Pearson, R. G. (1963). *J. Am. Chem. Soc.* **85**, 3533–3539.
Sheldrick, G. M. (1976). *SHELX76. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen.
Van Kralingen, C. G. & Reedijk, J. (1978). *Inorg. Chim. Acta*, **30**, 171–177.
Van Kralingen, C. G., Reedijk, J. & De Ridder, J. K. (1979). *Inorg. Chim. Acta*, **36**, 69–77.

Acta Cryst. (1987). C**43**, 1261–1264

Structures of Bis(aqua)bis(isoquinoline)copper(II) Perchlorate and of Tetrakis(isoquinoline)bis(trifluoromethanesulfonato)copper(II)

By K. Al Sarraj, J. Gouteron, S. Jeannin and Y. Jeannin

Laboratoire de Chimie des Métaux de Transition, UA n° 419, *Université Pierre et Marie Curie,*
4 *Place Jussieu,* 75232 *Paris, France*

(*Received* 13 *October* 1986; *accepted* 11 *February* 1987)

Abstract. Compound (1): $[Cu(C_9H_7N)_2(H_2O)_2](ClO_4)_2$, $M_r = 556{\cdot}5$, triclinic, $P\bar{1}$, $a = 7{\cdot}487\,(2)$, $b = 7{\cdot}621\,(2)$, $c = 10{\cdot}271\,(4)$ Å, $\alpha = 100{\cdot}82\,(2)$, $\beta = 108{\cdot}33\,(3)$, $\gamma = 99{\cdot}03\,(2)°$, $V = 531{\cdot}6\,(7)$ Å^3, $Z = 1$, $D_m = 1{\cdot}60$ (flotation in *n*-octane + 1,2-dibromoethane), $D_x = 1{\cdot}74$ Mg m^{-3}, μ(Mo $K\alpha$, $\lambda = 0{\cdot}71070$ Å) $= 1{\cdot}359$ mm^{-1}, $F(000) = 277$, $T = 294$ K, $R = 0{\cdot}041$ and $wR = 0{\cdot}035$ for 2741 significant reflections. Octahedral copper environment: two water molecules in *trans* positions and two isoquinolines in *trans* positions for basal plane, and two perchlorate anions in apical positions. Each perchlorate anion connected to three different copper atoms through semicoordinated O and through two hydrogen bonds. Compound (2): $[Cu(C_9H_7N)_4(CF_3SO_3)_2]$, $M_r = 877{\cdot}5$, triclinic, $P\bar{1}$, $a = 10{\cdot}882\,(3)$, $b = 18{\cdot}219\,(8)$, $c = 22{\cdot}384\,(3)$ Å, $\alpha = 67{\cdot}49\,(2)$, $\beta = 88{\cdot}64\,(2)$, $\gamma = 83{\cdot}34\,(3)°$, $V = 4071\,(4)$ Å^3, $Z = 4$, $D_m = 1{\cdot}30$ (flotation in *n*-octane + 1,2-dibromoethane), $D_x = 1{\cdot}43$ Mg m^{-3}, μ(Mo $K\alpha$, $\lambda = 0{\cdot}71070$ Å) $= 0{\cdot}643$ mm^{-1}, $F(000) = 1568$, $T = 294$ K, $R = 0{\cdot}071$ and $wR = 0{\cdot}049$ for 3540 significant reflections. Octahedral copper environment: four isoquinolines in basal plane and two monodentate anions semicoordinated through O in apical positions.

Introduction. A large number of studies have been carried out on complexes of copper(II) with pyridine. Some work involved isoquinoline, a monodentate aromatic amine of larger size than pyridine, but with similar donor properties. For example, from spectroscopic measurements, copper(II) complexes $[CuL_2Cl_2]$ (L = quinoline or isoquinoline) are supposed to have polymeric octahedral structures involving halogen bridges as for pyridine complexes (Patel & Ramana Rao, 1966). Also copper(II) complexes $[Cu(\mathrm{isoquin})_n(CF_3CO_2)_2]$ (where n = 2 or 4) have been studied by spectrometric methods (Agambar & Orrell, 1969). They are described as six-coordinated with trifluoroacetate (either mono- or bidentate). While looking for dinuclear copper complexes, we have synthesized and studied by X-ray diffraction $CuL_2(ClO_4)_2$ [compound (1)] and $CuL_4(CF_3SO_3)_2$ [compound (2)] with L = isoquinoline.

