

contributions were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = 4F_o/\sigma^2(I)$ with $\sigma^2(I) = [\sigma^2(I_c) + (pF_o)^2]$; final $R = 0.035$, $wR = 0.045$ ($w = 1$), $S = 3.0$, $(\Delta/\sigma)_{\max} = 0.10$; highest peak in the final difference Fourier map = $0.70 \text{ e } \text{Å}^{-3}$.

Final positional parameters are given in Table 1,* with selected bond distances and angles in Table 2.

* Lists of structure factors, anisotropic thermal parameters, and bond angles and distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55326 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU0328]

The atomic labelling scheme and molecular structure are shown in Fig. 1.

Related literature. This work is part of structural studies of uranyl with poorly coordinating anions in various solvents (Alcock & Esperas, 1977).

References

- ALCOCK, N. W. & ESPERAS, S. (1977). *J. Chem. Soc. Dalton Trans.* pp. 893–896.
 FRENZ, B. A. (1985). *Enraf-Nonius SDP-Plus Structure Determination Package*. Version 3.0. Enraf-Nonius, Delft, The Netherlands.
 JOHNSON, C. K. (1976). *ORTEP*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 WALKER, N. & STUART, D. (1983). *Acta Cryst.* **A39**, 158–166.

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Structure of the Dibenzyl Sulfoxide Adduct of *N*-Triphenylstannyl-1,2-benzisothiazol-3(2*H*)-one 1,1-Dioxide

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Abstract. [1,2-Benzisothiazol-3(2*H*)-one 1,1-dioxide-*N*](dibenzyl sulfoxide-*O*)triphenyltin(IV), $[\text{Sn}(\text{C}_{14}\text{H}_{14}\text{OS})(\text{C}_7\text{H}_4\text{NO}_2\text{S})(\text{C}_6\text{H}_5)_3]$, $[(\text{C}_6\text{H}_5)_3\text{SnNC}(\text{O})\text{C}_6\text{H}_4\text{SO}_2.\text{OS}(\text{CH}_2\text{C}_6\text{H}_5)_2]$, $M_r = 762.50$, monoclinic, $P2_1$, $a = 10.9026$ (7) Å , $b = 19.614$ (1) Å , $c = 16.8211$ (9) Å , $\beta = 94.512$ (5)°, $V = 3585.8$ (8) Å^3 , $Z = 4$, $D_x = 1.412 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71073 \text{ Å}$, $\mu = 8.64 \text{ cm}^{-1}$, $F(000) = 1552$, $T = 298 \text{ K}$, $R = 0.038$ for 4729 reflections [$I \geq 3\sigma(I)$]. The Sn atoms in the two independent molecules exhibit a *trans*- C_3SnNO trigonal bipyramidal geometry. In molecule *A*, the Sn—N bond [2.237 (8) Å] is short and the Sn—O bond [2.413 (7) Å] long; in molecule *B*, the Sn—N bond [2.253 (9) Å] is longer whereas the Sn—O bond [2.376 (7) Å] is shorter.

Experimental. The adduct was prepared by reacting triphenyltin hydroxide, saccharin and dibenzyl sulfoxide in 1/1/1 stoichiometric ratio in ethanol (Ng, Kuthubutheen, Zainudin, Chen, Kumar Das, Schulze, Molloy, Yip & Mak, 1991). A crystal of triphenyltin saccharin–dibenzyl sulfoxide measuring approximately $0.14 \times 0.22 \times 0.22 \text{ mm}$ was mounted

on an Enraf–Nonius CAD-4 diffractometer. Unit-cell parameters were fixed from 25 strong reflections in the $16 \leq \theta \leq 18^\circ$ thin shell. Intensity data were collected to $2\theta_{\max} = 50^\circ$ (collection range: h 0–12, k 0–21, l –19–19). 6251 reflections were collected, of which 4729 obeyed the $I \geq 3\sigma(I)$ criterion. Three reflections (0,16,0, 5,3,11 and 905) were used to monitor the intensity. Their intensities decreased by 5.7% over the 75 h of data collection, and a correction was applied to the data (average correction factor 1.02962). The two Sn atoms were obtained by direct methods, and the y coordinate of Sn1 was fixed at 0.25. The non-H atoms were derived from successive difference Fourier syntheses, and all the non-H atoms were refined anisotropically. H atoms were generated geometrically ($\text{C—H} = 0.95 \text{ Å}$, $B = 5 \text{ Å}^2$) and were included in the structure-factor calculations. Refinement based on F converged at $R = 0.038$ ($R_2 = 0.043$ for all reflections); 846 variables were refined, and unit weights were used; $\Delta/\sigma = 0.04$; $S = 2.353$; $(\Delta\rho)_{\max} = 0.41$ (6) $\text{e } \text{Å}^{-3}$. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B

