2·993 (2) Å and by three Sb(3) at 3·445 (2) Å, as in SbTe. Te(2) displays six equidistant Sb(1) neighbours at 3·176 (2) Å, as in Sb_2Te_3. Sb(1) is surrounded by six Te atoms, 3 Te(1) at 2·993 (2) Å and 3 Te(2) at 3·176 (2) Å, as in SbTe and Sb_2Te_3. The Sb(2) surrounding, which consists of six Sb atoms [3 Sb(3) at 2·902 (1) and 3 Sb(2) at 3·400 (2) Å], is like that in α-antimony. Sb(3) differs slightly from Sb(2) because it is bonded to 3 Te(1) at 3·455 (2) Å and 3 Sb(2) at 2·902 (1) Å. Bond length values in SbTe, Sb_2Te_3 and α-Sb are given for comparison in Table 2.

The layered structure obtained for Sb_2Te exhibits some similarities with the known structures of SbTe and Sb_2Te_3. Sb_2Te is a stacking of Sb_2Te_3 and Sb_2 in the ratio 1:2, and SbTe is known to consist of Sb_2Te_3–Sb_2–Te_3Sb_2. Thus, it may be now concluded that all known Sb–Te compounds are made from Sb_2Te_3 (five layers) as the basic sublattice component.

The authors wish to thank Mr M. Fedoroff, CECM, Vitry, for the neutron activation analysis.

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Acta Cryst. (1991). C**47**, 1143–1145

Redetermination of the Structure of the 80 K Superconductor $YBa_2Cu_4O_8$ by Time-of-Flight Neutron Powder Diffraction

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(*Received* 6 *July* 1990; *accepted* 9 *October* 1990)

Abstract. $M_r = 745·77$, orthorhombic, *Ammm*, $a = 3·8402\ (1)$, $b = 3·8708\ (1)$, $c = 27·2309\ (3)$ Å, $V = 404·8$ Å^3, $D_x = 6·119$ g cm^{-3}, $Z = 2$, $T = 295$ K, 912 reflections included, $R_{wp} = 5·62\%$, $R_{\rm exp} = 3·34\%$. Rietveld analysis of high-resolution time-of-flight powder neutron diffraction data has been used to redetermine the structure of the 80 K superconductor $YBa_2Cu_4O_8$ (the '124 phase'). The essential features of the structure previously reported have been confirmed. This study improves upon the precision of the previously reported structural parameters, obtained by powder neutron diffraction. The compound is stoichiometric, with no occupancy of the O(5) inter-chain site.

0108-2701/91/061143-03$03.00

Introduction. The structure of the 80 K superconductor $YBa_2Cu_4O_8$ (the '124 phase') was first reported by Marsh, Fleming, Mandich, DeSantolo, Kwo, Hong & Martinez-Miranda (1988) by X-ray diffraction from a thin film. The structure was found to differ from the $YBa_2Cu_3O_7$ ('123') structure in having an extra Cu–O chain layer intercalated between the Cu–O sheets. Subsequently, Fischer, Karpinski, Kaldis, Jilek & Rusiecki (1989) have presented a more accurate refinement from powder neutron diffraction data. In addition, Kaldis, Fischer, Hewat, Karpinski & Rusiecki (1989) have carried out a detailed structural study of the 124 system as a function of temperature and pressure. The pressure dependence of T_c in this system is one of its most interesting aspects, being one of the highest reported values for dT_c/dp, 0·55 K/kbar (Bucher, Karpinski, Kaldis & Wachter, 1989). We have recently undertaken a very detailed structural study of this system as a function of pressure (Yamada *et al.*, 1991) and, as a prelude to this, have redetermined the structure of 124 under ambient conditions. This brief report describes the results of this study.

Experimental. A sample with chemical composition $YBa_2Cu_4O_8$ was synthesized by a high-oxygen-pressure technique using hot isostatic pressing in a gas environment of argon with 20% oxygen (O_2-HIP). Starting materials, Y_2O_3, $BaCO_3$ and CuO, were thoroughly mixed and calcined twice at 1173 K for 12 h, with intermediate grinding. The reacted powder was pressed into a pellet and then HIP-treated in ($Ar + 20\%O_2$) gas environment of 100 MPa at 1283 K for 6 h.

Neutron powder diffraction data were collected by the time-of-flight method on the Special Environment Powder Diffractometer (SEPD) at the Intense Pulsed Neutron Source, Argonne (Jorgensen *et al.*, 1989), with the sample enclosed in a sealed vanadium can.

