Table 1. *Positional parameters* ($\times 10^4$, $\times 10^5$ *for* Ir) *and equivalent isotropic temperature factors* (Å^2 × 10) (*Hamilton*, 1959)

	x	y	z	B_{eq}
Ir	7259 (2)	8520 (1)	0*	22
Cl(1)	−185 (1)	565 (1)	−190 (4)	32
Cl(2)	1630 (1)	1147 (1)	202 (5)	41
P(1)	949 (2)	622 (1)	−2126 (3)	30
P(2)	480 (1)	1080 (1)	2115 (4)	30
N(1)	1028 (5)	278 (3)	558 (12)	31
N(2)	402 (5)	1418 (3)	−610 (12)	31
C(1)	1168 (7)	102 (4)	−1816 (16)	43
C(2)	1537 (9)	862 (5)	−2871 (19)	54
C(3)	396 (8)	574 (7)	−3368 (17)	55
C(4)	104 (7)	1563 (4)	1665 (19)	43
C(5)	−7 (7)	774 (6)	3115 (18)	52
C(6)	1048 (7)	1207 (5)	3264 (16)	46
C(7)	1422 (6)	91 (4)	−408 (17)	42
C(8)	391 (7)	1725 (4)	502 (19)	45
P(3)	1659 (2)	−248 (1)	3895 (5)	43
F(1)	2141 (8)	−162 (9)	2995 (24)	193
F(2)	1306 (6)	−405 (4)	2704 (18)	106
F(3)	1187 (9)	−305 (9)	4824 (23)	207
F(4)	2012 (8)	−110 (6)	5118 (21)	144
F(5)	1462 (11)	177 (5)	3716 (27)	182
F(6)	1858 (12)	−681 (6)	4122 (23)	177

* Parameter kept fixed for origin definition.

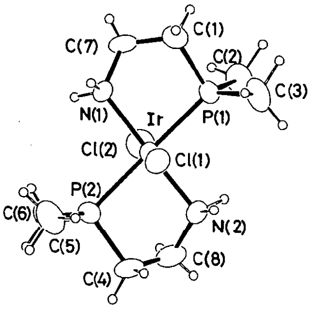

Fig. 1. An *ORTEP* (Johnson, 1965) drawing of the molecule with 50% probability ellipsoids. H atoms are represented by circles of radius 0.08 Å.

Table 2. *Selected bond lengths* (Å) *and bond angles* (°)

Ir—Cl(1)	2.359 (3)	Ir—P(2)	2.326 (4)
Ir—Cl(2)	2.356 (3)	Ir—N(1)	2.090 (10)
Ir—P(1)	2.325 (3)	Ir—N(2)	2.099 (10)
Cl(1)—Ir—Cl(2)	179.2 (1)	P(2)—Ir—N(1)	97.5 (3)
Cl(1)—Ir—P(1)	90.2 (1)	P(2)—Ir—N(2)	83.7 (3)
Cl(1)—Ir—P(2)	88.5 (1)	N(1)—Ir—N(2)	177.9 (4)
Cl(1)—Ir—N(1)	88.6 (3)	Ir—P(1)—C(1)	102.3 (4)
Cl(1)—Ir—N(2)	89.8 (3)	Ir—P(1)—C(2)	117.6 (7)
Cl(2)—Ir—P(1)	90.4 (1)	Ir—P(1)—C(3)	119.7 (6)
Cl(2)—Ir—P(2)	90.9 (1)	Ir—P(2)—C(4)	99.8 (5)
Cl(2)—Ir—N(1)	92.1 (3)	Ir—P(2)—C(5)	118.9 (6)
Cl(2)—Ir—N(2)	89.6 (3)	Ir—P(2)—C(6)	117.9 (5)
P(1)—Ir—P(2)	178.6 (1)	Ir—N(1)—C(7)	114.4 (9)
P(1)—Ir—N(1)	82.9 (3)	Ir—N(2)—C(8)	112.4 (9)
P(1)—Ir—N(2)	95.8 (3)		

Ir(OH)Ir(en)$_2$(OH)](S$_2$O$_6$)$_{3/2}$ClO$_4$.2.75H$_2$O, 2.054 (8)–2.087 (8) Å [en = ethylenediamine (Galsbøl, Larsen, Rasmussen & Springborg, 1986)].

