C(12)	0.6018 (6)	0.1549 (2)	0.9887 (4)	0.038 (1)
C(13)	0.5530 (6)	0.0940 (2)	0.9544 (4)	0.038 (1)
C(14)	0.4555 (6)	0.0807 (2)	0.8255 (4)	0.032 (1)
C(15)	0.4117 (7)	0.0772 (2)	0.2815 (5)	0.043 (1)
C(16)	−0.0952 (6)	0.0913 (2)	0.3179 (4)	0.040 (1)

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

Sn—O(1)	2.112 (3)	O(2)—C(1)	1.256 (5)
Sn—O(2)	2.503 (3)	O(3)—C(3)	1.353 (5)
Sn—O(4)	2.111 (3)	O(4)—C(8)	1.292 (5)
Sn—O(5)	2.577 (3)	O(5)—C(8)	1.254 (5)
Sn—C(15)	2.092 (5)	O(6)—C(10)	1.343 (5)
Sn—C(16)	2.092 (4)	C(1)—C(2)	1.460 (6)
O(1)—C(1)	1.301 (5)	C(8)—C(9)	1.482 (6)
O(1)—Sn—O(2)	56.0 (1)	Sn—O(5)—C(8)	82.2 (2)
O(1)—Sn—O(4)	82.9 (1)	O(1)—C(1)—O(2)	118.0 (4)
O(1)—Sn—O(5)	137.9 (1)	O(1)—C(1)—C(2)	119.8 (4)
O(1)—Sn—C(15)	106.5 (2)	O(2)—C(1)—C(2)	122.2 (4)
O(1)—Sn—C(16)	106.7 (2)	C(1)—C(2)—C(3)	120.4 (4)
O(2)—Sn—O(4)	138.9 (1)	C(1)—C(2)—C(7)	121.0 (4)
O(2)—Sn—O(5)	166.1 (1)	C(3)—C(2)—C(7)	118.6 (4)
O(2)—Sn—C(15)	90.0 (1)	O(3)—C(3)—C(2)	123.5 (4)
O(2)—Sn—C(16)	88.0 (2)	O(3)—C(3)—C(4)	116.8 (4)
O(4)—Sn—O(5)	55.0 (1)	O(4)—C(8)—O(5)	119.8 (4)
O(4)—Sn—C(15)	102.9 (2)	O(4)—C(8)—C(9)	118.6 (4)
O(4)—Sn—C(16)	105.7 (1)	O(5)—C(8)—C(9)	121.6 (4)
O(5)—Sn—C(15)	84.8 (1)	C(8)—C(9)—C(10)	120.1 (4)
O(5)—Sn—C(16)	87.3 (2)	C(8)—C(9)—C(14)	120.4 (4)
C(15)—Sn—C(16)	138.2 (2)	C(10)—C(9)—C(14)	119.5 (4)
Sn—O(1)—C(1)	101.5 (3)	O(6)—C(10)—C(9)	123.3 (4)
Sn—O(2)—C(1)	84.6 (2)	O(6)—C(10)—C(11)	117.7 (4)
Sn—O(4)—C(8)	102.9 (2)		

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation (1992). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1090). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N,N′-Bis[*trans*-2-phenyl-5-(triphenylstannylmethoxymethyl)-1,3-dioxan-5-yl]ethanediamide

JENNIFER-NICOLA ROSS,[a] JAMES L. WARDELL,[a] JOHN N. LOW[b]* AND GEORGE FERGUSON[c]

[a]*Department of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB9 2UE, Scotland,* [b]*Applied Physics and Electronic & Mechanical Engineering, University of Dundee, Dundee DD1 4HN, Scotland, and* [c]*Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario, Canada N1G 2W1. E-mail: j.n.low@dundee.ac.uk*

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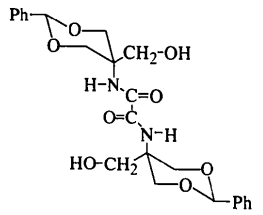
Abstract

The title compound, μ-{*N,N′*-bis[*trans*-5-(methoxymethyl)-2-phenyl-1,3-dioxan-5-yl]ethanediamide(2−)}-bis(triphenyltin), $[Sn_2(C_6H_5)_6(C_{26}H_{30}N_2O_8)]$, was isolated from the products of the (triphenylstannyl)-methylation of a 1:1 mixture of stereoisomers of *N,N′*-bis(5-hydroxymethyl-2-phenyl-1,3-dioxan-5-yl)ethanediamide. The molecule lies about an inversion centre and the unique Sn atom has distorted tetrahedral geometry, with Sn—C_{alkyl} and Sn—C_{aryl} distances of 2.154 (2) and 2.138 (2)–2.147 (3) Å, respectively, and C—Sn—C angles in the range 105.5 (1)–113.2 (1)°.

