Synthesis, crystal structures and spectroscopic properties of pure YSb$_2$O$_4$Br and YSb$_2$O$_4$Cl as well as Eu$^{3+}$- and Tb$^{3+}$-doped samples‡

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The quaternary halide-containing yttrium(II) oxidoantimonates(III) YSb$_2$O$_4$Cl and YSb$_2$O$_4$Br were synthesised through solid-state reactions from the binary components (Y$_2$O$_3$, Sb$_2$O$_3$ and YX$_3$, X = Cl and Br) at 750 °C in evacuated fused silica ampoules with eutectic mixtures of NaX and CsX (X = Cl and Br) as fluxing agents. YSb$_2$O$_4$Cl crystallizes tetragonally in the non-centrosymmetric space group $P4_2$2 with unit-cell parameters of $a = 773.56(4)$ pm and $c = 878.91(6)$ pm, whereas YSb$_2$O$_4$Br is monoclinic (space group: $P2_1/c$) with $a = 896.54(6)$ pm, $b = 780.23(5)$ pm, $c = 779.61(5)$ pm and $\beta = 91.398(3)^\circ$, both for $Z = 4$. The two new YSb$_2$O$_4$X compounds contain [YO$_6$]$^{12-}$ polyhedra, which are connected via four common edges to form $2\infty$ [YO$_8$]$_{13-}$ layers ($d$(Y$^{3+}$-O$^{2-}$) = 225–254 pm) without any Y$^{3+}$…X-bonds ($d$(Y$^{3+}$…X$^{-}$) > 400 pm). Moreover, all oxygen atoms belong to $\psi^1$-tetrahedral [SbO$_3$]$^{3-}$ units, which are either connected to four-membered rings [Sb$_2$O$_4$]$^{6-}$ in the chloride (Y$_2$[Sb$_2$O$_4$]Cl$_2$ for Z = 2) or endless chains in the bromide (Y$_{1/2}$[Sb$_2$O$_4$]Br$_{1/2}$ for Z = 8) by common vertices. With distances of 307 pm in YSb$_2$O$_4$Cl and 326 pm in YSb$_2$O$_4$Br there are no even substantial bonding Sb$^{5+}$…X$^{-}$ (X = Cl and Br) interactions at work. Luminescence spectroscopy on samples doped with trivalent europium and terbium showed an energy transfer from the oxidoantimonate(III) moieties as the sensitizer in the host structure onto the lanthanoid activators.

Introduction

In recent years, great attention has been paid to the structural diversity of rare-earth metal(III) oxidoarsenate(III) halides owing to the beneficial inorganic lone-pair antenna at the As$^{3+}$ cations for luminescence applications.1 Several representatives are known with the formula RE$_3$X$_2$[As$_2$O$_5$]$_4$ (RE = La–Lu, X = F–Br), but all their different crystal structures have the motif of isolated $\psi^1$-tetrahedral [AsO$_3$]$^{3-}$ anions in common. The fluoride derivatives (RE = Y, Ho, Tm–Lu)$^{1,3}$ crystallize in the tetragonal space group $P4_{1}/nnc$ with separated [AsO$_3$]$^{3-}$ units that form a lone-pair channel along [001], while the chloride derivatives crystallize monolithally (C2/c for La–Pr,$^{4,5}$ P2/c for Nd$^{5-7}$ and Sm$^{5-8}$) with a layered structure, in which the [AsO$_3$]$^{3-}$ units are linked to the chloride layers via weak secondary contacts. The three known bromide derivatives (RE = Pr, Sm, Eu)$^{5}$ crystallize again in the monoclinic system with space group P2/c and similar coordination features as the chloride derivatives. The motif of $\psi^1$-tetrahedral [AsO$_3$]$^{3-}$ is also present in the oxide-halide representatives RE$_3$O$\beta$Cl$_2$ and RE$_3$OBr$_2$[AsO$_3$], which crystallize tetragonally in the space groups $P4_{2}2$ or $P4_{2}2$ for (RE = Ce–Pr, Sm–Dy with X = Cl,$^{2,4,9}$ RE = Ce, Nd, Sm, Gd, Tb with X = Br$^2$). Like in the RE$_3$F$_2$[AsO$_3$]$_4$ cases (RE = Y, Ho, Tm–Lu), all RE$_3$O$_2$[AsO$_3$]$_2$ representatives have a lonepair tunnel structure of [AsO$_3$]$^{3-}$ units, but the rare-earth metal–oxygen linkage is different. Furthermore, there are RE$_3$O$_2$Cl$_2$ members (RE = Nd, Pr)$^{11,12}$ which crystallize monolithally in C2/m. Not only compounds with isolated [AsO$_3$]$^{3-}$ anions were synthesised, but also with additional oxidoantimonate(III) units of the pyroanionic species [As$_2$O$_5$]$^{3-}$ in the triclinic $R3\bar{m}$[As$_2$O$_5$][AsO$_3$] examples (RE = Sm–Gd with X = Cl,$^{4,13}$ RE = Y, Ho–Yb with X = Br$^{14}$). These also crystallize in a layered structure (space group: P1), in which both the [AsO$_3$]$^{3-}$ and the [As$_2$O$_5$]$^{3-}$ anions are bound to the halide layers via weak secondary contacts.

