	0·1609 (6)	0·2138 (6)	0·0609 (9)	4·3 (3)
C5	0·1233 (6)	0·1941 (5)	−0·0501 (8)	3·8 (3)
C6	0·0767 (7)	0·1429 (6)	0·2942 (8)	4·1 (3)
C7	−0·0107 (6)	0·1596 (6)	0·2422 (8)	4·2 (3)
C8	−0·0510 (6)	0·0794 (8)	0·2294 (8)	4·4 (3)
C9	0·0079 (7)	0·0170 (7)	0·2706 (8)	3·9 (3)
C10	0·0907 (6)	0·0553 (5)	0·3121 (7)	3·7 (3)
C11	0·2727 (8)	−0·0936 (7)	0·0968 (11)	6·8 (4)
C12	0·4095 (7)	0·0396 (7)	0·1071 (11)	6·0 (4)
C13	0·3177 (8)	−0·0227 (8)	−0·1186 (11)	6·9 (4)
C14	0·0907 (9)	0·3723 (7)	−0·1193 (11)	7·6 (5)
C15	0·0710 (8)	0·2363 (8)	−0·2940 (10)	6·7 (4)
C16	−0·0639 (7)	0·2548 (8)	−0·1461 (11)	6·6 (4)
C17	−0·1744 (9)	0·2561 (9)	0·1453 (16)	10·9 (7)
C18	−0·0058 (10)	0·3497 (8)	0·1844 (14)	9·8 (6)
C19	−0·0802 (19)	0·2846 (13)	0·3795 (14)	21·2 (14)
C20	0·2892 (7)	0·0471 (7)	0·3902 (10)	6·2 (4)
C21	0·1674 (10)	0·0292 (9)	0·5538 (10)	7·9 (5)
C22	0·1729 (10)	−0·1100 (7)	0·3895 (12)	8·1 (5)

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

O—U	2·096 (6)	C6—U	2·806 (9)
O′—U	2·129 (5)	C9—U	2·736 (9)
Cp1—U	2·499	C7—U	2·795 (9)
Cp2—U	2·494	C8—U	2·729 (9)
C1—U	2·735 (9)	C10—U	2·793 (9)
C2—U	2·778 (8)	U—U′	3·3927 (9)
C3—U	2·784 (9)	O—O′	2·520 (11)
C4—U	2·781 (9)	⟨Si—C⟩	1·857 (21)
C5—U	2·796 (9)		
Cp1—U—Cp2	123·38	O′—U—Cp2	109·40
O—U—Cp1	115·11	O—U—O	73·20 (23)
O—U—Cp2	109·78	U—O—U	106·80 (23)
O′—U—Cp1	115·14		

Cp1 and Cp2 are the centroids of atoms C(1)–C(5), C(6)–C(10) respectively. Primed atoms are at $-x, -y, -z$.

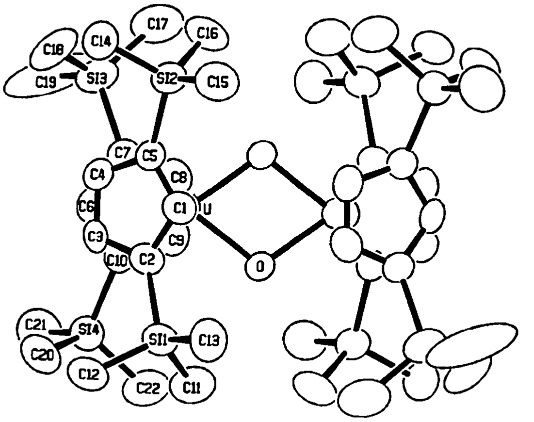

Fig. 1. *ORTEP* drawing of title complex, with thermal ellipsoids at 50% probability level.

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 geometry reported here is in agreement with that of an independent, but sparsely reported, determination (Blake, Lappert, Taylor, Attwood & Zhang, 1987). The Si atoms are from 0·3 to 0·5 Å out of the plane of the cyclopentadienyl rings and away from the U atoms.

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.

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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 51071 (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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Structure of Acetatobromocarbonylbis(triphenylphosphine)osmium(II)

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Abstract. $[OsBr(CO)(CH_3CO_2)\{P(C_6H_5)_3\}_2]$, $M_r = 881{\cdot}74$, monoclinic $P2_1/n$, $a = 9{\cdot}751\ (3)$, $b = 20{\cdot}334\ (4)$, $c = 17{\cdot}458\ (2)$ Å, $\beta = 96{\cdot}42\ (1)°$, $V = 3440\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}70$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 53{\cdot}32$ cm^{-1}, $F(000) = 1728$, $T = 295$ K, $R = 0{\cdot}046$, 3293 unique observed reflections. The compound was prepared by refluxing di(tetra-*n*-butylammonium) hexabromoosmate(IV) and triphenylphosphine in mixed acetic acid/acetic anhydride. The central Os atom is in distorted octahedral coordi-

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