Crystal and molecular structure of 3-(2-(1,10-phenanthrolyl))-5,6-diphenyl-1,2,4-triazine-chloroaquotriphenyltin(IV) (1:1)

M. F. C. LADD* AND D. C. POVEY

Department of Chemistry, University of Surrey Guildford, Surrey, GU2 5XH, UK

and

F. E. SMITH

Department of Chemistry, University of Prince Edward Island Charlottetown, Prince Edward Island, C1A 4P3, Canada

(Received August 30, 1982)

Abstract

3-(2-(1,10-Phenanthrolyl))-5,6-diphenyl-1,2,4-triazine-chloroaquotriphenyltin(IV) (1:1) crystallizes in the orthorhombic system: a = 19.195, b = 9.144, c = 21.642 Å, Z = 4, space group $Pca2_1$ (No. 29). The structure was determined using the procedure for difference structures (DIRDIF) with Cu $K\alpha$ diffractometer data, and refined by block-diagonal least squares to R = 0.031for 3287 observed reflections. The tin atom is 5-coordinate with the three phenyl groups forming the equatorial plane. A chlorine atom and a water molecule complete the coordination. The triazine moiety does not coordinate directly to the metal atom. The only interaction is due to two N···H-O hydrogen bonds formed between two nitrogen atoms from the ligand and the water molecule.

Introduction

Complex formation between neutral, planar, tridentate chelating agents and mono- and diorganotin(IV) halides and pseudo-halides is well known (Ferguson *et al.*, 1965; Einstein and Penfold, 1968; May and Curran, 1972; Pelizzi and Pelizzi, 1976). Seven-coordination at the tin(IV) atom has been proposed in several instances (May and Curran, 1972; Pelizzi and Pelizzi, 1976) and confirmed by crystal structure analysis for the 1:1 complex formed between dimethyltin diisothiocyanate and 2,2':6,2"-terpyridyl (Naik and Scheidt, 1973).

In contrast, the formation of stable 1:1 complexes between neutral planar tridentates and triorganotin halides or pseudo-halides is very unusual, and it appears that only one such series of complexes has been reported (Smith and Liengme, 1975). In this instance, the triazines (I) and (II), both of which bear a close structural relationship to terpyridyl, are reported to form stable 1:1 complexes with triphenyltin chloride and triphenyltin isothiocyanate.



Since no seven-coordinate tin(IV) complexes containing three tincarbon bonds have yet been confirmed, it was considered worthwhile to undertake a crystal structure analysis of the 1:1 complex formed between triphenyltin chloride and triazine (I).

Experimental

The triazine (I) was prepared from 1,10-phenanthroline-2-hydrazidine and benzil according to the general method of Case (1965). The compound was obtained from ethanol as yellow crystals. The complex between (I) and triphenyltin chloride was obtained in good yield by mixing hot ethanolic solutions containing stoichiometric quantities of the triazine and triphenyltin chloride. The complex was recrystallized from ethanol as buff polyhedra, m.p. 140°C. Elemental microanalysis was carried out by the Australian Microanalytical Service, CSIRO, Melbourne, Australia.

	$C_{45}H_{32}Cl$	N₅Sn, H₂O		
	С	н	N	Sn
Found (%)	66.54	4.23	8.66	14.4
Calculated (%)	66.32	4.21	8.59	14.6

Structure of C45H32CIN5Sn,H2O

Unit-cell and space-group data were obtained from Weissenberg and precession photographs. Accurate unit-cell dimensions were obtained by a weighted ($w = \tan \theta$) least-squares fit to the θ values of 13 reflections with resolved $\alpha_1 \alpha_2$ doublets, $\theta > 55^\circ$ (26 measurements) on a Siemens single-crystal diffractometer (AED). Crystal data are listed in Table 1.

A crystal of dimensions 0.2, 0.2, 0.4 mm parallel to *a*, *b*, and *c*, respectively, was selected for the intensity measurements. The intensities of 3476 symmetry-independent reflections with $(\sin \theta)/\lambda \le 0.60$ were measured on the diffractometer, using the 5-value measurement technique (Hoppe, 1965). A reference reflection was monitored after every 20 data reflections, and showed no significant change with time.

Each net intensity was assigned a variance, $\sigma^2(I)$, based upon the counting statistics. A total of 189 reflections was classified as "unobserved," using as a criterion $I/\sigma(I) < 3.0$. The intensities were corrected for Lorentz and polarization effects but not for absorption.