Table 1. Positional parameters and equivalent isotropic displacement parameters (Å²)

$$B_{eq} = (4/3)[a^2 B_{1,1} + b^2 B_{2,2} + c^2 B_{3,3} + ab(\cos\gamma)B_{1,2} + ac(\cos\beta)B_{1,3} + bc(\cos\alpha)B_{2,3}]$$

	x	y	z	B _{eq}
Sn1A	0.21344 (6)	0.25	0.29981 (4)	3.13 (1)
S2A	0.0611 (3)	0.1138 (1)	0.1959 (2)	3.59 (5)
S3A	0.3494 (2)	0.3004 (1)	0.4790 (2)	3.18 (5)
O4A	0.1280 (9)	0.0593 (5)	0.2333 (5)	5.8 (2)
O5A	-0.0592 (7)	0.1284 (5)	0.2219 (5)	4.8 (2)
O6A	0.2372 (6)	0.2541 (4)	0.1117 (4)	4.1 (1)
O7A	0.2785 (6)	0.3289 (4)	0.4048 (4)	3.6 (2)
N8A	0.1457 (7)	0.1833 (4)	0.1978 (5)	3.2 (2)
C9A	0.1726 (8)	0.2045 (5)	0.1226 (6)	2.9 (2)
C10A	0.1128 (9)	0.1583 (5)	0.0592 (6)	3.0 (2)
C11A	0.113 (1)	0.1664 (7)	-0.0229 (6)	4.6 (3)
C12A	0.045 (1)	0.1194 (7)	-0.0711 (7)	5.3 (3)
C13A	-0.014 (1)	0.0670 (7)	-0.0386 (7)	5.4 (3)
C14A	-0.014 (1)	0.0571 (6)	0.0451 (8)	4.7 (3)
C15A	0.0518 (9)	0.1052 (5)	0.0913 (6)	3.4 (2)
C16A	0.101 (1)	0.3328 (6)	0.2549 (6)	3.7 (2)
C17A	0.146 (1)	0.3996 (6)	0.2551 (6)	4.4 (3)
C18A	0.071 (10)	0.4523 (7)	0.2219 (8)	6.4 (3)
C19A	-0.050 (1)	0.4387 (7)	0.1935 (8)	5.6 (3)
C20A	-0.092 (1)	0.3735 (7)	0.1916 (7)	4.6 (3)
C21A	-0.0181 (9)	0.3194 (7)	0.2241 (6)	4.0 (2)
C22A	0.142 (1)	0.1880 (6)	0.3889 (6)	3.9 (2)
C23A	0.021 (1)	0.1988 (7)	0.4116 (7)	4.9 (3)
C24A	-0.022 (1)	0.1608 (8)	0.4756 (8)	6.7 (3)
C25A	0.053 (1)	0.1156 (8)	0.5159 (8)	7.2 (4)
C26A	0.169 (2)	0.1037 (7)	0.4931 (8)	7.1 (4)
C27A	0.216 (1)	0.1388 (7)	0.4284 (8)	5.6 (3)
C28A	0.4006 (9)	0.2278 (6)	0.2825 (6)	3.8 (3)
C29A	0.492 (1)	0.2773 (6)	0.2893 (6)	5.2 (3)
C30A	0.616 (1)	0.2610 (9)	0.2754 (7)	6.1 (4)
C31A	0.643 (1)	0.195 (1)	0.2561 (8)	7.6 (4)
C32A	0.554 (1)	0.1433 (9)	0.250 (1)	6.8 (4)
C33A	0.432 (1)	0.1612 (7)	0.2645 (8)	5.1 (3)
C34A	0.4438 (9)	0.3715 (6)	0.5205 (6)	3.6 (2)
C35A	0.549 (1)	0.3837 (6)	0.4678 (6)	3.9 (2)
C36A	0.653 (1)	0.3444 (9)	0.4800 (8)	6.2 (4)
C37A	0.754 (1)	0.356 (1)	0.435 (1)	8.6 (5)
C38A	0.748 (2)	0.411 (1)	0.379 (1)	10.4 (5)
