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Triphenylstannylferrocene and 1,1′-Bis(triphenylstannyl)ferrocene

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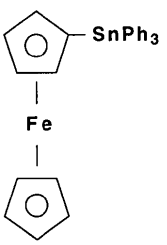
Abstract

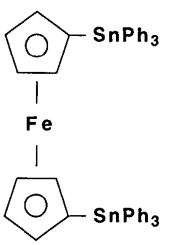
The coordination about the Sn atoms is tetrahedral for both triphenylstannylferrocene, $[FeSn(C_5H_4)(C_5H_5)(C_6H_5)_3]$ (1), and 1,1′-bis(triphenylstannyl)ferrocene, $[FeSn_2(C_5H_4)_2(C_6H_5)_6]$ (2), with C—Sn—C angles ranging from 106.4 (1) to 111.3 (1)°. The cyclopentadienyl rings in the ferrocene moieties of both compounds, (1) and (2), are planar with dihedral angles of 1.7 (2) and 1.4 (3)°, respectively, between the rings. However, the rings in (1) are eclipsed whereas in (2) the presence of two bulky triphenyltin groups attached in a *trans* configuration causes the rings to be slightly staggered, the largest torsion angle about the centroids of the two cyclopentadienyl rings being 19.4°. The mean Fe—C distances in (1) and (2) are 2.043 (7) and 2.049 (8) Å, respectively; the mean C—C distances within the cyclopentadienyl rings are 1.41 (1) and 1.42 (2) Å, respectively.

Comment

The structure and reactivity of 1,1′-bis(triphenylstannyl)ferrocene and other trialkyl- and triarylstannylferrocenes have been studied by Mössbauer and NMR spectroscopy (Roberts, Silver & Azizan, 1986) but their crystal structures were not determined. Little information is available for many such compounds, particularly the mono-triphenylstannylferrocene, owing to low yields and difficulties in separating the products from the reaction mixture. Hence, structural studies of both mono- and bis(triphenylstannyl)ferrocene will be useful in this context. The compounds (1) and (2) were prepared by lithiation of ferrocene followed by reaction with triphenylstannyl chloride as reported by Rausch & Ciappenelli (1967). The mono- and bis(triphenylstannyl)ferrocene were separated by preparative TLC using silica gel. Single crystals of optimum size for X-ray studies were grown from a 20:30 mixture of carbon disulfide–methanol at room temperature.



(1)



(2)

The coordination about the Sn atoms for both (1) and (2) is very close to ideal tetrahedral with C—Sn—C angles between 106.4 (1) and 111.3 (1)°. The tetrahedral angles for symmetrical tetraphenyltin (Chieh & Trotter, 1970) and tetrakis(4-methylphenyl)tin (Karipides & Wolfe, 1975) are 109.5 and 114.3°, respectively. The Sn—C(phenyl) bond distances are normal, between 2.130 (4) and 2.146 (3) Å, but the Sn—C(cyclopentadienyl) distances are slightly shorter, ranging from 2.102 (3) to 2.123 (3) Å.

The Fe···C distances in (1) and (2) are very regular with ranges of 2.028 (5)–2.055 (3) and 2.042 (4)–2.070 (3) Å, respectively. These distances are comparable with those found in ferrocene (Seiler & Dunitz, 1982) and its derivatives (Shawkataly, Kalil, Fun, Chinnakali, Teoh, Ito & Sawamura, 1993; Shawkataly, Fun, Chinnakali, Yip, Teoh, Ito & Sawamura, 1993; Einstein & Willis, 1980) and are also in the range of those found in structures containing the phenylferrocenyl unit where the Fe—C distances range from 1.957 to 2.212 Å with a mean of 2.041 Å. Structures with a crowded coordination sphere have a larger range of distances with a mean greater than 2.041 Å (Roberts, Silver, Yamin, Drew & Eberhardt, 1988). The non-bonded Fe···Sn distance in (1) is 3.678 Å while in (2) the distances of the Fe atoms from Sn(1) and Sn(2) are 3.742 and 3.746 Å, respectively. The mean C—C distances in the cyclopentadienyl rings of (1) and (2) are 1.41 (1) and 1.42 (2) Å. Suprisingly, despite the presence of bulky groups, these distances are not very different from those observed in ferrocene (mean = 1.407 Å).

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The cyclopentadienyl rings of (1) are eclipsed with the largest torsion angle about the centroids of the two cyclopentadienyl rings being 2.1 (3)°. However, compound (2) prefers a *trans* configuration and the presence of the two bulky triphenyltin groups results in the cyclopentadienyl rings being slightly staggered; the largest torsion angle about the centroids of the two cyclopentadienyl rings is 19.4 (4)°. The Sn atoms in (2) are coplanar with their bonded cyclopentadienyl ring, with torsion angles Sn(1)—C(22)—C(23)—C(19) and Sn(2)—C(24)—C(25)—C(26) of 179.7 (2) and 179.5 (2)°, respectively. The Sn atom in (1) deviates more from the bonded cyclopentadienyl plane with an Sn(1)—C(22)—C(23)—C(19) torsion angle of 176.0 (2)°.

The angles between least-squares planes in (1) and (2) are given in Table 5. The dihedral angle between the two cyclopentadienyl rings in compound (1) is 1.4 (3), and that in compound (2) is 1.7 (2)°. The phenyl rings are rotated such that their least-squares planes intersect each other and those of the cyclopentadienyl rings in order to reduce steric effects and therefore retain the tetrahedral coordination about the Sn atoms.

