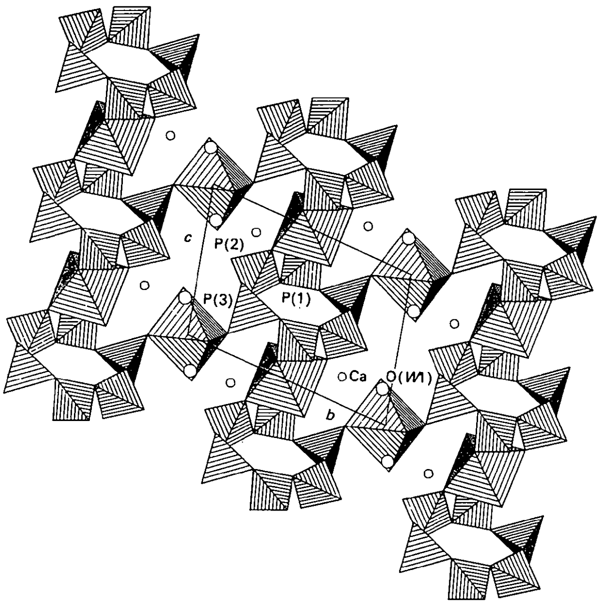

Fig. 2. Projection of the atomic arrangement of $Ca_2Li_2P_6O_{18}.8H_2O$ along the *a* axis.

As shown by Fig. 1, a projection along the *c* axis, the centrosymmetric P_6O_{18} ring anion located around the inversion centre at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ and the centrosymmetric Li(2)O_6 octahedron share two O atoms so as to build an infinite chain parallel to [110]. These chains are themselves interconnected along the **c** direction by the second lithium octahedron, Li(1)O_6, also centrosymmetric, so as to form planes parallel to [110] (Fig. 2). The connections between the phosphoric groups and the lithium octahedra are different. The Li(1)O_6 octahedron shares four O atoms with its two adjacent phosphoric groups while Li(2)O_6 shares two. These planes are themselves interconnected in a three-dimensional manner by the CaO_7 polyhedra and the hydrogen bonds whose main geometrical features are given in Table 2. The drawings were made with *STRUPLO* (Fischer, 1985).

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Structure of $Ho_2Ba_2Cu_{1+x}Pt_{1-x}O_8$ (x = 0·1)

By Yoko Saito, Kazutoshi Ukei, Toetsu Shishido and Tsuguo Fukuda

Institute for Materials Research, Tohoku University, Katahira, Aoba-ku, Sendai 980, *Japan*

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Abstract. $Ho_2Ba_2Cu_{1+x}Pt_{1-x}O_8$ ($x = 0{\cdot}1$), M_r = 978·0, orthorhombic, *Pcmn*, $a = 10{\cdot}303\,(2)$, $b = 5{\cdot}668\,(1)$, $c = 13{\cdot}178\,(3)$ Å, $V = 769{\cdot}6\,(3)$ Å^3, $Z = 4$, $D_x = 8{\cdot}44$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 50{\cdot}1$ mm^{-1}, $F(000) = 1648$, room temperature, final $R = 0{\cdot}064$ for 1473 [$|F_o| > 3\sigma(F_o)$] unique reflections. The crystal is isomorphous with $Er_2Ba_2Cu_{1{\cdot}1}Pt_{0{\cdot}9}O_8$ and is composed of a one-dimensional structure of Cu, O and Pt ions running parallel to the *b* axis.

Introduction. Recently, during the synthesis of single crystals of $ErBa_2Cu_3O_7$ which shows high-T_c superconductivity, a new compound $Er_2Ba_2Cu_{1{\cdot}1}Pt_{0{\cdot}9}O_8$ which incorporates the element Pt from a Pt crucible, was obtained (Shishido, Fukuda, Toyota, Ukei & Sasaki, 1987) and its structural studies were reported (Ukei, Shishido & Fukuda, 1988). The same type of compound with Y as the rare-earth element has been studied (Swinnea & Steinfink, 1987; Laligant, Ferey, Hervieu & Raveau, 1987).

As an extension of the investigations of this series of $R_2Ba_2(Cu,Pt)_2O_8$ (R = a rare-earth element) compounds, the Ho compound has been prepared to determine the crystal structure.

