Journal of Organometallic Chemistry
Elsevier Sequoia S.A., Lausanne
Printed in The Netherlands

PREPARATION AND STRUCTURE OF ORGANOANTIMONY(III) DERIVATIVES CONTAINING POTENTIALLY BIDENTATE LIGANDS. ANTIMONY–CARBON CLEAVAGE UPON COMPLEX FORMATION*

H. A. MEINEMA AND J. G. NOLTES
Organisch Chemisch Instituut TNO, Utrecht (The Netherlands)

(Received June 15th, 1970)

SUMMARY

Organoantimony(III) derivatives of the types $RSbX_2$ and R_2SbX in which X represents a *N,N*-diethyldithiocarbamate, *N,N*-diethylcarbamate, oxinate or tropolonate ligand have been synthesized and investigated by spectroscopic (IR, UV) methods. In the $RSbX_2$ compounds (R = phenyl, alkyl) the ligand X appears to be bidentate both in the solid state and in solution making the antimony pentacoordinate. The compounds $RSbOx_2$ are exceptional in that dissolution in benzene or chloroform causes rupture of the weak coordinate Sb–N bond. Compounds R_2SbX, in which R = alkyl, spontaneously disproportionate into a 1/1 mixture of R_3Sb and $RSbX_2$. That the redistribution equilibrium lies completely to the side of $RSbX_2$ is connected with the much reduced electrophilic reactivity of $RSbX_2$ as a result of pentacoordination. In contrast, compounds of the type Ph_2SbX appear to be stable. A four-coordinate structure is observed in the solid state, but three-coordination appears to be preferred in solution.

INTRODUCTION

There has recently been increasing interest in organoantimony coordination chemistry, and two groups have studied the preparation and properties of a series of hexacoordinate organoantimony(V) acetylacetonate[1-3] and oxinate[4,5] complexes. So far, however, the field of organoantimony(III) coordination chemistry has remained unexplored. Compounds of the types R_2SbX and $RSbX_2$ in which the group X is a potentially bidentate ligand are of special interest in that the antimony atom has a choice between three- and higher coordination, and so, we have studied the preparation and properties of a series of organostibines, R_2SbX and $RSbX_2$ in which R represents an alkyl or phenyl group and X a potentially chelating organic ligand. The present paper presents our results on organoantimony(III) compounds containing *N,N*-diethyldithiocarbamate, *N,N*-diethylcarbamate, oxinate and tyropolonate ligands.

---

* Part V in the series "Investigations on Organoantimony Compounds". For Part IV see ref. 6.

RESULTS AND DISCUSSION

Preparation

Recently we prepared a series of (diethylamino)organostibines, R_2SbNEt_2 and $RSb(NEt_2)_2$[6]. These compounds appear to be eminently suitable as starting materials for the preparation of other R_2SbX and $RSbX_2$ derivatives.

The insertion of CS_2 or CO_2 into the metal–nitrogen bond of organometalamines has been widely applied as a route to organometal-substituted dithiocarbamates and carbamates (for a recent review see ref. 7). Bis(*N*,*N*-diethyldithiocarbamato)-alkyl- and -phenylstibines and the corresponding bis(*N*,*N*-diethylcarbamate) derivatives were prepared in quantitative yield by this type of reaction:

$$RSb(NEt_2)_2 + 2\ CX_2 \rightarrow RSb[XC(X)NEt_2]_2 \quad (R = \text{phenyl or alkyl}; X = O \text{ or } S)$$

The corresponding reactions of Ph_2SbNEt_2 afford the expected diphenylstibine derivatives:

$$Ph_2SbNEt_2 + CX_2 \rightarrow Ph_2SbXC(X)NEt_2 \quad (X = O \text{ or } S)$$

However, the dialkylstibine derivatives show abnormal behaviour in that they are apparently unstable and spontaneously disproportionate into a 1/1 mixture of trialkylstibine and $RSb[XC(X)NEt_2]_2$:

$$2\ R_2SbNEt_2 + 2\ CX_2 \rightarrow RSb[XC(X)NEt_2]_2 + R_3Sb \quad (R = Me,\ Et \text{ or } Bu;\ X = O \text{ or } S)$$

