

The Crystal and Molecular Structure of Dichlorobis(δ -*N*-hydro-L-ornithinato)copper(II) Dihydrate

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The structure of dichlorobis(δ -*N*-hydro-L-ornithinato)copper(II) dihydrate has been determined by counter methods are refined to $R = 0.050$ for 1920 unique reflexions by least squares. The monoclinic unit cell, space group $P2_1$, has $a = 5.218(3)$, $b = 15.636(2)$, $c = 12.058(4)$ Å, $\beta = 93.37(2)^\circ$, $Z = 2$. The Cu atom is in a tetragonally elongated octahedral environment. A carboxylic O atom and the α -amino N atom from each ligand coordinate to the Cu atom in a *trans* square-planar configuration (Cu–O 1.96, Cu–N 1.98 Å). The octahedral environment is completed by the two Cl ions [Cu–Cl 2.916(2), 2.895(2) Å]. The neutrality of the molecule is maintained by protonation of the δ -nitrogen atom of each ornithine ligand. A three-dimensional hydrogen-bond network, including the water molecules, links the molecules in the crystal.

Introduction

The structure of cupric di-L-ornithinate dichloride dihydrate (Guha & Saha, 1970) has been reported, in which the complex crystallizes in the monoclinic space group $P2_1/c$ with $Z = 2$. This implies that the molecules must possess a centre of symmetry and hence the configuration must be DL and not LL as stated. A closer perusal of this report shows that the space group $P2_1/c$ was adopted on the grounds that some ten $h0l$ reflexions with l odd were Renninger reflexions (Renninger, 1937). If these were not Renninger reflexions then the possible space groups would be $P2_1$ or $P2_1/m$, of which only the former could be correct for an LL configuration. We therefore undertook to prepare the complex and investigate the problem. Table 1 summarizes the crystal data and compares them with those of Guha & Saha (1970).

A precession photograph of the $h0l$ zone for our crystal with Mo $K\alpha$ radiation and an exposure time of 72 h showed 129 $h0l$ reflexions of observable intensity, of which 47 have $l = 2n + 1$. We therefore undertook a redetermination of the structure.

Experimental

The complex was prepared by adding a stoichiometric amount of CuCl_2 in aqueous solution to a dilute aqueous solution of L-ornithine hydrochloride. The resultant solution was diluted twenty fold with methanol and then 2-propanol was added dropwise until the complex was on the point of precipitation. Slow evaporation of the solution yielded royal-blue needles elongated along *a*.

Analysis: calculated for $\text{C}_{10}\text{H}_{24}\text{N}_4\text{O}_4\text{Cl}_2\text{Cu} \cdot 2\text{H}_2\text{O}$, $C = 27.6$, $H = 6.5$, $N = 12.9\%$; found $C = 27.3$, $H = 6.5$, $N = 12.7\%$.

Crystal data

$\text{C}_{10}\text{H}_{28}\text{N}_4\text{O}_6\text{Cl}_2\text{Cu}$, $M_r = 434.8$, monoclinic, $a = 5.218(3)$, $b = 15.636(2)$, $c = 12.058(4)$ Å, $\beta = 93.37(2)^\circ$, $V = 982.1$ Å³, $D_m = 1.48$ g cm⁻³ (by flotation), $Z = 2$, $D_c = 1.47$; $F(000) = 454$, $\mu(\text{Cu } K\alpha) = 41.9$ cm⁻¹. Systematic absences: $0k0$ if $k = 2n + 1$, space group $P2_1$ or $P2_1/m$. Accurate cell parameters were obtained from a least-squares fit to diffractometer data.

