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INVESTIGATIONS ON ORGANOANTIMONY COMPOUNDS II[*]. PREPARATION AND CONFIGURATION OF ORGANO(OXINATO)-ANTIMONY(V) COMPOUNDS $R_nSbCl_{4-n}Ox$ ($n = 1$–4)

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SUMMARY

Organo(oxinato)antimony(V) compounds of the types $RSbCl_3Ox$, R_2SbCl_2Ox, $R_3SbClOx$ and R_4SbOx (R = alkyl and phenyl) have been synthesized and investigated by spectroscopic (UV, PMR, IR) methods. All the compounds are monomeric in benzene. In all the compounds except $R_3SbClOx$ (R = alkyl), which contains penta-coordinate antimony, the oxinato group acts as a bidentate ligand in benzene solution. The compounds $RSbCl_3Ox$ and R_2SbCl_2Ox are also hexa-coordinate in ethanol (100%) and chloroform, but partial or complete rupture of the Sb–N coordinate bond in $Ph_3SbClOx$ and R_4SbOx takes place in these polar solvents. The stability of the Sb–N coordinate bond in R_4SbOx follows the order of the *I*-effect of the group R.

Low-temperature PMR experiments show the occurrence of rapid pseudo-rotation for hexa-coordinate Me_4SbOx in toluene above $-70°$. At $-100°$ two types of methyl groups can be distinguished, but even at this temperature a limiting spectrum is not obtained. In methylene dichloride rapid pseudo-rotation takes place even at $-100°$. Similar results are obtained for the $-100°$ PMR spectra of $Me_4SbAcac$ in these solvents.

The spectral results are used in discussions of the possible configurations of the organo(oxinato)antimony(V) compounds.

INTRODUCTION

Organoantimony(V) compounds of the type R_nSbX_{5-n} are interesting from a structural point of view[1]. Compounds in which one of the groups X is a potentially chelating organic ligand are of special interest in that the antimony atom has a choice between penta- and hexa-coordination. Recently, one group of such compounds, *i.e.* organo(acetylacetonato)antimony(V) chlorides $R_nSbCl_{4-n}Acac$ ($n = 1,2,4$) has been independently studied by two groups[2-4]. With the exception of Ph_2SbCl_2Acac (partly penta-coordinate in chloroform solution[4]) such compounds contain hexa-coordinate antimony[2-4]. We have extended our studies to novel organo(oxinato)-

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antimony(V) compounds of the type $R_nSbCl_{4-n}Ox$ ($n=1$–4; R = alkyl or aryl) and herein report the results of these investigations.

RESULTS AND DISCUSSION

Preparation

Organo(oxinato)antimony(V) compounds of the type R_4SbOx were prepared by treating oxine (8-hydroxyquinoline) with R_4SbOMe (R = Me, Et[5], Bu, Ph[6]) in absolute methanol. Compounds of the type R_2SbCl_2Ox (R = Et, Bu) were obtained by the addition of an ethanol solution of oxine to a solution of the corresponding dialkylhydroxo(oxo)antimony compound[7] in a mixture of 4 *N* hydrochloric acid and ethanol. The methyl and phenyl derivatives were easily obtained by treating R_2SbCl_3

