2362 reflections
404 parameters
Only coordinates of H atoms refined

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	*y*	*z*	B_{eq}
Fe	0	0	0	1.99 (2)
P	0.02225 (7)	−0.00583 (8)	0.1896 (1)	3.05 (3)
O1	0.3848 (2)	−0.1798 (2)	−0.1424 (4)	4.6 (1)
O2	0.1631 (2)	0.4049 (2)	0.0290 (4)	4.7 (1)
O3	−0.0149 (2)	−0.0598 (2)	0.2545 (4)	5.0 (1)
O4	0.0264 (2)	0.0544 (3)	0.2704 (4)	6.0 (1)
O5	0.0939 (2)	−0.0314 (4)	0.2184 (4)	5.0 (1)
N1	0.0871 (2)	0.0343 (2)	−0.0263 (4)	2.10 (9)
N2	0.0327 (2)	−0.0899 (2)	−0.0166 (4)	2.21 (8)
C1	0.0670 (2)	0.1504 (2)	−0.0053 (5)	2.5 (1)
C2	0.1064 (2)	0.0978 (3)	−0.0247 (5)	2.4 (1)
C3	0.1731 (3)	0.1020 (3)	−0.0488 (5)	2.9 (1)
C4	0.1943 (3)	0.0424 (3)	−0.0649 (5)	3.0 (1)
C5	0.1414 (2)	−0.0011 (3)	−0.0499 (4)	2.5 (1)
C6	0.1451 (2)	−0.0674 (3)	−0.0549 (5)	2.5 (1)
C7	0.0938 (2)	−0.1086 (2)	−0.0379 (5)	2.3 (1)
C8	0.0975 (3)	−0.1778 (3)	−0.0456 (5)	3.0 (1)
C9	0.0390 (3)	−0.2008 (3)	−0.0307 (5)	3.0 (1)
C10	−0.0023 (3)	−0.1463 (2)	−0.0118 (5)	2.54 (9)
C11	0.0963 (2)	0.2166 (2)	0.0015 (5)	2.5 (1)
C12	0.0902 (3)	0.2610 (3)	−0.0862 (5)	3.6 (1)
C13	0.1138 (3)	0.3230 (3)	−0.0746 (6)	3.9 (1)
C14	0.1434 (3)	0.3413 (3)	0.0251 (5)	3.2 (1)
C15	0.1519 (3)	0.2979 (3)	0.1107 (6)	4.4 (2)
C16	0.1275 (3)	0.2362 (3)	0.0986 (6)	4.1 (1)
C17	0.1834 (4)	0.4280 (3)	0.1383 (7)	5.6 (2)
C18	0.2084 (2)	−0.0975 (3)	−0.0803 (5)	2.5 (1)
C19	0.2559 (3)	−0.0978 (3)	0.0009 (6)	3.4 (1)
C20	0.3143 (3)	−0.1257 (3)	−0.0230 (5)	3.7 (1)
C21	0.3256 (3)	−0.1540 (3)	−0.1283 (5)	3.3 (1)
C22	0.2790 (3)	−0.1548 (3)	−0.2089 (6)	3.9 (1)
C23	0.2214 (3)	−0.1266 (3)	−0.1841 (6)	3.6 (1)
C24	0.4018 (3)	−0.1984 (4)	−0.2551 (7)	5.7 (2)
C25	−0.0032 (4)	−0.0753 (4)	0.3754 (6)	6.4 (2)
C26	−0.0314 (3)	0.0911 (5)	0.2976 (7)	6.9 (2)
C27	0.1447 (3)	0.0081 (3)	0.2449 (6)	4.5 (1)

Table 2. *Selected geometric parameters* (Å, °)

