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

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Pd1	0.02848 (2)	0.96547 (2)	0.88343 (1)	0.02770 (3)
N1	−0.0989 (2)	1.0493 (2)	0.9144 (1)	0.0284 (3)
C1	−0.0825 (3)	1.1310 (2)	0.9216 (2)	0.0303 (3)
C2	0.0074 (2)	1.1822 (2)	0.8880 (2)	0.0344 (3)
C3	0.0103 (3)	1.1676 (2)	0.8056 (2)	0.0411 (3)
C4	−0.0027 (3)	1.2806 (2)	0.9031 (2)	0.0467 (4)
C5	0.1076 (3)	1.1449 (2)	0.9196 (2)	0.0353 (3)
N2	0.1253 (2)	1.0635 (2)	0.9291 (2)	0.0354 (3)
O1	−0.1469 (2)	1.1747 (2)	0.9661 (1)	0.0367 (3)
C6	−0.2114 (3)	1.1076 (3)	1.0018 (2)	0.0390 (3)
C7	−0.1985 (2)	1.0280 (2)	0.9532 (2)	0.0318 (3)
C8	−0.2910 (3)	1.0146 (2)	0.9001 (2)	0.0359 (3)
C9	−0.3254 (2)	1.0970 (2)	0.8634 (2)	0.0373 (3)
C10	−0.2753 (3)	1.1271 (3)	0.8014 (2)	0.0453 (3)
C11	−0.3048 (4)	1.2063 (3)	0.7709 (3)	0.0607 (4)
C12	−0.3836 (4)	1.2557 (3)	0.8020 (3)	0.0717 (4)
C13	−0.4355 (4)	1.2253 (3)	0.8622 (3)	0.0655 (4)
C14	−0.4077 (3)	1.1465 (3)	0.8922 (2)	0.0507 (3)
O2	0.1833 (2)	1.2005 (2)	0.9413 (2)	0.0496 (4)
C15	0.2731 (4)	1.1459 (3)	0.9601 (3)	0.0640 (4)
C16	0.2250 (3)	1.0564 (3)	0.9713 (3)	0.0457 (3)
C17	0.1951 (4)	1.0349 (4)	1.0503 (2)	0.0538 (3)
C18	0.1258 (4)	0.9577 (3)	1.0575 (2)	0.0599 (3)
C19	0.0220 (4)	0.9673 (4)	1.0786 (3)	0.0964 (3)
C20	−0.0424 (5)	0.8936 (4)	1.0826 (4)	0.1145 (4)
C21	−0.0006 (5)	0.8131 (4)	1.0626 (4)	0.1011 (4)
C22	0.1029 (5)	0.8037 (4)	1.0452 (4)	0.1060 (4)
C23	0.1666 (4)	0.8758 (3)	1.0428 (3)	0.0825 (4)
C24	−0.0388 (3)	0.8478 (2)	0.8359 (2)	0.0367 (3)
C25	0.0573 (3)	0.8714 (2)	0.8029 (2)	0.0353 (3)
C26	0.1477 (3)	0.8785 (2)	0.8464 (2)	0.0379 (3)
C27	0.2477 (3)	0.9189 (2)	0.8216 (2)	0.0397 (3)
C28	0.3416 (3)	0.8967 (3)	0.8565 (2)	0.0535 (3)
C29	0.4357 (3)	0.9359 (4)	0.8371 (3)	0.0608 (4)
C30	0.4364 (3)	0.9986 (4)	0.7834 (3)	0.0678 (4)
C31	0.3437 (3)	1.0227 (4)	0.7490 (2)	0.0657 (4)
C32	0.2495 (3)	0.9827 (3)	0.7678 (2)	0.0493 (3)
C33	−0.1413 (3)	0.8488 (2)	0.7987 (2)	0.0362 (3)
C34	−0.1608 (3)	0.9069 (3)	0.7426 (2)	0.0449 (3)
C35	−0.2582 (3)	0.9082 (3)	0.7085 (2)	0.0551 (4)
C36	−0.3359 (3)	0.8494 (4)	0.7287 (3)	0.0655 (4)
C37	−0.3163 (3)	0.7898 (3)	0.7830 (3)	0.0634 (4)
C38	−0.2197 (3)	0.7906 (3)	0.8189 (3)	0.0533 (4)
P1	0.5246 (1)	0.93473 (8)	0.08382 (6)	0.0585 (2)
F1	0.5871 (4)	0.8874 (3)	0.1444 (2)	0.1458 (4)
F2	0.4633 (3)	0.9876 (3)	0.1429 (2)	0.1039 (4)
F3	0.6113 (3)	1.0072 (3)	0.0811 (2)	0.0928 (4)
F4	0.5865 (4)	0.8856 (3)	0.0233 (2)	0.1211 (4)
F5	0.4625 (3)	0.9848 (3)	0.0236 (2)	0.1302 (4)
F6	0.4380 (4)	0.8631 (3)	0.0857 (3)	0.1382 (4)