Structure determination and refinement

The solution of this structure proved to be extremely difficult. The two possible choices for the space group, based upon systematic absences (*Pca2*₁, No. 29; *Pcam*, No. 57), was resolved by a consideration of the |E|-statistics, and the noncentrosymmetric space group was chosen. A three-dimensional $|E|^2 - 1$ Patterson synthesis indicated that the x coordinate of the tin atom was very close to zero, leading to pseudo-*B* centering in respect of this atom. The z coordinate of tin was fixed at $\frac{1}{2}$ in order to define the origin in that direction. A structure factor calculation, including an overall isotropic temperature factor and a scale factor derived from the calculation of normalized structure factors, gave a conventional *R*-factor of 0.38 and this followed by a Fourier synthesis revealed a fairly heavy peak within bonding

Molecular formula	$C_{45}H_{32}ClN_5Sn, H_2O$
M,	814.94
Crystal system	Orthorhombic
Space group	<i>Pca</i> 2 ₁ (No. 29)
a	19.195(1) Å
b	9.144(0)
с	21.642(1)
Vc	3799(1) Å ³
Ζ	4
D_x	$1.425(1) \text{ g cm}^{-3}$
<i>F</i> (000)	1656
$\mu(\operatorname{Cu} K\alpha)$	64.5 cm ^{-1}

Table 1. Crystal data

Table 2. Final positional and thermal parameters^a, with esd's in parentheses

Atom	<i>x</i> -	-y	Z ~	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sn	0.4911(0)	0.1788(0)		0.051(0)	0.026(0)	0.040(0)	0.000(0)	-0.000(0)	-0.001(0)
c	0.5910(1)	0.1289(2)	0.4287(1)	0.064(1)	0.054(1)	0.060(1)	0.004(1)	0.017(1)	-0.005(1)
N(1)	0.4131(3)	0.4740(5)	0.6503(2)	0.053(3)	0.035(2)	0.040(2)	-0.003(2)	-0.001(2)	-0.004(2)
N(2)	0.3677(2)	0.2228(5)	0.7048(2)	0.047(2)	0.038(2)	0.037(2)	-0.003(2)	-0.001(2)	-0.002(2)
N(3)	0.3297(2)	0.0014(5)	0.6298(2)	0.065(3)	0.040(3)	0.044(3)	-0.009(2)	-0.002(2)	-0.002(2)
N(4)	0.3092(3)	-0.1059(6)	0.5918(2)	0.062(3)	0.048(3)	0.049(3)	-0.013(2)	-0.006(2)	0.006(2)
N(5)	0.2876(2)	-0.1300(5)	0.7165(2)	0.044(2)	0.044(2)	0.043(3)	-0.003(2)	-0.002(2)	0.003(2)
C(I)	0.4328(3)	0.5997(6)	0.6239(3)	0.048(3)	0.039(3)	0.056(4)	0.002(2)	0.002(3)	0.001(3)
C(2)	0.4533(3)	0.7242(7)	0.6587(3)	0.051(3)	0.038(3)	0.068(4)	0.002(3)	-0.001(3)	0.001(3)
C(3)	0.4535(3)	0.7165(6)	0.7209(3)	0.052(3)	0.035(3)	0.068(4)	0.004(2)	-0.000(3)	-0.013(3)
C(4)	0.4332(3)	0.5862(6)	0.7502(3)	0.045(3)	0.042(3)	0.051(3)	0.001(2)	-0.002(3)	-0.013(3)
C(5)	0.4125(3)	0.4662(6)	0.7129(3)	0.034(2)	0.037(3)	0.043(3)	-0.000(2)	-0.003(2)	-0.003(2)
C(6)	0.4329(4)	0.5703(7)	0.8166(3)	0.072(4)	0.053(4)	0.046(3)	-0.005(3)	-0.004(3)	-0.016(3)
C(1)	0.4147(4)	0.4456(8)	0.8429(3)	0.069(4)	0.071(3)	0.041(3)	-0.006(4)	-0.009(3)	-0.009(3)
C(8)	0.3938(3)	0.3196(7)	0.8071(3)	0.051(3)	0.052(3)	0.045(3)	-0.001(3)	-0.007(3)	-0.004(3)
C(9)	0.3910(3)	0.3316(6)	0.7417(3)	0.039(3)	0.040(3)	0.039(3)	-0.002(2)	-0.001(2)	0.008(2)
C(10)	0.3485(3)	0.0974(6)	0.7320(3)	0.046(3)	0.046(3)	0.038(3)	-0.004(2)	0.002(2)	0.003(2)
C(11)	0.3532(3)	0.0721(7)	0.7955(3)	0.063(4)	0.050(3)	0.044(3)	-0.010(3)	-0.005(3)	0.006(3)
C(12)	0.3752(4)	0.1853(7)	0.8331(3)	0.073(4)	0.066(4)	0.035(3)	-0.011(3)	-0.006(3)	0.003(3)
C(13)	0.3211(3)	-0.0181(6)	0.6902(3)	0.038(3)	0.038(3)	0.049(3)	-0.004(2)	0.001(2)	-0.001(2)
C(14)	0.2614(3)	-0.2313(6)	0.6784(3)	0.034(3)	0.042(3)	0.055(3)	-0.001(2)	-0.002(3)	-0.000(3)
C(15)	0.2758(3)	-0.2241(6)	0.6145(3)	0.043(3)	0.039(3)	0.056(4)	-0.001(2)	-0.005(3)	-0.002(3)
C(16)	0.2598(3)	-0.3371(6)	0.5680(3)	0.040(3)	0.047(3)	0.060(4)	-0.004(2)	-0.003(3)	-0.010(3)
C(17)	0.2626(3)	-0.4857(6)	0.5801(3)	0.046(3)	0.041(3)	0.073(4)	0.001(3)	-0.003(3)	-0.005(3)
C(18)	0.2503(4)	-0.5867(7)	0.5344(4)	0.053(4)	0.047(3)	0.087(5)	0.001(3)	-0.002(3)	-0.020(3)
C(19)	0.2356(4)	-0.5419(9)	0.4754(4)	0.067(4)	0.065(4)	0.100(7)	-0.005(3)	0.008(4)	-0.037(4)
C(20)	0.2313(4)	-0.3933(9)	0.4620(4)	0.081(5)	0.086(5)	0.059(5)	0.002(4)	-0.005(4)	-0.023(4)
C(21)	0.2446(4)	-0.2918(7)	0.5069(4)	0.073(4)	0.054(5)	0.055(5)	-0.003(3)	-0.002(3)	-0.008(3)
C(22)	0.2170(3)	-0.3452(6)	0.7080(3)	0.043(3)	0.043(4)	0.055(3)	-0.005(3)	0.006(3)	-0.005(3)
C(23)	0.2368(4)	-0.4064(8)	0.7632(4)	0.066(5)	0.060(5)	0.070(5)	-0.018(3)	-0.002(4)	0.014(4)
C(24)	0.1957(4)	-0.5111(11)	0.7920(4)	0.110(7)	0.096(7)	0.085(6)	-0.040(6)	0.006(5)	0.026(5)
C(25)	0.1321(5)	-0.5498(12)	0.7634(5)	0.091(6)	0.101(7)	0.110(7)	-0.046(5)	0.009(6)	0.024(6)
C(26)	0.1125(4)	-0.4884(11)	0.7092(5)	0.059(4)	0.097(7)	0.108(7)	-0.035(4)	0.002(4)	0.006(6)
C(27)	0.1541(3)	-0.3835(9)	0.6810(4)	0.042(3)	0.080(5)	0.071(4)	-0.015(3)	-0.002(3)	0.003(4)
C(28)	0.4973(2)	0.4107(5)	0.4912(3)	0.034(2)	0.037(2)	0.022(3)	0.001(2)	-0.004(2)	-0.007(2)

