Table 3. *Non-bonded interionic distances* (Å) *corresponding to probable hydrogen bonds and corresponding angles* (°)

$O(12)\cdots C(8^{i})$	3·42 (2)	$O(31)\cdots C(2^{iii})$	3·47 (2)
$O(21)\cdots C(2^{ii})$	3·39 (2)		
$N(1)-O(12)\cdots C(8^{i})$	108 (1)	$O(12)\cdots C(8^{i})-C(7^{i})$	126 (1)
$N(2)-O(21)\cdots C(2^{ii})$	108 (1)	$O(21)\cdots C(2^{ii})-C(1^{ii})$	120 (1)
$N(3)-O(31)\cdots C(2^{iii})$	115 (1)		

Symmetry code (i) $-x+0{\cdot}5,-y-0{\cdot}5,-z+1$; (ii) $x,y+1,z$; (iii) $-x+1,-y,-z+1$.

oxygen would be 63·5 (five such angles), 116·5 (five such) and 180° (one such). In the nitrate groups (planar within the limits of e.s.d.'s) a number of oxygens exhibit large anisotropic thermal motions. This would explain the differences observed among N—O bond lengths and O—N—O angles.

The tetraethylammonium cation has the expected tetrahedral geometry. The large thermal vibrations of the C atoms are probably responsible for the differences between some interatomic distances and angles (Table 2). The crystal packing is illustrated in Fig. 3. Interionic distances and angles (Table 3) show that the connection between the $[U(NO_3)_6]^{2-}$ anions and the $[N(CH_2CH_3)_4]^{+}$ cations is realized through hydrogen bonds between nitrate oxygens and methyl carbons.

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Octacarbonyl-bis(μ-diphenylphosphido)-dirhenium and Heptacarbonyl-μ-diphenylantimonido-μ-diphenylphosphido-triphenylstibinedirhenium

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Abstract. $[Re_2(CO)_8\{P(C_6H_5)_2\}_2]$, $M_r = 966{\cdot}8$, monoclinic, $P2_1/n$, $a = 12{\cdot}183\ (5)$, $b = 16{\cdot}803\ (6)$, $c = 15{\cdot}707\ (5)$ Å, $\beta = 99{\cdot}99\ (4)°$, $V = 3167\ (2)$ Å^3, $Z = 4$, $D_x = 2{\cdot}026$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 74{\cdot}5$ cm^{-1}, $F(000) = 1824$, $T = 298\ (1)$ K, final $R = 0{\cdot}0407$ for 2895 unique observed reflections. $[Re_2(CO)_7\{P(C_6H_5)_2\}\{Sb(C_6H_5)_2\}\{Sb(C_6H_5)_3\}]$, $M_r = 1382{\cdot}7$, triclinic, $P\bar{1}$, $a = 11{\cdot}093\ (4)$, $b = 13{\cdot}352\ (5)$, $c = 17{\cdot}466\ (6)$ Å, $\alpha = 86{\cdot}50\ (3)$, $\beta = 75{\cdot}55\ (3)$, $\gamma = 67{\cdot}36\ (3)°$, $V = 2310\ (2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}987$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 65{\cdot}1$ cm^{-1}, $F(000) = 1304$, $T = 298\ (1)$ K, $R = 0{\cdot}0626$ for 4979 unique observed reflections. Each compound contains an edge-shared coordination octahedra pair with a nearly planar four-membered ring. The isovalent exchange of one bridging ring member enlarges the subtended bond angle of the remaining μ-P atom by about 7·8 (1)°. The mean value of the Re—(μ-Sb) bond length is 2·740 (1) Å and the terminal Re—Sb bond 2·671 (1) Å.

Introduction. The structures of the diamagnetic title compounds can be related to those of the A_2X_{10} class of molecules (A = metal atom, X = ligand) (Wells, 1984). Each of these dirhenium substances contains an edge-shared coordination octahedra pair with a heteronuclear four-membered $A_2(\mu\text{-}X)_2$ ring without a metal—metal bond. The structural parameters determined for the $Re_2(\mu\text{-}PPh_2)_2$ and $Re_2(\mu\text{-}PPh_2)(\mu\text{-}SbPh_2)$ rings are relevant to the study of the ring-bond-angle dependency on the isovalent exchange of one ring member. Furthermore, the present structural determination has

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continued our investigation into packing forces resulting from geometrical alterations of four-membered rings (Haupt, Preut & Wolfes, 1979).