Table 4. Selected geometric parameters (Å, °) for (2)

Pd1—N1	2.130 (3)	Pd1—C24	2.169 (3)
Pd1—N2	2.105 (3)	Pd1—C26	2.118 (3)
N2—Pd1—N1	84.5 (1)	C26—Pd1—N2	99.3 (1)
C24—Pd1—N1	108.0 (1)	C26—Pd1—C24	68.3 (1)

For compound (1), the origin has been fixed using polar axes restraints (Flack & Schwarzenbach, 1988). The allyl group of compound (1) is disordered; the position of the central C atom was split and the occupancies of the two alternative positions were refined holding the sum equal to 1.

For both compounds, data collection: CAD-4 Software (Enraf–Nonius, 1989); cell refinement: CAD-4 Software; data reduction: CRYSTALS (Watkin, 1990); program(s) used to solve structures: SHELXS86 (Sheldrick, 1985); program(s) used to refine structures: CRYSTALS.

Lists of anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1124). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Carruthers, J. R. & Watkin, D. J. (1979). *Acta Cryst.* A35, 698–699.
Enraf–Nonius (1989). *CAD-4 Software.* Enraf–Nonius, Delft, The Netherlands.
Flack, H. D. & Schwarzenbach, D. (1988). *Acta Cryst.* A44, 499–506.
Larson, A. C. (1970). *Crystallographic Computing,* edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 291–294. Copenhagen: Munksgaard.
Leutenegger, U., Umbricht, G., Fahrni, C., von Matt, P. & Pfaltz, A. (1992). *Tetrahedron,* 48, 2143–2156.
Pfaltz, A. (1993). *Acc. Chem. Res.* 26, 339–345.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* Univ. of Göttingen, Germany.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* A39, 158–166.
Watkin, D. (1990). *CRYSTALS. Structure Refinement Program.* Chemical Crystallography Laboratory, Oxford.

fac- and mer-Dipyridiniomethane Trichlorotrifluoroosmate(IV), fac- and mer-[(C$_5$H$_5$N)$_2$CH$_2$][OsCl$_3$F$_3$]

Clemens Bruhn and Wilhelm Preetz

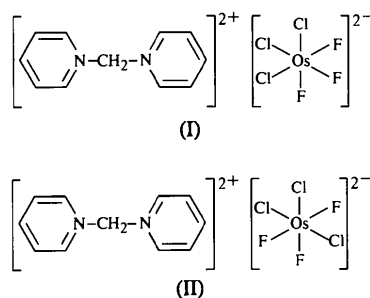*Institut für Anorganische Chemie, Universität Kiel, Olshausenstrasse 40, D-24098 Kiel, Germany*

Abstract

The structures of *fac-N,N'*-methylenedipyridinium trichlorotrifluoroosmate(IV), (I), and *mer-N,N'*-methylenedipyridinium trichlorotrifluoroosmate(IV), (II), *fac-* and *mer-*[(C$_5$H$_5$N)$_2$CH$_2$][OsCl$_3$F$_3$], have been determined by single-crystal X-ray diffraction at room temperature. The complex anions are completely ordered. The Os—F distances in (I) are equal to within 3 e.s.d.'s, as are the Os—Cl distances. The mean Os—F distance of 1.948 Å and Os—Cl distance of 2.3199 Å are typical for bonds altered in length by the *trans* influence. The asymmetric Cl—Os—F coordination axis in (II) has a longer Os—F distance [1.976 (3) Å] and a shorter Os—Cl distance [2.2782 (13) Å] than the symmetric F—Os—F and Cl—Os—Cl axes, which have mean bond lengths of 1.944 and 2.307 Å, respectively.

