C(12)	0.293 (2)	0.234 (2)	0.535 (1)	8.9 (6)
C(14)	0.376 (2)	0.109 (2)	0.447 (1)	9.8 (6)
C(15)	0.371 (2)	−0.024 (2)	0.419 (1)	7.8 (6)
C(17)	0.273 (2)	−0.087 (2)	0.302 (1)	7.5 (5)
C(18)	0.164 (1)	−0.110 (2)	0.265 (1)	6.8 (5)
C(20)	0.618 (2)	0.178 (2)	0.314 (1)	7.6 (5)

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

U environment			
U—O(1)	1.74 (1)	U—O(2)	1.76 (1)
U—N(1)	2.59 (2)	U—N(4)	2.61 (2)
U—N(7)	2.70 (2)	U—N(10)	2.76 (2)
U—N(13)	2.63 (2)	U—N(16)	2.68 (2)
O(1)—U—O(2)	174.9 (5)	O(1)—U—N(1)	86.2 (5)
N(1)—U—N(4)	64.7 (5)	N(1)—U—N(16)	65.0 (5)
N(4)—U—N(7)	63.4 (6)	N(7)—U—N(10)	61.7 (6)
N(10)—U—N(13)	57.7 (6)	N(13)—U—N(16)	64.3 (6)
Triflate			
S(1)—O(11)	1.48 (1)	S(2)—O(21)	1.44 (1)
S(1)—O(12)	1.44 (1)	S(2)—O(22)	1.44 (1)
S(1)—O(13)	1.42 (2)	S(2)—O(23)	1.44 (1)
S(1)—C(10)	1.74 (3)	S(2)—C(20)	1.77 (2)
C(10)—F(11)	1.42 (3)	C(20)—F(21)	1.30 (3)
C(10)—F(12)	1.33 (3)	C(20)—F(22)	1.33 (2)
C(10)—F(13)	1.27 (3)	C(20)—F(23)	1.34 (3)
O(11)—S(1)—O(12)	115.6 (8)	O(21)—S(2)—O(22)	114.8 (9)
O(11)—S(1)—O(13)	116.1 (9)	O(21)—S(2)—O(23)	115 (1)
O(11)—S(1)—C(10)	100 (1)	O(21)—S(2)—C(20)	102 (1)
O(12)—S(1)—O(13)	115 (1)	O(22)—S(2)—O(23)	114 (1)
O(12)—S(1)—C(10)	102 (1)	O(22)—S(2)—C(20)	107 (1)
O(13)—S(1)—C(10)	105 (1)	O(23)—S(2)—C(20)	101 (1)
S(1)—C(10)—F(11)	106 (2)	S(2)—C(20)—F(21)	113 (2)
S(1)—C(10)—F(12)	111 (2)	S(2)—C(20)—F(22)	113 (2)
S(1)—C(10)—F(13)	116 (2)	S(2)—C(20)—F(23)	111 (2)
F(11)—C(10)—F(12)	105 (2)	F(21)—C(20)—F(22)	109 (2)
F(11)—C(10)—F(13)	108 (2)	F(21)—C(20)—F(23)	106 (2)
F(12)—C(10)—F(13)	109 (2)	F(22)—C(20)—F(23)	105 (2)
Hexaaza-18-crown-6			
N(1)—C(2)	1.43 (2)	N(1)—C(18)	1.56 (2)
N(4)—C(3)	1.56 (2)	N(4)—C(5)	1.43 (3)
N(7)—C(6)	1.52 (3)	N(7)—C(8)	1.43 (3)
N(10)—C(9)	1.41 (3)	N(10)—C(11)	1.42 (3)
N(13)—C(12)	1.45 (3)	N(13)—C(14)	1.49 (3)
N(16)—C(15)	1.53 (3)	N(16)—C(17)	1.43 (2)
C(2)—C(3)	1.47 (3)	C(5)—C(6)	1.48 (3)
C(8)—C(9)	1.42 (3)	C(11)—C(12)	1.43 (3)
C(14)—C(15)	1.62 (3)	C(17)—C(18)	1.54 (3)
C(2)—N(1)—C(18)	109 (2)	C(3)—N(4)—C(5)	108 (2)
C(6)—N(7)—C(8)	116 (2)	C(9)—N(10)—C(11)	114 (2)
C(12)—N(13)—C(14)	113 (2)	C(15)—N(16)—C(17)	108 (2)
N(1)—C(2)—C(3)	106 (2)	C(2)—C(3)—N(4)	109 (2)
N(4)—C(5)—C(6)	104 (2)	C(5)—C(6)—N(7)	111 (2)
N(7)—C(8)—C(9)	110 (2)	C(8)—C(9)—N(10)	112 (2)
N(10)—C(11)—C(12)	115 (2)	C(11)—C(12)—N(13)	106 (2)
N(13)—C(14)—C(15)	105 (2)	C(14)—C(15)—N(16)	109 (2)
N(16)—C(17)—C(18)	106 (2)	C(17)—C(18)—N(1)	108 (2)

