$[Fe(tpp)(CN)_2]^-$ (Scheidt, Haller & Hatano, 1980) of 1·975 (2) Å is seen to be slightly longer. The Fe—C—N unit is linear with an observed angle of 179·1 (1)°. In common with all other mixed axial ligand derivatives of iron(III) porphyrins, (Adams, Rasmussen, Scheidt & Hatano, 1979; Scheidt, Lee, Geiger, Taylor & Hatano, 1982; Scheidt, Lee, Luangdilok, Haller, Anzai & Hatano, 1983; Nasri, Wang, Huynh, Walker & Scheidt, 1991) the porphyrin core in [Fe(oep)(CN)(py)] displays a significant S_4 ruffling.

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Structure of an Inclusion Compound of $Ag_2(SCN)_4$ and 5,7,7,12,14,14-Hexamethyl-1,4,8,11-tetraazatricyclo[9.3.1.1^{4,8}]hexadecane

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Abstract. $Ag_2(SCN)_4.C_{18}H_{36}N_4$, $M_r = 756·56$, monoclinic, $P2_1/n$, $a = 12·032$ (3), $b = 7·288$ (4), $c = 16·933$ (4) Å, $\beta = 90·44$ (4)°, $V = 1485$ (2) Å^3, $Z = 2$, $D_x = 1·691$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 16·035$ cm^{-1}, $F(000) = 764$, room temperature, final $R = 0·039$ for 2434 reflections with $I > 3\sigma(I)$. The crystal structure consists of infinite $[Ag_2(SCN)_4]_n$ chains and isolated 5,7,7,12,14,14-hexamethyl-1,4,8,11-tetraazatricyclo[9.3.1.1^{4,8}]hexadecane molecules. The shortest contact between the macrocyclic molecule and an Ag atom is 4·424 (3) Å. $[Ag_2(SCN)_4]_n$ forms a zigzag chain along **b** with an internal centre of symmetry.

Experimental. The title compound was obtained from the reaction of $AgK(SCN)_2$ with 5,7,7,12,14,14-hexamethyl-1,4,8,11-tetraazatricyclotetradecane in a water/ethanol solution. A colourless transparent single crystal with dimensions of 0·15 × 0·15 × 0·17 mm was used for X-ray structural analysis. Cell dimensions were obtained from a least-squares refinement of 25 reflections in the range $9 < \theta < 16°$. An Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation (λ = 0·71073 Å) was used. Diffraction intensities in the range $2 < \theta < 25°$ were measured using the $\omega/2\theta$ scan mode, index range h 0→14, k 0→8, l −20→20. The maximum scan time was 60 s. 3801 independent reflections were collected of which 2434 with $I > 3\sigma(I)$ were used in the structure refinement. Three standard intensity reflections monitored every 200 reflections showed no significant change in intensity. The data were corrected for Lorentz and polarization effects. An absorption correction was made with

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Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$B_{eq} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ac(\cos\beta)B(1,3)]$.