Experimental. Colorless crystals of $[Re_2(CO)_8(\mu\text{-}PPh_2)_2]$ prepared by heating $[Re_2(CO)_8(\mu\text{-}H)(\mu\text{-}PPh_2)]$ in a solvent mixture of methanol/trichloromethane (2/1) at 393 K in a glass bomb tube (Balsaa, 1987). Crystal size 0·22 × 0·32 × 0·60 mm, Nicolet *R*3*m*/V diffractometer, ω–2θ scan, scan speed 1·5–9·7° min^{-1}, graphite-monochromated Mo $K\alpha$; lattice parameters refined from 25 reflections, $5 \le 2\theta \le 25°$; three standard reflections recorded every 300 reflections: only random deviations, 4398 reflections measured, $2 \le 2\theta \le 45°$, $0 \le h \le 14$, $0 \le k \le 19$, $-17 \le l \le 17$, Lp correction, absorption correction *via* ψ scans, min./max. transmission 0·41/0·48, after merging ($R_{\text{int}} = 0{\cdot}021$) 4162 unique reflections, 1267 reflections considered unobserved, $I < 2\sigma(I)$; structure solution by Patterson method and ΔF synthesis, full-matrix least-squares refinement on F with anisotropic temperature factors for Re and P, 230 parameters refined, phenyl groups treated as rigid bodies (C—C 1·395, C—H 0·96 Å), phenyl H atoms geometrically fixed with $U_{\text{iso}} = 0{\cdot}09$ Å^2, $R = 0{\cdot}0407$, $wR = 0{\cdot}0447$, $w = 1/\sigma^2(F)$, $(\Delta/\sigma)_{\max} = 0{\cdot}001$, max. height in final ΔF map 0·8 e Å^{-3} near heavy-atom position; complex neutral-atom scattering factors corrected for anomalous dispersion (Cromer & Waber, 1974); program *SHELXTL-Plus* (Nicolet, 1987).

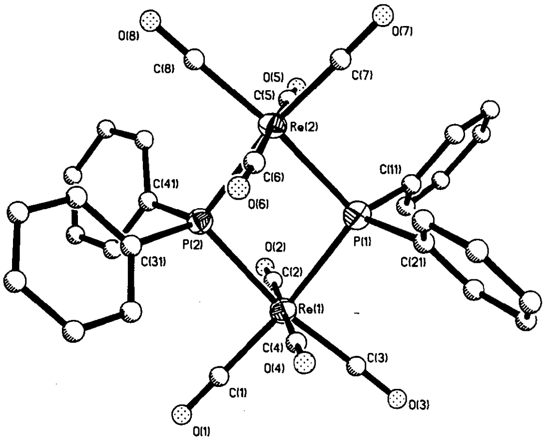

Fig. 1. General view of the molecule $[Re_2(CO)_8(\mu\text{-}PPh_2)_2]$.

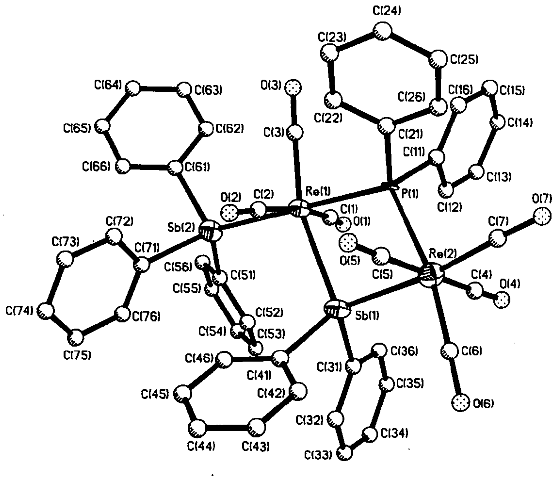

Fig. 2. General view of the molecule $[Re_2(CO)_7(\mu\text{-}PPh_2)(\mu\text{-}SbPh_2)(SbPh_3)]$.