TABLE 1

ANALYTICAL AND PHYSICAL DATA FOR SOME ORGANO(OXINATO)ANTIMONY(V) COMPOUNDS

Compound	M.p. (°C)	Analyses[a], found (calcd.) (%)					Mol.wt. Found (Calcd.)
		C	H	Cl	N	Sb	
$MeSbCl_3Ox$	142–144 (dec.)	31.66	2.90	25.65	3.83	30.62	392
		(31.01)	(2.32)	(27.52)	(3.62)	(31.52)	(387)
$PhSbCl_3Ox$	228–232 (dec.)	39.48	2.82	24.75	3.18	27.42	
		(40.09)	(2.45)	(23.71)	(3.12)	(27.09)	[b]
Me_2SbCl_2Ox	160–161	36.34	3.99	19.95	4.47	33.07	373
		(35.97)	(3.27)	(19.34)	(3.82)	(33.24)	(367)
Et_2SbCl_2Ox	140–141	38.79	4.22	18.51	4.00	30.34	417
		(39.49)	(4.05)	(18.00)	(3.52)	(30.88)	(395)
Bu_2SbCl_2Ox	102–104	45.15	5.54	16.04	3.17	26.80	526
		(45.23)	(5.32)	(15.74)	(3.10)	(27.05)	(451)
Ph_2SbCl_2Ox	209–210	50.60	3.37	15.17	3.10	24.69	487
		(51.32)	(3.26)	(14.46)	(2.85)	(24.85)	(491)
$Me_3SbClOx$	143–145	41.82	4.62	10.33	4.17	35.59	304
		(41.62)	(4.33)	(10.24)	(4.04)	(35.21)	(346)
$Et_3SbClOx$	78–79	46.62	5.55	9.22	3.82	31.30	338
		(46.39)	(5.41)	(9.13)	(3.61)	(31.40)	(388)
$Ph_3SbClOx$	198–205	61.09	4.14	6.29	2.75	22.13	530
		(60.78)	(3.93)	(6.66)	(2.70)	(22.88)	(533)
Me_4SbOx	85–87	48.27	4.99		4.38	37.88	308
		(47.85)	(5.24)		(4.29)	(37.34)	(326)
Et_4SbOx	oil	54.27	7.16		3.54	31.86	374
		(53.40)	(6.80)		(3.66)	(31.86)	(382)
Bu_4SbOx	72–73	59.70	8.65		2.45	25.22	448
		(60.74)	(8.56)		(2.83)	(24.62)	(494)
Ph_4SbOx[c]	189–193	68.62	4.78		2.50	22.36	597
		(69.01)	(4.56)		(2.43)	(21.20)	(574)

[a] Analyses were carried out at this Institute by Mr. W. J. Buis (C, H, N) and Mr. C. W. Dekker (Sb, Cl). [b] Not determined due to limited solubility in benzene. [c] During the course of this study, the preparation of hexa-coordinate Ph_4SbOx (m.p. 188–190°) has been reported[3].

(R = Me[8], Ph[9]) with oxine in dry ethanol solution. Similarly, reaction of $RSbCl_4$ (R = Me, Ph[10]) with oxine in absolute ethanol afforded $RSbCl_3Ox$. Attempts to prepare $R_3SbClOx$ by reacting R_3SbCl_2 with oxine were unsuccessful. However, these compounds were easily obtained by treating R_3SbCl_2 with sodium oxinate in dry ethanol in a 1/1 molar ratio.

All compounds with the exception of Et_4SbOx are crystalline solids, which except for the colourless $Me_3SbClOx$ and $Et_3SbClOx$ are yellow. Each of the antimony oxinates has been found to be monomeric in benzene.

Melting points, analytical and molecular weight data for the various compounds prepared are given in Table 1.

UV spectra

The shift to longer wavelength of the 320 nm absorption of oxine in the UV spectrum of an oxinato-metal complex is generally considered to be a reliable indication for the presence of a chelating oxine ligand[11–13]. The UV spectra of the organo(oxinato)antimony compounds $RSbCl_3Ox$ (R = Me, Ph), R_2SbCl_2Ox and R_4SbOx (R = Me, Et, Bu, Ph) in benzene solution each show an absorption at approximately 380 nm (ε 1.8–2.3 × 10^3). We therefore assume that in benzene solution, these compounds contain a chelated oxinate ligand. Noteworthy is the appearance of an additional band at 338 nm (ε 2.0–2.1 × 10^3) in the benzene solution spectra of the $(alkyl)_4SbOx$ compounds. This band, which is absent or very weak in the spectra of the other organoantimony(V) oxinates, also has been observed in the spectra of other chelated metal oxinates[11].

The magnitude of the shift of the 320 nm absorption in the UV spectrum of chelated oxinato complexes has been correlated with the complex stability[12] (*cf.* also ref. 14). The constant position of the oxinate absorption at approximately 380 nm in all our chelated complexes $R_nSbCl_{4-n}Ox$ (n = 1,2,4) is therefore noteworthy, since it can be reasonably assumed that progressive replacement of organic groups R by chlorine atoms will increase the effective nuclear charge of the antimony atom and

