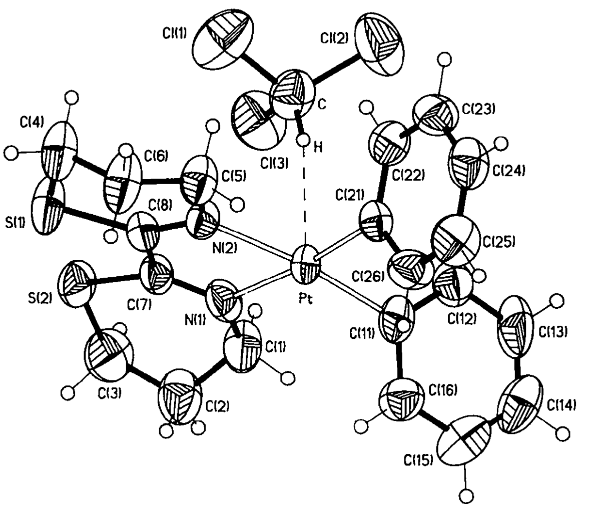

Fig. 1. Molecular structure with the atom-numbering scheme.

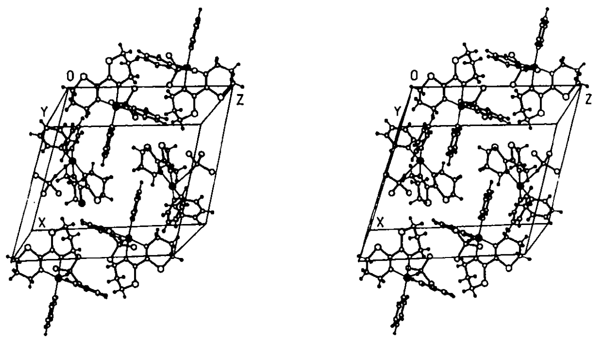

Fig. 2. A stereoscopic view of the packing.

chemically different. In ring *A*, five atoms, N(2), C(8), S(1), C(4) and C(5), are almost coplanar, while C(6) lies 0·63 Å out of this plane; in ring *B*, C(2) lies 0·62 Å out of the best plane through N(1), C(7), S(2), C(1) and C(3).

Bond distances within the rings are as expected, though it is noticeable that S(2)—C(7) and S(1)—C(8) [1·748 (7) and 1·735 (7) Å] are significantly shorter than S(2)—C(3) and S(1)—C(4) [1·79 (1) and 1·80 (1) Å]. The values, together with the value of 1·48 (1) Å for the C(7)—C(8) single-bond distance suggest a small degree of multiple-bond delocalization over the S—C—N system. There is a small twist around the C(7)—C(8) bond connecting the two six-membered rings [N(1)—C(7)—C(8)—N(2) torsion angle = 7·1 (9)°].

The two rings are not coplanar because of a strong S(1)···S(2) (3·005 Å) intramolecular contact which justifies the *trans* configuration observed in the free ligand (Huffman & Sattelberger, 1981). An intermolecular interaction between the H atom of chloroform and the metal atom may be present. The carbon to platinum separation is 3·44 Å. The Pt···H separation (for the H atom in calculated position) of 2·48 Å is in the range found for weak intramolecular *M*···H interactions (Crabtree, Holt, Lavin & Morehouse, 1985).

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Structure of Di(4-picolinium) Pentachloro(phenyl)stannate(IV)

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Abstract. $2C_6H_8N^+.[Sn(C_6H_5)Cl_5]^{2-}$, $M_r = 561{\cdot}34$, monoclinic, $P2_1/c$, $a = 14{\cdot}527\ (3)$, $b = 8{\cdot}454\ (2)$, $c = 18{\cdot}367\ (4)$ Å, $\beta = 94{\cdot}04\ (1)°$, $V = 2250{\cdot}06$ Å^3, $Z = 4$, $D_m = 1{\cdot}67$ Mg m^{-3}, $D_x = 1{\cdot}66$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 1602$ m^{-1}, $F(000) = 1112$, $T = 296$ K, final $R = 0{\cdot}027$ for 2724 unique reflexions. The title