A small fragment $0.3 \times 0.3 \times 0.2$ mm was cut from a larger crystal and used for the data collection. The intensities were collected on a Siemens AED four-circle diffractometer with monochromatic Cu $K\alpha$ radiation. A $\theta/2\theta$ scanning technique with integrated intensities was employed. For strong reflexions the beam was attenuated with Ni foils. Each reflexion in the $h0-18l$ layers for $\sin \theta < 0.60$ was recorded. 1924 unique reflexions gave counts for which $I > 2\sigma(I)$ and

Table 1. Comparison of crystal data

	Guha & Saha (1970)	Present work
<i>a</i>	5.18 Å	5.218 Å
<i>b</i>	15.57	15.636
<i>c</i>	11.90	12.058
β	93.3°	93.37°
Space group	$P2_1/c$	$P2_1$ or $P2_1/m$
D_m	1.501 g cm ⁻³	1.48 g cm ⁻³
<i>Z</i>	2	2

these were used for the analysis. The intensities were corrected for Lorentz, polarization and absorption effects (Craig, 1969) but not for extinction.

Scattering factors were taken from *International Tables for X-ray Crystallography* (1962), the Cu and Cl atoms being corrected for anomalous dispersion. All calculations were carried out with programs written by FSS for use on a Univac 1106 computer.

Table 2. *Final atomic coordinates (fractional, $\times 10^4$ for heavy atoms, $\times 10^3$ for H) with estimated standard deviations in parentheses*

	x	y	z
Cu	73 (2)	0	2448 (1)
Cl(1)	2671 (3)	-1522 (1)	1680 (1)
Cl(2)	-2523 (3)	1499 (1)	3248 (1)
O(1)	2072 (15)	-3423 (5)	2525 (5)
O(2)	-2287 (11)	3460 (4)	2565 (4)
O(11)	-2098 (7)	51 (4)	1081 (3)
O(12)	-2307 (10)	536 (4)	-640 (4)
O(21)	2155 (7)	-27 (4)	3863 (3)
O(22)	1590 (9)	-446 (4)	5600 (4)
N(11)	2517 (9)	674 (4)	1632 (4)
N(12)	4966 (9)	3929 (3)	641 (4)
N(21)	-2309 (9)	-755 (4)	3226 (4)
N(22)	-4691 (10)	-3789 (4)	4451 (4)
C(11)	-1193 (10)	481 (4)	299 (4)
C(12)	1311 (10)	960 (4)	548 (4)
C(13)	802 (11)	1922 (4)	539 (5)
C(14)	3253 (11)	2452 (4)	601 (5)
C(15)	2608 (12)	3388 (4)	632 (6)
C(21)	1222 (10)	-496 (4)	4593 (5)
C(22)	-791 (10)	-1155 (4)	4182 (4)
C(23)	-2453 (10)	-1499 (4)	5083 (5)
C(24)	-4239 (11)	-2231 (4)	4704 (5)
C(25)	-2844 (12)	-3072 (5)	4587 (6)
H(111)	300	110	205
H(112)	350	50	150
H(12)	200	75	50
H(131)	5	197	-13
H(132)	-10	192	108
H(141)	345	230	05
H(142)	475	243	55
H(151)	130	312	0
H(152)	160	360	115
H(121)	600	392	125
H(122)	590	382	35
H(123)	425	430	27
H(211)	-300	-117	275
H(212)	-325	-50	363
H(22)	0	-150	390
H(231)	-140	-165	575
H(232)	-350	-117	525
H(241)	-525	-233	387
H(242)	-525	-217	510
H(251)	-160	-292	405
H(252)	-150	-300	525
H(221)	-580	-383	480
H(222)	-570	-383	375
H(223)	-440	-448	437
H(1)	226	-282	226
H(2)	216	-379	185
H(3)	-236	284	278
H(4)	-175	380	325

Structure determination

For the space group $P2_1/m$ to be correct the L-ornithine molecule must undergo racemization, with the subsequent DL configuration. Thus the space group is $P2_1$, which imposes no symmetry constraints on the molecule and is confirmed by the successful refinement of the structure.

The positions of the Cu atom, Cl atoms, the atoms forming the two five-membered rings and the remaining carboxyl O atoms were obtained from a Patterson synthesis. The complete solution of the structure for the nonhydrogen atoms was obtained from a series of Fourier syntheses, each phased by an increasing number of atoms.