Attempts to prepare other $RSbX_2$ and R_2SbX derivatives *via* acidolysis of $RSb(NEt_2)_2$ or R_2SbNEt_2 compounds with monoprotic bidentate ligands gave similar results. $RSb(NEt_2)_2$ (R = Ph or alkyl) and Ph_2SbNEt_2 afforded the expected products upon reaction with 8-hydroxyquinoline (oxine) or tropolone:

$$RSb(NEt_2)_2 + 2\ HX \rightarrow RSbX_2 + 2\ HNEt_2$$
$$Ph_2SbNEt_2 + HX \rightarrow Ph_2SbX + HNEt_2 \quad (R = \text{phenyl or alkyl};\ HX = \text{oxine or tropolone})$$

The dialkyl compounds react with spontaneous disproportionation into a 1/1 mixture of R_3Sb and $RSbX_2$:

$$2\ R_2SbNEt_2 + 2\ HX \rightarrow RSbX_2 + R_3Sb + 2\ HNEt_2 \quad (R = \text{alkyl};\ HX = \text{oxine or tropolone})$$

Reaction of (halo)organostibines with the sodium salts of the corresponding organic ligands gives the same reaction products. Kupchik *et al.*[8,9] have described a few (dithiocarbamato)phenylstibines synthesized by this procedure. A patent application[10] claims the preparation of a series of (dithiocarbamato)organostibines, including the otherwise unknown dialkylantimony derivatives.

Melting points and analytical data for the various compounds prepared are given in Tables 1, 2 and 3.

Molecular weights of representative compounds were determined by osmometry and/or ebulliometry in benzene solution. The results showed that the *N*,*N*-diethyldithiocarbamate, the oxinate and the tropolonate derivatives are essentially monomeric in benzene solution. Because of the extreme sensitivity towards hydrolysis

TABLE 1

ANALYTICAL AND PHYSICAL DATA FOR A SERIES OF (*N,N*-DIETHYLDITHIOCARBAMATO)- AND (*N,N*-DIETHYLCARBAMATO)ORGANOSTIBINES

Compound	M.p. (°C)	Sb(%)	
		Found	Calcd.
N,N-diethyldithiocarbamates			
$\mathrm{MeSb[SC(S)NEt_2]_2}$	134–135	28.18	28.09
$\mathrm{EtSb[SC(S)NEt_2]_2}$	73–74	27.61	27.21
$\mathrm{BuSb[SC(S)NEt_2]_2}$	62–63	25.95	25.60
$\mathrm{PhSb[SC(S)NEt_2]_2}$	150–153[a]	24.76	24.57
$\mathrm{Ph_2SbSC(S)NEt_2}$	68–70[b]	28.55	28.70
N,N-diethylcarbamates			
$\mathrm{EtSb[OC(O)NEt_2]_2}$	66–67	31.72	31.78
$\mathrm{BuSb[OC(O)NEt_2]_2}$	[c]	30.40	29.61
$\mathrm{PhSb[OC(O)NEt_2]_2}$	75–79	28.30	28.23
$\mathrm{Ph_2SbOCONEt_2}$	90–91	30.90	31.04

[a] Ref. 9, m.p. 148–150°. [b] Ref. 8, m.p. 68–69°. [c] Oil.

