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Structure of N,N,N',N' -Tetramethylethylenediammonium Tetrachlorodioxosmate(VI)

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Abstract. $C_6H_{18}N_4^{2+}OsCl_4O_2^{2-}$, $M_r = 482.2$, monoclinic, $P2_1/n$, $a = 8.914(4)$, $b = 11.326(2)$, $c = 14.061(2)$ Å, $\beta = 94.74(2)^\circ$, $U = 1414.7$ Å³, $Z = 4$, $D_m = 2.21(3)$, $D_x = 2.263$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 96.98$ cm⁻¹, $F(000) = 912$, room temperature, final $R = 0.032$ for 2928 reflections [$F > 2\sigma(F)$]. The structure contains *trans*-[OsO₂Cl₂]²⁻ anions [Os–O = 1.718(4), 1.728(4), Os–Cl = 2.381(1)–2.397(1) Å] and [Me₂HNCH₂CH₂NHMe₂]²⁺ cations. The anion approximates to the D_{4h} point group and the cation has approximate C_i symmetry. There is hydrogen bonding between N and Cl atoms [minimum N(H)⋯Cl 3.221(6) Å]. There are no hydrogen bonds involving O atoms.

Introduction. The reaction of potassium osmate(VI), $K_2[OsO_2(OH)_4]$, with bidentate amine hydrohalides in water produces dioxosmium(VI) complexes, for example $[OsO_2X_2(\text{Me}_2\text{NCH}_2\text{CH}_2\text{NMe}_2)]$ ($X = \text{Cl}$ or Br) from $[\text{Me}_2\text{HNCH}_2\text{CH}_2\text{NHMe}_2]X_2$ (Harbron & Levason, 1987). Various attempts to produce single crystals of these materials by reaction over several days mostly produced orange-brown powders but one preparation afforded a few air-stable brown crystals, one of which was used for the present study.

Experimental. Density measured by flotation in carbon tetrachloride/1,2-dibromoethane. Preliminary data from photographic X-ray examination and accurate cell dimensions from 25 accurately centred reflections using an Enraf–Nonius CAD-4 diffractometer fitted with

graphite monochromator and Mo radiation. Intensities of 3796 reflections were recorded [$1 < \theta < 28^\circ$; $h(0-11)$, $k(0-14)$, $l(-18$ to 18)] using a crystal $0.4 \times 0.1 \times 0.1$ mm. The standard reflection showed decay (19%) during the experiment and this was allowed for in the data reduction. An empirical ψ -scan absorption correction was applied. (Transmission: max. 99.9, min. 85.9%). Systematic absences: $h0l$, $h + l \neq 2n$; $0k0$, $k \neq 2n$. After data reduction 3402 unique reflections remained, $R_{\text{int}} = 0.022$, of which 2933 with $F > 2\sigma(F)$ were used in the analysis. Two possible Os positions were identified from the Patterson synthesis and by repeated structure factor and electron density syntheses the correct Os position and the remaining non-H atoms were located. Evidence for some of the H atoms came from a difference electron density synthesis and H atoms bonded to C were introduced in geometrically calculated positions [$d(\text{C}-\text{H}) = 1.0$ Å] with a common refined temperature factor. Five reflections (020, $\bar{1}11$, 002, $\bar{1}13$, $\bar{2}02$) apparently affected by extinction were removed and full-matrix least-squares refinement minimizing $\sum w(\Delta F)^2$ converged to $R = 0.032$ {109 parameters, 2928 reflections, anisotropic (Os, Cl, O) and isotropic (N, C, H) atoms, $w = 1/[\sigma^2(F) + 0.0006F^2]$, max. shift/e.s.d. 0.4, $wR = 0.041$ }. The residual electron density was in the range 0.96 to -1.84 e Å⁻³. The H atoms bonded to N were not introduced into the model. Scattering factors for neutral atoms and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974). All calculations were carried out using *SHELX76* (Sheldrick, 1976), *ORTEP* (Johnson, 1965), *PLUTO* (Motherwell & Clegg, 1978) and *GEOM* (Mallinson, 1984; Mallinson & Muir, 1985) on an ICL