Comment

Octahedral mixed-ligand complexes of the type $[MCl_nF_{6-n}]^{2-}$ (M = Os, Pt; n = 0–6) have been used as standard species in vibrational and NMR spectroscopy to examine bond interactions arising from the mutual *trans* influence of F and Cl (Preetz, Ruf & Tensfeldt, 1984; Parzich, Peters & Preetz, 1993; Alyoubi, Greenslade, Foster & Preetz, 1990). However, no crystal structures have been available because of the statistical distribution of the ligands in A_2B-type lattices of high symmetry with alkali metal or alkylammonium cations (Keller & Homborg, 1976). Ordered structures can be obtained if doubly charged cations with less regular geometry are used to form AB-type salts (Garnier & Bele, 1994; Brüdgam & Hartl, 1986; Bruhn & Preetz, 1994a,b, 1995), such as the title compounds (I) and (II).

radius of Cl compared to F. Views of an asymmetric unit with the atom-labelling scheme for (I) and (II) are given in Figs. 1 and 2, respectively.

The dipyridiniomethane moiety shows closest H···F contacts to the complex anions of 2.36 Å, which may be described as very weak hydrogen bonds. The pyridine rings exhibit different interplanar angles in each of the cations. This has been postulated as one of factors leading to ordering in structures of this type (Bruhn, Drews, Meynhardt & Preetz, 1995). In (I), the normals to the pyridine rings form angles of 63.8 (3) and 61.9 (3)° with the normal to the plane through N1, C1 and N2, and angles of 45.9 (4) and 61.5 (4)° to the plane through N3, C2 and N4. In (II), the normals to the pyridine rings form angles of 84.3 (3) and 72.8 (3)° with the plane through CM, N1 and N2. Stereoscopic views of the unit cells are given in Figs. 3 and 4.
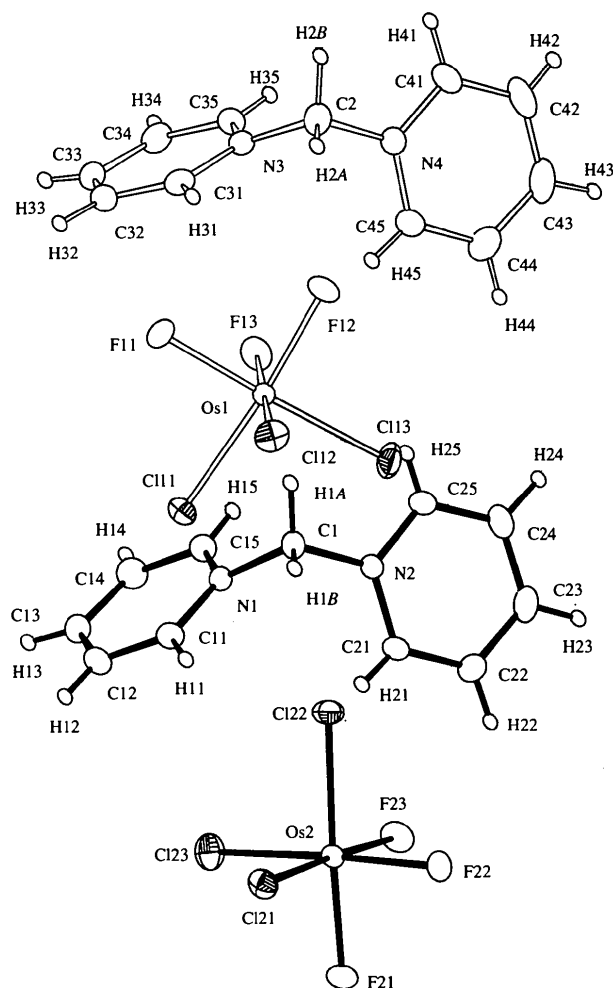

(I)

(II)

Compound (II) crystallizes in space group $P2_1/n$. The complex anion *mer*-$[OsCl_3F_3]^{2-}$ has local C_{2v} symmetry and contains three different axes: F—Os—F with Os—F distances of 1.941 (3) and 1.946 (3) Å, Cl—Os—Cl with Os—Cl distances of 2.3039 (15) and 2.3108 (15) Å, and F—Os—Cl with an Os—F distance of 1.976 (3) Å and an Os—Cl distance of 2.2782 (13) Å. These axes are almost linear (they are bent by less than 3°) and are orthogonal to one another, as expected for an octahedral coordination sphere. As a result of the *trans* influence in the asymmetric coordination axis the Os—F distance is 0.032 Å longer and the Os—Cl distance is 0.029 Å shorter than the corresponding average distances for the symmetric axes. Compound (I) crystallizes in space group $P\bar{1}$ with two formula units in the asymmetric unit. The *fac*-$[OsCl_3F_3]^{2-}$ anion has C_{3v} point symmetry and contains three asymmetric F—Os—Cl coordination axes, for which the Os—F distances would be expected to be equal and the Os—Cl distances would be expected to be equal. In fact, the Os—F distances vary over a range of 0.015 Å from 1.941 (2) to 1.956 (3) Å and the Os—Cl distances vary over a range of 0.017 Å from 2.3085 (14) to 2.3258 (11) Å as a result of steric interactions and weak hydrogen bonds between the cations and anions. The Cl—Os—Cl angles are wider [90.18 (5) to 94.28 (4)°] than the F—Os—F angles [86.75 (12) to 88.05 (12)°] because of the larger atomic

