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

Zr(1)—O(5)	1.908 (2)	Zr(1)—O(4)	2.084 (2)
Zr(1)—C(14)	2.503 (3)	Zr(1)—C(19)	2.514 (3)
Zr(1)—C(20)	2.526 (3)	Zr(1)—C(21)	2.531 (3)
Zr(1)—C(15)	2.536 (3)	Zr(1)—C(18)	2.542 (3)
Zr(1)—C(22)	2.549 (3)	Zr(1)—C(23)	2.550 (3)
Zr(1)—C(17)	2.555 (3)	Zr(1)—C(16)	2.572 (3)
V(1)—C(1)	1.896 (3)	V(1)—C(2)	1.906 (3)
V(1)—C(3)	1.926 (3)	V(1)—C(4)	2.096 (2)
V(1)—C(25)	2.266 (2)	V(1)—C(24)	2.282 (3)
V(1)—C(26)	2.285 (3)	V(1)—C(28)	2.300 (3)
V(1)—C(27)	2.311 (3)	O(1)—C(1)	1.154 (3)
O(2)—C(2)	1.154 (3)	O(3)—C(3)	1.145 (3)
O(4)—C(4)	1.280 (2)	O(5)—C(9)	1.415 (3)
C(4)—C(5)	1.540 (3)	C(5)—C(6)	1.493 (3)
C(6)—C(7)	1.312 (3)	C(7)—C(8)	1.488 (3)
C(8)—C(9)	1.536 (4)	C(9)—C(13)	1.527 (3)
C(9)—C(10)	1.539 (3)	C(10)—C(11)	1.528 (4)
C(11)—C(12)	1.530 (4)	C(12)—C(13)	1.523 (4)
C(14)—C(18)	1.394 (5)	C(14)—C(15)	1.404 (4)
C(15)—C(16)	1.396 (4)	C(16)—C(17)	1.404 (4)
C(17)—C(18)	1.395 (4)	C(19)—C(20)	1.393 (5)
C(19)—C(23)	1.402 (5)	C(20)—C(21)	1.398 (4)
C(21)—C(22)	1.393 (5)	C(22)—C(23)	1.383 (5)
C(24)—C(25)	1.396 (4)	C(24)—C(28)	1.410 (4)
C(25)—C(26)	1.402 (4)	C(26)—C(27)	1.394 (4)
C(27)—C(28)	1.390 (5)		
O(5)—Zr(1)—O(4)	104.41 (7)	C(1)—V(1)—C(2)	75.79 (11)
C(1)—V(1)—C(3)	115.93 (11)	C(2)—V(1)—C(3)	75.61 (12)
C(1)—V(1)—C(4)	71.24 (9)	C(2)—V(1)—C(4)	119.17 (10)
C(3)—V(1)—C(4)	75.12 (10)	C(4)—O(4)—Zr(1)	168.2 (2)
C(9)—O(5)—Zr(1)	172.3 (2)	O(1)—C(1)—V(1)	178.4 (2)
O(2)—C(2)—V(1)	177.5 (3)	O(3)—C(3)—V(1)	179.8 (3)
O(4)—C(4)—C(5)	112.9 (2)	O(4)—C(4)—V(1)	126.9 (2)
C(5)—C(4)—V(1)	120.1 (2)	C(6)—C(5)—C(4)	113.3 (2)
C(7)—C(6)—C(5)	125.3 (2)	C(6)—C(7)—C(8)	123.3 (2)
C(7)—C(8)—C(9)	111.4 (2)	O(5)—C(9)—C(13)	109.4 (2)
O(5)—C(9)—C(8)	108.2 (2)	C(13)—C(9)—C(8)	113.1 (2)
O(5)—C(9)—C(10)	109.1 (2)	C(13)—C(9)—C(10)	103.0 (2)
C(8)—C(9)—C(10)	113.9 (2)	C(11)—C(10)—C(9)	106.5 (2)
C(10)—C(11)—C(12)	106.8 (2)	C(13)—C(12)—C(11)	103.9 (2)
C(12)—C(13)—C(9)	104.2 (2)	C(18)—C(14)—C(15)	107.9 (3)
C(16)—C(15)—C(14)	108.1 (3)	C(15)—C(16)—C(17)	107.7 (3)
C(18)—C(17)—C(16)	108.1 (3)	C(14)—C(18)—C(17)	108.2 (3)
C(20)—C(19)—C(23)	107.9 (3)	C(19)—C(20)—C(21)	108.0 (3)
C(22)—C(21)—C(20)	107.7 (3)	C(23)—C(22)—C(21)	108.6 (3)
C(23)—C(22)—Zr(1)	74.3 (2)	C(21)—C(22)—Zr(1)	73.4 (2)
C(22)—C(23)—C(19)	107.8 (3)	C(22)—C(23)—Zr(1)	74.2 (2)
C(19)—C(23)—Zr(1)	72.5 (2)	C(25)—C(24)—C(28)	107.7 (3)
C(25)—C(24)—V(1)	71.5 (2)	C(28)—C(24)—V(1)	72.8 (2)
C(24)—C(25)—C(26)	107.8 (3)	C(24)—C(25)—V(1)	72.7 (2)
C(26)—C(25)—V(1)	72.8 (2)	C(27)—C(26)—C(25)	108.3 (3)
C(27)—C(26)—V(1)	73.3 (2)	C(25)—C(26)—V(1)	71.3 (2)
C(28)—C(27)—C(26)	108.1 (3)	C(28)—C(27)—V(1)	72.0 (2)
C(26)—C(27)—V(1)	71.4 (2)	C(27)—C(28)—C(24)	108.1 (3)
C(27)—C(28)—V(1)	72.9 (2)	C(24)—C(28)—V(1)	71.4 (2)