C(29)	0.4444(3)	0.4889(6)	0.4634(3)	0.052(3)	0.042(3)	0.052(3)	0.002(3)	-0.012(3)	0.005(3)
C(30)	0.4486(4)	0.6410(7)	0.4593(3)	0.067(4)	0.048(3)	0.056(4)	0.009(3)	-0.014(3)	0.009(3)
C(31)	0.5036(3)	0.7146(6)	0.4852(4)	0.067(4)	0.033(2)	0.075(7)	-0.003(2)	-0.008(3)	0.008(3)
C(32)	0.5577(3)	0.6383(7)	0.5115(3)	0.055(3)	0.048(3)	0.047(4)	-0.007(3)	-0.002(2)	-0.003(2)
C(33)	0.5548(3)	0.4871(6)	0.5156(2)	0.045(3)	0.036(3)	0.049(4)	-0.001(2)	-0.002(2)	-0.002(2)
C(34)	0.5441(3)	0.0834(6)	0.5785(3)	0.048(3)	0.032(3)	0.049(3)	-0.006(2)	-0.005(3)	0.001(2)
C(35)	0.6042(4)	-0.0027(7)	0.5704(3)	0.063(4)	0.051(4)	0.067(4)	0.011(3)	-0.005(3)	0.000(3)
C(36)	0.6385(4)	-0.0637(8)	0.6224(5)	0.070(5)	0.053(4)	0.113(7)	0.005(4)	-0.026(5)	0.007(5)
C(37)	0.6137(5)	-0.0337(9)	0.6813(4)	0.088(5)	0.063(4)	0.065(4)	-0.002(4)	-0.026(4)	0.010(4)
C(38)	0.5557(4)	0.0500(9)	0.6888(4)	0.089(5)	0.072(5)	0.061(4)	-0.017(4)	-0.025(4)	0.005(4)
C(39)	0.5216(4)	0.1081(8)	0.6383(3)	0.065(4)	0.050(3)	0.051(4)	0.002(3)	-0.004(3)	-0.001(3)
C(40)	0.4150(3)	0.0765(6)	0.4405(3)	0.060(3)	0.034(3)	0.045(3)	0.002(2)	-0.006(3)	0.003(2)
C(41)	0.4345(4)	-0.0089(8)	0.3905(3)	0.087(5)	0.057(4)	0.040(3)	-0.001(4)	0.003(3)	-0.006(3)
C(42)	0.3858(5)	-0.0721(9)	0.3528(3)	0.111(7)	0.073(5)	0.044(4)	-0.009(5)	-0.007(4)	-0.014(4)
C(43)	0.3180(5)	-0.0493(9)	0.3621(4)	0.093(6)	0.081(5)	0.065(5)	-0.002(4)	-0.025(4)	-0.014(4)
C(44)	0.2962(4)	0.0326(11)	0.4099(4)	0.072(5)	0.111(7)	0.083(5)	-0.00(5)	-0.021(4)	-0.027(5)
C(45)	0.3450(4)	0.0967(10)	0.4504(4)	0.059(4)	0.099(6)	0.068(5)	-0.002(4)	-0.011(4)	-0.026(4)
0(1)	0.3980(2)	0.2337(4)	0.5672(2)	0.056(2)	0.037(2)	0.040(2)	0.001(2)	0.006(2)	-0.003(2)
Atom	x	ų	z	U/A^2	Atom	×	ų	z	U/A^2
H(1)	0.428	0.597	0.576	0.035	H(27)	0.144	-0.336	0.635	0.093
H(2)	0.457	0.802	0.636	0.081	H(29)	0.401	0.440	0.452	0.060
H(3)	0.464	0.793	0.473	0.121	H(30)	0.414	0.694	0.441	0.060
(9)H	0.445	0.672	0.856	0.096	H(31)	0.499	0.821	0.492	0.052
H(7)	0.412	0.437	0.885	0.114	H(32)	0.597	0.697	0.526	0.057
H(11)	0.341	-0.025	0.813	0.061	H(33)	0.588	-0.447	0.539	0.087
H(12)	0.375	0.179	0.878	0.050	H(35)	0.621	-0.017	0.530	0.043
H(17)	0.273	-0.516	0.620	0.037	H(36)	0.689	-0.099	0.533	0.263
H(18)	0.249	-0.694	0.543	0.124	H(37)	0.654	-0.079	0.698	0.260
(61)H	0.233	-0.661	0.443	0.237	H(38)	0.545	0.074	0.737	0.149
H(20)	0.224	-0.345	0.428	0.085	H(39)	0.487	0.171	0.641	0.081
H(21)	0.241	-0.187	0.497	0.079	H(41)	0.484	-0.039	0.389	0.053
H(23)	0.279	-0.359	0.790	0.077	H(42)	0.396	-0.158	0.314	0.123
H(24)	0.221	-0.564	0.828	0.112	H(43)	0.269	-0.074	0.347	0.141
H(25)	0.111	-0.662	0.745	0.323	H(44)	0.244	0.045	0.410	0.087
H(26)	0.066	-0.507	0.702	0.067	H(45)	0.340	0.138	0.501	0.188
^a The temp $2U_{21}kh^{*}l_{2}$	erature factor	expression for th hydrogen atoms	ie nonhydrogen is exn(-2# ² 11)	atoms is exp -2 sin ² A)	$(-2\pi^2 \{ U_{11}(ha)$	$*)^{2} + U_{22}(kb*)$	$2^{2} + U_{33}(lc^{*})^{2} + 2$	$2U_{12}ha^{*}kb^{*}+20$	$U_{13}ha^{*}lc^{*} +$
4 NY23VN +	נ. ל) מווח זהו הוור	c IIJUI UZCII ALUIIIS	IS CAP(OT UN	SILL D).					

