tering factors and structure refinement for both structures: *SHELXTL-Plus* (Sheldrick, 1990), other programs: *PARST* (Nardelli, 1983). Atomic parameters for (I) and (II) are given in Table 1, selected bond lengths and angles in Table 2.* Figs. 1 and 2 show the molecular structures.

Related literature. Only a few other structures of Rh complexes containing one neutral monodentate ligand besides cod and Cl have been reported: $[\{RhCl(cod)\}_2(\mu\text{-}C_4H_4N_2)]$ (Halesha, Reddy, Sudhakar Rao & Manohar, 1983), $[RhCl(cod)P(p\text{-}C_6H_4F)_3]$ (Iglesias, del Pino, Corma, Garcia-Blanco & Martinez Carrera, 1987).

* Lists of structure factors, anisotropic displacement parameters, H-atom parameters and bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54986 (32 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SE0084]

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Structure of Chlorodioxotetrakis(tetrahydrofuran)uranium(VI) Pentachloro(tetrahydrofuran)uranate(IV)

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Abstract. $[UO_2Cl(C_4H_8O)_4][UCl_5(C_4H_8O)]$, M_r = 1081.3, triclinic, $P\bar{1}$, a = 9.272 (2), b = 13.031 (3), c = 15.240 (3) Å, α = 80.04 (3), β = 79.15 (3), γ = 73.46 (3)°, V = 1719.5 (6) Å^3, Z = 2, D_x = 2.088 Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, μ = 9.422 mm^{-1}, $F(000)$ = 1004, T = 293 K, R = 0.0639, wR = 0.0588 for 2544 unique observed [$F > 4.0\sigma(F)$] reflections. $[UO_2Cl(OC_4H_8)_4]^+$ is pentagonal biprismatic with the chloride and the four tetrahydrofuran ligands forming the equatorial plane. The mean U—O distances are 1.72 (1) (uranyl O atoms) and 2.43 (2) Å (thf O atoms) and U—Cl 2.637 (8) Å. Within the octahedral $[UCl_5(OC_4H_8)]^-$ the U—O distance is 2.389 (17) Å and the average U—Cl distance is 2.57 (1) Å.

Experimental. During an attempt to purify a compound which forms on the reaction between UCl_4 and $(CH_3)_3SiN{=}S{=}NSi(CH_3)_3$, green $[UO_2Cl(OC_4H_8)_4]^+.[UCl_5(OC_4H_8)]^-$ crystallized overnight from tetrahydrofuran (thf) at 250 K. A green plate of approximate dimensions 0.3 × 0.4 × 0.6 mm was used for data collection on a Siemens–Stoe AED2 diffractometer, with graphite-monochromated Mo $K\alpha$ radiation and profile-fitted 2θ–ω scans involving variable scan width and speed (Clegg, 1981). Lattice parameters were determined from a least-squares fit of 36 reflections with $20 < 2\theta < 25°$. 4513 reflections were measured in the range 2θ to 45° and $-9 \le h \le 9$, $-13 \le k \le 14$, and $-5 \le l \le 16$. *XEMP* (Sheldrick, 1985) was used to apply a semi-empirical absorption correction to the 4469 unique data in which a minimum to maximum transmission ratio of 0.3824/0.4933 was observed. Three standard reflections measured every 90 min showed about 30% decrease in intensity; an appropriate correction was applied.

The structure was solved with Patterson methods using *SHELXS*86 (Sheldrick, 1985) and 2544 reflections with $F > 4.0\sigma(F)$ were refined with 316 parameters using *SHELX*76 (Sheldrick, 1976); S = 1.6092, R = 0.0639, wR = 0.0588, where $w^{-1} = \sigma^2(F) + 0.0004F^2$. All non-H atoms were refined with anisotropic displacement coefficients and H atoms were included using a riding model with C—H = 0.96 Å and U(H) = 0.08 Å^2. In the final cycle the largest Δ/σ was 0.049 and the maximum and minimum $\Delta\rho$ were

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* ($Å^2 \times 10^3$)