Experimental. Compound (1): $1{\cdot}4 \times 10^{-4}$ mol isoquinoline poured into 4 ml of a solution of $CuClO_4.6H_2O$ in propanone. Precipitated white powder filtered after 24 h. Blue solution slowly evaporated 10 days under nitrogen, giving bright green air-stable crystals. Crystal: 0·5 × 0·2 × 0·1 mm; precessions and Laue: $P\bar{1}$; Nonius CAD-4 diffractometer, graphite monochromator; Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71070$ Å); unit cell: least squares on 25 reflections, $8 < \theta < 28°$; intensity measurements by θ–2θ scan; $(\sin\theta)/\lambda$ range: 0·04–

Table 1. *Atomic parameters for compound* (1) *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
Cu	0·0000	0·0000	0·0000	2·75 (1)
O	0·1669 (2)	0·2297 (2)	−0·0037 (2)	3·70 (5)
N	0·0343 (2)	0·1116 (2)	0·2011 (2)	3·07 (4)
C(1)	−0·1128 (3)	0·0913 (3)	0·2451 (2)	3·15 (5)
C(2)	−0·1005 (3)	0·1652 (3)	0·3844 (2)	3·12 (5)
C(3)	−0·2639 (4)	0·1388 (4)	0·4259 (3)	4·08 (7)
C(4)	−0·2463 (4)	0·2123 (4)	0·5621 (3)	4·62 (8)
C(5)	−0·0657 (5)	0·3188 (4)	0·6608 (3)	4·73 (9)
C(6)	0·0939 (4)	0·3463 (4)	0·6244 (3)	4·44 (7)
C(7)	0·0815 (3)	0·2693 (3)	0·4846 (2)	3·40 (6)
C(8)	0·2371 (3)	0·2878 (4)	0·4343 (3)	4·13 (7)
C(9)	0·2111 (3)	0·2113 (3)	0·2982 (3)	3·73 (6)
Cl	0·34891 (6)	−0·26676 (6)	0·05836 (5)	2·86 (1)
O(1)	0·3092 (3)	−0·0884 (2)	0·0917 (2)	4·46 (5)
O(2)	0·4390 (3)	−0·2734 (4)	−0·0458 (3)	5·97 (3)
O(3)	0·4712 (4)	−0·3023 (4)	0·1815 (3)	6·62 (9)
O(4)	0·1688 (2)	−0·3984 (2)	−0·0014 (3)	5·20 (7)

0108-2701/87/071261-04$01.50

Table 2. *Interatomic distances* (Å) *and angles* (°) *for compound* (1) *with e.s.d.'s in parentheses*

Cu—O	1·999 (1)	Cu—N	1·999 (2)
Cu—O(1)	2·461 (2)	N—C(9)	1·376 (3)
N—C(1)	1·315 (3)	C(2)—C(7)	1·420 (3)
C(1)—C(2)	1·404 (3)	C(8)—C(9)	1·349 (4)
C(7)—C(8)	1·414 (4)	C(3)—C(4)	1·362 (4)
C(2)—C(3)	1·415 (3)	C(5)—C(6)	1·358 (5)
C(4)—C(5)	1·414 (4)	Cl—O(3)	1·410 (2)
Cl—O(1)	1·439 (2)	Cl—O(4)	1·423 (2)
Cl—O(2)	1·431 (2)		
N—Cu—O	89·8 (1)	N—Cu—O(1)	87·5 (1)
O—Cu—O(1)	84·0 (1)	C(7)—C(2)—C(3)	119·9 (2)
C(1)—N—C(9)	117·6 (2)	C(2)—C(3)—C(4)	120·0 (2)
N—C(9)—C(8)	122·6 (2)	C(3)—C(4)—C(5)	119·9 (3)
C(9)—C(8)—C(7)	121·1 (2)	C(4)—C(5)—C(6)	121·5 (2)
C(8)—C(7)—C(2)	116·2 (2)	C(5)—C(6)—C(7)	120·0 (2)
C(7)—C(2)—C(1)	118·4 (2)	C(6)—C(7)—C(2)	118·6 (2)
C(2)—C(1)—N	124·1 (2)	O(2)—Cl—O(3)	110·0 (2)
O(1)—Cl—O(2)	109·2 (1)	O(2)—Cl—O(4)	108·2 (2)
O(1)—Cl—O(3)	110·2 (1)	O(3)—Cl—O(4)	111·4 (2)
O(1)—Cl—O(4)	107·7 (1)		

