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A Refinement of the Structure of Bisglycino-Copper(II) Monohydrate, $Cu(NH_2CH_2COO)_2.H_2O$

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The crystal structure of bisglycino-copper(II) monohydrate, which was originally determined by Tomita & Nitta in 1961, has been refined by three-dimensional least-squares methods. The molecular geometry of this *cis* complex is essentially the same as reported earlier, but the standard deviations of the distances have been reduced to a third of their previous values. The groups C_α–CO–O of both glycine molecules are highly planar, the entire chelate part of the complex only approximately so. The environment of the water molecule is trigonal. An error analysis of the reflection data is presented.

1. Introduction

The crystal structure of bisglycino-copper(II) monohydrate was determined from Weissenberg projection data by Tomita & Nitta (1961). They showed that two glycine molecules are coordinated to the copper(II) ion, forming a planar *cis* configuration. The irregular octahedral coordination is completed by two oxygen atoms: one belonging to a water molecule and the other a 'free' carboxyl oxygen atom of an adjacent asymmetric unit.

At the time of publication of the Osaka paper (Tomita & Nitta, 1961), the structure had been solved independently in Sydney as part of a program of accurate structure analyses of metal–peptide complexes. Since complete three-dimensional data were available in Sydney, it was agreed that an accurate refinement should be carried out and submitted as a joint publication. Unfortunately a systematic comparison of the Osaka (TN) and Sydney (FS) observations was impossible because the original Osaka records had been lost.

In this paper we do not make a detailed comparison of our results with the three metal glycinate structures which have been previously reported: bisglycino-nickel(II) dihydrate (Stosick, 1945*a*) and bisglycino-cadmium and zinc monohydrates (Low, Hirshfeld & Richards, 1959). These are *trans* structures and were not refined to high precision. Other structure determinations of *trans* α-amino acid chelate complexes, in increasing order of precision, are those of bis-α-aminobutyrato-copper(II) (Stosick, 1945*b*), bis-DL-prolinato-copper(II) dihydrate (Mathieson & Welsh, 1953) and bis-1′-aminocyclopentanecarboxylato-copper(II) (Barclay & Stephens, 1963).

2. Experimental

Crystals prepared from a neutral solution of copper(II) ion and glycine always grow as fine, light blue needles which are unsuitable for structure analysis on account of their small size. Okaya, Pepinsky, Takeuchi, Kuroya, Shimada, Gallitelli, Stemple & Beevers (1957) first reported the space group and approximate unit-cell dimensions of such crystals, but did not proceed with structure analysis.

It was accidentally found that triglycine present in the preparation allowed much larger crystals to form. Apparently the triglycine–copper(II) complex is both more stable and soluble than the glycine complex. It buffers the copper(II) ion concentration at a low value, allowing comparatively large ($0·3\times0·15\times0·15$

Table 1. *Unit-cell data of Tomita & Nitta* (1961) *and Freeman & Snow for bisglycino-copper*(*II*) *monohydrate*

$CuC_4H_8N_2O_4.H_2O$ F.W. = 228·67

Orthorhombic	TN	FS
$a(\sigma a)$ =	10·78 (0·010)	$10·86_6$ (0·017) Å
$b(\sigma b)$ =	5·208 (0·004)	5·220 (0·007) Å
$c(\sigma c)$ =	13·47 (0·020)	$13·50_2$ (0·021) Å
U =	756·5	765·9 $Å^3$
D_m =	$1·99_8$	1·99 $g.cm^{-3}$
Z =	4	4
D_x =	2·01	1·98 $g.cm^{-3}$
μ =	—	42·6 cm^{-1}
λ(Cu $K\alpha$) =	1·542	1·5405(α_1), 1·5443(α_2) Å

Space group: $P2_12_12_1$ (from systematic absences).

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Table 2. *Summary of the details of the least-squares refinement*

Cycle Nos.	Matrix approximation	Temperature factor	Final R*	Weighting scheme	Treatment of F_{unobs}	Machine program	
1–3	Diagonal	Mean isotropic	0·146	$\sqrt{w}=1/\Delta(F_o)$ from A, Fig. 1	omitted	ZEBRA	Schoone (1961)
4–7	Diagonal	Individual isotropic	0·126	As above	omitted	ZEBRA	Schoone (1961)
8–9	Full matrix	Individual isotropic	0·129	$\sqrt{w}=1/\sigma(F_o)$ from B, Fig. 2	$F_u=\frac{1}{2}F_{\min}$ $\sigma(F_u)=F_{\min}/\sqrt{(12)}$	IBM 7090	Busing & Levy (1961)
10–16	Full matrix	Individual anisotropic	0·091	As above	As above	IBM 7090	Busing & Levy (1961)

* R: Cycles 1–7, observed reflections only; cycles 8–16, all reflections.

mm^3) crystals of bisglycino-copper(II) monohydrate to form slowly. The crystals are orthorhombic needles exhibiting predominantly the form {101}. TN prepared similar crystals by buffering the *p*H of the solution with carbon dioxide.

The two sets of unit-cell data are compared in Table 1.

The unit-cell dimensions of TN had been fitted to the diffraction angles of eight planes in the range $10° \leq 2\theta \leq 20°$. Their diffractometer measurements were calibrated only for zero error by means of silica powder lines.

The values determined by FS were fitted by least-squares to the spacings of the α_1 and α_2 components of twelve reflections at high angles on Weissenberg photographs calibrated with platinum powder lines. These values were used in the subsequent structure refinement.

A 0·29 mm long crystal of rhombic cross-section, with diagonals $0{\cdot}16 \times 0{\cdot}175$ mm^2, was rotated about the needle axis to obtain $h0l$ to $h4l$ multiple-film Cu $K\alpha$ equi-inclination Weissenberg data. For inter-layer scaling the $hk0$ and $0kl$ spectra were recorded with crystals $0{\cdot}15 \times 0{\cdot}22 \times 0{\cdot}175$ mm^3 and $0{\cdot}19 \times 0{\cdot}16 \times 0{\cdot}18$ mm^3; the last mentioned length corresponds to the rotation axis in each case. The intensities were estimated visually and corrected for Lorentz-polarization and Tunnel effects, but not for absorption.

The 93 reflections which were recorded twice were used to correlate 886 non-symmetry-related reflections (85% of the accessible data), of which 71 were too weak to be observed.

3. Refinement

Least-squares refinement was commenced with the TN positional parameters. Six observed and three unobserved reflections were given zero weights, so that 877 reflections were actually included. Details of the refinement are given in Table 2.

A revision of the weighting scheme was made after cycle 7 (see § 4). Corrections for extinction were made to the 18 largest structure amplitudes, all of low θ, after cycle 13. The correction factor for the F_o values, found by the method of Pinnock, Taylor & Lipson (1956), was $1/\sqrt{(1-kI_o)}$, where I_o is the observed intensity and k was determined to be 6×10^{-5}.

The scattering factors used were those of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for carbon, nitrogen, oxygen and copper. The Cu$^+$ curve was used for copper for reasons mentioned in previous communications (Freeman, Smith & Taylor, 1961; Freeman, Robinson & Schoone, 1964). The real part of the anomalous scattering by copper was allowed for by subtracting 2·1 electrons from the scattering curve over the whole sin θ range (Dauben & Templeton, 1955).

At the end of the least-squares refinement the unweighted and weighted reliability factors for all reflections were, respectively, $R=0{\cdot}091$ and $R'=0{\cdot}103$. The final r.m.s. shifts in the coordinates were $\delta x=0{\cdot}014$, $\delta y=0{\cdot}0002$, $\delta z=0{\cdot}0002$ Å. A final difference Fourier synthesis had only two peaks of probable significance: these peaks were close to the copper atom. They could be explained by only slight differences between the copper atom parameters required, respectively, by the Fourier and least-squares weighting schemes.

The final coordinates are shown in Table 3(*a*), and the thermal parameters in Table 3(*b*). The observed and calculated structure factors are compared in Table 4.