C39A	0.643 (2)	0.4504 (9)	0.368 (1)	9.2 (5)
C40A	0.543 (1)	0.4373 (7)	0.4136 (8)	5.8 (3)
C41A	0.240 (1)	0.2947 (6)	0.5562 (6)	4.0 (2)
C42A	0.178 (1)	0.3604 (5)	0.5768 (6)	3.4 (2)
C43A	0.224 (1)	0.3984 (6)	0.6441 (7)	5.0 (3)
C44A	0.161 (1)	0.4565 (7)	0.6663 (7)	5.3 (3)
C45A	0.051 (1)	0.4755 (7)	0.6222 (7)	6.0 (3)
C46A	0.008 (1)	0.4381 (7)	0.5571 (9)	5.6 (3)
C47A	0.071 (1)	0.3808 (6)	0.5331 (8)	4.8 (3)
Sn1B	0.51082 (5)	0.24171 (5)	0.80724 (3)	2.68 (1)
S2B	0.6811 (2)	0.1059 (2)	0.7293 (2)	3.61 (6)
S3B	0.3776 (2)	0.3026 (1)	0.9747 (2)	2.95 (5)
O4B	0.6217 (8)	0.0510 (4)	0.7660 (5)	5.2 (2)
O5B	0.7976 (7)	0.1260 (5)	0.7639 (5)	5.7 (2)
O6B	0.4887 (6)	0.2319 (4)	0.6161 (4)	4.3 (2)
O7B	0.4309 (6)	0.3208 (4)	0.8969 (4)	3.5 (1)
N8B	0.5888 (7)	0.1709 (5)	0.7186 (5)	3.3 (2)
C9B	0.555 (1)	0.1840 (6)	0.6390 (6)	3.4 (2)
C10B	0.6128 (9)	0.1366 (5)	0.5845 (6)	3.2 (2)
C11B	0.596 (1)	0.1353 (7)	0.5003 (6)	4.4 (3)
C12B	0.664 (1)	0.0845 (7)	0.4642 (8)	6.1 (3)
C13B	0.739 (1)	0.0367 (7)	0.5075 (8)	5.1 (3)
C14B	0.753 (1)	0.0397 (7)	0.5899 (8)	5.1 (3)
C15B	0.687 (1)	0.0904 (6)	0.6260 (6)	3.9 (2)
C16B	0.3267 (9)	0.2083 (5)	0.7730 (5)	3.0 (2)
C17B	0.2289 (8)	0.2512 (8)	0.7773 (6)	4.3 (2)
C18B	0.108 (1)	0.2306 (8)	0.7521 (6)	5.2 (3)
C19B	0.092 (1)	0.1647 (9)	0.7230 (7)	5.7 (3)
C20B	0.190 (1)	0.1197 (7)	0.7224 (7)	5.2 (3)
C21B	0.309 (1)	0.1430 (6)	0.7468 (7)	4.1 (2)
C22B	0.597 (1)	0.3287 (6)	0.7601 (6)	4.1 (2)
C23B	0.707 (1)	0.3180 (7)	0.7213 (7)	4.9 (3)
C24B	0.764 (1)	0.3721 (8)	0.6850 (7)	7.0 (3)
C25B	0.713 (1)	0.4364 (8)	0.6911 (8)	7.8 (4)
C26B	0.608 (2)	0.4481 (7)	0.728 (1)	7.3 (4)
C27B	0.549 (1)	0.3934 (6)	0.7645 (8)	5.2 (3)
C28B	0.603 (1)	0.1937 (5)	0.9095 (6)	3.5 (2)
C29B	0.720 (1)	0.2140 (6)	0.9364 (6)	4.1 (2)
C30B	0.772 (1)	0.1875 (8)	1.0099 (7)	5.4 (3)
C31B	0.703 (1)	0.1427 (8)	1.0521 (8)	6.6 (3)
C32B	0.590 (1)	0.1208 (8)	1.0253 (9)	6.5 (4)
C33B	0.533 (1)	0.1472 (6)	0.9514 (7)	4.5 (3)

Table 1 (cont.)