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement parameters* (Å^2 × 10^3) *for* $C_{32}H_{20}O_8P_2Re_2$

	x	y	z	U_{eq}*
Re(1)	2378 (1)	1724 (1)	2192 (1)	29 (1)
Re(2)	360 (1)	3281 (1)	869 (1)	31 (1)
P(1)	2074 (3)	2469 (2)	773 (2)	30 (1)
P(2)	587 (2)	2471 (2)	2233 (2)	28 (1)
C(1)	2516 (12)	1314 (10)	3359 (9)	46 (5)
O(1)	2599 (9)	1077 (7)	4047 (7)	70 (5)
C(2)	1418 (13)	788 (11)	1779 (9)	55 (6)
O(2)	870 (10)	248 (7)	1591 (9)	86 (6)
C(3)	3718 (12)	1162 (8)	1959 (8)	39 (5)
O(3)	4515 (9)	862 (8)	1843 (7)	76 (5)
C(4)	3326 (12)	2679 (11)	2578 (8)	49 (6)
O(4)	3866 (10)	3216 (8)	2795 (7)	78 (5)
C(5)	−404 (10)	2389 (10)	166 (9)	42 (5)
O(5)	−819 (9)	1884 (8)	−228 (7)	75 (5)
C(6)	1176 (12)	4105 (9)	1618 (9)	46 (6)
O(6)	1628 (10)	4569 (7)	2095 (8)	75 (5)
C(7)	417 (11)	3891 (9)	−155 (10)	48 (6)
O(7)	424 (10)	4253 (8)	−788 (7)	80 (5)
C(8)	−1054 (13)	3800 (10)	1045 (10)	54 (6)
O(8)	−1838 (9)	4092 (8)	1137 (8)	86 (5)
C(11)	1913 (7)	1873 (5)	−229 (4)	40 (3)
C(12)	1825 (7)	1046 (5)	−226 (4)	42 (3)
C(13)	1623 (7)	629 (5)	−1006 (4)	62 (4)
C(14)	1509 (7)	1040 (5)	−1788 (4)	62 (4)
C(15)	1596 (7)	1868 (5)	−1791 (4)	58 (4)
C(16)	1798 (7)	2284 (5)	−1011 (4)	52 (4)
C(21)	3332 (5)	3047 (5)	644 (5)	33 (3)
C(22)	4286 (5)	2624 (5)	542 (5)	46 (4)
C(23)	5282 (5)	3028 (5)	521 (5)	55 (4)
C(24)	5323 (5)	3855 (5)	603 (5)	60 (4)
C(25)	4369 (5)	4277 (5)	706 (5)	58 (4)
C(26)	3373 (5)	3873 (5)	726 (5)	43 (4)
C(31)	619 (6)	3117 (5)	3189 (5)	30 (3)
C(32)	−353 (6)	3522 (5)	3283 (5)	38 (3)
C(33)	−352 (6)	4035 (5)	3981 (5)	61 (4)
C(34)	620 (6)	4144 (5)	4584 (5)	59 (4)
C(35)	1592 (6)	3740 (5)	4490 (5)	58 (4)
C(36)	1591 (6)	3226 (5)	3792 (5)	47 (4)
C(41)	−609 (6)	1834 (5)	2377 (5)	37 (3)
C(42)	−1610 (6)	1856 (5)	1791 (5)	42 (3)
C(43)	−2514 (6)	1399 (5)	1939 (5)	59 (4)
C(44)	−2418 (6)	920 (5)	2674 (5)	50 (4)
C(45)	−1417 (6)	898 (5)	3260 (5)	56 (4)
C(46)	−513 (6)	1355 (5)	3112 (5)	47 (4)

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

Colorless crystals of $[Re_2(CO)_7(\mu\text{-}PPh_2)(\mu\text{-}SbPh_2)(SbPh_3)]$ prepared with stoichiometric amounts of $[Re_2(CO)_8(\mu\text{-}H)(\mu\text{-}PPh_2)]$ and $SbPh_3$ in xylene solution at 463–473 K in a glass bomb tube (Haupt & Woyciechowski, 1988). Crystal size 0·12 × 0·24 × 0·38 mm, lattice parameters refined from 30 reflections, $5 \le 2\theta \le 25°$, ω–2θ scan, scan speed 3·5–