The complex was prepared as described in a previous paper (Folcher, Charpin, Costes, Keller & de Villardi, 1979). An excess of UO_3 was added to bring the pH within the range 4–5 and undissolved oxide was removed by filtration. The water was evaporated under vacuum and the resulting solid was redissolved in acetonitrile; crystals suitable for X-ray analysis were obtained by slow cooling.

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Molecular Adduct of Dimethyltin Dichloride with Methyl *N*-(2-Hydroxybenzylidene)-alaninate (1:2), $[SnCl_2(C_{11}H_{13}NO_3)_2(CH_3)_2]$

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Abstract

The structure of the 2:1 complex of methyl *N*-(2-hydroxybenzylidene)alaninate with dimethyltin dichloride, dichlorobis(2-{[1-(methoxycarbonyl)-ethyl]iminiomethyl}phenolato-*O*)dimethyltin, has been determined. The coordination of the Sn atom is octahedral, all *trans*, with bond distances Sn—Cl = 2.599 (1), Sn—O = 2.244 (3) and Sn—C = 2.111 (4) Å. The ligands are coordinated to Sn

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71447 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU1044]

through the phenolic O atoms, the hydroxylic H atoms being shifted to the imine N atoms giving rise to a zwitterion. There is intramolecular hydrogen bonding with N—H···O = 2.628 (4) Å.

Comment

N-Salicylidene-*α*-amino acid ester has been reported to form a trigonal bipyramidal adduct with triphenyltin(IV) chloride by Lee, Gabe, Khoo, Eng & Smith (1990). In addition, a shift of the phenolic proton to the imine N atom resulting in a zwitterionic configuration for the ligand has been observed. This is further substantiated by Bullock, Ladd, Povey & Tajmir-Riahi (1979) from the crystal structure and spectroscopic study of Schiff base complexes with calcium nitrate. In view of this unusual occurrence of the zwitterionic form of Schiff bases and its influence on the mode of coordination of the potentially bidentate neutral ligand, *N*-salicylidene-*α*-amino acid ester, to diorganotin dichloride, a complete X-ray structural investigation of the title adduct has been undertaken.

Preparation was according to Lee *et al.* (1990). Dimethyltin dichloride was used instead of triphenyltin chloride in a 1:2 chloroform–petroleum ether (60–80) mixture. Recrystallization from a similar mixture at room temperature gave soft crystals of melting point 388–390 K. Elemental analysis (NUS, Singapore) is as follows: calculated (found) for $C_{24}H_{32}N_2O_6SnCl_2$: C 45.45 (45.26), H 5.10 (5.13)%.

The Sn atom, which is on a symmetry centre, is bonded to two Cl atoms, two methyl groups and the phenolic O atoms of the two ligands, giving an all-*trans* octahedral geometry. The coordination bond angles involving the equivalent *trans* ligands are 180° by symmetry, the other angles range from 86.1 (1) to 93.9 (1)°. The Sn—C and Sn—Cl distances are normal and lie within the range for similar compounds (Lee *et al.*, 1990; Kamwaya & Khoo, 1985). The Sn—O bond length is close to those found in the Me_2SnCl_2–salicylideneaniline adduct [2.22 Å (Randaccio, 1973)]. The phenolic H atom has moved to the imine N atom forming an intramolecular hydrogen bond between O2 and N10 with N—H···O = 2.628 (4), N—H = 0.81 (4), O···H = 1.98 (4) Å and N—H···O = 137 (4)°.