Table 1. *Refined structural parameters for* $YBa_2Cu_4O_8$ *at room temperature*

Space group *Ammm* (No. 65); $Z = 2$, $a = 3{\cdot}8402\ (1)$, $b = 3{\cdot}8708\ (1)$, $c = 27{\cdot}2309\ (3)$ Å; 912 reflections included ($0{\cdot}52$ Å $< d < 3{\cdot}16$ Å); $R_{wp} = 5{\cdot}62\%$, $R_{\rm exp} = 3{\cdot}34\%$.

	Site	x	y	z	B(Å^2)
Y	2(*c*)	$\frac{1}{2}$	$\frac{1}{2}$	0	0·49 (2)
Ba	4(*h*)	$\frac{1}{2}$	$\frac{1}{2}$	0·13502 (7)	0·48 (3)
Cu1	4(*g*)	0	0	0·21296 (4)	0·43 (2)
Cu2	4(*g*)	0	0	0·06138 (4)	0·30 (2)
O1	4(*g*)	0	0	0·14562 (6)	0·73 (3)
O2	4(*h*)	$\frac{1}{2}$	0	0·05253 (6)	0·55 (3)
O3	4(*g*)	0	$\frac{1}{2}$	0·05214 (6)	0·47 (3)
O4	4(*g*)	0	$\frac{1}{2}$	0·21822 (7)	0·77 (3)

B is an isotropic temperature factor.

Table 2. *Metal-to-oxygen distances in* $YBa_2Cu_4O_8$ *at room temperature*

M—O	*d*(Å)	*M*—O	*d*(Å)
Cu1—O1	1·834 (2)	Ba—O1	2·7415 (3)
Cu1—O4	1·9407 (2)	Ba—O2	2·965 (2)
Cu1—O4′	1·8740 (2)	Ba—O3	2·963 (2)
Cu2—O1	2·294 (2)	Ba—O4	2·970 (2)
Cu2—O2	1·9352 (2)	Y—O2	2·407 (1)
Cu2—O3	1·9517 (3)	Y—O3	2·388 (1)

Discussion. Structural analysis by the Rietveld method (Von Dreele, Jorgensen & Windsor, 1982) was carried out using the high-resolution ($\Delta d/d = 0{\cdot}0035$ at $2\theta = 150°$) back-scattering data. The refinement included a total of 912 reflections in the range $0{\cdot}52 < d < 3{\cdot}2$ Å. In addition, the presence of a very small amount (< 2%) of CuO was apparent in the raw data. This was therefore included as a secondary phase in a two-phase Rietveld refinement. Initial parameters used were those of Fischer *et al.* (1989). Scattering lengths used were $b(\mathrm{Y}) = 0{\cdot}775 \times 10^{-12}$ cm, $b(\mathrm{Ba}) = 0{\cdot}525 \times 10^{-12}$ cm, $b(\mathrm{Cu}) = 0{\cdot}772 \times 10^{-12}$ cm and $b(\mathrm{O}) = 0{\cdot}580 \times 10^{-12}$ cm. In order to test for occupancy of the *inter*-chain O(5) site, the z and $B_{\rm iso}$ values for this atom were constrained to be equal to those of the *intra*-chain O(4). This resulted in marginally negative occupancy [$-0{\cdot}01$ (1)] for the O(5) site, and subsequently the occupancy was fixed at zero. Additionally, occupancies of all the O atoms were refined, with all being found to be fully occupied, within e.s.d.'s. The compound is therefore found to be stoichiometric, $YBa_2Cu_4O_8$. The final refinement converged to $R_{wp} = 5{\cdot}62\%$, $R_{\rm exp} = 3{\cdot}34\%$. Final refined structural parameters are given in Table 1,* with selected bond distances in Table 2. A portion of the final Rietveld fit at room temperature is given in Fig. 1.

Our results are in general agreement with those of Fischer *et al.* (1989) but are of improved precision due to the increased resolution, *d* spacing range, and quantity of data. The presence of a very small amount of CuO impurity in our sample is well accounted for by the two-phase refinement, and does not affect the accuracy of the refined parameters for 124. Our results on the high-pressure structural properties of the 124 system will be published shortly.