Fig. 1. View of the *fac*-$[OsCl_3F_3]^{2-}$ anions and the $[(C_5H_5N)_2CH_2]^{2+}$ cations in the asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level for non-H atoms. H atoms are drawn as spheres of arbitrary radii.
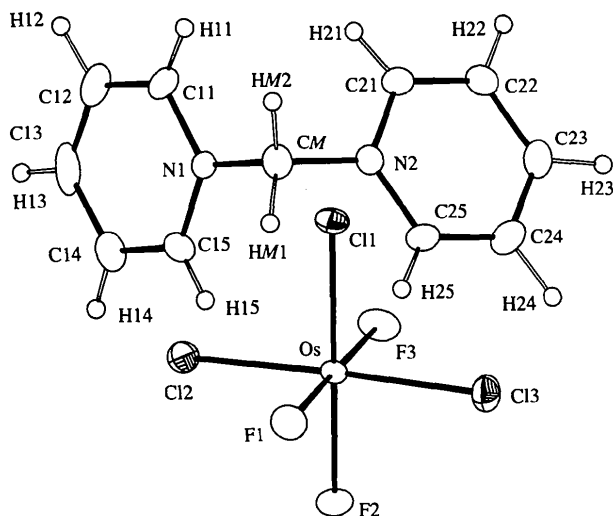
Fig. 2. View of the *mer*-[OsCl₃F₃]²⁻ anion and the [(C₅H₅N)₂CH₂]²⁺ cation in the asymmetric unit of (II). Displacement ellipsoids are drawn at the 50% probability level for non-H atoms. H atoms are drawn as spheres of arbitrary radii.
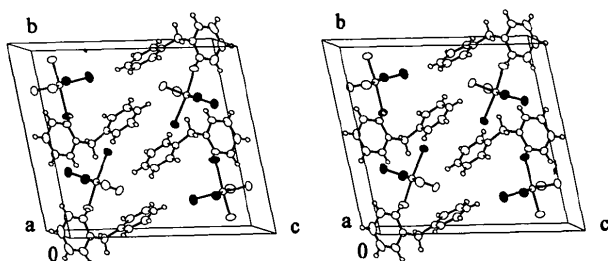

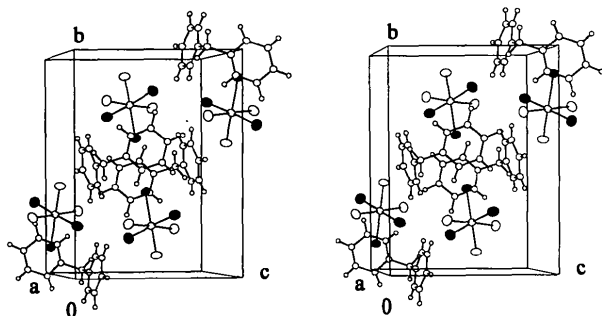Fig. 3. Stereoscopic plot showing a unit cell of (I).

Fig. 4. Stereoscopic plot showing a unit cell of (II).

Experimental

The reaction of K₂[OsCl₆] with BrF₃ at room temperature led to a mixture containing several compounds of the series [OsClₙF₆₋ₙ]²⁻, from which *fac*-[OsCl₃F₃]²⁻ and *cis*-[OsCl₂F₄]²⁻ were separated by ion-exchange chromatography on diethylaminoethylcellulose. They were then isolated as their tetra-*n*-butylammonium (TBA) salts. *cis*-[OsCl₂F₄]²⁻ was converted by stereospecific ligand exchange with HCl

gas in CH₂Cl₂ at 278 K to *mer*-[OsCl₃F₃]²⁻ in a 10% yield (Preetz, Ruf & Tensfeldt, 1984). The dipyridiniomethane salts were precipitated from a *N*,*N*-dimethylformamide solution of the TBA complex salts with dipyridiniomethane dibromide, [(C₅H₅N)₂CH₂]Br₂, and recrystallized from water/acetone to give suitable single crystals.