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compound crystallizes with a distorted K_2PtCl_6-type structure. The mean Sn—Cl bond length of the pentachloro(phenyl)stannate anion is about 0·06 Å longer than the Sn—Cl bond length of hexachlorostannates, which is a result of the stronger electron-donation ability of $C_6H_5^-$ compared with Cl^-. The differences in Sn—Cl bond lengths within the $(C_6H_5)SnCl_5^{2-}$ anion are determined by hydrogen bonding. A definite *cis* or *trans* influence of the electron donor $C_6H_5^-$ could not be observed.

Introduction. The *trans* influence of ligands is one of the best examined effects in coordination chemistry. In hexacoordinated compounds $L'ML_5$, where M is a transition metal atom, a weakening of the M—L bond *trans* to L' is observed when L' is a stronger electron donor than L. The influence of L' on the *cis* bonds is much smaller in magnitude (Shusterovitch, Porai-Koshits & Buslaev, 1975).

For hexacoordinated coordination compounds of tin in its highest oxidation state (+IV), the opposite effect is observed. X-ray diffraction and ^{35}Cl NQR results show that the electron donor $C_2H_5^-$ weakens the *cis* Sn—Cl bonds in $A_2EtSnCl_5$ compounds (A = univalent cation), while the effect on the *trans* Sn—Cl bond is much smaller in magnitude and probably of opposite sign (Storck & Weiss, 1989). This is seen by an increase of the Sn—Cl bond length, a decrease of the ^{35}Cl NQR frequency for the *cis* Cl atoms, a decrease of the Sn—Cl bond length and an increase of the ^{35}Cl NQR frequency for the *trans* Cl atoms of the pentachloro(ethyl)stannate(IV) compounds compared with hexachlorostannates.

The phenyl group $C_6H_5^-$ is also a stronger electron donor than Cl^-. The crystal structure of $(4\text{-}CH_3C_5H_4NH)_2[(C_6H_5)SnCl_5]$ is reported in this paper and the *cis* and *trans* influences are discussed.

