

Table 2. Selected bond distances (Å) and angles (°) with e.s.d.'s in parentheses

Se(1)	Se(2)	2.265 (3)	Se(2)	Se(1)	N(1)	90.6 (5)
Se(1)	N(1)	1.724 (15)	Se(1)	Se(2)	N(2)	91.5 (5)
Se(2)	N(2)	1.714 (15)	Se(4)	Se(3)	N(3)	91.0 (5)
Se(3)	Se(4)	2.253 (3)	Se(3)	Se(4)	N(4)	91.2 (5)
Se(3)	N(3)	1.733 (15)	Se(1)	N(1)	C(1)	118 (1)
Se(4)	N(1)	1.73 (2)	Se(2)	N(2)	C(1)	117 (1)
C(1)	N(1)	1.32 (2)	Se(3)	N(3)	C(5)	117 (1)
C(1)	N(2)	1.35 (2)	Se(4)	N(4)	C(5)	115 (1)
C(1)	C(2)	1.49 (2)	N(1)	C(1)	N(2)	122 (2)
C(3)	C(4)	1.31 (2)	N(1)	C(1)	C(2)	120 (2)
C(2)	C(3)	1.38 (2)	N(2)	C(1)	C(2)	118 (2)
C(2)	C(4)	1.39 (2)	C(1)	C(2)	C(3)	121 (2)
C(5)	N(3)	1.28 (2)	C(1)	C(2)	C(4)	121 (2)
C(5)	N(4)	1.33 (2)	C(3)	C(2)	C(4)	118 (2)
C(5)	C(6)	1.49 (3)	C(2)	C(3)	C(4)	123 (2)
C(6)	C(7)	1.39 (2)	C(2)	C(4)	C(3)	119 (2)
C(6)	C(8)	1.47 (2)	N(3)	C(5)	N(4)	126 (2)
C(7)	C(8 ⁺)	1.35 (2)	N(2)	C(5)	C(6)	117 (2)
Sb	F	1.816–1.895	N(4)	C(5)	C(6)	117 (2)
		1.849 (av.)	C(5)	C(6)	C(7)	120 (2)
			C(5)	C(6)	C(8)	123 (2)
			C(6)	C(7)	C(8)	117 (2)
			C(6)	C(7)	C(8 ⁺)	119 (2)
			C(6)	C(8)	C(7 ⁺)	124 (2)

scheme; the other cation was numbered in a corresponding manner.

Related literature. The structural parameters of the cation in this crystal can be compared to those found in the closely related monofunctional (PhCN₂Se₂)⁺ cation (Del bel Belluz, Cordes, Kristof, Kristof, Liblong & Oakley, 1990) and those of the corresponding sulfur-based dication (S₂N₂C—C₆H₄—CN₂S₂)²⁺ (Liblong, Oakley & Cordes, 1990).

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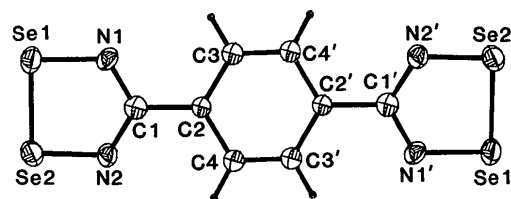


Fig. 1. ORTEP (Johnson, 1976) drawing and numbering scheme for the cation containing Se(1). The ellipsoids are drawn at the 30% probability surface and H atoms have been given arbitrary radii for figure clarity.

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Structure of Triphenyltin Adduct C₂₆H₂₈O₂Sn* Resulting from a Free-Radical Reaction

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Abstract. C₂₆H₂₈O₂Sn, *M_r* = 491.20, monoclinic, *P*2₁/*c*, *a* = 6.557 (2), *b* = 36.419 (8), *c* = 9.964 (4) Å, β = 110.23 (3)°, *V* = 2232.6 Å³, *Z* = 4, *D_x* = 1.462 Mg m⁻³, λ(Cu Kα) = 1.54178 Å, μ = 9.45 mm⁻¹, *F*(000) = 1000, *T* = 220 K, *R* = 0.055,

wR = 0.050 for 2569 observed reflections. During the course of some triphenyltin hydride-mediated cyclizations of activated and unactivated dienes, it was found that a major product resulted from addition of the hydride to the terminal double bond followed by intramolecular cyclization. The structure was analyzed by X-rays to determine the relative configuration of the ring junction which could not be established unambiguously by ¹H NMR studies. The

* (±)-*cis*-4-(Triphenylstannylmethyl)-2-oxabicyclo[3.3.0]octan-7-ol.