Compound (I)

Crystal data

[(C₅H₅N)₂CH₂][OsCl₃F₃]	Mo *Kα* radiation
M_r = 525.78	λ = 0.71069 Å
Triclinic	Cell parameters from 25
$P\bar{1}$	reflections
a = 7.223 (3) Å	θ = 5.88–10.43°
b = 13.9130 (15) Å	μ = 9.404 mm⁻¹
c = 14.9310 (15) Å	T = 293 (2) K
α = 100.655 (9)°	Prism
β = 101.33 (3)°	0.2 × 0.2 × 0.1 mm
γ = 90.32 (2)°	Orange
V = 1444.5 (5) Å³	
Z = 4	
D_x = 2.418 Mg m⁻³	

Data collection

Enraf–Nonius CAD-4-Turbo	R_{int} = 0.0128
four-circle diffractometer	θ_{max} = 25.00°
$\omega/2\theta$ scans	$h = 0 \rightarrow 8$
Absorption correction:	$k = -16 \rightarrow 16$
empirical (ψ scans)	$l = -17 \rightarrow 17$
T_{min} = 0.5209, T_{max} =	3 standard reflections
0.9986	monitored every 400
5531 measured reflections	reflections
5088 independent reflections	intensity decay: 1.2%
4709 observed reflections	
[$I > 2\sigma(I)$]	

Refinement

Refinement on F^2	$\Delta\rho_{max}$ = 1.039 e Å⁻³
$R[F^2 > 2\sigma(F^2)]$ = 0.0187	$\Delta\rho_{min}$ = −0.607 e Å⁻³
$wR(F^2)$ = 0.0503	Extinction correction:
S = 1.199	*SHELXL*93 (Sheldrick,
5088 reflections	1993)
458 parameters	Extinction coefficient:
All H-atom parameters	0.0064 (2)
refined	Atomic scattering factors
$w = 1/[\sigma^2(F_o^2) + (0.0247P)^2$	from *International Tables*
$+ 2.2353P]$	*for Crystallography* (1992,
where $P = (F_o^2 + 2F_c^2)/3$	Vol. C, Tables 4.2.6.8 and
$(\Delta/\sigma)_{max}$ = −0.002	6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å²) *for* (I)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Os1	0.08135 (2)	0.743535 (11)	0.716558 (10)	0.02207 (7)
Cl11	0.0457 (2)	0.58840 (8)	0.62422 (7)	0.0320 (2)
Cl12	−0.2303 (2)	0.74164 (9)	0.73219 (9)	0.0394 (3)
Cl13	0.1849 (2)	0.68734 (10)	0.85412 (8)	0.0479 (3)
F11	0.0098 (4)	0.7957 (2)	0.6033 (2)	0.0357 (6)
F12	0.1204 (4)	0.8786 (2)	0.7842 (2)	0.0408 (6)
F13	0.3427 (3)	0.7518 (2)	0.7024 (2)	0.0394 (6)
Os2	0.02144 (2)	0.228196 (11)	0.788450 (11)	0.02409 (7)

	x	y	z	U_{eq}
Cl21	-0.2961 (2)	0.22074 (9)	0.71695 (8)	0.0362 (2)
Cl22	0.0408 (2)	0.39441 (8)	0.78529 (8)	0.0368 (3)
Cl23	0.1186 (2)	0.18417 (10)	0.64789 (9)	0.0481 (3)
F21	0.0072 (4)	0.0905 (2)	0.7975 (2)	0.0393 (6)
F22	-0.0482 (4)	0.2566 (2)	0.9096 (2)	0.0404 (6)
F23	0.2838 (4)	0.2317 (2)	0.8551 (2)	0.0420 (7)
C1	0.4389 (7)	0.5242 (3)	0.7390 (3)	0.0305 (10)
N1	0.4770 (5)	0.4584 (3)	0.6562 (2)	0.0250 (7)
N2	0.5584 (5)	0.5044 (3)	0.8245 (2)	0.0249 (7)
C11	0.3386 (6)	0.3968 (3)	0.6020 (3)	0.0319 (10)
C12	0.3661 (7)	0.3425 (4)	0.5201 (3)	0.0362 (11)
C13	0.5364 (8)	0.3504 (4)	0.4942 (3)	0.0372 (11)
C14	0.6781 (8)	0.4107 (4)	0.5525 (4)	0.0397 (12)
C15	0.6469 (7)	0.4653 (4)	0.6329 (3)	0.0336 (10)
C21	0.5385 (7)	0.4175 (3)	0.8507 (3)	0.0298 (9)
C22	0.6380 (7)	0.4019 (4)	0.9333 (3)	0.0354 (10)
C23	0.7562 (7)	0.4754 (4)	0.9912 (3)	0.0403 (12)
C24	0.7749 (8)	0.5627 (4)	0.9640 (3)	0.0415 (12)
C25	0.6752 (7)	0.5766 (4)	0.8796 (3)	0.0350 (10)
C2	0.4345 (7)	0.0317 (4)	0.7720 (3)	0.0361 (11)
N3	0.4748 (5)	-0.0236 (3)	0.6853 (3)	0.0286 (8)
N4	0.5655 (5)	0.0096 (3)	0.8539 (2)	0.0291 (8)
C31	0.3276 (7)	-0.0660 (4)	0.6189 (4)	0.0377 (11)
C32	0.3550 (9)	-0.1103 (4)	0.5335 (4)	0.0446 (13)
C33	0.5337 (9)	-0.1131 (4)	0.5165 (4)	0.0432 (13)
C34	0.6842 (8)	-0.0722 (4)	0.5853 (4)	0.0377 (11)
C35	0.6523 (7)	-0.0270 (3)	0.6698 (3)	0.0303 (9)
C41	0.6735 (7)	0.0827 (4)	0.9121 (4)	0.0398 (11)
C42	0.7793 (8)	0.0663 (5)	0.9931 (4)	0.0507 (15)
C43	0.7785 (8)	-0.0249 (5)	1.0145 (4)	0.0500 (14)
C44	0.6716 (8)	-0.1007 (5)	0.9521 (4)	0.0464 (13)
C45	0.5634 (7)	-0.0819 (4)	0.8717 (3)	0.0357 (10)

