

Absorption correction: $h = 0 \rightarrow 8$
 ψ scan $k = -11 \rightarrow 11$
 $T_{\min} = 0.0898$, $T_{\max} = 0.1387$ $l = -15 \rightarrow 15$
 3360 measured reflections 3 standard reflections
 3078 independent reflections monitored every 97 reflections
 2773 observed reflections intensity decay: none
 $[I > 2\sigma(I)]$

Refinement

Refinement on F^2 $(\Delta/\sigma)_{\max} = 0.001$
 $R[F^2 > 2\sigma(F^2)] = 0.0230$ $\Delta\rho_{\max} = 0.638 \text{ e } \text{\AA}^{-3}$
 $wR(F^2) = 0.0502$ $\Delta\rho_{\min} = -0.829 \text{ e } \text{\AA}^{-3}$
 $S = 1.077$ Extinction correction: none
 3078 reflections Atomic scattering factors
 217 parameters from *International Tables*
 H atoms refined as riding for *Crystallography* (1992),
 $w = 1/[\sigma^2(F_o^2) + (0.0289P)^2]$ Vol. C, Tables 4.2.6.8 and
 where $P = (F_o^2 + 2F_c^2)/3$ 6.1.1.4)

C3—N3—C7	105.3 (4)	C6—C7—N3	109.7 (4)
C3—N3—Pt	123.9 (3)	C6—C7—C8	128.3 (5)
C7—N3—Pt	129.8 (3)	N3—C7—C8	122.0 (4)

Data collection: Siemens P3VAX system. Cell refinement: Siemens P3VAX system. Data reduction: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SZ1032). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
Pt	0.05135 (3)	0.28862 (2)	0.11701 (1)	0.03267 (7)
Cl	-0.2444 (2)	0.3823 (1)	0.0999 (1)	0.0500 (3)
N1	0.3091 (6)	0.2016 (4)	0.1199 (3)	0.045 (1)
C1	0.3444 (9)	0.1550 (6)	0.0108 (4)	0.054 (1)
C2	0.2940 (9)	0.2631 (6)	-0.0622 (4)	0.053 (1)
N2	0.0962 (7)	0.3028 (4)	-0.0382 (3)	0.045 (1)
N3	0.0199 (6)	0.2627 (4)	0.2715 (3)	0.0359 (9)
C3	-0.1110 (7)	0.1745 (5)	0.3104 (4)	0.038 (1)
S	-0.3066 (2)	0.0710 (1)	0.2618 (1)	0.0462 (3)
C4	-0.3393 (8)	0.0127 (6)	0.3878 (4)	0.051 (1)
C5	-0.2107 (8)	0.0695 (5)	0.4594 (4)	0.047 (1)
N4	-0.0799 (6)	0.1630 (4)	0.4143 (3)	0.041 (1)
C6	0.0814 (8)	0.2482 (5)	0.4449 (4)	0.045 (1)
C7	0.1421 (7)	0.3087 (5)	0.3566 (4)	0.038 (1)
C8	0.3113 (8)	0.4080 (5)	0.3452 (4)	0.041 (1)
C9	0.2855 (9)	0.5273 (5)	0.2945 (4)	0.053 (1)
C10	0.4442 (9)	0.6204 (6)	0.2854 (5)	0.061 (2)
C11	0.6243 (9)	0.5949 (6)	0.3246 (4)	0.056 (2)
C12	0.6504 (8)	0.4776 (6)	0.3744 (4)	0.053 (1)
C13	0.4927 (8)	0.3836 (5)	0.3866 (4)	0.045 (1)
N5	0.1489 (7)	-0.0980 (5)	0.2393 (4)	0.048 (1)
O1	0.2435 (6)	-0.0079 (4)	0.2920 (3)	0.064 (1)
O2	0.0363 (6)	-0.1817 (4)	0.2801 (4)	0.071 (1)
O3	0.1696 (9)	-0.1018 (5)	0.1440 (4)	0.094 (2)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Pt—N3	2.017 (4)	C3—N4	1.347 (6)
Pt—N2	2.026 (4)	C3—S	1.716 (5)
Pt—N1	2.029 (4)	S—C4	1.745 (5)
Pt—Cl	2.305 (1)	C4—C5	1.331 (7)
N1—C1	1.491 (7)	C5—N4	1.397 (6)
C1—C2	1.497 (8)	N4—C6	1.382 (6)
C2—N2	1.484 (7)	C6—C7	1.360 (7)
N3—C3	1.330 (6)	C7—C8	1.483 (7)
N3—C7	1.393 (6)		
N3—Pt—N2	175.6 (2)	N3—C3—N4	111.4 (4)
N3—Pt—N1	92.0 (2)	N3—C3—S	136.3 (4)
N2—Pt—N1	84.0 (2)	N4—C3—S	112.3 (3)
N3—Pt—Cl	92.0 (1)	C3—S—C4	88.8 (2)
N2—Pt—Cl	91.9 (1)	C5—C4—S	113.8 (4)
N1—Pt—Cl	175.5 (1)	C4—C5—N4	111.0 (5)
Cl—N1—Pt	107.6 (3)	C3—N4—C6	107.6 (4)
N1—C1—C2	108.5 (4)	C3—N4—C5	114.0 (4)
N2—C2—C1	108.1 (4)	C6—N4—C5	138.3 (4)
C2—N2—Pt	110.4 (3)	C7—C6—N4	106.0 (4)