The first rare-earth metal(III) oxidoarsenate(III) halide with the composition Nd$_{0.5}$Bi$_2.5$O$_4$Cl$^{14}$ was synthesised by Aurivillius. In this case, the rare-earth metal cation site is mixed with bismuth(III). Only ten years later, REBi$_2$O$_4$Cl phases (RE = Y, La, Nd)$^{15}$ were the first synthesised representatives without mixed...
occupation of the layers. Oppermann et al. extended the spectrum of these representatives at first with ErBi₂O₄Cl⁴⁸ and found all except the cerium representatives with the composition REBi₂O₄X (RE = La, Pr–Nd, Sm–Lu for X = Cl–I).¹⁶,¹⁷ All these representatives crystallize in the tetragonal space group P4/nmm and form layered structures, in which the rare-earth metal cations are surrounded cube-shaped by eight oxygen atoms [REO₈]¹³⁻. These cubes are linked to each other via common edges. The Bi³⁺ cations form square [BiO₄]²⁻ (ax = axial) with four oxygen, which are linked to each other via their corners to form layers as well.

The rare-earth metal(II) oxidoantimonate(III) halides have been neglected in previous research, except for La₃F₃[SB₂O₅]₄, which crystallizes analogously to the REBi₂O₄X family in the tetragonal space group P4/nmm. Furthermore, the analogous bromide derivative Sm₁.₅Sb₁.₅O₄Br was discovered, in which there is also a mixed occupation of the antimony position of the Sb³⁺ with Sm³⁺ cations just like in Sm₁.₃Sb₁.₇O₄Cl. In further studies, the derivatives of the other bromides with RE = Eu–Dy were discovered. They crystallize in the monoclinic space group P2₁/c, but with a different linkage of the antimony–oxygen polyhedra. Here ψ⁴⁻ tetrahedral [SbO₃]₁⁻ units with only three oxygen atoms are present, which are linked to each other via two corners to form edge-sharing, t = terminal), not showing any mixed occupation with RE³⁺ cations. Moreover, the luminescence of trivalent europium and terbium will be investigated and discussed. The oxidoantimonate(III) host structure promises to provide an energy transfer to enhance the luminescence of the 4f–4f activators. Trivalent antimony cations themselves show an efficient 5s–5p excitation that can be used as an antenna for lanthanoid activators⁵⁸–⁶⁸ and antimony(III) compounds have proven to transfer energy previously.⁵⁷,⁶⁸