Monoclinic
$P2_1/n$
$a = 9.676$ (5) Å
$b = 14.046$ (3) Å
$c = 10.476$ (3) Å
$\beta = 97.357$ (4)°
$V = 1412.1$ (9) Å³
$Z = 4$
$D_x = 2.473$ Mg m⁻³

Cell parameters from 25 reflections
$\theta = 4.18\text{--}11.79°$
$\mu = 9.620$ mm⁻¹
$T = 293$ (2) K
Prism
$0.2 \times 0.1 \times 0.1$ mm
Pink

Data collection

Enraf–Nonius CAD-4-Turbo four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical (ψ scans)
$T_{min} = 0.6329$, $T_{max} = 0.9987$
2627 measured reflections
2474 independent reflections
2033 observed reflections $[I > 2\sigma(I)]$

$R_{int} = 0.0166$
$\theta_{max} = 24.97°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 16$
$l = -12 \rightarrow 12$
3 standard reflections monitored every 400 reflections
intensity decay: 2.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0183$
$wR(F^2) = 0.0390$
$S = 1.094$
2474 reflections
230 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0142P)^2 + 0.4674P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.001$

$\Delta\rho_{max} = 0.518$ e Å⁻³
$\Delta\rho_{min} = -0.537$ e Å⁻³
Extinction correction: SHELXL93 (Sheldrick, 1993)
Extinction coefficient: 0.00092 (8)
Atomic scattering factors from International Tables for Crystallography (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 2. Selected geometric parameters (Å, °) for (I)

Os1—F11	1.941 (2)	C13—C14	1.371 (7)
Os1—F12	1.953 (3)	C14—C15	1.357 (7)
Os1—F13	1.946 (3)	C21—C22	1.355 (7)
Os1—Cl11	2.3194 (11)	C22—C23	1.377 (7)
Os1—Cl12	2.3085 (14)	C23—C24	1.364 (8)
Os1—Cl13	2.3207 (12)	C24—C25	1.370 (7)
Os2—F21	1.949 (3)	C2—N3	1.462 (6)
Os2—F22	1.944 (3)	C2—N4	1.473 (6)
Os2—F23	1.956 (3)	N3—C31	1.350 (6)
Os2—Cl21	2.3252 (14)	N3—C35	1.346 (6)
Os2—Cl22	2.3258 (11)	N4—C41	1.341 (6)
Os2—Cl23	2.3197 (13)	N4—C45	1.348 (6)
C1—N1	1.470 (6)	C31—C32	1.361 (8)
C1—N2	1.466 (5)	C32—C33	1.363 (9)
N1—C11	1.343 (6)	C33—C34	1.378 (8)
N1—C15	1.347 (6)	C34—C35	1.365 (7)
N2—C21	1.353 (6)	C41—C42	1.356 (8)
N2—C25	1.345 (6)	C42—C43	1.365 (9)
C11—C12	1.364 (7)	C43—C44	1.386 (9)
C12—C13	1.370 (7)	C44—C45	1.367 (7)