In addition to the metalloxycarbene complex, a toluene molecule, disordered about a crystallographic centre of inversion, was located. The positions of the six C atoms of the phenyl ring were refined as a rigid body with an occupancy factor of 0.5 and the methyl C atom was located by a difference Fourier analysis.

Data collection and cell refinement: *EXPRESS* (Enraf–Nonius, 1993). Data reduction: local programs. Structure solution: *SHELXS*86 (Sheldrick, 1985). Structure refinement: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SCHAKAL*92 (Keller, 1992).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving non-H atoms have been deposited with the IUCr (Reference: JZ1002). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tetrabutylbis[(*N*,*N*-diethylthiocarbamoylthio)acetato]distannoxane Dimer

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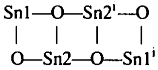
Abstract

In the centrosymmetric title compound, octabutyl-$1\kappa^2C,2\kappa^2C,3\kappa^2C,4\kappa^2C$-tetrakis[$\mu$-($N$,$N$-diethylthiocarbamoylthio)acetato]-$1{:}2\kappa^2O,O';2{:}3\kappa^2O;3{:}4\kappa^2O,O';1{:}4\kappa^2O$-bis-$\mu_3$-oxo-$1{:}2{:}3\kappa^3O;1{:}3{:}4\kappa^3O$-tetratin, $[Sn_4O_2(C_7H_{12}NO_2S_2)_4(C_4H_9)_8]$, one Sn atom is five-coordinate having *cis*-C_2SnO_3 trigonal bipyramidal geometry, whereas the other is six-coordinate having C_2SnO_4 skew-trapezoidal bipyramidal geometry with apical disposition of the butyl groups.

