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Crystal Structures of Copper(II) Complexes with ω-Amino Acids. I. Copper(II) Di-γ-aminobutyrate, $[Cu(NH_2CH_2CH_2CH_2COO)_2]_n$, and its Dihydrate, $[Cu(NH_2CH_2CH_2CH_2COO)_2.2H_2O]_n$

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The crystal structures of copper(II) di-γ-aminobutyrate (CuGABA-1) and its dihydrate (CuGABA-2) have been determined using three-dimensional X-ray data collected on Weissenberg photographs and estimated visually. Both crystals are monoclinic, space group $P2_1/c$ with two formula units in each unit cell. The cell dimensions are $a=7{\cdot}097$, $b=9{\cdot}693$, $c=7{\cdot}919$ Å, $\beta=107{\cdot}56°$ for CuGABA-1 and $a=5{\cdot}206$, $b=14{\cdot}406$, $c=8{\cdot}740$ Å, $\beta=109{\cdot}20°$ for CuGABA-2. The final R values are 0·078 and 0·077 for CuGABA-1 and CuGABA-2 respectively. The structure of CuGABA-1 consists of infinite one-dimensional chains, in which two γ-aminobutyrate ligands connect two copper(II) atoms by *trans* double-bridging, while that of CuGABA-2 consists of infinite two-dimensional networks. The water molecule in CuGABA-2 is not directly coordinated to the copper(II) atom, but links the neighbouring networks. The coordinations about the copper(II) atoms in the two crystals are quite similar, in spite of the difference in the modes of linkage of the ligands.

Introduction

There have been published a number of papers dealing with structures of metallic complexes with α-amino acids, and the five-membered chelate ring conformation with the central metallic atom has been established. Studies on the complex formation of ω-amino acids, $H_3N^+{-}[CH_2]_n{-}COO^-$, with metallic atoms are, on the other hand, very few and only two papers have been published dealing with the structures of copper(II) and nickel(II) complexes with β-alanine, in both of which six-membered chelate rings with the central metallic atom are reported (Tomita, 1961; Jose & Pant, 1965). Nakahara, Hidaka & Tsuchida (1956) prepared copper(II) di-γ-aminobutyrate dihydrate and copper(II) di-δ-aminovalerate, and suggested that in these complexes each ω-amino acidate ligand coordinates to one and the same copper(II) atom to form a seven- or eight-membered chelate ring. Bryan, Poljak & Tomita (1961) pointed out a possibility of forming a one-dimensional chain for the latter complex, in which two copper(II) atoms are double-bridged by two δ-aminovalerate ligands.

Saito and his coworkers (Kinoshita, Matsubara & Saito, 1959*a*, 1959*b* and Kinoshita, Matsubara, Higuchi & Saito, 1959) made an extensive study on the structures of copper(I) complexes with aliphatic dinitriles, $NC{-}[CH_2]_n{-}CN$ ($n=2,3,4$), and reported that nitrile groups at the two ends of the molecules are coordinated to two different copper(I) atoms with their nitrogen atoms forming one-, two- and three-dimensional polymeric complexes. ω-Amino acids have an analogy with aliphatic dinitriles in the sense that the distance between the two functional groups at the two ends increases with increasing number of methylenic groups. We may expect one-, two- or three-dimensional polymeric structures as possible structures for copper(II) complexes with ω-amino acids.

It is the purpose of the present study to look into the structural question mentioned above including the influence of the number of methylenic groups on the complex formation. Here we present the results of analyses of the crystal structures of copper(II) di-γ-aminobutyrate and its dihydrate.

Experimental

Preparation

A large excess of γ-aminobutyric acid was added to an aqueous solution of copper(II) chloride. Two types of crystals, one blue in colour and prismatic and the other violet and needle-shaped, were obtained at room temperature by diluting the solution with a mixture of methanol and ethanol; the total yield of the former crystal was much lower than that of the latter. These two crystals are designated hereafter in this paper as CuGABA-1 and CuGABA-2 respectively. Analyses of these crystals are as follows.

CuGABA-1	Anal. found C, 35·68; H, 6·35; N, 10·56%
Calcd. for	$Cu[NH_2(CH_2)_3COO]_2$ C, 35·70; H, 6·02; N, 10·48%
CuGABA-2	Anal. found C, 31·59; H, 6·84; N, 9·45%
Calcd. for	$Cu[NH_2(CH_2)_3COO]_2.2H_2O$ C, 31·54; H, 6·49; N, 9·27%

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CuGABA-2 is identified from its composition as the complex prepared by Nakahara *et al.* (1956) from copper(II) perchlorate and γ-aminobutyric acid, while CuGABA-1 is a new compound hitherto unreported. Applying the method of Nakahara *et* al. (1956), it was also possible to obtain along with CuGABA-2 a small quantity of CuGABA-1.

Crystal data

Both crystals were found to be monoclinic, space group $P2_1/c$, from oscillation and Weissenberg photographs taken with Cu $K\alpha$ radiation. The unit-cell dimensions of these crystals were determined from zero-layer Weissenberg photographs about [100] and [010], calibrated with superposed Si lines [a(Si) = 5·43075 Å at 25 °C; λ(Cu $K\alpha_1$) = 1·54051 and λ(Cu $K\alpha_2$) = 1·54433 Å]. The results are summarized in Table 1.

Table 1. *Crystal data*

CuGABA-1	CuGABA-2
$Cu(NH_2(CH_2)_3COO)_2$	$Cu(NH_2(CH_2)_3COO)_2\cdot 2H_2O$
F.W. 267·8	F.W. 303·8
Monoclinic; $P2_1/c$	Monoclinic; $P2_1/c$
a = 7·097 (2) Å	a = 5·206 (1) Å
b = 9·693 (1)	b = 14·406 (3)
c = 7·919 (4)	c = 8·740 (2)
β = 107·56 (4)°	β = 109·20 (2)°
V = 519·4 (3) Å^3	V = 619·10 (9) Å^3
D_m = 1·70 g.cm^{-3}	D_m = 1·63 g.cm^{-3}
D_x = 1·712	D_x = 1·630
Z = 2	Z = 2
μ(Cu $K\alpha$) = 30·5 cm^{-1}	μ(Cu $K\alpha$) = 28·7 cm^{-1}

The observed densities of the two crystals, as determined by the flotation method at room temperature, correspond to two formula units per unit cell. Accordingly the copper(II) atoms of the two crystals occupy the centres of symmetry.

Intensity data

Intensity data for CuGABA-1 were collected with Cu $K\alpha$ radiation on multiple-film equi-inclination, integrating Weissenberg photographs from layers 0–6 about [100] and layers 0–4 about [101], using the same prismatic crystal with dimensions of about 0·3 × 0·3 × 0·2 mm. Intensity data for CuGABA-2 were collected in the same way: layers 0–4 about [100], using a needle-shaped crystal of about 0·1 × 0·1 mm cross section, and layers 0–6 about [101], using a crystal cut to about 0·1 × 0·3 mm cross section. The intensities were estimated visually by comparison with a calibrated film strip, and were corrected for Lorentz and polarization factors, and for spot size. The two series of relative intensities were then cross-correlated and placed on an absolute scale using Wilson's (1942) method. The total number of independent reflexions was 1018 for CuGABA-1 and 1275 for CuGABA-2, of which non-zero reflexions numbered 864 and 886 respectively.

Structure determination

CuGABA-1

The positions of the copper(II) and the other non-hydrogen atoms were readily located from a three-dimensional sharpened Patterson function. The initial structure factor calculation for this approximate structure had an R value, $R=\sum|\Delta|/\sum|kF_o|$ $(\Delta=|kF_o|-|F_c|)$, of 0·19. The structure was refined by the least-squares method using a block-diagonal approximation for the normal equations. Four cycles of the refinement with isotropic thermal parameters and a further four cycles with anisotropic thermal parameters lowered R to 0·088. The quantity minimized was $\sum w\Delta^2$, with $w=1$ for all the observed reflexions. Reflexions too weak to be observed were rejected from the structural refinement. An attempt was then made to find the hydrogen atoms. The locations of all the eight hydrogen atoms were easily identified on a difference map computed at this stage.

In order to refine the coordinates of the hydrogen atoms, a new weighting function, $w=\exp(-P)$: $P=ax^2+by^2+cxy+dx+ey+f$, $(x=|kF_o|,\ y=\sin\theta/\lambda)$, was applied. The coefficients in the equation for P were evaluated by least-squares calculations from an analysis so that a two-dimensional distribution of

Table 2. *Final positional and thermal parameters of* CuGABA-1

Standard deviations as obtained from the block-diagonal matrix are indicated in parentheses. The form of the isotropic temperature factor is $\exp(-B\sin^2\theta/\lambda^2)$ and that of the anisotropic temperature factor is $\exp(-h^2\beta_{11}-k^2\beta_{22}-l^2\beta_{33}-hk\beta_{12}-kl\beta_{23}-lh\beta_{31})$.

	x	y	z	B
Cu	0·0000 (0)	0·0000 (0)	0·0000 (0)	
O(1)	0·2727 (5)	0·0227 (3)	0·1614 (5)	
O(2)	0·1672 (5)	0·2377 (4)	0·1244 (5)	
C(1)	0·2966 (6)	0·1506 (4)	0·1956 (6)	
C(2)	0·4849 (6)	0·1961 (5)	0·3336 (6)	
C(3)	0·6613 (6)	0·1001 (5)	0·3608 (6)	
C(4)	0·7361 (7)	0·0986 (5)	0·2007 (6)	
N	0·9039 (7)	0·0053 (4)	0·2154 (6)	
H(1)	0·455 (17)	0·208 (11)	0·440 (14)	2·8 (11)
H(2)	0·519 (13)	0·290 (9)	0·286 (11)	1·5 (9)
H(3)	0·623 (15)	0·004 (8)	0·386 (12)	1·7 (10)
H(4)	0·765 (12)	0·130 (9)	0·442 (11)	1·4 (9)
H(5)	0·628 (10)	0·072 (7)	0·099 (9)	0·6 (8)
H(6)	0·772 (14)	0·183 (9)	0·169 (12)	1·8 (9)
H(7)	0·860 (13)	−0·084 (8)	0·230 (11)	1·5 (9)
H(8)	1·013 (12)	0·035 (8)	0·322 (10)	1·0 (8)

β_{ij}s have been multiplied by 10^4.