	x	y	z	B _{eq}
C34B	0.5040 (9)	0.3087 (6)	1.0536 (6)	3.6 (2)
C35B	0.5620 (9)	0.3771 (6)	1.0573 (7)	3.8 (2)
C36B	0.544 (1)	0.4194 (7)	1.1217 (8)	5.1 (3)
C37B	0.602 (1)	0.4844 (7)	1.1266 (9)	7.0 (4)
C38B	0.676 (1)	0.5046 (8)	1.068 (1)	7.1 (4)
C39B	0.692 (1)	0.4643 (8)	1.005 (1)	6.8 (4)
C40B	0.638 (1)	0.3971 (7)	0.9999 (8)	4.9 (3)
C41B	0.2924 (9)	0.3805 (5)	1.0008 (6)	3.6 (2)
C42B	0.1661 (8)	0.3811 (5)	0.9587 (6)	3.0 (2)
C43B	0.069 (1)	0.3605 (6)	1.0009 (7)	4.2 (3)
C44B	-0.052 (1)	0.3613 (7)	0.9636 (7)	4.8 (3)
C45B	-0.071 (1)	0.3797 (7)	0.8863 (7)	4.8 (3)
C46B	0.026 (1)	0.4010 (7)	0.8435 (7)	4.6 (3)
C47B	0.145 (1)	0.4012 (6)	0.8803 (6)	3.9 (2)

Table 2. Selected bond distances (Å) and angles (°)

	Molecule A	Molecule B
Sn1—O7	2.413 (7)	2.376 (7)
Sn1—N8	2.237 (8)	2.253 (9)
Sn1—C16	2.14 (1)	2.15 (1)
Sn1—C22	2.13 (1)	2.13 (1)
Sn1—C28	2.13 (1)	2.14 (1)
S2—O4	1.414 (9)	1.422 (9)
S2—O5	1.443 (8)	1.411 (8)
S2—N8	1.645 (9)	1.626 (9)
S2—C15	1.76 (1)	1.77 (1)
S3—O7	1.522 (7)	1.517 (7)
S3—C34	1.84 (1)	1.84 (1)
S3—C41	1.83 (1)	1.86 (1)
O6—C9	1.22 (1)	1.23 (1)
N8—C9	1.38 (1)	1.38 (1)
C9—C10	1.51 (1)	1.48 (1)
O7—Sn1—N8	175.7 (3)	177.3 (3)
O7—Sn1—C16	84.2 (3)	89.3 (3)
O7—Sn1—C22	87.3 (3)	84.8 (3)
O7—Sn1—C28	90.1 (3)	87.1 (3)
N8—Sn1—C16	91.6 (4)	91.6 (3)
N8—Sn1—C22	95.1 (4)	92.6 (3)
N8—Sn1—C28	92.1 (3)	94.8 (4)
C16—Sn1—C22	116.8 (4)	125.2 (4)
C16—Sn1—C28	129.6 (4)	116.8 (4)
C22—Sn1—C28	112.8 (4)	117.3 (4)
O4—S2—O5	117.7 (5)	117.7 (2)
O4—S2—N8	110.4 (5)	110.1 (5)
O4—S2—C15	111.1 (5)	110.5 (5)
O5—S2—N8	110.6 (5)	110.8 (5)
O5—S2—C15	109.8 (5)	110.5 (5)
N8—S2—C15	94.9 (5)	95.5 (5)
O7—S3—C34	105.4 (5)	106.9 (4)
O7—S3—C41	106.7 (4)	104.0 (5)
C34—S3—C41	98.8 (5)	97.8 (5)
Sn1—O7—S3	117.8 (4)	125.3 (4)
Sn1—N8—S2	130.5 (5)	132.3 (4)
Sn1—N8—C9	116.4 (6)	116.1 (7)
S2—N8—C9	113.0 (7)	111.6 (7)
O6—C9—N8	122.8 (9)	123.4 (9)
O6—C9—C10	126.4 (9)	123.6 (9)
N8—C9—C10	110.8 (8)	112.9 (9)
C9—C10—C11	127 (1)	127.2 (9)
C9—C10—C15	112.0 (9)	111.4 (9)
S2—C15—C10	109.3 (7)	108.6 (8)
S2—C15—C14	127.7 (9)	127.6 (9)

and 2.3.1). All computations were performed using the *MolEN* structure determination package (Fair, 1990) on a DEC MicroVAX minicomputer. The atomic coordinates are listed in Table 1,* one of the

* Lists of structure factors, anisotropic thermal parameters, calculated H-atom positional parameters, and complete bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55302 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0550]

