$\Delta\rho_{min} = -0.29$ e Å^{-3}; atomic scattering factors were those incorporated in *SHELX*76.

Final atomic positional and isotropic displacement parameters for non-H atoms are given in Table 1. Table 2 lists interatomic distances and valence angles. Drawings of Si(NHCH$_3$)$_4$ are shown in Figs. 1 and 2.*

Related literature. The reactions of SiCl$_4$ with CH$_3$NH$_2$ in the gas phase (Drake & Westwood, 1971), without solvent at room temperature (Hagen & Callaway, 1972), in *n*-pentane (Andersch & Jansen, 1990) and in petroleum ether (Adrianov, Il'in, Talanov, Isakova & Sidorenko, 1976) have been reported in the literature. Based on an elementary analysis Adrianov suggested the same composition as determined for the title compound

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* Lists of structure factors, anisotropic displacement parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53013 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

for one of his products; however, no information about the molecular structure was given.

The electronic structures of SiR_4 (R = Me,Ph,NH-Ph,OPh) have been dicussed by Tatevosyan, Shuvalev & Kopylov (1982). One mole of SiCl$_4$ combines with four moles of R(C$_6$H$_4$)NH$_2$ (R = *o*-Et, NO$_2$, *p*-CHO) when mixed in benzene to give coloured, fairly stable, complexes (Pandey, 1972).

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SHORT COMMUNICATIONS

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible.*

Structural refinement of Y$_2$BaCuO$_5$ using neutron powder diffraction. By SHIYOU PEI,* A. P. PAULIKAS,†

B. W. VEAL† and J. D. JORGENSEN,† *Science and Technology Center for Superconductivity and Materials Science Division, Argonne National Laboratory, Argonne, IL 60439, USA*

Abstract

$M_r = 458.49$, orthorhombic, *Pbnm*, $a = 7.1342$ (1), $b = 12.1811$ (1), $c = 5.6580$ (1) Å, $V = 491.70$ (1) Å^3, $Z = 4$, $D_x = 6.197$ g cm^{-3}, 1907 reflections included, $R_{wp} = 0.0449$, $R_{exp} = 0.0293$. The crystal structure of Y$_2$BaCuO$_5$ has been refined from time-of-flight neutron powder diffraction data using Rietveld analysis. Our results confirm the structure determined earlier by X-ray work and permit a more precise determination of the position, occupancy, and temperature factor of atoms in this compound.

The Y$_2$BaCuO$_5$ compound was first discovered by Michel & Raveau (1982) and later became known as the 'green

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phase' or '211' phase (Hazen *et al.*, 1987) in the study of the Y$_2$O$_3$–BaO–CuO system where the high-T_c superconducting compound YBa$_2$Cu$_3$O$_7$ ('123' phase) was found (Wu *et al.*, 1987). The 211 phase is a semiconductor (Cava *et al.*, 1987) and exhibits magnetic ordering at 15 K (Weidinger *et al.*, 1988). The crystal structure of this compound (space group *Pbnm*) was solved by Michel & Raveau (1982) from powder X-ray diffraction data and later confirmed by Hazen *et al.* (1987) from single-crystal X-ray diffraction data. However, these X-ray measurements may have limited sensitivity to O atoms. Indeed, the original X-ray studies of YBa$_2$Cu$_3$O$_{7-x}$ did not reveal the unique ordering of oxygen vacancies later determined by neutron powder diffraction (Jorgensen, 1987). Additionally, the single-crystal work on Y$_2$BaCuO$_5$ provided only the atom positions with no estimates of uncertainties being reported (Hazen *et al.*, 1987). The site occupancies were not refined although microprobe analysis indicated

that the 211 samples studied could be yttrium (and accordingly oxygen) deficient (Hazen *et al.*, 1987). Thus, we report a structural study of the stoichiometric 211 compound based on the Rietveld refinement of neutron powder diffraction data to establish precisely all structural parameters of this compound.

A sample of Y_2BaCuO_5 weighing approximately 12 g was prepared from a stoichiometric mixture of Y_2O_3, $BaCO_3$ and CuO. The powders were mixed in a rotating ball mill, pressed into a pellet and heated at 1238 K in flowing O_2 for 17 h. After a furnace cool, the product was reground, repressed and heated for 19 h between 1253 and 1303 K, again in flowing O_2, followed by a furnace cool.