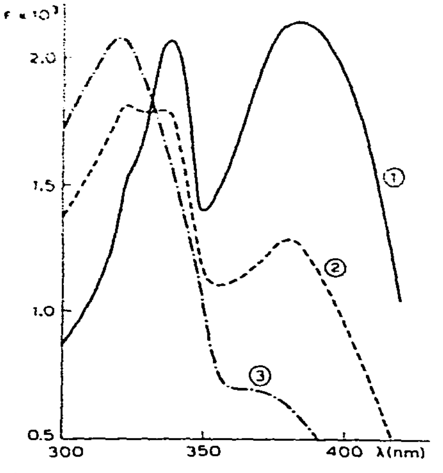


Fig. 1. Absorption spectra of $(CH_3)_4SbOx$. The molar extinction coefficient, ε, plotted against the wavelength λ (nm); 1 in benzene; 2 in chloroform; 3 in absolute ethanol.

hence the complex stability (*cf.* our PMR results). That the oxinate ligand is indeed weakly chelated in R_4SbOx follows from the UV spectra recorded in the more polar solvents ethanol (100%) and chloroform. Depending on the nature of R the Sb–N coordinate bond is partly or completely broken in these solvents, as indicated by the diminished extinction or complete disappearance of the 380 and 338 nm absorptions, which is accompanied by the commensurate appearance of the 320 nm absorption (see Fig. 1; *cf.* refs. 14 and 15 for a discussion of the UV spectrum of Ph_3SnOx in polar solvents). The results shown in Table 2 indicate that the strength of the Sb–N coordinate bond decreases in the order $Ph_4SbOx > Me_4SbOx \approx Et_4SbOx > Bu_4SbOx$, which agrees with the order of the inductive effect of the organic groups R.

TABLE 2

INFLUENCE OF SOLVENT POLARITY ON THE CONFIGURATION OF R_4SbOx COMPOUNDS

Compound	Coordination in[a]		
	C_6H_6	$CHCl_3$	C_2H_5OH
Ph_4SbOx	6	6	5, 6
Me_4SbOx[b]	6	5, 6	5
Et_4SbOx	6	5, 6	5
Bu_4SbOx	6	5	5

[a] 6: Hexa-coordination, 380 nm absorption present; 5: penta-coordination, 320 nm absorption present.
[b] This compound is hexa-coordinate in toluene and in methylene dichloride solution.

The UV spectra of compounds $RSbCl_3Ox$ and R_2SbCl_2Ox are the same in ethanol (100%) and chloroform as in benzene solution (single absorption at 380 nm) confirming the greater strength of the Sb–N coordinate bonds in these compounds. The absence of any solvent effect on the 380 nm band (a $\pi \rightarrow \pi^*$ absorption of the oxine ligand[16]) is noteworthy (*cf. e.g.* ref. 17b).

The compounds $R_3SbClOx$ with R = alkyl (Me or Et) contain a non-chelating oxine ligand (single absorption at 320 nm in benzene), whereas $Ph_3SbClOx$ contains a weakly chelated oxine ligand (single absorption at 365 nm) the Sb–N bond being ruptured in absolute ethanol or chloroform. The reflectance spectra of $Me_3SbClOx$ and $Et_3SbClOx$ (broad absorption band with the maximum at 320–330 nm) indicate that in the solid state also the oxine ligand is not chelated and that, consequently, the antimony atom in these compounds is penta-coordinate.

PMR spectra

PMR spectral data of methyl- and ethyl(oxinato)antimony(V) compounds in chloroform-*d* solution (27°) are tabulated in Table 3. Single signals were observed for the methyl and methylene proton resonances, which appear at progressively lower field upon increasing the number of chlorine atoms at antimony. For the $R_3SbClOx$ and R_4SbOx compounds, which the UV data show to contain a non- or weakly chelated oxinate ligand in chloroform solution, the δ-values of the signals due to the oxinate ligand differ only slightly from those of free oxine[18]. However, the spectra of the strongly chelated compounds $RSbCl_3Ox$ and R_2SbCl_2Ox show a considerable downfield shift of the 2- and 4-proton resonances (see Table 3). This shift is somewhat