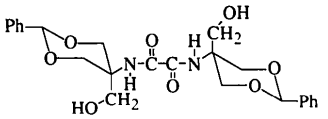
Comment

The crystal structure of a 1:1 mixture of stereoisomers of *N,N′*-bis(5-hydroxymethyl-2-phenyl-1,3-dioxan-5-yl)ethanediamide [(I*a*) and (I*b*)] has been reported recently (Ross, Wardell, Low & Ferguson, 1996). Reaction of Ph_3SnCH_2I with this 1:1 mixture in the presence of NaH in dimethylformamide solution was found to give a product mixture which exhibited four ^{119}Sn NMR chemical shift values in the region −145.2 to −139.8 p.p.m. (*i.e.* the region expected for compounds of the type Ph_3SnCH_2OR) (Cox, Doidge-Harrison, Howie & Wardell, 1991). Chromatographic separation of the product mixture resulted in the isolation of the crystalline title compound, (II) [NMR ($CDCl_3$): δ^{119}Sn −139.8 p.p.m.]. In order to both characterize this product unambiguously and determine its stereochemistry, the crystal structure determination of (II) was carried out.

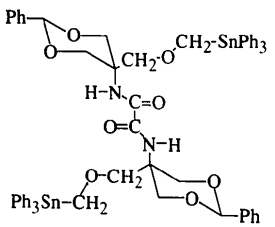
Analysis showed compound (II) (Fig. 1) to be clearly derived from the *trans* stereoisomer, (I*a*), of the (I*a*)/(I*b*) mixture, which had axial N—H moieties. The molecule of (II) lies about an inversion centre and the conformation is stabilized by intramolecular N—H···O bifurcated hydrogen bonding (Fig. 1 and Table 2). There are



(Ia)



(Ib)



(II)

no intermolecular hydrogen bonds and intermolecular contacts are of the van der Waals variety. In general, the core structure of (II) is similar to that of its precursor molecule (I*a*) and the 1,3-dioxane ring system has the same chair-like conformation as was found for (I*a*), with both the phenyl ring at the C2 position and the hydroxymethyl moiety at the C5 position equatorial.

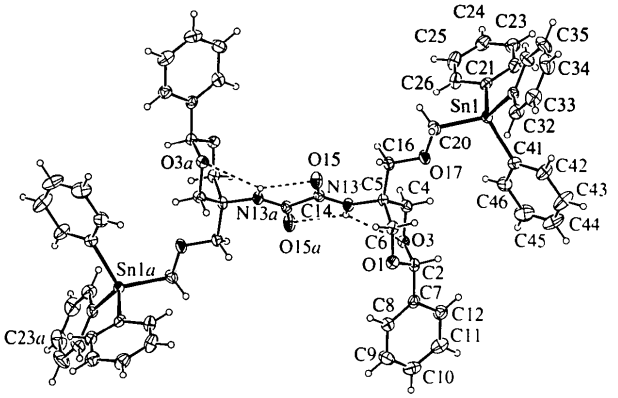


Fig. 1. A view of (II) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Atom labels with the suffix '*a*' refer to atoms at the equivalent position $1-x$, $-y$, $1-z$.

The unique Sn atom in compound (II) has a distorted tetrahedral coordination, with bond angles at the Sn atom varying between 105.5 (1) and 113.2 (1)°. The phenyl rings are arranged in an irregular propeller conformation with Sn—C_{aryl} bond lengths in the range 2.138 (2)–2.147 (3) Å and an Sn—C_{alkyl} distance of 2.154 (2) Å. These dimensions are within the expected regions, as seen by comparison with the structure of chloro(3-ethoxypropyl)diphenylstannane (Forrester, Howie, Ross, Low & Wardell, 1991), where the Sn—C_{aryl} bond lengths range from 2.116 (4) to 2.135 (6) Å and the Sn—C_{alkyl} bond length is 2.139 (6) Å. The Sn1···O17 1,3-intramolecular separation is 2.909 (2) Å, but the geometry at the Sn1 atom does not indicate any significant Sn···O interaction.

