A Novel Mixed-Valence Selenium(IV)/Selenium(VI) Oxo Compound: Crystal Structure Determination and X-ray Absorption Near Edge Structure Study of Erbium Selenite(IV) Selenate(VI) Hydrate, $Er(SeO_3)(SeO_4)_{1/2}\cdot H_2O$

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Erbium selenite(IV) selenate(VI) hydrate, $Er(SeO_3)(SeO_4)_{1/2}\cdot H_2O$, was prepared hydrothermally and the crystal structure determined using single-crystal X-ray diffraction. The monoclinic crystal structure (space group $C2$, a = 11.935 (2) Å, b = 6.881 (1) Å, c = 6.473 (1) Å, β = 103.79 (4)°, Z = 4) was solved by direct methods and the model refined to R = 2.36% and R_w = 2.69%. The structure contains both Se^{IV} in the form of selenite (SeO_3^{2-}) trigonal pyramids and Se^{VI} as selenate (SeO_4^{2-}) tetrahedra. The erbium is eight-coordinated. The structure can be described as a pseudolayered structure, the layers containing all the erbium, oxygen, and selenium(IV) atoms and being interconnected by selenium(VI) atoms which are disordered over two crystallographic sites. The X-ray absorption near edge structure (XANES) spectrum shows two maxima in the white-line feature at the correct energies for the presence of both Se^{IV} and the Se^{VI}.

Introduction

The oxo chemistry of selenium is confined to two oxidation states, Se(IV) and Se(VI), and compounds of both selenite, SeO_3^{2-}, and selenate, SeO_4^{2-}, have been known for well over 100 years.[1,2] Although the literature and applications of the rare earth selenites and selenates are not nearly as extensive as those of their sulfur-containing counterparts, they have been studied as potential precursors in the synthesis of rare earth phosphors[3] and as ion exchangers.[4,5] Due to the fact that the sulfate and selenate oxyanions have the same tetrahedral spatial configuration (the selenate anion being approximately 10% larger), their corresponding rare earth compounds are isostructural with only a few exceptions; for example, the series of octahydrates $R_2(SeO_4)_3\cdot 8H_2O$ (R = Pr–Lu, Y) is isostructural with the corresponding sulfate series and the two sets of compounds are made in a similar fashion.[6,7] The pentahydrate series, which have also been characterized by X-ray diffraction methods, show only small differences in structure, resulting in a change of space group, from the corresponding sulfates.[8,9]

The crystal chemistry of compounds containing the selenite ion are characterized by the stereochemically active lone pair of electrons on the selenium atom. The SeO_3^{2-} ion itself has C_{3v} symmetry with the selenium atom at the apex of a trigonal pyramid. There have been several recent investigations into the crystal structures of rare earth selenites, and a number of compounds with varied and interesting structures have resulted. These have included layered structures, such as $LaH(SeO_3)_2$,[10] $PrH_3(SeO_3)_2(Se_2O_5)$,[11] and the two series $RH(SeO_3)_2\cdot 2^1/_2H_2O$ and $RH(SeO_3)_2\cdot 2H_2O$ (R = Pr–Lu, Y),[12,13] and three-dimensional structures such as $NaY(SeO_3)_2$, $NaLa(SeO_3)_2$,[14] and $Na_{21}Y_{12}(SeO_3)_{28}(OH)$, which contains hydroxyl groups in small cavities created by the sodium–yttrium–selenite framework.[15] In contrast to those of the rare earth selenates, the structures of the rare earth selenites show little or no relationships to the structures of their sulfite counterparts. This and other differences, such as their differing thermal decomposition mechanisms, may be due to the different relative stabilities of the tetra- and hexavalent states[16] and the lower stability of the selenium–selenium bond compared to the sulfur–sulfur bond. The latter factor explains the marked difference between the oxygen-bridged diselenite anion and the nonsymmetrical, sulfur–sulfur-bonded disulfite anion.[17] The structures of the rare earth selenites seem to have a much closer relationship with those of the corresponding hydrogen phosphites, an example being the isostructural nature of the two dihydrate series $RH(SeO_3)_2\cdot 2H_2O$ and $RH(HPO_3)_2\cdot 2H_2O$ (R = Pr–Lu, Y).[18] Up to the present day, there have been two reported examples of compounds containing both Se(IV) and Se(VI) oxyanions in the same crystal structure, a lithium copper selenite/selenate, $Li_2Cu_3(SeO_3)_2(SeO_4)_2$,[19] and the rare mineral schmiederite, $Pb_2Cu_2(SeO_3)(SeO_4)_2$.[20] A compound of this kind has also been postulated in the decomposition of a hydrated lanthanum selenate.[21]