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
U(1)	2551 (1)	1596 (1)	3430 (1)	66 (1)
Cl(1)	−81 (8)	1137 (7)	3568 (5)	114 (4)
O(1)	1158 (16)	2211 (13)	4817 (11)	78 (8)
C(11)	218 (37)	3266 (28)	4849 (19)	137 (20)
C(12)	−778 (38)	3306 (29)	5679 (24)	126 (21)
C(13)	−451 (45)	2275 (40)	6201 (24)	201 (33)
C(14)	605 (40)	1580 (28)	5606 (19)	139 (21)
O(2)	4073 (16)	2396 (13)	4123 (10)	78 (8)
C(21)	4411 (34)	3399 (26)	3831 (21)	131 (19)
C(22)	5328 (48)	3541 (36)	4444 (26)	176 (33)
C(23)	5734 (45)	2562 (40)	5019 (26)	183 (32)
C(24)	4653 (37)	1973 (27)	4956 (19)	141 (20)
O(3)	5029 (18)	1468 (13)	2511 (11)	79 (8)
C(31)	6467 (32)	759 (26)	2747 (21)	130 (18)
C(32)	7568 (36)	817 (33)	1924 (30)	169 (27)
C(33)	6684 (60)	1476 (39)	1256 (25)	208 (38)
C(34)	5284 (28)	2046 (30)	1670 (22)	122 (19)
O(4)	2690 (17)	776 (17)	2083 (11)	83 (8)
C(41)	3178 (34)	−321 (27)	2051 (21)	118 (18)
C(42)	2794 (47)	−560 (28)	1253 (29)	171 (27)
C(43)	2062 (40)	444 (29)	778 (19)	124 (20)
C(44)	1778 (37)	1252 (23)	1378 (19)	118 (17)
O(5)	3334 (17)	367 (12)	4014 (10)	85 (8)
O(6)	1927 (16)	2838 (12)	2842 (9)	79 (7)
U(2)	7098 (1)	6162 (1)	1982 (1)	80 (1)
Cl(2)	6423 (10)	6906 (8)	392 (5)	151 (5)
Cl(3)	7429 (12)	4213 (7)	1693 (7)	156 (6)
Cl(4)	8034 (10)	5464 (8)	3523 (5)	137 (5)
Cl(5)	7259 (11)	8052 (7)	2175 (8)	164 (6)
Cl(6)	4308 (8)	6468 (8)	2693 (5)	137 (5)
O(7)	9696 (19)	5936 (16)	1312 (12)	104 (9)
C(71)	10392 (38)	6649 (29)	736 (25)	174 (22)
C(72)	12016 (40)	6327 (37)	843 (26)	163 (26)
C(73)	12220 (44)	5253 (31)	1323 (35)	184 (30)
C(74)	10832 (37)	5003 (29)	1437 (26)	165 (24)

1.29 and -1.81 e $Å^{-3}$. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic coordinates are given in Table 1, selected bond lengths and angles in Table 2.* Fig. 1 shows a drawing of the ions.

Related literature. The structures of $[UO_2Cl\{PhC(O)N(O)Ph\}].2thf$ (Smith & Raymond, 1979), $[UO_2Cl(O_2CCCl_3)(OPPh_3)_2].CH_2Cl_2$ (Alcock, Flanders, Bagnall & Ahmed, 1986), $[Et_4N]^+.[UO_2Cl(S_2PPh_2)_2]^-$ and $[Et_4N]^+.[UO_2Cl(S_2PMe_2)_2]^-$ (Storey, Zonnevijlle, Pinkerton & Schwarzenbach, 1983), which contain a UO_2Cl unit, and the $UCl_5(L)$ species $[UCl_3(EtCONEt_2)_4]^+.[UCl_5(EtCONEt_2)]^-$ (Bagnall, Beddoes, Mills & Li, 1982) and $[UCl_5(OPPh_3)]$ (Bombieri, Brown & Mealli, 1976), have been reported.

