C32	0.3503 (12)	0.3844 (4)	0.5434 (4)	0.071 (7)
C33	0.3035 (10)	0.4170 (4)	0.4887 (4)	0.070 (6)
C34	0.4230 (11)	0.4172 (4)	0.4558 (4)	0.074 (8)
C35	0.5405 (11)	0.3858 (4)	0.4902 (5)	0.082 (8)
Cl	0.7480 (2)	0.4619 (1)	0.9854 (1)	0.091 (2)

Table 2. *Selected geometric parameters* (Å, °)

La—O1	2.491 (4)	La—O2	2.478 (4)
La—O3	2.440 (4)	La—OW1	2.651 (5)
La—OW2	2.619 (5)	La—O4	2.449 (4)
La—O5	2.519 (4)	La—O6	2.478 (4)
Co1—P1	2.161 (2)	Co1—P2	2.161 (2)
Co1—P3	2.164 (2)	P1—O1	1.503 (4)
P1—O7	1.592 (6)	P1—O8	1.622 (5)
P2—O2	1.512 (4)	P2—O9	1.610 (6)
P2—O10	1.586 (7)	P3—O3	1.493 (4)
P3—O11	1.592 (9)	P3—O12	1.575 (8)
Co2—P4	2.166 (2)	Co2—P5	2.165 (2)
Co2—P6	2.160 (2)	P4—O4	1.479 (4)
P4—O13	1.598 (5)	P4—O14	1.584 (6)
P5—O5	1.494 (4)	P5—O15	1.601 (6)
P5—O16	1.589 (6)	P6—O6	1.487 (5)
P6—O17	1.639 (8)	P6—O18	1.523 (10)
O2···OW1	2.946 (6)	O1···O2	2.968 (6)
O6···O1	3.144 (6)	O6···OW1	3.108 (6)
O3···OW2	3.147 (6)	O3···O5	3.105 (7)
O5···O4	2.843 (6)	O4···OW2	2.918 (6)
OW1—La—O1	111.8 (1)	OW1—La—O2	69.8 (1)
OW1—La—O3	138.2 (1)	OW2—La—O1	143.5 (1)
OW2—La—O2	76.8 (1)	OW2—La—O3	76.9 (1)
OW2—La—OW1	75.9 (2)	O4—La—O1	144.3 (1)
O4—La—O2	141.4 (1)	O4—La—O3	116.3 (1)
O4—La—OW1	82.9 (1)	O4—La—OW2	70.2 (1)
O5—La—O1	80.9 (1)	O5—La—O2	145.1 (1)
O5—La—O3	77.5 (1)	O5—La—OW1	143.4 (1)
O5—La—OW2	114.9 (1)	O5—La—O4	69.9 (1)
O6—La—O1	78.6 (1)	O6—La—O2	121.0 (1)
O6—La—O3	144.2 (1)	O6—La—OW1	74.5 (1)
O6—La—OW2	136.0 (1)	O6—La—O4	74.4 (1)
O6—La—O5	74.8 (1)	O2—La—O1	73.3 (2)
O2—La—O3	73.4 (2)	O1—La—O3	75.1 (2)
O2···O1···O6	89.8 (2)	O1···O2···OW1	92.3 (2)
O5···O3···OW2	87.6 (2)	O2···OW1···O6	91.0 (2)
O3···OW2···O4	86.3 (2)	O5···O4···OW2	97.4 (2)
O3···O5···O4	88.4 (2)	O1···O6···OW1	85.9 (2)

The structure was solved by Patterson method and difference Fourier techniques and refined using full-matrix least squares on F. Final refinement was performed with anisotropic displacement parameters for all non-H atoms and fixed isotropic displacement parameters ($U_{iso} = 0.08$ Å^2) and calculated positions for all H atoms, except those at H$_2$O which were refined with isotropic displacement parameters and fixed positional parameters obtained from the difference Fourier function.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN* (Fair, 1990). Structure solution: *SHELX76* (Sheldrick, 1976). Structure refinement: *SHELX76*. Molecular graphics: *ORTEPII* (Johnson, 1976).

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Trimethyl[(trimethylstannyl)methyl]-ammonium Iodide

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Abstract

The crystal and molecular structure of the title compound, [Sn(C$_4$H$_{11}$N)(CH$_3$)$_3$]I, has been determined [alternative coordination nomenclature: (trimethyl)(trimethylammoniomethyl)tin(IV) iodide]. Both the Sn and N atoms are in tetrahedral environments.

