Acta Cryst. (1966). **20**, 824

The Crystal Structure of Cu(II) Succinate Dihydrate

By B. H. O'Connor* and E. N. Maslen,

Department of Physics, University of Western Australia, Nedlands, Western Australia

(*Received* 15 *September* 1965)

The crystal structure of copper(II) succinate dihydrate $Cu(OOC.CH_2)_2.2H_2O$, has been established by vector methods with three-dimensional single-crystal X-ray data. The atomic parameters were refined by the diagonal least-squares approximation, the final R index being 0·095. The crystal structure has $P\bar{1}$ space-group symmetry and the coordinates are defined most suitably relative to a cell of dimensions:

$$a=6\cdot437\pm0\cdot011,\ b=7\cdot623\pm0\cdot004,\ c=8\cdot081\pm0\cdot005\ \text{Å},$$
$$\alpha=103^\circ 54'\pm8',\ \beta=73^\circ 30'\pm21',\ \gamma=98^\circ 37'\pm13'.$$

The structure consists of infinite chains of covalently bonded binuclear units each of which closely resembles the copper(II) acetate monohydrate configuration. The Cu–Cu distance of 2·610 (1) Å confirms that there is magnetic exchange between the two copper atoms in each binuclear unit.

Introduction

Cu^{II}, with one unpaired spin per ion, has a theoretical spin-only magnetic moment of 1·73 BM. The value measured experimentally is usually in the vicinity of 1·8 BM owing to quenched spin-orbit coupling. However, compounds of divalent copper, in which there is magnetic coupling between the spins of pairs of copper atoms, have moments in the range 1·1–1·6 BM. The properties of these complexes have recently been reviewed by Figgis & Lewis (1964) and Kato, Jonassen & Fanning (1964).

The magnetic moments of the copper(II) salts of the α,ω-dicarboxylic acids ($HOOC–[CH_2]_n–COOH$, $n=0, 1, \cdots, 8$) have been measured at room temperature by Asai, Kishita & Kubo (1959). All members of the series examined, with the exception of the malonate ($n=1$), gave anomalous values for the magnetic moment. Copper(II) succinate dihydrate has a magnetic moment of 1·36 BM at 300°K. The steric requirements of the succinate group oppose the formation of a monomeric or dimeric molecule on energetic grounds. Asai and his colleagues proposed a model consisting of binuclear units which are linked covalently to form a two-dimensional polymeric structure. The current analysis has shown that the crystals are composed of infinite linear chains of covalently bonded binuclear units each of which closely resembles the copper(II) acetate monohydrate (van Niekerk & Schoening, 1953) configuration.

Experimental

Single crystals were kindly supplied by Dr B. N. Figgis of the Department of Chemistry, University of Western Australia. The material had been prepared by slowly introducing urea into a solution of succinic acid and copper sulphate. The crystals had a pinacoidal needle habit and cleaved easily along the needle axis into fibres. The material was practically insoluble in all common solvents. A stoichiometric analysis gave values of 29·43, 22·43 and 3·98% for the composition by weight for copper, carbon and hydrogen respectively, compared with calculated values of 29·47, 22·27 and 3·74% for the empirical formula $Cu(OOC.CH_2)_2.2H_2O$.

The needle axis was selected as the direction of **a**. Oscillation and equi-inclination Weissenberg exposures with this as rotation axis indicated that a primitive triclinic cell could be defined with $a=6\cdot4$ Å, $b^*=0\cdot136$ Å^{-1}, $c^*=0\cdot132$ Å^{-1} and $\alpha^*=78°$. Measurement of the ($h0l$) and ($hk0$) Weissenberg patterns gave approximate values for the remainder of the real and reciprocal cell parameters. Accurate values were derived by the back-reflexion method, the initial values being continually improved by applying the extrapolation method of Farquhar & Lipson (1946) to the triangulation technique of Buerger (1953). In order to select the correct triclinic space group symmetry, the zero moment test of Howells, Phillips & Rogers (1950) was applied. The calculated distribution functions were modified according to Sim's (1958) criterion for heavy atom structures. The experimental function was derived using the 1575 recorded reflexions (see below), and comparison with the calculated functions showed quite conclusively that the space group is $P\bar{1}$.

The Delaunay (1933) reduction was carried out in order to derive the reduced cell, *viz.* the cell which satisfies the conditions:

(i) $\alpha, \beta, \gamma > 90°$,
(ii) $c < a < b$ (Donnay & Nowacki, 1953),
and (iii) direction cosines of $\langle 111 \rangle \geq 0$.

The transformation which transposes the cell selected initially into the Delaunay cell is given by

---

* Present address: Metallurgy Division, United Kingdom Atomic Energy Research Establishment, Harwell, Berkshire, England.

$$\begin{bmatrix}\mathbf{a}_D\\ \mathbf{b}_D\\ \mathbf{c}_D\end{bmatrix} = \begin{bmatrix}001\\ 011\\ 10\bar{1}\end{bmatrix} \begin{bmatrix}\mathbf{a}\\ \mathbf{b}\\ \mathbf{c}\end{bmatrix}$$

It can be seen from the list of crystal data that the Delaunay cell is considerably less compact than the 'unreduced' cell. Since the least-squares refinement was to be conducted with the diagonal matrix approximation the parameters were expressed relative to the latter cell in keeping with the condition of the approximation that the cell axes should be close to orthogonal. The unreduced cell is subsequently referred to as the compact cell.

The reflexion intensities for Cu $K\alpha$ radiation were recorded by the equi-inclination Weissenberg method and were estimated by visual comparison. All layers corresponding to $h \le 6$ were collected from a crystal of dimensions $(0{\cdot}06 \times 0{\cdot}03 \times 0{\cdot}46)$ mm^3 mounted along the **a** direction. Correlation data were collected from a smaller crystal $(0{\cdot}15 \times 0{\cdot}01 \times 0{\cdot}05)$ mm^3, the third dimension corresponding to the extension of the crystal along the direction of **c**, which was selected as rotation axis. Absorption corrections were negligible compared with measurement errors for both samples. The final set of amplitudes comprised 1575 independent reflexions, of which 285 were indistinguishable from the film background.

Crystal data

$Cu(COO)_2(CH_2)_2 . 2H_2O$, copper(II) succinate dihydrate

F.W. 215·67

Space group, $P\bar{1}$ (triclinic)

$Z=2$

Cell parameters (with maximum possible errors):

Compact cell	$a=6{\cdot}437 \pm 0{\cdot}011$ Å	$\alpha=103°54' \pm\ 8'$
	$b=7{\cdot}623 \pm 0{\cdot}004$	$\beta=\ 73\ \ 30\ \pm 21$
	$c=8{\cdot}081 \pm 0{\cdot}005$	$\gamma=\ 98\ \ 37\ \pm 13$
Delaunay cell	$a=8{\cdot}081 \pm 0{\cdot}005$	$\alpha=\ 97\ \ 55\ \pm 17$
	$b=9{\cdot}685 \pm 0{\cdot}015$	$\beta=128\ \ 36\ \pm 13$
	$c=6{\cdot}294 \pm 0{\cdot}012$	$\gamma=\ 96\ \ 50\ \pm 14$

$U=367{\cdot}6$ Å^3

$D_m=1{\cdot}95 \pm 0{\cdot}02$ g.cm^{-3} (by flotation), $D_x=1{\cdot}945$

$\mu=42$ cm^{-1} for $\lambda=1{\cdot}5418$.

Structure solution

All non-hydrogen atoms were located by vector methods. A three-dimensional sharpened Patterson synthesis was computed with coefficients of the form $M(\theta)|F|^2$ where

$$M(\theta)=\exp\,(2\bar{B}\sin^2\theta/\lambda^2)/(\hat{f})^2$$

where $\bar{B}$ is the mean isotropic temperature factor coefficient for the structure and $\hat{f}$ is the mean unitary scattering factor. A trial value of 3·0 Å^2 was selected for $\bar{B}$. The copper–copper vector was prominent and corresponded to real-space coordinates (0·077, 0·150, 0·458) relative to the compact cell compared with the refined values (0·0790, 0·1551, 0·4559). The carbon and oxygen atoms were easily identified by evaluating the Buerger (1951) minimum function which is shown in Fig. 1. A preliminary set of structure factors was then computed for the model using the form factors listed in *International Tables for X-ray Crystallography* (1962). A plot of $ln(|F_o|/|F_c|)$ *versus* $\sin^2\theta/\lambda^2$ provided a $\bar{B}$ value of 1·4 Å^2 and a scale factor K, defined according to $KF_o=F_c\exp(-\bar{B}\sin^2\theta/\lambda^2)$. On applying B and K an R index† of 0·34 resulted. Since the agreement was promising for low order reflexions, least-squares parameter refinement was then commenced.

Parameter refinement

The parameters were refined by the block-diagonal approximation with Mair's (1962) program SFLS for the IBM 1620 computer. The program minimizes $\Sigma\, w(|F_o|-|F_c|)^2$.

Three different weighting schemes were employed during the analysis:

$$\begin{aligned}\sqrt{w}&=|F_o|/F^*,\ |F_o| \le F^*\\ &=F^*/|F_o|,\ |F_o| > F^* \qquad \text{(scheme 1)}\end{aligned}$$

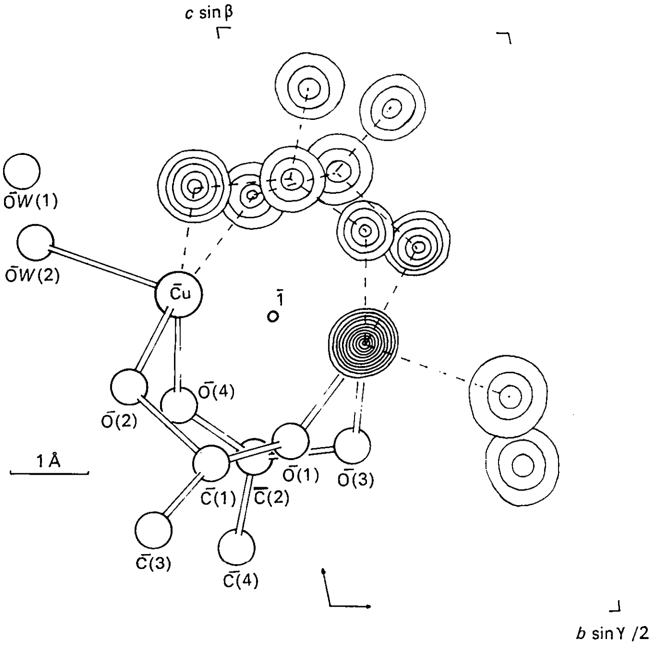


Fig. 1. Summary of the Buerger minimum function viewed down the a axis of the compact unit cell. Contours at arbitrary but equal intervals. The ball and spoke model completes the binuclear unit of the infinite chain.

---

† The R index calculated was

$$\frac{\Sigma\,||F_o|-|F_c||}{\Sigma\,|F_o|}\,,$$

each observed reflection being given unit weight. Unobservably weak reflexions were included only where $|F_c|$ exceeded the observable limit, and were assigned an $|F_o|$ value equal to the limit.

$$\sqrt{w}=1/[1+\{(|F_o|-b)/a\}^2]^{\frac{1}{2}} \qquad \text{(scheme 2)}$$

and

$$\sqrt{w}=1,\ |F_o|\le F^* \qquad\qquad$$
$$=F^*/|F_o|,\ |F_o|>F^*\ . \qquad \text{(scheme 3)}$$

Unobservably weak reflexions were given zero weight in the refinement. The scattering factors used were those given by Hanson, Harman, Lea & Skillman (1964) for wave functions derived from Slater's modification of the Hartree-Fock SCF method. Since these functions neglect anomalous dispersion effects, it was necessary to modify the copper scattering factor for the real component of the dispersion correction for Cu $K\alpha$ radiation, *i.e.* $-2{\cdot}1$ electrons (Dauben & Templeton, 1955). Dispersion corrections were negligible for the other atomic species. The standard deviations in the parameters were estimated in the usual manner from the inverse to the normal equations matrix.

The analysis is best considered in three stages – the initial refinement of non-hydrogen atoms, the location and refinement of hydrogen atoms and the final refinement of non-hydrogen atoms.

Stage 1

Three positional and six anisotropic thermal parameters per non-hydrogen atom and the scale factor K were refined through nine cycles. For the first five rounds of calculation scheme (1) was employed with an F^* value of 8 electrons and in rounds six to nine scheme (2) was used. The constants a and b for scheme (2) were derived in the manner described by Rietveld (1964). After two cycles R had fallen from 0·34 to 0·178, and thereafter fell steadily to 0·122. At this stage the positional parameter shifts were less than 0·5 of the corresponding parameter e.s.d. However, although the shifts for the thermal coefficients B_{11}, B_{22} and B_{33} were also small, a plot of $\ln(|F_o|/|F_c|)$ *versus* $\sin^2\theta/\lambda^2$ gave a $\Delta\bar{B}$ value of 0·2 Å^2 and a K of 0·985. Application of these corrections reduced R to 0·106. A systematic error of this nature had not been expected as Mair's program applies the scale to the $|F_o|$ – rather than $|F_c|$ – set which, according to Geller & Durand (1960), should lead to a scale factor which is too low. A close examination of the factors influencing the data was made but no satisfactory explanation has been found.

