greater than 3·80 Å and, consequently, are essentially non-bonding. The range of the Mo—O distances is 2·002 (9)–2·108 (11) (av. 2·055 Å), 2·048 (9)–2·119 (10) (av. 2·082 Å) and 2·030 (10)–2·193 (10) (av. 2·089 Å) in the linear, rhomboidal and octahedral Mo chains, respectively.

The two crystallographically inequivalent Gd cations are located in tunnels formed along the **b** direction. Their environments are similar and consist of eight O atoms placed at the vertices of a distorted bicapped trigonal prism. The average Gd—O distances are 2·421 and 2·443 Å for the Gd(1) and Gd(2) sites respectively. The shortest distance between Gd atoms is 4·294 (1) Å [Gd(1)—Gd(2)].

By using the bond-length–bond-strength formula (Brown & Wu, 1976) for the Mo—O bonds [$s = (d_{\rm Mo—O}/1{\cdot}882)^{-6{\cdot}0}$], the valence of each independent Mo atom was determined as follows: Mo(1) +2·63, Mo(2) +2·64, Mo(3) +2·21, Mo(4) +2·16, Mo(5) +3·28, Mo(6) +3·23, Mo(7) +3·26, Mo(8) +3·27, Mo(9) +3·55 and Mo(10) +3·55. From these values, we can deduce an average Mo oxidation state in the infinite Mo_2, Mo_4 and Mo_6 chains of +3·55, +3·26 and +2·41 respectively (+3·76, +3·38 and +2·34 in $Ca_{5{\cdot}45}Mo_{18}O_{32}$). The calculated valence of the molybdenum in $Gd_4Mo_{18}O_{32}$ is +2·91 which is in good agreement with that based on the stoichiometry, +2·89.

Isostructural compounds are also formed with Nd, Sm, Gd, Tb, Dy, Er, Tm, Yb, Lu and Y as the ternary element (Gougeon & McCarley, 1985).

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Acta Cryst. (1991). C47, 2029–2032

Mesitylbis[2-pyridinethiolato(1 −)]antimony(III)

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(*Received* 16 *February* 1991; *accepted* 22 *April* 1991)

Abstract. [$Sb(C_{19}H_{19}N_2S_2)$], $M_r = 461{\cdot}24$, triclinic, $P\bar{1}$, $a = 8{\cdot}168\,(21)$, $b = 10{\cdot}015\,(8)$, $c = 12{\cdot}964\,(12)$ Å, $\alpha = 76{\cdot}74\,(8)$, $\beta = 85{\cdot}73\,(12)$, $\gamma = 69{\cdot}22\,(7)°$, $V = 965\,(3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}587$ Mg m^{-3}, $\lambda(\text{Ag } K\alpha) = 0{\cdot}56087$ Å, $\mu = 0{\cdot}86$ mm^{-1}, $F(000) = 460$, $T = 291\,(1)$ K, final $R = 0{\cdot}040$ for 4227 unique observed reflexions [$F \geq 2·0\sigma(F)$]. In the title compound two planar 2-pyridinethiolate ligands chelate Sb *via* S [Sb—S (mean) = 2·500 (5) Å] and N [Sb—N (mean) = 2·689 (6) Å] forming together with C(mesityl) a distorted tetragonal pyramid around Sb, and leaving in the remaining half-sphere opposite to C(mesityl) space for a stereochemically active lone pair. Two symmetrically equivalent molecules are arranged such that the free half-spheres are directed at each other leading to Sb—Sb′ contacts of 3·787 (5) Å. Angles around Sb: S(1)—Sb(1)—S(2) = 85·27 (6), N(1)—Sb(1)—N(2) = 153·35 (16), S(1)—Sb(1)—C(1) = 98·32 (11), S(2)—Sb(1)—C(1) = 103·24 (15)°.

