JOURNAL OF SOLID STATE CHEMISTRY 108, 243–249 (1994)

A New Polymeric Antimony(III) Oxoalkoxide, $[Sr_2Sb_4O(OEt)_{14}]_n$: The Synthesis and Molecular Structure

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Received February 8, 1993; in revised form May 17, 1993; accepted May 19, 1993

A new bimetallic oxoalkoxide, with composition $Sr_2Sb_4O(OEt)_{14}$, has been synthesized by reacting $Sr(OEt)_2$ with an excess of $Sb(OEt)_3$ in toluene–ethanol under a dry nitrogen atmosphere. Colorless transparent crystals of the alkoxide have been studied by single crystal X-ray diffraction methods. The crystals have a monoclinic space group symmetry $P2_1/n$, with $a = 11.609(3)$ Å, $b = 22.006(5)$ Å, $c = 18.948(5)$ Å, $\beta = 105.08(2)°$, $V = 4673(2)$ Å^3, $Z = 4$, $D_x = 1.861(1)$ g · cm^{-3} and $M_r = 1309.1$. From the X-ray diffraction data collected at 163(1) K, refinements of the derived structure model against the amplitudes of the 3745 most significant reflections converged at an R-value of 0.043. The crystal structure consists of infinite parallel chains extending in the [100] direction. The polymer chains are formed by units of composition $[Sr_2Sb_3O(OEt)_{10}]^+$, that are linked by $[Sb(OEt)_4]^-$ groups through pairs of bridging ethoxy oxygen atoms. The parallel chains are approximately close packed. The oxygen coordination polyhedra around two five-coordinated antimony atoms share a common face. The Sr atoms are seven-coordinated by oxygen atoms. As all the ethoxy oxygen atoms are two-coordinated and the oxo oxygen four coordinated by the metal ions, the formula of the present strontium–antimony oxoethoxide can be written $[Sr_2Sb_4(\mu_4\text{-}O)(\mu\text{-}OEt)_{14}]_n$. © 1994 Academic Press, Inc.

INTRODUCTION

Metal alkoxides have applications as catalysts and as precursors in the so-called sol–gel route for preparation of, e.g., thin oxide layers and oxide materials with special physical properties (1, 2). Knowledge of the molecular structures of metal alkoxides is of vital importance to give a basis for the understanding of various physical and chemical properties, including the gelling process. Although one always has to consider the possibility that the molecular structure in the solid state might differ from the molecular structure in, e.g., diluted solutions, a determination of the crystal structure has the power of giving unambiguous structural information. Presently, a large number of bimetallic alkoxides have been prepared, but the amount of reliable experimental structural information based on, e.g., single crystal diffraction studies is still limited (see 3, 4). As regards bimetallic alkoxides containing trivalent antimony in particular, the preparation of only a limited number of compounds have been published (5–11), although the synthesis of the first bimetallic antimony alkoxide was reported by Meerwein and Bersin as early as 1929. Published experimental data, including both preparation and structural characterisation, for this type of alkoxides is even more limited and comprehend, viz. $Ni_5Sb_3O_2(OEt)_{15}(HOEt)_4$ (9), $Ni_6Sb_4O_4(OEt)_{16}(HOEt)_4$ (10), and $Mg_2Sb_4(OEt)_{16}$ (11). The present study describes the synthesis and structural investigation of a strontium–antimony complex with the chemical composition $Sr_2Sb_4O(OEt)_{14}$. The investigation is a part of a research program on heterometallic alkoxide complexes and their applications for the syntheses of new oxide materials, and is particularly focused towards complexes containing antimony.

EXPERIMENTAL

Preparation. All preparations were performed in a dry nitrogen atmosphere in a glove-box to prevent hydrolysis and decomposition. The toluene and ethanol used, were carefully dried with $Na_{(s)}$ and molecular sieves (3 Å), respectively, and finally saturated with $N_{2(g)}$. Despite the efforts to avoid any hydrolysis, the subsequent X-ray study (cf., below) shows that the obtained specimen contains oxo oxygen atoms. The origin of this oxo content is unclear, but might be due to traces of water or to decomposition of ethoxy groups during formation of the complexes.