Stage 2

The eight crystallographically independent hydrogen atoms were located from the $(\varrho_o-\varrho_c)$ synthesis, ϱ_c being derived for the non-hydrogen atoms. The methylene hydrogen atoms were clearly identified as well-shaped volumes of peak density about 0·6 e.Å^{-2}. Peaks of similar height were also observed in the vicinity of the water oxygen atoms although their interpretation was slightly ambiguous. One of the three peaks adjacent to OW(2) was close to the Cu–OW(2) bond and was therefore eliminated on chemical grounds. There were three regions of positive density adjacent to OW(1) and all three had to be considered as possible hydrogen atom sites. Inclusion of the nine hydrogen atoms in a structure factor refinement computation lowered the R index to 0·100. A relaxation factor of 0·25 was used to ensure convergence of the positional shifts. The hydrogen atoms were assigned the mean isotropic thermal parameter of the atom to which they are covalently bonded. A close study was made of the least-squares shifts for the hydrogen thermal parameters. However the shifts did not appear to be physically meaningful except in the case of one 'atom' for which the temperature factor diverged. It was concluded that this corresponded to an erroneous hydrogen position. A summary of the final difference synthesis, based on F_c values which do not include the effect of hydrogen atoms, is shown in Fig. 6. The regions of positive density in the vicinity of the hydrogen are well-shaped. Moreover the proposed positions for the water hydrogen atoms are consistent with the hydrogen bond system which is described below.

Stage 3

Refinement of the non-hydrogen atoms was continued in order to correct the B_{ij}'s for interactions with the hydrogen positional contributions during the initial refinement. Scheme (3) was applied with $F^*=16$ electrons. The R index dropped from 0·0991 to 0·0979 after the first cycle. Three more cycles reduced R to 0·0971 and the refinement was then terminated. At this stage the positional and B_{ij} shifts were less than 0·1 of the corresponding e.s.d. For observed terms only, R was 0·095.

A list of the final structure factors is given in Table 7. A summary of the final ϱ_o – synthesis is shown in Fig. 2.

Final structural model

The set of atomic coordinates for the compact cell and their standard deviations are listed in Table 1. The coordinates are also given relative to the Delaunay cell. The molecular configuration is shown in Figs. 1 and 3. The atoms form binuclear units which are linked by C–C bonds into linear polymeric chains. Each binuclear unit has a conformation which closely resembles the copper(II) acetate monohydrate (van Niekerk & Schoening, 1953) configuration.

The thermal parameters and their standard deviations are given in Table 2.

Discussion

The interatomic distances and associated angles are shown in Fig. 4, which presents a view of the binuclear unit down the **b** direction of the compact cell. The mean bond lengths are listed in Table 3 and compared with those in related Cu(II) carboxylate complexes.

Copper coordination

The coordination number of the copper atom is six. There are five oxygen ligands per copper, four of these being succinate oxygens and the fifth a water oxygen. The sixth ligand is the centrosymmetrically related copper atom. The coordination geometry therefore closely resembles that found in copper(II) acetate monohydrate, orthorhombic monopyridine copper(II) acetate (Hanic, Štempelová & Hanicová, 1964) and monoclinic monopyridine copper(II) acetate (Barclay & Kennard, 1961). The Cu–Cu distance of 2·610(1) Å, although significantly shorter than the length of 2·64 Å in copper(II) acetate monohydrate, still constitutes a very weak bond and in both complexes stability is conferred on the binuclear unit by four bridging carboxyl chains which link the copper atoms. Each copper atom is displaced by 0·197 Å from the equatorial plane of the octahedron (Table 4) towards the water oxygen. There are two hybridization schemes which are consistent with the coordination geometry. The first of these comprises four vacant $4(dsp^2)$ hybrids and a

Table 1. *Fractional atomic coordinates*

	Compact cell			Delaunay cell		
	x/a	*y/b*	*z/c*	*x/a*	*y/b*	*z/c*
Cu	0·07896 (9)	0·15506 (7)	0·45592 (7)	0·37982	0·15506	0·07896
O(1)	−0·3049 (5)	−0·0018 (4)	0·7023 (4)	0·3992	−0·0018	−0·3049
O(2)	−0·1763 (5)	0·2655 (4)	0·6332 (4)	0·1914	0·2655	−0·1763
O(3)	0·0958 (5)	−0·0805 (4)	0·7186 (4)	0·8949	−0·0805	0·0958
O(4)	0·2179 (5)	0·1887 (4)	0·6514 (4)	0·6806	0·1887	0·2179
C(1)	−0·3146 (6)	0·1638 (5)	0·7240 (5)	0·2456	0·1638	−0·3146
C(2)	0·1980 (6)	0·0741 (5)	0·7432 (5)	0·8671	0·0741	0·1980
C(3)	−0·4958 (7)	0·2499 (5)	0·8669 (5)	0·1212	0·2499	−0·4958
C(4)	0·2974 (7)	0·1258 (6)	0·8972 (5)	0·0688	0·1258	0·2974
O*W*(1)	−0·2951 (7)	0·3879 (5)	0·2468 (6)	0·5638	0·3879	−0·2951
O*W*(2)	0·2522 (5)	0·3838 (4)	0·3630 (5)	0·2314	0·3838	0·2522
H(31)	−0·513	0·357	0·881	0·011	0·357	−0·513
H(32)	−0·471	0·269	0·985	0·245	0·269	−0·471
H(41)	0·308	0·024	0·919	0·203	0·024	0·308
H(42)	0·205	0·208	0·981	−0·022	0·208	0·205
HW(11)	−0·221	0·500	0·267	0·546	0·500	−0·221
HW(12)	−0·276	0·306	0·333	0·751	0·306	−0·276
HW(21)	0·365	0·394	0·333	0·304	0·394	0·365
HW(22)	0·267	0·539	0·363	0·091	0·539	0·267

Table 2. *Anisotropic thermal parameters* referred to the compact cell*

	B_{11}	B_{22}	B_{33}	B_{23}	B_{13}	B_{12}
Cu	0·01360 (14)	0·00792 (9)	0·00935 (9)	0·00565 (15)	−0·00506 (19)	0·00342 (18)
O(1)	0·0166 (8)	0·0102 (5)	0·0140 (6)	0·0048 (9)	0·0045 (11)	0·0068 (10)
O(2)	0·0153 (8)	0·0088 (5)	0·0137 (6)	0·0049 (8)	0·0003 (11)	0·0040 (10)
O(3)	0·0194 (9)	0·0129 (6)	0·0135 (6)	0·0076 (8)	−0·0138 (11)	0·0011 (11)
O(4)	0·0236 (10)	0·0132 (6)	0·0126 (6)	0·0073 (9)	−0·0139 (12)	−0·0009 (12)
C(1)	0·0141 (10)	0·0115 (7)	0·0074 (6)	0·0046 (11)	−0·0110 (13)	0·0033 (13)
C(2)	0·0110 (10)	0·0104 (7)	0·0108 (7)	0·0038 (11)	−0·0032 (13)	0·0055 (13)
C(3)	0·0189 (12)	0·0085 (7)	0·0102 (7)	0·0003 (11)	−0·0086 (15)	0·0050 (13)
C(4)	0·0170 (12)	0·0142 (8)	0·0088 (8)	0·0026 (12)	−0·0053 (14)	0·0092 (16)
O*W*(1)	0·0334 (13)	0·0147 (7)	0·0277 (10)	0·0044 (13)	−0·0276 (19)	0·0083 (16)
O*W*(2)	0·0183 (9)	0·0115 (6)	0·0258 (9)	0·0188 (12)	−0·0010 (14)	0·0042 (12)

* Defined so that the atomic temperature factor is $\exp\{-(B_{11}h^2+B_{22}k^2+B_{33}l^2+B_{23}kl+B_{13}hl+B_{12}hk)\}$.

Table 3. *Mean covalent bond distances in* Cu(II) *succinate dihydrate and related salts*

	Cu(II) succinate dihydrate (Present analysis)	Cu(II) acetate monohydrate (van Niekerk & Schoening, 1953)	Orthorhombic monopyridine Cu(II) acetate (Hanic *et al.*, 1964)	Monoclinic monopyridine Cu(II) acetate (Barclay & Kennard, 1961)
Cu–Cu	2·610 (1) Å	2·64 Å	2·645 (3) Å	2·630 (3) Å
Cu–H_2O	2·102 (7)	2·20	2·186 (8)*	2·125 (7)*
Cu–O	1·975 (3)	1·97	1·955 (4)	1·981 (5)
C(sp^2)–O	1·562 (5)	1·33	1·246 (10)	1·239 (15)
C(sp^2)–C(sp^3)	1·497 (10)	1·52	1·537 (15)	1·503 (17)
C(sp^3)–C(sp^3)	1·508 (10)	—	—	—

* Cu–N bond.

vacant $4p_z$ orbital which accepts electron density from the water oxygen ligand. This is consistent with there being four equivalent Cu–O bonds of mean length 1·975(3) Å and a longer Cu–O*W* bond of length 2·102(7) Å. The displacement of the copper atom from the equatorial plane of the octahedron can be ascribed to repulsion between the filled $3d_{z^2}$, $3d_{xz}$ and $3d_{yz}$* orbitals of the copper pair (Figgis & Martin, 1956). The alternative hybridization scheme is a distorted $4(dsp^3)$ hybrid arrangement.

Figgis & Martin point out that an arrangement of four approximately planar Cu–O bonds will allow lateral overlap between the $3d_{x^2-y^2}$ orbitals to form a Cu–Cu δ-bond which, they contend, is responsible for magnetic coupling between the copper atoms. As the Cu–Cu distance of 2·610(1) Å is significantly shorter than the distance of 2·64 Å in copper(II) acetate monohydrate the succinate salts should therefore have a lower

---

* The *z* axis being defined along the Cu–Cu vector.

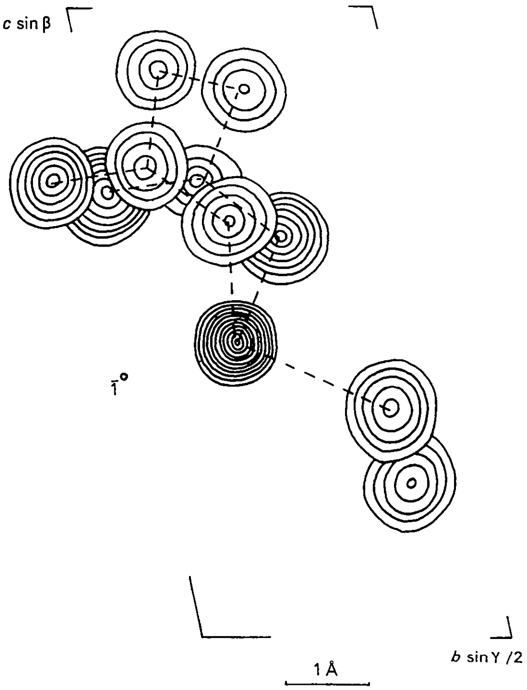


Fig. 2. Summary of the final ϱ_o synthesis viewed down the *a* axis of the compact cell. Contour intervals: 2 e.Å^{-3} for carbon and oxygen; 2·5 e.Å^{-3} for copper. First contour at 1 e.Å^{-3} for carbon and oxygen, 4 e.Å^{-3} for copper.

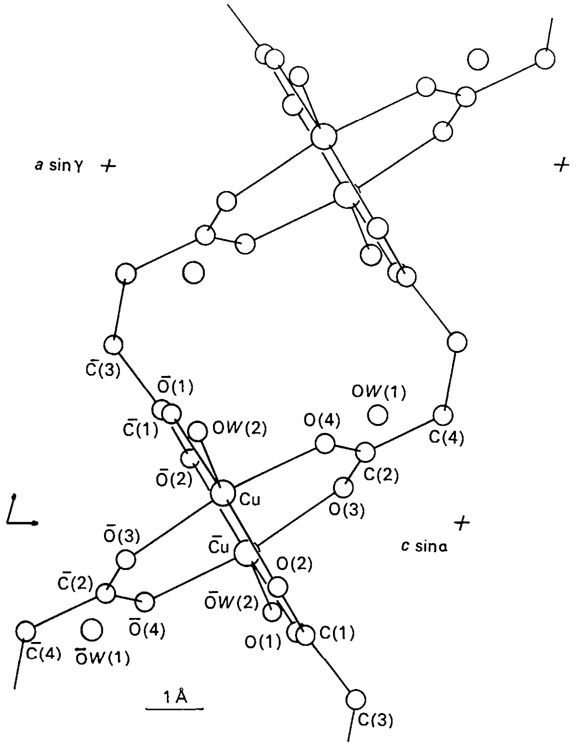


Fig. 3. Configuration viewed down the *b* axis of the compact cell.

