correlated with a hydrogen bond between these atoms. The existence of a hydrogen bond is also indicated by the position and broadness of υ(OH).

The bond distance between Sb and O(1) of the unidentately bonded carboxylate group is 2·280 (3) Å indicating an appreciably high ionic character. A similar situation was found in $(SbPh_4)CHO_2$, the analogous Sb—O distance being 2·223 Å (Bone & Sowerby, 1979). The Sb—O(H) bond of 1·975 (3) Å is significantly shorter than the Sb—O bonds in $Sb(OH)Ph_4$ (2·048 Å; Beauchamp, Bennett & Cotton, 1969) or in $Sb(OMe)Ph_4$ [2·061 (7) Å] and $Sb(OCH_3)_2Ph_3$ [mean value: 2·033 (8) Å; Kei-wei Shen, McEwen, La Placa, Hamilton & Wolf, 1968] which are considered to be essentially covalent.

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References

Beauchamp, A. L., Bennett, M. J. & Cotton, F. A. (1969). *J. Am. Chem. Soc.* **91**, 297–301.

Bone, S. P. & Sowerby, D. B. (1979). *J. Chem. Res.* (*M*), pp. 1029–1068.

Ferguson, G., Harris, G. S. & Khan, A. (1987). *Acta Cryst.* C**43**, 2078–2081.

Frenz, B. A. (1981). *Enraf–Nonius Structure Determination Package*, 4th ed., version 18. Enraf–Nonius, Delft, The Netherlands.

International Tables for X-ray Crystallography (1974). Vol. IV, Tables 2.2B and 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Kei-wei Shen, McEwen, W. E., La Placa, S. J., Hamilton, W. C. & Wolf, A. P. (1968). *J. Am. Chem. Soc.* **90**, 1718–1723.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Westhoff, T., Huber, F. & Preut, H. (1988). *J. Organomet. Chem.* **348**, 185–191.

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Structures of Bis[aquadimethyl(2,6-pyridinedicarboxylato)tin(IV) hydrate] and Bis[aquadi-*n*-butyl(2,6-pyridinedicarboxylato)tin(IV)]

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Abstract. (I) $\{[Sn(CH_3)_2(C_7H_3NO_4)(H_2O)].H_2O\}_2$, M_r = 699·79, monoclinic, $P2_1/c$, a = 11·576 (6), b = 13·266 (7), c = 7·747 (5) Å, β = 92·67 (5)°, V = 1188 (1) Å^3, Z = 2, D_x = 1·956 Mg m^{-3}, λ(Ag $K\alpha$) = 0·56087 Å, μ = 1·1 mm^{-1}, $F(000)$ = 688, T = 291 (1) K, final R = 0·040 for 2118 unique observed diffractometer data [$F \geq 2{\cdot}0\sigma(F)$]. (II) $[Sn(n\text{-}C_4H_9)_2(C_7H_3NO_4)(H_2O)]_2$, M_r = 832·08, tetragonal, $P4_2/n$, a = 17·684 (12), c = 11·148 (12) Å, V = 3486 (5) Å^3, Z = 4, D_x = 1·585 Mg m^{-3}, λ(Ag $K\alpha$) = 0·56087 Å, μ = 0·78 mm^{-1}, $F(000)$ = 1680, T = 291 (1) K, final R = 0·055 for 2410 unique observed diffractometer data [$F \geq 2{\cdot}0\sigma(F)$]. In both compounds Sn has a distorted pentagonal bipyramidal environment, the alkyl groups being in the apical positions. Both carboxylate groups of the tridentate ligand are unidentate. Two molecular units are linked *via* two asymmetric Sn—O(carboxylate)—Sn bridges to form a planar Sn—O—Sn—O ring. Four O atoms (2 × bridging, 1 × water, 1 × carboxylate) and one N atom form the equatorial plane. Linking of the dimeric units by hydrogen bonds is effected by the water of hydration in the Me_2Sn complex, and by the coordinated water molecule in the Bu_2Sn complex.