0·70 Å^{-1}; scan speed depending upon reflection; background measured for half of scan time, before and after every scan; scan range: 1·2° + 0·345 tanθ, two standard reflections measured every other hour; four octants measured: *hkl*, *$\bar{h}$kl*, *h$\bar{k}$l*, *$\bar{h}\bar{k}$l*; 3402 independent reflections collected and 2978 reflections kept [$F > 3\sigma(F)$]; computing program: *SHELX*76 (Sheldrick, 1976); method used to solve structure: Patterson; calculations performed on Gould-Concept 3287 computer; scattering factors: *International Tables for X-ray Crystallography* (1974); absorption corrections: not applied; secondary extinction: none observed; weighting scheme: $3\cdot97/\sigma^2$; average shift/e.s.d.: 0·005 in the last cycle; $N_{\rm reflections}/N_{\rm var.par.}$: 18; $R(F) = 0\cdot041$; $wR = 0\cdot035$; $S = 7\cdot6$; max. height in final difference Fourier synthesis: 0·8 e Å^{-3}; coordinates of seven ring hydrogens calculated and allowed to shift according to the movement of parent atoms with one refinable overall isotropic thermal parameter; water hydrogens located on a Fourier difference synthesis and set in refinement in fixed positions. Table 1 shows atomic parameters and Table 2 selected interatomic distances and bond angles.*

Compound (2): 2·2 × 10^{-3} mol isoquinoline poured into 10 ml of an ethanolic solution 0·011 mol l^{-1} of copper(II) trifluoromethanesulfonate. Blue solution evaporated under nitrogen for 3 weeks. Indigo-blue air-unstable crystals obtained. Data collection and refinement as for compound (1), but: crystal: 0·4 × 0·15 × 0·2 mm; unit cell: least squares on 25 reflections,