Refinement of the structure was by full-matrix least squares in which the function minimized was $\Sigma w\Delta^2$. The weight for each reflexion, w , was initially unity and in the final stages of refinement was given by $w = (15.0 + 0.1|F_o| + 0.1|F_o|^2)^{-1}$; for this latter weighting scheme the average values for $w\Delta^2$ for ranges of increasing $|F_o|$ were nearly constant. Reflexions for which $|F_o| < 0.3|F_o|$ were omitted from the least-squares analysis.

After four cycles of refinement, in which positional and individual isotropic thermal parameters for each atom were varied, a difference synthesis was calculated. This map showed no unusual features and yielded the positions of all the H atoms. These atoms were included in subsequent calculations with a thermal parameter of $B = 5.0 \text{ \AA}^2$, but were not refined.

Final refinement was carried out with anisotropic thermal parameters for all nonhydrogen atoms, and was terminated when the maximum shift in any parameter was less than 0.05σ . 1920 reflexions were included in the final cycle. The final value for R , based on all 1924 reflexions, was 0.050 and for $R' [= (\Sigma w\Delta^2 / \Sigma w|F_o|^2)^{1/2}]$ was 0.071.

The final atomic parameters are given in Tables 2 and 3.*

Discussion

The bond lengths and angles with their estimated standard deviations derived directly from the least-squares inverse matrix are given in Table 4. Fig. 1 shows a perspective drawing of the molecule and the labelling of the atoms, and Fig. 2 the packing of the molecules (Johnson, 1965). A three-dimensional hydrogen-bonding network, including the water molecules, is seen to

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32080 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

link the molecules; the donor–acceptor distances for these bonds are given in Table 5.

A carboxyl O atom and the α -amino N atom from each ligand coordinate to the Cu atom in a *trans* square-planar configuration, the Cu–O and Cu–N distances being 1.96 and 1.98 Å respectively. These values are in good agreement with those observed in similar complexes (Freeman, 1967). The octahedral environment about Cu is completed by the two Cl ions.

The two Cu–Cl distances [2.916(2) and 2.895(2) Å] are not equivalent, though nearly so, and are typical of those found in Cu^{II} complexes with tetragonally elongated octahedral environments (Hathaway & Billing, 1970). The directions of these bonds are almost collinear with the normal to the coordination square plane containing the Cu atom [plane 1, Table 6].

The location of the H atoms indicates that the neutrality of the molecule is maintained by the protonation

Table 3. *Final thermal parameters* ($\times 10^4$) *with estimated standard deviations in parentheses*

For all hydrogen atoms $B = 5.0 \text{ \AA}^2$. The anisotropic thermal parameters are in the form:
 $\exp[-(h^2 b_{11} + k^2 b_{22} + l^2 b_{33} + 2hkb_{12} + 2hkl_{13} + 2klb_{23})]$.

	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
Cu	167 (3)	30 (1)	25 (1)	–29 (1)	–17 (1)	11 (1)
Cl(1)	229 (5)	23 (1)	33 (1)	–1 (2)	–1 (2)	–4 (1)
Cl(2)	226 (5)	24 (1)	38 (1)	6 (2)	11 (2)	–6 (1)
O(1)	751 (37)	37 (3)	61 (5)	3 (9)	–15 (10)	–4 (3)
O(2)	511 (25)	37 (2)	34 (3)	22 (7)	–63 (7)	–5 (3)
O(11)	172 (12)	22 (1)	28 (2)	–21 (5)	–4 (4)	2 (2)
O(12)	415 (22)	28 (2)	34 (3)	–47 (6)	–62 (7)	12 (2)
O(21)	168 (13)	26 (2)	33 (2)	–19 (5)	–15 (5)	6 (2)
O(22)	321 (18)	31 (2)	24 (3)	–28 (6)	–28 (5)	3 (2)
N(11)	158 (17)	20 (2)	31 (3)	–7 (4)	–11 (6)	4 (2)
N(12)	169 (17)	14 (2)	45 (4)	–9 (5)	–12 (6)	4 (2)
N(21)	166 (16)	24 (2)	27 (3)	–11 (5)	–16 (6)	6 (2)
N(22)	261 (20)	22 (2)	40 (4)	6 (5)	–5 (7)	2 (2)
C(11)	189 (19)	12 (2)	28 (4)	–7 (5)	–13 (7)	0 (2)
C(12)	149 (18)	15 (2)	24 (3)	0 (5)	–4 (6)	0 (2)
C(13)	175 (18)	13 (2)	46 (4)	–7 (5)	–12 (7)	3 (2)
C(14)	153 (19)	16 (2)	48 (4)	–7 (5)	0 (7)	3 (3)
C(15)	174 (19)	15 (2)	64 (5)	–11 (6)	–16 (8)	3 (3)
C(21)	140 (17)	20 (2)	30 (4)	6 (6)	–2 (6)	1 (3)
C(22)	165 (18)	17 (2)	20 (3)	5 (5)	–4 (6)	2 (2)
C(23)	202 (19)	24 (2)	27 (3)	–18 (6)	10 (7)	4 (2)
C(24)	159 (17)	19 (2)	40 (4)	–5 (5)	4 (7)	3 (2)
C(25)	183 (20)	24 (3)	64 (5)	3 (7)	3 (8)	0 (3)

