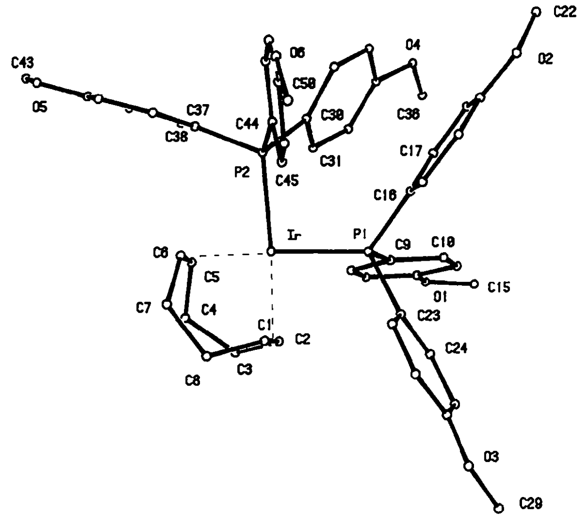

Fig. 1. Molecular structure and numbering scheme for $[Ir\{P(4\text{-}MeOC_6H_4)_3\}_2(cod)]^+$.

Chaloner, 1989), and is related to $[Ir(PCy_3)(py)(cod)][PF_6]$, which has been widely used as a catalyst for the reduction of hindered alkenes (Crabtree, 1979). The structures of $[Ir(PCy_3)(py)(cod)][PF_6]$ and $[Ir\{P(2\text{-}MeOC_6H_4)_3\}(py)][PF_6]$ have been determined, and show distortions due to the bulk of the phosphine ligands (Abbassioun, Hitchcock & Chaloner, 1989*a*). The conformation of the cod ligand is similar in these complexes. The structure of $[Ir(py)_2(cod)][BPh_4]$, containing the precursor cation, has also been established (Abbassioun, Hitchcock & Chaloner, 1989*b*)

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Structure of Difluorobis(trifluoromethyl)tellurium

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Abstract. $[TeF_2(CF_3)_2]$, $M_r = 303{\cdot}61$, monoclinic, $C2/c$, $a = 22{\cdot}065\ (4)$, $b = 9{\cdot}764\ (2)$, $c = 8{\cdot}981\ (3)$ Å, $\beta = 91{\cdot}66\ (2)°$, $V = 1934{\cdot}1\ (8)$ Å^3, $Z = 12$, $D_x = 3{\cdot}128$ Mg m^{-3}, $F(000) = 1632$, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 4{\cdot}71$ mm^{-1}, $T = 291\ (1)$ K, final $R = 0{\cdot}039$ for 2474 unique observed $[F \geq 4{\cdot}0\sigma(F)]$ diffractometer data. The asymmetric unit of the crystal contains one and a half $(CF_3)_2TeF_2$ molecules. The surrounding of each of the two independent Te atoms can roughly be described as a trigonal bipyramid with two C atoms and the non-bonding electron pair in the equatorial plane. The crystal contains intermolecular Te···F and F···F distances which are significantly shorter than the sum of the corresponding van der Waals radii.

Experimental. The title compound was prepared from $Te(CF_3)_2$ with XeF_2 (Naumann & Herberg,

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Table 1. *Atomic coordinates and equivalent isotropic displacement parameters* ($Å^2 \times 10^4$)

