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Reactions of 8-Aminoquinoline with Diorganotin Dichlorides

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The reactions between 8-aminoquinoline and R_2SnCl_2 (R = Me, Bu and Ph) have been investigated and the crystal structures of diphenyldichloro(8-aminoquinoline)tin(IV) **1** and 8-aminoquinolinium chloride **2** are reported. The smoke-grey crystals of **1** are triclinic with $a = 10.1879(10)$, $b = 10.6335(11)$, $c = 9.9927(9)$ Å, $\alpha = 97.426(7)$, $\beta = 81.853(7)$, $\gamma = 109.620(6)°$, $Z = 2$, space group $P\bar{1}$ and $R = 0.031$ for 4908 observed reflections. The molecules are octahedral with the phenyl groups *trans* to the 8-aminoquinoline, with $d(Sn–C) = 2.170(2)$, $d(Sn–N) = 2.313(2)$ and $d(Sn–Cl) = 2.509(1)$ Å. Orange crystals of **2** are monoclinic with $a = 6.9650(17)$, $b = 8.2078(19)$, $c = 15.4031(32)$ Å and $\beta = 94.380(11)°$, $Z = 4$, space group $P2_1/n$ and $R = 0.036$ for 788 observed reflections. The 8-aminoquinoline is protonated at the pyridyl nitrogen atom which is hydrogen-bonded to the chloride ion.

Extensive studies have been carried out on organotin compounds with symmetric *N,N'*-chelating agents such as 1,10-phenanthroline,[1] and with asymmetric ones, e.g., 2-aminomethylpyridine, because of the link between antitumour activity and the Sn–N bond distance in these compounds.[2–4] 8-Aminoquinoline is one such asymmetric bidentate ligand but, to the best of our knowledge, no reactions between it and diorganotin(IV) dihalides have been reported. However stable adducts with trichlorobutyltin[5] and with tin(II) chloride[6] have been reported. In this paper we report the reactions of R_2SnCl_2 (R = Ph, Bu or Me) with 8-aminoquinoline which were carried out in an attempt to prepare compounds displaying antitumour activity. Whereas Ph_2SnCl_2 yields a stable 1:1 adduct the reaction with Bu_2SnCl_2 or with Me_2SnCl_2 leads to protonation of the pyridine nitrogen atom and the formation of 8-aminoquinolinium chloride while the diorganotin moieties are hydrolysed and dehydrated to dimeric distannoxane, $[(R_2SnCl)_2O]_2$ for R = Me or Bu. All the reaction products were characterized by a variety of physical methods including crystal structure determinations of diphenyldichloro(8-aminoquinoline)-tin(IV) and of 8-aminoquinolinium chloride.

Experimental

Microanalyses. All microanalyses were determined with a Control Equipment Corporation model 240 XA elemental analyser at the School of Chemical Sciences, Universiti Sains Malaysia.

NMR spectra. The data were recorded on a Bruker AC-P 300 MHz or JOEL FX90 MHz NMR spectrometer. $CDCl_3$ and $(^2H_6)$DMSO were used as solvents, depending on the solubility of the sample, with TMS as the internal standard.

IR spectra. Spectra of the ligands and complexes were measured on a Perkin-Elmer Model 1725FT-IR spectrometer in the frequency range 4000–400 cm^{-1} with the samples prepared as KBr discs.

Reaction of Ph_2SnCl_2 with 8-aminoquinoline in chloroform. To a solution of 8-aminoquinoline (0.72 g, 5 mmol) in chloroform (4 ml), was added a solution of diphenyltin dichloride (1.72 g, 5 mmol) in 10 ml of chloroform. The mixture was gently heated on a hot plate and allowed to cool to room temperature as soon as the mixture started to boil (ca. 1 min). After about 5 h a precipitate had formed and the solution was filtered to give 2.2 g (90% yield) of the 1:1 adduct. Recrystallisation from chloroform afforded colourless crystals of dichlorodiphenyl (8-aminoquinoline)tin(IV) (m.p. 159–160 °C) suitable for structure determination. Anal. Calcd. for $C_{21}H_{18}Cl_2N_2Sn$: C 51.68, H 3.73, N 5.74%. Found: C 50.96, H 3.98, N 5.17%. IR (KBr, cm^{-1}) $\nu(NH_2)$ 3192, 3153. ^1H NMR ($CDCl_3$–[$^2H_6$]DMSO): δ 8.71 (1 H,