Table 2. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement parameters* (Å^2 × 10^3) *for* $C_{49}H_{35}O_7PRe_2Sb_2$

	x	*y*	*z*	U_{eq}*
Re(1)	2252 (1)	2049 (1)	7132 (1)	24 (1)
Re(2)	2112 (1)	4838 (1)	8142 (1)	35 (1)
Sb(1)	3457 (1)	2640 (1)	8125 (1)	31 (1)
Sb(2)	3238 (1)	−118 (1)	7137 (1)	32 (1)
P(1)	1361 (3)	4084 (2)	7117 (2)	16 (1)
C(1)	3769 (14)	1833 (11)	6231 (9)	35 (3)
O(1)	4720 (12)	1613 (9)	5714 (8)	62 (3)
C(2)	958 (14)	2055 (11)	8109 (9)	30 (3)
O(2)	220 (12)	2020 (9)	8718 (7)	59 (3)
C(3)	1129 (15)	2145 (11)	6439 (9)	36 (3)
O(3)	475 (11)	2213 (8)	6005 (7)	49 (3)
C(4)	3615 (18)	4981 (13)	7372 (11)	55 (5)
O(4)	4480 (14)	5124 (11)	6931 (9)	77 (4)
C(5)	616 (16)	4634 (12)	8884 (10)	47 (4)
O(5)	−314 (13)	4515 (10)	9325 (8)	73 (4)
C(6)	2803 (18)	5120 (14)	8951 (11)	55 (5)
O(6)	3301 (15)	5268 (12)	9468 (10)	93 (5)
C(7)	1069 (18)	6306 (14)	8032 (11)	58 (5)
O(7)	403 (13)	7260 (11)	8002 (8)	75 (4)
C(11)	1868 (9)	4544 (7)	6101 (4)	28 (3)
C(12)	3248 (9)	4208 (7)	5761 (4)	45 (4)
C(13)	3705 (9)	4498 (7)	4995 (4)	56 (5)
C(14)	2782 (9)	5125 (7)	4569 (4)	55 (4)
C(15)	1403 (9)	5461 (7)	4909 (4)	54 (4)
C(16)	946 (9)	5170 (7)	5675 (4)	48 (4)
C(21)	−531 (6)	4720 (6)	7338 (6)	31 (3)
C(22)	−1306 (6)	4087 (6)	7523 (6)	40 (4)
C(23)	−2709 (6)	4582 (6)	7717 (6)	56 (5)
C(24)	−3337 (6)	5711 (6)	7726 (6)	55 (4)
C(25)	−2562 (6)	6344 (6)	7541 (6)	52 (4)
C(26)	−1159 (6)	5849 (6)	7347 (6)	45 (4)
C(31)	5603 (7)	2111 (8)	7848 (6)	36 (3)
C(32)	6293 (7)	1682 (8)	8435 (6)	58 (5)
C(33)	7695 (7)	1311 (8)	8251 (6)	70 (5)
C(34)	8408 (7)	1368 (8)	7480 (6)	55 (4)
C(35)	7719 (7)	1796 (8)	6894 (6)	57 (5)
C(36)	6316 (7)	2168 (8)	7077 (6)	50 (4)
C(41)	3151 (11)	2034 (9)	9285 (6)	39 (4)
C(42)	2819 (11)	2697 (9)	9954 (6)	70 (5)
C(43)	2637 (11)	2275 (9)	10706 (6)	92 (7)
C(44)	2786 (11)	1191 (9)	10788 (6)	81 (6)
C(45)	3118 (11)	528 (9)	10119 (6)	101 (8)
C(46)	3300 (11)	950 (9)	9367 (6)	72 (6)
C(51)	5349 (7)	−918 (7)	6701 (6)	37 (3)
C(52)	6208 (7)	−566 (7)	6971 (6)	52 (4)
C(53)	7595 (7)	−1027 (7)	6646 (6)	59 (5)
C(54)	8123 (7)	−1842 (7)	6052 (6)	67 (5)
C(55)	7265 (7)	−2195 (7)	5783 (6)	59 (5)
C(56)	5878 (7)	−1733 (7)	6108 (6)	47 (4)
C(61)	2609 (11)	−901 (8)	6373 (6)	44 (4)
C(62)	2356 (11)	−424 (8)	5667 (6)	53 (4)
C(63)	1953 (11)	−929 (8)	5158 (6)	72 (6)
C(64)	1804 (11)	−1910 (8)	5355 (6)	75 (6)
C(65)	2057 (11)	−2387 (8)	6061 (6)	87 (7)
C(66)	2460 (11)	−1883 (8)	6570 (6)	61 (5)
C(71)	2835 (10)	−947 (8)	8190 (5)	40 (4)
C(72)	1504 (10)	−775 (8)	8573 (5)	58 (5)
C(73)	1222 (10)	−1305 (8)	9267 (5)	63 (5)
C(74)	2272 (10)	−2006 (8)	9580 (5)	63 (5)
C(75)	3604 (10)	−2178 (8)	9197 (5)	80 (6)
C(76)	3885 (10)	−1648 (8)	8503 (5)	59 (5)