Table 4. *Least-squares planes*

The coefficients *A*, *B*, *C* and *D* are defined so that $Ax+By+Cz+D=0$. The coefficient errors are maximum possible estimates. In the list of displacements from each plane the most significant figures of the positional standard deviations are enclosed in parentheses. Atoms listed below each broken line were excluded from the particular plane calculation.

Coefficients		Displacements		
A	0·7097 (14)	Cu	−0·0031 (6) Å	
B	0·0551 (8)	O(1)	−0·0038 (32)	
C	0·7024 (28)	O(2)	0·0037 (31)	
D	−3·431 (14)	C̄u	0·0032 (6)	
		C(1)	−0·0353 (41)	
		C(3)	0·1541 (43)	
A	0·7581 (29)	O(1)	0·0023 (32) Å	
B	0·0307 (28)	O(2)	0·0023 (31)	
C	0·6514 (60)	C(1)	−0·0063 (41)	
D	−3·165 (36)	C(3)	0·0018 (43)	
A	0·6959 (97)	Cu	0·0227 (6) Å	Cu–O–C–O–C̄u chains
B	−0·2876 (85)	O(3)	0·0277 (33)	
C	−0·658 (19)	O(4)	−0·0265 (33)	
D	1·462 (92)	C̄u	−0·0239 (6)	
		C(2)	−0·0445 (42)	
		C(4)	−0·2028 (46)	
A	0·699 (40)	O(3)	−0·0297 (33) Å	(COO)CH_2 groups
B	0·323 (95)	O(4)	0·0330 (33)	
C	−0·637 (48)	C(2)	0·0662 (42)	
D	1·42 (20)	C(4)	−0·0695 (46)	
A	0·2265 (2)	Ō(1)	0·0007 (32) Å	Equatorial ligand plane
B	0·9287 (19)	O(2)	−0·0007 (31)	
C	−0·2938 (3)	Ō(3)	−0·0007 (33)	
D	0·4988 (13)	O(4)	0·0007 (33)	
		C̄u	0·1973 (6)	

magnetic moment than the acetate. This is indeed the case. At 300·2°K the values are 1·36 BM (Figgis & Martin, 1965) and 1·42 BM (Figgis & Martin, 1956), respectively. The anomalous magnetic moment of the binuclear copper(II) carboxylates has also been attributed to exchange *via* interacting copper $3d_{z^2}$ orbitals (Forster & Ballhausen, 1962), *i.e.* σ-interaction. Since the shorter Cu–Cu distance in copper(II) succinate dihydrate will give rise to enhanced strength of both the δ- and σ-bond it is not possible to decide on the basis of this study whether magnetic exchange in this class of compounds is due to δ- or σ-exchange.

Succinate groups

The predicted carboxyl ion configuration has an O–C–O angle of 125·25° and two C–O bond distances of 1·27 Å (Pauling, 1962). Sutton (1958) lists the dimensions of the formate ion as 125·5° ± 1° and 1·25 ± 0·01 Å. The mean dimensions of 124·2(6)° and 1·256 (5) Å observed in copper(II) succinate dihydrate are in excellent agreement with these values. There is also conformity with the C–O distances given in Table 3 for related structures except for the value of 1·33 Å in copper(II) acetate monohydrate, which is of doubtful accuracy as the analysis of van Niekerk & Schoening was a two-dimensional study with final *R* values of 0·21 and 0·20.

Conformations of the α,ω-dicarboxylates of copper(II)

It is possible on the basis of the present study to predict structures for other copper(II)-α,ω-dicarboxylates:

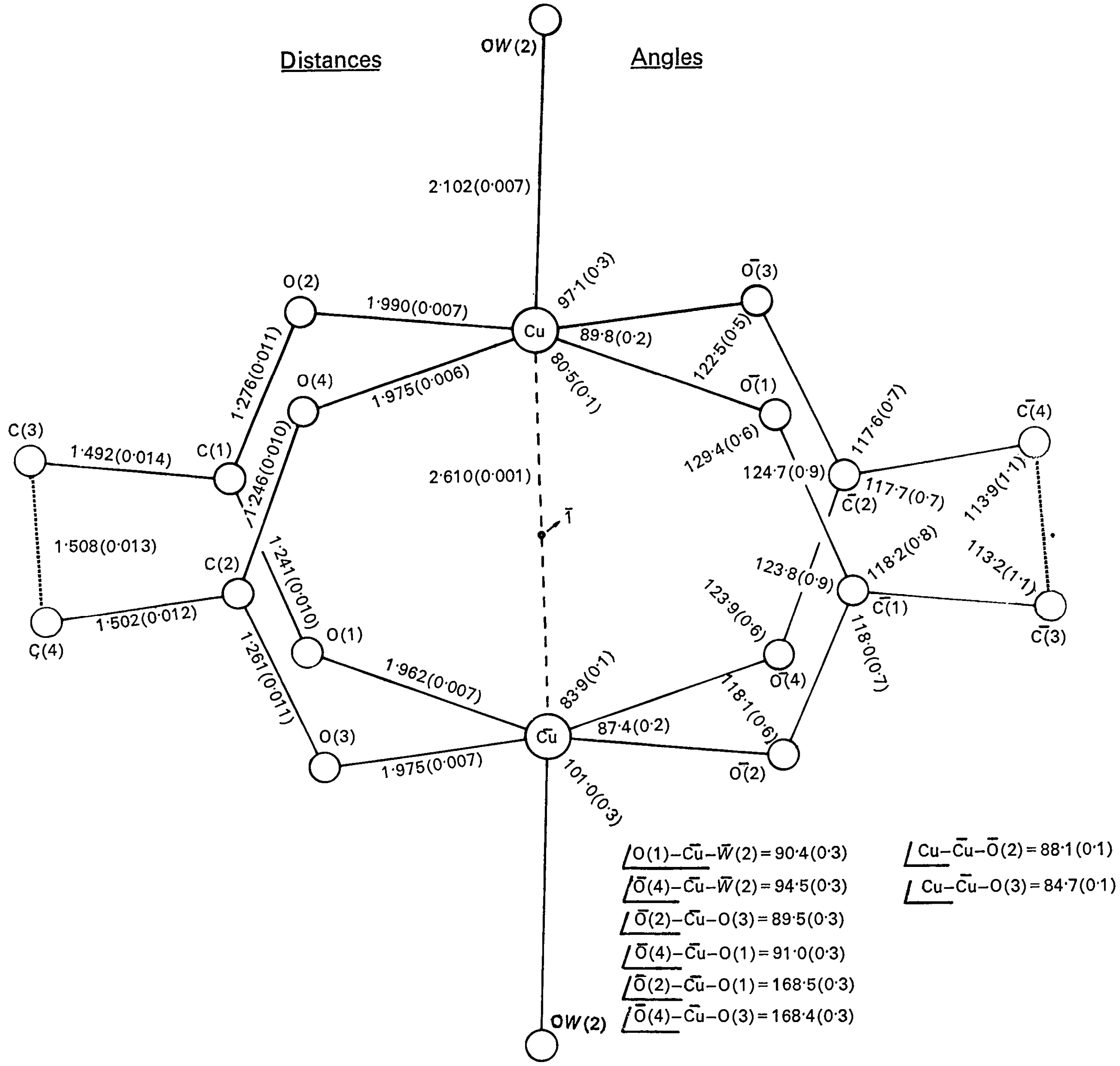
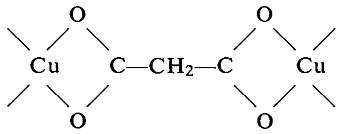


Fig. 4. Bond lengths (Å) and angles (°).

(i) Copper(II) oxalate, $\mathrm{Cu(COO)_2}$, has a magnetic moment of 1·20 BM at 288°K (Asai, Kishita & Kubo, 1959). There is therefore exchange between the copper atoms and it is probable that the structure consists of linear polymeric chains of binuclear units.

(ii) The 'normal' moment of the malonate, $\mathrm{Cu(COO)_2CH_2}$, indicates that the structure comprises linear polymeric chains of the form



(iii) Copper(II) glutarate, $\mathrm{Cu(COO)_2(CH_2)_3}$, which has an anomalous magnetic moment, probably has a structure resembling that of copper(II) succinate dihydrate. Single crystals of the two salts have the same morphology and cleave in a similar manner. An oscillation photograph of the glutarate showed that there is a repeat distance of 7·9 Å along the needle axis. This exceeds the corresponding distance in the succinate complex by 1·5 Å, the difference presumably being due to the additional C–C bond in the glutarate lying approximately parallel to the needle axis.

Hydrogen bond network

The interchain distances are tabulated in Table 5. Three of these distances represent O–H · · · O bonds. The hydrogen bond network is shown in Fig. 5 together with the important distances and angles associated with the donor and acceptor atoms. The O · · · O distances, 2·78Å [$\bar{\mathrm{O}}\bar{W}(2^{\mathrm{III}})\cdots\mathrm{O}(2^{\mathrm{I}})$] and 2·79Å [$\mathrm{O}W(1^{\mathrm{II}})\cdots\mathrm{OW}(2^{\mathrm{I}})$], conform with the length of the O–D · · · O bond (2·76 Å) in deuterated ice (Peterson & Levy, 1957). The third bond, $\bar{\mathrm{O}}(3^{\mathrm{I}})\cdots\mathrm{O}W(1^{\mathrm{I}})$ (2·94 Å), is considerably weaker than the other two. The angles subtended by the appropriate interatomic vectors confirm that all three contacts are hydrogen bonds. The carboxylate oxygen atom can be described as sp^2 and consequently a hydrogen bond directed towards the oxygen should be inclined at 120° to the adjacent Cu–O and C–O bonds. The angles $\mathrm{Cu^{I}}\text{–}\bar{\mathrm{O}}(3^{\mathrm{I}})\cdots\mathrm{O}W(1^{\mathrm{I}})$ and $\bar{\mathrm{C}}(2^{\mathrm{I}})\text{–}\mathrm{O}(3^{\mathrm{I}})\cdots\mathrm{O}W(1^{\mathrm{I}})$ are 111·7° and 117·3°, respectively, and are therefore fairly consistent with hydrogen bonding. The angle $\bar{\mathrm{O}}(3^{\mathrm{I}})\cdots\mathrm{O}W(1^{\mathrm{I}})\cdots\mathrm{O}W(2^{\mathrm{II}})$ of 117·4° lies reasonably close to the expected value of 109·5°. The angles subtended at $\mathrm{O}W(2)$ (103·2°, 117·6° and 134·9°) give further confirmation of the proposed hydrogen bond system, but, in view of the directional properties of lone pair orbitals, it is not possible to decide, on this basis alone, which atom acts as donor in the $\mathrm{O}W(2^{\mathrm{II}})\cdots\mathrm{O}W(1^{\mathrm{I}})$ bond. However, inspection of the $\varrho_o\text{–}\varrho_c$ synthesis in Fig. 6 shows clearly that, while $\mathrm{H}W(11)$ is not situated near a hydrogen bond vector, $\mathrm{H}W(12)$, $\mathrm{H}W(21)$ and $\mathrm{H}W(22)$ form hydrogen bonds. Hence both hydrogen atoms of the ligand water molecule participate in hydrogen bonding.

Table 5. *Interchain contacts less than* 4·0 Å

The equivalent positions refer to the compact cell. The 'bar' notation denotes the centric equivalent of an atom through the symmetry centre (0, 0, ½).

Equivalent positions	Atoms	Distance
I x, y, z))	$\bar{\mathrm{O}}\bar{W}(1^{\mathrm{III}})\text{–}\mathrm{O}(2^{\mathrm{I}})$	3·76 Å
II (1+x, y, z)		
III (x, 1+y, z)	$\bar{\mathrm{O}}\bar{W}(2^{\mathrm{III}})\text{–}\mathrm{O}(2^{\mathrm{I}})$	2·78*
IV (x, y, 1+z)		
V (−1+x, 1+y, z)	$\bar{\mathrm{C}}(4^{\mathrm{IV}})\text{——}\mathrm{O}(3^{\mathrm{I}})$	3·46
	$\bar{\mathrm{O}}\bar{W}(1^{\mathrm{V}})\text{–}\mathrm{C}(3^{\mathrm{I}})$	3·54
	$\mathrm{O}W(2^{\mathrm{III}})\text{–}\mathrm{C}(3^{\mathrm{I}})$	3·64
	$\bar{\mathrm{O}}(1^{\mathrm{IV}})\text{——}\mathrm{C}(4^{\mathrm{I}})$	3·60
	$\mathrm{O}W(2^{\mathrm{IV}})\text{–}\mathrm{C}(4^{\mathrm{I}})$	3·76
	$\bar{\mathrm{O}}(1^{\mathrm{V}})\text{——}\mathrm{O}W(1^{\mathrm{I}})$	3·63
	$\bar{\mathrm{O}}(3^{\mathrm{I}})\text{——}\mathrm{O}W(1^{\mathrm{I}})$	2·94*
	$\bar{\mathrm{C}}(2^{\mathrm{I}})\text{——}\mathrm{O}W(1^{\mathrm{I}})$	3·69
	$\bar{\mathrm{C}}(4^{\mathrm{I}})\text{——}\mathrm{O}W(1^{\mathrm{I}})$	3·81
	$\bar{\mathrm{C}}(1^{\mathrm{III}})\text{——}\mathrm{O}W(2^{\mathrm{I}})$	3·63
	$\bar{\mathrm{O}}\bar{W}(1^{\mathrm{III}})\text{–}\mathrm{O}W(2^{\mathrm{I}})$	3·90
	$\mathrm{O}W(1^{\mathrm{II}})\text{–}\mathrm{O}W(2^{\mathrm{I}})$	2·79*
	$\bar{\mathrm{O}}\bar{W}(2^{\mathrm{III}})\text{–}\mathrm{O}W(2^{\mathrm{I}})$	3·79

* Hydrogen bond.