The C—C distances within the phenyl rings for both (1) and (2) are normal with means of 1.381 (9) and 1.32 (2) Å respectively.

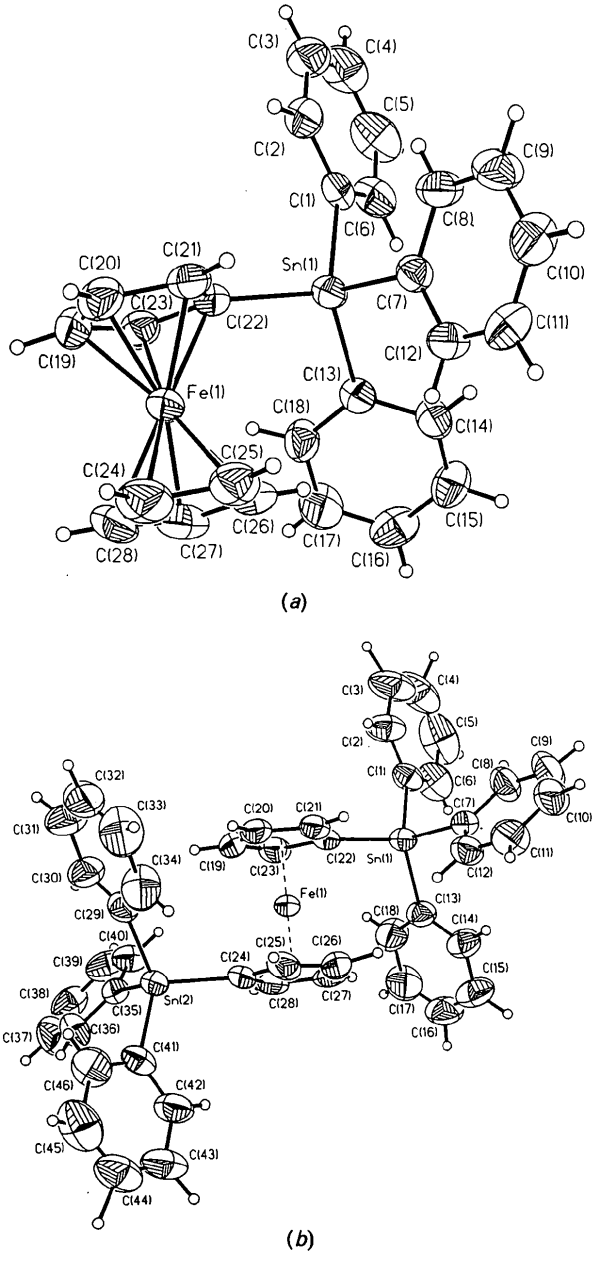

Fig. 1. Views of (*a*) triphenylstannylferrocene and (*b*) 1,1′-bis-(triphenylstannyl)ferrocene showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level.

Experimental

Compound (1)

Crystal data

$[FeSn(C_5H_4)(C_5H_5)(C_6H_5)_3]$	Mo $K\alpha$ radiation
$M_r = 535.0$	$\lambda = 0.7107$ Å
Monoclinic	Cell parameters from 23 reflections
$P2_1/c$	$\theta = 7.5$–$17.5°$
$a = 9.680$ (1) Å	$\mu = 1.731$ mm^{-1}
$b = 13.552$ (2) Å	$T = 298$ K
$c = 17.794$ (3) Å	Parallelepiped
$\beta = 100.36$ (1)°	0.40 × 0.28 × 0.18 mm
$V = 2296.2$ (6) Å^3	Dark orange
$Z = 4$	
$D_x = 1.548$ Mg m^{-3}	

Data collection

Siemens *P*4 diffractometer	$R_{int} = 0.0249$
$2\theta/\theta$ scans	$\theta_{max} = 27.5°$
Absorption correction: empirical	$h = 0 \rightarrow 12$
$T_{min} = 0.766$, $T_{max} = 0.946$	$k = 0 \rightarrow 17$
5611 measured reflections	$l = -23 \rightarrow 22$
5306 independent reflections	2 standard reflections monitored every 100 reflections
3934 observed reflections $[F > 4.0\sigma(F)]$	intensity variation: none

Refinement

Refinement on F	$w = 1/\sigma^2(F)$
$R = 0.0241$	$(\Delta/\sigma)_{max} < 0.001$
$wR = 0.0244$	$\Delta\rho_{max} = 0.29$ e Å^{-3}
$S = 1.20$	$\Delta\rho_{min} = -0.44$ e Å^{-3}
3934 reflections	Atomic scattering factors from *SHELXTL/PC* (Sheldrick, 1990)
367 parameters	
All H-atom parameters refined	

Compound (2)

Crystal data

$[FeSn_2(C_5H_4)_2(C_6H_5)_6]$
$M_r = 884.0$
Triclinic
$P\bar{1}$
$a = 10.860\ (2)$ Å
$b = 11.497\ (3)$ Å
$c = 15.868\ (3)$ Å
$\alpha = 85.86\ (2)°$
$\beta = 81.56\ (2)°$
$\gamma = 84.59\ (2)°$
$V = 1947.6\ (7)$ Å^3
$Z = 2$
$D_x = 1.507$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 24 reflections
$\theta = 7.5–17.5°$
$\mu = 1.670$ mm^{-1}
$T = 298$ K
Parallelepiped
$0.40 \times 0.34 \times 0.20$ mm
Dark orange