Table 4. *Bond lengths* (Å) *and angles* (°) *with estimated standard deviations in parentheses*

Cu–O(11)	1.946 (4)	C(13)–C(14)	1.522 (8)	C(21)–O(22)	1.253 (7)
Cu–N(11)	1.963 (5)	C(14)–C(15)	1.502 (8)	C(22)–N(21)	1.497 (7)
Cu–Cl(1)	2.916 (2)	C(15)–N(12)	1.493 (7)	C(21)–C(22)	1.533 (8)
C(11)–O(11)	1.272 (7)	Cu–O(21)	1.969 (4)	C(22)–C(23)	1.527 (7)
C(11)–O(12)	1.244 (7)	Cu–N(21)	1.989 (5)	C(23)–C(24)	1.529 (8)
C(12)–N(11)	1.486 (7)	Cu–Cl(2)	2.895 (2)	C(24)–C(25)	1.513 (9)
C(11)–C(12)	1.521 (7)	C(21)–O(21)	1.264 (8)	C(25)–N(22)	1.481 (9)
C(12)–C(13)	1.528 (8)				
O(11)–Cu–N(11)	85.3 (11)	C(11)–C(12)–C(13)	109.7 (28)	N(11)–Cu–N(21)	176.1 (15)
O(11)–Cu–Cl(1)	91.3 (9)	C(11)–C(12)–N(11)	109.9 (26)	Cu–O(21)–C(21)	113.6 (21)
O(11)–Cu–Cl(2)	89.4 (9)	C(13)–C(12)–N(11)	111.5 (20)	Cu–N(21)–C(22)	107.1 (20)
N(11)–Cu–Cl(1)	87.2 (9)	C(12)–C(13)–C(14)	113.0 (28)	O(21)–C(21)–O(22)	122.1 (32)
N(11)–Cu–Cl(2)	93.7 (10)	C(13)–C(14)–C(15)	110.0 (27)	O(21)–C(21)–C(22)	116.7 (30)
O(11)–Cu–N(21)	94.3 (11)	C(14)–C(15)–N(12)	111.5 (28)	O(22)–C(21)–C(22)	121.2 (28)
O(11)–Cu–O(21)	177.6 (12)	O(21)–Cu–N(21)	84.6 (11)	C(21)–C(22)–C(23)	114.4 (27)
Cl(1)–Cu–Cl(2)	178.9 (3)	O(21)–Cu–Cl(2)	88.5 (10)	C(21)–C(22)–N(21)	106.8 (27)
Cu–O(11)–C(11)	115.3 (20)	O(21)–Cu–Cl(1)	90.8 (10)	C(23)–C(22)–N(21)	113.5 (26)
Cu–N(11)–C(12)	110.4 (20)	N(21)–Cu–Cl(2)	90.2 (10)	C(22)–C(23)–C(24)	114.4 (29)
O(11)–C(11)–O(12)	122.4 (30)	N(21)–Cu–Cl(1)	88.9 (10)	C(23)–C(24)–C(25)	113.0 (29)
O(11)–C(11)–C(12)	117.8 (26)	O(21)–Cu–N(11)	96.0 (11)	C(24)–C(25)–N(22)	110.8 (28)
O(12)–C(11)–C(12)	119.8 (29)				

of the δ -nitrogen atom of each ornithine ligand. The two L-ornithine ligands are not equivalent and in particular show differences in the five-membered coordination rings. One may be considered as planar