TABLE 3

PMR SPECTRAL DATA FOR METHYL- AND ETHYL(OXINATO)ANTIMONY(V) COMPOUNDS IN CHLOROFORM-*d* AT 27°

Compound	R	Chemical shifts (δ^a)				
		$CH_3(-Sb)$	$CH_3(-CH_2Sb)$	$-CH_2(-Sb)$	2-H(Ox)	4-H(Ox)
$RSbCl_3Ox$	Me	2.86			9.58	8.64
R_2SbCl_2Ox	Me	2.38			9.36	8.58
	Et		1.30	2.82	9.31	8.64
$R_3SbClOx$	Me	2.08			8.76	8.23
	Et		1.42	2.45	8.70	8.20
R_4SbOx	Me	0.98			8.50	8.14
	Et		1.26	1.68	8.39	7.98
HOx					8.73	8.04

[a] ppm downfield from TMS.

greater for $MeSbCl_3Ox$ than for R_2SbCl_2Ox and this might reflect the greater Sb–N coordinative bond strength in $MeSbCl_3Ox$.

The UV data for Me_4SbOx (Table 2) show the presence of both the chelated and non-chelated configuration in chloroform solution. The observation of only one sharp singlet due to the methyl protons in chloroform-*d* at 27° points to a rapid equilibrium between stereochemically non-rigid penta- and hexa-coordinate species. As appears from the UV data Me_4SbOx is hexa-coordinate in toluene. The PMR spectrum in toluene-d_8 shows one sharp signal at 27° (δ 0.96 ppm), which broadens at −70°. At −80° the appearance of an additional broad signal is observed. At −100° the spectrum was resolved in two separate signals at δ 1.04 and 0.50 ppm with an

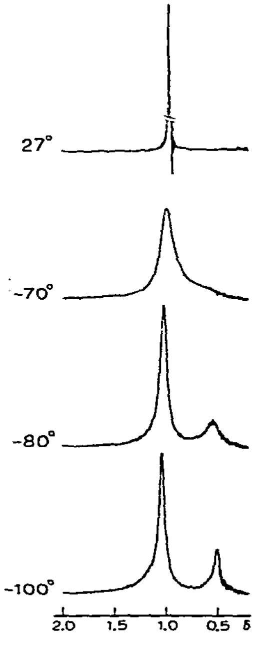
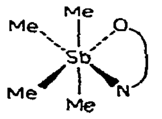
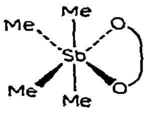


Fig. 2. Methyl proton resonance spectra of $(CH_3)_4SbOx$ in toluene-d_8 solution at various temperatures.

intensity ratio 3/1 (see Fig. 2). This indicates that, whereas at temperatures above −70° rapid pseudo-rotation occurs (*cf.* ref. 19), at −100° two types of methyl groups can be distinguished. The broadness of the signals suggest that at this temperature a limiting spectrum has not yet been obtained. Moreover, this spectrum would be expected to consist of three signals with a 2/1/1 intensity ratio (structure A) rather than the observed two signals with 3/1 intensity ratio. In methylene dichloride, in which solvent Me_4SbOx is also hexa-coordinate, the PMR spectrum showed even at −100° one sharp singlet at δ 1.11 ppm, indicating a considerable lower energy barrier for pseudo-rotation.



A



B

These results prompted us to investigate the PMR spectra of $Me_4SbAcac^4$ in toluene-d_8 and methylene dichloride at −100°. The results were consistent with those obtained for Me_4SbOx. Whereas the PMR spectrum of $Me_4SbAcac$ in toluene-d_8 at 27° shows one sharp methyl(Sb) resonance at δ 1.01 ppm, at −100° two broad, not fully resolved signals with an intensity ratio 1/1 are present at δ 0.86 and 1.15 ppm. Apparently, at this temperature the limiting spectrum for structure B (expected to consist of two separate signals with 1/1 intensity ratio) has not yet been reached. In methylene dichloride at −100° pseudo-rotation is still rapid as appears from the presence of one sharp methyl(Sb) singlet at δ 1.21 ppm.

Me_2SbCl_2Ox, which according to the UV spectrum is hexa-coordinate in methylene dichloride, showed in this solvent even at −100° one sharp singlet at δ 2.32 ppm. Whether this is due to equivalence of the methyl groups or still fast pseudo-rotation is obscure. Due to lack of solubility this experiment could not be carried out in toluene-d_8 solution.

