I. They also discuss possible factors affecting secondary Sn—halo bonds.

This compound was prepared under the auspices of the late Professor J. J. Zuckerman, formerly of the Department of Chemistry, University of Oklahoma, Norman, OK 73019, USA.

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Structure of (3,5,7,13,15,17-Hexaethyl-2,8,12,18-tetramethylporphinato)copper(II)

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Abstract. [Cu(C$_{36}$H$_{44}$N$_4$)], $M_r = 596.32$, monoclinic, $C2/c$, $a = 27.423$ (4), $b = 14.147$ (3), $c = 16.718$ (3) Å, $\beta = 107.60$ (1)°, $V = 6183$ (2) Å^3, $Z = 8$, $D_x = 1.281$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, μ(Mo $K\alpha$) = 0.768 mm^{-1}, $F(000) = 2536$, $T = 295$ K, $R = 0.0363$ for 5352 reflections [$|F_o| > 3\sigma(F_o)$]. The Cu^{II} ion is surrounded by four pyrrole N atoms of the porphinato ligand and the coordination plane defined by these atoms is virtually planar (to within 0.05 Å). The planar structure of the porphinato core is strongly distorted into a saddle-like form due to the steric hindrance between the *meso*-ethyl and β-pyrrolic ethyl substituents, the *meso*-C atoms being displaced from the coordination plane by 0.574 (3)–0.660 (3) Å.

Experimental. The compound was prepared by treatment of the free base porphyrin (Maruyama, Nagata & Osuka, 1988) with copper(II) acetate in a methanol–chloroform solution. Purple prismatic crystals were grown by the slow evaporation of a dichloromethane–hexane solution. A crystal with dimensions 0.54 × 0.62 × 0.51 mm was used for the X-ray diffractometry. Rigaku AFC-5 diffractometer operating at 50 kV, 20 mA, graphite-monochromated Mo $K\alpha$ radiation. Accurate cell parameters by least-squares fit using 41 reflections with 25

$< 2\theta < 30°$. Intensity data collected by θ–2θ scan, scan rate 3° min^{-1}, scan range $(1.3 + 0.5\tan\theta)°$, $2\theta_{max} = 55°$, h -38 to 38, k 0 to 19, l -23 to 0. Three standard reflections monitored every 50 reflections showed no crystal movement or decay. Intensity corrected by numerical absorption method based on Gaussian integration, transmission-factor range = 0.642–0.696. 7675 reflections measured, 5352 reflections with $|F_o| > 3\sigma(F_o)$ observed. The structure was solved by conventional heavy-atom method, refined on F by block-diagonal least squares. All the non-H atoms refined anisotropically. H atoms were located by difference synthesis and refined isotropically. Weighting scheme $w = [\sigma_c^2 + (0.030|F_o|)^2]^{-1}$; no extinction correction; $R(F) = 0.036$, $wR(F) =$
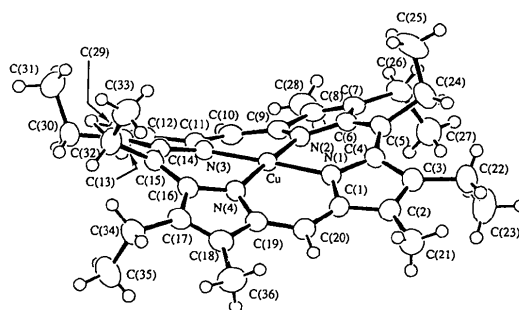

Fig. 1. *ORTEP* view of the molecule with the atom numbering. The thermal ellipsoids are given at 50% probability.

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Table 1. *Fractional coordinates ($\times 10^5$ for Cu; $\times 10^4$ for other atoms) and equivalent isotropic thermal parameters ($\mathring{A}^2$) for the non-H atoms*