Table 3. *Atomic parameters for compound* (2) *with e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$(Å^2)
Cu(1)	0·5117 (2)	0·1629 (1)	0·26344 (9)	3·89 (6)
N(11)	0·376 (1)	0·2437 (6)	0·2083 (5)	4·2 (2)
C(111)	0·396 (1)	0·2909 (8)	0·1485 (7)	4·1 (3)
C(112)	0·310 (1)	0·3568 (8)	0·1110 (7)	4·2 (3)
C(113)	0·340 (1)	0·4067 (9)	0·0480 (8)	5·6 (4)
C(114)	0·257 (1)	0·473 (1)	0·0136 (8)	6·2 (4)
C(115)	0·149 (1)	0·484 (1)	0·0438 (8)	5·6 (4)
C(116)	0·115 (1)	0·4379 (9)	0·1029 (7)	5·5 (3)
C(117)	0·200 (1)	0·3709 (9)	0·1403 (7)	4·9 (3)
C(118)	0·177 (1)	0·3206 (9)	0·2027 (8)	5·7 (4)
C(119)	0·266 (1)	0·2578 (8)	0·2347 (7)	4·6 (3)
N(12)	0·5107 (9)	0·2208 (6)	0·3244 (5)	4·1 (2)
C(121)	0·513 (1)	0·2984 (8)	0·3030 (7)	3·8 (3)
C(122)	0·512 (1)	0·3406 (8)	0·3444 (6)	3·9 (3)
C(123)	0·514 (1)	0·422 (1)	0·3212 (9)	6·6 (4)
C(124)	0·506 (2)	0·463 (1)	0·3632 (9)	7·5 (4)
C(125)	0·507 (2)	0·420 (1)	0·428 (1)	8·5 (5)
C(126)	0·510 (2)	0·338 (1)	0·455 (1)	8·1 (5)
C(127)	0·511 (1)	0·296 (1)	0·4113 (8)	5·8 (4)
C(128)	0·513 (1)	0·213 (1)	0·4341 (9)	6·3 (4)
C(129)	0·509 (1)	0·1797 (9)	0·3893 (7)	5·0 (3)
N(13)	0·6497 (9)	0·0815 (6)	0·3177 (5)	3·8 (2)
C(131)	0·638 (1)	0·0040 (8)	0·3414 (6)	3·4 (3)
C(132)	0·732 (1)	−0·0535 (8)	0·3768 (6)	3·9 (3)
C(133)	0·715 (1)	−0·1351 (9)	0·4010 (7)	5·2 (3)
C(134)	0·813 (2)	−0·191 (1)	0·4347 (8)	6·6 (4)
C(135)	0·921 (1)	−0·167 (1)	0·4455 (7)	5·8 (4)
C(136)	0·939 (1)	−0·0891 (9)	0·4252 (7)	5·5 (4)
C(137)	0·842 (1)	−0·0317 (8)	0·3893 (7)	4·5 (3)
C(138)	0·856 (1)	0·0519 (9)	0·3626 (7)	4·9 (3)
C(139)	0·760 (1)	0·1057 (9)	0·3290 (7)	4·8 (3)
N(14)	0·5195 (9)	0·1055 (6)	0·2020 (5)	3·7 (2)
C(141)	0·625 (1)	0·0846 (8)	0·1808 (6)	4·2 (3)
C(142)	0·638 (1)	0·0409 (8)	0·1404 (7)	4·4 (3)
C(143)	0·756 (1)	0·0171 (9)	0·1219 (7)	5·0 (3)
C(144)	0·765 (2)	−0·030 (1)	0·0864 (8)	6·4 (4)
C(145)	0·659 (2)	−0·053 (1)	0·0695 (8)	6·7 (4)
C(146)	0·543 (1)	−0·0305 (9)	0·0861 (7)	5·1 (3)
C(147)	0·530 (1)	0·0178 (8)	0·1238 (6)	4·2 (3)
C(148)	0·417 (1)	0·0397 (8)	0·1474 (7)	4·7 (3)
C(149)	0·415 (1)	0·0839 (8)	0·1842 (7)	4·5 (3)
Cu(2)	−0·0583 (1)	0·3349 (1)	0·72924 (9)	4·15 (6)
N(21)	0·1099 (9)	0·3156 (6)	0·6954 (5)	4·3 (2)
C(211)	0·191 (1)	0·2536 (8)	0·7278 (6)	3·7 (3)
C(212)	0·311 (1)	0·2406 (7)	0·7079 (6)	3·5 (3)
C(213)	0·389 (1)	0·1713 (8)	0·7432 (7)	4·4 (3)
C(214)	0·507 (1)	0·161 (1)	0·7212 (8)	5·8 (4)
C(215)	0·546 (1)	0·2160 (9)	0·6661 (7)	5·1 (3)
C(216)	0·472 (1)	0·2835 (9)	0·6303 (7)	4·9 (3)
C(217)	0·349 (1)	0·2976 (8)	0·6504 (6)	3·9 (3)
C(218)	0·262 (1)	0·3637 (9)	0·6147 (7)	4·9 (3)
C(219)	0·146 (1)	0·3711 (9)	0·6371 (7)	4·8 (3)
N(22)	−0·0821 (9)	0·2175 (6)	0·7529 (5)	3·7 (2)
C(221)	−0·098 (1)	0·1723 (8)	0·8132 (6)	3·9 (3)
C(222)	−0·098 (1)	0·0874 (7)	0·8342 (6)	3·3 (3)
C(223)	−0·106 (1)	0·0384 (8)	0·8995 (7)	4·6 (3)
C(224)	−0·101 (1)	−0·0423 (9)	0·9191 (8)	5·1 (3)
C(225)	−0·090 (1)	−0·0743 (9)	0·8726 (7)	5·0 (3)
C(226)	−0·083 (1)	−0·0305 (8)	0·8080 (7)	4·7 (3)
C(227)	−0·086 (1)	0·0541 (8)	0·7873 (6)	3·6 (3)
C(228)	−0·077 (1)	0·1040 (8)	0·7239 (7)	4·6 (3)
C(229)	−0·074 (1)	0·1842 (8)	0·7080 (7)	4·2 (3)
N(23)	−0·028 (1)	0·4496 (6)	0·7098 (5)	4·2 (2)
C(231)	−0·107 (1)	0·5114 (8)	0·6734 (7)	4·5 (3)
C(232)	−0·083 (1)	0·5910 (8)	0·6578 (7)	4·7 (3)
C(233)	−0·173 (1)	0·652 (1)	0·6197 (8)	6·1 (4)
C(234)	−0·147 (2)	0·730 (1)	0·6069 (8)	6·8 (4)
C(235)	−0·042 (2)	0·744 (1)	0·6292 (9)	7·9 (5)
C(236)	0·044 (2)	0·687 (1)	0·6678 (9)	7·2 (4)
C(237)	0·021 (1)	0·6051 (9)	0·6832 (7)	5·5 (3)
C(238)	0·104 (1)	0·5406 (9)	0·7221 (7)	5·6 (4)
C(239)	0·076 (1)	0·4655 (9)	0·7349 (7)	5·1 (3)
N(24)	−0·2272 (9)	0·3541 (6)	0·7641 (5)	4·0 (2)
C(241)	−0·246 (1)	0·3946 (7)	0·8018 (6)	3·7 (3)
C(242)	−0·365 (1)	0·4121 (7)	0·8230 (6)	3·4 (3)
C(243)	−0·380 (1)	0·4592 (9)	0·8613 (7)	5·3 (3)
C(244)	−0·497 (2)	0·479 (1)	0·8776 (8)	7·0 (4)
C(245)	−0·599 (2)	0·449 (1)	0·8597 (8)	6·7 (4)
C(246)	−0·585 (1)	0·4005 (9)	0·8275 (7)	5·1 (3)
C(247)	−0·465 (1)	0·3811 (8)	0·8074 (7)	4·2 (3)
C(248)	−0·442 (1)	0·3368 (8)	0·7685 (7)	4·9 (3)
C(249)	−0·326 (1)	0·3256 (9)	0·7470 (7)	5·0 (3)