Fe—P	2.255 (2)	C2—C3	1.438 (7)
Fe—N1	1.996 (4)	C3—C4	1.326 (8)
Fe—N2	1.995 (4)	C4—C5	1.445 (8)
N1—C2	1.376 (6)	C5—C6	1.376 (8)
N1—C5	1.388 (7)	C6—C7	1.393 (8)
N2—C7	1.370 (7)	C6—C18	1.505 (7)
N2—C10′	1.382 (6)	C7—C8	1.439 (7)
C1—C2	1.390 (7)	C8—C9	1.335 (8)
C1—C10′	1.384 (8)	C9—C10	1.442 (7)
C1—C11	1.505 (7)		
P—Fe—N1	88.6 (1)	C3—C4—C5	107.6 (5)
P—Fe—N1′	91.4 (1)	N1—C5—C4	109.4 (5)
P—Fe—N2	88.4 (1)	N1—C5—C6	125.6 (5)
P—Fe—N2′	91.6 (1)	C4—C5—C6	125.0 (5)
N1—Fe—N2	89.9 (2)	C5—C6—C7	124.1 (5)
N1—Fe—N2′	90.1 (2)	N2—C7—C6	125.9 (5)
C2—N1—C5	105.2 (4)	N2—C7—C8	110.1 (5)
C7—N2—C10	105.8 (4)	C6—C7—C8	124.0 (5)
C2—C1—C10′	124.5 (5)	C7—C8—C9	107.2 (5)
N1—C2—C1	125.0 (5)	C8—C9—C10	107.5 (5)
N1—C2—C3	110.2 (5)	N2—C10—C1′	125.8 (5)
C1—C2—C3	124.8 (5)	N2—C10—C9	109.4 (5)
C2—C3—C4	107.5 (5)	C1′—C10—C9	124.8 (5)

All the calculations were performed with a Digital MicroVAX 3100 using the *MolEN* package (Fair, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71728 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1063]

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1,4,7,10,13-Pentaoxacyclopentadecane (15-Crown-5) Sodium Iodide Complex, $C_{10}H_{20}O_5.NaI$

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Abstract

The Na cation forms a 1/1 complex with 15-crown-5 ether. In this complex, Na^+ is six-coordinated to the five macrocyclic O atoms (average Na—O 2.42 Å) and the I^- ion. The coordination geometry is pentagonal pyramidal. The Na^+ ion lies 0.798 (3) Å above the least-squares plane of the ether O atoms. All structural parameters of the crown ether are similar to those of related complexes.

Comment

To date, only one crystal structure for a sodium complex of 15-crown-5 ether has been published,

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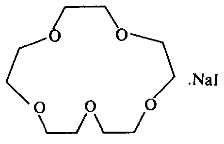
that being for the perchlorate salt (Shoham & Cohen, 1989). In the sodium perchlorate complex, the Na^+ ion is seven-coordinate. The NaI complex of 15-crown-5 ether is expected to be six-coordinate and the present study was initiated in order to examine the effect of seven- *versus* six-coordinate Na on the conformational features of the 15-crown-5 ether, as well as to examine changes in Na—O distances and coordination geometry.

In the 15-crown-5.NaI complex (I), the O atoms are strongly bonded to Na with the Na—O distances ranging from 2.398 (4) to 2.444 (4) Å. The mean Na—O distance is 2.42 Å, which is identical to that found for the 15-crown-5.$NaClO_4$ complex (Shoham & Cohen, 1989). The five O atoms of the macrocycle define a least-squares plane, with the following distances for each O atom from this plane: O1 0.025 (5), O2 −0.290 (5), O3 0.486 (5), O4 −0.454 (5) and O5 0.217 (5) Å. From this mean plane, the average deviation of the O-atom positions is 0.29 (4) Å, a value very similar to that (0.28 Å) found for the 15-crown-5.$NaClO_4$ complex (Shoham & Cohen, 1989). In the crown ether, three O atoms, O1, O3 and O5, are on one side of this mean plane, while O2 and O4 are on the other side pointing toward the Na^+ ion, which is situated 0.798 (3) Å out of the plane. In contrast, in the 15-crown-5.$NaClO_4$ complex, O3 and O5 are on one side of the mean plane, and O1, O2 and O4 are on the other side pointing toward the Na^+ ion (Shoham & Cohen, 1989), which is 0.77 Å out of the mean plane. Thus the nature of the counter ion influences the stereochemistry of the pentagonal pyramidal coordination.



(I)

The conformation of the 15-crown-5 moiety can be described *via* the 15 torsion-angle sequence $(\mathit{apsc} + \mathit{apapsc} - \mathit{ap})_2\mathit{apsc} + \mathit{ac} -$, which is the most common for this type of macrocyclic ether (Hazell, Hazell, Holm & Krogh, 1991). All the O—C—C—O torsion angles are close to the expected *gauche* (synclinal) conformation (60°) and all of the C—C—O—C angles are close to the *trans* (antiperiplanar) conformation (180°), with the exception of C3—O2—C2—C1 [−90.2 (5)°].

