groups (two singlets). {Ti[C$_5$H$_4$Si(CH$_3$)$_3$]$_2$Cl$_2$ shows only one singlet for the 18 methyl protons (Köpf & Klouras, 1982).}

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3,3-Di-*tert*-butyl-*N*,4,5,6,7-pentaphenyl-1,2-dihydro-1,2,3-diazastannepine-1,2-dicarboximide Monohydrate, C$_{44}$H$_{43}$N$_3$O$_2$Sn.H$_2$O

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Abstract. $M_r = 782 \cdot 6$, monoclinic, $C2/c$, $a = 22 \cdot 838$ (7), $b = 14 \cdot 526$ (5), $c = 26 \cdot 270$ (9) Å, $\beta = 115 \cdot 32$ (5)°, $U = 7878$ Å^3, $Z = 8$, $D_x = 1 \cdot 320$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0 \cdot 71069$ Å, $\mu = 0 \cdot 70$ mm^{-1}, $F(000) = 3232$, $T = 294$ (1) K. Final $R = 0 \cdot 057$ for 2500 unique observed diffractometer data. The central part of the asymmetric monomeric molecule is a seven-membered heterocycle consisting of a tetrahedrally coordinated Sn, two N and four C atoms. Two *tert*-butyl groups and four phenyl groups are bound to this ring. The two N atoms are part of an annellated *N*-phenylurazole ring.

Introduction. The ^{1}H NMR and ^{13}C NMR spectra of the 1:1 adduct of 1,1-di-*tert*-butyl-2,3,4,5-tetraphenyl-1-stanna-2,4-cyclopentadiene with 4-phenyl-3,4-di-hydro-1,2,4-triazole-3,5-dione indicated a 7-stanna-8,9,10-trinorbornene structure because of the existence of two non-equivalent *tert*-butyl groups. The high stability of the title compound (m.p. 498 K without decomposition), however, could not be explained in comparison with similar compounds such as the dimethyl- and diisopropylstanna analogues which decompose instantaneously with the elimination of the dialkylstannylene. Only the stanna-8,9,10-trinorbornene resulting from tetracyanoethylene and the 1,1-dimethyl-2,3,4,5-tetraphenylstannole can be isolated at 243 K (Grugel, Neumann & Schriewer, 1979). IR and mass spectrometry did not give clear information about the structure of the title compound.

Therefore an X-ray structure analysis has been undertaken.

Experimental. Colourless crystals obtained by recrystallization from methylene chloride/diethyl ether; $\omega/2\theta$ scan, scan speed 0·83–5° min^{-1} in θ, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$, crystal 0·12 × 0·20 × 0·30 mm, lattice parameters from least-squares fit with 25 reflexions in the range $6 \cdot 9 \le \theta \le 12 \cdot 1°$; three standard reflexions recorded every 3 h showed no decrease; 4027 reflexions measured, $2 \le \theta \le 20°$, $h -22 \rightarrow +22$, $k\ 0 \rightarrow +14$, $l\ 0 \rightarrow +25$; Lorentz–polarization and absorption correction *via* ψ scans, max./min. transmission 1·00/0·96; systematic absences (hkl) $h+k = 2n+1$, ($h0l$) $l = 2n+1$, possible space groups $C2/c$ and Cc; structure solution in space group $C2/c$ *via* Patterson function, ΔF syntheses and full-matrix least squares with 2500 observed reflexions [$|F_o| > 3\sigma(F_o)$], anisotropic temperature factor for Sn and isotropic for O, N, C, H; H atoms placed in geometrically calculated positions (C–H 0·95 Å) before last refinement cycles, not refined ($B = 5 \cdot 0$ Å^2); a ΔF synthesis with all non-H atoms of the molecule revealed two peaks which were interpreted as a split O atom of a water molecule [occupancy factor for O(3) 0·67 and for O(4) 0·33]; complex neutral-atom scattering factors from Cromer & Waber (1974) and Cromer (1974) (H: SDS, C: RHF), least-squares

refinement based on $|F|$ and 214 refined parameters, $R = 0.057$; unit weights, confirmed by analysis of variance of the mean $w\Delta^2$ with F_o, $(\Delta/\sigma)_{max} < 0.005$, no significant features in a final ΔF synthesis; programs: Enraf–Nonius SDP (Frenz, 1981), $ORTEP$II (Johnson, 1976), POP1 (van de Waal, 1976).