Table 6. *Ellipsoids of vibration referred to orthogonal cell axes*

	i	$(r_i^2)^{\frac{1}{2}}$	$a_{i\mathbf{a}}$	$a_{i\mathbf{b}'}$	$a_{i\mathbf{c}'}$
Cu	1	0·160 (2) Å	−0·907	0·062	0·414
	2	0·170 (3)	0·385	0·514	0·765
	3	0·136 (1)	−0·165	0·854	−0·491
O(1)	1	0·172 (4)	−0·489	−0·784	0·381
	2	0·238 (2)	0·479	0·123	0·868
	3	0·148 (6)	−0·728	0·607	0·316
O(2)	1	0·163 (4)	−0·868	−0·314	0·384
	2	0·219 (4)	0·368	0·112	0·923
	3	0·152 (5)	−0·332	0·942	0·018
O(3)	1	0·185 (3)	−0·481	−0·873	0·068
	2	0·210 (4)	−0·684	0·423	0·593
	3	0·168 (6)	0·547	−0·239	0·801
O(4)	1	0·204 (4)	0·899	−0·317	−0·299
	2	0·190 (6)	−0·381	−0·905	−0·186
	3	0·173 (3)	0·212	−0·282	0·935
C(1)	1	0·172 (7)	0·772	−0·473	−0·422
	2	0·179 (3)	0·453	0·877	−0·152
	3	0·114 (3)	0·443	−0·073	0·893
C(2)	1	0·137 (3)	0·896	−0·442	−0·028
	2	0·182 (6)	0·117	0·176	0·977
	3	0·173 (5)	−0·427	−0·879	0·210
C(3)	1	0·193 (4)	0·909	0·242	−0·337
	2	0·176 (7)	−0·394	0·251	−0·833
	3	0·145 (4)	−0·129	0·936	0·324
C(4)	1	0·169 (4)	−0·649	0·355	−0·672
	2	0·208 (8)	0·568	0·814	−0·118
	3	0·158 (2)	−0·505	0·458	0·730
O*W*(1)	1	0·227 (8)	−0·755	−0·298	−0·583
	2	0·296 (5)	−0·603	−0·032	0·797
	3	0·195 (3)	−0·256	0·953	−0·389
O*W*(2)	1	0·184 (3)	−0·980	0·171	0·101
	2	0·291 (8)	0·159	0·369	0·915
	3	0·150 (3)	0·119	0·913	−0·389

Thermal vibrations

The amplitudes, $\overline{(r_i^2)}^{\frac{1}{2}}$ of the three principal axes of the thermal ellipsoids and their direction cosines ($a_{i\mathbf{a}}$, $a_{i\mathbf{b}'}$, $a_{i\mathbf{c}'}$) relative to the orthogonal axial set **a**, **b**′, **c**′ (Cruickshank, 1956) are listed in Table 6. The orthogonal axes are defined so that **b**′ lies in the *xy* plane and **c**′ is mutually perpendiclar to **a** and **b**′. The ellipsoids are shown in Fig. 7 as viewed down the *a* axis. The principal axes of the ellipsoids as shown are proportional to the mean square displacements.

In a polymeric structure the covalent bonds which link the basic units hinder rigid-body motion. The

Table 7. *Observed and calculated structure factors*

H	K	L	F(OBS)	F(CALC)
0	0	1	-27.1	-33.1
		2	-11.2	-8.8
		3	6.0	5.7
		4	5.2	5.0
		5	-10.9	-10.1
		6	-2.9	-2.4
		7	4.7	4.3
		8	-2.7 U	-.2
		9	3.1	2.8
0	1	0	25.4	29.3
		1	-6.8	-5.8
		2	11.7	11.6
		3	-5.8	-5.9
		4	21.0	22.0
		5	-23.5	-23.3
		6	6.0	5.6
		7	-2.9	-2.9
		8	2.6 U	1.0
		9	1.9 U	.6
		-1	-14.8	-17.1
		-2	-9.2	-9.2
		-3	.9	1.2
		-4	-6.0	-6.7
		-5	7.3	6.9
		-6	-13.3	-11.2
		-7	15.0	13.3
		-8	-6.3	-5.9
		-9	2.3	1.9
0	2	0	-11.7	-11.9
		1	20.1	19.4
		2	-7.2	-7.0
		3	-6.9	-7.8
		4	20.3	20.7
		5	-12.9	-12.3
		6	6.9	6.2
		7	-7.3	-6.9
		8	2.4 U	1.9
		9	-3.0	-2.9
		-1	-4.6	-4.1
		-2	-20.0	-20.5
		-3	16.0	17.9
		-4	-9.1	-8.9
		-5	11.2	10.0
		-6	-14.8	-14.2
		-7	13.3	11.6
		-8	-4.6	-3.9
		-9	-2.3 U	-.7
0	3	0	-15.6	-16.0
		1	-1.6 U	0.0
		2	-5.8	-6.4
		3	7.4	8.3
		4	-3.3	-3.1
		5	4.2	3.5
		6	2.9 U	1.9
		7	-3.6	-2.9
		8	2.9	2.1
		-1	30.9	33.4
		-2	-32.3	-33.0
		-3	16.8	17.1
		-4	-4.8	-5.0
		-5	2.6 U	.9
		-6	2.9 U	.3
		-7	-3.0 U	-.6
		-8	2.7	2.5
		-9	-2.5	-2.2
0	4	0	-14.7	-14.5
		1	10.7	10.5
		2	-9.0	-8.8
		3	13.6	13.5
		4	-13.7	-14.6
		5	7.3	7.5
		6	2.7 U	0.0
		7	2.3 U	1.4
		8	-1.9	-.9
		-1	12.1	11.8
		-2	-4.8	-4.4
		-3	-2.4	-1.3
		-4	4.9	4.9
		-5	-7.3	-7.0
		-6	8.3	8.5
		-7	-5.8	-5.1
		-8	8.1	6.3
		-9	-6.7	-5.3
0	5	0	7.4	7.2
		1	2.4 U	.3
		2	-8.2	-7.4
		3	9.5	10.4
		4	-7.8	-8.1
		5	4.8	4.3
		6	-3.6	-2.5
		7	3.8	3.2
		-1	-12.1	-11.4
		-2	7.6	7.4
		-3	-9.4	-9.0
		-4	12.4	12.2
		-5	-7.9	-7.6
		-6	8.6	8.3
		-7	-6.9	-6.7
		-8	4.8	4.0
		-9	-4.1	-3.6
0	6	0	7.8	6.6
		1	-12.2	-11.0
		2	6.0	5.7
		3	-2.9 U	-1.7
		4	2.7 U	.6
		5	2.4 U	.4
		6	-1.6 U	-1.4
		-1	-9.0	-8.0
		-2	11.2	11.0
		-3	-7.7	-7.8
		-4	9.4	9.5
		-5	-7.2	-7.2
		-6	4.6	4.2
		-7	-2.6 U	-1.7
		-8	-4.5	-3.5
0	7	0	6.9	7.4
		1	-6.0	-5.6
		2	4.9	5.2
		3	-9.4	-7.7
		4	4.2	3.6
		5	-1.3 U	-2.0
		-1	-8.5	-8.2
		-2	9.0	8.7
		-3	3.0	1.0
		-4	-3.7	-3.5
		-5	2.7	.9
		-6	-5.8	-4.2
		-7	5.8	4.2
		-8	-5.5	-4.9
0	8	0	2.9	3.2
		1	-2.5 U	-.8
		2	2.9	2.4
		3	-4.2	-3.7
		-1	2.7 U	1.1
		-2	-3.3	-2.5
		-3	5.8	5.0
		-4	-12.1	-9.4
		-5	7.0	5.6
		-6	-5.9	-4.2
		-7	5.2	4.2
0	9	0	-7.0	-6.0
		1	4.8	4.4
		-1	7.1	5.6
		-2	-6.3	-5.3
		-3	4.3	3.1
		-4	-3.9	-4.3
1	0	0	20.7	18.8
		1	-27.7	-28.6
		2	32.2	33.6
		3	-15.7	-17.7
		4	16.7	15.6
		5	-2.6	-2.7
		6	3.7	3.4
		7	-4.3	-4.4
		8	-2.5	-3.4
		9	1.9 U	1.9
		-1	-32.3	-28.7
		-2	-3.8	-3.2
		-3	5.5	4.4
		-4	2.4	1.8
		-5	2.1 U	1.3
		-6	-3.9	-4.5
		-7	5.4	5.9
		-8	-3.5	-3.4
		-9	2.5	3.0
1	1	0	6.4	6.5
		1	-13.3	-12.2
		2	-7.6	-5.9
		3	-6.7	-7.3
		4	22.8	23.0
		5	-14.2	-15.6
		6	11.9	12.7
		7	-6.0	-5.5
		8	2.4	2.1
		9	-3.6	-3.6
		-1	2.3	2.5
		-2	-8.4	-9.4
		-3	3.9	5.0
		-4	-5.3	-5.8
		-5	14.2	15.2
		-6	-15.2	-16.9
		-7	10.2	10.4
		-8	-2.1 U	-1.9
		-9	1.5 U	0.0
1	2	0	-20.0	-19.7
		1	6.6	7.4
		2	-2.4	-3.0
		3	1.8	1.3
		4	1.9 U	.3