X-ray absorption spectroscopy using synchrotron radiation sources has been rapidly growing and it is now a very important technique for investigating the local structure and symmetry of materials and molecules by analysis of the extended X-ray absorption fine structure (EXAFS) and the X-ray absorption

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Table I. Crystallographic Data for $Er(SeO_3)(SeO_4)_{1/2}{\cdot}H_2O$

mol formula	$Er(SeO_3)(SeO_4)_{1/2}{\cdot}H_2O$	fw	381.5
a	11.935 (2) Å	space group	$C2$ (No. 5)
b	6.881 (1) Å	T	25 °C
c	6.473 (1) Å	λ	0.710 69 Å
β	103.79 (4)°	ρ_c	4.907 g cm^{-3}
V	516.3 (2) $Å^3$	μ	268.77 cm^{-1}
$F(000)$	676	$R(F)^a$	2.36%
Z	4	$R_w(F)^b$	2.69%

[a] $R(F) = \sum_h||F_o| - |F_c||/\sum_h|F_o|$. [b] $R_w(F) = [\sum_h w_h(|F_o - F_c|)^2/\sum_h w_h|F_o|^2]^{1/2}$.

near edge structure (XANES). One of the interesting features of XANES spectra is that the oxidation state of an element is reflected in the position of the absorption edges for that element.[22–26] Chemical shifts in the position of an X-ray absorption edge result from the changes in the screening of the core hole by the outer electrons and in the electronic structure of the valence electrons. As the oxidation state increases, the screening of the core levels decreases, driving the absorption threshold to higher energies.[27] A chemical shift in the same direction is often produced as the bonding becomes more covalent; this is a result of the energy gap between the filled "bonding" and empty "antibonding" orbitals increasing. As the absolute position of the X-ray absorption threshold is affected by a combination of these two effects, it can be used as an indicator of oxidation state only when compared to the positions of other absorption thresholds observed in spectra of similar compounds. XANES has been used in this way on a number of different occasions to reconcile valency ambiguity in transition metal compounds, one interesting example being V_2TiO_5, where one of the sites in the structure contains solely V^{III} while the other site contains both Ti and V. Analysis of the metal–oxygen distances in the structure could not distinguish whether this site was V^{III} and Ti^{IV} or V^{IV} and Ti^{III}. XANES spectroscopy allowed assignment of the species to be made as V^{III} and Ti^{IV}.[28] There have been fewer instances where XANES spectroscopy has been applied to compounds containing post transition elements, although examples include that of $Ga^{I}Ga^{III}Cl_4$.[29]

This paper describes the synthesis, crystal structure determination, XANES spectrum, and infrared spectrum of a new mixed-valence selenium compound, $Er(SeO_3)(SeO_4)_{1/2}{\cdot}H_2O$.

Experimental Section

Synthesis. The title compound was synthesized using a hydrothermal method. A mixture of $Er(NO_3)_3{\cdot}5H_2O$ (1 g) and SeO_2 (1 g) in 15 mL of water was heated to 473 K in a Teflon-lined steel autoclave for a period of 48 h. The solution was then cooled slowly, and a small amount of pale pink powder containing some small single crystals (maximum dimensions 0.15 × 0.15 × 0.1 mm) was recovered by suction filtration.