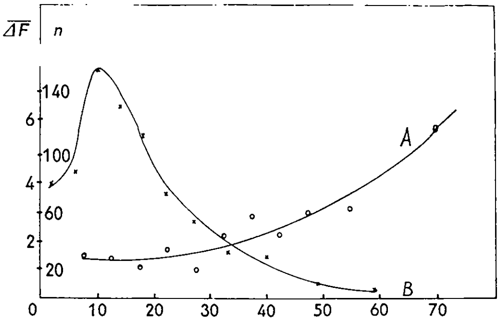

Fig. 1. A: The mean discrepancy $\overline{\Delta F}$ (average difference between two estimates of F_o in a range of F_o) plotted *versus* F_o. B: The population distribution of F_o: the number of reflections, n, in a range of F_o plotted *versus* F_o.

Table 3(*a*). *The positional parameters and their estimated standard deviations*

All values have been multiplied by 10^4

Atom	x	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$	Average s.d. (Å × 10^4)
Cu	1031	2	3403	4	4013	1	16
O(1)	813	8	5772	17	2903	6	82
O(2)	1242	9	6286	19	1314	6	90
O(3)	104	10	5568	18	4919	6	96
O(4)	−664	10	5797	22	6444	7	104
O(5_w)	2878	10	5558	28	4530	8	112
N(1)	1842	9	1152	21	3025	7	100
N(2)	1196	11	989	24	5176	8	110
C(1)	1938	13	2519	25	2074	10	124
C(2)	1285	9	5047	24	2085	9	114
C(3)	623	12	2162	34	6065	9	137
C(4)	−45	11	4676	26	5803	8	120

Table 3(*b*). *The final temperature factor parameters from the isotropic* (B) *and anisotropic* (B_{ij}) *refinements and their estimated standard deviations* (in Å^2)

The anisotropic temperature parameters have been multiplied by 10^4

Atom	B	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cu	1·86(0·05)	58(2)	161(7)	20(1)	17(3)	1(1)	4(2)
O(1)	1·86(0·17)	46(8)	179(35)	25(4)	13(13)	3(4)	−12(10)
O(2)	2·22(0·19)	78(9)	205(37)	23(4)	20(17)	12(5)	11(11)
O(3)	2·53(0·22)	102(10)	178(37)	23(4)	40(18)	9(6)	11(11)
O(4)	2·86(0·24)	84(10)	341(48)	31(5)	20(18)	20(6)	0(13)
O(5_w)	4·10(0·30)	74(10)	588(68)	50(6)	−88(22)	−35(7)	60(18)
N(1)	2·00(0·22)	36(8)	159(41)	32(5)	15(15)	−4(5)	11(13)
N(2)	2·74(0·27)	64(10)	266(51)	34(5)	37(21)	12(6)	28(14)
C(1)	2·50(0·30)	73(12)	130(46)	36(6)	1(20)	3(8)	6(15)
C(2)	1·55(0·23)	22(8)	129(44)	35(6)	−10(16)	−10(6)	2(2)
C(3)	2·42(0·28)	43(9)	476(79)	28(6)	5(22)	74(7)	32(20)
C(4)	1·62(0·24)	43(9)	219(51)	22(6)	1(19)	2(6)	−1(14)

Anisotropic temperature factor: $\exp[-(B_{11}h^2+B_{22}k^2+B_{33}l^2+2B_{12}hk+2B_{13}hl+2B_{23}kl)]$.

4. Weighting and error analysis

To obtain an estimate of the errors in the structure amplitudes $|F_o|$ (from here on called merely F_o), comparisons were made among the 93 structure amplitudes which were recorded twice. The variation of the mean discrepancy, $\overline{\Delta F}$ (average difference between the scaled observations in a range of F_o), with F_o is shown in Fig. 1. The population distribution of F_o based on all data is also given.

For the diagonal matrix calculations on the ZEBRA computer, a two-parameter weighting function $w = 200/\{200+(F_o-20)^2\}$ was fitted to a plot of $1/(\overline{\Delta F})^2$ *versus* F_o. The agreement between the weighting function and the inverse of curve A in Fig. 1 was best in the range $10<F_o<20$ near the population maximum. The weighting function diverged in the sense of higher weights at high values of F_o.

For the full-matrix calculations on an IBM 7090 computer, the observed reflections were given weights $w^{\frac{1}{2}}$, where $w^{-\frac{1}{2}}=\sigma(F_o)$ and the $\sigma(F_o)$ were read from a plot of $(1/\sqrt{2})\overline{\Delta F}$ *versus* F_o (Fig. 2, curve B). This doubled the weights which had been assigned in the diagonal matrix calculation, although the actual weighting function now more accurately represented the calculated weights. Our use of the 'population standard deviation' $\sigma(x)=\sqrt{[\Sigma(x_i-\bar{x})^2/(n-1)]}$ for small samples followed the method of Davies & Pearson (1934). For two observations of F_o, the standard deviation of $\sigma(F)$ is then $0{\cdot}603\,\sigma(F)$. The 'range estimate' of Tippett (1925), used by Ibers (1956), gives $\sigma(F)=0{\cdot}89F$ with standard deviation $0{\cdot}756\,\sigma(F)$.

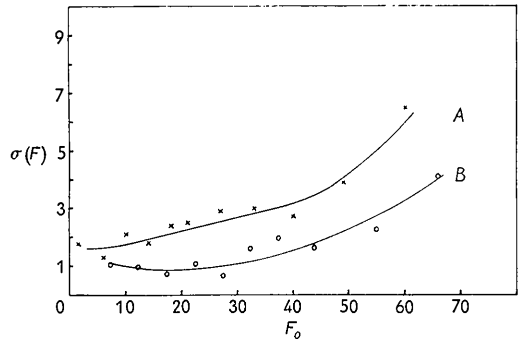

Fig. 2. A: $\sqrt{[\overline{|F_o-F_c|^2}]}$, the r.m.s. difference between the observed and calculated structure factors after cycle 13, found in ranges of F_o, plotted *versus* F_o. B: The standard deviation of F_o, $\sigma(F)=0{\cdot}707\overline{\Delta F}$, plotted *versus* F_o.

At the end of the refinement, the r.m.s. standard deviation in an observed structure amplitude of unit weight, $\sqrt{[\Sigma w|F_o-F_c|^2/(m-n)]}$, was found to be 2·14 instead of unity, the value expected when the weighting factors are given by $w=1/\sigma^2(F)$. This discrepancy

Table 4. *Observed and calculated structure amplitudes for bisglycino-copper(II) monohydrate*

Each group is headed by k and l. Subsequent lines contain h, F_{calc} and F_{obs}. The F_{calc} have been scaled by a factor of 1·0506. Values marked 'x' represent $\frac{1}{2}F_{\text{min}}$; values marked * represent $\frac{2}{3}F_{\text{min}}$; double-starred F's were excluded from least-squares cycles 8–16