$Sb(OEt)_3$ was prepared by adding $SbCl_3$ to an two-phase mixture of hexane and ethanol, through which $NH_{3(g)}$ was bubbled for 2 hr (12). The hexane phase was separated and evaporated until only $Sb(OEt)_3$ was left. The crude $Sb(OEt)_3$ was distilled in vacuum (76–80° C at $3 \cdot 10^{-1}$ Torr). $Sr(OEt)_2$ is easily prepared by simply dissolving $Sr_{(s)}$ (2.7 mmole) in a toluene–ethanol solution (14.4 ml

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0022-4596/94 $6.00

TABLE 1

Experimental Conditions for the Investigation of $[Sr_2Sb_4O(OEt)_{14}]_n$

Formula (X-ray study)	$[Sr_2Sb_4O(OEt)_{14}]_n$
Formula weight	$n \cdot 1309.1$ g · mole^{-1}
Space group	$P2_1/n$
Unit cell dimensions	$a = 11.609(3)$ Å, $b = 22.006(5)$ Å, $c = 18.948(5)$ Å, $\beta = 105.08(2)°$
Unit cell volume, V	4673(2) Å^3
Formula units per unit cell, Z	4
Calculated density, D_x	1.861(1) g · cm^{-3}
Radiation	Mo$K\alpha$
Wavelength, λ	0.71073 Å
Temperature, T	163(1) K
Crystal shape	Prismatic
Crystal size	0.09 × 0.13 × 0.22 mm^3
Diffractometer	Stoe 4-circle
Determination of unit cell	
Number of reflections used	18
2θ-range	16–24°
Intensity data collection	ω–2θ scan technique
Maximum $\sin(\theta)/\lambda$	0.53 Å^{-1}
Range of h, k and l	0 to 12, 0 to 23, and −19 to 19
Standard reflections	3
Intensity instability	<9%
Internal R	0.024
Number of collected reflections	6271
Number of unique reflections	5726
Number of observed reflections	3745
Criterion for significance	$I > 3 \cdot \sigma(I)$
Absorption correction	Numerical integration
Linear absorbtion coefficient	45.5 cm^{-1}
Transmission factor range	0.30–0.51
Structure refinement	Full matrix least squares
Minimization of	$\Sigma\, w(\Delta F)^2$
Anisotropic thermal parameters	Ni, Sb and O atoms
Isotropic thermal parameters	C and H atoms
Number of refined parameters	302
Weighting scheme	$(\sigma^2(F) + 0.0005\|F\|^2)$
Final R for observed reflections	0.043
Final R_w for observed reflections	0.054
Final R_w for all 5726 reflections	0.058
Final $(\Delta/\sigma)_{max}$	0.06
Final $\Delta\rho$ and $\Delta\rho$	1.0 and −0.9 $e^- \cdot$ Å^{-3}

and 3.8 ml, respectively). The title compound, with the composition $Sr_2Sb_4O(OEt)_{14}$, was prepared by reacting $Sr(OEt)_2$ with an excess (1 : 5) of $Sb(OEt)_3$ (2.7 mmole and 13.3 mmole, respectively) in a 4 : 1 (volume ratio) solution of toluene and ethanol. The estimated yields of the syntheses as judged by the Sr contents, are over 60%.

$Sr_2Sb_4O(OEt)_{14}$ is soluble in mixtures of toluene and ethanol. However, $Sr_2Sb_4O(OEt)_{14}$ decomposes within a day in the absence of ethanol in pure toluene. The yellow solutions obtained upon decomposition have not been further characterized. A solution of $Sr_2Sb_4O(OEt)_{14}$ in toluene–ethanol (4 : 1), was slowly evaporated under vacuum. During this process the solution became more concentrated in toluene. To avoid decomposition, small portions of a toluene–ethanol solution with a higher (2 : 1) ethanol content was added intermittently. Suitable transparent, needle shaped crystals grew from the solution over a few weeks.

