It has been suggested that the tendency towards formation of tetrahedrally coordinated species is due to the steric effects when *sec* alkyl groups are substituted on the N atoms of salicylaldimine (Sacconi, Paoletti & Ciampolini, 1963). However, substituents in the 3 and 5 positions of salicylaldimine inexplicably restore the planar coordination of the Ni^{II} ion.

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Structure of Tetramesityldistibane

By A. H. Cowley, C. M. Nunn and D. L. Westmoreland

Department of Chemistry, University of Texas at Austin, Austin, TX 78712-1167, *USA*

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Abstract. Tetra(2,4,6-trimethylphenyl)distibane, $C_{36}H_{44}Sb_2$, $M_r = 720{\cdot}3$, monoclinic, $P2_1/n$, $a = 12{\cdot}057\,(2)$, $b = 15{\cdot}992\,(2)$, $c = 17{\cdot}700\,(2)$ Å, $\beta = 99{\cdot}80\,(1)°$, $V = 3362{\cdot}9$ Å^3, $Z = 4$, $D_x = 1{\cdot}42$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71073$ Å, $\mu = 16{\cdot}4$ cm^{-1}, $F(000) = 1448$, $T = 296\,(1)$ K, $R = 0{\cdot}040$ for 3396 unique observed reflections. The crystal structure comprises discrete molecules which adopt an *anti* conformation of the non-bonding electron pairs. Molecular packing considerations preclude any extended interaction between Sb atoms of neighbouring molecules.

Introduction. In the course of our investigation into the reaction between mes_2SbLi (mes = mesityl = 2,4,6-trimethylphenyl) with group 11 and 12 chlorides we repeatedly obtained tetramesityldistibane together with reduction of the metal. In the light of previous structural studies of distibanes and their exhibition of thermochromic effects (Ashe, Butler & Diephouse, 1981), a study of the solid state structure and packing for this complex was carried out.

Experimental. Tetramesityldistibane was prepared by the oxidation of dimesitylstibido anion in the presence of CuCl. Yellow crystals were grown from hexane. A prism with approximate dimensions 0·25 × 0·30 × 0·30 mm was mounted under argon in a Lindemann capillary, and placed on an Enraf–Nonius CAD-4 diffractometer, using graphite-monochromated Mo $K\alpha$ X-radiation. Cell parameters were refined for 25 reflections within the range $26 \leq 2\theta \leq 30°$, measured by the diagonal slit method of centring. Data were collected at 296 (1) K, using ω–2θ scan mode for $3 \leq 2\theta \leq 48°$, variable scan rate 2–10° min^{-1} in ω, with scan width = $(0{\cdot}8 + 0{\cdot}35\tan\theta)°$. Range of *hkl* from 0, 0, −20 to 13, 18, 19. Two standard reflections were remeasured every 3600 s of X-ray exposure (no significant variation), and two orientation standards after every 100 reflections collected with a recalculation of the orientation matrix after any significant angular deviation. Empirical absorption correction applied to data, transmission coefficient range 0·94–1·00 (av. 0·98). Total reflections measured 5536, 5260 unique (averaged for observed and accepted reflections; 1·5 and 1·4% based on I and F_o respectively). 3396

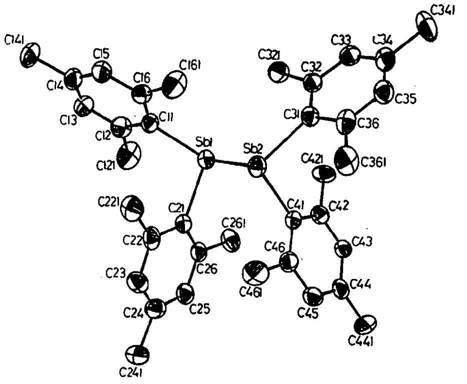

Fig. 1. The structure of tetramesityldistibane. Thermal motion is shown by 30% probability ellipsoids.