k	l	h	F_{calc}	F_{obs}
0	0			
		2	65.92	54.82
		4	86.82	92.92
		6	45.07	41.38
		8	17.53	19.51
		10	42.48	41.21
		12	1.57	x 2.29
0	1			
		1	75.65	** 51.87
		2	71.75	62.32
		3	86.83	52.31
		4	57.07	56.25
		5	47.37	47.73
		6	40.35	40.35
		7	1.10	x 3.24
		8	22.77	27.93
		9	22.73	27.60
		10	.98	x 3.52
		11	13.23	16.47
		12	16.41	16.29
		13	11.63	19.37
0	2			
		0	35.08	33.59
		2	29.96	39.12
		3	61.17	51.97
		4	19.95	22.27
		5	20.61	24.84
		6	.70	x 2.95
		7	32.57	34.65
		8	28.56	31.60
		9	25.44	29.79
		10	11.31	13.90
		11	12.59	14.75
		12	1.44	x 3.24
		13	17.72	21.32
0	3			
		1	2.82	10.85
		2	22.42	15.08
		3	11.25	15.32
		4	7.93	13.42
		5	77.76	81.47
		6	14.61	15.66
		7	12.30	14.09
		8	19.96	22.94
		9	28.31	30.45
		10	15.02	15.32
		11	8.78	11.23
		12	4.95	x 3.24
		13	9.05	10.19
0	4			
		0	52.74	47.02
		1	39.99	36.07
		2	34.18	36.17
		3	6.67	5.43
		4	57.23	53.30
		5	16.28	22.37
		6	45.60	43.55
		7	31.08	30.93
		8	5.29	9.04
		9	2.55	x 3.90
		10	10.34	14.19
		11	20.57	22.37
		12	3.28	x 4.00
0	5			
		1	5.59	9.42
		2	45.79	36.45
		3	80.64	29.60
		4	18.48	26.08
		5	10.77	20.19
		6	29.57	31.12
		7	12.74	14.75
		8	29.51	30.55
		9	13.89	15.25
		10	8.17	11.80
		11	3.91	x 3.43
		12	6.35	9.99
0	6			
		0	35.12	26.08
		1	.83	x 2.35
		2	10.13	13.04
		3	32.40	32.93
		4	37.34	36.74
		5	9.51	11.99
		6	30.06	32.36
		7	23.63	30.05
		8	9.99	12.19
		9	4.64	x 3.90
		10	15.87	23.22
		11	11.24	12.28
0	7			
		1	39.42	39.59
		2	37.64	38.93
		3	36.76	36.54
		4	33.27	37.22
		5	56.69	54.25
		6	12.24	13.90
		7	8.52	10.76
		8	17.76	19.51
		9	32.66	32.08
		10	12.58	14.94
		11	4.34	x 3.05
0	8			
		0	23.51	24.46
		1	29.61	34.26
		2	32.16	36.64
		3	55.01	56.28
		4	21.00	25.98
		5	12.21	23.51
		6	12.93	20.19
		7	38.34	38.45
		8	15.41	15.80
		9	11.68	13.90
		10	.57	x 3.24
		11	12.71	15.04
0	9			
		1	37.31	40.45
		2	52.70	50.35
		3	36.39	35.59
		4	33.50	35.03
		5	34.79	35.03
		6	5.70	** 3.90
		7	22.25	25.32
		8	22.56	23.70
		9	5.95	9.14
		10	.75	x 3.24
		11	8.87	x 3.52
0	10			
		0	75.65	80.43
		1	14.90	19.23
		2	24.27	28.55
		3	10.30	11.61
		4	36.12	31.89
		5	14.53	18.37
		6	15.09	18.94
		7	4.50	x 3.24
		8	13.17	11.33
		9	6.82	x 3.24
		10	23.86	22.65
0	11			
		1	35.79	39.98
		2	56.27	53.02
		3	10.60	15.42
		4	15.33	22.27
		5	7.02	11.14
		6	13.17	16.94
		7	4.70	x 3.14
		8	20.04	22.56
		9	9.66	11.14
		10	6.29	8.09
0	12			
		0	4.62	x 6.00
		1	17.15	21.70
		2	20.93	23.51
		3	15.74	22.94
		4	8.54	9.53
		5	11.92	12.95
		6	3.86	3.14
		7	15.75	22.18
		8	11.27	12.56
		9	5.34	x 2.76
0	13			
		1	13.01	14.75
		2	10.17	11.71
		3	1.76	x 3.51
		4	8.56	5.66
		5	15.77	16.37
		6	.56	x 3.24
		7	5.54	x 2.95
		8	1.16	x 2.33
		9	9.57	10.23
0	14			
		0	1.26	x 2.25
		1	9.03	10.25
		2	3.50	x 3.62
		3	20.23	19.70
		4	13.91	14.37
		5	2.77	x 3.14
		6	14.72	15.90
		7	15.33	14.75
0	15			
		1	3.17	x 3.14
		2	30.29	25.93
		3	4.30	x 2.95
		4	14.15	15.13
		5	2.30	x 2.67
		6	16.32	15.13
0	16			
		0	20.19	19.99
		1	9.24	12.28
		2	6.57	5.43
		3	3.06	x 2.35
		4	13.55	14.56
		5	11.10	8.95
0	17			
		1	11.58	11.52
		2	6.39	7.71
1	0			
		1	55.96	44.45
		2	101.59	** 117.64
		3	42.93	39.59
		4	17.19	16.37
		5	6.40	9.04
		6	6.91	5.76
		7	40.29	37.12
		8	33.29	31.79
		9	2.67	x 2.57
		10	3.11	x 2.57
		11	14.27	14.47
		12	14.33	13.13
		13	1.01	x 2.09
1	1			
		3	99.15	95.39
		4	59.50	58.42
		5	22.26	23.32
		6	41.99	41.69
		7	9.86	11.99
		8	23.37	25.51
		9	14.59	19.23
		10	16.62	19.61
		11	20.55	21.70
		12	1.26	* 2.57
		13	10.57	10.37
1	2			
		0	6.49	9.42
		2	31.08	34.07
		3	52.62	49.11
		4	37.57	37.31
		5	55.59	52.82
		6	35.52	37.50
		7	13.87	15.27
		8	15.75	15.94
		9	27.92	27.70
		10	14.30	15.42
		11	10.72	11.50
		12	.79	** 2.57
		13	9.72	9.14
1	3			
		0	26.41	27.70
		1	10.95	11.23
		2	40.74	41.58
		3	54.33	52.25
		4	24.95	26.94
		5	31.84	34.07
		6	21.54	23.32
		7	51.92	49.59
		8	11.50	14.56
		9	8.72	9.90
		10	5.97	* 3.14
		11	11.25	11.42
		12	11.85	11.14
		13	13.25	11.90
1	4			
		0	66.23	65.24
		1	29.20	30.54
		2	48.53	47.59
		3	27.47	30.36
		4	55.96	57.01
		5	42.25	42.26
		6	10.18	12.85
		7	25.92	25.93
		8	21.67	21.70
		9	15.26	19.31
		10	8.49	10.76
		11	13.67	14.55
		12	12.37	11.42
		13	5.72	4.95
1	5			
		0	97.56	105.17
		1	27.46	29.74
		2	36.04	36.83
		3	43.04	45.07
		4	39.26	35.45
		5	24.58	27.95
		6	35.44	37.95
		7	6.33	5.90
		8	10.63	12.15
		9	18.43	17.42
		10	19.53	21.99
		11	4.56	* 2.76
		12	4.30	* 2.38
		13	5.56	7.42
1	6			
		0	20.05	21.89
		1	55.57	52.35
		2	42.69	41.21
		3	37.06	39.12
		4	28.34	29.59
		5	36.07	37.31
		6	20.40	23.60
		7	20.52	21.70
		8	19.37	28.65
		9	20.55	20.18
		10	4.90	* 2.95
		11	7.53	11.92
		12	8.03	9.61
1	7			
		0	5.53	7.42
		1	25.54	26.08
		2	37.50	35.53
		3	37.72	37.03
		4	3.38	5.14
		5	26.92	29.41
		6	5.54	9.61
		7	34.18	32.36
		8	16.93	17.70
		9	11.20	12.56
		10	2.54	* 2.86
		11	14.87	15.32
1	8			
		0	20.28	19.70
		1	23.96	26.65
		2	18.51	21.32
		3	17.09	20.15
		4	37.45	37.03
		5	36.24	35.12
		6	33.19	31.69
		7	6.75	5.95