Result and discussion

The two rare-earth metal(II) oxidoantimonate(III) halides YSb₂O₄Cl and YSb₂O₄Br were formed from Y₂O₃ and Sb₂O₃ together with YX₃ (X = Cl and Br) as colourless square platelets. YSb₂O₄Cl crystallizes in the tetragonal space group P42/2, while YSb₂O₄Br adopts the monoclinic space group P2₁/c, just like the already known RESb₂O₄Br representatives with RE = Eu–Dy.²³–²⁴ The unit-cell parameters for YSb₂O₄Cl are a = 773.56(4) pm, c = 878.91(6) pm (c/a = 1.136), while a = 896.54(6) pm, b = 780.23(5) pm, c = 779.61(5) pm and β = 91.398(3)° apply for YSb₂O₄Br. The molar volumes of the bromides decrease from EuSb₂O₄Br (Vₚ = 84.36 cm³ mol⁻¹) to DySb₂O₄Br (Vₚ = 82.57 cm³ mol⁻¹) as consequence of the lanthanoid contraction.²⁹ Despite being half as heavy the Y³⁺ cation can be classified by its ionic radius between Dy³⁺ and Ho³⁺, which also holds here, indicated with the molar volume of 82.08 cm³ mol⁻¹ for YSb₂O₄Br. The molar volume of YSb₂O₄Cl is with 79.19 cm³ mol⁻¹ considerably smaller as compared to the bromide derivative, due to the lighter and smaller halide anion.

While the monoclinic YSb₂O₄Br shows two crystallographically distinct Y³⁺ positions, the tetragonal YSb₂O₄Cl only comprises one Y³⁺ position. In both cases, however, the Y³⁺ cations are surrounded by eight oxygen atoms that arrange themselves to square hemiprisms [YO₄]¹³⁻. The [YO₄]¹³⁻ polyhedra are linked with four of their edges via the oxygen atoms to form two-dimensional infinite layers according to the Niggli formula ²⁻∞\{ [YO e 8/2]¹⁻ \} (e = edge-sharing, Fig. 1). These layers run parallel to the (001) plane in YSb₂O₄Cl and to the (100) plane in YSb₂O₄Br. The distances between yttrium and the oxygen atoms range between 227 and 253 pm in YSb₂O₄Cl or

Fig. 1 Two-dimensional infinite layers ²⁻∞\{ [YO e 8/2]¹⁻ \} of edge-linked square hemiprisms [YO₄]¹³⁻ in the tetragonal crystal structure of YSb₂O₄Cl (top) and in the monoclinic crystal structure of YSb₂O₄Br (bottom).
achieve \([\text{SbO}_2]\). The bridging oxygen atoms show distances of 203 pm to the antimony(III) cations and are thus significantly longer than the terminal antimony–oxygen distances of 193–195 pm. Moreover, the terminal O1 atoms of (Sb1)\(^{3+}\) exhibit distances to the next (Sb2)\(^{3+}\) cation within the chain of \(d(\text{Sb2} \cdots \text{O1}) = 317\) pm (Fig. 2, red), which is approximately the same as that of the terminal O2 atom of (Sb2)\(^{3+}\) to the next (Sb1)\(^{3+}\) cation, \(d(\text{Sb1} \cdots \text{O2}) = 316\) pm, between the chains (Fig. 2, yellow). These meandering chains propagate along [001] and lie parallel within the (100) plane.

The motif of chains occurs more frequently in crystal structures of ternary or quarternary antimony(III)–oxygen compounds. However, Sb\(^{3+}\) has than a coordination number of four and forms square \(\psi^1\)-pyramids \([\text{SbO}_4]^{2-}\) edge-linked according to \(1 \propto \left\{ \begin{array}{c} \text{SbO} \\ \text{2/2} \\ \text{O} \\ \text{1/1} \end{array} \right\} \). Examples of representatives for this behaviour are \(\text{ASbO}_2\) (\(A = \text{K–Cs}\)),\(^{32-33}\) \(\text{BaSb}_2\text{O}_4\text{Cl}\),\(^{34}\) \(\text{PbSb}_2\text{O}_4\text{Cl}\)\(^{35}\) and \(\text{ZnSb}_2\text{O}_4\).\(^{36}\) Oxygen antimony chains

\[
\frac{1}{\propto} \left\{ \begin{array}{c} \text{SbO} \\ \text{2/2} \\ \text{O} \\ \text{1/1} \end{array} \right\}
\]

are present with their full \(C_{\infty v}\) symmetry. The structural motif of separated \(0 \propto \left\{ [\text{SbO}_4]^{2-}\right\}\) rings is also not novel, but found in valentinite \((\beta-\text{Sb}_2\text{O}_3)\)\(^{37}\). Here they are further connected, not isolated, and show a twisted configuration. The Sb\(^{3+}\)--O\(^{2-}\) bond lengths in both compounds correspond well with typical antimony–oxygen distances in both crystalline forms of \(\text{Sb}_2\text{O}_3\) or