The conformation of the 15-crown-5 ether in the present NaI complex is essentially identical to that found for the $NaClO_4$ complex (Shoham & Cohen, 1989). An *ORTEP* (Johnson, 1965) view of the complex and a stereoview of the unit-cell packing are shown in Figs. 1 and 2, respectively.

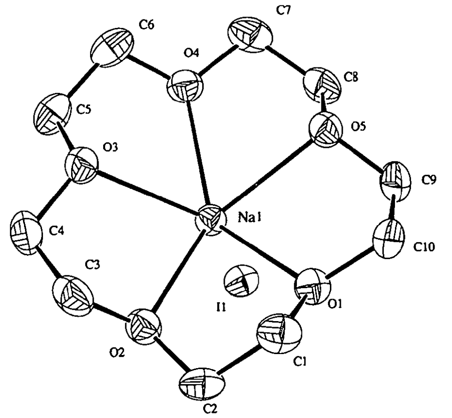


Fig. 1. *ORTEP* diagram of the structure of 15-crown-5.NaI with atomic numbering scheme.

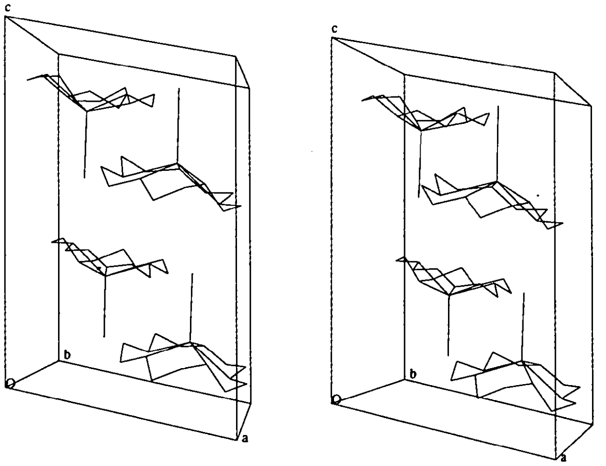


Fig. 2. Stereoview of the unit-cell packing for 15-crown-5.NaI.

Experimental

15-crown-5 ether and NaI were purchased from Aldrich and used without further purification. The 15-crown-5.NaI complex was obtained by mixing equimolar amounts of these reagents and crystals were afforded *via* slow evaporation from a CH_2Cl_2/EtOH (7:3) solution.

Crystal data

$[NaI(C_{10}H_{20}O_5)]$
$M_r = 370.16$
Monoclinic
$P2_1/c$
$a = 9.8719\ (24)$ Å
$b = 9.9071\ (20)$ Å
$c = 15.4065\ (23)$ Å
$\beta = 101.294\ (17)°$
$V = 1477.6\ (5)$ Å^3
$Z = 4$

$D_x = 1.664$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 20$–$25°$
$\mu = 2.22$ mm^{-1}
$T = 193$ K
Cube
0.2 × 0.2 × 0.2 mm
White

Data collection

Rigaku diffractometer
$\theta/2\theta$ scans (2° $\min^{-1}$) with profile analysis (Grant & Gabe, 1978)
Absorption correction: empirical
$T_{\min} = 0.139$, $T_{\max} = 0.175$
2772 measured reflections
2613 independent reflections
1988 observed reflections $[I > 2.5\sigma(I)]$

$R_{\rm int} = 0.065$
$\theta_{\max} = 24.9°$
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 18$
3 standard reflections monitored every 150 reflections intensity variation: 3.0%

Refinement

Refinement on F
$R = 0.037$
$wR = 0.052$
$S = 2.29$
1988 reflections
155 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{\max} = 0.012$

