has been presented in detail in a separate publication (Sax & Pletcher, 1969).

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The Crystal Structure of Bis-(*H*-pyrrole-2-aldimine)copper(II), $(C_5H_5N_2)_2$ Cu(II)

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The crystal structure of bis-(*H*-pyrrole-2-aldimine)copper(II) has been determined using three-dimensional data collected on a diffractometer equipped with a single-crystal orienter. The space group is $P2_1/c$ with $Z=2$; cell dimensions are $a=9{\cdot}845$ (2), $b=5{\cdot}562$ (3), $c=9{\cdot}604$ (3) Å and $\beta=103{\cdot}07$ (1)°. The Cu^{2+} ions occupy special positions (0,0,0) and $(0,\frac{1}{2},\frac{1}{2})$ and the molecule must lie on a centre of symmetry. The structure was elucidated by the heavy-atom method and was refined by the full-matrix least squares technique. The final R is 0·061. The Cu^{2+} ion is coordinated with four N atoms in a squareplanar arrangement; the two unique Cu–N distances are 1·97 (1) and 1·95 (1) Å. The molecule is essentially planar, the maximum deviation from the best least-squares plane being 0·06 Å.

Introduction

The preparation and chemical properties of pyrrole-2-aldimine chelates of copper have been reported by Enmart, Diehl & Collwitzer (1929) and Pfieffer, Hesse, Pfitzinger, Scholl & Theriot (1937). The electronic spectra of these chelates have been reported by Chakravorty & Kannan (1967). Stakleberg (1947) made some preliminary crystallographic studies of bis-(*H*-pyrrole-2-aldimine)copper (II), to which he assigned the space group $P2_1/c$. The complete crystal structure of any pyrrole-2-aldimine complex has not yet been reported in the literature. These chelates share common structural features with porphyrins which are of great biological significance. For these reasons and because of the interest in the study of coordination configurations of Cu^{2+}, the crystal structure of bis-(*H*-pyrrole-2-aldimine)copper(II) was determined.

Experimental

The crystals of bis-(*H*-pyrrole-2-aldimine)copper(II) were grown by slow evaporation of its solution in A.R. grade toluene. Flat prismatic crystals of deeppurple-brown colour were obtained. Suitable crystals of approximate dimensions 0·20 × 0·15 × 0·15 mm were chosen for X-ray studies.

Preliminary studies were carried out with rotation and Weissenberg photographs. The crystals were found to be monoclinic and showed the following systematic absences: $h0l$ reflexions absent when $l=2n+1$ and $0k0$ reflexions absent when $k=2n+1$. These systematic absences fix the space group uniquely as $P2_1/c$.

Table 1. *Crystal data*

$(C_5H_5N_2)_2Cu(II)$
Monoclinic, $P2_1/c$
$a=9{\cdot}845\pm0{\cdot}002$ Å
$b=5{\cdot}562\pm0{\cdot}003$
$c=9{\cdot}604\pm0{\cdot}003$
$\beta=103{\cdot}07\pm0{\cdot}01°$
$D_m=1{\cdot}59\pm0{\cdot}02$ g.cm^{-3}
$D_c=1{\cdot}62$ g.cm^{-3}
$\mu_c=28{\cdot}14$ cm^{-1}

Cell dimensions were determined on a General Electric XRD-6 diffractometer from observations on axial reflexions. Density was measured by flotation in an aqueous solutions of $ZnCl_2$. The cell parameters and other crystal data are listed in Table 1.

Three-dimensional intensity data were collected with Cu $K\alpha$ radiation using the stationary-crystal stationary-counter technique. Intensity measurements were made in shells of 2θ to a maximum of 100°. Beyond this point more than $\frac{2}{3}$ of the reflexions had intensities almost equal to the background and it was not considered worth while to collect data. Thus, intensity data for 730 reflexions were recorded. Reflexions whose intensities differed from background by less than one standard deviation of the background were classified as unobserved. There were 178 such reflexions.

The 020, 040, and 060 reflexions for which $\chi=90°$ were examined through 360° rotation of the angle φ, and no appreciable variation in their intensities was noticed. This indicated that the absorption correction was negligibly small. Consequently, no absorption correction was applied. The intensity data were corrected for background. Also, Lorentz and polarization corrections were applied.