	x	y	z	B_{eq}(Å^2)
Ag(1)	−0·04749 (4)	0·16743 (5)	−0·03575 (3)	5·075 (5)
S(1)	0·16871 (9)	0·1154 (1)	0·03363 (7)	4·40 (2)
S(2)	−0·0267 (1)	0·3631 (2)	−0·15426 (6)	4·90 (2)
N(1)	0·3253 (4)	−0·0024 (5)	−0·0773 (3)	4·88 (7)
N(2)	−0·0670 (4)	0·3304 (8)	0·0801 (3)	5·21 (7)
N(11)	0·4047 (2)	−0·3645 (3)	−0·1010 (1)	2·50 (3)
N(12)	0·5686 (2)	−0·5428 (3)	−0·0820 (1)	2·37 (3)
C(1)	0·2609 (3)	0·0408 (4)	−0·0314 (3)	3·82 (6)
C(2)	0·0282 (3)	0·5418 (6)	−0·1093 (2)	3·95 (5)
C(10)	0·3291 (2)	−0·4324 (4)	0·0360 (2)	2·65 (4)
C(11)	0·3342 (2)	−0·4874 (4)	−0·0510 (2)	2·70 (4)
C(12)	0·3994 (2)	−0·4163 (5)	−0·1879 (2)	3·13 (4)
C(13)	0·4558 (3)	−0·6020 (5)	−0·1999 (2)	3·48 (5)
C(14)	0·5750 (2)	−0·6134 (5)	−0·1643 (2)	3·09 (4)
C(21)	0·2806 (3)	−0·4131 (8)	−0·2197 (2)	4·53 (7)
C(22)	0·6043 (4)	−0·8172 (5)	−0·1622 (3)	4·71 (7)
C(23)	0·6593 (3)	−0·5140 (8)	−0·2160 (2)	4·45 (6)
C(00)	0·5254 (2)	−0·3608 (3)	−0·0783 (2)	2·46 (3)

Table 2. *Bond lengths* (Å), *bond angles* (°) *and torsion angles* (°)

Ag(1)—S(1)	2·871 (1)	Ag(1)—S(2)	2·476 (1)
Ag(1)—N(2)	2·307 (5)	Ag(1)—S(1^i)	2·526 (1)
S(1)—C(1)	1·661 (4)	S(2)—C(2)	1·645 (4)
N(1)—C(1)	1·146 (7)	N(2)—C(2)	1·152 (7)
N(11)—C(11)	1·500 (4)	N(11)—C(12)	1·520 (4)
N(11)—C(00)	1·500 (3)	N(12)—C(10)	1·463 (4)
N(12)—C(14)	1·488 (4)	C(12)—C(13)	1·528 (5)
C(10)—C(11)	1·528 (5)	C(13)—C(14)	1·554 (4)
C(12)—C(21)	1·524 (4)	C(14)—C(23)	1·527 (5)
C(14)—C(22)	1·527 (5)	N(12)—C(00)	1·426 (3)

S(1)—Ag(1)—S(2)	108·1 (1)	S(1)—Ag(1)—N(2)	79·5 (4)
S(2)—Ag(1)—N(2)	113·85 (4)	S(1)—Ag(1)—S(1^i)	114·1 (1)
S(2)—Ag(1)—S(1^i)	122·92 (1)	N(2)—Ag(1)—S(1^i)	110·22 (4)
Ag(1)—S(1)—C(1)	97·54 (3)	Ag(1)—S(2)—C(2)	97·10 (4)
Ag(1)—N(2)—C(2)	137·6 (1)	C(11)—N(11)—C(12)	112·25 (6)
C(11)—N(11)—C(00)	114·62 (6)	C(12)—N(11)—C(00)	106·63 (6)
C(10)—N(12)—C(00)	113·39 (6)	C(10)—N(12)—C(00)	113·39 (6)
C(14)—N(12)—C(00)	112·61 (7)	S(1)—C(1)—N(1)	176·84 (9)
S(2)—C(2)—N(2)	177·8 (1)	N(12)—C(10)—C(11)	116·18 (6)
N(11)—C(11)—C(10)	114·53 (6)	N(11)—C(12)—C(13)	109·47 (6)
N(11)—C(12)—C(21)	111·73 (7)	C(13)—C(12)—C(21)	112·5 (1)
C(12)—C(13)—C(14)	113·92 (8)	N(12)—C(14)—C(13)	106·84 (6)
N(12)—C(14)—C(22)	109·11 (8)	N(12)—C(14)—C(23)	114·28 (9)
C(13)—C(14)—C(22)	105·90 (9)	C(13)—C(14)—C(23)	111·62 (9)
C(22)—C(14)—C(23)	108·7 (1)	N(11)—C(00)—N(12)	108·95 (6)