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

Table 3. *Selected bond lengths* (Å) *and angles* (°)

$C_{32}H_{20}O_8P_2Re_2$		$C_{49}H_{35}O_7PRe_2Sb_2$	
Re(1)···Re(2)	3·928 (1)	Re(1)–Sb(1)	2·748 (2)
Re(1)–P(1)	2·526 (3)	Re(1)–Sb(2)	2·671 (1)
Re(1)–P(2)	2·528 (3)	Re(1)–P(1)	2·508 (3)
Re(2)–P(1)	2·520 (3)	Re(2)–Sb(1)	2·731 (1)
Re(2)–P(2)	2·513 (3)	Re(2)–P(1)	2·553 (4)
Re(2)–Re(1)–P(1)	38·8 (1)	Sb(1)–Re(1)–Sb(2)	104·1 (1)
Re(2)–Re(1)–P(2)	38·7 (1)	Sb(1)–Re(1)–P(1)	75·5 (1)
P(1)–Re(1)–P(2)	77·4 (1)	Sb(2)–Re(1)–P(1)	179·1 (1)
Re(1)–Re(2)–P(1)	39·0 (1)	Sb(1)–Re(2)–P(1)	75·1 (1)
Re(1)–Re(2)–P(2)	38·9 (1)	Re(1)–Sb(1)–Re(2)	98·4 (1)
P(1)–Re(2)–P(2)	77·8 (1)	Re(1)–P(1)–Re(2)	110·1 (1)
Re(1)–P(1)–Re(2)	102·2 (1)		
Re(1)–P(2)–Re(2)	102·4 (1)		

10° min^{-1}, 6317 reflections measured, $3 \leq 2\theta \leq 45°$, $-11 \leq h \leq 11$, $-14 \leq k \leq 14$, $0 \leq l \leq 18$, three standard reflections recorded every 400 reflections showed intensity loss of 6% during data collection, Lp correction, absorption correction applied *via* ψ scans, min./max. transmission 0·19/1·00, after merging (R_{int} = 0·046) 6076 unique reflections of which 1097 were considered unobserved, $I < 2{\cdot}5\sigma(I)$; structure solution by direct methods (*SHELXS*84, Sheldrick, 1985) and ΔF syntheses, full-matrix least-squares refinement based on *F* and 187 parameters, Re, Sb and P refined anisotropically, phenyl rings and H atoms treated as above, $R = 0{\cdot}0626$, $wR = 0{\cdot}0605$, $w = 1/\sigma^2(F)$, $(\Delta/\sigma)_{max} = 0{\cdot}062$, no significant features in final ΔF map; scattering factors and computer program as above.

Discussion. The structures of the title substances are shown in Figs. 1 and 2. Atomic parameters are given in Tables 1 and 2 and selected bond lengths and angles in Table 3.*

Whereas the compound $[Re_2(CO)_8(\mu\text{-}PPh_2)_2]$ crystallizes with four molecules in the monoclinic system, the other rhenium compound is triclinic with two molecules per unit cell. Each molecule of the different compounds has C_1 point-group symmetry and a nearly planar four-membered ring as the central molecular fragment. The dihedral angles of 4·6 (1)° in $Re_2(\mu\text{-}P)_2$ and 8·3 (1)° in $Re_2(\mu\text{-}P)(\mu\text{-}Sb)$ show a small deviation from the ring planarity that is generally preferred for the $M_2(\mu\text{-}P)_2$ ring type (*M* = transition metal) in crystalline solids (Deppisch, Schäfer, Binder & Leske, 1984). In our opinion each of the observed dihedral angles is related more to packing forces than to repulsive forces between non-bonded atoms in the neighborhood of the ring atoms because no remarkable C···O or O···O intramolecular contact lengths are present in the rhenium complexes. Furthermore, packing-energy calculations with the program *PCK*6 (Williams, 1972), based on the non-bonding intermolecular interactions between C and O atoms with C···C, C···O and O···O potential curves given by Mirsky (1978) and a summation limit of 6·0 Å, result in a lower energy for $[Re_2(CO)_8(\mu\text{-}PPh_2)_2]$ (−92 kJ mol^{-1}) than for $[Re_2(CO)_7(\mu\text{-}PPh_2)(\mu\text{-}SbPh_2)(SbPh_3)]$ (−116 kJ mol^{-1}).