IR spectra

The IR spectra of the organo(oxinato)antimony(V) compounds were run as

TABLE 4

RELEVANT INFRARED STRETCHING FREQUENCIES OF METHYL(OXINATO)ANTIMONY(V) COMPOUNDS IN THE 650–250 cm^{-1} REGION[a]

Compound	Stretching frequencies (cm^{-1})			
	Sb–C	Sb–O	[b]	Sb–Cl[c]
$MeSbCl_3Ox$	545	530	410	315
Me_2SbCl_2Ox	576[d]	520	400	290, 270
$Me_3SbClOx$	560[e,f]	515	370	< 250[g]
Me_4SbOx	535, 525[h]	497	365	

[a] The IR spectra of all organo(oxinato)Sb^V compounds show a strong absorption at ~620 cm^{-1}. [b] Assignment uncertain. [c] ν(Sb–Cl) for Me_3SbCl_2 at 275 cm^{-1} (*cf.* ref. 20). [d] Et_2SbCl_2Ox: $\nu(Sb–C)_{asym}$ not observed. [e] $\nu(Sb–C)_{asym}$ for Me_3SbCl_2 at 577 cm^{-1} (*cf.* ref. 20). [f] $Et_3SbClOx$: $\nu(Sb–C)_{asym}$ 534 cm^{-1}. [g] Increased absorption at 250 cm^{-1}. [h] $\nu(Sb–C)_{asym}$ for $Me_4Sb^+I^-$ at 570 cm^{-1} (*cf.* ref. 21).

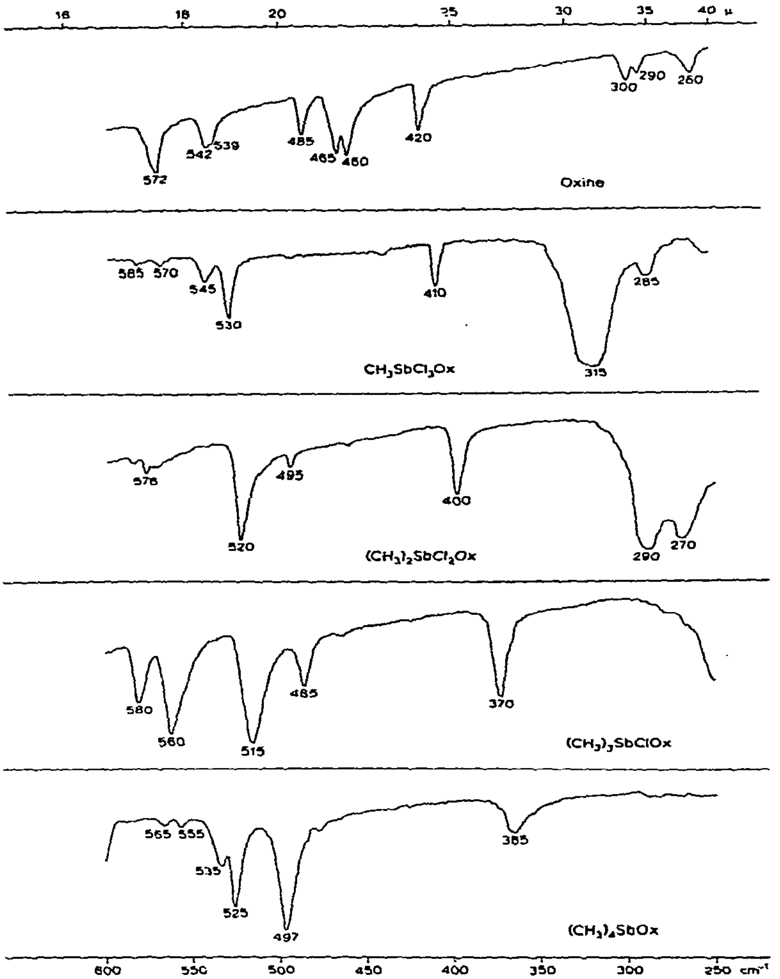


Fig. 3. Infrared spectra of oxine and methyl(oxinato)antimony(V) compounds in nujol between CsI discs.

solids in KBr in the 5000–400 cm^{-1} region. Spectra of the methyl derivatives Me_n-$SbCl_{4-n}Ox$ ($n = 1$–4) were also recorded as nujol mulls between CsI discs in the 600–250 cm^{-1} region. The latter spectra are shown in Fig. 3. Relevant frequencies are listed in Table 4.