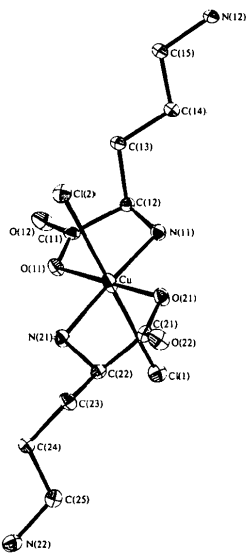


Fig. 1. Perspective drawing of the molecule and the labelling of the atoms. Thermal ellipsoids are scaled to include 35% probability.

[plane 2, Table 6], whereas the other shows significant distortion from planarity [plane 3, Table 6]. This distortion is similar to that observed in the structures of the L-asparagine complexes of Cu^{II} (Stephens, Vagg & Williams, 1975) and Zn^{II} (Stephens, Vagg & Williams, 1977). The reason for this distortion in the present structure is not readily evident.

As shown in Table 1, the crystal data for the present determination, which has an LL configuration, and

Table 5. *Proposed hydrogen bonds* (\AA): *donor-acceptor distances are given, with estimated standard deviations in parentheses*

	Acceptor at	
N(21)—H(211) \cdots Cl(1)	$(x - 1, y, z)$	3.347 (5)
N(12)—H(122) \cdots Cl(1)	$(1 - x, \frac{1}{2} + y, -z)$	3.202 (5)
O(1)—H(1) \cdots Cl(1)		3.164 (7)
N(11)—H(111) \cdots Cl(2)	$(1 + x, y, z)$	3.398 (5)
N(22)—H(221) \cdots Cl(2)	$(-1 - x, y - \frac{1}{2}, 1 - z)$	3.237 (6)
O(2)—H(3) \cdots Cl(2)		3.186 (7)
N(22)—H(222) \cdots O(1)	$(x - 1, y, z)$	2.848 (10)
N(12)—H(121) \cdots O(2)	$(1 + x, y, z)$	2.754 (8)
N(11)—H(112) \cdots O(11)	$(1 + x, y, z)$	3.083 (6)
N(12)—H(123) \cdots O(12)	$(-x, \frac{1}{2} + y, -z)$	2.869 (7)
O(1)—H(2) \cdots O(12)	$(-x, y - \frac{1}{2}, -z)$	2.806 (9)
N(21)—H(212) \cdots O(21)	$(x - 1, y, z)$	3.239 (6)
N(22)—H(223) \cdots O(22)	$(-x, y - \frac{1}{2}, 1 - z)$	2.963 (8)
O(2)—H(4) \cdots O(22)	$(-x, \frac{1}{2} + y, 1 - z)$	2.794 (8)

