Fig. 1. Perspective view of the complex molecule with the atom-labelling scheme.

A: Cu(*A*)···C(8*B*) = 3·116 (6) Å and Cu(*A*)···C(9*B*) = 3·142 (6) Å on one side, and Cu(*A*)···C(4*B*^{i}) = 3·061 (7) Å [(i) *x*, 0·5 − *y*, 0·5 + *z*] on the other side of the coordination plane. These copper–carbon interactions cause an arrangement of complex molecules parallel to *z*. Molecule *B* has somewhat longer Cu(*B*)···C distances: Cu(*B*)···C(8*A*) = 3·263 (6) Å and Cu(*B*)···C(9*A*) = 3·321 (7) Å on one side, and Cu(*B*)···C(4*A*^{ii}) = 3·149 (8) Å [(ii) *x*, 1 + *y*, *z*], on the other side of the coordination plane. These interactions give rise to chains running parallel to *y*.

Similar intermolecular copper–carbon contacts were observed in bis(3-methylpentane-2,4-dionato)-copper(II) (distance 3·216 Å) (Robertson & Truter, 1967), and similar intramolecular contacts in di-μ-{1-[2-(dimethylamino)phenyl]-2-(4-methylphenyl)-

1-propenyl-*N*,*C*}-tetracopper(I) (distances 3·28–3·56 Å; Smeets & Spek, 1987).

Other intermolecular contacts involve hydrogen bonding: O(*wA*)—H(*w*1)···O(1*B*) = 2·981 (7) Å [H(*w*1)···O(1*B*) = 2·155 (10) Å, O(*wA*)—H(*w*1)—O(1*B*) = 167 (2)°]; O(*wB*)—H(*w*3)···O(1*A*) = 2·973 (7) Å [H(*w*3)···O(1*A*) = 2·327 (9) Å, O(*wB*)—H(*w*3)—O(1*A*) = 171 (2)°]; O(*wB*)—H(*w*4)···N(1*B*^{i}) = 3·369 (9) Å [H(*w*4)···N(1*B*^{i}) = 2·404 (10) Å, O(*wB*)—H(*w*4)—N(1*B*^{i}) = 139 (2)°] [(i) = *x*, 0·5 − *y*, 0·5 + *z*] are established.

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Structure of Acetonitrile(dichloro)(phenyl)bis(triphenylstibine)rhodium(III)

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Abstract. [Rh(C$_{44}$H$_{38}$Cl$_2$NSb$_2$)], M_r = 998·1, monoclinic, *C*2/*c*, *a* = 13·816 (2), *b* = 13·472 (2), *c* = 22·075 (3) Å, β = 95·61 (2)°, *V* = 4089 Å^3, *Z* = 4, D_x = 1·62 Mg m^{-3}, λ(Mo *K*α) = 0·7107 Å, μ = 18·75 cm^{-1}, *F*(000) = 1960, *T* = 295 K, *R* = 0·051 for 2718 unique observed reflections. The structure consists of neutral complex molecules with an octahedral coordination sphere containing two chlorine ligands, two antimony atoms from triphenylstibine (SbPh$_3$), a nitrogen atom from acetonitrile (MeCN) and a carbon atom from phenyl (Ph). The two chlorine ligands are *trans* to each other and the Rh—Cl bond distances are 2·360 (2) Å. The Rh—N bond length of 2·163 (9) Å is somewhat large, indicating a significant *trans* influence of the Ph ligand. The Rh—Sb distance is 2·588 (1) Å.

Introduction. The chemistry of transition metal ions is very important for processes of biological significance. The study of inorganic, organic and organo-

