

*Acta Cryst.* (1984). **C40**, 903–904

**Structure of diaqua-bis[ $\mu$ -(*N*-salicylidene-*L*-phenylalaninato)-*O,N,\mu-O'*]-dicopper(II),  $C_{32}H_{30}Cu_2N_2O_8$ : a reappraisal.** By J. E. DAVIES, *Crystallographic Data Centre, University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW, England*

(Received 29 June 1983; accepted 18 January 1984)

**Abstract**

$M_r = 697.7$ , trigonal,  $P3_121$ ,  $a = 12.724$  (3),  $c = 16.476$  (3) Å,  $U = 2310$  Å<sup>3</sup>,  $Z = 3$ ,  $D_x = 1.51$  Mg m<sup>-3</sup>,  $F(000) = 1074$ . This structure has been reported [Hämäläinen, Turpeinen, Ahlgrén & Rantala (1978). *Acta Chem. Scand. Ser. A*, **32**, 549–553] as monoclinic,  $C2$ . The original  $C2$  reflexion data have been transformed to a trigonal setting and to check the space-group assignment the structure has been refined successfully in space group  $P3_121$  ( $R = 0.138$  for 2017 reflexions). Bond lengths and angles in the  $P3_121$  structure do not differ significantly from those already reported for the  $C2$  structure.

**Introduction**

The X-ray structure of the title compound was reported by Hämäläinen, Turpeinen, Ahlgrén & Rantala (1978) as monoclinic, space group  $C2$ ,  $a = 22.053$ ,  $b = 12.715$ ,  $c = 16.476$  Å,  $\beta = 89.98^\circ$ ,  $Z = 12$  monomeric units. However, the  $c$  axis of this  $C2$  cell is also the  $c$  axis of a trigonal cell with dimensions  $a = 12.724$ ,  $c = 16.476$  Å. The volume of the trigonal cell is one half that of the  $C2$  cell and therefore  $Z = 6$  for the trigonal structure. A set of structure factors calculated using the published  $C2$  coordinates\* very clearly indicates that the Laue symmetry is  $\bar{3}m$ . Examination of atomic coordinates transformed to the trigonal setting indicates that the true space group is  $P3_121$ . Fractional atomic coordinates ( $X/A, Y/B, Z/C$ ) in  $P3_121$  are related to fractional atomic coordinates ( $x/a, y/b, z/c$ ) in  $C2$  by the equation

$$\begin{pmatrix} X/A \\ Y/B \\ Z/C \end{pmatrix} = \begin{pmatrix} 1 & 1 & 0 \\ 2 & 0 & 0 \\ 0 & 0 & -1 \end{pmatrix} \begin{pmatrix} x/a \\ y/b \\ z/c \end{pmatrix} + \begin{pmatrix} 0.1725 \\ 0.0 \\ -0.1667 \end{pmatrix}.$$

**Experimental**

A list of structure factors† [ $F_o > 2\sigma(F_o)$ ] was obtained from the authors (Hämäläinen, 1981). Standard deviations of individual structure factors were not used; unit weights were assumed in all calculations. The  $C2$  reflexion data (2901 reflexions) were transformed to the  $P3_121$  setting and symmetry equivalents were averaged to give 2017 unique reflexions (merging  $R_{int} = 0.083$ ). The original  $C2$  atom coordinates were transformed to the  $P3_121$  setting as described above. Coordinates of symmetry-related atoms in  $P3_121$  (22 sets, each of 3 atoms) were averaged to yield a set of coordinates for the asymmetric unit of the trigonal structure; structure refined on  $F$  by three cycles of full-matrix least squares with complex neutral-atom scattering factors (*International Tables for X-ray Crystallography*, 1974) and individual isotropic temperature factors for all atoms; H

\* See deposition footnote.

† Counter measurements, Mo  $K\alpha$  radiation,  $\theta_{max} = 25^\circ$ .

Table 1. *Diaqua-bis[ $\mu$ -(*N*-salicylidene-*L*-phenylalaninato)-*O,N,\mu-O'*]-dicopper(II) atomic coordinates and isotropic thermal parameters*

Atom labelling as in Hämäläinen *et al.* (1978).

