

been observed for the shortest U—O bridge bond in similar tetranuclear uranyl complexes (Åberg, 1976; Perrin & Le Marouille, 1977).

The U—O distances in the uranyl groups are in the range of observed values for equatorial coordination when the uranyl oxygens are not further coordinated [mean value: 1.764 Å (*Gmelin Handbook of Inorganic Chemistry*, 1983)]; nevertheless the uranyl groups deviate slightly from linearity with O—U—O angles of 174.2 (12) and 174.0 (10)°, being bent towards the exterior of the molecular unit. The three negatively polarized ligands around U(1) seem to push the O(1) and O(2) uranyl oxygens towards the other two ligands, the two neutral water molecules O(*w*). The bisectrix of the O(1)—U(1)—O(2) angle is effectively directed towards the line joining these two water molecules. Likewise the four negatively polarized ligands around the U(2) atom must push the O(3) uranyl oxygens towards the fifth ligand, the neutral THF oxygen; the bisectrix of the O(3)—U(2)—O(3ⁱⁱⁱ) angle is effectively directed towards the O(5) atom.

Least-squares planes have been calculated through (a) all four uranium atoms U(1), U(1ⁱ), U(2) and U(2ⁱ), (b) the U(1) pentagon, (c) the U(2) pentagon and (d) the uranyl groups of U(1) and U(2). The angles between these planes are respectively (a,b) = 4 (1), (a,c) = 2 (1), (b,c) = 6 (1), (a,d) = 89 (1), (b,d) = 85 (1) and (c,d) = 91 (1)°. The largest deviations from the expected values of 0 and 90° occur for the angles with the plane through the U(1) pentagon; this could be explained by the fact that the bridging Cl and O atoms as well as the THF oxygens lie in the plane of the four U atoms while the water oxygens are 0.21 (4) Å out of

this plane. The THF's form dihedral angles of 89 (2)° with the plane of the four U atoms. The bond distances in the THF molecules are foreshortened compared with the values expected for a single conformation, the thermal vibrations principally of the terminal C atoms being large (Table 1). Such disorder in THF with the outermost carbon atoms elongated perpendicular to the ring has been observed in many other crystals Reynold, Zalkin & Templeton, 1977; Rietz, Edelstein, Ruben, Templeton & Zalkin, 1978; Smith & Raymond, 1979).

There are no short intermolecular interactions that could correspond to hydrogen bonds so that the stacking in the crystal is assumed to be mainly a result of van der Waals interactions.

References

- ÅBERG, M. (1976). *Acta Chem. Scand Ser. A*, **30**, 507–514.
 BEECKMAN, W., GOFFART, J., REBIZANT, J. & SPIRLET, M. R. (1986). *J. Organomet. Chem.* **307**, 23–37.
 Enraf–Nonius (1981). *Structure Determination Package*. Version 18. Enraf–Nonius, Delft.
Gmelin Handbook of Inorganic Chemistry (1983). 8th ed. Uranium Suppl. Vol. A6. Berlin: Springer-Verlag.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
 PERRIN, A. & LE MAROUILLE, J. Y. (1977). *Acta Cryst.* **B33**, 2477–2481.
 REYNOLD, J. G., ZALKIN, A. & TEMPLETON, D. H. (1977). *Inorg. Chem.* **16**, 3357–3359.
 RIETZ, R. R., EDELSTEIN, N. M., RUBEN, H. W., TEMPLETON, D. H. & ZALKIN, A. (1978). *Inorg. Chem.* **17**, 658–660.
 SMITH, W. L. & RAYMOND, K. N. (1979). *J. Inorg. Nucl. Chem.* **41**, 1431–1436.

