crystals were obtained by the slow evaporation of a solution of the complex in methanol/0.05 *M* $NaClO_4$.

Crystal data

$[Co(C_6H_9O_2)_2(C_6H_{14}Cl_2N_2)]ClO_4$
$M_r = 569.74$
Monoclinic
$P2_1/c$
$a = 17.229\,(3)$ Å
$b = 10.817\,(5)$ Å
$c = 14.851\,(6)$ Å
$\beta = 110.78\,(3)°$
$V = 2587.7\,(16)$ Å^3
$Z = 4$
$D_x = 1.462$ Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 9.5–11.8°$
$\mu = 1.016$ mm^{-1}
$T = 293\,(2)$ K
Thin plate
0.32 × 0.18 × 0.15 mm
Green

Data collection

Enraf–Nonius CAD-4 diffractometer
$2\theta/\omega$ scans
Absorption correction: empirical *via* ψ scans (North, Phillips & Mathews, 1968)
$T_{min} = 0.722$, $T_{max} = 0.859$
4307 measured reflections
4049 independent reflections
2735 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 23.98°$
$h = 0 \rightarrow 19$
$k = 0 \rightarrow 12$
$l = -16 \rightarrow 15$
3 standard reflections frequency: 60 min maximum variation: ±3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.160$
$S = 1.142$
4049 reflections
299 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0572P)^2 + 6.1844P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.033$
$\Delta\rho_{max} = 0.922$ e Å^{-3}
$\Delta\rho_{min} = -0.635$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Disorder was apparent in the position of Cl2. It was therefore refined as two part-atoms, with occupancies summing to 1.0.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: local program. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL* (Sheldrick, 1995). Software used to prepare material for publication: *SHELXL*93.

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Bis[aqua(chlorodifluoroacetato-*O*)triphenyltin–1,10-phenanthroline]

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Abstract

The coordinated water molecule in the centrosymmetric title compound $[Sn(C_2ClF_2O_2)(C_6H_5)_3(H_2O)]_2.2C_{12}H_8N_2$, engages in hydrogen bonding with two heterocycles. The Sn atom shows *trans*-C_3SnO_2 trigonal bipyramidal coordination.

Comment

An earlier study (Ng, Kumar Das & Kennard, 1996) documented the 'outer-sphere coordination' of 1,10-phenanthroline in dimeric bis[aqua(trifluoroacetato)triphenyltin–1,10-phenanthroline]. Despite the difference in electron-withdrawing ability of the F and Cl atoms, the bis[aqua(chlorodifluoroacetato)triphenyltin–1,10-phenanthroline] analogue, (I), is isomorphous with bis[aqua(trifluoroacetato)triphenyltin–1,10-phenanthroline], both compounds crystallizing with matching unit-cell dimensions. The latter compound displays a somewhat longer $Sn\cdots H_2O$ interaction [2.335 (4) Å] but its two hydrogen bonds [2.809 (6) and 2.814 (6) Å] are of the same length as those [2.799 (6) and 2.846 (6) Å] of the

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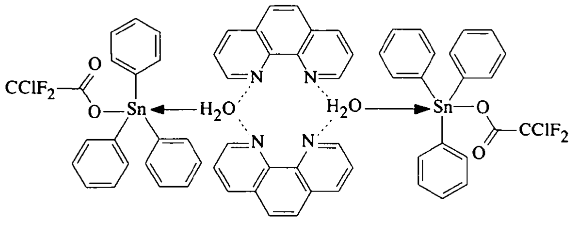
Supplementary data for this paper are available from the IUCr electronic archives (Reference: BS1025). Services for accessing these data are described at the back of the journal.

title compound. These hydrogen bonds are shorter than those found in bis[aquachlorotri(*p*-chlorophenyl)tin–1,10-phenanthroline] (Ng & Kumar Das, 1995), in agreement with the greater electron-withdrawing ability of the trifluoroacetato group compared with the chloro group.