TABLE 2

ANALYTICAL AND PHYSICAL DATA FOR A SERIES OF (OXINATO)ORGANOSTIBINES

Compound	M.p. (°C)	Analysis Found (calcd.) (%)			
		C	H	N	Sb
$\mathrm{MeSb(Ox)_2}$	~200 (decomp.)	53.33	3.82	6.69	28.11
		(53.69)	(3.55)	(6.59)	(28.64)
$\mathrm{EtSb(Ox)_2}$	~180 (decomp.)	54.48	4.05	6.18	27.46
		(54.70)	(3.90)	(6.37)	(27.23)
$\mathrm{PrSb(Ox)_2}$	~105 (decomp.)	55.28	4.50	5.87	26.53
		(55.67)	(4.22)	(6.18)	(26.86)
$\mathrm{BuSb(Ox)_2}$	~105 (decomp.)	56.38	4.73	5.85	25.73
		(56.57)	(4.53)	(5.99)	(26.06)
$\mathrm{PhSb(Ox)_2}$	~200 (decomp.)	58.35	3.81	4.77	25.15
		(59.17)	(3.51)	(5.75)	(24.19)
$\mathrm{Ph_2SbOx}$	79–82	59.35	4.10	3.48	29.20
		(60.03)	(3.83)	(3.33)	(28.97)

TABLE 3

ANALYTICAL AND PHYSICAL DATA FOR SOME (TROPOLONATO)ORGANOSTIBINES

Compound	M.p. (°C)	Sb(%)	
		Found	Calcd.
$\mathrm{EtSb(Trop)_2}$	~180 (decomp.)	30.44	31.04
$\mathrm{BuSb(Trop)_2}$	~100 (decomp.)	28.53	28.91
$\mathrm{PhSb(Trop)_2}$	~200 (decomp.)	27.80	27.66
$\mathrm{Ph_2SbTrop}$	80–82	30.37	30.73

of the carbamate derivatives, mol. wt. determinations gave erratic values for these compounds.

Infrared spectra

The infrared spectra of compounds $RSb[SC(S)NEt_2]_2$ (R = Ph or alkyl) were recorded in the 2000–400 cm^{-1} region. Apart from the characteristic absorptions of the R groups all spectra are essentially similar in solution (CCl_4 and CS_2) and in the solid state (Nujol mull and KBr pellets). They show a strong absorption band at 1480–1490 cm^{-1}, which has previously been associated with the vibration of C=N bond having partial double bond character[11,12]. Bonati *et al.*[13,14] recently showed that no definite evidence exists for the proposed correlation between the position of this band and the coordination behaviour of the dithiocarbamate ligand, but concluded that the region around 1000 cm^{-1} in particular does give indications of whether a complex contains a chelate- or an ester-type dithiocarbamate ligand. If the dithiocarbamate group acts as a bidentate ligand only one absorption band occurs in this region. A monodentate (ester-type) dithiocarbamate complex shows an additional absorption at slightly higher wave numbers due to an uncomplexed C=S group. Both the solution and the solid state spectra of compounds $RSb[SC(S)NEt_2]_2$ show only one intense absorption band in the region 980 ± 70 cm^{-1}, which strongly indicates the presence of bidentate dithiocarbamate ligands and therefore of five-coordinate antimony in these compounds (*cf.* refs. 12–15 for the presence of bidentate dithiocarbamate ligands in organogold[12], organotin[13,14] and organothallium[13,15] dithiocarbamates).

Comparison of the IR spectrum of $Ph_2SbSC(S)NEt_2$ in solution (CCl_4, CS_2) and in the solid state (nujol mull and KBr pellets) reveals that these spectra are essentially identical except for the 980 ± 70 cm^{-1} region. Whereas the solid state spectra show one intense absorption at 980 cm^{-1} and a weak shoulder at 1003 cm^{-1}, the solution spectra show medium intensity bands at 986 and 1006 cm^{-1} (see Fig. 1).

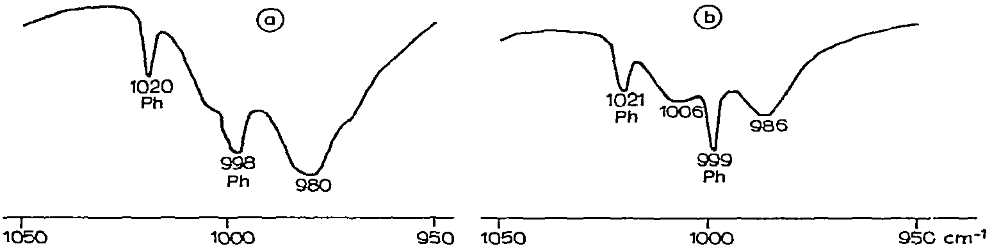


Fig. 1. Infrared spectra of $Ph_2SbSC(S)NEt_2$ in the C=S stretching region: (a) nujol mull; (b) in CS_2 solution.