metallic compounds capable of reacting with heteroaromatic bases has acquired greater interest after the discovery of antineoplastic properties shown by [cis-Pt(NH$_3$)$_2$Cl$_2$] and other transition metal complexes (Sherman & Lippard, 1987; Köpf-Maier & Köpf, 1987; Colamarino & Orioli, 1976). Most of the interest is devoted to platinum-group metal antitumour agents. Of the different oxidation states of rhodium, it was found that Rh^I, Rh^{II} and Rh^{III} give coordination compounds with anticancer properties (Alessio, Xu, Cauci, Mestroni, Quadrifoglio, Viglino & Marzilli, 1989; Giraldi, Zassinovich & Mestroni, 1974; Aoki, Moshino, Okada, Yamazaki & Sekizawa, 1986, and references therein; Aoki & Yamazaki, 1984, and references therein). Furthermore rhodium complexes of the type [trans-Rh^{III}L$_4$X$_2$]Y (L = substituted pyridine; X = Cl$^-$, Br$^-$; Y = Cl$^-$, Br$^-$, NO$_3^-$, ClO$_4^-$) showed high levels of antibacterial activity (Bromfield, Dainty, Gillard & Heaton, 1969).

In a previous study on the reactivity of platinum-group metal ions with substituted purines it was found that metal complexes containing the PPh$_3$ ligand act as convenient starting materials to produce crystalline Ru^{II}–6-thiopurine compounds (Cini, Cinquantini, Sabat & Marzilli, 1985). On the other hand, it is well known that ligands containing phosphorus, arsenic and antimony as donor atoms are involved in a great variety of metal complexes. However, the data relevant to antimony are rather sparse in comparison with phosphorus and arsenic (McAuliffe, 1987, and references therein). With the aim of preparing rhodium compounds able to react with aromatic bases we have carried out an extensive investigation on Rh^{III} complexes with stibines. Here we report on the synthesis of a new complex via the reaction of RhCl$_3$ with SbPh$_3$ in ethanol and subsequent crystallization from MeCN.

Experimental. On mixing RhCl$_3$.3H$_2$O (Merck) and SbPh$_3$ (Ega Chemie) in a 1:3 molar ratio in absolute ethanol (analytical grade Merck or BDH), at reflux for 3 h, a yellow microcrystalline powder was obtained. An investigation on the reaction of RhCl$_3$.3H$_2$O with SbPh$_3$ was previously reported (Hill & McAuliffe, 1974). The TLC analysis on silica gel (CH$_2$Cl$_2$ eluent) of the material obtained in our work, after recrystallization from CH$_2$Cl$_2$/ethanol, showed two distinct spots of about the same intensity. The product was then purified through a silica gel column (first yellow fraction, CH$_2$Cl$_2$ eluent). The solution was evaporated and the solid residue was mixed with MeCN (Merck, analytical grade). The suspension was refluxed for half an hour and then filtered. On concentration, the orange solution produced well formed orange prisms. The same crystals were obtained when the initial crude product was

directly crystallized from MeCN. The crystals were characterized by elemental analysis. Calcd C, 52·95; H, 3·84; N, 1·40. Found C, 53·12; H, 3·86; N, 1·41. The crystals are stable in air at room temperature for many months.

Crystal 0·30 × 0·30 × 0·35 mm; Philips PW1100 four-circle diffractometer, graphite-monochromated Mo Kα radiation; lattice parameters by least-squares refinement of 22 randomly selected reflections; intensity data $(-17 \le h \le 17, 0 \le k \le 17, 0 \le l \le 28)$ recorded in $\omega/2\theta$ scan mode for $5 \le 2\theta \le 55°$, scan speed 0·05°s^{-1}, scan width 0·85 + 0·35tan$\theta°$; three standard reflections ($\overline{3}$10, $\overline{3}$15, $2\overline{2}\overline{2}$) measured every 120 min showed no variation; 5014 reflections measured at 295 K, Lp and absorption corrections [numerical method as in SHELX76 (Sheldrick, 1976)], 2718 unique reflections with $F_o \ge 3\sigma(F)$.

Structure solved by using Patterson and difference Fourier methods. Difference maps based on the anisotropic full-matrix least-squares refinement on F including Rh, Sb, Cl, N, and C atoms revealed the positions of the H atoms of the Ph ligand and those of 10 H atoms of SbPh$_3$. The positional parameters of the 13 H atoms were refined in the final full-matrix least-squares cycles. The H atoms of the Ph ligand were given a common isotropic temperature factor U which refined at 0·049 (15) Å^2. This value is reasonably lower than the common temperature factor U for the H atoms of SbPh$_3$, refined at 0·059 (9) Å^2. Final refinement of 268 parameters gave $R = 0·051$, $wR = 0·055$, $S = 4·65$, weighting scheme $w = 1/[\sigma^2(F) + 0·059F^2]$; max. $\Delta/\sigma = 0·659$ for y coordinate of H(122); maxima in last $\Delta\rho$ map with 2·2, 2·2 and 1·8 e Å^{-3} were found at 0·91 Å from Sb, 0·81 Å from Rh and 0·87 Å from Sb respectively; min./max. of $-1·1/2·2$ e Å^{-3}.