The assignment of the Sb–C stretching frequencies deserves little comment. Like Me_2SbCl_3[1], Me_2SbCl_2Ox shows a very weak $\nu(Sb\text{–}C)_{asym}$. No distinct Sb–C absorptions were found in the spectra of the butyl derivatives. The phenyl compounds show Ph–Sb absorptions at 450–460 cm^{-1}.

The spectra of non-chelated $Me_3SbClOx$ contains bands at 580 and 485 cm^{-1} which are also present in the spectrum of oxine, but which are notably absent in the spectra of the chelated compounds.

The spectra of all compounds $R_nSbCl_{4-n}Ox$ show a strong absorption at 500–530 cm^{-1}. This band which is absent in the spectrum of oxine, but which has also been observed in the spectra of $Sb(OX)_3$[22], organotin[14,23,24] and other metal oxinates[25] is reasonably assigned to a metal–oxygen vibration.

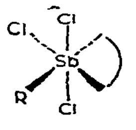
The spectra of the methyl derivatives $Me_nSbCl_{4-n}Ox$ ($n = 1$–4) show a medium intensity absorption in the region 365–410 cm^{-1}. This absorption, like the 500–530 cm^{-1} band, shifts to lower frequency upon progressive replacement of chlorine atoms by methyl groups. An absorption in this same region (380–410 cm^{-1}) present in the spectra of methyltin oxinates has been assigned by Okawara *et al.*[14] to the Sn–N stretching frequency. The 370 cm^{-1} band observed in the spectrum of $Me_3SbClOx$ cannot be due to an Sb–N absorption, since, as appears from the UV data, this compound does not contain an Sb–N bond. Likewise, the corresponding band observed in the spectra of the chelated compounds, is not believed to be due to a pure Sb–N vibration.

DISCUSSION

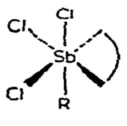
Until quite recently the chemistry of mono- and diorganoantimony(V) compounds has remained largely unexplored. The penta-coordinate halides $RSbX_4$ and R_2SbX_3 are thermally unstable compounds[26]; only $PhSbCl_4$[10] and R_2SbCl_3 ($R = Ph$[9], Me[8]) have been reported so far. However, replacement of one chlorine atom by a bidentate oxinato or acetylacetonato[2,4] ligand leads to quite stable compounds with hexa-coordinate antimony. Increased thermal stability resulting from increasing the coordination number of the metal is not uncommon (*e.g.* $R_2Au^{III}X$[17] and $RTlX_2$[27] compounds).

UV, PMR and IR spectroscopy yield valuable information on the occurrence and strength of Sb–N coordinate bonding in compounds $R_nSbCl_{4-n}Ox$, but the data do not allow definite structural assignments to be made. Compounds of the type $RSbCl_3Ox$ may be formulated with R in the equatorial (I) (R and O either in *cis*- or *trans*-position) or in the axial (II) position. Structure (I) which based on PMR evidence could be unambiguously assigned to $PhSbCl_3Acac$[2] may be favoured.

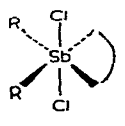
Several structures [*e.g.* (III)–(V)] are possible for compounds R_2SbCl_2Ox. The presence of one Sb–C and two Sb–Cl stretching frequencies in the spectrum of Me_2SbCl_2Ox and the observation of only one methyl proton resonance at −100° in the PMR spectrum favours structure (IV). The actual structure will probably be a considerably distorted octahedron.



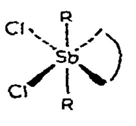
(I)



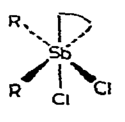
(II)



(III)

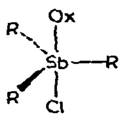


(IV)

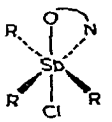


(V)

Compounds of the type R_3SbCl_2 are known to possess a trigonal bipyramidal structure with the two chlorine atoms occupying axial positions (*cf.* ref. 28). A similar structure (VI) for the non-chelated $R_3SbClOx$ (R = alkyl) compounds seems plausible (*cf.* the presence of one Sb–C stretching absorption in the IR). The observation that $R_3SbClOx$ (R = Me, Et) contrary to R_4SbOx compounds are non-chelated in benzene solution is rather intriguing, since one would expect the R_3SbCl group to be a better acceptor than the R_4Sb group. Obviously structure (VII) which would be obtained on hexa-coordination of (VI) is unstable.