Experimental. Colorless prisms of the title compound were obtained by slow evaporation of an aqueous solution of the stoichiometric amounts of 4-picoline, concentrated HCl, and $C_6H_5SnCl_3$. D_m = 1·67 Mg m^{-3}, measured pycnometrically with toluene as liquid. Crystal size 0·23 × 0·28 × 0·5 mm, Stoe–Stadi-4 four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, lattice parameters from setting of 60 reflexions with $7·45 \leq \theta \leq 21·3°$. Lorentz–polarization corrections, empirical absorption correction, min., max. transmission factors 0·5960, 0·7010; $(\sin\theta/\lambda)_{\max} = 0·5384$ Å^{-1}, $-15 \leq h \leq 5$, $0 \leq k \leq 9$, $-20 \leq l \leq 20$; three standard reflexions measured every 60 min showed no decay; ω–2θ scan. 4818 reflections measured, 2724 independent reflections with $F \geq 2\sigma(F)$, R_{int} = 0·015. Structure solved by direct methods with use of *SHELX76* (Sheldrick, 1976) refined by full-matrix least squares with minimization of $\sum w(|F_o| - |F_c|)^2$; $w = 1/\sigma^2(F_o)$. All H atoms except those of the methyl groups located in difference Fourier synthesis and refined with fixed isotropic temperature factors. 281 free parameters. Final R = 0·027, wR = 0·028 for 2724 reflexions with $F \geq 2\sigma(F)$, $(\Delta/\sigma)_{\max}$ = 0·007, $(\Delta/\sigma)_{\text{mean}}$ = 0·002, $(\Delta\rho)_{\max}$ = +0·49 e Å^{-3}, $(\Delta\rho)_{\min}$ = −0·34 e Å^{-3} in final difference Fourier synthesis. Atomic parameters are given in Table 1.* Bond lengths and angles are given in Table 2. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Table 1. *Positional and thermal parameters of di(4-picolinium)*$C_6H_5SnCl_5$; *equivalent isotropic thermal parameters for the non-*H *atoms* (Å^2 × 10^3) *with e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Sn(1)	0·2487 (1)	0·6512 (1)	0·1473 (1)	41 (1)
Cl(1)	0·2473 (1)	0·8340 (1)	0·0422 (1)	52 (1)
Cl(2)	0·4202 (1)	0·6505 (1)	0·1437 (1)	56 (1)
Cl(3)	0·2353 (1)	0·4276 (1)	0·0639 (1)	62 (1)
Cl(4)	0·0811 (1)	0·7027 (1)	0·1426 (1)	56 (1)
Cl(5)	0·2699 (1)	0·9058 (1)	0·2230 (1)	50 (1)
C(1)	0·2524 (3)	0·5091 (4)	0·2442 (2)	42 (3)
C(2)	0·3100 (4)	0·3827 (6)	0·2524 (3)	63 (4)
C(3)	0·3125 (5)	0·2897 (6)	0·3143 (3)	77 (6)
C(4)	0·2566 (4)	0·3183 (7)	0·3688 (3)	75 (5)
C(5)	0·1970 (5)	0·4453 (8)	0·3614 (2)	81 (5)
C(6)	0·1951 (4)	0·5402 (6)	0·2988 (2)	62 (4)
N(1)	−0·1122 (3)	0·6139 (5)	0·3633 (4)	79 (5)
C(7)	−0·0888 (4)	0·7005 (7)	0·3075 (3)	72 (4)
C(8)	−0·0183 (4)	0·8039 (6)	0·3170 (3)	63 (4)
C(9)	0·0287 (3)	0·8235 (5)	0·3839 (2)	53 (3)
C(10)	−0·0004 (4)	0·7328 (7)	0·4405 (3)	66 (4)
C(11)	−0·0702 (5)	0·6312 (7)	0·4291 (4)	80 (5)
C(12)	0·1077 (3)	0·9391 (6)	0·3946 (3)	83 (5)
N(2)	0·5792 (3)	0·5515 (5)	0·3898 (3)	79 (4)
C(13)	0·5801 (4)	0·6204 (7)	0·4541 (3)	80 (4)
C(14)	0·5154 (4)	0·7317 (6)	0·4649 (3)	72 (4)
C(15)	0·4510 (3)	0·7769 (5)	0·4100 (2)	50 (3)
C(16)	0·4552 (4)	0·7000 (7)	0·3448 (3)	72 (4)
C(17)	0·5188 (4)	0·5897 (7)	0·3348 (3)	84 (5)
C(18)	0·3802 (3)	0·9051 (6)	0·4216 (3)	73 (4)
H(1)*	0·339 (3)	0·359 (5)	0·217 (2)	
H(2)*	0·351 (3)	0·222 (5)	0·317 (2)	
H(3)*	0·263 (3)	0·237 (5)	0·415 (2)	
H(4)*	0·143 (3)	0·458 (5)	0·396 (2)	
H(5)*	0·157 (3)	0·620 (5)	0·293 (2)	
H(6)*	−0·152 (3)	0·557 (6)	0·360 (2)	
H(7)*	−0·124 (3)	0·694 (5)	0·258 (2)	
H(8)*	−0·002 (3)	0·874 (5)	0·280 (2)	
H(9)*	0·024 (3)	0·757 (5)	0·486 (2)	
H(10)*	−0·092 (3)	0·573 (5)	0·461 (2)	
H(11)*	0·616 (3)	0·483 (5)	0·385 (2)	
H(12)*	0·627 (3)	0·584 (5)	0·497 (2)	
H(13)*	0·517 (3)	0·790 (5)	0·508 (2)	
H(14)*	0·419 (3)	0·721 (5)	0·309 (2)	
H(15)*	0·525 (3)	0·545 (5)	0·288 (2)	

* H atoms were given fixed isotropic temperature factors of 0·06 Å^2.