Scattering factors for neutral atoms including f' and f'' from International Tables for X-ray Crystallography (1974, Vol. IV). Calculations performed with SHELX76 (Sheldrick, 1976) and SHELX86 (Sheldrick, 1986).

Discussion. Positional parameters and U values are given in Table 1.* A view of the structure is shown in Fig. 1. Interatomic distances and bond angles are reported in Table 2.

The crystal structure of the title compound contains discrete molecules with an octahedral coordination geometry. The metal centre, the MeCN molecule and C(1) and C(4) atoms of the Ph ligand lie on a twofold axis. As a consequence, the two SbPh$_3$ lig-

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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and* U_{eq} *values* $\times 10^3$ ($\times 10^4$ *for* Rh, Sb) *with e.s.d.'s in parentheses*

$$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	$U_{eq}(\text{Å}^2)$
Rh	5000	2739 (1)	2500	322 (4)
Sb	3995 (1)	2762 (1)	1447 (1)	344 (3)
Cl	3463 (1)	2674 (1)	2881 (1)	47 (1)
N(A)	5000	1133 (6)	2500	46 (6)
C(1A)	5000	293 (9)	2500	54 (9)
C(2A)	5000	-803 (10)	2500	106 (21)
C(1)	5000	4256 (8)	2500	42 (7)
C(2)	5422 (6)	4788 (6)	2058 (4)	45 (5)
C(3)	5429 (8)	5822 (6)	2060 (4)	63 (8)
C(4)	5000	6319 (10)	2500	64 (12)
C(111)	3442 (6)	1325 (6)	1195 (4)	43 (5)
C(112)	3025 (7)	764 (7)	1632 (5)	61 (8)
C(113)	2739 (9)	-207 (8)	1503 (7)	79 (10)
C(114)	2864 (9)	-598 (8)	938 (7)	82 (11)
C(115)	3224 (9)	-36 (9)	510 (6)	79 (10)
C(116)	3539 (7)	935 (7)	626 (5)	56 (6)
C(121)	2718 (6)	3643 (6)	1338 (4)	42 (5)
C(122)	2758 (8)	4641 (6)	1535 (5)	58 (6)
C(123)	1935 (8)	5234 (7)	1445 (5)	66 (8)
C(124)	1078 (10)	4826 (9)	1182 (5)	80 (9)
C(125)	1033 (8)	3861 (10)	987 (5)	72 (9)
C(126)	1847 (7)	3270 (7)	1080 (4)	52 (6)
C(131)	4631 (6)	3146 (6)	640 (3)	38 (4)
C(132)	5530 (7)	2747 (8)	539 (4)	60 (7)
C(133)	5934 (8)	2943 (10)	-1 (5)	75 (11)
C(134)	5447 (9)	3527 (11)	-447 (5)	81 (10)
C(135)	4556 (9)	3927 (8)	-350 (5)	75 (9)
C(136)	4131 (7)	3736 (7)	199 (4)	61 (7)