Data collection

Siemens $P4$ diffractometer
$2\theta/\theta$ scans
Absorption correction: empirical
$T_{min} = 0.700$, $T_{max} = 0.965$
9471 measured reflections
8999 independent reflections
6965 observed reflections $[F > 4.0\sigma(F)]$
$R_{int} = 0.0149$
$\theta_{max} = 27.5°$
$h = 0 \rightarrow 14$
$k = -14 \rightarrow 14$
$l = -20 \rightarrow 20$
2 standard reflections monitored every 200 reflections
intensity variation: none

Refinement

Refinement on F
$R = 0.0289$
$wR = 0.0273$
$S = 1.05$
6965 reflections
594 parameters
All H-atom parameters refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 1.29$ e Å^{-3}
$\Delta\rho_{min} = -0.97$ e Å^{-3}
Atomic scattering factors from *SHELXTL/PC*

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (1)

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

	x	y	z	U_{eq}
Sn(1)	0.0014 (1)	−0.0496 (1)	0.2523 (1)	0.036 (1)
Fe(1)	0.2323 (1)	0.0361 (1)	0.1226 (1)	0.039 (1)
C(1)	−0.1400 (3)	0.0139 (2)	0.3186 (2)	0.040 (1)
C(2)	−0.2403 (4)	0.0823 (3)	0.2858 (2)	0.050 (1)
C(3)	−0.3307 (4)	0.1255 (5)	0.3289 (3)	0.067 (2)
C(4)	−0.3201 (5)	0.1029 (4)	0.4046 (3)	0.076 (2)
C(5)	−0.2223 (5)	0.0357 (4)	0.4382 (3)	0.074 (2)
C(6)	−0.1332 (4)	−0.0096 (3)	0.3952 (2)	0.056 (1)
C(7)	−0.1133 (3)	−0.1582 (2)	0.1783 (2)	0.038 (1)
C(8)	−0.2587 (4)	−0.1509 (3)	0.1590 (2)	0.052 (1)
C(9)	−0.3362 (4)	−0.2170 (3)	0.1098 (3)	0.061 (1)
C(10)	−0.2708 (4)	−0.2930 (3)	0.0794 (2)	0.057 (1)
C(11)	−0.1272 (4)	−0.3023 (3)	0.0979 (2)	0.054 (1)
C(12)	−0.0485 (3)	−0.2352 (3)	0.1469 (2)	0.045 (1)
C(13)	0.1758 (3)	−0.1154 (2)	0.3263 (2)	0.038 (1)
C(14)	0.1670 (4)	−0.2115 (3)	0.3511 (2)	0.048 (1)
C(15)	0.2811 (5)	−0.2574 (3)	0.3955 (2)	0.058 (1)
C(16)	0.4061 (4)	−0.2087 (3)	0.4145 (2)	0.062 (1)
C(17)	0.4164 (4)	−0.1130 (4)	0.3905 (3)	0.067 (2)
C(18)	0.3020 (4)	−0.0666 (3)	0.3472 (2)	0.052 (1)
C(19)	0.1861 (4)	0.1829 (3)	0.1293 (2)	0.056 (1)
C(20)	0.0937 (4)	0.1382 (3)	0.0686 (2)	0.055 (1)
C(21)	0.0213 (3)	0.0617 (3)	0.0991 (2)	0.044 (1)
C(22)	0.0688 (3)	0.0581 (2)	0.1802 (2)	0.036 (1)
C(23)	0.1711 (3)	0.1339 (2)	0.1975 (2)	0.043 (1)
C(24)	0.3380 (5)	−0.0180 (3)	0.0423 (3)	0.067 (2)
C(25)	0.2703 (4)	−0.0954 (3)	0.0732 (2)	0.056 (1)
C(26)	0.3218 (4)	−0.0984 (3)	0.1524 (2)	0.052 (1)
C(27)	0.4211 (4)	−0.0235 (3)	0.1716 (3)	0.061 (1)
C(28)	0.4316 (4)	0.0263 (3)	0.1026 (3)	0.073 (2)

Table 2. *Bond lengths* (Å) *and angles* (°) *for* (1)

