SHORT-FORMAT PAPERS

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The Structure of $Y_2Ba_2CuPtO_8$

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Abstract. Diyttrium dibarium copper platinum oxide, $M_r = 839{\cdot}11$, orthorhombic, *Pnma*, $a = 13{\cdot}191\ (2)$, $b = 5{\cdot}680\ (1)$, $c = 10{\cdot}301\ (2)$ Å, $V = 771{\cdot}9\ (3)$ Å^3, $Z = 4$, $D_x = 7{\cdot}227$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 460{\cdot}42$ cm^{-1}, $F(000) = 1444$, $T = 298$ K, $R = 0{\cdot}050$, $wR = 0{\cdot}053$ for 1026 unique reflections, $F_o \geq 5\sigma(F_o)$. Y and Ba have sevenfold oxygen coordination. Y—O bond lengths vary from 2·22 (1)–2·40 (2) Å. Ba—O bond distances vary from 2·60 (1) to 2·92 (1) Å. The coordination about Pt is octahedral while that of Cu is square pyramidal. The average Pt—O distance is 2·04 (1) Å. The apical Cu—O distance is 2·12 (1) Å, and the average basal Cu—O distance is 1·99 (1) Å. The various polyhedra share edges and corners to create a three-dimensional framework.

Experimental. $Y_2Ba_2CuPtO_8$ crystals were isolated from a mixture of Y_2O_3, $BaCO_3$ and CuO heated to 1373 K in a Pt crucible. The mixture was prepared to synthesize $YBa_2Cu_3O_6$ (Swinnea & Steinfink, 1987), a high-temperature product of a 90 K superconductor (Steinfink, Swinnea, Sui, Hsu & Goodenough, 1987). When the crystals were examined optically in reflected light their appearances indicated that they were not single. The Weissenberg X-ray diffraction patterns from several crystals showed weak 'ghost' reflections due to a second crystal that formed a low-angle grain boundary with the main crystal. A metallic grey columnar parallelepiped, $0{\cdot}10 \times 0{\cdot}13 \times 0{\cdot}17$ mm, which also displayed such a second fragment, was selected and placed on a Syntex $P2_1$ automatic diffractometer operated in variable ω-scan mode (3–6° min^{-1}) with an incident-beam graphite monochromator. Lattice constants were obtained from a least-squares fit of 30 reflections in the range $24 < 2\theta < 30°$. Four standard reflections (162, 561, 812, 831) measured every 100 reflections did not show any significant change during data collection (<1%). 2248 reflections in the range

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* ($\times 10^4$) *with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}(Å^2)
Y(1)	0·0829 (1)	¼	0·4842 (2)	57 (5)
Y(2)	0·3580 (1)	¼	0·1875 (2)	61 (5)
Ba(1)	0·79347 (9)	¼	0·6863 (1)	79 (3)
Ba(2)	0·07777 (9)	¼	0·0919 (1)	89 (3)
Cu	0·0396 (2)	¼	0·7689 (2)	80 (6)
Pt	0·30630 (6)	¼	0·46701 (7)	52 (2)
O(1)	0·410 (1)	¼	0·610 (1)	77 (36)
O(2)	0·9928 (8)	0·506 (2)	0·3605 (9)	86 (25)
O(3)	0·7828 (8)	0·509 (2)	0·442 (1)	116 (28)
O(4)	0·205 (1)	¼	0·312 (2)	100 (39)
O(5)	0·6136 (7)	0·507 (2)	0·637 (1)	76 (25)

Table 2. *Interatomic distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Y(1)–[2]O(2)	2·27 (1)	O(1)–Cu–O(2) [2]	102·9 (4)
–[2]O(2)	2·34 (1)	O(1)–Cu–O(5) [2]	96·3 (4)
–[2]O(3)	2·36 (1)	O(2)–Cu–O(2) [2]	89·5 (6)
–[1]O(4)	2·40 (2)	O(2)–Cu–O(5) [2]	85·4 (4)
		O(5)–Cu–O(5) [1]	93·5 (6)
Y(2)–[2]O(2)	2·35 (1)	O(2)–Cu–O(5) [2]	160·8 (4)
–[2]O(3)	2·22 (1)		
–[1]O(4)	2·39 (2)	O(1)–Pt–O(3) [2]	93·3 (4)
–[2]O(5)	2·30 (1)	O(1)–Pt–O(5) [2]	92·0 (4)
		O(3)–Pt–O(3) [1]	84·6 (6)
Ba(1)–[1]O(1)	2·60 (1)	O(3)–Pt–O(4) [2]	88·8 (4)
–[2]O(3)	2·92 (1)	O(3)–Pt–O(5) [2]	94·9 (4)
–[2]O(4)	2·8404 (8)	O(4)–Pt–O(5) [2]	85·9 (4)
–[2]O(5)	2·83 (1)	O(5)–Pt–O(5) [1]	85·0 (5)
		O(1)–Pt–O(4) [1]	177·2 (6)
Ba(2)–[2]O(1)	2·85 (1)	O(3)–Pt–O(5) [2]	174·7 (4)
–[1]O(4)	2·82 (2)		
–[2]O(5)	2·82 (1)		
–[2]O(5)	2·92 (1)		
Cu–[1]O(1)	2·12 (1)		
–[2]O(2)	1·97 (1)		
–[2]O(5)	2·00 (1)		
Pt–[1]O(1)	2·01 (1)		
–[2]O(3)	2·03 (1)		
–[1]O(4)	2·09 (2)		
–[2]O(5)	2·04 (1)		