Experimental

The title compound was isolated by chromatographic separation [chromatotron; hexane/ethyl acetate (2:1 *v*/*v*) as eluant] of the products obtained from the addition of Ph_3SnCH_2I to a solution of stereoisomers of *N*,*N*′-bis(5-hydroxymethyl-2-phenyl-1,3-dioxan-5-yl)ethanediamide and NaH in dimethylformamide solution. It was recrystallized from ethyl acetate (m.p. 443–447 K).

Crystal data

$[Sn_2(C_6H_5)_6(C_{26}H_{30}N_2O_8)]$
M_r = 1198.5
Triclinic
$P\bar{1}$
a = 6.1770 (7) Å
b = 9.7470 (12) Å
c = 23.236 (2) Å
α = 86.944 (11)°
β = 89.599 (9)°
γ = 77.619 (9)°
V = 1364.5 (3) Å^3
Z = 1
D_x = 1.459 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 25 reflections
θ = 17.6–21.8°
μ = 0.973 mm^{-1}
T = 294 (1) K
Plate
0.44 × 0.31 × 0.08 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: five ψ scans at 4° steps (North, Phillips & Mathews, 1968)
T_{min} = 0.6811, T_{max} = 0.8029
5907 measured reflections
5907 independent reflections

4987 observed reflections [$I > 2\sigma(I)$]
θ_{max} = 26.9°
$h = -7 \rightarrow 7$
$k = 0 \rightarrow 12$
$l = -29 \rightarrow 29$
3 standard reflections
frequency: 120 min
intensity variation: 1.5%

Refinement

Refinement on F^2
$R(F)$ = 0.0268
$wR(F^2)$ = 0.0691
S = 1.049
5907 reflections
334 parameters
H atoms riding (*SHELXL* defaults, C—H 0.93–0.98, N—H 0.86 Å)

$w = 1/[\sigma^2(F_o^2) + (0.0404P)^2 + 0.2277P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.011
$\Delta\rho_{max}$ = 0.634 e Å^{-3}
$\Delta\rho_{min}$ = −0.589 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 (Å²)*

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

	x	y	z	U_{eq}
Sn1	0.11179 (3)	0.18480 (2)	0.17387 (1)	0.03449 (6)
C21	−0.1774 (4)	0.3432 (2)	0.19106 (11)	0.0383 (5)
C22	−0.3379 (5)	0.3980 (3)	0.14980 (12)	0.0459 (6)
C23	−0.5257 (5)	0.4968 (3)	0.16355 (14)	0.0556 (7)
C24	−0.5587 (5)	0.5401 (3)	0.21870 (15)	0.0596 (8)
C25	−0.4032 (5)	0.4851 (4)	0.26064 (14)	0.0620 (8)
C26	−0.2146 (5)	0.3888 (3)	0.24675 (12)	0.0509 (7)
C31	0.2718 (4)	0.2272 (3)	0.09503 (10)	0.0391 (5)
C32	0.4591 (5)	0.1327 (3)	0.07781 (12)	0.0513 (7)
C33	0.5683 (6)	0.1580 (4)	0.02733 (14)	0.0664 (9)
C34	0.4920 (7)	0.2776 (5)	−0.00640 (14)	0.0746 (10)
C35	0.3087 (6)	0.3729 (4)	0.00984 (14)	0.0726 (10)
C36	0.1963 (5)	0.3493 (3)	0.06051 (12)	0.0517 (7)
C41	0.0116 (4)	−0.0096 (3)	0.16355 (11)	0.0406 (5)
C42	0.0471 (5)	−0.0770 (3)	0.11286 (14)	0.0596 (8)
C43	−0.0288 (6)	−0.1987 (4)	0.1049 (2)	0.0833 (12)
C44	−0.1408 (7)	−0.2537 (4)	0.1475 (2)	0.0861 (12)
C45	−0.1787 (7)	−0.1891 (4)	0.1979 (2)	0.0849 (12)
C46	−0.1044 (5)	−0.0673 (3)	0.20671 (13)	0.0586 (8)
C20	0.3286 (5)	0.1726 (3)	0.24701 (11)	0.0454 (6)
O1	0.4356 (3)	−0.3130 (2)	0.38508 (7)	0.0405 (4)
C2	0.2137 (4)	−0.3198 (2)	0.37418 (10)	0.0356 (5)
O3	0.0660 (3)	−0.2040 (2)	0.39769 (7)	0.0361 (3)
C4	0.0938 (4)	−0.0749 (2)	0.36913 (10)	0.0369 (5)
C5	0.3302 (4)	−0.0558 (2)	0.37832 (9)	0.0330 (5)
C6	0.4897 (4)	−0.1881 (3)	0.35936 (10)	0.0387 (5)
C7	0.1650 (4)	−0.4526 (3)	0.40146 (10)	0.0386 (5)
C8	0.2917 (5)	−0.5265 (3)	0.44598 (12)	0.0535 (7)
C9	0.2313 (6)	−0.6428 (3)	0.47304 (14)	0.0652 (9)
C10	0.0479 (6)	−0.6861 (3)	0.45558 (14)	0.0617 (8)
C11	−0.0764 (6)	−0.6155 (3)	0.4099 (2)	0.0625 (8)
C12	−0.0193 (5)	−0.4985 (3)	0.38311 (13)	0.0509 (6)
N13	0.3540 (3)	−0.0415 (2)	0.44025 (8)	0.0357 (4)
C14	0.5135 (4)	0.0035 (2)	0.46668 (9)	0.0342 (5)
O15	0.6656 (3)	0.0479 (2)	0.44435 (7)	0.0521 (5)
C16	0.3697 (4)	0.0744 (3)	0.34301 (9)	0.0388 (5)
O17	0.2752 (3)	0.0726 (2)	0.28769 (7)	0.0501 (5)