Crystal Structure Determination. Intensity data were collected on a small single crystal (crystal dimensions 0.1 × 0.1 × 0.075 mm) using an Enraf-Nonius CAD-4F diffractometer operating with graphite-monochromated Mo Kα radiation ($\lambda = 0.710$ 69 Å) at room temperature. Unit cell dimensions were obtained by least-squares refinement of the 2θ values for 25 centered reflections with $2\theta > 22°$. The crystallographic data for $Er(SeO_3)(SeO_4)_{1/2}{\cdot}H_2O$ are summarized in Table I. A total of 876 observed unique intensities ($I > 3\sigma(I)$, $1 \leq 2\theta \leq 60°$) were corrected for Lorentz and polarization effects, and azimuthal scans were taken on three selected reflections so that a semiempirical absorption correction could be applied (minimum correction 1.56, maximum correction 3.24). The structure was solved using the direct-methods program SHELXS-86[30] and difference Fourier techniques. Final full-matrix least-squares refinement (83 parameters) included anisotropic thermal factors for all atoms, but with the thermal displacement parameters for Se(2) and Se(3) restrained to be equal. The hydrogen atoms in the structure could not be located in the final difference Fourier map. The final agreement factors are given in Table I. All crystallographic computations were done using the Oxford package CRYSTALS[31] on a Dec Microvax 3600 computer. Complex neutral-atom scattering factors were taken from ref 32.

Table II. Atomic Positions, Isotropic Thermal Parameters, and Occupancies (with ESD's) for $Er(SeO_3)(SeO_4)_{1/2}{\cdot}H_2O$

atom	x	y	z	U_{equ},[a] $Å^2$	occ
Er(1)	0.19929 (2)	0.961 (1)	0.85524 (4)	0.0073	
Se(1)	0.28605 (6)	0.958 (1)	0.3651 (1)	0.0102	
Se(2)	0.5	0.126 (1)	0.0	0.0067	0.26 (1)
Se(3)	0.5	0.804 (1)	0.0	0.0055	0.25 (1)
O(1)	0.1820 (5)	0.983 (3)	0.4996 (8)	0.0080	
O(2)	0.262 (2)	0.535 (2)	0.820 (3)	0.0104	
O(3)	0.256 (2)	0.277 (3)	0.816 (4)	0.0154	
O(4)	0.3963 (5)	0.970 (4)	0.882 (1)	0.0106	
O(5)	0.038 (1)	0.161 (2)	0.804 (2)	0.0215	
O(6)	0.020 (2)	0.767 (3)	0.788 (3)	0.0597	

[a] $U_{equ} = (U_1U_2U_3)^{1/3}$.

Table III. Selected Bond Distances (Å) for $Er(SeO_3)(SeO_4)_{1/2}{\cdot}H_2O$

Er(1)–O(1)	2.267 (5)	Se(1)–O(1)	1.686 (5)
Er(1)–O(2)	2.39 (1)	Se(1)–O(2)	1.71 (1)
Er(1)–O(2)	2.36 (1)	Se(1)–O(3)	1.70 (2)
Er(1)–O(3)	2.31 (1)		
Er(1)–O(3)	2.43 (1)	Se(2)–O(4)	1.68 (1)
Er(1)–O(4)	2.319 (5)	Se(2)–O(6)	1.751 (9)
Er(1)–O(5)	2.37 (1)		
Er(1)–O(6)	2.38 (2)	Se(3)–O(4)	1.72 (2)
		Se(3)–O(5)	1.758 (8)