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

	x	y	z	U_{eq}
Te(1)	0·0	0·06454 (4)	0·25	343
Te(2)	0·33436 (1)	0·31036 (3)	0·42007 (3)	367
F(1)	−0·0475 (1)	0·1038 (4)	0·0655 (3)	515
F(2)	0·3812 (2)	0·4267 (4)	0·2856 (4)	612
F(3)	0·2909 (1)	0·1493 (3)	0·4979 (4)	514
F(11)	−0·1089 (2)	0·2382 (5)	0·2679 (5)	774
F(12)	−0·0329 (2)	0·3312 (4)	0·3751 (7)	933
F(13)	−0·0761 (3)	0·1679 (6)	0·4797 (5)	1094
F(21)	0·2371 (2)	0·1895 (4)	0·2308 (5)	711
F(22)	0·2512 (2)	0·4009 (5)	0·1840 (6)	953
F(23)	0·3117 (2)	0·2487 (7)	0·1046 (5)	1096
F(24)	0·3725 (2)	0·0549 (5)	0·2831 (7)	1075
F(25)	0·4170 (2)	0·0907 (5)	0·4940 (5)	827
F(26)	0·4453 (2)	0·1941 (5)	0·3027 (7)	1046
C(11)	−0·0603 (3)	0·2136 (6)	0·3510 (7)	511
C(21)	0·2787 (3)	0·2827 (7)	0·2163 (7)	550
C(22)	0·3984 (2)	0·1486 (7)	0·3672 (7)	559

Table 2. *Bond distances* (Å) *and angles* (°), *torsion angles* (°) *and short intermolecular distances* (Å)

Te(1)—F(1)	1·973 (3)	F(22)—C(21)	1·332 (8)
Te(1)—C(11)	2·187 (6)	F(23)—C(21)	1·300 (8)
Te(2)—F(2)	1·972 (4)	F(24)—C(22)	1·307 (8)
Te(2)—F(3)	1·980 (3)	F(25)—C(22)	1·326 (8)
Te(2)—C(21)	2·191 (6)	F(26)—C(22)	1·280 (8)
Te(2)—C(22)	2·181 (6)	Te(1)···F(2*b*)	2·973 (3)
F(11)—C(11)	1·309 (7)	Te(2)···F(1*c*)	3·000 (3)
F(12)—C(11)	1·314 (7)	Te(2)···F(3*d*)	2·908 (3)
F(13)—C(11)	1·296 (8)	F(3)···F(3*d*)	2·670 (4)
F(21)—C(21)	1·302 (8)	F(26)···F(26*e*)	2·617 (7)
F(1)—Te(1)—F(1*a*)	157·6 (1)	Te(1)—C(11)—F(12)	111·5 (4)
F(1)—Te(1)—C(11*a*)	80·6 (2)	Te(1)—C(11)—F(11)	112·6 (4)
C(11)—Te(1)—C(11*a*)	96·5 (2)	F(22)—C(21)—F(23)	108·4 (6)
F(1)—Te(1)—C(11)	84·5 (2)	F(21)—C(21)—F(23)	108·1 (6)
C(21)—Te(2)—C(22)	94·6 (2)	F(21)—C(21)—F(22)	108·0 (5)
F(3)—Te(2)—C(22)	79·9 (2)	Te(2)—C(21)—F(23)	111·3 (4)
F(3)—Te(2)—C(21)	86·1 (2)	Te(2)—C(21)—F(22)	108·4 (4)
F(2)—Te(2)—C(22)	86·0 (2)	Te(2)—C(21)—F(21)	112·5 (4)
F(2)—Te(2)—C(21)	81·4 (2)	F(25)—C(22)—F(26)	107·6 (5)
F(2)—Te(2)—F(3)	160·3 (1)	F(24)—C(22)—F(26)	109·1 (6)
F(12)—C(11)—F(13)	106·8 (5)	F(24)—C(22)—F(25)	108·4 (6)
F(11)—C(11)—F(13)	109·6 (5)	Te(2)—C(22)—F(26)	112·7 (4)
F(11)—C(11)—F(12)	107·4 (5)	Te(2)—C(22)—F(25)	107·9 (4)
Te(1)—C(11)—F(13)	108·8 (4)	Te(2)—C(22)—F(24)	110·9 (4)
F(12)—C(11)—Te(1)—C(11*a*)	−24·8 (5)	F(24)—C(22)—Te(2)—C(21)	28·0 (5)
F(23)—C(21)—Te(2)—C(22)	34·7 (5)		

Symmetry codes: (*a*) $-x, y, 0·5-z$; (*b*) $x-0·5, y-0·5, z$; (*c*) $0·5+x, 0·5-y, 0·5+z$; (*d*) $0·5-x, 0·5-y, 1-z$; (*e*) $1-x, y, 0·5-z$.