Introduction. The structural effects and implications of the stereochemical activity of the lone pair in inorganic antimony(III) and organoantimony(III) compounds are still not understood fully. A specific problem in this respect is the formation of pairs of molecules showing intermolecular Sb···Sb′ interactions between the two molecules, in spite of the expected repulsion by the lone pairs. We report here the structure of mesitylbis[2-pyridinethiolato(1 −)]-antimony(III) as the first example of a mono-organoantimony(III) compound containing this type of Sb···Sb′ interaction.

Experimental. A solution of 4 mmol of sodium 2-pyridinethiolate (NaSP) (prepared from equimolar amounts of Na and HSP in ethanol) and 2 mmol of mesitylantimony dichloride in 60 ml ethanol was stirred for one day at 293 K. The precipitate was extracted with $CHCl_3$, and the title compound was crystallized by the addition of *n*-pentane to the extract. Yield 80%; m.p. 422 K; analysis (% found/calculated): C 49·2/49·5; H 4·0/4·2; N 6·0/6·1). Single crystals from $CHCl_3$/*n*-pentane. Approximate dimensions 0·29 × 0·13 × 0·06 mm. $\omega/2\theta$ scan, scan speed 1·1–1·7° min^{-1} in θ, scan width (1·2 + dispersion)°. Nonius CAD-4 diffractometer, graphite-monochromated Ag $K\alpha$ radiation; lattice parameters from least-squares fit with 25 reflexions up to 2θ = 26·2°; ω scans of low-order reflexions along the three crystal axes showed acceptable mosaicity; six standard reflexions ($\bar{2}\bar{2}6$, $22\bar{6}$, 261, $\bar{2}\bar{6}\bar{1}$, $\bar{2}26$, $2\bar{2}\bar{6}$) recorded every 2·5 h, only random deviations; 10 317 reflexions measured, $3·0 \leq 2 \leq 44·0°$, $-10 \leq h \leq 10$, $-13 \leq k \leq 13$, $-17 \leq l \leq 17$; after averaging 4805 unique reflexions, $R_{\rm int}$ = 0·037, 4227 reflexions with $F \geq 2·0\sigma(F)$; Lorentz–polarization correction and absorption correction *via* ψ scans, maximum/minimum transmission 1·00/0·93; structure solution by direct methods, ΔF syntheses and full-matrix least-squares refinement with anisotropic displacement factors for all non-H atoms and a common isotropic displacement factor for H atoms, which were placed in geometrically calculated positions (C—H 1·08 Å); refinement on F with 4227 reflexions and 218 refined parameters; $w = 1·0/[\sigma^2(F) + (0·005F^2)]$ which led to featureless analysis of variance in terms of sinθ and F_o; S = 0·72, R = 0·040, wR = 0·046; $(\Delta/\sigma)_{\rm max}$ = 0·09, no extinction correction; largest peak in final ΔF map ± 0·7 (3) e Å^{-3}; atomic scattering factors for neutral atoms and real and imaginary dispersion terms from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1.); programs: *Enraf–Nonius Structure Determination Package* (Frenz, 1985), *PARST* (Nardelli, 1983), *SHELX76* (Sheldrick, 1976), *SHELXTL-Plus* (Sheldrick, 1987), *PCK83* (Williams, 1984), *PLATON* (Spek, 1982), *MISSYM* (Le Page, 1987).

Discussion. The molecular structure of the title compound and the atom-numbering scheme are shown in Fig. 1 and a stereoscopic view of the unit cell is in Fig. 2. Positional parameters and the equivalent values of the anisotropic temperature factors for non-H atoms are given in Table 1,* bond lengths and

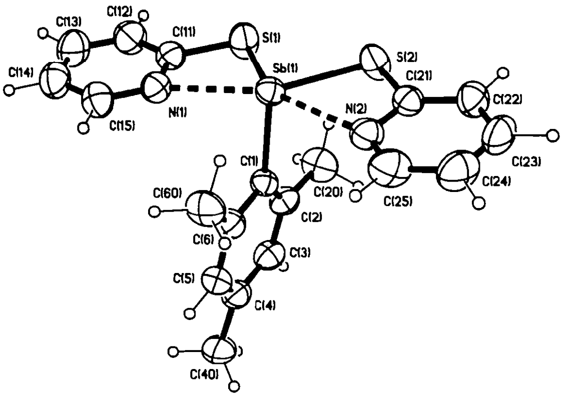

Fig. 1. General view (*SHELXTL-Plus* graphics) of the molecule, showing the atom-numbering scheme.