X-ray Absorption Spectroscopy. XANES measurements were made at the Science and Engineering Research Council Synchrotron Radiation Source (SRS), Daresbury, U.K., using station 9.2. A transmission sample was prepared by grinding a small amount of the title compound with boron nitride and placing it between two pieces of Sellotape. Small quantities of the reference compounds, $Na_2SeO_3{\cdot}5H_2O$ and $Na_2SeO_4{\cdot}10H_2O$, were prepared in a similar fashion. Spectra were recorded at room temperature using a Si(220) order sorting monochromator with 50% harmonic rejection over an energy range around the K absorption edge for selenium (12 475–12 896 eV; Se K edge is at 12 658 eV). A monochromator step size of 0.25 mdeg and a vertical beam height of 0.75 mm were employed. The monochromator band-pass was estimated to be approximately 2 eV; this is comparable to the expected core hole lifetime broadening. The energy scale was not calibrated, as Se foils were not available; however, the monochromator on this beam line has been demonstrated to show good repeatability, allowing a reliable comparison of the XANES spectra from the title compound and the reference materials.

Infrared Spectroscopy. The infrared spectrum of the title compound was collected on a Perkin-Elmer spectrometer in a pressed disk of dry cesium iodide.

Discussion

Final atomic parameters and selected bond distances and bond lengths for $Er(SeO_3)(SeO_4)_{1/2}{\cdot}H_2O$ are given in Tables II–IV. The structure contains isolated trigonal pyramidal selenite(IV) anions, with bond lengths and angles similar to those found in other compounds of this type (average Se–O distance 1.69 (1) Å, average O–Se–O angle 99.0 (5)°), and tetrahedral selenate(VI) groups (average Se–O distance 1.73 (1) Å, average O–Se–O angle 108.2 (9)°). However, taken by itself, the structure is not

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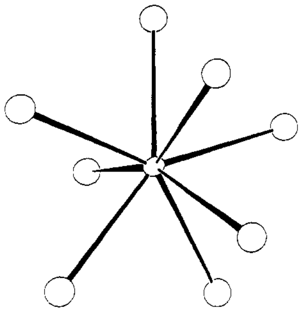

Figure 1. Distorted square antiprismatic coordination of oxygen around erbium in $Er(SeO_3)(SeO_4)_{1/2}\cdot H_2O$.

Table IV. Selected Bond Angles (deg) for $Er(SeO_3)(SeO_4)_{1/2}\cdot H_2O$

O(2)–Er(1)–O(1)	85.6 (3)	O(2)–Er(1)–O(1)	145.0 (3)
O(2)–Er(1)–O(2)	123.8 (3)	O(3)–Er(1)–O(1)	77.3 (4)
O(3)–Er(1)–O(2)	140.0 (2)	O(3)–Er(1)–O(2)	67.8 (3)
O(3)–Er(1)–O(1)	151.0 (4)	O(3)–Er(1)–O(2)	65.4 (3)
O(3)–Er(1)–O(2)	61.9 (2)	O(3)–Er(1)–O(3)	125.6 (3)
O(4)–Er(1)–O(1)	85.3 (2)	O(4)–Er(1)–O(2)	72.5 (6)
O(4)–Er(1)–O(2)	86.4 (5)	O(4)–Er(1)–O(3)	70.3 (6)
O(4)–Er(1)–O(3)	86.8 (5)	O(5)–Er(1)–O(1)	85.3 (3)
O(5)–Er(1)–O(2)	144.4 (5)	O(5)–Er(1)–O(2)	79.8 (5)
O(5)–Er(1)–O(3)	70.4 (5)	O(5)–Er(1)–O(3)	117.4 (4)
O(5)–Er(1)–O(4)	140.6 (6)	O(6)–Er(1)–O(1)	90.9 (4)
O(6)–Er(1)–O(2)	76.8 (5)	O(6)–Er(1)–O(2)	112.5 (4)
O(6)–Er(1)–O(3)	138.5 (5)	O(6)–Er(1)–O(3)	81.8 (5)
O(6)–Er(1)–O(4)	149.2 (6)	O(6)–Er(1)–O(5)	69.0 (3)
O(2)–Se(1)–O(1)	97.7 (6)	O(3)–Se(1)–O(1)	106.8 (6)
O(3)–Se(1)–O(2)	92.5 (3)		
O(4)–Se(2)–O(4)	100.0 (13)	O(6)–Se(2)–O(4)	106.5 (7)
O(6)–Se(2)–O(4)	119.2 (6)	O(6)–Se(2)–O(6)	106.1 (9)
O(4)–Se(3)–O(4)	97.1 (11)	O(5)–Se(3)–O(4)	113.6 (5)
O(5)–Se(3)–O(4)	106.9 (6)	O(5)–Se(3)–O(5)	117.0 (9)

