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INVESTIGATIONS ON ORGANOANTIMONY COMPOUNDS

VI*. PREPARATION AND PROPERTIES OF THERMALLY STABLE DIALKYLANTIMONY(V) COMPOUNDS OF THE TYPES $R_2Sb(OR')_3$, $R_2Sb(OAc)_3$ AND $R_2Sb(O)OH$**

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SUMMARY

Thermally stable dialkylantimony(V) compounds of the type R_2SbX_3 (X = OMe, OEt, or OAc) have been prepared in good yields and purity. Hydrolysis of the trialkoxodialkylantimony compounds, $R_2Sb(OR')_3$, results in the quantitative formation of hydroxo(oxo)dialkylantimony compounds, $R_2Sb(O)OH$. These compounds are thermally stable and not sensitive towards oxygen in the air. Therefore, they form suitable starting materials for the synthesis of a variety of dialkylantimony(V) compounds.

Variable-temperature PMR experiments point to the presence of a non-rigid structure for compounds R_2SbX_3 (R = Me, Et; X = OMe, OAc). At $-80°$ in toluene-d_8 dimeric $Me_2Sb(OMe)_3$ appears to be rigid within the PMR time scale. The PMR spectrum displays two methoxo group signals in a 2/1 intensity ratio for the terminal and bridging methoxo groups, respectively. Combined PMR and Raman data indicate a dimeric octahedral structure, in which the methyl groups occupy *trans*-positions, both in the solid state and in concentrated solutions.

Based on IR and Raman spectroscopic data, a hexacoordinate structure, in which one bidentate and two monodentate acetato ligands occupy equatorial and the two methyl groups occupy axial positions, is proposed for monomeric $Me_2Sb(OAc)_3$.

INTRODUCTION

So far alkylantimony(V) chemistry has been mainly restricted to the study of thermally stable, easily accessible R_3SbX_2 derivatives (*cf.* refs. 3–6 and refs. cited therein). Very few alkylantimony(V) compounds of the type R_2SbX_3 have been reported, the only known examples being trihalodimethylantimony compounds, Me_2SbX_3 ($X = F^4$, Cl^7, Br^7, I^7). These compounds are thermally unstable and decompose at room temperature into dihalomethylstibines and methyl halides. Com-

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* For Part V see ref. 1.
** Taken from the Ph.D. thesis of one of us (H.A.M.), ref. 2.

pounds of the type Me_2SbX_2Y containing different halogen atoms (chlorine and bromine) are also thermally unstable[3].

Recently we have succeeded in the preparation of thermally stable hexacoordinate dimethylantimony(V) compounds of the type Me_2SbX_2Y in which X represents a halogen atom and Y represents a monoprotic bidentate ligand such as an acetylacetonate[8] or an oxinate[9] group. The present paper deals with the successful preparation of thermally stable dialkylantimony(V) compounds of the type R_2SbX_3 in which X represents an alkoxo or acylato group. In addition, the preparation of novel hydroxo(oxo)dialkylantimony compounds $R_2Sb(O)OH$ is described. The structures of $Me_2Sb(OMe)_3$ and $Me_2Sb(OAc)_3$ are discussed in greater detail on the basis of IR, Raman and PMR spectroscopic studies.

RESULTS AND DISCUSSION

Preparation of trialkoxodialkylantimony compounds

The hitherto unknown trialkoxodialkylantimony compounds $R_2Sb(OR')_3$ (R = Me, Et, Pr, Bu; R' = Me, Et) have been prepared by essentially the same procedures as those reported for the preparation of dialkoxotrialkylantimony[10,11,12], $R_3Sb(OR')_2$, and pentaalkoxoantimony[13], $Sb(OR')_5$, compounds. Reaction of bromodialkylstibines[2,14,15], R_2SbBr (R = Et, Pr, Bu), with bromine at −20 to −40° in pentane solution results in the precipitation of tribromodialkylantimony compounds as pale yellow crystalline solids. Subsequent addition of the appropriate amount of sodium alkoxide in the corresponding alcohol results in the formation of the corresponding trialkoxodialkylantimony compounds.