Discussion. The structure of the title compound is shown in Fig. 1 ($ORTEP$) and in a stereoview in Fig. 2 (POP1). Positional parameters and the equivalent value of the anisotropic temperature coefficients β_{ij} of the Sn atom and isotropic parameters of the other non-H atoms are given in Table 1. Bond lengths and angles are given in Table 2.*

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a complete list of bond lengths and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39572 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
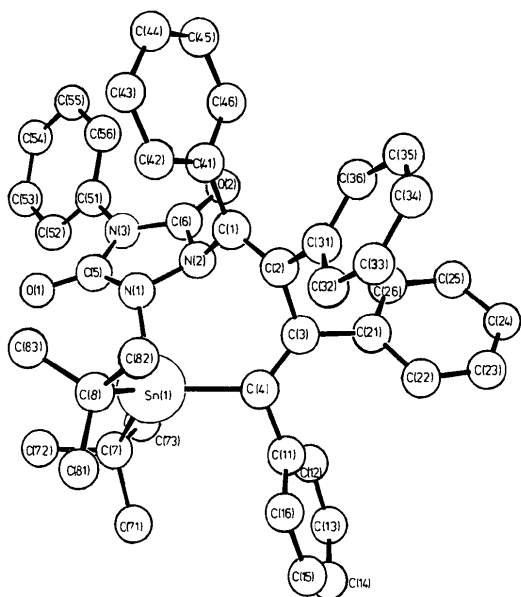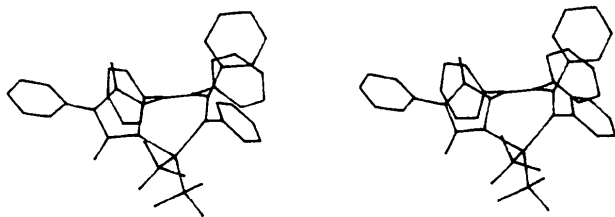
Table 1. *Fractional atomic coordinates and equivalent isotropic or isotropic temperature factors* ($\mathring{A}^2$) *with e.s.d.'s in parentheses*

	x	y	z	B_{eq}/B
Sn(1)	0·34832 (4)	0·07032 (6)	0·31858 (3)	2·99 (2)*
O(1)	0·3618 (4)	0·3141 (6)	0·3188 (3)	4·6 (2)
O(2)	0·3287 (4)	0·2656 (6)	0·4764 (3)	4·3 (2)
N(1)	0·3213 (4)	0·1910 (6)	0·3486 (3)	3·0 (2)
N(2)	0·3142 (4)	0·1748 (6)	0·3999 (3)	3·1 (2)
N(3)	0·3528 (4)	0·3160 (6)	0·4037 (3)	3·0 (2)
C(1)	0·2516 (5)	0·1333 (8)	0·3875 (4)	2·8 (2)
C(2)	0·2505 (5)	0·0419 (8)	0·3909 (4)	2·8 (2)
C(3)	0·3079 (5)	−0·0191 (8)	0·4081 (4)	3·1 (2)
C(4)	0·3452 (5)	−0·0262 (8)	0·3807 (4)	2·9 (2)
C(5)	0·3469 (5)	0·2764 (9)	0·3534 (4)	3·5 (3)
C(6)	0·3314 (5)	0·2536 (8)	0·4315 (4)	2·9 (2)
C(7)	0·4487 (5)	0·0966 (8)	0·3339 (5)	3·6 (3)
C(8)	0·2780 (6)	0·059 (1)	0·2317 (5)	4·7 (3)
C(11)	0·3980 (5)	−0·0960 (8)	0·3972 (4)	2·9 (2)
C(12)	0·4514 (5)	−0·0936 (8)	0·4477 (5)	3·7 (3)
C(13)	0·5016 (6)	−0·157 (1)	0·4606 (5)	5·2 (3)
C(14)	0·4975 (6)	−0·224 (1)	0·4231 (5)	5·1 (3)
C(15)	0·4447 (6)	−0·229 (1)	0·3738 (5)	5·3 (3)
C(16)	0·3948 (5)	−0·1661 (9)	0·3595 (5)	4·1 (3)
C(21)	0·3171 (5)	−0·0713 (9)	0·4603 (4)	3·1 (2)
C(22)	0·3198 (6)	−0·1663 (9)	0·4635 (5)	4·2 (3)
C(23)	0·3267 (6)	−0·208 (1)	0·5131 (5)	5·2 (3)
C(24)	0·3311 (6)	−0·160 (1)	0·5582 (5)	4·8 (3)
C(25)	0·3288 (6)	−0·066 (1)	0·5551 (5)	4·6 (3)
C(26)	0·3212 (5)	−0·0222 (9)	0·5057 (5)	3·9 (3)
C(31)	0·1893 (5)	−0·0103 (9)	0·3789 (5)	3·6 (3)
C(32)	0·1717 (6)	−0·081 (1)	0·3415 (5)	5·3 (3)
C(33)	0·1149 (8)	−0·132 (1)	0·3306 (7)	7·6 (4)
C(34)	0·0798 (7)	−0·110 (1)	0·3600 (6)	7·1 (4)
C(35)	0·0967 (7)	−0·043 (1)	0·3974 (6)	6·5 (4)
C(36)	0·1526 (6)	0·010 (1)	0·4069 (5)	4·7 (3)
C(41)	0·1961 (5)	0·1965 (8)	0·3678 (4)	2·8 (2)
C(42)	0·1606 (6)	0·2119 (9)	0·3120 (5)	4·4 (3)
C(43)	0·1092 (7)	0·275 (1)	0·2933 (6)	6·4 (4)
C(44)	0·0930 (6)	0·320 (1)	0·3307 (6)	5·6 (3)
C(45)	0·1282 (6)	0·308 (1)	0·3860 (5)	5·0 (3)
C(46)	0·1797 (6)	0·247 (1)	0·4049 (5)	4·8 (3)
C(51)	0·3722 (5)	0·4077 (8)	0·4214 (4)	3·2 (2)
C(52)	0·4247 (6)	0·444 (1)	0·4169 (5)	5·1 (3)
C(53)	0·4410 (6)	0·536 (1)	0·4329 (6)	5·6 (3)
C(54)	0·4045 (7)	0·587 (1)	0·4523 (6)	6·4 (4)
C(55)	0·3522 (6)	0·551 (1)	0·4562 (5)	5·6 (3)
C(56)	0·3359 (6)	0·4597 (9)	0·4409 (5)	4·3 (3)
C(71)	0·4811 (6)	0·009 (1)	0·3265 (5)	5·1 (3)
C(72)	0·4505 (6)	0·166 (1)	0·2918 (5)	4·8 (3)
C(73)	0·4841 (6)	0·133 (1)	0·3926 (5)	5·0 (3)
C(81)	0·3045 (9)	0·001 (1)	0·2006 (8)	9·5 (5)
C(82)	0·2175 (8)	0·020 (1)	0·2285 (7)	8·1 (5)
C(83)	0·2631 (9)	0·153 (1)	0·2075 (8)	9·4 (5)
O(3)	0·5440 (5)	0·4531 (8)	0·7168 (4)	3·6 (3)
O(4)	0·505 (1)	0·408 (2)	0·7139 (9)	4·6 (6)