Sn(1)—C(1)	2.142 (3)	Sn(1)—C(7)	2.146 (3)
Sn(1)—C(13)	2.140 (3)	Sn(1)—C(22)	2.123 (3)
Fe(1)—C(19)	2.047 (4)	Fe(1)—C(20)	2.042 (4)
Fe(1)—C(21)	2.039 (3)	Fe(1)—C(22)	2.055 (3)
Fe(1)—C(23)	2.042 (4)	Fe(1)—C(24)	2.038 (5)
Fe(1)—C(25)	2.049 (4)	Fe(1)—C(26)	2.047 (4)
Fe(1)—C(27)	2.045 (4)	Fe(1)—C(28)	2.028 (5)
C(1)—C(2)	1.391 (5)	C(1)—C(6)	1.389 (5)
C(2)—C(3)	1.391 (7)	C(3)—C(4)	1.367 (8)
C(4)—C(5)	1.371 (7)	C(5)—C(6)	1.394 (7)
C(7)—C(8)	1.392 (4)	C(7)—C(12)	1.387 (5)
C(8)—C(9)	1.377 (5)	C(9)—C(10)	1.370 (6)
C(10)—C(11)	1.376 (6)	C(11)—C(12)	1.388 (5)
C(13)—C(14)	1.383 (5)	C(13)—C(18)	1.380 (5)
C(14)—C(15)	1.384 (5)	C(15)—C(16)	1.366 (6)
C(16)—C(17)	1.373 (7)	C(17)—C(18)	1.381 (5)
C(19)—C(20)	1.408 (5)	C(19)—C(23)	1.414 (6)
C(20)—C(21)	1.413 (5)	C(21)—C(22)	1.434 (4)
C(22)—C(23)	1.421 (4)	C(24)—C(25)	1.401 (6)
C(24)—C(28)	1.408 (7)	C(25)—C(26)	1.408 (6)
C(26)—C(27)	1.397 (5)	C(27)—C(28)	1.422 (7)
C(1)—Sn(1)—C(7)	107.6 (1)	C(1)—Sn(1)—C(13)	109.9 (1)
C(7)—Sn(1)—C(13)	110.8 (1)	C(1)—Sn(1)—C(22)	110.7 (1)
C(7)—Sn(1)—C(22)	106.4 (1)	C(13)—Sn(1)—C(22)	111.3 (1)
C(19)—Fe(1)—C(20)	40.3 (2)	C(19)—Fe(1)—C(21)	68.0 (1)
C(20)—Fe(1)—C(21)	40.5 (2)	C(19)—Fe(1)—C(22)	68.6 (2)
C(20)—Fe(1)—C(22)	68.7 (1)	C(21)—Fe(1)—C(22)	41.0 (1)
C(19)—Fe(1)—C(23)	40.5 (2)	C(20)—Fe(1)—C(23)	67.9 (1)
C(21)—Fe(1)—C(23)	68.1 (1)	C(22)—Fe(1)—C(23)	40.6 (1)
C(19)—Fe(1)—C(24)	122.2 (2)	C(20)—Fe(1)—C(24)	106.5 (2)
C(21)—Fe(1)—C(24)	121.9 (2)	C(22)—Fe(1)—C(24)	158.5 (1)
C(23)—Fe(1)—C(24)	158.9 (2)	C(19)—Fe(1)—C(25)	158.1 (2)
C(20)—Fe(1)—C(25)	122.8 (2)	C(21)—Fe(1)—C(25)	108.1 (1)
C(22)—Fe(1)—C(25)	123.6 (1)	C(23)—Fe(1)—C(25)	160.0 (2)
C(24)—Fe(1)—C(25)	40.1 (2)	C(19)—Fe(1)—C(26)	159.5 (2)
C(20)—Fe(1)—C(26)	159.5 (1)	C(21)—Fe(1)—C(26)	124.5 (1)
C(22)—Fe(1)—C(26)	109.1 (1)	C(23)—Fe(1)—C(26)	124.6 (2)
C(24)—Fe(1)—C(26)	67.6 (2)	C(25)—Fe(1)—C(26)	40.2 (2)
C(19)—Fe(1)—C(27)	123.1 (2)	C(20)—Fe(1)—C(27)	158.3 (2)
C(21)—Fe(1)—C(27)	160.0 (2)	C(22)—Fe(1)—C(27)	123.6 (2)
C(23)—Fe(1)—C(27)	108.6 (2)	C(24)—Fe(1)—C(27)	68.5 (2)
C(25)—Fe(1)—C(27)	67.9 (2)	C(26)—Fe(1)—C(27)	39.9 (2)
C(19)—Fe(1)—C(28)	107.3 (2)	C(20)—Fe(1)—C(28)	121.8 (2)
C(21)—Fe(1)—C(28)	157.6 (2)	C(22)—Fe(1)—C(28)	159.8 (2)
C(23)—Fe(1)—C(28)	123.6 (2)	C(24)—Fe(1)—C(28)	40.5 (2)
C(25)—Fe(1)—C(28)	67.6 (2)	C(26)—Fe(1)—C(28)	67.4 (2)
C(27)—Fe(1)—C(28)	40.9 (2)	Sn(1)—C(1)—C(2)	120.2 (3)
Sn(1)—C(1)—C(6)	121.7 (2)	C(2)—C(1)—C(6)	118.0 (3)
C(1)—C(2)—C(3)	120.7 (4)	C(2)—C(3)—C(4)	120.3 (4)
C(3)—C(4)—C(5)	120.2 (5)	C(4)—C(5)—C(6)	120.0 (4)
C(1)—C(6)—C(5)	120.8 (4)	Sn(1)—C(7)—C(8)	119.4 (2)
Sn(1)—C(7)—C(12)	122.9 (2)	C(8)—C(7)—C(12)	117.7 (3)
C(7)—C(8)—C(9)	121.5 (4)	C(8)—C(9)—C(10)	120.2 (4)
C(9)—C(10)—C(11)	119.6 (4)	C(10)—C(11)—C(12)	120.4 (4)
C(7)—C(12)—C(11)	120.7 (3)	Sn(1)—C(13)—C(14)	120.1 (2)
Sn(1)—C(13)—C(18)	121.9 (2)	C(14)—C(13)—C(18)	117.9 (3)
C(13)—C(14)—C(15)	121.1 (3)	C(14)—C(15)—C(16)	120.3 (4)
C(15)—C(16)—C(17)	119.3 (4)	C(16)—C(17)—C(18)	120.5 (4)
C(13)—C(18)—C(17)	120.9 (4)	Fe(1)—C(19)—C(20)	69.7 (2)
Fe(1)—C(19)—C(23)	69.6 (2)	C(20)—C(19)—C(23)	107.9 (3)
Fe(1)—C(20)—C(19)	70.0 (2)	Fe(1)—C(20)—C(21)	69.6 (2)
C(19)—C(20)—C(21)	108.2 (3)	Fe(1)—C(21)—C(20)	69.9 (2)
Fe(1)—C(21)—C(22)	70.1 (2)	C(20)—C(21)—C(22)	108.6 (3)
Sn(1)—C(22)—Fe(1)	123.4 (1)	Sn(1)—C(22)—C(21)	123.6 (2)
Fe(1)—C(22)—C(21)	68.9 (2)	Sn(1)—C(22)—C(23)	130.0 (2)
Fe(1)—C(22)—C(23)	69.2 (2)	C(21)—C(22)—C(23)	106.2 (3)
Fe(1)—C(23)—C(19)	69.9 (2)	Fe(1)—C(23)—C(22)	70.2 (2)