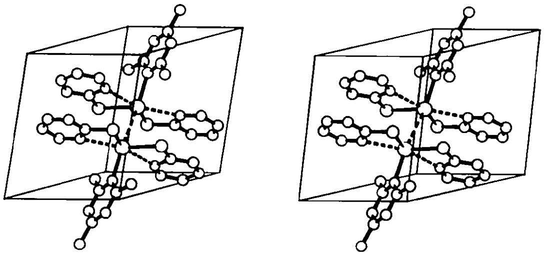

Fig. 2. Stereoscopic view (*SCHAKAL*; Keller, 1986) of the unit cell.

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* Lists of structure factors, anisotropic displacement and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54185 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates and equivalent isotropic displacement parameters* (Å^2 × 10^4)

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

	x	y	z	U_{eq}
Sb(1)	0·92212 (3)	0·61397 (3)	0·86299 (2)	421
S(1)	0·9162 (2)	0·4684 (1)	0·7304 (1)	613
S(2)	0·6222 (2)	0·6101 (1)	0·9101 (1)	580
N(1)	1·1986 (5)	0·5326 (4)	0·7373 (3)	486
N(2)	0·7345 (5)	0·7665 (4)	1·0051 (3)	513
C(1)	0·8727 (5)	0·8261 (4)	0·7543 (3)	386
C(2)	0·7513 (5)	0·8891 (4)	0·6708 (3)	453
C(20)	0·6157 (7)	0·8284 (6)	0·6511 (4)	649
C(3)	0·7564 (6)	1·0161 (5)	0·6000 (3)	504
C(4)	0·8719 (6)	1·0828 (4)	0·6103 (3)	516
C(40)	0·8808 (8)	1·2159 (5)	0·5307 (5)	690
C(5)	0·9869 (6)	1·0230 (5)	0·6966 (4)	510
C(6)	0·9884 (5)	0·8949 (4)	0·7685 (3)	446
C(60)	1·1166 (7)	0·8376 (6)	0·8599 (4)	628
C(11)	1·1297 (6)	0·4592 (5)	0·6915 (4)	502
C(12)	1·2200 (8)	0·3816 (6)	0·6162 (4)	652
C(13)	1·3899 (8)	0·3743 (7)	0·5939 (5)	711
C(14)	1·4631 (7)	0·4485 (7)	0·6425 (4)	666
C(15)	1·3632 (6)	0·5263 (6)	0·7127 (4)	576
C(21)	0·6015 (6)	0·7187 (4)	1·0024 (3)	462
C(22)	0·4554 (6)	0·7537 (5)	1·0662 (4)	558
C(23)	0·4491 (8)	0·8416 (6)	1·1351 (4)	684
C(24)	0·5833 (8)	0·8901 (6)	1·1390 (4)	727
C(25)	0·7265 (8)	0·8488 (6)	1·0750 (4)	668

Table 2. *Bond distances* (Å), *bond angles* (°), *least-squares planes* (Å) *and dihedral angles* (°)