	β_{11}	β_{22}	β_{33}	β_{12}	β_{23}	β_{31}
Cu	70 (2)	65 (1)	124 (2)	−4 (2)	−9 (2)	47 (3)
O(1)	59 (7)	52 (3)	147 (7)	−19 (7)	−57 (7)	53 (10)
O(2)	76 (7)	64 (3)	165 (7)	19 (8)	−8 (8)	−13 (10)
C(1)	56 (9)	64 (4)	102 (7)	−39 (9)	−46 (9)	75 (12)
C(2)	68 (9)	74 (4)	99 (7)	−17 (10)	−57 (10)	61 (12)
C(3)	61 (9)	72 (5)	89 (7)	−10 (10)	−12 (9)	30 (12)
C(4)	74 (9)	74 (5)	84 (7)	27 (10)	26 (9)	59 (12)
N	90 (10)	66 (4)	101 (7)	17 (8)	28 (8)	50 (13)

Table 3. *Observed and calculated structure factors for* CuGABA-1

H	K	L	FO	FC
0	2	0	59.64	64.81
0	4	0	59.04	66.63
0	6	0	35.16	35.30
0	8	0	34.95	37.31
0	10	0	22.10	21.70
0	12	0	14.45	14.16
0	1	1	61.71	68.63
0	2	1	66.25	-66.77
0	3	1	53.57	59.95
0	5	1	42.58	47.32
0	6	1	10.03	-8.29
0	7	1	28.25	30.40
0	9	1	19.27	19.61
0	10	1	6.58	-7.13
0	11	1	7.26	7.45
0	12	1	4.91	-5.01
0	0	2	33.10	-23.19
0	1	2	19.90	-18.71
0	2	2	19.97	22.65
0	3	2	9.48	8.44
0	4	2	29.05	32.13
0	5	2	19.54	-17.64
0	6	2	15.76	16.51
0	8	2	13.01	12.44
0	9	2	15.18	-15.67
0	10	2	6.97	6.97
0	11	2	2.52	2.93
0	12	2	5.43	5.79
0	3	3	17.69	15.69
0	5	3	16.09	17.84
0	6	3	5.80	-4.79
0	7	3	8.84	8.88
0	8	3	5.31	4.58
0	9	3	11.32	11.31
0	11	3	10.32	12.31
0	0	4	8.87	10.86
0	1	4	7.17	4.47
0	2	4	43.90	46.25
0	3	4	15.38	15.32
0	4	4	18.92	20.32
0	6	4	29.50	31.46
0	7	4	1.56	2.06
0	8	4	15.36	15.63
0	9	4	7.39	8.22
0	10	4	10.10	11.78
0	1	5	48.34	49.09
0	2	5	16.66	14.21
0	3	5	30.28	30.21
0	5	5	17.07	16.55
0	6	5	7.62	7.31
0	7	5	25.82	26.27
0	9	5	12.06	13.83
0	0	6	39.92	39.42
0	2	6	23.12	21.71
0	3	6	16.64	-15.91
0	4	6	18.80	17.63
0	5	6	10.31	9.95
0	6	6	18.43	17.13
0	8	6	7.56	8.99
0	9	6	2.62	3.82
0	1	7	5.58	5.35
0	2	7	7.30	-6.54
0	3	7	19.80	19.18
0	5	7	16.24	16.70
0	7	7	5.56	6.36
0	0	8	8.22	8.84
0	1	8	6.67	6.59
0	2	8	9.91	9.60
0	4	8	11.77	12.68
0	6	8	2.84	2.72
0	1	9	8.60	11.13
0	3	9	5.08	5.19
1	0	0	34.81	43.43
1	1	0	22.19	-24.73
1	2	0	65.49	62.96
1	3	0	36.93	33.62
1	4	0	60.47	66.16
1	5	0	14.85	-12.22
1	6	0	29.34	30.32
1	7	0	14.35	-12.99
1	8	0	32.15	31.59
1	9	0	17.55	-17.19
1	10	0	10.28	11.16
1	11	0	3.42	3.44
1	12	0	12.16	12.57
1	1	1	63.48	61.51
1	2	1	5.87	4.37
1	3	1	35.20	39.75
1	5	1	15.83	17.43
1	6	1	5.65	-5.62
1	7	1	15.74	15.61
1	9	1	25.46	26.02
1	10	1	6.60	-6.81
1	11	1	19.06	20.61
1	12	1	2.12	2.50
1	1	2	26.07	-22.56
1	2	2	24.14	30.47
1	3	2	12.37	4.34
1	4	2	8.05	11.10
1	5	2	9.05	-8.65
1	6	2	23.03	25.19
1	7	2	12.58	12.35
1	8	2	5.65	6.79
1	9	2	9.65	9.50
1	10	2	14.16	16.27
1	11	2	3.08	-2.95
1	1	3	20.60	20.51
1	2	3	15.48	13.59
1	3	3	27.83	30.35
1	4	3	5.70	5.15
1	5	3	14.01	13.65
1	6	3	6.46	5.67
1	7	3	26.93	26.86
1	8	3	8.82	9.12
1	9	3	4.40	4.76
1	10	3	5.55	5.73
1	11	3	5.19	6.24
1	0	4	44.29	47.39
1	1	4	16.61	14.73
1	2	4	40.49	42.44
1	3	4	15.72	-12.50
1	4	4	26.11	25.70
1	5	4	13.35	13.52
1	6	4	33.14	34.41
1	7	4	3.87	4.32
1	8	4	14.54	14.74
1	10	4	10.07	10.39
1	1	5	33.75	35.89
1	3	5	33.40	35.09
1	4	5	6.85	6.91
1	5	5	36.31	37.28
1	7	5	18.32	18.63
1	8	5	7.40	-8.58
1	9	5	11.37	12.41
1	0	6	26.03	25.03
1	1	6	4.30	4.73
1	2	6	23.88	23.30
1	4	6	20.73	19.52
1	6	6	2.52	1.84
1	7	6	7.68	-9.65
1	8	6	12.20	14.74
1	9	6	3.54	-4.11
1	1	7	10.49	10.64
1	2	7	1.87	-2.52
1	3	7	4.09	5.00
1	4	7	9.74	-9.65
1	5	7	4.02	4.37
1	6	7	2.08	2.25
1	0	8	5.15	5.74
1	1	8	3.21	-3.81
1	3	8	2.10	3.08
1	4	8	8.22	9.54
1	5	8	1.63	-2.22
1	1	9	8.35	9.32
-1	1	1	44.44	57.81
-1	2	1	38.24	35.75
-1	3	1	32.12	35.34
-1	4	1	2.72	-4.56
-1	5	1	62.34	62.65
-1	6	1	3.19	5.55
-1	7	1	28.29	27.11
-1	9	1	15.79	14.87
-1	10	1	2.32	2.50
-1	11	1	5.02	3.73
-1	12	1	1.83	2.45
-1	0	2	83.65	102.81
-1	1	2	34.68	-30.23
-1	2	2	15.78	20.57
-1	3	2	19.74	17.65
-1	4	2	39.04	42.07
-1	6	2	23.38	21.74
-1	7	2	5.06	-4.96
-1	8	2	23.53	22.47
-1	9	2	3.50	-2.79
-1	10	2	7.96	7.18
-1	12	2	7.37	7.22
-1	1	3	32.54	37.71
-1	2	3	12.35	-13.80
-1	3	3	47.25	51.14
-1	4	3	3.37	4.66
-1	5	3	26.76	26.37
-1	6	3	4.64	-6.38
-1	7	3	22.30	23.23
-1	8	3	6.14	-5.77
-1	9	3	15.45	14.38
-1	10	3	6.33	-6.37
-1	11	3	7.22	7.16
-1	0	4	38.26	37.78
-1	1	4	2.92	-3.89
-1	2	4	45.85	44.70
-1	3	4	8.57	7.05
-1	4	4	25.59	25.79
-1	5	4	13.18	-11.74
-1	6	4	20.32	19.47
-1	8	4	13.87	13.46
-1	9	4	10.24	-9.89
-1	10	4	21.43	21.44
-1	11	4	1.71	2.42
-1	1	5	23.60	23.83
-1	2	5	10.72	-10.80
-1	3	5	23.03	22.61
-1	4	5	4.16	-4.33
-1	5	5	15.93	14.98
-1	6	5	2.00	-2.53
-1	7	5	14.84	13.87
-1	8	5	10.70	10.68
-1	9	5	12.64	12.68
-1	0	6	6.54	-5.56
-1	1	6	7.31	7.57
-1	2	6	20.16	19.45
-1	4	6	12.74	12.09
-1	6	6	22.17	21.50
-1	7	6	4.00	4.56
-1	8	6	8.44	8.46
-1	9	6	5.20	5.55
-1	1	7	22.05	20.24
-1	2	7	10.35	10.31
-1	3	7	8.63	8.58
-1	5	7	14.72	13.44
-1	6	7	5.91	5.79
-1	7	7	12.39	11.38
-1	0	8	23.14	24.58
-1	1	8	2.58	-3.71
-1	2	8	15.57	15.57
-1	4	8	14.74	15.11
-1	5	8	7.10	6.99
-1	6	8	8.63	9.07
-1	3	9	13.61	16.88
-1	5	9	7.70	8.42
2	1	0	21.75	-19.45
2	2	0	24.01	28.30
2	3	0	4.55	8.51
2	4	0	44.25	40.98
2	5	0	16.61	-11.80
2	6	0	35.03	36.09
2	7	0	18.83	17.57
2	8	0	9.20	9.29
2	9	0	3.50	2.95
2	10	0	16.10	17.21
2	11	0	2.04	1.93
2	12	0	5.15	5.71
2	1	1	39.96	45.51
2	2	1	33.04	30.87
2	3	1	51.97	56.95
2	4	1	22.95	22.70
2	5	1	37.03	38.23
2	6	1	10.94	-9.39
2	7	1	36.20	36.85
2	8	1	6.30	5.74
2	9	1	14.74	13.90
2	10	1	3.80	3.79
2	11	1	14.09	13.24
2	0	2	59.03	61.43
2	1	2	29.65	22.80
2	2	2	73.17	72.84
2	4	2	11.54	10.79
2	5	2	12.47	8.91
2	6	2	33.57	32.48
2	7	2	3.47	2.59
2	8	2	21.70	21.94
2	10	2	15.15	15.58
2	11	2	2.35	2.31
2	1	3	44.72	42.17
2	2	3	15.88	-12.53
2	3	3	23.76	23.78
2	4	3	3.83	-4.54
2	5	3	31.17	29.69
2	6	3	12.31	11.24
2	7	3	17.53	16.51
2	8	3	3.56	-3.50
2	9	3	8.96	8.56
2	10	3	2.25	2.29
2	11	3	7.73	7.98
2	0	4	25.41	26.35
2	2	4	25.38	24.69
2	3	4	9.85	-7.71
2	4	4	31.84	30.34
2	5	4	7.62	6.89
2	6	4	10.78	10.09
2	7	4	11.32	-10.79
2	8	4	12.81	12.67
2	9	4	1.85	1.90
2	10	4	8.18	7.17
2	1	5	20.66	18.11
2	3	5	27.26	26.59
2	4	5	11.43	-10.55
2	5	5	17.61	17.40
2	7	5	11.63	11.21
2	9	5	11.47	12.80
2	0	6	19.80	18.63
2	1	6	4.58	3.88
2	2	6	6.36	5.13
2	3	6	5.98	5.75
2	4	6	22.28	22.68
2	5	6	5.02	-5.39
2	6	6	14.63	15.05
2	8	6	9.17	9.44
2	1	7	15.39	15.41
2	2	7	5.44	4.48
2	3	7	5.58	5.29
2	4	7	7.40	8.11
2	5	7	3.35	3.30
2	6	7	3.60	-4.07
2	7	7	6.33	7.77
2	0	8	9.74	10.01
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-6	6	6	8.46	9.24
-6	7	6	5.58	-6.57
-6	2	7	2.34	2.81
-6	3	7	16.07	17.91
-6	5	7	6.78	8.07
-6	6	7	3.24	-3.48
-7	3	3	20.76	21.54
-7	5	3	14.20	14.47
-7	6	3	2.81	2.40
-7	7	3	10.04	8.64
-7	0	4	13.33	12.48
-7	1	4	7.77	7.34
-7	2	4	11.70	11.00
-7	3	4	14.06	14.33
-7	4	4	12.24	11.78
-7	5	4	3.45	-3.71
-7	6	4	10.64	11.81
-7	7	4	2.12	2.61
-7	1	5	26.75	26.27
-7	3	5	15.45	15.13
-7	4	5	4.77	4.68
-7	5	5	10.12	9.61
-7	7	5	11.58	14.53
-7	0	6	22.28	20.51
-7	2	6	26.15	25.44
-7	3	6	3.57	-4.09
-7	4	6	12.68	12.68
-7	5	6	1.65	2.09
-7	6	6	17.16	18.04
-7	1	7	14.56	16.60
-7	2	7	5.04	-5.43
-7	3	7	9.20	10.06
-7	5	7	8.81	11.30
-8	0	4	6.19	6.92
-8	1	4	6.06	-7.08
-8	2	4	10.64	11.58
-8	4	4	5.39	6.41
-8	1	5	9.95	9.95
-8	2	5	2.46	3.50
-8	3	5	13.66	14.82
-8	4	5	2.69	-3.76
-8	5	5	10.39	11.53
-8	0	6	17.05	19.04
-8	2	6	16.47	15.35
-8	3	6	3.78	4.28
-8	1	7	9.27	11.19