---

* Lists of bond angles and distances and H-atom parameters for compound (2), and structure factors and anisotropic thermal parameters for both compounds have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43788 (44 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3 (*cont.*)

	x	y	z	B_{eq}(Å^2)
C(1)	0·867 (2)	0·266 (2)	0·139 (1)	8·5 (10)
F(11)	0·934 (1)	0·217 (1)	0·193 (1)	14·7 (9)
F(12)	0·938 (1)	0·3156 (9)	0·1005 (7)	13·0 (7)
F(13)	0·844 (1)	0·218 (1)	0·1118 (8)	12·9 (8)
S(1)	0·7345 (4)	0·3112 (3)	0·1630 (2)	5·7 (2)
O(11)	0·6699 (8)	0·2460 (6)	0·2003 (5)	5·6 (4)
O(12)	0·781 (2)	0·355 (1)	0·1934 (9)	12·7 (9)
O(13)	0·673 (1)	0·3583 (9)	0·1023 (7)	11·2 (6)
C(2)	0·296 (2)	−0·004 (2)	0·441 (1)	10·2 (11)
F(21)	0·593 (1)	0·0220 (9)	0·5365 (6)	14·4 (7)
F(22)	0·255 (2)	0·062 (1)	0·4545 (7)	17·4 (11)
F(23)	0·231 (1)	−0·0615 (9)	0·4761 (6)	12·7 (6)
S(2)	0·2792 (4)	0·0240 (3)	0·3579 (2)	6·0 (2)
O(21)	0·3602 (8)	0·0820 (6)	0·3315 (5)	5·5 (4)
O(22)	0·156 (1)	0·045 (1)	0·3433 (7)	11·6 (7)
O(23)	0·329 (2)	−0·052 (1)	0·355 (1)	16·3 (12)
C(3)	0·073 (3)	0·314 (2)	0·947 (1)	9·4 (12)
F(31)	0·104 (1)	0·3908 (9)	0·9130 (8)	12·3 (7)
F(32)	−0·043 (2)	0·325 (1)	0·9564 (9)	16·2 (10)
F(33)	0·136 (2)	0·284 (1)	0·9990 (7)	15·8 (9)
S(3)	0·1030 (4)	0·2581 (3)	0·8958 (2)	5·3 (2)
O(31)	0·233 (1)	0·2575 (8)	0·8840 (9)	11·0 (7)
O(32)	0·028 (1)	0·3032 (6)	0·8397 (5)	6·7 (4)
O(33)	0·065 (1)	0·1843 (6)	0·9334 (5)	8·1 (5)
C(4)	−0·140 (2)	0·387 (2)	0·507 (1)	13·3 (16)
F(41)	−0·195 (2)	0·304 (1)	0·530 (1)	22·2 (14)
F(42)	−0·022 (1)	0·3701 (9)	0·5084 (7)	11·9 (7)
F(43)	−0·183 (1)	0·4225 (8)	0·4487 (6)	10·7 (6)
S(4)	−0·2140 (5)	0·4109 (4)	0·5650 (2)	8·9 (3)
O(41)	−0·336 (1)	0·4194 (9)	0·5596 (6)	9·3 (6)
O(42)	−0·162 (2)	0·4994 (8)	0·532 (1)	20·5 (12)
O(43)	−0·147 (1)	0·3693 (6)	0·6230 (5)	6·3 (4)