Sb(1)—S(1)	2·509 (3)	C(3)—C(4)	1·364 (8)
Sb(1)—S(2)	2·492 (6)	C(4)—C(40)	1·507 (7)
Sb(1)—N(1)	2·679 (6)	C(4)—C(5)	1·393 (7)
Sb(1)—N(2)	2·699 (5)	C(5)—C(6)	1·399 (6)
Sb(1)—C(1)	2·183 (4)	C(6)—C(60)	1·509 (8)
S(1)—C(11)	1·755 (7)	C(11)—C(12)	1·390 (8)
S(2)—C(21)	1·755 (5)	C(12)—C(13)	1·376 (10)
N(1)—C(11)	1·323 (8)	C(13)—C(14)	1·376 (11)
N(1)—C(15)	1·341 (8)	C(14)—C(15)	1·365 (8)
N(2)—C(21)	1·339 (8)	C(21)—C(22)	1·386 (7)
N(2)—C(25)	1·342 (8)	C(22)—C(23)	1·377 (9)
C(1)—C(2)	1·399 (6)	C(23)—C(24)	1·356 (11)
C(1)—C(6)	1·396 (7)	C(24)—C(25)	1·378 (9)
C(2)—C(20)	1·501 (9)	Sb(1)···Sb(1')	3·787 (5)
C(2)—C(3)	1·399 (6)		

Sb(1')···Sb(1)—S(2)	88·99 (3)	C(20)—C(2)—C(3)	117·4 (6)
Sb(1')···Sb(1)—N(1)	104·97 (9)	C(2)—C(3)—C(4)	122·9 (5)
Sb(1')···Sb(1)—N(2)	72·42 (9)	C(3)—C(4)—C(5)	118·2 (6)
Sb(1')···Sb(1)—C(1)	146·02 (12)	C(3)—C(4)—C(40)	122·2 (6)
S(1)—Sb(1)—S(2)	85·27 (6)	C(40)—C(4)—C(5)	119·6 (7)
S(1)—Sb(1)—N(1)	60·49 (12)	C(4)—C(5)—C(6)	120·9 (7)
S(1)—Sb(1)—N(2)	145·51 (13)	C(1)—C(6)—C(5)	119·7 (6)
S(1)—Sb(1)—C(1)	98·32 (11)	C(5)—C(6)—C(60)	117·6 (6)
S(2)—Sb(1)—N(1)	154·76 (11)	C(1)—C(6)—C(60)	122·7 (6)
S(2)—Sb(1)—N(2)	60·56 (13)	S(1)—C(11)—N(1)	115·7 (5)
S(2)—Sb(1)—C(1)	103·24 (15)	N(1)—C(11)—C(12)	122·0 (8)
N(1)—Sb(1)—N(2)	153·35 (16)	S(1)—C(11)—C(12)	122·2 (7)
N(1)—Sb(1)—C(1)	82·61 (16)	C(11)—C(12)—C(13)	118·5 (7)
N(2)—Sb(1)—C(1)	86·07 (14)	C(12)—C(13)—C(14)	119·6 (7)
Sb(1)—S(1)—C(11)	90·0 (3)	C(13)—C(14)—C(15)	118·1 (9)
Sb(1)—S(2)—C(21)	91·0 (4)	N(1)—C(15)—C(14)	123·3 (8)
C(11)—N(1)—C(15)	118·4 (6)	S(2)—C(21)—N(2)	115·4 (5)
C(21)—N(2)—C(25)	117·9 (7)	N(2)—C(21)—C(22)	122·9 (6)
Sb(1)—C(1)—C(6)	113·2 (4)	S(2)—C(21)—C(22)	121·7 (6)
Sb(1)—C(1)—C(2)	126·9 (5)	C(21)—C(22)—C(23)	117·7 (8)
C(2)—C(1)—C(6)	119·7 (5)	C(22)—C(23)—C(24)	120·0 (7)
C(1)—C(2)—C(3)	118·5 (6)	C(23)—C(24)—C(25)	119·4 (7)
C(1)—C(2)—C(20)	124·1 (5)	N(2)—C(25)—C(24)	121·9 (8)

Symmetry code: (i) $-x+2,\ -y+1,\ -z+2$.