Absorptions due to the phenyl groups are observed at 1020 and 998 cm^{-1} (*cf.* ref. 16, which deals with the IR spectra of phenyl-substituted antimony compounds). This suggests that in the solid state $Ph_2SbSC(S)NEt_2$ contains a weakly bonded bidentate dithiocarbamate ligand (four-coordinate antimony) and that in solution rupture of the weak Sb–S coordinate bond takes place to make the antimony atom three-coordinate (*cf.* refs. 17 and 18 for the presence of a monodentate dithiocarbamate ligand in $RuNO[SC(S)NR_2]_3$).

The IR data suggest that the carbamate analogues $RSb[OC(O)NEt_2]_2$

likewise contain pentacoordinate antimony, both in the solid state and in solution (strong, broad carbonyl absorption of the bidentate carbamate ligands at 1550–1590 cm^{-1}). The antimony atom in $Ph_2SbOC(O)NEt_2$ in the solid state appears to be four-coordinate (broad carbonyl absorption at 1570–1590 cm^{-1}). However, the presence of a sharp strong band at 1620 cm^{-1} in benzene or CCl_4 indicates rupture of the Sb–O coordinate bond upon dissolution [*cf.* ref. 19, for a similar behaviour of $Me_3SnOC(O)NEt_2$].

The IR spectra of the oxinato(organo)stibines (see Table 2), which were run in the 2000–400 cm^{-1} region were not very informative. Apart from the characteristic bands due to alkyl or phenyl group absorptions these spectra are essentially similar. They all show a characteristic strong band in the region 510–520 cm^{-1}. This band which is absent in the spectrum of oxine also has been observed in the spectra of various other metal oxinates (*cf.* ref. 4 and references cited therein) and accordingly has been assigned to the metal–oxygen stretching vibration.

The IR spectra of the (tropolonato)organostibines were run in the 2000–400 cm^{-1} region, in the solid state (nujol mull and KBr pellet) and in $CDCl_3$ solution. Each of the spectra shows at least eight absorptions in the 1600–1200 cm^{-1} region. Detailed information on the IR spectra of tropolonate complexes has been recently given by Junge[20]. A comparison of Junge's data with the IR spectra of our complexes and the spectrum of tropolone methyl ether (prepared according to ref. 21) does not enable us to draw a definite conclusion as to whether the tropolonate ligand acts as a bidentate or monodentate ligand. However, based on the similarity of the spectra of copper(II) tropolonate[20], which is known to contain a chelating ligand[22], and of $RSb(Trop)_2$ we favour a structure with five-coordinate antimony for the latter compounds.

All the (tropolonato)organostibines show a characteristic absorption band in the region 500–510 cm^{-1}. This band which is absent in the spectra of tropolone and tropolone methyl ether also, has been observed in the spectra of organotin[23] and organothallium[15] tropolonates and accordingly has been assigned to a metal–oxygen stretching vibration.

UV spectra

The UV spectra of the (oxinato)organostibines, $RSbOx_2$ (R = Me, Et, Pr, Bu or Ph) and Ph_2SbOx in oxygen-free dry methylene chloride show an absorption band at 370–380 nm ($\varepsilon = 4.1$–4.3×10^3 for compounds $RSbOx_2$ and 2.1–2.2×10^3 for Ph_2SbOx). The presence of this absorption band in the spectra of metal oxinates is generally considered to be a reliable indication for the presence of a chelating oxinate ligand[24–26], and so we may with confidence assign structures with five- and four-coordinate antimony respectively to the compounds $RSbOx_2$ and Ph_2SbOx in methylene chloride.