$$R_2SbBr + Br_2 \xrightarrow[-20^\circ \text{ to } -40^\circ]{\text{pentane}} R_2SbBr_3$$

$$R_2SbBr_3 + 3\ NaOR' \xrightarrow[-20^\circ \text{ to } -40^\circ]{\text{pentane/R'OH}} R_2Sb(OR')_3 + 3\ NaBr$$

(R = Et, Pr, Bu; R' = Me, Et)

The reaction mixture has to be vigorously stirred and the reaction temperature should not be allowed to rise above −20°, particularly during the bromination of R_2SbBr, since at higher temperatures decomposition of R_2SbBr_3 followed by bromination of the decomposition products leads to the formation of large amounts of side-products, which prevent the isolation of pure $R_2Sb(OR')_3$. Trimethoxodimethylantimony has similarly been prepared from Me_2SbCl_3. The trimethoxo derivatives are obtained in yields up to 70%. The triethoxo derivatives, however, are obtained in much lower yields, and are more conveniently prepared by exchange between the trimethoxo derivatives and ethanol:

$$R_2Sb(OMe)_3 + EtOH_{\text{excess}} \rightarrow R_2Sb(OEt)_3 + 3\ MeOH$$

The boiling points and analytical data for all compounds prepared are given in Table 1. The trialkoxodialkylantimony compounds display a high thermal stability. No sensitivity towards oxidation by oxygen in the air has been observed, but contact with moist air results in rapid hydrolysis.

TABLE 1

BOILING POINTS AND ANALYTICAL DATA FOR SOME TRIALKOXODIALKYLANTIMONY COMPOUNDS

Compound	B.p. [°C (mm)]	Analyses, found (calcd.) (%)		
		C	H	Sb
$Me_2Sb(OMe)_3$	[a]	23.98 (24.52)	6.05 (6.17)	49.86 (49.71)
$Et_2Sb(OMe)_3$	74–80(0.2)	31.03 (30.80)	6.98 (6.96)	44.71 (44.63)
$Pr_2Sb(OMe)_3$	90–95(0.08)	36.05 (35.91)	7.60 (7.64)	40.25 (40.47)
$Bu_2Sb(OMe)_3$	88–90(0.15)	42.48 (40.16)	8.62 (8.21)	37.11 (37.02)
$Et_2Sb(OEt)_3$	70–72(0.2)	36.97 (38.13)	7.92 (7.94)	38.87 (38.67)
$Pr_2Sb(OEt)_3$	62–66(0.03)	41.98 (42.02)	8.55 (8.46)	35.98 (35.57)
$Bu_2Sb(OEt)_3$	82–86(0.06)	45.64 (45.32)	9.03 (8.90)	33.06 (32.83)

[a] M.p. 80–86° (softens at 60°), determined in a sealed capillary under dry nitrogen.

TABLE 2

PHYSICAL AND ANALYTICAL DATA FOR SOME TRIACETATODIALKYLANTIMONY COMPOUNDS

Compound	B.p. [°C (mm)]	M.p.[a] (°C)	Analyses, found (calcd.) (%)		
			C	H	Sb
$Me_2Sb(OAc)_3$		110–112	29.82 (29.20)	4.70 (4.56)	36.73 (37.02)
$Et_2Sb(OAc)_3$	98–102(0.06)	40–43	33.64 (33.64)	5.51 (5.32)	34.34 (34.12)
$Pr_2Sb(OAc)_3$	118–122(0.06)	55–57	37.20 (37.43)	5.68 (5.97)	31.96 (31.63)
$Bu_2Sb(OAc)_3$	146(0.5)		41.26 (40.70)	6.87 (6.54)	29.93 (29.49)

[a] Determined in a sealed capillary under dry nitrogen.