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![Fig. 2](image1.png)

**Fig. 2** One-dimensional infinite chains \(1 \propto \left\{ \begin{array}{c} \text{SbO} \\ \text{2/2} \\ \text{O} \\ \text{1/1} \end{array} \right\}\) of corner-linked \(\psi^1\)-tetrahedra \([\text{SbO}_3]^{3-}\) in the monoclinic crystal structure of \(\text{YSb}_2\text{O}_4\text{Br}\), which run parallel to the [001] direction. The contacts of the terminal oxygen atoms to the next, not directly bonded Sb\(^{3+}\) cations are shown in red within the chains and in yellow between the chains.

![Fig. 3](image2.png)

**Fig. 3** Isolated rings \([\text{SbO}_4]^{3-}\) of four cyclically vertex-linked \(\psi^1\)-tetrahedra \([\text{SbO}_3]^{3-}\) in the tetragonal crystal structure of \(\text{YSb}_2\text{O}_4\text{Cl}\) according to \(1 \propto \left\{ \begin{array}{c} \text{SbO} \\ \text{e} \\ \text{O} \\ \text{e} \\ \text{2/2} \\ \text{e} \end{array} \right\}\). The contacts of the terminal oxygen atoms to the next, not directly bonded Sb\(^{3+}\) cations are shown in yellow.
senarmontite: $d$(Sb–O) = 198 pm, $\beta$: valentinite: $d$(Sb–O) = 198–202 pm$^{39}$.

In YSb$_2$O$_4$Br there is only one crystallographic position for the halide anion, whereas in YSb$_2$O$_4$Cl two different ones of them are present. The halide anions show a minimum distance of $d$(Sb/Cl) = 307 pm to the nearest Sb$^{3+}$ cation in YSb$_2$O$_4$Cl and of $d$(Sb/Br) = 326 pm in YSb$_2$O$_4$Br. Their distances to the nearest Y$^{3+}$ cation amount to $d$(Y/Cl) = 420 pm for YSb$_2$O$_4$Cl and $d$(Y/Br) = 427 pm for YSb$_2$O$_4$Br. So at these distances, one can not speak of real coordination in either structure. Between each layer of Sb$^{3+}$ cations there is a layer of halide anions, which in the case of YSb$_2$O$_4$Br spreads out parallel to the (100) plane, but parallel to the (001) plane in the case of YSb$_2$O$_4$Cl. This halide layer has no contact or connection to any other layer, neither via X⋯⋯Sb$^{3+}$ nor via X⋯⋯Y$^{3+}$ bonds. However, the layer of Sb$^{3+}$ cations enjoys linkage to the layer of Y$^{3+}$ cations via all oxygen atoms according to $\frac{2}{\infty}$ {YSb$_2$O$_4^{3+}$} in both yttrium(III) oxidoantimonate(III) halides YSb$_2$O$_4$X (X = Cl and Br).

Fig. 4 shows an extended unit cell of YSb$_2$O$_4$Br with depicted coordination spheres of the Y$^{3+}$ and Sb$^{3+}$ cations. The same applies to Fig. 5, which shows the extended unit cell of YSb$_2$O$_4$Cl.

Since the yttrium cations are surrounded by oxidoantimonate layers, an energy transfer from these layers towards any cation doped on the yttrium site could be expected. This was verified via luminescence spectroscopy (Fig. 6 and 7), but apparently, the luminescence was quite different for all samples (Fig. 8).

Fig. 4 Extended tetragonal unit cell of YSb$_2$O$_4$Cl as viewed along [010].

Fig. 5 Extended monoclinic unit cell of YSb$_2$O$_4$Br as viewed along [001].

Fig. 6 Fluorescence spectra of YSb$_2$O$_4$Cl doped with either Eu$^{3+}$ (left) or Tb$^{3+}$ (right). $\lambda_{exc}$ describes the wavelength used to record the excitation spectrum, whereas $\lambda_{em}$ represents the wavelength, at which the emission spectrum was recorded.

