SHORT-FORMAT PAPERS

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Structures of Two Acetylacetonate Complexes: (I) Sb(acac)Cl_4, (II) [$NHEt_3$][Sn(acac)Cl_4]

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Abstract. (I) (Acetylacetonato)tetrachloroantimony(V), [$Sb(C_5H_7O_2)Cl_4$], $M_r = 362{\cdot}7$, orthorhombic, $C222_1$, $a = 6{\cdot}215$ (1), $b = 16{\cdot}026$ (2), $c = 11{\cdot}522$ (1) Å, $V = 1147{\cdot}6$ Å^3, $Z = 4$, $D_m = 2{\cdot}06$, $D_x = 2{\cdot}10$ Mg m^{-3}, λ(Mo $K\bar{\alpha}$) = 0·71073 Å, $\mu = 3{\cdot}32$ mm^{-1}, $F(000) = 688$, room temperature, $R = 0{\cdot}048$ for 1640 unique observed reflections. (II) Triethylammonium (acetylacetonato)tetrachlorostannate(IV), [$NH(C_2H_5)_3$][$Sn(C_5H_7O_2)Cl_4$], $M_r = 461{\cdot}8$, orthorhombic, *Pnma*, $a = 11{\cdot}199$ (1), $b = 12{\cdot}235$ (1), $c = 13{\cdot}820$ (1) Å, $V = 1893{\cdot}6$ Å^3, $Z = 4$, $D_m = 1{\cdot}62$, $D_x = 1{\cdot}62$ Mg m^{-3}, λ(Mo $K\bar{\alpha}$) = 0·71073 Å, $\mu = 1{\cdot}92$ mm^{-1}, $F(000) = 920$, room temperature, $R = 0{\cdot}050$ for 2625 unique observed reflections. Discrete chelate-complex molecules Sb(acac)Cl_4 of C_2 (approximate C_{2v}) point symmetry show distorted octahedral coordination at the central atom. Chelate-complex anions [Sn(acac)Cl_4]$^-$ of C_s (approximate C_{2v}) point symmetry with distorted octahedral coordination at the central atom and twofold (over the mirror plane) disordered cations $NHEt_3^+$ are linked *via* a weak hydrogen bond N—H···Cl; in the anion the Sn—Cl bond of the hydrogen acceptor atom is significantly lengthened.

Experimental. Single crystals were prepared by Teutsch & Schmidtke (1986). D_m by flotation. Syntex $P2_1$ diffractometer with graphite monochromator. Lattice parameters from setting angles of 15 strong reflections. Intensities by variable ω-scan technique, three standard reflections every 100 reflections showed only small random variations. No corrections for absorption. Criterion for observed reflections $I > 1{\cdot}96\sigma(I)$. (I) Crystal 0·5 × 0·5 × 0·3 mm. Lattice parameters from reflections with $38 < 2\theta < 40°$. 2009 unique reflections measured with $(\sin\theta)/\lambda$ up to 0·904 Å^{-1} and $0 \le h \le 11$, $0 \le k \le 28$, $0 \le l \le 20$; ten reflections with too large intensity and six with distinct asymmetric background excluded from structure refinement. (II) Crystal 0·3 × 0·3 × 0·2 mm. Lattice parameters from reflections with $32 < 2\theta < 35°$. 3559 unique reflections measured with $(\sin\theta)/\lambda$ up to 0·756 Å^{-1} and $0 \le h \le 16$, $0 \le k \le 18$, $0 \le l \le 20$; ten reflections with too large intensity, 17 with distinct asymmetric background, and 16 with $|F_o| \gg |F_c|$ at an advanced state of refinement and asymmetric profile discarded.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *of the non*-H *atoms with e.s.d.'s in parentheses*