Introduction. $Ph_2Sn(H_2O)L$ (H_2L = 2,6-pyridinedicarboxylic acid) has a polymeric chain structure in the crystalline state, the chains being formed by bidentate carboxylate groups and intrachain hydrogen bridges and being linked by interchain hydrogen bridges. Aside from this particular structural feature this compound earns attention since it adds one more example to the few organotin compounds with heptacoordinated Sn and since it proved to possess high *in vitro* activity against various leukemic and tumor cells (Gielen *et al.*, 1987). We now report on the structures of two

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$\mathrm{alkyl_2Sn(H_2O)}_nL$ compounds. They also show high *in vitro* antitumor activity (Gielen *et al.*, 1987) and contain heptacoordinated Sn but they have a distinctly different structure.

Experimental. (I) and (II) were synthesized from 2,6-pyridinedicarboxylic acid and the appropriate dialkyltin oxide (Gielen *et al.*, 1987). Single crystals from undried methanol. Approximate dimensions (I) 0·58 × 0·67 × 0·13 mm, (II) 0·3 × 0·4 × 0·3 mm; Nonius CAD-4 diffractometer, graphite-monochromated Ag $K\alpha$ radiation, $\omega/2\theta$-scan technique, scan speed (I) 2·9–6·7° min^{-1} and (II) 1·7–3·7° min^{-1} in θ, cell parameters from least-squares fit with 25 reflections up to (I) $2\theta = 26{\cdot}7°$ and (II) $2\theta = 28{\cdot}0°$ equally distributed in reciprocal space; six standard reflexions recorded every 2·5 h, only random deviations, 4958 (I) and 7390 (II) reflexions measured in the ranges (I) $1° \le \theta \le 20°$, $-9 \le h \le 9$, $0 \le k \le 16$, $-14 \le l \le 14$ and (II) $1° \le \theta \le 20°$, $-21 \le h \le 21$, $0 \le k \le 21$, $0 \le l \le 13$; after averaging: (I) 2247 ($R_{\text{int}} = 0{\cdot}053$) and (II) 3502 ($R_{\text{int}} = 0{\cdot}017$) unique reflexions, (I) 2118 and (II) 2410 with $F \ge 2{\cdot}0\sigma(F)$; Lorentz–polarization correction and absorption correction *via* ψ scans; max./min. transmission (I) 1·00/0·66, (II) 1·00/0·96; systematic absences (I) ($h0l$) $l = 2n + 1$, ($0k0$) $k = 2n + 1$ conform to space group $P2_1/c$, and (II) ($hk0$) $h + k = 2n + 1$, ($00l$) $l = 2n + 1$ conform to space group $P4_2/n$; structure solution *via* (I) Patterson function and (II) direct methods, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic temperature factor for H atoms, which were placed in geometrically calculated positions (C—H 0·96 Å); H atoms at O(6) were placed on the line connecting the atoms O(6) and O(4) and on the line connecting the atoms O(6) and O(5) ($1-x$, $-0{\cdot}5+y$, $0{\cdot}5-z$) at a distance of 0·96 Å from O(6); refinement on F with (I) 2118 and (II) 2410 reflections and (I) 155 and (II) 200 refined parameters; (I) $w = 1{\cdot}0/[\sigma^2(F) + 0{\cdot}001F^2]$; $S = 1{\cdot}76$, $R = 0{\cdot}040$, $wR = 0{\cdot}046$, $(\Delta/\sigma)_{\text{max}} = 0{\cdot}03$; 12 reflexions which show extinction effects were omitted; (II) $w = 1{\cdot}0/[\sigma^2(F_o) + 0{\cdot}001F_o^2]$; $S = 1{\cdot}30$, $R = 0{\cdot}055$, $wR = 0{\cdot}053$, $(\Delta/\sigma)_{\text{max}} = 0{\cdot}03$; five reflexions which show extinction effects were omitted; largest peak in final ΔF map (I) 0·9 e Å^{-3} near (0·83 Å) Sn(1), next peak has 0·7 e Å^{-3} and (II) ±0·4 e Å^{-3}; complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); programs: Enraf–Nonius *Structure Determination Package* (Frenz, 1985) for data reduction, *SHELXTL-Plus* (Sheldrick, 1987), *MULTAN*80 (Main *et al.*, 1980), *PARST* (Nardelli, 1982).