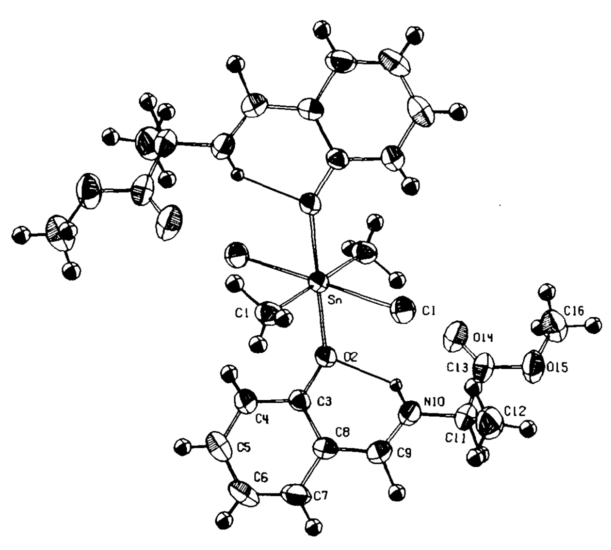

Fig. 1. A drawing of the title adduct with the atom-numbering scheme.

The organic ligand is monodentate bonding only *via* the phenolic oxygen. Atom O14 is 4.535 (3) Å from Sn and the very short C13—O14 distance of 1.187 (5) Å is typical for a non-bridging carbonyl group. A similar distance of 1.179 Å was reported for the corresponding carbonyl group in Ph_3SnCl–$2HOC_6H_4CH{=}NCH_2CO_2Et$ (Lee *et al.*, 1990).

Experimental

Crystal data

$[SnCl_2(C_{11}H_{13}NO_3)_2(CH_3)_2]$
M_r = 634.15
Monoclinic
$P2_1/n$
a = 12.092 (2) Å
b = 9.393 (2) Å
c = 12.507 (2) Å
β = 105.423 (9)°
V = 1369.4 (4) $Å^3$
Z = 2
D_x = 1.538 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 120 reflections
θ = 10.1-14.0°
μ = 1.17 mm^{-1}
T = 293 K
$\{10\bar{1}\}\{101\}(010)(1\bar{1}0)(\bar{1}\bar{1}0)$
0.74 × 0.46 × 0.11 mm
Yellow

Data collection

Huber four-circle diffractometer
ω-2θ step scans (50 steps, 1 s $step^{-1}$)
Absorption correction:
by integration from crystal shape
T_{min} = 0.584, T_{max} = 0.873
7858 measured reflections
3969 independent reflections

2824 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.058
θ_{max} = 30°
$h = -16 \rightarrow 16$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 17$
2 standard reflections monitored every 50 reflections
intensity variation: 10%

Refinement

Refinement on F
R = 0.042
wR = 0.052
S = 1.517
2824 reflections
224 parameters
Only coordinates of H atoms refined

$w = \{[\sigma_{cs}(F^2) + 1.03F^2]^{1/2} - |F|\}^{-1}$
$(\Delta/\sigma)_{max}$ = 0.13
$\Delta\rho_{max}$ = 1.6 (1) e $Å^{-3}$
$\Delta\rho_{min}$ = −2.2 (1) e $Å^{-3}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å²)