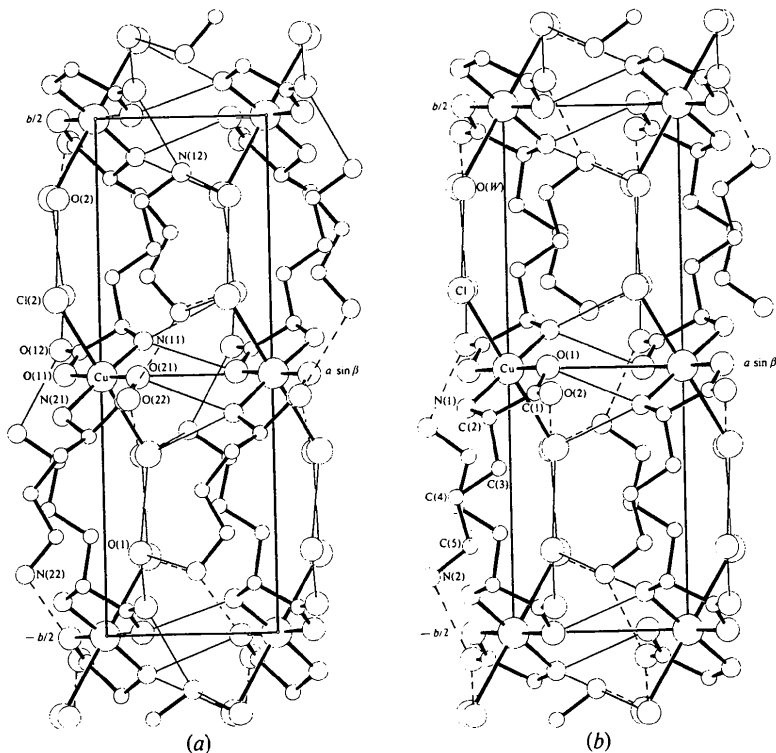


Fig. 2. Molecular packing in the crystal. Proposed hydrogen bonds are represented by thin lines; broken lines indicate that one of the atoms involved is from a molecule a cell away in c . (a) This work. (b) Taken from Guha & Saha (1970); for comparative purposes the signs of the x and z coordinates have been changed. The molecules represented are those with the Cu atoms at $z = 0$ or $-\frac{1}{2}$.

those of Guha & Saha (1970), for a DL configuration, are similar. Further it is evident from Fig. 2, in which the corresponding packing diagram for the DL structure is shown, that the two crystal structures are closely related. Therefore, we decided to investigate the possibility that the two sets of observed structure factors arose from the same structure, namely that with the LL configuration. In order to do this the space group for the earlier determination would have to be $P2_1$, and not $P2_1/c$; the argument concerning Renninger reflexions is therefore no longer a necessity.

An analysis of the two sets of data is given in Table 7; the R values for common reflexions in the various layers of data range from 0.217 to 0.239. This suggests that the two data sets do not arise from the same crystal structure. However, we attempted to obtain a solution for an LL configuration from the data of Guha & Saha (1970). The result of this analysis showed that

only a DL configuration is consistent with the data and that the resulting structure is similar to that found by those workers in the centrosymmetric space group $P2_1/c$.

From our studies we would conclude that the structure determined by Guha & Saha (1970) is that of the DL configuration. The preparative method used by these workers would be of interest since racemization has occurred during the preparation or recrystallization of the complex. With our preparative method, detailed earlier, we have not detected any racemized complex.

We thank Mr D. C. Craig of the School of Chemistry, University of New South Wales for his assistance with the collection of intensities and that School for the use of their diffractometer.

Table 6. Equations of the least-squares plane of best fit in the form $lX' + mY' + nZ' - p = 0$, where the orthogonalized coordinates X' , Y' , Z' are derived from the atomic coordinates X , Y , Z by: $X' = X \sin \beta$, $Y' = Y$, $Z' = X \cos \beta + Z$

Deviations (\AA) of the most relevant atoms from the planes are given in square brackets.

	l	m	n	p
Plane 1: Cu, O(11), O(21), N(11), N(21) [Cu 0.01, O(11) 0.05, O(21) 0.05, N(11) -0.06, N(21) -0.06]	-0.4517	0.8244	0.3412	0.9764
Plane 2: Cu, O(11), C(11), C(12), N(11) [Cu 0.04, O(11) -0.01, C(11) -0.04, C(12) 0.08, N(11) -0.07]	-0.4401	0.8116	0.3841	1.0765
Plane 3: Cu, O(21), C(21), C(22), N(21) [Cu -0.09, O(21) -0.01, C(21) 0.13, C(22) -0.22, N(21) 0.19]	-0.5491	0.7151	0.4325	1.3449
Plane 4: C(11), C(12), O(11), O(12) [C(11) -0.009, C(12) 0.003, O(11) 0.003, O(12) 0.003]	-0.5065	0.8006	0.3203	1.0518
Plane 5: C(21), C(22), O(21), O(22) [C(21) -0.010, C(22) 0.003, O(21), 0.004, O(22) 0.004]	0.7196	-0.6726	-0.1729	0.0388

Table 7. Comparison of the observed structure factors for the present structure with those of Guha & Saha (1970)

Data are presented for common reflexions in the layers collected by Guha & Saha.