distance of the tin atom, which was considered to be the chlorine atom, together with a considerable number of other peaks. Several cycles of structure-factor and Fourier calculations enabled a substantial part of the triazine to be assembled with reasonable geometry. Positional and isotropic least-squares refinement of the model reduced R to 0.18, but a subsequent electron density map failed to give any indication of the positions of the three phenyl rings which were present. Considerable time was spent attempting to produce a different model. There was every indication that both the tin and chlorine atoms were correct but no further progress was made, the three phenyl groups never being revealed.

Direct methods, using MULTAN 80 (*Main et al.*, 1980), were tried, but the results were not impressive. The position of the tin atom was revealed on nearly every occasion, with its smaller pseudo-related counterpart, together with a collection of peaks which could be assembled into part of the triazine, but never was there any direct indication of the phenyl groups, nor were they revealed in subsequent electron density maps phased on models obtained from the direct methods procedures.

Next the DIRDIF procedure (Beurskens *et al.*, 1980) was utilized. A preliminary run of the program used the proposed position of the tin atom as a starting model together with the position of the pseudo-center of symmetry $(0, 0, \frac{1}{4})$.

The resulting DIRDIF-Fourier map revealed the triazine minus one phenyl ring, three phenyl rings with poor geometry coordinated to the tin atom, the chlorine atom, and an indication of a pseudo-related tin atom. The model was developed by utilizing a Sim-weighted difference Fourier synthesis (available within the DIRDIF system); the resulting map revealed the complete structure.

Positional and isotropic thermal parameters and an overall scale factor, all refined by full-matrix least squares, reduced R to 0.14, the quantity minimized being $\Sigma w(|F_o| - |F_c|)^2$ with w = 1.

Four further cycles of block-diagonal least squares, now with anisotropic thermal parameters, gave R = 0.054.

A three-dimensional difference Fourier map revealed the positions of some of the hydrogen atoms, the remainder, except the water hydrogens, being calculated by geometry. At this point, a weighting scheme (w = 1 if $A < |F_o|$; otherwise, $w = (A/|F_o|)^2$ with A = 45) was applied to the data and considered to be satisfactory by the usual criteria.

A further four cycles of block-diagonal least-squares refinement was carried out, including the hydrogen atoms and with dispersion factors for the atoms; at the same time the polarity of the structure was checked. Coordinate set 1 gave $R_1 = 0.031$ and $R_{1w} = 0.040$, and coordinate set 2 gave $R_2 = 0.045$ and $R_{2w} = 0.048$. Set 1 is significantly better than set 2 at the 0.01 level (Hamilton, 1965), and the results listed correspond to set 1.