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

	x	*y*	*z*	U_{eq}
Sn	0.0	0.0	0.5	0.0316 (2)
Cl	0.05483 (10)	0.21486 (12)	0.39358 (9)	0.0502 (6)
C1	−0.1741 (4)	0.0615 (6)	0.4496 (4)	0.046 (2)
O2	0.0411 (2)	0.1427 (3)	0.6493 (2)	0.040 (1)
C3	−0.0227 (3)	0.2079 (4)	0.7048 (3)	0.033 (2)
C4	−0.1137 (4)	0.1393 (5)	0.7339 (4)	0.045 (2)
C5	−0.1777 (4)	0.2070 (6)	0.7936 (4)	0.054 (3)
C6	−0.1544 (5)	0.3464 (6)	0.8288 (4)	0.061 (3)
C7	−0.0666 (4)	0.4169 (5)	0.8033 (4)	0.055 (3)
C8	0.0003 (3)	0.3506 (4)	0.7398 (3)	0.038 (2)
C9	0.0897 (4)	0.4309 (5)	0.7141 (3)	0.043 (2)
N10	0.1549 (3)	0.3824 (4)	0.6564 (3)	0.043 (2)
C11	0.2456 (4)	0.4644 (5)	0.6277 (4)	0.051 (3)
C12	0.1998 (6)	0.5460 (7)	0.5213 (6)	0.068 (4)
C13	0.3389 (3)	0.3565 (5)	0.6216 (3)	0.045 (2)
O14	0.3359 (3)	0.2328 (3)	0.6401 (3)	0.059 (2)
O15	0.4243 (3)	0.4211 (3)	0.5929 (3)	0.058 (2)
C16	0.5215 (5)	0.3341 (7)	0.5895 (5)	0.065 (3)

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

Sn—Cl	2.599 (1)	C7—C8	1.418 (5)
Sn—O2	2.244 (3)	C8—C9	1.423 (6)
Sn—C1	2.111 (4)	C9—N10	1.286 (5)
O2—C3	1.318 (4)	N10—C11	1.461 (6)
C3—C4	1.403 (6)	C11—C12	1.508 (8)
C3—C8	1.415 (5)	C11—C13	1.533 (7)
C4—C5	1.367 (6)	C13—O14	1.187 (5)
C5—C6	1.386 (7)	C13—O15	1.327 (5)
C6—C7	1.359 (7)	O15—C16	1.441 (6)
O2—N10	2.628 (4)	O2···H10	1.98 (4)
N10—H10	0.81 (4)		
Cl—Sn—C1	90.2 (2)	C3—C8—C9	122.1 (3)
Cl—Sn—O2	86.1 (1)	C7—C8—C9	118.3 (4)
C1—Sn—O2	93.9 (1)	C8—C9—N10	123.7 (4)
Sn—O2—C3	133.2 (2)	C9—N10—C11	124.3 (4)
O2—C3—C4	122.1 (3)	N10—C11—C12	111.1 (4)
O2—C3—C8	120.6 (3)	N10—C11—C13	105.9 (3)
C4—C3—C8	117.2 (4)	C12—C11—C13	113.2 (4)
C3—C4—C5	121.7 (4)	C11—C13—O14	125.2 (4)
C4—C5—C6	121.1 (5)	C11—C13—O15	110.3 (4)
C5—C6—C7	119.4 (5)	O14—C13—O15	124.4 (4)
C6—C7—C8	121.0 (5)	C13—O15—C16	116.7 (4)
C3—C8—C7	119.6 (4)	O2···H10—N10	137 (4)

Cell dimensions were determined from reflections measured at four positions, ±2θ and high and low χ. Data were corrected for background, Lorentz and polarization effects, and for absorption. The structure was determined using *SHELXS86* (Sheldrick, 1986) and H atoms were located from a difference electron density map. Non-H atoms were refined anisotropically and H atoms isotropically using a modification of *ORFLS* (Busing, Martin & Levy, 1962).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and bond distances and angles including H-atom geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71378 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1077]

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Structure of Bis(2,2′-bipyridyl)diisocyanatonickel(II)

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Abstract

Blue–grey crystals of $[Ni(bipy)_2(NCO)_2]$ (where bipy is 2,2′-bipyridine) were prepared and its structure determined. There are discrete molecules with symmetry 2. The Ni^{II} ion is octahedrally coordinated by six N atoms, with some slight distortion towards a trigonal prism.

Comment

Recently, our group has studied complexes of copper(II) and nickel(II) ions with tridentate ligands, $[M(L_{III})(X)_2]$ (L_{III} = tridentate ligand; X = halide or pseudohalide), as precursors to polynuclear species (Cortés, Arriortua, Rojo, Solans & Beltrán, 1986; Cortés *et al.*, 1988; Arriortua *et al.*, 1988; Via, Arriortua, Rojo, Mesa & García, 1989). Knowledge