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

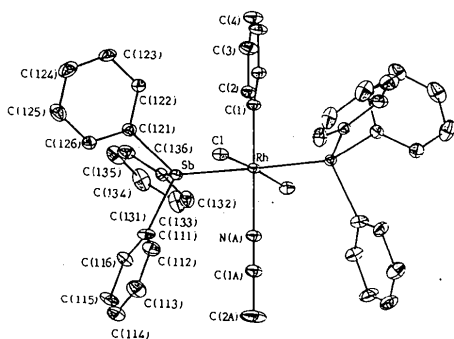|			
Rh—Sb	2.588 (1)	C(114)—C(115)	1.343 (18)
Rh—Cl	2.360 (2)	C(115)—C(116)	1.395 (14)
Rh—N(A)	2.163 (9)	C(116)—C(111)	1.379 (12)
Rh—C(1)	2.044 (10)	C(121)—C(122)	1.413 (11)
Sb—C(111)	2.135 (8)	C(122)—C(123)	1.387 (13)
Sb—C(121)	2.121 (8)	C(123)—C(124)	1.381 (15)
Sb—C(131)	2.127 (7)	C(124)—C(125)	1.370 (17)
N(A)—C(1A)	1.132 (14)	C(125)—C(126)	1.376 (14)
C(1A)—C(2A)	1.476 (17)	C(126)—C(121)	1.375 (12)
C(1)—C(2)	1.385 (9)	C(131)—C(132)	1.391 (12)
C(2)—C(3)	1.394 (11)	C(132)—C(133)	1.389 (14)
C(3)—C(4)	1.361 (12)	C(133)—C(134)	1.383 (16)
C(111)—C(112)	1.393 (12)	C(134)—C(135)	1.380 (16)
C(112)—C(113)	1.387 (13)	C(135)—C(136)	1.421 (13)
C(113)—C(114)	1.379 (17)	C(136)—C(131)	1.386 (12)
Sb—Rh—Sb'	178.6 (1)	Sb—C(111)—C(116)	121.2 (7)
Sb—Rh—Cl	84.1 (1)	C(112)—C(111)—C(116)	120.4 (8)
Sb'—Rh—Cl	95.9 (1)	C(111)—C(112)—C(113)	120 (1)
Sb—Rh—N(A)	90.7 (0)	C(112)—C(113)—C(114)	119 (1)
Sb—Rh—C(1)	89.3 (0)	C(113)—C(114)—C(115)	120 (1)
Cl—Rh—Cl'	175.7 (1)	C(114)—C(115)—C(116)	122 (1)
Cl—Rh—N(A)	87.9 (1)	C(115)—C(116)—C(111)	118 (1)
Cl—Rh—C(1)	92.1 (1)	Sb—C(121)—C(122)	119.4 (7)
N(A)—Rh—C(1)	180	Sb—C(121)—C(126)	121.8 (6)
Rh—N(A)—C(1A)	180	C(122)—C(121)—C(126)	118.8 (8)
N(A)—C(1A)—C(2A)	180	C(121)—C(122)—C(123)	119.8 (9)
Rh—C(1)—C(2)	121.1 (5)	C(122)—C(123)—C(124)	119.2 (9)
C(2)—C(1)—C(2')	118 (1)	C(123)—C(124)—C(125)	121 (1)
C(1)—C(2)—C(3)	121.1 (8)	C(124)—C(125)—C(126)	119 (1)
C(2)—C(3)—C(4)	119.4 (9)	C(125)—C(126)—C(121)	121.3 (9)
C(3)—C(4)—C(3')	121.0 (1)	Sb—C(131)—C(132)	119.2 (6)
Rh—Sb—C(111)	111.8 (2)	Sb—C(131)—C(136)	120.6 (6)
Rh—Sb—C(121)	118.4 (2)	C(132)—C(131)—C(136)	120.0 (8)
Rh—Sb—C(131)	121.7 (2)	C(131)—C(132)—C(133)	120 (1)
C(111)—Sb—C(121)	101.8 (3)	C(132)—C(133)—C(134)	121 (1)
C(111)—Sb—C(131)	99.7 (3)	C(133)—C(134)—C(135)	120 (1)
C(121)—Sb—C(131)	100.1 (3)	C(134)—C(135)—C(136)	121 (1)
Sb—C(111)—C(112)	118.2 (6)	C(135)—C(136)—C(131)	118.9 (9)

Fig. 1. *ORTEP* (Johnson, 1971) drawing of the title compound with the labelling scheme and thermal ellipsoids drawn at 30% probability.