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Table 1. Atomic coordinates and isotropic temperature factors ($\text{\AA}^2 \times 10^3$)

	x	y	z	U
Os	0.56219 (2)	0.24115 (2)	0.79051 (1)	24.1 (1)*
Cl(1)	0.4056 (2)	0.3595 (1)	0.6813 (1)	36.8 (7)*
Cl(2)	0.7198 (2)	0.4049 (1)	0.8363 (1)	48.1 (9)*
Cl(3)	0.4045 (2)	0.0787 (1)	0.7421 (1)	46.5 (8)*
Cl(4)	0.7122 (2)	0.1191 (2)	0.8987 (1)	43.2 (8)*
O(1)	0.6744 (5)	0.2092 (4)	0.7003 (3)	40.7 (23)*
O(2)	0.4452 (5)	0.2770 (4)	0.8782 (3)	39.5 (23)*
N(1)	0.4613 (6)	-0.2572 (4)	0.5824 (4)	30.0 (10)
N(2)	0.4127 (5)	-0.2939 (4)	0.8457 (3)	31.1 (10)
C(1)	0.3778 (8)	-0.3611 (6)	0.5419 (5)	45.1 (16)
C(2)	0.4348 (9)	-0.1531 (7)	0.5192 (5)	51.7 (18)
C(3)	0.4222 (7)	-0.2258 (5)	0.6801 (4)	30.0 (11)
C(4)	0.4632 (6)	-0.3256 (5)	0.7506 (4)	27.5 (11)
C(5)	0.4283 (8)	-0.3971 (6)	0.9115 (5)	46.6 (16)
C(6)	0.4984 (8)	-0.1877 (6)	0.8883 (5)	45.2 (16)

* Equivalent isotropic temperature factor from anisotropic thermal parameters: $U_{eq} = \frac{1}{3} \text{trace } U$.

2976 computer. The atomic parameters for non-H atoms are reported in Table 1.*

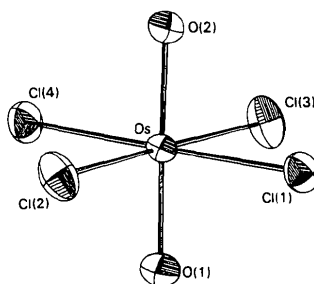
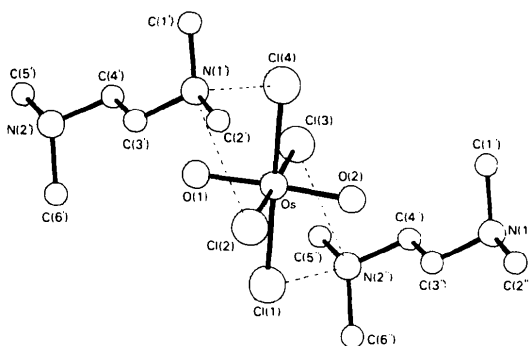
Discussion. Bond lengths and selected angles for the title compound are listed in Table 2. Figs. 1 and 2 show the anion and H-bonding arrangement respectively. The $[\text{OsO}_2\text{Cl}_4]^{2-}$ anion has been characterized in $\text{K}_2[\text{OsO}_2\text{Cl}_4]$ (Kruse, 1961). In the present example the anion has no crystallographic symmetry but approximates to the D_{4h} point group with a maximum angular deviation from the expected 90° of $2.9(2)^\circ$. The Os—Cl bonds show a spread of values differing by $0.016(2) \text{ \AA}$, presumably due to packing or H-bonding forces. The tetramethyldiammonium cation has been reported on a few occasions (Bisi-Castellani, Manotti-Lanfredi, Tiripicchio, Maresca & Natile, 1984) and the present geometry is in good agreement with the earlier studies, with the ion showing approximate C_i symmetry (Fig. 2). The anion and cation pack like the ions in the sodium chloride structure and there are short $\text{N(H)} \cdots \text{Cl}$ distances ($< 3.5 \text{ \AA}$) indicative of H bonding. Each N atom is H bonded to two Cl atoms and every Cl atom is involved in one H bond. Calculations in which an H atom bonded to N was introduced in a calculated position [$d(\text{N-H}) = 1.0 \text{ \AA}$] gave reasonable $\text{N-H} \cdots \text{Cl}$ angles in the range 115 to 164° for the unsymmetrical bifurcated H-bonding system. There are no $\text{N(H)} \cdots \text{O}$ hydrogen bonds present.

The X-ray analysis was performed on a minor crystalline product from the reaction mixture (*cf.* Bisi-Castellani *et al.*, 1984). Infrared spectroscopy

confirmed the presence of two materials since the principal product in our reaction, $[\text{OsO}_2\text{Cl}_2(\text{Me}_2\text{NCH}_2\text{CH}_2\text{NMe}_2)]$, has vibrational frequencies of $319 [\nu(\text{Os-Cl})]$ and $861 [\nu(\text{Os-O})] \text{ cm}^{-1}$ compared with 298 and 847 cm^{-1} for the title compound, $[\text{Me}_2\text{HNCH}_2\text{CH}_2\text{NHMe}_2][\text{OsO}_2\text{Cl}_4]$. The shift of 10 cm^{-1} in $\nu(\text{Os-Cl})$ frequency from 308 cm^{-1} in $\text{K}_2[\text{OsO}_2\text{Cl}_4]$ (Griffith, 1969) to 298 cm^{-1} may be correlated with the $\text{N(H)} \cdots \text{Cl}$ H bonding.

