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(–)-β-Isosparteine Copper(II) Chloride*

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Abstract. $C_{15}H_{26}N_2.CuCl_2$, orthorhombic, space group $P2_12_12_1$, $a=11{\cdot}397$ (16), $b=11{\cdot}560$ (10), and $c=12{\cdot}639$ (17) Å, $Z=4$, $M=368{\cdot}8$, $D_c=1{\cdot}47$, $D_m=1{\cdot}50$ g cm^{-3}. All four of the six-membered rings are found to be in the chair conformation. The copper(II) coordination is distorted tetrahedral with Cu–Cl distances of 2·255 (4) Å and Cu–N distances of 1·994 (6) Å.

Introduction. The copper(II) chloride complex of (–)-β-isoparteine was synthesized by Boschmann, Weinstock & Carmack (1974) and recrystallized from methanol. A yellow-green crystal of approximate dimensions 0·2 × 0·26 × 0·2 mm was selected for study. Aside from preliminary precession work all data were collected on a Picker FACS-1 diffractometer using graphite-monochromated Mo $K\alpha$ radiation. Systematic absences of $h00$ for $h=2n+1$, $0k0$ for $k=2n+1$ and $00l$ for $l=2n+1$ identified the space group as $P2_12_12_1$. Unit-cell dimensions were refined by a least-squares fit using the orientation angles of 12 carefully aligned reflections [λMo $K\alpha=0{\cdot}71069$ Å]. Intensities were collected in the range $1\leq 2\theta\leq 55°$, using the θ–2θ scan mode with a scan rate of 2° min^{-1} and 20 s background counts at both ends of the scan range of 2·5° + dispersion. Three reference reflections measured periodically showed no systematic changes. The data were corrected for Lorentz and polarization effects. Correction for absorption ($\mu=17{\cdot}0$ cm^{-1}) was not carried out as the crystal was lost before its dimensions could be accurately measured. Of the 2177 independent reflections (hkl) obtained 1583 were considered to be observed by the criterion $|F_o|^2\geq\sigma(|F_o|^2)$.

The structure was solved by standard Patterson and Fourier methods. The refinement was carried out by the full-matrix least-squares program of Busing, Martin & Levy (1962) minimizing $\sum w(|F_o|^2-|F_c|^2)^2$ with $w=1/\sigma^2(F_o^2)$. The positions of 20 of the 26 hydrogen atoms were determined from a difference map; the remaining 6 were not well defined. All 26 hydrogen atoms were subsequently placed and kept in calculated positions (C–H = 1·00 Å) with an isotropic thermal parameter $B=4$. The non-hydrogen atoms were refined anisotropically. The final R value ($R=\sum||F_o|-|F_c||/\sum|F_o|$) for 1583 reflections was 0·068, the R value for all data was 0·101. The corresponding values for the weighted R_{wF2} ($R_{wF2}=[\sum w(|F_o|^2-|F_c|^2)^2/\sum w|F_o|^4]^{1/2}$ were 0·090 and 0·097 respectively. Atomic scattering factors for Cu, Cl, C and N were obtained from *International Tables for X-ray Crystallography* (1968); for hydrogen, those of Stewart, Davidson & Simpson (1965) were used. Anomalous dispersion corrections for Cu and Cl were obtained from Cromer & Liberman (1970). The final positional and thermal parameters are given in Table 1.†

Discussion. The natural alkaloid sparteine, $C_{15}H_{26}N_2$, has been used medicinally as a muscle stimulant. The

† A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30774 (23 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England. Supplemental data are also available, in microfiche form only, for $2.00, from the Chemistry Department Library, Indiana University, Bloomington, Ind. 47401, U.S.A. Request Molecular Structure Center Report No. 7403.