$$* B_{eq} = \tfrac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

Fig. 1. General view of the molecule.

Fig. 2. Stereoscopic view of the molecule.

The central part of the asymmetric monomeric molecule is a seven-membered heterocycle consisting of an Sn, two N and four C atoms. Two *tert*-butyl groups and four phenyl groups are bound to this ring. The two N atoms are part of an annellated N-phenylurazole ring. The N and the three C atoms bound to the Sn form a distorted tetrahedron. The maximum deviations from the ideal tetrahedral angle ($109·47°$) amount to $11·1$ (4)° and are probably brought about by the ring strain and the repulsion of the two bulky *tert*-butyl groups. The Sn–N and the Sn–C bond lengths are in the normal range. Two short C–C bond distances in the seven-membered ring indicate double bonds. The torsion angles Sn(1)–C(4)–C(3)–C(2) $16·4$ (5), C(4)–C(3)–C(2)–C(1) $61·5$ (5), C(3)–C(2)–C(1)–N(2) $2·3$ (5)° show significant torsion around the C(4)–C(3) double bond and a strong deviation from

Table 2. *Selected bond lengths* (Å) *and angles* (°) *and ranges of bond lengths and angles*

C(1)–N(2)	1·45 (1)	C(5)–N(3)	1·39 (1)
C(1)–C(2)	1·33 (2)	N(3)–C(6)	1·38 (1)
C(2)–C(3)	1·48 (1)	C(6)–N(2)	1·37 (1)
C(3)–C(4)	1·33 (1)	C(5)–O(1)	1·23 (1)
C(4)–Sn(1)	2·174 (10)	C(6)–O(2)	1·22 (1)
Sn(1)–N(1)	2·120 (8)	N(3)–C(51)	1·42 (1)
N(1)–N(2)	1·44 (1)	Sn(1)–C(7)	2·185 (7)
N(1)–C(5)	1·36 (1)	Sn(1)–C(8)	2·164 (11)