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Trichloromethoxytin(IV) Dimethanolate, SnCl$_3$(OCH$_3$).2CH$_3$OH

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Abstract. $M_r = 320.19$, triclinic, $P\bar{1}$, $a = 7.552$ (2), $b = 8.114$ (2), $c = 9.714$ (4) Å, $\alpha = 93.88$ (2), $\beta = 102.04$ (3), $\gamma = 114.80$ (2)°, $V = 520.3$ (4) Å^3, $Z = 2$, $D_x = 2.045$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 29.57$ cm^{-1}, $F(000) = 308$, $T = 210$ (2) K, $R = 0.056$ for 2651 reflections with $F_o \geq 4\sigma(F_o)$. The structure

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Table 1. *Fractional positional parameters and equivalent isotropic atomic displacement parameters* (Å^2) *for* $SnCl_3(OCH_3).2CH_3OH$, *with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}
Sn(1)	0.45002 (7)	0.57653 (6)	0.34561 (5)	0.028 (1)
Cl(1)	0.6617 (3)	0.8234 (3)	0.2526 (3)	0.053 (1)
Cl(2)	0.1796 (4)	0.4338 (3)	0.1391 (2)	0.050 (1)
Cl(3)	0.3029 (4)	0.7501 (3)	0.4415 (2)	0.044 (1)
O(1)	0.6666 (7)	0.6353 (6)	0.5404 (5)	0.031 (2)
O(2)	0.5743 (9)	0.4103 (8)	0.2685 (6)	0.046 (3)
O(3)	0.3675 (12)	0.0634 (9)	0.2311 (8)	0.071 (4)
C(1)	0.8745 (13)	0.7680 (14)	0.5659 (11)	0.069 (4)
C(2)	0.7290 (21)	0.4444 (18)	0.1988 (18)	0.109 (9)
C(3)	0.2337 (22)	−0.0461 (16)	0.1057 (14)	0.088 (6)

Table 2. *Bond lengths* (Å) *and angles* (°) *for* $SnCl_3(OCH_3).2CH_3OH$, *with e.s.d.'s in parentheses*

Sn(1)—Cl(1)	2.355 (3)	O(1)—C(1)	1.439 (9)
Sn(1)—Cl(2)	2.351 (2)	O(2)—C(2)	1.408 (2)
Sn(1)—Cl(3)	2.378 (3)	O(3)—C(3)	1.372 (2)
Sn(1)—O(1)	2.101 (5)	O(2)···O(3)	2.536 (3)
Sn(1)—O(1^i)	2.086 (5)	O(2)···Cl(1^i)	3.304 (1)
Sn(1)—O(2)	2.117 (8)	O(3)···Cl(1^i)	3.389 (4)
O(1)—Sn(1)—O(1^i)	72.1 (2)	Cl(2)—Sn(1)—Cl(1)	98.3 (1)
O(1)—Sn(1)—O(2)	86.2 (2)	Cl(3)—Sn(1)—O(1)	92.5 (2)
O(1^i)—Sn(1)—O(2)	83.5 (2)	Cl(3)—Sn(1)—O(1^i)	93.0 (2)
Cl(1)—Sn(1)—O(1)	94.4 (2)	Cl(3)—Sn(1)—O(2)	176.5 (2)
Cl(1)—Sn(1)—O(1^i)	165.2 (2)	Cl(3)—Sn(1)—Cl(1)	93.8 (1)
Cl(1)—Sn(1)—O(2)	89.6 (2)	Cl(3)—Sn(1)—Cl(2)	93.8 (1)
Cl(2)—Sn(1)—O(1)	165.4 (2)	Sn(1)—O(1)—Sn(1^i)	107.9 (2)
Cl(2)—Sn(1)—O(1^i)	94.4 (2)	Sn(1)—O(1)—C(1)	123.0 (5)
Cl(2)—Sn(1)—O(2)	86.8 (2)	Sn(1)—O(2)—C(2)	133.5 (7)

