SHORT STRUCTURAL PAPERS

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Yttrium Diselenite Nitrate Trihydrate

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Abstract. $YNO_3(Se_2O_5).3H_2O$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}216\ (1)$, $b = 7{\cdot}100\ (2)$, $c = 20{\cdot}689\ (6)$ Å, $V = 913{\cdot}0$ Å^3, $Z = 4$, $D_x = 3{\cdot}22$ Mg m^{-3}, μ(Mo $K\alpha$) = 14·36 mm^{-1}, $R = 5{\cdot}1\%$ for 1311 reflections. The polyhedron around Y is a square antiprism with four O atoms from diselenite groups, one O atom from the nitrate group and three O atoms from water. The Y polyhedra are linked together by diselenite groups.

Introduction. A novel series of isostructural rare-earth compounds with the composition $RENO_3(Se_2O_5).3H_2O$, where RE is trivalent Y, Pr, Nd or Sm–Lu, have been synthetized and characterized by chemical and thermal analyses, IR spectroscopy and X-ray powder diffraction (Niinistö, Valkonen & Ylinen, 1979). To elucidate the structures of these compounds a crystallographic study was initiated for the Y compound grown as single crystals by slow evaporation of the aqueous solution.

A computer-controlled Syntex $P2_1$ (Fortran version) four-circle diffractometer with graphite-monochromatized Mo $K\alpha$ radiation was used in the determination of the cell parameters and the collection of intensities. The crystal was 0·1 × 0·1 × 0·7 mm and the temperature was 298 K. Cell dimensions were calculated on the basis of 25 reflections in the range $3° < 2\theta < 21°$ and refined by least squares. Intensities were collected in the interval $5° < 2\theta < 60°$ with the $\theta/2\theta$ technique and a scan speed of 1·5° min^{-1}. Of the 1596 unique reflections measured, 1311 had $I > 3\sigma(I)$.

Semi-empirical absorption corrections were made on the basis of φ-scan data with six different 2θ values. The largest measured relative reduction in intensity was from 1·000 to 0·160. This absorption apparently had a great effect because R was 13·7% before the data were corrected and 5·1% afterwards.

The space group was determined from systematic absences in the original reflections. Lorentz and polarization corrections were applied.

The structure was solved with *MULTAN* 78 (Main, 1978). The sites of Y and two Se atoms were determined from 160 E values >1·54. These positions were then refined with XRAY 76 (Stewart, 1976) and the subsequent difference map gave the sites of the remaining non-hydrogen atoms. The scattering factors were those of Cromer & Mann (1968) for neutral atoms. After refinement with isotropic temperature factors R was 6·7% and after block-diagonal refinement with anisotropic temperature factors the final R was 5·1%.* The H-atom positions could not be determined from the difference map.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34549 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional parameters for* $YNO_3(Se_2O_5).3H_2O$

The e.s.d.'s of the last figures are given in parentheses.

	x	y	z
Y	0·76957 (10)	0·18083 (9)	0·86614 (3)
Se(1)	0·96888 (12)	0·08261 (9)	0·70305 (3)
Se(2)	0·52486 (11)	0·31899 (9)	0·71533 (3)
O(1)	0·1165 (10)	0·5141 (8)	0·7242 (2)
O(2)	0·0500 (8)	0·3902 (7)	0·8430 (3)
O(3)	0·2761 (11)	0·6898 (9)	0·8313 (3)
O(4)	0·3397 (9)	0·8769 (7)	0·7186 (2)
O(5)	0·5342 (10)	0·5162 (8)	0·6708 (3)
O(6)	0·9555 (10)	0·1536 (8)	0·0913 (3)
O(7)	0·4115 (9)	0·9682 (8)	0·4024 (3)
O(8)	0·3441 (10)	0·1613 (9)	0·0335 (2)
O(9)	0·4661 (13)	0·2094 (10)	0·5168 (3)
O(10)	0·1555 (11)	0·5172 (7)	0·0426 (3)
O(11)	0·2653 (13)	0·2419 (10)	0·6030 (3)
N	0·3658 (11)	0·3066 (10)	0·5531 (4)

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Table 2. *Interatomic distances* (Å) *around* Y

The e.s.d.'s of the last figures are given in parentheses.