Preparation of triacetatodialkylantimony compounds

Attempts to prepare triacetatodialkylantimony compounds by treatment of R_2SbBr_3 at -20° to -40° with sodium acetate in THF solution were unsuccessful, probably because of the low solubility of sodium acetate in the reaction medium. On the other hand addition of acetic acid to trialkoxodialkylantimony compounds results in the immediate formation of the corresponding triacetato derivatives:

$$R_2Sb(OMe)_3+CH_3COOH_{excess} \rightarrow R_2Sb(OOCCH_3)_3+3\ MeOH$$

This reaction opens a general route to the preparation of triacylatodialkylantimony compounds. $Me_2Sb(OOCCH_3)_3$ has also been otained by treatment of Me_2SbCl_3 with silver acetate in benzene.

Like the trialkoxodialkylantimony compounds, the triacetatodialkylantimony compounds are stable towards oxidation by oxygen in the air but extremely sensitive towards hydrolysis by moist air. Physical and analytical data for the compounds prepared are given in Table 2.

Preparation of hydroxo(oxo)dialkylantimony compounds

Hydrolysis of trialkoxodialkylantimony compounds affords the hitherto practically unexplored hydroxo(oxo)dialkylantimony compounds.

$$R_2Sb(OR')_3 + 2\,H_2O \rightarrow R_2Sb(O)OH + 3\,R'OH$$
$$(R = Me, Et, Pr, Bu;\ R' = Me, Et)$$

The only report concerning such compounds involves the preparation of the methyl derivative by hydrolysis of the air-oxidation product of Me_2SbCl[7]. The diethyl, dipropyl and dibutyl derivatives can be recrystallized from THF or moist acetone. The methyl derivative is insoluble in the common organic solvents. However, the product obtained upon hydrolysis of $Me_2Sb(OMe)_3$ is essentially pure. The hydroxo(oxo)dialkylantimony compounds are colourless solids, which show a high thermal stability and, therefore, form very suitable starting materials for the synthesis of other dialkylantimony(V) compounds. Melting points and analytical data are given in Table 3.

TABLE 3

MELTING POINTS AND ANALYTICAL DATA FOR SOME HYDROXO(OXO)DIALKYLANTIMONY COMPOUNDS

Compound	M.p. (°C)	Analyses, found (calcd.) (%)		
		C	H	Sb
$Me_2Sb(O)OH$	[a]	12.99 (13.00)	3.68 (3.81)	65.06 (65.89)
$Et_2Sb(O)OH$	214–217	22.89 (22.57)	5.20 (5.17)	57.20 (57.22)
$Pr_2Sb(O)OH$	170–185	30.08 (29.91)	6.36 (6.23)	50.34 (50.56)
$Bu_2Sb(O)OH$	140–155[b]	35.82 (35.72)	7.27 (7.07)	45.62 (45.29)

[a] Does not melt. [b] Softens above 80°.

Results of molecular weight determinations

The molecular weights of the various trialkoxo- and triacetatodialkylantimony compounds have been measured by osmometry in benzene. However, obviously as a result of extreme sensitivity of these compounds towards hydrolysis, erratic values were obtained. Cryometry in benzene solution under a dry nitrogen atmosphere afforded reliable results. Triacetatodialkylantimony compounds $R_2Sb(OAc)_3$ (R = Me, Et, Pr) appear to be monomeric in benzene solution. For each compound the molecular weight was found to be independent of the concentration which varied within the range 0.5–1.2 wt. %. Trimethoxodialkylantimony compounds show a weak tendency towards association. For $Et_2Sb(OMe)_3$ the degree of association varies from

1.1–1.4 within the concentration range 0.4–1.1 wt. %. $Me_2Sb(OMe)_3$ most probably is a dissociating dimer. The degree of association increases from 1.4 to 1.9 when the concentration is increased from 0.4 to 1.5 wt. %.