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Table 1. *Positional parameters and their estimated standard deviations*

	x	y	z	B(Å^2)*
Sb1	0·21043 (5)	0·25438 (4)	0·42716 (3)	4·31 (1)
Sb2	0·03247 (5)	0·19851 (4)	0·50178 (3)	4·50 (1)
C11	0·2171 (7)	0·1728 (5)	0·3281 (5)	4·4 (2)
C12	0·3164 (8)	0·1798 (6)	0·2986 (5)	5·7 (2)
C13	0·3334 (8)	0·1318 (7)	0·2352 (5)	6·3 (3)
C14	0·2515 (9)	0·0774 (6)	0·2003 (5)	6·0 (2)
C15	0·1516 (8)	0·0717 (6)	0·2272 (5)	5·7 (2)
C16	0·1327 (7)	0·1208 (6)	0·2902 (5)	5·2 (2)
C21	0·3336 (7)	0·1942 (5)	0·5152 (5)	4·3 (2)
C22	0·3515 (7)	0·1056 (6)	0·5209 (5)	5·4 (2)
C23	0·4386 (8)	0·0750 (6)	0·5795 (5)	5·9 (2)
C24	0·5029 (8)	0·1295 (7)	0·6305 (5)	6·1 (3)
C25	0·4856 (7)	0·2139 (6)	0·6256 (5)	5·4 (2)
C26	0·3999 (7)	0·2463 (6)	0·5677 (5)	4·7 (2)
C31	−0·1165 (6)	0·2775 (5)	0·4652 (5)	4·3 (2)
C32	−0·1348 (7)	0·3347 (5)	0·4053 (5)	4·5 (2)
C33	−0·2338 (7)	0·3824 (6)	0·3944 (5)	5·2 (2)
C34	−0·3159 (8)	0·3717 (6)	0·4381 (6)	5·6 (2)
C35	−0·3025 (8)	0·3110 (6)	0·4951 (6)	5·6 (2)
C36	−0·2023 (8)	0·2652 (6)	0·5104 (6)	5·4 (2)
C41	0·0971 (7)	0·2675 (5)	0·6063 (4)	4·1 (2)
C42	0·0985 (7)	0·3550 (5)	0·6134 (5)	4·3 (2)
C43	0·1337 (7)	0·3935 (6)	0·6859 (5)	4·4 (2)
C44	0·1714 (7)	0·3474 (6)	0·7507 (5)	4·9 (2)
C45	0·1748 (8)	0·2595 (6)	0·7428 (5)	5·7 (2)
C46	0·1374 (8)	0·2194 (6)	0·6711 (5)	4·9 (2)
C121	0·4131 (9)	0·2371 (8)	0·3368 (7)	8·6 (3)
C141	0·271 (1)	0·0227 (7)	0·1324 (5)	7·6 (3)
C161	0·0178 (8)	0·1088 (8)	0·3176 (6)	7·8 (3)
C221	0·2788 (9)	0·0417 (6)	0·4695 (6)	6·9 (3)
C241	0·598 (1)	0·0909 (8)	0·6904 (6)	8·4 (3)
C261	0·3843 (8)	0·3406 (6)	0·5678 (6)	6·0 (2)
C321	−0·0544 (8)	0·3511 (7)	0·3489 (5)	5·8 (2)
C341	−0·4246 (9)	0·4276 (7)	0·4218 (7)	8·0 (3)
C361	−0·1901 (8)	0·2018 (8)	0·5763 (6)	7·2 (3)
C421	0·0650 (9)	0·4136 (5)	0·5452 (5)	5·6 (2)
C441	0·2075 (9)	0·3864 (7)	0·8284 (5)	6·6 (3)
C461	0·148 (1)	0·1242 (6)	0·6678 (6)	7·9 (3)

* Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $\frac{4}{3}(a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ab\beta_{12}\cos\gamma + ac\beta_{13}\cos\beta + bc\beta_{23}\cos\alpha)$.