Experimental. The crystal was prepared with reference to the preparation of $Er_2Ba_2Cu_{1{\cdot}1}Pt_{0{\cdot}9}O_8$ as described elsewhere (Shishido *et al.*, 1987). Crystal size 0·63 × 0·1 × 0·1 mm; Rigaku AFC-6A four-circle diffractometer, graphite-monchromated Mo $K\alpha$ radiation; lattice parameters from 16 reflections ($60 < 2\theta < 63°$); ω–2θ scan mode, scan speed 4°

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Table 1. *Atomic fractional coordinates and equivalent isotropic thermal parameters* (Å^2) (*Hamilton*, 1959)

$$B_{eq} = \tfrac{4}{3}\Sigma_i\Sigma_j B_{ij}\mathbf{a}_i\mathbf{a}_j.$$

	x	y	z	B_{eq}
Pt	0·5329 (1)	0·25	0·3065 (1)	0·38
Ho(1)	0·8126 (1)	0·25	0·3578 (1)	0·58
Ho(2)	0·9846 (1)	0·25	0·5823 (1)	0·56
Ba(1)	0·3138 (2)	0·25	0·7934 (2)	0·84
Ba(2)	0·5919 (2)	0·25	0·5774 (2)	0·90
Cu	0·2684 (4)	0·25	0·5392 (3)	0·66
O(1)	0·446 (2)	0·008 (4)	0·218 (1)	0·97
O(2)	0·636 (2)	0·004 (3)	0·385 (1)	0·88
O(3)	0·863 (2)	−0·007 (4)	0·493 (1)	0·91
O(4)	0·390 (2)	0·25	0·410 (2)	1·16
O(5)	0·685 (2)	0·25	0·207 (2)	0·73

Table 2. *Interatomic distances* (Å) *and angles* (°)

Pt—O(1)	2·01 (2)	Pt O(2)	2·04 (2)
Pt—O(4)	2·00 (2)	Pt—O(5)	2·05 (2)
Cu—O(2^{i})	2·01 (2)	Cu—O(3^{i})	1·98 (2)
Cu—O(4)	2·12 (2)		
Ho(1)—O(1^{vi})	2·24 (2)	Ho(1)—O(2)	2·32 (2)
Ho(1)—O(3)	2·36 (2)	Ho(1)—O(5)	2·39 (2)
Ho(2)—O(1^{vii})	2·37 (2)	Ho(2)—O(3)	2·26 (2)
Ho(2)—O(3^{viii})	2·31 (2)	Ho(2)—O(5^{vii})	2·39 (2)
Ba(1)—O(1^{i})	2·88 (2)	Ba(1)—O(1^{iv})	3·16 (2)
Ba(1)—O(2^{i})	2·81 (2)	Ba(1)—O(3^{v})	3·18 (2)
Ba(1)—O(4^{iv})	2·60 (2)	Ba(1)—O(5^{ii})	2·834 (1)
Ba(2)—O(1^{i})	3·09 (2)	Ba(2)—O(2)	2·92 (2)
Ba(2)—O(2^{i})	2·80 (2)	Ba(2)—O(3)	3·35 (2)
Ba(2)—O(4)	3·03 (2)	Ba(2)—O(4^{ii})	2·84 (2)
Ba(2)—O(5^{vii})	2·86 (2)		
O(1)—Pt—O(1^{iii})	86·2 (7)	O(2)—Pt—O(2^{iii})	86·2 (7)
O(1)—Pt—O(2)	93·6 (7)	O(1^{iii})—Pt—O(2^{iii})	93·5 (7)
O(1)—Pt—O(4)	93·7 (7)	O(2)—Pt—O(4)	92·1 (7)
O(1)—Pt—O(5)	88·4 (6)	O(2)—Pt—O(5)	85·9 (7)
O(1^{iii})—Pt—O(4)	93·6 (7)	O(2^{iii})—Pt—O(4)	92·1 (7)
O(1^{iii})—Pt—O(5)	88·5 (6)	O(2^{iii})—Pt—O(5)	85·9 (7)
O(2^{i})—Cu—O(2^{ii})	91·8 (7)	O(3)—Cu—O(3^{ii})	88·1 (8)
O(2^{i})—Cu—O(3^{i})	86·7 (8)		
O(2^{i})—Cu—O(4)	96·2 (7)	O(3^{i})—Cu—O(4)	103·7 (8)
O(2^{ii})—Cu—O(4)	96·2 (7)	O(3^{ii})—Cu—O(4)	103·7 (8)
Pt—O(4)—Cu	169·2 (13)		