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$J_{H2,H3}$ 5.6 Hz, $J_{H2,H4}$ 1.4 Hz, H2), 8.14 (1 H, $J_{H3,H4}$ 8.2 Hz, H4), 6.88 (1 H, $J_{H6,H7}$, 7.5 Hz, H7), 7.94–7.05 (m, 3 H, H3,H5,H6), 6.0 (s, 2 H, NH_2), 7.35–7.25 (12 H, Ph_2Sn) ^{13}C NMR ($CDCl_3$–[$^2H_6$]DMSO): δ 146.74 (C2), 121.18 (C3), 135.68 (C4), 113.81 (C5), 127.60 (C6), 108.78 (C7), 144.65 (C8), 137.28 (C9), 128.35 (C10), 154.88, 134.54, 127.33, 127.09 (Sn–Ph carbons).

Reaction of Bu_2SnCl_2 with 8-aminoquinoline in chloroform. A solution of 1.5 g (5 mmol) of Bu_2SnCl_2 in 5 ml of chloroform was added to a solution of 8-aminoquinoline (1.44 g, 10 mmol) in 5 ml chloroform in a 25 ml conical flask. The mixture was heated to boiling on a hotplate. On cooling, petroleum ether (b.p. 60–80 °C) was added dropwise (10 ml) to the mixture until the solution became turbid, and the solution was then allowed to stand in a freezer overnight. On filtration, a solid product weighing 1.1 g, m.p. 200–201 °C was obtained. Recrystallisation from methanol–chloroform (3:10) and dropwise addition of petroleum ether (7 drops) yielded dark red crystals, m.p. 207–209 °C which were identified as 8-aminoquinolinium chloride.[7] Anal. Calcd. for $C_9H_9ClN_2$: C 59.84, H 5.03, N 15.51%. Found: C 58.69, H 4.87, N 15.00%. IR (KBr, cm^{-1}): ν_{NH_2} 337, 3326; ν_{NH^+} 2727 (br). ^1H NMR ($CDCl_3$–[$^2H_6$]DMSO): δ 8.98 (1 H, $J_{H2,H3}$ 4.8 Hz, $J_{H2,H4}$ 1.5 Hz, H2), 8.70 (1 H, $J_{H3,H4}$ 8.5 Hz, H4), 7.89–7.30 (m, 4 H, H3,H5,H6,H7), 6.2 (br, 3 H NH_2/NH^+) ppm. ^{13}C NMR ($CDCl_3$–[$^2H_6$]DMSO): 144.06 (C2) 129.16 (C3), 142.77 (C4), 119.43 (C5), 121.39 (C6), 118.25 (C7), 136.62 (C8), 132.06 (C9), 129.57 (C10).

8-Aminoquinoline: ^1H NMR ($CDCl_3$–[$^2H_6$]DMSO): δ 8.73 (1 H, $J_{H2,H3}$ 4.2 Hz, $J_{H2,H4}$ 1.7 Hz, H2), 7.95 (1 H, $J_{H3,H4}$ 8.4 Hz, H3), 6.86 (1 H, $J_{H5,H7}$ 1.7 Hz, $J_{H6,H7}$ 9.5 Hz, H7), 7.38–7.00 (m, 3 H, H3,H5,H7), 5.0 (br, 2 H, NH_2). ^{13}C NMR ($CDCl_3$–[$^2H_6$]DMSO): δ 147.04 (C2), 121.19 (C3), 135.76 (C4), 115.08 (C5), 127.41 (C6), 109.54 (C7), 144.35 (C8), 138.08 (C9), 128.67 (C10).

8-Aminoquinolinium chloride was also isolated when the above reaction was carried out with equimolar quantities of the reagents.