PMR spectroscopy

PMR spectroscopy appeared very useful for the determination of the purity of the dialkylantimony(V) compounds prepared. Whereas according to their analytical data the trialkoxo- and triacetatodialkylantimony compounds appeared to have been isolated in good purity, PMR spectroscopy in some cases revealed the presence of trace amounts of side products. This contamination is explained in terms of the presence of traces of trialkylstibine and dibromoalkylstibine in the bromodialkylstibine starting materials. We have found that Bu_2SbBr obtained upon thermal cracking of Bu_3SbBr_2 contains traces of Bu_3Sb and $BuSbBr_2$ even after repeated distillation. Methylation of the Bu_2SbBr thus obtained resulted in the isolation of Bu_2SbMe contaminated with Bu_3Sb and $BuSbMe_2$. The same phenomenon was observed upon thermal cracking of Me_3SbBr_2, Me_2SbBr contaminated with Me_3Sb and $MeSbBr_2$ being isolated[16].

Table 4 shows the PMR spectral data for a series of R_2SbX_3 compounds (R = Me, Et; X = OMe, OAc).

TABLE 4

PMR SPECTRAL DATA FOR A SERIES OF R_2SbX_3 COMPOUNDS (R = Me, Et; X = OMe, OAc) IN BENZENE SOLUTION AT 25°

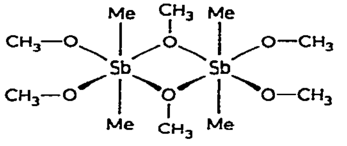
Compound	Chemical shifts, δ^a				
	CH_3-(Sb)	$-CH_2$-(Sb)	CH_3-(CH_2Sb)	$-OCH_3$	$-OOCCH_3$
$Me_2Sb(OMe)_3$	1.32			3.53	
$Et_2Sb(OMe)_3$		2.06	1.39	3.67	
$Me_2Sb(OAc)_3$	2.19				1.74
$Et_2Sb(OAc)_3$		2.85	1.41		1.80

[a] ppm downfield from TMS.

Low-temperature PMR spectra of $Me_2Sb(OAc)_3$ and $Et_2Sb(OAc)_3$ in toluene-d_8 show, even at −80°, a singlet due to the acetato group proton resonances. The observed magnetic equivalence of the three acetato groups might be explained in terms of a rigid trigonal bipyramidal structure in which three monodentate acetato ligands occupy equatorial positions, but this compound in fact has a non-rigid hexacoordinate structure (*cf.* the infrared and Raman data).

The PMR spectrum of $Me_2Sb(OMe)_3$ in benzene at 25° shows a sharp singlet for the methoxo group protons (see Table 4). However, at −80° in toluene-d_8 the spectrum displays two methoxo group signals in an approximately 2/1 intensity ratio at δ 3.57 and 3.35 ppm, respectively. These results are in accord with the presence in solution of dimeric $Me_2Sb(OMe)_3$ (*cf.* results of the molecular weight determinations). Whereas, at 25° exchange between bridging and terminal methoxo groups is very rapid, at −80° dimeric $Me_2Sb(OMe)_3$ appears to be rigid within the PMR time scale.

The presence of a very weak band at 570 cm^{-1} [$\nu_{asym.}$(Sb–C$_2$)] and a very strong band at 520 cm^{-1} [$\nu_{sym.}$(Sb–C$_2$)] in the Raman spectrum of crystalline $Me_2Sb(OMe)_3$ clearly indicates the *trans*-disposition of the two methyl groups bonded to antimony. Thus the combined PMR and Raman spectral data support the conclusion that $Me_2Sb(OMe)_3$ possesses a dimeric octahedral structure in which the two methyl groups occupy *trans*-positions (I), both in the solid state and in concentrated solutions.



(I)

Low-temperature PMR spectra of $Et_2Sb(OMe)_3$ in toluene solutions show even at $-80°$ only one signal for the methoxo group protons, but this is broadened.

The PMR spectra of the hydrolysis products of $R_2Sb(OMe)_3$ (R = Pr, Bu) recorded in acetone-d_6 solution confirm the formation of hydroxo(oxo)dialkylantimony compounds. A hydroxo group proton resonance signal is observed at δ 3.40 (R = Pr) and 3.58 ppm (R = Bu). Peak integration showed an alkyl/hydroxo group ratio of 2/1.

Infrared and Raman study of triacetatodimethylantimony.