No.	Plane through atoms	Equation of the plane
1	C(11),C(12),C(13),C(14),C(15),N(1)	$-0·110x + 0·665y - 0·739z = -3·55$ (4)
2	C(21),C(22),C(23),C(24),C(25),N(2)	$-0·217x + 0·678y - 0·702z = -4·23$ (5)
3	C(1),C(2),C(3),C(4),C(5),C(6)	$+0·454x - 0·689y - 0·565z = -7·28$ (5)
4	C(1),C(2),C(3),C(4),C(5),C(6),C(20),-C(40),C(60)	$+0·448x - 0·703y - 0·553z = -7·41$ (2)
5	Sb(1),S(1),C(11),N(1)	$-0·145x + 0·712y - 0·687z = -3·19$ (4)
6	Sb(1),S(2),C(21),N(2)	$-0·161x + 0·662y - 0·732z = -4·25$ (4)
7	Sb(1),S(2),C(21),N(2),S(1),C(11),N(1)	$-0·157x + 0·691y - 0·705z = -3·64$ (2)

Dihedral angles: 1,2 6·5 (2); 1,3 95·2 (2); 1,4 96·2 (1); 1,5 4·5 (1); 1,6 3·0 (2); 1,7 3·7 (2); 2,3 99·7 (2); 2,4 100·7 (1); 2,5 4·6 (2); 2,6 3·7 (2); 2,7 3·5 (2); 3,4 1·1 (1); 3,5 99·7 (1); 3,6 96·7 (1); 3,7 98·6 (1); 4,5 100·7 (1); 4,6 97·6 (1); 4,7 99·6 (1); 5,6 4·0 (1); 5,7 1·7 (1); 6,7 2·2 (1).

angles are summarized in Table 2. The Sb environment is best described as pseudo-octahedral. The coordination polyhedron around Sb is a distorted tetragonal pyramid with the C(1) of the mesityl group in the apical position. Space is left for a stereometrically active lone pair *trans* to C(1). Each molecule contains two planar bidentate 2-pyridinethiolate ligands. The two S atoms [S(1)—Sb(1)—S(2) = 85·27 (6)°] and the two N atoms [N(1)—Sb(1)—N(2) = 153·35 (16)°] are in *cis* positions. The SbSNC units and the mesityl-group skeleton are planar, and a plane can also be drawn through both chelating $SbS_2N_2C_2$ groups; the Sb atom lies 0·05 Å below this plane.

The Sb—S distances [Sb(1)—S(1) = 2·509 (3) Å, Sb(1)—S(2) = 2·492 (6) Å] are comparable with those in phenylbis[2-pyridinethiolato(1 −)]antimony(III) 2·503 (1) and 2·500 (1) Å (Preut, Huber & Hengstmann, 1988) and tris[2-pyridinethiolato(1 −)]-antimony 2·472 (2) Å (Bozopoulos, Kokkou, Rentzeperis & Karagiannidis, 1984). The two Sb—N distances [Sb(1)—N(1) = 2·679 (6), Sb(1)—N(2) = 2·699 (5) Å] are shorter than the corresponding Sb—N distances in tris[2-pyridinethiolato(1 −)]-antimony(III) 2·830 (7) Å (Bozopoulos, Kokkou, Rentzeperis & Karagiannidis, 1984).

Weak Sb···Sb′ interactions have to be assumed considering the short Sb(1)···Sb(1)′ ($-x+2$, $-y+1$, $-z+2$) contact [3·787 (5) Å] between neighbouring symmetrically equivalent molecules (see Fig. 2) which is less than the sum of the van der Waals radii (4·4 Å) (Pauling, 1968). The Sb(1)···Sb(1)′ contact is *trans* to the Sb(1)—C(1) bond so that Sb(1)′ formally completes a distorted octahedron around Sb(1). The angle Sb(1)′···Sb(1)—C(1) = 146·0° is in the range of corresponding angles in compounds with analogous intermolecular Sb···Sb′ interactions [tris(1-pyrrolidinecarbodithiolato)antimony(III) Sb′···Sb—S = 159·7° (Kavounis, Kokkou, Rentzeperis & Karagiannidis, 1980); *o*-ethylxanthatobis(quinolin-8-olato)antimony(III) Sb′···Sb—O = 143·5° (Hoskins, Tiekink & Winter, 1985); $(Sb_2OCl_6)^{2-}$ Sb′···Sb—O = 158·1° (Begley, Hall, Nunn & Sowerby, 1986)].