sufficiently precise to confirm unambiguously the mixed-valence nature of the compound, which is disordered (see below). It is unrelated to either of the two previously known mixed selenite/selenate structures. The erbium atoms are eight-coordinated by oxygens in a distorted square antiprism (Figure 1); five belong to selenite(IV) groups [O(1), 2 × O(2), 2 × O(3)], and the other three either are part of selenate(VI) groups or are water molecules. The structure can be described as having a pseudolayered type arrangement. The layers, which consist of the erbium and selenite(IV) polyhedra, are parallel to the *bc* plane and are interlinked into a three-dimensional structure by the disordered selenate(VI) groups. This arrangement produces small channels running parallel to the *c* axis. There is a close relationship between this structure and the structures of the two isostructural compounds $ErH(SeO_3)_2\cdot 2H_2O$[12] and $ErH(HPO_3)_2\cdot 2H_2O$,[18] which both have the same erbium–selenite(IV)/phosphite layers in them but are interconnected via protonated selenite(IV) or hydrogen phosphite groups instead of by selenate(VI) units. The close similarity is shown best by the close agreement of the unit cell constants of the unit cell face which is parallel to the layers, 6.881 (2) and 6.473 (1) Å for the title compound and 6.900 (8) and 6.451 (9) Å for $ErH(SeO_3)_2\cdot 2H_2O$ (the latter cell constants were calculated by least-squares refinement of reflection positions measured by powder X-ray diffraction). A view of the structure showing the pseudolayered arrangement and the small channels is shown in Figure 2.

The selenium(VI) atoms between the layers are disordered between two sites in the structure; their crystallographic occupancies were refined, and values of 0.26 (1) for Se(2) and 0.25 (1) for Se(3) were recorded (these sites are thus approximately half-occupied, as the occupation factor quoted here includes the multiplicity of the atomic site). Regardless of which site is occupied, both of the disordered selenium atoms are connected to O(4), while the O(5) and O(6) sites will contain either a water molecule or a selenate(VI) oxygen. Unfortunately this disorder could not be resolved in the current experiment, but a clue to its existence is given by the rather larger thermal displacement parameters seen for O(5) and O(6). The structure of the title compound, showing the two possible sites which the selenium(VI) atoms can occupy, can be seen in Figure 3.

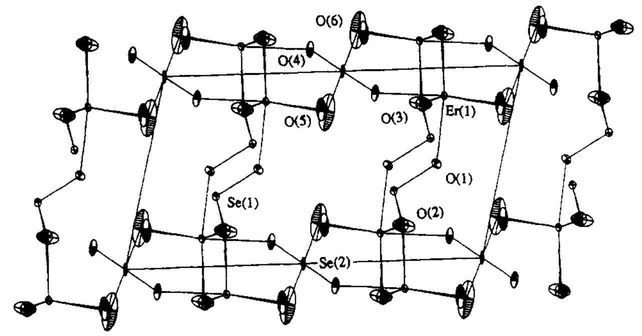

Figure 2. Structure of $Er(SeO_3)(SeO_4)_{1/2}\cdot H_2O$ viewed in the [010] direction.