	x	y	z	U_{eq}*
(I)				
Sb	0·0	0·12375 (2)	0·25	0·0413 (1)
Cl(1)	0·2160 (4)	0·2205 (1)	0·3439 (2)	0·0658 (5)
Cl(2)	0·2300 (4)	0·1147 (2)	0·4102 (3)	0·0897 (9)
O	0·1788 (8)	0·0335 (3)	0·3249 (5)	0·066 (1)
C(1)	0·3022 (20)	−0·0975 (5)	0·3834 (6)	0·083 (3)
C(2)	0·1576 (11)	−0·0455 (3)	0·3159 (4)	0·050 (1)
C(3)	0·0	−0·0849 (4)	0·25	0·057 (2)
(II), anion				
Sn	0·21565 (4)	0·25	0·04573 (3)	0·0492 (2)
Cl(1)	0·2955 (1)	0·1048 (2)	0·1390 (1)	0·0838 (6)
Cl(2)	0·0308 (2)	0·25	0·1336 (2)	0·0652 (6)
Cl(3)	0·3875 (2)	0·25	−0·0634 (2)	0·0773 (8)
O	0·1407 (4)	0·1317 (3)	−0·0453 (2)	0·065 (1)
C(1)	0·0216 (7)	0·0489 (6)	−0·1624 (5)	0·091 (3)
C(2)	0·0696 (4)	0·1501 (5)	−0·1163 (3)	0·057 (2)
C(3)	0·0334 (7)	0·25	−0·1490 (5)	0·062 (2)
(II), cation				
N	0·6684 (8)	0·25	0·0346 (5)	0·075 (3)
C(41)†	0·7088 (16)	0·3644 (19)	0·0495 (12)	0·104 (8)
C(42)†	0·6791 (13)	0·1823 (11)	0·1238 (9)	0·074 (4)
C(5)	0·6300 (8)	0·0697 (8)	0·1140 (7)	0·107 (3)
C(6)†	0·7392 (16)	0·1978 (15)	−0·0455 (9)	0·089 (5)
C(7)	0·7060 (11)	0·25	−0·1410 (8)	0·147 (8)

* $U_{eq} = \frac{1}{3}(U_{11}a^{*2}a^2 + U_{12}a^*b^*ab\cos\gamma + \ldots)$.
† Occupancy factor 0·5, owing to disorder.

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Table 2. *Selected interatomic distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

(I)			
Sb–Cl(1)	2·318 (2)	Sb–Cl(2)	2·339 (3)
Sb–O	2·017 (5)	O–C(2)	1·278 (7)
C(1)–C(2)	1·451 (11)	C(2)–C(3)	1·391 (7)
Cl(1)–Sb–Cl(1$^{\rm i}$)	96·1 (1)	Cl(1)–Sb–Cl(2)	91·5 (1)
Cl(1)–Sb–Cl(2$^{\rm i}$)	93·2 (1)	Cl(1)–Sb–O	87·7 (1)
Cl(1)–Sb–O$^{\rm i}$	176·1 (1)	Cl(2)–Sb–Cl(2$^{\rm i}$)	172·9 (1)
Cl(2)–Sb–O	87·4 (2)	Cl(2)–Sb–O$^{\rm i}$	87·5 (2)
O–Sb–O$^{\rm i}$	88·5 (3)	Sb–O–C(2)	128·2 (4)
O–C(2)–C(1)	117·5 (6)	O–C(2)–C(3)	124·5 (5)
C(1)–C(2)–C(3)	117·9 (5)	C(2)–C(3)–C(2$^{\rm i}$)	126·1 (6)

Symmetry code: (i) $-x, y, \frac{1}{2}-z$.

(II), anion			
Sn–Cl(1)	2·370 (2)	Sb–Cl(2)	2·400 (2)
Sn–Cl(3)	2·445 (2)	Sn–O	2·094 (4)
O–C2	1·283 (6)	C(1)–C(2)	1·493 (9)
C(2)–C(3)	1·365 (6)		
Cl(1)–Sn–Cl(1$^{\rm i}$)	97·1 (1)	Cl(1)–Sn–Cl(2)	92·9 (1)
Cl(1)–Sn–Cl(3)	92·2 (1)	Cl(1)–Sn–O	87·7 (1)
Cl(1)–Sn–O$^{\rm i}$	175·1 (1)	Cl(2)–Sn–Cl(3)	172·3 (1)
Cl(2)–Sn–O	87·6 (1)	Cl(3)–Sn–O	86·8 (1)
O–Sn–O$^{\rm i}$	87·5 (2)	Sn–O–C(2)	125·9 (4)
O–C(2)–C(1)	113·8 (5)	O–C(2)–C(3)	126·5 (5)
C(1)–C(2)–C(3)	119·6 (5)	C(2)–C(3)–C(2$^{\rm i}$)	127·1 (7)
(II), cation			
N–C(41)	1·49 (2)	N–C(42)	1·49 (1)
N–C(6)	1·50 (2)	C(41)–C(5$^{\rm i}$)	1·49 (2)
C(42)–C(5)	1·49 (2)	C(6)–C(7)	1·51 (2)
C(41)–N–C(42)	113 (1)	C(41)–N–C(6)	110 (1)
C(42)–N–C(6)	109 (1)	N–C(41)–C(5$^{\rm i}$)	114 (1)
N–C(42)–C(5)	114 (1)	N–C(6)–C(7)	110 (1)
(II), hydrogen bond*			
N···Cl(3)	3·425 (9)	N–H8	0·98
H8···Cl(3)	2·46	N–H8···Cl(3)	168

Symmetry code: (i) $x, \frac{1}{2}-y, z$.