C(19)—C(23)—C(22)	109.1 (3)	Fe(1)—C(24)—C(25)	70.4 (3)	C(2)—C(3)	1.375 (8)	C(3)—C(4)	1.318 (14)
Fe(1)—C(24)—C(28)	69.4 (3)	C(25)—C(24)—C(28)	107.6 (4)	C(4)—C(5)	1.364 (12)	C(5)—C(6)	1.433 (12)
Fe(1)—C(25)—C(24)	69.5 (3)	Fe(1)—C(25)—C(26)	69.8 (2)	C(7)—C(8)	1.393 (5)	C(7)—C(12)	1.386 (6)
C(24)—C(25)—C(26)	108.0 (3)	Fe(1)—C(26)—C(25)	70.0 (2)	C(8)—C(9)	1.377 (7)	C(9)—C(10)	1.368 (9)
Fe(1)—C(26)—C(27)	69.9 (2)	C(25)—C(26)—C(27)	109.2 (4)	C(10)—C(11)	1.366 (7)	C(11)—C(12)	1.383 (7)
Fe(1)—C(27)—C(26)	70.1 (2)	Fe(1)—C(27)—C(28)	68.9 (2)	C(13)—C(14)	1.389 (6)	C(13)—C(18)	1.372 (7)
C(26)—C(27)—C(28)	106.6 (4)	Fe(1)—C(28)—C(24)	70.1 (3)	C(14)—C(15)	1.377 (6)	C(15)—C(16)	1.360 (9)
Fe(1)—C(28)—C(27)	70.2 (3)	C(24)—C(28)—C(27)	108.7 (4)	C(16)—C(17)	1.365 (9)	C(17)—C(18)	1.403 (8)
				C(19)—C(20)	1.392 (7)	C(19)—C(23)	1.426 (7)
				C(20)—C(21)	1.417 (6)	C(21)—C(22)	1.437 (5)
				C(22)—C(23)	1.438 (5)	C(24)—C(25)	1.441 (7)
				C(24)—C(28)	1.420 (7)	C(25)—C(26)	1.380 (9)
				C(26)—C(27)	1.392 (13)	C(27)—C(28)	1.461 (9)
				C(29)—C(30)	1.385 (7)	C(29)—C(34)	1.377 (7)
				C(30)—C(31)	1.382 (7)	C(31)—C(32)	1.359 (11)
				C(32)—C(33)	1.356 (12)	C(33)—C(34)	1.408 (9)
				C(35)—C(36)	1.390 (5)	C(35)—C(40)	1.387 (6)
				C(36)—C(37)	1.389 (8)	C(37)—C(38)	1.355 (9)
				C(38)—C(39)	1.363 (8)	C(39)—C(40)	1.385 (8)
				C(41)—C(42)	1.381 (6)	C(41)—C(46)	1.359 (6)
				C(42)—C(43)	1.393 (7)	C(43)—C(44)	1.323 (9)
				C(44)—C(45)	1.359 (9)	C(45)—C(46)	1.431 (10)