$\Delta\rho_{\max} = 1.97$ e Å^{-3}
$\Delta\rho_{\min} = -0.61$ e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 0.138
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{\rm eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
I	0.24171 (4)	0.99635 (3)	0.108129 (22)	2.925 (16)
Na	0.25128 (21)	0.98425 (18)	0.30726 (13)	2.24 (8)
O1	0.4338 (4)	0.8289 (3)	0.36342 (24)	2.60 (16)
O2	0.1553 (4)	0.7735 (4)	0.3438 (3)	2.65 (16)
O3	0.0704 (4)	1.0234 (3)	0.3921 (3)	2.70 (16)
O4	0.2125 (4)	1.2223 (4)	0.3270 (3)	2.96 (17)
O5	0.4651 (4)	1.1010 (4)	0.3726 (3)	2.68 (16)
C1	0.3926 (6)	0.7211 (6)	0.4155 (4)	3.1 (3)
C2	0.2564 (6)	0.6686 (5)	0.3623 (4)	3.2 (3)
C3	0.0732 (6)	0.7871 (6)	0.4095 (4)	3.3 (3)
C4	−0.0177 (6)	0.9094 (7)	0.3848 (4)	3.4 (3)
C5	0.0017 (6)	1.1445 (6)	0.3577 (4)	3.3 (3)
C6	0.1047 (6)	1.2571 (6)	0.3728 (4)	3.5 (3)
C7	0.3340 (7)	1.3014 (5)	0.3508 (4)	3.6 (3)
C8	0.4502 (6)	1.2261 (6)	0.3224 (4)	3.3 (3)
C9	0.5742 (6)	1.0185 (6)	0.3554 (5)	3.7 (3)
C10	0.5653 (6)	0.8855 (6)	0.4018 (4)	3.4 (3)

Table 2. *Selected geometric parameters* (Å, °)