Discussion. Fig. 2 shows a projection of the unit cell of $(4\text{-}CH_3C_5H_4NH)_2[(C_6H_5)SnCl_5]$ onto the *ac* plane. The numbering scheme is given in Fig. 1. It is

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52480 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Intramolecular and intermolecular distances (Å) and angles (°) with e.s.d.'s in parentheses*

Sn(1)—Cl(1)	2·473 (1)	Cl(1)—Sn(1)—Cl(2)	86·2 (1)
Sn(1)—Cl(2)	2·497 (1)	Cl(1)—Sn(1)—Cl(3)	89·9 (1)
Sn(1)—Cl(3)	2·433 (1)	Cl(1)—Sn(1)—Cl(4)	84·7 (1)
Sn(1)—Cl(4)	2·469 (1)	Cl(1)—Sn(1)—Cl(5)	83·9 (1)
Sn(1)—Cl(5)	2·570 (1)	Cl(2)—Sn(1)—Cl(3)	91·0 (1)
Sn(1)—C(1)	2·145 (4)	Cl(2)—Sn(1)—Cl(4)	169·4 (1)
		Cl(2)—Sn(1)—Cl(5)	86·2 (1)
		Cl(3)—Sn(1)—Cl(4)	94·6 (1)
		Cl(3)—Sn(1)—Cl(5)	173·3 (1)
		Cl(4)—Sn(1)—Cl(5)	87·2 (1)
		Cl(2)—Sn(1)—C(1)	93·1 (1)
		Cl(3)—Sn(1)—C(1)	94·8 (1)
		Cl(4)—Sn(1)—C(1)	95·4 (1)
		Cl(5)—Sn(1)—C(1)	91·4 (1)
N(1)···Cl(1)	3·598 (6)	N(1)···H(6)···Cl(1)	137 (4)
H(6)···Cl(1)	3·01 (5)		
N(1)···Cl(4)	3·508 (5)	N(1)···H(6)···Cl(4)	112 (4)
H(6)···Cl(4)	3·16 (5)		
N(1)···Cl(5)	3·216 (5)	N(1)···H(6)···Cl(5)	149 (5)
H(6)···Cl(5)	2·55 (4)		
N(2)···Cl(1)	3·295 (5)	N(2)···H(11)···Cl(1)	142 (4)
H(11)···Cl(1)	2·64 (4)		
N(2)···Cl(2)	3·445 (5)	N(2)···H(11)···Cl(2)	127 (4)
H(11)···Cl(2)	2·90 (4)		
N(2)···Cl(5)	3·355 (5)	N(2)···H(11)···Cl(5)	134 (4)
H(11)···Cl(5)	2·76 (4)		

possible to transform the unit cell of (4-$CH_3C_5H_4$-$NH)_2[(C_6H_5)SnCl_5]$ into a distorted K_2PtCl_6-type unit cell. The $(C_6H_5)SnCl_5^{2-}$ ions form a distorted face-centered cell with the cations occupying the tetrahedral holes, whereby a cubic environment of eight cations around the anions results. (4-CH_3-$C_5H_4NH)_2[(C_6H_5)SnCl_5]$ is no exception to the series of hexacoordinated tin compounds A_2SnCl_6 and A_2RSnCl_5 (A = univalent cation) which crystallize in K_2PtCl_6 analogue structures. Three crystallographically inequivalent aromatic ring systems are found in the crystal structure – the phenyl group of the $(C_6H_5)SnCl_5^{2-}$ anion and two 4-picolinium cations. All three are nearly planar with a sum of the bond angles of 720·0 (5)° for the phenyl ring, 719·9 (5)° for 4-picolinium (I), and 720·1 (5)° for 4-picolinium (II) and a maximum deviation from the best plane of 0·004 Å for the phenyl ring, 0·013 Å for 4-picolinium (I), and 0·009 Å for 4-picolinium (II). The normals to the best planes of the two crystallographically inequivalent picolinium cations are nearly perpendicular (89·7°).