FT-IR spectra have been recorded (Mattson Polaris spectrometer) for several freshly prepared samples of $Sr_2Sb_4O(OEt)_{14}$ in KBr and toluene–ethanol (4 : 1) solution. The differences between the recorded IR spectra are discussed below. Analysis of the metal content using a scanning electron microscopy (JEOL 820) equipped for energy dispersive analysis of X-ray spectra (LINK AN10000), indicated Sr : Sb ratios of about 1 : 2 for the samples.

Structure determination. Crystals of suitable dimen-

TABLE 2

Fractional Atom Coordinates (× 10^4) for Non-hydrogen Atoms and Thermal Parameters (× 10^3 Å^2) with ESDs for $[Sr_2Sb_4O(OEt)_{14}]_n$

Atom	*x*	*y*	*z*	U_{iso}
Sb(1)	949(1)	4110(1)	3947(1)	27(1)
Sb(2)	261(1)	4977(1)	2566(1)	26(1)
Sb(3)	-141(1)	2547(1)	1430(1)	31(1)
Sb(4)	-4919(1)	3237(1)	1952(1)	30(1)
Sr(1)	2152(1)	3620(1)	2387(1)	27(1)
Sr(2)	-1600(1)	3628(1)	2441(1)	26(1)
O(1)	474(8)	4098(4)	2852(4)	20(3)
O(2)	-601(9)	4774(5)	3491(5)	39(4)
O(3)	1888(9)	4895(4)	3569(5)	32(4)
O(4)	2281(8)	3559(5)	3744(5)	33(4)
O(5)	-371(9)	3425(4)	3763(5)	35(4)
O(6)	1275(9)	4671(4)	1868(5)	34(4)
O(7)	-1348(8)	4669(4)	1904(5)	27(4)
O(8)	921(9)	2654(4)	2434(5)	34(4)
O(9)	1389(9)	2952(4)	1234(5)	31(4)
O(10)	-1509(8)	2537(4)	1992(5)	36(4)
O(11)	-710(10)	3404(4)	1346(5)	35(4)
O(12)	3932(8)	2879(4)	2556(5)	34(4)
O(13)	4215(9)	3985(4)	2141(6)	38(4)
O(14)	-3541(9)	3300(5)	2836(5)	35(4)
O(15)	-3681(9)	3785(5)	1592(5)	38(4)
C(2a)	-820(17)	5250(9)	3948(11)	67(6)
C(2b)	-2096(17)	5381(9)	3836(11)	79(7)
C(3a)	2377(18)	5357(8)	4057(10)	64(6)
C(3b)	2682(20)	5891(10)	3815(13)	96(8)
C(4a)	3194(15)	3346(8)	4367(9)	47(5)
C(4b)	4033(15)	3846(8)	4706(9)	54(5)
C(5a)	-245(19)	2966(9)	4351(10)	69(6)
C(5b)	-349(20)	3212(10)	5042(12)	93(8)
C(6a)	1399(16)	5114(8)	1370(10)	53(5)
C(6b)	1995(16)	4896(8)	825(10)	61(6)
C(7a)	-2225(14)	5131(7)	1761(8)	39(4)
C(7b)	-2240(15)	5489(7)	1071(9)	49(5)
C(8a)	816(14)	2273(6)	3028(8)	31(4)
C(8b)	889(16)	1617(7)	2890(9)	52(5)
C(9a)	1858(15)	2695(7)	697(9)	41(5)
C(9b)	2125(16)	3161(7)	163(9)	51(5)
C(10a)	-2391(16)	2099(8)	1794(9)	51(5)
C(10b)	-2631(19)	1811(9)	2453(11)	79(7)
C(11a)	-648(14)	3743(7)	726(8)	36(4)
C(11b)	-1237(15)	3448(8)	2(9)	50(5)
C(12a)	3772(15)	2240(7)	2460(9)	40(4)
C(12b)	4319(16)	1906(8)	3169(9)	56(5)
C(13a)	4813(15)	4564(8)	2288(9)	47(5)
C(13b)	4341(15)	4923(8)	2809(9)	51(5)
C(14a)	-3648(16)	3225(8)	3560(9)	51(5)
C(14b)	-3239(19)	3807(9)	3994(11)	81(7)
C(15a)	-3847(18)	3787(9)	814(10)	66(6)
C(15b)	-4743(18)	4196(9)	418(11)	80(7)