		5	-6.2	-6.1
		6	8.4	9.0
		7	-3.5	-3.6
		8	4.6	4.8
		9	-4.3	-4.5
		-1	24.4	24.0
		-2	-23.9	-22.1
		-3	13.9	14.0
		-4	-8.7	-8.4
		-5	7.2	7.7
		-6	-7.1	-7.9
		-7	7.1	7.9
		-8	-2.1 U	-.2
		-9	-4.0	-4.0
1	3	0	-22.8	-24.1
		1	12.6	13.3
		2	-8.3	-7.1
		3	5.6	5.9
		4	-7.9	-8.6
		5	6.9	7.3
		6	-2.6	-2.9
		7	-2.2 U	-.2
		8	2.6	2.5
		-1	25.9	26.7
		-2	-26.6	-26.0
		-3	7.9	7.6
		-4	9.6	9.8
		-5	-6.8	-6.8
		-6	4.0	4.1
		-7	-2.3 U	-1.7
		-8	2.1 U	1.6
		-9	-4.7	-5.2
1	4	0	-3.3	-3.3
		1	-4.3	-4.2
		2	-7.2	-7.8
		3	16.7	17.4
		4	-16.4	-16.4
		5	8.5	9.1
		6	-2.7	-2.6
		7	1.9 U	1.5
		8	-2.3	-2.1
		-1	3.1	3.8
		-2	4.6	6.0
		-3	-6.0	-4.9
		-4	4.6	6.5
		-5	-4.1	-5.6
		-6	7.1	8.5
		-7	-10.7	-11.3
		-8	4.8	5.0
1	5	0	6.4	6.7
		1	-8.7	-8.5
		2	4.2	4.1
		3	3.1	3.1
		4	-10.5	-10.4
		5	4.5	4.8
		6	1.9 U	.6
		7	2.7	2.2
		-1	-6.2	-7.2
		-2	17.3	18.7
		-3	-11.9	-12.9
		-4	4.8	5.6
		-5	-4.8	-5.2
		-6	3.5	3.9
		-7	-7.3	-7.0
		-8	3.8	3.6
1	6	0	5.9	5.6
		1	-6.8	-6.5
		2	8.9	9.1
		3	-5.2	-6.0
		4	2.2 U	1.7
		5	-4.8	-4.1
		6	1.3 U	1.4
		-1	-2.5	-2.0
		-2	11.6	12.3
		-3	-17.7	-18.7
		-4	5.2	5.7
		-5	2.6	2.6
		-6	-6.4	-5.6
		-7	2.9	3.0
		-8	-1.3 U	-1.0
1	7	0	2.3 U	1.2
		1	-8.5	-7.9
		2	14.1	11.7
		3	-4.1	-3.9
		4	3.6	2.7
		5	-4.2	-4.7
		-1	2.6	2.2
		-2	-2.5	-1.6
		-3	-2.7	-2.8
		-4	-3.1	-3.6
		-5	7.3	7.6
		-6	-4.9	-5.4
		-7	3.9	4.2
1	8	0	-3.8	-3.2
		1	-1.9 U	-1.8
		2	3.0	3.0
		3	-1.3 U	-.7
		-1	5.5	5.5
		-2	-8.5	-8.8
		-3	6.8	6.6
		-4	-4.1	-3.8
		-5	4.6	4.8
		-6	-3.3	-3.6
1	9	0	-4.2	-4.2
		1	2.9	3.0
		-1	4.1	4.4
		-2	-3.2	-3.0
		-3	3.0	2.9
		-4	-3.1	-2.8
1	-1	0	30.4	26.6
		1	-12.7	-11.7
		2	19.0	20.4
		3	6.8	6.0
		4	-2.6	-2.5
		5	1.9 U	.7
		6	-6.8	-7.0
		7	2.7	2.8
		8	-6.6	-6.2
		9	8.5	7.8
		10	-3.8	-4.2
1	-2	0	12.3	11.7
		1	20.0	23.1
		2	-13.4	-12.0
		3	1.3	.5
		4	-16.0	-12.7
		5	17.1	16.6
		6	-14.3	-15.3
		7	4.7	5.4
		8	-2.3 U	-2.2
		9	6.8	6.4
		10	-4.2	-4.2
1	-3	0	-11.5	-10.8
		1	10.8	10.9
		2	-13.8	-13.8
		3	11.5	12.3
		4	-22.6	-24.6
		5	19.0	19.5
		6	-8.7	-8.5
		7	2.4 U	.4
		8	3.1	4.1
		9	-1.9 U	-1.9
		10	1.2 U	.1
1	-4	0	-18.7	-20.3
		1	9.4	10.7
		2	-13.8	-14.0
		3	5.6	5.3
		4	2.0 U	1.9
		5	-2.1 U	-1.3
		6	2.3 U	.3
		7	-2.6	-3.1
		8	6.0	6.3
		9	-4.9	-5.7
		10	3.7	4.2
1	-5	0	-11.2	-11.1
		1	2.0 U	1.6
		2	7.0	7.7
		3	-8.2	-9.7
		4	8.2	9.8
		5	-8.5	-11.1
		6	9.0	10.3
		7	-4.8	-5.7
		8	3.7	4.1
		9	-2.4	-2.8
1	-6	0	6.9	6.9
		1	-12.5	-14.9
		2	10.4	11.3
		3	-2.5	-2.2
		4	2.6	2.4
		5	-10.2	-11.6
		6	13.8	14.7
		7	-4.8	-5.1
		8	-1.9 U	0.0
		9	1.3 U	1.6
1	-7	0	7.1	7.8
		1	-12.8	-14.7
		2	9.5	10.0
		3	-3.0	-3.2
		4	-2.4 U	-.2
		5	2.3 U	1.0
		6	3.1	3.0
		7	1.9 U	.2
		8	-4.1	-4.6
1	-8	0	-2.1 U	-.1
		1	-7.3	-6.3
		2	8.3	7.5
		3	2.4	2.1
		4	-7.8	-6.8
		5	9.2	8.5
		6	-6.0	-5.4
		7	3.0	3.3
1	-9	0	-3.5	-3.4
		1	4.4	4.6
		2	-4.2	-3.4
		3	7.1	7.0
		4	-5.3	-5.2
		5	2.3	2.4
		6	-2.9	-3.9
-1	1	1	-28.2	-26.4
		2	16.1	15.9
		3	-20.1	-21.3
		4	9.4	8.7
		5	-5.2	-5.9
		6	5.2	5.2
		7	2.3 U	.2
		8	-1.9 U	-.3
		9	1.6	1.8
-1	2	1	-4.8	-5.8
		2	9.0	8.7
		3	-15.7	-15.6
		4	12.2	12.9
		5	-13.5	-14.2
		6	5.5	6.3
		7	-2.5	-2.6
		8	4.6	3.9
-1	3	1	12.3	12.3
		2	-3.3	-2.8
		3	5.5	5.9
		4	-2.3 U	-.3
		5	-7.3	-7.8
		6	3.2	3.6
		7	-4.4	-5.1
		8	3.9	4.2
-1	4	1	20.1	22.1
		2	-12.3	-13.2
		3	7.0	6.7
		4	-2.4 U	-1.1
		5	4.0	4.1
		6	-2.9	-4.0
		7	-1.6 U	-1.7
-1	5	1	11.8	12.0
		2	-9.3	-9.5
		3	3.8	4.1
		4	-3.0	-2.8
		5	9.1	8.2
		6	-6.8	-6.1
-1	6	1	-2.4 U	-1.2
		2	3.3	3.9
		3	-2.9	-2.9
		4	-3.1	-4.2
		5	7.9	7.5
		6	-3.3	-4.1
-1	7	1	-4.1	-4.0
		2	4.9	5.5
		3	-4.7	-5.5
		4	2.7	2.4
		5	.7 U	.7
-1	8	1	-2.3	-1.8
		2	2.7	3.0
		3	-4.6	-4.6
-1	9	1	1.2 U	1.6
2	0	0	-18.8	-16.0
		1	4.7	3.4
		2	25.8	27.0
		3	-42.5	-43.5
		4	22.7	22.8
		5	-7.5	-7.7
		6	4.7	5.0
		7	-1.8 U	-1.0
		8	2.9	3.6
		9	-1.2 U	-.4
		10	-.7 U	-1.3
		-1	-3.1	-4.0
		-2	14.1	14.7
		-3	1.3 U	.8
		-4	-8.4	-9.5
		-5	4.9	6.2
		-6	-2.6	-2.9
		-7	7.3	7.1
		-8	-6.0	-7.6
2	1	0	-9.0	-9.6
		1	7.2	7.7
		2	6.3	6.6
		3	-15.2	-16.3
		4	4.9	5.7
		5	-3.0	-3.3
		6	5.8	6.6
		7	-9.4	-9.4
		8	8.5	8.6
		9	-3.7	-3.5
		-1	-2.6	-.4
		-2	-12.5	-11.5
		-3	19.0	22.3
		-4	-14.8	-17.9
		-5	10.5	11.9
		-6	-3.6	-4.0
		-7	1.6	2.3
		-8	-4.6	-4.1
2	2	0	-17.3	-18.7
		1	4.2	4.3
		2	-11.7	-11.9
		3	10.1	11.3
		4	-5.0	-6.3
		5	-1.6 U	-.2
		6	1.8 U	2.6
		7	-3.0	-3.9
		8	4.5	4.3
		9	-3.3	-3.9
		-1	17.3	24.6
		-2	-14.8	-14.7
		-3	12.7	14.4
		-4	-6.9	-7.8
		-5	4.5	5.1
		-6	-1.8 U	-.6
		-7	-4.4	-4.4
		-8	5.8	1.0
2	3	0	-11.9	-13.2
		1	15.1	18.0
		2	-16.4	-18.6
		3	11.9	13.9
		4	-9.3	-11.3
		5	3.0	3.0
		6	-1.5 U	-1.5
		7	4.3	4.3
		8	-1.9	-2.0
		-1	11.8	11.2
		-2	5.2	5.1
		-3	-2.6	-3.5
		-4	3.3	2.9
		-5	-5.2	-5.9
		-6	1.8	3.2
		-7	-6.4	-5.9
		-8	4.9	5.4
2	4	0	10.8	11.9
		1	1.4 U	1.1
		2	-5.4	-6.3
		3	6.3	7.8
		4	-6.4	-7.2
		5	3.6	4.1
		6	-6.9	-7.6
		7	6.2	6.6
		8	-2.3	-2.7
		-1	-3.8	-4.7
		-2	3.0	3.9
		-3	-9.4	-10.0
		-4	6.8	7.0
		-5	-8.2	-9.1