		8	11.77	12.47
		9	22.00	18.46
		10	5.12	3.51
		11	10.99	10.15
1	9			
		0	39.75	37.95
		1	42.57	40.48
		2	11.47	13.99
		3	27.00	26.17
		4	31.04	29.59
		5	10.29	12.25
		6	23.54	24.46
		7	17.07	17.13
		8	12.80	12.25
		9	11.40	11.04
		10	5.95	9.23
		11	12.58	11.90
1	10			
		0	8.35	5.95
		1	11.69	13.90
		2	44.84	42.36
		3	39.56	39.55
		4	16.24	15.66
		5	5.71	4.47
		6	16.57	17.61
		7	19.59	19.37
		8	16.57	15.90
1	11			
		0	36.14	36.64
		1	25.18	27.22
		2	24.70	26.84
		3	4.67	* 3.14
		4	21.06	21.42
		5	6.32	* 3.14
		6	23.73	25.22
		7	6.93	5.65
		8	7.74	10.85
		9	15.16	16.94
		10	12.34	10.47
1	12			
		0	31.22	29.13
		1	13.58	15.32
		2	21.13	21.51
		3	11.61	12.25
		4	19.94	20.15
		5	16.71	17.50
		6	15.23	17.59
		7	3.37	5.43
		8	11.92	11.80
		9	11.59	11.61
1	13			
		0	12.59	13.71
		1	19.34	17.61
		2	9.68	11.23
		3	25.31	25.79
		4	6.15	5.90
		5	12.35	11.30
		6	5.45	5.43
		7	22.55	18.94
		8	5.25	6.76
1	14			
		0	17.20	15.37
		1	5.56	8.76
		2	20.62	15.27
		3	4.89	* 2.76
		4	20.40	19.23
		5	14.14	12.15
		6	9.54	10.47
		7	7.27	6.47
		8	10.56	7.50
1	15			
		0	23.30	21.80
		1	6.85	7.33
		2	15.65	15.32
		3	14.56	14.75
		4	12.60	13.13
		5	11.99	10.15
		6	13.47	11.90
1	16			
		0	11.53	11.42
		1	9.16	9.33
		2	17.29	15.80
		3	8.35	5.57
		4	8.83	8.57
1	17			
		0	3.57	x 2.00
		1	11.07	5.57
2	0			
		0	26.65	31.12
		1	16.26	22.15
		2	19.05	22.94
		3	5.44	5.55
		4	27.76	26.75
		5	27.16	28.93
		6	20.10	23.03
		7	4.79	5.14
		8	10.26	11.50
		9	27.64	25.13
		10	2.51	x 2.57
		11	7.59	x 2.76
		12	.12	x 1.90
2	1			
		0	4.91	x 3.52
		2	39.62	38.93
		3	54.96	47.75
		4	21.36	21.50
		5	11.55	13.42
		6	24.51	24.15
		7	34.56	31.31
		8	16.41	14.56
		9	10.99	9.61
		10	4.93	** 4.19
		11	10.77	11.33
		12	1.90	* 2.37
2	2			
		0	10.34	14.25
		1	21.10	24.54
		2	65.26	63.10
		3	25.25	27.22
		4	40.67	40.07
		5	22.06	22.75
		6	48.72	45.21
		7	31.36	30.36
		8	26.15	28.93
		9	4.47	* 2.95
		10	17.35	15.42
		11	12.51	12.75
		12	11.85	12.15
2	3			
		0	75.34	77.67
		1	49.74	47.02
		2	28.26	27.95
		3	36.55	37.03
		4	55.45	50.92
		5	23.11	23.99
		6	41.45	40.45
		7	20.43	20.05
		8	2.02	* 3.05
		9	21.83	20.75
		10	18.80	17.99
		11	11.44	11.90
		12	3.89	* 2.19
2	4			
		1	44.25	39.79
		2	65.99	71.10
		3	21.26	21.59
		4	24.92	25.13
		5	27.66	27.79
		6	14.70	16.37
		7	8.39	5.66
		8	24.15	25.32
		9	25.58	25.03
		10	7.33	8.66
		11	14.43	14.37
		12	10.06	8.47
2	5			
		0	6.59	6.85
		1	67.76	71.77
		2	42.13	39.79
		3	69.07	63.49
		4	30.04	34.36
		5	15.13	17.51
		6	7.20	8.47
		7	35.61	35.31
		8	15.69	19.04
		9	15.52	15.13
		10	7.33	9.33
		11	15.44	13.04
		12	9.12	8.57
2	6			
		0	33.67	34.26
		1	65.89	61.11
		2	47.54	41.75
		3	15.83	17.13
		4	20.93	21.51
		5	30.54	31.50
		6	23.25	24.65
		7	1.63	* 2.95
		8	12.99	13.71
		9	24.88	23.51
		10	4.83	5.43
		11	12.14	11.42
		12	10.19	9.90
2	7			
		0	54.45	49.40
		1	19.57	24.65
		2	8.72	9.61
		3	1.84	* 2.57
		4	22.64	21.70
		5	17.43	17.70
		6	24.74	24.08
		7	14.74	14.37
		8	3.16	* 3.05
		9	12.43	13.13
		10	21.42	15.56
		11	10.06	10.55
2	8			
		0	9.26	11.25
		1	15.02	15.15
		2	29.56	27.79
		3	17.37	15.46
		4	28.35	25.89
		5	6.14	5.09
		6	16.50	15.56
		7	18.90	17.89
		8	14.14	13.90
		9	4.16	8.00
		11	9.22	8.57
2	9			
		0	15.62	15.50
		1	18.52	19.04
		2	10.93	12.15
		3	16.33	17.61
		4	21.02	20.75
		5	9.83	9.42
		6	18.42	18.15
		7	20.77	20.37
		8	15.35	14.75
		9	9.15	9.50
		10	6.06	7.04
		11	13.58	9.14
2	10			
		0	16.52	17.13
		1	22.16	22.56
		2	7.25	6.00
		3	9.20	10.47
		4	11.03	11.04
		5	35.83	31.69
		6	15.60	15.51
		7	.33	* 2.56
		8	9.10	9.42
		9	15.63	13.13
		10	6.29	6.76
2	11			
		0	6.51	7.90
		1	13.96	14.66
		2	9.50	11.71
		3	17.18	15.90
		4	14.99	14.15
		5	2.52	5.90
		6	9.66	9.52
		7	18.90	18.75
		8	10.40	10.56
		9	8.77	6.47
2	12			
		0	2.73	x 2.47
		1	19.52	18.55
		2	28.72	25.59
		3	15.26	14.37
		4	25.53	23.51
		5	16.78	15.50
		6	17.29	16.75
		7	9.97	9.61
		8	15.17	13.13
		9	5.79	5.91
2	13			
		0	35.39	35.12
		1	5.99	10.85
		2	5.73	5.71
		3	5.26	5.00
		4	29.92	26.94
		5	15.37	17.23
		6	16.75	17.23
		7	6.65	6.47
		8	5.04	5.43
2	14			
		0	20.61	20.55
		1	23.55	22.56
		2	22.14	22.08
		3	8.15	8.33
		4	8.37	8.57
		5	16.60	14.56
		6	9.06	9.33
		7	7.32	4.95
2	15			
		0	4.07	5.14
		1	11.59	10.15
		2	11.49	9.90
		3	14.47	13.61
		4	7.96	5.47
2	16			
		0	7.47	7.90
		1	14.25	11.90
		2	10.22	9.14
		3	8.09	6.19
3	0			
		1	23.54	26.05
		2	3.72	4.66
		3	51.33	45.16
		4	10.52	15.99
		5	3.90	x 1.62
		6	3.32	6.47
		7	35.06	34.17
		8	3.31	x 2.00
		9	8.56	7.71
		10	2.63	3.52
		11	11.15	5.57
3	1			
		0	3.22	6.57
		1	23.32	9.71
		2	30.83	26.56
		3	29.73	29.70
		4	31.65	32.55
		5	39.61	35.85
		6	15.47	17.23
		7	13.59	15.23
		8	20.22	16.09
		9	15.57	11.33
		10	5.04	5.43
		11	6.19	5.23
		12	6.62	7.61
3	2			
		0	54.52	47.75
		1	25.13	21.80
		2	33.50	28.17
		3	26.41	23.70
		4	33.36	30.55
		5	8.22	5.09
		6	25.32	27.79
		7	14.76	13.52
		8	20.19	17.04
		9	3.25	* 2.57
		10	17.35	14.47
		11	5.02	4.66
		12	2.65	2.95
3	3			
		0	8.66	9.71