Table 4. *Main interatomic distances* (Å) *and bond angles* (°) *for compound* (2) *with e.s.d.'s in parentheses*

Cu(1)–N(11)	2·01 (1)	Cu(2)–N(21)	2·01 (1)
Cu(1)–N(12)	2·02 (1)	Cu(2)–N(22)	2·04 (1)
Cu(1)–N(13)	2·031 (9)	Cu(2)–N(23)	2·03 (1)
Cu(1)–N(14)	2·02 (1)	Cu(2)–N(24)	2·03 (1)
Cu(1)–O(11)	2·472 (9)	Cu(2)–O(32)	2·49 (1)
Cu(1)–O(21)	2·434 (9)	Cu(2)–O(43)	2·41 (1)
N(11)–Cu(1)–N(12)	91·1 (5)	N(21)–Cu(2)–N(22)	89·9 (4)
N(12)–Cu(1)–N(13)	89·7 (5)	N(21)–Cu(2)–N(23)	89·1 (5)
N(13)–Cu(1)–N(14)	89·1 (5)	N(23)–Cu(2)–N(24)	90·8 (5)
N(11)–Cu(1)–N(14)	90·1 (5)	N(22)–Cu(2)–N(24)	90·1 (4)
O(11)–Cu(1)–N(11)	90·5 (4)	O(32)–Cu(2)–N(21)	92·0 (4)
O(11)–Cu(1)–N(12)	88·4 (4)	O(32)–Cu(2)–N(22)	90·8 (4)
O(11)–Cu(1)–N(13)	88·8 (3)	O(32)–Cu(2)–N(23)	86·2 (4)
O(11)–Cu(1)–N(14)	89·8 (4)	O(32)–Cu(2)–N(24)	87·4 (4)
O(21)–Cu(1)–N(11)	91·1 (4)	O(43)–Cu(2)–N(21)	89·6 (4)
O(21)–Cu(1)–N(12)	88·4 (4)	O(43)–Cu(2)–N(22)	90·6 (4)
O(21)–Cu(1)–N(13)	89·6 (3)	O(43)–Cu(2)–N(23)	92·5 (4)
O(21)–Cu(1)–N(14)	93·3 (4)	O(43)–Cu(2)–N(24)	90·9 (4)
C(111)–N(11)–Cu(1)	121·6 (9)	C(119)–N(11)–Cu(1)	120·3 (8)
C(111)–N(11)–C(119)	118· (1)	C(211)–N(21)–Cu(2)	122·8 (8)
C(219)–N(21)–Cu(2)	119·2 (8)	C(211)–N(21)–C(219)	118. (1)