Discussion. The structures of the two title compounds and the numbering schemes are shown in Figs. 1 and 2. Positional parameters and equivalent values U_{eq} of the anisotropic temperature factors for the non-H atoms are given in Table 1 and bond lengths and angles in Table 2.*

In both compounds (I) and (II) the molecules reside on a center of symmetry and two $R_2\mathrm{Sn}L(\mathrm{H_2O})$ units are linked *via* two asymmetric Sn—O—Sn bridges, such that a planar Sn—O—Sn—O ring is formed. Sn has a distorted pentagonal bipyramidal environment, the two C atoms of the R group being in the apical positions, N(1), the two bridging atoms O(1) and O(1$^{\text{i}}$), the atoms O(3) and O(5), the latter of the coordinated water molecule, forming the equatorial plane. Least-squares planes through the atoms Sn(1), O(1), O(1$^{\text{i}}$), N(1), O(3), O(5), (I) $\psi^2 = 90{\cdot}5$, (II) $\psi^2 = 112{\cdot}1$, show that in (I) the maximum deviation from this plane is 0·026 (4) Å [O(3)] and in (II) 0·039 (5) Å [N(1)]. The maximum deviations of the remaining atoms in the $\mathrm{(O_2C)_2C_5H_3N}$ groups from these planes are (I) 0·187 (4) Å [O(2)] and (II) 0·524 (5) Å [O(4)].

Both carboxylate groups of L in (I) and (II) are unidentate. In (I) the C=O group of that carboxylate group which is not participating in the formation of the

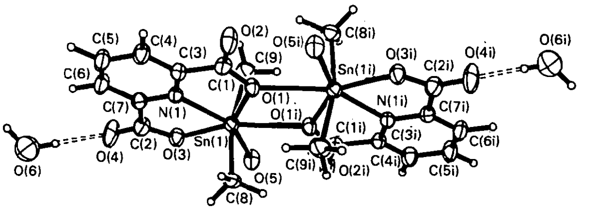

Fig. 1. General view (*SHELXTL-Plus* graphic) of $\{\mathrm{(CH_3)_2Sn[(O_2C)_2C_5H_3N](H_2O).H_2O}\}_2$ (I) showing the atom-numbering scheme [(i) corresponds to the symmetry operation: $-x, -y, -z$].

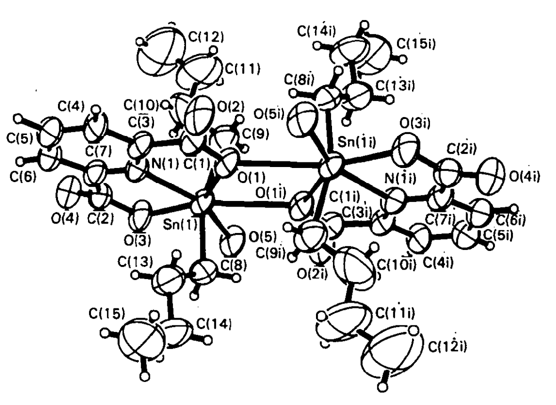

Fig. 2. General view (*SHELXTL-Plus* graphic) of $\{(n\text{-}\mathrm{C_4H_9})_2\mathrm{Sn[(O_2C)_2C_5H_3N](H_2O)}\}_2$ (II), showing the atom-numbering scheme [(i) corresponds to the symmetry operation: $-x, -y, -z$].

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* Lists of H-atom coordinates, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51381 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2 × 10^3)

$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

{$(CH_3)_2Sn[(O_2C)_2C_5H_3N](H_2O).H_2O$}$_2$ (I)

	x	y	z	U_{eq}
Sn(1)	0·17076 (2)	0·03331 (2)	0·09174 (3)	25
O(1)	0·0328 (3)	−0·1010 (2)	0·0020 (4)	29
O(2)	0·0145 (3)	−0·2648 (2)	−0·0591 (5)	45
O(3)	0·3518 (3)	0·0457 (2)	0·1915 (5)	36
O(4)	0·5155 (4)	−0·0312 (2)	0·2768 (8)	60
O(5)	0·1998 (3)	0·2042 (2)	0·1339 (5)	37
O(6)	0·6416 (4)	−0·1737 (4)	0·4993 (6)	70
N(1)	0·2509 (3)	−0·1253 (3)	0·1100 (4)	25
C(1)	0·0689 (4)	−0·1915 (3)	−0·0034 (5)	29
C(2)	0·4160 (4)	−0·0319 (3)	0·2182 (7)	36
C(3)	0·1918 (3)	−0·2079 (3)	0·0631 (5)	24
C(4)	0·2426 (4)	−0·3027 (3)	0·0797 (6)	37
C(5)	0·3556 (4)	−0·3107 (4)	0·1405 (7)	40
C(6)	0·4161 (4)	−0·2241 (4)	0·1834 (7)	38
C(7)	0·3607 (4)	−0·1317 (3)	0·1688 (5)	28
C(8)	0·1003 (5)	0·0302 (3)	0·3362 (6)	35
C(9)	0·2024 (4)	0·0513 (4)	−0·1693 (6)	38