Table 2. Bond lengths (\AA) and selected angles ($^\circ$)

Os—Cl(1)	2.397 (1)	N(1)—C(1)	1.481 (8)
Os—Cl(2)	2.383 (2)	N(1)—C(2)	1.484 (8)
Os—Cl(3)	2.381 (1)	N(1)—C(3)	1.489 (7)
Os—Cl(4)	2.382 (1)	N(2)—C(4)	1.490 (7)
Os—O(1)	1.718 (4)	N(2)—C(5)	1.490 (8)
Os—O(2)	1.728 (4)	N(2)—C(6)	1.521 (8)
		C(3)—C(4)	1.527 (8)
O(1)—Os—Cl(1)	89.3 (2)	O(2)—Os—Cl(4)	91.2 (2)
O(1)—Os—Cl(2)	89.8 (2)	Cl(1)—Os—Cl(2)	92.1 (1)
O(1)—Os—Cl(3)	89.5 (2)	Cl(1)—Os—Cl(3)	87.1 (1)
O(1)—Os—Cl(4)	91.1 (2)	Cl(2)—Os—Cl(4)	89.5 (1)
O(2)—Os—Cl(1)	88.4 (2)	Cl(3)—Os—Cl(4)	91.2 (1)
O(2)—Os—Cl(2)	90.1 (2)	O(1)—Os—O(2)	177.7 (2)
O(2)—Os—Cl(3)	90.5 (2)	Cl(2)—Os—Cl(3)	179.0 (1)
		Cl(1)—Os—Cl(4)	178.3 (1)

Fig. 1. The $[\text{OsO}_2\text{Cl}_4]^{2-}$ anion with ellipsoids drawn with 40% probability surfaces.Fig. 2. Diagram showing anion-to-cation hydrogen bonding (---). Symmetry code: (i) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$; (ii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond angles, torsion angles and selected non-bonded distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43285 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Bis(5,7-dichloro-8-quinolinolato-*N,O*)bis(pyridine)nickel(II)

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Abstract. $[\text{Ni}(\text{C}_9\text{H}_4\text{Cl}_2\text{NO})_2(\text{C}_5\text{H}_5\text{N})_2]$, $M_r = 643.00$, monoclinic, space group $P2_1/c$, $a = 12.6796$ (16), $b = 16.0408$ (16), $c = 14.5342$ (21) Å, $\beta = 113.28$ (1)°, $V = 2715.4$ (6) Å³, $Z = 4$, $D_m = 1.57$ (1), $D_x = 1.57$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.54184$ Å, $\mu = 5.022$ mm⁻¹, $F(000) = 1304$, $T = 290$ K, final $R = 0.058$, $wR = 0.060$ for 2177 observed reflections. The central Ni atom is pseudo-octahedrally coordinated by two 5,7-dichloro-8-quinolinol ligands and two pyridine ligands in *cis* positions. Distances and angles are normal [average Ni–N(quinoline) 2.080 (7), Ni–N(pyridine) 2.094 (8); Ni–O 2.045 (6) Å].

Introduction. The present paper describes the crystal structure of a new Ni^{II} compound with 5,7-dichloro-8-quinolinol. In previous work, we have reported several structures of Ni coordination compounds having C-substituted ethylenediamines or 5-chloro-8-quinolinol as ligands (García-Granda & Gómez-Beltrán, 1986). This investigation has been undertaken in order to obtain enough data to understand the

chemical and physicochemical behaviour of these transition-metal compounds.

Experimental. A dark yellow crystal of approximately 0.18 × 0.10 × 0.10 mm was used during the measurements. Density measured by flotation in carbon tetrachloride/heptane. Throughout the experiment Cu $K\alpha$ radiation was used with a graphite crystal monochromator on a Nonius CAD-4 single-crystal diffractometer. The unit-cell dimensions were determined from the angular settings of 25 reflections. The intensity data of 10 734 reflections (one half sphere up to $\theta = 70^\circ$), hkl range from (–15, –19, 0) to (15, 19, 17), were measured, using the ω – 2θ scan technique, with a scan angle of 1.50° and a variable scan rate with a maximum scan time of 60 s per reflection. The intensity of the primary beam was checked throughout the data collection by monitoring three standard reflections every 30 min. The final drift correction factors were between 1.00 and 1.05. On all reflections profile analysis was performed (Lehmann & Larsen, 1974; Grant & Gabe, 1978); empirical absorption correction was applied, using ψ scans (North, Phillips & Mathews, 1968) (correction factors were in the range 0.91 to 1.00). Symmetry-equivalent reflections were averaged, $R_{\text{int}} = \sum(I - \langle I \rangle) / \sum I = 0.054$, resulting in 5146 unique reflections of which 2177 were observed with $I > 3\sigma(I)$.

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