(VI)



(VII)

In the absence of more detailed information on the preferred orientation of the carbon and hetero atoms in the chelated compounds $RSbCl_3Ox$, R_2SbCl_2Ox and R_4SbOx a discussion of this subject is premature.

EXPERIMENTAL

General

The UV spectra were obtained with a Cary Model 15 Recording Spectrometer, using 1 cm cells. Anhydrous benzene, chloroform and ethanol were used as solvents. The concentrations of the solutions were such as to give optical densities in the range 0.1–2.0.

IR spectra were run on a Grubb–Parsons Spectromaster for the 5000–400 cm^{-1} and on a Perkin–Elmer 457 for the 600–250 cm^{-1} region.

PMR spectra were measured as 5–10% solutions using a Varian HA-100 spectrometer. Tetramethylsilane was used as an internal standard.

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

Analytical and physical data are given in Table 1.

Preparation of organo(oxinato)antimony(V) compounds

Methyltrichloro(oxinato)antimony. Methyldichloroantimony[8] 2.7 g (13 mmoles) dissolved in carbon tetrachloride (25 ml) was chlorinated at 0° with 0.73 g (13 mmoles) of chlorine dissolved in carbon tetrachloride (25 ml). Addition of 1.45 g (10 mmoles) oxine in absolute ethanol (25 ml) resulted in the formation of a deep yellow solution. After evaporation of the solvents and recrystallization from absolute ethanol 0.5 g $MeSbCl_3Ox$ was obtained. Yield 10%.

Dimethyldichloro(oxinato)antimony. Dimethyltrichloroantimony[8] 2.7 g (11 mmoles) was dissolved in absolute ethanol (25 ml) at 0°. Addition of 1.6 g (11 mmoles) oxine in absolute ethanol (25 ml) resulted in the formation of a deep yellow solution from which after partial evaporation of the solvent 2.0 g Me_2SbCl_2Ox was obtained. Yield 55%.

Diethyldichloro(oxinato)antimony. Diethylhydroxo(oxo)antimony[7] 0.42 g (2 mmoles) was dissolved in a mixture of 4 *N* hydrochloric acid (1.5 ml) and ethanol (10 ml). Addition of 0.3 g (2 mmoles) oxine resulted in the formation of a deep yellow solution. After evaporation of the solvents the remaining yellow solid afforded by recrystallization from 96% ethanol 0.49 g Et_2SbCl_2Ox. Yield 62%.

Trimethylchloro(oxinato)antimony. Trimethyldichloroantimony 6.0 g (25 mmoles) was dissolved in 100 ml absolute ethanol. Addition of 4.2 g (25 mmoles) sodium oxinate in 100 ml absolute ethanol resulted in the formation of a pale yellow solution and the precipitation of 1.4 g NaCl, which was removed by filtration. The filtrate afforded after evaporation of the solvent and recrystallization from absolute ethanol 4.5 g $Me_3SbClOx$ as a colourless crystalline solid. Yield 54%.

Phenyltrichloro-, diphenyldichloro-, dibutyldichloro-, triethylchloro- and triphenylchloro(oxinato)antimony. These compounds were isolated by recrystallization from ethanol ($PhSbCl_3Ox$, yield 25%; Ph_2SbCl_2Ox, yield 47%; Bu_2SbCl_2Ox, yield 67%), benzene/light petroleum ($Ph_3SbClOx$, yield 55%) and light petroleum (40–60°) ($Et_3SbClOx$, yield 51%).

Tetramethyl-, tetraethyl-, tetrabutyl- and tetraphenyl(oxinato)antimony. These compounds were obtained in nearly quantitative yield upon addition of oxine to a methanol solution of the corresponding tetraalkyl- or tetraphenylmethoxoantimony compounds. After evaporation of the solvent recrystallization from light petroleum (40–60°) afforded yellow crystalline compounds (R = Me, Bu, Ph). The ethyl derivative appeared to be a yellow oil at room temperature.

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