N(11)—C(12)—C(13)—C(14)	−52·2 (3)	C(12)—C(13)—C(14)—N(12)	49·0 (4)
C(13)—C(14)—N(12)—C(00)	−57·1 (3)	C(14)—N(12)—C(00)—N(11)	68·9 (6)
N(12)—C(00)—N(11)—C(12)	−67·0 (4)	C(00)—N(11)—C(12)—C(13)	58·3 (5)
C(12)—N(11)—C(11)—C(10)	−175·4 (8)	C(00)—N(11)—C(11)—C(10)	62·7 (6)
C(11)—N(11)—C(12)—C(13)	−68·0 (5)	C(11)—N(11)—C(12)—C(21)	57·3 (4)
C(00)—N(11)—C(12)—C(21)	−176·4 (9)	C(11)—N(11)—C(00)—N(12)	57·9 (4)
C(00)—N(12)—C(14)—C(22)	−171·2 (7)	C(00)—N(12)—C(14)—C(23)	66·9 (6)
C(21)—C(12)—C(13)—C(14)	−177·0 (9)	C(12)—C(13)—C(14)—C(22)	165·2 (7)
C(12)—C(13)—C(14)—C(23)	−76·6 (6)		

Symmetry code: (i) $-x, -y, -z$.

transmission factors in the range 0·85–0·99. The structure was solved from Patterson and subsequent electron-density difference maps. All 18 H atoms were located from these maps. The structure was refined (on F) by full-matrix least squares with isotropic thermal parameters for H atoms and anisotropic temperature factors for non-H atoms. The final discrepancy factors were $R = 0·039$ and $wR = 0·050$. Unit weights were used and the number of parameters was 236. Maximum shift/e.s.d. in the final cycle was 2·55 and no residual electron density

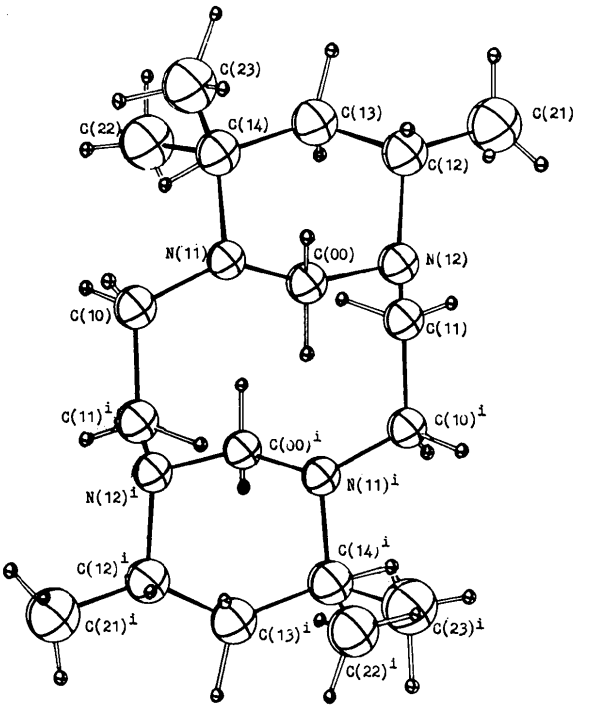

Fig. 1. Perspective drawing of the macrocyclic molecule. Symmetry code: (i) $-x, -y$.

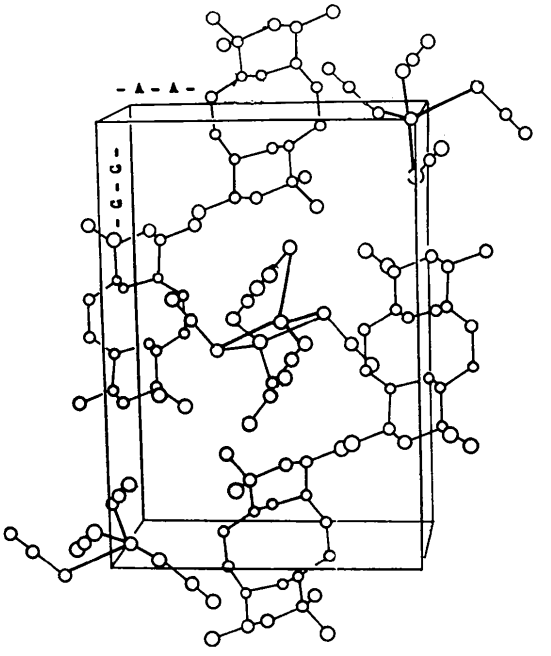

Fig. 2. Packing diagram of the title compound.

