C17	0.5924 (4)	0.6973 (3)	0.1896 (5)	0.110 (5)
C18	0.5143 (4)	0.6753 (2)	0.2141 (4)	0.075 (4)
C101	0.3215 (3)	0.4941 (2)	0.4993 (3)	0.037 (2)
C102	0.1891 (3)	0.4721 (2)	0.6219 (4)	0.062 (3)
C103	0.1185 (3)	0.4371 (2)	0.3891 (4)	0.043 (3)
C104	0.0248 (3)	0.4447 (2)	0.3085 (4)	0.051 (3)
C105	−0.0295 (3)	0.4016 (2)	0.2439 (4)	0.072 (3)
C106	0.0112 (4)	0.3478 (2)	0.2579 (4)	0.080 (4)
C107	0.1050 (4)	0.3374 (2)	0.3336 (5)	0.087 (4)
C108	0.1570 (3)	0.3829 (2)	0.3981 (4)	0.071 (3)
C109	0.1256 (3)	0.5644 (2)	0.4432 (4)	0.044 (3)
C110	0.1143 (3)	0.5861 (2)	0.3450 (4)	0.061 (3)
C111	0.0654 (4)	0.6371 (2)	0.3077 (4)	0.077 (4)
C112	0.0287 (4)	0.6666 (2)	0.3693 (5)	0.099 (4)
C113	0.0392 (4)	0.6477 (2)	0.4678 (6)	0.107 (5)
C114	0.0880 (4)	0.5958 (2)	0.5045 (4)	0.068 (4)
C201	0.5618 (3)	0.5605 (2)	0.6727 (3)	0.033 (2)
C202	0.6254 (4)	0.6829 (2)	0.7465 (4)	0.080 (3)
C203	0.7215 (3)	0.5847 (2)	0.8996 (3)	0.037 (2)
C204	0.7374 (3)	0.5818 (2)	1.0065 (4)	0.055 (3)
C205	0.8309 (4)	0.5670 (2)	1.0851 (4)	0.065 (3)
C206	0.9075 (3)	0.5556 (2)	1.0550 (4)	0.063 (3)
C207	0.8949 (3)	0.5568 (2)	0.9505 (4)	0.057 (3)
C208	0.8027 (3)	0.5712 (2)	0.8732 (4)	0.050 (3)
C209	0.4993 (3)	0.6127 (2)	0.8421 (4)	0.067 (3)
C210	0.4525 (5)	0.5669 (3)	0.8525 (7)	0.176 (7)
C211	0.3758 (7)	0.5700 (7)	0.8869 (9)	0.39 (2)
C212	0.3457 (5)	0.6076 (5)	0.9173 (6)	0.26 (1)
C213	0.3869 (6)	0.6629 (4)	0.9059 (6)	0.217 (8)
C214	0.4664 (6)	0.6626 (3)	0.8706 (6)	0.160 (7)

Table 12. *Geometric parameters* (Å, °) *for* (VI)

Ni—P	2.368 (1)	Si1—C101	1.908 (5)
Ni—O11	2.001 (3)	Si2—C201	1.914 (4)
Ni—O12	2.013 (3)	O11—C101	1.249 (6)
Ni—O21	2.002 (3)	O12—C101^{i}	1.275 (5)
Ni—O22	2.013 (3)	O21—C201	1.259 (5)
Ni—Ni^{i}	2.7079 (8)	O22—C201^{i}	1.268 (5)
P—Ni—O11	93.78 (8)	O21—Ni—Ni^{i}	79.19 (8)
P—Ni—O12	100.23 (9)	O22—Ni—Ni^{i}	87.17 (8)
P—Ni—O21	90.86 (8)	Ni—O11—C101	132.0 (2)
P—Ni—O22	103.07 (8)	Ni—O12—C101^{i}	117.9 (3)
P—Ni—Ni^{i}	166.66 (5)	Ni—O21—C201	129.7 (2)
O11—Ni—O12	166.0 (1)	Ni—O22—C201^{i}	119.2 (3)
O11—Ni—O21	91.5 (1)	Si1—C101—O11	119.5 (3)
O11—Ni—O22	88.5 (1)	Si1—C101—O12^{i}	116.6 (3)
O11—Ni—Ni^{i}	77.78 (8)	O11—C101—O12^{i}	123.9 (4)
O12—Ni—O21	87.8 (1)	Si2—C201—O21	116.9 (3)
O12—Ni—O22	88.8 (1)	Si2—C201—O22^{i}	118.5 (3)
O12—Ni—Ni^{i}	88.35 (9)	O21—C201—O22^{i}	124.5 (4)
O21—Ni—O22	166.0 (1)		