Acta Cryst. (1987). **C43**, 839–842

Structure of *trans*-Dibromotetrakis(pyridine)rhodium(III) Bromide Hexahydrate

BY MARIEL M. MUIR, GRISELL M. GOMEZ, JAMES A. MUIR AND SERGIO SANCHEZ

Departments of Chemistry and Physics, University of Puerto Rico, Rio Piedras, PR 00931, USA

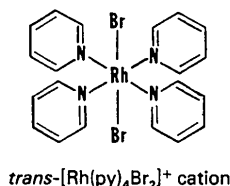
(Received 20 June 1986; accepted 29 October 1986)

Abstract. $[\text{RhBr}_2(\text{C}_5\text{H}_5\text{N})_4]\text{Br}\cdot 6\text{H}_2\text{O}$, $M_r = 767.2$, monoclinic, $P2_1/c$, $a = 12.663$ (3), $b = 10.703$ (3), $c = 21.585$ (7) Å, $\beta = 98.19$ (2)°, $V = 2895.6$ Å³, $Z = 4$, $D_x = 1.76$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 46.20$ cm⁻¹, $F(000) = 1512$, $T = 296$ K, final $R = 0.047$ for 2438 observed reflections. The crystal structure is composed of *trans*- $[\text{Rh}(\text{py})_4\text{Br}_2]^+$ cations packed in layers separated by Br⁻ anions and H₂O molecules. The Rh atom has a nearly regular octahedral geometry with an average Rh—N bond length of

2.069 (2) Å and an average Rh—Br bond length of 2.478 (1) Å.

Introduction. The study of photoaquation reactions of Rh^{III} complexes has increased in the past few years (Muir & Huang, 1973; Ford & Petersen, 1975; Clark & Petersen, 1981; Muir, Zinner, Paguaga & Torres, 1982). For complexes of the type *trans*- $[\text{Rh}(A)_4X_2]^+$, where *A* is a heterocyclic amine and *X* is Cl or Br, both photoaquation of halide and amine have been obtained

with stereoretention. The quantum yields for these processes depend on the basicity of the amine (Muir *et al.*, 1982; Sanchez, 1985). A single-crystal X-ray diffraction study of the title complex (shown below) has been undertaken to ascertain the full three-dimensional configuration of the complex, in order to study the relationship between structure and photochemical behavior of other tetraamminerhodium(III) complexes.



Experimental. Pyridine (4.80 g, 60.7 mmol) was added slowly to 25 mL of an aqueous solution of RhBr₃·2H₂O (2.63 g, 6.95 mmol), and a red precipitate formed. The reaction mixture was heated for 2 h and an orange precipitate formed. The resulting product was a mixture which included 16% *trans*-[Rh(py)₄Br₂]Br and 24.8% *mer*-[Rh(py)₃Br₃]. These products were separated by the use of the difference in their solubilities. The title compound was recrystallized from ethyl acetate (Sanchez, 1985).

Diffraction data were collected by the Molecular Structure Corp. and refined in this laboratory. Orange cubic crystal, D_m not determined, $0.20 \times 0.20 \times 0.20$ mm, Enraf-Nonius CAD-4 diffractometer, monochromated Mo $K\alpha$, ω - θ scan, scan speed $2-20^\circ \text{ min}^{-1}$ in ω , scan width $0.7 + 0.35 \tan \theta$ ($^\circ$), $2\theta_{\text{max}} = 45^\circ$, lattice parameters from least squares of 25 reflections in the range $5 < \theta < 11^\circ$. Systematic absences $0k0$ ($k = 2n + 1$) and $h0l$ ($l = 2n + 1$); $\pm h + k + l$ with $|h| \leq 13$, $k \leq 11$, $l \leq 23$; three standard reflections, intensities constant within experimental error; total unique reflections measured 4026, 3028 non-zero, 2438 with $F > 4\sigma(F)$. Absorption corrections from ψ scans, transmission factors 0.880 to 0.997. Structure was solved with *SHELX76* (Sheldrick, 1976), heavy-atom, difference-Fourier and full-matrix least-squares refinement methods using the 2438 observed reflections. All calculations were made using an IBM-4381 computer. Isotropic then anisotropic refinement on F of all non-H atoms, H atoms were not used; 308 parameters refined, $R = 0.047$, $wR = 0.046$, $w^{-1} = \sigma^2(F)$, $S = 1.5$; $\sigma(F_o)^2 = [\sigma(I)^2 + (0.05I)^2]^{1/2}/Lp$; difference syntheses showed a max. density of $0.81 \text{ e } \text{\AA}^{-3}$ (min. $-0.73 \text{ e } \text{\AA}^{-3}$); max. $\Delta/\sigma = -0.024$. Neutral-atom scattering factors (*International Tables for X-ray Crystallography*, 1974) were used, except for Rh^{III}, and were corrected for anomalous dispersion (Cromer & Liberman, 1970). An isotropic extinction parameter which refined to $x = 0.00053$ was applied and the calculated structure factor became $F = F(1 - xF^2/\sin \theta)$.