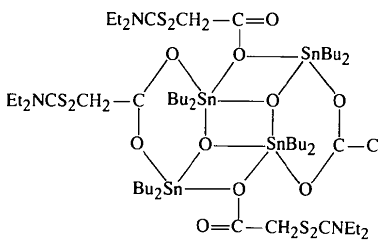
Comment

Dicarboxylatotetraorganodistannoxanes $[(R_2CO_2Sn\text{—}O\text{—}SnO_2CR_2)_2O]_2$ are often formed instead of diorganotin dicarboxylate esters when diorganotin oxides are reacted with carboxylic acids and are the incompletely hydrolyzed products. The dimeric compounds contain the



core unit. Distannoxanes are generally centrosymmetric compounds whose five-coordinate Sn1 atom displays *cis*-trigonal bipyramidal coordination. The Sn2 atom is basically five-coordinate also, but its geometry is distorted towards skew-trapezoidal bipyramidal coordination owing to a long carbonyl O···Sn interaction (Ng, Chen & Kumar Das, 1991; Ng, Kumar Das, Yip & Mak, 1994).

In the title compound, (I), the two monomeric halves are linked by a short intradimer bond [Sn2—O5^{i} 2.043 (4) Å]. One type of carboxylato anion bridges the Sn1—O5—Sn2 unit through the three-atom carboxyl group [Sn1—O1 2.264 (5), Sn2—O2^{i} 2.291 (5) Å] to give Sn—O bonds of nearly equal length. The Sn1 atom has *cis*-trigonal bipyramidal coordination, but the distortion of the geometry is severe [C1—Sn1—C5 139.6 (3)°].



(I)

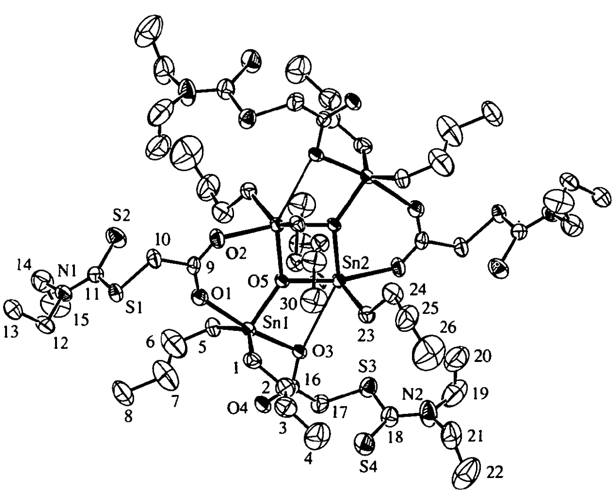


Fig. 1. The atomic labeling scheme for the title dimer (C atoms are labeled by number only). Displacement ellipsoids are plotted at the 30% probability level.

The other type of anion bridges through the ester O atom only, resulting in a short Sn—O bond [Sn1—O3 2.179 (4) Å] and a relatively weak Sn···O interaction [Sn2···O3 2.784 (4) Å]. If this interaction is regarded as a formal dative bond, the coordination geometry is skew-trapezoidal bipyramidal in which the α-C atoms of the butyl groups extend over the long edge of the trapezoidal plane.

Experimental

The title distannoxane was obtained instead of the expected dibutylbis[(*N*,*N*-diethylthiocarbamoylthio)acetato]tin compound from the condensation of dibutyltin oxide and (*N*,*N*-diethylthiocarbamoylthio)acetic acid (1:2 molar ratio). The reactants were heated in a small volume of ethanol until the oxide had dissolved completely. The compound that precipitated from the cool solution was recrystallized from ethanol to give well defined crystals.