		1	14.69	16.37
		2	71.92	69.01
		3	13.02	13.61
		4	26.01	26.46
		5	13.50	13.99
		6	35.70	29.41
		7	7.13	8.35
		8	25.57	23.51
		9	10.79	9.14
		10	8.13	7.04
		11	1.34	* 1.52
		12	11.05	11.42
3	4			
		0	39.22	40.26
		1	39.03	38.55
		2	14.48	16.75
		3	44.87	41.31
		4	21.53	22.05
		5	19.35	21.23
		6	15.31	15.50
		7	30.35	27.22
		8	4.04	6.57
		9	3.63	6.95
		10	14.50	11.33
		11	14.36	11.71
		12	5.52	5.71
3	5			
		0	8.15	9.71
		1	36.27	38.93
		2	23.66	24.27
		3	19.03	19.99
		4	4.49	3.71
		5	30.70	29.70
		6	5.06	7.04
		7	10.84	13.94
		8	13.15	16.56
		9	15.93	16.19
		10	4.15	4.47
		11	15.35	11.71
3	6			
		0	23.92	24.37
		1	34.70	37.31
		2	6.99	9.42
		3	35.49	36.64
		4	6.20	8.09
		5	25.83	29.03
		6	14.00	15.04
		7	22.20	21.13
		8	7.52	7.80
		9	6.99	8.00
		10	12.40	11.42
		11	10.43	7.90
3	7			
		0	11.37	13.42
		1	22.42	24.27
		2	45.83	44.64
		3	16.17	17.04
		4	25.82	26.35
		5	21.97	21.59
		6	23.52	24.54
		7	5.70	7.04
		8	15.58	17.99
		9	10.79	11.33
		10	3.73	3.05
3	8			
		0	51.93	50.54
		1	16.34	15.94
		2	16.62	18.94
		3	13.56	17.89
		4	25.87	29.60
		5	8.01	8.47
		6	17.56	15.08
		7	9.31	10.47
		8	15.89	15.61
		9	2.54	5.71
		10	14.51	13.23
3	9			
		0	2.56	4.09
		1	21.29	24.54
		2	17.77	19.50
		3	14.15	14.66
		4	16.59	16.66
		5	25.38	25.13
		6	5.74	7.52
		7	.86	3.52
		8	11.76	14.09
		9	13.08	12.15
3	10			
		0	7.96	5.25
		1	14.09	16.37
		2	7.24	9.99
		3	22.30	23.89
		4	8.94	11.83
		5	2.50	5.71
		6	13.02	16.66
		7	20.72	19.42
		8	4.56	6.09
		9	8.68	8.28
3	11			
		0	7.38	5.95
		1	17.08	20.18
		2	12.57	14.47
		3	14.33	14.56
		4	11.94	14.37
		5	25.20	26.54
		6	9.15	5.95
		7	5.54	5.09
		8	9.05	5.09
3	12			
		0	26.72	27.41
		1	10.01	10.09
		2	10.99	11.42
		3	9.39	11.23
		4	22.59	23.41
		5	2.12	* 1.52
		6	17.82	17.38
		7	7.10	6.00
		8	5.38	4.09
3	13			
		0	4.45	4.28
		1	10.66	11.71
		2	24.10	23.89
		3	8.99	9.23
		4	12.50	13.33
		5	3.72	4.95
		6	12.59	12.18
3	14			
		0	20.76	20.37
		1	10.87	12.37
		2	5.76	4.95
		3	13.85	14.25
		4	11.59	12.15
		5	6.16	6.25
3	15			
		0	.27	x 1.14
		1	15.13	16.56
		2	6.41	5.43
		3	9.72	7.90
4	0			
		0	35.50	37.31
		1	46.63	51.49
		2	5.13	5.43
		3	7.10	8.00
		4	34.09	32.93
		5	35.52	31.60
		6	19.84	18.15
		7	6.44	7.42
		8	5.22	8.57
		9	11.91	11.23
		10	9.45	5.09
		11	10.77	10.76
4	1			
		0	31.13	32.54
		1	29.54	35.45
		2	25.82	24.84
		3	22.97	22.37
		4	14.53	14.47
		5	12.72	12.37
		6	16.15	15.23
		7	17.13	13.50
		8	10.65	5.76
		9	6.30	7.33
		10	10.90	9.14
		11	13.86	11.71
4	2			
		0	11.03	13.99
		1	22.52	21.99
		2	38.74	32.17
		3	21.62	22.18
		4	29.95	26.17
		5	5.32	3.62
		6	13.30	11.04
		7	12.57	11.23
		8	18.40	15.51
		9	12.33	10.76
		10	4.30	5.14
		11	4.08	5.51
4	3			
		0	31.57	35.55
		1	29.19	33.22
		2	11.06	11.42
		3	12.90	12.66
		4	14.23	13.13
		5	19.56	17.61
		6	10.04	10.25
		7	7.40	6.95
		8	10.52	9.04
		9	16.23	13.23
		10	12.71	11.99
		11	2.05	5.51
4	4			
		0	27.33	30.17
		1	9.54	13.23
		2	8.37	9.80
		3	18.16	17.99
		4	21.42	18.75
		5	21.53	19.04
		6	14.49	12.25
		7	16.20	17.42
		8	10.00	10.47
		9	6.04	6.47
		10	7.83	8.25
4	5			
		0	15.57	18.56
		1	10.27	11.33
		2	4.91	9.42
		3	17.05	17.51
		4	16.83	15.04
		5	8.61	5.76
		6	10.20	10.56
		7	17.96	17.89
		8	9.87	10.25
		9	3.49	4.85
		10	2.90	4.76
4	6			
		0	8.17	9.42
		1	5.38	9.99
		2	16.35	15.15
		3	19.05	19.23
		4	14.46	16.75
		5	25.05	23.28
		6	13.31	11.99
		7	10.22	11.33
		8	5.93	10.27
		9	6.72	6.95
		10	7.31	7.04
4	7			
		0	23.14	24.94
		1	25.73	27.13
		2	27.26	29.60
		3	14.98	17.61
		4	20.38	20.27
		5	21.37	23.32
		6	14.92	15.08
		7	5.92	6.66
		8	7.52	7.52
		9	15.04	15.61
4	8			
		0	13.67	15.13
		1	27.61	29.22
		2	26.88	25.17
		3	22.14	22.46
		4	15.19	15.94
		5	5.37	10.25
		6	10.74	11.61
		7	13.64	15.42
		8	18.08	18.15
		9	5.19	9.14
4	9			
		0	25.29	25.89
		1	17.22	15.37
		2	20.84	22.18
		3	21.25	23.13
		4	14.79	16.47
		5	4.43	7.61
		6	13.28	17.04
		7	11.55	13.52
		8	7.66	7.90
4	10			
		0	11.63	11.99
		1	21.49	22.18
		2	1.68	* 1.14
		3	9.07	11.04
		4	12.41	15.13
		5	14.79	17.70
		6	9.29	5.95
		7	3.80	5.51
4	11			
		0	17.37	18.56
		1	9.96	11.52
		2	18.63	19.23
		3	16.69	18.66
		4	9.26	10.65
		5	7.94	5.95
		5	13.27	13.23
4	12			
		0	2.95	3.43
		1	10.55	12.56
		2	12.57	14.55
		3	9.39	11.23
		4	5.23	9.42
4	13			
		0	14.25	15.70
		1	8.95	10.55
		2	5.74	5.76
		3	3.37	3.71
		4	6.59	7.52
4	14			
		0	9.04	7.42
5	1			
		0	36.81	37.41
5	2			
		0	12.81	15.99
5	3			
		0	14.56	16.75
5	4			
		0	4.04	4.95
5	5			
		0	11.89	14.37
5	6			
		0	18.35	19.23
5	7			
		0	5.53	6.85
5	8			
		0	2.21	x 2.09
5	9			
		0	22.51	22.75
5	10			
		0	2.14	x 3.24
5	11			
		0	11.15	9.99
5	0			
		1	11.75	12.47
		2	24.10	28.36
		3	7.18	5.09
		4	15.57	18.46
		5	8.14	9.33
		6	14.84	15.61
		7	4.88	6.47
		8	11.95	11.33
6	0			
		0	9.48	12.15
		1	2.35	x 2.09
		2	4.47	4.55
		3	5.72	6.28
		4	17.31	15.15
		5	6.56	5.00
		6	8.30	6.55
6	1			
		0	.60	x 1.90
6	2			
		0	7.59	9.04
6	3			
		0	7.34	9.71
6	4			
		0	21.04	19.99
6	5			
		0	5.16	5.55
6	6			
		0	20.87	22.15