Discussion. The atomic coordinates and equivalent isotropic temperature factors are listed in Table 1, and bond lengths and angles in Table 2.* An *ORTEP* plot (Johnson, 1976) of the complex cation and the atom numbering is shown in Fig. 1, and the packing of the molecules (Motherwell, 1976) in Fig. 2. The Rh has a nearly regular octahedral geometry with the following average bond lengths and angles: Rh-N 2.069 (2), Rh-Br 2.478 (1) Å, Br-Rh-Br 179.2 (0.1) and N-Rh-N 90.0 (0.4) $^\circ$. The average Rh-Br bond length is about 0.14 Å longer than the analogous Rh-Cl bond length [2.33 (1) Å] (Acharya, Tavale & Guru, 1984; Colamarino & Orioli, 1976). The same difference can be observed in trihalotris(phosphine)-rhodium complexes. The average Rh-Cl distance for two *trans* chlorides is 2.36 (1) Å (Skapski & Stephens, 1973) and the average Rh-Br distance for two *trans* bromides is 2.50 (3) Å (English, 1979). This may be explained in terms of the higher *trans* influence of Br compared to Cl and the larger size of Br, and is

* Lists of structure factors, anisotropic thermal factors and least-squares-planes information have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43540 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atom coordinates and equivalent isotropic temperature factors* (\AA^2) *for trans*-[Rh(py)₄Br₂]Br·6H₂O