{$(n\text{-}C_4H_9)_2Sn[(O_2C)_2C_5H_3N](H_2O)$}$_2$ (II)

	x	y	z	U_{eq}
Sn(1)	0·07127 (2)	−0·10102 (2)	0·01872 (4)	51
O(1)	0·0090 (2)	0·0008 (2)	0·1255 (3)	56
O(2)	−0·0153 (3)	0·0500 (3)	0·3067 (4)	80
O(3)	0·1416 (3)	−0·1995 (3)	0·0541 (4)	64
O(4)	0·1748 (3)	−0·2866 (3)	0·1897 (4)	67
O(5)	0·0886 (3)	−0·1508 (3)	−0·1749 (4)	71
N(1)	0·0891 (3)	−0·1098 (3)	0·2195 (4)	51
C(1)	0·0141 (4)	0·0026 (4)	0·2395 (5)	57
C(2)	0·1519 (4)	−0·2240 (4)	0·1626 (5)	54
C(3)	0·0616 (3)	−0·0571 (4)	0·2945 (5)	54
C(4)	0·0776 (4)	−0·0621 (5)	0·4157 (6)	65
C(5)	0·1206 (4)	−0·1220 (4)	0·4570 (6)	66
C(6)	0·1484 (4)	−0·1760 (4)	0·3783 (5)	61
C(7)	0·1296 (4)	−0·1684 (4)	0·2578 (5)	51
C(8)	0·1599 (4)	−0·0251 (4)	−0·0177 (6)	64
C(9)	−0·0342 (4)	−0·1572 (5)	0·0135 (6)	72
C(10)	−0·0397 (6)	−0·2255 (7)	0·084 (1)	165
C(11)	−0·1095 (7)	−0·2683 (7)	0·087 (1)	151
C(12)	−0·1073 (8)	−0·3358 (9)	0·158 (1)	228
C(13)	0·1995 (4)	0·0103 (5)	0·0898 (6)	82
C(14)	0·2592 (5)	0·0645 (6)	0·0579 (8)	103
C(15)	0·2890 (6)	0·1079 (7)	0·161 (1)	138

Table 2. *Bond distances* (Å) *and angles* (°)

	(I)	(II)
Sn(1)—O(1)	2·471 (3)	2·422 (4)
Sn(1)—O(1^{i})	2·593 (3)	2·783 (4)
Sn(1)—O(3)	2·206 (4)	2·176 (5)
Sn(1)—O(5)	2·312 (3)	2·352 (5)
Sn(1)—N(1)	2·301 (4)	2·265 (5)
Sn(1)—C(8)	2·097 (5)	2·104 (7)
Sn(1)—C(9)	2·086 (5)	2·114 (8)
O(1)—C(1)	1·272 (5)	1·275 (7)
O(2)—C(1)	1·226 (5)	1·238 (8)
O(3)—C(2)	1·280 (5)	1·298 (8)
O(4)—C(2)	1·218 (7)	1·215 (8)
N(1)—C(3)	1·334 (5)	1·344 (8)
N(1)—C(7)	1·333 (5)	1·330 (8)
C(1)—C(3)	1·507 (6)	1·482 (9)
C(2)—C(7)	1·512 (6)	1·500 (9)
C(3)—C(4)	1·390 (6)	1·383 (8)
C(4)—C(5)	1·374 (7)	1·38 (1)
C(5)—C(6)	1·378 (7)	1·39 (1)
C(6)—C(7)	1·386 (6)	1·390 (8)
C(8)—C(13)		1·52 (1)
C(9)—C(10)		1·44 (2)
C(10)—C(11)		1·45 (2)
C(11)—C(12)		1·43 (2)
C(13)—C(14)		1·47 (1)
C(14)—C(15)		1·48 (1)
O(4)···O(6)	2·905 (7)	
O(6)···O(5^{ii})	2·691 (6)	
N(1)—Sn(1)—C(9)	94·3 (2)	96·8 (2)
N(1)—Sn(1)—C(8)	95·7 (2)	97·5 (2)
O(5)—Sn(1)—C(9)	89·7 (2)	85·1 (2)
O(5)—Sn(1)—C(8)	87·2 (2)	88·0 (2)
O(5)—Sn(1)—N(1)	146·3 (1)	149·7 (2)
O(3)—Sn(1)—C(9)	97·1 (2)	97·7 (3)
O(3)—Sn(1)—C(8)	95·1 (2)	96·9 (2)
O(3)—Sn(1)—N(1)	70·9 (1)	71·7 (2)
O(3)—Sn(1)—O(5)	75·4 (1)	78·0 (2)
O(1)—Sn(1)—C(9)	87·2 (2)	87·8 (2)