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}
Fe(1)	0.4837 (1)	0.7482 (1)	0.7260 (1)	0.056 (1)
Sn(1)	0.2758 (1)	0.9565 (1)	0.8653 (1)	0.045 (1)
Sn(2)	0.7319 (1)	0.5345 (1)	0.6258 (1)	0.044 (1)
C(1)	0.3002 (4)	1.1205 (4)	0.9136 (2)	0.056 (1)
C(2)	0.3458 (5)	1.2093 (4)	0.8581 (4)	0.079 (2)
C(3)	0.3527 (7)	1.3201 (4)	0.8834 (6)	0.110 (3)
C(4)	0.3168 (8)	1.3441 (6)	0.9637 (7)	0.126 (4)
C(5)	0.2701 (7)	1.2609 (9)	1.0223 (6)	0.126 (4)
C(6)	0.2619 (6)	1.1454 (5)	0.9964 (4)	0.090 (2)
C(7)	0.1070 (3)	0.9814 (3)	0.8087 (2)	0.046 (1)
C(8)	0.0245 (4)	1.0800 (4)	0.8256 (3)	0.071 (2)
C(9)	−0.0836 (5)	1.0987 (5)	0.7890 (4)	0.094 (2)
C(10)	−0.1111 (5)	1.0213 (5)	0.7344 (4)	0.090 (2)
C(11)	−0.0319 (4)	0.9237 (4)	0.7171 (3)	0.080 (2)
C(12)	0.0764 (4)	0.9035 (3)	0.7540 (3)	0.061 (1)
C(13)	0.2508 (4)	0.8221 (3)	0.9647 (2)	0.055 (1)
C(14)	0.1410 (4)	0.7666 (4)	0.9758 (3)	0.064 (1)
C(15)	0.1218 (6)	0.6735 (4)	1.0345 (3)	0.083 (2)
C(16)	0.2124 (6)	0.6350 (4)	1.0830 (3)	0.089 (2)
C(17)	0.3212 (6)	0.6880 (5)	1.0747 (4)	0.100 (2)
C(18)	0.3410 (5)	0.7822 (5)	1.0145 (3)	0.083 (2)
C(19)	0.6192 (4)	0.8608 (3)	0.6907 (3)	0.068 (2)
C(20)	0.5329 (4)	0.8748 (3)	0.6332 (3)	0.066 (2)
C(21)	0.4146 (4)	0.9078 (3)	0.6796 (2)	0.052 (1)
C(22)	0.4255 (3)	0.9132 (3)	0.7684 (2)	0.046 (1)
C(23)	0.5558 (4)	0.8834 (3)	0.7739 (3)	0.061 (1)
C(24)	0.5605 (3)	0.5815 (3)	0.7006 (3)	0.055 (1)
C(25)	0.4441 (4)	0.6064 (3)	0.6669 (4)	0.079 (2)
C(26)	0.3496 (5)	0.6333 (4)	0.7324 (6)	0.114 (3)
C(27)	0.3982 (7)	0.6264 (4)	0.8092 (5)	0.106 (3)
C(28)	0.5322 (5)	0.5934 (3)	0.7900 (3)	0.075 (2)
C(29)	0.7690 (4)	0.6610 (3)	0.5223 (2)	0.055 (1)
C(30)	0.8712 (5)	0.7258 (4)	0.5184 (3)	0.072 (2)
C(31)	0.8951 (7)	0.8141 (5)	0.4559 (4)	0.102 (3)
C(32)	0.8153 (8)	0.8385 (6)	0.3973 (4)	0.114 (3)
C(33)	0.7142 (8)	0.7773 (7)	0.3985 (4)	0.117 (3)
C(34)	0.6902 (5)	0.6867 (6)	0.4617 (3)	0.091 (2)
C(35)	0.8799 (3)	0.5237 (3)	0.7013 (2)	0.047 (1)
C(36)	0.9691 (4)	0.4286 (4)	0.6985 (3)	0.070 (2)
C(37)	1.0628 (5)	0.4193 (5)	0.7502 (4)	0.090 (2)
C(38)	1.0703 (5)	0.5043 (5)	0.8036 (4)	0.085 (2)
C(39)	0.9848 (5)	0.5991 (5)	0.8069 (3)	0.081 (2)
C(40)	0.8903 (4)	0.6096 (3)	0.7563 (3)	0.062 (1)
C(41)	0.7177 (3)	0.3654 (3)	0.5809 (3)	0.055 (1)
C(42)	0.6478 (5)	0.2862 (3)	0.6320 (3)	0.075 (2)
C(43)	0.6389 (6)	0.1739 (4)	0.6070 (4)	0.091 (2)
C(44)	0.6993 (6)	0.1394 (5)	0.5332 (4)	0.093 (2)
C(45)	0.7701 (7)	0.2139 (7)	0.4811 (5)	0.106 (3)
C(46)	0.7780 (5)	0.3307 (5)	0.5045 (3)	0.082 (2)

Table 4. *Bond lengths* (Å) *and angles* (°) *for* (2)