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* Lists of structure factors, anisotropic temperature factors, H-atom coordinates and a complete list of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51277 (37 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

A comparison of the structural ring parameters from $Re_2(\mu\text{-}P)_2$ to $Re_2(\mu\text{-}P)(\mu\text{-}Sb)$ shows that the angle subtended at the Re atoms is only reduced by about 2·3 (1)°, whereas that at the P atoms is distinctly enlarged by 7·8 (1)°. This means that the previously mentioned isovalent exchange realizes for the remaining μ-P atom a better approximation to an idealized sp^3-hybridization state in such rings of the type $M_2(\mu\text{-}P)_2$ (Mason & Mingos, 1973). In addition, the last-named ring type has, in the absence of steric expanded groups bound to the ring atoms, the following ring bond angles (av.): P—M—P 75–78° and M—P—M 102–105°. Each range of these bond angles has been found not only for different transition-metal atoms M but also for various ligands attached at the ring atoms (Deppisch *et al.*, 1984). Compared to the μ-P bond-angle alteration in the title substances, the examined exchange of isovalent bridging atoms has a more distinct effect.

The average Re—P bond lengths of both the rhenium compounds do not differ significantly. The covalent single-bond length of an Re—Sb bond has been measured for the first time; its value of 2·671 (1) Å in the terminal coordinated $SbPh_3$ group is shorter than that of 2·740 (1) Å (av.) in the bridging diphenylantimonido group.

Intermolecular distances in the title compounds do not indicate interactions greater than van der Waals forces.

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Tris(trimethylsilylcyclopentadienyl)uranium(III)

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Abstract. [$\{(CH_3)_3SiC_5H_4\}_3U$], $M_r = 649{\cdot}87$, orthorhombic, *Pbca*, $a = 22{\cdot}630\,(8)$, $b = 29{\cdot}177\,(10)$, $c = 8{\cdot}428\,(3)$ Å, $V = 5564{\cdot}8$ Å^3, $Z = 8$, $D_x = 1{\cdot}551$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 56{\cdot}64$ cm^{-1}, $F(000) = 2536$, $T = 296$ K, $R = 0{\cdot}041$ [2251 data, $F^2 > 2\sigma(F^2)$]. The U atom is bonded to the three cyclopentadienyl rings in a pentahapto fashion and is in the plane of the ring centroids. The U to ring distances are 2·54, 2·47 and 2·51 Å, and the average U—C distance is 2·78 (4) Å.

Introduction. The title compound was prepared as part of a systematic synthetic and X-ray crystallographic investigation of the role of steric effects on the stereochemistry of trivalent metallocenes. Trivalent uranium metallocenes form compounds of the type $(RC_5H_4)_3U(L)$ where R is either H or CH_3 and L is a Lewis base such as tetrahydrofuran (Wasserman, Zozulin, Moody, Ryan & Salazar, 1983), tetrahydrothiophene (Zalkin & Brennan, 1985), 4-dimethylaminopyridine (Zalkin & Brennan, 1987), trimethylphosphine (Brennan & Zalkin, 1985), or 1,2-bis(dimethylphosphino)ethane (Zalkin, Brennan & Andersen, 1987). All of these molecules may be described as four-coordinate complexes of trivalent uranium (defining the midpoint of the cyclopentadienyl ring centroid as occupying one coordination position) with a distorted tetrahedral stereochemistry. Increasing the size of the substituent on the cyclopentadienyl ring to Me_3Si gives monomeric base-free $(Me_3SiC_5H_4)_3U$ (Brennan, Andersen & Zalkin, 1986), the title compound, the structure of which was determined in order

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