ands (also the two Cl atoms) are *trans* to each other. The Rh—Cl and Rh—Sb distances of 2·360 (2) and 2·588 (1) Å, respectively, are in agreement with corresponding values previously found in other rhodium-(III) complexes (Lamprecht, Leipoldt & Van Biljon, 1984; Colamarino & Orioli, 1976; Mague, 1970). The Rh—C distance is 2·044 (10) Å; this appears to be longer than the Rh—C length (1·99 Å) recently found for [Rh^{III}(2-phenylpiridine)$_2$(2,2'-bipyridine)]$^+$ (Zilian, Maeder, von Zelewski & Güdel, 1989) which contains the two Rh—C bonds *cis* to each other and *trans* to the Rh—N bonds. For [Rh^{III}(2-C$_6$H$_4$N(O)O)$_2$Cl(PPh$_3$)] (Vicente, Martin, Solans &

Font-Altaba, 1989) the Rh—C lengths are 2·04 (2) and 1·96 (2) Å when the carbon donor is *trans* to PPh$_3$ and to O(NO)C$_6$H$_4$ respectively. The Rh—C lengths average 2·05 (2) Å in [Rh^{III}(1,3-diphenyl-1,3-propanedionato)(SbPh$_3$)$_2$Ph$_2$] (Lamprecht *et al.*, 1984).

The Rh—N bond length of 2·163 (9) Å reveals a significant *trans* influence of the Ph ligand. It has to be pointed out that the Rh—N(CMe) distances average 2·022 (7) Å for the two MeCN molecules *trans* to each other in [Rh^{III}(PPh$_3$)$_2$(MeCN)$_3$NO]$^{2+}$ (Kelly, Welch & Woodward, 1977).

The acetonitrile N—C distance is 1·132 (14) Å in the present structure, while free nitrile N—C lengths average 1·136 (10) Å (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). For N metal bound nitriles, the N—C lengths average 1·133 (20) Å (Orpen, Brammer, Allen, Kennard, Watson & Taylor, 1989). It has to be pointed out that the IR spectrum of the title complex in the solid state as Nujol mulls or KBr pellets does not show any absorption band attributable to N—C triple bond stretching vibration in the 2000–2400 cm^{-1} region.

The angles of the coordination sphere have almost regular values, the largest deviation being found for the Sb—Rh—Cl angle [84·1 (1)°]. The Cl—Rh—C—C torsion angle is 34·3° so that the steric hindrance between the Ph and the Cl ligand should not be high. The Cl—Rh—Cl' angle is 175·7 (1)°.

The Sb atom has an approximate tetrahedral geometry with Sb—C distances averaging 2·128 (2) Å. This value is in agreement with the mean values of 2·145 and 2·188 Å previously found for other compounds (Lamprecht et al., 1984; Mague, 1970). The C—Sb—C angles average 100·5 (3)° while previously reported mean values are 101·0° (Lamprecht et al., 1984) and 102·3° (Mague, 1970). The Rh—Sb—C bond angles range from 111·8 (2) to 121·7 (2)°.

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(1,3-Diamino-2-propanol-N,N')diiodoplatinum(II) and (1,3-Diamino-2-propanol-N,N')dichloroplatinum(II)

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Abstract. (1) [PtI$_2$(C$_3$H$_{10}$N$_2$O)], M_r = 539·0, monoclinic, $P2_1/n$, a = 10·756 (2), b = 7·211 (1), c = 12·113 (1) Å, β = 90·20 (1)°, V = 939·3 (3) Å^3, Z = 4, D_x = 3·81 g cm^{-3}, graphite-monochromated Mo $K\alpha$ radiation, λ = 0·71069 Å, μ = 223 cm^{-1}, $F(000)$ = 936, T = 298 K, R = 0·046 for 1080 unique reflections with $I > 3\sigma(I)$. (2) [PtCl$_2$(C$_3$H$_{10}$N$_2$O)], M_r = 356·1, monoclinic, $P2_1/n$, a = 8·497 (3), b = 8·784 (2), c = 10·735 (5) Å, β = 105·79 (3)°, V = 771·0 (5) Å^3, Z = 4, D_x = 3·07 g cm^{-3}, graphite-monochromated Mo $K\alpha$ radiation, λ = 0·71069 Å, μ = 198 cm^{-1}, $F(000)$ = 648, T = 298 K, R = 0·054 for 1456 unique reflections with $I > 3\sigma(I)$. In both compounds the amine ligand coordinates to the central metal ion