H atoms. Structure refinement converged to $R = 0.038$, $wR = 0.043$ and $S = 2.21$ for 267 parameters. Maximum shift/e.s.d. in final cycle was 1·64 and no residual electron density > |0·36| e Å^{-3} was found. Calculations were carried out on a PDP11/34 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).

The fractional coordinates and thermal parameters for non-H atoms are given in Table 1* and selected bond lengths and angles in Table 2. A perspective drawing of the molecule is shown in Fig. 1.

Related literature. The cluster molecule has a 'butterfly-like' four-membered-ring skeleton composed of two Fe and two S atoms. The Fe(1)—Fe(2) bond of 2·254 (2) Å arises from the d^2sp^3 orbitals of Fe forming a bent metal–metal bond (Dahl, Martell & Wampler, 1961). Each Fe is surrounded by three carbonyls and two S atoms forming a regular tetragonal pyramid. The average Fe—C and C—O bond lengths compare well with distances found in other compounds, $Fe_2(CO)_6(COH)_2(CH_3C_2CH_3)$ (Hock & Mills, 1958), $C_8H_8Fe(CO)_3$ (Dickens & Lipscomb, 1961), $Fe_2(CO)_6(C_6H_5C_2H)_3$ (King, 1962) and $[C_2N_5SFe(CO)_3]_2$ (Dahl & Wei, 1963).

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* Lists of structure-factor amplitudes, anisotropic displacement parameters for the non-H atoms, H-atom parameters, C—H bond lengths and calculations of least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54070 (12 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 (Cyano)(2,3,7,8,12,13,17,18-octaethylporphinato)(pyridine)iron(III) Chloroform Solvate

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Abstract. $[Fe(C_{36}H_{44}N_4)(CN)(C_5H_5N)].CHCl_3$, $M_r = 813.12$, monoclinic, $P2_1/n$, $a = 10.345$ (2), $b = 14.748$ (2), $c = 27.302$ (3) Å, $\beta = 92.12$ (3)°, $V = 4162.6$ Å^3, $Z = 4$, $D_x = 1.30$, $D_m = 1.29$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0.71073$ Å, $\mu = 5.92$ cm^{-1}, $F(000) = 1708$, $T = 293$ K, $R = 0.046$ for 5193 unique observed $[F > 3\sigma(F)]$ reflections. The iron(III) atom is six-coordinate with an average Fe—N(p) distance of 1·980 (4) Å and axial Fe—C and Fe—N distances of 1·934 (4) and 2·087 (3) Å, respectively.

Experimental. The title compound was synthesized as part of a resonance Raman study of iron–ligand vibrations (Uno, Hatano, Nishimura & Arata, 1988). D_m measured by flotation. A purple, air-stable crystal of [Fe(oep)(CN)(py)] recrystallized from $CHCl_3$ with approximate dimensions of 0·31 × 0·27 × 0·27 mm was mounted on the end of a glass fiber. All measurements were performed with graphite-monochromated Mo $K\alpha$ radiation on an Enraf–Nonius CAD-4 diffractometer. Intensity data were measured by θ–2θ scans at a scan rate of 3°θ min^{-1}. Data were collected to a maximum 2θ of 54·9°, and standard θ–2θ CAD-4 moving-crystal moving-counter background measurements were used. 25 reflections used for measuring lattice parameters

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Table 1. *Fractional coordinates*

The e.s.d.'s of the least significant digits are given in parentheses.

$$B_{eq} = (4/3)[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + (2ab\cos\gamma)\beta_{12} + (2ac\cos\beta)\beta_{13} + (2bc\cos\alpha)\beta_{23}].$$