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Dipyridiniomethane *cis*- and *trans*-Difluorotetrachloroosmate(IV), *cis*- and *trans*- $[(C_5H_5N)_2CH_2][OsCl_4F_2]$

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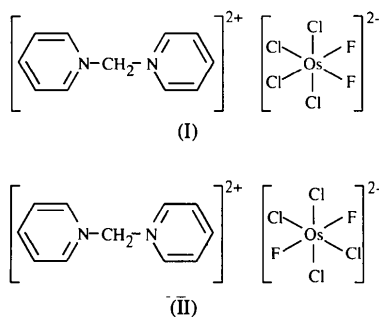
Abstract

In the structures of *N,N'*-methylene-dipyridinium *cis*-difluorotetrachloroosmate(IV), (I), and *N,N'*-methylene-dipyridinium *trans*-difluorotetrachloroosmate(IV),

(II), *cis*- and *trans*- $[(C_5H_5N)_2CH_2][OsCl_4F_2]$, the *trans* influence in the F—Os—Cl axes of (I) lengthens the average Os—F distance to 1.948 Å and shortens the average Os—Cl distance to 2.316 Å, compared with those of the symmetrically coordinated axes F—Os—F in (II) and Cl—Os—Cl in (I) and (II), respectively, with mean values of 1.926 and 2.337 Å observed for Os—F and Os—Cl, respectively.

Comment

Octahedrally coordinated complexes of the type $[MF_nCl_{6-n}]^{2-}$ ($M = Os, Ir, Pt; n = 0-6$) have been synthesized systematically *via* stereospecific substitution reactions and characterized using vibrational and NMR spectroscopy to examine bond interactions due to the mutual *trans* influence of F and Cl (Preetz, Ruf & Tensfeldt, 1984; Groth & Preetz, 1987; Alyoubi, Greenslade, Foster & Preetz, 1990; Parzich, Peters & Preetz, 1993). However, the *trans* influence on bond distances in mixed halogeno complexes had not been studied extensively until recently because of statistical disorder of the ligands in high-symmetry A_2B lattices with alkali metal or spherical alkylammonium cations (Keller & Homborg, 1976). Obviously, ordered structures can be obtained if bent and doubly charged cations like dipyridiniomethane (Brüdgam & Hartl, 1986) are used to form *AB*-type salts (Bruhn & Preetz, 1994*a,b*, 1995*a,b*; Bruhn, Drews, Meynhardt & Preetz, 1995). In the course of our work on mixed F/Cl complexes of Os^{IV} , we isolated the title compounds (I) and (II) by ion-exchange chromatography.