TABLE 3

Selected Bond Distances in (Å) with ESDs for $[Sr_2Sb_4O(OEt)_{14}]_n$

Sb(1)–O(1)	2.00(1)
Sb(1)–O(2)	2.31(1)
Sb(1)–O(3)	2.26(1)
Sb(1)–O(4)	2.08(1)
Sb(1)–O(5)	2.11(1)
Sb(2)–O(1)	2.01(1)
Sb(2)–O(2)	2.28(1)
Sb(2)–O(3)	2.31(1)
Sb(2)–O(6)	2.10(1)
Sb(2)–O(7)	2.08(1)
Sb(3)–O(8)	1.99(1)
Sb(3)–O(9)	2.11(1)
Sb(3)–O(10)	2.13(1)
Sb(3)–O(11)	1.99(1)
Sb(4)–O(12)	2.12(1)
Sb(4)–O(13)	2.01(1)
Sb(4)–O(14)	2.00(1)
Sb(4)–O(15)	2.12(1)
Sr(1)–O(1)	2.56(1)
Sr(1)–O(4)	2.54(1)
Sr(1)–O(6)	2.62(1)
Sr(1)–O(8)	2.58(1)
Sr(1)–O(9)	2.59(1)
Sr(1)–O(12)	2.59(1)
Sr(1)–O(13)	2.68(1)
Sr(2)–O(1)	2.55(1)
Sr(2)–O(5)	2.58(1)
Sr(2)–O(7)	2.56(1)
Sr(2)–O(10)	2.56(1)
Sr(2)–O(11)	2.60(1)
Sr(2)–O(14)	2.65(1)
Sr(2)–O(15)	2.55(1)

sions of the $Sr_2Sb_4O(OEt)_{14}$ specimen were selected and mounted into glass capillary tubes (∅ = 0.5 mm) within the glove box. The crystals are very fragile, which makes them difficult to handle, and they start to decompose when removed from their mother liquid. By lowering the temperature, the rate of decomposition was found to be negligible. After a suitable crystal was successfully mounted, it was rapidly transferred to a STOE 4-circle single crystal X-ray diffractometer equipped with cryostat ($N_{2(g)}$ gas stream) operating at 163 K.

Preliminary investigations of a crystal, using Mo*K*α radiation, indicated a primitive monoclinic space group symmetry. Although the selected crystal had a small twin component (<10%), it was possible to find the orientation matrix for the major crystal, which was used for the subsequent X-ray intensity data collection. The unit cell parameters, a = 11.609(3) Å, b = 22.006(5) Å, c = 18.948(5) Å and β = 105.09(2)°, were refined from accurate θ set-