Table 4. *Bond distances and angles for* CuGABA-1

Standard deviations are added in parentheses. The superscript A refers to the equivalent position ($\bar{x}+1,\bar{y},\bar{z}$).

Distances (Å)	
Cu—O(1)	1·982 (3)
Cu···O(2)	2·641 (4)
Cu^A—N	2·015 (5)
C(1)—O(1)	1·269 (5)
C(1)—O(2)	1·249 (5)
C(1)—C(2)	1·513 (6)
C(2)—C(3)	1·522 (6)
C(3)—C(4)	1·511 (7)
C(4)—N	1·471 (7)
C(2)—H(1)	0·93 (13)
C(2)—H(2)	1·04 (9)
C(3)—H(3)	1·01 (9)
C(3)—H(4)	0·87 (7)
C(4)—H(5)	0·96 (6)
C(4)—H(6)	0·91 (9)
N—H(7)	0·94 (9)
N—H(8)	1·00 (7)

Angles (°)	
O(1)—Cu—N^A	88·2 (2)
O(1)—Cu····O(2)	54·7 (1)
O(2)···Cu—N^A	83·8 (2)
C(1)—O(1)—Cu	106·7 (3)
C(1)—O(2)···Cu	76·4 (3)
O(1)—C(1)—C(2)	118·0 (4)
O(2)—C(1)—C(2)	119·8 (4)

Angles (°)	
O(1)–C(1)–O(2)	122·1 (4)
C(1)–C(2)–C(3)	115·7 (4)
C(2)–C(3)–C(4)	111·7 (4)
C(3)–C(4)–N	114·9 (4)
C(4)–N—Cu^A	114·9 (3)
C(1)–C(2)–H(1)	107 (8)
C(3)–C(2)–H(1)	111 (8)
C(1)–C(2)–H(2)	104 (5)
C(3)–C(2)–H(2)	108 (5)
H(1)–C(2)–H(2)	111 (9)
C(2)–C(3)–H(3)	110 (5)
C(4)–C(3)–H(3)	110 (5)
C(2)–C(3)–H(4)	112 (5)
C(4)–C(3)–H(4)	101 (5)
H(3)–C(3)–H(4)	113 (7)
C(3)–C(4)–H(5)	108 (4)
N—C(4)–H(5)	109 (4)
C(3)–C(4)–H(6)	115 (6)
N—C(4)–H(6)	107 (6)
H(5)–C(4)–H(6)	103 (7)
C(4)–N—H(7)	107 (5)
Cu^A–N—H(7)	107 (5)
C(4)–N—H(8)	107 (4)
Cu^A–N—H(8)	110 (4)
H(7)–N—H(8)	111 (6)

$\langle w\Delta^2\rangle$ was made as uniform as possible.* Further refinement of the structure was continued using the new weighting scheme with the inclusion of the hydrogen atoms, to which isotropic thermal motions were assigned. The least-squares procedure was interrupted when all parameter shifts were less than one third of the calculated standard deviations. In each least-squares cycle the weighting scheme was checked and the coefficients in the equation for P were analysed for subsequent cycles; the final values of these coefficients were 0·000799, 55·9, 0·349, −0·115, −58·2 and 13·2 respectively. The final R and R_w values, $R_w=\{\sum w\Delta^2/\sum w|kF_o|^2\}^{1/2}$ (Hamilton, 1965), for all the data are 0·078 and 0·089. The positional and thermal parameters are given in Table 2, and the observed and calculated structure factors in Table 3. Table 4 gives the bond distances and angles.

CuGABA-2

The structure determination of CuGABA-2 was carried out by the same procedure as that used for CuGABA−1. Nine of the ten hydrogen atoms were easily located on an electron-density difference map, and a weak broad peak was found, which could be taken as the remaining hydrogen atom. The weighting scheme described above was applied in the final refinement with the inclusion of the ten hydrogen atoms. The coefficients in the equation for P were −0·00139, 6·89, −0·218, 0·194, −3·96 and −0·249 respectively. The final R and R_w values for all the observed reflexions are 0·077 and 0·091.

The positional and thermal parameters are listed in Table 5 together with their standard deviations. The standard deviations of CuGABA-2 are larger than those of CuGABA-2, because of the higher temperature factors for CuGABA-2. The observed and calculated structure factors are given in Table 6, and the bond distances and angles, together with their standard deviations, in Table 7.

Table 5. *Final positional and thermal parameters of* CuGABA-2

Standard deviations as obtained from the block-diagonal matrix are indicated in parentheses. The form of the isotropic temperature factor is $\exp(-B\sin^2\theta/\lambda^2)$ and that of the anisotropic temperature factor is $\exp(-h^2\beta_{11}-k^2\beta_{22}-l^2\beta_{33}-hk\beta_{12}-kl\beta_{23}-lh\beta_{31})$.

	x	y	z	B
Cu	0·0000 (0)	0·0000 (0)	0·0000 (0)	
O(1)	0·3015 (10)	0·0831 (4)	0·1113 (6)	
O(2)	0·1797 (13)	0·0533 (6)	0·3257 (7)	
C(1)	0·3311 (15)	0·0905 (7)	0·2632 (11)	
C(2)	0·5814 (20)	0·1413 (9)	0·3665 (14)	
C(3)	0·5262 (20)	0·2438 (8)	0·3655 (16)	
C(4)	0·7885 (19)	0·3007 (6)	0·4427 (15)	
N	0·7203 (12)	0·3978 (4)	0·4463 (7)	
O(W)	0·2225 (14)	0·4225 (6)	0·1553 (8)	
H(1)	0·637 (27)	0·119 (10)	0·499 (16)	3·3 (25)
H(2)	0·742 (15)	0·134 (6)	0·302 (9)	5·8 (31)
H(3)	0·428 (13)	0·259 (5)	0·229 (8)	6·1 (31)
H(4)	0·364 (21)	0·252 (8)	0·443 (13)	8·8 (38)
H(5)	0·862 (28)	0·280 (10)	0·543 (16)	0·0 (12)
H(6)	0·944 (38)	0·294 (13)	0·387 (20)	2·7 (22)
H(7)	0·542 (22)	0·412 (9)	0·340 (14)	4·3 (31)
H(9)	0·623 (24)	0·409 (11)	0·526 (15)	0·0 (14)
H(8)	0·209 (23)	0·431 (9)	0·044 (14)	5·0 (28)
H(10)	0·127 (16)	0·464 (6)	0·174 (9)	0·0 (14)

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* An application of this weighting scheme is described elsewhere (Furusaki & Watanabé, 1972).

Table 5 (*cont.*)

β_{ij}s have been multiplied by 10^4

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{23}	β_{31}
Cu	252 (4)	31 (1)	96 (2)	6 (3)	11 (2)	70 (4)
O(1)	268 (19)	39 (3)	95 (7)	−34 (12)	−1 (7)	84 (19)
O(2)	368 (25)	102 (6)	119 (9)	−104 (20)	−25 (12)	158 (25)
C(1)	252 (28)	55 (5)	165 (14)	−61 (19)	−60 (13)	108 (32)
C(2)	415 (41)	72 (7)	233 (20)	−124 (26)	−64 (19)	19 (47)
C(3)	393 (42)	60 (6)	297 (24)	−70 (24)	−98 (19)	155 (51)
C(4)	461 (41)	31 (4)	310 (24)	−50 (21)	−77 (15)	290 (53)
N	272 (22)	35 (3)	100 (9)	−28 (14)	8 (8)	24 (22)
O(*W*)	483 (31)	82 (5)	123 (10)	56 (20)	45 (11)	−12 (27)

Description and discussion of the structure

The crystal structure of CuGABA-1

As shown in Fig. 1, no isolated chelate complexes are found in the crystal. The γ-aminobutyrate ligands are coordinated to two copper(II) atoms by means of the *trans* double-bridging, and this linkage is repeated with successive copper(II) atoms to form an infinite one-dimensional chain parallel to [100].