The relation between the structure of a metal acetate and the positions of the infrared absorption bands of the acetato ligand has been extensively studied (*cf.* ref. 17). For organometal acetates infrared spectroscopic studies have concentrated on the investigation of organotin acetates of the types R_3SnOAc^{18-23} and $R_2Sn(OAc)_2{}^{24}$. The only infrared study of organoantimony acetates[5] deals with the elucidation of the structure of $Me_3Sb(OAc)_2$. The results point to a trigonal bipyramidal structure for this compound, in which the two acetato groups occupy the apical positions. We have applied IR and Raman spectroscopy to elucidate the molecular structure of $Me_2Sb(OAc)_3$.

The infrared spectra in nujol and in CCl_4 and benzene solution show three absorption bands in the COO antisymmetric stretching region. A strong and sharp band at 1723 cm^{-1} points to the presence of acetic acid. Despite extreme precautions obviously some decomposition of $Me_2Sb(OAc)_3$ due to hydrolysis has taken place. In addition, two broad absorption bands are present, one in the region 1680–1620 cm^{-1}, the other in the region 1600–1540 cm^{-1}. In nujol, band maxima were observed at 1640 and 1568 cm^{-1}, in benzene solution at 1666 and 1567 cm^{-1}. In general, antisymmetric COO stretching absorptions for ester-type acetato ligands are observed in the frequency region 1700–1600 cm^{-1}, whereas for bidentate acetato ligands these absorptions are observed in the region 1600–1540 cm^{-1}. Therefore, the results are indicative for the presence of both monodentate ester-type and bidentate acetato groups in $Me_2Sb(OAc)_3$.

In the frequency region 600–200 cm^{-1} the IR spectrum was run as a nujol mull between CsI discs, whereas the Raman spectrum was run on the crystalline solid. The spectra are shown in Fig. 1. Data and tentative assignments of the absorption

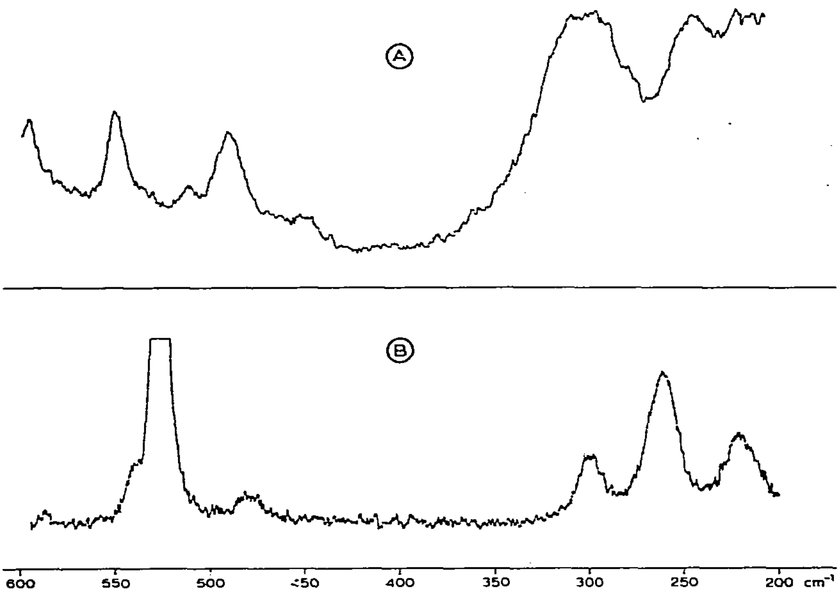


Fig. 1. Vibrational spectra of triacetatodimethylantimony: A. Infrared spectrum in Nujol between CsI discs; B. Raman spectrum of the crystalline solid.