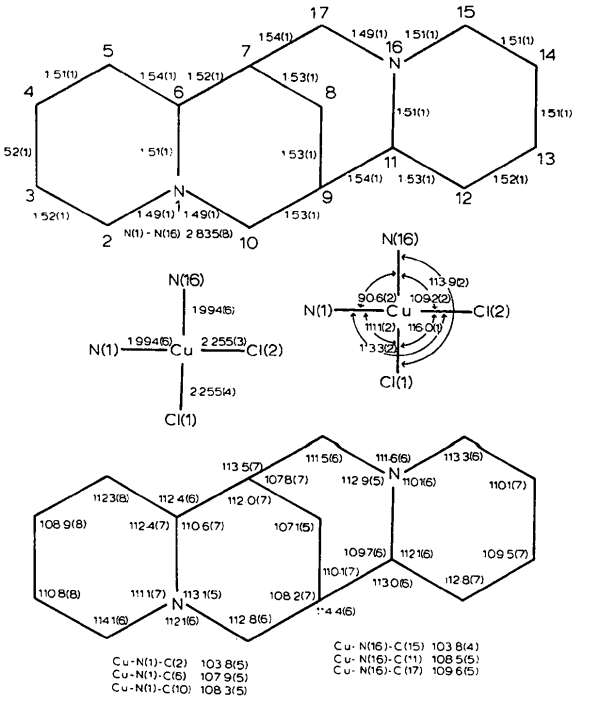

Fig. 1. Intermolecular bond distances and angles for β-isosparteine $CuCl_2$. Estimated standard deviations of the least significant digits are in parentheses.

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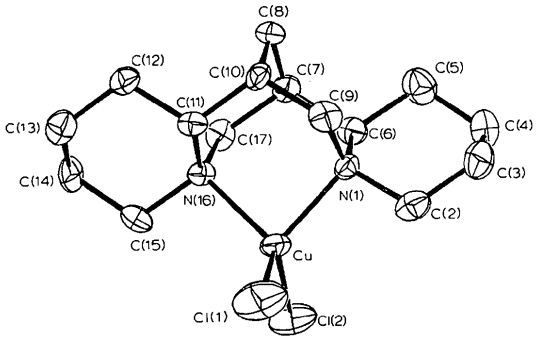

Fig. 2. Conformation and atomic numbering scheme of $C_{15}H_{26}N_2.CuCl_2$. The thermal ellipsoids are drawn at the 50% probability level (Johnson, 1965). The view is approximately perpendicular to the *yz* plane. Hydrogen atoms are omitted.

Table 1. *Final atomic parameters*

(*a*) Positional parameters ($\times 10^4$)

	x	*y*	*z*
Cu	246 (1)	679 (1)	1387 (1)
Cl(1)	1992 (2)	947 (3)	2190 (2)
Cl(2)	−1308 (2)	428 (2)	2467 (2)
N(1)	360 (5)	−517 (6)	254 (4)
C(2)	651 (8)	−1596 (8)	841 (6)
C(3)	535 (8)	−2692 (8)	189 (7)
C(4)	−691 (9)	−2796 (8)	−272 (8)
C(5)	−952 (8)	−1731 (8)	−920 (7)
C(6)	−821 (7)	−609 (9)	−270 (6)
C(7)	−1076 (7)	467 (7)	−922 (6)
C(8)	−108 (8)	711 (8)	−1730 (5)
C(9)	1022 (7)	925 (7)	−1108 (6)
C(10)	1314 (7)	−171 (8)	−489 (7)
C(11)	896 (7)	2016 (7)	−422 (6)
C(12)	826 (7)	3129 (7)	−1075 (6)
C(13)	658 (7)	4201 (9)	−394 (6)
C(14)	−415 (7)	4049 (7)	296 (6)
C(15)	−296 (8)	2968 (7)	961 (6)
N(16)	−139 (5)	1885 (5)	313 (4)
C(17)	−1241 (7)	1561 (7)	−251 (7)

(*b*) Anisotropic thermal parameters ($\times 10^4$)

The anisotropic thermal parameters are in the form $\exp[-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23})]$.