The atoms N, O(1), O(2) and those related by the centre of symmetry form a distorted octahedron about the copper(II) atom (see Fig. 2). The nearest neighbours of the copper(II) atom are two N and two O(1) atoms, which form an almost square coordination. The coordination plane is expressed by equation $-0{\cdot}0734X+0{\cdot}9936Y-0{\cdot}0859Z=0$, where X, Y and Z are in Å and are referred to the orthogonal axes a, b and c^* (this reference is also used for the planes described below). The Cu–N and Cu–O(1) distances and the N–Cu–O(1) angle are comparable with the corresponding average values $2{\cdot}00_8$, $1{\cdot}96_6$ Å and 84° obtained from a large number of reported copper(II) complexes with amino acids and peptides (Freeman, 1966). The remaining two O(2) atoms occupy the two apical positions located at a distance 2·641 Å from the copper(II) atom above and below the coordination plane. The O(1)–Cu···O(2) and N–Cu···O(2) angles are 54·7 and 83·8°, and the line passing through the O(2) and Cu atoms makes an angle 33·3° with the normal to the coordination plane. This double carboxylate–copper(II) interaction (Freeman, 1966) is analogous to those found in copper(II) glutamate dihydrate (Gramaccioli & Marsh, 1966), glycylglycylglycinatocopper(II) chloride sesquihydrate (Freeman, Robinson & Schoone, 1964) and diammine-(*o*-phthalato)copper(II) (Cingi, Guastini, Musatti & Nardelli, 1970), where the values corresponding to the Cu···O(2) distance are 2·59, 2·82 and 2·80 Å respectively.

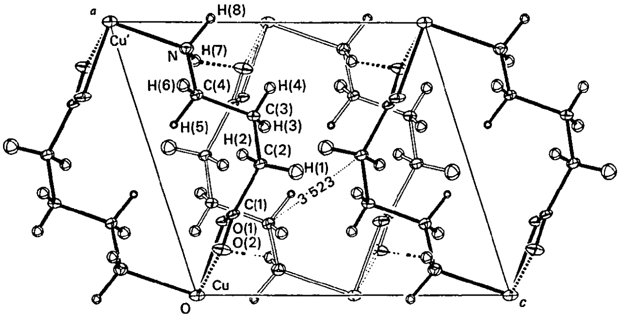
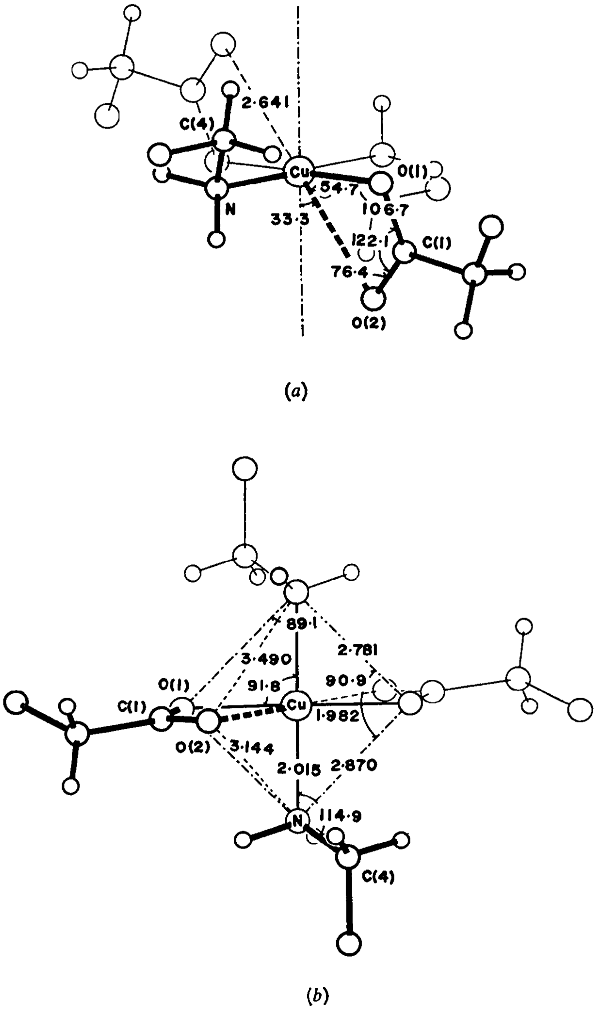


Fig. 1. The crystal structure of CuGABA-1 viewed along the *b* axis.



(*a*)

(*b*)

Fig. 2. The coordination about the copper(II) atom in CuGABA-1. Projections are (*a*) along the direction making an inclination angle of 10° from the coordination plane and (*b*) normal to the coordination plane.