	x	y	z	U_{eq}^*
Rh	1.0162 (1)	0.2721 (1)	0.3823 (1)	0.030 (3)
Br(1)	0.9908 (1)	0.5003 (1)	0.3925 (1)	0.040 (9)
Br(2)	1.0392 (1)	0.0437 (1)	0.3710 (1)	0.050 (16)
Br(3)	0.4992 (1)	0.5845 (1)	0.8792 (1)	0.066 (3)
N(1)	0.9126 (7)	0.2796 (8)	0.2991 (4)	0.034 (3)
N(2)	0.8884 (7)	0.2402 (8)	0.4300 (4)	0.033 (6)
N(3)	1.1179 (7)	0.2724 (9)	0.4664 (4)	0.037 (9)
N(4)	1.1446 (7)	0.3011 (8)	0.3343 (4)	0.035 (8)
C(1)	0.9400 (10)	0.2257 (11)	0.2470 (6)	0.047 (8)
C(2)	0.8751 (10)	0.2371 (12)	0.1894 (6)	0.058 (6)
C(3)	0.7775 (10)	0.3058 (12)	0.1845 (6)	0.054 (2)
C(4)	0.7501 (9)	0.3577 (11)	0.2408 (5)	0.048 (9)
C(5)	0.8198 (9)	0.3434 (11)	0.2964 (5)	0.036 (2)
C(6)	0.8179 (8)	0.1465 (10)	0.4118 (5)	0.036 (9)
C(7)	0.7377 (9)	0.1173 (11)	0.4454 (6)	0.042 (6)
C(8)	0.7258 (9)	0.1843 (12)	0.5005 (6)	0.045 (3)
C(9)	0.7963 (9)	0.2828 (11)	0.5178 (5)	0.044 (6)
C(10)	0.8791 (9)	0.3070 (10)	0.4837 (5)	0.036 (18)
C(11)	1.1049 (9)	0.1891 (11)	0.5126 (6)	0.045 (4)
C(12)	1.1632 (10)	0.1963 (12)	0.5718 (6)	0.051 (3)
C(13)	1.2418 (10)	0.2901 (13)	0.5840 (6)	0.054 (11)
C(14)	1.2573 (9)	0.3717 (12)	0.5353 (6)	0.046 (12)
C(15)	1.1948 (8)	0.3615 (11)	0.4778 (5)	0.035 (14)
C(16)	1.2334 (8)	0.2304 (11)	0.3485 (6)	0.048 (20)
C(17)	1.3222 (10)	0.2497 (14)	0.3160 (6)	0.056 (20)
C(18)	1.3174 (11)	0.3429 (14)	0.2696 (6)	0.063 (9)
C(19)	1.2267 (11)	0.4165 (13)	0.2574 (6)	0.062 (8)
C(20)	1.1417 (9)	0.3922 (11)	0.2907 (5)	0.042 (4)
O(1)	0.5981 (8)	0.3575 (9)	0.7976 (4)	0.079 (20)
O(2)	0.5006 (7)	0.1535 (8)	0.9655 (4)	0.076 (3)
O(3)	0.6003 (7)	0.1168 (8)	0.8580 (4)	0.071 (1)
O(4)	0.5025 (8)	0.3763 (8)	0.6747 (4)	0.073 (20)
O(5)	0.3963 (7)	0.3787 (8)	0.9709 (4)	0.081 (11)
O(6)	0.6200 (7)	0.4458 (9)	0.5759 (4)	0.077 (4)

* Equivalent isotropic U calculated from anisotropic U . $U_{\text{eq}} = (U_{11} + U_{22} + U_{33})/3$.

consistent with the quantum yields obtained for photoaquation of halide in tetraaminerrhodium(III) complexes ($\Phi_{\text{Br}} > \Phi_{\text{Cl}}$) (Sanchez, 1985).

Table 2. Bond lengths (Å) and angles (°) in *trans*-[Rh(py)₄Br₂][Br·6H₂O]