1982). Colourless crystals were obtained by slow sublimation *in vacuo* at 343 K. Crystal size ~0·47 × 0·37 × 0·90 mm, D_m not determined, $\omega/2\theta$ scan, scan speed 0·50–2·50° min^{-1} in θ; Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$; lattice parameters from least-squares fit with 25 reflections up to $2\theta = 33·78°$; ω scans of low-order reflections along the three crystal axes showed acceptable mosaicity; three standard reflections (18,0,0, 006, 080) recorded every 2·5 h, showed intensity loss up to 40% over 344·03 h of X-ray exposure; 6199 reflections measured, $4·0 \le 2\theta \le 60·0°$, $-31 \le h \le 31$, $-13 \le k \le 13$, $0 \le l \le 12$; after averaging ($R_{int} = 0·018$): 2814 unique reflections, 2474 with $F \ge 4·0\sigma(F)$; Lorentz–polarization correction, decay correction (maximum correction factor 1·70, minimum 0·97, average 1·16) and absorption correction *via* ψ scans, max./min. transmission 1·00/0·92 (average 0·95); systematic absences (hkl) $h+k=2n+1$ and ($h0l$) with $h=2n+1$ and $l=2n+1$ conform to space groups $C2/c$ and Cc; structure solution in space group $C2/c$ *via* Patterson function, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all atoms; refinement on F with 2474 reflections and 150 refined parameters; $w = 1·0/[\sigma^2(F) + (0·0037F^2)]$ which led to featureless analysis of variance in terms of $\sin\theta$ and F_o; $S = 2·68$, $R = 0·039$, $wR = 0·043$ ($R = 0·044$, $wR = 0·060$ with all data), $(\Delta/\sigma)_{max} = 0·01$, largest peak in final ΔF map $\pm 2·1$ (4) e Å^{-3} near Te and

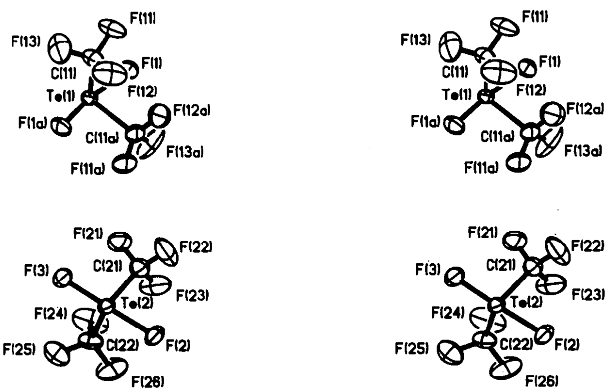

Fig. 1. Stereoscopic view (*SHELXTL-PLUS*) of the molecules, showing the atomic numbering scheme [symmetry code: (*a*) $-x, y, 0·5-z$].

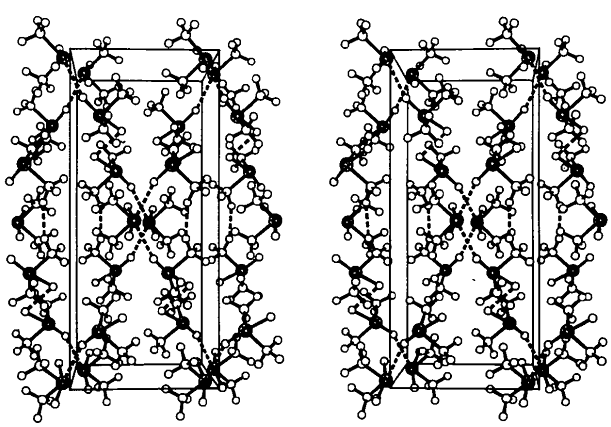

Fig. 2. Stereoscopic view (*SCHAKAL*) of the unit cell (**a** vertical, **b** horizontal). Short intermolecular Te···F contacts lower than 3·0 Å and F···F contacts lower than 2·7 Å are indicated by broken lines.