$$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Cu	47456 (1)	30208 (2)	2606 (1)	2·4
N(1)	5292 (1)	2049 (1)	509 (1)	2·7
N(2)	5191 (1)	3779 (1)	1178 (1)	2·6
N(3)	4206 (1)	4001 (1)	−12 (1)	2·6
N(4)	4315 (1)	2294 (1)	−702 (1)	2·6
C(1)	5333 (1)	1356 (2)	−42 (1)	2·8
C(2)	5792 (1)	814 (2)	311 (2)	3·3
C(3)	6022 (1)	1157 (2)	1092 (1)	3·2
C(4)	5707 (1)	1944 (2)	1218 (1)	2·9
C(5)	5810 (1)	2573 (2)	1903 (1)	3·0
C(6)	5609 (1)	3491 (2)	1832 (1)	2·9
C(7)	5816 (1)	4308 (2)	2363 (1)	3·1
C(8)	5535 (1)	5070 (2)	1998 (1)	3·0
C(9)	5131 (1)	4729 (2)	1279 (1)	2·7
C(10)	4722 (1)	5261 (1)	818 (1)	2·9
C(11)	4279 (1)	4919 (1)	249 (1)	2·7
C(12)	3813 (1)	5445 (2)	−80 (1)	3·1
C(13)	3446 (1)	4836 (2)	−496 (1)	3·0
C(14)	3697 (1)	3916 (2)	−473 (1)	2·7
C(15)	3491 (1)	3092 (2)	−922 (1)	2·9
C(16)	3802 (1)	2402 (2)	−1132 (1)	2·7
C(17)	3669 (1)	1770 (2)	−1854 (1)	3·1
C(18)	4106 (1)	1318 (2)	−1855 (1)	3·3
C(19)	4499 (1)	1608 (2)	−1111 (1)	2·9
C(20)	4975 (1)	1188 (2)	−808 (1)	3·1
C(21)	5962 (1)	21 (2)	−140 (2)	4·4
C(22)	6550 (1)	833 (2)	1601 (2)	4·2
C(23)	6964 (1)	1495 (3)	1541 (2)	6·4
C(24)	6129 (1)	2222 (2)	2760 (2)	4·4
C(25)	5817 (1)	2152 (2)	3360 (2)	6·0
C(26)	6295 (1)	4411 (2)	3097 (1)	3·9
C(27)	6767 (1)	4522 (2)	2812 (2)	5·1
C(28)	5617 (1)	6078 (2)	2257 (2)	3·9
C(29)	3765 (1)	6493 (2)	45 (2)	4·3
C(30)	2900 (1)	5134 (2)	−931 (2)	3·7
C(31)	2563 (1)	5149 (2)	−355 (2)	5·6
C(32)	2917 (1)	2913 (2)	−1140 (2)	3·9
C(33)	2806 (1)	2000 (2)	−765 (2)	4·8
C(34)	3178 (1)	1693 (2)	−2570 (2)	3·9
C(35)	3159 (1)	2367 (2)	−3276 (2)	5·4
C(36)	4199 (1)	652 (2)	−2490 (2)	4·6

$0·051$, $S = 1·27$ for 5352 independent reflections, 547 parameters refined. Maximum and minimum peak heights in difference Fourier map 0·35 and $-0·22\, e\,\mathring{A}^{-3}$, $(\Delta/\sigma)_{max} = 0·5$. Atomic scattering factors and anomalous-scattering corrections from *International Tables for X-ray Crystallography* (1974). All the calculations were made by *UNICS*III (Sakurai & Kobayashi, 1979) and *ORTEP* (Johnson, 1965). Deviations from the least-square planes and their e.s.d.'s were calculated by the procedure proposed by Ito (1981).

Atomic parameters are given in Table 1.* Bond lengths and angles for non-H atoms and selected interatomic short contacts are given in Table 2. The molecular structure and the atomic numbering scheme are shown in Fig. 1.

Table 2. *Bond distances ($\mathring{A}$), bond angles (°) and selected interatomic short contacts ($\mathring{A}$) within a molecule*

Cu—N(1)	1·983 (2)	C(8)—C(9)	1·448 (3)
Cu—N(2)	1·965 (2)	C(8)—C(28)	1·488 (3)
Cu—N(3)	1·978 (2)	C(9)—C(10)	1·377 (3)
Cu—N(4)	1·973 (2)	C(10)—C(11)	1·384 (3)
N(1)—C(1)	1·374 (3)	C(11)—C(12)	1·438 (3)
N(1)—C(4)	1·380 (2)	C(12)—C(13)	1·347 (3)
N(2)—C(6)	1·385 (2)	C(12)—C(29)	1·508 (3)
N(2)—C(9)	1·372 (3)	C(13)—C(14)	1·468 (3)
N(3)—C(11)	1·367 (3)	C(13)—C(30)	1·514 (3)
N(3)—C(14)	1·380 (3)	C(14)—C(15)	1·409 (3)
N(4)—C(16)	1·381 (3)	C(15)—C(16)	1·408 (3)
N(4)—C(19)	1·369 (3)	C(15)—C(32)	1·524 (3)
C(1)—C(2)	1·439 (3)	C(16)—C(17)	1·458 (3)
C(1)—C(20)	1·378 (3)	C(17)—C(18)	1·359 (4)
C(2)—C(3)	1·356 (3)	C(17)—C(34)	1·510 (3)
C(2)—C(21)	1·503 (4)	C(18)—C(19)	1·438 (3)
C(3)—C(4)	1·465 (3)	C(18)—C(36)	1·498 (4)
C(3)—C(22)	1·511 (3)	C(19)—C(20)	1·384 (3)
C(4)—C(5)	1·409 (3)	C(22)—C(23)	1·500 (5)
C(5)—C(6)	1·402 (3)	C(24)—C(25)	1·507 (5)
C(5)—C(24)	1·518 (3)	C(26)—C(27)	1·516 (4)
C(6)—C(7)	1·462 (3)	C(30)—C(31)	1·523 (5)
C(7)—C(8)	1·359 (3)	C(32)—C(33)	1·508 (4)
C(7)—C(26)	1·511 (3)	C(34)—C(35)	1·506 (4)