The existence of hydrogen bonds leads to another view of the crystal structure. Two 4-picolinium cations are associated with each $(C_6H_5)SnCl_5^{2-}$ anion. Hydrogen bonds occur only within this group of three ions, so that the crystal structure can be regarded as composed of discrete (4-$CH_3C_5H_4NH)_2$-$[(C_6H_5)SnCl_5]$ building blocks. The N atoms of the 4-picolinium rings are positioned at the triangle which is built up by three neighboring Cl atoms of the anion.

The strength of the Sn—Cl bonds is reflected by their length. For a given octahedral anion $[SnCl_6^{2-}$, $(C_2H_5)SnCl_5^{2-}$, $(C_6H_5)SnCl_5^{2-}]$ surrounded by different cations the mean Sn—Cl bond length is nearly constant (Table 3). No cation influence on the mean properties of the anion is observed. The mean bond strength is determined by the donor strength of the ligands, showing that $C_2H_5^-$ and $C_6H_5^-$ are stronger donors than Cl^-.

Although the donor activity of $C_6H_5^-$ and $C_2H_5^-$ does not differ much, one observes for the (C_2H_5)-$SnCl_5^{2-}$ compounds that only the *cis* Sn—Cl bonds

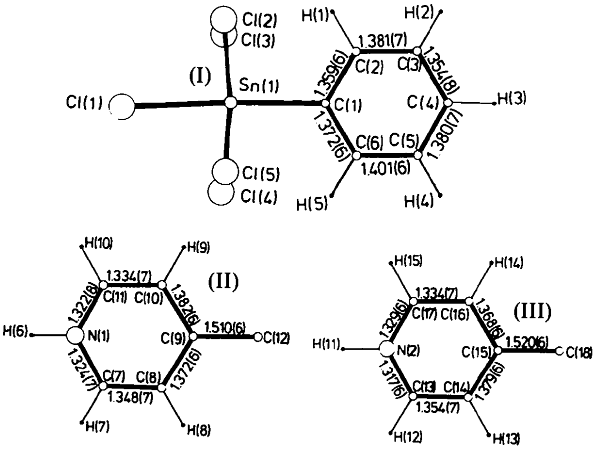

Fig. 1. Atomic numbering scheme as used in Table 1. The molecules are labeled (I), (II) and (III). C—C and C—N bond distances are given with e.s.d.'s in parentheses.

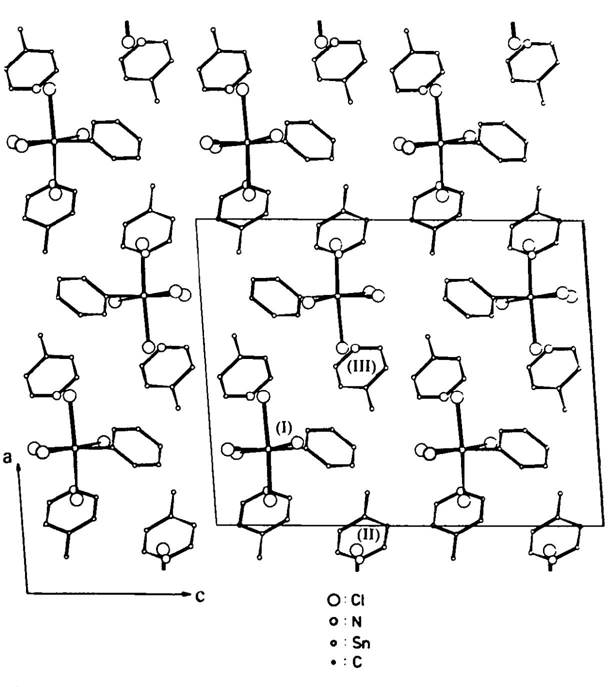

Fig. 2. Projection of the unit cell of $(4-CH_3C_5H_4NH)_2[(C_6H_5)$-$SnCl_5]$ along the *b* axis onto the *ac* plane. The molecules labeled (I), (II) and (III) are shown in Fig. 1.