Structure of C45H32ClN5Sn,H2O

Scattering factors were taken from *International Tables for X-Ray Crystallography* (1974). Computations were performed by local programs and the x-RAY 72 system (Stewart *et al.*, 1972). The final atomic parameters are listed in Table 2, and the atom numbering scheme is shown in Fig. 1.

Discussion

The structure shown in Figs. 2 and 3 was most unexpected. The triazine moiety is not directly coordinated to the tin atom but is hydrogen-bonded to the coordinated water molecule. Thus, the tin atom is only five-coordinate, and essentially trigonal bipyramidal, with the three phenyl groups occupying the equatorial positions. The axial positions are taken up by the chlorine atom and the oxygen atom of the water molecule. The triazine moiety is held in the complex by hydrogen bonds between the water molecule and two nitrogen atoms, one from the phenanthroline ring system, N(1), and the other from the triazine ring system, N(3). Bond lengths and bond angles are listed in Table 3.

The measured bond lengths are all within the ranges expected for the structure described. The average Sn-C distance between the tin atom and the three phenyl groups is 2.15 Å, which is, as would be expected, longer than that found (2.09 Å) in the tetrahedral compound triphenyltin isothiocyanate (Domingos and Sheldrick, 1974) and between those values reported for the five-coordinate trigonal bipyramidal complexes triphenyltin isocyanate (2.12 Å) and dichlorotriphenylstannate(IV) (2.19 Å) (Harrison *et al.*, 1978).



Fig. 1. Diphenyltriazine phenanthroline and triphenyltin moieties, showing the atom numbering.



Fig. 2. Diphenyltriazine phenanthroline moiety and its linkage to tin.





Structure of C45H32ClN5Sn,H2O

C(1) - C(2)	1.420(9)	C(2)-C(3)	1.348(10)
C(3) - C(4)	1.405(8)	C(4) - C(5)	1.420(8)
C(5) = N(1)	1.358(7)	N(1)-C(1)	1 338(7)
C(4) - C(6)	1 443(9)	C(6) - C(7)	1 321(10)
C(7) - C(8)	1.445(10)	C(8) - C(9)	1.420(8)
C(0) = C(5)	1.440(8)	C(0) = C(1)	1.420(0)
V(3) = C(10)	1.440(0)	C(9) + N(2)	1.331(7)
N(2) = C(10)	1.340(7)	C(10) - C(11)	1.396(8)
C(11) - C(12)	1.383(9)	C(12) - C(8)	1.397(9)
C(10)-C(13)	1.487(8)		
C(13)-N(5)	1.336(7)	N(5)-C(14)	1.338(8)
C(14)-C(15)	1.411(9)	C(15)-N(4)	1.350(8)
N(4) - N(3)	1.339(7)	N(3)-C(13)	1.330(8)
C(15)-C(16)	1.475(9)	C(14)-C(22)	1.491(8)
C(16)-C(17)	1.385(8)	C(22)-C(23)	1.373(10)
C(17) - C(18)	1.374(10)	C(23) - C(24)	1.390(12)
C(18) - C(19)	1.370(12)	C(24) - C(25)	1.413(14)
C(19) - C(20)	1 396(12)	C(25) - C(26)	1 355(15)
C(20) - C(21)	1.390(12) 1.381(11)	C(26) - C(27)	1.390(12)
C(21) = C(21)	1.331(11)	C(27) = C(27)	1.396(0)
C(21) = C(10)	1.415(10)	$C(27)^{-}C(22)$	1.360(9)
C(28)-C(29)	1.379(8)	C(34)-C(35)	1.406(9)
C(29)-C(30)	1.396(9)	C(35)-C(36)	1.418(12)
C(30)-C(31)	1.371(10)	C(36)-C(37)	1.387(9)
C(31)-C(32)	1.375(9)	C(37)-C(38)	1.361(13)
C(32)-C(33)	1.386(8)	C(38)-C(39)	1.381(11)
C(33) - C(28)	1.410(7)	C(39) - C(34)	1.383(9)
C(40) - C(41)	1 387(9)	Sn-C(28)	2 133(5)
C(41) - C(42)	1 370(11)	Sn-C(34)	2 163(6)
C(42) - C(43)	1 333(13)	Sn = C(40)	2.163(6)
C(42) = C(43)	1.335(13)	$S_n C^1$	2.101(0)
C(44) = C(44)	1.044(10)	$S_{n} = O(1)$	2.303(2)
C(44) = C(43)	1.411(12)	311-0(1)	2.339(4)
C(43) = C(40)	1.372(10)		
C(1)-C(2)-C(3)	119.3(6)	C(2)-C(3)-C(4)	119.6(6)
C(3) - C(4) - C(5)	118.4(6)	C(4) - C(5) - N(1)	121.7(5)
C(5)-N(1)-C(1)	118.2(5)	N(1)-C(1)-C(2)	122.7(6)
C(3)-C(4)-C(6)	122.4(6)	C(5)-C(4)-C(6)	119.2(5)
C(4)-C(6)-C(7)	121.1(6)	C(6)-C(7)-C(8)	122.0(6)
C(7)-C(8)-C(9)	118.9(5)	C(8)-C(9)-C(5)	119.1(5)
C(7)-C(8)-C(12)	123.7(6)	C(5)-C(9)-N(2)	118.0(5)
C(9)-C(5)-C(4)	119.7(5)	N(1) - C(5) - C(9)	118.6(5)
C(8) - C(9) - N(2)	123.0(5)	C(9) - N(2) - C(10)	117.5(5)
N(2) - C(10) - C(11)	123.8(5)	C(10) - C(11) - C(12)	118.3(6)
C(11) = C(12) = C(8)	120.0(6)	C(12) = C(8) = C(9)	117 4(5)
N(2) = C(10) = C(13)	116 0(5)	C(11) = C(10) = C(13)	120.2(5)
C(10)-C(13)-N(3)	117.3(5)	C(10)-C(13)-N(5)	120.2(5) 117.1(5)
N(2) C(12) N(5)	125 6(5)	C(12) N(5) $C(14)$	116 ((5)
N(3) - U(13) - N(3)	123.0(3)	C(13) - N(3) - C(14)	110.0(5)
N(5) - C(14) - C(15)	119.9(5)	C(14) - C(15) - N(4)	119.1(5)
C(15) - N(4) - N(3)	120.2(5)	N(4) - N(3) - C(13)	117.9(5)
N(4) - C(15) - C(16)	114.3(5)	C(14)-C(15)-C(16)	126.5(5)
C(15)-C(14)-C(22)	124.5(5)	N(5)-C(14)-C(22)	115.6(5)

