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## 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*

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**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) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 2.06$  g cm<sup>-3</sup>,  $\text{Mo } K\alpha$ ,  $\lambda = 0.71069$  Å,  $\mu = 81.41$  cm<sup>-1</sup>,  $F(000) = 984$ ,  $T = 295$  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<sup>VI</sup> 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<sup>VI</sup> 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  $\alpha$ -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,  $\omega$ - $2\theta$  scans, scan speed  $6^\circ \text{ min}^{-1}$ , maximum of 3 scan repetitions to obtain  $\sigma(F)/F < 0.10$ . Lattice parameters

\* To whom all correspondence should be addressed.

from least-squares fit of 20 reflections in  $2\theta$  range 6–13°. 3230 reflections measured ( $h-13$  to 13,  $k 0$  to 18,  $l 0$  to 11); 3046 reflections unique, 1972 observed with  $I > 3\sigma(I)$ ,  $(\sin\theta/\lambda)_{\max} = 0.595 \text{ \AA}^{-1}$ ,  $R_{\text{int}} = 0.6\%$  for 184 equivalent reflections. Two standard reflections (021 and 230) varied by 0.61 and 0.96% respectively. Data corrected for Lp and absorption (numerical method, transmission range 0.43–0.77). Most non-H atomic positions located by direct methods, remainder by difference Fourier synthesis. Full-matrix least-squares refinement of  $F$  magnitudes performed for all non-H positional and anisotropic thermal parameters; H atoms placed in geometrically correct positions ( $C-H = 0.95 \text{ \AA}$ ) but not refined. Convergence yielded  $R = 0.036$ ,  $wR = 0.042$  [ $w = 1/\sigma^2(|F_o|)$ ],  $S = 1.18$ , and  $(\Delta/\sigma)_{\max} = 0.0009$ . Final difference synthesis showed  $(\Delta\rho)_{\max} = 1.21$  and  $(\Delta\rho)_{\min} = -1.39 \text{ e \AA}^{-3}$ , both near the Os position. No extinction corrections; atomic scattering factors and anomalous-dispersion corrections from Cromer & Waber (1974). All computer programs from the TEXSAN structure analysis package (Molecular Structure Corporation, 1985).

**Discussion.** Positional and equivalent isotropic thermal parameters are given in Table 1 while Table 2 lists relevant interatomic distances and angles.\*

The Os atom of the complex is six-coordinate with the bonding ligand atoms arranged in a distorted octahedron (Fig. 1). The nitrogen atoms of the pyridine ligands, N(1) and N(2), the binding oxygen atoms of the salicylate ion, O(3) and O(4), and the Os atom form the central plane of the molecule which also includes the carbon atom of the carboxylate group, C(1). Various least-squares planes were calculated, together with dihedral angles between them. The dihedral angle between the central plane and that of the pyridine ligand containing N(1) is 90.52°, while the angle with the second pyridine is 102.50°. The initial accommodation of the six-membered chelate ring formed by the salicylate ligand is a 94.8(3)° O(3)–Os–O(4) bond angle. The remaining angles around Os in the central plane are less than 90°. The plane formed by the phenyl group, the carboxylate carbon, C(1), and the oxo oxygen, O(4) is flexed 14.89° out of the central plane along an O(4)–C(1) hinge line. The plane containing the osmyl group, O(1)–Os–O(2), forms a 90.33° dihedral angle with the central plane, but the osmyl is not linear. The O(1)–Os–O(2) angle is 169.8(3)°. The oxygen atoms of the osmyl are directed between the pyridine ligands. The molecules interact through normal van der Waals forces.

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43610 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Positional and equivalent isotropic thermal parameters for the non-H atoms and their e.s.d.'s

$$B_{\text{eq}} = (8\pi^2/3)(U_{11} + U_{22} + U_{33} + 2U_{12}\cos\gamma + 2U_{13}\cos\beta + 2U_{23}\cos\alpha).$$