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cis-fused oxabicyclo[3.3.0] system contains a triphenylstannylmethyl moiety which is in the concave face of the molecule.

Table 1. Final atomic coordinates and their *e.s.d.*'s ($\times 10^4$, Sn $\times 10^5$) and U_{eq} ($\text{\AA}^2 \times 10^3$) for C₂₆H₂₈O₂Sn

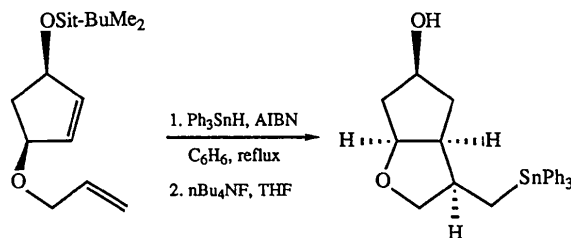
$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	U_{eq}
Sn	32959 (11)	10727 (1)	19400 (6)	64
O(1)	9832 (15)	2778 (2)	3116 (9)	156
O(2)	3407 (14)	2400 (2)	420 (8)	112
C(1)	8814 (17)	2573 (2)	1925 (12)	101
C(2)	6770 (17)	2734 (2)	965 (11)	108
C(3)	5338 (17)	2422 (3)	105 (12)	92
C(4)	3378 (17)	2054 (2)	1168 (10)	100
C(5)	4764 (14)	1804 (2)	730 (10)	72
C(6)	6542 (17)	2069 (2)	656 (11)	89
C(7)	8222 (16)	2179 (2)	2188 (9)	88
C(8)	5620 (14)	1469 (2)	1717 (9)	77
C(11)	489 (14)	981 (2)	57 (8)	63
C(12)	-803 (15)	1265 (2)	-695 (10)	83
C(13)	-2679 (17)	1198 (3)	-1825 (11)	100
C(14)	-3303 (17)	843 (3)	-2247 (10)	88
C(15)	-2027 (18)	559 (3)	-1533 (11)	98
C(16)	-146 (16)	628 (2)	-400 (9)	84
C(21)	5071 (15)	572 (2)	2430 (9)	66
C(22)	5866 (15)	423 (2)	1433 (10)	85
C(23)	7146 (18)	110 (3)	1747 (11)	105
C(24)	7657 (17)	-55 (3)	3079 (12)	94
C(25)	6909 (16)	92 (3)	4066 (11)	87
C(26)	5612 (15)	403 (2)	3733 (9)	77
C(31)	2197 (14)	1208 (2)	3647 (8)	62
C(32)	2805 (15)	1539 (2)	4430 (9)	73
C(33)	2023 (17)	1631 (2)	5491 (10)	82
C(34)	679 (16)	1403 (3)	5862 (10)	81
C(35)	91 (15)	1071 (3)	5139 (10)	83
C(36)	813 (15)	977 (2)	4022 (10)	74

Table 2. Bond distances (\AA), bond angles and torsion angles ($^\circ$) with *e.s.d.*'s for C₂₆H₂₈O₂Sn