Fig. 7 Luminescence spectra of YSb$_2$O$_4$Br doped with either Eu$^{3+}$ (left) or Tb$^{3+}$ (right). $\lambda_{exc}$ describes the wavelength used to record the excitation spectrum, whereas $\lambda_{em}$ represents the wavelength, at which the emission spectrum was recorded.

Fig. 8 Comparison of the visible luminescence of the four samples YSb$_2$O$_4$Cl:Eu$^{3+}$ (a), YSb$_2$O$_4$Cl:Tb$^{3+}$ (b), YSb$_2$O$_4$Br:Eu$^{3+}$ (c), and YSb$_2$O$_4$Br:Tb$^{3+}$ (d).
YSb2O4Cl:Eu3+ shows weak, orange-red luminescence. In the spectrum, the weak emission is represented by the low signal-to-noise ratio. The excitation spectrum is dominated by the broad charge-transfer within the oxidoantimonate host structure peaking at 297 nm. Another band at 467 nm can be attributed to 4f–4f transitions of Eu3+. The emission spectrum features the main emission bands typical for trivalent europium. The band peaking at 612 nm, attributed to the emission \( ^{5}D_{0} \rightarrow ^{7}F_{2} \), is much more intense than that for \( ^{5}D_{0} \rightarrow ^{7}F_{1} \), normally located around 595 nm. This supports the experimentally obtained site symmetry of yttrium, as the comparably strong hypersensitive transition is a very good probe for the absence of a local inversion center.

The Tb3+-doped sample of YSb2O4Cl shows two different emissions (green and blue) that mix to give a turquoise colour impression. The broad emission in the blue regime with the maximum around 485 nm can be attributed to the host structure \(^{5}P_{1,2} \rightarrow ^{1}S_{0}\) transition of the Sb3+ lone-pair cation), which has been observed for LaOBr:Sb3+ (510 nm) and GdSb2O4Br (455 nm) as well and even matches with pure antimony(m) chlorides such as CsNaSbCl6 upon excitation between 255 to 280 nm. Three sharp bands assigned to the 4f–4f transitions \( ^{5}D_{1} \rightarrow ^{7}F_{j} \) \((j = 3, 4, 5)\) were also recorded. The excitation spectrum features two 4f–4f transitions with their respective maxima at 374 and 483 nm. The charge-transfer transition of the host structure is blue-shifted compared to the Eu3+-doped sample and peaks at 294 nm. In both spectra apparently an energy transfer between the host structure and the lanthanoid activator happens upon excitation to enhance the luminescence, but in the Tb3+-doped compound this transfer is obviously incomplete causing a characteristic turquoise emission colour.

The oxidoantimonate bromides show a similar luminescence, when doped with trivalent europium or terbium, but significantly more intense (“heavy-atom effect”).\(^{1}\) YSb2O4Br:Eu3+ exhibits an excitation spectrum, in which the charge-transfer band of the oxidoantimonate host structure is even more dominating compared to any 4f–4f transition of Eu3+.

It is blue-shifted about 50 nm compared to the chloride. While the same bands were observed as in the oxidoantimonate chloride, their relative intensities are decidedly stronger.

The excitation band around 393 nm, normally the most prominent one, can be only seen as a slight shoulder. In the emission spectrum, the band attributed to the hypersensitive transition \( ^{5}D_{0} \rightarrow ^{7}F_{2} \) is once again noticeably more intense than that for \( ^{5}D_{0} \rightarrow ^{7}F_{1} \) transition, since the yttrium cation occupies a site without inversion symmetry.

The emission spectrum of YSb2O4Br:Tb3+ consists of a very broad band, which we assign to the charge-transfer transition of the host structure. The sharp band of the \( ^{5}D_{4} \rightarrow ^{7}F_{6} \) transition with a maximum around 540 nm is typically the most intense band in Tb3+ spectra, the other bands are not observed, due to restrictions on the recorded wavelength regime. Like in the case above, the excitation is blue-shifted and therefore not in the accessible region of the spectrum. Interestingly, the spectrum does not feature either the 4f–5d excitation or any of the typical 4f–4f transitions of Tb3+. This indicates that the Tb3+ cations are almost exclusively excited via the energy transfer from the oxidoantimonate(m) system \( ^{7}S_{0} \rightarrow ^{5}P_{1,2} \) transition of the Sb3+ lone-pair cation and/or \( O^{2-} \rightarrow Sb^{3+} \) charge-transfer excitation).