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* Lists of structure factors, anisotropic thermal parameters, H-atom positions, and bond distances and angles, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55162 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0562]

Table 2. *Selected bond lengths* (Å) *and angles* (°)

$[UO_2Cl(thf)_4]^+$		$[UCl_5(thf)]^-$	
U(1)—Cl(1)	2.637 (8)	U(2)—Cl(2)	2.573 (8)
U(1)—O(1)	2.397 (16)	U(2)—Cl(3)	2.574 (10)
U(1)—O(2)	2.445 (19)	U(2)—Cl(4)	2.584 (8)
U(1)—O(3)	2.434 (15)	U(2)—Cl(5)	2.580 (10)
U(1)—O(4)	2.440 (20)	U(2)—Cl(6)	2.556 (7)
U(1)—O(5)	1.724 (14)	U(2)—O(7)	2.389 (17)
U(1)—O(6)	1.709 (13)		
Cl(1)—U(1)—O(1)	75.7 (5)	Cl(2)—U(2)—Cl(3)	91.3 (3)
Cl(1)—U(1)—O(2)	145.5 (4)	Cl(2)—U(2)—Cl(4)	174.2 (3)
Cl(1)—U(1)—O(3)	144.2 (5)	Cl(3)—U(2)—Cl(4)	90.6 (3)
Cl(1)—U(1)—O(4)	74.3 (4)	Cl(2)—U(2)—Cl(5)	89.1 (4)
Cl(1)—U(1)—O(6)	92.9 (6)	Cl(3)—U(2)—Cl(5)	170.2 (3)
Cl(1)—U(1)—O(5)	92.2 (6)	Cl(4)—U(2)—Cl(5)	88.1 (4)
O(1)—U(1)—O(2)	69.9 (5)	Cl(2)—U(2)—Cl(6)	93.0 (3)
O(1)—U(1)—O(3)	140.1 (6)	Cl(3)—U(2)—Cl(6)	95.3 (3)
O(2)—U(1)—O(3)	70.3 (6)	Cl(4)—U(2)—Cl(6)	92.3 (3)
O(1)—U(1)—O(4)	150.0 (6)	Cl(5)—U(2)—Cl(6)	94.4 (3)
O(2)—U(1)—O(4)	140.1 (5)	Cl(2)—U(2)—O(7)	86.5 (5)
O(3)—U(1)—O(4)	69.9 (6)	Cl(3)—U(2)—O(7)	86.5 (6)
O(1)—U(1)—O(5)	89.7 (6)	Cl(4)—U(2)—O(7)	88.1 (5)
O(2)—U(1)—O(5)	88.4 (7)	Cl(5)—U(2)—O(7)	83.8 (6)
O(3)—U(1)—O(5)	87.8 (6)	Cl(6)—U(2)—O(7)	178.1 (5)
O(4)—U(1)—O(5)	92.2 (7)		
O(1)—U(1)—O(6)	91.7 (6)		
O(2)—U(1)—O(6)	87.5 (7)		
O(3)—U(1)—O(6)	88.0 (6)		
O(4)—U(1)—O(6)	89.1 (7)		
O(5)—U(1)—O(6)	174.9 (8)		

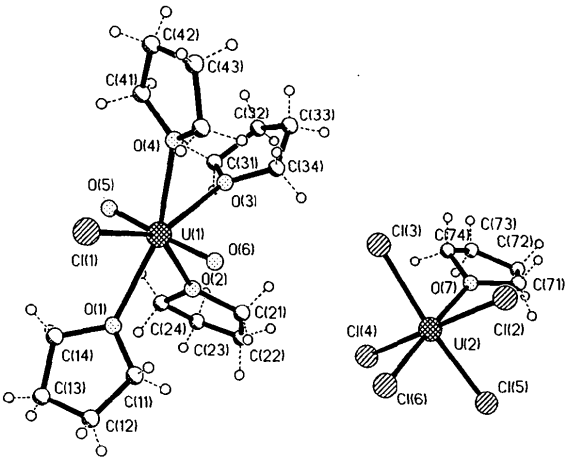

Fig. 1. Structure of $[UO_2Cl(OC_4H_8)_4]^+$ and $[UCl_5(OC_4H_8)]^-$.

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