±0·6 (4) e Å^{-3} in the remaining parts of the cell; no extinction correction; atomic scattering factors for neutral atoms and real and imaginary dispersion terms from *International Tables for X-ray Crystallography* (1974); programs: Enraf–Nonius *Structure Determination Package* (Frenz, 1985), *PARST* (Nardelli, 1983), *SHELXTL-PLUS* (Sheldrick, 1987), *SCHAKAL* (Keller, 1986), *PCK*83 (Williams, 1984), *PLATON* (Spek, 1982), *MISSYM* (Le Page, 1987). The molecules and the numbering scheme are shown in Fig. 1 and a stereoscopic view of the unit cell in Fig. 2. Positional parameters and the equivalent values of the displacement parameters are given in Table 1.* Bond lengths and angles, torsion angles and short intermolecular distances are given in Table 2.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52581 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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9-Ethylguaninium Tetrachloroaurate(III) Hydrate

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Abstract. 2-Amino-9-ethyl-6-oxo-1,6-dihydro-7H^+,9H-purinium tetrachloroaurate(III) hydrate, $C_7H_{10}N_5O^+.AuCl_4^-.H_2O$, M_r = 536·98, monoclinic, $P2_1/n$, a = 7·345 (2), b = 14·976 (4), c = 13·366 (5) Å, β = 93·35 (3)°, V = 1467·7 (8) Å^3, Z = 4, D_x = 2·430 Mg m^{-3}, $F(000)$ = 1008, λ(Mo $K\alpha$) = 0·71073 Å, μ = 10·74 mm^{-1}, T = 291 (1) K, final R = 0·039 for 2378 unique observed [$F \geq 3·0\sigma(F)$] diffractometer data. The title compound consists of discrete 9-ethylguaninium cations, tetrachloroaurate(III) anions and water molecules. The $[AuCl_4]^-$ anion is planar with Au—Cl distances in the range 2·277 (2)–2·283 (2) Å.

Experimental. Orange-yellow needles of the title compound were obtained in 81% yield by cocrystallization of 9-ethylguanine and $NaAuCl_4$ in 0·1 *M* HCl and subsequent recrystallization from water at 276 K. A crystal of size ~0·26 × 0·04 × 0·12 mm was used, D_m was not determined. Intensity data were collected with $\omega/2\theta$ scans, scan speed 4–15° min^{-1} in θ and scan width 1·2° + dispersion. A Nicolet *R3m/V* diffractometer with graphite-monochromated Mo $K\alpha$ radiation was used. The lattice parameters were determined from least-squares fit of 28 reflections with $2\theta \leq 33·09°$; ω scans of low-order reflections along the three crystal axes showed acceptable mosaicity. Six standard reflections (200, 020, 002, $\bar{2}00$, $0\bar{2}0$, $00\bar{2}$) were recorded every 2·5 h, only random deviations were detected during 158 h of X-ray exposure; 10023 reflections with 3·0 $\leq 2\theta \leq$ 50·0°, $-9 \leq h \leq 9$, $-18 \leq k \leq 18$, $-16 \leq l \leq 16$ were measured. The data were corrected for Lorentz–polarization and absorption effects, the latter *via* ψ scans; the max./min. transmission factors were 1·00/0·53. The intensities were averaged (R_{int} = 0·065) to 2593 unique reflections, 2378 of which had $F \geq 3·0\sigma(F)$. The systematic absences ($h0l$) $h + l = 2n + 1$, ($0k0$) $k = 2n + 1$ conform to space group $P2_1/n$. The structure was solved from Patterson function and $\Delta\rho$ maps. It was refined (on F) using full-matrix least squares with anisotropic temperature factors for

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