The UV spectra of the same compounds recorded in dry, oxygen-free benzene immediately after the preparation of the solution likewise show an absorption band at 370–380 nm. However, within one minute the intensity of this absorption decreases and a new band appears at 320 nm (see Fig. 2). This indicates that in benzene the evidently weak Sb–N coordinate bond is broken, resulting in the Sb atom becoming three-coordinate in both $RSbOx_2$ (R = Ph or alkyl) and Ph_2SbOx. The same phenomenon was observed in chloroform solution.

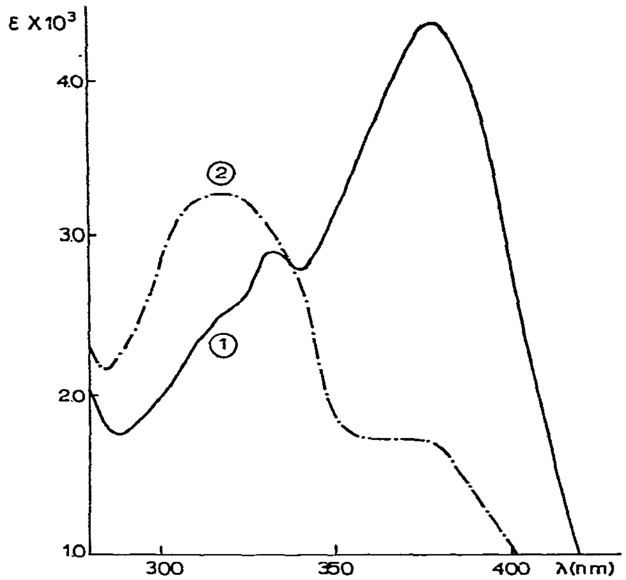


Fig. 2. UV absorption spectra of $BuSbOx_2$ in benzene solution. The molar extinction coefficient ε plotted against the wavelength λ (nm): 1. immediately after the preparation of the solution; 2. after standing for about 1 min.

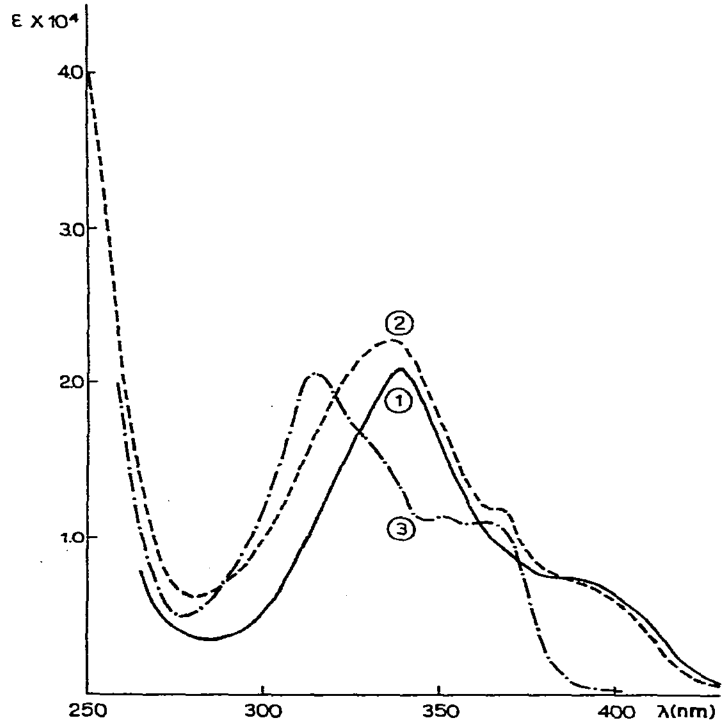


Fig. 3. UV absorption spectra of $PhSb(Trop)_2$: 1. in benzene; 2. in chloroform; 3. in methylene chloride.