Neutron powder diffraction data were collected using the Special Environment Powder Diffractometer (SEPD) at the Argonne Intense Pulsed Neutron Source (Jorgensen *et al.*, 1989). Although time-of-flight data were collected simultaneously at several different scattering angles, only the high-resolution back-scattering data ($\Delta d/d \simeq 0.0035$ at $2\theta = 150°$) were used for full structural analysis by the Rietveld method (Von Dreele, Jorgensen & Windsor, 1982). The refinement included a total of 1907 Bragg reflections, covering a d-spacing range of 0.53–3.3 Å, and used the results of Michel & Raveau (1982) as starting values. The refinement quickly converged and yielded an excellent fit to the powder diffraction profile (Fig. 1).* Refined atom positions, isotropic temperature factors, site occupancies, and metal–oxygen distances are reported in Tables 1 and 2. The refined atom positions agree well with the earlier X-ray results but are determined to higher precision. Additionally, our refinement shows that all Y atoms, as well as O atoms, have similar isotropic temperature factors (0.17 Å^2 for yttrium and 0.44 Å^2 for oxygen).

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* The numbered intensity of each measured point on the profile has been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53413 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
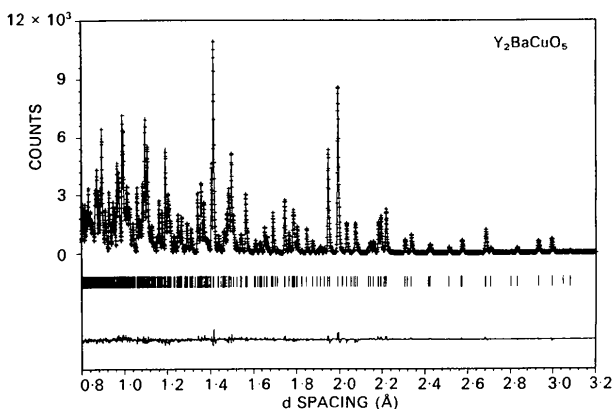

Fig. 1. Portion of the Rietveld refinement profile for orthorhombic (*Pbnm*) Y_2BaCuO_5 at room temperature. Plus marks (+) are the raw data. The solid line is the calculated profile. Tick marks below the profile mark the positions of allowed reflections. A difference curve (observed minus calculated) is plotted beneath. Background has been fit as part of the refinement but subtracted prior to plotting.

Table 1. *Refined structural parameters for* Y_2BaCuO_5 *at room temperature: scattering lengths for* Y, Ba, Cu *and* O *are, respectively,* 0.775, 0.525, 0.772 *and* 0.5805 *in units of* 10^{-12} cm

	Site	x	y	z	$B(Å^2)$	Occupancy
Y1	4(c)	0.1159 (2)	0.2881 (1)	$\frac{1}{4}$	0.16 (2)	0.98 (1)
Y2	4(c)	0.3957 (2)	0.0736 (1)	$\frac{1}{4}$	0.17 (2)	0.98 (1)
Ba	4(c)	0.0694 (3)	0.0946 (2)	$\frac{1}{4}$	0.50 (5)	0.98 (1)
Cu	4(c)	0.2870 (2)	0.3406 (1)	$\frac{1}{4}$	0.25 (3)	1
O1	8(d)	0.1655 (1)	0.4327 (1)	0.5070 (2)	0.42 (2)	2.01 (1)
O2	8(d)	0.3563 (2)	0.2273 (1)	0.5043 (3)	0.47 (3)	2.00 (1)
O3	4(c)	0.0796 (2)	0.0998 (1)	$\frac{1}{4}$	0.43 (4)	1.01 (1)

Table 2. *Metal-to-oxygen distances* (Å) *in* Y_2BaCuO_5 *at room temperature*

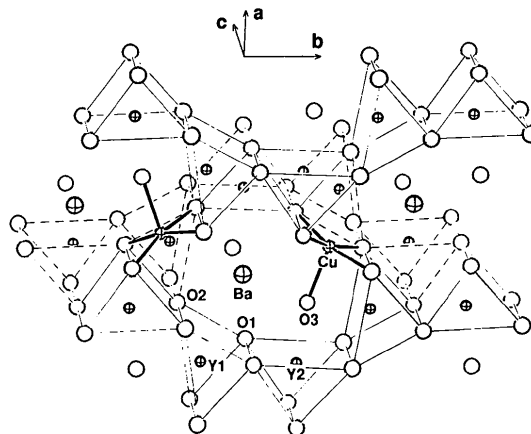|			
Ba—O1 × 2	3.059 (2)	Y1—O1 × 2	2.311 (2)
Ba—O1 × 2	3.245 (2)	Y1—O2 × 2	2.358 (2)
Ba—O2 × 2	2.956 (2)	Y1—O2 × 2	2.323 (2)
Ba—O2 × 2	3.014 (2)	Y1—O3 × 1	2.309 (2)
Ba—O3 × 2	2.8307 (3)	Y2—O1 × 2	2.367 (2)
Ba—O3 × 1	2.596 (1)	Y2—O1 × 2	2.291 (2)
Cu—O1 × 2	1.976 (2)	Y2—O2 × 2	2.378 (2)
Cu—O2 × 2	2.020 (2)	Y2—O3 × 1	2.277 (2)
Cu—O3 × 1	2.210 (2)		

Fig. 2. The room-temperature structure of Y_2BaCuO_5 viewed nearly along the c axis. The full and dashed lines indicate the edges of YO_7 trigonal prisms centered at $z = \frac{1}{4}$ and $\frac{3}{4}$, respectively. Note that some edges of the trigonal prisms are omitted and only one row of the trigonal prisms centered at $z = \frac{3}{4}$ is shown.