* Coordinates of H(8) not refined.

Heavy-atom method, least-squares refinement based on F magnitudes, observed reflections only, weighted according to $w=[\sigma^2(F)+c|F_o|^2]^{-1}$. H atoms located by difference-Fourier method or geometrical calculation. Complex atomic scattering factors from *International Tables for X-ray Crystallography* (1974). (I) In final refinement 56 parameters varied: one scale factor, coordinates and anisotropic thermal parameters of all non-H atoms (H atoms constrained at C atoms with C–H equal to 0·96 Å and fixed isotropic thermal parameters); $c = 0{\cdot}0009$, $wR = 0{\cdot}064$, $S = 1{\cdot}618$; all Δ/σ in last cycle 0·00; $\Delta\rho_{\rm min}$ and $\Delta\rho_{\rm max}$ in final difference Fourier synthesis −2·2 and +2·0 e Å^{-3} near Sb atom, respectively. (II) In final refinement 109 parameters varied: one scale factor, coordinates and anisotropic thermal parameters of all non-H atoms (H atoms: in the anion constrained at C atoms with C–H equal to 0·96 Å, in the cation with coordinates invariant; all isotropic thermal parameters fixed); $c = 0{\cdot}00075$, $wR = 0{\cdot}087$, $S = 2{\cdot}270$; all Δ/σ in last cycle 0·00; $\Delta\rho_{\rm min}$ and $\Delta\rho_{\rm max}$ in final difference Fourier synthesis −1·4 and +0·8 e Å^{-3} near Sn atom, respectively.

The atomic parameters of all non-H atoms are listed in Table 1,* selected interatomic distances and angles in Table 2. Fig. 1 shows the molecules with atom labels, Fig. 2 stereo plots of the crystal structures. All calculations and drawings performed with the Nicolet (1983) *SHELXTL* program system on a Data General Eclipse S/140 computer.

For (I) the absolute configuration as given was suggested by the refinement; with the approximate molecular symmetry C_{2v} the inverted (enantiomorphic) structure yielded the only slightly higher values $R = 0{\cdot}050$, $wR = 0{\cdot}065$, $S = 1{\cdot}665$. For (II) refinement in the non-centrosymmetric subgroup $Pna2_1$ with the cation ordered was also performed, but found not convincing.

Related literature. Polarized crystal spectra of the complexes in the visible and ultraviolet region (Teutsch

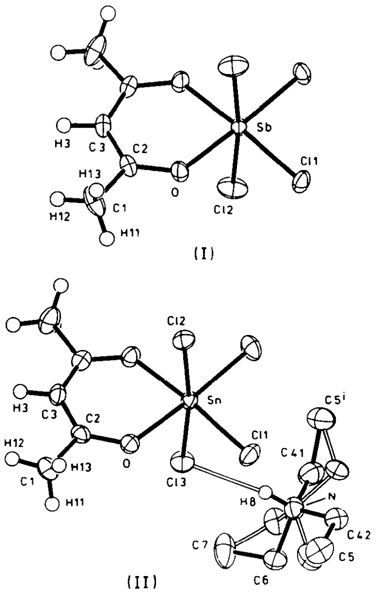

Fig. 1. (I) The molecule Sb(acac)Cl$_4$ [C_2 axis through atoms Sb, C(3), H(3)]. (II) The hydrogen-bonded anion [Sn(acac)Cl$_4$]$^-$ and cation NHEt$_3^+$, of which the two possible orientations are drawn with different kinds of bonds [C_s plane through atoms Sb, Cl(2), Cl(3), C(3), H(3); N, C(7), H(8)]. All ellipsoids correspond to 20% probability level, H atoms with arbitrary size (their half-occupied positions in the cation are omitted for clarity).