Fe(1)—C(19)	2.042 (4)	Fe(1)—C(20)	2.045 (4)	C(19)—Fe(1)—C(20)	39.8 (2)	C(19)—Fe(1)—C(21)	67.3 (2)
Fe(1)—C(21)	2.045 (3)	Fe(1)—C(22)	2.070 (3)	C(20)—Fe(1)—C(21)	40.5 (2)	C(19)—Fe(1)—C(22)	68.9 (1)
Fe(1)—C(23)	2.044 (4)	Fe(1)—C(24)	2.059 (3)	C(20)—Fe(1)—C(22)	69.2 (1)	C(21)—Fe(1)—C(22)	40.9 (1)
Fe(1)—C(25)	2.048 (5)	Fe(1)—C(26)	2.044 (6)	C(19)—Fe(1)—C(23)	40.8 (2)	C(20)—Fe(1)—C(23)	68.2 (2)
Fe(1)—C(27)	2.046 (6)	Fe(1)—C(28)	2.048 (4)	C(21)—Fe(1)—C(23)	67.8 (2)	C(22)—Fe(1)—C(23)	40.9 (1)
Sn(1)—C(1)	2.139 (4)	Sn(1)—C(7)	2.142 (4)	C(19)—Fe(1)—C(24)	108.1 (1)	C(20)—Fe(1)—C(24)	115.1 (2)
Sn(1)—C(13)	2.134 (3)	Sn(1)—C(22)	2.117 (3)	C(21)—Fe(1)—C(24)	147.8 (2)	C(22)—Fe(1)—C(24)	170.1 (2)
Sn(2)—C(24)	2.102 (3)	Sn(2)—C(29)	2.133 (3)	C(23)—Fe(1)—C(24)	130.9 (2)	C(19)—Fe(1)—C(25)	130.0 (2)
Sn(2)—C(35)	2.130 (4)	Sn(2)—C(41)	2.145 (4)	C(20)—Fe(1)—C(25)	107.6 (2)	C(21)—Fe(1)—C(25)	115.8 (2)
C(1)—C(2)	1.379 (6)	C(1)—C(6)	1.360 (7)	C(22)—Fe(1)—C(25)	147.9 (2)	C(23)—Fe(1)—C(25)	169.5 (2)
				C(24)—Fe(1)—C(25)	41.1 (2)	C(19)—Fe(1)—C(26)	167.1 (3)
				C(20)—Fe(1)—C(26)	129.2 (3)	C(21)—Fe(1)—C(26)	108.6 (2)
				C(22)—Fe(1)—C(26)	116.7 (2)	C(23)—Fe(1)—C(26)	150.5 (3)
				C(24)—Fe(1)—C(26)	68.3 (2)	C(25)—Fe(1)—C(26)	39.4 (3)
				C(19)—Fe(1)—C(27)	152.0 (3)	C(20)—Fe(1)—C(27)	167.6 (3)
				C(21)—Fe(1)—C(27)	130.2 (2)	C(22)—Fe(1)—C(27)	108.8 (2)
				C(23)—Fe(1)—C(27)	118.9 (2)	C(24)—Fe(1)—C(27)	69.0 (2)
				C(25)—Fe(1)—C(27)	67.1 (3)	C(26)—Fe(1)—C(27)	39.8 (4)
				C(19)—Fe(1)—C(28)	117.6 (2)	C(20)—Fe(1)—C(28)	148.3 (2)
				C(21)—Fe(1)—C(28)	170.6 (2)	C(22)—Fe(1)—C(28)	131.6 (2)
				C(23)—Fe(1)—C(28)	110.2 (2)	C(24)—Fe(1)—C(28)	40.5 (2)
				C(25)—Fe(1)—C(28)	67.9 (2)	C(26)—Fe(1)—C(28)	68.3 (2)
				C(27)—Fe(1)—C(28)	41.8 (2)	C(1)—Sn(1)—C(7)	106.5 (1)
				C(1)—Sn(1)—C(13)	111.8 (1)	C(7)—Sn(1)—C(13)	107.3 (1)
				C(1)—Sn(1)—C(22)	109.7 (1)	C(7)—Sn(1)—C(22)	108.2 (1)
				C(13)—Sn(1)—C(22)	113.1 (1)	C(24)—Sn(2)—C(29)	110.5 (1)
				C(24)—Sn(2)—C(35)	110.2 (1)	C(29)—Sn(2)—C(35)	108.8 (1)
				C(24)—Sn(2)—C(41)	106.8 (1)	C(29)—Sn(2)—C(41)	111.3 (1)
				C(35)—Sn(2)—C(41)	109.3 (1)	Sn(1)—C(1)—C(2)	119.5 (3)
				Sn(1)—C(1)—C(6)	122.6 (3)	C(2)—C(1)—C(6)	117.6 (4)
				C(1)—C(2)—C(3)	122.5 (6)	C(2)—C(3)—C(4)	120.0 (6)
				C(3)—C(4)—C(5)	120.8 (8)	C(4)—C(5)—C(6)	119.5 (8)
				C(1)—C(6)—C(5)	119.5 (6)	Sn(1)—C(7)—C(8)	119.8 (3)
				Sn(1)—C(7)—C(12)	122.5 (2)	C(8)—C(7)—C(12)	117.7 (4)
				C(7)—C(8)—C(9)	120.8 (4)	C(8)—C(9)—C(10)	120.4 (5)
				C(9)—C(10)—C(11)	119.9 (5)	C(10)—C(11)—C(12)	120.2 (5)
				C(7)—C(12)—C(11)	120.9 (4)	Sn(1)—C(13)—C(14)	118.2 (3)
				Sn(1)—C(13)—C(18)	123.4 (3)	C(14)—C(13)—C(18)	118.2 (4)
				C(13)—C(14)—C(15)	121.6 (5)	C(14)—C(15)—C(16)	119.3 (5)
				C(15)—C(16)—C(17)	120.9 (5)	C(16)—C(17)—C(18)	119.8 (6)
				C(13)—C(18)—C(17)	120.2 (5)	Fe(1)—C(19)—C(20)	70.2 (2)
				Fe(1)—C(19)—C(23)	69.7 (2)	C(20)—C(19)—C(23)	108.9 (4)
				Fe(1)—C(20)—C(19)	70.0 (2)	Fe(1)—C(20)—C(21)	69.7 (2)
				C(19)—C(20)—C(21)	107.5 (4)	Fe(1)—C(21)—C(20)	69.7 (2)
				Fe(1)—C(21)—C(22)	70.5 (2)	C(20)—C(21)—C(22)	110.0 (4)
				Fe(1)—C(22)—Sn(1)	126.7 (1)	Fe(1)—C(22)—C(21)	68.6 (2)
				Sn(1)—C(22)—C(21)	125.1 (3)	Fe(1)—C(22)—C(23)	68.6 (2)
				Sn(1)—C(22)—C(23)	130.0 (3)	C(21)—C(22)—C(23)	104.9 (3)
				Fe(1)—C(23)—C(19)	69.5 (2)	Fe(1)—C(23)—C(22)	70.5 (2)
				C(19)—C(23)—C(22)	108.7 (4)	Fe(1)—C(24)—Sn(2)	127.1 (2)
				Fe(1)—C(24)—C(25)	69.0 (2)	Sn(2)—C(24)—C(25)	123.9 (4)
				Fe(1)—C(24)—C(28)	69.3 (2)	Sn(2)—C(24)—C(28)	129.9 (3)
				C(25)—C(24)—C(28)	106.2 (4)	Fe(1)—C(25)—C(24)	69.9 (3)
				Fe(1)—C(25)—C(26)	70.1 (3)	C(24)—C(25)—C(26)	109.5 (6)
				Fe(1)—C(26)—C(25)	70.4 (3)	Fe(1)—C(26)—C(27)	70.2 (4)