Compound (I) crystallizes in space group $P2_1/n$ with two independent formula units in the asymmetric unit and with all atoms in general positions. The complex anion whose atoms have 1 as the first digit in their label is completely ordered. The Os1—Cl13 [2.341 (2) Å] and Os1—Cl14 [2.335 (2) Å] distances are typical for symmetrically coordinated Cl—Os—Cl axes and comparable with those axes in (II). The mutual *trans* influence of Cl and F is noticeable by a slight shortening of the Os1—Cl12 [2.306 (2) Å] and Os1—Cl11 [2.327 (2) Å] bonds, whereas the Os1—F11 [1.945 (4) Å] and Os1—F12 [1.946 (4) Å] distances are lengthened compared

with the values of the symmetrically coordinated axes in (II). The complex anion whose atoms have 2 as the first digit in their label exhibits unusual values for the anisotropic displacement parameters of F22 and Cl24 and the Os2—F22 [2.054 (4) Å] and Os2—Cl24 [2.288 (3) Å] bond distances. This is related to a packing disorder affecting these particular positions, presumably as a result of a 90° tilting around the Cl21—Os2—F21 axis. The Os2—Cl21 [2.316 (2) Å] and Os2—Cl22 [2.322 (2) Å] distances show typical values for Os—Cl bonds shortened by *trans* influence, and Os2—F21 is concomitantly lengthened to 1.953 (4) Å. In both anions the expected octahedral geometry is slightly distorted due to the larger atomic radii of the Cl ligands compared with F. The Cl13—Os1—Cl14 and Cl23—Os2—Cl24 angles are bent to 174.94 (8) and 173.80 (9)°, avoiding contact with Cl11, Cl12 and Cl21, Cl22, respectively.

The almost perfect ordering of the mixed-ligand complex anions in the lattice is presumably conferred by the bent geometry of the dipyridiniomethane dication. In addition, the free orientation of the pyridine rings around the *CM*—N axes enables optimal adaptivity to the steric and topologic features of the anions. The normals to the planes through the pyridine rings carrying the labels *A* and *B* form angles of 56.9 (7) and 62.8 (7)°

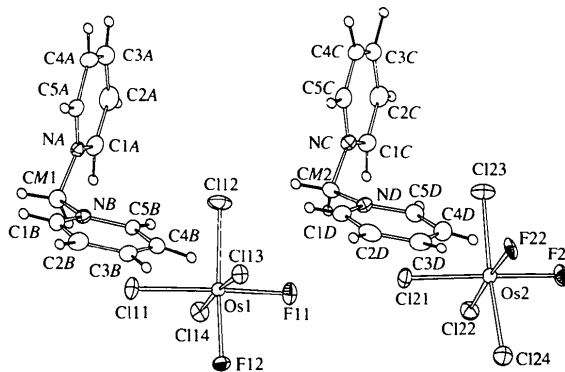


Fig. 1. View of an asymmetric unit of (I) with the atom-labelling scheme. Ellipsoids represent the 30% probability level; H atoms are drawn as spheres of arbitrary radii.

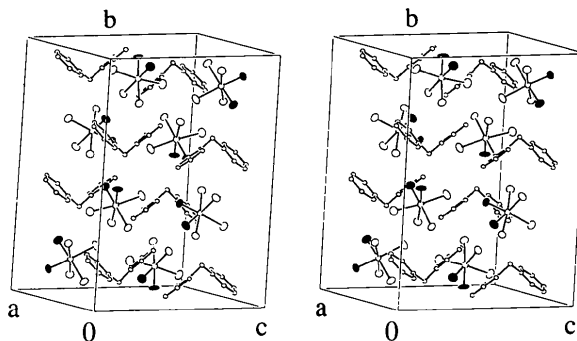


Fig. 2. Stereoscopic view of (I) with H atoms omitted for clarity.

with the plane through NA, CM1 and NB. The pyridine rings labelled C and D exhibit corresponding angles of 68.0(7) and 56.2(7)°, respectively, with the plane through NC, CM2 and ND.