Comment

The Sn—C$_{sp^3}$ bond lengths in the title compound, (I), range from 2.126 (6) to 2.182 (5) Å and valency angles about Sn range from 102.7 (2) to 113.7 (2)°. The Sn—CH$_2$ bond length is longer than all three Sn—Me bond

(I)
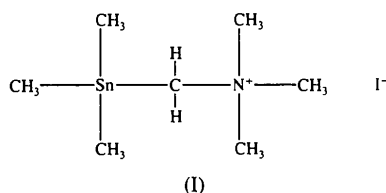
lengths, which are not significantly different from one another. The N—C_{sp^3} bond lengths range from 1.485 (6) to 1.499 (6) Å and the valency angles about N range from 108.6 (4) to 110.4 (4)°. The smaller variation in bond lengths and angles about N, in comparison to Sn, indicates a better tetrahedral environment for the N atom. This feature is also indicated by observing the atom displacements from the base of the tetrahedron formed by the mean plane through the three methyl C atoms and comparing the differences in displacements to the bond lengths. For Sn, the base is formed by C1, C2 and C3; the displacements are 0.644 (4) for Sn and 2.811 (6)Å for C4. The difference in the displacements, 2.167 (7) Å, compares with the Sn—C4 bond length of 2.182 (5) Å. Similarly for N, the base is formed by C5, C6 and C7 with displacements of 0.511 (5) and 2.006 (6) Å for N and C4, respectively, and the difference in displacements, 1.495 (8) Å, corresponds to the N—C4 bond length of 1.495 (6) Å. The slight distortion of the tetrahedral molecular framework for tetramethyltin has been discussed previously (Krebs, Henkel & Dartmann, 1989). In-plane bending is observed at C4 as the Sn—C4—N angle of 121.4 (3)° is significantly greater than the normal C_{sp^3} value.

The crystal structure of the Si analogue of (I), $(CH_3)_3SiCH_2(CH_3)_3N^+.I^-$, has been reported (Gurkova, Gusev, Abrosimova, Alexeev & Voronkov, 1981) and shows a slightly wider spread of angles about N (105.9–
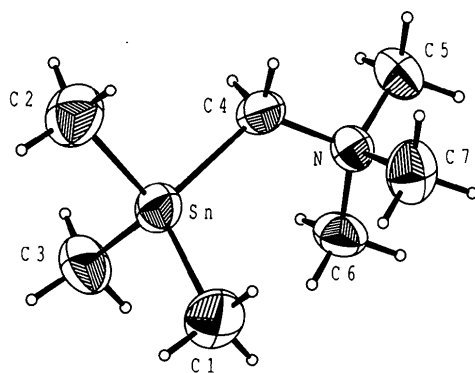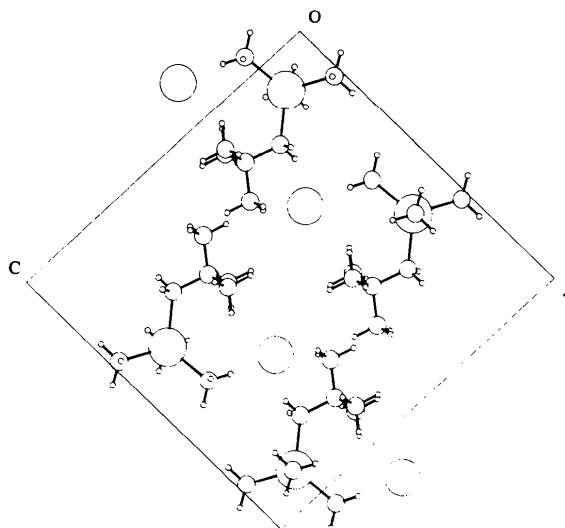

Fig. 2. A crystal packing diagram.

113.5°) as calculated from coordinates in the Cambridge Structural Database (1994, file reference 36621). The shortest $I^-\cdots N^+$ separation is 4.397 (1) in (I) and 4.41 Å in the Si analogue.

Experimental

The title compound was synthesized by reacting $Me_3SnCH_2NMe_2$ and MeI and was subsequently recrystallized from chloroform.

Crystal data

$[Sn(C_4H_{11}N)(CH_3)_3]I$	Mo $K\alpha$ radiation
$M_r = 363.83$	$\lambda = 0.71069$ Å
Monoclinic	Cell parameters from 250
$P2_1/a$	reflections
$a = 13.544$ (3) Å	$\theta = 3.02–25.04°$
$b = 6.8416$ (8) Å	$\mu = 4.246$ mm^{-1}
$c = 14.2204$ (6) Å	$T = 150$ (2) K
$\beta = 94.044$ (8)°	Lozenge
$V = 1314.5$ (3) Å^3	$0.4 \times 0.2 \times 0.15$ mm
$Z = 4$	Colourless
$D_x = 1.838$ Mg m^{-3}	

Data collection

Delft Instruments FAST diffractometer	5284 measured reflections
Area detector	1975 independent reflections
Absorption correction:	1690 observed reflections
refined from ΔF	$[I > 2\sigma(I)]$
(*DIFABS*; Walker & Stuart, 1983)	$R_{int} = 0.056$
	$\theta_{max} = 25.04°$
$T_{min} = 0.25, T_{max} = 0.51$	$h = -15 \rightarrow 16$
	$k = -7 \rightarrow 8$
	$l = -16 \rightarrow 14$

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} = -0.012$
$R[F^2 > 2\sigma(F^2)] = 0.034$	$\Delta\rho_{max} = 1.321$ e Å^{-3}
$wR(F^2) = 0.096$	$\Delta\rho_{min} = -0.656$ e Å^{-3}

Fig. 1. The atomic arrangement in (1). Displacement ellipsoids are plotted at the 50% probability level.