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* Lists of anisotropic thermal parameters, coordinates and isotropic thermal parameters of the H atoms, further interatomic distances and angles including H atoms, and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44768 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

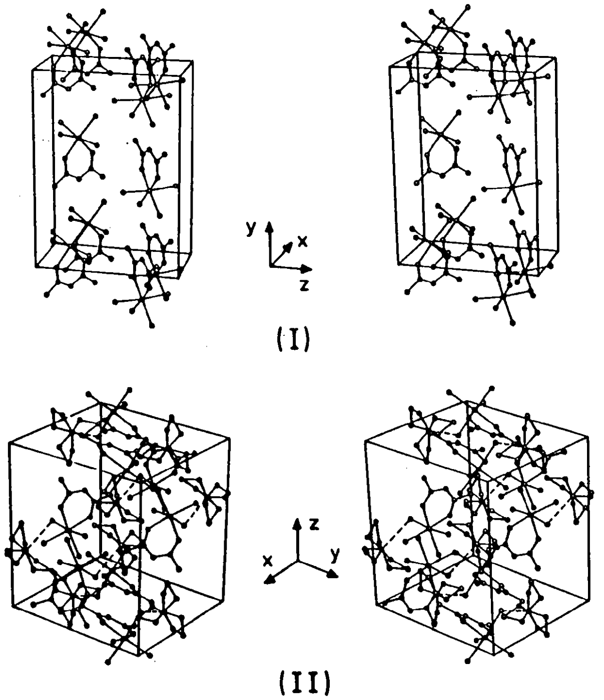

Fig. 2. Stereo plots of the crystal structures. Atoms with arbitrary size (H atoms omitted), hydrogen bonds with dotted lines.

& Schmidtke, 1986). Crystal structure analyses of related compounds: Sb(acac)$MeCl_3$, Sb(acac)Me_2Br_2, Sb(acac)Ph_2Cl_2 (Kanehisa, Onuma, Uda, Hirabayashi, Kai, Yasuoka & Kasai, 1978); $Sn(acac)_2Me_2$, $Sn(acac)_2Cl_2$ (Miller & Schlemper, 1973, 1978).

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(η-Cyclopentadienyl)[methoxy(methyl)carbene]bis(triphenylphosphine)ruthenium(II) Hexafluorophosphate

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Abstract. $C_{44}H_{41}F_6OP_3Ru$, $M_r = 893{\cdot}8$, orthorhombic, $P2_12_12_1$, $a = 11{\cdot}934\,(4)$, $b = 14{\cdot}858\,(4)$, $c = 22{\cdot}287\,(4)$ Å, $V = 3952\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}502$ Mg m^{-3}, Mo $K\bar{\alpha}$ radiation, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}531$ mm^{-1}, $F(000) = 1824$, $T = 293\,(2)$ K, $R = 0{\cdot}046$ for 3219 observed reflections. The crystal structure of the title compound is comprised of discrete $[Ru\{C(OCH_3)CH_3\}\{P(C_6H_5)_3\}_2(\eta\text{-}C_5H_5)]^+$ cations and $[PF_6]^-$ anions. In the cation the Ru atom exists in a distorted octahedral environment with one face being occupied by the η-C_5H_5 ring and the other by the two P atoms [Ru—P 2·333 (2), 2·336 (2) Å; P—Ru—P 101·3 (1)°] and C(6), at 1·931 (9) Å, of the $C(OCH_3)CH_3$ group [P—Ru—C 86·6 (2), 89·8 (2)°].

Experimental. The complex was prepared in 62% yield from $[Ru(NCCH_3)\{P(C_6H_5)_3\}_2(\eta\text{-}C_5H_5)][PF_6]$ and $HC_2Si(CH_3)_3$ in CH_3OH; crystals were obtained from $CH_3OH/CDCl_3$. Found: C, 58·9; H, 4·6; $C_{44}H_{41}F_6OP_3Ru$ requires C, 58·9; H, 4·6%. IR (Nujol): ν(CO) 1270*s*, ν(PF) 840*vs*(*br*). ^{1}H NMR: $\delta(CDCl_3$; p.p.m.) 3·02*s* (CH_3), 3·28*s* (OCH_3), 4·75*s* (C_5H_5), 7·0–7·4*m* (C_6H_5). ^{13}C NMR: $\delta(CDCl_3$; p.p.m.) 46·4*s* (CH_3), 60·7*s* (OCH_3), 91·4*s* (C_5H_5), 128·2–136·1*m* (C_6H_5), 309·2*t*, *J*(CP) 0·2Hz (Ru*C*). FAB MS: M$^+$ at *m*/*z* 749. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\bar{\alpha}$ radiation; ω:2θ scan technique. Cell parameters on crystal 0·50 × 0·18 × 0·50 mm from least-squares

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