$> |0.991|$ e Å^{-3} was found. Calculations were performed on a PDP 11/34 computer with the *SDP* system (B. A. Frenz & Associates, Inc., 1982). Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B).

Fractional coordinates and equivalent isotropic temperature factors for non-H atoms are given in Table 1.* Bond distances and angles are given in Table 2. Fig. 1 shows the perspective drawing of the macrocycle molecule. Fig. 2 shows the packing drawing of the title compound.

Related literature. In recent years, there has been a growing interest in polycyclic polyamines (Whimp, Baily & Curtis, 1970; Hay, Fraser & Ferguson, 1987). Metal complexes of Cu^{2+}, Ni^{2+}, Co^{2+}, Cr^{3+} with macrocyclic tetraaza ligands have been studied extensively (House, Hay & Ali, 1983). The crystal structure of the bridged tetraaza macrocyclic molecule 5,7,7,12,14,14-hexamethyl-1,4,8,11-tetraazatricyclo[9.3.1.1.^{4,8}]hexadecane has previously been determined (Alcock, More & Mok, 1980) as well as the structure of 1,4,8,11-tetraazatricyclo[9.3.1.1^{4,8}]-hexadecane (Gabe, Le Page & Prasad, 1982). The characteristic infinite chains of $[Ag_2(SCN)_4]_n$ are the same as in $Zn[Ag(SCN)_2]$ (Lu, Huang & Huang, 1982).

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, least-squares planes and C—H bond distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54125 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of the Complex [*N*-(*p*-Chlorophenyl)iminodiacetato]copper(II)

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Abstract. $[Cu(C_{10}H_8ClNO_4)]$, $M_r = 305.17$, orthorhombic, *Pbca*, $a = 8.783$ (2), $b = 9.304$ (3), $c = 26.993$ (5) Å, $V = 2206$ (3) Å^3, $Z = 8$, $D_x = 1.838$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 22.28$ cm^{-1}, $F(000) = 1224$, room temperature, $R = 0.037$, $wR = 0.045$ for 634 observed reflections with $I > 3\sigma(I)$. Cu is coordinated by one amino N and three carboxyl O atoms in an approximately square-planar configuration. Two five-membered rings with an envelope conformation are formed. As the N and two carboxyl O atoms are from one ligand and the third O atom is from another, a chain structure is formed.

Experimental. Crystals of the title compound were obtained by the slow evaporation of an ethanol solution of *p*-chlorophenyliminodiacetic acid and $Cu(ClO_4)_2$. A green single crystal (0.15 × 0.14 × 0.14 mm) was used for the data collection on an Enraf–Nonius CAD-4 four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation (λ = 0.71073 Å). Unit-cell dimensions were obtained from a least-squares fit of 25 reflections in the range $12.8 < 2\theta < 24.1°$. Intensities in the range $2 < 2\theta < 25°$ (h: 0→10, k: 0→11, l: 0→32) were measured using the $\omega/2\theta$ scan mode. Three standard reflections monitored after each group of 200 measurements showed no significant change in intensity. Maximum counting time for each reflection was 60 s. Systematic absences $0kl$ for $k = 2n + 1$, $h0l$ for $l = 2n + 1$ and $hk0$ for $h = 2n + 1$ indicated space group *Pbca*. 2263 unique reflections were collected. 634 with $I > 3\sigma(I)$

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