Crystal data

$[Sn_4O_2(C_7H_{12}NO_2S_2)_4(C_4H_9)_8]$
M_r = 1916.87
Triclinic
$P\bar{1}$
a = 11.8709 (8) Å
b = 13.625 (2) Å
c = 13.8088 (14) Å
α = 88.684 (9)°
β = 75.199 (7)°
γ = 72.618 (7)°
V = 2057.2 (3) Å^3
Z = 1
D_x = 1.547 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 8–10°
μ = 1.452 mm^{-1}
T = 298 K
Block
0.28 × 0.21 × 0.14 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
7591 measured reflections
7208 independent reflections
4697 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int}$ = 0.0272
$\theta_{\rm max}$ = 24.98°
$h = 0 \rightarrow 14$
$k = -15 \rightarrow 16$
$l = -15 \rightarrow 16$
3 standard reflections frequency: 60 min intensity decay: 2.9%

Refinement

Refinement on F^2
$R(F)$ = 0.0501
$wR(F^2)$ = 0.1133
S = 1.025
7208 reflections
388 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0422P)^2 + 0.3089P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max}$ = 0.001
$\Delta\rho_{\rm max}$ = 0.460 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.506 e Å^{-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)

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

	x	y	z	U_{eq}
Sn1	0.16618 (4)	0.51355 (4)	0.26968 (3)	0.0498 (2)
Sn2	−0.12148 (4)	0.52959 (4)	0.45381 (3)	0.04069 (15)
S1	0.6214 (2)	0.3932 (2)	0.24971 (15)	0.0574 (5)
S2	0.5663 (2)	0.1967 (2)	0.3130 (2)	0.0853 (7)
S3	−0.2731 (2)	0.6148 (2)	0.2180 (2)	0.0694 (6)
S4	−0.2112 (2)	0.8031 (2)	0.1428 (2)	0.1047 (10)
O1	0.3469 (5)	0.4643 (5)	0.3109 (4)	0.084 (2)
O2	0.3227 (4)	0.4551 (4)	0.4733 (4)	0.0633 (14)
O3	−0.0197 (4)	0.5547 (4)	0.2531 (3)	0.0514 (12)
O4	0.0779 (5)	0.5626 (4)	0.0948 (4)	0.0673 (15)
O5	0.0763 (4)	0.5051 (3)	0.4136 (3)	0.0404 (10)
N1	0.7249 (6)	0.2172 (5)	0.1461 (5)	0.065 (2)
N2	−0.4218 (7)	0.8033 (6)	0.2592 (7)	0.109 (3)
C1	0.1973 (7)	0.6583 (6)	0.2501 (6)	0.066 (2)
C2	0.0999 (8)	0.7507 (7)	0.2333 (7)	0.079 (3)
C3	0.1322 (9)	0.8498 (7)	0.2239 (7)	0.091 (3)
C4	0.0352 (11)	0.9377 (8)	0.1988 (10)	0.141 (5)
C5	0.2244 (8)	0.3652 (6)	0.1929 (6)	0.080 (3)
C6	0.3385 (13)	0.3319 (13)	0.1138 (10)	0.160 (6)
C7	0.3756 (13)	0.3949 (13)	0.0517 (11)	0.170 (7)
C8	0.4980 (10)	0.3545 (12)	−0.0349 (8)	0.158 (6)
C9	0.3857 (6)	0.4493 (5)	0.3859 (6)	0.050 (2)
C10	0.5232 (6)	0.4235 (6)	0.3734 (5)	0.061 (2)
C11	0.6438 (6)	0.2597 (6)	0.2305 (5)	0.054 (2)
C12	0.7953 (8)	0.2739 (7)	0.0761 (6)	0.077 (3)
C13	0.9186 (8)	0.2623 (8)	0.0961 (7)	0.097 (3)
C14	0.7569 (10)	0.1062 (7)	0.1198 (8)	0.096 (3)
C15	0.6812 (12)	0.0880 (8)	0.0542 (9)	0.134 (5)
C16	−0.0153 (6)	0.5626 (5)	0.1579 (5)	0.048 (2)
C17	−0.1308 (7)	0.5683 (7)	0.1279 (6)	0.070 (2)
C18	−0.3087 (7)	0.7493 (6)	0.2076 (6)	0.069 (2)
C19	−0.5313 (13)	0.7445 (10)	0.3019 (11)	0.150 (6)
C20	−0.5150 (13)	0.7381 (10)	0.3934 (10)	0.167 (6)
C21	−0.4686 (11)	0.9161 (8)	0.2603 (10)	0.124 (4)
C22	−0.5374 (12)	0.9511 (10)	0.1787 (13)	0.187 (7)
C23	−0.1725 (7)	0.6916 (5)	0.4463 (5)	0.060 (2)
C24	−0.2208 (9)	0.7526 (6)	0.5433 (7)	0.088 (3)
C25	−0.2362 (12)	0.8686 (9)	0.5342 (10)	0.128 (4)
C26	−0.3187 (16)	0.9184 (11)	0.4738 (13)	0.211 (8)
C27	−0.1139 (6)	0.3924 (5)	0.3813 (5)	0.051 (2)
C28	−0.1327 (7)	0.3069 (6)	0.4485 (6)	0.063 (2)
C29	−0.1139 (10)	0.2070 (7)	0.3932 (7)	0.093 (3)
C30	−0.1268 (11)	0.1207 (8)	0.4605 (9)	0.129 (4)