Structure determination

With two molecules to a unit cell of space group $P2_1/c$, the molecules must lie on the centre of symmetry. Therefore, the Cu^{2+} ions were assigned the special positions (0,0,0) and $(0,\frac{1}{2},\frac{1}{2})$ and all other atoms were located by a series of three-dimensional Fourier and difference Fourier syntheses. In all these calculations

Table 2. *Atomic positions* (× 10⁴) *and anisotropic thermal parameters* (× 10⁴)

Estimated standard deviations are given in parentheses. Temperature factors are at the form:

$$\exp\left[-(h^2b_{11}+k^2b_{22}+l^2b_{33}+2hkb_{12}+2hlb_{13}+2klb_{23})\right]$$

Positions

	x	y	z
Cu^{2+}	0	0	0
N(1)	1287 (10)	2648 (17)	−160 (10)
N(2)	1492 (9)	−875 (15)	1629 (8)
C(1)	2482 (14)	2543 (23)	813 (13)
C(2)	2600 (11)	644 (17)	1798 (11)
C(3)	3687 (12)	−78 (27)	2931 (13)
C(4)	3199 (14)	−2213 (25)	3474 (14)
C(5)	1829 (14)	−2603 (23)	2641 (13)
H(1)	1290	2890	−260
H(2)	2560	2790	770
H(3)	3800	60	2960
H(4)	3210	−2380	3570
H(5)	1730	−2850	2690

Thermal parameters

	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
Cu^{2+}	156 (2)	316 (7)	129 (3)	−32 (5)	37 (2)	−7 (5)
N(1)	161 (14)	334 (37)	142 (13)	−52 (19)	33 (11)	9 (19)
N(2)	120 (12)	226 (31)	112 (11)	−5 (15)	28 (9)	−12 (15)
C(1)	160 (18)	460 (55)	168 (19)	−47 (26)	53 (15)	−33 (27)
C(2)	123 (15)	275 (44)	115 (14)	3 (18)	32 (10)	−28 (17)
C(3)	174 (17)	431 (49)	180 (18)	53 (35)	48 (14)	−57 (35)
C(4)	183 (21)	404 (55)	182 (20)	28 (28)	36 (16)	0 (28)
C(5)	193 (21)	390 (52)	148 (17)	44 (26)	64 (15)	14 (25)

an overall temperature factor of 4·8 Å^2, as determined by the method of Wilson (1942), was used. Atomic positions from electron density peaks were deduced following the method of Ladell & Katz (1954).

The structure was refined by the full-matrix least-squares technique, using the adapted version of program *ORFLS* (Busing, Martin & Levy, 1962). In least-squares calculations all observed reflexions were given unit weights and the unobserved zero weights. Anisotropic temperature factors were included in the refinement during the last three cycles and the *R*, on observed reflexions only, was 0·063.

A three-dimensional difference Fourier synthesis at this stage revealed two out of five hydrogen atoms. The other three hydrogen atoms were assigned such that the C–H bond was 1·08 Å and coplanar with the two adjacent bonds, making equal angles with them. One cycle of full-matrix least-squares refinement was performed, including the hydrogen atoms but varying only the positional and anisotropic temperature-factor parameters of non-hydrogen atoms. This reduced *R* to 0·061 for observed reflexions only. The *R* with unobserved reflexions included was 0·080. The least-squares refinement was stopped at this stage because the maximum shift in any parameter was less than $\frac{1}{3}$ its σ.

Scattering factors for the calculations were taken from *International Tables for X-ray Crystallography* (1962).

Final atomic coordinates are listed in Table 2. Hydrogen atoms are numbered from H(1) to H(5) corresponding to the respective atoms N(1), C(1), C(3),

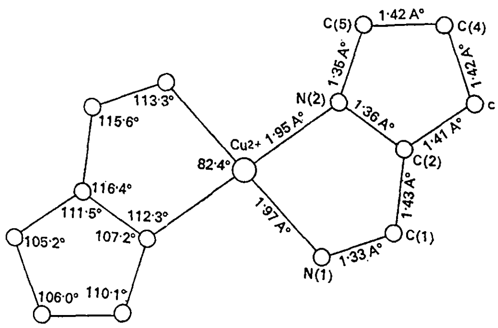

Fig. 1. *X–Z* projection of the crystal structure.