Table 6. *Observed and calculated structure factors for* CuGABA-2

H	K	L	FO	FC
0	2	0	68.59	75.00
0	4	0	11.79	10.77
0	6	0	37.95	-31.21
0	8	0	19.47	19.87
0	10	0	45.04	38.11
0	12	0	29.36	30.00
0	14	0	17.88	16.27
0	16	0	9.75	8.38
0	1	1	53.46	56.98
0	2	1	34.11	-31.32
0	3	1	32.22	29.41
0	4	1	17.29	-20.67
0	5	1	32.06	38.58
0	7	1	21.15	20.39
0	9	1	46.00	43.71
0	11	1	37.48	37.39
0	13	1	12.74	13.32
0	15	1	13.55	12.24
0	17	1	5.81	5.40
0	0	2	37.27	42.13
0	1	2	34.82	34.90
0	2	2	23.12	23.15
0	3	2	27.05	-28.88
0	4	2	39.05	39.81
0	5	2	9.74	-9.24
0	6	2	42.57	43.35
0	8	2	35.85	36.08
0	9	2	19.09	17.80
0	10	2	35.19	36.30
0	11	2	6.09	5.76
0	12	2	15.02	14.43
0	14	2	14.23	13.28
0	15	2	7.49	-6.82
0	16	2	9.83	9.83
0	18	2	6.54	7.96
0	1	3	82.76	90.15
0	2	3	17.55	13.51
0	3	3	38.27	39.22
0	5	3	29.21	28.07
0	7	3	19.11	19.42
0	8	3	9.79	-8.15
0	9	3	24.61	22.74
0	11	3	17.92	16.57
0	12	3	9.31	9.48
0	13	3	15.09	15.24
0	14	3	3.49	-3.58
0	15	3	11.16	11.81
0	17	3	8.32	7.23
0	0	4	4.80	5.02
0	1	4	5.20	5.48
0	2	4	25.46	30.22
0	4	4	35.58	36.15
0	5	4	9.10	-8.83
0	6	4	40.95	40.33
0	7	4	7.00	-6.92
0	8	4	29.12	28.41
0	9	4	6.80	-7.52
0	10	4	25.93	26.29
0	11	4	8.03	7.96
0	12	4	9.95	10.26
0	14	4	10.80	9.60
0	16	4	9.74	6.82
0	1	5	6.47	8.29
0	2	5	8.16	8.59
0	3	5	26.36	23.73
0	4	5	4.58	-4.27
0	5	5	37.20	37.27
0	7	5	35.06	35.56
0	9	5	23.10	24.45
0	11	5	9.78	8.99
0	13	5	10.77	10.22
0	15	5	10.62	7.95
0	0	6	33.01	31.13
0	1	6	9.09	7.29
0	2	6	29.48	31.29
0	3	6	18.86	19.88
0	4	6	28.35	28.85
0	5	6	5.89	5.97
0	6	6	19.79	20.62
0	7	6	6.57	-6.47
0	8	6	15.87	13.89
0	9	6	16.02	-14.61
0	10	6	4.77	5.27
0	12	6	8.65	9.99
0	14	6	11.65	10.48
0	1	7	12.71	12.20
0	2	7	11.43	11.91
0	3	7	23.48	22.30
0	5	7	19.98	20.23
0	6	7	5.76	-5.20
0	7	7	15.12	15.58
0	8	7	6.80	-11.49
0	9	7	12.00	11.21
0	11	7	10.34	10.03
0	13	7	10.47	9.92
0	0	8	17.15	15.23
0	2	8	14.55	13.26
0	4	8	12.52	11.99
0	6	8	10.67	10.74
0	8	8	12.89	12.53
0	10	8	8.07	9.25
0	1	9	14.36	15.98
0	3	9	15.59	15.13
0	5	9	9.21	9.33
0	7	9	6.33	5.86
0	9	9	5.20	3.29
0	2	10	8.38	8.21
0	4	10	9.83	10.06
1	1	0	5.62	2.74
1	2	0	70.96	67.70
1	3	0	73.88	-64.00
1	4	0	53.35	56.85
1	5	0	45.09	-48.21
1	6	0	52.28	54.06
1	8	0	18.22	17.11
1	9	0	11.36	12.26
1	10	0	26.68	28.08
1	11	0	17.17	17.43
1	12	0	11.43	11.65
1	14	0	14.45	15.70
1	16	0	9.32	11.25
1	18	0	8.67	9.59
1	1	1	10.72	12.63
1	2	1	53.45	43.39
1	3	1	26.28	25.84
1	5	1	65.94	63.73
1	6	1	6.66	5.72
1	7	1	46.38	54.33
1	8	1	14.35	-12.98
1	9	1	27.94	28.82
1	11	1	9.16	8.80
1	12	1	6.33	5.25
1	13	1	17.78	17.78
1	15	1	13.74	14.86
1	17	1	10.94	11.36
1	0	2	13.93	-8.05
1	1	2	15.46	11.54
1	2	2	27.60	29.47
1	3	2	41.48	36.83
1	4	2	65.91	68.04
1	5	2	14.77	15.26
1	6	2	51.70	56.32
1	8	2	30.59	33.58
1	9	2	20.34	-21.52
1	10	2	11.06	11.76
1	11	2	8.20	7.57
1	12	2	11.69	12.18
1	14	2	11.12	12.43
1	16	2	10.23	10.66
1	17	2	3.76	-2.27
1	1	3	36.61	35.83
1	3	3	49.72	51.55
1	4	3	26.68	-23.26
1	5	3	33.66	34.12
1	6	3	11.57	-10.89
1	7	3	26.86	27.13
1	9	3	18.02	17.82
1	10	3	6.38	4.74
1	11	3	11.54	13.09
1	13	3	15.68	15.51
1	15	3	9.44	10.58
1	17	3	6.76	9.39
1	0	4	16.43	19.00
1	1	4	12.81	8.89
1	2	4	23.42	18.48
1	3	4	14.28	15.56
1	4	4	36.00	35.90
1	5	4	10.18	9.76
1	6	4	32.28	32.10
1	8	4	27.03	29.81
1	9	4	10.49	-10.26
1	10	4	4.35	6.27
1	12	4	15.90	16.22
1	14	4	11.21	13.98
1	16	4	8.91	10.03
1	1	5	15.79	14.03
1	2	5	10.54	10.45
1	3	5	37.11	35.58
1	4	5	17.75	17.28
1	5	5	29.57	31.03
1	6	5	6.09	6.70
1	7	5	20.07	23.12
1	8	5	6.71	-6.39
1	9	5	10.75	10.11
1	10	5	6.80	-6.41
1	11	5	9.17	11.27
1	13	5	8.80	12.44
1	15	5	7.52	9.45
1	0	6	34.91	34.51
1	2	6	34.52	31.48
1	3	6	9.35	-9.22
1	4	6	13.32	16.24
1	5	6	9.50	-7.47
1	6	6	9.50	7.65
1	8	6	6.57	9.03
1	10	6	9.59	11.65
1	12	6	11.29	14.70
1	14	6	8.51	11.38
1	1	7	18.99	18.47
1	3	7	13.37	14.28
1	5	7	11.33	11.04
1	7	7	9.82	12.16
1	9	7	9.63	10.48
1	11	7	9.43	11.34
1	2	8	11.52	12.03
1	4	8	11.85	14.76
1	6	8	14.45	14.68
1	8	8	8.13	9.79
1	10	8	8.15	7.73
1	1	9	13.04	13.01
1	3	9	7.42	9.85
1	5	9	8.04	7.88
-1	1	1	61.75	70.50
-1	2	1	8.46	-8.65
-1	3	1	29.08	34.69
-1	4	1	16.19	-8.45
-1	5	1	39.12	44.83
-1	6	1	17.21	-19.24
-1	7	1	23.98	27.36
-1	9	1	33.11	33.04
-1	11	1	22.99	24.26
-1	12	1	8.92	-7.59
-1	13	1	16.42	15.45
-1	14	1	6.84	6.09
-1	15	1	12.38	12.00
-1	17	1	8.03	7.06
-1	0	2	109.83	112.98
-1	1	2	58.12	-57.47
-1	2	2	35.30	38.37
-1	3	2	22.48	-21.42
-1	4	2	13.94	14.70
-1	5	2	11.69	-11.10
-1	6	2	11.07	13.35
-1	7	2	17.26	16.54
-1	8	2	26.58	27.26
-1	10	2	41.82	38.82
-1	12	2	23.50	26.36
-1	14	2	15.74	16.17
-1	16	2	9.10	9.02
-1	1	3	62.80	64.01
-1	2	3	14.31	10.37
-1	3	3	42.26	43.14
-1	4	3	22.98	19.14
-1	5	3	29.54	31.53
-1	6	3	17.75	16.94
-1	7	3	12.85	13.98
-1	8	3	7.98	7.03
-1	9	3	28.32	27.02
-1	11	3	27.28	27.87
-1	12	3	6.38	-7.96
-1	13	3	12.33	11.69
-1	15	3	10.02	12.85
-1	17	3	7.72	6.32
-1	0	4	68.91	74.47
-1	1	4	9.65	-6.87
-1	2	4	40.47	36.10
-1	3	4	25.11	-24.80
-1	4	4	30.30	30.09
-1	5	4	19.20	-16.83
-1	6	4	22.40	21.59
-1	8	4	26.25	24.96
-1	9	4	8.55	8.11
-1	10	4	24.56	23.33
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-4	0	8	9.30	10.48
-4	1	8	4.30	5.23
-4	2	8	11.58	12.56
-4	3	8	7.17	8.39
-4	4	8	12.86	14.03
-4	5	8	2.79	3.50
-4	6	8	9.53	10.62
-4	7	8	5.55	-4.90
-4	8	8	6.36	8.27
-4	10	8	6.14	5.14
-4	3	9	8.84	9.74
-4	5	9	5.24	6.63
-4	6	9	3.70	-3.75
-4	7	9	6.83	6.10
-4	8	9	3.53	-3.62
5	0	0	10.58	12.12
5	1	0	6.09	6.41
5	2	0	11.38	13.45
5	4	0	15.82	16.33
5	6	0	18.23	19.66
5	8	0	8.55	8.03
5	10	0	7.52	5.09
5	1	1	10.25	10.82
5	2	1	6.47	3.15
5	3	1	12.81	12.84
5	5	1	14.41	16.30
5	6	1	8.65	-1.68
5	7	1	7.61	8.73
5	9	1	6.42	6.47
-5	3	1	13.23	13.85
-5	5	1	21.68	24.91
-5	7	1	15.97	17.11
-5	8	1	2.64	-3.63
-5	9	1	8.69	11.23
-5	11	1	6.42	6.37
-5	0	2	24.37	23.46
-5	1	2	3.64	3.28
-5	2	2	12.33	11.13
-5	3	2	8.22	7.22
-5	4	2	13.65	14.39
-5	6	2	12.14	10.99
-5	7	2	3.35	3.12
-5	8	2	11.06	10.95
-5	10	2	9.26	10.24
-5	1	3	28.43	25.78
-5	3	3	13.51	14.00
-5	5	3	9.69	9.28
-5	6	3	5.90	5.11
-5	7	3	9.07	6.74
-5	8	3	3.83	-0.64
-5	9	3	8.79	10.17
-5	10	3	4.53	-2.62
-5	11	3	11.92	10.49
-5	0	4	17.34	13.49
-5	2	4	13.60	10.47
-5	3	4	13.56	-7.95
-5	4	4	18.61	15.23
-5	6	4	12.81	11.30
-5	8	4	11.62	11.78
-5	10	4	10.59	11.73
-5	1	5	20.03	19.17
-5	3	5	9.59	10.71
-5	5	5	7.51	7.12
-5	7	5	7.80	7.95
-5	9	5	10.39	10.21
-5	11	5	7.87	9.25
-5	0	6	25.04	22.21
-5	2	6	20.21	19.30
-5	10	6	9.37	10.11
-5	1	7	9.73	9.48
-5	2	7	5.53	-5.85
-5	3	7	8.65	9.17
-5	4	7	7.46	-7.99
-5	5	7	8.07	8.03
-5	7	7	7.03	9.40
-5	9	7	10.18	10.39
-5	0	8	7.18	7.02
6	0	0	11.57	11.95
6	2	0	10.82	10.82
6	4	0	10.70	10.04
-6	1	1	6.80	7.47
-6	2	1	4.39	-5.21
-6	4	2	11.57	11.88
-6	6	2	14.52	14.73
-6	3	1	10.88	9.53
-6	4	1	3.47	-4.96
-6	5	1	12.50	13.40
-6	6	1	4.00	-4.20
-6	2	2	6.05	6.27
-6	1	3	6.47	6.79
-6	2	3	4.01	4.59
-6	3	3	9.69	10.38
-6	4	3	2.64	4.21
-6	5	3	18.05	14.72
-6	7	3	7.87	7.86
-6	0	4	10.67	10.15
-6	2	4	7.65	8.19
-6	4	4	9.73	10.35
-6	6	4	12.09	11.51
-6	1	5	9.17	5.21
-6	3	5	8.74	7.52
-6	5	5	15.62	11.51
-6	7	5	8.10	7.57
-6	0	6	10.36	9.97
-6	2	6	6.70	7.00
-6	4	6	11.80	10.43
-6	6	6	5.27	7.11

The atoms C(2), C(3), C(4), N and Cu lie approximately on a plane expressed by $0{\cdot}5348X+0{\cdot}7574Y+0{\cdot}3747Z=3{\cdot}805$. The deviations of these atoms from the plane are $-0{\cdot}010$ for C(2), $-0{\cdot}003$ for C(3), 0·023 for C(4), $-0{\cdot}002$ for N and $-0{\cdot}009$ Å for Cu. The dihedral angle between the C(1)C(2)C(3) and C(2)C(3)C(4) planes is 68·4°; the conformation about the C(2)–C(3) bond is almost *gauche*. The carboxylate group is planar within 0·01 Å from the plane expressed by $0{\cdot}6782X+0{\cdot}1182Y-0{\cdot}7254Z=0{\cdot}2067$, and is rotated around the C(1)–C(2) bond by 23·9° from the C(1)C(2)C(3) plane and by 80° from the C(1)C(2)H(1) plane. Almost the same conformation has been recently found in free γ-aminobutyric acid (Tomita & Fujiwara, 1971). The two ligand molecules and the two copper(II) atoms form a fourteen-membered ring (see Fig. 1). The internal rotation angles around each bond in the ring are illustrated in Fig. 3. It is interesting to see that the ring conformation is similar to those observed in diazacyclotetradecanes (Dunitz & Meyer, 1965 and Brown, 1966), except for the N–Cu–O(1) angle and the conformation around the C(1)–C(2) bond.

The bond distances and angles of the ligand are in good agreement with those reported for other amino acids. The difference between the C(1)–O(1) and C(1)–O(2) distances reflects the strong bonding of the O(1) atom to the copper(II) atom. The C(4)–N distance is slightly shorter than the average value, 1·482 Å, given by Freeman (1966). The three C–C bond distances are within the range 1·511 to 1·522 Å, the average being 1·515 Å. The C(1)–C(2)–C(3), C(2)–C(3)–C(4), C(3)–C(4)–N and C(4)–N–Cu angles are significantly larger than the regular tetrahedral angle, 109·5°, as is usually observed in saturated hydrocarbon chains. The six C–H and two N–H bond distances are found to be within the range 0·87 to 1·04 Å, their average value being 0·96 Å. The bond angles of carbon and nitrogen atoms involving hydrogen atoms are all near to 109·5°. These results suggest the adequacy of the new weighting scheme applied in the present analysis.