Sn—C(8)	2.166 (9)	C(1)—C(7)	1.533 (12)
Sn—C(11)	2.150 (8)	C(2)—C(3)	1.533 (14)
Sn—C(21)	2.127 (8)	C(3)—C(6)	1.509 (14)
Sn—C(31)	2.122 (8)	C(4)—C(5)	1.456 (14)
O(1)—C(1)	1.364 (13)	C(5)—C(6)	1.535 (14)
O(2)—C(3)	1.408 (15)	C(5)—C(8)	1.545 (11)
O(2)—C(4)	1.468 (11)	C(6)—C(7)	1.593 (13)
C(1)—C(2)	1.472 (16)		
C(8)—Sn—C(11)	115.7 (3)	O(2)—C(3)—C(6)	105.5 (8)
C(8)—Sn—C(21)	104.3 (3)	C(2)—C(3)—C(6)	106.6 (9)
C(8)—Sn—C(31)	112.0 (3)	O(2)—C(4)—C(5)	105.4 (8)
C(11)—Sn—C(21)	107.6 (3)	C(4)—C(5)—C(6)	100.6 (8)
C(11)—Sn—C(31)	108.0 (3)	C(4)—C(5)—C(8)	115.4 (8)
C(21)—Sn—C(31)	109.0 (3)	C(6)—C(5)—C(8)	114.4 (7)
C(3)—O(2)—C(4)	109.4 (8)	C(3)—C(6)—C(5)	104.1 (8)
O(1)—C(1)—C(2)	114.1 (9)	C(3)—C(6)—C(7)	103.6 (8)
O(1)—C(1)—C(7)	116.1 (9)	C(5)—C(6)—C(7)	113.4 (8)
C(2)—C(1)—C(7)	104.9 (8)	C(1)—C(7)—C(6)	101.8 (7)
C(1)—C(2)—C(3)	108.1 (9)	Sn—C(8)—C(5)	118.6 (6)
O(2)—C(3)—C(2)	110.0 (9)		
Ring A		Ring B	
C(1)—C(2)—C(3)—C(6)	3.6 (11)	C(3)—O(2)—C(4)—C(5)	-25.2 (10)
C(2)—C(3)—C(6)—C(7)	19.9 (10)	O(2)—C(4)—C(5)—C(6)	37.0 (9)
C(3)—C(6)—C(7)—C(1)	-35.4 (9)	C(4)—C(5)—C(6)—C(3)	-36.1 (9)
C(6)—C(7)—C(1)—C(2)	38.0 (9)	C(5)—C(6)—C(3)—O(2)	21.8 (10)
C(7)—C(1)—C(2)—C(3)	-26.7 (11)	C(6)—C(3)—O(2)—C(4)	1.2 (11)
Inter-rings		Substituents	
C(1)—C(2)—C(3)—O(2)	117.5 (9)	C(8)—C(5)—C(4)—O(2)	160.6 (7)
C(1)—C(7)—C(6)—C(5)	-147.5 (8)	C(8)—C(5)—C(6)—C(3)	-160.5 (8)
C(2)—C(3)—O(2)—C(4)	-113.4 (9)	C(8)—C(5)—C(6)—C(7)	-48.6 (10)
C(7)—C(6)—C(5)—C(4)	75.8 (9)	O(1)—C(1)—C(2)—C(3)	-154.8 (9)
		O(1)—C(1)—C(7)—C(6)	164.8 (9)

Experimental. The reaction shown below gave a major product which after deprotection led to nicely crystalline alcohol in 92% yield (2 steps). The triphenyltin adduct was subjected to X-ray analysis.



Crystal of C₂₆H₂₈O₂Sn, with dimensions 0.040 \times 0.085 \times 0.40 mm, was obtained from hexanes. Unit-cell dimensions from 25 centered reflections in the range $20 < \theta < 25^\circ$. Enraf-Nonius CAD-4 diffractometer using graphite-monochromatized Cu *K* α radiation, $2\theta_{max} = 140.0^\circ$. The data were collected in the ω -scan mode, $\Delta\omega = (1.00 + 0.14\tan\theta)^\circ$. The orientation was checked every 200 measurements. The intensities of seven reflections (checked every hour) presented a maximum fluctuation of 6.2%. 4207 measured reflections of which 2569 had $I > 3\sigma(I)$. h 0– \pm 7, k 0–44, l 0–12.

The structure was solved using *MULTAN80*.^{*} Block-diagonal least-squares refinement based on $|F_o|$, all non-H atoms anisotropic, H-atom coordinates were fixed at idealized positions ($d_{C-H} = 1.08 \text{ \AA}$), with $U_{iso} = 0.10 \text{ \AA}^2$ (phenyl) and $U_{iso} = 0.15 \text{ \AA}^2$ (others), for H atoms involved in hydrogen bonding, coordinates refined and U_{iso} fixed. Final $R = 0.055$, $wR = 0.050$ {weights derived from the counting statistics, $w = 1/[\sigma^2(F) + 0.00005F^2]}$, $S = 2.35$, (shift/ σ) mean 0.016, max. 0.21. The residual

^{*} The programs used here are modified versions of: *NRC-2*, data reduction, *NRC-10*, bond distances and angles and *NRC-22*, mean planes (Ahmed, Hall, Pippy & Huber, 1973); *MULTAN80*, multiresolution program (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); and *ORTEP* (Johnson, 1965).