Evaporation of the filtrate gave 1.3 g of a brownish solid, m.p. 68–76 °C. Recrystallisation (3 ×) from hexanes yielded a white solid, m.p. 104–107 °C (lit.[8] 112.5 °C), IR (KBr, cm^{-1}) ν_{Sn-O} 597, 588, 562. The solid was identified as 1,2,3,4-tetrabutyl-1,3-dichlorodistannoxane, $[(Bu_2SnCl)_2O]_2$ by comparison with an authentic sample prepared according to literature methods.[8]

Reaction of Me_2SnCl_2 with 8-aminoquinoline in chloroform. Equimolar quantities (2 mmol) of dimethyltin dichloride and 8-aminoquinoline were dissolved in 12 ml of chloroform and heated to boiling. The solution was cooled overnight and 0.31 g of a solid, m.p. 150–160 °C was obtained. Repeated recrystallisation (2 ×) from chloroform–methanol (10:3) yielded red crystals identified as 8-aminoquinolinium chloride. Evaporation of the filtrate gave a brownish-grey solid, m.p. 300 °C. IR (KBr, cm^{-1}) ν_{Sn-O} 600, 550; ν_{Sn-CH_3} 564, 535. The powder contained a few colourless crystals which were identified as 1,1,3,3-tetramethyl-1,3-dichlorodistannoxane. The powder itself gave a difractogram that showed that it was largely amorphous, but with weak lines (d_{obs} = 8.98, 6.06, 5.91 Å) that could not be identified from the JCPDS powder index.[9]

Crystal structure determinations. Cell dimensions were determined from reflections measured at ±2θ. Intensities were measured at 294 K using a Huber 4-circle diffractometer with graphite-monochromatized Mo Kα radiation using an ω–2θ scan. Crystal data for **1** and **2** are given in Table 1. Crystals found in the grey product of the reaction with Me_2SnCl_2 were identified from cell dimensions and refinement on a partial data set as 1,1,3,3-tetramethyl-1,3-dichlorodistannoxane, the structure of which has previously been determined.[10-12]

Data were corrected for background, Lorentz and polarization effects, decay and absorption. Structures were determined using SIR92[13] and subsequent electron density maps. Structures were refined by the least-squares minimization of $\Sigma w(|F_o|-|F_c|)^2$ using a modification of ORFLS.[14] All non-hydrogen atoms were refined anisotropically, hydrogen atom coordinates were determined from difference electron-density maps and were refined with isotropic displacement parameters. The thermal motion of the aminoquinoline groups was analysed, assuming them to be rigid bodies, using TLS,[15] and the bond distances were corrected for libration. The atomic scattering factors were from Ref. 16 as were the anomalous scattering corrections for Sn.

Powder diffraction. Powder diffractograms were recorded with a Stoe transmission (Guinier) type diffractometer using Cu $K\alpha_1$ radiation (λ = 1.540981 Å) selected by a curved Ge monochromator. Intensities were measured with a position-sensitive detector.

Results and Discussion

Diphenyldichloro(8-aminoquinoline)tin(IV). Elemental analysis indicates that diphenyltin dichloride formed a 1:1 adduct with 8-aminoquinoline which behaves as a bidentate ligand through the amino and pyridyl nitrogens. In the ^1H NMR spectrum of the adduct, the broad absorption band at 6 ppm assigned to the two free amino protons was shifted downfield by about 1 ppm on adduct formation thus indicating the involvement of the amino protons. This was supported by the infrared spectrum of the adduct. The ν_{N-H} asymmetric and symmetric stretching vibrations recorded at 3450 and 3349 cm^{-1}, respectively, for 8-aminoquinoline[17] were reduced by about 200 cm^{-1} to 3192 and 3153 cm^{-1} upon coordination. This is in agreement with the fact that diorganotin dihalides show a strong tendency to form octahedral 1:1 adducts with *N,N'*-bidentate ligands.[18] As studies have shown

Table 1. Crystal data for diphenyldichloro(8-aminoquinoline)tin(IV) **1** and 8-aminoquinolinium chloride **2**.