One of the two hydrogen atoms of the amino group, H(7), participates in hydrogen bonding between the polymeric chains; the distances and angles with regard to this hydrogen bond are given in Table 8. The other hydrogen atom, H(8), does not participate in hydrogen bonding. As shown in Fig. 1, a considerably close approach of aliphatic carbon atoms exists between the

Table 8. *Distances and angles involving hydrogen bonding in* CuGABA-1

Standard deviations are added in parentheses. The superscript *B* refers to the equivalent position $(1-x,\ y-\frac{1}{2},\ \frac{1}{2}-z)$.

Distances (Å)	
N -----O(2^{B})	2·993 (6)
H(7)---O(2^{B})	2·11 (9)

Angles (°)	
N -----H(7)----O(2^{B})	155 (8)
H(7)---O(2^{B})—C(1^{B})	119 (2)
N -----O(2^{B})—C(1^{B})	126·1 (3)
H(7)---O(2^{B})···Cu^{B}	144 (2)
N -----O(2^{B})···Cu^{B}	143·0 (2)

Table 7. *Bond distances and angles for* CuGABA-2

Standard deviations are added in parentheses. The superscripts, i and ii, refer to the equivalent positions $(x-1,\frac{1}{2}-y,z-\frac{1}{2})$ and $(x+1,\frac{1}{2}-y,\frac{1}{2}+z)$.

Distances (Å)	
Cu—N^{i}	2·015 (5)
Cu—O(1)	1·959 (6)
Cu···O(2)	2·796 (6)
C(1)—O(1)	1·290 (11)
C(1)—O(2)	1·221 (12)
C(1)—C(2)	1·508 (13)
C(2)—C(3)	1·504 (18)
C(3)—C(4)	1·545 (13)
C(4)—N	1·446 (11)
C(2)—H(1)	1·15 (14)
C(2)—H(2)	1·16 (9)
C(3)—H(3)	1·16 (7)
C(3)—H(4)	1·25 (13)
C(4)—H(5)	0·89 (13)
C(4)—H(6)	1·08 (22)
N—H(7)	1·10 (10)
N—H(8)	1·00 (15)
O(W)-H(9)	0·96 (13)
O(W)-H(10)	0·83 (9)

Angles (°)	
O(1)-Cu—N^{i}	94·0 (2)
O(1)-Cu····O(2)	51·8 (2)
N^{i}—Cu····O(2)	90·6 (2)
C(1)-O(1)—Cu	111·5 (5)
C(1)-O(2)···Cu	73·4 (6)
O(1)-C(1)—C(2)	116·3 (8)

Angles (°)	
O(2)-C(1)—C(2)	120·2 (8)
O(1)-C(1)—O(2)	123·2 (9)
C(1)-C(2)—C(3)	110·3 (8)
C(2)-C(3)—C(4)	112·3 (9)
C(3)-C(4)—N	109·6 (7)
C(4)-N—Cu^{ii}	123·3 (5)
C(1)-C(2)—H(1)	110 (7)
C(3)-C(2)—H(1)	106 (7)
C(1)-C(2)—H(2)	107 (5)
C(3)-C(2)—H(2)	105 (5)
H(1)-C(2)—H(2)	119 (8)
C(2)-C(3)—H(3)	102 (3)
C(4)-C(3)—H(3)	112 (3)
C(2)-C(3)—H(4)	105 (6)
C(4)-C(3)—H(4)	112 (6)
H(3)-C(3)—H(4)	113 (7)
C(3)-C(4)—H(5)	106 (8)
N—C(4)—H(5)	109 (8)
C(3)-C(4)—H(6)	116 (11)
N—C(4)—H(6)	109 (11)
H(5)-C(4)—H(6)	106 (14)
C(4)-N—H(7)	108 (5)
Cu^{ii}-N—H(7)	114 (5)
C(4)-N—H(8)	110 (9)
Cu^{ii}-N—H(8)	102 (9)
H(7)-N—H(8)	95 (10)
H(9)-O(W)-H(10)	105 (10)

polymeric chains. The shortest C···C distance, 3·523 Å, is less than (by 0·477 Å) twice the van der Waals radius (2Å) of a methylenic group. This can be explained by the hydrogen atoms on the carbon atoms in question being in favourable positions to prevent close approaches between the hydrogens (see Fig. 4).

The apparent magnitudes and orientations of the principal axes of the temperature-factor ellipsoids are given in Table 9. The directions of maximum motion of the methylenic carbon and nitrogen atoms are nearly perpendicular to the zigzag plane. The same holds for those of the two oxygen atoms with respect to the carboxylate plane.

Table 9. *Description of the thermal ellipsoids in* CuGABA – 1

$B_i = 8\pi^2 u_i^2$ where u_i is the root-mean-square displacement corresponding to the ith axis of the ellipsoid. C_{ia}, C_{ib} and C_{ic} are the direction cosines of the ith axis with respect to the crystal axes, a, b and c.

	i	B_i	u_i	C_{ia}	C_{ib}	C_{ic}
Cu	1	1·28	0·127	−0·9682	−0·0353	0·0562
	2	2·42	0·175	0·0962	−0·9664	−0·2561
	3	2·94	0·193	0·2309	0·2541	−0·9651
O(1)	1	1·02	0·113	−0·9269	−0·2456	0·0093
	2	1·64	0·144	0·3363	−0·8793	−0·4231
	3	3·80	0·219	0·1666	0·4081	−0·9060
O(2)	1	1·24	0·126	−0·3718	0·1867	−0·1686
	2	2·40	0·174	0·1101	0·9756	0·1481
	3	4·47	0·238	−0·4773	−0·1159	0·9745
C(1)	1	0·78	0·099	−0·9751	−0·2163	0·2483
	2	1·70	0·147	−0·1795	0·6433	0·7638
	3	3·05	0·197	−0·1299	0·7345	−0·5958
C(2)	1	1·17	0·122	−0·9984	−0·0068	0·2479
	2	1·63	0·144	−0·0486	0·5844	0·7869
	3	3·40	0·207	0·0266	0·8115	−0·5646
C(3)	1	1·11	0·119	0·9334	0·0829	0·0511
	2	2·10	0·163	0·3588	−0·2236	−0·9723
	3	2·74	0·186	0·0030	0·9711	−0·2283
C(4)	1	1·24	0·125	0·9827	−0·1734	−0·2349
	2	1·78	0·150	0·1187	0·3227	−0·9311
	3	2·96	0·193	0·1420	0·9305	0·2791
N	1	1·59	0·142	−0·8779	0·2760	−0·1082
	2	2·08	0·162	−0·4778	−0·5579	0·7911
	3	2·80	0·188	−0·0312	0·7826	0·6022

The crystal structure of CuGABA-2

The crystal structure of CuGABA-2 is shown in Fig. 5. The principal difference between the structures of CuGABA-1 and CuGABA-2 is found in the mode of linkage of the ligand between two copper(II) atoms. In contrast to CuGABA-1, two copper(II) atoms are connected by a single bridge of the ligand to form an infinite two-dimensional network extending parallel to (201), and each copper(II) atom is located at the knot of the network. The nearest neighbours of the copper(II) atom are N, O(1) and those atoms related by the centre of symmetry; these atoms forms a square planar coordination. This coordination plane is expressed by $-0{\cdot}4781X-0{\cdot}1629Y+0{\cdot}8631Z=0$. The Cu–N and Cu–O(1) distances and the N–Cu–O(1) angle are comparable with the corresponding values observed in CuGABA-1. The second neighbours, the O(2) atoms, occupy the apical positions of the distorted octahedron. The O(1)–Cu···O(2) and N–Cu···O(2) angles are 51·8 and 90·6°, and the Cu···O(2) direction makes an angle

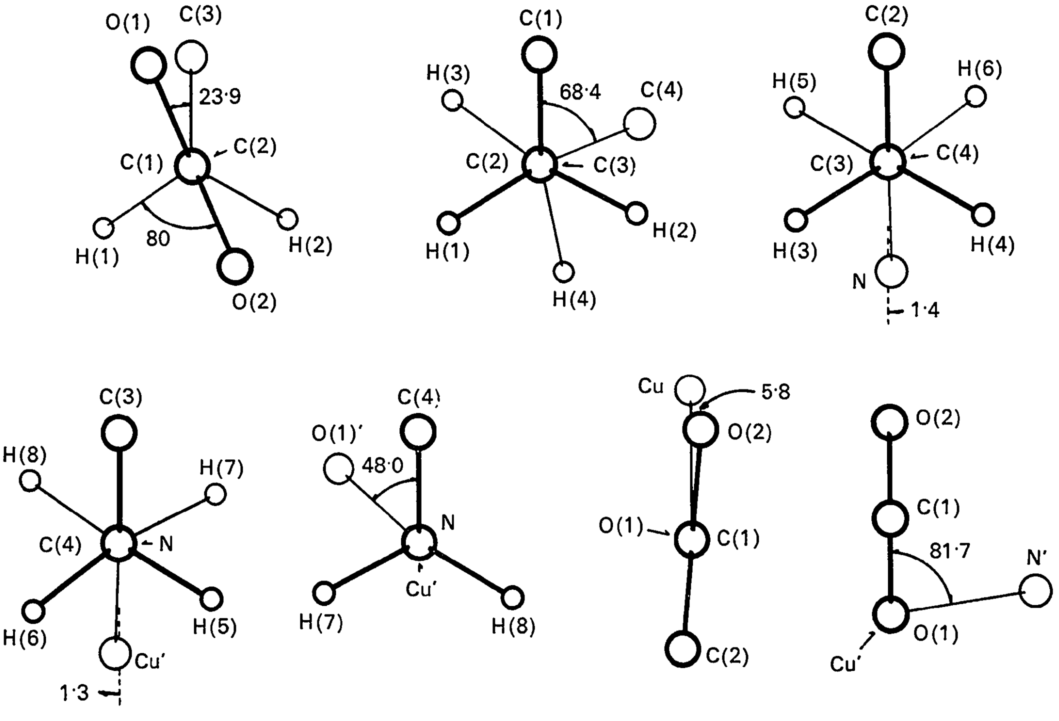
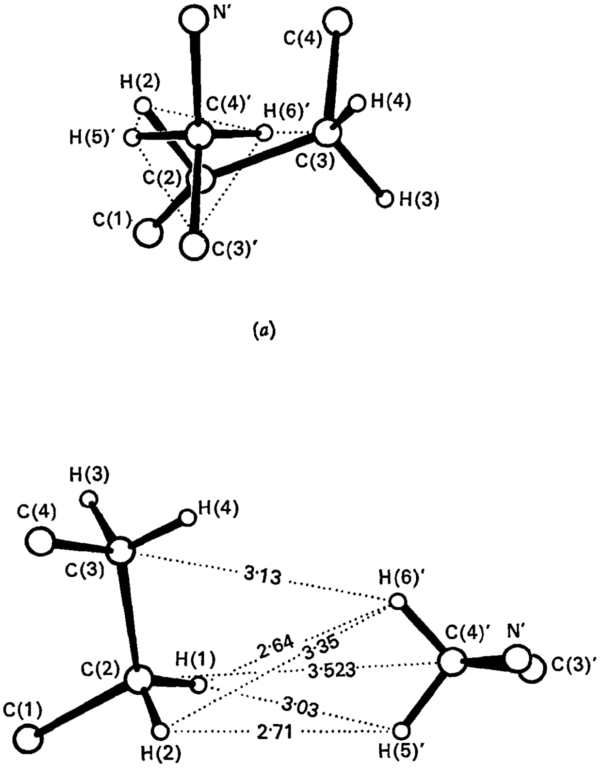


Fig. 3. Internal rotation angles around each bond in the ring of CuGABA-1.