	x	y	z	$B_{\text{eq}}(\text{\AA}^2)$
Os	0.598792 (38)	0.113131 (27)	0.751197 (44)	2.99 (2)
O(1)	0.61512 (66)	0.14107 (44)	0.92524 (73)	3.6 (3)
O(2)	0.60930 (71)	0.09055 (47)	0.57579 (72)	4.1 (4)
O(3)	0.48815 (68)	0.21232 (45)	0.70891 (81)	4.1 (4)
O(4)	0.46556 (67)	0.03260 (44)	0.76974 (81)	3.8 (4)
O(5)	0.31390 (80)	0.28026 (53)	0.69464 (97)	5.5 (5)
N(1)	0.72424 (72)	0.01399 (51)	0.80129 (82)	2.8 (4)
N(2)	0.75251 (78)	0.19585 (53)	0.72846 (87)	3.2 (4)
C(1)	0.3660 (12)	0.21184 (79)	0.6990 (12)	4.1 (6)
C(2)	0.2979 (10)	0.13062 (60)	0.6910 (10)	3.2 (5)
C(3)	0.3478 (10)	0.05114 (78)	0.7231 (10)	3.5 (5)
C(4)	0.2699 (11)	−0.01999 (76)	0.7090 (12)	4.2 (6)
C(5)	0.1503 (11)	−0.01334 (87)	0.6681 (14)	4.8 (6)
C(6)	0.0991 (11)	0.0660 (10)	0.6374 (13)	5.2 (7)
C(7)	0.1725 (11)	0.13575 (75)	0.6502 (12)	4.2 (6)
C(8)	0.7681 (10)	−0.03521 (67)	0.7051 (11)	3.3 (5)
C(9)	0.8538 (12)	−0.09608 (68)	0.7340 (13)	4.3 (6)
C(10)	0.8986 (11)	−0.10927 (77)	0.8709 (14)	4.6 (6)
C(11)	0.8537 (14)	−0.05943 (96)	0.9727 (13)	6.3 (8)
C(12)	0.7683 (13)	0.00092 (83)	0.9330 (13)	5.2 (7)
C(13)	0.7883 (11)	0.21845 (84)	0.6012 (12)	4.5 (6)
C(14)	0.8830 (13)	0.27179 (95)	0.5833 (14)	5.6 (7)
C(15)	0.9482 (12)	0.30467 (85)	0.6943 (16)	5.4 (7)
C(16)	0.9180 (12)	0.28158 (86)	0.8269 (14)	5.1 (7)
C(17)	0.8195 (11)	0.22656 (78)	0.8378 (12)	4.2 (6)

Table 2. Bond distances (Å), bond angles (°), and e.s.d.'s for the non-hydrogen atoms

Os	O(1)	1.729 (7)	C(2)	C(3)	1.40 (1)		
Os	O(2)	1.738 (7)	C(2)	C(7)	1.40 (2)		
Os	O(4)	1.957 (7)	C(3)	C(4)	1.41 (2)		
Os	O(3)	2.008 (7)	C(4)	C(5)	1.35 (2)		
Os	N(1)	2.119 (8)	C(5)	C(6)	1.40 (2)		
Os	N(2)	2.159 (9)	C(6)	C(7)	1.37 (2)		
O(3)	C(1)	1.34 (1)	C(8)	C(9)	1.36 (1)		
O(4)	C(3)	1.37 (1)	C(9)	C(10)	1.39 (2)		
O(5)	C(1)	1.22 (1)	C(10)	C(11)	1.38 (2)		
N(1)	C(8)	1.33 (1)	C(11)	C(12)	1.37 (2)		
N(1)	C(12)	1.34 (1)	C(13)	C(14)	1.36 (2)		
N(2)	C(17)	1.33 (1)	C(14)	C(15)	1.35 (2)		
N(2)	C(13)	1.36 (1)	C(15)	C(16)	1.39 (2)		
C(1)	C(2)	1.49 (1)	C(16)	C(17)	1.40 (2)		
O(1)	Os	O(2)	169.8 (3)	O(5)	C(1)	O(3)	117 (1)
O(1)	Os	O(4)	95.9 (3)	O(5)	C(1)	C(2)	122 (1)
O(1)	Os	O(3)	90.8 (3)	O(3)	C(1)	C(2)	120 (1)
O(1)	Os	N(1)	87.2 (3)	C(3)	C(2)	C(7)	118 (1)
O(1)	Os	N(2)	85.3 (3)	C(3)	C(2)	C(1)	126 (1)
O(2)	Os	O(4)	93.4 (3)	C(7)	C(2)	C(1)	116 (1)
O(2)	Os	O(3)	92.7 (3)	O(4)	C(3)	C(2)	128 (1)
O(2)	Os	N(1)	88.8 (3)	O(4)	C(3)	C(4)	114 (1)
O(2)	Os	N(2)	85.3 (3)	C(2)	C(3)	C(4)	118 (1)
O(4)	Os	O(3)	94.8 (3)	C(5)	C(4)	C(3)	122 (1)
O(4)	Os	N(1)	88.5 (3)	C(4)	C(5)	C(6)	120 (1)
O(4)	Os	N(2)	176.7 (3)	C(7)	C(6)	C(5)	119 (1)
O(3)	Os	N(1)	176.3 (3)	C(6)	C(7)	C(2)	122 (1)
O(3)	Os	N(2)	88.3 (3)	N(1)	C(8)	C(9)	124 (1)
N(1)	Os	N(2)	88.5 (3)	C(8)	C(9)	C(10)	119 (1)
C(1)	O(3)	Os	126.7 (7)	C(11)	C(10)	C(9)	118 (1)
C(3)	O(4)	Os	121.6 (7)	C(12)	C(11)	C(10)	118 (1)
C(8)	N(1)	C(12)	116 (1)	N(1)	C(12)	C(11)	124 (1)
C(8)	N(1)	Os	122.6 (7)	C(14)	C(13)	N(2)	123 (1)
C(12)	N(1)	Os	120.8 (7)	C(15)	C(14)	C(13)	120 (1)
C(17)	N(2)	C(13)	116 (1)	C(14)	C(15)	C(16)	119 (1)
C(17)	N(2)	Os	122.1 (7)	C(15)	C(16)	C(17)	118 (1)
C(13)	N(2)	Os	122.0 (7)	N(2)	C(17)	C(16)	124 (1)