Compound	**1**	**2**
FW/g mol^{-1}	488.01	180.64
Crystal system	Triclinic	Monoclinic
Space group	$P\bar{1}$	$P2_1/n$
Cell parameters (294 K)		
a/Å	10.1879(10)	6.9650(17)
b/Å	10.6335(11)	8.2078(19)
c/Å	9.9927(9)	15.4031(32)
α/°	97.426(7)	90.0
β/°	81.853(7)	94.380(11)
γ/°	109.620(6)	90.0
V/Å^3	1005.5(2)	878.0(4)
Calculated density (294 K)/g cm^{-3}	1.612	1.366
Formula units per cell	2	4
Linear absorption coefficient μ/cm^{-1}	15.489	3.752
Range of transmission factors	0.716–0.766	0.939–0.991
Crystal size/mm	0.19 × 0.44 × 0.22	0.286 × 0.17 × 0.11
No. of reflections measured	6159	1776
No. of unique reflections	5864	1445
No. with $I > 3\sigma$	4908	788
Maximum value of θ	30.0	25.0
Refinement on F		
$R = \Sigma(\|F_o\| - \|F_c\|)/\Sigma\|F_o\|$	0.031	0.036
$R_w = [\Sigma w(\|F_o\| - \|F_c\|)^2/\Sigma\|F_o\|^2]^{1/2}$	0.041	0.043
$S = \Sigma w(\|F_o\| - \|F_c\|)^2/(N_o - N_v)$	0.968	0.996
N_o	3489	788
N_v	308	145
Weighting scheme $w^{-1} = [\sigma_{cs}(F^2) + (1+A)F^2]^{1/2} - \|F\|$		
A	0.02	0.03
Maximum Δ/σ	0.03	0.04
$\Delta\rho_{max}$/e Å^{-3}	1.2(1) near tin	0.22(4)
$\Delta\rho_{min}$/e Å^{-3}	−0.8(1)	−0.15(4)
Extinction correction		None
$(I/I_{corr})_{max}$	0.957	

that organotin compounds complexed to *N,N'* bifunctional ligands having Sn–N bond lengths greater than 2.39 Å exhibit anti-tumour activity[2,19] a crystal structural analysis was undertaken to determine the Sn–N bond distance and also which of the four possible stereoisomers occurs.

Description of structure. The molecular structure of **1** is shown in Fig. 1. The tin is octahedrally coordinated to two phenyl groups which are *trans* to the nitrogen atoms of the 8-aminoquinoline ligand. Sn–N(sp^2) = 2.321(3) Å, which is surprisingly longer than Sn–N(sp^3) = 2.305(3) Å, Sn–Cl distances are 2.502(1) and 2.518(1) Å, and the Sn–C distances are 2.168(3) and 2.172(3) Å. The Cl–Sn–Cl angle is 162.07(3)°, the Sn–Cl bonds being bent away from the phenyl groups, the C–Sn–C angle is opened out to 106.9(1)°, and the N–Sn–N angle is only 71.90(9)°. The phenyl groups are twisted out of the N–Sn–N plane by various extents, the torsion angle N1–Sn–C11–C12 being −124.4(2)°, and N2–Sn–C21–C22 is −83.0(3)°. The geometry is thus similar to that found in related compounds.[20,21] The Sn–N bonds are found to be shorter than those in the active compounds which also differ in that they have chlorine atoms *trans* to the nitrogens of the bifunctional ligand. Fractional atomic coordinates are listen in Table 2.

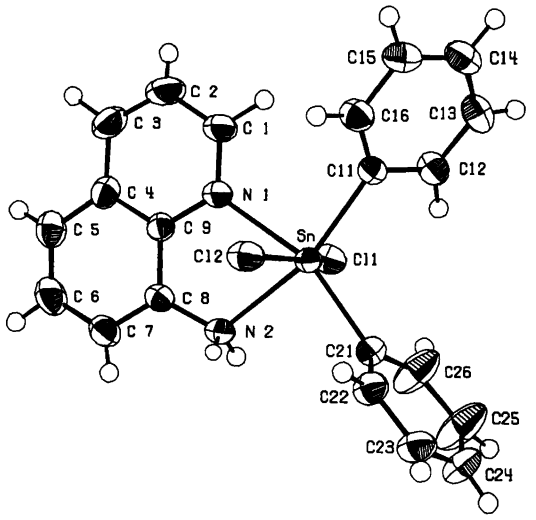

Fig. 1. ORTEPII[29]drawing of **1** showing the atomic numbering. Ellipsoids are drawn at the 50% probability level.

8-Aminoquinolinium chloride. 8-Aminoquinoline is protonated at the pyridyl nitrogen in the presence of dibut-

Table 2. Diphenyldichloro(8-aminoquinoline)tin(IV) **1**. Fractional coordinates and U_{eq}[a] (in Å^2).