The magnitude of the shift of the 320 nm absorption band of oxine in chelated oxinate complexes has been correlated with the complex stability[25]. However, this correlation apparently does not exist for organoantimony oxinates as both for the (oxinato)organostibines discussed in this paper and for a series of hexacoordinate (oxinato)organoantimony(V) compounds, $R_nSbCl_{4-n}Ox$ with $n=1$, 2, or 4, which contain Sb–N coordinative bonds with greatly diverging stabilities[4], a constant position of the oxinate absorption at ~380 nm has been observed.

UV spectra of $Ph_2SbTrop$ and $PhSb(Trop)_2$ were run in benzene, chloroform and methylene chloride (Figs. 3 and 4). The methylene chloride solution spectra resemble the UV spectrum of chelated $Cu(Trop)_2$[21,22]. The benzene and chloroform solution spectra of $Ph_2SbTrop$ markedly differ from that in methylene chloride and resemble the spectrum of tropolone methyl ether[21] (Fig. 4), suggesting that the Sb–O coordinative bond in $Pg_2SbTrop$ is broken upon dissolution in these solvents.

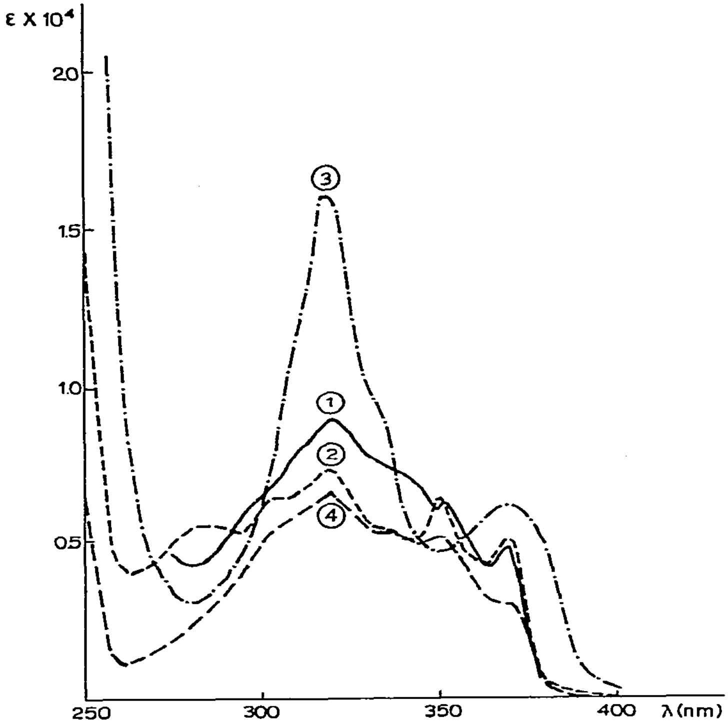


Fig. 4. UV absorption spectra of $Ph_2SbTrop$: 1. in benzene; 2. in chlorform; 3. in methylene chloride. UV absorption spectrum of tropolone methyl ether: 4. in methylene chloride.

DISCUSSION

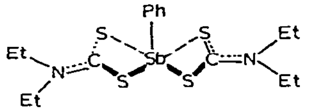
The stability of organostibines R_2SbX and $RSbX_2$ appears to depend on the nature of X. Compounds containing a halogen[27] or methoxy[27] group are observed to be fairly stable at room temperature but very sensitive towards oxidation by air-oxygen[28]. The present study reveals that when X represents a bidentate ligand, the thermal and oxidative stability of compounds $RSbX_2$ is considerably enhanced, probably as a result of the Sb atom being pentacoordinate. However, corresponding compounds of the type R_2SbX in which R = alkyl appear to be extremely unstable and spontaneously disproportionate according to:

$$2\,R_2SbX \rightarrow R_3Sb + RSbX_2$$

This redistribution reaction presumably proceeds via electrophilic attack of antimony on carbon. This reaction which in principle is reversible lies completely at the right side because of the much reduced electrophilic reactivity of antimony in $RSbX_2$ as a result of pentacoordination. The electron withdrawing phenyl substituents in Ph_2SbX stabilize the antimony–ligand bond and favour tetracoordination. Nevertheless, it is somewhat surprising that the Ph_2SbX compounds discussed in this paper do not show any tendency towards disproportionation even after several hours of reflux in benzene.