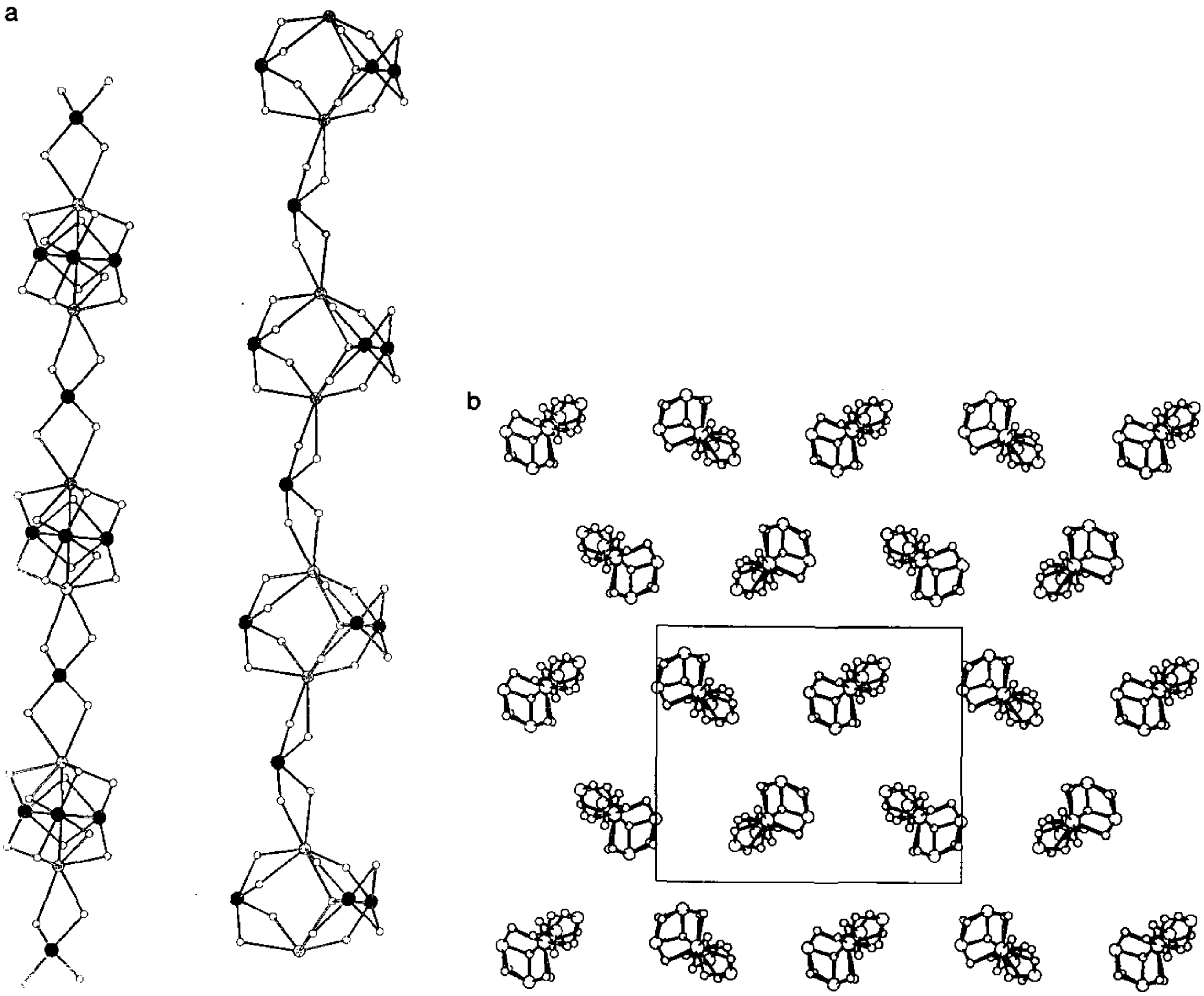

FIG. 1. (a) The parallel polymer chains of $[Sr_2Sb_4O(OEt)_{14}]_n$, viewed perpendicular to the [100] direction. Sb, Sr, and O atoms are drawn as black filled, gray filled, and unfilled circles, respectively. Carbon and hydrogen atoms are omitted. (b) Packing of the chains of $[Sr_2Sb_4O(OEt)_{14}]_n$. Viewed along the [100] direction, [010] is horizontal, [001] vertical and the origin is the lower left corner. Carbon and hydrogen atoms are omitted.

tings of 18 well-centered reflections. The intensity data were corrected for background, Lorentz, polarization, and absorption effects. The possible space group symmetry, $P2_1/n$ (No. 14), was deduced from the systematic extinctions among the diffraction data. Intensity statistics further supported a centrosymmetric space group symmetry. Further details on the experimental conditions are given in Table 1.

Preliminary positions of the antimony atoms were obtained by direct methods, using the program package SHELXS-86 (13). The positions of the other nonhydrogen atoms were found from subsequent calculations of difference electron density ($\Delta\rho$) maps, using the program system SHELX-76 (14) . The hydrogen atoms were positioned with ideal geometry and refined by constraining them to geometrically predicted positions around the carbon atoms (C–H distances of 1.0 Å and bond angles of 109.5°). Least-squares refinement of the structure model, using atomic X-ray scattering factors for neutral atoms from the "International Tables for X-ray Crystallography" (15), yielded an R-value of 0.043. Details on the final structural refinement are given in Table 1. Geometrical calculations on the structural model were performed with *inter alia* the program PLATON (16). The final atomic coordinates of the nonhydrogen atoms are listed in Table 2.