Atom	x	y	z	U_{eq}
Sn	0.18610(2)	0.18110(2)	0.25066(2)	0.0286(1)
Cl1	0.18892(8)	0.14893(9)	0.49378(7)	0.0417(4)
Cl2	0.19351(7)	0.14280(9)	−0.00387(7)	0.0401(3)
N1	0.3225(2)	0.0412(3)	0.2204(3)	0.037(1)
N2	0.0383(2)	−0.0375(3)	0.2393(3)	0.033(1)
C1	0.4599(3)	0.0851(4)	0.2181(5)	0.055(2)
C2	0.5390(4)	−0.0022(5)	0.1985(6)	0.069(2)
C3	0.4744(4)	−0.1359(4)	0.1815(4)	0.055(2)
C4	0.3279(3)	−0.1863(3)	0.1870(3)	0.040(2)
C5	0.2503(4)	−0.3244(4)	0.1753(4)	0.048(2)
C6	0.1082(4)	−0.3662(4)	0.1845(4)	0.054(2)
C7	0.0352(4)	−0.2735(4)	0.2039(4)	0.045(2)
C8	0.1062(3)	−0.1384(3)	0.2152(3)	0.033(1)
C9	0.2540(3)	−0.0926(3)	0.2067(3)	0.033(1)
C11	0.3681(3)	0.3605(3)	0.2489(3)	0.031(1)
C12	0.3759(4)	0.4551(3)	0.3598(3)	0.042(2)
C13	0.4871(4)	0.5746(4)	0.3637(4)	0.050(2)
C14	0.5923(4)	0.6008(4)	0.2573(4)	0.047(2)
C15	0.5858(3)	0.5085(4)	0.1469(4)	0.047(2)
C16	0.4729(3)	0.3900(4)	0.1418(3)	0.042(2)
C21	0.0028(3)	0.2465(3)	0.2827(3)	0.036(1)
C22	−0.0646(4)	0.2554(4)	0.1772(3)	0.044(2)
C23	−0.1757(3)	0.3071(4)	0.1976(4)	0.048(2)
C24	−0.2200(4)	0.3497(5)	0.3253(4)	0.056(2)
C25	−0.1530(6)	0.3431(7)	0.4315(5)	0.086(4)
C26	−0.0433(5)	0.2894(6)	0.4111(4)	0.072(3)

[a] $U_{eq} = 1/3\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \boldsymbol{a}_i \boldsymbol{a}_j$.

ylin dichloride or dimethyltin dichloride in chloroform or in methanol. This is confirmed by the IR spectrum, where the stretching vibration for $-NH^+$ is observed in the range 2812–2700 cm^{-1} and there is only a small shift, <80 cm^{-1}, of the asymmetric and symmetric ν_{N-H} vibrations. Elemental analysis confirmed that the compound had a molecular formula consistent with 8-aminoquinolinium chloride. ^{1}H and ^{13}C NMR spectra of the protonated species are also in agreement with protonation at the pyridyl nitrogen, the H-2 (8.98 ppm) and H-4 (8.70 ppm) protons being shifted downfield (H-2 and H-4 of 8-aminoquinoline are 8.73 and 7.95 ppm, respectively[22]). Furthermore, protonation at the pyridyl nitrogen of 8-aminoquinoline produces changes of −3.0, +8.0 and +7.0 ppm in the C-2, -3 and -4 shielding respectively. Similar changes are also observed for the protonation of pyridine which produces changes of −7.8, +5.0 and +12.4 ppm in the C-2, -3 and -4 shieldings.[23] Since there are reports[24,25] indicating that singly protonated 8-aminoquinoline can undergo tautomerism at the pyridyl and amino nitrogens, we therefore carried out an X-ray structure analysis to confirm the above findings.