(I)

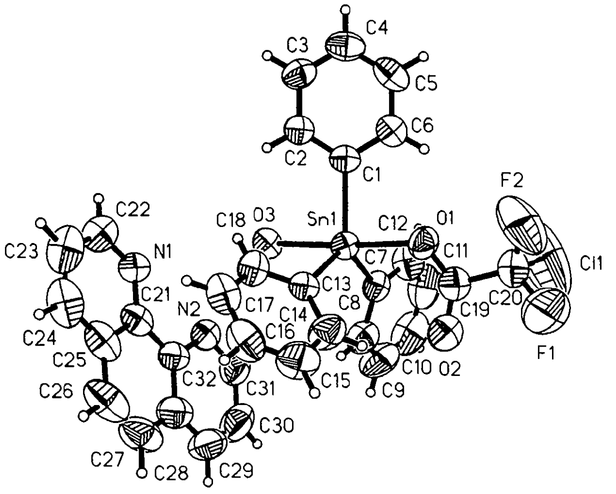


Fig. 1. *ZORTEP* (Zsolnai & Pritzkow, 1996) plot of the asymmetric unit of bis[aqua(chlorodifluoroacetato)triphenyltin–1,10-phenanthroline] with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

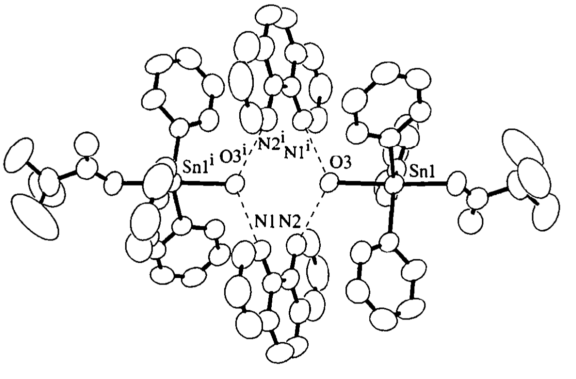


Fig. 2. *ZORTEP* (Zsolnai & Pritzkow, 1996) plot of bis[aqua(chlorodifluoroacetato)triphenyltin-1,10-phenanthroline] showing the hydrogen bonds [O3···N1^i 2.799 (6), O3···N2 2.846 (6) Å and N1^i···O3···N2 109.5 (2)°; symmetry code: (i) $-x$, $1-y$, $1-z$].

Experimental

Equimolar quantities of triphenyltin hydroxide, chlorodifluoroacetic acid and 1,10-phenanthroline were heated in a small volume of ethanol. The title complex separated on slow cooling of the filtered solution.

Crystal data

$[Sn(C_2ClF_2O_2)(C_6H_5)_3(H_2O)]_2.2C_{12}H_8N_2$
$M_r = 1355.36$
Triclinic
$P\bar{1}$
$a = 9.515\,(1)$ Å
$b = 12.234\,(1)$ Å
$c = 12.787\,(1)$ Å
$\alpha = 89.888\,(8)°$
$\beta = 97.377\,(9)°$
$\gamma = 90.290\,(9)°$
$V = 1476.3\,(3)$ Å^3
$Z = 1$
$D_x = 1.525$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 12.0–13.0°$
$\mu = 1.004$ mm^{-1}
$T = 298\,(2)$ K
Block
0.50 × 0.45 × 0.30 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scan
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{min} = 0.592$, $T_{max} = 0.740$
5533 measured reflections
5190 independent reflections

4446 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 25.39°$
$h = 0 \rightarrow 11$
$k = -14 \rightarrow 14$
$l = -15 \rightarrow 15$
3 standard reflections frequency: 60 min intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.145$
$S = 1.087$
5190 reflections
378 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.1033P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.176$ e Å^{-3}
$\Delta\rho_{min} = -1.802$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Sn1—C1	2.175 (5)	Sn1—O1	2.186 (3)
Sn1—C7	2.123 (5)	Sn1—O3	2.299 (3)
Sn1—C13	2.130 (5)		
C1—Sn1—C7	117.8 (2)	C7—Sn1—O1	93.0 (2)
C1—Sn1—C13	119.8 (2)	C7—Sn1—O3	88.6 (2)
C1—Sn1—O1	88.8 (2)	C13—Sn1—O1	94.0 (2)
C1—Sn1—O3	88.1 (2)	C13—Sn1—O3	87.4 (2)
C7—Sn1—C13	122.0 (2)	O1—Sn1—O3	176.9 (1)

The final difference map had a peak larger than 1 e Å^{-3} near the Sn atom. Water H atoms were located and refined. All other H atoms were placed in calculated positions with $U(H) = 1.5U_{eq}(C)$.