Compound (II) crystallizes in space group $P\bar{1}$ with two independent Os atoms residing on inversion centres [Os1 on (0,0,0) and Os2 on (1/2,1/2,1/2)]. The bond distances within the linear F—Os—F axis are similar, as are those within the Cl—Os—Cl axis, with averaged values of 1.926 Å for Os—F and 2.340 Å for Os—Cl. In both complex anions, the axes are almost perfectly orthogonal.

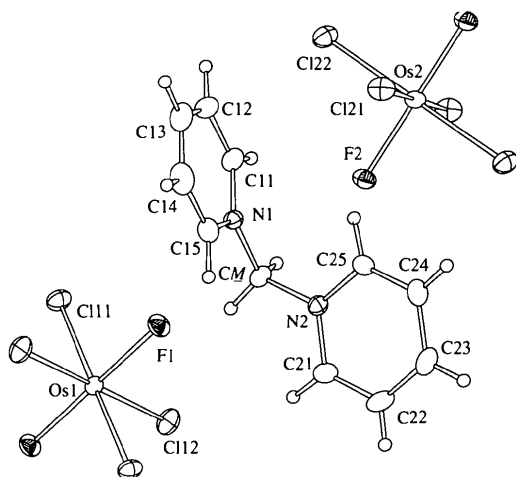


Fig. 3. View of both independent complex anions and the dication of (II) with the atom-labelling scheme. Ellipsoids represent the 30% probability level; H atoms are drawn as spheres of arbitrary radii.

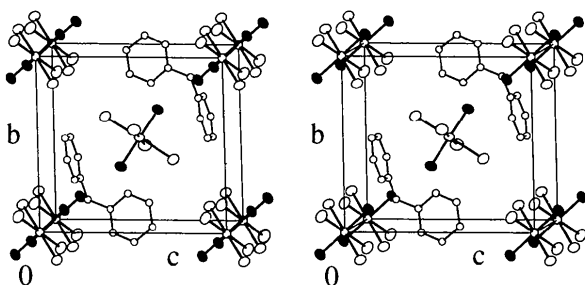


Fig. 4. Stereoscopic view of (II) with H atoms omitted for clarity.

In the dipyridiniomethane dication the angles between the normals to the pyridine rings make angles of 62.8(2) and 40.2(3)° with the normal to the plane through CM, N1 and N2.

Experimental

By the reaction of $[\text{OsCl}_6]^{2-}$ with BrF_3 at room temperature, a mixture containing several compounds of the series $[\text{OsF}_n\text{Cl}_{6-n}]^{2-}$ ($n = 0-6$) is formed, from which $\text{cis-}[\text{OsCl}_4\text{F}_2]^{2-}$ and $\text{cis-}[\text{OsF}_4\text{Cl}_2]^{2-}$ have been separated by ion-

exchange chromatography on diethylaminoethyl-cellulose and finally isolated as their tetra-*n*-butylammonium (TBA) salts. By stereospecific ligand exchange with HCl gas in CH_2Cl_2 at 278 K, $\text{cis-}[\text{OsF}_4\text{Cl}_2]^{2-}$ is converted to $\text{trans-}[\text{OsCl}_4\text{F}_2]^{2-}$ in 50% yield (Preetz, Ruf & Tensfeldt, 1984). The dipyridiniomethane salts were precipitated from a *N,N*-dimethylformamide solution of the TBA salts using dipyridiniomethane dibromide, $[(\text{C}_5\text{H}_5\text{N})_2\text{CH}_2]\text{Br}_2$, and recrystallized from water/acetone to give suitable single crystals.

Compound (I)

Crystal data

$(\text{C}_{11}\text{H}_{12}\text{N}_2)[\text{OsCl}_4\text{F}_2]$

$M_r = 542.23$

Monoclinic

$P2_1/n$

$a = 10.269(4) \text{ \AA}$

$b = 19.958(3) \text{ \AA}$

$c = 15.066(3) \text{ \AA}$

$\beta = 103.80(2)^\circ$

$V = 2998.6(14) \text{ \AA}^3$

$Z = 8$

$D_x = 2.402 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71069 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 3.19-12.68^\circ$