might be attributed to factors such as the presence of systematic errors in the experimental data.

On the other hand, the function

$$\surd[\Sigma|F_o-F_c|^2/\Sigma(\Delta F)^2]\,,$$

which was derived in intervals of increasing F_o from the curves in Fig. 2, was found to be $2{\cdot}15 \pm 0{\cdot}035$. This is consistent with the greater part of the discrepancy being attributed to an over-estimation of the weighting factors by a factor of about two. This corresponds to an under-estimate of $\sigma(F)$ from the limited selection of the doubly estimated intensities, a result which is not unexpected. (An error only in the *scale* of the weights does not affect the final parameters or their calculated standard deviations in the computational method used in this refinement).*

Aspects of the two-dimensional (TN) and three-dimensional (FS) refinements of the structure are compared below:

	2-dimensional	3-dimensional
Number of reflections	254	877
Final R value	0·165	0·091
$\bar{\sigma}(l)$* (Cu–O)	0·03 Å	0·010 Å
$\bar{\sigma}(l)$* (Cu–N)	0·03	0·010
$\bar{\sigma}(l)$* (O–C)	0·05	0·015
$\bar{\sigma}(l)$* (N–C)	0·06	0·016
$\bar{\sigma}(l)$* (C–C)	0·06	0·017

* TN: average e.s.d.'s from formulae of Cruickshank (1949). FS: average e.s.d.'s from final least-squares cycle.

The bond lengths found by TN do not differ by more than 1·5 e.s.d.'s from the new values. The e.s.d.'s, however, have been reduced to less than one-third of their previous values by the three-dimensional refinement.

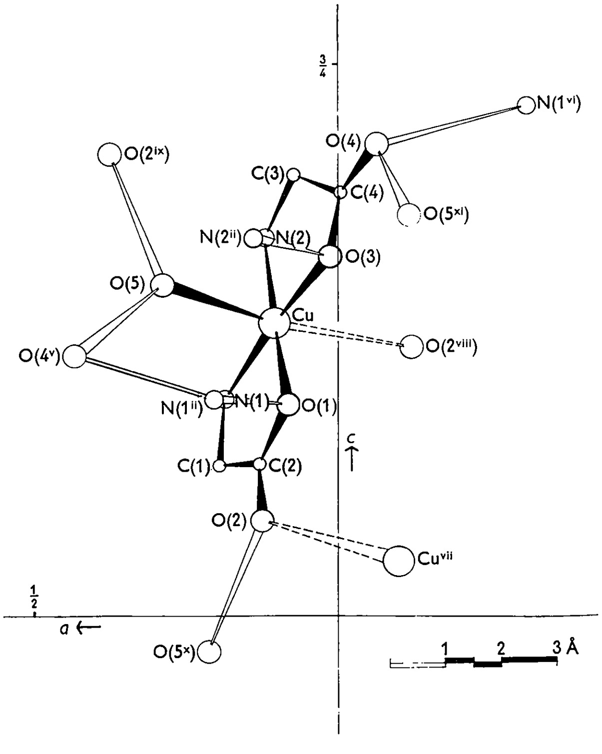

Fig. 3. Bisglycino-copper(II) monohydrate: the asymmetric unit at (x, y, z) with its immediate environment. Covalent bonds, ———; hydrogen-bonds, ═══; weak copper–oxygen interactions, ====.

5. Discussion of the structure

Fig. 3 illustrates the asymmetric unit at (x, y, z) and its immediate environment. Interatomic distances and intervector angles are listed in Tables 5 and 6. Arabic numerals have been used to distinguish atoms of the same type within an asymmetric unit and Roman superscripts for atoms not belonging to the asymmetric unit at (x, y, z).

Table 5. *Interatomic distances and their estimated standard deviations*

Distance	d	$\sigma(d)$
Cu–O(1)	1·95$_7$ Å	0·009 Å
Cu–O(3)	1·94$_6$	0·009
Cu–N(1)	1·98$_4$	0·010
Cu–N(2)	2·02$_1$	0·011
Cu–O(5)	2·40$_4$	0·011
N(1)–C(1)	1·47$_3$	0·016
N(2)–C(3)	1·48$_4$	0·017
C(1)–C(2)	1·49$_8$	0·017
C(3)–C(4)	1·54$_1$	0·018
C(2)–O(1)	1·27$_5$	0·014
C(2)–O(2)	1·22$_6$	0·015
C(4)–O(3)	1·29$_1$	0·015
C(4)–O(4)	1·24$_3$	0·015
Cu · · · O(2$^{\text{viii}}$)	2·74$_2$	0·009
N(1) O(1)	2·66$_3$	0·014
N(2) O(2)	2·69$_1$	0·016
N(1) N(2)	2·98$_9$	0·014
O(1) O(2)	2·83$_1$	0·012

Hydrogen bond distances

Distance	Equivalent distance	d	$\sigma(d)$
O(5) · · · O(2$^{\text{ix}}$)	O(2) · · · O(5$^{\text{x}}$)	2·76$_5$ Å	0·014 Å
O(5) · · · O(4$^{\text{v}}$)	O(4) · · · O(5$^{\text{xi}}$)	2·80$_4$	0·015
O(1) · · · N(1$^{\text{ii}}$)	N(1) · · · O(1$^{\text{iii}}$)	3·02$_7$	0·013
O(3) · · · N(2$^{\text{ii}}$)	N(2) · · · O(3$^{\text{iii}}$)	3·08$_6$	0·015
N(1) · · · O(4$^{\text{iv}}$)	O(4) · · · N(1$^{\text{vi}}$)	2·98$_2$	0·014

Shortest non-bonded inter-complex distances

O(1) · · · C(1$^{\text{vii}}$)	C(1) · · · O(1$^{\text{viii}}$)	3·13 Å
O(1) · · · N(1$^{\text{vii}}$)	N(1) · · · O(1$^{\text{viii}}$)	3·15
O(1) · · · C(2$^{\text{vii}}$)	C(2) · · · O(1$^{\text{viii}}$)	3·19

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* Dr J. A. Ibers has pointed out to us that even if the standard deviations of the observed structure amplitudes had been on an absolute scale, we blundered by using incorrect values for some of the F_u and $\sigma(F_u)$ (Table 2). For 36 unobserved reflections in centric projections, our values were correct. For 32 general reflections we used $|F_u| = 0{\cdot}5F_{\min}$ instead of $0{\cdot}67F_{\min}$ and $\sigma(F_u) = F_{\min}/\surd 12$ instead of $F_{\min}/\surd 18$ (Ibers, 1956). Owing to the over-weighting of the $808F_o$, the 32 slightly incorrect values of F_u were included with too *low* relative weights. The effect on the final parameters is considered to be negligible.