Table 3. *Mean* Sn—Cl *bond distances* (Å) *in hexacoordinated chlorostannate*(IV) *compounds*

Compound	All Sn—Cl bonds	*cis* Sn—Cl bonds	*trans* Sn—Cl bonds	Reference
$[(CH_3)_4N]_2SnCl_6$	2·424 (3)	—	—	(*a*)
$[C_5H_5NH]_2SnCl_6$	2·431 (1)	—	—	(*b*)
$[4\text{-}ClC_5H_4NH]_2SnCl_6$	2·432 (4)	—	—	(*c*)
$[(CH_3)_4N]_2(C_2H_5)SnCl_5$	2·495 (4)	2·516 (3)	2·410 (4)	(*d*)
$[C_5H_5NH]_2(C_2H_5)SnCl_5$	2·495 (2)	2·519 (1)	2·400 (2)	(*d*)
$[CH_3NH_3]_2(C_2H_5)SnCl_5$	2·502 (1)	2·522 (1)	2·426 (1)	(*d*)
$[4\text{-}CH_3C_5H_4NH]_2(C_2H_5)SnCl_5$	2·500 (1)	2·526 (1)	2·397 (1)	(*d*)
$[4\text{-}CH_3C_5H_4NH]_2(C_6H_5)SnCl_5$	2·488 (1)	2·492 (1)	2·473 (1)	(*e*)

References: (*a*) Nielsen & Berg (1980); (*b*) Borchers & Weiss (1987); (*c*) Gearhart, Brill, Welsh & Wood (1973); (*d*) Storck & Weiss (1989); (*e*) this work.

are weakened through the influence of the $C_2H_5^-$ group, while the *trans* Sn—Cl bonds are slightly shorter than in $SnCl_6^{2-}$ compounds (‘*trans*-strengthening and ‘*cis*-weakening’). Within the $(C_6H_5)SnCl_5^{2-}$ anion of $(4\text{-}CH_3C_5H_4NH)_2[(C_6H_5)SnCl_5]$, *cis* and *trans* bonds are weakened. This may be a result of hydrogen bonding. In none of the four $(C_2H_5)SnCl_5^{2-}$ compounds of Table 3 were short $N\cdots Cl_{trans}$ distances observed (only the Cl atoms, *cis* to the ethyl group, are hydrogen bonded), while in $(4\text{-}CH_3C_5H_4NH)_2[(C_6H_5)SnCl_5]$, the *trans* Cl atom has N···Cl distances of 3·295 and 3·598 Å. This can be compared with Cl(5), where N···Cl(5) distances of 3·216 and 3·355 Å are accompanied by a lengthening of 0·078 Å over the mean *cis* Sn—Cl bond length. Therefore, hydrogen bonds may lengthen the *trans* Sn—Cl bond by up to 0·06 Å – enough to compensate for the *trans* influence of the $C_6H_5^-$ group.

The ^{35}Cl NQR signals observed for $(4\text{-}CH_3C_5H_4NH)_2[(C_6H_5)SnCl_5]$ are weak. Only two of the expected five signals showed a sufficient intensity to be recognized unambiguously as NQR resonances. The ^{35}Cl NQR frequencies are 15·92 and 14·66 MHz at room temperature.

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Structure of Bis{*N*-[2-(2-pyridyl)ethyl]-3-ethoxysalicylideneaminato}copper(II)

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Abstract. $[Cu(C_{16}H_{17}N_2O_2)_2]$, $M_r = 602{\cdot}19$, monoclinic, $P2_1/n$, $a = 13{\cdot}246\,(5)$, $b = 7{\cdot}155\,(3)$, $c = 15{\cdot}123\,(4)$ Å, $\beta = 92{\cdot}25\,(4)°$, $V = 1432{\cdot}2$ Å^3, $Z = 2$, $D_x = 1{\cdot}397$ g cm^{-3}, $\lambda(Cu\ K\alpha) = 1{\cdot}54184$ Å, $\mu = 13{\cdot}950$ cm^{-1}, $F(000) = 630$, $T = 293$ K. The final R value is 0·045 for 1896 significant $[I > 3\sigma(I)]$ reflections. In the centrosymmetric compound the Cu atom is bonded to an imine N atom and a phenolate

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