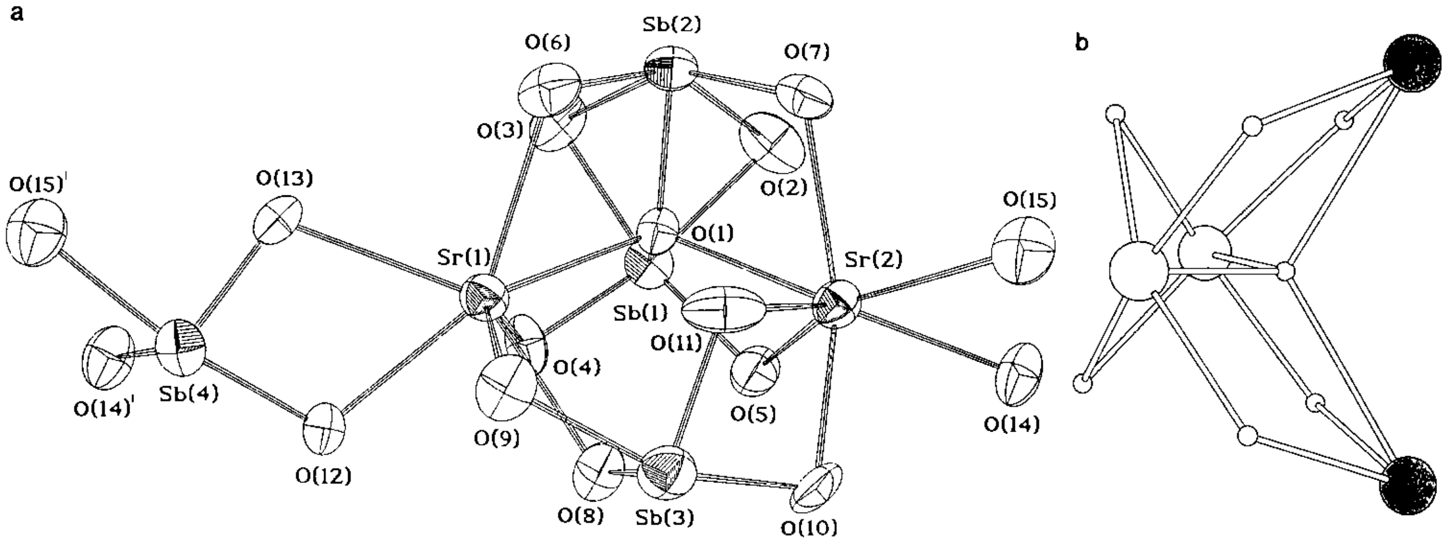

FIG. 2. (a) Thermal ellipsoid plot (ellipsoids drawn at 75% probability) perpendicular to [100], showing the metal and oxygen atoms (with labels) in the polymeric units of $Sr_2Sb_4O(OEt)_{14}$. The oxygen atoms with primed labels denote translation related atoms in an adjacent unit cell. (b) Tetrameric oxoalkoxide fragment $[Sr_2Sb_2O(OEt)_6]^{2+}$, showing the metal and oxygen positions, within $[Sr_2Sb_4O(OEt)_{14}]_n$.

STRUCTURE DESCRIPTION AND DISCUSSION

The crystal structure consists of parallel polymer chains (Fig. 1a), formed by rather globular units of the composition $[Sr_2Sb_3O(OEt)_{10}]^+$, that are linked by $[Sb(OEt)_4]^-$ groups via bridging ethoxy oxygen atoms, to give $[Sr_2Sb_4O(OEt)_{14}]_n$ chains resembling "strings of pearls" that extend in the [100] direction. The polymer chains can, when viewed along the [100] direction (Fig. 1b), roughly be described as being closely packed. As there are no intermolecular hydrogen bonds present, the structure is held together mainly by van der Waals forces. Polymeric chains have earlier been observed in some other alkoxides, as the bimetallic tin alkoxides $[M\text{Sn}(\mu\text{-O}^t\text{Bu})_3]_n$ (where M = Li, Na, K, Rb, or Cs) (4) and $[Tl_2Sn(OEt)_6]_n$ (17). Fig. 2a shows a thermal ellipsoid plot (18) of the metal and oxygen atoms within a polymer unit, together with the atom labeling scheme used for the metal and the oxygen atoms. The α and β carbon atoms of the ethoxy groups have been labeled with the number of the oxygen atoms followed by the letters a and b, respectively.