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

In compound (II), the chloroform solvent molecules exhibit rotational disorder. Four possible positions of the Cl atoms around C301, labeled as Cl1—Cl4, were each assumed to have occupation probabilities of 3/4 and were refined with a rigid-body approximation. The relatively large *R* value for (II) may be the result of the disorder of the $CHCl_3$ molecule.

The programs used to solve the structures were *Xtal*3.0 *FOURR* and *Xtal*3.0 *SIMPEL* (Hall & Stewart, 1990). The structures were refined using *Xtal*3.0 *CRYSLQ*, molecular graphics were produced using *Xtal*3.0 *ORTEP* and material produced for publication with *Xtal*3.0 *BONDLA* and *Xtal*3.0 *CIFIO*. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B) were used for non-H atoms. For the H atoms the values were taken from Stewart, Davidson & Simpson (1965).

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55199 (105 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1007]

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Structure of [2-(Fluorodimethylstannyl)-ethyl]diphenylphosphine Oxide

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Abstract

The atoms bound to Sn form a distorted trigonal bipyramid with the O and F atoms in the apical positions [Sn—O 2.454(3), Sn—F 2.035(2) Å; O—Sn—F 172.66(9)°] and the C atoms in the equatorial positions [Sn—C 2.123(5), 2.124(4), 2.158(3) Å; C—Sn—C 123.9(2), 115.7(2), 118.2(2)°; C—Sn—F 93.6(1), 94.3(1), 97.3(1)°; C—Sn—O 80.5(1), 85.5(1), 89.2(2)°]. The atoms bound to P form a slightly distorted tetrahedron with bond angles in the range 106.2(2)-112.1(2)°. The five-membered ring has an envelope conformation; the atoms P, O, Sn and C(3) are nearly coplanar.

Comment

The compounds [2-(bromodimethylstannyl)ethyl]diphenylphosphine sulfide (Preut, Godry & Mitchell, 1992*a*) and [2-(chlorodimethylstannyl)ethyl]diphenylphosphine

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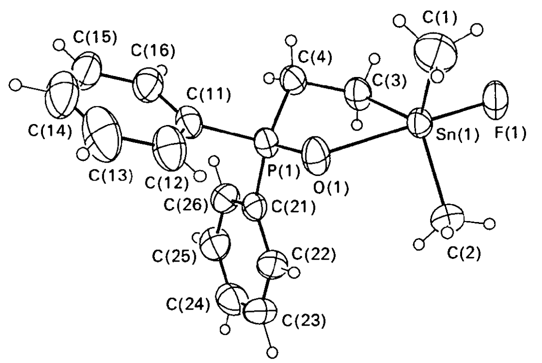

Fig. 1. General view (*SHELXTL-Plus* graphic) of the molecule showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries. H atoms are represented as spheres of arbitrary radius.