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$4 < 2\theta < 60°$, $-4 \le h \le 18$, $-7 \le k \le 7$, $-2 \le l \le 14$ were measured and yielded 1230 unique reflections, $R_{int} = 0{\cdot}034$. Of those, 1026 were considered observed on the basis that $F_o \ge 5\sigma(F_o)$. The data were corrected for Lorentz and polarization effects. An analytical absorption correction was made; transmission factors ranged from 0·012 to 0·069; $\sigma(F_o)$ was calculated from counting statistics. The structure was solved and refined with *SHELX*76 (Sheldrick, 1976). The cation positions were obtained by direct methods, and oxygen positions from a difference Fourier map. A full-matrix least-squares refinement of 77 parameters minimized $\sum w(|F_o| - |F_c|)^2$, $w = 1{\cdot}0/[\sigma^2(F_o) + 0{\cdot}0015F_o^2]$. The refinement was carried out with anisotropic thermal parameters and an extinction correction $F_o = F_o(1 - 5 \times 10^{-8}F_o^2/\sin\theta)$; $S = 1{\cdot}4$, $(\Delta/\sigma)_{max} = 0{\cdot}0000$, $R = 0{\cdot}050$, $wR = 0{\cdot}053$ for 1026 observed and $R = 0{\cdot}061$, $wR = 0{\cdot}060$ for all reflections. A final $\Delta\rho$ map gave peaks <1 e Å^{-3} except in the vicinity of heavy atoms where 5 e Å^{-3} ripples were observed. Scattering factors for neutral atoms, corrected for real and imaginary parts of dispersion, were obtained from *International Tables for X-ray Crystallography* (1974). Positional and thermal parameters are listed in Table 1, bond lengths and angles are given in Table 2, and a stereographic view of the structure is shown in Fig. 1.*

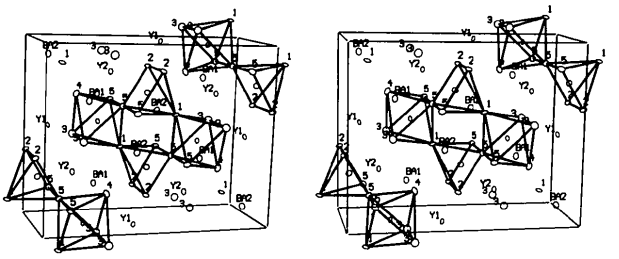

Fig. 1. Stereoview of the structure of $Y_2Ba_2CuPtO_8$. The *a* axis is horizontal and *c* vertical. Cu is in square-pyramidal and Pt in octahedral coordination.

Related literature. The structure of $YBa_2Cu_3O_{7-y}$ has been determined by neutron powder diffraction analysis (Cox, Moodenbaugh, Hurst & Jones, 1987; Capponi *et al.*, 1987; Beno *et al.*, 1987; Beech, Miraglia, Santoro & Roth, 1987). The structure of the related $YBa_2Cu_3O_6$ has been determined by single-crystal X-ray analysis by Swinnea & Steinfink (1987).

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* Tables of anisotropic thermal parameters and of structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44229 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tetraphenylphosphonium-octachlorodirhenat(III)–Dichloromethan (1/2)

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Abstract. $[P(C_6H_5)_4]_2[Re_2Cl_8].2CH_2Cl_2$, $M_r = 1504{\cdot}7$, triclinic, $P\bar{1}$, $a = 10{\cdot}615$ (3), $b = 11{\cdot}589$ (2), $c = 12{\cdot}345$ (1) Å, $\alpha = 84{\cdot}11$ (1), $\beta = 71{\cdot}23$ (2), $\gamma = 70{\cdot}65$ (2)°, $V = 1350{\cdot}1$ Å^3, $Z = 1$, $D_x = 1{\cdot}84$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 4{\cdot}896$ mm^{-1}, $F(000) = 728$, $T = 293$ K, $R = 0{\cdot}041$ for 2873 observed independent reflexions. $[P(C_6H_5)_4]_2[Re_2Cl_8].2CH_2Cl_2$ was prepared by the reaction of $[P(C_6H_5)_4]_2[Re_2Cl_9]$ with *N,N'*-dichloro-1,4-benzoquinone diimine in CH_2Cl_2 solution; it crystallizes upon cooling of the solution. The

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