Y–O(1)	2·324 (5)	Y–O(6)	2·442 (6)
Y–O(2)	2·341 (5)	Y–O(7)	2·369 (6)
Y–O(4)	2·340 (5)	Y–O(8)	2·404 (5)
Y–O(5)	2·349 (6)	Y–O(10)	2·459 (6)

Table 3. *Interatomic distances* (Å) *and angles* (°) *in the diselenite group*

The e.s.d.'s of the last figures are given in parentheses.

Se(1)–O(1)	1·668 (5)	O(1)–Se(1)–O(2)	104·7 (3)
Se(1)–O(2)	1·669 (5)	O(1)–Se(1)–O(3)	101·8 (3)
Se(1)–O(3)	1·844 (6)	O(2)–Se(1)–O(3)	93·4 (3)
Se(2)–O(3)	1·816 (6)	O(3)–Se(2)–O(4)	102·5 (3)
Se(2)–O(4)	1·658 (5)	O(3)–Se(2)–O(5)	96·1 (3)
Se(2)–O(5)	1·677 (5)	O(4)–Se(2)–O(5)	103·2 (3)
		Se(1)–O(3)–Se(2)	124·6 (3)

Table 4. *Interatomic distances* (Å) *and angles* (°) *in the nitrate group with e.s.d.'s and the distances* (Å) *of atoms from the least-squares plane in the nitrate group*

N–O(9)	1·196 (10)	O(9)–N–O(10)	120·8 (7)
N–O(10)	1·277 (9)	O(9)–N–O(11)	123·3 (7)
N–O(11)	1·291 (10)	O(10)–N–O(11)	115·8 (7)

	Distance		Distance
N	0·02 (1)	O(10)	−0·01 (1)
O(9)	−0·01 (1)	O(11)	−0·01 (1)

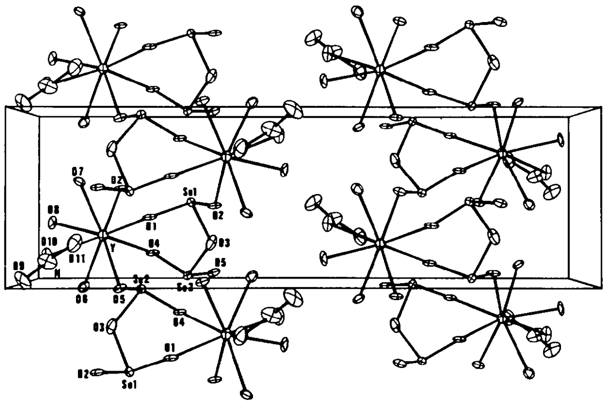

Fig. 1. The structure of $YNO_3(Se_2O_5).3H_2O$. **a** is vertical and **c** horizontal.

Table 5. *Distances* (Å) *of* O *atoms from least-squares planes in the square antiprism*

Plane 1	Distance	Plane 2	Distance
O(2)	0·04 (4)	O(1)	−0·09 (10)
O(4)	−0·04 (4)	O(5)	0·09 (10)
O(6)	0·04 (4)	O(7)	0·09 (10)
O(8)	−0·04 (4)	O(10)	−0·09 (10)

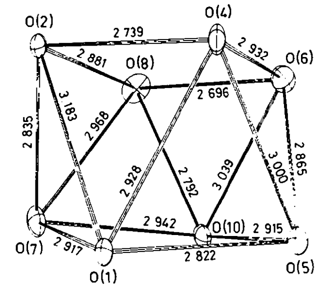

Fig. 2. O–O distances (Å) in the YO_8 polyhedron. E.s.d.'s are 0·008 Å in all distances.

Discussion. Positional parameters are listed in Table 1 and the interatomic distances in Tables 2, 3 and 4. The content of one unit cell is shown in Fig. 1.

Y–O distances, 2·324–2·459 Å, are in good agreement with distances found for eight coordination: 2·185–2·483 Å for $Y_2Ti_2O_7$ (Becker & Will, 1970), 2·21–2·41 Å for YBO_3 (Newnham, Redman & Santoro, 1963) and 2·299–2·443 Å for YVO_4 (Baglio & Gashurov, 1968). The range of Y–O distances in this work is somewhat smaller than the ranges in the literature. The O atoms of the diselenite groups are slightly closer to the Y atom than the O atoms of water and the nitrate group.

The Y atom is surrounded by eight O atoms, four from diselenite groups, one from the nitrate group and three from the water molecules. The polyhedron is a slightly distorted square antiprism. The test of Lippard & Russ (1968) gave the angle between two trapezoidal planes as 81·6°, which in ideal cases should be 90·0° for a dodecahedron and 77·4° for a square antiprism.

