Table 1. *Results of the refinement of the structure of* YbO(OH)

Scattering amplitudes are, Yb: 12·62, O: 5·803 and H: −3·7409 fm. Scale factor = 0·0131 (1), $R(F)$ = 5·3%.

	Atom site	x	y	z	B (Å^2)
Yb	2(*e*)	0·1918 (7)	0·75	0·331 (1)	0·9
O(1)	2(*e*)	0·058 (2)	0·75	0·771 (3)	−0·1 (2)
O(2)	2(*e*)	0·571 (2)	0·75	0·755 (2)	1·0 (2)
H	2(*e*)	0·582 (3)	0·75	0·988 (5)	2·6 (3)

not change the atomic positions and B values significantly. Hence, we may conclude that the powder-profile-refinement technique is insensitive to small changes of the scattering amplitudes when used on the available powder diffraction data of YbO(OH) and TbO(OH).

Discussion. The investigation confirms that TbO(OH) and YbO(OH) have the same monoclinic structure, that TbO(OH) orders antiferromagnetically at 7·6 K and that YbO(OH) shows no magnetic transition down to 4·2 K. The two hydroxide oxides also exist in a more dense tetragonal modification (Gondrand & Christensen, 1971; Christensen & Hazell, 1972). For YbO(OH) the metal–oxygen distances are from 2·22 (1) to 2·45 (5) Å in the tetragonal modification and from 2·25 (1) to 2·36 (1) Å in the monoclinic modification. The O—H distance in the monoclinic form of YbO(OH) is 0·98 (1) Å and the structure is not hydrogen bonded.

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Acta Cryst. (1981). B**37**, 427–429

Diammonium Copper Bis(selenate) Hexahydrate

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(*Received* 26 *June* 1980; *accepted* 17 *September* 1980)

Abstract. $Cu(NH_4)_2(SeO_4)_2.6H_2O$, M_r = 493·5, monoclinic, $P2_1/c$, a = 6·424 (1), b = 12·547 (1), c = 9·351 (2) Å, β = 105·62 (5)°, V = 725·8 (2) Å^3, Z = 2, D_c = 2·26 Mg m^{-3}. Final R = 0·050 for 1269 observed reflexions. The Cu atom has the characteristic 4 + 2 distorted coordination.

Introduction. The crystal structures of many double sulphates are known. This is not the case for the related Se compounds, with the exception of $CuK_2(SeO_4)_2.6H_2O$ (Whitnall, Kennard, Nimmo & Moore, 1975). In this paper the crystal structure of $Cu(NH_4)_2(SeO_4)_2.6H_2O$ is studied to establish its relation to that of the above-mentioned sulphates and selenate: in particular the coordination of the metal atom and the arrangement of the hydrogen bonds.

A pale-green crystal was mounted on a Nonius CAD-4 diffractometer. The cell dimensions were refined by least-squares fitting of the θ values of 20 reflexions. The intensities of all 1348 unique reflexions with $1 < \theta < 70°$ were measured at 295 K with monochromatic Cu $K\alpha$ radiation (λ = 1·54178 Å) and an $\omega/2\theta$ scan technique. There was no appreciable change in the periodically monitored standard reflexions. The intensities were corrected for Lorentz and polarization effects and 1269 of these were considered as observed with $I > 2\sigma(I)$. No absorption correction was applied because of the irregular shape of the crystal.

Scattering factors for neutral atoms and anomalous-dispersion corrections for Cu and Se were taken from *International Tables for X-ray Crystallography* (1974). The structure was solved by *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1977). The set of phases with the highest figure of merit gave 50% of the non-hydrogen atoms, the remainder being located in a difference synthesis. Anisotropic full-matrix least-squares refinement with unit weights led to R = 0·059.

0567-7408/81/020427-03$01.00

Successive Fourier syntheses calculated with reflexions having $\sin\theta/\lambda < 0.5$ Å^{-1} showed all the H atoms. In order to prevent bias on $\langle \Delta F\rangle$ *vs* $\langle F_o\rangle$ or $\langle \sin\theta/\lambda\rangle$, the last steps of the refinement were carried out with weights $w = w_1 w_2$ where $w_1 = 1/(a + b|F_o|)^2$ and $w_2 = 1/(c + d\sin\theta/\lambda)$ with the coefficients shown in Table 1 and calculated by *PESOS* (Martínez-Ripoll & Cano, 1975). The thermal parameters of the H atoms were considered isotropic and held invariant in the calculations. The final $R = 0.050$ and $R_w = (\sum w\Delta^2/\sum w|F_o|^2)^{1/2} = 0.061$. Table 2 shows the final atomic parameters.[*] Table 3 gives the bond lengths and angles.