$9{\cdot}5 < \theta < 10{\cdot}5°$; $(\sin\theta)/\lambda$ range: 0·04–0·48 Å^{-1}; scan range: $1{\cdot}10° + 0{\cdot}345 \tan\theta$; four standard reflections measured every other hour; 8373 independent reflections collected, and 6293 reflections kept [$F > 3\sigma(F)$]; method used to solve structure: *MULTAN* (Germain, Main & Woolfson, 1971); weighting scheme: $1{\cdot}47/\sigma^2$; average shift/e.s.d.: 0·08 in the last cycle; $N_{\text{reflections}}/N_{\text{var.par.}}$: 5·6 (refined by blocks); $R(F) = 0{\cdot}071$; $wR = 0{\cdot}049$; max. height in final difference Fourier synthesis: 0·3 e Å^{-3}; $S = 1{\cdot}47$; refinement carried out with isotropic thermal parameters for isoquinoline atoms and anisotropic thermal parameters for copper and anion atoms (better R than with each ligand as rigid group), owing to the large number of parameters; H coordinates calculated and kept fixed. Tables 3 and 4 show atomic parameters, selected interatomic distances and angles.

Discussion. Compound (1). The unit cell contains one $[CuL_2(H_2O)_2](ClO_4)_2$ entity with Cu located on an inversion centre. The copper environment is octahedral (Fig. 1), with a planar basal plane: two oxygens of two water molecules in *trans* positions and two nitrogens of two ligand molecules in *trans* positions. O(1) and O′(1) of apical perchlorate anions are semicoordinated (Table 2) to Cu and the Cu–O(1) vector is perpendicular to the basal plane. O(2) and O(4) of each perchlorate anion build hydrogen bonds with oxygen

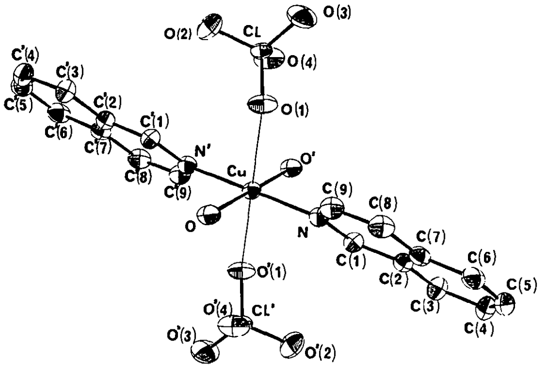

Fig. 1. *ORTEP* view (Johnson, 1976) of the molecule for compound (1) (atoms X and X' are related through the inversion centre).

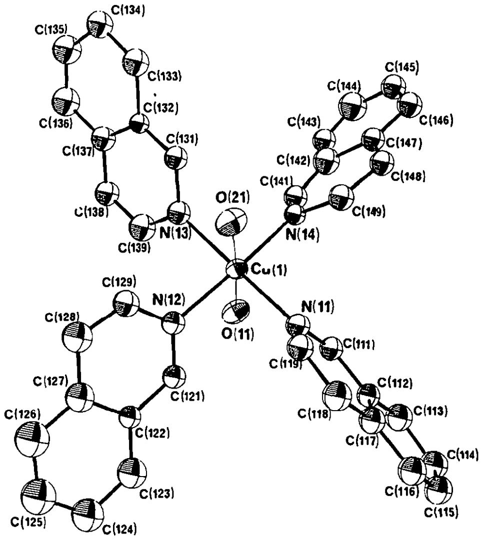

Fig. 2. *ORTEP* view (Johnson, 1976) of one of the two molecules constituting the asymmetric unit for compound (2).