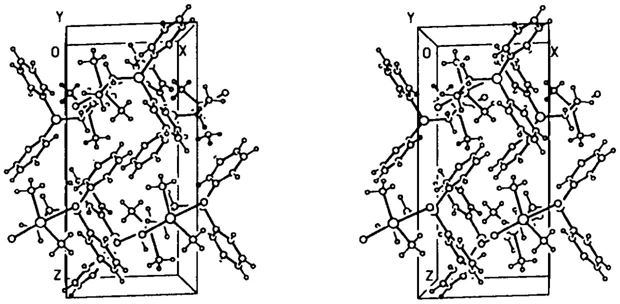

Fig. 2. Stereoscopic view (*SHELXTL-Plus* graphic) of the unit cell.

selenide (Preut, Godry & Mitchell, 1992*b*) are isomorphous and crystallize in the space group $P2_12_12_1$. We are undertaking a series of crystal structure determinations of the compounds $C_{16}H_{20}XYPSn$ (X = O, S, Se; Y = F, Cl, Br, I) in order to investigate systematically the influence of stepwise variations in the composition of the molecules on their geometry and packing in the crystal.

Experimental

Crystal data

$C_{16}H_{20}FOPSn$
M_r = 397.00
Monoclinic
$P2_1/n$
a = 7.768 (2) Å
b = 13.524 (4) Å
c = 16.066 (4) Å
β = 91.91 (2)°
V = 1686.9 (8) Å^3
Z = 4
D_x = 1.563 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 50 reflections
θ = 7.8-15.0°
μ = 1.62 mm^{-1}
T = 293 (1) K
Plate
0.30 × 0.28 × 0.14 mm
Colourless

Data collection

Nicolet *R*3*m*/*V* diffractometer
$\theta/2\theta$ scans

$R_{\rm int}$ = 0.038
$\theta_{\rm max}$ = 50.0°
$h = -10 \rightarrow 10$

Absorption correction: empirical
$T_{\rm min}$ = 1.00, $T_{\rm max}$ = 0.90
6390 measured reflections
2992 independent reflections
2493 observed reflections
[$F>4.0\sigma(F)$]

$k = 0 \rightarrow 17$
$l = 0 \rightarrow 20$
6 standard reflections
frequency: 150 min
intensity variation: none

Refinement

Refinement on F
Final R = 0.027
wR = 0.038
S = 0.81
2493 reflections
182 parameters
Only H-atom U's refined
Calculated weights
$w = 1/[\sigma^2(F)+0.0010F^2]$

$(\Delta/\sigma)_{\rm max}$ = 0.007
$\Delta\rho_{\rm max}$ = 0.45 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.77 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Data collection: Nicolet *R*3*m*/*V* software, release 4.11. Cell refinement: Nicolet *R*3*m*/*V* software. Data reduction: Nicolet *R*3*m*/*V* software. Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1987). Program(s) used to refine structure: *SHELXTL-Plus*. Software used to prepare material for publication: *PARST* (Nardelli, 1983), *PLATON* (Spek, 1982), *MISSYM* (Le Page, 1987) and *PCK*83 (Williams, 1984).

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

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Sn(1)	0.19861 (3)	0.04711 (2)	0.27952 (2)	0.0381
P(1)	−0.0820 (1)	0.22077 (6)	0.31989 (6)	0.0358
F(1)	0.4197 (3)	0.0084 (2)	0.2234 (2)	0.0569
O(1)	−0.0718 (3)	0.1105 (2)	0.3336 (2)	0.0485
C(1)	0.2683 (7)	−0.0051 (4)	0.4007 (3)	0.0731
C(2)	0.0392 (5)	−0.0441 (3)	0.2018 (3)	0.0532
C(3)	0.2364 (5)	0.2032 (2)	0.2589 (3)	0.0464
C(4)	0.1341 (4)	0.2685 (2)	0.3172 (3)	0.0408
C(11)	−0.1967 (4)	0.2822 (3)	0.4008 (2)	0.0414
C(12)	−0.3219 (6)	0.2281 (3)	0.4407 (3)	0.0607
C(13)	−0.4185 (7)	0.2753 (5)	0.5027 (3)	0.0824
C(14)	−0.3859 (7)	0.3711 (4)	0.5227 (3)	0.0781
C(15)	−0.2607 (7)	0.4238 (4)	0.4840 (3)	0.0690
C(16)	−0.1656 (6)	0.3797 (3)	0.4222 (2)	0.0526
C(21)	−0.1941 (4)	0.2516 (3)	0.2232 (2)	0.0371
C(22)	−0.2982 (5)	0.1817 (3)	0.1838 (3)	0.0515
C(23)	−0.3926 (5)	0.2067 (4)	0.1124 (3)	0.0658
C(24)	−0.3834 (6)	0.3003 (4)	0.0799 (3)	0.0667
C(25)	−0.2795 (6)	0.3719 (3)	0.1185 (2)	0.0569
C(26)	−0.1852 (5)	0.3474 (3)	0.1903 (2)	0.0462