F11—Os1—F12	87.53 (12)	F22—Os2—F23	87.00 (13)
F11—Os1—F13	88.05 (12)	F21—Os2—F23	87.18 (12)
F13—Os1—F12	86.92 (12)	F22—Os2—Cl23	175.40 (9)
F11—Os1—Cl12	90.08 (9)	F21—Os2—Cl23	89.55 (9)
F12—Os1—Cl12	90.87 (10)	F23—Os2—Cl23	90.09 (10)
F13—Os1—Cl12	177.16 (9)	F22—Os2—Cl21	89.89 (10)
F11—Os1—Cl11	87.74 (9)	F21—Os2—Cl21	90.88 (9)
F12—Os1—Cl11	174.25 (9)	F23—Os2—Cl21	176.42 (9)
F13—Os1—Cl11	89.66 (9)	C123—Os2—Cl21	92.90 (5)
Cl12—Os1—Cl11	92.40 (5)	F22—Os2—Cl22	90.76 (9)
F11—Os1—Cl13	176.29 (9)	F21—Os2—Cl22	177.29 (9)
F13—Os1—Cl13	88.85 (9)	F23—Os2—Cl22	91.63 (9)
F12—Os1—Cl13	90.27 (9)	C123—Os2—Cl22	92.88 (5)
Cl12—Os1—Cl13	92.94 (5)	Cl21—Os2—Cl22	90.18 (5)
Cl11—Os1—Cl13	94.28 (4)	N2—C1—N1	111.5 (3)
F22—Os2—F21	86.75 (12)	N3—C2—N4	111.7 (4)

Compound (II)

Crystal data

$[(C_5H_5N)_2CH_2][OsCl_3F_3]$
$M_r = 525.78$

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (II)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Os	0.44963 (2)	0.225211 (13)	0.55497 (2)	0.02124 (8)
Cl1	0.52155 (13)	0.37830 (9)	0.53482 (12)	0.0323 (3)
Cl2	0.26497 (14)	0.27513 (10)	0.65670 (14)	0.0411 (3)
Cl3	0.62901 (14)	0.16791 (10)	0.45067 (13)	0.0423 (3)
F1	0.5627 (3)	0.2095 (2)	0.7206 (3)	0.0395 (7)
F2	0.3839 (3)	0.0931 (2)	0.5714 (3)	0.0398 (7)
F3	0.3345 (3)	0.2371 (2)	0.3892 (3)	0.0414 (8)
N1	0.6841 (4)	0.5026 (3)	0.8098 (3)	0.0261 (9)
N2	0.8449 (4)	0.4624 (3)	0.6601 (4)	0.0275 (9)
CM	0.8323 (5)	0.4957 (4)	0.7934 (5)	0.0328 (12)
C11	0.6200 (7)	0.5884 (4)	0.7916 (5)	0.0407 (14)
C12	0.4815 (7)	0.5958 (5)	0.8046 (5)	0.050 (2)
C13	0.4108 (7)	0.5178 (6)	0.8396 (5)	0.052 (2)
C14	0.4773 (6)	0.4304 (5)	0.8561 (5)	0.0418 (14)
C15	0.6148 (6)	0.4248 (4)	0.8400 (5)	0.0323 (12)
C21	0.8126 (5)	0.5226 (4)	0.5602 (5)	0.0305 (11)
C22	0.8210 (6)	0.4917 (4)	0.4376 (5)	0.0343 (12)
C23	0.8646 (5)	0.3999 (4)	0.4177 (5)	0.0375 (13)
C24	0.8983 (6)	0.3411 (4)	0.5213 (6)	0.0431 (14)
C25	0.8866 (6)	0.3729 (4)	0.6428 (5)	0.0367 (13)

Table 4. *Selected geometric parameters* (Å, °) *for* (II)

Os—F1	1.941 (3)	N2—C21	1.351 (6)
Os—F2	1.976 (3)	N2—C25	1.340 (6)
Os—F3	1.946 (3)	C11—C12	1.369 (9)
Os—Cl1	2.2782 (13)	C12—C13	1.367 (10)
Os—Cl2	2.3039 (15)	C13—C14	1.387 (10)
Os—Cl3	2.3108 (15)	C14—C15	1.365 (8)
N1—C11	1.358 (7)	C21—C22	1.369 (7)
N1—C15	1.341 (6)	C22—C23	1.380 (8)
N1—CM	1.469 (6)	C23—C24	1.369 (8)
N2—CM	1.492 (6)	C24—C25	1.367 (7)
F1—Os—F3	178.35 (12)	C11—Os—Cl2	91.13 (5)
F1—Os—F2	88.07 (13)	F1—Os—Cl3	90.66 (10)
F3—Os—F2	90.28 (13)	F3—Os—Cl3	89.24 (11)
F1—Os—Cl1	92.64 (9)	F2—Os—Cl3	89.06 (10)
F3—Os—Cl1	89.01 (9)	Cl1—Os—Cl3	91.61 (5)
F2—Os—Cl1	179.02 (9)	Cl2—Os—Cl3	177.09 (5)
F1—Os—Cl2	90.17 (10)	C15—N1—C11	121.5 (5)
F3—Os—Cl2	89.85 (11)	N1—CM—N2	109.2 (4)
F2—Os—Cl2	88.18 (10)		