38·3° with the normal to the coordination plane. It is interesting to see that the coordination about the copper(II) atom is quite similar to that found in CuGABA-1 (see Fig. 6). However, the Cu···O(2) distances is longer than that in CuGABA-1 by 0·155 Å and it appears that the difference in colour between CuGABA-1 and CuGABA-2 is due to this difference in the Cu···O(2) distances.



(a)

(b)

Fig. 4. The closest approaches of methylenic groups between the two different polymeric chains in CuGABA-1. (a) View along the direction of the sum vector of C(4)'–H(5)' and C(4)'–H(6'), and (b) a view along the normal to the H(5)'C(4)'H(6)' plane.

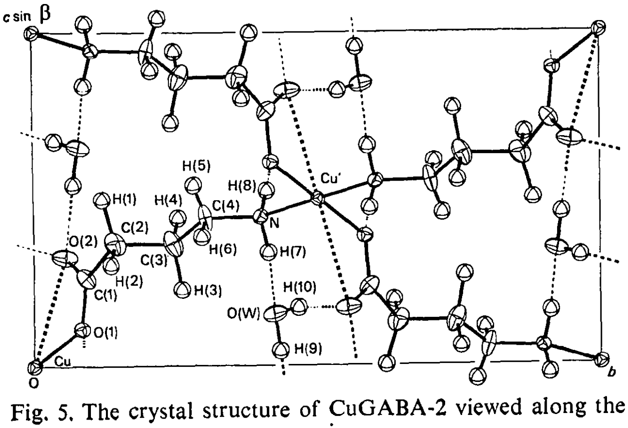
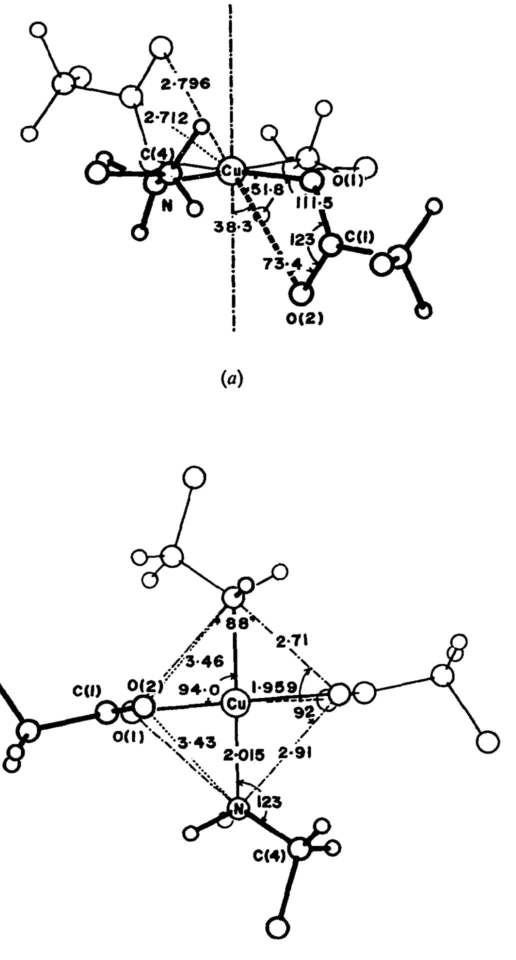


Fig. 5. The crystal structure of CuGABA-2 viewed along the *a* axis.



(a)

(b)

Fig. 6. The coordination about the copper(II) atom in CuGABA-2. Projections are (a) along the direction making an inclination angle of 10° from the coordination plane and (b) normal to the coordination plane.

The carboxylate group is planar within 0·02 Å of the plane expressed by $-0\cdot512X+0\cdot844Y-0\cdot158Z=0\cdot243$; the copper(II) atom deviates by 0·243 Å from this plane. That the C(1)–O(1) distance is longer than the C(1)–O(2) distance by 0·07 Å, reflects the strong bonding of the O(1) atom to the copper(II) atom. The C(4)–N distance is somewhat shorter than the average value given by Freeman (1966). Such a short distance has been found in several other metallic complexes of amino acids in recent years; *e.g.* 1·45 Å in *cis*-bis-(D-alaninato)copper(II) (Gillard Mason, Payne & Robertson, 1969) and in *trans*-bis-(DL-*N*,*N*-diethyl-α-alaninato)copper(II) (Nash & Schaefer, 1969) and 1·446 Å in *cis*-diaquo-bis-(L-serinato)-nickel-(II) (Van der Helm & Hossain, 1969). The three C–C bond distances are within the range 1·50 to 1·55 Å, the average value being 1·52 Å. The three bond angles C(1)–C(2)–C(3), C(2)–C(3)–C(4) and C(3)–C(4)–N, in the methylenic chain are definitely larger than 109·5° as are those found in CuGABA-1. The large value, 123·5°, found for the C(4)–N–Cu angle may be due to the very close approach of the O(1) atom of the adjacent ligand in the network to the C(4) atom. The two hydrogen atoms attached to the C(4) atom are located at positions rather distant from the O(1) atom in question (see Fig. 7). Covalent bond distances and angles involving hydrogen atoms are all very close to the corresponding normal values.

The methylenic chain including the C(1) and N atoms assumes an almost planar conformation. The mean plane is expressed by $-0{\cdot}516X-0{\cdot}100Y+0{\cdot}851Z=1{\cdot}284$ and the deviations of the atoms from the plane are −0·07 for C(1), 0·07 for C(2), 0·06 for C(3), −0·07 for C(4) and 0·01 Å for N. Slight internal rotations are observed from the planar *trans* conformation around the C–C and C–N bonds (see Fig. 8), as has almost invariably been observed in other similar compounds. The carboxylate plane is rotated by 85° from the C(1)C(2)C(3) plane around the C(1)–C(2) bond. The conformation of the part of the amino acid residue, C(1), C(2), C(3), C(4) and N, is almost the same as that found in γ-aminobutyric acid hydrochloride (Tomita, 1965), where the carboxyl group is coplanar with the corresponding C(1)C(2)C(3) plane.

The crystal contains two water molecules in the unit cell. The water oxygen atom, O(*W*), is not coordinated

Table 10. *Distances and angles involving hydrogen bonding in* CuGABA – 2

Standard deviations are added in parentheses. The superscripts refer to the equivalent positions:

ii	$x+1, \frac{1}{2}-y, \frac{1}{2}+z$	iii	$x, \frac{1}{2}-y, \frac{1}{2}+z$
iv	$x, \frac{1}{2}-y, z-\frac{1}{2}$	v	$\bar{x}, \frac{1}{2}+y, \frac{1}{2}-z$

Distances (Å)

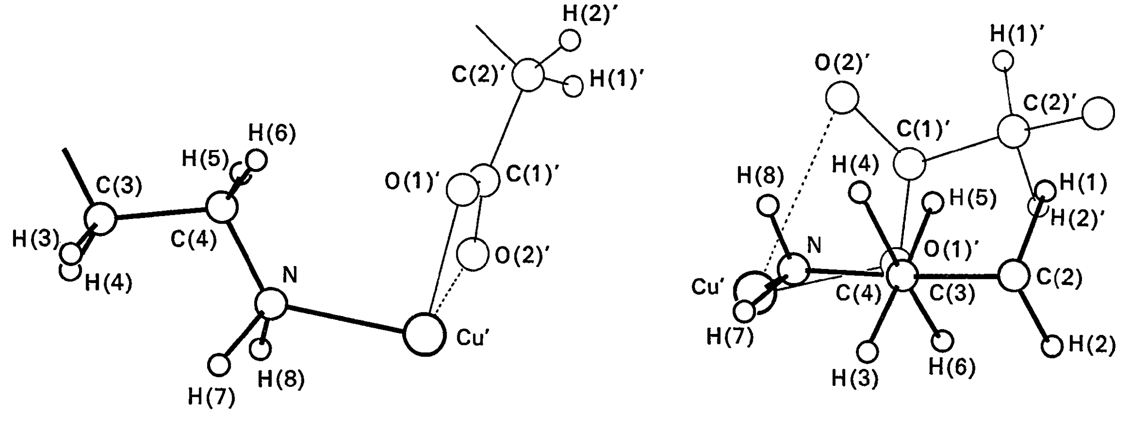
N-------O(W)	2·977 (8)
N-------O(1^{iii})	2·996 (9)
O(W)---O(2^{iv})	2·837 (10)
O(W)---O(2^{v})	2·862 (11)
H(7)----O(W)	1·90 (10)
H(8)----O(1^{iii})	2·05 (14)
H(9)----O(2^{iv})	1·88 (13)
H(10)---O(2^{v})	2·05 (9)

Angles (°)

N——H(8)-----O(1^{iii})	157 (11)
C(4)—N-------O(1^{iii})	108·8 (5)
Cu^{ii}—N-------O(1^{iii})	113·9 (2)
C(1^{iii})–O(1^{iii})---H(8)	122 (4)
C(1^{iii})–O(1^{iii})---N	128·9 (5)
N——H(7)----O(*W*)	174 (10)

Angles (°)