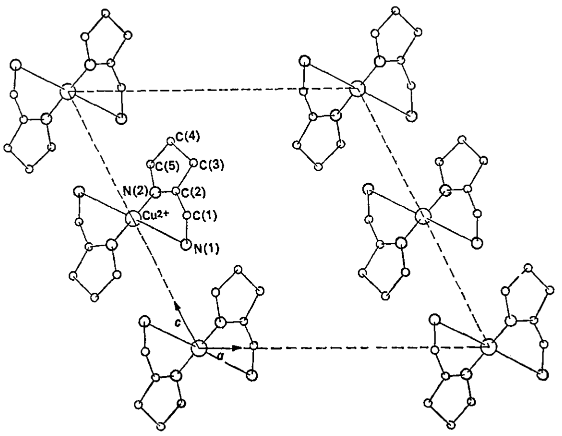

Fig. 2. A molecule of bis-(*H*-pyrrole-2-aldimine)copper(II).

Table 3. *Observed and calculated structure factors*

* indicates the unobserved reflexions

H	K	L	Y/OBS	YCALC	H	K	L	YOBS	YCALC	H	K	L	YOBS	YCALC	H	K	L	YOBS	YCALC	H	K	L	YOBS	YCALC
1	0	0	693	693	-4	0	6	166	151	-9	1	1	94	119	8	1	4	26	11*	4	1	8	26	21*
2	0	0	234	286	-5	0	6	180	175	-10	1	1	26	23*	-9	1	4	26	19*	-5	1	8	28	19*
3	0	0	207	222	6	0	6	113	97	10	1	1	25	37*	9	1	4	23	3*	5	1	8	24	29*
4	0	0	119	142	-6	0	6	173	168	0	1	2	267	264	-10	1	4	44	47	-6	1	8	28	20*
5	0	0	206	232	7	0	6	88	94	1	1	2	287	291	0	1	5	304	291	-7	1	8	27	10*
6	0	0	282	275	-7	0	6	166	201	-1	1	2	641	617	1	1	5	444	412	-8	1	8	26	18*
7	0	0	235	241	-8	0	6	134	127	2	1	2	46	49	-1	1	5	232	233	0	1	9	51	39
8	0	0	218	210	-9	0	6	57	53	-2	1	2	618	596	2	1	5	224	240	1	1	9	72	92
9	0	0	75	62	-10	0	6	42	34	3	1	2	205	184	-2	1	5	133	140	-1	1	9	74	69
10	0	0	26	2*	0	0	8	109	108	-3	1	2	167	172	3	1	5	117	123	2	1	9	65	77
0	0	2	155	159	1	0	8	140	124	4	1	2	45	52	-3	1	5	232	228	-2	1	9	146	146
1	0	2	459	478	-1	0	8	193	194	-4	1	2	280	290	4	1	5	192	189	3	1	9	59	61
-1	0	2	796	805	2	0	8	111	114	5	1	2	141	136	-4	1	5	365	352	-3	1	9	130	124
2	0	2	296	300	-2	0	8	205	205	-5	1	2	108	117	5	1	6	83	90	-4	1	9	114	102
-2	0	2	916	952	3	0	8	45	54	6	1	2	75	90	-5	1	5	195	198	-5	1	9	80	77
3	0	2	476	446	-3	0	8	128	113	-7	1	2	25	32*	6	1	5	58	51	-6	1	9	57	67
-3	0	2	103	110	4	0	8	65	70	7	1	2	27	26*	-6	1	5	194	187	-7	1	9	25	53*
4	0	2	388	418	-4	0	8	27	23*	-8	1	2	27	10*	7	1	5	89	89	0	1	10	25	13*
-4	0	2	215	238	5	0	8	64	69	8	1	2	78	0*	-7	1	5	191	178	1	1	10	29	30
5	0	2	46	40	-5	0	8	83	76	-9	1	2	28	5*	8	1	5	66	69	-1	1	10	30	33
-5	0	2	294	292	-6	0	8	177	172	9	1	2	27	20*	-8	1	5	79	67	-2	1	10	26	7*
6	0	2	146	145	-7	0	8	95	103	-10	1	2	27	12*	-9	1	5	47	39	-3	1	10	26	11*
-6	0	2	89	80	-8	0	8	50	52	10	1	2	23	10*	-10	1	5	41	37	-4	1	10	25	6*
7	0	2	230	206	-9	0	8	24	61*	0	1	3	231	240	0	1	6	24	17*	-5	1	10	39	38
-7	0	2	125	123	0	0	10	59	58	1	1	3	276	288	1	1	6	22	19	-6	1	10	38	44
8	0	2	84	74	1	0	10	105	116	-1	1	3	130	151	2	1	6	48	47	0	2	0	162	113
-8	0	2	220	214	-1	0	10	26	14*	2	1	3	338	355	-2	1	6	109	108	1	2	0	229	220