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*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1030). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1992). *SIR92. Program for Crystal Structure Solution.* Univ. of Bari, Italy.
Alyoubi, O. A., Greenslade, D. J., Foster, M. J. & Preetz, W. (1990). *J. Chem. Soc. Dalton Trans.* pp. 381–383.
Brüdgam, I. & Hartl, H. (1986). *Acta Cryst.* C42, 866–868.
Bruhn, C., Drews, H. H., Meynhardt, B. & Preetz, W. (1995). *Z. Anorg. Allg. Chem.* In the press.
Bruhn, C. & Preetz, W. (1994a). *Acta Cryst.* C50, 1555–1557.
Bruhn, C. & Preetz, W. (1994b). *Acta Cryst.* C50, 1687–1690.
Bruhn, C. & Preetz, W. (1995). *Acta Cryst.* C51, 865–867.
Enraf–Nonius (1993). *CAD-4-PC.* Version 1.2. Enraf–Nonius, Delft, The Netherlands.
Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis.* Enraf–Nonius, Delft, The Netherlands.
Garnier, E. & Bele, M. (1994). *Acta Cryst.* C50, 994–996.
Johnson, C. K. (1976). *ORTEPII.* Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Keller, H.-L. & Homborg, H. (1976). *Z. Anorg. Allg. Chem.* 422, 261–265.
Parzich, E., Peters, G. & Preetz, W. (1993). *Z. Naturforsch. Teil B*, 48, 1169–1174.
Preetz, W., Ruf, D. & Tensfeldt, D. (1984). *Z. Naturforsch. Teil B*, 39, 1100–1109.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* Univ. of Göttingen, Germany.

μ-(1,2,5,6-η:3,4,7,8-η)-1,3,5,7-Cyclooctatetraene-bis[dimethylplatinum(II)]

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Abstract

μ-(1,2,5,6-η:3,4,7,8-η)-1,3,5,7-Cyclooctatetraene-bis[dimethylplatinum(II)], [Pt₂(C₈H₈)(CH₃)₄], consists of two Pt(CH₃)₂ groups bridged by a single cyclooctatetraene molecule. The coordination about each Pt atom is approximately square planar, if each olefinic group is considered as a single ligand. The molecule contains a twofold axis passing through the Pt atoms and the center of the cyclooctatetraene molecule.

Comment

As part of a study concerned with the synthesis and properties of metal–olefin compounds, the NMR spectrum of the title compound, (I), was examined for potential long-range coupling by the Pt¹⁹⁵ nuclei. Here we report its crystal structure.

(I)
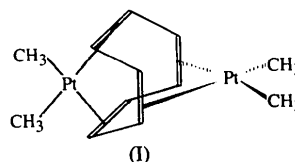
The molecular structure and atom-numbering scheme are shown in Fig. 1. The molecule consists of two dimethylplatinum groups bridged by a cyclooctatetraene molecule. The molecule displays a twofold axis passing through the Pt atoms and the center of the cyclooctatetraene molecule. The coordination about each Pt atom is approximately square planar with the third and fourth bonds of the platinum directed towards the midpoints

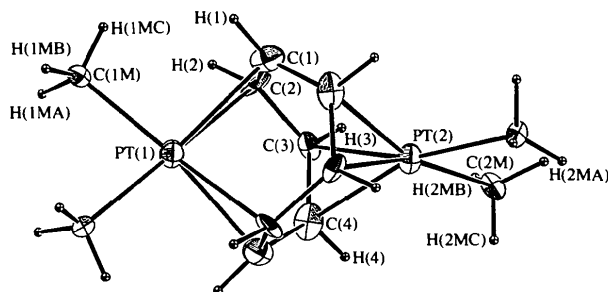Fig. 1. *ORTEP* (Johnson, 1965) view of the title compound with displacement ellipsoids plotted at the 25% probability level.