This is in contrast to the refinement of Michel & Raveau (1982) in which the isotropic temperature factors were significantly different for inequivalent Y atoms and inequivalent O atoms. Furthermore, our refinement yields full occupancies (within two standard deviations) for all sites.

The structure of Y_2BaCuO_5 consists of chains of edge-sharing YO_7 trigonal prisms running parallel to the b axis (Fig. 2). The chains on the sheets having approximately constant z values are cross linked by Ba and Cu atoms. Each Ba atom is coordinated to 5 O atoms forming a distorted CuO_5 tetragonal pyramid. Neighboring YO_7 chains on successive sheets are linked directly by edge sharing of the YO_7 trigonal prisms.

Note added in proof: During the refereeing process of this paper, we learned similar structural work on Y_2BaCuO_5

had also been done by Sato & Nakada (1989) using single-crystal X-ray diffraction, and by Hunter *et al.* (1990) using powder neutron diffraction. We found that our refined atomic coordinates agreed within e.s.d.'s with those by Sato & Nakada (1989) although our refined temperature factors were somewhat smaller.

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Structure of 3-amino-4*H*-1,2,4-benzothiadiazine 1,1-dioxide (1). Corrigendum.

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Abstract

The crystal structure of (1) was described as monoclinic, space group *Cc* with $a = 14.259$ (2), $b = 22.539$ (3), $c = 8.741$ (3) Å, $\beta = 114.10$ (2)°, $Z = 12$. [Bombieri, Demartin, Braghiroli, Todeschi & Di Bella (1989). *Acta Cryst.* C**45**, 1905–1908]. The application of the *MISSYM* program [Le Page (1987). *J. Appl. Cryst.* **20**, 264–269] has shown that it should rather have been described as rhombohedral, space group *R3c* with $a = 22.539$ (3), $c = 8.741$ (3) Å, $Z = 18$. The refinement carried out in the correct space group

converged to $R = 0.025$ and $wR = 0.030$ [686 independent reflections with $I > 2\sigma(I)$], without significant differences in the molecular parameters.

The vectors defining the new cell edges are [0·0, 1·0, 0·0], [1·5, −0·5, 1·0] and [0·0, 0·0, −1·0]. The asymmetric unit in the space group *R3c* is a single molecule with the refined coordinates reported in Table 1.*

The molecular parameters in the revised structure are not significantly different from those reported for the structure refined in the space group *Cc*; all the bond lengths and angles agree within three e.s.d.'s with the averaged values given in the previous determination (Bombieri *et al.*, 1989).

* Lists of crystallographic data, selected bond lengths and angles, H-atom coordinates, anisotropic temperature factors and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53000 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Table of positional parameters with e.s.d.'s in parentheses*

	x	*y*	*z*	$B(\text{Å}^2)$
S	0·26563 (3)	0·05361 (2)	0·412	3·06 (1)
O(1)	0·31674 (9)	0·09133 (8)	0·2963 (2)	4·36 (4)
O(2)	0·25317 (9)	0·09749 (8)	0·5107 (2)	4·48 (4)
N(2)	0·2886 (1)	0·00952 (9)	0·5076 (3)	3·98 (5)
N(3)	0·2791 (1)	−0·08904 (9)	0·5982 (3)	4·05 (5)
N(4)	0·19365 (8)	−0·09722 (8)	0·4429 (2)	3·05 (4)
C(3)	0·2536 (1)	−0·05799 (9)	0·5148 (3)	2·79 (4)
C(5)	0·0971 (1)	−0·1185 (1)	0·2855 (3)	3·75 (6)
C(6)	0·0646 (1)	−0·0942 (1)	0·1938 (4)	4·81 (7)
C(7)	0·0940 (2)	−0·0252 (1)	0·1619 (4)	5·88 (8)
C(8)	0·1566 (1)	0·0205 (1)	0·2266 (4)	5·31 (7)
C(9)	0·1605 (1)	−0·07321 (9)	0·3507 (2)	2·81 (4)
C(10)	0·1888 (1)	−0·0033 (1)	0·3220 (3)	3·28 (5)

Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as: $(4/3)[a^2 B(1,1) + b^2 B(2,2) + c^2 B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

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