9	0	2	57	51	-2	0	10	46	45	-2	1	3	692	624	-3	1	6	75	68	2	2	0	532	542
-9	0	2	208	201	-3	0	10	43	117	3	1	3	428	439	-4	1	6	113	97	3	2	0	452	469
10	0	2	60	53	-4	0	10	98	102	-3	1	3	548	579	-5	1	6	85	85	4	2	0	246	265
-10	0	2	67	64	-5	0	10	67	68	4	1	3	218	219	-6	1	6	27	4	5	2	0	252	282
0	0	4	419	421	-6	0	10	34	42	-4	1	3	225	243	6	1	6	27	7*	6	2	0	159	144
1	0	4	416	397	1	1	0	27	24	5	1	3	208	214	-7	1	6	28	19*	7	2	0	114	101
-1	0	4	492	503	2	1	0	104	112	-5	1	3	316	312	7	1	6	25	16*	8	2	0	138	118
2	0	4	578	566	3	1	0	46	36	6	1	3	194	173	-8	1	6	28	11*	9	2	0	35	36
-2	0	4	152	155	4	1	0	18	14*	-6	1	3	183	182	-9	1	6	27	6*	10	2	0	24	29*
3	0	4	177	179	5	1	0	152	151	7	1	3	29	27	-10	1	6	24	7	0	2	1	53	68
-3	0	4	150	137	6	1	0	60	55	-7	1	3	80	95	0	1	7	222	222	1	2	1	117	82
4	0	4	69	77	7	1	0	160	164	-8	1	3	106	94	1	1	7	73	71	-1	2	1	78	9
-4	0	4	219	205	8	1	0	27	19*	-8	1	3	28	40*	-1	1	7	276	261	2	2	1	148	152
5	0	4	190	180	9	1	0	28	42*	9	1	3	77	70	2	1	7	101	109	-2	2	1	138	124
-5	0	4	326	330	10	1	0	26	13*	-9	1	3	88	64	-2	1	7	202	193	3	2	1	59	32
6	0	4	78	58	0	1	1	538	540	-10	1	3	103	95	3	1	7	119	123	-3	2	1	137	131
-6	0	4	340	352	1	1	1	129	127	0	1	4	37	54	-3	1	7	155	149	4	2	1	133	130
7	0	4	59	60	-1	1	1	948	983	1	1	4	24	49	4	1	7	68	68	-4	2	1	88	78
-7	0	4	157	152	2	1	1	420	429	-1	1	4	33	33	-4	1	7	109	104	5	2	1	30	42
8	0	4	116	103	-2	1	1	290	297	-2	1	4	25	30	5	1	7	77	83	-5	2	1	49	23
-8	0	4	56	58	3	1	1	263	268	2	1	4	20	16*	-5	1	7	158	150	6	2	1	72	62
9	0	4	56	68	-3	1	1	289	327	3	1	4	49	54	6	1	7	56	75	-6	2	1	93	82
-9	0	4	67	58	4	1	1	329	328	-3	1	4	177	176	-6	1	7	126	116	-7	2	1	26	7*
-10	0	4	87	71	-4	1	1	340	369	4	1	4	73	83	-7	1	7	54	75	7	2	1	27	27*
-8	0	5	28	0*	5	1	1	312	304	-4	1	4	39	27	-8	1	7	116	116	-8	2	1	28	25*
0	0	6	371	368	-5	1	1	190	184	5	1	4	43	44	-9	1	7	25	70*	8	2	1	28	13*
1	0	6	302	297	6	1	1	198	198	-5	1	4	158	149	0	1	8	28	52*	-9	2	1	27	13*
-1	0	6	232	231	-6	1	1	195	208	6	1	4	90	88	1	1	8	62	72	9	2	1	27	18*
2	0	6	200	204	7	1	1	206	206	-6	1	4	77	61	-2	1	8	27	8*	-10	2	1	25	25*
-2	0	6	188	174	-7	1	1	173	161	-7	1	4	26	17*	2	1	8	28	19*	10	2	1	23	1*
3	0	6	214	209	8	1	1	95	81	7	1	4	28	12*	3	1	8	79	86	0	2	2	116	137
-3	0	6	[illegible]	[illegible]	-8	1	1	159	154	-8	1	4	27	27*	-4	1	8	28	13*	1	2	2	246	235