Table 2. *Geometric parameters* (Å, °)

Sn(1)—F(1)	2.035 (2)	C(11)—C(16)	1.382 (5)
Sn(1)—O(1)	2.454 (3)	C(12)—C(13)	1.418 (7)
Sn(1)—C(1)	2.123 (5)	C(13)—C(14)	1.357 (8)
Sn(1)—C(2)	2.124 (4)	C(14)—C(15)	1.372 (8)
Sn(1)—C(3)	2.158 (3)	C(15)—C(16)	1.391 (6)
P(1)—O(1)	1.509 (3)	C(21)—C(22)	1.384 (5)
P(1)—C(4)	1.801 (3)	C(21)—C(26)	1.401 (5)
P(1)—C(11)	1.803 (4)	C(22)—C(23)	1.383 (6)
P(1)—C(21)	1.804 (3)	C(23)—C(24)	1.371 (7)
C(3)—C(4)	1.530 (5)	C(24)—C(25)	1.394 (7)
C(11)—C(12)	1.391 (6)	C(25)—C(26)	1.386 (6)
C(2)—Sn(1)—C(3)	123.9 (2)	P(1)—C(4)—C(3)	108.1 (2)
C(1)—Sn(1)—C(3)	115.7 (2)	P(1)—C(11)—C(16)	122.2 (3)
C(1)—Sn(1)—C(2)	118.2 (2)	P(1)—C(11)—C(12)	117.4 (3)

O(1)—Sn(1)—C(3)	80.5 (1)	C(12)—C(11)—C(16)	120.4 (4)
O(1)—Sn(1)—C(2)	85.5 (1)	C(11)—C(12)—C(13)	118.8 (4)
O(1)—Sn(1)—C(1)	89.2 (2)	C(12)—C(13)—C(14)	119.8 (5)
F(1)—Sn(1)—C(3)	93.6 (1)	C(13)—C(14)—C(15)	121.2 (5)
F(1)—Sn(1)—C(2)	94.3 (1)	C(14)—C(15)—C(16)	120.1 (5)
F(1)—Sn(1)—C(1)	97.3 (2)	C(11)—C(16)—C(15)	119.6 (4)
F(1)—Sn(1)—O(1)	172.66 (9)	P(1)—C(21)—C(26)	120.7 (3)
C(11)—P(1)—C(21)	106.2 (2)	P(1)—C(21)—C(22)	119.6 (3)
C(4)—P(1)—C(21)	108.7 (2)	C(22)—C(21)—C(26)	119.6 (3)
C(4)—P(1)—C(11)	109.6 (2)	C(21)—C(22)—C(23)	119.8 (4)
O(1)—P(1)—C(21)	112.1 (2)	C(22)—C(23)—C(24)	120.6 (4)
O(1)—P(1)—C(11)	112.0 (2)	C(23)—C(24)—C(25)	120.5 (4)
O(1)—P(1)—C(4)	108.3 (2)	C(24)—C(25)—C(26)	119.2 (4)
Sn(1)—O(1)—P(1)	109.6 (1)	C(21)—C(26)—C(25)	120.2 (4)
Sn(1)—C(3)—C(4)	113.3 (2)		
O(1)—Sn(1)—C(3)—C(4)	24.9 (2)	O(1)—P(1)—C(4)—C(3)	47.9 (3)
C(3)—Sn(1)—O(1)—P(1)	4.2 (2)	Sn(1)—C(3)—C(4)—P(1)	−45.7 (3)
C(4)—P(1)—O(1)—Sn(1)	−26.9 (2)		