Table 3. Bond lengths/Å and bond angles/deg, with esd's in parentheses

$\begin{array}{cccccccccccccccccccccccccccccccccccc$				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(15)-C(16)-C(17)	123.3(6)	C(14)-C(22)-C(23)	120.1(6)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(15)-C(16)-C(21)	118.4(5)	C(14)-C(22)-C(27)	119.5(6)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(17)-C(16)-C(21)	118.2(6)	C(23)-C(22)-C(27)	120.3(6)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(16)-C(17)-C(18)	121.1(7)	C(22)-C(23)-C(24)	121.0(7)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(17)-C(18)-C(19)	120.4(6)	C(23)-C(24)-C(25)	117.7(9)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(18)-C(19)-C(20)	120.5(8)	C(24)-C(25)-C(26)	121.2(9)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(19)-C(20)-C(21)	119.1(8)	C(25)-C(26)-C(27)	120.3(8)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(20)-C(21)-C(16)	120.7(6)	C(26)-C(27)-C(22)	119.4(7)
$\begin{array}{ccccccc} C(29)-C(30)-C(31) & 120.5(6) & C(35)-C(36)-C(37) & 119.5(7)\\ C(30)-C(31)-C(32) & 120.1(6) & C(36)-C(37)-C(38) & 120.1(8)\\ C(31)-C(32)-C(33) & 120.2(6) & C(37)-C(38)-C(39) & 120.6(7)\\ C(32)-C(33)-C(28) & 120.1(5) & C(38)-C(39)-C(34) & 122.0(6)\\ C(33)-C(28)-C(29) & 118.9(5) & C(39)-C(34)-C(35) & 117.6(6)\\ \hline\\ C(40)-C(41)-C(42) & 121.2(7) & \\ C(41)-C(42)-C(43) & 120.7(7) & \\ C(42)-C(43)-C(44) & 120.5(8) & \\ C(43)-C(44)-C(45) & 120.2(8) & \\ C(44)-C(45)-C(40) & 119.8(7) & \\ C(45)-C(40)-C(41) & 117.6(6) & \\ \hline\\ C(28)-Sn-C(34) & 116.4(2) & C(28)-Sn-Cl & 94.8(1)\\ C(34)-Sn-C(40) & 127.7(2) & C934)-Sn-Cl & 92.9(1)\\ C(40)-Sn-C(28) & 114.5(2) & C(40)-Sn-Cl & 94.1(1)\\ \hline\\ C(28)-Sn-O(1) & 83.4(2) & Cl-Sn-O(1) & 178.2(1)\\ C(34)-Sn-O(1) & 87.6(2) & \\ C(40)-Sn-O(1) & 86.9(2) & \\ \hline\end{array}$	C(28)-C(29)-C(30)	120.2(6)	C(34)-C(35)-C(36)	120.2(7)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(29)-C(30)-C(31)	120.5(6)	C(35)-C(36)-C(37)	119.5(7)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(30)-C(31)-C(32)	120.1(6)	C(36)-C(37)-C(38)	120.1(8)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(31)-C(32)-C(33)	120.2(6)	C(37)-C(38)-C(39)	120.6(7)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(32)-C(33)-C(28)	120.1(5)	C(38)-C(39)-C(34)	122.0(6)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(33)-C(28)-C(29)	118.9(5)	C(39)-C(34)-C(35)	117.6(6)
$\begin{array}{cccc} C(41)-C(42)-C(43) & 120.7(7) \\ C(42)-C(43)-C(44) & 120.5(8) \\ C(43)-C(44)-C(45) & 120.2(8) \\ C(44)-C(45)-C(40) & 119.8(7) \\ C(45)-C(40)-C(41) & 117.6(6) \\ \hline \\ C(28)-Sn-C(34) & 116.4(2) & C(28)-Sn-Cl & 94.8(1) \\ C(34)-Sn-C(40) & 127.7(2) & C934)-Sn-Cl & 92.9(1) \\ C(40)-Sn-C(28) & 114.5(2) & C(40)-Sn-Cl & 94.1(1) \\ C(28)-Sn-O(1) & 83.4(2) & Cl-Sn-O(1) & 178.2(1) \\ C(34)-Sn-O(1) & 87.6(2) \\ C(40)-Sn-O(1) & 86.9(2) \\ \hline \end{array}$	C(40)-C(41)-C(42)	121.2(7)		