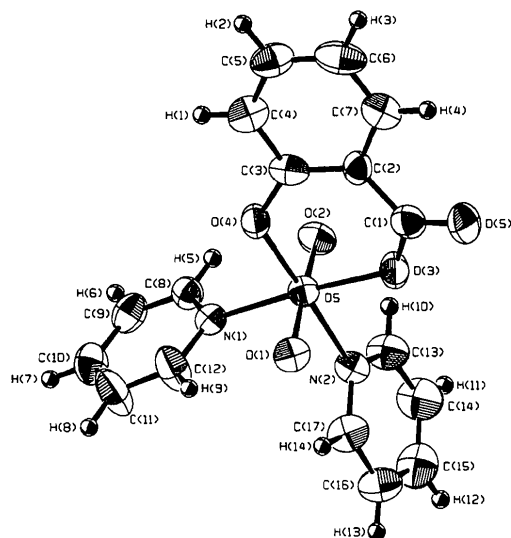


Fig. 1. Molecular structure and numbering scheme.

$\alpha$ -Hydroxycarboxylate complexes of osmyls comprise a new class of osmyl-containing compounds. The general structural characteristics of the complex studied in this paper are consistent with osmyls in general, and spectroscopy (Hinckley & Kibala, 1986) indicates that this compound is broadly representative of the class. The six-membered chelate ring found in

this compound is unusual. The larger ring size is accommodated by an expansion of the interior bond angles within the chelate ring, accompanied by a flex in the ring to relieve strain. The supposed electronic stabilization of a planar unflexed molecule is evidently unimportant in this case. The structure of this single compound is consistent with the notion that the small chelate ring size which characterizes osmyl complexes of all classes is due to steric factors arising out of the size of the osmium atom, and to chemical constraints associated with their synthesis.

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## Structure of Sodium *N*-(*p*-Aminobenzenesulfonyl)acetamide Monohydrate (Sodium Sulfacetamide Monohydrate)

BY H. C. PATEL AND T. P. SINGH

*Department of Biophysics, All India Institute of Medical Sciences, New Delhi 110 029, India*

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**Abstract.**  $\text{Na}^+ \cdot \text{C}_8\text{H}_{10}\text{N}_2\text{O}_3\text{S}^- \cdot \text{H}_2\text{O}$ ,  $M_r = 255.24$ , monoclinic,  $P2_1/c$ ,  $a = 6.617$  (2),  $b = 23.771$  (4),  $c = 7.014$  (1) Å,  $\beta = 103.12$  (3)°,  $V = 1074.2$  (2) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.580$  (1),  $D_x = 1.578$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 2.48$  mm<sup>-1</sup>,  $F(000) = 532$ ,  $T = 293$  K, final  $R = 0.069$  for 2161 observed reflections. The asymmetric unit contains one sulfacetamide molecule, one Na atom and one water molecule. The Na atom has fivefold coordination in the structure. The bonding around the S atom is slightly distorted from the ideal tetrahedral symmetry. The sulfacetamide molecule shows a *gauche* conformation about the S–N bond

with a torsion angle of 81.3 (5)°. The crystal structure is stabilized by an extended network of interdigitating hydrogen bonds, van der Waals forces and intermolecular coordinations through Na atoms.

**Introduction.** Recent studies on the action of sulfonamides on enzymatic extracts obtained from different bacterial species (McCullough & Maren, 1973; Ho, Corman, Morse & Schneider, 1975) have definitely shown that the target enzyme catalyses the formation of dihydropteroate from *p*-aminobenzoate (PAB) and (hydroxymethyl)dihydropteridine pyrophosphate