	x	y	z	B_{eq}(Å^2)
Fe	0·58572 (5)	0·19978 (3)	0·105991 (17)	2·51
N(1)	0·56555 (25)	0·06639 (17)	0·11158 (10)	2·67
N(2)	0·53432 (27)	0·21324 (18)	0·17472 (10)	2·93
N(3)	0·61407 (27)	0·33233 (18)	0·10171 (10)	2·81
N(4)	0·63703 (25)	0·18612 (17)	0·03744 (10)	2·65
N(5)	0·77874 (27)	0·18208 (19)	0·12893 (11)	3·10
N(6)	0·3029 (3)	0·21854 (26)	0·06876 (14)	5·00
C(1)	0·4077 (4)	0·21223 (24)	0·08275 (13)	3·26
C(*a*1)	0·6025 (3)	0·00250 (22)	0·07824 (13)	2·85
C(*a*2)	0·5176 (3)	0·01826 (22)	0·14985 (13)	2·99
C(*a*3)	0·4799 (3)	0·14789 (24)	0·20365 (13)	3·11
C(*a*4)	0·5420 (4)	0·29054 (25)	0·20289 (13)	3·48
C(*a*5)	0·6119 (4)	0·39492 (24)	0·13958 (13)	3·44
C(*a*6)	0·6342 (3)	0·38301 (22)	0·06052 (13)	3·01
C(*a*7)	0·6612 (3)	0·25479 (23)	0·00452 (12)	2·83
C(*a*8)	0·6638 (3)	0·10666 (22)	0·01306 (12)	2·68
C(*b*1)	0·5823 (3)	−0·08770 (22)	0·09683 (13)	3·13
C(*b*2)	0·5288 (3)	−0·07769 (23)	0·14139 (14)	3·18
C(*b*3)	0·4480 (4)	0·18643 (25)	0·25020 (13)	3·56
C(*b*4)	0·4920 (4)	0·27244 (28)	0·25089 (14)	4·24
C(*b*5)	0·6332 (4)	0·48504 (24)	0·12066 (15)	3·61
C(*b*6)	0·6431 (4)	0·47838 (23)	0·07175 (14)	3·43
C(*b*7)	0·7069 (3)	0·21679 (23)	−0·04052 (11)	2·95
C(*b*8)	0·7090 (3)	0·12589 (24)	−0·03483 (12)	3·00
C(*m*1)	0·4704 (3)	0·05770 (23)	0·19166 (13)	3·20
C(*m*2)	0·5831 (4)	0·37354 (26)	0·18706 (14)	3·76
C(*m*3)	0·6533 (4)	0·34605 (23)	0·01485 (13)	3·16
C(*m*4)	0·6490 (3)	0·02160 (22)	0·03287 (13)	3·05
C(11)	0·6208 (4)	−0·17265 (24)	0·07110 (15)	3·69
C(12)	0·7634 (5)	−0·1914 (3)	0·07625 (22)	5·85
C(21)	0·4922 (4)	−0·14984 (26)	0·17727 (16)	4·16
C(22)	0·5974 (6)	−0·1677 (4)	0·21617 (21)	6·72
C(31)	0·3788 (5)	0·13684 (29)	0·28921 (14)	4·31
C(32)	0·2348 (5)	0·1343 (4)	0·27923 (19)	6·14
C(41)	0·4979 (7)	0·3404 (4)	0·29411 (20)	6·09
C(42)	0·3869 (7)	0·3942 (5)	0·29234 (25)	8·97
C(51)	0·6385 (5)	0·56961 (27)	0·15291 (16)	4·69
C(52)	0·7660 (6)	0·5817 (4)	0·17938 (22)	7·15
C(61)	0·6634 (4)	0·55312 (26)	0·03454 (16)	4·34
C(62)	0·8055 (5)	0·5659 (3)	0·02477 (18)	5·66
C(71)	0·7570 (4)	0·27306 (27)	−0·08145 (13)	3·82
C(72)	0·8915 (5)	0·3088 (3)	−0·07025 (16)	5·19
C(81)	0·7583 (4)	0·05567 (27)	−0·06936 (13)	3·79
C(82)	0·8969 (5)	0·0326 (4)	−0·05780 (21)	6·68
C(2)	0·8108 (4)	0·14091 (29)	0·17122 (15)	4·36
C(3)	0·9366 (5)	0·1223 (3)	0·18559 (17)	5·27
C(4)	1·0346 (4)	0·1454 (3)	0·15584 (20)	5·17
C(5)	1·0036 (4)	0·1873 (3)	0·11177 (17)	4·69
C(6)	0·8748 (4)	0·20529 (27)	0·10044 (14)	4·02
C(7)	0·1805 (5)	0·3911 (3)	0·12191 (19)	5·80
Cl(1)	0·02671 (17)	0·43154 (12)	0·10591 (8)	9·23
Cl(2)	0·29706 (17)	0·47170 (12)	0·10913 (6)	8·51
Cl(3)	0·18842 (25)	0·36553 (13)	0·18401 (7)	10·30

Table 2. *Averaged bond distances* (Å) *and angles* (°) *in* [Fe(oep)(CN)(py)]