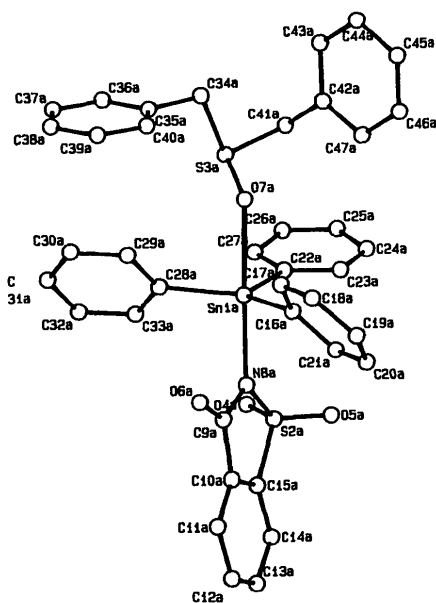


Fig. 1. One of the two crystallographically independent molecules of triphenyltin saccharin-dibenzyl sulfoxide.

two molecules (molecule *A*) is shown in Fig. 1. Selected bond distances and angles are listed in Table 2. The absolute structure was not determined.

Related literature. The Sn—N bond distances are somewhat longer and the Sn—O distances somewhat shorter than those [Sn—N 2.240 (7) – 2.242 (8); Sn—O 2.394 (8) – 2.409 (7) Å (Ng, Chen, Kumar Das & Mak, 1989*a,b*)] found in the ethanol, glycolic acid and dimethylformamide adducts. The Sn—O bonds in the dibenzyl sulfoxide adduct are longer than the Sn—O bond [2.319 (10) Å] found in the *cis* trigonal bipyramidal 1/1 adduct with dimethyltin dichloride (Ng & Rheingold, 1989).

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References

- FAIR, C. K. (1990). *MolEN Structure Determination System*. Delft Instruments, X-ray Diffraction BV, Röntgenweg 1, 2624 BD Delft, The Netherlands.
- NG, S. W., CHEN, W., KUMAR DAS, V. G. & MAK, T. C. W. (1989*a*). *J. Organomet. Chem.* **376**, 277–281.
- NG, S. W., CHEN, W., KUMAR DAS, V. G. & MAK, T. C. W. (1989*b*). *J. Organomet. Chem.* **379**, 247–249.
- NG, S. W., KUTHUBUTHEEN, A. J., ZAINUDIN, A., CHEN, W., KUMAR DAS, V. G., SCHULZE, B., MOLLOY, K. C., YIP, W.-H. & MAK, T. C. W. (1991). *J. Organomet. Chem.* **403**, 101–109.
- NG, S. W. & RHEINGOLD, A. L. (1989). *J. Organomet. Chem.* **378**, 339–345.

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Structure of *trans*-Dicyanobis(triphenylphosphine)platinum(II) Dimethanol Solvate

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Abstract. [Pt(CN)₂(C₁₈H₁₅P)₂].2CH₄O, *M_r* = 835.790, triclinic, *P* $\bar{1}$, *a* = 7.736 (1), *b* = 10.406 (2), *c* = 12.111 (3) Å, α = 111.00 (1), β = 104.07 (1), γ = 90.65 (1)°, *V* = 877.7 (3) Å³, *Z* = 1, *D_x* = 1.581 g cm⁻³, λ (Mo *K* α) = 0.71073 Å, μ = 41.63 cm⁻¹, *F*(000) = 416, *T* = 233 K, final conventional *R* = 0.0174 and *wR* = 0.0177, for 214 variable parameters and 2303 reflections with $|F_o|^2 > 3\sigma|F_o|^2$. The structure consists of a discrete monomeric four-coordinated square-planar Pt^{II} center with *trans* cyanide groups. The Pt—C distance is 1.991 (4) Å and the Pt—P distance is 2.332 (1) Å.

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Experimental. [Pt(CN)₂(PPh₃)₂] was synthesized according to the procedure of Bailar & Itatani (1967). IR ν (CN) 2130 cm⁻¹. Colorless crystals were obtained from a warm solution of C₆H₆ and CH₃OH which was allowed to cool slowly to room temperature. The compound crystallizes with solvent (CH₃OH) in the lattice. A multifaceted crystal of approximate dimensions 0.4 × 0.20 × 0.10 mm was mounted in a random orientation on a glass fiber. Data were collected at 233 K by using the ω -scan technique in bisecting geometry on a Nicolet R3m/E diffractometer, with graphite-monochromated Mo *K* α radiation. The low-temperature device used has not been previously reported and therefore we