$\mu = 9.228 \text{ mm}^{-1}$

$T = 293(2) \text{ K}$

Prismatic

$0.2 \times 0.2 \times 0.1 \text{ mm}$

Orange

Data collection

Enraf-Nonius CAD-4 diffractometer

$\omega/2\theta$ scans

Absorption correction:

ψ scans (MolEN; Fair, 1990)

$T_{\min} = 0.73$, $T_{\max} = 0.99$

4995 measured reflections

4707 independent reflections

3751 observed reflections

$[I > 2\sigma(I)]$

$R_{\text{int}} = 0.0282$

$\theta_{\text{max}} = 23.99^\circ$

$h = 0 \rightarrow 11$

$k = 0 \rightarrow 22$

$l = -17 \rightarrow 16$

3 standard reflections

frequency: 240 min

intensity decay: 1.2%

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.0290$

$wR(F^2) = 0.0756$

$S = 1.182$

4707 reflections

455 parameters

All H atoms refined

freely with isotropic

displacement parameters

except for H1B which

was placed in a calculated

position

$w = 1/[\sigma^2(F_o^2) + (0.031P)^2$

$+ 6.61P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = -0.7$

$\Delta\rho_{\text{max}} = 1.09 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -1.55 \text{ e \AA}^{-3}$

Extinction correction:

SHELXL93 (Sheldrick, 1993)

Extinction coefficient:

0.00363(10)

Atomic scattering factors

from *International Tables*

for *Crystallography* (1992),

Vol. C, Tables 4.2.6.8 and

6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (I)

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Os1	0.23191 (3)	0.357482 (15)	0.70256 (2)	0.02221 (12)
F11	0.3767 (5)	0.2964 (2)	0.6957 (3)	0.0406 (12)
F12	0.2558 (5)	0.3968 (2)	0.5893 (3)	0.0345 (11)

Cl11	0.0610 (2)	0.43337 (11)	0.7057 (2)	0.0392 (5)
Cl12	0.2123 (3)	0.30555 (13)	0.8355 (2)	0.0529 (7)
Cl13	0.0830 (2)	0.28241 (10)	0.61091 (14)	0.0328 (5)
Cl14	0.3898 (2)	0.43320 (10)	0.78335 (14)	0.0353 (5)
Os2	-0.25242 (3)	0.10469 (2)	0.70726 (2)	0.02411 (12)
F21	-0.1041 (4)	0.0428 (2)	0.7105 (4)	0.0409 (12)
F22	-0.3813 (4)	0.0295 (2)	0.6491 (4)	0.0412 (13)
Cl21	-0.4280 (2)	0.17887 (11)	0.70040 (14)	0.0354 (5)
Cl22	-0.0986 (2)	0.18813 (11)	0.7664 (2)	0.0377 (5)
Cl24	-0.2490 (3)	0.12843 (13)	0.5593 (2)	0.0509 (6)
Cl23	-0.2583 (2)	0.06831 (13)	0.8536 (2)	0.0489 (6)
CM1	-0.2743 (10)	0.4072 (5)	0.7444 (6)	0.035 (2)
NA	-0.2425 (6)	0.3610 (3)	0.8235 (4)	0.0263 (15)
C1A	-0.1216 (9)	0.3651 (5)	0.8817 (6)	0.038 (2)
C2A	-0.0950 (10)	0.3275 (6)	0.9596 (7)	0.048 (3)
C3A	-0.1915 (11)	0.2860 (5)	0.9782 (6)	0.046 (3)
C4A	-0.3140 (10)	0.2811 (5)	0.9161 (6)	0.042 (2)
C5A	-0.3375 (9)	0.3186 (5)	0.8380 (6)	0.032 (2)
NB	-0.3174 (6)	0.3700 (3)	0.6577 (4)	0.0250 (14)
C1B	-0.4408 (8)	0.3789 (4)	0.6053 (6)	0.036 (2)
C2B	-0.4804 (9)	0.3493 (5)	0.5222 (6)	0.039 (2)
C3B	-0.3927 (9)	0.3079 (5)	0.4939 (6)	0.037 (2)
C4B	-0.2669 (9)	0.2957 (4)	0.5482 (6)	0.033 (2)
C5B	-0.2300 (8)	0.3285 (4)	0.6314 (5)	0.026 (2)
CM2	0.2379 (9)	0.1516 (4)	0.7364 (6)	0.030 (2)
NC	0.2776 (6)	0.1127 (3)	0.8203 (4)	0.0251 (14)
C1C	0.4032 (9)	0.1183 (5)	0.8732 (6)	0.034 (2)
C2C	0.4375 (9)	0.0861 (5)	0.9563 (6)	0.040 (2)
C3C	0.3448 (9)	0.0478 (5)	0.9827 (6)	0.038 (2)
C4C	0.2183 (9)	0.0407 (4)	0.9266 (6)	0.036 (2)
C5C	0.1859 (9)	0.0736 (4)	0.8451 (6)	0.034 (2)
ND	0.2117 (6)	0.1072 (3)	0.6546 (4)	0.0268 (15)
C1D	0.0915 (9)	0.1129 (5)	0.5944 (6)	0.035 (2)
C2D	0.0702 (11)	0.0787 (5)	0.5151 (7)	0.043 (2)
C3D	0.1660 (12)	0.0378 (5)	0.4958 (7)	0.048 (3)
C4D	0.2844 (11)	0.0308 (5)	0.5589 (7)	0.046 (2)
C5D	0.3090 (10)	0.0662 (4)	0.6379 (6)	0.036 (2)