Table 7 (*cont.*)

H	K	L	F(OBS)	F(CALC)
		-6	7.1	7.7
		-7	-6.7	-6.2
		-8	4.9	5.1
2	5	0	7.6	7.7
		1	-6.7	-7.3
		2	6.8	8.5
		3	-1.9 U	-.5
		4	2.0 U	1.1
		5	-2.2 U	-.9
		6	-4.6	-4.4
		7	3.0	3.1
		-1	-9.7	-10.0
		-2	10.0	10.2
		-3	-13.5	-14.2
		-4	5.3	6.3
		-5	-1.8 U	-1.8
		-6	1.6 U	2.7
		-7	-1.3 U	-.6
		-8	-.7 U	-.1
2	6	0	3.0	3.3
		1	-4.9	-5.8
		2	6.9	7.7
		3	-5.8	-6.2
		4	7.1	6.6
		5	-1.3	-2.1
		6	.9	1.5
		-1	-9.2	-9.7
		-2	7.6	8.3
		-3	-3.6	-4.4
		-4	-1.8 U	-.8
		-5	6.7	6.9
		-6	-4.5	-5.1
		-7	2.9	3.2
2	7	0	-2.0	-2.2
		1	-4.7	-4.9
		2	5.2	4.8
		3	-5.8	-5.4
		4	4.8	4.6
		5	-.5	-1.7
		-1	5.0	6.1
		-2	-4.5	-4.1
		-3	3.7	3.5
		-4	-4.1	-4.0
		-5	6.7	5.8
		-6	-3.7	-4.2
2	8	0	-6.0	-5.5
		1	2.3	2.5
		2	-1.6	-.2
		3	-1.3	-.6
		-1	6.4	6.9
		-2	-4.8	-4.9
		-3	4.4	4.3
		-4	-4.9	-4.8
		-5	3.7	4.1
2	9	0	-1.0 U	-1.9
		-1	.7 U	.7
2	-1	0	17.1	17.3
		1	-6.0	-6.3
		2	1.2	2.1
		3	-22.4	-25.2
		4	20.2	21.7
		5	3.1	4.0
		6	-3.2	-2.8
		7	1.8 U	2.1
		8	-1.8	-2.8
		9	4.2	4.2
		10	-6.0	-6.9
2	-2	0	6.4	7.0
		1	11.7	11.0
		2	-6.6	-6.4
		3	5.2	6.0
		4	-4.0	-4.2
		5	13.8	15.0
		6	-6.8	-8.4
		7	1.8 U	1.8
		8	-6.9	-6.9
		9	6.4	6.0
		10	-5.2	-6.0
2	-3	0	-6.9	-7.3
		1	6.9	7.4
		2	1.6	.9
		3	13.2	14.5
		4	-16.5	-18.4
		5	16.9	19.4
		6	-16.0	-17.4
		7	2.6	3.4
		8	-1.8	-.5
		9	1.6	.8
		10	-.9	-.1
2	-4	0	-15.4	-15.3
		1	20.0	21.8
		2	-7.3	-7.6
		3	2.3	2.8
		4	-11.7	-12.9
		5	10.4	11.0
		6	-4.8	-5.2
		7	-2.6	-3.3
		8	4.2	5.1
		9	-1.3	-2.5
		10	2.6	3.9
2	-5	0	-17.9	-19.6
		1	13.4	14.4
		2	-2.2	-2.7
		3	-7.6	-7.3
		4	2.7	4.2
		5	-5.8	-6.9
		6	6.9	8.5
		7	-4.9	-6.5
		8	4.8	4.9
		9	-3.1	-3.6
		10	3.4	3.9
2	-6	0	1.6 U	1.6
		1	-6.2	-6.7
		2	4.9	5.9
		3	-7.9	-9.1
		4	5.6	9.8
		5	-6.6	-7.7
		6	6.0	7.2
		7	-7.2	-7.7
		8	5.9	5.4
		9	-.9 U	-1.3
2	-7	0	8.5	9.2
		1	-8.3	-8.1
		2	9.4	8.6
		3	-7.3	-7.8
		4	5.6	5.9
		5	-4.9	-4.8
		6	3.3	2.9
		7	-1.5 U	-1.0
		8	.9	.4
2	-8	0	5.3	4.7
		1	-7.1	-6.1
		2	4.0	3.5
		3	-2.3	-2.2
		4	1.6 U	.7
		5	1.6 U	.5
		6	-3.9	-3.8
		7	6.9	7.2
2	-9	0	.9 U	1.1
		1	-1.2 U	-.8
		2	-4.7	-4.5
		3	5.2	5.3
		4	-4.4	-4.4
		5	5.0	5.5
-2	1	1	-36.6	-34.7
		2	23.8	24.1
		3	-4.3	-4.5
		4	1.6 U	1.6
		5	-1.8 U	-1.0
		6	1.8 U	.7
		7	2.3	2.6
		8	-3.7	-3.6
-2	2	1	-42.0	-43.0
		2	24.8	26.0
		3	-6.4	-6.6
		4	4.3	4.0
		5	-3.9	-5.2
		6	7.3	7.2
		7	-3.3	-3.3
-2	3	1	3.1	4.0
		2	-4.2	-4.3
		3	-6.2	-5.7
		4	4.2	4.7
		5	-5.3	-6.5
		6	9.7	9.2
		7	-4.5	-5.1
-2	4	1	15.7	15.7
		2	-9.4	-10.0
		3	2.3	1.9
		4	-1.8	-2.7
		5	-1.6 U	-1.6
		6	1.3 U	1.5
-2	5	1	12.7	14.3
		2	-1.8 U	-.3
		3	2.6	2.9
		4	-8.2	-7.5
		5	6.4	5.5
		6	-5.2	-5.2
-2	6	1	1.8 U	.9
		2	-1.8 U	-1.0
		3	4.7	4.7
		4	-4.9	-5.2
		5	6.8	6.5
-2	7	1	-8.6	-7.6
		2	3.1	3.3
		3	-1.3 U	-.3
		4	-.9 U	-.2
-2	8	1	-2.5	-2.5
		2	4.2	4.3
		3	-4.1	-5.5
-2	9	1	-.7 U	-1.2
3	0	0	-.6 U	0.0
		1	-2.2	-2.2
		2	1.3	1.1
		3	-15.0	-15.6
		4	22.6	21.9
		5	-16.3	-15.7
		6	4.1	4.6
		7	-4.3	-4.6
		8	6.7	5.6
		9	-4.6	-4.2
		-1	3.5	2.9
		-2	-4.2	-4.0
		-3	9.6	9.4
		-4	-7.2	-6.6
		-5	6.0	5.4
		-6	-8.4	-7.2
		-7	6.6	5.6
		-8	-5.2	-6.2
3	1	0	-4.9	-5.6
		1	4.6	5.2
		2	-1.2 U	-.6
		3	-3.2	-3.6
		4	-2.4	-.7
		5	-2.5	-1.5
		6	3.3	2.8
		7	-7.0	-5.6
		8	10.7	8.0
		9	-8.5	-6.4
		-1	19.1	17.2
		-2	-17.8	-17.9
		-3	21.3	23.2
		-4	-14.3	-14.2
		-5	-2.0 U	-.9
		-6	1.9 U	.6
		-7	1.6 U	1.0
		-8	-1.8	-2.1
3	2	0	-1.2 U	-1.2
		1	19.5	22.7
		2	-20.0	-20.9
		3	13.2	13.7
		4	-7.0	-6.9
		5	2.6	2.0
		6	-3.3	-2.8
		7	-2.6 U	-1.2
		8	3.2	3.3
		-1	5.5	5.8
		-2	-13.5	-11.9
		-3	8.4	8.5
		-4	-6.0	-6.5
		-5	-2.0	-3.0
		-6	5.6	5.0
		-7	-3.1	-2.9
		-8	4.1	3.4
3	3	0	2.1	2.2
		1	8.9	9.6
		2	-14.2	-16.5
		3	16.7	21.1
		4	-6.0	-6.7
		5	.9 U	.6
		6	-4.8	-5.6
		7	4.8	4.6
		8	-3.7	-4.1
		-1	-7.8	-8.1
		-2	2.9	2.4
		-3	-7.7	-7.7
		-4	3.3	3.8
		-5	-2.0	-3.0
		-6	7.9	6.3
		-7	-5.2	-4.8
3	4	0	4.0	3.8
		1	-4.6	-5.1
		2	1.8 U	.7
		3	1.9	1.9
		4	2.0	2.3
		5	2.3	2.4
		6	-4.7	-5.5
		7	6.0	6.4
		-1	-6.8	-7.5
		-2	4.8	4.8
		-3	-9.3	-9.8
		-4	13.9	14.3
		-5	-8.3	-8.1
		-6	4.2	3.5
		-7	-1.3 U	-1.0
3	5	0	12.5	13.0
		1	-12.7	-13.0
		2	2.0	2.8
		3	-3.3	-4.2
		4	6.3	7.0
		5	-1.9	-1.9
		6	-1.1 U	-.8
		-1	-5.8	-5.9
		-2	2.0	2.6
		-3	-4.3	-4.4
		-4	6.4	7.8
		-5	-1.8	-2.3
		-6	-3.0	-2.9
		-7	2.6	3.1
3	6	0	11.5	9.9
		1	-11.9	-8.9
		2	6.0	3.9
		3	-3.1	-3.5
		4	3.0	3.3
		5	-4.3	-4.7
		6	3.0	3.2
		-1	-3.3	-2.3
		-2	-5.4	-3.8
		-3	6.8	6.0
		-4	-4.6	-3.8
		-5	3.5	2.8
		-6	-3.6	-3.7
3	7	0	-1.8 U	-1.3
		1	3.3	3.0
		2	-1.1 U	-1.3
		3	-1.5	-1.9
		4	2.9	3.2
		-1	2.4	2.5
		-2	-6.8	-6.3
		-3	7.9	6.2
		-4	-3.1	-3.1
		-5	3.3	2.8
3	8	0	-4.6	-4.6
		1	2.8	4.4
		-1	4.7	3.5
		-2	-3.3	-2.9
		-3	4.2	4.2
3	-1	0	9.9	9.8
		1	-5.2	-4.8
		2	17.3	16.1
		3	-26.1	-25.0
		4	14.1	15.3
		5	-10.9	-10.4
		6	3.6	2.9
		7	2.6 U	1.1
		8	-2.6 U	-1.2
		9	4.3	2.6
		10	-1.5 U	-.5
3	-2	0	18.8	18.1
		1	-17.6	-17.8
		2	9.3	8.9
		3	-1.6 U	0.0
		4	-9.3	-8.5
		5	2.6	2.3
		6	2.6 U	.6
		7	10.1	8.1
		8	-8.9	-7.0
		9	7.6	6.2
		10	-4.6	-3.5
3	-3	0	-2.2	-1.5
		1	4.8	4.2
		2	-10.4	-9.9
		3	5.2	5.3
		4	-10.6	-9.2
		5	16.2	16.0
		6	-6.8	-6.2
		7	8.3	7.0
		8	-10.5	-8.8
		9	7.6	5.6
		10	-3.6	-3.0
3	-4	0	-14.8	-16.9
		1	12.1	12.5
		2	-8.3	-7.5
		3	12.5	13.0
		4	-13.4	-14.3
		5	14.5	14.7
		6	-4.2	-3.6
		7	-2.6 U	-1.7
		8	-3.8	-2.8
		9	2.3 U	.1
		10	1.5 U	1.1
3	-5	0	-6.8	-6.7
		1	11.5	12.2
		2	-10.5	-10.8
		3	11.3	11.7
		4	-2.6 U	-2.0
		5	-2.6 U	-2.0
		6	2.6 U	1.3
		7	-10.0	-7.4
		8	8.3	6.3
		9	-5.5	-4.4
3	-6	0	1.6 U	1.0
		1	4.3	4.0
		2	1.9 U	2.0
		3	-7.3	-8.4
		4	7.7	8.9
		5	-7.6	-8.2
		6	4.7	3.6
		7	-6.9	-4.7
		8	7.8	6.4
		9	-6.0	-5.3
3	-7	0	9.0	7.4
		1	-9.0	-7.7
		2	5.5	5.1
		3	-8.7	-7.5
		4	11.7	9.7
		5	-12.5	-10.5
		6	5.6	4.5
		7	2.3 U	.4
		8	1.9 U	.3
3	-8	0	8.6	8.1
		1	-6.0	-5.3
		2	-1.8 U	-.6
		3	-1.8 U	-1.5
		4	7.6	6.7
		5	-7.1	-6.0
		6	1.5 U	.1
		7	3.7	3.5
3	-9	0	4.2	4.5
		1	-2.0	-2.1
		2	-1.8 U	-.6
		3	2.9	2.8
		4	-3.9	-3.7
-3	1	1	-20.7	-17.5
		2	11.7	11.4
		3	-2.3	-1.3
		4	-1.9 U	-.7
		5	2.0 U	2.3
		6	-2.6	-3.3
		7	4.8	4.2
-3	2	1	-20.6	-17.7
		2	22.8	19.4
		3	-7.9	-7.5
		4	1.9 U	1.0
		5	-2.6	-2.2
		6	5.2	4.5
		7	-1.8	-2.0
-3	3	1	-7.1	-7.0
		2	10.8	10.2
		3	-6.0	-5.5
		4	7.2	7.4
		5	-8.2	-7.3
		6	8.5	6.7
		7	-3.8	-3.9
-3	4	1	2.6	2.2
		2	-1.9	-2.1
		3	2.4	3.0
		4	1.9 U	1.6
		5	-3.3	-3.7
		6	5.5	5.4
-3	5	1	4.3	4.4
		2	-11.7	-10.4
		3	7.6	6.5
		4	-6.3	-4.9
		5	2.6	2.2
-3	6	1	5.5	4.8
		2	-10.7	-9.7
		3	8.5	6.7
		4	-5.8	-4.8
-3	7	1	-2.5 U	-.1
		2	-2.3 U	-.9
		3	2.3	1.9
-3	8	1	-7.1	-6.0
		2	3.8	3.8
4	0	0	1.0	1.1
		1	.8	.9
		2	4.8	4.2
		3	-1.3	-.5
		4	1.6	1.1
		5	-7.4	-8.7
		6	9.2	10.7
		7	-7.8	-9.9
		8	5.4	5.8
		9	-2.5	-2.7
		-1	9.4	9.3
		-2	-11.9	-12.0
		-3	5.2	5.6
		-4	-9.0	-9.2
		-5	7.3	7.7
		-6	-5.2	-5.6
		-7	2.6	3.3
4	1	0	-14.7	-13.4
		1	9.4	9.3
		2	-7.2	-7.1
		3	11.3	11.9
		4	2.7	.8
		5	-3.8	-4.0
		6	1.3 U	.1
		7	-2.6	-2.6
		8	3.3	3.0
		9	-3.5	-3.8
		-1	16.0	16.4
		-2	-18.3	-18.1
		-3	4.2	3.7
		-4	-.9 U	-.5
		-5	1.6	2.1
		-6	-.7 U	-.3
		-7	.7 U	.6
4	2	0	-7.6	-7.4
		1	10.5	10.2
		2	-17.8	-15.6
		3	13.3	14.0
		4	-4.8	-4.6
		5	6.4	7.2
		6	-4.0	-5.1
		7	2.0	2.3
		8	-1.0	-1.2
		9	-.3 U	-1.9
		-1	-.7 U	-.7
		-2	-3.8	-3.8
		-3	3.2	2.4
		-4	4.1	4.2
		-5	-2.9	-3.0
		-6	3.2	3.3
		-7	-3.2	-3.2
4	3	0	6.3	6.9
		1	-2.6	-.5
		2	-9.7	-10.1
		3	3.8	4.1
		4	-2.2	-2.2
		5	7.6	8.8
		6	-6.8	-7.1
		7	5.2	5.6
		8	-1.9	-1.7
		-1	-6.8	-6.8
		-2	3.6	3.9
		-3	-3.9	-3.3
		-4	9.3	8.4
		-5	-6.8	-7.0
		-6	3.1	3.6
		-7	-2.1	-2.6
4	4	0	6.2	6.1
		1	-5.0	-4.8
		2	3.6	3.5
		3	-4.8	-5.3
		4	1.3 U	1.4
		5	1.3 U	.5
		6	-2.6	-2.5
		7	4.1	4.3
		-1	-5.8	-5.8
		-2	8.2	8.6
		-3	-9.3	-9.5
		-4	7.9	8.6
		-5	-4.4	-4.5
		-6	-.5 U	-.6

Table 7 (*cont.*)