Table 3 (*cont.*)

-1	2	2	480	466
2	2	2	295	240
-2	2	2	459	461
3	2	2	272	270
-3	2	2	317	314
4	2	2	278	277
-4	2	2	397	396
5	2	2	283	282
-5	2	2	213	214
6	2	2	257	248
-6	2	2	134	168
7	2	2	64	57
-7	2	2	164	158
-8	2	2	28	21*
8	2	2	28	18*
9	2	2	68	46
-9	2	2	60	53
-10	2	2	49	88
0	2	3	56	52
1	2	3	50	41
-1	2	3	24	32
2	2	3	140	119
-2	2	3	75	79
3	2	3	41	42
-3	2	3	63	52
4	2	3	101	86
-4	2	3	96	95
5	2	3	62	85
-5	2	3	101	85
-6	2	3	25	23*
6	2	3	27	19*
-7	2	3	27	22*
7	2	3	26	9*
-8	2	3	28	23*
8	2	3	27	14*
-9	2	3	27	5*
9	2	3	24	4*
-10	2	3	25	7*
0	2	4	158	205
1	2	4	270	249
2	2	4	278	269
-2	2	4	239	241
3	2	4	221	235
-3	2	4	657	654
4	2	4	211	223
-4	2	4	322	323
5	2	4	181	178
-5	2	4	95	101
6	2	4	57	60
-6	2	4	157	154
7	2	4	46	41
-7	2	4	93	82
8	2	4	69	53
-8	2	4	86	72
-9	2	4	84	65
-10	2	4	42	47
0	2	5	69	66
1	2	5	52	34
-1	2	5	27	31
2	2	5	45	52
-2	2	5	54	65
3	2	5	76	90
-4	2	5	112	108
-5	2	5	40	43
6	2	5	36	40
-6	2	5	52	55
-7	2	5	28	45*
7	2	5	26	4*
-8	2	5	28	16*
-9	2	5	27	1*
-10	2	5	24	1*
0	2	6	248	249
1	2	6	179	177
-1	2	6	222	219
-2	2	6	258	213
3	2	6	106	104
-3	2	6	112	112
4	2	6	172	173
-4	2	6	213	209
5	2	6	84	104
-5	2	6	231	222
-6	2	6	68	58
-7	2	6	85	91
-8	2	6	112	106
-9	2	6	25	28*
0	2	7	27	18*
-1	2	7	27	5*
1	2	7	27	27*
-2	2	7	27	25*
2	2	7	28	41*
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-4	2	7	44	47
-5	2	7	28	27*
5	2	7	25	4*
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-7	2	7	27	3*
-8	2	7	26	22*
-9	2	7	24	20*
0	2	8	46	44
1	2	8	36	29
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2	2	8	106	112
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3	2	8	80	80
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4	2	8	48	58
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-5	2	8	85	78
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0	2	9	26	8*
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1	2	9	26	4*
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-3	2	10	76	73
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3	3	0	139	141
4	3	0	24	16*
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6	3	0	27	37*
7	3	0	23	23*
8	3	0	27	24*
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0	3	1	366	363
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2	3	1	296	275
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3	3	1	215	216
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-5	3	1	138	141
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-6	3	1	171	166
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-7	3	3	105	101
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0	3	4	24	25*
1	3	4	51	51
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2	3	4	63	60
3	3	4	92	91
4	3	4	146	172
-5	3	4	27	5*
5	3	4	28	20*
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-9	3	5	57	58
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1	3	6	27	9*
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3	3	6	73	73
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-4	3	6	75	82
5	3	6	32	37
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0	3	8	27	11*
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0	4	0	407	393
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4	4	0	140	122
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6	4	0	94	97
7	4	0	62	60
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0	4	1	25	7*
1	4	1	37	40
-1	4	1	47	54
2	4	1	38	39
-2	4	1	33	38
-3	4	1	79	91
4	4	1	48	52
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5	4	1	28	2*
6	4	1	43	57
7	4	1	37	49
-7	4	1	41	30
-8	4	1	25	27*
8	4	1	23	17*
0	4	2	197	195
1	4	2	372	295
-1	4	2	198	191
2	4	2	181	181
-2	4	2	198	188
3	4	2	142	139
-3	4	2	85	77
4	4	2	128	120
-4	4	2	137	133
-5	4	2	141	146
6	4	2	30	50
-6	4	2	85	98
7	4	2	92	109
-7	4	2	63	94
-8	4	2	70	73
0	4	3	26	3*
-1	4	3	26	16*
1	4	3	27	10*
-2	4	3	48	52
-3	4	3	45	53
-4	4	3	27	18*
4	4	3	28	22*
5	4	3	57	63
-5	4	3	96	89
-6	4	3	53	61
-7	4	3	27	11*
7	4	3	23	2*
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0	4	4	224	221
1	4	4	125	115
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2	4	4	127	138
-2	4	4	129	122
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6	4	4	60	65
-6	4	4	144	132
-7	4	4	75	86
-8	4	4	54	75
0	4	5	28	19*
1	4	5	33	35
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2	4	5	28	9*
3	4	5	47	55
-4	4	5	28	35*
4	4	5	26	21*
-5	4	5	27	11*
5	4	5	24	13*
-6	4	5	36	34
7	4	5	37	14
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-7	4	6	59	62
0	4	7	26	8*
1	4	7	32	33
-2	4	7	27	24*
2	4	7	24	29*
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-4	4	7	34	46
-5	4	7	25	9
-6	4	7	23	14
0	4	8	66	76
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-2	4	8	45	55
-3	4	8	49	46
-4	4	8	41	44
1	5	0	28	15*
2	5	0	28	6*
3	5	0	37	31
4	5	0	27	10*
5	5	0	26	15*
6	5	0	24	29*
0	5	1	64	61
1	5	1	127	120
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3	5	1	96	104
-3	5	1	134	123
4	5	1	77	79
-4	5	1	150	157
5	5	1	77	76
-5	5	1	82	78
6	5	1	41	35
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0	5	2	28	42*
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2	5	2	27	13
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3	5	2	31	31
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-5	5	2	44	48
-6	5	2	25	6*
0	5	3	161	171
1	5	3	95	95
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2	5	3	57	50
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3	5	3	93	94
-3	5	3	104	107
4	5	3	57	60
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5	5	3	53	61
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0	5	4	27	37*
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1	5	4	27	0*
-2	5	4	27	20*
2	5	4	26	17*
-3	5	4	34	31
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4	5	4	23	3*
-5	5	4	39	46
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0	5	5	40	44
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0	5	6	24	5*
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1	5	6	23	4*
-2	5	6	24	34*
-3	5	6	24	7*
0	6	0	25	4*
1	6	0	37	40
2	6	0	118	121
3	6	0	108	109
0	6	1	25	9*
-1	6	1	25	8*
1	6	1	24	14*
2	6	1	30	34
-3	6	1	23	1*
0	6	2	69	76
1	6	2	54	103
1	6	2	57	103
-1	6	2	40	49
-2	6	2	45	44
-1	6	3	26	40