O(2), O(4), O(6) and O(8) form one square plane of the antiprism and O(1), O(5), O(7) and O(10) the other. Distances of each O atom from the least-squares planes (Table 5) show that the atoms lie in the plane. The angle between these planes is 2·50°. In an ideal square antiprism the diagonals of the different square planes form an angle of 45°. In this case diagonals O(2)–O(6) and O(1)–O(10) form an angle of 48·9° and O(2)–O(6) and O(5)–O(7) 41·9°.

O–O distances in the square antiprism are given in Fig. 2. O–O–O angles of the square plane of the square antiprism vary between 88·2 and 96·8°.

The geometry of the diselenite group is normal, with the distances and angles given in Table 3. Being quadridentate, with O(1) and O(4) connected to the same Y and O(2) and O(5) to neighbouring Y atoms, the group is thus connected to three Y square antiprisms. O(3) is bonded to two Se atoms and the angle Se(1)–O(3)–Se(2) is 124·6 (3)°. The Se–O(3) distance is the longest of the three Se–O distances, as expected.

Table 6. *Additional distances* (<3·5 Å) *around water O atoms*

Distances in the YO_8 polyhedron are not given.

O(6)–O(2)	2·880 (8)	O(7)–O(3)	3·070 (8)
O(3)	3·123 (8)	O(5)	2·795 (8)
O(7)	3·196 (8)	O(8)	3·275 (8)
O(8)	2·980 (8)	O(9)	2·941 (8)
O(9)	3·043 (8)	O(11)	3·015 (8)
O(11)	3·308 (8)		

O(8)–O(9)	3·281 (8)
O(10)	2·798 (8)
O(11)	3·273 (8)

The nitrate group is connected as a unidentate ligand to the Y atom with the longest Y–O distance. The nitrate group is almost planar, as shown in Table 4.

The structure forms layers in the *xy* plane with $0\cdot0 < z < 0\cdot5$ or $0\cdot5 < z < 1\cdot0$ (Fig. 1). These layers are formed from Y polyhedra and diselenite groups, which are joined together through common O atoms. The layers are connected by hydrogen bonds, which are probably formed between the water and the nitrate O atoms. The possible bond distances are listed in Table 6. It is, however, impossible to decide which O atoms participate in the hydrogen bonds because the positions of the H atoms could not be determined.

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Gold(III) Chloride Oxide

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Abstract. AuClO, $M_r = 248\cdot42$, trigonal, $R\bar{3}$, $a = 8\cdot148\ (3)$ Å, $\alpha = 113\cdot45\ (4)°$, $U = 341\cdot6$ Å^3, $Z = 6$, $D_m = 7\cdot45\ (10)$, $D_x = 7\cdot24$ Mg m^{-3}, μ(Mo $K\alpha$) = 65·1 mm^{-1}. $R = 0\cdot064$ for 653 unique reflexions. The polymeric structure contains $[\text{–Au(Cl)–O–}]_6$ rings in which the Cl atoms project inwards and alternately above and below. These rings are linked by $[\text{–Au–O–}]_2$ squares with Au···Au = 3·073 (2) Å. The coordination geometry at Au is approximately square planar, with Au–O 2·07 (1) (*trans* to Cl), 1·99 (2) and 2·01 (2) Å (*cis*), and Au–Cl 2·244 (4) Å.

Introduction. We first reported the preparation of gold(III) chloride oxide, AuClO, in microcrystalline form (Schwarzmann, Schulze & Mohn, 1974). We have since succeeded in growing larger single crystals (prisms up to 1 mm in length) by a similar method (Rumpel, 1975; quoted in Brauer, 1978); from such a crystal we have determined the structure of AuClO.

Intensities were measured on a Stoe four-circle diffractometer with monochromated Mo $K\alpha$ radiation and a crystal 0·3 × 0·15 × 0·15 mm. A complete sphere of data was collected up to $2\theta = 70°$. Cell constants were obtained by least squares from angle measurements of 27 strong high-angle reflexions. Lp corrections were applied, as were empirical absorption corrections based on measurements of strong equivalent reflexions at different azimuthal angles. The 4872 reflexions were then merged to give 669 unique reflexions, of which 653 had $F > 5\sigma(F)$.

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