H	K	L	F(OBS)	F(CALC)
4	5	0	7.9	8.2
		1	-9.3	-8.2
		2	7.9	8.3
		3	-6.8	-6.5
		4	2.4	2.8
		5	-2.5	-2.5
		6	.8	1.3
		-1	-4.4	-4.4
		-2	-.9 U	-.2
		-3	.9 U	.5
		-4	.8 U	1.1
		-5	.7 U	.5
		-6	-2.5	-2.8
4	6	0	2.6	2.6
		1	-6.4	-6.2
		2	3.8	3.6
		3	-1.2	-1.2
		4	2.5	2.5
		5	-3.6	-3.8
		-1	4.1	4.0
		-2	-6.9	-6.6
		-3	6.2	5.6
		-4	-2.5	-2.5
		-5	2.4	2.6
4	7	0	-2.9	-2.8
		1	2.7	3.0
		2	-2.6	-3.3
		3	1.6	2.2
		-1	4.5	4.0
		-2	-2.9	-2.8
		-3	3.1	3.2
4	-1	0	2.6	2.6
		1	-16.6	-15.8
		2	23.2	21.8
		3	-11.7	-12.0
		4	3.3	2.9
		5	-8.2	-8.7
		6	9.4	11.1
		7	-5.5	-6.7
		8	.9	1.1
		9	-1.2 U	-.2
4	-2	0	8.6	8.5
		1	-21.5	-21.3
		2	23.9	22.7
		3	-13.7	-13.5
		4	2.6	2.7
		5	1.2	1.4
		6	-3.0	-3.2
		7	1.0	1.5
		8	-3.9	-4.2
		9	4.6	4.6
4	-3	0	1.7	2.1
		1	-2.4	-.3
		2	4.2	3.8
		3	.9	1.4
		4	-9.0	-9.4
		5	3.8	4.5
		6	-3.6	-3.9
		7	6.9	7.2
		8	-7.9	-8.8
		9	8.4	8.5
4	-4	0	-2.7	-3.3
		1	3.8	4.4
		2	-11.9	-11.9
		3	15.2	15.6
		4	-13.7	-14.3
		5	3.8	4.2
		6	-1.3	-1.8
		7	7.1	8.0
		8	-4.4	-4.8
		9	3.3	3.1
4	-5	0	-3.6	-4.2
		1	.9	.9
		2	-9.7	-10.6
		3	13.0	13.4
		4	-11.2	-12.4
		5	6.3	7.4
		6	3.1	3.5
		7	-1.9	-2.3
		8	1.2	1.9
		9	-3.0	-4.0
4	-6	0	-8.2	-9.0
		1	-.9 U	-.3
		2	.9	1.2
		3	2.9	3.1
		4	.9 U	.6
		5	-2.5	-3.5
		6	7.3	7.8
		7	-5.5	-5.8
		8	1.3	2.1
		9	-4.5	-5.8
4	-7	0	-.8	-.9
		1	-2.3	-2.3
		2	6.4	7.3
		3	-4.8	-5.7
		4	7.8	9.0
		5	-7.0	-8.0
		6	3.8	4.5
		7	-3.8	-4.5
		8	1.3	1.7
4	-8	0	5.2	6.7
		1	-2.2	-2.8
		2	3.9	4.8
		3	-4.4	-5.2
		4	3.5	3.9
		5	-3.9	-4.2
		6	2.9	2.8
		7	-1.8	-1.9
4	-9	2	.5	.9
		3	-.8	-1.2
		4	.5 U	.3
-4	1	1	4.2	4.2
		2	4.4	4.5
		3	-3.8	-2.3
		4	-5.0	-6.0
		5	6.2	5.9
		6	-6.0	-5.8
-4	2	1	-2.7	-1.7
		2	7.0	6.8
		3	-7.0	-7.5
		4	3.9	4.4
		5	-.8	-1.1
		6	-.7	-.8
-4	3	1	-8.0	-8.2
		2	12.1	12.7
		3	-8.4	-10.6
		4	6.3	7.0
		5	-3.5	-3.7
		6	2.3	2.3
-4	4	1	3.8	3.8
		2	3.8	3.9
		3	-5.2	-5.6
		4	3.8	4.4
		5	-2.7	-2.9
-4	5	1	7.0	8.1
		2	-6.9	-7.7
		3	5.5	5.9
		4	-.7 U	-.2
		5	-.3 U	-.7
-4	6	1	7.6	8.1
		2	-6.9	-8.1
		3	4.5	5.1
		4	-.3 U	-1.1
-4	7	1	2.3	2.1
		2	-4.2	-4.5
-4	8	1	-4.8	-5.1
5	0	0	-13.9	-12.8
		1	6.3	5.4
		2	-1.3	-1.1
		3	1.3	1.1
		4	.9 U	.2
		5	1.2 U	1.2
		6	6.2	7.4
		7	-9.5	-9.7
		8	4.5	4.5
		9	-2.4	-2.3
		-1	4.9	4.8
		-2	-4.8	-5.2
		-3	7.9	8.3
		-4	-6.2	-7.0
		-5	5.9	6.8
		-6	-.9	-1.4
5	1	0	-16.7	-15.1
		1	17.3	16.3
		2	-16.8	-14.8
		3	2.6	2.6
		4	-2.4	-1.9
		5	5.2	5.5
		6	1.2 U	.7
		7	-1.2 U	-.5
		8	1.3	.7
		9	-.5 U	-.2
		-1	6.7	6.3
		-2	1.2	1.5
		-3	1.2 U	.2
		-4	-1.2 U	-.1
		-5	1.3	1.8
		-6	-.7 U	-.4
5	2	0	-4.2	-4.1
		1	5.2	5.4
		2	-5.5	-5.1
		3	2.5	2.5
		4	-7.1	-7.8
		5	7.7	7.7
		6	-6.7	-6.8
		7	4.4	5.9
		8	-.7 U	-.8
		-1	2.5	2.3
		-2	2.5	3.0
		-3	-3.9	-3.8
		-4	3.8	3.9
		-5	-3.9	-4.3
		-6	2.5	3.8
5	3	0	4.3	4.1
		1	-2.0	-1.1
		2	3.5	3.7
		3	-3.1	-3.7
		4	-2.7	-2.9
		5	5.3	5.9
		6	-8.2	-7.3
		7	3.3	3.4
		-1	-7.1	-6.4
		-2	4.0	5.2
		-3	-4.3	-4.5
		-4	5.2	5.7
		-5	-4.4	-5.4
5	4	0	9.4	9.0
		1	-3.9	-4.4
		2	2.5	2.5
		3	-3.7	-4.1
		4	4.0	4.2
		5	-.9 U	-.6
		6	-2.1	-1.6
		-1	-9.5	-10.3
		-2	6.3	6.9
		-3	-1.2 U	-1.5
		-4	.9 U	.2
		-5	-.7 U	-.2
5	5	0	3.0	2.5
		1	-2.1	-2.3
		2	4.9	4.8
		3	-7.0	-6.3
		4	4.4	4.5
		5	-2.9	-2.8
		-1	-1.6	-1.8
		-2	1.2 U	.8
		-3	2.5	3.0
		-4	-3.8	-4.0
5	6	0	-2.7	-3.3
		1	3.8	3.6
		2	1.2	1.0
		3	-3.0	-3.3
		4	1.0 U	2.3
		-1	2.3	2.8
		-2	-3.0	-2.9
		-3	2.3	2.6
5	-1	0	2.3	1.6
		1	-7.3	-6.9
		2	10.6	10.2
		3	-6.0	-4.7
		4	7.3	6.9
		5	-5.0	-5.2
		6	4.6	5.5
		7	-9.3	-10.0
		8	6.4	6.9
		9	-2.2	-2.1
5	-2	0	5.5	5.1
		1	-8.2	-7.7
		2	19.0	16.8
		3	-16.8	-15.8
		4	9.4	9.2
		5	-3.9	-4.1
		6	1.2 U	.2
		7	-1.2 U	-.4
		8	1.2 U	.5
		9	.8 U	.4
5	-3	0	6.9	6.6
		1	-9.4	-10.3
		2	7.9	7.8
		3	-.8	-.7
		4	-.9	-1.0
		5	-1.2 U	-.9
		6	-2.9	-3.2
		7	6.7	7.8
		8	-7.1	-6.8
		9	2.3	2.2
5	-4	0	2.1	1.5
		1	-1.2 U	-.2
		2	-6.8	-5.8
		3	10.4	10.4
		4	-7.8	-7.8
		5	3.8	3.9
		6	-6.4	-5.7
		7	6.2	6.9
		8	-7.1	-7.5
		9	3.5	3.6
5	-5	0	-6.3	-7.6
		1	4.0	5.3
		2	-2.6	-2.9
		3	8.2	8.5
		4	-11.9	-12.6
		5	4.7	5.3
		6	-2.6	-2.4
		7	2.3	2.6
		8	-1.8	-2.1
		9	.7	.7
5	-6	0	-5.6	-7.0
		1	4.2	5.3
		2	-4.2	-4.6
		3	2.7	3.1
		4	-4.0	-4.5
		5	-1.2 U	-.5
		6	3.2	3.8
		7	-2.4	-2.7
		8	4.2	4.8
5	-7	0	-3.8	-4.0
		1	2.2	2.2
		2	-1.2 U	-.4
		3	-5.8	-5.9
		4	5.0	5.2
		5	-2.2	-2.5
		6	6.4	6.2
		7	-4.7	-5.5
5	-8	1	-3.9	-4.5
		2	5.2	5.8
		3	-4.8	-5.6
		4	4.7	5.7
		5	-1.2	-1.6
		6	1.9	2.2
-5	1	1	.9 U	0.0
		2	-5.8	-6.2
		3	3.9	4.4
		4	-6.0	-6.5
		5	6.9	7.2
-5	2	1	-2.0	-1.5
		2	1.2	1.4
		3	-5.0	-5.7
		4	1.2 U	1.0
		5	.8 U	1.0
-5	3	1	-3.6	-3.3
		2	8.5	9.1
		3	-9.3	-10.2
		4	3.9	3.9
		5	-1.2	-1.1
-5	4	1	-2.5	-2.1
		2	6.0	6.5
		3	-6.4	-7.1
		4	2.9	3.8
-5	5	1	6.2	6.1
		2	-1.2 U	-1.0
		3	-.8 U	-.9
-5	6	1	7.1	7.5
		2	-2.2	-2.7
-5	7	1	1.5	1.9
6	0	0	-5.5	-5.7
		1	10.7	9.6
		2	-9.4	-8.7
		3	1.6	1.6
		4	-1.0	-1.2
		5	-1.2	-1.0
		6	1.2 U	.6
		7	-1.2 U	-.6
		8	3.0	4.4
		-1	6.4	5.9
		-2	-4.4	-4.6
		-3	5.2	5.8
		-4	-3.5	-3.6
6	1	0	-6.2	-5.3
		1	11.7	12.4
		2	-8.2	-7.7
		3	3.9	4.0
		4	-6.2	-6.7
		5	5.3	5.7
		6	-4.6	-4.6
		7	1.0 U	.1
		8	.5 U	.5
		-1	-1.2	-2.0
		-2	2.1	2.2
		-3	1.2	1.0
		-4	-.8 U	-1.1
6	2	0	-1.2	-1.6
		1	2.6	3.1
		2	1.2	.4
		3	-1.2	-.9
		4	-3.1	-3.4
		5	7.3	7.8
		6	-6.7	-6.9
		7	1.2	1.6
		-1	-2.6	-2.7
		-2	4.7	4.6
		-3	-3.5	-3.1
		-4	2.6	3.5
6	3	0	4.9	4.1
		1	-3.8	-3.7
		2	5.0	5.5
		3	-2.6	-3.2
		4	-1.2 U	-1.1
		5	2.3	2.5
		6	-1.2	-1.1
		-1	-5.3	-5.4
		-2	7.3	6.6
		-3	-3.0	-3.4
		-4	.5	.8
6	4	0	5.2	7.0
		1	-4.4	-4.3
		2	4.9	4.7
		3	-3.1	-3.3
		4	2.1	1.9
		5	-1.9	-2.5
		-1	-6.9	-6.0
		-2	1.3	1.5
		-3	.5 U	1.4
6	5	0	-.8 U	-.5
		1	.8 U	.4
		2	.8 U	.6
		3	-.8 U	-1.5
		-1	-.8 U	-.5
		-2	-.8 U	-1.8
6	-1	0	-1.0 U	-.1
		1	-1.0	-.9
		2	-3.2	-2.4
		3	-4.8	-4.3
		4	5.8	5.9
		5	-4.6	-4.4
		6	6.4	6.9
		7	-3.9	-3.8
		8	3.9	4.2
6	-2	0	2.7	2.7
		1	-7.3	-7.5
		2	4.6	4.4
		3	-4.2	-4.0
		4	9.4	9.0
		5	-10.5	-8.6
		6	9.7	8.2
		7	-3.1	-3.0
		8	-1.0 U	-.5
6	-3	0	8.7	8.3
		1	-7.6	-9.5
		2	7.0	6.7
		3	-2.6	-2.2
		4	5.2	4.9
		5	-2.6	-2.6
		6	-1.2	-1.4
		7	1.2	1.6
		8	-2.1	-2.6
6	-4	0	6.3	6.5
		1	-2.5	-2.5
		2	1.2	1.5
		3	1.6	1.7
		4	-3.7	-3.8
		5	4.9	5.2
		6	-6.8	-6.6
		7	5.9	5.6
		8	-3.9	-4.1
6	-5	0	-2.1	-1.7
		1	4.9	5.1
		2	-7.6	-7.1
		3	5.3	5.5
		4	-1.2	-2.1
		5	3.2	3.1
		6	-7.9	-7.4
		7	6.4	7.3
		8	-3.1	-3.3
6	-6	0	-2.9	-3.1
		1	6.4	7.0
		2	-7.3	-8.9
		3	5.0	5.8
		4	-1.2 U	-.4
		5	1.0 U	.1
		6	-2.9	-3.0
		7	1.0 U	.5
6	-7	1	2.0	2.9
		2	-2.5	-3.1
		3	2.3	2.8
		4	1.0 U	.1
		5	-3.7	-5.2
		6	2.3	3.5
-6	1	1	4.7	4.4
		2	-6.9	-6.7
		3	7.8	8.5
		4	-2.6	-3.2
-6	2	1	-3.5	-3.1
		2	1.2 U	.2
		3	1.6	1.6
		4	-.8 U	0.0
-6	3	1	-7.0	-6.6
		2	1.2	1.6
		3	-3.7	-3.4
-6	4	1	-5.8	-5.6
		2	2.9	2.6
		3	-2.6	-3.6
-6	5	1	1.0 U	1.2
		2	.8 U	1.0
7	0	0	-4.9	-5.3
		1	3.9	4.3
		2	-4.9	-3.6
		3	6.4	5.7
		4	-4.0	-4.5
		-1	5.2	5.4
		-2	-3.1	-3.5
7	1	0	-1.2 U	0.0
		1	-2.2 U	-.4
		2	-2.6 U	-1.6
		3	6.9	6.5
		4	-6.9	-7.4
		-1	1.9 U	1.2
		-2	1.9 U	0.0
7	2	0	1.0 U	2.0
		1	-2.0 U	-.5
		2	2.5 U	1.5
		3	2.1 U	.5
		4	-2.5	-2.6
		-1	-2.8	-3.8
		-2	2.9	4.0
7	3	0	2.9	3.3
		1	-3.7	-4.1
		2	4.4	4.3
		3	-1.9	-2.0
		4	1.6 U	2.0
		-1	-3.8	-4.6
		-2	1.0	1.3
7	-1	0	-5.8	-6.3
		1	3.9	4.5
		2	2.9 U	.6
		3	-2.5 U	-.1
		4	-2.7 U	-.5
7	-2	0	2.9	3.0
		1	-2.3	-1.7
		2	2.7 U	1.1
		3	-3.1	-2.9
		4	2.7	2.7
7	-3	0	6.9	6.8
		1	-6.6	-6.1
		2	2.7 U	2.6
		3	-7.0	-6.1
		4	4.9	4.3
7	-4	0	4.9	5.0
		1	-7.2	-7.0
		2	2.9	2.5
		3	-2.3 U	-1.4
		4	2.5	2.0
7	-5	0	1.8	2.0
		1	1.4 U	.6
		2	-4.6	-4.4
		3	4.9	4.7
-7	1	1	6.7	6.6