C(4) and C(5) to which they are bonded. The observed and calculated structure factors are given in Table 3.

Discussion of the structure

The crystal structure is shown in a (010) projection in Fig. 1. The Cu^{2+} ion forms a square-planar arrangement with the four nitrogen atoms of a complex molecule (two nitrogen atoms from each ligand molecule). Two *H*-pyrrole-aldimine molecules are related to each other through the centre of symmetry lying at Cu^{2+}, so that the Cu^{2+} ion lies exactly on the plane made by N(1), N(2), N(1′), N(2′) (primed numbers are related to unprimed ones by the centre of symmetry at Cu^{2+}). The two unique Cu–N distances, *i.e.*, Cu–N(1) and Cu–N(2) are 1·97 and 1·95 Å respectively.

The bond lengths and angles are listed in Table 4 and shown in Fig. 1. The observed Cu–N bond lengths are in good agreement with the following reported structures: 1·97 and 1·98 Å in $[Cu(en)_2(H_2O)Br]Br$, 1·98 and 1·99 Å in $[Cu(en)_2(H_2O)Cl]Cl$ (Mazzi, 1953); 2·01 and 2·02 Å in $Cu(en)_2(NO_3)_2$ (Komiyama & Lingafelter, 1964); 1·97 and 2·04 Å in $K_2Cu(NH_2CH_2CONCH_2COO)_2 . 6H_2O$ (Sugihara, Ashida, Sasada & Kakudo, 1968); 1·99 and 2·02 Å in $Cu(en)_2(SCN)_2$ (Brown & Lingafelter, 1964).