Table 1. *Coefficients for the weighting scheme*

	a	*b*		*c*	*d*
$\lvert F_o\rvert < 60$	1·13	0·12	$\sin\theta/\lambda < 0.35$ Å^{-1}	2·54	−5·34
$60 < \lvert F_o\rvert$	−2·14	0·06	$0.35 < \sin\theta/\lambda < 0.57$	0·66	0·23
			$\sin\theta/\lambda > 0.57$	−24·36	44·02

Table 2. *Atomic parameters* ($\times 10^4$, *for* H $\times 10^3$) *for* $Cu(NH_4)_2(SeO_4)_2.6H_2O$

E.s.d.'s are given in parentheses. ($U = 0.022$ Å^2 for H atoms.)

	x	*y*	*z*	U_{eq} (Å^2)†
N	8570 (9)	1594 (5)	11424 (7)	284 (18)
Cu	5000	0	5000	206 (4)
Se	2479 (1)	1356 (1)	9176 (1)	185 (2)
O(1)	4744 (7)	1811 (3)	8959 (5)	275 (13)
O(2)	1268 (7)	605 (3)	7772 (4)	243 (13)
O(3)	873 (7)	2358 (3)	9253 (6)	322 (15)
O(4)	2879 (10)	666 (4)	10678 (5)	438 (18)
O(5)	2197 (7)	641 (3)	5075 (5)	228 (13)
O(6)	5331 (7)	1072 (4)	3448 (5)	231 (13)
O(7)	6779 (8)	1126 (4)	6764 (5)	289 (15)
H1(O5)	183 (17)	60 (8)	580 (13)	
H2(O5)	210 (16)	135 (9)	479 (12)	
H1(O6)	494 (19)	154 (9)	368 (13)	
H2(O6)	446 (17)	93 (8)	251 (13)	
H1(O7)	792 (20)	98 (9)	702 (12)	
H2(O7)	642 (18)	115 (8)	744 (14)	
H1(N)	843 (17)	99 (10)	1160 (12)	
H2(N)	896 (16)	198 (9)	1224 (12)	
H3(N)	738 (19)	161 (9)	1082 (12)	
H4(N)	970 (16)	181 (8)	1080 (11)	

† $U_{\text{eq}} = \frac{1}{3}\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

Table 3. *Bond lengths* (Å) *and angles* (°) *in* $Cu(NH_4)_2(SeO_4)_2.6H_2O$

E.s.d.'s	σ (Å)	σ (°)
Involving Cu atoms	0·005	0·18
Involving Se atoms	0·005	0·24
Involving other atoms	0·007	
Involving H atoms	0·11	11

Cu–O(5)	1·990	Se–O(1)	1·626
–O(6)	2·031	–O(2)	1·633
–O(7)	2·237	–O(3)	1·641
		–O(4)	1·611
O(5)–Cu–O(6)	91·6	H1(O5)–O(5)–H2(O5)	107
O(5)–Cu–O(7)	90·4	H1(O6)–O(6)–H2(O6)	107
O(6)–Cu–O(7)	88·7	H1(O7)–O(7)–H2(O7)	105
O(1)–Se–O(2)	110·5	H1(N)–N–H2(N)	112
O(1)–Se–O(3)	109·3	H1(N)–N–H3(N)	92
O(1)–Se–O(4)	111·1	H1(N)–N–H4(N)	120
O(2)–Se–O(3)	108·0	H2(N)–N–H3(N)	125
O(2)–Se–O(4)	108·7	H2(N)–N–H4(N)	104
O(3)–Se–O(4)	109·2	H3(N)–N–H4(N)	105

Discussion. $Cu(NH_4)_2(SeO_4)_2.6H_2O$ is isomorphous with the double sulphates and selenate mentioned above. The Cu atom is located at a centre of symmetry

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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 35660 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 4. *Hydrogen-bond distances* (Å) *and angles* (°)

For e.s.d.'s see Table 3.