Work at Argonne is supported by the US Department of Energy, Division of Basic Energy Sciences – Materials Sciences, under Contract No. W-31-109-

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53638 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

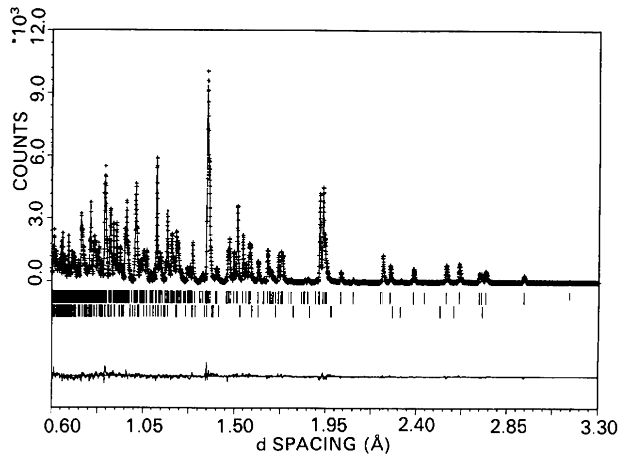

Fig. 1. Portion of the final Rietveld fit for $YBa_2Cu_4O_8$ at room temperature. Plus marks (+) represent the raw data, the solid line is the calculated profile. A difference (obs. − calc.) plot is shown beneath. The two sets of tick marks represent reflection positions for $YBa_2Cu_4O_8$ (upper) and CuO (lower).

ENG-38 (JDJ) and the National Science Foundation, Science and Technology Center for Superconductivity, under grant No. DMR 88-09854 (PL, SP).

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Acta Cryst. (1991). C**47**, 1145–1148

Structure of Trisodium Monohydroxogermanate(IV) Pentahydrate

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(*Received* 22 *April* 1987; *accepted* 2 *January* 1991)

Abstract. $Na_3[GeO_3(OH)].5H_2O$, $M_r = 296{\cdot}6$, orthorhombic, *Pbca*, $a = 11{\cdot}889\,(6)$, $b = 11{\cdot}039\,(2)$, $c = 13{\cdot}090\,(2)$ Å, $V = 1717{\cdot}9$ Å^3, $Z = 8$, $D_m = 2{\cdot}18$, $D_x = 2{\cdot}29$ Mg m^{-3}, λ(Mo $K\bar{\alpha}$) = 0·71073 Å, $\mu = 3{\cdot}68$ mm^{-1}, $F(000) = 1128$, $T = 296$ K, $R = 0{\cdot}034$ for 1266 independent reflections. Isostructural relation established with $Na_3[SiO_3(OH)].5H_2O$. All H atoms located from ΔF map. The structure of $Na_3[GeO_3(OH)].5H_2O$ shows centrosymmetric dimeric units of hydrogen bonded $[GeO_3(OH)]^{3-}$ tetrahedra. The hydrogen bonds of the edge-sharing tetrahedra are short and strong [O1···O3 = 2·568 (3) Å]. The Na atoms show very distorted Na—O polyhedra, two Na atoms with coordination number 7 and one with coordination number 6. The face-sharing Na—O polyhedra show a wave-like extension along [001]. A coordination number of 5 is observed for all water molecules with two H and three Na atoms coordinating the O atom.

Introduction. There are a limited number of phase analogues in the series of sodium silicate and sodium germanate hydrates known so far which have the hydroxoorthoanions $[XO_{4-x}(OH)_x]^{(4-x)-}$ with $X =$ Si,Ge, $x = 1,2$ in common. Four hydrate phases are known in the silicate series $Na_2[SiO_2(OH)_2].nH_2O$ with $n = 4,5,7,8$. Corresponding germanates show $n = 5$ (Jamieson & Dent Glasser, 1967) and $n = 6$ (Nowotny & Szekely, 1952; Bauren & Ingri, 1967; Schmid, Ketterer, Felsche & Hiller, 1988). We report here on the structure of sodium germanate pentahydrate $Na_3[GeO_3(OH)].5H_2O$ which is the first analogue known of the silicate series $Na_3[SiO_3(OH)].nH_2O$ which shows $n = 5$ (Smolin, Shepelev & Butikova, 1973), $n = 2$ (Schmid, Huttner & Felsche, 1979; Schmid, Zsolnai, Felsche & Huttner, 1980; Schmid & Felsche, 1983) and $n = 1$ (Schmid & Felsche, 1990).

In the context of our research program which is concerned with the mechanisms of melting or peritectical decomposition of the water-rich hydrates of oxosalts we are interested in the structural details especially in the hydrogen bonding of the phases of both series. This is because of the unique thermal properties of the dihydroxo hydrates $Na_2[SiO_2(OH)_2].nH_2O$ of the system Na_2O—SiO_2—H_2O

0108-2701/91/061145-04$03.00