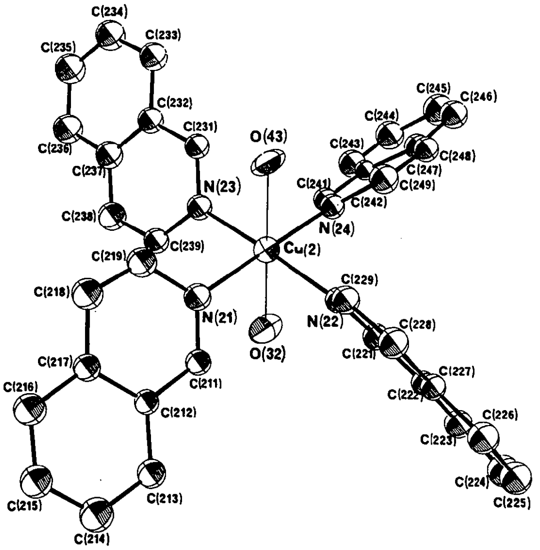

Fig. 3. *ORTEP* view (Johnson, 1976) of the other molecule constituting the asymmetric unit for compound (2).

atoms of two water molecules belonging to two other entities. Distances O(2)–first water oxygen and O(4)–second water oxygen are 2·79 and 2·84 Å, with angles around the corresponding hydrogens equal to 171 and 156·3°, respectively. Each perchlorate anion is then connected to three different Cu atoms through a first O(2)—O_{H_2O} hydrogen bond, and through a second O(4)—O_{H_2O} hydrogen bond involving a different water molecule, in addition to semicoordinated O(1).

Compound (2). The unit cell contains four $[CuL_4(CF_3SO_3)_2]$ molecules corresponding to two asymmetric units (Fig. 2 and 3). For both coppers, the environment is octahedral. The basal plane includes four N atoms of four isoquinoline molecules arranged as propeller blades. Two trifluoromethanesulfonate anions are semicoordinated by oxygen in apical positions (Table 4). The dihedral angle between the two basal planes of Cu(1) and Cu(2) is 81°.

References

Agambar, C. A. & Orrell, K. G. (1969). *J. Chem. Soc. A*, p. 897.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

International Tables for X-ray Crystallography (1974). Vol. IV. Tables 2.2B and 2.3.1. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.

Patel, R. N. & Ramana Rao, D. V. (1966). *Indian J. Chem.* **1**, 36.

Sheldrick, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1987). C**43**, 1264–1267

Structure of Bis(pyrazine)-5,10,15,20-tetraphenylporphinatoiron(II), TPPFe(pyz)$_2$

By Wolfgang Hiller

Institut für Anorganische Chemie der Universität Tübingen, Auf der Morgenstelle 18, *D*-7400 *Tübingen, Federal Republic of Germany*

and Michael Hanack and Martin G. Mezger

Institut für Organische Chemie der Universität Tübingen, Auf der Morgenstelle 18, *D*-7400 *Tübingen, Federal Republic of Germany*

(*Received* 11 *August* 1986; *accepted* 27 *February* 1987)

Abstract. $[Fe(C_{44}H_{28}N_4)(C_4H_4N_2)_2]$, $M_r = 828\cdot77$, monoclinic, $P2_1/c$, $a = 13\cdot157\ (4)$, $b = 16\cdot000\ (5)$, $c = 19\cdot350\ (5)$ Å, $\beta = 93\cdot15\ (5)°$, $V = 4067\cdot3$ Å^3, $Z = 4$, $D_x = 1\cdot353$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1\cdot54184$ Å, $\mu = 33\cdot395$ cm^{-1}, $F(000) = 1720$, $T = 293$ K. The final R value is 0·052 for 3612 significant $[I > 3\sigma(I)]$ reflections. The octahedral FeN_6 coordination group exhibits equatorial Fe—N bond lengths from 1·975 (3) to 1·993 (2) Å; the different Fe—N bonds to the axial pyrazine ligands [1·970 (3), 2·010 (3) Å] are attributable to steric repulsions.

Introduction. The stereochemical parameters of the six-coordinate iron(II) porphyrins are of interest because of their physical properties and as models for conducting bridged macrocyclic metal complexes (Hanack, 1983, 1985). Only two structures of bis(amine) derivatives of tetraphenylporphinatoiron(II) (TPPFe) are known. These are the compounds with the bis-axial ligands piperidine (Radonovich, Bloom & Hoard, 1972) and 1-methylimidazole [the latter has been cited (Scheidt, 1977) but, to the best of our knowledge, not yet published (Steffen, Reed, Sayler &

0108-2701/87/071264-04$01.50