Table 2. Selected geometric parameters (\AA , $^\circ$) for (I)

Os1—F11	1.945 (4)	NB—C1B	1.336 (10)
Os1—F12	1.946 (4)	NB—C5B	1.348 (10)
Os1—Cl12	2.306 (2)	C1B—C2B	1.356 (12)
Os1—Cl11	2.327 (2)	C2B—C3B	1.364 (13)
Os1—Cl14	2.335 (2)	C3B—C4B	1.376 (12)
Os1—Cl13	2.341 (2)	C4B—C5B	1.384 (12)
Os2—F21	1.953 (4)	CM2—NC	1.457 (10)
Os2—F22	2.054 (4)	CM2—ND	1.489 (10)
Os2—Cl24	2.288 (3)	NC—C5C	1.342 (11)
Os2—Cl21	2.316 (2)	NC—C1C	1.349 (10)
Os2—Cl22	2.322 (2)	C1C—C2C	1.377 (13)
Os2—Cl23	2.335 (2)	C2C—C3C	1.352 (13)
CM1—NB	1.476 (11)	C3C—C4C	1.379 (13)
CM1—NA	1.479 (11)	C4C—C5C	1.362 (13)
NA—C1A	1.341 (10)	ND—C1D	1.351 (11)
NA—C5A	1.349 (11)	ND—C5D	1.361 (11)
C1A—C2A	1.365 (14)	C1D—C2D	1.348 (13)
C2A—C3A	1.371 (15)	C2D—C3D	1.363 (15)
C3A—C4A	1.380 (14)	C3D—C4D	1.360 (15)
C4A—C5A	1.367 (13)	C4D—C5D	1.355 (13)
F11—Os1—F12	87.0 (2)	F21—Os2—Cl24	87.8 (2)
F11—Os1—Cl12	89.5 (2)	F22—Os2—Cl24	83.9 (2)
F12—Os1—Cl12	176.4 (2)	F21—Os2—Cl21	178.8 (2)
F11—Os1—Cl11	177.5 (2)	F22—Os2—Cl21	91.85 (13)
F12—Os1—Cl11	90.53 (15)	Cl24—Os2—Cl21	90.97 (8)
Cl12—Os1—Cl11	93.01 (9)	F21—Os2—Cl22	89.4 (2)
F11—Os1—Cl14	89.4 (2)	F22—Os2—Cl22	176.4 (2)
F12—Os1—Cl14	88.81 (15)	Cl24—Os2—Cl22	93.32 (9)
Cl12—Os1—Cl14	91.81 (9)	Cl21—Os2—Cl22	90.56 (8)
Cl11—Os1—Cl14	90.07 (8)	F21—Os2—Cl23	88.6 (2)
F11—Os1—Cl13	88.1 (2)	F22—Os2—Cl23	90.9 (2)
F12—Os1—Cl13	86.67 (15)	Cl24—Os2—Cl23	173.80 (9)
Cl12—Os1—Cl13	92.56 (8)	Cl21—Os2—Cl23	92.61 (9)
Cl11—Os1—Cl13	92.23 (8)	Cl22—Os2—Cl23	91.70 (8)
Cl14—Os1—Cl13	174.94 (8)	NB—CM1—NA	111.2 (7)
F21—Os2—F22	88.2 (2)	NC—CM2—ND	111.0 (7)