Data collection: *CAD*-4 *VAX*/*PC* (Enraf–Nonius, 1988). Cell refinement: *CAD*-4 *VAX*/*PC*. Data reduction: *NRCVAX*

(Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai & Pritzkow, 1996). Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: KH1135). Services for accessing these data are described at the back of the journal.

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Pentakis(tetrahydrofuran)barium Bis(carbazolate) Tetrahydrofuran Solvate

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Abstract

Reaction of carbazole with barium metal in dry tetrahydrofuran yields colourless blocks of the title compound, $[Ba(C_4H_8O)_5](C_{12}H_8N)_2.C_4H_8O$. The structure contains sevenfold-coordinated barium counter cations, which are surrounded by two carbazole monoanions and five tetrahydrofuran molecules in a C_2 symmetric environment.

Comment

Numerous structures of alkaline and alkaline earth organometallic compounds have contributed to the rationalization of their chemical and physical properties. The effects of metal cation solvation on the structure of the complexes are well known, especially for the respective sodium complexes (Bock, Näther, Havlas, John & Arad, 1994; Bock *et al.*, 1992; Schade & Schleyer, 1987), whereas analogous investigations for the heavier alkaline and the doubly charged alkaline earth ions are still missing. The structure of the title compound was determined as part of a study of both solvent and counter cation effects in carbazolate anion salts. In the presence of strong solvating agents like tetraglyme, crown ethers or cryptands, alkali metal ions form monomers (Bock, Arad, Näther & Havlas, 1997; Bock, Arad & Näther, 1997), contrary to the otherwise crystallizing dimers. According to their structures, in the dimers, the nitrogen centre of one anion is σ-coordinated to the alkali metal ion, whereas the coordination of the second anion depends predominantly on the cation size. Small cations such as lithium are σ-coordinated, medium-sized ones such as sodium exhibit mixed π–σ contacts, and the rather large caesium cation is π-coordinated to the other carbazole anion (Bock, Arad, Näther & Havlas, 1997; Gregory, Bremer, Schleyer, Klusener & Brandsma, 1989; Hacker, Kaufmann, Schleyer, Mahdi & Dietrich, 1987). In carbazole–barium–tris(dimethoxyethane), the only structurally characterized barium carbazolate to date, the barium dication is only σ-coordinated to both carbazolate anions (Mösges, Hampel, Kaupp & Schleyer, 1992).

The crystal structure of the title compound, (I), shows the metal complex to be located around a twofold axis, which passes through the barium dication and the O atom of one tetrahydrofuran ligand. In the selected setting for the non-centrosymmetric space group, this solvent molecule is oriented into the direction of the negative *b* axis. Of the four crystallographically independent solvent molecules, one is not connected to the barium counter dication and found disordered around the twofold axis in holes of the crystal packing. Its shortest intermolecular C—C distances to a neighbouring molecule amounts to 3.7 Å. The barium dication exhibits a coordination number of seven and is surrounded by two carbazole anions and five tetrahydrofuran molecules in an asymmetric environment. The coordination polyhedron around Ba1 can be described as a strongly distorted pentagonal bipyramid with the oxygen centres in the pentagonal plane and the nitrogen centres at both peaks. The Ba1—N1 distances amount to 2.772 (2) Å and the Ba—O distances to 2.773 (2) (two contacts), 2.790 (2) (two contacts) and 2.785 (2) Å. The barium dication is located 1.475 (4) Å above the carbazole plane. The angle between Ba1, N1 and the centroid of the five-membered ring amounts to 146.7 (1)°.

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