C(25)—C(26)—C(27)	109.5 (6)	Fe(1)—C(27)—C(26)	70.0 (4)
Fe(1)—C(27)—C(28)	69.2 (3)	C(26)—C(27)—C(28)	107.2 (6)
Fe(1)—C(28)—C(24)	70.2 (2)	Fe(1)—C(28)—C(27)	69.0 (3)
C(24)—C(28)—C(27)	107.6 (5)	Sn(2)—C(29)—C(30)	119.3 (3)
Sn(2)—C(29)—C(34)	122.3 (3)	C(30)—C(29)—C(34)	118.2 (4)
C(29)—C(30)—C(31)	121.8 (5)	C(30)—C(31)—C(32)	118.9 (6)
C(31)—C(32)—C(33)	121.4 (6)	C(32)—C(33)—C(34)	119.8 (7)
C(29)—C(34)—C(33)	119.9 (6)	Sn(2)—C(35)—C(36)	121.1 (3)
Sn(2)—C(35)—C(40)	121.8 (3)	C(36)—C(35)—C(40)	117.1 (4)
C(35)—C(36)—C(37)	121.1 (5)	C(36)—C(37)—C(38)	120.4 (5)
C(37)—C(38)—C(39)	119.6 (5)	C(38)—C(39)—C(40)	120.8 (5)
C(35)—C(40)—C(39)	120.9 (4)	Sn(2)—C(41)—C(42)	119.5 (3)
Sn(2)—C(41)—C(46)	122.7 (3)	C(42)—C(41)—C(46)	117.8 (4)
C(41)—C(42)—C(43)	121.7 (5)	C(42)—C(43)—C(44)	120.5 (5)
C(43)—C(44)—C(45)	119.9 (6)	C(44)—C(45)—C(46)	120.5 (6)
C(41)—C(46)—C(45)	119.5 (5)		

Table 5. *Angles (°) between least-squares planes*

Plane 1 is defined by atoms C(19) to C(23), plane 2 by C(24) to C(28), plane 3 by C(1) to C(6), plane 4 by C(7) to C(12) and plane 5 by C(13) to C(18). In each case the atoms are coplanar with the largest deviation being 0.006 Å.

Angle between planes	Compound (1)	Compound (2)
1 and 2	1.7 (2)	1.4 (3)
1 and 3	92.2 (2)	93.0 (3)
1 and 4	84.4 (2)	132.2 (3)
1 and 5	131.0 (2)	61.7 (3)
2 and 3	91.4 (2)	92.6 (3)
2 and 4	86.5 (2)	131.0 (3)
2 and 5	132.4 (2)	60.6 (3)
3 and 4	112.7 (2)	97.1 (3)
3 and 5	80.0 (2)	105.2 (3)
4 and 5	55.7 (2)	70.6 (3)

Structure (1) was solved by direct methods, structure (2) by Patterson methods and both structures were refined by full-matrix least-squares methods. All H atoms were located from difference Fourier maps. The *SHELXTL/PC* (Sheldrick, 1990) program package was used for all calculations.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: LI1082). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Dipyridiniomethane *cis*-Dichlorotetrafluoroosmate(IV)

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Abstract

The structure of dipyridiniomethane *cis*-dichlorotetrafluoroosmate(IV) {or *N,N*-methylenedipyridinium dichlorotetrafluoroosmate(IV), *cis*-$[(C_5H_5N)_2CH_2][OsF_4Cl_2]$}, has been determined by single-crystal X-ray diffraction at room temperature. The complex anions are completely ordered in the lattice. As a result of the *trans* influence in the two asymmetric axes Cl—Os—F, the Os—F distances of 1.941 (4) and 1.953 (5) Å are slightly lengthened compared to those of the symmetric F—Os—F axis of 1.931 (5) and 1.934 (4) Å. Correspondingly, the Os—Cl distances of 2.324 (2) and 2.335 (2) Å are shortened in comparison with those of 2.364 Å in the octahedral complex anion of $K_2[OsCl_6]$.

Comment

We are currently studying the effects of altered symmetry in octahedral mixed-ligand complex ions of the type $[MX_nY_{6-n}]^{2-}$ (M = Re, Tc, Os, Ir, Pt; $X \neq Y$ = F, Cl, Br, I) on the chemical and physical properties. The main emphasis is placed on vibrational and NMR spectra of compounds containing F ligands (Preetz, Ruf & Tensfeldt, 1984; Parzich, Peters & Preetz, 1993; Alyoubi, Greenslade, Foster & Preetz, 1986). Bond interactions resulting from the mutual *trans* influence between different ligands may be described by force-field calculations if the structural parameters are known (Erlhöfer & Preetz, 1989; Preetz & Irmer, 1990). Unfortunately, up to now most of the structure analyses have failed because of the statistical arrangement of the mixed-halogeno complex ions in lattices of high symmetry and, consequently, no detailed information about bond lengths and angles has been available. Total or partial disorder has been observed in complex