Compound (II)

Crystal data

(C₁₁H₁₂N₂)[OsCl₄F₂] $M_r = 542.23$

Triclinic

 $P\bar{1}$ $a = 7.7562 (5) \text{\AA}$ $b = 10.0127 (8) \text{\AA}$ $c = 11.0057 (9) \text{\AA}$ $\alpha = 89.649 (7)^\circ$ $\beta = 70.346 (6)^\circ$ $\gamma = 75.112 (6)^\circ$ $V = 774.76 (14) \text{\AA}^3$ $Z = 2$ $D_x = 2.324 \text{ Mg m}^{-3}$

Data collection

Enraf-Nonius CAD-4

diffractometer

 $\omega/2\theta$ scans

Absorption correction:

 ψ scans (MolEN; Fair, 1990) $T_{\min} = 0.73$, $T_{\max} = 1.00$

3007 measured reflections

2795 independent reflections

Refinement

Refinement on F^2 $R[F^2 > 2\sigma(F^2)] = 0.0148$ $wR(F^2) = 0.0389$ $S = 1.132$

2795 reflections

233 parameters

All H-atom parameters

refined

 $w = 1/[\sigma^2(F_o^2) + (0.018P)^2 + 0.31P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ Mo $K\alpha$ radiation $\lambda = 0.71069 \text{\AA}$

Cell parameters from 25

reflections

 $\theta = 4.01\text{--}11.85^\circ$ $\mu = 8.928 \text{ mm}^{-1}$ $T = 293 (2) \text{ K}$

Prismatic

 $0.3 \times 0.2 \times 0.1 \text{ mm}$

Orange

2354 observed reflections

 $[I > 2\sigma(I)]$ $R_{\text{int}} = 0.0122$ $\theta_{\max} = 29.94^\circ$ $h = 0 \rightarrow 9$ $k = -13 \rightarrow 11$ $l = -13 \rightarrow 14$

3 standard reflections

frequency: 240 min

intensity decay: 2.4%

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (II)

	$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$			
	x	y	z	U_{eq}
Os1	0	0	0	0.02399 (7)
F1	-0.1246 (3)	0.1371 (2)	0.1459 (2)	0.0355 (5)
Cl11	0.19078 (12)	0.14176 (9)	-0.10534 (9)	0.0360 (2)
Cl12	-0.22903 (14)	0.10522 (10)	-0.09175 (9)	0.0415 (2)
Os2	1/2	1/2	1/2	0.02644 (7)
F2	0.5527 (3)	0.3321 (2)	0.3933 (2)	0.0365 (5)
Cl21	0.18906 (13)	0.56678 (10)	0.49972 (10)	0.0407 (2)
Cl22	0.59682 (13)	0.61427 (9)	0.31263 (9)	0.0401 (2)
CM	0.4380 (6)	0.1528 (4)	0.2233 (4)	0.0337 (8)
N1	0.3252 (4)	0.2828 (3)	0.1953 (3)	0.0278 (6)
C11	0.3931 (7)	0.3947 (4)	0.1807 (4)	0.0428 (10)
C12	0.2893 (9)	0.5154 (4)	0.1519 (4)	0.0590 (13)
C13	0.1193 (9)	0.5206 (5)	0.1389 (5)	0.066 (2)
C14	0.0535 (7)	0.4053 (5)	0.1534 (4)	0.0552 (12)
C15	0.1595 (6)	0.2855 (4)	0.1809 (4)	0.0392 (9)
N2	0.3345 (4)	0.1099 (3)	0.3509 (3)	0.0265 (6)