Rh Br(1)	2.477 (2)	C(8)–C(9)	1.397 (16)
Rh Br(2)	2.478 (1)	C(9)–C(10)	1.386 (15)
Rh N(1)	2.068 (9)	C(10)–N(2)	1.381 (13)
Rh N(2)	2.070 (8)	N(3)–C(11)	1.369 (14)
Rh N(3)	2.069 (9)	C(11)–C(12)	1.388 (16)
Rh N(4)	2.071 (9)	C(12)–C(13)	1.410 (17)
N(1) C(1)	1.355 (13)	C(13)–C(14)	1.409 (17)
C(1) C(2)	1.394 (17)	C(14)–C(15)	1.378 (16)
C(2) C(3)	1.432 (17)	C(15)–N(3)	1.360 (13)
C(3) C(4)	1.430 (17)	N(4)–C(16)	1.354 (13)
C(4) C(5)	1.394 (15)	C(16)–C(17)	1.421 (16)
C(5) N(1)	1.354 (13)	C(17)–C(18)	1.408 (18)
N(2) C(6)	1.362 (13)	C(18)–C(19)	1.386 (19)
C(6) C(7)	1.363 (15)	C(19)–C(20)	1.403 (16)
C(7) C(8)	1.416 (15)	C(20)–N(4)	1.358 (14)
Br(1) Rh Br(2)	179.2 (0.1)	N(1)–C(1)–C(2)	121.1 (1.1)
N(1) Rh N(3)	177.4 (0.4)	C(1)–C(2)–C(3)	120.2 (1.2)
N(2) Rh N(4)	179.0 (0.3)	C(2)–C(3)–C(4)	116.9 (1.2)
N(1) Rh Br(1)	87.9 (0.2)	C(3)–C(4)–C(5)	119.1 (1.2)
N(1) Rh Br(2)	91.3 (0.2)	C(4)–C(5)–N(1)	122.4 (1.1)
N(2) Rh Br(1)	89.8 (0.2)	C(5)–N(1)–C(1)	120.2 (1.0)
N(2) Rh Br(2)	89.8 (0.2)	N(2)–C(6)–C(7)	121.4 (1.1)
N(2) Rh N(1)	89.6 (0.3)	C(6)–C(7)–C(8)	120.5 (1.1)
N(3) Rh Br(1)	89.5 (0.3)	C(7)–C(8)–C(9)	117.8 (1.1)
N(3) Rh Br(2)	91.2 (0.3)	C(8)–C(9)–C(10)	120.0 (1.1)
N(3) Rh N(2)	89.9 (0.3)	C(9)–C(10)–N(2)	120.7 (1.0)
N(4) Rh Br(1)	91.1 (0.2)	C(10)–N(2)–C(6)	119.5 (0.9)
N(4) Rh Br(2)	89.2 (0.2)	N(3)–C(11)–C(12)	122.2 (1.1)
N(4) Rh N(1)	90.4 (0.3)	C(11)–C(12)–C(13)	118.9 (1.2)
N(4) Rh N(3)	90.1 (0.4)	C(12)–C(13)–C(14)	118.2 (1.1)
Rh N(1) C(1)	119.9 (0.8)	C(13)–C(14)–C(15)	120.1 (1.1)
Rh N(1) C(5)	119.9 (0.7)	C(14)–C(15)–N(3)	121.7 (1.1)
Rh N(2) C(6)	120.4 (0.7)	C(15)–N(3)–C(11)	118.9 (1.0)
Rh N(2) C(10)	119.9 (0.7)	N(4)–C(16)–C(17)	119.6 (1.2)
Rh N(3) C(11)	120.9 (0.8)	C(16)–C(17)–C(18)	120.2 (1.2)
Rh N(3) C(15)	120.1 (0.8)	C(17)–C(18)–C(19)	118.9 (1.3)
Rh N(4) C(16)	119.4 (0.8)	C(18)–C(19)–C(20)	118.6 (1.3)
Rh N(4) C(20)	120.5 (0.8)	C(19)–C(20)–N(4)	122.6 (1.2)
		C(20)–N(4)–C(16)	120.1 (1.0)
Intramolecular contacts in the Br·6H ₂ O unit (Å)			
Br(3)···O(1)	3.346 (10)	O(1)···O(3)	2.884 (13)
Br(3)···O(4)	3.332 (10)	O(1)···O(4)	2.763 (13)
Br(3)···O(5)	3.347 (10)	O(2)···O(3)	2.823 (12)
		O(2)···O(5)	2.759 (14)
		O(4)···O(6)	2.873 (13)

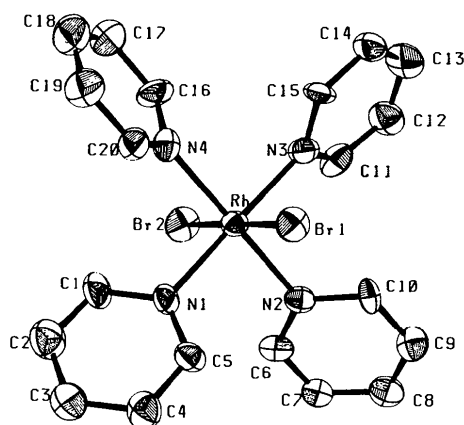


Fig. 1. Labeling of atoms in the *trans*-[Rh(py)₄Br₂]⁺ cation (ORTEP diagram, 50% probability ellipsoids).