The asymmetric units are denoted as follows:

(i)	$\bar{x}$	y	z
(ii)	x	$1+y$	z
(iii)	x	$-1+y$	z
(iv)	$\frac{1}{2}+x$	$\frac{1}{2}-y$	$1-z$
(v)	$\frac{1}{2}+x$	$1\frac{1}{2}-y$	$1-z$
(vi)	$-\frac{1}{2}+x$	$\frac{1}{2}-y$	$1-z$
(vii)	$\bar{x}$	$\frac{1}{2}+y$	$\frac{1}{2}-z$
(viii)	$\bar{x}$	$-\frac{1}{2}+y$	$\frac{1}{2}-z$
(ix)	$\frac{1}{2}-x$	$1-y$	$\frac{1}{2}+z$
(x)	$\frac{1}{2}-x$	$1-y$	$-\frac{1}{2}+z$
(xi)	$-\frac{1}{2}+x$	$1\frac{1}{2}-y$	$1-z$

Molecules are linked in spirals parallel to *b* by bonds from carboxyl oxygen atoms to copper(II) ions (O(2$^{\text{viii}}$) · · · Cu, O(2) · · · Cu$^{\text{vii}}$ *etc.* in Fig. 3). This copper–oxygen interaction and the coordination of one water molecule make the copper(II) ion coordination sixfold. Chelate molecules related by the lattice translation *b* are linked by two hydrogen bonds from amino nitrogen to carboxyl oxygen (N(1) · · · O(1) = 3·03 Å, N(2) · · · O(3) = 3·09 Å). The spiral chains are cross-linked by hydrogen bonds from water to carboxyl oxygen (O(5) · · · O(4$^{\text{v}}$) = 2·80 Å, O(5) · · · O(2$^{\text{ix}}$) = 2·76 Å. There is also a hydrogen bond, N(1) · · · O(4$^{\text{iv}}$) = 2·99 Å, acting chiefly parallel to (101).

Table 6. *Intervector angles and their estimated standard deviations*

Angle	θ	$\sigma(\theta)$
O(1)–Cu–N(1)	85·0°	0·4°
O(3)–Cu–N(2)	85·4	0·4
N(1)–Cu–N(2)	96·6	0·4
O(1)–Cu–O(3)	92·9	0·4
N(1)–Cu–O(2$^{\text{viii}}$)	93·0	0·4
N(2)–Cu–O(2$^{\text{viii}}$)	87·3	0·4
O(1)–Cu–O(2$^{\text{viii}}$)	91·2	0·4
O(3)–Cu–O(2$^{\text{viii}}$)	82·5	0·4
O(5)–Cu–O(2$^{\text{viii}}$)	170·9	0·4
N(1)–Cu–O(5)	95·9	0·4
N(2)–Cu–O(5)	89·6	0·4
O(1)–Cu–O(5)	91·7	0·4
O(3)–Cu–O(5)	88·8	0·4
Cu–N(1)–C(1)	109·3	0·8
Cu–N(2)–C(3)	109·6	0·8
Cu–O(1)–C(2)	115·3	0·8
Cu–O(3)–C(4)	115·8	0·8
N(1)–C(1)–C(2)	112·6	1·0
N(2)–C(3)–C(4)	111·3	1·0
C(1)–C(2)–O(1)	117·4	1·0
C(1)–C(2)–O(2)	118·3	1·0
O(1)–C(2)–O(2)	124·3	1·0
C(3)–C(4)–O(3)	117·5	1·0
C(3)–C(4)–O(4)	119·7	1·0
O(3)–C(4)–O(4)	122·8	1·0
Cu$^{\text{vii}}$–O(2)–C(2)	112·6	0·8
Hydrogen bond angles	θ	$\sigma(\theta)$
Cu–O(5) · · · O(2$^{\text{ix}}$)	112·3°	0·8°
Cu–O(5) · · · O(4$^{\text{v}}$)	130·8	0·8
O(2$^{\text{ix}}$)–O(5) · · · O(4$^{\text{v}}$)	116·7	0·9
Cu–N(1) · · · O(1$^{\text{iii}}$)	115·0	0·8
Cu–N(1) · · · O(4$^{\text{iv}}$)	116·3	0·8
C(1)–N(1) · · · O(1$^{\text{iii}}$)	115·4	1·0
C(1)–N(1) · · · O(4$^{\text{iv}}$)	108·1	1·0
Cu–N(2) · · · O(3$^{\text{iii}}$)	116·8	0·8
C(3)–N(2) · · · O(3$^{\text{iii}}$)	108·0	0·8
C(2)–O(1) · · · N(1$^{\text{iii}}$)	100·0	1·0
Cu–O(1) · · · N(1$^{\text{iii}}$)	120·0	0·8
C(2)–O(2) · · · O(5$^{\text{x}}$)	122·9	1·0
Cu$^{\text{vii}}$–O(2) · · · O(5$^{\text{x}}$)	108·2	0·8
Cu–O(3) · · · N(2$^{\text{iii}}$)	113·8	0·8
C(4)–O(3) · · · N(2$^{\text{iii}}$)	106·0	1·0
C(4)–O(4) · · · N(1$^{\text{vi}}$)	119·9	1·0
C(4)–O(4) · · · O(5$^{\text{xi}}$)	107·4	1·0
N(1$^{\text{vi}}$) · · · O(4) · · · O(5$^{\text{xi}}$)	80·2	0·9

The ligands

The carboxyl oxygen atoms not strongly bound to the copper(II) ion, O(2) and O(4), each have two weak interactions (Fig. 3), whereas of the two amino nitrogen atoms, N(2) has one hydrogen bond and N(1) has two. Although the glycine ligands thus have different environments, the greatest difference between them occurs in the carbon–carbon bond-lengths (0·04 Å or 2·2$\sigma(d)$). This is not considered significant.

Table 7. *Details of plane of best fit*

Each plane is represented by $lx'+my'+nz'+k=0$ with respect to orthogonal axes, where x', y', z' are coordinates in Å

Plane	l	m	n	k
1	+0·856039	+0·456178	+0·243102	−3·081509
2	+0·806339	+0·511710	+0·296596	−3·544961
3	+0·865199	+0·437638	+0·244753	−3·021608

Atom	Deviation from plane (Å) 1	2	3	Coordinate s.d. (σ) (Å)	$w=K/\sigma^2$
Cu	+0·0059*	−0·1256*	+0·0512*	0·0016	0
O(1)	+0·0003		+0·0189	0·0082	131
O(2)	+0·0005			0·0090	111
O(3)		+0·0028	−0·0268	0·0096	100
O(4)		+0·0034		0·0104	88
N(1)	−0·1027*		−0·0288	0·0100	89
N(2)		−0·1623*	+0·0368	0·0110	63
C(1)	+0·0000$_2$			0·0124	47
C(2)	−0·0010			0·0114	67
C(3)		+0·0054		0·0137	43
C(4)		−0·0088		0·0120	63

The starred deviation values correspond to atoms not included in the least-squares plane.

Angles between planes

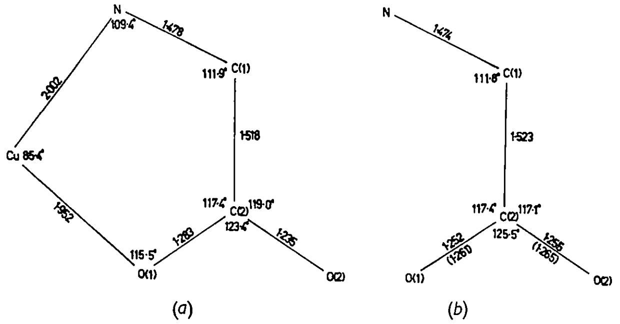
Plane	1	2	3
2	5·4°	—	6·2°
3	0·2°	6·2°	—

The C_α–CO–O groups are planar (planes 1, 2 of Table 7). The greatest deviation is 0·009 Å of atom C(4) from plane 2. The angle between planes 1 and 2 is 5·4°, indicating that the chelate part of the complex is only approximately planar. The amino nitrogen atoms N(1) and N(2) deviate from planes 1 and 2 by 0·103 Å and 0·162 Å respectively. The tetrahedral environment of N(1) with two hydrogen bonds is more symmetrical than that of N(2) with one hydrogen bond. This qualitatively accounts for the difference of 0·059 Å between their respective deviations.