X–H···O	Relative position of the second atom	*X*–H	H···O	*X*···O	*X*–H···O
O(5)–H1(O5)···O(2)	x,y,z	0·78	1·97	2·744	172
O(5)–H2(O5)···O(3)	$x, \frac{1}{2}-y, z-\frac{1}{2}$	0·93	1·81	2·695	159
O(6)–H1(O6)···O(1)	$x, \frac{1}{2}-y, z-\frac{1}{2}$	0·69	2·10	2·742	156
O(6)–H2(O6)···O(4)	$x, y, z-1$	0·92	1·77	2·693	178
O(7)–H1(O7)···O(2)	$1+x, y, z$	0·73	2·13	2·858	178
O(7)–H2(O7)···O(1)	x,y,z	0·73	2·16	2·847	156
N–H1(N)···O(2)	$1-x, \bar{y}, 2-z$	0·78	2·08	2·854	168
N–H2(N)···O(3)	$1+x, \frac{1}{2}-y, \frac{1}{2}+z$	0·88	2·12	2·968	161
N–H3(N)···O(1)	x,y,z	0·82	2·09	2·894	167
···O(4)*	$1+x, y, z$		2·53	3·247	123
N–H4(N)		1·08			
···O(3)*	$1+x, y, z$		1·93	2·974	162

* Asymmetrically bifurcated.

and has the typical 4 + 2 distorted coordination, as in $Cu(NH_4)_2(SO_4)_2.6H_2O$ (Brown & Chidambaram, 1969) and $CuK_2(SeO_4)_2.6H_2O$ (Whitnall *et al.*, 1975). However, the difference between the two bond distances in the equatorial plane of the CuO_6 octahedra, Cu–O(5) and Cu–O(6) [1·990 (5), 2·031 (5) Å], is less than in $CuK_2(SeO_4)_2.6H_2O$ (Whitnall *et al.*, 1975) and the S compounds.

The bond lengths and angles for the anion are as expected and similar to those in $CuK_2(SeO_4)_2.6H_2O$ (Whitnall *et al.*, 1975). All the water molecules form either inter- or intramolecular hydrogen bonds connecting the O atoms coordinated to Cu and Se. In the same way, all the H atoms of NH_4^+ are bonded to O atoms of the anion.

The geometry implied in these hydrogen bonds is given in Table 4.

All the hydrogen bonds, including O(5)–H···O(3) [O(8)–H···O(4) in Brown & Chidambaram's (1969) numbering] and the bifurcated $N{-}H(4)\genfrac{}{}{0pt}{}{\cdot\cdot O(4)}{\cdot\cdot O(3)}$, agree with those found by Brown & Chidambaram (1969).

Most of the calculations were carried out with XRAY 70 (Stewart, Kundell & Baldwin, 1970). Thanks are due to the staff of the Computing Center of JEN (Madrid) for the facilities provided on a Univac 1100/80 computer.

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Acta Cryst. (1981). B**37**, 429–431

Structure of Dimercury(II) Germanate(IV)

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(*Received* 17 *October* 1979; *accepted* 29 *September* 1980)

Abstract. Synthetic Hg_2GeO_4, orthorhombic, *Fddd*, $a_o = 6{\cdot}603\ (1)$, $b_o = 10{\cdot}596\ (2)$, $c_o = 11{\cdot}485\ (2)$ Å, $Z = 8$, $D_x = 8{\cdot}89$ Mg m^{-3}. The *R* values obtained are $R = 0{\cdot}076$, and $R_w = 0{\cdot}061$ for 683 independent reflections. The compound is isostructural with thenardite, $Na_2SO_4(V)$. The structure consists of isolated $[GeO_4]$ tetrahedra (Ge–O distance 1·753 Å) and Hg atoms with an irregular sixfold coordination. Two Hg–O distances are very short (2·08 Å) and four rather long (2·66, 2·64 Å).

Introduction. Hg_2GeO_4 was synthesized hydrothermally by Röpke & Eysel (1978) at 673 K and 2×10^8 Pa in a welded gold capsule. Hg_2GeO_4 is the only known compound in the system $HgO–SiO_2–GeO_2$. Single-crystal investigations established that it is isostructural with thenardite [$Na_2SO_4(V)$]. On the basis of geometrical considerations a rough structure proposal was given by Mehrotra, Hahn, Eysel, Röpke & Illguth (1978). The aim of the present paper is a structure refinement with particular emphasis on the O coordination of Hg.

A crystal $0{\cdot}10 \times 0{\cdot}09 \times 0{\cdot}25$ mm was used for data collection on an automatic Philips PW 1100 four-circle diffractometer with graphite-monochromatized Mo $K\alpha$ radiation ($\lambda = 0{\cdot}7107$ Å) and a θ–2θ scan ($\theta_{\max} = 50°$). The intensities of 4231 reflections were measured; of these, 693 independent reflections had $I \geq 3\sigma(I)$ and were used for the subsequent refinement. The standard deviations, $\sigma(I)$, were estimated using the formula of Stout & Jensen (1968).

0567-7408/81/020429-03$01.00