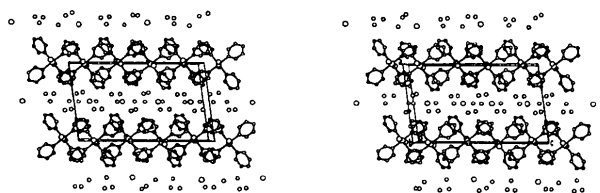


Fig. 2. Packing diagram of *trans*-[Rh(py)₄Br₂][Br·6H₂O] molecules in the unit cell.

The average C–N and C–C distances [1.36 (1), 1.40 (2) Å] and bond angles [120 (2)°] for coordinated pyridine are similar to those previously reported (Rozière, Lehmann & Potier, 1979; Acharya *et al.*, 1984). The pyridine rings are all essentially planar with no atom deviating from the least-squares plane by more than 0.023 (12) Å [average $\chi^2 = 5.8 (0.5)$]. The average dihedral angle between the RhN₄ plane and the pyridine planes is 45 (4)° and the average dihedral angle between two *trans* pyridine moieties is 84.2 (0.5)°. This type of coordination about the Rh³⁺ ion, with the pyridines oriented in a propeller-like manner about the Br–Rh–Br bond axis, has been observed in previously reported structures (Rozière *et al.*, 1979; Acharya *et al.*, 1984). The closest intramolecular contact involving the non-H atoms of pyridines is 2.34 (2) Å [C(16)···C(20)].

From the packing diagram it can be seen that the [Rh(py)₄Br₂]⁺ cations are tightly packed in layers, separated by more loosely populated regions containing the Br[−] anions and the water molecules in an irregular arrangement. Each Br[−] anion has three O atoms close enough for hydrogen bonding. The average O···Br intramolecular contact is 3.342 (8) Å and the average O···O intramolecular contact is 2.82 (6) Å (Table 2). These distances are indicative of hydrogen bonding of the type O–H···Br and O–H···O (Hamilton & Ibers, 1968). In anion/water structures four or six coordination is rare and most atoms have only one, two or three nearest neighbors. This type of anion/water structure has been observed in Cl·6H₂O units [$\langle \text{O} \cdots \text{Cl} \rangle$ 3.14 (2) and $\langle \text{O} \cdots \text{O} \rangle$ 2.8 (2) Å] (Chang & Jeffrey, 1970).

This research has been supported in part by grant RR-8102 from the Division of Research Resources, US National Institutes of Health. We thank the campus computer center for use of computer resources and Dr C. L. Barnes for providing the facilities to obtain the ORTEP and packing diagrams, and for useful discussions.

References

- ACHARYA, K. R., TAVALE, S. S. & GURU, T. N. (1984). *Acta Cryst.* **C40**, 1327–1328.
 CHANG, S. C. & JEFFREY, G. A. (1970). *Acta Cryst.* **B26**, 673–683.
 CLARK, S. F. & PETERSEN, J. D. (1981). *Inorg. Chem.* **20**, 280–281.

- COLAMARINO, P. & ORIOLI, P. (1976). *J. Chem. Soc. Dalton Trans.* pp. 845–848.
- CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1896.
- ENGLISH, R. B. (1979). *Cryst. Struct. Commun.* **8**, 167–172.
- FORD, P. C. & PETERSEN, J. D. (1975). *Inorg. Chem.* **14**, 1404–1408.
- HAMILTON, W. C. & IBERS, J. A. (1968). *Hydrogen Bonding in Solids*, pp. 16–21. New York: W. A. Benjamin.
- International Tables for X-ray Crystallography* (1974). Vol. IV, Tables 2.2B and 2.3.1. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.
- MOTHERWELL, W. D. S. (1976). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- MUIR, M. M. & HUANG, W.-L. (1973). *Inorg. Chem.* **12**, 1831–1835.
- MUIR, M. M., ZINNER, L. B., PAGUAGA, L. A. & TORRES, L. M. (1982). *Inorg. Chem.* **21**, 3448–3450.
- ROZIÈRE, J., LEHMANN, M. S. & POTIER, J. (1979). *Acta Cryst.* **B35**, 1099–1102.
- SANCHEZ, S. (1985). PhD Thesis, Univ. of Puerto Rico, Rio Piedras.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- SKAPSKI, A. C. & STEPHENS, F. A. (1973). *J. Chem. Soc. Dalton Trans.* pp. 1789–1793.