Symmetry code: (i) $-x+1, -y, -z+1$; (ii) $-x+1, y+\frac{1}{2}, -z+1$; (iii) $x, -y+\frac{1}{2}, z$; (iv) $-x+\frac{1}{2}, y, z+\frac{1}{2}$; (v) $x-\frac{1}{2}, -y, -z+\frac{1}{2}$; (vi) $x+\frac{1}{2}, -y, -z+\frac{1}{2}$; (vii) $-x+1\frac{1}{2}, y, z+\frac{1}{2}$; (viii) $-x+2, -y, -z+1$.

min^{-1} (ω); data collection up to $2\theta = 65°$; three standard reflections with no significant intensity variation; 5301 reflections measured (h: $-15 \rightarrow 15$, k: $-8 \rightarrow 8$, l: $0 \rightarrow 19$), of which 1473 unique reflections with $|F_o| > 3\sigma(F_o)$ were considered observed, $R_{int} = 0{\cdot}061$, absorption correction ($16{\cdot}04 < A^* < 192{\cdot}37$), correction for Lorentz and polarization effects.

From the possible space groups, *Pcmn* and *Pc*2$_1$*n*, *Pcmn* was adopted from statistical distribution of the observed intensities. *Pcmn* proved to be the correct choice also in the later stages of the structure determination.

On the assumption that this compound is isomorphous with $Er_2Ba_2Cu_{1{\cdot}1}Pt_{0{\cdot}9}O_8$, the structure was solved using the atomic parameters of the Er compound in the first stage of the structure determination. For the population parameter x of Cu atoms at the Pt position, 0·1 was used from a consideration of the results of EPMA analysis and the R value. Refinement by full-matrix least squares on F of positional and anisotropic thermal parameters for all atoms converged to a final $R = 0{\cdot}064$, $wR = 0{\cdot}077$ [$w = 1/\sigma^2(F)$] and $(\Delta/\sigma)_{max} = 0{\cdot}28$ in the last cycle. The final difference Fourier map shows maximum and minimum heights of 11·1 and $-11{\cdot}0$ e Å^{-3}. The value of g for the isotropic secondary extinction (Zachariasen, 1967) is $5{\cdot}7\,(2) \times 10^{-5}$.

All calculations were carried out using the *UNICS*III program system (Sakurai & Kobayashi, 1979) and the full-matrix least-squares program *RADIEL* (Coppens, Guru Row, Leung, Stevens, Becker & Yang, 1979). Atomic scattering factors for the O^{2-} ion were taken from Tokonami (1965), and those for other ions from *International Tables for X-ray Crystallography* (1974).

Discussion. Final atomic coordinates and selected interatomic distances and angles are presented in Tables 1 and 2.* The structure of the title compound is essentially the same as described for $Er_2Ba_2Cu_{1{\cdot}1}Pt_{0{\cdot}9}O_8$. The coordination of the Cu and Pt atoms is shown in Fig. 1. A distorted square-pyramidal coordination of the Cu ion and a distorted octahedral coordination of the Pt ion are typical types of coordination for Cu^{2+} and Pt^{4+} ions, respectively. As shown in Fig. 2, double —O—Pt—O—Cu— zigzag chains compose a one-dimensional column-like structure running along the b axis.

The measurements of temperature-dependent conductivity have made it clear that the conductance is proportional to $\exp(-T^{-1/2})$ and exhibits semiconductivity with a narrow energy gap. This result might be explained by the Mott–Hubbard model (Toyota, Koorevaar, van den Berg, Kes, Mydosh, Shishido, Saito, Kuroda, Ukei, Sasaki & Fukuda, 1989).