Table 4. *Bond lengths and bond angles*

Estimated standard deviations are given in parentheses.

Lengths in Å ($\sigma \times 10^3$)		Angles in degrees (σ)	
Cu^{2+}–N(1)	1·972 (9)	N(2)–Cu^{2+}–N(1)	82·4 (0·3)
Cu^{2+}–N(2)	1·950 (8)	Cu^{2+}–N(1)–C(1)	113·3 (0·8)
N(1)–C(1)	1·328 (14)	N(1)–C(1)–C(2)	115·6 (1·0)
N(2)–C(2)	1·360 (12)	C(2)–N(2)–Cu^{2+}	112·3 (0·6)
N(2)–C(5)	1·353 (15)	C(5)–N(2)–C(2)	107·2 (0·8)
C(1)–C(2)	1·406 (16)	N(2)–C(2)–C(3)	111·4 (0·9)
C(2)–C(3)	1·401 (15)	C(2)–C(3)–C(4)	105·2 (0·9)
C(3)–C(4)	1·424 (19)	C(3)–C(4)–C(5)	106·0 (1·1)
C(4)–C(5)	1·421 (16)	C(4)–C(5)–N(2)	110·1 (1·0)
		C(1)–C(2)–N(2)	116·4 (0·9)

The N(1)–Cu–N(2) angle was 82·3° in the present structure. This compares with 83° in $K_2Cu(NH_2CH_2CONCH_2COO)_2 . 6H_2O$ (Sugihara *et al.*, 1968); 85° in $Cu(en)_2(SCN)_2$ (Brown & Lingafelter, 1964); 86° in $Cu(en)_2(BF_4)_2$ (Brown, Lee & Melsom, 1968); 89° in $[Cu(en)_2(H_2O)Cl]Cl$ and 90° in $[Cu(en_2)_2(H_2O)Br]Br$ (Mazzi, 1953).

In the present structure both carbon atoms of the ring formed by Cu^{2+}, N(1), N(2), C(1) and C(2) are above the N–Cu–N plane by 0·098 and 0·054 Å respectively. A similar case is that of $K_2Cu(NH_2CH_2CONH_2COO)_2 . 6H_2O$ (Sugihara *et al.*, 1968) in which both the carbon atoms of the ethylenediamine ring are above the N–Cu–N plane by 0·406 and 0·302 Å, but in all other similar structures mentioned above one carbon atom of the ethylenediamine ring is above and the other below the plane.

Bis-(*H*-pyrrole-2-aldimine)copper(II) molecules in the present structure are essentially planar, the maximum deviation of any atom from the least-squares plane being only 0·06 Å. The equation of the least-squares plane in orthogonal Ångström space passing through the atoms in the asymmetric unit is:

$$-0·4042\,x+0·5647\,y+0·7195\,z=0\,. \qquad (1)$$

The distances of the constituent atoms from the above plane are listed in Table 5.

Table 5. *Deviation of atoms from the best least-squares plane* [*equation*(1)]

	Deviation (Å)
Cu^{2+}	0·0
N(1)	0·018
N(2)	0·063
C(1)	0·010
C(2)	0·020
C(3)	−0·012
C(4)	−0·035
C(5)	0·015

The pyrrole ring in the present structure is planar with an average N–C bond length of 1·355 Å and an average C–C bond length of 1·415 Å. The average bond angle is 108°. Corresponding values in ibogaine (Arai, Coppola & Jeffrey, 1960) are 1·395, 1·430 Å and 108°; in Ni(II) 2,4-diacetyldeuteroporphyrin-*IX* dimethyl ester (Hamor, Caughey & Hoard, 1965) 1·38, 1·41 Å and 108°; in methoxyiron(III)-mesoporphyrin-*IX* dimethyl ester (Hoard, Hamor, & Hamor Caughey, 1965) 1·398, 1·426 Å and 108°; and in tetraphenyl porphyrin (Silvers & Tulinsky, 1967) 1·368, 1·410 Å and 108°.