Fe—N(*p*)	1·980 (4)	C(*a*)—C(*m*)—C(*a*)	125·6 (3)
N(*p*)—C(*a*)	1·378 (6)	N(*p*)C(*a*)C(*m*)	124·4 (5)
C(*a*)—C(*b*)	1·443 (5)	C(*a*)N(*p*)C(*a*)	105·4 (5)
C(*a*)—C(*m*)	1·377 (5)	C(*b*)C(*a*)C(*m*)	125·2 (5)
C(*b*)—C(*b*)	1·352 (7)	N(*p*)C(*a*)C(*b*)	110·4 (6)
C(*b*)—C(*n*1)	1·512 (18)	C(*a*)C(*b*)C(*b*)	106·9 (6)
C(*n*1)—C(*n*2)	1·49 (4)	C(*b*)C(*b*)C(*n*1)	128·8 (4)
		C(*a*)C(*b*)C(*n*1)	124·3 (6)
		C(*b*)—C(*n*1)—C(*n*2)	112·0 (11)

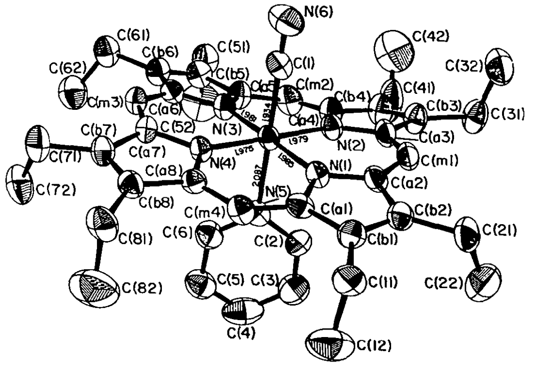

Fig. 1. *ORTEP* (Johnson, 1976) diagram and numbering scheme for all heavy atoms. 50% probability surfaces. Distances are given in Å.

$10·30 < \theta < 14·08°$. Range of *hkl*: $0\rightarrow13$, $-18\rightarrow18$, $-35\rightarrow35$, 23 219 reflections measured, 9576 unique, 5193 with $F > 3\sigma(F)$ considered observed. Merging $R = 0·025$ for 9573 duplicates. Four standard reflections, 0·3% intensity decrease. The data were corrected for Lorentz, polarization and absorption effects using the Blessing (1987) suite for data reduction. Empirical absorption was based on azimuthal (ψ) scans of seven reflections. Solved by direct methods: *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Full-matrix least squares minimized $\sum w(\Delta F)^2$ with a total of 478 variables. The H atoms were positioned according to idealized geometry (C—H = 0·95 Å) and given isotropic B's of 1·3 times B of the attached atom. All non-H atoms refined with anisotropic temperature factors. $R = 0·046$, $wR = 0·060$, $S = 1·71$. Weighting scheme $w = 1/[\sigma(F_o)]^2$. Final difference peak 0·71 e Å^{-3} near Cl(3), $(\Delta/\sigma)_{max} = 0·11$. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV). A locally modified version of *ALLS* (Lapp & Jacobson, 1979) used for structure refinement and *ORFFE* (Busing, Martin & Levy, 1964) for structural parameters with e.s.d.'s. Table 1 gives atomic coordinates and Table 2 averaged bond distances and angles.* Fig. 1 gives the atomic numbering of the independent atoms and a perspective drawing of this molecule.

Related literature. The average Fe—N(*p*) distance of 1·980 (4) Å is typical of that for low-spin porphinatoiron(III) complexes (Scheidt & Reed, 1981). The axial Fe—C(CN) distance of 1·934 (4) Å and the axial Fe—N(py) distance of 2·087 (3) Å are comparable, but possibly slightly longer, than the 1·908 (4) and 2·075 (3) Å values found for [Fe(tpp)(CN)(py)] (Scheidt, Lee, Luangdilok, Haller, Anzai & Hatano, 1983). The unique Fe—C(CN) distance in

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and complete bond distance and angle tables have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54090 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

$[Fe(tpp)(CN)_2]^-$ (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. $Ag_2(SCN)_4.C_{18}H_{36}N_4$, $M_r = 756{\cdot}56$, monoclinic, $P2_1/n$, $a = 12{\cdot}032\ (3)$, $b = 7{\cdot}288\ (4)$, $c = 16{\cdot}933\ (4)$ Å, $\beta = 90{\cdot}44\ (4)°$, $V = 1485\ (2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}691$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 16{\cdot}035$ cm^{-1}, $F(000) = 764$, room temperature, final $R = 0{\cdot}039$ 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 ($\lambda = 0{\cdot}71073$ 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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