N(1)—Cu—N(2)	87·84 (7)	C(7)—C(8)—C(28)	128·0 (2)
N(1)—Cu—N(3)	178·54 (8)	C(9)—C(8)—C(28)	124·8 (2)
N(1)—Cu—N(4)	92·17 (7)	N(2)—C(9)—C(8)	109·8 (2)
N(2)—Cu—N(3)	92·48 (7)	N(2)—C(9)—C(10)	125·1 (2)
N(2)—Cu—N(4)	176·94 (8)	C(8)—C(9)—C(10)	124·9 (2)
N(3)—Cu—N(4)	87·43 (7)	C(9)—C(10)—C(11)	126·2 (2)
Cu—N(1)—C(1)	124·3 (1)	N(1)—C(11)—C(10)	124·5 (2)
Cu—N(1)—C(4)	128·9 (2)	N(3)—C(11)—C(12)	110·0 (2)
C(1)—N(1)—C(4)	106·7 (2)	C(10)—C(11)—C(12)	125·2 (2)
Cu—N(2)—C(6)	128·8 (2)	C(11)—C(12)—C(13)	107·8 (2)
Cu—N(2)—C(9)	124·2 (1)	C(11)—C(12)—C(29)	124·0 (2)
C(6)—N(2)—C(9)	107·0 (2)	C(13)—C(12)—C(29)	128·2 (2)
Cu—N(3)—C(11)	124·4 (1)	C(12)—C(13)—C(14)	106·4 (2)
Cu—N(3)—C(14)	128·9 (1)	C(12)—C(13)—C(30)	123·1 (2)
C(11)—N(3)—C(14)	106·7 (2)	C(14)—C(13)—C(30)	130·4 (2)
Cu—N(4)—C(16)	129·1 (2)	N(3)—C(14)—C(13)	108·9 (2)
Cu—N(4)—C(19)	123·8 (1)	N(3)—C(14)—C(15)	123·0 (2)
C(16)—N(4)—C(19)	106·9 (2)	C(13)—C(14)—C(15)	127·6 (2)
N(1)—C(1)—C(2)	110·1 (2)	C(14)—C(15)—C(16)	122·0 (2)
N(1)—C(1)—C(20)	124·5 (2)	C(14)—C(15)—C(32)	118·9 (2)
C(2)—C(1)—C(20)	125·3 (2)	C(16)—C(15)—C(32)	119·0 (2)
C(1)—C(2)—C(3)	107·4 (2)	N(4)—C(16)—C(15)	123·1 (2)
C(1)—C(2)—C(21)	123·9 (2)	N(4)—C(16)—C(17)	108·9 (2)
C(3)—C(2)—C(21)	128·7 (2)	C(15)—C(16)—C(17)	127·8 (2)
C(2)—C(3)—C(4)	106·7 (2)	C(16)—C(17)—C(18)	106·9 (2)
C(2)—C(3)—C(22)	121·4 (2)	C(16)—C(17)—C(34)	129·9 (2)
C(4)—C(3)—C(22)	131·0 (2)	C(18)—C(17)—C(34)	122·6 (2)
N(1)—C(4)—C(3)	109·1 (2)	C(17)—C(18)—C(19)	107·2 (2)
N(1)—C(4)—C(5)	122·8 (2)	C(17)—C(18)—C(36)	129·5 (2)
C(3)—C(4)—C(5)	127·9 (2)	C(19)—C(18)—C(36)	123·3 (2)
C(4)—C(5)—C(6)	122·6 (2)	N(4)—C(19)—C(18)	109·9 (2)
C(4)—C(5)—C(24)	118·5 (2)	N(4)—C(19)—C(20)	124·9 (2)
C(6)—C(5)—C(24)	118·8 (2)	C(18)—C(19)—C(20)	124·9 (2)
N(2)—C(6)—C(5)	123·3 (2)	C(1)—C(20)—C(19)	126·5 (2)
N(2)—C(6)—C(7)	108·8 (2)	C(3)—C(22)—C(23)	112·8 (2)
C(5)—C(6)—C(7)	127·6 (2)	C(5)—C(24)—C(25)	111·7 (2)
C(6)—C(7)—C(8)	107·0 (2)	C(7)—C(26)—C(27)	111·7 (2)
C(6)—C(7)—C(26)	130·7 (2)	C(13)—C(30)—C(31)	113·7 (2)
C(8)—C(7)—C(26)	121·6 (2)	C(15)—C(32)—C(33)	111·5 (2)
C(7)—C(8)—C(9)	107·2 (2)	C(17)—C(34)—C(35)	112·8 (2)