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

consists of dimeric $SnCl_3(OCH_3).2CH_3OH$ molecules possessing crystallographic $\bar{1}$ symmetry. The Sn atoms are octahedrally coordinated and bridged by two μ_2-OCH_3 groups. The two additional methanol molecules in the unit cell which complete the crystal structure are bonded *via* hydrogen bonds to the two solvent molecules coordinated to the Sn atoms.

Experimental. Trichloromethoxytin(IV) dimethanolate was unexpectedly formed by the reaction of phenyltin trichloride with an excess of anhydrous methanol. Suitable single crystals of the air-sensitive compound were obtained by slow evaporation of the organic solvent.

X-ray diffraction experiments were performed at $T = 210\,(2)$ K with an Enraf–Nonius CAD-4 diffractometer using graphite-monochromated Mo $K\alpha$ radiation and a colourless transparent single crystal of approximate dimensions 0.34 × 0.28 × 0.31 mm which was embedded on the top of a glass fibre in epoxy resin to protect it from moisture. Unit-cell parameters were obtained by least-squares refinement of the setting angles of 25 well centered reflections in the range $20.14 \leq 2\theta \leq 28.76°$. 3190 intensity data were collected using an $\omega/2\theta$ scan mode to $2\theta_{max} = 50°$ ($h_{min,max} = -10,10$; $k_{min,max} = -11,11$; $l_{min,max} = 0,13$). Four standard reflections measured every 200 reflections and every hour of X-ray exposure indicated no significant crystal movement or intensity decay over the period of data collection. Data were corrected for Lorentz and polarization effects, but not for those of absorption. 154 reflections measured twice afforded a merging R_{int} value of 0.037. Of the 2936 unique reflections, 2651 with $F_o \geq 4\sigma(F_o)$ were used for structure solution and refinement.

The structure was solved by direct methods with the program *SHELXS*86 (Sheldrick, 1986) and refined by full-matrix least squares with the program *SHELX*76 (Sheldrick, 1976) minimizing $\Sigma w(\Delta F)^2$ with $w = 1/\sigma^2(F)$. Final refinement ($\Delta/\sigma \leq 0.01$) using 91 parameters converged to conventional R values of $R = 0.056$ and $wR = 0.056$. With the exception of the H atoms all atoms were refined with anisotropic thermal parameters. Beside two peaks (2.10/1.89 e Å^{-3}) less than 0.9 Å apart from Sn(1) the final difference electron density map showed only insignificant features ≤ 0.9 e Å^{-3} ($\Delta\rho_{min} = -1.56$ e Å^{-3}). Neutral-atom scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV) and corrected for the real part of the anomalous dispersion. The fractional coordinates and equivalent isotropic displacement factors are listed in Table 1.* Selected bond lengths and angles are listed in Table 2. Fig. 1 shows the perspective view of the dimeric molecule and the atomic numbering scheme used. Fig. 1 was designed by the program *KPLOT* (Hundt, 1979) and executed with the program *ORTEP* (Johnson, 1965).

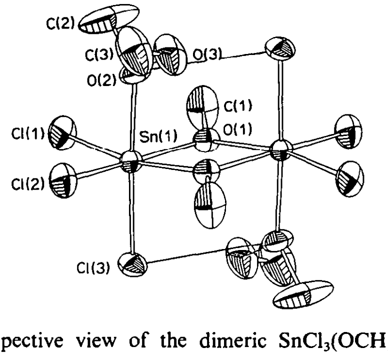

Fig. 1. Perspective view of the dimeric $SnCl_3(OCH_3).2CH_3OH$ molecule showing the atomic numbering scheme used.