Table 2. *Selected bond lengths* (Å) *and angles* (°), *and torsion angles* (°) *for tetramesityldistibane*

Numbers in parentheses are estimated standard deviations in the least significant digits.

Sb1—Sb2	2·8481 (9)	Sb2—Sb1—C11	108·4 (2)
Sb1—C11	2·198 (8)	Sb2—Sb1—C21	90·3 (2)
Sb1—C21	2·185 (8)	C11—Sb1—C21	101·2 (3)
Sb2—C31	2·201 (8)	Sb1—Sb2—C31	109·0 (2)
Sb2—C41	2·181 (8)	Sb1—Sb2—C41	92·6 (2)
		C31—Sb2—C41	96·6 (3)
C11—Sb1—Sb2—C31	−99·11 (32)	C21—Sb1—Sb2—C31	158·96 (31)
C11—Sb1—Sb2—C41	162·96 (31)	C21—Sb1—Sb2—C41	61·03 (30)

reflections with $F \geq 3\sigma F$ used for structure refinement.

Structure solved by Patterson and difference syntheses, full-matrix least-squares refinement of F. All non-H atoms were anisotropically refined. Scattering factors including f' and f'' were taken from *International Tables for X-ray Crystallography* (1974); calculations using a VAX computer and Enraf–Nonius (1984) *SDP*/VAX programs. Final refinement included 343 variables, $R = 0{\cdot}040$, $wR = 0{\cdot}061$, $w = 4F_o^2/[\sigma^2_{\text{counting}} + (0{\cdot}04F_o^2)^2]$, max. shift/e.s.d. = 0·02, max. and min. difference Fourier map excursions 0·6 (1) and 0·0 (1) e Å^{-3}.

Discussion. Final parameters are presented in Table 1.* Tetramesityldistibane exists as discrete molecules with no short intramolecular contacts, as displayed in Fig. 1. The important bond lengths and angles are given in Table 2. The Sb1—Sb2 bond length of 2·8481 (9) Å is comparable with the value of 2·844 (1) Å determined for tetraphenyldistibane (Becker, Freudenblum & Witthauer, 1982). With respect to the Sb1—Sb2 bond the mesityl groups adopt a staggered conformation with an *anti* conformation of the non-bonding electron pairs. This conformation was similarly observed for the solid state structures of tetramesityldiphosphane (Baxter, Cowley, Davis & Riley, 1981) and tetraphenyldistibane (first reported by von Deuten & Rehder, 1980), which display twofold and $\bar{1}$ symmetry, respectively.

Table 2 gives the torsion angles for the central Sb_2C_4 core. C11—Sb1—Sb2—C31 gives a torsion angle of 99·11°, with the phenyl rings C11–C16 and C31–C36 lying almost coplanar (19·7 and 12·1° respectively) with the Sb—Sb vector, and close to orthogonal to one another (91·8°). C21—Sb1—Sb2—C41 gives a torsion angle of 61·03°, with the phenyl rings C21–C26 and C41–C46 lying at 72·5 and 68·7° with respect to the Sb—Sb vector, and close to parallel to one another (27·0°). Presumably this molecular orientation is a result of steric demands.

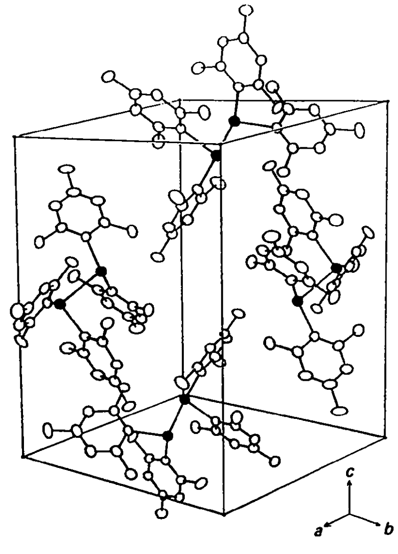

Fig. 2. Unit-cell packing diagram for tetramesityldistibane.