Table 8. *Parameters of thermal vibration ellipsoids*

μ_i, r.m.s. component of thermal displacement along principal axes i (Å × 10^3); θ_{ik} angles with respect to the cell axes k (degrees); φ_i, angles between principal axes and the normal to the plane of the coordination square (degrees). Estimated standard deviations in these quantities are also given

Atom	Axis i	$\mu_i(\sigma\mu_i)$	$\theta_{i1}(\sigma\theta_{i1})$	$\theta_{i2}(\sigma\theta_{i2})$	$\theta_{i3}(\sigma\theta_{i3})$	$\varphi_i(\sigma\varphi_i)$
Cu	1	133(3)	84(4)	113(11)	24(12)	81(3)
	2	145(4)	71(3)	149(9)	113(12)	100(3)
	3	191(3)	20(3)	71(3)	87(2)	13(3)
O(1)	1	134(16)	113(17)	51(18)	48(20)	84(17)
	2	165(13)	111(64)	64(51)	137(26)	117(70)
	3	174(14)	41(57)	50(43)	98(53)	27(69)
O(2)	1	134(13)	105(9)	102(23)	19(10)	94(7)
	2	164(16)	71(12)	160(16)	96(23)	98(11)
	3	227(13)	25(9)	74(11)	72(6)	9(10)
O(3)	1	136(16)	88(18)	125(48)	34(49)	90(11)
	2	150(17)	71(6)	141(46)	123(50)	101(6)
	3	257(13)	19(6)	74(6)	81(5)	11(6)
O(4)	1	144(15)	117(6)	84(10)	28(7)	97(6)
	2	213(15)	73(18)	157(20)	75(12)	94(20)
	3	245(14)	33(12)	68(20)	67(8)	8(10)
O(5)	1	137(17)	41(7)	85(7)	49(8)	30(10)
	2	208(17)	63(9)	50(6)	128(9)	59(10)
	3	331(17)	138(4)	41(5)	63(4)	88(4)
N(1)	1	132(20)	45(39)	135(37)	93(30)	73(44)
	2	150(18)	127(42)	144(41)	125(23)	155(37)
	3	182(15)	112(18)	114(19)	35(23)	106(22)
N(2)	1	154(18)	95(58)	124(49)	34(39)	95(28)
	2	165(19)	160(14)	56(50)	72(64)	110(12)
	3	236(17)	51(10)	52(10)	62(9)	21(10)
C(1)	1	133(24)	90(13)	8(20)	98(20)	68(13)
	2	181(17)	79(31)	98(20)	167(28)	98(29)
	3	210(18)	11(31)	88(13)	79(31)	23(16)
C(2)	1	98(24)	24(20)	73(29)	73(11)	8(29)
	2	136(22)	75(28)	162(28)	80(20)	97(29)
	3	186(16)	109(10)	85(19)	20(12)	90(11)
C(3)	1	139(21)	127(25)	100(10)	39(22)	113(20)
	2	172(20)	143(25)	78(10)	124(24)	138(16)
	3	263(21)	86(9)	16(9)	75(9)	58(9)
C(4)	1	142(19)	109(43)	87(28)	19(43)	90(40)
	2	161(17)	161(45)	87(76)	109(43)	152(76)
	3	174(20)	87(73)	4(61)	92(36)	62(76)



(*a*) (*b*)

Fig. 4. Comparison of the average bond lengths and angles of the chelate rings of bisglycino-copper(II) monohydrate (*a*) with those obtained for glycine (*b*) by Marsh (1958).

The average bond lengths and angles of the chelate are compared with those obtained by Marsh (1957) for α-glycine in Fig. 4. The standard deviations in the averages have not been estimated since the two chelate molecules do not represent independent determinations. It can be seen that the bond lengths and angles agree well except for those of the carboxyl group. In the chelate the C(2)–O(1) bond has lengthened and the C(2)–O(2) bond has shortened compared with the glycine values; the difference of 0·05 Å has probable significance. Differences of this magnitude are observed in amino acid salts (Hahn, 1957) and chelates (Barclay & Stephens, 1963), in carboxylic acids when the proton is attached to the carboxyl oxygen (Nardelli, Fava & Giraldi, 1962) and in peptide–metal complexes (Strandberg, Lindqvist & Rosenstein, 1961; Freeman *et al.*, 1964). Coordination to a metal presumably stabilizes the resonance form in which C(2)–O(1) is a single bond.

Environments of the copper ion and water molecule

Two glycine molecules are chelated to the copper(II) ion. The ligand atoms, O(1), O(3), N(1) and N(2) are not strictly co-planar, the deviations from their best least-squares plane (plane 3, Table 7) ranging from 0·019 Å to 0·037 Å. The copper(II) ion lies 0·05 Å out of this plane on the side of the water molecule (Cu–O(5) = 2·405 Å). A weak interaction (Cu–O(2^{viii}) = 2·74 Å) with an adjacent carboxyl oxygen completes the distorted octahedral coordination of the copper(II) ion.

The water molecule additionally makes two close approaches to carboxyl oxygen atoms at distances of 2·76 and 2·80 Å (Fig. 3). The angle $O(2^{ix})$–O(5)–$O(4^{v})$ is 116·7°, making the assignment of these contacts as hydrogen bonds reasonable. The distance of the water oxygen atom from the best plane fitted to itself and its three neighbours Cu, $O(2^{ix})$ and $O(4^{v})$ is 0·06 Å, and the sum of the three angles about it is 359·8°. Approximately trigonal water environments of this kind, whilst occurring in the minority of structures, are not uncommon (Clark, 1963).

Thermal analysis

The parameters μ_{ik} and θ_{ik} of the thermal vibration ellipsoids for all atoms (other than hydrogen) are shown in Table 8. These were calculated by the function and error program of Busing & Levy (1961, p. 140) and represent, respectively, the magnitudes of the principal axes of the vibration ellipsoids and their angles with respect to the cell axes. The angles between the same principal axes and the normal to the plane of the atoms O(1), O(3) and N(1) have also been calculated. Except for the atoms C(2), C(3) and N(1) the atoms of the chelate have their maximum amplitudes of vibration roughly normal to the coordination plane. The water molecule has the largest maximum vibration of any atom and this makes an angle of 8·6° with the normal to the plane of the atoms to which it is bonded ($O(4^{v})$, $O(2^{ix})$ and Cu).

The vibrations of the atoms N(1), O(1), C(1), C(2) and O(2) are smaller than those of the corresponding atoms of the other glycine molecule. The general trend of the magnitudes of the thermal vibrations is the same in both glycine molecules. In particular, the trigonal carbon atoms C(2) and C(4) have the smallest thermal parameters and the 'outer' oxygen atoms O(2) and O(4) the largest.

We cannot explain the behaviour of atoms C(2) and C(3). The atom C(2) has a relatively large vibrational component in the plane of the chelate in a direction approximately parallel to the vector between carboxyl oxygen atoms O(1) and O(2). A similar result was found for this atom in glycine by Marsh (1957) who thought it might be an artifact caused by the electron density associated with the C(2)–O(1) bond (*i.e.* C(1)–O(1) using Marsh's labels). Anomalous values of the thermal vibrations of trigonal carbon atoms in potassium bisbiureto cuprate(II) tetrahydrate have also been reported (Freeman *et al.*, 1961). Little physical significance, however, is accorded these phenomena, especially in view of the omission of hydrogen atoms from the present refinement.

Non-bonded contacts

The three shortest contacts between adjacent complexes are made by O(1) with the atoms $C(1^{vii})$, $N(1^{vii})$ and $C(2^{vii})$. These distances are included in Table 5. All other contacts exceed 3·2 Å.

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