C(21)···C(22)	3·089 (4)	C(29)···C(30)	3·106 (3)
C(22)···C(24)	3·209 (4)	C(30)···C(32)	3·163 (4)
C(24)···C(26)	3·155 (4)	C(32)···C(34)	3·201 (4)
C(26)···C(28)	3·070 (4)	C(34)···C(36)	3·128 (4)

Related literature. The synthesis of the free base of the title compound has been reported by Maruyama *et al.* (1988), who also studied the conformation of 5,15-dialkylporphyrins in solution by ^{1}H NMR. Two crystal structures have been reported for octaalkylporphyrins with alkyl substituents at the 5,15-positions (Collman, Chong, Jameson, Oakley, Rose,

Schmittou & Ibers, 1981; Lay, Buchler, Kenny & Scheidt, 1986). Both revealed pronounced non-planarity of the porphinato core owing to steric hindrance between the alkyl substituents.

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N-(Diméthyl-4,6 pyridyl-2) Phényl-3 Propènamide-(E) Hydrate (2/1)

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Abstract. $2C_{16}H_{16}N_2O.H_2O$, $M_r = 522.65$, monoclinic, $P2_1/n$, $a = 14.208$ (3), $b = 14.764$ (3), $c = 28.592$ (5) Å, $\beta = 103.76$ (2)°, $V = 5825$ (4) Å^3, $Z = 8$, $D_x = 1.192$ Mg m^{-3}, λ(Mo $K\bar{\alpha}$) $= 0.7107$ Å, $\mu = 0.072$ mm^{-1}, $F(000) = 2224$, $T = 295$ (1) K, $R = 0.044$ for 3584 independent reflections. Bond lengths and angles agree with those obtained for related compounds. The four organic molecules in the asymmetric unit have similar geometries. Each of them is approximately planar with a delocalized orbital over the amide group. The largest value of the dihedral angle between the phenyl-ring plane and that of the ethylenic bond is 5.5°. So the π electrons of the ethylenic bond are conjugated with those of the phenyl ring. In the four molecules, the dihedral angles between the two ring planes are respectively 18.0, 1.9, 7.8 and 1.9°. This compound proved to
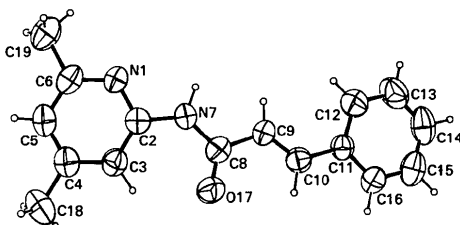
have anti-inflammatory properties. It has been studied in order to determine its precise molecular geometry and, therefore, to determine the critical values of the steric parameters which allow pharmacological activity.

Partie expérimentale. Cristal approximativement parallélépipédique: 0,19 × 0,21 × 0,32 mm. Dimensions de la maille déterminées avec 25 réflexions telles
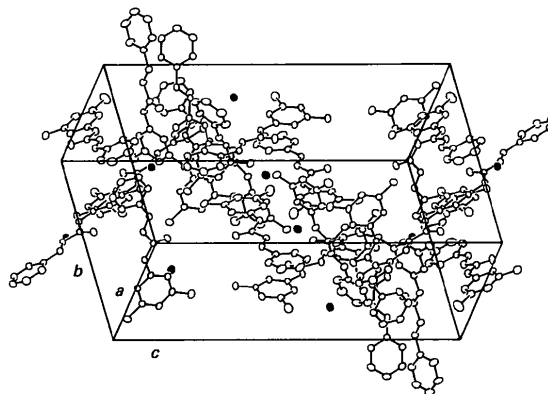

Fig. 1. Dessin de la molécule 1 et numéros attribués à ses atomes.

Fig. 2. Vue de la structure en perspective. Les ellipsoides noirs représentent les atomes d'oxygène des molécules d'eau. a est orienté de l'avant vers l'arrière de la figure, b du bas vers le haut et c de la gauche vers la droite.