Table 1 lists the most important crystallographic data for YSb2O4Br and YSb2O4Cl, while Table 2 gives the atomic coordinates, Wyckoff positions and equivalent isotropic displacement parameters. Table 3 contains selected bond lengths and interatomic distances for YSb2O4Cl and YSb2O4Br.

### Experimental section

**Synthesis of YSb2O4Cl and YSb2O4Br**

The yttrium(m) oxoantimonate(m) halides YSb2O4X (X = Cl and Br) were synthesised at elevated temperatures via solid-state reactions in evacuated silica ampoules (Quarz- und Glasbläserei Müller, Berlin-Adlershof; inner diameter: 10 mm, wall thickness: 1 mm, length: 40 mm). Yttrium oxide (Y2O3, ChemPur: 99.9%, 37.68 mg) and yttrium bromide (YBr3, Aldrich: 99.9%, 57.49 mg) were used as reactants according to eqn (1). Eutectic mixtures of...
sodium chloride (NaCl, Merck: 99.9%, 126 mg) and cesium chloride (CsCl, Aldrich: 99.9%, 674 mg) were used for YSb2O4Cl and those of sodium bromide (NaBr, Merck: 99.9%, 203 mg) and cesium bromide (CsBr, ChemPur: 99.9%, 597 mg) for YSb2O4Br to improve the reaction speed and the crystal growth as fluxing agents. As doping material europium sesquioxide (Eu2O3, ChemPur: 99.9%, 1.7 mg) or terbium chloride (TbCl3, Aldrich: 99.9%, 1.0 mg) were used for YSb2O4Cl and europium sesquioxide (Eu2O3, ChemPur: 99.9%, 1.5 mg) or terbium bromide (TbBr3, Aldrich: 99.9%, 1.4 mg) for YSb2O4Br.

The reactants were weighed into glassy silica ampoules under inert gas (argon) inside a glove box (Glovebox Systemtechnik, GS Mega E-line), sealed under dynamic vacuum and then subjected to a defined temperature program in a muffle furnace (Nabertherm, L 9/12). This was heated at a rate of 150 K h^{-1} to 750 °C, held there for two days, cooled with 5 K h^{-1} to 666 °C, held for another three days, cooled with 5 K h^{-1} to 530 °C, again held for two days, then cooled with 10 K h^{-1} to 480 °C and finally quenched to room temperature by cutting off the power to the closed furnace. The recovered product samples were washed with 500 ml demineralised water and then dried for 2 h in a drying oven at 120 °C. Under a stereomicroscope, colourless flat, square platelets were visible, clearly larger for YSb2O4Cl than for YSb2O4Br.

### Table 1

| Distance | YSb2O4Cl | Distance | YSb2O4Br |
|----------|----------|----------|----------|
| Y1−O1    | (4)      | Y−O2     | (1)      |
| Y1−O2    | (2)      | Y−O1     | (1)      |
| Y2−O1    | (2)      | Y−O′1    | (1)      |
| Y2−O2    | (2)      | Y−O2     | (1)      |
| Y1−Sb    | (2)      | Y−O4     | (1)      |
| Y2−Sb    | (2)      | Y−O2     | (1)      |
| Sb−O1    | (1)      | Y−O′1    | (1)      |
| Sb−O2    | (1)      | Y−O3     | (1)      |
| Sb−O′1   | (1)      | Y−Sb2    | (1)      |
| C1⋯Y1    | (2)      | Y−Sb1    | (1)      |
| C1⋯Y2    | (2)      | Y−Sb′1   | (1)      |
| C2⋯Y1    | (2)      | Y−Sb′2   | (1)      |
| C1⋯Sb    | (4)      | Sb1−O1   | (3)      |
| C1⋯Sb′   | (4)      | Sb1−O4   | (3)      |
| C2⋯Sb′   | (4)      | Sb2−O4   | (3)      |
| Br⋯Y     | (1)      | Sb2−O3   | (1)      |
| Br⋯Y′    | (1)      | Sb2−O1   | (1)      |
| Br⋯Sb1   | (1)      | Sb2−O3   | (1)      |