C(2)–C(1)–N(2)	116·3 (7)	N(2)–N(1)–C(5)	106·3 (8)
C(2)–C(1)–C(41)	127·5 (7)	N(1)–N(2)–C(1)	110·4 (7)
N(2)–C(1)–C(41)	115·9 (9)	N(1)–N(2)–C(6)	108·1 (8)
C(1)–C(2)–C(3)	125·3 (7)	C(1)–N(2)–C(6)	118·9 (6)
C(1)–C(2)–C(31)	122·1 (8)	N(1)–C(5)–N(3)	109·1 (8)
C(3)–C(2)–C(31)	112·6 (9)	N(1)–C(5)–O(1)	126 (1)
C(2)–C(3)–C(4)	125 (1)	N(3)–C(5)–O(1)	151 (1)
C(2)–C(3)–C(21)	109·9 (7)	C(5)–N(3)–C(6)	108·6 (9)
C(4)–C(3)–C(21)	125·6 (9)	C(5)–N(3)–C(51)	126·0 (8)
C(3)–C(4)–Sn(1)	126·0 (8)	C(6)–N(3)–C(51)	125·2 (8)
C(3)–C(4)–C(11)	121·2 (9)	N(3)–C(6)–N(2)	107·7 (8)
Sn(1)–C(4)–C(11)	112·1 (5)	N(3)–C(6)–O(2)	127 (1)
C(4)–Sn(1)–N(1)	98·4 (3)	N(2)–C(6)–O(2)	125·7 (9)
C(4)–Sn(1)–C(7)	110·0 (4)	Sn(1)–C(7)–C(71)	111·0 (7)
C(4)–Sn(1)–C(8)	119·5 (4)	Sn(1)–C(7)–C(72)	109·7 (6)
N(1)–Sn(1)–C(7)	103·7 (3)	Sn(1)–C(7)–C(73)	108·2 (5)
N(1)–Sn(1)–C(8)	104·8 (4)	Sn(1)–C(8)–C(81)	109·7 (8)
C(7)–Sn(1)–C(8)	117·0 (4)	Sn(1)–C(8)–C(82)	110·3 (9)
Sn(1)–N(1)–N(2)	112·3 (6)	Sn(1)–C(8)–C(83)	108 (1)
Sn(1)–N(1)–C(5)	127·5 (5)		

In the phenyl rings C–C bond distances and C–C–C angles are in the range 1·33 (2) to 1·42 (2) Å [mean 1·37 (2) Å] and 117 (1) to 123 (1)° [mean 120 (1)°]. The C–C bond distances to the phenyl rings and in the *tert*-butyl groups are in the range 1·46 (1) to 1·52 (2) Å [mean 1·49 (2) Å] and the C–C–C angles in the *tert*-butyl groups are in the range 107 (1) to 111 (1)° [mean 109·6 (1)°].

coplanarity of the two double bonds. The dihedral angles between the planes through these double bonds – least-squares planes through Sn(1), C(4), C(11), C(3), C(2), C(21) and through C(3), C(2), C(31), C(1), N(2), C(41) – and the planes through the corresponding phenyl groups bound to the sp^2-hybridized C atoms are in the range 53·5 (5) to 84·2 (5)° and therefore a significant resonance between the π-systems does not exist. A least-squares plane through the atoms C(5), C(6), N(1), N(2), N(3), O(1), O(2) shows that no atom deviates significantly from planarity. The dihedral angle between this plane and the least-squares plane through the phenyl group bound to the urazole ring is 45·0 (5)°.

Intermolecular distances do not indicate interactions exceeding van der Waals forces.

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Trichloro(phenylimido)bis(triphenylphosphine)rhenium(V) Dichloromethane (1/1), [ReCl₃(C₆H₅N){P(C₆H₅)₃}₂].CH₂Cl₂

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Abstract. $M_r = 920·85$, triclinic, $P\bar{1}$, $a = 12·296$ (4), $b = 16·961$ (5), $c = 11·022$ (4) Å, $\alpha = 74·00$ (5), $\beta = 99·72$ (5), $\gamma = 108·46$ (5)°, $V = 2088$ (1) Å³, D_m(flotation) $= 1·61$, $D_x = 1·60$ g cm⁻³ for $Z = 2$, Mo $K\alpha$, $\lambda = 0·7107$ Å, $\mu = 34$ cm⁻¹, $F(000) = 996$, room temperature. $R = 0·045$ and $Rw = 0·049$ for 5812 observed reflections. The coordination about Re is distorted octahedral with two apical triphenylphosphines and with the phenylimido group (coordinated to Re *via* the N) and the three Cl atoms in the basal plane. Distances are: Re–P 2·503 (2) and 2·490 (2) Å; Re–Cl 2·415 (2), 2·411 (2), 2·402 (2) Å; Re–N 1·726 (6) Å. The Re–N–C angle is close to linearity at 172·6 (6)°.