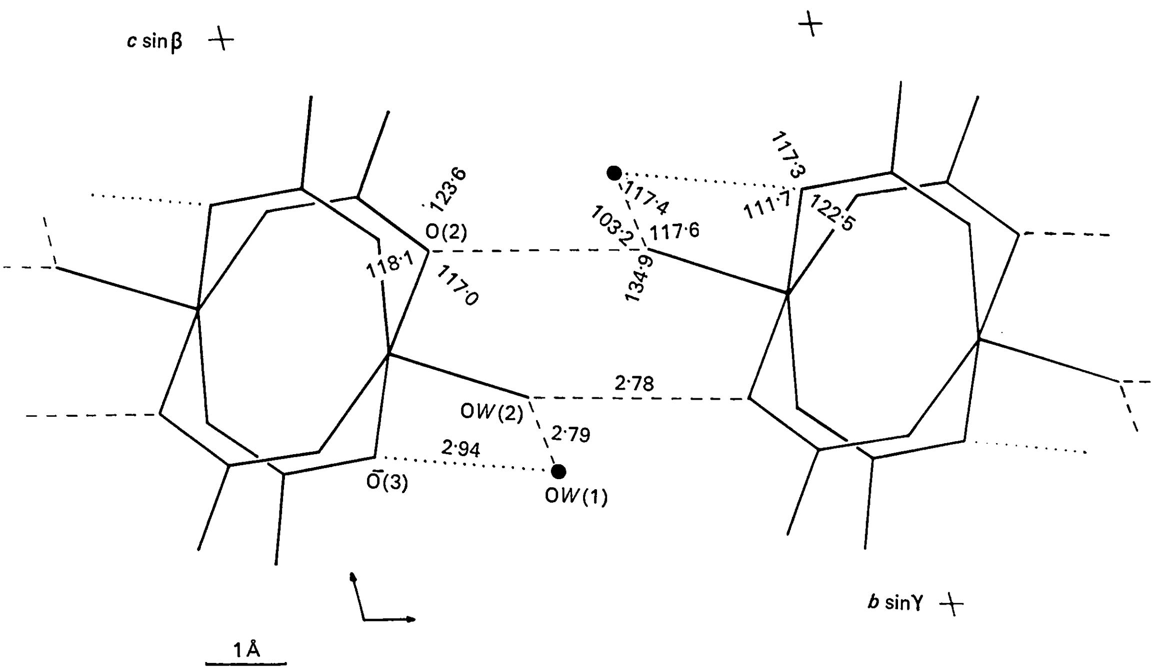


Fig. 5. Hydrogen bond system. The important distances and angles are indicated.

basic motion of copper(II) succinate dihydrate in the absence of such constraints would probably consist of rigid body vibrations of the carboxyl groups about the Cu–Cu vector. However it appears from Fig. 7 that the carboxyl groups do not execute such vibrations, although the atoms do undergo anisotropic motions. As expected, the carboxyl oxygen atoms and the two sp^3 carbon atoms librate about the appropriate covalent bonds. The sp^2 carbon atoms have comparable motions. The preferred mode of C(2) is approximately normal to the plane defined by O(4), C(2), C(4) and O(3). Similarly C(1) shows a strong preference to vibrate normal to its sp^2 plane.

The most interesting feature of the thermal analysis is that the water molecules both have elongated ellipsoids. The preferred vibration of O*W*(2) is almost perpendicular to the Cu–O*W*(2) bond. O*W*(1) also has a pronounced anisotropic motion even though it does not bond covalently. The preferred mode is in fact approximately normal to the plane defined by its hydrogen bonds. Hence the anisotropic motion of OW(1) can be ascribed to the constraining influence of its hydrogen bonds.

Helpful advice from Dr B. N. Figgis is gratefully acknowledged. One of us (B. H. O'Connor) is obliged to Imperial Chemical Industries of Australia and New Zealand Ltd for the award of a fellowship during the course of this study.

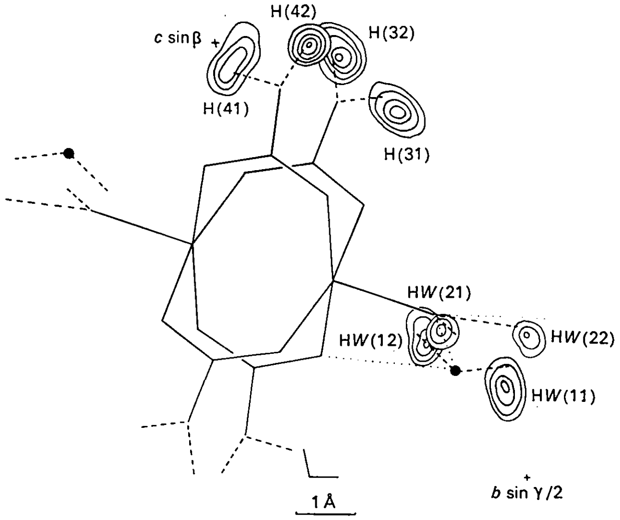


Fig. 6. Summary of the *a* axis view of the $\varrho_o - \varrho_c$ synthesis, hydrogen atom contributions being excluded from the ϱ_c computation. Contour interval: 0·1 Å^{-3}; zero contour at 0·3 e.Å^{-3}. The broken lines represent covalent bonds which involve hydrogen atoms and the dotted lines denote hydrogen bonds.

References

Asai, O., Kishita, M. & Kubo, M. (1959). *J. Phys. Chem.* **63**, 96.

Barclay, G. A., & Kennard, C. H. L. (1961). *J. Chem. Soc.* p. 5244.

Buerger, M. J. (1951). *Acta Cryst.* **4**, 531.

Buerger, M. J. (1953). *X-ray Crystallography*, Chap. 19, New York: John Wiley.

Cruickshank, D. W. J. (1956). *Acta Cryst.* **9**, 747.

Dauben, C. H. & Templeton, D. H. (1955). *Acta Cryst.* **8**, 841.

Delaunay, B. (1933). *Z. Kristallogr.* **84**, 109.

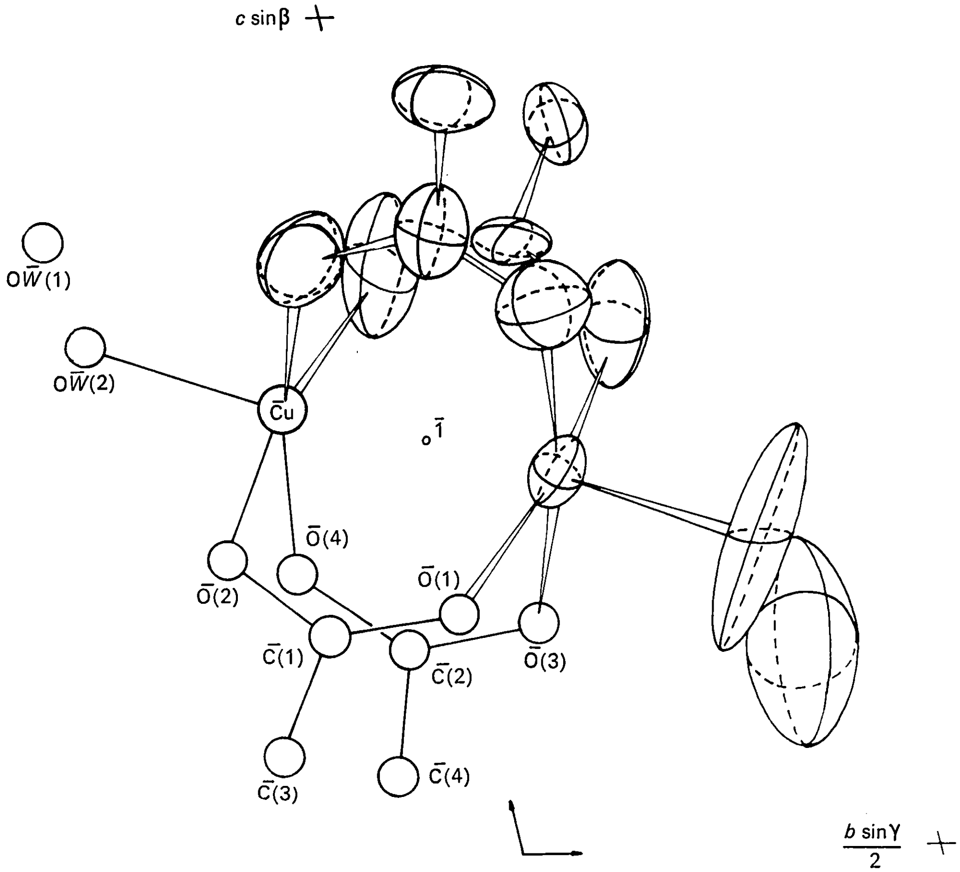


Fig. 7. Ellipsoids of vibration viewed down the *a* axis.

DONNAY, J. D. H. & NOWACKI, W. (1953). *Crystal Data*, p. 141, New York: The Geol. Soc. of America.

FARQUHAR, M. C. M. & LIPSON, H. (1946). *Proc. Phys. Soc. Lond.* **58**, 200.

FIGGIS, B. N. & LEWIS, J. (1964). *Progr. Inorg. Chem.* **6**, 37.

FIGGIS, B. N. & MARTIN, R. L. (1956). *J. Chem. Soc.* p. 3837.

FIGGIS, B. N. & MARTIN, D. J. (1965). In the press.

FORSTER, L. S. & BALLHAUSEN, C. J. (1962). *Acta Chem. Scand.* **16**, 1385.

GELLER, S. & DURAND, J. L. (1960). *Acta Cryst.* **13**, 325.

HANIC, F., ŠTEMPELOVÁ, D. & HANICOVÁ, K. (1964). *Acta Cryst.* **17**, 633.

HANSON, H. P., HERMAN, F., LEA, J. D. & SKILLMAN, S. (1964). *Acta Cryst.* **17**, 1040.

HOWELLS, E. R., PHILLIPS, D. C. & ROGERS, D. (1950). *Acta Cryst.* **3**, 210.

International Tables for X-ray Crystallography (1962). Vol. III, p. 201. Birmingham: Kynoch Press.

KATO, M., JONASSEN, H. B. & FANNING, J. C. (1964). *Chem. Rev.*, **64**, 99.

MAIR, G. A. (1962). Private communication.

NIEKERK, J. N. VAN, & SCHOENING, F.R.L. (1953). *Acta Cryst.* **6**, 227.

PAULING, L. (1962). *The Nature of the Chemical Bond.* London: Oxford Univ. Press.

PETERSON, S. W. & LEVY, H. A. (1957). *Acta Cryst.* **10**, 70.

RIETVELD, H. M. (1964). Ph. D. Thesis, University of Western Australia.

SIM, G. A. (1958). *Acta Cryst.* **11**, 123.

SUTTON, L. E. (1958). *Tables of Interatomic Distances and Configurations in Molecules and Ions.* London: The Chemical Society.