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

Sn1—C20	2.154 (2)	C4—C5	1.529 (3)
Sn1—C21	2.147 (3)	C5—N13	1.464 (3)
Sn1—C31	2.138 (2)	C5—C6	1.529 (3)
Sn1—C41	2.141 (2)	C5—C16	1.537 (3)
O1—C2	1.412 (3)	N13—C14	1.328 (3)
O1—C6	1.430 (3)	C14—O15	1.217 (3)
C2—O3	1.422 (3)	C14—C14^{i}	1.556 (4)
C2—C7	1.498 (3)	C16—O17	1.418 (3)
O3—C4	1.434 (3)		
C20—Sn1—C21	106.2 (1)	C21—Sn1—C31	113.2 (1)
C20—Sn1—C31	111.7 (1)	C21—Sn1—C41	108.5 (1)
C20—Sn1—C41	111.9 (1)	C31—Sn1—C41	105.5 (1)

D—H···A	D—H	H···A	D···A	D—H···A
N13—H13···O3	0.86	2.51	2.837 (3)	103
N13—H13···O15^{i}	0.86	2.26	2.681 (2)	110

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

Compound (II) crystallized in the triclinic system and space group $P\bar{1}$ was assumed and confirmed by the analysis. Examination of the structure with *PLATON* (Spek, 1995) showed that there were no solvent-accessible voids in the crystal lattice.

Data collection: *CAD-4/PC Software* (Enraf–Nonius, 1992). Cell refinement: *SET4* and *CELDIM* (Enraf–Nonius, 1992). Data reduction: *DATRD2* in *NRCVAX*94 (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SOLVER* in *NRCVAX*94. Program(s) used to refine structure: *NRCVAX*94 and *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976) in *PLATON*. Software used to prepare material for publication: *NRCVAX*94, *SHELXL*93 and *WordPerfect*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: AB1356). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 1963–1966

catena-Poly[bis(*O*,*O*′-diethyldithiophosphato-*S*)zinc(II)-μ-4,4′-bipyridyl-*N*:*N*′]

DUO-LIN ZHU,[a] YUN-PENG YU,[a] GOU-CONG GUO,[b] HONG-HUI ZHUANG,[b] JIN-SHUN HUANG,[b] QI LIU,[c] ZHENG XU[c]* AND XIAO-ZENG YOU[c]

[a]*Department of Chemistry, Zhenjiang Teachers College, Zhenjiang 212003, People's Republic of China,* [b]*National Key Laboratory of Structure Chemistry, Fuzhou 350002, People's Republic of China, and* [c]*Coordination Chemistry Institute, National Key Laboratory of Coordination Chemistry, Nanjing University, Nanjing 210008, People's Republic of China*

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Abstract

The crystal structure of $[Zn\{(C_2H_5O)_2S_2P\}_2(C_{10}H_8N_2)]$ contains polymeric zigzag chains. The asymmetric unit comprises two independent zinc centres having similar distorted-tetrahedral coordination geometries. Each Zn