TABLE 5

IR AND RAMAN DATA OF $Me_2Sb(OAc)_3$ IN THE 600–200 cm^{-1} FREQUENCY REGION

IR (nujol mull)	Raman (solid)	Assignment
550	540	$\nu_a(Sb\text{–}C_2)$
	526	$\nu_s(Sb\text{–}C_2)$
511		
491		$\rho(COO)$[a]
	480	
296	299	$\nu_a(Sb\text{–}O_2)$ [b]
	261	$\nu_s(Sb\text{–}O_2)$ [b]
245		$\nu_a(Sb\text{–}O_2)$ [c]
220	221	$\nu_s(Sb\text{–}O_2)$ [c]

[a] According to ref. 17. [b] Due to ester-type acetato group. [c] Due to bidentate acetato group.

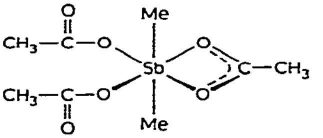
bands are tabulated in Table 5.

The appearance of a very weak antisymmetric $Sb–C_2$, and the presence of a very strong symmetric $Sb–C_2$ stretching band in the Raman spectrum of crystalline $Me_2Sb(OAc)_3$ together with the absence of a symmetric $Sb–C_2$ stretching absorption in the IR spectrum, clearly indicate the *trans*-disposition of the two methyl groups bound to antimony.

The presence of two different types of acetato ligands, already indicated by the IR spectrum in the 1700–1500 cm^{-1} region is confirmed by the appearance of two sets of $Sb–O_2$ stretching absorptions. The strong IR band at 296 cm^{-1} and the

Raman bands at 299 (weak) and 261 (medium) cm^{-1} are indicative of the presence of two ester-type bonded acetato groups in *cis*-position. These absorptions are observed in the same region as the Sb–O_2 stretching frequencies of $R_3Sb(OAc)_2$ compounds which contain ester-type bonded acetato ligands[25]. Recently Hester studied the IR and Raman spectra of Me_3SnOAc[23]. This compound was found to contain a bidentate acetato group in the solid state[18–23]. The far-infrared spectrum of a nujol mull showed two absorptions at 258 and 208 cm^{-1} which were tentatively assigned to the antisymmetric and symmetric Sn–O_2 stretching modes, respectively. Therefore, although the data are limited, we suggest that the absorptions observed in the 200–250 cm^{-1} region are due to SbO_2 stretching vibrations of a bidentate acetato group.

The combined IR and Raman spectral data support the conclusion that $Me_2Sb(OAc)_3$ possesses an octahedral structure in which, apart from possible minor distortions, two methyl groups occupy axial positions, whereas two ester-type and one bidentate acetato group occupy the equatorial positions (II).



(II)

In this connection, it is of interest to recall the presence of only one signal for the acetato group protons, in the PMR spectrum of this compound. Apparently, even at −80°, $Me_2Sb(OAc)_3$ has a non-rigid octahedral structure, the time-averaged situation being, however, that of the three acetato groups bound to antimony, two are of the monodentate ester-type, whereas the third one acts as a bidentate ligand.

The presence of two types of acetato groups in one molecule is not unprecedented, since Kurosawa and Okawara[26] recently reported the presence of both an ester-type and a bidentate acetato ligand in $MeTl(OAc)_2$ and $PhTl(OAc)_2$.

CONCLUSION

So far, the chemistry of dialkylantimony(V) derivatives has been seriously hampered by the thermal instability of trihalodialkylantimony compounds. The successful preparation of thermally stable trimethoxodialkylantimony compounds and of hydrolytically and thermally stable hydroxo(oxo)dialkylantimony compounds has now opened a route to the synthesis of new types of diorganoantimony(V) compounds.

EXPERIMENTAL PART

General

Since most of the organoantimony compounds mentioned in this paper are oxygen and/or moisture-sensitive, all manipulations were performed in an atmosphere of dry, oxygen-free nitrogen, unless indicated otherwise. Liquids were handled by the syringe technique.

PMR spectra were recorded by Miss L. Veldstra, using a Varian Associates HA-100 NMR spectrometer.

Far-infrared absorption spectra were run by Messrs R. Elst and G. C. Schoemaker at the Laboratory for Inorganic Chemistry of the University of Amsterdam, using Beckman IR 7 and IR 11 spectrometers. Raman spectra were run by Mr. W. Gabes on a Coderg PH 1 spectrometer, equipped with a CRL model 53 MG Kr/Ar laser using 6471 Å radiation, at the Laboratory for Inorganic Chemistry of the University of Amsterdam.