Layer	Number	$\Sigma F $	$\Sigma F_{G\&S} $	K	$\Sigma F - K \times F_{G\&S} $	R^*
0kl	204	4088.93	3642.80	1.122	977.14	0.239
1kl	355	7991.69	6963.89	1.148	1736.50	0.217
2kl	300	6324.62	5309.70	1.191	1412.03	0.223
3kl	288	4804.58	3951.60	1.216	1093.72	0.228
4kl	240	3280.02	2568.50	1.277	727.89	0.222

$$* R = \Sigma|F - K \times F_{G\&S}| / \Sigma|F|.$$

References

- CRAIG, D. C. (1969). *AEDRED*, a data reduction program for the Siemens diffractometer. School of Chemistry, Univ. of New South Wales, Sydney, Australia.
- FREEMAN, H. C. (1967). *Advanc. Protein Chem.* **22**, 257–424.
- GUHA, S. & SAHA, N. N. (1970). *Acta Cryst.* **B26**, 2073–2079.
- HATHAWAY, B. J. & BILLING, D. E. (1970). *Chem. Rev.* **5**, 143–207.
- International Tables for X-ray Crystallography* (1962). Vol. III, pp. 202–204, 215. Birmingham: Kynoch Press.
- JOHNSON, C. K. (1965). *ORTEP*. Oak Ridge National Laboratory Report ORNL-3794, revised 1971.
- RENNINGER, M. Z. (1937). *Z. Kristallogr.* **97**, 107–121.
- STEPHENS, F. S., VAGG, R. S. & WILLIAMS, P. A. (1975). *Acta Cryst.* **B31**, 841–845.
- STEPHENS, F. S., VAGG, R. S. & WILLIAMS, P. A. (1977). *Acta Cryst.* **B33**, 433–437.

Acta Cryst. (1977). **B33**, 443–446

The Crystal Structure of an Unusual Bicyclic Phosphazene $N_4P_4(NMe_2)_5(NHEt)(NEt)$

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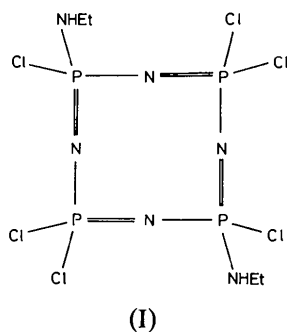
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The structure of the title compound has been determined by the heavy-atom method and refined by least squares to $R = 0.008$. $a = 10.81$, $b = 17.52$, $c = 28.48$ Å, $\beta = 94.8^\circ$, space group $C2/c$, 921 reflexions. The molecule is a bicyclic phosphazene with an ethylamine group bridging two P atoms. The exocyclic P–N length appears to be related to the deviation from coplanarity of the three atoms bonded to the N atom.

In an investigation into the reaction of the phosphazene (I) with an excess of Me_2NH an unusual crystalline product was obtained (Keat *et al.*, 1976). The structural formula of this product has been determined by X-ray crystallography (Cameron *et al.*, 1975).



Experimental

The crystals were provided by S. Krishnamurthy and A. C. Sau, Indian Institute of Science, Bangalore.

Crystal data

$C_{14}H_{41}N_{11}P_4$, $M_r = 463.3$, $a = 10.81$ (1), $b = 17.52$ (1), $c = 28.48$ (2) Å, $\beta = 94.8$ (2)°, $D_m = 1.21$, $Z = 8$, $D_c = 1.205$ g cm⁻³. Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å, $\mu = 26.6$ cm⁻¹; systematic extinctions: hkl $h + k = 2n + 1$, $h0l$ $l = 2n + 1$, space group $C2/c$.

The intensities of 921 reflexions were estimated from equi-inclination Weissenberg photographs (0–10, k , l) by the Harwell film-scanning service.

The positions of four P atoms were determined from an unsharpened Patterson function and the locations of the remaining non-hydrogen atoms found in the subsequent F_o synthesis.