Description of structure. The molecular structure of **2**, which is shown in Fig. 2, confirms that protonation takes place at the pyridyl nitrogen. The Cl ion is hydrogen bonded to the pyridyl nitrogen atom, N1–Cl = 3.030(7), N1–NH1 = 0.87(4) and Cl–NH1 = 2.16(4) Å, and the

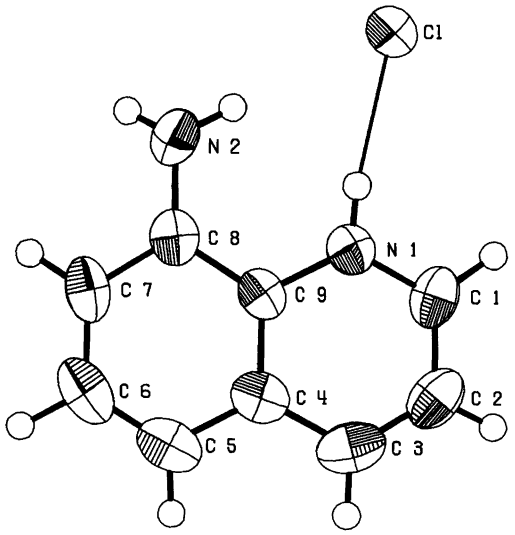

Fig. 2. ORTEPII[29] drawing of **2** showing the atomic numbering. Ellipsoids are drawn at the 50% probability level.

N–H–Cl angle is 172(3)°. The C–N(sp^3) distance is considerably shorter than that in the tin complex, 1.358(6) as against 1.439(4) Å. This difference of 0.081(7) Å may be attributable to thermal vibration but is almost as large, 0.075(7) Å, after correction for rigid body libration. The C–N–H angles are 110(3) and 111(3)° for the tin complex, and were 118(3) and 119(3)°, which also suggests a degree of double bonding in the C–NH_2 bond in the 8-aminoquinolinium cation.

Fractional atomic coordinates are listed in Table 3, and bond distances for the 8-aminoquinoline groups, corrected for thermal motion, are compared in Table 4.

Supplementary material. Further details of the structural work have been deposited with the Cambridge Crystallographic Data Centre, Cambridge, UK.

Reaction of diorganotin dichloride with 8-aminoquinoline. Though 2-aminomethylpyridines are reported to form

Table 3. 8-Aminoquinolinium chloride **2**. Fractional coordinates and U_{eq}[a] (in Å^2).

Atom	x	y	z	U_{eq}
Cl	0.28603(19)	0.07645(13)	−0.15286(6)	0.071(1)
N1	0.2414(4)	0.4046(5)	−0.0671(2)	0.045(2)
N2	0.3620(6)	0.1623(5)	0.0574(3)	0.063(3)
C1	0.1846(6)	0.5116(6)	−0.1275(3)	0.054(2)
C2	0.1524(6)	0.6713(6)	−0.1043(3)	0.060(3)
C3	0.1781(6)	0.7163(6)	−0.0205(3)	0.062(3)
C4	0.2431(5)	0.6035(5)	0.0451(2)	0.050(2)
C5	0.2800(7)	0.6452(6)	0.1335(3)	0.064(3)
C6	0.3470(6)	0.5298(7)	0.1913(3)	0.068(3)
C7	0.3756(6)	0.3706(6)	0.1661(2)	0.058(3)
C8	0.3390(5)	0.3202(5)	0.0804(2)	0.048(2)
C9	0.2736(4)	0.4416(5)	0.0194(2)	0.041(2)

[a] $U_{eq} = 1/3\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \boldsymbol{a}_i \boldsymbol{a}_j$.

Table 4. Comparison of bond distances (Å) of 8-aminoquinoline in **1** and **2** after correction for thermal libration.

	1	**2**
N1–C1	1.323(4)	1.328(5)
N1–C9	1.363(4)	1.378(5)
N2–C8	1.442(4)	1.367(6)
C1–C2	1.405(5)	1.393(7)
C2–C3	1.356(6)	1.351(7)
C3–C4	1.409(5)	1.431(6)
C4–C5	1.419(5)	1.419(6)
C4–C9	1.421(4)	1.419(6)
C5–C6	1.363(6)	1.369(7)
C6–C7	1.405(6)	1.394(7)
C7–C8	1.378(4)	1.400(6)
C8–C9	1.419(4)	1.432(5)

1:1 adducts with diorganotin dichlorides,[26] 8-aminoquinoline was found to form a stable adduct only with diphenyltin dichloride. With a weaker Lewis like dibutyltin dichloride or with dimethyltin dichloride, whose Lewis acidity[27] is between that of diphenyltin and dibutyltin dichlorides, it is protonated at the pyridyl nitrogen whilst the diorganotin moieties are hydrolysed and dehydrated to the dimeric distannoxane. This is in agreement with the findings that the 1:1 adducts formed between R_2SnCl_2 (R = Me or Ph)[11,28] and 2-aminobenzothiazole and between dimethyltin dichloride and potassium ethylxanthate[12] hydrolysed upon recrystallization to distannoxane.