C21	0.3521 (6)	-0.0266 (4)	0.3614 (4)	0.0369 (9)
C22	0.2727 (6)	-0.0715 (4)	0.4789 (4)	0.0449 (10)
C23	0.1771 (6)	0.0211 (4)	0.5856 (4)	0.0449 (10)
C24	0.1606 (6)	0.1595 (4)	0.5721 (4)	0.0431 (10)
C25	0.2413 (5)	0.2030 (4)	0.4546 (3)	0.0316 (8)

Table 4. Selected geometric parameters (\AA , $^\circ$) for (II)

Os1—F1	1.923 (2)	C11—C12	1.370 (6)
Os1—C111	2.3353 (8)	C12—C13	1.361 (8)
Os1—C112	2.3488 (9)	C13—C14	1.365 (8)
Os2—F2	1.929 (2)	C14—C15	1.365 (6)
Os2—C121	2.3323 (9)	N2—C25	1.344 (4)
Os2—C122	2.3444 (9)	N2—C21	1.346 (4)
CM—N1	1.465 (4)	C21—C22	1.362 (5)
CM—N2	1.488 (4)	C22—C23	1.365 (6)
N1—C15	1.341 (5)	C23—C24	1.370 (6)
N1—C11	1.343 (5)	C24—C25	1.357 (5)
F1 ⁱ —Os1—C111	90.10 (6)	F2—Os2—C121	90.59 (7)
F1—Os1—C111	89.90 (6)	F2 ⁱⁱ —Os2—C122	90.44 (7)
F1—Os1—C112 ⁱ	90.31 (7)	F2—Os2—C122	89.56 (7)
F1—Os1—C112	89.69 (7)	C121 ⁱⁱ —Os2—C122	90.46 (3)
C111—Os1—C112	90.37 (3)	C121—Os2—C122	89.54 (3)
F2—Os2—C121 ⁱⁱ	89.41 (7)	N1—CM—N2	111.0 (3)

Symmetry codes: (i) $-x, -y, -z$; (ii) $1-x, 1-y, 1-z$.

In each compound the major residual features in the ΔF synthesis lie close to the metal atoms.

For both compounds, data collection: *CAD-4-PC* (Enraf-Nonius, 1993); cell refinement: *CAD-4-PC*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structures: *SIR92* (Altomare *et al.*, 1992); program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1027). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Sodium 3 α ,7 α ,12 α -Trihydroxy-5 β -cholan-24-oate Ethanolate (Sodium Cholate Ethanolate)

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Abstract

The crystal structure of sodium cholate ethanolate, $\text{Na}^+\cdot\text{C}_{24}\text{H}_{39}\text{O}_5^-\cdot\text{C}_2\text{H}_6\text{O}$, recrystallized from absolute ethanol by slow evaporation, has been determined. This structure is the first report of a sodium cholate structure solely solvated by an organic molecule.

Comment

Our research into methyl rotation in the solid state led us to a number of compounds containing multiple methyl groups, including the two bile acids, cholic acid and deoxycholic acid. While most previous structural research has focused on deoxycholic acid (3 α ,12 α -dihydroxy-5 β -cholan-24-oic acid) and its many inclusion compounds (Giglio, 1984), more recent research has examined the many possible solvated structures of cholic acid (3 α ,7 α ,12 α -trihydroxy-5 β -cholan-24-oic acid). Many cholic acid structures have been solved, including cholic acid complexed with water (Lessinger, 1982; Lessinger & Low, 1993), acetophenone (Miki *et al.*, 1988), valerolactone (Miki, Kasai, Shibakami, Take-moto & Miyata, 1991), benzene (Nakano, Sada & Miyata, 1994), aniline (Caira, Nassimbeni & Scott, 1993; Shibakami & Sekiya, 1994), 3-fluoroaniline (Shibakami & Sekiya, 1994), nitrobenzene (Caira, Nassimbeni & Scott, 1993), acetonitrile (Scott, 1995), and a number of