Na—I	3.0532 (20)	O3—C5	1.429 (7)
Na—O1	2.398 (4)	O4—C6	1.430 (7)
Na—O2	2.405 (4)	O4—C7	1.420 (7)
Na—O3	2.442 (4)	O5—C8	1.453 (7)
Na—O4	2.418 (4)	O5—C9	1.418 (7)
Na—O5	2.444 (4)	C1—C2	1.523 (8)
O1—C1	1.441 (7)	C3—C4	1.512 (9)
O1—C10	1.432 (7)	C5—C6	1.496 (9)
O2—C2	1.431 (6)	C7—C8	1.504 (9)
O2—C3	1.422 (7)	C9—C10	1.509 (9)
O3—C4	1.416 (7)		
I—Na—O1	105.12 (11)	Na—O3—C4	109.5 (3)
I—Na—O2	109.47 (11)	Na—O3—C5	106.2 (3)
I—Na—O3	130.52 (12)	C4—O3—C5	113.6 (4)
I—Na—O4	96.53 (11)	Na—O4—C6	116.7 (3)
I—Na—O5	104.44 (11)	Na—O4—C7	115.1 (3)
O1—Na—O2	70.20 (13)	C6—O4—C7	114.4 (4)
O1—Na—O3	119.03 (15)	Na—O5—C8	101.3 (3)
O1—Na—O4	134.96 (15)	Na—O5—C9	106.1 (3)
O1—Na—O5	68.16 (13)	C8—O5—C9	113.2 (4)
O2—Na—O3	69.47 (13)	O1—C1—C2	105.6 (4)
O2—Na—O4	137.56 (15)	O2—C2—C1	111.5 (4)
O2—Na—O5	131.51 (15)	O2—C3—C4	106.9 (5)
O3—Na—O4	68.12 (13)	O3—C4—C3	107.1 (4)
O3—Na—O5	111.56 (14)	O3—C5—C6	108.0 (5)
O4—Na—O5	68.39 (13)	O4—C6—C5	107.0 (5)
Na—O1—C1	113.7 (3)	O4—C7—C8	107.7 (4)
Na—O1—C10	117.0 (3)	O5—C8—C7	105.8 (4)
C1—O1—C10	113.1 (4)	O5—C9—C10	107.4 (5)
Na—O2—C2	112.7 (3)	O1—C10—C9	106.2 (5)
Na—O2—C3	113.0 (3)	C2—O2—C3	113.6 (4)
I—Na—O1—C1	−126.6 (3)	I—Na—O1—C10	98.6 (3)
O2—Na—O1—C1	−21.0 (2)	O2—Na—O1—C10	−155.8 (3)
O3—Na—O1—C1	30.3 (2)	O3—Na—O1—C10	−104.6 (3)
O4—Na—O1—C1	117.5 (3)	O4—Na—O1—C10	−17.3 (2)
O5—Na—O1—C1	133.6 (3)	O5—Na—O1—C10	−1.2 (2)
I—Na—O2—C2	88.9 (3)	I—Na—O2—C3	−140.7 (3)
O1—Na—O2—C2	−10.7 (2)	O1—Na—O2—C3	119.6 (3)
O3—Na—O2—C2	−144.0 (3)	O3—Na—O2—C3	−13.6 (2)
O4—Na—O2—C2	−146.7 (3)	O4—Na—O2—C3	−16.3 (2)
O5—Na—O2—C2	−42.9 (3)	O5—Na—O2—C3	87.5 (3)
I—Na—O3—C4	76.9 (3)	I—Na—O3—C5	−46.1 (3)
O1—Na—O3—C4	−73.2 (3)	O1—Na—O3—C5	163.8 (3)
O2—Na—O3—C4	−21.6 (3)	O2—Na—O3—C5	−144.6 (3)
O4—Na—O3—C4	156.4 (3)	O4—Na—O3—C5	33.4 (3)
O5—Na—O3—C4	−149.4 (3)	O5—Na—O3—C5	87.6 (3)
I—Na—O4—C6	128.8 (3)	I—Na—O4—C7	−93.1 (3)
O1—Na—O4—C6	−112.2 (3)	O1—Na—O4—C7	26.0 (3)
O2—Na—O4—C6	0.3 (3)	O2—Na—O4—C7	138.5 (3)
O3—Na—O4—C6	−2.5 (3)	O3—Na—O4—C7	135.7 (3)
O5—Na—O4—C6	−128.3 (3)	O5—Na—O4—C7	9.9 (3)
I—Na—O5—C8	51.0 (2)	I—Na—O5—C9	−67.4 (3)
O1—Na—O5—C8	151.8 (3)	O1—Na—O5—C9	33.4 (3)
O2—Na—O5—C8	−175.5 (3)	O2—Na—O5—C9	66.1 (3)
O3—Na—O5—C8	−94.4 (3)	O3—Na—O5—C9	147.2 (3)
O4—Na—O5—C8	−40.3 (3)	O4—Na—O5—C9	−158.7 (4)
Na—O1—C1—C2	46.7 (3)	C10—O1—C1—C2	−176.7 (6)
Na—O1—C10—C9	−29.0 (3)	C1—O1—C10—C9	−164.2 (6)
Na—O2—C2—C1	39.8 (3)	C3—O2—C2—C1	−90.2 (5)
Na—O2—C3—C4	44.7 (3)	C2—O2—C3—C4	174.7 (6)
Na—O3—C4—C3	52.3 (3)	C5—O3—C4—C3	170.8 (6)
Na—O3—C5—C6	−61.9 (3)	C4—O3—C5—C6	177.7 (6)
Na—O4—C6—C5	−27.5 (3)	C7—O4—C6—C5	−166.0 (6)
Na—O4—C7—C8	22.2 (2)	C6—O4—C7—C8	161.3 (6)
Na—O5—C8—C7	68.6 (3)	C9—O5—C8—C7	−178.3 (6)
Na—O5—C9—C10	−62.3 (3)	C8—O5—C9—C10	−172.5 (6)
O1—C1—C2—O2	−57.1 (3)	O2—C3—C4—O3	−65.4 (4)
O3—C5—C6—O4	60.0 (4)	O4—C7—C8—O5	−62.5 (4)
O5—C9—C10—O1	61.2 (4)		

The structure was solved by direct methods and refined by full-matrix least squares. H-atoms positions were found by difference Fourier synthesis and refined isotropically. All calculations were performed using the *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989) package.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: CR1080). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Di-μ-bromo-tetrabromotetrakis(trimethylphosphine)dirhodium(III)

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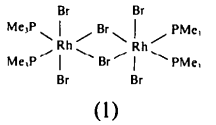
Abstract

The title structure, $[Rh_2Br_6(C_3H_9P)_4]$ (1), consists of neutral $[\mu\text{-}Br_2\text{-}\{RhBr_2(PMe_3)_2\}_2]$ bromide-bridged rhodium dimers. The Rh—P bond lengths are 2.273 (5) and 2.266 (5) Å.