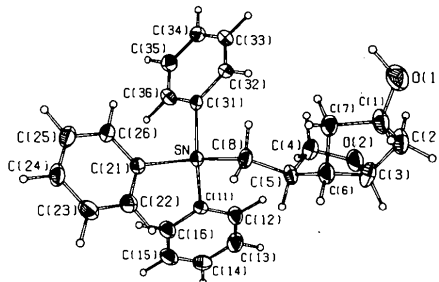


Fig. 1. An *ORTEP* diagram of the title compound with the H atoms. The atomic numbering is shown. Ellipsoids represent 50% probability.

electron density fluctuation on final difference Fourier synthesis gave two peaks of $0.65\text{--}0.55\text{ e \AA}^{-3}$, respectively, at $1.1\text{--}1.2\text{ \AA}$ from Sn, with background of $\pm 0.45\text{ e \AA}^{-3}$.

The scattering factors were taken from Cromer & Mann (1968) for Sn, O and C, and from Stewart, Davidson & Simpson (1965) for H.

The final coordinates for the non-H atoms are given in Table 1, selected bond distances and angles in Table 2.* Fig. 1 shows a thermal-ellipsoid plot with the atom numbering.

Related literature. For the preparation of some related compounds *via* a tin-mediated radical cycli-

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, distances and angles related to the phenyl groups, to the weighted least-squares planes and to H bonding, and a stereoview of the unit cell have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52288 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 1,3-Dinitro-1,3-diazacyclopentan-2-one

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Abstract. $\text{C}_3\text{H}_4\text{N}_4\text{O}_5$, $M_r = 176.08$, orthorhombic, $P2_12_12_1$, $a = 5.912(1)$, $b = 7.928(2)$, $c = 13.951(2)\text{ \AA}$, $V = 653.9(2)\text{ \AA}^3$, $Z = 4$, $D_x = 1.789\text{ Mg m}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71073\text{ \AA}$, $\mu = 0.16\text{ mm}^{-1}$, $F(000) = 360$, $T = 295\text{ K}$, final $R = 0.036$, $wR = 0.046$ for 653 observed reflections. Excluding H atoms the molecule is approximately planar to within $\pm 0.27\text{ \AA}$. Even without the possibility of hydrogen bonding there are some very close intermolecular $\text{O}\cdots\text{N}$ approaches ($2.91\text{--}2.96\text{ \AA}$).

Experimental. Colorless, $0.36 \times 0.28 \times 0.20\text{ mm}$ data crystal, synthesized and crystallized by Clifford Coon of the Lawrence Livermore Laboratory, Livermore, California. Automated Nicolet *R3m* diffractometer with incident-beam graphite monochromator; 25 centered reflections within $18 \leq 2\theta \leq 30^\circ$ used for determining cell parameters. Data corrected for Lorentz and polarization effects, but not for absorption. $2\theta_{\text{max}} = 50^\circ$; range of hkl : $-6 \leq h \leq 7$, $0 \leq k \leq 9$, $0 \leq l \leq 16$, standards, 400, 060, 0.0.10, monitored every

zation reaction, see also Fish, Kuivila & Tyminsky (1967).

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100 reflections with random variation of 2.0% over data collection, $\theta/2\theta$ mode, scan width $[2\theta(K\alpha_1) - 1.0]$ to $[2\theta(K\alpha_2) + 1.0]^\circ$, scan rate a function of count rate (8° min^{-1} minimum, 30° min^{-1} maximum); 1366 reflections measured, 700 unique, $R_{\text{int}} = 0.015$, 653 observed [$F_o > 3\sigma(F_o)$].

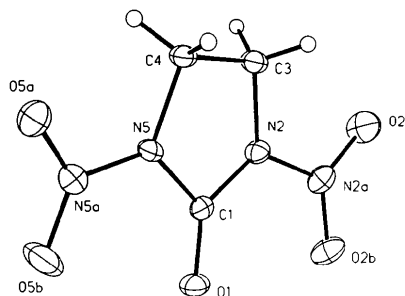


Fig. 1. Perspective drawing of the results of the X-ray study on the cyclopentane. Thermal ellipsoids are shown at the 20% probability level.