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* Lists of structure factors and anisotropic thermal parameters and complete lists of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52566 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The Sb—C bond lengths [2·181–2·201 (8) Å] show no significant variation dependent upon the mesityl ring orientation. The sum of the angles at Sb1 and Sb2 is 299·9 and 298·2° respectively, which is larger than the values observed for tetraphenyldistibane in which the sum of the angles at the Sb atoms was 284·7 (von Deuten & Rehder, 1980) and 285·2° (Becker, Freudenblum & Witthauer, 1982).

Fig. 2 displays the unit-cell packing for tetramesityldistibane. Thermochromic effects in distibane complexes have been seen to occur (*e.g.* Becker, Freudenblum & Witthauer, 1982; Ashe, Ludwig, Oleksyszyn & Huffman, 1984), where the Sb atoms are arranged in collinear chains with the Sb–Sb separations well below the van der Waals separation of 4·4 Å. For tetramesityldistibane the Sb2—Sb2 vectors are not collinear with neighbouring molecules, with the closest intramolecular contact between Sb atoms being 6·40 Å. This far exceeds the van der Waals separation and precludes the possibility of extended bonding between the Sb atoms of neighbouring molecules.

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Structure of 5-(2,2,6,6-Tetramethyl-1-oxyl-1,2,5,6-tetrahydropyridin-4-yl)penta-2,4-diyn-1-ol

By Vladimir A. Igonin, Valery E. Shklover* and Yuri T. Struchkov

A. N. Nesmeyanov Institute of Organoelement Compounds of the Academy of Sciences of the USSR,
28 Vavilov St, B-334, Moscow, USSR

and Olga L. Lazareva and Georgi A. Vinogradov

Institute of Chemical Physics of the Academy of Sciences of the USSR, 4 Kosygina St, B-334, Moscow, USSR

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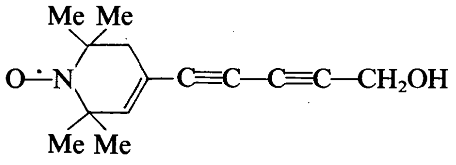
Abstract. $C_{14}H_{18}NO_2$, $M_r = 232$, monoclinic, $P2_1/n$, $a = 5{\cdot}760\,(1)$, $b = 22{\cdot}064\,(4)$, $c = 10{\cdot}520\,(1)$ Å, $\beta = 103{\cdot}98\,(1)°$, $V = 1297{\cdot}3\,(4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}19$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}86$ cm^{-1}, $F(000) = 125$, $T = 150$ K, $R = 0{\cdot}058$ for 1386 reflections. The six-membered heterocycle has a distorted half-chair conformation. The N atom of the paramagnetic centre is displaced from the plane of the three bonded atoms by 0·120 (3) Å. The N—O distance is 1·279 (4) Å, the N—C distances are 1·491 and 1·482 (4) Å. The molecules are linked by O—H···O 'head-to-tail' hydrogen bonds into infinite chains along [10$\bar{1}$] which in their turn form layers parallel to (010). Packing of the molecules in the crystal satisfies the conditions for solid state polymerization of diacetylenes; however, due to the fixation of molecular positions in the crystal by hydrogen bonds this particular compound is quite stable relative to the solid state polymerization.

Introduction. An X-ray structural study of 5-(2,2,6,6-tetramethyl-1-oxyl-1,2,5,6-tetrahydropyridin-4-yl)penta-2,4-diyn-1-ol (I), prepared by the conventional technique of the cross-coupling of monosubstituted acetylenes (Chodkiewich, 1957), has been carried out and the possibility of its solid state polymerization is discussed.



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* To whom correspondence should be addressed.