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

Preparation of trialkoxodialkylantimony compounds

Trimethoxodimethylantimony. Trichlorodimethylantimony[7] (7.3 g, 28 mmoles) was added to a solution of sodium methoxide (87 mmoles) in methanol (60 ml). After stirring for 1 h the precipitate of sodium chloride was removed by filtration. The methanol solution was evaporated and pentane (50 ml) was added to the colourless residue. The mixture was heated to boiling and subsequently cooled and filtrated. Upon evaporation the filtrate afforded 3.5 g of colourless $Me_2Sb(OMe)_3$, m.p. 80–86°. Yield 50.5%.

Trimethoxodiethylantimony. Bromine (16.0 g, 100 mmoles) in pentane (50 ml) was added dropwise to a vigorously stirred solution of Et_2SbBr[7,15] (26.3 g, 100 mmoles) in pentane (50 ml) kept at −20 to −40°. The bromine solution decolourized immediately and pale yellow Et_2SbBr_3 precipitated. At −40° sodium methoxide (300 mmoles) in absolute methanol (150 ml) was added dropwise, while vigorous stirring was maintained. Afterwards, the mixture was allowed to come to room temperature and the pentane/methanol mixture was evaporated under reduced pressure. Benzene (75 ml) was added, the mixture was heated to boiling and subsequently cooled and filtered. Upon distillation 16.5 g of pure $Et_2Sb(OMe)_3$ [b.p. 74–80° (0.2 mm)] was isolated. Yield 60.4%. Traces of impurities, probably $Et_nSb(OMe)_{(3-n)}$ ($n=0$–2), sublime out of the crude reaction product just below the boiling point of $Et_2Sb(OMe)_3$. These have to be removed before distillation.

Trimethoxodipropyl- and trimethoxodibutylantimony. These compounds were isolated in up to 70% yields by essentially the same procedure as described for the diethylantimony derivative. Minor quantities of solid impurities have to be sublimed out of the crude reaction product before distillation.

Triethoxodiethyl-, triethoxodipropyl- and triethoxodibutylantimony. The compounds were obtained in quantitative yield by repeated refluxing of the corresponding trimethoxo derivatives with excess of EtOH and subsequently distilling off of a mixture of MeOH and EtOH. Finally the triethoxodialkylantimony compounds were distilled *in vacuo.*

Preparation of triacetatodialkylantimony compounds

Triacetatodimethyl-, triacetatodiethyl-, triacetatodipropyl- and triacetatodibutylantimony. These compounds were obtained in quantitative yields upon addition of acetic acid to a benzene solution of the corresponding trimethoxodialkylantimony compounds in a 3/1 molar ratio. After removal of volatile products *in vacuo* the triacetatodialkylantimony compounds were obtained in high purity.

Triacetatodimethylantimony. Silver acetate (15.0 g, 90 mmoles) was added to a solution of Me_2SbCl_3[7] (7.7 g, 30 mmoles) in benzene (120 ml). The reaction mixture was stirred for two days and subsequently filtered over filtrapid. The filtrate was evaporated *in vacuo*, leaving behind a colourless product which was recrystallized from benzene/hexane (1/2) yielding 5.6 g (57%) of $Me_2Sb(OAc)_3$, m.p. 110–112°.

Preparation of hydroxo(oxo)dialkylantimony compounds

Hydroxo(oxo)dimethyl-, hydroxo(oxo)diethyl-, hydroxo(oxo)dipropyl- and hydroxo(oxo)dibutylantimony. These compounds were obtained as colourless solids upon hydrolysis of the corresponding trialkoxodialkylantimony compounds in water. The diethylantimony derivative was recrystallized from THF, the dipropyl- and dibutylantimony derivatives from acetone and acetone/water, respectively. All manipulations were performed in air. Yields were almost quantitative.

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