The title compound was prepared by the dropwise addition of hydrogen peroxide to a stirred suspension of [2-(fluorodimethylstannyl)ethyl]diphenylphosphine in acetone until the solution became clear. Recrystallization of the crude product from methylene chloride/hexane yielded colourless crystals, m.p. 432–433 K. The yield was 42%. $ZC_{16}H_{20}ClOPSn$: C 48.4% (calculated), 48.3% (found), H 5.1% (calculated), 5.3% (found). [2-(Fluorodimethylstannyl)ethyl]diphenylphosphine was prepared by stirring [2-(chlorodimethylstannyl)ethyl]diphenylphosphine with a fivefold molar excess of potassium fluoride in a degassed mixture of water and methanol for 12 h. The white precipitate was washed with water/methanol and vacuum dried; m.p. > 473 K, yield 78%. The crystal was mounted on the tip of a glass fibre with shellac. The lattice constants were determined from a symmetry-constrained least-squares fit. ω scans of low-order reflections along the three crystal axes showed acceptable mosaicity. H atoms were placed in calculated positions and a common isotropic displacement parameter refined. The weighting scheme used led to an analysis of variance which was featureless in terms of sinθ and F. A search for higher symmetries was performed using *MISSYM* (Le Page, 1987).

Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55286 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1011]

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Structure of a Dichromium(II) Complex with Bridging Betaine Ligands, $[Cr_2(O_2CCH_2NMe_3)_4(OH_2)_2][ZnCl_4]_2.2H_2O$

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Abstract

The structure of diaqua-tetrakis(μ-*N*,*N*,*N*-trimethylglycinato-*O*,*O'*)-dichromium(II) bis[tetrachlorozincate(II)] dihydrate is described. A Cr—Cr quadruple bond of length 2.446 (2) Å is bridged by four zwitterionic betaine ligands with Cr—O bond lengths in the range 2.016 (4)–2.027 (3) Å. The axial coordination site of each Cr atom, opposite the Cr—Cr bond, is occupied by a water molecule with Cr—O 2.243 (5) Å. The cationic charge is balanced by tetrahedral $[ZnCl_4]^{2-}$ anions. The axial aqua ligand is hydrogen bonded to additional water of crystallization.

Comment

Chromium(II) acetate monohydrate has been known for about a century and a half. With its deep red colour, feeble paramagnetism and acetate-bridged binuclear structure (van Niekerk, Schoening & de Wet, 1953; Cotton, de Boer, Laprade, Pipal & Ucko, 1970, 1971), it differs markedly from the blue mononuclear chromium(II) salts. It is also the prototype of a large variety of quadruple-bonded complexes (Cotton & Walton, 1982) which contain simple alkyl or aryl carboxylates as bridging ligands and either neutral or anionic ligands in the axial positions (Ford, Larkworthy, Povey & Roberts, 1983; Larkworthy & Roberts, 1982).

In contrast, the analogous complexes in which the carboxyl groups of amino acids act as bridges remain relatively unexplored. Two types of chromium(II) complex with glycine were reported by Herzog & Kalies (1967). The first of these, $[Cr(O_2CCH_2NH_2)_2.H_2O]$, appears, from magnetic susceptibility measurements, to be a mononuclear high-spin complex. The second, originally formulated as $[Cr(O_2CCH_2NH_2)_2.2HBr.2H_2O]$, was subsequently shown, along with the chloride analogue, to contain the binuclear cation $[Cr_2(O_2CCH_2NH_3)_4Br_2]^{2+}$, with zwitterionic glycine bridging ligands (Ardon, Bino,

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