### Table 2

| Atom | Wyckoff site | x/a | y/b | z/c | Ueq/pm² |
|------|--------------|-----|-----|-----|---------|
| Y1   | 2a           | 0   | 0   | 0   | 120(6)  |
| Y2   | 2c           | 0   | 1/2 | 0   | 118(6)  |
| Sb   | 8g           | 0.24016(8) | 1/2 | 0.0153(4) | 203(4) |
| O1   | 8g           | 0.0629(9)  | 2437(9) | 0.1351(11) | 165(17) |
| O2   | 8g           | 0.4648(9)  | 2483(9) | 0.1705(11) | 147(17) |
| Cl1  | 2b           | 0   | 0   | 1/2 | 265(19) |
| Cl2  | 2c           | 0   | 1/2 | 0   | 250(18) |

### Table 3

| Distance | YSb2O4Cl | Distance | YSb2O4Br |
|----------|----------|----------|----------|
| Y1−O1    | (4)      | Y−O2     | (1)      |
| Y1−O2    | (2)      | Y−O1     | (1)      |
| Y2−O1    | (2)      | Y−O′1    | (1)      |
| Y2−O2    | (2)      | Y−O2     | (1)      |
| Y1−Sb    | (2)      | Y−O4     | (1)      |
| Y2−Sb    | (2)      | Y−O2     | (1)      |
| Sb−O1    | (1)      | Y−O′1    | (1)      |
| Sb−O2    | (1)      | Y−O3     | (1)      |
| Sb−O′1   | (1)      | Y−Sb2    | (1)      |
| C1⋯Y1    | (2)      | Y−Sb1    | (1)      |
| C1⋯Y2    | (2)      | Y−Sb′1   | (1)      |
| C2⋯Y1    | (2)      | Y−Sb′2   | (1)      |
| C1⋯Sb    | (4)      | Sb1−O1   | (3)      |
| C1⋯Sb′   | (4)      | Sb1−O4   | (3)      |
| C2⋯Sb′   | (4)      | Sb2−O4   | (3)      |
| Br⋯Y     | (1)      | Sb2−O3   | (1)      |
| Br⋯Y′    | (1)      | Sb2−O1   | (1)      |
| Br⋯Sb1   | (1)      | Sb2−O3   | (1)      |
| Br⋯Sb1′  | (1)      | Sb2−O1   | (1)      |
| Br⋯Sb1″  | (1)      | Sb2−O3   | (1)      |
| Br⋯Sb1‴  | (1)      | Sb2−O1   | (1)      |
| Br⋯Sb2″  | (1)      | Sb2−O3   | (1)      |
| Br⋯Sb2‴  | (1)      | Sb2−O1   | (1)      |

The luminescence spectra of all samples were measured using a Horiba Fluoromax-4 spectrometer scanning from 220 to 800 nm at room temperature. Therefore, finely ground powder samples were filled into the sample holder and subsequently placed in the sample chamber. These measurements were conducted and evaluated with the program FluorEssence. All excitation spectra were corrected to consider the xenon-lamp spectrum.

### Conclusions

With YSb2O4Br another representative of the known series RESb2O4Br (RE = Eu−Dy) could be synthesised and structurally characterised. Thus it also shows the structural motif of \( \psi \)-tetrahedral [SbO3]3⁻ groups linked to a meandering chain of the formula

\[
\begin{align*}
\text{SbO}_3 & \quad 2/2 \\
\text{O}_3 & \quad 1/1
\end{align*}
\]

via common vertices. As first tetragonal representative of the rare-earth metal(III)
obtained. Although its structure shows many similarities to the monoclinic YSb₂O₅Br (space group: P2₁/c) representative, it exhibits a different corner-linkage of the \( \phi^{3}\)-tetrahedral \( \text{[SbO}_3]^{2−} \) units resulting in closed \( \infty \{ \text{[SbO}_3]^{4+} \} \) rings.

The luminescence spectra of samples doped with trivalent europium or terbium confirmed the lack of inversion symmetry around the yttrium cations in both structures, as well as an efficient energy transfer between the oxidoantimonate(III) layers and the lanthanoid(III)-activator cations.

Author contributions

The manuscript was written through contributions of all authors.

Conflicts of interest

There are no conflicts to declare.

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