Financial assistance from the Fonds der Chemischen Industrie is gratefully acknowledged.

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Acta Cryst. (1991). C**47**, 2032–2035

Synthesis and Structure of 4-Ferrocenyl-2′-methyl-4′-nitroazobenzene*

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(*Received* 4 *February* 1991; *accepted* 25 *March* 1991)

Abstract. [Fe($C_{23}H_{19}N_3O_2$)], $M_r = 425{\cdot}3$, monoclinic, $P2_1/c$, $a = 15{\cdot}020\ (9)$, $b = 7{\cdot}555\ (2)$, $c = 18{\cdot}134\ (15)$ Å, $\beta = 112{\cdot}22\ (6)^\circ$, $V = 1905$ Å^3, $Z = 4$, $D_x = 1{\cdot}483$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 8{\cdot}28$ cm^{-1}, $F(000) = 880$, $T = 293$ K, $R = 0{\cdot}052$ for 2252 observed reflections. The azobenzene residue has the *trans* configuration, C—N—N—C torsion angle 177·2 (7)°, N═N bond length 1·238 (6) Å. The cyclopentadienyl rings of the ferrocenyl residue are eclipsed with Fe—C distances 2·005 (7) to 2·053 (6) Å, mean 2·034 (4) Å. The rings are inclined at 2·7 (6)° to one another. There does not appear to be any significant electron delocalization along the length of the molecule.

Introduction. There has recently been considerable interest in materials which exhibit non-linear optical (NLO) properties (*e.g.* Chemla & Zyss, 1987) in connection with optical data transmission and processing technologies. The necessary (but not sufficient) condition for a material to exhibit the desired NLO properties is that it should consist of polarizable dipolar molecules which crystallize in a non-centrosymmetric space group. The title compound contains a ferrocenyl residue which acts as electron donor and a nitro group which acts as electron acceptor linked by a polarizable chain of atoms, thus meeting the first criterion for NLO behaviour. Unfortunately, however, it crystallizes in the centrosymmetric space group $P2_1/c$. The related compound *cis*-[(η^5-C_5H_5)Fe(η^5-C_5H_4)—CH═CHC_6H_4-4-NO_2], however, crystallizes in the non-centrosymmetric space group *Cc* and shows relatively high NLO properties of 62 times that of urea taken as standard (Green, Marder, Thompson, Bandy, Bloor, Kolinsky & Jones, 1987).

Experimental. The synthesis of the title compound followed the method of Little & Clark (1960). A mixture of 2-nitroso-5-nitrotoluene (3·6 g, 22 mmol) and 4-aminophenylferrocene (5·0 g, 18 mmol) in glacial acetic acid (400 cm^3) was stirred at room temperature for 5 d. Water (1000 cm^3) and dichloromethane (100 cm^3) were added and the organic layer separated and evaporated to dryness to yield the crude product. This was purified by column chromatography using gradient elution from 50% dichloromethane/*n*-hexane to 100% dichloromethane. The major purple band collected in 70% dichloromethane/*n*-hexane afforded deep purple crystals. A crystal of size 0·4 × 0·4 × 0·1 mm selected from this material was mounted on an Enraf—Nonius CAD-4 diffractometer and cell dimensions were determined from the setting angles of 24 reflections in the range $10 < \theta < 15^\circ$. Intensity data were

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0108-2701/91/102032-04$03.00