$S = 0.920$
1974 reflections
93 parameters
H atoms in riding mode
$w = 1/[\sigma^2(F_o^2) + (0.0613P)^2]$
  where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none
Atomic scattering factors
from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	U_{eq}
I	−0.12456 (2)	−0.24984 (5)	0.32861 (3)	0.0598 (2)
Sn	0.09678 (2)	0.14396 (6)	0.14059 (2)	0.0528 (2)
N	0.1698 (3)	0.2392 (5)	0.3565 (3)	0.0419 (9)
C1	−0.0515 (4)	0.2442 (11)	0.1537 (5)	0.078 (2)
C2	0.1598 (5)	0.2709 (11)	0.0221 (4)	0.081 (2)
C3	0.1012 (4)	−0.1689 (10)	0.1363 (4)	0.074 (2)
C4	0.2004 (3)	0.2311 (8)	0.2575 (3)	0.0491 (12)
C5	0.2593 (4)	0.2559 (8)	0.4226 (4)	0.0551 (13)
C6	0.1151 (4)	0.0566 (9)	0.3792 (4)	0.0583 (13)
C7	0.1051 (4)	0.4136 (9)	0.3695 (4)	0.067 (2)

Table 2. *Selected geometric parameters* (Å, °)

Sn—C2	2.126 (6)	N—C7	1.499 (6)
Sn—C3	2.142 (7)	N—C6	1.499 (6)
Sn—C1	2.143 (5)	N—C4	1.495 (6)
Sn—C4	2.182 (5)	N—C5	1.485 (6)
C2—Sn—C3	111.9 (3)	C7—N—C4	110.4 (4)
C2—Sn—C1	111.7 (3)	C6—N—C4	110.2 (4)
C3—Sn—C1	110.5 (3)	C7—N—C5	108.6 (4)
C2—Sn—C4	102.7 (2)	C6—N—C5	108.7 (4)
C3—Sn—C4	106.1 (2)	C4—N—C5	109.3 (4)
C1—Sn—C4	113.7 (2)	N—C4—Sn	121.4 (3)
C7—N—C6	109.5 (4)		

All non-H atoms were given anisotropic temperature factors; the methylene and methyl H atoms were given isotropic temperature factors and were allowed to ride on the C atoms to which they are attached.

Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SNOOPI* (Davies, 1983).

The use of the SERC X-ray crystallographic service at Cardiff University is gratefully acknowledged.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1164). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Gadolinium(III) Bicyclic Phthalocyanine

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Abstract

A new gadolinium bicyclic phthalocyanine complex, {14,28-[3,3′-iminobis(1-isoindolylideneamino)]phthalocyaninato(3−)}gadolinium(III), [Gd($C_{48}H_{24}N_{13}$)], has been obtained from gadolinium and 1,2-dicyanobenzene under a low pressure of nitrogen at 483 K. The crystals consist of separate molecules of the title compound. The bicyclic hexadentate phthalocyaninato ligand is composed of six isoindole units and forms a distorted trigonal prism by coordination *via* its six N-donor atoms, one from each isoindole ring. Gd—N distances range from 2.231 (6) to 2.282 (6) Å. The conjugation of the inner 16-membered ring is interrupted by the two sp^3-hybridized C atoms.

Comment

The crystal structure of gadolinium(III) bicyclic phthalocyanine, GdPcc, consists of discrete molecules of six-coordinate Gd^{III} surrounded by the six isoindole N atoms of the bicyclic phthalocyaninato chelate ligand.

GdPcc
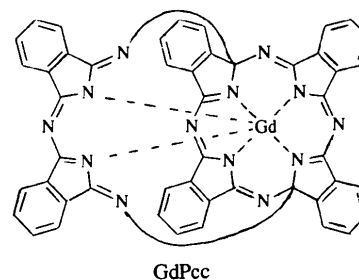
The bicyclic phthalocyaninato ligand consists of the phthalocyaninato skeleton of four isoindole units which is bridged by two additional isoindole moieties. The six N-donor atoms of the whole bicyclic phthalocyaninato ligand form a distorted trigonal prism. The macrocyclic hexadentate chelate ligand can also be described as two phthalocyaninato anions having one common half. It can be assumed that the macrocyclic ligand is less stable than the phthalocyaninato anion as the conjugated π-electron system of the inner 16-membered ring is interrupted at the two sp^3-hybridized bridgehead atoms, C(9) and C(25), of the ring linkage to the bicyclo system.