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References

1. Sammes, P. G. and Yahioglu, G. *Chem. Soc. Rev.* (1994) 327.
2. Crowe, A. J., Smith, P. J. and Atassi, G. *Inorg. Chim. Acta 93* (1984) 179.
3. Kabanos, T. A., Keramidas, A. D., Mentzafos, D., Russo, U., Terzin, A. and Tsangaris, J. M. *J. Chem. Soc., Dalton Trans.* (1992) 2729.
4. Saxena, A. K. and Huber, F. *Coord. Chem. Rev. 95* (1989) 109.
5. Mullins, F. P. *Can. J. Chem. 48* (1970) 1677.
6. Doskey, M. A. and Curran, C. *Inorg. Chim. Acta 3* (1969) 169.
7. Buckingham, J., Ed., *Dictionary of Organic Compounds*, 5th ed., Chapman & Hall, New York 1982, Vol. 1.
8. Alleston, D. L., Davies, A. G., Hancock, M. and White, R. F. M. *J. Chem. Soc.* (1963) 5469.
9. JCPDS International Centre for Diffraction Data, 1601 Park Lane, Swarthmore, PA 19081-2389, USA.
10. Graziani, R., Casellato, U. and Plazzogna, G. *Acta Crystallogr., Sect. C 39* (1983) 1188.
11. Harrison, P. G., Begley, M. J. and Molloy, K. C. *J. Organomet. Chem. 186* (1980) 213.
12. Dakternieks, D., Gable, R. W. and Hoskins, B. F. *Inorg. Chim. Acta 85* (1984) L43.
13. Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. and Camalli, M. *J. Appl. Crystallogr. 27* (1994) 435.
14. Busing, W. R., Martin, K. O. and Levy, H. A. ORFLS. Report ORNL-TM 305, Oak Ridge National Laboratory, Tennessee, USA 1962.
15. Schomaker, V. and Trueblood, K. N. *Acta Crystallogr., Sect. B 24* (1968) 63.
16. *International Tables for X-Ray Crystallography*, Vol. IV, Kynoch Press, Birmingham 1974, pp. 72–98.
17. Engelter, G., Jackson, G. E., Knight, C. L. and Thornton, D. A. *J. Coord. Chem. 20* (1989) 289.
18. Biddle, B. N., Gray, J. S. and Crowe, A. J. *Appl. Organomet. Chem. 1*(1987) 261.
19. Crowe, A. J., Smith, P. J. Cardin, C. J., Parge, H. E. and Smith, F. E. *Cancer Lett. 24* (1984) 45.
20. Das, V. G. K., Wei, C., Keong, Y. C. and Mak, T. C. W. *J. Organomet. Chem. 41* (1986) 299.
21. Das, V. G. K., Keong, Y. C., Wei, C., Smith, P. J. and Mak, T. C. W. *J. Chem. Soc., Dalton Trans.* (1987) 129.
22. Kook, A. M., Smith, S. L. and Brown, E. V. *Org. Magn. Reson. 22* (1984) 730.
23. Stothers, J. B. *Carbon-13 NMR Spectroscopy*, Academic Press, New York 1972, p. 212.
24. Ebert, L. *Z. Phys. Chem. (Leipzig) 121* (1926) 385.
25. Zavala, P. J., Radcliffe, M. D. and Schulman, S. G. *Talanta, 32* (1985) 285.
26. Crowe, A. J. and Smith, P. J. *J. Organomet. Chem. 224* (1982) 223.
27. Poller, R. C. *The Chemistry of Organotin Compounds*, Logos, London 1970, Chap. 11.
28. Harrison, P. G. and Molloy, K. *J. Organomet. Chem. 152* (1978) 63.
29. Johnson, C. K., ORTEPII. Report ORNL-5138, Oak Ridge National Laboratory, Tennessee, USA 1976.

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