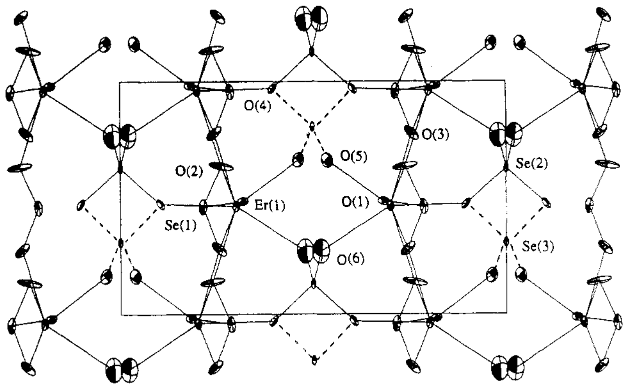

Figure 3. Structure of $Er(SeO_3)(SeO_4)_{1/2}\cdot H_2O$ viewed in the [001] direction.

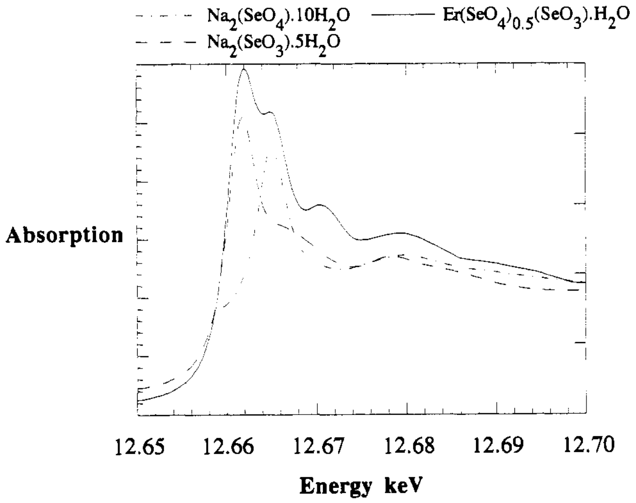

Figure 4. X-ray absorption near edge spectrum of $Er(SeO_3)(SeO_4)_{1/2}\cdot H_2O$.

The mixed-valence nature of the title compound is confirmed by the XANES spectrum (Figure 4). A splitting is observed in the white line, with two maxima occurring at 12662.0 and 12664.9 eV. The reference compounds $Na_2SeO_3\cdot 5H_2O$ and $Na_2SeO_4\cdot 10H_2O$ show single maxima in their white lines at energies of 12661.8 and 12665.0 eV, respectively, corresponding almost exactly to the energies of the maxima for the two features seen in the title compound and clearly identifying them as arising from the presence of both Se^{IV} and Se^{VI}. The splitting of 2.9 eV observed between the two oxidation states is very similar to those observed for Ga^I and Ga^{III} in $GaCl_2$ (3.3 eV) and in $GaCl_3$ and $GaAlCl_4$ (3.1 eV)[29] and also for As and As in As_2O_3 and As_2O_5 (4.3 eV).[33]

The infrared spectrum of the compound shows features in the 400–2000-cm^{-1} energy range which can be tentatively assigned to the normal modes given in Table V. The assignment of the selenite(IV) vibrational modes is done by comparison with the IR spectrum of $ErH(SeO_3)_2 \cdot 2H_2O$,[12] where the selenite group is in an extremely similar environment, and the assignment of the selenate(VI) modes is done by comparison with the IR spectra of other lanthanide selenates.[34]

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Table V. Band Maxima (cm^{-1}) and Assignments for $Er(SeO_3)(SeO_4)_{1/2} \cdot H_2O$

785	$\nu_1(SeO_3)$	1650	$\delta(H_2O)$
719	$\nu_3(SeO_3)$	1364	$\delta(OH)$
454	$\nu_2(SeO_3)$		
917	$\nu_3(SeO_4)$	517	$\nu(Er{-}OH_2)$ wag
899	$\nu_3(SeO_4)$	668	$\nu(Er{-}OH_2)$ rock
881	$\nu_3(SeO_4)$		
839	$\nu_1(SeO_4)$		
420	$\nu_4(SeO_4)$		

Supplementary Material Available: Tables giving crystal data and details of the structure solution and refinement and anisotropic thermal parameters (2 pages). Ordering information is given on any current masthead page.

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