$\begin{array}{ccccc} C(42)-C(43)-C(44) & 120.5(8) \\ C(43)-C(44)-C(45) & 120.2(8) \\ C(44)-C(45)-C(40) & 119.8(7) \\ C(45)-C(40)-C(41) & 117.6(6) \\ \hline \\ C(28)-Sn-C(34) & 116.4(2) & C(28)-Sn-Cl & 94.8(1) \\ C(34)-Sn-C(40) & 127.7(2) & C934)-Sn-Cl & 92.9(1) \\ C(40)-Sn-C(28) & 114.5(2) & C(40)-Sn-Cl & 94.1(1) \\ C(28)-Sn-O(1) & 83.4(2) & Cl-Sn-O(1) & 178.2(1) \\ C(34)-Sn-O(1) & 87.6(2) \\ C(40)-Sn-O(1) & 86.9(2) \\ \hline \end{array}$	C(41)-C(42)-C(43)	120.7(7)		
$\begin{array}{cccc} C(43)-C(44)-C(45) & 120.2(8) \\ C(44)-C(45)-C(40) & 119.8(7) \\ C(45)-C(40)-C(41) & 117.6(6) \\ \\ C(28)-Sn-C(34) & 116.4(2) & C(28)-Sn-Cl & 94.8(1) \\ C(34)-Sn-C(40) & 127.7(2) & C934)-Sn-Cl & 92.9(1) \\ C(40)-Sn-C(28) & 114.5(2) & C(40)-Sn-Cl & 94.1(1) \\ \\ C(28)-Sn-O(1) & 83.4(2) & Cl-Sn-O(1) & 178.2(1) \\ C(34)-Sn-O(1) & 87.6(2) \\ C(40)-Sn-O(1) & 86.9(2) \\ \end{array}$	C(42)-C(43)-C(44)	120.5(8)		
$\begin{array}{cccc} C(44)-C(45)-C(40) & 119.8(7) \\ C(45)-C(40)-C(41) & 117.6(6) \\ \\ C(28)-Sn-C(34) & 116.4(2) & C(28)-Sn-Cl & 94.8(1) \\ C(34)-Sn-C(40) & 127.7(2) & C934)-Sn-Cl & 92.9(1) \\ C(40)-Sn-C(28) & 114.5(2) & C(40)-Sn-Cl & 94.1(1) \\ \\ C(28)-Sn-O(1) & 83.4(2) & Cl-Sn-O(1) & 178.2(1) \\ C(34)-Sn-O(1) & 87.6(2) \\ C(40)-Sn-O(1) & 86.9(2) \end{array}$	C(43)-C(44)-C(45)	120.2(8)		
$\begin{array}{cccc} C(45)-C(40)-C(41) & 117.6(6) \\ C(28)-Sn-C(34) & 116.4(2) & C(28)-Sn-Cl & 94.8(1) \\ C(34)-Sn-C(40) & 127.7(2) & C934)-Sn-Cl & 92.9(1) \\ C(40)-Sn-C(28) & 114.5(2) & C(40)-Sn-Cl & 94.1(1) \\ C(28)-Sn-O(1) & 83.4(2) & Cl-Sn-O(1) & 178.2(1) \\ C(34)-Sn-O(1) & 87.6(2) \\ C(40)-Sn-O(1) & 86.9(2) \end{array}$	C(44)-C(45)-C(40)	119.8(7)		
$\begin{array}{cccccc} C(28)-Sn-C(34) & 116.4(2) & C(28)-Sn-Cl & 94.8(1) \\ C(34)-Sn-C(40) & 127.7(2) & C934)-Sn-Cl & 92.9(1) \\ C(40)-Sn-C(28) & 114.5(2) & C(40)-Sn-Cl & 94.1(1) \\ C(28)-Sn-O(1) & 83.4(2) & Cl-Sn-O(1) & 178.2(1) \\ C(34)-Sn-O(1) & 87.6(2) & \\ C(40)-Sn-O(1) & 86.9(2) & \\ \end{array}$	C(45)-C(40)-C(41)	117.6(6)		
$\begin{array}{cccc} C(34)-Sn-C(40) & 127.7(2) & C934)-Sn-Cl & 92.9(1) \\ C(40)-Sn-C(28) & 114.5(2) & C(40)-Sn-Cl & 94.1(1) \\ C(28)-Sn-O(1) & 83.4(2) & Cl-Sn-O(1) & 178.2(1) \\ C(34)-Sn-O(1) & 87.6(2) & \\ C(40)-Sn-O(1) & 86.9(2) & \\ \end{array}$	C(28)-Sn-C(34)	116.4(2)	C(28)-Sn-Cl	94.8(1)
C(40)-Sn-C(28) 114.5(2) C(40)-Sn-Cl 94.1(1) C(28)-Sn-O(1) 83.4(2) Cl-Sn-O(1) 178.2(1) C(34)-Sn-O(1) 87.6(2) C(40)-Sn-O(1) 178.2(2) C(40)-Sn-O(1) 86.9(2) 178.2(2) 178.2(2)	C(34)-Sn-C(40)	127.7(2)	C934)-Sn-Cl	92.9(1)
C(28)-Sn-O(1) 83.4(2) Cl-Sn-O(1) 178.2(1) C(34)-Sn-O(1) 87.6(2) </td <td>C(40)-Sn-C(28)</td> <td>114.5(2)</td> <td>C(40)-Sn-Cl</td> <td>94.1(1)</td>	C(40)-Sn-C(28)	114.5(2)	C(40)-Sn-Cl	94.1(1)
C(34)-Sn-O(1) 87.6(2) C(40)-Sn-O(1) 86.9(2)	C(28)-Sn-O(1)	83.4(2)	Cl-Sn-O(1)	178.2(1)
C(40)-Sn-O(1) 86.9(2)	C(34)-Sn-O(1)	87.6(2)		
	C(40)-Sn-O(1)	86.9(2)		