	$x$	$y$	$z$	$U(\text{Å}^2)$
Cu	0.7984 (2)	0.9307 (2)	-0.2553 (1)	0.030 (1)
O(1)	0.746 (1)	0.876 (1)	-0.147 (1)	0.028 (3)
O(2)	0.845 (1)	0.978 (1)	-0.369 (1)	0.035 (3)
O(3)	0.771 (2)	0.972 (2)	-0.490 (1)	0.055 (4)
O(4)	0.954 (1)	0.934 (1)	-0.245 (1)	0.032 (3)
N	0.636 (1)	0.877 (1)	-0.297 (1)	0.025 (3)
C(1)	0.542 (2)	0.807 (2)	-0.262 (1)	0.038 (4)
C(2)	0.530 (2)	0.758 (2)	-0.176 (1)	0.031 (4)
C(3)	0.413 (2)	0.670 (2)	-0.148 (1)	0.027 (4)
C(4)	0.399 (2)	0.633 (2)	-0.067 (1)	0.045 (6)
C(5)	0.499 (2)	0.672 (2)	-0.018 (2)	0.049 (6)
C(6)	0.618 (2)	0.754 (2)	-0.044 (1)	0.038 (5)
C(7)	0.635 (2)	0.800 (2)	-0.125 (1)	0.036 (5)
C(8)	0.760 (2)	0.958 (2)	-0.417 (1)	0.020 (4)
C(9)	0.632 (2)	0.912 (2)	-0.381 (1)	0.033 (5)
C(10)	0.604 (2)	1.016 (2)	-0.388 (1)	0.038 (5)
C(11)	0.473 (2)	0.977 (2)	-0.370 (1)	0.031 (5)
C(12)	0.430 (2)	0.973 (2)	-0.291 (2)	0.050 (6)
C(13)	0.306 (3)	0.927 (3)	-0.277 (2)	0.079 (9)
C(14)	0.223 (3)	0.891 (3)	-0.344 (2)	0.078 (9)
C(15)	0.262 (3)	0.892 (3)	-0.419 (2)	0.063 (8)
C(16)	0.385 (3)	0.936 (3)	-0.433 (2)	0.065 (7)

atoms not included; refinement converged with  $R = 0.138$ ,  $(\Delta/\sigma)_{max} = 0.03$ ,  $\Delta\rho_{max} \pm 2e \text{ Å}^{-3}$  in the vicinity of the Cu atom. Final coordinates are in Table 1; \* all calculations performed with *SHELX76* (Sheldrick, 1976).

**Discussion**

The structure is illustrated in Fig. 1.† There are no significant differences between bond lengths and angles in the  $C2$  and  $P3_121$  structures. The original description of the coordination geometry (Hämäläinen *et al.*, 1978) is correct, *i.e.* 'the tridentate *N*-salicylidene-*L*-phenylalanine group occupies three of the square-planar coordination sites, with the oxygen atom of the water molecule completing the plane. The fifth coordinated atom, the salicylidene oxygen of the monomeric partner, lies at the apex of the [square] pyramid.'

In  $P3_121$ , the two halves of the dimeric molecule are related by a diad axis which is approximately parallel to the two Cu–O(4) bonds, where O(4) is the coordinated water oxygen atom (see Fig. 1). All O(4) atoms are close (1.02 Å)

\* Structure factors, bond lengths and angles and diagrams showing the weighted  $hk0$  reciprocal-lattice section calculated with the  $C2$  atomic coordinates, and aspects of the crystal and molecular structure have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39203 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

† Other diagrams have been deposited.

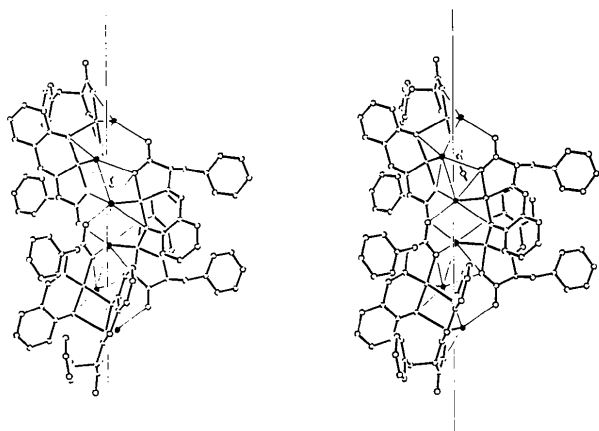


Fig. 1. Stereoscopic diagram of three molecules viewed perpendicular to the triad ( $c$ ) axis (also illustrated). The water oxygen atoms [O(4)] are shaded and all O( $n$ )-O(4) ( $n = 1,2,3$ ) distances less than  $3 \cdot 10 \text{ \AA}$  are indicated with fine lines.

to screw triad axes of the type  $0,0,z$  and there is an extensive hydrogen-bonded network continuously spiralling along these screw triad axes (see Fig. 1). There are six O(4)-O( $n$ ) ( $n = 1,2,3$ ) distances less than  $3 \cdot 10 \text{ \AA}$ ;\* each of these contacts may be associated with a hydrogen bond.

I thank Professor R. Hämäläinen for very kindly providing me with a list of the original  $C2$  structure factors. Figures were drawn with *ORTEP* (Johnson, 1965).

\* Values have been deposited.

#### References

- HÄMÄLÄINEN, R. (1981). Private communication.  
 HÄMÄLÄINEN, R., TURPEINEN, U., AHLGRÉN, M. & RANTALA, M. (1978). *Acta Chem. Scand. Ser. A*, **32**, 549-553.  
*International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press.  
 JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Oak Ridge, Tennessee.  
 SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.