Redistributions similar to those reported in this paper have been observed in organobismuth chemistry. Whereas $Ph_2SbSC(S)NEt_2$ is stable in refluxing benzene, attempts[9] to prepare the bismuth analogue resulted in a spontaneous decomposition into Ph_3Bi, $PhBi[SC(S)NEt_2]_2$ and $Bi[SC(S)NEt_2]_3$. Okawara *et al.*[29] observed the same phenomenon upon addition of pyridine to Ph_2BiX (X = Cl or Br), the products $PhBiX_2 \cdot 2Py$ and Ph_3Bi being isolated.

A discussion of the structure of the organostibines described in this paper must be tentative. It seems plausible that the weak coordinative bonds present in $RSbX_2$ (R = Ph and alkyl) and in Ph_2SbX do not cause considerable distortions of the bond angles normally observed in three-coordinate antimony(III) compounds (bond angles of ~90°). $PhSb[SC(S)NEt_2]_2$ might possess a pseudo-octahedral structure with the Sb–C bond perpendicular to the plane containing the non-equivalent Sb–S bonds (*cf.* the X-ray structure of $PhAs[SC(S)NEt_2]_2$[30]).



Similar structures may be considered for the other $RSbX_2$ derivatives.

EXPERIMENTAL PART

General

All reactions were carried out under oxygen-free dry nitrogen. IR spectra were run on a Grubb–Parsons Spectromaster. The UV spectra were obtained with a Cary model 15 recording spectrometer, using 1 cm cells. Anhydrous oxygen-free benzene, chloroform and methylene chloride were used as solvents. The concentrations of the solutions were such as to give optical densities in the range 0.1–2.0.

Molecular weights were measured on 0.2–0.5% solutions in benzene using a Mechrolab dynamic vapour pressure osmometer, and a Gallenkamp ebulliometer.

Analytical and melting point data are given in Tables 1–3. Analyses were carried out at this Institute by Mr. W. J. Buis (C, H, N) and Mr. C. W. Dekker (Sb).

Preparation of (N,N-diethyldithiocarbamato)organostibines

Bis(N,N-diethyldithiocarbamato)ethylstibine

Method 1. Carbon disulphide (3 ml) was added dropwise to a solution of $EtSb(NEt_2)_2$ (2.56 g) in benzene (15 ml). The highly exothermic reaction was controlled by cooling with an ice bath. After partial evaporation of the benzene solution, pentane was added and 2.95 g (76%) of $EtSb[SC(S)NEt_2]_2$ was precipitated as a pale yellow crystalline solid.

Method 2. Carbon disulphide (1 ml) in pentane (10 ml) was added dropwise to a solution of Et_2SbNEt_2 (2.92 g) in pentane (20 ml) at 0°. A pale yellow crystalline solid precipitated which appeared to be pure $EtSb[SC(S)NEt_2]_2$ (1.65 g; 64%). Distillation of the pentane solution gave pure Et_3Sb (b.p. 51°/13 mm).

The compounds $RSb[SC(S)NEt_2]_2$ (R = Me, Bu or Ph) and $Ph_2SbSC(S)NEt_2$ were obtained similarly in yields of 80 to 90%.

Preparation of (N,N-diethylcarbamato)organostibines

These compounds were prepared by the procedure used for the corresponding dithiocarbamate derivatives. Carbon dioxide was bubbled through pentane solutions of (diethylamino)organostibines, R_2SbNEt_2 and $RSb(NEt_2)_2$.

Preparation of (oxinato)- and (tropolonato)organostibines

Both (oxinato)- and (tropolonato)organostibines were prepared by the addition of oxine or tropolone to a pentane solution of (diethylamino)organostibines in the required molar ratio. The compounds precipitated in nearly quantitative yield from the reaction mixture.

ACKNOWLEDGEMENTS

This research has been supported by N.V. Billiton Maatschappij, the Hague. Thanks are due to Mr. H. F. Martens for assistance in the experimental work and to Mrs. R. de Ridder for running IR and UV spectra and for molecular weight determinations.