Two of the antimony atoms, Sb(1) and Sb(2), are five-coordinated by four ethoxy oxygen atoms and by the oxo oxygen atom. The square pyramidal coordination polyhedra around two adjacent Sb(1) and Sb(2) atoms are face-sharing, as three of the coordinating oxygen atoms, two ethoxy and the oxo oxygen atom, are common to the two polyhedra. Such face-sharing have not been observed in other bimetallic antimony oxo alkoxides, where the antimony ions do not share any coordination elements. In this connection, it should be noted that the present compound was prepared with a large excess of $Sb(OEt)_3$, which probably promotes the occurrence of antimony ions with common coordinating atoms. If the stereo active lone pair regions are also taken into account, the coordination geometries around these two antimony atoms can be regarded as distorted octahedra. The two other antimony atoms, Sb(3) and Sb(4), atoms are both four-coordinated by ethoxy oxygen atoms and have the approximate coordination geometries of distorted trigonal bipyramids (tbp), if the lone pair regions are included. The four coordinated antimony ion Sb(3) of the globular unit $[Sr_2Sb_3O(OEt)_{10}]^+$ is not linked via bridging oxygen atoms to any of the other (five coordinated) antimony ions within the unit. The observed coordination geometries around all four antimony ions and the uneven bond distances observed are in accordance with those found in other bimetallic antimony alkoxides (see 9–11). A more thorough discussion on the general features of four-coordinated antimony can be found in the paper on $Mg_2Sb_4(OEt)_{16}$ (11). By calculating the values of the bond valence sums (bvs-values), using the parameters given by Brown and Altermatt (19), any major anomalies in the bond distance distributions (and coordination) is likely to be revealed. In the present case the bvs-values of the antimony atoms are 3.23, 3.20, 3.25, and 3.18 for the Sb(1–4) atoms, respectively. The bvs-values for trivalent antimony ions have in bimetallic alkoxides found to be slightly higher than the formal charge (9–11).

The strontium atoms are seven coordinated by oxygen atoms. The coordination polyhedra can possibly be described as very distorted monocapped octahedra. The two strontium ions atoms, Sr(1) and Sr(2), have bond distance