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

Sn1—O1	2.264 (5)	Sn2—O2^i	2.291 (5)
Sn1—O3	2.179 (4)	Sn2—O5	2.192 (4)
Sn1—O5	2.020 (4)	Sn2—O5^i	2.043 (4)
Sn1—C1	2.112 (8)	Sn2—C23	2.114 (7)
Sn1—C5	2.139 (8)	Sn2—C27	2.107 (7)
O1—Sn1—O3	171.2 (2)	O5^i—Sn2—C23	107.8 (2)
O1—Sn1—O5	90.5 (2)	O5^i—Sn2—C27	108.6 (2)
O1—Sn1—C1	84.7 (3)	C23—Sn2—O2^i	88.2 (3)
O1—Sn1—C5	86.3 (3)	C23—Sn2—O5	96.1 (2)
O3—Sn1—O5	80.8 (2)	C27—Sn2—O2^i	86.3 (2)
O3—Sn1—C1	99.6 (2)	C27—Sn2—O5	96.4 (2)
O3—Sn1—C5	95.1 (3)	C27—Sn2—C23	143.3 (3)
O5—Sn1—C1	109.8 (2)	C9—O1—Sn1	139.4 (5)
O5—Sn1—C5	109.5 (3)	C9—O2—Sn2^i	134.7 (5)
C1—Sn1—C5	139.6 (3)	C16—O3—Sn1	108.6 (4)
O5—Sn2—O2^i	168.5 (2)	Sn1—O5—Sn2^i	136.6 (2)
O5^i—Sn2—O2^i	91.8 (2)	Sn1—O5—Sn2	120.0 (2)
O5^i—Sn2—O5	76.7 (2)	Sn2^i—O5—Sn2	103.3 (2)

Symmetry code: (i) $-x, 1-y, 1-z$.

H atoms were allowed to ride on their parent C atoms with displacement factors 1.5 times those of the C atoms.

Data collection: *CAD*-4 *VAX/PC Fortran System* (Enraf–Nonius, 1988). Cell refinement: *CAD*-4 *VAX/PC Fortran System*. Data reduction: *Xtal*3.0 (Hall & Stewart, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1020). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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μ-Phenylphosphido-μ-[(pentacarbonylmanganese)phenylphosphido]-bis(tetracarbonylrhenium)

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Abstract

A μ_3-phenylphosphido group bridges an Mn and two Re atoms in the title compound, $[Re_2(C_6H_5PH)\{C_6H_5PMn(CO)_5\}(CO)_8]$ (IUPAC name: tridecacarbonyl-1κ^5C,2κ^4C,3κ^4C-μ_3-phenylphosphanediido-1:2:3κ^3P-μ-phenylphosphanido-2:3κ^2P-manganesedirhenium). The Re atoms each have four carbonyl ligands and a common μ-