Anisotropic thermal parameters are listed in Table 2. From these parameters, the vibration amplitudes along the three principal axes of the thermal ellipsoids and also the orientation of these axes have been calculated using the IBM 7044 program *ANTEMP* (Dwivedi, 1970). It has been shown by other authors (Srivastava & Lingafelter, 1966; Werner, 1964) that the temperature factors can compensate for errors in data due to absorption and dispersion; since these corrections have not been applied, perhaps the absolute magnitudes of the vibration amplitudes are of not much significance. Relative magnitudes of vibration amplitudes and theorientation of ellipsoids are shown in Fig. 3.

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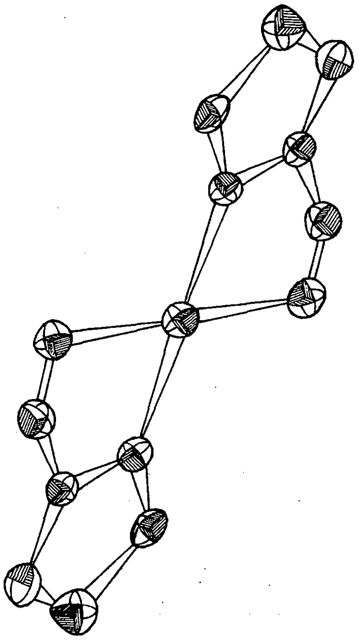

Fig. 3. *X–Z* projection of the crystal structure showing thermal ellipsoids.

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The Crystal Structure of α-Bromoacetophenone

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The crystal structure of α-bromoacetophenone has been determined using three-dimensional X-ray diffraction data. The compound crystallizes in the orthorthombic system with $a = 9{\cdot}74_3$, $b = 18{\cdot}93_5$, $c = 4{\cdot}22_2$ Å. Space group is $P2_12_12_1$, with four molecules in the unit cell. The structure has been refined by the least-squares method with individual anisotropic temperature factors for each atom to an R value of 0·107. In the crystal, the molecules are loosely separated by van der Waals distances all greater than 3 Å, the molecules themselves being arranged in two distinct layers, making an angle of ~122° with each other.

The spatial geometry of the acetophenone group and Br–C linkages in the group have not been reported before. The present structure analysis was attempted with this objective in view, as part of a larger programme in this department for determining the crystal structures of simple organic molecules.

The compound α-bromoacetophenone ($C_6H_5CO.CH_2.Br$) or phenacyl bromide crystallizes as colourless

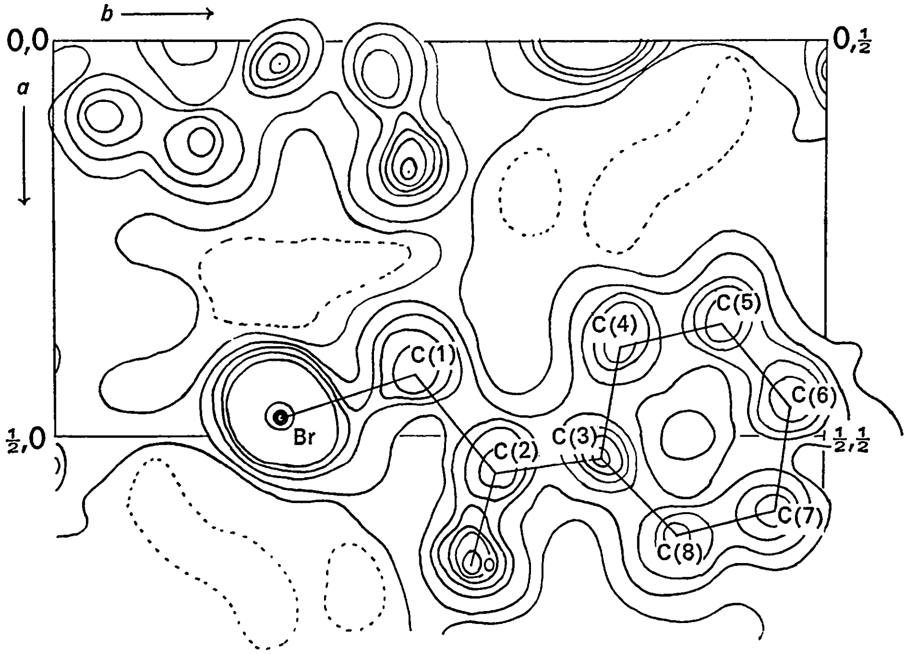

Fig. 1. Electron density projection looking down the [001] axis. Contours are at intervals of 1·0 e.Å^2.