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Cu	54 (1)	56 (1)	23 (0)	4 (1)	−4 (1)	3 (1)
Cl(1)	89 (3)	172 (4)	65 (2)	−16 (3)	−44 (2)	18 (2)
Cl(2)	87 (2)	130 (3)	43 (1)	−6 (2)	25 (2)	14 (2)
N(1)	43 (5)	34 (6)	29 (4)	1 (1)	−3 (4)	5 (4)
C(2)	88 (10)	71 (9)	41 (6)	8 (8)	−7 (7)	10 (7)
C(3)	81 (11)	52 (9)	67 (8)	22 (8)	−8 (7)	6 (7)
C(4)	98 (11)	45 (9)	69 (8)	−7 (8)	−1 (8)	−5 (7)
C(5)	71 (9)	66 (10)	60 (7)	−11 (8)	−14 (7)	−13 (7)
C(6)	44 (7)	49 (8)	34 (5)	−11 (7)	3 (5)	−2 (7)
C(7)	48 (8)	46 (9)	33 (5)	−5 (7)	−21 (5)	2 (6)
C(8)	103 (10)	52 (7)	22 (4)	12 (9)	2 (5)	−4 (6)
C(9)	60 (8)	33 (8)	35 (6)	5 (6)	17 (5)	7 (5)
C(10)	33 (7)	65 (9)	47 (6)	4 (7)	21 (6)	−4 (6)
C(11)	34 (7)	52 (8)	27 (5)	−9 (6)	1 (5)	−1 (6)
C(12)	52 (7)	54 (8)	34 (6)	−5 (6)	3 (5)	7 (5)
C(13)	64 (8)	54 (8)	54 (6)	−4 (8)	−2 (6)	6 (7)
C(14)	52 (8)	34 (8)	58 (6)	18 (6)	−4 (6)	−4 (6)
C(15)	61 (7)	59 (8)	35 (5)	6 (8)	10 (6)	−15 (5)
N(16)	31 (6)	46 (6)	25 (4)	14 (5)	−2 (4)	1 (4)
C(17)	25 (6)	49 (8)	45 (6)	8 (6)	−3 (6)	3 (6)

possibility that the biological actions depend upon interactions with calcium ions has prompted investigations on the complexes of sparteine. The reactivity and properties of the copper(II) derivatives of sparteine and its two diastereoisomers are being studied by Boschmann, Weinstock & Carmack (1974), who suggested this structure determination to us.

The interatomic distances and angles for non-hydrogen atoms are shown in Fig. 1. The conformation of the molecule is shown in Fig. 2. No unusual bond distances or angles were found. The distances also agree well with those found in 7-hydroxy-β-isosparteine (Pinkerton & Steinrauf, 1967). All four of the six-membered rings are in the chair conformation.

The molecule posesses a nearly perfect twofold axis of rotation along a line through C(8) and Cu. All bonds related by the presumed twofold axis agree to within 0·02 Å and the corresponding angles agree to within 2°. As another measure of agreement, the midpoints of lines joining atoms related by the presumed axis should of course lie on the twofold axis and a least-squares fit of all such midpoints to a straight line gave a standard deviation of 0·029 Å.

Intermolecular distances less than 3·80 Å are given in Table 2. The molecules are oriented in the unit cell with the long direction [C(4)–C(13)] approximately along the *y* axis and with Cu–C(8) approximately along *z*. The contacts appear to be usual van der Waals distances.

Table 2. *Intermolecular distances between atoms less than* 3·80 Å *apart* (*excluding hydrogen atoms*)

Cl(1)···C(10)	$\frac{1}{2}-x$, $-y$, $\frac{1}{2}+z$	3·63 Å
Cl(2)···C(7)	$-\frac{1}{2}-x$, $-y$, $\frac{1}{2}+z$	3·76
Cl(2)···C(3)	$-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$	3·78
Cl(2)···C(14)	$-x$, $-\frac{1}{2}+y$, $\frac{1}{2}-z$	3·79
C(3)····C(13)	x, $-1+y$, z	3·67
C(4)····C(14)	x, $-1+y$, z	3·73
C(11)···C(17)	$\frac{1}{2}+x$, $\frac{1}{2}-y$, $-z$	3·75
C(12)···C(17)	$\frac{1}{2}+x$, $\frac{1}{2}-y$, $-z$	3·76
C(13)···C(17)	$\frac{1}{2}+x$, $\frac{1}{2}-y$, $-z$	3·73

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