Comment

As part of an investigation of the products arising from the irradiation of $[Rh\{P(CH_3)_3\}_2(CO)Cl]$ (2) (Tanaka, 1989) in the presence of aromatic hydrocarbons, a reaction mixture containing both phenylrhodium(III) hydride complexes and the starting complex, (2), was treated with an excess of dibromomethane in an attempt to obtain the corresponding phenylrhodium(III) bromide derivatives. On standing, red crystals of the neutral Rh^{III} dimer, di-μ-bromo-tetrabromotetrakis(trimethylphosphine)dirhodium(III), (1), precipitated from the reaction mixture. In this paper, the structure of $[Rh_2\{P(CH_3)_3\}_4Br_6]$, (1), is reported.



(1)

The structure consists of neutral Rh dimers. The Rh atoms are bridged by two Br^- ligands and are each coordinated to other Br^- ligands in positions *trans* to each other and *cis* to the bridging ligands and the two trimethylphosphine ligands. The trimethylphosphine ligands are *cis* to each other and *trans* to the bridging ligands. The chloro complex $[Rh_2(^nBu_3P)_4Cl_6]$ (Muir, Muir & Rivera, 1974) has an analogous arrangement around one of the Rh atoms, but has *trans* phosphines and *cis* chloro ligands around the other. The Rh—P bond lengths in the present structure [2.273 (5) and 2.266 (5) Å] are longer than the *cis* Rh—P bonds in the chloro structure [2.256 (15) Å] but shorter than the *trans* Rh—P bonds [2.321 (12) Å].

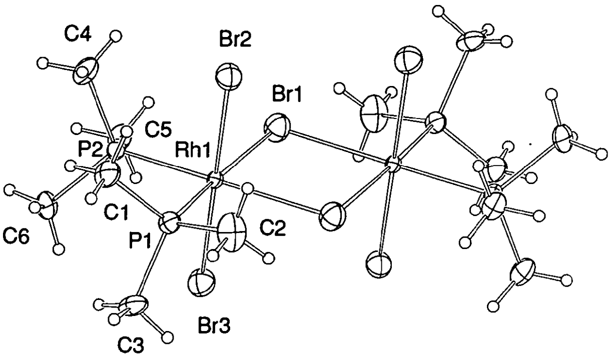


Fig. 1. *ORTEP* (Johnson, 1965) plot of $[Rh_2\{P(CH_3)_3\}_4Br_6]$.

Experimental

$[Rh\{P(CH_3)_3\}_2(CO)Cl]$ was synthesized in 65% yield (after sublimation and recrystallization from ether) using an analogous procedure to that outlined for the synthesis of $[Rh(PPh_3)_2(CO)Cl]$ from $[Rh_2(CO)_4Cl_2]$ (McCleverty & Wilkinson, 1966). A solution of $[Rh\{P(CH_3)_3\}_2(CO)Cl]$ (0.5 ml, 125 m*M*) in benzene/tetrahydrofuran (1:3 *v*/*v*) was irradiated under nitrogen at 233 K for 48 h with a 125 W medium-pressure mercury-vapour lamp. The resulting mixture was treated with dibromomethane (100 µl, 253 K, 2 d) and then warmed to room temperature. Crystals of $[Rh_2\{P(CH_3)_3\}_4Br_6]$ (1) precipitated from the reaction mixture over several days.

Crystal data

$[Rh_2Br_6(C_3H_9P)_4]$
$M_r = 989.58$
Orthorhombic
Pcab
$a = 10.971\ (2)$ Å
$b = 15.562\ (2)$ Å
$c = 16.459\ (3)$ Å
$V = 2810.0\ (11)$ Å^3
$Z = 4$
$D_x = 2.339$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 10\text{-}15°$
$\mu = 9.730$ mm^{-1}
$T = 294$ K
Prism
0.22 × 0.22 × 0.15 mm
Red

Data collection

Enraf-Nonius CAD-4*F* diffractometer
$\omega\text{-}2/3\theta$ scans
Absorption correction: empirical
$T_{min} = 0.313$, $T_{max} = 0.410$
2811 measured reflections
2024 independent reflections

1351 observed reflections $[I > 2.5\sigma(I)]$
$\theta_{max} = 25°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 19$
3 standard reflections
frequency: 60 min
intensity variation: none

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