C(4)—N-------O(W)	104·3 (5)
Cu^{ii}—N-------O(W)	117·9 (3)
H(9)—O(W)---H(7)	128 (8)
H(10)—O(W)---H(7)	108 (7)
O(W)–H(9)----O(2^{iv})	179 (10)
C(1^{iv})—O(2^{iv})---H(9)	124 (4)
C(1^{iv})—O(2^{iv})---O(W)	123·6 (6)
Cu^{iv}···O(2^{iv})---H(9)	163 (4)
Cu^{iv}···O(2^{iv})---O(W)	162·9 (3)
O(W)–H(10)---O(2^{v})	166 (8)
C(1^{v})—O(2^{v})---H(10)	153 (3)
C(1^{v})—O(2^{v})---O(W)	155·2 (7)
Cu^{v}···O(2^{v})---H(10)	80 (2)
Cu^{v}···O(2^{v})---O(W)	82·7 (3)
O(2^{v})--O(W)---O(2^{iv})	99·4 (3)
O(2)--O(W)---N	118·3 (3)
O(2^{iv})--O(W)---N	129·3 (3)



(*a*) (*b*)

Fig. 7. Interatomic close approaches between the ligands in the network, viewed (*a*) perpendicular to the C(3)C(4)N plane and (*b*) parallel to the C(3)–C(4) bond. C(4)---O(1′) 3·09, C(4)---C(1′), 3·62, H(5)---O(1′) 2·93, H(5)---C(1′) 3·17, H(5)---H(3)′ 3·64, H(5)---H(4)′ 2·91, H(6)---O(1)′ 2·84 Å.

directly to the copper(II) atom, but is located in the cavity within the network and participates in hydrogen bonding as an acceptor for the N–H(7) group. The two hydrogen atoms of the water molecule, H(9) and H(10), both participate in hydrogen bonding as donors for the two O(2) atoms in the adjacent networks above and below (see Fig. 5). In this way the water molecule plays an important role in the formation of the crystal. Yet another hydrogen bond, N–H(8)···O(1), is found between the networks. The distances and angles with regard to these hydrogen bonds are given in Table 10.

The magnitudes and direction cosines of the principal axes of the thermal motion of the atoms, as derived from the parameters given in Table 5, are listed in Table 11. The directions of maximum motion of the methylenic carbon atoms are all nearly perpendicular to the zigzag plane, and the two oxygen atoms of the carboxylate group have their directions of maximum amplitude almost perpendicular to the carboxylate plane.

All the calculations were carried out on a FACOM 270-20 at the Computing Centre of this University, with crystallographic programs developed in our laboratory by A. Furusaki, A. Takenaka and others. Figs. 1 and 5 were drawn using the program *DEAM* (Takenaka, 1972). The atomic scattering factors used for structure-factor calculations were obtained by using two-point interpolation from tables of f_j *vs.* an equal interval of sin θ/λ (0·01), which were prepared by six-point interpolation of the values taken from *International Tables for X-ray Crystallography* (1962).

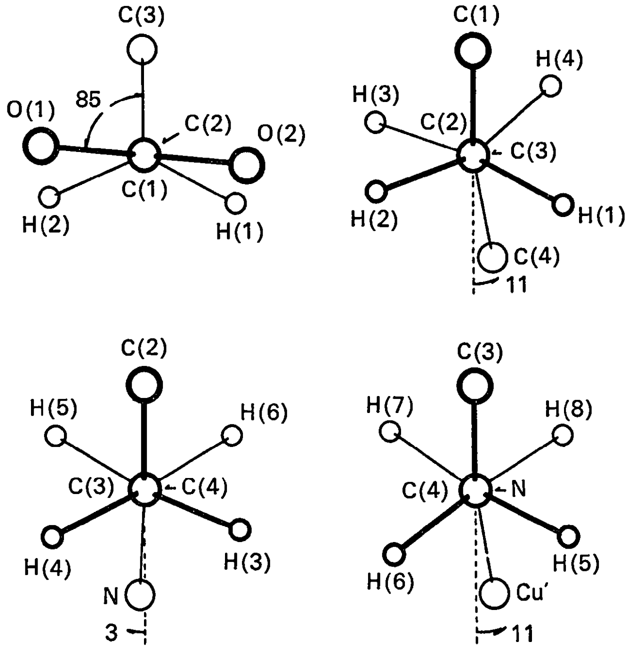


Fig. 8. Internal rotation angles around the C–C and C–N bonds in CuGABA-2.

Table 11. *Description of the thermal ellipsoids in* CuGABA – 2

$B_i = 8\pi^2 u_i^2$ where u_i is the root-mean-square displacement corresponding to the ith axis of the ellipsoid. C_{ia}, C_{ib} and C_{ic} are the direction cosines of the ith axis with respect to the crystal axes, *a*, *b* and *c*.

	i	B_i	u_i	C_{ia}	C_{ib}	C_{ic}
Cu	1	2·21	0·167	−0·5213	0·4829	−0·4932
	2	2·61	0·182	−0·6364	−0·7695	0·1592
	3	3·01	0·195	0·5660	−0·4216	−0·8551
O(1)	1	2·31	0·171	0·7382	0·4171	0·2582
	2	2·68	0·184	−0·4182	−0·3388	0·9334
	3	3·53	0·211	0·5301	−0·8448	−0·2431
O(2)	1	3·05	0·197	−0·8288	−0·1471	0·7823
	2	3·22	0·202	0·5061	0·2231	0·6205
	3	8·89	0·336	−0·2431	0·9637	−0·0243
C(1)	1	2·07	0·162	0·8015	0·3977	0·1584
	2	3·63	0·214	0·5961	−0·5899	−0·7103
	3	5·94	0·274	0·0468	0·7027	−0·6859
C(2)	1	2·50	0·178	0·6723	0·4929	0·3007
	2	6·98	0·297	0·5969	−0·8023	−0·2065
	3	8·04	0·319	−0·4378	−0·3374	0·9310
C(3)	1	2·97	0·194	0·6300	0·6217	0·2325
	2	4·78	0·246	0·7434	−0·6561	−0·3670
	3	9·60	0·349	0·2246	0·4278	−0·9007
C(4)	1	2·05	0·161	0·1028	0·9544	0·2308
	2	4·28	0·233	0·9910	−0·1224	−0·2754
	3	9·01	0·338	0·0852	0·2721	−0·9332
N	1	2·11	0·163	0·6250	0·1860	0·5106
	2	2·70	0·185	−0·2578	−0·8680	0·4855
	3	3·92	0·223	0·7376	−0·4622	−0·7983
O(*W*)	1	2·48	0·177	−0·3845	0·2578	−0·7108
	2	6·26	0·282	0·9168	−0·0172	−0·6781
	3	7·15	0·301	0·1192	0·9659	0·1779

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Die Strukturbestimmung der Isomaltulose, $C_{12}H_{22}O_{11}.H_2O$

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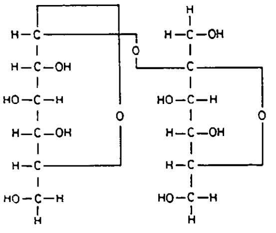
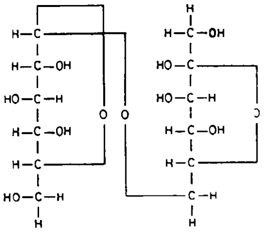
The crystal structure of 6-[α-D-glucopyranosyl]-D-fructofuranose, chemical formula $C_{12}H_{22}O_{11}.H_2O$, also called isomaltulose, has lattice parameters $a=9{\cdot}052$, $b=12{\cdot}155$, $c=14{\cdot}127$ Å, space group $P2_12_12_1$. The phase problem could be solved by application of the tangent formula. The refinement of the diffractometer data by least-squares methods led to a final R value of 0·039. The conformation of the furanosyl ring is $_3T^4$, but very near to E_3. The (1 → 6) linkage between the pyranosyl and furanosyl parts of the molecule is found to be different from the comparable bond in planteose. One intramolecular and several intermolecular hydrogen bonds could be found. Every oxygen atom of an O–H group participates in two hydrogen bonds, as donor as well as an acceptor.

Die Isomaltulose entsteht bei der Einwirkung von aus dem Zuckerfabrikationsprozess stammenden Bakterien auf Saccharose in einer bakteriell-enzymatischen Transferierungsreaktion (Weidenhagen & Lorenz 1957*a*, *b*). Die dabei wirkende Transglucosylase spaltet zuerst die Saccharose unter Bildung eines Glucose-Enzym-Komplexes und freier D-Fructose. In einem zweiten Reaktionsschritt wird dann die gebundene D-Glucose auf freie Fructose in 1,6-Position unter Bildung von Isomaltulose übertragen (Mauch & Schmidt-Berg-Lorenz, 1964*a*, *b*).


Saccharose


Isomaltulose

α-D-Glucopyranosyl-β-D-fructofuranosid + Enzym → α-Glucose-Enzym + Fructose → 6[α-D-Glucopyranosyl]-D-fructofuranose + Enzym.

Auffallend ist die ausserordentlich feste Bindung von 1 Mol Wasser an die Isomaltulose, das erst im Vakuum bei hoher Temperatur kurz unterhalb des Schmelzpunktes vollständig abgegeben wird. Aus diesem Grunde wurde vorübergehend sogar eine chemische Bindung des Wassers als Ketohydrat diskutiert. Durch chemische Nachweismethoden konnte jedoch eine solche Acetalbindung später ausgeschlossen werden (Loss, 1967).

Obwohl die Konstitution des Moleküls bereits weitgehend mit chemischen Methoden untersucht war, sollten durch die Röntgenstrukturanalyse weitere Informationen sowohl über den Molekülaufbau, als auch über das Kristallgitter und die darin vorliegenden Wasserstoffbrückenbindungen gewonnen werden.

Experimente

Die Kristalle wurden vom Institut für Zuckerindustrie der Technischen Universität Berlin gezüchtet und uns freundlicherweise zur Verfügung gestellt.

Aus orientierenden Drehkristall- und Weissenbergaufnahmen konnten vorläufige Gitterkonstanten und wegen der rhombischen Intensitätssymmetrie und der Auslöschungen $h00$, $0k0$ und $00l$ für ungerade Ordnungen die Raumgruppe zu $P2_12_12_1$ bestimmt werden.

Auf einem automatischen Einkristalldiffraktometer der Fa. Siemens (AED) wurden die genauen Gitterkonstanten und die Intensitäten der unabhängigen Reflexen nach der Fünfwertmethode vermessen. Hierbei wurde ein Kristall einer Grösse von ca 0,2 × 0,2 × 0,3 mm verwendet. Als unbeobachtet wurden Reflexe eingestuft, deren Intensität kleiner als der doppelte statistische Fehler war. Auf eine Absorptionskorrektur wurde wegen der isotropen Kristallform und des relativ geringen linearen Absorptions-Koeffizienten verzichtet.

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