REFERENCES

1 H. A. Meinema and J. G. Noltes, *J. Organometal. Chem.*, 16 (1969) 257.
2 N. Nishii, M. Shindo, Y. Matsumura and R. Okawara, *Inorg. Nucl. Chem. Lett.*, 5 (1969) 529.
3 N. Nishii, Y. Matsumura and R. Okawara, *Inorg. Nucl. Chem. Lett.*, 5 (1969) 703.
4 H. A. Meinema, E. Rivarola and J. G. Noltes, *J. Organometal. Chem.*, 17 (1969) 71.
5 Y. Kawasaki, *Inorg. Nucl. Chem. Lett.*, 5 (1969) 805.
6 H. A. Meinema and J. G. Noltes, *Inorg. Nucl. Chem. Lett.*, 6 (1970) 241.
7 K. Jones and M. F. Lappert, *Organometal. Chem. Rev.*, 1 (1966) 67.
8 E. J. Kupchik and P. J. Calabretta, *Inorg. Chem.*, 4 (1965) 973.
9 E. J. Kupchik and C. T. Theisen, *J. Organometal. Chem.*, 11 (1968) 627.
10 M&T Chemicals Inc., *Neth. Appln.*, 6,505,218; *Chem. Abstr.*, 64 (1966) 9766e.
11 J. Chatt, L. A. Duncanson and L. M. Venanzi, *Nature (London)*, 177 (1956) 1042.
12 H. J. A. Blaauw, R. J. F. Nivard and G. J. M. van der Kerk, *J. Organometal. Chem.*, 2 (1964) 236.
13 F. Bonati, S. Cenini and R. Ugo, *J. Organometal. Chem.*, 9 (1967) 395.
14 F. Bonati, S. Cenini and R. Ugo, *J. Organometal. Chem.*, 10 (1967) 257.
15 H. Kurosowa and R. Okawara, *J. Organometal. Chem.*, 14 (1968) 225.

16 G. O. DOAK, G. G. LONG AND L. D. FREEDMAN, *J. Organometal. Chem.*, 4 (1965) 82.
17 A. DOMENICANO, A. VACIAGO, L. ZAMBONELLI, P. L. LOADER AND L. M. VENANZI, *Chem. Commun.*, (1966) 476.
18 B. F. G. JOHNSON, K. H. AL-OBAIDI AND J. A. MCCLEVERTY, *J. Chem. Soc. A*, (1969) 1668.
19 T. A. GEORGE, K. JONES AND M. F. LAPPERT, *J. Chem. Soc.*, (1965) 2157.
20 H. JUNGE, *Spectrochim. Acta, Part A*, 24 (1968) 1957.
21 J. W. COOK, A. R. GIBB, R. A. RAPHAEL AND A. R. SOMMERVILLE, *J. Chem. Soc.*, (1951) 503.
22 J. M. ROBERTSON, *J. Chem. Soc.*, (1951) 1222.
23 M. KOMURA, T. TANAKA AND R. OKAWARA, *Inorg. Chim. Acta*, 2 (1968) 321.
24 T. MOELLER AND A. J. COHEN, *J. Amer. Chem. Soc.*, 72 (1950) 3546.
25 K. SONE, *J. Amer. Chem. Soc.*, 75 (1953) 5207.
26 T. MOELLER AND F. L. PUNDSACK, *J. Amer. Chem. Soc.*, 76 (1954) 617.
27 E. A. BESOLOVA, V. A. FOSS AND I. F. LUTSENKO, *Zh. Obshch. Khim.*, 38 (1968) 267; *Chem. Abstr.*, 69 (1968) 52243a.
28 H. A. MEINEMA, unpublished results.
29 R. OKAWARA, K. YASUDA AND M. INOUE, *Bull. Chem. Soc. Jap.*, 39 (1966) 1823.
30 R. BALLY, *Acta Crystallogr.*, 23 (1967) 295.