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52287 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

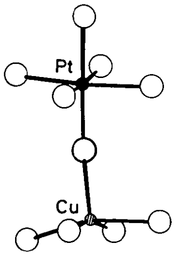

Fig. 1. The coordination geometry of the Pt and Cu ions, surrounded by O ions. Atom key: open circles O, filled circle Pt, shaded circle Cu.

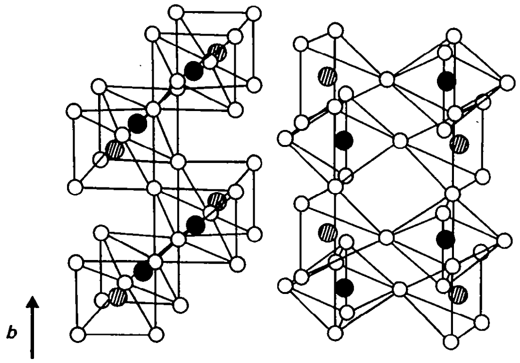

Fig. 2. Column-like units containing the double zigzag chains of Cu, O and Pt ions. Atom key: open circles O, filled circles Pt, shaded circles Cu.

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Structure of a Triclinic Rubidium Ammonium Triaqua-μ_3-oxo-hexa-μ-sulfato-triferrate(III) Tetrahydrate, $Rb_{2{\cdot}74}(NH_4)_{2{\cdot}26}Fe_3O(SO_4)_6.7H_2O$

By K. Mereiter

Institut für Mineralogie, Kristallographie und Strukturchemie, Technische Universität Wien, Getreidemarkt 9, *A*-1060 *Vienna, Austria*

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Abstract. $Rb_{2{\cdot}74}(NH_4)_{2{\cdot}26}Fe_3O(SO_4)_6.7H_2O$, $M_r = 1160{\cdot}9$, triclinic, $P\bar{1}$, $a = 9{\cdot}783$ (3), $b = 9{\cdot}586$ (3), $c = 18{\cdot}389$ (4) Å, $\alpha = 95{\cdot}30$ (2), $\beta = 93{\cdot}19$ (2), $\gamma = 118{\cdot}12$ (2)°, $V = 1504{\cdot}8$ (4) Å^3, $Z = 2$, $D_x = 2{\cdot}57$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 62{\cdot}3$ cm^{-1}, $F(000) = 1140$, $T = 295$ K, final $R = 0{\cdot}033$ for 4528 observed reflections. The structure contains discrete trinuclear $[Fe_3(H_2O)_3O(SO_4)_6]^{5-}$ units arranged in close-packed pseudotrigonal layers parallel to (001) at $z \sim 1/4$ and $z \sim 3/4$. The units are tied together by Rb and NH_4 ions which are statistically but not evenly distributed over five independent cation sites. Four Rb/NH_4 bonded water molecules complete the structure. Average bond lengths are: S—O = 1·470 (22) Å, Fe—O = 2·006 (50) Å for all types of O and 1·924 (17) Å for the oxo O atom coordinated by three Fe atoms (e.s.d.'s for individual bond lengths ≤ 0·004 Å).

Introduction. Studies in the system A_2SO_4–$Fe_2(SO_4)_3$–H_2O, where A = Na, K, Rb, Cs, NH_4 or Tl or combinations thereof, yielded many novel hydrated basic iron sulfates, many of which are now known to contain the trinuclear anion $[Fe_3(H_2O)_3O(SO_4)_6]^{5-}$ as a fundamental constituent. Two subgroups of this family of salts have been investigated in some detail; the first one comprises crystals containing sodium and potassium with trigonal or hexagonal symmetry, complicated disorder phenomena and merohedral twinning (Scordari & Milella, 1983, 1984; Giacovazzo, Scordari & Menchetti, 1975), the second subgroup comprises crystals with potassium, rubidium and thallium, monoclinic symmetry and essentially ordered structures (Mereiter & Völlenkle, 1978, 1980; Mereiter, 1980). The title compound, which is pseudotrigonal triclinic in unit-cell dimensions, is of interest as a possible link between the two subgroups.

Experimental. Solutions of 2 g Rb_2SO_4, 1·5 g $(NH_4)_2SO_4$ and 4·5 g $Fe_2(SO_4)_3$ in 40 g H_2O were slowly evaporated at 340–350 K and yielded large