	(I)	(II)
O(1)—Sn(1)—C(8)	88·1 (2)	87·6 (2)
O(1)—Sn(1)—N(1)	67·1 (1)	68·2 (2)
O(1)—Sn(1)—O(5)	146·6 (1)	142·1 (2)
O(1)—Sn(1)—O(3)	138·0 (1)	139·9 (2)
C(8)—Sn(1)—C(9)	166·2 (2)	162·2 (3)
O(1)—Sn(1)—O(1^{i})	65·1 (1)	65·1 (1)
N(1)—Sn(1)—O(1^{i})	133·3 (2)	133·3 (2)
O(3)—Sn(1)—O(1^{i})	155·0 (1)	155·0 (1)
O(5)—Sn(1)—O(1^{i})	77·0 (1)	77·0 (1)
C(8)—Sn(1)—O(1^{i})	82·1 (2)	82·1 (2)
C(9)—Sn(1)—O(1^{i})	80·4 (2)	80·4 (2)
Sn(1)—O(1)—C(1)	118·8 (3)	118·5 (4)
Sn(1)—O(3)—C(2)	122·1 (3)	121·1 (4)
Sn(1)—N(1)—C(7)	117·0 (3)	116·5 (4)
Sn(1)—N(1)—C(3)	122·3 (3)	121·1 (4)
C(3)—N(1)—C(7)	120·6 (4)	122·4 (5)
O(1)—C(1)—O(2)	126·6 (4)	126·2 (6)
O(2)—C(1)—C(3)	117·9 (4)	118·0 (5)
O(1)—C(1)—C(3)	115·5 (4)	115·8 (5)
O(3)—C(2)—O(4)	125·8 (4)	125·6 (6)
O(4)—C(2)—C(7)	118·9 (4)	120·6 (6)
O(3)—C(2)—C(7)	115·3 (4)	113·8 (5)
N(1)—C(3)—C(1)	116·0 (4)	116·2 (5)
C(1)—C(3)—C(4)	123·4 (4)	124·5 (6)
N(1)—C(3)—C(4)	120·6 (4)	119·3 (6)
C(3)—C(4)—C(5)	119·6 (4)	119·2 (6)
C(4)—C(5)—C(6)	118·9 (5)	120·8 (6)
C(5)—C(6)—C(7)	119·3 (5)	117·4 (6)
C(2)—C(7)—C(6)	124·4 (4)	123·9 (6)
N(1)—C(7)—C(6)	120·9 (4)	121·0 (6)
N(1)—C(7)—C(2)	114·6 (4)	115·2 (5)
Sn(1)—C(8)—C(13)		116·9 (5)
Sn(1)—C(9)—C(10)		115·9 (6)
C(9)—C(10)—C(11)		121 (1)
C(10)—C(11)—C(12)		115 (1)
C(8)—C(13)—C(14)		114·1 (7)
C(13)—C(14)—C(15)		113·9 (8)
O(4)···O(6)···O(5^{ii})	119·8 (2)	

Symmetry operations: (i) $-x, -y, -z$; (ii) $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$.

Sn—O—Sn bridge formation links a second water molecule *via* a hydrogen bond [O(4)···O(6) = 2·905 (6) Å]. In this context it is interesting to note that the density of (I) is appreciably higher than that of (II), which, like $Ph_2SnL(H_2O)$ (Gielen *et al.*, 1987), does not contain a second water molecule per R_2SnL unit. It is inferred that this second H_2O may contract the lattice in the case of the Me_2Sn compound by hydrogen bonding, while a similar contraction is prevented by the more voluminous R groups in (II) and $Ph_2SnL(H_2O)$.