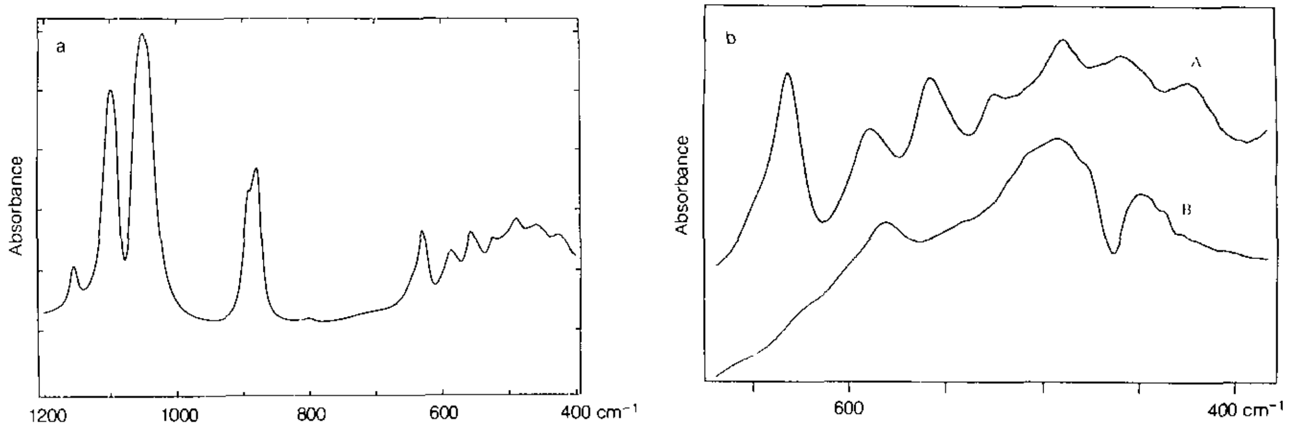

FIG. 3. (a) FT-IR spectra of $[Sr_2Sb_4O(OEt)_{14}]_n$ mixed with KBr and (b) mixed with KBr (A) and toluene-ethanol (4 : 1) solution with a $Sr_2Sb_4O(OEt)_{14}$ concentration of 0.5 M (B), in the M–O region 650–400 cm^{-1}. The dip at 463 cm^{-1} in the lower curve (B) is due to background effects from the solvent.

averages of 2.60(5) and 2.58(4) Å, respectively. The two longest Sr–O bonds (2.68 and 2.65 Å) are formed to oxygen atoms (O(13) and O(14)) that are equatorial in the tbp-coordination geometry of the bridging Sb(4) atom. The calculated bvs-values (1.94 and 2.02) for the strontium ions agree well with their formal charges.

By disregarding the terminal ethoxy groups a "tetrameric" oxoalkoxide fragment ($[Sr_2Sb_2O(OEt)_6]^{2+}$) of the globular unit $[Sr_2Sb_3O(OEt)_{10}]^+$ is obtained (Fig. 2b). The fragment, which consist of the four metal ions that coordinate the μ_4-oxo ion has a close resemblance to the "tetrameric" units observed in *inter alia* $Al_4O(O^iBu)_{11}H$ (20), $Al_{10}O_4(OEt)_{22}$ (21), and $Ce_4O(O^iPr)_{13}(HO^iPr)$ (22), and also some resemblance to the units in the bimetallic oxoalkoxide carboxylate $Cd_4Sn_4O_2(O_2CMe)_{10}(OCH_2{}^tBu)_{10}$ (23). One major difference being that, while the two antimony atoms are linked by *inter alia* two ethoxy oxygen bridges there is only one such bridge in the aluminum alkoxides. By considering this fragment as a major building unit, the complete $[Sr_2Sb_4O(OEt)_{14}]_n$ chain is formed, by adding two more $[Sb(OEt)_4]^-$ units, to give a neutral complex and fulfil the high coordination number (seven) of the two strontium atoms.

Preliminary results show that the structure of $Sr_2Sb_4O(OEt)_{14}$ is related to that of a Ba–Sb oxoalkoxide, $Ba_2Sb_4O(OEt)_{14}(HOEt) \cdot Ba_2Sb_4O(OEt)_{14}$ (24), which, however, contains two different types of polymeric chains. One of the chain types is very similar to that found in the present structure, while the other one differs significantly. The latter contains an extra ethanol group in each polymer unit, that coordinates one of the barium atoms and participates in an intramolecular hydrogen bond. The root mean square deviation between superposed (25) related polymeric units of the two alkoxides is 0.22 Å, which is of about the same magnitude as the difference between the ionic radii of the Sr^{2+} and Ba^{2+} ions.

The geometries of the ethoxy groups are rather normal. The averages of the O–C(a) and C(a)–C(B) bond distances and of the O–C(a)–C(b) bond angles are 1.42(3) Å, 1.48(5) Å, and 113.2(3)°, respectively.

The FT-IR spectra of $Sr_2Sb_4O(OEt)_{14}$, in solid state (mixed with KBr) and in toluene–ethanol [4 : 1] solution (0.5 M) are shown in Fig. 3. In Fig. 3a the spectrum of $Sr_2Sb_4O(OEt)_{14}$ in KBr shows the absorption from the C–O and M–O stretchings in the region 1200–400 cm^{-1}. The spectra of the solid and the solution are particularly different in the M–O stretching region, 650–400 cm^{-1} which are shown in Fig. 3b. As the polymeric chain structure is not expected to be retained in the solution such a difference is anticipated.

ACKNOWLEDGMENT

This research has been financially supported by the Swedish Natural Science Research Council.

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