Acta Cryst. (1987). **C43**, 842–844

Structure of *trans*-Dioxo(*o*-oxobenzoato)dipyridineosmium(VI)

BY C. C. HINCKLEY* AND P. A. KIBALA

Department of Chemistry and Biochemistry, Southern Illinois University, Carbondale, IL 62901, USA

AND P. D. ROBINSON

Department of Geology, Southern Illinois University, Carbondale, IL 62901, USA

(Received 30 August 1986; accepted 2 December 1986)

Abstract. $[\text{OsO}_2(\text{C}_7\text{H}_4\text{O}_3)(\text{C}_5\text{H}_5\text{N})_2]$, $M_r = 516.51$, monoclinic, $P2_1/n$, $a = 10.967$ (3), $b = 15.832$ (6), $c = 9.624$ (5) Å, $\beta = 94.27$ (3)°, $V = 1666$ (2) Å³, $Z = 4$, $D_x = 2.06$ g cm⁻³, $\text{Mo K}\alpha$, $\lambda = 0.71069$ Å, $\mu = 81.41$ cm⁻¹, $F(000) = 984$, $T = 295$ K, $R = 0.036$, 1972 unique observed reflections. The Os is bound to six atoms in a distorted octahedron. The *trans* O=Os=O osmyl group is bent at an angle of 169.1 (3)°. The *o*-oxobenzoato group binds to Os through O atoms, forming a six-membered chelate ring which is hinged along its center line in a manner that forces the attached benzene ring 14.9° out of the central plane of the octahedron. The pyridine ligands are bound to Os through their N atoms.

Introduction. Recently we reported the synthesis and spectral characterization of a group of osmyl complexes with hydroxycarboxylic acids (Hinckley & Kibala, 1986). The central feature of the compounds is a chelate ring formed with the osmium atom through the simultaneous binding of the carboxyl and hydroxo functions of the acid. In this paper, we report the structure of one of the group.

There are several classes of osmyl compounds which contain *trans* O=Os=O groups. Among them are salts, such as $\text{K}_2[\text{OsO}_2(\text{OH})_4]$ (Griffith, 1967), and amino acid complexes, such as $\text{OsO}_2(\text{glycinato})_2$ (Roth &

Hinckley, 1981), in which the osmyl groups are linear. Pyridine-containing Os^{VI} monoesters are asymmetric around the osmium atom and contain osmyl groups which are bent (Conn, Kim, Suddath, Blattman & Rich, 1974). In addition, the chelate rings formed by the binding of the diols with the osmium atoms in the monoesters contain five atoms (Brumley & Hinckley, 1980). This structural feature is typical of Os^{VI} complexes (Griffith & Raub, 1980). The compound described in this paper is the first reported example of an osmyl complex containing a six-membered chelate ring.

The initial interest in α -hydroxycarboxylic acid complexes of osmyls, as with the recently reported amino acid complexes (Roth & Hinckley, 1981), is one of existence. Osmyl complexes of amino acids and hydroxycarboxylic acids are potential intermediates in osmium tetroxide oxidations of amino acids and carbohydrates, respectively. The fact that stable osmyl complexes exist for them reveals limits to the oxidizing power of osmyls. Structural features of the compounds which are of particular interest are the linearity of the osmyl group and the chelate ring geometry.

Experimental. Crystal $0.41 \times 0.11 \times 0.03$ mm, transparent, yellow-brown, bladed. Rigaku AFC-5 diffractometer, graphite-monochromatized $\text{Mo K}\alpha$ radiation, ω - 2θ scans, scan speed 6° min^{-1} , maximum of 3 scan repetitions to obtain $\sigma(F)/F < 0.10$. Lattice parameters

* To whom all correspondence should be addressed.