In (II) a short intermolecular distance O(4)···O(5) ($\bar{y}$, $-\frac{1}{2}+x$, $\frac{1}{2}+z$) of 2·707 (7) Å exists, indicating hydrogen bridging between the dimeric units.

The Sn—N distance in (I) and in (II) is significantly shorter than in the polymeric $Ph_2SnL(H_2O)$ [2·338 (3) Å; Gielen *et al.*, 1987], which has a very similar environment around Sn. In other comparable R_2Sn compounds the Sn—N distance has also been found to be slightly larger (see Gielen *et al.*, 1987). The distance Sn—O(5) is distinctly longer than in $Ph_2Sn(H_2O)L$ [2·271 (3) Å; Gielen *et al.*, 1987] and in $[Bu_3Sn(OH_2)_2]^+$ [2·326 (5) and 2·295 (4) Å; Davies, Goddard, Hursthouse & Walker, 1986] but significantly shorter than in $Me_3SnO_2CCH_2NMe_2.H_2O$ (2·386 Å; Jurkschat, Tzschach, Zschunke, Grand & Albrand, 1987).

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References

Davies, A. G., Goddard, J. P., Hursthouse, M. B. & Walker, N. P. C. (1986). *J. Chem. Soc. Dalton Trans.* pp. 1873–1877.

Frenz, B. A. (1985). *Enraf–Nonius Structure Determination Package, SDP-Plus*, V3.0. Enraf–Nonius, Delft, The Netherlands.

Gielen, M., Joosen, E., Mancilla, T., Jurkschat, K., Willem, R., Roobol, C., Bernheim, J., Atassi, G., Huber, F., Hoffmann, E., Preut, H. & Mahieu, B. (1987). *Main Group Met. Chem.* **10**, 147–167.

International Tables for X-ray Crystallography (1974). Vol. IV, Tables 2.2B and 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Jurkschat, K., Tzschach, A., Zschunke, A., Grand, A. & Albrand, J. P. (1987). Ref. 21 in Gielen, M. *et al.*, (1987).

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Nardelli, M. (1982). *PARST. A System of Computer Routines for Calculating Molecular Parameters from Results of Crystal Structure Analysis.* Univ. of Parma, Italy.

Sheldrick, G. M. (1987). *SHELXTL-Plus* (*Release* 2) *for Nicolet R3m/V Crystallographic Systems for Solving, Refining, and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen, Federal Republic of Germany.

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Structure of Bis(*μ*-pyridine-2-thione-*μ*-*S*)-bis[iodo(pyridine-2-thione-*S*)copper(I)]

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Abstract. $[Cu(C_5H_5NS)_2I]_2$, $M_r = 825·55$, monoclinic, $C2/c$, $a = 13·139$ (2), $b = 9·888$ (1), $c = 20·405$ (4) Å, $\beta = 94·92$ (1)°, $V = 2641·1$ (7) Å^3, $Z = 4$, $D_m = 2·065$, $D_x = 2·076$ Mg m^{-3}, m.p. 323 K, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 4·321$ mm^{-1}, $F(000) = 1583·9$, $T = 296$ (2) K. Final R value 0·0235 for 1767 observed reflections with $F_o \geq 3·0\sigma(F_o)$. The structure consists of dimers, $[Cu(C_5H_5NS)_2I]_2$, each having a twofold symmetry axis and a $(CuIS_2)_2$ core in *cis* conformation stabilized by two strong intramolecular I···H—N hydrogen bonds. Each Cu atom has a distorted tetrahedral coordination with one terminal and two bridging S atoms and a terminal I atom. The two tetrahedra are joined along the S(2)···S(2′) edge. The Cu···Cu separation is 3·139 Å.

Introduction. Complexes of copper with pyridine-2-thione (py2SH) have been known for some time and continue to be investigated (Raper, 1985). Recently the structures of copper halide complexes $[Cu(py2SH)_3]_2X_2$, (X = Cl, Br), consisting of centrosymmetric dimers (Stergioudis, Kokkou, Rentzeperis & Karagiannidis, 1987), have been reported. Since the stereochemistry of Cu^I with soft ligands is very flexible, it was deemed worthwhile to carry out also the X-ray

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