Table 3. Continued

The length of the tin-chlorine bond is 2.48 Å, which also fits into the range reported by Harrison (1974) for the tin-chlorine bond in a series of complexes similar to the one here considered.

One of the most interesting features of the structure is the hydrogen bonding scheme mentioned above. The N-O distances [N(1)-O(1), 2.85 Å;N(3)-O(1), 2.83 Å] are well within the range predicted for this type of $O-H \cdots N$ hydrogen bond and there may also be some interaction via N(2)-O(1), 3.04 Å (Sutton, 1958). The hydrogen bonding described here between the coordinated water molecule and the triazine moiety is similar to that displayed by 1,10-phenanthroline monohydrate (Donnay *et al.* 1965).

Acknowledgments

We are grateful to Prof. Paul Beurskens for introducing us to DIRDIF and for assisting with its use in solving this structure.

References

- Beurskens, P. T., and Van den Hark, Th. E. M. (1980) in *Theory and Practice of Direct Methods in Crystallography*, M. F. C. Ladd and R. A. Palmer, eds. (Plenum Press, New York), p. 255 and references therein.
- Case, F. H. (1965) J. Org. Chem. 30, 931.
- Domingos, A. M., and Sheldrick, G. M. (1974) J. Organomet. Chem. 67, 257.
- Donnay, G., Donnay, J. D. H., and Harding, M. J. C. (1965) Acta Crystallogr. 19, 688.
- Einstein, F. W. B., and Penfold, B. R. (1968) J. Chem. Soc. A, 3019.
- Ferguson, J. E., Roper, W. R., and Wilkins, C. J. (1965) J. Chem. Soc. A, 3716.
- Hamilton, W. C. (1965) Acta Crystallogr. 18, 502.
- Harrison, P. G., King, T. J., and Richards, J. A. (1974) J. Chem. Soc. Dalton Trans. 1723.
- Harrison, P. G., Molloy, K., Phillips, R. C., Smith, P. J., and Crowe, A. J. (1978) J. Organomet. Chem. 160, 421.
- Hoppe, W. (1965) Angew. Chem. 77, 484.
- International Tables for X-Ray Crystallography (1974) Vol. IV, J. A. Ibers and W. C. Hamilton, eds., (Kynoch Press, Birmingham).
- Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. and Woolfson, M. M. (1980) MULTAN 80: A System of Computer Programs for the Automatic Solution of Crystal Structures from X-Ray Diffraction Data. University of York, England, and Louvain, Belgium.
- May, J. C. and Curran, C., (1972) J. Organomet. Chem. 39, 289.
- Naik, D. V., and Scheidt, W. R. (1973) Inorg. Chem. 12, 272.
- Pelizzi, C., and Pelizzi, G. (1976) Inorg. Chim. Acta. 18, 139.
- Smith, F. E., and Liengme, B. V. (1975) J. Organomet. Chem. 91, C31.
- Stewart, J. M., Kruger, G. J., Ammon, H. L., Dickinson, C. H., and Hall, S. R. (1972) The x-RAY System-Version of June 1972. Tech. Rep. TR-192. Computer Science Center, University of Maryland, College Park, Maryland.
- Sutton, L. E., ed. (1958) Tables of Interatomic Distances and Configuration in Molecules and Ions (The Chemical Society, London).

British Library Lending Division Supplementary Publication No. 66031 contains 34 pages of structure factor tables.