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* Lists of structure factors, anisotropic displacement parameters and a diagram of the crystal packing have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54832 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BX0564]

Related literature. The structural features of the title compound are consistent with those of other dimeric tin(IV) chloride and mono-organotin trichloride derivatives such as $SnCl_3(OR).ROH$ [R = Me (Sterr & Mattes, 1963), R = Et (Webster & Collins, 1974)]; $SnCl_3(OH).H_2O.L$ (Barnes, Sampson & Weakly, 1980); $Sn(O^iPr)_4.^iPrOH$ (Hampden-Smith, Wark, Rheingold & Huffman, 1991; Reuter & Kremser, 1991); and $RSn(OH)Cl_2.H_2O$ [R = Et (Lecomte, Protas & Devaud, 1976), R = ^{n}Bu (Holmes, Shafieezad, Chandrasekhar, Holmes & Day, 1988), R = ^{i}Pr, ^{i}Bu (Puff & Reuter, 1989)]. In all these cases the two octahedrally coordinated Sn atoms are bridged by two μ_2-OH or μ_2-OR groups. Differences only exist with respect to crystal packing and the formation of additional hydrogen bridges to further solvent molecules. In the present case a second methanol molecule is bonded *via* a short hydrogen bond to the methanol molecule which coordinates directly with the Sn atom and forms a weak hydrogen bond to the adjacent Cl atom.

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Acta Cryst. (1992). C**48**, 1114–1116

Structure of Aqua{[3,3′-(1,3-propanediyldinitrilo)bis(2-butanone oximato)]-*N*,*N*′,*N*″,*N*‴}(2,2,2-trifluoroethyl)cobalt(III) Hexafluorophosphate

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Abstract. $[Co(C_{11}H_{19}N_4O_2)(C_2H_2F_3)(H_2O)]PF_6$, M_r = 544.2, monoclinic, $P2_1/c$, a = 6.906 (3), b = 14.545 (3), c = 21.236 (9) Å, β = 99.05 (2)°, V = 2106 (1) Å^3, Z = 4, D_x = 1.72 g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, μ = 9.8 cm^{-1}, $F(000)$ = 1104, T = 293 K, R = 0.046 for 3583 unique reflections with $I > 3\sigma(I)$. The axial fragment of the distorted octahedral polyhedron of Co is characterized by Co—C and Co—O distances of 2.007 (4) and 2.066 (3) Å, respectively. The geometry of the CH_2CF_3 group departs significantly from that expected for sp^3 hybridization at the C atom bearing the F substituents (*cis* steric influence).

Experimental. The complex was prepared as previously reported (Parker, Bresciani-Pahor, Zangrando, Randaccio & Marzilli, 1985). Red–brown crystal with approximate dimensions 0.20 × 0.20 × 0.60 mm, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, lattice constants by least squares refinement of setting angles of 25 reflections with $14 \leq \theta \leq 22°$. Measurements carried out at room temperature, three standard reflections measured every 4000 s showed no significant variation in intensity, ω–2θ scan; scan angle $(0.70 + 0.35\tan\theta)°$. 5403 reflections measured in the range $3 \leq \theta \leq 28°$, with $-9 \leq h \leq 9$, $0 \leq k \leq 19$, $0 \leq l \leq 27$. The intensities of 3583 unique reflections with $I > 3\sigma(I)$ were corrected for Lorentz–polarization effects and for absorption based on empirical ψ scans ($T_{\max,\min}$ = 99.7, 86.6%), and used for structure determination. The structure was solved by conventional Patterson and Fourier methods and refined through full-matrix least-squares calculations with $\sum w(|F_o| - |F_c|)^2$ minimized. 316 parameters refined, anisotropic temperature factors for all

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